

4.0 EXPERIMENT ON DETERMINATION OF CHLORIDES

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4.0 EXPERIMENT ON DETERMINATION OF CHLORIDES

PREAMBLE:

“How to determine chlorides in Water and Wastewater”.

Test procedure is in accordance to IS: 3025 (Part 32) - Reaffirmed 2003.

In addition to our Indian Standard, we also discuss in brief regarding the procedure stated in

- (1) APHA Standard Methods for the Examination of Water and Wastewater - 20th Edition. Method 4500 - Cl⁻ - B.
- (2) Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, USEPA, Method 9253.

4.1 AIM

To determine the chlorides of given water sample with the stipulations as per **IS: 3025 (Part 32) - Reaffirmed 2003.**

4.2 INTRODUCTION

Chlorides are widely distributed as salts of calcium, sodium and potassium in water and wastewater. In potable water, the salty taste produced by chloride concentrations is variable and dependent on the chemical composition of water. The major taste producing salts in water are sodium chloride and calcium chloride. The salty taste is due to chloride anions and associated cations in water. In some water which is having only 250 mg /L of chloride may have a detectable salty taste if the cat-ion present in the water is sodium. On the other hand, a typical salty taste may be absent even if the water is having very high chloride concentration for example 1000 mg /L.

This is because the predominant cation present in the water is not sodium but either calcium or magnesium may be present.

4.2.1 Environmental Significance

- Chlorides associated with sodium (Sodium Chloride) exert salty taste when its concentration is more than 250 mg/L. These impact a salty taste to water. Chlorides are generally limited to 250 mg/L in water supplies intended for public water supply.

In many areas of the world where water supplies are scarce, sources containing as much as 2000 mg/L are used for domestic purposes without the development of adverse effect, once the human system becomes adapted to the water.

- It can also corrode concrete. Magnesium chloride in water generates hydrochloric acid after heating which is also highly corrosive and creates problem in boilers.
- Chloride determinations in natural waters are useful in the selection of water supplies for human use.
- Chloride determination is used to determine the type of desalting apparatus to be used.
- Chloride determination is used to control pumping of ground water from locations where intrusion of seawater is a problem.
- Chlorides interfere in the determination of chemical oxygen demand (COD).

4.3 PRINCIPLE

The amount of chloride present in water can be easily determined by titrating the given water sample with silver nitrate solution.

The silver nitrate reacts with chloride ion according to 1 mole of AgNO_3 reacts with 1 mole of chloride. The titrant concentration is generally 0.02 M.

Silver chloride is precipitated quantitatively, before red silver chromate is formed. The end of titration is indicated by formation of red silver chromate from excess silver nitrate.

The results are expressed in mg/L of chloride (Cl^- with a molecular weight of 35.453 g/mol).

4.4 MATERIALS REQUIRED

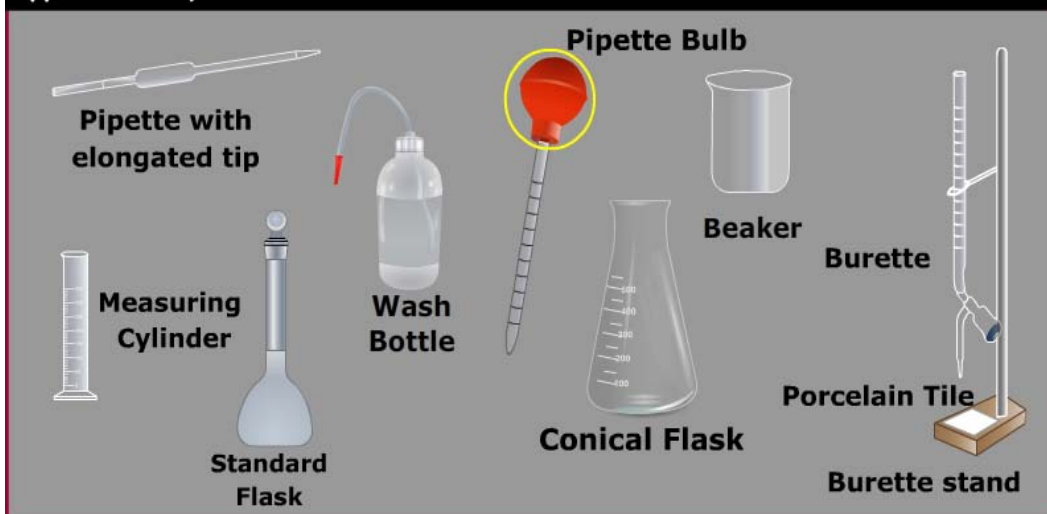
4.4.1 APPARATUS REQUIRED

1. Burette with Burette stand and porcelain tile
2. Pipettes with elongated tips
3. Conical flask (Erlenmeyer Flask)
4. Standard flask
5. Beaker
6. Wash bottle

