Designation: D7889 – 13

# Standard Test Method for
# Field Determination of In-Service Fluid Properties Using IR Spectroscopy[1]

This standard is issued under the fixed designation D7889; 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 test method describes the use of a grating spectrometer to analyze properties of an in-service fluid sample which are indicative of the status of that fluid and related machinery.

1.2 This test method provides a means for the assessment of in-service fluid properties using infrared spectroscopy. It describes a methodology for sampling, performing analysis, and providing key in-service fluid properties with a self-contained unit that is meant for field use. It provides analysis of in-service fluids at any stage of their useful life, including newly utilized fluid.

1.3 In particular, these key in-service fluid properties include oxidation, nitration, sulfation, soot, and antiwear additives. They are applicable for hydrocarbon type (API Group I-IV) fluids from machinery lubricants, including reciprocating engine oils, turbine oils, hydraulic oils, and gear oils.

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

1.4.1 *Exception*—The unit for wavenumbers is in $cm^{-1}$.

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

D7412 Test Method for Condition Monitoring of Phosphate Antiwear Additives in In-Service Petroleum and Hydrocarbon Based Lubricants by Trend Analysis Using Fourier Transform Infrared (FT-IR) Spectrometry

D7414 Test Method for Condition Monitoring of Oxidation in In-Service Petroleum and Hydrocarbon Based Lubricants by Trend Analysis Using Fourier Transform Infrared (FT-IR) Spectrometry

D7415 Test Method for Condition Monitoring of Sulfate By-Products in In-Service Petroleum and Hydrocarbon Based Lubricants by Trend Analysis Using Fourier Transform Infrared (FT-IR) Spectrometry

D7418 Practice for Set-Up and Operation of Fourier Transform Infrared (FT-IR) Spectrometers for In-Service Oil Condition Monitoring

D7624 Test Method for Condition Monitoring of Nitration in In-Service Petroleum and Hydrocarbon-Based Lubricants by Trend Analysis Using Fourier Transform Infrared (FT-IR) Spectrometry

D7669 Guide for Practical Lubricant Condition Data Trend Analysis

D7720 Guide for Statistically Evaluating Measurand Alarm Limits when Using Oil Analysis to Monitor Equipment and Oil for Fitness and Contamination

D7844 Test Method for Condition Monitoring of Soot in In-Service Lubricants by Trend Analysis using Fourier Transform Infrared (FT-IR) Spectrometry

E131 Terminology Relating to Molecular Spectroscopy

E168 Practices for General Techniques of Infrared Quantitative Analysis

E932 Practice for Describing and Measuring Performance of Dispersive Infrared Spectrometers

E1655 Practices for Infrared Multivariate Quantitative Analysis

E2412 Practice for Condition Monitoring of In-Service Lubricants by Trend Analysis Using Fourier Transform Infrared (FT-IR) Spectrometry

E2617 Practice for Validation of Empirically Derived Multivariate Calibrations

## 3. Terminology

3.1 For definitions of terms relating to infrared spectroscopy used in this test method, refer to Terminology E131. For

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[1] This test method 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.

definition of terms related to infrared-based in-service fluid condition monitoring, refer to Practice D7418.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *absorbance units (AU), n*—units of measurement of the raw absorbance spectrum which is obtained using the definition described in Section 8 (Theory for a Single Compound Analysis) of Practice E168, which is not normalized for pathlength.

3.2.2 *cell background, n*—a single-beam spectrum that is obtained on a clean, empty wipe-clean transmission cell.

3.2.3 *mid-infrared grating spectrometer, n*—a spectrometer which operates in the mid-infrared spectral range, between at least 960 $cm^{-1}$ and 3040 $cm^{-1}$ and creates an infrared spectrum by means of a reflective diffraction grating.

3.2.3.1 *Discussion*—Such a grating spectrometer may be of any of a variety of designs and optical configurations. Example designs include monochrometer-type systems wherein the grating is rotated to a single point infrared detector, or array-type systems which utilize an infrared detector array at the output and a fixed grating. Example optical configurations include Rowland-Circle and Czerny-Turner systems. Typical infrared detectors are uncooled thermal detectors such as thermopile or pyroelectric-based sensors.

3.2.4 *reporting units, n*—specifies the reporting units of the fluid analysis property.

3.2.5 *self-contained field apparatus, n*—a mid-infrared grating spectrometer which is of the form factor to allow it to operate as an independent device suitable for field use.

3.2.6 *wipe-clean transmission cell, n*—an infrared transmission cell which is specifically tailored for field use.

3.2.6.1 *Discussion*—In particular, the cell may be utilized and cleaned with a towel or rag and without the use of reagents or chemicals of any sort, making it convenient for use as a field device. Such transmission cells may accomplish this using mechanisms for quick open/close of the cell such as by means of a mechanical lever, demountable screw or press fit, or magnetic coupling. To correct for any cell fringing effects, the cell utilizes a wedged design both on the interior faces and exterior faces of the cell windows: The cell windows themselves are wedged at an angle of less than 0.5 degrees. The spacing between the two windows is wedged at an angle of approximately 0.013 degrees. The cell is designed to be nominally 100 µm in pathlength, with ZnSe windows.

