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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 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:*[2]

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

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

E2412 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 E131.

3.1.2 *Fourier transform infrared (FT-IR) spectrometry, n*—form of infrared spectrometry in which an interferogram is

[1] 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.

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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.

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 (5-8), as described in Practice E2412. 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 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 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 (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, respectively. The actual cell pathlength obtained can be determined from the interference fringes in the spectrum recorded

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

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^{-1}
Data spacing: 2 cm^{-1}
Apodization: Triangular
Scanning range: 4000 to 550 cm^{-1}
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 $N^{1/2}$.

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 D4057.

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.[4, 5]

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. The frequency of background checks shall be determined by the individual laboratory.

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

[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.

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

9.1.2.1 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.

9.1.2.2 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 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 12).

9.1.3 *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 9.1.2, 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.

9.2 *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 9.1 and using the operating parameters specified in 6.1. If spectral normalization is being used (see 5.2.2), the absorption spectrum should subsequently be normalized (see 10.2) to a pathlength of 0.100 mm.

9.3 *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 9.1 and using the operating parameters specified in 6.1. If spectral normalization is being used (see 5.2.2), the absorption spectrum should subsequently be normalized (see 10.2) to a pathlength of 0.100 mm.

9.4 *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 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 4—As a general guideline, the integrated absorbance after normalization to a pathlength of 0.100 mm (10.2) should be >2 for petroleum and ester-based oils.

10. Reporting of Spectral Data

10.1 All spectra must be processed in units of absorbance as a function of wavenumber.

10.2 *Pathlength Normalization*—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 to account for pathlength variations, either by multiplying all data points in the absorption spectra by a pathlength correction factor (spectral normalization) or by multiplying the results from the respective test methods by a pathlength correction factor (see 5.2.2). Various instrument manufacturers provide a normalization procedure in their software. The manual procedure is described in 10.2.1.

10.2.1 *Manual Procedure for Pathlength Normalization*—The pathlength of the cell is calculated according to Appendix X2, and the pathlength correction factor is calculated as follows:

$$\textit{Pathlength correction factor} = 0.100\ \textit{mm/Actual pathlength} \quad (1)$$

To perform spectral normalization, all the data points of the absorption spectrum are multiplied by the pathlength correction factor. Most software packages for FT-IR instruments include spectral multiplication as one of their spectral data manipulation functions.

10.3 *Reporting for Direct Trend Analysis*—Absorption spectra over the range of 4000 to 550 cm^{-1} are stored to disk and used for measuring various in-service oil condition monitoring parameters (see the respective test methods). If spectral normalization has not been performed, the pathlength correction factor (see 10.2) for each spectrum must be reported; most software packages for FT-IR instruments provide a spectrum title or comments box in which this value may be entered and stored with the spectral data.

10.4 *Reporting for Differential Trend Analysis*—The normalized absorption spectrum of the reference oil is subtracted from the normalized absorption spectrum of the in-service oil sample using a 1:1 subtraction factor to generate the differential spectrum of the in-service oil sample. Most software packages for FT-IR instruments include spectral subtraction as one of their spectral data manipulation functions. Differential spectra over the range of 4000 to 550 cm^{-1} are stored to disk and used for measuring various in-service oil condition monitoring parameters (see the respective test methods). If spectral normalization has not been performed, the pathlength correction factor (see 10.2) for each spectrum must be reported; most software packages for FT-IR instruments provide a spectrum title or comments box in which this value may be entered and stored with the spectral data.

11. Instrument Performance Checks

11.1 Periodically, the performance of the FT-IR instrument should be monitored using the Level 0 procedure of Practice E1421. If significant change in performance is noted, then testing should be suspended until the cause of the performance change is diagnosed and corrected.

11.2 Alternative instrument performance tests conforming to the recommendations of Guide E1866 may be substituted for the Practice E1421 test.

12. Sample Spectral Noise Test

12.1 The sample spectral noise test is designed to ensure that the noise level in the sample spectrum, including the amplitude of the fringe pattern that may be superimposed on the spectrum, especially when using ZnSe cells or when ratioing against an empty cell background, is acceptable. This test shall be carried out each day that the instrument is in operation. To calculate the sample spectrum noise level, fill the cell with a check fluid, and record its absorption spectrum. The noise level is calculated by measuring the maximum peak-to-peak noise in milliabsorbance units (mAU) over the frequency range of 2000-1900 cm^{-1}. The software of most commercial spectrometers includes a noise calculation that can be used to measure peak-to-peak noise over a selected frequency range. The peak-to-peak noise level can be improved by increasing the number of scans, whereby the noise level is decreased by a factor of $N^{1/2}$ when the number of scans is increased by a factor of N. Various fringe reduction techniques outlined in Appendix X1 can be used to help reduce sample spectral "noise" caused by fringing. Acceptable sample spectral noise levels shall be determined during a round robin and will be available on or before July 1, 2009.

