Silica

DOC316.53.01337

Heteropoly Blue Method[1]

3 to 1000 µg/L SiO_2

Method 8282

Reagent Solution

Scope and application: For pure 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 the sample cell and adapter requirements.

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

Table 1 Instrument-specific information

Instrument	Adapter	Sample cell
DR 6000	LZV902.99.00020	2410212 
DR 5000	A23618	
DR 3900, DR 3800	—	
DR 2800	LZV585 (B)	
DR 1900	—	

Before starting

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.
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 solution	0.5 mL
Citric Acid F Reagent solution	0.5 mL
Molybdate 3 Reagent solution	0.5 mL
Pipet, TenSette®, 0.1 to 1.0 mL with tips	1

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

Sample collection

- Samples must be analyzed immediately after collection and cannot be preserved for later analysis.

- Collect samples in clean plastic bottles with tight-fitting caps. Do not use glass bottles, which will contaminate the sample.
- Soak the sample containers for several hours in a solution of one part Molybdate 3 Reagent to 50 parts of high quality deionized water of low silica concentration. Fully rinse with low-level silica water, drain and close. Repeat this cleaning periodically.
- Make sure to get a representative sample. It the sample is taken from a spigot or faucet, let the water flow for at least 1 or 2 minutes. Do not adjust the flow because this can add particulates.

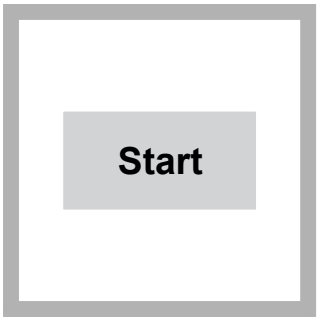
Clean the sample cell

Fully clean the plastic sample cell to remove traces of silica.

Note: *It is not necessary to clean the plastic sample cell before each test. Clean only when contamination has occurred.*

1. Add 1 ml of the Molybdate 3 Reagent to the plastic sample cell, then fill with deionized water.
2. Put on the cap. Wait 15 to 30 minutes.
3. Remove the cap. Rinse several times with low-level silica water or the sample.
4. When not in use, keep the cap on.

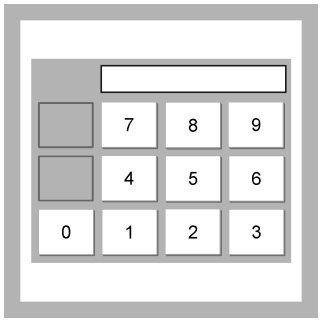
Heteropoly Blue procedure



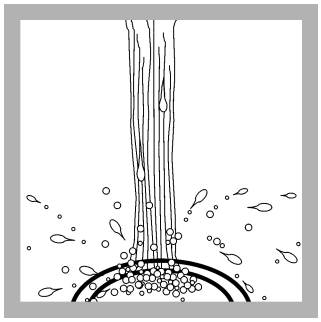
1. Start program **645 Silica ULR**. For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.

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

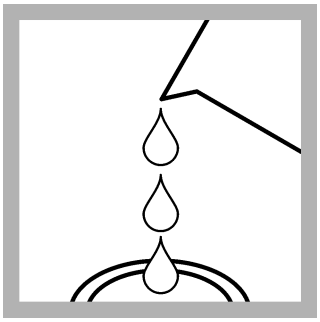
2. Push **Reagent Blank On**.



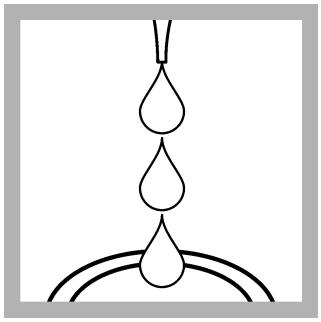
3. Enter the value that is recorded on the bottle of Molybdate 3 Reagent Solution.



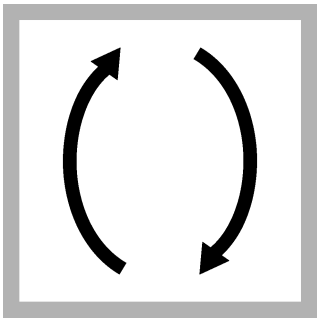
4. Rinse a clean plastic sample cell and the cap three times with the sample. Do not let the interior surface of the cap touch surfaces that can have contamination.



5. Fill the sample cell to the 25-mL mark with sample.

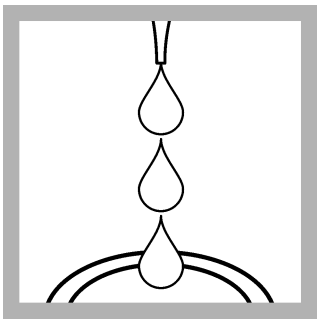


6. Use a pipet to add 0.5 mL of Molybdate 3 Reagent solution to the sample cell.

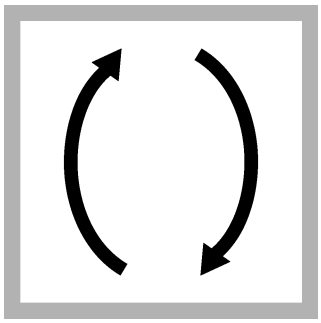


7. Put the cap on the vial. Invert several times to mix.

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



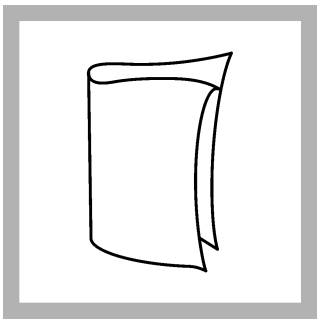
9. After the timer expires, use a pipet to add 0.5 mL of Citric Acid F Reagent solution to the sample cell.



