



# SLOVENSKI STANDARD

## SIST ENV 13999-1:2002

01-februar-2002

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**Lepila - Kratkotrajna metoda za merjenje emisijskih lastnosti lepil z malo topila ali brez njega po uporabi - 1. del: Splošni postopek**

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

Klebstoffe Kurzzeit-Verfahren zum Messen der Emissionseigenschaften von lösemittelfarmen oder lösemittelfreien Klebstoffen nach der Applikation

Adhésifs - Méthode de mesurage a court terme des caractéristiques émissives des adhésifs a teneur faible ou nulle en solvant apres application - Partie 1: Mode opératoire général

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EUROPEAN PRESTANDARD  
PRÉNORME EUROPÉENNE  
EUROPÄISCHE VORNORM

**ENV 13999-1**

May 2001

ICS 83.180

English version

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

This European Prestandard (ENV) was approved by CEN on 7 March 2001 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, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

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## Foreword

This European Prestandard has been prepared by Technical Committee CEN/TC 193 "Adhesives", the secretariat of which is held by AENOR.

This Prestandard consists of four parts:

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

Annex A is normative.

Annex B is informative.

This Prestandard 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, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

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## Introduction

Solvent-free and low-solvent adhesives can show considerable long-term emissions of volatile chemical compounds. Any documentation of the emission characteristics requires a test method which includes all relevant and potentially hazardous substances.

The test should:

- be reliable and reproducible
- give results in a short time to be useful for decisions for development projects
- verify that carcinogenic or sensitising volatile substances are absent
- characterise the emission properties of the adhesive

This can be achieved by sampling the atmosphere around the applied adhesive kept in an environmental test chamber at controlled ambient conditions.

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## 1 Scope

This European Prestandard describes a conventional standard method for assessing the potential emissions from adhesives after their application.

This Prestandard applies only to "solvent free" and "low solvent" adhesives as they are defined in EN 923. The adhesives shall be applicable at room temperature.

## 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 923	Adhesives - Terms and definitions
EN 1066	Adhesives - Sampling
EN 1067	Adhesives - Examination and preparation of samples for testing
ENV 13419-1	Building products - Determination of the emission of volatile organic compounds - Part 1: Emission test chamber method.
ISO 554	Standard atmospheres for conditioning and/or testing – Specifications
ISO/DIS 16000-6:2000	Indoor Air- Part 6: Determination of volatile organic compounds in indoor and chamber air by active sampling on TENAX TA, thermal desorption and gas-chromatography MSD/FID.

## 3 Terms and definitions

For the purposes of this Prestandard, the terms and definitions given in EN 923, ENV 13419-1, ISO/DIS 16000-6:2000 and the following term and definition apply.

### 3.1

#### **specific cumulative emission (SCE)**

total volatile organic compounds (TVOC) per unit of test area emitted in the time interval between 24 h and 240 h

## 4 Principle

The test determines the emissions of volatile substances from adhesive coatings. Very volatile and mainly particle bound compounds are not detected with the described procedures. The test is performed in an emission test chamber (Figure 1) at specified constant temperature, relative humidity, air mixing and air exchange.

A measurement of the concentrations of the compounds of interest in the air of the exhaust pipe is considered representative of the air in the whole test chamber.

Sampling and analysis of the volatile organic compounds (VOC) shall be done as described in Part 2 of this Prestandard.

Sampling and analysis of the volatile aldehydes shall be done as described in Part 3 of this Prestandard.

Sampling and analysis of the volatile isocyanates shall be done as described in Part 4 of this Prestandard.

The test includes:

- a) a qualitative and quantitative determination of carcinogenic and sensitising substances in an early stage after the application of the adhesive sample,
- b) a determination of the VOC specific emission rate at three points of time,
- c) a determination of specific cumulative VOC emission in the interval between 24 h and 240 h.

## 5 Safety

Persons using this Prestandard shall be familiar with normal laboratory practice.

This Prestandard does not purport to address all the safety problems, if any, associated with its use.

It is the responsibility of the user to establish safety and health practices and to ensure compliance with any European or national regulatory conditions.

## 6 Apparatus

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The test apparatus shall comprise the following main components.

