

FerroZine® Method¹

Method 8147

0.009 to 1.400 mg/L Fe

Reagent Solution Pillows

Scope and application: For water and seawater.

¹ Adapted from Stookey, L.L., Anal. Chem., 42(7), 779 (1970).



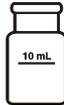
Test preparation

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

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.

Clean all glassware with 6.0 N (1:1) hydrochloric acid, then fully rinse with deionized water to remove contaminants.

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.

If the sample contains rust, refer to [Interferences](#) on page 3.

The FerroZine Iron Reagent can crystallize or precipitate if kept at cold temperatures during shipment. The reagent quality is not affected. Put the reagent in warm water to dissolve the precipitate.

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

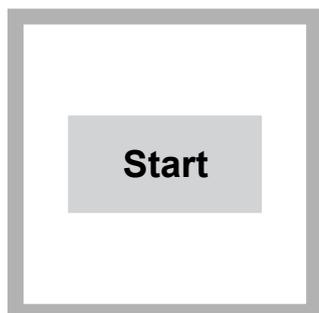
Description	Quantity
FerroZine Iron Reagent Solution Pillows, OR	1
FerroZine Iron Reagent Solution	0.5 mL
Cylinder, 25-mL graduated mixing, with stopper	1
Clippers for solution pillows	1
Sample cells (For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.)	2

Refer to [Consumables and replacement items](#) on page 5 for order information.

Sample collection and storage

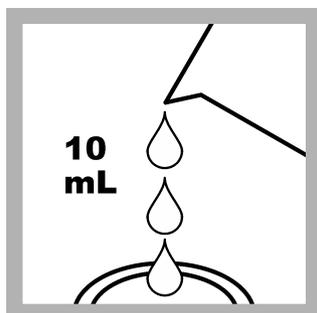
- Collect samples in clean glass or plastic bottles that have been cleaned with 6 N (1:1) hydrochloric acid and rinsed with deionized water.
- To preserve samples for later analysis, adjust the sample pH to less than 2 with concentrated nitric acid (approximately 2 mL per liter). No acid addition is 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 10% ammonium hydroxide solution. Do not exceed pH 5 to prevent precipitation of the iron.
- Correct the test result for the dilution caused by the volume additions.

Solution pillow or bulk solution procedure

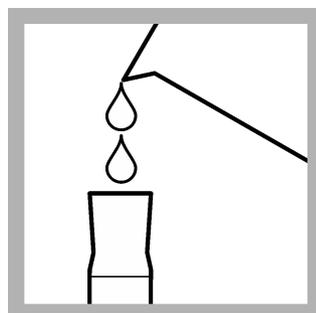


1. Start program **260 Iron, FerroZine**. For information about sample cells, adapters or light shields, refer to [Instrument-specific information](#) on page 1.

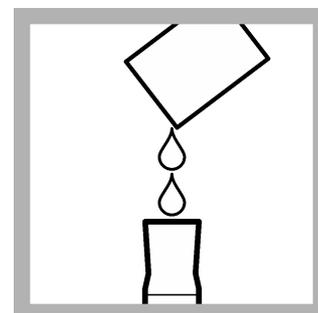
Note: Although the program name can be different between instruments, the program number does not change.



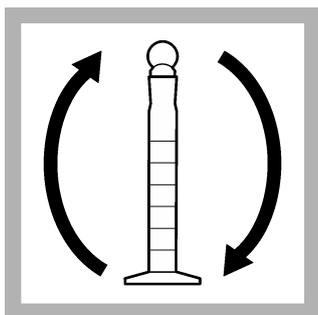
2. **Prepare the blank:** Fill the sample cell with 10 mL of sample.



3. **Prepare the sample:** Fill a mixing cylinder to the 25-mL line with sample.



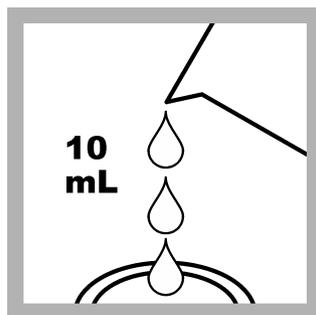
4. Add the contents of one FerroZine Iron Reagent Solution Pillow to the mixing cylinder.



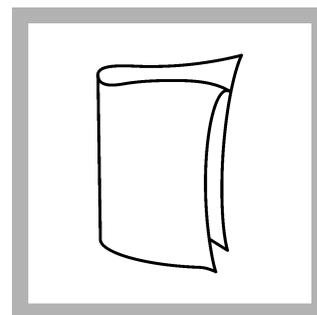
5. Put the stopper on the mixing cylinder. Invert the mixing cylinder several times to mix.



