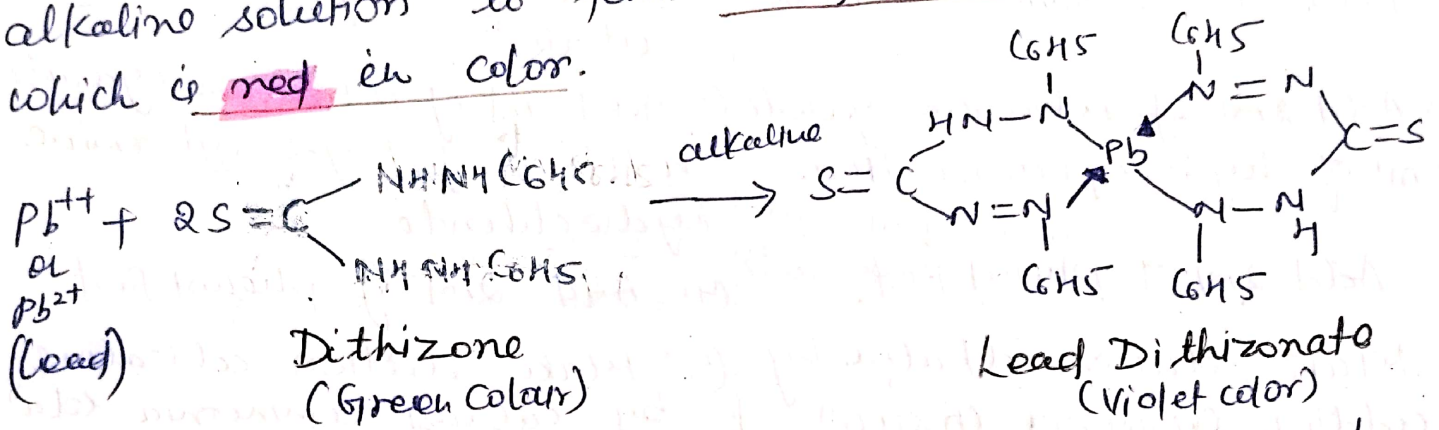


Limit Test of Lead

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.

Principle - Limit test of lead is based on the reaction of lead and diphenyl thio carbazone (dithizone) in alkaline solution to form lead dithizonate complex which is red in color.



⇒ Dithizone is green in color in chloroform and lead-dithizonate 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 dithizonate 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.

Notes - (1) $\text{C}_6\text{H}_{11}\text{NO}_7$ Ammonium citrate, KOCN potassium cyanide, hydroxylamine hydrochloride is used to make pH optimum so interference and influence of other impurities have been eliminated.

(2) Phenol red is used as indicator to develop

the colour at the end of process.

Procedure:- [Separating Funnel]

Test sample

(10)

(sample)

① A known quantity of sample solution is transferred in separating funnel.

(9 mL H₂O + 40 g malic acid + 9 mmol NH₄SO₄)

② Add 6 ml of Ammonium citrate.

③ Add 2 ml of potassium cyanide & 1 ml of hydroxylamine HCl.

(To develop colour at the end of process)

④ Add 2 ml of phenol Red.

⑤ Make solution alkaline by adding ammonia solution.

⑥ Extract with 5 ml of dithizone until it become green.

⑦ 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. (Dissolve 10 mg of dithizone in 1000 ml of chloroform)

⑨ Add 4 ml of Ammonium cyanide

⑩ Shake for 30 mins and observe the color.

chloroform
green

(7-10)

(18)

(2)

Standard Sample

(Std soln)

① 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.

③ Add 2 ml of potassium cyanide and 2 ml of hydroxylamine hydrochloride.

④ Add 2 ml of phenol Red.

⑤ Make solution alkaline by adding ammonia solution.

⑥ Extract with 5 ml of dithizone until it become green.

⑦ 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.

