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Rubber — Determination of the mass concentration of tyre and road wear particles (TRWP) in soil and sediment — Pyrolysis-gas chromatography/mass spectrometry (GC/MS) method

Caoutchouc — Détermination de la concentration massique en particules d'usure des pneumatiques et de la route (TRWP) dans le sol et les sédiments — Méthode par pyrolyse-GC/MS

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO document should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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This document was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products.*

This first edition cancels and replaces (ISO/TS 21396:2017), which has been technically revised.

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The main changes are as follows:

— addition of information regarding instrument conditions for the microfurnace pyrolyser and resistive pyrolyser to 6.2.5.1;

— addition of recommendations for sample chemical pre-treatment to 10.5.4;

— revision of the calculation method to account for differences in elastomer microstructure between calibration standards and marketplace average tread;

— addition of 4-phenylcyclohexene marker to 4., 8., 10.1, 10.3, 10.4.2, 10.6.3.4, 11.1, 11.4, Annex C, Annex D and Annex E.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

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Introduction

Tyre and road wear particles (TRWP) are formed as a result of tread abrasion from the road surface, and subsequent particle release to the environment. TRWP consist of tyre tread particles which include incorporated material from the road surface (see Reference [1]). The elastomeric fraction in TRWP contained in soil or sediment materials is quantified in this document by direct pyrolysis-GC/MS (gas chromatography/mass spectrometry) analysis. Mass concentration can be expressed on the basis of the rubber polymer, tyre tread, or TRWP. This method has been used to measure the TRWP concentration in soil and sediment samples (see References [2], [3], and [4]). The airborne concentration of TRWP in the $PM_{2.5}$ and PM_{10} fraction has also been characterized by a similar method (see References [5], [6], and [14]).

Specific chemical markers are generated from intact TRWP by pyrolysis of sample specimens. The chemical markers consist of characteristic and specific pyrolysis dimeric fragments of passenger and truck tyre tread polymers including butadiene rubber, styrene-butadiene rubber, and isoprene rubber. The polymer fragments generated by sample pyrolysis are subsequently separated by gas chromatography (GC) and identified by mass spectrometry (MS). The TRWP mass concentration is calculated based on market average polymer use rates in tread, and prior characterization of the mineral content of TRWP. Rubber polymer specificity is achieved by quantification of dimeric polymer fragments consisting of two monomer units (see References [7], and [8]). Repeatability is achieved by the use of a deuterated internal standard of similar polymeric structure to the tyre tread polymers. The internal standard corrects for variable analyte recovery caused by sample size, matrix effects, and temporal variation in instrument response. The method is suitable for monitoring changes in soil or sediment TRWP concentrations over time.

NOTE Tyre and road wear particles are a discrete mass of elongated particles generated at the frictional interface between the tyre and roadway surface during the service life of a tyre. The particles consist of tyre tread enriched with mineral encrustations from the roadway surface.

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Rubber — Determination of the mass concentration of tyre and road wear particles (TRWP) in soil and sediment — Pyrolysis-gas chromatography/mass spectrometry (GC/MS) method

WARNING 1 — Persons using this document should be familiar with normal laboratory practice. This document 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.

WARNING 2 — Certain procedures specified in this document can involve the use or generation of substances, or the generation of waste, that can constitute a local environmental hazard. Reference should be made to appropriate documentation on safe handling and disposal after use.

1 Scope

This document specifies a method for the determination of the mass concentration (µg/g) of tyre and road wear particles (TRWP) in soil or sediment environmental samples.

This document establishes principles for soil or sediment sample collection, the generation of pyrolysis fragments from the sample, and the quantification of the generated polymer fragments.

Sample Document

This document is applicable to the environmental samples from soil or sediment.

This document is not applicable to the environmental samples from ambient air and ambient water.

