# Iron, Total

DOC316.53.01520

## FerroZine® Method[1]

**Method 10287**

**1.00 to 100 µg/L Fe (1-inch cell)**

**Reagent Solution**

**Scope and application:** For ultrapure water.

[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 specific instruments.

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 | The fill line is to the right. | 2495402   |
| DR 5000<br>DR 3900 | The fill line is toward the user. | |

### Before starting

This analysis is extremely sensitive to contamination. Make sure that the samples and equipment do not become contaminated. All of the materials that can contact an item that is used in the analysis are potential sources of contamination. Contamination sources include: paper towels, lab wipes, cardboard, filter paper, pipet tips, water, bottles and bottle lids.

Before analysis, carefully pour the FerroZine Iron Reagent from the bulk bottle into a clean dropping bottle. This transfer decreases contamination from solids that can collect at the bottom of the bulk bottle. Do not pour the solids into the dropping bottle. Safely discard the reagent that contains the solids.

Always use the same lot of reagent to measure the color of the reagent and the sample.

Before first use, make a 12-mL fill line on a 20-mm reaction tube. Use a pipet (rinse pipet tips with deionized water) to add 12-mL of deionized water into a 20-mm reaction tube. Use a permanent marker to show the 12-mL fill line on the reaction tube. Use this fill line to measure the 12-mL volume of the sample during the digestion procedure.

Before first use, clean all glassware with 1:1 hydrochloric acid solution, then fully rinse with deionized water.

Before first use, clean the 20-mm reaction tubes. To clean the 20-mm reaction tubes, add a blank solution (deionized water and reagent) to each reaction tube and digest for 24 hours. Use these reaction tubes only for this procedure. Keep a blank solution in the reaction tubes and sample cell during storage.

DR 6000 only: This method is sensitive to absorbance changes of less than 0.001 absorbance units. Before analysis, wait 30 minutes to let the instrument become stable. Set the instrument to "continuous" read. Zero the instrument on air (no cell). Monitor the absorbance values. When the absorbance does not change, the instrument is ready to use.

Make sure that the sample cell is in the same position in the cell compartment for each reading. Use the same sample cell to measure the color of the reagent and the sample.

**Use a fume hood for all analyses.**

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.

Make sure that samples are at room temperature or warmer so the reaction tube does not break when it is put in the DRB200.

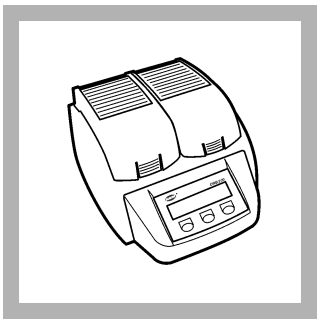
| |
|---|
| Review the Safety Data Sheets (MSDS/SDS) for the chemicals that are used. Use the recommended personal protective equipment. |
| Dispose of reacted solutions according to local, state and federal regulations. Refer to the Safety Data Sheets for disposal information for unused reagents. Refer to the environmental, health and safety staff for your facility and/or local regulatory agencies for further disposal information. |

## Items to collect

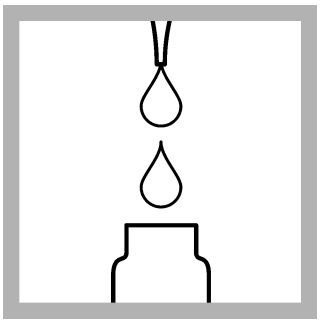
| Description | Quantity |
|---|---|
| FerroZine Iron Reagent Solution | 0.24 mL |
| DRB200 reactor | 1 |
| Digestion vials, 20-mm | 2 |
| Dropping bottle, 59-mL | 1 |
| Pipet, adjustable volume, 0.2–1.0 mL and 1.0 and 5.0 mL with pipet tips | varies |
| Water, deionized | varies |
| Sample cell (For 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 7 for order information.

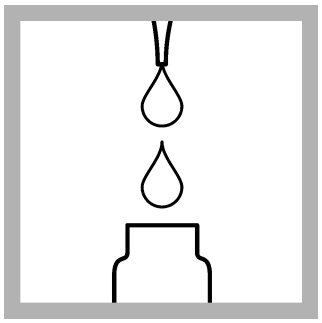
## Digestion procedure



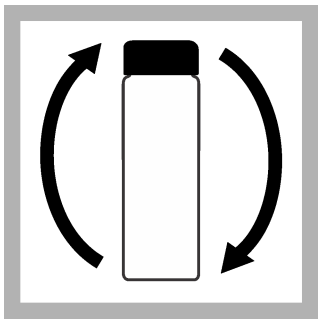
**1.** Set the DRB200 reactor power to on. Set the temperature to 135 °C.



