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LIMIT TEST

Definition

Limit test is semi quantitative test designed to identify and control small amounts of impurity which is likely to be present in pharmaceuticals.

In these tests, the test opalescence / turbidity / color/ stain produced by the reaction of specified amount of impurity in the test sample with the reagent is compared with the standard opalescence / turbidity / color/ stain produced by the reaction of known amount of impurity [standard] with the reagent.

LIMIT TEST FOR CHLORIDE

AIM

To perform the limit test for chloride on a given sample as per Indian pharmacopoeia and report on its standard.

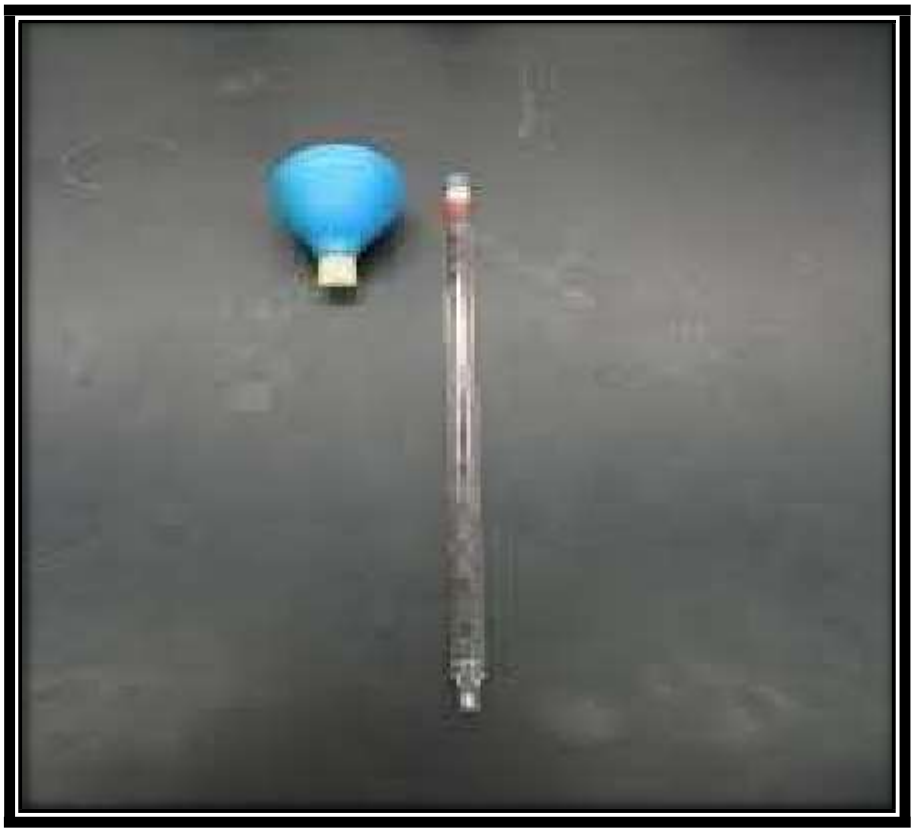
Apparatus required:

Nessler's cylinder

Glass rod-2

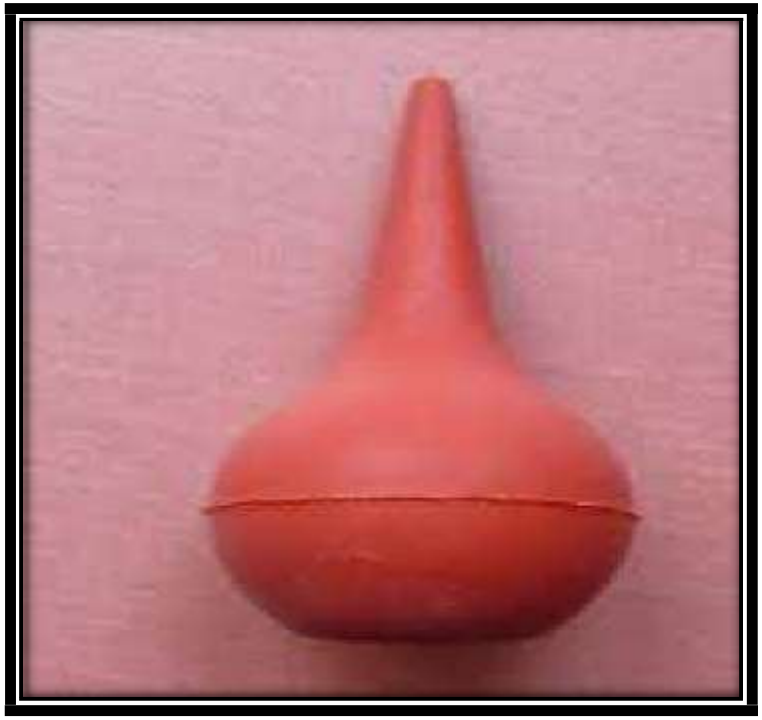
Measuring cylinder

Pipette



Dropper

Rubber stopper



Chemicals Required

Dilute nitric acid:

Dissolve 106 ml of concentrated Nitric acid in 1000 ml of water.

