Iron, Total

Method 8365

Scope and Application: For cooling water containing molybdate-based treatment; digestion is required for determining total iron. See SECTION 1 for digestion procedure. The estimated detection limit for program number 2160 is 0.025 mg/L.

* Adapted from G. Frederick Smith Chemical Co., The Iron Reagents, 3rd ed. (1980)

1. Press the soft key under HACH PROGRAM.

Select the stored program number for iron, FerroMo, by pressing 2160 with the numeric keys.

Press: ENTER

Note: Analyze samples as soon as possible to prevent air oxidation of ferrous iron to ferric iron, which is not determined. See Sample Collection, Storage and Preservation following these steps. Adjust the pH of preserved samples before analysis.

Note: The Flow Cell and Sipper Modules can be used with this procedure.

2. The display will show:

HACH PROGRAM: 2160 Iron, FerroMo

The wavelength (λ), 590 nm, is automatically selected.

Note: Rinse glassware with a 1:1 Hydrochloric Acid Solution. Rinse again with deionized water. These two steps will remove iron deposits which can cause slightly high results.

Note: For best results, determine a reagent blank for each new lot of reagent as follows. Prepare a reagent blank by repeating steps 3 through 11, using deionized water as the sample. Zero the instrument on deionized water by pressing the soft key under ZERO. Insert the reagent blank and the blank value will be displayed. Correct for the reagent blank by pressing the soft keys under OPTIONS, (MORE), and then BLANK:OFF. Enter the reagent blank value and press ENTER. Repeat for each new lot of reagent.

Note: Determination of total iron requires digestion; see SECTION 1 for procedures.

3. Fill a 50-mL graduated mixing cylinder with 50 mL of water to be tested.

Note: Sample pH is important in this test and should be between 3 and 5; see pH discussion in the Interferences section.

Note: For proof of accuracy, use a 0.4-mg/L iron standard solution (preparation given in the Accuracy Check section) in place of the sample.

4. Add the contents of one FerroMo Iron Reagent 1 Powder Pillow to the graduated mixing cylinder. Stopper and invert several times to dissolve the reagents. This is the prepared sample.
5. Fill a clean matched sample cell to the 25-mL mark with prepared sample. Save the remaining 25 mL of prepared sample for Step 8.

6. Add the contents of one FerroMo Iron Reagent 2 Powder Pillow to the sample cell. Swirl to dissolve the reagents. This is the developed sample. 
   Note: A blue color will develop if iron is present.

7. Press the soft key under START TIMER. A 3-minute reaction period will begin.

8. Fill a second sample cell with the remaining 25 mL of prepared sample from Step 5 (the blank).

9. When the timer beeps, insert the blank into the cell holder. Close the light shield.

10. Press the soft key under ZERO. The display will show:
    0.000 mg/L Fe
    Note: If you are using a reagent blank correction, the display will show the correction.
    Note: For alternate concentration units, press the soft key under OPTIONS. Then press the soft key under UNITS to scroll through the available options. Press ENTER to return to the read screen.

11. Place the developed sample into the cell holder. Close the light shield. The results in mg/L iron (or chosen units) will be displayed.
    Note: For samples containing high levels of molybdate (≥100 mg/L as MoO₄²⁻ or MoO₃), read the sample immediately after zeroing the blank.
Inteferences

<table>
<thead>
<tr>
<th>Interfering Substance</th>
<th>Interference Levels and Treatments</th>
</tr>
</thead>
<tbody>
<tr>
<td>pH</td>
<td>A sample pH of less than 3 or greater than 4 after the addition of reagent may inhibit color formation, cause the developed color to fade quickly or result in turbidity. Adjust the sample pH in the graduated cylinder before the addition of reagent to between 3 and 5 by using a pH meter or pH paper and adding, dropwise, an appropriate amount of iron-free acid or base such as 1.0 N Sulfuric Acid Standard Solution or 1.0 N Sodium Hydroxide Standards Solution. Make a volume correction if significant volumes of acid or base are used; see Section 1.2.2 Correcting for Volume Additions.</td>
</tr>
</tbody>
</table>

Sample Collection, Storage and Preservation

Collect samples in acid-washed glass or plastic bottles. To preserve samples, adjust the sample pH to 2 or less with hydrochloric acid (about 2 mL per liter). Samples preserved in this manner can be stored up to six months at room temperature. If only dissolved iron is to be reported, filter sample immediately after collection through a 0.45 micron filter or equivalent medium before addition of hydrochloric acid.

Before testing, adjust the sample pH to 3–5 with 5.0 N Sodium Hydroxide Standard Solution. Do not exceed pH 5 as iron may precipitate. Correct test results for volume additions; see Section 1.2.2 Correcting for Volume Additions.

