# **DPD Method Powder Pillows**

# USEPA accepted for reporting wastewater and drinking water analyses\*

*Note:* This product has not been evaluated to test for chlorine and chloramines in medical applications in the United States.

# **Using Powder Pillows**





**1.** Enter the stored program number for free and total chlorine (Cl<sub>2</sub>) powder pillows.

#### Press: PRGM

The display will show:

#### PRGM ?

Note: For most accurate results, perform a Reagent Blank Correction using deionized water. 2. Press: 114 ENTER The display will show mg/L, Cl2 and the ZERO icon.



**3.** Fill a sample cell with 10 mL of sample (the blank).

Note: Samples must be analyzed immediately and cannot be preserved for later analysis.



**4.** Place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

<sup>\*</sup> Procedure is equivalent to USEPA method 330.5 for wastewater and Standard Method 4500-Cl G for drinking water.

# CHLORINE, FREE, continued



5. Press: ZERO

The cursor will move to the right, then the display will show:

#### 0.00 mg/L Cl2

*Note:* If Reagent Blank Correction is on, the display may flash "limit".



**6.** Fill another cell with 10 mL of sample.



**7.** Add the contents of one DPD Free Chlorine Powder Pillow for 25-mL samples to the sample cell (the prepared sample). Cap the cell and swirl vigorously to dissolve the powder.

*Note:* A pink color will develop if free chlorine is present.



**8.** Immediately place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.

*Note:* Perform Step 9 within one minute of reagent addition.



## 9. Press: READ

The cursor will move to the right, then the result in mg/L chlorine will be displayed. Note: If the sample temporarily turns yellow after reagent addition, or the display flashes "limit", it is due to high chlorine levels. Dilute a fresh sample and repeat the test. A slight loss of chlorine may occur during dilution. Multiply the result by the dilution factor. Or, use the High Range Free Chlorine test, program #8. *Note:* Standard Adjust may be performed using a prepared standard.

# Sampling 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 l 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 pretreatment is necessary.

Do not use the same sample cells for free and total chlorine. If trace iodide from the total chlorine reagent is carried over into the free chlorine determination, monochloramine will interfere. It is best to use separate, dedicated sample cells for free and total chlorine determinations.

A common error in testing for chlorine is introduced when a representative sample is not obtained. If sampling from a tap, let the water flow for at least 5 minutes to ensure a representative sample. Let the container overflow with the sample several times, then cap the sample container so there is no headspace (air) above the sample. If sampling with a sample cell, rinse the cell several times with the sample, then carefully fill to the 10-mL mark. Perform the analysis immediately.

# **Accuracy Check**

#### Standard Additions Method (using powder pillows)

- a) Snap the top off a LR Chlorine PourRite Ampule Standard Solution.
- **b**) Use a TenSette Pipet to add 0.1 mL of the standard to the reacted sample (this is the spiked sample). Swirl to mix.
- c) Re-zero the instrument using the original sample (the blank).
- **d**) Place the spiked sample in the cell holder and press **READ**. Record the results.

e) Calculate the concentration of mg/L chlorine added to the sample:

 $mg/L \ Chlorine \ added \ = \ \frac{0.1(vol. \ standard \ added) \times Label \ value \ (mg/L \ Cl_2)}{10.1(sample + standard \ volume)}$ 

f) The spiked sample result (step d) should reflect the analyzed sample result + the calculated mg/L  $Cl_2$  added (step e).

# **Method Performance**

In a single laboratory using a standard solution of 1.50 mg/L chlorine and two representative lots of reagents with the instrument, a single operator obtained a standard deviation of  $\pm 0.02$  mg/L chlorine.

# Interferences

Interfering Substance	Interference Level and Treatment
Acidity	Greater than 150 mg/L CaCO <sub>3</sub> . May not develop full color or color may fade instantly. Neutralize to pH 6-7 with 1 N sodium hydroxide. Determine amount to be added on separate sample aliquot, then add the same amount to the sample being tested. Correct for volume addition.
Alkalinity	Greater than 250 mg/L CaCO <sub>3</sub> . May not develop full color or color may fade instantly. Neutralize to pH 6-7 with 1 N sulfuric acid. Determine amount to be added on separate sample aliquot, then add the same amount to the sample being tested. Correct for volume addition.
Bromine	Interferes at all levels
Chlorine Dioxide	Interferes at all levels
Chloramines, organic	May interfere
Hardness	No effect at less than 1,000 mg/L as CaCO <sub>3</sub>
Iodine	Interferes at all levels
Manganese, Oxidized (Mn <sup>4+</sup> , Mn <sup>7+</sup> ) or Chromium, Oxidized (Cr <sup>6+</sup> )	<ol> <li>Adjust sample pH to 6-7.</li> <li>Add 3 drops potassium iodide (30 g/L) to a 25-mL sample.</li> <li>Mix and wait one minute.</li> <li>Add 3 drops sodium arsenite (5 g/L) and mix.</li> <li>Analyze 10 mL of the treated sample as described in the procedure.</li> <li>Subtract the result from this test from the original analysis to obtain the correct chlorine concentration.</li> </ol>
Monochloramine	Causes a gradual drift to higher readings. When read within 1 minute after reagent addition, 3 mg/L monochloramine causes less than a 0.1 mg/L increase in the reading.
Ozone	Interferes at all levels

