#### **EXPERIMENT NO:-4**

# Aim: To determine chloride ion in a given water sample by argentometric method

### Theory:

Chlorides are present in water usually as NaCl,  $MgCl_2$  and  $CaCl_2$ . Although chlorides are not harmful as such, their concentration above 250 ppm imparts a peculiar taste to the water thus rendering the water unacceptable for drinking purpose. Further existence of unusually high concentrations of chloride in a water sample indicates pollution from domestic sewage or from industrial waste water. Salts like  $MgCl_2$  may undergo hydrolysis under the high pressure and temperature prevailing in the boiler, generating hydrochloric acid which causes corrosion in boiler parts. Chlorides in the form of  $MgCl_2$  and  $CaCl_2$  cause permanent hardness and are a source of trouble not only at our house hold but also in industries.

By Argentometric method, estimation of chlorides in a water sample can be quantitatively carried out in a neutral medium, since silver hydroxide gets precipitated in alkaline medium leading to erroneous results and in acidic medium some chromate is converted to dichromate.

$$Ag^{+} + OH^{-} = AgOH; 2CrO_{4}^{2-} + 2H^{+} \rightarrow 2HCrO_{4}^{-} \rightarrow Cr_{2}O_{7}^{2-} + H_{2}O_{4}^{-}$$

When silver nitrate is added to a solution of sodium chloride containing potassium chromate, Ag<sup>+</sup> first reacts with Cl<sup>-</sup> to give a white precipitate of AgCl and not Ag<sub>2</sub>CrO<sub>4</sub>.

$$Ag^{+} + Cl^{-}$$
 AgCl (white precipitate)  $(K_{SP1} = 1.2 \times 10^{-10})$ 

$$2Ag^{+} + CrO_{4}^{2-}$$
 Ag<sub>2</sub>CrO<sub>4</sub> (Red precipitate)  $(K_{SP2} = 1.7 \times 10^{-12})$ 

Where the  $K_{sp}$  values in parenthesis indicate their respective solubility products (the greatest possible value of ionic product for a saturated solution, beyond which precipitation occurs).

If the solubility of the solids is indicated by  $S_1$  and  $S_2$ 

Then, 
$$K_{SP1} = S_1 \times S_1$$
 or,  $S_1^2 = K_{SP1}$  or,  $S_1 = \sqrt{K_{SP1}} = 1.732 \times 10^{-5}$ 

$$K_{SP2} = (2S_2)^2 \times S_2$$
 or,  $4S_2^3 = K_{SP2}$  or,  $S_2 = (K_{SP2}/4) = 1.076 \times 10^{-4}$ 

Thus solubility of Ag<sub>2</sub>CrO<sub>4</sub> being more than that of AgCl, the former will start to precipitate after complete precipitation of later. So, during the titration the solution will be turbid due to appearance of AgCl and the equivalence point of the precipitation titration is

indicated by the appearance of red color (Ag<sub>2</sub>CrO<sub>4</sub>) from yellow color (K<sub>2</sub>CrO<sub>4</sub>). From this titre value concentration of total chloride can be calculated.

#### Apparatus and Reagents:

Burette

• Pipette (10ml and 25ml)

Conical flask (100ml)

Beaker (100ml)

Funnel

primary standard sodium chloride solution secondary standard silver nitrate solution potassium chromate indicator

water sample

#### Procedure:

Step-1: Standard (~ N/50) sodium chloride solution and silver nitrate solution is provided

#### Step-2: Standardization of silver nitrate solution

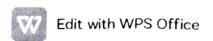
Pipette out 10 ml of ( $\sim$  N/50) sodium chloride solution in a 100 ml conical flask, add 4-5 drops of potassium chromate indicator into it and titrate the resulting solution with ( $\sim$  N/50) silver nitrate solution taken in burette. Add drop wise silver nitrate solution from burette until yellow color start changing to brown red colour. The titration is repeated two times to get a concordant reading.

#### Step-3: Estimation of chloride content

Pipette out 25 ml water sample in a 100 ml conical flask, add 4-5 drops of potassium chromate indicator into it and titrate the resulting solution with

(~ N/50) silver nitrate solution taken in burette. Add drop wise silver nitrate solution from burette until yellow colour start changing to brown red colour. The titration is repeated two times to get a concordant reading.

#### Result and Calculations:



## Table-1: Standardization of silver nitrate solution

Strength of sodium chloride solution (S<sub>1</sub>) = ....... (N)

No of observation	Volume of sodium chloride solution (V <sub>1</sub> ml)	Burette Reading		Volume of AgNO <sub>3</sub> required (ml)	Mean volume of AgNO <sub>3</sub> required (V <sub>2</sub> ml)
		Initial (ml)	Final (ml)		
1	10				
2	10				
3	10				

We know,  $V_1S_1 = V_2S_2$ 

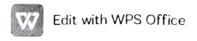
 $S_2 = V_1S_1/V_2$  (N)

Table-2: Estimation of chloride content:

Strength of AgNO<sub>3</sub> ( $S_2$ ) = .....(N)

No of observation	Volume of Water sample taken (ml)	Burette Reading		Volume of AgNO <sub>3</sub> required (ml)	Mean volume of AgNO3 required (V')ml
		Initial (ml)	Final (ml)		
1	25				
2	25				
3	25				

1000 ml 1 (N) AgNO $_3$  = 35.5 gm of chloride ion



V' ml  $S_2$  (N) AgNO $_3$  = (35. 5 X V' X  $S_2$ ) / 1000 gm of chloride ion

= x gm of chloride ion

Now, 25 ml water sample contains x gm of chloride ion

- $\therefore$  10<sup>6</sup> ml of water sample contains (x X 10<sup>6</sup>) / 25 gm of chloride ion
- $\therefore$  Chloride content in the given water sample = (x X 10<sup>6</sup>) / 25 ppm

#### Precaution:

- i. Before starting of the experiment the whole apparatus should be washed with distilled water which is free of chlorine.
- ii. Carefully handle silver nitrate and avoid any skin contact since silver nitrate is corrosive and leaves blacks spots.
- iii. Add same amount of indicator each time.

#### Conclusion:

The chloride content in a given water sample is =...... ppm