JHARKHAND
Rai University
UGC RECOGNISED UNIVERSITY
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PRACTICAL LAB MANUAL

Pharmaceutical Chemistry

D. Pharm Ist Year

LIST OF EXPERIMENTS

S. No.	EXPERIMENTS
1	To determine the strength of a given unknown solution of HCl by titrating it against with the help of a known solution of NaOH using phenolphthalein indicator.
2	To determine the strength of a given unknown solution of NaOH by titrating it against with the help of a known solution of HCl using methyl orange indicator.
3	Determination of the concentration (strong) of a given NaOH solution by titrating it against a standard (M/20) solution of C2H2O4 using phenolphthalein indicator.
4	To perform the limit test of chloride in the given sample of tap water.
5	To perform the limit test for sulphate in the given sample of tap water.
6	To perform the limit test for chloride in the given sample of sodium bicarbonate.
7	To perform the limit test for chloride in the given sample of sodium hydroxide.
8	To perform the limit test for chloride in the given sample of sodium acetate.
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10	To perform the limit test for sulphates in the given sample of sodium citrate.
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22	To perform the assay of ammonium chloride.
23	To prepare and standardize 200 ml of 0.1 M silver nitrate solution..
24	To perform the assay of magnesium sulphate.
25	To perform the assay of calcium gluconate.

EXPERIMENT NO -1

OBJECT: - To determine the strength of a given unknown solution of HCl by titrating it against with the help of a known solution of NaOH using phenolphthalein indicator.

REFERENCE

1. Parle A., " Pharmaceutical Chemistry 1 Laboratory Manual", CBS Publishers and Distributors Pvt. Ltd., Ed Ist, 2008, pp 59.

REQUIREMENTS: -

Chemical required: NaOH Solution, HCl solution, phenolphthalein indicator

Apparatus required: - burette, conical flask, and beaker

PROCEDURE:-

(a) Take a burette and wash it with distilled water.

(b) Rinse and fill the solution HCl N/10 with the help of a funnel and set the initial burette reading as zero. Clamp it vertically to the burette stand.

(c) Rinse the pipette with water and then with the given NaOH solution.

(d) Pipette out 10ml of given NaOH (N/10) solution into a conical flask and add one or two drops of methyl orange.

(e) Titrate it against the HCl (N/10) solution taken in the burette till the color of the solution in the conical flask changes from a yellowish color to pink color

(f) Note down the final burette reading.

(g) Repeat the titration until concordant values are obtained.

OBSERVATION:

No of observation	Volume of NaOH solution in ml	Burette reading		Different	Constant	Indicator used
		Initial	Final			

Calculation:

$$N_1V_1=N_2V_2$$

RESULT:- The strength of given unknown solution oh HCl is

EXPERIMENT NO -2

OBJECT: - To determine the strength of a given unknown solution of NaOH by titrating it against with the help of a known solution of HCl using methyl orange indicator.

REFERENCE

Parle A., "Pharmaceutical Chemistry 1 Laboratory Manual", CBS Publishers and distributors Pvt. Ltd., Ed Ist, 2008, pp 55.

REQUIREMENTS: -

Chemical required: NaOH Solution, HCl solution, methyl orange indicator

Apparatus required: - burette, conical flask, and beaker.

PROCEDURE:-

Take a burette and wash it with distilled water.

Rinse and fill the solution HCl N/10 with the help of a conical funnel and set the initial burette reading as zero. Clamp it vertically to the burette stand. Rinse the pipette with water and then with the given NaOH solution.

(a) Pipette out 10ml of given NaOH (N/10) solution into a conical flask and add one or two drops of methyl orange.

(b) Titrate it against the HCl(N/10) solution taken in the burette till the color of the solution in the conical flask changes from a yellowish color to pink color

Note down the final burette reading.

(c) Repeat the titration until concordant values are obtained.

OBSERVATION:

No of observation	Volume of HCl solution in mL	Burette reading		Different	Constant	Indicator used
		Initial	Final			

Calculation:

$$N_1V_1=N_2V_2$$

RESULT:- The strength of the given unknown solution oh NaOH is

EXPERIMENT NO -3

OBJECT: - Determination of the concentration (strong) of a given NaOH solution by titrating it against a standard (M/20) solution of $C_2H_2O_4$ using phenolphthalein indicator.

REFERENCE

Parle A. ,"Pharmaceutical Chemistry 1 Laboratory Manual", CBS Publishers and Distributors Pvt. Ltd., Ed Ist, 2008, pp 59.

REQUIREMENTS: -

Chemical required: NaOH Solution, HCl solution, phenolphthalein indicator, $C_2H_2O_4$

Apparatus required: - burette, conical flask, and beaker.

PROCEDURE:-

(a) Take a burette and wash it with distilled water.

(b) Rinse and fill the solution HCl N/10 with the help of a conical funnel and set the initial burette reading as zero. Clamp it vertically to the burette stand.

(c) Rinse the pipette with water and then with the given NaOH solution.

(d) Pipette out 10ml of given NaOH (N/10) solution into a conical flask and add one or two drops of methyl orange.

(e) Titrate it against the HCl(N/10) solution taken in the burette till the color of the solution in the conical flask changes from a yellowish color to pink color

(f) Note down the final burette reading.

(g) Repeat the titration until concordant values are obtained.

OBSERVATION:

No of observation	Volume of NaOH solution in mL	Burette reading		Different	Constant	Indicator used
		Initial	Final			

Calculation:

$$N_1V_1=N_2V_2$$

RESULT:- The strength of a given unknown solution of strong NaOH is

EXPERIMENT NO -4

OBJECT: - To perform the limit test for chloride in a given sample of tap water.

REFERENCE: 1. Parle A.," Pharmaceutical Chemistry 1 Laboratory Manual", CBS Publishers and Distributors Pvt. Ltd., Ed Ist, 2008, pp 59.

