Iron, Total

DOC316.53.01314

FerroVer® Method[1]

Method 10249

0.1 to 3.0, 1.0 to 30.0 and 10.0 to 300.0 mg/L Fe

Powder Pillows

Scope and application: For oil and gas field waters; digestion is required for total iron determinations.[2]

[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 all of the instruments that have the program for this test. The table also shows sample cell and orientation requirements for reagent addition tests, such as powder pillow or bulk reagent tests.

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

Table 1 Instrument-specific information

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 

Before starting

Install the instrument cap on the DR 900 cell holder before ZERO or READ is pushed.

To make sure that all forms of the metal are measured, digest the sample with heat and acid. Use the mild or vigorous digestion. Refer to the Water Analysis Guide for more information.

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.

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

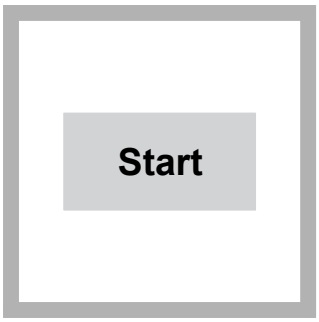
Description	Quantity
FerroVer® Iron Reagent Powder Pillow, 10-mL	1
EDTA solution, 1M	2 drops
Sample cells (For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.)	1

Refer to Consumables and replacement items on page 6 for order information.

Sample collection and storage

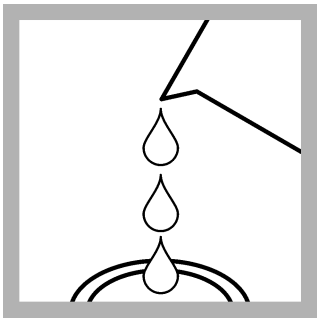
- Collect samples in clean glass or plastic bottles that have been cleaned with 6 N (1:1) hydrochloric acid and rinsed with deionized water.
- To preserve samples for later analysis, adjust the sample pH to less than 2 with concentrated nitric acid (about 2 mL per liter). No acid addition is necessary if the sample is tested immediately.
- To measure only dissolved iron, filter the sample immediately after collection and before acidification.
- Keep the preserved samples at room temperature for a maximum of 6 months.
- Before analysis, adjust the pH to 3–5 with 5.0 N sodium hydroxide standard solution.
- Correct the test result for the dilution caused by the volume additions.

Test procedure



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

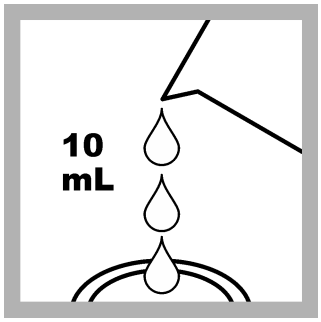
Note: *Although the program name can be different between instruments, the program number does not change.*



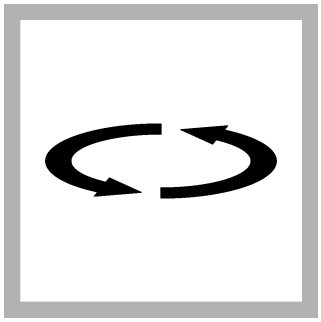
2. Fill a clean sample cell with sample:

- Use 10 mL of sample for the 0.02 to 3.0 mg/L range.
- Use 1.0 mL of sample for the 0.2 to 30.0 mg/L range with a dilution factor of 10.
- Use 0.1 mL of sample for the 2.0 to 300.0 range with a dilution factor of 100.

Note: *Refer to* Set the dilution factor *on page 4.*

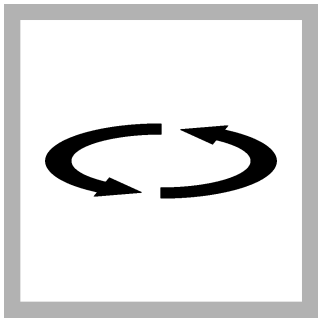


3. If the sample volume is less than 10 mL, add deionized water to the 10-mL line.

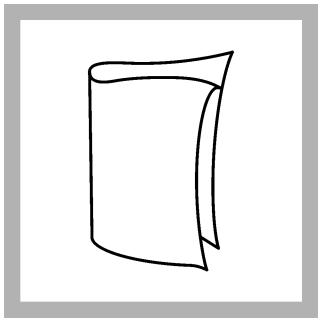


4. Swirl to mix.

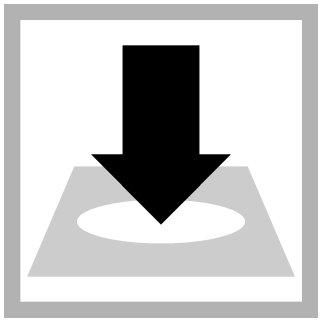
5. Add 2 drops of 1 M EDTA Solution to the sample.



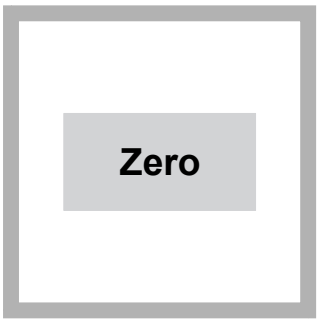
6. Swirl to mix.



