

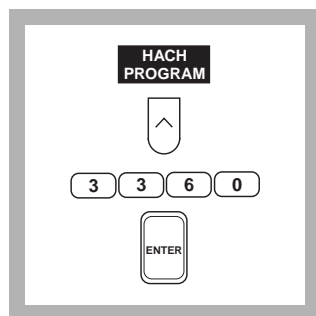


## Method 8186

## Heteropoly Blue Method\* LR (0 to 1.600 mg/L as SiO<sub>2</sub>)

**Scope and Application:** For water and seawater  
The estimated detection limit for program number 3360 is 0.01 mg/L SiO<sub>2</sub>.

\* Adapted from *Standard Methods for the Examination of Water and Wastewater*.



**1.** Press the soft key under **HACH PROGRAM**.

Select the stored program number for low range silica by pressing **3360** with the numeric keys.

Press: **ENTER**

**Note:** If samples cannot be analyzed immediately, see *Sample Collection, Storage and Preservation* following these steps.

**Note:** The Flow Cell and Sipper Modules are recommended for this procedure. Use a 25-mL sample and reagents with the Flow Cell Module.



**2.** The display will show: **HACH PROGRAM: 3360 Silica, LR**

The wavelength ( $\lambda$ ), **815 nm**, is automatically selected.

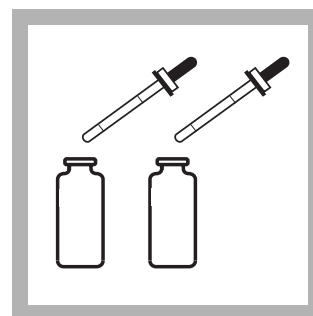
**Note:** For best results, determine a reagent blank for each new lot of reagent as follows. Prepare a reagent blank by repeating steps 3 through 12, using low silica deionized water as the sample. Zero the instrument on deionized water by pressing the soft key under **ZERO**. Insert the reagent blank 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.



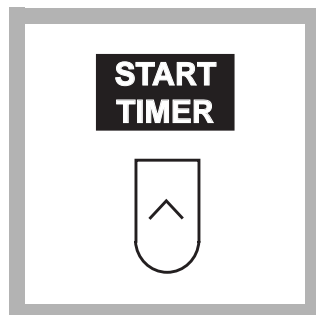
**3.** Fill two sample cells with 10 mL of sample.

**Note:** For proof of accuracy, use a 0.50-mg/L Silica Standard Solution in place of the sample (see *OPTIONAL REAGENTS AND STANDARDS*).

**Note:** For turbid samples, see the *Interferences* section following these steps.



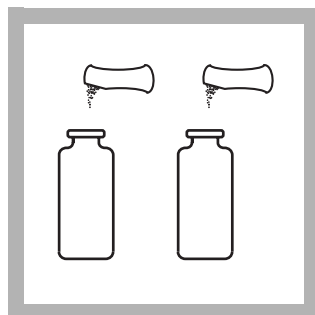
**4.** Add the 0.5 mL of Molybdate 3 Reagent to each sample cell. Swirl to mix.



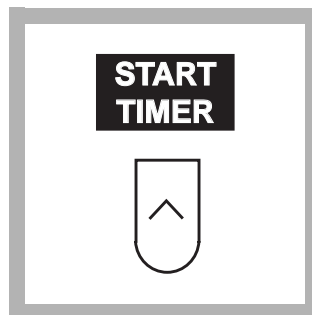
**5.** Press the soft key under **START TIMER**.

A 4-minute reaction period will begin.

**Note:** Reaction time depends on sample temperature. The time given is for samples at 20 °C (68 °F). If the sample temperature is 10 °C (50 °F), wait 8 minutes. If the sample temperature is 30 °C (86 °F), wait 2 minutes.



**6.** When the timer beeps, add the contents of one Citric Acid Reagent Powder Pillow to each sample cell. Swirl to mix.



**7.** Press the soft key under **START TIMER**.

A one-minute reaction period will begin. The destruction of possible phosphate interference occurs during this period.

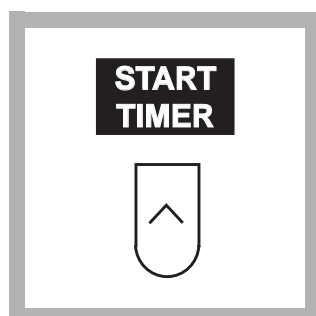
**Note:** Reaction time depends on sample temperature. The time given is for samples at 20 °C (68 °F). If the sample temperature is 10 °C (50 °F), wait 2 minutes. If the sample temperature is 30 °C (86 °F), wait 30 seconds.



