

BS ISO 4656:2012



BSI Standards Publication

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

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National foreword

This British Standard is the UK implementation of ISO 4656:2012. It supersedes BS ISO 4656:2007 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee PRI/50, Rubber - Raw, natural and synthetic, including latex and carbon black.

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

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

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Date	Text affected
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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.

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 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, $\rho_{25} = 1,042 \text{ Mg/m}^3$ to $1,047 \text{ Mg/m}^3$;

or

4.2 Paraffin oil, having a kinematic viscosity of $10 \text{ mm}^2/\text{s}$ to $34 \text{ mm}^2/\text{s}$ (10 cSt to 34 cSt) at $40 \text{ }^\circ\text{C}$.

4.3 Standard reference blacks (SRBs).¹⁾

5 Apparatus

5.1 Absorptometer²⁾, 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 system³⁾. Absorptometers of types A and B use a spring and a dashpot having a damper control valve operating

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 $(0,067 \pm 0,000 4) \text{ cm}^3/\text{s}$ [$(4,0 \pm 0,025) \text{ cm}^3/\text{min}$]. Instructions for checking the burette are given in Annex C.

5.6 Oven, gravity-convection type, capable of temperature regulation to within $\pm 1 \text{ }^\circ\text{C}$ at $125 \text{ }^\circ\text{C}$ and temperature uniformity within $\pm 5 \text{ }^\circ\text{C}$.

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 $125 \text{ }^\circ\text{C}$, 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 $23 \text{ }^\circ\text{C} \pm 5 \text{ }^\circ\text{C}$. The temperature of the mixing chamber should be kept below $30 \text{ }^\circ\text{C}$.

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:

$$\text{Normalized value} = (\text{measured value} \times \text{slope}) + y\text{-intercept}$$

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: $(129,5 \pm 1,5)$ cm³/100 g; SRB F6: $(133,6 \pm 3,3)$ cm³/100 g; SRB F7: $(129,3 \pm 2,0)$ cm³/100 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).

A.5 Procedure

A.5.1 Procedure using type A compression cylinder (see Figure A.1)

A.5.1.1 Place the bottom seal plate and the compression cylinder (A.3.3) in the correct position in the hydraulic press (A.3.4). Move the handle of the seal plate to check its position in the support plate. Rotate the cylinder to be certain it fits on the seal plate.

A.5.1.2 Weigh out, to the nearest 0,1 g, a mass of dried carbon black in accordance with Table A.1.

Table A.1 — Mass of dried carbon black weighed out for compression

Type of carbon black	Mass weighed out
	g
N630, N642, N700 series, except N765	30
N800 and N900 series, SRB D7 and SRB D8	45
All other types (including N765)	25

NOTE In order to have sufficient carbon black for the oil absorption test after compression, the mass weighed out is increased by 5 g compared to the mass of the test portion given in Table 1.

A.5.1.3 Pour this sample into the compression cylinder.

A.5.1.4 Place the piston in the cylinder with the nylon spacer next to the carbon black. Rotate the piston slightly while pressing it by hand into the cylinder as far as possible.

A.5.1.5 Adjust alignment of the piston, cylinder and power ram to maintain freedom of movement.

A.5.1.6 Compress the black to 165 MPa. Make certain full pressure is applied, hold for 1 s, then release.

NOTE The oil gauge pressure level is dependent on the bore diameter of the hydraulic cylinder and the surface area of the end of the ram. For the ENERPAC RC256⁴⁾ single-acting cylinder, the oil pressure required to compress the black to 165 MPa (24 000 psi) is 5 710 psi and for the double-acting RR308 it is 4 357 psi, giving 131 kN (29 450 lbf) on the piston of 7,916 cm² area.

A.5.1.7 Raise the power ram enough to remove the bottom seal plate. Then lower the ram to press the piston and carbon black through the cylinder into the steel beaker (A.3.6).

A.5.1.8 Wipe the cylinder, piston and bottom seal plate free of carbon black dust and replace the bottom seal plate and cylinder in the press stand as described in A.5.1.1.

