Chlorine Field Methods

Drinking Water Field Method Training

October/November 2013

Arizona Department of Health Services

Chlorine Addition Flow Chart

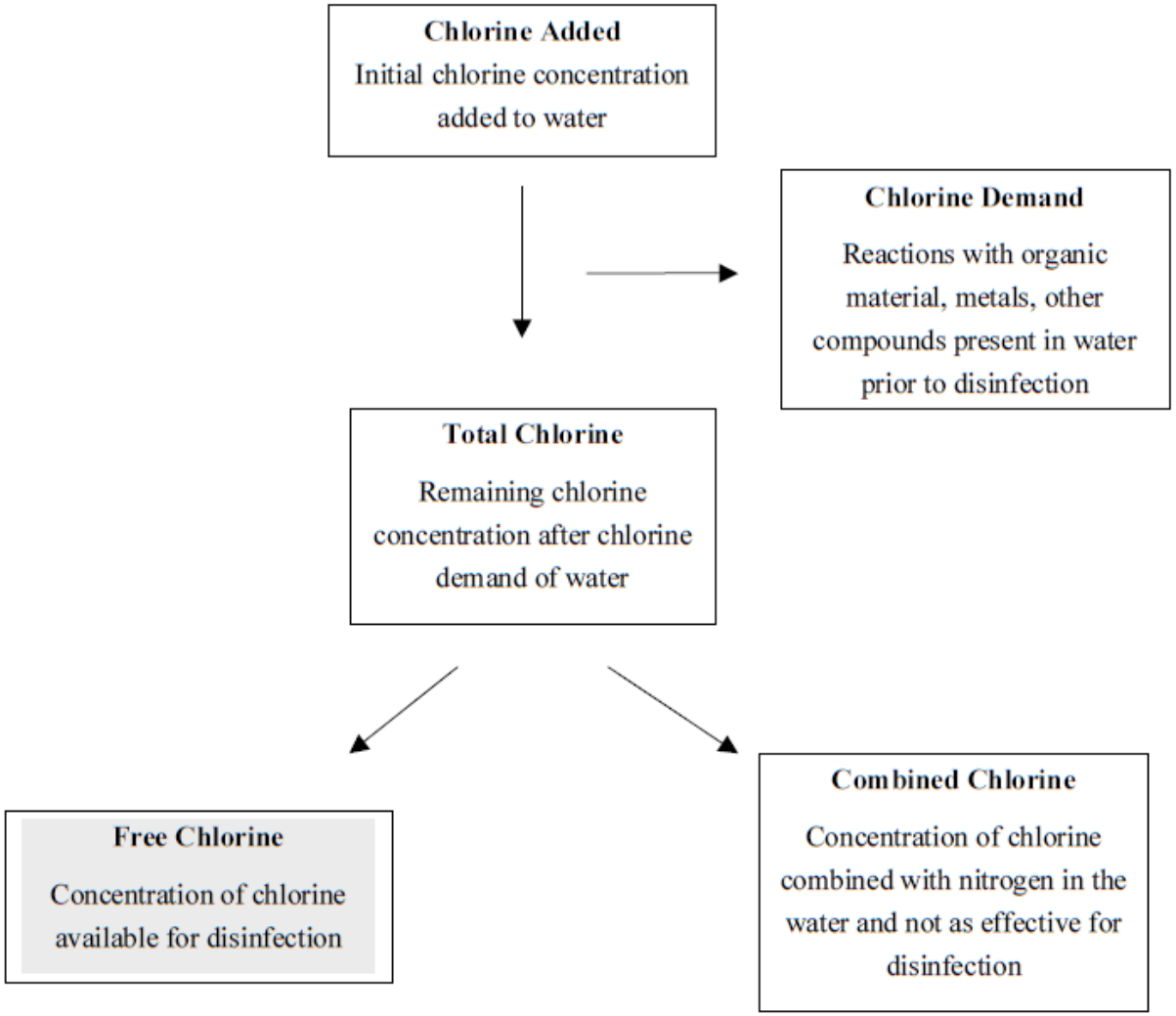

Table IV-6 Sample Containers, Preservation and Holding Times for Regulated Parameters

Parameter/ Method	Preservative	Sample Holding Time	Extract Holding Time and Storage Conditions	Suggested Sample Size	Type of Container
Metals (except Hg)	HNO_3 pH<2	6 months		1 L	Plastic or Glass
Mercury	HNO_3 pH<2	28 days		100 mL	Plastic or Glass
Alkalinity	Cool, 4C	14 days		100 mL	Plastic or Glass
Asbestos	Cool, 4C	48 hours		1 L	Plastic or Glass
Chloride	none	28 days		100 mL	Plastic or Glass
Residual Disinfectant	none	immediately		200 mL	Plastic or Glass
Color	Cool, 4C	48 hours		100 mL	Plastic or Glass
Conductivity	Cool, 4C	28 days		100 mL	Plastic or Glass
Cyanide	Cool, 4C, Ascorbic acid (if chlorinated), NaOH pH>12	14 days		1 L	Plastic or Glass