REQUIREMENTS: -

Chemical required: Silver nitrate, dilute nitric acid, sodium chloride, etc.

Apparatus required: - Measuring cylinder, glass rod, and Nessler's cylinder.

THEORY

Limit tests are quantitative or semi-quantitative test designed to identify and control a small number of impurities which are likely to be present in the substance. This test involves the reaction of silver nitrate with soluble chloride to form the ppt. of silver chloride which is insoluble in dilute HNO_3. The extent of the precipitation depends upon the amount of silver chloride formed i.e. on the number of chloride ions present in the substance. The opalescence produced in the test solution is compared with a reference/standard solution under the same experimental conditions.

$$\mathbf{Cl^- + AgNO_3 \rightarrow AgCl \downarrow + NO_3^-}$$

ppt.

$$\mathbf{NaCl + AgNO_3 \rightarrow AgCl \downarrow + NaNO_3}$$

ppt.

PROCEDURE

STANDARD - 1mL of 0.05845% w/v solution of NaCl is taken in Nessler's cylinder Add 10 mL of Dil. HNO_3 Make up the volume up to 50 ml with distilled water. Now add 1mL of silver nitrate to this solution. Stirrer the solution with glass road and allow to stand for 5 minutes.

TEST

Dissolve a specified quantity of substances as per I.P. monograph in10mL of distilled water. Add 10 mL of Dil. Nitric acid. Makeup the volume up to 50 mL with tap water. Now add 1mL of silver nitrate to this solution. Stirrer the solution with glass road and allow to stand for 5 minutes.

OBSERVATION

The opalescence of the test solution is less/more than the standard solution. If the opalescence of the test solution has been less than the standard opalescence, the sample will pass the limit test.

RESULT

A limit test for chloride was performed.

EXPERIMENT NO -5

OBJECT: To perform a limit test for sulphate in a given sample of tap water.

REFERENCES:

Parle A., "Pharmaceutical Chemistry 1 Laboratory Manual", CBS Publishers and Distributors Pvt. Ltd.,Ed Ist, 2008, pp 59

REQUIREMENTS

Chemical required: Hydrochloric acid, Barium sulphate, and Barium chloride

Apparatus required: Measuring cylinder, glass rod, pipette and Nessler's cylinder, etc.

THEORY

Limit tests are quantitative or semi-quantitative test designed to identify and control a small number of impurities which are likely to be present in the substance. This test involves the reaction of Barium chloride with soluble sulphate to form the precipitate of Barium sulphate which is insoluble in dilute hydrochloric acid. The Barium sulphate precipitate is white in color.

REACTION - $SO4^{--} + BaCl2 \rightarrow BaSO4 + Cl^-$

(White ppt.)

$$Na_2SO_4 + BaCl_2 \rightarrow BaSO_4 + NaCl$$

(White ppt.)

PROCEDURE

STANDARD - Take 1mL of 0.1089 w/v of Na_2SO_4 or K_2SO_4 as per I.P. in Nessler cylinder. Add 2 mL of dilute Hydrochloric acid. Makeup the volume up to 45 ml with distilled water. Add in this solution of 5 mL of Barium sulphate reagent. Stirrer the solution with a glass rod and allow to stand for 5 minutes.

TEST

Dissolve the specific quantity of test substances in 10 mL of tap water. Add 2 mL of dilute Hydrochloric acid. Makeup the volume up to 45 ml with distilled water. Add in this solution of 5 mL of Barium sulphate reagent Stirrer the solution with a glass rod and allow to stand for 5 minutes.

OBSERVATION

The opalescence of the test solution is less/more than the standard solution. If the opalescence of the test solution has been less than the standard opalescence, the sample will pass the limit test.

RESULT

Limit test for sulphate was performed

EXPERIMENT NO -6

OBJECT: - To perform the limit test for chloride in a given sample of sodium bicarbonate.

REFERENCE

Parle A., "Pharmaceutical Chemistry 1 Laboratory Manual", CBS Publishers and Distributors Pvt. Ltd., Ed Ist, 2008, pp 59

REQUIREMENTS: -

Chemical required: sodium bicarbonate, dilute nitric acid, 0.1 M silver nitrate solution, etc.

Apparatus required: - Measuring cylinder, glass rod, and Nessler's cylinder, Beaker.

THEORY

Limit tests are quantitative or semi-quantitative test designed to identify and control a small number of impurities which are likely to be present in the substance. This test involves the reaction of silver nitrate with soluble chloride to form the ppt. of silver chloride which is insoluble in dilute HNO_3. The extent of the precipitation depends upon the amount of silver chloride formed i.e. on the number of chloride ions present in the substance. The opalescence produced in the test solution is compared with a reference/standard solution under the same experimental conditions.

$$\mathbf{Cl^- + AgNO_3 \rightarrow AgCl \downarrow + NO_3^-}$$

ppt.

$$\mathbf{NaCl + AgNO_3 \rightarrow AgCl \downarrow + NaNO_3}$$

ppt.

PROCEDURE STANDARD - 10 ml of chloride standard solution is taken in Nessler's cylinder. Mix it with 5 ml of water. Add 10 mL of Dil. HNO_3 Make up the volume up to 50 ml with distilled water. Now add 1mL of silver nitrate to this solution. Stirrer the solution with glass road and allow to stand for 5 minutes.

TEST

Dissolve a specified quantity of substances as per I.P. monograph in10mL of distilled water. Add 10 mL of Dil. Nitric acid. Makeup the volume up to 50 mL with tap water. Now add 1mL of silver nitrate to this solution. Stirrer the solution with glass road and allow to stand for 5 minutes.

OBSERVATION

The opalescence of the test solution is less/more than the standard solution. If the opalescence of the test solution has been less than the standard opalescence, the sample will pass the limit test.

RESULT

Limit test for chloride was performed

EXPERIMENT NO -7

OBJECT: - To perform a limit test for chloride in a given sample of sodium hydroxide.

