DRAFT FOR DEVELOPMENT

DD CEN/TS 14243:2010

Materials produced from end of life tyres — Specification of categories based on their dimension(s) and impurities and methods for determining their dimension(s) and impurities

ICS 13.030.50; 83.160.01

BSI British Standards

National foreword

This Draft for Development is the UK implementation of CEN/TS 14243:2010.

This publication is not to be regarded as a British Standard.

It is being issued in the Draft for Development series of publications and is of a provisional nature. It should be applied on this provisional basis, so that information and experience of its practical application can be obtained.

Comments arising from the use of this Draft for Development are requested so that UK experience can be reported to the international organization responsible for its conversion to an international standard. A review of this publication will be initiated not later than 3 years after its publication by the international organization so that a decision can be taken on its status. Notification of the start of the review period will be made in an announcement in the appropriate issue of Update Standards.

According to the replies received by the end of the review period, the responsible BSI Committee will decide whether to support the conversion into an international Standard, to extend the life of the Technical Specification or to withdraw it. Comments should be sent to the Secretary of the responsible BSI Technical Committee at British Standards House, 389 Chiswick High Road, London W4 4AL.

The UK participation in its preparation was entrusted to Technical Committee PRI/73, Industrial rubber products.

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.

Compliance with a British Standard cannot confer immunity from legal obligations.

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English Version

Materials produced from end of life tyres - Specification of categories based on their dimension(s) and impurities and methods for determining their dimension(s) and impurities

Matériaux produits à partir de pneumatiques en fin de vie - Spécification de catégories basées sur leur(s) dimension(s) et impuretés et méthodes pour déterminer leur(s) dimension(s) et impuretés

Materialen aus Altreifen - Festlegung von Klassen anhand ihrer Abmessung(en) und Verunreinigungen und Verfahren zur Bestimmung ihrer Abmessung(en) und Verunreinigungen

This Technical Specification (CEN/TS) was approved by CEN on 4 January 2010 for provisional application.

The period of validity of this CEN/TS is limited initially to three years. After two years the members of CEN will be requested to submit their comments, particularly on the question whether the CEN/TS can be converted into a European Standard.

CEN members are required to announce the existence of this CEN/TS in the same way as for an EN and to make the CEN/TS available promptly at national level in an appropriate form. It is permissible to keep conflicting national standards in force (in parallel to the CEN/TS) until the final decision about the possible conversion of the CEN/TS into an EN is reached.

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Foreword

This document (CEN/TS 14243:2010) has been prepared by Technical Committee CEN/TC 366 “Project Committee - Tyre recycling”, the secretariat of which is held by UNI.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This document supersedes CEN/CWA 14243:2002.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to announce this Technical Specification: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and the United Kingdom.

Introduction

This Technical Specification is the first in a foreseen series of Technical Specifications. Such series would cover the testing programmes needed to characterise each product category as shown on the figure below.

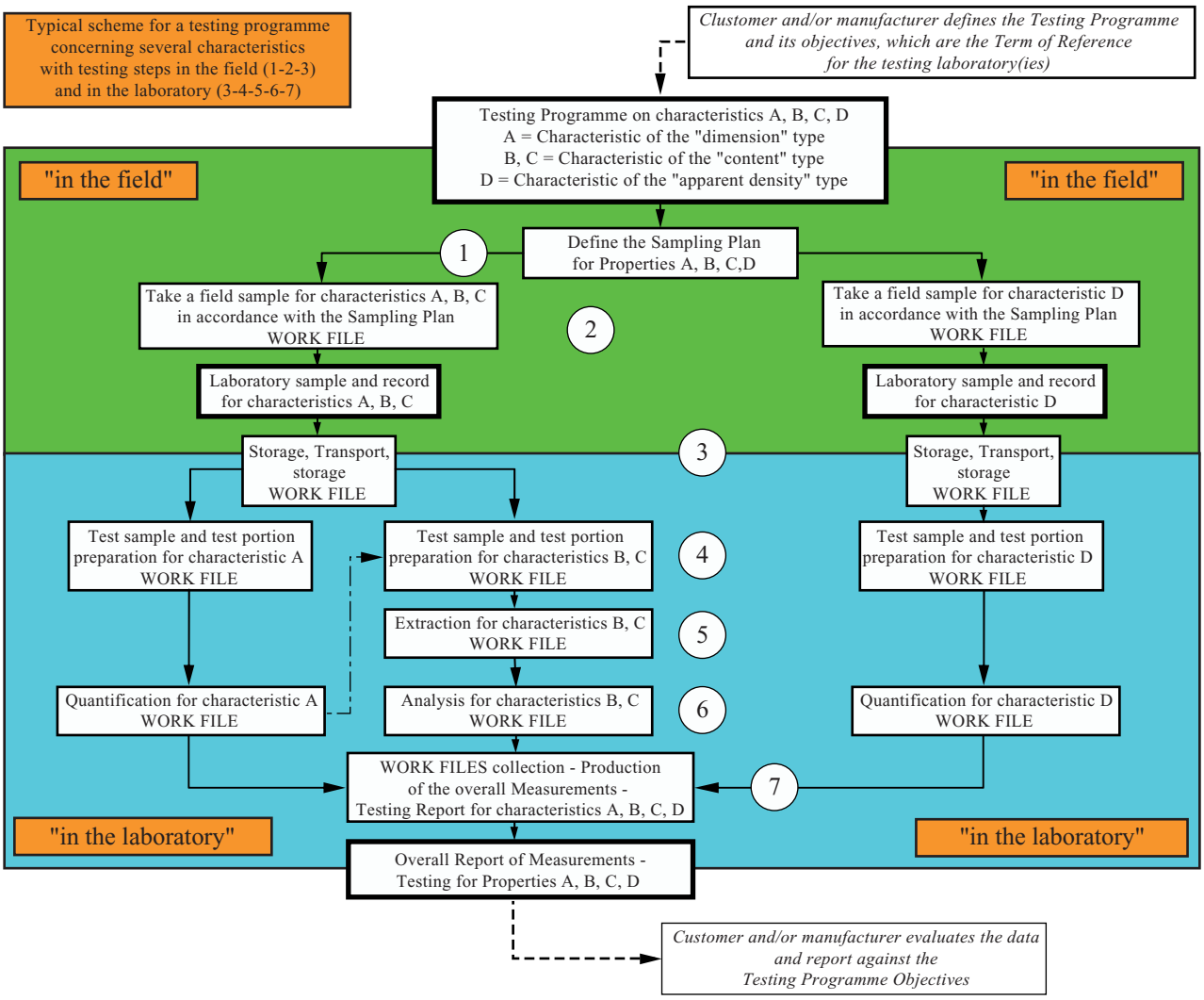

Figure 1 — Typical scheme for a testing programme concerning several characteristics with testing steps in the field and in the laboratory

End-of-life tyres consist mainly of passenger and commercial vehicle tyres, and truck tyres manufactured for distribution in the European market. Products from end-of-life tyres are used as a secondary raw material finding a wide range of applications. The principal categories of materials from end-of-life tyres are defined on the basis of their dimension(s) according to this Technical Specification. Determination of additional dimensions/impurities according to the test methods specified in normative Annexes A, B, C and D may be performed if required by agreement between the producer and the customer or at the producer's own will.

European Standards are needed for the production, trade and use of the materials from end-of-life tyres. They are also useful for buyers of materials, regulators, controllers and laboratories.

The materials produced at different stages of the treatment processes, primarily by size reduction, of end-of-life tyres are as follows:

— cuts: typically 300 mm and above;

— shreds: typically 20 mm – 400 mm;

— chips: typically 10 mm – 50 mm;

— granulates: typically 0,8 mm – 20 mm;

— powders: typically under 0,8 mm;

— steel;

— textiles.

They may be distinguished mainly according to their dimension(s).

1 Scope

This Technical Specification provides definitions for the categories of materials produced from end-of-life tyres based on their dimension(s) or impurities. It also provides test methods for the determination of the dimension(s) of the materials produced from all categories of end-of-life tyres at all steps of the treatment process as well as for the determination of impurities.

The test methods described in this Technical Specification include sample collection and the preparation of a representative sample based on a sampling plan for the purpose of determining dimensions and impurities.

This Technical Specification does not cover the operational performance or fitness for use of the materials which are deemed to be a function of agreement between the producer and the customer.

NOTE This Technical Specification does not cover downstream products prepared from the materials, including reclaimed or devulcanized rubber, surface-modified powders and pyrolytic output.

This Technical Specification does not purport to address safety concerns associated with its use. It is the responsibility of the user of this Technical Specification to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

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.

