



# SLOVENSKI STANDARD

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Nadomešča:  
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**Kakovost zunanje zraka - Standardna metoda za določevanje koncentracije benzena - 1. del: Vzorčenje s črpanjem in določevanje s plinsko kromatografijo po termični desorpciji**

Ambient air quality - Standard method for measurement of benzene concentrations - Part 1: Pumped sampling followed by thermal desorption and gas chromatography

Außenluft - Verfahren zur Messung von Benzolkonzentrationen - Teil 1: Probenahme mit einer Pumpe, gefolgt von Thermodesorption und Gaschromatographie

Qualité de l'air ambiant - Méthode normalisée pour le mesurage de la concentration en benzène - Partie 1 : Prélèvement par pompage suivi d'une désorption thermique et d'une chromatographie en phase gazeuse

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EUROPEAN STANDARD

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## Ambient air quality - Standard method for measurement of benzene concentrations - Part 1: Pumped sampling followed by thermal desorption and gas chromatography

Qualité de l'air ambiant - Méthode normalisée pour le mesurage de la concentration en benzène - Partie 1 : Prélèvement par pompage suivi d'une désorption thermique et d'une chromatographie en phase gazeuse

Außenluft - Verfahren zur Messung von Benzolkonzentrationen - Teil 1: Probenahme mit einer Pumpe, gefolgt von Thermodesorption und Gaschromatographie

This European Standard was approved by CEN on 20 November 2023.

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## EN 14662-1:2023 (E)

### European foreword

This document (EN 14662-1:2023) has been prepared by Technical Committee CEN/TC 264 "Air quality", the secretariat of which is held by DIN.

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of an identical text or by endorsement, at the latest by June 2024, and conflicting national standards shall be withdrawn at the latest by June 2024.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN shall not be held responsible for identifying any or all such patent rights.

This document supersedes EN 14662-1:2005.

In comparison with the previous edition, the following technical modifications have been made:

- inclusion of the option of multi-tube sampling devices such that a series of samples can be collected sequentially and/or in parallel without user intervention in the field;
- specification of associated 'type testing' requirements to allow multi-tube sampling devices to be tested and approved;
- inclusion of gas standard calibration options, alongside liquid spiking, in line with best practice;
- extensive updates to technical aspects, including uncertainty calculations, in line with the latest standards.

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.

According to the CEN-CENELEC Internal Regulations, the national standards organisations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Republic of North Macedonia, Romania, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Türkiye and the United Kingdom.





## EN 14662-1:2023 (E)

### 3.2

#### **calibration gas mixture**

traceable gas mixture of sufficient stability and homogeneity whose composition is properly established for use in the calibration of a measuring instrument or for the validation of a measurement or gas analytical method

### 3.3

#### **certified reference material**

##### **CRM**

reference material (RM) characterized by a metrologically valid procedure for one or more specified properties, accompanied by an RM certificate that provides the value of the specified property, its associated uncertainty, and a statement of metrological traceability

[SOURCE: ISO Guide 30:2015, 2.1.2 [2]]

### 3.4

#### **combined standard uncertainty**

standard uncertainty of the result of a measurement when that result is obtained from the values of a number of other quantities, equal to the positive square root of a sum of terms, the terms being the variances or covariances of these other quantities weighted according to how the measurement result varies with changes in these quantities

[SOURCE: JCGM 100:2008, 2.3.4 [3]]

### 3.5

#### **competent authority**

organization which implements the requirements of EU Directives and regulates installations, which complies with the requirements of applicable European Standards

Note 1 to entry: In ambient air quality monitoring this is an authority that performs one or more of the tasks listed in Article 3 of Directive 2008/50/EC.

### 3.6

#### **competent body**

organization which can demonstrate its competence for a specific task to the competent authority in the Member State

Note 1 to entry: It is recommended that the competent body performing the required tests be able to demonstrate that it works in conformity with the requirements of internationally accepted standards for test laboratories.

Note 2 to entry: EN ISO/IEC 17025 is the harmonized internationally accepted standard that applies.

Note 3 to entry: A formal accreditation by a member body of the European Accreditation Organization to EN ISO/IEC 17025 is a demonstration of conformity.

### 3.7

#### **desorption efficiency**

ratio of the mass of analyte desorbed from a sampler to that collected

### 3.8

#### **expanded uncertainty**

quantity defining an interval about the result of a measurement that may be expected to encompass a large fraction of the distribution of values that could reasonably be attributed to the measurand

Note 1 to entry: The fraction may be viewed as the coverage probability or level of confidence of the interval.

