Designation: D7415 – 09

Standard 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[1]

This standard is issued under the fixed designation D7415; 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 covers monitoring sulfate by-products in in-service petroleum and hydrocarbon based diesel crankcase engine and motor oils that have a sulfur content of greater than 500 ppm. This test method should not be employed when low-sulfur fuels are used for combustion.

1.2 This test method uses Fourier Transform Infrared (FT-IR) spectrometry for monitoring build-up of sulfate by-products in in-service petroleum and hydrocarbon based lubricants as a result of normal machinery operation. Sulfate by-products can result from the introduction of sulfur from combustion or from the oxidation of sulfur-containing base oil additives. This test method is designed as a fast, simple spectroscopic check for monitoring of sulfate by-products in in-service petroleum and hydrocarbon based lubricants with the objective of helping diagnose the operational condition of the machine based on measuring the level of sulfate by-products in the oil.

1.3 Acquisition of FT-IR spectral data for measuring sulfate by-products in in-service oil and lubricant samples is described in Practice D7418. In this test method, measurement and data interpretation parameters for sulfate by-products using both direct trend analysis and differential (spectral subtraction) trend analysis are presented.

1.4 This test method is based on trending of spectral changes associated with sulfate by-products of in-service petroleum and hydrocarbon based lubricants. Warnings or alarm limits can be set on the basis of a fixed minimum value for a single measurement or, alternatively, can be based on a rate of change of the response measured, see Ref (1).[2]

1.4.1 For direct trend analysis, values are recorded directly from absorption spectra and reported in units of absorbance per 0.1 mm pathlength.

1.4.2 For differential trend analysis, values are recorded from the differential spectra (spectrum obtained by subtraction of the absorption spectrum of the reference oil from that of the in-service oil) and reported in units of 100*absorbance per 0.1 mm pathlength (or equivalently absorbance units per centimetre).

1.4.3 In either case, maintenance action limits should be determined through statistical analysis, history of the same or similar equipment, round robin tests or other methods in conjunction with the correlation of sulfate by-product changes to equipment performance.

NOTE 1—It is not the intent of this test method 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.

1.5 This test method is for petroleum and hydrocarbon based lubricants and is not applicable for ester based oils, including polyol esters or phosphate esters.

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

1.6.1 *Exception*—The unit for wave numbers is cm^{-1}.

1.7 *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:*[3]

D445 Test Method for Kinematic Viscosity of Transparent

[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 on In-Service Lubricant Testing and Condition Monitoring Services.

Current edition approved July 1, 2009. Published August 2009. DOI: 10.1520/D7415-09.

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

[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.

and Opaque Liquids (and Calculation of Dynamic Viscosity)
D974 Test Method for Acid and Base Number by Color-Indicator Titration
D2896 Test Method for Base Number of Petroleum Products by Potentiometric Perchloric Acid Titration
D4739 Test Method for Base Number Determination by Potentiometric Hydrochloric Acid Titration
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
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
D7418 Practice for Set-Up and Operation of Fourier Transform Infrared (FT-IR) Spectrometers for In-Service Oil Condition Monitoring
E131 Terminology Relating to Molecular Spectroscopy
E2412 Practice for Condition Monitoring of In-Service Lubricants by Trend Analysis Using Fourier Transform Infrared (FT-IR) Spectrometry

3. Terminology

3.1 *Definitions*—For definitions of terms relating to infrared spectroscopy used in this test method, refer to Terminology E131. For definitions of terms related to in-service oil condition monitoring, refer to Practice D7418.

3.2 *machinery health, n*—qualitative expression of the operational status of a machine subcomponent, 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.

4. Summary of Test Method

4.1 This test method uses FT-IR spectrometry to monitor sulfate by-product in in-service petroleum and hydrocarbon based lubricants. The FT-IR spectra of in-service oil samples are collected according to the protocol for either direct trend analysis or differential trend analysis described in Practice D7418, and the levels of sulfate by-products are measured using the peak height or area measurements described herein.

5. Significance and Use

5.1 An increase in sulfate material can be an indicator of oil degradation caused by oxidation of sulfur in the oil and sulfur in fuel. It can also indicate the breakdown or oxidation of some key additives in the oil such as antiwear and extreme pressure additives as well as blow-by concerns. As oxidized sulfur from blow-by enters the lubricant, it will consume the overbase additive to generate sulfate by-products. Monitoring of sulfate by-products is therefore an important parameter in determining overall machinery health and in determining additive depletion and should be considered in conjunction with data from other tests such as atomic emission (AE) and atomic absorption (AA) spectroscopy for wear metal analysis (Test Method D5185), physical property tests (Test Methods D445, D2896, and D6304), base number tests (Test Methods D974 and D4739) and other FT-IR oil analysis methods for nitration (Practice E2412), oxidation (Test Method D7414), additive depletion (Test Method D7412), breakdown products and external contaminants (Practice E2412), which also assess elements of the oil's condition, see Refs (1-6)

6. Interferences

6.1 Various additive packages, especially those containing detergents, dispersants, demulsifiers and overbase additives, will interfere with the sulfate by-products measurement.

6.2 Contaminants such as esters, polyols, glycols and alcohols will also interfere with the measurement of sulfate by-products.

6.3 Oxidation by-products can be a major source of interference in the measurement of sulfate by-products. Because of this interference, the low levels of sulfate by-products associated with the use of low-sulfur fuels for combustion cannot be adequately measured.

7. Apparatus

7.1 Fourier transform infrared spectrometer equipped with sample cell, filter (optional) and pumping system (optional) as specified in Practice D7418.

7.2 *FT-IR Spectral Acquisition Parameters*—Set FT-IR spectral acquisition parameters according to instructions in Practice D7418.

8. Sampling

8.1 Obtain a sample of the in-service oil and a sample of the reference oil (required only for differential trend analysis) according to the protocol described in Practice D7418.

9. Preparation and Maintenance of Apparatus

9.1 Rinse, flush, and clean the sample cell, inlet lines, and inlet filter according to instructions in Practice D7418.

9.2 Monitor cell pathlength as specified in Practice D7418.

10. Procedure

10.1 Collect a background spectrum according to the procedure specified in Practice D7418.

10.2 *Differential Trend Analysis Only*—Collect the absorption spectrum of a reference oil sample according to the procedure specified in Practice D7418.

10.3 Collect the absorption spectrum of an in-service oil sample according to the procedure specified in Practice D7418.

10.4 *Data Processing*—All data are normalized to a pathlength of 0.100 mm according to the procedure specified in Practice D7418.