

Iron, Total

DOC316.53.01314

USEPA¹ FerroVer[®] Method²

Method 10249

0.1-3.0, 1.0-30.0 and 10.0-300.0 mg/L

Powder Pillows

Scope and Application: For oil and gas field waters; 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

How to use instrument-specific information

[Table 1](#) displays requirements that may vary between instruments. To use this table, select an instrument then read across to find the corresponding information required to perform this test.

Table 1 Instrument-specific information

Instrument	Sample cell	Cell orientation	Adapter
DR 5000	2495402	Fill line faces user	A23618
DR 3900	2495402	Fill line faces user	
DR 3800, DR 2800, DR 2700	2495402	Fill line faces right	

Before starting the test:

Digestion is required for determining total iron for EPA reporting purposes. Use the mild or vigorous digestion. Refer to the *Water Analysis Guide* for more information.

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.

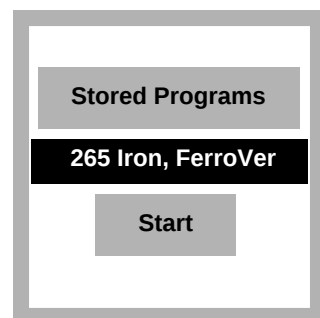
Adjust pH of stored samples before analysis.

Collect the following items:

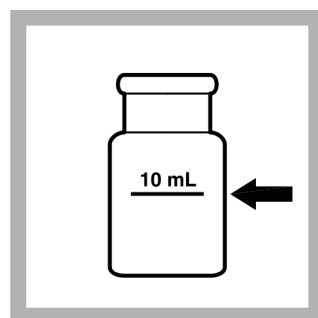
Description	Quantity
Powder Pillow Test:	
FerroVer [®] Iron Reagent Powder Pillow	1
Sample Cells (see Table 1)	1

See [Consumables and replacement items](#) for reorder information.

FerroVer method for powder pillows



1. Select the test.
Insert an adapter if required (see [Table 1](#)).
Refer to the user manual for orientation.

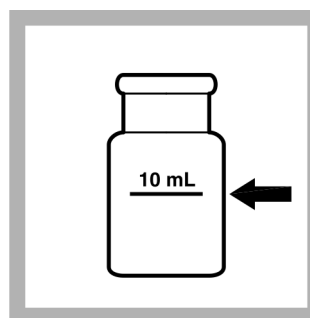


2. Prepared sample: Fill a clean sample cell with 10 mL of sample for 0.02-3.0 mg/l range.

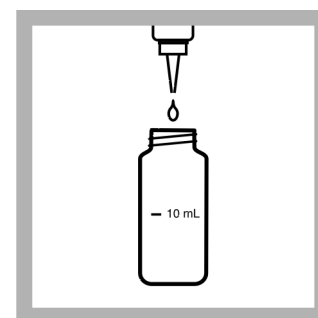
Use 1.0 mL of sample for the 0.2-30.0 mg/L range; enter Dilution Factor of 10. See Set the Dilution Factor.

Use 0.1 mL of sample for the 2.0-300.0 range. Use Dilution Factor of 100. See Set the Dilution Factor.

Note: Use a TenSette or glass pipet to measure 0.1 mL or 1.0 mL.



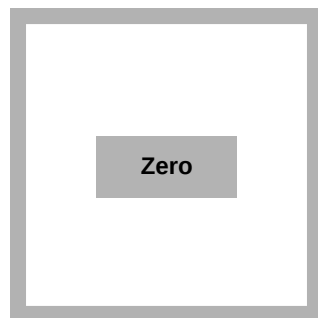
3. If sample volume is less than 10 mL, add deionized water to the 10-mL line. Swirl to mix.



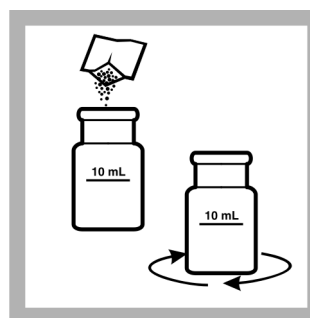
4. Add 2 drops of EDTA Solution 1M to the sample. Swirl to mix.



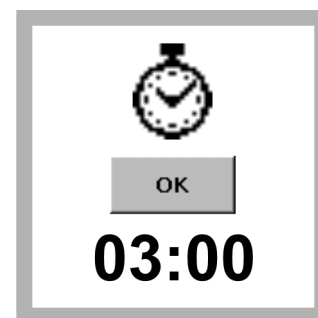
5. Insert the sample into the cell holder.



6. Zero the instrument. The display will show: 0.0 mg/L Fe



7. Remove the cell and add the contents of one FerroVer Iron Reagent Powder Pillow to the sample cell. Swirl to mix. Accuracy is not affected by undissolved powder.



8. Start the instrument timer. A three-minute reaction period begins. If iron is present, an orange color will form.

FerroVer method for powder pillows (continued)



9. When the timer expires, insert the prepared sample into the cell holder.

READ the results in mg/L Fe.

Interferences

The known interferences are shown in [Table 2](#).