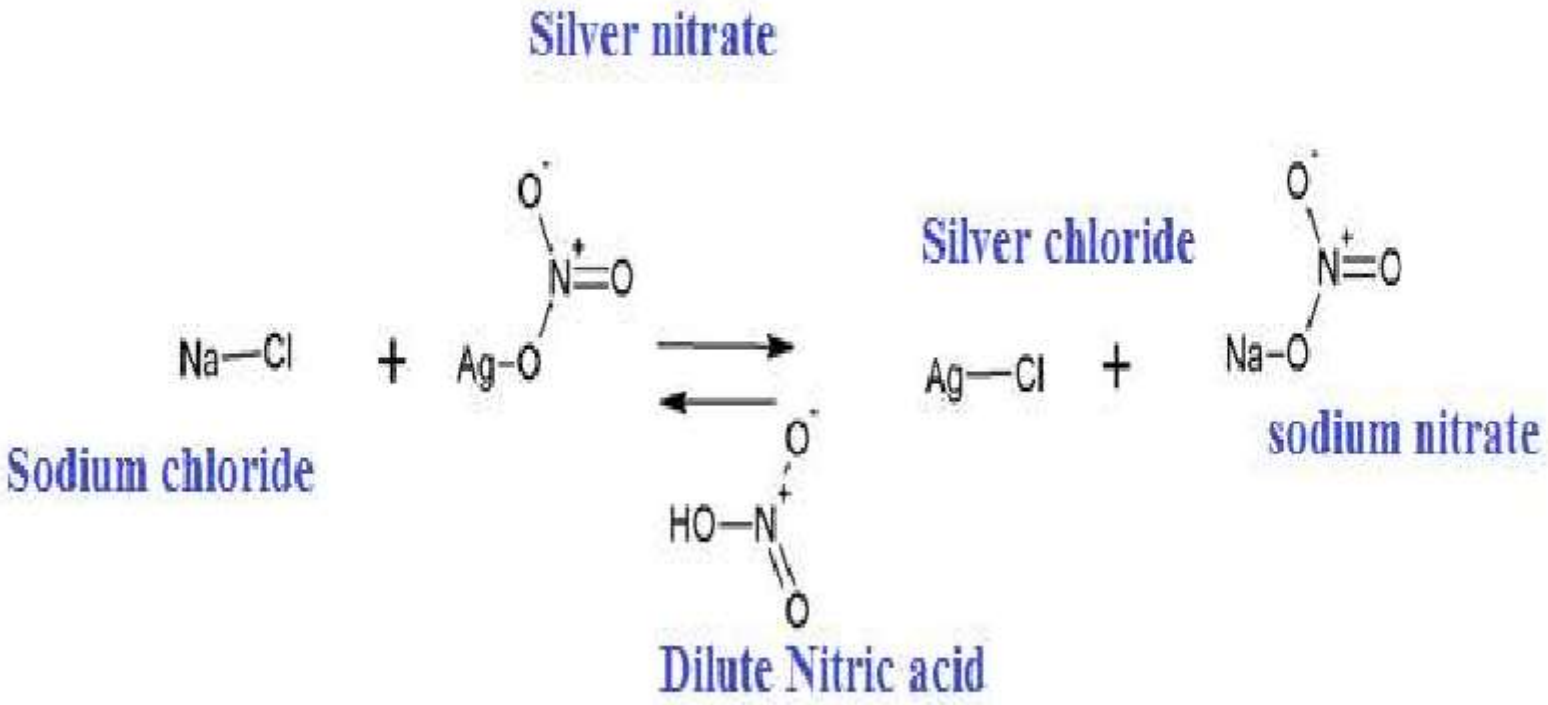
5 % w/v Silver nitrate solution:

Dissolve 5 g of silver nitrate in 100 ml of distilled water.

