Designation: D7214 – 20

Standard Test Method for Determination of the Oxidation of Used Lubricants by FT-IR Using Peak Area Increase Calculation[1]

This standard is issued under the fixed designation D7214; 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 test method was jointly developed with "Groupement Francais de Coordination" (GFC), technical committee LM5 and "Coordinating European Council" (CEC) Surveillance Group T-048 for the purpose of monitoring the oxidation stability of artificially aged automotive transmission fluids. This test method has been used in the CEC L-48-A-00 method as an end of test measurement parameter.

1. Scope*

1.1 This test method covers the determination of the oxidation of used lubricants by FT-IR (Fourier Transform Infrared Spectroscopy). It measures the concentration change of constituents containing a carbonyl function that have formed during the oxidation of the lubricant.

1.2 This test method may be used to indicate relative changes that occur in an oil under oxidizing conditions. The test method is not intended to measure an absolute oxidation property that can be used to predict performance of an oil in service.

1.3 This test method was developed for transmission oils which have been degraded either in service, or in a laboratory test, for example a bulk oxidation test. It may be used for other in-service oils, but the stated precision may not apply.

1.4 The results of this test method may be affected by the presence of other components with an absorbance band in the zone of 1600 cm^{-1} to 1800 cm^{-1}. Low PAI values may be difficult to determine in those cases. Section 6 describes these possible interferences in more detail.

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.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products

D6299 Practice for Applying Statistical Quality Assurance and Control Charting Techniques to Evaluate Analytical Measurement System Performance

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

2.2 *CEC Standard:*

CEC L-48-A-00 Oxidation Stability of Lubricating Oils Used in Automotive Transmissions by Artificial Aging[3]

[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.

[3] Available from Coordinating European Council (CEC), c/o Interlynk Administrative Services, Ltd., P.O. Box 6475, Earl Shilton, Leicester, LE9 9ZB, U.K.

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

3. Terminology

3.1 *Definitions*—For terminology relating to molecular spectroscopic methods, refer to Terminology E131.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *carbonyl region, n*—region of the FT-IR spectrum corresponding to the absorbance of compounds containing a carbonyl function. Depending on the nature of the carbonyl compounds, this region is usually located between approximately 1820 cm^{-1} and 1650 cm^{-1}.

3.2.2 *differential spectrum, n*—FT-IR absorbance spectrum resulting from the subtraction of the fresh oil from the used oil.

3.2.3 *PAI (peak area increase), n*—area of the carbonyl region of the differential FT-IR spectrum, divided by the cell pathlength in millimetres. In this standard, PAI refers to a relative measurement of the oxidation of a used lubricant by FT-IR.

4. Summary of Test Method

4.1 FT-IR spectra of the fresh oil and of the used oil are recorded in a transmission cell of known pathlength. Both spectra are converted to absorbance and then subtracted. Using the resulting differential spectrum, a baseline is set under the peak corresponding to the carbonyl region around 1650 cm^{-1} and 1820 cm^{-1} and the area created by this baseline and the carbonyl peak is calculated. The area of the carbonyl region is divided by the cell pathlength in millimeters and this result is reported as Peak Area Increase (PAI).

5. Significance and Use

5.1 The PAI is representative of the quantity of all the compounds containing a carbonyl function that have formed by the oxidation of the lubricant (aldehydes, ketones, carboxylic acids, esters, anhydrides, etc.). The PAI gives representative information on the chemical degradation of the lubricant which has been caused by oxidation.

5.2 This test method was developed for transmission oils and is used in the CEC L-48-A-00 test (Oxidation Stability of Lubricating Oils Used in Automotive Transmissions by Artificial Aging) as a parameter for the end of test evaluation.

6. Interferences

6.1 Some specific cases (very viscous oil, use of ester as base stock, high soot content) may require a dilution of the sample and a specific area calculation, which are described in 13.1 – 13.3. In those cases, the result is corrected by a dilution factor, which is applied to the sample.

7. Apparatus

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

7.1.1 *Transmission Cell,* with windows of potassium bromide, having a known pathlength of approximately 0.025 mm to 0.1 mm.

7.1.1.1 Other cell window types such as Zinc Selenide, with a known pathlength of approximately 0.025 mm to 0.1 mm, may also be used. ZnSe is known to have a greater durability in the presence of moisture.

7.1.2 *Syringe, Automated, or Semi-Automated Device (Pumping System),* with adequate volume to fill the cell.

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

8. Reagents and Materials

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

8.2 *Heptane,* used as cleaning solvent. Other solvents and solvent mixtures may be used provided they adequately clean the cell(s) between samples. A 50/50 mixture of cyclohexane and toluene has been found to be useful in cleaning cells after highly contaminated and degraded samples have been run. (**Warning—**Flammable.)

8.3 *PAO4,* used as dilution oil (PAO4: PolyAlphaOlefin with a kinematic viscosity at 100 °C of approximately 4 mm^2/s).

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 Determine the cell pathlength daily as specified in Practice D7418.

9.2.1 Cell path shall be determined whenever maintenance is performed on the cell.

9.3 *Instrument Performance Checks* shall be performed in accordance with Practice D7418.

10. Preparation of Sample of Used Oil

10.1 Refer to Practice D4057 (Manual Sampling) or Practice D4177 (Automatic Sampling) for proper sampling techniques.

10.2 When sampling used lubricants, the specimen shall be representative of the system sampled and shall be free of contamination from external sources. As used oil can change appreciably in storage, test samples as soon as possible after removal from the lubricating system and note the dates of sampling and testing.

10.3 If the sample of used oil contains visible sediment, heat to 60 °C ± 5 °C in the original container and agitate until all of the sediment is homogeneously suspended in the oil.

10.4 If the original container is a can or if it is glass and more than three-fourths full, transfer the entire sample to a clear-glass bottle having a capacity at least one third greater than the volume of the sample.