BS ISO 15825:2017

BSI Standards Publication

Rubber compounding ingredients — Carbon black — Determination of aggregate size distribution by disc centrifuge photosedimentometry

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National foreword

This British Standard is the UK implementation of ISO 15825:2017. It supersedes BS ISO 15825:2015 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee PRI/50, Raw materials (including latex) for use in the rubber industry.

A list of organizations represented on this committee can be obtained on request to its secretary.

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Published by BSI Standards Limited 2017

ISBN 978 0 580 92835 2

ICS 83.040.20

Compliance with a British Standard cannot confer immunity from legal obligations.

This British Standard was published under the authority of the Standards Policy and Strategy Committee on 31 March 2017.

Amendments/corrigenda issued since publication

Date	Text affected

BS ISO 15825:2017

INTERNATIONAL STANDARD

ISO 15825

Third edition
2017-03

Rubber compounding ingredients — Carbon black — Determination of aggregate size distribution by disc centrifuge photosedimentometry

Ingrédients de mélange de caoutchouc — Noir de carbone — Détermination de la distribution dimensionnelle des agrégats par photosédimentométrie avec centrifugeuse à disque

Reference number
ISO 15825:2017(E)

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Foreword

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This document was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 3, *Raw materials (including latex) for use in the rubber industry*.

This third edition cancels and replaces the second edition (ISO 15825:2015), which has been technically revised:

— to correct Figure A.1;

— to update the precision data in Annex B following a new interlaboratory trial programme (ITP) conducted in 2015 and 2016.

Rubber compounding ingredients — Carbon black — Determination of aggregate size distribution by disc centrifuge photosedimentometry

1 Scope

This document specifies a method for determining the size distribution of carbon black aggregates, using a disc centrifuge photosedimentometer. This technique is based on the hydrodynamic behaviour of carbon black in a centrifugal field. The determination of the aggregate size distribution is important in the evaluation of carbon black used in the rubber industry.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 1124, *Rubber compounding ingredients — Carbon black shipment sampling procedures*

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at http://www.iso.org/obp

3.1 General terms

3.1.1
carbon black aggregate
discrete, rigid colloidal entity that is the smallest dispersible unit in a suspension

Note 1 to entry: It is composed of extensively coalesced particles.

3.1.2
spin fluid
inert liquid injected into the disc prior to the sample, through which aggregates sediment

Note 1 to entry: Alkaline conditions minimize agglomeration of dispersed aggregates in most cases.

3.1.3
dispersion fluid
liquid in which aggregates are dispersed

3.1.4
Stokes equation
mathematical description of the sedimentation of a spherical particle:

$$D_{st} = \sqrt{\frac{1{,}8 \times 10^{16} \eta \ln\left(\frac{R}{S}\right)}{\left(\rho_1 - \rho_2\right)\omega^2 t}}$$

where

D_{st} is the Stokes diameter (nm);

η is the viscosity of the spin fluid (Pa·s);

R is the distance of the photodetector from the centre of rotation (cm);

S is the distance of the air-liquid interface from the centre of rotation (cm);

t is the time of centrifugation (s);

ρ_1 is the density of the carbon black (Mg/m^3);

ρ_2 is the density of the spin fluid (Mg/m^3);

ω is the rotational velocity (rad/s).

3.1.5
particle density
density of the aggregate in Mg/m^3

Note 1 to entry: For carbon black, 1,86 × 10^3 kg/m^3 (1,86 g/cm^3) is used as a typical value.

3.2 Terms concerning aggregate dimensions

3.2.1
Stokes diameter
D_{st}
diameter of a sphere which sediments in a viscous medium in a centrifugal or gravitational field according to the Stokes equation

Note 1 to entry: A non-spherical object, such as a carbon black aggregate, may also be represented in terms of an equivalent Stokes diameter if it is considered as behaving as a smooth, rigid sphere of the same density and with the same sedimentation rate as the object.

Note 2 to entry: For carbon black, Stokes diameter is expressed in nanometres (nm).

3.2.2
mean diameter
average diameter
D_{mean}
average diameter calculated from the differential mass distribution curve

Note 1 to entry: It represents the first moment of the differential distribution.