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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 17257, *Rubber — Identification of polymers — Pyrolytic gas-chromatographic method using mass-spectrometric detection*

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

3 Terms and definitions

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

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

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

3.1
deuterated internal standard
polymer made of monomer containing at least one deuterium molecule added to a sample in a fixed amount that is nearly identical to the target analyte used to correct for instrument drift and matrix interference

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3.2
dry mass
mass of solid dried in an oven for a specified time and at a specified temperature

3.3
wet mass
mass of solid before drying in an oven

3.4
monitoring
repeated measurement to follow changes over a period of time

3.5
per cent dry mass
dry mass (3.2) of sample solids expressed as a percentage of the sample *wet mass* (3.3)

3.6
pyrolysis analysis
decomposition of organic polymeric molecules into characteristic fragments separated by gas chromatography (GC) and quantified by mass spectrometry (MS)

4 Principle

Tyre tread polymer is quantified using internal standard calibration and the peak area of characteristic fragment ions corresponding to dimers of the raw polymer by pyrolysis-GC/MS. The thermal decomposition products of cross-linked natural rubber (NR) (CAS Registry Number®[1)] 9006-04-6), styrene-butadiene rubber (SBR) (CAS RN® 9003-55-8) and butadiene rubber (BR) (CAS RN® 9003-17-2) polymers depend on the abundance of polymers in the sample. SBR pyrolysis generates butadiene (CAS RN® 106-99-0), 4-vinylcyclohexene (butadiene dimer;VCH) (CAS RN® 100-40-3), styrene (CAS RN® 100-42-5), and 4-phenylcyclohexene (PCH) (CAS RN® 4994-16-5), whereas BR generates butadiene monomer and VCH (see Reference [9]). NR is associated with isoprene (CAS RN® 78-79-5) monomer and dipentene (isoprene dimer;DP) (CAS RN® 138-86-3). Synthetic poly(1,4-isoprene) rubber (IR) (CAS RN® 9003-31-0), a structural analogue of NR, also yields dipentene upon pyrolysis, and is used in calibration standards. Quantification of the synthetic fraction (SBR and BR) by the VCH marker is preferred based on the increased response ratio and contribution as compared to the PCH marker, which estimates the synthetic fraction based on SBR only (see Reference [11]).

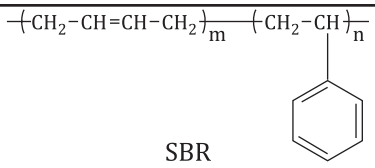
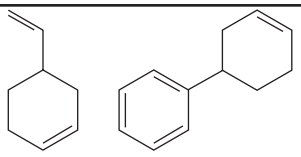
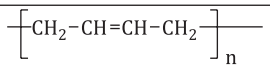
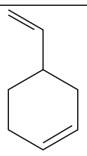
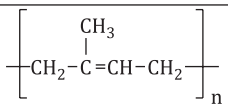
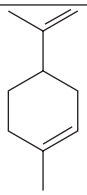
The dimer fragments have good specificity for rubber polymers, whereas both anthropogenic and natural organic substances are sources of the monomer markers (see Reference [7]). Therefore, the monomeric pyrolysis marker compounds are subject to interference from non-TRWP environmental sources, and are not suitable for quantification of TRWP mass concentration in soil or sediment. One well-known example is styrene, which is generated from pyrolysis of both SBR and diesel exhaust particles (see Reference [10]). The tyre polymers and pyrolysis fragment dimers used for quantification of TRWP are shown in Table 1.

1) CAS Registry Number® is a trademark of the American Chemical Society (ACS). This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

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Table 1 — Dimeric pyrolysis products of tyre rubber polymer

Polymer formula	Dimer
 SBR	 VCH PCH
 BR	 VCH
 NR	 DP

5 Reagents and materials

Use only reagents of recognized analytical grade.

5.1 Chloroform, analytical grade (non-deuterated CAS RN® 67-66-3 or deuterated CAS RN® 865-49-6).

5.2 Helium, purity 99,999 5 % by volume. (CAS RN® 7440-59-7).

5.3 Potassium hydroxide 10 % (100 g/l), purity >86 % by volume. (CAS RN® 1310-58-3).

5.4 Purified water, Type I ultrapure.

5.5 Deuterated poly(1,4-isoprene) (d-IR), for internal standard.

5.6 Deuterated polystyrene polybutadiene (d-PSPB), random copolymer for internal standard.

5.7 Poly(1,4-isoprene) rubber (IR) (CAS RN® 9003-31-0), random copolymer for calibration polymer.

5.8 Styrene-butadiene rubber (SBR), for calibration polymer.

6 Apparatus

6.1 Soil or sediment sampling — Equipment and consumable supplies

6.1.1 Sampling device

The sampling device shall be suitable for the collection of soil or sediment samples. Suitable devices include pre-cleaned stainless steel hand trowels, coring tools, or clamshell dredge devices. The collection of a sediment sample with a clamshell dredge device is illustrated in Figure 1.

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