
Adhézifs - Méthode de courte durée de mesurage des propriétés d'émission des adhésifs a faible teneur en solvants ou exempts de solvants apres application - Partie 2: Dosage des composés organiques volatils

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

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

Adhésifs - Méthode de courte durée de mesurage des propriétés d'émission des adhésifs a faible teneur en solvants ou exempts de solvants apres application - Partie 2: Dosage des composés organiques volatils

Ta slovenski standard je istoveten z: ENV 13999-2:2002

ICS:

83.180

Lepila

Adhesives

SIST ENV 13999-2:2003**en**

iTeh STANDARD PREVIEW
(standards.iteh.ai)

SIST ENV 13999-2:2003

<https://standards.iteh.ai/catalog/standards/sist/8c17058b-24b0-44b0-b1f4-8c874e5ccc05/sist-env-13999-2-2003>

ICS 83.180

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 courte durée de mesurage des propriétés d'émission des adhésifs à faible teneur en solvants ou exempts de 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 Prestandard (ENV) was approved by CEN on 16 October 2002 as a prospective standard for provisional application.

The period of validity of this ENV is limited initially to three years. After two years the members of CEN will be requested to submit their comments, particularly on the question whether the ENV can be converted into a European Standard.

CEN members are required to announce the existence of this ENV in the same way as for an EN and to make the ENV available promptly at national level in an appropriate form. It is permissible to keep conflicting national standards in force (in parallel to the ENV) until the final decision about the possible conversion of the ENV into an EN is reached.

CEN members are the national standards bodies of Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

<https://standards.iteh.ai/catalog/standards/sist/8c17058b-24b3-44b0-b1f4-8c874e5ccc05/sist-env-13999-2-2003>



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

Management Centre: rue de Stassart, 36 B-1050 Brussels

Contents

Foreword.....	3
1 Scope	4
2 Normative references	4
3 Principle	4
4 Safety	4
5 Reagents and materials.....	5
5.1 Volatile organic compounds for calibration, of gas chromatographic grade.	5
5.2 Methanol, of gas chromatographic grade.	5
5.3 Sorbent material.....	5
5.4 Carrier gas	5
6 Apparatus	5
6.1 Sorbent tubes.....	5
6.2 Sorbent tube unions	5
6.3 Syringes.....	5
6.4 Sampling pump.....	5
6.5 Tubing.....	5
6.6 Flow meter calibrator.....	5
6.7 Gas chromatograph.....	6
6.8 Thermal desorption apparatus	6
6.9 Injection facility for preparing standards by liquid spiking	6
7 Procedure	6
7.1 Preparation of sampling tubes	6
7.1.1 Preconditioning.....	6
7.1.2 Conditioning.....	6
7.1.3 Storage of conditioned sorbent tubes before sampling.....	6
7.2 Test chamber air sampling	7
7.3 Storage of loaded sampling tubes	7
7.4 Blank tubes.....	7
7.5 Preparation of calibration blend solutions for liquid spiking	7
7.5.1 Solution containing approximately 10 mg/ml of each liquid component	7
7.5.2 Solution containing approximately 1000 µg/ml of each liquid component	7
7.5.3 Solution containing approximately 100 µg/ml of each liquid component	7
7.5.4 Solution containing approximately 10 µg/ml of each liquid component	7
7.5.5 Stability of calibration blend solutions.....	8
7.6 Preparation of calibration sorbent tubes spiked with standards	8
7.7 Analysis	8
7.7.1 Desorption	8
7.7.2 Gas chromatographic analysis.....	8
7.7.3 Calibration	9
8 Identification and calculation	9
8.1 Identification of single VOC's	9
8.1.1 Identification Classes.....	9
8.2 Concentration of single VOC's in the sampled air	9
8.3 Calculation of TVOC _{ENV 13999}	10
9 Precision and Bias.....	10
10 Test report	10
11 Quality control.....	11
Bibliography	12

iTech STANDARD PREVIEW
(standards.iteh.ai)

[https://standards.iteh.ai/catalog/standards/sist/8c17058b-24f3-44b0-b1f4-](https://standards.iteh.ai/catalog/standards/sist/8c17058b-24f3-44b0-b1f4-8c874e5ccc05/sist-env-13999-2-2003)

[8c874e5ccc05/sist-env-13999-2-2003](https://standards.iteh.ai/catalog/standards/sist/8c17058b-24f3-44b0-b1f4-8c874e5ccc05/sist-env-13999-2-2003)

Foreword

This document ENV 13999-2:2002 has been prepared by Technical Committee CEN/TC 193, "Adhesives", the secretariat of which is held by AENOR.

This document includes a Bibliography.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to announce this European Prestandard : Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

iTeh STANDARD PREVIEW (standards.iteh.ai)

SIST ENV 13999-2:2003

<https://standards.iteh.ai/catalog/standards/sist/8c17058b-24b0-44b0-b1f4-8c874e5ccc05/sist-env-13999-2-2003>

1 Scope

This European Prestandard specifies a method for the determination of single volatile organic compounds (VOC's) and of the total amount of volatile organic compounds in the exhaust air of an emission test chamber after application of an adhesive (TVOC_{ENV13999}). 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's.

