DR/4000 PROCEDURE

SILICA

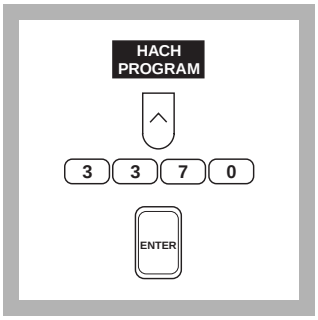
Method 8282

Heteropoly Blue Method*

ULR (0 to 1000.0 µg/L as SiO_2)

Scope and Application: *For testing trace levels of soluble silica in pure and ultrapure water. The estimated detection limit for program number 3370 is 1.0 µg/L.*

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



1. Press the soft key under ***HACH PROGRAM.***

Select the stored program for ultra low range silica by pressing **3370** with the numeric keys.

Press: **ENTER**

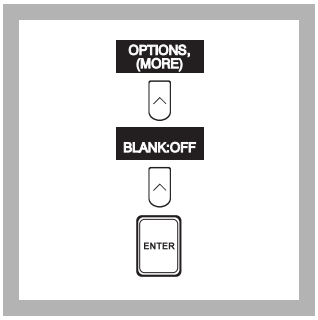
Note: *If samples cannot be analyzed immediately, see Sample Collection, Storage and Preservation following these steps.*

Note: *The Flow Cell and Sipper Modules must be used with this procedure. See Summary of Method.*

2. The display will show:

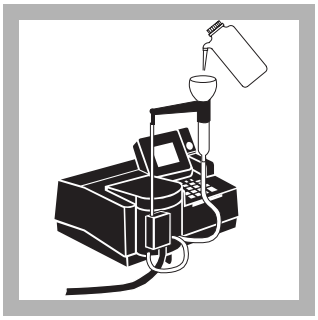
HACH PROGRAM: 3370 Silica, ULR

The wavelength (λ), **815 nm**, is automatically selected.



3. Account for the Molybdate 3 reagent blank by pressing the soft keys under ***OPTIONS***, ***(MORE),*** and then ***BLANK:OFF***. Enter the reagent blank value found on the Molybdate 3 Reagent label and press **ENTER** until the read screen appears.

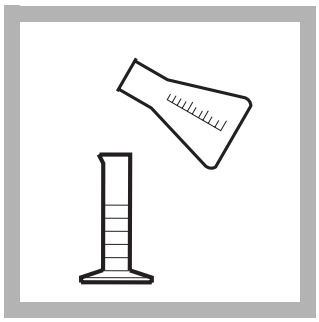
Note: *Reagent blank values printed on analyzer reagent containers vary because the reagent dilutions vary according to the instrument. For this method, use the 1234D analyzer reagent blank value for a 3.78-liter volume of Molybdate 3 Reagent (Cat No. 1995-17). For a Series 5000, 2.9-liter volume of Molybdate 3 Reagent (Cat. No. 1995-03), multiply the reagent blank on the label by 1.09. For 100-mL (Cat No. 1995-32) and 1-liter (Cat. No. 1995-53) volumes, use the lab blank values on the bottle labels.*



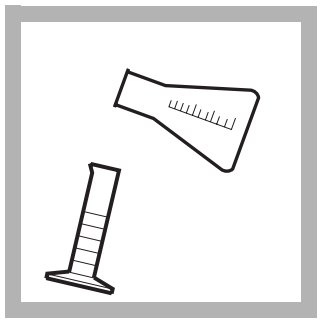
4. Insert the Flow Cell or Sipper Module and flush with 50 mL of low-silica deionized water.

Note: *Clean labware carefully; see more information in Labware following these steps.*

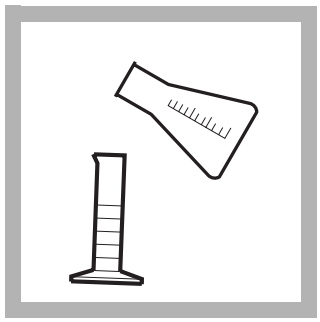
5. Fill two clean 250-mL Erlenmeyer flasks to overflowing with the sample to be tested.



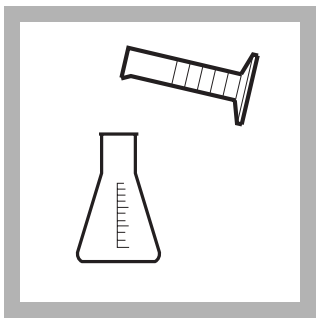
6. Fill a clean 50-mL plastic graduated cylinder with sample from one of the flasks and then discard the cylinder contents.



