

1,10-Phenanthroline Method¹

Method 8146
0.02 to 3.00 mg/L Fe²⁺
Powder Pillows or AccuVac[®] Ampuls

Scope and application: For water, wastewater, seawater, brine solutions, produced waters and hydraulic fracturing waters.

¹ Adapted from Standard Methods for the Examination of Water and Wastewater, 15th ed. 201 (1980).



Test preparation

Instrument-specific information

Table 1 shows sample cell and orientation requirements for reagent addition tests, such as powder pillow or bulk reagent tests. Table 2 shows sample cell and adapter requirements for AccuVac Ampul tests. The tables also show all of the instruments that have the program for this test.

To use the table, select an instrument, then read across to find the applicable information for this test.

Table 1 Instrument-specific information for reagent addition





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 

Table 2 Instrument-specific information for AccuVac Ampuls

Instrument	Adapter	Sample cell
DR 6000 DR 5000 DR 900	—	2427606 
DR 3900	LZV846 (A)	
DR 1900	9609900 or 9609800 (C)	
DR 3800 DR 2800 DR 2700	LZV584 (C)	2122800 

Before starting

Samples must be analyzed immediately after collection and cannot be preserved for later analysis.

Install the instrument cap on the DR 900 cell holder before ZERO or READ is pushed.

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.

Items to collect

Powder pillows

Description	Quantity
Ferrous Iron Reagent Powder Pillows, 25-mL	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 6 for order information.

AccuVac Ampuls

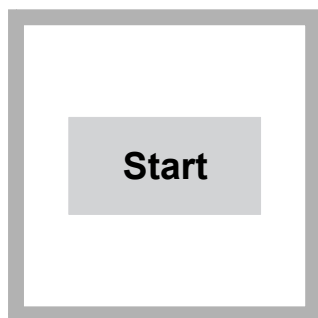
Description	Quantity
Ferrous Iron Reagent AccuVac Ampuls	1
Beaker, 50-mL	1
Sample cells (For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.)	1
Stopper for 18-mm tubes and AccuVac Ampuls	1

Refer to [Consumables and replacement items](#) on page 6 for order information.

Sample collection

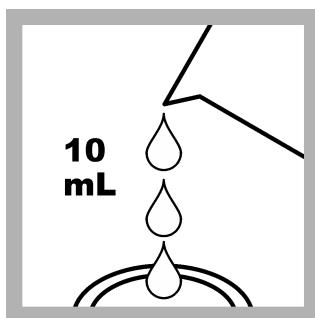
- Samples must be analyzed immediately after collection and cannot be preserved for later analysis.
- Collect samples in clean glass or plastic bottles with tight-fitting caps. Completely fill the bottle and immediately tighten the cap.
- Prevent agitation of the sample or exposure to air.

Powder pillow procedure

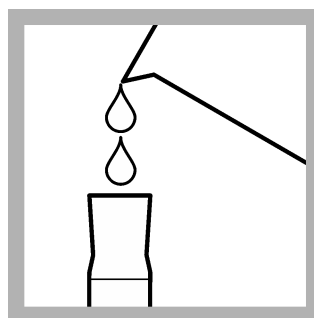


1. Start program **255 Iron, Ferrous**. For information about sample cells, adapters or light shields, refer to [Instrument-specific information](#) on page 1.

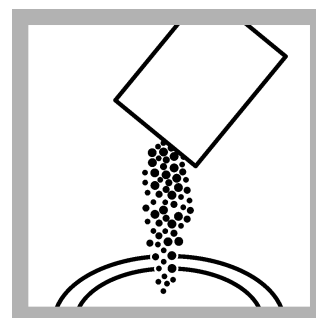
Note: Although the program name can be different between instruments, the program number does not change.



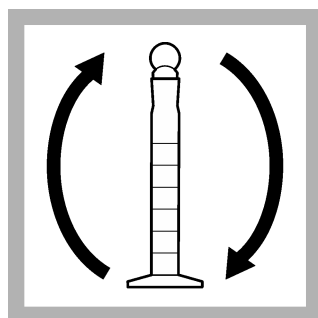
2. **Prepare the blank:** Fill the sample cell with 10 mL of sample.



