SLOVENSKI STANDARD
SIST EN 14662-3:2005

01-september-2005

Kakovost zunanjega zraka - Standardna metoda za določ evanje koncentracije benzena - 3. del: Avtomatsko vzorč enje s č rpanjem in določ evanje z in-situ plinsko kromatografijo

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

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

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

Ta slovenski standard je istoveten z: **EN 14662-3:2005**

ICS:

13.040.20	Kakovost okoljskega zraka	Ambient atmospheres

SIST EN 14662-3:2005 **en,fr,de**

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 14662-3

May 2005

ICS 13.040.20

English version

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

Qualité de l'air ambiant - Méthode pour le mesurage des concentrations en benzène - Partie 3 - Echantillonnage par pompage automatique suivi d'une chromatographie en phase gazeuse in situ

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

This European Standard was approved by CEN on 21 March 2005.

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Ref. No. EN 14662-3:2005: E

Contents

Foreword

This European Standard (EN 14662-3:2005) 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of an identical text or by endorsement, at the latest by November 2005, and conflicting national standards shall be withdrawn at the latest by November 2005.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 EU Directive 2000/69/EC and EU Directive 96/62 EC.

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

1 Scope

This part of EN 14662 is in accordance with the generic methodology selected as the basis of the European Union Reference Method for the determination of benzene in ambient air [1] for the purpose of comparison of measurement results with limit values with a one-year reference period.

The standard describes guidelines for measurements with, and type approval of, automated gas chromatographs. The use of automated instruments gives this part a different structure compared to the other parts including the procedure for selecting an appropriate automated gas chromatograph by means of type approval tests.

Requirements for use in the field are also described.

The standard is applicable to measurements of airborne benzene vapour in the concentration range from 0 µg/m^3 to 50 µg/m^3 (Standardised to 101,3 kPa and 293 K). This concentration range represents the certification range for benzene in the type approval test.

2 Normative references

The following referenced documents are indispensable for the application 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.

ENV 13005, *Guide to the expression of uncertainty in measurement*

EN ISO 14956, *Air quality - Evaluation of the suitability of a measurement procedure by comparison with a required measurement uncertainty (ISO 14956:2002)*

EN ISO/IEC 17025 General requirements for the competence of testing and calibration laboratories (ISO/IEC 17025:1999)

ISO 5725-2:1994, *Accuracy (trueness and precision) of measurement methods and results - Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

ISO 5725-3:1995, *Accuracy (trueness and precision) of measurement methods and results - Part 3: Intermediate measures of the precision of a standard measurement method*

ISO 6142: 2001 Gas analysis – Preparation of calibration gas mixtures - Gravimetric method

ISO 6143 *Gas analysis – Determination of the composition of calibration gas mixtures – Comparison methods*

ISO 6144 *Gas analysis - Preparation of calibration gas mixtures - Static volumetric method*

ISO 6145 (all parts) *Gas analysis - Preparation of calibration gas mixtures - Dynamic volumetric methods*

3 Terms and definitions

For the purposes of this European Standard the following terms and definitions apply.

3.1
Ambient air
outdoor air in the troposphere

3.2
Ambient temperature
temperature at the sample inlet outside the monitoring station

3.3
Availability of the analyser
fraction of the total time period for which valid measuring data of the ambient air concentration is available

3.4
Calibration
comparison of the analyser response to a known gas concentration with a known uncertainty

3.5
Carry-over (memory effect)
influence of the previous measurement due to the retention of benzene within the instrument

3.6
Certification range
concentration range for which the analyser is type approved

3.7
Combined standard uncertainty
calculation result of combining the standard uncertainties calculated from all performance characteristics specified in this standard according to the prescribed procedure given in this standard

3.8
Designated body
body (which can be a laboratory) which has been designated for a specific task (type approval tests and/or QA/QC activities in the field) by a competent authority in the Member States

NOTE It is recommended that the designated body is accredited for the specific task according to EN ISO/IEC 17025

