

Silicomolybdate Method

Method 8185
1 to 100 mg/L SiO₂ (HR, spectrophotometers)
Powder Pillows
1 to 75 mg/L SiO₂ (HR, colorimeters)

Scope and application: For water and seawater.





Test preparation

Instrument-specific information

Table 1 shows all of the instruments that have the program for this test. The table also shows sample cell and orientation requirements for reagent addition tests, such as powder pillow or bulk reagent tests.

To use the table, select an instrument, then read across to find the applicable information for this test.

Table 1 Instrument-specific information

Instrument	Sample cell orientation	Sample cell
DR 6000 DR 3800 DR 2800 DR 2700 DR 1900	The fill line is to the right.	2495402 
DR 5000 DR 3900	The fill line is toward the user.	
DR 900	The orientation mark is toward the user.	2401906 

Before starting

Install the instrument cap on the DR 900 cell holder before ZERO or READ is pushed.

The sample temperature must be between 15–25 °C (59–77 °F) for accurate results.

Use the Standard Adjust option with each new lot of reagent for the best results. Refer to the Standard solution method in [Accuracy check](#) on page 4.

Review the Safety Data Sheets (MSDS/SDS) for the chemicals that are used. Use the recommended personal protective equipment.

Dispose of reacted solutions according to local, state and federal regulations. Refer to the Safety Data Sheets for disposal information for unused reagents. Refer to the environmental, health and safety staff for your facility and/or local regulatory agencies for further disposal information.

Items to collect

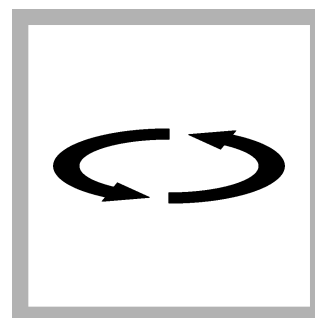
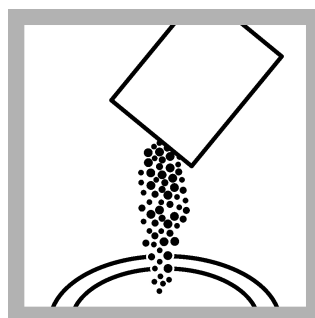
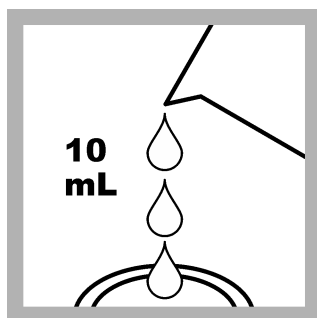
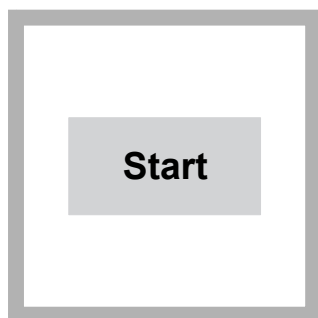
Description	Quantity
High Range Silica Reagent Set	1
Sample cells (For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.)	2

Refer to [Consumables and replacement items](#) on page 5 for order information.

Sample collection

- Collect samples in clean plastic bottles with tight-fitting caps. Do not use glass bottles, which will contaminate the sample.
- Analyze the samples as soon as possible for best results.
- If prompt analysis is not possible, keep the samples at or below 6 °C (43 °F) for up to 28 days.
- Let the sample temperature increase to room temperature before analysis.

Powder pillow procedure



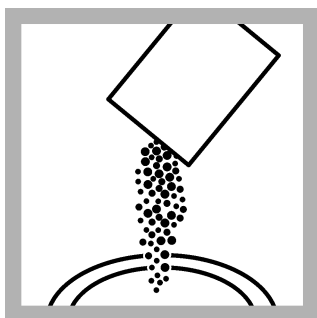
1. Start program 656 Silica HR. For information about sample cells, adapters or light shields, refer to [Instrument-specific information](#) on page 1.

Note: Although the program name can be different between instruments, the program number does not change.

