Designation: ~~D6595 – 00 (Reapproved 2011)~~ D6595 – 16

Standard Test Method for
Determination of Wear Metals and Contaminants in Used Lubricating Oils or Used Hydraulic Fluids by Rotating Disc Electrode Atomic Emission Spectrometry[1]

This standard is issued under the fixed designation D6595; 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~~ Scope*

1.1 This test method covers the determination of wear metals and contaminants in used lubricating oils and used hydraulic fluids by rotating disc electrode atomic emission spectroscopy (RDE-AES).

1.2 This test method provides a quick indication for abnormal wear and the presence of contamination in new or used lubricants and hydraulic fluids.

1.3 This test method uses oil-soluble metals for calibration and does not purport to relate quantitatively the values determined as insoluble particles to the dissolved metals. Analytical results are particle size dependent and low results may be obtained for those elements present in used oil samples as large particles.

1.4 The test method is capable of detecting and quantifying elements resulting from wear and contamination ranging from dissolved materials to particles approximately 10 μm in size.

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

1.5.1 The preferred units are mg/kg (ppm by mass).

1.6 *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.*

---

[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.

Current edition approved ~~May 1, 2011~~July 1, 2016. Published ~~August 2011~~July 2016. Originally approved in 2000. Last previous edition approved in ~~2005~~2011 as ~~D6595–00(2005).~~D6595 ~~DOI: 10.1520/D6595-00R11.~~ – 00 (2011). DOI: 10.1520/D6595-16.

***A Summary of Changes section appears at the end of this standard**

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products

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 determination of the values of the significant parameters by comparison with values indicated by a set of reference standards.

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.

3.2 *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 *contaminant, n*—material in 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 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.

3.2.6 *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 *profiling, n*—to set the actual position of the entrance slit to produce optimum measurement intensity.

3.2.8 *standardization, n*—the process of reestablishing and correcting a calibration curve through the analysis of at least two known oil standards.

3.2.9 *uptake rate, n*—the amount of oil sample that is physically carried by the rotating disc electrode into the arc for analysis.

3.2.10 *wear metal, n*—material resulting from damage to a solid surface due to relative motion between that surface and a contacting substance or substances.

4. Summary of Test Method

4.1 Wear metals and contaminants in a used oil test specimen are evaporated and excited by a controlled arc discharge using the rotating disk technique. The radiant energies of selected analytical lines and one or more references are collected and stored by way of photomultiplier tubes, charge coupled devices or other suitable detectors. A comparison is made of the emitted intensities of the elements in the used oil test specimen against those measured with calibration standards. The concentrations of the elements present in the oil test specimen are calculated and displayed. They may also be entered into a data base for processing.

5. Significance and Use

5.1 *Used Lubricating Oil*—The determination of debris in used oil is a key diagnostic method practiced in machine condition monitoring programs. The presence or increase in concentration of specific wear metals can be indicative of the early stages of wear if there are baseline concentration data for comparison. A marked increase in contaminant elements can be indicative of foreign materials in the lubricants, such as antifreeze or sand, which may lead to wear or lubricant degradation. The test method identifies the metals and their concentration so that trends relative to time or distance can be established and corrective action can be taken prior to more serious or catastrophic failure.

---

[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.

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. 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 *Viscosity Effects*—Differences in viscosity of used oil samples will cause differences in uptake rates. Internal references of the instrument will compensate for a portion of the differences. In used oil applications, the hydrogen ~~486.10 nm~~ 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.

6.3 *Particulate*—When large particles over ~~10 µm~~ 10 µm in size are detected, the analytical results will be lower than the actual concentration they represent. Large particles may not be effectively transported by the rotating disk electrode sample introduction system into the arc, nor will they be fully vaporized by the spark.

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 *Rotating Disc Electrode Atomic Emission Spectrometer,* a simultaneous spectrometer consisting of excitation source, 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.

7.3 *Heated Ultrasonic Bath (Recommended),* an ultrasonic bath to heat and homogenize used oil samples to bring particles into homogeneous suspension. The ultrasonic bath shall be used on samples containing large amount of debris and those that have been in transit or stored for ~~48 hours~~ 48 h or longer.

8. Reagents and Materials

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

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, non-chlorinated, rapid evaporating, and non-film producing solvent, to remove spilled or splashed oil sample in the sample stand.

8.4 *Disc Electrode,* a graphite disc 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~~100 mg ~~mg/kg~~ /kg for wear metals and contaminants, and ~~900~~900 mg ~~mg/kg~~ /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.

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		