Note 2 to entry: In the software of the Brookhaven disc centrifuge the mass distribution is called “Volume (Mass)” and mean diameter is reported as “Mean”.

Note 3 to entry: D_{mean} is used for reporting purposes only.

3.2.3
median
D_{50}
x-value of the point on the mass distribution curve at which 50 % by mass of the test sample is larger and 50 % by mass of the test sample is smaller

Note 1 to entry: It represents the median value of the distribution.

Note 2 to entry: In the software of the Brookhaven disc centrifuge the median Stokes diameter is reported as "d50".

Note 3 to entry: D_{50} is used for reporting purposes only.

3.2.4
mode
D_{mode}
value at which the most frequent diameter occurrence is observed, which is portrayed as a peak in the distribution curve

Note 1 to entry: In some cases, there may be more than one mode indicated.

Note 2 to entry: D_{mode} is used for reporting purposes only.

3.2.5
lower quartile
x-value of the point on the mass distribution curve at which 75 % of the sample is larger, and 25 % smaller

3.2.6
upper quartile
x-value of the point on the mass distribution curve at which 75 % of the sample is smaller, and 25 % larger

3.2.7
quartile ratio
ratio of upper quartile to lower quartile

Note 1 to entry: In the software of the Brookhaven disc centrifuge the quartile ratio is reported as "d75/d25".

3.2.8
ΔD-50
width of the plot of the mass distribution measured at the half-maximum point of the mode, which is a measure of the breadth of the aggregate size distribution

Note 1 to entry: In the software of the Brookhaven disc centrifuge ΔD 50 is reported as "FWHM" (full width at half maximum).

4 Significance and use

Disc centrifuge photosedimentometry produces a rapid mass-differential aggregate size distribution, by continuously measuring the solution turbidity as a function of centrifugation time. In order to obtain a true mass distribution, a light scattering correction shall be applied.

An example of a mass distribution curve is given in Annex A.

5 Apparatus

5.1 Disc centrifuge photosedimentometer (DCP)[1], capable of rotational speeds of 1 000 r/min to 11 000 r/min or greater, with integral spin feed-back control (accuracy and stability of rotational speed better than ± 0,05 %), spin fluid volume from 10 cm^3 to 20 cm^3, stable temperature of spin fluid, stroboscope to monitor the rotating disc both for stability and streaming anomalies, and an appropriate optical turbidity measuring device.

5.2 Energy meter, capable of measuring the energy consumption (in kWh) of the probe-type sonicator.

The energy meter is inserted between an electrical plug of the laboratory and the plug of the power supply cord of the sonicator. The actual energy consumption is indicated on a digital display.

5.3 Probe-type sonicator[2], typically with a nominal power of 200 W or more.

The sonicator should be capable of providing a measured power consumption of at least 60 W. This has been found to be an effective means of dispersing carbon black into discrete aggregates. See Clause 8 for further details.

NOTE Cylindrical tips with 12,7 mm (1/2 inch) diameter have been found to be suitable.

6 Reagents and materials

Unless otherwise stated, use only reagents of recognized reagent grade[3].

6.1 Water, distilled or deionized, grade 3 as defined in ISO 3696.

6.2 Ethanol, absolute.

6.3 Surfactant, non-ionic type[4], 0,02 % to 0,05 % (by mass) solution.

6.4 Dodecane, ≥ 98 % purity (GC grade).

6.5 Spin fluid: Water (6.1) containing surfactant (6.3) which may be adjusted to pH 9,0 to pH 10,0 using 0,1 mol/dm^3 NaOH.

6.6 Dispersion fluid: A solution of 20 cm^3 of ethanol (6.2) and 80 cm^3 of water (6.1) containing a surfactant (6.3). The solution may be adjusted to a pH value between 9,0 and pH 10,0 using 0,1 mol/dm^3 NaOH.

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1) BI-DCP Particle Sizer is available from Brookhaven Instruments Corporation, 750 Blue Point Rd., Holtsville, NY 11742, USA, www.brookhaveninstruments.com. Joyce Loebl DCF 4 is no longer available. It is an example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of these products.