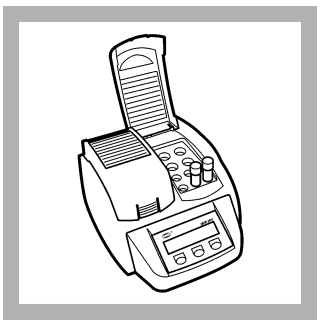
**2. Prepare the sample:** Pour 12 mL of sample into a 20-mm reaction tube.



**3.** Use the dropping bottle to add 8 drops of the FerroZine Reagent Solution to the reaction tube.

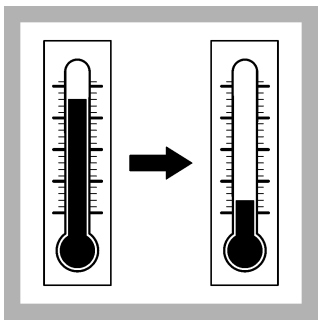


**4.** Tighten the cap on the reaction tube and invert to mix.



**5.** Insert the reaction tube in the preheated DRB200 reactor. Close the lid.

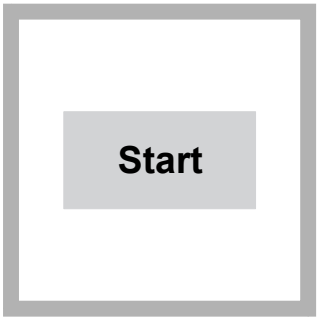

**6.** Keep the reaction tube in the reactor for 30 minutes.



**7.** When the timer expires, carefully remove the reaction tube from the reactor. Let the temperature of the reaction tube decrease to room temperature.

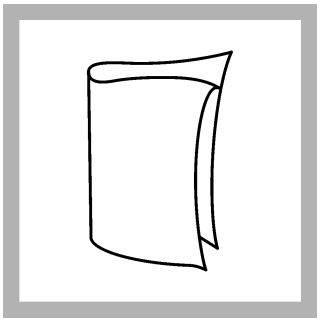
## Measure the color of the reagent

Each time the dropping bottle is filled, measure the color of the FerroZine Reagent Solution in the dropping bottle. Subtract the color of the FerroZine Reagent Solution from the result of the colorimetric procedure.

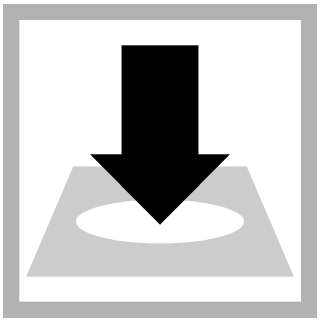



**1.** Start program **269 Iron, Total Rapid Liquid**. For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.

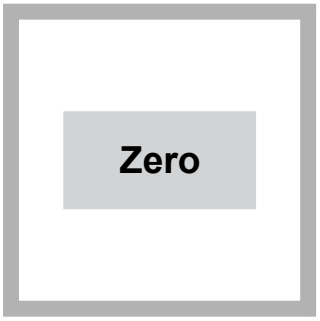
**2.** Add 10 mL of deionized water to the sample cell.



**3.** Clean the sample cell.

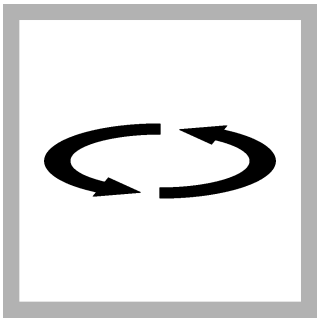


**4.** Insert the sample cell into the cell holder.

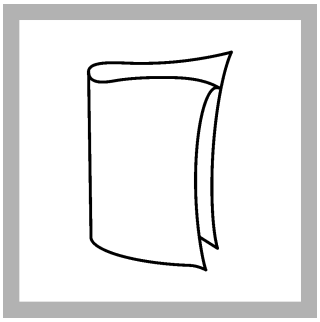



**5.** Push **ZERO**. The display shows 0.0 µg/L Fe.

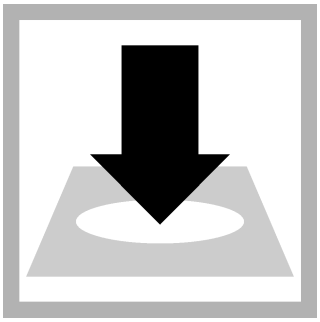
**6.** Use the dropping bottle to add 8 drops of the FerroZine Reagent Solution to the sample cell.



