

Silicomolybdate Method**Method 8185****HR (1 to 100 mg/L)****Powder Pillows**

Scope and Application: For water and seawater

**Test preparation**

How to use instrument-specific information

The *Instrument-specific information* table displays requirements that may vary between instruments. To use this table, select an instrument then read across to find the corresponding information required to perform this test.

Table 1 Instrument-specific information

Instrument	Sample cell	Cell orientation	Adapter
DR 5000	2495402	Fill line faces user	A23618
DR 2800	2495402	Fill line faces right	—
DR 2700	2495402	Fill line faces right	—
DR/2500	2427606	—	—
DR/2400	2427606	—	—

Before starting the test:

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

Collect the following items:

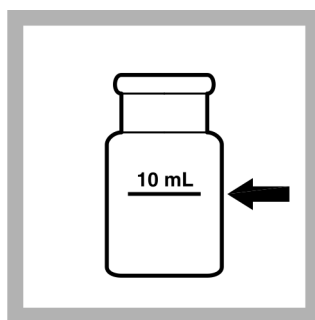
Description	Quantity
High Range Silica Reagent Set	1
Sample Cell (see <i>Instrument-specific information</i>)	2

See *Consumables and replacement items* for reorder information.

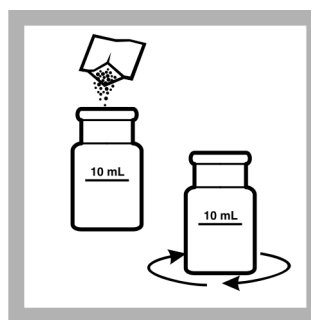
Silicomolybdate method for powder pillows



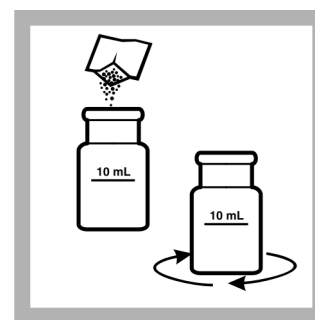
1. Select the test. Insert an adapter if required (see [Instrument-specific information](#)).



2. Fill a sample cell with 10-mL of sample.



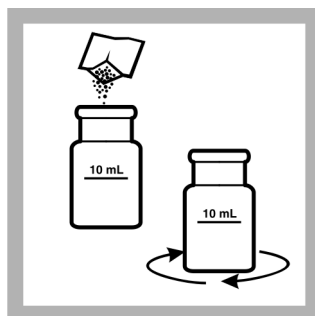
3. **Prepared Sample:** Add the contents of one Molybdate Reagent Powder Pillow for High Range Silica to the sample cell. Swirl until completely dissolved.



4. Add the contents of one Acid Reagent Powder Pillow for High Range Silica. Swirl to mix. A yellow color will develop if silica or phosphorus is present.



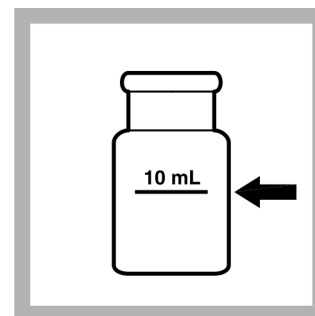
5. Start the instrument timer. A ten-minute reaction period will begin.



6. When the timer expires, add the contents of one Citric Acid Powder Pillow to the sample cell. Swirl to mix. Any yellow color due to phosphorus is removed in this step.



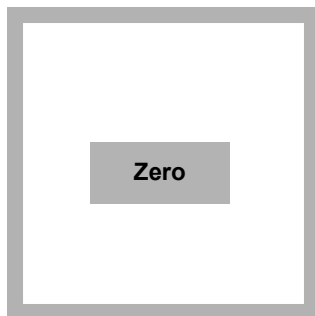
7. Start the instrument timer. A two-minute reaction period will begin. Perform steps 3–11 within three minutes after the timer expires.



8. **Blank Preparation:** Fill a second sample cell with 10 mL of the original sample.



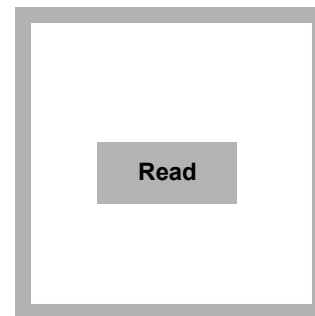
9. Wipe the blank and insert the blank into the cell holder.



10. **ZERO** the instrument. The display will show: 0 mg/L SiO₂



11. Wipe the prepared sample and insert the prepared sample into the cell holder.



12. **READ** the results in mg/L SiO₂.

