# SLOVENSKI STANDARD
# oSIST prEN ISO 23322:2021

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**Barve in laki - Določevanje topil v premazih, ki vsebujejo samo organska topila - Plinska kromatografska metoda (ISO 23322:2021)**

Paints and varnishes - Determination of solvents in coating materials containing organic solvents only - Gas-chromatographic method (ISO 23322:2021)

Lösemittel für Beschichtungsstoffe - Bestimmung der Lösemittel in ausschließlich organische Lösemittel enthaltenden Beschichtungsstoffen - Gaschromatographisches Verfahren (ISO 23322:2021)



Peintures et vernis - Détermination des solvants dans les produits de peinture contenant uniquement des solvants organiques - Méthode par chromatographie en phase gazeuse (ISO 23322:2021)



**Ta slovenski standard je istoveten z:** **prEN ISO 23322**

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INTERNATIONAL STANDARD

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# Paints and varnishes — Determination of solvents in coating materials containing organic solvents only — Gas-chromatographic method

*Peintures et vernis — Détermination des solvants dans les produits de peinture contenant uniquement des solvants organiques — Méthode par chromatographie en phase gazeuse*



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# Foreword

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This document was prepared by Technical Committee ISO/TC 35, *Paints and varnishes*, Subcommittee SC 16, *Chemical analysis*.



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# Paints and varnishes — Determination of solvents in coating materials containing organic solvents only — Gas-chromatographic method

## 1 Scope

This document specifies a method for the gas-chromatographic determination of the qualitative and quantitative composition of solvents contained in a product. The method is applicable to coating materials containing solely organic solvents (generally called conventional coating materials) and binder solutions and non-aqueous dispersions containing solely organic solvents.

The method defined in this document is not applicable for determination of volatile organic compounds (VOC) and semi-volatile organic compounds (SVOC) content.

NOTE For determination of VOC and SVOC, see ISO 11890-2.

## 2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.



ISO 4618, *Paints and varnishes — Terms and definitions*



ISO 15528, *Paints, varnishes and raw materials for paints and varnishes — Sampling*

## 3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 4618 apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

## 4 Units

The analytical results are expressed as a mass fraction.

## 5 Principle

The volatile fraction of the sample of product under test is separated by gas chromatography. Either a hot sample injection system, a cold sample injection system or a headspace injection system can be used, depending on the product type. After the components have been identified, they are quantified from the peak areas using the internal standard method.

# 6 Apparatus

## 6.1 Gas chromatograph

### 6.1.1 General

The gas chromatograph shall be suitable for use with capillary separation columns and meet the conditions specified in 6.1.2 to 6.1.4.

All of the instrumental parts coming into contact with the test sample shall be made of a material, e.g. glass, which is resistant to the sample and will not change it chemically.

### 6.1.2 Sample injection system

#### 6.1.2.1 General

The method provides a choice between three sample injection systems:

— hot-injection system with sample splitter;

— cold-injection system with sample splitter;

— headspace injector.

#### 6.1.2.2 Hot-injection system



The instrument shall have a variable-temperature injection block with sample splitter. The injection temperature shall be capable of being set to an accuracy of 1 K. Standard operating temperature shall be between 250 °C and 280 °C.



NOTE It is useful to use silanized glass wool to retain non-volatile constituents. The active sides of silanized glass wool can be a sink for organic compounds and significantly influence the recovery rate in the lower range of the method. The occurrence of adsorption is revealed by peak tailing, in particular with components of low volatility and/or high polarity.

#### 6.1.2.3 Cold-injection system

The cold-injection system shall be provided with temperature programming for heating from ambient to 300 °C including a sample splitter for split operation.

NOTE It is useful to use silanized glass wool to retain non-volatile constituents. The active sides of silanized glass wool can be a sink for organic compounds and significantly influence the recovery rate in the lower range of the method. The occurrence of adsorption is revealed by peak tailing, in particular with components of low volatility and/or high polarity.

#### 6.1.2.4 Headspace injection

It shall be possible to set the following values:

— controlled sample temperature: 150 °C;

— controlled transfer line and dispensing valve temperatures: 160 °C;

— temperature hold time: 4 min.

### 6.1.3 Oven

The oven shall be capable of being heated between 40 °C and 300 °C, both isothermally and under programmed temperature control. It shall be possible to set the oven temperature to within 1 K. The

 

final temperature of the temperature program shall not exceed the maximum operating temperature of the separation column (see manufacturer's instructions).

