

ANEXO 6

**Procedimientos
HACH 4000/DR**

COLOR LIBRE RESIDUAL

HACH DR/4000
PROCEDURE



DR/4000 PROCEDURE

CHLORINE, TOTAL

✓ Method 8167

DPD Method*

Powder Pillows or AccuVae® Ampuls

(0 to 2.00 mg/L)

Scope and Application: For testing residual chlorine and chloramines in water, wastewater, estuary water and seawater, USEPA-accepted for reporting^{***} for drinking and wastewater analyses. The estimated detection limit for program numbers 1450 and 1460 is 0.01 mg/L Cl_2 .

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

^{***} Procedure is equivalent to USEPA method 330.5 and Standard Method 4500- Cl_2 G for drinking water and wastewater analyses.

Using Powder Pillows



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

Select the stored program number for free chlorine (Cl_2) by pressing **1450** 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 between samples. Use a 25-mL sample and reagents with the Flow Cell Module.



2. The display will show:

**HACH PROGRAM: 1450
Chlorine, F&T**

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 5, 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 sample cell with 10 mL of sample.

Note: For sample 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 the sample cell for 20 seconds to mix.

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

CHLORINE, TOTAL, continued



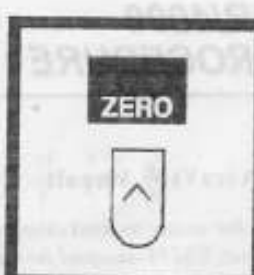
5. Press the soft key under **START TIMER**.

A 3-minute reaction period will begin.

Note: Perform steps 6 and 7 during this time period.



6. Fill another sample cell (the blank) with 10 mL of sample. Place it into the cell holder. Close the light shield.



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

The display will show:

0.00 mg/L Cl_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. Results in mg/L chlorine (or chosen units) will be displayed.

Note: If the sample temporarily turns yellow after reagent addition, or the display shows **OVER!**, dilute a fresh sample and repeat the test. A slight loss of chlorine may occur because of the dilution. Multiply the result by the appropriate dilution factor; see Section 1.2.8 Sample Dilution Techniques.

COLORURO

HACH DR/4000
PROCEDURE



DR/4000 PROCEDURE

CHLORIDE

Method 8113

Mercuric Thiocyanate Method*

(0 to 25.00 mg/L Cl^-)

Scope and Application: For water and wastewater.

The estimated detection limit for program number 1400 is 0.24 mg/L Cl^- .

* Adapted from Zall, et al., *Analytical Chemistry*, 28 (11) 1665 (1956)



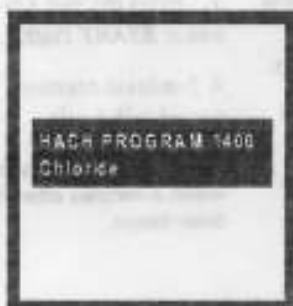
1. Press the soft key under **HACH PROGRAM**.

Select the stored program number for chloride (Cl^-) by pressing **1400** with the numeric keys.

Press: **ENTER**

Note: If samples cannot be analyzed immediately, see *Sample Collection, Storage and Preservation* following these steps.

Note: The Flow Cell and Sipper Modules cannot be used with this procedure.



2. The display will show:
HACH PROGRAM: 1400 Chloride

The wavelength (λ), 455 nm, is automatically selected.



3. Fill a sample cell with 25 mL of sample (the prepared sample).

Note: Filter turbid samples through moderately rapid filter paper before analysis.

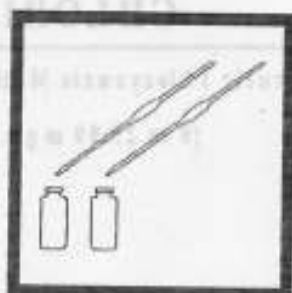
Note: For proof of accuracy, use a 10.0 mg/L chloride standard solution (see *Accuracy Check*) in place of the sample.

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 10, 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.



4. Fill another sample cell with 25 mL of deionized water (the blank).

CHLORIDE, continued



5. Pipet 2.0 mL of Mercuric Thiocyanate Solution into each sample cell. Swirl to mix.



6. Pipet 1.0 mL of Ferric Ion Solution into each sample cell. Swirl to mix.

Note: An orange color will develop if chloride is present.



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

A 2-minute reaction period will begin.

Note: Read the sample within 5 minutes after the timer beeps.



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 Cl⁻

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. Place the prepared sample into the cell holder. Close the light shield. Results in mg/L chloride (or chosen units) will be displayed.