**7.** Swirl to mix.

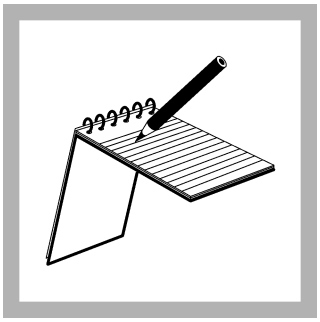


**8.** Clean the sample cell.



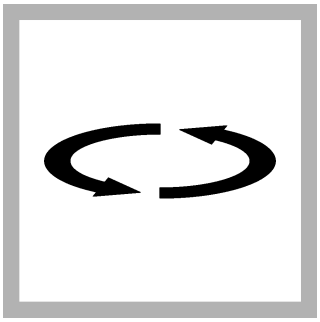
**9.** Insert the sample cell into the cell holder.


**10.** Push **READ**. Results show in µg/L Fe.

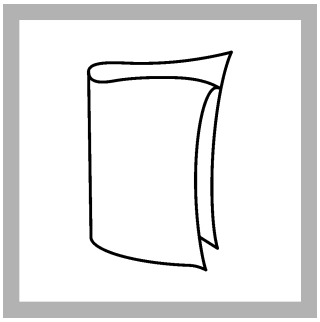


**11.** Record the result.

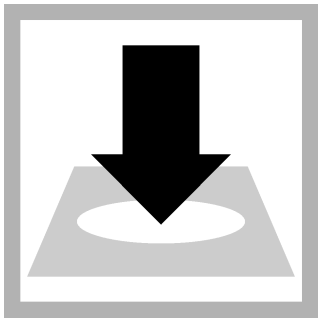
**12.** Use the dropping bottle to add 8 more drops of the FerroZine Reagent Solution to the sample cell.



**13.** Swirl to mix.

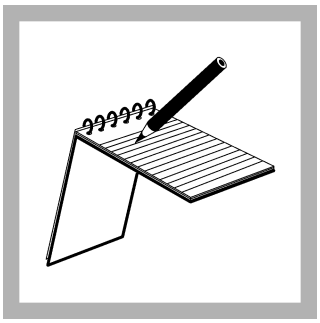


**14.** Clean the sample cell.

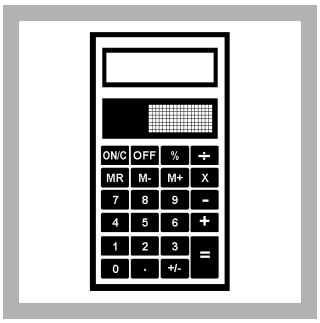


**15.** Insert the sample cell into the cell holder.

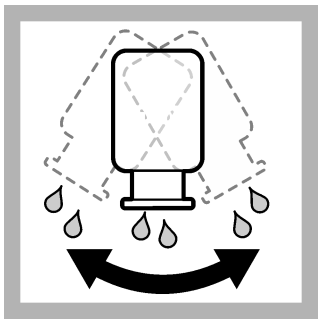

**16.** Push **READ**. Results show in µg/L Fe.



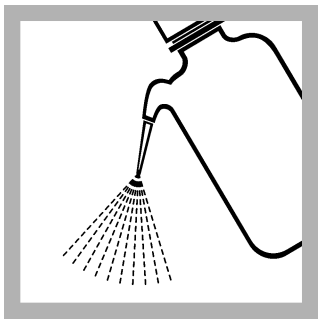
**17.** Record the result.



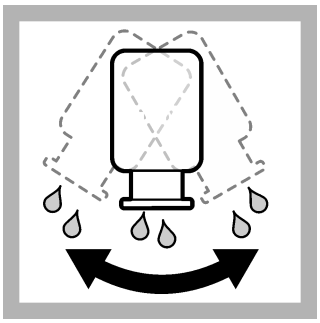

**18.** Subtract the result from step 11 from the result of step 17. The result is the color of the FerroZine Reagent Solution.



