Date 27/11/20

Expt. No. 1 Page No. 1

Aim :-

To study the various apparatus / equipment used in the laboratory scale.

Reference :-

"Ali Mohammad", A text book of practical Pharmaceutical chemistry Edition - Ist, 1995 Published by CBS publishers and distributors.

Theary :-

1. Digital balance machine :-

The Digital balance used in the general chemistry Lab. It is very sensitive istrument used For the weighing substance to the mg Lable.

2. Beakar :-

A Beakar is generally a cylindiral container with a flat bootam. Most also have a small spout to aid pouring. Bikars are avialable in a wide range of sizes from 1 mm up to several liters.

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3. Conical Flask:-

The Flask has a conical base that almost extends into small cylindirical Neck. It is also non as titration Flask. It is also used to mixing the chemical liquids and measured.

4. Pippete :-

A Pippete is a laboratory tools. Commonly used in chemistry, Biology, medicine to transport a measured volume of liquids.

5. Burner:-

A Burner named often refered burner. It is a common piece of laboratory equipment that produce gas flame which is used for heating.

6. Filter paper :-

Filter paper is a semi-permeable paper barrier placed perpendicular to a liquid or air flow. It is used to separate or filtrate the fine substances from liquids.

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Expt. No. Page No. 3

7. Measuring Cylinder :-

A measuring cylinder is a piece of laboratory glass ware. It is used to measure the volume of liquids. It is used to accurately measure the value of chemical for use in reactions.

8. Test Tube :-

A Test Tube is a glass or plastic container that is much longer. It is a common piece of laboratory glass ware constisting of a finger light length of glass or clear plastic tubing, plastic tubing open at the top and closed at the bottam.

9. Test Tube Holder :-

A test tube holder is a used to Hold test tubes. It is Used for Holding a test tube in place when ithe tube is hot or should not be touched.

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10. Analytical balance :-

A Analytical balance is a class of balance designed to measure small malls in the sub mg range. The measuring span of a analytical balance is incide a transeparent one closer with doors so that dust does not collect and so any air currents in the room do not effect the balance operation.

11. Tripod stand :-

Tripod is a portable three leged platform equpment it is usually made of light weight metal such as steinless steel and Alluminium. It can be moved with in the Lab.

Result :-

The verious apparatus / equpment used in the laboratory was studied.

Teacher's Signature _____

Observations :-

Sr. No.	Identification test	observation	Inference
1.	Identification test for sodium ion :- 2 ml sample + 2 ml of 15% Potassium carbonate solution + 4 ml freshly prepared antimonite solution. Boil and then cool.	White PPT	Sodium ion present.
2.	1 ml sample + 1 ml acetic acid + excess of magnesium uranyl acetate solution.	Yellow crystal	Sodium ion present.
3	Identification test for chloride ion :- 3 ml sample + 1 ml dilute nitric acid + few drops of silver nitrate solution	white PPT	Chloride ion present
4.	1 ml sample + 0.2 g Potassium dichromate + 1 ml sulphuric acid	White PPT	Chloride ion present.

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Expt. No. 2

Page No. 5

Aim :-
To perform identification test for sodium chloride.

Reference :-
"Gupta G.D and Sharma Shailesh", A practical manual of pharmaceutical chemistry I. Edition - IInd 2017 Aug. Published by Nirali prakashan Pune. P. No 67.

Requirements :-

1. Apparatus :-
Test Tube, test tube Holder, Burner, filter paper, glass rod, Funel.

2. Chemical :-
Sodium chloride, Potassium carbonate, Antimonate solution, Ammonia solution, Sulphuric acid, Potassium di chromate, di phenyl carbazide solution.

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5	2 ml sample + 2 ml of 2M acetic acid solution. Close the tube with bent at right angles. Heat gently and collect the gas in 5 ml barium hydroxide solution. Place filter paper strip moistened with 0.1 ml of diphenylcarbazide soln over the mouth of the test-tube	Paper turned violet-red	Chloride ion present

Date

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Theory :-

These identification test are generally based on reactions of anions and cations present in the drug molecules. On the basis of anions and cations present, the quality of drug is established.

Sodium chloride is used as an electrolyte replenisher and pharmaceutical aid.

Procedure :-

Prepare a solution of sample by dissolving 1 g in 20 ml water.

Indentification Taste for Sodium ion :-

1. Take 2 ml sample solution and add 2 ml of 15% w/v solution of potassium carbonate. Heat to the boil. No precipitate formed. Add 4 ml of freshly prepared Antimonite solution and heat to boil.

Than cool in ice bath and Rub the inside wall of the test tube with glass rod.

A dense white precipitate of sodium antimonide is formed.

$KSbO_3 + Na^+ \longrightarrow NaSbO_3 + K^+$

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Acedefy 1 ml of sample solution with acetic acid Add excess of magnesium uranyl Acetated solution.