### 6.1 Support

A stainless steel plate or a glass plate shall be used as substrate for the adhesive to be applied.

### 6.2 Adhesive coating device

A device able to ensure an uniform coating of the adhesive to the support (6.1) shall be used.

### 6.3 Emission test chamber (see Figure 1)

All the components of the chamber and connected pipes and tubes that will be in contact with the vapours shall be low emitting and low adsorbing and shall not contribute to the emission test chamber background concentration. Polished stainless steel or glass shall be used to build the chamber structure. The internal volume of the chamber shall not be less than 4 l, preferably from 100 l to 500 l.

The emission test chamber shall be airtight in order to avoid uncontrolled air exchange with external air. The emission test chamber shall be operated slightly above atmospheric pressure to avoid any influence from the laboratory atmosphere. The emission test chamber is considered sufficiently airtight if at least one of the following requirements is fulfilled:

- the air leakage is less than  $\frac{1}{1000}$  of the chamber volume per minute at an overpressure of 1 000 Pa
- the air leakage in operating condition is less than 1% of the supply airflow rate.



## 6.4 Air supply and mixing facilities

The emission test chamber shall have facilities (e.g. electronic mass flow-meter controllers) capable of continuously controlling the air exchange rate at a fixed value with an accuracy of  $\pm 3\%$ . The supply and exhaust air system shall be designed to ensure proper mixing of the air inside the chamber and to keep the air velocity at the surface of the test specimen in the range from 0,1 m/s to 0,3 m/s. The supply air shall be very clean and pre-filtered, not containing any volatile compounds at a level higher than the required test chamber background concentrations of 10  $\mu\text{g}/\text{m}^3$  TVOC and 2  $\mu\text{g}/\text{m}^3$  for any single component. The control of background concentration shall be carried out with the empty support (6.1) inside the test chamber (6.3).

**NOTE** A four-step filter is suggested:

filtration membrane particle size (maximum 0,5  $\text{mg}/\text{m}^3$  breakthrough);

activated charcoal (maximum 0,003  $\text{mg}/\text{m}^3$  breakthrough);

high efficiency compressed air dryer (dew point  $-40\text{ }^\circ\text{C}$  at least) ;

high filtration grade ( $< 0,5\text{ mg}/\text{m}^3$  particles breakthrough at  $21\text{ }^\circ\text{C}$ ).

## 6.5 Air sampling devices

The sampling devices as described in Parts 2, 3 and 4 of this Prestandard shall take the sample air directly from the exhaust air. The devices shall be connected as near as possible to the test chamber and held at the same temperature. The sum of sampling air flows shall be smaller than the supply air flow to the emission test chamber.

**NOTE** Placing the whole apparatus in a room conditioned at  $(23 \pm 2)\text{ }^\circ\text{C}$  is recommended.

## 6.6 Devices for desorption and analysis

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Devices for desorption and analysis shall be used as described in Parts 2, 3 and 4 of this Prestandard.

## 7 Adhesive sample

Take the adhesive sample to be tested in accordance with EN 1066. Examine and prepare this sample for testing in accordance with EN 1067.

### 7.1 Pre-conditioning

A pre-conditioning of the adhesive sample at the temperature of operation  $(23 \pm 2)\text{ }^\circ\text{C}$  shall be performed for at least 24 h before the test is started. To avoid loss of material the adhesive sample shall be kept in a sealed container.

### 7.2 Preparation of a test specimen

Test specimens shall be prepared not later than eight weeks after manufacture of the adhesive sample. The adhesive sample shall be unpacked in the testing laboratory only immediately before starting the test.

Unless otherwise specified choose a support giving the loading factor (the ratio between the area of application and the chamber volume)  $(0,40 \pm 0,02)\text{ m}^2/\text{m}^3$ . The loading factor shall be recorded in the test report. Different adhesives designed for the same application shall be tested at the same loading factor.

**NOTE** This loading factor corresponds to a totally covered floor in a residence with 2,5 m high walls.

Mechanically stir the sample until homogeneous. Weigh the support (6.1) to the nearest  $\pm 0,1\text{g}$ . Spread an adequate amount of the adhesive to be tested by a suitable coating device (6.2) following the manufacturer's instructions until the total surface of the support is covered by a bubble free adhesive coating of uniform surface structure and equal coat weight. Take the coat weight as recommended by the manufacturer. If a range of coat weights is recommended by the manufacturer, the highest coat weight shall be taken.

