SLOVENSKI STANDARD
SIST EN 16339:2013

01-december-2013

Zunanji zrak - Metoda za določevanje koncentracije dušikovega dioksida z difuzijskim vzorčenjem

Ambient air - Method for the determination of the concentration of nitrogen dioxide by diffusive sampling

Luftqualitãt - Methode zur Bestimmung der Konzentration von Stickstoffdioxid mittels Passivsammler

Air ambiant - Méthode pour la détermination de la concentration du dioxyde d'azote au moyen d'échantillonneurs par diffusion

Ta slovenski standard je istoveten z: **EN 16339:2013**

ICS:

13.040.20	Kakovost okoljskega zraka	Ambient atmospheres

SIST EN 16339:2013 **en,fr,de**

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EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 16339

July 2013

ICS 13.040.20

English Version

Ambient air - Method for the determination of the concentration of nitrogen dioxide by diffusive sampling

Air ambiant - Méthode pour la détermination de la concentration du dioxyde d'azote au moyen d'échantillonneurs par diffusion

Außenluft - Bestimmung der Konzentration von Stickstoffdioxid mittels Passivsammler

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Ref. No. EN 16339:2013: E

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Foreword

This document (EN 16339:2013) 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of an identical text or by endorsement, at the latest by January 2014, and conflicting national standards shall be withdrawn at the latest by January 2014.

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Introduction

Experience gained across the European Union (EU) in implementing EU ambient air quality legislation [1] has shown that, generally, for nitrogen dioxide (NO_2), meeting the annual average limit value of 40 µg/m^3 is more problematic than meeting the 1-h limit value of 200 µg/m³ [2].

EU Directive 2008/50/EC [1] stipulates that European Union Member States shall apply the reference measurement methods and criteria specified in the Directive. For NO_2 monitoring in ambient air, the reference method being that described in EN 14211:2012 [3]. However, a Member State may use any other method that provides results equivalent to that of the reference method, to be demonstrated in accordance with the Guide for the demonstration of equivalence of ambient air monitoring methods [4]. The GDE devotes specific paragraphs to methods based on diffusive sampling.

For the measurement of longer-term average concentrations of nitrogen dioxide for comparison with the annual average limit value diffusive sampling is an attractive alternative to fixed monitoring using the reference methodology described in EN 14211 because of

— small size of diffusive samplers;

— no requirement for electric power;

— potential for covering areas with a high spatial density;

— cost effectiveness.

Consequently, diffusive samplers can partially substitute and supplement fixed monitoring as an instrument for the assessment of air quality, provided that they fulfil the specific Data Quality Objectives given in [1].

At the time of publication of this standard, no full demonstration of equivalence according to [4] has been performed. However, some studies have compared NO_2 annual average concentrations measured by chemiluminescence and by diffusive samplers [5], [6], [7] and [8]. These have shown the potential of diffusive sampling to meet the data quality objective of 15 % expanded uncertainty for fixed measurements [1].

The methodology described in this standard can be applied to obtain air quality information with a relatively high spatial density that can be used to complement the appropriate siting of fixed monitoring stations, or in the validation of dispersion models.

Further, the methodology described can be used for simultaneously measuring sulphur dioxide (SO_2) when using ion chromatography as the method of analysis. The analytical method is described in [9], [10] and [11].

This standard has been prepared based on the findings of reviews of implemented diffusive samplers in the European Union [12].

The methodology described in this standard may also be used to determine NO_2 in indoor air. Appropriate strategies for NO_2 measurement in indoor air are described in EN ISO 16000-15.

1 Scope

This European Standard specifies a method for the sampling and analysis of NO_2 in ambient air using diffusive sampling followed by extraction and analysis by colorimetry or ion chromatography (IC). It can be used for the NO_2 measurement in a concentration range of approximately 3 μg/m³ to 130 μg/m³. A sample is typically collected for a period of 1 to 4 weeks [13], with exposure periods depending on the design of the samplers and the concentration levels of NO_2.

Several sorbents can be used for trapping NO_2 in ambient air using a diffusive sampler. This standard specifies the application of triethanolamine as the reagent.

Nitrous acid and peroxyacetyl nitrate are the major chemical interferences of sorption by triethanolamine. However, in ambient air monitoring over long sampling times, both contaminants are generally present at low concentrations relative to NO_2. Moreover, these species can also interfere with the measurement of NO_2 when applying the EU reference method for NO_2 monitoring based on chemiluminescence (see [2]).

