# Silica

DOC316.53.01132

## Heteropoly Blue Method[1]

**Method 8186**

**0.010 to 1.600 mg/L $SiO_2$ (spectrophotometers)**

**Powder Pillows**

**0.01 to 1.60 mg/L $SiO_2$ (colorimeters)**

**Scope and application:** For boiler and ultrapure water.

[1] Adapted from Standard Methods for the Examination of Water and Wastewater.

## 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<br>DR 3800<br>DR 2800<br>DR 2700<br>DR 1900 | The fill line is to the right. | 2495402<br>  |
| DR 5000<br>DR 3900 | The fill line is toward the user. | |
| DR 900 | The orientation mark is toward the user. | 2401906<br>  |

### Before starting

| |
|---|
| Install the instrument cap on the DR900 cell holder before ZERO or READ is pushed. |
| The reaction times in the test procedure are for samples that are at 20 °C (68 °F). If the sample temperature is 10 °C (50 °F), wait 8 minutes for the first (4-minute) reaction time and 2 minutes for the second (1-minute) reaction time. If the sample temperature is 30 °C (86 °F), wait 2 minutes for the first (4-minute) reaction time and 30 seconds for the second (1-minute) reaction time. |
| To test for very low concentrations, use the ULR rapid liquid method for the best results. |
| 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

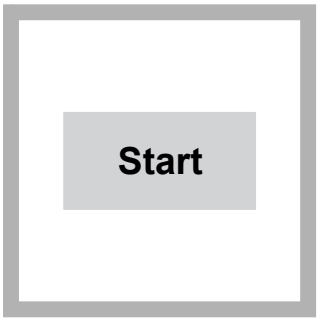
| Description | Quantity |
|---|---|
| Amino Acid F Reagent powder pillows, 10-mL | 1 |
| Citric acid powder pillows, 10-mL | 2 |
| Molybdate 3 Reagent solution | 1 mL |
| Sample cells (For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.) | 2 |

Refer to Consumables and replacement items on page 5 for order information.

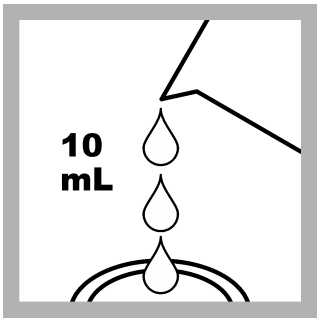
## Sample collection

- 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.

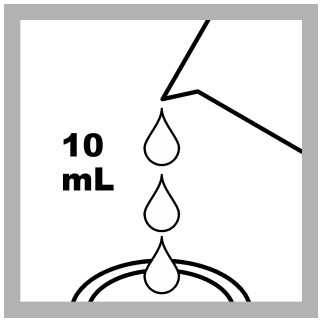
## Powder pillow procedure



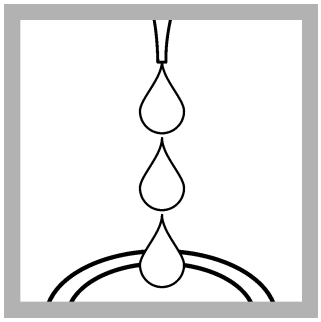

**1.** Start program **651 Silica LR**. For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.



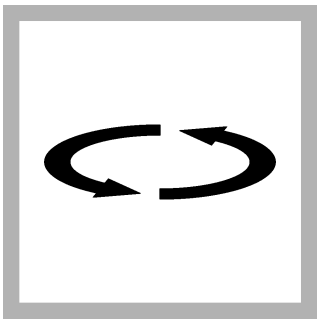

**2. Prepare the blank:** Fill a sample cell with 10 mL of sample.




**3. Prepare the sample:** Fill a second sample cell with 10 mL of sample.

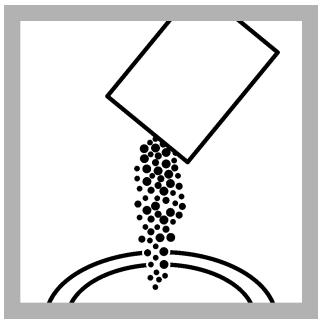


**4.** Add 14 drops of Molybdate 3 reagent solution to each cell.

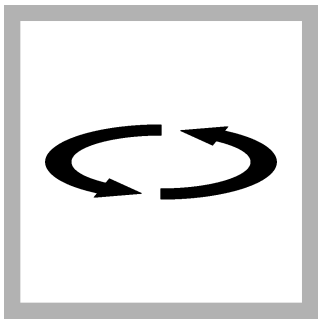


**5.** Swirl to mix.


**6.** Start the instrument timer. A 4-minute reaction time starts.



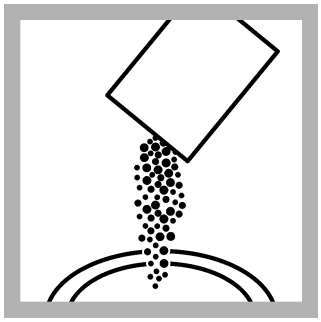
**7.** After the timer expires, add the contents of one Citric Acid Reagent powder pillow to each sample cell.