2) Sonoplus 2220, equipped with Sonotrode UW 2200 and horn DH 13 G, is available from BANDELIN electronic GmbH & Co. KG, Heinrichstraße 3-4, D-12207 Berlin, www.bandelin.com. It is an example of a suitable product available commercially. This information is given for the convenience of users of this documentand does not constitute an endorsement by ISO of this product.

3) *Reagent Chemicals: American Chemical Society Specifications*, American Chemical Society, Washington DC, USA. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Reagent Chemicals and Standards*, by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, USA, and the *United States Pharmacopoeia*.

4) Nonidet P-40, from Shell Chemicals, has been found suitable for this application. This information is given for the convenience of users of this documentand does not constitute an endorsement by ISO of the product named. Any other equivalent non-ionic type of surfactant may be used.

7 Sampling

Select carbon black samples from larger-sized lots at random, in either pelletized or non-pelletized form, in accordance with ISO 1124. Label and retain samples for storage or further analysis.

8 Calibration

8.1 The following procedure shall ensure that carbon black agglomerates are completely dispersed into aggregates.

8.2 Prepare a sample of ITRB (or ITRB−2) following the instructions in Clause 9.

8.3 Select sonication energy and sonication mode (e.g. pulsed mode) in such way that 0,005 kWh (18 kJ) are applied. This can typically be achieved by a power of 60 W and a sonication time of 5 min.

8.4 Start sonication and press on start button of the energy-meter, which is plugged in between supply plug and plug of the power cord of the sonicator.

8.5 Stop sonication after 5 min, press stop button on energy-meter and read energy consumption, expressed in kWh.

8.6 If the ITRB or ITRB−2 is entirely dispersed, it will give a mean Stokes diameter ("Mean") of 105 nm ± 5 nm (99 nm ± 5 nm for ITRB−2).

8.7 Test ITRB or ITRB−2 as a standard carbon black on a regular basis before testing actual samples.

8.8 If the value of the standard is too high, increase sonication time and/or power or change the tip of the sonicator.

NOTE The tips of the sonicator are consumed with time.

9 Preparation of test sample

9.1 Weigh 20 mg of carbon black in a weighing vessel.

If the software cannot handle high turbidity values, reduce the sample mass.

9.2 Add to 20 cm^3 of dispersion fluid (6.6).

9.3 Disperse with ultrasonic energy for the time found during calibration (Clause 8), with the dispersing container immersed in a cooling medium, such as iced water, to minimize the heating effect of the sonic energy during sonication. The temperature of the test sample shall be approximately the same as ambient temperature, to minimize thermal gradients in the disc.

Test samples shall be subjected to further sonication if there is any indication of streaming, or more than 1 h has elapsed since sonication.

10 Computer and software setup

Input the appropriate parameters.

a) File name.

b) Sample designation.

c) Fluid temperature: enter the actual temperature, displayed by the instrument after having run the test, for the calculation.

d) Fluid density and fluid viscosity: do not enter a figure, but choose the option “spin fluid = water”.

e) Disc speed.

f) Choose light scattering correction (Mie correction, carbon black).

11 Initiation of procedure

11.1 Set the rotational speed. In general, 8 000 r/min to 11 000 r/min for reinforcing grades and 4 000 r/min to 6 500 r/min for semi-reinforcing grades is suitable. Prior to the test, a 30 min warm-up phase at the chosen speed is necessary. Make sure that the spin fluid used in 11.3 is at room temperature. Keep the air filter of the test instrument clean at all time so that a temperature rise at the rotating disc cell during testing is avoided.

NOTE Effective air ventilation can be achieved by keeping the ventilation window of the instrument open.

11.2 Inject 1,0 cm^3 of ethanol (6.2) and start the centrifuge.

11.3 Inject carefully 15 cm^3 of the spin fluid (6.5) to underlay the ethanol.

11.4 Inject 0,1 cm^3 of dodecane (6.4) on top of the gradient layer to reduce evaporative cooling.

11.5 Allow stabilization of spin fluid, typically for 3 min.

11.6 Set turbidity to zero on the DCP photodetector, if required. This step may be optional, depending on the instrument used in the procedure.

The use of “cut” and “boost” controls is not recommended since it leads to poor reproducibility of the test results.

