Designation: ~~D7844 – 12~~ D7844 – 18

Standard Test Method for Condition Monitoring of Soot in In-Service Lubricants by Trend Analysis using Fourier Transform Infrared (FT-IR) Spectrometry[1]

This standard is issued under the fixed designation D7844; 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 pertains to field-based monitoring soot in diesel crankcase engine oils as well as in other types of engine oils where soot may contaminate the lubricant as a result of a blow-by due to incomplete combustion of in-service fuels.

1.2 This test method uses FT-IR spectroscopy for monitoring of soot build-up in in-service lubricants as a result of normal machinery operation. Soot levels in engine oils rise as soot particles contaminate the oil as a result of exhaust gas recirculation or a blow-by. This test method is designed as a fast, simple spectroscopic check for monitoring of soot in in-service lubricants with the objective of helping diagnose the operational condition of the machine based on measuring the level of soot in the oil.

1.3 Acquisition of FT-IR spectral data for measuring soot in in-service oil and lubricant samples is described in Standard Practice D7418. In this test method, measurement and data interpretation parameters for soot using both direct trend analysis and differential (spectral subtraction) trend analysis are presented.

1.4 This test method is based on trending of spectral changes associated with soot in in-service lubricants. For direct trend analysis, values are recorded directly from absorbance spectra and reported in units of 100*absorbance per 0.1 mm pathlength. For differential trend analysis, values are recorded from the differential spectra (spectrum obtained by subtraction of the spectrum of the reference oil from that of the in-service oil) and reported in units of 100*absorbance per 0.1 mm pathlength (or equivalently absorbance units per centimeter). Warnings or alarm limits can be set on the basis of a fixed maximum value for a single measurement or, alternatively, can be based on a rate of change of the response measured (**1**).[2] In either case, such maintenance action limits should be determined through statistical analysis, history of the same or similar equipment, round robin tests or other methods in conjunction with the correlation of soot levels to equipment performance.

NOTE 1—It is not the intent of this test method to establish or recommend normal, cautionary, warning, or alert limits for any machinery. Such limits should be established in conjunction with advice and guidance from the machinery manufacturer and maintenance group.

1.5 This test method is primarily for petroleum/hydrocarbon based lubricants but is also applicable for ester based oils, including polyol esters or phosphate esters.

1.6 This method is intended as a field test only, and should be treated as such. Critical applications should use laboratory based methods, such as Thermal Gravimetric (TGA) analysis described in Standard Method D5967, Annex A4.

1.7 *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.*

1.8 *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:*[3]

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

[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] The boldface numbers in parentheses refer to the list of references at the end of this standard.

[3] 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**

D2896 Test Method for Base Number of Petroleum Products by Potentiometric Perchloric Acid Titration
D5185 Test Method for Multielement Determination of Used and Unused Lubricating Oils and Base Oils by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)
D5967 Test Method for Evaluation of Diesel Engine Oils in T-8 Diesel Engine
D6304 Test Method for Determination of Water in Petroleum Products, Lubricating Oils, and Additives by Coulometric Karl Fischer Titration
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
E131 Terminology Relating to Molecular Spectroscopy
E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods
E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method
E2412 Practice for Condition Monitoring of In-Service Lubricants by Trend Analysis Using Fourier Transform Infrared (FT-IR) Spectrometry

3. Terminology

3.1 *Definitions*—For definitions of terms relating to infrared spectroscopy used in this test method, refer to Terminology E131.

3.2 *Definitions*—For definition of terms related to in-service oil condition monitoring, refer to Practice D7418.

3.3 *Definitions of Terms Specific to This Standard:*

3.3.1 *machinery health, n*—a qualitative expression of the operational status of a machine sub-component, component or entire machine, used to communicate maintenance and operational recommendations or requirements in order to continue operation, schedule maintenance, or take immediate maintenance action.

4. Summary of Test Method

4.1 This test method uses FT-IR spectrometry to monitor soot levels in in-service lubricants. The test method is meant to serve as a field-based method to provide an indicator of soot level. The FT-IR spectra of in-service oil samples are collected according to the protocol described in Standard Practice D7418 and the levels of soot are measured using the absorption intensity measurement described herein. The values obtained for the sample of the in-service oil are compared to the value for a sample of new reference oil using either direct trend analysis or differential trend analysis approaches.

5. Significance and Use

5.1 An increase in soot material can lead to increased wear, filter plugging and viscosity, which is usually a consideration for diesel engines, although it may also be an indicator of carburetor or injector problems in other fuel systems. Monitoring of soot is therefore an important parameter in determining overall machinery health and should be considered in conjunction with data from other tests such as atomic emission (AE) and atomic absorption (AA) spectroscopy for wear metal analysis (Test Method D5185), physical property tests (Test Methods D445, D6304 and D2896), and other FT-IR oil analysis methods for oxidation (Test Method D7414), sulfate by-products (Test Method D7415), nitration (Test Method D7624), additive depletion (Test Method D7412), and breakdown products and external contaminants (Practice E2412), which also assess elements of the oil's condition (**1-6**).

6. Interferences

6.1 High levels of water ~~(>5%)~~(>5 %) will interfere with the soot measurement in internal combustion engine crankcases. Other interferences include high levels of sludge or insolubles. These interferences will increase the measured soot values.

7. Apparatus

7.1 *Fourier Transform Infrared Spectrometer,* equipped with sample cell, filter and pumping system (optional) as specified in Standard Practice D7418.

7.2 *FT-IR Spectral Acquisition Parameters*—Set FT-IR spectral acquisition parameters according to instructions in Standard Practice D7418.