Silica

DOC316.53.01130

Heteropoly Blue Method[1]

Method 8282

ULR 3 to 1000 µg/L SiO_2

Pour-Thru Cell

Scope and application: For testing trace levels of soluble silica in pure and ultrapure water.

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

Test preparation

Instrument specific table

Table 1 shows all of the instruments that have the program for this test. The table also shows sample cell and orientation 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	Sample cell orientation	Pour-Thru Kit	Adapter	Sipper Cell Kit
DR 6000	The flow path is to the right.	LZV899, LZQ105 and LZQ102	—	LQV157.99.20002
DR 3800		5940400	LZV585 (B)	—
DR 2800		5940400	LZV585 (B)	—
DR 2700		5940400	LZV585 (B)	—
DR 1900		LZV899	—	—
DR 5000	The flow path is toward the user.	LZV479	—	—
DR 3900		LZV899	—	LQV157.99.10002

Before starting

Refer to the instrument documentation for Pour-Thru cell and module assembly and installation. Make sure to install the Pour-Thru cell correctly.
To protect the Pour-Thru Cell from contamination when not in use, invert a small beaker over the top of the glass funnel.
Refer to Clean the labware on page 5 and to Clean the Pour-Thru Cell on page 5 to clean the Pour-Thru Cell and all labware.
Refer to Prepare the reagents on page 4 to prepare the Amino Acid F Reagent.
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	1
Citric Acid F Reagent	1
Molybdate 3 Reagent	1

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Items to collect (continued)

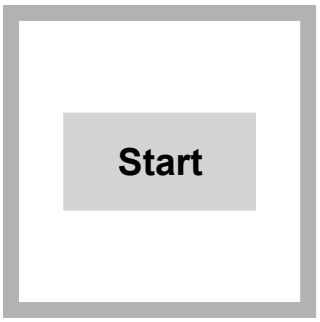
Description	Quantity
Cylinder, graduated, 50 mL, polypropylene	1
Flask, Polymethylpentene, screw cap, 250-mL	2
Pipet, TenSette®, 0.1–1.0 mL	1
Pipet tips, for TenSette® Pipet, 0.1–1.0 mL	2
Pour-Thru cell kit (refer to Table 1 on page 1)	1

Refer to Consumables and replacement items on page 6 for order information.

Sample collection

- Analyze the samples immediately. The samples 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.

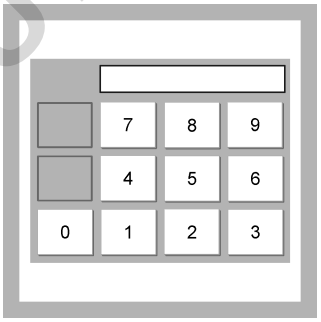
Test procedure



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



2. Set the reagent blank option to on to automatically subtract the reagent blank value from the sample results.

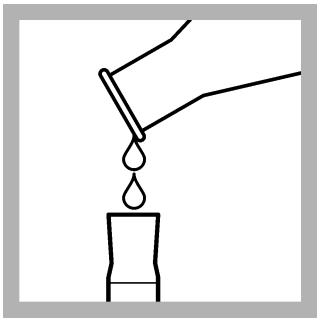


3. Enter the reagent blank value. The value is printed on the Molybdate 3 reagent label.

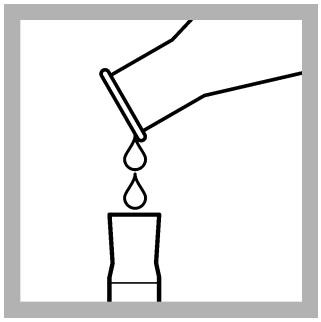


4. Fill two clean 250-mL Erlenmeyer flasks to overflowing with sample.

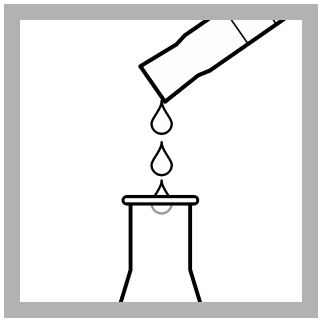
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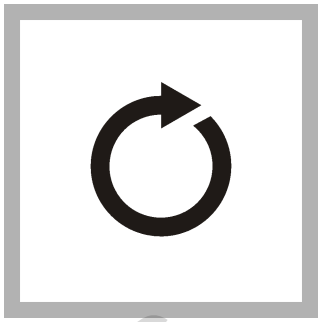
5. Fill a clean 50-mL plastic graduated cylinder with sample from one of the flasks. Then discard the contents of the cylinder. Do this procedure three times.



