

SNS COLLEGE OF PHARMACY AND HEALTH SCIENCES

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Pharmacy Council of India, New Delhi.
Coimbatore -641035*

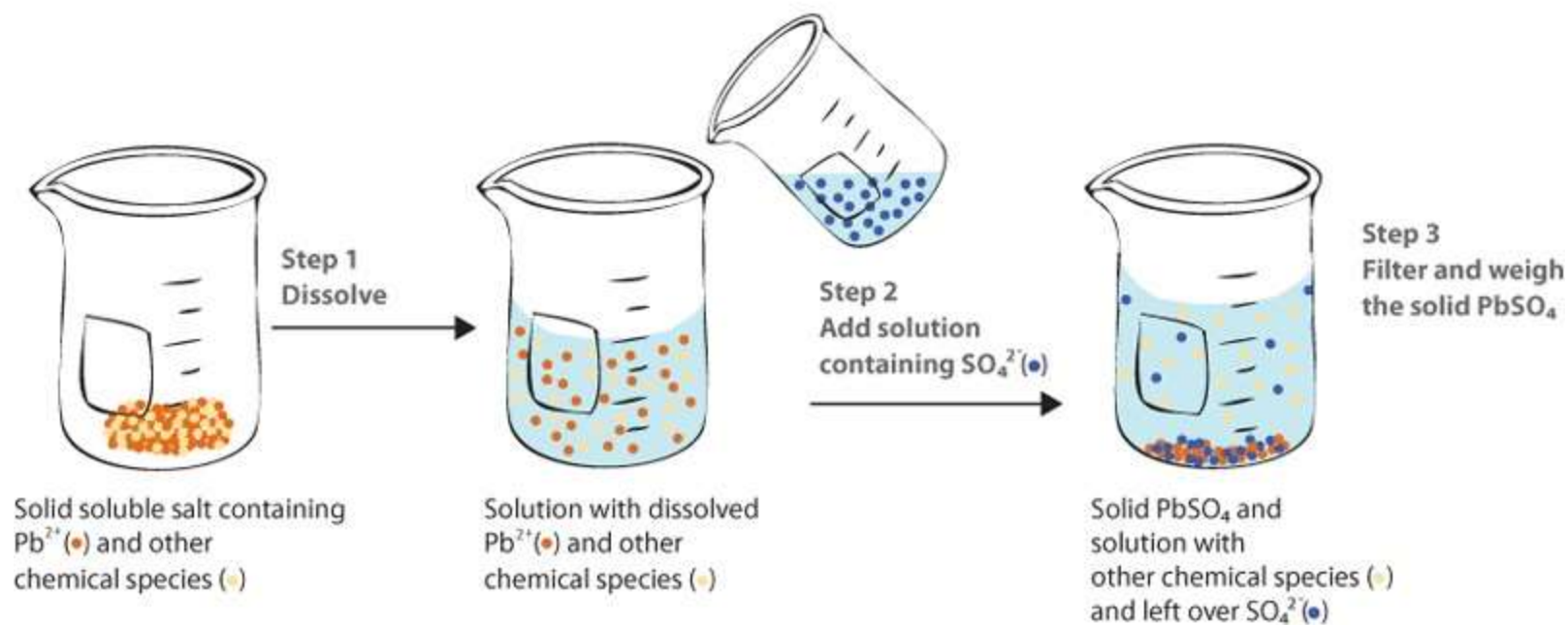
PROGRAM: B.PHARM I YEAR / I SEM.