7. Repeat the rinsing of the cylinder three times from the same sample flask, discarding each rinse.

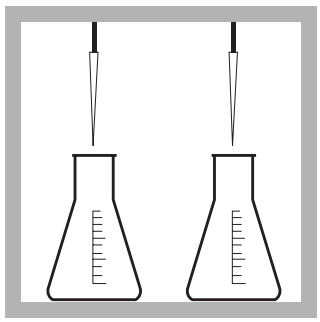


8. Fill this rinsed cylinder to the 50-mL mark with sample from the same flask, discarding any remaining sample in the flask.



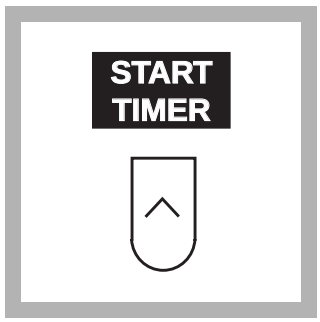
9. Pour the contents of the 50-mL cylinder back into the original flask.

10. Repeat steps 6–10 for the second flask containing sample, then continue with Step 11.



11. Using a TenSette Pipet, add 1.0 mL of Molybdate 3 Reagent to each flask. Swirl to mix.

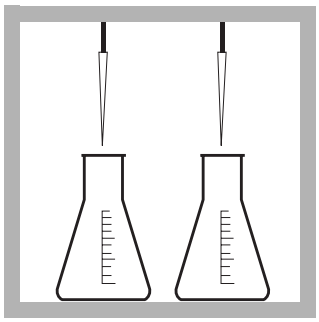
Note: *The TenSette Pipet is recommended for convenient reagent addition. An all-plastic 1.0-mL dropper is also available (See OPTIONAL EQUIPMENT AND SUPPLIES).*



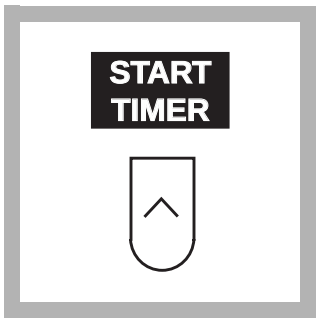
12. Press the soft key under ***START TIMER***.

A 4-minute reaction period will begin.

Note: *Reaction time depends on sample temperature. The time given is for samples at 20 °C (68 °F). If the sample temperature is 10 °C (50 °F), wait 8 minutes. If the sample temperature is 30 °C (86 °F), wait 2 minutes.*



13. When the timer beeps, add 1.0 mL of Citric Acid F Reagent to each flask. Swirl to mix.

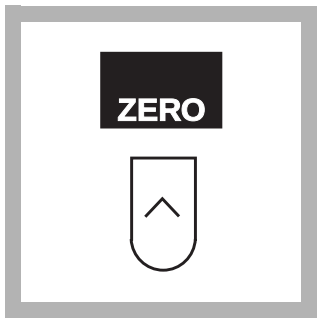


14. Press the soft key under ***START TIMER***.

A one-minute reaction period will begin. The destruction of possible phosphate interference occurs during this period.

Note: *Reaction time depends on sample temperature. The time given is for samples at 20 °C (68 °F). If the sample temperature is 10 °C (50 °F), wait 2 minutes. If the sample temperature is 30 °C (86 °F), wait 30 seconds.*

15. When the timer beeps, pour the contents of one flask into the Flow-Thru Cell or draw the contents into the Sipper Cell.



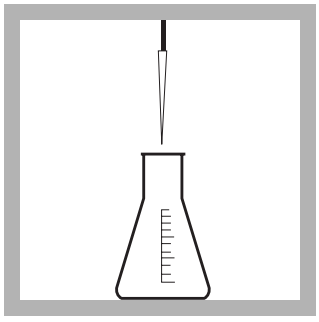
16. After the flow has stopped, press the soft key under ***ZERO***.

The display will show:

-0.0 µg/L SiO_2

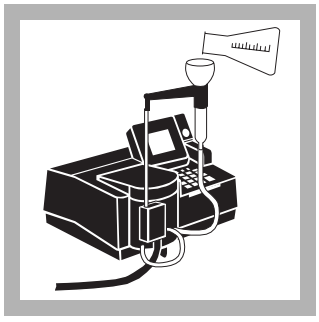
Note: *f you are using a reagent blank correction, the display will show the correction.*

Note: *For alternate concentration units press the soft key under* ***OPTIONS****. Then press the soft key under* ***UNITS*** *to scroll through the available options. Press* **ENTER** *to return to the read screen.*

