Standard Test Method for Carbon Black—Oil Absorption Number (OAN)[1]

This standard is issued under the fixed designation D2414; 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.

This standard has been approved for use by agencies of the U.S. Department of Defense.

ε[1] NOTE—Corrected 5.4 editorially in October 2016.

1. Scope

1.1 This test method covers the determination of the oil absorption number of carbon black.

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

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.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and Calculation of Dynamic Viscosity)

D1218 Test Method for Refractive Index and Refractive Dispersion of Hydrocarbon Liquids

D1765 Classification System for Carbon Blacks Used in Rubber Products

D1799 Practice for Carbon Black—Sampling Packaged Shipments

D1900 Practice for Carbon Black—Sampling Bulk Shipments

D4052 Test Method for Density, Relative Density, and API Gravity of Liquids by Digital Density Meter

D4483 Practice for Evaluating Precision for Test Method Standards in the Rubber and Carbon Black Manufacturing Industries

D4821 Guide for Carbon Black—Validation of Test Method Precision and Bias

D5554 Test Method for Determination of the Iodine Value of Fats and Oils

2.2 *DIN Standards:*[3]

DIN 16945 Testing of resins, hardeners and accelerators, and catalyzed resins

DIN EN ISO 660 Animal and vegetable fats and oils - Determination of acid value and acidity

3. Summary of Test Method

3.1 In this test method, oil is added by means of a constant-rate buret to a sample of carbon black in the mixer chamber of an absorptometer. As the sample absorbs the oil, the mixture changes from a free-flowing state to one of a semiplastic agglomeration, with an accompanying increase in viscosity. This increased viscosity is transmitted to the torque-sensing system of the absorptometer. When the viscosity of the mixture reaches a predetermined torque level, the absorptometer and buret will shut off simultaneously. The volume of oil added is read from the direct-reading buret. The volume of oil per unit mass of carbon black is the oil absorption number.

3.2 Either DBP, paraffin or epoxidized sunflower oils are acceptable for use with most standard pelleted grades of carbon black including N-series carbon blacks found in Classification D1765. OAN testing using paraffin oils or epoxidized sunflower oils on some standard blacks and specialty blacks including powder products may result in unacceptable differences as compared to OAN testing with DBP oil. Paraffin and epoxidized sunflower oils are considered non-hazardous; some paraffin oils are FDA approved. For any of the oils, Sections 8 – 11 (Calibration, Procedure, Calculation, and Report) are to be consistent with the oil selected for use. Referee testing between suppliers and users should use DBP oil until such time that precision data are available for alternate oils.

[1] This test method is under the jurisdiction of ASTM Committee D24 on Carbon Black and is the direct responsibility of Subcommittee D24.11 on Carbon Black Structure.

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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 Deutsches Institut fur Normung e.V.(DIN), Burggrafenstrasse 6, 10787 Berlin, Germany, http://www.din.de.

4. Significance and Use

4.1 The oil absorption number of a carbon black is related to the processing and vulcanizate properties of rubber compounds containing the carbon black.

5. Apparatus[4]

5.1 *Balance,* analytical, with an 0.01-g sensitivity.

5.2 *Oven,* gravity-convection type, capable of maintaining 125° ± 5°C.

5.3 *Spatula,* rubber, 100-mm.

5.4 *Absorptometer,* equipped with a constant-rate buret that delivers 4 ± 0.024 cm^3/min.

5.5 *Desiccator.*

6. Reagent and Standards

6.1 *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.[5] 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.

6.2 *n-Dibutyl Phthalate,* having a density of 1.042 to 1.047 Mg/m^3 at 25°C and a relative density of 1.045 to 1.050 at 25°C.

6.3 *Paraffin Oil,* having a kinematic viscosity of 10 to 34 mm^2/s (cSt) at 40°C.

NOTE 1—Three paraffin oils have been found suitable including Marcol 82 from Exxon, 80/90 White Oil from Conoco-Phillips, and LC1 oil from Lab Chemicals, Germany. All three oils are pharmaceutical or food grade oil, or both, based on available data.

6.4 *Epoxidized Fatty Acid Ester (EFA),* meeting the specifications listed in Annex A4. It is recommended to store the product at temperatures between 7 and 30°C. If stored in sealed original containers, the product is stable for at least 12 months. For handling and safety, please refer to safety data sheet.

