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.

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Standard Practice for Condition Monitoring of In-Service Lubricants by Trend Analysis Using Fourier Transform Infrared (FT-IR) Spectrometry[1]

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.

1.3 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.*

[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.

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

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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

D8321 Practice for Development and Validation of Multivariate Analyses for Use in Predicting Properties of Petroleum Products, Liquid Fuels, and Lubricants based on Spectroscopic Measurements

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

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.

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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° 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.

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NOTE 2—Identical scanning acquisition parameters should be used for all samples to be trended.

8.2 *Background Collection:*

8.2.1 The single-beam background collection (empty system reference scanned and stored on an FT-IR spectrometer) should be performed frequently enough such that ambient changes in atmospheric water vapor levels and other changing ambient conditions do not significantly affect the sample results (see Practice E1421). The frequency of background checks should be determined by the individual laboratory conditions and sampling technique; for example, at the completion of each run when an autosampler is used.

8.2.2 Note that changing water vapor levels will have the strongest effect, as water vapor is a strong infrared absorber. A water vapor check may be included in the software to monitor the intensity of the water vapor in the single-beam background spectrum. For example, the water vapor bands superimposed on the single-beam spectrum at 1540 cm^{-1}, 1559 cm^{-1}, and 1652 cm^{-1} may be measured relative to the average of baseline points at 1609 cm^{-1} to 1582 cm^{-1}. Acceptable limits for operation can be set; for example, measured peaks due to water vapor superimposed on the single-beam background should not be more than 10 % of the single-beam intensity.

8.2.3 Most of the research and development work used in the development of this practice used a background collection at least every 2 h. 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.

8.3 *Cell Pathlength Check*—A cell pathlength check is needed to verify the pathlength consistency of the cell. Results are referenced to 0.100 mm as mentioned in 6.1.2. This check is particularly important for water-soluble salt cell windows (for example, KBr). For systems using a fixed flow cell, the check can be performed at the same time as the background collection. Different instrument manufacturers may use different techniques for cell pathlength checks that may require the use of a reference or calibration fluid(s). A fringe-based method for determining cell pathlength is discussed in the appendix. Manufacturers' instructions and recommendations should be considered.

9. Procedures, Calculation, and Reporting

9.1 *Sample Introduction*—A representative sample is introduced into the infrared transmission cell, either manually or by an automatic pumping system. Autosamplers that hold a variety of oil sample container sizes are available from several manufacturers.

9.2 *Sample Integrity Check*—To ensure accurate and consistent results, the infrared spectrum of the sample should be checked to verify that the cell is completely filled and that air bubbles passing through the cell during data collection are not affecting the results. Multiple, automatic, computerized interpretation methods exist for this procedure. A sample integrity check based on measurement of the absorbance intensity over the wavenumber range from 3000 cm^{-1} to 1090 cm^{-1} is suitable for multiple lubricant types. The exact absorbance intensity will depend on the spectral resolution and the pathlength of the cell being used. The manufacturer's suggestions and recommendations should be considered.

9.2.1 Petroleum based lubricants have their maximum absorbance in the 3000 cm^{-1} to 2800 cm^{-1} range (or transmittance value close to 0 %T).

9.2.2 Ester based lubricants have their maximum absorbance in the 1390 cm^{-1} to 1090 cm^{-1} range (or transmittance value close to 0 %T).

9.3 *Sample System Cleaning and Checks*—To ensure the minimum amount of sample cross-contamination or sample carry-over, either a minimum volume of the next sample can be flushed, or a volatile solvent can be flushed through the cell and the cell dried. If the cell is dried, the amount of absorbance from either the previous sample or residual wash solvent in the sample cell can be checked. This check is performed by the same spectral analysis operation as described above. The maximum absorbance intensity should be below a preset threshold in the monitoring region (that is, CH stretch in petroleum based fluids). For most petroleum and synthetic lubricants and wash solvents, this intensity will be less than 0.2 absorbance units. The optimal threshold will depend upon the specific system configuration, in that some systems are designed to "push-out" the residual oil sample and wash solvent with the next sample. The manufacturer's suggestions and recommendations should be considered.