Yellow crystalline precipetate of sodium magnicium uranyl acitate is formed.

$$Na^+ + Mg^{2+} + 3UO_2^- + 9CH_3COO^- \longrightarrow NaMg(UO_2)_3(CH_3COO)_9$$

Indentification taste for chlorine ion:-

1. Take 1 ml of diulte HNO_3 and add 3 ml of sample solution.
   Add few drops of silver nitrate solution.
   white precipitate of silver chloride is formed.
   Add diulte ammonia, the precipitate is disolveNed due to formation of complex.

$$NaCl + AgNO_3 \longrightarrow AgCl + NaNO_3$$

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2. Take small Quantity of sample solution in a test tube.
Add 0.2 g of potassium dichromate and 2 ml of H_2SO_4. place a filter paper striap moistened with 0.1 ml of di phenyl carbazied solution over the mouth of the test tube the paper turns violet red.

Result:-
The given sample is identified as sodium chloride.

Observations :-

Sr No	Identification test	Observation	Inference
1	Identification test for ammonium ion :- Sample + Sodium hydroxide soln	Smell of ammonia	Ammonium ion Present
2.	Sample + Sodium hydroxide solution. Red litmus paper at the mouth of test tube	Red litmus paper turns blue	Ammonium ion present.
3.	Sodium cobalt nitrate test :- 2ml sample + 2ml of 10% Sodium cobalt nitrate	Yellow PPT	Ammonium Chloride Present
4.	2ml sample + 0.2g light MgO → Generation of air → This air is passed through 0.1N HCl + 0.05 ml methylene red solution	Yellow colour solution	Ammonium chloride present
5	Identification test for chloride ion :- 3ml sample + 1ml dilute HNO_3 + 2 drops of Silver nitrate solution	White PPT	Chloride ion present

Date

Expt. No. 3 Page No. 9

Aim :-
To perform Identification test of ammonium chloride (NH_4Cl).

Reference :- "Gupta G.D and Sharma Shailesh". A practical manual of pharmaceutical chemistry I. Published by Nirali prakashan. Edition I, Aug 2017, P.No. 47.

Requirement :-

(i) Apparatus :-
Test tube, Test tube holder, Filter paper, Burner, Glass rod, Funnel, Beaker.

(ii) Chemical :-
NH_4Cl, HNO_3, NaOH, HCl, $AgNO_3$, NH_3, H_2SO_4, potassium dichromate, Diphenyl carbazide.

Theory :-
Identification test are performed to determine the quality of drug so that drugs can be used safely. these identification test are generally based on reaction of anions (-) and cations (+)

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6.	1 ml sample + 0.2 g Potassium dichromate + 1 ml H_2SO_4 + 2 drops of silver nitrate solution. Place filter paper moistened with 0.1 ml of diphenyl carbazide solution	Paper turns Violet - red in colour	chloride ion present.

Date

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present in the drug molecule.

→ Ammonium chloride is a odourless, colourless or white crystalline powder with Saline taste. (नमकीन)

→ It is soluble in water and slightly soluble in ethanol. It is used as an expectorant, diuretics.

Procedure :-

Identification test for ammonium ion :-

1. Heat a small amount of NH_4Cl solution and add the NaOH solution.

$$NH_4Cl + NaOH \longrightarrow NH_3\uparrow + H_2O + NaCl$$

2. Take the solution of NH_4Cl and add few drops of 10% w/v cobalt nitrate solution Yellow P.P.T of ammonium cobalt nitrate.

$$3NH_4Cl + Na_3[Co(NO_2)_6] \longrightarrow$$

Sodium cobalt Nitrate

$$(NH_4)_3[Co(NO_2)_6] + NH_4NO_3$$

ammonium cobalt nitrate — ammonium Nitrate.

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Date

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Identification test for chloride ion :-

1. Take 3ml of NH_4Cl solution and add 1ml of dilute HNO_3 solution and add few drops of silver nitrate ($AgNO_3$) solution white P.P.T of Silver nitrate is formed on addition of ammonia solution the P.P.T is dissolved due to formation of complex.

$$NH_4Cl + AgNO_3 \longrightarrow AgCl + NH_4NO_3$$

2. Add 0.2gm of potassium dichromate and 1ml of H_2SO_4 to a small quantity of NH_4Cl in a test tube.
   Place a filter paper strip moistened with 0.1ml of diphenyl carbazied solution over the mouth of test tube.
   The paper turns violet red.

Result :-
The given sample is identified as ammonium chloride.

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- **Observations :**

Sr. No.	Identification test	Observation	Inference
1.	**Identification test for sodium ion :** 2 ml sample + 2 ml of 15% potassium carbonate solution + 4 ml freshly prepared antimonite solution. Boil and then cool.	White precipitate	**Sodium ion** ☑ Present ☐ Absent
2.	**Identification test for sodium ion :** 1 ml sample + 1 ml acetic acid + excess of magnesium uranyl acetate solution.	Yellow crystal	**Sodium ion** ☐ Present ☑ Absent
3.	**Identification test for thiosulphate ion :** 5 ml sample + 2 ml dilute hydrochloric acid.	White precipitate	**Thiosulphate ion** ☑ Present ☐ Absent
4.	**Identification test for thiosulphate ion :** 5 ml sample + 2 ml ferric chloride test solution.	Dark violet colour which disappears quickly	**Thiosulphate ion** ☑ Present ☐ Absent
5.	**Identification test for thiosulphate ion :** 5 ml sample + few drops of iodine solution.	Colour is discharged	**Thiosulphate ion** ☑ Present ☐ Absent

- **Result :** The given sample is identified as sodium thiosulphate.

