FIRST YEAR DIPLOMA IN PHARMACY

PH

A LABORATORY MANUAL FOR PHARMACEUTICAL CHEMISTRY I

Maharashtra State Board of Technical Education, Mumbai
(Autonomous) (ISO 9001 : 2015) (ISO / IEC 27001 : 2013)

VISION

To ensure that the Diploma level Technical Education constantly matches the latest requirements of technology and industry and includes the all-round personal development of students including social concerns and to become globally competitive, technology led organization.

MISSION

To provide high quality technical and managerial manpower, information and consultancy services to the industry and community to enable the industry and community to face the changing technological and environmental challenges.

QUALITY POLICY

We, at MSBTE are committed to offer the best in class academic services to the students and institutes to enhance the delight of industry and society. This will be achieved through continual improvement in management practices adopted in the process of curriculum design, development, implementation, evaluation and monitoring system along with adequate faculty development programmes.

CORE VALUES

MSBTE believes in the followings:

- Education industry produces live products.
- Market requirements do not wait for curriculum changes.
- Question paper is the reflector of academic standards of educational organization.
- Well designed curriculum needs effective implementation too.
- Competency based curriculum is the backbone of need based program.
- Technical skills do need support of life skills.
- Best teachers are the national assets.
- Effective teaching learning process is impossible without learning resources.

A Laboratory Manual for

Pharmaceutical Chemistry - I

(Inorganic Pharmaceutical Chemistry)

(0806)

Maharashtra State

Board of Technical Education, Mumbai

(Autonomous) (ISO-9001-2015) (ISO/IEC 27001:2013)

Maharashtra State Board of Technical Education,
(Autonomous) (ISO 9001 :2015) (ISO/IEC 27001 : 2013)
4th Floor, Government Polytechnic Building, 49, Kherwadi,
Bandra (East), Mumbai - 400051.
(Printed on June, 2014)

MAHARASHTRA STATE BOARD OF TECHNICAL EDUCATION

Certificate

This is to certify that Mr. / Ms. _____

Roll No. _____, of **First Year Diploma in Pharmacy** has completed the term work satisfactorily in **Pharmaceutical Chemistry-I (0806)** for the academic year 200 _____ to 200 _____ as prescribed in the MSBTE curriculum.

Place : _____ Enrollment No.: _____

Date : _____ Exam. Seat No.: _____

Subject Teacher **Principal**

Seal of Institution

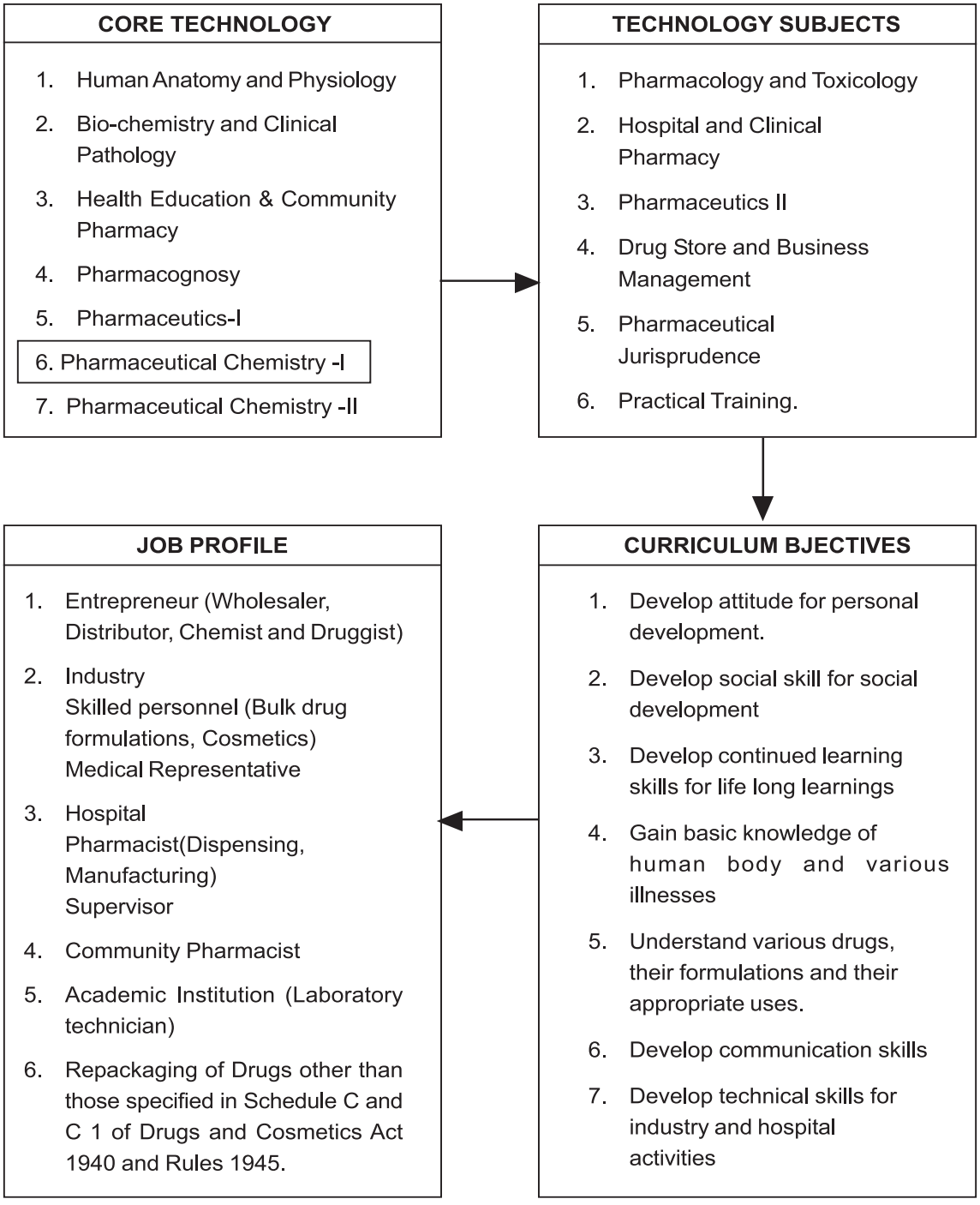
LEARNING OVERVIEW

IMPORTANCE OF SUBJECT :

1. The practicability of obtaining a reasonable standard of purity in raw material or in final product /drug/medicine.
2. To decrease the cumulative and toxic effects of impurities present in drugs.
3. By performing the tests like
   a. Assays
   b. Limit tests
   c. Identification tests

   One can uphold the practicability of drugs for internal use.
4. After performing the tests, the final product of good commercial quality can be obtained.

LINK/BLOCK DIAGRAM SHOWING INTER RELATIONSHIP OF SUBJECT AREAS, CURRICULUM OBJECTIVE AND JOB PROFILE

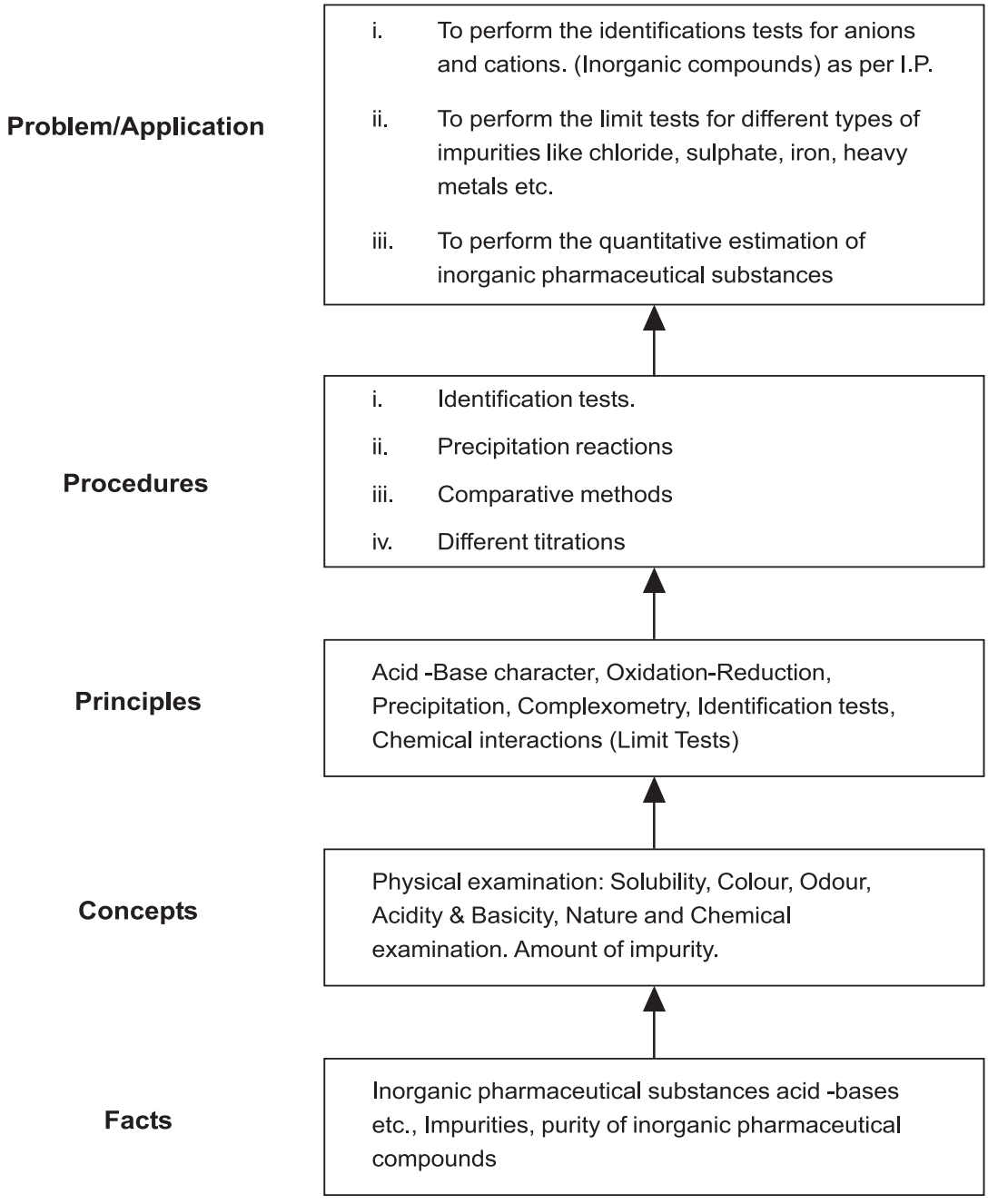


GRAPHICAL STRUCTURE OF SUBJECT AREA

FIRST YEAR DIPLOMA IN PHARMACY

PHARMACEUTICAL CHEMISTRY- I

(Inorganic Pharmaceutical Chemistry)



DEVELOPMENT OF SKILLS

A. Intellectual Skills:

1. To identify the impurity in the given sample. I1
2. To understand the concept of assay. I2
3. To identify the titrant and titrate. I3
4. To understand the concept of factor calculation. I4
5. To understand the concept of percentage purity. I5
6. To understand the difference between iodimetry and iodometric type of titration. I6
7. To understand the concept of volume strength calculation. I7
8. To understand the concept of complex formation. I8
9. To understand the concept of back titration. I 9
10. To understand the concept of blank titration I10.
11. To understand the concept of direct titration I11.
12. To understand the concept of argentimetry. I12
13. To understand the concept of identification test. I13
14. To understand the chemical reaction of sample. I14

B. Motor Skills:

1. To observe the opalescence in the given sample. M1
2. To compare the opalescence of test solution with standard solution. M2
3. To observe the colour intensity in test solution and standard solutionM3
4. To compare the colour intensity of test solution with standard solution. M4
5. To observe the stain produced by the standard and test solution M5
6. To compare the stain produced by the standard and test solution. M6
7. To observe the correct meniscus of solution in burette. M7
8. To observe the formation of carbon dioxide in the reaction. M8
9. To observe the colour change at the end point of titration. M9
10. To observe the precipitate formation in titration. M10
11. To identify the physical properties of given compound. M11
12. To observe the solubility in water and other solvent. M12
13. To observe the colour developed in the test solution. M13
14. To observe the precipitate formed in the test solution. M14
15. To observe the efflorescence of the compound. M15
16. To observe the evolution of gas from the test tube. M16
17. To observe the colour change in the flame of test. M17

GRID TABLE

Following table gives grid of the experiments and related intellectual and motor skills.

- ❖ Teacher shall ensure development of generic skills during the practical.
- ❖ Students are expected to focus on acquiring specific skills mentioned therein.

Sr. No.	Experiment No. & Title	Intellectual Skill	Motor skill
1	Introduction to Laboratory	--	--
2	Study of Laboratory Equipment &	--	--
	Glass Wares		
3	Limit Test for chloride	I1	M1, M2
4	Limit Test for Sulphate	I1	M1.M2
5	Limit Test for Iron	I1	M3, M4
6	Limit Test for Heavy Metal	I1	M3, M4
7	Limit Test for Arsenic	I1	M5, M6
8	Assay of Sodium bicarbonate	I2, I3, I4, I5	M7, M8, M9
9	Assay of Boric Acid	I2, I3, I4, I5	M7, M9
10.	Assay of Zinc Oxide	I2, I3, I4, I5, I9,I10	M7, M9
11.	Assay of Ferrous Sulphate	I2, I3, I4, I5	M7, M9
12.	Assay of Iodine	I2, I3, I4, I5, I6	M7, M9
13.	Assay of Hydrogen Peroxide	I2, I3, I4, I5, I7	M7, M9
14.	Assay of Magnesium Sulphate	I2, I4, I5, I8	M7, M9
15.	Assay of Calcium Gluconate	I2, I4, I5, I8	M7, M9
16.	Assay of Sodium Chloride (Mohr's Method)	I4, I5, I11, I12	M9, M10
17.	Assay of Ammonium Chloride (Volhard's Method)	I2,I3,I4,I5,I9,I10	M9, M10
18.	Identification Tests for Sodium Chloride.	I13,	M11M12, M13, M14
19.	Identification Tests for Sodium bicarbonate	I13	M11, M12, M13
20.	Identification Tests for Magnesium Sulphate	I13	M11, M12, M14

Sr. No.	Experiment No. & Title	Intellectual Skill	Motor skill
21.	Identification Tests for Ferrous Sulphate.	I13	M11, M12, M15
22.	Identification Tests for Sodium Acetate	I13	M11, M12, M13
23.	Identification Tests for Hydrogen Peroxide.	I13, I14	M13, M16
24.	Identification Tests for Boric Acid.	I13	M11, M12, M17
25.	Identification Tests for Ammonium Chloride	I13	M11, M12, M13,

NOTE: numbers indicates identified skills

STRATEGY FOR IMPLEMENTATION

It is suggested that 40 to 50% experiments shall be completed in first term and remaining experiments in second term.

GUIDELINES FOR TEACHERS

Teachers shall discuss the following points with students before starting of practicals of the subject.

1. **Learning Overview:** To develop better understanding of importance of the subject. To know related skills to be developed such as intellectual skills and motor skills.
2. **Link/Block Diagram:** Context of the subject in the form of link diagram showing interrelationship of various subject areas, curriculum objectives and job profile.
3. **Graphical structure:** In this topics and sub topics are organized in a systematic way so that ultimate purpose of learning the subject is achieved. This is arranged in the form of fact, concept, principle, procedure and application.
4. **Know your laboratory work:** To understand the layout of laboratory including water, electrical and gas connection specifications of Equipment/Instruments/Materials, procedure, working in groups, planning time etc. Also to know total amount of work to be done in the laboratory.
5. Teacher shall ensure that required equipment are in working condition before starting of experiment, also keep Standard Operating Procedures of equipments.
6. Explain prior concepts, specific apparatus/instrument/machine to the students before starting of each experiment.
7. Involve students' activity at the time of conduct of each experiment.
8. List of questions is given at the end of each experiment. Teacher shall instruct the student to attempt all questions given at the end of each experiment/exercise. Teacher shall ensure that each student writes the answers to the allotted questions in the laboratory manual after the performance is over.
9. Teacher shall assess the performance of students continuously as per the norms prescribed by MSBTE.
10. Teacher shall ensure that the respective skills and competencies are developed in the students after the completion of the practical exercise.
11. Teacher is expected to share the skills and competencies to be developed in the students.
12. Teacher may provide additional knowledge and skills to the students even though not covered in the manual but are expected from the students by the industries or profession.
13. Teachers shall ensure that industrial, analytical laboratory visits recommended in the manual are covered.
14. Teacher may suggest the students to refer additional related literature of the technical papers/ reference books/seminar proceedings, etc.
15. During assessment teacher is expected to ask questions to the students to tap their achievements regarding related knowledge and skills so that students can prepare while submitting record of the practicals. Focus should be given on development of enlisted skills rather than theoretical/codified knowledge.
16. Teacher shall enlist the skills to be developed in the students that are expected by the industry and profession.
17. Teacher shall organize group discussions/brain storming sessions/seminars to facilitate the exchange of knowledge amongst the students.

18. Teacher shall ensure that revised CIAAN-2004 norms are followed simultaneously and progressively.
19. Teacher shall give more focus on hands on skills and shall actually share the same.
20. Teacher shall also refer to the Circular No. MSBTE/D-50/Pharm Lab Manual/2006/3160 dated 4th May 2006 for additional guidelines.
21. Teacher shall instruct the students that dummy compound can also be given in place of genuine compound for identification tests experiments, in the examination.
22. While setting practical examination, teacher shall see that the students will be able to complete the practical, considering time required for viva and synopsis.
23. Teacher shall assign experiments to the student in such a way that they have to refer Indian Pharmacopoeia and should see that the students are well acquainted with the Pharmacopoeia.
24. Teacher may assign one or more experiments in a practical depending on the time requirement of the experiments.
25. Teachers shall explain all subtitles of the experiment thoroughly to the students before starting of the actual experiment.

INSTRUCTIONS FOR STUDENTS

Students shall read the points given below for understanding the theoretical concepts and practical applications.

1. Students should note the following essential requirements for pharmaceutical chemistry practical: I-card, practical manual, rough journal, locker key, laboratory coat, weight box, match box, test tube holder, soap, washing powder, napkin, butter paper etc.
2. Listen carefully to the lecture given by teacher about importance of subject, curriculum philosophy, graphical structure and skills to be developed, information about equipment, instruments, procedures, method of continuous assessment, tentative plan of work in laboratory and total amount of work to be done in a year. Student shall clear all his doubts before starting the practical.
3. Students shall note down important points in the rough journal, when teacher is explaining the experiments.
4. Students shall undergo study visit of the laboratory for types of equipments, instruments, and material to be used, before performing experiments.
5. Read the write up of each experiment to be performed a day in advance.
6. Organize the work and arrange the experiment by procuring required apparatus and chemicals.
7. Follow time management and complete all experiment in time.
8. Understand the purpose of experiment and its practical implications.
9. Students may record the initial observations in rough notebook. However all observations shall be recorded in the manual and obtain initials of the teacher. Finally after calculations and conclusion the student shall obtain the signature of the teacher.
10. Students should maintain discipline in the laboratory. Any misbehavior or indiscipline in the laboratory may lead to serious mishap.
11. Students should handle hazardous chemicals carefully. They should wear hand gloves while handling corrosive chemicals like bromine, concentrated acids and alkalies etc.

12. Students should wash their hands thoroughly with soap, before leaving laboratory.
13. Write the answers to the questions allotted by the teacher during practical hours if possible or afterwards, before next practical.
14. Student should not hesitate to ask any difficulty faced during conduct of practical/exercise.
15. The student shall study all the questions given in the laboratory manual and practice to write the answers to these questions.
16. Students shall visit the recommended industries and analytical laboratory. Student shall also write brief report of the visit at the end of manual.
17. Student shall develop maintenance and working skills as expected by the industries or medical shops.
18. Student shall develop the habit of pocket discussion/group discussion related to the experiments/ exercises so that exchange of knowledge/skills can take place.
19. Student shall attempt to develop related hands-on-skills and gain confidence.
20. Student shall focus on development of skills rather than theoretical or codified knowledge.
21. Student shall visit the nearby medical shop, industries, laboratories, technical exhibitions, trade fair etc. Even though not included in the lab manual. In short, students should have exposure to the area of work right in the student hood.
22. Student shall insist for the completion of recommended laboratory work, industrial visits, answers to the given questions, etc.
23. Student shall develop the habit of evolving more ideas, innovations, skills etc. than included in the scope of the manual.
24. Student shall refer technical, pharmaceutical, health magazines, and proceedings of the seminars, refer websites related to the scope of the subjects and update their knowledge and skills.
25. Student should develop the habit of not to depend totally on teachers but to develop self-learning techniques with the help of library and group discussions.
26. Student should develop the habit to react with the teacher without hesitation with respect to the academics involved.
27. Student should develop habit to submit the practicals exercise continuously and progressively on the scheduled dates and should get the assessment done.
28. Student should be well prepared while submitting the write up of the exercise. This will develop the continuity of the studies and he will not be over loaded at the end of the term.
29. Do not use cell-phone in the laboratory.

List of Experiments & Record of Progressive Assessment

Sr. No.	Experiment No.with Title	Page No	Date of Perfor-mance	Date of submi - ssion	Assess-ment Max. Marks 10	Sign. of teacher & Remarks
1.	Introduction to laboratory.	1				
2.	Study of laboratory equipment & glassware.	5				
3.	Limit test for chloride.	12				
4.	Limit test for sulphate.	17				
5.	Limit test for iron.	22				
6.	Limit test for heavy metal.	27				
7.	Limit test for arsenic.	32				
8.	Assay of sodium bicarbonate.	38				
9.	Assay of boric acid.	44				
10.	Assay of zinc oxide.	50				
11.	Assay of ferrous sulphate.	56				
12.	Assay of iodine.	62				
13.	Assay of hydrogen peroxide.	68				
14.	Assay of magnesium sulphate.	75				
15.	Assay of calcium gluconate.	81				
16.	Assay of sodium chloride.	86				
17.	Assay of ammonium chloride.	90				
18.	Identification tests of sodium chloride.	96				
19.	Identification tests of sodium bicarbonate.	101				
20.	Identification tests of magnesium sulphate.	106				

Sr. No.	Experiment No.with Title	Page No	Date of Perfor-mance	Date of submi - ssion	Assess-ment Max. Marks 10	Sign. of teacher & Remarks
21.	Identification tests of ferrous sulphate	111				
22.	Identification tests of sodium acetate.	116				
23.	Identification tests of hydrogen peroxide.	121				
24.	Identification tests of boric acid.	125				
25.	Identification tests of ammonium chloride	130				
					Total Marks ……. Average Marks out of 10……*	

* To be transferred to Proforma of CIAAN-200 (Proforma I-1)

(**NOTE :** The guidelines for conduct of Annual Practical Examination are enclosed in the end at page number 142)

Experiment No. 1

1.0 Title :

To know your inorganic pharmaceutical chemistry-I laboratory.

2.0 Prior Concepts :-

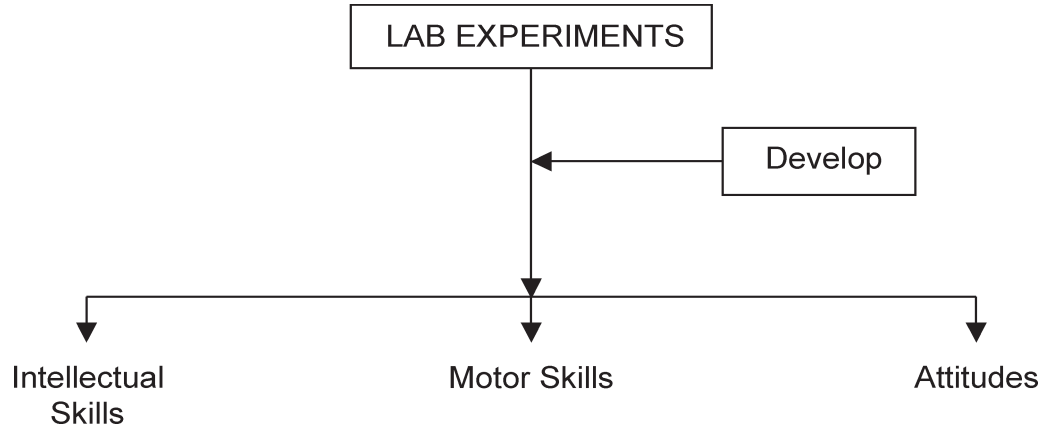
1. Definition of chemistry and classification of branches of it.
2. Inorganic chemistry and its contents.
3. Application of inorganic chemistry.

3.0 New Concepts :-

1. Inorganic pharmaceutical chemistry.
2. Applications of inorganic pharmaceutical chemistry in pharmaceutical sciences.
3. Definition of pharmaceutical sciences.

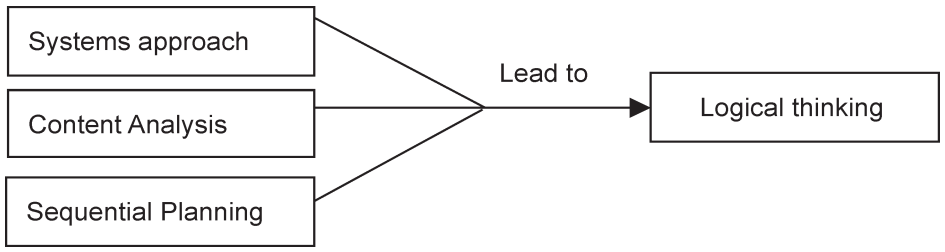
Proposition 1 :-

Laboratory experiments are expected to develop intellectual skills, motor skills and attitudes in students.



