



SLOVENSKI STANDARD

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SIST EN 14212:2005

Zunanji zrak - Standardna metoda za določevanje koncentracije žveplovega dioksida z ultravijolično fluorescenco

Ambient air - Standard method for the measurement of the concentration of sulphur dioxide by ultraviolet fluorescence

Außenluft - Messverfahren zur Bestimmung der Konzentration von Schwefeldioxid mit Ultraviolett-Fluoreszenz

Qualité de l'air ambiant - Méthode normalisée pour le mesurage de la concentration en dioxyde de soufre par fluorescence UV

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EUROPEAN STANDARD

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ICS 13.040.20

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English Version

Ambient air - Standard method for the measurement of the concentration of sulphur dioxide by ultraviolet fluorescence

Qualité de l'air ambiant - Méthode normalisée pour le mesurage de la concentration en dioxyde de soufre par fluorescence U.V.

Luftqualität - Messverfahren zur Bestimmung der Konzentration von Schwefeldioxid mit Ultraviolett-Fluoreszenz

This European Standard was approved by CEN on 10 May 2012.

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EN 14212:2012 (E)**Foreword**

This document (EN 14212:2012) 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of an identical text or by endorsement, at the latest by February 2013, and conflicting national standards shall be withdrawn at the latest by February 2013.

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 14212:2005.

The technical changes made since EN 14212:2005 are listed in Annex I of this European Standard.

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, 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.

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1 Scope

This European Standard specifies a continuous measurement method for the determination of the concentration of sulphur dioxide present in ambient air based on the ultraviolet fluorescence measuring principle. This standard describes the performance characteristics and sets the relevant minimum criteria required to select an appropriate ultraviolet fluorescence analyser 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 sulphur dioxide present in ambient air up to 1000 $\mu\text{g}/\text{m}^3$. This concentration range represents the certification range for SO_2 for the type approval test.

NOTE 1 Other ranges may be used depending on the levels present in ambient air.

NOTE 2 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 sulphur dioxide in zones classified as rural areas, urban-background areas and traffic-orientated locations and locations influenced by industrial sources.

The results are expressed in $\mu\text{g}/\text{m}^3$ (at 20 °C and 101,3 kPa).

NOTE 3 1 000 $\mu\text{g}/\text{m}^3$ of SO_2 corresponds to 376 nmol/mol of SO_2 .

This standard contains information for different groups of users.

Clauses 5 to 7 and Annexes C and D contain general information about the principles of sulphur dioxide measurement by ultraviolet fluorescence analyser and sampling equipment.

Clause 8 and Annex E are specifically directed towards test houses and laboratories that perform type-approval testing of sulphur dioxide 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 sulphur dioxide analyser based on the type-approval test results.

Clauses 9 to 11 and Annexes F and G are directed towards monitoring networks performing the practical measurements of sulphur dioxide 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.

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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 methods (ISO 6144)*

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 6145-10, *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)*

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

ENV 13005:1999, *Guide to the expression of uncertainty in measurement*

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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.

[SOURCE: JCGM 200:2012 (VIM) [2]]

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

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**

measuring system 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 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.

Note 3 to entry: Often, the first step alone in the above definition is perceived as being calibration.

[SOURCE: JCGM 200:2012 (VIM) [2]]

Note 4 to entry In the context of this standard, calibration is a comparison of the analyser response to a known gas concentration with a known uncertainty when the information obtained from the comparison is used for the successive adjustment (if needed) of the analyser.

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3.6**certification range**

concentration range for which the analyser is type-approved

3.7**check**

verification that the analyser is still operating within specified performance limits

3.8**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 co-variances of these other quantities weighted according to how the measurement result varies with changes in these quantities

[SOURCE: ENV 13005:1999]

3.9**coverage factor**

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

[SOURCE: ENV 13005:1999]

3.10**designated 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

EN 14212:2012 (E)**3.11****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 $3,3x(s_z/B)$ where s_z is the standard deviation of analyser response at zero measurand concentration (see 8.4.5) and B is the slope of the calibration function [3].

3.12**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 characterised 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: ENV 13005:1999]

Note 3 to entry: For the purpose of this European 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 %.

