SLOVENSKI STANDARD
SIST EN 14662-3:2016

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Zunanji zrak - Standardna metoda za določevanje koncentracije benzena - 3. del: Avtomatsko vzorčenje s črpanjem in določevanje s plinsko kromatografijo na kraju samem (in situ)

Ambient air - Standard method for the measurement of benzene concentrations - Part 3: Automated pumped sampling with in situ gas chromatography

Außenluft - Referenzverfahren zur Bestimmung von Benzolkonzentrationen - Teil 3: Automatische Probenahme mit einer Pumpe mit gaschromatographischer In-situ-Bestimmung

Air ambiant - Méthode normalisée de mesurage de la concentration en benzene - Partie 3: Méthode à chromatographie en phase gazeuse automatique

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English Version

Ambient air - Standard method for the measurement of benzene concentrations - Part 3: Automated pumped sampling with in situ gas chromatography

Qualité de l'air ambiant - Méthode normalisée pour le mesurage de la concentration en benzène - Partie 3: Prélèvement par pompage automatique avec analyse chromatographique en phase gazeuse sur site

Außenluft - Messverfahren zur Bestimmung von Benzolkonzentrationen - Teil 3: Automatische Probenahme mit einer Pumpe und gaschromatographische In-situ-Bestimmung

This European Standard was approved by CEN on 17 July 2015.

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European foreword

This document (EN 14662-3:2015) 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of an identical text or by endorsement, at the latest by May 2016, and conflicting national standards shall be withdrawn at the latest by May 2016.

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

This document supersedes EN 14662-3:2005.

This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association and supports Essential Requirements of the Council Directive 2008/50/EC [1].

Details of significant technical changes between this European Standard and the previous edition are:

— Clause 8 has been brought in line with other Standards dealing with type approval of gas analysers;

— In 9.4 and 9.6, performance requirements have been modified or removed and additional performance criteria and tests have been introduced for repeatability at span level;

— In 9.5, formulae have been introduced for software adjustment of the raw analyser signal after calibration;

— In Annexes E and F, uncertainty calculations have been modified to be in conformity with EN ISO 14956.

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

1 Scope

This European Standard specifies a semi-continuous measurement method for the determination of the concentration of benzene present in ambient air based on automated sampling and analysis by gas chromatography. This European Standard describes the performance characteristics and sets the relevant minimum criteria required to select an appropriate automated gas chromatograph (GC) by means of type approval tests. It also includes the evaluation of the suitability of an analyser for use in a specific fixed site so as to meet the data quality requirements as specified in Annex I of Directive 2008/50/EC [1] and requirements during sampling, calibration and quality assurance for use.

The method is applicable to the determination of the mass concentration of benzene present in ambient air in the range up to 50 µg/m³ benzene. This concentration range represents the certification range for the type approval test.

Other ranges may be used depending on the levels present in ambient air.

NOTE 1 When the standard is used for other purposes than for measurements required by Directive 2008/50/EC, the ranges and uncertainty requirements may not apply.

The method covers the determination of ambient air concentrations of benzene in zones classified as rural areas, urban-background areas and traffic-orientated locations and locations influenced by industrial sources.

The results are expressed in µg/m³ (at 20 °C and 101,3 kPa).

NOTE 2 50 µg/m³ of benzene corresponds to 15,4 nmol/mol of benzene.

This European Standard contains information for different groups of users.

Clauses 5 to 7 and Annexes C and D contain general information about the principles of benzene measurement by automated gas chromatography and sampling equipment.

Clause 8 and Annex E are specifically directed towards test houses and laboratories that perform type-approval testing of benzene analysers. These sections contain information about:

— type-approval test conditions, test procedures and test requirements;

— analyser performance requirements;

— evaluation of the type-approval test results;

— evaluation of the uncertainty of the measurement results of the benzene analyser based on the type-approval test results.

Clauses 9 to 11 and Annex F are directed towards monitoring networks performing the practical measurements of benzene in ambient air. These sections contain information about:

— initial installation of the analyser in the monitoring network and acceptance testing;

— ongoing quality assurance/quality control;

— calculation and reporting of measurement results;

— evaluation of the uncertainty of measurement results under practical monitoring conditions.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN 15267-1, *Air quality — Certification of automated measuring systems — Part 1: General principles*

EN 15267-2, *Air quality — Certification of automated measuring systems — Part 2: Initial assessment of the AMS manufacturer's quality management system and post certification surveillance for the manufacturing process*

EN ISO 6142, *Gas analysis — Preparation of calibration gas mixtures — Gravimetric method (ISO 6142)*

