



Designation: **D7418–22** **D7418 – 23**

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

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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.*

1.6 *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.*

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

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
- 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

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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.

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

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.

5.5 It is recommended to trend the in-service oil against the new oil to help identify any interference and the influence on measurements. In some oils the contributions from additive packages and synthetic ester based oils may be so high that the desired parameter cannot be reliably measured.

5.6 When the baseline of a sample is raised (as occurs with high soot content) or the presence of an interferent creates an intense peak in the same region of the desired parameter, the influence of the interferent may be reduced through dilution of the sample as described in Section 11 and Appendix X3. In those cases, the result is corrected by the dilution factor and reported.

5.6.1 If differential analysis is required, the reference oil must also be diluted with the same dilution factor and material prior to analysis. This diluted reference is then used in the subsequent differential calculation.

6. Apparatus

6.1 *Fourier Transform Infrared (FT-IR) Spectrometer*—All FT-IR instruments suitable for use in this practice must be configured with a source, beamsplitter and detector suitable for spectral acquisition over the mid-infrared range of 4000 cm^{-1} to 550 cm^{-1} . FT-IR spectrometer's IR source and interferometer should be in a sealed compartment to prevent harmful, flammable, or explosive vapors from reaching the IR source and air-cooled source.

6.1.1 *Detectors*—The standard configuration of detectors include a room temperature deuterated triglycine sulfate (DTGS), Silicon (Si), indium gallium arsenide (InGaAs), indium antimonide (InSb), lithium tantalate (LiTaO₃), complementary metal-oxide semiconductor (CMOS) array/linear variable filters (LVF) or photoacoustic detectors.

NOTE 2—Photoconductive detectors such as mercury cadmium telluride (MCT) should not be used owing to inadequate linearity of the detector response.

6.1.2 *Beamsplitters*—Beamsplitters can include potassium bromide (KBr), germanium-coated potassium bromide (Ge/KBr), cesium iodide (CsI), or zinc selenide (ZnSe).

6.2 *Sample Cell*—The sample cell employed for in-service oil condition monitoring is a transmission cell with a fixed pathlength that can be inserted in the optical path of the FT-IR spectrometer. Cell window material and cell pathlength considerations are stated below.

6.2.1 *Cell Window Material*—ZnSe is commonly used as the window material for condition monitoring and is recommended because of its resistance to water. Sample cells constructed of materials other than ZnSe may be used; however, to address all the various methods associated with condition monitoring, the window material should transmit IR radiation over the range of 4000 cm⁻¹ to 550 cm⁻¹. KCl and KBr are common cell window materials that meet this requirement but these are water-soluble salts and should not be used if oil samples containing moisture are frequently run through the cell, as contact with water will cause the windows to fog and erode rapidly. In addition, Coates and Setti (8) have noted that oil nitration products can react with KCl and KBr windows, depositing compounds that are observed in the spectra of later samples. On the basis of this report, KCl and KBr windows should not be used with samples of gasoline or natural gas engine oils as well as other types of lubricants where nitration by-products may form due to the combustion process or other routes of nitration formation.

6.2.1.1 When ZnSe is used as the window material, the reflections of the infrared beam that occur at the inner faces of the windows cause fringes to be superimposed on the oil spectrum; these must be minimized using physical or computational techniques as presented in Appendix X1. Because KCl and KBr have lower refractive indices than ZnSe, the use of these window materials avoids observable fringes in the oil spectrum.

6.2.2 *Cell Pathlength*—The standard cell pathlength to be employed for in-service oil condition monitoring is 0.100 mm; however, in practical terms, pathlengths ranging from 0.080 mm up to 0.120 mm are suitable, with values outside this range leading to either poor sensitivity or non-linearity of detector response, respectively. The actual cell pathlength obtained can be determined from the interference fringes in the spectrum recorded with an empty cell or by recording the spectrum of a check fluid; details for calculating cell pathlength are presented in Appendix X2. The reporting units of the various in-service oil condition monitoring parameter test methods are based on a pathlength of 0.100 mm (see the respective test methods). Accordingly, all data must be normalized to a pathlength of 0.100 mm, either by multiplying all data points in the absorption spectra by a pathlength correction factor (spectral normalization) or by multiplying the results of the respective test methods by a pathlength correction factor (see 12.2). The normalization procedure is usually part of the software provided by instrument manufacturers.