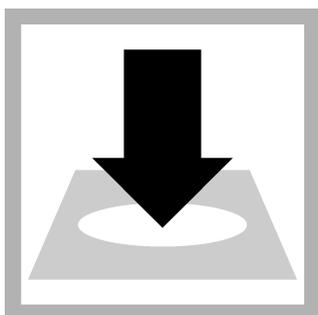
6. Start the instrument timer. A 5-minute reaction time starts.
A purple color shows if iron is present in the sample.



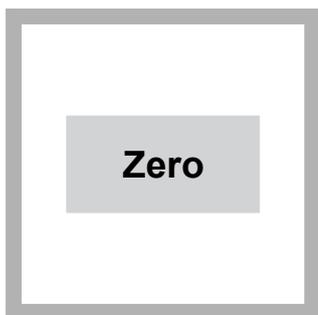
7. When the timer expires, pour 10 mL of the prepared sample from the mixing cylinder into the second sample cell.



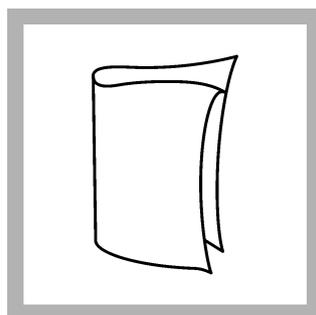
8. Clean the blank sample cell.



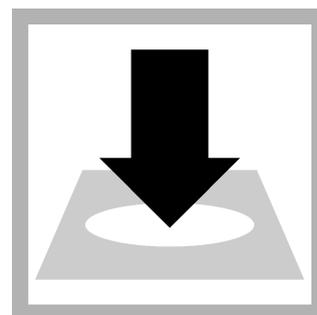
9. Insert the blank into the cell holder.



10. Push **ZERO**. The display shows 0.000 mg/L Fe.



11. Clean the prepared sample cell.



12. Insert the prepared sample into the cell holder.



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

Interferences

Interfering substance	Interference level
Strong chelants (EDTA)	Interfere at all levels. Use the FerroVer [®] or TPTZ methods for these samples. Use the TPTZ method for low iron concentrations.
Cobalt	May give slightly high results.
Copper	May give slightly high results.
Hydroxides	Add 1 mL of FerroZine Iron Reagent to 50 mL of sample, then boil the sample for 1 minute in a boiling water bath. Let the sample cool to room temperature, then start the instrument timer. Return the sample volume to 50 mL with deionized water.

Interfering substance	Interference level
Magnetite (black iron oxide) or Ferrites	Add 1 mL of FerroZine Iron Reagent to 50 mL of sample, then gently boil the sample for 20 to 30 minutes in a boiling water bath. <i>Note: Do not let the sample boil dry. A purple color forms if iron is present.</i> Let the sample cool to room temperature. Return the sample volume to 50 mL with deionized water. Continue with the test procedure after the timer step.
Rust	Add 1 mL of FerroZine Iron Reagent to 50 mL of sample, then boil the sample for 1 minute in a boiling water bath. Let the sample cool to room temperature, then start the instrument timer. Return the sample volume to 50 mL with deionized water.

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, 10 mg/L Fe
 - 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 25-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
 - 500-mL volumetric flask, Class A
 - 5-mL volumetric pipet, Class A and pipet filler safety bulb
 - Deionized water
1. Prepare a 1.0-mg/L iron standard solution as follows:
 - a. Use a pipet to add 5.00 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.

- 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
260	1.000 mg/L Fe	0.985–1.015 mg/L Fe	0.009 mg/L Fe

Summary of method

The FerroZine[®] Iron Reagent forms a purple-colored complex with trace amounts of iron in samples that are buffered to a pH of 3.5. This method is applicable for determining trace levels of iron in chemical reagents and glycols. With digestion, this method can be used to analyze samples that contain magnetite (black iron oxide) or ferrites. The measurement wavelength is 562 nm for spectrophotometers or 560 nm for colorimeters.

Consumables and replacement items

Required reagents

Description	Quantity/test	Unit	Item no.
FerroZine ^{®1} Iron Reagent Solution	0.5 mL	500 mL	230149
OR			
FerroZine [®] Iron Reagent Solution Pillow	1	50/pkg	230166

¹ FerroZine is a registered trademark of Hach Company.

Required apparatus

Description	Quantity/test	Unit	Item no.
Clippers for plastic pillows	1	each	96800
Mixing cylinder, graduated, 25-mL with stopper	1	each	2088640
Sample cells, 10-mL square, matched pair	2	2/pkg	2495402

Recommended standards and apparatus

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
Metals Drinking Water Standard, LR for Cu, Fe, Mn	500 mL	2833749
Flask, volumetric, Class A, 500-mL glass	each	1457449
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 5.00-mL	each	1451537
Pipet filler, safety bulb	each	1465100

Optional reagents and apparatus

Description	Unit	Item no.
Ammonium Hydroxide, 10%	100 mL MDB	1473632
Hydrochloric Acid Solution, 6 N (1:1)	500 mL	88449
Nitric Acid, concentrated	500 mL	15249



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