Designation: D7889 – 21

Standard Test Method for Field Determination of In-Service Fluid Properties Using IR Spectroscopy[1]

This standard is issued under the fixed designation D7889; 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 describes the use of a grating spectrometer to analyze properties of an in-service fluid sample which are indicative of the status of that fluid and related machinery.

1.2 This test method provides a means for the assessment of in-service fluid properties using infrared spectroscopy. It describes a methodology for sampling, performing analysis, and providing key in-service fluid properties with a self-contained unit that is meant for field use. It provides analysis of in-service fluids at any stage of their useful life, including newly utilized fluid.

1.3 In particular, these key in-service fluid properties include oxidation, nitration, sulfation, soot, and antiwear additives. They are applicable for hydrocarbon type (API Group I-IV) fluids from machinery lubricants, including reciprocating engine oils, turbine oils, hydraulic oils, and gear 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.

1.4.1 *Exception*—The unit for wavenumbers is in cm^{-1}.

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

1.6 *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]

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

D7412 Test Method for Condition Monitoring of Phosphate Antiwear Additives in In-Service Petroleum and Hydrocarbon Based Lubricants by Trend Analysis Using Fourier Transform Infrared (FT-IR) Spectrometry

D7414 Test Method for Condition Monitoring of Oxidation in In-Service Petroleum and Hydrocarbon Based Lubricants by Trend Analysis Using Fourier Transform Infrared (FT-IR) Spectrometry

D7415 Test Method for Condition Monitoring of Sulfate By-Products in In-Service Petroleum and Hydrocarbon Based Lubricants by Trend Analysis Using Fourier Transform Infrared (FT-IR) Spectrometry

D7418 Practice for Set-Up and Operation of Fourier Transform Infrared (FT-IR) Spectrometers for In-Service Oil Condition Monitoring

D7624 Test Method for Condition Monitoring of Nitration in In-Service Petroleum and Hydrocarbon-Based Lubricants by Trend Analysis Using Fourier Transform Infrared (FT-IR) Spectrometry

D7669 Guide for Practical Lubricant Condition Data Trend Analysis

D7720 Guide for Statistically Evaluating Measurand Alarm Limits when Using Oil Analysis to Monitor Equipment and Oil for Fitness and Contamination

D7844 Test Method for Condition Monitoring of Soot in In-Service Lubricants by Trend Analysis using Fourier Transform Infrared (FT-IR) Spectrometry

E131 Terminology Relating to Molecular Spectroscopy

E168 Practices for General Techniques of Infrared Quantitative Analysis

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E691 Practice for Conducting an Interlaboratory Study to

[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 D02.96.03 on FTIR Testing Practices and Techniques Related to 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.

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

Determine the Precision of a Test Method
E932 Practice for Describing and Measuring Performance of Dispersive Infrared Spectrometers
E1655 Practices for Infrared Multivariate Quantitative Analysis
E2412 Practice for Condition Monitoring of In-Service Lubricants by Trend Analysis Using Fourier Transform Infrared (FT-IR) Spectrometry
E2617 Practice for Validation of Empirically Derived Multivariate Calibrations

3. Terminology

3.1 For definitions of terms relating to infrared spectroscopy used in this test method, refer to Terminology E131. For definition of terms related to infrared-based in-service fluid condition monitoring, refer to Practice D7418.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *absorbance units (AU), n*—units of measurement of the raw absorbance spectrum which is obtained using the definition described in Section 8 (Theory for a Single Compound Analysis) of Practice E168, which is not normalized for pathlength.

3.2.2 *cell background, n*—a single-beam spectrum that is obtained on a clean, empty wipe-clean transmission cell.

3.2.3 *mid-infrared grating spectrometer, n*—a spectrometer which operates in the mid-infrared spectral range, between at least 960 cm^{-1} and 3040 cm^{-1} and creates an infrared spectrum by means of a reflective diffraction grating.