11.7 Inject 0,25 cm^3 of the test sample, prepared as in Clause 9, into the spinning disc, and immediately start the computer for data acquisition. For injecting the test sample, it is recommended to use a syringe with a needle having an inner diameter of 1,19 mm (16 gauge).

11.8 Read the temperature of the chamber measured by the integrated thermocouple.

11.9 Inspect the disc for hydrodynamic instability or streaming, which may be seen as vortices of sample originating from the dark band of layered carbon black, spiralling towards the outer boundary of the spin fluid. A normal run will produce a smooth, diffuse, circular band of carbon black moving outward towards the perimeter of the disc.

11.10 Continue to run until the turbidity has returned close to the baseline, then stop the run. lf the baseline condition is not reached within 1 h, repeat the test with an increased rotational speed. If the baseline condition is not reached within 1 h even though maximum rotational speed (11 000 rpm) is applied, continue the test until the turbidity has returned close to the baseline.

11.11 Read the temperature of the chamber. lf different from the initial value, use the average of the starting and ending temperatures for calculation. The temperature difference shall not exceed 4 °C.

11.12 The acquired data will be automatically stored. For calculation of the results, use a light-scattering correction. Refer to the user’s manual to find how to activate this feature.

11.13 Remove fluid from the disc, thoroughly clean the disc with water, and dry with a clean paper towel or soft cloth.

12 Test report

The test report shall include the following information:

a) all information necessary for identification of the sample tested;

b) a reference to this document, i.e. ISO 15825;

c) the test parameters (sample mass, sonication time and energy, rotational speed of disc);

d) the type of instrument and software used;

e) the test results based on the mass distribution (aggregate dimensional properties as defined in 4.6);

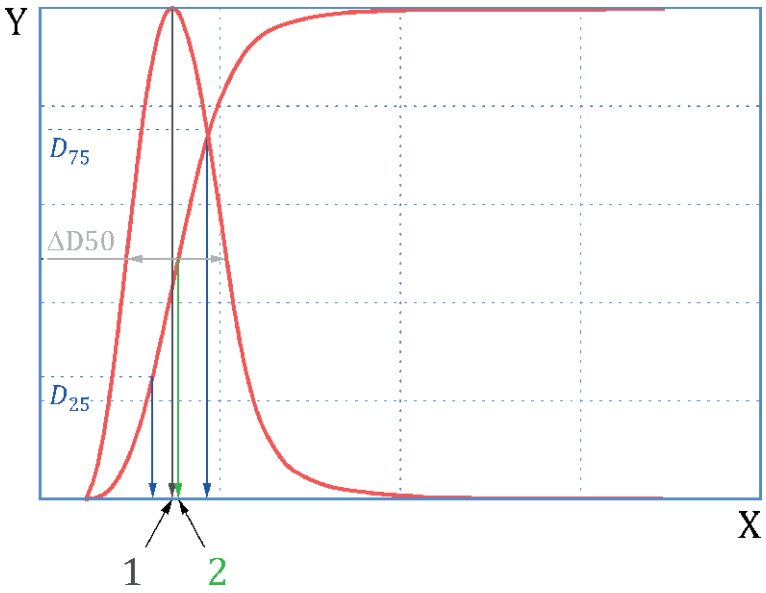
f) any deviations from the procedure specified;

g) any unusual features (anomalies) observed during the test;

h) the date of the test.

Annex A
(informative)

Example of a mass distribution curve



Key

1 mode
2 mean
X diameter
Y cumulative/differential distribution

NOTE $\frac{D_{75}}{D_{25}}$ = quartile ratio

Figure A.1 — Example of a mass distribution curve

Annex B
(informative)

Precision

B.1 The precision of this test method was determined in accordance with ISO/TR 9272. Refer to this document for terminology and other statistical details.

B.2 The precision results give an estimate of the precision to be expected. The precision parameters shall not be used for acceptance/rejection testing of any group of materials without documentation that they are applicable to those particular materials and the specific testing protocols that include this test method.

B.3 A type 1 precision interlaboratory trial programme was conducted. Both the repeatability and the reproducibility determined represent short-term testing conditions. Thirteen laboratories tested four different carbon blacks on two different days in duplicate. However, not all the laboratories tested all the samples, therefore $p \leq 13$, $q = 4$ and $n = 4$. A test result is the value obtained from a single determination. Acceptable difference values were not measured.