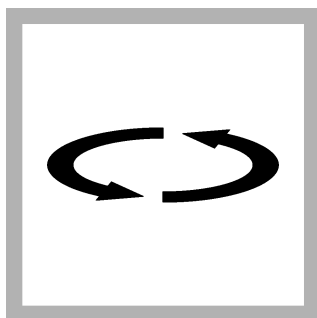
2. Prepare the sample: Fill a sample cell with 10 mL of sample.

3. Add the contents of one Molybdate Reagent Powder Pillow for High Range Silica to the sample cell.

4. Swirl until the reagent is completely dissolved.



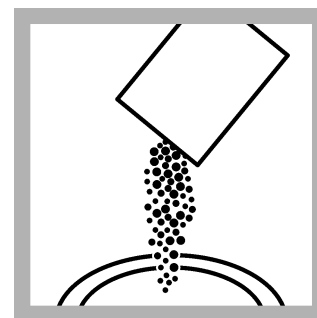
5. Add the contents of one Acid Reagent Powder Pillow for High Range Silica. A yellow color shows if silica or phosphorus is present in the sample.



6. Swirl to mix.



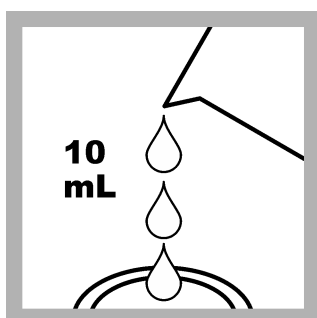
7. Start the instrument timer. A 10-minute reaction time starts.



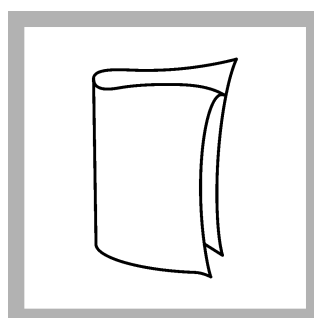
8. When the timer expires, add the contents of one Citric Acid Powder Pillow to the sample cell and swirl to mix. Any yellow color caused by phosphorous is removed during this step.



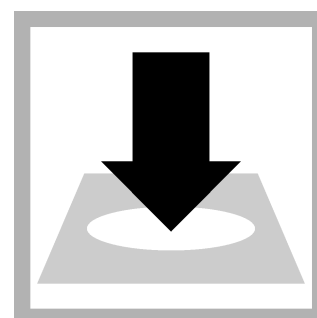
9. Start the instrument timer. A 2-minute reaction time starts. Complete the rest of the procedure within 3 minutes after the timer expires.



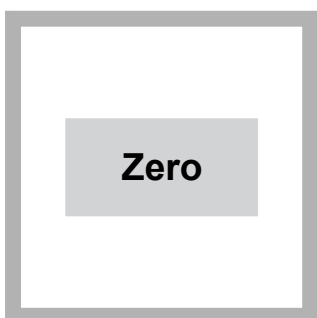
10. Prepare the blank: Fill a second sample cell with 10 mL of the original sample.



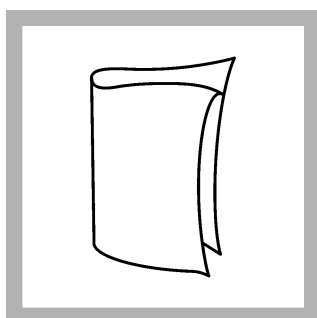
11. When the timer expires, clean the blank sample cell.



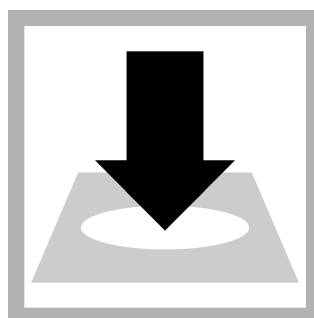
12. Insert the blank into the cell holder.



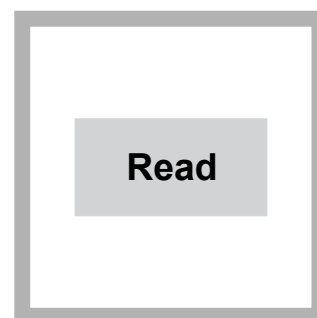
13. Push **ZERO**. The display shows 0 mg/L SiO₂.



