

## Pharmaceutical Chemistry Laboratory Course

### EXPERIMENT: Gravimetry

Aim: To quantify an unknown analyte by a precipitation reaction

#### Introduction

In gravimetric analysis, the substance to be determined is converted into a definite compound which can be separated in a pure state and weighed. The procedure involves isolating and weighing an element or compound in as pure a form as possible, the element or compound is separated from a definite portion (weight or volume) of the substance being examined, and the weight of the constituent in the sample calculated from the weight of the product.

In pharmaceutical analysis, the product to be weighed is obtained by one of the following procedures:

- (i) volatilisation or ignition
- (ii) solvent extraction
- (iii) precipitation from solution

#### Analysis by precipitation

The most common and most general type of gravimetric determination involves precipitation from solution of the anion or cation to be determined either in the form of an insoluble compound of definite composition, or an insoluble compound which leaves a residue of definite composition upon ignition. The techniques involved are precipitation, filtration, washing of the precipitate and drying or ignition of the residues to constant weight. Precipitation methods rely upon the production of a substance the solubility of which is negligible under the conditions employed.

## ANALYSIS OF EPSOM SALTS FOR SULPHATE CONTENT

### Procedure:

1. Weigh out accurately approximately 0.4 g of the epsom salts provided into a 500 mL beaker.
2. Dissolve in 25 mL distilled water and add 2 drops concentrated HCl.
3. Dilute to about 200 mL with distilled water and heat solution to boiling, using a hot plate.
4. Remove from the heater and add about 14 mL warm BaCl<sub>2</sub> DROPWISE, with stirring.
5. When the precipitate settles, add a further 3 mL of BaCl<sub>2</sub>.
6. Test for complete precipitation by adding a drop of BaCl<sub>2</sub> and checking for precipitate formation as drop descends through the solution.
7. Heat solution on a water bath for about one (1) hour.
8. Filter under suction through a pre-weighed sintered glass crucible.
9. Wash precipitate (4 to 5 times) with small volumes of ethanol followed by ether.
10. Leave under suction for a few minutes and dry in an oven.
11. Weigh as BaSO<sub>4</sub>

### Exercise :

- 1) Determine the SO<sub>4</sub><sup>2-</sup> content of Epsom salt.
- 2) Determine the % mass of MgSO<sub>4</sub>·7H<sub>2</sub>O as compared to Manufacturer.