

11.0 EXPERIMENT ON DETERMINATION OF RESIDUAL CHLORINE

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11.0 EXPERIMENT ON DETERMINATION OF RESIDUAL CHLORINE

PREAMBLE:

“How to determine residual chlorine in Water and Wastewater”.

Test procedure is in accordance to IS: 3025 (Part 26) - 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 C.
- (2) Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, USEPA, Method 330.2.

11.1 AIM

To determine residual chlorine in the given water sample with the stipulations as per **IS: 3025 (Part 26) - Reaffirmed 2003**.

11.2 INTRODUCTION

- ✓ Why we should measure residual chlorine?
- ✓ What do we mean by the term “Residual Chlorine”?
- ✓ What is the source or reason for presence of chlorine in water or waste water?
- ✓ Is it mandatory to measure residual chlorine in water or waste water?

Before learning what is residual chlorine and why we need to measure residual chlorine. Let us first understand the importance of adding chlorine.

Treated or filtered water is deemed to be fit for consumption only if it is devoid of disease producing microorganism.

Chlorination is primarily adopted to destroy or deactivate disease-producing microorganisms in the public water supplies and polluted rivers.

Chlorine is usually added to water in gaseous form or as sodium or calcium hypochlorite.

It has been practiced over several years.

When chlorine is added to water, some of the chlorine reacts first with organic materials and metals in the water and is not available for disinfection (this is called the chlorine demand of the water).

The remaining chlorine concentration after the chlorine demand is accounted for is called total chlorine.

Total chlorine is further divided into: 1) the amount of chlorine that has reacted with nitrates and is unavailable for disinfection which is called combined chlorine and, 2) the free chlorine, which is the chlorine available to inactivate disease-causing organisms, and thus a measure to determine the potability of water

The word "residual" means "remainder" or "that which is left", and as the name suggests the chlorine residual test is used to measure the amount of chlorine remaining in the water at the time the test is made.

The chlorine residual is usually tested in finished water which is ready to be released into the distribution system, although operators must also ensure that there is adequate residual at the extreme ends of the distribution system.

Although the pros and cons of disinfection with chlorine have been extensively debated, it remains the most widely used chemical for disinfection of water.

Excess Chlorination may produce adverse effects. Potentially carcinogenic chloroorganic compounds such as chloroform may be formed.

To fulfill the primary purpose of chlorination and to minimize any adverse effects, it is essential that proper testing procedures be used.

Several methods for measurement of total residual chlorine are available including iodometric methods, amperometric titration methods, and N,N-diethyl-p-phenylenediamine (DPD) methods.

In this module we are going to learn Iodometric method of residual chlorine determination.

11.2.1 ENVIRONMENTAL SIGNIFICANCE

Chlorine residuals determination is used to control chlorination of domestic and industrial wastewaters.

Active chlorine (free and combined) should be determined at each stage in the treatment process of drinking water and in the water mains in order to guarantee bacteriologically impeccable water.

Chlorine determination is important to avoid bad odour and change in the taste of water.

It is determined in the swimming pools to avoid ill effects due to excess chlorination.

Determination of chlorine residual in water distribution is useful to find the source of contamination or leakage points, so as to supply wholesome water to the consumer.

Thus, the main purpose for the chlorination of water supplies and polluted waters serves primarily to destroy or deactivate disease-producing micro-organisms.

11.3 PRINCIPLE

The starch-iodide titration method, one of the oldest methods for determining chlorine, is very non-specific for oxidants and generally is used for total chlorine testing at levels above 1 mg/L Cl_2 .

Chlorine will liberate free iodine from potassium iodide (KI) solutions at pH 8 or less. The liberated iodine is titrated with a standard solution of sodium thiosulphate ($\text{Na}_2\text{S}_2\text{O}_3$) with starch as the indicator.

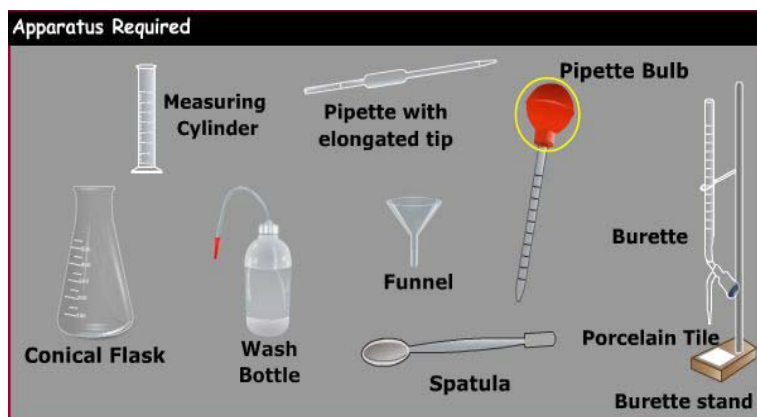
This method is based on reaction with thiosulfate solution

The end point of the titration is indicated by the disappearance of the blue-colored, starch-iodide complex.

