



A Honeywell Company

## **Trace Silicon in Petroleum Liquids by ICP-MS**

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### **UOP Method 1006-14**

#### **Scope**

This method is for the determination of silicon (Si) in petroleum liquids using an Inductively Coupled Plasma - Mass Spectrometer (ICP-MS). Silicon is typically present in petroleum liquids as soluble organic silicones, but the method will also detect other forms of silicon such as the generally insoluble inorganic silicon salts and silica. The method is primarily used to analyze reformer charge stocks for silicones, but may also be used for refinery charge stocks as heavy as vacuum gas oil (VGO). The lower limit of quantitation is less than 0.1 mg/kg (mass-ppm) as silicon. Less sensitive tests for trace silicon are UOP Method 796, "Silicon in Petroleum Liquids by ICP-OES," (to 1 mg/kg) and ASTM Method D7757, "Silicon in Gasoline and Related Products by Monochromatic Wavelength Dispersive X-ray Fluorescence Spectrometry," (to 3 mg/kg).

#### **References**

ASTM Specification D1193, "Reagent Water," [www.astm.org](http://www.astm.org)

ASTM Practice D7455, "Sample Preparation of Petroleum and Lubricant Products for Elemental Analysis," [www.astm.org](http://www.astm.org)

ASTM Method D7757, "Silicon in Gasoline and Related Products by Monochromatic Wavelength Dispersive X-ray Fluorescence Spectrometry," [www.astm.org](http://www.astm.org)

ASTM Practice D7876, "Sample Decomposition Using Microwave Heating (With or Without Prior Ashing) for Atomic Spectroscopic Elemental Determination in Petroleum Products and Lubricants," [www.astm.org](http://www.astm.org)

UOP Method 796, "Silicon in Petroleum Liquids by ICP-OES," [www.astm.org](http://www.astm.org)

UOP Method 999, "Precision Statements in UOP Methods," [www.astm.org](http://www.astm.org)

#### **Outline of Method**

The sample is treated initially with water and nitric acid to reduce the volatility of silicon, followed by hydrofluoric acid, after which the samples are digested. The digestate is quantitatively transferred to an autosampler tube using purified water. Indium is added as a monitoring standard and the mixture is diluted to the desired volume. The concentration of silicon in the resulting solution is determined by ICP-MS.

|                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                            |
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| <p>IT IS THE USER'S RESPONSIBILITY TO ESTABLISH APPROPRIATE PRECAUTIONARY PRACTICES AND TO DETERMINE THE APPLICABILITY OF REGULATORY LIMITATIONS PRIOR TO USE. EFFECTIVE HEALTH AND SAFETY PRACTICES ARE TO BE FOLLOWED WHEN UTILIZING THIS PROCEDURE. FAILURE TO UTILIZE THIS PROCEDURE IN THE MANNER PRESCRIBED HEREIN CAN BE HAZARDOUS. SAFETY DATA SHEETS (SDS) OR EXPERIMENTAL SAFETY DATA SHEETS (ESDS) FOR ALL OF THE MATERIALS USED IN THIS PROCEDURE SHOULD BE REVIEWED FOR SELECTION OF THE APPROPRIATE PERSONAL PROTECTION EQUIPMENT (PPE).</p> |
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## Apparatus

References to catalog numbers and suppliers are included as a convenience to the method user. Other suppliers may be used.

*Balance*, readable to 0.0001 g

*Beaker*, PTFE, 600-mL, Fisher Scientific, Cat. No. 02-586-1K

*Bottles*, polypropylene, narrow mouth, screw cap, 125-mL, VWR, Cat. No. 16067-066

*Bottle*, wash, VWR, Cat. No. 2402-0500, for deionized water

*Cover*, PTFE, Milestone, Cat. No. MCL0435, with o-ring, Cat. No. OR0138, used to cover the stainless steel vessel in the microwave oven

*Cylinders*, graduated, Class B, 5-, 10-, 25-, and 100-mL, VWR, Cat. Nos. 14201-610, -462, -364, and -718, respectively

