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Rubber compounding ingredients — Carbon black — Determination of oil absorption number (OAN) and oil absorption number of compressed sample (COAN)

Ingrédients de mélange du caoutchouc — Noir de carbone — Détermination de l'indice d'absorption d'huile (OAN) et de l'indice d'absorption d'huile d'échantillons comprimés (COAN)

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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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 4656 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 second edition cancels and replaces the first edition (ISO 4656:2007), which has been technically revised. In addition to a number of minor editorial changes, the following changes have been made:

— a warning concerning the dangers of dibutyl phthalate has been added at the beginning of Clause 4;

— the current standard reference black, SBR D8, has been added to Table 1;

— in A.3.7, “mesh” has been replaced by “nominal size of openings”;

— in Annex A concerning the preparation of compressed test portions, A.5.1.2, A.5.1.11, A.5.2.1 and A.5.2.14 have been rewritten to specify, for each type of carbon black, the mass of carbon black to be taken for the compression procedure and the mass to be subsequently taken as the test portion for determination of the absorption number of the compressed sample;

— the precision data (previously in Clause 10) have been moved to an informative annex (Annex D);

ISO/TR 9272:2005 has been moved from the normative references clause to a bibliography.

Rubber compounding ingredients — Carbon black — Determination of oil absorption number (OAN) and oil absorption number of compressed sample (COAN)

WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

1 Scope

Oil absorption number (OAN) is a measure of the ability of a carbon black to absorb liquids. This property is a function of the structure of the carbon black. Either dibutyl phthalate (DBP) or paraffin oil is acceptable for use with standard pelleted grades, including N-series carbon blacks found in ASTM D1765, although OAN testing using paraffin oil on some speciality blacks and powder blacks might result in unacceptable differences as compared to OAN testing using DBP oil. While studies have shown the two oils to give comparable precision, paraffin oil offers the advantage of being non-hazardous.

This International Standard specifies a method using an absorptometer for the determination of the oil absorption number of carbon black for use in the rubber industry.

The same method is used for the determination of the oil absorption number of compressed samples of carbon black. The procedure for the preparation of the compressed samples is described in Annex A.

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.

ISO 1126, *Rubber compounding ingredients — Carbon black — Determination of loss on heating*

ASTM D1765, *Standard Classification System for Carbon Blacks Used in Rubber Products*

ASTM D4821, *Standard Guide for Carbon Black — Validation of Test Method Precision and Bias*

3 Principle

Oil 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 either a preset value or a defined percentage of the maximum torque, calculated from the recorded torque curve.

4 Reagents

WARNING — Dibutyl phthalate is classified as a "substance of very high concern" (SVHC) as it is considered toxic for reproduction. Its use or marketing is regulated in some countries. Wear suitable protective clothing. Replace by paraffin oil if technically feasible.

4.1 Dibutyl phthalate, ρ_{25} = 1,042 Mg/m^3 to 1,047 Mg/m^3;

or

4.2 Paraffin oil, having a kinematic viscosity of 10 mm^2/s to 34 mm^2/s (10 cSt to 34 cSt) at 40 °C.

4.3 Standard reference blacks (SRBs).[1)]

5 Apparatus

5.1 Absorptometer[2)], consisting of the elements given in 5.2 to 5.5.

Two different basic kinds of absorptometer are in use:

a) electronic models (types C, E and DABS), equipped with a load cell and a digital torque indicator;

b) mechanical models (types A and B), which are older models based on springs and mechanical torque indicators.

Even though the mechanical instruments are no longer commercially available, they may still be used.

5.2 Mixing chamber, stainless steel.

Other chamber materials, such as soft- or hard-anodized aluminium, are acceptable provided they give an acceptable reading for SRB F after calibration (see 8.2.9). The surface finish of the mixing chamber is critical for the maintainance of proper calibration, and the mixing chamber shall not be modified to achieve calibration.

It is recommended that new replacement chambers be pre-polished for 16 h to minimize the effects of chamber surface changes on calibration during their initial use.