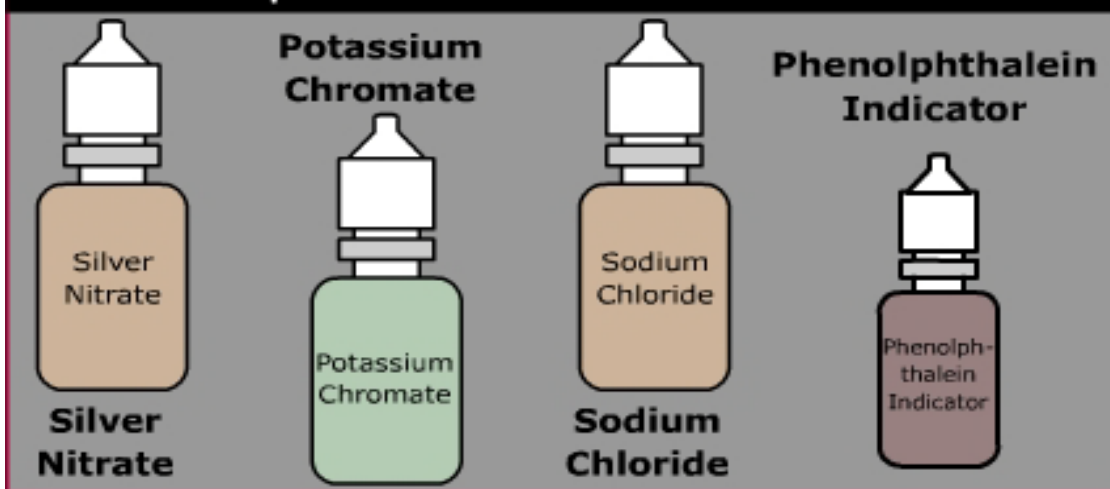
4.4.2 CHEMICALS REQUIRED

1. Silver nitrate
2. Phenolphthalein Indicator
3. Sodium chloride
4. Potassium chromate

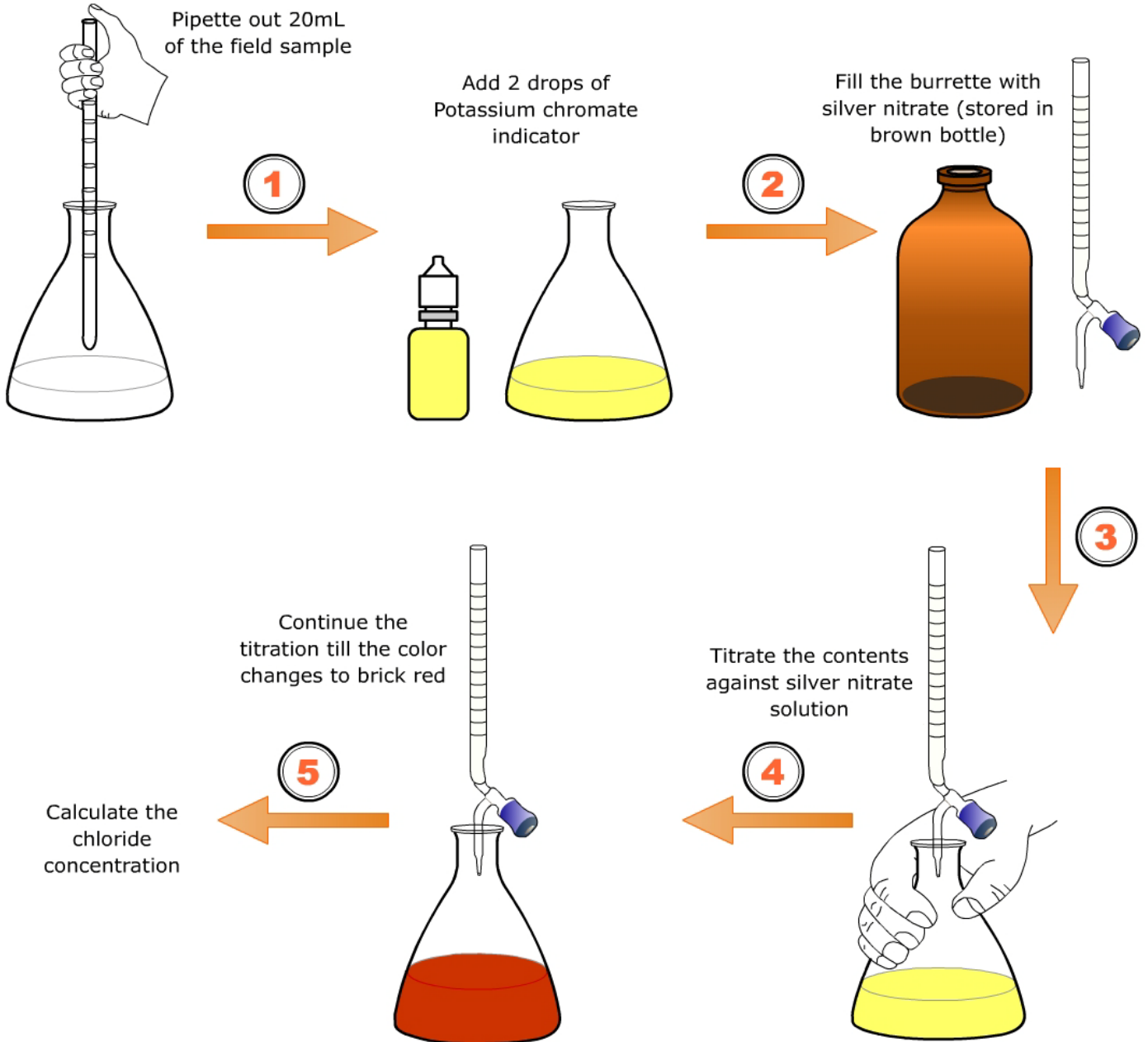
Apparatus Required



Chemicals Required



PROCEDURE CHART



4.5 SAMPLE HANDLING AND PRESERVATION

Preservation of sample is not practical. Because biological activity will continue after a sample has been taken, changes may occur during handling and storage.

If Analysis is to be carried out within two hours of collection, cool storage is not necessary. If analysis can not be started within the two hours of sample collection to reduce the change in sample, keep all samples at 4⁰ C.

Do not allow samples to freeze. Do not open sample bottle before analysis.

Begin analysis within six hours of sample collection

4.5.1 PRECAUTIONS

- AgNO₃ should be stored in a brown amber bottle and should not be exposed to sunlight.
- While handling AgNO₃, care should be taken so that it is not spilled on your skin.
- If it spills on your skin, the scar will remain at least for ten to fifteen days.

4.6 PROCEDURE

4.6.1 PREPARATION OF REAGENTS

Standard Sodium Chloride Solution

- Switch on the Electronic balance, keep the weighing pan, and set the reading to zero.
- Weigh 1.648g of Sodium chloride
- Transfer the contents to the beaker containing distilled water. Using glass rod, dissolve the contents thoroughly.
- Transfer the contents in the beaker to a 100 mL standard flask; fill distilled water up to 100 mL mark.
- Transfer it to 100mL standard flask using funnel

Standard Silver Nitrate (0.0282 N)

- Weigh 4.791g of Silver nitrate and transfer it to the beaker with distilled water.
