SLOVENSKI STANDARD
SIST-TS CEN/TS 17307:2019

01-junij-2019

Snov iz izrabljenih avtomobilskih gum - Granulati in praški - Identifikacija elastomerov: odkrivanje piroliznih produktov v raztopinah z uporabo plinske kromatografije in masne spektrometrije

Material derived from End-of-Life tyres - Granulates and powders - Elastomers identification: Gas-chromatography and mass-spectrometric detection of pyrolysis products in solution

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Material aus Altreifen - Granulat und Mehle - Identifizierung von Elastomeren: Gaschromatographie und massenspektrometrische Detektion von Pyrolyseprodukten in Lösung

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Matériaux obtenus à partir de pneumatiques en fin de vie - Granulats et poudrette - Identification des élastomères : Détection par chromatographie en phase gazeuse et spectrométrie de masse des produits de pyrolyse en solution

Ta slovenski standard je istoveten z: **CEN/TS 17307:2019**

ICS:

71.040.50	Fizikalnokemijske analitske metode	Physicochemical methods of analysis
83.160.01	Avtomobilske pnevmatike na splošno	Tyres in general

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TECHNICAL SPECIFICATION

SPÉCIFICATION TECHNIQUE

TECHNISCHE SPEZIFIKATION

CEN/TS 17307

March 2019

ICS 83.160.01

English Version

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
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European foreword

This document (CEN/TS 17307:2019) 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.

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Introduction

WARNING — Persons using this Euopean Standard should be familiar with normal laboratory practice. This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

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1 Scope

This document specifies a method for the identification of the elastomers in granulates or powder derived from End-of-Life Tyres.

The method specified is a qualitative method only.

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.

ISO 1407, *Rubber — Determination of solvent extract*

3 Terms and definitions

No terms and definitions are listed in this document.

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

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4 Principle

A sufficient amount of granules or powder is compacted and homogenized in a laboratory mill and a small aliquot of the homogenized sample is then solvent extracted and subjected to pyrolysis at elevated temperature. Few drops of the liquid pyrolysis products are then diluted in dichloromethane for the GC/MS analysis. The use of the mass-spectrometric detector is a mean for improving the sensitivity and reliability of the identification of the elastomers present in low or trace amount, with threshold limit estimated to about 5 %.

The use of this standard pre-supposes sufficient working knowledge of the principles and techniques of gas chromatography/mass-spectrometry (GC/MS) for the analyst to perform the operations described and interpret the results correctly.

5 Reagents

5.1 Dichloromethane

5.2 Acetone

5.3 Nitrogen, for flushing the pyrolysis product.

6 Apparatus

All reagents shall be of analytical grade

6.1 Extraction apparatus. The apparatus specified in ISO 1407 is satisfactory.

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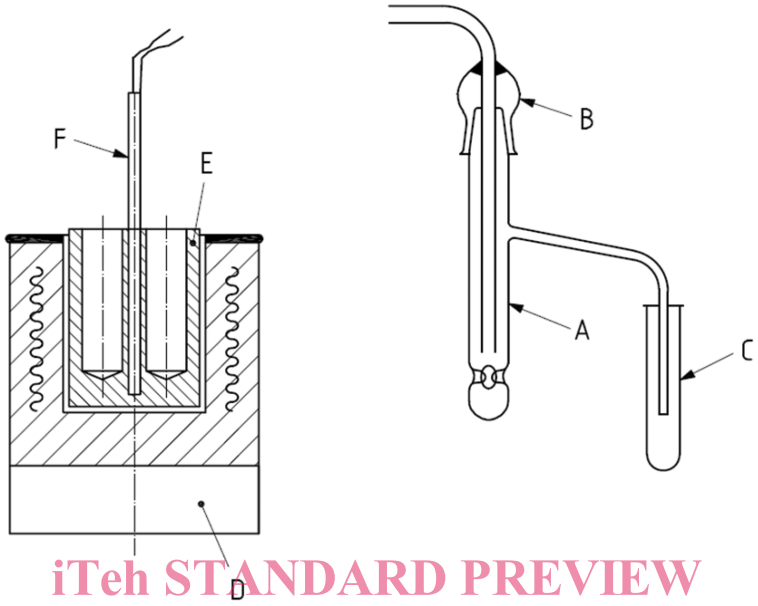
6.2 Pyrolysis apparatus (see Figure 1), comprising a glass tube A having inward projections to prevent the sample from falling to the bottom of the tube, and a lateral condenser tube. The tube A has a standard ground-glass joint B that carries a small glass adductor tube for the connection to flowing nitrogen. A collecting tube C is placed under the condenser tube. A thermo-regulated electric furnace D accommodates an aluminium block E with holes for one or more tubes A.