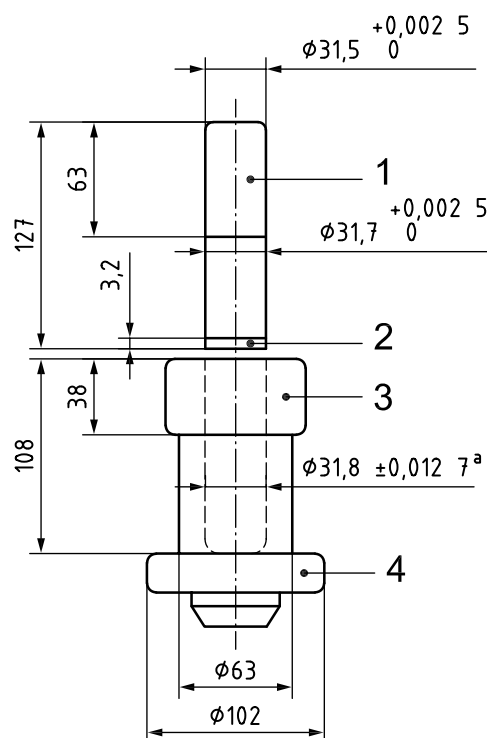
A.5.1.9 Break up the carbon black with the spatula (A.3.5) so that the largest agglomerate is 0,25 cm wide. Brush the compressed black through the sieve (A.3.7) into the receiver pan and pour the black back into the cylinder. Ensure that no black is lost during this procedure.

A.5.1.10 Repeat A.5.1.4 to A.5.1.9, compressing the carbon black a total of four times. Retain the black after the fourth compression. If the compressed black is not to be tested within 15 min after compression, store it in the desiccator (A.3.9), or re-dry it for 1 h at 125 °C as specified in Clause A.4 prior to testing.

4) ENERPAC RC256 and RR308 are the trade names of equipment supplied by ENERPAC. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of the equipment named. Equivalent equipment may be used if it can be shown to lead to the same results.

A.5.1.11 For the determination of the oil absorption, weigh out, to the nearest 20 mg, a mass of the compressed carbon black in accordance with Table 1.

Dimensions in millimetres



Key

- 1 piston
- 2 nylon spacer
- 3 cylinder
- 4 bottom seal plate

^a Inside diameter (inside finish 0,304 8 µm).

Figure A.1 — Essential features of type A compression cylinder and piston

A.5.2 Procedure using type B compression cylinder (see Figure A.2)

A.5.2.1 Weigh out, to the nearest 0,1 g, a mass of dried carbon black in accordance with Table A.1.