EN 933-1, *Tests for geometrical properties of aggregates — Part 1: Determination of particle size distribution — Sieving method*

EN 933-2, *Tests for geometrical properties of aggregates — Part 2: Determination of particle size distribution — Test sieves, nominal size of apertures*

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

ISO 3310-1:2000, *Test sieves — Technical requirements and testing — Part 1: Test sieves of metal wire cloth*

ISO 3310-2:1999, *Test sieves — Technical requirements and testing — Part 2: Test sieves of perforated metal plate*

3 Terms and definitions

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

3.1
sample
amount of material taken from a population and intended to provide information on the population

3.2
sub-sample
portion of a sample

3.3
increment
portion of material extracted in a single operation of the sampling device

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[Adapted from ISO 13909:2001 (all parts)]

3.4
characteristic
property which helps to identify or differentiate items of a given population

[ISO 3534-1:2006]

NOTE The characteristic may be either quantitative (by variables) or qualitative (by attributes).

3.5
lot
defined quantity of material for which a characteristic is to be determined

NOTE In sampling standards the lot is also designated as the "scale" as the stated size or volume that is considered appropriate for assessing the material. It follows that variations occurring in the material on any finer scale than this are deemed not to be of relevance.

3.6
combined sample
sample consisting of all the increments taken from a lot

NOTE A combined sample is a quantity of material, representative of the lot for which the quality is to be determined.

3.7
field sample
sample taken in the field and from which laboratory samples are produced

3.8
laboratory sample
sample or sub-sample sent to or received by the laboratory

[IUPAC definition [1]]

NOTE 1 When the laboratory sample has been prepared (reduced) by subdivision, mixing, or crushing, or by a combination of these processes, it becomes the test sample. A laboratory sample that requires no preparation can be used directly as the test sample. A test portion is removed from the test sample for testing or analysis purposes. The laboratory sample is the final sample from the point of view of sample collection, but it is the initial sample from the point of view of the laboratory.

NOTE 2 Several laboratory samples can be prepared and sent to different laboratories, or they can be sent to the same laboratory for different purposes. In the latter case, they are generally considered to be a single laboratory sample and documented as such.

3.9
test sample
sample prepared from the laboratory sample, from which the test portions are removed for testing or for analysis

[IUPAC definition]

3.10
test portion
quantity or volume removed from the test sample for analysis purposes, generally of known weight or volume

[IUPAC definition[1]]

3.11
population
totality of items, or total volume of material, to be investigated by sampling

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NOTE The population will generally be a convenient, well-defined subset of the overall population (e.g. a year's production of material) that is believed to be typical of that wider population.

3.12
representative sample
sample in which the characteristic(s) of interest is (are) present with a reliability appropriate for the purposes of the testing programme

3.13
representative
sample resulting from a sampling plan that can be expected to reflect adequately the properties of interest in the parent population

[ISO 11074:2005]

3.14
probabilistic sampling
sampling conducted according to the statistical principles of sampling

3.15
judgemental sampling
sampling undertaken from a practically convenient (perhaps relatively small) sub-population, not conducted fully in accordance with the statistical principles of sampling

3.16
sample division
reduction of the mass of a sample or sub-sample

3.17
nominal top size
aperture size of the sieve used in the determination of the particle size distribution through which at least 95 % by mass of the material passes

[Adapted from ISO 13909:2001 (all parts)]

3.18
cuts
result of mechanical processes by which end-of-life tyres are fragmented, ripped or torn into irregularly formed pieces typically larger than 300 mm in size

3.19
shreds
result of mechanical processes by which end-of-life tyres are fragmented, ripped or torn into irregular pieces of typically 20 mm to 400 mm in any dimension

3.20
format
<of shreds> format specified in this Technical Specification based on the distribution of the maximum projected length of shreds produced from end-of-life tyres

3.21
chips
result of mechanical processes by which end-of-life tyres are fragmented, ripped or torn into irregularly shaped pieces of typically 10 mm to 50 mm in size

3.22
granulates
result of processing rubber to reduce it in size into finely dispersed particles between typically 0,8 mm and 20 mm

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3.23
powder
result of processing rubber to reduce it in size to achieve finely dispersed particles of typically under 0,8 mm

3.24
steel
result of processing end-of-life tyres by which steel wires are separated from textile and rubber fractions

3.25
textiles
result of processing end-of-life tyres by which textile fibres are separated from steel and rubber fractions

3.26
filaments
filiform metallic and/or textile parts protruding from pieces of shreds, cuts and chips considered as very small shreds

3.27
free steel
fraction of the steel not embedded in granulates or powders or chips considered as large granulates which can be separated with a magnetic process

3.28
free textile
fraction of the textile content in granulates or powders or chips considered as large granulates which can be separated as small ball during sieving

4 Categories of products, based mainly on their dimensions, produced from end-of-life tyres

4.1 Categories

The materials produced at different stages of the treatment processes primarily by size reduction of end-of-life tyres may be distinguished mainly according to their dimension(s):

— cuts: typically 300 mm and above;

— shreds: typically 20 mm – 400 mm;

— chips: typically 10 mm – 50 mm;

— granulates: typically 0,8 mm – 20 mm;

— powders: typically under 0,8 mm;

— steel;

— textiles.

The actual range of dimensions (including lower and higher dimensions and associated percentage) is to be determined in the contract between producer and customer, according to the measurement method specified in this Technical Specification.

The dimensions of cuts, shreds, chips, granulates and powders, as well as the impurities content of other materials, i.e. steel and textile, are to be measured according to the test methods specified in this Technical Specification. Determination of additional dimensions/impurities according to the test methods specified in

normative Annexes A, B, C and D may be performed if required by agreement between the producer and the customer or at the producer's own will.

NOTE The term dimension(s) is used since several materials cannot be considered as a collection of spherical particles characterised by a diameter but are rather characterised by several dimensions.

4.2 Testing programme

When performing a testing programme for determining product dimensions, all the different measurement/testing steps are to be considered and specified by means of standards dealing each with one or several of those steps, thus securing the needed coherence-coordination between these different testing steps. The different steps are:

— sampling plan;

— taking field sample(s) to obtain laboratory sample(s);

— storage, transport, storage of the sample(s);

— preparation of test portion(s) from the laboratory sample(s);

— pre-treatment, e.g. drying (if needed);

— quantification, analysis, calculations;

— overall test report.

When undertaking some or all of these measurement steps, a testing laboratory shall operate with appropriate equipment and competent personnel so as to fulfil the applicable requirements specified in the present Technical Specification. This includes calibration of equipment, e.g. of scales.

5 Determination of dimensions for granulates and powders and for chips considered as very large granulates

5.1 General

Particle size and related parameters are key measurements for product classification of size-reduced materials such as granulates, powders and chips considered as very large granulates. Particle size analysis is the first measurement of consistency and forms the basis for material grading. Before being able to obtain an accurate particle size distribution, a representative sample of the material to be tested must be taken.

Depending on the agreed test programme between the producer and the customer, or on the producer's own will, the determination of dimensions specified in this clause for granulates and powders and for chips considered as very large granulates may be followed by the additional determination(s) of the free steel content and/or the free textile content and/or the other impurities as specified in normative Annexes B, C and D.

5.2 Preparation of the sampling plan

5.2.1 Principles of correct sampling

The main principle of sampling is to obtain a representative sample(s) from a lot of material from which a characteristic is to be determined. If the lot is to be represented by a sample then every particle in the lot must have an equal probability of being included in the sample. When these principles cannot be applied in practice, the sampler shall note the limitations in the sampling plan and sampling report.

5.2.2 General

In general, it is difficult to take samples in a way that ensues truly representative sampling when material is stationary (for example in a stockpile, big bag or silo). It is easier when the material is moving (for example on a conveyor belt). Therefore, sampling from moving material is to be preferred wherever possible.

NOTE The determination of other properties than dimension may result in different sampling requirements. This is the case for the determination of physical properties such as apparent density or chemical composition.

5.2.3 Sampling plan

A written sampling plan shall be prepared before samples are taken according to 5.2.4 to 5.2.7. The number of increments shall be not less than the minimum number of increments specified in 5.2.6.

A form for the sampling plan shall be prepared containing the following minimum information:

— the name of the producer;

— a unique identification number of the sample;

— the name of the sampler;

— the location(s), date and time of sampling;

— the nominal top size;

— the lot identification number which is to be tested (based on 5.2.3);

— reference to this Technical Specification;

— any deviation from this Technical Specification.

Once completed, this form becomes the sampling certificate.

5.2.4 Definition of lot size

The lot size shall be defined by the producer in accordance with requested specifications and is a fixed quantity for which a characteristic is to be determined. The lot size (M_{lot}) may be defined by the producer as:

— a fixed quantity produced between machine settings;

— a fixed quantity in a production day/shift;

— a fixed quantity minimum of 100 t (10 t for powders).

The lot size is based on production quality management decisions or specific customer requirements.