6.5 ASTM D24 Standard Reference Blacks, SRB.[6]

7. Sampling

7.1 Samples shall be taken in accordance with Practices D1799 and D1900.

8. Calibration and Standardization

8.1 *Absorptometer:*

8.1.1 *Model*—Three different types of absorptometers are in use: (*1*) early models based on springs and mechanical indication of torque (Type A and B), (*2*) second generation absorptometers equipped with load cells and digital torque display (Type E[7]), and (*3*) current model absorptometers which are designed with a torque measuring system that includes a micro-computer and software to continuously record torque and oil volume with time (Types H and C and modified Type E[7]). Types A, B, and E[7] are designed to stop mixing at a predetermined, fixed torque level, which is the recommended procedure for measuring hard or tread blacks (calibration Procedure A). The computer controlled models (Types H and C and modified Type E[7]) are required for running calibration Procedure B, the recommended torque curve analysis for the determination of the end-point of soft or carcass blacks. The Type H and C and modified Type E[7] absorptometers can also provide an end-point at a fixed or predetermined torque level such that these types of absorptometers are well-suited for measuring OAN of both hard and soft carbon blacks. Several components influence the calibration: the dynamometer torque spring or the load cell, the torque limit switch or the indicator set point, the damper (oil damper or electronic damping), and the mixing head consisting of two counter rotating blades and a mixing bowl. It is necessary that all of these components are in good condition and are properly adjusted to achieve acceptable calibration.

8.1.2 *Mixing Bowl*—Typically the absorptometer is delivered with either a surface-treated stainless steel or anodized aluminum mixing bowl. These bowls are considered acceptable provided they give the correct reading for the appropriate SRB reference standards. The surface finish of the mixer chamber is critical for maintaining proper calibration, and the bowl should not be modified to achieve calibration.

NOTE 2—Stainless steel chambers have been found satisfactory for the test when they are manufactured to a roughness value (Ra) of 2.5 ± 0.4 µm (100 ± 15 µin.) based upon 8 measurements. No single measurement should be greater than 3.6 µm (140 µin.) or less than 1.5 µm (60 µin.). Stainless steel bowls purchased with an absorptometer have been pre-polished for 16 h to minimize bowl surface changes affecting calibration during their initial use. It is recommended that new replacement stainless steel bowls should also be pre-polished in the same manner (see Annex A3).

8.2 *Calibration:*

8.2.1 *Rotor Blades*—The speed of the motor driving the rotor blades is either fixed (Type A and B) or has to be set (Type E, C, and H) to 125 r/min. Due to a gear, one blade spins at 125 r/min, the other blade at 250 r/min.

8.2.2 *Constant-Rate Buret*—The delivery rate of the buret is to be 4 cm^3/min. See Annex A1 for detailed instructions on the procedure for calibration check of the constant-rate buret.

8.2.3 *Spring Tension (Type A and B)*—It is recommended that the torque spring is adjusted so that the SRB F standard will develop a maximum torque between 70 % and full-scale deflection. This is achieved by selecting the appropriate spring strength and adjusting the spring tension in accordance with the instructions of the manufacturer.

[4] All apparatus are to be operated and maintained in accordance with the manufacturers' directions for optimum performance.

[5] *Reagent Chemicals, American Chemical Society Specifications* , American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K. and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

[6] The sole source of supply of ASTM Standard Reference Blacks known to the committee at this time is Laboratory Standards and Technologies, 227 Somerset, Borger, TX 79007, http://carbonstandard.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.

[7] Type E absorptometers can be modified with additional hardware and micro-computer system.

NOTE 3—The absorptometers Type E, C, and H are calibrated by the manufacturer to give a direct reading of torque in mNm; this calibration should not be modified in order to achieve a desired level of torque. If calibration is necessary, refer to the instrument manufacturer's recommendations. The instrument torque calibration should not be confused with the torque limit switch described in 8.2.5.

8.2.4 *Damper*—For the Type A absorptometer, it is recommended to keep the valve of the oil damper fully closed. The Type B absorptometer shall provide a full-scale recovery of 3 ± 0.5 s; the valve has to be adjusted accordingly. The Type E absorptometer has an electronic damping option and Types C and H have appropriate software damping. Make sure that these damping options are activated.

