



CHEMISTRY

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In this class..

Estimation of amount of chloride content
in a water sample

Expt. No. : 3

Drinking water standards



INDIAN STANDARD SPECIFICATIONS FOR DRINKING WATER IS: 10500

S.NO.	Parameter	Requirement desirable Limit	Remarks
1.	Colour	5	May be extended up to 50 if toxic substances are suspected
2.	Turbidity	10	May be relaxed up to 25 in the absence of alternate
3.	pH	6.5 to 8.5	May be relaxed up to 9.2 in the absence
4.	Total Hardness	300	May be extended up to 600
5.	Calcium as Ca	75	May be extended up to 200
6.	Magnesium as Mg	30	May be extended up to 100
7.	Copper as Cu	0.05	May be relaxed up to 1.5
8.	Iron	0.3	May be extended up to 1
9.	Manganese	0.1	May be extended up to 0.5
10.	Chlorides	250	May be extended up to 1000
11.	Sulphates	150	May be extended up to 400
12.	Nitrates	45	No relaxation
13.	Fluoride	0.6 to 1.2	If the limit is below 0.6 water should be rejected, Max. Limit is extended to 1.5

Drinking water standards require chloride level not to exceed
250 mg/l (salinity)

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Experiment



□ Aim :

To estimate the amount of chloride in a water sample by **Mohr's method**

□ Materials required:

Conical flask, 100 mL standard flask, pipette, burette, funnel, glass rod.

□ Chemicals required :

Silver nitrate solution (AgNO_3), standard NaCl solution, K_2CrO_4 indicator, sample water and distilled water.

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Precipitation reaction



- ❑ A precipitation reaction refers to the **formation of an insoluble salt** when two solutions containing soluble salts are combined.
- ❑ The insoluble salt that falls out of solution is known as the **precipitate**, hence the reaction's name.
- ❑ Precipitation reactions can help determine the presence of **various ions in solution**.



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Argentometric titration (precipitation)



- ❑ A titration involving the **silver (I) ion**.
- ❑ Used to determine the **amount of chloride** present in a sample. The sample solution is titrated **against a solution of silver nitrate of known concentration**.
- ❑ **The indicator (potassium chromate)** is added to visualize the endpoint, demonstrating presence of silver ions, solubility product of silver chromate exceeded, and it forms a **brick reddish-brown precipitate**.
- ❑ This stage is taken as evidence that all chloride ions have been consumed and only **excess silver ions have reacted with chromate ions**.

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Mohr's method (1855 by Karl Friedrich Mohr)



- ☐ It is an example of precipitation reaction.
- ☐ The reaction between chloride and silver nitrate is direct and simple. It proceeds as follows:



- ☐ The completion of the reaction in this case is observed by employing potassium chromate (K_2CrO_4) solution as an indicator.
- ☐ K_2CrO_4 indicator will not be precipitated as Ag_2CrO_4 , until all the chlorides in the solution have been precipitated as AgCl

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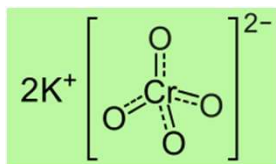
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Potassium chromate indicator



- ☐ Potassium chromate is an inorganic compound with the formula (K_2CrO_4).
- ☐ It is the potassium salt of the chromate anion.



- ☐ Before the addition of any silver nitrate the chromate indicator gives the clear solution a **bright lemon-yellow color.**

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Principle



- ☐ Before the equivalence point there is an excess of the chloride ion in solution.
- ☐ As the silver ions are added, the chloride ion concentration slowly decreases and the amount of precipitate, silver chloride, increases.
- ☐ At the equivalence point, all of the chloride ions in solution have reacted and no more precipitate will form.
- ☐ After the equivalence point, the excess silver ion concentration increases and is in excess.
- ☐ The equivalence point of a precipitation titration is defined as being the point at which the precipitate no longer forms.
- ☐ Silver cations are reduced to silver metal in bright light. Therefore silver solutions are typically stored in dark colored containers and titrations are performed in low light

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Principle contd..



- ☐ Before the titration endpoint, addition of Ag^+ ions leads to formation of silver chloride precipitate, making the solution cloudy.
- ☐ At the end point all the Cl^- ions have precipitated. The slightest excess of Ag^+ precipitates with the chromate indicator giving a slight red-brown coloration.

$$2\text{AgNO}_3 + \text{K}_2\text{CrO}_4 \rightarrow \text{Ag}_2\text{CrO}_4 + 2\text{KNO}_3$$
- ☐ If addition of Ag^+ is continued past the endpoint, further silver chromate precipitate is formed and a stronger red-brown color results.



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Principle, requirements



- ☐ The **rate of reaction** between the precipitant and the substance to be precipitated **must be fast**.
- ☐ There should **be no co-precipitation**. The precipitate should be **insoluble**.
- ☐ A **suitable indicator** should be used to locate the end point of the titration (for colorless precipitate) .
- ☐ The process of precipitation titration is completely based on the **concept of solubility product**. The solubility product constant (K_{sp}) describes the equilibrium between a solid and its constituent ions in a solution.

