BS EN 13999-2:2013

BSI Standards Publication

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

National foreword

This British Standard is the UK implementation of EN 13999-2:2013. It supersedes BS EN 13999-2:2007 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee PRI/52, Adhesives.

A list of organizations represented on this committee can be obtained on request to its secretary.

ISBN 978 0 580 78121 6

ICS 83.180

This British Standard was published under the authority of the Standards Policy and Strategy Committee on 31 December 2013.

Amendments issued since publication

Date	Text affected

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 lösemittelarmen oder lösemittelfreien Klebstoffen nach der Applikation - Teil 2: Bestimmung flüchtiger organischer Verbindungen

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

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Ref. No. EN 13999-2:2013: E

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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of an identical text or by endorsement, at the latest by June 2014, and conflicting national standards shall be withdrawn at the latest by June 2014.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*

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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 ($TVOC_{EN\ 13999}$) 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*

3 Principle

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[1] TA [1] or equivalent sorbent materials) shall be used.

For the determination of very volatile carcinogenic organic compounds (e.g. vinyl chloride), Carbopack X [1] 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. Carbopack 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

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) Carbopack 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.

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 ± 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 ± 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 ~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.

6.5.3 Solution containing approximately 100 µg/ml of each liquid component

Accurately weigh approximately 10 mg 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.

6.5.4 Solution containing approximately 10 µg/ml of each liquid component

Introduce 50 ml of methanol into a 100 ml volumetric flask. Add 10 ml of solution 6.5.3. Make up to 100 ml with methanol, stopper and shake to mix.

6.5.5 Stability of calibration blend solutions

Prepare fresh standard solutions weekly or more frequently if evidence is noted of deterioration, e.g. condensation reactions between alcohols and ketones. Determine stability of the calibration solutions as part of the quality assurance protocol of the laboratory.

6.6 Preparation of calibration sorbent tubes spiked with standards

Prepare spiked sorbent tubes by injecting aliquots of standard solutions onto clean sorbent tubes as follows. Pass inert purge gas at 100 ml/min through the sorbent tube and inject a 1 µl to 5 µl aliquot of an appropriate standard solution. After appropriate purging, seal the tube. Prepare fresh standard tubes with each batch of samples.

6.7 Analysis

6.7.1 General

Desorb and analyse the VOC from the sampling tubes as soon as possible (preferably within 2 weeks, but not later than 4 weeks). Separate individual compounds on non-polar or slightly polar (5 % phenyl / 95 % poly methylsiloxane) capillary columns in a gas chromatograph, identify them with the mass spectrometric detector and quantify them using either the flame ionisation detector chromatogram, or the total ion current chromatogram taken from the mass spectrometric detector (if chromatographic separation is incomplete, use the selected ion chromatogram for quantification).

Analyse blank tubes, calibration standards and quality check standards in sequence with the samples.

For $\text{TVOC}_{\text{EN 13999}}$ determination, all volatile organic compounds (TVOC+TVVOC+TSVOC) appearing on a non-polar gas chromatographic column shall be included and calculated in accordance with 7.3.

NOTE TVOC = Total volatile organic compounds, TVVOC = Total Very volatile organic compounds and TSVOC = Total semi-volatile organic compounds.

6.7.2 Desorption

Select the desorption parameters (i.e. desorption time and desorption gas flow rate for thermal desorption) achieving desorption efficiency for n-octadecane better than 95 %, for more details see EN ISO 16017-1. Parameters shall allow analysis of any volatile organic compounds in the range of at least n-pentane (n-C_5) to eicosane (n-C_{20}).

NOTE Typical desorption conditions for VOC analysis using the secondary cold trap are:

- desorption temperature 200 °C to 320 °C,
- desorption time 5 min to 15 min,
- desorption gas flow rate 20 ml/min to 50 ml/min,
- cold trap high temperature 250 °C to 280 °C,

- cold trap low temperature −30 °C to 0 °C,
- cold trap sorbent Tenax TA,
- transfer line temperature 220 °C.

6.7.3 Gas chromatographic analysis

Determine the operation parameters of the gas chromatographic analysis by analysing standard solutions and tubes as well as sampled tubes. The parameters shall allow analysis of any volatile organic compounds in the range of at least n-pentane (n-C_5) to n-eicosane (n-C_{20}).

EXAMPLE As an example, the following conditions are suggested:

- column: poly (dimethyl-siloxane) 30 m × 0,25 mm × 1,0 µm or equivalent (poly(methylphenylsiloxane) containing 95 % methyl and 5 % phenyl groups);
- injector: splitless or split mode (split ratio from 100:1 to 10:1, depending on the measured concentration);
- carrier: helium 36 kPa;
- GC oven programme: 35 °C or 40 °C for 5 min, 5–10 °C/ min to 300 °C for 5 min to 10 min.

6.7.4 Calibration

Determine compound specific response factors and the linearity of FID/MS for compounds of interest by calibrating the analytical system with standard solutions (6.5) injected into the sampling tube (5.5). At least three-point calibration over the linear range shall be performed.

7 Identification and calculation

7.1 Identification of single VOC

7.1.1 General

For identification of single VOC, analyse the samples with mass spectrometric detector operating in the scan mode. Identify single VOC detected in the sample by comparing their mass spectra from the mass spectrometric detector total ion chromatogram with the mass spectra of pure standard compounds if available, or with commercially available compilations of mass spectra libraries. User generated libraries may also be used.

Assume a positive identification if both the mass spectrum in the chromatogram matches a standard spectrum and if the retention time corresponds with the retention time of the standard compound.

