Designation: D7417 – 10

Standard Test Method for Analysis of In-Service Lubricants Using Particular Four-Part Integrated Tester (Atomic Emission Spectroscopy, Infrared Spectroscopy, Viscosity, and Laser Particle Counter)[1]

This standard is issued under the fixed designation D7417; 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 quantitative analysis of in-service lubricants using an automatic testing device that integrates these varied technologies: atomic emission spectroscopy, infrared spectroscopy, viscosity, and particle counting.

1.2 This is suited for in-service lubricating oils having viscosities in the range between ISO 10 and ISO 320 and properties in the ranges given in Tables 1 and 2.

1.3 This test method may be used to establish trends in wear and contamination of in-service lubricants and may not give equivalent numerical results to current ASTM test methods.

1.4 This test method is not intended for use with crude oil.

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

1.6 *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.* (Specific hazard statements are given in Section 9 and 11.3.)

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and Calculation of Dynamic Viscosity)

D2270 Practice for Calculating Viscosity Index from Kinematic Viscosity at 40 and 100°C

D2896 Test Method for Base Number of Petroleum Products by Potentiometric Perchloric Acid Titration

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

D4739 Test Method for Base Number Determination by Potentiometric Hydrochloric Acid Titration

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

D7042 Test Method for Dynamic Viscosity and Density of Liquids by Stabinger Viscometer (and the Calculation of Kinematic Viscosity)

E2412 Practice for Condition Monitoring of In-Service Lubricants by Trend Analysis Using Fourier Transform Infrared (FT-IR) Spectrometry

2.2 *ISO Standards:*[3]

ISO 4406:99 Hydraulic Fluid Power Solid Contaminations Code

ISO 11171 Automatic Particle Counter Calibration Procedures

3. Terminology

3.1 *Definitions:*

3.1.1 *electrode*, *n*—*in an integrated tester*, set of two (upper and lower) used in excitation of wear metals during emission spectroscopic testing.

3.1.2 *emission spectrometer*, *n*—component used to report elements in parts per million in lubricants. This process measures 20 different wear/additive metals that can be present in the used lubricant after the oil has been in service for a period of time. Test Method D6595 can be used for reference or definition.

3.1.3 *infrared spectrometer*, *n*—component used to report condition and contamination of the lubricant (for example, water, oxidation, fuel dilution (gasoline and diesel), nitration, glycol, soot, calculated viscosity, and base number). Practice E2412 can be used for reference or definition.

3.1.4 *integrated tester*, *n*—instrument used to analyze in-service lubricants for maintenance, preventative maintenance and service recommendations. This instrument utilizes any

[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.96 on In-Service Lubricant Testing and Condition Monitoring Services.

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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.

[3] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

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TABLE 1 Element Test Parameters Measured, Calculated, and Reported

Element	Low Range, mg/kg	High Range, mg/kg	Element	Low Range, mg/kg	High Range, mg/kg
Aluminum	5 to 100	NA	Molybdenum	10 to 1000	NA
Barium	25 to 150	150 to 2000	Nickel	5 to 100	NA
Boron	5 to 100	100 to 1000	Phosphorous	100 to 600	600 to 4000
Calcium	25 to 500	500 to 9000	Potassium	10 to 1000	1000 to 4000
Chromium	8 to 100	NA	Silicon	5 to 150	150 to 3000
Copper	5 to 500	500 to 1000	Sodium	10 to 1000	NA
Iron	6 to 1000	1000 to 3000	Tin	6 to 100	NA
Lead	6 to 150	NA	Titanium	8 to 100	NA
Magnesium	5 to 100	100 to 3000	Vanadium	7 to 100	NA
Manganese	5 to 100	NA	Zinc	8 to 100	100 to 4000

TABLE 2 Physical Properties Parameters Measured, Calculated, and Reported

NOTE—Review Test Method D4739 and D2896 for particular lubricating oil applications.

