



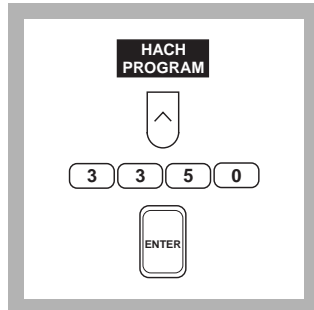
Method 8185

Silicomolybdate Method

HR (0 to 100.0 mg/L)

Scope and Application: For water and seawater.

The estimated detection limit for program number 3350 is 0.3 mg/L as SiO₂.



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

Select the stored program number for high range silica by pressing **3350** with the numeric keys.

Press: **ENTER**

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

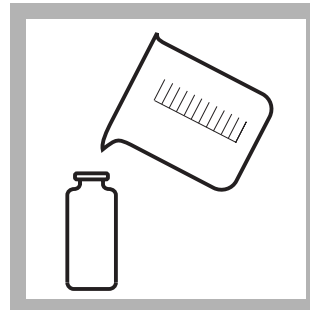
Note: The Flow Cell and Sipper Modules can be used with this procedure. Use a 25-mL sample and reagents with the Flow Cell Module.



2. The display will show: **HACH PROGRAM: 3350 Silica, HR**

The wavelength (λ), **452 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 ultra 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 a sample cell with 10 mL of sample.

Note: For proof of accuracy, use a 50-mg/L Silica Standard Solution in place of the sample (see *OPTIONAL REAGENTS AND STANDARDS*).

Note: Sample temperature should be 15–25 °C (59–77 °F).

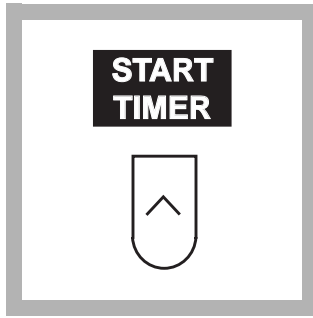


4. Add the contents of one Molybdate Reagent Powder Pillow for High Range Silica to the sample cell (the prepared sample). Swirl to mix.



5. Add the contents of one Acid Reagent Powder Pillow for High Range Silica. Swirl to mix.

Note: A yellow color will develop if silica or phosphorus is present.

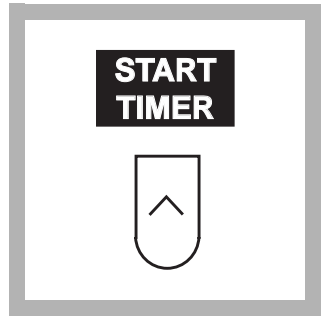


6. Press the soft key under **START TIMER**. A 10-minute reaction period will begin.



7. When the timer beeps, add the contents of one Citric Acid Powder Pillow to the sample cell. Swirl to mix.

Note: Any yellow color due to phosphorus is removed in this step.

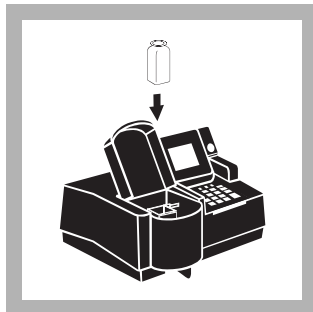


8. Press the soft key under **START TIMER**. A 2-minute reaction period will begin.

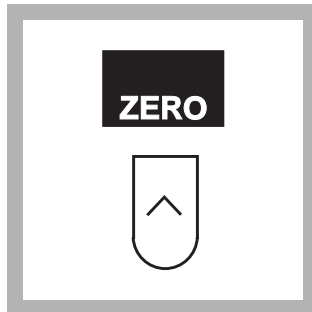
Note: Perform steps 9-12 within 3 minutes after the timer beeps.



9. When the timer beeps, fill a second sample cell with 10 mL of the original sample (the blank).



10. Place the blank in the cell holder. Close the light shield.



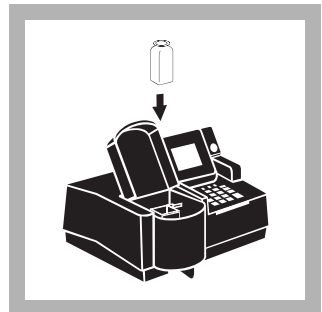
11. Press the soft key under **ZERO**.

The display will show:

0.0 mg/L SiO₂

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.



12. Place the prepared sample in the cell holder. Close the light shield. Results in mg/L silica (SiO₂) (or chosen units) will be displayed.