3.2.3.1 *Discussion*—Such a grating spectrometer may be of any of a variety of designs and optical configurations. Example designs include monochrometer-type systems wherein the grating is rotated to a single point infrared detector, or array-type systems which utilize an infrared detector array at the output and a fixed grating. Example optical configurations include Rowland-Circle and Czerny-Turner systems. Typical infrared detectors are uncooled thermal detectors such as thermopile or pyroelectric-based sensors.

3.2.4 *reporting units, n*—specifies the reporting units of the fluid analysis property.

3.2.5 *self-contained field apparatus, n*—a mid-infrared grating spectrometer which is of the form factor to allow it to operate as an independent device suitable for field use.

3.2.6 *wipe-clean transmission cell, n*—an infrared transmission cell which is specifically tailored for field use.

3.2.6.1 *Discussion*—In particular, the cell may be utilized and cleaned with a towel or rag and without the use of reagents or chemicals of any sort, making it convenient for use as a field device. Such transmission cells may accomplish this using mechanisms for quick open/close of the cell such as by means of a mechanical lever, demountable screw or press fit, or magnetic coupling. To correct for any cell fringing effects, the cell utilizes a wedged design both on the interior faces and exterior faces of the cell windows: The cell windows themselves are wedged at an angle of less than 0.5 degrees. The spacing between the two windows is wedged at an angle of approximately 0.013 degrees. The cell is designed to be nominally 100 µm in pathlength, with ZnSe windows.

4. Summary of Test Method

4.1 This test method utilizes a self-contained field apparatus to provide detailed information concerning the condition status of in-service fluids. In particular, it provides readings of oxidation, antiwear additive, sulfation, nitration and soot levels in hydrocarbon type (API Group I-IV) fluids.

4.1.1 An absorbance spectrum of the sample under test is obtained. For the background spectrum, the cell background is used.

4.1.1.1 Test Methods have been developed for Fourier-transform infrared (FTIR) devices using absorbance spectra obtained using Practice D7418. Particular Test Methods developed for in-service monitoring of hydrocarbon type (API Group I-IV) fluids include those established for Oxidation (Test Method D7414, Procedure A), Antiwear Additive (Test Method D7412, Procedure A), Sulfation (Test Method D7415, Procedure A), Nitration (Test Method D7624, Procedure A), and Soot (Test Method D7844, Procedure A). These test methods have served to establish the signature infrared spectroscopic behavior associated with key in-service monitoring properties. This test method provides property values based on the examination of each property's signature infrared spectroscopic behavior. The essence of this test method is to capture the underlying chemical trends associated with each property for in-service fluid analysis using a self-contained field apparatus and coupled wipe-clean transmission cell.

4.1.2 From the infrared absorbance spectrum obtained with the self-contained field apparatus, properties of the in-service fluid are calculated. In particular, those properties in Table 1 are calculated by the device and presented to the user on the display. Additional properties using infrared calibration methods may be calculated and displayed depending on the particular fluid being analyzed and availability of calibrations for that fluid.

4.1.2.1 Infrared spectra generated by the described instrument type can be used to provide a further set of properties of interest to in-service fluid analysis of hydrocarbon type (API Group I-IV) fluids. Such properties must be calibrated to the particular fluid blend, and may be generated using ASTM guidelines which govern the creation of such calibrations. Such calibrations may be built from either standard regression methods as described in Practice E168 or as described in Practice E2412. Further, they may also be multivariate calibrations, described in Practice E1655 and Practice E2617. Example properties include Acid Number (AN), Base Number (BN), water contamination, ethylene glycol, fluid mixture content, and antioxidant depletion. It should be noted that, due to the fact that these calibrations are sample-specific, this test method does not provide a prescription for calculating such properties.

TABLE 1 In-Service Lubricant Properties Reported by the Test Method

Property	Reporting Units
Oxidation	Abs/0.1 mm
Antiwear Additive	Abs/0.1 mm
Sulfation	Abs/0.1 mm
Nitration	Abs/cm
Soot	Abs/cm