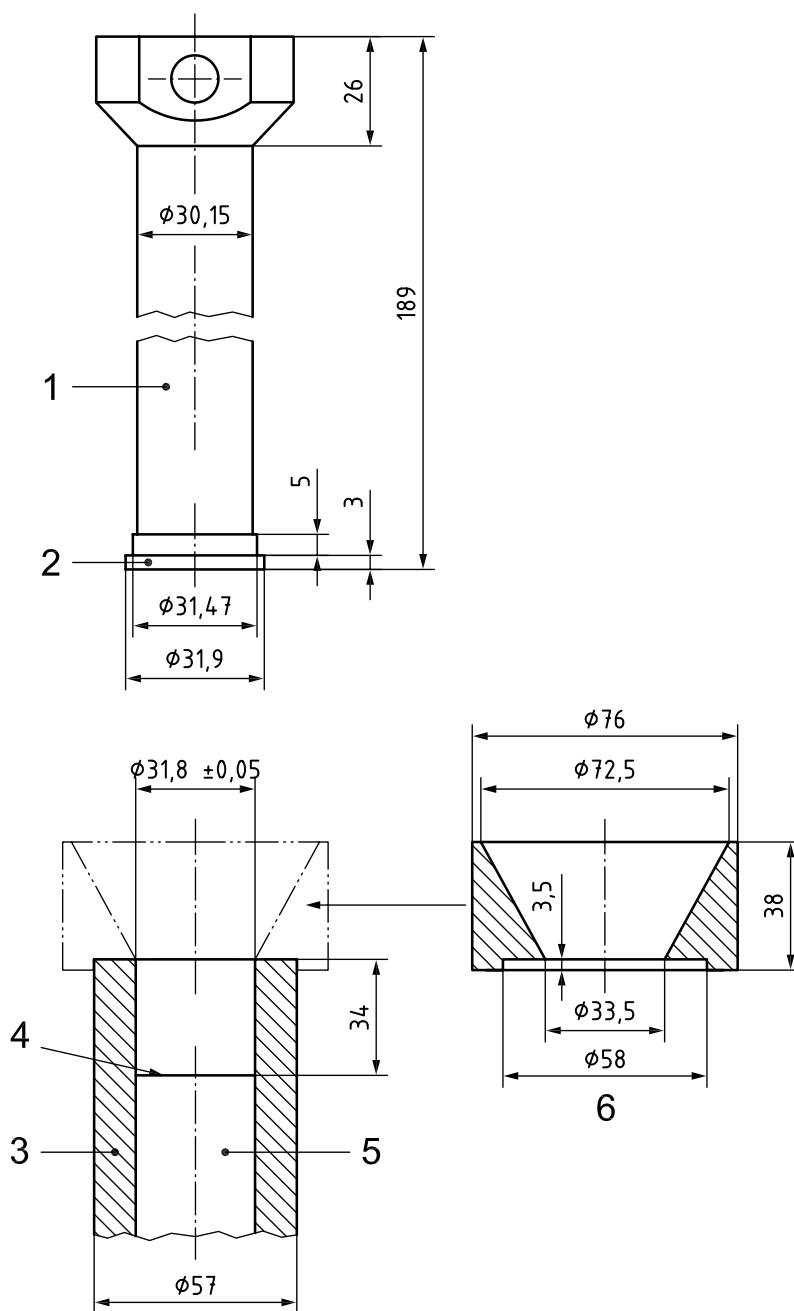
A.5.2.2 Pour the carbon black into the compression cylinder.

A.5.2.3 Compress the black to 165 MPa by operating the ram using a downward movement to open the right-hand valve. Press until the gauge located above the apparatus reaches 103 MPa (15 000 psi) and release immediately.

NOTE The oil gauge pressure level is dependent on the base diameter of the hydraulic cylinder and the cross-sectional area of the piston. For this apparatus, 103 MPa (15 000 psi) on the gauge exerts 165 MPa on the piston.

A.5.2.4 Raise the ram by operating the right-hand valve in an upwards direction. Continue until the ram is approximately 4 cm clear of the cylinder, i.e. level with the top of the conical collar.

A.5.2.5 Raise the cylinder piston by operating the left-hand valve in an upwards direction. Continue until the compressed sample is clear of the cylinder and is broken by contact with the raised ram. The carbon black will fall into the conical collar.



Key

- 1 piston
- 2 nylon spacer
- 3 cylinder
- 4 top
- 5 ejection piston
- 6 loading funnel

Figure A.2 — Essential features of type B compression cylinder and piston

A.5.2.6 Break up the carbon black with a spatula, so that the largest agglomerate is 0,25 cm wide. Ensure that no black is lost during this procedure.

A.5.2.7 Lower the piston and allow the black to fall back into the cylinder. If necessary, brush the inside of the collar with the brush (A.3.8) to return all the black to the cylinder.

A.5.2.8 Repeat steps A.5.2.3 to A.5.2.7 on a further two occasions.

A.5.2.9 Remove the collar and repeat step A.5.2.3 once again.

A.5.2.10 Raise the ram to its top position using the right-hand valve control.

A.5.2.11 Push the carbon black out of the cylinder by raising the piston using the left-hand valve control.

A.5.2.12 Return the piston to its low position by using the left-hand valve control.

A.5.2.13 Transfer the black to the steel beaker. If the compressed black is not to be tested within 15 min after compression, store it in the desiccator (A.3.9), or re-dry it for 1 h at 125 °C as specified in Clause A.4 prior to testing.

A.5.2.14 For the determination of the oil absorption, weigh out, to the nearest 20 mg, a mass of the compressed carbon black in accordance with Table 1.

A.6 Additional information

Data on the oil absorption number of a compressed sample of standard reference black are given in ASTM D4821.

