
Snov iz izrabljenih avtomobilskih gum - Jeklena žica - Ugotavljanje deleža nekovinskih materialov

Materials produced from end of life tyres - Steel wire - Determination of the non-metallic content

Aus Altreifen gewonnene Materialien - Stahldrähte - Bestimmung der nicht-metallischen Bestandteile

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Pneus usagés non réutilisables (PUNR) - Fils métalliques - Détermination de la teneur en matériaux non métalliques

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83.160.01	Avtomobilske pnevmatike na splošno	Tyres in general

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**Materials produced from end of life tyres - Steel wire -
Determination of the non-metallic content**

Pneus usagés non réutilisables (PUNR) - Fils
métalliques - Détermination de la teneur en matériaux
non métalliques

Aus Altreifen gewonnene Materialien - Stahldrähte -
Bestimmung der nicht-metallischen Bestandteile

This Technical Specification (CEN/TS) was approved by CEN on 21 January 2019 for provisional application.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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CEN/TS 17308:2019 (E)**European foreword**

This document (CEN/TS 17308:2019) has been prepared by Technical Committee CEN/TC 366 “Materials obtained from End-of-Life Tyres (ELT)”, 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 shall not be held responsible for identifying any or all such patent rights.

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

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1 Scope

This document provides two different methods for the quantitative estimation of non-metallic content remaining adhered to the steel wire obtained from the recovery of materials from end-of-life tyres.

The pyrolysis method is considered as the reference method while the hydrostatic method is considered as an in-situ method.

This European Standard includes sample collection and the preparation of representative samples based on a sampling plan for the purpose of their characterization.

This European Standard does not cover the operational performance or fitness for use of the materials which are deemed to be a function of agreements between the manufacturer and the customer.

This European Standard does not purport to address all the safety concerns, if any, associated with its use. This European Standard does not establish appropriate safety and health practices and does not determine the applicability of regulatory limitations prior to its use.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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.

CEN/TS 14243, *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*

3 Terms and definitions (standards.iteh.ai)

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

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- IEC Electropedia: available at <http://www.electropedia.org/>
- ISO Online browsing platform: available at <http://www.iso.org/obp>

3.1

sample

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

3.2

increment

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

[SOURCE: ISO 13909-1:2016, 3.15: modified – "fuel" has been replaced with "material"]

3.3

characteristic

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

Note 1 to entry: The characteristic may be either quantitative (by variables) or qualitative (by attributes).

3.4

lot

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

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Note 1 to entry: In sampling standards, the lot is also designated 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.

3.5 combined sample

sample consisting of all the increments taken from a lot

Note 1 to entry: A combined sample is a quantity of material, representative of the lot for which the quality is to be determined.

3.6 field sample

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

3.7 laboratory sample

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

[SOURCE: IUPAC definition]

Note 1 to entry: 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 to entry: 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.

3.8 test sample

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

[SOURCE: IUPAC definition]

3.9 population

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

Note 1 to entry: 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.

3.10 representative sample

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

[SOURCE: IUPAC definition]

3.11 probabilistic sampling

sampling conducted according to the statistical principles of sampling

3.12**steel wire**

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

4 Testing programme

When performing a testing programme for determining product characteristics, 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 and coordination between these different testing steps. The steps are:

- a) sampling plan;
- b) taking field sample(s);
- c) storage of the sample(s), transport;
- d) pre-treatment, e.g. drying (if needed);
- e) quantification, analysis, calculations;
- f) 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 fulfill the applicable requirements specified in the present European Standard. This includes calibration of equipment, e.g. calibration of scales.

5 Determination of non-metallic content in steel wire**5.1 General**

Percentage of non-metallic content is a key measurement required for product classification of steel wire. Before being able to obtain an accurate value, a representative sample of the material to be tested shall be taken.

5.2 Preparation of sampling plan and laboratory sample**5.2.1 Principle 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 shall have an equal probability of being included in the sample (i.e. probabilistic sampling). These principles cannot be fully applied in practice with steel wire, so a different procedure is defined to ensure as much as possible the probabilistic sampling.

NOTE The objective of this procedure is to take samples in a way that is truly representative of the material produced. It is easier when the material is moving (for example on a conveyor belt). Therefore, sampling from moving material will be preferred whenever possible.

5.2.2 Sampling plan

A written sampling plan shall be prepared, before taking samples according to 5.2.4 to 5.2.8. 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:

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- a) the name of the producer;
- b) a unique identification number of the sample;
- c) the name of the sampler;
- d) the location(s), date and time of sampling;
- e) the lot identification number which is to be tested (based on 5.2.3);
- f) reference to this European Standard;
- g) any deviation from this European Standard.

Once completed, this form becomes the sampling certificate.

5.2.3 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 determined. The lot size M_{lot} may be defined by the producer as:

- a) a fixed quantity produced between machine settings;
- b) a fixed quantity in a production day/shift;
- c) a fixed quantity minimum of 10 t.

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

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5.2.4 Sampling point and apparatus

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Based on health and safety assessments and producer equipment, a fixed point for the collection of sample increments shall be chosen. 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.

Sample increments shall be collected with the same frequency within the lot in order to represent the whole lot.

5.2.5 Size of a sample increment

Both criteria shall be fulfilled:

- mass of minimum increment: 0,5 kg;
- volume of minimum increment: 2 dm³.

NOTE Mass and volume have been calculated according to existing experience.

5.2.6 Number of sample increments

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

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

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.

5.2.7 Metrological characteristics of sampling

The subclauses will be updated with the results of the second phase of the validation.

5.2.8 Visual assessment

The increments are 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 laboratory sample(s)

Increment(s) and combined sample(s) are placed in a sealed sample box in such a way that no material is lost during storage and transportation. In the case of a combined sample send to an external laboratory, it is stored dry and the sampling certificate attached. See 7.2 for pretreatment.

5.4 Laboratory sample(s) and test portion(s)

5.4.1 General principle

The shape and the size of the (steel wire do not allow performing subdivision of the sample and quartering without significantly affecting the sample composition. Sample duplication can only be performed by taking several sample increments at the same instant. Whenever more increments are joined, they can no longer be divided in test portions without changing their composition.

5.4.2 Laboratory sample

The laboratory sample can then be obtained by joining four or more increments from the same lot. Analysis of the laboratory sample will be conducted as described in 6 for the reference method or 7 for the in-situ method.

6 Determination by pyrolysis method

6.1 Principle

This method is based on decomposition in an inert atmosphere of a sample of steel wire taken after one of the mechanical grinding treatments of ELTs. Both steel and non-metallic content are weighed to evaluate the quantities of each components

6.2 Apparatus

6.2.1 Analytical scales

The analytical scales used to weigh the laboratory sample and the steel wire shall be accurate to ± 1 g.

6.2.2 Pyrolysis set-up

The pyrolysis set-up comprises:

- a base-mounted resistor furnace;