5.2.5 Sampling point and apparatus

Based on health and safety assessments and producer equipment, a fixed sampling point for the collection of sample increments shall be chosen for each material fraction to be monitored. Sampling shall be carried out using a sample box or other suitable equipment. The sampling box is passed through the stream of falling material so that it uniformly cuts the full flow of falling material. The box shall be large enough so that it does not become overloaded. Automatic systems fulfilling these criteria may also be used.

5.2.6 Size of a sample increment

The sampling box shall have a capacity of not less than:

V_{min} = 0,5 for d < 10;

V_{min} = 0,05 × d for 10 ≤ d ≤ 20;

V_{min} = 5 dm^3 (chips considered as very large granulates)

where

V_{min} in cubic decimetres is the minimum capacity of the sampling box; and

d is the nominal top size in millimetres.

The sampler shall record the approximate capacity of the sampling device $V_{increment}$ in cubic decimetres.

5.2.7 Number of increments

The minimum number of increments to be taken from a lot depends on the nominal top size of the granulate material to be sampled. The granulate shall be assigned by the sampler to one of two groups in Table 1.

Table 1 — Classification of material according to size

Group 1	Group 2
nominal top size < 10 mm	nominal top size is between 10 mm and 20 mm

For sampling from moving material:

Group 1: $n = 3 + 0{,}025 \times M_{lot}$ size of granulates from 0,8 mm to 10 mm and powders under 0,8 mm

Group 2: $n = 5 + 0{,}040 \times M_{lot}$ size of granulates from 10 mm to 20 mm and chips considered as very large granulates

where

n is the minimum permitted number of increments rounded off to the higher nearest whole number;

M_{lot} is the mass of the lot in tonnes.

Sample collection is carried out according to the sample plan and the increments are collected manually using the sampling box. Each increment is placed in a separate container and sent to the testing laboratory. Increments shall be taken at regular intervals during the discharge of the lot. The time of increment collection is recorded in the sampling plan.

5.2.8 Metrological characteristics of sampling

When this Technical Specification was adopted by CEN, the test specified in this clause had not been validated and no data on robustness, repeatability and reproducibility was available.

5.2.9 Visual assessment

The increments are to be assessed visually and observations on the quality should be noted as additional information and recorded according to quality management, best practice policies or specific customer requirements.

5.3 Storage and transport of sample(s)

Samples are to be stored in such a way that material will not be lost during transportation. The samples are to be stored dry and in a sealed sample box. The sampling certificate is to be attached.

5.4 Preparation of the laboratory sample(s) and test portion(s)

5.4.1 Principles of correct sample preparation and sample division

The main purpose of sample preparation is that the increment samples collected from the whole lot are reduced to one or more test samples that are in general smaller than the original sample based on sample division. The aim of sample division is therefore to reduce the mass of the sub-samples or to make several duplicate sub-samples out of the original sample.

Each test sample shall be a representative of the original sample based on the principle that the composition of the sample as taken on site shall not be changed during the steps of sample preparation for testing.

5.4.2 Test portion preparation

The increments that have been collected are mixed together in a container of suitable size to form a combined sample. In this case the combined sample is the laboratory sample.

As regards the method of particle size analysis to determine the particle size distribution (dimensions), the combined sample shall be reduced to a suitable size such that sufficient material is still provided for all the tests to be performed. In particular the calculation shall take into account the need for duplicate test portions, for extra material in case dubious results are obtained and the need for sampling with replacement. The combined sample shall be reduced using the principles of sample division using either:

— a riffle splitter;

— a rotary sample divider; or

— other equivalent device.

5.4.3 Apparatus for sample division

a) Riffle Splitter

A sample splitter shall have at least 16 slots and an even number with adjacent slots directing material into two different sub-samples. The width of the slots shall be at least three times the nominal top size of the material to be riffled to prevent bridging.

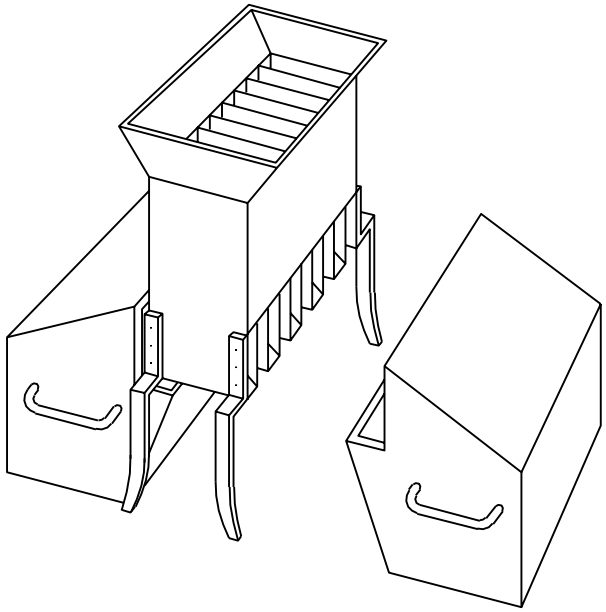

Figure 2 — Example of a riffle box

b) Rotary Sample Divider

A rotary sample divider shall be used according to its manufacturer's manual. The inner dimensions of the equipment where the sample is fed shall be at least three times as wide as the nominal top size to be processed.

5.4.4 Procedure

The combined sample is mixed with a scoop and sent through the sample divider until the sample reduction is complete. A final mass quantity of at least 150 g is used for the purposes of the particle size analysis depending on the size of the sieving system in order to fulfil the non-overloading criteria given in 5.5.6.

5.5 Particle size analysis

5.5.1 General principles

A sample is subjected to mechanical sieving through two or three dimensional oscillating sieves, sorting the particles in decreasing size classes by mechanical means. All apparatus shall conform to the general requirements of EN 933-1, EN 933-2, ISO 565, ISO 3310-1 and ISO 3310-2. The sample shall be completely dry before particle size analysis.

NOTE In case of moisture, drying should be performed by putting the sample inside an oven of circulating air at a temperature of (110 ± 2) °C. After 30 min the sample is taken out of the oven and the sample is given time to reach room temperature. The particle size analysis should not be performed before 30 min after the extraction of the sample from the oven.

5.5.2 Sieves

For the sieve analysis, an appropriate number of sieves is required and shall be chosen according to the size specification of the sample to be tested. The geometry of the apertures shall be either circular or square, the thickness of the sieves, the distances of the holes and the diameter of the holes shall be in accordance with

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ISO 3310-1 and or ISO 3310-2. The frame of the sieves shall have a height that enables the sieves to contain the samples and allow for the free movement of the sample during the sieving process.

The number of sieves that are used may vary according to the particle size distribution that is to be determined. However, the supplementary row R 20 from ISO 3310-1:2000 and/or ISO 3310-2:1999 is to be preferred. It is not permitted to jump between principal and supplementary rows within a series.

5.5.3 Flat brush

For cleaning the sieves, a flat brush is required.

5.5.4 Mechanical vibrating equipment

A mechanical device that applies a vibrating movement in at least two dimensions is to be used.

NOTE Equipment may include a test sieve shaker which has a timer and amplitude settings.

5.5.5 Scales

Scales capable of measuring the mass of the sample to be sieved to the nearest 0,1 g.

5.5.6 Procedure

Assemble and operate the mechanical shaking device with the appropriate sieve sizes with decreasing aperture size ending with the collecting pan. The amount of sample depends primarily on the maximum particle size, the number of sieves in the sieve stack and their openings. The sieves shall not be overloaded. Criteria for non overloading are given in Annex G Sieving.

NOTE 1 For rubber granulate, practice has shown that different sizes of sieving system fulfil these requirements with a sample quantity of at least 150 g.

Before sieving starts the mass of all individual sieves should be checked on the scales used for determination of sieving fractions.

Weigh the sample to the nearest 0,1 g and spread the test sample in an even layer on the top sieve. Using a brush, remove all traces from the sample box. Secure the cover plate (lid) and begin the sieving operation.

The duration of the screening operation is fixed at:

— 10 min ± 15 s for granulates;

— 10 min ± 15 s for chips considered as very large granulates;

— 10 min ± 15 s for powders.

Weigh the retained net material on each sieve and in the collecting pan with an accuracy of 0,1 g and record the masses as expressed in 5.5.7.

NOTE 2 Avoid the loss of particles from the screens. If a particle sticks in a sieving hole, it should be removed and added to the fraction which remained on the sieve (as it did not pass the hole).

5.5.7 Calculation

Record the various masses on a test data sheet alongside the corresponding sieve size. If there is more than a 1 % difference between the total mass of the test portions M_1 and the total mass of all the fractions in percent then the test shall be repeated.

Calculate the mass of material retained on each sieve as a percentage of M_1.

