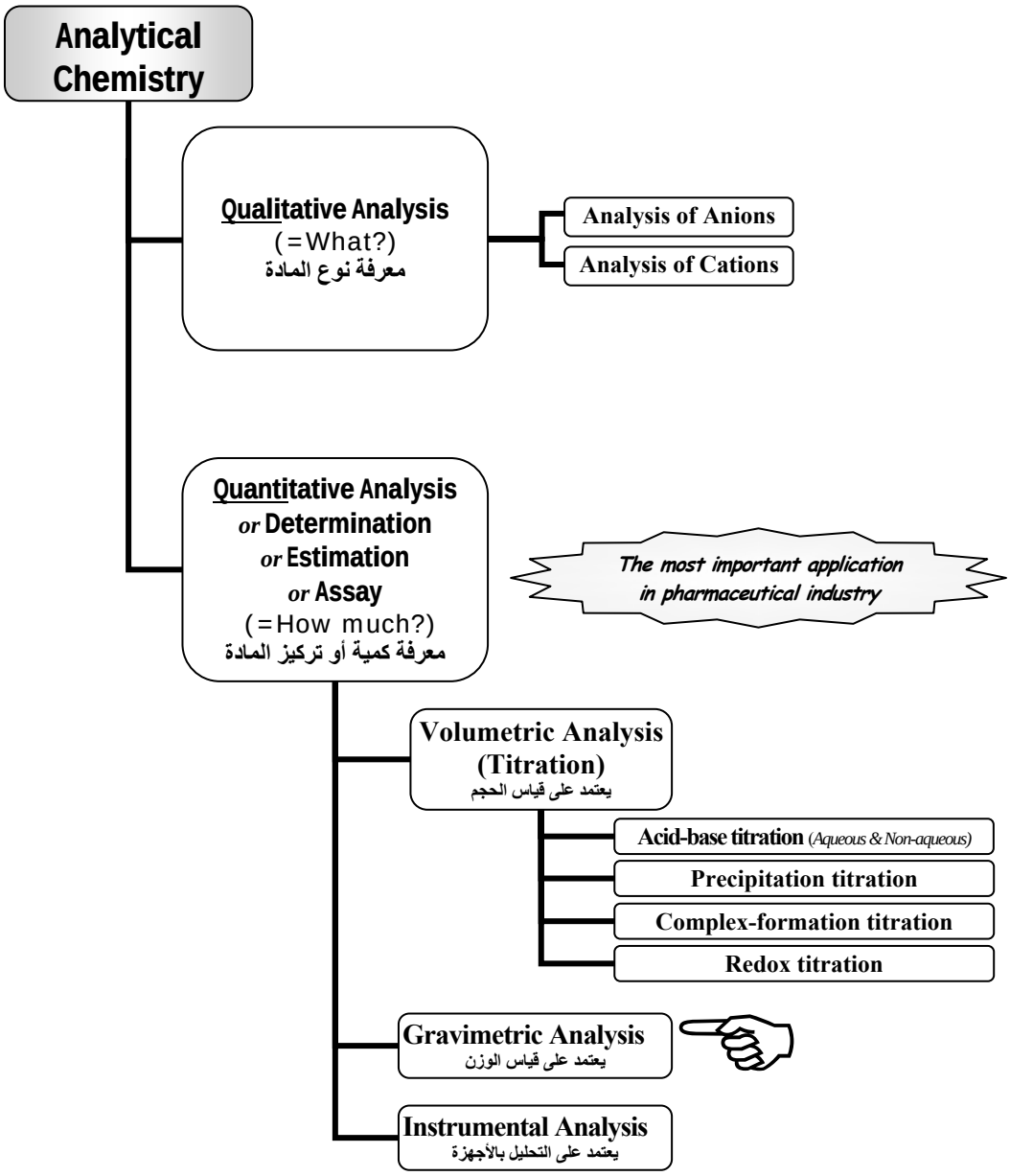
Gravimetric Analysis

Definition of Gravimetric Analysis :

It is a type of quantitative analysis that involves weighing of the constituent under determination. IN OTHER WORDS, It is the process of isolating and weighing an element or compound in a pure form.

