Iron, Total

DOC316.53.01053

USEPA[1] FerroVer® Method[2] — Method 8008

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

Scope and application: For water, wastewater and seawater; digestion is required for determining total iron.

[1] USEPA approved for reporting wastewater analysis, Federal Register, June 27, 1980; 45 (126:43459).
[2] Adapted from Standard Methods for the Examination of Water and Wastewater.

Test preparation

Instrument-specific information

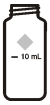
Table 1 shows sample cell and orientation requirements for reagent addition tests, such as powder pillow or bulk reagent tests. Table 2 shows sample cell and adapter requirements for AccuVac Ampul tests. The tables also show all of the instruments that have the program for this test.

To use the table, select an instrument, then read across to find the applicable information for this test.

Table 1 Instrument-specific information for reagent addition

Instrument	Sample cell orientation	Sample cell
DR 6000 DR 3800 DR 2800 DR 2700 DR 1900	The fill line is to the right.	2495402 
DR 5000 DR 3900	The fill line is toward the user.	
DR 900	The orientation mark is toward the user.	2401906 

Table 2 Instrument-specific information for AccuVac Ampuls

Instrument	Adapter	Sample cell
DR 6000 DR 5000 DR 900	—	2427606 
DR 3900	LZV846 (A)	
DR 1900	9609900 or 9609800 (C)	
DR 3800 DR 2800 DR 2700	LZV584 (C)	2122800 

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Before starting

Install the instrument cap on the DR 900 cell holder before ZERO or READ is pushed.
The reagent in this test procedure converts all soluble iron and most insoluble forms of iron in the sample to soluble ferrous iron for measurement. For regulatory reporting, however, the sample must be digested with heat and acid to make sure that all forms of the metal are measured.
For the best results, measure the reagent blank value for each new lot of reagent. Replace the sample with deionized water in the test procedure to determine the reagent blank value. Subtract the reagent blank value from the sample results automatically with the reagent blank adjust option.
For turbid samples, treat the blank with one 0.1-g scoop of RoVer Rust Remover. Swirl to dissolve.
Review the Safety Data Sheets (MSDS/SDS) for the chemicals that are used. Use the recommended personal protective equipment.
Dispose of reacted solutions according to local, state and federal regulations. Refer to the Safety Data Sheets for disposal information for unused reagents. Refer to the environmental, health and safety staff for your facility and/or local regulatory agencies for further disposal information.

Items to collect

Powder pillows

Description	Quantity
FerroVer® Iron Reagent Powder Pillows, 10-mL[1]	1
Sample cells. (For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.)	2

[1] FerroVer is a registered trademark of Hach Company.

Refer to Consumables and replacement items on page 7 for order information.

AccuVac Ampuls

Description	Quantity
FerroVer® Iron Reagent AccuVac® Ampul	1
Beaker, 50-mL	1
Sample cells (For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.)	1
Stopper for 18-mm tubes and AccuVac Ampuls	1

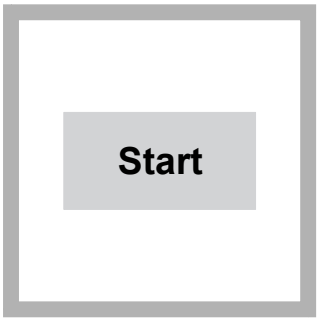
Refer to Consumables and replacement items on page 7 for order information.

Sample collection and storage

- Collect samples in clean glass or plastic bottles that have been cleaned with 6 N (1:1) hydrochloric acid and rinsed with deionized water.
- To measure only dissolved iron, filter the sample immediately after collection and before acidification.
- To preserve samples for later analysis, adjust the sample pH to less than 2 with concentrated nitric acid (approximately 2 mL per liter). No acid addition is necessary if the sample is tested immediately.
- Keep the preserved samples at room temperature for a maximum of 6 months.
- Before analysis, adjust the pH to 3–5 with 5 N sodium hydroxide solution.
- Correct the test result for the dilution caused by the volume additions.