6.2.2.1 *Discussion*—However, if sample dilution is employed (see Appendix X3), longer pathlengths may become suitable. For example, for dilution with odorless mineral spirits (OMS) in a 2:1 OMS:oil sample ratio, a pathlength of 0.200 mm has proven suitable.

NOTE 3—For purposes of interlaboratory comparison of results, spectral normalization should be performed.

6.3 *Filter (optional)*—The use of a particulate filter with a mesh size of 0.100 mm or less to trap any large particles present in the sample is strongly recommended to prevent cell clogging.

6.4 *Sample Pumping System (optional)*—A pumping system capable of transporting oil to be analyzed into the transmission cell and of emptying and flushing the cell with solvent between samples may be used instead of manual cell loading. Commercial vendors offer various pumping systems that may differ in the type of pump, tubing, and transmission cell. Depending on the sample handling system employed and the viscosity of the oils analyzed, a wash/rinsing solvent may be run between samples to minimize sample-to-sample carryover as well as keep the cell and inlet tubing clean; commercial vendors may recommend specific solvent rinse protocols.

6.4.1 *Hydrocarbon Leak Alarm*—When a sample pumping system is used, an independent flammable vapor sensor and alarm system is strongly recommended. The purpose of this alarm system is to alert the operator when a leak occurs in the tubing, connectors or transmission cell.

7. FT-IR Spectral Acquisition Parameters

7.1 The spectral acquisition parameters are specified below. Because the spectral resolution, data spacing, and apodization affect the FT-IR spectral band shapes, these specifications must be adhered to:

Spectral resolution: 4 cm⁻¹

Data spacing: 2 cm⁻¹

Apodization: Triangular

Scanning range: 4000 cm⁻¹ to 550 cm⁻¹

Spectral format: Absorbance as a function of wavenumber

7.2 The number of scans co-added and hence the scan time will depend on the desired spectral noise level (see Section 14), whereby an increase in scan time by a factor of N will decrease the level of noise by a factor of $N^{1/2}$.

8. Sampling

8.1 *Sample Acquisition*—The objective of sampling is to obtain a test specimen that is representative of the entire quantity. Thus, laboratory samples should be taken in accordance with the instructions in Practice D4057.

8.2 *Sample Preparation*—Filtering the sample using a filter described in 6.3 prior to loading the cell with the sample is highly recommended. An exception to this recommendation may be made when oil samples are diluted (see Appendix X3).

9. Preparation and Maintenance of Apparatus

9.1 *Rinsing, Washing and Check Solvents*—A variety of hydrophobic solvents may be used to clean the cell and rinse the lines between samples as well as serving as a check fluid to monitor pathlength. Typical solvents include hexanes, cyclohexane, heptane or odorless mineral spirits (OMS). Health and safety issues on using, storing, and disposing of check or cleaning/wash solvents will not be covered here. Local regulations and Material Safety Data Sheets (MSDS) should be consulted.

9.2 *Sample Cell and Inlet Filter*—The cell should be flushed with the designated rinse/wash solvent at the start and end of analytical runs to clean the cell. Immediately following flushing of the cell, an absorption spectrum of the empty cell (see 10.1.2.2) should be recorded to check for build-up of material on the cell windows. If an inlet filter is used, the filter shall also be checked for particle build-up and its effect on sample flow rate.

9.3 *Check Fluid and Pathlength Monitoring*—The purpose of a check fluid is to verify proper operation of the FT-IR spectrometer/transmission cell combination, as well as any associated sample introduction and cleaning hardware. It is recommended that an absorption spectrum of the check fluid be recorded when a new or re-assembled cell is initially used and archived to disk as a reference spectrum against which subsequent spectra of the check fluid may be compared. The spectrum of the check fluid may also be used to calculate the pathlength of the sample cell to normalize all data to 0.100 mm and to monitor changes in the cell pathlength over time, where significant changes may imply wear or contamination on the cell windows and should prompt remedial action. To serve as a check fluid, a solvent must have consistent spectral characteristics (lot-to-lot) and a measurable (on-scale) IR absorption band for cell pathlength calculation; for more details, see X2.2. One IR manufacturer uses heptane, another uses OMS, and other commercial products are available.^{4,5}

