

Chemical Reaction:**Note: -**

- 1. Lead Standard Solution:** On the day of use, dilute 10 mL of lead nitrate stock solution with water to 100 mL. A control comparison solution prepared with 2.0 mL of lead standard solution contains, when compared to a solution representing 1.0 g of the substance under examination, the equivalent of 20 ppm lead.
- 2. Lead Nitrate Stock Solution:** Dissolve 0.1598 g of lead nitrate in 100 mL of water to which has been added 1 mL of nitric acid, then dilute to 1000 mL with water.
- 3. Dilute Acetic Acid:** Dilute 5.7 mL of glacial acetic acid to 100 mL with water.
- 4. Dilute Ammonia Solution:** Dilute 42.5 mL of strong ammonia solution to 100 mL with water.
- 5. Dilute Sodium Hydroxide:** A 5.0% w/v solution of sodium hydroxide.
- 6.** The colour developed in the sample and standard solution is compared by keeping the Nessler cylinders against dark background and observing side by side.

Practical - 4

Date: .../.../.....

Aim: To perform the limit test for heavy metals in the given sample as per I.P.

Reference:.....
.....

Requirements:

Apparatus/Equipment required:.....

Chemical required:.....
.....

Principle

The limit test for heavy metals is based on the reaction of metallic impurities with hydrogen sulphide in acidic medium; the reaction product will be the sulphides of the respective metals. The sulphides so formed are distributed in colloidal state and produce brownish or black colour solution. Metals that response to this test are lead, mercury, bismuth, arsenic, antimony, tin, cadmium, silver, copper, and molybdenum. The metallic impurities in substances are expressed as parts of lead per million parts of the substance. The usual limit as per Indian Pharmacopoeia is 20 ppm.

Procedure:

The Indian Pharmacopoeia has adopted three methods for the limit test for heavy metals.

Method I: Use for the substance which gives clear colorless solution under the specific condition. Take two 50 mL Nessler Cylinders. Label one as “Test” and the other as “Standard”.

| Test Solution | Standard Solution |
|---|--|
| Into a 50 ml Nessler cylinder place 25 ml of the solution prepared as per the test as directed in the individual monograph or dissolve the specified quantity of the substance under examination in sufficient distilled water to produce 25 ml | Take 1 mL of standard lead solution (20 ppm, Pb) and dilute to 25 ml with water |
| Adjust the pH between 3 to 4 by adding dilute acetic acid or dilute ammonia solution | Adjust the pH between 3 to 4 by adding dilute acetic acid or dilute ammonia solution |
| Dilute with water to about 35 ml and mix | Dilute with water to about 35 ml and mix |
| Add freshly prepared 10 ml of hydrogen sulphide solution and mix | Add freshly prepared 10 ml of hydrogen sulphide solution and mix |
| Dilute with water to 50 ml | Dilute with water to 50 ml |
| Keep aside for 5 min | Keep aside for 5 min |
| Observe the Turbidity | Observe the Turbidity |

*Compare the opalescence/turbidity produced by the test solution with a standard solution.

Method II: Use for the substance which do not give clear colorless solution under the specific condition. Take two 50 ml Nessler Cylinders. Label one as “Test” and the other as “Standard”.

| Test Solution | Standard Solution |
|---|---|
| <p>Weigh in a suitable crucible specific quantity of test substance, moisten with sulphuric acid and ignite carefully at a low temperature until thoroughly charred. Add 2 ml of nitric acid and 5 drops of sulphuric acid and heat cautiously until white fumes are no longer evolved.</p> <p>Ignite, preferably in a muffle furnace, at 500°C to 600 °C, until the carbon is completely burnt off. Allow to Cool, add 4 ml of hydrochloric acid, digest on a water-bath for 15 minutes, uncover and slowly evaporate to dryness on water-bath.</p> <p>Moisten the residue with 1 drop of hydrochloric acid, add 10 ml of hot water and digest for 2 minutes. Add ammonia solution dropwise until the solution is just alkaline to litmus paper, dilute to 25 ml with water.</p> | <p>Take 1 mL of standard lead solution (20 ppm, Pb) and dilute to 25 ml with water</p> |
| <p>Adjust the pH between 3 to 4 by adding dilute acetic acid or dilute ammonia solution and filter if necessary</p> | <p>Adjust the pH between 3 to 4 by adding dilute acetic acid or dilute ammonia solution</p> |
| <p>Dilute with water to about 35 ml and mix</p> | <p>Dilute with water to about 35 ml and mix</p> |
| <p>Add freshly prepared 10 ml of hydrogen sulphide solution and mix</p> | <p>Add freshly prepared 10 ml of hydrogen sulphide solution and mix</p> |
| <p>Dilute with water to 50 ml</p> | <p>Dilute with water to 50 ml</p> |
| <p>Keep aside for 5 min</p> | <p>Keep aside for 5 min</p> |
| <p>Observe the Turbidity</p> | <p>Observe the Turbidity</p> |

*Compare the opalescence/turbidity produced by the test solution with a standard solution.

Method III: Use for the substance which gives clear colorless solution in sodium hydroxide solution. Take two 50 mL Nessler Cylinders. Label one as “Test” and the other as “Standard”.

| Test Solution | Standard Solution |
|--|--|
| <p>Into a 50 mL Nessler cylinder place 25 ml of the solution prepared as per the test as directed in the individual monograph or dissolve the specified quantity of the substance under examination in sufficient in a mixture of 20 ml of distilled water and</p> | <p>Take 1 mL of standard lead solution (20 ppm, Pb) and dilute to 25 ml with water</p> |

| | |
|---|--|
| 5 ml of dilute sodium hydroxide solution | |
| Dilute with water to 50 ml and mix | Add 5 ml dilute sodium hydroxide solution and dilute with water to 50 ml and mix |
| Add 5 drops of sodium sulphide solution and mix | Add 5 drops of sodium sulphide solution and mix |
| Keep aside for 5 min | Keep aside for 5 min |
| Observe the Turbidity | Observe the Turbidity |

*Compare the opalescence/turbidity produced by the test solution with a standard solution.

Observation:

Observation: The colour produce in sample solution should not be greater than standard solution. If colour produces in sample solution is less than the standard solution, the sample will pass the limit test of heavy metals and vice versa.

Result:.....

Questions Bank

1. Write the chemical formula of sodium sulphide.
2. Write the chemical formula of Acetic acid.
3. What is the meaning of standard lead solution?
4. Write the formula of sulphuric acid.
5. What is the muffle furnace?
6. What is the full form of pH?
7. How to prepare freshly hydrogen sulphide?
8. What is the meaning of 5.0% w/v solution of sodium hydroxide.
9. What is the meaning of dilution?
10. What is the meaning of heavy metals?