



# Standard Test Method for Silicon in Engine Coolant Concentrates by Atomic Absorption Spectroscopy<sup>1</sup>

This standard is issued under the fixed designation D 6129; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This test method covers the determination of silicon in the range from 200 to 500 ppm in engine coolant concentrates by atomic absorption. This method is as accurate and precise as photometric methods, while requiring considerably less operator time and avoiding problems with reagent instability.

1.1.1 Coolants with silicon content outside of this range may be analyzed by this method by suitably adjusting the sample size. Care should be taken to ensure that the glycol content of the working standards corresponds to that of the sample solution being analyzed.

1.2 The values stated in SI units are to be regarded as the standard.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

## 2. Referenced Documents

### 2.1 ASTM Standards:

D 1176 Test Method for Sampling and Preparing Aqueous Solutions of Engine Coolants or Antirusts for Testing Purposes<sup>2</sup>

## 3. Summary of Test Method

3.1 The sample is dissolved in water and the silicon content is determined from a standard curve established at the time the sample is run. An atomic absorption spectrometer with a nitrous oxide-acetylene flame is used.

## 4. Apparatus

4.1 *Atomic Absorption Spectrometer*, with nitrous oxide-acetylene flame.

4.2 *Hollow-Cathode Lamp*, for silicon.

## 5. Reagents

5.1 Reagent grade chemicals shall be used in all tests.<sup>3</sup>

5.2 References to water shall be understood to mean distilled water or water of equal quality.

5.3 *Standard Silicon Solution*, 1000 mg/L (see Note 1).

5.4 *Ethylene Glycol* (propylene glycol should be used when analyzing PG-based coolants).

5.5 *Sodium Hydroxide*, 5 % in water (see Note 1). When preparing from solid sodium hydroxide avoid prolonged exposure to the atmosphere. DO NOT STORE IN GLASS.

5.6 Working standards: 0, 20, 40, and 60 mg/L silicon.

5.6.1 Prepare these solutions by pipetting 2.0, 4.0, and 6.0 mL of the 1000 mg/L standard silicon solution into separate 100 mL volumetric flasks. Add 10.0 mL of ethylene glycol and 2.0 mL of 5 % sodium hydroxide solution to each flask and dilute to volume with water. Prepare the zero standard solution by adding 10.0 mL ethylene glycol and 2.0 mL 5 % sodium hydroxide solution to another 100 mL volumetric flask and diluting to volume with water.

NOTE 1—Standard silicon solutions and sodium hydroxide solutions are available from most laboratory supply companies.

## 6. Sampling

6.1 Sample material in accordance with Test Method D 1176.

## 7. Procedure

7.1 To the nearest 0.001 g, weigh 5 g of sample into a 50 mL volumetric flask. Dissolve in and dilute to volume with water.

7.2 Using the nitrous oxide-acetylene flame, optimize the instrument operating conditions for silicon determination at the 251.6 nm spectral line according to the manufacturer's recommendations.

7.3 Obtain the approximate absorbance reading of the sample solution versus the zero standard solution (containing glycol and sodium hydroxide).

7.4 Choose the working standard solutions that will give absorbance readings lower and higher than that of the sample.

<sup>1</sup> This specification is under the jurisdiction of ASTM Committee D15 on Engine Coolants and is the direct responsibility of Subcommittee D15.04 on Chemical Properties.

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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 15.05.

<sup>3</sup> *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC) Rockville, MD.

7.5 Set the instrument to zero absorbance with the zero standard solution.

7.6 Determine and record the absorbance of each working standard solution.

7.7 Determine and record the absorbance of the sample solution.

7.8 Recheck the standards to verify the calibration. If the calibration has changed, repeat 7.5-7.8. When running multiple samples, up to five samples may be run between recalibrations.

7.9 Plot a standard curve using absorbance versus mg/L silicon (see Note 2).

7.10 From the standard curve determine the mg/L of silicon contained in the sample solution.

NOTE 2—If the instrument in use has the capability of internally calibrating and directly displaying the concentration contained in the sample solution, manual construction of a calibration curve is, of course, unnecessary.

## 8. Calculation

8.1 Calculate the silicon content of the sample as follows:

$$\text{silicon, ppm} = \frac{M \times 500\,000}{\text{sample wt (g)} \times 10\,000} \quad (1)$$

where:

$M$  = silicon in the sample solution, mg/L.

8.2 The silicon content may be expressed as specific compounds by use of the following conversion calculations:

$$\text{ppm Na}_2\text{SiO}_3 = \text{ppm Si} \times 4.344 \quad (2)$$

$$\text{ppm Na}_2\text{SiO}_3 \cdot 5\text{H}_2\text{O} = \text{ppm Si} \times 7.553$$

$$\text{ppm SiO}_2 = \text{ppm Si} \times 2.138$$

## 9. Precision and Bias

9.1 *Precision*—The repeatability standard deviation, for a sample containing 230 ppm Si, has been determined to be 3 ppm. The 95 % repeatability limit has been determined to be 8 ppm. The reproducibility standard deviation has been determined to be 8 ppm. The 95 % reproducibility limit has been determined to be 22 ppm.

9.2 *Bias*—No information can be presented on the bias of the procedure in this test method as no material having an accepted reference value is available.

## 10. Keywords

10.1 atomic absorption; silicon; engine coolants; spectroscopy

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