Note: The results can be expressed as silicon (Si). Press the soft keys under **OPTIONS**, 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	High levels of Fe ²⁺ and Fe ³⁺ interfere.
Phosphate	Does not interfere below 50 mg/L PO ₄ ³⁻ . At 60 mg/L PO ₄ ³⁻ , a negative 2% interference occurs. At 75 mg/L PO ₄ ³⁻ the interference is negative 11%.
Sulfides (S ²⁻)	High levels interfere.
Turbidity	Eliminated by zeroing the instrument with the original sample.

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 *Standard Methods for the Examination of Water and Wastewater* 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 treatment.

Sample Collection, Storage and Preservation

Collect samples in clean plastic bottles (glass is not recommended). Analyze sample as soon as possible after collection. If prompt analysis is not possible, store samples at 4 °C (39 °F) for up to 7 days. Warm samples to room temperature before analyzing.

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 **ENTER** to accept the default standard concentration (mg/L), 1000.
- d. Press the soft key under **ENTRY DONE**.
- e. Open a 1000-mg/L Silica Standard Solution.
- f. Use the TenSette Pipet to add 0.1 mL, 0.3 mL, and 0.5 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

To check test accuracy, use the 50-mg/L Silica Standard Solution listed under *OPTIONAL REAGENTS AND STANDARDS*. Analyze according to the HR Silica procedure described above using deionized water as the blank.

To adjust the calibration curve using the reading obtained with the 50.0-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: 50.0 mg/L SiO₂

Program	95% Confidence Limits
3350	49.7–50.3 mg/L SiO ₂

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

Estimated Detection Limit

Program	EDL
3350	0.3 mg/L SiO ₂

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 40 CFR part 136, appendix B, see Section 1.5.1.

Sensitivity

Program Number: 3350

Portion of Curve	ΔAbs	ΔConcentration
0.010 Abs	0.010	1.006 mg/L
50.0 mg/L	0.010	1.036 mg/L
90.0 mg/L	0.010	1.060 mg/L

See Section 1.5.3 *Sensitivity Explained* for more information.

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. Silica is then determined by measuring the remaining yellow color.

Calibration Standard Preparation

To perform a silica calibration using the Silicomolybdate method, prepare calibration standards containing 5.0, 10.0, 20.0, 50.0, 80.0, and 100.0 mg/L silica (SiO₂) as follows:

- a. Into each of six different 100-mL Class A volumetric flasks, pipet 0.50, 1.00, 2.00, 5.00, 8.00, and 10.00 mL of a 1000-mg/L Silica Standard Solution. Only use Class A pipets.
- b. Dilute each flask to volume with ultra-low silica deionized water. Stopper each flask and then invert several times to mix.
- c. Using the Silicomolybdate 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.

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

	Cat. No.
High Range Silica Reagent Set for 10-mL samples (100 tests)	24296-00
Includes: (1) 21074-69, (1) 21062-69, (1) 21073-69	

Description	Quantity Required		Cat. No.
	per test	Unit	
Acid Reagent Powder Pillows for High Range Silica	1	100/pkg	21074-69
Citric Acid Powder Pillows	1	100/pkg	21062-69
Molybdate Reagent Powder Pillows for High Range Silica	1	100/pkg	21073-69
Water, deionized	10 mL	4 liters	272-56

REQUIRED EQUIPMENT AND SUPPLIES

Clippers, for opening powder pillows	1	each	968-00
DR/4000 1-Inch Cell Adapter	1	each	48190-00

OPTIONAL REAGENTS AND STANDARDS

Silica Standard Solution, 10-mg/L	500 mL	1403-49
Silica Standard Solution, 50-mg/L	200 mL	1117-29
Silica Standard Solution, 1000-mg/L	500 mL	194-49
Sodium Bicarbonate	454 g	776-01
Sulfuric Acid Standard Solution, 1.000 N	100 mL MDB	1270-32

OPTIONAL EQUIPMENT AND SUPPLIES

Description	Unit	Cat. No.
Clippers, shears, 7¼-inch	each.....	23694-00
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
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, 0.50-mL.....	each.....	14515-34
Pipet, volumetric, Class A, 1.00-mL.....	each.....	14515-35
Pipet, volumetric, Class A, 2.00-mL.....	each.....	14515-36
Pipet, volumetric, Class A, 5.00-mL.....	each.....	14515-37
Pipet, volumetric, Class A, 8.00-mL.....	each.....	14515-08
Pipet, volumetric, Class A, 10.00-mL.....	each.....	14515-38
<i>Standard Methods for the Examination of Water and Wastewater</i> , 18th edition.....	each.....	22708-00
Thermometer, -10 to 110 °C.....	each.....	1877-01



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