Heteropoly Blue Method*

1. Enter the stored program number for low range silica (SiO₂).
   Press: PRGM
   The display will show: PRGM ?

2. Press: 90 ENTER
   The display will show mg/L, SiO₂ and the ZERO icon.

3. Fill two sample cells to the 10-mL line with sample.

4. Add 15 drops of Molybdate 3 Reagent to each sample cell. Swirl to mix.
   Note: For greatest accuracy, hold dropping bottle vertical.

5. Press:
   TIMER ENTER
   A 4-minute reaction period will begin.
   Note: Reaction 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.

6. After the timer beeps, add the contents of one Citric Acid Reagent Powder Pillow to each sample cell. Swirl to mix.

7. The display will show:
   1:00 TIMER 2
   Press: ENTER
   A 1-minute reaction period will begin. Phosphate interference is eliminated during this period.
   Note: The time given is for samples at 20 °C (68 °F). If the sample temperature is 10 °C (50 °F), wait two minutes. If the sample is 30 °C (86 °F), wait 30 seconds.

8. After the timer beeps, add the contents of one Amino Acid F Reagent Powder Pillow to one of the sample cells (the prepared sample). Invert to mix.
   Note: The sample cell without the Amino Acid F Reagent is the blank.

* Adapted from Standard Methods for the Examination of Water and Wastewater.
9. The display will show:
   **2:00 TIMER 3**

   Press: **ENTER**

   A 2-minute reaction period will begin.

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

10. After the timer beeps, place the blank (solution without Amino Acid F Reagent) into the cell holder. Tightly cover the sample cell with the instrument cap.

11. Press: **ZERO**

   The cursor will move to the right, then the display will show:
   **0.00 mg/L SiO2**

12. Place the sample into the cell holder. Tightly cover the sample cell with the instrument cap.

13. Press: **READ**

   The cursor will move to the right, then the result in mg/L SiO2 will be displayed.

   *Note: Use of the Standard Adjust feature with each new lot of reagent is highly recommended. See Accuracy Check.*
Sampling and Storage

Collect samples in clean plastic bottles. Analyze samples as soon as possible after collection. If prompt analysis is not possible, store samples for up to 28 days by cooling to 4 °C (39 °F) or below. Warm samples to room temperature before analysis.

Accuracy Check

Standard Additions Method

a) Open a Silica Standard Solution Bottle, 25 mg/L SiO₂.

b) Using the TenSette Pipet, add 0.1, 0.2, and 0.3 mL of standard to three 10-mL samples. Mix thoroughly.

c) Analyze each sample as described above. The silica concentration should increase 0.25 mg/L for each 0.1 mL of standard added.

d) If these increases do not occur, see Standard Additions in Section 1 for more information.

Standard Adjust

To adjust the calibration curve using the reading obtained with the 1.00-mg/L Standard Solution (see Optional Reagents), press the SETUP key and scroll (using the arrow keys) to the STD setup option. Press ENTER to activate the standard adjust option. Then enter 1.00 to edit the standard concentration to match that of the standard used. Press ENTER to complete the adjustment. See Section 1, Standard Curve Adjustment for more information.

Method Performance

Precision

In a single laboratory, using standard solutions of 1.00 mg/L silica and two representative lots of reagent and a instrument, a single operator obtained a standard deviation of ±0.025 mg/L silica.

Estimated Detection Limit (EDL)

The estimated detection limit for program 90 is 0.020 mg/L SiO₂. For more information on the estimated detection limit, see Section 1. If testing for very low levels of silica, use the ultra-low range silica method on the Hach DR/2010 or DR/4000 Spectrophotometers.
Interferences

<table>
<thead>
<tr>
<th>Interfering Substance</th>
<th>Interference Levels and Treatments</th>
</tr>
</thead>
<tbody>
<tr>
<td>Color</td>
<td>Eliminated by zeroing the instrument with the original sample.</td>
</tr>
<tr>
<td>Phosphate</td>
<td>Phosphate does not interfere at levels less than 50 mg/L PO₄. At 60 mg/L PO₄, an interference of -2% occurs. At 75 mg/L PO₄ the interference is -11%.</td>
</tr>
<tr>
<td>Iron</td>
<td>Large amounts of iron interfere.</td>
</tr>
<tr>
<td>Slow reacting forms of silica</td>
<td>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 adding citric acid) may help in lieu of the bicarbonate pretreatment.</td>
</tr>
<tr>
<td>Sulfides</td>
<td>Interfere at all levels</td>
</tr>
<tr>
<td>Turbidity</td>
<td>Eliminated by zeroing the instrument with the original sample.</td>
</tr>
</tbody>
</table>

Reagent Preparation

To prepare Amino Acid F Reagent Solution, dissolve 11.4 grams of Amino Acid F Reagent Powder in 100 mL of 1.0 N Sodium Hydroxide Solution. The solution is stable for at least one month if stored in a plastic bottle.