Note 2 to entry: To associate a specific level of confidence with the interval specified by the expanded uncertainty requires explicit or implicit assumptions regarding the probability distribution characterized by the measurement result and its combined standard uncertainty (3.4). The level of confidence that can be attributed to the interval can be known only to the extent to which such assumptions may be justified.

[SOURCE: JCGM 100:2008, 2.3.5] [3]

### 3.9

#### **reference material**

##### **RM**

material, sufficiently homogeneous and stable with respect to one or more specified properties, which has been established to be fit for its intended use in a measurement process

[SOURCE: ISO Guide 30:2015, 2.1.1] [2]

### 3.10

#### **repeatability condition of measurement**

##### **repeatability condition**

condition of measurement, out of a set of conditions that includes the same measurement procedure, same operators, same measuring system, same operating conditions and same location, and replicate measurements on the same or similar objects over a short period of time

[SOURCE: JCGM 200:2012, 2.20] [4]

### 3.11

#### **retention volume**

elution volume at peak maximum of a small aliquot of a benzene vapour eluted from the tube by air or chromatographic carrier gas

### 3.12

#### **safe sampling volume**

##### **SSV**

volume of air that can be safely drawn through a sampler under prescribed conditions without significant breakthrough of benzene

Note 1 to entry: See EN ISO 16017-1 for more information.

### 3.13

#### **sample delivery**

inert assembly of components needed to transfer the air to the sampling device (3.15) or sequential sampling device (3.17) inlet without impacting the concentration of benzene

### 3.14

#### **sampler**

sampling tube packed with a known mass of sorbent to retain benzene from ambient air

**EN 14662-1:2023 (E)****3.15****sampling device**

device capable of exposing one or more samplers (3.14) to a constant flow of air for one prescribed sampling period, until the required volume of air has been sampled

**3.16****sampling efficiency**

ratio of the mass of analyte collected by a sampler (3.14) to that applied

**3.17****sequential sampling device**

automated sampling device capable of storing multiple samplers (3.14) and exposing them in sequence to a constant flow of air for prescribed periods without user intervention, until all the required volumes of air have been collected in the samplers

**3.18****signal to noise ratio**

measure that compares the level of the signal to the level of background noise

Note 1 to entry: It is also possible to estimate the chromatographic detection limit with the aid of the signal-to-noise ratio. For example, the mean noise level can be estimated across a distance of ten half-peak widths before and after the peak. The threefold of this noise level is then defined as the detection limit [5].

**3.19****standard uncertainty**

uncertainty of the result of a measurement expressed as a standard deviation

[SOURCE: JCGM 100:2008, 2.3.1] [3]

**3.20****uncertainty (of measurement)**

parameter, associated with the results of a measurement, that characterises the dispersion of values that could reasonably be attributed to the measurand

Note 1 to entry: The parameter may be, for example, a standard deviation (or given multiple of it), or the half width of an interval having a stated level of confidence.

Note 2 to entry: Uncertainty of measurement comprises, in general, many components. Some of these components may be evaluated from the statistical distribution of the results of a series of measurements and can be characterized by experimental standard deviations. The other components, which can also be characterized by standard deviations, are evaluated from assumed probability distributions based on experience or other information.

Note 3 to entry: It is understood that the result of a measurement is the best estimate of the value of a measurand, and that all components of uncertainty, including those arising from systematic effects, such as components associated with corrections and reference standards, contribute to this dispersion

[SOURCE: JCGM 100:2008, 2.2.3] [3].

Attention is drawn to the fact that the terms Ambient Air and Limit Value are specified in Directive 2008/50/EC [1].

## 4 Method description

### 4.1 Principle

A measured volume of sample air is drawn through a sampler (3.14). Provided suitable sorbents are chosen, benzene is retained by the sorbent and thus is removed from the flowing air stream. The collected vapour (on each sampler) is desorbed by heat and is transferred under inert carrier gas into a gas chromatograph equipped with a chromatography column and a suitable detector, where it is analysed. The equipment shall be calibrated by one of the following means:

- Vapour or liquid spiking onto a sampler;
- Reference material (3.9) in sampler.

### 4.2 Reagents and materials

The determination of the impurities contained in each material (gas or liquid) used in the preparation has an impact on the uncertainty (3.20) associated with the content of the component. Requirements for the purity analysis of reagent and materials used in the preparation of calibration standards for gases are described in ISO 19229:2019 [7].

Use only volumetric glassware and syringes that are calibrated to ensure traceability of volume to primary standards.

#### 4.2.1 Benzene

Benzene is required for calibration purposes, using either vapour spiking (4.2.6 to 4.2.7) or liquid spiking (4.2.8 to 4.2.10) onto samplers (3.14). The benzene used shall be of a minimum purity of 99,5 %.

