This document is an instructional module package prepared in objective form for use by an instructor familiar with the laboratory procedures for determining the combined chlorine residual of a wastewater sample. Included are objectives, instructor guides, student handouts, and transparency masters. This module considers the amperometric, DPD, electrode and color comparator kit methods.

(Author/RH)
CHLORINE ANALYSIS - WASTEWATER

Training Module 5.125.2.77

Prepared for the

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by

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September, 1977
The mention of trade names, or use of manufacturers technical bulletins, diagrams depicting specific equipment, or the commercial product in this module is for illustration purposes, and does not constitute endorsement or recommendation for use by Kirkwood Community College nor by the Iowa Department of Environmental Quality.
Module Title: Chlorine in Wastewater

Topics: Introduction, Amperometric Method, DPD Method, Electrode Method, Comparator kit Methods, Summary

Objectives:
Upon completion of this module the participant should be able to:
- Determine the residual chlorine level in wastewater.

Instructional Aids:
Handouts

Instructional Approach:
Lecture, Lab, Discussion

References:
- Standard Methods
- EPA Effluent Monitoring Procedure
- AWWA - Water Chlorination Principles and Practices
- Manuals for Amperometric Titrators

Class Assignments:
Instructional Aids

Handouts

Handouts may be copied directly.

Lab supplies and apparatus

Supplies and apparatus should be supplied per handouts so that participants may work in groups of 2 or 3.
Module No: Residual Chlorine in Wastewater

Submodule Title: 

Topic: Introduction

Objectives:

When the participant completes this topic they should be able to:

1. Differentiate between free and residual chlorine.
2. List four methods for total residual chlorine analysis.
3. Indicate and demonstrate proper sampling procedures.
4. Explain why a sample cannot be preserved.

Instructional Aids:

None

Instructional Approach:

Lecture

References:

Standard Methods
AWWA - Water Chlorination Principles and Practices

Class Assignments:
### Instructor Notes:

### Instructor Outline:

1. **a.** Differentiate between free and residual chlorine.
   
   **b.** Indicate when each is tested.

2. **List and describe methods for chlorine analysis.**
   
   **a.** Kits
   
   **b.** Amperometric
   
   **c.** DPD Spectrophotometric
   
   **d.** Orthotolidine
   
   **e.** Titrmetric

3. **Discuss sampling for chlorine.

4. **Explain why a sample cannot be preserved.**
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**Objectives:**

When the participant completes this topic they should be able to:

1. Identify the proper apparatus and reagents needed for the amperometric method for free and residual chlorine analysis.
2. Conduct a free and a residual chlorine test using the amperometric method given proper test equipment and reagents.
3. Translate the raw results of the tests into proper units of expression.

**Instructional Aids:**

- Handout
- Lab supplies per handout

**Instructional Approach:**

- Lecture
- Lab

**References:**

1. Manuals for Amperometric Titrators
2. Standard Methods, 14th Ed.
3. EPA effluent Monitoring Procedures
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Effluent Monitoring Procedure: Amperometric Determination of Total Residual Chlorine in Wastewater

1. Analysis Objectives
2. Brief Description of Analysis*

1. The operator will be able to perform an amperometric titration for the determination of total residual chlorine in a sample of wastewater treatment plant effluent.

2. Residual chlorine present in wastewater is in the form of combined chlorine. A "Back-Titration" procedure is used to determine the phenylarsene oxide excess and a formula used to calculate the concentration of total residual chlorine in the sample.

General Description of Equipment used in the Process

A. Capital Equipment

1. Amperometric Titrator Assembly - Wallace and Tiernan*

B. Reusable

1. 1 pipette (1 ml capacity)
2. 1 pipette (5 ml capacity)
3. 1 sample cup (to contain 200 ml)
4. 1 plastic squeeze bottle

* Mention of a specific brand name does not constitute endorsement by the U.S. Environmental Protection Agency.