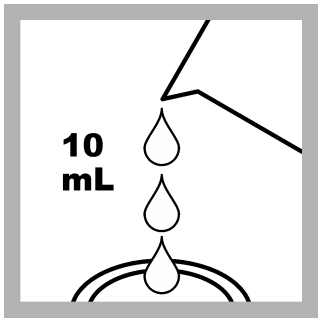
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Powder pillow procedure

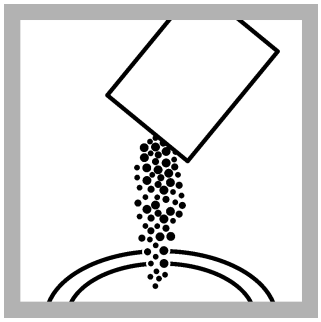


1. Start program **265 Iron, FerroVer**. For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.

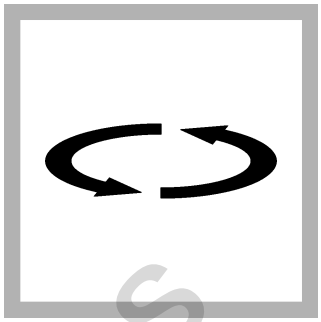
Note: *Although the program name can be different between instruments, the program number does not change.*



2. Prepare the sample: Fill a sample cell with 10 mL of sample.



3. Add the contents of one FerroVer Iron Reagent Powder Pillow to the sample cell.

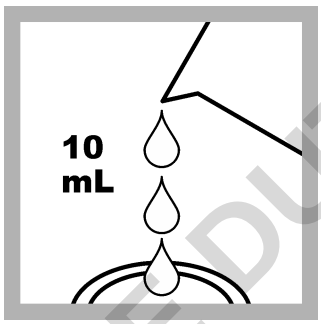


4. Swirl the sample cell to mix. Undissolved powder will not affect accuracy.

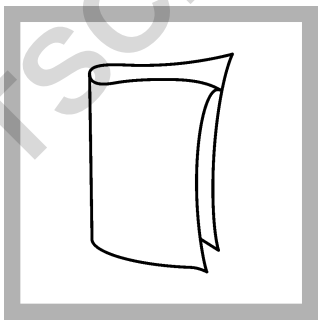


5. Start the instrument timer. A 3-minute reaction time starts.

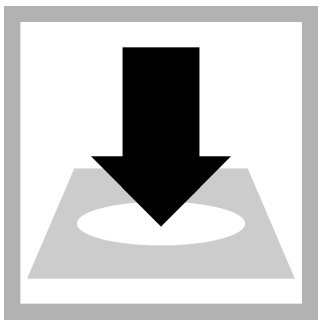
An orange color will show if iron is present. Let samples that contain rust react for 5 minutes or more.



6. Prepare the blank: Fill a second sample cell with 10 mL of sample.



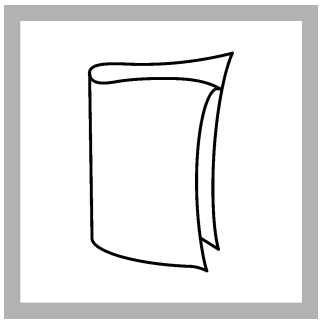
7. Clean the blank sample cell.



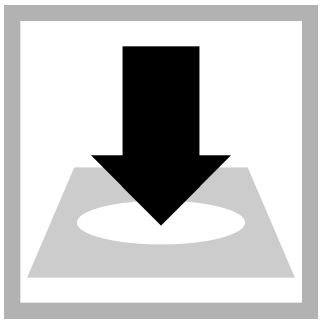
8. When the timer expires, insert the blank into the cell holder.



9. Push **ZERO**. The display shows 0.00 mg/L Fe.



10. Clean the prepared sample cell.



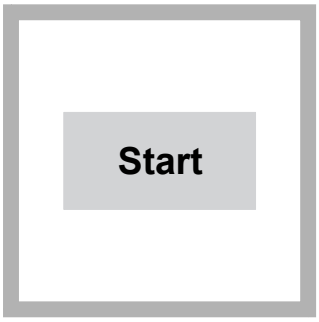
11. Insert the prepared sample into the cell holder.



12. Push **READ**. Results show in mg/L Fe.

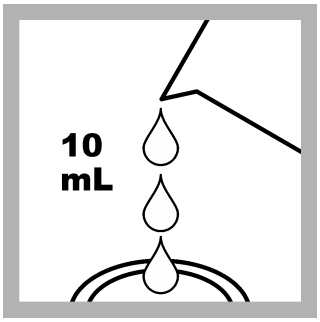
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AccuVac procedure

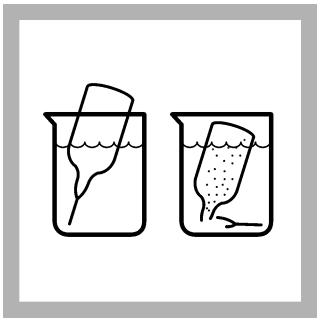


1. Start program **267 Iron, FerroVer AV**. For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.

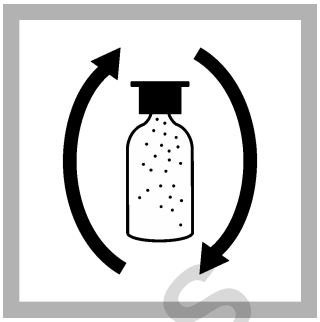
Note: *Although the program name can be different between instruments, the program number does not change.*



2. Prepare the blank: Fill the sample cell with 10 mL of sample.



3. Prepare the sample: Collect at least 40 mL of sample in a 50-mL beaker. Fill the AccuVac Ampul with sample. Keep the tip immersed while the AccuVac Ampul fills completely.

