



Standard Practice for Analysis of In-Service Lubricants Using a Particular Five- Part (Dielectric Permittivity, Time-Resolved Dielectric Permittivity with Switching Magnetic Fields, Laser Particle Counter, Microscopic Debris Analysis, and Orbital Viscometer) Integrated Tester¹

This standard is issued under the fixed designation D7416; 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 practice covers procedures for analysis of in-service lubricant samples using a particular five-part (dielectric permittivity, time-resolved dielectric permittivity with switching magnetic fields, laser particle counter, microscopic debris analysis, and orbital viscometer) integrated tester to assess machine wear, lubrication system contamination, and lubricant dielectric permittivity and viscosity. Analyzed results trigger recommended follow-on actions which might include conducting more precise standard measurements at a laboratory. Wear status, contamination status, and lubricant dielectric permittivity and viscosity status are derived quantitatively from multiple parameters measured.

1.2 This practice is suitable for testing incoming and in-service lubricating oils in viscosity grades 32 mm²/s at 40°C to 680 mm²/s at 40°C having petroleum or synthetic base stock. This practice is intended to be used for testing in-service lubricant samples collected from pumps, electric motors, compressors, turbines, engines, transmissions, gearboxes, crushers, pulverizers, presses, hydraulics and similar machinery applications. This practice addresses operation and standardization to ensure repeatable results.

1.3 This practice is not intended for use with crude oils.

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

1.5 *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 practice 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. Referenced Documents

2.1 ASTM Standards:²

D341 Test Method for Viscosity-Temperature Charts for Liquid Petroleum Products

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

D924 Test Method for Dissipation Factor (or Power Factor) and Relative Permittivity (Dielectric Constant) of Electrical Insulating Liquids

D1298 Test Method for Density, Relative Density (Specific Gravity), or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products

E617 Specification for Laboratory Weights and Precision Mass Standards

E1951 Guide for Calibrating Reticles and Light Microscope Magnifications

D6300 Practice for Determination of Precision and Bias Data for Use in Test Methods for Petroleum Products and Lubricants

2.2 ISO Standards:³

ISO 11171 Hydraulic fluid power—Calibration of automatic particle counters for liquids

3. Terminology

3.1 Definitions:

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 International Organization for Standardization (ISO), 1, ch. de la Voie-Creuse, Case postale 56, CH-1211, Geneva 20, Switzerland, <http://www.iso.ch>.

3.1.1 *integrated tester*, *n*—automated, or semi-automated stand alone instrument utilizing multiple technologies to provide diagnostic recommendations (on-site or in-line) for condition monitoring of in-service lubricants.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *chemistry index (Chem Index)*, *n*—parameter computed from dielectric permittivity increase compared to new oil. The value is equal to dielectric difference multiplied by 100.

3.2.2 *chemistry status (Chem Status)*, *n*—diagnostic severity ranking having 0 to 100 score based on the highest alarm indication of dielectric permittivity and viscosity measurements.

3.2.3 *counts ≥ 4* , *n*—sensor 3 measured particle counts per mL for particles $\geq 4\ \mu\text{m}$.

3.2.4 *counts ≥ 6* , *n*—sensor 3 measured particle counts per mL for particles $\geq 6\ \mu\text{m}$.

3.2.5 *counts ≥ 10* , *n*—sensor 3 measured particle counts per mL for particles $\geq 10\ \mu\text{m}$.

3.2.6 *counts ≥ 14* , *n*—sensor 3 measured particle counts per mL for particles $\geq 14\ \mu\text{m}$.

3.2.7 *counts ≥ 18* , *n*—sensor 3 measured particle counts per mL for particles $\geq 18\ \mu\text{m}$.

3.2.8 *counts ≥ 22* —sensor 3 measured particle counts per mL for particles $\geq 22\ \mu\text{m}$.

3.2.9 *counts ≥ 26* —sensor 3 measured particle counts per mL for particles $\geq 26\ \mu\text{m}$.

3.2.10 *counts ≥ 32* —sensor 3 measured particle counts per mL for particles $\geq 32\ \mu\text{m}$.

3.2.11 *counts ≥ 38* —sensor 3 measured particle counts per mL for particles $\geq 38\ \mu\text{m}$.

3.2.12 *contaminant status (Cont Status)*, *n*—diagnostic severity ranking having 0 to 100 score based on the highest alarm indication of all contamination related parameters.

3.2.13 *dual-screen patch maker*, *n*—apparatus with screens to support individual (most often) or stacked (occasionally for size segregation) filter patches used to extract solid particles from in-service lubricating fluid as the fluid is evacuated from sensor 2 test chamber. This item is often referred to simply as “patch maker.”

3.2.14 *ferrous index (Fe Index)*, *n*—ferrous density type parameter measuring relative concentration and size of magnetically responsive iron particles $\geq 5\ \mu\text{m}$ collected on a dielectric permittivity sensor.

3.2.15 *large contaminant droplet (LCont D)*, *n*—indication reporting sensor 2 detects presence of free-water drops in oil.

3.2.16 *large contaminant ferrous (LCont Fe)*, *n*—indication reporting sensor 2 detects presence of very large ferrous-metal particles in oil, which are often the kind produced by abrasive wear mechanisms.

3.2.17 *large contaminant non-ferrous (LCont NF)*, *n*—indication reporting sensor 2 detects presence of very large non-ferrous-metal particles in oil, which are often the kind produced by abrasive wear mechanisms.

3.2.18 *orbital viscometer*, *n*—four-pole, magnetically driven, orbital viscometer.

3.2.19 *new oil*, *n*—sample of as-purchased new oil as supplied by a manufacturer for use to measure baseline reference values for the following reference oil properties: dielectric permittivity, specific gravity (Test Method **D1298**), kinematic viscosity at 40°C (Test Method **D445**), kinematic viscosity at 100°C (Test Method **D445**), and sensor 2 water factor.

3.2.20 *particular five-part integrated tester*, *n*—integrated tester including these five parts:^{4,5} sensor 1 (dielectric permittivity sensor), sensor 2 (time-resolved dielectric permittivity sensor with switching magnetic fields),^{5,6} sensor 3 (laser particle counter),^{5,7} dual-screen patch maker (initial step in microscopic debris analysis),^{5,8} and orbital viscometer.^{5,9}

3.2.21 *particle count ppm by volume $< 6\ \mu\text{m}$ (PC Vol $< 6\ \mu\text{m}$)*, *n*—volume of particulate debris detected using a laser particle counter in size range $\geq 4\ \mu\text{m}$ and $< 6\ \mu\text{m}$ compared to volume of oil $\times 10^{-6}$.

3.2.22 *particle count ppm by volume $\geq 6\ \mu\text{m}$ and $< 14\ \mu\text{m}$ (PC Vol 6-14 μm)*, *n*—volume of particulate debris detected using a laser particle counter in size range $\geq 4\ \mu\text{m}$ and $< 6\ \mu\text{m}$ compared to volume of oil $\times 10^{-6}$.

3.2.23 *particle count ppm by volume $\geq 14\ \mu\text{m}$ (PC Vol $\geq 14\ \mu\text{m}$)*, *n*—volume of particulate debris detected using a laser particle counter in size range $\geq 14\ \mu\text{m}$ compared to volume of oil $\times 10^{-6}$.

