CLIL - Gravimetric methods

Gravimetric methods are quantitative methods that are based on measuring the mass of a pure compound to which the analyte is chemically related. Since weight can be measured with greater accuracy than almost any other fundamental property, gravimetric analysis is potentially one of the most accurate classes of analytical methods. The mass of this product then provides a measure of the analyte concentration. In precipitation gravimetry, the analyte is converted to a sparingly soluble precipitate. This precipitate is then filtered, washed free of impurities, converted to a product of known composition by suitable heat treatment, and weighed. For example, a precipitation method for determining calcium in natural waters involves the addition of oxalate anions ($\mathbf{C_2O_4^{2-}}$) as a precipitating agent produces:

$$Ca^{2+}_{(aq)} + C_2O_4{}^{2-}_{(aq)} \rightarrow CaC_2O_{4(s)}$$

The precipitate $\mathbf{CaC_2O_4}$ is filtered, then dried and ignited to convert it entirely to calcium oxide:

$$CaC_2O_{4(s)} \rightarrow CaO_{(s)} + CO_{(g)} + CO_{2(g)}$$

After cooling, the precipitate is weighed, and the calcium content of the sample is then computed. The steps required in gravimetric analysis, after the sample has been dissolved, can be summarized as follows: preparation of the solution, precipitation, digestion, filtration, washing, drying or igniting, weighing and finally calculation.

1. The preparation of the solution may involve several steps including adjustment of the pH of the solution in order for the precipitate to occur quantitatively and get a precipitate of desired properties, removing interferences... etc.
2. Precipitation requires addition of a precipitating agent solution to the sample solution. Upon addition of the first drops of the precipitating agent, supersaturation occurs, then nucleation starts to occur where every few molecules of precipitate aggregate together forming a nucleus. At this point, addition of extra precipitating agent will either form new nuclei (precipitate with small particles) or will build up on existing nuclei to give a precipitate with large particles.
3. Digestion of the Precipitate: The precipitate is left hot (below boiling) for 30 min to 1 hour in order for the particles to be digested. Digestion involves dissolution of small particles and reprecipitation on larger ones resulting in particle growth and better precipitate characteristics.
4.The washing operation is useful to remove all the substances that can be absorb by the precipitate and falsify the next weighing. To avoid filtering problems with surface adsorption may be reduced by careful washing of the precipitate. Filtration should be done in appropriate sized Goosh or ignition ashless filter paper.
5. Drying and Ignition: The purpose of drying (heating at about 120-150°C in an oven) is to remove the remaining moisture while the purpose of ignition in a muffle furnace at temperatures ranging from 600-1200°C is to get a material with exactly known chemical structure so that the amount of analyte can be accurately determined. The precipitate is converted to a more chemically stable form. For instance, calcium ion might be precipitated using oxalate ion, to produce calcium oxalate ($\mathbf{CaC_2O_4}$) which is hydrophil, therefore it is better to be heated to convert it into $\mathbf{CaCO_3}$ or **CaO**. The $\mathbf{CaCO_3}$ formula is preferred to reduce weighing errors.
6. Weighing the precipitate: The precipitate can not be weighed with the necessary accuracy in place on the filter paper; nor can the precipitate be completely removed from the filter paper in order to weigh it. The precipitate can be carefully heated in a crucible until the filter paper has burned away; this leaves only the precipitate. The precipitate is weighed at constant weight, repeating the weighing several times until two successive weighings do not diverge by 0.0003 g.

Adapted from: faculty.ksu.edu.sa

TEACHING AIMS:

- Understanding the meaning of gravimentric analysis;
- Understanding the meaning of precipitation;
- Understanding the meaning of digestion;
- Understanding the differences between washing and filtration;
- Understanding the differences between Drying and Ignition;
- Understanding the different methods of gravimentric analysis;

EXERCISE

1 Read the text and complete the scheme below

	In a muffle furnace at temperatures ranging from 600-1200°C is to get a material with exactly known chemical structure so that the amount of analyte can be accurately determined
	At constant weight, repeating the weighing several times until two successive weighings do not diverge by 0.0003 g
	It should be done in appropriate sized Goosh or ignition ashless filter paper
	It is useful to remove all the substances that can be absorb by the precipitate and falsify the next weighing
	We must heating precipitate at about 120-150°C in an oven to remove the remaining moisture
	It requires addition of a precipitating agent solution to the sample solution
	It involves dissolution of small particles and reprecipitation on larger ones resulting in particle growth and better precipitate characteristics