TECHNICAL REPORT

ISO/TR 16098

First edition
2012-09-01

Reclaimed rubbers and reclaimed crumb rubbers — Evaluation of dispersion in rubber mixes

Caoutchoucs régénérés et poudrettes de caoutchoucs régénérés — Évaluation de la dispersion dans les mélanges de caoutchouc

Reference number
ISO/TR 16098:2012(E)

COPYRIGHT PROTECTED DOCUMENT

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

Contents

Page

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.

In exceptional circumstances, when a technical committee has collected data of a different kind from that which is normally published as an International Standard ("state of the art", for example), it may decide by a simple majority vote of its participating members to publish a Technical Report. A Technical Report is entirely informative in nature and does not have to be reviewed until the data it provides are considered to be no longer valid or useful.

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/TR 16098 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*.

Reclaimed rubbers and reclaimed crumb rubbers — Evaluation of dispersion in rubber mixes

1 Scope

This Technical Report specifies standard materials, standard test formulations, equipment and processing methods for evaluating the dispersion of reclaimed rubbers and reclaimed crumb rubbers in rubber mixes.

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 2393, *Rubber test mixes — Preparation, mixing and vulcanization — Equipment and procedures*

ISO 23529, *Rubber — General procedures for preparing and conditioning test pieces for physical test methods*

3 Preparation of test mixes for the evaluation of reclaimed and crumb rubbers dispersion

3.1 General

The ability of reclaimed or crumb rubbers to disperse in a rubber compound is studied by light macroscopy. These materials shall be mixed in a white masterbatch using a mill mixing procedure. After vulcanization, a slice of a bar specimen shall be used to evaluate the dispersion.

3.2 Standard test formulation

3.2.1 White masterbatch

The masterbatch formulation for the evaluation of the dispersion is given in Table 1.

The materials used shall be national or international standard reference materials. If no standard reference materials are available the materials used shall be agreed between the interested parties.

Table 1 — Masterbatch formulation

Material	Parts by mass
Natural rubber TSR 3L	100,00
Stearic acid [a]	1,50
Zinc oxide [a]	3,00
TiO_2 anatase	105,00
Liquid paraffin	15,00
Total	224,50

[a] Powder materials shall be used (standard curing ingredients used in the industry).

3.2.2 Incorporation formulation for reclaimed rubber or rubber crumb

The incorporation formulation is given in Table 2.

The materials used shall be national or international standard reference materials. If no standard reference materials are available the materials used shall be agreed between the interested parties.

Table 2 — Incorporation formulation for evaluation of reclaimed rubber dispersion

Material	Parts by mass
White masterbatch	100,00
Reclaimed or crumb rubber	10,00 [c]
Sulfur[a]	2,50
TBBS[b]	1,00
Total	113,50

[a] Powder materials shall be used (standard curing ingredients used in the industry).

[b] N-tert-butyl-benzothiazole-2-sulfenamide. This is supplied in powder form having an initial insoluble-matter content, determined in accordance with ISO 11235, of less than 0,3 %. The material shall be stored at room temperature in a closed container and the insoluble matter shall be checked every 6 months. If this is found to exceed 0,75 %, the material shall be discarded or recrystallized.

[c] This crumb rubber loading of 10 parts was estimated to be sufficient to assess the dispersion level in the rubber matrix.

3.3 Procedure

3.3.1 Equipment and procedure

Equipment and procedure for the preparation, mixing and vulcanization shall be in accordance with ISO 2393.

3.3.2 Mixing procedure

3.3.2.1 White masterbatch – Internal mixing

		Duration (min)	Cumulative time (min)
a)	Adjust the temperature of the internal mixer to achieve a final mix temperature of 120 °C in about 5 min. Close the discharge door, set the rotor at 50 rpm, start the rotor and raise the ram.	0	0
b)	Load the rubber. Lower the ram and allow the natural rubber to be masticated.	1,0	1,0
c)	Raise the ram and add about 75 % of the TiO_2, taking care to avoid any loss. Then lower the ram and allow the batch to mix.	1,0	2,0
d)	Raise the ram and add the rest of TiO_2 and liquid paraffin, taking care to avoid any loss. Then lower the ram and allow the batch to mix.	1,0	3,0
e)	Raise the ram and add the zinc oxide and the stearic acid taking care to avoid any loss. Then lower the ram and allow the batch to mix.	1,0	4,0
f)	Raise the ram and clean the mixer throat and the top of the ram. Lower the ram and allow the batch to mix.	1,0	5,0
	Total time (max.)	**5,0**	

g) Turn off the rotor, raise the ram and discharge the batch. Record the maximum batch temperature.

h) Immediately pass the batch four times through a laboratory mill with its mill opening set at 2,5 mm and at a temperature of 50 °C ± 5 °C. Check-weigh the batch (see ISO 2393). If the mass of the batch differs from the theoretical value by more than + 0,5 % or - 1,5 %, discard the batch and remix.

i) Leave the batch for 30 min to 24 h after mixing, if possible at standard temperature and humidity as defined in ISO 23529.