3.2.24 *particle count ppm by volume total (PC Vol Total)*, *n*—volume of all particulate debris detected using a laser particle counter in size range $\geq 4\ \mu\text{m}$ compared to volume of oil $\times 10^{-6}$.

3.2.25 *Sensor 1*, *n*—dielectric permittivity sensor having oil-filled cavity between central oscillating electrode and grounded concentric-shell.

3.2.26 *Sensor 2*, *n*—concentric-electrical-trace-type time-resolved dielectric permittivity sensor using a ceramic fiber filled printed circuit board and including pair of coaxial, switching electromagnets proximate to the underside of the surface supporting the concentric electrical traces.

3.2.27 *Sensor 2 water factor*, *n*—proportional measure of time-resolved-dielectric permittivity per 1% emulsified water-in-oil.

3.2.28 *Sensor 3*, *n*—light-blocking-type (also called light-extinction-type) laser particle counter sensor.

⁴ The analyzer is described in and covered by the following U.S. Patents: 5,262,732; 5,394,739; 5,604,441; 5,614,830; 5,656,767; 5,674,401; 5,817,928; 6,064,480; 6,418,799; 6,582,661; 7,027,959; and 7,065,454. The sole source of supply of the apparatus known to the committee at this time is Machinery Health Management, Emerson Process Management, 835 Innovation Drive, Knoxville, TN 37932.

⁵ 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 time-resolved dielectric sensor with switching electromagnets is described in and covered by U.S. Patent 5,604,441.

⁷ Sensor 3 uses methods described in and covered by U.S. Patents 6,064,480 and 7,065,454.

⁸ The patch maker with dual screens is described in and covered by U.S. Patent 6,418,799.

⁹ The orbital viscometer is described in and covered by U.S. Patent 5,394,739. The sole source of supply of the apparatus known to the committee at this time is Machinery Health Management, Emerson Process Management, 835 Innovation Drive, Knoxville, TN 37932.

TABLE 1 Oil and Solvent Solubility^A

NOTE—Y=Yes, N=No

Oil Class	Dielectric	Original Lamp Oil or Kerosine	Ultra Pure Lamp Oil	Original Lamp Oil + Fluid B	Toluene	Hexane	Fluid A	Fluid B	Fluid C	
Mineral Oil	2.1–2.4	Y	Y	Y	Y	Y	Y	Y	N	Most industrial lubricants Synthetic Hydrocarbon Diester Polyol Ester + Polyalkylene Glycol Polyalkylene Glycol Phosphate Ester
PAO	2.1– 2.4	Y	Y	Y	Y	Y	Y	Y	N	
Diester	3.4– 4.3	Y	Y	Y	Y	Y	Y	Y	Y	
POE + PAG	4.6– 4.8	Y	Y	Y	Y	Y	Y	Y	Y	
PAG	6.6–7.3	N	N	N	Y	Y	N	N	N	
PhE	6.0–7.1	sometimes	N	Y	Y	Y	N	Y	Y	

^A (Warning—Both Toluene and Isopropyl Alcohol have flash points below room temperature. They require an explosion proof vacuum pump.)

3.2.29 *system debris, n*—calculated volume of debris in entire oil compartment (PC Vol Total multiplied by volume of oil compartment).

3.2.30 *orbital viscosity at 25°C (Visc 25C)*—orbital viscometer viscosity measurement reported as absolute viscosity (mPa × s at 25°C).

3.2.31 *orbital viscosity at 40°C (Visc 40C)*—orbital viscometer viscosity measurement reported as kinematic viscosity (mm²/s) at 40°C.

3.2.32 *percent change in viscosity at 40°C (Visc%Chng)*—parameter comparing Visc 40C between new in-service oil.

3.2.33 *wear debris analysis classification (WDA classification)*—microscopic debris analysis classification method that closely identifies particulate debris from an oil sample.

3.2.34 *wear debris analysis severity (WDA severity)*—score-type parameter or alarming system assigned by an analyst that reflects a qualitative assessment of risk to machine health as evidenced by microscopic viewing of collected contamination and wear debris.

3.2.35 *wear status*—diagnostic severity ranking having 0 to 100 score based on the highest alarm indication of all wear related parameters.

4. Summary of Practice

4.1 *Measurements Made*—The particular five-part integrated tester sequentially measures viscosity, dielectric permittivity, water-in-oil, ferrous debris, particle count and distribution, and microscopic wear debris analysis for in-service oil samples.

4.1.1 Absolute viscosity is measured based on speed of an orbiting steel ball forced by controlled magnetic fields. Temperature of fluid under test is also measured.

4.1.2 Dielectric permittivity is measured using a concentric-shell-type capacitive sensor.

4.1.3 Water-in-oil and ferrous debris are each measured using time-resolved dielectric sensor and are differentiated by using a switching dual-coil electromagnet.

4.1.4 Particle counting is measured using a laser particle counter gated to detect and count individual particles at eight size ranges.

4.1.5 Microscopic wear debris analysis is performed after collecting solids on a filter patch and placing the filter patch under an optical microscope.

4.2 *Computer Application Software*—A computer application software program guides the test sequence and provides analysis, diagnostic determination, data storage, and reporting.

5. Significance and Use

5.1 *In-plant Oil Analysis*—The particular five-part integrated tester practice is primarily used by plant maintenance personnel desiring to perform on-site analysis of as-received and in-service lubricating oils.

5.2 *Detect Common Lubrication Problems*—The software application interprets data from integration of multiple sensing technologies to detect common lubrication problems from inadvertent mixing of dissimilar lubricant viscosity grades and from particulate or moisture contamination. The redundant views of ferrous particulates (sensor 2), all particulates larger than 4-µm (sensor 3), and all solid particulates larger than filter patch pore size (patch maker) provides screening for oil wetted mechanical system failure mechanisms from incipient to catastrophic stages.

5.3 *Supported by Off-Site Lab Analysis*—The particular five-part integrated tester is normally used in conjunction with an off-site laboratory when exploring the particular nature of an alarming oil sample. An off-site laboratory should be consulted for appropriate additional tests.

6. Interferences

6.1 *Wrong Solvent Selection*—The particular five-part integrated tester testing almost always requires the use of dilution with a solvent that is soluble with the in-service lubricant being tested. All petroleum-based and most synthetic lubricants dissolve very well in kerosine or lamp oil, so this is most often used. However, certain synthetics remain immiscible in these solvents. See 8.3 and Table 1. It is therefore very important to verify solubility of synthetic-based lubricants being tested with the diluents and cleaning solvents being used. To do this, add a 50:50 mixture of solvent and sample in a bottle, shake vigorously, and allow settling for 1 min. Layered fluids or emulsion are signs of insolubility. This is likely to cause erroneous measurements using sensors 2 and 3.

6.2 *Improper Sampling Techniques*—Interferences can be produced by improper sampling techniques. Practice D4177 should be followed. Samples collected from cold, not operating machinery are not likely to properly represent contaminants and wear debris since these settle when the system is not hot



FIG. 1 Example of Layout for a Particular Five-Part Integrated Tester

and flowing. Interferences may be produced by contaminated bottles, uncapped new bottles, and incorrectly labeled sample bottles.

