Iron, Total

✔Method 8008 — FerroVer® Method*

Powder Pillows or AccuVac® Ampuls — (0.02 to 3.00 mg/L)

Scope and Application: For water, wastewater, and seawater; digestion is required for determining total iron; USEPA approved for reporting wastewater analysis**

* Adapted from *Standard Methods for the Examination of Water and Wastewater*
** *Federal Register*, June 27, 1980; 45 (126:43459)

Tips and Techniques

- Digestion is required for determining total iron for EPA reporting purposes. See *Section 4* on page *43* for the digestion procedure.
- For more accurate results, determine a reagent blank value for each new lot of reagent. Follow the procedure using deionized water in place of the sample. Subtract the reagent blank value from the final results or perform a reagent blank adjust. See the instrument manual for more information on *Running a Reagent Blank*.
- After adding reagent, an orange color will form if iron is present.
- Accuracy is not affected by undissolved powder.

Powder Pillows — Method 8008

1. Touch

Hach Programs.

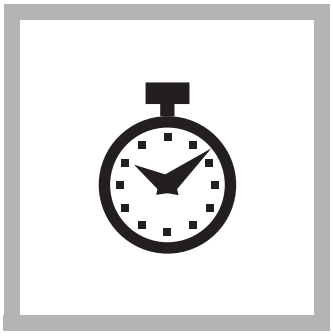
Select program

265 Iron, FerroVer.

Touch **Start**.

2. Fill a clean, round sample cell with 10 mL of sample.

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



4. Touch the timer icon.

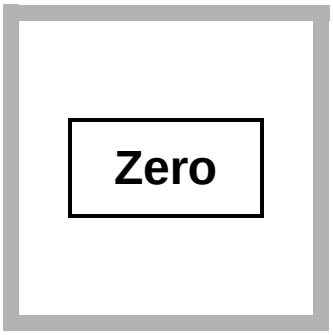
Touch **OK**.

A three-minute reaction period will begin.

(Allow samples that contain rust to react for at least 5 minutes.)

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

6. When the timer beeps, place the blank into the cell holder.



7. Touch **Zero**.

The display will show:

0.00 mg/L Fe

8. Place the prepared sample into the cell holder.

Results will appear in mg/L Fe.

AccuVac Ampul — Method 8008

1. Touch

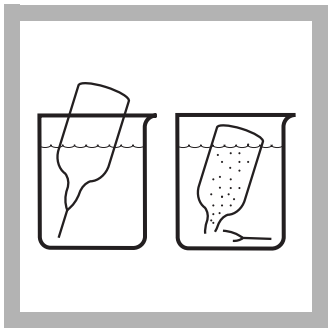
Hach Programs.

Select program

267 Iron, FerroVer AV.

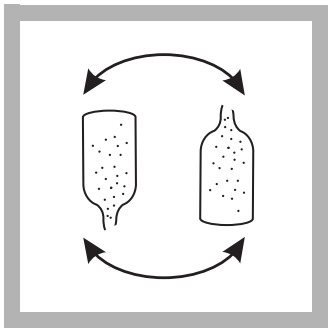
Touch **Start**.

2. Fill a sample cell with 25 mL of sample. Collect at least 40 mL of sample in a 50-mL beaker.

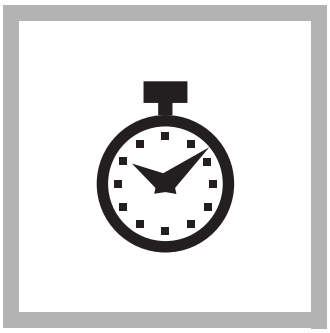


3. Fill a FerroVer Iron AccuVac® Ampul with sample.

Keep the tip immersed while the ampule fills completely.



4. Quickly invert the ampule several times to mix. Wipe off any liquid or fingerprints.



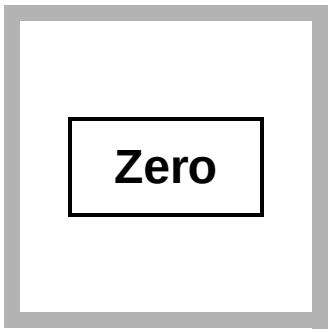
5. Touch the timer icon.

Touch **OK**.

A three-minute reaction period will begin.

(Samples that contain rust should react for at least 5 minutes.)

6. When the timer beeps, place the blank into the cell holder.



7. Touch **Zero**.

The display will show:

0.00 mg/L Fe

8. Place the AccuVac Ampul into the cell holder.

Results will appear in mg/L Fe.

Interferences

Interfering Substance	Interference Levels and Treatments
Calcium, Ca^{2+}	No effect at less than 10,000 mg/L as $CaCO_3$.
Chloride, Cl^-	No effect at less than 185,000 mg/L.
Copper, Cu^{2+}	No effect. Masking agent is contained in FerroVer Reagent.
High Iron Levels	Inhibit color development. Dilute sample and re-test to verify results.
Iron Oxide	Requires mild, vigorous or Digesdahl digestion. After digestion, adjust sample to pH 3–5 with sodium hydroxide (Cat. No. 2450-32), then analyze.
Magnesium	No effect at 100,000 mg/L as calcium carbonate.
Molybdate Molybdenum	No effect at 50 mg/L as Mo.
High Sulfide Levels, S^{2-}	**1.** Treat in fume hood or well-ventilated area. Add 5 mL hydrochloric acid, ACS (Cat. No. 134-49) to 100 mL sample in a 250-mL Erlenmeyer flask. Boil 20 minutes. **2.** Cool. Adjust pH to 3–5 with Sodium Hydroxide (Cat. No. 2450-32). Readjust volume to 100 mL with deionized water. **3.** Analyze.
Turbidity	**1.** Add 0.1 g scoop of RoVer® Rust Remover (Cat. No. 300-01) to the blank. Swirl to mix. **2.** Zero the instrument with this blank. **3.** If sample remains turbid, add three 0.2 g scoops of RoVer to a 75-mL sample. Let stand 5 minutes. **4.** Filter through a Glass Membrane Filter (Cat. No. 2530-00) and Filter Holder (Cat No. 2340-00). **5.** Use filtered sample in steps 2 and 5.
Extreme Sample pH	Adjust pH to 3–5. See *Section 3.3 Interferences* on page *30.*
Highly Buffered Samples	Adjust pH to 3–5. See *Section 3.3 Interferences* on page *30.*

