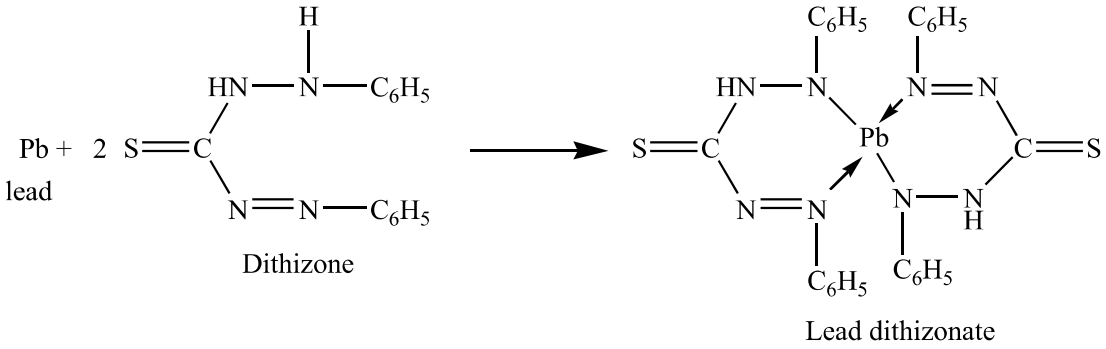
Limit test of Lead (Pb)

Principle:

- Limit test of Lead (Pb) is based on the reaction between lead (impurity) and Diphenylthiocarbazone (Dithizone) in chloroform solution and alkaline medium to form Lead-dithizone complex, which is red in color.
- Dithizone is green in color in chloroform and lead-dithizone complex is violet in color, so the resulting color at the end of process is red.



- Dithizone in chloroform, extract lead from alkaline aqueous solution as lead dithizone complex (violet in color).
- In this experiment, ammonium citrate, potassium cyanide, hydroxylamine hydrochloride are used to extract and discard and interfering metal ions (other than lead) at optimum pH in the form of complex.

Apparatus: (Separating Funnel)

Procedure:

S. No.	Test Sample	Standard Sample
1.	A known quantity of sample solution is transferred in separating funnel	A standard lead solution is prepared equivalent to the amount of lead permitted in the sample under examination.
2.	Add 6 ml of ammonium citrate	Add 6 ml of ammonium citrate
3.	Add 2 ml of potassium cyanide and 2 ml of hydroxylamine hydrochloride	Add 2 ml of potassium cyanide and 2 ml of hydroxylamine hydrochloride
4.	Add 2 ml of phenol red	Add 2 ml of phenol red
5.	Make solution alkaline by adding ammonia solution	Make solution alkaline by adding ammonia solution
6.	Extract with 5 ml of diathizone until it become green	Extract with 5 ml of diathizone until it become green
7.	Combined dithizone extract are shaken for 30 mins with 30 ml nitric acid and dithizone layer is discarded	Combined dithizone extract are shaken for 30 mins with 30 ml nitric acid and dithizone layer is discarded
8.	To the acid solution add 5 ml of standard dithizone solution	To the acid solution add 5 ml of standard dithizone solution
9.	Add 4 ml of ammonium cyanide	Add 4 ml of ammonium cyanide
10.	Shake for 30 mins and observe the color	Shake for 30 mins and observe the color

Notes:

- Ammonium citrate, potassium cyanide, hydroxylamine hydrochloride is used to make pH optimum so interference and influence of other impurities have been eliminated.
- Phenol red is used as indicator to develop the color at the end of process.

Observation:

The intensity of the color of complex will depend on the amount of lead in the solution. The color produced in the sample solution should not be greater than standard solution. If color produces in sample solution is less than the standard solution, the sample will pass the limit test of lead and vice versa.