## 4. Summary of Test Method

4.1 This test method utilizes a self-contained field apparatus to provide detailed information concerning the condition status of in-service fluids. In particular, it provides readings of oxidation, antiwear additive, sulfation, nitration and soot levels in hydrocarbon type (API Group I-IV) fluids.

4.1.1 An absorbance spectrum of the sample under test is obtained. For the background spectrum, the cell background is used.

4.1.1.1 Test Methods have been developed for Fourier-transform infrared (FTIR) devices using absorbance spectra obtained using Practice D7418. Particular Test Methods developed for in-service monitoring of hydrocarbon type (API Group I-IV) fluids include those established for Oxidation (Test Method D7414, Procedure A), Antiwear Additive (Test Method D7412, Procedure A), Sulfation (Test Method D7415, Procedure A), Nitration (Test Method D7624, Procedure A), and Soot (Test Method D7844, Procedure A). These test methods have served to establish the signature infrared spectroscopic behavior associated with key in-service monitoring properties. This test method provides property values based on the examination of each property's signature infrared spectroscopic behavior. The essence of this test method is to capture the underlying chemical trends associated with each property for in-service fluid analysis using a self-contained field apparatus and coupled wipe-clean transmission cell.

4.1.2 From the infrared absorbance spectrum obtained with the self-contained field apparatus, properties of the in-service fluid are calculated. In particular, those properties in Table 1 are calculated by the device and presented to the user on the display. Additional properties using infrared calibration methods may be calculated and displayed depending on the particular fluid being analyzed and availability of calibrations for that fluid.

4.1.2.1 Infrared spectra generated by the described instrument type can be used to provide a further set of properties of interest to in-service fluid analysis of hydrocarbon type (API Group I-IV) fluids. Such properties must be calibrated to the particular fluid blend, and may be generated using ASTM guidelines which govern the creation of such calibrations. Such calibrations may be built from either standard regression methods as described in Practice E168 or as described in Practice E2412. Further, they may also be multivariate calibrations, described in Practice E1655 and Practice E2617. Example properties include Acid Number (AN), Base Number (BN), water contamination, ethylene glycol, fluid mixture content, and antioxidant depletion. It should be noted that, due to the fact that these calibrations are sample-specific, this test method does not provide a prescription for calculating such properties.

4.2 The results of the test method can be compared against pre-defined or user-defined limits so as to judge the condition of the in-service lubricant. Warning and alarm limits exceedences which may be pre-defined or set by the user are indicated by the property and associated value being highlighted using coding established in Guide D7720, with either green (favorable alarm level designation showing acceptable condition), yellow (intermediate level alarm designation warning a fault condition is present and will likely need attention in the future), or red (high level alarm designation showing significant deterioration) indicated by the self-contained field apparatus.

**TABLE 1 In-Service Lubricant Properties Reported by the Test Method**

| Property | Reporting Units |
|---|---|
| Oxidation | Abs/0.1 mm |
| Antiwear Additive | Abs/0.1 mm |
| Sulfation | Abs/0.1 mm |
| Nitration | Abs/cm |
| Soot | Abs/cm |

## 5. Significance and Use

5.1 This test method provides a means for obtaining useful in-service fluid analysis properties in the field. It is not to be confused with laboratory or portable FTIR devices which provide measurements per the existing Test Methods listed in 4.1.1.1. Each of these monitored properties has been shown over time to indicate either contamination in the fluid system or a particular breakdown modality of the fluid, which is critical information to assess the health of the fluid as well as the machinery. By utilizing the field device, it is possible for those operating machinery, in locations and situations where it is not practical to gather a sample for the laboratory, to obtain quality in-service fluid analysis. This may be due to the need to have an analysis done in real-time, on-the-spot to maximize the operational hours of equipment, or to have the analysis performed at a location where no laboratory analysis is available.

## 6. Interferences

6.1 Spectral interferences due to very high levels of external contamination in the fluid can yield errors with these measurements. Common contaminants include the presence of API Group V lubricants at levels exceeding 5 % and antifreeze mixes at similar levels.

## 7. Apparatus

7.1 A self-contained mid-infrared grating spectrometer with a coupled wipe-clean transmission cell as defined in Section 3.

7.2 This spectrometer shall have specific performance characteristics indicated in 7.3 – 7.5, with a Spectral Format in the form of absorbance as a function of wavenumber reported at a digital resolution of 2 cm$^{-1}$.

7.3 *Signal-to-Noise Ratio (S/N)*—shall be adequate to provide the desired precision as indicated in Section 17. Practically, this means that, over the range of measurement, the standard deviation of the obtained absorbance should be less than 0.001 AU. Based on the capabilities of the spectrometer system, this may be achieved by co-adding a number of scans to improve the S/N as needed.