REFERENCE

Parle A., "Pharmaceutical Chemistry 1 Laboratory Manual", CBS Publishers and distributors Pvt. Ltd., Ed Ist, 2008, pp 59

REQUIREMENTS: -

Chemical required: sodium hydroxide, dilute nitric acid, 0.1 M silver nitrate solution, etc.

Apparatus required: - Measuring cylinder, glass rod, and Nessler's cylinder, Beaker.

THEORY

A limit test is a quantitative or semi-quantitative test designed to identify and control a small number of impurities which are likely to be present in the substance. This test involves the reaction of silver nitrate with soluble chloride to form the ppt. of silver chloride which is insoluble in dilute HNO_3. The extent of the precipitation depends upon the amount of silver chloride formed i.e. on the number of chloride ions present in the substance. The opalescence produced in the test solution is compared with a reference/standard solution under the same experimental conditions.

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

ppt.

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

ppt.

PROCEDURE

STANDARD -10 ml of chloride standard solution is taken in Nessler's cylinder. Mix it with 5 ml of water. Add 10 mL of Dil. HNO_3 Make up the volume up to 50 ml with distilled water. Now add 1mL of silver nitrate to this solution. Stirrer the solution with glass road and allow to stand for 5 minutes.

TEST - Dissolve a specified quantity of substances as per I.P. monograph in10mL of distilled water. Add 10 mL of Dil. Nitric acid. Makeup the volume up to 50 mL with tap water. Now add 1mL of silver nitrate to this solution. Stirrer the solution with glass road and allow to stand for 5 minutes.

OBSERVATION

The opalescence of the test solution is less/more than the standard solution. If the opalescence of the test solution has been less than the standard opalescence, the sample will pass the limit test.

RESULT

A limit test for chloride was performed.

EXPERIMENT NO- 8

OBJECT: - To perform the limit test for chloride in a given sample of sodium acetate.

REFERENCE

Parle A., "Pharmaceutical Chemistry 1 Laboratory Manual", CBS Publishers and Distributors Pvt. Ltd., Ed Ist, 2008, pp 59

REQUIREMENTS: -

Chemical required: sodium acetate, dilute nitric acid, 0.1 M silver nitrate solution, etc.

Apparatus required: - Measuring cylinder, glass rod, and Nessler's cylinder, Beaker.

THEORY

Limit tests are quantitative or semi-quantitative test designed to identify and control a small number of impurities which are likely to be present in the substance. This test involves the reaction of silver nitrate with soluble chloride to form the ppt. of silver chloride which is insoluble in dilute HNO_3. The extent of the precipitation depends upon the amount of silver chloride formed i.e. on the number of chloride ions present in the substance. The opalescence produced in the test solution is compared with a reference/standard solution under the same experimental conditions.

$$\mathbf{Cl^- + AgNO_3 \rightarrow AgCl \downarrow + NO_3^-}$$

ppt.

PROCEDURE

STANDARD - 10 ml of chloride standard solution is taken in Nessler's cylinder. Mix it with 5 ml of water. Add 10 mL of Dil. HNO_3 Make up the volume up to 50 ml with distilled water. Now add 1mL of silver nitrate to this solution. Stirrer the solution with glass road and allow to stand for 5 minutes.

TEST

Dissolve a specified quantity of substances as per I.P. monograph in10mL of distilled water. Add 10 mL of Dil. Nitric acid. Makeup the volume up to 50 mL with tap water. Now add 1mL of silver nitrate to this solution. Stirrer the solution with glass road and allow to stand for 5 minutes.

OBSERVATION

The opalescence of the test solution is less/more than the standard solution. If the opalescence of the test solution has been less than the standard opalescence, the sample will pass the limit test.

RESULT

The limit test for chloride was performed.

EXPERIMENT NO -9

OBJECT: To perform a limit test for sulphate in a given sample of sodium bicarbonate.

REFERENCES

Parle A., "Pharmaceutical Chemistry 1 Laboratory Manual", CBS Publishers and distributors Pvt. Ltd., Ed Ist, 2008, pp 59.

REQUIREMENTS

Chemical required: sodium bicarbonate, ethanol, potassium sulphate, 5M acetic acid, HCl, and Barium chloride

Apparatus required: Measuring cylinder, glass rod, pipette and Nessler's cylinder, etc.

THEORY

Limit tests are quantitative or semi-quantitative test designed to identify and control the small number of impurities which are likely to be present in the substance. This test involves the reaction of Barium chloride with soluble sulphate to form the precipitate of Barium sulphate which is insoluble in dilute hydrochloric acid. The Barium sulphate precipitate is white in color.

REACTION: $SO_4^{--} + BaCl_2 \rightarrow BaSO_4 + Cl^-$

(White ppt.)

$$Na_2SO_4 + BaCl_2 \rightarrow BaSO_4 + NaCl$$

(White ppt.)

PROCEDURE

STANDARD - Take 1ml of 0.1089 w/v of Na_2SO_4 or K_2SO_4 as per I.P. in Nessler cylinder. Add 2 ml of dilute Hydrochloric acid. Makeup the volume up to 45 ml with distilled water. Add in this solution of 5 mL of Barium sulphate reagent Stirrer the solution with a glass rod and allow to stand for 5 minutes.

TEST

Dissolve the specific quantity of test substances in 10 mL of tap water. Add 2 mL of dilute Hydrochloric acid. Makeup the volume up to 45 ml with distilled water. Add in this solution of 5 mL of Barium sulphate reagent Stirrer the solution with a glass rod and allow to stand for 5 minutes.

OBSERVATION - The opalescence of the test solution is less/more than the standard solution. If the opalescence of the test solution has been less than the standard opalescence, the sample will pass the limit test.

RESULT - Limit test for sulphate was performed

EXPERIMENT NO -10

OBJECT: To perform a limit test for sulphate in a given sample of sodium citrate.

