

PHARMACEUTICAL ANALYSIS – BP102T

UNIT: III PRECIPITATION TITRATION, COMPLEXOMETRIC TITRATION,
GRAVIMETRY AND DIAZOTISATION TITRATION

CLASS:29

TOPIC: ESTIMATION OF BARIUM SULPHATE

Estimation of Barium Sulphate:

The amount of sulphate is determined quantitatively as barium sulphate, BaSO₄ by gravimetric analysis.

This determination consists of slowly adding a dilute solution of barium chloride to a hot solution of the sulphate slightly acidified with concentrated HCl.



The white precipitate is filtered off, washed with water, dried in the oven, and weighed as barium sulphate. The percentage of sulphate is calculated from the weight of barium sulphate.

PROCEDURE

(a) Preparation of porcelain Gooch crucible

1. Dry the porcelain, Gooch crucible in the oven at 110° - 120°C for 30 minutes.
2. Cool it in the desiccator.
3. Weigh the crucible, together with a piece of filter paper.
4. Repeat the above until constant weights are obtained.

b) Precipitation of BaSO₄

- Pipette 25.0 mL of the given sulphate solution into a 250 mL beaker
- Add 5 drops of concentrated HCl and about 50 mL of water.
- Caution: Concentrated HCl is very corrosive, add in the fume hood.
- Heat to boiling and, with vigorous stirring, add dropwise from a measuring cylinder 10 mL of 10% barium chloride solution.
- Cover the beaker with a watch glass and digest (heat to just below boiling) for 20 minutes.
- Test for complete precipitation by adding a few drops of BaCl_2 to the clear supernatant liquid.

(b) Washing and Filtration of BaSO_4 precipitate

1. Decant the clear supernatant by filtration (use a weighed filter paper) at the vacuum pump, using a pre-weighed crucible.
2. Wash and swirl the precipitate with about 20 mL of warm deionised water.
3. Use a 'rubber-policeman' to dislodge any particles on the beaker.
4. Allow to settle.
5. Decant the liquid through the filter paper.
6. Repeat the washing and decanting at least 2 more times.
7. Discard the filtrate.

(c) Drying and weighing of BaSO_4 precipitate

1. Dry the crucible in the oven at 150°C for about 1/2 hour.
2. Cool the crucible with BaSO_4 precipitate in a desiccator for about 10 minutes.

3. Weigh the crucible when it is cooled down.
4. The difference between this weight and the empty crucible (plus filter paper) is the weight of BaSO_4 precipitate. (Note: You may repeat Steps 1 to 4 until a constant weight of the precipitate is obtained.)

RRCP