5.3 Motor-driven rotors, which fit into the mixing chamber (5.2) and rotate at a frequency of 13 rad/s (125 rpm).

5.4 Torque-sensing device, consisting of a load cell for measuring the torque curve.

Absorptometers of types C and DABS have an integrated data acquisition system for recording the torque curve and calculating the end-point. A type E absorptometer can be equipped with an external data acquisition system[3)]. Absorptometers of types A and B use a spring and a dashpot having a damper control valve operating

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1) Standard reference blacks are available from Laboratory Standards and Technologies, 227 Somerset Street, Borger, TX 79007, USA, Tel/Fax: +1 806 273 3006, E-mail: jwbal@cableone.net, Web site: www.carbonstandard.com. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of the products produced by this company.

2) Suitable apparatus is available from Brabender GmbH & Co KG, Kulturstr. 51-55, 47055 Duisburg, Germany, Tel: +49 203 7788-0, Fax: +49 203 7788-100, E-mail: brabender@brabender.com, Web site: www.brabender.com, and from HITEC Luxembourg, 5, rue de l'Église, L-1458 Luxembourg, Tel: +352 49 84 78-1, Fax: +352 40 13 03, E-mail: info@hitec.lu, Web site: www.hitec.lu. 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. Equivalent apparatus may be used if it can be shown to lead to the same results.

3) This apparatus is commercially available from HITEC, Luxembourg, (see Footnote 2). 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. Equivalent products may be used if they can be shown to lead to the same results.

a torque limit switch which automatically stops the rotors (5.3) and the burette (5.5) when a preselected torque is reached. Instructions for checking and adjusting the settings are given in Annex B.

5.5 Constant-rate burette, pre-set to deliver oil at a rate of (0,067 ± 0,000 4) cm^3/s [(4,0 ± 0,025) cm^3/min]. Instructions for checking the burette are given in Annex C.

5.6 Oven, gravity-convection type, capable of temperature regulation to within ±1 °C at 125 °C and temperature uniformity within ±5 °C.

5.7 Balance, accurate to 10 mg.

5.8 Desiccator.

5.9 Spatula, made from soft material to avoid scratching the mixing chamber.

6 Sample preparation

Dry the mass of carbon black specified in 8.3 for 1 h in the oven (5.6) set at 125 °C, using the drying procedure specified in ISO 1126. Allow to cool to ambient temperature in the desiccator (5.8). Keep the dried sample in the desiccator until ready for testing.

7 Conditions of test

The test should preferably be carried out at a temperature of 23 °C ± 5 °C. The temperature of the mixing chamber should be kept below 30 °C.

8 Procedure

8.1 Checking of the absorptometer and constant-rate burette

Proceed as described in Annexes B and C.

8.2 Calibration and normalization of the absorptometer

8.2.1 Follow the procedure specified in 8.3 and 8.4, using one series of standard reference blacks (SRBs) as indicated in ASTM D4821. Each standard reference black shall be tested a sufficient number of times to establish firm measured values.

Certain semi-reinforcing carbon blacks, i.e. blacks of the N500, N600 and N700 series and thermal blacks (N900), might fail to give an end-point due to insufficient torque level. Therefore the preferred method for testing soft-grade blacks is to record the torque curve using a pen-writer or a data acquisition system and to read the end-point at 70 % of the maximum of the curve. If this approach is applied, make sure that the calibration is performed in the same way.

8.2.2 Test the six standard reference blacks in duplicate to establish the average measured value. Additional values are added periodically on a weekly basis. The rolling average of the measured values is computed from the latest four values.

When only highly reinforcing or semi-reinforcing carbon blacks are tested, the calibration may be limited to either the three hard-grade carbon black standards (A, B, C) or the three soft-grade standards (D, E, F).

8.2.3 Perform a regression analysis using the standard value of the carbon black standard (y-value) and the rolling average of the measured value (x-value). It is recommended that separate soft- and hard-black calibration curves be maintained.

8.2.4 Normalize the values of all subsequent determinations as follows:

Normalized value = (measured value × slope) + y-intercept

8.2.5 Alternatively, a table of numbers may be generated, based on the regression equation, to find the correspondence between a measured and calibrated value.

8.2.6 For SRBs that consistently give measured values which are outside the expected variability, the test apparatus shall be recalibrated.

8.2.7 When any absorptometer or calibration changes occur, a new calibration curve shall be initiated as described in 8.2.1.

8.2.8 In most instances, if proper calibration cannot be achieved by following 8.2.2 to 8.2.5, it will be necessary to replace the mixer chamber with one having a better surface finish.

