Designation: D7899 – 13

Standard Test Method for Measuring the Merit of Dispersancy of In-Service Engine Oils with Blotter Spot Method[1]

This standard is issued under the fixed designation D7899; 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 a procedure for determination of the merit of dispersancy of diesel crankcase engine oils as well as other types of engine oils where pollutants of diverse sources such as soot from combustion, metallic particles from wear, corrosion of mechanical parts, and insoluble products resulting from the oxidation of the oil may contaminate the lubricant.

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

1.3 *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.*

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.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

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

3. Terminology

3.1 *Definitions:*

3.1.1 *diesel crankcase engine oils, n*—an engine oil used in the crankcase of the internal combustion diesel engine.

3.1.1.1 *Discussion*—It may contain additives to enhance certain properties. Inhibition of engine rusting, deposit formation, valve train wear, oil oxidation, and foaming are examples.

3.1.2 *diesel engine, n*—a reciprocating or rotary engine in which ignition of the main fuel charge, as it is introduced to the combustion chamber, shall be by the heat of compression of the charge of combustion air, during regular operation of the engine from idle speeds up to full speed, regardless of whether miscellaneous methods to augment such heat of compression are used to facilitate starting of the engine under normal conditions or under low ambient temperature conditions or low intake air temperature conditions.

3.1.2.1 *Discussion*—Engines that are designed to operate with a continuously hot spot or bulb or other device to facilitate ignition or combustion, or both, of low cetane fuels, or any fuels slow to ignite or to burn, or both, shall be considered to be diesel engines for purposes of this test method.

3.1.3 *engine oil, n*—a liquid that reduces friction or wear, or both, between the moving parts within an engine; removes heat, particularly from the underside of pistons; and serves as a combustion gas sealant for piston rings.

3.1.3.1 *Discussion*—It may contain additives to enhance certain properties. Inhibition of engine rusting, deposit formation, valve train wear, oil oxidation, and foaming are examples.

3.1.4 *oxidation, n*—of engine oil, the reaction of the oil with an electron acceptor, generally oxygen, which can produce deleterious acidic or resinous materials often manifested as sludge formation, varnish formation, viscosity increase, or corrosion, or a combination thereof.

3.1.5 *sludge, n*—in internal combustion engines, a deposit, principally composed of insoluble resins and oxidation products from fuel combustion and the lubricant that does not drain from engine parts but can be removed by wiping with a cloth.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *dispersancy*—the property that allows oil to suspend and carry away pollutants of diverse sources such as soot from combustion, metallic particles from wear, corrosion of mechanical parts, and insoluble products resulting from the aging of the oil.

[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 D02.96.02 on Chemistry for the Evaluation of 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.

4. Summary of Test Method

4.1 A drop of oil is deposited on a piece of specific filter paper held level and not resting on a surface. The filter paper is placed in an oven set at 80 °C for 1 h. The oil wicks across the paper. The oil spot is illuminated with a constant and homogenous LED (light emitting diodes) backlight. A CCD (charge-coupled device) camera positioned on the other side of the paper takes a picture of the spot in black and white mode. The software analyzes each pixel of the picture. Dispersancy characteristics of the oil are judged by how far the oil drop spreads, how large the central sooty area is, and how homogeneous the opacity of the spot is in comparison with a theoretical reference diameter of 32 mm.

5. Significance and Use

5.1 Dispersancy is the property that allows oil to suspend and carry away pollutants of diverse sources such as soot from combustion, metallic particles from wear, corrosion of mechanical parts, and insoluble products resulting from the aging of the oil.

5.2 When poured on a specific filter paper, oil that is properly dispersing soot and other insolubles produces an evenly graduated spot. The distribution of the different zones (Fig. 1) will reflect the status of oil dispersancy.

5.3 While the oil spreads out on the filter paper, the oil carries contaminants, and due to the lamination phenomenon of the oil film, the particles of same size deposit on the paper on the same concentric zones.

5.4 This test method provides a simple technique for condition monitoring of the dispersancy property of in-service lubricants.

5.5 An oil that is properly dispersing soot and other insolubles produces an evenly graduated blotter (see Fig. 2—Spot 1). A ring of light debris on the outer circumference of the circular spot also indicates that the oil has retained its dispersancy properties.

