



Designation: D8315 – 20

Standard Test Method for Determination of Wear Metals and Contamination Elements in Used Industrial Oils by Sweeping Flat Electrode Atomic Emission Spectrometry¹

This standard is issued under the fixed designation D8315; 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 wear metals and contaminants in used industrial oils by sweeping flat electrode atomic emission spectroscopy (SFE-AES).²

1.2 Industrial oil includes lubricant oil, gear box oil, hydraulic fluid, compressor oil, turbine oil, synthetic oils, and other petroleum oils.

1.3 Method working range for every element is evaluated by equations in 15.2.1 and tabulated in Table 6.

1.4 Though this technique is designed to analyze non-suspended particles in lubricant samples, the precision statements published here were established solely from homogeneous oil samples per Practice D6300 requirements. Non-suspended particles, which are inhomogeneous by nature, were not sampled and evaluated for deriving precision statements for this test method (see Annex A1).

1.5 This test method provides a quick indication for abnormal wear and the presence of contamination in new or used industrial oils by immediately reporting:

- 1.5.1 Normal fine particles of specific wear metals;
- 1.5.2 Non-suspendable particles of specific wear metals and of contamination elements;
- 1.5.3 Less populated large particles (10 μm to 50 μm) of specific wear metals;
- 1.5.4 Contamination elements; and
- 1.5.5 Additive elements.

1.6 This test method uses oil-soluble elements for calibration and does not purport to relate quantitatively the values determined as insoluble particles to the dissolved metals.

¹ This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.03 on Elemental Analysis.

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² The sole source of supply of the apparatus (COA Oil & Grease Analyzer and accessories) known to the committee at this time is CYCP Oil Analyzers, LLC., 7 Beths Rd, Shrewsbury, MA 01545, www.cycp-oilanalyzers.com. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.

Analytical results are particle size dependent and low results may be obtained for those elements present in used oil samples as large particles (referenced by Test Methods D5185 and D6595).

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

1.8 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.9 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

- 2.1 *ASTM Standards*:³
 - D4057 Practice for Manual Sampling of Petroleum and Petroleum Products
 - D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products
 - D5185 Test Method for Multielement Determination of Used and Unused Lubricating Oils and Base Oils by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)
 - D6300 Practice for Determination of Precision and Bias Data for Use in Test Methods for Petroleum Products, Liquid Fuels, and Lubricants
 - D6595 Test Method for Determination of Wear Metals and Contaminants in Used Lubricating Oils or Used Hydraulic Fluids by Rotating Disc Electrode Atomic Emission Spectrometry

³ 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.

3. Terminology

3.1 Definitions:

3.1.1 *burn*, *vt*—in *emission spectroscopy*, to vaporize and excite a specimen with sufficient energy to generate spectral radiation.

3.1.2 *calibration*, *n*—the process by which the relationship between signal intensity and elemental concentration is determined for a specific element analyzed.

3.1.3 *calibration curve*, *n*—the graphical or mathematical representation of a relationship between the assigned (known) values of standards and the measured responses from the measurement system.

3.1.4 *calibration standard*, *n*—a standard having an accepted value (reference value) for use in calibrating a measurement instrument or system.

3.1.5 *emission spectroscopy*, *n*—measurement of energy spectrum emitted by or from an object under some form of energetic stimulation; for example, light, electrical discharge, and so forth.

3.1.6 *graphite disc electrode*, *n*—a soft form of the element carbon manufactured into the shape of a disc for use as a counter electrode in arc/spark spectrometers for oil analysis.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *arc discharge*, *n*—a self-sustaining, high current density, high temperature discharge, uniquely characterized by a cathode fall nearly equal to the ionization potential of the gas or vapor in which it exists.

3.2.2 *check sample*, *n*—a reference material usually prepared by a laboratory for its own use as a calibration standard, as a measurement control standard, or for the qualification of a measurement method.

3.2.3 *contamination element*, *n*—material resulting from contamination into an oil sample that may cause abnormal wear or lubricant degradation.

3.2.4 *counter electrode*, *n*—either of two graphite electrodes in an atomic emission spectrometer across which an arc or spark is generated.

3.2.5 *graphite flat electrode*, *n*—a soft form of the element carbon manufactured to possess a flat surface for use as both holding oil test specimen and a counter electrode in arc/spark spectrometers for oil analysis.

3.2.6 *graphite rod electrode*, *n*—a soft form of the element carbon manufactured into the shape of a rod for use as a counter electrode in arc/spark spectrometers for oil analysis.