8.2.5 *Torque Limit Switch (TLS) or the Indicator Set Point*—If the end-point of the test is determined by a fixed torque limit, the setting of the TLS, also called indicator set-point, has to be selected using one of the following three procedures:

8.2.5.1 *Procedure A: End-Point at Fixed Torque Level*—This "classical" method is well suited for most hard or tread blacks but may lead to problems when low-torque carcass blacks are to be tested; proceed to Procedure B for low-torque carbon blacks. For Type A, B, and E absorptometers, adjust the TLS or the indicator set point in such way that the current SRB F standard gives the correct target value within the limits as defined in Guide D4821. For Type E, C, and H absorptometers dedicated to testing tread blacks only, there is no advantage to setting the TLS based on the SRB F standard; for these absorptometers, set the TLS to 3500 mNm for DBP oil, or 4000 mNm for paraffin oil.

8.2.5.2 *Procedure B: End-Point at 70 % of the Maximum Torque*—Certain carcass blacks and thermal blacks may fail to give an end-point due to insufficient torque level. Therefore, the preferred method for testing soft blacks is to record the torque curve using a chart-recorder or a data acquisition system and to read the end-point at 70 % of the maximum of the torque achieved. Set the TLS or the indicator set point to full scale in order to disable the automatic shut-off of the absorptometer.

8.2.5.3 *Procedure C: End-Point at a Fixed, But Reduced Torque Level*—Requires use of SRB-5 series standards. See Test Method D2414 – 00.

8.3 *Normalization:*

8.3.1 Physically calibrate the test apparatus including TLS adjustment using the instructions in 8.2.

8.3.2 Test the six ASTM Standard Reference Blacks (SRBs) in duplicate to establish the average measured value. Additional values are added periodically, typically on a weekly basis. The rolling average of the measured values is computed from the latest four values.

NOTE 4—When only tread- or carcass-type carbon blacks are to be tested, the calibration can be limited to either the three tread- (A, B, C) or the three carcass-type (D, E, F) carbon black standards.

8.3.3 Perform a regression analysis using the standard value of the standard (*y* value) and the rolling average measured value (*x* value). Separate carcass and tread calibration curves should be maintained.

8.3.4 Normalize the values of all subsequent samples as follows:

$$\text{Normalized value} = (\text{measured value} \times \text{slope}) + y - \text{intercept} \quad (1)$$

8.3.5 For normalized values of the SRBs that are consistently outside the x-chart limits listed in Guide D4821, the test apparatus should be recalibrated in accordance with 8.2.

8.3.6 When any absorptometer or calibration changes occur, a new calibration curve must be initiated as described in 8.3.2.

8.3.7 In most instances, if proper calibration cannot be achieved by following 8.2 or 8.3.2 – 8.3.4, it will be necessary to replace the mixer chamber with one of proper surface finish. Review Appendix X1.

9. Procedure

9.1 Dry an adequate sample for 1 h in the specified oven set at 125°C. Prior to testing, cool the sample in a desiccator for a minimum of 30 min.

9.2 Weigh the sample to the nearest 0.01 g. The recommended masses are as follows:

Carbon Black	Mass, g
N630, N642, and N700 series, except N765	25
N800 and N900 series, SRB D-7 and D-8	40
All others	20

9.3 It is recommended that a testing temperature of 23 ± 5°C be maintained, as measured by a thermocouple in the mixing bowl. If a temperature controllable mixing bowl is not available, keep the bowl temperature below 30°C and comply with Note 5 and Note 6 while running the samples.

NOTE 5—If the absorptometer has remained idle for more than 15 min and a temperature controllable bowl is not being used, a 10-min warm-up sample must be run before beginning a test. It is important that the mixer chamber temperature be kept uniform. Preferably, allow 5 min between the end of one test and the start of another.

NOTE 6—It is important that the temperature of the bowl be the same for machine calibration as for oil absorption testing. ASTM task group work has shown that an increase in bowl temperature can cause higher values that increased variability in bowl temperatures cause increased test variability.