Interfering Substance	Interference Level and Treatment
Peroxides	May interfere
Extreme sample pH and highly buffered samples	Adjust to pH 6-7.

## **Summary of Method**

Chlorine in the sample as hypochlorous acid or hypochlorite ion (free chlorine or free available chlorine) immediately reacts with DPD (N,N-diethyl-p-phenylenediamine) indicator to form a pink color which is proportional to the chlorine concentration.

## **Pollution Prevention and Waste Management**

Samples treated with sodium arsenite for manganese or chromium interferences will be hazardous wastes as regulated by Federal RCRA for arsenic (D004). Refer to the appropriate Material Safety Data Sheet (MSDS) for more information on proper disposal of these materials.

#### **Instrument Setup**

The following procedure will add program 114 to a DR/820, DR/850 or DR/890 instrument.

- 1. Turn on the instrument by pressing the ON key.
- **2.** Press the **SETUP** key.
- 3. Press the DOWN arrow key until the prompt line shows USER.
- **4.** Press the ENTER key.
- 5. Enter "8138", followed by ENTER.

# *Note:* The arrow keys can be used to scroll and review or change numbers at any time.

**6.** Refer to the following table and enter the number from the "Enter" column that corresponds to line number 1 on the display. Press **ENTER**. Repeat for lines 2–56 on the display.

# CHLORINE, FREE continued

Line Number	Enter	Line Number	Enter
1	114	29	0
2	24	30	0
3	74	31	0
4	0	32	0
5	0	33	0
6	0	34	0
7	0	35	0
8	0	36	0
9	0	37	0
10	0	38	0
11	0	39	0
12	63	40	0
13	219	41	0
14	34	42	0
15	200	43	1
16	0	44	164
17	0	45	0
18	0	46	4
19	0	47	10
20	67	48	0
21	108	49	180
22	50	50	0
23	0	51	0
24	0	52	0
25	0	53	0
26	0	54	223
27	0	55	0
28	0	56	255

#### **REQUIRED REAGENTS & APPARATUS (Using Powder Pillows)**

Quantity Required			
Per Test	Unit	Cat. No.	
1 pillow	100/pkg	1407099	
2	6/pkg	2401906	
	Quantity Required Per Test 1 pillow 	Quantity Required Per Test Unit 1 pillow 100/pkg 	

# **OPTIONAL REAGENTS**

Description	Unit	Cat. No.
Chlorine Standard Solution, PourRite ampule, 25-30 mg/L, 2 mL	.20/pkg	2630020
Potassium Iodide Solution, 30 g/L100 mL	<sup>*</sup> MDB	34332
Sodium Arsenite, 5 g/L 100 mL	* MDB	
Sodium Hydroxide Standard Solution, 1.000 N 100 mL	* MDB	104532
Sulfuric Acid Standard Solution, 1.000 N 100 mL	* MDB	127032
Water, deionized	4 L	

# **OPTIONAL APPARATUS**

Description	Unit	Cat. No.
Cylinder, graduated, 25 mL	each	
pH Meter, HQ30d, single-input, multi-parameter	each	.HQ30D53000000
pH probe, standard gel, 1-m cable	each	PHC10101
pH Paper, 1 to 11 pH units	5 rolls/pkg	
Pipet, TenSette, 0.1 to 1.0 mL	each	
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg	
Pipet Tips, for 19700-01 TenSette Pipet	1000/pkg	
PourRite Ampule Breaker	each	2484600

#### For Technical Assistance, Price and Ordering

In the U.S.A.—Call 800-227-4224 Outside the U.S.A.—Contact the Hach office or distributor serving you.

<sup>\*</sup> Marked Dropper Bottle - contact Hach for larger sizes.



FOR TECHNICAL ASSISTANCE, PRICE INFORMATION AND ORDERING: In the U.S.A. – Call toll-free 800-227-4224 Outside the U.S.A. – Contact the HACH office or distributor serving you. On the Worldwide Web – www.hach.com; E-mail – techhelp@hach.com HACH COMPANY WORLD HEADQUARTERS Telephone: (970) 669-3050 FAX: (970) 669-2932

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