3.2.7 *less populated large particles*, *n*—wear metal particles that cause emission intensity surges against most populated particles or background in SFE-AES test; whether they are suspendable depends on both specific physical properties of the particles and of oil fluid.

3.2.8 *non-suspendable particles*, *n*—wear metal and contamination elements that are not oil soluble and cannot remain in suspension without disturbance in a specific fluid sample. Non suspendable particles are always in suspension under working conditions of machinery and can be collected into a fluid sample via sampling practices described by Practices

D4057 and **D4177**; though the concentrations (in ppm) of non-suspendable particles do not characterize the sizes of particles, their values provide the partition of large and heavy particles against fine and small ones in fluid bulk.

3.2.9 *sparkling surface*, *n*—the surface of a graphite flat electrode facing to a graphite rod electrode; on the surface test specimen is attached and spark is generated for vaporizing the test specimen.

3.2.10 *standardization*, *n*—the process of re-establishing and correcting a calibration curve through the analysis of at least two known oil standards.

3.2.11 *suspendable particles*, *n*—wear metal and contamination elements that are not oil-soluble but remain in suspension in specific fluid bulk; suspendable particles might be captured by RDE-AES; they can be less assessed when oil sample is diluted by solvent; in most cases, suspendable particles of wear metals are fine particles and come from normal wear of moving parts.

3.2.12 *sweeping arc discharge*, *n*—the positions of two counter electrodes have relative motion during arc discharge in order for test specimen on sparking surface to be burned sequentially.

3.2.13 *wear metal*, *n*—material resulting from damage to a solid surface due to relative motion between that surface and a contacting substance or substances; wear metal is not oil-soluble.

3.2.14 *XY sweeping table*, *n*—a mechanical device for carrying the graphite flat electrode and moving the electrode under the rod electrode in pre-programmed two horizontal dimensions (X and Y).

3.3 Abbreviations:

3.3.1 **AES**—Atomic Emission Spectroscopy

4. Summary of Test Method

4.1 A used oil test specimen is placed on the sparking surface of a XY-movable (sweep-able) graphite flat electrode. A graphite rod electrode directs arc discharge to a point on the flat electrode surface. Evaporation and excitation of the specimen is implemented with controlled XY-motion of the flat electrode. The radiant energies of selected analytical lines at each point of the sparking surface are collected by a photon collection device and stored into computer memory.

4.2 In one test mode test fluid is well disturbed and the non-suspendable particles and the suspended particles are introduced onto spark gap. The test result represents total concentration of particles in the fluid bulk. In the second test mode, the fluid undergoes settling. Because the fluid is introduced to the spark gap by pouring it down, only the top part of the fluid, which only contains suspendable particles, is sparked. The second test result represents the concentration of suspendable particles. The concentration of non-suspended particles can be calculated by finding the difference of two tests of the same test fluid.

4.3 In one mode the measured concentrations from all points of the sparking surface can be combined into one average concentration for the element in the entire area. In a

second mode of analysis each reading is considered separately with the understanding that each spark is analyzing a different area of an inhomogeneous sample. With either numerical or graphical analysis an understanding of the particle size and distribution in the sampled area can be obtained in this second mode.

5. Significance and Use

5.1 *Used Industrial Oil*—The detection of large particles are important inputs for used industrial lubricant condition monitoring. For wear metals, these particles, in size, are represented by those between 20 μm and 50 μm in engine oil, 80 μm or greater in gear-box oil. In desert or windy areas, large sand and dust particles can enter in-service lubricant. The concentrations contributed from large particles can be more sensitive to serious or catastrophic failure of industrial equipment than those from 10 μm or less. In spectroscopic analysis, excluding large particles significantly under-reports the concentrations of wear and contamination elements. The corresponding results may not represent the actual state of in-service lubricant. Because this test method posts less limitation on the size of wear metal particles while still reporting normal fine wear particles, it provides a means to assess wear and contamination elements in a comprehensive range of the size of particulates and raises the fidelity of spectroscopic analysis of in-service lubricant.

5.2 *Non-suspendable Particles in Used Industrial Oil*—The increase of non-suspendable particles suggests excessive wear or poor sealing of machinery, or both. Large amounts of such particulate in industrial oil bulk itself are harmful to moving parts of machinery. This test method provides another means to identify the presence or absence of the non-suspendable particles for machinery condition monitoring.

6. Interferences

6.1 *Spectral*—Most spectral interferences can be avoided by judicious choice of spectral lines. High concentrations of additive elements can have an interfering influence on the spectral lines used for determining wear metals (referenced by Test Methods D5185 and D6595). Instrument manufacturers usually compensate for spectral interferences during factory calibration. A background correction system, which subtracts unwanted intensities on either side of the spectral line, shall also be used for this purpose. When spectral interferences cannot be avoided with spectral line selection and background correction, the necessary corrections shall be made using the computer software supplied by the instrument manufacturer.

