Iron, Total

DOC316.53.01051

TPTZ Method[1] **Method 8112**

0.012 to 1.800 mg/L Fe (spectrophotometers) **Powder Pillows**

0.04 to 1.80 mg/L Fe (colorimeters)

Scope and application: For water, wastewater and seawater.

[1] Adapted from G. Frederic 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
DR6000 DR3800 DR2800 DR2700 DR1900	The fill line is to the right.	2495402 
DR5000 DR3900	The fill line is toward the user.	
DR900	The orientation mark is toward the user.	2401906 

Before starting

Install the instrument cap on the DR900 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.
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

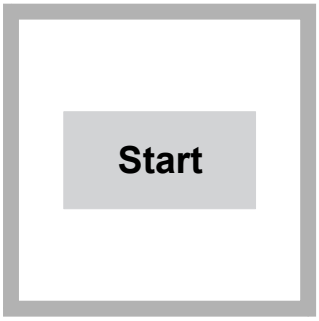
Description	Quantity
TPTZ Iron Reagent Powder Pillows, 10 mL	1
Sample cells. (For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.)	2

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

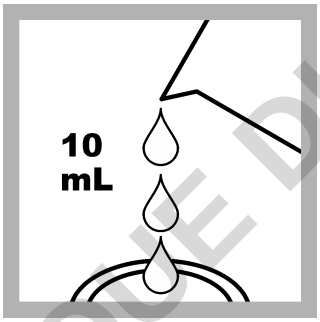
Sample collection and storage

- 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 3–4 with 5 N sodium hydroxide standard solution. Do not exceed pH 5 or iron may precipitate.
- Correct the test result for the dilution caused by the volume additions.

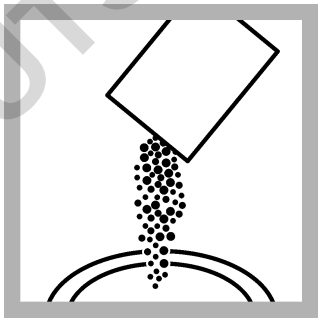
Test procedure



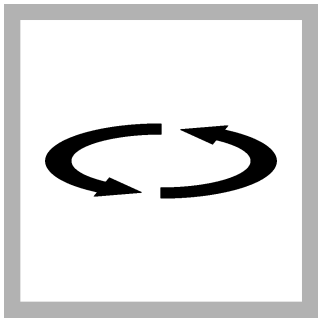
1. Start program **270 Iron, TPTZ**. For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.



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



3. Add the contents of one 10-mL TPTZ Iron Reagent Powder Pillow to the prepared sample.

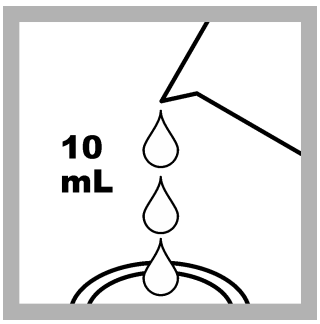


4. Swirl the sample cell for at least 30 seconds to mix.

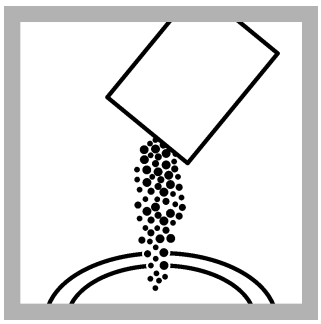


5. Start the instrument timer. A 3-minute reaction time starts.

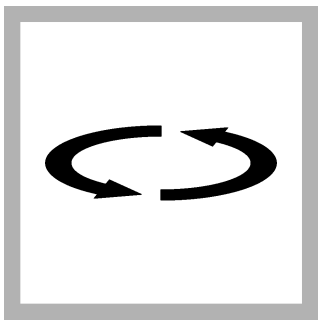
Prepare the blank during the reaction time.



6. Prepare the blank: Fill a sample cell with 10 mL of deionized water.

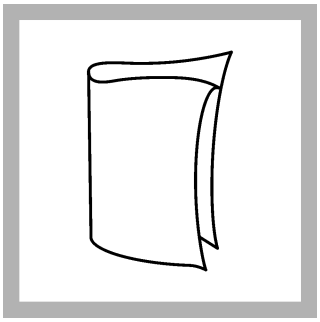


7. Add the contents of the contents of one 10-mL TPTZ Iron Reagent Powder Pillow to the blank.

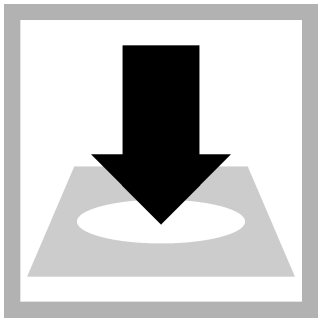


8. Swirl the sample cell for at least 30 seconds to mix.

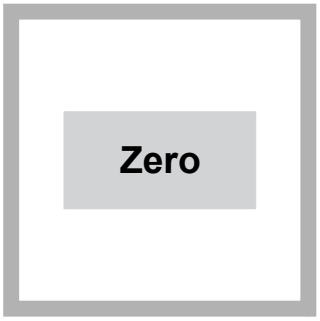
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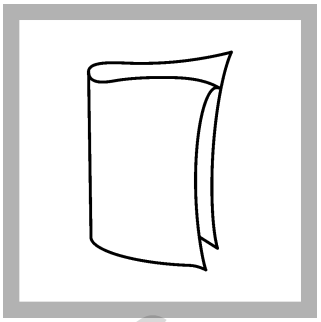
9. When the timer expires, clean the blank sample cell.



