CHAPTER-04

Experiment: 04

TO PERFORM AND REPORT THE LIMIT TEST FOR HEAVY METAL IN THE GIVEN SAMPLE OF SODIUM CHLORIDE AS PER INDIAN PHARMACOPOEIA

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Aim:

To perform and report the limit test for heavy metal in the given sample of sodium chloride as per I.P.

Requirements:

A. Glassware & Instruments:

a. Measuring cylinder

b. Pipette

c. Beaker

d. Nessler's cylinder

e. Glass rod

f. Analytical balance

B. Chemicals & Reagents:

1. Standard Pb Solution (20ppm Pb)
2. NaCl
3. Dextrose
4. Distilled water (DW)
5. dil. CH_3COOH
6. Pot. Iodide
7. Fe free ammonia
8. Freshly prepared H_2S solution

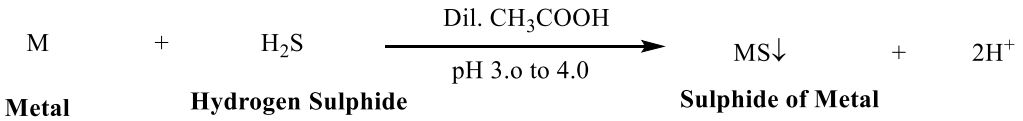
Theory:

The limit test for heavy metals is based on the reaction of metallic contaminants with hydrogen sulphide in an acidic environment to generate a solution with a brownish hue. Lead, mercury, bismuth, arsenic, antimony, tin, cadmium, silver, copper, and molybdenum respond to this test. Metallic impurities in substances are represented as part of lead per million parts of substance. The standard Indian Pharmacopoeia limit is 20.

Principle:

In the presence of dilute acetic acid, the interaction between hydrogen sulphide and certain heavy metals such as lead, iron, copper, nickel, cobalt, and bismuth results in the creation of sulphides of the respective metals. The resulting sulphides are spread in a colloidal condition and produce a solution with a brownish hue. The colour intensity produced by the test is compared to the colour intensity produced by the standard solution.

Reaction:



Procedure:

Procedure for Test Sample	Procedure for Reference Sample
➢ Accurately weigh 4 g of sodium chloride and add to labeled Nesslers cylinder (T). ➢ Add 2 ml of dilute acetic acid and mix well, then add sufficient water to produce 25 ml. ➢ Adjust the pH with dilute acetic acid or dilute ammonia solution in between 3.0 to 4.0. ➢ Dilute with water to 35 ml and mix well. ➢ Add 10 ml of freshly prepared hydrogen sulphide solution. ➢ Mix and dilute to 50 ml with water. ➢ Allow to stand for 5 minutes. ➢ View downwards over a white	➢ In to a 50 ml labeled Nesslers cylinder (R). Pipette 1.0 ml of lead standard solution (20 ppm Pb). ➢ Dilute with DW to 25 ml. ➢ Adjust the pH with dilute acetic acid or dilute ammonia solution in between 3.0 to 4.0. ➢ Dilute with distilled water about 35 ml and mix with glass rod. ➢ Add 10 ml of freshly prepared hydrogen sulphide solution. ➢ Mix and dilute to 50 ml with water. ➢ Allow to stand for 5 minutes.

surface and compare with that of standard.	

Observation:

The colour intensity produced by the NaCl test solution is……. (less/equal/greater) than that of the standard solution.

Results:

The given sample of NaCl..... (complies/does not comply) the limit test for heavy metal as per the I.P.

Viva Questions:

- Why is a limit test for heavy metals conducted?
- Write the technique for preparing standard solutions for heavy metal limit tests.
- Write the principle underlying the heavy metal limit test.
- Write the technique for preparing sodium chloride test solution.
- Write the technique for preparing potassium iodide test solution.
- Write the technique for preparing dextrose test solution.
- Explain the rationale for the following usage. i. A solution of diluted acetic acid, ii. A solution of diluted ammonia, and iii. A freshly made solution of hydrogen sulphides.
- Can hydrogen sulphides solution be replaced by another substance? Justify by way of example.

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