# CEN/TS 17510:2020

NBN

# Materials obtained from end-of-life tyres - Determination of the specific surface area of powders - Method based on krypton adsorption

Valid from 09-10-2020

ICS: 13.030.50, 83.160.01

Bureau for Standarisation
Rue Joseph II 40 PO box 6
1000 Brussels

T. +32 2 738 01 11
F. +32 2 733 42 64
info@nbn.be

BTW BE0880.857.592
IBAN BE41 0003 2556 2110
BIC Code BPOTBEB1

www.nbn.be

# TECHNICAL SPECIFICATION
# SPÉCIFICATION TECHNIQUE
# TECHNISCHE SPEZIFIKATION

**CEN/TS 17510**

October 2020

ICS 83.160.01; 13.030.50

English Version

# Materials obtained from end-of-life tyres - Determination of the specific surface area of powders - Method based on krypton adsorption

Matériaux produits à partir de pneus usagés non réutilisables - Détermination de la surface spécifique des poudrettes - Méthode fondée sur l'adsorption de krypton

Materialien aus Altreifen - Bestimmung der spezifischen Oberfläche von Mehlen - Verfahren basierend auf Kryptonadsorption

This Technical Specification (CEN/TS) was approved by CEN on 24 August 2020 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.

CEN members are the national standards bodies of 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, Republic of North Macedonia, Romania, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and United Kingdom.

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

**CEN-CENELEC Management Centre: Rue de la Science 23, B-1040 Brussels**



Ref. No. CEN/TS 17510:2020 E

**CEN/TS 17510:2020 (E)**

# Contents

Page

**European foreword** ... **3**
**Introduction** ... **4**
**1 Scope** ... **5**
**2 Normative references** ... **5**
**3 Terms and definitions** ... **5**
**4 Principle** ... **6**
**5 Apparatus** ... **7**
**5.1 General** ... **7**
**6 Procedure** ... **7**
**6.1 Sample preparation** ... **7**
**6.2 Analysis** ... **8**
**6.3 Analysis data** ... **8**
**7 Test report** ... **9**
**Bibliography** ... **10**

# European foreword

This document (CEN/TS 17510:2020) 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, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Republic of North Macedonia, Romania, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

# Introduction

Specific surface area ($A_S$) is a parameter of great importance when it comes to physical characterization of materials such as granulates and powders from rubber materials. Like other physical characteristics, specific surface area could influence the performance of materials in its different applications.

Depending on the type of sample to be characterized, several different methods can be used for the determination of the specific surface area, generally based on different physical principles. The most widespread and useful method used in materials characterization is gas adsorption, either through gravimetric or volumetric methods.

For very low surface area samples the traditional volumetric method of nitrogen adsorption at 77 K or Argon at 87 K shows some important limitations. Alternatively, for absolute areas as low as 0,05 $m^2g^{-1}$ the suitable method for $A_S$ determination is krypton adsorption at 77 K.

# 1 Scope

This document specifies a method for the determination of low specific surface area of powders ELTs rubber by measuring the amount of physically adsorbed krypton gas and applying the theoretical multipoint Brunauer, Emmett and Teller (BET) method.

This document defines a specific method for powders taking into account that, in order to obtain an accurate value of specific surface area, a representative sample of the material to be tested is taken according to the principle that every particle of the sample that represents the lot have an equal probability of being included in the sample.

# 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.

EN 14243-1:2019, *Materials obtained from end of life tyres — Part 1: General definitions related to the methods for determining their dimension(s) and impurities*

# 3 Terms and definitions

For the purposes of this document, the terms and definitions given in EN 14243-1:2019 and the following 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 https://www.iso.org/obp

**3.1**
**adsorption**
enrichment of the adsorptive gas at the external and accessible internal surfaces of a solid material

[SOURCE: ISO 15901-2:2006]

**3.2**
**adsorbate**
adsorbed gas

[SOURCE: 15901-2:2006]

**3.3**
**adsorptive**
gas or vapour to be adsorbed

[SOURCE: ISO 15901-2:2006]

**3.4**
**adsorbent**
solid material on which adsorption occurs

[SOURCE: ISO 15901-2:2006]

**3.5**
**isotherm**
relationship between the amount of gas adsorbed and the equilibrium pressure of the gas, at constant temperature

[SOURCE: ISO 15901-2:2006]

**3.6**
**surface area**
extent of available surface area as determined by a given method under stated conditions

[SOURCE: ISO 15901-1:2016]

**3.7**
**specific surface area**
absolute surface area of the sample divided by sample mass

**3.8**
**relative pressure**
ratio of the equilibrium adsorption pressure, $p$, to the saturation vapour pressure, $p°$, at analysis temperature

[SOURCE: ISO 15901-3:2007]

# 4 Principle

The determination of the specific surface area undergoes by the determination of the amount of adsorbate required to cover the external and accessible internal pore surface of the adsorbent with a complete monolayer of adsorbate. The monolayer capacity is then calculated by using the BET equation:

$$\frac{p/p°}{n_a\left[1-\left(p/p°\right)\right]} = \frac{1}{n_m C} + \frac{C-1}{n_m C} \times \left(p/p°\right) \quad (1)$$

where $n_a$ is the amount adsorbed at the relative pressure $p/p°$, $n_m$ is the monolayer capacity and C is a constant dependent on isotherm shape. By plotting $\frac{p/p°}{n_a\left[1-\left(p/p°\right)\right]}$ against $p/p°$ a linear relation is achieved and the $n_m$ value determined from slope and y-intercept values. The range of linearity of BET is usually restricted between 0,05 and 0,35, depending on the materials this range can be wider or shorter.

For low specific surface areas, lower than 1 $m^2g^{-1}$, the recommended method is krypton adsorption at 77 K (liquid nitrogen temperature). Since at 77 K krypton is below its triple point temperature, there are some studies that suggest the adsorbate could be in a liquid-like state and therefore it is more adequate to use the value of p° for supercooled liquid when plotting the BET.

The test is performed by automated equipment following the principle that gas is admitted to the sample cell, which is at a constant temperature of 77 K, and the amount of adsorbed gas is recorded after equilibration is achieved and plotted as relative pressure, $p/p°$.