3.13**fall time**

difference between the response time (fall) and the lag time (fall)

3.14**independent measurement**

individual measurement that is not influenced by a previous individual measurement by separating two individual measurements by at least four response times

Note 1 to entry: The largest value of response time (rise) and response time (fall) are intended.

3.15**individual measurement**

measurement averaged over a time period equal to the response time of the analyser

Note 1 to entry: The largest value of response time (rise) and response time (fall) are intended.

Note 2 to entry: This definition differs from the meaning of the concept "individual measurement" in Directive 2008/50/EC [1].

3.16**influence quantity**

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

[SOURCE: ENV 13005:1999]

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**lag time**

time interval from the moment at which a step change of sample concentration occurs at the inlet of the analyser to the moment at which the output reading reaches a level corresponding to a predefined change of the stable output reading

3.20**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]]

3.21**long term drift**

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

3.22**monitoring station**

enclosure located in the field in which an analyser has been installed to monitor concentrations of one or more ambient air pollutants

3.23**parallel measurements**

measurements from different analysers, sampling from one and the same sampling manifold starting at the same time and ending at the same time

3.24**performance characteristic**

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

3.25**performance criterion**

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

3.26**period of unattended operation**

time period over which the drift complies with the performance criterion for long term drift

3.27**repeatability (of results of measurement)**

closeness of the agreement between the results of successive individual measurements of sulphur dioxide carried out under the same conditions of measurement

Note 1 to entry: These conditions include:

- a) the same measurement procedure;
- b) the same observer;
- c) the same analyser, used under the same conditions;
- d) at the same location;
- e) repetition over a short period of time.

3.28**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:

- a) the same measurement procedure;
- b) two identical analysers, used under the same conditions;

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- c) at the same monitoring station;
- d) the period of unattended operation.

3.29**residence time inside the analyser**

time period for the sampled air to be transported from the inlet of the analyser to the reaction chamber

3.30**residence time in the sampling system**

time period for the sampled air to be transferred to the inlet of the analyser

3.31**response time**

time interval from the instant at which a step change of sample concentration occurs at the inlet of the analyser to the instant at which the output reading reaches a level corresponding to a predefined change of the output reading

3.32**sampled air**

part of ambient air that is transferred through the sampling inlet and sampling system for subsequent measurement

3.33**sample gas temperature**

temperature of the sampled gas at the sample inlet

Note 1 to entry: The term "gas" may refer to a test gas used in type-approval testing or to ambient air transferred to the analyser.

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3.34**sampling system**

assembly of components needed to transfer ambient air to the analyser

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3.35**short-term drift**

difference between zero or span readings at the beginning and end of a 12 h period

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3.36**standard uncertainty**

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

[SOURCE: ENV 13005:1999]

3.37**surrounding temperature**

temperature of the air directly surrounding the analyser

3.38**total residence time**

sum of the residence time in the sampling system and the residence time inside the analyser

3.39**type approval**

decision taken by a designated body that the pattern of an analyser conforms to specified requirements

3.40**type approval test**

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

3.41**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: ENV 13005:1999]

4 Abbreviated terms

FEP perfluoro-ethylene-propylene;

MFC mass flow controller;

PTFE polytetrafluoroethylene.

5 Principle**5.1 General**

This standard describes the method for measurement of the concentration of sulphur dioxide in ambient air by means of ultraviolet fluorescence. The requirements, the specific components of the ultraviolet fluorescence 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 test 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 15% for fixed measurements or 25 % for indicative measurements, as specified in Annex 1 of Directive 2008/50/EC [1]. Requirements and recommendations for quality assurance and quality control are given for the measurements in the field (see 9.4).

5.2 Measuring principle

UV (ultraviolet) fluorescence is based on the emission of light by SO₂ molecules excited by UV radiation when they return to their ground state:

The first reaction step is:



In the second step the excited SO₂^{*} molecule returns to its ground state, emitting an energy hν' according to the reaction:



The intensity of the fluorescence radiation is proportional to the number of SO₂ molecules in the detection volume and is therefore proportional to the concentration of SO₂.

Therefore:

$$F = k \times c_{\text{SO}_2} \quad (3)$$

where

F is the intensity of fluorescence radiation;

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k is the factor of proportionality;

c_{SO_2} is the concentration of SO_2 .

Before entering the fluorescence analyser, the air sample is passed through a filter in order to exclude interferences caused by contamination with particles.

