INTERNATIONAL STANDARD

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Rubber compounding ingredients — Carbon black — Determination of individual pellet crushing strength

Ingrédients de mélange du caoutchouc — Noir de carbone — Détermination de la force d'écrasement des granules individuels

Reference number
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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 8942 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*.

It cancels and replaces ISO/TR 8942:1988, which has been technically revised.

Introduction

Carbon black for the rubber industry is generally pelletized to reduce dust and facilitate handling. The property of individual pellet crushing strength, among several other properties, may have an influence on the dispersion of carbon black in polymers, on bulk handling and on conveying properties. For these purposes, carbon black is mostly pelletized to around 1 mm in diameter, but the pellet size can reach 1,7 mm. Therefore, in most of the existing test methods used to measure the individual pellet crushing strength, the pellets are selected in sizes ranging from 1 mm to 1,7 mm.

The individual pellet crushing strength is the force necessary to crush one pellet, and the value measured is absolute (i.e. neither per unit volume nor per unit surface area of the pellet). The value varies with the size of the pellet. Therefore, a narrower size range in the sample generally gives a narrower distribution of the measured individual pellet crushing strength.

This International Standard provides two methods for determining the individual pellet crushing strength of carbon black:

— Method A, specified in ASTM D5230, which uses a test sample of a certain pellet size range that is widely used in the industry for typical grades, prepared by passing a sample through a sieve that has an aperture of 1,4 mm to 1,7 mm.

— Method B, which uses a test sample prepared using a sieve that has an aperture of 1,0 mm. This method is used when a more precise result is required, such as in process control or in order to meet a customer's specification.

Rubber compounding ingredients — Carbon black — Determination of individual pellet crushing strength

1 Scope

This International Standard specifies two methods for the determination of the individual pellet crushing strength of carbon black for use in the rubber industry:

— method A: using pellets of size ranging from 1,4 mm to 1,7 mm;

— method B: using pellets of size 1 mm.

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 565, *Test sieves — Metal wire cloth, perforated metal plate and electroformed sheet — Nominal sizes of openings*

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

ASTM D5230, *Standard Test Method for Carbon Black — Automated Individual Pellet Hardness*

3 Terms and definitions

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

3.1
individual pellet crushing strength
force required to crush (i.e. fracture) a carbon black pellet under specified conditions

4 Principle

A number of pellets within the diameter range 1,4 mm to 1,7 mm (method A) or of diameter 1,0 mm (method B) are selected by sieving a carbon black sample (in method B, this is achieved by selecting only those pellets which, during sieving, become lodged in the apertures of a 1,0 mm aperture sieve). The pellets selected are placed one at a time between two parallel plates of a device capable of applying a force. The force which is being applied when the pellet fractures is recorded.

5 Method A

Testing is carried out in accordance with ASTM D5230.

6 Method B

6.1 Apparatus

Usual laboratory apparatus, plus the following:

6.1.1 Manual pellet hardness tester.

Required is a device capable of applying a force at a constant rate and of measuring the force at which the pellet fractures.

The device shall:

— have two parallel plates (a baseplate and a force-application plate) which remain horizontal throughout the whole of the test;

— be capable of applying a force which increases at a constant rate of between 5 cN/s[1)] and 25 cN/s;

— possess a means of measuring this force with an accuracy of 1 cN;

— be capable of being calibrated using deadweights or a force-measuring device that verifies the accuracy of the equipment over the range being used for the test.

The baseplate and force-application plate shall be firmly fixed in order to prevent the pellet from rolling, or otherwise moving, before the start of the test.

Simple, accurate bench dial scales having a 50 g or 200 g dial and with the force-application plate positioned above the scale pan may be used.

6.1.2 Automatic pellet hardness tester[2)], capable of measuring the crushing force to an accuracy of ±0,4 cN and having the following characteristics:

— the ability to load a pellet automatically on to the plate from the pellet feed device;

— the ability to apply the crushing force at a constant rate;

— a control unit which takes the instrument through the test cycle which comprises crushing the pellet under controlled conditions, measuring the crushing force and storing the result and, finally, cleaning the pellet fragments from the plate surface before starting the next cycle;

— an algorithm for detecting the end-point of each individual test, i.e. the maximum force reached before the first occurrence of a reduction in force;

— a software programme for calculating the data required in 6.4 for a specified number of pellets.

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1) 1 cN = 1,019 gf.

2) The following automatic pellet hardness testers are available commercially:

— AS 2000 from Asahisouken Co. Ltd., 3-13-3 Meikeshinmei chuo-ku, Niigata City, Niigata 950-0943, Japan;

— Individual Pellet Hardness Tester from Jaron Technologies LLC, 2338 Duncan St., Pampa, TX 79065, USA (formerly Titan Specialties);

— IPHT from HITEC Luxembourg, 5 rue de l'Église, L-1458 Luxembourg.

This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of the instruments named.

6.1.3 Sieve shaker.

6.1.4 Sieve, complying with the requirements of ISO 565, preferably having a nominal aperture size of 1,0 mm. A sieve with an aperture size of 1,4 mm or 1,7 mm (or some other value) may, however, be used by agreement between the interested parties.

NOTE There is a marked tendency for larger particle sizes to have a higher individual crushing strength. The use of a 1 mm sieve to isolate pellets of diameter 1 mm, as described in 6.2.3 and 6.2.4, is intended to avoid this problem.

