

#### SUMMARY OF METHOD

Iron(II) ions form an orange-red complex with 1.10-phenanthroline. Any iron(III) ions present in the water sample are reduced to iron(II) ions by ascorbic acid before the complex is formed. This method complies with regulations in many countries and therefore can often be used for reporting purposes. Contact your local **Hach** distributor to determine if this method can be used for regulatory reporting purposes in your country.

#### SHELF LIFE OF TEST REAGENT

The test reagents are stable at room temperature for 30 months as indicated by the expiration date on the package.

#### SAMPLE COLLECTION, PRESERVATION AND STORAGE

Collect samples in acid-cleaned glass or plastic containers. No acid addition is necessary if analyzing the samples immediately. To preserve samples, adjust the pH to 2 or less with concentrated nitric acid (about 2.00 ml per liter). Preserved samples may be stored up to six months at room temperature. If reporting only dissolved free iron, filter sample immediately after collection and before adding nitric acid.

Before analysis, adjust the pH to between 3 and 5 with 5.0 N sodium hydroxide standard solution. Do not exceed pH 5 as iron may precipitate. Correct the test results for volume additions.

#### INTERFERENCES

The ions listed in the table have been individually checked up to the given concentrations. Cumulative effects and the influence of other ions have not been evaluated. There is no interference from:

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1000 mg/L: Cl<sup>-</sup>

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500 mg/L: Ca<sup>2+</sup>

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100 mg/L: Ag<sup>+</sup>

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70 mg/L: Cd<sup>2+</sup>

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50 mg/L: Co<sup>2+</sup>, Zn<sup>2+</sup>, Pb<sup>2+</sup>, CO<sub>3</sub><sup>2-</sup>, Hg<sup>2+</sup>, Cr<sup>3+</sup>, Cr<sup>6+</sup>

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25 mg/L: Ni<sup>2+</sup>

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10 mg/L: Cu<sup>2+</sup>

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5 mg/L: Sn<sup>2+</sup>

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Higher amounts of copper, nickel, and tin cause high-bias results.

**Total iron including undissolved iron and complexed iron can only be determined after digesting with the Metal Prep Set HCT 200. (Total iron measuring range 0.12 – 6.00 mg/L).**

#### ACCURACY CHECK

##### Standard Solution Method

Prepare a 3.00 mg/L Fe standard solution by pipetting 0.30 ml of 1000 mg/L Fe into a 100 ml volumetric flask. Dilute to the mark with deionized water. Stopper and invert to mix. Prepare this solution daily. Perform the iron procedure as described.

##### Standard Addition Method

- Measure 100 ml of sample into three mixing cylinders.
- Using a pipette add 0.10, 0.20 and 0.30 ml of 1000 mg/L Fe standard solution to the three samples. Mix well.
- Analyze each sample using the procedure.  
 The Fe concentration should increase 1.00 mg/L for each 0.10 ml of standard added.

#### METHOD PERFORMANCE

##### Precision

In a single laboratory, using a standard solution of 3.00 mg/L Fe and two representative lots of reagent, a single operator obtained a standard deviation of +/- 0.15 mg/L on a total of 14 samples.

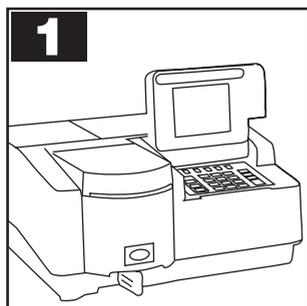
##### TIPS AND TECHNIQUES

- Adjust pH of preserved samples to between pH 3 – 5 before analysis. Water samples with pH < 2 affects the reagent.
- The outside of the vial must be clean before placing it in the instrument. Wipe it down with a damp towel, followed by a dry one.
- For a proof of accuracy, use the standard solution or standard addition method. (See Accuracy Check).
- See your Instrument's Procedure Manual for instructions on adjusting the calibration curve if needed.
- The temperature of the water sample and the sample vial should be between 15 – 25°C (59 – 77°F).