2 Normative references

This European Prestandard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text, and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Prestandard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

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

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

ENV 13419-1, *Building products - Determination of the emission of volatile organic compounds - Part 1: Emission test chamber method.*

EN ISO 16017-1:2000, *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 16017-1:2000).*

ISO/DIS 16000-6, *Indoor air - Part 6: Determination of volatile organic compounds in indoor and chamber air by active sampling on TENAX TA sorbent, thermal desorption and gas chromatography using MSD/FID.*

3 Principle

An emission test chamber as described in ENV 13999-1 or in ENV 13419-1 shall be used.

A sufficient volume of test chamber air is drawn with an appropriate flow through one (or more) sorbent tubes in series containing a solid sorbent. Volatile organic compounds (VOC's) are retained by the sorbent tube, which is subsequently analysed in the laboratory. The VOC's 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 Safety

This Prestandard does not purport to address all of the safety concerns, if any associated with its use. The user of this Prestandard should be familiar with common laboratory practice including general safety measures. It is the responsibility of the user of this Prestandard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

5 Reagents and materials

5.1 Volatile organic compounds for calibration, of gas chromatographic grade.

5.2 Methanol, of gas chromatographic grade.

5.3 Sorbent material

For general purpose Tenax¹⁾ TA [1] (poly(2,6-diphenyl-*p*-phenylene oxide)), particle size (0,18 to 0,25) mm i.e. (60 to 80) mesh shall be used.

For the determination of very volatile carcinogenic organic compounds (e.g. vinylchloride) Chromosorb 106¹⁾ (polystyrene) or equivalent sorbent materials as described in EN ISO 16017-1 shall be used.

5.4 Carrier gas

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

6 Apparatus

6.1 Sorbent tubes

Sorbent tubes, made from stainless steel or glass, that can be filled with at least 200 mg of Tenax TA sorbent (5.3) 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.

NOTE 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.

Fill Chromosorb¹⁾ or equivalent tubes as described in EN ISO 16017-1.

6.2 Sorbent tube unions

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

6.3 Syringes

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

6.4 Sampling pump

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

6.5 Tubing

PE (polythene) 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.

6.6 Flow meter calibrator

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

1) Chromosorb 106 and Tenax TA are an example of suitable products available commercially. This information is given for the convenience of users of this European Prestandard and does not constitute an endorsement by CEN of this product.

6.7 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.

6.8 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.

6.9 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.

7 Procedure

7.1 Preparation of sampling tubes

7.1.1 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 ENV 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.

NOTE EN ISO 16017-1 suggests doing this by keeping the tubes at a temperature of 330 °C for at least 18 h using carrier gas flow of 100 ml/min

Pack one end of the sampling tube 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.

7.1.2 Conditioning

Recondition the pre-cleaned sampling tubes prior to each use at temperature of 300 °C for 10 min (Tenax TA) under inert carrier gas flow of (50 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 sorbent tube blank level is acceptable if artefact peaks are no larger than 10 % of the typical areas of the analytes of interest.

7.1.3 Storage of conditioned sorbent tubes before sampling

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

7.2 Test chamber air sampling

Assemble the sampling line. Prepare the test chamber sampling port as described in ENV 13999-1 or in ENV 13419-1.

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 50 % 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.

NOTE The recommended sampling flow rate is in the range of (50 to 200) ml/min. Safe sampling volumes, i.e. the amount of gas that can be sampled without breakthrough of VOC's, are listed in Table 3 in EN ISO 16017-1:2000. In general, the recommended sampling volume is (1 to 10) l for sampling tubes filled with 200 mg of Tenax TA.

7.3 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 you use other sealing material than PTFE/metal then analyse the tubes within two days maximum after sampling.

NOTE 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 and not later than one month after collection.

7.4 Blank tubes

Take blank sampling tubes equivalent to the ones to be used for sampling. Subject the blank tubes to the same handling procedure in the laboratory as the sample tubes except for the actual period of sampling, i.e. repeat the sampling procedure up to the point of actual sample collection. Do not perform sampling but repeat normal post-sampling procedure for the tube. Mark, store, and analyse the blank tubes in sequence with the actual samples.

For blank control of the test chamber see ENV 13999-1.

7.5 Preparation of calibration blend solutions for liquid spiking

7.5.1 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 (5.2), stopper and shake to mix.

7.5.2 Solution containing approximately 1000 µg/ml of each liquid component

Introduce 50 ml of methanol into a 100 ml volumetric flask. Add 10 ml of solution 7.5.1. Make up to 100 ml with methanol, stopper and shake to mix.

7.5.3 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 (5.2), stopper and shake to mix.

7.5.4 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 7.5.3. Make up to 100 ml with methanol, stopper and shake to mix.