7.4 *Spectral Resolution*—shall be approximately 1.5 % of frequency being measured across the measurement range. For example, at 1000 cm$^{-1}$, the spectral resolution should be 15 cm$^{-1}$. In order to qualify this resolution, a simple test using a 40 micron film of polystyrene, a standard reference material for grating instruments as discussed in Practice E932 may be performed. The absorbance spectrum of this material measured with the spectrometer should show a peak at band number 12 (1028 cm$^{-1}$) of approximately 0.29 AU and a peak at band number 2 (2924 cm$^{-1}$) of approximately 0.56 AU. Other spectral resolutions may provide accurate results as well but the calculation parameters listed in Table 2 and Section 17 may be different from those listed.

7.5 *Spectral Range*—shall cover the frequencies necessary for calculation of all properties described in the method, which is 960 cm$^{-1}$ to 3040 cm$^{-1}$.

**TABLE 2 Specification for Calculation of Each In-Service Fluid Property**

| Property | Measurement, (cm$^{-1}$) | Baseline(s), (cm$^{-1}$) | Reporting Units |
|---|---|---|---|
| Oxidation | Average<br>1800–1670 | Average<br>1815 to 1805 | Abs/0.1 mm |
| Antiwear Additive | Average<br>1025–960 | Average<br>1060 to 1030 and<br>1812 to 1803 | Abs/0.1 mm |
| Sulfation | Average<br>1180–1120 | Average<br>1210 to 1200 and<br>1115 to 1105 | Abs/0.1 mm |
| Nitration | 100·Single<br>Point at 1630 | Average<br>1607 to 1597 and<br>1847 to 1837 | Abs/cm |
| Soot | 100·Single<br>Point at 2000 | None | Abs/cm |

## 8. Reagents and Materials

8.1 The only materials required to make a measurement are either a shop rag or lint-free paper towel to clean the wipe-clean transmission cell. No other materials or reagents are necessary.

## 9. Hazards

9.1 The apparatus utilizes a certified Li-Ion battery.

## 10. Sampling, Test Specimens, and Test Units

10.1 A sample of in-service fluid should be obtained. A minimum quantity of approximately 50 µL is needed to obtain one set of measurements as defined in Table 2. The sample should be representative of the system. If such equipment is available, the sample is preferably obtained as described in Practice D4057.

## 11. Preparation of Apparatus

11.1 A quality collection of the infrared absorbance spectrum is assured by several internal quality checks, which include check fluid, pathlength, clean cell, and loaded cell monitoring.

11.1.1 Check fluid and pathlength monitoring (described in Practice D7418) are performed on a periodic basis according to manufacturer's recommendations.

11.1.2 In order to verify that the wipe-clean transmission cell is empty and clean, a cell background is taken in real-time and a raw absorbance spectrum is calculated using a previously archived, known, empty, and clean cell background.

11.1.2.1 By measuring the maximum peak height between 3000 cm$^{-1}$ and 2800 cm$^{-1}$ relative to a baseline at 2700 cm$^{-1}$, it can be determined whether the cell is clean. When the absorbance value is greater than a pre-set limit of 0.2 AU, the cell is considered not clean.

11.1.2.2 This check is performed before any cell background to be used in the calculation of fluid properties is obtained, and the user is warned if the check fails.

11.1.3 A cell loading check (as described in Practice D7418) is performed on each loaded sample to ensure that the cell is fully loaded. Such a check is performed according to manufacturer's recommendations.

11.1.3.1 If the cell is determined to not be fully loaded, a message that the cell is not fully loaded is displayed alongside the results of the analysis. It should be noted that due to the nature of the design for field use, the wipe clean transmission cell has no need for fringe correction.

11.2 Ensure that wipe-clean transmission cell is clean by visual inspection.

11.3 Gather approximately two drops of sample (on the order of 50 µL).

11.3.1 This may be accomplished using a dropper, syringe, or simply by using drops from a fluid dipstick, for example.

## 12. Calibration and Standardization

12.1 A check fluid is supplied with the apparatus as a qualification test. This fluid should be run after each set of 100 samples to ensure that the unit remains within calibration.

12.2 If this qualification test fails, the user should follow manufacturer's recommendations regarding calibration and standardization.

## 13. Conditioning

13.1 The field apparatus does not require conditioning nor does the sample being analyzed.

## 14. Procedure

14.1 Whenever prompted, collect a cell background on the device.

14.1.1 The cell background must be taken periodically according to (*a*) the time from the last cell background (must not be greater than 30 min) and (*b*) the current device temperature relative to the temperature at which the previous cell background was obtained (must not be greater than 3 K) exceeding maximum allowable thresholds.

