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ABSTRACT

This document is an instructional module package prepared in objective form for use by an instructor familiar with the procedures for chlorine residual analysis. It includes objectives, an instructor guide, and student handouts. The module addresses the determination of combined and free residual chlorine in water supply samples using three different methods: (1) Amperometric, (2) DPD spectrophotometric, and (3) color comparator kit. (Author/RH)

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CHLORINE ANALYSIS - WATER
Training Module 5.260.2.77

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September, 1977

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Module No:	Module Title: Chlorine in Water
Approx. Time:	Topics: Introduction Amperometric Method DPD Spectrophotometric Method Use of Comparator of Kit Methods Summary
Objectives: Upon completion of this module the participant will be able to: Determine the amount of free and residual chlorine in a water sample.	
Instructional Aids: Handouts	
Instructional Approach: Lecture Lab Discussion	
References: Standard Methods, 14th Ed. EPA Effluent Monitoring Procedures	
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:	Module Title: Chlorine in Water
	Submodule Title:
Approx. Time:	Topic: Introduction
Objectives: When the participant completes this topic they should be able to: <ol style="list-style-type: none">1. Differentiate between free and residual chlorine.2. List four methods for free and 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:	

Module No:	Topic: Introduction
Instructor Notes:	Instructor Outline:
	<ol style="list-style-type: none">1. a. Differentiate between free and residual chlorine. b. Indicate when each is tested.2. List and describe methods for chlorine analysis.<ol style="list-style-type: none">a. Kitsb. Amperometricc. DPD Spectrophotometricd. Orthotolidinee. Titrimetric3. Discuss sampling for chlorine4. Explain why a sample cannot be preserved.

Module No:	Module Title: Chlorine in Water
	Submodule Title:
Approx. Time:	Topic: Amperometric Method
Objectives: When the participant completes this topic they should be able to: <ol style="list-style-type: none">1. Identify the proper apparatus and reagents needed for the amperometric method of residual chlorine analysis.2. Conduct a chlorine residual test by the amperometric method given proper test equipment and reagents.3. Translate the raw results of test into proper units of expression.	
Instructional Aids: Handout Lab supplies per handout.	
Instructional Approach: Lecture Lab	
References: <ol style="list-style-type: none">1. Manuals for Amperometric Titrators2. Standard Methods, 14th Ed.3. EPA effluent Monitoring Procedures	
Class Assignments:	

Module No:	Topic:
Instructor Notes:	Instructor Outline:
Handout Page 9 - 17	<ol style="list-style-type: none">1. List and identify apparatus and reagents needed for the method.2. Demonstrate the free and residual chlorine test. Have participant conduct test.3. Demonstrate calculation procedure. Have participants do calculations.

Effluent Monitoring Procedure: Amperometric Determination of Free and Combined Residual Chlorine in Water

1. Analysis Objectives

2. Brief Description of Analysis*

1. The operator will be able to perform an amperometric titration for the determination of free and combined residual chlorine in water.
2. Free available residual chlorine and combined residual chlorine are titrated successively using an amperometric titrator. The free available residual chlorine is titrated first. The sample pH is then dropped to 4 by adding buffer solution pH4 and then potassium iodide is added to the sample. The first titration will represent the free available residual chlorine while the second titration will represent the combined residual chlorine.

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)

*Standard Methods for the Examination of Water and Wastewater, 13th Ed., 1971. APHA, Washington, D. C., p. 112.

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

4. 1 plastic squeeze bottle

C. Consumable*

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)

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

A. Equipment Preparation

1. Set up titrator on work bench.
 - a. Electric outlet 110 volt required.
 - b. Amperometric titrator assembly available from Wallace and Tiernan Corporation.
2. Select proper pipette for titration
 - a. Two pipettes are furnished with the titrator. The 1 ml pipette is generally used when the residual is less than 1 mg/l. A 5 ml pipette is for use with higher residuals.
3. Lightly grease the lower end and insert it in the top of the pump unit on the side of the titrator.
4. Fill the pump squeeze bottle about 2/3 to 3/4 full with phenylarsene oxide solution.
5. Screw the bottle on to the pump
 - a. It is easier to turn the bottle than the cap.
6. Pour sufficient electrolyte tablets into the cell unit to fill the chamber about 2/3 full.
7. Add enough distilled water to cover the tablets.
 - a. Use a plastic squeeze bottle.
8. Plug the cell unit into the titrator.
 - a. The cell is so designed that it cannot be plugged in except in the correct position.
9. Examine the titrator cup. The cup has a line indicating the 200 ml level.
 - a. Whenever the term "sample" is used in these instructions it shall mean a 200 ml volume of the water to be tested.