**19.** Discard the solution from the sample cell. Shake to remove excess solution from the sample cell.

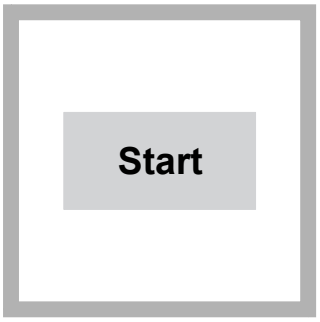


**20.** Rinse the sample cell with deionized water.



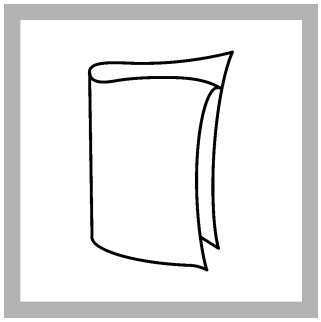
**21.** Discard the solution from the sample cell. Shake to remove excess solution from the sample cell.

## Colorimetric procedure

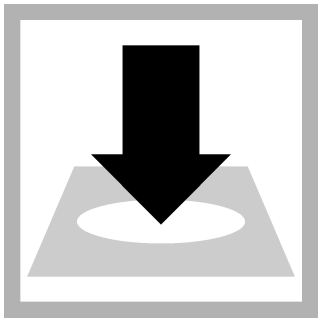



**1.** Start program **269 Iron, Total Rapid Liquid**. For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.

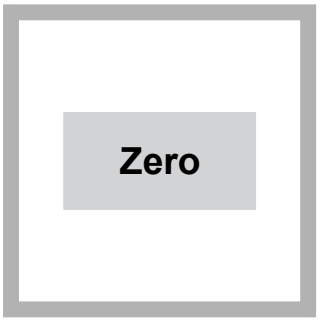
**2.** Add 10 mL of deionized water to the sample cell. The same sample cell must be used to measure the color of the reagent and the sample.



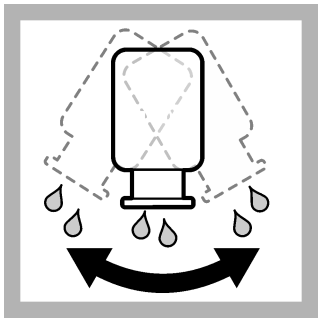
**3.** Clean the sample cell.



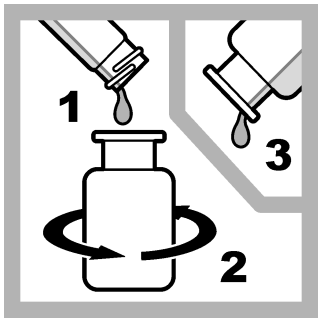
**4.** Insert the sample cell into the cell holder.



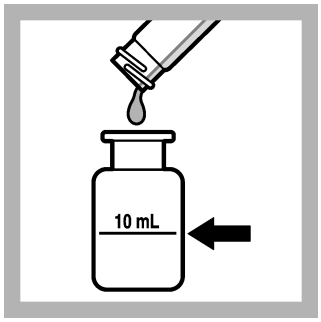

**5.** Push **ZERO**. The display shows 0.0 µg/L Fe.



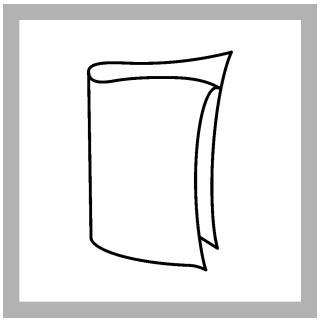
**6.** Discard the solution from the sample cell. Shake to remove excess solution from the sample cell.



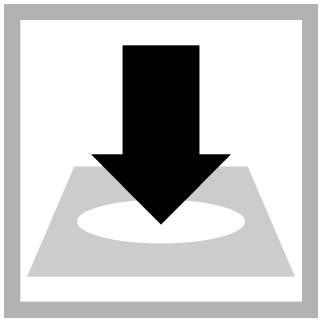

**7.** Rinse the sample cell with a small portion of the digested sample.




**8.** Pour 10 mL of the digested sample into the same sample cell.

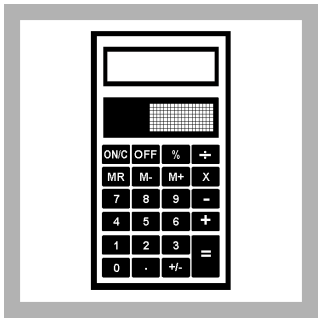


**9.** Clean the sample cell.



**10.** Insert the sample cell into the cell holder.


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




**12.** Subtract the color of the FerroZine Reagent Solution from the result to get the concentration of total iron in the sample. Refer to Measure the color of the reagent on page 3.

