
International Standard



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Rubber compounding ingredients — Carbon black — Determination of dibutylphthalate absorption number — Part 1 : Method using absorptometer

*Ingrédients de mélange du caoutchouc — Noir de carbone — Détermination de l'indice d'absorption de phtalate de dibutyle —
Partie 1 : Méthode à l'absorptiomètre*

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Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 4656/1 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*.

ISO 4656/1 was first published in 1978. This second edition cancels and replaces the first edition, of which it constitutes a minor revision.

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0 Introduction

The degree of aggregation of carbon black particles affects the vulcanizate and other properties of rubber mixes in which the black is incorporated. The space between the agglomerates of carbon black is dependent on the degree of aggregation of the black. The volume of this space may be estimated from the volume of dibutylphthalate absorbed per unit mass of carbon black. The dibutylphthalate absorption is therefore an indication of the degree of aggregation and agglomeration of the carbon black.

1 Scope and field of application

This part of ISO 4656 specifies a method using an absorptometer for the determination of the dibutylphthalate absorption number of carbon black for use in the rubber industry.

NOTE — ISO 4656/2 specifies a method based on the use of a "plastograph" or "plasticorder".

2 References

ISO 1126, *Carbon black for use in the rubber industry — Determination of loss on heating*.

ISO 6809, *Rubber compounding ingredients — Carbon black — Standard reference blacks*.

3 Principle

Dibutylphthalate is added to a test portion of the carbon black which is kept in motion by means of rotating blades. As the liquid is added, the mixture changes from a free-flowing powder to a semi-plastic mass. The end-point for the determination is reached when the torque resulting from this change in viscous properties attains a pre-set value.

4 Reagent

Dibutylphthalate, ρ_{25} 1,045 to 1,050 g/cm³.

5 Apparatus

5.1 Absorptometer¹⁾, type A or type B (see annex A), consisting of the following essential parts.

5.1.1 Mixing chamber.

5.1.2 Motor-driven rotors, which fit into the mixing chamber (5.1.1) and rotate at a frequency of 2,08 Hz.

5.1.3 Torque-sensing device, consisting of a spring and a dashpot having a damper control valve operating a torque-limit switch which automatically stops the rotors (5.1.2) and the burette (5.1.4) when a pre-selected torque is reached. Instructions for checking and adjusting the settings are given in annex A.

5.1.4 Constant-rate burette, pre-set to deliver dibutylphthalate at a rate of $4,0 \pm 0,024$ cm³/min. Instructions for checking the burette are given in annex B.

5.2 Oven, gravity convection type, capable of being controlled at 105 ± 2 °C or 125 ± 2 °C.

5.3 Balance, accurate to 0,01 g.

5.4 Desiccator.

5.5 Apparatus capable of pulverizing carbon black²⁾, if pulverizing is found to be necessary (see note under 8.2).

5.6 Spatula.

1) This apparatus is available commercially. Details may be obtained from the Secretariat of ISO/TC 45 (BSI) or from the ISO Central Secretariat.

2) A coffee bean grinder is suitable.

6 Sample preparation

Dry an amount of the sample of carbon black sufficient for at least three test portions (see 8.3) for 1 h in the oven (5.2), controlled at $105 \pm 2 \text{ }^{\circ}\text{C}$ or $125 \pm 2 \text{ }^{\circ}\text{C}$, as specified in ISO 1126. Allow to cool to ambient temperature in the desiccator (5.4). Keep the dried sample in the desiccator until ready for testing.

7 Conditions of test

The test should preferably be carried out at ambient conditions of either $23 \pm 2 \text{ }^{\circ}\text{C}$ and $(50 \pm 5) \%$ relative humidity or $27 \pm 2 \text{ }^{\circ}\text{C}$ and $(65 \pm 5) \%$ relative humidity.

It is recommended that the reagent and apparatus be allowed to stand in the test room long enough to reach ambient temperature.

8 Procedure

8.1 Checking the absorptometer and constant rate burette

Proceed as described in annexes A and B.

8.2 Calibration of the absorptometer

Follow the procedure specified in 8.3 and 8.4, using standard reference blacks indicated in ISO 6809.

NOTE — Some machines, particularly those with highly polished rotors and chambers, may give high and inconsistent results for N 650, N 660 and N 683 blacks because of erratic torque development near the end-point. Sometimes no end-point is obtained at all.

In these cases, it is recommended that such blacks be pulverized before weighing.

Alternatively, the spring tension may be reduced and/or the damper valve opened further.

If such changes are made, it will be necessary to recalibrate the apparatus using all the standards.

In some cases, the correct value may be obtained by using a torque-limit setting of less than 5. (See 8.4.2.)

Work is in progress from which it may be possible to recommend a suitable chamber specification to avoid these problems.

Each standard reference black shall be tested a sufficient number of times to establish firm measured values.

If, after checking and adjustment, an apparatus is still found to give values outside the accepted ranges, the regression of the standard values on the measured values shall be calculated by the method of least squares. Alternatively, a graph of observed versus accepted values may be plotted.

The test values of subsequent samples shall be corrected by applying the appropriate equation or graph.

The standard reference blacks shall be retested periodically and if necessary new equations shall be calculated or new graphs shall be plotted.

8.3 Test portion

Weigh, to the nearest 0.02 g, a mass of the dried sample of carbon black in accordance with table 1.

Table 1 — Mass of test portion

Type of carbon black	Mass of test portion g
N 472	15
N 630, N 642 and N 700 series, except N 765 and N 785	25
N 800 and N 900 series	40
All other types	20

NOTE — With high bulk density blacks which do not sufficiently fill the mixing chamber (5.1.1), it may be necessary to use a larger test portion of carbon black so that enough torque is developed to activate the torque-limit switch.

8.4 Determination

8.4.1 Transfer the test portion (8.3) to the mixing chamber (5.1.1) of the absorptometer (5.1), calibrated as specified in 8.2.

8.4.2 Replace the mixing chamber cover. Set the torque-limit switch (see 5.1.3) to 5. (See note under 8.2.) Check that the speed selector (if fitted) of the torque recording instrument is in the correct position.

8.4.3 Check the reagent flow by swinging the delivery tube over a waste container and switching on. The burette (5.1.4) should deliver an air-free stream of the dibutyl-phthalate (clause 4), and the delivery tube should be free of air bubbles. Switch off. Re-centre the delivery tube over the mixing chamber and switch to "automatic feed".

8.4.4 Set the burette digital counter to zero.

8.4.5 Press the start button.

8.4.6 Record the burette digital counter reading after the instrument has stopped.

8.4.7 Dismantle the mixing chamber, and clean the blades of the rotor (5.1.2) and the mixing chamber with the spatula (5.6).

NOTE — The cleaning process may be simplified by adding some dry black and operating the absorptometer before dismantling, while the burette is re-filling.

8.4.8 Re-assemble the mixing chamber.

9 Expression of results

The dibutylphthalate absorption number D of the carbon black is given, in cubic centimetres per 100 g, by the formula

$$D = \frac{V}{m} \times 100$$