Date _____

Date : _____ Marks : _____/10

EXPERIMENT NO. 27

- **Aim :** To perform identification test for sodium thiosulphate.
- **Requirements :**
  Chemicals : Sodium thiosulphate ($Na_2S_2O_3.5H_2O$), iodine solution, bromine solution, dilute hydrochloric acid, ferric chloride solution.
  Apparatus : Test tubes, burner, filter paper, glass rod, funnel, test tube holder, dropper *etc.*
- **Theory :** Identification tests are performed to determine the quality of drug so that drugs can be used safely. These identification tests are generally based on reactions of anions and cations present in the drug molecule. On the basis of anions and cations present, the quality of drug is established. Sodium thiosulphate is used as antidote in cyanide poisoning. It exists as an odourless, colourless crystalline mass or a coarsely crystalline powder with saline taste. It is deliquescent in moist air and efflorescent in dry air at temperature above 30°C. Sodium thiosulphate is identified on basis of identification tests of sodium and thiosulphate ions and also on the basis of its reducing properties.
- **Procedure :** Prepare a solution of sample by dissolving 1 g in 20 ml water and perform the following tests :

Identification tests for sodium ion :

1. Take 2 ml of the above solution and add 2 ml of 15% *w/v* solution of potassium carbonate. Heat to boil. No precipitate is formed. Add 4 ml of freshly prepared antimonite solution and heat to boil. Cool in ice bath and rub the inside wall of the test tube with glass rod, if necessary. A dense white precipitate of sodium antimonate is formed.

$$KSbO_3 + Na^+ \longrightarrow NaSbO_3 + K^+$$

2. Acidify 1 ml of the above solution with acetic acid. Add an excess of magnesium uranyl acetate solution. Yellow crystalline precipitate of sodium magnesium uranyl acetate is formed.

$$Na^+ + Mg^{2+} + 3UO_2^- + 9CH_3COO^- \longrightarrow NaMg(UO_2)_3(CH_3COO)_9$$

Identification tests for thiosulphate ion :

1. Dissolve 0.1 g of the sample in 5 ml of water and add 2 ml of hydrochloric acid. A white precipitate is formed which soon turns yellow due to separation of sulphur and sulphur dioxide is evolved which is recognised by its odour.

$$Na_2S_2O_3 + HCl \longrightarrow S + SO_2 + H_2O + 2NaCl$$

2. Dissolve 0.1 g of the sample in 5 ml of water and add 2 ml of ferric chloride test solution. A dark violet colour is produced which quickly disappears.
3. To 10% *w/v* solution of sample, add few drops of iodine solution. The colour is discharged. It occurs due to oxidation-reduction reaction.

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

- **Result :** The given sample is identified as sodium thiosulphate.

- **Observations :**

Sr. No.	Identification test	Observation	Inference
1	**Identification test for iodine :** Gentle heating of sample On condensation	Violet colour Bluish black crystal	**Iodine** □ Present □ Absent
2	**Identification test for iodine :** Sample + Potassium iodide + Starch mucilage On boiling of solution	Deep blue colour solution Blue colour disappears	**Iodine** □ Present □ Absent

- **Result :** The given sample is identified as iodine.

Date :_____ Marks :_____/10

EXPERIMENT NO. 23

- **Aim :** To perform identification test for iodine.
- **Requirements :**
  Chemicals : Iodine, potassium iodide, starch mucilage.
  Apparatus : Test tubes, burner, glass rod *etc.*
- **Theory :** Identification tests are performed to determine the quality of drug so that drugs can be used safely. Iodine is a topical anti-infective. It occurs as heavy, bluish-black, rhombic prism or plates with a metallic lustre and characteristic odour. It is volatile at room temperature. It is slightly soluble in water, soluble in ethanol, freely soluble in carbon disulphide, chloroform, ether, carbon tetrachloride and concentrated aqueous solution of iodides.
- **Procedure :**
  Identification tests for iodine :
  1. On gentle heating, iodine gives violet coloured vapours, which when condensed, form a bluish-black crystalline sublimate.
  2. Dissolve few crystals of iodine in potassium iodide solution and add few drops of starch mucilage. A deep blue colour is produced which disappears on boiling, but reappears on cooling.
- **Result :** The given sample is identified as iodine.

VIVA VOCE QUESTIONS

Observations

Sr. No	Identification test	observation	Inference
	Identification test for magnesium ion :-		
1.	2ml sample + 1ml of dilute ammonia solution which is dissolved in 1ml of 2M ammonium chloride solution	White PPT Precipitate gets dissolved	Magnesium ion present
2.	1ml sample + 0.25M disodium hydrogen phosphate solution	White PPT	Magnesium ion present.
3.	0.5ml sample + 0.2ml of 0.1% titan yellow hydroxide	Red PPT	Magnesium ion present
	Identification test for sulphate ion :-		
4.	1ml sample + 1ml of barium chloride solution	White PPT	Sulphate ion present
5.	1ml sample + 1ml of lead acetate solution	White PPT	Sulphate ion present.

Date

Expt. No. 6 Page No. 18

Aim :-
To Perform the identification test for the given sample of magnesium sulphate.

Reference :- "Gupta G.D., Sharma Shailesh" A Practical manual of Practical chemistry 1st Edition 2nd - 2017, Published by Nirali Prakashan Pune, Page No - 163.