** Consumable reagents listed are available from Wallace & Tiernan Industrial Products Division, 25 Main St., Belleville, JH 07109.
C. Consumables

1. 1 bottle phenylarsene oxide solution 0.00564 N (16 ounce)
2. 1 bottle pH 4 buffer solution (4 ounce)
3. 1 bottle pH 7 buffer solution (4 ounce)
4. 1 bottle potassium iodide solution (4 ounce)
5. 1 bottle sodium chloride electrolyte tablets (8 ounce)
6. Standard iodine solution 0.1 N
7. Standard iodine titrant 0.0282
8. Potassium iodide crystals
9. Iodine crystals, purified
A. Reagents Standard iodine solution. 1.0 N-Iodine standard solution (0.0282N)

1. Dissolve 40.0 grams of potassium iodide (KI) in 50 ml of distilled water. Add 12.7 g. of iodine crystals and stir until solution is complete.

2. Dilute to one liter with distilled water.
   a. Store the solution in a dark bottle

3. Transfer 25 grams of potassium iodide into a one liter volumetric flask
   a. Use a trip balance

4. Add 200 ml of distilled water and swirl to dissolve.
   a. Use a graduate cylinder

5. Add 285 ml of 0.1 N iodine solution and dilute to the mark with distilled water.

B. Determination of total residual chlorine

1. Set up titrator and plug into a source of 115 volt, single phase, 60 cycle A.C. current.

2. Add sample water to the cup. Adjust the level to the line:
   a. The volume of sample is 200 ml.

3. Place the cup on the titrator.
   a. The top edge of the cup should go behind the cup guide post.
   b. The bottom of the cup should rest on the support post.

4. Turn the switch to start the agitator.

5. Add 5 ml of phenylarsene oxide solution to the sample and mix.
   a. Use a 5 ml pipette.

6. Add 4.0 ml of pH 4.0 buffer solution to the sample and mix.

7. Add 1.0 ml of KI solution.
8. Rotate the adjusting knob so that the microammeter pointer reads about 20 on the scale.

9. Add 0.0282 N iodine solution in small increments.
   a. Use a 1 ml volumetric pipette graduated in 0.1 ml.
   b. The standard reagent bottle, pump, pipette, and applicator tubing cannot be used for this purpose since the plastic components may react with the iodine solution and change its strength.
   c. As iodine is added to the sample, the pointer remains practically stationary until the end-point. Each increment of iodine solution causes a temporary deflection of the microammeter to the right, but the pointer drops back to about its original position. The true end-point is reached with a small addition of iodine solution giving a definite and permanent pointer deflection to the right (up-scale).

10. Note the volume of iodine solution used to reach the end-point.
    a. Calculate the total residual chlorine as follows:

       \[
       \text{mg/l chlorine} = \frac{\text{total volume of iodine}}{\text{phensylarsene oxide used in titration (step 5)}}
       \]

    b. Example of calculation:
       1. Total phenylarsene oxide used in step 5 = 5.0 ml.
       2. ml of iodine required to reach the end-point in step 9 = 0.6 ml.

       \[
       \text{mg/l chlorine} = \frac{5.0 - (5.0 - 0.6)}{5.0} = \frac{5.0 - 3.0}{2.0} = 2.0
       \]
c. The accuracy of the above procedure depends on the volume of the sample (step 2), the strength of the phenylarsene oxide solution (0.00564N) which is quite stable, and the strength of the iodine solution (0.0282 N) which is subject to deterioration with time. If the iodine is not 0.0282 N, it must be standardized by the following procedure.

C. Standardization of iodine solution

1. Add 5.0 ml of phenylarsene oxide solution to 195 ml of dechlorinated water.
   a. Chlorine-demand-free water: Add sufficient chlorine to distilled water to destroy the ammonia. The amount of chlorine required will be about ten times the amount of ammonia nitrogen present; in no case produce an initial residual of less than 1.0 mg/l free chlorine. Allow the chlorinated water to stand overnight or longer; then expose to direct sunlight until all residual chlorine is discharged. Use distilled water free from ammonia and nitrite to produce the chlorine demand free water. Check chlorine residual by amperometric titration.