Table 2 Interfering substances

Interfering substance	Interference level
Barium, Ba ²⁺	The dilution of samples to measure iron lowers most barium concentrations below barium interference levels. No effects are seen on analyzed solutions which contain less than 500 mg/L barium. No effects are seen when 1.0 or 0.1 mL of sample is used in Step 5. A turbidity may show at higher levels. Add 5 drops of EDTA Solution to the solution in Step 5 and repeat the test. 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.
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 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 acid¹, ACS to 100 mL sample in a 250 mL Erlenmeyer flask. Boil 20 minutes. 2. Cool. Adjust pH to 3–5 with Sodium Hydroxide¹. Readjust volume to 100 mL with deionized water. 3. Analyze using FerroVer method for powder pillows.
Strontium, Sr ²⁺	Strontium by itself does not interfere. Strontium in combination with Barium will cause a precipitate to form. The dilution of samples to measure iron lowers most strontium concentration below interference levels. No effects are seen on analyzed solutions when 1.0 or 0.1 mL of sample is used in step 4. A turbidity may start at higher levels. Add 5 drops of EDTA Solution to the solution in Step 5 and repeat the analysis again. Allow the sample to react for 5 minutes.
Extreme Sample pH	Adjust pH to 3–5.
Highly Buffered Samples	Adjust pH to 3–5.

¹ See [Optional reagents and apparatus](#).

Sample collection, preservation and storage

- 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.

Set the dilution factor

The instrument 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.
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)

Required for accuracy check:

- Iron Voluette® Ampule Standard, 25 mg/L
 - Ampule breaker
 - TenSette Pipet and Pipet Tips
1. After reading test results, leave the sample cell (unspiked sample) in the instrument.
 2. Select Options>More>Standard Additions from the instrument menu.
 3. Accept the default values for standard concentration, sample volume and spike volumes. After the values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.
 4. Open the standard solution ampule.
 5. Prepare a 0.1 mL sample spike by adding 0.1 mL of standard to 10 mL of unspiked sample. Start the instrument timer. After the timer expires, 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. Start the instrument timer. After the timer expires, 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. Start the instrument timer. After the timer expires, read the result.
 8. Select **GRAPH** to view the results. Select **IDEAL LINE** (or best-fit) to compare the standard addition results to the theoretical 100% recovery.

Standard solution method

Note: Refer to the instrument user manual for specific software navigation instructions.

Required for accuracy check:

- Iron Standard Solution, 100 mg/L
 - 100-mL volumetric flask
 - Class A volumetric pipet, 2 mL
 - Deionized water
 - Pipet filler
1. Prepare a 2.00-mg/L Fe standard solution as follows:
 - a. Pipet 2.00 mL of Iron Standard Solution, 100 mg/L, into a 100 mL volumetric flask.
 - b. Dilute to the mark with deionized water. Mix well. Prepare this solution daily.
 2. Use the 2.00 mg/L Fe standard solution in place of the sample. Follow the [FerroVer method for powder pillows](#) test procedure.
 3. To adjust the calibration curve using the reading obtained with the Standard Solution, select **Options > More > Standard Adjust** from the instrument menu.
 4. Turn on the Standard Adjust feature and accept the displayed concentration. If an alternate concentration is used, enter the concentration and adjust the curve to that value. Mixed-parameter standards are also available to simulate various matrices.

Method performance

Program	Standard	Precision 95% Confidence Limits of Distribution	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. Test results are measured at 510 nm.

Consumables and replacement items**Required reagents**

Description	Quantity/Test	Unit	Catalog number
FerroVer® Iron Reagent Powder Pillows (for 10-mL sample)	1	100/pkg	2105769
EDTA Solution 1M 2 drops	2 drops	50 mL	2241926

Iron, Total

Recommended standards

Description	Unit	Catalog number
Iron Standard Solution, 100 mg/L	100 mL	1417542
Iron Standard Solution, 10 mL Voluette® Ampule, 25 mg/L as Fe	16/pkg	1425310
Water, deionized	4 L	27256
Pipet, TenSette, 0.1–1.0 mL	each	1970001
Pipet Tips, for TenSette Pipet 1970001	50/pkg	2185696
Pipet Tips, for TenSette Pipet 1970001	1000/pkg	2185628
Flask, volumetric, Class A, 100 mL	each	1457442
Pipet, volumetric, Class A, 2.00 mL	each	1451536
Pipet Filler, safety bulb	each	1465100

Optional reagents and apparatus

Description	Unit	Catalog number
Hydrochloric Acid, concentrated	500 mL	13449
Nitric Acid, concentrated	500 mL	15249
Sodium Hydroxide Standard Solution, 5.0 N	100 mL	245032
Glass Membrane Filter, 47 mm	100/pkg	253000
Glass Membrane Filter Holder	each	234000
RoVer Rust Remover	454 g	30001
Spoon, measuring, 0.1 g	each	51100



FOR TECHNICAL ASSISTANCE, PRICE INFORMATION AND ORDERING:
In the U.S.A. – Call toll-free 800-227-4224
Outside the U.S.A. – Contact the HACH office or distributor serving you.
On the Worldwide Web – www.hach.com; E-mail – techhelp@hach.com

HACH COMPANY
WORLD HEADQUARTERS
Telephone: (970) 669-3050
FAX: (970) 669-2932