NOTE 7—In the event that an endpoint is not obtained (maximum torque < TLS) when using an absorptometer with a fixed TLS such as Type B or E, it is acceptable to mill pelleted carbon blacks using a coarse grinder such as a coffee mill. The carbon black should be milled for only a few seconds to allow sufficient grind time to change the pellets to powder form. High-speed micronizing mills and air-jet mills are not acceptable, as they can reduce the carbon black structure.

9.4 Transfer the sample to the absorptometer mixer chamber and replace the chamber cover. For Type H, close the safety door surrounding the mixing chamber.

9.5 Position the buret delivery tube over the hole in the mixer chamber cover, and for Types A, B, or E set the buret digital counter to zero (Types C and H have automatic reset). Insure the buret delivery tubes have no air bubbles.

9.6 Activate the "start" button. On the Type E absorptometer, activate both "start" buttons simultaneously. The apparatus will operate until one of the following conditions are met: 1) sufficient torque has developed to activate the torque-limit switch, which will halt the absorptometer and buret; 2) the sample torque has reached a maximum and then dropped below maximum torque for a preset period of time (using Procedure B).

9.7 Record the volume of oil used as indicated by the buret digital counter.

9.8 Dismantle the mixer chamber and clean the mixing blades and chamber with a rubber spatula and reassemble.

9.9 Mixing chamber cleanup can be aided by the addition of dry carbon black to the mixing chamber prior to disassembly, and the use of the preset cleanup cycle for Types E, C, and H (use of water to aid cleanup is not recommended).

NOTE 8—It is not necessary to clean and polish the mixing blades and chamber with a solvent, but it is recommended to remove all visible residues by wiping the chamber and mixing blade surfaces.

10. Calculation

10.1 Calculate the oil absorption number of the sample to the nearest 0.1 $10^{-5}m^3/kg$ ($cm^3/100$ g) as follows:

$$\text{Oil absorption number, } 10^{-5}\text{m}^3/\text{kg} = \frac{A}{B} \times 100 \qquad (2)$$

where:

A = volume of oil used, cm^3, and
B = mass of tested sample, g.

11. Report

11.1 Report the following information:

11.1.1 Proper identification of the sample,

11.1.2 Oil (DBP, paraffin, or epoxidized sunflower oil),

11.1.3 Method for end-point determination (Procedure A, B or C in 8.2),

11.1.4 Sample mass, if different than shown in 9.2, and

11.1.5 The result obtained from the individual determination is reported to the nearest 0.1 $10^{-5}m^3$ /kg ($cm^3/100$ g).

12. Precision and Bias

12.1 These precision statements have been prepared in accordance with Practice D4483. Refer to this practice for terminology and other statistical details.

12.2 Interlaboratory precision program (ITP) information was conducted as detailed in Table 1. Both repeatability and reproducibility represent short-term (daily) testing conditions. The testing was performed using two operators in each laboratory performing the test once on each of two days (total of four tests). A test result is the value obtained from a single determination. Acceptable difference values were not measured. The between operator component of variation is included in the calculated values for r and R.

12.3 The precision results in this precision and bias section give an estimate of the precision of this test method with the materials used in the particular interlaboratory programs described in 12.2. The precision parameters should not be used for acceptance or rejection testing of any group of materials without documentation that they are applicable to those particular materials and the specific testing protocols of the test method. Any appropriate value may be used from Table 2.

12.4 The results of the precision calculations for this test are given in Table 2. The materials are arranged in ascending "mean level" order.

12.5 *Repeatability*—The **pooled relative** repeatability, (r), of this test has been established as 1.2 %. Any other value in Table 2 may be used as an estimate of repeatability, as appropriate. The difference between two single test results (or determinations) found on identical test material under the repeatability conditions prescribed for this test will exceed the repeatability on an average of not more than once in 20 cases in the normal and correct operation of the method. Two single test results that differ by more than the appropriate value from Table 2 must be suspected of being from different populations and some appropriate action taken.

NOTE 9—Appropriate action may be an investigation of the test method procedure or apparatus for faulty operation or the declaration of a significant difference in the two materials, samples, and so forth, which generated the two test results.