Pool Kits

Color Wheels

Not Approved Methods

Test Strips

Approved Methods

40 CFR § 141.131

Methodology	SM (19th or 20th ed)	SM Online[2]	ASTM method	EPA method	Residual measured[1]			
					Free Cl_2	Combined Cl_2	Total Cl_2	ClO_2
Amperometric Titration	4500–Cl D	4500–Cl D–00	D 1253–86 (96), 03		X	X	X	
Low Level Amperometric Titration	4500–Cl E	4500–Cl E–00					X	
DPD Ferrous Titrimetric	4500–Cl F	4500–Cl F–00			X	X	X	
DPD Colorimetric	4500–Cl G	4500–Cl G–00			X	X	X	
Syringaldazine (FACTS)	4500–Cl H	4500–Cl H–00			X			
Iodometric Electrode	4500–Cl I	4500–Cl I–00					X	
DPD	4500–ClO_2D							X
Amperometric Method II	4500–ClO_2E	4500–ClO_2E–00						X
Lissamine Green Spectrophotometric				327.0 Rev 1.1				X

Arizona Approved

SenSafe™ Free Chlorine

SenSafe™ Free Chlorine
Part Number: 480602
Detection Range:
0, 0.05, 0.1, 0.2, 0.4, 0.8, 1, 1.2, 1.6, 2, 3, 4, 5, 6, 7, 8, 9, 11, 15, 20, 25 ppm (mg/L)
Total Test Time: 1 Minute and 20 Seconds
Number of Tests: 50 Packets of 1

view details

Retail Price: **$28.49**

Method # (D99-003): Free Chlorine Species ($HOCl$ and OCl^-) by Test Strip

Industrial Test Systems, Inc.
1875 Langston St.
Rock Hill, SC 29730
(803) 329-9712

Revision 3.0
November 21, 2003

Approve Methods

Chlorite				
Amperometric titration		4500–ClO_2E[8]	4500–ClO_2E–00[8]	
Spectrophotometry	327.0 Rev 1.1[8]			
Ion chromatography	300.0, 300.1, 317.0 Rev 2.0, 326.0			D 6581–00

40 CFR § 141.131

Daily Chlorite Monitoring Methods. Analyzed Immediately. No preservation.

Chlorite				
Amperometric titration		4500–ClO_2E[8]	4500–ClO_2E–00[8]	
Spectrophotometry	327.0 Rev 1.1[8]			
Ion chromatography	300.0, 300.1, 317.0 Rev 2.0, 326.0			D 6581–00

40 CFR § 141.131

Approve Methods

Chlorite				
Amperometric titration		4500–ClO_2E[8]	4500–ClO_2E–00[8]	
Spectrophotometry	327.0 Rev 1.1[8]			
Ion chromatography -Confirmation Method -Preserved with EDA -14 Day Hold Time -Licensed Lab	300.0, 300.1, 317.0 Rev 2.0, 326.0			D 6581–00

40 CFR § 141.131

Approved DPD Colorimetric Methods (Field)

Free and Total Chlorine

- SM 4500-Cl-G (Hach 8021 & 8167)
  - American Public Health Association et al., Standard Methods for the Examination of Water and Wastewater (20th ed. 1998), available from American Public Health Association, 800 I St., NW, Washington, DC 20001.

Chlorine Dioxide (Hach 10126)

- SM 4500-ClO2-D

Note: Hach has a special price for these for Arizona trainees

0 item(s

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< product 1

Pocket Colorimeter™ II, Chlorine (Free & Total), Mid Range/High

Overview | Details | Downloads | Accessories | Similar Products | Reviews

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Product #: 5870062
USD Price: $420.00
Ships within 3 days

Quantity 1

Add to Cart

Optional Accessories

Pocket Colorimeter II Custom Holster
USD Price: $22.15
» Add to Order

Calibration

Most meters (including Hach) are factory calibrated and do not need routine calibration. The meter must be checked, however, for test bias by performing an Accuracy Check or Calibration Check (See QA/QC below).

General Procedure

Chlorine, Free

DOC316.53.01023

USEPA DPD Method[1] **Method 8021**

(0.02 to 2.00 mg/L) **Powder Pillows or AccuVac® Ampuls**

Scope and Application: For testing free chlorine (hypochlorous acid and hypochlorite ion) in water, treated waters, estuary and seawater. USEPA accepted for reporting for drinking water analyses.[2]

[1] Adapted from *Standard Methods for the Examination of Water and Wastewater.*

[2] Procedure is equivalent to USEPA and Standard Method 4500-Cl G for drinking water.

Test preparation

How to use instrument-specific information

The *Instrument-specific information* table displays requirements that may vary between instruments. To use this table, select an instrument then read across to find the corresponding information required to perform this test.