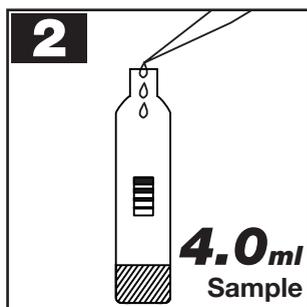
##### SAFETY

Good safety habits and laboratory techniques should be used throughout the procedure. Consult the Material Safety Data Sheet for information specific to the reagents used.

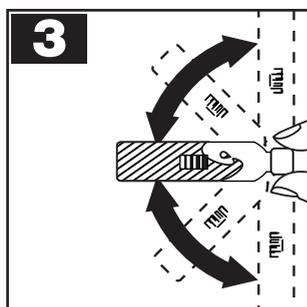
**PROCEDURE for Free Iron**  
applies to DR/4000, DR/2500 DR/2010 and DR/800



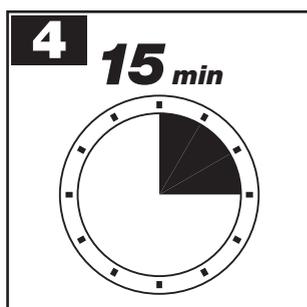
1. Enter program number and set up the instrument as explained for each instrument type.



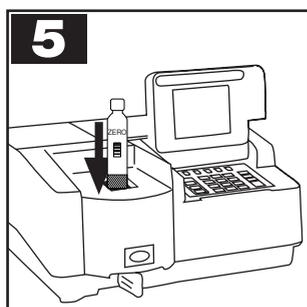
2. Pipet **4.0 ml** sample into the sample vial.



3. Close sample vial and invert a few times, until the freeze-dried contents are completely dissolved.



4. Start the instrument timer.  
A **15-minute** reaction period will begin.



5. Zero the instrument using the zero vial (white cap) provided and read the sample.

**PROCEDURE for Total Iron:**

After digestion with the Metal Prep Set HCT 200, follow the procedure steps 1 – 5.

**MEASURING RANGE:**  
0.10 - 5.00 mg/L Free-Fe / 0.12 - 6.00 mg/L Tot-Fe

**Note**

If this is the first time you have run this revision of the test on your DR/4000, you must contact your local **Hach** distributor for an upgrade of your software (Cat.No. 49544-00). Software Version number 2.39 contains the factory **UniCell™** programs.

**INSTRUMENT SET-UP**

1. Press the soft key under **HACH PROGRAM** (or **USER PROGRAM** if the method was entered as a **USER PROGRAM**).  
Enter program number **2130** (or **159** if the method was entered as a **USER PROGRAM**).  
Press **ENTER**.
2. The display will show:  
**HACH PROGRAM: 2130**  
**Iron, HCT 159**  
The wavelength ( $\lambda$ ), **485 nm**, is automatically selected.
3. Insert the *Test N'Tube* adapter into the cell holder by sliding it under the thumb screw and into the alignment grooves. Fasten in place by tightening the thumb screw.
4. Follow the procedure steps.  
(**Note:** If measuring Total Iron and performed the sample digestion using the HCT 200 Metal Prep Set, select the Total Iron alternate form under the Options soft key menu).
5. Wipe the outside of the zero vial.  
Place the zero vial into the adapter.  
Close the light shield.
6. Press the soft key under **ZERO**.  
The display will show: **0.00 mg/L Free-Fe** or **0.00 mg/L Tot-Fe** if the Total Iron alternate form was selected.
7. Clean the outside of the sample vial.
8. Place the prepared sample vial into the adapter. Close the light shield. Results in **mg/L Free-Fe** or **Tot-Fe** are displayed.

**Note**

If the test overranges, repeat the **UniCell™** test with diluted sample.

