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**Rubber — Trapping and identification  
of volatile components of rubber  
fumes with active sampling on a  
poly(2,6-diphenylphenylene oxide)  
type sorbent, using thermodesorption  
and gas chromatographic method with  
mass spectrometric detection**

*Caoutchouc — Piégeage et identification des composés volatils des  
fumées de procédés du caoutchouc, par échantillonnage actif sur un  
sorbant de type poly(oxyde de 2,6-diphénylphénylène), en utilisant  
une méthode par thermodésorption et chromatographie en phase  
gazeuse avec détection par spectrométrie de masse*

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

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The committee responsible for this document is ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Testing and analysis*.

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# Rubber — Trapping and identification of volatile components of rubber fumes with active sampling on a poly(2,6-diphenylphenylene oxide) type sorbent, using thermodesorption and gas chromatographic method with mass spectrometric detection

## 1 Scope

This Technical Specification specifies a qualitative method of thermodesorption – gas chromatography – mass spectrometry (TD-GC-MS) for the identification of volatile components in rubber fumes, after trapping on a solid sorbent based on 2,6-diphenylphenylene-oxide polymer resin. It is applicable to a screening of emissions from the processing of rubber compounds in the ambient workplace and storage environment.

**CAUTION — Persons using this Technical Specification should be familiar with the procedures for gas chromatography – mass spectrometry measurement and analysis. All the operative details for the application and set-up of the GC-MS are assumed to be in agreement with the operative instructions provided by the manufacturer. Therefore, the detailed procedure for the operation is not included in this Technical Specification. This Technical Specification specifies a qualitative method which is not aimed at quantitative analyses.**

## 2 Terms and definitions

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

### 2.1

#### **semi-volatile organic compound**

##### **SVOC**

organic compound whose boiling point is in the range from (240 to 260) °C to (380 to 400) °C

Note 1 to entry: This classification has been defined by the World Health Organization.[4]

Note 2 to entry: Boiling points of some compounds are difficult or impossible to determine because they decompose before they boil at atmospheric pressure. Vapour pressure is another criterion for the classification of compound volatility that may be used for the classification of organic chemicals. SVOCs have vapour pressures of between  $10^{-2}$  kPa and  $10^{-8}$  kPa.

### 2.2

#### **volatile organic compound**

##### **VOC**

organic compound whose boiling point is in the range from (50 to 100) °C to (240 to 260) °C

Note 1 to entry: This classification has been defined by the World Health Organization.[4]

Note 2 to entry: Boiling points of some compounds are difficult or impossible to determine because they decompose before they boil at atmospheric pressure. Vapour pressure is another criterion for the classification of compound volatility that may be used for the classification of organic chemicals. VOCs generally have saturation vapour pressures at 25 °C greater than  $10^2$  kPa.

### 2.3

#### **very volatile organic compound**

##### **VVOC**

organic compound whose boiling point is in the range from <0 °C to (50 to 100) °C

Note 1 to entry: This classification has been defined by the World Health Organization.[4]

Note 2 to entry: Boiling points of some compounds are difficult or impossible to determine because they decompose before they boil at atmospheric pressure. Vapour pressure is another criterion for the classification of compound volatility that may be used for the classification of organic chemicals. VVOCs typically have vapour pressures of greater than 15 kPa.

### 3 Principle

Rubber fumes are sampled on an adsorbent support using a pump. They are recovered from the trap by thermal desorption and the substances composing the desorbed fume are identified by the mass spectrometer. The method identifies the components adsorbed on the trap support used, except benzene.

The actual composition of the emissions depends on the selection of ingredients used for compounding and on the thermal and mechanical conditions applied to the rubber. Moreover, environmental humidity might interfere with the sorption capability of the sorbent material.

The sorbent tube is used for the trapping of volatile (VOC) (boiling point  $>50\text{ }^{\circ}\text{C}$  to  $100\text{ }^{\circ}\text{C}$ ) and semi-volatile (SVOC) (boiling point  $>240\text{ }^{\circ}\text{C}$ ) organic compounds in the C6 to C26 range, which are chemically stable against a desorption temperature of  $200\text{ }^{\circ}\text{C}$ . Very volatile compounds (VVOC) (boiling point approximately  $50\text{ }^{\circ}\text{C}$  to  $100\text{ }^{\circ}\text{C}$ ) are only partially retained by the sorbent. Other sorbents based on carbon molecular sieve or by multi-sorbent bed tube may be more appropriate in this case.

The upper limit of the useful range is set by the sorptive capacity of the sorbent used and by the linear dynamic range of the gas chromatograph column and detector or by the sample-splitting capability of the analytical instrumentation used. The sorptive capacity is measured as a breakthrough volume of air, which determines the maximum air volume that shall not be exceeded when sampling.

NOTE Small amounts of benzene could be created by the thermal decomposition of the sorbent.

### 4 Sampling

#### 4.1 Equipment

**4.1.1 Trap support**, poly(2,6-diphenylphenylene oxide)<sup>1)</sup>, of quantity 180 mg to 200 mg, of particle size 0,18 mm to 0,25 mm (60/80 mesh), and of specific surface  $20\text{ m}^2/\text{g}$  to  $35\text{ m}^2/\text{g}$ . Another quantity, particle size or specific surface may be chosen if the test result is proven to be equivalent.

**4.1.2 Adsorbent tubes**, stainless steel tube.

#### 4.1.3 Calibrated pump

Calibrate the pump with the sorbent tube assembly inline, using a calibrated external flowmeter.

One end of the calibrated flowmeter shall be kept at atmospheric pressure to ensure proper operation.

#### 4.2 Operating conditions

##### 4.2.1 Trap support

Recondition the trap sorbent material before sampling, heating it at  $300\text{ }^{\circ}\text{C}$  under inert gas for 1 h (minimum) to 8 h (maximum). Check the cleaning of the trap support by GC-MS analysis.

Recondition tubes stored for more than four weeks before sampling.

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1) One example of poly(2,6-diphenylphenylene oxide) is Tenax TA®, which is an example of a suitable product available commercially. This information is given for the convenience of users of this Technical Specification and does not constitute an endorsement by ISO of this product.