Silica

DOC316.53.01472

Silicomolybdate Method

Method 8185

1 to 100 mg/L SiO_2

Powder Pillows

Scope and application: For water and seawater.

Test preparation

Before starting

The sample temperature must be between 15-25 °C (59-77 °F) for accurate results.
Always do tests in sample cells. Do not put the instrument in the sample or pour the sample into the cell holder.
Make sure that the sample cells are clean and there are no scratches where the light passes through them.
Rinse the sample cell and cap with the sample three times before the sample cell is filled.
Make sure that there are no fingerprints or liquid on the external surface of the sample cells. Wipe with a lint-free cloth before measurement.
Cold waters can cause condensation on the sample cell or bubbles in the sample cell during color development. Examine the sample cell for condensation or bubbles. Remove condensation with a lint-free cloth. Invert the sample cell to remove bubbles.
Install the instrument cap over the cell holder before ZERO or READ is pushed.
After the test, immediately empty and rinse the sample cell. Rinse the sample cell and cap three times with deionized water.
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

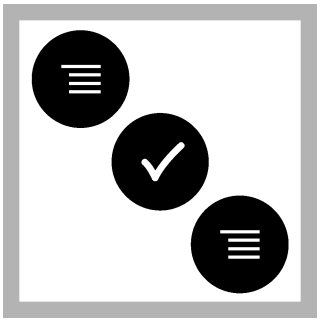
Description	Quantity
High Range SIlica Reagent Set, 10-mL	1
Sample cells, 25-mm (10 mL)	2

Refer to Consumables and replacement items on page 4 for order information.

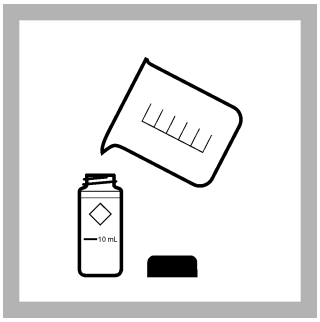
Sample collection

- 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 7 days.
- Let the sample temperature increase to room temperature before analysis.

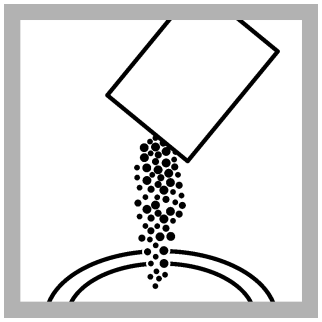
Powder pillow procedure



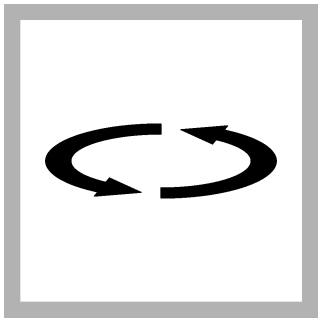
1. Set the instrument to mg/L SiO_2. Refer to the instrument documentation.



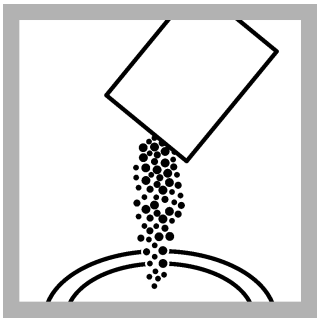
2. Prepare the sample: Fill a sample cell to the 10-mL mark with sample.



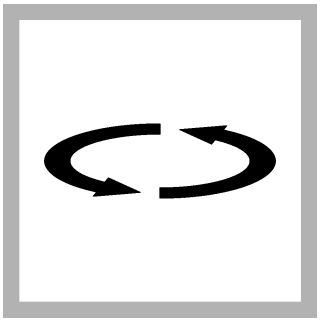
3. Add the contents of one Molybdate Reagent Powder Pillow for High Range Silica to the sample cell.



4. Swirl until the reagent is completely dissolved.

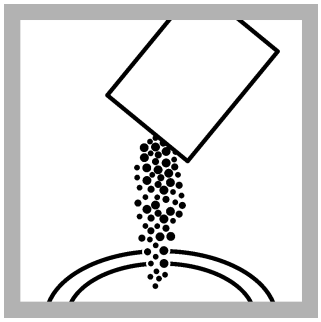


5. Add the contents of one Acid Reagent Powder Pillow for High Range Silica.
A yellow color will show if silica or phosphorus is in the sample.



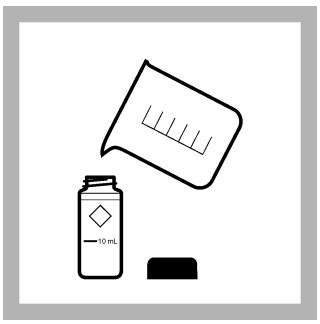
6. Swirl to mix.