REFERENCES

Singh H.R., Kapoor V.K. "Practical Pharmaceutical chemistry", Vallabh Prakashan, Ed Ist, 2008, pp 57.

REQUIREMENTS

Chemical required: sodium citrate, ethanol, potassium sulphate, 5M acetic acid, HCl, and Barium chloride

Apparatus required: Measuring cylinder, glass rod, pipette and Nessler's cylinder, etc.

THEORY

Limit test is a quantitative or semi-quantitative test designed to identify and control a small quantity of impurities which are likely to be present in the substance This test involves the reaction of Barium chloride with soluble sulphate to form the precipitate of Barium sulphate which is insoluble in dilute hydrochloric acid. The Barium sulphate precipitate is white in color.

REACTION - $SO4^{--} + BaCl2 \rightarrow BaSO4 + Cl^-$

(White ppt.)

$$Na_2SO_4 + BaCl_2 \rightarrow BaSO_4 + NaCl$$

(White ppt.)

PROCEDURE

STANDARD - Take 1mL of 0.1089 w/v of Na_2SO_4 or K_2SO_4 as per I.P. in Nessler cylinder. Add 2 ml of dilute Hydrochloric acid. Makeup the volume up to 45 ml with distilled water. Add in this solution of 5 mL of Barium sulphate reagent. Stirrer the solution with a glass rod and allow to stand for 5 minutes.

TEST

Dissolve the specific quantity of test substances in 10 mL of tap water. Add 2 mL of dilute Hydrochloric acid. Makeup the volume up to 45 ml with distilled water. Add in this solution of 5 mL of Barium sulphate reagent. Stirrer the solution with a glass rod and allow to stand for 5 minutes.

OBSERVATION

The opalescence of the test solution is less/more than the standard solution. If the opalescence of the test solution has been less than the standard opalescence, the sample will pass the limit test.

RESULT - A limit test for sulphate was performed.

EXPERIMENT NO 11

OBJECT: To perform a limit test for sulphate in a given sample of sodium dihydrogen phosphate dihydrate.

REFERENCES

Singh H.R., Kapoor V.K. "Practical Pharmaceutical chemistry", Vallabh Prakashan, Ed Ist, 2008, pp 57.

REQUIREMENTS

Chemical required: sodium dihydrogen phosphate dihydrate, ethanol, potassium sulphate, 5M acetic acid, HCl, and Barium chloride

Apparatus required: Measuring cylinder, glass rod, pipette and Nessler's cylinder, etc.

THEORY

Limit test are quantitative or semi-quantitative test designed to identify and control the small quantity of impurities which are likely to be present in the substance

This test involves the reaction of Barium chloride with soluble sulphate to form the precipitate of Barium sulphate which is insoluble in dilute hydrochloric acid. The Barium sulphate precipitate is white in color.

REACTION

$$SO_4^{--} + BaCl_2 \rightarrow BaSO_4 + Cl^-$$

(White ppt.)

$$Na_2SO_4 + BaCl_2 \rightarrow BaSO_4 + NaCl$$

(White ppt.)

PROCEDURE

STANDARD

Take 1mL of 0.1089 w/v of Na_2SO_4 or K_2SO_4 as per I.P. in Nessler cylinder. Add 2 mL of dilute Hydrochloric acid. Makeup the volume up to 45 ml with distilled water. Add in this solution of 5 mL of Barium sulphate reagent Stirrer the solution with a glass rod and allow to stand for 5 minutes.

TEST

Dissolve the specific quantity of test substances in 10 mL of tap water. Add 2 mL of dilute Hydrochloric acid. Makeup the volume up to 45 ml with distilled water. Add in this solution of 5 mL of Barium sulphate reagent Stirrer the solution with a glass rod and allow to stand for 5 minutes.

OBSERVATION

The opalescence of the test solution is less/more than the standard solution. If the opalescence of the test solution has been less than the standard opalescence, the sample will pass the limit test.

RESULT

A limit test for sulphate was performed.

EXPERIMENT NO 12

OBJECT: To perform the identification tests for the given sample of ammonium chloride.

REFERENCES

Parle A., "Pharmaceutical Chemistry 1 Laboratory Manual", CBS Publishers and Distributors Pvt. Ltd., Ed Ist, 2008, pp 59.

REQUIREMENTS

Chemical required: Ammonium chloride, sodium hydroxide, red litmus paper, sodium cobalt nitrite, nitric acid, silver nitrate solution, dilute ammonia solution, potassium dichromate, sulphuric acid, diphenyl carbazide solution.

Apparatus required: test tubes, test tube stands, test tube holders, glass rod.

THEORY

Any process that can provide a qualitative determination of the ions present in a simple inorganic compound is based upon knowledge of acid/base chemistry, redox chemistry, and solubility. In this regard, the identification of a single pure compound is therefore very much simpler than the identification of a mixture. This experiment deals only with the identification of simple compounds, ie those that contain only one cation and one anion.

CEDURE

STANDARD Take 1mL of 0.1089 w/v of Na_2SO_4 or K_2SO_4 as per I.P. in Nessler cylinder. Add 2 mL of dilute Hydrochloric acid. Make up the volume up to 45 ml with distilled water. Add in this solution of 5 mL of Barium sulphate reagent. Stirrer the solution with a glass rod and allow to stand for 5 minutes.

TEST

Dissolve the specific quantity of test substances in 10 mL of tap water. Add 2 ml of dilute Hydrochloric acid. Makeup the volume up to 45 ml with distilled water. Add in this solution of 5 mL of Barium sulphate reagent. Stirrer the solution with a glass rod and allow to stand for 5 minutes.

OBSERVATION

The opalescence of the test solution is less/more than the standard solution. If the opalescence of the test solution has been less than the standard opalescence, the sample will pass the limit test.

RESULT

Limit test for sulphate was performed

EXPERIMENT NO -13

OBJECT: To perform the identification test of ammonium chloride.