Proposition 2 :-

Logical thinking is developed in students through systems approach, content analysis and sequential planning of laboratory work.



Proposition 3 :-

In the laboratory, students will perform the experiments by using various, apparatus like, burettes, pipettes, Nesslers cylinder, Arsenic limit test apparatus, thermometer etc.

4.0 Procedure:-

1. Standard Operating Procedures (SOP's) for analytical balance.
2. Listen to the lecture given by teacher about importance of subject, curriculum philosophy and graphical structure.
3. Skills to be developed, information about equipment, instruments. SOP's

   (Standard Operating Procedures) of continuous assessment and tentative plan of work in laboratory.
4. Visit the laboratory for types of equipments, instruments and materials to be used while performing experiments.

5.0 Observations:- (Student shall write the information)

Sr. No.	Name of Equipment/Instrument	Specifications Size/diameter/capacity	Use in Laboratory	Use in Industry
1.				
2.				
3.				
4.				
5.				
6.				
7.				
8.				
9.				
10.				

Give five chemicals / reagents on the rack		Specifications Size/diameter/capacity	Use in Laboratory	Use in Industry
1.				
2.				
3.				
4.				
5.				

Give two names of Scientists displayed in laboratory		Work of Scientist		
1.				
2.				
Give two names of two charts displayed in laboratory		Title of Chart		
1.				
2.				
Give two names of two experimental set ups in laboratory		Purpose of Experiment		
1.				
2.				

6.0 Questions:- Answer Q..... from Group A and Q....., Q..... from Group B and Q....., Q..... from Group C. Question numbers are to be allotted by the teacher.

Group A:

1. What is the importance of the link diagram of the curriculum of the subject?
2. List two types of skills given in the experiment.
3. How graphical structure is useful in understanding the scope of the subject?
4. Write the chronological sequence of terms of graphical structure. (Refer graphical structure)
5. List the sub heads of curriculum for the subject.

Group B:

1. Differentiate between the inorganic pharmaceutical chemistry-I laboratory and pharmaceutics laboratory.
2. Draw layout of inorganic pharmaceutical chemistry laboratory-I.
3. Name any five apparatus used in inorganic pharmaceutical chemistry practical.
4. Enlist the acids and bases (reagents) used in inorganic pharmaceutical chemistry.
5. Write the instructions for inorganic pharmaceutical chemistry laboratory.

Group C:

1. Write a note on fire hazards in inorganic pharmaceutical chemistry laboratory.
2. Write the names of elements of VII A group of periodic table.
3. List out the reference books for inorganic pharmaceutical chemistry laboratory experiments.
4. State the procedure for cleaning of glass apparatus?
5. What care is to be taken while using inflammable chemicals?

(Space for answers)

Experiment No. 2

1.0 Title :

To study the different laboratory equipments and general glasswares used in pharmaceutical chemistry-I practical.

2.0 Prior Concepts :

Laboratory equipments, glasswares, constructions and applications of laboratory equipments.

3.0 New Concepts :

1. Analytical weighing balances and different weights.
2. Different glasswares.

4.0 Study of Laboratory Equipments:

4.1 Weighing Balance:

In the experiments involving quantitative analysis, weighing is an important operation.

The tool/equipment employed for weighing purpose is called as a 'balance'/weighing balance.

There are various balances with varied degrees of sensitivity as Follows

1. Physical balance.
2. Analytical balance.
3. Triple beam balance.
4. Torsion type balance.
5. Single pan electronic balance.
6. Platform electronic balance.

Amongst above all types of balances, generally 'analytical balance' is used for any quantitative analysis.

4.2 Analytical balance:

The followings are the important parts of the analytical balance

1. Supports, 2. Pans, 3. Plumb line, 4. Beam, 5. Pointer, 6. Graduated scale and 7. Knob.

The most important part of the analytical balance is beam 4 which is an equal-armed lever furnished with long vertical pointer 5, by the deflection of which the movement of the beam can be judged. The lower end of the pointer swings in front of scale 6 with divisions for reading the amplitude of swing. The scale has a zero line in the middle and 10 divisions on either side of it. When the beam is horizontal (the pans are balanced) the pointer should rest opposite the zero line of the scale.

The beam has agate knife-edges at its extremes, supporting stirrups from which balance pans 2 are suspended. Another agate or steel knife-edge is fixed exactly in the middle of the beam on its bottom side. This knife-edge faces downwards and supports the beam.

To prevent the knife-edge from becoming dull under the weight of the beam and pans. The balance is equipped with a special device called an arrest. It bears the weight of the beam and pans when weighing is not being done and the balance is at rest. The arrest is operated by means of milled knob 7 underneath the base plate in the middle and in front of the balance (sometimes the arrest knob is at one side of the balance). To put the balance in its operating position the arrest is lowered by rotating the knob, which releases the beam and sets its knife-edges on their cushions. To stop the movement of the beam, the knob is rotated in the opposite direction as far as it will go, which raises the arrest. A balance with its arrest raised is said to be arrested. When not in use and during loading or unloading of the pans, the balance should be arrested.

4.2 Weight box:

The weights are made up of heavy metals alloy and are cylindrical in shape, each with a knob at the top. An ordinary set of weight contains, 100 g, 50 g, 20 g, 10 g, 5 g, 2 g and 1 g weights. Weights smaller than 1 g are called as 'milligram' (mg) weights or fractional weights. They are leaf shaped foils of aluminum or other suitable metal with one of the sides turned up for picking up with forceps.

The fractional weights have 3 sets of 500 mg, 200 mg, 100 mg, 50 mg, 20 mg, 10 mg, 5 mg, 2mg and 1 mg weights. Weights smaller than 10 mg are generally not used. For this purpose 'rider' is used.

4.3 Rules in weighing:

To keep the balance in working order and to obtain accurate results the following rules should be observed:

1. Sit squarely in front of the balance while weighing.
2. Keep no objects inside the balance case except beakers with calcined calcium chloride (to absorb moisture), which should be placed in the back corners of the case. Keep the balance meticulously clean. If anything gets spilled on the pan or the case bottom, sweep it out immediately with a soft brush. Wipe the pans with chamois leather; never use liquids of any kind to clean them.
3. Align the balance strictly vertical by its plumb line. Do not move the balance after alignment; otherwise it will have to be readjusted.
4. When weighing, open only the side doors of the case; do not raise its front door.
5. Never place the substance being weighed directly on the pan or on paper. Weigh solids on a watch glass or in a beaker, and volatile and hygroscopic substances and liquids in weighing bottles. Glassware used for weighing should be clean and dry.
6. Objects being weighed should have the same temperature as the weighing room. To ensure this, keep them in a desiccator in the weighing room for at least 15 or 20 minutes before weighing.

7. Never load the balance above its maximum load (200 g).
8. Change the load on the balance only after fully arresting it.
9. Remove or add substance being weighed only outside the case.
10. When observing pointer deflections, keep the case doors shut.
11. Never touch weights, pans, or balance beam. Handle weights only with plastic-tipped forceps.
12. Place weights on the right-hand pan, arranging them in the centre to avoid skewing of the pan, which may distort the reading.
13. Open the box of weights only when weighing.
14. Before and after using, keep the rider on the carrier hook clear of the beam scale.
15. After finishing weighing, move the carrier inside the case.
16. Having finished weighing, arrest the balance and check your record of the results. Without opening the case doors, remove the rider from the beam scale using the carrier rod, open the right-hand case door, replace the weights in their places in the box, open the left-hand case door, remove the object weighed from the scale pan and close the case door.

When you have finished weighing, clean the balance with a brush or a piece of chamois leather to remove possible spillage or other dirt.

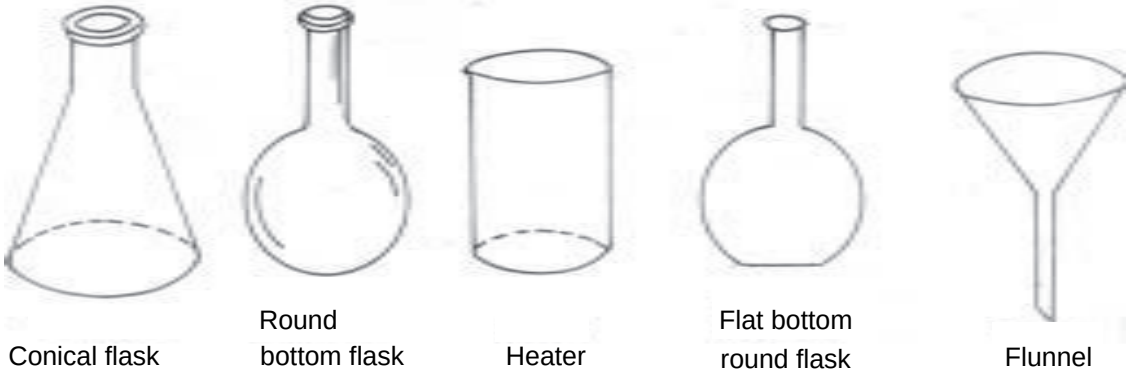
4.4 Glassware:

The pharmaceutical chemistry-I laboratory require different glassware for smooth conduction of practical viz:

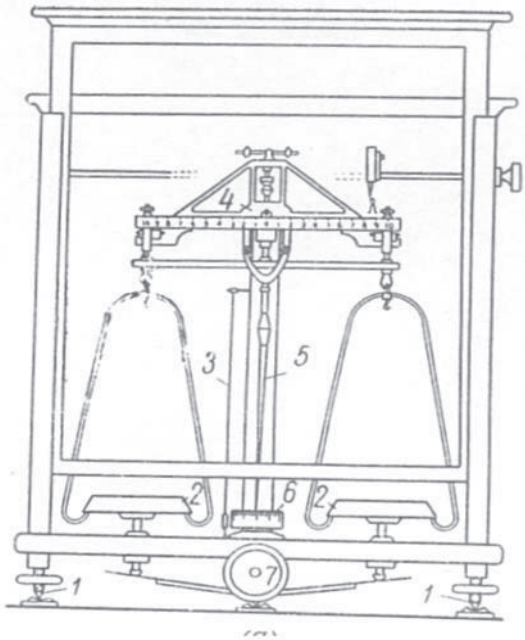
1. Burette, 2. Pipettes (graduated and volumetric).
3. Conical flask. 4. Measuring cylinder. 5. Beaker.
6. Volumetric flask.7. Test tube. 8. Reagent bottle.

The glassware's are made up of borosilicate glass. These are fragile, so care should be taken during the use of these glassware.

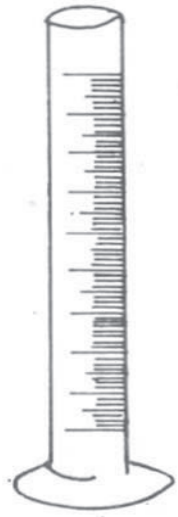
5.0 Diagrams:



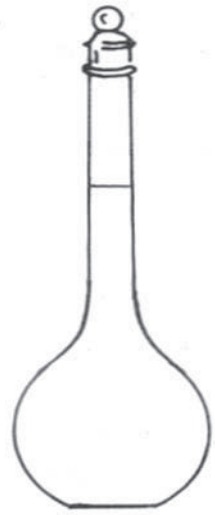
Conical flask | Round bottom flask | Heater | Flat bottom round flask | Flunnel



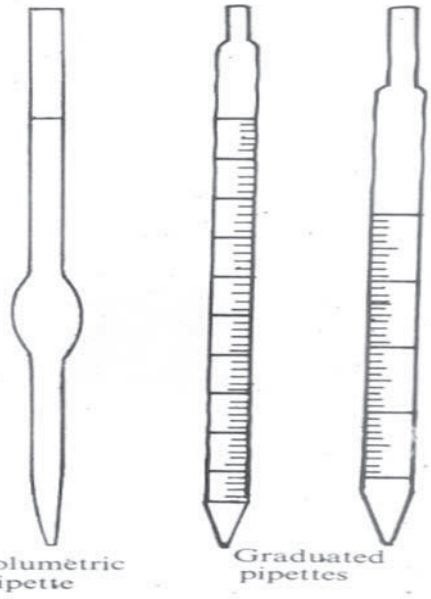
Analytical balance



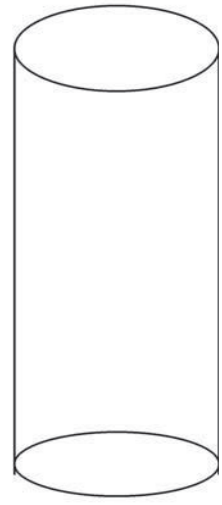
Measuring cylinder



Volumetric Flask



Test tube



Nesslers cylinder

6.0 Equipments / Instruments in Laboratory:

Sr. No.	Name of Equipment/Instrument	Specifications Size/Diameter/Capacity	Use in Laboratory	Use in Industry
1.	Analytical balance.			
2.	Electronic balance.			
3.	Burette.			
4.	Pipette:pipette			
	i. Graduated pipette			
	ii. Volumetric pipette			
5.	Conical flask.			
6.	Measuring cylinder.			
7.	Beaker.			
8.	Volumetric flask.			
9	Test tube.			
10.	Reagent bottle.			
11.	Nesslers cylinder.			

7.0 Questions:

(Answer the following questions, Q.… Q…..Q.…and Q.…., Question Number to be allotted by the teacher)

1. Enlist the balances used in weighing.
2. State the meaning of analytical balance.
3. Enlist four main parts of analytical balance with its working.
4. Write the rules of weighing.
5. How 5 mg samples can be weighed on analytical balance?
6. Write the use of rider.
7. State the meaning of fractional weight box.
8. State the procedure for cleaning of the analytical balance?
9. Write the maximum and minimum load weighed on analytical balance.
10. Enlist the glassware used in pharmaceutical chemistry-I laboratory.
11. Write the procedure for weighing of hygroscopic, volatile and liquid substances in analytical balance?
12. Differentiate volumetric pipette and graduated pipette.

(Space for answers)

(Space for answers)

Experiment No. 3

1.0 Title :

Limit test for chloride

(To perform and report the limit test for chloride on the given samples as per Indian Pharmacopoeia).

1. Sodium bicarbonate.
2. Potassium permanganate
3. Magnesium trisilicate

2.0 Prior Concepts :-

Impurities, sources of impurities, types of impurities.

3.0 New Concepts: Principle

Proposition 1: Chemical Interaction

Limit test for chloride depends upon the interaction of chlorides with silver nitrate in the presence of dilute nitric acid.

Proposition 2: Precipitation/Opalescence

Deposition of solid particle of chloride as silver chloride.

Proposition 3: Comparison of test sample with standard

Chloride as impurity is present in very small quantities. In the chemical interaction the precipitation of silver chloride appears as opalescence. It is compared under uniform conditions of illumination with standard opalescence in Nesslers cylinders to draw inference.

Proposition 4: Importance of limit tests:

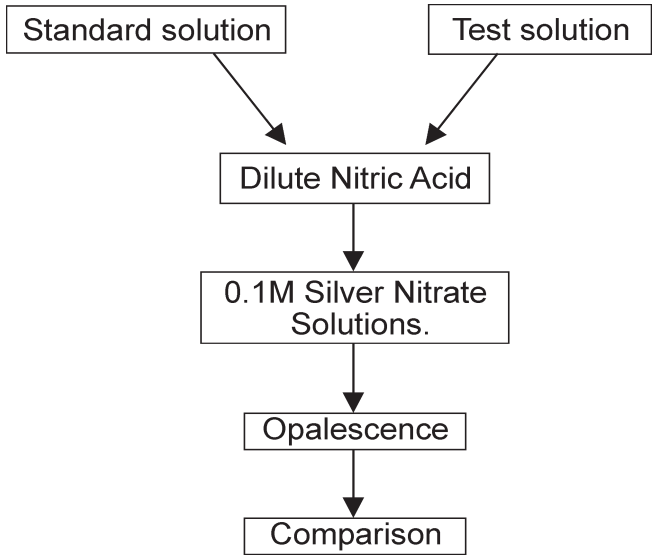
1. To find out the amount of harmful impurities.
2. To find out the avoidable/unavoidable amount of impurities.

Chemical Reaction:

$$Cl^- + AgNO_3 \xrightarrow{\text{Dilute } HNO_3} AgCl\downarrow + NO^-_3$$

(Soluble) (Soluble) (Precipitate) (Soluble)

General Concept Structure:



4.0 Learning Objectives:

4.1 Intellectual Skills:

1. To identify impurities in the given sample.

4.2 Motor Skills:

1. To observe the turbidity /opalescence in Nesslers cylinder.
2. To compare opalescence of test solution with standard solution.

5.0 Apparatus:

5.1 Materials:

A pair of Nesslers cylinder, Wooden stands, Glass rod, Pipette (10 ml, 25 ml) and Measuring cylinder (100 ml).

5.2 Chemicals:

Sodium bicarbonate, Potassium permanganate, Magnesium Trisilicate, 0.1 M Silver nitrate, dilute nitric acid and Sodium chloride, Chloride standard solution (25 ppm), distilled water and ethanol (95%).

6.0 Stepwise Procedure:

Standard solution	Test solution of sodium bicarbonate	Test solution of potassium permanganate	Test solution of magnesium trisilicate
1. Take 10 ml of chloride standard solution. (25 ppm Cl) in labeled Nesslers cylinder (S). 2. Add 5 ml of distilled water. 3. Add 10 ml of dilute nitric acid and mix well. 4. Dilute to 50 ml with distilled water. 5. Add 1 ml of 0.1 M silver nitrate solution. 6. Stir immediately with glass rod and allow to stand for 5 minutes, protect from light.	1. Weigh accurately 1.25 g of sodium bicarbonate 2. Dissolve it in 15 ml of distilled water in a labeled Nesslers cylinder (T). 3. Add 2 ml of dilute nitric acid and mix well. 4. Dilute to 50 ml with distilled water. 5. Add 1 ml of 0.1 M silver nitrate solution. 6. Stir immediately with glass rod and allow to stand for 5 minutes, protect from light. 7. View transversely, against a black background. 8. Compare the opalescence produced with that of standard solution.	1. Weigh accurately 1.50 g of potassium permanganate and transfer it in 250 ml conical flask. 2. Add 50 ml of distilled water, heat on water bath. 3. Add gradually 6 ml of ethanol (95%) and cool. 4. Dilute to 60 ml with distilled water and filter, the filtrate is colourless. 5. Take 40 ml of above filtrate in a labeled Nesslers cylinder (T) 6. Add 10 ml of dilute nitric acid. 7. Add 1 ml of 0.1 M silver nitrate solution. 8. Stir immediately with glass rod and allow to stand for 5 minutes, protect from light. 7. View transversely, against a black background. 8. Compare the opalescence produced with that of standard solution.	1. Weigh accurately 2.0 g of magnesium trisilicate and transfer it in 250 ml conical flask. 2. Add the mixture of 4 ml of nitric acid and 4 ml of distilled water and heat to boiling, shaking frequently. 3. Add 12 ml of distilled water, allow to cool, filter or centrifuge to obtain a clear solution. 4. Dilute the filtrate to 20 ml with distilled water. 5. Take 0.5 ml of above solution in a labeled Nesslers cylinder (T). 6. Dilute to 50 ml with distilled water. 7. Add 1 ml of 0.1 M silver nitrate solution. 6. Stir immediately with glass rod and allow to stand for 5 minutes, protect from light. 7. View transversely, against a black background. 8. Compare the opalescence produced with that of standard solution.

7.0 Observations:

1. Opalescence produced by sodium bicarbonate test solution is ..... (less/same/more) intense than that of standard solution.
2. Opalescence produced by potassium permanganate test solution is ..... (less/same/more) intense than that of standard solution.
3. Opalescence produced by magnesium trisilicate test solution is .... (less/same/more) intense than that of standard solution.

8.0 Results:

1. The given sample of sodium bicarbonate..... (complies/ does not comply) the limit test for chlorides as per the Indian Pharmacopoeia 1996.
2. The given sample of potassium permanganate..... (complies/ does not comply) the limit test for chlorides as per the Indian Pharmacopoeia 1996.
3. The given sample of magnesium trisilicate..... (complies/ does not comply) the limit test for chlorides as per the Indian Pharmacopoeia 1996.

9.0 Questions:

(Answer the following questions, Q.... Q.....Q....and Q....., Question Number to be allotted by teacher)

1. State the meaning of the term opalescence.
2. Write the principle of limit test for chlorides.
3. State the chemical equation of chloride limit test.
4. Give the reason for formation of opalescence.
5. Write the procedure for preparation of standard solution for chloride limit test I.P.
6. Write the procedure for preparation of sodium bicarbonate test solution for chloride limit test I.P.
7. Write the procedure for preparation of potassium permanganate test solution for chloride limit test I.P.
8. Write the procedure for preparation of magnesium trisilicate test solution for chloride limit test I.P.
9. State the reason for use of silver nitrate in the chloride limit test.
10. Why nitric acid is used in the chloride limit test give the reasons.

10.0 Reference:

Indian pharmacopoeia 1996

(Space for Answers)

Experiment No. 4

1.0 Title :

Limit test for sulphate.

(To perform and report the limit test for sulphate on the given samples as per Indian Pharmacopoeia).

1. Purified water
2. Sodium bicarbonate
3. Calcium gluconate.

2.0 Prior Concepts :-

Opalescence, Impurities, Supersaturation, Parts per millions.

3.0 New Concepts :- Principle

Proposition 1: Chemical Interaction

Limit test for sulphate depends upon the interaction of soluble sulphate with barium chloride in presence of alcohol and potassium sulphate.

Proposition 2: Opalescence/ precipitation

Deposition of solid particles of sulphate as barium sulphate.

Note: (barium chloride, alcohol and a small amount of potassium sulphate is used for this purpose, alcohol prevents supersaturation and potassium sulphate increases sensitivity of the test by giving ionic concentration in the reagent which just exceeds the solubility product of barium sulphate.)

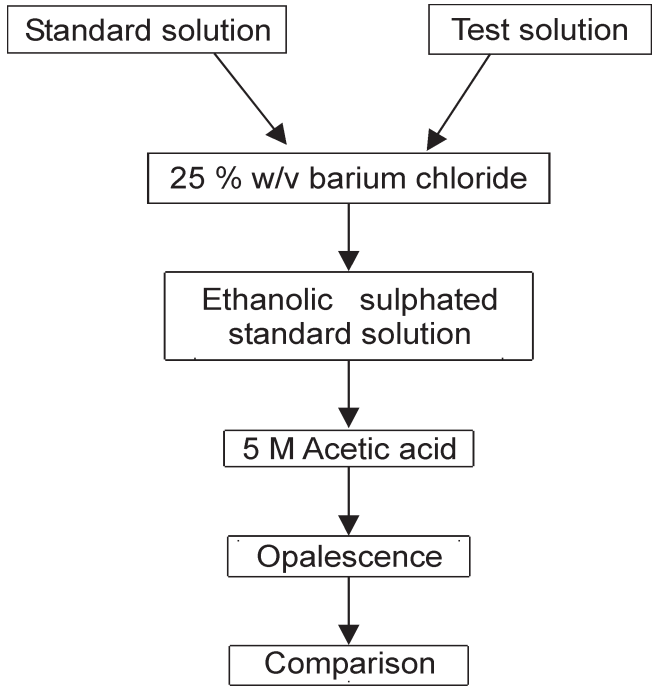
Proposition 3: Comparison of test Sample with standard

The opalescence/turbidity produced by sample under test is compared with standard sample opalescence/turbidity against the dark background.

Chemical Reaction:

$$BaCl_2 + SO^{2-}_4 \rightarrow BaSO_4 \downarrow +2Cl^-$$

General concept Structure:



4.0 Learning Objectives:

4.1 Intellectual Skills:

1. To identify impurities in the given sample.

4.2 Motor Skills:

1. To observe the opalescence in the given samples.
2. To compare the opalescence of test solution with standard solution.

5.0 Apparatus :-

5.1 Materials:

A pair of Nesslers cylinder, Wooden stands Glass rod, Pipette (10 ml and 25 ml) and Measuring Cylinder (100 ml).

5.2 Chemicals:

Sodium bicarbonate, calcium gluconate, purified water, alcohol, barium sulphate, ethanolic sulphated standard solution (10 ppm SO_4), 5 M acetic acid, barium chloride and 2 M hydrochloric acid.

6.0 Stepwise Procedure:

Standard solution	Test solution of purified water	Test solution of sodium bicarbonate	Test solution of calcium gluconate
1. Take 1 ml of 25% w/v solution of barium chloride in the Nesslers cylinder (S). 2. Add 1.5 ml of ethanolic sulphate standard solution(10 ppm SO_4) mix and allow to stand for 1 minute. 3. Add 0.15 ml of 5 M acetic acid. 4. Add sufficient water to produce 50 ml, stir immediately with glass rod. 5. Allow to stand for 5 minutes.	1. Take 10 ml of purified water in a labeled Nesslers cylinder (T). 2. add 0.1 ml of 2M hydrochloric acid. 3. And add 0.1 ml of 25% w/v barium chloride solution. 4. Observe the appearance of the solution for 1 hour. (Appearance of solution does not change for at least 1 hour)	1. Take 1 ml of 25% w/v solution of barium chloride in the labeled Nesslers cylinder (T). 2. Add 1.5 ml of ethanolic sulphate standard solution(10 ppm SO_4) mix and allow to stand for 1 minute. 3. Weigh accurately 1.0 g of sodium bicarbonate and add to a labeled Nesslers cylinder. 4. Add 10 ml distilled water, neutralise with hydrochloric acid and dilute to 15 ml with distilled water. 5. Add 0.15 ml of 5 M acetic acid. 6. Add sufficient water to produce 50 ml, stir immediately with glass rod and allow to stand for 5 minutes. 7. View transversely against a black background. 8. Compare opalescence with that of standard solution.	1. To 1 ml of 25% w/v solution of barium chloride in a labeled Nesslers cylinder (T). 2. add 1.5 ml of ethanolic sulphate standard solution, mix and allow to stand for 1 minute. 3. Weigh accurately 1.0 g calcium gluconate and add in labeled Nesslers cylinder. 4. Dissolve it by adding 15 ml distilled water. 5. Add 0.15 ml of 5 M acetic acid. 6. Add sufficient distilled water to produce 50 ml, stir immediately with glass rod and allow to stand for 5 minutes. 7. View transversely against a black background. 8. Compare opalescence with that of standard sulphate solution.