B. Determination of Free Available Residual Chlorine

1. Plug the electric power plug into a source of 115 volt, single phase, 60 cycle A.C. current.
2. Fill the pipette with phenylarsene oxide solution.
 - a. Alternately squeeze and release the squeeze bottle.
3. Remove all air from the pipette and plastic tubing by rotating the red knob in the stem unit 1/4 turn counter-clockwise.
 - a. The pipette should drain through the plastic tubing.
4. Catch the discarded solution in a 50 ml beaker.
5. Refill the pipette to the top (zero) calibration mark.
6. Add sample water to the cup. Adjust the level to the line.
 - a. The volume of sample is 200 ml.
7. 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.
 - c. The plastic tubing from the pump should be submerged in the sample about 1/16 inch. If necessary, adjust the tubing on the guide post to obtain this condition.
8. Add 1 ml of buffer solution pH 7 to the water sample.
 - a. The droppers furnished with the titrator are 1 ml units. A dropper full of solution should be used wherever 1 ml of solution is called for.
 - b. The pH of the sample is between 6.0 and 7.5 it is not necessary to add buffer.

9. Start the agitator by turning the switch to "ON".
10. Adjust the meter to make the pointer read maximum on the scale.
 - a. Rotating the adjusting knob clockwise should increase the reading.
 - b. If the pointer is above maximum when the adjusting knob is rotated completely counter-clockwise, then the titration should be started with the knob in this position.
11. Start adding small amounts of titrant and note the deflection of the meter scale after each addition.
 - a. If free available chlorine is present in the sample and if the pointer is on scale at the beginning of the titration, then the first addition of titrant should cause a definite pointer movement to the left. If the pointer goes below zero then it should be brought back on scale by rotating the adjusting knob clockwise.
12. Continue the addition of small amounts of titrant until the addition of titrant no longer causes a deflection of the needle.
 - a. In most waters the end-point of the reaction is just passed when the addition of a small amount of titrant no longer deflects the pointer to the left.
 - b. The amount of titrant used in the titration is then read from the pipette and the last increment is subtracted from the pipette reading and the resultant figure represents the free available residual chlorine in mg/l.
13. Subtract the last reading from the previous reading.
14. The reading on the pipette represents the amount of free available chlorine in mg/l.

15. Turn instrument "OFF".

16. Record your result.

C. Determination of combined residual chlorine

1. Repeat steps 1 through 7 of the free available chlorine procedure if the free available chlorine determination has not been performed.

a. The general procedure for measuring total residual chlorine is the same as that given for measuring free available residual chlorine.

2. If you have just completed the free chlorine determination, you can continue the use of the same sample for this determination.

3. Add 1 ml of buffer solution pH 4 to the sample.

a. Use the dropper to add the buffer solution.

4. Add 1 ml of potassium iodide solution to the water sample.

a. Use the dropper to add the potassium iodide solution.

b. When potassium iodide is added, the pointer may first deflect to the left and then go up-scale.

5. Follow steps 9 through 16 if the previous procedure for the determination of free available chlorine. In this case the result is reported as combined residual chlorine.

a. Free available residual chlorine and combined residual chlorine may be measured in one sample by combining the two procedures.

b. The free available chlorine is measured first. The sample pH is then dropped to 4 by adding buffer solution pH 4 and then potassium iodide.

- c. If combined residual chlorine is present, the pointer will deflect to the right when potassium iodide is added.
- d. The first titration will represent the free available residual chlorine while the second titration will represent the combined residual chlorine.