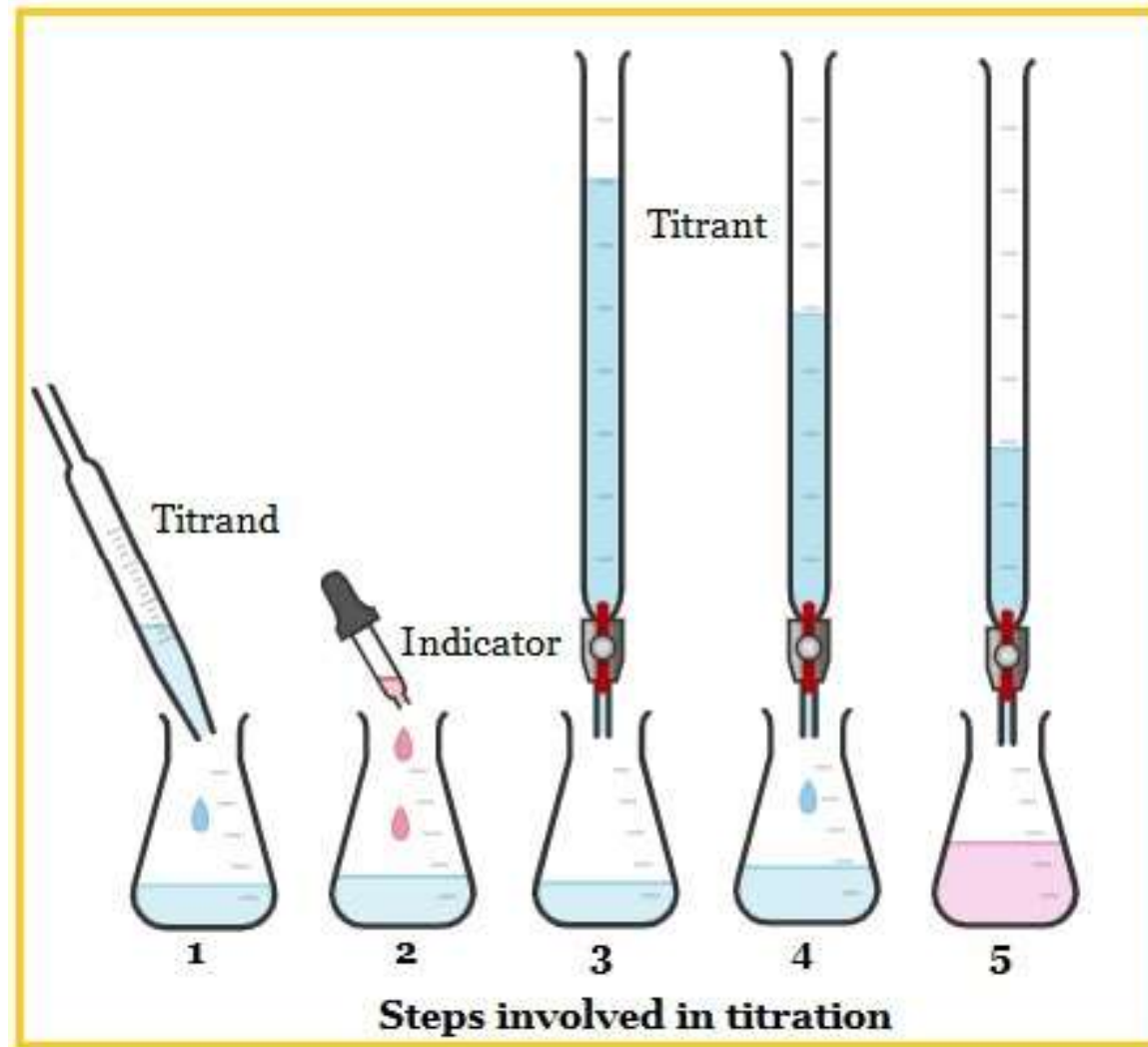
COURSE NAME : BP102T. PHARMACEUTICAL ANALYSIS -UNIT III.

SUB TOPIC :Precipitation titrations:-

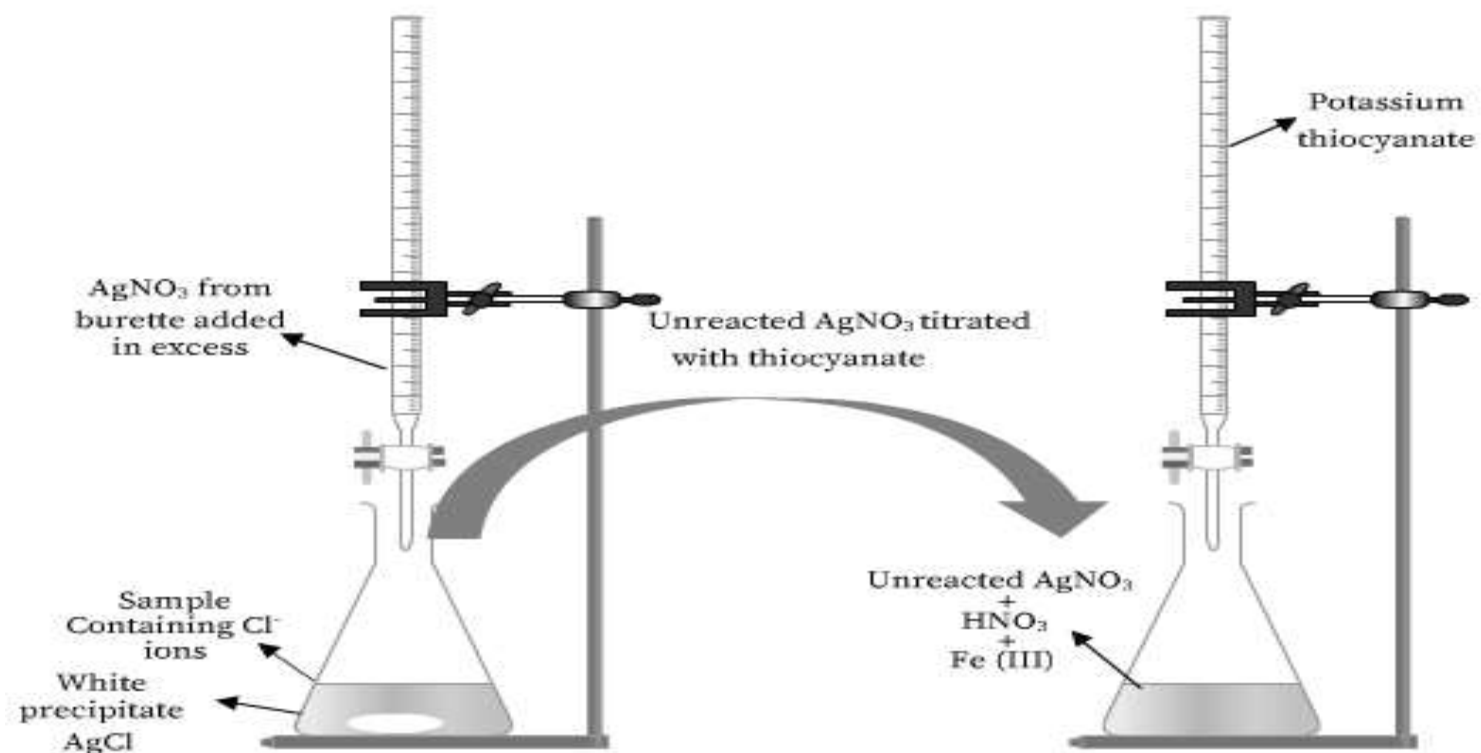
Mohr's method, Volhard's, Modified Volhard's, Fajans method and
Estimation of Sodium chloride.

