



Designation: D6130 – 11

Standard Test Method for Determination of Silicon and Other Elements in Engine Coolant by Inductively Coupled Plasma-Atomic Emission Spectroscopy¹

This standard is issued under the fixed designation D6130; 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 (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of silicon in engine coolant by inductively coupled plasma-atomic emission spectroscopy (ICP-AES). Silicon can be determined as low as the range of 5 ppm by this test method. Other elements also found in engine coolant can be determined by this method. This test method is applicable to the determination of dissolved or dispersed elements.

1.2 This test method is applicable to both new and used engine coolant.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.4 *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:²

D1193 Specification for Reagent Water

D1176 Practice for Sampling and Preparing Aqueous Solutions of Engine Coolants or Antirusts for Testing Purposes

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

¹ This test method is under the jurisdiction of ASTM Committee D15 on Engine Coolants and Related Fluids and is the direct responsibility of Subcommittee D15.04 on Chemical Properties.

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² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

2.2 US EPA Standards:

Method 6010, Inductively Coupled Plasma Method, SW-846, Test Methods for Evaluating Solid Waste³

Method 200.7, Inductively Coupled Plasma - Atomic Emission Spectrometric Method for Trace Element Analysis of Water And Wastes, EPA-600/4-79-020, revised 1984³

3. Summary of Test Method

3.1 Elements in solution are determined, either sequentially or simultaneously, by ICP-AES. New or used engine coolants are prepared by dilution. Samples and standards are introduced to the nebulizer using a peristaltic pump and the aerosol is injected into an argon-supported inductively coupled plasma. The high temperature of the plasma atomizes the sample and produces atomic emission intensities at wavelengths associated with the desired elements. Emission intensity is proportional to concentration. Elemental determinations are made by comparing standard and sample emission intensities.

4. Significance and Use

4.1 Some engine coolants are formulated with silicon containing additives. This test method provides a means of determining the concentration of dissolved or dispersed elements which give an indication of this additive content in the engine coolant.

5. Interferences

5.1 Interferences may be categorized as follows:

5.1.1 *Spectral*—Light emission from spectral sources other than the element of interest may contribute to apparent net signal intensity. Sources of spectral interference include direct spectral line overlaps, broadened wings of intense spectral lines, ion-atom recombination continuum emission, molecular band emission and stray (scattered) light from the emission of elements at high concentrations. Avoid overlaps by selecting alternate analytical wavelengths.

5.1.2 *Physical*—Physical interferences are effects associated with sample nebulization and transport processes such as viscosity and particulate contamination.

³ Available from U. S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, OH 45268.

5.1.3 *Background*—High background effects from scattered light, etc., can be compensated for by background correction adjacent to the analyte line.

5.1.4 *Chemical*—Chemical interferences are caused by molecular compound formation, ionization effects, and thermochemical effects associated with sample vaporization and atomization in the plasma. Normally these effects are not pronounced and can be minimized by careful selection of operating conditions (incident power, plasma observation position, etc.).

6. Apparatus

6.1 *Spectrometer*—An inductively coupled plasma emission spectrometer of the simultaneous or sequential type including RF generator, torch, nebulizer, spray chamber, recommended peristaltic pump and host computer.

7. Reagents and Materials

7.1 *Purity of Chemicals*—Reagent grade or better chemicals shall be used for preparation of all standards and samples. Other grades may be used provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 *Purity of Water*—References to water shall be understood to mean deionized water.

7.3 *Standard Stock Solutions*—Certified solutions may be purchased or prepared from high purity grade chemicals or metals (See Method 6010, SW-846, Method 200.7). Standards contain 1000 mg/L of the element of interest. Salts should be dried as indicated.

7.4 *Calibration Standards*—Prepare the standards in volumetric flasks using appropriate volumes of each stock solution to cover the expected concentration range of the samples. Elements in multielement standards should be shown to be compatible and stable. Compensate for differences in standard/sample matrix by using an appropriate amount of ethylene glycol and/or an internal standard. Suggested combinations and analytical lines are in **Table 1**. Validate calibration standards. Monitor stability.

8. Sampling

8.1 Collect sample in accordance with Practice **D1176**.

TABLE 1 Analytical Wavelengths for ICP-AES Determination of Elements in Engine Coolant

Element	Wavelength, nm
	Mixed Standard 1
Silicon	251.612, 288.158, 252.851, 252.411
Molybdenum	202.030, 204.598
Boron	249.773
Phosphorus	214.914, 178.29
	Mixed Standard 2
Aluminum	308.215, 394.401, 369.152
Lead	220.353
Zinc	213.856
Iron	259.94, 259.837, 238.204
Copper	324.754, 219.226
Magnesium	279.079, 280.270, 279.553
Calcium	317.933, 393.37, 396.847, 315.887
Sodium	588.995, 589.592

9. Calibration and Standardization

9.1 Set the up instrument according to the manufacturer's instructions. Warm it up at least 20 min.

9.2 Profile and calibrate the instrument according to manufacturer's recommended procedures with the blank and standards, aspirating the standard for at least 30 s to allow the instrument to equilibrate prior to signal integration. Water should be run for an additional 60 s after standards containing boron. Calibration should be validated and stability of standards should be monitored.