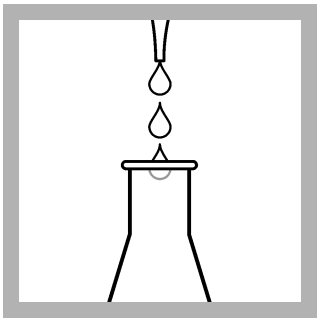
6. Fill the rinsed cylinder to the 50-mL mark with sample from the same flask. Discard any remaining sample in the flask.



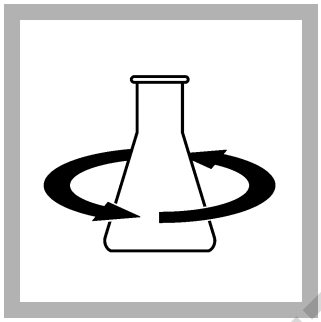
7. Pour the contents of the 50-mL cylinder back into the original flask.



8. Do steps 5 to 7 again for the second sample flask.



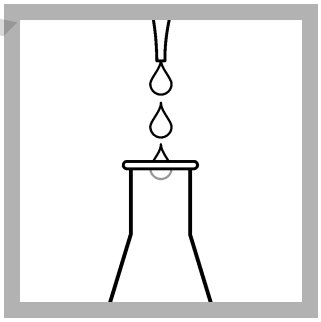
9. Use a TenSette Pipet to add 1.0 mL of Molybdate 3 Reagent to each flask.



10. Swirl to mix.



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



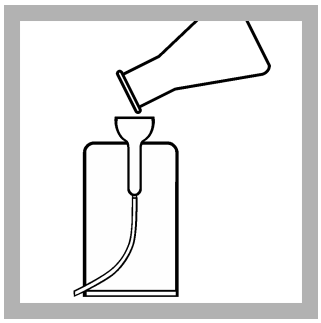
12. When the timer expires, add 1.0 mL of Citric Acid F Reagent to each flask.



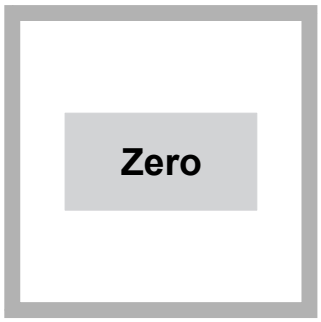
13. Swirl to mix.



14. Start the instrument timer. A 1-minute reaction time starts.
The destruction of possible phosphate interference occurs during this period.

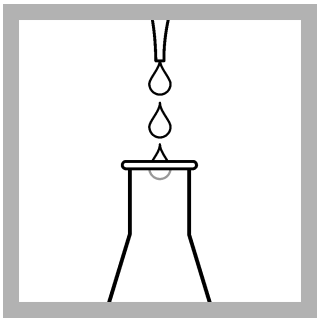


15. When the timer expires, pour the contents of one flask into the Pour-Thru Cell.

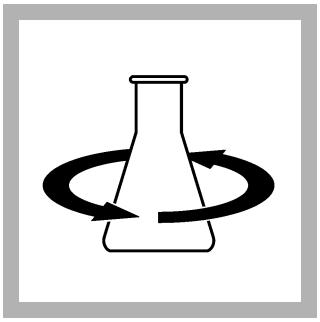


16. When the flow stops, push **ZERO**. The display shows 0 µg/L SiO_2.

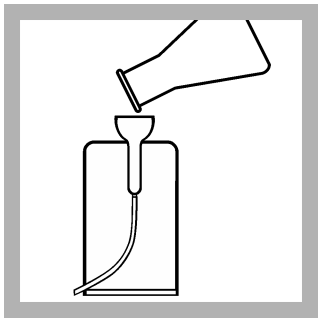
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17. Add 1.0 mL of Amino Acid F Reagent to the remaining flask.



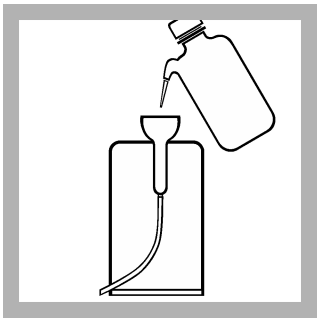
18. Swirl to mix.
A faint blue color forms if silica is in the sample.



19. Wait at least 15 seconds, then pour the contents of the second flask into the Pour-Thru Cell.



20. Push **READ**. Results show in µg/L SiO_2.



21. Flush the Pour-Thru Cell with at least 50-mL of deionized water immediately after use.

Interferences

Interfering substance	Interference level
Color	Eliminated by zeroing the instrument with the blank (follow procedure).
Iron	Interferes at high levels.
pH (extreme)	Adjust pH to less than 7.
Phosphate (PO_4^{3-})	Interferes at levels greater than 50 mg/L PO_4^{3-}.
Sulfides	Interfere at all levels.
Turbidity	Eliminated by zeroing the instrument with the blank (follow procedure).