Calculate the cumulative percentage of M_1 passing each sieve, to the nearest decimal point.

Plot the cumulative percentage as a function of particle size using the sieve size, expressed in millimetres for sizes of 1 mm and above and in microns for sizes under 1 mm, unless otherwise specified.

From the plot, determine the particle size at which the passing cumulative percentage is 90 % and take this as the upper limit of the dimension of the granulate fraction. Likewise, determine the particle size at which the passing cumulative percentage is 10 % and take this as the lower limit. The two limits are a measure of the particle size range of the material.

In compliance testing, more than 90 % by mass of the material shall be smaller in size than the upper defined limit and less than 10 % by mass of the material in size shall not be smaller than the lower defined limit.

5.6 Metrological characteristics

When this Technical Specification was adopted by CEN, the test specified in this clause had not been validated and no data on robustness, repeatability and reproducibility was available.

5.7 Test Report

The test report for the particle size distribution shall include at least the following information:

— identification of the testing laboratory and the testing date;

— identification of the company having requested the test;

— reference to this Technical Specification;

— identification of the product with reference to the lot;

— the mass of the sample;

— type of sieving machine and settings used (when available time frequency and amplitude);

— information on which sieves were used (ISO 3310-1 or ISO 3310-2);

— conditions and observations, e.g. visual quality, free of contamination;

— test results expressed as upper and lower limits of the dimensions (see 5.5.7) and graphical representation;

— any deviations from this Technical Specification.

6 Determination of dimensions for shreds and cuts and for chips considered as very small shreds

6.1 Definition of shreds

In Clause 6 "shreds" are material produced from end-of-life tyres by primary shredding. Primary shredding means the processing of end-of-life tyres by shredding, crushing or fragmenting while maintaining in the obtained shreds an average global composition similar to that of the end-of-life tyres.

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Depending on the agreed test programme between the producer and the customer, or at the producer's own will, the determination of dimensions specified in this clause for shreds and cuts and for chips considered as very small shreds may be followed by the additional determination of filaments as specified in normative Annex A.

6.2 Principles of sampling

The main principle of sampling is to obtain a representative sample(s) from a whole lot (of defined material) from which a characteristic is to be determined. If the lot is to be represented by a sample then every particle in the lot must have an equal probability of being included in the sample (i.e. probabilistic sampling). When these principles cannot be applied in practice, the sampler shall define a procedure as close as possible to probabilistic sampling in his judgement (i.e. judgemental sampling) and note the limitations in the sampling plan and sampling report.

In general, it is difficult to take samples in a way that ensues truly representative sampling when a material is stationary (for example in a stockpile, big bag or silo). Regarding shreds, it is necessary to take samples when the material is in movement.

NOTE The determination of other properties than dimensions may result in different sampling requirements. This is the case for the determination of physical properties such as apparent density or chemical composition.

6.3 Preparation of the sampling plan for determining dimensions

The first step is to identify the properties required in the testing programme and the lot in relation to which they are defined, for instance "maximum projected length on a shreds production of 300 t".

The lot size is based on production quality management decisions or specific customer requirements. The lot size may be defined by the producer as a fixed quantity produced between machine settings or a fixed quantity in a production day/shift/week or a fixed quantity. With regards to certain pieces exhibiting large dimensions, the tools for taking an increment shall be sufficiently large in order that the large pieces are equally sampled. This results typically for shreds in increments of more than 100 pieces (3 kg to 15 kg) (50 pieces for cuts).

NOTE 1 Larger increments would slightly improve the sampling quality, while increasing the size of the field sample, therefore complicating the size reduction into laboratory sample(s). It is preferable to increase the number of increments, thus increasing directly the representativeness.

For reference testing (contractual or pre-contractual) concerning shreds dimension(s) the field sample is a composite sample constituted of three increments taken at dates selected randomly along the period of time during which the lot is produced. When at one date there are no shreds at the sampling location, the increment is taken at another date preselected prior to sampling, unless this is caused by a major change in the production process (see below).

For routine testing concerning the dimension(s) of shreds the field sample consists of one increment taken at a date selected randomly along the period of time during which the lot is produced. When at this date there are no shreds at the sampling location, the increment is taken at another date preselected prior to sampling, unless this is caused by a major change in the production process (see below).

During a sampling step the production process may be interrupted by a major change. Such a major change would induce in the sample two different subpopulations, before and after the major change. Therefore it is necessary to consider a lot before and a lot after the major change. Major changes may occur on the feed and on the production process.

NOTE 2 The random taking of three increments allows having a first approximation of the variability inside the considered lot. The random taking of one increment in routine testing allows also another evaluation of the variability when considering the lots obtained under comparable conditions.

6.4 Procedure for taking the field sample and producing the laboratory sample(s)

The taking of the increment(s) is done at the output of the conveyer in the falling zone. The used tool is typically a rectangular open scoop, i.e. without an edge on one of the long side so as to be capable of "cutting" the entire flow. Such a scoop (see Annex F) shall have:

— a width *L* at least 1,5 times the width of the falling flow;

— a depth *P* at least 2/3 of the width *L* and at least 2,5 times the higher dimension HDF;

— an edge height H equal at least to 1/3 of the width *L* and at least two times the higher dimension HDF.

This scoop is moved for instance with a loader according to a detailed procedure adapted to the site condition. This scoop can be the bucket of a loader provided that its cleanliness is compatible with the requirement of the dimension measurement, i.e. the absence of deposit. The taken increment is considered as valid if both criteria are fulfilled:

— the increment consists of at least 100 pieces (after sieving of fines) (50 pieces for cuts);

— the mass of the increment does not exceed 15 kg for shreds.

NOTE The minimum size of the increment is specified as a minimum number of pieces in view of the statistical evaluation of the measurements of the maximum projected length of each piece. A specification in mass would result in significant differences depending on the size of the pieces.

For the determination of dimension(s), each increment constitutes a laboratory sample for such determination. If there are several increments, the dimension of each increment is determined and the average is calculated. This is easier than to reduce the size of the field sample prior to measurement of the dimension.

6.5 List of symbols and abbreviations

This subclause lists the symbols and abbreviations used in other subclauses of Clause 6 dedicated to shreds:

— LDF Lower Dimension of the Format (millimetres);

— HDF Higher Dimension of the Format (millimetres);

— *L* Maximum projected Length (millimetres);

— MS Mass of the laboratory Sample (kilograms);

— MF Mass of the Fine pieces (kilograms);

— MLM Mass of the Loose Metal wires (kilograms);

— NCC Number of Central Classes;

— NCR Number of Classes in the Range LDF — HDF;

— TNP Total Number of Pieces in the sample not including the fine pieces;

— MPF Mass Percentage of the Fine pieces;

— MPM Mass Percentage of the loose Metal wires;

— NPL Number Percentage of Large pieces;

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— MPL Mass Percentage of Large pieces (optional);

— NPC Number Percentage of the NCC Central Classes;

— MPC Mass Percentage of the NCC Central Classes (optional).

6.6 Principle of determining the dimension(s)

A laboratory sample of at least TNP > 100 (50 for cuts) that do not pass through the LDF mm sieve is taken for the test. The MS mass (in kilograms) of this laboratory sample is weighed to within ± 0,01 kg. Any elements consisting solely of metal wires released from the pieces of end-of-life tyre are not counted in the TNF pieces. They are collected and weighed together (MLM kg).

After passing through a LDF mm sieve, the mass (in kilograms) of the fine pieces (MF) is weighed to within ± 0,01 kg. The pieces that do not pass through the sieve (without loose metal wires) are used to determine the maximum lengths and constitute the test portion for determination purposes.

Each piece of this test portion is treated individually. As these pieces are not usually flat, the largest length is defined as the largest length projected onto a plane on which the piece in question lies. This length is measured to within ± 5 mm, without deforming the piece and excluding filaments.

The measurements of the different maximum projected lengths *L* are used to build a histogram that is characteristic of the distribution of the pieces of the test portion, i.e. the laboratory sample without the fine pieces and without the loose metal wires. This histogram consists of the large pieces class (larger than the HDF threshold dimension of the large pieces) and NCR = 7 classes of the same width between the LDF and HDF dimensions.

Three characteristics of the histogram are extracted from these measurements:

a) the number percentage of large pieces (NPL) (%) (and optionally, the mass percentage of large pieces (MPL) (%)) corresponding to the pieces larger than the higher dimension of the HDF format (HDF is one of the characteristics of the format of the product under consideration, for example 350 mm);

b) the mass percentage of the fine pieces (MPF) (%) = 100 × MF/MS (mass percentage of the pieces passing through the sieve with a mesh of LDF mm) (LDF is one of the characteristics of the product format under consideration, for example 25 mm);

the number percentage (NPC) (%) (and optionally the mass percentage (MPC) (%)) of the pieces in the number of NCC central classes (2-3-4-5-6) amongst the NCR = 7 classes between the lower and higher dimensions of the format (LDF and HDF).