Requirement :-

Chemicals :- Magnesium sulphate, ammonia solution, 2M ammonium chloride solution, 0.25M disodium hydrogen phosphate solution, 0.1% w/v solution of titan yellow, Sodium hydroxide, barium chloride, lead acetate.

Apparatus :- Test tubes, burner, Filter paper, glass rod, Funnel, test tube holder, dropper etc.

Theary :-
Identification tests are performed to determine the quality of drug so that drugs can be used safely. These identification tests are generally based on reactions of anions and cations present in the drug molecule. On the basis of anions and cations present, the

Teacher's Signature

quality of drug is established. Magnesium sulphate ($MgSO_4.7H_2O$) is also called Epsom salt. It occurs as odourless, colourless crystals with cool, saline and bitter taste. It gives efflorescence in warm dry air. Its one part is soluble in one part of water, but slightly soluble in ethanol and dissolves slowly in 1 part of glycerine. Magnesium sulphate is identified on the basis of identification tests of magnesium and sulphate ions.

Procedure :-

Prepare a solution of sample by dissolving 1g in 20ml water and perform following tests:

Identification tests for magnesium ion :-

1. Take 2ml of the above solution and add 1ml of dilute ammonia solution. White PPT of magnesium hydroxide is formed which gets dissolved on addition of 1ml of 2M ammonium chloride solution.

$$MgSO_4 + 2NH_4OH \longrightarrow Mg(OH)_2 + NH_4SO_4$$

2. Take 1ml of above solution and add 1ml of 0.25M disodium hydrogen phosphate solution. White PPT of magnesium ammonium phosphate appears.

Teacher's Signature

$Mg^{2+} + NH_3 + HPO^{4-} \longrightarrow Mg(NH_4)PO_4$

3. To 0.5 ml of a neutral or slightly acidic solution of magnesium sulphate, 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 is developed which is gradually settled to give a bright red precipitate.

Identification tests For Sulphate ion :-

1. Add 1ml of barium chloride solution to above solution. White PPT is formed due to formation of $MgSO_4$.

$MgSO_4 + BaCl_2 \longrightarrow BaSO_4 + MgCl$

2. Add 1ml of lead acetate solution to 1ml of above solution. white PPT is formed due to formation of lead sulphate.

$MgSO_4 + (CH_3COO)_2Pb \longrightarrow PbSO_4 + 2CH_3COOH$

Result :-

The given sample is identified as magnesium sulphate.

Observations :-

Sr. No.	Identification test	observation	Inference
1	Identification test for Na ion: 2 ml sample + 2 ml of 15% Potassium carbonate soln + 4 ml Freshly prepared antimonite soln. Boil and then Cool	White PPT	Sodium ion Present
2.	2 ml sample + 1 ml acetic acid + excess of magnesium uranyl acetate soln	Yellow crystal	Sodium ion present.
3.	Identification test for bicarbonate ion- Boil the above soln	CO_2 is evolved	Bicarbonate ion Present.
4.	2 ml sample + 2 ml magnesium sulphate solution Boil and cool.	White PPT	Bicarbonate ion present.

Date

Expt. No. 7 Page No. 21

Aim :-
To perform identification test for Sodium bicarbonate.

Reference :- "Gupta G.D, Sharma Shailesh"; A Practical manual of Pharmaceutical Chemistry 1st. Edition 2nd - 2017, Published by Nirali Prakashan Pune, Page No - 65.

Requirements :-

Chemicals - Sodium bicarbonate, potassium carbonate antimonite solution acetic acid etc.

Apparatus - Test tube, burner, Filter paper, glass rod, Funnel, dropper etc.

Theory :-
These identification test are generally based on reactions of anions and cations present in the drug molecule. On the basis of anions and cations present the quality of drug is established.

Procedure :-
Prepare a solution of sample by dissolving 1g in 20 ml water and perform following test -

Teacher's Signature

5	2ml sample + 2ml of 2M acetic acid solution. Close the tube with bent at right angles. Heat gently and collect the gas in 5ml barium hydroxide solution	white PPT	Bicarbonate ion present

Date

Expt. No. Page No. 22

Identification tests for sodium ion :-

1. Take 2ml of the above solution add 2ml of 15% w/v solution of potassium carbonate and heat to boil. No precipitate is formed. Add 4ml of freshly prepared antimonite solution and heat to boil. A dense white PPT of sodium antimonite is formed.

$$KSbO_3 + Na^+ \longrightarrow NaSbO_3 + K^+$$

2. Acidify 1ml of the above sol^n with acetic acid. Add an excess of magnesium uranyl acetate soln. Yellow crystalline PPT of sodium magnesium uranyl acetate is formed.

$$Na^+ + Mg^{2+} + 3UO_2^{2-} + 9CH_3COO^- \longrightarrow NaMg(UO_2)_3(CH_3COO)_9$$

Identification tests for bicarbonate ion :-

1. Boil the above solution carbon dioxide is evolved.

$$2NaHCO_3 \longrightarrow Na_2CO_3 + H_2O + CO_2$$

Date

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2. To 1 ml of above solution add 1ml of magnesium sulphate solution. No precipitate is formed. Boil the soln white PPT appears due to formation of magnesium carbonate.

$$MgSO_4 + NaHCO_3 \longrightarrow MgCO_3 + H_2O + CO_2$$

3. To 2ml of above soln, add 2ml of 2M 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 5ml of barium hydroxide solution. A white PPT is formed that dissolves on addition of an excess dilute hydrochloric acid.

$$NaHCO_3 + CH_3COOH \longrightarrow CH_3COONa + H_2O + CO_2$$

$$CO_2 + Ba(OH)_2 \longrightarrow BaCO_3 + H_2O$$

$$BaCO_3 + 2HCl \longrightarrow BaCl_2 + H_2O + CO_2$$

Result :-

The given sample is identified as sodium bicarbonate.

