Iron, Total

DOC316.53.01052

FerroMo Method[1] **Method 8365**

0.01 to 1.80 mg/L Fe **Powder Pillows**

Scope and application: For cooling water that contains molybdate-based treatment.

[1] Adapted from G. Frederick Smith Chemical Co., The Iron Reagents, 3rd ed. (1980).

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.
Wash all glassware with detergent. Rinse with tap water. Rinse again with 1:1 hydrochloric acid solution. Rinse a third time with high-quality deionized water. These steps will remove deposits that can cause slightly high results.
If the sample contains 100 mg/L or more molybdate (MoO_4^{2-}), read the sample immediately after the instrument zero.
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.

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Items to collect

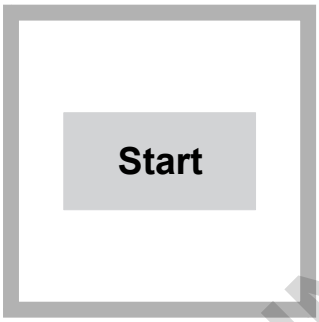
Description	Quantity
FerroMo® Reagent 1 Powder Pillow	1
FerroMo® Reagent 2 Powder Pillow	1
Cylinder, graduated mixing, 25-mL with stopper	1
Cylinder, graduated mixing, 50-mL with stopper	1
Sample cells (For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.)	2

Refer to Consumables and replacement items on page 5 for order information.

Sample collection and storage

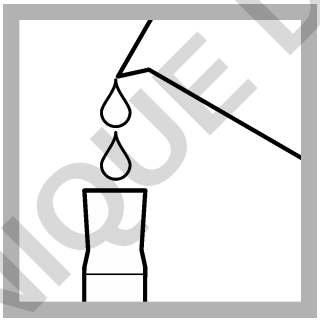
- Collect samples in clean glass or plastic bottles that have been cleaned with 6 N (1:1) hydrochloric acid and rinsed with deionized water.
- To preserve samples for later analysis, adjust the sample pH to less than 2 with concentrated hydrochloric acid (about 2 mL per liter). No acid addition is necessary if the sample is tested immediately.
- To measure only dissolved iron, filter the sample through a 0.45-micron filter or equivalent medium 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 N sodium 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.

Powder pillow procedure

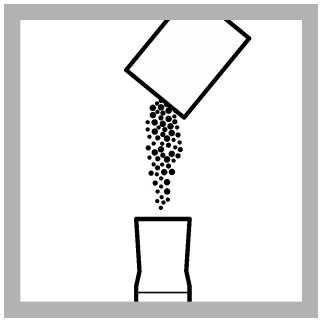


1. Start program **275 Iron, FerroMo**. For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.

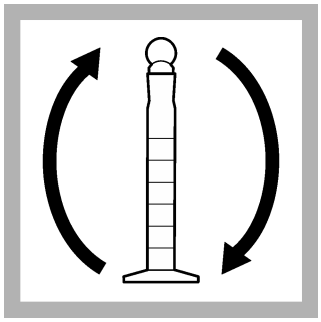
Note: *Although the program name can be different between instruments, the program number does not change.*



2. Prepare the sample: Fill a 50-mL mixing cylinder with 50 mL of sample.

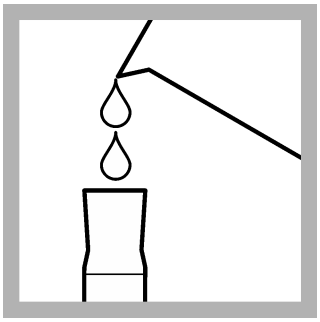


3. Add the contents of one FerroMo Iron Reagent 1 Powder Pillow to the mixing cylinder.

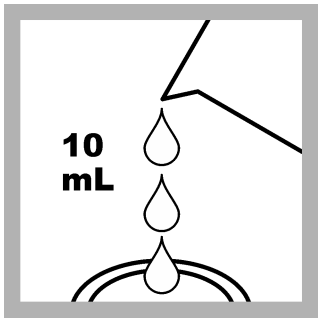


4. Close the cylinder. Invert several times to mix.

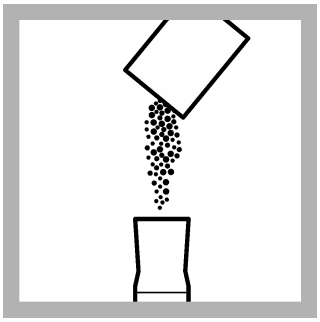
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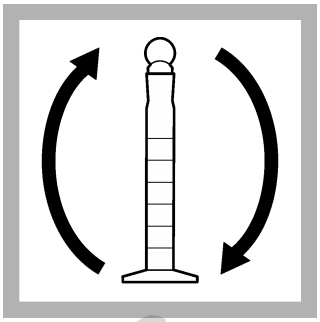
5. Fill a clean 25-mL mixing cylinder to the 25-mL mark with the prepared sample. Use the rest of the sample to prepare the blank.



6. Prepare the blank: Fill a second sample cell with 10 mL of the prepared sample.



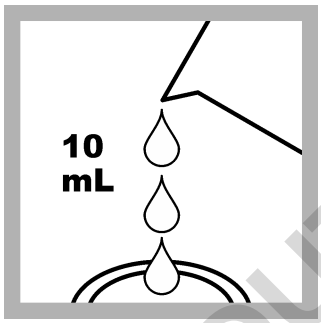
7. Develop the sample: Add the contents of one FerroMo Iron Reagent 2 Powder Pillow to the prepared sample in the 25-mL mixing cylinder.