14. Clean the prepared sample cell.



15. Insert the prepared sample into the cell holder.



16. Push **READ**. Results show in mg/L SiO₂.

Interferences

Interfering substance	Interference level
Color	Does not interfere when the original sample is used to zero the instrument.
Iron	Large amounts of both ferrous and ferric iron interfere.
Phosphate	Does not interfere at levels less than 50 mg/L PO ₄ ³⁻ . At 60 mg/L PO ₄ ³⁻ , an interference of -2% occurs. At 75 mg/L PO ₄ ³⁻ , the interference is -11%.

Interfering substance	Interference level
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 the citric acid is added) can help as an alternative to the bicarbonate pretreatment.
Sulfides	Interfere at all levels.
Turbidity	Small amounts of turbidity do not interfere when the original sample is used to zero the instrument.

Accuracy check

Standard additions method (sample spike)

Use the standard additions method (for applicable instruments) to validate the test procedure, reagents and instrument and to find if there is an interference in the sample.

Items to collect:

- Silica Standard Solution, 1000-mg/L
 - Pipet, TenSette®, 0.1–1.0 mL
 - Pipet tips
1. Use the test procedure to measure the concentration of the sample, then keep the (unspiked) sample in the instrument.
 2. Go to the Standard Additions option in the instrument menu.
 3. Select the values for standard concentration, sample volume and spike volumes.
 4. Open the standard solution.
 5. Prepare three spiked samples: use the TenSette pipet to add 0.1 mL, 0.2 mL and 0.3 mL of the standard solution, respectively, to three 10-mL portions of fresh sample. Mix well.
 6. Use the test procedure to measure the concentration of each of the spiked samples. Start with the smallest sample spike. Measure each of the spiked samples in the instrument.
 7. Select **Graph** to compare the expected results to the actual results.

Note: *If the actual results are significantly different from the expected results, make sure that the sample volumes and sample spikes are measured accurately. The sample volumes and sample spikes that are used should agree with the selections in the standard additions menu. If the results are not within acceptable limits, the sample may contain an interference.*

Standard solution method

Use the standard solution method to validate the test procedure, the reagents and the instrument.

Items to collect:

- Silica Standard Solution, 50-mg/L
1. Use the test procedure to measure the concentration of the standard solution.
 2. Compare the expected result to the actual result.

Note: *The factory calibration can be adjusted slightly with the standard adjust option so that the instrument shows the expected value of the standard solution. The adjusted calibration is then used for all test results. This adjustment can increase the test accuracy when there are slight variations in the reagents or instruments.*

Method performance

The method performance data that follows was derived from laboratory tests that were measured on a spectrophotometer during ideal test conditions. Users can get different results under different test conditions.

Program	Standard	Precision (95% confidence interval)	Sensitivity Concentration change per 0.010 Abs change
656	50 mg/L SiO ₂	48–52 mg/L SiO ₂	1.0 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. The measurement wavelength is 452 nm for spectrophotometers or 420 nm for colorimeters.

Consumables and replacement items

Required reagents

Description	Quantity/test	Unit	Item no.
Water, deionized	varies	4 L	27256
High Range Silica Reagent Set, 10-mL	—	100 tests	2429600
Includes:			
Acid Reagent Powder Pillow for High Range Silica, 10-mL	1	100/pkg	2107469
Citric Acid Powder Pillow, 10-mL	2	100/pkg	2106269
Molybdate Reagent Powder Pillow for High Range Silica, 10-mL	1	100/pkg	2107369

Recommended standards

Description	Unit	Item no.
Silica Standard Solution, 50-mg/L as SiO ₂	200 mL	111729
Silica Standard Solution, 1000-mg/L as SiO ₂	500 mL	19449

Optional reagents and apparatus

Description	Unit	Item no.
Sodium Bicarbonate	454 g	77601
Sulfuric Acid Standard Solution, 1 N	100 mL MDB	127032
Bottle, sampling, with cap, low density polyethylene, 250-mL	12/pkg	2087076
Thermometer, non-mercury, -10 to +225 °C	each	2635700



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