10. Procedure for Collecting FT-IR Spectra

10.1 *Background Collection*—Collect a single-beam background spectrum at the beginning of each run and frequently enough thereafter such that changes in atmospheric water vapor levels and other changing ambient conditions do not significantly affect the sample results (for example, every 30 min). Four methods may be used to collect single-beam background spectra: (1) collecting an air (open-beam) background spectrum, (2) collecting a cell background spectrum, (3) collecting an air (open-beam)

⁴ Conostan Division, Conoco Inc. 1000 South Pine, Ponca City, OK 74602-1267.

⁵ Thermal-Lube Inc., 255 avenue Labrosse, Pointe-Claire, QC H9R 1A3.

background spectrum and a cell reference spectrum, or (4) air background with corrected cell reference. The background spectrum shall be acquired using the operating parameters specified in 7.1.

NOTE 4—It should be noted that changes in atmospheric conditions, such as humidity and temperature, can change the background spectrum. The frequency of background checks shall be determined by the individual laboratory.

10.1.1 *Air Background*—Collect a single-beam background spectrum with no cell in the sample compartment.

10.1.2 *Cell Background*—Collect a single-beam cell background with the clean empty cell in the sample compartment.

10.1.2.1 To use an empty cell background, either physical or computational fringe reduction methods (see Appendix X1) must be employed so as to reduce the superimposition of fringes from the spectrum of the empty cell onto the sample absorption spectrum.

10.1.2.2 To verify that the cell is empty and clean, an absorption spectrum of the empty cell should be collected using a previously collected or archived single-beam air (open-beam) spectrum as the background spectrum. Measure the maximum peak height between 3000 cm⁻¹ and 2800 cm⁻¹ relative to a baseline at 2700 cm⁻¹. If this value is <0.2 absorbance units, then the cell is adequately clean for recording an empty cell background. This spectrum may also be used to verify that the fringe reduction technique employed meets the criterion of sample spectral peak-to-peak noise (see Section 14).

10.1.3 *Air Background with Cell Reference*—Collect a single-beam background spectrum with no cell in the sample compartment. Obtain a cell reference spectrum by collecting a single-beam empty cell background spectrum, according to the procedure outlined in 10.1.2, and ratioing it against the newly acquired air background spectrum to give the absorption spectrum of the empty cell. This absorption spectrum is then subtracted in a 1:1 ratio from the absorption spectra of the samples collected using an air background.

10.1.4 *Air Background with Corrected Cell Reference*—Collect a cell reference absorbance spectrum according to the procedure outlined in 10.1.3. If any visible interference fringing is present after fringe-reduction methods have been employed, smooth the spectrum by convolution with a triangular function having full width at half-height (that is, FWHH) of 33 cm⁻¹ and unit area, or by an equivalent procedure. To account for the increase in reflection losses seen with an empty cell, subtract 0.067 (in absorbance) from the spectrum. The resultant spectrum is then subtracted in a 1:1 ratio from the absorption spectra of the samples collected using an air background. This procedure is appropriate only for cells held perpendicular to the optical axis and having two uncoated parallel-faced ZnSe windows. Wedging of the sample volume by a small angle (<<1°) does not affect the validity of the procedure.

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10.2 *Reference Collection (Differential Trend Analysis Only)*—The reference oil sample should be shaken or agitated to ensure that a representative sample is taken from the bottle. Introduce the sample into the infrared transmission cell, either manually or using an automatic pumping system. Collect the absorption spectrum of the reference oil using the single-beam background spectrum collected as described in 10.1 and using the operating parameters specified in 7.1. If spectral normalization is being used (see 6.2.2), the absorption spectrum should subsequently be normalized (see 12.2) to a pathlength of 0.100 mm.