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



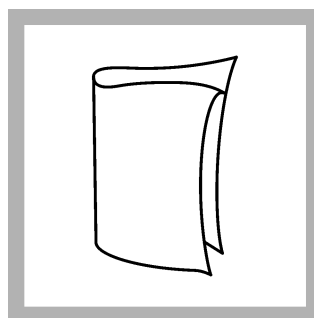
4. Add the contents of one Ferrous Iron Reagent Powder Pillow to the mixing cylinder. An orange color shows if ferrous iron is present in the sample.



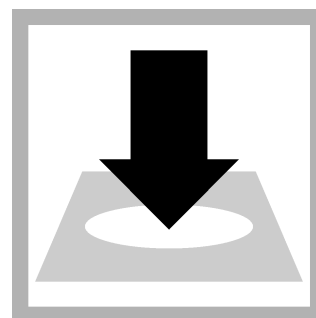
5. Put the stopper on the mixing cylinder. Invert the mixing cylinder several times to mix. Undissolved powder does not affect accuracy.



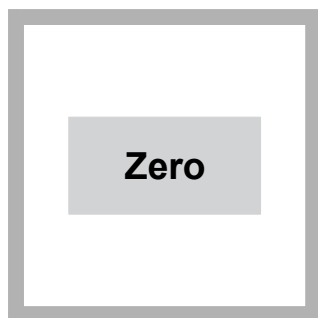
6. Start the instrument timer. A 3-minute reaction time starts.



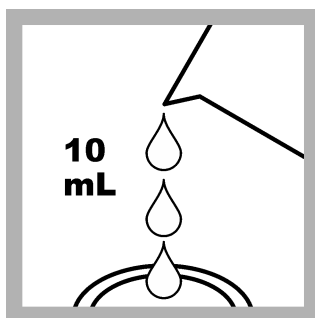
7. When the timer expires, clean the blank sample cell.



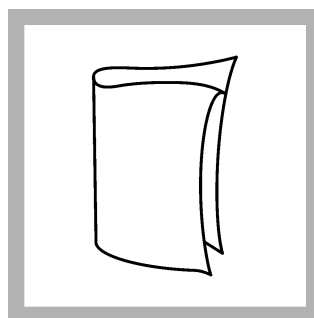
8. Insert the blank into the cell holder.



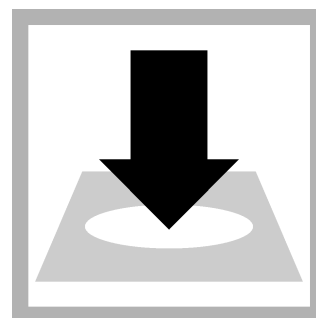
9. Push **ZERO**. The display shows 0.00 mg/L Fe^{2+} .



10. Fill a second sample cell with 10 mL of the reacted prepared sample.



11. Clean the prepared sample cell.



12. Insert the prepared sample into the cell holder.



Read

13. Push **READ**. Results show in mg/L Fe²⁺.

AccuVac Ampul procedure



Start

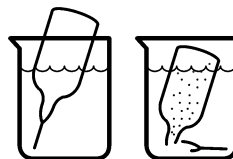
1. Start program **257 Iron, Ferrous AV**. For information about sample cells, adapters or light shields, refer to [Instrument-specific information](#) on page 1.

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

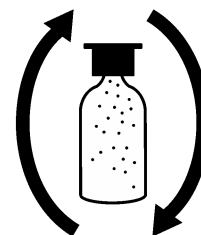


10 mL

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



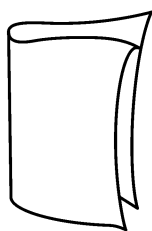
3. **Prepare the sample:** Collect at least 40 mL of sample in a 50-mL beaker. Fill the AccuVac Ampul with sample. Keep the tip immersed while the AccuVac Ampul fills completely.