- Transfer the contents in the beaker to a 100 mL standard flask, fill distilled water up to 100 mL mark.
- Standardize it against 0.0282 N NaCl solution. Store it in an amber bottle.

Potassium Chromate Indicator

- Weigh 25 g of Potassium Chromate. Transfer it to the beaker contains distilled water. Add few drops of Silver Nitrate solution until slight red precipitate is formed.
- Allow it to stand for 12 hours. After 12 hours filter the solution using filter paper and dilute the filtrate to 1000 mL using distilled water.

4.6.2 TESTING OF WATER SAMPLE

- Before starting the titration rinse the burette with silver nitrate solution. Fill the burette with silver nitrate solution of 0.0282 N. Adjust to zero and fix the burette in stand.
- Take 20 mL of the sample in a clean 250mL conical flask
- Add 1 mL of Potassium Chromate indicator to get light yellow color
- Titrate the sample against silver nitrate solution until the color changes from yellow to brick red. i.e., the end point.
- Note the volume of Silver nitrate added (**A**).
- The value of titration is 3.3 mL.
- Repeat the procedure for concordant values.

Blank Titration

- Take 20 mL of the distilled water in a clean 250mL conical flask
- Add 1 mL of Potassium Chromate indicator to get light yellow color
- Titrate the sample against silver nitrate solution until the color changes from yellow to brick red. i.e., the end point.
- Note the volume of silver nitrate added for distilled water (B).
- The value of titration is 0.2 mL

4.7 CALCULATION

4.7.1 TABLE

Sample No	Volume of Sample (mL)	Burette Reading (mL)		Volume of AgNO ₃ (mL)
		Initial	Final	
1.				
2.				
Blank (B)				

Burette solution: Silver Nitrate

Pipette solution: Sample

Indicator: Potassium chromate

End point: Appearance of Brick red color.

