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Diploma in Pharmacy 1st Year
Pharmaceutical Chemistry
Experiment
To perform the limit test for heavy metals.

Aim:

To perform the limit test for heavy metals.

Reference :

‘ Dr. Gupta G.D. , Dr. Sharma Shailish , Kaur Baljeet ’ “Practical Manual of Pharmaceutical Chemistry” Published by Nirali Prakashan, Page no 12 - 15

Requirements:

Apparatus Required : Analytical balance, Glass rod, Nessler's cylinder,

Chemicals Required : dil. acetic acid solution, dil ammonium solution, hydrogen sulphide solution, sulphuric acid, hydrochloric acid,standard lead solution,distilled water

Theory:

- This limit test is carried out for determining the content of metallic impurities coloured by sulphide ion, under specific conditions. The monographs prescribe the limit for heavy metals in terms of amount of lead parts per million of the substance (by weight). This has been determined by visually comparing the colour produced by the substance with that of a control prepared from a standard lead solution.
- Limit tests for heavy metals are based on the reaction between a solution of a heavy metal and a saturated solution of H_2S in an acidic medium. A reddish/black colour result is compared with the standard solution of lead nitrate solution.

$$(Pb + H_2S \rightarrow PbS + H_2)$$

Procedure:

Following are the methods which are prescribed in monographs and can be used for determining the amount of heavy metals:

→ **Standard Solution :** 2ml of standard lead solution is pipetted out in 50ml Nessler cylinder and diluted up to 25ml with water. The pH is adjusted between 3-4 using dilute acetic acid or dilute ammonia solution. After pH adjustment the solution is diluted up to 35ml with water.

→ **Test Solution :** 25ml of test solution is prepared in a 50ml Nessler cylinder and pH is adjusted between 3-4 using dilute acetic acid or dilute ammonia solution. After pH adjustment the solution is diluted up to 35ml with water.

→ **Procedure :** 10ml of freshly prepared hydrogen sulphide solution is added into both the cylinders containing standard and test solution and diluted up to 50ml with water. After dilution, the solution is kept aside over a white surface for 5 minutes and viewed downwards. The test solution colour is lighter than the standard solution colour.

Observation

- The intensity of colour produced in the sample solution should not exceed that of the standard solution. If the colour intensity of the sample solution is less than the standard solution, the sample will pass the limit test of iron and vice versa.

Result: The limit test of heavy metal was performed.