



Designation: D7418 – 23

Standard Practice for Set-Up and Operation of Fourier Transform Infrared (FT-IR) Spectrometers for In-Service Oil Condition Monitoring¹

This standard is issued under the fixed designation D7418; 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.

INTRODUCTION

This practice describes the instrument set-up and operation parameters for using FT-IR spectrometers for in-service oil condition monitoring. The following parameters are typically monitored for petroleum and hydrocarbon based lubricants: water, soot, oxidation, nitration, phosphate antiwear additives, fuel dilution (gasoline or diesel), sulfate by-products and ethylene glycol. Measurement and data interpretation parameters are standardized to allow operators of different FT-IR spectrometers to obtain comparable results by employing the same techniques. Two approaches may be used to monitor in-service oil samples by FT-IR spectrometry: (1) direct trend analysis and (2) differential (spectral subtraction) trend analysis. The former involves measurements made directly on in-service oil samples, whereas the latter involves measurements obtained after the spectrum of a reference oil has been subtracted from the spectrum of the in-service oil being analyzed. Both of these approaches are described in this practice, and it is up to the user to determine which approach is more appropriate.

1. Scope*

1.1 This practice covers the instrument set-up and operation parameters for using FT-IR spectrometers for in-service oil condition monitoring for both direct trend analysis and differential trend analysis approaches.

1.2 This practice describes how to acquire the FT-IR spectrum of an in-service oil sample using a standard transmission cell and establishes maximum allowable spectral noise levels.

1.3 Measurement and integrated parameters for individual in-service oil condition monitoring components and parameters are not described in this practice and are described in their respective test methods.

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, health, and environmental 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, 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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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

D4175 Terminology Relating to Petroleum Products, Liquid Fuels, and Lubricants

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

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

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

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

- E131** Terminology Relating to Molecular Spectroscopy
E168 Practices for General Techniques of Infrared Quantitative Analysis
E1421 Practice for Describing and Measuring Performance of Fourier Transform Mid-Infrared (FT-MIR) Spectrometers: Level Zero and Level One Tests
E1866 Guide for Establishing Spectrophotometer Performance Tests

3. Terminology

3.1 Definitions:

3.1.1 For definitions of terms used in this standard, see Terminology **D4175**.

3.1.2 For definitions of terms relating to infrared spectroscopy used in this practice, refer to Terminology **E131**.

3.1.3 *Fourier transform infrared (FT-IR) spectrometry, n*—form of infrared spectrometry in which an interferogram is obtained; this interferogram is then subjected to a Fourier transform calculation to obtain an amplitude-wavenumber (or wavelength) spectrum.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *condition monitoring, n*—field of technical activity in which selected physical parameters associated with an operating machine are periodically or continuously sensed, measured and recorded for the interim purpose of reducing, analyzing, comparing and displaying the data and information so obtained and for the ultimate purpose of using interim result to support decisions related to the operation and maintenance of the machine. (**1**, **2**)³

3.2.2 *direct trend analysis, n*—monitoring of the level and rate of change over operating time of measured parameters (**2**, **3**) using the FT-IR spectrum of the in-service oil sample, directly, without any spectral data manipulation such as spectral subtraction.

3.2.3 *differential trend analysis, n*—monitoring of the level and rate of change over operating time of measured parameters using the FT-IR spectra of the in-service oil samples, following subtraction of the spectrum of the reference oil.

3.2.4 *in-service oil, n*—lubricating oil that is present in a machine that has been at operating temperature for at least one hour.

3.2.4.1 *Discussion*—Sampling an in-service oil after a short period of operation will allow for the measurement of a base point for trend analysis; the minimum sampling time should be at least one hour after oil change or topping-off.

3.2.5 *reference oil, n*—sample of a lubricating oil whose spectrum is subtracted from the spectrum of an in-service oil for differential trend analysis.

3.2.5.1 *Discussion*—The most commonly employed reference oil is a sample of the new oil. It should be noted, however, that the continued use of the same reference oil after any top-off of lubricant may lead to erroneous conclusions, unless the added lubricant is from the same lot and drum as the in-service oil. This possibility is averted if a sample of the

in-service oil is taken after a short period of operation following top-off of the lubricant (see 3.2.4.1) and is employed thereafter as the reference oil.

4. Significance and Use

4.1 This practice describes to the end user how to collect the FT-IR spectra of in-service oil samples for in-service oil condition monitoring. Various in-service oil condition monitoring parameters, such as oxidation, nitration, soot, water, ethylene glycol, fuel dilution, gasoline dilution, sulfate by-products and phosphate antiwear additives, can be measured by FT-IR spectroscopy (**4-7**). Changes in the values of these parameters over operating time can then be used to help diagnose the operational condition of various machinery and equipment and to indicate when an oil change should take place. This practice is intended to give a standardized configuration for FT-IR instrumentation and operating parameters employed in in-service oil condition monitoring in order to obtain comparable between-instrument and between-laboratory data.

5. Interferences

5.1 High levels of water contamination will interfere with measurements in the ranges 3600 cm^{-1} to 3000 cm^{-1} and 1690 cm^{-1} to 1575 cm^{-1} . This can affect values determined for oxidation by Test Method **D7414** and nitration by Test Method **D7624** as the baseline for both procedures are in this affected region. Any calibration models using this region may also be affected.

5.2 Soot directly affects the ability of the laser to penetrate the sample and will cause increasing sample baseline absorbance due to decreasing transmittance.

NOTE 1—Refer to Test Method **D7844** Fig. 1 for a demonstration of the effect of increasing soot concentration on sample transmittance.

5.2.1 It is recommended to use caution when interpreting measurements for all parameters in samples with calculated soot concentrations exceeding 2.5 Absorbance values.

5.3 High levels of ester-based, polyols, glycols, and alcohols interfere with measurements in the range 1260 cm^{-1} to 1000 cm^{-1} . This can affect sulfation and phosphate anti-wear measurements and any calibration models using this region.

5.4 Various additive packages containing carbonyl compounds (aldehydes, ketones, and esters) absorb in the range 1745 cm^{-1} to 1680 cm^{-1} . Their presence can affect oxidation and nitration measurements and any calibration models using this region.

5.4.1 Additive packages containing esters and carboxylic acids, such as some dispersants, viscosity index improvers, pour point depressants, and rust inhibitors, can give false positives for oxidation. In addition, oils mixed with any synthetic ester-based oil products will also give very high values for oxidation. In some oils the contributions from additive packages and synthetic ester-based oils may be so high that oxidation cannot be reliably measured.

5.4.2 Additive packages containing detergents (sulfonates, phenates and salicylate), dispersants, demulsifiers will interfere with the sulfate by-products measurement.

³ The boldface numbers in parentheses refer to a list of references at the end of this standard.