11.4 MATERIALS REQUIRED

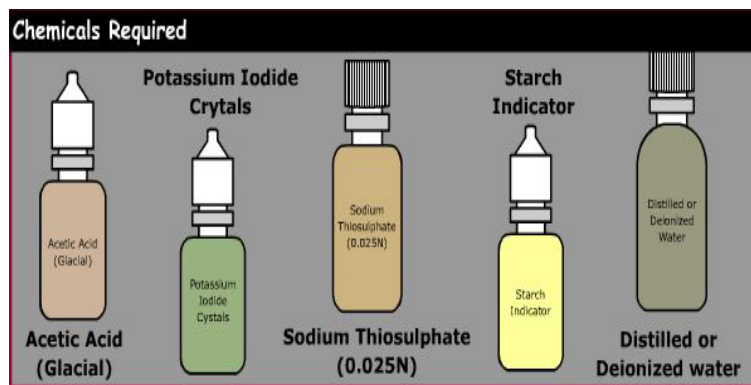
11.4.1 APPARATUS REQUIRED

1. Burette & Burette Stand
2. Porcelain Tile
3. Pipettes with elongated tips
4. Pipette Bulb
5. Wash Bottle
6. 250 mL Graduated Cylinder
7. 500 mL Conical Flask (Erlenmeyer flask)