REFERENCES

Parle A., "Pharmaceutical Chemistry 1 Laboratory Manual", CBS Publishers and Distributors Pvt. Ltd., Ed Ist, 2008, pp 59.

REQUIREMENTS

Chemical required: Ammonium chloride, sodium hydroxide, red litmus paper, sodium cobalt nitrite, nitric acid, silver nitrate solution, dilute ammonia solution, potassium dichromate, sulphuric acid, diphenyl carbazide solution.

Apparatus required: test tubes, test tube stands, test tube holders, glass rod.

THEORY

Any process that can provide a qualitative determination of the ions present in a simple inorganic compound is based upon knowledge of acid/base chemistry, redox chemistry, and solubility. In this regard, the identification of a single pure compound is therefore very much simpler than the identification of a mixture. This experiment deals only with the identification of simple compounds, ie those that contain only one cation and one anion.

PROCEDURE

REACTIONS OF AMMONIUM IONS

S.NO	TEST	OBSERVATION	INFERENCE
1	Take a few mg of sample and add sodium hydroxide solution. Heat. Bring moist red litmus paper near the mouth of the test tube.		
2	Dissolve 10 mg of sample in water. Add 1ml of freshly prepared 10% w/v solution of sodium cobalt nitrite.		

REACTION 0F CHLORIDES

S. NO	TEST	OBSERVATION	INFERENCE
1	Dissolve 2mg of sample in 2ml of water. Acidify with dilute nitric acid. Add 0.5 ml of silver nitrate solution. Shake and allow to stand addammonia + nitric acid.		

2	Mix 10 mg sample with 0.2 gm of potassium dichromate in a test tube.Add 1 ml of sulfuric acid. Place a filter paper strip, moistened with 0.1 ml of diphenyl carbazide solution, over the mouth of the test tube.		

RESULT: An identification test of ammonium chloride was performed.

EXPERIMENT NO -14

OBJECT: To perform the identification test of sodium chloride.

REFERENCE

1. Singh H.R., Kapoor V.K. "Practical Pharmaceutical chemistry", Vallabh Prakashan, Ed Ist, 2008, pp 19-20.
2. Chatwal GR, "Pharmaceutical chemistry inorganic" Himalaya publishing house, Ed 5th, 2010, pp 256-257

REQUIREMENTS

Chemical required: Sodium chloride, Potassium carbonate, potassium antimonite solution, acetic acid, magnesium uranyl acetate solution, nitric acid, silver nitrate solution, dilute ammonia solution, potassium dichromate, sulphuric acid, etc

Apparatus required: Volumetric flask, conical flask, Burette, Pipette, Glass rod.

THEORY

Any process that can provide a qualitative determination of the ions present in a simple inorganic compound is based upon knowledge of acid/base chemistry, redox chemistry, and solubility. In this regard, the identification of a single pure compound is therefore very much simpler than the identification of a mixture. This experiment deals only with the identification of simple compounds, ie those that contain only one cation and one anion.

PROCEDURE

REACTIONS OF SODIUM IONS

S.NO	TEST	OBSERVATION	INFERENCE
1	Take 2 ml of the test solution, add 2 ml of 15% solution of potassium carbonate. Heat to boiling. Add 4 ml of potassium antimonite solution. Heat to boiling. Cool in ice.		
2	To 2 ml of the test, solution add 0.5 ml of 1 M acetic acid followed by a large excess of magnesium uranyl acetate solution.		

REACTION 0F CHLORIDES

S. NO	TEST	OBSERVATION	INFERENCE
1	Dissolve 2mg of sample in 2ml of water. Acidify with dilute nitric acid. Add 0.5 ml of silver nitrate solution.Shake and allow to stand.add ammonia+nitric acid.		
2	Mix 10 mg sample with 0.2 gm of potassium dichromate in a test tube..Add 1 ml of sulfuric acid. Place a filter paper strip, moistened with 0.1ml of diphenyl carbazide solution,over the mouth of the test tube.		

RESULT:

An identification test of sodium chloride was performed.

EXPERIMENT NO -15

OBJECT: To perform the identification test of sodium bicarbonate.

REFERENCE

3. Singh H.R., Kapoor V.K. "Practical Pharmaceutical chemistry", Vallabh Prakashan, Ed Ist, 2008, pp 19-20.
4. Chatwal GR, "Pharmaceutical chemistry inorganic" Himalaya publishing house, Ed 5th, 2010, pp 256-257

REQUIREMENTS

Chemical required: Sodium bicarbonate, Potassium carbonate, potassium antimonite solution, acetic acid, magnesium uranyl acetate solution, nitric acid, silver nitrate solution, dilute ammonia solution, potassium dichromate, sulphuric acid, etc

Apparatus required: Volumetric flask, conical flask, Burette, Pipette, Glass rod.

THEORY

Any process that can provide a qualitative determination of the ions present in a simple inorganic compound is based upon knowledge of acid/base chemistry, redox chemistry, and solubility. In this regard, the identification of a single pure compound is therefore very much simpler than the identification of a mixture. This experiment deals only with the identification of simple compounds, ie those that contain only one cation and one anion.

PROCEDURE

REACTIONS OF SODIUM IONS

S.NO	TEST	OBSERVATION	INFERENCE
1	Take 2 ml of the test solution, add 2 ml of 15% solution of potassium carbonate. Heat to boiling. Add 4 ml of potassium antimonite solution. Heat to boiling. Cool in ice.		
2	To 2 ml of the test, solution add 0.5 ml of 1 M acetic acid followed by a large excess of magnesium uranyl acetate solution.		

REACTION 0F BICARBONATES

S. NO	TEST	OBSERVATION	INFERENCE
1	Boil the aqueous solution of the sample.		
2	To the aqueous solution of the sample add magnesium sulphate		

RESULT:

An identification test of sodium bicarbonate was performed.