Table 102 Instrument-specific information

Instrument	Powder pillows		AccuVac Ampuls	
	Sample cell	Cell orientation	Sample cell	Adapter
DR 6000	2495402	Fill line faces right	2427606	—
DR 5000	2495402	Fill line faces user	2427606	—
DR 3900	2495402	Fill line faces user	2427606	LZV846 (A)
DR 3800, DR 2800, DR 2700	2495402	Fill line faces right	2122800	LZV584 (C)

Before starting the test:

If the test over-ranges, dilute the sample with a known volume of high quality, chlorine demand-free water and repeat the test. Some loss of chlorine may occur due to the dilution. Multiply the result by the dilution factor. Alternatively, samples with high chlorine concentrations may be analyzed directly without dilution by using Method 10069, Chlorine, Free HR, or Method 10245, Chlorine Free MR .
The SwifTest Dispenser for Free Chlorine can be used in place of the powder pillow in step 4.
Analyze samples immediately. Do not preserve for later analysis.
The sample cell shown is a generic representation. Refer to *Instrument-specific information* for the correct sample cell and adapter configuration.

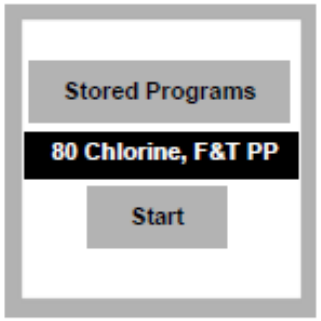
General Procedure

Sample collection, preservation and storage

- Analyze samples for chlorine immediately after collection. Free chlorine is a strong oxidizing agent and it is unstable in natural waters. It reacts rapidly with various inorganic compounds and more slowly oxidizes organic compounds. Many factors, including reactant concentrations, sunlight, pH, temperature and salinity influence decomposition of free chlorine in water.
- Avoid plastic containers since these may have a large chlorine demand.
- Pretreat glass sample containers to remove any chlorine demand by soaking in a dilute bleach solution (1 mL commercial bleach to 1 liter of deionized water) for at least 1 hour. Rinse thoroughly with deionized or distilled water. If sample containers are rinsed thoroughly with deionized or distilled water after use, only occasional pre-treatment is necessary.

General Procedure

Powder pillow procedure



1. Select the test.

Insert an adapter if required (see *Instrument-specific information*).

Refer to the user manual for orientation.

2. **Blank Preparation:** Fill a sample cell with 10 mL of sample.

3. Wipe the blank and insert it into the cell holder.

ZERO the instrument.

The display will show:

0.00 mg/L Cl_2

4. **Prepared Sample:** Fill a second cell with 10 mL of sample.

Add the contents of one DPD Free Chlorine Powder Pillow to the sample cell.

5. Swirl the sample cell for 20 seconds to mix.

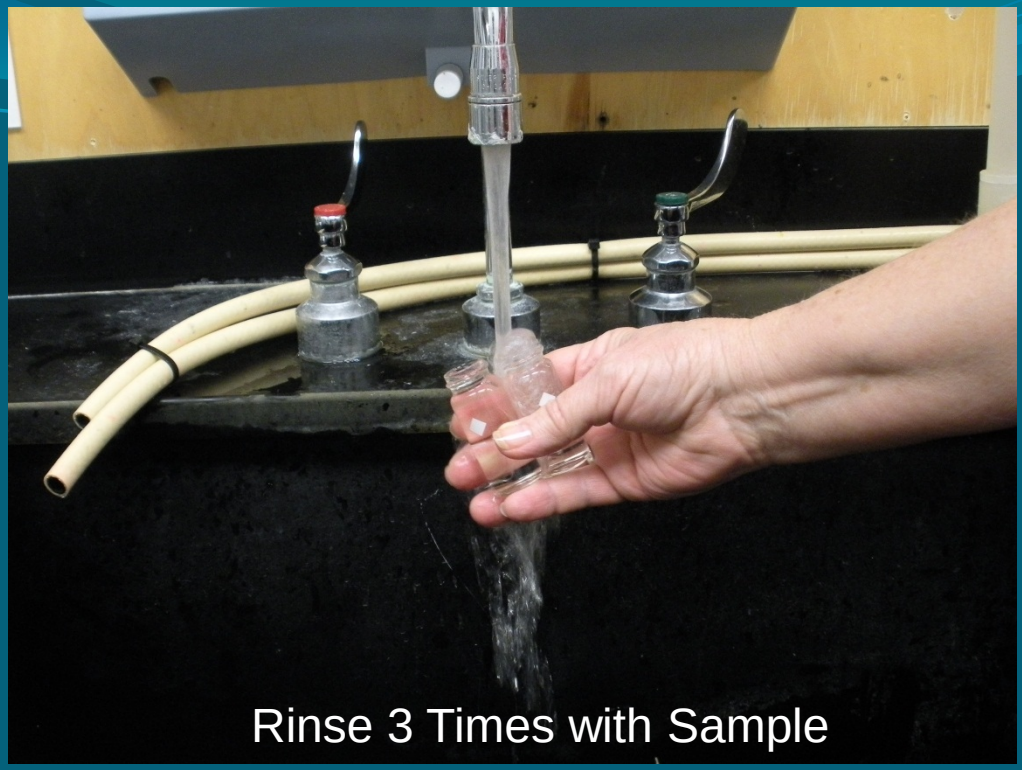
A pink color will develop if chlorine is present. Proceed to step 6 immediately.