*Flasks*, volumetric, polypropylene, Class B, 25-, 50-, 100-, and 1000-mL, VWR, Cat. Nos. 83008-964, -974, -984, and 83009-008, respectively; several of each required

*Fume hood*

*Hot plate*, VWR, Cat. No. 11301-006

*ICP-MS*, PerkinElmer, DRC-e, equipped with an AS10 autosampler

*Microwave oven*, 15-vessel capacity, with closed digestion vessels, quartz tubes and lids, CryoLab chiller, and computer, Milestone, UltraWAVE

*Pipet*, automatic, 10-100- $\mu$ L, VWR, Cat. No. 89079-968; calibrated and/or certified according to manufacturer's recommendations

*Pipet*, automatic, 100-1000- $\mu$ L, VWR, Cat. No. 89079-974; calibrated and/or certified according to manufacturer's recommendations

*Pipet*, electronic, 1–10-mL, EDP 3-Plus, Rainin, Cat. No. SE3-10mL; calibrated and/or certified according to manufacturer's recommendations

*Regulator*, argon or nitrogen, two-stage, high purity, delivery pressure range 30-700 kPa (4-100 psi), Matheson Tri-Gas, Model 3122-580

*Timer*, capable of timing two hours, VWR, Cat. No. 62344-440

*Watch glass*, PTFE, 125-mm, Fisher Scientific, Cat. No. 02-617-1F

## Reagents and Materials

References to catalog numbers and suppliers are included as a convenience to the method user. Other suppliers may be used. Unless otherwise specified, references to water mean deionized water.

*Argon*, 99.995% minimum purity

*Cleaning compound*, Alconox detergent, VWR, Cat. No. 21835-032

*Dry ice*

*Gloves*, neoprene/natural rubber, VWR, Cat. No. 32917-212 (size 10)

*Gloves*, Viton, VWR, Cat. No. 89236-768 (size 10)

*Hydrofluoric acid*, concentrated, trace metals grade, VWR, Cat. No. EM-HX0628-7

*Hydrogen peroxide*, 29-32%, VWR, Cat. No. VW3690-5

*Indium metal solution*, aqueous, 1000- $\mu\text{g/mL}$ , SPEX CertiPrep, Cat. No. PLIN2-2X, for use as a monitoring solution

*Indium metal stock solution*, aqueous, 100- $\mu\text{g/mL}$ . To prepare 50 mL, pipet 5 mL of the 1000- $\mu\text{g/mL}$  indium metal solution into a 50-mL volumetric flask. Fill to the mark with deionized water. Cap and invert several times to mix. The solution should remain stable for two months.

*Indium monitoring standard solution*, 10- $\mu\text{g/mL}$ . To prepare 100 mL, pipet 10.0 mL of the 100- $\mu\text{g/mL}$  indium metal stock solution into a 100-mL volumetric flask. Fill to the mark with deionized water. Cap and invert several times to mix. Store in a 125-mL polypropylene bottle. The solution should remain stable for two months.

*Lubricant*, dry spray, PTFE, W. W. Grainger, Cat. No. 4JB33, or local supply

*Nitric acid*, concentrated, trace metals grade, VWR, Cat. No. EM-NX0408-2

*Nitric acid*, 2%. Pipet 20 mL of concentrated nitric acid into a 1000-mL volumetric flask containing ~800 mL of deionized water; fill to mark with deionized water. Cap and invert several times to mix.

*Nitrogen*, 99.995% minimum purity, optional, see *Microwave Digestion*, Step 6

*O-rings*, replacement, for microwave oven, Milestone, Cat. No. OR0159

*O-ring*, top of chamber, replacement, Milestone, Cat. No. OR0170

*Pipet*, glass, dropping, VWR, Cat. No. 52950-206

*Pipet tips*, 10-mL, Rainin, Cat. No. RC-10 mL

*Pipet tips*, 1-200- $\mu\text{L}$ , VWR, Cat. No. 53508-810

*Pipet tips*, 100-1250- $\mu\text{L}$ , VWR, Cat. No. 53508-922

*Silicon standard solution*, 1000- $\mu\text{g/mL}$ , SPEX CertiPrep, Cat. Nos. PLSi9-2X.