6.1.5 Bottom-receiver pan.

6.1.6 Sieve cover.

6.1.7 Sample splitter, single-stage riffle type.

6.1.8 Shallow container, e.g. a dish approximately 300 mm long.

6.2 Sampling and selection of pellets for use as test pieces

6.2.1 Take samples in accordance with the requirements of ISO 1124.

6.2.2 Pass a sufficient quantity of carbon black through the sample splitter (6.1.7) to obtain a representative sample of approximately 100 g.

6.2.3 Assemble the bottom-receiver pan (6.1.5) and sieve (6.1.4) correctly. Transfer the 100 g sample of the riffled carbon black to the sieve, place the sieve cover (6.1.6) on the sieve and transfer the assembly to the sieve shaker (6.1.3). Shake the sieve assembly for $60\,^{+10}_{0}$ s with the minimum of energy input so as to avoid pellet breakdown. If a hammer-type mechanical shaker is used, the hammer shall be at rest.

6.2.4 Remove the pellets left on the sieve by turning it upside-down. Then hold the sieve upside-down over a shallow container (6.1.8) and tap the sieve gently so that the pellets lodged in the apertures fall into the container. Repeat these operations until about 50, 100 or 150 1 mm pellets, depending how many it is intended to test (see 6.2.5), have been obtained. With these pellets at one end of the container, slightly tilt and gently shake the container so that the most spherical pellets roll to the opposite end. Choose 20, 50 or 100 (see 6.2.5) of the most spherical of these pellets.

6.2.5 The number of pellets tested is 20 for normal applications, although, by agreement between the interested parties, either 50 or 100 pellets may be tested if a more precise result is required.

6.3 Procedure

6.3.1 Manual procedure

6.3.1.1 Select one pellet and, using a soft brush, position it in the centre of the baseplate, just under the force-application plate (see 6.1.1).

6.3.1.2 Carefully bring the force-application plate into contact with the pellet. Exercise great care when testing soft pellets which tend to fracture prematurely due to impact on initial contact.

6.3.1.3 Apply force at a constant rate of 25 cN/s until the pellet fractures. Record the force required.

6.3.1.4 Clean the baseplate and force-application plate.

6.3.1.5 Repeat the operations in 6.3.1.1 to 6.3.1.4 until all 20 (or 50 or 100) pellets have been tested.

6.3.2 Automatic procedure

Use an automatic pellet hardness tester operated in accordance with the manufacturer's instructions. Allow the machine to continue operating until all 20 (or 50 or 100) pellets have been tested.

6.4 Calculation and expression of results

6.4.1 Irrespective of the number of pellets tested, calculate and report the average value, in centinewtons, of the forces recorded in 6.3.1 or 6.3.2, rounded to the nearest millinewton (i.e. the nearest 0,1 gf). Report, in addition, the maximum value, in centinewtons, of the forces recorded in 6.3.1 or 6.3.2.

6.4.2 When 50 or 100 pellets have been tested, the following may also be reported, in centinewtons, rounded to the nearest whole number:

— the average value of the five highest forces recorded in 6.3.1 or 6.3.2;

— the average of the highest 10 % of the individual forces recorded in 6.3.1 or 6.3.2.

7 Test report

The test report shall include the following information:

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

b) a reference to this International Standard (i.e. ISO 8942:2010);

c) the method used (A or B);

d) details of the sieve apparatus used;

e) if method B was used, the aperture size of the sieve used to select pellets for use as test pieces;

f) the number of pellets tested;

g) if method A was used, the average pellet crushing strength, reported in accordance with ASTM D5230, or, if method B was used, the average pellet crushing strength and the maximum individual crushing strength, reported in accordance with 6.4.1;

h) other crushing strength values, if required (see e.g. 6.4.2);

i) details of any deviations from the procedure specified in this International Standard as well as details of any incident that might have influenced the results;

j) the date of the test.

Annex A
(informative)

Precision and bias

A.1 Precision

A.1.1 The precision of this test method was determined in accordance with ISO/TR 9272. Refer to this document for terminology and other statistical details.

A.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 supporting documentation that shows that they are applicable to those particular materials, and without the specific testing protocols that include this test method.

A.1.3 A type 1 precision interlaboratory test programme was conducted. Both the repeatability and the reproducibility determined represent short-term testing conditions. Five laboratories tested one carbon black sample using each method three times on different days (i.e. $p = 5$, $q = 1$, $n = 3$). A test result is the value obtained from a single determination. Acceptable difference values were not measured.

A.1.4 The results of the precision calculations are given in Table A.1. The results obtained when testing using the method specified in ISO/TR 8942:1988 are also given in the table for comparison purposes.

A pellet size of 1,0 mm was used when testing using method B of this International Standard. A pellet size range of 1,4 mm to 1,7 mm was used when testing using ISO/TR 8942:1988.

Table A.1 — Individual pellet crushing strength

Test method used	Mean	Within laboratory			Between laboratories		
		s_r	r	(r)	s_R	R	(R)
Method B of ISO 8942:2010	14,97	0,958	2,71	18,1	1,636	4,63	30,9
Method specified in ISO/TR 8942:1988	36,01	2,307	6,53	18,1	5,983	16,93	47,0

s_r is the within-laboratory standard deviation;

r is the repeatability (in measurement units);

(r) is the repeatability (as a percentage);

s_R is the between-laboratory standard deviation;

R is the reproducibility (in measurement units);

(R) is the reproducibility (as a percentage).