Prepare the reagents

Amino Acid F Reagent Solution is available in 100-mL bottles or a package of 20 unit-dose ampules. The bottled reagent is stable for maximum of 1 year if the bottle is kept closed when not in use. The reagent ampule is sealed under argon and is more stable with a shelf life of more than 1 year.

Prepare other volumes of Amino Acid F Reagent as follows:

1. Dissolve the contents of one bottle of Amino Acid F Reagent Powder in one bottle of Amino Acid Reagent Dilution Solvent. These reagents are available as the Amino Acid F Reagent Package.
2. Install a bottle-top dispenser on the Amino Acid F, Molybdate 3 and Citric Acid Reagent bottles for fast reagent addition.
3. As an alternative, larger or smaller volumes can be prepared. Dissolve Amino Acid F Reagent Powder in Amino Acid F Reagent Solvent at a ratio of 11 grams per 100 mL of reagent solvent.

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This reagent has a limited stability. Verify the test performance routinely with the 1-mg/L (1000 µg/L) Silica Standard Solution. Reduced sensitivity at high concentrations (1000 µg/L) indicates reagent instability. If the measured concentration is less than 950 µg/L, prepare fresh Amino Acid F Reagent Solution.

Clean the labware

Fully clean all containers that are used in this test to remove any traces of silica. Use plastic containers for all analysis and storage because glass can contaminate the sample with silica. Small bottles or flasks with screw-type closures work well.

1. Clean containers (do not use phosphate detergents), then rinse with high quality deionized water of low-level silica concentration.
2. Soak for 10 minutes with a 1:50 dilution of Molybdate 3 Reagent in low-level silica water.
3. Rinse repeatedly with either low-level silica water or the sample before use. Keep containers tightly closed when not in use.
4. Fill the Pour-Thru Cell with this same mixture of Molybdate 3 and water. Soak for 10 minutes.
5. Rinse with low-level silica water.

Clean the Pour-Thru Cell

The Pour-Thru Cell can collect a buildup of products with color, especially if the reacted solutions stay in the cell for long periods of time after measurement.

1. Rinse the Pour-Thru Cell with a 1:5 dilution of ammonium hydroxide solution to remove the color.
2. Fully rinse with deionized water.
3. Put a cover on the Pour-Thru Cell funnel when it is not in use.

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:

- 1-mg/L (1000-µg/L) Silica standard
- TenSette Pipet and Pipet tips
- 250-mL plastic Erlenmeyer flasks (3x)

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 0.2 mL, 0.4 mL and 0.6 mL of the standard solution, respectively, to three 50 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.*

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Standard solution method

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

Items to collect:

- 500-µg/L SiO_2 standard solution

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 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 Concentration change per 0.010 Abs change
645	500 µg/L silica	496–504 µg/L silica	13 µg/L silica

Summary of method

A number of modifications are necessary to adapt the Low Range Silica method for analyzing trace levels in the Ultra Low Range method. It is absolutely necessary to use the 1-inch Pour-Thru Cell and liquid reagents. The Pour-Thru Cell increases the reproducibility of the optics and reduces the instability of the readings that result from moveable sample cells. Liquid reagents produce more reproducible readings and lower blank values by eliminating slight turbidity that can remain with powdered reagents.

Silica and phosphate in the sample react with molybdate ions under acidic conditions to form yellow silicomolybdic acid complexes and phosphomolybdic acid complexes. Addition of citric acid destroys the phosphate complexes. Amino Acid F Reagent 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).

Consumables and replacement items

Required reagents

Description	Quantity/test	Unit	Item no.
ULR Silica Reagent Set (using Amino Acid F solution, 100 tests) includes:			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
ULR Silica Reagent Set (using Amino Acid F ampules, 40 tests) includes:			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

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Required apparatus

Description	Quantity/test	Unit	Item no.
Cylinder, graduated, 50 mL, polypropylene	1	each	108141
Flask, Polymethylpentene, screw cap, 250 mL	1	each	2089846
Pipet, TenSette®, 0.1–1.0 mL	1	each	1970001
Pipet tips, for TenSette® Pipet, 0.1–1.0 mL	2	50/pkg	2185696

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

Optional reagents and apparatus

Description	Unit	Item no.
Water, deionized	4 L	27256
Ammonium Hydroxide, 58%	500 mL	10649
Citric Acid Reagent Solution	500 mL	2254249
Molybdate 3 Reagent Solution	500 mL	199549
Molybdate 3 Reagent Solution	1 L	199553
PourRite® Ampule Breaker, 2-mL	each	2484600
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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