This standard describes the application of a tube-type sampler with either a cylindrical or a slightly conical tube. Its typical uptake rate is about 1 cm³/min. Only for this sampler type sufficient evidence of validation has been found in a literature survey [12].

The relative expanded uncertainty of NO_2 measurements performed using these tube-type diffusive samplers can potentially be lower than 25 % for individual measurements. When aggregating results to form annual average values, the relative expanded uncertainty can be further reduced to levels below 15 % due to the reduction of random effects on uncertainty [6].

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and 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.

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

3 Terms and definitions

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

3.1
certified reference material
reference material [3.8], characterized by a metrologically valid procedure for one or more specified properties, accompanied by a certificate that provides the value of the specified property, its associated uncertainty, and a statement of metrological traceability

[SOURCE: ISO Guide 35:2006]

3.2
combined standard uncertainty
standard measurement uncertainty [3.10] that is obtained using the individual standard measurement uncertainties associated with the input quantities in a measurement model

[SOURCE: JGCM 200:2012]

3.3
desorption efficiency
ratio of the mass of analyte desorbed from a sampling device to that applied

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[SOURCE: EN 13528-2:2002]

3.4
diffusive sampler
device which is capable of taking samples of gases or vapours from the atmosphere at a rate controlled by a physical process such as gaseous diffusion through a static air layer or a porous material and/or permeation through a membrane, but which does not involve the active movement of air through the device

[SOURCE: EN 13528-1:2002]

Note 1 to entry: Active normally refers to the pumped movement of air.

3.5
diffusive uptake rate
rate at which the diffusive sampler collects a particular gas or vapour from the atmosphere.

[SOURCE: EN 13528-1:2002]

Note 1 to entry: The uptake rate is usually expressed in units of (pg/(nmol/mol)/min) or (cm^3/min).

Note 2 to entry: pg/(nmol/mol)/min is equivalent to ng/(μmol/mol)/min.

3.6
expanded (measurement) uncertainty
product of a combined standard measurement uncertainty and a factor larger than the number one

[SOURCE: JCGM 200:2008]

Note 1 to entry: The factor depends upon the type of probability distribution of the output quantity in a measurement model and on the selected coverage probability.

Note 2 to entry: The term "factor" in this definition refers to a coverage factor.

3.7
field blank
sealed sampler drawn from the same batch as the samplers being used for NO_2 monitoring. This sampler is taken unopened to the field and returned together with exposed samplers after the sampling is completed

Note 1 to entry: This blank is only used for quality control purposes.

Note 2 to entry: A transport blank is considered to be a special case of a field blank. A transport blank is taken to the exposure site, left unopened and returned to the laboratory immediately after placement or collection of the samplers. Transport blanks may be used when regular field blanks reveal an unacceptable level of nitrite to investigate the possibility of contamination of samplers during transport.

3.8
laboratory blank
sealed sampler drawn from the same batch as the samplers being used for NO_2 monitoring which is stored in a refrigerator during sampling of the exposed samplers.

3.9
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: JGCM 200:2012]

3.10
standard (measurement) uncertainty
measurement uncertainty expressed as a standard deviation

[SOURCE: JGCM 200:2012]

3.11
uncertainty (of measurement)
non-negative parameter characterizing the dispersion of the quantity values being attributed to a measurand, based on the information used

Note 1 to entry: For footnotes to the definition the reader is referred to the parent document JGCM 200:2012.

[SOURCE: JGCM 200:2012]

4   Principle of the method

The diffusive sampler is exposed to air for a measured time period. NO_2 migrates through the sampler diffusion path and is collected as nitrite by reaction with triethanolamine (TEA). TEA is coated onto a suitable support. Supports that have been demonstrated to be suitable in practice are (see Annex A):

⎯ a series (2 or 3) of circular stainless steel grids with a fine mesh size;

⎯ a cylindrical stainless steel grid with a fine mesh size;

⎯ a cellulose-fibre filter.

A number of pathways have been proposed for the reaction of nitrogen dioxide with triethanolamine. More details can be found in [14].

The diffusive uptake rate is determined either by numerical calculation based on Fick's first law of diffusion (see EN 13528-3) or through calibration by exposure to standard atmospheres, and/or by field comparison of diffusive samplers measurements with measurements carried out using the EU reference method (EN 14211). This latter approach has been described in [5], [6], [7] and [18].

Values of and equations to calculate diffusive uptake rates associated with different diffusive samplers are given in Annex C.

NOTE 1   The theory of performance of diffusive samplers is given in EN 13528-3 together with information on possible saturation of the sorbent, the effect of transients and the effect of face velocity. This standard explains the dependence of diffusion uptake rates on the concentration level of pollutants and sampling time.