12.6 *Reproducibility*—The **pooled relative** reproducibility, (R), of this test method has been established as 3.1 %. Any other value in Table 2 may be used as an estimate of reproducibility, as appropriate. The difference between two single and independent test results found by two operators working under the prescribed reproducibility conditions in different laboratories on identical test material will exceed the reproducibility on an average of not more than once in 20 cases in the normal and correct operation of the method. Two single test results produced in different laboratories that differ by more than the appropriate value from Table 2 must be suspected of being from different populations and some appropriate investigative or technical/commercial action taken.

TABLE 1 SRB8 ITP Information

SRB8 Material	Grade	Producer	Test Period	Number of Labs (M/H/L) D2414
SRB-8A	N326	Continental	March 2008	64 (1/0/0)
SRB-8A2	N326	Continental	March 2013	72 (0/1/1)
SRB-8B	N134	Cabot	June 2009	66 (0/0/0)
SRB-8B2	N134	Cabot	March/April 2014	40 (3/4/3)
SRB-8C	HS Tread	Columbian	September 2010	66 (2/1/0)
SRB-8D	LS Carcass	Cabot	March 2009	67 (0/2/0)
SRB-8E	N660	Orion	September 2008	57 (1/0/0)
SRB-8F	N683	Orion	March 2010	67 (1/1/0)
SRB-8F2	N683	Orion	March 2015	62 (1/0/0)
SRB-8G[A]	N990	Cancarb	Last half of 1996	Unknown

[A] SRB-8G was produced and approved in the last half of 1996 as SRB-5G and has continued to be included in the current SRB sets since that time. At the time it was produced and approved it was D24's practice to only publish the within-laboratory standard deviation, Sr, and associated limits. The between-laboratory standard deviation, SR, was never published and since the data is no longer available it is not possible to calculate or publish the SR values and corresponding limits. The SRB G material was only tested for NSA, STSA, and OAN per the test method version available in 1996.

TABLE 2 Precision Parameters for Test Method D2414, OAN Method (Type 1 Precision)[A]

Units	10^{-5} m³/kg (cm³/100 g)						
Material	Mean Level	Sr	r	**(r)**	SR	R	**(R)**
SRB-8C	174.9	0.50	1.41	**0.8**	1.08	3.04	**1.7**
SRB-8B2	125.2	0.42	1.19	**1.0**	0.97	2.74	**2.2**
SRB-8B	123.5	0.45	1.26	**1.0**	0.91	2.57	**2.1**
SRB-8A2	71.5	0.46	1.31	**1.8**	1.56	4.42	**6.2**
SRB-8A	70.9	0.46	1.31	**1.8**	0.93	2.64	**3.7**
SRB-8F	132.0	0.41	1.16	**0.9**	0.91	2.59	**2.0**
SRB-8E	87.8	0.36	1.02	**1.2**	1.30	3.68	**4.2**
SRB-8D	36.9	0.26	0.73	**1.9**	1.09	3.09	**8.1**
Average	103.0						
Pooled Values		0.42	1.19	**1.2**	1.11	3.15	**3.1**

[A] The preferred precision values are shown in bold text.

12.7 *Bias*—In test method terminology, bias is the difference between an average test value and the reference (true) test property value. Reference values do not exist for this test method since the value or level of the test property is exclusively defined by the test method. Bias, therefore, cannot be determined.

13. Keywords

13.1 carbon black; n–dibutyl phthalate; oil absorption number; paraffin oil

ANNEXES

(Mandatory Information)

A1. CALIBRATION CHECK OF CONSTANT-RATE BURET

A1.1 Scope

A1.1.1 The constant-rate buret is an integral part of the absorption-measuring system. Failure of the buret to deliver the specified amount of reagent to the carbon black will result in erroneous absorption readings. This annex provides a method for checking the delivery rate of the constant-rate buret. One of the reasons for the incorrect absorption values (caused by incorrect reagent delivery by the automatic buret) is entrapped air in the plastic tubing or the delivery tube, especially above the nozzle. This trouble source should be checked first.

A1.2 Apparatus

A1.2.1 *Stop Watch.*

A1.2.2 *Beaker,* 150-cm³.

A1.3 Procedure

A1.3.1 Ensure that all seals and tubing are in good condition.

A1.3.2 Fill the buret and delivery tubes with oil. Ensure that all air is removed from the system.

A1.3.3 With the buret completely full, set the stopcock to the delivery position. Run the buret on "deliver" until a constant flow is obtained from the delivery tube.