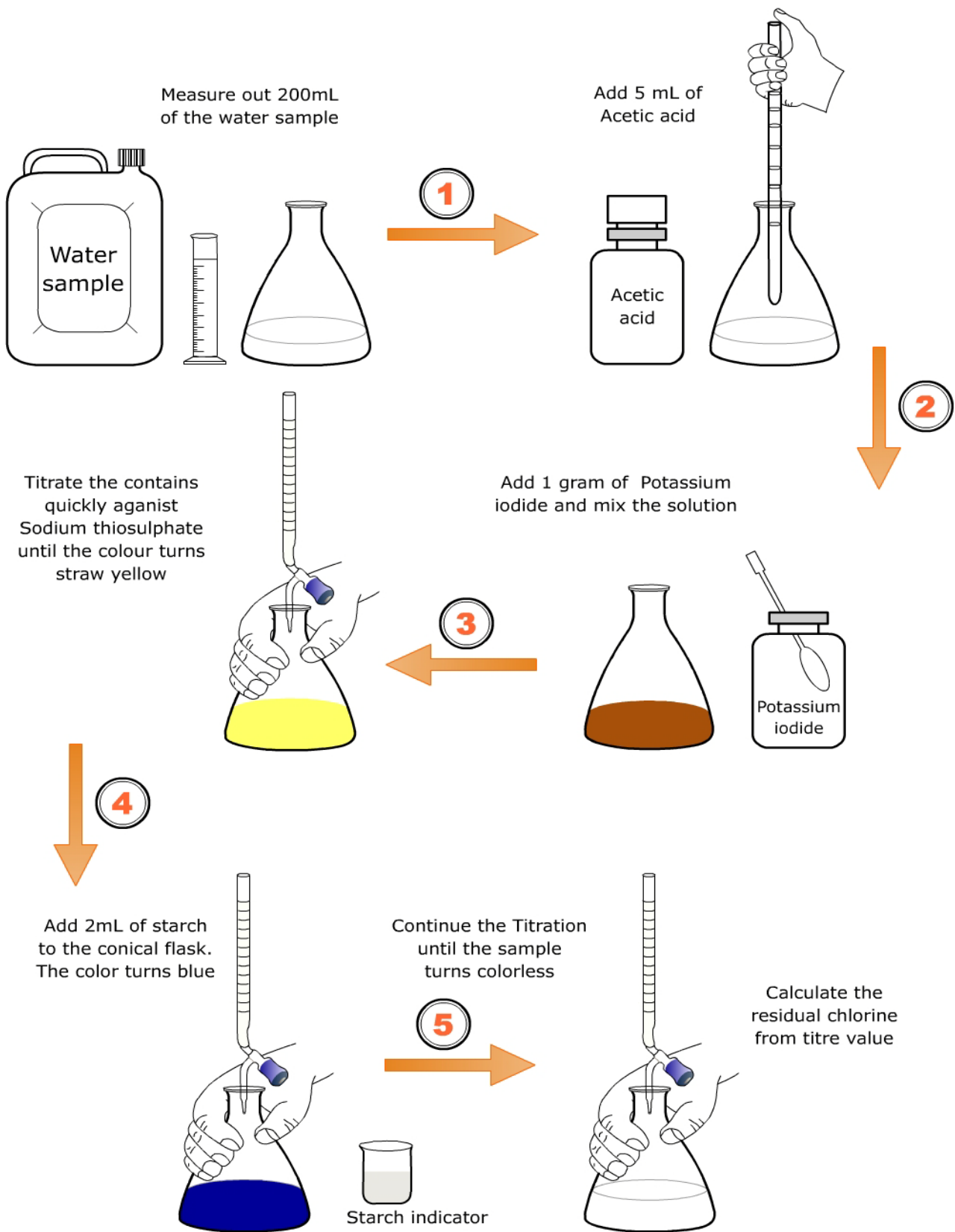


11.4.2 CHEMICALS REQUIRED

1. Acetic Acid, Conc. (glacial)
2. Potassium Iodide, KI, crystals
3. Sodium thiosulphate
4. Starch indicator
5. Distilled or Deionized Water



PROCEDURE CHART



11.5 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.

11.5.1 PRECAUTIONS

The following precautions should be observed while performing the experiment:

- This experiment is a basic experiment and hence there will not be any major difficulties in performing the experiment. The entire procedure should be done in quick time without exposing the solutions to the ambient air.
- Do not expose the potassium iodide crystals in the air. If possible do the experiment in iodine flask instead of conical flask.
- Chlorine in water solutions is not stable. As a result, its concentration in samples decreases rapidly.
- Samples to be analyzed for chlorine cannot be stored or preserved. Tests must be started immediately after sampling. Therefore, samples taken for the chlorine residual test must be grab samples only and excessive agitation must be avoided.
- Exposure to sunlight or other strong light, air, or agitation will further reduce the quantity of chlorine present in solutions.

11.6 PROCEDURE

There are two different methods available to estimate the residual chlorine as per our IS Code.

- 1) **Iodometric Method:** This method is more precise than colorimetric method where residual concentration exceeds 1mg/L, but for lower concentration it is not so accurate.
- 2) **Stabilized Neutral Ortho-Toluidine method:** This method is useful to determine free available chlorine and combined chlorine. This method is sensitive to low residual chlorine concentrations.

11.6.1 PREPARATION OF REAGENTS

Sodium Thiosulphate solution (0.01N)

Weigh approximately 2.482 g of sodium thiosulphate ($\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$).

Transfer to the beaker and dissolve it in boiled distilled water.

Transfer it to the standard flask and make it up to 1000 mL.

11.6.2 TESTING OF SAMPLE

- Rinse the burette with sodium thiosulphate and then fill the burette with sodium thiosulphate.
- Fix the burette to the stand.
- Take 200 mL of a given sample in a conical flask.
- Add 5 mL Acetic acid. To acidify the sample. It is used to reduce the pH between 3 and 4 in the conical flask.
- Add about 1 g Potassium Iodide (KI) measured using the spatula and dissolve it by thoroughly mixing it with stirring rod.
- Perform the titration quickly, since iodine liberate faster.
- Titrate the solution with standard $\text{Na}_2\text{S}_2\text{O}_3$ solution until the yellow color of liberated Iodine is almost faded out. (Pale yellow color)
- Add 1 mL of starch solution and continue the titration until the blue color disappears.
- In many cases residual chlorine is very low and starch needs to be added before starting up the titration.
- Note down the burette reading (to know the volume of sodium thiosulphate added).
- The reading is 10.6 mL

11.7 CALCULATION

To determine Residual Chlorine in the given water sample, the readings are required to be tabulated.