Notes:

The fundamental chemical procedure involved in the amperometric titrator is the neutralization of an oxidizing agent (free available chlorine) in a sample of water by the addition of a reducing agent of known strength. Immersed in the sample cell unit which produces a small direct current which is proportional to the free chlorine present on a microammeter which is connected to the cell current decreases, and the microammeter pointer moves down scale. The end point of the reaction occurs when enough reducing agent has been added to just neutralize all of the free chlorine in the sample. When this point is reached, the further addition of a small amount of reducing agent no longer deflects the pointer to the left. On the titrator, the sample volume and the strength of the reducing agent have been selected to make 1 milliliter of reducing agent equivalent to one milligram per liter of chlorine. When the endpoint is reached, therefore, the volume of reducing agent used represents the chlorine concentration in mg/l.

Under the conditions specified in the titration procedure, the titration can be used to distinguish between free available residual chlorine and combined residual chlorine because the reducing agent employed reacts readily with free chlorine but does not react with combined chlorine. If either combined or total residual chlorine is to be measured, potassium iodide

is added to the sample to produce an amount of free iodine which is equivalent to the original residual chlorine. The reducing agent reacts readily with free iodine so that the titration can be carried out in a manner similar to that used for free available residual chlorine determination.

The electrolyte used in the inner chamber of the cell has a tendency to crystallize out on the contact springs and in the terminals of the cell unit. This may slightly corrode the electrical contacts between the various units. Improper electrical connections cause erratic microammeter pointer readings during the titration. Should any crystals accumulate on the plastic cell unit, these parts should be washed off with warm water. CAUTION: Never use water warmer than 100° F, as hot water softens the plastic. When the titrator is not to be used for extended periods, the cell unit should be washed out to remove all electrolyte tables and solution, and stored dry.

If free available residual chlorine determinations are to be made after potassium iodide has been used in preceding titrations, the cell unit should be rinsed off in several sample cups of water to remove traces of potassium iodide solution and buffer solution pH 4.

Occasionally, when potassium iodide is added to the sample, the pointer will drop to the left and will not come back on scale even though the potentiometer is turned completely clockwise. Under these conditions, the cell unit is said to have lost its sensitivity to iodine. This situation is likely to arise if the titrator has been used to determine free chlorine only for extended periods of time, i.e. the cell unit has not been exposed to iodine for prolonged periods.

The sensitivity of the cell unit can be restored by adding enough free iodine to the distilled water in the sample jar to create a yellowish color. The free iodine may be in the form of tincture of iodine or may be obtained by adding potassium iodide to a strong chlorine solution. Agitate the sample for two or three minutes and then allow the cell unit to stand in the iodine solution for 10 to 15 minutes. After this treatment, the cell unit should be rinsed off thoroughly to remove all traces of iodine.

The main requirement as far as electrolyte tablets are concerned is to have saturated electrolyte solution inside the cell unit at all times. Theoretically, this requirement is not as long as any tablets and water are in the cell unit. The actual water level inside the cell unit cannot be controlled since this level tends to equalize with (or even go below) the water level in the sample jar through the porous wicking.

Module No:	Module Title:
	Submodule Title:
Approx. Time:	Topic:
	DPD Spectrophotometric Method
Objectives: When the participant completes this topic they should be able to: <ol style="list-style-type: none"> 1. Identify the proper apparatus and reagents needed for the DPD Spectrophotometric method of residual chlorine analysis. 2. Conduct a chlorine residual 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: <ol style="list-style-type: none"> 1. Standard Methods, 14 Ed. 2. Manual for Sanitary Chemistry and Sanitary Microbiology 3. Linn-Benton Community College, Albany, Oregon 	
Class Assignments:	

Module No:	Topic:
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Instructor Notes:	Instructor Outline:
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Handout
Page 20 - 30

1. List and identify apparatus and reagents needed for the method.
2. Demonstrate the free and residual chlorine test.
Have participant conduct test.
3. Demonstrate calculation procedure.
Have participants do calculations.

FREE CHLORINE RESIDUAL
DPD/SPECTROPHOTOMETRIC METHOD

Introduction

Drinking water, sewage effluents and swimming pools are treated with chlorine to eliminate possible pathogenic bacteria.