2. Titrate with the iodine solution.
   a. The end point is reached when a small addition of iodine gives a pointer deflection to the right (up scale) which holds for 15 to 20 seconds. If 1.0 ml of iodine solution neutralizes the 5.0 ml of phenylarsene oxide solution, the iodine solution is 0.0282 N. If the iodine solution has deteriorated, the volume of iodine solution to reach the end-point (something greater than 1.0 ml) is equal to 5 ml of phenylarsene oxide solution.
b. "Back titration" for residual chlorine may be made with weaker than 0.0282 N iodine solutions. The attached chart can be used to determine the excess phenylarsene oxide by following step 12 and subsequent steps.

12. Locate on line "A" the ml of iodine equal to 5.0 ml of iodine equal to 5.0 ml of phenylarsene oxide.

13. Locate on line "C" the volume of iodine as determined in step 9.

14. Determine where a line connecting these points crosses line "B".
   a. This is the excess phenylarsene oxide.
   b. As expressed in the formula, the mg/l of chlorine residual is the excess phenylarsene oxide subtracted from the total.
   c. Example of calculation:
      1. Total phenylarsene oxide = 10.0 ml
      2. ml iodine equal to 5.0 ml phenylarsene oxide = 1.2 ml
      3. ml iodine to reach end point of "back titration" = 0.4
      4. Excess phenylarsene oxide (from chart) = 1.6 (approx.)
      5. mg/l chlorine residual = 10 - 1.6 = 8.4
EXAMPLE

Total phenylarsene oxide = 10 ml.
ml. iodine equal to 5 ml. phenylarsene oxide = 1.2 ml.
ml. iodine to reach end point of "Back Titration" = 0.4
Excess phenylarsene oxide (from chart) = 1.8 (approximately).
ppm chlorine residual = 10 - 1.6 = 8.4
Module No:

Module Title: Chlorine in Wastewater

Submodule Title:

Approx. Time:

Topic: DPD Spectrophotometric Method

Objectives:

When the participant completes this topic they should be able to:

1. Identify the proper apparatus and reagents needed for the DPD.
2. Conduct a free and residual chlorine test using the DPD spectrophotometric method.
3. Translate the raw results of the method into proper units of expression.

Instructional Aids:

Handout
Lab supplies per handout

Instructional Approach:

Lecture
Lab

References:

1. Standard Methods, 14th Ed.
2. Manual for Sanitary Chemistry and Sanitary Microbiology
3. Linn-Benton Community College, Albany, Oregon

Class Assignments:
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| Handout Page 17 - 22 | 1. List and identify apparatus and reagents needed for the method.  
2. Demonstrate the residual chlorine test.  
   Have participant conduct test.  
3. Demonstrate calculation procedure.  
   Have participants do calculations. |
TOTAL CHLORINE RESIDUAL
DPD/SPECTROPHOTOMETRIC METHOD

Equipment

Bausch & Lomb Spectronic 20 or
Bausch & Lomb Mini Spectronic 20
Graduated cylinder, 25 ml, 100 ml
Clippers, for opening powder pillows
Pipette, volumetric, 50 ml
Pipettes, 1 ml, 10 ml
One liter volumetric flask

Reagents

DPD total chlorine reagent powder pillows or.
Buffer and DPD Solution

1. Buffer
Anhydrous potassium dihydrogen phosphate
Anhydrous disodium hydrogen phosphate
Distilled water
Disodium ethylenediamine tetraacetate dihydrate also called
(Ethylenedinitrilo)tetraacetic acid sodium salt mercuric chloride

2. DPD Solution (N, N-diethyl-p-phenylenediamine) DPD Oxalate or
p-amino-N,N diethylaniline sulfate
Sulfuric acid
Distilled water
Potassium iodide
Reagent Preparation

1. **PHOSPHATE BUFFER SOLUTION**
   
   Dissolve 24 g anhydrous disodium hydrogen phosphate, Na$_2$PO$_4$, and 46 g anhydrous potassium dihydrogen phosphate, KH$_2$PO$_4$, in distilled water. Combine this solution with 100 mls distilled water in which 0.8 g disodium ethylenediamine tetraacetate dihydrate, also called (ethylenedinitrilo) tetraacetic acid sodium salt, have been dissolved. Dilute to 1 liter with distilled water and add 0.02 g mercuric chloride to prevent mold growth and to prevent interference in the test by iodide ions in the reagents.