- The volume of water sample taken is 20 mL.
- The silver nitrate is taken in the Burette.
- For the first titration, the initial reading is 0 mL. The final reading is 3.3 mL.
- The volume of silver nitrate consumed to get the end point is 3.3 mL.
- For the second titration, the initial reading is 0 mL. The final reading is 3.3 mL.
- The volume of water sample taken is 20 mL.
- The silver nitrate is taken in the Burette.
- For the first titration, the initial reading is 0 mL. The final reading is 3.3 mL.
- The volume of silver nitrate consumed to get the end point is 3.3 mL.
- For the second titration, the initial reading is 0 mL. The final reading is 3.3 mL.
- The volume of silver nitrate consumed to get the end point is 3.3 mL.
- For the first and second titration, the burette reading is same so we have achieved concordant value. We can go for the calculations
- For the blank titration the end point is attained within the few drops of silver nitrate
- So the burette reading is 0.2 mL.
- Total amount of Chlorides mg/L of the given water sample is equal to
- Volume of AgNO_3 used for sample minus AgNO_3 used for blank multiplied by Normality multiplied by 35.45 multiplied by 1000 divided by Volume of sample taken
- Here the volume of silver nitrate used for sample is 3.3 mL and for blank is 0.2 mL
- Normality is 0.0282 N
- volume of sample taken is 20 mL Substituting the values in the formula and calculating we get the value 155 mg/L

4.7.2 DATA SHEET

DETERMINATION OF CHLORIDES DATA SHEET

Date Tested : August 30, 2010
Tested By : CEM Class, Group A
Project Name : CEM, NITTTR Lab
Sample Number : BH1
Sample Location : Perungudi (Lat 12' 57" 31.74 & Long 80'14" 8.82)
Sample Description : Surface water

Sl.No.	Volume of Sample (mL)	Burette Reading (mL)		Volume of EDTA (mL)
		Initial	Final	
1.	20	0	3.3	3.3
2.	20	0	3.3	3.3
Blank (B)	20	0	0.2	0.2

Specimen Calculation:

Volume of Silver Nitrate for sample (V_s) = 3.3 mL

Volume of Silver Nitrate for Blank (V_B) = 0.2 mL

Normality of EDTA = 0.0282 N

Volume of Sample = 20.0 mL

Equivalent weight of Chlorine = 35.45

$$\text{Chlorides mg/L} = \frac{(V_s - V_B) * \text{Normality} * 35.45 * 1000}{\text{volume of sample taken}}$$

To convert the sample size from mL to L, multiply the result by 1,000 mL/L

$$\text{Chlorides mg/L} = (3.3 - 0.2) \times 0.0282 \times 35.45 \times 1000 / 20$$

$$= 155 \text{ mg/L}$$

4.8 INTERPRETATION OF RESULTS

The amount of chloride present in the given water sample is = **155 mg/L**.

4.9 INFERENCE

The high concentrations of chloride ions mostly results in an unpleasant salty taste of water and it also aides the corrosion of plumbing system. Very high chloride content of water may also produce laxative effect. An upper limit of 250 mg/L has been set for the chloride ions. An increase in the normal chloride content of your water may indicate possible pollution from human sewage, animal manure or industrial wastes. As all aware the sea water is full of sodium chloride, the chloride levels will be much higher compared to the fresh water sources.

4.10 EVALUATION

1. The limit of chlorides in drinking water as per IS code is

- a) 200 ppm
- b) 225 ppm
- c) 250 ppm
- d) 500 ppm

2. Silver nitrate is stored in a brown bottle

- a) to avoid decomposition by sun light
- b) because it is dark in colour
- c) because the solution is colourless
- d) to avoid heat

3. The colour of Silver Chromate is

- a) Milky White
- b) pale Yellow
- c) Colourless
- d) Brick Red

4. When both hardness and chloride content are very high above 500 mg/L, then the water will be

- a) Non salty in nature
- b) Fit for drinking
- c) Salty in nature
- d) Soft water

5. Presence of chloride can corrode_____.

- a) GI pipes
- b) Rubber tubes
- c) PVC pipes
- d) Glass pipes

6. The chloride concentration in sewage is

- a) More concentrated than the municipal water supplied
- b) Equal concentration to the municipal water supplied
- c) Less concentrated than the municipal water supplied
- d) Only in trace

7. Chloride consumed by human beings

- a) Pass through the fecal matter as it is
- b) Gets changed into other forms
- c) Gets disappeared in the body
- d) Stored in bones

8. Chloride gives salty taste to water particularly when present as ____.

- a) Sodium chloride
- b) Magnesium chloride
- c) Potassium chloride
- d) Zinc chloride

9. The point at which a clear visual change is observed after the reaction between titrant and titrates is called

- a) End point
- b) Equivalence point
- c) Equal point
- d) Double equivalence point

10. Most common ion in the water is

- a) Fluoride
- b) Nitrate
- c) Chloride
- d) Sulphate

KEY TO ITEMS:

- 1) c
- 2) a
- 3) d
- 4) c
- 5) a
- 6) a
- 7) a
- 8) a
- 9) a
- 10) c