EXPERIMENT NO -16

OBJECT: To perform the identification test of magnesium sulphate.

REFERENCE

1. Singh H.R., Kapoor V.K. "Practical Pharmaceutical chemistry", Vallabh Prakashan, Ed Ist, 2008, pp 19-20.
2. Chatwal GR, "Pharmaceutical chemistry inorganic" Himalaya publishing house, Ed 5th, 2010, pp 256-257

REQUIREMENTS

Chemical required: Magnesium sulphate, 2 M ammonium chloride, 0.25 M disodium hydrogen phosphate, dilute ammonia solution lead acetate solution, ammonium acetate solution, potassium dichromate, dilute hydrochloric acid, etc

Apparatus required: Volumetric flask, conical flask, Burette, Pipette, Glass rod.

THEORY

Any process that can provide a qualitative determination of the ions present in a simple inorganic compound is based upon knowledge of acid/base chemistry, redox chemistry, and solubility. In this regard, the identification of a single pure compound is therefore very much simpler than the identification of a mixture. This experiment deals only with the identification of simple compounds, ie those that contain only one cation and one anion.

PROCEDURE

REACTIONS OF MAGNESIUM IONS

S.NO	TEST	OBSERVATION	INFERENCE
1	Dissolve 15 mg of sample in 2 ml of water. Add 1 ml of dilute ammonia solution.		
2	Add 1ml of 2 M solution of Ammonium chloride.		

REACTION 0F SULPHATE

S. NO	TEST	OBSERVATION	INFERENCE
1	Dissolve 50mg of sample in 5 ml of water. Acidify with dilute hydrochloric acid. And 1 ml of barium chloride.		
2	Dissolve 50 mg sample with 5 ml of water. Add 2 ml of lead acetate solution. Divide into two parts. • To the first part add ammonium acetate solution. • To the second part add sodium hydroxide solution.		

RESULT:

An identification test of magnesium sulphate was performed.

EXPERIMENT NO -17

OBJECT: To perform the identification test of barium sulphate.

REFERENCE

1. Singh H.R., Kapoor V.K. "Practical Pharmaceutical chemistry", Vallabh Prakashan, Ed Ist, 2008, pp 19-20.
2. Chatwal GR, "Pharmaceutical chemistry inorganic" Himalaya publishing house, Ed 5th, 2010, pp 256-257

REQUIREMENTS

Chemical required: Barium sulphate, barium chloride solution, dilute hydrochloric acid, silver nitrate solution,dilute ammonia solution, potassium dichromate,sulphuric acid, etc

Apparatus required: Test tube, test tube stand, test tube holder, Pipette, Glass rod.

THEORY

Any process that can provide a qualitative determination of the ions present in a simple inorganic compound is based upon knowledge of acid/base chemistry, redox chemistry and solubility. In this regard, the identification of a single pure compound is therefore very much simpler than the identification of a mixture. This experiment deals only with the identification of simple compounds, ie those that contain only one cation and one anion.

PROCEDURE

REACTIONS OF BARIUM IONS

S.NO	TEST	OBSERVATION	INFERENCE
1	Take 1ml of filtrate, acidify with dilute HCl +add 0.2 ml of barium chloride solution.		
2	Add 0.1 ml of iodine solution to the suspension obtained in the above test. Add stannous chloride solution dropwise. Boil the mixture.		

REACTION 0F SULPHATE IONS

S. NO	TEST	OBSERVATION	INFERENCE
1	Dissolve 50mg of sample in 5 ml of water. Acidify with dilute hydrochloric acid. Add 1 ml of barium chloride.		
2	Dissolve 50 mg sample with 5 ml of water. Add 2 ml of lead acetate solution. Divide into two parts. • To the first part add ammonium acetate solution. • To the second part add sodium hydroxide solution.		

RESULT:

An identification test of barium sulphate was performed.

EXPERIMENT NO -18

OBJECT: To perform the identification test of iodine.

REFERENCE

Singh H.R., Kapoor V.K. "Practical Pharmaceutical chemistry", Vallabh Prakashan, Ed Ist, 2008, pp 19-20.

Chatwal GR, "Pharmaceutical chemistry inorganic" Himalaya publishing house, Ed 5th, 2010, pp 256-257

REQUIREMENTS

Chemical required: Iodine, starch solution

Apparatus required: Volumetric flask, conical flask, Burette, Pipette, Glass rod.

THEORY

Any process that can provide a qualitative determination of the ions present in a simple inorganic compound is based upon knowledge of acid/base chemistry, redox chemistry, and solubility. In this regard, the identification of a single pure compound is therefore very much simpler than the identification of a mixture. This experiment deals only with the identification of simple compounds, i.e. those that contain only one cation and one anion.

PROCEDURE

S.NO	TEST	OBSERVATION	INFERENCE
1	• Heat the sample gently in a test tube. • Allow it to condense.		
2	Prepare a saturated solution of iodine. Add starch solution. • Heat • Cool		

RESULT:

Identification test of iodine was performed.

EXPERIMENT NO -19

OBJECT: To perform assay of iodine IP.

REFERENCE

1. Singh H.R., Kapoor V.K. "Practical Pharmaceutical chemistry", Vallabh Prakashan, Ed Ist, 2008, pp 19-20.
2. Chatwal GR, "Pharmaceutical chemistry inorganic" Himalaya publishing house, Ed 5th, 2010, pp 256-257

STANDARDS

Iodine contains nlt 99.5% and nmt 100.5% of I.

REQUIREMENTS

Chemical required: Iodine , potassium iodide, 2 M acetic acid, 0.1 M sodium thiosulphate solution,starch solution

Apparatus required: Iodine flask, burette, pipette,beaker, etc.

THEORY

This is an iodometric type of oxidation-reduction titration. In this titration iodine solution is used as oxidizing agent while sodium thiosulphate solution is used as reducing agent.