Sample Collection, Storage and Preservation

Collect samples in acid-cleaned glass or plastic containers. No acid addition is necessary if analyzing the sample immediately. To preserve samples, adjust the pH to 2 or less with concentrated nitric acid (about 2 mL per liter) (Cat. No. 152-49). Preserved samples may be stored up to six months at room temperature. Before analysis, adjust the pH to between 3 and 5 with 5.0 N Sodium Hydroxide Standard Solution (Cat. No. 2450-32). Correct the test result for volume additions; see *Section 3.1.3 Correcting for Volume Additions* on page *23*.

If only dissolved iron is to be determined, filter the sample before acid addition.

Accuracy Check

Standard Additions Method (Sample Spike)

1. After reading test results, leave the sample cell (unspiked sample) in the instrument.
2. Touch **Options**. Touch **Standard Additions**. A summary of the standard additions procedure will appear.
3. Touch **OK** to accept the default values for standard concentration, sample volume, and spike volumes. Touch **Edit** to change these values. After values are accepted, the unspiked sample reading will appear in the top row. See *Standard Additions* in the instrument manual for more information.
4. Snap the neck off an Iron Voluette Ampule Standard, 50-mg/L.
5. Prepare a 0.1 mL sample spike by adding 0.1 mL of standard to the unspiked sample. Touch the timer icon. After the timer beeps, read the result.
6. Prepare a 0.2 mL sample spike by adding 0.1 mL of standard to the 0.1 mL sample spike. Touch the timer icon. After the timer beeps, read the result.
7. Prepare a 0.3 mL sample spike by adding 0.1 mL of standard to the 0.2 mL sample spike. Touch the timer icon. After the timer beeps, read the result. Each addition should reflect approximately 100% recovery.

Note: *For AccuVac Ampuls, fill three mixing cylinders (Cat. No. 1896-41) with 50-mL of sample and spike with 0.2 mL, 0.4 mL, and 0.6 mL of standard. Transfer 40 mL from each of the three mixing cylinders to three 50-mL beakers (Cat. No. 500-41H). Analyze each standard addition sample as described in the procedure above. Accept each standard additions reading by touching* **Read***. Each addition should reflect approximately 100% recovery.*

8. After completing the sequence, touch **Graph** to view the best-fit line through the standard additions data points, accounting for matrix interferences. Touch **View: Fit**, then select **Ideal Line** and touch **OK** to view the relationship between the sample spikes and the “Ideal Line” of 100% recovery.

See *Section 3.2.2 Standard Additions* on page *26* for more information.

Standard Solution Method

1. Prepare a 1.00-mg/L Fe standard solution by pipetting 1.00 mL of Iron Standard Solution, 100-mg/L, into a 100-mL volumetric flask. Dilute to the mark with deionized water. Stopper and invert to mix. Prepare this solution daily. Perform the iron procedure as described above.
2. To adjust the calibration curve using the reading obtained with the 1.00 mg/L Standard Solution, touch **Options** on the current program menu. Touch **Standard Adjust**.

3. Touch **On**. Touch **Adjust** to accept the displayed concentration. If an alternate concentration is used, touch the number in the box to enter the actual concentration, then touch **OK**. Touch **Adjust**.

See *Section 3.2.4 Adjusting the Standard Curve* on page *29* for more information.

Method Performance

Precision

Standard: 1.000 mg/L Fe

Program	95% Confidence Limits of Distribution
265	0.989–1.011 mg/L Fe
267	0.977–1.023 mg/L Fe

See *Section 3.4.3 Precision* on page *33* for more information, or if the standard concentration did not fall within the specified range.

Sensitivity

Program	Portion of Curve	ΔAbs	ΔConcentration
265	Entire range	0.010	0.022 mg/L Fe
267	Entire range	0.010	0.023 mg/L Fe

See *Section 3.4.5 Sensitivity* on page *34* for more information.

Summary of Method

FerroVer Iron Reagent converts all soluble iron and most insoluble forms of iron in the sample to soluble ferrous iron. The ferrous iron reacts with the 1,10 phenanthroline indicator in the reagent to form an orange color in proportion to the iron concentration. Test results are measured at 510 nm.

Required Reagents

Description	Quantity Required per test	Unit	Cat. No.
FerroVer® Iron Reagent Powder Pillows (for 10-mL sample)	1 pillow	100/pkg	21057-69
or			
FerroVer® Iron Reagent AccuVac® Ampuls	1 ampul	25/pkg	25070-25

Required Apparatus

Sample Cells, 10-mL, w/cap	2	6/pkg	24276-06
Beaker, 50-mL	1	each	500-41H

Required Standards

Iron Standard Solution, 100-mg/L		100 mL	14175-42
Iron Standard Solution, 10-mL Voluette® Ampule, 50-mg/L as Fe		16/pkg	14254-10
Metals Drinking Water Standard, LR for Cu, Fe, Mn		500 mL	28337-49
Metals Drinking Water Standard, HR for Cu, Fe, Mn		500 mL	28336-49
Water, deionized		4 liters	272-56

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