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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

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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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For an explanation of 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 www.iso.org/iso/foreword.html.

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

This first edition cancels and replaces the first edition Technical Specification (ISO/TS 22640:2018), which has been technically revised.

The main changes are as follows:

— an error in definition 3.1 has been corrected;

— the references have been updated.

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.

Introduction

This document is one of a family of standards that provides guidance on tyre and road wear particles (TRWP). Another document, ISO 22638[1)], 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 (see References [1] and [2]). Characteristics of TRWP are likely to differ from that of the manufactured tread due to the chemical and mechanical alterations during driving.

This 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 polycyclic aromatic hydrocarbons (PAH) content. Knowing the physical and chemical properties of TRWP can assist in future analysis regarding the environmental fate and toxicity of the particles.

1) Under preparation. Stage at the time of publication: ISO/DIS 22638:2023.

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

1 Scope

This document establishes 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 International Standards, but also 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 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
tyre and road wear particles
TRWP
discrete mass of elongated particles generated at the frictional interface between the tyre 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 (see 4.2) and particle size distribution (see 4.3).

4.2 Morphology

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

— shape;

— elongation/aspect ratio;

— 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 distribution

Particle size distribution shall be determined using transmission optical microscopy and laser diffraction in accordance with 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 are:

— general composition;

— metallic content;

— tyre element content;

— PAH content of TRWP.

Existing methods published by ISO, ASTM, DIN and AFNOR are available to sufficiently characterize these chemical compositions in TRWP, see the 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;

— mineral filler.

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

NF T46-047 may be used as an alternative to ISO 9924-3.

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

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

5.3 Metallic content

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 is provided in ASTM D1971. Three sample digestion methods are provided in the guidance.

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

5.4 Tyre element content

Elements commonly associated with tyre formulation include silicon, sulfur and zinc. The concentrations of these three elements in TRWP shall be measured in accordance with ISO 14869-3 and ISO 11885 in combination. Specifically, TRWP samples shall be digested in accordance with ISO 14869-3 and the elemental content shall be determined in accordance with ISO 11885.

Guidance on microwave assisted digestion of a sample with a mixture of hydrofluoric acid (HF), nitric (HNO_3) and hydrochloric (HCl) acids is provided in ISO 14869-3. 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 can be found in trace amounts in oils used in tyre formulation. German document AfPS GS 2019:01 PAK specifies methods for the extraction and determination of 16 PAH compounds. Three extraction methods using hexane/acetone mixture are specified. PAHs are quantified in the extract using one of two chromatography methods described. Total aromaticity in rubber products through extraction and NMR spectroscopy are characterized in ISO 21461. This method can be used in lieu of AfPS GS 2019:01 PAK 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 22640:2023;

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) pp. 652–659

[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** pp. 476–487

[3] NF T46-047, *Rubber and rubber products - Determination of composition of vulcanizates and uncured compounds by thermogravimetry*

[4] ISO 22638[2)], *Rubber — Generation and collection of tyre and road wear particles (TRWP) — Road simulator laboratory method*

Relevant standards for the determination of particle size

[5] ASTM E2651, *Standard Guide for Powder Particle Size Analysis*

[6] JIS Z 8825, *Particle Size Analysis — Laser diffraction methods*

Relevant standards for the determination of rubber and rubber products composition

[7] ASTM D5774, *Standard Test Methods For Rubber — Chemical Chemical Analysis Of Extractables*

[8] JIS K 6229, *Rubber — Determination of solvent extract*

[9] ASTM D6370, *Standard Test Method for Rubber — Compositional Analysis by Thermogravimetry (TGA)*

[10] ASTM D297, *Standard Test Methods for Rubber Products — Chemical Analysis*

[11] JIS K 6226-1, *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*

[12] JIS K 6226-2, *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

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

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

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

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

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

2) Under preparation. Stage at the time of publication: ISO/DIS 22638:2023.

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

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

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

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

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

[23] 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)*

[24] 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)*

[25] 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)*

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

[27] 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)*

[28] 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)*

[29] 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)*

[30] 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)*

[31] 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)*

[32] 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*

[33] 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)*

[34] 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)*

[35] 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)*

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

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