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SIST EN 13999-2:2014

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EUROPEAN STANDARD  
NORME EUROPÉENNE  
EUROPÄISCHE NORM

**EN 13999-2**

December 2013

ICS 83.180

Supersedes EN 13999-2:2007

English Version

**Adhesives - Short term method for measuring the emission  
properties of low-solvent or solvent-free adhesives after  
application - Part 2: Determination of volatile organic compounds**

Adhésifs - Méthode de mesurage rapide des  
caractéristiques émissives des adhésifs à teneur faible ou  
nulle en solvants après application - Partie 2: Dosage des  
composés organiques volatils

Klebstoffe - Kurzzeit-Verfahren zum Messen der  
Emissionseigenschaften von lösemittelfreien oder  
lösemittelfreien Klebstoffen nach der Applikation - Teil 2:  
Bestimmung flüchtiger organischer Verbindungen

This European Standard was approved by CEN on 19 October 2013.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the CEN-CENELEC Management Centre or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the CEN-CENELEC Management Centre has the same status as the official versions.

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EUROPEAN COMMITTEE FOR STANDARDIZATION  
COMITÉ EUROPÉEN DE NORMALISATION  
EUROPÄISCHES KOMITEE FÜR NORMUNG

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## Foreword

This document (EN 13999-2:2013) has been prepared by Technical Committee CEN/TC 193 “Adhesives”, the secretariat of which is held by AENOR.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by June 2014, and conflicting national standards shall be withdrawn at the latest by June 2014.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This document supersedes EN 13999-2:2007.

EN 13999-2:2013 includes the following significant technical changes with respect to EN 13999-2:2007:

- a) change in the calculation of total VOC;
- b) harmonization of the testing conditions to EN ISO 16000-9.

EN 13999, under the general title *Adhesives — Short-term method for measuring the emission properties of low-solvent or solvent-free adhesives after application*, consists of the following parts:

- *Part 1: General procedure*
- *Part 2: Determination of volatile organic compounds*
- *Part 3: Determination of volatile aldehydes*
- *Part 4: Determination of volatile diisocyanates*

**SAFETY STATEMENT** — Persons using this document should be familiar with the normal laboratory practice, if applicable. 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 regulatory conditions.

**ENVIRONMENTAL STATEMENT** — It is understood that some of the material permitted in this European Standard may have negative environmental impact. As technological advantages lead to acceptable alternatives for these materials, they will be eliminated from this European Standard to the extent possible.

At the end of the test, the user of this European Standard should take care to carry out an appropriate disposal of the wastes, according to local regulation.

According to the CEN-CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

**EN 13999-2:2013 (E)****1 Scope**

This European Standard specifies a method for the determination of single volatile organic compounds (VOC) and of the total amount of volatile organic compounds (TVOC<sub>EN 13999</sub>) in the exhaust air of an emission test chamber after application of a low-solvent or solvent-free adhesive as defined in EN 923:2005+A1:2008. The method is based on use of a solid sorbent with subsequent desorption and gas chromatographic analysis. The method is applicable to measurement of non-polar and slightly polar VOC.

**2 Normative references**

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN 1232, *Workplace atmospheres - Pumps for personal sampling of chemical agents - Requirements and test methods*

EN 13999-1, *Adhesives - Short term method for measuring the emission properties of low-solvent or solvent-free adhesives after application - Part 1: General procedure*

EN ISO 16000-9, *Indoor air - Part 9: Determination of the emission of volatile organic compounds from building products and furnishing - Emission test chamber method (ISO 16000-9)*

EN ISO 16017-1, *Indoor, ambient and workplace air - Sampling and analysis of volatile organic compounds by sorbent tube/thermal desorption/capillary gas chromatography - Part 1: Pumped sampling (ISO 16017-1)*

ISO 16000-6, *Indoor air - Part 6: Determination of volatile organic compounds in indoor and test chamber air by active sampling on Tenax TA® sorbent, thermal desorption and gas chromatography using MS or MS-FID*

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**3 Principle**

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A sufficient volume of test chamber air is drawn with an appropriate flow through a sorbent tube (or several tubes in series) containing a solid sorbent.

Volatile organic compounds (VOC) are retained by the sorbent tube, which is subsequently analysed in the laboratory. The VOC are desorbed from the sampling tube and transferred by a flow of inert carrier gas into a capillary gas chromatograph coupled to a mass spectrometric detector (MS) and to a flame ionisation detector (FID) or only to a mass spectrometric detector.

