Designation: D7686 – 11

Standard Test Method for
Field-Based Condition Monitoring of Soot in In-Service Lubricants Using a Fixed-Filter Infrared (IR) Instrument[1]

1. Scope

1.1 This test method pertains to field-based monitoring of soot in diesel crankcase engine oils as well as in other types of engine oils where soot may contaminate the lubricant as a result of a blow-by due to incomplete combustion of fuels. It is applicable to oils having soot levels of up to 12%.

1.2 This test method uses filter-based infrared technology for monitoring of soot build-up in in-service petroleum and hydrocarbon-based lubricants as a result of normal machinery operation. Soot levels in engine oils rise as soot particles contaminate the oil as a result of exhaust gas recirculation from blow-by. This test method is designed as a fast, simple and field capable spectroscopic check for soot in in-service hydrocarbon-based lubricants with the objective of helping diagnose the operational condition of the machine based on measuring the level of soot in the oil.

1.3 This test method is intended as a field test only, and should be treated as such. Critical applications should use laboratory based methods, such as Thermal Gravimetric Analysis (TGA) described in Test Method D5967, Annex A4.

1.4 Acquisition of spectral data for measuring soot in in-service oil and lubricant samples with the use of a fixed-filter IR instrument is described in this test method. Calibration against prepared soot standards is also described.

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 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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D5967 Test Method for Evaluation of Diesel Engine Oils in T-8 Diesel Engine

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

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.

3.2 *Definitions*—For definition of terms related to in-service oil condition monitoring, refer to Practice D7418.

4. Summary of Test Method

4.1 This test method uses filter-based infrared spectrometry to monitor levels of soot in in-service petroleum and hydrocarbon based lubricants. The test method is meant to serve as a field-based method to provide an indicator of soot level. A well homogenized sample of oil is applied to a Horizontal Attenuated Total Reflectance (HATR) crystal. Infrared light is totally internally reflected through the HATR crystal and then passed through a broadband infrared filter centered on 3.9 µm. The amount of infrared light reaching a detector is converted to a soot concentration via a calibration curve generated from the instrument response on various soot standards.

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[1] This test method 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.

5. Significance and Use

5.1 This test method provides a simple field-based technique for condition monitoring of soot in in-service lubricants associated with combustion engines, machinery, and equipment used in industry and by the military. Critical applications should use laboratory based test methods, such as Thermal Gravimetric Analysis (TGA) described in Test Method D5967, Annex A4. Infrared spectroscopy is a well established laboratory method for evaluating soot levels in lubricants. This test method can be used to monitor soot build-up in lubricants and can indicate whether soot has accumulated to an extent which could significantly degrade the performance of the oil. High soot content can compromise lubricant performance and cause filter and oil passage blockage. Soot concentration should be considered in conjunction with data from other condition monitoring tests as described in Practice E2412 to determine whether the oil should be replaced to minimize machinery wear or failure, or both.

6. Interferences

6.1 Some types of dirt or wear debris may cause a positive bias. In most cases the bias will be small and will likely also affect Thermal Gravimetric Analysis (TGA) as described in Test Method D5967.

6.2 Large amounts of free water in an oil sample may cause a positive bias.

7. Apparatus

7.1 *Filter Based Infrared Spectrometer:*

7.1.1 *Infrared Spectrometer*—The type of apparatus suitable for this test method consists of a tungsten source, a 4 reflection cubic zirconia horizontal attenuated total reflectance cell, an infrared filter paired to a pyroelectric detector, an A/D converter and a microprocessor capable of storing an internal calibrations and converting instrument response into soot concentration.

7.1.2 *Infrared Filter Specifications*—A broadband infrared filter with a peak wavelength centered between 3.9 to 4.0 µm, and a filter half width of 0.15 ± 0.05 µm shall be used.

7.1.3 Other spectrometer configurations may provide adequate results; however the precision and bias data listed with this test method was collected based on these apparatus specifications. Any modifications may result in precision and or bias that differ from the numbers listed in this test method.

8. Reagents and Materials

8.1 *Calibration Standards*—Calibration standards shall consist of unused and soot–free 15w-40 diesel motor oil adulterated with various concentrations of sub micron particle size, high structure carbon black dispersed in the oil and mixed thoroughly. The resulting soot concentration shall be determined by Thermal Gravimetric Analysis (TGA) as described in Test Method D5967. Five standards uniformly covering a range of approximately 0 to 12% soot are required. Precision information for Thermal Gravimetric Analysis (TGA) in accordance with Test Method D5967 can be found in Annex A1.

9. Sampling, Test Specimens, and Test Units

9.1 *General Requirements:*

9.1.1 It is recommended that in-service oil or lubricant samples to be analyzed by this test method are sampled using procedures outlined in Practice D7418.