*Sulfuric acid*, concentrated, trace metals grade, VWR, Cat. No. EM-SX1248-6

*Towels*, paper, disposable, local supply

*Tubes*, autosampler, 50 mL, with caps, PerkinElmer, Cat. No. BO193234

*Vials*, PTFE, 15-mL, Milestone, Cat. No. HB00086, with caps, Cat. No. DD00134

*Water*, deionized, ASTM D1193 Type III reagent grade water, unless otherwise specified

*Water*, hot, tap

*Wipers*, Kimwipes Ex-L, VWR, Cat. No. 21905-026

The following materials are not required to perform the analyses described herein and are not cited in the *Procedure* portion of the method. However, they may be useful for reference purposes, for verification of instrument performance, or as an aid in instrument maintenance.

*Kerosene solvent*, metals free, VWR, Cat. No. AA36447-K7 or SPEX, Cat. No. KER-BLK-G

*Reference material*, SPEX CertiPrep 23 Multi-Element Standard, 100- $\mu\text{g/g}$  (mg/kg) each, SPEX CertiPrep, Cat. No. S23 1004. Dilute by weight in kerosene to 10  $\mu\text{g/g}$ . Alternatively, a reference standard for silicon may be prepared using polydimethylsiloxane as described in the *Appendix*.

## Procedure

The analyst is expected to be familiar with general laboratory practices, the technique of ICP-MS, and the equipment being used. Additional information regarding sample preparation may be found in ASTM Practice D7455, "Sample Preparation of Petroleum and Lubricant Products for Elemental Analysis." Additional information regarding the use of microwave digestion for sample decomposition may be found in ASTM Practice D7876, "Sample Decomposition Using Microwave Heating (With or Without Prior Ashing) for Atomic Spectroscopic Elemental Determination in Petroleum Products and Lubricants." Dispose of all supplies and samples in an environmentally safe manner according to applicable regulations.

**CAUTION:** All subsequent steps involving additions of reagents and heating of samples must be performed in a properly operating fume hood, and appropriate personal protective equipment must be worn. See the safety data sheet (SDS) and any local requirements for each material used.

### Preparation of Standards

Standards are required for analytes to be determined. Stock standard solution is prepared first, from which lower level, matrix matched, calibration standard solutions are then prepared.

#### ***Preparation of Silicon Stock Standard Solution***

Prepare stock standard solution of 10- $\mu$ g/mL concentration of silicon as follows:

1. Pipet 1.0 mL of the 1000- $\mu$ g/mL silicon standard solution into a 100-mL polypropylene volumetric flask.
2. Dilute to the mark with deionized water. Label as Stock Standard A. The solution should remain stable for two months.

### Cleaning of PTFE Vials and Caps

Clean the PTFE vials and caps as follows:

1. Place PTFE vials and caps in a 600-mL beaker, cover with 2% nitric acid, and place a watch glass on top of the beaker.
2. Place the beaker on a hot plate. Set the hot plate to 200 °C and bring to a boil. Adjust the temperature of the hot plate to maintain a slow boil. Boil for two hours.
3. Remove the beaker from the hot plate and allow it to cool.
4. Decant the acid and dispose of it in an environmentally safe manner according to applicable regulations.
5. Rinse the PTFE vials, caps, beaker, and watch glass with deionized water, and air dry.
6. Store the vials and caps in the beaker, covered with the watch glass until needed.

### Microwave Digestion

The blank and the reference are digested in the same manner as the samples.

1. Weigh 0.25-0.30 g of each sample or reference material to the nearest 0.0001 g into the previously cleaned and air dried vials. Using a pipet or graduated cylinder, add 1 mL of water. Using a graduated cylinder, add 1.5 to 2 mL of concentrated nitric acid (HNO<sub>3</sub>) and 0.3 mL of hydrofluoric acid (HF) to the PTFE vials.