Precipitation titrations: Precipitation titration is a titration based on formation of an insoluble precipitate.





Precipitation Titration – Principle:



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First, Cl^- is precipitated by excess AgNO_3



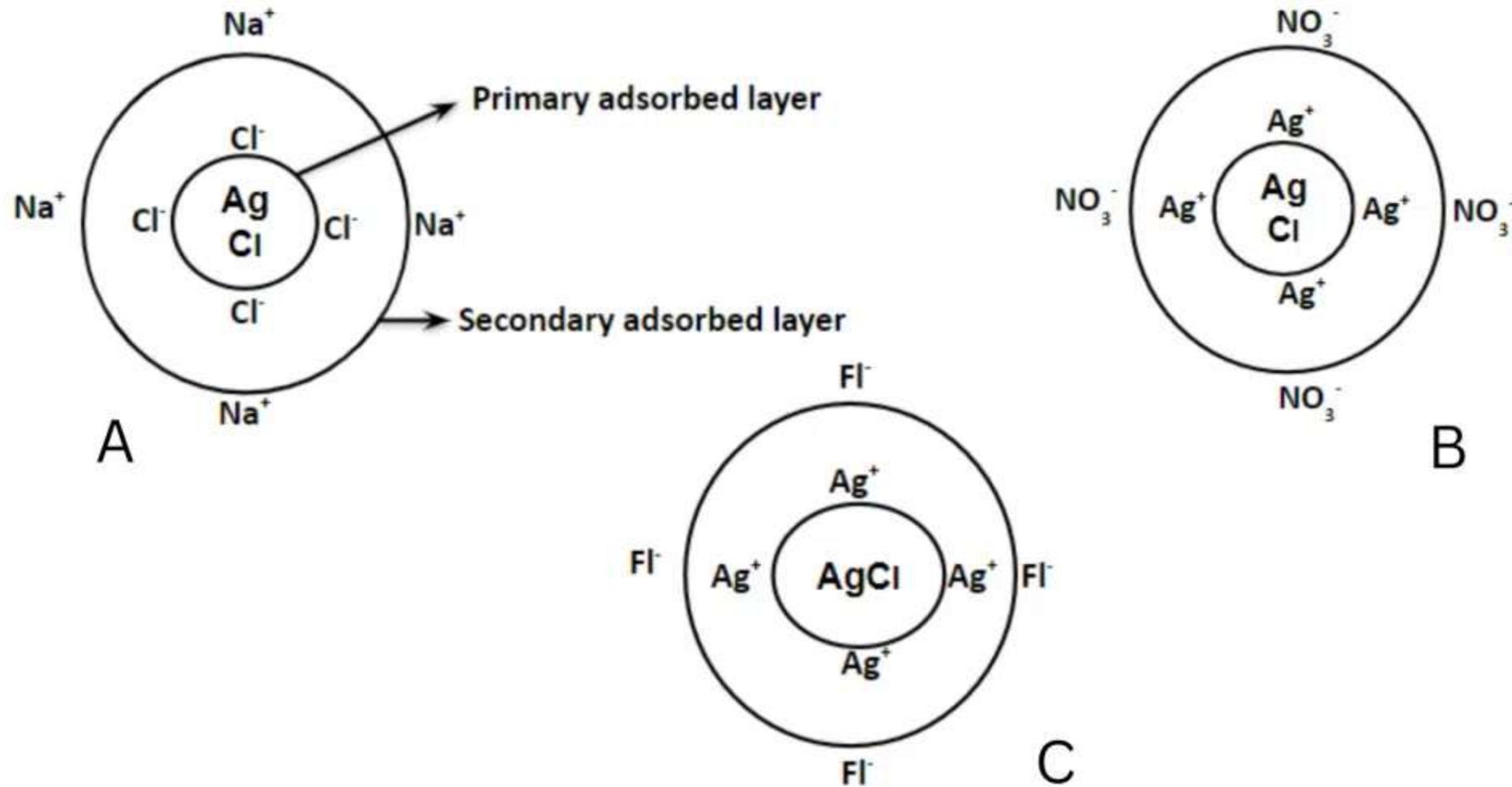
Removing $\text{AgCl}(\text{s})$ by filtration / washing