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. Acid reduces the yellow silicomolybdic acid to an intense blue color, which is proportional to the silica concentration.
### REQUIRED REAGENTS

<table>
<thead>
<tr>
<th>Description</th>
<th>Cat. No.</th>
<th>Quantity Required</th>
<th>Units</th>
</tr>
</thead>
<tbody>
<tr>
<td>Low Range Silica Reagent Set, 10 mL sample (100 tests)</td>
<td>24593-00</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Includes: (1) 22540-69, (1) 21062-69 (2) 1995-26</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Amino Acid F Reagent Powder Pillows</td>
<td>22540-69</td>
<td>1 pillow</td>
<td>100/pkg</td>
</tr>
<tr>
<td>Citric Acid Powder Pillows</td>
<td>21062-69</td>
<td>2 pillows</td>
<td>100/pkg</td>
</tr>
<tr>
<td>Molybdate 3 Reagent</td>
<td>1995-26</td>
<td>28 drops</td>
<td>50 mL SCDB</td>
</tr>
</tbody>
</table>

### REQUIRED APPARATUS

<table>
<thead>
<tr>
<th>Description</th>
<th>Cat. No.</th>
<th>Quantity Required</th>
<th>Units</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample Cell, 10-20-25 mL, w/ cap</td>
<td>24019-06</td>
<td>2</td>
<td>6/pkg</td>
</tr>
</tbody>
</table>

### OPTIONAL REAGENTS

<table>
<thead>
<tr>
<th>Description</th>
<th>Cat. No.</th>
<th>Quantity Required</th>
<th>Units</th>
</tr>
</thead>
<tbody>
<tr>
<td>Silica Standard Solution, 1.00 mg/L SiO₂</td>
<td>1106-49</td>
<td>500 mL</td>
<td></td>
</tr>
<tr>
<td>Silica Standard Solution, 25 mg/L SiO₂</td>
<td>21225-31</td>
<td>236 mL</td>
<td></td>
</tr>
<tr>
<td>Sodium Bicarbonate, ACS</td>
<td>776-01</td>
<td>454 g</td>
<td></td>
</tr>
<tr>
<td>Sodium Hydroxide Standard Solution, 1.000 N</td>
<td>1045-53</td>
<td>900 mL</td>
<td></td>
</tr>
<tr>
<td>Sulfuric Acid Standard Solution, 1.0 N</td>
<td>1270-53</td>
<td>1000 mL</td>
<td></td>
</tr>
</tbody>
</table>

### OPTIONAL APPARATUS

<table>
<thead>
<tr>
<th>Description</th>
<th>Cat. No.</th>
<th>Quantity Required</th>
<th>Units</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bottle, 118 mL, polyethylene, oblong</td>
<td>23184-06</td>
<td>6/pkg</td>
<td></td>
</tr>
<tr>
<td>Dropper, 0.5- &amp; 1.0-mL marks</td>
<td>23185-06</td>
<td>6/pkg</td>
<td></td>
</tr>
<tr>
<td>Pipet, serological, 2 mL, poly</td>
<td>2106-36</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Pipet, TenSette, 0.1 to 1.0 mL</td>
<td>19700-01</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Pipet Tips, for 19700-01 Pipet</td>
<td>21856-96</td>
<td>50/pkg</td>
<td></td>
</tr>
<tr>
<td>Pipet Tips, for 19700-01 Pipet</td>
<td>21856-28</td>
<td>1000/pkg</td>
<td></td>
</tr>
<tr>
<td>Standard Methods for the Examination of Water and Wastewater</td>
<td>22708-00</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Thermometer, - 20 to 110 °C, Non-Mercury</td>
<td>26357-02</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

For Technical Assistance, Price and Ordering
In the U.S.A.—Call 800-227-4224
Outside the U.S.A.—Contact the Hach office or distributor serving you.