14.2 Place the two drops of in-service fluid on the wipe-clean transmission cell.

14.2.1 Once the wipe-clean transmission cell is loaded with sample, the user initiates a measurement. This measurement comprises gathering the single-beam infrared spectrum of the fluid.

14.3 From that single-beam spectrum (=P(counts(ν))), the last saved cell background (=B(counts(ν))), and a pathlength normalization of the resulting spectrum (=PL(microns)), the infrared absorbance spectrum (=A(ν)) of the fluid is calculated by the apparatus:

$$A(\nu) = -(100/\mathrm{PL})\cdot\log_{10}(\mathrm{P}(\nu)/\mathrm{B}(\nu))\ [\mathrm{Abs/0.1\ mm}] \qquad (1)$$

14.3.1 The units of A(ν), the infrared absorbance spectrum, as indicated are Abs/0.1 mm.

## 15. Calculation or Interpretation of Results

15.1 Calculation of in-service fluid properties proceeds according to the spectral analysis detailed in Table 2. These analyses proceed using the signature infrared absorption bands gleaned from the obtained infrared absorbance spectrum of the fluid.

15.1.1 Properties are obtained from the absorbance spectrum using the measurement and baseline regions listed in Table 2. Properties are presented to the user in the reporting units specified.

15.1.1.1 Oxidation is obtained from calculating the average absorbance (Abs/0.1 mm) in the measurement region between 1800 $\mathrm{cm}^{-1}$ and 1670 $\mathrm{cm}^{-1}$ $\mathrm{Abs}_{,av}$(1800 to 1670) and performing a weighted subtraction from the average baseline absorbance (Abs/0.1 mm) in the region between 1815 $\mathrm{cm}^{-1}$ and 1805 $\mathrm{cm}^{-1}$ $\mathrm{Abs}_{,av}$(1815 to 1805). The resulting units of measurement are Abs/0.1 mm:

$$\begin{aligned}\text{Oxidation} = {} & 130\cdot\mathrm{Abs}_{,av}(1800\ \text{to}\ 1670)\\ & -38.5\cdot\mathrm{Abs}_{,av}(1815\ \text{to}\ 1805)\ [\mathrm{Abs/0.1\ mm}]\end{aligned} \qquad (2)$$

15.1.1.2 Antiwear additive is obtained from calculating the average absorbance (Abs/0.1 mm) in the measurement region between 1025 $\mathrm{cm}^{-1}$ and 960 $\mathrm{cm}^{-1}$ $\mathrm{Abs}_{,av}$(1025 to 960) and performing a weighted subtraction from the average baseline absorbance (Abs/0.1 mm) in the region between 1060 $\mathrm{cm}^{-1}$ and 1030 $\mathrm{cm}^{-1}$ $\mathrm{Abs}_{,av}$(1060 to 1030) as well as the region between 1812 $\mathrm{cm}^{-1}$ and 1803 $\mathrm{cm}^{-1}$ $\mathrm{Abs}_{,av}$(1812 to 1803). The resulting units of measurement are Abs/0.1 mm:

$$\begin{aligned}\text{Antiwear Additive} = {} & 65\cdot\mathrm{Abs}_{,av}(1025\ \text{to}\ 960)\\ & -8.4\cdot\mathrm{Abs}_{,av}(1060\ \text{to}\ 1030)\\ & -10.8\cdot\mathrm{Abs}_{,av}(1812\ \text{to}\ 1803)[\mathrm{Abs/0.1\ mm}]\end{aligned} \qquad (3)$$

15.1.1.3 Sulfation is obtained from calculating the average absorbance (Abs/0.1 mm) in the measurement region between 1180 $\mathrm{cm}^{-1}$ and 1120 $\mathrm{cm}^{-1}$ $\mathrm{Abs}_{,av}$(1180 to 1120) and performing a weighted subtraction from the average baseline absorbance (Abs/0.1 mm) in the region between 1210 $\mathrm{cm}^{-1}$ and 1200 $\mathrm{cm}^{-1}$ $\mathrm{Abs}_{,av}$(1210 to 1200) as well as the region between 1115 $\mathrm{cm}^{-1}$ and 1105 $\mathrm{cm}^{-1}$ $\mathrm{Abs}_{,av}$(1115 to 1105). The resulting units of measurement are Abs/0.1 mm:

$$\begin{aligned}\text{Sulfation} = {} & 60\cdot\mathrm{Abs}_{,av}(1180\ \text{to}\ 1120) - 11\cdot\mathrm{Abs}_{,av}(1210\ \text{to}\ 1200)\\ & -5\cdot\mathrm{Abs}_{,av}(1115\ \text{to}\ 1105)[\mathrm{Abs/0.1\ mm}]\end{aligned} \qquad (4)$$