Accuracy Check

Standard Additions Method

a. Leave the unspiked sample in the sample cell compartment. Verify that the units displayed are in mg/L. Select standard additions mode by pressing the soft keys under OPTIONS, (MORE) and then STD ADD.

b. Press ENTER to accept the default sample volume (mL), 50.

c. Press ENTER to accept the default standard concentration (mg/L), 100.

d. Press the soft key under ENTRY DONE.

e. Open an Iron Voluette Ampule Standard, 100-mg/L Fe.

f. Use the TenSette Pipet to add 0.1 mL, 0.2 mL and 0.3 mL of standard, respectively to three 50-mL samples and mix each thoroughly.

g. Analyze each standard addition sample as described above. Accept the standard additions reading by pressing the soft key under READ each time. Each addition should reflect approximately 100% recovery.

h. After completing the sequence, the display will show the extrapolated concentration value and the “best-fit” line through the standard additions data points, accounting for matrix interferences.

i. See Section 1.4.1 Standard Additions for more information.

Standard Solution Method

Using Class A glassware, prepare a 0.400-mg/L iron standard solution by pipetting 4.00 mL of Iron Standard Solution, 100-mg/L, into a 1-liter volumetric flask. Dilute to the mark with deionized water. Prepare this solution daily. Perform the iron procedure as described above.
To adjust the calibration curve using the reading obtained with the 0.400-mg/L Fe Standard Solution, press the soft keys under OPTIONS, (MORE) then STD: OFF. Press ENTER to accept the default concentration, the value of which will depend on the selected units. If an alternate concentration is used, enter the actual concentration and press ENTER to return to the read screen. See Section 1.5.5 Adjusting the Standard Curve for more information.

### Method Performance

#### Precision

Standard: 0.40 mg/L Fe

<table>
<thead>
<tr>
<th>Program</th>
<th>95% Confidence Limits</th>
</tr>
</thead>
<tbody>
<tr>
<td>2160</td>
<td>0.387–0.413 mg/L Fe</td>
</tr>
</tbody>
</table>

For more information on determining precision data and method detection limits, refer to Section 1.5.

### Estimated Detection Limit

<table>
<thead>
<tr>
<th>Program</th>
<th>EDL</th>
</tr>
</thead>
<tbody>
<tr>
<td>2160</td>
<td>0.025 mg/L Fe</td>
</tr>
</tbody>
</table>

For more information on derivation and use of Hach’s estimated detection limit, see Section 1.5.2. To determine a method detection limit (MDL) as defined by the 40 CFR part 136, appendix B, see Section 1.5.1.

#### Sensitivity

Program Number: 2160

<table>
<thead>
<tr>
<th>Portion of Curve</th>
<th>∆Abs</th>
<th>∆Concentration</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.010 Abs</td>
<td>0.010</td>
<td>0.0112 mg/L</td>
</tr>
<tr>
<td>0.200 mg/L</td>
<td>0.010</td>
<td>0.0113 mg/L</td>
</tr>
<tr>
<td>1.620 mg/L</td>
<td>0.010</td>
<td>0.0121 mg/L</td>
</tr>
</tbody>
</table>

See Section 1.5.3 Sensitivity Explained for more information.

### Calibration Standard Preparation

To perform an iron calibration using the FerroMo Method, use a 10-mg/L Iron Standard Solution (Cat. No. 140-49). Prepare calibration standards containing 0.30, 0.60, 0.90, 1.20, 1.50, and 1.80 mg/L Fe as follows:

- a. Into six different 100-mL volumetric flasks, pipet 3.00, 6.00, 9.00, 12.00, 15.00, and 18.00 mL of the 10 mg/L Iron Standard Solution using Class A glassware.
- b. Dilute to the mark with deionized water. Mix thoroughly.
- c. Using the FerroMo method and the calibration procedure described in the User-Entered Programs section of the DR/4000 Spectrophotometer Instrument Manual, generate a calibration curve from the standards prepared above.
Summary of Method

FerroMo Iron Reagent 1 contains a reducing agent combined with a masking agent. The masking agent eliminates interference from high levels of molybdate. The reducing agent converts precipitated or suspended iron, such as rust, to the ferrous state. FerroMo Iron Reagent 2 contains the indicator combined with a buffering agent. The indicator reacts with ferrous iron in the sample, buffered between pH 3 and 5, resulting in a deep blue-purple color.

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. For additional information, refer to Section 1.

Pollution Prevention and Waste Management

For information on pollution prevention and waste management, refer to Section 1.

