
International Standard



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**Rubber compounding ingredients — Carbon black —
Determination of dibutylphthalate 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*

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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.

International Standard ISO 4656/2 was developed by Technical Committee ISO/TC 45, *Rubber and rubber products*, and was circulated to the member bodies in March 1980.

It has been approved by the member bodies of the following countries:

Belgium	Germany, F. R.	South Africa, Rep. of
Brazil	Hungary	Spain
Bulgaria	India	Sweden
Canada	Italy	Thailand
China	Korea, Rep. of	Turkey
Czechoslovakia	Mexico	United Kingdom
Denmark	Netherlands	USA
Egypt, Arab Rep. of	Poland	USSR
France	Romania	

No member body expressed disapproval of the document.

Rubber compounding ingredients — Carbon black — Determination of dibutylphthalate absorption number — Part 2 : Method using plastograph or plasticorder

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 by a mass of carbon black. The dibutylphthalate absorption is therefore an indication of the degree of aggregation of the carbon black.

1 Scope and field of application

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

NOTE — ISO 4656/1 specifies a method based on the use of an absorptometer.

2 Reference

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

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 viscosity properties attains a pre-set value calculated from a torque curve.

4 Reagent

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

5 Apparatus

5.1 **Plastograph or plasticorder**¹⁾, consisting of the following parts :

5.1.1 **Special mixing chamber**.¹⁾

5.1.2 **Constant-rate burette**, which delivers 4 cm³/min.¹⁾

5.1.3 **Inductive shut-off selector**.

5.1.4 **Recorder**, for recording torque.

5.2 **Oven**, gravity convection type, capable of being controlled at 105 ± 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 the note to 8.2).

5.6 **Spatula**.

6 Sample preparation

Dry an adequate amount of the sample of carbon black for 1 h in the oven (5.2), controlled at 105 ± 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.

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

2) A coffee bean grinder is suitable.

7 Conditions of test

The test should preferably be carried out at ambient conditions of either 23 ± 2 °C and 50 ± 5 % relative humidity or 27 ± 2 °C and 65 ± 5 % relative humidity.

It is recommended that the dibutylphthalate 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 blacks¹⁾ (see table 1).

Table 1 — Standard reference blacks

Standard reference blacks	Dibutylphthalate absorption number
	cm ³ /100 g
A ₂	71,0 ± 1
B ₂	97,5 ± 1
C ₂	122,7 ± 1
D ₂	129,5 ± 1
E ₂	90,3 ± 1
F ₂	65,1 ± 1

NOTE — 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.

Each standard reference black shall be tested a sufficient number of times to establish firm measured values. Two test results shall be considered acceptable if their difference does not exceed 1,6 cm³/100 g.

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. It may be necessary to use two equations or graphs, one for F₂, E₂ and D₂ standard

reference blacks and one for A₂, B₂ and C₂ standard reference blacks.

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 2.

Table 2 — Mass of test portion

Type of carbon black	Mass of test portion, g
N 472	15
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).

8.4.2 Start the plasticorder, then the recorder (5.1.4) and finally the flow of dibutylphthalate. The flow of dibutylphthalate will shut off inductively at the pre-determined torque level (generally at about 400 plastograph units). Record the volume of dibutylphthalate used.

Alternatively, register the whole torque curve without inductive shut off and evaluate this on the basis of the 70 % value.

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

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

8.4.4 Re-assemble the mixing chamber.

1) Specifications for standard reference blacks will form the subject of ISO 6809.

9 Expression of results

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

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

where

V is the volume, in cubic centimetres, of dibutylphthalate used;

m is the mass, in grams, of the test portion.

10 Test report

The test report shall include the following particulars :

- a) a reference to this International Standard;
- b) a complete identification of the sample;
- c) the conditions of test;
- d) the mass of test portion used;
- e) the results obtained from the individual determinations and their average.

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Annex A

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 to 20 units

Paper advance : 4 cm/min

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Annex B

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

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

B.3 Apparatus

B.3.1 Stop-watch.

B.3.2 Beaker, of capacity 150 cm³.

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

B.3.4 Plastic tubing, resistant to swelling by dibutylphthalate.

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 the dibutylphthalate (clause B.2). 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 the nearest 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 formula

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

where

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

ρ is the density, in grams per cubic centimetre, of the reagent.

B.6.2 The constant-rate burette is operating satisfactorily if the requirements of table 3 are met.

Table 3 — 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

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