IODINE

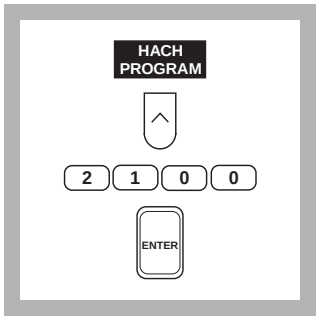
Method 8031 **DPD Method***

Powder Pillows or AccuVac® Ampuls **(0 to 7.00 mg/L)**

Scope and Application: *For testing dissolved iodine residual used as disinfectant in process water, treated water, estuary and seawater. The estimated detection limit for program numbers 2100 and 2110 is 0.04 mg/L I_2.*

* Adapted from Palin, A.T., *Inst. Water Eng., 21 (6)*, 537-547 (1967)

Using Powder Pillows



1. Press the soft key under ***HACH PROGRAM***.

Select the stored program number for iodine (I_2) by pressing **2100** with the numeric keys.

Press: **ENTER**

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

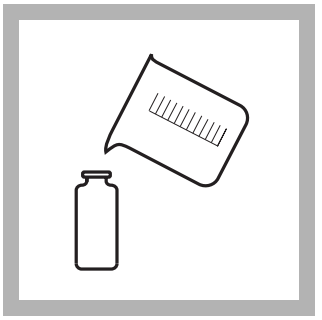
Note: *The Flow Cell and Sipper Modules can be used with this procedure if rinsed with deionized water immediately after analysis. Use a 25-mL sample and reagents with the Flow Cell Module.*

2. The display will show:

HACH PROGRAM: 2100 Iodine

The wavelength (λ), **530 nm**, is automatically selected.

Note: *For best results, determine a reagent blank for each new lot of reagent as follows. Prepare a reagent blank by repeating steps 3 through 8, using deionized water as the sample. Zero the instrument on deionized water by pressing the soft key under* ***ZERO****. Insert the reagent blank and the blank value will be displayed. Correct for the reagent blank by pressing the soft keys under* ***OPTIONS, (MORE)****, and then* ***BLANK:OFF****. Enter the reagent blank value and press* **ENTER***. Repeat for each new lot of reagent.*

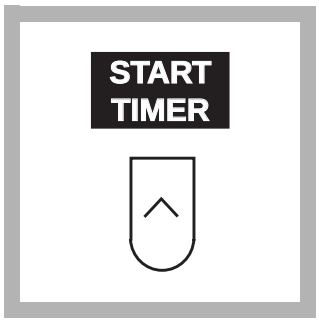


3. Fill a cell with 10 mL of sample.

Note: *For samples with extreme pH, see Interferences section.*

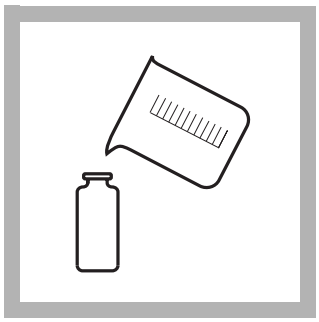
4. Add the contents of one DPD Total Chlorine Powder Pillow to the sample cell (the prepared sample). Swirl to mix.

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

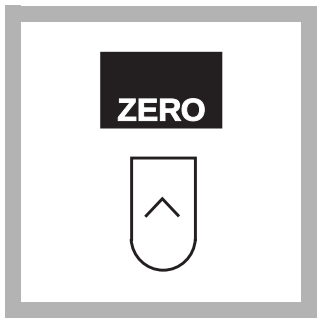


5. Press the soft key under ***START TIMER***.

A 3-minute reaction period will begin.



6. Fill a second sample cell with 10 mL of sample (the blank). Place it into the cell holder.



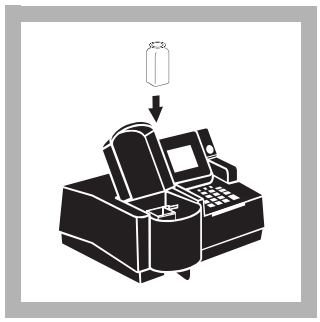
7. Press the soft key under ***ZERO***.