6. Within one minute of adding the reagent, insert the prepared sample into the cell holder.

Results are in mg/L Cl_2.

Note: Hach Method Procedures Work For About 85% of The Necessary SOP.

2. Blank Preparation:
Fill a sample cell with 10 mL of sample.


Rinse 3 Times with Sample

General Procedure

3. Wipe the blank and insert it into the cell holder.

ZERO the instrument.

The display will show:

0.00 mg/L Cl_2

General Procedure

3. Wipe the blank and insert it into the cell holder.

ZERO the instrument.

The display will show:

0.00 mg/L Cl_2

General Procedure

3. Wipe the blank and insert it into the cell holder.

ZERO the instrument.

The display will show:

0.00 mg/L Cl_2

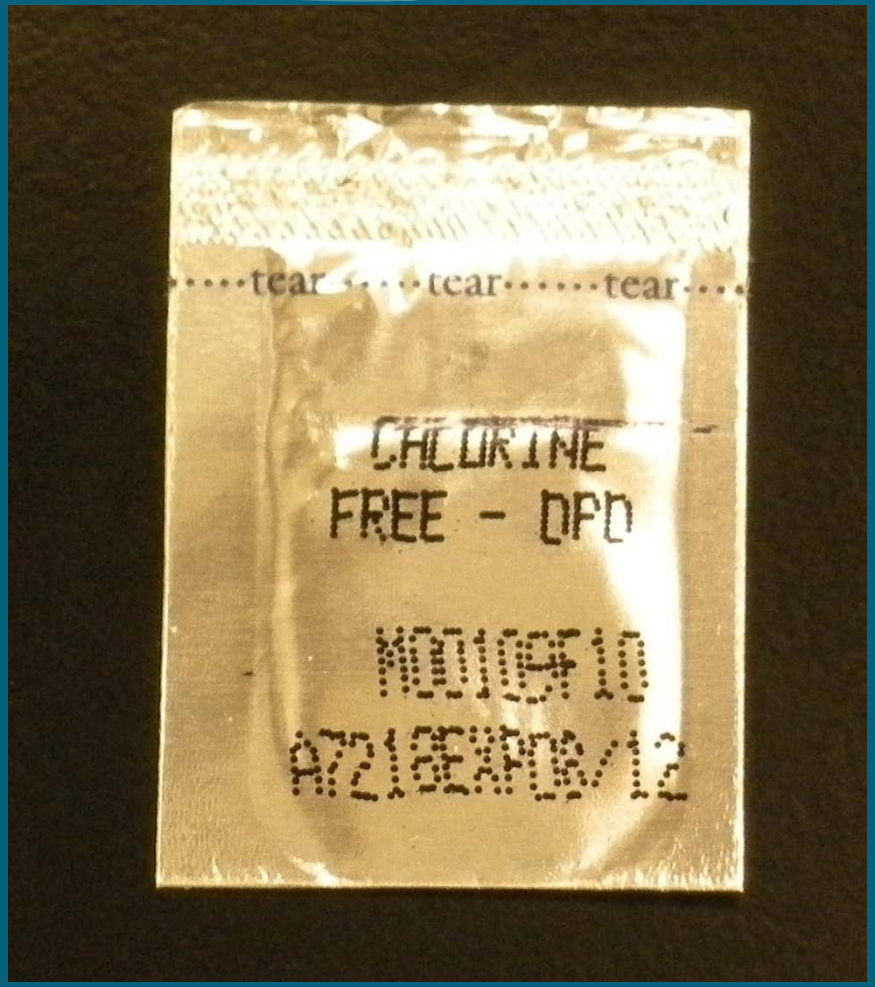
Cover On

0.00 mg/L

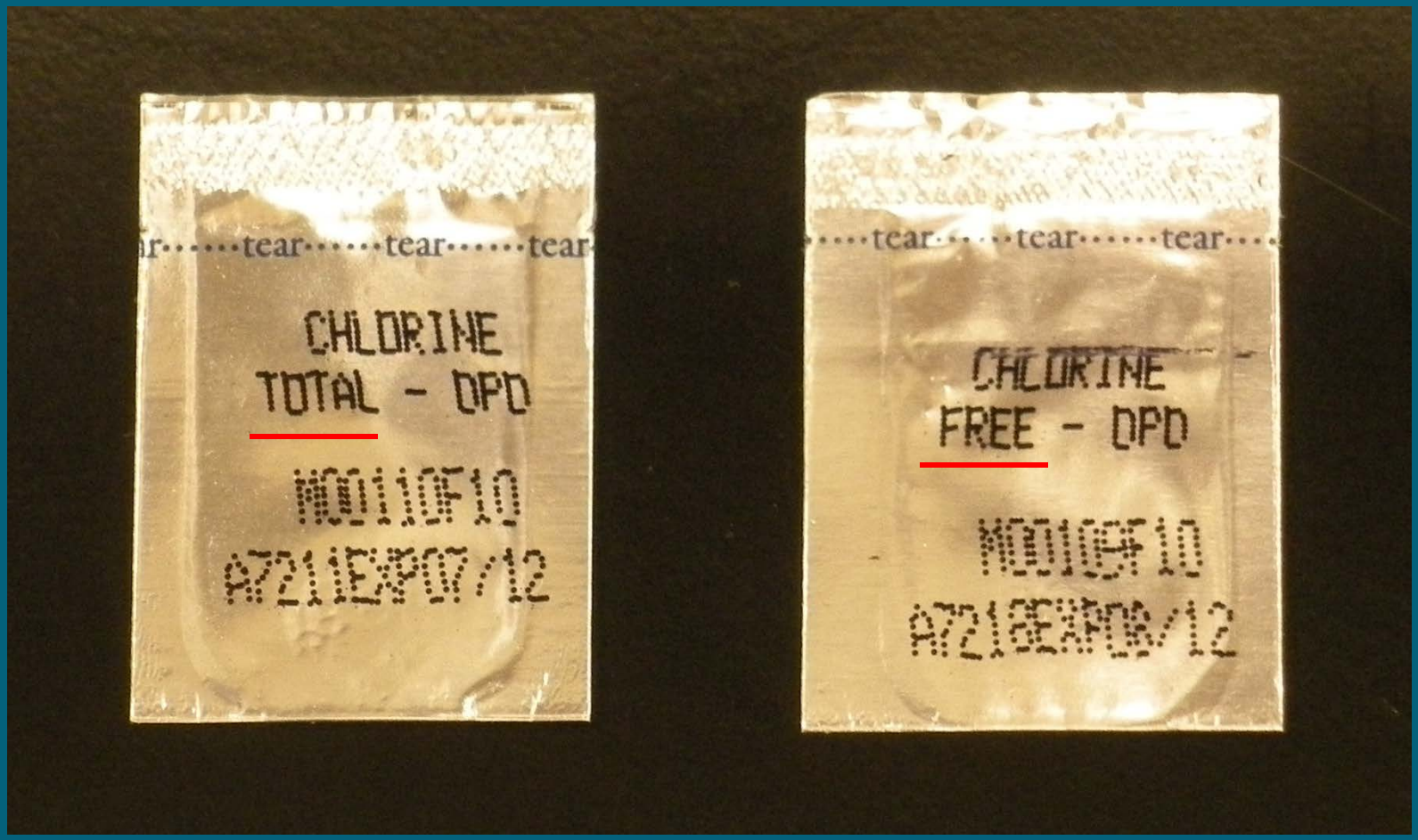
General Procedure

4. Prepared Sample:
Fill a second cell with 10 mL of sample.

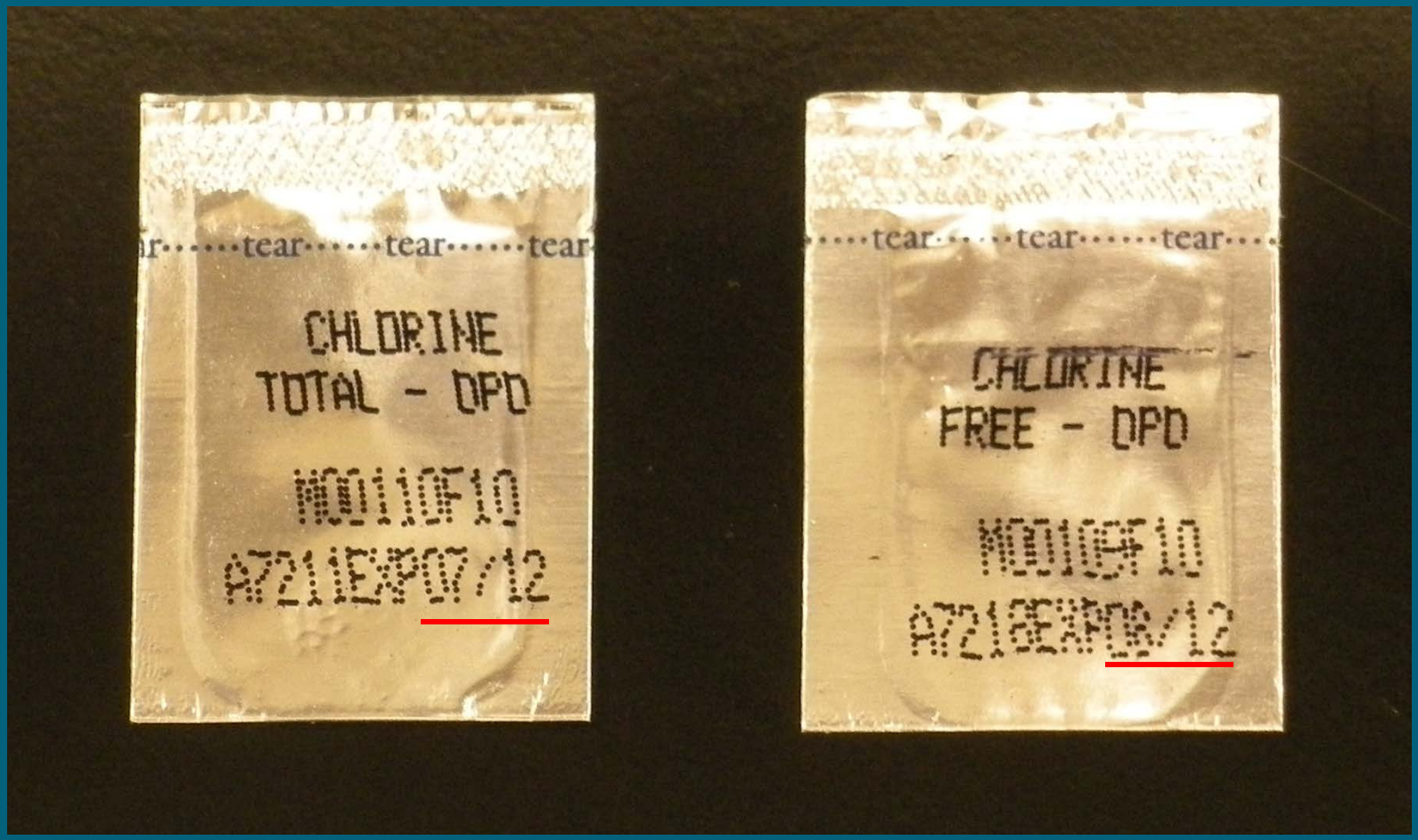
Add the contents of one DPD Free Chlorine Powder Pillow to the sample cell.



