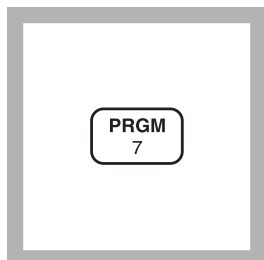


**Scope and Application:** For oil and gas field waters; digestion is required for determining total iron  
USEPA approved for reporting wastewater analysis (digestion is required)

## Test procedure



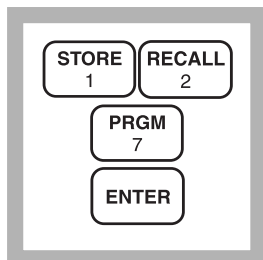
**1. Push: PRGM**

The display shows:

**PRGM ?**

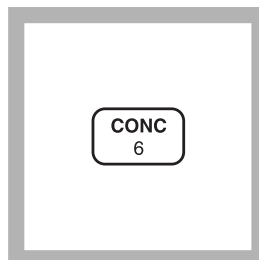
Initial setup: go to “Instrument setup” on page 4 to add the program to the instrument.

**Note:** Adjust the pH of stored samples before analysis.



**2. Push: 127 ENTER**

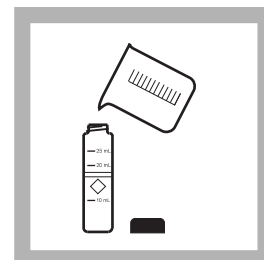
The display shows **mg/L, Fe LR, Fe MR or Fe HR** and the **ZERO** icon.



**3. Push: CONC** to select the test range.

**Fe LR 0–3.0 mg/L Fe  
Fe MR 0–30.0 mg/L Fe  
Fe HR 0–300.0 mg/L Fe**

Identify test ranges: First analyze samples with unknown iron concentrations with the Fe HR test. If no iron is found in step 13, analyze the samples with the MR and LR test ranges.



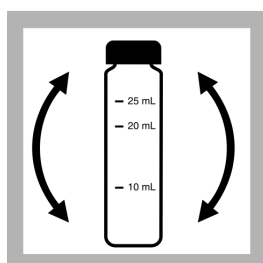
**4. Add the specified sample volume to a clean sample cell.**

**Fe LR: 10 mL  
Fe MR: 1.0 mL  
Fe HR: 0.1 mL**

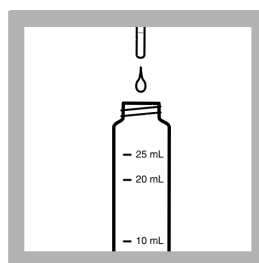
Use a TenSette or glass pipet to measure the 1.0 or 0.1 mL sample sizes.



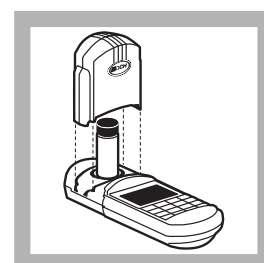
**5. If the sample volume is less than 10 mL, fill the sample to the 10 mL line with deionized or iron-free water.**



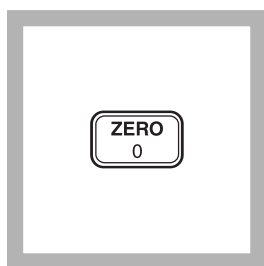
**6. Tighten the cap on the sample cell and invert to mix.**



**7. Remove the cap from the sample cell and add 2 drops of 1 M EDTA Solution to the sample. Tighten the cap on the sample cell and invert to mix.**



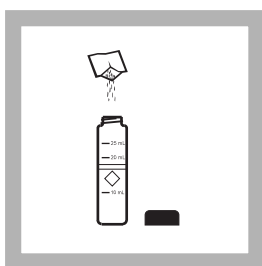
**8. Put the sample cell into the cell holder. Put the instrument cap over the sample cell.**



**9. Push: ZERO**

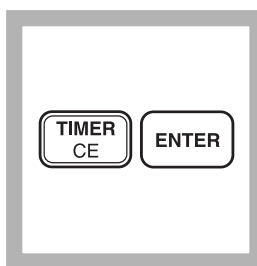
The cursor moves to the right, then the display shows:

**0.0 mg/L Fe LR, Fe MR or Fe HR**



**10. Remove the cap and add the contents of one FerroVer Iron Reagent Powder Pillow to the sample cell. Tighten the cap on the sample cell and invert to mix.**

The accuracy is not affected by undissolved powder.