**LIST OF REQUIRED REAGENTS**

Iron-Fe		
<b>UniCell™</b> HCT 159	23/pkg	HCT 159

**OPTIONAL REAGENTS**

Metal Prep Set		
<b>UniCell™</b> HCT 200	50 digestions	HCT 200
Iron Standard		
1000 mg/L as Fe	100 ml	2271-42
Sodium Hydroxide		
Standard Solution	1000 ml	2450-53
(5.0 N)		
Nitric Acid, concentrated	500 ml	152-49

**OPTIONAL APPARATUS**

Cylinder, mixing – 100 ml	each	20886-42
Flask, volumetric 100 ml	each	547-42
Pipettor, (Jencons) 1-5 ml	each	27951-00
Replacement tips for 27951-00	pk/100	27952-00
Pipettor, (Jencons) 100-1000 $\mu$ l	each	27949-00
Replacement tips for 27949-00	pk/400	27950-00
pH Paper	pk/100	26013-00

**MEASURING RANGE:**

0.10 - 5.00 mg/L Free-Fe / 0.12 - 6.00 mg/L Tot-Fe

**Note**

If this is the first time you have run this revision of the test on your DR/2500, you must contact your local **Hach** distributor for an upgrade of your software (Instrument Version Number 1.11 and Hach Program version number 27).

**INSTRUMENT SET-UP**

1. Touch: **Hach Programs**.  
Select program **276, Iron HCT 159**.  
Then touch: **Start**.
2. Follow the procedure steps.  
*(Note: If measuring Total Iron and performed the sample digestion using the HCT 200 Metal Prep Set, select the Total Iron alternate form under Options).*
3. Wipe the outside of the zero vial.  
Place it into the cell holder.
4. Touch: **Zero**. The display will show:  
**0.00 mg/L Free-Fe or Tot-Fe** if the Total Iron alternate form was selected.
5. Clean the outside of the prepared sample vial and place it into the cell holder. Read the results.

**MEASURING RANGE:**

0.10 - 5.00 mg/L Free-Fe / 0.12 - 6.00 mg/L Total-Fe

**Note**

If this is the first time you have run this test on your DR/890, you must first perform the DR/890 User Calibration procedure at the end of the next section (see Instrument Methods).

**INSTRUMENT SET-UP**

1. Press **PRGM**. The display will show **PRGM ?**  
Enter the user program number that the **UniCell™ Iron HCT 159** was entered in and press **ENTER**. The display will show **USER** and **mg/L** as the units. These will be in the Concentration Display Form unit chosen when the method was entered in the instrument.
2. Insert the COD/TNT Adapter into the cell holder by rotating the adapter until it drops into place.  
Then, push down to make sure it is completely inserted.
3. Follow the procedure steps.  
*(Note: Ensure that the correct Iron unit Free or Total was used when the method was entered in the instrument).*
4. Clean the outside of the Zero vial and place it in the adapter. Push down on the top of the vial until it seats completely into the adapter. Tightly cover the vial with the instrument cap and press **ZERO**.
5. Clean the outside of the sample vial and place it in the vial adapter. Push down on the top of the vial until it seats completely into the adapter. Tightly cover the vial with the instrument cap and press **READ**.
6. Results will be in **mg/L** of the Display Form unit chosen when the method was entered.

**MEASURING RANGE:**

0.10 - 5.00 mg/L Free Fe / 0.12 - 6.00 mg/L Total Fe

**Note**

If this is the first time you have run this test on your DR/2010, you must contact your local **Hach** distributor for an upgrade of your software or perform the Manual Program Update for the DR/2010 (see Instrument Methods).

**INSTRUMENT SET-UP**

1. The display should read: **Enter Program #**
2. Enter **276** on the keypad.
3. Rotate the wavelength dial until the small display shows: **485 nm**.  
When the correct wavelength is dialed in, the display will quickly show: **Zero Sample**.
4. Insert the COD Vial Adapter into the cell holder with the marker to the right.
5. Follow the procedure steps.  
*(Note: If measuring Total Iron and performed the sample digestion using the HCT 200 Metal Prep Set, select the Total Iron alternate form).*
6. Wipe the outside of the zero vial.  
Place the vial into the adapter.  
Place the cover on the adapter.
7. Press: **ZERO**. The display will show:  
**Zeroing...** then: **0.00 mg/L Free Fe** or **0.00 mg/L Total Fe** if the Total Iron alternate form was selected.
8. Clean the outside of the sample vial.
9. Place the vial into the adapter. Place the cover on the adapter.
10. Press: **READ**. The display will show: **Reading...**  
then the results are displayed in **mg/L Free Fe** or **Total Fe**.

**Note**

If the test overranges, repeat the **UniCell™** test with diluted sample.