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Stationary source emissions — Determination of volume concentration of oxygen — Standard reference method: Paramagnetism

National foreword

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European foreword

This document (EN 14789:2017) has been prepared by Technical Committee CEN/TC 264 “Air quality”, the secretariat of which is held by DIN.

This document supersedes EN 14789:2005.

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of an identical text or by endorsement, at the latest by July 2017, and conflicting national standards shall be withdrawn at the latest by July 2017.

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.

Annex E provides details of significant technical changes between this document and the previous edition.

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, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

1 Scope

This European Standard specifies the standard reference method (SRM) based on the paramagnetic principle for the determination of the oxygen concentrations in flue gases emitted to the atmosphere from ducts and stacks. It includes the sampling and the gas conditioning system as well as the analyser.

This European Standard specifies the performance characteristics to be determined and the performance criteria to be fulfilled by portable automated measuring systems (P-AMS) based on this measurement method. It applies to periodic monitoring and the calibration or control of automated measuring systems (AMS) permanently installed on a stack, for regulatory or other purposes.

This European Standard specifies criteria for demonstration of equivalence of an alternative method (AM) to the SRM by application of EN 14793:2017.

This European Standard has been validated during field tests on waste incineration, co-incineration and large combustion plants and on a recognized test bench. It has been validated for sampling periods of 30 min in the range from 3 % to 21 %. Oxygen concentration values, expressed as volume concentrations, are used to allow results of emission measurements to be standardised to the oxygen reference concentration and dry gas conditions required e.g. by EU Directive 2010/75/EC on industrial emissions.

NOTE The characteristics of installations, the conditions during field tests and the values of repeatability and reproducibility in the field are given in Annex A.

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 14793:2017, *Stationary source emission — Demonstration of equivalence of an alternative method with a reference method*

EN 15259:2007, *Air quality - Measurement of stationary source emissions - Requirements for measurement sections and sites and for the measurement objective, plan and report*

EN 15267-4:2017, *Air quality — Certification of automated measuring systems — Part 4: Performance criteria and test procedures for automated measuring systems for periodic measurements of emissions from stationary sources*

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

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.

NOTE In this European Standard, the volume concentration of oxygen is expressed in percent.

3.1
standard reference method
SRM
reference method prescribed by European or national legislation

[SOURCE: EN 15259:2007]

3.2
reference method
RM
measurement method taken as a reference by convention, which gives the accepted reference value of the measurand

Note 1 to entry: A reference method is fully described.

Note 2 to entry: A reference method can be a manual or an automated method.

Note 3 to entry: Alternative methods can be used if equivalence to the reference method has been demonstrated.

[SOURCE: EN 15259:2007]

3.3
measurement method
method described in a written procedure containing all the means and procedures required to sample and analyse, namely field of application, principle and/or reactions, definitions, equipment, procedures, presentation of results, other requirements and measurement report

[SOURCE: EN 14793:2017]

3.4
alternative method
AM
measurement method which complies with the criteria given by this European Standard with respect to the reference method

Note 1 to entry: An alternative method can consist of a simplification of the reference method.

[SOURCE: EN 14793:2017]

3.5
measuring system
set of one or more measuring instruments and often other devices, including any reagent and supply, assembled and adapted to give information used to generate measured quantity values within specified intervals for quantities of specified kinds

[SOURCE: JCGM 200:2012]

3.6
automated measuring system
AMS

entirety of all measuring instruments and additional devices for obtaining a result of measurement

Note 1 to entry: Apart from the actual measuring device (the analyser), an AMS includes facilities for taking samples (e.g. probe, sample gas lines, flow meters and regulator, delivery pump) and for sample conditioning (e.g. dust filter, pre-separator for interferents, cooler, converter). This definition also includes testing and adjusting devices that are required for functional checks and, if applicable, for commissioning.

Note 2 to entry: The term “automated measuring system” (AMS) is typically used in Europe. The term “continuous emission monitoring system” (CEMS) is also typically used in the UK and USA.

[SOURCE: EN 15267-4:2017]

3.7
portable automated measuring system
P-AMS

automated measuring system which is in a condition or application to be moved from one to another measurement site to obtain measurement results for a short measurement period

Note 1 to entry: The measurement period is typically 8 h for a day.

Note 2 to entry: The P-AMS can be configured at the measurement site for the special application but can be also set-up in a van or mobile container. The probe and the sample gas lines are installed often just before the measurement task is started.

[SOURCE: EN 15267-4:2017]

3.8
calibration

set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring method or measuring system, and the corresponding values given by the applicable reference

Note 1 to entry: In case of automated measuring systems (AMS) permanently installed on a stack the applicable reference is the standard reference method (SRM) used to establish the calibration function of the AMS.

Note 2 to entry: Calibration should not be confused with adjustment of a measuring system.

3.9
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: The adjustment can be made directly on the instrument or using a suitable calculation procedure.