Chlorine can be present in water as free available chlorine or as combined available chlorine. Both forms can exist in the same water and be determined together as the total available chlorine. Free chlorine is present as hypochlorite ion. Combined chlorine exists as mono, di, or trichloramine.

Equipment:

Bausch and Lomb Spectronic 20 or
Bausch and Lomb Mini Spectronic 20
Clippers, for opening powder pillows
Pipette, Volumetric, 50 ml
Pipette, 1 & 10 ml in 1/10 ml graduations
One liter volumetric Flask

Reagents

DPD Free Chlorine Reagent Powder Pillow or
Buffer & 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

1. PHOSPHATE BUFFER

Dissolve 24 g anhydrous disodium hydrogen phosphate, Na_2HPO_4 , and 46 g anhydrous potassium dihydrogen phosphate, KH_2PO_4 , in distilled water. Combine this solution with 100 ml 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 free available chlorine test caused by any trace amounts of iodide in the reagents.

2. N, N-DIETHYL-P-PHENYLENEDIAMINE (DPD)

Dissolve 1 g DPD oxalate or 1.5 g p-amino-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 1 liter, store in a brown glass-stoppered bottle. Discard when this solution becomes discolored.

Sample Preparation

1. Take a sample using a clean 100 ml graduated cylinder. Fill to the 100 ml mark.

2. Place 5 mls each of buffer reagent and DPD indicator solution in the graduated cylinder and mix (0.5 g DPD free chlorine reagent powder may be used). A reading must be made no longer than 1 minute after the reagent addition.

Procedure

1. Bausch & Lomb Spectronic 20
 - a. Adjust the wavelength to 530 nm
 - b. Cover the empty sample compartment and adjust the zero control so as to obtain a reading of exactly 0% transmittance.
 - c. Place a test tube containing a portion of untreated sample into the compartment and adjust the Full Scale Control so as to obtain a reading of exactly 100% transmittance.
 - d. Place a test tube containing the treated sample into the sample compartment and read the % transmittance. Refer to the table on the next page.

2. Bausch & Lomb Mini Spectronic 20

- a. Adjust wavelength to 530 nm.
- b. Using the one half inch adapter and insert, place the opaque rod in the sample compartment and check the zero adjustment. Adjust to a reading of exactly zero % transmittance.
- c. Place a 25 mm cell containing a portion of the untreated water sample into the sample compartment and adjust the Full Scale Control for a meter reading of exactly 100% transmittance.
- d. Place a 25 mm cell containing the treated sample into the sample compartment and read the % transmittance. Refer to the table for free chlorine as listed under procedures for Spectronic 20.

NOTES:

1. It is not necessary for all of the particles to dissolve to obtain an accurate reading.
2. If the sample turns yellow when adding DPD Free Chlorine Reagent or measures less than 10% transmittance, the free chlorine concentration is too high and a sample dilution is needed. A slight loss of chlorine may occur to the dilution.

TENS #T	%T UNITS									
	0	1	2	3	4	5	6	7	8	9
10	4.00	3.84	3.68	3.54	3.42	3.30	3.18	3.08	2.98	2.88
20	2.80	2.71	2.63	2.55	2.48	2.41	2.34	2.28	2.21	2.15
30	2.09	2.04	1.98	1.93	1.88	1.82	1.78	1.73	1.68	1.64
40	1.59	1.55	1.51	1.47	1.43	1.39	1.35	1.31	1.28	1.24
50	1.20	1.17	1.14	1.10	1.07	1.04	1.01	0.98	0.95	0.92
60	0.89	0.86	0.83	0.80	0.78	0.75	0.72	0.70	0.67	0.74
70	0.62	0.60	0.57	0.56	0.52	0.50	0.48	0.46	0.43	0.41
80	0.39	0.37	0.34	0.32	0.30	0.28	0.26	0.24	0.22	0.20
90	0.18	0.16	0.14	0.13	0.11	0.09	0.07	0.05	0.04	0.02

Table taken from Page 2-29, Procedures, Chemical Lists and Glassware for Water and Wastewater Analysis, 2nd Edition, Hach Chemical Co.