6.3 Particle Count Interferences—Sensor 3 is a light obscuration type laser particle counter and is therefore subject to interferences from air bubbles, dark fluids, and emulsified water. Follow manufacturer recommended procedures to avoid these interferences. Interference caused by false counts from air bubbles may be caused by inadequate degassing with the 30 mL syringe in an inverted position. For very dark in-service lubricants, when sensor 3 gives an alert that oil sample may be too dark, retest with additional dilution. Avoid false counts from emulsified water-in-oil by using the water masking step in 8.4.

6.4 Coincidence Errors—Sensor 3 laser particle counter will experience abnormally high coincidence errors when it attempts to count more than 20 000 particles per mL. Coincidence errors result when more one particle is in the laser window at one time. When two small particles are coincidentally in the laser window, they are effectively counted like they are a single particle having double cross-section. This artificially lowers measured counts at small sizes and inflates the counts at larger sizes. Coincidence errors >10% are not acceptable. Avoid coincidence by performing second dilution prior to sensor 3 testing. For example, if you dilute 4-to-1, then you can expect to detect less than $5 \times 20\,000$ or 100 000 particles per mL.

7. Apparatus

7.1 Bench Top Setup—The particular five-part integrated tester^{5,10} with example arrangement shown in Fig. 1.

7.2 Core Analyzer—The core analyzer as shown on right in Fig. 1 incorporates sensor 1 (dielectric permittivity sensor), sensor 2 (time-resolved-dielectric-permittivity measurement with switching electromagnet), sensor 3 (laser particle counting sensor), and a patch maker as outlined in the following

paragraphs. The core analyzer connects directly to the balance, orbital viscometer, and computer application. In addition, it creates filter patches for wear debris analysis (WDA) classification and WDA severity determination.

7.2.1 Sensor 1 includes a concentric-shell-type-capacitor dielectric permittivity measurement sensor. Test fluid is injected into a port and pushed up around the annular space between a central metal electrode and the electrically grounded outer metal shell. By filling the cavity from the bottom, air is displaced as the fluid rises to fill the entire volume of the sensor. It takes approximately 8 mL of test fluid to completely fill the tubing and sensor. Dielectric permittivity measurement is made by measuring capacitance of the calibrated sensor. The concentric-shell-type-capacitor dielectric permittivity is calibrated to within $\pm 1\%$ of known values for standardization fluids A, B, and C.

7.2.2 Sensor 2 includes (1) a concentric-trace-type-capacitor dielectric permittivity measurement sensor mounted above (2) a dual-coil electromagnet suitable for introducing switching magnetic fields through the sensing surface.

7.2.2.1 Sensor 2 performs a time-resolved-dielectric-permittivity measurement involving 360 separate measurements taken at 0.5-s intervals covering a total elapsed test time of 3 min. During the first 2 min, the switching electromagnet produces alternating axial-field and then radial-field patterns which orient and re-orient ferrous particles on the sensor surface at 0.5-s intervals, synchronous with the sequential dielectric permittivity measurements. After 2 min, the field patterns are modified to draw ferrous debris entirely away from the concentric-trace-type capacitor sensor. The maximum ferrous concentration on the sensor is detected at the end of 240 measurements (2 min) and the maximum non-ferrous particle and water concentration is detected at the end of 360 measurements (3 min). After the test sequence is completed, the electromagnet coils are pulsed through a demagnetization or degaussing sequence so that ferrous debris are easily flushed away during cleaning.

7.2.2.2 The concentric-trace-type-capacitor dielectric permittivity measuring sensor is constructed using gold plated copper traces on a ceramic-fiber reinforced PTFE (polytetrafluoroethylene) substrate with a grounded metal backplane. The sensor is calibrated to within $\pm 2\%$ of standard values for standardization fluids A, B, and C.

7.2.2.3 The dual-coil electromagnet is used to collect and manipulate ferrous debris on the sensor surface. The dual-coil electromagnet produces -300 ± 30 G field strength on the sensor surface above the center-post when both coils are powered with fields adding, and it produces 70 ± 2 G field strength when the magnets when the polarity of the outer coil is reversed so it effectively overpowers the inner coil. These relative field strengths allow the analyzer to gather ferrous debris above the center-post, orient ferrous debris in axial and radial geometric pattern, and sweep ferrous debris off the sensor surface.

7.2.3 Sensor 3 includes a laser particle counting sensor mounted immediately below a Luer-tip injection port. Sample test fluid is diluted, homogenized, drawn into 30 mL syringe, degassed, and then injected via the Luer-tip port through the

¹⁰ The sole source of supply of the apparatus known to the committee at this time is A520010 Comprehensive Minilab, apparatus, and accessories, available from Machinery Health Management, Emerson Process Management, 835 Innovation Drive, Knoxville, TN 37932.

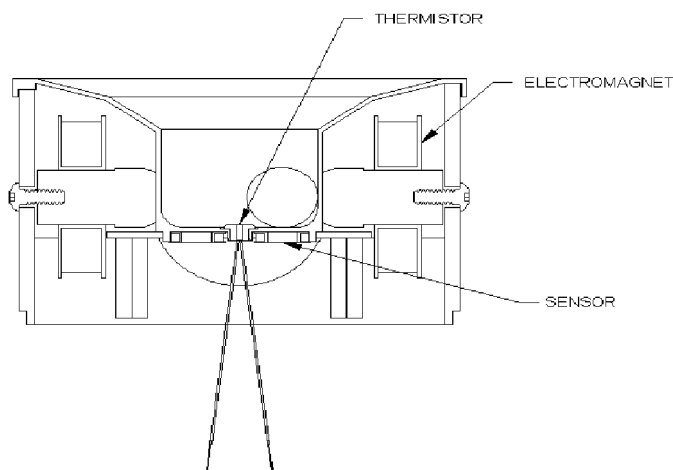


FIG. 2 Cross Section View of Orbital Viscometer

laser window. Flow rate through the particle counter is maintained at constant 50 mL/min using a stepper-motor controlled syringe pump. Sensor 3 is standardized using MTD standardization fluid in accordance with **ISO 11171**.

7.3 Orbital Viscometer—The orbital viscometer measures absolute viscosity, also called dynamic viscosity, at a particular temperature in units of centigrade (°C). A cross section of the orbital viscometer is shown in **Fig. 2**. The ~12 mL sample test cup is filled with test fluid and contains a steel ball that orbits around the base of the test cup under the influence of four sequentially powered electromagnets. Under the circular path of the ball are four ball-position sensors. Each time the ball passes over one sensor, the next electromagnet is energized, thereby the orbital speed of the ball is a function of test fluid absolute viscosity. A temperature sensor in the center-bottom of the sample test cup measures temperature. A fan inside the orbital viscometer constantly pulls air around the electromagnet coils to keep the orbital viscometer from self heating. The computer application software processes and controls the orbital viscometer, and uses the viscosity-temperature relationship as defined in Test Method **D341** to translate values from room temperature absolute viscosity (mPa × s) into 25°C absolute viscosity (mPa × s) and into 40°C kinematic viscosity (mm²/s). To do this, the application software uses laboratory measured values from its new oil database: specific gravity, kinematic viscosity at 40°C, and kinematic viscosity at 100°C.