B.4 The results of the precision calculations are given in Table B.1, Table B.2. and Table B.3. Outliers have been removed. The number of laboratories remaining after outlier deletion is reported in Tables B.1 to B.3.

Table B.1 — Mean diameter, D_{mean}

Material	Number of laboratories	Mean nm	Within laboratory			Between laboratories		
			s_r	r	(r)	s_R	R	(R)
SRB-B8 (N134)	11	69,6	1,41	3,94	5,66	2,29	6,40	9,20
ITRB (N330)	11	103,4	1,65	4,61	4,46	3,33	9,32	9,01
ITRB-2 (N330)	10	101,4	1,46	4,09	4,04	2,63	7,37	7,27
N772	11	233,5	3,68	10,3	4,42	7,49	21,0	8,98
Pooled or averaged values		126,9	2,26	6,39	5,03	4,45	12,6	9,93

s_r is the within-laboratory standard deviation (in nm)
r is the repeatability (in nm)
(r) is the repeatability (in %)
s_R is the between-laboratory standard deviation (in nm)
R is the reproducibility (in nm)
(R) is the reproducibility (in %)

Table B.2 — Mode, D_{mode}

Material	Number of laboratories	Mean nm	Within laboratory			Between laboratories		
			s_r	r	(r)	s_R	R	(R)
SRB-B8 (N134)	10	59,2	0,84	2,36	3,98	1,34	3,75	6,33
ITRB (N330)	12	86,9	1,57	4,38	5,04	2,93	8,19	9,42
ITRB-2 (N330)	13	94,2	1,32	3,69	3,92	3,13	8,76	9,30
N772	12	181,9	3,54	9,92	5,46	11,7	32,8	18,0
Pooled or averaged values		105,6	2,09	5,91	5,60	6,27	17,7	16,8

s_r is the within-laboratory standard deviation (in nm)
r is the repeatability (in nm)
(r) is the repeatability (in %)
s_R is the between-laboratory standard deviation (in nm)
R is the reproducibility (in nm)
(R) is the reproducibility (in %)

Table B.3 — Full width at half maximum ΔD-50

Material	Number of laboratories	Mean nm	Within laboratory			Between laboratories		
			s_r	r	(r)	s_R	R	(R)
SRB-B8 (N134)	10	48,4	0,97	2,72	5,61	1,72	4,82	9,94
ITRB (N330)	11	76,5	1,29	3,60	4,70	3,14	8,80	11,5
ITRB-2 (N330)	12	73,5	1,62	4,54	6,18	3,73	10,4	14,2
N772	11	223,6	4,04	11,3	5,05	10,3	28,9	12,9
Pooled or averaged values		105,5	2,32	6,56	6,22	5,77	16,3	15,5

s_r is the within-laboratory standard deviation (in nm)
r is the repeatability (in nm)
(r) is the repeatability (in %)
s_R is the between-laboratory standard deviation (in nm)
R is the reproducibility (in nm)
(R) is the reproducibility (in %)

B.5 The precision for the pooled values of the mean diameter, the mode and for ΔD-50 may be expressed as follows.

a) The repeatability (r) has been established as 5,0 % for the mean diameter, 5,6 % for the mode and 6,8 % for ΔD-50. Two single test results (or determinations) that differ by more than 5,0 %, 5,6 % or 6,8 %, respectively, shall be considered suspect and dictate that some appropriate investigative action be taken.

b) The reproducibility (R) has been established as 9,9 % for the mean diameter, 16,8 % for the mode and 18,0 % for ΔD-50. Two single test results (or determinations), produced in separate laboratories, that differ by more than 9,9 %, 16,8 % or 22,9 %, respectively, shall be considered suspect and dictate that some appropriate investigative action be taken.

B.6 In test method terminology, bias is the difference between an average test value and a reference (true) test property value. Reference values do not exist for this test method since the value of the test property is exclusively defined by the test method. Bias, therefore, cannot be determined.

Bibliography

[1] ISO/TR 9272, *Rubber and rubber products — Determination of precision for test method standards*

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