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

PROCEDURE

Weigh accurately about 0.2 gm of iodine by method of difference. Transfer it to an iodine flask containing 1 gm of potassium iodide and 2 ml of water. Add 1 ml of 2M acetic acid. Dissolve completely and add 50 ml of water. Titrate with 0.1 M sodium thiosulphate solution. When the solution in the conical flask becomes pale yellow add 2ml of starch solution.Continue titration until it becomes colourless. Note the burette reading.

RESULT:

The percentage purity of I in the given sample of iodine is % w/w.

EXPERIMENT NO 20

OBJECT: To perform limit test of iron in given sample.

REFERENCE:-

Singh H.R., Kapoor V.K. "Practical Pharmaceutical chemistry", Vallabh Prakashan, Ed Ist, 2008, pp 58.

REQUIREMENTS

Chemical required: Thioglycolic acid, citric acid, Ammonia solution, ferric ammonium sulphate.

Apparatus required: Measuring cylinder, glass rod, pipette and Nessler's cylinder.

THEORY

Limit test are quantitative or semi-quantitative test designed to identify and control small quatity of impurities which are likely to be present in the substance**.**

The limit test of iron is based on the reaction between iron and thioglycolic acid in the presence of citric acid in a ammonical solution. Citric acid prevents precipitation of iron with Ammonia. A deep reddish purple colour is formed.

Ferrous thioglycolate is colourless in acidic medium but in alkaline medium it gives purple colour.

$$2HSCH_2COOH + Fe_3+ \rightarrow Fe\ (HSCH_2COO)_2 + 2H^+$$

PROCEDURE

STANDARD

Take standard 1.5 ml of Iron solution. Add 1.5 ml of iron free citric acid to this solution and 1.5 ml of thioglycollic acid and make the solution alkaline. Volume make up to 50 ml. Stare the solution allow to stand for 5 minutes.

TEST

Dissolve specific quantity of substances being examined dissolved in water. Add 1.5 ml of iron free citric acid to this solution and 1.5 ml of thioglycollic acid and make the solution alkaline. Volume make up to 50 ml. Stare the solution allow to stand for 5 minutes

RESULT

Limit test for Iron was performed.

EXPERIMENT NO 21

OBJECT: To perform limit test of heavy metals in given sample of sodium chloride.

REFERENCE:-

Singh H.R., Kapoor V.K. "Practical Pharmaceutical chemistry", VallabhPrakashan, Ed Ist, 2008, pp 58.

REQUIREMENTS

Chemical required: Thioglycolic acid, citric acid, Ammonia solution, ferric ammonium sulphate.

Apparatus required: Measuring cylinder, glass rod, pipette and Nessler's cylinder.

THEORY

Limit test of heavy metals is based on the reaction of metallic impurities with hydrogen sulfide in acidic medium to form brownish colour solution. Metals that response to this test are lead, mercury, bismuth, arsenic, antimony, tin, cadmium, silver, copper, and molybdenum. The metallic impurities in substances are expressed as parts of lead per million parts of the substance. The usual limit as per Indian Pharmacopoeia is 20

PROCEDURE STANDARD

Take 2 ml of standard lead solution and dilute to 25 ml with water. Adjust the pH between 3 to 4 by adding dilute acetic acid 'Sp' or dilute ammonia solution 'Sp. Dilute with water to 35 ml. Add freshly prepared 10 ml of hydrogen sulphide solution and dilute with water to 50 ml. Allow to stand for five minutes, view downwards over a white surface.

TEST

Weigh specific quantity of test substance, moisten with sulphuric acid and ignite on a low flame till completely charred. Add few drops of nitric acid and heat it. Allow to cool and add 4 ml of hydrochloric acid and evaporate to dryness. Moisten the residue with 10 ml of hydrochloric acid and digest for two minutes Neutralize with ammonia solution and make just acid with acetic acid. Adjust the pH between 3 to 4 and filter if necessary. Dilute with water to 35 ml. Add freshly prepared 10 ml of hydrogen sulphide solution. Dilute with water to 50 ml. Allow to stand for five minutes. View downwards over a white surface.

RESULT

Limit test for heavy metals was performed. The color produce in sample solution should not be greater than standard solution. If color produces in sample solution is less than the standard solution, the sample will pass the limit test of heavy metals and vice versa.

EXPERIMENT NO 22

OBJECT: To perform assay of hydrogen peroxide solution (20 volume) IP 1996.

REFERENCE

Parle A., "Pharmaceutical chemistry I Laboratory Mannual", CBS Publishers and distributors Pvt. Ltd, Ed Ist, 2008, pp 109-110.

STANDARDS

Hydrogen peroxide solution (20 vol.) contains not less than 5% w/v and nmt 7% w/v of H_2O_2 corresponding to about 20 times its volume of available oxygen.

REQUIREMENTS

Chemical required: H_2O_2 , potassium iodide, 1 M sulphuric acid, 0.02 M potassium permaganate.

Apparatus required: conical flask, burette, pipette, beaker, etc.

THEORY

This is an oxidation-reduction titration of permanganometery.

$$\mathbf{2\ KMnO_4 + 3\ H_2SO_4 \rightarrow K_2SO_4 +}$$

$$\mathbf{2MnSO_4 + 3H_2O + 5[O]}$$

$$\mathbf{5[H_2O_2] + [O] \rightarrow H_2O + O_2}$$

$$\mathbf{2\ KMnO_4 + 3\ H_2SO_4 + 5\ H_2O_2 \rightarrow}$$

$$\mathbf{K_2SO_4 + 2MnSO_4 + 8H_2O + 5[O]_2}$$

PROCEDURE

Rinse the pipette with the dil. Hydrogen peroxide solution. Using pipette transfer 1 ml of dil. Hydrogen peroxide solution to dry and clean conical flask. Add 20 ml of 1 M sulphuric acid. Rinse the burette with 0.002 M potassium permagnetane and fill it with this on up to zero mark. Titrate it with potassium permagnete solution until the solution become pink. Note the burette reading.