3.9
Detection limit
lowest concentration level in ambient air for which the instrument does not give a zero response

3.10
Expanded uncertainty
for the purposes of this standard, the combined uncertainty of the measurement multiplied by a factor of 2 and so expressed with a level of confidence of 95 %

3.11
Independent measurement
individual measurement that is not influenced by a previous individual measurement

3.12
Individual measurement
measurement averaged over a time period equal to the sampling time

3.13
Influence quantity
quantity that affects the result of a measurement

3.14
Interference
response of the analyser to interferents

3.15
Interferent
single gaseous component in the sampled air, other than benzene, that affects the output signal of the analyser

3.16
International (measurement) standard
standard recognised by an international agreement to serve internationally as the basis for assigning values to other standards of the quantity concerned

3.17
Lack of fit
deviation of the average of a series of measurements at the same concentration from the linear regression line

3.18
Limit value
concentration level fixed on the basis of scientific knowledge, with the aim of avoiding, preventing or reducing harmful effects on human health, as stated in the EU legislation

3.19
Linearity
ability of a series of data to be modelled by a linear regression line

3.20
Long term drift
change in zero or span readings over the period of unattended operation

3.21
Measurand
particular quantity subject to measurement. For the purpose of this EN-standard it is benzene in air

3.22
Monitoring station
enclosure located in the field in which an automated benzene-analyser has been installed in such a way that its performance and operation complies with the prescribed requirements

3.23
Operational range
concentration range for which the analyzer is calibrated. It shall not exceed the certification range

3.24
Parallel measurement
two measurements from different analysers, sampling from one and the same sampling manifold starting at the same time and ending at the same time

3.25
Performance characteristic
one of the parameters assigned to equipment in order to define its performance, e.g. linearity

3.26
Performance criterion
limiting quantitative numerical value assigned to a performance characteristic, to which conformance is tested

3.27
Repeatability (of results of measurement)
closeness of the agreement between the results of successive measurements of the same measurand carried out under the same conditions of measurement [2]

NOTE 1 These conditions are called laboratory repeatability conditions and include:
- the same measurement procedure;
- the same analyser, used under the same conditions;
- at the same location;
- repetition over a short period of time;
- the same observer.

NOTE 2 In this EN-standard the repeatability is expressed as a value with a level of confidence of 95 %.

3.28
Response time
time interval between the instant when a stimulus is subjected to a specified abrupt change and the instant when the response reaches and remains within specified limits around its final steady value.

NOTE For the analyzers described in this Standard, that perform measurements integrated over a certain time period, the response time is at least one measurement cycle.

3.29
Reproducibility under field conditions
closeness of the agreement between the results of parallel measurements with two analysers in ambient air carried out under the same conditions of measurement

NOTE 1 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.

NOTE 2 In this EN-standard the reproducibility under field conditions is expressed as a value with a level of confidence of 95 %

3.30
Sampled air
ambient air that has been sampled through the sampling inlet and sampling system

3.31
Sampling inlet
entrance to the sampling system where the ambient air is collected from the atmosphere

3.32
Short term drift
change in zero or span measurement results over a 24 hour period

3.33
Sorbent
materials that can collect benzene from sample air with near 100 % efficiency and release it again by heating in a flow of a carrier gas e.g. nitrogen, helium or hydrogen

3.34
Standard uncertainty
uncertainty of the result of a measurement expressed as a standard deviation [ENV 13005:1999]

3.35
Temperature of surrounding air
temperature of the air directly surrounding the analyser, ie the temperature inside the monitoring station or laboratory

3.36
Transfer standard
working standard that is routinely used to calibrate or check measuring instruments and is traceable to an international standard and can be moved (transferred) from place to place

3.37
Type approval
decision taken by a designated laboratory that the pattern of an analyser conforms to the requirements as laid down in this EN-standard (comparable to the "pattern approval" as described by the OIML [4])

3.38
Type approval test
examination of two or more analysers of the same pattern which are submitted by a manufacturer to a designated body; this examination includes the test necessary for approval of the pattern (comparable to "pattern evaluation" as described by the OIML [4])