7.0 Observations:

1. There is..... (Change/no change) in appearance of test solution of purified water after 1 hour.
2. Opalescence produced by sodium bicarbonate test solution is.....(less/ same/more) intense than that of standard solution.
3. Opalescence produced by calcium gluconate test solution is……..…..... (less/ same/more) intense than that of standard solution.

8.0 Results:

1. The given sample of purified water.....(complies/does not comply) the limit test for sulphate as per the Indian pharmacopoeia 1996.
2. The given sample of sodium bicarbonate..... (complies /does not comply) the limit test for sulphate as per the Indian pharmacopoeia 1996.
3. The given samples of calcium gluconate..... (complies/does not comply) the limit test for sulphate as per the Indian pharmacopoeia 1996.

9.0 Questions:

(Answer the following questions, Q.… Q…..Q.…and Q….., Question Number to be allotted by the teacher)

1. State the meaning of the term impurities as applicable to drug.
2. Define supersaturation.
3. State the meaning of opalescence.
4. Write the procedure for preparation of standard solution for the test of sulphate impurities.
5. Write the procedure for preparation of test solution of purified water for test of sulphate impurities.
6. Write the procedure for preparation of test solution of sodium bicarbonate for test of sulphate impurities.
7. Write the procedure for preparation of test solution of calcium gluconate for test of sulphate impurities.
8. State the use of barium chloride and alcohol in limit test of sulphate.

10.0 Reference:

Indian pharmacopoeia 1996

(Space for Answers)

(Space for Answers)

Experiment No. 5

1.0 Title :

Limit test for Iron.

(To perform and report the limit test for Iron on the given samples as per Indian Pharmacopoeia).

1. Zinc sulphate.
2. Sodium chloride.
3. Magnesium sulphate.

2.0 Prior Concepts :-

Limit test, sources of iron impurities, colour intensity.

3.0 New Concepts: Principle

Proposition 1: Chemical Interaction

Limit test for iron based upon the chemical reaction of iron in ammonical solution in presence of iron free citric acid with thioglycollic acid, it forms ferrous thioglycolate complex.

Proposition 2: Colouration

A purple colour is formed due to formation of ferrous thioglycolate complex. The original state of iron is unimportant as thioglycollic acid reduces ferric (Fe^{3+}) to ferrous (Fe^{2+}). This test is very sensitive making use of 20 % citric acid, which forms, complex with other metal ion, eliminates interference of other metal ions.

Note:-(purple colour is developed only in alkaline medium, so ammonia solution is used, but ammonia solution forms precipitate with iron, therefore citric acid is added to form ammonium citrate buffer which stabilizes ferrous thioglycolate complex).

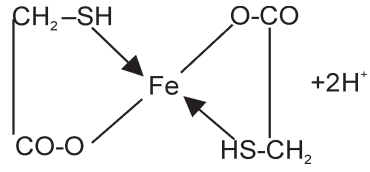
Proposition 3: Comparison with standard

The colour produced by sample under test is compared with the coloured produced by the standard solution.

Chemical Reactions:

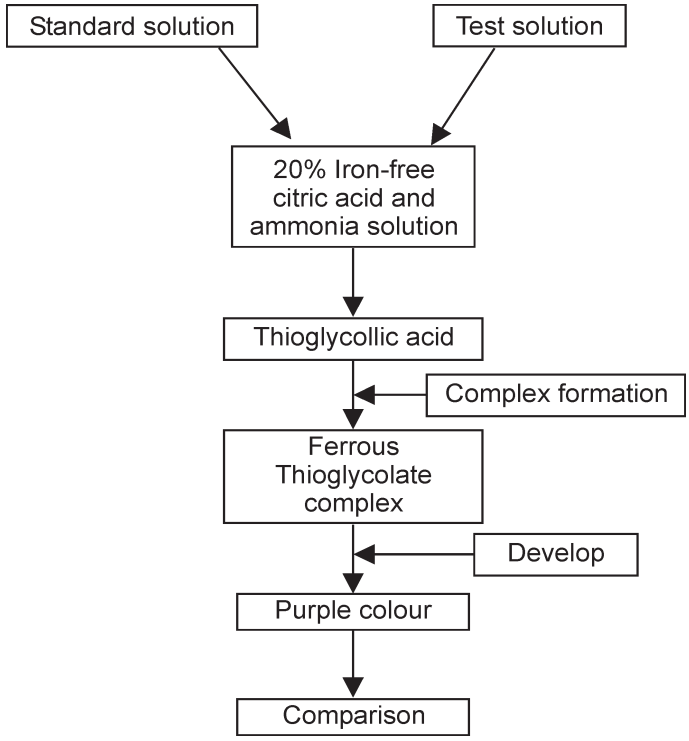
1.2 $Fe^{+++} + 2\ CH_2–SH\text{-}COOH \rightarrow 2\ Fe^{++} + HCOO\text{-}CH_2\text{-}S\text{-}S\text{-}CH_2\text{-}COOH + 2H^+$

Ferric Thioglycollic acid Ferrous

2. $Fe^{++} + 2\ CH_2SH\text{-}COOH$ —(Dil.ammonia, Citric acid)→ 

Ferrous Thioglycollic acid (Ferrous thioglycolate complex)

General concept structure:



4.0 Learning Objectives :-

4.1 Intellectual Skills:

1. To identify impurities in the given sample.

4.2 Motor Skills:

1. To observe the colour intensity in test solution and standard solution.
2. To compare the colour intensity of test solution with standard solution.

5.0 Apparatus :-

5.1 Materials:

A pair of Nesslers cylinder, wooden stands, Glass rod, Pipette (1 ml, 2 ml and 10ml), Measuring cylinder (100 ml), White tile, Glass beaker, analytical weighing balance and weight box.

5.2 Chemicals:

Standard solution of iron (20 ppm Fe), 20% w/v iron free citric acid, thioglycollic acid, zinc sulphate, sodium chloride, magnesium sulphate, iron free ammonia and distilled water.

6.0 Stepwise Procedure:

Standard solution	Test solution for zinc sulphate	Test solution for sodium chloride	Test solution for magnesium sulphate
1. Take 2.0 ml of iron standard solution (20 ppm Fe) in labeled Nesslers cylinder (S). 2. Add 2 ml of a 20% w/v solution of iron-free citric acid. 3. And add 0.1 ml of thioglycollic acid, mix well, make alkaline with iron free ammonia solution. 4. Dilute to 50 ml with water and allow to stand for 5 minutes.	1. Weigh accurately 2.5 g of zinc sulphate and dissolve in sufficient carbon dioxide free water to produce 50 ml In a beaker. Take 2.0 ml of solution and diluted to 10 ml with water in a labeled Nesslers cylinder (T). 2. Add 2 ml of a 20% w/v solution of iron-free citric acid. 3. And add 0.1 ml of thioglycollic acid, mix well and make alkaline with iron free ammonia solution. 4. Dilute to 50 ml with water and allow to stand for 5 min. 5. View the colour intensity against white background and compare with that of standard.	1. Weigh accurately 2g of sodium chloride and dissolve in 20 ml of water in a labeled Nesslers cylinder (T). 2. Add 2 ml of a 20% w/v solution of iron-free citric acid. 3. And add 0.1 ml of thioglycollic acid, mix well and make alkaline with iron-free ammonia solution. 4. Dilute to 50 ml with water and allow to stand for 5 min. 5. View the colour intensity against white background and compare with that of standard.	1. Weigh accurately 5 g of magnesium sulphate and dissolve in sufficient carbon dioxide-free water to 50 ml in a beaker. Take 2 ml of solution in a labeled Nesslers cylinder (T). 2. Add 2 ml of a 20% w/v solution of iron-free citric acid. 3. And add 0.1 ml of thioglycollic acid, mix well and make alkaline with iron free ammonia solution. 4. Dilute to 50 ml with water and allow to stand for 5 min. 5. View the colour intensity against white background and compare with that of standard.

7.0 Observations:

1. The colour intensity produced in zinc sulphate test solution is..... (less/same/more) than the colour intensity produced in standard solution.
2. The colour intensity produced in sodium chloride test solution is..... (less/same/more) than the colour intensity produced in standard solution.
3. The colour intensity produced in magnesium sulphate test solution is .....(less/same/more) than the colour intensity produced in standard solution.

8.0 Results:

1. The given sample of zinc sulphate..... (complies/ does not comply) the limit test for iron as per the Indian Pharmacopoeia.
2. The given samples of sodium chloride..... (complies/ does not comply) the limit test for iron as per the Indian Pharmacopoeia.
3. The given sample of magnesium sulphate..... (complies/ does not comply) the limit test for iron as per the Indian Pharmacopoeia.

9.0 Questions:

(Answer the following questions, Q.... Q.....Q....and Q....., Question Number to be allotted by the teacher)

1. Why limit test for iron is performed?
2. What is the principle of limit test for iron.
3. Why ammonia solution and citric acid solution are used in iron limit test?
4. Why deep reddish purple colour is developed in iron limit test?
5. Write chemical reaction involved in 'limit test for iron'.
6. Write the procedure for preparation of standard solution of iron.
7. Write the procedure for preparation of zinc sulphate test solution.
8. Write the procedure for preparation of sodium chloride test solution.
9. Write the procedure for preparation of magnesium sulphate test solution.
10. How to observe the colour intensity?
11. Explain the role of thioglycollic acid in iron limit test.

10.0 Reference:

Indian pharmacopoeia 1996

(Space for Answers)

(Space for Answers)

Experiment No. 6

1.0 Title :

Limit test for heavy metals

(To perform and report limit test for heavy metals on the given samples as per Indian pharmacopoeia).

1. Sodium chloride
2. Potassium iodide
3. Dextrose.

2.0 Prior Concepts :-

Sources of impurities, colloidal state, metal impurities.

3.0 New Concepts :-

Propositions: Principle

Proposition 1: Chemical Interaction

Limit test for heavy metal is based upon the reaction between hydrogen sulphide and certain heavy metals such as lead, iron, copper, nickel, cobalt and bismuth resulting in the formation of sulphides of the respective metals in presence of dilute acetic acid.

Proposition 2: Colouration

The sulphides so formed are distributed in a colloidal state and produce brownish coloured solution.

Proposition 3: Comparison of sample with standard

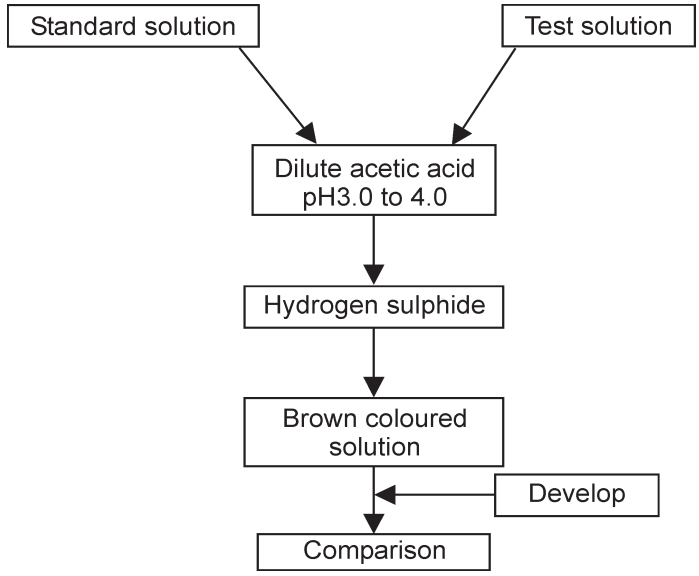
The intensity of colour produced in test is compared with intensity of colour produced in standard solution.

Chemical Reaction:

$$M + H_2S \xrightarrow[pH\ 3.0\ to\ 4.0]{dilute\ acetic\ acid} MS\downarrow + 2H^+$$

Metal Hydrogen sulphide Sulphide of metal

General concept structure:



4.0 Learning Objectives :-

4.1 Intellectual Skills:

1. To identify impurities in the given sample.

4.2 Motor Skills:

1. To observe the colour intensity in the test and standard solution.
2. To compare the colour intensity of test and standard solutions

5.0 Apparatus :-

5.1 Materials:

A pair of Nesslers cylinder, Glass rod, Pipette (10 ml), Measuring cylinder (100 ml), White tile, glass beaker, analytical weighing balance and weight box.

5.2 Chemicals:

Lead standard solution (20 ppm Pb), dilute acetic acid, dilute ammonia solution, freshly prepared hydrogen sulphides solution, distilled water, sodium chloride, potassium iodide and dextrose.

6.0 Stepwise Procedure: Method A (I.P. 1996)

Standard solution	Test solution of sodium chloride	Test solution of potassium iodide	Test solution of dextrose
1. In to a 50 ml labeled Nesslers cylinder (S) Pipette 1.0 ml of lead standard solution (20 ppm Pb). 2. Dilute with distilled water to 25 ml. 3. Adjust the pH with dilute acetic acid or dilute ammonia solution in between 3.0 to 4.0. 4. Dilute with distilled water about 35 ml and mix with glass rod. 5. Add 10 ml of freshly prepared hydrogen sulphide solution. 6. Mix and dilute to 50 ml with water. 7. Allow to stand for 5 minutes.	1. Accurately weigh 4 g of sodium chloride and add to labeled Nesslers cylinder (T). 2. Add 2 ml of dilute acetic acid and mix well, then add sufficient water to produce 25 ml. 3. Adjust the pH with dilute acetic acid or dilute ammonia solution in between 3.0 to 4.0. 4. Dilute with water to 35 ml and mix well. 5. Add 10 ml of freshly prepared hydrogen sulphide solution. 6. Mix and dilute to 50 ml with water. 7. Allow to stand for 5 minutes. 8. View downwards over a white surface and compare with that of standard.	1. Accurately weigh 2 g of potassium iodide and add to labeled Nesslers cylinder (T). 2. Add 2 ml of dilute acetic acid and mix well, then add sufficient water to produce 25 ml. 3. Adjust the pH with dilute acetic acid or dilute ammonia solution in between 3.0 to 4.0. 4. Dilute with water to 35 ml and mix well. 5. Add 10 ml of freshly prepared hydrogen sulphide solution. 6. Mix and dilute to 50 ml with water. 7. Allow to stand for 5 minutes. 8. View downwards over a white surface and compare with that of standard.	1. Accurately weigh 4 g of dextrose and dissolve in 10 ml water in a labeled Nesslers cylinder (T). 2. Add 2 ml of dilute acetic acid and mix well, then add sufficient water to produce 25 ml. 3. Adjust the pH with dilute acetic acid or dilute ammonia solution in between 3.0 to 4.0. 4. Dilute with water to 35 ml and mix well. 5. Add 10 ml of freshly prepared hydrogen sulphide solution. 6. Mix and dilute to 50 ml with water. 7. Allow to stand for 5 minutes. 8. View downwards over a white surface and compare with standard.

7.0 Observations:

1. The colour intensity produced in sodium chloride test solution is..... (less/same/more) than the colour intensity produced in standard solution.
2. The colour intensity produced in potassium iodide test solution is..... (less/same/more) than the colour intensity produced in standard solution.
3. The colour intensity produced in dextrose test solution is..... (less/same/more) than the colour intensity produced in standard solution.

8.0 Results:

1. The given sample of sodium chloride.....(complies/does not comply) the limit for heavy metal as per the Indian pharmacopoeia.
2. The given sample of potassium iodide.....(complies/does not comply) the limit for heavy metal as per the Indian pharmacopoeia.
3. The given sample of dextrose.....(complies/does not comply) the limit for heavy metal as per the Indian pharmacopoeia.

9.0 Questions:

(Answer the following questions, Q.... Q.....Q....and Q....., Question Number to be allotted by the teacher)

1. Why limit test for heavy metals is performed?
2. Write the procedure for preparation of standard solution for limit test for heavy metals.
3. Write the principle involved in the limit test for heavy metals.
4. Write the procedure for preparation of test solution of sodium chloride.
5. Write the procedure for preparation of test solution of potassium iodide.
6. Write the procedure for preparation of test solution of dextrose.
7. State reasons for uses of following.
   i. Dilute acetic acid
   ii. Dilute ammonia solution
   iii. Freshly prepared solution of hydrogen sulphides.
8. Is it possible to replace hydrogen sulphides solution by any other means? Justify with example.

10.0 Reference:

Indian pharmacopoeia 1996

(Space for Answers)

(Space for Answers)

Experiment No. 7

1.0 Title :

Limit test for arsenic.

(To perform and report limit test for arsenic on the given sample of sodium acetate as per Indian Pharmacopoeia).

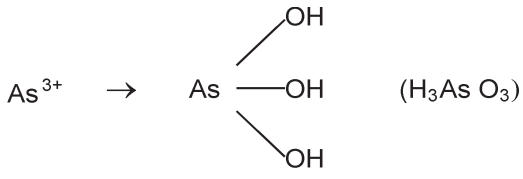
2.0 Prior Concepts :-

Sources of arsenic impurities, parts per millions.

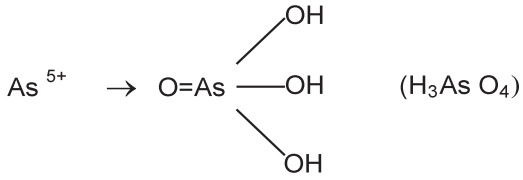
3.0 New Concepts: Principle

Proposition 1:

Limit test for arsenic is based on semiquantitative determination of arsenic impurities in the test sample of drug. The sample is dissolved in stannated acid, which converts the arsenic impurities to arsenious acid or arsenic acid depending upon valency state of arsenic impurity present in the test sample.



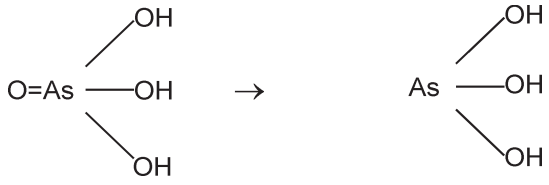
Trivalent arsenic — Arsenious acid (trivalent)



Pentavalent arsenic — Arsenic acid (pentavalent)

Proposition 2:

When acidic solution of sample treated is with reducing agent (stannous chloride) converts pentavalent arsenic acid into the trivalent arsenious acid.



Proposition 3:

$Zn^{+2} + 2\ SnHCl_3 \rightarrow ZnCl_2 + 2\ SnCl_2 + 2\ [H]$

Proposition 4:

The arsenious acid is then converted into gaseous arsenious hydride
(arsine gas) with the help of nascent hydrogen (which is produced by zinc and hydrochloric acid.

$$H_3AsO_3 + 3H_2 \rightarrow \underset{\text{Arsine gas}}{AsH_3} + 3\ H_2O$$

Proposition 5:

Arsine gas is carried through the tube by the steam of hydrogen and out through the mercuric chloride paper. A reaction occurs between arsine and mercuric chloride, which produces yellow colour stain.

$$2AsH_3 + HgCl_2 \rightarrow \begin{matrix} AsH_2 \\ Hg \\ AsH_2 \end{matrix} + 2HCl$$

(Yellow in colour)

Proposition 6:

The stain produced by test sample compared with that of standard arsenic solution.
The limit of arsenic is expressed as part per millions.

Note:

1. All solutions used in the test should be free from arsenic impurities and denoted as AsT.
2. The presences of small quantity of stannous chloride in the hydrochloric acid ensure the rapid reaction between the acid and the zinc and steady evolution of H_2 gas.
3. Lead acetate cotton plug prevent the formation of black stain to mercuric chloride paper produced by sulphide impurities which are present in zinc.
4. potassium iodide is converted to hydriodic acid in presence hydrochloric acid which reduces unreacted pentavalent arsenic to trivalent arsenic.

4.0 Learning Objectives:

4.1 Intellectual Skills:

1. To identify the impurity in the given sample.

4.2 Motor Skills:

1. To observe the stain produced by test sample and standard.
2. To compare the stain produced by test sample with standard.

5.0 Apparatus :-

5.1 Materials:

Apparatus for limit test of arsenic, cotton wool and mercuric chloride paper.

(Note: Student shall refer the I.P. for specification of arsenic limit test apparatus)

5.2 Chemicals:

Arsenic standard solution (10 ppm As), stannated hydrochloric acid (AsT), lead acetate, zinc (AsT) and 1 M potassium iodide.

6.0 Diagram :-

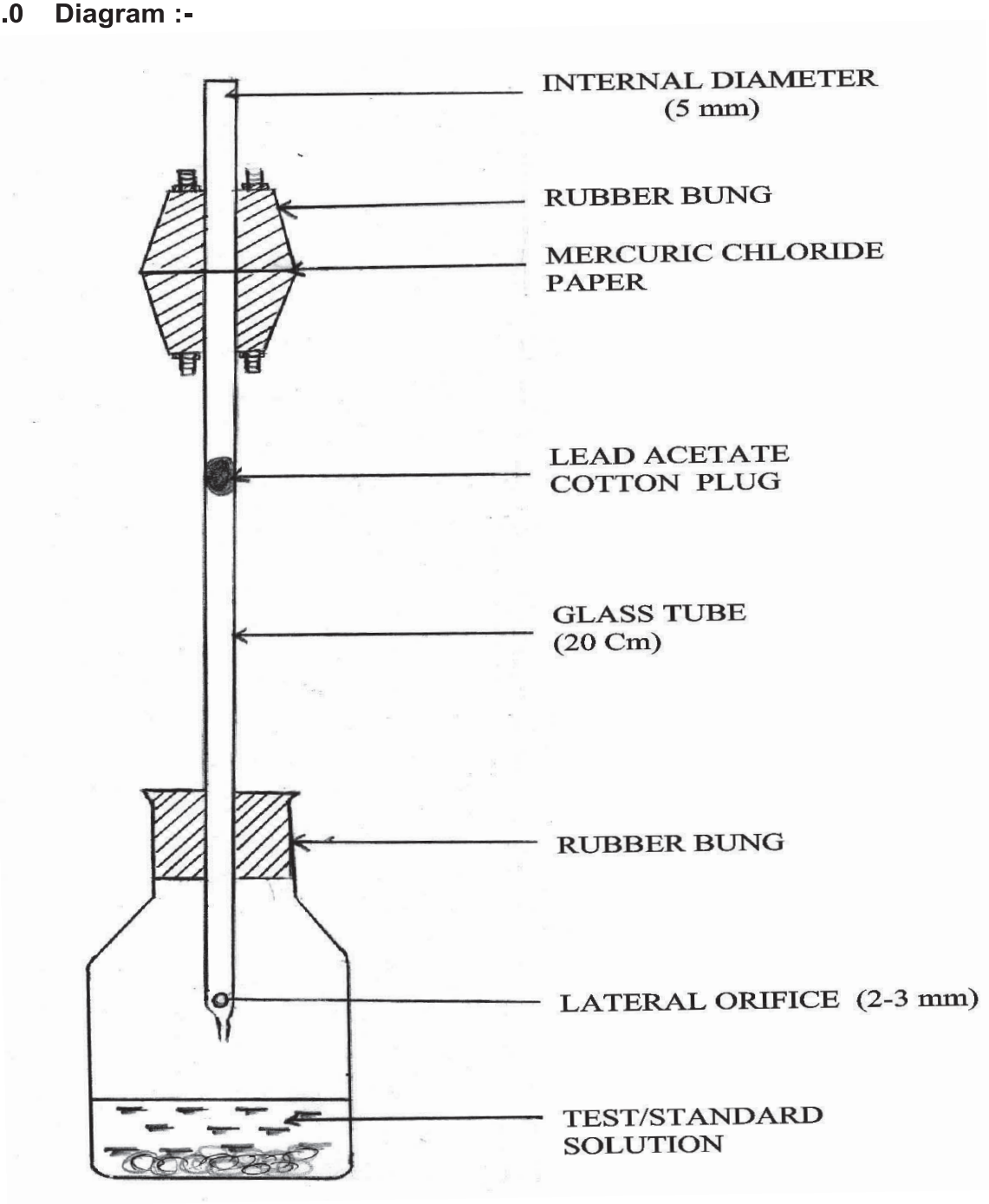


FIG : APPARATUS FOR ARSENIC LIMIT TEST

7.0 Stepwise procedure:

The glass tube is lightly packed with cotton wool (previously moistened with lead acetate solution). A piece of mercuric chloride paper is placed on top of the lower rubber bung and other rubber bung is placed in such a manner that the boring of the two rubber bung meets to from true tube.

Standard solution	Test solution of sodium acetate
1. Take 1 ml of arsenic standard solution (10 ppm As). 2. Dilute to 50 ml with water in the glass bottle/flask. 3. Add 5 ml of 1M potassium iodide. 4. And add 10 g zinc (AsT), mix it well. 5. Immediately assemble the apparatus and immerse the bottle/flask in a water bath at a temperature such that a uniform evolution of gas is maintained. 6. Allow the reaction to take place for 40 minutes. **Note: -** AsT = Tested for arsenic.	1. Accurately weigh 5 g of sodium acetate and dissolve in 50 ml water in the glass bottle/flask. 2. Add 15 ml of stannated hydrochloric acid (AsT). 3. To this add 5 ml of 1 M potassium iodide. 4. Add 10 g of zinc AsT. 5. Immediately assemble the apparatus and immerse the bottle/flask in a water bath at a temperature such that a uniform evolution of gas is maintained. 6. Allow the reaction to take place for 40 minutes, after 40 minutes any stain produced by test sample on mercuric chloride paper is compared with that of standard.

