



Designation: **D 6130 – 97a (Reapproved 2003)**

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 D 6130; 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 the 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:*

D 1193 Specification for Reagent Water²

¹ This test method is under the jurisdiction of ASTM Committee D15 on Engine Coolants and is the direct responsibility of Subcommittee D15.04 on Chemical Properties. Current edition approved Oct. 10, 1997. Published July 1998. Originally published as D 6130-97. Last previous edition D 6130-97.

D 1176 Standard Practice for Sampling and Preparing Aqueous Solutions of Engine Coolants or Antirusts for Testing Purposes³
E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method⁴

2.2 US EPA Standards:

Method 6010, Inductively Coupled Plasma Method, SW-846, Test Methods for Evaluating Solid Waste⁵

Method 200.7, Inductively Coupled Plasma - Atomic Emission Spectrometric Method for Trace Element Analysis of Water And Wastes, EPA-600/4-79-020, revised 1984⁵

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.

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 D 1176.

9. Calibration and Standardization

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

² Annual Book of ASTM Standards, Vol 11.01.

³ Annual Book of ASTM Standards, Vol 15.05.

⁴ Annual Book of ASTM Standards, Vol 14.02.

⁵ Available from U. S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, OH 45268.

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 |

9.2 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 *Precision*—The precision of this test method for silicon was determined by statistical examination of interlaboratory results according to Practice E 691 is as follows:⁶

13.1.1 *Repeatability*— The difference between successive test results obtained by the same operator on the same test material would, in the long run, in the normal and correct operation of the test method, be expected to be as indicated in Table 2. Average relative repeatability, that is, within laboratory consistency, is 6 %.

13.1.2 *Reproducibility*— The difference between two single and independent results, obtained by different operators working in different laboratories on identical test materials would, in the long run, in the normal and correct operation of this test method, be expected to be as indicated in Table 2. The relative reproducibility never exceeds twice the repeatability and on average is about 1-1/2 times, at 9 %, the repeatability.

NOTE 1—Ten laboratories and six samples were involved in this interlaboratory study. Data from only seven laboratories are available.

⁶ Supporting data are available from ASTM Headquarters. Request RR: D15-1013.



TABLE 2 Summary of Precision Parameters

| Sample | Average, µg/mL | Repeatability | | Reproducibility | |
|--------|-------------------|--------------------|-------------|--------------------|-------------|
| | | Absolute, µg/mL | Relative, % | Absolute, µg/mL | Relative, % |
| B | 246 | 20.6 | 8 | 27.4 | 11 |
| D | 250 | 16.1 | 6 | 28.1 | 11 |
| A | 513 | 28.0 | 5 | 55.7 | 11 |
| F | 515 | 28.0 | 5 | 35.5 | 7 |
| C | 645 | 26.4 | 4 | 48.6 | 8 |
| E | 649 | 36.4 | 6 | 53.0 | 8 |

13.2 *Bias*—Since there is no accepted reference material suitable for determining the bias for this test method, bias has not been determined.

14. Keywords

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

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