



Designation: E2412 – 23

Standard Practice for Condition Monitoring of In-Service Lubricants by Trend Analysis Using Fourier Transform Infrared (FT-IR) Spectrometry¹

This standard is issued under the fixed designation E2412; 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 practice covers the use of FT-IR in monitoring additive depletion, contaminant buildup and base stock degradation in machinery lubricants, hydraulic fluids and other fluids used in normal machinery operation. Contaminants monitored include water, soot, ethylene glycol, fuels and incorrect oil. Oxidation, nitration and sulfonation of base stocks are monitored as evidence of degradation. The objective of this monitoring activity is to diagnose the operational condition of the machine based on fault conditions observed in the oil. Measurement and data interpretation parameters are presented to allow operators of different FT-IR spectrometers to compare results by employing the same techniques.

1.2 This practice is based on trending and distribution response analysis from mid-infrared absorption measurements. While calibration to generate physical concentration units may be possible, it is unnecessary or impractical in many cases. Warning or alarm limits (the point where maintenance action on a machine being monitored is recommended or required) can be determined through statistical analysis, history of the same or similar equipment, round robin tests or other methods in conjunction with correlation to equipment performance. These warning or alarm limits can be a fixed maximum or minimum value for comparison to a single measurement or can also be based on a rate of change of the response measured (1).² This practice describes distributions but does not preclude using rate-of-change warnings and alarms.

NOTE 1—It is not the intent of this practice 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 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 The boldface numbers in parentheses refer to the list of references at the end of this standard.

1.3 Spectra and distribution profiles presented herein are for illustrative purposes only and are not to be construed as representing or establishing lubricant or machinery guidelines.

1.4 This practice is designed as a fast, simple spectroscopic check for condition monitoring of in-service lubricants and can be used to assist in the determination of general machinery health through measurement of properties observable in the mid-infrared spectrum such as water, oil oxidation, and others as noted in 1.1. The infrared data generated by this practice is typically used in conjunction with other testing methods. For example, infrared spectroscopy cannot determine wear metal levels or any other type of elemental analysis. The practice as presented is not intended for the prediction of lubricant physical properties (for example, viscosity, total base number, total acid number, etc.). This practice is designed for monitoring in-service lubricants and can aid in the determination of general machinery health and is not designed for the analysis of lubricant composition, lubricant performance or additive package formulations.

1.5 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.6 *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.*

1.7 *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.*

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

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
- D4057** Practice for Manual Sampling of Petroleum and Petroleum Products
- D4175** Terminology Relating to Petroleum Products, Liquid Fuels, and Lubricants
- D5185** Test Method for Multielement Determination of Used and Unused Lubricating Oils and Base Oils by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)
- D6304** Test Method for Determination of Water in Petroleum Products, Lubricating Oils, and Additives by Coulometric Karl Fischer Titration
- 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
- E1655** Practices for Infrared Multivariate Quantitative Analysis

2.2 ISO Standard:⁴

- ISO 13372** Condition monitoring and diagnostics of machines—Vocabulary

3. Terminology

3.1 Definitions:

3.1.1 For definitions of terms used in this practice, refer to 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*—a form of infrared spectrometry in which an interferogram is obtained; this interferogram is then subjected to a Fourier transform to obtain an amplitude-wavenumber (or wavelength) spectrum. **E131**

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *condition monitoring, n*—a 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 (ISO 13372).

3.2.2 *in-service oil, n*—as applied in this practice, a lubricating oil that is present in a machine which has been at operating temperature for at least one hour.

3.2.2.1 *Discussion*—Sampling a in-service oil after at least one hour of operation will allow for the measurement of a base point for later trend analysis.

3.2.2.2 *Discussion*—Any subsequent addition of lubricant (for example, topping off) may change the trending baseline, which may lead to erroneous conclusions.

3.2.3 *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.

3.2.4 *new oil, n*—an oil taken from the original manufacturer's packaging, prior to being added to machinery.

3.2.5 *reference oil, n*—see *new oil*.

3.2.6 *trend analysis, n*—as applied in this practice, monitoring of the level and rate of change over operating time of measured parameters (**1**).

4. Summary of Practice

4.1 Periodic samples are acquired from the engine or machine being monitored. An infrared absorbance spectrum of the sample is acquired, typically covering the range of 4000 cm⁻¹ to 550 cm⁻¹, with sufficient signal-to-noise (S/N) ratio to measure absorbance areas of interest. Exact data acquisition parameters will vary depending on instrument manufacturer but most systems should be able to collect an absorbance spectrum adequate for most measurements in less than one minute. Features in the infrared spectrum indicative of the molecular level components of interest (**1, 2**) (that is, water, fuel, antifreeze, additive, degradation, and so forth) are measured and reported. Condition alerts and alarms can then be triggered according to both the level and the trends from the monitored system.

5. Significance and Use

5.1 Periodic sampling and analysis of lubricants have long been used as a means to determine overall machinery health. Atomic emission (AE) and atomic absorption (AA) spectroscopy are often employed for wear metal analysis (for example, Test Method **D5185**). A number of physical property tests complement wear metal analysis and are used to provide information on lubricant condition (for example, Test Methods **D445**, **D2896**, and **D6304**). Molecular analysis of lubricants and hydraulic fluids by FT-IR spectroscopy produces direct information on molecular species of interest, including additives, fluid breakdown products and external contaminants, and thus complements wear metal and other analyses used in a condition monitoring program (**1, 2-6**).