6.2 *Particulate*—When large particles are detected, the analytical results will be lower than the actual concentration they represent. Large particles may not be fully vaporized by the spark (referenced by Test Methods D5185 and D6595).

6.3 *Particle Suspension*—Large particles can only remain in suspension for a short time after vibration, depending on the viscosity of test specimen. Wear metal and contamination elements would be significantly under-reported if the test specimen is not mixed thoroughly immediately before it is poured down to sparking surface.

6.4 *Less Populated Particles*—If large particles are not on the route of sparking, they will not be detected and reported. On the other hand, false results may be reported if a lot of small particles are aggregated either spatially or by bonded forces, or both.

6.5 *Poor Maintenance*—Because test specimen is burned in the sparking chamber, ashes and combustion residuals can be adsorbed or deposited everywhere in the chamber. Dry paper tissues can be used to clean the chamber surfaces in order to maintain the instrument in a good and operable condition. Particular attention should be paid to the groove of the rod electrode holder, the lens, and the fixture of flat electrode, which shall be cleaned on a daily basis. Also, the fixture of graphite flat electrode shall be cleaned after every sample.

6.6 *Viscosity Effects*—Differences in viscosity of used oil samples will cause differences in sample film thickness on the flat electrode. Internal references of the instrument will compensate for a portion of the differences. In used oil applications, the hydrogen 486.10 nm spectral line has become the industry standard for use as an internal reference. Without a reference, trended data on used oil will be adversely affected if the sample base stock has a different viscosity from the base line samples (referenced by Test Method D6595).

7. Apparatus

7.1 *Electrode Sharpener*—An electrode sharpener is necessary to remove the contaminated portion of the rod electrode remaining from the previous determination. It also forms a new 160° angle on the end of the electrode. Electrode sharpeners are not required for instruments using a pre-shaped disc electrode as the counter electrode.

7.2 *Sweeping Flat Electrode Atomic Emission Spectrometer*, a simultaneous spectrometer consisting of an excitation source (which uses a graphite flat electrode lying on an XY sweeping table), polychromator optics and a readout system. Suggested elements and wavelengths are listed in Table 1. When multiple wavelengths are listed, they are in the order of preference or desired analytical range.⁴

⁴ The method and apparatus for emission spectroscopy utilizing a flat plate for sample holding and excitation is covered by a patent (U.S. Patent #8559004). Interested parties are invited to submit information regarding the identification of an alternative(s) to this patented item to the ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend.

TABLE 1 Elements and Recommended Wavelengths

Element	Wavelength, nm	Element	Wavelength, nm
Aluminum	308.21	Nickel	341.48
Barium	230.48, 455.40	Phosphorus	255.32, 214.91
Boron	249.67	Potassium	766.49
Calcium	393.37, 445.48	Silicon	251.60
Chromium	425.43	Silver	328.07, 243.78
Copper	324.75, 224.26	Sodium	588.89, 589.59
Iron	259.94	Tin	317.51
Lead	283.31	Titanium	334.94
Lithium	670.78	Tungsten	400.87
Manganese	403.07, 294.92	Vanadium	290.88, 437.92
Magnesium	280.20, 518.36	Zinc	213.86
Molybdenum	281.60		

8. Reagents and Materials

8.1 *Base Oil*, a 75 cSt base oil free of analyte to be used as a calibration blank or for blending calibration standards, or both.

8.2 *Check Samples*, an oil standard or sample of known concentration which is periodically analyzed as a go/no go sample to confirm the need for standardization based on an allowable 10 % accuracy limit.

8.3 *Cleaning Solution*, an environmentally safe, nonchlorinated, rapid evaporating, and non-film producing solvent, to remove spilled or splashed oil sample in the sample stand.

8.4 *Graphite Flat Electrode*, a graphite flat electrode of high-purity graphite (spectroscopic grade). Dimensions of the electrodes shall conform to those shown in Fig. 1.

8.5 *Glass Cleaning Solution*, capable of cleaning and removing splashed oil sample from the quartz window that protects the entrance lens and fiber optic. Isopropyl rubbing alcohol or ammonia-based window cleaner has been found to be suitable for this purpose.

8.6 *Organometallic Standards*, single or multi-element blended standards for use as the high concentration standard for instrument standardization purposes or for use as a check sample to confirm calibration. Typical concentrations in the upper calibration point standard for used oil applications is 100 mg/kg for wear metals and contaminants, and 1200 mg/kg for additive elements.