2. Prepare an additional three vials as digestion acid blanks by adding 1 mL water, 1.5 to 2 mL concentrated nitric acid (HNO<sub>3</sub>) and 0.3 mL of hydrofluoric acid (HF) to each pre-cleaned, air dried, PTFE vials.
  - One vial is used for preparing the blank, two are used for standard preparation. This is to ensure matrix matching with the samples.
3. Close the digestion vessels and place them into the sample holder.
4. Set up the microwave as described below. Refer to the UltraWAVE Operator Manual for details. Add the basic load/absorption media as follows: Using graduated cylinders, add 130 mL of water, 5 mL of 30% hydrogen peroxide, and 2 mL of sulfuric acid into the PTFE vessel that goes into the stainless steel pressure vessel. This acts as absorption media.
  - Ensure that all o-rings are properly cleaned and greased.
5. Check the water level in the CryoLab chiller.
6. Turn on the gas (nitrogen or argon), CryoLab chiller, computer, and UltraWAVE.
7. Program the system using the settings shown in Table 1.
  - Other conditions may be used provided they produce equivalent results.

**Table 1**  
**Digestion Conditions**

| <b><u>Nr.</u></b> | <b><u>t(min)</u></b> | <b><u>E (w)</u></b> | <b><u>T1 °C</u></b> | <b><u>T2 °C</u></b> | <b><u>P</u></b> |
|-------------------|----------------------|---------------------|---------------------|---------------------|-----------------|
| <b>1</b>          | <b>00:30:00</b>      | <b>1500</b>         | <b>230</b>          | <b>60</b>           | <b>150</b>      |
| <b>2</b>          | <b>00:35:00</b>      | <b>1500</b>         | <b>230</b>          | <b>60</b>           | <b>150</b>      |

Nr. 1 is for reaching to the prescribed condition,  
and Nr. 2 is holding at that condition.

8. Load the samples into the UltraWAVE.
9. Press the down arrow on the touch screen monitor to lower the sample holder to level 1, check that everything is OK, then press the down arrow again to lower it to level 2. At this point the sample holder is immersed into the absorption media.
10. Manually close the clamps.
11. Check the method and parameters, and then start the run.
  - At the end of the digestion process, after the materials have cooled to 30 °C or lower on the readout, open the UltraWAVE clamps, press the up arrow on the monitor to lift the sample holder, wait for the fumes to exhaust through the vent, and press the up arrow again to lift the sample holder fully. Cover the stainless steel vessel with the PTFE cover to protect it from possible dripping of the acid condensate.
12. Carefully remove the sample holder with the PTFE vials and place it on disposable paper towels.
13. Lift each vial from the sample holder and wipe the bottom on a paper towel. Quantitatively transfer the contents of each vial into separate 50-mL autosampler tubes using deionized water as the diluent.
14. Add 80 µL of the 10-µg/mL indium monitoring standard solution to each tube containing samples.
15. Fill each tube to the 40 mL mark with water. Cap and invert several times to thoroughly mix the contents.
16. Immediately after use, to avoid corrosion of the UltraWAVE's stainless steel pressure vessel, dispose of the absorption media, and rinse with water. Carefully clean and wipe dry other

exposed parts. Spray lubricant on a wiper and wipe as described in the UltraWAVE Operator Manual. Also clean the PTFE container and PTFE vials.

### Preparation of Blank and Calibration Standard Solutions

Prepare calibration standards and a blank as follows:

1. Into one of the three 50-mL autosampler tubes containing the digestate of the acid blank, pipet 200  $\mu\text{L}$  of 10.0- $\mu\text{g}/\text{mL}$  silicon standard stock solution. Label as calibration standard solution, 50-ng/mL
2. Into a second tube containing the digestate of the acid blank, pipet 400  $\mu\text{L}$  of the 10.0- $\mu\text{g}/\text{mL}$  silicon standard stock solution. Label as calibration standard solution, 100-ng/mL.
  - The third flask containing the digestate of the acid blank will be used for the blank preparation. (Initially dilute to 25-mL volume and transfer 3 mL of the diluted digestate to another 50-mL tube and 1 drop of HF).
  - The blank can be also prepared by pipetting 0.1 mL of nitric acid and 0.1 mL of hydrofluoric acid into a 50 mL tube containing ~25 ml of water.
3. Add 80  $\mu\text{L}$  of 10- $\mu\text{g}/\text{mL}$  indium internal standard solution to each tube.
4. Dilute to the 40-mL mark with water and mix the contents.