Teacher's Signature

Observation :-

Sr. No.	Identification test	observation	Inference
1	Identification test for zinc ion 0.2 g sample + 5 ml water + 0.2 N. NaOH solution	White PPT	zinc ion present
2	0.2 g sample + 5 ml water + 1 ml dilute sulphuric acid + one drop of 0.1 % cupric sulphate soln + 2 ml ammonium mercuric thiocyanate solution	Violet PPT	zinc ion present
3	0.2 g sample + 5 ml water + 2 ml of Potassium Ferrocyanide solution	White PPT	zinc ion present
4	Identification test for sulphate ion :- 1 ml sample + 1 ml of $BaCl_2$ soln	White PPT	Sulphate ion present
5	1 ml sample + 1 ml of lead acetate solution	White PPT	Sulphate ion present

Date

Expt. No. 8. Page No. 24

Aim :-
To perform identification test for Zinc sulphate.

Reference :- "Gupta G.D, Sharma Shailesh", A practical manual of Pharmaceutical Chemistry 1st Edition 2nd - 2017, Published by Nirali Prakashan Pune, Page No - 71.

Requirements :- Chemicals - Zinc sulphate ($ZnSO_4 \cdot 7H_2O$), sodium hydroxide, NH_4Cl, Na_2SO_4, dilute H_2SO_4, 0.1 % w/v soln of cupric sulphate.

Apparatus - Test tubes, burner, Filter paper, glass rod, funnel, test tube holder, dropper.

Theory :-
Identification tests are performed to determine the quality of drug that drugs can be used safely. These identification tests are generally based on reactions of anions and cations present in the drug molecule. On the basis of anions and cations present, the quality of drug is established. Zinc sulphate is used as an powder.

Teacher's Signature

Procedure :-

Identification tests for zinc ion :-

① Dissolve 0.2g of the sample in 5ml of water. Add 0.2 ml of sodium hydroxid soln white PPT is produced.

② Dissolve 0.2g of the sample in 5ml of water. Acidify with dilute H_2SO_4 and add one drop of a 0.1% w/v soln of cupric sulphate, and 2ml of ammonium mercuric thiocyanate solution violet PPT is formed.

Identification tests for Sulphate ion :-

① Add 1 ml of barium chloride soln to sample soln white PPT is formed due to formation of zinc sulphate.

$$ZnSO_4 + BaCl_2 \longrightarrow BaSO_4 + ZnCl.$$

② Add 1 ml of lead acetate soln to 1 ml of sample soln, white PPT is formed due to formation of lead sulphate.

$$ZnSO_4 + (CH_3COO)_2Pb \longrightarrow PbSO_4 + 2CH_3COOH.$$

Result :- The given sample is identified as zinc sulphate.

Observation :-

Sr. No	Identification test	observation	Inference.
1.	Identification test for barium ion :- Barium residue + HCl solution → Evolved gas on fire	Yellowish green Flame	Barium ion Present
2.	Wash the residue with water, then residue + 5ml dilute HCl → Filter. Filtrate + 3ml dilute H_2SO_4	White PPT	Barium ion Present
3.	Identification test for sulphate ion :- Filtrate + 2ml $BaCl_2$	White PPT	Sulphate ion Present
4.	Filtrate + 2ml lead sulphate	White PPT	Sulphate ion Present

Date

Expt. No. 9 Page No. 26

Aim :-
To perform identification test for barium sulphate.

Reference :- "Gupta G.D, Sharma Shailesh", A practical manual of Pharmaceutical chemistry Ist Edition 2nd -2017, Published by Nirali Prakashan Pune, Page No - 49.

Requirements :- Chemicals - Barium sulphate, Sodium carbonate, barium chloride, lead acetate soln, HCl, dilute H_2SO_4.

Apparatus - Test tube, burner, filter paper, glass rod, Funnel etc.

Theory :-
These identification tests are generally based on reactions of anions and cations present in the drug molecule. On the basis of anions and cations present, the quality of drug is established. Barium sulphate is used as diagnostic aid.

Procedure :- Boil 0.2 g of barium sulphate with 5ml of 50% sodium carbonate soln for 5 minutes.

$BaSO_4 + Na_2CO_3 \longrightarrow Na_2SO_4 + BaCO_3$.

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Identification tests for barium ion :-

1. Dissolve a portion of residue in hydrochloric acid and perform the flame test. Yellowish green colour of flame indicates presence of $BaSO_4$.