General Procedure



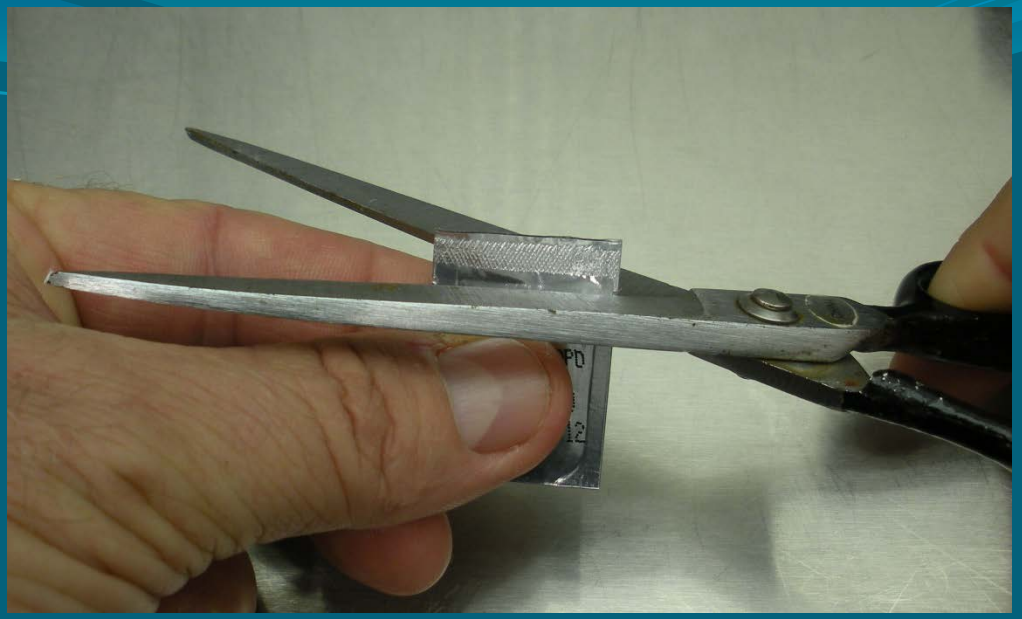
General Procedure



General Procedure

4. Prepared Sample:
Fill a second cell with 10 mL of sample.

Add the contents of one DPD Free Chlorine Powder Pillow to the sample cell.



General Procedure

5. Swirl the sample cell for 20 seconds to mix.

A pink color will develop if chlorine is present. Proceed to step 6 immediately.

General Procedure

6. Within one minute of adding the reagent, insert the prepared sample into the cell holder.

Results are in mg/L Cl_2.

General Procedure

6. Within one minute of adding the reagent, insert the prepared sample into the cell holder.

Results are in mg/L Cl_2.

Cover On

⩽ 0.31 mg/L

General Procedure

Chlorine, Total

DOC316.53.01027

USEPA[1] DPD Method[2]

Method 8167

(0.02 to 2.00 mg/L)

Powder Pillows or AccuVac® Ampuls

Scope and Application: For testing residual chlorine and chloramines in water, wastewater, estuary water and seawater; USEPA-accepted[1] for reporting for drinking and wastewater analyses.

[1] Procedure is equivalent to USEPA method and Standard Method 4500-Cl G for drinking water and wastewater analyses.