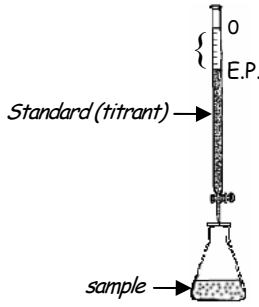
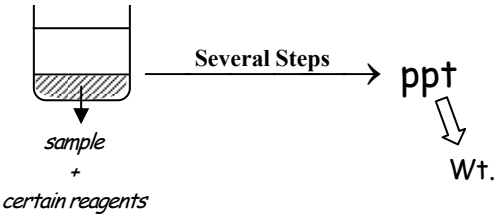
⇒ ***Remember:***

Classification of Analytical Chemistry



Comparison between

Volumetric Analysis (Titration) & Gravimetric Analysis

	Volumetric Analysis (Titration)	Gravimetric Analysis
Diagram:		
Sample:	Liquid or Solid	Liquid or Solid
Result obtained:	E.P. (Vol. consumed from titrant) Ex: 4 ml	Weight of the ppt Ex: 4 g
Required:	Concentration of the sample	Concentration of the sample
Steps of Calculations:	1st: Calc. of **equivalence** factor (F). 2nd: Calc. of Concentration.	1st: Calc. of **gravimetric** factor (F). 2nd: Calc. of Concentration.

Examples of gravimetric determinations:

1) Gravimetric determination of NaCl:

$$NaCl + AgNO_3 \longrightarrow \underset{\text{ppt}}{AgCl\downarrow} + NaNO_3$$

filtered, dried and weighed and then from the weight of the ppt, the concentration of NaCl sample can be calculated.

2) Gravimetric determination of Potash alum [$KAl(SO_4)_2.12H_2O$]:

$$2\ KAl(SO_4)_2.12H_2O \longrightarrow \underset{\text{gelatinous ppt}}{2\ Al(OH)_3\downarrow} \xrightarrow{\text{ignition}} \underset{\text{ppt}}{Al_2O_3\downarrow}$$

Not suitable for weighing.

More suitable for weighing & from its weight, the concentration of Potash alum sample can be calculated.

Calculations for Gravimetric Analysis { 2 STEPS }:

1st step: Calculation of the Gravimetric Factor OR Conversion Factor (F):

Gravimetric Factor is the weight of the sample equivalent 1 g of the ppt.

$$F = \frac{m \times (\text{M.W. of } \textbf{substance sought})}{n \times (\text{M.W. of } \textbf{substance weighed})}$$

(substance sought: sample; substance weighed: final ppt)

No Unit

m & n are integers that make the molecular weights in the numerator (البسط) & denominator (المقام) chemically equivalent.

N.B. Gravimetric factor (F) is completely different from Equivalence factor (F).

2nd step: Calculation of the Concentration:

If the sample is liquid (solution):

$$\text{Concn.} = \frac{\text{Wt. of the ppt} \times F \times \mathbf{1000}}{\text{Volume taken from the sample}} = \ldots\ldots\ldots \textbf{ g/L}$$

$$= \frac{\text{Wt. of the ppt} \times F \times \mathbf{100}}{\text{Volume taken from the sample}} = \ldots\ldots\ldots \textbf{ g\% (i.e. g/100 ml)}$$

If the sample is solid (powder):

$$\text{Concn. or Purity} = \frac{\text{Wt. of the ppt} \times F \times \mathbf{100}}{\text{Wt. taken from the sample}} = \ldots\ldots\ldots \textbf{ g\% (i.e. g/100 g)}$$

Example: *In the determination of $MgSO_4.7H_2O$:*

The reaction can be summarized as follows:

$$2\ MgSO_4.7H_2O \longrightarrow 2\ MgNH_4PO_4.6H_2O \downarrow \xrightarrow{\text{ignition}} Mg_2P_2O_7 \downarrow$$

(final ppt)

sample | *Mag. amm. phosphate* | *Mag.pyrophosphate*

$$2\ (MgSO_4.7H_2O) \equiv 1\ (Mg_2P_2O_7)$$

(m: sample; n: final ppt)

$$F = \frac{2 \times (\text{M.W. of } MgSO_4.7H_2O)}{1 \times (\text{M.W. of } Mg_2P_2O_7)}$$

Then the concentration can be calculated from one of the above mentioned equations:

$$\text{Concn.} = \frac{\text{Wt. of the ppt} \times F \times \mathbf{1000}}{\text{Volume taken from the sample}} = \ldots\ldots\ \textbf{g/L}$$

Derivation of Gravimetric Factor (F):