15.1.1.4 Nitration is calculated by measuring the absorbance (Abs/0.1 mm) of the sample at 1630 $\mathrm{cm}^{-1}$ Abs(1630), and performing a weighted subtraction from the average baseline absorbance (Abs/0.1 mm) in the region between 1607 $\mathrm{cm}^{-1}$ and 1597 $\mathrm{cm}^{-1}$ $\mathrm{Abs}_{,av}$(1607 to 1597) as well as the region between 1847 $\mathrm{cm}^{-1}$ and 1837 $\mathrm{cm}^{-1}$ $\mathrm{Abs}_{,av}$(1847 to 1837). The units of this calculation are Abs/0.1 mm. The final result is obtained by multiplying the result by 100, yielding a result in units of Abs/cm (100*Abs/0.1 mm = Abs/0.001 mm = Abs/cm). This allows for easy-to-read numerical results in a typical numerical range > 1 Abs/cm. The calculation is detailed as follows:

$$\begin{aligned}\text{Nitration} = {} & 100\cdot(\mathrm{Abs}\ (1630)\\ & -0.89\cdot\mathrm{Abs}_{,av}(1607\ \text{to}\ 1597)\\ & -0.11\cdot\mathrm{Abs}_{,av}(1847\ \text{to}\ 1837))\ [\mathrm{Abs/\ cm}]\end{aligned} \qquad (5)$$

15.1.1.5 Soot is calculated by measuring the absorbance of the sample at 2000 $\mathrm{cm}^{-1}$ (Abs/0.1 mm) and multiplying the result by 100, yielding a result in units of Abs/cm. This allows for numerical results in a typical range of 0-100 Abs/cm.

15.1.2 In the following we provide examples for each of the five (5) properties detailed in 15.1.1:

15.1.2.1 *Oxidation Example*—Fig. 1 presents a schematic illustration of the calculation for the oxidation property. In this case, the average value of absorbance in the oxidation measurement region is approximately 0.1216 Abs/0.1 mm, and the baseline average is -0.005 Abs/0.1 mm. This yields an oxidation value of 16 Abs/0.1 mm:

$$\text{Oxidation} = 130 \cdot (0.1216) - 38.5 \cdot (-\ 0.005) = 16\,[\text{Abs/0.1 mm}] \tag{6}$$

15.1.2.2 *Antiwear Additive Example*—Fig. 2 presents a schematic illustration of the calculation for the antiwear additive property. In this case, the average value of absorbance in the antiwear additive measurement region is approximately 0.385 Abs/0.1 mm, and the baseline averages are 0.298 Abs/0.1 mm and 0.165 Abs/0.1 mm for the low and high frequency baselines, respectively. This yields an antiwear additive value of 20.7 Abs/0.1 mm:

$$\begin{aligned}\text{Antiwear Additive} &= 65 \cdot (0.385) - 8.4 \cdot (0.298) - 10.8 \cdot (0.165) \\ &= 20.7\,[\text{Abs/0.1 mm}]\end{aligned} \tag{7}$$

15.1.2.3 *Sulfation Example*—Fig. 3 presents a schematic illustration of the calculation for the sulfation property. In this case, the average value of absorbance in the sulfation measurement region is approximately 0.258 Abs/0.1 mm, and the baseline averages are 0.168 Abs/0.1 mm and 0.285 Abs/0.1 mm for the low and high frequency baselines, respectively. This yields a sulfation value of 11.5 Abs/0.1 mm:

$$\begin{aligned}\text{Sulfation} &= 60 \cdot (0.258) - 11 \cdot (0.285) - 5 \cdot (0.168) \\ &= 11.5\,[\text{Abs/0.1 mm}]\end{aligned} \tag{8}$$

15.1.2.4 *Nitration Example*—Fig. 4 presents a schematic illustration of the calculation for the nitration property. In this case, the absorbance at 1630 cm$^{-1}$ is 0.246 Abs/0.1 mm, and the baseline averages are 0.22 Abs/0.1 mm and 0.004 Abs/0.1 mm for the low and high frequency baselines, respectively. This yields a nitration value of 5 Abs/cm:

$$\begin{aligned}\text{Nitration} &= 100 \cdot (0.246 - 0.89 \cdot (0.22) - 0.11 \cdot (0.004)) \\ &= 5\,[\text{Abs/cm}]\end{aligned} \tag{9}$$

15.1.2.5 *Soot Example*—Fig. 5 presents a schematic illustration of the calculation of soot. In this case, the absorbance at 2000 cm$^{-1}$ is 0.31 Abs/0.1 mm. Multiplying this value by 100 we obtain a soot value of 31 Abs/cm:

$$\text{Soot} = 100 \cdot (0.31) = 31\,[\text{Abs/cm}] \tag{10}$$

15.1.3 These methods are chosen so as to give the user a reported value which does not rely on any spectral reference. This is in keeping with the apparatus being used as a field tool.

15.1.4 Limits for each particular fluid of interest may be tailored considering the typical range of that particular fluid.