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Principle

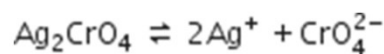


- ☐ Silver can form a precipitate by reaction with chloride (AgCl ; $K_{sp} = 1.8 \times 10^{-10}$) and with chromate (Ag_2CrO_4 ; $K_{sp} = 1.9 \times 10^{-12}$).



$$K_{sp} = S \times S \quad (K_{sp} = 1.8 \times 10^{-10})$$

$$S = \sqrt{K_{sp}} = 1.34 \times 10^{-5}$$



solubility product

$$K_{sp} = (2S)^2 \times S = 4S^3$$

$$K_{sp} = (1.1 \times 10^{-12})$$

$$S = \sqrt[3]{\frac{K_{sp}}{4}} = 0.65 \times 10^{-4}$$

AgCl is less soluble than Ag_2CrO_4 so if Ag^+ ions are added to a solution containing BOTH Cl^- ions and CrO_4^{2-} ions, the added Ag^+ ions will have a greater attraction for the Cl^- ions, so the precipitate of AgCl will form first.

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Key points

- Has to be performed at a neutral or weak basic solution of pH 7-9 (or 6-10)
- In a lower pH (acid solution)

$$\text{CrO}_4^{2-}(\text{aq}) + \text{H}^+(\text{aq}) \rightarrow \text{H}_2\text{CrO}_4$$

$$\text{H}_2\text{CrO}_4 \leftrightarrow 2\text{H}^+(\text{aq}) + \text{CrO}_4^{2-}(\text{aq})$$
- In a higher pH (basic solution)

$$\text{Ag}^+(\text{aq}) + \text{OH}^-(\text{aq}) \rightarrow \text{AgOH}(\text{s})$$
- It is important to rinse the burette with the titrant before beginning the titration
- AgNO_3 to be stored in brown bottle

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Procedure

Titration I : Standardization of silver nitrate solution

- ❑ **10 ml of standard NaCl solution (0.017 N)** is pipetted out into a clean conical flask.
- ❑ **4-5 drops of 2% K_2CrO_4** indicator is added to it. The solution turns **bright lemon yellow** in color.
- ❑ It is titrated against **AgNO_3 solution** taken in the burette.
- ❑ During each addition of AgNO_3 , the content in the conical flask is **shaken well**. At the end point, **yellow color changes into reddish brown** (which does not disappear even after shaking).
- ❑ The titration is repeated till the **concordant value** is obtained

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Procedure Titration II : Estimation of chloride



- ☐ The given chloride solution is **diluted to 100 ml** using distilled water in a standard flask. Exactly **10 ml** of this solution is pipetted out into a clean conical flask.
- ☐ To this solution **4-5 drops of 2% K_2CrO_4** indicator is added and is titrated against standardized $AgNO_3$ solution from the burette.
- ☐ The addition of $AgNO_3$ solution is continued until the solution produced a **permanent reddish-brown color**. The titration is repeated till the concordant value is obtained.
- ☐ From the **volume of $AgNO_3$ consumed**, the strength of chloride and hence its **amount is calculated**.

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Tabular column



Table I : Standardization of silver nitrate solution

S.No.	Volume of NaCl solution (ml)	Burette Reading (ml)		Concordant Value (ml)	Indicator
		Initial	Final		
					2 % Potassium chromate (1 mL)

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Calculation



Titration I : Standardization of silver nitrate solution

$$\begin{aligned}
 \text{Normality of NaCl Solution} &= \\
 \text{Volume of NaCl Solution} &= \\
 \text{Volume of AgNO}_3 \text{ Solution} &= V_2 \text{ (end point)} \\
 \text{Normality of AgNO}_3 \text{ Solution} &= \text{---} ? N_2 \\
 N_2 &= V_1 \times N_1 / V_2
 \end{aligned}$$

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Tabular column



Table II : Estimation of chloride

S.No.	Volume of chloride solution (ml)	Burette Reading (ml)		Concordant Value (ml)	Indicator
		Initial	Final		
					2 % Potassium chromate (1 mL)

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Calculation



Titration II : Estimation of chloride content

$$\begin{aligned}
 \text{Volume of chloride solution} &= 20 \text{ ml } (V_1) \\
 \text{Normality of chloride solution} &= \text{---} ? N_1 \\
 \text{Volume of AgNO}_3 \text{ solution} &= V_2 \text{ (end point)} \\
 \text{Normality of AgNO}_3 \text{ solution} &= N_2 \text{ (from tit I)} \\
 N_1 &= V_2 \times N_2 / V_1
 \end{aligned}$$

Amount of chloride ion present in the water of the given solution

$$\frac{N_1 \times \text{Equivalent wt of Cl}}{10} \quad (35.45)$$

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Table 1



S No	Volume of pipette solution (mL)	Burette reading		Concordant value (mL)	Indicator used
		Initial (mL)	Final (mL)		
1	10	0	13.4	13.4	K ₂ CrO ₄
2	10	0	13.4		

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Table 2



S No	Volume of pipette solution (mL)	Burette reading		Concordant value (mL)	Indicator used
		Initial (mL)	Final (mL)		
1	10	0	19.7	19.7	K ₂ CrO ₄
2	10	0	19.7		

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Drawbacks



- ☐ The Mohr's method suffers with a drawback as it has an inherent titration error.
- ☐ The end point is not observed immediately after the equivalence point but after a little over titration, i.e., after adding a little extra silver nitrate.
- ☐ A definite amount of indicator must be used to provide a certain concentration of chromate ions, otherwise silver chromate may form too soon or not soon enough.
- ☐ Caution should be made to notice indicator color change as it can varies person to person. The usual range is 0.2 to 0.4 mL of titrant.

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Result



☐ Amount of chloride present in the given solution is
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Thank you all for your attention

Information presented here were collected from various sources –
textbooks, articles, manuscripts, internet and newsletters. All the
researchers and authors of the above mentioned sources are greatly
acknowledged.