8.0 Observations :

The stain produced in sodium acetate test sample is.....(less/same/ more) than the stain produced in standard.

9.0 Result:

The given sample of sodium acetate.....(complies/does not comply) the limit test for arsenic as per the Indian Pharmacopoeia.

10.0 Questions:

(Answer the following questions, Q.… Q…..Q.…and Q.…., Question Number to be allotted by the teacher)

1. Write the role of stannous chloride in arsenic limit test.
2. How nascent hydrogen is produce in the limit test of arsenic.
3. What is the role of zinc and hydrochloric acid in limit test of arsenic?
4. How you identify the arsenic impurities present in given sample.

5. Write the principle involved in arsenic limit test.
6. Write the role of mercuric chloride paper.
7. Name the apparatus used for arsenic limit test.
8. Write the procedure for preparation of standard solution.
9. Write the procedure for preparation of sodium acetate test solution.
10. In which forms arsenic impurities present in drugs.
11. Write the steps involved in formation of arsine gas.
12. Draw the well-labeled diagram of arsenic limit test apparatus.
13. Why potassium iodide is used in arsenic limit test.
14. Why lead acetate soaked cotton plug is used in arsenic limit test.

11.0 Reference:

Indian pharmacopoeia 1996

(Space for Answers)

(Space for Answers)

Experiment No. 8

1.0 Title :

Assay of sodium bi-carbonate.

(To determine % w/w of $NaHCO_3$ in a given sample of sodium bicarbonate)

2.0 Prior Concepts :-

Acids, bases, Titration, Neutralization, Normality, Indicator and Arrhenius acid base theory.

3.0 New Concepts :-

Proposition 1:

This assay is based upon acid base reaction in which sodium bicarbonate act as week base and hydrochloric acid as strong acid.

According to Arrhenius theory, acid on dissociation gives hydrogen ion (H+) i.e. proton and base gives hydroxyl ion (OH-). Proton and hydroxyl ion on interaction, produces water. Therefore, it is also called as neutralization reaction.

It is expressed by following equations,

1. $HCl \longleftrightarrow H^+ + Cl^-$
2. $NaOH \longleftrightarrow Na^+ + OH^-$
3. $H^+ + OH^- \longrightarrow H_2O$

Proposition 2:

In this assay sodium bicarbonate is titrated against 1 M hydrochloric acid by using methyl orange as an indicator. At the end point, the colour changes from colourless to pink.

Proposition 3:

Factors:

I. Factor for standardization:

Each ml of 1 M Hydrochloric acid ≡ 0.05299 g of Na_2CO_3

II. Factor for Assay:

Each ml of 1 M Hydrochloric acid ≡ 0.08401 g of $NaHCO_3$

Standards:

Sodium bicarbonate contains not less than 99.0 percent and not more than101.0 percent of $NaHCO_3$.

Chemical Reactions:

I. Chemical Reaction for standardization:

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

II. Chemical Reaction for Assay:

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

4.0 Learning Objectives :-

4.1 Intellectual Skills:

1. To understand the concept of assay.
2. To identify titrant and titrate in titration.
3. To understand the concept of factor calculation.
4. To understand the concept of percentage purity.

4.2 Motor Skills:

1. To observe the correct meniscus of solution in burette.
2. To observe the formation of carbon dioxide in the reaction.
3. To observe the colour change at the end point of titration.

5.0 Apparatus :-

5.1 Materials:

Burette (50ml), Pipette (10 ml & 25 ml), Beaker (100 ml), conical flask (250 ml), analytical weighing balance and weight box.

5.2 Chemicals:

Sodium bicarbonate, sodium carbonate, hydrochloric acid (approx.1M), methyl orange and methyl red.

6.0 Stepwise procedure:

I. Standardization of Hydrochloric acid.

1. Weigh accurately 1.5 g of anhydrous sodium carbonate (previously heated at about 270° C for 1 hour in an oven)
2. Dissolve in 100 ml of water in a 250 ml conical flask.
3. Add 0.1 ml of methyl red solution as indicator.
4. Add hydrochloric acid slowly from a burette, with constant stirring until solution becomes faintly pink.
5. Heat the solution to boiling, cool and continue the titration until the faint pink colour is no longer affected by continued boiling.
6. Heat again to boiling and titrate further as necessary
7. Repeat the procedure for three times and calculate the molarity of hydrochloric acid.

II. Assay of Sodium bicarbonate:

1. Weigh accurately 1.5 g of sodium bicarbonate.
2. And dissolve in 50 ml of carbon dioxide-free water in a 250 ml conical flask.
3. Add 0.2 ml of methyl orange solution as indicator.
4. Titrate the solution with standardized hydrochloric acid (apporx.1M), until the solution become pink.
5. Repeat the procedure for three times and calculate the percentage purity of sodium bicarbonate.

7.0 Observations:

I. Standardization of Hydrochloric acid.

1. **Content of Conical Flask:-** 1.5 g of sodium carbonate + 100 ml of water + 0.1 ml methyl red.
2. **Solution in burette:-** Given hydrochloric acid.
3. **Indicator:-** Methyl red.
4. **Endpoint:-** Colourless to faint pink.

Observation table:

Sr.No.	Burette reading (ml)			Mean burette Reading (ml)
	Initial	Final	Difference	
1.				
2.				
3.				

II. Assay of Sodium bicarbonate:

1. **Weight if sample: 1.5 g**
2. **Solution in Conical Flask:-** 1.5 g of sodium bicarbonate + 50 ml of water + 0.2 ml methyl orange.
3. **Solution in burette:-** Standardized hydrochloric acid (approx.1M)
4. **Indicator:-** Methyl orange.
5. **Endpoint:-** Colourless to pink.

Observation table:

Sr.No.	Burette reading (ml)			Mean burette Reading (Ml)
	Initial	Final	Difference	
1.				
2.				
3.				

8.0 Calculations:

I. Factor calculation:

From the reaction,

$HCl \equiv NaHCO_3$

$36.46 \equiv 84.01$

1000 ml of 1 M Hydrochloric acid $\equiv$ 84.01 g of $NaHCO_3$

$\therefore$ Each ml of 1 M Hydrochloric acid $\equiv$ 0.08401 g of $NaHCO_3$

II. Standardization calculation:

$$\text{Molarity of hydrochloric acid} = \frac{\text{Wt of sodium carbonate}}{\text{Burette reading X } 0.05299}$$

= .....

= .....

III. Percentage purity calculation:

1 ml of 1M hydrochloric acid $\equiv$ 0.08401 g of $NaHCO_3$

$$\text{1 ml of 'm' M Hydrochloric acid} = \frac{0.08401 \text{X m g of } NaHCO_3}{M}$$

$$\therefore \text{ 'x' ml of 'm' M hydrochloric acid} = \frac{0.08401 \text{ X m X x g of } NaHCO_3}{M}$$

= .....

= .....

$$\therefore \text{ 'w' g of sample contains} = \frac{0.08401 \text{ X m X x g of } NaHCO_3}{M}$$

= .....

= ..... g of $NaHCO_3$

$$\therefore \text{ 100 g of sample contains} = \frac{0.08401 \text{ X m X x X 100 g of } NaHCO_3}{M \text{ X w}}$$

= .....

= .....%$NaHCO_3$

(Where, x = Burette reading of assay, w = weight of sodium bicarbonate, and m = Calculated molarity and M = Factor molarity)

9.0 Results:

1. Molarity of hydrochloric acid is .....M.
2. The given sample of sodium bicarbonate contains. ……..... % w/w of $NaHCO_3$.

10.0 Questions:

(Answer the following questions, Q.… Q…..Q.…and Q….., Question Number to be allotted by the teacher)

1. Write the principle of sodium bicarbonate assay.
2. What is neutralization reaction?
2. What is the procedure for standardization of hydrochloric acid?
3. Write any two neutralization reactions.
4. Write the formula for percentage purity calculation.
5. Write the formula for normality calculation.
6. Write factor calculation for assay of sodium bicarbonate.
7. Write two acid-base theories with examples.
8. Enlist five acids and bases used in laboratory.
9. Write two pharmaceutical uses of sodium bicarbonate.
10. Find out two market preparations, which contain sodium bicarbonate.
11. Write the I.P.standards for sodium bicarbonate.

11.0 Reference:

Indian pharmacopoeia 1996.

(Space for Answers)

(Space for Answers)

Experiment No. 9

1.0 Title :

Assay of Boric Acid.

(To determine the % w/w of H_3BO_3 in a given sample of boric acid)

2.0 Prior Concepts :-

Acids, bases, Titration, Neutralization, Normality, Primary standard, Secondary standard, Indicator and Arrhenius acid base theory.

3.0 New Concepts: Acid-Base Titration

Proposition 1:

This assay is based on acid-base type of titration in which boric acid, a very weak acid and is to be titrated against strong alkali like sodium hydroxide. It does not give sharp end point, so glycerin is added to form glyceroboric acid complex, which acts as strong monobasic acid and is strong enough to titrate against standard solution of sodium hydroxide using phenolphthalein as indicator.

(Note:- The acidity of boric acid can also be increased by the other polyhydric compounds like mannitol)

Proposition 2:

Factor: -

I. Factor for Standardization:

1. Each ml of 1M sodium hydroxide ≡ 0.02042 g of $C_8H_5KO_4$

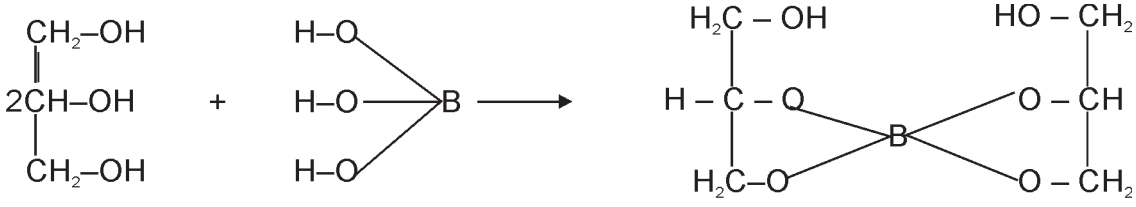
II. Factor for Assay:

2. Each mi of 1M sodium hydroxide ≡ 0.06183 g of H_3BO_3

Standards: -

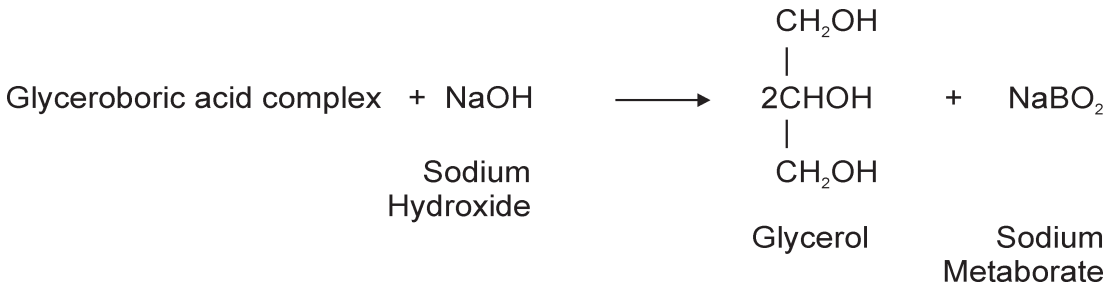
Boric acid contains not less than 99.5 Percent and not more than 100.5 percent of H3BO3, calculated with reference to the dried substance.

Chemical reactions for Assay: -

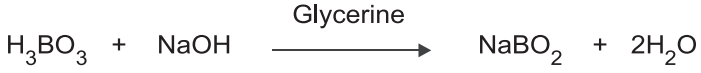


Glycerol Boric acid Glyceroboric acid complex

$+ H_3O^+ + 2H_2O$



Net reaction:



4.0 Learning Objectives :-

4.1 Intellectual Skills:

1. To understand the concept of assay.
2. To identify titrant and titrate in titration.
3. To understand the concept of factor calculation.
4. To understand the concept of percentage purity.

4.2 Motor Skills:

1. To observe the correct meniscus of solution in burette.
2. To observe the colour change at the end point of titration.

5.0 Apparatus :-

5.1 Material:

Burette (50ml), pipette (10 ml & 25 ml), beaker (100 ml), conical flask (250 ml), measuring cylinder (100 ml), glass funnel (small) etc.

5.2 Chemicals:

Boric acid, Potassium hydrogen phthalate, Sodium hydroxide, (apporx.1M), glycerin, phenolphthalein.

6.0 Stepwise procedure:

I. Standardization of sodium hydroxide.

1. Weigh accurately 5 g of potassium hydrogen phthalate.
2. Dissolve it in 75 ml of carbon dioxide free water.
3. Add 0.1 ml of phenolphthalein solution as indicator.
4. Fill the burette with given sodium hydroxide solution.
5. Remove the air bubble and adjust the zero level.
6. Titrate the solution in conical flask by running the solution from burette till the colour changes from colourless to pink.
7. Report the reading by repeating it for three times and calculate the molarity of sodium hydroxide.

II. Assay of Boric acid:

1. Weigh accurately 2 g of given sample of boric acid in conical flask.
2. Add mixture of 50 ml water and 100 ml glycerin, previously neutralized to phenolphthalein solution and dissolve the sample completely.
3. To this mixture add few drop of phenolphthalein indicator.
4. Add the standardized sodium hydroxide (aprox.1M) from burette.
5. Until colour change from colourless to pink.
6. Report the burette reading three times and calculate the percentage purity by using factor.

7.0 Observations:

I. Standardization of sodium hydroxide.

1. **Content of conical flask:-** 5 g of potassium hydrogen phthalate + 75 ml of water and phenolphthalein.
2. **Solution in burette: -**Given sodium hydroxide solution.
3. **Indicator: -** phenolphthalein solution.
4. **End point: -**Colourless to pink.

Observation table:

Sr.No.	Burette reading (ml)			Mean burette Reading (ml)
	Initial	Final	Difference	
1				
2				
3				

II. Assay of Boric acid:

1. Weight of sample: 2g
2. Content of conical flask: - 2g boric acid+ 50 ml water + 100ml glycerin and phenolphthalein.
3. Solution in burette: - Standardized sodium hydroxide solution (approx 1M).
4. Indicator: - phenolphthalein solution.
5. End point: -Colourless to pink.

Observation table: -

Sr.No.	Burette reading (ml)			Mean burette Reading (ml)
	Initial	Final	Difference	
1				
2				
3				

8.0 Calculations:

I. Factor Calculation:

NaOH	≡	H_3BO_3
40	≡	61.83
1000 ml of 1 M sodium hydroxide	≡	61.83 g of H_3BO_3
Each ml of 1 M sodium hydroxide	≡	0.06183 g of H_3BO_3

II. Standardization calculation:

$$\text{Molarity of sodium hydroxide} = \frac{\text{Wt of potassium hydrogen phthalate}}{\text{Burette reading x 0.02042}}$$

= …….....

= …….....M

III. Percentage purity calculation:

Each ml of 1 M sodium hydroxide ≡ 0.06183 g of H_3BO_3

1 ml of 'm' M sodium hydroxide ≡ 0.06183 g of H_3BO_3

∴ 'x' ml of 'm' M sodium hydroxide = $\frac{0.06183 \times m \times x}{M}$ g of H_3BO_3

= …….

= …… ……..

∴ 'w' g of sample contains = $\frac{0.06183 \times m \times x}{M}$ g of H_3BO_3

= …….

= …… g of H_3BO_3

∴ 100 g of sample contains = $\frac{0.06183 \times m \times x \times 100}{M \times w}$ g of H_3BO_3

= …….

= ……% H_3BO_3

(Where, x = Burette reading of assay, w = weight of boric acid, m = calculated molarity and M = factor Molarity)

9.0 Results:

1. Molarity of sodium hydroxide is.....M.
2. The given sample of boric acid contains …….. % w/w of H_3BO_3

10.0 Questions:

(Answer the following questions, Q.… Q…..Q.…and Q….., Question Number to be allotted by the teacher)

1. Write the principle of boric acid assay.
2. Write the procedure for standardization of sodium hydroxide.
3. Write the formula for percentage purity calculation.
4. Write the factor calculation of boric acid.
5. Write the uses of boric acid.
6. Write the assay procedure for boric acid.
7. State the use of glycerine in boric acid assay.
8. Write the chemical reaction involved in boric acid assay.
9. Write the standards of boric acid.
10. Write the factor of boric acid assay.

11.0 Reference:

Indian pharmacopoeia 1996

(Space for Answers)

(Space for Answers)

Experiment No. 10

1.0 Title :

Assay of zinc oxide.

(To determine % w/w of ZnO in a given sample of zinc oxide)

2.0 Prior Concepts:

Acids, bases, Titration, Neutralization, Normality, Indicator and Arrhenius acid base theory.

3.0 New Concepts:

Proposition 1:

This assay is based upon acid base type of titration. Zinc oxide reacts with acid very slowly, so the back titration is useful for the assay of zinc oxide. In this assay the zinc oxide is added to definite amount of a sulphuric acid (capable of reacting with the substance under test), which is taken in excess and is titrated with standard solution of sodium hydroxide. Sulphuric acid solution, which is excess, is back titrated. From the volume of sulphuric acid consumed, percentage of zinc oxide can be calculated. (**Note:** Ammonium chloride is added to prevent the precipitation of zinc hydroxide, which interferes with end point detection.)

Proposition 2 :

Factors:

I. Factor for standardization:

Each ml of 1M sodium hydroxide ≡ 0.02042 g of $C_8H_5KO_4$

II. Factor for Assay:

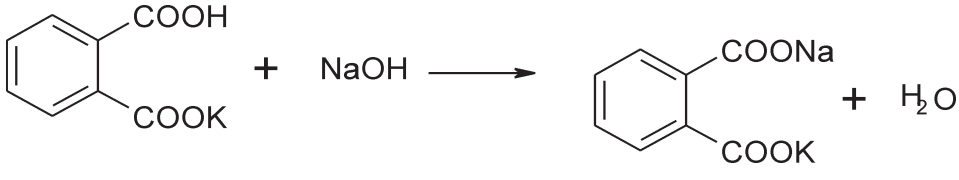
Each ml of 1 N Sulphuric acid ≡ 0.04069 g of ZnO

Standards:

Zinc oxide contains not less than 99.0 percent not more than 100.5 percent of ZnO, calculated with reference to the ignited substance.

Chemical Reactions:

I. Chemical Reaction for standardization:



Potassium Hydrogen Phthalate Sodium Potassium Phthalate

II. Chemical Reaction for Assay:

1. $ZnO + H_2SO_4 \longrightarrow ZnSO_4 + H_2O$

2. $H_2SO_4 + 2NaOH \longrightarrow Na_2SO_4 + 2H_2O$

4.0 Learning Objectives :-

4.1 Intellectual Skills:

1. To understand the concept of assay.
2. To identify titrant and titrate in titration.
3. To understand the concept of factor calculation.
4. To understand the concept of percentage purity.
5. To understand the concept of back titration.
6. To understand the concept of blank titration.

4.2 Motor Skills:

1. To observe the correct meniscus of solution in burette.
2. To observe the colour change at the end point of titration.

5.0 Apparatus :-

5.1 Materials:

Burette (50ml), Pipette (10 ml & 25 ml), Beaker (100 ml), conical flask (250 ml, analytical weighing balance and weight box.

5.2 Chemicals:

Zinc oxide, ammonium chloride, sulphuric acid solution (1 N), sodium hydroxide Solution, potassium hydrogen phthalate, phenolphthalein and methyl orange.

6.0 Stepwise procedure:

I. Standardization of Sodium Hydroxide.

1. Weigh accurately 5 g of potassium hydrogen phthalate.
2. Dissolve it in 75 ml of carbon di oxide free water in conical flask.
3. Add 0.1 ml of phenolphthalein solution as indicator.
4. Fill the burette with given sodium hydroxide solution.
5. Remove the air bubble and adjust the zero level.
6. Titrate the solution in conical flask by running the solution from burette till the colour changes from colourless to pink.
7. Report the reading by repeating it for three times and calculate the normality of sodium hydroxide.

II. Assay of Zinc oxide:

1. Weigh accurately 1.5 g of zinc oxide.
2. Dissolve with 2.5 g of ammonium chloride in 50.0 ml of 1N sulphuric acid with the add of gentle heat, if necessary.
3. Add few drops of methyl orange solution.
4. Titrate the excess sulphuric acid with 1 N sodium hydroxide.

7.0 Observations:

I. Standardization of sodium hydroxide.

1. **Solution in conical flask: -** 5 g of potassium hydrogen phthalate + 75 ml of water and phenolphthalein.
2. **Solution in burette: -**Given sodium hydroxide solution.
3. **Indicator: -** phenolphthalein solution.
4. **End point: -**Colourless to pink.

Observation table:

Sr.No.	**Burette reading** (ml)			**Mean burette Reading** (ml)
	Initial	**Final**	**Difference**	
1.				
2.				
3.				

II. Assay of Zinc oxide:

a. Back titration:

1. **Weight of sample:** 1.5 g of zinc oxide
2. **Solution in Conical Flask: -** 1.5 g of zinc oxide +2.5 g of ammonium chloride + 50 ml of 1 N sulphuric solution.
3. **Solution in burette: -** Standardized sodium hydroxide (approx.1N)
4. **Indicator: -** Methyl orange.
5. **End point: -**Orange yellow to red.

Observation table:

Sr.No.	**Burette reading** (ml)			**Mean burette Reading** (ml)
	Initial	**Final**	**Difference**	
1.				
2.				
3.				

b. Blank titration:-

1. Solution in Conical Flask: - 2.5 g of ammonium chloride + 50 ml of 1 N sulphuric solution.
2. Solution in burette: - Standardized sodium hydroxide (approx.1N)
3. Indicator: - Methyl orange.
4. End point: -Orange yellow to red.

Observation table:

Sr.No.	Burette reading (ml)			Mean burette Reading (ml)
	Initial	Final	Difference	
1.				
2.				
3.				

8.0 Calculations:

I. Factor calculation:

From the reaction:

$$2H_2SO_4 \equiv ZnO$$

$$\equiv \frac{81.38}{2}$$

$$\equiv 40.69$$

1000 ml of 1 N sulphuric acid ≡ 40.69 g of ZnO

Each ml of 1 N sulphuric acid ≡ 0.04069 of ZnO

II. Standardization calculation:

$$\text{Normality of sodium hydroxide} = \frac{\text{Wt of potassium hydrogen phthalate}}{\text{Burette reading x 0.02042}}$$

= …….....

= ……N

III. Percentage purity calculation: -

(**Note:** 1. The excess volume of sulphuric acid added = 50 ml

2. The volume of sodium hydroxide reacted with unused sulphuric acid in flask.

3. The volume of sulphuric acid utilized by the sample is x ml = blank reading - back reading = ……. ml)

Each ml of 1 N Sulphuric acid ≡ 0.04069 of ZnO

If 1 ml of 'n' N Sulphuric acid = $\frac{0.04069 X n}{N}$ g of ZnO

∴ 'x' ml of 'n' N Sulphuric acid = $\frac{0.04069 \text{ X n X x}}{N}$ g of ZnO

= .....

= .....……

$$\therefore \text{ 'w' g of sample contains} = \frac{0.04069 \times n \times x \text{ g of ZnO}}{N}$$

= ……..

= …… g of ZnO

$$\therefore \text{ 100 g of sample contains} = \frac{0.04069 \times n \times x \times 100 \text{ g of ZnO}}{N \times W}$$

= ……

= ……g of ZnO

i.e., % purity of = ……. % w/w of ZnO

(Where, x = 50 ml - Burette reading of assay, w = weight of zinc oxide, and n = Calculated normality and N = Factor normality)

9.0 Results:

1. Normality of sodium hydroxide is .....N.
2. The given sample of zinc oxide contains. …….....% w/w of ZnO.

10.0 Questions:

(Answer the following questions, Q.... Q...Q....and Q.... Question Number to be allotted by the teacher)

1. Why zinc oxide is back titrated?
2. Write the principle involved in assay of zinc oxide.
3. Write the role of ammonium chloride in assay of the zinc oxide.
4. Write the chemical reactions involved in assay of zinc oxide.
5. Write the procedure for standardization of sodium hydroxide.
6. Write the procedure for assay of zinc oxide.
7. Write the factor calculation for assay of zinc oxide.
8. Write the standards for assay of zinc oxide.
9. Write the factor for assay of zinc oxide.

11.0 Reference:

Indian pharmacopoeia 1985.

(Space for Answers)

(Space for Answers)

Experiment No. 11

1.0 Title :

Assay of Ferrous Sulphate.

(To determine the % w/w of Fe SO_4. $7H_2O$ in a given sample of ferrous sulphate)

2.0 Prior Concepts:

Redox titration, Standardization, Primary standard, Secondary standard, Indicator.

3.0 New Concepts: Redox Titration

Proposition 1:

Oxidation is the loss of electron and reduction is the gain of electron. These two processes always occur simultaneously or they go hand in hand i.e. oxidation can never occur without reduction or vice versa. Such reactions are called oxidation-reduction reactions or simply redox reactions.