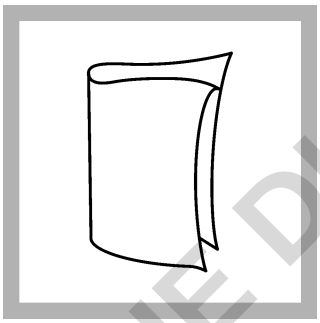


4. Quickly invert the AccuVac Ampul several times to mix. Undissolved powder will not affect accuracy.



5. Start the instrument timer. A 3-minute reaction time starts.

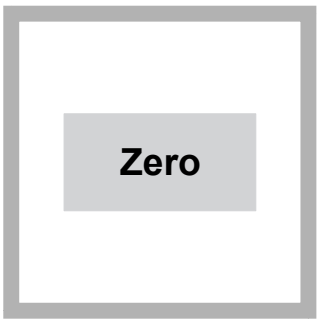
An orange color will show if iron is present. Let samples that contain rust react for 5 minutes or more.



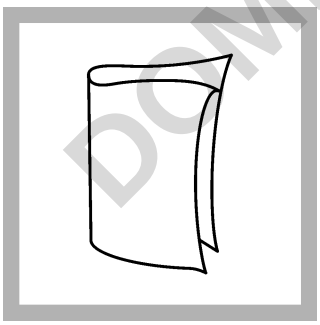
6. Clean the blank sample cell.



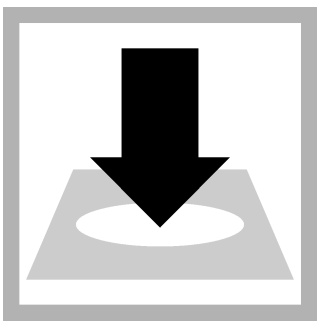
7. When the timer expires, insert the blank into the cell holder.



8. Push **ZERO**. The display shows 0.00 mg/L Fe.



9. Clean the AccuVac Ampul.



10. Insert the prepared sample AccuVac Ampul into the cell holder.



11. Push **READ**. Results show in mg/L Fe.

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Interferences

Interfering substance	Interference level
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	A mild, vigorous or Digesdahl digestion is necessary. After digestion, adjust the sample pH to 3–5 with sodium hydroxide, then analyze.
Magnesium	No effect at 100,000 mg/L as $CaCO_3$.
Molybdate molybdenum	No effect at 50 mg/L as Mo.
High sulfide levels, S^{2-}	Pretreat the sample in a fume hood or in an area with sufficient airflow before analysis: **1.** Add 5 mL of 6.0 N (1:1) hydrochloric acid solution to 100 mL of sample in a 250-mL Erlenmeyer flask. **2.** Boil for 20 minutes. **3.** Let the solution cool to room temperature. **4.** Adjust the pH to 3–5 with 5 N sodium hydroxide solution. **5.** Add deionized water until the volume is 100 mL. **6.** Use the treated sample in the test procedure.
Turbidity	Pre-treat the sample before analysis: **1.** Add one 0.1-g scoop of RoVer® Rust Remover to the blank. Swirl to mix. **2.** If the sample remains turbid, add three 0.2-g scoops of RoVer Rust Remover to 75 mL of sample. Let stand 5 minutes. **3.** Filter through a 0.45-micron membrane filter and filter holder. **4.** Use the treated sample in the test procedure.
Highly buffered samples or extreme sample pH	Can prevent the correct pH adjustment of the sample by the reagents. Sample pre-treatment may be necessary. Adjust the pH to 3–5.

Accuracy check

Standard additions method (sample spike)

Use the standard additions method (for applicable instruments) to validate the test procedure, reagents and instrument and to find if there is an interference in the sample.