#### 4.2.2 Dilution solvent

A dilution solvent is required for preparing calibration blend solution for liquid spiking (4.2.8). The presence of benzene impurity in the solvent or any other gas chromatographic interferent with benzene shall be assessed and it shall be lower than 10 % of the lowest mass of benzene introduced as a calibration standard. During the assessment of the purity the same volume shall be introduced as the volume of the lowest calibration standard.

Methanol is frequently used. Alternative solvents may be used provided they do not interfere with the gas chromatographic analysis, either by co-elution or by altering detector response.

#### 4.2.3 Dilution gas

Dilution gas, such as dry air or nitrogen, is required for preparing calibration gas mixtures (3.2), for preparation of calibration standards in samplers (3.14) and for sampler cleaning. The presence of benzene impurity in the gas or any other gas chromatographic interferent with benzene shall be lower than 10 % of the lowest mass of benzene introduced as a calibration standard. During the assessment of the purity the same volume shall be introduced as the volume of the lowest calibration standard.

#### 4.2.4 Inert gas for sampler conditioning

The purity of the inert gases used for sampler (3.14) conditioning, typically nitrogen or helium, shall be such that the conditioned samplers meet the blank requirements specified in Table 1.

Oxygen filters are recommended in the inert gas supply line to minimize the risk of sorbent oxidation.

**EN 14662-1:2023 (E)****4.2.5 Calibration standards in samplers**

Calibration standards in samplers (3.14) are preferably prepared by loading the required amount of benzene in vapour phase on the samplers from calibration gas mixtures (3.2) (see 4.2.6 and 4.2.7) as this procedure most closely resembles the practical sampling situation.

NOTE 1 The loading ranges given in 4.2.7 to 4.2.10 are not mandatory and approximate to the application range given in the scope.

If this way of preparation is not practicable, calibration standards may be prepared by a benzene liquid spiking procedure (see 4.2.8 to 4.2.10) provided that the accuracy of the spiking technique is either:

- a) established by using procedures giving spiking levels fully traceable to primary standards of mass, or,
- b) confirmed by comparison with reference materials (3.9) (if available), standards produced using calibration gas mixtures, or results of reference measurement procedures.

NOTE 2 See 4.2.10 relating to the mass of solvent retained in the calibration standard.

To minimize matrix effects, calibration standards should be closely matched to the samplers with respect to sorbent type, particle size and mass of sorbent.

Calibration standards are required at a minimum of 5 mass levels for a full calibration (4.7.3) and the mass range of benzene shall cover the maximum range of benzene masses that are expected to be collected/trapped during field monitoring.

Maximum permitted uncertainty (3.20) in the preparation of calibration standards in samplers is 5 % with a level of confidence of 95 %.

**4.2.6 Preparation of calibration gas mixtures**

Prepare calibration gas mixtures (3.2) of known concentrations of benzene by a recognized procedure. Methods described in EN ISO 6142-1 [8], EN ISO 6144 [9] and EN ISO 6145-1 [10] are suitable. If the procedure is not applied under conditions that will allow the establishment of full metrological traceability of the generated concentrations to primary standards of amount of substance, mass and/or volume, the concentrations need to be confirmed using an independent procedure, e.g. EN ISO 6143 [11] or ISO 12963 [12].

For the conversion of the calibration gas mixture composition into mass concentration (mass per volume) use e.g. EN ISO 14912 [13].

NOTE Calibration gas mixtures can be prepared or obtained at the full range of benzene ambient concentration levels. The lower concentrations ( $0,5 \mu\text{g}/\text{m}^3$  to  $5 \mu\text{g}/\text{m}^3$ ) are typically obtained by dynamic methods (ISO 6145-1).

**4.2.7 Calibration standards in samplers prepared by vapour spiking of calibration gas mixtures**

Prepare loaded samplers (3.14) by passing an accurately known volume of the calibration gas mixture (3.2) through the sampling end of conditioned samplers e.g. by means of a constant controlled flow using a pump as described in 4.5 and 4.6. The volume of calibration gas sampled shall not exceed the safe sampling volume (3.12) of the sorbent. After loading the sampler is disconnected and sealed. Prepare fresh standards representing benzene levels in the samples corresponding to the concentration range of  $0,5 \mu\text{g}/\text{m}^3$  to  $50 \mu\text{g}/\text{m}^3$  with each batch of samples or use spiked control samples to ensure consistency of detector response.

NOTE In ambient air monitoring, benzene masses collected typically range from 5 ng to 5  $\mu\text{g}$  per sampler. Therefore, loading volumes of 1 l of a  $5 \mu\text{g}/\text{m}^3$  calibration standard to 10 l of a  $500 \mu\text{g}/\text{m}^3$  calibration standard, will allow covering the lower and upper concentration ranges.