Excess Ag^+ is titrated with KSCN in the presence of Fe^{3+}



When Ag^+ has been consumed, a red complex forms as a result of:





Mechanism of adsorption

TYPES OF TITRATION

ACID-BASE TITRATION

- ④ Neutralization of an acid with a base
- ④ Purpose: Determining acid or base concentration
- ④ Indicator: Phenolphthalein, methyl orange
- ④ Example: NaOH vs HCl



REDOX TITRATION

- ④ Oxidation-reduction reaction
- ④ Purpose: Determining oxidizing or reducing agents
- ④ Indicator: Self-indicating
- ④ Example: Vitamin C determination



COMPLEXOMETRIC TITRATION

- ④ Complex formation between a metal ion and a ligand
- ④ Purpose: Determining metal ion concentration
- ④ Indicator: Eriochrome-Black-T
- ④ Example: Ca^{2+} , Mg^{2+} in water

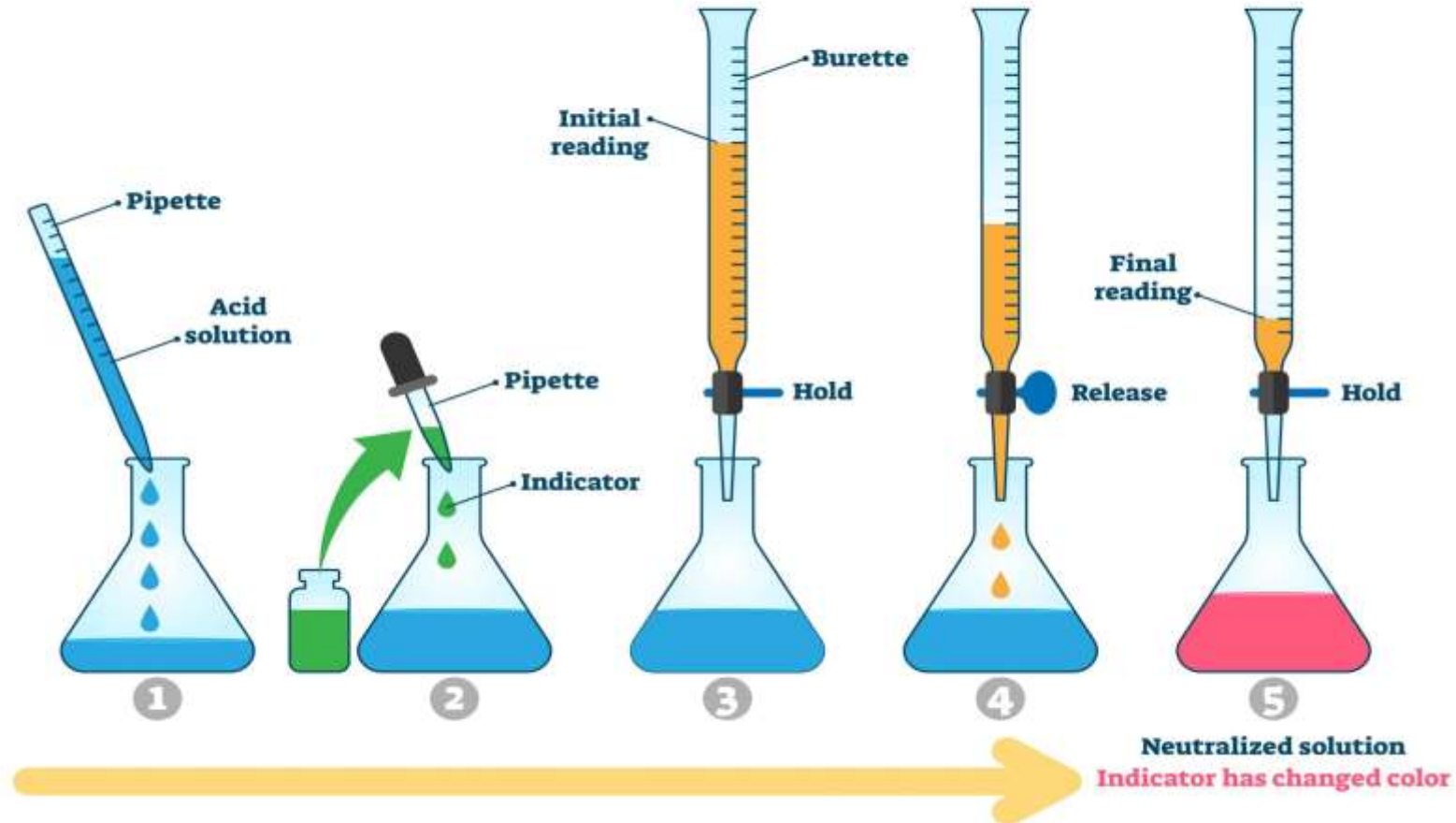


PRECIPITATION TITRATION

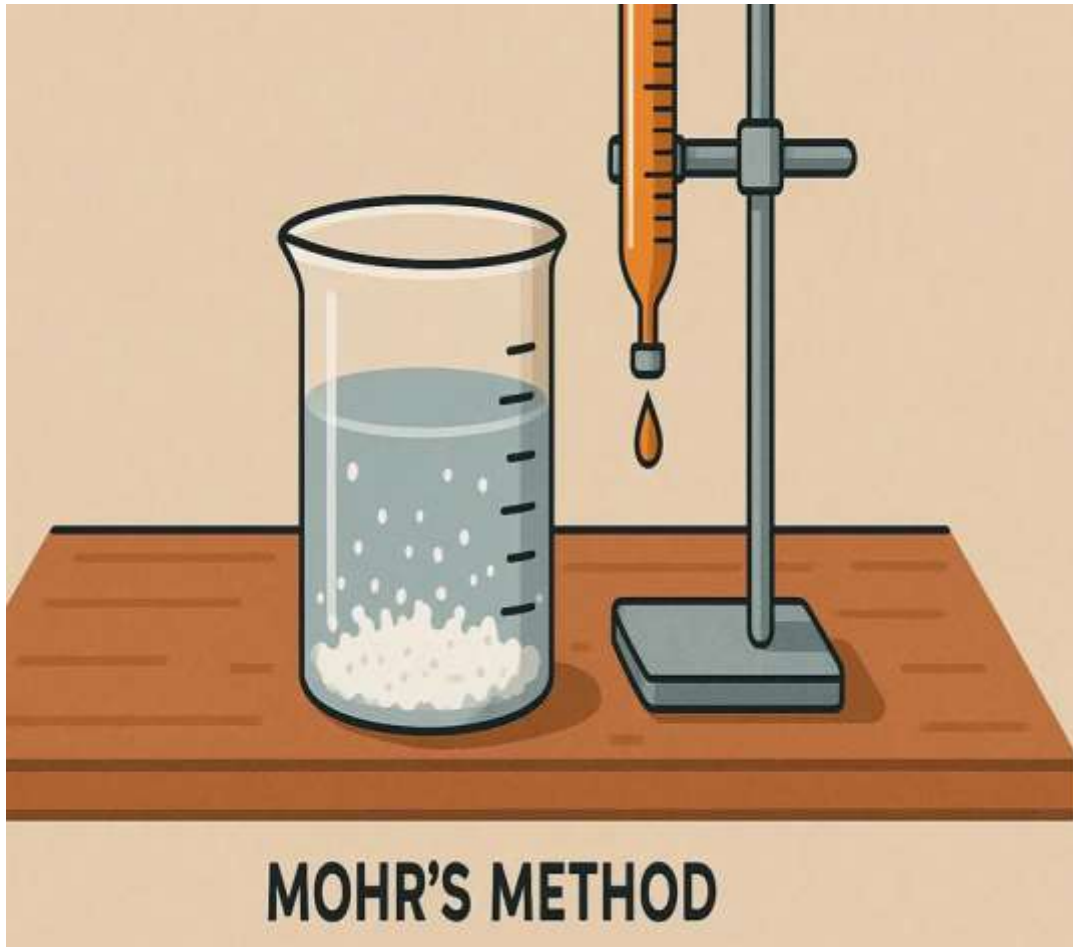
- ④ Precipitate formation
- ④ Purpose: Quantifying ions forming insoluble salts
- ④ Indicator: Chromate ion
- ④ Example: Cl^- in water



