Iron, Total

★Method 8008 — FerroVer® Method[1]

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[2]

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

[2] *Federal Register*, June 27, 1980; 45 (126:43459)

Test Preparation

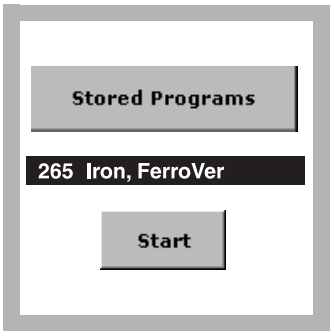
Before starting the test:

Digestion is required for determining total iron for EPA reporting purposes.
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 user manual for more information.

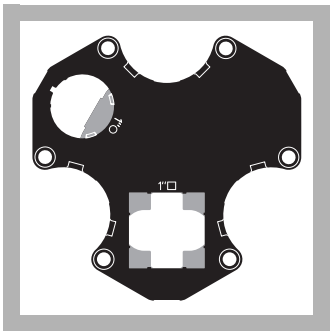
Collect the following items:	**Quantity**
FerroVer® Iron Reagent Powder Pillow or AccuVac® Ampul	1
Sample Cells,1-inch square, 10 mL	2
Beaker, 50-mL	1

Note: *Reorder information for consumables and replacement items is on page 6.*

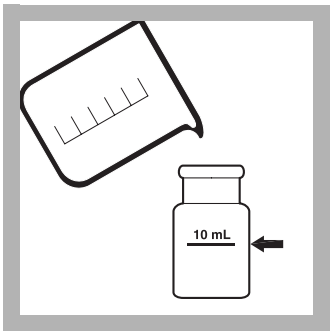
Powder Pillows — Method 8008



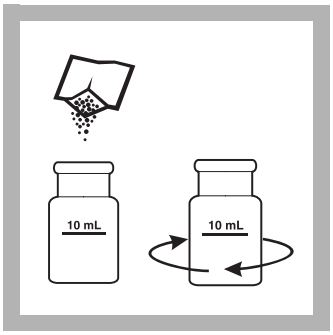
1. Select the test.



2. Insert the Multi-cell Adapter with the 1-inch square cell holder facing the user.



3. Prepared Sample: Fill a clean square sample cell with 10 mL of sample.



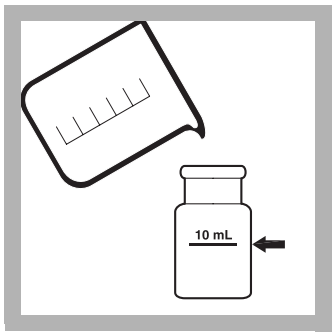
4. Add the contents of one FerroVer Iron Reagent Powder Pillow to the sample cell. Swirl to mix.

An orange color will form, if iron is present

5. Press **TIMER**>**OK**.

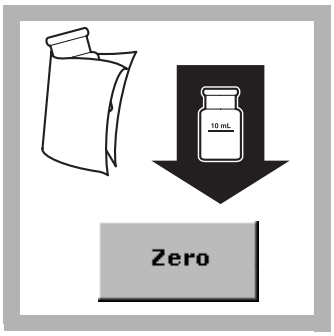
A three-minute reaction period will begin.

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



6. Blank Preparation:

Fill a second square sample cell with 10 mL of sample.



7. When the timer expires, insert the blank into the cell holder with the fill line facing the user.

Press **ZERO**.

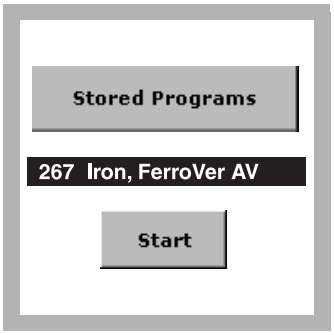
The display will show:

0.00 mg/L Fe

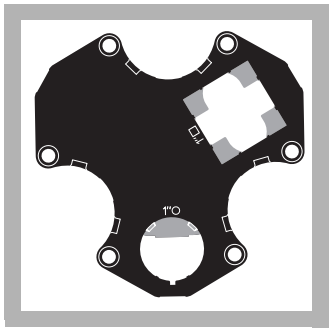
8. Place the prepared sample into the cell holder with the fill line facing the user.

Results are in mg/L Fe.

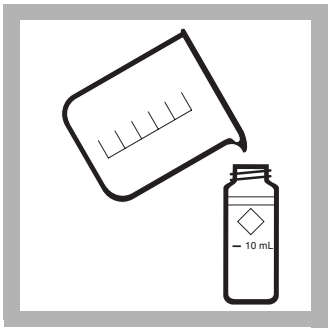
AccuVac Ampul — Method 8008



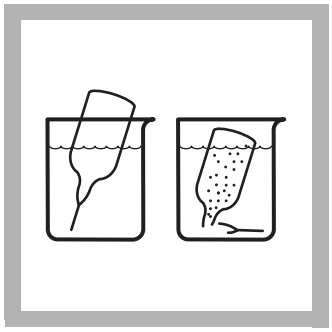
1. Select the test.