COLOR

HACH DR/4000
PROCEDURE



DR/4000 PROCEDURE

COLOR

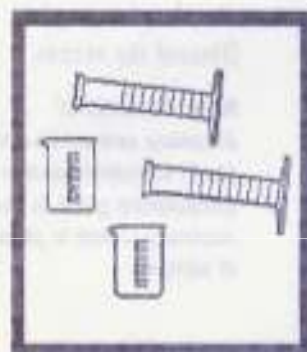
Method 10048

ADM1 Weighted Ordinate Method*

(0 to 250 units Pt-Co)

Scope and Application: For colored waters and wastewaters having color characteristics significantly different from platinum-cobalt standards, as well as to those similar in hue to the standards. Turbid samples must be filtered prior to analysis. The estimated detection limit for program number 1660 is 3 ADM1 (American Dye Manufacturers Institute) color value.

* Adapted from Allen, et. al., 1973, Determination of color of water and wastewater by means of ADM1 Color Values. *Proc. 26th Ind. Waste Conf.*, Purdue Univ., Ind., Int. Ser. No. 142,661



1. If the sample is not turbid, omit Steps 2-5. Pour two 100-mL aliquots of sample into separate beakers. Adjust the pH of one of the aliquots to 7.6; leave the other aliquot as is.

Note: Use 10 N sodium hydroxide or concentrated sulfuric acid to adjust the pH. Use 0.1 N acid or base near the end point.

Note: If sample cannot be analyzed immediately, see Sample Collection, Storage and Preservation following these steps.



2. Assemble the filtering apparatus (membrane filter, filter holder, filter flask and aspirator).



3. Rinse filter by pouring approximately 50 mL of original sample aliquot in the beaker through the filter. Discard the rinse.



4. Pour about 50 mL of original sample aliquot in the beaker through the filter. Label the flask "Original".

COLOR, continued

Repeat Steps 2-4 for adjusted sample



HACH PROGRAM:1660
Color:ADM



5. Repeat Steps 2-4 for the pH-adjusted sample. Label the flask "pH adjusted".

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

Select the stored program for ADM color value by pressing 1660 with the numeric keys.

Press: **ENTER**

Note: The Flow Cell and Sipper Modules can be used with this procedure. The Carousel Module cannot be used.

7. The display will show:

HACH PROGRAM:1660
Color:ADM

The starting wavelength (λ), 700 nm, is automatically selected.

8. Fill a 1-inch square sample cell with the pH-adjusted filtered sample (the sample). Discard the excess.

Note: For proof of accuracy, use a 100-unit Go-Pt standard solution (preparation given in the Accuracy Check in place of sample).



ZERO



9. Fill another sample cell with deionized water (the blank).

10. Place the blank into the cell holder and close the light shield.

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

Starting at 700 nm, the instrument will read the percent transmittance (%T) at 10 nm intervals until reaching 400 nm.

12. When prompted, place the sample in the cell holder and close the light shield.

COLOR, continued



13. Press the soft key under **START**.

Starting at 700 nm, the instrument will read the percent transmittance (%T) at 10-nm intervals until reaching 400 nm. Once finished, the instrument will display the ADM1 color value of the pH adjusted sample.

14. Repeat Steps 8-13 for the original filtered sample. For US EPA reporting, report both results.

Sample 5	
700 nm	99.9
600 nm	99.9
500 nm	99.9
400 nm	99.9
ADM1	1.0

HIERRO

HACH DR/4000
PROCEDURE



DR/4000 PROCEDURE

IRON, TOTAL

✓ Method 8008

FerroVer Method[†]

Powder Pillows or AccuVac[®] Ampuls

(0 to 3,000 mg/L)

Scope and Application: For water, wastewater and seawater, digestion is required for determining total iron; USEPA approved for reporting wastewater analysis.[‡] See Section 2 for digestion procedure. The estimated detection limits for program numbers 2163 and 2170 are 0.008 and 0.007 mg/L Fe, respectively.

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

[‡] Federal Register, June 27, 1989; 45 (126:43459)

Using Powder Pillows



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

Select the stored program number for iron (Fe), FerroVer, method by pressing 2165 with the numeric keys.

Press: **ENTER**

Note: If samples cannot be analyzed immediately, see Sample Collection, Storage and Preservation following these steps. Adjust pH of preserved samples before analysis.

Note: The Flow Cell and Sipper Modules can be used with this procedure. Use a 25-mL sample and reagents with the Flow Cell Module.