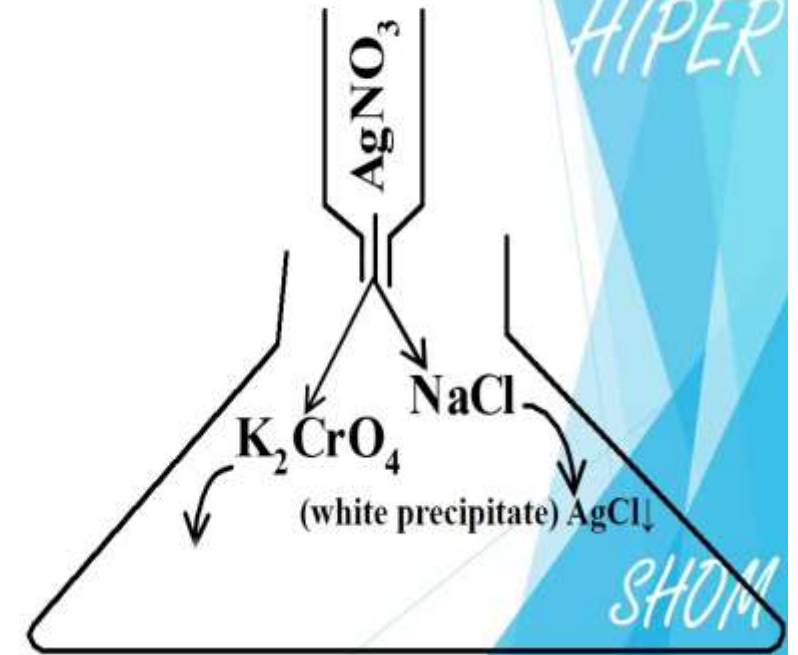
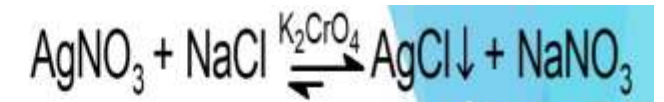
TITRATION



Mohr's method, Volhard's, Modified:-



Mohr's method:

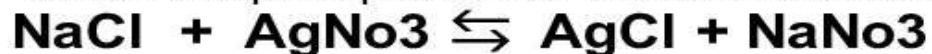


Principle of Mohr's method

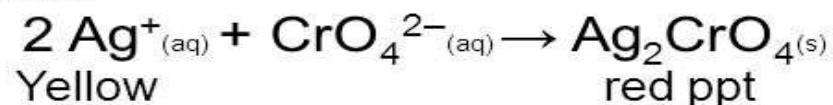
Mohr's method is a direct titration using standard solution of silver nitrate in a neutral medium (why?).

Chloride is titrated with AgNO_3 solution. A soluble chromate salt (K_2CrO_4) is added as the indicator. This produces a yellow color solution.

When the precipitation of the chloride is complete.

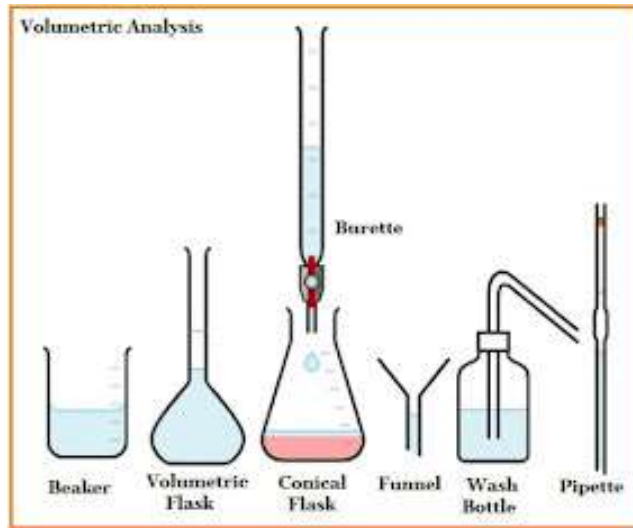


At the end point: The first excess of Ag^+ reacts with the indicator to precipitate red silver chromate as a second precipitate after precipitation of all chlorides as silver chloride.



The Mohr's method must be performed at a pH about 8.

This method is useful for determining Cl^- in neutral or unbuffered solutions such as drinking water.