Interferences

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 instead of the bicarbonate treatment.

Table 2 Interfering substances

Interfering substance	Interference level
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⁻)	All levels interfere.
Turbidity	Eliminated by zeroing the instrument with the original sample.

Sample collection, preservation and storage

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

Accuracy check

Standard additions method (sample spike)

Required for accuracy check:

- Silica Standard Solution, 1000 mg/L
 - TenSette Pipet and Pipet Tips
1. After reading test results, leave the sample cell (unspiked sample) in the instrument. Verify the chemical form.
 2. Select standard additions from the instrument menu:

Instrument	Navigate to:
DR 5000	OPTIONS>MORE>STANDARD ADDITIONS
DR 2800	OPTIONS>MORE>STANDARD ADDITIONS
DR 2700	OPTIONS>MORE>STANDARD ADDITIONS
DR/2500	OPTIONS>STANDARD ADDITIONS
DR/2400	OPTIONS>STANDARD ADDITIONS

3. Accept the default values for standard concentration, sample volume and spike volumes. After the values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.

* See [Optional reagents and apparatus](#).

4. Open the standard solution.
5. Use the TenSette Pipet to prepare spiked samples: add 0.1 mL, 0.2 mL and 0.3 mL of standard to three 10-mL portions of fresh sample.
6. Follow the [Silicomolybdate method for powder pillows](#) test procedure for each of the spiked samples, starting with the 0.1 mL sample spike. Measure each of the spiked samples in the instrument.
7. Select **GRAPH** to view the results. Select **IDEAL LINE** (or best-fit) to compare the standard addition results to the theoretical 100% recovery.

Standard solution method

Note: Refer to the instrument user manual for specific software navigation instructions.

Required for accuracy check:

- Silica Standard Solution, 50-mg/L
1. Use the Silica Standard Solution, 50-mg/L in place of the sample. Use deionized water as the blank. Follow the [Silicomolybdate method for powder pillows](#) test procedure.
 2. To adjust the calibration curve using the reading obtained with the standard solution, navigate to Standard Adjust in the software.

Instrument	Navigate to:
DR 5000	OPTIONS>MORE>STANDARD ADJUST
DR 2800	OPTIONS>MORE>STANDARD ADJUST
DR 2700	OPTIONS>MORE>STANDARD ADJUST
DR/2500	OPTIONS>STANDARD ADJUST
DR/2400	OPTIONS>STANDARD ADJUST

3. Turn on the Standard Adjust feature and accept the displayed concentration. If an alternate concentration is used, enter the concentration and adjust the curve to that value.

Method performance

Program	Instrument	Standard	Precision—95% Confidence Limits of Distribution	Sensitivity—DConcentration per 0.010 DABs
656	DR 5000	50 mg/L SiO ₂	48–52 mg/L SiO ₂	1.0 mg/L SiO ₂
	DR 2800	50 mg/L SiO ₂	48–52 mg/L SiO ₂	1.0 mg/L SiO ₂
	DR 2700	50 mg/L SiO ₂	48–52 mg/L SiO ₂	1.0 mg/L SiO ₂
	DR/2500	50 mg/L SiO ₂	46.9–53.1 mg/L SiO ₂	1.2 mg/L SiO ₂
	DR/2400	50 mg/L SiO ₂	43.2–56.8 mg/L SiO ₂	1.1 mg/L SiO ₂

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. Test results are measured at 452 nm.

Consumables and replacement items

Required reagents

Description	Quantity/Test	Unit	Catalog number
High Range Silica Reagent Set for 10-mL samples (100 tests), includes:			2429600
Acid Reagent Powder Pillows for High Range Silica	1	100/pkg	2107469
Citric Acid Powder Pillows	1	100/pkg	2106269
Molybdate Reagent Powder Pillows for High Range Silica	1	100/pkg	2107369
Water, deionized	10 mL	4 L	27256

Recommended standards

Description	Unit	Catalog number
Silica Standard Solution, 50-mg/L	200 mL	111729
Silica Standard Solution, 1000-mg/L	500 mL	19449

Optional reagents and apparatus

Description	Unit	Catalog number
Sodium Bicarbonate	454 grams	77601
Sulfuric Acid 1.00 N	100 mL	127032
Sampling Bottle with cap, low density polyethylene, 250 mL	12/pkg	2087076
Thermometer, Non-Mercury, -10 to 225 °C	each	2635700



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; E-mail – techhelp@hach.com

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Telephone: (970) 669-3050
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