8.2.9 Adjust the torque-limit switch in such a way that the F-grade of the SRB series in use gives a value within its acceptable range [SRB F5: (129,5 ± 1,5) cm^3/100 g; SRB F6: (133,6 ± 3,3) cm^3/100 g; SRB F7: (129,3 ± 2,0) cm^3/100 g]. After calibration, this setting shall not be changed.

8.2.10 Note that, for type C, E and DABS absorptometers:

— the torque-sensing system is preset at 3 s damping;

— if a data acquisition system is used, the values of subsequent determinations are automatically corrected by the software.

8.2.11 For type A and B absorptometers:

— check that the speed selector (if fitted) of the torque-recording instrument is in the correct position;

— it is mandatory to apply the normalization as described in 8.2.2 to 8.2.9 for the calculation of the correct values of the oil absorption number.

8.3 Test portion

8.3.1 OAN determinations

Weigh out, to the nearest 20 mg, a mass of dried carbon black in accordance with Table 1.

Table 1 — Mass of test portion for determination of oil absorption

Type of carbon black	Mass of test portion g
N630, N642, N700 series, except N765	25
N800 and N900 series, SRB D7 and SRB D8	40
All other types (including N765)	20

With high-bulk-density blacks which do not sufficiently fill the mixing chamber, it might be necessary to use a larger test portion of carbon black, so that enough torque is developed to activate the torque limit switch.

8.3.2 COAN determinations

See Annex A.

8.4 Determination

8.4.1 Transfer the test portion (see 8.3) to the mixing chamber of the absorptometer, calibrated as specified in 8.2.

8.4.2 Replace the mixing-chamber cover.

8.4.3 Check the reagent flow by swinging the delivery tube over the waste container and switching on. The burette (5.5) shall deliver an air-free stream of the oil (4.1 or 4.2), and the delivery tube shall 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 counter to zero. If a data acquisition system is used, this is done automatically by the system.

8.4.5 Press the start button.

8.4.6 Record the burette counter reading after the instrument has stopped.

8.4.7 Dismantle the mixing chamber and clean the blades of the rotors (5.3) and the mixing chamber with the spatula (5.9).

The cleaning process may be simplified by adding some dry black and operating the absorptometer before dismantling while re-filling the burette.

8.4.8 Re-assemble the mixing chamber.

9 Expression of results

The oil absorption number OAN (or COAN, if the sample has been compressed prior to the test) of the carbon black, expressed in cubic centimetres per 100 g, is given by the following equation:

$$\text{OAN (or COAN)} = \frac{V}{m} \times 100$$

where

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

m is the mass, in grams, of the test portion (see 8.3).

Normalize the measured value using the equation in 8.2.4.

10 Precision and bias

See Annex D.

11 Test report

The test report shall include the following particulars:

a) a reference to this International Standard;

b) all details necessary for the complete identification of the sample;

c) the drying temperature used (125 °C);

d) whether the carbon black was compressed before testing (COAN) or not (OAN);

e) the conditions of test;

f) the mass of the test portion used;

g) the method used for the determination of the end-point (fixed torque-limit switch setting or 70 % of maximum torque);

h) the value of the torque-limit switch setting;

i) the results obtained from the individual determinations, and their average;

j) the date of the test.

Annex A
(normative)

Preparation of compressed test portions

A.1 Scope

This annex specifies procedures for the preparation of samples for the determination of the oil absorption number of compressed samples of carbon black for use in the rubber industry.

A.2 Principle

A quantity of dry carbon black is compressed four times at a pressure of 165 MPa and then the oil absorption number is determined as described in this International Standard.

A.3 Apparatus

A.3.1 Balance, accurate to 10 mg.

A.3.2 Oven, gravity-convection type, capable of temperature regulation to within ±1 °C at 125 °C and temperature uniformity within ±5 °C.

A.3.3 Compression cylinder and piston (type A or B).

A.3.4 Hydraulic press, electrically operated, having sufficient power to force the piston into the cylinder during compression of the sample at a rate of 4,2 mm/s and to attain a final pressure of 165 MPa on the sample.

A.3.5 Spatula.

A.3.6 Steel beaker.

A.3.7 Sieve, 850 µm nominal size of openings.

A.3.8 Brush, 38 mm stiff bristle.

A.3.9 Desiccator.

A.4 Sample preparation

Dry an adequate amount of the sample of carbon black for 1 h in the oven (A.3.2) set at 125 °C. Allow to cool to ambient temperature in the desiccator (A.3.9).