Volhard Method

➤ Used as a procedure for titrating Ag^+ ; determination of Cl^- requires a back-titration

- First, Cl^- is precipitated by excess AgNO_3



- Excess Ag^+ is titrated with KSCN in the presence of Fe^{3+}



- When Ag^+ has been consumed, a red complex forms



❑ Volhard Method :

Jacob Volhard (1834-1910)

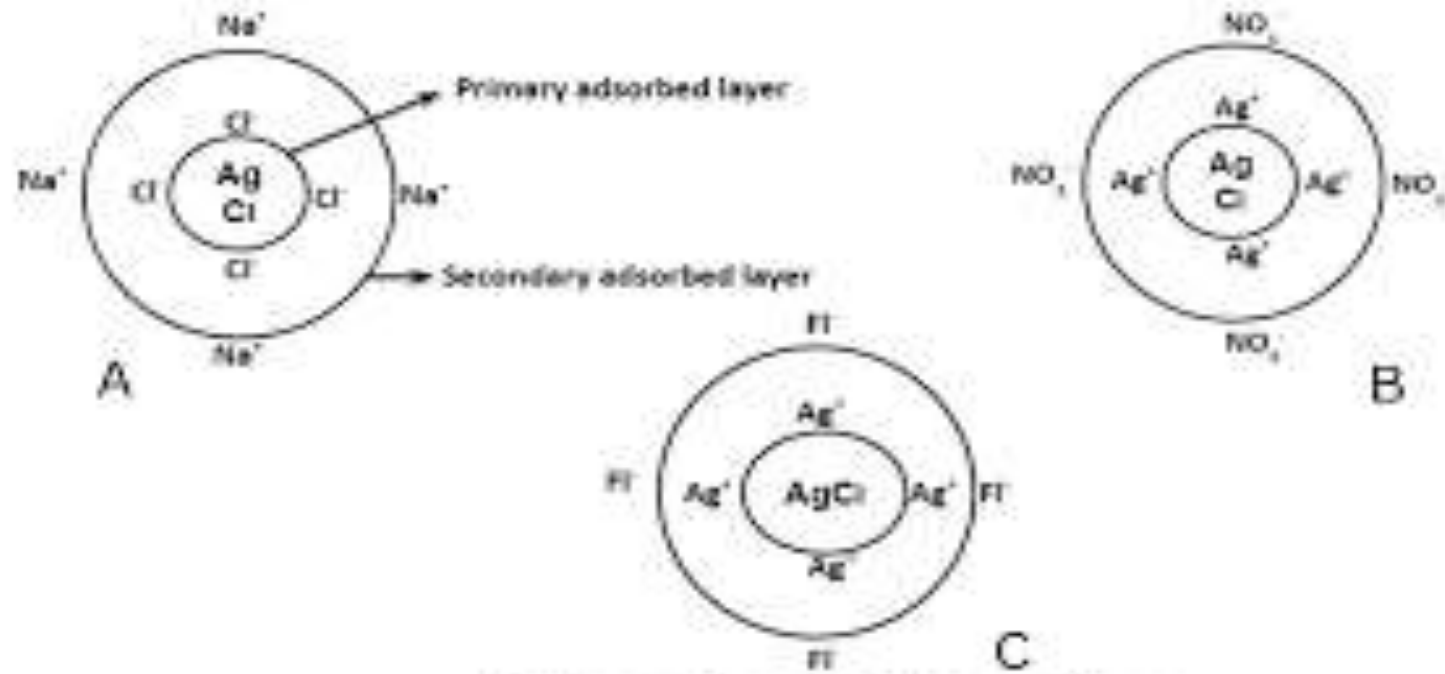


This is an indirect method for chloride determination where an excess amount of standard Ag^+ is added to the chloride solution containing Fe^{3+} as an indicator. The excess Ag^+ is then titrated with standard SCN^- solution until a red color is obtained which results from the reaction:



Difference between Mohr and Volhard method

<i>Mohr method</i>	<i>Volhard method</i>
Silver nitrate is used as the titrant and chloride ion solution as the analyte	Excess silver nitrate is used to precipitate chlorides, which is then titrated with potassium or sodium thiocyanate
It is a direct method of titration	It is an indirect method of titration
Potassium chromate is used as an indicator	Ferric ammonium sulfate is used as an indicator
It requires neutral or alkaline conditions	It requires acidic conditions
It is performed at room temperature	It is performed at temperatures below 20°C



Mechanism of adsorption

Fajans Method :

