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Rubber — Framework for physical and chemical characterization of tyre and road wear particles (TRWP)

Caoutchouc — Lignes directrices pour la caractérisation physique et chimique des particules émises par l'usure des pneumatiques et de la route (TRWP)

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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 documents 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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For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www. iso. org/iso/foreword. html.

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

Introduction

This document is the second in a series that provides guidance pertaining to tyre and road wear particles (TRWP). The first document, ISO/TS 22638, specifies how to generate TRWP that are to be used for future analysis such as physical and chemical characterization. This document provides a framework of existing international standards to perform such analysis on TRWP.

TRWP are formed from the friction between a tyre and roadway surface. The particles are subsequently released into nearby soil and sediment ecosystems. As such, there is interest in studying the composition of TRWP in the environment (Kreider et al. 2010; Unice et al. 2015). Characteristics of TRWP are likely to differ from that of the manufactured tread due to the chemical and mechanical alterations during driving.

The document describes testing strategies and considerations for assessing the physical and chemical properties of interest in TRWP. Specifically, guidance is provided on how to qualitatively and quantitatively assess physical properties including morphology and particle size distribution, as well as determining the chemical characteristics such as general composition, metallic content, tyre element content and PAH content. Knowing the physical and chemical properties of TRWP can assist in future analysis regarding the environmental fate and toxicity of the particles.

Rubber — Framework for physical and chemical characterization of tyre and road wear particles (TRWP)

1 Scope

This document provides a framework for the characterization of physical and chemical properties of tyre and road wear particles (TRWP) using published analytical standards. It is applicable to laboratory-generated TRWP and TRWP collected in the environment.

NOTE This framework focuses primarily on published ISO standards, but includes standards published by other entities such as ASTM and AFNOR. A brief summary and justification for each standard required to characterize the physical and chemical properties of interest are provided.

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*

ISO 9924-3, *Rubber and rubber products — Determination of the composition of vulcanizates and uncured compounds by thermogravimetry — Part 3: Hydrocarbon rubbers, halogenated rubbers and polysiloxane rubbers after extraction*

ISO 11885, *Water quality — Determination of selected elements by inductively coupled plasma optical emission spectrometry (ICP-OES)*

ISO 13320, *Particle size analysis — Laser diffraction methods*

ISO 14869-3, *Soil quality — Dissolution for the determination of total element content — Part 3: Dissolution with hydrofluoric, hydrochloric and nitric acids using pressurised microwave technique*

3 Terms and definitions

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 https://www.electropedia.org/

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

3.1
tyre and road wear particles
TRWP
discrete mass of elongated particles generated at the frictional interface between the road and the pavement surface during the service life of a tyre

Note 1 to entry: The particles consist of tyre tread enriched with mineral encrustations from the roadway surface.

4 Physical characterization strategies

4.1 General

TRWP, whether generated naturally or in a laboratory setting, inherently have different physical properties than naïve tyre rubber because of the mechanical process in which they are created. Physical characteristics of interest for TRWP include morphology and particle size distribution.

4.2 Morphology

The following physical characteristics shall be determined to qualitatively and quantitatively assess TRWP morphology:

— shape;

— elongation/aspect ratio; and

— circularity.

Scanning electron microscopy (SEM) shall be used to qualitatively assess the morphology of TRWP. SEM images show the deposited mineral encrustations and shape of TRWP.

Transmission optical microscopy shall be used to quantitatively assess the elongation and circularity of TRWP. Elongation is determined from the particle aspect ratio, or the ratio of length to width.

4.3 Particle size

Particle size distribution shall be determined using transmission optical microscopy and laser diffraction following ISO 13320. Transmission optical microscopy should be capable of quantifying the number and volume of particles used in the laser diffraction method. ISO 13320 provides guidance on instrumentation and size distribution measurement of particles ranging from 0,1 µm to 3 mm in a two-phase system. Special instructions are provided for particles measuring outside of this size range.

5 Chemical characterization strategies

5.1 General

Although the chemicals used in the manufacture of tyre compound are known, the chemical composition will differ throughout the service life of a tyre. TRWP include pavement components that vary from the chemical composition of tyre compound. The chemical content of TRWP is of interest to assess potential toxicity of particles to aquatic and sediment ecosystems. Four chemical composition groups to investigate consists of the:

— general composition;

— metallic content;

— tyre element content; and

— PAH content of TRWP.

Existing methods published by ISO, ASTM, DIN and AFNOR are available to sufficiently characterize these chemical compositions in TRWP. See Bibliography.