5.6 A blotter indicating a high soot load, but even graduation, suggests the oil is still fit for service, but should be watched closely for degradation (see Fig. 2—Spot 2).

5.7 When dispersancy begins to fail, the insolubles begin to form a dense ring on the exterior of the absorbing oil drop as in Fig. 2—Spot 3. A brown or yellow stain on the blotter spot indicates oxidation.

5.8 Fig. 2—Spot 4 indicates the characteristic dense black dot and sharp periphery that indicates sludge and the loss of dispersancy as the particles have settled in the center and the oil has wicked outward.

5.9 From a maintenance perspective, when the ring begins to form around the exterior of the oil blotter, it is time to look at scheduling a drain. If the black dot is allowed to form, the situation is problematic because the undispersed portion of soot

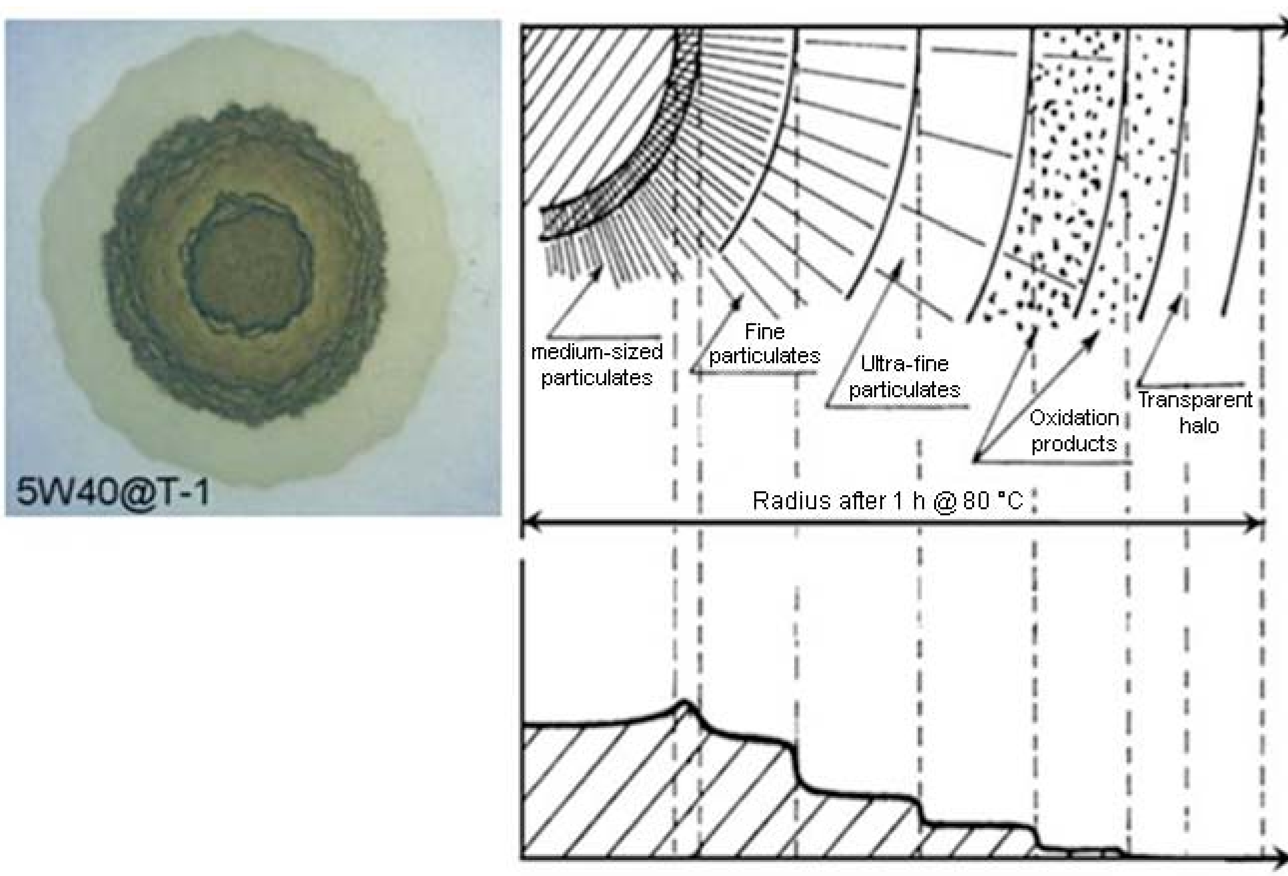

FIG. 1 Oil Spot Example and Scheme of the Distribution of the Different Zones

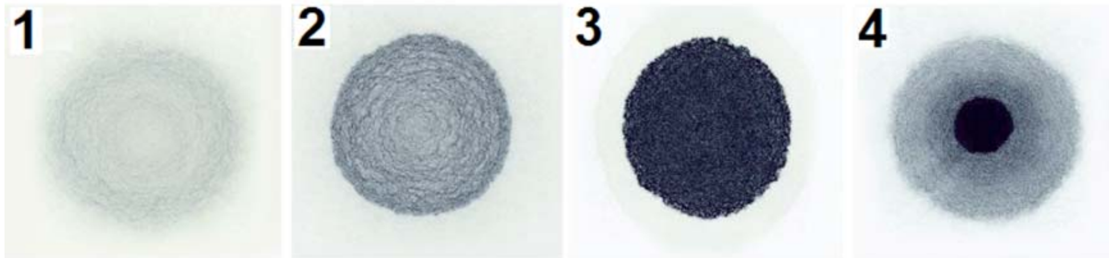

FIG. 2 Oil Spot Examples

that has deposited upon surfaces will not be removed by the oil change. Often, several changes made at frequent intervals will be required to effectively scour the engine clean. Also, if dispersancy performance degrades at an unusually rapid pace, a more extensive review of combustion and ring performance should be undertaken.

6. Apparatus

6.1 *Positive Displacement Pipette,* Class A to deliver 20 µl.

6.2 *Filter Paper,* having the following characteristics:

Paper density	84
Paper thickness	0.16 mm
Type	Qualitative
Particle retention	<2 µm
Filtration	Slow
Filtration Herzberg	750
Material	cotton liners and cellulose

NOTE 2—The filter paper characteristics are critical for the quality of the results.

6.3 *Filter Paper Holder,* made of two parts of rigid material, with 50 mm ± 1 mm holes, the filter paper is positioned between the upper part and the lower part. The minimum distance between the holes shall be 65 mm to prevent the oil spots from overlapping. See Fig. 3.

