

SLOVENSKI STANDARD SIST-TS CEN/TS 17985:2024

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Gradbeni proizvodi - Ocenjevanje sproščanja nevarnih snovi - Metode določevanja N-nitrozaminov v vzorcih zraka, pridobljenih v skladu s standardom EN 16516

Construction Products - Assessment of release of dangerous substances - Methods for the determination of N-nitrosamines in air samples derived by EN 16516

Bauprodukte: Bewertung der Freisetzung gefährlicher Stoffe - Verfahren zur Bestimmung von N-Nitrosaminen in Luftproben, die nach EN 16516 gewonnen wurden

Produits de construction : Évaluation de l'émission de substances dangereuses - Méthodes de détermination des N-nitrosamines dans des échantillons d'air prélevés conformément à l'EN 16516

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English Version

Construction products: Assessment of release of dangerous substances - Methods for the determination of N-nitrosamines in air samples derived by EN 16516

Produits de construction : Évaluation de l'émission de substances dangereuses - Méthodes de détermination des N-nitrosamines dans des échantillons d'air prélevés conformément à l'EN 16516 Bauprodukte: Bewertung der Freisetzung gefährlicher Stoffe - Verfahren zur Bestimmung von N-Nitrosaminen in Luftproben, die nach EN 16516 gewonnen wurden

This Technical Specification (CEN/TS) was approved by CEN on 25 September 2023 for provisional application.

The period of validity of this CEN/TS 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 CEN/TS can be converted into a European Standard.

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European foreword

This document (CEN/TS 17985:2023) has been prepared by Technical Committee CEN/TC 351 "Construction products: Assessment of release of dangerous substances", the secretariat of which is held by NEN.

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Any feedback and questions on this document should be directed to the users' national standards body. A complete listing of these bodies can be found on the CEN website.

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Introduction

This document describes a test procedure for the determination of N-nitrosamines in air samples derived by EN 16516:2017+A1:2020 that is able to determine N-nitrosamines at a level of $\leq 0.2 \, \mu g/m^3$.

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1 Scope

This document describes a test procedure for sampling, elution, detection, and quantification of N-nitrosamines in air samples derived from a test chamber according to EN 16516:2017+A1:2020. The following N-nitrosamines are covered:

- N-Nitrosodimethylamine, CAS No. 62-75-9,
- N-Nitrosomethylethylamine, CAS No. 10595-95-6,
- N-Nitrosodiethylamine, CAS No. 55-18-5,
- N-Nitrosodipropylamine, CAS No. 621-64-7,
- N-Nitrosodiisopropylamine, CAS No. 601-77-4,
- N-Nitrosodibutylamine, CAS No. 924-16-3,
- N-Nitrosopiperidine, CAS No. 100-75-4,
- N-Nitrosopyrrolidine, CAS No. 930-55-2 and
- N-Nitrosomorpholine, CAS No. 59-89-2.

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.

EN 16516:2017+A1:2020, Construction products: Assessment of release of dangerous substances — Determination of emissions into indoor air

3 Terms and definitions

For the purposes of this document, the terms and definitions given in EN 16516:2017+A1:2020 and the following apply.

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

- ISO Online browsing platform: available at https://www.iso.org/obp/
- IEC Electropedia: available at https://www.electropedia.org/

3.1

internal standard

compound of known concentration added to a sample to facilitate the qualitative identification and/or quantitative determination of the sample components

[SOURCE: ISO 16000-6:2021, 3.10]

3.2

N-nitrosamine

substance characterised by the -N-N=O functional group, usually formed by the reaction of an amine with a nitrosating agent at acidic pH

Note 1 to entry: The reacting amines primarily are secondary amines.

Note 2 to entry: An example for a nitrosating agent is nitrite.

[SOURCE: EN 71-12:2016, 3.3]

4 Abbreviations

For the purposes of this document the following abbreviations apply.

GC-TEA Gas chromatography - thermal energy analysis

HPLC-MS/MS High performance liquid chromatography - tandem mass spectrometry

LOD Limit of detection

LOQ Limit of quantification

NDBA N-Nitrosodibutylamine

NDEA N-Nitrosodiethylamine

NDiPA N-Nitrosodiisopropylamine

NDMA N-Nitrosodimethylamine 2000 2000

NDPA N-Nitrosodipropylamine

NMEA N-Nitrosomethylethylamine

NMOR N-Nitrosomorpholine Ment Preview

NPIP N-Nitrosopiperidine

NPYR N-Nitrosopyrrolidine TS CEN/TS 17985:2024

5 Sampling of N-nitrosamines from test chamber air

N-nitrosamines in air samples are collected on special cartridges and are analysed either by GC-TEA or HPLC-MS/MS.

Air samples shall be collected in duplicate. For this, N-nitrosamines from test chamber air shall be collected simultaneously (or immediately sequentially) on two samplers.