6. Apparatus

6.1 Required Components:

6.1.1 *Fourier Transform Infrared Spectrometer (FT-IR)*—Instrument is configured with a source, beamsplitter and

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.

⁴ Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, <http://www.ansi.org>.

detector to adequately cover the mid-infrared range of 4000 cm^{-1} to 550 cm^{-1} . Most work has been done on systems using a room temperature deuterated triglycine sulfate (DTGS) detector, air-cooled source and Germanium coating on Potassium Bromide (Ge/KBr) beamsplitter. Alternate source, beamsplitter and detector combinations covering this range are commercially available but have not been investigated for use in this practice. Other detectors may be suitable but should be used with caution. In particular, liquid nitrogen cooled Mercury Cadmium Telluride (MCT) detectors are known to exhibit significant nonlinearities.

6.1.2 Infrared Liquid Transmission Sampling Cell—Sampling cells can be constructed of zinc selenide (ZnSe), barium fluoride (BaF_2), potassium bromide (KBr), or other suitable window material, with a pathlength of 0.1 mm (100 μm), parallel ($<0.5^\circ$ variance) cell spacer. Acceptable pathlength ranges are from 0.080 mm to 0.120 mm. Outside this range, poor sensitivity or data nonlinearity can occur. For the data provided in this document, the cells used were ZnSe, NaCl, or KBr as the measurements ranged from 4000 cm^{-1} to 700 cm^{-1} . Some cell material information is given below.

Material	Comments	Transmission Range, cm^{-1}
ZnSe	see 6.1.2.1	4000 – 550
KBr	susceptible to water damage	4000 – 400
NaCl	susceptible to water damage	4000 – 650
BaF_2	ammonium salts can damage	4000 – 850
CaF_2	ammonium salts can damage	4000 – 1100

Results should be corrected to 0.100 mm pathlength to account for cell path variation and improve data comparison to other instruments using this practice.

6.1.2.1 Due to the large refractive index change when the infrared beam passes from air into the ZnSe windows, fringe reduction is necessary to provide consistent results. Fringe reduction can be achieved electronically, optically or mechanically for ZnSe cells. For further explanation, see [Appendix X1](#). Care should be taken in selecting window materials to ensure that the desired parameters can be measured within the transmission region of that material and compatibility with the specific application; for example, salt windows (KBr, NaCl, KCl) can be used and may not require fringe correction but are susceptible to damage from water contamination in the oil. Coates and Setti (3) have noted that oil nitration products can react with salt windows, depositing compounds that are observed in later samples.

6.1.3 Cell Flushing/Cleaning Solvent—The ideal solvent to flush the cell between samples to minimize carryover should have no significant absorption in the condition monitoring areas of interest and should dry quickly when air is pumped through the system. Typical wash solvents used for common petroleum and some synthetic lubricants are technical grade, light aliphatic hydrocarbons such as heptane or cyclohexane. Other solvents may be required for more specialized synthetic lubricants. Health and safety issues on using, storing, and disposing of these solvents will not be covered here. Local regulations and Material Safety Data Sheets (MSDS) should be consulted.

6.2 Optional Components:

6.2.1 Sample Pumping System—A pumping system capable of transporting the sample to the transmission cell, emptying the cell and flushing the cell between samples may be used. Many commercial vendors offer various configurations of pump types, tubing and transmission cells for this type of application. It should be noted that non-homogeneity might occur if the oils are left standing for too long.

6.2.2 Filter—The use of a particulate filter (for example, 0.090 mm) to trap large particles is strongly recommended to prevent cell clogging when a pumping system is used. If a particulate filter is not used, the cell should be back-flushed regularly to prevent clogging.

6.2.3 Sealed Sample Compartment—The system configuration should be consistent with preventing harmful, flammable or explosive vapors from reaching the IR source.

6.2.4 Hydrocarbon Leak Alarm—When a sample pumping system is used, an independent flammable vapor sensor and alarm system should be used to alert the operator when a leak occurs in the tubing, connectors or transmission cell. This alarm system is strongly recommended when a pumping system is used to pump samples and wash solvents into an enclosed area.

6.2.5 Check Fluid—A check fluid or quality control fluid can be analyzed as needed for individual laboratory quality control and procedure issues and for comparison to other laboratories. One IR manufacturer has used heptane. A check sample should be a material that provides consistent results using the methods presented in the annexes to this practice. The purpose of this quality control fluid is to verify proper operation of the FT-IR spectrometer/transmission cell combinations, as well as any associated sample introduction and cleaning hardware.

7. Sampling and Sample Handling

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 [D4057](#).

7.2 Sample Preparation—No sample preparation is required. Laboratory samples should be shaken or agitated to ensure a representative sample is taken from the bottle.

8. Instrumentation Preparation

8.1 Spectral Acquisition Parameters:

8.1.1 Spectral Resolution— 8 cm^{-1} or better (lower numeric value).

8.1.2 Data Point Spacing Resolution— 4 cm^{-1} or better (lower numeric value).

8.1.3 Typical Range— 4000 cm^{-1} to 550 cm^{-1} (see [6.1.2](#)).

8.1.4 Spectral Format—Absorbance as a function of wavenumber.

8.1.5 Other Optical, Electronic Filtering and Interferogram Computational Parameters—These parameters should be as recommended by the manufacturer or as determined necessary for adequate measurement quality. Individual parameters and settings will vary depending on instrument manufacturer but most FT-IR spectrometers should be able to collect an adequate spectrum in less than one minute.