9.4 *Data Processing*—All spectra will be processed in units of absorbance as a function of wavenumber. Calculated data must be corrected to the reference pathlength of 0.100 mm prior to reporting to account for cell pathlength variation that will be seen in commercially available cells. Any other spectral data treatment should occur prior to calculating results from the spectrum.

9.4.1 Spectral data processing results can be trended directly from the in-service oil spectrum (direct trending). The only spectral data treatment is the correction of the spectrum or results to the 0.100 mm reference pathlength and the application of fringe reduction algorithms to the spectrum, if required.

9.4.2 Spectral data processing results can also be obtained by spectral subtraction processing, which requires a reference spectrum (spectral subtraction). Where spectral subtraction is used, processing of results is done from the difference spectrum that is generated by subtracting the appropriate new oil reference spectrum from the spectrum of the in-service oil sample. The in-service oil spectrum and new oil reference spectrum must both be corrected to the reference pathlength of 0.100 mm prior to subtraction and a 1:1 subtraction factor used. The subtracted spectral results can be trended over time and treated in a manner similar to those collected using the direct infrared trending method.

9.4.2.1 The most commonly used reference is a sample of new oil. If possible, the new oil should be from the same lot and drum as the in-service oil. An alternate approach that might yield a more representative reference would be to take a sample of oil one hour after the oil has reached operating temperatures.

9.4.3 Post-analysis data treatment can use simple multipliers and other scaling techniques; for example, "value × 100" at the request of maintenance personnel for ease in evaluation and presentation (see Annex A1).

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9.5 *Spectral Analysis of Sample Data*—Selected spectral regions containing information relevant to condition monitoring are measured and reported. The regions analyzed are specific to different lubricating fluid types. New oil sample parameters can be used as the point from which to trend when initially implementing an analysis process for a lubricant type. Statistical analysis shown in the annexes also provides examples. Details of the spectral analysis process can be found in the annexes to this Practice.

10. Effects of Oil Formulation

10.1 Differences in oil formulations can affect the results reported for the various measurements described in Annex A1. For example, Fig. 1 shows spectra of four 10W-30 oils in the carbonyl region where oxidation is measured for petroleum lubricants. In this example, absorbances for carbonyl-containing additives in these unused oil formulations contribute nearly a factor of 2 difference in the oxidation result measured by direct trending.

10.2 Results should be:

10.2.1 Interpreted relative to values measured for unused oil of the same formulation, or

10.2.2 Trended directly from the component sample history.

10.3 Distribution profiles for results for different oil formulations should typically not be combined unless justified by field experience in condition monitoring programs.

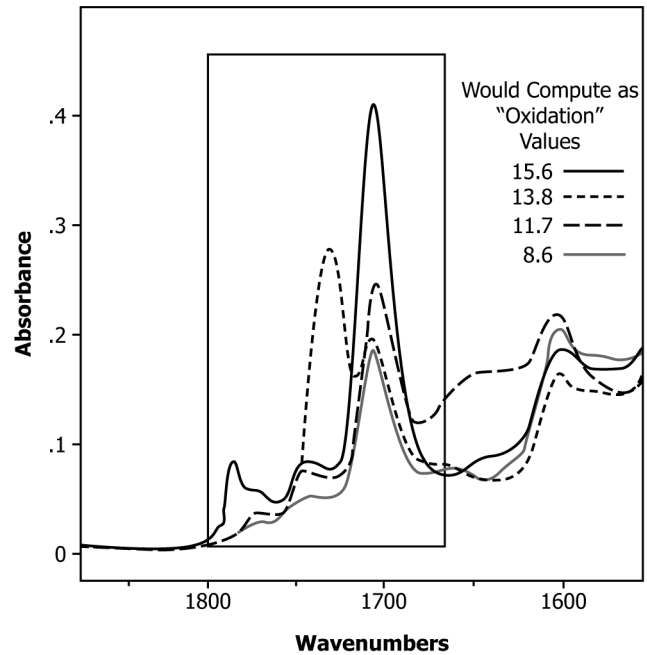

FIG. 1 Example of Carbonyl Containing Components in New Oil Formulations

11. Keywords

11.1 additive packages; base stock degradation; condition monitoring; contamination; Fourier transform infrared; FT-IR; fuel; glycol; infrared; IR; lubricating oils; nitration; oxidation; petroleum based extreme pressure lubricants; petroleum lubricants; polyol ester synthetic lubricants; soot; sulfates; trend analysis; water