#### 6.1.4 Detector

##### 6.1.4.1 General

One of the following two detectors shall be used.

##### 6.1.4.2 Mass spectrometer (MS) or other mass-selective detector (MSD)

To prevent condensation, the detector temperature shall be at least 10 K above the maximum oven temperature.

##### 6.1.4.3 Flame ionization detector

The flame ionization detector (FID) is operated at temperatures between 230 °C and 300 °C. To prevent condensation, the detector temperature shall be at least 10 K above the maximum oven temperature. The detector gas supply, injection volume, split ratio and gain setting shall be optimized so that the signals (peak areas) used for the calculation are proportional to the amount of substance.

#### 6.1.5 Capillary separation column

The column shall be made of glass or fused silica. Columns of sufficient length to resolve volatiles and of maximum internal diameter 0,32 mm, of a suitable polarity and with a suitable film thickness shall be used.



#### 6.1.6 Analytical system performance criteria



The analytical system performance criteria shall be demonstrated. The resolution, $R$, of the peaks to be separated shall be at least 1,5.

For the compounds under investigation it has to be ensured that the sample concentration lies within the quantification range of the analytical system.

NOTE The limit of quantification can deviate for single compounds. If necessary, the compound specific limit of quantification can be determined for the considered single compound(s).

### 6.2 Injection syringe

The injection syringe for hot or cold injection systems shall have a capacity of at least twice the volume of the sample to be injected into the gas chromatograph.

### 6.3 Data processing

Suitable software shall be used for integration, calibration, quantification and other data handling processes.

### 6.4 Sample vial

A suitable sample vial is one made of chemically inert material, for example glass, which can be sealed for example with a rubber membrane having a coating of poly(tetrafluoroethylene) (PTFE). The vessel shall be filled to about 90 % of capacity.

# 7 Reagents

## 7.1 General

Table 1 shows a non-exhaustive list of an internal standards and extraction solvents.

**Table 1 — List of reagents and their function**

| Reagent | CAS-No[a] | Abbreviation | Function |
|---|---|---|---|
| *n*-tetradecane | CAS-No 629–59–4 | C14 | internal standard |
| diethyladipate | CAS-No 141–28–6 | DEA | internal standard |
| acetonitrile | CAS-No 75–05–8 | ACN | extraction solvent |
| methanol | CAS-No 67–56–1 | MEOH | extraction solvent |
| acetone | CAS-No 67-64-1 | AC | extraction solvent |
| tetrahydrofuran | CAS-No 109–99–9 | THF | extraction solvent |
| [a] CAS-No: Chemical Abstracts Service Registry Number. | | | |

## 7.2 Internal standard

The internal standard should be a compound which is not present in the sample and is completely separated from the other components in the chromatogram. It shall be inert with respect to the sample constituents, stable in the required temperature range, and of known purity. The preferred internal standard is DEA.



NOTE If DEA is not suitable as internal standard, internal standards such as glycol ethers can be suitable.

## 7.3 Gases



**7.3.1 Carrier gas:** Dry oxygen-free helium, nitrogen or hydrogen having a purity of at least 99,995 % (volume fraction).

**7.3.2 Detector gases:** Hydrogen having a purity of at least 99,995 % (volume fraction) and (synthetic) air-free of organic compounds.

**7.3.3 Auxiliary gas:** Nitrogen or helium of the same quality as the carrier gas.

Suitable filters shall be installed in the gas chromatograph connection pipes to adsorb residual impurities (see the gas chromatograph operating instructions).

## 7.4 Calibration substances

The solvent used for the calibration shall have a purity of at least 99 % (mass fraction) or shall be of known purity.

## 7.5 Extraction solvent

For better handling of the direct injection, the sample may be diluted with suitable extraction solvents (e.g. acetone, CAS-No 67-64-1, methanol, CAS-No 67-56-1, acetonitrile, CAS-No 75-05-8 or tetrahydrofuran, CAS-No 109-99-9). The extraction solvents shall have a purity of at least 99 % (mass fraction) or be of known purity and shall not contain any substances which interfere with the determination by for example causing overlapping peaks in the chromatogram. Always carry out a separate run injecting the solvent alone in order to observe contaminants and possible interference peaks, especially in trace analysis.

 