2. Wash the other portion of residue with small successive quantities of water. To the residue, add 5ml of dilute HCl and Filter it. To the Filtrate, add 0.3 ml of dilute H_2SO_4.

$$BaCO_3 + HCl \longrightarrow BaCl_2 + CO_2 + H_2O$$
$$BaCl_2 + H_2SO_4 \rightarrow BaSO_4 + NaCl$$

Identification tests for sulphate ion :-

1. Add 1 ml of barium chloride soln to a part of Filtrate white PPT is formed.
$$Na_2SO_4 + BaCl_2 \longrightarrow BaSO_4 + NaCl.$$

2. Add 1ml of lead acetate soln to 1ml of filtrate. white PPT is formed due to formatio of lead sulphate.
$$Na_2SO_4 + (CH_3COO)_2Pb \longrightarrow PbSO_4 + 2CH_3COOH$$

Result :- The given sample is identified as barium sulphate.

Teacher's Signature

Observation :-

Sr. No	Identification test	observation	Inference
1	Identification test for boric acid :- Dissolve 0.1g of sample (gentle warm) + 5 ml methyl alcohol + few drops of H_2SO_4 Ignite the soln → Flame test	Green flame	Boric acid Present
2	3g sample + 90 ml boiling water and cool.	Acidic solution	Boric acid Present

Date

Expt. No. 10 Page No. 28

Aim :-
To perform identification test for boric acid.

Reference :- "Gupta G.D, Sharma Shailesh", A practical manual of Pharmaceutical chemistry Ist, Edition - 2nd - 2017, Published by Nirali Prakashan Pune, Page No - 51.

Requirements :- Chemicals - Boric acid, methanol, Sulphuric acid.

Apparatus - Test tubes, burner, glass rod, Funnel etc.

Theory :-
Identification tests are performed to determine the quality of drug so that drugs can be used safely. Boric acid is a local anti-infective. It is also used as dusting powder. It is a colourless or white crystalline powder. It is odourless and taste is slightly acidic and bitter, with a sweetish after taste. Boric acid is sparingly soluble in water and ethanol, and is freely soluble in boiling water, boiling ethanol and glycerine.

Teacher's Signature

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Expt. No. Page No. 29

Procedure :-

Identification tests for boric acid :-

1. Dissolve 0.1 g of boric acid with gentle warming in 5ml of methyl alcohol containing few drops of sulphuric acid. Ignite the solution and perform the flame test. The flame has green border due to formation of methyl bromate.

$$H_3BO_3 + CH_3OH \longrightarrow B(OCH_3) + 3H_2O$$

2. Dissolve 3g boric acid in 90ml of boiling water and cool. The solution is faintly acidic.

Result :-

The given sample is identified as boric acid.

Observation :-

Sr. No	Identification test	Observation	Inference
1.	Identification test for borax :- 5% solution of sample + HCl → moisten a piece of turmeric paper with this solution	Colour of paper become pink or red	Borax present
2.	Ignite the mixture of borax with sulphuric acid and alcohol in a porcelain dish	Greenish flame	Borax present

Date

Expt. No. 11 Page No. 30

Aim :-
To perform identification test for borax

Reference :- "Gupta G.D, Sharma shailesh", A Practical manual of Physical chemistry Ist edition 2nd - 2017, Published by Nirali prakashan pune, Page No- 53.

Requirements :- Chemicals - Borax, HCl, turmeric paper, NaOH, H_2SO_4.

Apparatus - Test tubes, burner, glass rod, porcelain dish etc.

Theory :-
Identification tests are performed to determine the quality of drug so that drugs can be used safely. Borax is a bacteriostatic and pharmaceutical aid. It is available as transparent, colourless crystals. It is odourless compound with saline taste and is alkaline. It is slightly soluble in water, soluble in boiling water and insoluble in alcohol.

Teacher's Signature

Procedure :-

Identification tests for borax :-

1. Acidify 5% solution of borax with hydrochloric acid and moisten a piece of turmeric paper with this solution and make it dry. The colour of the paper becomes pink or brownish red. Put dilute ammonia sol^n or solution of Sodium hydroxide on the paper. The colour of paper changes to blue or greenish black.

2. Ignite the mixture of borax with sulphuric acid and alcohol in a porcelain dish. The greenish tinged flame is produced.

Result :-

Hence the given sample is identified as borax.

Observation :-

Standard solution	Test solution
More opalescence	more opalescence
Equal opalescence	Equal opalescence.
Less opalescence than test solution	less opalescence than standard solution

Date

Expt. No. 12. Page No. 32

Aim :-
To perform limit test for chloride in given compound.

Reference :- "Gupta G.L, Sharma Shailesh"; A Practical manual of Pharmaceutical chemistry Vol, Edition 2nd - 2017, Published by Nirali Prakashan Pune, Page No - 75.

Requirements :-

Chemicals - Chloride standard solution (25 ppm), dilute nitric acid, distilled water, 0.1M silver nitrate solution.

Apparatus - Nessler cylinder, glass rod, beaker, dropper, volumetric flasks, pipette etc.

Theory :-
Limit tests are performed to check the limits of impurities present in pharmaceutical substance. These tests involve comparison of opalescence, turbidity or colour of solutions of test substances with standard substance.

$$NaCl + AgNO_3 \xrightarrow{Dil.\ HNO_3} AgCl + NaNO_3$$

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Procedure :-

1. Preparation of chloride standard solution :-

Dilute 5 volumes of 0.0824% w/v of sodium chloride solution to 100 volumes with water. It gives standard chloride solution of 25 ppm.