## Interferences

| Interfering substance | Interference level |
|---|---|
| Strong chelants (EDTA) | Interfere at all levels. |
| Cobalt | Can give slightly high results. |
| Copper | Can give slightly high results. |

## Accuracy check

### Standard additions method (blank spike)

Use the standard additions method (for applicable instruments) to validate the test procedure, the reagents and the instrument.
Items to collect:

- 1-mg/L Iron Standard Solution
- Pipet, adjustable volume, 0.2–1.0 mL
- Pipet tips

1. Use the test procedure to measure the concentration of the blank, then keep the (unspiked) blank 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. Insert the blank into the cell holder. Push **Zero**.
5. Prepare three spiked blanks: use a pipet to add 0.10 mL, 0.30 mL and 0.50 mL of the standard solution, respectively, to three 15-mL portions of fresh sample. Mix well.
6. Use the test procedure to measure the concentration of each of the spiked blanks. Start with the smallest blank spike. Measure each of the spiked blanks 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 blank spikes are measured accurately. The sample volumes and blank 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-mg/L Iron Standard Solution
- 6.0 N Hydrochloric Acid Standard Solution
- 500-mL volumetric flask, Class A
- 5-mL volumetric pipet, Class A and pipet filler safety bulb
- Deionized water

1. Prepare a 10-µg/L Fe standard solution as follows:
   a. Add about 300 mL of metal-free water to the flask.
   b. Use a pipet to add 1 mL of 6.0 N Hydrochloric Acid Standard Solution to the 500-mL volumetric flask.
   c. Use a pipet to add 5.00 mL of Iron Standard Solution to the 500-mL volumetric flask.
   d. Dilute to the mark with metal-free 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. The result should have a value of approximately 10-µg/L.

## 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<br>Concentration change per 0.010 Abs change |
|---|---|---|---|
| 269 | 10 µg/L Fe | 9.6–10.4 µg/L Fe | 8.7 µg/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. Use this method to find trace levels of iron in high purity water. This method may be used to analyze samples that contain insoluble iron oxides, which includes magnetite and hematite. These oxides are dissolved during the digestion procedure. The measurement wavelength is 562 nm.

## Consumables and replacement items

### Required reagents

| Description | Quantity/Test | Unit | Item no. |
|---|---|---|---|
| FerroZine® Iron Reagent Solution | 0.24 mL | 500 mL | 230149 |
| Water, deionized | varies | 4 L | 27256 |

### Required apparatus

| Description | Quantity/test | Unit | Item no. |
|---|---|---|---|
| Dropping bottle, assembly | 59 mL | 6/pkg | 2937606 |
| DRB200 Digital Reactor, 12 x 13 mm and 8 x 20 mm vial wells, 115 VAC | 1 | each | DRB200-04 |
| DRB200 Digital Reactor, 12 x 13 mm and 8 x 20 mm vial wells, 230 VAC | 1 | each | DRB200-08 |
| Digestion vials, 20-mm | 1 | 5/pkg | LZP065 |

### Recommended standards

| Description | Unit | Item no. |
|---|---|---|
| Iron Standard Solution, 1-mg/L Fe | 500 mL | 13949 |

### Optional reagents and apparatus

| Description | Unit | Item no. |
|---|---|---|
| Flask, volumetric, Class A, 500 mL, glass | each | 1457449 |
| Hydrochloric Acid Solution, 6 N (1:1) | 500 mL | 88449 |
| Pipet, adjustable volume, 0.2–1.0 mL | each | BBP078 |
| Pipet, adjustable volume, 1.0–5.0 mL | each | BBP065 |
| Pipets, adjustable volume, includes one 0.2–1.0 mL and one 1.0–5.0 mL pipet plus tips | each | LZP320 |

**Optional reagents and apparatus (continued)**

| Description | Unit | Item no. |
|---|---|---|
| Pipet tips, for 0.2–1.0 mL pipet | 100/pkg | BBP079 |
| Pipet tips, for 1.0–5.0 mL pipet | 75/pkg | BBP068 |
| Pipet, volumetric 5.00-mL | each | 1451537 |
| Pipet filler, safety bulb | each | 1465100 |

**FOR TECHNICAL ASSISTANCE, PRICE INFORMATION AND ORDERING:**
In the U.S.A. – **Call toll-free 800-227-4224**
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FAX: (970) 669-2932



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