A1.3.4 Stop the buret and set the digital counter to zero.

A1.3.5 Position a tared 150-cm³ beaker under the delivery tube.

A1.3.6 Simultaneously start the buret and stop watch.

A1.3.7 After 2 min, stop the buret and record the digital counter reading.

A1.3.8 Weigh and record the amount of reagent delivered.

A1.3.9 Refill the buret.

A1.3.10 Repeat A1.3.3 – A1.3.9, changing the delivery time in A1.3.7 to 4 and 8 min.

A1.4 Calculation

A1.4.1 Calculate the volume of oil from the delivered mass and density (A1.3.8) as follows:

$$\text{Delivery, cm}^3 = \frac{\text{mass delivered}}{\text{oil density}} \quad (A1.1)$$

A1.5 Acceptable Results

A1.5.1 The calculated delivery should be within the following limits of the digital counter reading:

Time, min	Volume, cm³	Tolerance, cm³
2	8	±0.05
4	16	±0.10
8	32	±0.20

A1.6 Oil Density

A1.6.1 Oil density is necessary to calculate the volume of oil delivered from a buret as described in A1.4. Oil densities may be analyzed using calibrated hydrometers or density meters at specified temperatures. Typical densities for both DBP and paraffin oil have been obtained from different laboratories measuring different lots of oils as shown in Table A1.1. Epoxidized sunflower oil densities were obtained from the vendors technical data and other laboratory measurements.

NOTE A1.1—New oil densities will be added to Table A1.1 as alternative oils are identified and demonstrated as suitable for OAN testing, and as density data are made available to D24.

A1.6.2 Variation in oil density has been observed between different labs and lots of oil. typical DBP density at 23°C was observed to vary from 1.044 to 1.050 g/cm³, and typical Marcol 82 density at 23°C was observed to vary from 0.843 to

0.846 g/cm³. Until further guidelines are made available by D24, it is an acceptable practice to utilize an average density value from Table A1.1, or either lot data at a specified temperature.

A1.6.3 Selection of the appropriate oil density from Table A1.1 is dependent on the temperature of the oil in use. It is suggested that a temperature measurement be made on the oil in the buret reservoir, and the appropriate density used in A1.4.

A1.6.4 Conoco-Phillips 80/90 White Oil density is reported at 15.6°C (60°F) as 0.855 g/cm³; LC1 oil density is reported at 15°C (59°F) as 850 kg/m³ (0.85 g/cm³).

A1.6.5 Epoxidized sunflower oil density is reported at 20°C as 0.900 to 0.910 g/cm³. Additional density data at three temperatures was reported as follows: 20.0°C as 0.9033 g/cm³, 25.0°C as 0.8998 g/cm³, and 30.0°C as 0.8933 g/cm³.

TABLE A1.1 Oil Density

	Typical DBP Oil Density			Typical Marcol 82 Paraffin Oil Density		
Temp °C	Lab 1 g/cm³	Lab 2 g/cm³	Average g/cm³	Lab 1 g/cm³	Lab 2 g/cm³	Average g/cm³
20.0	1.052	1.047	1.049	0.845	0.848	0.846
21.0	1.051	1.046	1.049	0.844	0.847	0.846
22.0	1.050	1.045	1.048	0.844	0.846	0.845
23.0	1.050	1.044	1.047	0.843	0.846	0.844
24.0	1.049	1.043	1.046	0.842	0.845	0.844
25.0	1.048	1.043	1.045	0.841	0.844	0.843
26.0	1.047	1.042	1.044	0.841	0.844	0.842
27.0	1.046	1.041	1.044	0.840	0.843	0.842
28.0	1.046	1.040	1.043	0.839	0.842	0.841
29.0	1.045	1.039	1.042	0.839	0.842	0.840
30.0	1.044	1.038	1.041	0.838	0.841	0.840

A2. DETERMINATION OF MAXIMUM TORQUE

A2.1 Scope

A2.1.1 On some instruments the SRB F-6 (an N683 carbon black) will not develop sufficient torque to produce acceptable test precision. This is an indication that other similar type carbon blacks may also test with poor precision.