The display will show:

0.00 mg/L I_2

Note: *If you are using a reagent blank correction, the display will show the correction.*

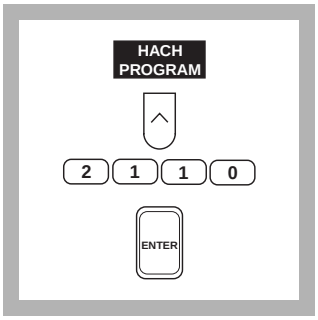
Note: *For alternate concentration units, press the soft key under* ***OPTIONS****. Then press the soft key under* ***UNITS*** *to scroll through the available options. Press* **ENTER** *to return to the read screen.*



8. Within 3 minutes after the timer beeps, place the prepared sample into the cell holder. Close the light shield. Result in mg/L I_2 (or chosen units) will be displayed.

Note: *If the sample temporarily turns yellow after reagent addition, or the display reads* **OVER!**, *dilute a fresh sample. Repeat the test. A slight loss of iodine may occur because of the dilution. Apply the appropriate dilution factor; see Section* 1.2.6 Sample Dilution Techniques.

Using AccuVac Ampuls



1. Press the soft key under ***HACH PROGRAM***.

Select the stored program number for iodine (I_2) by pressing **2110** with the numeric keys.

Press: **ENTER**

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

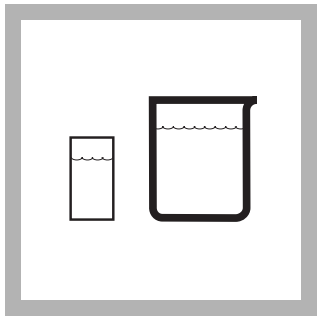
2. The display will show:

HACH PROGRAM: 2110
Iodine, AV

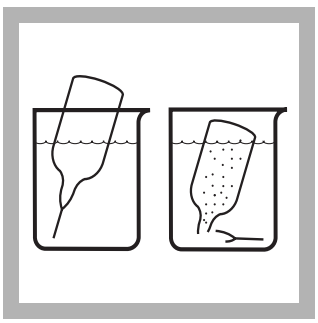
The wavelength (λ), **530 nm**, is automatically selected.

Note: *For best results determine a reagent blank for each new lot of reagent. See the note under Step 2 of the powder pillow method.*

3. Insert the AccuVac Ampul Adapter by sliding it under the thumb screw and into the alignment grooves. Fasten with the thumb screw.

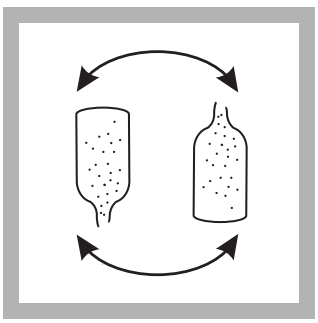


4. Fill a zeroing vial (the blank) with at least 10 mL of sample. Collect at least 40 mL of sample in a 50-mL beaker.



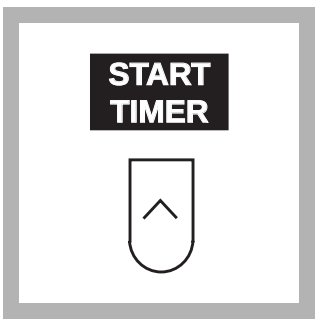
5. Fill a DPD Total Chlorine Reagent AccuVac Ampul with sample.

Note: *Keep the tip immersed while the ampul fills completely.*



6. Quickly invert the ampul several times to mix. Wipe off any liquid or fingerprints.

Note: *A pink color will form if iodine is present.*

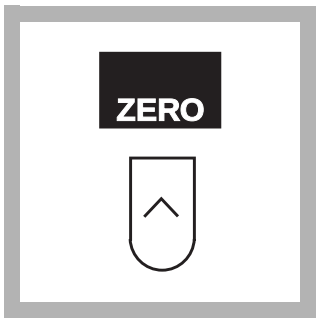


7. Press the soft key under ***START TIMER***.

A 3-minute reaction period will begin.

