

Analytical Chemistry

```
graph TD; A[Analytical Chemistry] --> B[Qualitative Analysis]; A --> C[Quantitative Analysis]; B --> B1[Identification of unknown]; C --> C1[concentration]; C1 --> D[Solid]; C1 --> E[Solution]; D --> D1[g%]; E --> E1[g/L];
```

Qualitative Analysis

Identification of unknown

Quantitative Analysis

Determination of Sample
concentration

Solid

g%

Solution

g/L

Gravimetric Analysis



Quantitative
analysis

(Gravi = weight , metry = to measure)

Definition:

Is the process of isolating by precipitation , and weighing of a final product with known, pure, stable and definite chemical structure.

Steps of gravimetric analysis:

- 1- Precipitation
- 2- Ageing
- 3- Filtration
- 4- Washing
- 5- Drying or igniting
- 6- Weighing
- 7- Calculation

1- Precipitation:

A-Properties precipitating reagents:

Ideally, a Gravimetric precipitating agent should react specifically or at least selectively with the analyte.

B-Properties of good precipitates:

- 1- Quantitatively precipitated.
- 2- Pure (not Contaminated).
- 3- Suitable physical form (Easily filtered and washed free of contaminants).
4. Of known chemical composition after it is dried or ignited.

C- Particle size of the ppt:

1-Colloidal ppt.:

- Tiny particles pass through filter paper.
- Show no tendency to settle from solution.



2 - Amorphous ppt.:

- Large surface area ????
- Aggregates of nuclei easily transformed into colloidal state.

3-Crystalline ppt.:

- Particles with large size .
- Tend to settle spontaneously.
- Easily Washed and retained on the filter paper.



***Obtained by precipitation from hot dilute solution with stirring and adding the precipitating agent slowly dpwise.**

2- Ageing (crystal growth):

- Happens when a freshly formed precipitate is left in the solution from which it precipitates.
- It results in **cleaner** and **bigger** particles.

3- Filtration:

Done by decantation ??????

4- Washing:

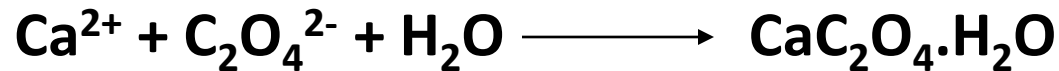
- To remove surface adsorbed impurities .
- Wash solution ???

5- Drying (< 250 °C) Or Ignition (> 250°C -1200°C).

Gravimetric Determination of Calcium as Calcium Oxalate Monohydrate

Principle:

CaCl_2 is precipitated as $\text{CaC}_2\text{O}_4 \cdot \text{H}_2\text{O}$ in alkaline medium, the ppt. is washed with dilute ammonium oxalate and then weighed.



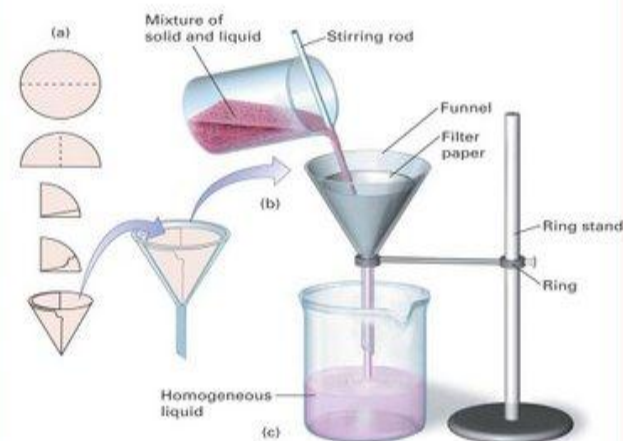
Procedure:

1- In a 250 mL beaker , Take 10 mL sample + 150 mL dist. water + 2 mL dil. HCl+ 2 dps M.R. (**Red color**)+ 5 mL saturated ammonium oxalate, heat.