8.6.1 Standards have a shelf-life and shall not be used to standardize an instrument if they have exceeded the expiration date.

8.7 *Counter Electrode*—The counter electrode can be either a rod or a plate. The counter electrode must be high-purity graphite (spectroscopic grade). Dimensions of the counter electrodes shall conform to those shown in Fig. 2.

8.8 *Quality Control Samples*, preferably are portions of one or more liquid petroleum materials that are stable and repre-

sentative of the samples of interest. These QC samples can be used to check the validity of the testing process as described in Section 13.

9. Hazards

9.1 Used oil can contain hazardous material from component source or the contamination process, or both. Follow applicable regulations and procedures when disposing of used oil.

9.2 Incorrect installation of counter electrodes can cause damage of arc discharge system. Follow instructions of the manufacturer’s manual for installing the electrodes.

10. Sampling

10.1 The used oil sample taken for the analysis must be representative of the entire system. Good sampling procedures are key to good analyses and samples must be taken in accordance with Practice D4057.

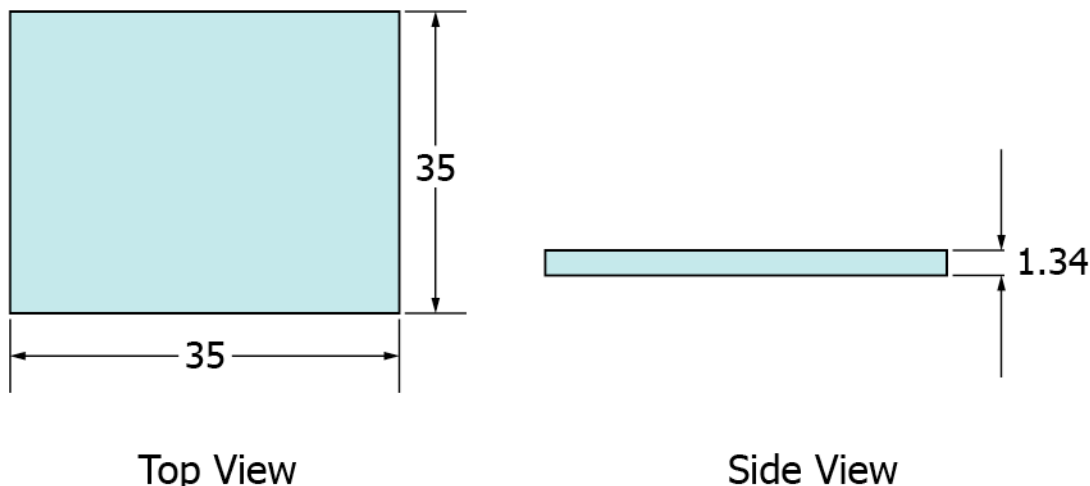
11. Preparation of Apparatus

11.1 *Homogenization*—Used oil samples may contain particulate matter and, in order to be representative, must always be vigorously shaken or vibrated prior to pouring a test specimen to the sparking surface of a graphite flat electrode.

11.2 *Large Wear Metal and Foreign Particles*—Large particles cannot suspend in oil for very long. In order to capture them, used oil samples must be immediately placed on the sparking surface right after being shaken or vibrated. Ultrasonic homogenization may not always be effective for agitating large or less populated particles to suspend.