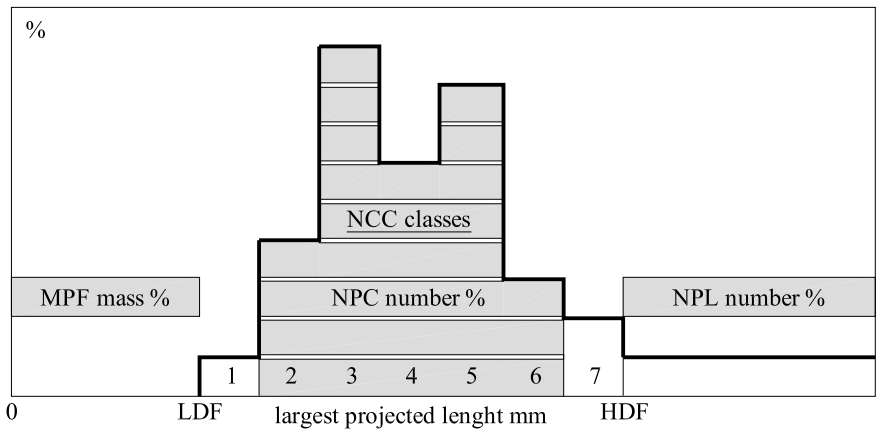

Figure 3 — Example of a histogram

6.7 Equipment

— Vessel large enough to contain at least 100 pieces (50 for cuts);

— illuminated measurement plane with a ruler graduated in mm that can measure lengths up to 500 mm (1 000 mm for cuts) on this measurement plane;

— scales to within 0,01 kg;

— circular mesh sieve in accordance with ISO 565 or ISO 3310-1 with a mesh of LDF (in millimetres).

6.8 Procedure for quantification of maximum projected length (manual)

— Identify the LDF and HDF dimensions in the test specifications and calculate the limits of the seven classes of the same width. Deduce the limits of the central range defined by NCC classes 2-3-4-5-6;

NOTE 1 HDF and LDF should be different enough in order to define in practice the seven classes.

— take a laboratory sample of the shredded material, in accordance with the sampling scheme (a vessel can be used to check that for the site in question, the TNP criterion > 100 elements after sieving at LDF mm is satisfied);

— weigh the laboratory sample (MS) (in kilograms);

— sieve the laboratory sample at LDF mm;

NOTE 2 To make the sieving easier, the coarsest fractions can be removed manually, ensuring that they do not contain any fines.

— weigh the undersized material, i.e. the MF fine pieces (in kilograms) small enough to pass through the LDF mm sieve;

— the elements that do not pass through the sieve are used to determine the maximum lengths as follows: place each element not passing through the sieve on the flat measurement surface and use the ruler to measure to within ± 5 mm the largest length projected onto the measurement plane, without deforming the piece and excluding filaments.

  Any foreign body identified during this step shall be reported with a warning that it may not be representative since the sampling plan is not designed for such foreign bodies;

— obtain the total mass of any elements consisting solely of metal wires released from the pieces of end-of-life tyre (and which are not counted in the TNP pieces) (MLM) (in kilograms);

— calculate the mass percentage of the loose metal wires (MPM) = 100 × MLM/MS;

— calculate the mass percentage of the fine pieces (MPF) = 100 × MF/MS;

— from the projected lengths excluding filaments, calculate the numerical percentages per class (and optionally the mass percentages);

NOTE 3 The histogram traced from these percentages gives an overview of the distribution of the maximum projected dimensions excluding filaments.

— calculate the number percentage of large pieces (NPL) (%) (and optionally the mass percentage (MPL) (%)) corresponding to the piece larger than the higher dimension of the HDF format;

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— calculate the number percentage (NPC) (%) (and optionally the mass percentage (MPC) (%)) of the pieces in the NCC central classes amongst the NCR classes between the lower and higher dimensions of the format (LDF and HDF).

6.9 Metrological characteristics

When this Technical Specification was adopted by CEN, the test specified in this clause had not been validated and no data on robustness, repeatability and reproducibility was available.

6.10 Measurement report

To conform to this Technical Specification, the measurement report shall include at least the following information:

a) general:

   1) reference to this technical specification;

   2) any deviation from this technical specification;

   3) kind of determination;

   4) address of the sampling body and of the testing laboratory;

   5) body responsible for the whole testing (sampling body or quantification body);

b) sampling plan, taking of the field sample and producing the laboratory sample(s):

   1) name of the sampler;

   2) place where the sample(s) were taken possibly with photograph;

   3) date/hour of increment taking;

   4) population and lot;

   5) weighing results at the different sampling steps;

   6) storage conditions;

c) preparation of the test portion from the laboratory sample:

   1) the mass of the laboratory sample (MS) (in kilograms);

   2) report on any eventual observed foreign body;

NOTE Presently there is no systematic test method available for foreign bodies especially for efficient sampling procedures.

d) measurement of the lengths and statistical assessment:

   1) the name(s) of the person(s) performing the measurement;

   2) the mass (in kilograms) of the fine pieces (MF);

   3) the results of the measurement of the projected lengths excluding filaments (in millimetres);

   4) the mass of the loose metal wires (MLM) (in kilograms);

5) the mass percentage of the loose metal wires (MPM) (%);

6) the mass percentage of the fine pieces (PMF) (%);

7) the number percentage of large pieces (PNG) (%) corresponding to the piece larger than the higher dimension of the HDF format (in millimetres);

8) the num percentage (PNC) (%) of the fractions in the NCC central classes amongst the NCR classes between the lower and higher dimensions of the LDF (in millimetres) and HDF (in millimetres) format.

7 Determination of characteristics of the separated fractions steel and textiles

7.1 General

In addition to rubber, textile and steel are two further materials obtained from the recycling of end-of-life tyres. These materials generally appear at different stages of the recycling processes in the form of fluff for textiles and steel wires. The size and quality of the textile and steel depends on the individual recycling process, and in particular, at which stage in the recycling process they are extracted. The textile and steel products usually have free or attached residual rubber content and reciprocally free or attached residual steel and textile content. These products are generally handled in bulk and can be baled or compressed to facilitate handling and transport. Examples are given in informative Annex E.

7.2 Preparation of sampling plan for steel and textiles and method for sampling

7.2.1 Principle of sampling

The main principle of sampling is to obtain a representative sample(s) from a lot of material from which a characteristic is to be determined. If the lot is to be represented by a sample then every particle in the lot must have an equal probability of being included in the sample (i.e. probabilistic sampling). When these principles cannot be applied in practice, the sampler shall define a procedure as close as possible to probabilistic sampling in his judgement (i.e. judgemental sampling) and note the limitations in the sampling plan and sampling report.

In general, it is difficult to take samples in a way that ensues truly representative sampling when a material is stationary (for example in a stockpile, big bag or silo). Regarding steel and textile, it is recommended to take samples when the materials are in movement.

7.2.2 Sampling plan

A written sampling plan shall be prepared before samples are taken according to this Technical Specification. The sampling plan shall specify the objectives of the testing program through consultation with all involved parties. These involved parties are e.g. the client, the producer of the textiles or steel and the sampler. The sampling plan shall specify the primary objectives of the testing program. A form for the sampling plan shall be prepared containing the following minimum information:

— a unique identification number of the sample;

— the name of the sampler;

— the date and time of sampling;

— the lot identification number which is to be tested;

— the name of the company.

Once completed, this form becomes the sampling certificate.

7.2.3 Definition of a lot and determining lot size

The lot size shall be defined by the producer and is a fixed quantity for which the quality is to be determined. The lot size (M_{lot}) may be defined by the producer as:

— a fixed quantity produced between machine settings;

— a fixed quantity in a production day/shift;

— a fixed quantity minimum of 100 t.

The lot size is based on production quality management decisions or specific customer requirements.

7.2.4 Determination of the number of increments

The minimum number of increments depends on the size of the lot to be sampled.

$$n = 4 + 0{,}01 \times M_{lot}$$

where

n is the minimum permitted number of increments rounded off to the nearest whole number;

M_{lot} is the mass of the lot in tonnes.

7.2.5 Determination of minimum sample size

a) Textile

1) mass of minimum sample: 0,2 kg;

2) volume of minimum sample: 2 dm^3.

b) Steel

1) mass of minimum sample: 0,5 kg;

2) volume of minimum sample: 2 dm^3.

NOTE Mass and volume have been calculated according to existing experience and the principles described in Annex D of prEN 15442:2009.

7.2.6 Determination of the minimum increment size

a) Textiles:

1) mass of minimum increment size: 0,2 kg;

2) volume of minimum increment: 2 dm^3.

b) Steel:

1) mass of minimum increment: 0,5 kg;

2) volume of minimum increment: 2 dm^3.