Note: *Perform steps 8–9 during the three minute period.*

8. Place the blank into the cell holder. Close the light shield.



9. Press the soft key under ***ZERO***.

The display will show:

0.00 mg/L I_2

Note: *If you are using a reagent blank correction, the display will show the correction.*

Note: *For alternate concentration units, press the soft key under* ***OPTIONS****. Then press the soft key under* ***UNITS*** *to scroll through the available options. Press* **ENTER** *to return to the read screen.*

10. Within 3 minutes after the timer beeps, place the AccuVac Ampul into the cell holder. Close the light shield. Result in mg/L iodine will be displayed.

Note: *If the sample temporarily turns yellow after sample addition, or the display shows* **OVER!** *dilute a fresh sample. Repeat the test. A slight loss of iodine may occur because of the dilution. Apply the appropriate dilution factor; see Section* 1.2.6 Sample Dilution Techniques*.*

Interferences

Table 1 Interfering Substances and Suggested Treatments

Interfering Substance	Interference Level and Treatment
Acidity	Greater than 150 mg/L $CaCO_3$. 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 (See Section *1.2.2 Correcting for Volume Additions*).
Alkalinity	Greater than 250 mg/L $CaCO_3$. 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 (See Section *1.2.2 Correcting for Volume Additions*).
Bromine	Interferes at all levels
Chlorine and chloramines	Causes a positive interference at all levels
Chlorine Dioxide	Interferes at all levels
Chloramines, organic	May interfere
Hardness	No effect at less than 1,000 mg/L as $CaCO_3$
Manganese, Oxidized (Mn^{4+}, Mn^{7+}) or Chromium, Oxidized (Cr^{6+})	**1.** Adjust sample pH to 6–7. **2.** Add 3 drops potassium iodide (30 g/L) to a 25-mL sample. **3.** Mix and wait 1 minute. **4.** Add 3 drops sodium arsenite (5 g/L) and mix. **5.** Analyze 10 mL of the treated sample as described in the procedure. **6.** Subtract the result from this test form the original analysis to obtain the correct iodine concentration.
Ozone	Interferes at all levels
Peroxides	May interfere
Extreme sample pH	Adjust to pH 6–7. See Section *1.3.1 pH Interference*.
Highly Buffered Samples	Adjust to pH 6–7. See Section *1.3.1 pH Interference.*

Sample Collection, Storage and Preservation

Collect samples in clean, dry glass containers. If sampling from a tap, allow the water to flow at least 5 minutes to ensure a representative sample. Avoid excessive agitation and exposure to sunlight when sampling. Allow several volumes of water to overflow the container and cap the container so there is not headspace above the sample. If sampling with a DR cell, rinse the cell several times with the sample, then carefully fill to the 10-mL mark. Proceed with the analysis immediately.

Accuracy Check

Standard Additions Method (Using Powder Pillows)

a. Leave the unspiked sample in the sample cell compartment. Verify that the units displayed are in mg/L. Select standard additions mode by pressing the soft keys under ***OPTIONS, (MORE)*** and then ***STD ADD***.

b. Press **ENTER** to accept the default sample volume (mL), 10.

c. Multiply the average chlorine concentration shown on the certificate enclosed with the LR Voluettes by 3.6 to obtain the equivalent

concentration of iodine. When prompted for the standard concentration, use the numeric keys to enter the calculated iodine value. Press **ENTER**.

d. Press the soft key under ***ENTRY DONE***.

e. Snap the neck off a LR Chlorine Voluette Ampule Standard, 20–30 mg/L Cl_2.

f. Use the TenSette Pipet to add 0.1 mL, 0.2 mL and 0.3 mL of standard, respectively to three 10-mL samples and mix each thoroughly.

g. Analyze each standard addition sample as described above. Accept the standard additions readings by pressing the soft key under ***READ*** each time. Each addition should reflect approximately 100% recovery.

h. After completing the sequence, the display will show the extrapolated concentration value and the "best-fit" line through the standard additions data points, accounting for matrix interferences.

i. See Section *1.4.1 Standard Additions* for more information.