EN ISO 6143, *Gas analysis — Comparison methods for determining and checking the composition of calibration gas mixtures (ISO 6143)*

EN ISO 6144, *Gas analysis — Preparation of calibration gas mixtures — Static volumetric method (ISO 6144)*

EN ISO 6145-4, *Gas analysis — Preparation of calibration gas mixtures using dynamic volumetric methods — Part 4: Continuous syringe injection method (ISO 6145-4)*

EN ISO 6145-6, *Gas analysis — Preparation of calibration gas mixtures using dynamic volumetric methods — Part 6: Critical orifices (ISO 6145-6)*

EN ISO 6145-7, *Gas analysis — Preparation of calibration gas mixtures using dynamic volumetric methods — Part 7: Thermal mass-flow controllers (ISO 6145-7)*

EN ISO 6145-8, *Gas analysis — Preparation of calibration gas mixtures using dynamic volumetric methods — Part 8: Diffusion method (ISO 6145-8)*

EN ISO 6145-9, *Gas analysis — Preparation of calibration gas mixtures using dynamic volumetric methods — Part 9: Saturation method (ISO 6145-9)*

EN ISO 6145-10, *Gas analysis — Preparation of calibration gas mixtures using dynamic volumetric methods — Part 10: Permeation method (ISO 6145-10)*

EN ISO 14956, *Air quality — Evaluation of the suitability of a measurement procedure by comparison with a required measurement uncertainty (ISO 14956)*

ISO/IEC Guide 98-3:2008, *Uncertainty of measurement — Part 3: Guide to the expression of uncertainty in measurement (GUM:1995)*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
adjustment
set of operations carried out on a measuring system so that it provides prescribed indications corresponding to given values of a quantity to be measured

Note 1 to entry: Types of adjustment of a measuring system include zero adjustment of a measuring system, offset adjustment, and span adjustment (sometimes called gain adjustment).

Note 2 to entry: Adjustment of a measuring system should not be confused with calibration, which is a prerequisite for adjustment.

Note 3 to entry: In the context of this European Standard, adjustment is performed on measurement data rather than on the analyser.

[SOURCE: JCGM 200:2012 (VIM), Note 3 to entry has been modified, [2]]

3.2
ambient air
outdoor air in the troposphere, excluding workplaces as defined by Directive 89/654/EEC where provisions concerning health and safety at work apply and to which members of the public do not have regular access

[SOURCE: 2008/50/EC [1]]

3.3
analyser
analytical instrument that provides an output signal which is a function of the concentration, partial pressure, flow or temperature of one or more components of a gas mixture

3.4
availability of the analyser
fraction of the total time period for which valid measuring data of the ambient air concentration is available from an analyser

3.5
calibration
operation that, under specified conditions, in a first step, establishes a relation between the quantity values with measurement uncertainties provided by measurement standards and corresponding indications with associated measurement uncertainties and, in a second step, uses this information to establish a relation for obtaining a measurement result from an indication

Note 1 to entry: A calibration may be expressed by a statement, calibration function, calibration diagram, calibration curve, or calibration table. In some cases, it may consist of an additive or multiplicative correction of the indication with associated measurement uncertainty.

Note 2 to entry: Calibration should not be confused with adjustment of a measuring system, often mistakenly called “self-calibration”, nor with verification of a calibration.

[SOURCE: JCGM 200:2012 (VIM), modified, 3rd note has been deleted]

3.6
carry-over (memory effect)
influence of the previous measurement due to the retention of benzene within the instrument

3.7
certification range
concentration range for which the analyser is type approved

3.8
check
verification that the analyser is still operating within specified performance limits

3.9
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: ISO/IEC Guide 98-3:2008]

3.10
coverage factor
numerical factor used as a multiplier of the combined standard uncertainty in order to obtain an expanded uncertainty

[SOURCE: ISO/IEC Guide 98-3:2008]

3.11
competent body
body which has been designated for a specific task (type approval tests and/or QA/QC activities in the field) by the competent authority in the Member States

3.12
detection limit
smallest concentration of a measurand that can be reliably detected by a specific measurement process

Note 1 to entry: The detection limit is calculated as $3{,}3x(sr_z/B)$, where sr_z is the standard deviation of instrument response at zero measurand concentration and B is the slope of the calibration function [5].

Note 2 to entry: In principle, the response of the instruments described in this standard to a zero concentration of benzene should be zero. Consequently, sr_z may be determined by repeatedly measuring a low concentration of benzene, e.g., 10 % of the level of the annual limit value (see 8.4.4).