NOTE Mass and volume have been calculated according to existing experience and the principles described in Annex D of prEN 15442:2009.

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7.3 Handling and storage of samples

The sampling plan shall identify the procedure(s) selected for packaging, preservation, storage, and transport of the laboratory sample. The samples shall be kept in a dry room in sealed packaging. Samples that are stored shall be pre-dried if required. The sampling certificate is to be attached.

7.4 Determination of characteristic of the separated fractions steel and textiles by manual sorting of all components

7.4.1 General principles

A sample is subjected to manual sorting of its components (rubber, steel and textiles) and the posterior weighing of those components. The components may be separated directly by hand and/or using sieves and/or magnets. The textile and steel fractions may have residual rubber attached.

7.4.2 Equipment

Sieves are used to separate the textile and steel from the residual rubber powder or rubber granulates. For this purpose appropriate sieves shall be chosen. The geometry of the apertures shall be either circular or square, the thickness of the sieves, the distances of the holes and the diameter of the holes shall be in accordance with ISO 3310-1 and or ISO 3310-2. The frame of the sieves shall have a height that enables the sieves to contain the samples and all for a free movement of the sample during the sieving process.

Magnets are used to separate remaining ferrous particles inside the raw sample and also inside pre-separated fractions.

Scales: a scales capable of measuring the mass of the sorted fractions to the nearest 0,1 g.

7.4.3 Procedure

— If required, reduce the original sample to a smaller but representative sample by size reduction;

— weigh the sample;

— pour the sample over a well lit working table with millimetric reference;

— take reference picture;

— sort components of the sample according to pre-defined fractions: manually, using sieves and using hand magnets;

— the remaining textile or steel fluff in the sieve should be carefully recovered;

— the rubber powder or rubber granulate should be carefully recovered;

— then proceed to further separation of components and weighing of fractions;

— weigh each fraction;

— take reference photographs of each fraction;

— all fraction, free steel, free textile, rubber and mixed components (rubber+steel, rubber+textile) are collected separately and weighed;

— the fractions are reported as percentage of the original mass of the test sample;

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— complete the test report.

7.5 Calculation

Record the various masses on a test data sheet alongside the corresponding separated fractions. If there is more than a 1 % (for textiles) or 5 % (for steel) difference between the total mass of the separated fractions and the mass of the sample, then the test shall be repeated.

7.6 Metrological characteristics

When this Technical Specification was adopted by CEN, the test specified in this clause had not been validated and no data on robustness, repeatability and reproducibility was available.

7.7 Report

The test report shall include at least the following information:

— identification of the testing laboratory and the testing method;

— identification of the testing date;

— reference to this Technical Specification;

— identification of the product tested with reference to the lot;

— identification of the sample tested and the mass of the sample;

— photographs of the sample and the separated fractions: rubber, textiles and steel;

— conditions and observations, e.g. unusual occurrences during the test procedure which may affect the result;

— conditions and observations, e.g. visual quality, contamination of the sample;

— test results as demonstrated in the present Technical Specification.

Annex A
(normative)

Evaluation of protruding filaments for shreds and cuts and chips considered as very small shreds

A.1 General

Depending on the agreed test programme between the producer and the customer, or at the producer's own will, after the determination of dimensions specified in Clause 6 for shreds and cuts and for chips considered as very small shreds, the additional determination of filaments may be carried out according to this normative Annex A.

A.2 Principle of the evaluation

The method for evaluating the "filaments" of shredded materials determines two parameters:

a) ANPF = Average number per Piece of Filaments longer than MLF1;

b) NPF = Number Percentage of pieces having at least one Filament longer than MLF2.

This evaluation is based on a measurement by image analysis as specified in the following subclauses determining the filaments as filiform metallic and/or textile protruding wires (see 3.28) at least as long as MLF mm. The evaluation is generally combined with a determination by image analysis of the largest projected length (excluding filaments).

A.3 Sampling

A laboratory sample shall be obtained according to the principles of sampling set out in 6.2, the preparation of the sampling plan for determining dimensions set out in 6.3 and the procedure for taking the field sample and producing the laboratory sample(s) set out in 6.4.

A.4 List of symbols and abbreviations

This subclause lists the symbols and abbreviations used in other subclauses of this Annex A dedicated to filaments:

— MLF Minimum length of a filament (in millimetres);

— MLF1 Minimum length of a filament (in millimetres) for the criterion average number of filaments per piece;

— MLF2 Minimum length of a filament (in millimetres) for the criterion number percentage of pieces having at least one filament;

— ANPF Average Number per Piece of Filaments longer than MLF1;

— NPF Number Percentage of pieces having at least one Filament longer than MLF2;

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— LDF Lower Dimension of the Format (in millimetres);

— HDF Higher Dimension of the Format (in millimetres);

— MLM The Mass of the Loose Metal wires (in kilograms) as defined in Clause 6;

— MPM The Mass Percentage of the loose Metal wires (%) as defined in Clause 6.

A.5 Principle for determining dimension(s) by image analysis

This method is in general identical to the manual method, except that the quantification of the filaments of each piece is performed by image analysis and not manually as specified in the dimensional determination in Clause 6.

NOTE 1 The manual method described in 6.8 was defined for the determination of the maximum projected length, without taking into account the filaments. However it is not generally considered as easy to apply this method to quantifying the filaments in the same way by hand on at least 100 pieces each exhibiting, in general, several filaments. Such practical limitations are not encountered with the optical method by image analysis as described in this annex.

NOTE 2 General information on the determination of dimensions by image analysis can be found in the standard quoted [2] in the bibliography.

A.6 Equipment

— Vessel large enough to contain at least 100 pieces (50 for cuts);

— automated measurement system using a process for the determination, by image analysis, of the largest projected length of each piece. This automated system is used to measure lengths (projected length of pieces or length of filaments) to within ± 5 mm up to 500 mm (1 000 mm for cuts);

— scales to weigh within 0,01 kg;

— circular mesh sieve in accordance with ISO 565 or ISO 3310-1 with a mesh of LDF (in millimetres).

A.7 Procedure

Proceed as specified in 6.8 with the following specific requirements for the optical measurement by image analysis of the maximum projected length and for the filaments:

— put the pieces that do not pass through the sieve on the flat image acquisition area of the automated measurement system. Place the pieces so that the largest area is in contact with the flat surface.

  Any foreign body identified during this step shall be reported with a warning that it may not be representative since the sampling plan is not designed for such foreign bodies;

— ensure that the pieces are completely separated, especially the filaments of each piece are completely separated from the filaments of the other pieces. Ensure that they are all within the acquisition area;

— capture the image of the arranged pieces;

— repeat the operation until all the pieces in the sample have been dealt with;

— start to process the images with the measurement equipment to determine the largest projected length of each of the TNF pieces in the sample and to determine the filaments in each piece of the sample;

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— calculate ANPF, the Average Number per Piece of Filaments longer than MLF1;

— calculate NPF, the Number Percentage of pieces having at least one filament longer than MLF2.

A.8 Metrological characteristics

When this Technical Specification was adopted by CEN, the test specified in this annex had not been validated and no data on robustness, repeatability and reproducibility was available.

A.9 Measurement report

To conform to this Technical Specification, the measurement report shall include at least the following information:

a) general:

   1) reference to this Technical Specification;

   2) any deviation from this Technical Specification;

   3) kind of determination;

   4) address of the sampling body and of the testing laboratory;

   5) body responsible for the whole testing (sampling body or quantification body);

b) sampling plan, taking of the field sample and producing the laboratory sample(s):

   1) name of the sampler;

   2) place where the sample(s) were taken possibly with photograph;

   3) date/hour of increment taking;

   4) population and lot;

   5) results of weighing at the different sampling steps;

   6) storage conditions;

c) preparation of the test portion from the laboratory sample:

   1) the mass of the laboratory sample (MS) (in kilograms);

   2) description of any eventual foreign body;

d) measurement of lengths and statistical assessment:

   1) the mass (in kilograms) of the fine pieces (MF);

   2) the results of the measurement of the projected lengths excluding filaments (in millimetres);

   3) the results of the measurement of the filaments;

   4) the mass of the loose metal wires (MLM) (in kilograms);

5) the mass percentage of the loose metal wires (MPM) (%);

6) the mass percentage of the fine pieces (PMF) (%);

7) the number percentage of large pieces (PNG) (%) corresponding to the piece larger than the higher dimension of the HDF format (in millimetres);

8) the number percentage (PNC) (%) of the fractions in the NCC central classes amongst the NCR classes between the lower and higher dimensions of the LDF (in millimetres) and HDF (in millimetres) format;

9) ANPF, the Average Number per Piece of Filaments longer than MLF1;

10) NPF, the Number Percentage of pieces having at least one filament longer than MLF2.