NOTE 1—The viscosity measurement is performed at room temperature and converted to 40°C using calculation based on user supplied new oil information.

7.4 Microscope—A microscope enables the operator to perform analytical wear debris analysis of 25-mm diameter filter patches. As a minimum the microscope shall provide viewing stage with top-lighting and magnification from 30 to 230×. The microscope imaging system is standardized on the computer display (pixels per micron) using application software and a captured image from a NIST traceable length standard (see **12.5**).

7.5 Computer Application Software—The computer application software provides the functionality for the particular five-part integrated tester: an electronic user interface, hierar-

chical equipment database to store and analyze and manage data, step-by-step testing guide, imbedded logic for data interpretation, and automatic reporting tools.

7.6 Solvent Filtration Device—This device is used for dispensing the diluting and cleaning solvents. The device filters solvents from the pressurized dispenser by passing the fluid through a 0.8-μm (typical) filter patch.

8. Reagents and Materials

8.1 Disposable Luer-tip 10-mL Syringes—Syringes shall be supplied individually wrapped to prevent contamination. Syringes are all plastic with no rubber seals on plunger. Rubber materials may have material compatibility problems with some solvents. Syringes are not reusable.

8.2 Disposable Luer-tip 30-mL Syringes—Syringes shall be supplied individually wrapped to prevent contamination. Syringes are all plastic with no rubber seals on plunger. Rubber materials may have material compatibility problems with some solvents. Syringes are not reusable.

8.3 Cleaning and Diluting Solvent—Sensors 1, 2, and 3 are all cleaned using a solvent that is soluble with the base oils tested. See **Table 1** for oil and solvent solubility. Fluids A, B, and C are standardization fluids. See **12.2.1**.

8.4 Water Masking Solvent—Water masking eliminates false particle count interference from water droplets. Water masking solvents are selected to dissolve water-in-oil. See **Table 2** for water masking solvent materials and procedures.^{5,11}

8.5 Filter Patches—The WDA filter patches are 25-mm diameter circular patches with ~5 μm pore size or another pore size selected to retain desired size range of particulate debris of interest when performing analytical WDA.

8.6 Cleaning Swabs—These are used to clean sensor 2.

8.7 Paper Goods—Paper goods often used with the particular five-part integrated tester include label stock, printer paper, and tissue papers.

9. Hazards

9.1 Flammable Solvents—All particular five-part integrated tester setup and operations shall be approved by an on-site industrial hygiene specialist considering the particular situation. One should be particularly cautious if optional highly flammable solvents such as heptane, hexane, toluene, or isopropyl alcohol are used. Among other safety practices, be sure to keep the area well ventilated, and eliminate spark sources in the vicinity of highly flammable solvents. Those wishing to minimize flammability risks may prefer to use kerosine or lamp oil for diluents and cleaning solvents, and to use dipropylene glycol n-butyl ether for water masking.

10. Sampling and Test Specs and Units

10.1 Sample Bottles—The sample bottles, typically 100 mL in size, are used for testing. Occasionally, sample bottles should be tested using solvent to determine the particle count contribution from the bottles. It is important to use bottles that are significantly cleaner (at least two ISO codes) than the fluids

¹¹ This water masking method is described in and covered by U.S. Patent 6,064,480.

TABLE 2 Particle Counting Water Masking Options

Water Masking Solvent Mixture	Dipropylene Glycol n-Butyl Ether (fp 205°F) ^A	Toluene (fp 50°F) ^A	Isopropyl Alcohol (fp 54°F) ^A	Ratio (diluted sample to masking fluid) ^B	Application
Mask 1	100%			Add until clear	Particle count with water-in-oil
Mask 2		75%	25%	1:2	Particle count with water-in-oil
Mask 3		25%	75%	1:2	Particle counting diluents for water- glycol hydraulics

^A fp = flash point.

^B These solvent blends are added to the already diluted 1:1 sample after filling sensor 2 test chamber.

being sampled. This may require use of certified clean sample bottles when in-service lubricants are especially clean.

10.2 *Active Zone Sampling*—Obtain a representative sample of the in-service lubricating oil sample from an active zone of the machinery system where the oil is already warm, well agitated, and flowing

10.3 *Sampling Practices*—Practices D4057 or D4177 provide guidance for obtaining such representative samples.

11. Preparation of Apparatus

11.1 *Operator Instruction Manual*—Prepare the instrument for operation in accordance with the manufacturer's instructions.

11.2 *Clean Check*—Perform the following clean checks of sensor 1, sensor 2, sensor 3 and orbital viscometer at the start of each test day.

11.2.1 *Sensor 1 Clean Check*—Flush approximately 10 mL of solvent through sensor 1 test position. Evacuate with vacuum. Perform sensor 1 clean check. Pass if clean check test result is less than or equal to 1.20 dielectric units.

11.2.2 *Sensor 2 Clean Check*—Flush approximately 20 mL of solvent through sensor 2 test position. Evacuate with vacuum. Gently dry flat surface of sensor 2 with cotton swab. Perform sensor 2 clean check. Pass if clean check test result is less than or equal to 1.40 dielectric units.

11.2.3 *Sensor 3 Clean Check*—Perform particle count test using 100% solvent. Compare these results to most recent values for solvent standardization (12.3). Pass if particle counts $\geq 4 \mu\text{m}$ test result is within 25% of average solvent standardization value.

11.2.4 *Orbital Viscometer Clean Check*—Wipe, feel, and visually inspect cleanliness of the orbital viscometer test cup and steel ball.

11.3 *Database Setup*—Use the computer application software to create user database defining equipment and including measurement points. Each measurement point shall have appropriate alarm limit sets and new oil designation from an associated new oil database.

11.3.1 User database defines all equipment, analysis parameters, alarm limit sets, and includes links to an associated new oil database.

11.3.2 New oil database defines the following new oil properties corresponding to in-service lubricants being tested: dielectric permittivity, specific gravity (Test Method D1298), kinematic viscosity at 40°C (Test Method D445), kinematic viscosity at 100°C (Test Method D445), and sensor 2 water factor. Check for completeness of the new oil database, determine and add any missing property values as follows:

11.3.2.1 Dielectric permittivity value for new oil database entry is determined by testing a sample of neat new oil with sensor 1. Result is automatically stored in the new oil database.

11.3.2.2 Specific gravity and kinematic viscosity values for new oil database entries are determined by lab report results from sending samples of that new oil to a qualified laboratory for testing in accordance with Test Method D1298 and Test Method D445 respectively or in accordance with other relevant ASTM test methods. Results are manually entered into the new oil database.

11.3.2.3 Sensor 2 water factor value in a new oil database is a measure for rate of change in sensor 2 dielectric from the effects of moisture settling and adsorption per 1 % emulsified water-in-oil. This new oil database entry is determined by testing with sensor 2 using a prepared new oil sample.

(1) The prepared water factor test sample is a blended mixture of new oil with a known quantity of water. Typically this requires 1 drop of water into 50 ± 1 mL of new oil. However, other amounts of water, more or less, may be used if this amount does not generate a meaningful measured response from sensor 2 testing.