10.2.1 The reference oil sample may be diluted (see Appendix X3).

10.3 *Sample Collection*—The in-service oil samples to be analyzed should be shaken or agitated to ensure that a representative sample is taken from the bottle. Introduce each sample into the infrared transmission cell, either manually or using an automatic pumping system. Collect the absorption spectrum of each oil sample using the single-beam background spectrum collected as described in 10.1 and using the operating parameters specified in 7.1. If spectral normalization is being used (see 6.2.2), the absorption spectrum should subsequently be normalized (see 12.2) to a pathlength of 0.100 mm.

10.3.1 Peak area shall be reported as absorbance per pathlength: absorbance/0.100 mm.

10.3.2 And is calculated as per one of these two equivalent equations:

$$\int_{\text{firstpt}}^{\text{endpt}} \left(\frac{\text{peakslice}}{\text{freq}} \right) d(\text{freq}) * dv = \text{spectral area} \quad (1)$$

10.3.2.1 Alternatively:

$$\frac{\text{numfreq} * \left\{ \text{average}_{\text{start-endrange}} (Absorbance(v) - BL(v)) \right\}}{\text{spectral area}} = \quad (2)$$

10.3.2.2 Where:

$$\frac{\text{average absorbance}_{\text{freq.range}}}{\text{number of frequencies in area of frequency range}} = \quad (3)$$

10.3.3 *Frequency Width*—An x-axis length shall have the width of the frequency slices (dv) as 1.0 cm⁻¹ units.

10.3.3.1 For example, in Test Method **D7414**:

$$\text{Oxidation} = 130^*$$

$$\left\{ \text{average}_{1670 \rightarrow 1800} (Absorbance(v) - BL(v)) \right\} \left[\frac{Absorbance}{0.100\text{mm}} \right] \quad (4)$$

10.3.4 *Baseline Correction*—Peakslice are all baseline corrected as stated in 10.2.1 and shown in **Appendix X2**.

10.3.5 *Number Frequencies*—num freq is a count of the number of frequencies within the defined integration area. The number is unitless, since it is an x-axis frequency count. The frequency slice is 1.0 cm⁻¹ units wide.

10.3.6 *Average Slices*—Average is the mathematical average of the peak slices across the defines area, subtracted for BL (baseline absorption at the slice frequency location). The unit is absorbance. And when normalized against the pathlength the unit is Abs/0.1 mm.

10.4 *Cell Loading Check (optional)*—A mechanism to verify that the cell is fully loaded with oil (no air bubbles) is useful to avoid misleading data. The integrated absorbance value calculated for the CH region between 2754 cm⁻¹ and 3039 cm⁻¹ relative to that in an archived absorption spectrum for a cell that has been ascertained to be fully loaded (by careful visual inspection) provides a simple means by which to check cell loading. The integrated absorbance values used to ascertain that the cell is fully loaded shall be determined by individual laboratories.

NOTE 5—As a general guideline, the integrated absorbance after normalization to a pathlength of 0.100 mm (12.2) should be >2 for petroleum and ester-based oils.

10.4.1 *Peak Height*—Peak height is a special case (or subset) measurement of area where dv=1 and the number of slices = 1. The measurement frequency shall also be reported with the peak height value to help define where the peak height is measured.

$$\int_{p_1}^{p_2} \frac{\text{peakslice}}{\text{freq}} d(\text{freq}) * dv = \text{spectral height} \quad (5)$$

10.4.2 *Baseline Correction*—Where peakslice are all baseline corrected values as stated in 10.2.1 and shown in **Appendix X2**.

10.4.3 *Integration*—Where the integration is across one point and dv = 1. dv still has the unit of frequency.

10.4.4 *Normalization*—The spectral height is normalized to 0.1 mm pathlength. However, in an attempt to put both area and peak height with similar number ranges, the peak height result are multiplied by 100. Yielding peak height with units of Abs/0.1 mm * 100, or Abs/cm.

10.5 *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 before the next sample collection.

10.6 *Sample Carryover Efficacy Check (optional)*—The efficacy of the flushing protocol may be assessed by consecutively analyzing three in-service oil samples and comparing the concentration of the parameter of interest. Samples 1 and 3 shall have a low or zero concentration of the parameter (L1 and L2 respectively). Sample 2 shall have a high concentration (H1) of the same