[2] Adapted from *Standard Methods for the Examination of Water and Wastewater.*

Test preparation

How to use instrument-specific information

The *Instrument-specific information* table displays requirements that may vary between instruments. To use this table, select an instrument then read across to find the corresponding information required to perform this test.

Table 110 Instrument-specific information

Instrument	Powder pillows		AccuVac Ampuls	
	Sample cell	Cell orientation	Sample cell	Adapter
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DR 5000	2495402	Fill line faces user	2427606	—
DR 3900	2495402	Fill line faces user	2427606	LZV846 (A)
DR 3800, DR 2800, DR 2700	2495402	Fill line faces right	2122800	LZV584 (C)

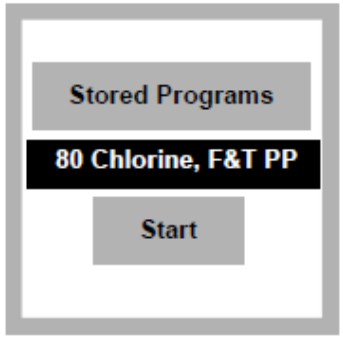
Before starting the test:

Samples must be analyzed immediately and cannot be preserved for later analysis

General Procedure

General Procedure

DPD method for powder pillows



1. Select the test.

Insert an adapter if required (see *Instrument-specific information*).

2. Fill a sample cell with 10 mL of sample.

3. Prepared Sample:

Add the contents of one DPD Total Chlorine Powder Pillow to the sample cell.

Swirl the sample cell for 20 seconds to mix.

4. Start the instrument timer.

A three-minute reaction period will begin. Perform steps 5 and 6 during this time period.

5. Blank Preparation: Fill a second sample cell with 10-mL of sample.

6. Wipe the blank sample cell and insert it into the cell holder.

ZERO the instrument. The display will show: 0.00 mg/L Cl_2

7. Within three minutes after the timer expires, wipe the prepared sample and insert it into the cell holder.

READ the results in mg/L Cl_2.

Accuracy check

Standard additions method (Sample spike)
Required for accuracy check:

- Chlorine Standard Solution, 2-mL PourRite® Ampule, 25–30 mg/L
- Breaker, PourRite Ampules
- Pipet, TenSette®, 0.1–1.0 mL and tips

1. After reading test results, leave the sample cell (unspiked sample) in the instrument.
2. Select Options>More>Standard Additions from the instrument menu.
3. Enter the average chlorine concentration shown on the label of the ampule container.
4. A summary of the standard additions procedure will be displayed. Press **OK** to accept the default values for standard concentration, sample volume and spike volumes. After the values are accepted, the unspiked sample reading will appear in the top row.
5. Open one Voluette ampule standard.
6. Prepare spiked samples: add 0.1 mL, 0.2 mL and 0.3 mL of standard to three 10-mL portions of fresh sample.

   Note: *For AccuVac® Ampuls, add 0.4 mL, 0.8 mL and 1.2 mL of standard to three 50-mL portions of fresh sample.*

7. Follow the test procedure for each of the spiked samples using the powder pillows or AccuVac ampules, starting with the smallest sample spike. Measure each of the spiked samples in the instrument.
8. Select **GRAPH** to view the results. Select **IDEAL LINE** (or best-fit) to compare the standard addition results to the theoretical 100% recovery.

Note: *If results are not within acceptable limits (± 10%), be sure that the sample volumes and sample spikes are measured accurately. The sample volumes and sample spikes that are used should agree with the selections in the standard additions menu. If all procedures are followed correctly but the standard additions results are not within acceptable limits, the sample may contain an interference.*

QA/QC (Round 1)

- Sample Blank
- Accuracy check (Calibration Check)
  - 0.5-1.0 mg/L for Total & Free Chlorine for surface water testing.
  - 0.5-4.0 mg/L for ground water testing.
  - Once per month per meter.
  - 80-120% of expected value

-Need Written Records for Blank & CC.

SpecCheck Secondary Gel Standards Set, DPD Chlorine - LR

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Product #: 2635300
USD Price: $145.00
Available

Quantity 1

Add to Cart

★★★★★

Write the first review | Follow this product

DPD Chlorine, Low Range, 0-2.00 mg/L as Cl_2 .
Used to confirm consistent instrument response.
Set contains four vials filled with gels that simulate the test color at various concentrations.

- Fast and Convenient
- Stable

Optional Accessories

Disposable Wipes, 11 x 22 c
280 per package
USD Price: $5.55
» Add to Order

Chlorine Check Standard Snips

The easiest way to QC your routine chlorine analysis. Our certified residual chlorine standard snips deliver the exact volume required without pipetting. To use, fill a flask or cuvette to about half the target final volume. Cut open a snip with a sharp pair of scissors. Pour the contents into the cuvette or flask. Bring to volume with reagent water. Available in 3 concentrations each for both colorimetric and amperometric titration methods. Packaged as 25 snips per pack.