(2) In order to achieve a consistent, representative, homogeneous water-in-oil sample, it is important to blend water into new neat oil samples for at least 1 min. Then the blended sample shall age for at least 1 h before commencing testing.

(3) To calculate sensor 2 water factor, the computer application software instructs an operator to enter the actual number of water drops (0.5, 1, 2, 3, etc.) per 50 mL oil, and then to perform sensor 2 test in accordance with 13.3.2, 13.3.3, and 13.3.7. The computer application software uses the test results to calculate sensor 2 water factor calculation assuming that 1 drop water in ~50 mL oil is equivalent to ~741 ppm water-in-oil, 2 drops water in 50 mL oil is ~1082 ppm water-in-oil; 1 drop water in 100 mL oil is ~320 ppm, and so forth.

(4) Sensor 2 water factor is a measure of demulsibility characteristics for base oil plus additives. Formulated lubricating oils having good demulsibility, for example, good water-shedding, generally exhibit relatively high sensor 2 water factor values, typically >3 . Those with poor demulsibility generally exhibit relatively low sensor 2 water factor values, typically <2 .

11.4 *Vacuum System Check*—Verify that the vacuum system is functioning and retains at least 50 % initial vacuum for 30 s after the pump stops. To test if the vacuum system holds vacuum, place a hand or finger over each respective opening

for sensor 1, sensor 2, and degassing sensor 3 ports, and observe the pressure gauge needle after the vacuum pump is stopped.

12. Standardization and Verification

12.1 Standardization:

12.2 *Standardization Fluids*—The following control fluids are used for standardizing and verifying sensors 1, 2, and 3, and orbital viscometer in accordance with the equipment manufacturer's procedures using the computer application.

12.2.1 *Fluids A, B, and C*—These dielectric permittivity sensor 1 and sensor 2 are standardized using Fluids A, B, and C, which are standardization fluids sourced exclusively from the equipment manufacturer. Note the supplier's dielectric permittivity values marked on each bottle.

12.2.1.1 Fluid A is petroleum oil, Fluid B is vegetable oil, and Fluid C is phosphate ester oil.

12.2.1.2 The dielectric permittivity value for each batch of Fluids A, B, and C is measured by the equipment manufacturer using a "sensor 1" type instrument that was standardized using four reagent grade pure fluids having well characterized room temperature dielectric permittivity (Test Method **D924**): hexane (1.887), octamethylcyclotetrasiloxane (2.024), cyclohexane (2.390) and decyl alcohol (7.900). This practice allows the integrated tester user to handle common oils (Fluids A, B, and C) and avoid handling the relatively flammable or toxic pure fluids.

12.2.2 *Medium Test Dust (MTD) Standardization and Verification Fluid*—Sensor 3 is a laser particle counter, is standardized in accordance with **ISO 11171** using NIST traceable particulate concentrations of MTD. This particle counting fluid is also used for verification testing of sensor 3.

12.2.3 *Orbital Viscometer Standardization Fluids*—Standardization of the orbital viscometer is accomplished using eight ISO viscosity grade (VG) oils typically including: VG 14, VG 32, VG 68, VG 100, VG 80W90, VG 220, VG 460, and VG 680. Each fluid shall be supplied with laboratory measured viscosities at 40°C and 100°C (Test Method **D445**) and specific gravity (Test Method **D1298**). The viscosity grades for eight oils used to standardize the orbital viscometer may vary, but the range shall include at least one with viscosity grade above and another with viscosity grade below the range of in-service lubricants being tested. This is intended to avoid extrapolation errors.

12.3 *Solvent Standardization*—Whenever the source or dispensing mechanism for diluting and water-masking solvents changes, perform standardization to determine representative particle count values by performing three sequential tests on each fluid.

12.4 *Balance Calibration Mass*—A NIST traceable (Specification **E617**) mass is supplied with the balance to verify operation and, if necessary, to calibrate the balance used with the analyzer.

12.5 *Microscope Standardization*—The microscope system should be standardized following the equipment manufacturer's procedures in the application software upon initial setup and whenever one of these parts of the system setup is replaced: camera, computer image resolution, or microscope.

12.6 *Microscope Length Standard*—The NIST traceable microscope length-standard (Guide **E1951**) is used to calibrate the size represented by images displayed on the display for the computer application software. This is supplied with the WDA microscope.

12.7 *Verification*—Perform quarterly verification testing following the equipment manufacturer's procedures in the application software using standardization fluids (**12.2**).

13. Procedure

13.1 *Initial Setup*—Use application software to create a user database and an associated new oil database.

13.2 Orbital Viscometer Testing Procedure:

13.2.1 Viscosity test should be performed prior to the diluting step in preparation for sensor 2 and sensor 3 testing.

13.2.2 Visually inspect the viscometer test cup to see the test cup is clean and already contains the steel ball.

13.2.3 Pour sample lubricating oil into the test cup, filling to the top of the cylindrical shoulder. This is approximately 12 mL and does not have to be precise.

13.2.4 Use the software selection to run the viscosity test and at the completion of the test, save the result using the application software.

13.2.5 Evacuate the test cup using either a vacuum or syringe.

13.2.6 Extract the steel ball using a magnet. Immediately remove the ball from the magnet using a tissue, wipe the ball, and place the ball on the table. Wipe remaining fluid from the cup. Visually inspect the cup. Place the steel ball back in the cup.

13.3 Core Analyzer Testing Procedure:

13.3.1 *Test Using Sensor 1*—Open the wrapper on a brand new 10-mL Luer-tip syringe and fill the syringe with oil taken directly from the sample bottle. Be careful to avoid bringing air in with the sample. Secure the Luer-tip self-locking-tapered-tip into the sensor 1 port and follow instructions on computer display to complete the dielectric permittivity test. This takes about 10 s.

13.3.2 *Prepare to Test Using Sensor 2*—Follow the instructions on the computer display for tare weighing, diluting, and vigorously shaking the oil sample in its original bottle.

13.3.3 *Begin Test Using Sensor 2*—Add approximately 20 mL of the diluted sample into the sensor 2 port. Do this by filling to the top of the tapered portion of the sample chamber. The sensor 2 test will self-start.

13.3.4 *Perform Test 3*—During the 3-min interval while testing with sensor 2, continue to follow instructions on the computer display.

13.3.4.1 Open and fill a brand new 30-mL Luer-tip syringe with diluted sample. Secure the Luer-tip self-locking-tapered-tip into the sensor 3 degassing port, and degas the syringe. During degassing, the vacuum may produce a cloud of entrained air bubbles in the diluted sample volume. This cloud rises toward the vacuum port, leaving behind transparent fluid in the syringe. Do not be concerned if the vacuum causes bubble stream(s) to be produced from air leaking around the syringe plunger seal. It will dissipate quickly after vacuum is released.

