INTERNATIONAL STANDARD

ISO 4656-2

Second edition
1991-12-01

Rubber compounding ingredients — Carbon black — Determination of dibutyl phthalate absorption number —

Part 2: Method using plastograph or plasticorder

Ingrédients de mélange du caoutchouc — Noir de carbone — Détermination de l'indice d'absorption de phtalate de dibutyle — Partie 2: Méthode au plastographe ou au plasticorder

Reference number
ISO 4656-2:1991(E)

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 4656-2 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*.

This second edition cancels and replaces the first edition (ISO 4656-2:1981), of which it constitutes a technical revision.

ISO 4656 consists of the following parts, under the general title *Rubber compounding ingredients — Carbon black — Determination of dibutyl phthalate absorption number*:

— *Part 1: Method using absorptometer*

— *Part 2: Method using plastograph or plasticorder*

Annexes A and B form an integral part of this part of ISO 4656.

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Introduction

The degree of aggregation of carbon black particles affects the vulcanizate and other properties or 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 particles. The volume of this space may be estimated from the volume of dibutyl phthalate absorbed by a given mass of carbon black. The dibutyl phthalate absorption is therefore an indication of the degree of aggregation of the carbon black.

Rubber compounding ingredients — Carbon black — Determination of dibutyl phthalate absorption number —

Part 2:
Method using plastograph or plasticorder

1 Scope

This part of ISO 4656 specifies a method using a plastograph or plasticorder for the determination of the dibutyl phthalate absorption number of carbon black for use in the rubber industry.

NOTE 1 ISO 4656-1:1985, *Rubber compounding ingredients — Carbon black — Determination of dibutyl phthalate absorption number — Part 1: Method using absorptometer*, specifies a method based on the use of an absorptometer.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 4656. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this part of ISO 4656 are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 1126:1985, *Rubber compounding ingredients — Carbon black — Determination of loss on heating.*

ISO 6809:1989, *Rubber compounding ingredients — Carbon black — Standard reference blacks.*

3 Principle

Dibutyl phthalate is added drop by drop 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 viscosity properties attains a pre-set value of 400 torque units or 70 % of the maximum achievable torque value, calculated from a torque curve.

4 Reagent

4.1 Dibutyl phthalate, ϱ_{25} 1,045 Mg/m^3 to 1,050 Mg/m^3.

5 Apparatus

5.1 Plastograph or **plasticorder**[1], consisting of the elements given in 5.1.1 and 5.1.4.

5.1.1 Special mixing chamber.

5.1.2 Constant-rate burette, which delivers 4 cm^3/ min.

5.1.3 Inductive shut-off selector.

5.1.4 Recorder, for recording torque.

1) Plastograph and plasticorder are examples of suitable apparatus available commercially. This information is given for the convenience of users of this part of ISO 4656 and does not constitute an endorsement by ISO of either apparatus.

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

5.3 Balance, accurate to 0,01 g.

5.4 Desiccator.

5.5 Apparatus capable of pulverizing carbon black[2], if pulverizing is found to be necessary (see 8.2, note 2).

5.6 Spatula.

6 Sample preparation

Dry an adequate amount of the sample of carbon black for 1 h in the oven (5.2) maintained at 105 °C ± 2 °C or 125 °C ± 2 °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 °C ± 2 °C and (50 ± 5) % relative humidity or 27 °C ± 2 °C and (65 ± 5) % relative humidity.

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

8 Procedure

8.1 Adjustment and checking of the plastograph or plasticorder and constant-rate burette

Proceed as described in annexes A and B.

8.2 Calibration of the plastograph or plasticorder

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

NOTE 2 Some machines, particularly those with highly polished rotors and chambers, may give high and inconsistent results for N 650, N 660, N 683 and N 765 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 out the test portion.

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 a new graph 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

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).

8.4.2 Start the plastograph or plasticorder (5.1) together with the torque recorder (5.1.4) and then the drop-by-drop flow of dibutyl phthalate (4.1). The flow of dibutyl phthalate will shut off inductively at the predetermined torque level of 400 plastograph units (total measuring range 0 to 1 000 units). Record the volume of dibutyl phthalate used.