Example: *In the determination of $MgSO_4.7H_2O$:*

$$2\ (MgSO_4.7H_2O) \longrightarrow 1\ (Mg_2P_2O_7)$$

sample *final ppt*

The Gravimetric Factor (F) means:

the weight of the sample ($MgSO_4.7H_2O$) equivalent **1 g** of the ppt ($Mg_2P_2O_7$).

$$2 \text{ moles of } MgSO_4.7H_2O \longrightarrow 1 \text{ mole of } Mg_2P_2O_7$$

$$\text{i.e. } 2 \times (\text{M.W.})_g \text{ of } MgSO_4.7H_2O \longrightarrow 1 \times (\text{M.W.})_g \text{ of } Mg_2P_2O_7$$

$$\therefore (?)\ g \text{ of } MgSO_4.7H_2O \longrightarrow \mathbf{1\ g} \text{ of } Mg_2P_2O_7$$

$$\therefore F = 1 \times \frac{2 \times (\text{M.W. of } MgSO_4.7H_2O)}{1 \times (\text{M.W. of } Mg_2P_2O_7)}$$

$$\therefore F = \frac{2 \times (\text{M.W. of } MgSO_4.7H_2O) \longrightarrow \text{sample}}{1 \times (\text{M.W. of } Mg_2P_2O_7) \longrightarrow \text{final ppt}}$$

Basic Steps for Any Gravimetric Analysis

1. Sampling & Preparation for precipitation.
2. Precipitation.
3. Digestion (i.e. Leaving the ppt in contact with the soln. for a considerable time).
4. Filtration & Washing.
5. Drying OR Ignition.
6. Weighing & Calculations.

Required Glassware for Gravimetric Analysis (الأدوات المطلوبة):

- **Beaker 250 ml.**
- **10-ml bulb pipette.**
- **10-ml graduated pipette.**
- **10-ml measuring cylinder.**
- **Filter papers (Whatmann no.1).**
- **Funnel.**
- **Flask.**
- **Watch glass.**
- **Test tube.**

Exp.(8): Gravimetric Determination of $Na_2SO_4.10\ H_2O$ (USP 2007)

Principle :

It depends on the precipitation of SO_4^{--} as $BaSO_4$ by adding $BaCl_2$ to a hot solution of SO_4^{--} slightly acidified with HCl.

$$\underset{\textit{sample}}{SO_4^{--}} + \underset{\textit{BaCl}_2\textit{ (ppting agent)}}{Ba^{2+}} \longrightarrow \underset{\textit{white ppt}}{BaSO_4 \downarrow}$$

Procedure :

Sampling & Preparation for Precipitation

1- Transfer 10 ml of the sample of sodium sulphate into a 250 ml beaker, Dilute to about 150 ml with water.

2- Add about 10 ml dilute HCl, and heat to boiling.

Precipitation

3- Remove the flame, and add slowly with constant stirring (*by glass rod*), a hot 2 % $BaCl_2$ solution in slight excess (*about 10 ml heated in a test tube*).

4- **Test for complete precipitation** by leaving the precipitate to settle, and then adding to the clear supernatant liquid, drops of the precipitating agent (2% $BaCl_2$), noting if any turbidity appears.

If turbidity appears → add more drops of $BaCl_2$ soln. and repeat the test.

If no turbidity → complete the next steps.

Digestion

5- Cover the beaker with a watch glass, and place it in a water bath almost at the boiling point for about one hour.

Filtration & Washing

6- Decant the supernatant liquid in a suitable ashless filter paper and wash the precipitate in the beaker by decantation two times with hot water.

Decantation means: Pouring off the clear upper portion (supernatant) of a fluid leaving a sediment or a ppt at the bottom WHILE Washing by Decantation means: mixing some of the washing solution with the ppt, then allow the ppt to settle down again and then the clear supernatant is decanted.

⬇

Repeat washing & filtration until all the ppt is filtered

⬇

Drying ➔ ***Weighing & Calculations***

Calculations :

$$1\ (Na_2SO_4.10H_2O) \equiv 1\ (BaSO_4)$$

m = sample, n = ppt

$$F = \frac{1 \times (\text{M.W. of } Na_2SO_4.10H_2O)}{1 \times (BaSO_4)}$$

$$\text{Concn.} = \frac{\text{Wt. of the ppt} \times F \times \mathbf{1000}}{\text{Volume taken from the sample}} = \ldots\ldots\ldots\ \mathbf{g/L}$$

Best wishes