## 16. Report

16.1 Properties are calculated as described in Section 15 and reported in units as described in Table 2.

16.2 *Trending and Alarm Limits*—As defined by the user, the apparatus will acquire, report, and log the fluid measured properties on a periodic basis. The user may monitor this trending behavior and as well set alarm limits in the apparatus for each particular fluid being analyzed. Guidance on performing trending analysis and setting alarm limits on in-service lubricants is provided in Guide D7669 and Guide D7720.

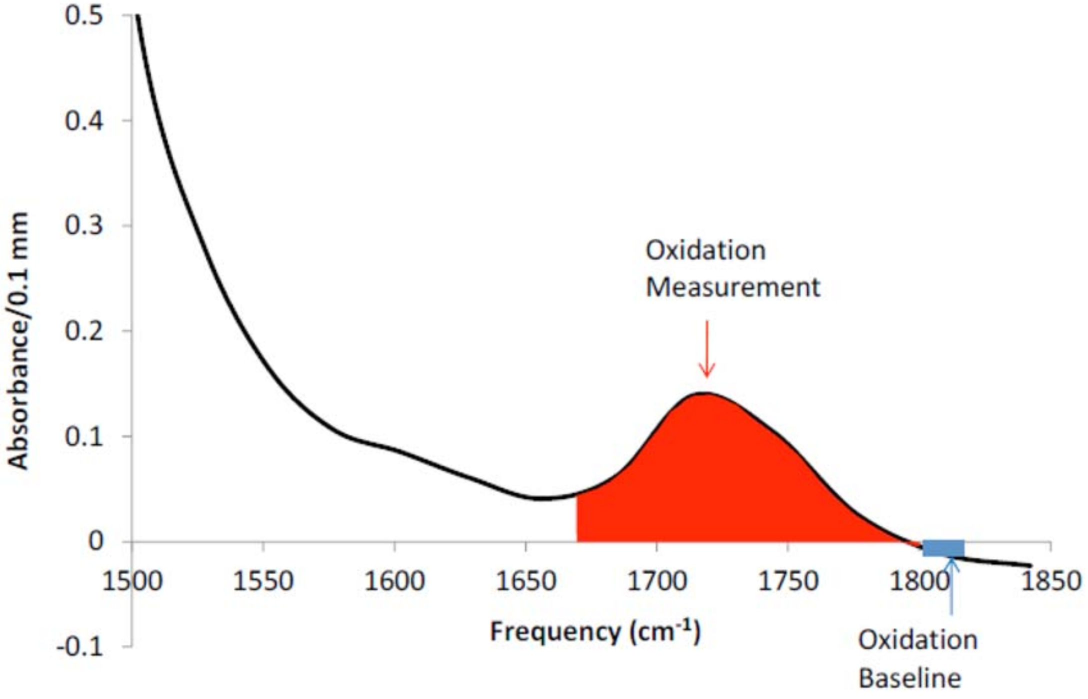

**FIG. 1 Illustration of Oxidation Measurement as Specified in Table 2**

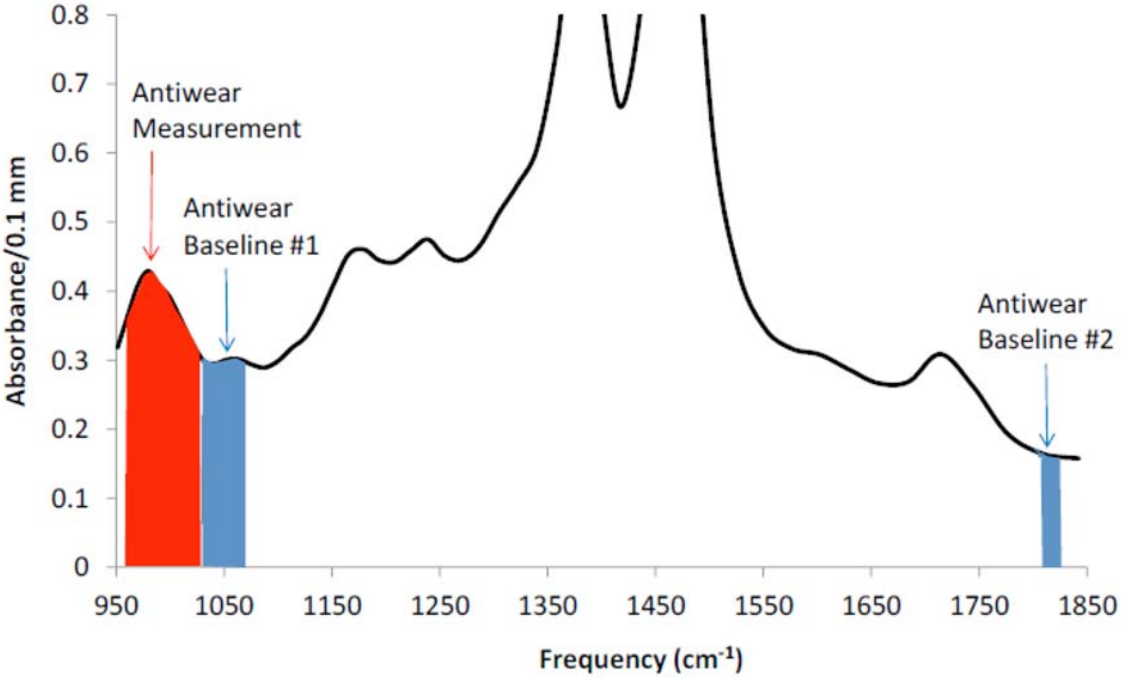