2. **N, N-DIETHYL-P-PHENYLENEDIAMINE (DPD)**
   
   Dissolve 1 g DPD oxalate or 1.5 g p-aminophenol-N,N diethylaniline sulfate in chlorine-free distilled water containing 8 ml of 1+3 (25%) sulfuric acid and 0.2 g disodium ethylenediamine tetraacetate dihydrate also called (ethylenedinitrilo) tetraacetic acid sodium salt. Make up to one liter, store in a brown glass-stoppered bottle. Discard when this solution becomes discolored.

3. **POTASSIUM IODIDE**
   
   Use as dry crystals.

Sample Preparation

1. Take a water sample by filling a clean 100 ml graduated cylinder to the 100 ml mark with sample solution.

2. Add the contents of one DPD Total Chlorine Reagent Powder Pillow (0.5 g DPD Powder) and mix. A red color will develop if chlorine is present. Wait at least 3 minutes but not over 6 minutes before taking reading.
Place 5 ml's each of buffer reagent and DPD indicator solution in the graduated cylinder and mix. Add 100 ml's sample and mix. Add 1 g of potassium iodide crystals and mix to dissolve. Allow to stand at least 3 minutes but not over 6 minutes for color development.

**Procedure**

1. Bausch & Lomb Spectronic 20
   - a. Adjust the wavelength to 530 nm.
   - b. Cover the empty sample compartment and adjust the zero control for a reading of exactly 0% transmittance.
   - c. Place a test tube containing a portion of the untreated sample into the sample compartment and adjust the Full Scale Control for a reading of exactly 100% transmittance.
   - d. Place a test tube containing the treated sample into the sample compartment and read transmittance. Refer to the table of available chlorine in mg/l vs. % transmittance shown under Free Chlorine Analysis.

2. Bausch & Lomb Mini Spectronic 20
   - a. Adjust the wavelength to 530 nm.
   - b. Using the one half inch adapter and insert, place the opaque rod in the sample compartment and zero the instrument.
   - c. Place a 25-nm cell containing a portion of the untreated sample into the sample compartment and adjust the Full Scale Control for a reading of 100% transmittance.
   - d. Place a 25-nm cell containing the treated sample into the sample compartment and read % transmittance. Refer to the table of available
chlorine in mg/l vs. % transmittance shown under Free Chlorine Analysis.

NOTES:

1. If the sample turns yellow when adding the DPD reagent or measures less than 10% transmittance, the chlorine concentration is too high and a sample dilution is necessary. A slight loss of chlorine may occur due to dilution.

2. If the sample contains more than 2000 mg/l alkalinity or acidity as CaCO₃, the sample may not develop the full amount of color or it may fade instantly. To overcome this interference, pretreat the sample to about 2000 mg/l alkalinity or acidity or establish a pH range between 5 and 8. Use an appropriate amount of acid or base that does not contain ammonium or chloride ions. Test the treated sample immediately.
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<tr>
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Table taken from Page 2-29, Procedures, Chemical Lists and Glassware for Water and Wastewater Analysis
Module No: 1
Module Title: Chlorine in Wastewater
Submodule Title: 

Approx. Time: 
Topic: Chlorine Electrode Method

Objectives:
When the participant completes this topic they should be able to:

1. Identify the proper apparatus and reagents needed for the residual chlorine electrode method.
2. Conduct a chlorine residual test using the residual chlorine electrode.
3. Translate the raw results of the method into proper units of expression.