6.4 *Oven,* capable of maintaining a temperature of 80 °C ± 4 °C. It shall be equipped with shelves to position the paper holders. It shall not be equipped with forced air convection.

6.5 *Dispersancy Tester*[3], (Fig. 4) shall be equipped with backlight illumination, a digital camera connected to a computer to analyze and record the oil spot image, and the ability to compute the dispersancy number according to the described method.

6.5.1 *Digital Camera, associated to its dedicated software,* type WVGA shall have a minimum resolution of 752 × 480 pixels.

6.5.2 *Illumination Source,* shall provide a uniform light on the entire surface of the oil spot.

6.6 *Neutral Density Filter or ND Filter,* for instrument calibration, to be used to reduce the intensity of light by a definite ratio, without affecting the tonal rendition of colors.

6.6.1 Neutral density filter with 50 % light transmission.

6.6.2 Neutral density filter with 10 % light transmission.

NOTE 3—Kodak (trademark) Wratten2 filters[4] have been found satisfactory for this application.

6.7 *Reference Spots,* two reference spots printed in black on the filter paper specified in 6.2, one with a diameter of 35 mm ± 0.2 mm, one with a diameter 20 mm ± 0.2 mm.

NOTE 4—A special calibration kit including the two filters and two reference spots is available from the instrument manufacturer.

7. Sampling and Preparation of Samples

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

7.2 Protect samples from excessive temperatures prior to testing.

7.3 Do not test samples stored in containers with significant leakage. Discard and obtain a new sample if significant leaks are detected.

7.4 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 container.

8. Preparation of Apparatus

8.1 Before use, the instrument needs to be calibrated according to the procedure described in Section 10. This calibration can be performed by the end user.