3.39
Uncertainty
parameter associated with the result of a measurement that characterises the dispersion of the values that could reasonably be attributed to the measurand

4 Symbols and abbreviated terms

A	intercept of the calibration function
A_a	availability of the analyser
B	slope of the calibration function
b_j	sensitivity coefficient of c for influence quantity x_j at $C = c_{test}$
c	measured value of the measurand
c_{test}	value of the measurand at which the required measurement uncertainty is given
$\bar{c}$	average value of individual measurements of the measurand
d	drift of measured value on input quantity Y_i at $C = c_{test}$
E_{ss}	collection efficiency of sample system
i	index of input quantities Y
j	index of influence quantities X
k	coverage factor
n	total number of input quantities; last number
m	total number of influence quantities
p	index of the performance characteristic
p_{max}	maximum number of performance characteristics considered
r	Repeatability
$s(x_j)$	standard deviation of x_j at $C = c_{test}$
$t_{0,95}$	95 percentile of the t-distribution
U_c	combined expanded uncertainty of c at $C = c_{test}$ expressed as a 95 % confidence interval

u_c combined standard uncertainty of c at $C = c_{test}$

u_r partial standard uncertainty of c due to repeatability of input quantity Y_i at y_i corresponding to $C = c_{test}$

u_R partial standard uncertainty of c due to reproducibility of input quantity Y_i at y_i corresponding to $C = c_{test}$

u standard uncertainty of input quantity Y_i

x_j jth influence quantity

X_l lack of fit

y_i individual measurand

Y carry over or memory effect of measurand

5 Principles

A measured volume of sample air is drawn or forced through a sorbent tube. Provided suitable sorbents are chosen, benzene is retained by the sorbent tube and thus is removed from the flowing air stream. The collected benzene (on each tube) is desorbed by heat and is transferred by inert carrier gas into a gas chromatograph equipped with a capillary column and a flame ionisation detector or other suitable detector, where it is analysed. Prior to entering the column the sample is 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 is equipped with a single sampling trap and the other is equipped with two or more traps. The single trap instrument samples for only part of the time in each cycle whereas the multitrap instrument samples continuously as illustrated in Figures 1 and 2 respectively. Typical cycling times are between 15 minutes and 1 hour.

