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)

bsi.

...making excellence a habit.™

Document Sharing Hub

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 It supersedes BS ISO 4656:2007 which is withdrawn.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.

© The British Standards Institution 2012. Published by BSI Standards Limited 2012

ISBN 978 0 580 77727 1

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 August 2012.

Amendments issued since publication

Date	Text affected

Documents
Document Sharing Hub

Full document details: BS ISO 4656:2012

INTERNATIONAL STANDARD

ISO 4656

Second edition
2012-07-15

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)

Reference number
ISO 4656:2012(E)

Documents
Document Sharing Hub

© ISO 2012

Full document details: BS ISO 4656:2012

COPYRIGHT PROTECTED DOCUMENT

© ISO 2012

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying and microfilm, without permission in writing from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

Documents
Document Sharing Hub
© ISO 2012 – All rights reserved

Full document details: BS ISO 4656:2012

Contents

Page

Foreword ... iv
1 Scope ... 1
2 Normative references ... 1
3 Principle ... 1
4 Reagents ... 2
5 Apparatus ... 2
6 Sample preparation ... 3
7 Conditions of test ... 3
8 Procedure ... 3
8.1 Checking of the absorptometer and constant-rate burette ... 3
8.2 Calibration and normalization of the absorptometer ... 3
8.3 Test portion ... 4
8.4 Determination ... 5
9 Expression of results ... 5
10 Precision and bias ... 5
11 Test report ... 5
Annex A (normative) **Preparation of compressed test portions** ... 7
Annex B (normative) **Checking and adjustment of torque and hydraulic damping** ... 12
Annex C (normative) **Checking of the constant-rate burette** ... 14
Annex D (informative) **Precision and bias** ... 16
Bibliography ... 18

© ISO 2012 – All rights reserved

Documents
Document Sharing Hub
Full document details: BS ISO 4656:2012

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.

© ISO 2012 – All rights reserved
Documents
Document Sharing Hub
Full document details: BS ISO 4656:2012

INTERNATIONAL STANDARD ISO 4656:2012(E)

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.

© ISO 2012 – All rights reserved

Documents
Document Sharing Hub
Full document details: BS ISO 4656:2012

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

---

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.

© ISO 2012 – All rights reserved
Documents
Document Sharing Hub
Full document details: BS ISO 4656:2012

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.

© ISO 2012 – All rights reserved

Documents
Document Sharing Hub
Full document details: BS ISO 4656:2012