**FIG. 2 Illustration of Antiwear Additive Measurement as Specified in Table 2**

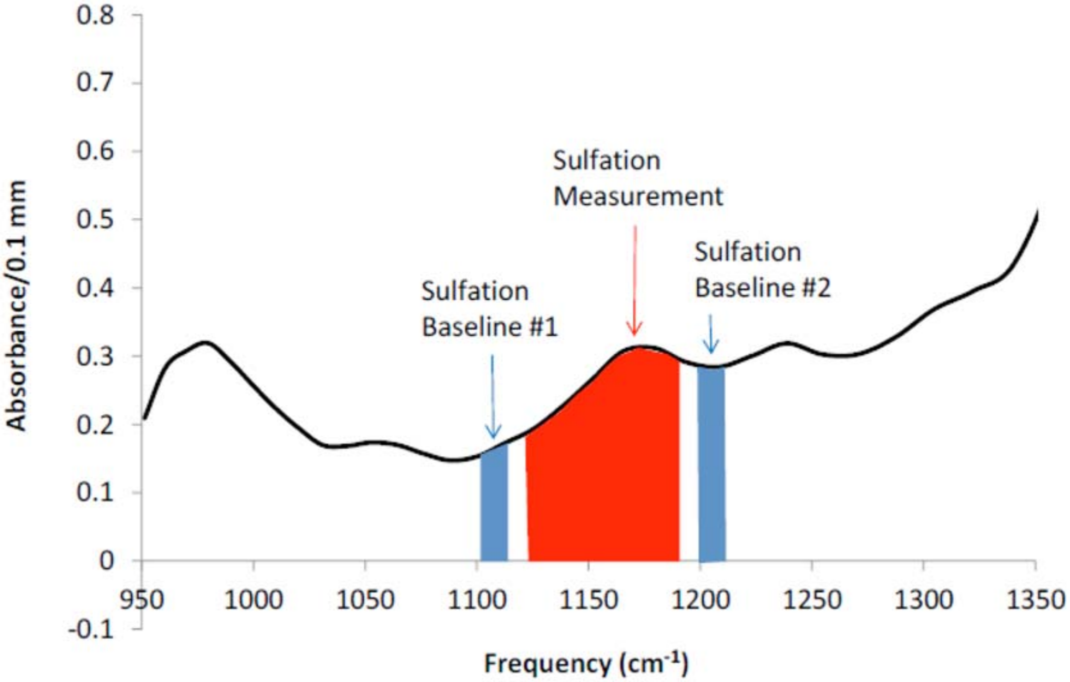

**FIG. 3 Illustration of Sulfation Measurement as Specified in Table 2**

16.3 *Effects of Fluid Formulation*—For each measured property, the reported values as well as the trending tendencies are in general different for each fluid type. The apparatus may be designed so that each fluid type may be tracked separately with trend histories being viewed and alarm limits set on the apparatus per these fluid types.

## 17. Precision and Bias

17.1 A temporary precision statement including repeatability is reported in Table 3 based on analyzing five samples tested thirty times each in the same laboratory. Full precision and bias

