

FerroZine® Rapid Liquid Method¹

Method 8147

0.009 to 1.400 mg/L Fe

Pour-Thru Cell

Scope and application: For boiler, cooling and natural waters.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.

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	Pour-Thru Kit	Adapter
DR 6000	The flow path is to the right.	LQV157.99.20002	—
DR 3800		5940400	LZV585 (B)
DR 2800		5940400	LZV585 (B)
DR 2700		5940400	LZV585 (B)
DR 1900		LZV899	—
DR 5000	The flow path is toward the user.	LZV479	—
DR 3900		LQV157.99.10002	—

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	1.0 mL
Cylinder, graduated, 50-mL, polypropylene	1
Dispenser, fixed volume, 1.0-mL, with bottle	1

Items to collect (continued)

Description	Quantity
Flask, Erlenmeyer, Polymethylpentene, screw cap, 125-mL	1
Water, deionized	varies
Pour-Thru Cell (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 5 for order information.

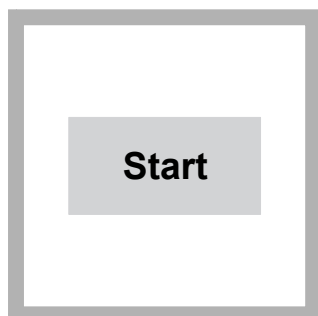
Sample collection and storage

- 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.
- 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. Do not exceed pH 5 or iron can precipitate.
- Correct the test result for the dilution caused by the volume additions.

Labware storage

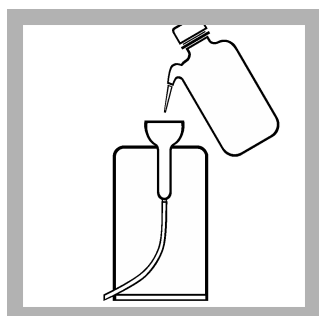
- Before the test procedure, it is necessary to fully clean all containers to remove iron contamination.
- Rinse labware and the Pour-Thru Cell with a 1:1 HCl solution or with a 1:50 dilution of FerroZine® Reagent. Rinse several times with deionized water.
- Keep flasks tightly closed when not in use. Dedicate these containers for iron analysis only.
- Only occasional treatment with HCl or FerroZine is necessary if the containers are consistently rinsed and the caps put on tightly after use.

Test procedure

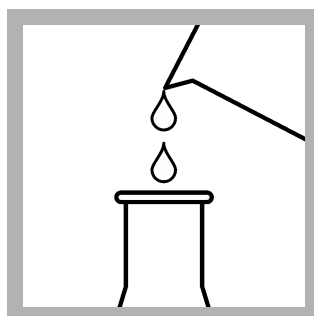


1. Start program 261 Iron, FerroZine RL. For information about sample cells, adapters or light shields, refer to [Instrument-specific information](#) on page 1.

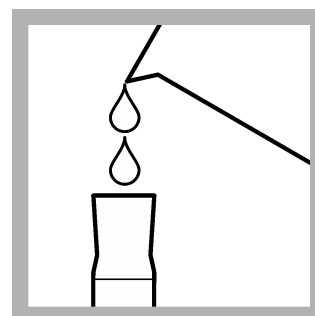
Note: Although the program name can be different between instruments, the program number does not change.



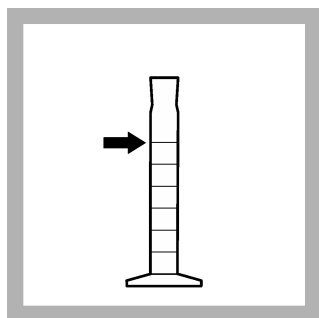
2. Flush the Pour-Thru Cell with 50 mL of deionized water.



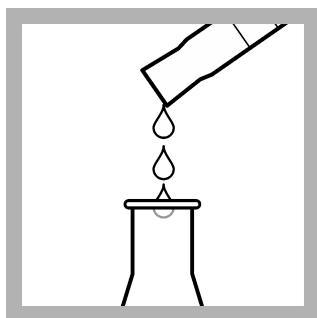
3. Rinse a clean 125-mL Erlenmeyer flask with the sample three times.



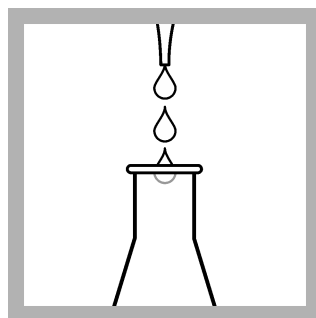
4. Rinse a clean 50-mL plastic graduated cylinder with the sample three times.



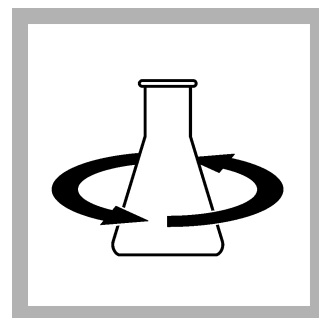
5. Fill the graduated cylinder to the 50-mL mark with sample.



