Designation: D7418 – 21

Standard Practice for Set-Up and Operation of Fourier Transform Infrared (FT-IR) Spectrometers for In-Service Oil Condition Monitoring[1]

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.*

1.6 *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

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:*

[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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[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**

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. 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 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.

5.1.1 *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 ($LiTaO_3$), complementary metal-oxide semiconductor (CMOS) array/linear variable filters (LVF) or photoacoustic detectors.

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

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

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 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.

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

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