The nitrite formed in the sampler is subsequently extracted. The resulting extract is analyzed by:

⎯ colorimetry after derivatization of the nitrite, using the Griess-Saltzman method [15];

⎯ ion chromatography [16].

The Griess-Saltzman derivatization consists of reacting nitrite with a mixture of sulphanilamide and N-(naphthyl-1) ethylenediamine dihydrochloride in dilute orthophosphoric acid (see Table 1). The absorbance of the azo dye formed is measured at approximately 540 nm.

NOTE 2   In practice, when applying colorimetry, the derivatization agent solution is directly used for desorption.

NOTE 3   When using ion chromatography, sulphur dioxide can be determined simultaneously [9], [10] and [11].

The analytical system is calibrated by means of solutions of nitrite in water with accurately known concentrations.

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NOTE 4 An example of commercially available European sampler based on a sorption media not containing triethanolamine is given in the informative Annex B.2 (badge-type A with typical uptake rate of about 12 cm³/min).

5 Materials

5.1 Sampling

5.1.1 Diffusive samplers

5.1.1.1 Description

Descriptions of the tube-type sampler with cylindrical and with conical tube are given in Annex A. The descriptions hold for sampler designs that have a proven practical validity.

NOTE 1 A radial-type sampler, with typical uptake rate about 70 cm³/min, exists and is widely used in the EU. Limited validation data are available for this sampler; it is therefore described in the informative Annex B.1.

NOTE 2 A badge-type NO_2 diffusive sampler exists that is based on the application of triethanolamine. Limited validation data is available for this sampler. The sampler is described in the informative Annex B.3 (badge-type B with typical uptake rate of about 12 cm³/min).

The sampler may include a turbulence barrier or a protective device (5.1.2) in order to avoid effects of turbulence inside the diffusion path during sampling.

When a turbulence barrier or a protective device is considered an integral part of the sampler, the performance of the sampler shall be validated including the turbulence barrier or protective device.

5.1.1.2 Preparation

The preparation of the sampler consists of the coating of a support with triethanolamine from a solution in water, methanol or acetone. To this solution a wetting agent may be added to facilitate the coating. In principle, one of the procedures specified in Annex A shall be used for the coating.

Preparation procedures are taken from references describing tube-type samplers with a cylindrical tube. Three preparation methods are given in Annex A. These preparation methods have proven to be effective in practice. Other methods may be used provided that their suitability has been satisfactorily demonstrated.

5.1.1.3 Triethanolamine (TEA)

Purity $\geq$ 99 %.

TEA has a melting point of approximately 20 °C depending on its purity. When using volumetric techniques for measuring quantities of TEA, the TEA should be handled at temperatures well above its melting point. Alternatively, gravimetry may be used.

5.1.1.4 Acetone

For the preparation of TEA coating solutions. Purity ≥ 99,9 %.

5.1.1.5 Ultrapure water

For the preparation of TEA coating solutions. Its conductivity shall be equal or less than 0,1 µS/cm.

5.1.1.6 Wetting agent compound

A wetting agent, for example BRIJ 35®[1] [15, 16], may be used for the preparation of TEA coating solutions. The wetting agent shall not contain significant nitrite impurities.

5.1.1.7 Storage and shelf-life of samplers

After preparation, samplers shall be sealed and stored under cool conditions, for example at temperatures between 0 °C and 4 ºC, in the dark, in order to minimize any undesired reactions. Samplers are stable for up to one year [16] when prepared according to the preparation method described in A.1.3. and stored under the above conditions. Tube-type samplers shall be stored in a position that minimizes the probability of the absorbing solution moving onto the tube walls, for example in a vertical position with the TEA at the bottom of the tube.

When storing samplers for periods ≥ 3 months, subsets shall be analyzed periodically to check that blank development does not occur.

5.1.2 Protective device

Consisting of a protective cover or box to be used, where appropriate, to prevent:

— the entrance of coarse particles or water droplets into the sampler during sampling;

— exposure to direct sunlight;

— exposure to high wind velocities.

NOTE 1   EN 13528-3 gives general recommendations for the design of protective devices.

NOTE 2   In Annex A protective devices are described in for the tube-type samplers; in Annex B protective devices are described for the other samplers.

Protective devices should be used consistently over time when measuring at a specific site for a prolonged period, or over space when performing monitoring campaigns, in order to ensure the internal consistency of sampler behaviour.