13. Keywords

13.1 condition monitoring; differential trend analysis; Fourier transform infrared; FT-IR; hydrocarbon based lubricants; infrared; IR; lubricants; oils; petroleum lubricants

APPENDIXES

(Nonmandatory Information)

X1. FRINGING EFFECTS IN INFRARED LIQUID TRANSMISSION SAMPLE CELLS AND FRINGE REDUCTION METHODS

X1.1 When light passes from one medium into another medium of different refractive index, its velocity changes and some reflection occurs, such that the amount of light transmitted through the interface is less than 100%. When light passes through a transmission cell, two such interfaces are encountered owing to the difference in refractive index between the cell window material and the sample. If the two windows are parallel, some of the light that is reflected back from the second window is again reflected back from the first window. Because this reflected light is then traveling in the original direction with a phase delay from the original beam, it interferes with the original beam to produce a sinusoidal interference pattern. The result is that a spectrum plotted in wavenumbers shows a regular sine wave (fringes) superimposed on the spectrum of the material between the windows.

X1.2 Such interference effects are particularly strong when light passes through the air gap in an empty transmission cell having parallel windows because of the large refractive index difference between air and the cell window material. This optical effect can be used to accurately measure the pathlength of an empty cell, either from the resulting spectral fringes (see X2.1) or from the phase delay observed in the interferogram (see Practice E168).

X1.3 When a cell is filled with a sample having a refractive index similar to that of the cell window material, the fringe pattern is weakened to negligible levels. Such is the case when the spectra of oils are recorded in cells assembled with low refractive index salt windows (for example, KCl, KBr) because the refractive index of these windows is fairly similar to that of oils. However, when the spectrum of an oil is recorded using a cell assembled with window materials having a high refractive index (for example, ZnSe), a readily observable fringe pattern is superimposed on the oil spectrum because of the significant refractive index difference between the cell window material and the oil. Because fringe patterns vary with pathlength, they can cause significant variations in peak height and area measurements, compromising between-instrument comparisons. A variety of fringe reduction techniques are available to minimize fringing when high-refractive-index windows such as ZnSe are used or in instances where the background is taken through an empty cell. These include:

(1) Turning the cell holder at an angle away from the normal so that the empty cell fringes are minimized (Brewster's angle) and locking it into that position.

(2) Using cell windows with antireflective coatings

(3) Wedging the windows so that they are not parallel to each other, for example by using angled spacers

(4) Software-based, automated fringe reduction procedures provided by spectrometer manufacturers which locate and digitally remove the secondary (phase-delayed) centerburst from the interferogram recorded for the empty cell.

X1.4 Please consult with the manufacturer as to which fringe reduction method is appropriate for your system. Whichever technique is used (1– 4), its effectiveness can be validated by measuring the peak-to-peak noise level in the spectrum of a check fluid (see 12.1) over the range of 2000 to 1900 cm^{-1}. Fig. X1.1 illustrates the effectiveness of fringe reduction when an absorption spectrum of mineral oil in a ZnSe cell is recorded with the cell holder turned to Brewster's angle rather than placed normal to the IR beam.

