International Standard ISO 4656/1

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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.

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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/cm^3.

5 Apparatus

5.1 Absorptometer[1], 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 ± 0,024 cm^3/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 black[2], 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 ± 2 °C or 125 ± 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 ± 2 °C and (50 ± 5) % relative humidity or 27 ± 2 °C and (65 ± 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

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

where

V is the volume, in cubic centimetres of dibutylphthalate used (8.4.6);

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 International Standard;

b) a complete identification of the sample;

c) the conditions of test;

d) the mass of test portion used;

e) the torque-limit switch setting;

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

g) the drying temperature used (105 °C or 125 °C).

Annex A

Checking and adjustment of torque spring and hydraulic damping

(This annex forms an integral part of the Standard.)

A.1 Apparatus

A.1.1 Spring scale, range 0 to 150 N.

A.1.2 String, of length 450 mm, looped at each end. (For type A absorptometer only.)

A.1.3 Torque shaft gauge. (For type A absorptometers only.)[1)]

A.1.4 Stop-watch.

A.2 Absorptometer type A

A.2.1 Checking

Secure the loop from one end of the string (A.1.2) to the nut protruding from the rear of the differential dynamometer (see figure 1).

Wrap the string twice around the dynamometer body in a counter-clockwise direction.

Slip the hook at the bottom of the spring scale (A.1.1) into the free loop of the string (see figure 1).

Set the manual/automatic switch to "automatic", start the absorptometer and set the torque-limit switch to 5.

Apply a constant upward pull to the spring scale until the torque indicator reaches 0 and the instrument shuts off.

Read the force applied by the spring scale.

If the indicated force is between 80 and 90 N, the setting is satisfactory.

A.2.2 Adjustment of the spring

If the instrument has been dismantled since it was manufactured, check the position of the dynamometer coupling collar and torque shaft height and relocate if necessary. Do this by fitting the gauge (A.1.3) between the dynamometer coupling collar and the spring support block. Adjust by raising or lowering the dynamometer collar as necessary to conform to the dimensions of the gauge (see figure 1). Check for the proper height of the torque shaft by inverting the gauge and placing it on top of the torque shaft and spring support block, as shown in figure 1.

Adjust the height by loosening the dynamometer collar and the stop collar and sliding the torque shaft up or down as necessary.

If the spring scale reading is too high, this is an indication of excessive friction or improper torque spring adjustment. Check the entire system for lubrication, worn gears, dry bearings or binding in the measuring head of the gear train due to hardened grease. If all these aspects are satisfactory, adjust the torque spring to give a reading between 80 and 90 N. Correct by adjusting or replacing the spring.

A.2.3 Adjustment of the dashpot damper valve

Check that the dashpot is full of the correct oil[2)]. Adjust the damper valve to provide a full-scale recovery time for the torque-sensing system of 11 ± 1 s. Do this by lifting the assembly by hand and releasing.

A.3 Absorptometer type B

A.3.1 Checking

Place the hook of the spring scale (A.1.1) underneath the rod to which the iso-elastic spring is attached (see figure 2).

Set the manual/automatic switch to "automatic". Start the absorptometer and set the torque shut-off selector to 5.

Apply a constant upward pull to the spring scale until the torque indicator reaches 0 and the instrument shuts off.

Read the force applied from the spring scale.

If the indicated force is between 17,5 and 25 N, the setting is satisfactory.

A.3.2 Adjustment of the iso-elastic spring

If the spring scale reading is outside the above limits, check for excessive friction, proper lubrication, worn gears, dry bearings and hardened grease. Correct by adjusting the screw at the bottom of the spring or move the counter-balance weight.

A.3.3 Adjustment of the dashpot damper valve

Check that the dashpot is full of the correct oil[2)]. Adjust the damper valve (black knob on the rear of the machine) to provide a full-scale recovery time of 3 ± 0,5 s. Do this by lifting the assembly by hand and releasing.

1) See footnote 1) on page 1.

2) General Electric silicone 96 or equivalent.