9.1.2 Protect samples from excessive temperatures prior to testing.

9.1.3 Do not test samples stored in leaky containers. Discard and obtain a new sample if leaks are detected.

9.2 When analyzing samples using this test method, shake the sample vigorously until the sample is adequately homogenized and no sediment is adhered to the bottom or sides of the vessel.

10. Preparation of Apparatus

10.1 Before use, the instrument needs to be calibrated according to the procedure described in Section 11. This calibration can be performed by the instrument manufacturer prior to delivery of the instrument to the end user.

10.2 Before analyzing samples, the instrument should be allowed to warm up for at least 1 h to ensure the analyzer is stabilized.

10.3 Before use, the instrument should be qualified by running quality control check samples consisting of a known soot concentration as determined by Test Method D5967 that span the range that the instrument is calibrated in. These samples should consist of at least one sample in the bottom third of the range, one sample in the middle third of the range and one sample in the top third of the range. Apply the control charting procedures described in Practice D6299 to establish that the analyzer is in-statistical-control. Any system that is found to be out of statistical control cannot be used until the root cause(s) of out-of-control is identified and corrected.

11. Calibration and Standardization

11.1 *Calibration Standards*—Refer to 8.1 for calibration standard description.

11.2 *Calibration:*

11.2.1 Equilibrate all samples to the temperature of the laboratory (15°C to 27°C) prior to analysis

11.2.2 Allow the instrument to warm up for at least one hour before attempting a calibration.

11.2.3 Calibration standards should be run in a randomized order.

11.2.4 Clean the HATR sample crystal of any residual oil or other contamination by applying about 1 mL of unused and soot–free oil to the crystal and wiping across the crystal in one direction with a disposable wipe. Avoid rubbing back and forth since this can recontaminate the crystal.

11.2.5 Introduce approximately 1 mL of unused, soot–free oil sample of the same type as the oils to be analyzed to the instrument, ensuring that the crystal is completely covered with sample. Remove any bubbles from the sample if needed.

11.2.6 Obtain a baseline detector response (zero) according to the manufacturer's instructions with the unused oil sample on the crystal. A zero should be obtained before the first use and once every two hours the instrument is in use.

11.2.7 Wipe the sample off using a disposable wipe once the sampling is complete.

11.2.8 Shake the first calibration standard vigorously until the sample is adequately homogenized and no sediment is adhered to the bottom or sides of the vessel.

11.2.9 Introduce approximately 1 mL of the first calibration standard to the instrument, ensuring that the crystal is completely covered with sample. Remove any bubbles from the sample if needed by piercing the bubbles with the sample applicator.

11.2.10 Obtain an infrared detector response according to the manufacturer's instructions and divide this by the baseline detector response. The ratio may be calculated internally by the instrument. The acquisition of a detector response should be initiated within 10 s of placing the sample on the crystal in order to reduce any errors due to sample settling.

11.2.11 Record the resulting response for later use in determining the calibration curve.

11.2.12 Wipe the sample off using a disposable wipe, then apply about 1 mL of unused and soot–free oil to the crystal and wipe across the crystal in one direction with a disposable wipe to clean the crystal of any soot residue. Avoid rubbing back and forth since this can recontaminate the crystal.

11.2.13 Repeat 11.2.7-11.2.11 for each calibration standard. Standards should be run in a randomized order.

11.2.14 Repeat steps 11.2.3-11.2.12 twice more, so that each calibration standard is run three times. Average the three instrument responses recorded for each calibration standard and compute the difference from the mean for each of the three responses. If a response differs by more than 0.1x + 2 mAU from the mean response for that standard, where x is the particular instrument response value, then the value should be excluded as an outlier and another standard run at that concentration level.

11.2.15 After removal of outliers, compute the average response at each concentration level. These concentration versus response values should determine a point to point curve, with a linear fit applied between adjacent points. This curve should be used to convert instrument response values to weight % soot. The point to point curve may be generated by the instrument microprocessor.

12. Procedure

12.1 Equilibrate the samples to between 15°C and 27°C before analysis.

12.2 Clean the HATR sample crystal of any residual oil or other contamination by applying about 1 mL of unused and soot–free oil to the crystal and wiping across the crystal in one direction with a disposable wipe. Avoid rubbing back and forth since this can recontaminate the crystal.

12.3 Introduce approximately 1 mL of an unused, soot–free sample of the same type as the oils to be analyzed to the instrument, ensuring that the crystal is completely covered with sample. Remove any bubbles from the sample if needed by piercing the bubbles with the sample applicator.

12.4 Obtain a baseline detector response (zero) according to the manufacturer's instructions with the unused oil sample on the crystal. A zero should be obtained before the first use and once every two hours the instrument is in use.

12.5 Wipe the sample off using a disposable wipe once the sampling is complete.

12.6 Shake unknown samples vigorously until the sample is adequately homogenized and no sediment is adhered to the bottom or sides of the vessel.