ANNEXES

(Mandatory Information)

A1. MEASUREMENT OF MOLECULAR PARAMETERS IN VARIOUS SYSTEMS—DIRECT TRENDING

A1.1 This annex does not purport to discuss all lubricant types. Measurement parameters for petroleum lubricants (for example, crankcase), extreme pressure petroleum lubricants and polyol esters are presented. As data becomes available, other lubricant types can be added to the annex.

NOTE A1.1—It is not the intent of this practice to establish or recommend normal, cautionary, warning or alert limits for any machinery or fluids. Such limits should be established in conjunction with advice and guidance from the machinery manufacturer and maintenance group.

A1.2 *Petroleum Lubricants (Typically Diesel Engines)*—Monitoring of diesel crankcase oil is one of the most common applications of lubricant condition monitoring. Condition monitoring in these systems is divided into contaminant monitoring (typically water, soot, fuel, glycol) and oil degradation monitoring (typically oxidation and nitration). Sulfation degradation products may arise from lubricant component breakdown but commonly arise from the by-products of sulfur–containing diesel fuels. Measuring contamination from gasoline is also possible but not as widely applied, as comparatively few gasoline engines are enrolled in condition monitoring programs. In addition, monitoring of the zinc dialkyldithiophosphate (ZDDP) based antiwear component of the additive package is also possible. The most common FT-IR condition monitoring parameters for crankcase engines are presented in Table A1.1, with some spectral measurement examples presented as a guide in using band areas. Throughout these examples, the use of integrated band area is preferred as noted in Practice E168 because it has been "found to be more accurate than peak-height measurements because one is, in effect, averaging multipoint data."

A1.2.1 *Water:*

A1.2.1.1 Water contamination is monitored in diesel crankcase lubricants by measuring the hydrogen-bonded OH stretch region given in Table A1.1. An example of varying levels of water contamination is shown in Fig. A1.1. In the following examples (except soot) the infrared spectrum is [illegible]

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the described baseline, giving a visual example of how the integrated absorbance area is measured. Measurement of these band areas by computer assisted techniques is common in most infrared manufacturers' software packages. For the water measurement in crankcase oils, the area under the curve between 3500 cm^{-1} and 3150 cm^{-1} is shaded, showing an example of the measurement described above.

A1.2.1.2 *Water Interferences*—High soot levels (~10 % w/w solids) may interfere with water measurements in diesel engines, but interference has not been seen until the soot limit has been exceeded (that is, > 3 % to 5 % w/w solids). As a condition limit (soot) has already triggered, action should be taken irrespective of water. Exact quantitative measurement of soot is difficult (that is, % w/w) due to multiple infrared contributing factors as well as the many different soot measurement methods available.

A1.2.2 *Soot:*

A1.2.2.1 Soot loading is measured from the baseline offset at 2000 cm^{-1} as described in Table A1.1. Fig. A1.2 shows some examples of spectra showing low, intermediate, high and very high soot loading levels (increasing levels from 1 through 5).

A1.2.2.2 *Soot Interference*—High water levels have been observed to interfere with the measurement of soot in internal combustion engine crankcases. However, this interference does not become significant until the water level is on the order of >5 % (50 000 ppm), levels which will immediately condemn the lubricant and require immediate maintenance action irrespective of any other indicators.

A1.2.3 *Oxidation, Nitration and Sulfation:*

A1.2.3.1 Unlike the previous examples, oxidation, nitration and sulfation breakdown products in crankcase oils cannot be easily quantified by comparison to pure prepared standards. Here, there are a large number of different oxidation and nitration compounds that can be produced and gradually build up in the oil. Fig. A1.3 shows the measurement areas for oxidation and nitration product buildup monitoring, with the sulfation region highlighted in Fig. A1.4.