A2.1.2 In order to obtain acceptable test precision in these situations, it is necessary that the absorptometer be adjusted mechanically or electronically for Type E absorptometers so that the F-6 SRB will develop a maximum torque of at least 70 % of full scale. This procedure gives the needed instructions to determine the maximum torque developed by a carbon black sample.

A2.2 Procedure

A2.2.1 Set the torque pointer to 10 on the Set Scale. For Type E absorptometers, move the shut-off alarm set point to maximum scale. This makes 100 % of the torque range available.

NOTE A2.1—Torque limit switch settings should always be made with the instrument stopped and the mixing chamber empty.

A2.2.2 Start the apparatus having followed 9.1 – 9.6 for testing SRB F-6.

A2.2.3 As the sample begins to develop viscosity and the torque increases, the pointer will move down the scale towards zero. The maximum % torque, T_{max}, developed by the sample is as follows:

$$T_{max} = (10 - N_{min}) \times 10 \quad (A2.1)$$

where:

N_{min} = the lowest pointer reading.

A3. PRE-POLISHING PROCEDURE FOR NEW REPLACEMENT STAINLESS STEEL BOWLS

A3.1 Scope

A3.1.1 It is recommended that new replacement stainless steel bowls manufactured with a 2.5 ± 0.4 µm (100 ± 15 µin.) roughness be pre-polished for 16 h prior to their use for oil absorption testing. This will minimize the calibration changes for the absorptometer that will probably occur without the pre-polishing.

A3.2 Reagents

A3.2.1 *Carbon Black* (SRB F-8).

A3.2.2 *n-Dibutyl Phthalate or Paraffin Oil.*

A3.3 Procedure

A3.3.1 Weigh 25 g of SRB F-8 carbon black and transfer this sample into the absorptometer mixing chamber.

A3.3.2 Turn on the absorptometer and add 35 cc of oil.

NOTE A3.1—Relieve the torque limit switch to prevent automatic shutoff. It may be necessary to increase the spring tension.

A3.3.3 Allow the absorptometer to run continuously for 16 h.

NOTE A3.2—The absorptometer bowl must be securely covered during this time to prevent the loss of sample to be able to achieve adequate pre-polishing action.

A3.3.4 After 16 h, stop the absorptometer, empty the sample, and clean the mixing chamber and blades. Allow the chamber to cool to room temperature.

A3.3.5 Check and adjust the torque-switch setting and the spring tension before proceeding with calibration following the standard testing procedure.

A4. EPOXIDIZED FATTY ACID ESTER (EFA) OIL SPECIFICATION

A4.1 See Table A4.1.

TABLE A4.1 Specifications of EFA Oil For Use in OAN/COAN Test

Test	Method	Units	Min.	Max.
Refractive index (20°C)	ASTM D1218	nD20	1.4530	1.4590
Acid number	DIN EN ISO 660	mg KOH/g	0.0	3.0
Iodine number	ASTM D5554-95	gI2/100g	0.0	5.0
Epoxy oxygen content	DIN 16945	wt. %	3.5	4.5
Density (20°C)	ASTM D4052	g/cm^3	0.900	0.920
Viscosity, kinematic (40°C)	ASTM D445	mm^2/s	10.0	30.0

APPENDIX

(Nonmandatory Information)

X1. DIAGNOSTIC PROCEDURES FOR MONITORING OAN MIXING CHAMBERS

X1.1 Scope

X1.1.1 Diagnostic procedures for monitoring the condition of a mixing bowl surface when using DBP or paraffin oils are described in X1.2 and X1.3, and an assessment of the mixing chamber mechanical operation in X1.4. For oil absorptometers used to measure the OAN of carcass or soft grades (or other nonreinforcing carbon blacks), the mixing bowl surface must be monitored to insure it is capable of producing acceptable OAN data. This criterion does not apply to tread or hard blacks (that is, reinforcing carbon blacks).

X1.1.2 Diagnostic procedures for determining the condition of an OAN mixing bowl have been developed based on the characteristics of SRB F-8 (N683) with DBP and paraffin oils. For an oil absorptometer system in good condition, the typical maximum torque level for F-8 is approximately 4500 to 5000 mNm with DBP and paraffin oils. A typical torque value at 70 % of maximum torque is 3500 to 4000 mNm using DBP and paraffin oils.