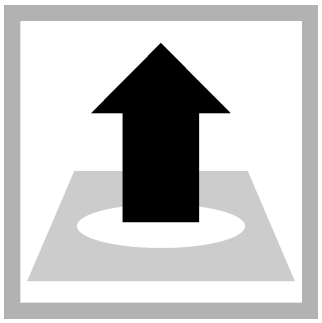
7. Clean the sample cell.



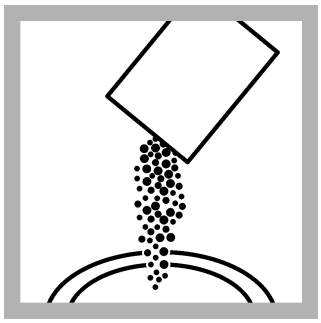
8. Insert the sample cell into the cell holder.



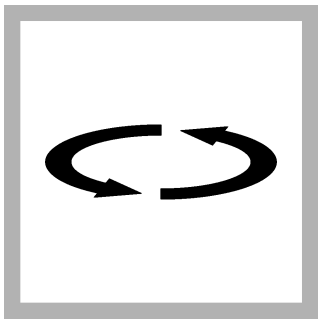
9. Push **ZERO**. The display shows 0.0 mg/L Fe.



10. Remove the sample cell from the cell holder.



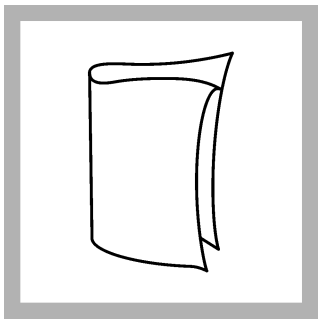
11. Add the contents of one FerroVer Iron Reagent Powder Pillow to the sample cell.



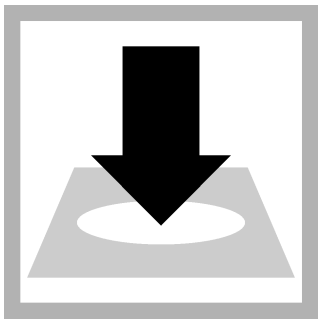
12. Swirl to mix. Accuracy is not affected by undissolved powder.

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

If iron is present in the sample, an orange color will show.



14. When the timer expires, clean the sample cell.



15. Insert the sample cell into the cell holder.

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

Interferences

Interfering substance	Interference level
Barium, Ba^{2+}	The dilution of samples lowers most barium concentrations below interference levels. No effects are seen on analyzed samples that contain less than 50 mg/L of Ba. No effects are seen when a 1.0 or 0.1 mL sample volume is used in the test procedure. A turbidity may show at higher levels. Use 5 drops of EDTA Solution in the test procedure and allow the sample to react for 5 minutes.
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.

Interfering substance	Interference level
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.
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.
Strontium, Sr^{2+}	Strontium by itself does not interfere. Strontium in combination with Barium will cause a precipitate to form. The dilution of samples lowers most strontium concentrations below interference levels. No effects are seen on analyzed samples that contain less than 50 mg/L of combined Ba and Sr. No effects are seen when a 1.0 or 0.1 mL sample volume is used in the test procedure. A turbidity may show at higher levels. Use 5 drops of EDTA Solution in the test procedure and allow the sample to react for 5 minutes.
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 sample pH to 3–5 before the test is started. Correct the test result for the dilution from the volume addition.

Set the dilution factor

Instruments that have a dilution factor option can include the dilution factor in the result and show the concentration of the original, undiluted sample. For example, if the sample is diluted by a factor of 10, the instrument multiplies the result by 10 and shows the calculated result in the instrument display.

1. Select **Options>More>Dilution** factor from the instrument menu.

   Note: *DR 1900: Select* ***Options>Advanced Options>Dilution Factors>On****.*

   Note: *Colorimeters include a dilution factor when the chemical form is set. Go to* ***Options>Advanced Options>Chemical Form*** *and select LR, MR or HR.*
2. Enter the dilution factor:
   - 1 mL sample diluted to 10 mL: dilution factor is 10.
   - 0.1 mL sample diluted to 10 mL: dilution factor is 100.
3. Push **OK** to confirm. Push **OK** again.
4. Push **RETURN** to go back to the measurement screen.

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.
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 safety bulb
- Deionized water

1. Prepare a 2.00 mg/L iron standard solution as follows:
   a. Use a pipet to add 2.00 mL of 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

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.

Consumables and replacement items

Required reagents

Description	Quantity/test	Unit	Item no.
FerroVer® Iron Reagent Powder Pillow[1], 10-mL	1	100/pkg	2105769
EDTA Solution, 1 M	2 drops	50 mL SCDB	2241926

[1] FerroVer is a registered trademark of Hach Company

Recommended standards

Description	Unit	Item no.
Iron Standard Solution, 100-mg/L Fe	100 mL	1417542
Iron Standard Solution, 10-mL Voluette® Ampule, 25-mg/L Fe	16/pkg	1425310
Water, deionized	4 L	27256
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
Flask, volumetric, Class A, 100-mL glass	each	1457442
Pipet, volumetric, Class A, 2-mL	each	1451536
Pipet filler, safety bulb	each	1465100

Optional reagents and apparatus

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

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