**8.** When the timer beeps, add the contents of one Amino Acid F Reagent Powder Pillow to one of the sample cells. Swirl to mix. This is the prepared sample.

**Note:** The sample cell without the Amino Acid F Reagent is the blank.

**Note:** If testing for very low levels of silica, use the ultra low range silica method.

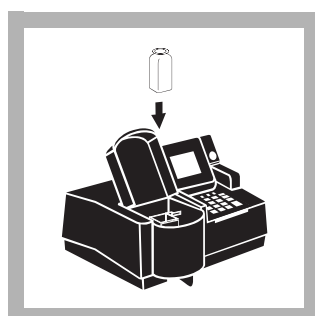


**9.** Press the soft key under **START TIMER**.

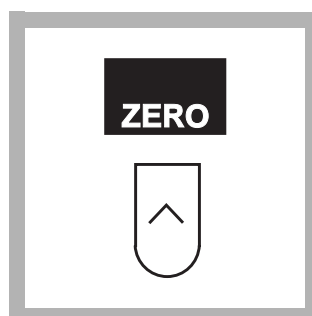
A 2-minute reaction period will begin.

**Note:** A blue color will develop if silica is present.

**Note:** Wiping the cells with a damp cloth, followed by a dry one removes fingerprints and other marks that may affect measurements.



**10.** When the timer beeps, place the blank in the cell holder. Close the light shield.



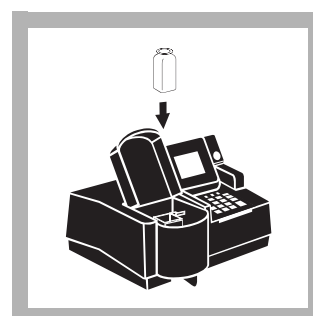
**11.** Press the soft key **ZERO**.

The display will show:

**0.00 mg/L SiO<sub>2</sub>**

**Note:** If you are using a reagent blank correction, the display will show the correction.

**Note:** For alternate concentration units press the soft keys under **OPTIONS**, **(MORE)**, then **UNITS** to scroll through the available options. Press **ENTER** to return to the read screen.



**12.** Place the prepared sample in the cell holder. Close the light shield. Results in mg/L SiO<sub>2</sub> (or chosen units) will be displayed.

**Note:** The results can be expressed as silicon (Si). Press the soft keys under **OPTIONS**, **(MORE)**, then **FORM**: to scroll through the options. Press **ENTER** to return to the read screen.

## Interferences

**Table 1 Interfering Substances and Suggested Treatments**

Interfering Substance	Interference Levels and Treatments
Color	Eliminated by zeroing the instrument with the original sample
Iron	Large amounts interfere
Phosphate	Does not interfere at levels less than 50 mg/L PO <sub>4</sub> . At 60 mg/L PO <sub>4</sub> , an interference of -2% occurs. At 75 mg/L PO <sub>4</sub> the interference is -11%.
Slow reacting forms of silica	Occasionally a sample contains silica which reacts very slowly with molybdate. The nature of these "molybdate-unreactive" forms is not known. A pretreatment with sodium bicarbonate, then sulfuric acid will make these forms reactive to molybdate. The pretreatment is given in <i>Standard Methods for the Examination of Water and Wastewater</i> under Silica-Digestion with Sodium Bicarbonate. A longer reaction time with the sample and the molybdate and acid reagents (before adding citric acid) may help in lieu of the bicarbonate pretreatment.
Sulfides	Interfere at all levels
Turbidity	Eliminated by zeroing the instrument with the original sample

## Sample Collection, Storage and Preservation

Collect samples in clean plastic bottles. Analyze samples as soon as possible after collection. If prompt analysis is not possible, store samples for up to 28 days by cooling to 4 °C (39 °F) or below. Warm samples to room temperature before analysis.

## Accuracy Check

### Standard Additions Method

- a. Leave the unspiked sample in the sample 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), 10.0.
- c. Press **25.00** and then press **ENTER** to accept the standard concentration (mg/L), 25.
- d. Press the soft key under **ENTRY DONE**.
- e. Open a 25-mg/L Silica Standard Solution bottle.
- f. Use the TenSette Pipet to add 0.2 mL, 0.4 mL and 0.6 mL of standard, respectively to three 10-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

Use the 1.00-mg/L SiO<sub>2</sub> Standard Solution listed under Optional Reagents and Standards in place of the sample. Perform the silica procedure as described above.