11.7.1 TABLE

Sample No.	Temperature of Sample (°C)	Volume of Sample (mL)	Burette Reading (mL)		Volume of Titrant (mL) (Na ₂ S ₂ O ₃ solution used)	Residual Chlorine (mg/L)
			Initial	Final		

Burette Solution: Sodium Thiosulphate

Pipette Solution: Sample

Indicator: Starch

End point : Disappearance of blue color

- The volume of water sample taken is 200 mL. Sodium Thiosulphate is taken in the burette.
- For the First titration the initial reading is 0 mL and the final reading is 1.4 mL
- The volume of sodium thiosulphate consumed to get the end point is 1.4 mL
- For Second titration the initial reading is 0 mL and the final reading is 1.3 mL
- The volume of sodium thiosulphate consumed to get the end point is 1.3 mL
- For Third titration the initial reading is 0mL and the final reading is 1.3 mL
- The volume of sodium thiosulphate consumed to get the end point is 1.3 mL
- For second and third titration the burette reading is same so, we have achieved concordant values. we can go for the calculations.

11.7.2 DATA SHEET

DETERMINATION OF RESIDUAL CHLORINE 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 S Long 80'14" 8.82)
Sample Description : Surface water

TABULATION

Sample No	Volume of Sample (mL)	Burette Reading (mL)		Volume of Titrant (mL) (Na ₂ S ₂ O ₃ solution used)
		Initial	Final	
1.	200	0	1.4	1.4
2.	200	0	1.3	1.3
3.	200	0	1.3	1.3

Specimen Calculation:

Volume of Sodium thiosulphate V_1 = 1.3 mL

Normality of Sodium thiosulphate N_1 = 0.01 N

Volume of Sample V_2 = 200.0 mL

Equivalent weight of Chlorine = 35.45

$$\text{Residual Chlorine} = \frac{\text{Volume of Sodium thiosulphate} \times N \times 35.45 \times 1000}{\text{Volume of sample taken}}$$

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

$$\text{Residual Chlorine (mg/L)} = 1.3 \times 0.01 \times 35.45 \times 1000 / 200$$

$$= 2.3 \text{ mg/L}$$

11.8 INTERPRETATION OF RESULTS

The amount of residual chlorine in the given sample of water = **2.3 mg/L**

11.9 INFERENCE

Sample provided for the experiment is obtained from over head tank supplied by the government agency. Residual chlorine is the water measured to be 2.3 mg/L. It is more than permissible amount. Active chlorine should be present at each stage of water treatment and distribution. The residual chlorine at the consumers end should be 0.2 mg/l. Presence of excessive chlorine gives bad odour and taste and is harmful also. It may lead to cancer, skin and eye irritation. To avoid the excess chlorination, water sample need to be boiled before the domestic use.

11.10 EVALUATION

1. Disinfection means

- a) Killing of disease producing bacteria and other microorganisms
- b) Killing of all bacteria and other microorganisms
- c) Removing infection from water
- d) Removing sewage from water

2. Bleaching powder is mixed in water for

- a) Making it clean
- b) Disinfection of water
- c) Adjusting its pH
- d) Making it soft water

3. Residual chlorine means the Chlorine

- a) required for the disinfection of water normally
- b) required for the disinfection of water in the rainy season
- c) available after completion of the disinfection
- d) required as the superchlorination

4. Potable water is

- a) tasty water
- b) wholesome water
- c) Mineral water
- d) water free from disease producing elements and bacteria.

5. The amount of residual chlorine in water should be
- a) 0.2 mg/litre
 - b) 2.0 mg/litre
 - c) 2.5 mg/litre
 - d) 4.0 mg/litre
6. Residual chlorine is detected in water by _____.
- a) Erichrome black T
 - b) Bleaching powder
 - c) Methyl orange
 - d) Orthotolidine
7. Chlorine is often added to wastewater for disinfection before effluent discharge
A potential problem with this procedure is
- a) Toxic chlorinated hydrocarbons may be formed
 - b) Chlorine contributes to depletion of the ozone layer
 - c) Chlorine gas is poisonous and may threaten nearby homes
 - d) chlorine is a nonrenewable resource and may soon be depleted
8. Water requires _____ mg/L of chlorine to destroy the microorganisms present.
- a) 2
 - b) 3
 - c) 4
 - d) 1
9. Chlorination is effective if the pH is _____.
- a) 7.2 to 8.5
 - b) 6.8 to 7.2
 - c) 6 to 7
 - d) Neutral
10. Titration should be carried out with
- a) Neutral pH
 - b) pH 10 - 11
 - c) pH 3 - 4
 - d) No criteria

KEY TO ITEMS:

1) a

2) b

3) c

4) d

5) a

6) d

7) a

8) a

9) b

10) c