Instructional Aids:
Handout

Instructional Approach:
Lecture
Lab

References:
Methods Manual for Chlorine Electrode

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<td>2. Demonstrate the residual chlorine test. Have participant conduct test.</td>
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RESIDUAL CHLORINE ELECTRODE

Procedure for Samples Between 0.2 and 20 ppm

Prepare 1 ppm standardizing solution:

Note: Always mix the three reagents and allow two minutes for reaction before diluting to 200 ml with distilled water. The reaction between iodate and iodide in an acid solution is extremely slow after dilution.

1. Pipet 1 ml residual chlorine standard (100 ppm as Cl₂), 1 ml iodide reagent, and 1 ml acid reagent into a 150 ml beaker. Use only 1 ml of each solution, and do not add water. Swirl for two minutes to allow complete reaction.

2. Add 99 ml distilled water and mix thoroughly.

3. Store this 1 ppm standardizing solution in a 4-oz., amber glass, wide mouth packer bottle for calibration. Prepare a fresh solution every day.

Measurement Using 701 or 801 A digital pH/mv meter

1. Scale 4-cycle semilogarithmic graph paper with 000.0 mv at the center of the linear axis and 1 ppm at the center of the logarithmic axis. The linear scale should cover a range from -60 mv to 60 mv.

2. Plot one point at 000.0 mv and 1 ppm. Plot a second point at 29 mv and 10 ppm. Draw a straight line connecting the two points and extrapolate it to 0.01 ppm.

This calibration curve can be used for all measurements between 0.05 and 10 ppm Cl₂. There is no need to prepare a new curve each time the electrode is calibrated. The portion of the curve below 0.2 ppm is used for low-level measurements.
3. Place the electrode in the 1 ppm standardizing solution so that the reference element is submerged.
4. Turn the function switch of the meter to a relative millivolt mode. Set the meter reading to 000.0 by turning the calibration control knob on the 701 or by pressing the restandardization button on the 801A.
5. Remove the electrode from the solution. Blot dry. Replace the cap on the storage bottle.
6. Pipet 1 ml iodide reagent into a 150 ml beaker and add 1 ml (20 drops) acid reagent. Add 100 ml sample.
7. Mix thoroughly and let stand for about two minutes to allow complete reaction.
8. Place the electrode in the sample so that the reference element is submerged. Record the potential reading. Determine the total residual chlorine level in the sample from the calibration curve.
9. Recalibrate every two hours by repeating steps 3 through 5 above.
### Module

**Module No:**  
**Module Title:** Chlorine in Wastewater  
**Submodule Title:**

### Approx. Time:

### Topic:
Use of Comparator or Kit Methods

### Objectives:

When the participant completes this topic they should be able to:

1. Indicate why the kit methods are less accurate than standard methods.
2. Conduct a residual chlorine and free chlorine test using kit methods and compare the results to a standard method.

### Instructional Aids:

- Handout
- Lab supplies per handout

### Instructional Approach:

- Lecture
- Lab

### References:

1. Standard Methods
2. Kit Instructions

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CHLORINE RESIDUAL
(Comparator)

Introduction

The amount of chlorine remaining in wastewater samples, following chlorination varies rapidly with time since chlorine is unstable in water. Therefore, samples to be analyzed for chlorine residual values cannot be stored and tests must be started immediately after grab sampling. Avoid exposing the samples to excessive light and agitation.

Equipment

Color comparators
a. La Motte Model #P-38 VW & R #66151-007
b. Wallace-Tiernan Model #U-2374
c. Hach DPD Free & Total Chlorine Test Kit, Model #CN-66

Reagents

1. ORTHOTOLIDINE
   No longer available from chemical supply houses.
2. DPD FREE CHLORINE REAGENT POWDER PILLOWS  HACH #14070-99
3. DPD TOTAL CHLORINE REAGENT POWDER PILLOWS  HACH #14064-99

Procedure

(For Wallace-Tiernan Model)

1. CLEAN COMPARATOR GLASS CELLS
   Use hot soapy water and a soft test tube brush. Rinse thoroughly with final distilled water rinse. Let cells drain dry.