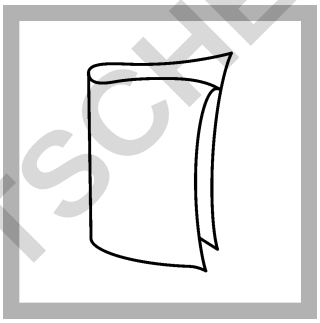
8. Put the stopper on the mixing cylinder. Invert the mixing cylinder several times to mix.
A blue color will show if iron is present in the sample. A small amount of undissolved reagent will not affect the results of the test.



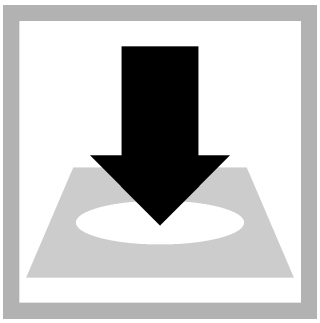
9. Start the instrument timer. A 3-minute reaction time starts.



10. When the timer expires, pour 10 mL of the developed sample into a sample cell. This is the prepared sample for the test.



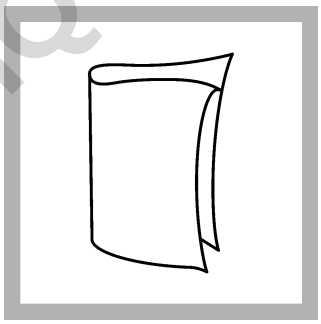
11. Clean the blank sample cell.



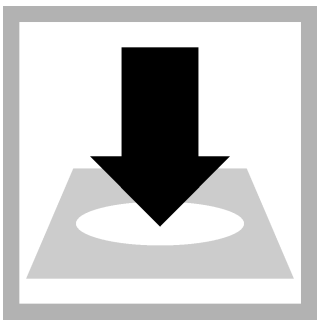
12. Insert the blank into the cell holder.



13. Push **ZERO**. The display shows 0.00 mg/L Fe.



14. Clean the developed sample.



15. Insert the developed sample into the cell holder.



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

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Interferences

Interfering substance	Interference level
pH	After the addition of reagent, a sample pH of less than 3 or more than 4 may inhibit color formation, cause the developed color to fade quickly or result in turbidity. Adjust the sample pH to between 3 and 8 in the graduated cylinder before the addition of reagent: **1.** Add by drops an applicable amount of iron-free acid or base such as 1 N sulfuric acid solution or 1 N sodium hydroxide solution. **2.** Make a volume correction if significant volumes of acid or base are used.

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, 50 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 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:

- 100 mg/L iron standard solution
- 100-mL volumetric flask, Class A
- 1-mL volumetric pipet, Class A and pipet filler
- Deionized water

1. Prepare a 1.00-mg/L iron standard solution as follows:
   a. Use a pipet to add 1.0 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.

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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
275	1.00 mg/L Fe	0.98–1.02 mg/L Fe	0.01 mg/L Fe

Summary of method

FerroMo Iron Reagent 1 contains a reducing agent combined with a masking agent. The masking agent removes interference from high levels of molybdate. The reducing agent converts precipitated or suspended iron, such as rust, to the ferrous state. FerroMo Iron Reagent 2 contains the indicator combined with a buffering agent. The indicator reacts with ferrous iron in the sample, buffered between pH 3 and 5, which results in a deep blue-purple color. The measurement wavelength is 590 nm for spectrophotometers or 610 nm for colorimeters.

Consumables and replacement items

Required reagents

Description	Quantity/test	Unit	Item no.
FerroMo® Iron Reagent Set	1	100/pkg	2544800
Includes:			
FerroMo®[1] Reagent 1 Powder Pillow	1	25/pkg	2543768
FerroMo® Reagent 2 Powder Pillow	1	50/pkg	2543866

[1] FerroMo is a registered trademark of Hach Company.

Required apparatus

Description	Quantity/test	Unit	Item no.
Mixing cylinder, graduated, 25-mL with stopper	1	each	2088640
Mixing cylinder, graduated, 50-mL, with stopper	1	each	2088641

Recommended standards

Description	Unit	Item no.
Iron Standard Solution, 100-mg/L Fe	100 mL	1417542
Iron Standard Solution, 1-mg/L Fe	500 mL	13949
Iron Standard Solution, 10-mL Voluette® Ampule, 50-mg/L Fe	16/pkg	1425410
Water, deionized	4 L	27256

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Optional reagents and apparatus

Description	Unit	Item no.
Flask, volumetric, Class A, 100-mL glass	each	1457442
Pipet, volumetric, Class A, 1.00-mL	each	1451535
Pipet filler, safety bulb	each	1465100
Sodium Hydroxide Standard Solution, 1.0 N	100 mL MDB	104532
Sodium Hydroxide Standard Solution, 5.0 N	100 mL MDB	245032
Sulfuric Acid Standard Solution, 1 N	100 mL MDB	127032
Hydrochloric Acid Solution, 6.0 N (1:1)	500 mL	88449

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