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Lab Manual



Pharmaceutical Analysis [BP102T]



HR PATEL INSTITUTE OF PHARMACEUTICAL
EDUCATION AND RESEARCH

By,

Mr. Prashant B. Patil

Assistant Professor,

H R Patel Institute of Pharmaceutical Education and Research,

Shirpur

Experiment No: 3

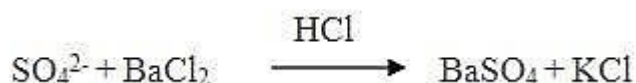
Aim: To perform limit test of given sample of Sulphate

Reference: Jooti Gupta, Mohit Sanduja and Madhuri Jover, A practical book of pharmaceutical inorganic chemistry, Nirali Prakashan, 1st Edition 2017, Page no. 1 - 18

Requirement: Nessler's cylinder, measuring cylinder, glass rod, potassium sulphate, standard solution, HCl, barium sulphate reagent and distilled water

Principle:

Limit test of sulphate is based on the reaction of soluble sulphate with barium chloride in presence of dilute hydrochloric acid to form barium sulphate which appears as solid particles (turbidity) in the solution and Barium sulphate reagent contains barium chloride, sulphate free alcohol and small amount of potassium sulphate.

**Procedure:**

Test Sample	Standard Compound
Specific weight of compound is dissolved in water or solution is prepared as directed in the pharmacopoeia and transferred in Nessler cylinder	Take 1ml of 0.1089 % W/V solution of potassium sulphate in Nessler cylinder
Add 2ml of dilute hydrochloric acid	Add 2ml of dilute hydrochloric acid
Dilute to 45 ml in Nessler cylinder	Dilute to 45 ml in Nessler cylinder
Add 5ml of barium sulphate reagent	Add 5ml of barium sulphate reagent
Keep aside for 5 min	Keep aside for 5 min
Observe the Turbidity	Observe the Turbidity

Observation:

The turbidity produce in sample solution should not be greater than standard solution. If

turbidity produces in sample solution is less than the standard solution, the sample will pass the limit test of sulphate and vice versa.

Reasons:

1. Hydrochloric acid helps to make solution acidic.
2. Potassium sulphate is used to increase the sensitivity of the test by giving ionic.
3. Concentration in the reagent and alcohol helps to prevent super saturation.

Experiment No: 4

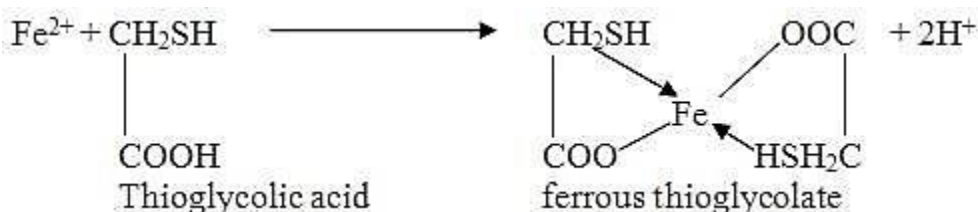
Aim: To perform limit test of given sample Iron

Reference: Jooti Gupta, Mohit Sanduja and Madhuri Jover, A practical book of pharmaceutical inorganic chemistry, Nirali Prakashan, 1st Edition 2017, Page no. 1 - 18

Requirement: Nesselar's cylinder, measuring cylinder, glass rod, citric acid, thioglycollic acid, ammonia solution, standard iron solution, and sulphuric acid

Principle:

Limit test of Iron is based on the reaction of iron in ammonical solution with thioglycollic acid in presence of citric acid to form iron thioglycolate which is pale pink to deep reddish purple in color.



Procedure:

Test sample	Standard compound
Sample is dissolved in specific amount of water and then volume is made up to 40 ml	2 ml of standard solution of iron diluted with water upto 40ml
Add 2 ml of 20 % w/v of citric acid (iron free)	Add 2 ml of 20 % w/v of citric acid (iron free)
Add 2 drops of thioglycollic acid	Add 2 drops of thioglycollic acid
Add ammonia to make the solution alkaline and adjust the volume to 50 ml	Add ammonia to make the solution alkaline and adjust the volume to 50 ml
Keep aside for 5 min	Keep aside for 5 min

Color developed is viewed vertically and compared with standard solution	Color developed is viewed vertically and compared with standard solution
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Earlier ammonium thiocyanate reagent was used for the limit test of iron. Since thioglycolic acid is more sensitive reagent, it has replaced ammonium thiocyanate in the test.

