IRON, Ferrous

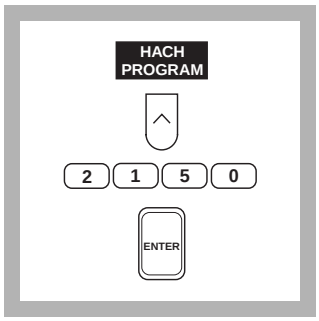
Method 8146 **1,10 Phenanthroline Method***

Powder Pillows or AccuVac® Ampuls **(0 to 3.000 mg/L)**

Scope and Application: *For water, wastewater and seawater. The estimated detection limit for program numbers 2150 and 2155 are 0.008 and 0.007 mg/L Fe^{2+}, respectively.*

* Adapted from *Standard Methods for the Examination of Water and Wastewater,* 15th ed. 201 (1980)

Using Powder Pillows



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

Select the stored program number for ferrous iron (Fe^{2+}), by pressing **2150** with the numeric keys.

Press: **ENTER**

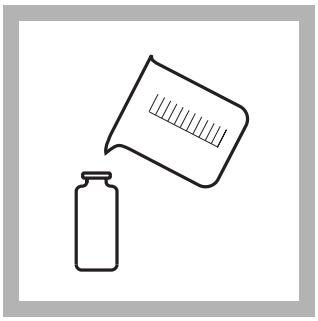
Note: *Analyze samples as soon as possible to prevent air oxidation of ferrous iron to ferric iron, which is not determined. See Sample Collection, Storage and Preservation following these steps.*

Note: *The Flow Cell and Sipper Modules can be used with this procedure.*

2. The display will show:

HACH PROGRAM: 2150
Iron, Ferrous

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



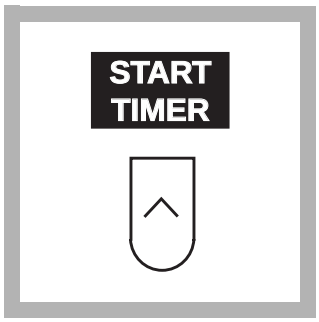
3. Fill a clean sample cell with 25 mL of sample.

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

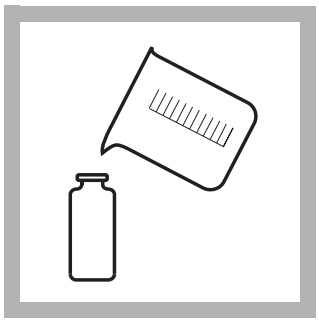
4. Add the contents of one Ferrous Iron Reagent Powder Pillow to the sample cell (the prepared sample). Swirl to mix.

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

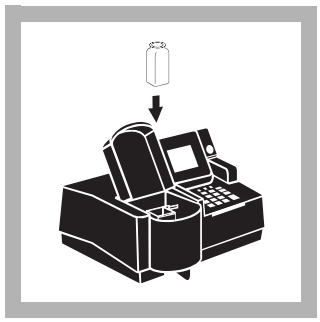


5. Press the soft key under ***START TIMER***.

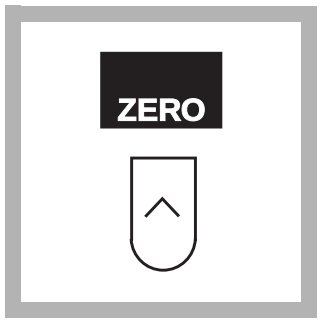
A 3-minute reaction period will begin.



6. Fill a second sample cell with 25 mL of sample (the blank).



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



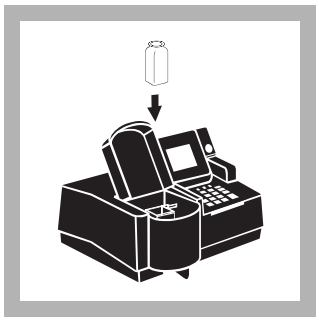
8. Press the soft key under ***ZERO***.