5.1.3 Support

A device capable of positioning the sampler at an appropriate height and distance from obstacles to warrant undisturbed sampling.

5.1.4 Instructions for use

The manufacturer shall make available a manual or instruction sheet for the handling of the samplers. These shall be followed in order to ensure proper operation of the sampler.

5.2 Analysis

5.2.1 General

During the analysis, use only reagents of known purity appropriate to the application.

Use only volumetric glassware, pipettes and syringes that are calibrated to ensure traceability of volume to primary standards. Volumetric glassware shall be cleaned with ultra pure water (see 5.2.2.1).

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[1] BRIJ 35® is an example of a suitable product available commercially. This information is given for the convenience of users of this European Standard and does not constitute an endorsement by CEN of this product.

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5.2.2 Reagents

5.2.2.1 Ultra pure water

To be used for the preparation of reagents, calibration solutions and chromatography eluents. Its conductivity shall be equal or less than 0,1 µS/cm.

5.2.2.2 Sodium nitrite

To be used for the preparation of calibration standards and check solutions (see 7.2 and 9.1).

The sodium nitrite shall be of a minimum established purity of 99,5 %. The sodium nitrite shall be dried before preparation of the solution by placing a small quantity of sodium nitrite on a container (for example a Petri-dish) placed in an oven (5.2.3.4) at 102 °C ± 2 °C until completely dry. In practice, it is sufficient to wait for between 1 and 2 h. The small quantity of sodium nitrite shall be cooled at ambient temperature before weighing. It is necessary to cool in a dry atmosphere, for example using a desiccator (see 5.2.3.6). Special care shall be taken to avoid contamination during handling by using gloves and a glass container for weighing.

It is also possible to use commercially available certified standards (typically at the level of 1 g/l of nitrite) to eliminate the possibility of errors due to inaccurate standard preparation.

5.2.2.3 Orthophosphoric acid (PA)

To be used for the preparation of colorimetric reagent. 85 % in water (density =1,70 g/cm^3). Only use acid of high purity.

NOTE   Commercial trade qualifications of appropriate purity grades are "type AR grade", "pro analysis grade" and "grade trace analysis".

5.2.2.4 Sulphanilamide (SA)

To be used for the preparation of the colorimetric reagent. Purity ≥ 99 % (see Table 1).

5.2.2.5 Sulphanilic acid (SAc)

To be used for the preparation of the colorimetric reagent. Purity ≥ 99 % (see Table 1).

5.2.2.6 N-(naphthyl-1) ethylene diamine dihydrochloride (NEDD)

To be used for the preparation of the colorimetric reagent. Purity ≥ 98 %.

5.2.2.7 Colorimetric reagent

The compositions of the colorimetric reagent are given in Table 1.

Table 1 — Commonly used compositions of reagents[a]

Sampler type	Reagent A	Reagent B	Reagent mixture
Tube-type sampler with a cylindrical tube	Dissolve 10 g of SA in 300 ml of water. Add 25 ml of PA. Add water up to 500 ml	Dissolve 70 mg NEDD in 300 ml water; add water up to 500 ml	Equal volumes of reagents A and B
Tube-type sampler with a slightly conical tube	Dissolve 10 g of SAc and 25 ml of PA in 500 ml water. Add 500 ml of water and heat the solution to boiling	Dissolve 72,8 mg NEDD in 100 ml water	After cooling of solution A to room temperature, the two solutions are combined. The colour reagent is ready after 24 h
[a] The compositions specified have been demonstrated to be fit for purpose in the validation studies of the two sampler types.			

It is important to ensure that the pH of the solution being analysed remains below 2 [18, 19] when:

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— the proportions of the reagent are modified;

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— the sample to be analyzed is diluted; dilutions shall be performed with the colorimetric reagent only.

5.2.2.8   Sodium carbonate

Anhydrous, purity ≥ 99,8 %. To be used for the preparation of the eluent for ion chromatography.

5.2.2.9   Sodium bicarbonate

Purity ≥ 99,0 %. To be used for the preparation of the eluent for ion chromatography.

5.2.2.10   Eluent for ion chromatography

The composition of the eluent will depend on the ion chromatography column being used. Generally, the eluent is an aqueous solution of sodium carbonate and sodium bicarbonate.

5.2.3   Equipment

5.2.3.1   Colorimeter

With wavelength settings in the range of 500 to 600 nm. The volumes of the cuvettes of the colorimeter shall be consistent with the volumes of mixed reagents used for derivatization (see Table 2).

5.2.3.2   Ion chromatograph

With anion column and conductivity detector. Select a column that allows sufficient separation of chloride and nitrite.