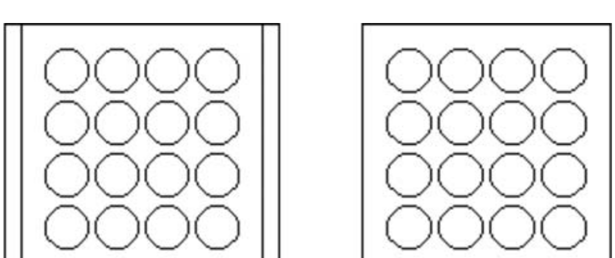

FIG. 3 Filter Paper Holder

[3] The sole source of supply of the apparatus (model DT 10 and DT 100DL—Dispersancy Tester) known to the committee at this time is AD Systems, P.A. Portes de la Suisse Normande, Allée de Cindais, 14320 Saint andré sur Orne, France, http://www.adsystems-sa.com. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

[4] The sole source of supply of the apparatus known to the committee at this time is Kodak Wratten2, http://www.kodak.com. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

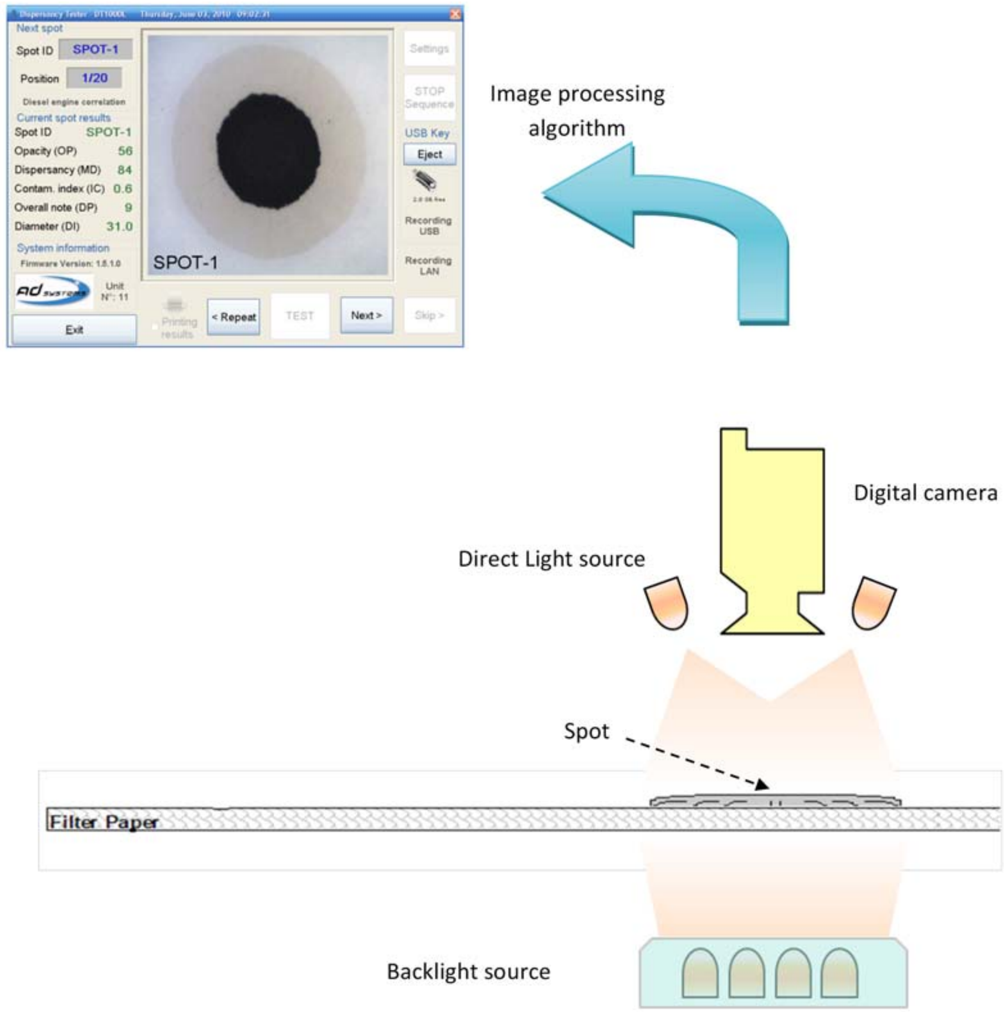

FIG. 4 Dispersancy Tester Principle

8.2 Before analyzing samples, the instrument should be allowed to warm up for at least 15 min to ensure that the analyzer is stabilized.