Annex B
(normative)

Determination of free steel content for granulates and powders and chips considered as very large granulates by the method of magnetic separation

B.1 General

Depending on the agreed test programme between the producer and the customer, or at the producer's own will, after the determination of dimensions specified in Clause 5 for granulates and powders and chips considered as very large granulates the additional determination of free steel may be done according to this normative Annex B.

B.2 Principle of the determination

The objective of this determination is to find the quantity of free steel contained in granulates, powders or chips considered as very large granulates by using a very specific method, that is the one where steel particles are separated from the rubber particles through the use of a magnet.

Difficulties may be encountered for large particles since steel may be incorporated in the rubber. When the magnet drags rubber with steel, measure and report the quantity of rubber that incorporates steel.

B.3 Sampling

Preparation of the sampling plan shall be done according to 5.2. Storage and sample transportation shall be done according to 5.3. The preparation of the laboratory sample and test portion shall be done according to 5.4 but the final mass quantity for the purpose of magnetic separation shall be a combined sample consisting of as many samples (increments) as needed to reach a total mass as close as possible to 500 g.

B.4 Equipment

— A tray made with nonmagnetic material for spreading the sample and obtaining a thickness of less than 2 cm (for instance 60 cm by 60 cm);

— a permanent magnet made of neodymium with an intensity of minimum 1 tesla and an area of not less than 2 cm^2;

— the scales for weighing the laboratory sample shall have an accuracy of 0,1 g;

— the scales for weighing the metal wire shall have an accuracy of at least 0,01 g.

B.5 Procedure

— Break the aggregates in order to secure proper particle separation.

— Place the dry, crumbled up samples in the tray and level off in a 2 cm thick maximum layer.

— Hold the permanent magnet in the hand, pass it over the sample at a distance of less than 1 cm.

— Recover in the container all the fragments adhering to the magnet, i.e. the free steel particles and the rubber particles incorporating steel.

— Mix the sample in the tray after the first pass of the magnet and reconstitute a 1 cm to 2 cm layer.

— Pass the magnet over the sample again. If elements are still recovered by the magnet, repeat the mixing and magnet passing operation. These two operations will be carried out until the magnet is exempt from all adhering elements when passing it over the sample (time should be not less than 2 min).

— Weigh the recovered free steel particles and rubber particles incorporating steel.

B.6 Calculation

The ferrous wire content in percent is given by:

$$M_p = (M_{metal} \times 100) / M_0$$

where

M_p is the percentage of free steel in the laboratory sample;

M_0 is the mass, in grams, of the laboratory sample;

M_{metal} is the mass, in grams, of ferromagnetic elements recovered by the permanent magnet.

B.7 Metrological characteristics

When this Technical Specification was adopted by CEN, the test specified in this annex had not been validated and no data on robustness, repeatability and reproducibility was available.

The minimum precision of the scales specified in B.4, combined with the mass of the test sample, should allow for the correct quantification of collected free steel greater than 0,05 %.

B.8 Measurement report

To conform to this Technical Specification, the measurement report shall include at least the following information:

a) reference to this Technical Specification;

a) any deviation from this Technical Specification;

b) address of the sampling body and of the testing laboratory;

c) body responsible for the whole testing (sampling body or quantification body);

d) name of the sampler;

e) description of where the sample(s) were taken optionally with photograph;

f) date/hour of increment taking;

g) population and lot;

h) weighing results at the different sampling steps;

i) storage conditions;

j) the mass of the laboratory sample (MS) (in kilograms);

k) the name(s) of the person(s) performing the measurement;

l) the results of the measurement of the free steel content.

Annex C
(normative)

Determination of free textile content for granulates and powders and chips considered as very large granulates by the method of the "small ball" agglomeration

C.1 General

Depending on the agreed test programme between the producer and the customer, or at the producer's own will, after the determination of dimensions specified in Clause 5 for granulates and powders and chips considered as very large granulates the additional determination of free textile may be done according to this normative Annex C.

NOTE This method does not take into account the textile fibres embedded in the granulates.

C.2 Principle of the determination

The objective of this determination is to find the quantity of free textile contained in granulates, powders or chips considered as very large granulates by extracting this impurity from the rubber in the form of small balls during the process of sieving.

NOTE The process generating the small ball may be disturbed in presence of large particles.

C.3 Sampling

Preparation of the sampling plan shall be done according to 5.2. Storage and sample transportation shall be done according to 5.3. The preparation of the laboratory sample and test portion shall be done according to 5.4. Very small particles of rubber may be entrapped in the small balls. They could be dislodged from the small balls.

C.4 Equipment

— Same as those used for sieving analysis (5.5.2);

— the scales for weighing the small balls shall have an accuracy of 0,01 g.

C.5 Procedure

— Same as those used for sieving analysis (5.5.6).

— After the sieving of the material for the size distribution determination, on every sieve it could be found one or more small balls that have been formed by the free textile during the sieving time.

— Collect all these small balls from every screen.

— If they have entrapped very small particles of rubber granulate, it is possible to dislodge these particles by shaking the small balls.

— At the end of this procedure weigh all the "textile balls" in grams.

C.6 Calculations

The free textile content in percentage is given by the formula:

$$T_p = (T_{ball} \times 100) / M_0$$

where

T_p is the percentage of free textile in the sample;

M_0 is the mass, in grams, of the laboratory sample;

T_{ball} is the mass, in grams, of all the "textile balls" extracted.

C.7 Metrological characteristics

When this Technical Specification was adopted by CEN, the test specified in this annex had not been validated and no data on robustness, repeatability and reproducibility was available.

The minimum precision of the scales specified in C.4, combined with the mass of the test sample, should allow for the correct quantification of the collected free textile greater then 0,1 %.

C.8 Measurement report

To conform to this Technical Specification, the measurement report shall include at least the following information:

a) reference to this Technical Specification;

b) any deviation from this Technical Specification;

c) address of the sampling body and of the testing laboratory;

d) body responsible for the whole testing (sampling body or quantification body);

e) name of the sampler;

f) description of where the sample(s) were taken optionally with photograph;

g) date/hour of increment taking;

h) population and lot;

i) weighing results at the different sampling steps;

j) storage conditions;

k) the mass of the laboratory sample (MS) (in kilograms);

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l) the name(s) of the person(s) performing the measurement;

m) the results of the measurement of the free textile fraction.

Annex D
(normative)

Determination of other impurities content for granulates and powders and chips considered as very large granulates by the method of a saline solution

D.1 General

Depending on the agreed test programme between the producer and the customer, or at the producer's own will, after the determination of dimensions specified in Clause 5 for granulates and powders and chips considered as very large granulates, the additional determination of other impurities may be done according to this normative Annex D.

D.2 Principle of the determination

The objective of this determination is to find the quantity of other dispersed impurities (other than steel and textile contained in granulates or powders or chips considered as very large granulates) by using a densimetry method. The principle uses the differences of density between granulate or powder or chip considered as very large granulate and the most frequent impurities coming from processing tyres such as glass, sand or non-magnetic metals. This method is applicable only to such impurities which are dispersed among the material to be sampled.

NOTE The testing process may be disturbed in presence of large particles.

D.3 Sampling

Preparation of the sampling plan will be carried out according to 5.2. Storage and sample transportation shall be carried out according to 5.3. The preparation of the laboratory sample and test portion shall be carried out according to 5.4 but the final mass quantity for the purpose of the determination of impurities by the method of the saline solution will be a combined sample consisting of as many samples (increments) as needed to reach a total mass as close as possible to 150 g, after the free steel determination.

D.4 Equipment

— Beaker with a capacity of 2 000 cm^3;

— funnel;

— filter paper;

— container to collect the saline water with impurities;

— oven with a heating capacity to achieve (105 ± 2) °C;

— the scales for weighing the laboratory sample shall have an accuracy of 0,1 g;

— the scales for weighing the other impurities shall have an accuracy of at least 0,01 g.

D.5 Procedure

— Prepare a saline solution consisting of 1 l of water and 300 g of salt (sodium chlorine), to obtain a solution with a density higher than 1,25 g/cm^3.

NOTE It is very important to achieve a complete dissolution of the salt in water. For this purpose it is possible to heat the water and help the salt to be dissolved in water. If this alternative is used, let the solution achieve room temperature before carrying out the test.

— Before adding the test material to the solution it is necessary to completely clean the test sample of any steel and textile. For this purpose the evaluation of free steel and textile should be determined by carrying out the test methods described in Annexes B and C on the same sample.

— Introduce the saline solution into the beaker and add the test material to it.

— Add to the saline water a drop of detergent in order to reduce the occluded air bubbles in the faces of the particle due to the surface tension.