3.13
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 defined by the expanded uncertainty requires explicit or implicit assumptions regarding the probability distribution characterized by the measurement result and its combined standard uncertainty. The level of confidence that may be attributed to this interval can be known only to the extent to which such assumptions may be justified.

[SOURCE: ISO/IEC Guide 98-3:2008]

Note 3 to entry: For the purpose of this standard the expanded uncertainty is the combined standard uncertainty multiplied by a coverage factor k=2 resulting in an interval with a level of confidence of 95 %.

3.14
independent measurement
individual measurement that is not influenced by a previous individual measurement

3.15
individual measurement
measurement over a time period equal to the cycle time of the analyser

Note 1 to entry: Cycle time is defined as the time taken for the analyser to complete all the required functions to report a single measurement. This may include some or all of the following: sample collection, sample trapping, sample desorbing, chromatographic analysis, analyser preparation for next measurement cycle.

3.16
influence quantity
quantity that is not the measurand but that affects the result of the measurement

[SOURCE: ISO/IEC Guide 98-3:2008]

3.17
interferent
component of the air sample, excluding the measured constituent, that affects the output signal

3.18
lack of fit
maximum deviation from the linear regression line of the average of a series of measurement results at the same concentration

3.19
limit value
level fixed on the basis of scientific knowledge, with the aim of avoiding, preventing or reducing harmful effects on human health and/or the environment as a whole, to be attained within a given period and not to be exceeded once attained

[SOURCE: 2008/50/EC [1]]

3.20
long term drift
difference between zero or span readings over a determined period of time (e.g. period of unattended operation)

3.21
monitoring station
enclosure located in the field in which an analyser has been installed to monitor concentrations of one or more ambient air pollutants in such a way that its performance and operation complies with the prescribed requirements

3.22
parallel measurements
measurements from analysers of same type and model, sampling from one and the same sampling manifold starting at the same time and ending at the same time

3.23
performance characteristic
one of the parameters assigned to equipment in order to define its performance

3.24
performance criterion
limiting quantitative numerical value assigned to a performance characteristic, to which conformance is tested

3.25
period of unattended operation
time period over which the drift complies with the performance criterion for long term drift

3.26
repeatability (of results of measurement)
closeness of the agreement between the results of successive individual measurements of benzene carried out under the same conditions of measurement

Note 1 to entry: These conditions include:

— the same measurement procedure;

— the same observer;

— the same analyser, used under the same conditions;

— at the same location;

— repetition over a short period of time.

3.27
reproducibility under field conditions
closeness of the agreement between the results of simultaneous measurements with two analysers in ambient air carried out under the same conditions of measurement

Note 1 to entry: These conditions are called field reproducibility conditions and include:

— the same measurement procedure;

— two identical analysers, used under the same conditions;

— at the same monitoring station;

— the period of unattended operation.

3.28
sampled air
ambient air that has been sampled through the sampling inlet and sampling system

3.29
sample gas temperature
temperature of the sampled gas at the sample inlet outside the monitoring station

3.30
sampling device
component of the analyser which samples an accurately known volume of ambient air

Note 1 to entry: For the purpose of this European Standard, typical devices may consist of a mass-flow controller and timed switching valve, or a sampling syringe.

3.31
sampling system
assembly of components needed to transfer the sampled air to the analyser

3.32
short-term drift
difference between zero or span readings at the beginning and end of a 12-hour period

3.33
standard uncertainty
uncertainty of the result of a measurement expressed as a standard deviation

[SOURCE: ISO/IEC Guide 98-3:2008]

3.34
surrounding temperature
temperature of the air directly surrounding the analyser (temperature inside the monitoring station or laboratory)

3.35
type approval
decision taken by a competent body that the pattern of an analyser conforms to specified requirements

3.36
type approval test
examination of two or more analysers of the same pattern which are submitted by a manufacturer to a competent body including the tests necessary for approval of the pattern

3.37
uncertainty (of measurement)
parameter associated with the result of a measurement that characterises the dispersion of the values that could reasonably be attributed to the measurand

[SOURCE: ISO/IEC Guide 98-3:2008]