2. PLACE UNTREATED SAMPLE IN GLASS CELL
   Designated as the "blank" cell and place in right hand slot of comparator.
3. PLACE ORTHOTOLIDINE REAGENT IN GLASS CELL DESIGNATED "OT"

   Use 0.5 ml reagent for each 10 mls of sample.

4. ADD MEASURED VOLUME OF SAMPLE

   The volume is usually 10 or 15 mls.

5. MIX REAGENT AND SAMPLE

   Careful use of clean stirring rod is recommended.

6. WAIT FIVE MINUTES

7. PLACE GLASS CELL IN LEFT HAND SLOT OF THE COMPARATOR

   Rotate the standard color disc until the color of the standard, as
   seen through the untreated sample, most closely matches the color
   of the treated sample.

8. READ THE CHLORINE VALUE FROM THE STANDARD COLOR WHEEL

9. EXPRESS THE RESIDUAL VALUE AS:

   Total residual chlorine (mg/l)

Procedure (For LaMotte Model)

1. CLEAN COMPARATOR/GLASS CELL, AS ABOVE

2. ADD SAMPLE TO MARK ON GLASS CELL

3. ADD 8 DROPS ORTHOTOLIDINE REAGENT TO GLASS CELL

4. MIX REAGENT AND SAMPLE, AS ABOVE

5. WAIT FIVE MINUTES

6. PLACE GLASS CELL IN COMPARATOR

   Put in the slot closest to the comparator window whose color most
   nearly matches that of the sample.

7. DETERMINE WHICH WINDOW MATCHES THE COLOR OF THE SAMPLE

8. READ THE CHLORINE RESIDUAL VALUE NEXT TO THE WINDOW
9. **EXPRESS THE CHLORINE RESIDUAL VALUE AS:**

   Total residual chlorine (mg/l)

**NOTE:** These procedures measure total residual chlorine. If free residual chlorine is desired, read immediately after the addition of orthotolidine without the five minute wait.

If combined residual chlorine is desired subtract the free value from the total value.

**Hach Test Kit Total Chlorine Residual**

1. **COLLECT SAMPLE**
2. **CLEAN GLASS CELL AS ABOVE**
3. **PLACE UNCHLORINATED SAMPLE INTO BOTH CELLS**
4. **ADD REAGENTS**
   - Use nail clippers to open powder pillows
5. **IF POSITIVE TEST DPD CANNOT BE USED ON THIS WATER SAMPLE**
6. **IF RESULTS OF THIS REAGENT ADDITION ARE NEGATIVE, RINSE BOTH CELLS AND ADD CHLORINATED SAMPLE TO BOTH CELLS**
7. **ADD DPD TOTAL RESIDUAL CHLORINE REAGENT POWDER PILLOW**
8. **STIR REAGENTS AND SAMPLE**
9. **WAIT UNTIL FULL COLOR DEVELOPS, FIVE MINUTES**
11. **READ AND RECORD IN mg/l TOTAL RESIDUAL CHLORINE**

**NOTE:** If free residual chlorine is desired, carry out the steps described above using free residual chlorine powder pillows in place of total chlorine residual powder pillows. Record as free residual chlorine in mg/l.
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<td>SUMMARY</td>
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**Objectives:**

When the participant completes this topic they should be able to:

1. Compare and contrast methods for residual chlorine and select a method best suited for an individual plant.

**Instructional Aids:**

All handouts

**Instructional Approach:**

Discussion

**References:**

Standard Methods

**Class Assignments:**
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<td>Compare and contrast the methods for chlorine.</td>
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### Module Title:
Chlorine in Wastewater

### Approx. Time:

### Submodule Title:
EVALUATION

### Objectives:

#### Practical

Determining the amount of residual chlorine in a given sample using one of the methods from this module. Reagent apparatus and procedure sheets will be provided.