Designation: D 2414 – 02

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

This standard is issued under the fixed designation D 2414; 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 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:*

D 1799 Practice for Carbon Black—Sampling Packaged Shipments[2]

D 1900 Practice for Carbon Black—Sampling Bulk Shipments[2]

D 4483 Practice for Determining Precision for Test Method Standards in the Rubber and Carbon Black Industries[2]

D 4821 Guide for Carbon Black—Validation of Test Method Precision and Bias[2]

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[3]

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 or paraffin oil is acceptable for use. While studies have shown either oil to exhibit comparable precision, paraffin oil offers the advantage of being non-hazardous; even FDA approved grades are available. For either oil, 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 is available for paraffin oil.

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*, [5] 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.[6] 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

[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 Absorptive Properties of Carbon Black.

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[2] *Annual Book of ASTM Standards*, Vol 09.01.

[3] *Annual Book of ASTM Standards*, Vol 14.02.

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

[5] Available from C. W. Brabender Instruments, Inc., 50 E. Wesley St., South Hackensack, NJ 07606 and from HITEC Luxembourg, 5 Rue de l'Eglise, L-1458 Luxembourg.

[6] *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.

mm^2/s (cSt) at 40° C[7]

6.4 ASTM D24 Standard Reference Blacks, SRB-6.[8]

7. Sampling

7.1 Samples shall be taken in accordance with Practices D 1799 and D 1900.

8. Calibration and Standardization

8.1 *Absorptometer*:

8.1.1 *Model*—Two different types of absorptometers are in use: older models based on springs and mechanical indication of torque (Brabender Type A and B) and the current ones (Brabender Type E and Hitec Type H) equipped with a load cell and a digital torque display. 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 a velvetized stainless steel mixing bowl. Other chamber materials like aluminum, soft- or hard-anodized, are also acceptable provided they give the correct reading for the SRB F-6 after calibration. The surface finish of the mixer chamber is critical for maintaining proper calibration, and the bowl should not be modified to achieve calibration.

NOTE 1—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 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 *Spring tension (Type A and B)*—It is recommended that the torque spring is adjusted so that the SRB F-5 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.

NOTE 2—The absorptometers Type E 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 for SRB F–6.

8.2.3 *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 Type H has an appropriate software damping. Make sure that these damping options are activated.

8.2.4 *Torque limit switch or the indicator set point*—If the end-point of the test is determined by a fixed torque limit, the setting of the torque limit switch, also called indicator set-point, has to be selected using one of the following three procedures:

8.2.4.1 Procedure A: *End-point at fixed torque level*—This "classical" method is well suited for tread blacks but often leads to problems when low-torque carcass blacks are to be tested. Adjust the torque limit switch or the indicator set point in such way that the SRB F-6 gives a value of 133.6 ± 3.3 cm^3/100 g.

8.2.4.2 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[9] and to read the end-point at 70 % of the maximum of the torque achieved. Set the torque limit switch or the indicator set point to full scale in order to disable the automatic shut-off of the absorptometer.

8.2.4.3 Procedure C: *End-point at a fixed, but reduced torque level*—The reduced value on SRB F-6 still needs to be established. For now, if procedure C is desirable, use SRB-5 series standards.

8.2.5 *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.

8.3 *Standardization*:

8.3.1 Physically calibrate the test apparatus 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 3—When only tread or carcass type carbon blacks are to be tested, the calibration can be limited to either the three tread (A-6, B-6, C-6) or the three carcass (D-6, E-6, F-6) type 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). It is recommended that separate carcass and tread calibration curves be maintained.

NOTE 4—Note that the standard value of SRB F-6 (133.6 cm^3/ 100g) has to be used as the y value in the regression, regardless of the absorptometer calibration procedure (A, B or C) used in 8.2.5.

8.3.4 Normalize the values of all subsequent samples as follows:

[7] Three paraffin oils have been found suitable including Marcol 82 and Marcol 9 from Exxon and Sunpar LW107 from SUNOCO.

[8] The sole source of supply of the apparatus known to the committee at this time is Laboratory Standards and Technologies, 227 Somerset, Borger, TX 79007. 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.

[9] A data acquisition system designed for recording and evaluating oil measurements is available from HITEC Luxembourg, 5 Rue de l'Eglise, L-1458 Luxembourg