7. Set and start a timer for 10 minutes. A 10-minute reaction time starts.

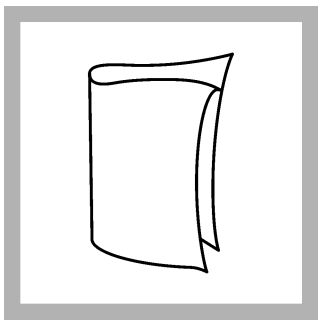


8. When the timer expires, add the contents of one Citric Acid Powder Pillow to the sample cell and swirl to mix.
Any yellow color caused by phosphorous is removed during this step.

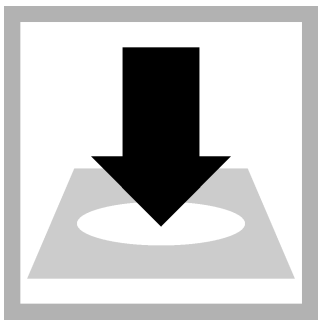
9. Set and start a timer for 2 minutes. A 2-minute reaction time starts.



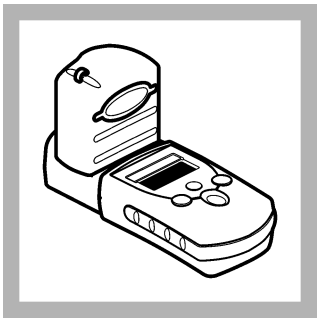
10. Prepare the blank: Fill a sample cell to the 10-mL mark with sample. Close the sample cell.



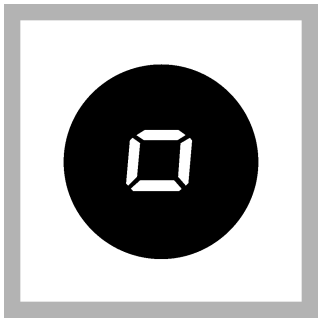
11. When the timer expires, clean the blank sample cell.



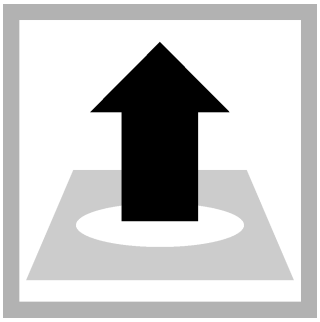
12. Insert the blank into the cell holder. Point the diamond mark on the sample cell toward the keypad.



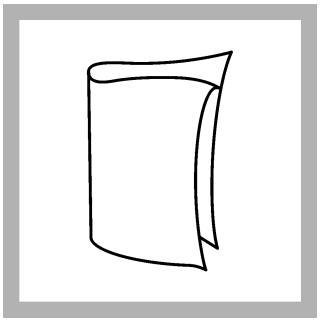
13. Install the instrument cap over the cell holder.



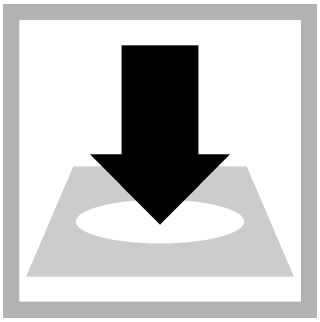
14. Push **ZERO**. The display shows “0”.



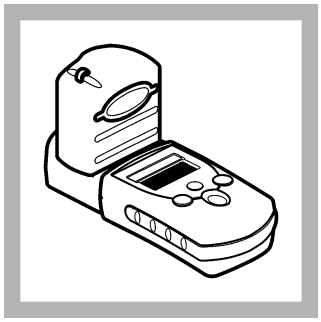
15. Remove the sample cell from the cell holder.



16. Clean the prepared sample cell.



17. Within 3 minutes after the timer expires, insert the prepared sample into the cell holder. Point the diamond mark on the sample cell toward the keypad.



18. Install the instrument cap over the cell holder.

19. Push READ. Results show in mg/L silica (SiO_2).

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%.
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 *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 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

Use the standard additions method 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
- Sample, 70 mg/L SiO_2 or less
- Pipet, TenSette®, 0.1–1.0 mL and tips

1. 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.
2. 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.
3. Compare the expected result to the actual result. The expected silica concentration increase is 10 mg/L for each 0.1 mL of standard that is added.

Standard solution method

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

Items to collect:

- Silica Standard Solution, 10-, 25- or 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 calibration 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 Pocket Colorimeter II during ideal test conditions. Users can get different results under different test conditions.

Precision (95% confidence interval)
50 ± 2 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.

Consumables and replacement items

Required reagents

Description	Quantity/test	Unit	Item no.
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

Required apparatus

Description	Quantity/test	Unit	Item no.
Sample cells, 10-mL round, 25 mm x 60 mm	2	6/pkg	2427606

Optional reagents and apparatus

Description	Unit	Item no.
Silica Standard Solution, 1000-mg/L as SiO_2	500 mL	19449
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
Pipet, TenSette®, 0.1–1.0 mL	each	1970001
Pipet tips for TenSette® Pipet, 0.1–1.0 mL	50/pkg	2185696
Pipet tips for TenSette® Pipet, 0.1–1.0 mL	1000/pkg	2185628
Thermometer, –10 to 110 °C	each	187701

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04/2014, Edition 1