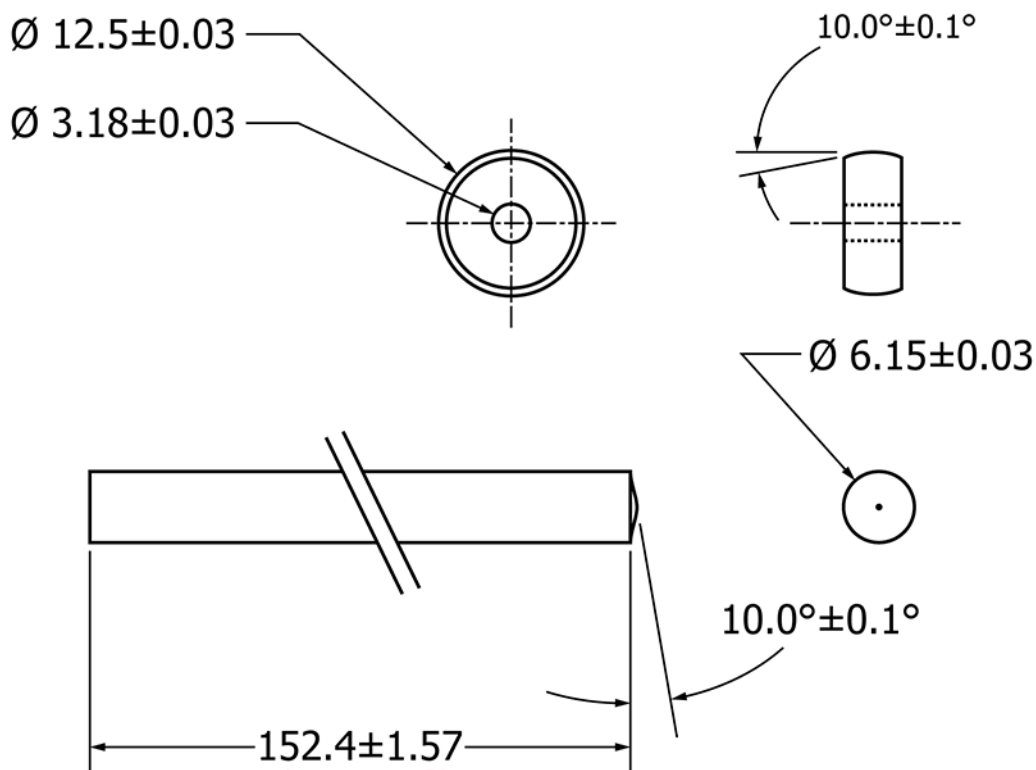
11.3 *Non-suspendable Particles Test:*

11.3.1 *Prepare Test Specimen for the Test of Suspendable Particles*—Do not disturb the sample bottle for 20 min to 30 min, so that non-suspendable particles will settle down to the bottom of the sample bottle. The settling time of the non-suspendable particles depends on the viscosity of the specimen, and can be determined empirically. In most cases,



NOTE 1—All dimensions are in millimeters (± 0.05); material is high-purity graphite (spectroscopic grade).

FIG. 1 Graphite Flat Electrode



NOTE 1—All dimensions are in millimeters; material is high-purity graphite (spectroscopic grade).

FIG. 2 Graphite Counter Electrode

<https://standards.iteh.ai>

after settling 30 min, all non-suspendable particles will be cleared out of the major bulk of the specimen.

11.3.2 *Prepare Test Specimen for the Test of Total Particles (Suspendable Plus Non-suspendable)*—Follow 11.2 for the same test specimen used in 11.3.1.

11.4 *Validation Check*—A go/no go standardization check can be performed with one or more check samples to confirm calibration prior to the analysis of routine samples. A calibration standard or known oil sample can be used for this purpose. The optical profile and standardization routine recommended by the instrument manufacturer shall be performed if the validation check fails to meet the $\pm 10\%$ accuracy guidelines for each element of interest.

12. Calibration and Standardization

12.1 *Multipoint Calibration Curve*—A software user interface is provided to conduct multi-point calibration and can be used by users if they feel needed. The calibration curve for each element can be established or re-established through the analysis of organometallic standards at known concentrations and correction factors are set or reset to produce a linear response. The wavelengths used for the calibration software are listed in Table 1. The linear range must be established once for the particular instrument being used. This is accomplished by running intermediate standards between the blank and the working standard and by running standards containing higher concentrations than the working standard. For operational details, consult manufacturer manual and software updates.

12.2 *Routine Standardization*—A minimum of a two-point routine standardization shall be performed if the instrument fails the validation check or at the start of each working shift. A minimum of three analyses shall be made using the blank and working standard.

13. Procedure

13.1 *Installation of Graphite Flat Electrode*—A new graphite flat electrode and a new or re-pointed graphite rod electrode must be used for each analysis. A laboratory grade paper towel shall be used during the installation of the flat electrode in order to protect it from contamination from the fingers. Cleaning procedures recommended by the manufacturer shall be followed to avoid cross contamination or buildup of spilled sample. Test specimen must be introduced after the flat electrode has been properly installed.

13.2 *Introduction of Fluid Samples*—Used oil samples and oil standards shall be poured on the surface of the flat electrode prior to analysis. At least 1 mL capacity is needed to cover the entire surface of the flat electrode. Exercise care to pour the sample consistently to the same level on the flat electrode. However, the actual sample volume retained on the flat electrode depends on the viscosity of a sample and cannot always be repeatable among various types of samples, which is not critical as the impact of unrepeatable sample volume held by the flat electrode can be compensated in the calculation.

13.3 *Analysis of Non-suspendable Particles*—Analyze the same test specimen twice: