
**Ambient air — Determination of the
mass concentration of tire and road
wear particles (TRWP) — Pyrolysis-
GC-MS method**

*Air ambient — Détermination de la concentration en masse de
particules provenant de l'usure des pneumatiques et des chaussées
(TRWP) — Méthode par pyrolyse-GC/SM*

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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 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 146, *Air quality*, Subcommittee SC 3, *Ambient atmospheres*.

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.[3] The elastomeric fraction in TRWP contained in PM_{2,5} or PM₁₀ is quantified in this document by direct pyrolysis-GC-MS analysis of a sample filter. Mass can be expressed on the basis of the rubber polymer, tyre tread, or TRWP. This method has been used to measure the airborne concentration of TRWP in the PM₁₀ fraction for three geographically separated regions.[4] The TRWP concentration in soil and sediment has also been characterized by a similar method.[5]

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 and identified by mass spectroscopy. 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.[6][7] 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 ambient air TRWP concentrations over a specified averaging time.

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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 and to ensure compliance with any national regulatory conditions.

WARNING 2 — Certain procedures specified in this document may involve the use or generation of substances, or the generation of waste, that could 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 airborne concentration ($\mu\text{g}/\text{m}^3$), mass concentration ($\mu\text{g}/\text{g}$) and mass fraction (%) of tyre and road wear particles (TRWP) in ambient particulate matter (PM) samples.

This document establishes principles for air sample collection, the generation of pyrolysis fragments from the sample, and the quantification of the generated polymer fragments. The quantified polymer mass is used to calculate the fraction of tyre tread in PM and concentration of tyre tread in air. These quantities are expressed on a TRWP basis, which includes the mass of tyre tread and mass of road wear encrustations, and can also be expressed on a tyre rubber polymer or tyre tread basis.

Air sample collection is on quartz fibre filters with size-selective input in a range of $\text{PM}_{2,5}$ or PM_{10} . The method is suitable for the determination of TRWP in indoor or outdoor atmospheres.

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 4225, *Air quality — General aspects — Vocabulary*

ISO 7708:1995, *Air quality — Particle size fraction definitions for health-related sampling*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 4225 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 <http://www.iso.org/obp>

3.1

ambient air

outdoor air to which people, plants, animals, or material may be exposed

3.2

averaging time

interval of time over which the air quality has been expressed as an average

3.3

continuous sampling

sampling, without interruptions, throughout an operation or for a predetermined time

3.4

deuterated compound

compound containing at least one *deuterium* (3.5) molecule

3.5

deuterium

minor and stable isotope of hydrogen with one proton and one neutron

3.6

internal standard

compound 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

3.7

measurement period

interval of time between first and last measurement

3.8

monitoring

repeated measurement to follow changes over a period of time

3.9

natural background concentration

concentration of a given species in a pristine air mass in which anthropogenic emissions are negligible

3.10

particle aerodynamic diameter

diameter of a sphere of density 1 g/cm³ with the same terminal velocity due to gravitational force in calm air as the particle, under the prevailing conditions of temperature, pressure, and relative humidity

3.11

particle

small discrete mass of solid or liquid matter

3.12

particulate matter — 2,5 µm

PM_{2,5}

airborne *particles* (3.11) passing a size selective inlet with 50 % efficiency cut-off at a *particle aerodynamic diameter* (3.10) of 2,5 µm

Note 1 to entry: See Thoracic Convention in ISO 7708:1995, Clause 6.

3.13

particulate matter — 10 µm

PM₁₀

airborne *particles* (3.11) passing a size selective inlet with 50 % efficiency cut-off at an aerodynamic diameter of 10 µm

Note 1 to entry: See High-Risk Respirable Convention in ISO 7708:1995, Clause 7.

3.14**pyrolysis analysis**

decomposition of organic polymeric molecules into characteristic fragments separated by gas chromatography and quantified by mass spectroscopy

Note 1 to entry: The principle of sample decomposition is the application of thermal energy to a sample encapsulated in a pyrolyser in the absence of oxygen. Secondary reactions are minimized by rapid heating of the pyrolyser to the target temperature.

3.15**sampling time**

interval of time over which a single sample is taken

3.16**tyre and road wear particles****TRWP**

discrete mass of elongated *particles* (3.11) generated at the frictional interface between the tread of the tyre and the roadway 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.

3.17**thoracic convention**

mass fraction of inhaled *particles* (3.11) which penetrate beyond the larynx

3.18**respirable convention**

target specification for sampling instruments when the respirable fraction is of interest

4 Symbols and abbreviated terms**4.1 Symbols of units (see also ISO 4226)**

µg	microgram (10 ⁻⁶ gram)
cm	centimeter (10 ⁻² meter)
m ³	cubic meter
cm ²	square centimeter
µg/m ³	microgram per cubic meter

4.2 Abbreviated terms

BdD	vinylcyclohexene (butadiene dimer)
d-BdD	deuterated butadiene dimer
d-IpD	deuterated isoprene dimer
d-PI	deuterated polyisoprene
d-PB	deuterated polybutadiene
BR	butadiene rubber
GC-MS	gas chromatograph/mass spectrometer