NOTE X1.1—A replacement mixing bowl can be used to replace a worn bowl. The mixing drive should also be evaluated for mechanical condition as described in X1.4.

X1.2 Monitoring Mixing Chamber Surface Conditions Using Measured TLS with DBP and Paraffin Oils

X1.2.1 For absorptometers which are setup for "Measured TLS," monitor the TLS value of SRB F-8 using an x-chart. The definition of TLS or torque limit switch is the torque at which the SRB F-8 equals the specified target value in cm^3/100g as found in Guide D4821. The TLS value can be measured by the instrument software.

X1.2.2 TLS values for "Measured TLS" are typically 1500 to 5000 mNm. New mixing bowls which have been pre-polished should have TLS values of approximately 3500 to 5000 mNm. As the mixing bowl wears, the TLS value is reduced. When the TLS value reaches 1800 mNm or less, the mixing bowl should be evaluated for replacement using ASTM SRB's as described in X1.2.3.

X1.2.3 If the following criterion are exceeded, the mixing bowl should be replaced:

X1.2.3.1 Normalized OAN values are outside ASTM SRB tolerances for OAN testing.

X1.2.3.2 Average differences in measured and normalized OAN values are greater than ±3 to 4 cm^3/100 g.

X1.3 Monitoring Mixing Chamber Surface Conditions Using Fixed TLS with DBP and Paraffin Oils

X1.3.1 For absorptometers which are setup for fixed or pre-set TLS level as described in Test Method D2414-11, Subsection 8.2.5.1, regularly monitor the raw or measured value of the SRB F-8 standard using an x-chart.

X1.3.2 SRB F-8 raw or measured OAN values for a new mixing bowl which has been prepolished should be approximately 3 to 4 cm^3/100 g less than target. New chambers always give raw OAN measurements low versus target. If the raw value is outside the 3 to 4 cm^3/100 g less than target, the chamber should undergo further pre-polishing.

X1.3.3 As the mixing bowl is used the raw F-8 OAN will slowly increase. When the F-8 measured OAN value is 3 to 4 cm^3/100 g greater than target the mixing chamber should be evaluated for replacement.

X1.4 Determining the Mechanical Condition of an OAN Mixing Chamber

X1.4.1 An OAN mixing chamber consists of two major components including a mixing bowl and a mixing drive. A mixing drive includes a back-plate, two rotors, bushings, bearings, gear drive, etc. The back-plate and rotors are typically constructed of either stainless steel or aluminum.

X1.4.2 Aluminium mixing bowls typically have an anodized surface treatment or hardcoat finish. Once the finish is visibly worn off of an aluminum mixing bowl it should be replaced. Components which are physically damaged should be replaced. The mixing bowl should be removed and the drive components inspected. Bushings can be inspected by applying side-toside force to the rotors to check for excessive movement.

X1.4.3 A diagnostic measurement used to assess and monitor the general condition of the mixing drive bearings and rotors is to monitor the idle torque. This is accomplished by first ensuring the mixing chamber has been thoroughly cleaned, then reassemble as if starting a new test. Start the motor and after several seconds observe the average torque reading. This is the idle torque.

X1.4.4 The idle torque of typical mixing chambers will vary from a low level of 20 to 30 mNm up to higher levels of 100 to 150 mNm. Most new mixing chambers will exhibit an idle torque less than 100 mNm. The idle torque of an absorptometer mixing chamber should be monitored regularly such that when a problem occurs that causes the idle torque to rise above a typical level, it is recognized and can be investigated.

X1.4.5 Generally when idle torque levels exceed 150 to 200 mNm or a significant rise in idle torque is observed, this is an indication of one of several possible problems:

X1.4.5.1 The mixing assembly is not properly cleaned.

X1.4.5.2 The torque sensor may need calibrated. Refer to the manufacturer's procedures.

X1.4.5.3 There may be carbon black-oil paste between the rotors and bushings or inside the bearing assembly. Correcting this condition requires disassembly and cleaning the bearing assembly and possible replacement of the rotor bushings which may have excess wear. It is not recommended the user perform this maintenance, but to send the unit to the manufacturer or other shop familiar with the equipment and specifications. In some cases the bearings may be damaged and require replacement.

X1.4.5.4 Some mixing chambers have bearings which require proper alignment. Refer to the manufacturers alignment procedures.