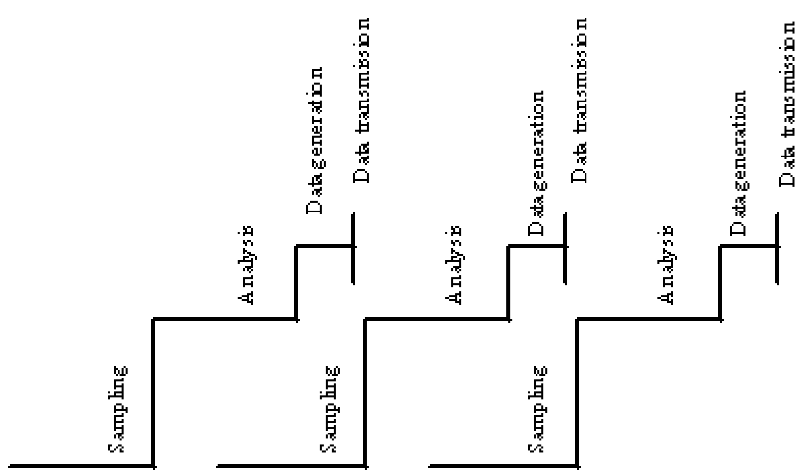

Figure 1 sampling by single trap

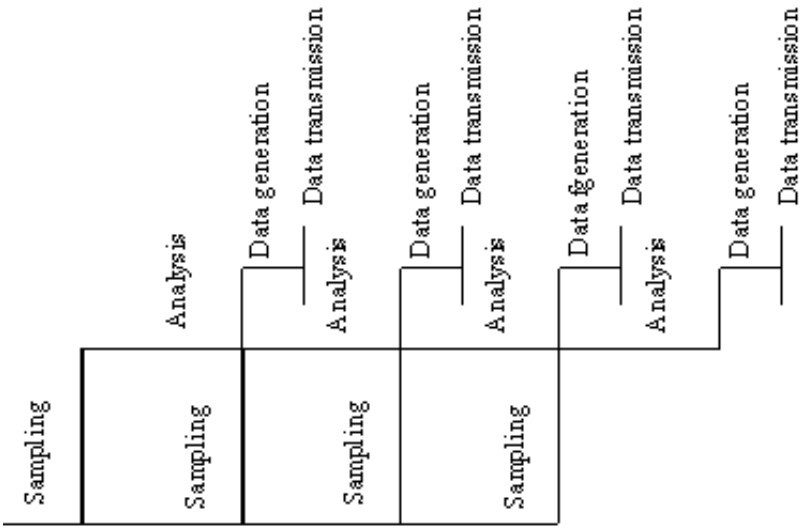

Figure 2 sampling by multitrap

6 Reagents and materials

6.1 Measurement standards of Benzene

Standard atmospheres of known concentrations of benzene shall be prepared by recognised procedures as described in ISO 6142, ISO 6144 and ISO 6145. If the procedure is not applied under conditions that will allow the establishment of full traceability of the generated concentrations to standards of mass or volume, or if the chemical inertness of the generation system cannot be guaranteed, the concentrations need to be confirmed using an independent procedure or a reference gas traceable to national standard.

The concentrations of measurement standard atmospheres shall have an expanded uncertainty of less than 5%.

An internationally accepted certified matrix reference material may also be used.

6.2 Transfer standard/span gas

A transfer standard is needed for quality control to check the regular function of the analyser and to allow correction of drift. The concentration needs to be known but traceability can be obtained from the standard atmospheres of 6.1. The benzene concentration should be between 70% and 90% of the certification range. Transfer standards have to be stable during the period of usage.

The concentrations of transfer standard atmospheres shall have an expanded uncertainty of less than 7,5%.

Methods for transfer standard preparation are listed in Table 1.

Table 1 - Methods for preparation of transfer standards

Method	Description	Traceability/ Standard to be used
Gravimetry	Gas cylinder with a content of benzene in the range 70%-90% of the certification range	ISO 6142 ISO 6143
Permeation tubes	Mass loss of benzene from a permeable tube into a zero air stream	ISO 6145-10
Dynamic dilution	Dynamic blending of cylinder gas containing high benzene concentration (mg/m³) with zero air	ISO 6145-7
Diffusion	Mass loss of benzene from a diffusion tube into a zero air stream	ISO 6145-8

NOTE Practical examples of the application of these methods, with performance characteristics, are described in VDI 2100 Part 4 [5].

6.3 Zero air

Zero air shall have a content of benzene lower than the detection limit and can be provided either as synthetic air from a cylinder or from a compressor followed by an air purifier system.

NOTE The chromatograms of zero air samples provide a check of the benzene content. Where available an independent measurement method should be used for purity analysis.

7 Apparatus

Some typical automated gas chromatograph systems, with their operational parameters for trapping, focusing trap/pre-column, capillary column and detectors are listed in Annex D.

The following equipment is also needed.

7.1 Calibration facility

Calibration standards shall be traceable to internationally accepted standards and meet the requirements of 6.1 and 6.2.

7.2 Sample inlet, sample line and filter

The sampling line and the residence time of the sample shall be as short as practicable. The line and the filter shall be chemically inert to benzene, made of a material such as glass, stainless steel or Per-Fluoro-Carbon (FEP or PFA). Calibration or span gases shall be used to test the inertness of the complete sampling train. The influence of the sample line, filter and inlet shall be less than 2% of the signal output of the analyser at a concentration close to the Limit Value.

NOTE The sample line may need to be moderately heated to avoid condensation of water. Condensation may occur in the case of high ambient temperature and/or humidity.