



ANALYTICAL PROCEDURES

For DR/800 Instruments

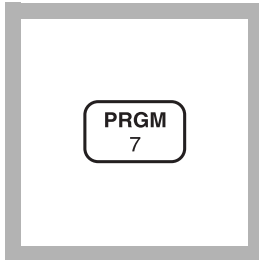
CHLORINE, TOTAL, UHR

Method 10070

DPD Method*

(0.0–10.0 mg/L Cl₂)

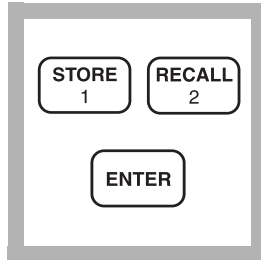
For testing higher levels of total chlorine (free and combined)
in drinking water, cooling water,
industrial process waters, or treated wastewater



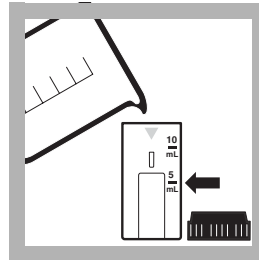
1. Enter the user program number for Chlorine, UHR.

Press: **PRGM**
The display will show:
PRGM?

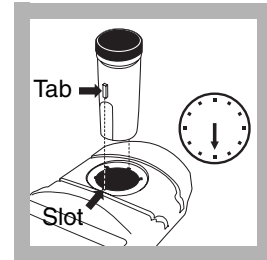
Note: If the chlorine is typically less than 2.0 mg/L, use method 8167, program number 9.



2. Press:
12 ENTER
The display will show
mg/L Cl₂
then: **ZERO**



3. Fill the 10-mL/1-cm cell to the 5-mL line with sample.

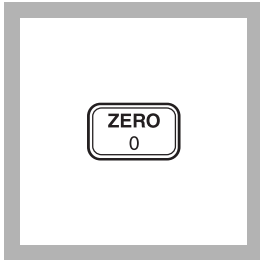


4. Place the sample cell into the instrument. Cover the sample cell tightly with the instrument cap.

Note: Place the cell into the cell holder as illustrated. The sample cell tab should be at the 6 o'clock position and completely seated in the cell holder slot.

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

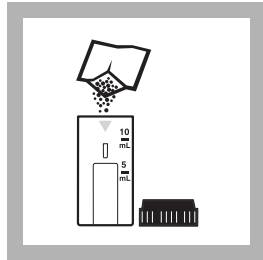
CHLORINE, TOTAL, UHR, continued



5. Press: ZERO

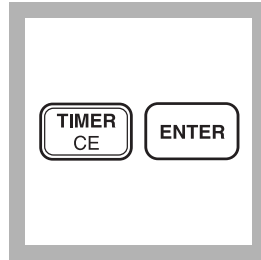
The cursor will move to the right, then the display will show:

0.0 mg/L Cl₂



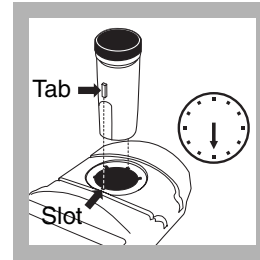
6. Remove the sample cell from the cell holder and add the contents of one DPD Total Chlorine Reagent pillow to the sample. Cap and shake the sample cell about 20 seconds to dissolve.

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



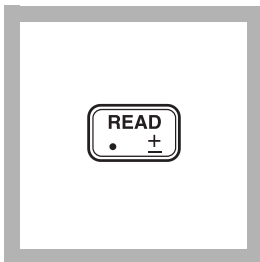
7. Press: TIMER ENTER

A 3-minute reaction period will begin.



8. Within 3 minutes after the timer beeps, place the sample cell into the instrument. Cover the sample cell tightly with the instrument cap.

Note: Place the cell into the cell holder as illustrated. The sample cell tab should be at the 6-o'clock position and completely seated in the cell holder slot.



9. Press: READ

The cursor will move to the right. The result in mg/L chlorine (as Cl₂) will be displayed.

CHLORINE, TOTAL, UHR, continued

Sampling and Storage

Analyze samples for chlorine immediately after collection. Free and combined chlorine are strong oxidizing agents and react rapidly with various compounds. Many factors such as sunlight, pH, temperature, and sample composition will influence decomposition of chlorine in water.

- Avoid plastic containers which may have a large chlorine demand.
- Pretreat glass sample containers to remove chlorine demand by soaking in a dilute bleach solution (1 mL of commercial bleach to 1 liter of deionized water) for at least one 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.
- Use separate, dedicated sample cells for free and total chlorine determinations. If trace iodide from the total chlorine reagent is carried over to the free chlorine test, monochloramine could interfere.
- A common error in testing for chlorine is failure to obtain a representative sample. If sampling from a tap, let the water flow for at least five minutes to ensure a representative sample. Let the sample container overflow with sample several times. Cap the container so there is no air above the sample.
- If sampling with a sample cell, rinse the cell several times with the sample, then carefully fill to the 5-mL mark. Proceed with the chlorine test immediately.

Accuracy Check

1. Fill three mixing cylinders (Cat. No. 20886-38) with 5-mL of sample.
2. Snap the neck of a HR Chlorine Ampule Standard, 50–75 mg/L Cl₂. Using the TenSette® Pipet, add 0.1 mL, 0.2 mL, and 0.3 mL of standard, respectively, to each cylinder and mix thoroughly.