4 Abbreviated terms

AMS automated measuring system

MFC mass flow controller

PTFE polytetrafluoroethylene

QA quality assurance

QC quality control

5 Principle

5.1 General

This European Standard describes the method for measurement of the concentration of benzene in ambient air by means of automated sampling and analysis by gas chromatography (GC). The requirements, the specific components of the GC analyser and its sampling system are described. A number of performance characteristics with associated minimum performance criteria are given for the analyser. The actual values of these performance characteristics for a specific type of analyser shall be determined in a so-called type approval test for which procedures have been described. The type approval test comprises a laboratory and a field test. The selection of a type approved analyser for a specific measuring task in the field is based on the calculation of the expanded uncertainty of the measurement method. In this expanded uncertainty calculation the actual values of various performance characteristics of a type approved analyser and the site-specific conditions at the monitoring station are taken into account (see 9.6). The expanded uncertainty of the method shall not exceed 25 % for fixed measurements or 30 % for indicative measurements, as specified in Annex I of Directive 2008/50/EC. Requirements and recommendations for quality assurance and quality control are given for the measurements in the field (see 9.4).

5.2 Measuring principle

A measured volume of sample air is drawn or forced through a sampling trap (sorbent tube). Provided suitable sorbents are chosen, benzene is retained by the sorbent tube and is quantitatively removed from the flowing air stream. The collected benzene is desorbed by heat and is transferred by inert carrier gas into a gas chromatograph equipped with a capillary column and a suitable detector (see 7.6), where it is analysed. Prior to entering the column the sample may be concentrated either on a cryo-trap, which is heated to release the sample into the column, or on a pre-column, where higher boiling hydrocarbons are removed from the pre-column by back flush.

Two general types of instruments are used. One type samples for only part of the time in each cycle whereas the other type of instrument samples continuously as illustrated in Figures 1 and 2 respectively. Typical cycle times are between 15 min and 1 h.

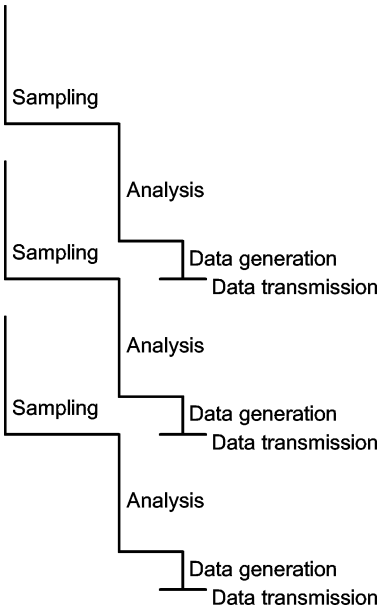

Figure 1 — Sampling by single trap

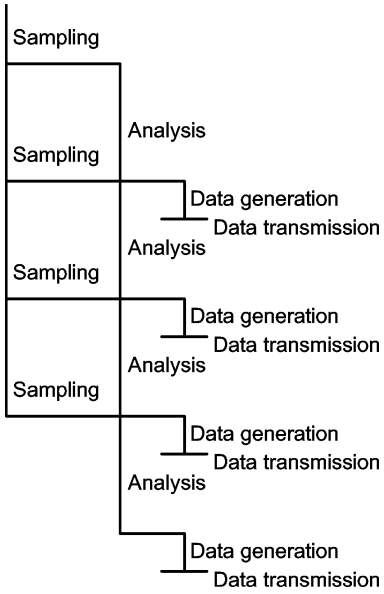

Figure 2 — Sampling by multi-trap

Special attention has to be paid to gases that may co-elute with benzene on the chromatographic column selected, such as hydrocarbons with similar boiling points.

The final results for reporting shall be expressed in µg/m³ using standard conversion factors (see Clause 10).

5.3 Type approval test

The type approval test is based on the evaluation of performance characteristics determined under a prescribed series of tests. In this European Standard, test procedures are described for the determination of the actual values of the performance characteristics for at least two analysers in a laboratory and the same analysers in the field operated in parallel in both cases. The type approval laboratory tests shall not include the sampling inlet or sampling manifold and external data acquisition system, but shall include analyser sampling line and filter. The type approval field test may include a sampling inlet and a sampling manifold. However, the influence of these components on the test results shall be minimized by proper maintenance.

A competent body shall perform these tests. The evaluation for type approval of an analyser is based on the calculation of the expanded uncertainty in the measuring result based on the numerical values of the tested performance characteristics and compared with a prescribed maximum uncertainty.

The type approval of an analyser and subsequent QA and QC procedures provide evidence that the defined requirements concerning data quality laid out in Annex I of Directive 2008/50/EC [1] can be satisfied.

Appropriate experimental evidence shall be provided by:

— type approval tests performed under conditions of intended use of the specified method of measurement, and

— calculation of expanded uncertainty of results of measurement by reference to ISO/IEC Guide 98-3:2008.