Items to collect:

- Iron Voluette® Ampule Standard, 25 mg/L
- Ampule Breaker
- Pipet, TenSette®, 0.1–1.0 mL and tips

1. Use the test procedure to measure the concentration of the sample, then keep the (unspiked) sample in the instrument.
2. Go to the Standard Additions option in the instrument menu.
3. Select the values for standard concentration, sample volume and spike volumes.
4. Open the standard solution.
5. Prepare three spiked samples: use the TenSette pipet to add 0.1 mL, 0.2 mL and 0.3 mL of the standard solution, respectively, to three 10-mL portions of fresh sample. Mix well.

   Note: *For AccuVac® Ampuls, add 0.2 mL, 0.4 mL and 0.6 mL of the standard solution to three 50-mL portions of fresh sample.*

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6. Use the test procedure to measure the concentration of each of the spiked samples. Start with the smallest sample spike. Measure each of the spiked samples in the instrument.
7. Select **Graph** to compare the expected results to the actual results.

   Note: *If the actual results are significantly different from the expected results, make sure that the sample volumes and sample spikes are measured accurately. The sample volumes and sample spikes that are used should agree with the selections in the standard additions menu. If the results are not within acceptable limits, the sample may contain an interference.*

Standard solution method

Use the standard solution method to validate the test procedure, the reagents and the instrument.

Items to collect:

- Iron standard solution, 100 mg/L
- 100-mL volumetric flask, Class A
- 2-mL volumetric pipet, Class A and pipet filler
- Deionized water

1. Prepare a 2.00-mg/L iron standard solution as follows:
   a. Use a pipet to add 2 mL of the 100-mg/L iron standard solution into the volumetric flask.
   b. Dilute to the mark with deionized water. Mix well. Prepare this solution daily.
2. Use the test procedure to measure the concentration of the prepared standard solution.
3. Compare the expected result to the actual result.

   Note: *The factory calibration can be adjusted slightly with the standard adjust option so that the instrument shows the expected value of the standard solution. The adjusted calibration is then used for all test results. This adjustment can increase the test accuracy when there are slight variations in the reagents or instruments.*

Method performance

The method performance data that follows was derived from laboratory tests that were measured on a spectrophotometer during ideal test conditions. Users can get different results under different test conditions.

Program	Standard	Precision (95% Confidence Interval)	Sensitivity Concentration change per 0.010 Abs change
265	2.00 mg/L Fe	1.99–2.01 mg/L Fe	0.021 mg/L Fe
267	2.00 mg/L Fe	1.98–2.02 mg/L Fe	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. The measurement wavelength is 510 nm for spectrophotometers or 520 nm for colorimeters.

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Consumables and replacement items

Required reagents

Description	Quantity/Test	Unit	Item no.
FerroVer® Iron Reagent Powder Pillow[1], 10-mL	1	100/pkg	2105769
OR			
FerroVer® Iron Reagent AccuVac® Ampul	1	25/pkg	2507025

[1] FerroVer is a registered trademark of Hach Company

Required apparatus

Description	Quantity/Test	Unit	Item no.
Beaker, 50-mL	1	each	50041H
Stoppers for 18-mm tubes and AccuVac Ampuls	2	6/pkg	173106

Recommended standards and apparatus

Description	Unit	Item no.
Flask, volumetric, Class A, 100-mL glass	each	1457442
Iron Standard Solution, 100-mg/L Fe	100 mL	1417542
Iron Standard Solution, 10-mL Voluette® Ampule, 25-mg/L Fe	16/pkg	1425310
Metals Drinking Water Standard, LR for Cu, Fe, Mn	500 mL	2833749
Metals Drinking Water Standard, HR for Cu, Fe, Mn	500 mL	2833649
Pipet filler, safety bulb	each	1465100
Pipet, TenSette®, 0.1–1.0 mL	each	1970001
Pipet tips for TenSette® Pipet, 0.1–1.0 mL	50/pkg	2185696
Pipet tips for TenSette® Pipet, 0.1–1.0 mL	1000/pkg	2185628
Pipet, volumetric, Class A, 2-mL	each	1451536
Water, deionized	4 L	27256

Optional reagents and apparatus

Description	Unit	Item no.
AccuVac® Ampul Snapper	each	2405200
Mixing cylinder, graduated, 50-mL	each	189641
Filter, glass fiber membrane, 1.5-micron, 47-mm	100/pkg	253000
Filter membrane filter holder, 47-mm	each	234000
Hydrochloric Acid, concentrated	500 mL	13449
Nitric Acid, concentrated	500 mL	15249
RoVer Rust Remover	454 g	30001
Sodium Hydroxide Standard Solution, 5.0 N	100 mL MDB	245032
Spoon, measuring, 0.1-g	each	51100

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