A1.2.3.2 *Oxidation, Nitration and Sulfation Interferences*—As in the soot measurement, very high water levels can generate false positives for oxidation and nitration. However, water levels of this magnitude will immediately condemn the lubricant. Very high (>5 %) glycol levels in a crankcase oil may start interfering with sulfation measurement, but again contaminant levels of this magnitude would dictate immediate maintenance action. Various additive packages, such as detergents, dispersants, antioxidants, overbase additives, etc. may also generate significant absorbance in the condition monitoring regions of interest. Blends of petroleum lubricants with significant amounts of ester, whether part of the base-stock package or as an additive, will absorb strongly in the oxidation area. These lubricants are not presented at this time.

A1.2.4 *Fuel Contamination:*

A1.2.4.1 The possibility of fuel contamination may be indicated in diesel crankcase lubricants by measuring the peak at 810 cm^{-1}. Spectral characteristics of diesel (Figs. A1.5 and A1.6) and other fuels noted in Table A1.1 have been found to vary. Work is currently active on other IR measurement areas and techniques. The measurement listed can be used as a guideline but is not intended to be the only infrared based fuel contamination measurement. An independent test, such as viscosity change, flash point, or gas chromatography can be used to confirm an indication of fuel presence in the FT-IR spectrum of the oil.

A1.2.5 *Glycol Antifreeze Contamination:*

A1.2.5.1 Glycol contamination is monitored in diesel crankcase lubricants by measuring the carbon-oxygen stretch region as noted in Table A1.1. Spectral characteristics of glycol contamination are shown in Fig. A1.6.

A1.2.5.2 Ethylene glycol will interfere with the ability to accurately quantify water level when present since it also contains hydroxyl groups. However, the converse is not true since glycol has other spectral features that are used for detection and quantification. Therefore, when glycol is present, water can be detected but not reliably quantified using FT-IR spectroscopy. This is not considered a problem because of the greater significance the presence of glycol has to engine operation. As with fuel, the presence of glycol can be confirmed by gas chromatography or a colorimetric test, or more commonly, corroborated using elemental analysis results for sodium and boron.

A1.3 *Extreme Pressure (EP) Fluids (Typically Petroleum Gear or Hydraulic Fluids)*:

A1.3.1 In addition to the above crankcase oil analysis, condition monitoring of gear and hydraulic oil is also widely applied. In these systems, the most common parameters measured are water contamination and oxidative breakdown of the oil, which are presented in Table A1.2.

A1.3.2 *Water:*

A1.3.2.1 As water is the most common contaminant in crankcase oils, it is also the most common contaminant in gearboxes and hydraulic systems. In these systems, unlike the crankcase oils, however, interactions between water and the EP additives alter the infrared response, and thus water is measured differently than in the crankcase lubricants. Fig. A1.7 demonstrates this different response of water. Water contamination is manifested as a general, horizontal baseline offset of the entire infrared spectrum. Here, the integrated area for the spectrum representing 3000 ppm (0.3 %) water is shaded. While this measurement becomes the principal water measurement in EP fluid systems, very high water levels (greater than 2 %) will begin to show a similar hydrogen-bonded OH stretch band as seen in the crankcase oils.

A1.3.2.2 *Water Interferences*—As the principal water measurement is based on the integrated absorbance with no local baseline correction, soot, dirt and high concentrations of infrared scattering particulates will generate higher than expected readings for water. However, typical gearboxes and hydraulic systems will not contain particulate levels high enough to cause a significant baseline offset and tilt. Wear metal analysis, particle counting or other applicable tests should condemn gear and hydraulic systems that manifest such extreme particulate levels.

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A1.3.3 *Oxidation:*

A1.3.3.1 The oxidative breakdown measurement shown in Fig. A1.8 in petroleum EP fluids is the same as in the petroleum-based crankcase fluids discussed in A1.2.3.2. Note that while Fig. A1.8 also shows an increase in sulfation by-products, not all EP systems will show this effect.

A1.4 *Synthetic Polyol Ester Lubricants (Typically Aero-Derivative Gas Turbines)*:

A1.4.1 Condition monitoring of high-performance aircraft turbine engines is widely applied in both the military and commercial aviation maintenance industries. In addition, many aero-derivative gas turbines are used in power generation, marine transport and other non-aeronautical applications. In these systems, the primary lubricant is a synthetic polyol ester and is available under a variety of different military specifications and commercial item descriptions and brand names. Table A1.3 lists the condition monitoring properties of interest measured by FT-IR along with the band measurement area and the baseline point(s).