For carbon blacks of ASTM groups N 500 to N 700, the 400 plastograph units correspond generally with the 70 % value of the maximum achievable torque level.

2) A coffee bean grinder is suitable.

For carbon blacks of ASTM groups N 100 to N 400, the 400 plastograph units correspond only approximately with the 70 % value of the maximum achievable torque level. Therefore calculate the dibutyl phthalate absorption number on the basis of the 70 % torque of the registered torque curve.

For carbon blacks that do not reach a plastograph/plasticorder torque level of 400 units, register the whole torque curve without inductive shut-off and evaluate the amount of absorbed dibutyl phthalate on the basis of the 70 % torque level.

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

NOTE 3 The cleaning process may be simplified by adding some dry black and operating the instrument before dismantling, while the burette (5.1.2) is re-filling.

8.4.4 Re-assemble the mixing chamber.

9 Expression of results

The dibutyl phthalate absorption number D of the carbon black, expressed in cubic centimetres per 100 g, is given by the equation

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

where

V is the volume, in cubic centimetres, of dibutyl phthalate used in 8.4.2;

m is the mass, in grams, of the test portion (8.3).

10 Test report

The test report shall include the following particulars:

a) a reference to this part of ISO 4656;

b) all details necessary for the complete identification of the sample;

c) the drying temperature used;

d) the conditions of test;

e) the mass of test portion used;

f) the results obtained from the individual determinations and their average.

Annex A
(normative)

Adjustment and checking of the plastograph or plasticorder

Adjust and check the parameters of the plastograph or plasticorder as follows:

Position of the suspension: 1:1

Time for damping from 1 000 mp (1 000 plastograph units) to 100 mp (100 plastograph units): 7 s.

Rotational frequency of the mixing chamber: 125 min^{-1} (2,08 Hz)

Adjustment of the balance head to set the torque to 1 000 mp for full deflection (1 000 plastograph units): Position × 5

Initial loading: None

Pointer deflection for the unloaded mixing chamber (zero value): 5 units to 20 units

Paper advance: 4 cm/min

Annex B
(normative)

Checking of the constant-rate burette

B.1 General

The constant-rate burette is an integral part of the absorption measuring system. Failure of the burette to deliver the specified amount of reagent to the carbon black will result in erroneous absorption readings.

B.2 Reagent

B.2.1 Dibutyl phthalate, ϱ_{25} 1,045 Mg/m^3 to 1,050 Mg/m^3.

B.3 Apparatus

B.3.1 Stop-watch.

B.3.2 Beaker, of capacity 150 cm^3.

B.3.3 Balance, accurate to 0,01 g.

B.3.4 Plastic tubing, resistant to swelling by dibutyl phthalate.

B.4 Preliminary check

Ensure that air is not trapped in the plastic tubing (B.3.4) or the delivery tube, especially above the nozzle. Trapped air can cause incorrect reagent delivery.

B.5 Checking procedure

Check that the "O" ring and plastic tubing have not become softened by the reagent, and assemble the burette.

Fill the burette and delivery tubes with dibutyl phthalate (B.2.1). Ensure that all air is removed from the system.

With the burette completely full, set the stopcock to the delivery position. Run the burette on "delivery" until constant flow is obtained from the delivery tube.

Stop the burette and set the digital counter to zero.

Weigh the beaker (B.3.2) to 0,01 g and position it under the delivery tube.

Simultaneously start the burette and the stop-watch (B.3.1).

At 2 min exactly, stop the burette and record the digital counter reading. Weigh and record the mass of reagent delivered. Repeat this operation using time-intervals of 4 min and 8 min.

B.6 Assessment of checking

B.6.1 Calculate the volume V of reagent delivered, in cubic centimetres, using the equation

$$V = \frac{m_1}{\varrho}$$

where

m_1 is the mass, in grams, of reagent delivered;

ϱ is the density, in megagrams per cubic metre, of the reagent.

B.6.2 The constant-rate burette is operating satisfactorily if the requirements of table B.1 are met.

Table B.1 — Burette requirements

Time min	Counter reading	Volume of reagent delivered cm^3
2	8,00 ± 0,05	8,00 ± 0,05
4	16,00 ± 0,05	16,00 ± 0,10
8	32,00 ± 0,05	32,00 ± 0,20