2. Preparation of test opalescence :-

Dissolve the specified quantity of the substance under examination in water or prepare a solution as directed in the individual monograph and transfer it to a nessler cylinder.

3. Preparation of standard opalescence :-

Take a mixture of 10 ml of chloride standard soln and 5 ml of water in a Nessler cylinder.

4. Observation :- After 5 min, view both standard and test cylinder transversely against a black background.

Result :- The opalescence of both solutions is compared. The given compound the limit test for chloride.

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observation :-

Standard solution	Test solution
more opalescence	more opalescence
Equal opalescence	Equal opalescence
less opalescence than test solution	less opalescence than standard solution

Expt. No. 13 Date

Page No. 34

Aim :-
To perform limit test for sulphate in given compound.

Reference :- "Gupta, G.P., Sharma Shailesh", A Practical manual of Pharmaceutical Chemistry Ist Edition 2nd - 2017, Published by Nirali Prakashan Pune, Page No - 77.

Requirements :-

Chemicals - Standard sulphate solution (10ppm), ethanolic standard sulphate solution (10ppm), 25% w/v barium chloride solution, 5M acetic acid, distilled water.

Apparatus - Nesseler cylinder, glass rod, beaker, dropper, volumetric flasks (100ml), pippete (100ml) etc.

Theory :-
Limit test for sulphate is based on reaction of barium chloride and soluble sulphate in acidic solution. The opalescence produced by test substances should be less intense than that of standard subs. to pass the limit test for chloride.

$BaCl_2 + Na_2SO_4 \longrightarrow BaSO_4\downarrow + 2NaCl$
white ppt

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Date

Expt. No.

Procedure :-

1. Preparation of ethanolic sulphate standard solution :-

Dilute 1 volume of 0.181% w/v of Potassium sulphate solution in 30% ethanol to 100 volumes with 30% ethanol. It gives ethanolic standard sulphate solution of 100 PPM.

2. Preparation of sulphate standard solution :-

Dilute 1 volume of 0.181% w/v of potassium sulphate solution in distilled water to 100 volumes with distilled water. It gives standard sulphate solution of 10 PPM.

3. Preparation of test opalescence :-

To 10ml of a 25% w/v solution of barium chloride in a Nessler cylinder, add 2.5 ml of ethanolic sulphate standard solution (10 PPM SO_4), mix and allow to stand for 1 minute. Add 15 ml of the solution prepared as directed in the monograph or a solution of the specified quantity of the substance under examination in 15ml of water and 0.15 ml of 5M acetic acid.

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4. Preparation of standard opalescence :-

To 1 ml of a 25% w/v solution of barium chloride in a Nessler cylinder, add 1.5 ml of ethanolic sulphate standard solution (10 ppm SO_4). Mix and allow to stand for 5 minutes. Add 15 ml of sulphate standard soln (10 ppm SO_4) in 15 ml of water and 0.15 ml of 5M acetic acid. Add sufficient water to produce 50 ml solution. Stir immediately with a glass rod and allow to stand for 5 minutes.

5. Observation :-

After 5 minutes, view both standard and test cylinder transversely against a black background.

Result :-

The opalescence of both solutions is compared. The given compound the limit test for sulphate.

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Observations :-

Standard solution	Test Solution
more opalescence	more opalescence
Equal opalescence	Equal opalescence
Less opalescence than test solution	Less opalescence than standard solution.

Date

Expt. No. 14 Page No. 37

Aim :-
To perform limit test for iron in given compound.

Reference :- "Gupta G.D, Sharma Shailesh", A practical manual of pharmaceutical chemistry Ist, Edition 2nd - 2017, Published by Nirali prakashan Pune, Page No - 79.

Requirements :-

Chemicals - Iron standard solution (20ppm), 20% w/v iron free citric acid, thioglycollic acid, iron free ammonia solutions, distilled water.

Apparatus - Nessler cylinder, glass rod, beaker, dropper, volumetric flasks (100 ml), pipette (100 ml) etc.

Theory :-
Limit tests are performed to check the limits of impurities present in pharmaceutical substances. These tests involve comparison of opalescence turbidity or colour of solutions of test substances with standard substances. The extent of opalescence turbidity, colour is affected by presence

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of impurities present in substance, variation in time and method of performance of test.

$2HSCH_2COOH + Fe^{2+}/Fe^{3+} \longrightarrow Fe(HSCH_2COOH)_2 + 2H^+$

Thioglycollic acid — Ferrous thioglycollate.

Procedure :-

1. Preparation of iron standard solution :-

Dilute 2 volumes of 0.1726 % w/v of ferric ammonium sulphate in 0.05 M sulphuric acid to 10 volumes with water. It gives Standard iron solution of 20 PPM.

2. Preparation of test colour :-

Dissolve the specified quantity of the substances under examination in 40 ml water, or prepare a solution (10 ml) as directed in the monograph, and transfer to a Nessler cylinder. Add 2 ml of a 20% w/v solution of iron-free citric acid and 0.1 ml of thioglycollic acid. Mix them and make alkaline with iron-free ammonia solution. Dilute it to 50 ml with water and allow to stand for 5 minutes.

