Designation: ~~D7417 – 10~~ D7417 – 17

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~~ 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~~ safety, health, and ~~health~~environmental practices and determine the applicability of regulatory limitations prior to use.* (Specific hazard statements are given in Section 9 and 11.3.)

1.7 *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:*[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 °C 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

[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 D02.96.07 on Integrated Testers, Instrumentation Techniques for In-Service Lubricants.

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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.

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

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

Physical Property	Range
~~Water, wt%~~	~~0.1 to 3~~
Water, % by mass	0.1 to 3
~~Glycol, wt%~~	~~0.1 to 2~~
Glycol, % by mass	0.1 to 2
~~Soot, wt%~~	~~0.1 to 4~~
Soot, % by mass	0.1 to 4
~~Fuel Dilution, wt%~~	~~0.1 to 15~~
Fuel Dilution, % by mass	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 40 °C, cSt (optional)	30 to 320
~~Viscosity 100°C, cSt (optional)~~	~~5 to 25~~
Viscosity 100 °C, cSt (optional)	5 to 25
Viscosity Index	5 to 150
~~Base Number, mg KOH/g~~	~~1.0 to 17~~
Base Number, mg/g KOH	1.0 to 17

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 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~~40 °C or ~~100°C~~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.

[4] The sole source of supply of the apparatus ~~(OSA Lab Four Part Analyzer~~ (MicroLab 40 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.~~ Spectro Scientific, Inc., One Executive Dr., Suite 101, Chelmsford, MA 01824, www.spectrosci.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.

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,~~ 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~~ 120 mL sample bottle from the equipment being tested, preferably within ~~30 min~~ 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 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~~ 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~~ 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~~ 10.8 cm high, 8.9 cm in diameter, and an opening of no less than ~~1.6 cm~~ 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~~±2 mg ~~mg/kg~~ /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 during sampling and throughout analytical process. Follow applicable regulations and procedures when disposing of used oil.

10. Sampling

10.1 When possible, obtain sample within 30 min of equipment shutdown so that wear metals are retained in suspension and are removed with the sample. Use every possible method to keep the sample clear from external contamination. The integrated tester is capable of analyzing in-service lubricants having viscosities in the range of ISO 10 to ISO 320, including hydraulic, transmission, and power steering lubricants. Refer to Practice D4057 when preparing sample for processing.

11. Preparation of Apparatus

11.1 Turn the main power switch of the integrated tester to "ON," and push the red reset button to activate the computer and all components.

11.2 The integrated tester will run a diagnostics process while warming up. After the diagnostic page appears, it is a good practice to allow the integrated tester to have a warming up period of ~~10 min.~~10 min.

11.3 Fill the flush container with the cleaning fluid; this is a nonflammable product. (**Warning—**Empty the waste fluid into an approved waste disposal container in accordance with local laws.)

11.4 Fill the printer paper tray if using printer in conjunction with unit.

11.5 *Check Electrode Gap*—The gap between upper and lower electrodes shall be checked routinely to verify the proper operation of the emission spectrometer. A specially designed gapping tool is supplied with each integrated tester for setting the proper electrode gap. Electrode gap can ONLY be adjusted before elemental standardization (calibration).

11.6 *Clean Electrodes*—The outer and inner surfaces of the upper electrode, as well as the lower electrode, shall be cleaned with sanitary cleaning swabs.

11.6.1 Open the integrated tester spark chamber door. Using a clean dry cotton swab, wipe the electrodes clean of all sample residues (see Note 2).

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[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.

11.6.2 Using another clean dry cotton swab, wipe the inside of the upper electrode holder by inserting the swab down through the center hole of the brass holder (see Note 2).

NOTE 2—Do not use cleaning fluid when cleaning electrodes. Always use a clean dry cotton swab when cleaning electrodes.

11.6.3 When testing for viscosity, no special requirements are needed to prepare the oil sample.

11.6.4 When particle count testing is required, the process requires placing the in-service oil sample bottle into the sonic bath for a ~~1 min~~ 1 min period as mentioned in the operations manual. This will prepare the sample for processing.

12. Calibration and Standardization

12.1 *Baseline Test*—Verify system cleanliness by running a baseline test that uses the system's cleaning fluid. This process is performed under the "Standardization" button on the main screen of the integrated tester. Baseline testing ensures that the atomic emission spectrometer is not contaminated and will give a pass or fail indication on the computer screen to let the operator know if it is acceptable to proceed with the next sample. The specifications are ~~±5~~ ± 5 mg ~~mg/kg~~ /kg on all wear and additive metals except phosphorus which is ~~±10~~ ± 10 mg ~~mg/kg~~ /kg.

12.2 If the baseline test fails to meet the required cleanliness range of the atomic emission spectrometer, the system will require flushing until a passing baseline test is achieved. The baseline test process should be run every ~~24 h.~~ 24 h.

12.3 *Standardization*—Standardize the unit's atomic emission spectrometer by using an atomic emission test standard fluid as mentioned in 8.1.3 (see Note 3).