A1.4.2 *Water:*

A1.4.2.1 Just as the infrared measurement for water was adjusted to account for the different interactions in the formulations in crankcase and EP oils, a different water measurement area is also required for the polyol esters. Fig. A1.9 shows the area under the curve that is integrated for the determination of water contamination in these systems, with the measurement highlighted for a sample containing 1000 ppm of added water. Note that the water in these systems shows up as a broad band, similar to what is observed for water in the crankcase oils, but the strongest response occurs at higher frequencies than in the case of the crankcase oils (~3700 cm^{-1} to 3600 cm^{-1} for polyol esters versus 3500 cm^{-1} to 3150 cm^{-1} for crankcase oils).

A1.4.2.2 *Water Interferences*—The most significant interference found in the determination of water is interference from the polyol ester lubricant breakdown I (see A1.4.3). Under severe conditions of lubricant degradation, this band will begin to overlap and contribute to the integrated water measurement area. As seen below in Fig. A1.10 however, this effect is only seen when the lubricant is already severely degraded, which dictates maintenance action from the degradation irrespective of the actual water level.

A1.4.3 *Ester Base-Stock Breakdown:*

A1.4.3.1 As the polyol esters are a different chemical system than petroleum based lubricants, degradation of the polyol ester lubricant produces different breakdown products. The most common degradation pathway in ester based lubricants is the conversion of the ester into organic acids and alcohols.

A1.4.3.2 *Ester Base-Stock Breakdown I*—The resulting polyol ester degradation products are first seen between 3595 cm^{-1} and 3500 cm^{-1}, and the measurement is noted as ester base-stock breakdown I in Table A1.3 and highlighted in Fig. A1.10. As this area is closely associated with the water measurement area, a localized, single–point baseline at 3595 cm^{-1} provides a correction for low levels of water buildup (Fig. A1.10).

A1.4.3.3 *Ester Base-Stock Breakdown II*—In addition to the breakdown area I, a second area associated with the traditional OH stretch (as measured for water in crankcase oils) also increases as the lubricant breaks down. This ester base-stock breakdown II area is also monitored as a measurement of degradation of the polyol ester lubricants. The breakdown II region is also highlighted in Fig. A1.10.

A1.4.3.4 *Ester Base-Stock Breakdown Interferences*—As noted above in A1.4.2.2 where excessive base-stock breakdown interferes with the water measurement, a similar effect has also been noted with the lubricant breakdown measurement. Excessive water levels may cause the lubricant breakdown reading to be higher than the actual level. Once again however, water levels of this magnitude will condemn the lubricant irrespective of the actual breakdown level.

A1.4.4 *Antiwear Components:*

A1.4.4.1 While the antiwear compounds used in crankcase oils and polyol ester lubricants are typically different species, the most common compounds used for both oils have a phosphate functional group. For this reason, the measurement area developed for monitoring levels and trends of ZDDP has been found to be equally useful for monitoring tricresyl phosphate (TCP). Fig. A1.11 shows varying levels of TCP blended into a polyol ester lubricant. As previously noted, building calibration curves for measurement parameters (when pure or prepared standards are available) is possible. However, this is not necessary, as lubricant condition monitoring requires only reliable, repeatable measurements. Correlation of FT-IR measurements to physical values is not necessary.

A1.4.5 *Fuel Contamination:*

A1.4.5.1 Fuel contamination is monitored in polyol ester lubricants by measuring the peak at 810 cm^{-1} as given in section A1.2.4.

A1.4.6 *Other Fluid Contamination:*

A1.4.6.1 In addition to fuel contamination, foreign oils and hydraulic fluids may contaminate lubricating oils (for example, polyol ester contaminated by a petroleum based fluid). In most cases, identifying the presence of a foreign fluid is all that is required to generate an appropriate maintenance response. The wide variety of potential contaminants suggests an equally wide variety of measurement methods may be desirable. In addition, multiple frequency distributions may also be required and are not given here. The measurement areas given in Table A1.3 demonstrate the measurement used to indicate the presence of petroleum oils, phosphate ester oils, or polyalphaolefin (PAO)/diester blend oils contaminating polyol ester oils. Fig. A1.12 shows an example of polyol ester oil contaminated by a polyalphaolefin (PAO)/diester blend oil.