### ICP-MS Determination of Analytes

The ICP-MS is calibrated with prepared standards using the autosampler. Three calibration points are obtained by analyzing a blank, 50- and 100-ng/mL silicon standards. Samples are then analyzed and the 50- and 100-ng/mL standards are reanalyzed as samples at the end of the run.

1. Set up the instrument according to the manufacturer's recommended conditions, see Table 2.

**Table 2**  
**ICP-MS Parameters**

|                                                   |                                                                                 |
|---------------------------------------------------|---------------------------------------------------------------------------------|
| <b>ICP/MS instrument:</b>                         | <b>Perkin Elmer DRC-e (DRC Mode)</b>                                            |
| <b>Analytes:</b>                                  | <b>Si is measured as <math>^{44}\text{SiO}</math></b>                           |
| <b>Internal standard:</b>                         | <b><math>^{115}\text{In}</math> ( internal standard or monitoring standard)</b> |
| <b>RF power</b>                                   | <b>1500 W</b>                                                                   |
| <b>Plasma gas flow:</b>                           | <b>15 L/min</b>                                                                 |
| <b>Auxiliary gas Flow rate</b>                    | <b>1.2 L/min</b>                                                                |
| <b>Nebulizer</b>                                  | <b>Nebulizer Cross Flow II</b>                                                  |
| <b>Nebulizer gas flow rate</b>                    | <b>~0.85 L/min</b>                                                              |
| <b>DRC(<math>\text{O}_2</math>) gas flow rate</b> | <b>~0.8 L/min</b>                                                               |
| <b>Spray chamber:</b>                             | <b>Ryton<sup>TM</sup> Scott-type</b>                                            |
| <b>Ion sampling and skimmer cone</b>              | <b>nickel cone</b>                                                              |
| <b>Sampling distance</b>                          | <b>10 mm from load coil</b>                                                     |
| <b>Sample wash</b>                                |                                                                                 |
| <b>Peristaltic pump speed</b>                     | <b>-20 rpm</b>                                                                  |
| <b>Uptake</b>                                     | <b>30 seconds</b>                                                               |
| <b>Wash</b>                                       | <b>60 seconds</b>                                                               |
| <b>Resolution:</b>                                | <b>standard</b>                                                                 |
| <b>Detector mode</b>                              | <b>dual</b>                                                                     |

|                               |              |
|-------------------------------|--------------|
| <b>Dwell time</b>             | <b>50 ms</b> |
| <b>Sweeps per reading</b>     | <b>20</b>    |
| <b>Readings per replicate</b> | <b>1</b>     |
| <b>Replicates</b>             | <b>3</b>     |

2. Transfer aliquots of the digested samples, blank, and calibration standards into autosampler vials.
3. Enter the locations of all the solutions to be analyzed into the instrument data system.
4. Run the blank, calibration standards, and the samples. At the end of the series, analyze the blank and calibration standards as samples.
  - If the standards run at the end of the series do not repeat within the repeatability allowable difference shown under *Precision*, check the apparatus and technique, and after fixing any problems found, rerun the samples.
5. Dispose of the remaining solutions in an environmentally safe manner according to applicable regulations.

### ***Viscous Liquids or Meltable Solids***

If the sample is a solid or a very viscous liquid at room temperature and cannot be readily dissolved in the solvent:

1. Loosen the sample bottle cap and heat the sample in an oven at 100 °C for approximately 30 minutes. Also place a glass dropper pipet in the oven.
  - Heating the sample above 100 °C is not recommended as it may result in the loss of volatile silicone compounds.
2. Place a precleaned PTFE vial and cap (removed from vial) on the balance and obtain a tare weight.
3. Using thermal gloves or other protection, remove the pipet and sample bottle from the oven, tighten the cap on the sample bottle and shake to mix.
4. Using the hot pipet, transfer 0.25 to 0.30 g of the hot sample into the tared PTFE vial. Record the weight to the nearest 0.01 g and continue with Step 2 and following under *Microwave Digestion*.