Standard Additions Method (Using AccuVac Ampuls)

a. Leave the unspiked sample in the sample cell compartment. Verify that the units displayed are in mg/L. Select standard additions mode by pressing the soft keys under ***OPTIONS, (MORE)*** and then ***STD ADD***.

b. Press **ENTER** to accept the default sample volume (mL), 25.

c. Multiply the average chlorine concentration shown on the certificate enclosed with the LR Voluettes by 3.6 to obtain the equivalent concentration of iodine. When prompted for the standard concentration, use the numeric keys to enter the calculated iodine value. Press **ENTER**.

d. Press the soft key under ***ENTRY DONE***.

e. Snap the neck off a LR Chlorine Voluette Ampule Standard, 20–30 mg/L Cl_2.

f. Use graduated cylinder to measure 25 mL of sample into each of three 50-mL beakers. Use a TenSette Pipet to add 0.2, 0.4 and 0.6 mL of standard, respectively, to each of the 25-mL samples. Swirl gently to mix

g. Fill a DPD Total Chlorine AccuVac Ampul completely from each beaker and analyze each standard addition sample as described above. Accept the standard additions readings by pressing the soft key under ***READ*** each time. Each addition should reflect approximately 100% recovery.

h. After completing the sequence, the display will show the extrapolated concentration value and the "best-fit" line through the standard additions data points, accounting for matrix interferences.

i. See Section *1.4.1 Standard Additions* for more information.

Method Performance

Precision

Standard: 3.50 mg/L I_2

Program	95% Confidence Limits
2100	3.47–3.54 mg/L I_2
2110	3.47–3.54 mg/L I_2

For more information on determining precision data and method detection limits, refer to Section *1.5*.

Estimated Detection Limit

Program	EDL
2100	0.04 mg/L I_2
2110	0.04 mg/L I_2

For more information on derivation and use of Hach's estimated detection limit, see Section *1.5.2*. To determine a method detection limit (MDL) as defined by the 40 CFR part 136, appendix B, see Section *1.5.1*.

Sensitivity

Program Number: 2100

Portion of Curve:	ΔAbs	ΔConcentration
Entire Range	0.010	0.066 mg/L

Program Number: 2110

Portion of Curve:	ΔAbs	ΔConcentration
Entire Range	0.010	0.071 mg/L

See Section *1.5.3 Sensitivity Explained* for more information.

Summary of Method

Iodine reacts with DPD (N, N-diethyl-p-phenylenediamine) to form a pink color which is proportional to the total iodine concentration.

Safety

Good safety habits and laboratory techniques should be used throughout the procedure. Consult the *Material Safety Data Sheet* for information specific to the reagents used. For additional information, refer to Section *1*.

Pollution Prevention and Waste Management

Samples treated with sodium arsenite for manganese or chromium interferences will be hazardous wastes as regulated by the Federal RCRA for arsenic (D004). See Section *1* for more information on proper disposal of these materials.

REQUIRED REAGENTS AND STANDARDS (Using Powder Pillows)

Description	Quantity Required per test	Unit	Cat. No.
DPD Total Chlorine Reagent Powder Pillows	1 pillow	100/pkg	21056-69

REQUIRED REAGENTS AND STANDARDS (Using AccuVac Ampuls)

DPD Total Chlorine Reagent AccuVac Ampuls	1 ampul	25/pkg	25030-25

REQUIRED EQUIPMENT AND SUPPLIES (Using Powder Pillows)

DR/4000 1-inch Cell Adapter	1	each	48190-00

REQUIRED EQUIPMENT AND SUPPLIES (Using AccuVac Ampuls)

DR/4000 AccuVac Ampul Adapter	1	each	48187-00
Beaker, 50-mL	1	each	500-41
Sample Cell, 10-mL, with cap	1	each	21228-00

OPTIONAL REAGENTS AND STANDARDS

Chlorine Standard Solution, 1-mL Voluette Ampule, 20–30 mg/L Cl_2		20/pkg	26300-20
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 liters		272-56

OPTIONAL EQUIPMENT AND SUPPLIES

AccuVac Snapper		each	24052-00
Ampule Breaker Kit		each	21968-00
Cylinder, graduated, 25-mL, poly		each	1081-40
Graph Paper, linear		100/pkg	22313-00
pH Meter, *sens**ion**™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
DR/4000 Carousel Module Kit		each	48070-02
DR/4000 Flow Cell Module Kit, 1-inch		each	48070-04
DR/4000 Flow Cell Module Kit, 1-cm		each	48070-05
DR/4000 Sipper Module Kit, 1-inch		each	48090-03

* Contact Hach for larger sizes.

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