



# Standard Practice for Set-Up and Operation of Fourier Transform Infrared (FT-IR) Spectrometers for In-Service Oil Condition Monitoring<sup>1</sup>

This standard is issued under the fixed designation D 7418; 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 the standard. The values given in parentheses are for information only.

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

### 2. Referenced Documents

2.1 *ASTM Standards:*<sup>2</sup>

**D 4057** Practice for Manual Sampling of Petroleum and Petroleum Products

**E 131** Terminology Relating to Molecular Spectroscopy

**E 168** Practices for General Techniques of Infrared Quantitative Analysis

**E 1421** Practice for Describing and Measuring Performance of Fourier Transform Mid-Infrared (FT-MIR) Spectrometers: Level Zero and Level One Tests

**E 1866** Guide for Establishing Spectrophotometer Performance Tests

**E 2412** Practice for Condition Monitoring of Used Lubricants by Trend Analysis Using Fourier Transform Infrared (FT-IR) Spectrometry

### 3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms relating to infrared spectroscopy used in this practice, refer to Terminology **E 131**.

<sup>1</sup> This practice is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.96 on In-Service Lubricant Testing and Condition Monitoring Services.

Current edition approved Dec. 1, 2007. Published February 2008.

<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

3.1.2 *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)<sup>3</sup>

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 (5-8), as described in Practice E 2412. 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. Apparatus

5.1 *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 to 550  $\text{cm}^{-1}$ . The standard configuration includes a room temperature deuterated triglycine sulfate (DTGS) detector, an air-cooled source, and a germanium-coated potassium bromide (Ge/KBr) beamsplitter, although a zinc selenide (ZnSe) beamsplitter may also be used. The 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.

NOTE 1—Photoconductive detectors such as mercury cadmium telluride (MCT) should not be used owing to inadequate linearity of the detector response.

5.2 *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.

5.2.1 *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  $\text{cm}^{-1}$  to 550  $\text{cm}^{-1}$ . 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 (4) 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.

5.2.1.1 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.

5.2.2 *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 up to 0.120 mm are suitable, with values outside this range leading to either poor sensitivity or non-linearity of detector response,

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

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 [10.2](#)). The normalization procedure is usually part of the software provided by instrument manufacturers.

**NOTE 2**—For purposes of interlaboratory comparison of results, spectral normalization should be performed.

**5.3 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.

**5.4 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.

**5.4.1 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.

## 6. FT-IR Spectral Acquisition Parameters

**6.1** 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<sup>-1</sup>

Data spacing: 2 cm<sup>-1</sup>

Apodization: Triangular

Scanning range: 4000 to 550 cm<sup>-1</sup>

Spectral format: Absorbance as a function of wavenumber

**6.2** The number of scans co-added and hence the scan time will depend on the desired spectral noise level (see [Section 12](#)), whereby an increase in scan time by a factor of *N* will decrease the level of noise by a factor of *N*<sup>1/2</sup>.

## 7. Sampling

**7.1 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 D 4057](#).

**7.2 Sample Preparation**—Filtering the sample using a filter described in [5.3](#) prior to loading the cell with the sample is highly recommended.

## 8. Preparation and Maintenance of Apparatus

**8.1 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.

**8.2 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 [9.1.2.2](#)) 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.

**8.3 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.<sup>4, 5</sup>

## 9. Procedure for Collecting FT-IR Spectra

**9.1 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). Three methods may be used to collect single-beam background spectra: (1) collecting an air (open-beam) background spectrum, (2) collecting a cell background spectrum, or (3) collecting an air (open-beam) background spectrum and a cell reference spectrum. The background spectrum shall be acquired using the operating parameters specified in [6.1](#).

**NOTE 3**—It should be noted that changes in atmospheric conditions, such as humidity and temperature, can change the background spectrum.

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

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