⑨ Add 4 ml of Ammonium cyanide

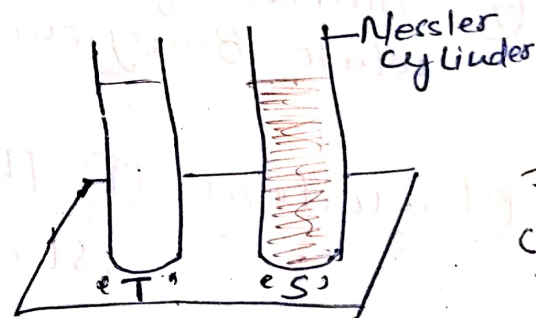
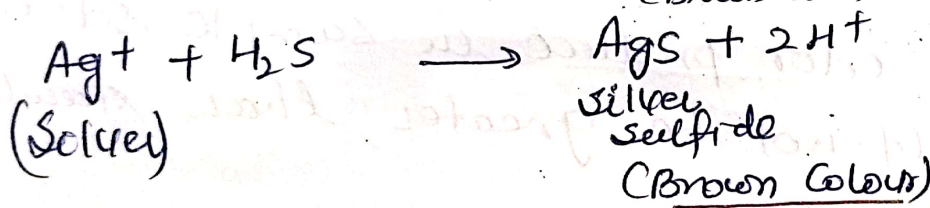
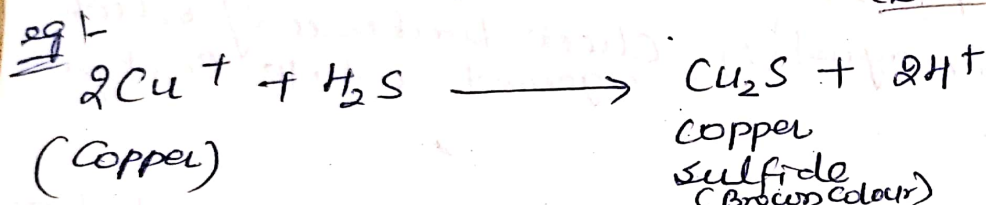
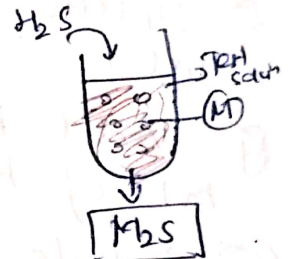
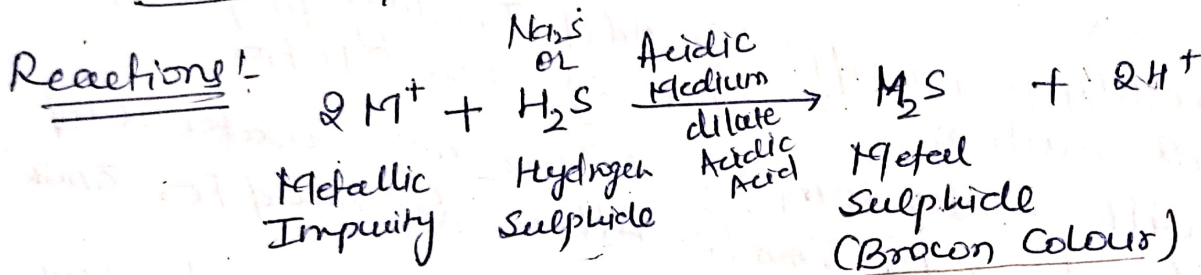
⑩ Shake for 30 mins and observe the color.

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 produced in sample solution is less than the standard solution the sample will pass the limit test of lead and

Limit Test for Heavy Metals (High degree of toxicity)

Principle:- Limit test of Heavy metals is based on the reaction of metallic impurities with Hydrogen Sulphide (H_2S) in acidic medium to form brownish colour solution. (produce)

⇒ Metals that response to this test are ^(Pb)lead, ^(Sn)tin, ^(Hg)mercury, ^(Bi)Bismuth, ^(As)arsenic, ^(Sb)antimony, ^(Cu)copper and ^(Mo)molybdenum.



- The metallic impurities in substances are expressed as parts of lead per million parts of the substance.
- The usual limit as per Indian Pharmacopoeia is 20 ppm. (If more than that sample will be rejected).

Procedure:- The Indian Pharmacopoeia has adopted 3 methods for the limit test of Heavy metals.

Method-1 → Use for the substance which gives clear colorless solution under the specific condition.

$$\left(ppm = \frac{\text{mass of solute (g)}}{\text{mass of solution (g)}} \times 10^6 \right)$$

Test

- ① Solution is prepared as per the monograph and 25 ml is transferred in Nessler's cylinder.
- ② Adjust the pH between ^(Acid) 3 to 4 by adding dilute Acetic Acid ^(7.81 type) or by adding Ammonia solution ^(21.1 type).
- ③ Dilute with water to 35 ml.
- ④ Add Freshly prepared 10 ml of H_2S solution.
- ⑤ Dilute with water 50 ml.
- ⑥ Allow to stand for 5 min.
- ⑦ Check ^(due to metallic sulphide) Darkness of solution against white Background.

Standard

- ① Take 2 ml of standard lead solution ^(20 ppm) and dilute to 25 ml with water.
- ② Adjust the pH ^{to} 3 to 4 by adding dilute Acetic acid or by adding Ammonia solution.
- ③ Dilute with water to 35 ml.
- ④ Add Freshly prepared 10 ml of H_2S solution.
- ⑤ Dilute with water 50 ml.
- ⑥ Allow to stand for 5 min.
- ⑦ Check Darkness of solution against white Background.

Observation:- ① The color produced in sample solution should not be greater than standard solution.

- ② If colour produced in sample solution is less than the standard solution, the sample will pass the limit test of Heavy metals.