Proposition 2 :

When potassium permanganate is used as an oxidizing agent in the titration it is known as permanganate titration. These titrations are mainly conducted in acidic solution. The ability of $KMnO_4$ solution to oxidize is due to the conversion of the MnO4- (permanganate) ion to Mn2+ (Manganese) in acidic solution.

$$MnO_4^- \longrightarrow Mn^{+2},$$

$$MnO_4^- + 8H^+ + 5e^- \rightarrow Mn^2 + 4H_2O.$$

1. Solutions containing MnO_4^- ions are purple in colour.
2. Solutions of salts containing Mn^{2+} ions are colourless.

Therefore, a permanganate solution is decolourised when added to a solution of a reducing agent. The moment there is an excess $KMnO_4$, the solution becomes purple. Thus, the permanganate ion can act as a self-indicator, especially in acidic conditions.

Proposition 3:

Assay of ferrous sulphate depends upon oxidation-reduction type of titration where Fe^{2+} (ferrous ion), are readily oxidized by potassium permanganate in acidic solution (H_2SO_4) into Fe^{3+} (ferric ion). Thus ferrous sulphate acts as a reducing agent.

Proposition 4:

$$MnO_4^- + 4H^+ + 3e^- \rightarrow MnO_2 + 2H_2O.$$

Factor:

Each ml 0.1N of potassium permanganate ≡ 0.02780 g of $FeSO_4$, $7H_2O$

Standards: -

Ferrous sulphate contains not less than 98.0 percent and not more than 105.0 percent of $FeSO_4$, $7H_2O$.

Chemical Reactions:

I. Chemical reaction for standardization:

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

II. Chemical reactions for assay:

1. $2KMnO_4 + 3H_2SO_4 \rightarrow K_2SO_4 + 2MnSO_4 + 3H_2O + 5(O)$

2. $2FeSO_4 + H_2SO_4 + (O) \rightarrow Fe_2(SO_4)_3 + H_2O$

Net Reaction:

3. $2KMnO_4 + 8H_2SO_4 + 10FeSO_4 \rightarrow K_2SO_4 + 2MnSO_4 + 5Fe_2(SO_4)_3 + 8H_2O$

4. $Fe^{+++} + \bar{e} \xrightarrow[\text{oxidation}]{} Fe^{++}$

5. $Mno_4^- + 8H^+ + 5e^- \xrightarrow{\text{reduction}} Mn^{+2} + 4H_2O$

4.0 Learning Objectives :-

4.1 Intellectual Skills:

1. To understand the concept of assay.
2. To identify titrant and titrate in titration.
3. To understand the concept of factor calculation.
4. To understand the concept of percentage purity.

4.2 Motor Skills:

1. To observe the correct meniscus of solution in burette.
2. To observe the colour change at the end point of titration.

5.0 Apparatus :-

5.1 Materials:

Burette (50ml), pipette (10 ml & 25 ml), beaker (100 ml), conical flask (10 ml), measuring cylinder (50 ml), glass funnel (small) etc.

5.2 Chemicals:

Ferrous sulphate, potassium permanganate, primary standard oxalic acid (0.1N), dilute sulphuric acid and distilled water.

6.0 Stepwise procedure:

I. Standardization of potassium permanganate solution:

1. Take 10 ml of 0.1 N oxalic acid solutions in conical flask.
2. Add 10 ml dilute sulphuric acid.
3. Heat up to 70oC and titrate this heated solution against given solution of potassium permanganate.
4. Report the reading by repeating it for three times and calculate the normality of potassium permanganate.

II. Assay of Ferrous Sulphate:

1. Weigh accurately 1 g of sample
2. Dissolve in 20 ml distilled water in conical flask.
3. To this add 10 ml dilute sulphuric acid.
4. Titrate this solution against standardized solution of potassium permanganate.
5. Report the burette reading three times and calculate the percentage purity by using factor.

7.0 Observations:

I. Standardization of potassium permanganate solution:

1. **Content of conical flask: -** 10 ml oxalic acid +20 ml dilute sulphuric acid.
2. **Solution in burette: -** Given potassium permanganate.
3. **Indicator: -** potassium permanganate (Self indicator).
4. **End point: -** colourless to light pink.

Observation table:

Sr.No.	Burette reading (ml)			Mean burette Reading (ml)
	Initial	Final	Difference	
1				
2				
3				

II. Assay of Ferrous Sulphate:

1. Content of conical flask: - 1g sample + 20 ml distilled water +10 ml dilute sulphuric acid.
2. Solution in burette: - Standardized potassium permanganate(approx. 0.1 N)
3. Indicator: -Self indicator.
4. End point: - colourless to light pink.

Observation table:

Sr.No.	Burette reading (ml)			Mean burette Reading (ml)
	Initial	Final	Difference	
1				
2				
3				

8.0 Calculations:

I. Factor Calculation:

From the reaction:

$2KMnO_4 \equiv 10FeSO_4, 7H_2O \equiv 5(O)$

$2\ (158) \equiv 10\ (278) \equiv 5(16)$

$316 \equiv 278 \equiv 8$

1000 ml 1 N $KMnO_4$ solution $\equiv$ 278 g of $FeSO_4, 7H_2O$

Each ml of 0.1 N $KMnO_4$ solution $\equiv$ 0.0278 g of $FeSO_4, 7H_2O$

II. Standardization calculation:

Potassium permanganate = Oxalic acid

$$N_1 \times V_1 = N_2 \times V_2$$

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

N_1 = ……

N_1 = ……N

III. Percentage purity calculation:

Each ml of 0.1 N $KMnO_4$ solution $\equiv$ 0.0278 g of $FeSO_4, 7H_2O$

If 1ml of 0.1 N $KMnO_4$ solution $\equiv$ 0.0278 g of $FeSO_4, 7H_2O$

$\therefore$ 'x' ml of 'n' N potassium permanganate = $\frac{0.0278 \times n \times x}{N}$ g of $FeSO_4, 7H_2O$

= …….

= …….

'w' g of sample contains = $\frac{0.0278 \times n \times x}{N}$ g of $FeSO_4, 7H_2O$

= .....

= .....

$\therefore$ 100 g of sample contains = $\frac{0.0278 \text{ X n X x X } 100}{\text{NXw}}$ g of $FeSO_4, 7H_2O$

= .....

= .....

(Where, x = Burette reading, w = weight of ferrous sulphate, n = calculated normality, N = factor normality)

9.0 Results:

1. The normality of potassium permanganate solution is .....N.
2. The given sample of hydrated ferrous sulphate contains.....% w/w of $FeSO_4$,$7H_2O$.

10.0 Questions:

(Answer the following questions, Q.... Q.....Q....and Q....., Question Number to be allotted by the teacher)

1. Give properties of potassium permanganate, give their chemical reaction.
2. Which indicator is used in this titration?
3. State the meaning of oxidation-reduction type of titration.
4. How potassium permanganate acts as self-indicator?
5. State the meaning of Oxidation, Reduction.
6. Write the reactions involved in assay of ferrous sulphate.
7. Which primary standard solution is used to standardize potassium permanganate solution?
8. Write the procedure for preparation of 0.1N oxalic acid solution.
9. Write principle involve in assay of ferrous sulphate.
10. Write standard and factor of assay of ferrous sulphate.

11.0 Reference:

1. Indian pharmacopoeia 1985.
2. Indian pharmacopoeia 1996.

(Space for Answers)

(Space for Answers)

Experiment No. 12

1.0 Title :

Assay of Iodine.

(To determine % w/w of I in a given sample of iodine)

2.0 Prior Concepts:

Standardization, Iodimetric flask, Indicator,

3.0 New Concepts: Redox titration

Proposition 1:

Assay of iodine is based upon Iodimetry (Redox) type of titration in which standard solution of iodine is used. Iodine is slightly soluble in water it made soluble by adding potassium iodide, which forms polyiodides (KI_3), where free iodine acts as oxidizing agent, the solution is titrated against the standard solution of reducing agent, sodium thiosulphate using starch solution as an indicator. The end point is determined by colour change from blue to colourless.
In iodometry type of titration liberated iodine is used for titration.

Proposition 2 :

These are the titration, which involve the titration of free iodine (direct titration) with standard sodium thiosulphate; starch solution is used as an indicator to detect the end point of titration. Starch shows end point depending upon the structural changes. Starch is polysaccharide, which contains β-amylose and amylopectine. β-amylose, which is straight chain structure, changes to spiral form on absorption of iodine leading to bluish colouration while amylopectine which is branched chain structure, absorbs iodine to give insoluble complex.

(**Note: -** Starch solution is added near to the end point rather than at the initial stages of titration to prevent formation of insoluble complex of amylopectine with the iodine, which may interfere with the end point of titration. Higher temperature and presence of alkalis should be avoided in the titration to get correct / proper end point of the titration)

Proposition 3:

Factor:

I. Factor for Standardization:

Each ml of 0.1 M sodium thiosulphate ≡ 0.002784 g of $KBrO_3$

II. Factor for Assay:

Each ml of 0.1 M sodium thiosulphate ≡ 0.01269 g of I

Standards:

Iodine contains not less than 99.5 percent and not more than 100.5 percent of I.

Chemical reaction for assay:

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

Sodium Thiosulphate	Iodine		Sodium tetrathionate		Sodium iodide

4.0 Learning Objectives :-

4.1 Intellectual Skills:

1. To understand the concept of assay.
2. To identify titrant and titrate in titration.
3. To understand the concept of factor calculation.
4. To understand the concept of percentage purity.
5. To understand the difference between Iodimetry & Iodometry type of titration.

4.2 Motor Skills:

1. To observe the correct meniscus of solution in burette
2. To observe the colour change at the end point of titration.

5.0 Apparatus :-

5.1 Materials:

Iodine flask with stopper (250 ml), burette (50ml), pipette (10 ml, 25 ml), conical flask (250 ml), measuring cylinder (50 ml), beakers (100 ml)

5.2 Chemicals:

Potassium bromate, 2 M acetic acid, potassium iodide, iodine, sodium thiosulphate, starch solution and 2 M hydrochloric acid

6.0 Stepwise procedure:

I. Standardization of Sodium thiosulphate solution:

1. Weigh accurately 0.200 g of potassium bromate and dissolve in sufficient water to produce 250.0 ml in a 250 ml volumetric flask.
2. Take 50.0 ml of above solution and add 2 g of potassium iodide and 3 ml of 2M hydrochloric acid.
3. And titrate with the given sodium thiosulphate solution using starch solution as an indicator, added towards the end of the titration, until the blue colour is discharged.
4. Repeat the procedure for three times and calculate the molarity of sodium thiosulphate.

II. Assay of Iodine:

1. Weigh accurately about 0.2 g of given sample of iodine and transfer it into a iodine flask containing 1g of potassium iodide (KI), 2 ml of distilled water and 1 ml of 2 M acetic acid.
2. Dissolve the iodine by shaking and add 50 ml of distilled water, shake it well and keep side in dark for 15 minutes.
3. Titrate the solution against standardized sodium thiosulphate (aprox.0.1M) solution by using starch solution as an indicator, added towards the end of the titration, colour change from blue to colourless.
4. Repeat the procedure for three times and calculate the percentage purity by using the factor.

7.0 Observations:

I. Standardization of Sodium thiosulphate solution:

1. Content of iodine flask: - 50 ml solution of potassium bromate + 2 g of potassium iodide + 3 ml of 2 M hydrochloric acid.
2. Solution in burette: -Given Sodium thiosulphate solution.
3. Indicator: -Starch solution (Freshly prepared)
4. End point: - Blue colour to colourless.

Observation table:

Sr.No.	Burette reading (ml)			Mean burette Reading (ml)
	Initial	Final	Difference	
1				
2				
3				

II. Assay of Iodine

1. Content of iodine flask: - 0.2 g of iodine + 1 g of potassium iodide + 2 ml water + 1 ml of 2 M acetic acid + 50 ml water.
2. Solution in burette: - Standardized sodium thiosulphate solution (Aporx.0.1M).
3. Indicator: - Starch solution (freshly prepared).
4. End point: - Blue colour to colourless.

Observation table:

Sr.No.	Burette reading (ml)			Mean burette Reading (ml)
	Initial	Final	Difference	
1				
2				
3				

8.0 Calculations:

I. Factor Calculation:

From the above reaction,

$Na_2S_2O_3, 5H_2O \equiv I$

$248.17 \equiv 253.81$

1000 ml of 1 M sodium thiosulphate $\equiv$ 126.9 g of I

1ml of 1 M sodium thiosulphate $\equiv$ 0.1269 g of I

Each ml of 0.1 M sodium thiosulphate $\equiv$ 0.01269 g of I

II. Standardization calculation:

$$\text{Molarity of sodium thiosulphate} = \frac{\text{Wt of potassium bromate}}{\text{Burette reading x 0.002784}}$$

= .....

= .....M

III. Percentage purity calculation:

Each ml of 0.1 M sodium thiosulphate ≡ 0.01269 g of I

1 ml of 'm' M sodium thiosulphate ≡ $\frac{0.01269 \times m}{M}$ g of I

= .....

'x' ml of 'm' M sodium thiosulphate = $\frac{0.01269 \times m \times x}{M}$ g of I

= .....

= .....

∴ 'w' g of sample contains = $\frac{0.01269 \times m \times x}{M}$ g of I

= .....

= .....g of I

∴ 100 g of sample contains = $\frac{0.01269 \times m \times x \times 100}{M \times w}$ g of I

= .....

= .....% w/w of I

(Where, x = burette reading of assay, w = weight of iodine and m = calculated molarity and M = Factor molarity)

9.0 Results:

1. Molarity of sodium thiosulphate is .....M.
2. The given sample of Iodine contains .....% w/w of I.

10.0 Questions:

(Answer the following questions, Q.... Q...Q....and Q.... Question Number to be allotted by the teacher)

1. Differentiate between Iodometry & Iodimetry type of titration.
2. List out the different indicators uses in iodine titration.
3. Why starch shows blue colouration with iodine?
4. State the names of inorganic compounds, which are assayed by Iodimetry.

5. State the reason for use of potassium iodide in the assay of iodine.
6. State the reason for use of iodine flask for iodine assay.
7. Write the method of standardization of sodium thiosulphate solution.
8. Is it possible to find out percentage purity of iodine other than 'Redox' titration? Describe in detail.
9. Write the I.P. procedure for preparation of starch solution as indicator.

11.0 Reference:

Indian pharmacopoeia 1996.

(Space for Answers)

(Space for Answers)

Experiment No. 13

1.0 Title :

Assay of Hydrogen peroxide.

(To determine the % w/v of H_2O_2 in a given sample of hydrogen peroxide)

2.0 Prior Concepts:

Titration, titrate, titrant, oxidation and reduction reactions, oxidizing agent, reducing agents, decomposition of hydrogen peroxide.

3.0 New Concepts:

Proposition 1:

This assay is based upon the oxidation-reduction (permanganate) type of titration in which solution of potassium permanganate is acts as an oxidizing agent and hydrogen peroxide also act as an oxidizing agent but in presence of strong oxidizing agent like potassium permanganate hydrogen peroxide acts as reducing agent.

Proposition 2:

The ability of potassium permanganate solution to oxidize is due to the conversion of the MnO_4^- ion to Mn++ in acidic solution but MnO_4 ion is reduced by reducing agent like hydrogen peroxide solution containing MnO_4 ion are purple in colour, solution of salts containing Mn++ ions are colourless hence a permanganate solution is decolourised by reducing agent as longer as MnO4 is present in the solution. The moment there is an excess addition of potassium permanganate solution becomes purple thus permanganate ion act as an indicator.

Proposition 3:

In this assay dilute sulphuric acid is used for the conversion of MnO_4^- to Mn^{++}

$$MnO_4^- + 8H+ + 5e \xrightarrow[\text{condition}]{\text{acidic}} Mn^{++} + 4H_2O$$

But in alkaline or neutral condition there is a formation of brown colour precipitate of manganese dioxide (MnO_2), which interferes with detection of end point hence acid is used.

$$MnO_4^- + 4H+ + 3e \xrightarrow[\text{condition}]{\text{alkaline/neutral}} MnO_2 + 2H_2O$$

Proposition 4:

Factor:

Each ml of 0.1 N KMnO4 ≡ 0.0017 g of H_2O_2

Standards:

Hydrogen peroxide solution (20 Vol) contains not less than 5.0 percent w/v not more than 7.0 percent w/v H_2O_2, corresponding to about 20 times its volume of available oxygen.

Chemical Reactions:

I. Chemical reaction for standardization:

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

II. Chemical reaction for assay:

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

4.0 Learning Objectives :-

4.1 Intellectual Skills:

1. To understand the concept of assay.
2. To identify titrant and titrate in titration.
3. To understand the concept of factor calculation.
4. To understand the concept of percentage purity.
5. To understand the concept of volume strength calculation.

4.2 Motor Skills:

1. To observe the correct meniscus of solution in burette.
2. To observe the colour change at the end point of titration.

5.0 Apparatus :-

5.1 Material:

Burette (50ml), pipette (10 ml & 25 ml), beaker (100 ml), conical flask (10 ml), measuring cylinder (50 ml), glass funnel (small) etc.

5.2 Chemicals:

Hydrogen peroxide solution (6% w/v), standard potassium permanganate solution, oxalic acid (0.1N), dilute sulphuric acid and distilled water.

6.0 Stepwise procedure:

I. Standardization of potassium permanganate solution:

1. Take 10 ml of 0.1 N oxalic acid solutions in conical flask.
2. Add 10 ml dilute sulphuric acid.
3. Heat up to 70^oC and titrate this heated solution against given solution of potassium permanganate.
4. Report the reading by repeating it for three times and calculate the normality of potassium permanganate.

II. Assay of Hydrogen peroxide:

1. Take 10 ml of given hydrogen peroxide in volumetric flask. Dilute up to 100 ml mark.
2. Pipette out 25 ml diluted solution in conical flask.
3. To this add 20 ml dilute sulphuric acid.
4. Titrate this solution against standardized potassium permanganate.
5. Appearance of permanent pink colour indicates end-point.
6. Report the burette reading three times and calculate the percentage content by using factor.

7.0 Observations:

I. Standardization of potassium permanganate solution:

1. **Content of conical flask: -** 10 ml oxalic acid +20 ml dilute sulphuric acid.
2. **Solution in burette: -** Given potassium permanganate.
3. **Indicator: -** potassium permanganate (Self indicator).
4. **End point: -** colourless to light pink.

Observation table:

Sr.No.	Burette reading (ml)			Mean burette Reading (ml)
	Initial	Final	Difference	
1				
2				
3				

II. Assay of Ferrous Sulphate:

1. **Content of conical flask: -** 25 ml diluted solution of hydrogen peroxide + 20 ml dilute sulphuric acid.
2. **Solution in burette: -** standardized solution of potassium permanganate.
3. **Indicator: -** potassium permanganate (Self indicator).
4. **End point: -** colourless to light pink.

Observation table:

Sr.No.	Burette reading (ml)			Mean burette Reading (ml)
	Initial	Final	Difference	
1				
2				
3				

8.0 Calculations:

I. Factor Calculation:

From the reaction,

$$2KMnO_4 \equiv 5H_2O_2 \equiv 5(O)$$
$$2\ (158) \equiv 5\ (34) \equiv 5(16)$$
$$316 \equiv 17 \equiv 8$$

1000 ml 1 N $KMnO_4$ solution $\equiv$ 17 g of H_2O_2

Each ml of 0.1 N $KMnO_4$ solution $\equiv$ 0.0017 g of H_2O_2

II. Standardization calculation:

Potassium permanganate = Oxalic acid

$$N_1 \times V_1 = N_2 \times V_2$$

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

N_1 = ……

N_1 =……N

III. Percentage purity calculation (Y):

Each ml of 0.1 N $KMnO_4$ solution $\equiv$ 0.0017 g of H_2O_2

If 1ml of 0.1 N $KMnO_4$ solution $\equiv$ 0.0017 g of H_2O_2

Therefore,

'x' ml of 'n' N potassium permanganate = $\frac{0.0017 \times n \times x}{N}$ g of H_2O_2

= …….

= …….

If 25 ml of diluted sample contains = $\frac{0.0017 \times n \times x}{N}$ g of H_2O_2

= …….

= …….

$\therefore$ 100 ml of diluted sample contains = $\frac{0.0017 \times n \times x \times 100}{N \times 25}$ g of H_2O_2

= …….

Y = ……g of H_2O_2 is present in 10 ml of given undiluted sample.

If 10 undiluted sample contains = $\frac{0.0017 \times n \times x \times 100}{N \times 25}$ g of H_2O_2

Y = ……..

Therefore, 100 ml of given undiluted sample contains = $\frac{0.0017 \times n \times x \times 100}{N \times 25} \times \frac{100}{10}$

= ……..

Y = ……% w/v of H_2O_2.

(Where, x = Burette reading of assay, n = calculated normality, N = factor normality)

IV. Volume strength calculation:

$$2H_2O_2 \rightarrow 2H_2O + O_2 \uparrow$$

$\therefore$ 68 g of H_2O_2 gives 32 g of oxygen $\equiv$ 22.4 liters of oxygen at NTP (Normal temperature and pressure).

$\therefore$ 1 gm of H_2O_2 will give $\frac{22400}{68}$ ml of oxygen $\equiv$ 329.4 ml of oxygen

Let, concentration of H_2O_2 = y = ……% w/v

$\therefore$ 10 ml undiluted hydrogen peroxide contain $\frac{y}{100}$ = ……..g of O_2

$\therefore$ Contain $\frac{y}{100}$ g of H_2O_2 i.e., 10 ml of H_2O_2 gives $\frac{22400}{68} \times \frac{y}{100}$ = …….V is

the strength of given hydrogen peroxide.

9.0 Results:

1. The normality of potassium permanganate solution is ……N.
2. The given solution of hydrogen peroxide is ……% w/v.
3. The volume strength of given hydrogen peroxide is ……V.

10.0 Questions:

(Answer the following questions, Q.… Q…..Q.…and Q….., Question Number to be allotted by the teacher)

1. Write the properties of potassium permanganate with chemical reaction.
2. Which indicator is used in this titration?
3. State the meaning of oxidation-reduction type of titration.
4. How potassium permanganate acts as self-indicator?
5. State the meaning of Oxidation, Reduction.
6. Write the reactions involved in assay of hydrogen peroxide.
7. Which primary standard solution is used to standardize potassium permanganate solution?
8. Write the procedure for preparation for primary standard solution.
9. Write the principle and reaction involved in assay of hydrogen peroxide.
10. State the meaning of 20V of hydrogen peroxide.
11. Write the category of hydrogen peroxide.

12. Why hydrogen peroxide solution is stored in plastic containers.
13. How to calculate the volume strength of 6%w/v of hydrogen peroxide?
14. Find out commercial/ pharmaceutical uses of hydrogen peroxide.
15. Write standards and factor for calculation assay of hydrogen peroxide.

11.0 Reference:

1. Indian pharmacopoeia 1985.
2. Indian pharmacopoeia 1996.

(Space for Answers)

(Space for Answers)

Experiment No. 14

1.0 Title :

Assay of Magnesium Sulphate.

(To determine the % w/w of $MgSO_4$ in a given sample of Magnesium sulphate)

2.0 Prior Concepts:

Assay, Titration, Titrant, Titrate, Indicator, Normality, Buffer, co-ordinate, chelates.

3.0 New Concepts: Complxometric titration

Proposition 1:

This assay is based upon complexometric type of titration in which simple metal ion is transformed into complex ion by addition of reagent which is known as 'ligand'. The complex formed is stable and water-soluble.

Proposition 2:

In this titration metal ion accepts electrons and ligand (complexing or chelating agent) donate it. As in a ligand molecule, there is presence of at least one lone pair of electrons through which co-ordinate linkage with metal ion takes place. Ligand molecule usually posses oxygen, nitrogen or sulphur in one or more number in their structure.

Proposition 3:

Ligands are classified depending upon their number of sites for attachment viz unidentate (single site), bidentate (two sites) and multidentate (many sites). Disodium edetate is multidentate ligand, which forms complex with metal ion by donating lone pair of electrons in presence of strong ammonia ammonium chloride buffer. The end point is determined by addition of mordant black-11 as an indicator, the colour changes from pale pink to blue.

Proposition 4:

Factors:

I. Factor for standardization.

Each ml of 0.05 M disodium edetate = 0.000654 g of Zn.

II. Factor for Assay.

Each ml of 0.05 M disodium edetate = 0.00602 g of $Mg\ SO_4$

Standards:

Magnesium sulphate contains not less than 99.0 percent and not more than 100.5 percent of $MgSO_4$, calculated with reference to dried substance.

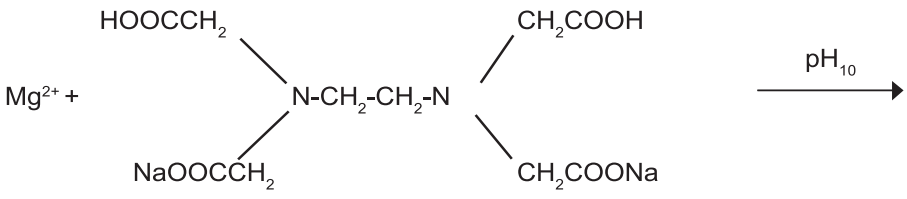
Chemical Reaction: -

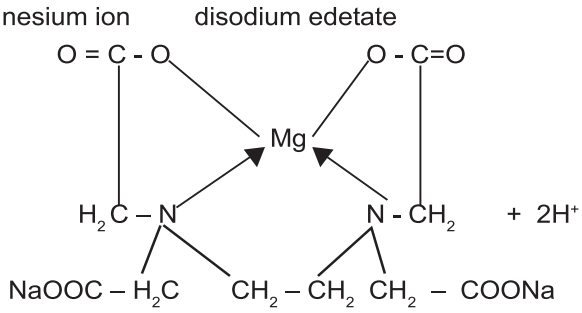
The process of complex formation can be represented by the reactions.

$$Mg^{2+} + Na_2H_2Y \longleftrightarrow Na_2(MgY) + 2H^+$$

Metal complex

Where, Na_2H_2V – disodium edetate.





