

FerroZine® Rapid Liquid Method¹

Method 8147 Pour-Thru Cell

0.009 to 1.400 mg/L Fe

Scope and Application: For boiler, cooling and natural waters

¹ Adapted from Stookey, L.L., *Anal.Chem.*, 42 (7) 779 1970.



Test preparation

How to use instrument-specific information

The *Instrument-specific information* table displays requirements that may vary between instruments. To use this table, select an instrument then read across to find the corresponding information required to perform this test.

Table 202 Instrument-specific information

Instrument	Pour-thru Kit	Cell orientation	Adapter
DR 6000	LQV175.99.20002	Arrow faces right	_
DR 5000	LZV479	_	_
DR 3900	LQV157.99.10002	Align cell flow arrows with arrows on cell compartment	_
DR 3800, DR 2800, DR 2700	5940400	1-inch (round) path aligned with arrow on the adapter	LZV585 (B)

Before starting the test:

If sample contains rust, see Interferences.

Digestion is required for total iron determination.

For more accurate results, determine a reagent blank value for each new lot of reagent. Follow the procedure using deionized water in place of the sample. Subtract the reagent blank value from the final results or perform a reagent blank adjust.

If iron is present, a purple color will form after adding the reagent

Rinse glassware with a 1:1 Hydrochloric acid¹ solution. Rinse again with deionized water. This will remove residual iron that may interfere

Prepare the Pour-Thru cell by preparing a solution of 1 mL Ferrozine Reagent per 50 mL of deionized water. Pour this into the cell and allow to stand for approximately 5 minutes to react with any trace iron in the cell and cell tubing. Flush with deionized water.

FerroZine Iron Reagent may crystallize or precipitate when exposed to cold temperatures during shipment; reagent quality is not affected. Place the reagent bottle in warm water to dissolve.

Make sure the Pour-Thru cell is completely seated in the sample cell compartment.

¹ See Optional reagents and apparatus.

Collect the following items:

Description	Quantity
FerroZine® Iron Reagent Solution	1.0 mL
Water, deionized	varies
Cylinder, graduated, 50 mL poly	1
Dispenser, fixed volume, 1.0 mL, with bottle	1
Flask, Erlenmeyer, PMP w/cap, 125 mL	2
Pour-Thru Cell Module (see Instrument-specific information)	1

See Consumables and replacement items for reorder information.

FerroZine Rapid Liquid Method for Pour-Thru Cell



1. Select the test.

Insert an adapter if required (see *Instrument-specific information*).

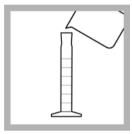
Refer to the user manual for orientation.



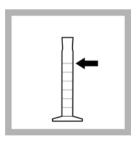
2. Flush the Pour-Thru Cell with 50 mL of deionized water.



3. Rinse two clean 125 mL Erlenmeyer flasks with the sample three times.



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



5. Fill the rinsed cylinder to the 50 mL mark with sample.



6. Pour the contents of the 50 mL cylinder into one of the flasks.



7. Prepared sample: Add 1.0 mL of FerroZine Iron Reagent Solution to flask using the Dispenser. Swirl to mix.



8. Start the instrument timer.

A five-minute reaction period will begin.

FerroZine Rapid Liquid Method for Pour-Thru Cell (continued)

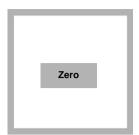


9. Blank preparation:
Measure a second 50 mL
portion of sample into the
graduated cylinder and
pour the contents into the
second flask.



10. When the timer expires the display will show "mg/L Fe."

Pour the contents of the flask containing the blank into the Pour-Thru Cell



11. When the flow stops, **ZERO** the instrument.

The display will show:

0.000 mg/L Fe



12. Pour the contents of the flask containing the prepared sample into the Pour-Thru Cell.



13. When the flow stops, **READ** the results in mg/L Fe.



14. Flush the Pour-Thru Cell with at least 50 mL of deionized water immediately after use.

Interferences

Table 203 Interfering substances

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	
Hydroxides	Boil the sample with the FerroZine® Iron Reagent added to it from step 7 of the test procedure for 1 minute in a boiling water bath. Cool to 24 °C (75 °F) before proceeding with step 9. Return the sample volume to 50 mL with deionized water.	
Magnetite (black iron oxide) or Ferrites	 Fill a 50 mL graduated cylinder with 50 mL of sample. Transfer the sample into a clean glass 125 mL Erlenmeyer flask. Add 1.0 mL of FerroZine Iron Reagent Solution¹ and swirl to mix. Place the flask on a hot plate or over a flame and bring to a boil. Continue boiling gently for 20 to 30 minutes. <i>Do not allow to boil dry.</i> Return the boiled sample to the graduated cylinder. Rinse the Erlenmeyer flask with small amounts of deionized water and empty into the graduated cylinder. A purple color will develop if iron is present. Return the sample volume to the 50 mL mark with deionized water. Pour the solution into a 125 mL Erlenmeyer flask and swirl to mix. Proceed with steps 9–14 of the test procedure. 	
Rust	Boil the sample, with the FerroZine® Iron Reagent added to it from step 7, for 1 minute in a boiling water bath. Cool to 24 °C (75 °F) before proceeding with step 9. Return the sample volume to 50 mL with deionized water.	

¹ See Optional reagents and apparatus.