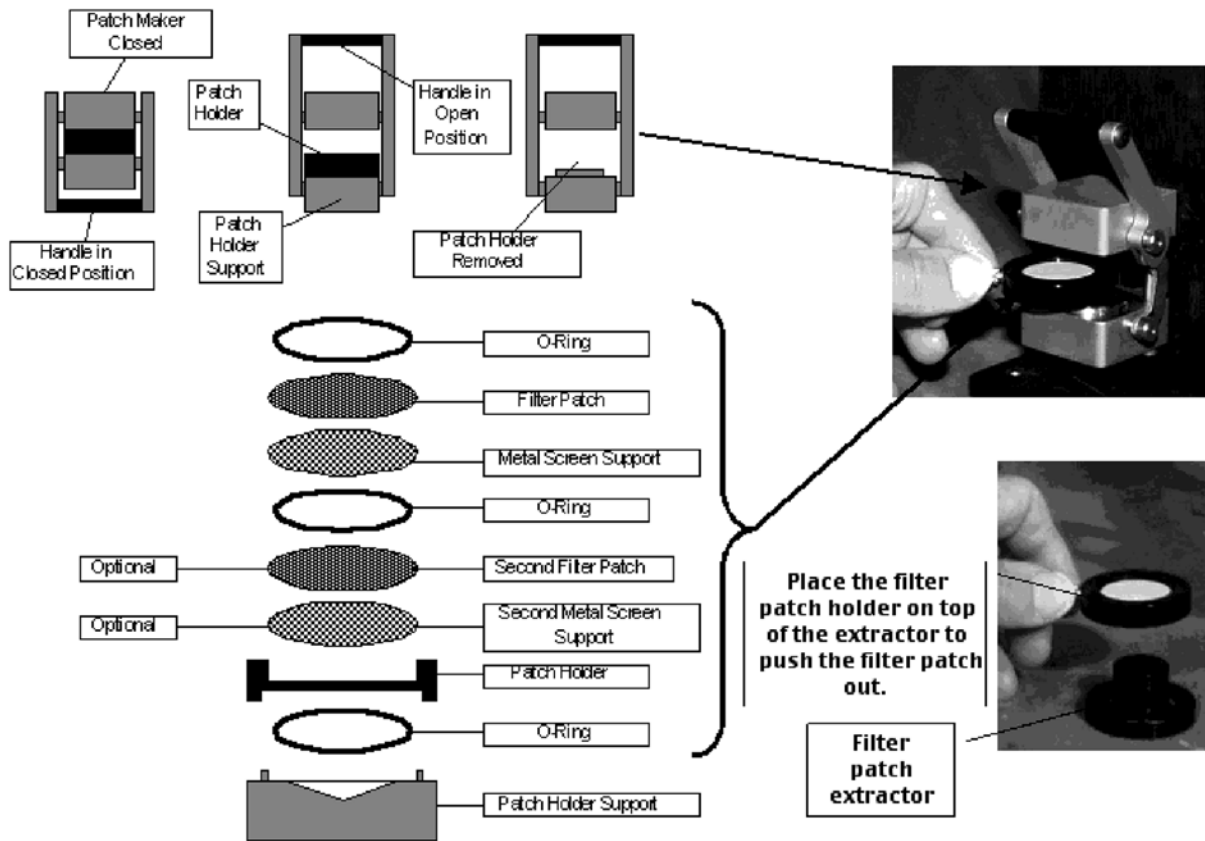


FIG. 3 Wear Debris Patch Maker Filter Assembly Diagram

13.3.4.2 When degassing is complete, expel sufficient gas from the Luer-tip end of the syringe so less than 5 mL measured by syringe markings remain in the syringe. Keep in mind the syringe plunger stops at the 5 mL mark on the syringe during testing. Immediately invert the 30-mL syringe and insert the tip into the sensor 3 test port.

13.3.4.3 Observe that there are not any visible air bubbles near the downward pointing Luer-tip end of the syringe. Bubble(s) should be dislodged by thumping the syringe with a fingertip or similar soft object.

13.3.4.4 Then press the button to begin sensor 3 test sequence.

13.3.5 *Clean Up Sensor 1 Test Port*—After completing test with sensor 3, the lighted LED and message on the computer display will guide the user to clean up sensor 1 test port. Remove the 10-mL syringe from sensor 1 port, then evacuate and clean sensor 1 by flushing with solvent. Perform sensor 1 clean check to verify that sensor 1 is adequately clean.

13.3.6 *Prepare for WDA*—Install a filter patch and close the filter patch holder. Fig. 3 displays a diagrammatic representation of the filter patch maker filter assembly.

13.3.7 *Clean Up Sensor 2 Test Port*—When instructed to do so by the lighted LED and message on the computer display, evacuate sensor 2, flush with solvent, and wipe sensor 2 carefully using a cotton swab. Perform sensor 2 clean check to verify that Sensor 2 is adequately clean.

13.3.8 *Transfer WDA Filter Patch*—Remove the filter patch from the filter patch holder, visually inspect, and place the

patch in a tray or on a card marked with sample identification. This tray or card will hold the patch for microscopic WDA classification and WDA severity determination.

13.4 *Wear Debris Analysis Procedure:*

13.4.1 Receive the filter patch at some point following the analyzer test sequence.

13.4.2 Open the WDA tab on the computer application software, and verify that the correct measurement point and sample are shown on the computer display.

13.4.3 Place the filter patch on the microscope stage and begin inspecting the patch using relatively low magnification (30 to 50×) in order to get a general indication as to what is on the filter patch and where points of interest are located.

13.5 Use the image capture capability to document interesting portions of the filter patch.

13.6 Perform qualitative particle classification. Use the computer application software WDA tab and identify up to five groups of particulate debris on the filter patch and document the characteristics using the inspection grid on the computer display. For each particulate debris group, be sure to identify the WDA classification. For wear debris, be sure to also identify the WDA severity. Experienced users inspect the filter patch under microscope magnification and immediately annotate findings on the computer application software WDA table. New users employ the look-up atlas in the computer application software, finding similar examples in the software image atlas.

TABLE 3 Quantitative Parameters Measured, Calculated, and Reported Using the Five-Part Integrated Tester

Parameter	Description
Wear Status	Diagnostic severity ranking (score) based on the highest alarm indication of all wear related parameters
Cont Status	Diagnostic severity ranking (score) based on the highest alarm indication of all contamination related parameters
Chem Status	Diagnostic severity ranking (score) based on the highest alarm indication of dielectric permittivity and viscosity measurements
Chem Index	Dielectric permittivity increase compared to new oil $\times 100$
Dielectric	Dielectric permittivity of in-service oil
Fe Index	Relative measure of magnetically responsive iron particles $\geq 5 \mu\text{m}$ collected on a dielectric permittivity sensor
% Water	Percent water-in-oil
LCont D	Indication of free-water-in-oil
LCont Fe	Indication of detected large ferrous-metal particles in oil ($\geq 60 \mu\text{m}$)
LCont NF	Indication of detected large non-ferrous-metal particles in oil ($\geq 60 \mu\text{m}$)
Visc 40C	Viscosity at 40°C in mm^2/s
Visc%Chng	Percent change in viscosity at 40°C in mm^2/s from reference to in-service oil
Visc 25C	Viscosity at 25°C in $\text{mPa} \times \text{s}$
PC Vol < 6 μ	PPM by volume for particles $\geq 4 \mu\text{m}$ and $< 6 \mu\text{m}$
PC Vol 6-14 μ	PPM by volume for particles $\geq 6 \mu\text{m}$ and $< 14 \mu\text{m}$
PC Vol $\geq 14\mu$	PPM by volume for particles $\geq 14 \mu\text{m}$
PC Vol Total	PPM by volume for particles $\geq 4 \mu\text{m}$
System Debris	Calculated volume of debris in entire oil compartment (PPM $\times$ compartment)
Cnts ≥ 4	Particle counts $\geq 4 \mu\text{m}$
Cnts ≥ 6	Particle counts $\geq 6 \mu\text{m}$
Cnts ≥ 10	Particle counts $\geq 10 \mu\text{m}$
Cnts ≥ 14	Particle counts $\geq 14 \mu\text{m}$
Cnts ≥ 18	Particle counts $\geq 18 \mu\text{m}$
Cnts ≥ 22	Particle counts $\geq 22 \mu\text{m}$
Cnts ≥ 26	Particle counts $\geq 26 \mu\text{m}$
Cnts ≥ 32	Particle counts $\geq 32 \mu\text{m}$
Cnts ≥ 38	Particle counts $\geq 38 \mu\text{m}$
ISO ≥ 4	ISO 11171 code for particles $\geq 4 \mu\text{m}$
ISO ≥ 6	ISO 11171 code for particles $\geq 6 \mu\text{m}$
ISO ≥ 14	ISO 11171 code for particles $\geq 14 \mu\text{m}$
WDA Severity	Analyst determination of wear severity based on microscopic WDA

