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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*

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## Foreword

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International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. 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.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 15825 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*.

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# 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 referenced documents are indispensable for the application 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.

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

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

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

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

NOTE It is composed of extensively coalesced particles.

**4.2**
**spin fluid**
inert liquid injected into the disc prior to the sample, through which aggregates sediment

NOTE Alkaline conditions minimize agglomeration of dispersed aggregates in most cases.

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**4.3**
**dispersion fluid**
liquid in which aggregates are dispersed

**4.4**
**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);

$\eta$ 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);

$\rho_1$ is the density of the carbon black (Mg/m$^3$);

$\rho_2$ is the density of the spin fluid (Mg/m$^3$);

$\omega$ is the rotational velocity (rad/s).

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**4.5**
**particle density**
density of the aggregate in Mg/m$^3$

NOTE For carbon black, 1,86 × 10$^3$ kg/m$^3$ (1,86 g/cm$^3$) may be used as a typical value.

### 4.6 Terms concerning aggregate dimensions

**4.6.1**
**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 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 For carbon black, Stokes diameter is expressed in nanometres (nm).

**4.6.2**
**median Stokes diameter**
$D_{st}$ (for reporting purposes)
$x$-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 It therefore represents the median value of the distribution.


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**4.6.3**
**mode**
value at which the most frequent diameter occurrence is observed, which is portrayed as a peak in the distribution curve

NOTE In some cases, there may be more than one mode indicated.

**4.6.4**
**lower quartile**
$x$-value of the point on the mass distribution curve at which 75 % of the sample is larger, and 25 % smaller

**4.6.5**
**upper quartile**
$x$-value of the point on the mass distribution curve at which 75 % of the sample is smaller, and 25 % larger

**4.6.6**
**quartile ratio**
ratio of upper quartile to lower quartile

**4.6.7**
$\Delta$D-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

## 5 Apparatus

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**5.1 Disc centrifuge photosedimentometer (DCP)**[1], capable of rotational speeds of 1 000 r/min to 10 000 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.

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**5.2 Probe-type sonicator**, with 100 W or more output power. This has been found to be an effective means of dispersing material into discrete aggregates. Although a microtip probe may be acceptable for limited use, a 12,7 mm (1/2 inch) diameter tip with 50 W at the tip is better suited for routine use.[2]

**5.2.1** To find out the time necessary for the best sonication, use standard reference black B5[3] or industry reference black 7 and sonicate it. Sufficient deagglomeration of the carbon black will give a mode of 113 nm ± 5 nm. Vary the time of the sonication to reach this value. Use this time when analysing carbon black samples. Redo this procedure when you change the tip of the probe.

**5.2.2** The tips of the sonicator are consumed with time, therefore it is recommended:

a) to test a standard (for example a standard reference black) before testing actual samples;

b) to change the tip if the mode value is increased by more than 3 nm compared to previous values.

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1) The following examples of DCP instruments have been found to be acceptable: BI-DCP Particle Sizer, available from Brookhaven Instruments Corporation, 750 Blue Point Rd., Holtsville, NY 11742, USA, and Joyce-Loebl DCF-4 (no longer available). This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of the apparatus named. Other apparatus may be used provided it complies with the specified requirements.

2) Suitable examples are the Model W-225 and Model W-380 Probe Sonicator, available from Heat Systems Ultrasonic, 1938 New Highway, Farmingdale., NY 11735, USA. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of the apparatus named. Other apparatus may be used provided it complies with the specified requirements.

3) SRB B5 is no longer commercially available.

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## 6 Reagents and materials

Unless otherwise stated, use only reagents of recognized reagent grade[4].

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

**6.2 Ethanol,** absolute.

**6.3 Surfactant,** non-ionic type[5], 0,02 % to 0,05 % (by mass) solution.

**6.4 Dodecane,** $\geqslant$ 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.

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## 8 Preparation of test sample

**8.1** Weigh 10 mg of carbon black in a weighing vessel. If this mass is too high, use a 5 mg test sample.

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Some software cannot handle high turbidity values. In such cases, use smaller test samples.

**8.2** Add to 50 cm$^3$ of dispersion fluid (6.6).

**8.3** Disperse with ultrasonic energy for the time found in 5.2.1, 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.

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4) *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*.

5) 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.


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## 9 Computer and software setup

Input the appropriate parameters, including file name, fluid temperature, density and viscosity, test sample identification, spin fluid volumes and disc speed. The specific sequence and parameters will depend on the particular software used; two appropriate and typical examples are furnished by the vendors of Joyce-Loebl and Brookhaven Instruments.[6)]

Light-scattering corrections appropriate to carbon black shall be applied. Software to do this is available from Brookhaven.

## 10 Initiation of procedure

**10.1** Set the rotational speed. In general, 8 000 r/min to 10 000 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 10.3 is at room temperature.

**10.2** Inject 0,2 $cm^3$ of ethanol (6.2) and start the centrifuge.

**10.3** Inject carefully an appropriate volume of the spin fluid (6.5), usually 10 $cm^3$ to 20 $cm^3$, to underlay the ethanol.

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

**10.5** Wait for 3 min.

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**10.6** 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.

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**10.7** Inject 0,25 $cm^3$ of the test sample, prepared as in Clause 8, into the spinning disc, and immediately start the computer for data acquisition.

**10.8** Read the temperature of the chamber measured by the integrated thermocouple.

**10.9** 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.

**10.10** Continue to run until the turbidity has returned close to the baseline, then stop the run. If the baseline condition is not reached within 1 h, repeat the test with an increased rotational speed.

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

**10.12** 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.

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

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6) Joyce-Loebl and Brookhaven are examples of instruments 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 instruments.

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