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 & 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_2HPO_4 , and 46 g anhydrous potassium dihydrogen phosphate, KH_2PO_4 , in distilled water.

Combine this solution with 100 ml 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-amino-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.

Or

Place 5 mls each of buffer reagent and DPD indicator solution in the graduated cylinder and mix. Add 100 mls 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 CaCO_3 , 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.

TENS
#T

%T-UNITS

	0	1	2	3	4	5	6	7	8	9
10	4.00	3.84	3.68	3.54	3.42	3.30	3.18	3.08	2.98	2.88
20	2.80	2.71	2.63	2.55	2.48	2.41	2.34	2.28	2.21	2.15
30	2.09	2.04	1.98	1.93	1.88	1.82	1.78	1.73	1.68	1.64
40	1.59	1.55	1.51	1.47	1.43	1.39	1.35	1.31	1.28	1.24
50	1.20	1.17	1.14	1.10	1.07	1.04	1.01	0.98	0.95	0.92
60	0.89	0.86	0.83	0.80	0.78	0.75	0.72	0.70	0.67	0.74
70	0.62	0.60	0.57	0.56	0.52	0.50	0.48	0.46	0.43	0.41
80	0.39	0.37	0.34	0.32	0.30	0.28	0.26	0.24	0.22	0.20
90	0.18	0.16	0.14	0.13	0.11	0.09	0.07	0.05	0.04	0.02

Table taken from Page 2-29, Procedures, Chemical Lists and Glassware for Water and Wastewater Analysis, 2nd Edition, Hach Chemical Co.

Module No:	Module Title:
Approx. Time:	Submodule Title:
Objectives: When the participant completes this topic they should be able to: <ol style="list-style-type: none">1. Indicate why the kit methods are less accurate than standard methods.2. Conduct a residual chlorine test using a kit method and compare the results to a standard method.	Topic: Use of comparator or Kit methods
	Instructional Aids: Handout Lab supplies per handout
Instructional Approach: Lecture Lab	
References: <ol style="list-style-type: none">1. Standard Methods2. Kit Instructions	
Class Assignments:	

Module No:	Topic:
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Instructor Notes:	Instructor Outline:
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Handout
Page 32 - 34

1. Discuss the use of comparator or kit methods for chlorine.
Indicate when chlorine kits are accepted.
2. Conduct a chlorine test using a kit method.

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

Reagents1. ORTHOTOLIDINE

No longer available from chemical supply houses.

2. DPD FREE CHLORINE REAGENT POWDER PILLOWS HACH #14070-993. DPD TOTAL CHLORINE REAGENT POWDER PILLOWS HACH #14064-99Procedure

(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 MINUTES7. 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 WHEEL9. EXPRESS THE RESIDUAL VALUE AS:

Total residual chlorine (mg/l)

Procedure

(For La Motte Model)

1. CLEAN COMPARATOR GLASS CELL, AS ABOVE2. ADD SAMPLE TO MARK ON GLASS CELL3. ADD 8 DROPS ORTHOTOLIDINE REAGENT TO GLASS CELL4. MIX REAGENT AND SAMPLE, AS ABOVE5. WAIT FIVE MINUTES6. 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 SAMPLE8. 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 IN 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
10. ROTATE COLOR WHEEL UNTIL THE COLOR OF THE STANDARD, AS SEEN THROUGH THE UNTREATED SAMPLE, MOST CLOSELY MATCHES THE TREATED SAMPLE
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.

Module No:	Module Title: Chlorine in Water
	Submodule Title:
Approx. Time:	Topic: SUMMARY
Objectives: When the participants complete this topic they should be able to: <ol style="list-style-type: none">1. Compare and contrast methods for residual and free chlorine and select a method best suited for an individual plant.	
Instructional Aids: All handouts	
Instructional Approach: Discussion	
References: Standard Methods	
Class Assignments:	

Module No:

Topic:

Instructor Notes:

Instructor Outline:

Compare and contrast the methods for chlorine.

Discuss:

Speed

Cost

Accuracy

Precision

Module No:	Module Title: Chlorine in Water
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.