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Module-1

Theory of Volumetric and Gravimetric Analysis

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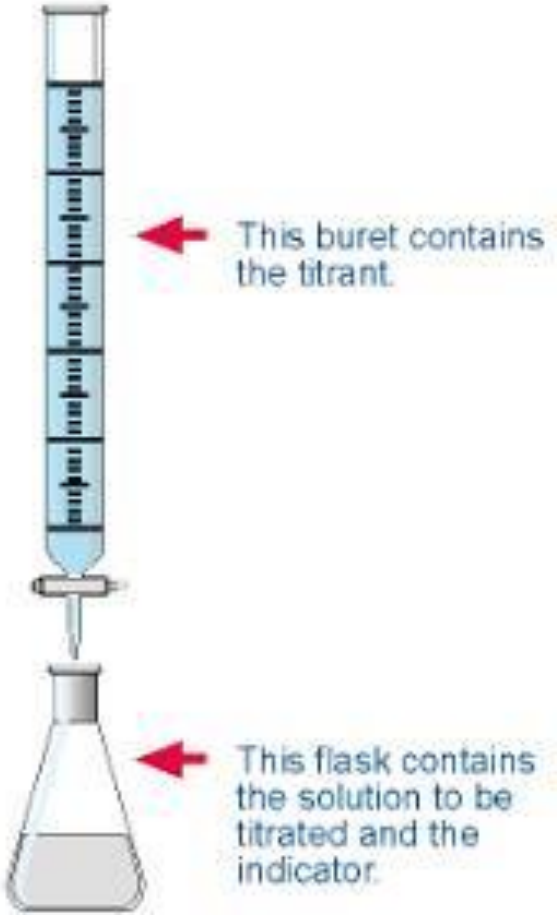
Objective



Gravimetric Analysis

WHAT ARE SOME OF THE KEY THINGS WE WILL LEARN FROM THIS TOPIC?

- What is gravimetric analysis?
- Steps of a gravimetric analysis: precipitation, digestion, filtration, washing, drying, weighing, calculation
- The solubility product, the common ion effect
- Gravimetric calculations (key equations)

What is gravimetric analysis?

1914
for his work.

- Gravimetry is among the most accurate analytical techniques (but it
  is tedious!).

- T. W. Richards used it to determine
  atomic weights.

- He received the Nobel Prize in

Theodore W. Richards

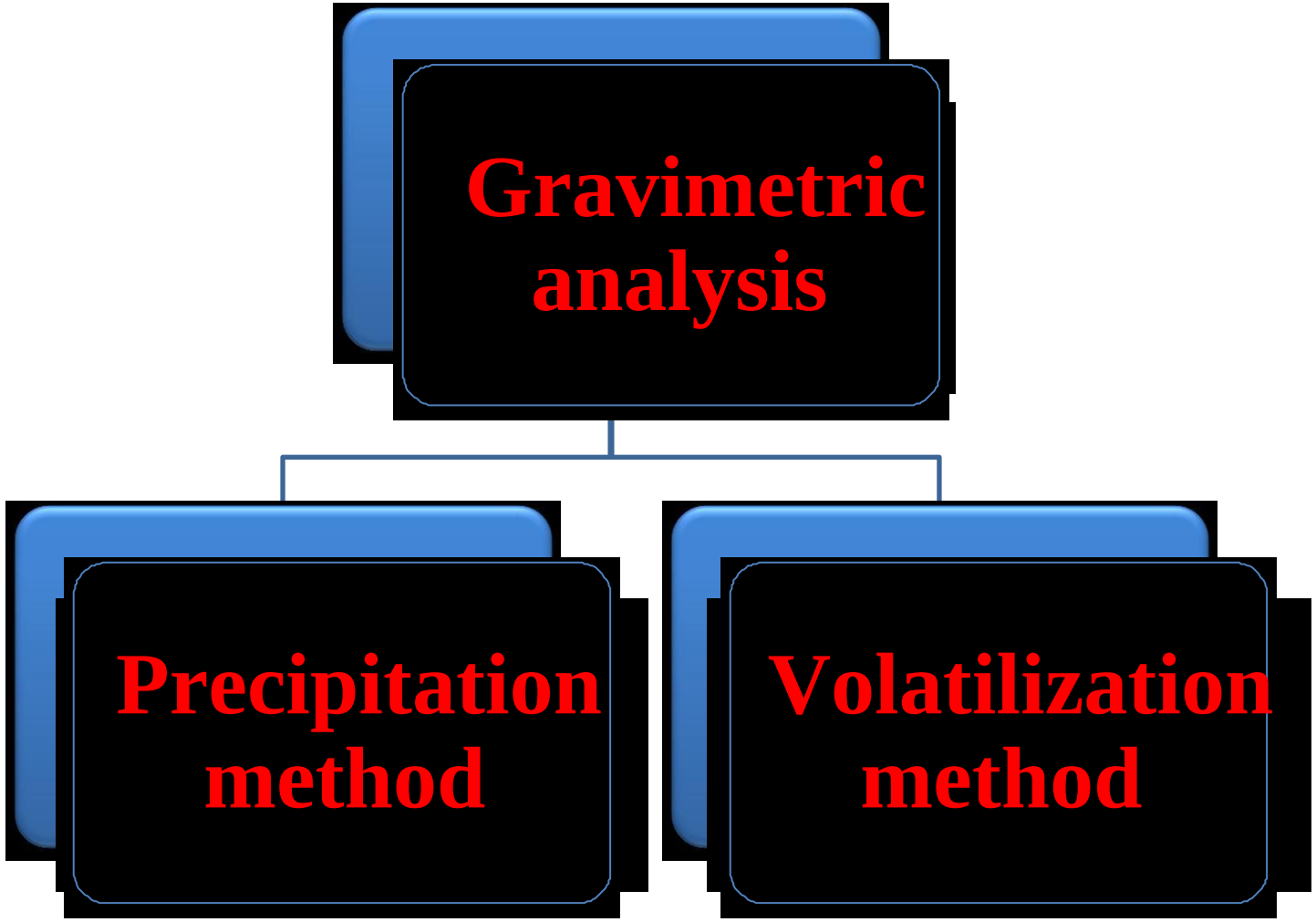
- Gravimetric analysis is one of the most accurate and precise methods of macro-quantitative analysis.