Dimensions in millimetres

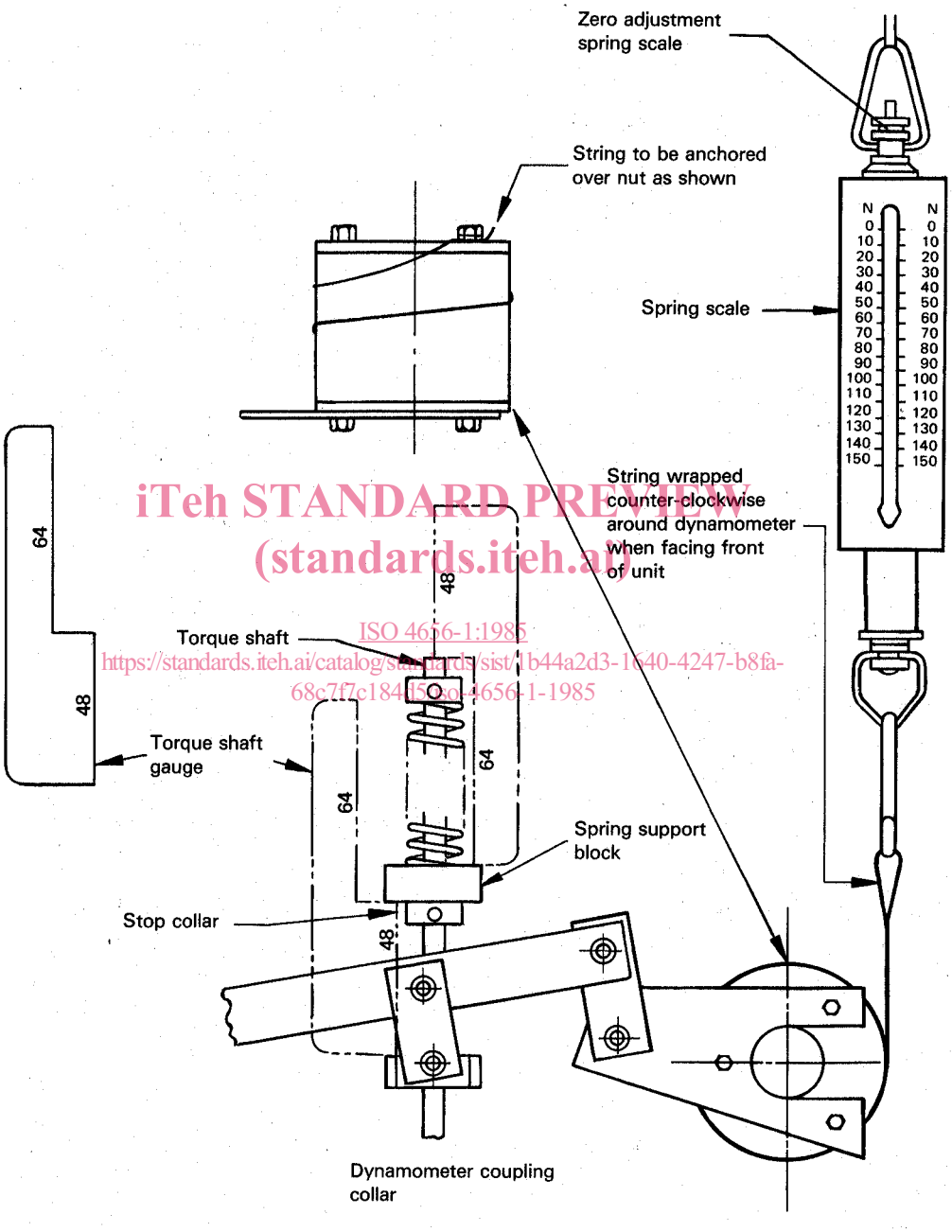

Figure 1 — Absorptometer type A

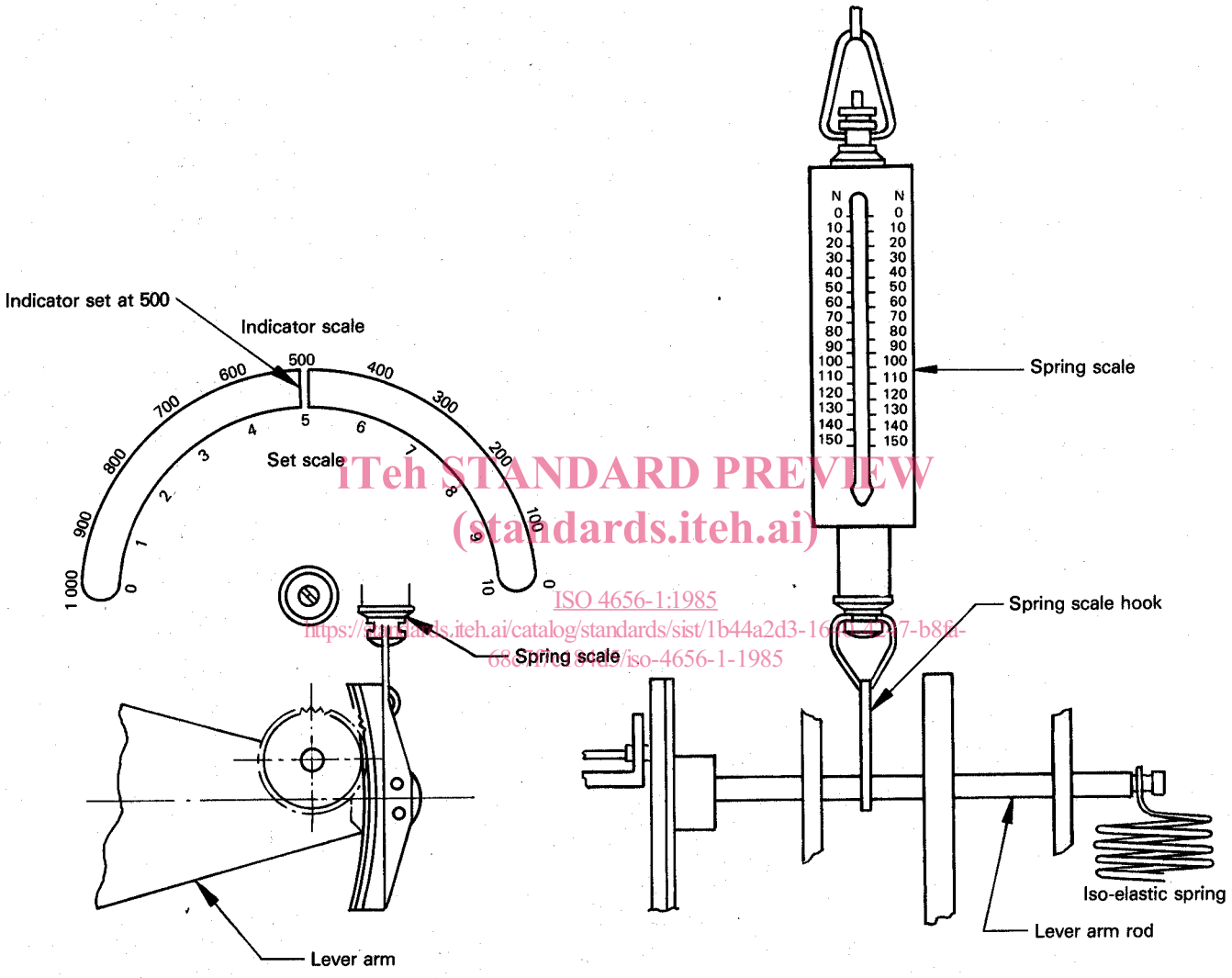

Figure 2 — Absorptometer type B

Annex B

Checking of the constant-rate burette

(This annex forms an integral part of the Standard.)

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

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

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 2 are met.

Table 2 — Burette requirements

Time	Counter reading	Volume of reagent delivered
min		cm³
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