**NOTE** The coat weight of an adhesive after application depends on the type of adhesive and its use, surface structure and formulation. To obtain comparable results it is thus essential that materials be tested using the spreading techniques and coat weights recommended by the manufacturer.

Weigh the plate again to the nearest  $\pm 0,1\text{g}$  and adjust the adhesive weight to  $\pm 5\%$  of the target coat weight by adding more or by removing parts of the sample. Record coat weight and loading factor in the test report.

The whole procedure shall not take longer than 5 min.

## 8 Procedure

### 8.1 Number of determinations

Sampling at the chamber outlet and analyses shall be made in duplicate. Both individual values and the mean value shall be given in the test report.

### 8.2 Preconditioning of the test chamber

Clean and condition the test chamber by flushing with clean air at test temperature for at least 24 h and check for contamination before any test as described in ENV 13419-1.

### 8.3 Operation of the test chamber

The test chamber shall be operated at standard climate conditions as described in ISO 554 (23 °C, 50 % relative humidity). The temperature of the chamber and the humidity of the supply air shall be monitored continuously and controlled within  $\pm 2\text{ °C}$  and  $\pm 5\%$  relative humidity. The supply air flow shall be adjusted to an air exchange rate of  $(0,5 \pm 0,025)$  per hour.

Unless otherwise specified in this Prestandard, the specifications of ENV 13419-1 for the operation of a test chamber apply.

### 8.4 Starting of the test

Place the prepared test specimen (7.2) into the centre of the chamber (6.3). Consider this instant as time  $t_0$  (zero) for all the time measurements throughout the test.

### 8.5 Sampling and analysis of emitted substances

Perform air sampling according to the following scheduling:

24 hours

48 hours

240 hours.

Analyse the VOC in the 24 hours, the 48 hours and the 240 hours air samples. Take the 24 hours air samples for determination of carcinogenic and sensitising substances.

Sample from test chamber air and determine VOC in the time intervals  $(24 \pm 1)$  h,  $(48 \pm 1)$  h and  $(240 \pm 1)$  h after starting the test in compliance with Part 2 of this Prestandard.

Sample from test chamber air and determine aldehydes in the time interval  $(26 \pm 1)$  h after starting the test in compliance with Part 3 of this Prestandard.

Sample from test chamber air and determine diisocyanates in the time interval  $(28 \pm 1)$  h after starting the test in compliance with Part 4 of this Prestandard.

NOTE Other sampling times can be useful for special purposes such as the determination of emission decay curves.

## 9 Expression of results

### 9.1 Calculations

Calculate the chamber air concentrations, the specific emission rates and the specific cumulative emission (SCE) from the results obtained according to Parts 2,3 and 4 of this Prestandard by the following equations:

**Chamber air concentration = Mass in air sample/ Volume collected,**

where the mass shall be expressed in micrograms and the volume in cubic metres

**Specific emission rate = Chamber air concentration x air exchange flow/test area**

where the chamber air concentration shall be expressed in micrograms/cubic metres, air exchange flow in cubic metres/hour, and the test area in squared metres .

NOTE This “specific emission rate” is equivalent to the one defined in ENV 13419-1 as “SERA”.

**Specific cumulative emission = (Mass of TVOC emitted between 24 h and 240 h)/test area**

where the mass of TVOC shall be expressed in micrograms and the test area in squared metres, calculated as described in 9.3.2

### 9.2 Carcinogenic and sensitising compounds concentration

Search the results of sampling and analysis according to subclause 8.5 for carcinogenic and sensitising compounds.

Report the concentration (micrograms/cubic metres) and specific emission rate (micrograms/(squared metres x hours)) and the nature of any detected carcinogenic and sensitising compounds.

The compounds to be taken into consideration are defined in Annex A according to Directive 67/548/EEC on dangerous substances and further amendments and adaptations. Annex A contains an example list of relevant compounds.

The procedures for identification and quantification depend on the appropriate analytical technique and are described in Parts 2, 3 and 4 of this Prestandard respectively.