Experiment No. 06

Aim: *To perform the limit test of Lead in the given unknown sample.*

Requirements:

(a) Glassware: Separating funnel, measuring cylinder, glass rod, dropper, beaker etc.

(b) Chemicals: Test sample, standard lead nitrate solution, dithizone extraction solution, dithizone standard solution, ammonium citrate, potassium cyanide, hydroxylamine hydrochloride, phenol red, ammonia solution, ammonium cyanide, dil. nitric acid etc.

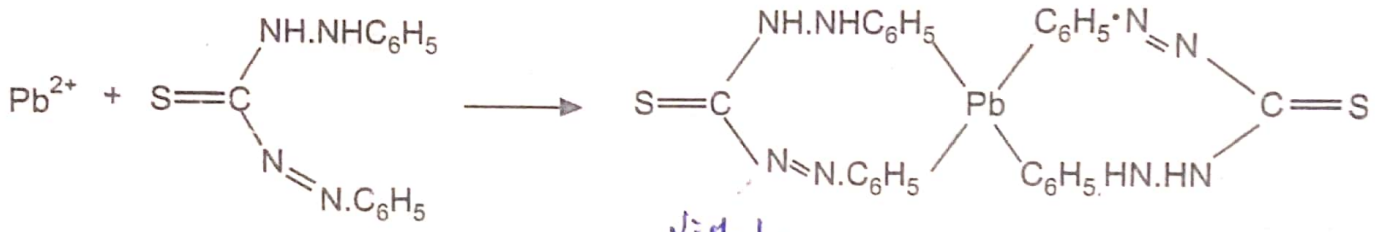
Lead is a most undesirable impurity in medical compounds and comes through use of sulphuric acid, lead lined apparatus and glass bottles use for storage of chemicals.

Common sources of lead impurities are:

1. Equipment used for manufacturing.
2. Storage container.
3. From packaging material.

Principle:

Limit test of lead is based on the reaction of lead and diphenylthiocarbazone (dithizone) in alkaline solution which leads to the formation of lead dithizone complex which is violet in colour.



Lead ion **Dithizone** violet **Lead dithizonate**

In this test, Nesslers' cylinders are not used, instead it is performed by extraction with the help of a separating funnel.

The original colour of dithizone in chloroform is green while the lead dithizonate complex is violet in colour. The intensity of the violet colour of the complex depends upon the quantity of lead present in the solution which is compared with that of the standard colour produced by treating standard solution containing definite amount of lead in the similar manner.

Intensity of this violet colour is compared in both the test and standard solution in a chloroform solvent medium.

Reagent Preparations:

- **Preparation of standard lead solution (1 ppm Pb):**
  - Dissolve 0.400 g of lead nitrate in water containing 2 ml of dilute nitric acid and add sufficient water to produce 250.0 ml. This gives standard lead solution (1% Pb).
  - Standard lead solution (1 ppm Pb) is prepared by diluting 1 volume of standard lead solution (1% Pb) to 1000 volumes with water.
- **Preparation of dithizone extraction solution:**

  Dissolve 30 mg of dithizone in 1000 ml of chloroform and add 5 ml of ethanol (95%). The solution is stored in a refrigerator. Before use, the solution is shaken with about half of its volume of 1% v/v nitric acid solution and the acid is discarded.
- **Preparation of dithizone standard solution:**

  Dissolve 10 mg of dithizone in 1000 ml of chloroform.

Procedure:

Test sample	Standard compound	Inference/ Reasons
1. A known quantity of sample solution is transferred in a separating funnel.	1. A standard lead solution is prepared equivalent to the amount of lead permitted in the sample under examination.	1. Suspected sample is taken which might have an impurity of lead.
2. Add 6 ml of ammonium citrate.	2. Add 6 ml of ammonium citrate.	2. Ammonium citrate is added to maintain optimum pH and to prevent the formation of undesired precipitates.

... (Contd.)

Test sample	Standard compound	Inference/ Reasons
3. Add 2 ml of potassium cyanide and 2 ml of hydroxylamine hydrochloride.	3. Add 2 ml of potassium cyanide and 2 ml of hydroxylamine hydrochloride.	3. Cyanide forms complexes with all the interfering metals in the solution.
4. Add 2 drops of phenol red indicator.	4. Add 2 drops of phenol red indicator.	4. It is used as an indicator to develop colour at the end of the process.
5. Make solution alkaline by adding ammonia solution.	5. Make solution alkaline by adding ammonia solution.	5. Ammonia provides basic conditions to develop red colour.
6. Extract with 5 ml of dithizone until it becomes green.	6. Extract with 5 ml of dithizone until it becomes green.	6. At least 2-3 extractions are done in separating funnel and mix all the extracts in the end.
7. Combined dithizone extracts are shaken for 30 min. with 30 ml of nitric acid and the chloroform layer is discarded.	7. Combined dithizone extracts are shaken for 30 min. with 30 ml of nitric acid and the chloroform layer is discarded.	7. Acidic layer (contains free lead Pb^{2+}) is separated from chloroform. Nitric acid make lead free from chloroform layer.
8. To the acid solution, add 5 ml of standard dithizone solution.	8. To the acid solution, add 5 ml of standard dithizone solution.	8. Violet colour appears due to complex lead dithizonate.
9. Add 4 ml of ammonium cyanide.	9. Add 4 ml of ammonium cyanide.	9. Maintain optimum pH.
10. Shake for 30 min. and observe the colour.	10. Shake for 30 min. and observe the colour.	10. Comparison of colour.

Conclusion:

If the colour produced in the sample solution is less than the standard solution, the sample will pass the limit test of lead and vice versa.