LIMIT TESTS

1. LIMIT TEST FOR CHLORIDES

Apparatus Required

Nessler cylinders

Glass rod

Stand

Chemicals Required

Dilute Nitric acid (10%)

Silver nitrate (5%)

Sodium chloride

Principle

It is based upon the chemical reaction between silver nitrate and soluble chlorides in presence of dilute nitric acid to give opalescence of silver chloride. If the Opalescence produced is compared with the standard solution. If the opalescence in the sample is less than the standard, it passes the test. If it is more than the standard, it fails the test.

$$Cl^- + AgNO_3 \xrightarrow{\text{dil } HNO_3} AgCl\downarrow + No_3$$

Procedure

Take two 50 ml Nessler Cylinders. Label one as "Test" and the other as 'Standard'.

Test	Standard
Dissolve the specified quantity of the substance in distilled water and transfer Add 10 ml of dil. Add 10ml of dil. HNO_3. Dilute to 50ml with water and add 1 ml of silver nitrate solution. Stir immediately with glass rod and allow to stand for 5 minutes.	Place 1ml of 0.05845% w/v solution of NaCl and transfer to Nessler cylinder. Add 10ml of dil.HNO_3. Dilute to 50ml with water and add 1 ml of silver nitrate solution. Stir immediately with a glass rod and allow to stand for 5 minutes.

LIMIT TEST FOR SULPHATES

Apparatus Required

Nessler cylinders

Glass rod

Stand

Chemicals Required

1.Dilute Hydrochloric acid

2.Barium sulphate reagent.

Mix 15ml of 0.5m $BaCl_2$,55ml of water and 20ml of sulphate free alcohol and add 5 ml of 0.0181% w/v potassium sulphate. Dilute to 10ml with water and mix.

0.5m $BaCl_2$ Barium chloride in 1000 ml of water.

Principle

It is based upon the chemical reaction between Barium chloride and soluble sulphate in presence of dilute Hydrochloric acid . The turbidity produced is compared with the standard solution. Barium sulphate reagent contains barium chloride, sulphate – free alcohol and small quantity of potassium sulphate. The inclusion of the small quantity of potassium sulphate in the reagent increase the sensitivity of the test. Alcohol prevents super saturation and more uniform turbidity develops. If the turbidity produced in the test is more intense than the standard turbidity it fails the test . otherwise, it passes the test.

$$SO_4^{2-} + BaCl_2 \xrightarrow{\text{dil Hcl}} BaCl_2\downarrow + 2Cl^-$$

Procedure

Take two 50 ml Nessler Cylinders. Label one as “Test” and the other as ‘Standard’.

Test	**Standard**
Dissolve the sample in distilled water and transfer to a Nessler cylinder. Add 2 ml of dilute Hydrochloride acid. Dilute to 45 ml with water and add 5ml of Barium	Place 1ml of 0.1089% w/v solution of potassium sulphate and 2 ml of dil. HCI in a Nessler’s cylinder. Dilute to 45ml with water and add 5ml of Barium

sulphate reagent. Stir immediately with a glass rod and allow to stand for 5 minutes.

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LIMIT TEST FOR IRON

Apparatus Required

Nessler cylinders

Glass rod

Stand

Chemicals Required

1. Standard Iron Solution :0.1726 gm of ferric ammonium Sulphate and dissolve in 10ml of 0.1 N H_2SO_4 and sufficient water to produce 1000 ml.
2. 0.1 N H_2SO_4 :4.904 gm in 1000ml of water.
3. 20% Iron free citric acid.
4. Thioglycollic acid
5. Ammonia solution.

Principle

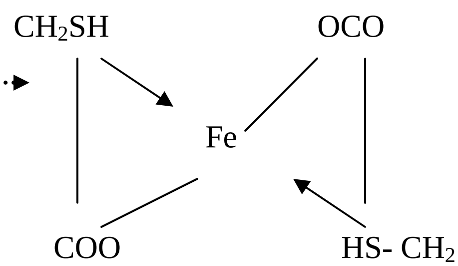
The test depends upon the reaction between ferrous iron and thioglycollic acid in the presence of ammonia When a pale pink to deep reddish purple colour is produced. Ferric iron is reduced to ferrous iron by the thioglycollic acid and the compound produced is ferrous thioglycollate . Citric acid forms a soluble complex with iron and prevents its precipitation by ammonia as ferrous hydroxide. Ferrous thioglycollate is colourless in neutral or acid solutions. The colour develops only in the presence of alkali. It is stable in the absence of air but fades when exposed to air due to oxidation to the ferric compound. Therefore the colours should be compared immediately after the time allowed for full development of colours is over.

$2Fe^{+++} + 2CH_2SH.COOH$ ....► $2Fe^{++}$ + $S.CH_2.COOH$

Ferric Iron Thioglycollic acid   Ferrous Iron |

$S.CH_2.COO$ $+2H^+$

$2Fe^{+++} + 2CH_2SH.COOH$ ....►



$+2H^+$

Ferrous thioglycollate

(Coordination compound)

Procedure

Take two 50ml Nessler cylinders. Label one as ‘Test’ and the others as ‘standard’.

Test

Dissolve a specified quantity of the substance in 20 ml of water and transfer to Nessler cylinder. Add 2 ml of a 20% w/v solution of iron –free citric acid and 0.1 ml of thioglycollic acid and mix. Make alkaline with iron-free ammonia solution. Dilute to 50ml with water and and allow to stand for 5 minutes.

Standard

Dilute 2 ml of standard Iron solution with 20ml of water in a Nessler cylinder. Add 2 ml of a 20% w/v solution of iron – free citric acid and 0.1 ml of thioglycollic acid and mix. Make alkaline with iron-free ammonia solution. Dilute to 50ml with water and allow to stand for 5 minutes.

LIMIT TEST FOR HEAVY METALS

Apparatus Required

Nessler cylinders

Glass rod

Stand

Chemicals Required

(i) Dilute $CH_3.COOH$(10% v/v)

(ii) Dilute Ammonia (10% v/v)

(iii) Hydrogen sulphide solution (Saturated of H_2S).

(iv) Standard lead solution

10ml of the lead nitrate stock solution diluted to 100 ml with water.(20 ppm of lead).

Lead nitrate stock solution

Dissolve 0.1598 gm of lead nitrate in 100 ml of water , add 1 ml of con. HNO_3 and dilute to 1000 ml with water.

Principle

It is based on the reaction between the solution Heavy metals and a saturated solution of Hydrogen sulphides. In acidic media, it produces reddish / black colour with Hydrogen sulphide which is compared with standard lead nitrate solution.

$Pb + H_2S \cdots\cdots\blacktriangleright Pbs + H_2$

Procedure

Take two 50 ml Nessler Cylinders. Label one as “Test” and the other as ‘Standard’.

Test	Standard
Place 25ml of the solution and adjust the pH 3-4 by using dilute acetic acid or dilute ammonia. Dilute to 35ml with water and mix. Add 10ml of freshly prepared H2S solution. Dilute to 50ml with water. Mix well and allow to stand	2 ml of standard lead solution is taken in a Nessler cylinders and dilute 25 ml with water. Adjust the pH 3-4 by using dilute acetic acid or dilute ammonia. Dilute to 35ml with water and mix. Add 10ml of freshly H_2S

for five minutes.

solution. Dilute to 50ml with water. Mix and allow to stand for five minutes.

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