2- Add NH_3 dpwise till alkaline (**Yellow colour**) while stirring, boil

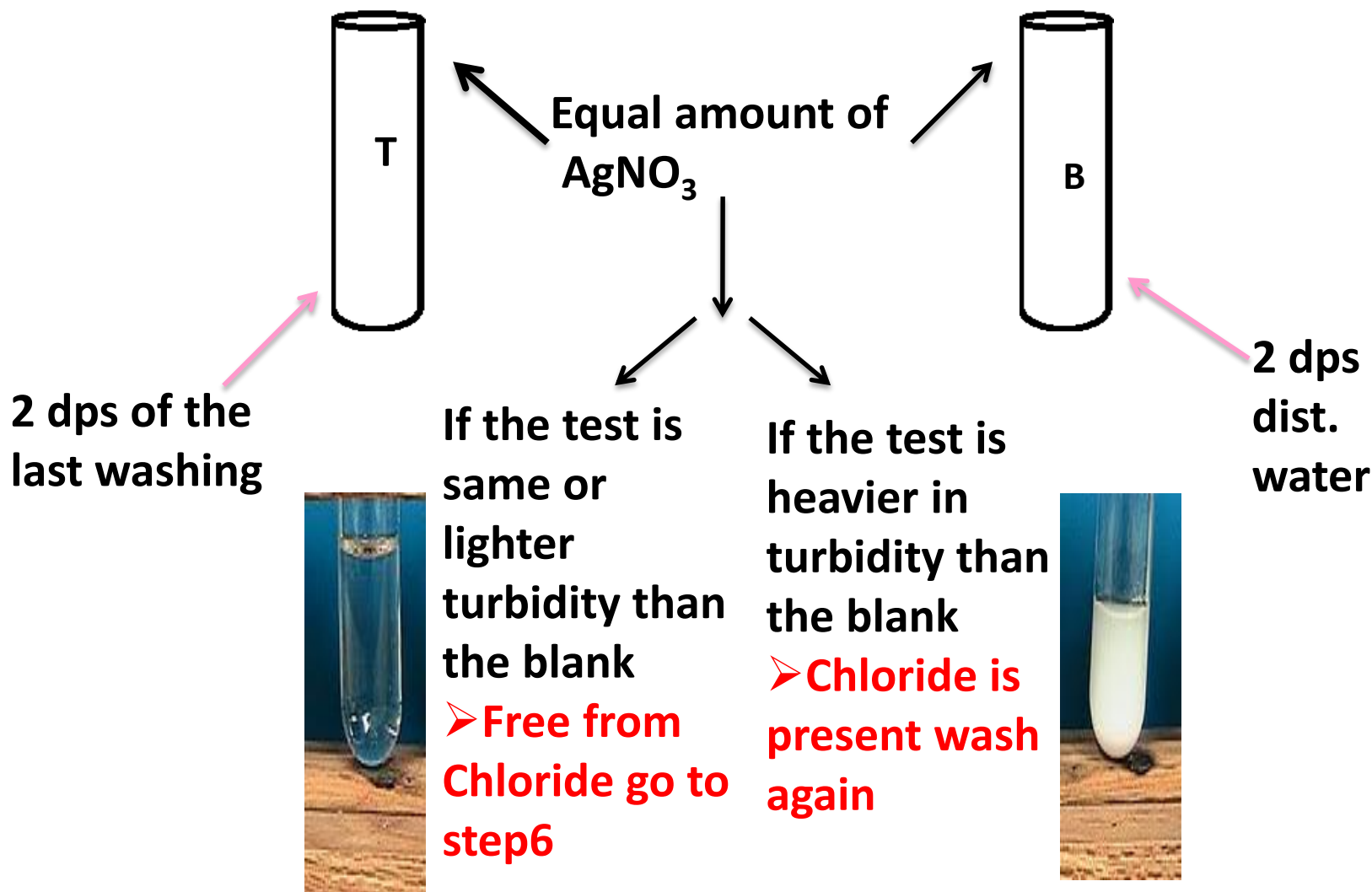
3- Ageing for 10 min.

4- Filtration by decantation on 2 matched filter paper.



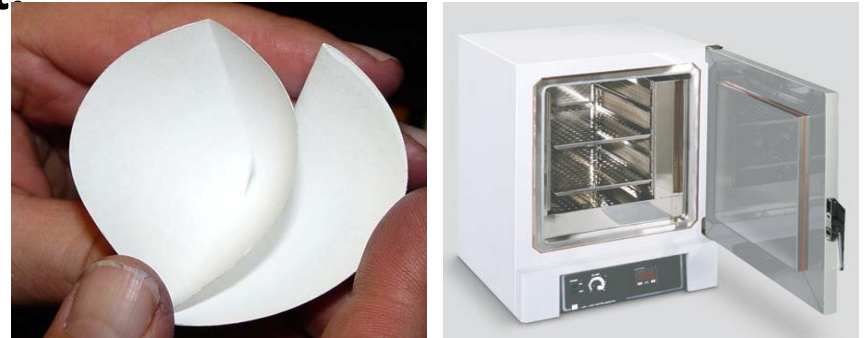
5- Wash the ppt. till free from Cl^- (4×10 mL 1% amm. oxalate), every time wait till complete drainage of the last wash.

➤ Test for Chloride:



6- Transfer the **ppt on the filter paper**, dry in the oven at 110 °C for 1 hr, weigh and re-dry till constant weight.

Calculation:



111 g
?

146 g
wt of ppt (known)

$$\text{Conc. of CaCl}_2 = \text{Wt of ppt} * \frac{111}{146} * \frac{1000}{10} = \dots\dots\dots \text{g/L}$$

