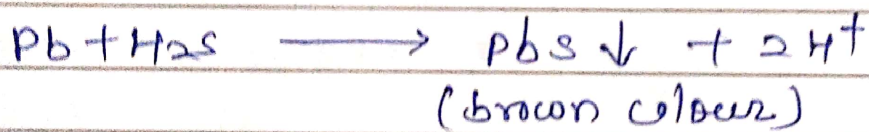


Limit test for Heavy Metal

Principle:

- It is based on the reaction of metallic impurities with H_2S in acidic medium to form brownish colour solution.
- Metals that response this test are Pb, Hg, Bi, As, Sb, Sn, Cd, Ag, Cu & Mb.
- heavy metal impurities compared, a standard colour obtained by the reaction of known quantity of lead with saturated solution of H_2S .



* Indian pharmacopoeia has adopted '3' method for limit test of heavy metals.

Method 1 : It is used for the substance which gives a clear, colourless solution under specified condition.

Method 2 : For substance which does not give a clear, colourless solution.

Method 3 : Substance which gives clear, colourless solution in $NaOH$ medium.

Procedure

Method 1

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Test solution

Standard solⁿ

1. Sample solution is prepared as per I.P. & 25 ml of solution is transferred into H.C. Transfer 1.0 ml of standard Lead solution and dilute to 25 ml in H₂O.
2. Add dil ammonia or CH₃COOH to adjust pH 3-4. — do —
3. dilute to 35 ml in distilled H₂O & mix well. — do —
4. Add 10 ml, freshly prep^d H₂S, mix & dilute to 50 ml in H₂O. — do —
5. Allow to stand for 5 min & observe. — do —

Method - 2

Test

standard

1. Take prescribe quantity of sample. 1 ml standard lead sol diluted to 25 ml in H₂O
2. moisten in H₂SO₄ & ignite on a low flame till complete charring of sample. Add dil ammonia or CH₃COOH to adjust pH 3-4.
3. Add 2-3 drops of HNO₃ & heated to 500°C & cool it. dilute to 35 ml in dist H₂O & mix well.

TestStandard

- | | |
|--|---|
| 1) Sample is dissolved in 20 ml of H ₂ O | Standard sol ⁿ is prepared by using 1.0 ml of standard Lead solution |
| 2) Add 5 ml NaOH | ↓
Add 5 ml NaOH |
| 3) Makeup vol ⁿ 50 ml | — do — |
| 4) Add 5 drop of H ₂ S sol ⁿ , mix & keep for 5 min. | Add 5 drops of sodium sulphide solution mix well & kept for 5 min |
| 5) Observe | observe |

Observation

If test colour intensity is less than standard, sample pass limit test & vice-versa.