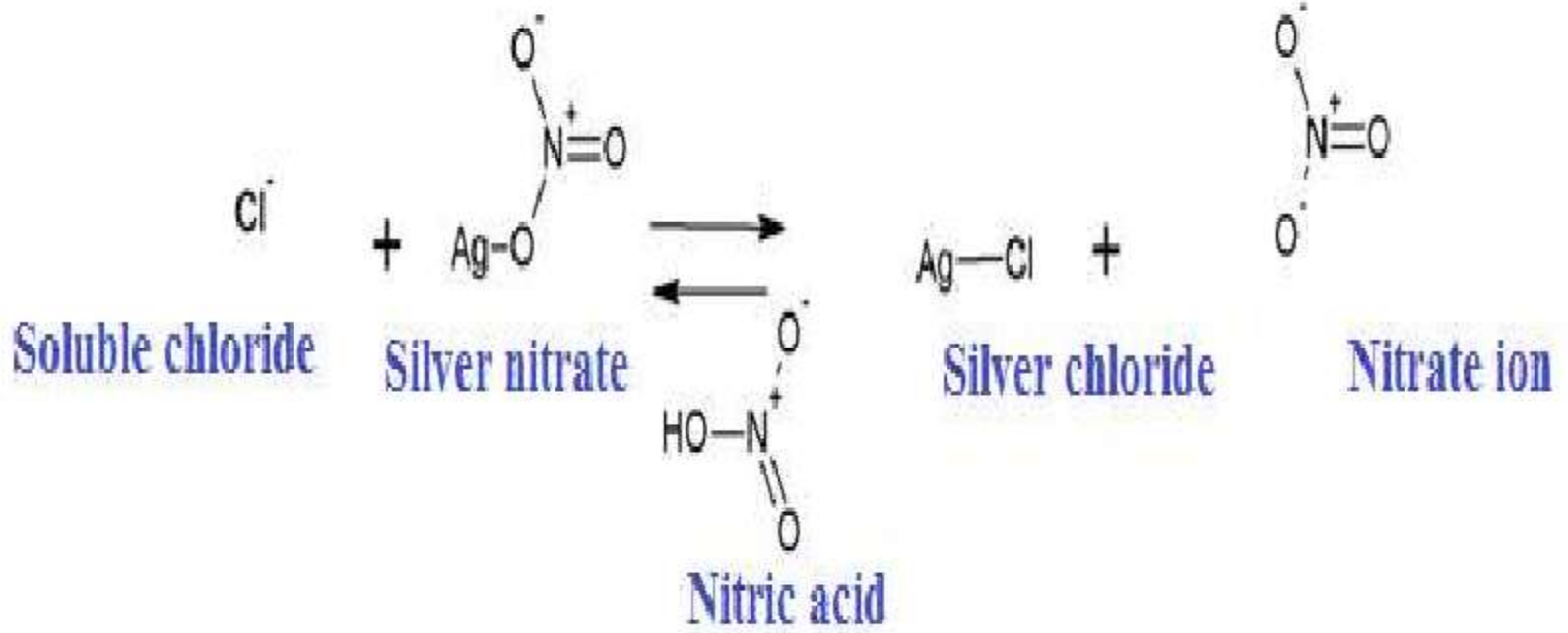
Standard sodium chloride solution: 0.05845%

Dissolve 0.05845 g of sodium chloride in 100ml of distilled water.

Principle -Reaction Standard:



Reaction Test :



Nitric acid - why

- It extracts a common ion effect by furnishing nitrate ions and thereby suppression of dissociation of silver chloride.
- Dilute nitric acid is used to dissolve other impurities if present.

Common ion effect:

It is defined as the suppression of the degree of dissociation of a weak electrolyte containing a common ion.

Standard	Test
Pipette out 1 ml of standard sodium chloride solution into the Nessler's cylinder marked as standard	Dissolve the specified quantity of given sample in distilled water in Nessler's cylinder marked as test
Add 10 ml of dilute nitric acid	Add 10 ml of dilute nitric acid
Dilute it with distilled water to 50 ml	Dilute it with distilled water to 50 ml

Finally add 1ml of silver nitrate solution	**Finally add 1ml of silver nitrate solution**
Immediately stir with a glass rod and allow it to stand for five minutes	Immediately stir with a glass rod and allow it to stand for five minutes
Observe it under black back ground	Observe it under black back ground

- Distilled water must be used because chloride present in the tap water will interfere the result.
- Same glass rod should not be used because it will affect your observation.
- Silver nitrate is photosensitive store it in amber colour bottle.

Black dots will be produced if silver nitrate is in contact with skin.

Black spots

Silver nitrate gets into the skin, and there the skin reacts with the silver, so it becomes silver oxide particles. It doesn't dissolve in anything but strong acids. it's only in the dead layers on top, not in the lower layers where it would be permanent, like a tattoo. Once the stain becomes fully developed, it is usually impossible to remove, but it will wear off naturally.

- **Sample I [Pass Sample] :**
- **Observation:**
- The opalescence produced in the test solution is lesser than standard solution.
- **Inference:**
- The given substance passes the limit test for chloride as per Indian pharmacopoeia when compared with that of a standard substance.

- **Sample II [Fail Sample] :**
- **Observation:**
- The opalescence produced in the test solution is more than standard solution.
- **Inference:**
- The given substance fails the limit test for chloride as per Indian pharmacopoeia when compared with that of a standard substance.

Opalescence : It is a type of dichroism seen in highly dispersed systems with little opacity