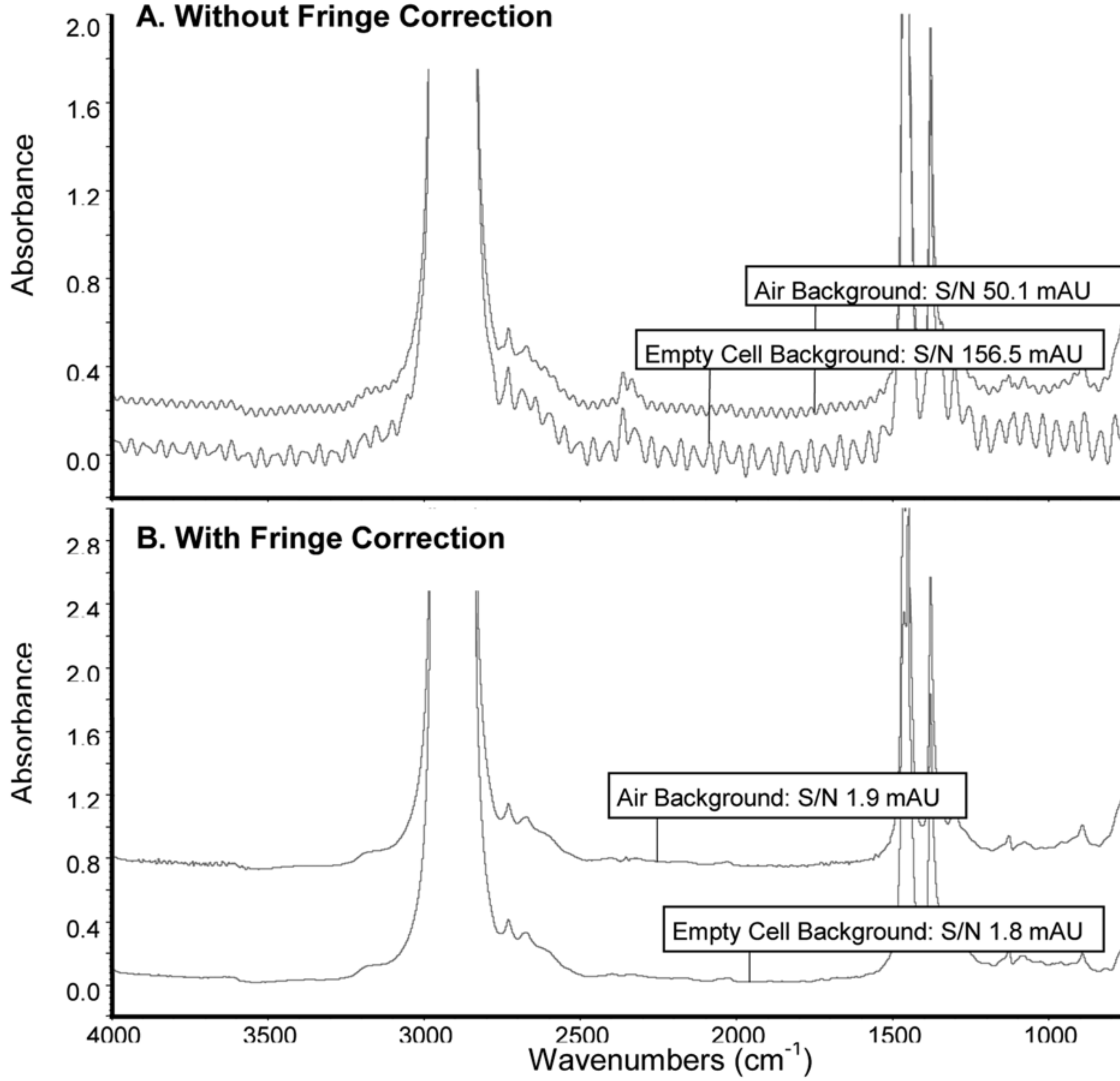

FIG. X1.1 Spectrum of a Mineral Oil Recorded Using an Air and an Empty Cell Background Before (A) and After (B) Fringe Reduction by Tilting the Cell Holder at Brewster's Angle

X2. SAMPLE CELL PATHLENGTH CALCULATION

X2.1 *Calculation using Interference Fringes*—Assemble a cell using a spacer of approximately 0.100 mm. Calculate the infrared cell pathlength as follows:

X2.1.1 *Acquire the FT-IR Spectrum of the Empty Cell*—Collect an absorption spectrum of the empty cell in the sample compartment (without using fringe reduction techniques). The spectrum will display a sinusoidal wave pattern (if no such wave pattern is observed, the windows are not clean or the windows are not parallel to each other).

NOTE X2.1—When cell windows have been wedged to reduce interference fringes (Appendix X1), interference fringes may not be observed in the absorption spectrum of the empty cell. In this instance, the cell pathlength must be determined from the spectrum of a check fluid as described in X2.2.

X2.1.2 *Calculation*—Choose two minima of the sinusoidal wave pattern separated by about 20 measurable interference fringes in a region of the IR spectrum of the empty cell where the fringes are regular. Count the number of interference fringes, N, between the higher and lower wavenumbers (units of cm^{-1}), referred to as $\nu 1$ and $\nu 2$, respectively, as illustrated in Fig. X2.1.