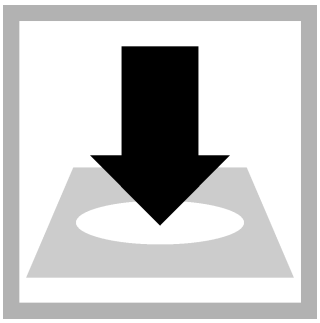
10. Insert the blank into the cell holder.



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



12. Clean the prepared sample cell.



13. Insert the prepared sample into the cell holder.



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

Interferences

Interferences were tested with an iron concentration of 0.5 mg/L Fe. The following do not interfere with this method when present up to the levels shown.

Interfering substance	Interference level
Cadmium	4.0 mg/L
Chromium^{3+}	0.25 mg/L
Chromium^{6+}	1.2 mg/L
Cobalt	0.05 mg/L
Copper	0.6 mg/L
Cyanide	2.8 mg/L
Manganese	50.0 mg/L
Mercury	0.4 mg/L
Molybdenum	4.0 mg/L
Nickel	1.0 mg/L
Nitrite Ion	0.8 mg/L

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Interfering substance	Interference level
Color or turbidity	If the sample, without a TPTZ Iron Reagent Powder Pillow, has a color or turbidity more than the blank (deionized water plus TPTZ Iron Reagent), then use the sample as the blank. Refer to the powder pillow procedure.
pH	After the addition of reagent, a sample pH of less than 3 or more than 4 may inhibit color formation. The developed color fades quickly or causes turbidity. Adjust the sample pH in the sample cell before the addition of reagent: **1.** Use a pH meter or pH paper to measure the current pH. **2.** Add an applicable amount of iron-free acid or base such as 1.0 N Sulfuric Acid Standard Solution or 1.0 N Sodium Hydroxide Standard Solution to adjust the sample pH to between 3 and 4.[1] **3.** 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 Standard Solution, 10 mg/L Fe
- Pipet, TenSette®, 0.1–1.0 mL and pipet tips
- Mixing cylinders, 10 mL (3)

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 10-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
- 500-mL volumetric flask, Class A
- 5-mL volumetric pipet, Class A and pipet filler safety bulb
- Deionized water

1. Prepare a 1.00 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.

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

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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 calibration 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 small 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
270	1.000 mg/L Fe	0.989–1.011 mg/L Fe	0.011 mg/L Fe

Summary of method

The TPTZ Iron Reagent forms a deep blue-purple color with ferrous iron (Fe^{2+}). The indicator is combined with a reducing agent that converts precipitated or suspended iron, such as rust, to the ferrous state. The amount of ferric iron (Fe^{3+}) can be determined as the difference between the results of a ferrous iron test and the concentration of total iron. 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.
TPTZ Iron Reagent Powder Pillow, 10 mL	1	100/pkg	2608799

Required apparatus

Description	Quantity/test	Unit	Item no.
Sample cells, 10-mL square, matched pair	2	2/pkg	2495402

Recommended standards

Description	Unit	Item no.
Iron Standard Solution, 100-mg/L Fe	100 mL	1417542
Iron Standard Solution, 10-mg/L Fe	500 mL	14049
Iron Standard Solution, 1-mg/L Fe	500 mL	13949
Metals Drinking Water Standard, LR for Cu, Fe, Mn	500 mL	2833749
Metals Drinking Water Standard, HR for Cu, Fe, Mn	500 mL	2833649
Water, deionized	4 L	27256

Optional reagents and apparatus

Description	Unit	Item no.
Mixing cylinder, graduated, 50 mL	each	189641
Nitric Acid, concentrated	500 mL	15249

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Optional reagents and apparatus (continued)

Description	Unit	Item no.
Sodium Hydroxide Solution, 5 N	50 mL	245026
Sulfuric Acid Standard Solution, 1 N	100 mL MDB	127032
Sodium Hydroxide Standard Solution, 1.0 N	100 mL MDB	104532
Pipet, TenSette®, 0.1–1.0 mL	each	1970001
Pipet tips for TenSette® Pipet, 0.1–1.0 mL	50/pkg	2185696
Flask, volumetric, Class A, 500 mL, glass	each	1457449
Pipet, volumetric 5.00-mL	each	1451537
Pipet filler, safety bulb	each	1465100

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