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Key

A glass tube for sample

B ground-glass joint

C collecting tube

D thermo-regulated electric furnace

E aluminium block, bored to hold tubes

F thermocouple

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Figure 1 — Pyrolysis apparatus

6.3 The chromatographic equipment consists of three associated parts:

6.3.1 Gas chromatograph equipped with chromatographic columns; capillary column in fused silica of 0,20 mm diameter and 30 m long, of apolar type stationary phase – film of dimethyl polysiloxane, 1 µm thick;

6.3.2 Mass detector in electron impact mode.

6.3.3 These elements are connected with an acquisition and data processing system.

6.4 Glassware

6.4.1 Vials of 2 ml capacity: caps with silicone septum, PTFE lined.

6.4.2 Capillary pipettes.

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7 Procedure

7.1 On a laboratory mill, thinly sheet, to about 0,25 mm to 0,5 mm thickness, a representative sample of about 0,5 kg of the rubber granulates or powder.

7.2 Extract with acetone (5.2) 2 g to 5 g of the homogenized sample in accordance with the procedure given in ISO 1407.

7.3 Depending on the nature of the composition of the unknown vulcanisate and of the type of apparatus used, place 0,5 g to 2 g of the extracted, dried test sample in the pyrolysis tube A (see Figure 1).

7.4 Bring the electric furnace D to 525 °C ± 50 °C and hold within this temperature range. This temperature range is recommended to obtain rapid pyrolysis without excessive degradation or carbonization. A temperature of 475 °C is advised, however, to obtain the maximum quantity of pyrolysate for NR, IR, BR, SBR, IIR, BIIR and CIIR.

7.5 Pass a slow stream of nitrogen (5.3) through the pyrolysis tube A and introduce the tube containing the prepared test sample into a hole in the aluminium block E. Nitrogen serves to displace air, prevent oxidation and facilitate transfer of the pyrolysis products into the collecting tube C. Maintain the nitrogen flow at 10 cm^3/min ± 2 cm^3/min.

7.6 Continue the heating to complete distillation, i.e. for about 10 min.

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7.7 Place a few drops (1 mg to 5 mg) of the homogenized pyrolysate into the vial (6.4.1), add about 1 ml of dichloromethane (5.1) and seal the vials with the cap and silicon septum.

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7.8 Inject 1 µl of the dichloromethane solution in the GC/MS analyser and acquire the data. The following conditions were tested for a quadrupole mass detector: other condition can be used provided that they provides similar results:

7.8.1 Gas-chromatograph set-up:

7.8.1.1 Carrier gas: helium

7.8.1.2 Constant pressure: 70 kPa: corresponding to a linear velocity of approximately 40 cm/s

7.8.1.3 Injector temperature: 300 °C

7.8.1.4 Injector: Splitless with gold-plated seal, Injection volume: 1,0 µL

7.8.1.5 Thermal program:

Isothermal for 4 min at 50 °C

Heating from 50 °C to 300 °C at the rate of 10 °C/min

Isothermal for 10 min at 300 °C

7.8.2 Mass spectrometric detector

7.8.2.1 Transfer line temperature: 280 °C

7.8.2.2 Source temperature: 230 °C