**8.** Swirl to mix.

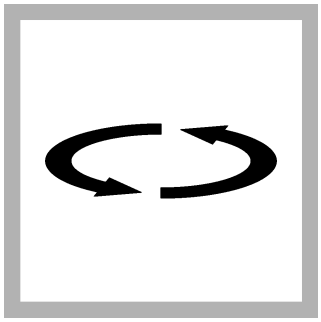

**9.** Start the instrument timer. A 1-minute reaction time starts.

The destruction of possible phosphate interference occurs during this period.



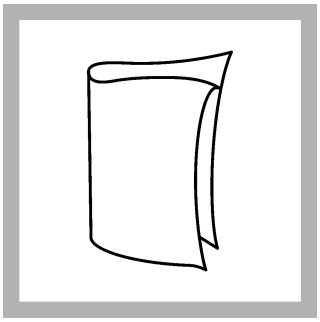
**10.** After the timer expires, add the contents of one Amino Acid F Reagent powder pillow to the prepared sample cell.

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

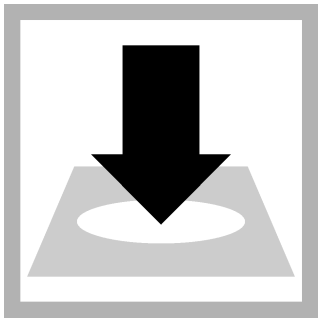


**11.** Swirl to mix.

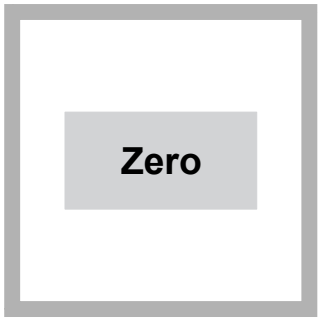

**12.** Start the instrument timer. A 2-minute reaction time starts.
A blue color shows if silica is present.



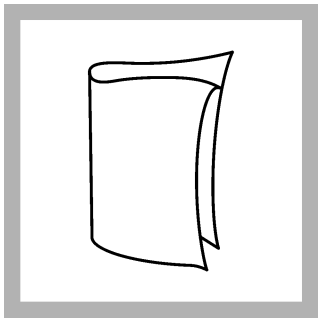
**13.** When the timer expires, clean the blank sample cell.



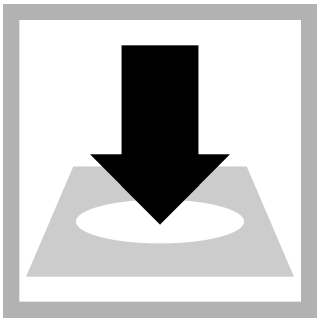
**14.** Insert the blank into the cell holder.




**15.** Push **ZERO**. The display shows 0.000 mg/L $SiO_2$. .



**16.** Clean the prepared sample cell.



**17.** Insert the prepared sample into the cell holder.


**18.** Push **READ**. Results show in mg/L $SiO_2$.

## 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%. |

| 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 *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 (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, 25 mg/L $SiO_2$
- 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, 1.00-mg/L $SiO_2$

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 small 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<br>Concentration change per 0.010 Abs change |
|---|---|---|---|
| 651 | 1.000 mg/L $SiO_2$ | 0.990–1.010 mg/L $SiO_2$ | 0.012 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. An amino acid is then added to reduce the yellow silicomolybdic acid to an intense blue color, which is proportional to the silica concentration. The measurement wavelength is 815 nm for spectrophotometers (DR 1900: 800 nm) or 610 nm for colorimeters.

## Consumables and replacement items

### Required reagents

| Description | Quantity/Test | Unit | Item no. |
|---|---|---|---|
| Silica Reagent Set, low range, includes: | — | 100 tests | 2459300 |
| Amino Acid F Reagent Powder Pillow, 10 mL | 1 | 100/pkg | 2254069 |
| Citric Acid Powder Pillow, 10 mL | 2 | 100/pkg | 2106269 |
| Molybdate 3 Reagent Solution | 1 mL | 50 mL | 199526 |

### Recommended standards and apparatus

| Description | Unit | Item no. |
|---|---|---|
| Silica Standard Solution, 1-mg/L $SiO_2$ | 500 mL | 110649 |
| Silica Standard Solution, 25 mg/L as $SiO_2$ | 236 mL | 2122531 |
| Water, deionized | 4 L | 27256 |

### Optional reagents and apparatus

| Description | Unit | Item no. |
|---|---|---|
| Sodium Bicarbonate | 454 g | 77601 |
| Sulfuric Acid Solution, 1.00 N | 1000 mL | 127053 |
| 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 |

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