3.3.2.2 Incorporation – Mill mixing

The standard laboratory mill batch mass, in grams, shall be based on twice the formulation mass. The surface temperature of the rolls shall be maintained at 50 °C ± 5 °C throughout the mixing.

A good rolling bank at the nip of the rolls shall be maintained during mixing. If this is not obtained with the nip settings specified below, small adjustments to the mill openings may be necessary.

		Duration (min)	Cumulative time (min)
a)	Band the white masterbatch on the roll with the mill set at 50 °C ± 5 °C and at a 1,2 mm opening.	2,0	2,0
b)	Add the reclaimed rubber. Do not cut the band until it is completely incorporated.	0,5	2,5
c)	Make a 3/4 cut from each side, allowing 15 s between each cut.	0,5	3,0
d)	Add the TBBS and the sulfur evenly across the rolls, still set at an opening of 1,2 mm.	2,0	5,0
e)	Make three 3/4 cuts from each side, allowing 15 s between each cut.	2,0	7,0
f)	Cut the batch from the mill. Set the mill opening at 1,2 mm and pass the rolled batch endwise through the rolls six times, introducing it from each end alternately.	2,0	9,0
	Total time (max.)	**9,0**	

g) Sheet the batch to approximately 10 mm. Check-weigh the batch (see ISO 2393). If the mass of the batch differs from the theoretical value by more than + 0,5 % or - 1,5 %, discard the batch and remix.

h) Prepare blanks for ISO bar test pieces (see 4.3).

i) After mixing and prior to vulcanization, condition the batch for at least 2 h but not more than 24 h, if possible at standard laboratory temperature and humidity as defined in ISO 23529.

3.3.3 Vulcanization procedure

Vulcanize test pieces (see 4.3) at 150 °C ± 5 °C for 15 min.

4 Measurement of the dispersion of reclaimed rubbers and crumbs rubbers

4.1 General

This method is a testing method which determines the surface fraction (i.e. the fraction of surface occupied by reclaimed rubber or crumbs) and the various sizes of reclaimed rubbers or crumb rubbers particles in a white masterbatch by using a light macroscope and a numerical camera.

4.2 Apparatus

4.2.1 Razor blade, with only one edge, set in a suitable device.

4.2.2 Cut-out device, with a mechanism of levers in order to make a vertical cut and a clamp for the razor blade.

4.2.3 Optical macroscope, equipped with a numerical camera.

4.2.4 Lamp for the macroscope, giving a strong light intensity.

NOTE The observations are made with an indirect lighting and an angle of approximately 60°.

4.3 Test piece

By using the cut-out device, cut a test piece from the ISO bar having a cross-section of approximately 8 mm thickness and 10 mm width. The surface must be as flat as possible. Do not touch the surface which is used for measurement. The blade shall be replaced before each new cut, to avoid stripes on the cut.

4.4 Test procedure

Arrange the sample on the macroscope sample holder.

Adjust the diaphragm of the light macroscope and the acquisition time of the numerical camera to enhance the contrast.

Adjust the angle of the lamp illuminating the sample surface, in order to decrease the intensity of the bright points on the sample surface.

Use a magnification, G = x20, in order to see enough objects on the photograph, observe the various classes of particles and underline their distribution in the masterbatch.

Take enough photographs on different zones of the sample for the image processing.

NOTE 10 photographs are enough for dispersed particles.

4.5 Image processing

Use commercial image processing software.

NOTE For example AnalySis software.

Convert the colour image (24 bits) to an image in levels of gray (8 bits) and reverse the contrast.

Use some mathematical filters to increase the contrast of fillers and enhance the image (noise reduction smoothing).

Convert this to a black and white image (binary contrast image).

4.6 Measurement of Equivalent Circular Diameter (ECD)

Equivalent Circular Diameter (ECD) is the diameter of a circle with area equal to that of the projection of the particle at a plane; relevant for imaging methods of particle characterization used in microscopy. The ECD is also referred to as the Heywood diameter.

Due to the image magnification, the measurement of the ECD does not include the particles of size lower than 5 µm.

Calculate a number average ECD from all the individual particle ECDs. A particle size distribution curve can also be relevant.

4.7 Measurement of surface fraction

Surface fraction is the percentage of the surface occupied by particles. The value is determined for each photograph by image analysis on the binary image.

With all surface fractions, a number average surface fraction is calculated for each sample.

5 Test report

The test report shall include the following:

a) a reference to this International Standard;

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

c) the reference materials used to prepare the test mix;

d) the ambient conditions in the laboratory during preparation of the test mix;

e) any unusual features noted during the determination;

f) details of any operation not included in this International Standard or in the International Standard to which reference is made, as well as of any operation regarded as optional;

g) the results and the units in which they have been expressed;

h) the date of the test.

Bibliography

[1] ISO 11235, *Rubber compounding ingredients — Sulfenamide accelerators — Test methods*

ICS 83.040.20

Price based on 7 pages