RESULT:

The percentage purity of H_2O_2 in the given sample of iodine is % w/v.

EXPERIMENT NO -23

OBJECT: To perform assay of ammonium chloride IP 1996.

REFERENCE

Parle A., "Pharmaceutical chemistry I Laboratory Mannual", CBS Publishers and distributors Pvt. Ltd, Ed Ist, 2008, pp 86-87.

STANDARDS

Ammonium Chloride contains not less than 99.00% and not more than 100.5% of NH_4Cl

.

REQUIREMENTS

Chemical required: Ammonium chloride ,formaldehyde solution, potassium iodide,

0.1 M sodium hydroxide solution.

Apparatus required: conical flask, burette, pipette, beaker, etc.

THEORY

This is an alkalimetric titration.

$$\mathbf{NH_4Cl + H_2O \rightarrow NH_4OH + HCl}$$

$$\mathbf{NH_4OH + HCHO \rightarrow CH_2{=}NH + 2H_2O}$$

$$\mathbf{HCl + NaOH \rightarrow NaCl + H_2O}$$

PROCEDURE

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

RESULT:

The percentage purity of ammonium chloride in the given sample is % w/w.

EXPERIMENT NO -24

OBJECT: To prepare and standardize 200 ml of 0.1 M of silver nitrate solution.

REFERENCE

Parle A., "Pharmaceutical chemistry I Laboratory Mannual", CBS Publishers and distributors Pvt. Ltd, Ed Ist, 2008, pp 117-118.

REQUIREMENTS

Chemical required: Silver nitrate, sodium chloride, acetic acid ,methanol, eosin solution.

Apparatus required: conical flask, burette, pipette, beaker, etc.

THEORY

Mohr method of determination of chlorides by titration with silver nitrate is one of the oldest titration methods still in use - it was researched and published by Karl Friedrich Mohr in 1856. The idea behind is very simple - chlorides are titrated with the silver nitrate solution in the presence of chromate anions. End point is signalled by the appearance of the red silver chromate. Intense yellow colour of chromate may make detection of first signs of formation of red silver chromate precipitation difficult. As some excess of silver must be added before precipitate starts to form, if concentration of titrant is below 0.1M, we may expect singificant positive error. To correct for this error we can determine a blank, titrating a solution of the indicator potassium chromate with standard silver nitrate solution. To make result more realistic we can add small amount of chloride free calcium carbonate to the solution to imitate the white silver precipitate.

PROCEDURE

Dissolve 2.75 g of ceric ammonium nitrate in 1 N nitric acid to obtain 100 ml of solution, and filter. Standardize the solution as follows.

Accurately measure 10 ml of freshly standardized 0.1 N ferrous ammonium sulfate VS into a flask, and dilute with water to about 100 ml. Add 1 drop of nitrophenanthroline TS, and titrate with the ceric ammonium nitrate solution to a colorless endpoint.

RESULT:

The exact molarity of the prepared silver nitrate solution is M.

EXPERIMENT NO 25

OBJECT: To perform assay of Magnesium sulphate IP 1996.

REFERENCE

Parle A., "Pharmaceutical chemistry I Laboratory Mannual", CBS Publishers and distributors Pvt. Ltd, Ed Ist, 2008, pp 139-140.

STANDARDS

Magnesium sulphate contains not less than 99.00% and not more than 100.5% of $MgSO_4$.

REQUIREMENTS

Chemical required: Ammonium chloride ,strong ammonia, ammonium chloride solution.

Apparatus required: conical flask, burette, pipette, beaker, etc.

THEORY

This is a complexometric titration (Direct titration).

PROCEDURE

Your unknown for this experiment is a solution. When you obtain your unknown, you need to quantitatively transfer it to a 100 mL volumetric flask and dilute it to the mark, resulting in the "prepared" unknown solution. You are to report the results of this "prepared" unknown. Transfer exactly 10 mL of the prepared unknown solution into 3 or 4 Erlenmeyer flasks. Add approximately 15 mL of pH 10 buffer in the hood and 20 or 25 mL of distilled, deionized water to each flask. Add a few crystals of Eriochrome Black T indicator -- it is crucial that you only add enough indicator to produce a light, wine-red color. Titrate each solution with your standardized EDTA solution to a clear blue color. .Report your results as percent magnesium (% w/v) in your "prepared" unknown sample. Also report the 95% confidence interval.

RESULT:

The percentage purity of magnesium sulphate in the given sample is % w/w.

EXPERIMENT NO 26

OBJECT: To perform assay of Calcium gluconate IP 1996.

REFERENCE

Parle A., "Pharmaceutical chemistry I Laboratory Mannual", CBS Publishers and distributors Pvt. Ltd, Ed Ist, 2008, pp 143-144.

STANDARDS

Calcium gluconate contains not less than 98.5% and not more than 102.0% of $C_{12}H_{22}CaO_4.H_2O$.

REQUIREMENTS

Chemical required: Calcium gluconate ,strong ammonia, magnesium sulphate,EDTA solution.

Apparatus required: conical flask, burette, pipette, beaker, etc.

THEORY

This a replacement type of complexometric titration.Addition of magnesium sulphate ensure sharp change in colour.

PROCEDURE

Weigh accurately about 0.5 g of the dried sample and dissolve in 5 ml of dilute hydrochloric acid. Add 50 ml of water, 25 ml of sodium hydroxide TS and about 0.1 g of 2-hydroxy-1-(2'-hydroxy-4'-sulfo-1'-naphthylazo)-3-naphthoic acid. Titrate with 0.05 M EDTA immediately. At the end-point, the red colour changes completely to blue. Each ml of 0.05 M EDTA is equivalent to 22.42 mg of C12H22CaO14 · H2O.

RESULT:

The percentage purity of magnesium sulphate in the given sample is % w/w.