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5.2.3.3 Sample shaker

For the extraction of nitrite from the sampling support. A vibrating tray or a vortex shaker have been found to be acceptable.

5.2.3.4 Oven

For drying parts of diffusive samplers before assembling. Only use electric ovens and do not use gas ovens because flame combustion generates oxides of nitrogen.

5.2.3.5 Balance

Laboratory balance with a resolution $\leq$ 0,1 mg.

5.2.3.6 Desiccator

Airtight chamber made of glass containing a substance with a strong affinity for water such as calcium chloride or silica gel.

6 Sampling

6.1 Calibration of the diffusive uptake rate

Information about the diffusive uptake rates and (potential) effects of concentration and environmental factors is provided in Annex C. This information will cover most practical conditions under which the samplers are used. When a sampler is to be used outside the range of concentration and environmental factors specified by the manufacturer, the diffusive uptake rate and its uncertainty shall be determined experimentally. This shall be done – where practicable – by a combination of laboratory and field experiments. Information about experimental approaches and evaluation of results can be found in [4].

6.2 Siting criteria

When measuring for the purpose of checking compliance with the requirements of EU Directive 2008/50/EC, the siting criteria of this Directive for fixed monitoring shall be met.

Information on siting criteria can also be found in [18]. Further information for determining appropriate monitoring sites for diffusive samplers can be found in [20] and [21].

6.3 Protective measures

Samplers shall be transported to and from the sampling site in suitable plastic containers and/or sealable clean plastic bags in order to avoid contamination during transport.

During sampling the sampler shall be protected from influences of environmental conditions that can adversely affect its performance, for example by using a protective device (see 5.1.2).

When a protective device is used, it shall be suitable for the particular sampler used.

A protective cover or box may be an integral part of the sampler, thereby affecting its uptake rate (see Annex A).

6.4 Exposure

All handling of samplers shall be done in such a way that contamination of the sampler or re-usables such as caps is avoided. Follow the manufacturer's instructions for specific sampling handling, for example, for opening and closing of samplers.

Mount the sampler at the appropriate site, using the support (5.1.3), and fit the protective cover (5.1.2) if needed.

The actual permissible period of sampling may vary with the sampler design, e.g., due to the presence of a membrane. Different designs of diffusive samplers have different diffusive uptake rates; the exposure time shall be such that the mass of nitrite is within the minimum and maximum capacity of the sampler specified by the manufacturer.

NOTE   A simple method for predicting the mass of nitrite in a sampler is to multiply the sampling time by the expected highest NO_2 concentration and the nominal uptake rate.

Prepare field blanks by using samplers identical to those used for sampling and subjecting them to the same handling procedure as the samples except that they will be kept unopened. The number of blanks shall be ≥ 10 % of the number of samplers exposed. When the number of samplers exposed simultaneously exceeds 30, it is sufficient to use 3 field blanks.

After exposure, collect and close the samplers, and store these as described below.

6.5 Storage after sampling

After sampling, samples shall be sealed and stored under cool conditions, for example at temperatures between 0 °C and 4 ºC, in the dark in order to minimize any undesired reactions.

Although samples may be stable for up to 4 months [11] when stored under the above conditions, it is strongly recommended to analyze the samples as soon as possible after exposure.

6.6 Auxiliary information

Information about average air temperatures and pressures during sampling may be necessary for the conversion of measurement results to conditions of standard temperature and pressure. These data may be obtained from measurements or from local meteorology stations.

Information about the average temperature, relative humidity and wind speed may also be required to check that the sampler has been used under the conditions for which it has been validated, or as parameters for the calculation of the effective uptake rate during the sampling period.

7   Analytical procedure

7.1 Safety precautions

This standard does not purport to address all of the safety concerns associated with its use. It is the responsibility of the user of this standard to establish appropriate health and safety practices and determine the applicability of regulatory limitations prior to use.

7.2 Calibration

Calibration shall be performed by analyzing a series of solutions of nitrite in mixed reagent for colorimetry or in water or eluent for ion chromatography.

A full calibration shall include at least 6 calibration standards.

For ion chromatography it is recommended to use concentrations of the calibration standards of 0,0, 0,1, 0,2, 0,4, 0,8, 1,5 and 2,0 µg/ml of nitrite.

For colorimetry it is recommended to use concentrations of the calibration standards of 0,0, 0,1, 0,2, 0,4, 0,6, 0,8 and 1,0 µg/ml of nitrite. Higher concentrations are not recommended because of potential problems with linearity [22].