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TABLE A1.1 Petroleum Lubricant (for example, Crankcase) Condition Monitoring Parameters—Direct Trending

Component	Measurement Area, cm^{-1}	Baseline Point(s), cm^{-1}	Reporting[A]
Water	Area 3500 to 3150	Minima 4000 to 3680 and 2200 to 1900	Report Value as Measured
Soot Loading	Absorbance intensity at 2000	None	Value × 100
Oxidation	Area 1800 to 1670	Minima 2200 to 1900 and 650 to 550	Report Value as Measured
Nitration	Area from 1650 to 1600	Minima 2200 to 1900 and 650 to 550	Report Value as Measured
Antiwear Components (Phosphate based, typically ZDDP)	Area 1025 to 960	Minima 2200 to 1900 and 650 to 550	Report Value as Measured
Gasoline	Area 755 to 745	Minima 780 to 760 and 750 to 730	Report Value as Measured
Diesel (JP-5, JP-8)[B]	Area 815 to 805	Minima 835 to 825 and 805 to 795	(Value + 2) × 100
Sulfate by-products	Area 1180 to 1120	Minima 2200 to 1900 and 650 to 550	Report value as measured
Ethylene Glycol Coolant	Area 1100 to 1030	Minima 1130 to 1100 and 1030 to 1010	Report value as measured

[A] Reporting values in absorbance/0.1 mm (see 6.1.2).
[B] Spectral characteristics of diesel and other noted fuels have been found to vary. Work is currently active on other IR measurement areas and techniques. The measurement listed can be used as a guideline but is not intended to be the only infrared based fuel contamination measurement. Checking suspect fuel sources is suggested to verify presence of indicator absorbance bands.

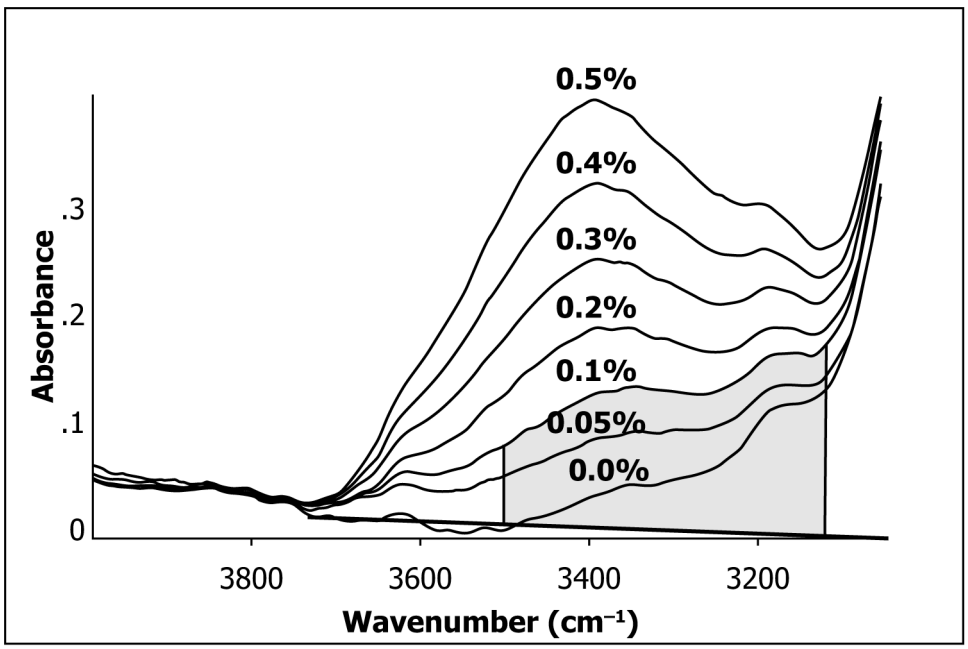

FIG. A1.1 Example of Integrated Band Measurement Area for Water in Crankcase Oil

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