— Stir the mixture for at least 3 min to permit a good exchange of granulate from the top to the bottom and to avoid the formation of agglomerates that can retain impurities inside them.

— Once the equilibrium is achieved, withdraw the granulates floating on the top of the solution using the stirring bar.

— After all material has been withdrawn, pour carefully the salty solution into the filter paper previously placed in the funnel.

— Wash the impurities retained in the filter paper adding 1 l of water and eliminate salt from it.

— Introduce the filter into the stove and let it dry completely. Withdraw the material retained in the filter.

— Allow enough time to achieve room temperature before weighing all impurities.

D.6 Calculations

The content of other impurities obtained from the test is expressed as a percentage of the total mass of material and is given by:

$$\% \text{ MFM} = M_1 / M_0 \times 100$$

where

MFM is the percentage of other impurities in the laboratory sample;

M_0 is the mass, in grams, of the laboratory sample;

M_1 is the mass, in grams, of impurities recovered in the filter paper.

D.7 Metrological characteristics

When this Technical Specification was adopted by CEN, the test specified in this annex had not been validated and no data on robustness, repeatability and reproducibility was available.

The minimum precision of the scales specified in D.4, combined with the mass of the test sample, should allow for correct quantification of the collected other impurities greater than 0,1 %.

D.8 Measurement report

To conform to this Technical Specification, the measurement report shall include at least the following information:

a) reference to this Technical Specification;

b) any deviation from this Technical Specification;

c) address of the sampling body and of the testing laboratory;

d) body responsible for the whole testing (sampling body or quantification body);

e) name of the sampler;

f) description of where the sample(s) were taken optionally with photograph;

g) date/hour of increment taking;

h) population and lot;

i) weighing results at the different sampling steps;

j) storage conditions;

k) the mass of the laboratory sample (MS) (in kilograms);

l) the name(s) of the person(s) performing the measurement;

m) the results of the measurement of the other impurities.

Annex E
(informative)

Example of applications of Clause 7 to steel and textiles

E.1 Example for textiles

E.1.1 Sampling

Take textile increments.

Minimum 2 dm^3 or 0,2 kg.

E.1.2 Sample

From increment produce sample.

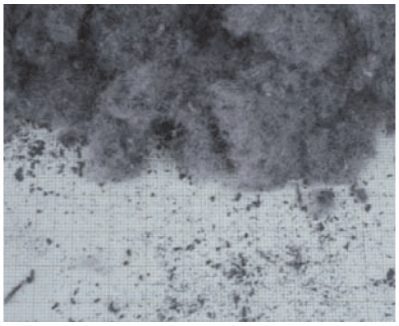
If required apply sample reduction size.

Weigh the sample.

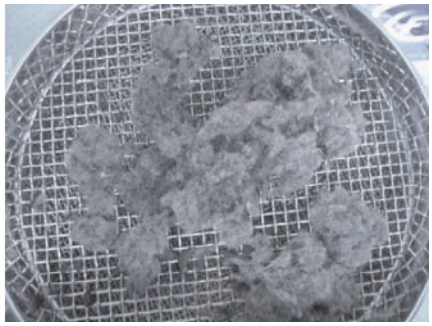
Take reference photograph.

M_{sample} = 205,5 g.

a) Textile sampling



b) Partial view of test sample



c) Textile separation

Figure E.1

E.1.3 Separation of fractions textile, steel, rubber

Use a sieving machine and hand magnets. For example from sieve 7,1 mm down to 0,25 mm.

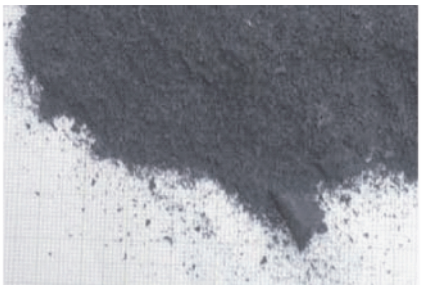
Take note of sieving time and amplitudes.

NOTE A primary separation could be made moving the sample by hand.

E.1.4 Separation of steel using hand magnet

From the fractions obtained in the sieving equipment, separate steel using hand magnet.

a) Textile fraction



b) Rubber fraction

c) Steel fraction

Figure E.2

E.1.5 Sorted fractions

Weigh each fractions.

Take reference photographs: textiles, rubber and steel.

E.1.6 Results

Table E.1

Fractions	**Mass** g	**%**
Sample	205,5	100
Textile	140,2	68,2
Rubber	64,1	31,2
Steel	1,2	0,6

E.2 Example on steel

E.2.1 Sampling

Take increments steel: minimum 2 dm^3 or 0,5 kg.

E.2.2 Sample

From increments, produce sample.

If required apply sample reduction size.

Weigh the sample.

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Take reference photographs.

M_{sample} = 2,0 kg.

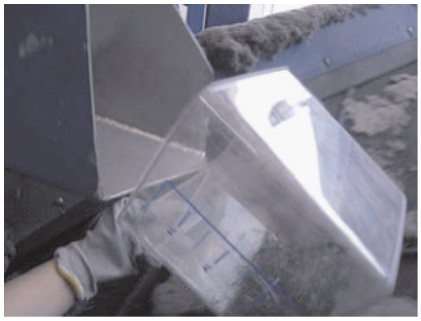
E.2.3 Separation of fractions

Use a sieving machine and hand magnets.

For example from sieve 15 mm down to 2,5 mm.

Take note of sieving time and amplitudes.

NOTE A primary separation could be made moving the sample "by hand".



a) location of steel sampling

b) steel sample

Figure E.3

E.2.4 Separation of steel using hand magnet

From the fractions obtained in the sieving equipment, separate steel using hand magnet.

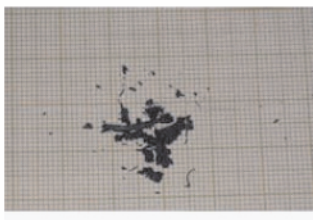
E.2.5 Sorted fractions

Weight each fraction.

Take reference pictures: steel, steel+rubber, rubber, textile.

a) steel fraction

b) steel plus rubber fraction



c) rubber fraction

Figure E.4

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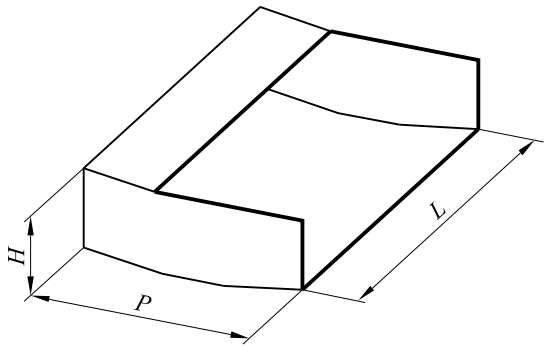
E.2.6 Results

Table E.2

Fractions	**Mass** g	**%**
Sample	519,0	100
Steel	364,3	70,2
Steel+Rubber	114,2	22,0
Rubber	36,7	7,1
Textiles	3,8	0,7

Annex F
(informative)

Example of scoop



Key

L Width

P Depth

H Edge height

Width *L* at least 1,5 times the width of the falling flow
Depth *P* at least 2/3 of the width *L* and at least 2,5 times the higher dimension HDF
Edge height *H* equal at least to 1/3 of the width *L* and at least two times the higher dimension HDF

Figure F.1

Annex G
(informative)

Sieving

Different factors influence the quality of sieving tests. Part 1 of DIN 66165 addresses them in detail and the requirements on load below are adapted from this Technical Specification.

The quantity of the material to be sieved, termed "charge", should be limited so as to enable each particle to meet a free sieve aperture within a reasonable time, and not to exceed the permissible loading of the sieving medium. The charge volume should therefore not exceed twice the maximum volume of the residue permitted at the completion of sieving.

It should be ensured, by selecting the appropriate sieve aperture sizes for each sieve within a set, that the charge is uniformly distributed over the whole set. If necessary, preliminary sieving should be carried out. The minimum quantity of the individual sieve fractions obtained should meet the requirements in respect of sample size and the precision of the balance used.

Table G.1

Nominal aperture size of sieving medium mm	**Maximum permissible volume of residue per unit area of sieving medium** cm^3/dm^2
125	580
90	470
63	370
45	280
31,5	230
22,4	180
16	145
11,2	115
8	90
5,6	70
4	55
2,8	45
2	35
1,4	30
1	20
0,71	18
0,5	14
0,355	11
0,25	9

(continued)

Table G.1 *(continued)*

Nominal aperture size of sieving medium mm	**Maximum permissible volume of residue per unit area of sieving medium** cm^3/dm^2
0,18	7
0,125	6
0,09	4
0,063	4
0,045	3
0,032	2
0,02	2
0,016	1,5
0,01	1

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