Assign identification classes as given in 7.1.2 to all compounds with test chamber concentrations at least above 2 µg/m^3 (when expressed in toluene equivalents).

7.1.2 Identification classes

CLASS 1: DEFINITELY IDENTIFIED

The mass spectrum and the retention time of the signal peak match those of a pure chemical standard.

CLASS 2: PROBABLY IDENTIFIED

The mass spectrum of the signal peak matches a reference spectrum of a standard library and the retention time is in the expected time interval, and there is additional information supporting the identity (such as knowledge of the composition of the tested product), but there is no pure standard available.

CLASS 3: POSSIBLY IDENTIFIED

The mass spectrum of the signal peak matches a reference spectrum of a standard library and the retention time is in the expected time interval but there is no additional information and no pure standard available.

CLASS 4: NOT IDENTIFIED

The mass spectrum of the signal peak does not match reference spectra of a standard library, or it matches with many reference spectra to a comparable degree, not indicating a clear decision.

7.2 Concentration of single VOC in the sampled air

Calculate the volume V_s, in litres, for each air sample. Calculate the quantity q, in µg, of any identified compound in the sample tubes using their individual response factor. Calculate the quantity q, in µg, of any unidentified compound in the sample tubes using the calibration response factor for toluene. Calculate the concentration C_m, in µg/m^3, of any compounds in the sampled chamber air correcting for blanks as follows:

$$C_m = (q_{sample} - q_{blank}) \times 1\,000 / V_s \qquad (1)$$

where

C_m	is the concentration of analyte in the air sample, in µg/m^3;
q_{sample}	is the quantity of analyte in the sample tubes (all tubes in series), in µg;
q_{blank}	is the quantity of analyte in the test chamber blank, in µg;
V_s	is the volume of air sampled, in litres.

NOTE In case of high-emitting samples, analyte loss by breakthrough can be avoided by air sampling onto 2 tubes connected in series. Then, the breakthrough B is calculated as:

$$B = q_{sample} \times 100 / (q_{sample} - q_{control}) \qquad (2)$$

where

B	is the breakthrough, in %;
q_{sample}	is the quantity of analyte in the sample tube, in µg;
$q_{control}$	is the quantity of analyte in the control tube, in µg.

In case of a breakthrough higher than 25 %, repeat the test with different air sampling conditions (see 6.2).

7.3 Calculation of TVOC, TVVOC, TSVOC and TVOC$_{EN\ 13999}$

Classify the analyte identification quality as described in 7.1.2. Then calculate the total amount of volatile organic compounds (TVOC$_{EN\ 13999}$) as follows:

a) consider all the compounds found in the chromatogram representing at least 2 µg/m^3 (when expressed as toluene equivalents);

b) quantify all peaks with the response factor of toluene. If MS is used for quantification as toluene equivalents, full-scan mode shall be used;

c) calculate the total of all compounds appearing on a non-polar or slightly polar gas chromatographic column between and inclusive n-hexane (n-C_6) and n-hexadecane (n-C_{16}) as TVOC. Calculate the total of all compounds before n-hexane (n-C_6) as TVVOC and the total of all compounds after n-hexadecane (n-C_{16}) as TSVOC. Calculate the sum of TVOC, TVVOC and TSVOC as the $TVOC_{EN\ 13999}$.

8 Precision and bias

Determine the repeatability and the accuracy of this method as part of method validation, preferably by sampling from known atmospheres of 10^{-12} level. If a laboratory has no access to such atmospheres, repeatability shall be determined by repeated sampling from a constant atmosphere. Accuracy can be determined by analysing commercially available control or reference samples.

NOTE The repeatability of sampling of non-polar hydrocarbons from cylinder atmospheres containing six VOC could be shown for Tenax TA to be better than ± 10 % for 2 l samples and better than ± 12 % for 0,5 l samples.

9 Test report

The test report shall include:

a) reference to this European Standard, i.e. EN 13999-2;

b) purpose of the measurement;

c) identification of the test specimen in the test chamber;

d) time and date of the sampling;

e) description of the sampling procedure, especially the sampling duration;

f) description of the analytical procedure;

g) detection limits of the analytical method;

h) concentrations of identified compounds provided with CAS-numbers including calculation and calibration principles used;

i) $TVOC_{EN\ 13999}$ concentration;

j) ratio of the total amount of identified single compounds to the $TVOC_{EN\ 13999}$;

k) operation not included in this European Standard or in the International Standard to which reference is made or regarded as optional.

10 Quality control

An appropriate level of quality control shall be employed as described in ISO 16000-6, including:

- preparation of blank samples by using tubes identical to those used for sampling and subject them to the same handling procedure as the sample tubes except for the actual period of sampling;
- controlling the collection efficiency and sample breakthrough by using and analysing back-up tubes or by taking samples of different sampling volumes;
- controlling desorption efficiency of VOC either by using internal standards or by second desorption of the sampling tube. To follow the stability of the response factors, a standard mixture of selected, representative compounds is analysed in sequence with the actual samples;

- determination of the repeatability of the measuring method. The relative standard deviation of ≤ 15 % should be reached. The recovery of n-octadecane shall be at least 95 %;
- tracing calibration to at least one of these;
- commercially available certified reference samples or certified calibration gases;
- self-produced dynamic calibration gases;
- primary standards being quantified by weighing.

It is strongly recommended that all laboratories undertaking the determination of volatile organic compounds in test chamber air should participate in an external quality assessment scheme and/or in laboratory intercomparisons.

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