Magnesium edetate complex

$Mg^{+2} + (CH_2)_2 \bullet N_2 \bullet (CH_2COONa)_2 \bullet (CH_2COOH)_2 \rightarrow (CH_2)_2 \bullet N_2 \bullet (CH_2COONa)_2 \bullet (CH_2COO)_2 Zn + 2H^+$

4.0 Learning Objectives:

4.1 Intellectual Skills:

1. To understand the concept of assay.
2. To understand the concept of complex formation.
3. To understand the concept of factor calculation.
4. To understand the concept of percentage purity.

4.2 Motor Skills:

1. To observe the colour change at the end point of titration.

5.0 Apparatus :

5.1 Materials:

Burette (50ml), pipettes (10 ml & 25 ml), conical flask (250 ml), burette stand.

5.2 Chemicals:

Magnesium sulphate, Disodium edetate (Aprox 0.05 M), strong ammonia-ammonium chloride buffer, mordant black-11, granulated zinc, 2M sodium hydroxide, dilute hydrochloric acid, bromine water and ammonia buffer.

6.0 Stepwise Procedure :

I. Standardization of disodium edetate:

1. Weigh accurately about 0.8 g of granulated zinc; dissolve by gentle warming in 12 ml of dilute hydrochloric acid and 0.1 ml of bromine water.
2. Boil to remove excess bromine, cool and add sufficient water to produce 200 ml in a conical flask.
3. Pipette 20 ml of the resulting solution into a flask and nearly neutralize with 2 M sodium hydroxide.
4. Dilute to about 150 ml of water, add sufficient ammonia buffer pH 10 to dissolve the precipitate and add 5 ml in excess.
5. Add 50 mg of mordant black-11 mixture.
6. Titrate the solution with the disodium edetate solution until the solution turns green.

II. Assay of magnesium sulphate:

1. Accurately weigh 0.3 g of dried sample and add in 250 ml conical flask.
2. Dissolve the sample in 50 ml of water.
3. Add 10 ml strong ammonia-ammonium chloride buffer solution and mix well.
4. To this mixture add 0.1 g of mordant black-11.
5. Titrate the solution in conical flask with standardized (aprox. 0.05 M) disodium edetate from the burette.
6. End point determining by colour change from pink to blue.

7.0 Observation :

I. Standardization of disodium edetate:

1. **Content of conical flask:-** 0.8 g zinc + 12 ml dilute hydrochloric acid + 0.1 ml bromine water + 200 ml water → pipetted 20 ml + 2 M sodium hydroxide (to neutralized) + 150 ml water + ammonia (to dissolve ppt) buffer + 50 mg mordant black -11(II).
2. **Solution in burette:-** Given disodium edetate (aprox. 0.05 M)
3. **Indicator:-** Mordant black 11(II).
4. **End point:-** pink to bottle green.

Observation table:

Sr.No.	Burette reading (ml)			Mean burette Reading (ml)
	Initial	Final	Difference	
1				
2				
3				

II. Assay of magnesium sulphate:

1. **Weight of sample: -** 0.3 g magnesium sulphate (dried)
2. **Content of conical flask: -** 0.3 g of sample + 50 ml water + 10 ml strong ammonia-ammonium chloride buffer solution + 0.1 g of mordant black-11(II).
3. **Solution in burette: -** Standardized disodium edetate (aprox. 0.05 M)
4. **Indicator: -** Mordant black-11 (II).
5. **End point: -** Pink to blue.

Observation table:

Sr.No.	Burette reading (ml)			Mean burette Reading (ml)
	Initial	Final	Difference	
1				
2				
3				

8.0 Calculations :

I. Factor Calculation: -

Now from the reaction,

Disodium edetate ≡ magnesium sulphate

$C_{10}H_{14}N_2Na_2O_8, 2H_2O \equiv MgSO_4$

$372 \equiv 120.47$

1000 ml of 1 M disodium edetate ≡ 120.47 g of $MgSO_4$

∴ 1 ml of 0.1 M disodium edetate ≡ 0.012047 g of $MgSO_4$

∴ Each ml of 0.05 M disodium edetate ≡ 0.0060235 g of $MgSO_4$

II. Standardization calculation: -

$$\text{Molarity of disodium edetate} = \frac{\text{weight of granulated zinc}}{\text{Burette reading X 0.000654}}$$

$$= \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots$$

III. Calculation for Percentage purity: -

$$\text{1 ml of 'm' M disodium edetate} = \frac{0.0060235 \text{ X m g of } MgSO_4}{M}$$

$$= \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots$$

$$= \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots$$

$$\therefore \text{ 'X ' ml of 'm' M disodium edetate} = \frac{0.0060235 \text{ X m X x g of } MgSO_4}{M}$$

$$= \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots$$

$$= \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots$$

$$\therefore \text{ 'w' gm of sample contains} = \frac{0.0060235 \text{ X m X x g of } Mg\ SO_4}{M}$$

$$= \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots$$

$$= \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots$$

$$\therefore \text{ 100 g of sample contains} = \frac{0.0060235 \text{ X m X x X 100 g of } MgSO_4}{M \text{ X w}}$$

$$= \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots$$

$$= \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \text{ \% of } MgSO_4$$

(Where x = burette reading of assay, w = weight of magnesium sulphate,

m = calculated molarity and M = factor molarity)

9.0 Results :

1. Molarity of disodium edetate is ……..M.
2. The given sample of magnesium sulphate contains…….% w/w of $MgSO_4$.

10.0 Questions:

(Answer the following questions, Q.… Q…Q.…and Q.… Question Number to be allotted by the teacher)

1. Write the assay method for magnesium sulphate?
2. State the meaning of complexation.
3. Write the principle of complexometric titrations.
4. Write the chemical reactions involved in magnesium sulphate assay.
5. Write the procedure for magnesium sulphate assay.
6. Write the formula for determination of percent w/w.
7. State the reason for formation of complex with disodium edetate.
8. State the reason for use of strong ammonia-ammonium chloride buffer solution.
9. State the meaning of buffer.
10. Draw the structure of disodium edetate.

11. Draw the structure of metal complex.
12. Why disodium edetate known as multidentate ligand?
13. Write the contents of Mordant black-11 (II) mixture.

11.0 Reference:

Indian Pharmacopoeia 1996.

(Space for answers)

(Space for answers)

Experiment No. 15

1.0 Title :

Assay of Calcium Gluconate.

(To determine % w/w of $C_{12}H_{22}CaO_{14}H_2O$ in given sample of calcium gluconate)

2.0 Prior Concepts:

Titration, Assay, Titrant, Titrate, Indicators, Normality.

3.0 New Concepts: Complexometric titration

Proposition 1:

This assay is based upon complexometric type of titration in which simple metal ion is transformed into complex ion by addition of reagent which is known as 'ligand' (complexing agent). The complex formed is stable and water-soluble.

Proposition 2:

In this titration metal ion accepts electrons and ligand donates it. In a ligand molecule, there is presence of at least one lone pair of electrons through which co-ordinate linkage with metal ion takes place. Ligand molecule usually posses oxygen, nitrogen or sulphur in one or more number in their structure.

Proposition 3:

Ligands are classified depending upon their number of sites for attachment viz unidentate (single site), bidentate (two sites) and multidentate (many sites). Disodium edetate is multidentate ligand which form complex with metal ion by donating lone pair of electrons in presence of strong ammonia solution, the end point is determined by addition of mordant black-11 as an indicator, the colour changes from red to blue.

(**Note:** 5 ml of 0.05 M magnesium sulphate solution is added before the titration, this is done to make end point sharp)

Proposition 4:

Factors:

I. Factor for standardization.

Each ml of 0.05 M disodium edetate = 0.000654 g of Zn.

II. Factor for Assay.

Each ml of 0.05 M disodium edetate = 0.02242 g of $C_{12}H_{22}CaO_{14}H_2O$.

Standards:

Calcium gluconate contains not less than 98.5 percent and not more than 102.0 percent of $C_{12}H_{22}CaO_{14}H_2O$.

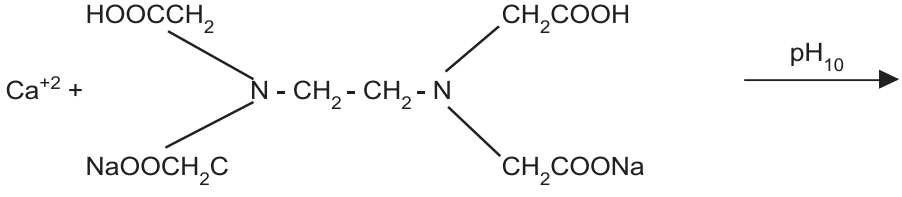
Chemical Reaction: -

The process of complex formation can be represented by the reactions.

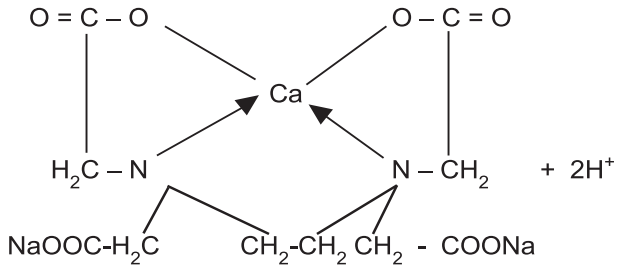
$$Ca^{2+} + Na_2H_2V \longleftrightarrow Na_2(CaV) + 2H^+$$

Metal complex

Where, Na_2H_2V - disodium edetate.



Calcium ion disodium edetate



Calcium edetate complex

4.0 Learning Objectives:

4.1 Intellectual Skills:

1. To understand the concept of assay.
2. To understand the concept of complex formation.
3. To understand the concept of factor calculation.
4. To understand the concept of percentage purity.

4.2 Motor Skills:

1. To observe the colour change at the end point of titration.

5.0 Apparatus :

5.1 Materials:

Burette (50ml), pipette (10 ml, 25 ml), conical flask (250 ml).

5.2 Chemicals:

Calcium gluconate, Disodium edetate (aprox. 0.05 M), strong ammonia solution, mordant black-11, magnesium sulphate solution (approx. 0.05 M), granulated zinc, 2M sodium hydroxide, dilute hydrochloric acid, bromine water and ammonia buffer.

6.0 Stepwise Procedure :

I. Standardization of disodium edetate:

1. Weigh accurately about 0.8 g of granulated zinc, dissolve by gentle warming in 12 ml of dilute hydrochloric acid and 0.1 ml of bromine water.
2. Boil to remove excess bromine, cool and add sufficient water to produce 200 ml in a conical flask.
3. Pipette 20 ml of the resulting solution into a flask and nearly neutralize with 2M sodium hydroxide.
4. Dilute to about 150 ml of water, add sufficient ammonia buffer pH 10 to dissolve the precipitate and add 5 ml in excess.
5. Add 50 mg of Mordant black 11 mixture.
6. Titrate the solution with the disodium edetate solution until the solution turns green.

II. Assay of calcium gluconate:

1. Accurately weigh 0.5 g of sample and add in 250 ml conical flask.
2. Dissolve the sample in 50 ml of warm water and cool it.
3. Add 5 ml of 0.05 M magnesium sulphate solution and 10 ml of strong ammonia solution mix well.
4. And add 0.1 gm of mordant black-11.
5. This solution in conical flask titrate with standardized disodium edetate (aprox. 0.05 M) in burette.
6. End point is determined by colour change from red to blue.

7.0 Observation :

I. Standardization of disodium edetate:

1. **Content of conical flask:-** 0.8 g zinc + 12 ml dilute hydrochloric acid + 0.1 ml bromine water + 200 ml water → pipetted 20 ml + 2 M sodium hydroxide + 150 ml water + ammonia buffer + 50 mg mordant black 11(II).
2. **Solution in burette:-** Given disodium edetate (aprox. 0.05 M)
3. **Indicator:-** Mordant black 11(II).
4. **End point:-** Solution becomes green.

Observation table:

Sr.No.	Burette reading (ml)			Mean burette Reading (ml)
	Initial	Final	Difference	
1				
2				
3				

II. Assay of calcium gluconate:

1. **Weight of sample: -** 0.5 g of calcium gluconate.
2. **Content of conical flask: -** 0.5 g of sample + 50 ml water + 5 ml of 0.05 M $MgSO_4$ solution + 10 ml strong ammonia solution + 0.1 g of Mordant black 11(II).
3. **Solution in burette: -** Standardized disodium edetate (aprox 0.05M)
4. **Indicator: -** Mordant black-11 (II).
5. **End point: -** Red to blue.

Observation table:

Sr.No.	Burette reading (ml)			Mean burette Reading (ml)
	Initial	Final	Difference	
1				
2				
3				

8.0 Calculations :

I. Factor Calculation: -

From the reaction,

Disodium edetate ≡ Calcium gluconate

$C_{10}H_{14}N_2Na_2O_8, 2H_2O \equiv C_{12}H_{22}O_{14}\ Ca\ H_2O$

1000 ml of 1 M disodium edetate ≡ 448.4 g of $C_{12}H_{22}Ca\ O_{14}\ H_2O$

1 ml of 0.1 M disodium edetate ≡ 0.04484 g of $C_{12}H_{22}Ca\ O_{14}\ H_2O$

Each ml of 0.05 M disodium edetate ≡ 0.02242 g $C_{12}H_{22}Ca\ O_{14}\ H_2O$

II. Standardization calculation:-

$$\text{Molarity of disodium edetate} = \frac{\text{Weight of granulated zinc}}{\text{Burette reading X } 0.000654}$$

=.....M

III. Calculation of Percentage purity:

1 ml of 'm' M disodium edetate solution = $\frac{0.02242 \text{ g} \times \text{m g}}{\text{M}}$ $C_{12} H_{22} Ca O_{14} H_2O$

=.....

=.....

'x' ml of 'm' M disodium edetate solution = $\frac{0.02242 \times \text{m} \times \text{x g}}{\text{M}}$ of $C_{12} H_{22} Ca O_{14} H_2O$

= .....

= .....

$\therefore$ 'w ' g of sample contains = $\frac{0.02242 \times \text{m} \times \text{x g}}{\text{M}}$ of $C_{12} H_{22} Ca O_{14} H_2O$

= .....

= .....

$\therefore$ 100 g of sample contains = $\frac{0.02242 \times \text{m} \times \text{x} \times 100}{\text{M} \times \text{w}}$ g of $C_{12} H_{22} Ca O_{14} H_2O$

= .....

= .....

(Where x = burette reading of assay, w = weight of calcium gluconate, m = calculated molarity and M = factor molarity)

9.0 Results :

1. Molarity of disodium edetate is .....M.
2. The given sample of calcium gluconate contains .....% w/w of $C_{12} H_{22} Ca O_{14} H_2O$.

10.0 Questions:

(Answer the following questions, Q.... Q...Q....and Q.... Question Number to be allotted by the teacher)

1. Write the weight of sample used as per I.P.
2. Write the principle involved in assay of calcium gluconate.
3. State the use of disodium edetate.
4. Write the procedure for calcium gluconate assay.
5. Write the chemical reaction involved in calcium gluconate assay.
6. Write the apparatus used in titration.
7. Draw the structure of disodium edetate.
8. Draw the structure of calcium gluconate.
9. Write the standards of calcium gluconate as per I.P. 1996.
10. Draw the structure of calcium edetate complex.
11. Write the factor calculation for calcium gluconate assay.
12. Write the content of mordant black 11 and how it is to be prepared.
13. Write the meaning of following
    a. Ligand
    b. Chelating agent
    c. Sequestering agent

11.0 Reference:

Indian Pharmacopoeia 1996

(Space for answers)

Experiment No. 16

1.0 Title :

Assay Of Sodium Chloride. (By Mohr's Method)

(To Determine % W/W Of Nacl In A Given Sample Of Sodium Chloride)

2.0 Prior Concepts:

Titration, Assay, Precipitation, Tritrate, Direct Titration, Titrant, Indicator And Normality.

3.0 New Concepts: Argentimetric Titration

Proposition 1:

This assay based upon argentimetric type of titration where silver nitrate solution used as a titrant. When silver nitrate solution is run into solution of sodium chloride a precipitate of silver chloride is formed, the end point is at which all chlorides are precipitated as silver chloride and next drop of silver nitrate causes precipitation of silver chromate by reacting with indicator potassium chromate.

Proposition 2:

Factor:

Each ml of 0.1N Silver Nitrate is equivalent to 0.005844 g of NaCl.

Standards:

Sodium Chloride contains not less than 99.5 percent and not more than 100.5 percent of NaCl.

Chemical Reaction:

$$\underset{\text{Sodium Chloride}}{NaCl} + \underset{\text{Silver Nitrate}}{AgNO_3} \rightarrow \underset{\text{Silver Chloride}}{AgCl\downarrow} + \underset{\text{Sodium Nitrate}}{NaNO_3}$$

$$\underset{\text{Potassium chromate}}{K_2CrO_4} + \underset{\text{Silver Nitrate}}{2AgNO_3} \rightarrow \underset{\text{Pot Nitrate}}{2KNO_3} + \underset{\text{Silver chromate (red ppt)}}{Ag_2CrO_4\downarrow}$$

4.0 Learning Objectives:

4.1 Intellectual Skills:

1. To understand the concept of Argentimetric.
2. To understand concept of factor calculation.
3. To determine the concept of percentage purity.
4. To understand the concept of direct titration.

4.2 Motor Skills:

1. The ability to observe the precipitation formation in titration.
2. To observe the colour change at the end point of titration.

5.0 Apparatus :

5.1 Material:

Burette (50ml), pipette (10,25 ml), beaker (100 ml), Stoppered Conical flask (250 ml), Measuring cylinder (50 ml), Glass funnel (small) etc.

5.2 Chemicals:

Sodium Chloride, Silver nitrate solution, potassium chromate solution, acetic acid and methanol.

6.0 Stepwise procedure:

I. Standardization of silver nitrate solution.

1. Weigh accurately 0.1 g of sodium chloride previously dried at 110° C for 2 hours.
2. Dissolve it in 5 ml distilled water.

3. Add 5 ml of acetic acid and 50 ml of methanol.
4. Add few drops of potassium chromate (5%), stir preferably with magnetic stirrer.
5. Fill the burette with given solution of silver nitrate.
6. Remove the air bubble and adjust the zero level.
7. Titrate the solution in conical flask by running the solution from burette. The appearance of red precipitate indicates the end point.
8. Report the reading by repeating it for three times and calculate the normality of silver nitrate.

II. Assay of sodium chloride. (I.P. 1966 Second Edition)

1. Weigh accurately 0.25 g of sodium chloride.
2. Dissolve it in about 50 ml water.
3. Dissolve it completely by shaking.
4. Titrate with 0.1 N silver nitrate solution using solution of potassium chromate as an indicator.
5. End point of the titration is yellow to red.

7.0 Observation :

I. Standardization of silver nitrate: -

1. **Content of conical flask: -** 0.1 g sodium chloride + 5 ml distilled water + 5 ml acetic acid + 50 ml methanol.
2. **Solution in burette: -** Given Solution of silver nitrate.
3. **Indicator: -** potassium chromate (5%).
4. **End point: -** Appearance of red precipitate.

Observation table:

Sr.No.	Burette reading (ml)			Mean burette Reading (ml)
	Initial	Final	Difference	
1				
2				
3				

II. Assay of sodium chloride: -

1. **Content of conical flask-** 0.25 g of sodium chloride + 50 ml distilled water.
2. **Solution in burette-**Standardized silver nitrate solution.
3. **Indicator-**Potassium chromate solution.
4. **End point -** yellow to red.

Observation table:

Sr.No.	Burette reading (ml)			Mean burette Reading (ml)
	Initial	Final	Difference	
1				
2				
3				

8.0 Calculation:

I. Factor Calculation:

From the reaction -

$AgNO_3 \equiv NaCl$

$170 \equiv 58.45$

1000 ml of 1N silver nitrate ≡ 58.45 g of NaCl.

Each ml of 0.1N Silver nitrate = 0.005845 g of NaCl

II. Standardization calculation:

$$\text{Normality of Silver nitrate} = \frac{\text{Wt of sodium chloride}}{\text{Burette reading x } 0.005845}$$

= .....

= .....N

III. Percentage purity calculation:

Each ml of 0.1N Silver Nitrate ≡ 0.005845 g of NaCl

If 1 ml of 0.1N Silver Nitrate ≡ 0.005845 g of NaCl

'x' ml of 'n' N silver nitrate $= \frac{0.005845 \text{X n X x}}{\text{N}}$ g of NaCl

= .....

= .....

∴ 'W' ml of sample contains $= 0.005845 \frac{\text{X m X x}}{\text{N}}$ g of NaCl

= .....

= .....

∴ 100 ml of sample contains $= \frac{0.005845 \text{ X n X x X } 100}{\text{NX W}}$ g of NaCl

= .....

= .....

(Where, x = burette reading, W = weight of sample taken for analysis,
n = obtained normality, and N = factor normality)

9.0 Results:

1. Normality of silver nitrate is ……....N
2. The given sample of sodium chloride contains . ……..% w/w of NaCl.

10.0 Questions:

(Answer the following questions, Q.... Q...Q....and Q.... Question Number to be allotted by the teacher)

1. Which method is used for assay analysis of sodium chloride?
2. Why this method is known as 'direct titration' method.
3. Name the indicator used in this method.
4. Give the reaction involved in assay of sodium chloride.
5. Why this type of titration is known as 'Argentimetric titration'.
6. Write the factor given in the I.P.
7. Give the standards for sodium chloride according to I.P. on dried substance.
8. Write the principle involved in assay of sodium chloride.
9. Write the preparation 0.9% w/v solution of sodium chloride.
10. Why 0.9% w/v NaCl solution known as normal saline solution.
11. Write the difference between Mohr's method and Volhard's method.

11.0 Reference:

Pharmacopoeia of India second edition 1966.

(Space for answers)

Experiment No. 17

1.0 Title :

Assay of Ammonium Chloride (Volhard's method)

(To determine the % w/w of NH_4Cl in a given sample of ammonium chloride)

2.0 Prior Concepts:

Titration, assay, precipitation, tritrate, titrant, indicator, back titration and normality.

3.0 New Concepts: Argentimetric Titration

Proposition 1:

This assay is based upon argentimetric (precipitation) type of titration. Precipitation is the combination of two ionic species to form a very insoluble product. In this method silver ion reacts with thiocyanate ion to form insoluble silver thiocyanate.

$$Ag^+ + SCN^- \rightarrow AgSCN\downarrow$$

Insoluble silver thiocyanate

Proposition 2:

In this assay titration is carried out with standard silver nitrate solution and a standard solution of ammonium thiocyanate, ferric ammonium sulphate is used as an indicator, the end point of titration is indicated by the appearance of persistent reddish colour due to formation of ferric ferro- thiocyanate complex.

Proposition 3:

Assay of ammonium chloride which contain halide (Cl^-) ion, which do not react with thiocyanate (SCN^-) ion, therefore, it is assayed by back titration, Volhard's method, in which fixed volume of halide (ammonium chloride) is used along with excess of silver nitrate solution, acidified with nitric acid. A part of silver nitrate reacts with the halide ion to form an insoluble silver chloride complex in presence of nitrobenzene.

(Note: Nitrobenzene protects the reaction of silver chloride precipitate with ammonium thiocyanate by forming a protective film around silver chloride particles).

Then unconsumed or excess of silver nitrate is back titrated with the standard ammonium thiocyanate solution. The amount of silver nitrate that reacted with the halide (Cl^-) is found as the difference between the known amount of silver nitrate and the excess of silver nitrate by carrying out back titration.

Proposition 4:

Factors:

I. Factor for standardization of silver nitrate:

Each ml of 0.1 N silver nitrate ≡ 0.005844 g of NaCl.

II. Factor for standardization of ammonium thiocyanate:

Each ml of 0.1N silver nitrate ≡ 0.007612 g of NH_4SCN.

III. Factor for assay:

Each ml of 0.1 N silver nitrate ≡ 0.005349 g NH_4Cl.

Standards: -

Ammonium chloride contains not less than 99.0 percent and not more than 100.5 percent of NH4Cl, calculated with reference to the dried substance.

Chemical reactions:

I. Chemical reaction for standardization:

i. $NaCl + AgNO_3 \rightarrow AgCl\downarrow + NaNO_3$

Sodium chloride — silver nitrate — silver chloride — sodium nitrate

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

Potassium chromate — Silver chromate

II. Chemical reaction for assay:

i. $NH_4Cl + AgNO_3 \rightarrow AgCl\downarrow + NH_4NO_3$

Ammonium chloride — silver nitrate — silver chloride — ammonium nitrate

ii. $AgNO_3 + NH4\ SCN \rightarrow AgSCN\downarrow + NH4\ NO_3$

Excess silver nitrate — ammonium thiocyanate — silver thiocyanate — ammonium nitrate

iii. $FeNH_4(SO_4)_2 + 3\ NH_4SCN \rightarrow Fe(SCN)_3 + 2(NH_4)_2SO_4$

4.0 Learning Objectives:

4.1 Intellectual Skills:

1. To understand the concept of assay.
2. To identify titrant and titrate in titration.
3. To understand the concept of factor calculation.
4. To understand the concept of percentage purity.
5. To understand the concept of back titration.
6. To understand the concept of blank titration.

4.2 Motor Skills:

1. The ability to observe the precipitation formation in titration.
2. To observe the colour change at the end point of titration.