14. Interpretation of Results

14.1 Diagnostic logic in the computer application software assists the user with interpretation of results for particular five-part integrated tester tests and user observations to determine machine wear condition, lubrication system contamination condition, and lubricant dielectric permittivity and viscosity condition. Table 3 summarizes the list of quantitative parameters measured.

14.2 Machine wear condition determination is based on automatic measurements made using a time-resolved-dielectric sensor with switching magnetic field, and as needed on visual microscopic WDA. Measured parameters contributing to machine wear condition include ferrous index, large ferrous indication, large nonferrous indication, WDA classification, and WDA severity.

14.3 Lubrication system contamination condition determination is based on automatic measurements made using a time-resolved-dielectric sensor, on a laser particle counter sensor. Measured parameters contributing to lubrication system contamination include: water-in-oil (percent), count of particles in eight different size ranges, volumetric parts-per-million (ppm v/v versus μm) distribution for particulate debris, particulate debris between 4 and 6 μm (ppm v/v), particulate debris from 6 to 14 μm (ppm v/v), particulate debris $\geq 14 \mu\text{m}$ (ppm v/v), total systems debris (mL), and visual or microscopic debris classification.

14.4 Lubricant dielectric permittivity and viscosity condition determination is based on automatic measurements made using a viscosity sensor and a dielectric permittivity sensor.

Measured parameters include viscosity, and dielectric permittivity (also called *dielectric constant* or simply *dielectric*).

15. Report

15.1 The computer application software provides the user dozens of reporting options.

15.2 As a minimum the report should include at least the overall machine wear status, lubricant system contamination status, and lubricant dielectric permittivity and viscosity status.

15.2.1 The user will usually report the numerical values for measurements made during the using a particular five-part integrated tester. When a particular value crosses and alarm condition, that value is normally flagged with a corresponding color indication.

15.2.2 Reports often include photographs from microscopic WDA.

15.2.3 Reports often include computer-generated observations, recommended actions, and analyst's notes. Example reports are given in Figs. 4-6.

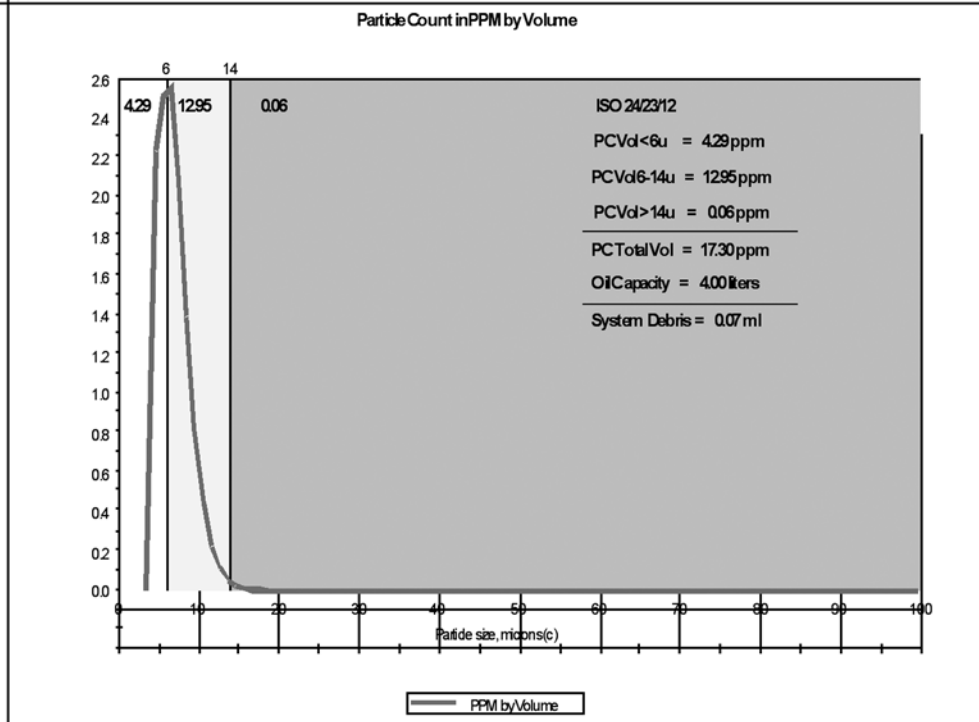
16. Precision and Bias ¹²

16.1 *Precision*—Values for repeatability and Reproducibility precision with 95% confidence is reported in Table 4. Reproducibility values were computed for three status values, including wear status, contamination status, and chemistry

¹² Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D02-1646.

Particle Count in PPM by Volume

Database:	Example.rbm	Point:	P2-Honda Engine
Area:	A17-ASTMR repeatability Study	Sample:	78911
Equipment:	E1-R repeatability study	Sample Date:	7/10/2006 10:51:05 AM

[illegible]

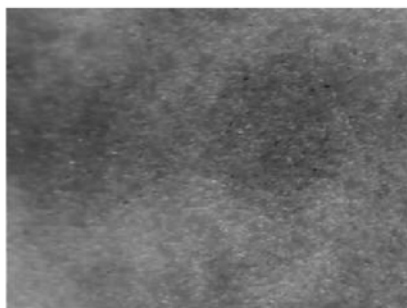
Wear Debris Analysis Summary for Sample 78911

Grp	Conc.	Avg Size	Max Size	Aspect	Shape	Color	Texture	Composition	Classificatio	WDA Seven
1	Few	Fine<6u	Med 14-40u	1:2	Chunks	Black	Oxidized	Ferrous	Adhesive W	Normal
2	Moderate	Fine<6u	Large 40-100	1:2	Thermal	Stew	Amorphous	Organic	Lubric Debris	Normal

FIG. 4 Example of “Particle Count PPM by Volume” Report

WDA & Diagnosis Summary

Database:	Example.rbm	Meas. Point:	P2- Honda Engine
Area:	A17 - ASTM R repeatability Study	Sample No:	78911
Equipment:	E1- R repeatability study	Sample Date:	7/10/06 10:51 am



100 x image

Mild adhesion and organic lube degradation on filter patch.