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3. Preparation of standard opalescence :-

Take 2ml of iron standard solution (20 ppm) in a Nessler cylinder. Add 2ml of 20% w/v solution of iron-free citric acid and 0.1ml of thioglycollic acid. Mix them and make alkaline with iron-free ammonia solution. Dilute it to 50ml with water and allow to stand for 5 minutes.

4. Observation :-

After 5 minutes, view both standard and test cylinder to compare intensity of colour produced.

Result :-

The opalescence of both solutions is compared. The given compound the limit test for iron.

Observations :-

Sr. No	Volume of $CaCl_2$ solution taken (ml)	Burette reading (ml)		Volume of EDTA solution used (ml)
		Initial	Final	
1	10			
2	10			
3	10			

Average volume of EDTA solution used (V_1) =

Average volume of $CaCl_2$ solution taken (V_2) = 10 ml

Calculations :- The molarity of 0.5 M EDTA solution is calculated by using the following Formula

$$\frac{M_1V_1}{n_1} = \frac{M_2V_2}{n_2}$$

M_1 = Molarity of EDTA
V_1 = volume of EDTA
n_1 = 1

M_2 = Molarity of $CaCl_2$ = 0.05 M
V_2 = Volume of $CaCl_2$ = 10 ml
n_2 = 1

$$M_1 = \frac{n_1M_2V_2}{n_2V_1} = \frac{1 \times 0.05 \times 10}{1 \times}$$

Date

Expt. No. 15 Page No. 40

Aim :- To perform assay of a given sample of Calcium gluconate.

Reference :- "Gupta G.D, Sharma Shailesh." A Practical manual of Pharmaceutical chemistry Ist Edition 2nd - 2017, published by Nirali Prakashan pune, Page No - 39.

Requirement :-

Chemicals :- Calcium gluconate, 0.05 M EDTA soln, 0.05 M Calcium chloride soln, 0.05 M magnesium sulphate solution, eriochrome black-T indicator, ammonia buffer (pH 10), distilled water etc.

Apparatus :- Burette, pipette (10 ml), Conical Flask, beaker, Volumetric flask, Funnel, glass rod etc.

Theory :-

Calcium gluconate is Calcium D-gluconate monohydrate. Calcium gluconate contains not less than 98.5% and not more than 102% of $C_{12}H_{22}CaO_{14} \cdot H_2O$. Its molecular weight is 448.4 g/mol. It exists as a white,

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Assay of calcium gluconate :-

Sr. No.	Volume of Calcium gluconate solution (ml)	Burette reading (ml)		Volume of EDTA solution used (ml)
		Initial	Final	
1.	50			
2	50			
3	50			

Average volume of EDTA used =

IP Factor or equivalent factor :-

Each ml of 0.05 M EDTA = 0.02 g of $C_{12}H_{22}CaO_{14}$

Calculations :- The percentage purity of given sample of Calcium gluconate is calculated by using the Following formula:

% of Calcium gluconate = $\frac{\text{Volume of EDTA used} \times \text{IP factor} \times 100 \times M}{\text{weight of calcium gluconate (g)} \times M}$

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Crystalline powder or granules. Calcium gluconate is a mineral supplement and used in treatment of hypokalemia, hypocalcemia and magnesium sulphate overdose. This titrations are the reactions in which a metal ion is converted into a stable complex ion by addition of reagent or a ligand.

$C_{12}H_{22}CaO_{14} + Na_2H_2Y + 2NH_3 \rightarrow CaNa_2Y + (NH_4)_2C_{12}H_{22}CaO_{14} \cdot H_2O$

Procedure :-

Standardisation of EDTA :-

1. Preparation of 0.05 M disodium edetate solution :-

Weigh accurately 18.6 g of disodium edetate and dissolve it in approximately 900 ml of water.

2. Preparation of 0.05 M calcium chloride solution :-

Weigh accurately 14.7 g of calcium chloride and dissolve it in approximately 900 ml of water. Transfer this solution to a volumetric flask (1000 ml) and make up the volume. Shake it well to mix properly.

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Where,
M_p EDTA is the calculated molarity of EDTA solution,
M_T EDTA is the theoretical molarity of EDTA solution.

% Purity of calcium gluconate = $\frac{\text{ml of EDTA Used} \times 0.05 \times \text{MEDTA}}{0.5 \times 0.05}$

$= \frac{\text{ml EDTA} \times \text{MEDTA}}{\text{MEDTA}} \times 88$

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3. Preparation of Ammonia buffer (pH 10) :-

Dissolve 5.4 g of ammonium chloride in 20 ml of water.

4. Titration :-

Rinse and fill the burette with 0.05M EDTA solution with the help of Funnel. Pipette out 10ml of 0.05M calcium chloride solution into a clean and dry conical flask and add 2-3 drops of Eriochrome black T indicator to it.

Assay :-

1. Preparation of 0.05 M Magnesium sulphate solution :-

Dissolve 12.5 g of magnesium sulphate in sufficient water to produce 1000 ml.

2. Assay of Calcium gluconate :-

Weigh accurately about 0.5 g of calcium gluconate and dissolve it in 50ml of warm water.

Result :- The percentage purity of given sample (Almost) of Calcium gluconate is .

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