### **Calculations**

All calculations are performed by the instrument data system after the sample mass and dilutions are entered, and sample results are reported directly in mg/kg. If all the data is not entered into the instrument data system, the results will be reported in ng/mL. Use Equation 1 to calculate the final sample concentration.

- If any analyte's concentration is more than 20% above that of the highest standard, dilute the sample further and reanalyze.

$$\text{Trace metal concentration, mg/kg} = \frac{40 C}{W} \quad (1)$$

where:

**C** = analyte in the analyzed solution as reported by the instrument, ng/mL

**W** = sample weight, g

**40** = diluted volume of the digest, mL

Report results in mg/kg to two significant figures.

## Precision

Precision statements were determined using UOP Method 999, "Precision Statements in UOP Methods," from precision data obtained using an autosampler.

### Repeatability and Site Precision

A nested design was carried out for determining metals in organics by two analysts, with each analyst performing analyses on four separate days, performing two analyses each day for a total of 16 analyses. Using a stepwise analysis of variance procedure, the within-day and within-lab estimated standard deviations (esd) were calculated at the concentration means listed in Table 3. Two analyses performed in one laboratory by the same analyst on the same day should not differ by more than the repeatability allowable differences shown in Table 3 with 95% confidence. Two analyses performed in one laboratory by different analysts on different days should not differ by more than the site precision allowable differences shown in Table 3 with 95% confidence.

**Table 3**  
**Repeatability and Site Precision, mg/kg**

| <u>Mean</u> | <u>Repeatability</u>       |                                 | <u>Site Precision</u>      |                                 |
|-------------|----------------------------|---------------------------------|----------------------------|---------------------------------|
|             | <u>Within-<br/>Day esd</u> | <u>Allowable<br/>Difference</u> | <u>Within-<br/>Lab esd</u> | <u>Allowable<br/>Difference</u> |
| 5.19        | 0.204                      | 0.63                            | 0.238                      | 0.94                            |

The data in Table 3 represent short-term estimates of the repeatability of the method. When the test is run routinely, use of a control standard and a control chart is recommended to generate an estimate of long-term repeatability.

### Reproducibility

There is insufficient data to calculate the reproducibility of the test at this time.

## Time for Analysis

The elapsed time for one analysis is approximately 2 hours, depending upon sample preparation. The labor requirement for one analysis is 1 hour. In groups of 5 or more samples, the labor requirement for each analysis is 0.5 hour.

## Suggested Suppliers

Fisher Scientific, 300 Industry Dr., Pittsburgh, PA 15275, USA, 1-412-490-8300,  
www.fishersci.com

Matheson Tri-Gas, 150 Allen Rd. #302, Basking Ridge, NJ 07920, USA, 1-908-991-9200,  
www.mathesontrigas.com

Milestone Inc., 25 Controls Dr., Shelton, CT 06484, USA, 1-203-925-4240, www.milestonesci.com

National Institute of Standards and Technology (NIST), Standard Reference Materials Group, 100  
Bureau Dr., Gaithersburg, MD 20899, USA, 1-301-975-6776, www.nist.gov

PerkinElmer Analytical Instruments, 710 Bridgeport Ave., Shelton, CT 06484-4794, USA, 1-203-  
925-4600, www.perkinelmer.com

Rainin Instrument Co., Mack Road, Woburn, MA 01801, USA, 1-800-472-4646, www.rainin.com

SPEX CertiPrep, Inc., 203 Norcross Ave., Metuchen, NJ 08840, USA, 1-732-549-7144,  
www.spexcsp.com

VWR International, 1310 Goshen Parkway, West Chester, PA 19380, USA, 1-610-431-1700,  
[www.vwr.com](http://www.vwr.com)

W.W. Grainger, Inc., 100 Grainger Parkway, Lake Forest, IL 60045-5201, USA, 1-847-535-1000,  
[www.grainger.com](http://www.grainger.com)

## Appendix

### Preparation of Silicon Reference Standards from Polydimethylsiloxane

A silicon reference standard, in addition to the calibration standard, is useful for reference purposes, for verification of instrument performance, and as an aid in instrument maintenance.