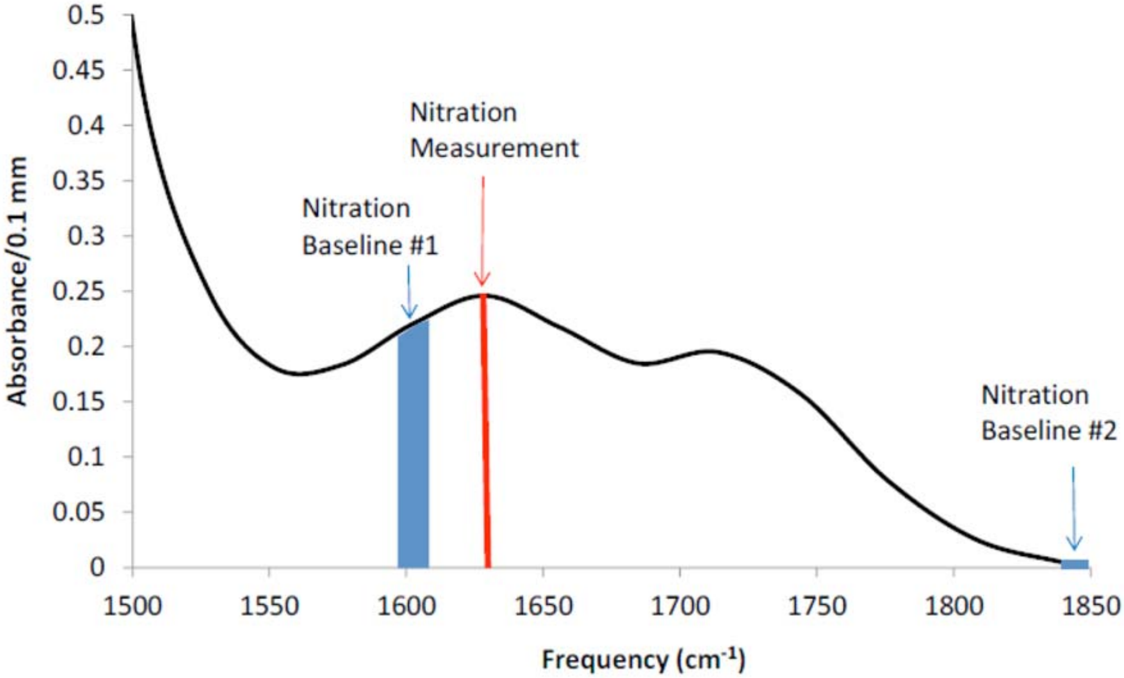

FIG. 4 Illustration of Nitration Measurement as Specified in Table 2

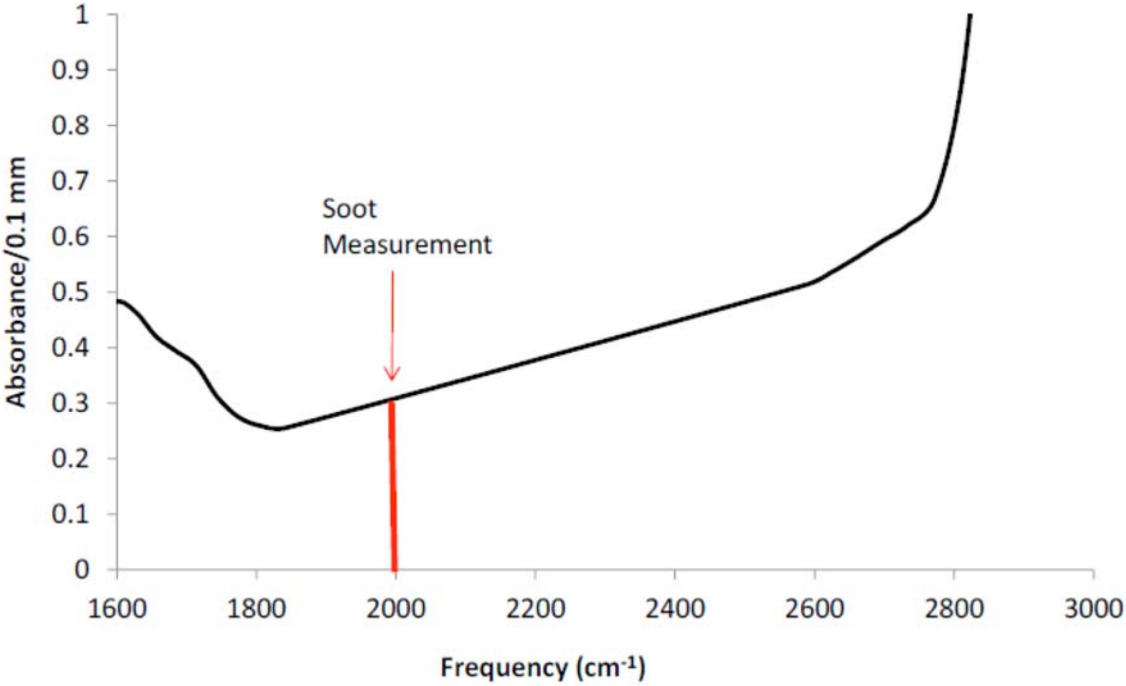

FIG. 5 Illustration of Soot Measurement as Specified in Table 2

TABLE 3 Temporary Precision Statement

| Property | Typical Repeatability | Lower Range | Upper Range | Units |
|---|---|---|---|---|
| Oxidation | 0.20 | 5.76 | 32.24 | Abs/0.1 mm |
| Nitration | 0.53 | 0.46 | 18.28 | Abs/cm |
| Sulfation | 0.31 | 15.81 | 39.45 | Abs/0.1 mm |
| Antiwear Additive | 0.38 | 9.67 | 16.57 | Abs/0.1 mm |
| Soot | 0.43 | -5.65 | 57.65 | Abs/cm |

statements based on interlaboratory round robin testing will be determined within five years of adoption of this standard method.

## 18. Keywords

18.1 antiwear additives; condition monitoring; direct trending; field-based devices; grating spectrometers; handheld devices; infrared spectroscopy; in-service fluid analysis; multivariate analysis; nitration; oxidation; soot; sulfation

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