Physical Property	Range
Water, wt%	0.1 to 3
Glycol, wt%	0.1 to 2
Soot, wt%	0.1 to 4
Fuel Dilution, wt%	0.1 to 15
Oxidation, abs.	0.1 to 50
Nitration, abs.	0.1 to 35
Calculated Viscosity - IR	4 to 35 (100° cSt)
Viscosity 40°C, cSt (optional)	30 to 320
Viscosity 100°C, cSt (optional)	5 to 25
Viscosity Index	5 to 150
Base Number, mg KOH/g	1.0 to 17

combination of the following: emission spectrometer, infrared device, viscometer, and particle counter.[4]

3.1.5 *sample transport system*, *n—in an integrated tester*, computer controlled assembly that directs the oil samples throughout the integrated tester.

3.1.6 *spark chamber*, *n—in an integrated tester*, area housing the upper and lower electrodes for emission spectrometer.

3.1.7 *viscometer*, *n—in an integrated tester*, a viscometer using calibrated measurements similar to a kinematic viscometer capable of reporting viscosity at 40°C or 100°C in centistokes (cSt), and providing a calculated viscosity index. The results are also used to determine fuel dilution in diesel lubricants. Reference to Test Method D445, Practice D2270, or Test Method D7042 can be used as a reference for viscosity definition. Although the integrated tester does not print out SI units for measuring viscosity, reporting in mm^2/s can be determined.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *electrode gap*, *n—in an integrated tester*, specific distance between the upper and lower electrodes in the spark chamber.

3.2.2 *particle counter*, *n*—component in this particular integrated tester that is used to count particles using laser and high-resolution digital counter reporting in 4 µm(c), 6 µm(c), 14 µm(c) or 2 µm, 5 µm, 15 µm, using ISO 11171 calibration method and or ISO 4406:99 reporting method.

3.2.3 *sanitized cleaning swabs*, *n—in an integrated tester*, used to clean the electrodes after each sample analysis.

3.3 *Abbreviations:*

AES = Atomic Emission Spectroscopy
IR = Infrared
OEM = Original Equipment Manufacturer

4. Summary of Test Method

4.1 A sample of in-service lubricant is collected into a clean, new 120 mL sample bottle from the equipment being tested, preferably within 30 min of equipment shutdown. Lubricant description and service information should be recorded for proper evaluation. The integrated tester is prepared for analysis according to the operations manual and on-screen prompts. The lubricant sample is placed into the sample transport system and is analyzed using available integrated devices. The application software guides the entire procedure, controls the transfer of the sample, stores data, and generates on screen and printed results with a printed generic recommendation of the lubricant's physical condition.

5. Significance and Use

5.1 The integrated tester is primarily used to perform on-site analysis of in-service lubricants used in the automotive, highway trucking, mining, construction, off-road "mining," marine, industrial, power generation, agriculture, and manufacturing industries.

5.2 The immediate results of analysis of in-service lubricants are critical when performing proactive and preventative

[4] The sole source of supply of the apparatus (OSA Lab Four Part Analyzer and accessories) known to the committee at this time is On-Site Analysis, Inc., 7108 Fairway Drive, Suite 130, Palm Beach Gardens, FL 33418 (manufacturing division in Marlborough, MA), www.on-siteanalysis.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.

maintenance. On-site oil analysis, when used in conjunction with these programs, allows continuous system monitoring and contamination control potentially improving equipment "up-time" and equipment life.

6. Interferences

6.1 *Sample Size*—Using less than the required 130 mL of sample for the analysis may result in erroneous numbers.

6.2 *High Concentration*—Of a contaminant such as water and or soot in the IR device might cause other parameters to be blocked out in the IR band resulting in a default returned value of not applicable or "n/a" for these other parameters.

7. Apparatus

7.1 *Integrated Tester*—The particular integrated tester will incorporate two or more of the following sensors: an emission spectrometer, an infrared device, a viscometer, or a particle counter.

7.2 *Emission Spectrometer*—Consists of an excitation source, spark stand, and optical system.

7.3 *Infrared Device*—Consists of an infrared source, sample cell, and optical system.

7.4 *Dual Temperature Viscometer*—Consists of a temperature controlled sample reservoir and electronic control system.