2. The display will show: **HACH PROGRAM: 2165 Iron, FerroVer**

The wavelength (λ), 510 nm, is automatically selected.

Note: Determination of total iron requires digestion. See Section 2.

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 clean sample cell with 10 mL of sample.

Note: For proof of accuracy, use a 1.0 mg/L iron standard solution (preparation given in the Accuracy Check section) in place of the sample.

Note: For turbid samples, or non-preserved samples with extreme pH, see the Interferences section.



4. Add the contents of one FerroVer Iron Reagent Powder Pillow for 10-mL sample to the sample cell (the prepared sample). Swirl to mix.

Note: An orange color will form if iron is present.

Note: Accuracy is not affected by undissolved powder.

IRON, TOTAL, continued



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

A 3-minute reaction period will begin.

Note: Samples containing visible rust should be allowed to react for at least 3 minutes.



6. Fill another sample cell (the blank) with 10 mL of sample.



7. When the timer beeps, place the blank into the cell holder. Close the light shield.



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

The display will show:

0.000 mg/L Fe

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

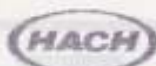
*Note: For alternate concentration units, press the soft key under **OPTIONS**. Then press the soft key under **UNITS** to scroll through the available options. Press **ENTER** to return to the read screen.*



9. Place the prepared sample into the cell holder. Close the light shield. Results in mg/L iron (or chosen units) will be displayed.

NITRATOS

**HACH DR/4000
PROCEDURE**



DR/4000 PROCEDURE

NITRATE

Method 8171

Cadmium Reduction Method

Powder Pillows or AccuVae® Ampuls

MR, (0 to 5.0 mg/L NO_3^- -N)

Scope and Application: For water, wastewater and seawater.

The estimated detection limit for program numbers 2520 and 2523 are 0.1 and 0.1 mg/L NO_3^- -N, respectively.

Using Powder Pillows



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

Select the stored program number for mid range nitrate by pressing **2520** with the numeric keys.

Press: **ENTER**

Note: If samples cannot be analyzed immediately, see *Sample Collection, Storage and Preservation* following these steps. Adjust the pH of preserved samples before analysis.



2. The display will show: **HACH PROGRAM: 2520**
Nitrate MR

The wavelength (λ), **400 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 9, 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 sample cell with 10 mL of sample.



4. Add the contents of one NitraVer 5 Nitrate Reagent Powder Pillow (to the prepared sample). Stopper.



NITRATE, continued



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

Shake the cell vigorously until the timer beeps in one minute.

Note: A deposit of unoxidized metal will remain after the NitraVer 5 dissolves. The deposit will not affect results.

Note: Shaking time and technique influence color development. For most accurate results, make successive tests on a 1.00 mg/L Nitrate Nitrogen Standard solution (listed under **OPTIONAL REAGENTS AND STANDARDS**). Adjust shaking times to obtain the correct result.



6. When the timer beeps, press the soft key under **START TIMER**.

A 5-minute reaction period will begin.

Note: An amber color will develop if nitrate nitrogen is present.



7. When the timer beeps, fill a second sample cell with 10 mL of sample (the blank). Place the blank into the cell holder.



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

The display will show:

0.0 mg/L $\text{NO}_3^- \text{--N}$

Note: If you have entered 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.



9. Place the prepared sample into the cell holder. Close the light shield. The result in mg/L nitrate nitrogen ($\text{NO}_3^- \text{--N}$) will be displayed.

Note: Measure sample within two minutes after timer beeps.

Note: The result can be expressed as mg/L nitrate (NO_3^-). Press the soft key under **OPTIONS** and then **FORM** to scroll through the available options.

Note: Rinse the sample cell immediately after use to remove all cadmium particles. Retain the spent sample for proper hazardous waste disposal for cadmium.

NITRITOS

HACH DR/4000
PROCEDURE



DR/4000 PROCEDURE

Method 8153

Powder Pillows

Scope and Application: For water and wastewater.
The estimated detection limit for program number 2600 is 1 mg/L NO_2^- .

* Adapted from McAlpine, R. and Sogatz, B., *Qualitative Chemical Analysis*, New York, 476, 575 (1933).

NITRITE

Ferric Sulfate Method[†]

HR, (1 in 250 mg/L NO_2^-)



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

Select the stored program number for high range nitrite by pressing **2600** with the numeric keys.

Press: **ENTER**

Note: If samples cannot be analyzed immediately, see *Sample Collection, Storage and Preservation* following these steps.

Note: The Flow Cell and Sipper Modules cannot be used with this procedure.