6. Pour the contents of the 50-mL graduated cylinder into the flask.



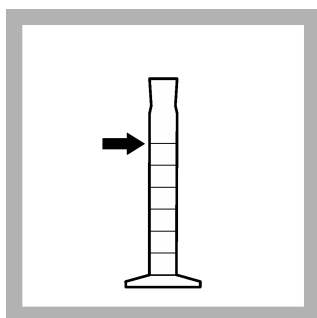
7. Prepare the sample: Use the bottle-top dispenser to add 1.0 mL of FerroZine Iron Reagent Solution to the flask.



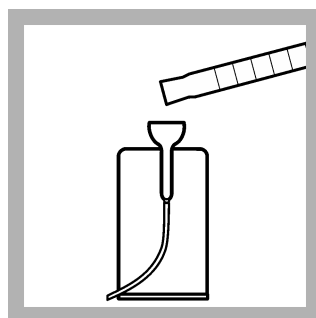
8. Swirl to mix.



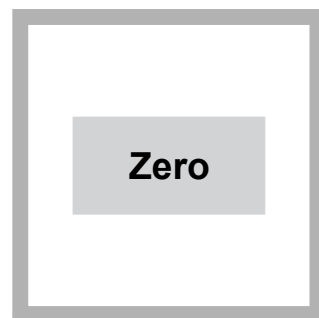
9. Start the instrument timer. The reaction time starts.



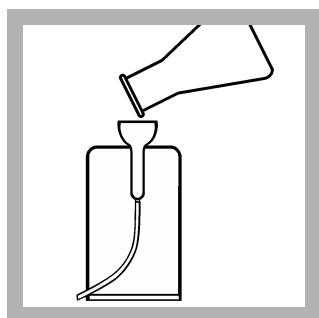
10. Prepare the blank: Measure a second 50 mL portion of the sample into the graduated cylinder.



11. When the timer expires, pour the contents of the graduated cylinder with the blank sample into the Pour-Thru Cell.



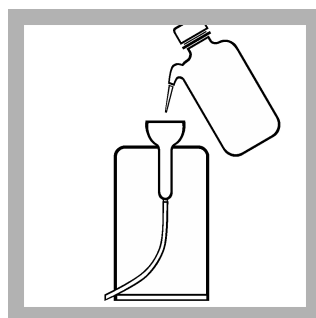
12. When the flow stops, push **ZERO**. The display shows 0.000 mg/L Fe.



13. Pour the contents of the flask with the prepared sample into the Pour-Thru Cell.



14. When the flow stops, push **READ**. Results show in mg/L Fe.



15. Immediately after use, flush the Pour-Thru Cell with at least 50 mL of deionized water.

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.

Interfering substance	Interference level
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.
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. Note: Do not let the sample boil dry. A purple color forms if iron is present. 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:

- 25-mg/L Iron Voluette Ampule Standard with ampule breaker
 - 50-mL graduated mixing cylinders (3x)
 - 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 0.2 mL, 0.4 mL and 0.6 mL of the standard solution, respectively, to three 50-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:

- 1.0-mg/L or 100-mg/L Iron Standard Solution
 - 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 Iron Standard Solution, 100 mg/L Fe, into a 500 mL volumetric flask. standard solution into the volumetric flask. As an alternative, purchase a prepared 1.0 mg/L iron standard solution.

- 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.

Clean the Pour-Thru Cell

The Pour-Thru Cell can collect a buildup of products with color, especially if the reacted solutions stay in the cell for long periods of time after measurement. Rinse with a 1:5 dilution of Ammonium Hydroxide with several rinses with deionized water to remove the color. Put a cover on the Pour-Thru Cell after use.

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
261	1.000 mg/L Fe	0.997–1.003 mg/L Fe	0.009 mg/L Fe

Summary of Method

The FerroZine® Iron Reagent forms a purple complex with trace amounts of iron in samples that are buffered to a pH of 3.5. This method is applicable to determine trace levels of iron in chemical reagents and glycols. With digestion, this method is also used to analyze samples that contain magnetite (black iron oxide) or ferrites. The measurement wavelength is 562 nm.

Consumables and replacement items

Required reagents

Description	Quantity/Test	Unit	Item no.
FerroZine® Iron Reagent Solution	1 mL	500 mL	230149
Water, deionized	varies	4 L	27256

Required apparatus

Description	Quantity/test	Unit	Item no.
Cylinder, graduated, 50-mL, polypropylene	1	each	108141
Dispenser, fixed volume, 1.0 mL	1	each	2111302
Flask, Erlenmeyer, PMP with cap 125-mL	2	each	2089843

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
Iron Standard Solution, 1-mg/L Fe	500 mL	13949

Optional reagents and apparatus

Description	Unit	Item no.
Ammonium Hydroxide, 10%	100 mL MDB	1473632
Ammonium Hydroxide, 58%	500 mL	10649
Hydrochloric Acid Solution, 6 N (1:1)	500 mL	88449
Nitric Acid, concentrated	500 mL	15249
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, volumetric 5.00-mL	each	1451537
Pipet filler, safety bulb	each	1465100
Water, deionized	4 L	27256



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