Colorimetric	Part#	Price		Amperometric	Part#	Price
.100 mg/L	QCI-118	$45.00		0.1 mg/L	QCI-148	$45.00
1.00 mg/L	QCI-123	$45.00		1.0 mg/L	QCI-149	$45.00
2.50 mg/L	QCI-117	$45.00		2.0 mg/L	QCI-150	$45.00

NSI Solutions, Inc.

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Volumetric Flask, Class A, Glass, 200mL, Pk6

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Volumetric Flask, Class A, Capacity 200mL., Material of Construction Glass, Tolerance 0.15ml, Stopper Size 14/15, Color Clear, Includes Stopper

Grainger Item #	5YHZ8
Price (pk.)	**$49.60**
Package Qty.	6
Brand	GRAINGER APPROVED VENDOR
Mfr. Model #	5YHZ8
UNSPSC #	41121804
Ship Qty.	1
Sell Qty. (Will-Call)	1
Ship Weight (lbs.)	1.75
Availability	**Item ships within 4 business days from supplier**

HACH

ShareThis Prin

Chlorine Standard Solution, 25-30 mg/L as Cl_2 , pk/20 - 2 mL PourRite Ampules (NIST)

Overview | Details | Parameter/Reagent | Downloads | Accessories | Similar Products | Reviews

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Product #: 2630020
USD Price: $40.85
Available

Quantity 1

Add to Cart

★★★★★

Write the first review | Follow this product

For accuracy checks in chlorine determinations. Pack of 20 2-mL ampules. Lot-specific concentration displayed on label.

- Simple Accuracy Checks
- Minimize waste
- Reliable results

Optional Accessories | View all

image coming soon

Pipet Tips, for TenSette Pipet 1970010, 1.0-10.0 mL, Non-Sterile, pk/250
USD Price: $43.85
» Add to Order

Pipet Tips, for TenSette Pipet 1970001, 0.1-1.0 mL, pack of 50
USD Price: $12.45
» Add to Order

» Shipping Policy and Rates
» Return Policy
» Hach Warranty
» Terms and Conditions

0.5 ml Pipetman

Formula for Hach Standard

$$\frac{(0.5 \text{ ml of standard added}) \times (25\text{-}30 \text{ mg/L } Cl_2)}{10.5 \text{ ml (sample cell volume + standard added)}}$$

= 1.19-1.43 mg/L

(12 ml sample cell volume + 0.5 ml standard added = 1.00-1.2 mg/L)

Approved Amperometric Methods (Field)

Free, Total and Combined Chlorine

- SM 4500-Cl-D & -E
  - American Public Health Association et al., Standard Methods for the Examination of Water and Wastewater (20th ed. 1998), available from American Public Health Association, 800 I St., NW, Washington, DC 20001.

Chlorine Dioxide

- SM 4500-ClO2-E

Chlorite

- SM 4500-ClO2-E

Autotitrator
Or
AutoCat 9000
Amperometric

Part Number	Item Description	Qty	Unit Price	Ext. Price
5008100	AUTOCAT 9000 COMPLETE UNIT	1	$3,571.00	$3,571.00
			Grand Total	$3,571.00

General Procedure

-SM 4500-ClO2 E (Chlorine Dioxide and Chlorite)

This procedure entails four successive titrations of combinations of chlorine species (30-45 minutes). Subsequent calculations determine the concentration of each species.

QA/QC

- Accuracy check (Standard)
  - 0.5-1.0 mg/L for Chlorite
  - Once per month per meter. Recommend weekly.
  - 80-120% of expected value
  - No check for chlorine dioxide!

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Analytical Solutions

CERTIFIED ANALYTICAL STANDARDS SINCE 1976

ULTRA easy,
ULTRA smart!

Product Details

Chlorite Standard

Material Safety Data Sheet / Components List

1 Analyte(s) @ 1000 µg/mL in water **Min 3 month, max 6 month expiration date**

Catalog #	Sale Unit	Price	Quantity	
ICC-012	125 mL	$45.65	1	Add To Cart
ICC-012-5	Multi-component Kit	$146.05	1	Add To Cart

METHOD 334.0: DETERMINATION OF RESIDUAL CHLORINE IN DRINKING WATER USING AN ON-LINE CHLORINE ANALYZER

Version 1.0

September 2009

Steven C. Wendelken, Derek E. Losh, and Patricia S. Fair
Office of Ground Water and Drinking Water

TECHNICAL SUPPORT CENTER
OFFICE OF GROUND WATER AND DRINKING WATER
U. S. ENVIRONMENTAL PROTECTION AGENCY
CINCINNATI, OHIO 45268

In-Line Meters

- 40 CFR § 141.131 footnote 2) – Instruments used for continuous monitoring must be calibrated with a grab sample measurement at least every five days, or with a protocol approved by the state.
- EPA Method 334.0 (Now Arizona Approved)

  -Analyze grab sample and compare to analyzer and adjust analyzer to agree with the grab sample measurement unit the analyzer is within ±0.1 mg/L or ± 15% of grab sample measurement.

  -Routine calibration check: Grab sample weekly compared to analyzer measurement.

Questions?