Sample collection, preservation and storage

- · Collect samples in acid-washed glass or plastic bottles.
- To preserve samples, adjust the sample pH to 2 or less with concentrated Nitric Acid, ACS*
 (about 2 mL per liter). Samples preserved in this manner can be stored up to six months at room temperature.
- If only reporting dissolved iron, filter the sample immediately after collection and before adding nitric acid.
- Before testing, adjust the sample pH to 3–5 with Ammonium Hydroxide, 10%. Do not exceed pH 5 or iron may precipitate.
- Correct test results for volume additions. Refer to the Water Analysis Guide for more information.

^{*} See Optional reagents and apparatus.

Labware

- All containers used in this test must be cleaned thoroughly to remove any traces of iron.
- 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. If containers are rinsed and capped after each use, only occasional treatment with HCl or FerroZine[®] is necessary.

Cleaning the Pour-Thru Cell

The Pour-Thru Cell may accumulate a buildup of color products, especially if the reacted solutions are allowed to stand in the cell for long periods after measurement. Remove the color by rinsing with a 1:5 dilution of Ammonium Hydroxide*, followed by several rinses with deionized water. Cover the Pour-Thru Cell after use.

Accuracy check

Standard additions method (sample spike)

Required for accuracy check:

- Iron Voluette® Ampule Standard, 25 mg/L Fe
- · Ampule breaker
- · TenSette Pipet and Pipet Tips
- Graduated mixing cylinder, 50 mL (3)
- 1. After reading test results, leave the sample cell (unspiked sample) in the instrument.
- 2. Select Options>More>Standard Additions from the instrument menu.
- Accept the default values for standard concentration, sample volume and spike volumes. After the values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.
- 4. Open the standard solution ampule.
- Use the TenSette Pipet to prepare spiked samples: add 0.2 mL, 0.4 mL and 0.6 mL of standard to three 50 mL portions of fresh sample.
- Follow the FerroZine Rapid Liquid Method for Pour-Thru Cell test procedure for each of the spiked samples starting with the 0.2 mL sample spike. Measure each of the spiked samples in the instrument.
- Select GRAPH to view the results. Select IDEAL LINE (or best-fit) to compare the standard addition results to the theoretical 100% recovery. Each addition should reflect approximately 100% recovery.

Standard solution method

Note: Refer to the instrument user manual for specific software navigation instructions.

Required for accuracy check:

- 1.0 mg/L Iron Standard Solution or
- 100 mg/L Iron Standard Solution
- · Deionized water
- 500 mL Class A volumetric flask

- 5 mL Class A volumetric pipet and filler
- To check the accuracy, use a 1.0 mg/L Iron Standard Solution or prepare a 1.0 mg/L iron working solution as follows:
 - a. Pipet 5.00 mL of iron standard solution, 100 mg/L Fe, into a 500 mL volumetric flask.
 - b. Dilute to volume with deionized water. Prepare this solution daily.
- 2. Follow the FerroZine Rapid Liquid Method for Pour-Thru Cell test procedure.
- 3. To adjust the calibration curve using the reading obtained with the 1.00-mg/L Standard Solution, select Options>More>Standard Adjust from the instrument menu.
- 4. Turn on the Standard Adjust feature and accept the displayed concentration. If an alternate concentration is used, enter the concentration and adjust the curve to that value. Mixed-parameter standards are also available to simulate various test matrices.

Method performance

Program	Standard	Precision 95% Confidence Limits of Distribution	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 colored complex with trace amounts of iron in samples that are buffered to a pH of 3.5. This method is applicable for determining trace levels of iron in chemical reagents and glycols and with digestion can be used to analyze samples containing magnetite (black iron oxide) or ferrites. The test results are measured at 562 nm.

Consumables and replacement items

Required reagents

Description	Quantity/Test	Unit	Catalog number
FerroZine® Iron Reagent Solution	1 mL	500 mL	230149
Water, deionized	varies	4 L	27256

Required apparatus

Description	Quantity	Unit	Catalog number
Cylinder, graduated, 50-mL, poly	1	each	108141
Dispenser, fixed volume, 1.0-mL	1	each	2111302
Flask, Erlenmeyer, PMP w/cap, 125-mL	2	each	2089843

Recommended standards and apparatus

Description	Unit	Catalog number
Flask, volumetric, Class A, 500 mL	each	1457449
Iron Standard Solution, 100-mg/L Fe	100 mL	1417542
Iron Standard Solution, Voluette® ampule, 25-mg/L Fe, 10-mL	16/pkg	1425310
Iron Standard Solution, 1 mg/L Fe	500 mL	13949
Metals Drinking Water Standard, LR for Cu, Fe, Mn	500 mL	2833749
Pipet, TenSette 0.1–1.0 mL	each	1970001
Pipet Tips, for TenSette Pipet 1970001	50/pkg	2185696
Pipet Tips, for TenSette Pipet 1970001	1000/pkg	2185628
Pipet, volumetric, Class A, 5.00 mL	each	1451537
Pipet Filler, safety bulb	each	1465100

Optional reagents and apparatus

Description	Unit	Catalog number
Ammonium Hydroxide, ACS, 58%	500 mL	10649
Ammonium Hydroxide, ACS, 10%	100 mL MDB	1473632
Hydrochloric Acid, 1:1, 6N	500 mL	88449
Nitric Acid ACS, concentrated	500 mL	15249

