

# Limit test for lead

## BACKGROUND-

The test as prescribed by B.P, the sodium sulphide reacts with lead salts to give a brownish colour solution and the intensity depends upon the amount of lead present in the sample, if the content of lead is high then it gives a colloidal brownish black precipitate of lead sulphide. Two solutions are prepared that is primary and auxillary just to compare under identical conditions, as elimination can be done through this process of any unknown entity. Therefore, the exact quantity of lead can be determined.

The limit test for lead followed by I.P and U.S.P prescribes the reagent to be lead and thiocarbazon.

**Aim:** Aim of this experiment is to carry out the limit test for lead of the given samples.

## REQUIREMENTS-

**Sample:** 4 gm Dextrose ( $C_6H_{12}O_6$ )

**Apparatus:** Analytical Balance

Glass Rod

Nessler's cylinder

Separating flasks

Beakers

Separating flask ring

Test tubes

**Chemicals:** Dilute acetic acid solution,

Dilute ammoniasolution,

Freshly prepared hydrogen sulphide solution,

Sulphuric acid,

Hydrochloric acid,

Standard lead solution

Distilled water

Ammonium citrate solution Sp

Hydroxyl amine hydrochloride solution Sp

Phenol-red solution

Strong ammonia solution

Potassium cyanide solution Sp

Dithizone extraction solution

1% v/v solution of nitric acid

Chloroform

Dithizone standard solution

## **PROCEDURE-**

### **Method 1**

#### **Preparation of standard solution**

The 2 g standard is dissolved in water with 7 ml of acetic acid to which 2 ml of dilute lead solution is added. Then the solution is made alkaline with ammonia and 1 ml of potassium cyanide is added. The necessary steps are done like if colour required then being added after filtration of solution is done. The solution is diluted to 50 ml with water and 2 drops of sodium sulphide is added and stirred.

#### **Preparation of test solution**

In the primary solution 4 g of substance is dissolved in water in the presence of 10 ml acetic acid. Then, the solution is made alkaline with ammonia and 1 ml of potassium cyanide is added. The solution is filtered and colouring matter is added if required, and then diluted upto 50 ml with water. At last 2 drops of sodium sulphide solution is added and stirred.

### **Method 2**

#### **Preparation of standard solution**

A specified volume of lead standard solution (1 ppm Pb) equivalent to the amount of lead permitted in the substance being examined is transferred into a separator, 6 ml of ammonium citrate solution Sp., 2 ml of hydroxylamine hydrochloride solution Sp. and two drops of phenol red solution are added and the solution is made just alkaline (red in colour) by the addition of strong ammonia solution. The solution is cooled if necessary and 2 ml of potassium cyanide solution sp. is added. Immediately the solution is extracted with several quantities, each of 5 ml of dithizone extraction solution, draining off each extract into another separating funnel, until the dithizone extraction solution retains its green colour. The combined dithizone solutions is shaken for 30 seconds with 30 ml of a 1% v/v solution of nitric acid and the chloroform layer is discarded. To the acid solution exactly 5 ml of dithizone standard solution is added and shaken for 30 seconds. The colour of chloroform layer is observed.

#### **Preparation of test solution**

The given sample (as specified in the monograph) is dissolved in water and transferred into a separator, 6 ml of ammonium citrate solution Sp., 2 ml of hydroxylamine hydrochloride solution Sp. And two drops of phenol red solution are added. Then the solution is made just alkaline (red in colour) by the addition of strong ammonia solution. The solution is cooled if necessary and 2 ml of potassium cyanide solution Sp. is added. Immediately the solution is extracted

with several quantities, each of 5ml of dithizone extraction solution, draining off each extract into another separating funnel, until the dithizone extraction solution retains its green colour. The combined dithizone solutions are shaken for 30 seconds with 30 ml of a 1% v/v solution of nitric acid and the chloroform layer is discarded. To the acid solution exactly 5 ml of dithizone standard solution is added and shaken for 30 seconds. The colour of chloroform layer is observed.

### **CONCLUSION-**

The colour intensity observed in the test solution was less than the standard solution. Hence the given samples had passed the limit test for iron and those are of pharmaceutical grade.

### **REFERENCES-**

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