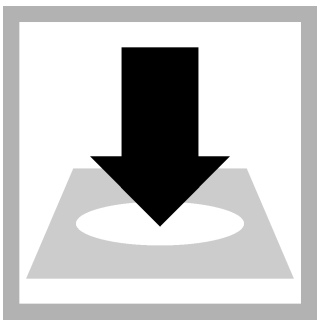
10. Put the cap on the vial. Invert several times to mix.

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

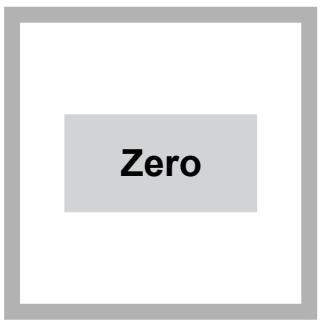
The addition of citric acid destroys possible phosphate interference.



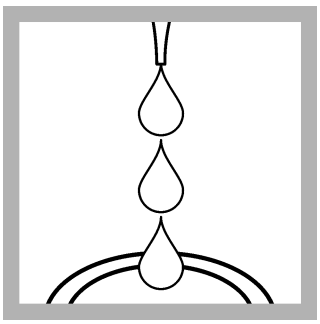
12. Clean the prepared sample cell.



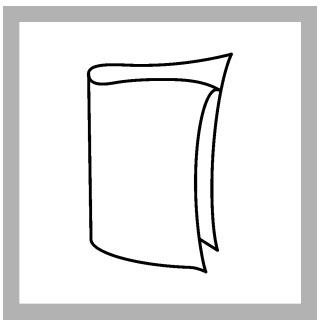
13. Insert the prepared sample into the cell holder.



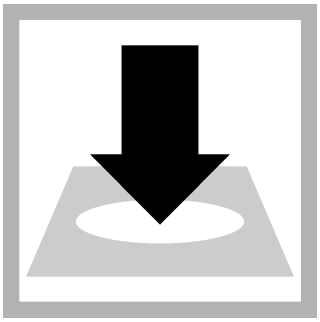
14. Push **ZERO**. The display shows 0 µg/L SiO_2.



15. Use a pipet to add 0.5 mL of Amino Acid F Reagent solution to the sample cell.
A blue color shows if silica is present. Wait a minimum of 15 seconds for full color development.



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.
Highly buffered samples or extreme sample pH	Can prevent the correct pH adjustment of the sample by the reagents. Sample pre-treatment may be necessary. Adjust the pH to less than 7.
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%.
Sulfides	Interfere at all levels.
Turbidity	Does not interfere when the original sample is used to zero the instrument.

Reagent preparation (optional)

To prepare larger or smaller volumes of Amino Acid F Reagent Solution, dissolve Amino Acid F Reagent Powder in Amino Acid F Reagent Solvent at a ratio of 11 grams per 100 mL of reagent solvent. These reagents are available as the Amino Acid F Reagent Package. This prepared solution has limited stability, use the standard solution method to make sure that the reagent continues to give the correct result.

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 µg/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 25 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, 500-µg/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 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
645	500 µg/L SiO_2	496–504 µg/L SiO_2	13 µg/L SiO_2

Summary of method

A number of changes are necessary to adapt the Low Range Silica method to analyze trace levels in the Ultra Low Range method. Liquid reagents are used because they give more reproducible readings and lower blank values. Powdered reagents are not used because they can cause light turbidity.

Silica and phosphate in the sample react with molybdate ions under acidic conditions to form yellow silicomolybdic acid complexes and phosphomolybdic acid complexes. The addition of citric acid destroys the phosphate complexes. Amino Acid F Reagent is then added to decrease the yellow silicomolybdic acid to an intense blue color, which is proportional to the silica concentration. The measurement wavelength is 815 nm (DR 1900: 800 nm).

Consumables and replacement items

Required reagents

Description	Quantity/Test	Unit	Item no.
Silica Reagent Set, ultra low range, includes:	—	100 tests	2553500
Molybdate 3 Reagent Solution	2 mL	100 mL	199532
Citric Acid Reagent Solution	2 mL	100 mL	2254232
Amino Acid F Reagent	1 mL	100 mL	2386442
Silica Reagent Set, ultra low range, includes:	—	40 tests	2581400
Molybdate 3 Reagent Solution	2 mL	100 mL	199532
Citric Acid Reagent Solution	2 mL	100 mL	2254232
Amino Acid F Reagent, 1.2-mL ampule	1	20/pkg	2386420

Required apparatus

Description	Quantity/Test	Unit	Item no.
Pipet, TenSette®, 0.1–1.0 mL	1	each	1970001
Pipet Tips, for TenSette® Pipet, 0.1–1.0 mL	5	50/pkg	2185696
Sample cell, 1-inch square plastic, with cap	1	12/pkg	2410212

Recommended standards

Description	Unit	Item no.
Silica Standard Solution, 1-mg/L SiO_2	500 mL	110649
Silica Standard Solution, 500-µg/L as SiO_2	3.78 L	2100817
Water, deionized	4 L	27256

Optional reagents and apparatus

Description	Unit	Item no.
Ammonium Hydroxide, 58%	500 mL	10649
Molybdate 3 Reagent Solution	2.9 L	199503
Molybdate 3 Reagent Solution	3.78 L	199517
Molybdate 3 Reagent Solution	100 mL	199532
Molybdate 3 Reagent Solution	1 L	199553
PourRite® Ampule Breaker, 2-mL	each	2484600
Amino Acid F Reagent Package	each	2254117
Sampling bottle with cap, low density polyethylene, 500-mL	12/pkg	2087079
Thermometer, non-mercury, –10 to +225 °C	each	2635700

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