5.2 General composition

General composition specifies the general chemical classes comprised in TRWP. These shall include the following chemical classes:

— plasticizers and oils;

— polymers;

— carbon black; and

— mineral filler.

ISO 1407 shall be used in combination with ISO 9924-3 to determine the general composition of TRWP. Specifically, ISO 1407 shall be used to determine the appropriate solvent extraction method and ISO 9924-3 shall be used to determine the general composition using thermogravimetry.

NF T 46-047 may be used as an alternative to ISO 9924-3.

Provided in ISO 1407 are four quantitative methods to determine the material extractable from natural and/or synthetic raw rubbers. Recommended solvents are provided for the vulcanized and unvulcanized rubber for each rubber family as given in ISO 1407.

Following preliminary extraction, ISO 9924-3 shall be used to quantify the chemical classes of TRWP. This method is applicable to rubbers with a hydrocarbon, halogenated hydrocarbon, and polysiloxane backbones.

5.3 Metallic content

ASTM D1971 provides a comprehensive framework to determine the metallic content of TRWP by digestion of samples prior to atomic absorption spectrophotometry, plasma emission spectrometry, or plasma mass spectrometry. Three sample digestion methods are provided in the guidance.

NOTE Analytical ASTM methods referenced in the study include D1976, D3919, D4691 and D4190 (atomic absorption spectrophotometry, plasma emission spectrometry) and D5673 (plasma mass spectrometry).

5.4 Tyre element content

Elements commonly associated with tyre formulation include silicon, sulfur, and zinc. To measure the concentrations of these three elements in TRWP, ISO 14869-3 and ISO 11885 shall be used in combination. Specifically, ISO 14869-3 shall be used to digest TRWP samples and ISO 11885 shall be used to determine the elemental content.

ISO 14869-3 provides guidance on microwave assisted digestion of a sample with a mixture of hydrofluoric acid (HF), nitric (HNO_3) and hydrochloric (HCl) acids. The subsequent solution is acceptable for use in inductively coupled plasma optical emission spectrometry analysis as described in ISO 11885.

5.5 PAH content

PAHs may be found in trace amounts in oils used in tyre formulation. French standard XP X 33-012 and German Standard AFPS GS 2014 01 PAK specifies methods for the extraction and determination of 16 PAH compounds. Provided in the standard are three extraction methods using hexane/acetone mixture. PAHs are quantified in the extract using one of two chromatography methods described. ISO 21461 characterizes total aromaticity in rubber products through extraction and NMR spectroscopy. This method can be used in lieu of XP X33-012 to evaluate total aromaticity, rather than determining specific PAH compound concentrations.

6 Test report

The test report shall include at least the following information:

a) all information necessary for identifying the TRWP sample;

b) reference to this document (i.e ISO/TS 22640);

c) date and time of testing;

d) identity of analyst;

e) equipment and test conditions;

f) description of chemicals, standards, and reagents;

g) test results obtained;

h) any anomalies that occurred during the tests;

i) any additional information mandated by the standard referenced in this framework.

Bibliography

[1] KREIDER M. L., PANKO J. M., MCATEE B. L., SWEET L. I., FINLEY B. L. Physical and chemical characterization of tire-related particles: Comparison of particles generated using different methodologies. Sci. Total Environ. 2010, **408** (3), 652–9

[2] UNICE K. M., BARE J. L., KREIDER M. L., PANKO J. M. Experimental methodology for assessing the environmental fate of organic chemicals in polymer matrices using column leaching studies and *OECD 308* water/sediment systems: Application to tire and road wear particles. Sci. Total Environ. 2015, **533**, 476-87

[3] NF T 46 047:2014, Rubber and rubber products - Determination of composition of vulcanizates and uncured compounds by thermogravimetry

[4] ISO/TS 22638, *Rubber — Generation and collection of tyre and road wear particles (TRWP) — Road simulator laboratory method*

[5] XP X 33-012, Characterization of sludges —Determination of polynuclear aromatic hydrocarbons (PAH) and polychlorinated biphenyls (PCB)

Relevant standards for the determination of particle size

[6] ASTM E2651-13 *Standard Guide for Powder Particle Size Analysis*

[7] JIS Z 8825:2013, *Particle Size Analysis — Laser diffraction methods*

Relevant standards for the determination of rubber and rubber products composition

[8] ASTM D5774-95*Standard test methods for rubber — Chemical analysis of extractables*

[9] JIS K 6229:2015, *Rubber — Determination of solvent extract*

[10] ASTM D6370-99*Standard Test Method for Rubber — Compositional Analysis by Thermogravimetry (TGA)*