- In this process the analyte is selectively converted to an insoluble form.

- The separated precipitate is dried or ignited, possibly to another form, and is accurately weighed.

- From the weight of the precipitate and a knowledge of its chemical composition, we can calculate the weight of analyte in the desired form.

Types of Gravimetric Analysis



Precipitation method:

- Analyte must first be converted to a solid (precipitate) by precipitation with an appropriate reagent.

- The precipitates from solution is filtered, washed, purified (if necessary) and weighed.

Volatilization method

- In this method the analyte or its decomposition products are volatilised (dried) and then collected and weighed.

- or alternatively, the mass of the volatilise product is determined indirectly by the loss of mass of the sample.

Example for Precipitation

- Calcium can be determined gravimetrically by precipitation of calcium oxalate and ignition of the oxalate ion to calcium oxide.

↓

- $Ca^{2+} + C_2O_4^{2-} \rightarrow CaC_2O_4$

  $CaC_2O_4 \rightarrow CaO + CO_2 + CO$

- The precipitate thus obtained are weighed and

the mass of calcium oxide is determined

Example for Volatilisation

- The analyte or its decomposition products are volatilised at a suitable temperature.

- The volatile product is then collected and weighed, i.e. the mass of the product is indirectly determined from the loss in mass of the sample.

- Example, Water can be separated from most inorganic

compounds by ignition.

- The evolved water can then be absorbed on any one of several solid desiccants.

- The weight of water evolved may be calculated from the gain in weight of the absorbent.

- Not all insoluble precipitates are well suited for gravimetric analysis.

Advantages of Gravimetric Analysis

- Accurate and precise: Gravimetric analysis is potentially more accurate and more precise than volumetric analysis.

- Gravimetric analysis avoids problems with temperature fluctuations, calibration errors, and other problems associated with volumetric analysis.

- It is an ABSOLUTE method. i.e. it involves direct measurements without ant form of calibration.

- Relatively inexpensive

Disadvantages

But there are potential problems with gravimetric analysis that must be avoided to get good results:

- Proper lab technique is critical
- Careful and time consuming.
- Very clean glassware.
- Very accurate weighing.
- Co-precipitation.

Steps in a gravimetric analysis

1. Preparation of the solution
2. Precipitation
3. Digestion
4. Filtration
5. Washing
6. Drying or ignition
7. Weighing
8. Calculation

1.Preparation of analyte solution

- Gravimetric analysis usually involves precipitation of analyte from solution. To prepare the analyte solution, which may need:

1. – Preliminary separation to separate potential interferences before precipitating analyte

2. Adjustment of solution condition (pH/temp/volume/concentration of test substance) to
maintain low solubility of precipitate & max precipitate
formation.

- Example: Calcium oxalate insoluble in basic Medium

3. – Most of the substances are readily soluble in water and can be used as such.

4. - Some required special treatment as treatment with HCl, HNO_3, Aqua regia or fusing with basic flux

2.Precipitation process :

- The precipitating reagent is added at a concentration that favors the formation of a "good“ precipitate.

- This may require low concentration, extensive heating (often described as "digestion"), or

careful control of the pH.

- A large excess precipitating reagent should be avoided because this increases chances of adsorption on ppt.

- Test for completeness of precipitation

- No new ppt should be formed after addition of drop of ppting agent.

3.Digestion of the Precipitate

- Let precipitate stand in contact with mother liquor (the solution from which it was precipitated)

- This process is called digestion, or Ostwald ripening. The small particles tend to dissolve and precipitate on the surfaces of the larger crystals.

- Digestion make larger crystals, reduce

surface

contamination, reduce crystals imperfection.

4. Filtration