



Designation: D2414 – 21

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.

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 of 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

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.

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.

³ Available from Deutsches Institut für 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. Apparatus⁴

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

5.2 *Oven*, gravity-convection type, capable of maintaining $125^{\circ} \pm 5^{\circ}\text{C}$.

5.3 *Spatula*, rubber, 100-mm.

5.4 *Absorptometer*, equipped with a constant-rate buret that delivers $4 \pm 0.024 \text{ cm}^3/\text{min}$.

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 E⁷), 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 E⁷). Types A, B, and E⁷ 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 E⁷) 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 $2.5 \pm 0.4 \mu\text{m}$ ($100 \pm 15 \mu\text{in.}$) based upon 8 measurements. No single measurement should be greater than $3.6 \mu\text{m}$ ($140 \mu\text{in.}$) or less than $1.5 \mu\text{m}$ ($60 \mu\text{in.}$). 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 $4 \text{ cm}^3/\text{min}$. 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.