9. Calibration of Apparatus

9.1 The calibration of the instrument shall be performed every time it is switched on. Perform the calibration according to the manufacturer's instructions. The camera brightness and contrast are automatically adjusted by measuring the two standard filters specified in 6.6. The spot dimension measurement is verified by measuring the printed spots specified in 6.7.

10. Procedure

10.1 Position a new sheet of filter paper in the filter paper holder and place it on the bench.

10.2 Shake the sample vigorously until the sample is adequately homogenized and no sediment is adhered to the bottom or sides of the container.

10.3 With the positive displacement pipette, sample 20 µl of sample, wipe the outer surface of the pipette tip from excess of oil, and inject the sample in the center of the hole provided in the holder.

10.4 If several different samples are tested, repeat steps 10.2 and 10.3 with all samples to be tested (Fig. 5). Several different oil spots can be prepared on the same sheet. If this is the case, it is recommended to write the sample identification under each spot.

10.5 The paper holder with the paper sheet is then placed in horizontal position for 60 min ± 2 min in a laboratory drying oven adjusted to 80 °C.

10.6 After 60 min ± 2 min, remove the paper holder from the oven. (**Warning—**The paper holder surface is at 80 °C. Use protecting gloves to take the holder out of the oven.)

10.7 The paper filter is extracted from the holder. After the 60 min drying phase at 80 °C, the oil spot may continue its expansion. In order to obtain precise results, the oil spots must be analyzed within a maximum of 60 min after removal from the oven.

10.8 Key in all sample details. Position the oil spot in front of the CCD camera and the image of the oil spot is displayed. The oil spot must be positioned within the displayed red circle. Press the test key to initiate the test. For more details, refer to the instruction manual of the apparatus manufacturer.

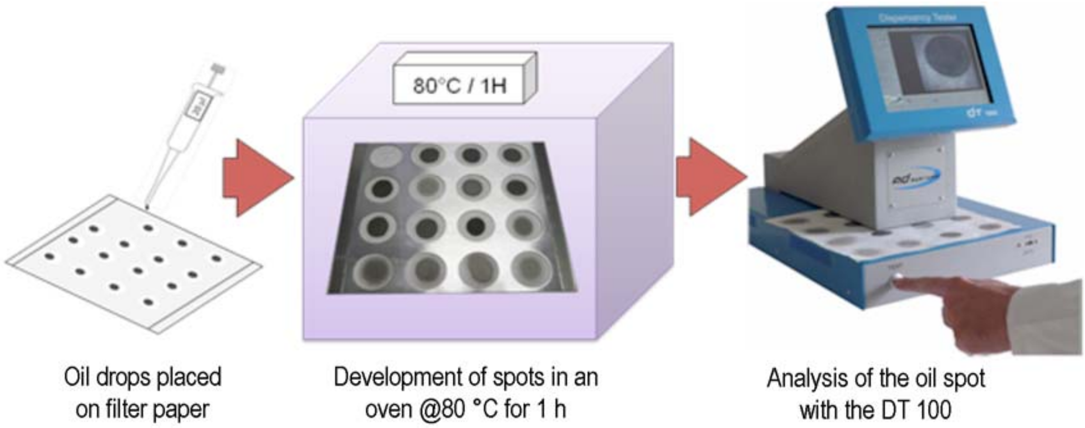

FIG. 5 Synoptic Showing the Oil Spot Preparation

10.9 Once the test is initiated, the following steps will be achieved:

10.9.1 *Step 1*—Oil Spot detection and size measurement.

10.9.1.1 The software determines the edges and the center of the oil spot. *r1* and *r2* in Fig. 6 are determined by converting in mm the number of pixels on *r1* and *r2* lines. The diameter of the oil spot is determined by the addition of *r1* + *r2*.

10.9.2 *Step 2*—Contamination Index—IC calculation.

10.9.2.1 The contamination level (IC) is determined by analyzing the oil spot opacity. The software determines the level of gray (from 0 to 255) of each pixel included in the oil spot. The oil spot opacity is the sum of all opacities of all pixels included in the oil spot image divided by the total number of pixels of the oil spot image. Using an established correlation table, the software determines the IC corresponding to the measured opacity. The IC measuring limits are: 0 % to 4.8 %.