**4 Reagents and materials****4.1 Volatile organic compounds for calibration**

Compounds of gas chromatographic grade.

**4.2 Methanol**

Methanol of gas chromatographic grade.

### 4.3 Sorbent material

For general purpose, poly(2,6-diphenyl-p-phenylene oxide, particle size (0,18 to 0,25) mm i.e. (60 to 80) mesh (Tenax<sup>1)</sup> TA [1] or equivalent sorbent materials) shall be used.

For the determination of very volatile carcinogenic organic compounds (e.g. vinyl chloride), Carbo-pack X<sup>1)</sup> or equivalent sorbent materials as described in EN ISO 16017-1 shall be used.

### 4.4 Carrier gas

Helium, argon or nitrogen with a purity 99,999 % or better.

## 5 Apparatus

### 5.1 Sorbent tubes

Sorbent tubes made from stainless steel or glass that can be filled with at least 200 mg of Tenax TA sorbent (4.3) are used. For example, tubes with 6 mm external diameter, 5 mm internal diameter and 90 mm long fulfil the requirement and are used in many commercial thermal desorbing devices.

It is recommended to use unsilanised glass wool or other suitable mechanism, e.g. stainless steel frit to retain the sorbent in the tube and metal screw caps and polytetrafluoroethylene (PTFE) ferrules for closing the sorbent tube. Carbo-pack X (4.3) or equivalent tubes are filled as described in EN ISO 16017-1.

### 5.2 Sorbent tube unions

Two sorbent tubes may be connected in series during sampling with metal screw cap couplings with PTFE ferrules.

### 5.3 Syringes

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A precision 10 µl liquid syringe readable to 0,1 µl.

### 5.4 Sampling pump

The pump shall fulfil the requirements of EN 1232 or equivalent.

### 5.5 Tubing

PE [poly(ethylene)] or PTFE tubing of appropriate diameter to ensure a leak-proof fit to both pump and sample tube.

Sampling tubes shall not be used with plastic tubing other than PTFE upstream of the sorbent to avoid interference from contaminants.

### 5.6 Flow meter calibrator

Bubble meter or other appropriate suitable device for gas flow calibration.

### 5.7 Gas chromatograph

A capillary gas chromatograph coupled to a mass spectrometric detector and to a flame ionisation detector or only to a mass spectrometric detector shall be used in accordance with EN ISO 16017-1 and ISO 16000-6.

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1) Carbo-pack X and Tenax TA® are examples of suitable products available commercially. This information is given for the convenience of users of this European Standard and does not constitute an endorsement by CEN of this product.

**EN 13999-2:2013 (E)****5.8 Thermal desorption apparatus**

Apparatus for two-stage thermal desorption of sorbent tubes and transfer of desorbed vapours via an inert gas flow into a gas chromatograph is required. A typical apparatus contains a mechanism for holding the tubes to be desorbed whilst they are heated and purged simultaneously with inert carrier gas. The desorption temperature and time is adjustable, as is the carrier gas flow rate. The apparatus may also incorporate additional features, such as automatic sample tube loading, leak testing, and a cold trap or another suitable device to concentrate the desorbed sample. The desorbed sample, contained in the purge gas, is routed to the gas chromatograph and capillary column via a heated transfer line.

**5.9 Injection facility for preparing standards by liquid spiking**

Use a commercially available unit for preparation of calibration standards or any other device allowing reproducible preparation of calibration standards.

**5.10 Emission test chamber**

An emission test chamber as described in EN 13999-1 or in EN ISO 16000-9 shall be used.

**6 Procedure****6.1 Preparation of sampling tubes****6.1.1 Preconditioning**

Use commercially pre-cleaned sampling tubes containing Tenax TA or sampling tubes that are preconditioned and filled in the laboratory as described for Tenax TA in this clause. For special purposes such as determination of specific carcinogenic volatile organic compounds as foreseen in EN 13999-1, other sorbent materials as described in EN ISO 16017-1 can be taken.

Remove trace impurities of manufactured Tenax TA before using it for VOC sampling. The success of the cleaning procedure is checked by performing analysis of the blank sorbent.

Tube blanks are acceptable if no individual interfering artefact peaks exceed 2 ng and if TVOC levels do not exceed 20 ng after subtraction of non-interfering VOC artefacts.