10. Sample Preparation

10.1 Dilute the sample with deionized water so the concentration of the element(s) of interest is in the linear detection range of the instrument. Generally a $1/20$ or $1/50$ dilution for used engine coolant and a $1/100$ dilution for engine coolant concentrate are sufficient. Samples may be prepared by weight to volume or by volume to volume. Be certain when preparing dilutions by volume that the entire sample aliquot is transferred. Filter or centrifuge samples that contain particulate.

11. Procedure

11.1 Aspirate the prepared samples into the calibrated instrument using the same conditions established for the calibration procedure. Rinse sufficiently to prevent carryover. Run water an additional 60 s between samples containing boron.

11.2 Run a blank and an instrument check standard (a calibration standard, calibration verification or standard engine coolant) every ten samples or as established to be necessary for the instrument. Analyze a blank and check standard at the end of each run. The concentration shall be within $\pm 5\%$ of the expected value. If the concentration is out of range, correct the problem, recalibrate the instrument and rerun the samples in question.

11.3 Matrix spikes and duplicates may be performed as quality control procedures if sample concentrations are suspect due to contamination, spectral interferences or trace levels of the element of interest.

11.4 Perform the corrections and calculations, including dilution factors, using the instrument host computer.

12. Report

12.1 Samples prepared by weight to volume dilution may be reported in ppm by weight or % by weight depending on the concentration of the element of interest. Samples prepared by volume may be reported as g/L, mg/L, $\mu\text{g/mL}$, etc. These units may be converted to ppm weight or % by weight using the density of the sample:

$$\text{concentration (ppm by wt)} = \frac{\text{concentration } (\mu\text{g/mL})}{\text{density (g/mL)}} \quad (1)$$

13. Precision and Bias

13.1 The precision of this test method is based on an interlaboratory study conducted in 2009. A total of nine laboratories participated in this study, testing samples of six

different coolants for their metals content. Every test result represents an individual determination, and all participants were asked to report four replicate test results for each metal/coolant combination. Practice E691 was followed for the design and analysis of the data; the details are given in RR:D15-1030.

13.1.1 *Repeatability Limit, r*—Two test results obtained within one laboratory shall be judged not equivalent if they differ by more than the *r* value for that material; *r* is the interval representing the critical difference between two test results for the same material, obtained by the same operator using the same equipment on the same day in the same laboratory.

13.1.1.1 Repeatability limits are listed in Tables 2-13.

13.1.2 *Reproducibility limit, R*—Two test results shall be judged not equivalent if they differ by more than the *R* value for that material; *R* is the interval representing the critical difference between two test results for the same material, obtained by different operators using different equipment in different laboratories.

13.1.2.1 Reproducibility limits are listed in Tables 2-13.

13.1.3 The above terms (*repeatability limit* and *reproducibility limit*) are used as specified in Practice E177.

13.1.4 Any judgment in accordance with statements 13.1.1 and 13.1.2 would have an approximate 95% probability of being correct.

13.2 *Bias*—At the time of the study, there was no accepted reference material suitable for determining the bias for this test method, therefore no statement on bias is being made.

13.3 The precision statement was determined through statistical examination of 1,872 test results, submitted by nine laboratories, on twelve metals, in six coolants.

13.3.1 The six coolant types were described as follows:

- Coolant A
- Coolant B
- Coolant C
- Coolant D
- Coolant E
- Coolant F

13.4 To judge the equivalency of two test results, it is recommended to choose the coolant material that is closest in characteristics to the test material.

14. Keywords

14.1 engine coolant; inductively coupled plasma-atomic emission spectroscopy; silicon

TABLE 2 Aluminum (µg/mL)

Material	Average, \bar{x}	Repeatability Standard Deviation, S_r	Reproducibility Standard Deviation, S_R	Repeatability Limit, <i>r</i>	Reproducibility Limit, <i>R</i>
Sample A	6.4	8.0	9.7	22.4	27.2
Sample B	2.9	3.0	3.2	8.3	9.0
Sample C	0.5	0.7	0.7	2.0	2.1
Sample D	2.9	4.4	4.9	12.4	13.9
Sample E	5.2	3.7	4.8	10.2	13.5
Sample F	5.6	6.1	7.0	17.2	19.7

^aThe average of the laboratories' calculated averages.