It is important to check results obtained on a standard reference black known to be near to that of the sample, to ensure that the result is within the required range.

Annex B (normative)

Checking and adjustment of torque and hydraulic damping

B.1 Electronic absorptometers — Types C, E and DABS

Electronic absorptometers are calibrated by the manufacturer to give a direct reading of torque in mN·m. This calibration shall not be modified in order to achieve a desired level of torque. If recalibration is necessary, refer to the instrument manufacturer's recommendations.

B.2 Mechanical absorptometer — Type A

B.2.1 Apparatus

B.2.1.1 Spring scale, range 0 N to 150 N.

B.2.1.2 String, of length 450 mm, looped at each end.

B.2.1.3 Torque shaft gauge.

B.2.1.4 Stop-watch.

B.2.2 Checking

Secure the loop at one end of the string (B.2.1.2) to the nut protruding from the rear of the differential dynamometer. Wrap the string twice around the dynamometer body in the counter-clockwise direction.

Slip the hook at the bottom of the spring scale (B.2.1.1) into the free loop of the string.

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 N and 90 N, the setting is satisfactory.

B.2.3 Adjustment of the spring

If the instrument has been dismantled since it was manufactured, check the position of the dynamometer coupling collar and the torque shaft mass and relocate if necessary. Do this by fitting the gauge (B.2.1.3) 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. 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.

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 insufficient lubrication, worn gears, dry bearings or binding in the measuring head of the gear train due to hardened grease. If all these checks are satisfactory, adjust or replace the torque spring so that the reading is between 80 N and 90 N.

B.2.4 Adjustment of the dashpot damper valve

Check that the dashpot is full of the correct oil (General Electric silicone 96 or equivalent⁵⁾). Adjust the damper valve to provide a full-scale recovery time for the torque-sensing system of $11 \text{ s} \pm 1 \text{ s}$. Do this by lifting the assembly by hand and releasing.

B.3 Mechanical absorptometer — Type B

B.3.1 Apparatus

B.3.1.1 Spring scale, range 0 N to 150 N.

B.3.1.2 Stop-watch.

B.3.2 Checking

Place the hook of the spring scale (B.3.1.1) underneath the rod to which the iso-elastic spring is attached.

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 N and 25 N, the setting is satisfactory.

B.3.3 Adjustment of the iso-elastic spring

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

B.3.4 Adjustment of the dashpot damper valve

Check that the dashpot is full of the correct oil (General Electric silicone 96 or equivalent⁵⁾). Adjust the damper valve (black knob on the rear of the machine) to provide a full-scale recovery time of $3 \text{ s} \pm 0,5 \text{ s}$. Do this by lifting the assembly by hand and releasing.

5) General Electric silicone 96 is an example of a suitable oil available commercially. 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.

Annex C (normative)

Checking of the constant-rate burette

C.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.

C.2 Reagents

C.2.1 Dibutyl phthalate, $\rho_{25} = 1,042 \text{ Mg/m}^3$ to $1,047 \text{ Mg/m}^3$ (see warning at beginning of Clause 4);

or

C.2.2 Paraffin oil, having a kinematic viscosity of $10 \text{ mm}^2/\text{s}$ to $34 \text{ mm}^2/\text{s}$ (10 cSt to 34 cSt) at $40 \text{ }^\circ\text{C}$.

C.3 Apparatus

C.3.1 Stop-watch.

C.3.2 Beaker, of capacity 150 cm^3 .

C.3.3 Balance, accurate to 10 mg.

C.3.4 Plastic tubing, resistant to swelling by oil.

C.4 Preliminary check

Ensure that air is not trapped in the plastic tubing (C.3.4) or the delivery tube, especially above the nozzle. Trapped air can cause incorrect reagent delivery.

C.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 oil (C.2.1 or C.2.2). 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 a constant flow is obtained from the delivery tube.

Stop the burette and set the digital counter to zero.

Weigh the beaker (C.3.2) to the nearest 10 mg and position it under the delivery tube.

Simultaneously start the burette and the stop-watch (C.3.1).