NOTE Usually keeping the tubes at a temperature of  $(320 \pm 10)$  °C for at least 3 h using carrier gas flow of 50 ml/min to 100 ml/min is sufficient.

Pack one end of the sampling tubes with unsilanised glass wool or stainless steel gauze. Weigh the appropriate amount of adsorbent. Transfer the adsorbent into the tube e.g. using suction. Prior to storage or conditioning seal the open end of the tube in the same manner as was done with the other end. To maintain the sorption capacity of the sampling tube, use no less than 200 mg of sorbent per tube.

**6.1.2 Conditioning**

Recondition the pre-cleaned sampling tubes prior to each use at temperature of  $(320 \pm 10)$  °C for 30 min (Tenax TA) under inert carrier gas flow of 50 ml/min to 100 ml/min to remove trace organic volatiles possibly trapped on the tube.

Analyse the tubes, using routine analytical parameters, to ensure that the thermal desorption blank is sufficiently small. If the blank is unacceptable, repeat the conditioning procedure.

The tube blank is acceptable if no individual interfering artefact peaks exceed 2 ng and if TVOC levels do not exceed 20 ng after subtraction of non-interfering VOC artefacts. If the blank is unacceptable, recondition the tubes by desorbing them for 10 min under a 50 ml/min to 100 ml/min flow rate of inert carrier gas at a



temperature  $\sim 20$  °C higher than that used for analysis and then repeat the analysis. If after repeated conditioning, the blank is still unacceptable, the tubes shall be re-packed.

It is recommended to seal the conditioned sorbent tubes with metal screw caps fitted with PTFE ferrules and store the tubes in an emission free container at room temperature. Conditioned sampling tubes should be used within two weeks.

Tubes stored for more than two weeks should be reconditioned before sampling. If other sealing material than PTFE/metal is used then the tubes should be used for sampling within 1 week.

## 6.2 Test chamber air sampling

Assemble the sampling line. Prepare the test chamber sampling port as described in EN 13999-1 or EN ISO 16000-9. If the sampling flow rate is determined with a calibrator, start the pump, note and record the sampling flow rate.

Connect the sampler to the chamber sampling port, note and record the time the sampler was connected. Never use more than 80 % of the test chamber supply air flow rate as the combined sampling flow rates. At the end of the sampling period, disconnect the sampler from the chamber sampling port, note and record the time of disconnection, repeat the sampling flow determination and turn off the pump.

Disconnect the sampling tube from the sampling line and seal both ends tightly.

The recommended sampling flow rate is in the range of 20 ml/min to 200 ml/min. Safe sampling volumes, i.e. air samples, shall be collected in duplicate, but using different pump flows such that two different volumes of air (such as 5 l and 2 l, or 5 l and 1 l) are collected simultaneously (or immediately sequentially).

## 6.3 Storage of loaded sampling tubes

Seal the loaded sampling tubes tightly using screw cap fittings with PTFE ferrules and store the tubes in an emission free container at ambient room temperature. If other sealing material than PTFE/metal is used, then analyse the tubes within two days maximum after sampling.

The effect of storage on loaded VOC from indoor or chamber air is not known, although certain experiences [2] suggest, that they may be stable over several months at room temperature. To avoid possible changes, the samples should be analysed as soon as possible (preferably within 2 weeks, but not later than 4 weeks).

## 6.4 Blank tubes

One or two of the conditioned tubes from the batch selected for each sampling exercise shall be retained as laboratory blank tubes. These are noted. They shall be analysed with the sampled tubes and shown to meet the performance specification detailed above (i.e.  $< 2$  ng of any individual interfering VOC and  $< 20$  ng total VOC after subtraction of non-interfering VOC artefacts). For blank control of the test chamber, see EN 13999-1.

## 6.5 Preparation of calibration blend solutions for liquid spiking

### 6.5.1 Solution containing approximately 10 mg/ml of each liquid component

Accurately weigh approximately 1 g of substance or substances of interest into a 100 ml volumetric flask, starting with the least volatile substance. Make up to 100 ml with methanol (4.2), stopper and shake to mix.

### 6.5.2 Solution containing approximately 1 000 µg/ml of each liquid component

Introduce 50 ml of methanol into a 100 ml volumetric flask. Add 10 ml of solution 6.5.1. Make up to 100 ml with methanol, stopper and shake to mix.