Gravimetric method of analysis :

Gravimetric methods: The quantitative methods that are based on determining the mass of a pure compound to which the analyte is chemically related.

The principle of Gravimetric Analysis:

The principle behind the gravimetric analysis is that the mass of an ion in a pure compound and can be determined. Later, used to find the mass percent of the same ion in a known quantity of an impure compound.

Types of Gravimetric Analysis

• Precipitation gravimetry: The analyte is separated from a solution of the sample as a precipitate and is converted to a compound of known composition that can be weighed.

• Volatilization gravimetry: The analyte is separated from other constituents of a sample by converting it to a gas of known chemical composition that can be weighed.

• Electrogravimetry: The analyte is separated by deposition on an electrode by an electrical current.

Properties of gravimetric analysis:

1- **Tradition method**
2- **Cheap, easily available apparatus, simple to carry out**
3- **Slow, especially when accurate results are required**
4- **Wide range of concentration (ng-kg)**
5- **No calibration required except for the balance**
6- **Accurate**

Precipitation gravimetry

The analyte is converted to a sparingly soluble precipitate that isλ then filtered, washed free of impurities and converted to a product of known composition by suitable heat treatment and weighed.

Ex:

Calcium in water

$2NH_3 + H_2C_2O_4 \rightarrow 2NH_4^+ + C_2O_4^{-2}$

$Ca^{+2}(aq) + C_2O_4^{-2}(aq) \rightarrow CaC_2O_4(s)$

filtered, dried, ignited

$CaC_2O_4 + \Delta \rightarrow CaO(s) + CO(g) + CO_2$

The step required in gravimetric analysis:

1- Precipitation
2- Digestion
3- Filtration
4- Washing
5- Drying and igniting
6- Weighing
7- Calculation

Gravimetric factor(GF):

the weight of one substance is converted into the corresponding weight of another substance through substance through multiplication by asset of constant terms .

GF=a/b *gfw substance sought / gfw substance weighed

Species I sought	Species weighed 2	GF
In	In2O3	2/1*gfw In/gfw In2O3
HgO	Hg5(IO6)2	5/1*gfw HgO /gfw Hg5(IO6)2
I	Hg5(IO6)2	2/1*gfw I/gfw Hg5(IO6)2
K3PO4	K2PtCl6	2/3*gfw K3PO4/gfw K2PtCl6
K3PO4	Mg2P2O7	2/1*gfw K3PO4/gfw Mg2P2O7

Example: Calculate the amount of sulphate as barium sulphate from sodium sulphate. Solution of sodium sulphate (Na_2SO_4) is treated with solution of barium chloride ($BaCl_2$) to get precipitates of barium sulphate ($BaSO_4$). The precipitates are then washed, dried and ignited to get free from impurities and then weighed.

$Na_2SO_4 + BaCl_2 \rightarrow BaSO_4 + 2\ NaCl$

Mol. Weight of $BaSO_4$ = 233.42 gm
Mol. Weight of SO_4^- = 96.06 gm

Suppose obtained weight of $BaSO_4$ precipitates = X · gm
233.42 gm of $BaSO_4$ = 96.06 gm of $SO4^-$ ions
X · gm of $BaSO_4$ = ?

$BaSO_4 = 96.06 \cdot X / 233.32 = 0.411X$ gm of $SO4^-$ ions

Suppose 25 ml solution is consumed, then

25 ml solution contains = 0.411X gm of $SO4^-$ ions
1000ml solution conatins?
1000ml solution conatins = $0.411X \cdot 1000 / 25 = 16.44X$ gm of $SO4^-$ ions

Example: Calculate the amount of zinc oxide from zinc sulphate.
A solution of zinc sulphate is boiled to convert it into zinc carbonate by adding solution of sodium carbonate. Sodium carbonate is added to precipitate zinc completely as zinc carbonate. Precipitates of zinc carbonate is boiled for few minutes to convert it into zinc oxide and collected in a tarred Gooch crucible. Precipitates are washed with hot water until it gets free from alkali and then dried, ignited and weighed to a constant weight.

$ZnSO_4 + Na_2CO_3 \rightarrow ZnCO_3 + Na_2SO_4$

$ZnCO_3 \rightarrow ZnO + CO_2$

$ZnSO_4 = ZnCO_3 = ZnO$
$ZnSO_4 = ZnO$

Mol. Weight of $ZnSO_4 = 168$ gm
Mol. Weight of ZnO = 81.38 gm
81.38 gm of ZnO = 168 gm of $ZnSO_4$
1 gm of ZnO =?
$= 168 \cdot 1 / 81.38$
= 1.984 gm

Example: Calculate the amount of Boric acid from Borax.

Borax is an alkaline substance, and reacts with conc. HCl to form Boric acid. Boric acid is freely soluble in boiling water and precipitated out in cold water. To get high grade of Boric acid, Borax is treated with conc. HCl as it is volatile in nature and won't left any residual traces on crystal surface of Boric acid.

Weight and dissolve 5 gm of Borax in 15 ml of distilled water. Add 7 ml of conc. HCl, mix thoroughly with glass rod and mark the original volume with glass rod. Evaporate the solution till the volume reduces to half of the original volume. Allow to cool at room temperature. Keep it aside for few min and add ice water. Filter the residue under suction and dry it in air. Weight the compound preparation.

$Na_2B_4O_7.10H_20 \rightarrow 4H_3BO_3 + 5H_20 + 2NaCl$

Mol wt of Borax = 381.37gm
Mol wt of Boric acid = 61.83gm
Practical yield: X gm

381.37 gm of Borax = 4 × 61.83 gm of Boric acid
X gm of Borax = ?
= X · 4 · 61.83 / 381.37
= 0.674X gm of Boric acid

Example: The calcium in a 200 mL sample of a natural water was determined by precipitating the cation as CaC_2O_4. The precipitate was filtered, washed, and ignited in a crucible with empty mass of 26.6002 g. The mass of the crucible and CaO(56.099 g/mol) was 26.7134 g. Calculate the concentration of Ca(40.078 g/mol) in the water in units of grams per 100 mL.

The mass of CaO is

26.7134 g –26.6002 g = 0.1132 g

The number of moles Ca in the sample is equal to the number of moles CaO

Amount Ca = 0.1132 g CaO x $\frac{1\text{mol CaO}}{56.077\text{ g}}$ x $\frac{1\text{ mol Ca}}{\text{mol CaO}}$ = 2.0168e-3 mol Ca

Mass Ca/100mL = $\frac{2.0186\text{e-}3 \times 40.078\text{ g Ca/mol Ca}}{200\text{ ml sample}}$ x 100 mL = **0.04045 g/ 100ml**