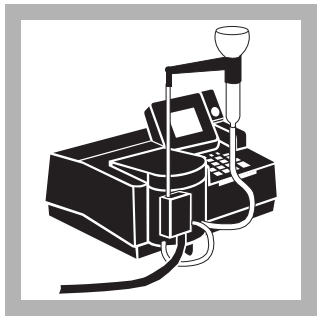


17. Add 1.0 mL of Amino Acid F Reagent to the remaining flask. Swirl to mix.

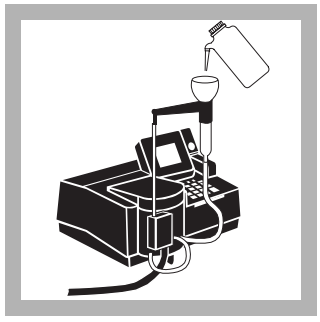
Note: *A faint blue color will develop if silica is present.*



18. Wait 15 seconds (but no more than 5 minutes) for color formation. Transfer the contents of the second flask into the Flow-Thru Cell or draw them into the Sipper Cell.



19. After the flow has stopped, results in µg/L SiO_2 (or chosen units) will be displayed.



20. Flush the Flow-Thru or Sipper Cell with at least 50 mL of deionized water immediately after use.

Interferences

Table 1 Interfering Substances and Suggested Treatments

Interfering Substance	Interference Levels and Treatments
Color	Eliminated by zeroing the instrument with the original sample (follow procedure)
Iron	Interferes at levels greater than 1 mg/L
pH (extreme)	Adjust pH to less than 7. See Section *1.3.1 pH Interference.*
Phosphate (PO_4^{3-})	Interferes at levels greater than 50 mg/L PO_4^{3-}
Sulfides	Interfere at high levels
Turbidity	Eliminated by zeroing the instrument with the original sample (follow procedure)

Sample Collection, Storage and Preservation

Use only plastic containers with tight-fitting closures. Do not use glass containers; they will contaminate the sample with silica. Soak sampling containers with a solution made of one part Molybdate 3 Reagent to 50 parts of high quality deionized water of low silica concentration. Fill completely and let stand for several hours. Rinse thoroughly with low-level silica water, drain and close. Repeat this cleaning periodically.

Allow the sample stream to flow for 1–2 minutes before collection. Do not adjust the flow during the sampling period as this may introduce particulates. Rinse the container well with sample before collecting the portion for analysis. Analyze as soon as possible.

Reagent Preparation

Amino Acid F Reagent Solution is available in either 100-mL bottles or a package of 20 unit-dose ampules. The bottled reagent is stable for up to one year if the bottle is kept closed when not in use. The ampuled reagent is sealed under argon and is more stable with a shelf life greater than 1 year. Reduced sensitivity at high concentrations (>1000 µg/L) indicates reagent instability. Check the bottled reagent on a routine basis by performing an analysis on a 1-mg/L Silica Standard Solution (Cat. No. 1106-49). If the concentration is less than 950 µg/L, use a fresh bottle of Amino Acid F Reagent Solution.

Prepare larger or smaller volumes of Amino Acid F Reagent by dissolving Amino Acid F Reagent Powder in Amino Acid F Reagent Solvent at a ratio of 11 grams per 100 mL. These reagents are available as the Amino Acid F Reagent Package listed under *OPTIONAL EQUIPMENT AND SUPPLIES*. This prepared solution has limited stability; test routinely with the 1-mg/L Silica Standard Solution as above.

If running a large number of samples, the variable volume dispensers are convenient (see *OPTIONAL EQUIPMENT AND SUPPLIES*). The dispensers are made of fluoropolymer plastic. Do not use a dispenser with glass bottles or glass parts; they will contaminate the reagent with silica.

Labware

All containers used in this test must be cleaned thoroughly 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 caps work well.

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

Cleaning the Flow-Thru or Sipper Cell

The Flow-Thru or Sipper Cell may accumulate a buildup of colored products, especially if the reacted solutions are allowed to stand in the cell for long periods after measurement. Remove the color by rinsing with a 1:5 dilution of ammonium hydroxide, followed by several deionized water rinses. Cover the Flow-Thru Cell when it is not in use.

Accuracy Check

Standard Additions Method

a. Leave the unspiked sample in the sample compartment. Verify that the units displayed are in µg/L. Select standard additions mode by pressing the soft keys under ***OPTIONS, (MORE)*** and then ***STD ADD***.

b. Press **ENTER** to accept the default sample volume (mL), 50.0.

c. Use either a 1-mg/L or 10-mg/L (1000 or 10,0000 µg/L) Silica Standard Solution. When prompted for the standard concentration, enter the silica concentration that will be used (either 1000- or 10,000-µg/L). Press **ENTER**.

d. Press the soft key under ***ENTRY DONE***.

e. Use the TenSette Pipet to add 0.1 mL, 0.2 mL and 0.3 mL of standard, respectively, to three 50-mL samples and mix each thoroughly.

f. Analyze each standard addition sample as described above. Accept the standard additions reading by pressing the soft key under ***READ*** each time. Each addition should reflect approximately 100% recovery.

g. After completing the sequence, the display will show the extrapolated concentration value and the “best-fit” line through the standard additions data points, accounting for matrix interferences.

h. See Section *1.4.1 Standard Additions* for more information.