CHLORINE, TOTAL, UHR, continued

- Analyze each standard addition sample as described in the procedure. Record each result.
- Calculate the concentration of mg/L chlorine added to each sample.

$$\text{mg/L chlorine added} = \frac{\text{volume of standard added} \times \text{label value of Cl}_2 \text{ standard ampule}}{\text{sample volume} + \text{volume of standard added}}$$

The spiked sample results should reflect the analyzed sample result plus the calculated mg/L Cl₂ added to each sample. If these increases do not occur, see Standard Additions in Section 1 of the DR/800 Instrument Manual for more information.

Method Performance

Precision

In a single laboratory, using a chlorine standard solution of 5.05 mg/L Cl₂ and representative lots of reagent, a single operator obtained a standard deviation of ± 0.05 mg/L Cl₂.

Estimated Detection Limit

The estimated detection limit for Method 10070 is 0.05 mg/L Cl₂. For more information on the estimated detection limit, see Section 1 of a DR/850 or DR/890 Procedure Manual.

Interferences

Interfering Substance	Interference Levels and Treatments
Acidity	Greater than 150 mg/L CaCO ₃ . May not develop full color or color may fade instantly. <ol style="list-style-type: none">Neutralize to pH 6–7 with 1 N Sodium Hydroxide.Determine amount to be added on a separate sample aliquot, then add the same amount to the sample being tested.Correct for volume addition.
Alkalinity	Greater than 250 mg/L CaCO ₃ . May not develop full color or color may fade instantly. <ol style="list-style-type: none">Neutralize to pH 6–7 with 1 N Sulfuric Acid.Determine amount to be added on a separate sample aliquot, then add the same amount to the sample being tested.Correct for volume addition.

CHLORINE, TOTAL, UHR, continued

Interfering Substance	Interference Levels and Treatments
Bromine, Br ₂	Interferes at all levels
Chlorine Dioxide, ClO ₂	Interferes at all levels
Chloramines, organic	May interfere
Iodine, I ₂	Interferes at all levels
Manganese, oxidized (Mn ⁴⁺ , Mn ⁷⁺) or Chromium, oxidized (Cr ⁶⁺)	<ol style="list-style-type: none"> 1. Adjust sample pH to 6–7. 2. Add 2 drops Potassium Iodide (30 g/L) to a 5-mL sample. 3. Mix and wait 1 minute. 4. Add 2 drops of Sodium Arsenite (5 g/L) and mix. 5. Analyze the treated sample as described in the procedure. 6. Subtract the result from this test from the original analysis to obtain the correct chlorine concentration.
Ozone	Interferes at all levels
Peroxides	May interfere
Extreme sample pH or highly buffered samples	Adjust the sample pH to 6–7 with Sulfuric Acid or Sodium Hydroxide

Summary of Method

The range of analysis using the DPD method for total chlorine can be extended by adding more indicator in proportion to sample volume. Thus, a larger fill powder pillow of DPD Total Chlorine Reagent is added to a 5-mL sample portion.

The combined chlorine oxidizes iodide in the reagent to iodine. The iodine reacts with DPD (N,N-diethyl-p-phenylenediamine) along with free chlorine present in the sample to form a pink color which is proportional in intensity to the total chlorine concentration.

Instrument Setup

The following procedure will add this method as a new Hach program to a DR/850 or DR/890.

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**.

CHLORINE, TOTAL, UHR, continued

4. Press the **ENTER** key.
5. Enter “8138”, followed by **ENTER**.
6. Key the number in the “Enter” column corresponding to line number 1 on the display. Press **ENTER**. Repeat for lines 2–56 on the display.

Line Number	Enter	Line Number	Enter
1	12	29	0
2	24	30	0
3	73	31	0
4	0	32	0
5	0	33	0
6	0	34	0
7	0	35	0
8	62	36	0
9	55	37	0
10	23	38	0
11	88	39	0
12	64	40	0
13	113	41	0
14	242	42	0
15	18	43	0
16	0	44	110
17	0	45	0
18	0	46	0
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	236
27	0	55	0
28	0	56	255

CHLORINE, TOTAL, UHR, continued

REQUIRED REAGENTS

Description	Quantity Required		Cat. No.
	Per Test	Unit	
DPD Total Chlorine Reagent Powder Pillows, 25-mL.....	1.....	100/pkg.....	14064-99

REQUIRED APPARATUS

Sample Cell, 10-mL/1-cm.....	1.....	2/pkg.....	48643-02
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OPTIONAL REAGENTS

Chlorine Standard Solution, 2-mL Voluette® Ampule, 50–75 mg/L.....	20/pkg.....	14268-20
Potassium Iodide Solution, 30-g/L.....	100 mL MDB.....	343-32
Sodium Arsenite Solution, 5-g/L.....	100 mL MDB.....	1047-32
Sodium Hydroxide Standard Solution, 1.00 N.....	100 mL MDB.....	1045-32
Sulfuric Acid Standard Solution, 1.000 N.....	100 mL MDB.....	1270-32
Water, deionized.....	4 L.....	272-56

OPTIONAL APPARATUS

Ampule Breaker Kit.....	each.....	24846-00
Cylinder, graduated, 10-mL, mixing.....	each.....	20886-38
pH Meter, sens <i>ion</i> ™1, portable.....	each.....	51700-00
Pipet, TenSette®, 0.1 to 1.0 mL.....	each.....	19700-01
Pipet Tips, for 19700-01 TenSette Pipet.....	50/pkg.....	21856-96



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