X2.1.3 Calculate the pathlength using the following formula:

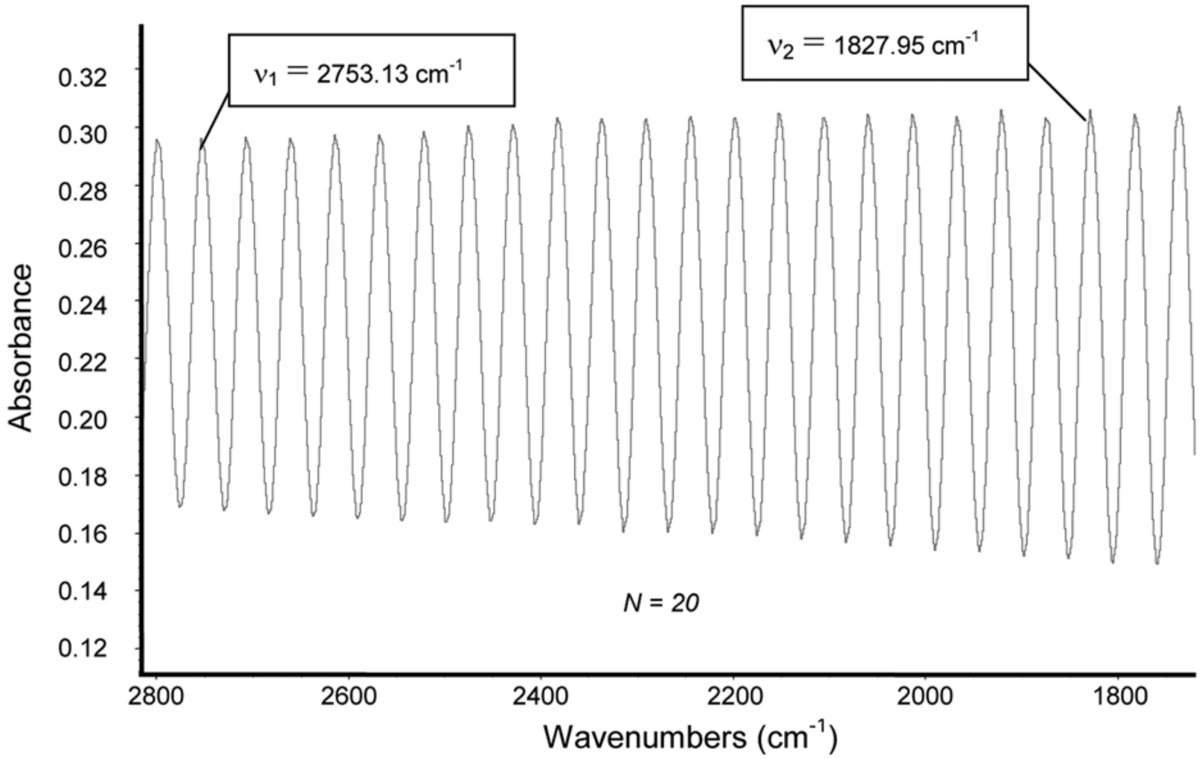

FIG. X2.1 Spectrum of an Empty Cell Used for Calculation of Cell Pathlength

$$E = 1/2 \cdot N/(\nu 1 - \nu 2) \cdot (10\ mm/cm) = 5 \cdot N/(\nu 1 - \nu 2) \quad \text{(X2.1)}$$

where:

E = cell pathlength in mm, and
N = number of fringes between $\nu 1$ (cm^{-1}) and $\nu 2$ (cm^{-1}).

X2.2 *Calculation Using Check Fluid*—To calculate the sample cell pathlength using a check fluid (see 8.3), assemble three cells using alkali halide windows (such as KCl or KBr) and a spacer of 0.050, 0.100, and 0.150 mm, respectively. For each cell assembled, collect an FT-IR spectrum through the empty cell to obtain its fringe pattern and calculate its pathlength following the instructions in X2.1. Subsequently, load each cell with the check fluid and measure the absorbance maximum (peak height) of a peak that is on-scale (maximum absorbance at a pathlength of 0.15 mm should be <1.2 and minimum absorbance at a pathlength of 0.05 mm should be >0.3 to ensure adequate sensitivity and linearity) relative to a single-point baseline located at a wavenumber where the check fluid has no absorption. The FWHM (full width at half-maximum) of the peak selected should not be narrower than the resolution used to acquire the FT-IR spectrum of the check fluid. Plot the absorbance values obtained from the peak height measurements versus their respective pathlength values for each of the assembled cells. Carry out a linear regression to obtain an equation which has the following form:

$$y = mx + b \quad \text{(X2.2)}$$

where:

m = slope,
b = intercept,
x = absorbance values, and
y = pathlength values.

X2.3 The linear regression equation should have an SD of <0.005 mm for the pathlength calibration to be workable; if the SD is >0.005 mm, then recalibrate. This equation can then be used to calculate the pathlength of any cell assembled at any time thereafter by recording the spectrum of the check fluid and measuring the absorbance value of the peak selected above. Solve for y, the pathlength value, using the linear equation above. This procedure is effective as long as the spectral characteristics of the check fluid used are consistent from lot to lot.

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