The display will show:

0.000 mg/L Fe^{2+}

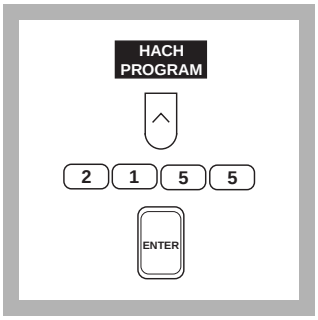
Note: *If you are using a reagent blank correction, the display will show the correction.*

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.*



9. Place the prepared sample into the cell holder. Close the light shield. The results in mg/L Fe^{2+} (or chosen units) will be displayed.

Using AccuVac Ampuls



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

Select the stored program number for ferrous iron (Fe^{2+}) by pressing **2155** with the numeric keys.

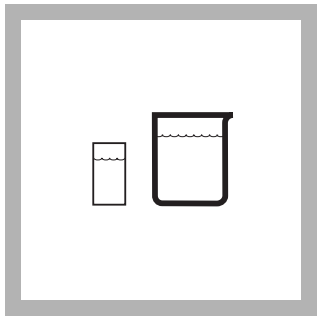
Press: **ENTER**

Note: *Analyze samples as soon as possible to prevent air oxidation of ferrous iron to ferric iron, which is not determined. See Sample Collection, Storage and Preservation following these steps.*

2. The display will show:

HACH PROGRAM: 2155
Iron, Ferrous AV

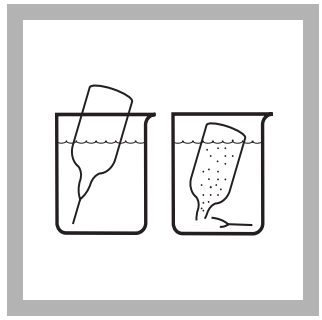
The wavelength (λ), **510 nm**, is automatically selected.



3. 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.

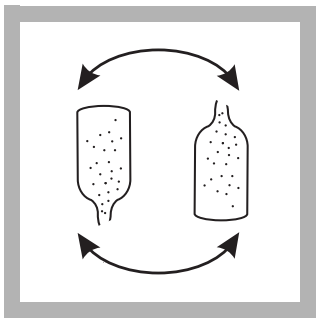
Note: *For proof of accuracy, a 1.0-mg/L ferrous iron standard solution (preparation given in the Accuracy Check section) can be used in place of the sample.*

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.*



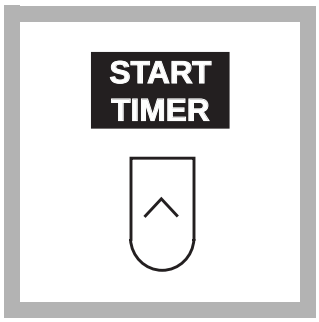
4. Fill a Ferrous Iron AccuVac Ampul with sample.

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



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

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



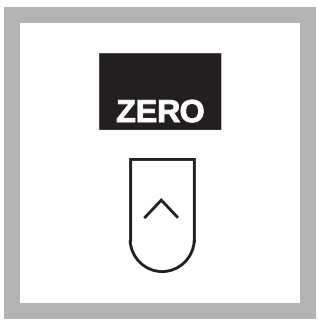
6. Press the soft key under ***START TIMER***.

A 3-minute reaction period will begin.

Note: *Complete Step 7 during the reaction period.*

7. Insert the AccuVac Ampul Adapter into the sample cell module by sliding it under the thumb screw and into the alignment grooves. Fasten with the thumb screw.

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



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

The display will show:

0.000 mg/L Fe^{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. Place the AccuVac Ampul into the cell holder. Close the light shield. Results in mg/L Fe^{2+} (or chosen units) will be displayed.

Sample Collection, Storage and Preservation

Collect samples in plastic or glass bottles. Analyze samples as soon as possible after collection.

Accuracy Check

Standard Solution Method

Prepare a ferrous iron stock solution (100-mg/L Fe^{2+}) by dissolving 0.7022 grams of Ferrous Ammonium Sulfate, hexahydrate, in deionized water. Dilute to one liter in a Class A volumetric flask. In a 100-mL Class A volumetric flask, dilute 1.00 mL of this solution to 100 mL with deionized water to make a 1.0-mg/L standard solution. Prepare this solution immediately before use. Perform the iron procedure as described above.

