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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
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Foreword

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 3, *Raw materials (including latex) for use in the rubber industry*.

This second edition cancels and replaces the first edition (ISO 15825:2004), which has been technically revised. It also incorporates the Technical Corrigendum ISO 15825:2004/Cor.1:2006. The main changes are as follows.

— Clause 4 has been revised to update some terms and definitions.

— Clause 8 has been revised to specify the sonication parameters in more details and to include the use of an energy meter. New target values for Industry Tint Reference Blacks ITRB and ITRB-2 have been reported.

— Clause 10 has been revised to provide the exact entries to be made by the user when using the test instrument's software.

— Clause 11 has been revised regarding the quantities of fluids used in the disc centrifuge.

— Precision data from the first edition of this International Standard have been moved to an informative Annex B.

— A Bibliography has been added.

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

1 Scope

This International Standard 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, in whole or in part, are normatively referenced in this document and are indispensable for its application. 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 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 has to be applied.

4 Terms and definitions

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

4.1 General terms

4.1.1
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.

4.1.2
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.

4.1.3
dispersion fluid
liquid in which aggregates are dispersed

4.1.4
Stokes equation
mathematical description of the sedimentation of a spherical particle:

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

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).

4.1.5
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) should be used as a typical value.

4.2 Terms concerning aggregate dimensions

4.2.1
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).

4.2.2
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”

4.2.3
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".

4.2.4
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.

4.2.5
lower quartile
x-value of the point on the mass distribution curve at which 75 % of the sample is larger, and 25 % smaller

4.2.6
upper quartile
x-value of the point on the mass distribution curve at which 75 % of the sample is smaller, and 25 % larger

4.2.7
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".

4.2.8
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).

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.

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 International Standard and does not constitute an endorsement by ISO of these products.

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 pH 9,0 to pH 10,0 using 0,1 mol/dm^3 NaOH.

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 Conditions for sonication

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

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

8.1.3 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.

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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 International Standard and 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 International Standard and does not constitute an endorsement by ISO of the product named. Any other equivalent non-ionic type of surfactant may be used.

8.1.4 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.

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

8.1.6 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).

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

8.1.8 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 as described in 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.