A measured volume of 100 l of air from the emission test chamber is drawn at a controlled flow rate (from 1,5 l/min to 2,0 l/min) through the sampler at specified times (as specified in EN 16516:2017+A1:2020) during the emission test. The vapour-phase N-nitrosamines present in the chamber air are trapped on the samplers as the air passes through. The sampler shall prevent the formation of N-nitrosamines from amines, usually by use of an inhibitor (Example: Thermosorb-N Air Sampling Cartridges from Ellutia). The loaded samplers may be stored or transported for a maximum of seven days at room temperature before elution for analysis.

To confirm the absence of the N-nitrosamines, an additional sampling is recommended 24 h after the start of the emission test due to the high volatility of the N-nitrosamines.

NOTE Materials containing vulcanising agents have a potential to form N-nitrosamines.

6 Preparation for subsequent analysis

6.1 General

The samplers are extracted in opposite direction of air sampling.

6.2 Elution for subsequent analysis with GC-TEA

The loaded samplers are placed in an upright position with the "air-in" side on top. $100\,\mu$ l internal standard solution (see 7.1.2) are added with a syringe and left for 5 min. The sampler is turned around that the "air-out" side of sampling is up and a 2 ml-vial is placed under the "air-in" side of the tube. The sampler is eluted with 2 ml dichloromethane/methanol 3:1 mixture. The eluates are then analysed by GC-TEA as described in 7.1.2.

NOTE Approximately 1 ml of extract is received, internal standard method compensates for varying extract volume.

6.3 Elution for subsequent analysis with HPLC-MS/MS

The loaded samplers are eluted with 1,5 ml methanol and made up to 2 ml. After shaking, the eluates are left to settle for 5 min and then filtrated through a 0,2 μ m membrane filter. The eluates are then analysed by HPLC-MS/MS as described in 7.1.3.

NOTE Crystallization is sometimes observed after elution, therefore it is advisable to leave the eluates for 10 min before filtration.

6.4 Storage of eluted samples

The eluates can be stored for at least 14 days in a refrigerator protected from light at a temperature below 4 °C without analyte losses. Tight-sealing vials are required to prevent the loss of the solvent.

7 Analysis and quantification ment Preview

7.1 Analytical method of determination

7.1.1 General

The analytical step shall be carried out twofold for each eluate.

7.1.2 Calibration and analysis with GC-TEA

A calibration shall be carried out before analysis of samples.

Commercially available standard solutions (single or mixture) may be used for calibration. N-Nitroso-propylbutylamine (NPBA) is used as internal standard.

Examples of stock solutions:

- Analyte stock solution (1 μ g/ml of each analyte): 100 μ l of the commercially available standard solution (100 μ g/ml in methanol) is filled-up in a 10 ml brown glass flask to the mark with a mixture of dichloromethane/methanol 3:1.
- NPBA stock solution (1 μ g/ml): 1 ml of the commercially available standard solution (1 000 μ g/ml in methanol) is filled-up in a 10 ml brown glass flask to the mark with methanol. 100 μ l of this solution is filled-up in a 10 ml brown glass flask to the mark with a mixture of dichloromethane/methanol 3:1.

The calibration solutions are prepared in 2 ml brown glass vials from the stock solutions according to Table 1.

Table 1 — Scheme for preparation of calibration solutions for GC-TEA

| Calibration solution | Analyte stock solution | NPBA stock solution [μl] | Fill up volume ^a [µl] | Concentration analytes [ng/ml] | Concentration NPBA [ng/ml] | |
|--|------------------------|--------------------------------|--|--------------------------------|----------------------------------|--|
| 1 | 0 | 100 | 900 | 0 | 100 | |
| 2 | 25 | 100 | 875 | 25 | 100 | |
| 3 | 50 | 100 | 850 | 50 | 100 | |
| 4 | 100 | 100 | 800 | 100 | 100 | |
| 5 | 150 | 100 | 750 | 150 | 100 | |
| 6 | 250 | 100 | 650 | 250 | 100 | |
| 7 | 350 | 100 | 550 | 350 | 100 | |
| 8 | 500 | 100 | 400 | 500 | 100 | |
| a A mixture of dichloromethane/methanol 3:1 is used. | | | | | | |

Example conditions for GC-TEA analysis:

— GC-Column: CP WAX 52 CB, 30 m × 0,25 mm × 0,25 μm

— Carrier gas: Helium 5,0, flow rate 1,2 ml/min

Injector:
 PTV (solvent vent mode)

— Temperature program PTV: hold at 39 °C for 0,05 min, increase temperature with

720 °C/min to 250 °C and hold for 10 min

— Volume of injection: $5 \mu l$

— Temperature program: hold at 60 °C for 2 min, increase temperature with 20 °C/min

to 150 °C and hold for 9 min, increase temperature with

30 °C/min to 220 °C and hold for 2 min

— TEA interface: 250 °C

— Pyrolysis oven: 500 °C

A chromatogram of a calibration solution is shown in Figure 1.