

---

---

**Rubber — Determination of mass concentration of tire and road wear particles (TRWP) in soil and sediments — Pyrolysis-GC/MS method**

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



This document is a preview generated by EMS



**COPYRIGHT PROTECTED DOCUMENT**

© ISO 2017, Published in Switzerland

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized otherwise in any form or by any means, electronic or mechanical, including photocopying, or posting on the internet or an intranet, without prior written permission. Permission can be requested from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office  
Ch. de Blandonnet 8 • CP 401  
CH-1214 Vernier, Geneva, Switzerland  
Tel. +41 22 749 01 11  
Fax +41 22 749 09 47  
copyright@iso.org  
www.iso.org

# Contents

	Page
<b>Foreword</b> .....	<b>iv</b>
<b>Introduction</b> .....	<b>v</b>
<b>1 Scope</b> .....	<b>1</b>
<b>2 Normative references</b> .....	<b>1</b>
<b>3 Terms and definitions</b> .....	<b>1</b>
<b>4 Principle</b> .....	<b>2</b>
<b>5 Reagents</b> .....	<b>2</b>
<b>6 Apparatus</b> .....	<b>3</b>
<b>7 Specimen preparation laboratory</b> .....	<b>5</b>
<b>8 Measuring range</b> .....	<b>5</b>
<b>9 Limit of detection</b> .....	<b>5</b>
<b>10 Procedure</b> .....	<b>6</b>
10.1 General.....	6
10.2 Sample collection.....	6
10.3 Deuterated internal standard preparation.....	6
10.4 Calibration curve preparation.....	7
10.4.1 Stock solutions.....	7
10.4.2 Calibration curves.....	7
10.5 Sample preparation.....	8
10.5.1 Oven drying.....	8
10.5.2 Sieving and homogenization.....	8
10.6 Sample measurement.....	8
10.6.1 Sample mass.....	8
10.6.2 Internal standard addition.....	9
10.6.3 Pyrolysis-gas chromatograph/mass spectrometer measurement.....	9
<b>11 Analysis</b> .....	<b>9</b>
11.1 TRWP detection limit.....	9
11.2 Quantity of tyre polymer in the sample.....	9
11.3 Mass concentration of TRWP.....	10
<b>12 Performance characteristics</b> .....	<b>10</b>
12.1 General.....	10
12.2 Specific performance characteristics.....	10
12.3 Method detection limit.....	10
<b>13 Test report</b> .....	<b>10</b>
<b>Annex A (informative) Recipe for calibration curves and stock solutions</b> .....	<b>12</b>
<b>Annex B (informative) Curie-point pyrolyser</b> .....	<b>14</b>
<b>Annex C (informative) Representative calibration curves and pyrograms</b> .....	<b>15</b>
<b>Annex D (normative) Calculation of TRWP detection limits</b> .....	<b>20</b>
<b>Annex E (normative) Calculation of results using dimer markers</b> .....	<b>21</b>
<b>Bibliography</b> .....	<b>23</b>

## 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](http://www.iso.org/directives)).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see [www.iso.org/patents](http://www.iso.org/patents)).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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](http://www.iso.org/iso/foreword.html).

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

## 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 (Kreider et al. 2010<sup>[1]</sup>). The elastomeric fraction in TRWP contained in soil or sediment materials is quantified in this document by direct pyrolysis-GC/MS 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 from three geographically separated regions (Unice et al. 2012<sup>[2]</sup>; Unice et al. 2013<sup>[3]</sup>). The airborne concentration of TRWP in the PM<sub>10</sub> fraction has also been characterized by a similar method (Panko et al. 2013<sup>[4]</sup>).

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 (Kitamura et al. 2007<sup>[5]</sup>; Harada et al. 2009<sup>[6]</sup>). 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.



# Rubber — Determination of mass concentration of tyre and road wear particles (TRWP) in soil and sediments — Pyrolysis-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 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 soil or sediment mass concentration ( $\mu\text{g/g}$ ) of tyre and road wear particles (TRWP) in 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. The quantified polymer mass is used to calculate the concentration of TRWP in soil or sediment. 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.

**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.

## 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 7270-1, *Rubber — Analysis by pyrolytic gas-chromatographic methods — Part 1: Identification of polymers (single polymers and polymer blends)*

## 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 <http://www.electropedia.org/>
- ISO Online browsing platform: available at <https://www.iso.org/obp>

### 3.1

#### **deuterated internal standard**

compound 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

### 3.2

#### **dry mass**

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