5.0 Apparatus :

5.1 Material:

Burette (50ml), pipette (10 ml & 25 ml), beaker (100 ml), conical flask (10 ml), volumetric flask (100 ml).

5.2. Chemicals:

Ammonium chloride sample, nitric acid, nitrobenzene, silver nitrate solution, ammonium thiocyanate solution, ferric ammonium sulphate and distilled water.

6.0 Stepwise procedure:

I. Standardization of silver nitrate solution.

1. Weigh accurately 0.1 g of sodium chloride previously dried at 110° C for 2 hours.
2. Dissolve it in 5 ml distilled water.
3. Add 5 ml of acetic acid and 50 ml of methanol.
4. Add few drops of potassium chromate (5%), stir preferably with magnetic stirrer.
5. Fill the burette with given solution of silver nitrate.
6. Remove the air bubble and adjust the zero level.
7. Titrate the solution in conical flask by running the solution from burette. The appearance of red precipitate indicates the end point.
8. Report the reading by repeating it for three times and calculate the normality of silver nitrate.

II. Standardization of ammonium thiocyanate.

1. Pipette out 30 ml of previously standardized silver nitrate solution into glass stoppered flask, dilute with 50 ml distilled water.
2. Add 2 ml of nitric acid and 2 ml of ferric ammonium sulphate solution.
3. Titrate this solution with given solution of ammonium thiocyanate.
4. Appearance of red-brown colour indicates and point.

III. Assay of ammonium chloride:

1. Weigh accurately about 0.1 g of ammonium chloride.
2. Dissolve in 20 ml of distilled water and add a mixture of 1.5 ml of concentrated nitric acid, 2.5 ml of nitrobenzene.
3. Add 25 ml of 0.1 N silver nitrate solutions. Shake well vigorously for 2 minutes.
4. Then titrate above solution with ammonium thiocyanate solution using ferric ammonium

sulphate as an indicator.

5. Perform blank titration.
6. Report the reading by repeating it for three times and calculate the percentage purity. (Note: This method is performed in acidic condition only).

7.0 Observation :

I. Standardization of silver nitrate: -

1. **Content of conical flask: -** 0.1 g sodium chloride + 5 ml distilled water + 5 ml acetic acid + 50 ml methanol.
2. **Solution in burette: -** Given Solution of silver nitrate.
3. **Indicator: -** potassium chromate (5%).
4. **End point: -** Appearance of red precipitate.

Observation table:

Sr.No.	Burette reading (ml)			Mean burette Reading (ml)
	Initial	Final	Difference	
1				
2				
3				

II. Standardization of ammonium thiocyanate: -

1. **Content of conical flask: -** 30 ml of standardized solution of silver nitrate + 50 ml distilled water + 2 ml nitric acid.
2. **Solution in burette: -**Given Solution of ammonium thiocyanate.
3. **Indicator: -** ferric ammonium sulphate solution.
4. **End point: -**Appearance of red-brown colour.

Observation table:

Sr.No.	Burette reading (ml)			Mean burette Reading (ml)
	Initial	Final	Difference	
1				
2				
3				

III. Assay of Ammonium Chloride: -

a. Back titration

1. **Content of conical flask: -** 0.1 g ammonium chloride+ 20 ml distilled water + 1.5 ml concentrated nitric acid + 2.5 ml nitrobenzene + 25 ml silver nitrate (aprox.0.1 N)
2. **Solution in burette: -** Standardized solution of ammonium thiocyanate.
3. **Indicator: -** Ferric ammonium sulphate.
4. **End point: -** Colourless to reddish brown.

Observation table:

Sr.No.	Burette reading (ml)			Mean burette Reading (ml)
	Initial	Final	Difference	
1				
2				
3				

8.0 Calculation:

I. Factor Calculation:

$AgNO_3 \equiv NH_4Cl$

169 ≡ 53.49

1000 ml of 1 N silver nitrate ≡ 53.49 g of NH_4Cl

1ml of 0.1 N silver nitrate ≡ 0.05349 g of NH_4Cl

1ml of 0.1 N silver nitrate ≡ 0.005349 g NH_4Cl

II. Standardization calculation for silver nitrate:

$$\text{Normality of silver nitrate} = \frac{\text{Wt of sodium chloride}}{\text{Burette reading} \times 0.005844}$$

= ……

= ……N

III. Standardization calculation for ammonium thiocyanate:

Ammonium thiocyanate = Silver nitrate

$N_1 \times V_1 = N2 \times V_2$

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

= ……

=……N

IV. Percentage purity calculation:

Note : The volume of silver nitrate solution utilized by the sample

X ml = blank reading - back reading =…… ml.)

Each ml of 0.1 N silver nitrate ≡ 0.005349 g NH_4Cl

If 1 ml of 'n' N silver nitrate ≡ 0.005349 X n g NH_4Cl

'x' ml of 'n' N silver nitrate = $\frac{0.005349 \times n \times x}{N}$ g of NH_4Cl

= ……

= ……

∴ 'w' g of sample contains = $\frac{0.005349 \times n \times x}{N}$ g of NH_4Cl

= ……

= ……

∴ 100 g of sample contains = $\frac{0.005349 \times n \times x \times 100}{NX\ w}$ g of NH_4Cl

= ……

= ……

(Where, x = Blank reading - back reading, w = weight of ammonium chloride, n = obtained normality, and N = factor normality)

9.0 Results:

1. Normality of silver nitrate is.....N.
2. Normality of ammonium thiocyanate is ……N.
3. The given sample of ammonium chloride contains …….. % w/w of NH_4Cl

10.0 Questions:

(Answer the following questions, Q.… Q…Q.…and Q.… Question Number to be allotted by the teacher)

1. State the principle of ammonium chloride assay
2. Is it possible to use another method for assay of ammonium chloride? Justify your answer by describing the method.
3. Write the factor calculation for ammonium chloride.
4. State the role of nitric acid in this assay.
5. Write the chemical reaction involved in ammonium chloride assay.
6. Write the assay procedure for ammonium chloride.
7. Write the formula for percentage purity calculation.
8. Write the factor for ammonium chloride assay.
9. Why these titrations are called argentimetric titrations.

11.0 Reference:

1. Indian pharmacopoeia 1985
2. Indian pharmacopoeia 1996

(Space for answers)

(Space for answers)

Experiment No. 18

1.0 Title :

Identification test for sodium chloride.

(To perform and report identification tests on given sample of sodium chloride as per Indian Pharmacopoeia)

2.0 Prior Concepts:

Identification tests, Standards, Category, Solubility.

3.0 New Concepts: Monograph of sodium chloride

Proposition 1:

Chemical Formula: - NaCl

Molecular weight: - 58.44

Proposition 2: Description

It gives organoleptic properties of the given compound.

Nature: -Crystals or crystalline powder.

Colour: - Colourless or white.

Odour: - Odourless.

Proposition 3: Solubility

Statements of solubility's are indicated by a descriptive phrase and are intended to apply at 20° C to 30° C.

Sodium chloride is freely soluble in water and slightly more soluble in boiling water; practically insoluble in ethanol.

The following table indicates the meaning of the terms used in statements of approximate solubility's.

Description Term	Approximate volume of solvent in milliliters per gram of solute
Very soluble	Less than 1
Freely soluble	From 1 to 10
Soluble	From 10 to 30
Sparingly soluble	From 30 to 100
Slightly soluble	From 100 to 1000
Very slightly soluble	From 1000 to 10,000
Insoluble or practically insoluble	More than10, 000

Proposition 4: Identification test.

This test is described to verify the article being examined in accordance with the label on container. Failure of these test indicate that the article may be mislabeled or substituted.

Standards: -Sodium chloride contains not less than 99.0 percent and not More than 100.5 percent of NaCl, calculated with reference to the dried substances.

Category: -Pharmaceutical aid (tonicity agent); fluid and electrolyte replenisher.

4.0 Learning Objectives:

4.1 Intellectual Skills:

1. To understand the concept of identification tests.

4.2 Motor Skills:

1. To identify the physical properties of given sample.
2. To observe the solubility in water and other solvents.
3. To observe the colour developed in test solution.
4. To observe the precipitate formed in test solution.

5.0 Apparatus :

5.1 Materials:

Test tubes, test tube stand, Graduated Pipettes (0.5 ml,1 ml & 10 ml)

5.2 Chemicals:

Sodium chloride, potassium carbonate, potassium antimonate solution, sulphuric acid, 1M acetic acid, magnesium uranyl acetate solution, dilute nitric acid, silver nitrate solution, potassium dichromate, diphenylecarbazide, 15% w/v potassium carbonate solution and dilute ammonia solution.

6.0 Stepwise procedure:

1. Procedure for Physical Tests:

Description:

Observe the given compound critically for the following description. The compound is colourless crystals or white, crystalline powder, odourless.

Solubility:

Perform solubility test in the different solvents. The compound is freely soluble in water and slightly more soluble in boiling water; practically insoluble in ethanol.

2. Procedure for Identification Tests:

A. Dissolve 0.1 g of the substance being examined (equivalent to about 2 mg of chloride ion), in 2 ml of water. Acidify with dilute nitric acid, and add 0.5 ml of silver nitrate solution, shake and allow to stand; A curdy, white precipitate formed, which is insoluble in nitric acid but soluble, after being well washed with water, in dilute ammonia solution, from which it is reprecipitated by the addition of dilute nitric acid.

B. Introduce into a test tube 0.5 g of the substance being examined (equivalent to about 10 mg of chloride ion), add 0.2 g of potassium dichromate and 1 ml of sulphuric acid. Place a filter paper strip moistened with 0.1 ml of diphenylcarbazide solution over the mouth of test tube; The paper turns violet red. (Do not bring the moistened paper into contact with the potassium dichromate solution).

C. Dissolve 0.1 g of substance being examined in 2 ml of water. Add 2 ml of 15% w/v solution of potassium carbonate and heat to boiling no precipitate is produced. Add 4 ml of freshly prepared potassium antimonate solution and heat to boiling. Allow to cool in ice and if necessary scratch the inside of test tube with glass rod; a dense, white precipitate is formed.

D. Acidify a solution of the substance being examined with 1 M acetic acid and add a large excess of magnesium uranyl acetate solution; a yellow, crystalline precipitate is formed.

7.0 Observation :

Test	Observation	Inference*
1. Physical Tests:		
i. Nature		
ii. Colour		
iii. Odour	……..	……..
iv. Solubility		
a. Water	……..	……..
b. Ethanol	……..	……..
2. Identification Tests:		
i. Test A:		
	……..	……..
	……..	……..
ii. Test B:		
	…….	…….....
	…….	…….....
iii. Test C:		
	…….	……..
iv. Test D:	……	…….....
	……	……..
	……	……..

*If observation is as per given in the procedure, then write, "complies the test "; if not then write "does not comply the test".

8.0 Result:

The given sample of sodium chloride was found to complies the tests…..…… and does not comply the tests …. ……..….. for identification as per I.P.

9.0 Questions:

(Answer the following questions, Q.… Q…Q.…and Q.… Question Number to be allotted by the teacher)

1. Write the physical properties of sodium chloride.
2. Sodium chloride used as a tonicity agent, why?
3. Write two identification tests for sodium chloride.
4. What is the content of table salt?
5. Write the storage conditions for sodium chloride.
6. Write category of sodium chloride.
7. Write chemical formula and molecular weight of sodium chloride.
8. Why sodium chloride is best known as taste enhancer.
9. Mention the standard of dried sodium chloride given in I.P. 1996.

10. Write the procedure for preparation of isotonic solution of sodium chloride.
11. Write four pharmaceutical uses of sodium chloride.
12. Write the meaning of organoleptic properties.
13. Enumerate the use of electrolyte replenisher.

10.0 Reference:

Indian pharmacopoeia 1996.

(Space for answers)

(Space for answers)

Experiment No. 19

1.0 Title :

Identification test for sodium bicarbonate.

(To perform and report identification tests on given sample of sodium bicarbonateas per Indian Pharmacopoeia)

2.0 Prior Concepts:

Identification tests, Standards, Category, Solubility.

3.0 New Concepts: Monograph of sodium bicarbonate.

Proposition 1:

Chemical formula: - $NaHCO_3$

Molecular weight: - 84.01

Proposition 2: Description

It gives organoleptic properties of the given compound.

Nature: - Crystalline powder or small, opaque, mono clinic crystals.

Colour: - White.

Odour: - Odourless.

Proposition 3: Solubility

Statements of solubility's are indicated by a descriptive phrase and are intended to apply at 20°c to 30°c.

Sodium bicarbonate is freely soluble in water and practically insoluble in ethanol (95%).

The following table indicates the meaning of the terms used in statements of approximate solubility's.

Description Term	Approximate volume of solvent in milliliters per gram of solute
Very soluble	Less than 1
Freely soluble	From 1 to 10
Soluble	From 10 to 30
Sparingly soluble	From 30 to 100
Slightly soluble	From 100 to 1000
Very slightly soluble	From 1000 to 10,000
Insoluble or practically insoluble	More than10, 000

Proposition 4: Identification test.

This test is described to verify the article being examined in accordance with the label on the container. Failure of these test indicate that the article may be mislabeled or substituted.

Standards :- Sodium bicarbonate contains not less than 99.0 percent and not more than 101.0 percent of $NaHCO_3$.

Category :- Electrolyte replenisher; systemic alkaliser.

4.0 Learning Objectives:

4.1 Intellectual Skills:

1. To understand the concept of identification tests.

4.2 Motor Skills:

1. To identify the physical properties of given sample.
2. To observe the solubility in water and other solvents.
3. To observe the colour developed in test solution.

5.0 Apparatus :

5.1 Materials:

Test tubes, test tube stand, Graduated pipettes (0.5 ml, 1 ml, 10 ml)

5.2 Chemicals:

Sodium bicarbonate, 15% w/v potassium carbonate, potassium antimonate, dilute hydrochloric acid, solution, magnesium sulphate,(1M & 2M) acetic acid, phenolphthalein, magnesium uranyl acetate solution and barium hydroxide.

6.0 Stepwise procedure:

1. Procedure for Physical Tests:

Description:

Observe the given compound critically for the following descriptions. The compound is white crystalline powder or small, opaque, mono clinic crystals.

Solubility:

Perform solubility test in the different solvents. The compound is freely soluble in water and practically insoluble in ethanol 95%.

2. Procedure for Identification Tests:

A. Solutions of substance, when boiled liberate carbon dioxide.

B. Treat a solution of substance being examined with a solution of magnesium sulphate; no precipitate is formed (distinction from carbonates). Boil; awhite precipitate is formed.

C. Introduce into a test tube 0.1 g of substance being examined suspended in 2 ml of water. Add 2 ml of 2 M acetic acid, close the tube immediately using a stopper fitted with a glass tube bent at two right angles, heat gently and collect the gas in 5 ml of barium hydroxide solution; a white precipitate is formed that dissolves on addition of an excess of dilute hydrochloric acid.

D. Dissolve 0.1 g of substance being examined in 2 ml of water. Add 2 ml of 15% w/v solution of potassium carbonate and heat to boiling no precipitate is produced. Add 4 ml of freshly prepared potassium antimonate solution and heat to boiling. Allow to cool in ice and if necessary scratch the inside of test tube with glass rod; a dense, white precipitate is formed.

E. Acidify a solution of the substance being examined with 1 M acetic acid and add a large excess of magnesium uranyl acetate solution; a yellow, crystalline precipitate is formed.

7.0 Observation Table :

Test	Observation	Inference*
1. Physical Tests:		
i. Nature	.....	.....
ii. Colour	.....	.....
iii. Odour	.....	.....
iv. Solubility		
a. Water	.....	.....
b. Ethanol 95%	.....	.....
2. Identification Tests:		
i. Test A:	.....	.....
	.....	.....
ii. Test B:	.....	.....
	.....	.....
iii. Test C:	.....	.....
	.....	.....
iv. Test D:	.....	.....
	.....	.....
v. Test E:	.....	.....
	.....	.....

*If observation is as per given in the procedure, then write "complies the test "; if not then write "does not comply the test".

8.0 Result:

The given sample of sodium bicarbonate was found to complies the tests……..…… …….and does not comply the tests …. …….. ….. for identification as per I.P.

9.0 Questions:

(Answer the following questions, Q.... Q...Q....and Q.... Question Number to be allotted by the teacher)

1. Write the physical properties of sodium bicarbonate.
2. Write two identification tests for sodium bicarbonate.
3. How you will store the sodium bicarbonate.
4. Write category of sodium bicarbonate.
5. Write chemical formula and molecular weight of sodium bicarbonate.
6. Mention the standard of dried sodium bicarbonate given in I.P. 1996.
7. Find out two pharmaceutical uses of sodium bicarbonate.
8. Name the cation and anion formed after ionization of sodium chloride.
9. State the meaning of opaque and monoclinic.

10.0 Reference:

Indian pharmacopoeia 1996

(Space for answers)

(Space for answers)

Experiment No. 20

1.0 Title :

Identification test for magnesium sulphate.

(To perform and report identification tests on given sample of magnesium sulphate as per Indian Pharmacopoeia).

2.0 Prior Concepts:

Identification tests, Standards, Category, Solubility.

3.0 New Concepts: Monograph of magnesium sulphate

Proposition 1:

Chemical Formula: - $MgSO_4 . 7H_2O$

Molecular weight: - 246.47

Proposition 2: Description

It gives organoleptic properties of the given compound.

Nature: - Crystals or crystalline powder.

Colour: - Colourless or white.

Odour: - Odourless.

Proposition 3: Solubility

Statements of solubility's are indicated by a descriptive phrase and are intended to apply at 20° c to 307°c.

Magnesium sulphate is very soluble in boiling water; freely soluble in water; actically insoluble in ethanol (95%).

The following table indicates the meaning of the terms used in statements of approximate solubility's.

Description Term	Approximate volume of solvent in milliliters per gram of solute
Very soluble	Less than 1
Freely soluble	From 1 to 10
Soluble	From 10 to 30
Sparingly soluble	From 30 to 100
Slightly soluble	From 100 to 1000
Very slightly soluble	From 1000 to 10,000
Insoluble or practically insoluble	More than10, 000

Proposition 4: Identification test.

This test is described to verify the article being examined in accordance with the label on container. Failure of these test indicate that the article may be mislabeled or substituted.

Standards:- Magnesium sulphate contains not less than 99.0 percent and not more than 100.5 percent of $MgSO_4$, calculated with reference to the dried substances.

Category:- Osmotic laxative; used in treatment of electrolyte deficiency.

4.0 Learning Objectives:

4.1 Intellectual Skills:

1. To understand the concept of identification tests.

4.2 Motor Skills:

1. To identify the physical properties of given sample.
2. To observe the solubility in water and other solvents.
3. To observe the precipitate formed in test solution.

5.0 Apparatus :

5.1 Materials:

Test tubes, test tube stand, Graduated pipettes (0.5 ml, 1 ml & 10 ml)

5.2 Chemicals:

Magnesium sulphate, dilute hydrochloric acid, barium chloride, iodine solution, stannous chloride, dilute ammonia, lead acetate solution, sodium hydroxide, ammonium acetate solution, 2M ammonium chloride, 0.25M disodium hydrogen phosphate, 0.1% w/v titan yellow and 0.1 M sodium hydroxide solution.

6.0 Stepwise procedure:

1. Procedure for Physical Tests:

Description:

Observe the given compound critically for the following descriptions. The compound is colourless crystals or white, crystalline powder, odourless.

Solubility:

Perform solubility test in the different solvents. The compound is freely soluble in water; very soluble in boiling water and practically insoluble in ethanol 95%.

2. Procedure for Identification Tests:

A. Dissolve about 15 mg of the substance being examined in 2 ml of water. Add 1 ml of dilute ammonia solution; a white precipitate formed that is redissolved by adding 1 ml of 2 M ammonium chloride. Add 1 ml of 0.25 M disodium hydrogen phosphate; a white crystalline precipitate is formed.

B. To 0.5 ml of a neutral or slightly acid solution of the substance being examined add 0.2 ml of a 0.1% w/v solution of titan yellow and 0.5 ml of 0.1 M sodium hydroxide; a bright red turbidity develops which gradually settles to give a bright red precipitate.

C. Dissolve about 50 mg of the substance being examined in 5 ml of water. Add 1 ml of dilute hydrochloric acid and 1 ml of barium chloride solution; a white precipitate is formed.

D. Add 0.1 ml of iodine solution to the suspension obtained in test C; the suspension remains yellow (distinction from sulphites and dithionites) but is decolourised by adding, drop wise, stannous chloride solution (distinction from iodates). Boil the mixture; no colour precipitate is formed (distinction from selenates and tungstates).

E. Dissolve about 50 mg of the substance being examined in 5 ml of water. Add 2 ml of lead acetate solution; a white precipitate, soluble in ammonium acetate solution and in sodium hydroxide solution, is produced.

7.0 Observation Table :

Test	Observation	Inference*
1. Physical Tests:		
i. Nature	……..	……..
ii. Colour	……..	……..
iii. Odour	……..	……..
iv. Solubility		
a. Water	……..	……..
b. Ethanol	……..	……..
2. Identification Tests:		
i. Test A:	……..	……..
	……..	……..
ii. Test B:	……..	……..
	……..	……..
iii. Test C:	……..	……..
	……..	……..
iv. Test D:	…….. ……..	…….. ……..
v. Test E:	…….. ……..	…….. ……..

*If observation is as per given in the procedure, then write, "complies the test "; if not then write "does not comply the test".

8.0 Result:

The given sample of magnesium sulphate was found to complies the tests……..……..……. and does not comply the tests …. ……..……... for identification as per I.P.

9.0 Questions:

(Answer the following questions, Q.... Q...Q....and Q.... Question Number to be allotted by the teacher)

1. Write the observations of all physical tests for magnesium sulphate.
2. Write the solubility parameters for magnesium sulphate.
3. Which anions and cations are formed after ionization of magnesium sulphate.
4. Write two identification tests for magnesium and sulphate ions.
5. Write standards of magnesium sulphate on dried substance.
6. Write the indicator used to identify magnesium ion.
7. Write the category of magnesium sulphate.
8. Write four pharmaceutical applications of magnesium sulphate.
9. Write chemical formula and molecular weight of magnesium sulphate.
10. State the meaning of osmotic laxative.

10.0 Reference:

Indian pharmacopoeia 1996.

(Space for answers)

(Space for answers)

Experiment No. 21

1.0 Title :

Identification test for Ferrous Sulphate.

(To perform and report identification tests on given sample of ferrous sulphate as per Indian Pharmacopoeia).

2.0 Prior Concepts:

Identification tests, Standards, Category, Solubility.

3.0 New Concepts: Monograph of ferrous sulphate.

Proposition 1:

Chemical Formula: - $FeSO_4$, $7H_2O$

Molecular weight: - 278.01

Proposition 2: Description

It gives organoleptic properties of the given compound.

Nature: - Crystals or crystalline powder.

Colour: - Bluish Green or light green.

Odour: - Odourless

Proposition 3: Solubility

Statements of solubility's are indicated by a descriptive phrase and are intended toapply at 20° C to 30° C.

Ferrous sulphate is freely soluble in water; very soluble in boiling water; practically insoluble in ethanol (95%).

The following table indicates the meaning of the terms used in statements of approximate solubility's.

Description Term	Approximate volume of solvent in milliliters per gram of solute
Very soluble	Less than 1
Freely soluble	From 1 to 10
Soluble	From 10 to 30
Sparingly soluble	From 30 to 100
Slightly soluble	From 100 to 1000
Very slightly soluble	From 1000 to 10,000
Insoluble or practically insoluble	More than10, 000

(**Note:** It is efflorescent in air, on exposure to moist air, the crystals rapidly oxidize and become brown)

Proposition 4: Identification test.

This test is described to verify the article being examined in accordance with the label on container. Failure of these test indicate that the article may be mislabeled or substituted.

Standards: - Ferrous sulphate contains not less than 98.0 percent and not more than 105.0 percent of $FeSO_4$, $7H_2O$.

Category: - Haematinic.

Dose: - Prophylactic 300 mg; Therapeutic 600 to 900 mg.

4.0 Learning Objectives:

4.1 Intellectual Skills:

1. To understand the concept of identification tests.

4.2 Motor Skills:

1. To identify the physical properties of given sample.
2. To observe the solubility in water and other solvents.
3. To observe the efflorescence of compound.

5.0 Apparatus :

5.1 Materials:

Test tubes, test tube stand, Graduated pipettes (0.5 ml, 1 ml & 10 ml)

5.2 Chemicals:

Dilute hydrochloric acid, barium chloride, iodine solution, stannous chloride solution, dilute sulphuric acid, 0.1 % w/v 1,10 phenanthroline and 0.1M ceric ammonium sulphate, potassium ferricyanide, dilute hydrochloric acid, sodium hydroxide, potassium ferrocyanide.

6.0 Stepwise procedure:

1. Procedure for Physical Tests:

Description:

Observe the given compound critically for the following description. The compound is bluish green crystals or light green, crystalline powder, odourless.

Solubility:

Perform solubility test in the different solvents. The compound is freely soluble in water; very soluble in boiling water and practically insoluble in ethanol 95%.

2. Procedure for Identification Tests:

A. Dissolve 0.5 g of substance being examined in 2 ml of water(equivalent to about 10 mg of iron).Add 2 ml of dilute sulphuric acid and 1 ml of a 0.1 % w/v solution of 1, 10-phenonthroline; and intense red colour which discharged by addition of a slight excess of 0.1 M ceric ammonium sulphate is produced.

B. To 1 ml of a solution containing not less than 1 mg of iron. Add 1 ml of potassium ferricyanide solution: a dark blue precipitate is formed that is insoluble in dilute hydrochloric acid and is decomposed by sodium hydroxide solution.