7.5 *Particle Counter*—Consists of a sophisticated sensor cell using red laser light extinction technology utilizing the ISO 11171 calibration and ISO 4406:99 reporting.

7.6 *Sample Container*—Sample container of no less than 118 mL that is free of contaminants shall be used for the in-service oil samples and shall be discarded after use. The maximum dimensions of 10.8 cm high, 8.9 cm in diameter, and an opening of no less than 1.6 cm shall be used.

7.7 *Sonic Bath*—Water-filled, vibrating tool used to be sure that all of the contamination is in suspension in the lubricant sample. This is mostly used when preparing a lubricant sample for a particle count analysis procedure.

7.8 *Computer Application Software and Operations Manual*—The computer application software provides the functionality for the particular integrated tester; an electronic user with help screens and a condensed reference manual for quick reference, interface, hierarchical equipment database to store, analyze and manage data, embedded logic for data interpretation; and automatic reporting tools. A complete operations manual and troubleshooting guide accompany the software application.

8. Reagents and Materials

8.1 The particular integrated tester uses the following supplies which are available from the manufacturer to ensure accurate operation of the integrated tester:

8.1.1 *Electrodes*—A high-purity silver electrode set (upper and lower).

8.1.2 *Cleaning Fluid*—An environmentally safe mineral oil, to clean the instruments internal flow system. The cleaning solution in most cases can also be used as the base oil for the calibration of the instrument. This product is a nonflammable product consisting of technical grade Semtol White Mineral Oil.[5]

8.1.3 *Test Standard*—This solution is intended for use as a calibration standard for the integrated tester. It is a multi-element solution of Primol N352[6] that was prepared with neutral oil as a functional antioxidant for specific concentrations. The certified concentrations are based upon the assayed concentrations of the raw materials and the gravimetric procedures used to prepare the final standard. The uncertainty associated with each certified concentration is ±2 mg/kg (parts per million). In order to verify these certified values, the final solution was analyzed by plasma emission spectroscopy (ICP).

NOTE 1—The manufacturer guarantees the accuracy of this solution until the expiration date shown, provided it is kept tightly capped and stored in original bottle under normal laboratory conditions. Do not refrigerate or store in direct sunlight. Minimize exposure to moisture or high humidity. It is recommended that the solution be thoroughly mixed, by shaking the bottle, prior to use.

8.1.4 *High Viscosity Fluid*—For use with viscometer only. This fluid is blended with known viscosities and is periodically analyzed to confirm calibration or to recalibrate the viscometer. It consists of a highly refined mineral oil and a detergent/dispersant engine oil additive package.

8.1.5 *Low Viscosity Fluid*—For use with viscometer only. This fluid is blended with known viscosities and is periodically analyzed to confirm calibration or to recalibrate the viscometer. It consists of a highly refined mineral oil and a detergent/dispersant engine oil additive package.

8.1.6 *Reference Oil*—Used to verify accuracy of physical property test results reported by infrared device. The reference oil has known physical properties which are tested against the infrared device using the software to determine standardization. It consists of a highly refined mineral oil combined with zinc alkyl dithiophosphate.

8.1.7 *Sanitary Cleaning Swabs*—Used to clean the electrodes after each sample analysis. Operator shall use contaminant free cotton swabs that are not treated with sodium from the hypochlorite bleaching process. Check the manufacturer's specifications of the cleaning swabs before use to determine if the bleaching process has been performed.

9. Hazards

9.1 Used oil can contain hazardous material from component source and or the contamination process. Wear appropriate personal protective equipment to prevent repeated or prolonged contact with used oil. Follow proper oil handling procedures

[5] The sole source of supply of the apparatus known to the committee at this time is Sonneborn, Inc., 771 Old Saw Mill River Rd., Tarrytown, NY 10591-6716. 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.

[6] The sole source of supply of the apparatus known to the committee at this time is ExxonMobil (Corporate Headquarters), 5959 Las Colinas Blvd., Irving, TX 75039-2298. 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.