At 2 min exactly, stop the burette and record the digital counter reading. Weigh and record the mass of the reagent delivered. Repeat this operation using time-intervals of 4 min and 8 min.

C.6 Assessment of check

Calculate the volume V of reagent delivered, in cubic centimetres, using the equation:

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

where

m_1 is the mass, in grams, of reagent delivered;

ρ is the density, in megagrams per cubic metre, of the reagent.

The constant-rate burette is operating satisfactorily if the requirements of Table C.1 are met.

Table C.1 — Burette requirements

Time min	Counter reading	Volume of reagent delivered cm ³
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

Annex D (informative)

Precision and bias

D.1 Precision

D.1.1 The precision of this test method was determined in accordance with ISO/TR 9272:2005, level 2 method. Refer to ISO/TR 9272:2005 for terminology and other statistical details.

D.1.2 The precision results give an estimate of the precision to be expected. The precision parameters should 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.

D.1.3 A type 1 precision interlaboratory-trials programme was conducted. Both the repeatability and the reproducibility determined represent short-term testing conditions.

For OAN, 16 laboratories tested two carbon blacks twice on each of two different days. Therefore $p = 16$, $q = 2$ and $n = 4$.

For COAN, 15 laboratories tested two carbon blacks twice on each of two different days. Therefore, $p = 15$, $q = 2$ and $n = 4$.

A test result is the value obtained from a single determination. Acceptable difference values were not measured.

D.1.4 The results of the precision calculations are given in Table D.1 (OAN) and Table D.2 (COAN), with the materials arranged in descending order of mean oil absorption number. Outliers have been removed. The number of laboratories remaining after outlier deletion is given in Tables D.1 and D.2.

Table D.1 — Oil absorption number (OAN)

Material	Number of laboratories	Mean OAN cm ³ /100 g	Within-laboratory			Between laboratories		
			s_r	r	(r)	s_R	R	(R)
A (N220)	13	113,6	0,37	1,04	0,92	0,77	2,17	1,91
B (N762)	13	67,5	0,47	1,33	1,97	1,14	3,24	4,79
Average value	13	90,6						
Pooled values			0,42	1,20	1,54	0,98	2,76	3,65

The symbols used are defined as follows:

- s_r is the within-laboratory standard deviation;
- r is the repeatability (in measurement units);
- (r) is the repeatability (in percent);
- s_R is the between-laboratory standard deviation;
- R is the reproducibility (in measurement units);
- (R) is the reproducibility (in percent).

Table D.2 — Oil absorption number of compressed sample (COAN)

Material	Number of laboratories	Mean COAN cm ³ /100 g	Within-laboratory			Between laboratories		
			s_r	r	(r)	s_R	R	(R)
A (N220)	11	98,0	0,33	0,94	0,96	0,54	1,54	1,57
B (N762)	11	60,0	0,42	1,19	1,99	1,12	3,16	5,27
Average value	11	79,0						
Pooled values			0,38	1,07	1,56	0,88	2,49	3,89

The symbols used are defined as follows:

s_r is the within-laboratory standard deviation;
 r is the repeatability (in measurement units);
(r) is the repeatability (in percent);
 s_R is the between-laboratory standard deviation;
 R is the reproducibility (in measurement units);
(R) is the reproducibility (in percent).

D.1.5 The precision for the pooled values of the oil absorption number may be expressed as follows:

a) Repeatability

The repeatability r has been established as 1,2 cm³/100 g for OAN and 1,07 cm³/100 g for COAN. Two single test results (or determinations) that differ by more than 1,2 cm³/100 g (for OAN) or 1,07 cm³/100 g (for COAN) should be considered suspect and dictate that some appropriate investigative action be taken.

b) Reproducibility

The reproducibility R has been established as 2,76 cm³/100 g for OAN and 2,49 cm³/100 g for COAN. Two single test results (or determinations), produced in separate laboratories, that differ by more than 2,76 cm³/100 g (for OAN) or 2,49 cm³/100 g (for COAN) should be considered suspect and dictate that some appropriate investigative action be taken.

D.2 Bias

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 9272:2005, *Rubber and rubber products — Determination of precision for test method standards*

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