12.3.1 If any device in the particular integrated tester fails to meet the required accuracy range of the test standard, a full standardization shall be performed (see Note 3).

NOTE 3—A standardization check can be run at any time to verify calibration and cleanliness of the integrated tester. A detailed standardization process is located within the operations manual.

12.3.2 This process must be run when reference oil test fails as outlined in the instrument operations manual.

12.4 *Reference Oil Test*—See 8.1.6. This process is recommended every 96 operational hours. This process will determine if the IR component of the integrated tester is clean of soot/solids and other debris from previous samples processed. The software for the particular integrated tester will determine the pass/fail criteria and will post a message on the computer screen stating whether standardization was completed successfully.

12.5 *Errors*—If the instrument returns an "error" message, refer to the troubleshooting guide in the operations manual.

12.6 *Calibration of Particle Counter*—Calibration shall be done by the manufacturer and should be done approximately every 1000 particle samples processed or every 2 years, whichever comes first.

13. Conditioning

13.1 No preconditioning is required for lubricant samples except when a particle count is required. If a particle count is required, the lubricant sample should be obtained hot and within 30 min of equipment shutdown. Lubricant sample must be shaken in a sonic bath immediately prior to testing to ensure all material is suspended uniformly.

14. Procedure

14.1 Collect lubricant sample into a clean, new sample bottle; at least ~~118 mL~~ 118 mL of the lubricant being tested is recommended.

14.2 Make sure the integrated tester is in the proper software/test mode for application being tested (for example, automotive, trucking, off-road, industrial, etc.).

14.3 Place the filled sample bottle underneath the sipper/extraction tube of the integrated tester. This device is located either in the middle of the unit or on the top right hand corner of the integrated tester.

14.4 Follow the on-screen instructions. Answer all screen prompts in the order that they appear on the touch screen. This process will lead the operator through the equipment and oil usage information: component type; oil type; oil viscosity; miles or hours, or both, that the oil has been in service; time on component being sampled; etc.

14.5 The accuracy of the information entered is crucial to allow the software to process the oil sample correctly. Review all of the vehicle and in-service lubricant information entered and make any necessary changes.

14.6 Press "START," and follow any on-screen instructions, including placing lubricant sample into the sample transport system.

14.7 The integrated tester has an option of two delivery systems using internal valves and pumps. If the integrated tester has a particle counter, the delivery system runs separately from the atomic emission spectrometer, viscometer and IR delivery system.

14.8 The analysis process will take up to 15 min to complete. The instrument transfers oil automatically to the selected devices within the integrated tester. 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 lubricants physical condition.

15. Calculation or Interpretation of Results

15.1 Based on the information entered before sample processing and the results obtained from analysis of the test sample, the software program performs all required calculations and generates a data report. Optionally, a diagnostics and results interpretation report may be printed.

15.2 Test results can include equipment wear ratings (for example, normal, abnormal or severe) and maintenance recommendations.

16. Report

16.1 Report test results as follows:

16.1.1 *Wear Metals and Contaminant Elements*—Report to the nearest part per million.

16.1.2 *Water, Glycol, Soot, and Fuel Dilution*—Report either in whole values as weight % or in the abbreviated format of "present" or "not present."

16.1.3 *Base Number*—Report in mg of KOH/g. Test Method D4739 can be used for reference and definition base number.

16.1.4 *Oxidation and Nitration*—Report in absorbance units (Abs).

16.1.5 *Viscosity*—Report in centistokes (cSt) at ~~100°C~~100 °C or ~~40°C,~~40 °C, or both. If viscosity results at both temperatures are measured, report viscosity index.

16.1.6 *Particle Count*—Report by particle size and number of particles found.

16.2 The computer software displays the test results on the screen. The results can then be printed automatically in one of several print format options. An example of a typical report is shown in Appendix X1.

17. Precision and Bias[7]

17.1 The precision statements are based on results from a four-laboratory study conducted in November 2004. The study involved five samples (five instruments; five operators), comprising used oil from various in-service applications. Statistical analysis revealed spectroscopic and instrumental differences. The differences are included in the estimated repeatability.

17.2 *Repeatability*—The difference between successive results obtained by the same operator with the same apparatus under constant operating conditions, on identical test materials would, in the long run, in the normal and correct operation of the practice, exceeds the values shown in Table 3 in only one case in twenty.

17.3 *Reproducibility*—The reproducibility of this test method is not currently available. The reproducibility will be available within five years following the approval of this test method.

17.4 *Bias*—The measured quantity is an effective value for which there is not an established reference material available for measurement.

18. Keywords

18.1 arc-spark emission spectrometry; baseline; check flush; contaminants; electrodes; infrared spectrometry; in-line dual temperature viscometer; in-service lubricants testing; integrated tester; kinematic viscosity; particle counter; standardization; test standard; wear metals

[7] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1702. Contact ASTM Customer Service at service@astm.org.