2. The display will show:
HACH PROGRAM: 2600
Nitrite, HR

The wavelength (λ), **585 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 key under **OPTIONS, (MORE)**, and then **BLANK/OFF**. Enter the reagent blank value and press **ENTER**. Repeat for each new lot of reagent.



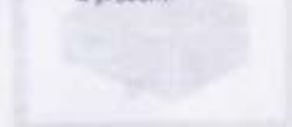
3. Fill a sample cell with 10 mL of sample.

Note: For proof of accuracy, use a 200 mg/L nitrite solution in place of the sample. See the *Accuracy Check* section for preparation.



4. Add the contents of one NiriVer 2 Nitrite Reagent Powder Pillow, stopper and shake to dissolve (the prepared sample).

Note: A greenish-brown color will develop if nitrite is present.



NITRITE, continued



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

A 10-minute reaction period will begin. It is critical to leave the sample undisturbed on a flat surface for the reaction period or low results may occur.



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



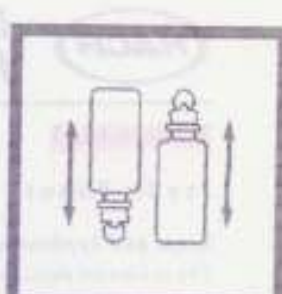
7. Press the soft key under **ZERO**.

The display will show:

0 mg/L NO_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. Gently invert the prepared sample twice. Remove the stopper.

Note: Avoid excessive mixing or low results may occur.



9. Place the prepared sample into the cell holder. Close the light shield. Results in mg/L NO_2^- (or chosen units) will be displayed.

*Note: The results can be expressed as nitrite nitrogen ($\text{NO}_2^- \text{--N}$) or as sodium nitrite (NaNO_2). Press the soft keys under **METHOD OPTIONS** and then **FORM** to scroll through the available options.*

TURBIDEZ

HACH DR/4000
PROCEDURE



DR/4000 PROCEDURE

TURBIDITY

Method 10047

Attenuated Radiation Method (Direct Reading)
(0 to 5000 Formazin Attenuation Units*)

Scope and Application: For testing turbidity in water, wastewater, estuary water, seawater and industrial process water. Results may not be used for compliance reporting. The estimated detection limit for program number 3750 is 14 Formazin Attenuation Units (FAUs).

* A Formazin Attenuation Unit (FAU) is equivalent to a Nephelometric Turbidity Unit (NTU).



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

Select the stored program number for turbidity in FAUs by pressing 3750 with the numeric keys.

Press: **ENTER**

Note: If samples cannot be analyzed immediately, see Sample Collection, Preservation and Storage following these steps.

Note: The Flow Cell and Sipper Modules cannot be used with this procedure.

Note: Results are given in FAU (Formazin Attenuation Units), not Nephelometric Turbidity Units. An FAU is equivalent to a NTU when measuring formazin. They are not necessarily equivalent when measuring samples or other types of standards.



2. The display will show:

**HACH PROGRAM: 3750
Turbidity, Absorb**

The wavelength (λ), 860 nm, is automatically selected.



3. Use a set of matched sample cells. Fill one of the clean stoppered sample cells to the 10-mL mark with deionized water (the blank). Stopper.

Note: For highly colored samples, filter a portion of the sample and use it in place of the deionized water. See **OPTIONAL EQUIPMENT AND SUPPLIES** for labware.

Note: For colored samples, see the **Interferences** section.



4. Rinse the other matched sample cell with sample. Then fill the sample cell to the 10-mL mark with sample. Stopper (this is the prepared sample).

TURBIDITY, continued



5. Wipe the sides of both sample cells using a clean, soft cloth.

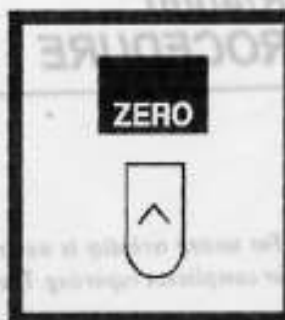
Note: Handle the sample cells by grasping the top of the cell.

Note: Apply a small amount of silicone oil to the outside of the sample cells. This minimizes the effects of surface defects on the measurement.



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

Note: Avoid disturbing the liquid in the sample cell.



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

The display will show:

0 FAU



8. Gently invert the prepared sample several times. Immediately place it into the cell holder. Close the light shield. Results in FAU turbidity will be displayed.

Note: Do not shake the sample. Shaking causes air bubbles, which will cause falsely high turbidity readings.