



Standard Test Method for Condition Monitoring of Soot in In-Service Lubricants by Trend Analysis using Fourier Transform Infrared (FT-IR) Spectrometry¹

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

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

2. Referenced Documents

2.1 *ASTM Standards*:³

- [D445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids \(and Calculation of Dynamic Viscosity\)](#)
- [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 \(ICP-AES\)](#)

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.

- [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
- [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%) 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](#).

8. Sampling

8.1 Obtain a sample of the in-service oil and the reference oil according to the protocol described in Standard Practice [D7418](#).

9. Preparation and Maintenance of Apparatus

9.1 Rinse, flush and clean the sample cell, lines and inlet filter according to instructions in Standard Practice [D7418](#).

9.2 Monitor cell pathlength as specified in Standard Practice [D7418](#).

10. Procedure and Calculation

10.1 Collect a background spectrum according to the procedure specified in Standard Practice [D7418](#).

10.2 Collect the spectrum of a reference oil sample according to the procedure specified in Standard Practice [D7418](#).

10.3 Collect the spectrum of an in-service oil sample according to the procedure specified in Standard Practice [D7418](#).

10.4 *Data Processing*—All data are processed to give direct trend analysis and differential trend analysis spectra normalized to 0.100 mm according to the procedure specified in Standard Practice [D7418](#).

10.5 *Calculation of Soot Value*—Soot has no specific frequency of absorption in the infrared spectrum, but causes a baseline shift and tilt in the spectrum due to light scattering. As such, soot is calculated from the normalized oil sample spectrum by directly measuring the absorbance intensity at

2000 cm^{-1} using no baseline. For differential trending, soot is directly measured from the difference spectrum also using the absorbance intensity at 2000 cm^{-1} . Fig. 1 illustrates the effect that soot (increasing from 1 to 5) has on the baseline of the spectra of diesel crankcase oils in-service and the measurement frequency used to monitor soot.

10.6 Reporting:

10.6.1 *Procedure A (Direct Trend Analysis)*—Values are reported in units of $100 \times \text{absorbance} / 0.100 \text{ mm}$; e.g., #4 in Fig. 1, the soot value equals 78.

10.6.2 *Procedure B (Differential Trend Analysis)*—Values are reported in units of $100 \times \text{absorbance} / 0.100 \text{ mm}$ (or equivalently absorbance units per centimeter).

10.7 *High Soot Values*—In samples where the value for soot is $>150 \text{ Abs./0.1 mm}$, which corresponds approximately to a soot content of $>3\%$, then it is necessary to dilute the oil sample with the new oil or mineral spirits prior to analysis to obtain reliable results. The measured value obtained from the diluted oil sample spectrum is then multiplied by the dilution factor to give the soot value for the original sample. Tests in which oils having soot contents of 2.9% and 10.8% were diluted in odorless mineral spirits (OMS) at a level of 0.5 g oil/16 mL OMS and their FTIR spectra were recorded at 15-min intervals showed no settling of soot over a period of 450 min.

10.8 *Sample Carryover*—To ensure the minimum amount of sample-to-sample cross-contamination or carryover, either a minimum volume of the subsequent sample or a solvent rinse is used to flush out the previous sample. The efficacy of the flushing protocol may be assessed by consecutively analyzing an oil having a low (or zero) soot level (L1, e.g., a fresh oil) and an in-service oil sample having a high soot level (H1) followed by a second run of the oil sample having a low soot level (L2) and then calculating the percent carryover (PC) as follows: $\text{PC} = [(L2 - L1)/H1] \times 100$, where L1, H1, and L2 are the values measured for soot (for calculating values for soot,

see 10.5) for the samples run in the indicated sequence. The calculated PC should be $<5\%$.

11. Reporting

11.1 *Trending*—Data shall be recorded and reported at selected time intervals during the lubricant's life. Ideally, soot values would be compared to that of the newly formulated oil and plotted over time to visualize the relative changes in soot and to determine when there needs to be an oil change, albeit other parameters may dictate this change earlier. Sampling and reporting time intervals for soot are based on the type of machinery and its previous history associated with this parameter.

11.2 *Statistical Analysis and Alarm Limits*—For statistical analysis and setting alarm limits, refer to Standard Practice E2412, Section A3, "Distribution Profiles and Statistical Analysis".

11.3 *Effects of Oil Formulation*—The compositions of various oil formulations can have an effect on the results reported for soot value, and values from two different oil formulations should not be compared. Results should be interpreted relative to values measured for unused oils of the same formulation or trended directly from the sample history.

12. Precision and Bias

12.1 *Precision*—The precision of the test method has not yet been determined by formal interlaboratory study. Preliminary examinations of repeatability have shown that the difference between repetitive results obtained by the same operator in a given laboratory applying the same test method with the same apparatus under constant operating conditions on identical test material within short intervals of time would in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in 20:

repeatability = 0.90 absorbance units/0.100 mm for Procedure A (direct trend analysis)

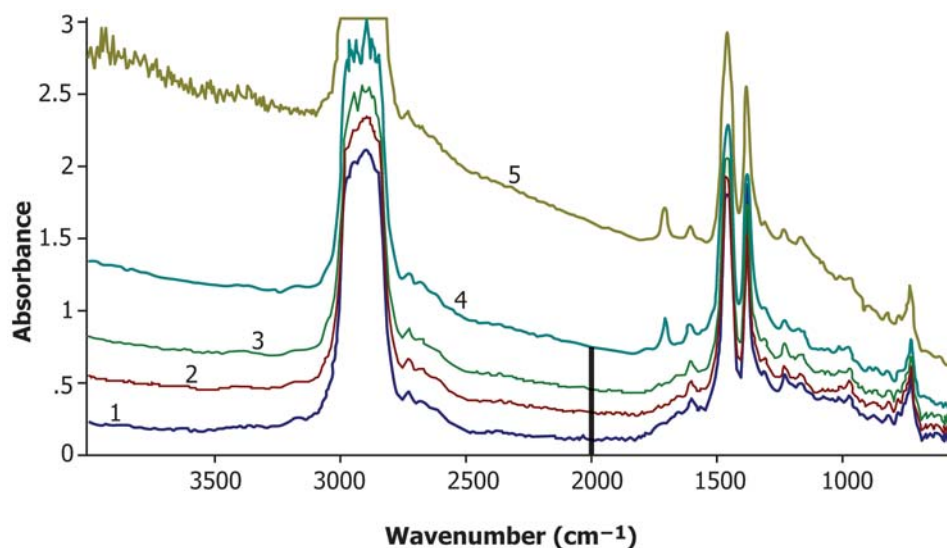


FIG. 1 Soot Measurements in Diesel Crankcase Oils

repeatability = 0.86 absorbance units/cm for Procedure B (differential trend analysis)

12.1.1 The reproducibility of this test method is being determined and will be available on or before May 1, 2018.

12.2 *Bias*—The procedures in this test method have no bias because the soot values can be defined only in the terms of the test method and no accepted reference method or value is available.

13. Keywords

13.1 condition monitoring; differential trend analysis; direct trend analysis; Fourier transform infrared; FT-IR; hydrocarbon based lubricants; infrared; in-service petroleum lubricants; IR; lubricants; oils; soot

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