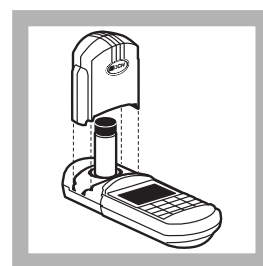


**11. Push: TIMER ENTER**

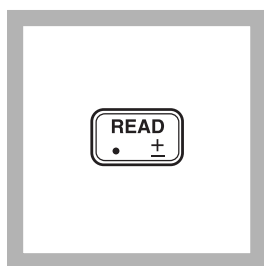
A three-minute reaction period starts.

An orange color will form if iron is present.

If the sample contains visible rust or if five drops of EDTA were used in step 7, the sample should be allowed to react for at least five minutes.



**12. Put the prepared sample into the cell holder. Put the instrument cap over the sample cell.**



**13. Push: READ**

The cursor will move to the right, then the result for the selected test range in mg/L iron (Fe) is shown.

**Notice!** Do not push the CONC key at the end of the test to change the range. The result is applicable only to the test range that was selected in step 3.

**Note:** For best results use the Standard Adjust option. Refer to the procedures manual for more information about Standard Adjust.

---

## Sampling and Storage

Collect samples in acid-cleaned glass or plastic containers. No acid addition is necessary if the sample is analyzed immediately. To preserve samples, adjust the pH to 2 or less with nitric acid (about 2 mL per liter). Keep preserved samples in a storage up to six months at room temperature. Adjust the pH to between 3 and 5 with 5.0 N Sodium Hydroxide Standard Solution before analysis. Correct the test result for volume additions; refer to Section 1 *Correcting for Volume Additions* of the procedures manual for more information. Filter the sample before the acid addition to find dissolved iron only.

## Accuracy Check

### Standard Additions Method

1. Fill three sample cells with sample as specified in steps 4 and 5 of the test procedure.
2. Open a 25 mg/L Iron Voluette Ampule Standard Solution.

3. Use the TenSette Pipet to add 0.1, 0.2, and 0.3 mL of the 25 mg/L standard, respectively, to the samples and mix fully.
4. Complete the test procedure for each sample.
5. Examine the results. The iron concentration should increase by 0.25 mg/L for Fe LR, 2.5 mg/L for Fe MR and 25.0 mg/L for Fe HR for each 0.1 mL of standard that is added.
6. If these increases do not occur, refer to Section 1 *Standard Additions* of the procedures manual for troubleshooting information.

#### Standard Solution Method

Prepare a 1.0-mg/L iron standard solution.

- Dilute 1.00 mL of Iron Standard Solution, 100 mg/L Fe, to 100 mL with deionized water.

Use 10 mL of the standard in step 4 to complete the Fe LR procedure for powder pillows. Results should be between 0.90 mg/L and 1.10 mg/L Fe.

### Method Performance

#### Precision

In a single laboratory, with a standard solution of 2.0 mg/L Fe and two of powder pillows with the instrument, a single operator got a standard deviation of  $\pm 0.02$  mg/L Fe.

#### Estimated Detection Limit (EDL)

The EDL for program 127 is 0.1 mg/L Fe. For more information on derivation and use of the estimated detection limit, refer to Section 1 of the procedures manual.

### Interferences

#### Interfering Substances and Suggested Treatments

Interfering Substance	Interference Level and Treatment
Barium, Ba <sup>2+</sup>	The dilution of samples to measure iron lowers most barium concentrations below interference levels. No effects are seen on analyzed solutions which contain less than 50 mg/L of Ba. No effects are seen when 1.0 or 0.1 mL of sample is used in step 4. A turbidity may show at higher levels. Add 5 drops of EDTA Solution to the solution in step 5 and do the analysis again. Allow the sample to react for 5 minutes.
Calcium, Ca <sup>2+</sup>	No effect at less than 10,000 mg/L as CaCO <sub>3</sub>
Chloride, Cl <sup>-</sup>	No effect at less than 185,000 mg/L.
Copper, Cu <sup>2+</sup>	No effect. Masking agent is contained in FerroVer Iron Reagent.
High Iron Levels	Prevents color development. Dilute the sample and test again to verify results.
Magnesium	No effect at 100,000 mg/L as CaCO <sub>3</sub> .
Molybdate, Molybdenum	No effect at 25 mg/L as Mo.
High Sulfide Levels, S <sup>2-</sup>	<ol style="list-style-type: none"> <li>1. Prepare in fume hood or well-ventilated area. Add 5 mL HCl to 100 mL sample in a 250-mL Erlenmeyer flask. Boil for 20 minutes.</li> <li>2. Cool. Adjust the pH to 3–5 with NaOH. Adjust the volume to 100 mL with deionized water.</li> <li>3. Analyze.</li> </ol>
Strontium, Sr <sup>2+</sup>	Strontium by itself does not interfere. Strontium in combination with Barium will cause a precipitate to form. The dilution of samples to measure iron lowers most strontium concentrations below interference levels. No effects are seen on analyzed solutions which contain less than 50 mg/L of combined Ba and Sr. No effects are seen when 1.0 or 0.1 mL of sample is used in step 4. A turbidity may start at higher levels. Add 5 drops of EDTA Solution to the solution in Step 5 and do the analysis again. Allow the sample to react for 5 minutes
Sample pH (extreme)	Adjust pH to 3-5. Refer to <i>Interferences</i> in Section 1.