10.9.3 *Step 3*—Merit of Dispersancy—MD calculation.

10.9.3.1 To determine MD, the software needs to calculate two intermediary parameters, DB (DB comes from the French term *Démérite Brut*, which means gross demerit) and ratio.

10.9.3.2 Homogeneity of soot distribution within the oil spot is assessed by the DB value. The distribution of the particles in the oil spot is assessed by the ratio value. The DB and the ratio values are not reported. Combination of both DB and ratio gives the value of the merit of dispersancy (MD).

$$MD = (1 - DB)\cdot(Ratio \cdot 100)$$

10.9.3.3 To calculate DB and ratio, the software determines 16 zones in the oil spot with a width of 1 mm each, which corresponds to the ideal reference oil spot of 32 mm diameter.

10.9.3.4 Based on the assumption that the oil spot has a symmetrical form, the processing of the image is performed in two rectangular areas with a length of 16 mm and a height of 11 pixels, which is approximately 1 mm (Fig. 7).

10.9.3.5 Because the oil spot could be oval, the calculation is done separately for *X* and *Y* coordinates (blue rectangles).

10.9.3.6 DB is calculated as follows:

$$DBx = \sum_{i=1}^{n} \frac{|Oxi - Oc|}{(n - 2)\cdot Oc} \quad (1)$$

where:

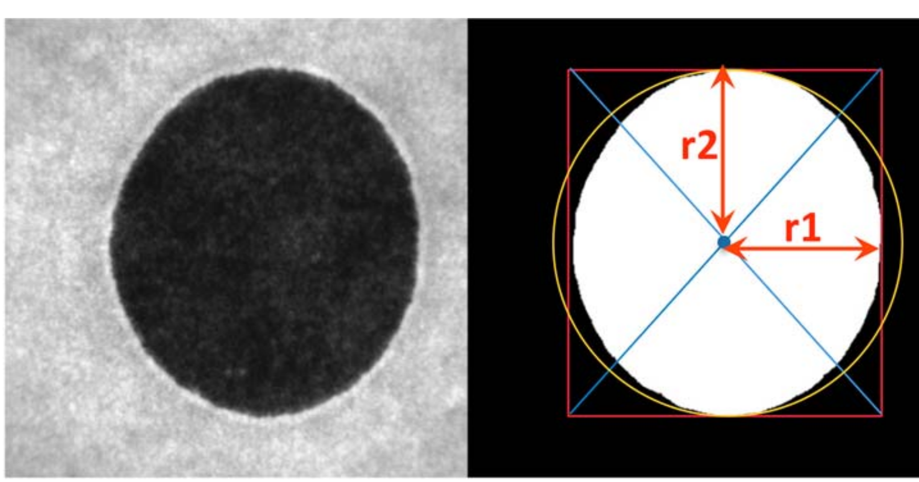

FIG. 6 Principle to Determine *r1* and *r2*

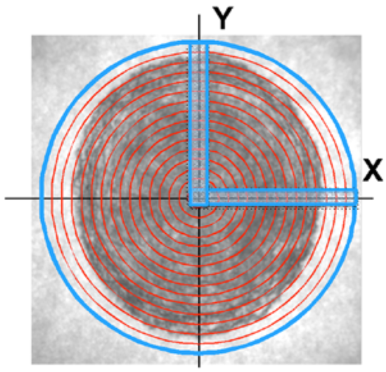

FIG. 7 Sixteen (16) Zones in the Oil Spot

n = number of pixels per mm,
Oxi = opacity of zone i on X direction, and
Oc = overall average oil spot opacity.

$$DBy = \sum_{i=1}^{n} \frac{|Oyi - Oc|}{(n - 2) \cdot Oc} \quad (2)$$

where:

n = number of pixels per mm,
Oyi = opacity of zone i on Y direction, and
Oc = overall average oil spot opacity.

$$DB = \frac{DBx + DBy}{2} \quad (3)$$

10.9.3.7 The ratio is calculated as follows (Fig. 8):

10.9.3.8 The median opacity is calculated for the X and Y axis respectively.

10.9.3.9 Beginning from the oil spot center, the opacity value of each pixel is cumulated until the sum exceeds Om where:

$$OM = \left(\sum_{1=i}^{Rt} Oi \right) / 2 \quad (4)$$

10.9.3.10 The distance between the oil spot center and the point where the sum is equal to Om is called Rmx for X axis and Rmy for Y axis.