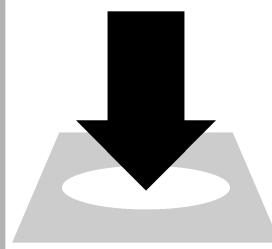
4. Quickly invert the AccuVac Ampul several times to mix.



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



6. When the timer expires, clean the blank sample cell.

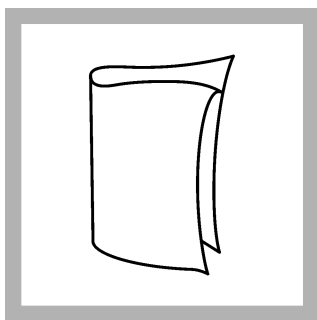


7. Insert the blank into the cell holder.

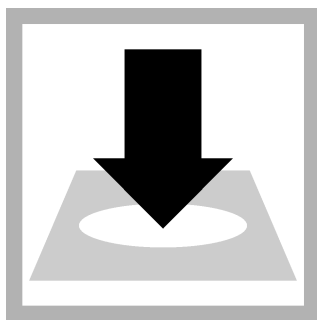


Zero

8. Push **ZERO**. The display shows 0.00 mg/L Fe²⁺.



9. Clean the AccuVac Ampul.



10. Insert the prepared sample AccuVac Ampul into the cell holder.



11. Push **READ**. Results show in mg/L Fe²⁺.

Accuracy check

Standard solution method

Use the standard solution method to validate the test procedure, the reagents and the instrument.

Items to collect:

- Ferrous Ammonium Sulfate, hexahydrate
- 1-L volumetric flask, Class A
- 100-mL volumetric flask, Class A
- 2-mL volumetric pipet, Class A and pipet filler
- Deionized water

1. Prepare a 100-mg/L Fe²⁺ ferrous iron stock solution as follows:
 - a. Add 0.7022 g of ferrous ammonium sulfate, hexahydrate into a 1-L volumetric flask.
 - b. Dilute to the mark with deionized water. Mix well.
2. Prepare a 2-mg/L ferrous iron standard solution as follows:
 - a. Use a pipet to add 2.00 mL of the 100-mg/L Fe²⁺ ferrous iron stock solution into a 100-mL volumetric flask.
 - b. Dilute to the mark with deionized water. Mix well. Prepare the standard solution immediately before use.
3. Use the test procedure to measure the concentration of the prepared standard solution.
4. 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
255	2.00 mg/L Fe ²⁺	1.99–2.01 mg/L Fe ²⁺	0.021 mg/L Fe ²⁺
257	2.00 mg/L Fe ²⁺	1.98–2.02 mg/L Fe ²⁺	0.023 mg/L Fe ²⁺

Summary of method

The 1,10-phenanthroline indicator in the Ferrous Iron Reagent reacts with ferrous iron (Fe^{2+}) in the sample to form an orange color in proportion to the iron concentration. Ferric iron (Fe^{3+}) does not react. The ferric iron concentration can be determined by subtracting the ferrous iron concentration from the results of a total iron test. The measurement wavelength is 510 nm for spectrophotometers or 520 nm for colorimeters.

Consumables and replacement items

Required reagents

Description	Quantity/test	Unit	Item no.
Ferrous Iron Reagent Powder Pillow, 25-mL	1	100/pkg	103769
OR			
Ferrous Iron Reagent AccuVac [®] Ampul	1	25/pkg	2514025

Required apparatus

Description	Quantity/test	Unit	Item no.
Beaker, 50-mL	1	each	50041H
Stoppers for 18-mm tubes and AccuVac Ampul	1	6/pkg	1448000

Recommended standards and apparatus

Description	Unit	Item no.
Balance, analytical, 80 g x 0.1 mg 100–240 VAC	each	2936701
Ferrous Ammonium Sulfate, hexahydrate, ACS	113 g	1125614
Flask, volumetric, Class A, 1000-mL glass	each	1457453
Pipet filler, safety bulb	each	1465100
Pipet, volumetric, Class A, 1.00-mL	each	1451535
Water, deionized	4 L	27256
Wipes, disposable	280/pkg	2097000



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