Date 14-12-21

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Aim :-

To perform the limit test for chlorides of the given test sample.

Reference :- Dr. Gupta Jyoti, Sandya Mohit, Groover Madhuri, P'ceutical Inorganic Chemistry, Nirali Prakashan, Page no.- 14 -15

Requirement :-

(a) Glasswares : Nessler's cylinder, measuring cylinder and glass rod.

(b) Chemicals : Dil. Nitric acid (10%), 0.1 M silver chloride, distilled water.

The chloride limit test is designed to determine the allowable limit of chloride present in a sample.

Principle :

Limit test of chloride is based on the precipitation reaction. The precipitates of chloride develop on reaction of soluble chloride with silver nitrate in the presence of dilute nitric acid to form silver chloride, which appears as solid particles (opalescence) in the solution. The intensity of turbidity depends on the amount of chlorides present in the test substance.

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

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Reagent Preparations

- Dil. Nitric Acid : 106 ml conc. HNO_3 is diluted to 1000 ml with water.
- Silver Nitrate Solution : 5 g of $AgNO_3$ is dissolved in 100 ml of water.
- Standard Sodium Chloride solution :
  Dissolve 0.05845 g of NaCl in 100 ml distilled water.
- Test sample :

(i) Dextrose : Dissolve 1 g in 10 ml distilled water to make the the test sample.

(ii) Sodium Bicarbonate : Dissolve 1 g in 10 ml distilled water.

Procedure :

Test Sample	Standard compound	Reasons
• 1 ml volume of compound / test sample is dissolved in water or solution is prepared as directed in the pharmacopoeia and	• Take 1 ml of 0.05845 % w/v solution of sodium chloride in Nessler's cylinder.	• The aqueous solution will leach out all the chloride ions present in the sample and make them ready to react with silver nitrate.

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transferred in Nesslers' cylinder	–	
• Add 10 ml of dil. nitric acid.	• Add 10 ml of dil. nitric acid.	• Dil. nitric acid is added in the limit test of chloride to make the solution acidic and helps silver chloride precipitate to make solution turbid at the end of process.
• Dilute to 50ml in Nesslers' cylinder.	• Dilute to 50 ml in Nesslers' cylinder	• For comparison of opalescence, equal volume of both is taken.
• Add 1 ml of 0.1M $AgNO_3$ solutions, stir properly and keep aside for 5min	Add 1ml of 0.1M $AgNO_3$ solution, stir properly and keep aside for 5 min.	The Ag^+ ions will react with Cl^- ions to form opalescence of silver chloride. $Ag^+ + Cl^- \rightarrow AgCl$.
Observe the opalescence/turbidity	Observe the opalescence/turbidity	Compare after 5min.

Conclusion:

If opalescence produced in the sample solution is less than the standard solution, the sample will pass the limit test of chloride and vice versa.

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