To adjust the calibration curve using the reading obtained with the 1.00 mg/L Standard Solution, press the soft keys under **OPTIONS, (MORE)** then **STD:OFF**. Press **ENTER** to accept the displayed concentration, the value of which depends 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: 1.00 mg/L SiO<sub>2</sub>

Program	95% Confidence Limits
3360	0.950–1.050 mg/L SiO <sub>2</sub>

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

### Estimated Detection Limit

Program	EDL
3360	0.01 mg/L SiO <sub>2</sub>

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: 3360

Portion of Curve	ΔAbs	ΔConcentration
0.010 Abs	0.010	0.012 mg/L
0.80 mg/L	0.010	0.011 mg/L
1.44 mg/L	0.010	0.011 mg/L

See Section 1.5.3 *Sensitivity Explained* for more information.

## Calibration Standard Preparation

Preparing silica standards is difficult. Standards are easily contaminated and should be made by a trained chemist.

To perform a silica calibration using the Heteropoly Blue method, prepare calibration standards containing 0.20, 0.50, 0.80, 1.20 and 1.60 mg/L silica as follows:

- a. Into five different 100-mL volumetric flasks, pipet 2.00, 5.00, 8.00, 12.00 and 16.00 mL of a 10.00-mg/L Silica Standard Solution using Class A glassware.

- b. Dilute each flask to volume with ultra-low silica deionized water. Stopper and invert several times to mix.
- c. Using the Heteropoly Blue 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

Silica and phosphate in the sample react with molybdate ion under acidic conditions to form yellow silicomolybdic acid complexes and phosphomolybdic acid complexes. Addition of citric acid destroys the phosphate complexes. An Amino Acid is then added to reduce the yellow silicomolybdic acid to an intense blue color, which is proportional to the silica concentration.

### 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

Low Range Silica Reagent Set (100 tests) .....			<b>Cat. No.</b>
			24593-00
Includes: (1) 22540-69, (1) 21062-69 (2) 1995-26, (1) 1117-02			
	<b>Quantity Required</b>		
<b>Description</b>	<b>per test</b>	<b>Unit</b>	<b>Cat. No.</b>
Amino Acid F Reagent Powder Pillows (for 10-mL sample) .....	1 pillow .....	100/pkg .....	22540-69
Citric Acid Powder Pillows .....	2 pillows .....	100/pkg .....	21062-69
Molybdate 3 Reagent Solution .....	1.0 mL .....	50 mL .....	1995-26

### REQUIRED EQUIPMENT AND SUPPLIES

DR/4000 1-Inch Cell Adapter .....	1 .....	each .....	48190-00
-----------------------------------	---------	------------	----------

### OPTIONAL REAGENTS AND STANDARDS

Amino Acid F Reagent Powder .....	410 g .....	22833-55
Silica Standard Solution, 0.5-mg/L SiO <sub>2</sub> .....	3.78 liter .....	21008-17
Silica Standard Solution, 1-mg/L SiO <sub>2</sub> .....	500 mL .....	1106-49
Silica Standard Solution, 10-mg/L SiO <sub>2</sub> .....	500 mL .....	1403-49
Silica Standard Solution, 50-mg/L SiO <sub>2</sub> .....	200 mL .....	1117-29
Silica Standard Solution, 25-mg/L as SiO <sub>2</sub> .....	236 mL .....	21225-31
Sodium Bicarbonate .....	454 g .....	776-01
Sodium Hydroxide Standard Solution, 1.00 N .....	900 mL .....	1045-53
Sulfuric Acid Standard Solution, 1.0 N .....	1000 mL .....	1270-53

## OPTIONAL EQUIPMENT AND SUPPLIES

Description	Unit	Cat. No.
Bottle, 118 mL, polyethylene, oblong.....	6/pkg.....	23184-06
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
Dropper, 0.5- & 1.0-mL marks, glass.....	6/pkg.....	23185-06
Flask, volumetric, Class A, 100-mL .....	each.....	14574-42
Flask, volumetric, Class A, 100-mL .....	6/pkg.....	14574-72
Flask, volumetric, Class A, 250-mL .....	each.....	14574-66
Pipet, Mohr, serological, 2-mL, poly .....	each.....	2106-36
Pipet, TenSette, 0.1 to 1.0 mL .....	each.....	19700-01
Pipet Tips, for 19700-01 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 Filler, safety bulb.....	each.....	14651-00
<i>Standard Methods for the Examination of Water and Wastewater</i> , 18th edition.....	each.....	22708-00
Thermometer, -10 to 110 °C.....	each.....	1877-01



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