REQUIRED REAGENTS AND STANDARDS

FerroMo Iron Reagent Set (100 tests) ........................................................................................... ..........25448-00
Includes: (4) 25437-68, (2) 25438-66

<table>
<thead>
<tr>
<th>Description</th>
<th>Quantity Required</th>
<th>Unit</th>
<th>Cat. No.</th>
</tr>
</thead>
<tbody>
<tr>
<td>FerroMo Iron Reagent 1 Powder Pillows</td>
<td>1 pillow</td>
<td>25/pkg</td>
<td>25437-68</td>
</tr>
<tr>
<td>FerroMo Iron Reagent 2 Powder Pillows</td>
<td>1 pillow</td>
<td>50/pkg</td>
<td>25438-66</td>
</tr>
</tbody>
</table>

REQUIRED EQUIPMENT AND SUPPLIES

Clippers, for opening powder pillows ............. each 968-00
Cylinder, graduated mixing, 50-mL, w/ stopper ...... each 1896-41
DR/4000 1-Inch Cell Adapter ........................ each 48190-00

OPTIONAL REAGENTS AND STANDARDS

<table>
<thead>
<tr>
<th>Description</th>
<th>Unit</th>
<th>Cat. No.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hydrochloric Acid, ACS</td>
<td>500 mL</td>
<td>134-49</td>
</tr>
<tr>
<td>Iron Standard Solution, 100 mg/L</td>
<td>100 mL</td>
<td>14175-42</td>
</tr>
<tr>
<td>Iron Standard Solution, 10-mL Voluette ampule, 25 mg/L Fe</td>
<td>16/pkg</td>
<td>14253-10</td>
</tr>
<tr>
<td>Iron Standard Solution, 10 mg/L Fe</td>
<td>500 mL</td>
<td>140-49</td>
</tr>
<tr>
<td>Sodium Hydroxide Standard Solution, 1.00 N</td>
<td>100 mL MDB</td>
<td>1045-32</td>
</tr>
<tr>
<td>Sodium Hydroxide Standard Solution, 5.0 N</td>
<td>100 test MDB</td>
<td>2450-32</td>
</tr>
<tr>
<td>Sulfuric Acid Standard Solution, 1.00 N</td>
<td>100 mL MDB</td>
<td>1270-32</td>
</tr>
<tr>
<td>Water, deionized</td>
<td>4 liters</td>
<td>272-56</td>
</tr>
</tbody>
</table>

OPTIONAL EQUIPMENT AND SUPPLIES

Ampule Breaker Kit ..................................... each 21968-00
Aspirator, Nalgene vacuum pump ...................... each 2131-00
Digesdahl Apparatus, 115 VAC, 50/60 Hz .......... each 23130-20
Digesdahl Apparatus, 230 VAC, 50/60 Hz .......... each 23130-21
DR/4000 Carousel Module Kit ...................... each 48070-02
DR/4000 Flow Cell Module Kit, 1-inch ............ each 48070-04
DR/4000 Flow Cell Module Kit, 1-cm ............... each 48070-05
DR/4000 Sipper Module Kit, 1-inch ............... each 48090-03
Filter Disks, glass fiber, 47-mm .................. 100/pkg 2530-00
Filter Holder, membrane ................................ each 2340-00
Flask, filtering, 500-mL ............................. each 546-49
IRON, Total, continued

Flask, volumetric, Class A, 100-mL ................................................................. each .................. 14574-42
Flask, volumetric, Class A, 1000-mL, with stopper............................................. each .................. 14574-53
pH Paper, pH 1.0 to 11.0 ........................................................................................................ 5 rolls/pkg ................. 391-33
pH Meter, sension™, portable .............................................................................................. each .................. 51700-00
Pipet Filler, safety bulb ......................................................................................................... each .................. 14651-00
Pipet, serological, 2-mL ....................................................................................................... each .................. 532-36
Pipet TenSette, 0.1 to 1.0 mL .............................................................................................. each .................. 19700-01
Pipet Tips, for 19700-01 TenSette Pipet ............................................................................. 50/pkg ................. 21856-96
Pipet, volumetric, Class A, 1.00-mL .................................................................................. each .................. 14515-35
Pipet, volumetric, Class A, 2.00-mL .................................................................................. each .................. 14515-36
Pipet, volumetric, Class A, 3.00-mL .................................................................................. each .................. 14515-03
Pipet, volumetric, Class A, 4.00-mL .................................................................................. each .................. 14515-04
Pipet, volumetric, Class A, 5.00-mL .................................................................................. each .................. 14515-37
Pipet, volumetric, Class A, 6.00-mL .................................................................................. each .................. 14515-06
Pipet, volumetric, Class A, 8.00-mL .................................................................................. each .................. 14515-08
Pipet, volumetric, Class A, 9.00-mL .................................................................................. each .................. 14515-09
Pipet, volumetric, Class A, 10.00-mL ............................................................................... each .................. 14515-38