ASTM INTERNATIONAL

Designation: D2414 – 23

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

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

1.4 *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]

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 Fatty Acid Ester (EFA) 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 or EFA 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 EFA oils are considered non-hazardous; some paraffin oils are FDA approved. For any of the oils, Sections 8 – 12 (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.

---

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.

---

[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.

8. Oil Absorptometers, Instrument Calibration, and End-Point Procedures

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]) absorptometers can provide an end-point at a fixed or predetermined torque level such that these types of absorptometers are well-suited for measuring OAN of all types of carbon blacks.

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 on 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 *Instrument Calibration*—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.

8.2.1 *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.

---

[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 8.3.1.

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 *Stainless Mixing Bowls*—When replacing an old mixing bowl with a new one, further pre-polish may be needed after installation even if those mixing bowls are typically already pre-polished by the manufacturer. The surface finish of the mixer chamber is critical for maintaining proper calibration, and the bowl shall not be modified to achieve calibration other than described in Annex A3.

8.3 *End-Point Procedures:*

8.3.1 *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:

8.3.1.1 *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.

8.3.1.2 *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.

9. Normalization using Standard Reference Blacks (SRB)

9.1 Physically calibrate the test apparatus including TLS adjustment using the instructions in 8.3.

9.2 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. An alternative to periodic normalizations are daily additions of SRB normalization tests such that the normalization curve is continuously maintained. The rolling average of the measured values is computed from the latest four values.

9.3 For a tread or carcass-type normalization curve, all three SRBs should be included.

9.4 For a tread or carcass-type normalization curve, because of the differing grades in SRB series and material ages, all three SRBs should normally be from the same series (example: series-9). However, when the normalization range provided by a given SRB set does not cover the normalization range needed, follow Guide D4821 Subsection 8.2 to choose the proper alternative SRB material to be used in the normalization curve.

Note 4—When only tread- or carcass-type carbon blacks are to be tested, the normalization can be limited to either the three tread SRBs (example: 9A, 9B, 9C for typical OAN levels or 9A, 9C, 9H for high OAN levels > 125 10^{-5} m^3/kg (cm^3/100 g)) or the three carcass-type SRBs (example: 9D, 9E, 9F).

9.5 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 normalization curves should be maintained.

9.6 Normalize the values of all subsequent samples using the regression equation as follows:

$$\text{Normalized value} = (\text{measured value} * \text{slope}) + \text{intercept} \qquad (1)$$

9.7 For normalized values of the SRBs that are consistently outside the x-chart limits listed in Guide D4821, the instrument calibration and end-point procedures in Section 8 should be reviewed.

9.8 When any absorptometer or calibration changes occur, a new normalization curve must be initiated as described in Section 9.

9.9 In most instances, if normalized values of the SRB's are not within x-chart limits provided in Guide D4821, and the normalizations are current, it will be necessary to replace the mixer chamber with one of proper surface finish. Review Appendix X1.

10. Procedure

10.1 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.

Note 5—Very fluffy or low density material, for example, non-beaded, may require a pre-densification step prior to testing. Fill sufficient material for OAN testing into a paper bag, close the bag properly and start carefully compressing the sample by hand. Let the air pass through the paper bag and then compress more rigorously to densify the sample so that the desired sample mass fits into the mixing chamber. Failure to densify samples can lead to high uncertainty in OAN results. If this densification step did not reduce sample volume sufficiently for the weights listed in 10.2, refer to Note 6.

10.2 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, D-8 and D-9	40
All others	20*

*See Note 6 for exceptions.

Note 6—Non-ASTM grades may differ significantly in their Oil Adsorption Number from the N-grades; therefore, the 20-g sample mass listed above for "All Others" might result in under- or overfilling of the OAN mixing chamber. In these cases, alternative sample masses can be used for grades that are NOT included in Classification D1765.

10.3 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 7 and Note 8 while running the samples.

NOTE 7—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 8—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.

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

10.5 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.

10.6 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).

NOTE 9—In the event that an endpoint is not obtained (maximum torque <TLS) or an unexpected value is obtained, 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.

10.7 For Type B and E oil absorptometers, record the volume of oil used as indicated by the buret digital counter. For Type C and H oil absorptometers, the normalized OAN test results are obtained directly from the instrument software. A summary of recommended end-point type is shown below for the two general types of carbon black:

Recommended End-Point Procedure and Results

CB Type	End-Point Procedure	Result*
Hard/Tread	A	Normalized
Soft/Carcass	B	Normalized

*OAN test results should always be normalized to standard reference blacks (SRB) when reported.

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

10.9 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 10—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.

11. Calculation

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

$$\text{Oil absorption number, } 10^{-5} \text{m}^3/\text{kg} = \left\{ \frac{A}{B} \times 100 \times C \right\} + D \quad (2)$$

where:

A = volume of oil used, cm^3,
B = mass of tested sample, g,
C = slope of normalization curve (see Section 9), and
D = y-intercept of normalization curve (see Section 9).

12. Report

12.1 Report the following information:

12.1.1 Proper identification of the sample,

12.1.2 Oil (DBP, paraffin, or EFA),

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

12.1.4 Sample mass, if different than shown in 10.2, and

TABLE 1 Precision Parameters for Test Method D2414, Method N/A (Type 1 Precision)[A]

Material	Period	Units Number of Laboratories (M/H/L)[B]	10^{-5} m^3/kg (cm^3/100 g) Mean Level	Sr	r	(r)	SR	R	(R)
SRB-9A	Mar 2013	77(0/1/1)	71.5	0.47	**1.33**	1.9	1.55	**4.38**	6.1
SRB-9B	Mar 2016	87(0/2/0)	98.9	0.40	**1.14**	1.1	1.11	**3.14**	3.2
SRB-9D	Mar 2018	75(2/2/1)	34.5	0.25	**0.72**	2.1	0.72	**2.03**	5.9
SRB-9E	Aug 2016	81(1/3/0)	89.2	0.44	**1.24**	1.4	0.89	**2.53**	2.8
SRB-9F	Mar 2015	74(1/0/0)	134.5	0.53	**1.51**	1.1	1.59	**4.51**	3.3
SRB-9G	Aug 2017	74(0/2/1)	38.6	0.23	**0.64**	1.7	1.76	**4.98**	12.9
SRB-9C	Aug 2019	79(1/2/0)	124.4	0.44	**1.24**	1.0	1.23	**3.49**	2.8
SRB-9A2	Aug 2018	76(0/2/0)	71.4	0.34	**0.97**	1.4	1.18	**3.34**	4.7
SRB-9H	Mar/Apr 2020	74(1/1/0)	178.3	0.57	**1.63**	0.9	1.92	**5.43**	3.0
Average			94.0						
Pooled Values				0.42	**1.19**	1.3	1.38	**3.90**	4.1

[A] Preferred precision shown in bold text.
[B] M = number of outliers for Mean; H = number of outliers for High variation; L = number of outliers for Low variation.