10.9.3.11 The ratio is a measure of how well balanced the contamination matter is through the oil spot:

$$\text{Ratio} = 2 \cdot (Rmx + Rmy) / 2\ Rt$$

10.9.3.12 If the ratio is equal to one, the distribution of particles is considered homogeneous throughout the oil spot.

10.9.3.13 The lower the ratio value, the closer the black sooty particles are to oil spot center.

10.9.3.14 MD is then calculated as follows:

$$\text{MD} = (1 - \text{DB}) \cdot (\text{Ratio} \cdot 100)$$

10.9.4 *Step 4*—Weighted Demerit—DP calculation (DP comes from the French term *Démérite Pondéré*, which means weighted demerit).

10.9.5 DP is a combined performance rating (overall note), which is helpful for trend condition monitoring of the performance of the lubricant. Two parameters are used for this calculation, the merit of dispersancy (MD) and the contamination index (IC).

10.9.6 DP is calculated as follows:

$$\text{DP} = (100 - \text{MD}) \cdot \text{IC}$$

11. Report

11.1 Record the results from the apparatus, as the merit of dispersancy of the sample obtained by the procedure, and reference this test method.

11.2 The measurement of the merit of dispersancy is the primary measurement of this test method. However, two other parameters measured by this test method can be recorded, the contamination index (IC) and the weighted demerit (DP). If these parameters are requested, record the results from the apparatus, as the contamination index (IC) and the weighted

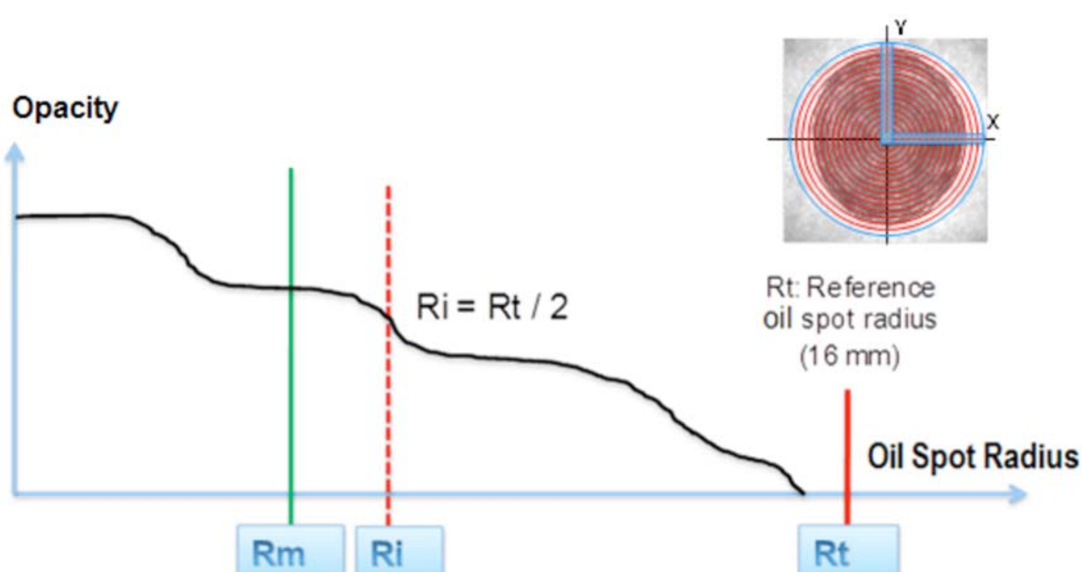

FIG. 8 Calculation of the Ratio

demerit (DP) of the sample obtained by the procedure, and reference this test method.

12. Precision[5]

12.1 The repeatability standard deviation from a single operator, 31 different samples, and one laboratory with four different instruments has been determined to be 1.7.

[5] An interlaboratory study of this test method is being conducted and a complete precision statement is expected to be available on or before the end of 2014.

13. Keywords

13.1 condition monitoring; diesel engine oils; dispersancy; in-service petroleum lubricants; lubricants; oils; oxidation; soot