2. Insert the Multi-cell Adapter with the 25-mm round cell holder facing the user.



3. Blank Preparation: Fill a round sample cell with 10 mL of sample.



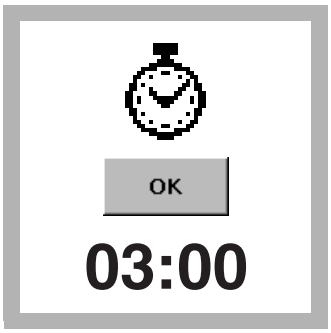
4. Prepared Sample: Collect at least 40 mL of sample in a 50-mL beaker.

Fill a FerroVer Iron AccuVac® Ampul with sample.

Keep the tip immersed while the ampule fills completely.

5. Quickly invert the ampule several times to mix.

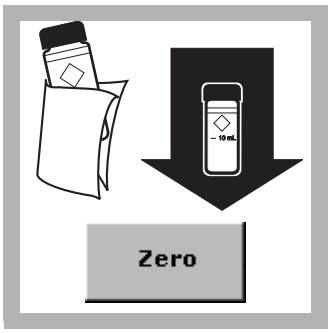
Wipe off all liquid and fingerprints.



6. Press **TIMER**>**OK**.

A three-minute reaction period will begin.

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



7. When the timer expires, insert the blank into the cell holder.

Press **ZERO**.

The display will show:

0.00 mg/L Fe

8. Insert the AccuVac Ampul into the cell holder.

Results are in mg/L Fe.

Interferences

Table 1 Interfering Substances and Levels

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, 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[1], ACS to 100 mL sample in a 250-mL Erlenmeyer flask. Boil 20 minutes. **2.** Cool. Adjust pH to 3–5 with Sodium Hydroxide[1]. Readjust volume to 100 mL with deionized water. **3.** Analyze.
Turbidity	**1.** Add 0.1 g scoop of RoVer® Rust Remover 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 and Filter Holder[1]. **5.** Use filtered sample in steps 6 and 3.
Extreme Sample pH	Adjust pH to 3–5.
Highly Buffered Samples	Adjust pH to 3–5.

[1] See Optional Reagents and Apparatus on page 6.

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). 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. Correct the test result for volume additions.

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. Press **OPTIONS>MORE**. Press **STANDARD ADDITIONS**. A summary of the standard additions procedure will appear.
3. Press **OK** to accept the default values for standard concentration, sample volume, and spike volumes. Press **EDIT** to change these values. After values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.
4. Snap the neck off an Iron Voluette Ampule Standard, 25-mg/L.
5. Prepare a 0.1 mL sample spike by adding 0.1 mL of standard to the unspiked sample. Press 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. Press 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. Press 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* 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*. Analyze each standard addition sample as described in the procedure above. Accept each standard additions reading by pressing* ***READ****. Each addition should reflect approximately 100% recovery.*

8. After completing the sequence, press **GRAPH** to view the best-fit line through the standard additions data points, accounting for matrix interferences. Press **IDEAL LINE** to view relationships between the sample spikes and the “Ideal Line” of 100% recovery.

* See Optional Reagents and Apparatus on page 6.

Standard Solution Method

1. Prepare a 2.00-mg/L Fe standard solution by pipetting 2.00 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 standard solution, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.

3. Press **ON**. Press **ADJUST** to accept the displayed concentration. If an alternate concentration is used, press the number in the box to enter the actual concentration, then press **OK**. Press **ADJUST**.

Method Performance

Precision
Standard: 2.00 mg/L Fe

Program	95% Confidence Limits of Distribution
265	1.99–2.01 mg/L Fe
267	1.98–2.02 mg/L Fe

Sensitivity

Program	Portion of Curve	ΔAbs	ΔConcentration
265	Entire range	0.010	0.021 mg/L Fe
267	Entire range	0.010	0.023 mg/L Fe

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.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
FerroVer® Iron Reagent Powder Pillows (for 10-mL sample)	1	100/pkg	21057-69
OR			
FerroVer® Iron Reagent AccuVac® Ampuls	1	25/pkg	25070-25

Required Apparatus (Powder Pillows)

Description	Quantity/Test	Unit	Cat. No.
Sample Cells, 1-inch square, 10 mL, matched pair	2	2/pkg	24954-02

Required Apparatus (AccuVacs)

Description	Quantity/Test	Unit	Cat. No.
Sample Cells, 10-mL, with cap	1	6/pkg	24276-06
Beaker, 50-mL	1	each	500-41H

Recommended Standards and Apparatus

Description	Unit	Cat. No.
Iron Standard Solution, 100-mg/L	100 mL	14175-42
Iron Standard Solution, 10-mL Voluette® Ampule, 25-mg/L as Fe	16/pkg	14253-10
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 L	272-56
Pipet, TenSette, 0.1–1.0 mL	each	19700-01
Pipet Tips, for TenSette Pipet 19700-01	50/pkg	21856-96
Pipet Tips, for TenSette Pipet 19700-01	1000/pkg	21856-28
Flask, volumetric, Class A, 100 mL	each	14574-42
Pipet, volumetric, Class A, 2.00 mL	each	14515-36
Pipet Filler, safety bulb	each	14651-00

Optional Reagents and Apparatus

Description	Cat. No.
Beaker, 50-mL	500-41H
Cylinder, mixing	1896-41
Hydrochloric Acid, concentrated	134-49
Nitric Acid, concentrated	152-49
Sodium Hydroxide Standard Solution, 5.0 N	2450-32
Glass Membrane Filter	2530-00
Glass Membrane Filter Holder	2340-00

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