[11] ASTM D297-15*Standard Test Methods for Rubber Products — Chemical Analysis*

[12] JIS K 6226-1:2003, *Rubber and rubber products — Determination of the composition of vulcanizates and uncured compounds by thermogravimetry — Part 1: Butadiene, ethylene-propylene copolymer and terpolymer, isobutene-isoprene, isoprene and styrene-butadiene rubbers*

[13] JIS K 6226-2:2003, *Rubber and rubber products — Determination of the composition of vulcanizates and uncured compounds by thermogravimetry — Part 2: Acrylonitrile-butadiene and halobutyl rubbers*

Relevant standards for the determination of metallic content

[14] ISO 15587-1:2002, *Water quality — Digestion for the determination of selected elements in water — Part 1: Aqua regia digestion*

[15] ISO 15587-2:2002, *Water quality — Digestion for the determination of selected elements in water — Part 2: Nitric acid digestion*

[16] ISO 8288:1986, *Water quality — Determination of cobalt, nickel, copper, zinc, cadmium and lead — Flame atomic absorption spectrometric methods*

[17] ISO 9174:1998, *Water quality — Determination of chromium — Atomic absorption spectrometric methods*

[18] ISO 12020:1997, *Water quality — Determination of aluminium — Atomic absorption spectrometric methods*

[19] ISO 15586:2003, *Water quality — Determination of trace elements using atomic absorption spectrometry with graphite furnace*

[20] ISO 17294-2:2016, *Water quality — Application of inductively coupled plasma mass spectrometry (ICP-MS) — Part 2: Determination of selected elements including uranium isotopes*

[21] DIN 38405-1, *German standard methods for the examination of water, waste water and sludge; anions (group D); determination of chloride ions (D1)*

[22] DIN 38405-4, *German standard methods for the examination of water, waste water and sludge; anions (group D); determination of fluoride (D4)*

[23] DIN 38405-5, *German standard methods for the examination of water, waste water and sludge; anions (group D); determination of sulphate ions (D5)*

[24] DIN 38405-7, *German standard methods for the examination of water, waste water and sludge; anions (group D); Part 7: Determination of cyanides in low polluted water by liquid chromatography of ions or potentiometric titration (D 7)*

[25] DIN 38405-9, *German standard methods for the examination of water, waste water and sludge; anions (group D); Part 9: Spectrometric determination of nitrate (D 9)*

[26] DIN 38405-13, *German standard methods for the examination of water, waste water and sludge; anions (group D); Part 13: Determination of cyanides (D 13)*

[27] DIN 38405-17, *German standard methods for the examination of water, waste water and sludge; anions (group D); Determination of Borate Ions (D 17)*

[28] DIN 38405-21, *German standard methods for the examination of water, waste water and sludge; anions (group D); determination of dissolved silicate by spectrometry (D 21)*

[29] DIN 38405-23, *German standard methods for the examination of water, waste water and sludge; anions (group D); Part 23: Determination of selenium by atomic absorption spectrometry (D 23)*

[30] DIN 38405-24, *German standard methods for the examination of water, waste water and sludge; anions (group D); photometric determination of chromium (VI) using 1.5-diphenylcarbonohydrazide (D 24)*

[31] DIN 38405-26, *German standard methods for the examination of water, waste water and sludge; anions (group D); Part 26: Determination of dissolved sulfide by spectrometry (D26)*

[32] DIN 38405-27, *German standard methods for the examination of water, waste water and sludge; anions (group D); Part 27: Determination of sulfide by gas extraction method (D 27)*

[33] DIN 38405-29, *German standard methods for the examination of water, waste water and sludge; anions (group D); Part 29: Spectrometric determination of nitrate with sulfosalicylic acid (D 29); ISO 7890-3:1988, modified*

[34] DIN 38405-32, *German standard methods for the examination of water, waste water and sludge; anions (group D); Part 32: Determination of antimony by atomic absorption spectrometry (D 32)*

[35] DIN 38405-33, *German standard methods for the examination of water, waste water and sludge; anions (group D); Part 33: Determination of iodide by spectrometry (D 33)*

[36] DIN 38405-35, *German standard methods for the examination of water, waste water and sludge; anions (group D); Part 35: Determination of arsenic - Method by graphite furnace atomic absorption spectrometry (GF-AAS) (D 35)*

[37] DIN 38406-1, *German standard methods for the examination of water, waste water and sludge; cations (group E); determination of iron (E1)*

[38] DIN 38406-2, *German standard methods for the examination of water, waste water and sludge; cations (group E); determination of manganese (E 2)*