This sample revealed a few distribution of black chunks in fine<6u size from adhesive wear, moderate distribution of straw thermal in fine<6u size from lube degrade.

Gp	Conc.	Avg Size	Max Size	Aspect	Shape	Color	Texture	Composition	Classification	WDA Severe
1	Few	Fine<6u	Med 14-40u	1:2	Chunks	Black	Oxidized	Ferrous	Adhesive We	Normal
2	Moderate	Fine<6u	Large 40-100	1:2	Thermal	Straw	Amorphous	Organic	Lube Degrad	Normal

FIG. 5 Example of “WDA & Diagnosis Summary” Report

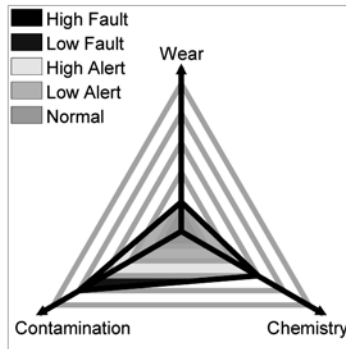
Sample - PPM

Database:	Example.rbm	Point:	P2 - Honda Engine
Area:	A17 - ASTM Repeatability Study	Sample:	78911
Equipment:	E1 - Repeatability study	Sample Date:	7/10/2006 10:51:05 AM

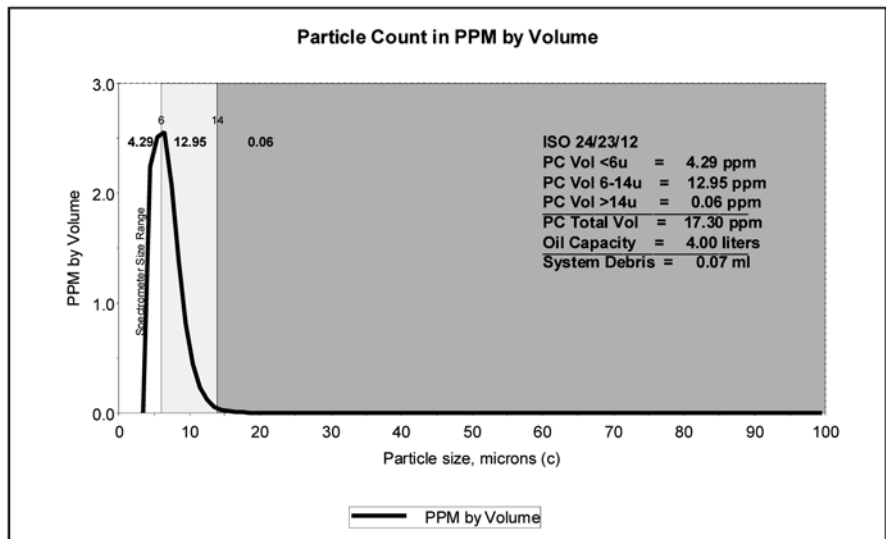
Wear	0
Ferrous Idx	0.0
LCont Ferrous	0
LCont NonFe	0
WDA Severity	1

Contamination	56
Contam Idx	0.0
% Water	0.0000
LCont Droplet	0
PC Vol Total	17.30
Cnts >4	119,549
Cnts >6	67,929
Cnts >10	1,185
Cnts >14	32.4
Cnts >18	1.6
Cnts >22	.2
Cnts >26	0.0
Cnts >32	0.0
Cnts >38	0.0
Cnts >56	0.0
Cnts >70	0.0
ISO >4	24
ISO >6	29
ISO >14	12
NAS 1638	15
PC Vol < 6u	4.29
PC Vol 6-14u	12.95
PC Vol >14u	.06
System Debris	.07

Chemistry	28
Chemical Idx	6.2
Dielectric	2.265
DV Visc 40C	47
DV Visc %Chng	-4
DV Visc 25C	83



Ref Oil Pennzoil - Multi-Grade - 5W30



Observations
Particle counting indicates contaminati

Actions
Filter lubricant
Check for flow surge
Check for source of contamination

Estimated water content = 0.0000% assuming 3.849 water factor.
This sample revealed a few distribution of black chunks in fine<6u size from adhesive wear, moderate distribution of straw thermal in fine<6u size from lube degrade.

TABLE 4 Calculated Repeatability and Reproducibility Precision

Parameter	Units	Repeatability	Reproducibility
Wear Status	score	29.00	10.6 ^A
Cont Status	score	11.27	12.3
Chem Status	score	2.87	6.4
Chem Index	permittivity $\times$ 100	6.83	8.5
Dielectric	permittivity	0.06	0.085
Fe Index	index value	10.22	23.3
Visc 40C	mm ² /s @ 40°C	8.40	15.8
Visc%Chng	%	2.77	14.7
Visc 25C	mPa $\times$ s @ 25°C	2.38	16.0
PC Vol < 6 μ m	ppm by volume	2.26 ^B	0.92
PC Vol 6-14 μ m	ppm by volume	5.97 ^C	2.17
PC Vol $\geq$ 14 μ m	ppm by volume	1.49	5.80
PC Vol Total	ppm by volume	5.55	7.91
ISO $\geq$ 4	code value	<1	1.7
ISO $\geq$ 6	code value	<1	1.2
ISO $\geq$ 14	code value	1.54	2.0
counts (<15 cts/mL)	counts/mL		12.5
counts (>15 cts/mL)	% of median value		99.9%

^A Wear status Reproducibility R = 10.6 is suspect because this value is inconsistent with earlier repeatability value r = 29.00. Wear status is a score type unit derived by applying alarm limits to ferrous index measurements for which R = 23.2 and r = 10.22. Wear status measurement scores in do not have sufficient degrees of freedom as required by Practice D6300 due to the effect of lowest ferrous index alarm limit = 6 which truncated wear status scores = 0 for 9 of 13 samples.

^B PC vol <6 repeatability r = 2.26 is suspect. A statistical worst case value for this should be r = 0.92 based on seven labs testing thirteen samples in the interlaboratory study. Note that repeatability r = 2.26 is from a different study (population) than Reproducibility R = 0.92 in this table.

^C PC vol 6-14 repeatability r = 5.97 is suspect. A statistical worst case value for this should be r = 2.17 based on seven labs testing thirteen samples in the interlaboratory study. Note that repeatability r = 5.97 is from a different study (population) than Reproducibility R = 0.92 in this table.

status. In addition, each supporting parameter includes associated reproducibility values.

16.2 Sample Populations—Reproducibility precision is calculated based on thirteen samples tested at seven different field laboratories. Repeatability precision is calculated based on two samples tested thirty times each at a single laboratory and on a third sample tested ten times each by two different laboratories. Each field laboratory was operated by the assigned operator who normally operated the respective five-part integrated tester setup.

17. Keywords

17.1 condition monitoring; contamination status; debris analysis; dielectric permittivity; ferrous density; ferrous index; in-service oil; machinery health; particle counting; ppm distribution; time-resolved-dielectric; viscosity; wear status

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