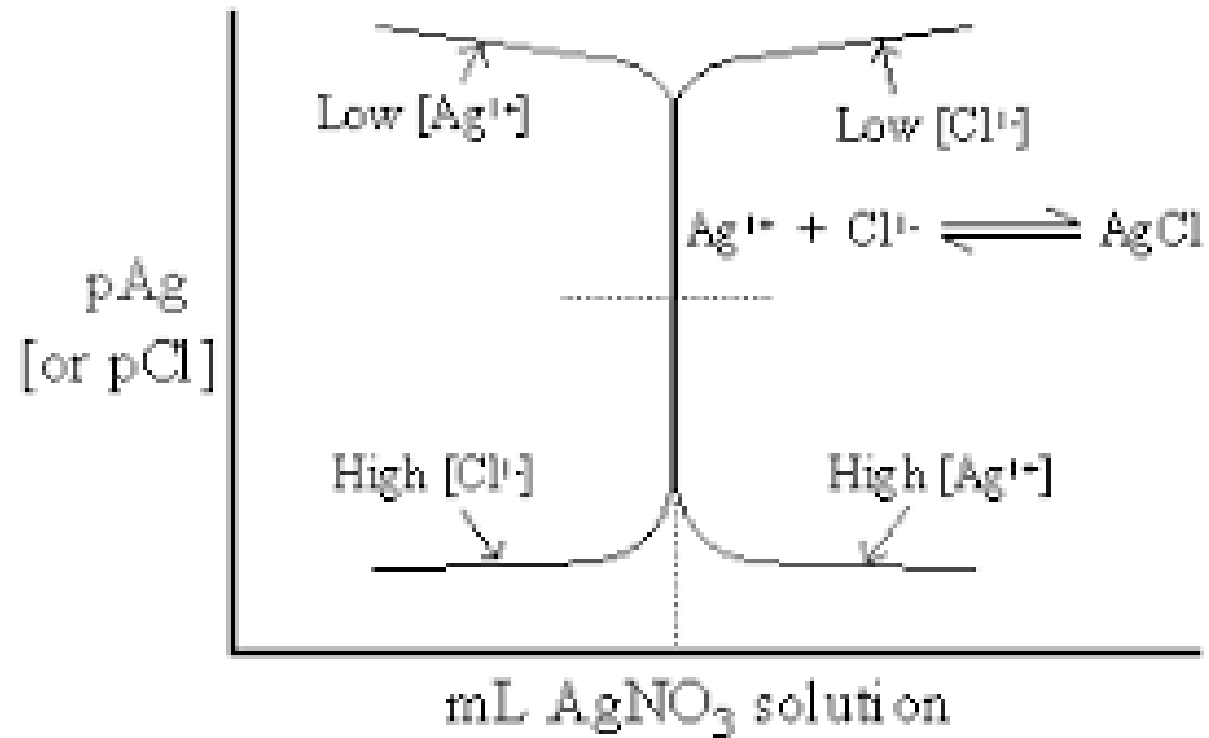
Kazimierz Fajans (1887-1975)



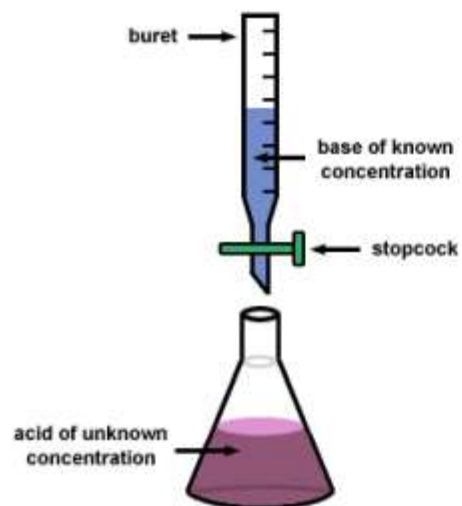
Fluorescein and its derivatives are adsorbed to the surface of colloidal AgCl. After all chloride is used, the first drop of Ag⁺ will react with fluorescein (FI⁻) forming a reddish color.



Among these methods, the Volhard Method is widely used because we can detect the end point of precepitation titration very well.



Mohr Titration for Sodium Quantification



ASSESSMENT QUESTIONS:-

Mohr's Method:

1. Calculate the chloride ion concentration in a sample if 25 mL of NaCl solution requires 30.0 mL of 0.1 M AgNO_3 to reach the endpoint.

Assessment Question:

How would you calculate the molarity of NaCl in the given example? Show your calculation.

Volhard's Method:

2. If an excess of 0.1 M AgNO_3 is added to a halide solution and requires 20.0 mL of 0.1 M KSCN for back-titration, calculate the concentration of halides in the sample.

Assessment Question:

Given the above information, how would you determine the concentration of halide ions in the sample?

Recommended Books:

1. A.H. Beckett & J.B. Stenlake's, Practical Pharmaceutical Chemistry Vol I & II, Stahlone, Press of University of London.
2. A.I. Vogel, Text Book of Quantitative Inorganic analysis.
3. P. Gundu Rao, Inorganic Pharmaceutical Chemistry.
4. Bentley and Driver's Textbook of Pharmaceutical Chemistry.
5. John H. Kennedy, Analytical chemistry principles.

