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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

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ISO/TR 8942 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products.*

The reasons which led to the decision to publish this document in the form of a technical report type 2 are explained in the Introduction.

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0 Introduction

Carbon black for the rubber industry is generally pelletized to reduce dust and improve handling. The property of individual pellet crushing strength, among several other properties, can have an influence on the dispersion of carbon black in polymers, on bulk handling, and on conveying properties.

Following international efforts to develop a method for the determination of individual pellet hardness, it was agreed that a type 2 technical report should be prepared, since the results of two round-robin tests had shown that, although within-laboratory repeatability of the method under investigation was moderately acceptable, between-laboratories reproducibility was too great to allow the preparation of an International Standard.

It has been agreed to consider the investigation of a new apparatus. Since this will take a considerable amount of time, and since many laboratories are still using the apparatus that was featured in the round-robin tests, it was agreed that this Technical Report should be issued in the interim.

1 Scope and field of application

This Technical Report describes a method of determining the individual pellet crushing strength of carbon black for use in the rubber industry.

2 References

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

ISO 1124, *Rubber compounding ingredients — Carbon black shipment sampling procedures.* [1]

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3 Principle

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A number of pellets with a narrow range of diameters is selected by sieving a carbon black sample. These pellets are placed one at a time between two parallel plates on a device capable of applying a force. The force applied when the pellet breaks is recorded.

4 Apparatus

Usual laboratory apparatus, and

4.1 Device capable of applying a force at a constant rate and of measuring the force as the pellet breaks.

The device shall

— have two parallel plates which remain truly horizontal throughout the complete test, with the upper plate transparent so that the pellet under test is clearly visible;

— be capable of applying a force at a constant rate of between 5 and 25 cN/s*;

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

— have the base plate and the force applicator firmly fixed so as not to cause the pellet to roll or move, immediately prior to the test;

— be capable of being calibrated by dead-weights or by a force-measuring device that will verify the precision of the equipment over the range to be tested.

NOTE — An accurate, simple plate balance with a 200 g dial can be used as a measuring instrument. A force-application device is positioned above the balance plate.

1) At present at the stage of draft. (Revision of ISO 1124 : 1983 and ISO 1310 : 1974.)

* 1 cN/s ≈ 1,019 gf/s

4.2 Sieve shaker.

4.3 Sieves, complying with the requirements of ISO 565, and having nominal aperture sizes of 1,4 mm and 1,7 mm.

4.4 Bottom-receiver pan.

4.5 Top sieve cover.

4.6 Sample splitter, single-stage riffle type.

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

5 Sample preparation

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

5.2 Pass a sufficient quantity of carbon black through the sample splitter (4.6) to obtain a representative sample of approximately 100 g.

5.3 Stack the sieves (4.3) in the following order from bottom to top: bottom receiver pan (4.4), 1,4 mm aperture sieve, 1,7 mm aperture sieve. Transfer the 100 g sample of the riffled black to the 1,7 mm aperture sieve, install the top sieve cover (4.5) and transfer the assembly to the sieve shaker (4.2). 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 should be at rest.

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5.4 Take the 1,4 mm aperture sieve and pour about 45 pellets from it into one end of the shallow container (4.7). Slightly tilt and gently shake this container to cause the most spherical pellets to roll to the opposite end.

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5.5 Pour about 25 of the most spherical pellets into a position close to the test equipment.

6 Procedure

6.1 Select one pellet and, using a soft brush, position it on the base plate centrally under the pressure foot of the force-application device (4.1).

6.2 Carefully bring the pressure foot into contact with the pellet.

Care is important when testing soft pellets which tend to fracture prematurely due to impact on initial contact.

6.3 Apply a force at a constant rate of between 5 and 25 cN/s, preferably 15 cN/s, until the pellet fractures. Record the force required.

NOTE — Lower force rates tend to give more repeatable results.

6.4 Clean the base plate and pressure foot.

6.5 Repeat the operations in 6.1 to 6.4 until 20 pellets have been tested.

7 Expression of results

7.1 Calculate the average of the 20 values and report this value to the nearest centinewton.

7.2 Calculate other values (e.g. the average of the five highest results) and report them if required.

8 Precision

8.1 Coefficients of variation of up to 30 % have been found within 20 spherical pellets and within 20 non-spherical pellets.

8.2 Spherical pellets have given higher crushing strength values than non-spherical pellets. Ratios of up to 1,8 have been found.

9 Test report

The test report shall include the following information:

a) a reference to this Technical Report;

b) the complete identification of the sample;

c) the details of the apparatus used;

d) the rate of force application;

e) the average pellet crushing strength of 20 pellets, in centinewtons;

f) other crushing strength values, if required;

g) any deviations from this recommended procedure.

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