Method Performance

Precision
Standard: 500 µg/L silica

Program	95% Confidence Limits
3370	498.2–501.8 µg/L silica

For more information on determining precision data and method detection limits, refer to Section *1.5*.

Estimated Detection Limit

Program	EDL
3370	1.0 µg/L silica

For more information on derivation and use of Hach's estimated detection limit, see Section *1.5.2*. To determine a method detection limit (MDL) as defined by the 40 CFR part 136, Appendix B, see Section *1.5.1*.

Sensitivity
Program Number: 3370

Portion of Curve	ΔAbs	ΔConcentration
Entire Range	0.010	11.84 µg/L

See Section *1.5.3 Sensitivity Explained* for more information.

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 one-inch Flow-Thru or Sipper Cell with liquid reagents. The Flow-Thru or Sipper Cell increases the reproducibility of the optics and reduces the instability of the readings that result from moveable sample cells. Liquid reagents ensure reproducible readings and lower blank values by eliminating slight turbidity that may remain when using powdered reagents. In addition, the liquid reagents are directly used with Hach process analyzers for continuous silica measurement.

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. 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.

Safety

Good safety habits and laboratory techniques should be used throughout the procedure. Consult the *Material Safety Data Sheet* for information specific to the reagents used. For additional information, refer to *Section 1*.

Pollution Prevention and Waste Management

For information on pollution prevention and waste management, refer to *Section 1*.

REQUIRED REAGENTS AND STANDARDS

	Cat. No.
ULR Silica Reagent Set (using Amino Acid F solution, 100 tests)	25535-00
Includes: (2) 1995-32, (2) 22542-32, (1) 23864-42	
ULR Silica Reagent Set (using Amino Acid F ampules, 40 tests)	25814-00
Includes: (1) 1995-32, (1) 22542-32, (2) 23864-20	

Description	Quantity Required per test	Unit	Cat. No.
Amino Acid F Reagent Solution	1.0 mL	100 mL	23864-42
or			
Amino Acid F Reagent Solution, 1.2-mL Ampules	1 each	20/pkg	23864-20
Citric Acid Reagent Solution	2 mL	500 mL	22542-49
Molybdate 3 Reagent Solution	2.0 mL	1 L	1995-53

REQUIRED EQUIPMENT AND SUPPLIES

Cylinder, graduated, 50-mL, poly	1	each	1081-41
DR/4000 Flow Cell Module Kit, 1-inch	1	each	48070-04
or			
DR/4000 Flow Cell Module Kit, 1-cm	1	each	48070-05
or			
DR/4000 Sipper Module Kit, 1-inch		each	48090-03
Flask, Erlenmeyer, 250-mL, PMP, w/cap	2	each	20898-46
Pipet, TenSette, 0.1- to 1.0-mL	1	each	19700-01
Pipet Tips, for 19700-01 Pipet	5	50/pkg	21856-96

OPTIONAL REAGENTS AND STANDARDS

Amino Acid F Reagent Package, includes:			23531-03
Amino Acid F Reagent Powder		308 g	23532-55
Amino Acid F Dilution Solvent		2.7 liters	23530-03
Ammonium Hydroxide, ACS		500 mL	106-49
Citric Acid F Reagent Solution		100 mL MDB	22542-32
Molybdate 3 Reagent Solution		2.9 L	1995-03
Molybdate 3 Reagent Solution		3.78 L	1995-17
Molybdate 3 Reagent Solution		100 mL	1995-32
Silica Standard Solution, 1 mg/L SiO_2		500 mL	1106-49
Silica Standard Solution, 10 mg/L SiO_2		500 mL	1403-49

OPTIONAL EQUIPMENT AND SUPPLIES

Beaker, polypropylene, 100-mL		each	1080-42
Bottle, 1000-mL, for use w/ variable volume dispenser		6/pkg	7137-54
Dispenser, variable volume, 1.0- to 5.0-mL		each	23121-37
Dropper, 0.5 & 1.0 mL marks, plastic		10/pkg	21247-10
Flask, Erlenmeyer, 250-mL, PMP w/ cap		4/pkg	20898-76
Pipet Tips, for 19700-01 TenSette Pipet		1000/pkg	21856-28
PourRite Ampul Breaker		each	24846-00
Standard Methods for the Examination of Water and Wastewater, 18th edition		each	22708-00
Thermometer, –10 to 110 °C		each	1877-01

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