The sampled air is scrubbed to remove any interference by aromatic hydrocarbons that may be present. A hydrocarbon scrubber device is used to achieve this.

The sampled air is then introduced into a reaction chamber, where it is irradiated by UV light in the wavelength range between 200 nm and 220 nm. The UV fluorescent light emitted in the wavelength range of 240 nm to 420 nm, is optically filtered and then converted to an electrical signal by a UV detector, for example, a photomultiplier tube.

The response of the analyser is proportional to the number of SO_2 molecules in the reaction chamber. Therefore, temperature and pressure either need to be kept constant, or, if variation of these parameters occurs, the measured values need to be corrected.

The concentration of sulphur dioxide is directly measured in volume/volume units (if the analyser is calibrated using a volume/volume standard). The final results for reporting are expressed in $\mu\text{g}/\text{m}^3$ 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 system 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 system. However, the influence of these components on the test results shall be minimised by proper maintenance.

A designated 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 ENV 13005.

5.4 Field operation and quality control

Prior to the installation and operation of a type-approved analyser at a monitoring station, an expanded uncertainty calculation shall be performed with the actual values of the performance, obtained during the type approval tests, and the site-specific conditions at that monitoring station. This calculation shall be used to demonstrate that the type-approved analyser meets the requirements for all applicable limit values under the actual conditions present at that specific monitoring station.

After the installation of the approved analyser at the monitoring station, its correct functioning shall be tested.

Requirements for quality assurance and quality control are given for the operation and maintenance of the sampling system, as well as for the analyser, to ensure that the uncertainty of subsequent measurement results obtained in the field is not compromised.

6 Sampling

6.1 General

Depending on the installation of the ultraviolet fluorescence analyser at a monitoring station, a single sampling line for the analyser may be chosen. Alternatively, sampling can take place from a sampling system consisting of a common sampling inlet with a sampling manifold to which other analysers and equipment may be attached. Conditions and layout of the sampling system will contribute to the uncertainty of the measurement; to minimise this contribution to the expanded uncertainty, requirements for the sampling equipment are given in the following sub-clauses.

NOTE In Annex B, different arrangements of the sampling equipment are schematically presented.

The following factors may, through decrease in the concentration of sulphur dioxide, contribute to the uncertainty of the measurement when considering the sampling as an integral part of the measurement:

- loss of sulphur dioxide in the sampling system;
- loss of sulphur dioxide in the particle filter.

These factors are recognised to be relevant, but currently cannot be quantified for lack of appropriate assessment methods. As a consequence, the contributions of these factors are not considered in the uncertainty assessment applied in this standard. The effect of these factors is minimised through minimum requirements (see 6.3) and application of appropriate QA/QC measures (see 9.4 to 9.6) and maintenance (see 9.7).

6.2 Sampling location

The location where the ambient air shall be sampled and analysed is not specified as this depends strongly on the category of a monitoring station (such as measurements taken in e.g. a rural area or background area). Guidance and criteria on sampling points on a micro scale are given in Annex III of Directive 2008/50/EC [1].

6.3 Sampling system

6.3.1 Construction

The sampling system shall include a sampling inlet and may include the following components:

- a sampling line or manifold;
- a particle filter placed between the sampling line or manifold and the inlet of the analyser;
- a sampling pump in case a sampling manifold is used.

The sample inlet shall be constructed in such a way that ingress of rainwater into the sampling line or manifold is prevented. The sampling line or manifold shall be as short as practical to minimise the residence time.

In the case where a sampling manifold is used, an additional pump is necessary with sufficient capacity to fulfil the sampling requirements stated in the previous sub-clauses (see also 6.5 and Annex B).

The material of the sample inlet as well as the sampling line or manifold can influence the composition of the sample. In practice, the best materials, such as polytetrafluoroethylene (PTFE), perfluoro-ethylene-propylene (FEP), borosilicate glass or stainless steel, shall be used. The influence of the material of the sampling inlet and line or manifold on the measured concentrations of sulphur dioxide due to losses shall be < 2,0 %.

NOTE This value can be achieved when the quality assurance and quality control requirements (see Clause 9) are followed.

The sampling line or manifold may be moderately heated to avoid condensation. Condensation may occur in the case of high ambient temperature and/or humidity.