To adjust the calibration curve using the reading obtained with the 1.0 mg/L Fe^{2+} Standard Solution, press the soft keys under ***METHOD OPTIONS, (MORE)*** then ***STD: OFF***. Press **ENTER** to accept the default concentration, the value of which will depend on the selected units. If an alternate concentration is used, enter the actual concentration and press **ENTER** to return to the read screen. See Section *1.5.5 Adjusting the Standard Curve* for more information.

Method Performance

Precision

Standard: 1.000 mg/L Fe

Program	95% Confidence Limits
2150	0.997–1.003 mg/L Fe
2155	0.997–1.003 mg/L Fe

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

Estimated Detection Limit

Program	EDL
2150	0.008 mg/L Fe
2155	0.007 mg/L Fe

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: 2150

Portion of Curve:	ΔAbs	ΔConcentration
Entire Range	0.010	0.0210 mg/L

Program Number: 2155

Portion of Curve:	ΔAbs	ΔConcentration
Entire Range	0.010	0.0226 mg/L

See Section *1.5.3 Sensitivity Explained* for more information.

Calibration Standard Preparation

Preparing ferrous standards is difficult. These standards are very unstable and degrade rapidly. Only a trained chemist should prepare these standards.

To perform an ferrous iron calibration using the 1,10 phenanthroline method, prepare calibration standards containing 0.50, 1.00, 2.00, and 3.00 mg/L ferrous iron as follows:

a. Prepare a 100 mg/L ferrous iron stock solution by dissolving 0.7022 grams of Ferrous Ammonium Sulfate, hexahydrate, in deionized water. Dilute to one liter in a Class A volumetric flask. Stopper and invert several times to mix. Prepare this solution just before use.

b. Into four different 100-mL Class A volumetric flasks, pipet 0.50, 1.00, 2.00 and 3.00 mL of the 100-mg/L ferrous iron stock solution. Dilute each flask to volume with deionized water. Stopper and invert each flask to mix. Prepare these standards just before use.

c. Using the 1,10 phenanthroline method and the calibration procedure described in the *User-Entered Programs* section of the *DR/4000 Spectrophotometer Instrument Manual*, generate a calibration curve from the standards prepared above.

Summary of Method

The 1,10 phenanthroline indicator in the Ferrous Iron Reagent reacts with ferrous iron in the sample to form an orange color in proportion to the iron concentration. Ferric iron does not react. The ferric iron (Fe^{3+}) concentration can be determined by subtracting the ferrous iron concentration from the results of a total iron test.

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

For information on pollution prevention and waste management, refer to Section *1*.

REQUIRED REAGENTS AND STANDARDS (Using Powder Pillows)

Description	Quantity Required per test	Unit	Cat. No.
Ferrous Iron Reagent Powder Pillows	1 pillow	100/pkg	1037-69

REQUIRED REAGENTS AND STANDARDS (Using AccuVac Ampuls)

Ferrous Iron Reagent AccuVac Ampuls	1 ampul	25/pkg	25140-25

REQUIRED EQUIPMENT AND SUPPLIES (Using Powder Pillows)

Clippers, for opening powder pillows	1	each	968-00
DR/4000 1-Inch Cell Adapter	1	each	48190-00

REQUIRED EQUIPMENT AND SUPPLIES (Using AccuVac Ampuls)

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

OPTIONAL REAGENTS AND STANDARDS

Ferrous Ammonium Sulfate, hexahydrate, ACS	113 g	11256-14
Water, deionized	4 liters	272-56

OPTIONAL EQUIPMENT AND SUPPLIES

AccuVac Snapper	each	24052-00
Balance, electronic, 110 VAC	each	26104-00
Balance, electronic, 220 VAC	each	26104-02
Clippers, shears, 7 ¼-inch	each	23694-00
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	48070-03
Flask, volumetric, 100-mL, Class A	each	14574-42
Flask, volumetric, 1000-mL, Class A, with stopper	each	14574-53
pH Meter, *sens**ion**™1*, portable	each	51700-00
pH Paper, pH 1.0 to 11.0	5 rolls/pkg	391-33
Pipet, volumetric, 0.50-mL, Class A	each	14515-34
Pipet, volumetric, 1.00-mL, Class A	each	14515-35
Pipet, volumetric, 2.00-mL, Class A	each	14515-36
Pipet, volumetric, 3.00-mL, Class A	each	14515-03
Pipet Filler, safety bulb	each	14651-00

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