Observation:

The purple color produced in sample solution should not be greater than standard solution. If purple color produced in sample solution is less than the standard solution, the sample will pass the limit test of iron and vice versa.

Reasons:

1. Citric acid helps precipitation of iron by ammonia by forming a complex with it.
2. Thioglycolic acid helps to oxidize iron (II) to iron (III).
3. Ammonia to make solution alkaline.

Experiment No: 5

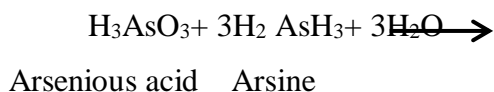
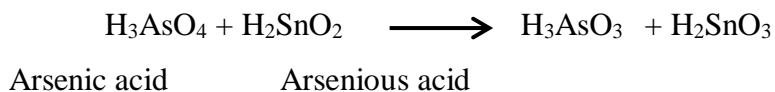
Aim: To perform limit test of given sample of Arsenic

Reference: Jooti Gupta, Mohit Sanduja and Madhuri Jover, A practical book of pharmaceutical inorganic chemistry, Nirali Prakashan, 1st Edition 2017, Page no. 1 - 18

Requirement: Gutzeit's apparatus, measuring cylinder, conical flask, glass rod, dropper, test solution, stannous chloride solution, stannous hydrochloric acid, standard arsenic solution, granulated zinc, potassium iodide, mercuric chloride paper etc.

Principle:

Limit test of Arsenic is based on the reaction of arsenic gas with hydrogen ion to form yellow stain on mercuric chloride paper in presence of reducing agents like potassium iodide. It is also called as Gutzeit test and requires special apparatus. Arsenic, present as arsenic acid in the sample is reduced to arsenious acid by reducing agents like potassium iodide, stannous acid, zinc, hydrochloric acid, etc. Arsenious acid is further reduced to arsine (gas) by hydrogen and reacts with mercuric chloride paper to give a yellow stain.



The depth of yellow stain on mercuric chloride paper will depend upon the quality of arsenic present in the sample.

Procedure:**Test Solution**

1. The test solution is prepared by dissolving specific amount in water and stannated HCl (arsenic free) and kept in a wide mouthed bottle.
2. To this solution 1 gm of KI, 5 ml of stannous chloride acid solution and 10 gm of zinc

is added (all this reagents must be arsenic free)

3. Keep the solution aside for 40 min and stain obtained on mercuric chloride paper is compared with standard solution.

Standard solution:

A known quantity of dilute arsenic solution is kept in wide mouthed bottle and rest procedure is followed as described in test solution.

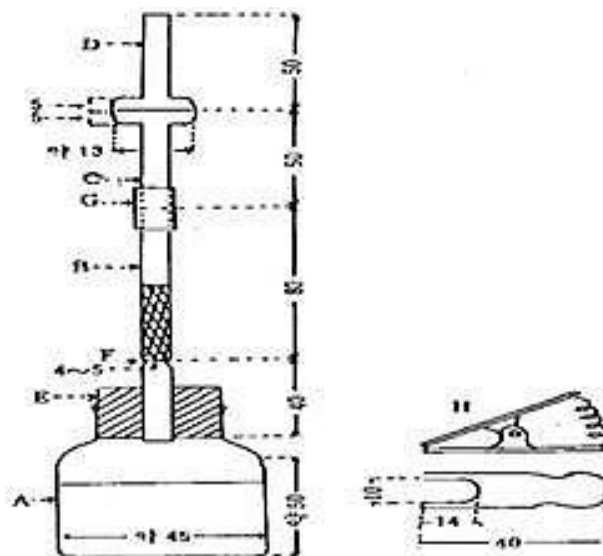


Figure: Arsenic Limit test apparatus

Where,

- A. Approximately 60 ml generator bottle with 40 ml indicating line
- B. Glass tube with 6.5 mm inner diameter
- C. and D: a ground joint glass tube with 6.5 mm inner diameter and 18 mm outer diameter at the joint. Inner joint and the outer joint form a concentric circle.
- E. Rubberstopper
- F. Narrow part of the glass tube B. Glass wool is inserted up to this part.
- G. Rubber board (Lead acetate cotton plug)
- H. Clamp

Reasons:

1. Stannous chloride is used for complete evolution of arsine.
2. Zinc, potassium iodide and stannous chloride is used as a reducing agent.
3. Hydrochloric acid is used to make the solution acidic.
4. Lead acetate pledger or papers are used to trap any hydrogen sulphide which may be evolved along with arsine.