Limit test of Heavy metal

Principle:

- □ Limit test of Heavy metals is based on the reaction of metallic impurities with Hydrogen sulfide (H₂S) or Sodium Sulfide in an acidic medium to produce metal sulphides which gives Brown color. Here lead is used to make standard solution.
- □ Heavy metal like: Cobalt, tin, Manganese, Bismuth, Antimony, Silver, Arsenic, lead etc.

Heavy Metals + H_2S / Na_2S $\xrightarrow{acidic medium}$ Sulphides of heavy metals (Brown coloration)

Procedure:

Method A & B: Method A is used for substance gives clear colorless solution under specific condition given in monograph, while Method B is for those substance which does not give clear colorless solution.

Method C & D: Method C is used for the substances which gives clear colourless solution in sodium hydroxide, while Method D is used for substance which does not gives clear colourless solution in NaOH solution.

S.No.	Test Sample	Standard Sample
1.	sample prepared according to direction of I.P. in a Nessler cylinder labelled as	Place 2 ml of standard lead solution (0.02mg lead) in a Nessler cylinder labelled as 'Standard' and dilute with distilled water to 25 ml
2.		Adjust with dil. Acetic acid or ammonia to a pH b/t 3 to 4 and dilute with water to 35 ml.
3.	Add 10 ml of freshly prepared saturated solution of hydrogen sulphide	Add 10 ml of freshly prepared saturated solution of hydrogen sulphide
4.	Make the volume to 50 ml with distilled water and mix.	Make the volume to 50 ml with distilled water and mix.
5.	Allow to stand for 5 min	Allow to stand for 5 min

Procedure for Method A

Procedure for Method B

It is similar to method A except in this case the coloured substance (Test sample) is given special treatment (sulphuric acid, ignition, nitric acid, ignition, HCl and finally digestion with water) to make it colourless before preparing its solution

Procedure for Method C

Test Sample	Standard Sample	
 Place 25 ml of the solution of the test sample prepared according to direction of I.P. in a Nessler cylinder labelled as 'Test'. 	 Place 2 ml of standard lead solution (0.02mg lead) in a Nessler cylinder labelled as 'Standard' and dilute with distilled water to 25 ml. 	
2. Add 5 ml of dil sodium hydroxide solution		
3. Add distilled water to make 50 ml and mix.		
4. Add 5 drops of sodium sulphide solution and mix.		
5. 6. Allow to stand for 5 mins and compare the color		

Procedure for Method D

□ To the cylinders containing test sample and standard solution add 2 ml of acetate buffer of pH 3.5 and mix/ to each of the cylinder add 1.2 ml thioacetamide reagent. Allow to stand for two minutes and view downwards over a white surface and compare intensity of color in both.

Observation:

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