#### Apparatus

References to catalog numbers and suppliers are included as a convenience to the method user. Other suppliers may be used. The following items are required in addition to, or as an alternative to, those listed in the body of the method.

*Bath*, ultrasonic, VWR, Cat. No. 98000-328

#### Reagents and Materials

The following items are required in addition to, or as an alternative to, those listed in the body of the method.

*Kerosene solvent*, metals free, VWR, Cat. No. AA36447-K7 or SPEX, Cat. No. KER-BLK-G

*Silicone*, 100% polydimethylsiloxane (37.87% silicon), 200 Fluid, 10 cSt, Dow Corning

#### Procedure

Prepare a nominally 50- $\mu\text{g/g}$  silicon standard from polydimethylsiloxane as follows. If a purchased silicon standard is to be used in place of polydimethylsiloxane, skip this part and proceed with the preparation of the nominally 10- $\mu\text{g/g}$  silicon reference standard as described in the body of the method.

1. Weigh  $2.64 \pm 0.01$  g of 100% polydimethylsiloxane (37.87% silicon) into a clean 125-mL bottle and record the weight to the nearest 0.01 g. Add  $97.36 \pm 0.01$  g of kerosene and record the weight to the nearest 0.01 g. Mix by shaking and place in an ultrasonic bath for approximately 10 minutes. Label as "Stock Silicon Standard A." It will contain approximately 10000- $\mu\text{g/g}$  silicon.
2. Weigh  $0.500 \pm 0.01$  g of Stock Silicon Standard A into a clean 125-mL bottle and record the weight to the nearest 0.01 g. Add  $99.5 \pm 0.01$  g of kerosene and record the weight to the nearest 0.01 g. Mix by shaking, and place in an ultrasonic bath for approximately 10 minutes. Label as "Stock Silicon Standard B." It will contain approximately 50- $\mu\text{g/g}$  silicon.
3. Weigh  $20 \pm 0.01$  g of Stock Silicon Standard B into a clean 125-mL bottle and record the weight to the nearest 0.01 g. Add  $80 \pm 0.01$  g of kerosene and record the weight to the nearest 0.01 g. Mix by shaking and place in an ultrasonic bath for approximately 10 minutes. Label as "Reference Silicon Standard." It will contain approximately 10- $\mu\text{g/g}$  silicon.
4. Weigh 0.25-0.30 g of the Reference Silicon Standard and digest per as described in the body of the method and use as a reference standard.
5. Calculate the concentration of silicon in Stock Silicon Standards A and B using Equations A1 and A2:

$$\text{Concentration of silicon (Si), Stock Silicon Standard A, } \mu\text{g/g} = 378700 \frac{A}{A+B} \quad (\text{A1})$$

where:

**A** = weight of polydimethylsiloxane from Step 1, g

**B** = weight of kerosene from Step 1, g

$$378700 = 37.87 \times 10000$$

where:

**37.87** = concentration of Si in polydimethylsiloxane, mass-%

**10000** = factor to convert mass-% to  $\mu\text{g/g}$

$$\text{Concentration of silicon (Si), Stock Silicon Standard B, } \mu\text{g/g} = F \frac{D}{D + E} \quad (\text{A2})$$

where:

**D** = weight of Stock Silicon Standard A from Step 2, g

**E** = weight of kerosene from Step 2, g

**F** = concentration of Si in Stock Silicon Standard A, from Equation A1,  $\mu\text{g/g}$

### Suggested Supplier

Dow Corning Corporation, Midland, MI 48686, USA, 1-989-496-7875, [www.dowcorning.com](http://www.dowcorning.com)