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Diploma in Pharmacy 1st Year

Pharmaceutical Chemistry

Experiment

To perform limit test for chloride in given compound

Aim:

To perform limit test for chloride in given compound.

Reference :

‘ Dr. Gupta G.D. , Dr. Sharma Shailish , Kaur Baljeet ’ “Practical Manual of Pharmaceutical Chemistry” Published by Nirali Prakashan, Page no 5 - 8

Requirements:

Apparatus Required: Nessler's cylinder, glass rod, beaker, dropper, volumetric flasks (100 ml), pipette (10 ml), etc.

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

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, and colour is affected by presence of impurities present in substance, variation in time and method of performance of test.
- Limit test for chloride is based on reaction of silver nitrate with soluble chlorides to form precipitate of silver chloride which is insoluble in dilute nitric acid.
- The extent of precipitate depends upon the amount of silver chloride formed or amount of chloride ions present in the substance.
- The opalescence produced is compared with standard opalescence from standard solution containing a fixed amount of chloride under same experimental

conditions. The opalescence produced by test substance should be less intense than that of standard substance to pass the limit test for chloride.

Procedure :

- **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.
- **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's cylinder. Add 10 ml of dilute nitric acid, except when nitric acid is used in the preparation of the solution, dilute to 50 ml with water and add 1 ml of 0.1 M silver nitrate. Stir immediately with a glass rod and allow to stand for 5 minutes protected from light.
- **Preparation of standard opalescence :** Take a mixture of 10.0 ml of chloride standard solution (25 ppm Cl) and 5 ml of water in a Nessler's cylinder. Add 10 ml of dilute nitric acid, dilute it to 50 ml with water and add 1 ml of 0.1 M silver nitrate. Stir immediately with a glass rod and allow to stand for 5 minutes protected from light.
- **Observation :** After 5 minutes, view both standard and test cylinder transversely against a black background.

Result: The colour obtained in the test solution is lighter than the standard solution, It means limit test is passed.

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