12.7 Introduce approximately 1 mL of unknown sample to the instrument, ensuring that the entire crystal surface is covered with sample. Remove any bubbles from the sample if needed by piercing the bubbles with the sample applicator.

12.8 Obtain an infrared detector response and divide this by the baseline detector response according to the manufacturer's instructions. The ratio may be calculated internally by the instrument. The acquisition of a detector response should be initiated within 10 seconds of placing the sample on the crystal in order to reduce any errors due to sample settling.

12.9 Determine the soot concentration according to the point-to-point calibration generated in Section 11. This may be automatically computed by an instrument microprocessor.

12.10 Wipe the sample off using a disposable wipe, then apply about 1 mL of unused and soot–free oil to the crystal and wipe across the crystal in one direction with a disposable wipe to clean the crystal of any soot residue. Avoid rubbing back and forth since this can recontaminate the crystal.

13. Report

13.1 Report the following information:

13.1.1 Weight % Soot by Test Method D7686, to the nearest 0.1%.

14. Precision and Bias

14.1 *Precision*—The repeatability has been determined to be:

$$0.016\ X^{1.2}\ \text{wt}\ \% \qquad (1)$$

where X is the calculated soot concentration in weight %. The reproducibility of this test method is being determined and will be available on or before December 1, 2014.

15. Keywords

15.1 condition monitoring; diesel engine oil; hydrocarbon-based lubricants; infrared; in-service petroleum lubricants; IR; lubricants; oils; soot

ANNEX

(Mandatory Information)

A1. TEST METHOD D5967 SOOT PRECISION DATA

A1.1 The Thermal Gravimetric Analysis (TGA) found in Test Method D5967 does not currently contain any precision data. To facilitate the use of this test method to quantify calibration standards, the precision data in Table A1.1 is provided courtesy of ASTM Test Monitoring Center, Carnegie Mellon University, 6555 Penn Avenue, Pittsburgh, PA 15206, http://www.astmtmc.cmu.edu.

TABLE A1.1 Test Method D5967 TGA Soot Statistics

Date	Sample ID	Avg Soot, %	r	R
Mar-95	NO2	0.1	0.11	0.25
	UO2	4.8	0.14	0.18
	UO3	4.3	0.09	0.16
Jun-95	NO2	0.1	0.14	0.33
	UO4	4.0	0.19	0.26
	UO5	3.9	0.11	0.28
Sep-95	NO2	0.1	0.05	0.22
	UO6	4.3	0.09	0.22
	UO7	5.0	0.14	0.27
Dec-95	NO2	0.1	0.13	0.27
	UO8	4.5	0.11	0.29
	UO9	3.8	0.08	0.21
Mar-96	NO2	0.1	0.11	0.30
	UO10	4.1	0.10	0.22
	UO11	4.2	0.09	0.19
Jun-96	NO2	0.1	0.13	0.26
	UO12	4.6	0.14	0.24
	UO13	4.2	0.17	0.26
Sep-96	NO2	0.1	0.17	0.30
	UO14	4.6	0.09	0.29
Dec-96	NO2	0.2	0.10	0.33
	UO15	4.3	0.12	0.33
	UO16	3.850	0.18	0.31
Apr-97	NO2	0.2	0.10	0.30
	UO17	3.7	0.13	0.28
	UO18	5.0	0.13	0.30
Oct-97	UO21	4.8	0.09	0.34
	UO22	5.8	0.10	0.20
Jan-98	UO23	1.8	0.26	0.36
May-98	UO24	2.0	0.10	0.23
	UO25	5.1	0.18	0.28
Jul-98	UO16	3.8	0.08	0.19
	UO18	4.9	0.12	0.29
Nov-98	UO26	2.3	0.10	0.22
	UO27	4.4	0.08	0.14
Mar-99	UO28	2.5	0.11	0.24
	UO29	4.1	0.14	0.23
Jun-99	UO30	1.8	0.12	0.23
	UO31	5.1	0.11	0.13
Sep-99	UO17	3.7	0.10	0.25
	UO27	4.4	0.12	0.21
Dec-99	UO19	4.0	0.13	0.33
	UO28	2.5	0.13	0.39
Jul-00	UO32	7.2	0.10	0.27
	UO33	6.6	0.12	0.27
Jan-01	UO34	5.3	0.07	0.18
	UO37	9.0	0.08	0.24
Mar-02	UO38	6.0	0.12	0.21
	UO39	5.6	0.11	0.20
	UO40	6.8	0.11	0.22
	UO41	5.6	0.10	0.26
	UO42	6.0	0.11	0.25
Oct-02	UO43	4.6	0.13	0.26
	UO44	6.2	0.15	0.29
	UO45	6.5	0.13	0.27
	UO46	6.5	0.15	0.35
			r	R
Averages[A]			0.12	0.26

[A] Note that there is no observed concentration level dependence for r or R.

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