Interfering Substance	Interference Level and Treatment
Highly Buffered Samples	Adjust pH to 3-5. Refer to <i>Interferences</i> in <i>Section 1</i> .

### Summary of Method

FerroVer Iron Reagent reacts with all soluble iron and most insoluble forms of iron in the sample to produce soluble ferrous iron. Ferrous iron reacts with 1,10-phenanthroline indicator in the reagent to form an orange color in proportion to the iron concentration.

### Instrument setup

This procedure adds the program 127 to a DR/820, DR/850 or DR/890 instrument.

1. Push the **ON** key to turn on the instrument.
2. Push the **SETUP** key.
3. Push the down arrow key until the prompt line shows USER.
4. Push the **ENTER** key.
5. Push the numbers **8138**, then push **ENTER**.
6. Refer to *Table 1*. Find the number from the Enter column that corresponds to Line Number 1 on the display. Push these numbers on the keypad, then push **ENTER**. Continue to add the numbers that correspond to each line number on the display.

**Note:** Use the arrow keys to scroll and review or change numbers at any time.

Table 1 Instrument setup

Line Number	Enter	Line Number	Enter
1	127	29	82
2	24	30	70
3	73	31	101
4	0	32	32
5	0	33	72
6	0	34	82
7	0	35	65
8	0	36	32
9	0	37	0
10	0	38	0
11	0	39	66
12	64	40	200
13	24	41	0
14	81	42	0
15	235	43	0
16	0	44	33
17	0	45	128
18	0	46	0
19	0	47	15
20	70	48	0
21	101	49	180
22	32	50	0
23	76	51	0
24	82	52	0
25	70	53	0
26	101	54	132
27	32	55	0
28	77	56	255

# Iron, Total

## REQUIRED REAGENTS & APPARATUS (Using Powder Pillows)

Description	Quantity Per Test	Unit	Item No.
FerroVer Iron Reagent Powder Pillows	1 pillow	100/pkg	2105769
Sample cell, 10-20-25 mL, with screw cap	1	6/pkg	2401906
EDTA Solution 1M	2 drops	50 mL	2241926

## OPTIONAL REAGENTS

Description	Unit	Item No.
Hydrochloric Acid Standard Solution, 6 N	500 mL	88449
Iron Standard Solution, 100 mg/L	100 mL	1417542
Iron Ampule Standard, 25 mg/L	16/pkg	1425310
Sodium Hydroxide Standard Solution, 5.0 N	100 mL MDB	245032
Water, deionized	4 L	27256

## OPTIONAL APPARATUS

Description	Unit	Item No.
Ampule Breaker, Voluette Ampules	1	2196800
Cylinder, graduated, poly, 25 mL	1	108140
Cylinder, graduated, poly, 100 mL	1	108142
Flask, Erlenmeyer, 250 mL	1	50546
Flask, volumetric, Class A, 100 mL	1	1457442
Pipet Filler, safety bulb	1	1465100
Pipet, serological, 2 mL	1	53236
Pipet, TenSette, 0.1 to 1.0 mL	1	1970001
Pipet Tips, for 1970001 TenSette Pipet	50/pkg	2185696
Pipet Tips, for 1970001 TenSette Pipet	1000/pkg	2185628
Pipet, volumetric, Class A, 1.00 mL	1	1451535
Pipet, TenSette 1.0 - 10.0 ml	1	1970010
Pipet Tips, for 1970010 TenSette Pipet	50/pk	2199796
Pipet Tips, for 1970010 TenSette Pipet	250/pk	2199725



FOR TECHNICAL ASSISTANCE, PRICE INFORMATION AND ORDERING:  
In the U.S.A. – Call toll-free 800-227-4224  
Outside the U.S.A. – Contact the HACH office or distributor serving you.  
On the Worldwide Web – [www.hach.com](http://www.hach.com); E-mail – [techhelp@hach.com](mailto:techhelp@hach.com)

HACH COMPANY  
WORLD HEADQUARTERS  
Telephone: (970) 669-3050  
FAX: (970) 669-2932