C. To 1 ml of solution containing not less than 1 mg of iron. Add 1 ml of potassium ferrocyanide solution; a white precipitate is formed which rapidly becomes blue and is insoluble in dilute hydrochloric acid.

D. Dissolve about 50 mg of the substance being examined in 5 ml of water. Add 1 ml of dilute hydrochloric acid and 1 ml of barium chloride solution; a white precipitate is formed.

E. Add 0.1 ml of iodine solution to the suspension obtained in test C; the suspension remains yellow (distinction from sulphites and dithionites) but is decolourised by adding, drop wise, stannous chloride solution (distinction from iodates). Boil the mixture; no coloured precipitate is formed (distinction from selenates and tungstates).

F. Dissolve about 50 mg of the substance being examined in 5 ml of water. Add 2 ml of led acetate solution; a white precipitate, soluble in ammonium acetate solution and in sodium hydroxide solution, is produced.

7.0 Observation Table :

Test	Observation	Inference*
1. Physical Tests:		
i. Nature	.....	.....
ii. Colour	.....	.....
iii. Odour	.....	.....
iv. Solubility	.....	.....
a. Water	.....	.....
b. Ethanol 95%		
Test	**Observation**	**Inference***
2. Identification Tests:		
i. Test A:	.....	.....
	.....	.....
ii. Test B:	.....	.....
	.....	.....
iii. Test C:	.....	.....
	.....	.....
iv. Test D:	.....	.....
	.....	.....
v. Test E:	.....	.....
	.....	.....
vi. Test F:		
	.....	.....
	.....	.....

*If observation is as per given in the procedure, then write, "complies the test "; if not then write "does not comply the test".

8.0 Result:

The given sample of ferrous sulphate was found to complies the tests. ……..…… and does not comply the tests …. …….. for identification as per I.P.

9.0 Questions:

(Answer the following questions, Q.... Q...Q....and Q.... Question Number to be allotted by the teacher)

1. Write the observations of all physical tests for ferrous sulphate.
2. Write the solubility parameters of ferrous sulphate.
3. Which anions and cations are formed after ionization of ferrous sulphate?
4. Write two identification tests for ferrous salt and sulphate ions.
5. Write the standards of ferrous sulphate.
6. Write the category of ferrous sulphate.
7. Write two pharmaceutical applications of ferrous sulphate.
8. Write the chemical formula and molecular weight of ferrous sulphate.
9. State the meaning of following terms.
   i. Prophylactic
   ii. Therapeutic
   iii. Efflorescent
   iv. Haematinic
10. Write two reagents used to identify ferrous salt.

10.0 Reference:

Indian pharmacopoeia 1996.

(Space for answers)

(Space for answers)

Experiment No. 22

1.0 Title :

Identification test for sodium acetate.

(To perform and report identification tests on given sample of sodium acetate as per Indian Pharmacopoeia).

2.0 Prior Concepts:

Identification tests, Standards, Category, Solubility.

3.0 New Concepts: Monograph of sodium acetate

Proposition 1:

Chemical Formula: - $C_2H_3NaO_2,3H_2O$ (CH_3 COONa,$3H_2O$)

Molecular weight: - 136.08

Proposition 2: Description

It gives organoleptic properties of the given compound.

Nature: - Crystals or crystalline powder.

Colour: - Colourless or white.

Odour: - Odourless.

Proposition 3: Solubility

Statements of solubility's are indicated by a descriptive phrase and are intended to apply at 20° C to 30°C.

Sodium acetate is very soluble in water and soluble in ethanol (95%).

The following table indicates the meaning of the terms used in statements of approximate solubility's.

Description Term	Approximate volume of solvent in milliliters per gram of solute
Very soluble	Less than 1
Freely soluble	From 1 to 10
Soluble	From 10 to 30
Sparingly soluble	From 30 to 100
Slightly soluble	From 100 to 1000
Very slightly soluble	From 1000 to 10,000
Insoluble or practically insoluble	More than10, 000

Proposition 4: Identification test.

This test is described to verify the article being examined in accordance with the label on container. Failure of these test indicate that the article may be mislabeled or substituted.

Standards: - Sodium acetate contains not less than 99.0 percent and not more than 101.0 percent of $C_2H_3NaO_2$,$3H_2O$, calculated with reference to the dried substance.

Category:- Pharmaceutical aid (for peritoneal dialysis fluid)

4.0 Learning Objectives:

4.1 Intellectual Skills:

1. To understand the concept of identification tests.

4.2 Motor Skills:

1. To identify the physical properties of given sample.
2. To observe the solubility in water and other solvents.
3. To observe the colour developed in test solution.

5.0 Apparatus :

5.1 Materials:

Test tubes, test tube stand, graduated pipettes (0.5 ml, 1 ml & 10 ml)

5.2 Chemicals:

Sodium acetate, 15% w/v potassium carbonate, potassium antimonate solution, sulphuric acid, 1 M acetic acid, magnesium uranyl acetate solution, ethanol 95%, oxalic acid and dilute ammonia solution.

6.0 Stepwise procedure:

1. Procedure for Physical Tests:

Description:

Observe the given compound critically for the following description. The compound is colourless crystals or white, crystalline powder, odourless.

Solubility:

Perform solubility tests in the different solvents. The compound is very soluble in water and soluble in ethanol 95%.

2. Procedure for Identification Tests:

A. Dissolve 0.1 g of substance being examined in 2 ml of water. Add 2 ml of 15% w/v solution of potassium carbonate and heat to boiling no precipitate is produced. Add 4 ml of freshly prepared potassium antimonate solution and heat to boiling. Allow to cool in ice and if necessary scratch the inside of test tube with glass rod; a dense, white precipitate is formed.

B. Acidify a solution of the substance being examined with 1 M acetic acid and add a large excess of magnesium uranyl acetate solution; a yellow, crystalline precipitate is formed.

C. Heat the substance being examined with an equal quantity of oxalic acid; acidic vapours with the characteristic odour of acetic acid are liberated.

D. Warm 1 g of substance being examined with 1 ml of sulphuric acid and 3 ml of ethanol (95%); ethyl acetate, recognizable by its odour, is evolved.

E. Dissolve about 30 mg of the substance being examined in 3 ml of water; add successively 0.25 ml of dilute ammonia solution. Heat carefully to boiling; within a few minutes a blue precipitate or dark blue colour is produced.

7.0 Observation Table :

Test	Observation	Inference*
1. Physical Tests:		
i. Nature	.....	.....
ii. Colour	.....	.....
iii. Odour	.....	.....
iv. Solubility	.....	.....
a. Water	.....	.....
b. Ethanol 95%		
2. Identification Tests:		
i. Test A:	.....	.....
	.....	.....
ii. Test B:	.....	.....
	.....	.....
iii. Test C:	.....	.....
	.....	.....
iv. Test D:	.....	.....
	.....	.....
v. Test E:	.....	.....
	.....	.....

*If observation is as per given in the procedure, then write, "complies the test "; if not then write, "does not comply the test".

8.0 Result:

The given sample of sodium acetate was found to complies the tests..... ..... and does not comply the tests .... for identification as per I.P.

9.0 Questions:

(Answer the following questions, Q.... Q...Q....and Q.... Question Number to be allotted by the teacher)

1. Write the Physical properties of sodium acetate.
2. Write two identification tests for sodium acetate.
3. Write the storage conditions of sodium acetate.

4. Write category of sodium acetate.
5. Write chemical formula and molecular weight of sodium acetate.
6. Mention the standards of dried sodium acetate given in I.P. 1996.
7. Write two pharmaceutical uses of sodium acetate.
8. State the meaning of peritoneal dialysis.

10.0 Reference:

Indian pharmacopoeia 1996.

(Space for answers)

(Space for answers)

Experiment No. 23

1.0 Title :

Identification test for hydrogen peroxide.

(To perform and report identification tests on given sample of hydrogen peroxide asper Indian Pharmacopoeia).

2.0 Prior Concepts:

Identification tests, Category, Standards.

3.0 New Concepts: Monograph of sodium hydroxide.

Proposition 1:

Chemical Formula: - H_2O_2

Strength of Solution: - 6% w/v

Proposition 2: Description

It gives organoleptic properties of the given compound.

Nature: - Clear liquid.

Colour: - Colourless.

Odour: - Odourless.

Proposition 3: Identification test.

This test is described to verify the article being examined in accordance with the label on container. Failure of these test indicate that the article may be mislabeled or substituted.

Standards: - Hydrogen peroxide solution (20 Vol) contains not less than 5.0 percent w/v not more than 7.0 percent w/v H_2O_2, corresponding to about 20 times its volume of available oxygen.

Category: - Antiseptic and deodorant.

(**Note: -** It decomposes vigorously in contact with oxidisable organic matter and with certain metals and also it allows becoming alkaline.)

4.0 Learning Objectives:

4.1 Intellectual Skills:

1. To understand the concept of identification tests.
2. To understand the chemical reactions of sample.

4.2 Motor Skills:

1. To observe the evolution of gas from test solution.
2. To observe the colour change in the test solution.

5.0 Apparatus :

5.1 Materials:

Test tubes, test tube stand, graduated Pipettes (0.5 ml, 1 ml & 10 ml)

5.2 Chemicals:

20 V hydrogen peroxide, 1 M sulphuric acid, 0.02 M potassium permanganate, potassium chromate and ether.

6.0 Stepwise procedure:

1. Procedure for Physical Tests:

Description:

Observe the given compound critically for the following descriptions. The compound is clear, colourless liquid; odourless.

Solubility:

Perform solubility tests in the different solvents. The compound is very soluble in water and soluble in ethanol 95%.

2. Procedure for Identification Tests:

A. To 1 ml add 0.2 ml of 1M sulphuric acid and 0.25 ml of 0.02 M potassium permanganate; the solution becomes colourless with evolution of gas.

B. Shake 0.05 ml with 2 ml of 1M sulphuric acid, 2 ml ether and 0.05 ml of potassium chromate solution; the ether layer becomes blue.

7.0 Observation Table :

Test	Observation	Inference*
1. Physical Tests:		
i. Nature	.....	.....
ii. Colour	.....	.....
iii. Odour	.....	.....
2. Identification Tests:		
i. Test A:	.....	.....
	.....	.....
ii. Test B:	.....	.....
	.....	.....

*If observation is as per given in the procedure, then write "complies the test "; if not then write "does not comply the test".

8.0 Result:

The given sample of hydrogen peroxide was found to complies the tests…. …..... and does not comply the tests ……. …… ….. for identification as per I.P.

9.0 Questions:

(Answer the following questions, Q.... Q...Q....and Q.... Question Number to be allotted by the teacher)

1. State the meaning of identification tests.
2. State the meaning of 20 vol of hydrogen peroxide.
3. Which property of hydrogen peroxide decolorized the potassium permanganate solution.
4. Why hydrogen peroxide is used for dressing of wounds and cuts.
5. Why hydrogen peroxide is stored at cool place or below 15 0C.
6. Write the I.P. standards of hydrogen peroxide.
7. Why hydrogen hydroxide should not stored for longer period of time.
8. Write the category of hydrogen peroxide.
9. Write four marketed preparations of hydrogen peroxide.
10. Why hydrogen peroxide is stored in plastic containers.
11. Write two identification tests of hydrogen peroxide.
12. Write the labeling instructions for hydrogen peroxide.
13. State the meaning of aseptic and deodorant action.

10.0 Reference:

Indian pharmacopoeia 1996.

(Space for answers)

(Space for answers)

Experiment No. 24

1.0 Title :

Identification test for boric acid.

(To perform and report identification tests on given sample of boric acid as per Indian Pharmacopoeia).

2.0 Prior Concepts:

Identification tests, Standards, Category, Solubility.

3.0 New Concepts: Monograph of Boric acid

Proposition 1:

Chemical Formula: - H_3BO_3

Molecular weight: - 61.83

Proposition 2: Description

It gives organoleptic properties of the given compound.

Nature: - Crystalline powder, shiny plates, unctuous to the touch.

Colour: - White or colourless.

Odour: - Odourless.

Proposition 3: Solubility

Statements of solubility's are indicated by a descriptive phrase and are intended toapply at 20° C to 30°C.

Boric acid is soluble in water and in ethanol (95%); freely soluble in boiling water, in boiling ethanol (95%) and in glycerine.

The following table indicates the meaning of the terms used in statements of approximate solubility's.

Description Term	Approximate volume of solvent in milliliters per gram of solute
Very soluble	Less than 1
Freely soluble	From 1 to 10
Soluble	From 10 to 30
Sparingly soluble	From 30 to 100
Slightly soluble	From 100 to 1000
Very slightly soluble	From 1000 to 10,000
Insoluble or practically insoluble	More than10, 000

Proposition 4: Identification test.

This test is described to verify the article being examined in accordance with the label on container. Failure of these test indicate that the article may be mislabeled or substituted.

Standards:- Boric acid contains not less than 99.5 per cent and not more than 100.5 per cent of H_3BO_3, calculated with reference to the dried substance.

Category:- Local anti-infective.

4.0 Learning Objectives:

4.1 Intellectual Skills:

1. To understand the concept of identification tests.

4.2 Motor Skills:

1. To identify the physical properties of given sample.
2. To observe the solubility in water and other solvents.
3. To observe the colour change in flame of test.

5.0 Apparatus :

5.1 Materials:

Test tubes, test tube stand, graduated pipettes (0.5 ml, 1 ml & 10 ml)

5.2 Chemicals:

Boric acid, methanol (95%), glycerin, ethanol 95% and sulphuric acid.

6.0 Stepwise procedure:

1. Procedure for Physical Tests:

Description:

Observe the given compound critically for the following description. The compound is white, crystalline powder colourless shiny plates unctuous to touch or white crystals; odourless.

Solubility:

Perform solubility test in the different solvents. The compound is soluble in water and in ethanol (95%); freely soluble in boiling water, in boiling ethanol (95%) and in glycerin.

2. Procedure for Identification Tests:

A. Dissolve 0.1 g by gentle warming with 5 ml of methanol to which a few drops of sulphuric acid have been added. Ignite the solution; the flame has green border.

B. Dissolve 3.0 g in 90 ml of boiling water, cool; the solution is faintly acidic to litmus(pH 3.8 to 4.8)

7.0 Observation Table :

Test	Observation	Inference
1. Physical Tests:		
i. Nature	……...	……..…..….
ii. Colour	……..	……..……...
iii. Odour	……..	……..……...
iv. Solubility	……..	
a. Water		……..……...
b. Ethanol (95%).	……..	……..……...
c. Boiling water.	……..	……..……...
d. Boiling ethanol (95%)	……	……..……...
2. Identification Tests:		
i. Test A:	……	…….
	……	…….
ii. Test B:	……	…….
	.....	…….

*If observation is as per given in the procedure, then write "complies the test "; if not then write "does not comply the test".

8.0 Result:

The given sample of boric acid was found to complies the tests…...……..…….. and does not comply the tests …. …… for Identification as per I.P.

9.0 Questions:

(Answer the following questions, Q.… Q…Q.…and Q.… Question Number to be allotted by the teacher)

1. Write the properties of boric acid.
2. Write two synonyms of boric acid.
3. Why boric acid is not used internally.
4. Write two identification tests of boric acid.
5. Write category of boric acid.
6. How you check the solubility of boric acid in following solvents:

a. Water

b. Boiling water

c. Glycerine

d. Ethanol 95%

e. Boiling ethanol 95%.

7. Whether boric acid is weak acid or strong acid.

8. State the meaning of unctuous to touch.

9. Write the standards of dried H_3BO_3 given in I.P. 1996.

10. Write chemical formula and molecular weight of boric acid.

11. Write four pharmaceutical uses of boric acid.

12. State the meaning of ignites.

13. State the meaning of antiinfective.

10.0 Reference:

Indian pharmacopoeia 1996.

(Space for answers)

(Space for answers)

Experiment No. 25

1.0 Title :

Identification test for Ammonium chloride.

(To perform and report identification tests on given sample of Ammonium chloride as per Indian Pharmacopoeia).

2.0 Prior Concepts:

Identification tests, Standards, Category, Solubility.

3.0 New Concepts: Monograph of ammonium chloride

Proposition 1:

Chemical Formula: - NH_4Cl

Molecular weight: - 53.44

Proposition 2: Description

It gives organoleptic properties of the given compound.

Nature: - Crystals or Crystalline powder.

Colour: - Colourless or white.

Odour: - Odourless.

Proposition 3: Solubility

Statements of solubility's are indicated by a descriptive phrase and are intended to apply at 20° C to 30°C.

Ammonium chloride is freely soluble in water; sparingly soluble in ethanol (95%).

The following table indicates the meaning of the terms used in statements of approximate solubility's.

Description Term	Approximate volume of solvent in milliliters per gram of solute
Very soluble	Less than 1
Freely soluble	From 1 to 10
Soluble	From 10 to 30
Sparingly soluble	From 30 to 100
Slightly soluble	From 100 to 1000
Very slightly soluble	From 1000 to 10,000
Insoluble or practically insoluble	More than10, 000

Proposition 4: Identification test.

This test is described to verify the article being examined in accordance with the label on container. Failure of these test indicate that the article may be mislabeled or substituted.

Standards:- Ammonium chloride contains not less than 99.0 percent and not more than 100.5 percent of NH_4Cl, calculated with reference to the dried substances.

Category:- Expectorant; diuretic; systemic acidifier.

4.0 Learning Objectives:

4.1 Intellectual Skills:

1. To understand the concept of identification tests.

4.2 Motor Skills:

1. To identify the physical properties of given sample.
2. To observe the solubility in water and other solvents.
3. To observe the colour developed in test solution.

5.0 Apparatus :

5.1 Materials:

Test tubes, test tube stand, Graduated pipette (0.5 ml, 1 ml & 10 ml) and litmus paper.

5.2 Chemicals:

Ammonium chloride, sodium hydroxide, light magnesium oxide, 0.1M hydrochloric acid, methyl red solution and freshly prepared 10% w/v solution of sodium cobaltinitrite, diphenylcarbazide solution, dilute ammonia solution, dilute nitric acid, potassium dichromate and sulphuric acid

6.0 Stepwise procedure:

1. Procedure for Physical Tests:

Description:

Observe the given compound critically for the following description. The compound is colourless crystals or white, crystalline powder, odourless.

Solubility:

Perform solubility tests in the different solvents. The compound is freely soluble in water and sparingly soluble in ethanol (95%).

2. Procedure for Identification Tests:

A. Heat the few mg of substance being examined with sodium hydroxide solution; ammonia is evolved, which is recognizable by its odour and by its action on moist red litmus paper, which turns blue.

B. To the solution of ammonium chloride add 0.2 g of light magnesium oxide. Pass a current of air through the mixture and direct the gas that is evolved to just beneath the surface of mixture of 1 ml of 0.1 M hydrochloric acid and 0.05 ml of methyl red solution; the colour of solution changes to yellow. On addition of 1 ml of a freshly prepared 10% w/v solution of sodium cobaltinitrite, a yellow colour precipitate is formed.

C. Dissolve 0.1 g of the substance being examined (equivalent to about 2 mg of chloride ion), in 2 ml of water. Acidify with dilute nitric acid, and add 0.5 ml of silver nitrate solution, shake and allow to stand; A curdy, white precipitate formed, which is insoluble in dilute nitric acid but soluble, after being well washed with water, in dilute ammonia solution, from which it is reprecipitated by the addition of dilute nitric acid.

D. Introduce into a test tube 0.5 g of the substance being examined (equivalent to about 10 mg of chloride ion), add 0.2 g of potassium dichromate and 1 ml of sulphuric acid. Place a filter paper strip moistened with 0.1 ml of diphenylcarbazide solution over the mouth of test tube; The paper turns violet red. (Do not bring the moistened paper into contact with the potassium dichromate solution).

7.0 Observation Table :

Test	Observation	Inference*
1. Physical Tests:		
i. Nature	.....	.....
ii. Colour	.....	.....
iii. Odour	.....	.....
iv. Solubility	.....	.....
a. Water	.....	.....
b. Ethanol	.....	.....
2. Identification Tests:		
i. Test A:	.....	.....
	.....	.....
ii. Test B:	.....	.....
	.....	.....
iii. Test C:	.....	.....
	.....	.....
iv. Test D:	.....	.....
	.....	.....

*If observation is as per given in the procedure, then write, "complies the test "; if not then write "does not comply the test".

8.0 Result:

The given sample of ammonium chloride was found to complies the tests…….. …..... and does not comply the tests…….. for Identification as per I.P.

9.0 Questions:

(Answer the following questions, Q.... Q...Q....and Q.... Question Number to be allotted by the teacher)

1. Write the Physical properties of ammonium chloride.
2. Write two identification tests for ammonium chloride.
3. Write the storage condition for ammonium chloride.
4. Write category of ammonium chloride.
5. Write chemical formula and molecular weight of ammonium chloride.
6. Write the standard of dried ammonium chloride given in I.P. 1996.
7. Write four pharmaceutical uses of ammonium chloride.

8. State the meaning of following:
   i. Expectorant
   ii. Diuretic
   iii. Systemic acidifier

10.0 Reference:

Indian pharmacopoeia 1996.

(Space for answers)

(Space for answers)

MAHARASHTRA STATE BOARD OF TECHNICAL EDUCATION

PATTERN OF ANNUAL PRACTICAL EXAMINATION
(Specimen Question Paper)

Sub: Pharmaceutical chemistry-I **Max. Marks:** 80

Class: F.Y.D.Pharm **Time:** 3 hrs.

Q.1. Write synopsis on the following. 20

(Details are given on the next page)

Q.2. Major experiment 30

a. Standardize the given sample of…….....

b. Find out the amount of .....……..... present in the given sample.

(That is % w/v of ……..)

Q.3. Minor experiment 20

Carry out the limit test for .....……..… …….given sample bearing your Table no and state whether it complies with I.P. limit or not.

OR

Identification tests for given sample as per I.P.

Q.4. Viva voce. 10

80 marks

Detail of Question No. 1 (Synopsis)

a. One:" question bit" shall be based on the principle involved in the assay of inorganic compound. 4

Viz. out line the principle involved in the assay of ferrous sulphate.

b. One " question bit" shall be based on the reactions involved in the assay of inorganic compound 4

Viz: - Give the reactions involved in the assay of ammonium chloride I.P. 66 or I.P. 96.

c. One "question bit" shall be based on limit test. 4

Viz. Write in brief the principle and reactions involved in the limit test for Sulphate I.P. 85.

d. One "question bit" shall be based on standardization. 4

Viz: state the procedure for standardization of 0.1 N potassium permanganate.

e. One "question bit" shall be based on factor calculation 4

Viz: Calculate the factor used in the assay of sodium bicarbonate.
(Given: - Atomic weights- Na-23, H-1, C-12 & O-16

OR

e. One: "question bit" may be based on medicinal use/uses of Inorganic compounds.

Viz. Mention the medicinal uses of: -

i. Calcium Gluconate

ii. Boric Acid.

iii. Copper Sulphate.

Detail of Question No. 2 (Major Experiment)

Viz. Determine the % w/v of $FeSO_4.7H_2O$ present in the given Ferrous sulphate sample solution (A).

Procedure: To the given sample solution (A), add 10 ml of dilute sulphuric acid and titrate with standardized potassium permanganate solution.

Factor:- Each ml of 0.1 N Potassium permanganate solution ≡ 0.0278 g of $FeSO_4.7H_2O$ above experiment is divided into three parts for the distribution of Marks.

Part 1st 5

Standardization of given potassium permanganate solution.
(Deduct 1 mark for the fluctuation of normality by +/- 0.01N)

Part 2nd Determination of sample Reading 20
(Deduct 1 marks for the fluctuation of reading by +/- 0.2 ml)

Part 3rd Calculations 5

30 Marks

Detail of Question No. 3 (Minor Experiment)

Viz:- Carry out the Limit Test for Sulphate I.P. 96, on the given sample (I) and state whether it complies the test or not? 20

* After performing the above Limit test on the given sample (I) student will report whether the sample (I) complies the Limit test or not?

OR

Q.3. Identification Tests as per I.P.

Detail of question No. 4

The external examiner shall take viva on the experiments performed in complete academic year and some question on theory related to the experiments performed.

Q.4. Viva 10

List of Laboratory Manuals Developed by MSBTE For Diploma In Pharmacy

First Year

1.	Pharmaceutics - I	(0805)
2.	Pharmaceutical Chemistry - I	(0806)
3.	Pharmacognosy	(0807)
4.	Biochemistry and Clinical Pathology	(0808)
5.	Human Anatomy and Physiology	(0809)

Second Year

1.	Pharmaceutics - II	(0811)
2.	Pharmaceutical Chemistry - II	(0812)
3.	Pharmacology and Toxicology	(0813)
4.	Hospital and Clinical Pharmacy	(0816)

PHARMACIST'S OATH

- I swear by the Code of Ethics of Pharmacy Council of India in relation to the community and shall act as an integral part of health care team.
- I shall uphold the laws and standards governing my profession.
- I shall strive to perfect and enlarge my knowledge to contribute to the advancement of pharmacy and public health.
- I shall follow the system, which I consider best for pharmaceutical care and counseling of patient.
- I shall endeavour to discover and manufacture drugs of quality to alleviate sufferings of humanity.
- I shall hold in confidence the knowledge gained about the patients in connection with my professional practice and never divulge unless compelled to do so by the law.
- I shall associate with organizations having their objectives for betterment of Profession of Pharmacy and make contribution to carry out the work of those organizations.
- While I continue to keep this oath unviolated, may it be granted to me to enjoy life and practice of pharmacy respected by all, at all times!
- Should I trespass and violate this oath, may the reverse be my lot!

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