3.10
span gas

test gas used to adjust and check a specific point on the response line of the measuring system

3.11
measurand

particular quantity subject to measurement

[SOURCE: EN 15259:2007]

Note 1 to entry: The measurand is a quantifiable property of the stack gas under test, for example mass concentration of a measured component, temperature, velocity, mass flow, oxygen content and water vapour content.

3.12
interference

negative or positive effect upon the response of the measuring system, due to a component of the sample that is not the measurand

3.13
influence quantity

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

Note 1 to entry: Influence quantities are e.g. presence of interfering gases, ambient temperature or pressure of the gas sample.

3.14
ambient temperature

temperature of the air around the measuring system

3.15
measurement site

place on the waste gas duct in the area of the measurement plane(s) consisting of structures and technical equipment, for example working platforms, measurement ports, energy supply

Note 1 to entry: Measurement site is also known as sampling site.

[SOURCE: EN 15259:2007]

3.16
measurement plane

plane normal to the centreline of the duct at the sampling position

Note 1 to entry: Measurement plane is also known as sampling plane.

[SOURCE: EN 15259:2007]

3.17
measurement port

opening in the waste gas duct along the measurement line, through which access to the waste gas is gained

Note 1 to entry: Measurement port is also known as sampling port or access port.

[SOURCE: EN 15259:2007]

3.18
measurement line

line in the measurement plane along which the measurement points are located, bounded by the inner duct wall

Note 1 to entry: Measurement line is also known as sampling line.

[SOURCE: EN 15259:2007]

3.19
measurement point

position in the measurement plane at which the sample stream is extracted or the measurement data are obtained directly

Note 1 to entry: Measurement point is also known as sampling point.

[SOURCE: EN 15259:2007]

3.20
performance characteristic

one of the quantities (described by values, tolerances, range) assigned to equipment in order to define its performance

3.21
response time

duration between the instant when an input quantity value of a measuring instrument or measuring system is subjected to an abrupt change between two specified constant quantity values and the instant when a corresponding indication settles within specified limits around its final steady value

Note 1 to entry: By convention time taken for the output signal to pass from 0 % to 90 % of the final variation of indication.

3.22
short-term zero drift

difference between two zero readings at the beginning and at the end of the measurement period

3.23
short-term span drift

difference between two span readings at the beginning and at the end of the measurement period

3.24
lack of fit

systematic deviation, within the measurement range, between the measurement result obtained by applying the calibration function to the observed response of the measuring system measuring test gases and the corresponding accepted value of such test gases

Note 1 to entry: Lack of fit can be a function of the measurement result.

Note 2 to entry: The expression “lack of fit” is often replaced in everyday language by “linearity” or “deviation from linearity”.

3.25
repeatability in the laboratory

closeness of the agreement between the results of successive measurements of the same measurand carried out under the same conditions of measurement

Note 1 to entry: Repeatability conditions include:

- same measurement method;
- same laboratory;
- same measuring system, used under the same conditions;
- same location;
- repetition over a short period of time.

Note 2 to entry: Repeatability can be expressed quantitatively in terms of the dispersion characteristics of the results.

Note 3 to entry: In this European Standard the repeatability is expressed as a value with a level of confidence of 95 %.

3.26
repeatability in the field

closeness of the agreement between the results of simultaneous measurements of the same measurand carried out with two sets of equipment under the same conditions of measurement

Note 1 to entry: These conditions include:

- same measurement method;
- two sets of equipment, the performances of which are fulfilling the requirements of the measurement method, used under the same conditions;
- same location;
- implemented by the same laboratory;
- typically calculated on short periods of time in order to avoid the effect of changes of influence parameters (e.g. 30 min).

Note 2 to entry: Repeatability can be expressed quantitatively in terms of the dispersion characteristics of the results.

Note 3 to entry: In this European Standard, the repeatability under field conditions is expressed as a value with a level of confidence of 95 %.

3.27
reproducibility in the field
closeness of the agreement between the results of simultaneous measurements of the same measurand carried out with several sets of equipment under the same conditions of measurement

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

- same measurement method;
- several sets of equipment, the performances of which are fulfilling the requirements of the measurement method, used under the same conditions;
- same location;
- implemented by several laboratories.

Note 2 to entry: Reproducibility can be expressed quantitatively in terms of the dispersion characteristics of the results.

Note 3 to entry: In this European Standard, the reproducibility under field conditions is expressed as a value with a level of confidence of 95 %.

3.28
residence time in the measuring system
time period for the sampled gas to be transported from the inlet of the probe to the inlet of the measurement cell

3.29
uncertainty
parameter associated with the result of a measurement, that characterises the dispersion of the values that could reasonably be attributed to the measurand

3.30
standard uncertainty
u
uncertainty of the result of a measurement expressed as a standard deviation

3.31
combined uncertainty
u_c
standard uncertainty attached to the measurement result calculated by combination of several standard uncertainties according to the principles laid down in ISO/IEC Guide 98-3 (GUM)

3.32
expanded uncertainty
U
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

$$U = k \times u_{\mathrm{c}}$$

Note 1 to entry: In this European Standard, the expanded uncertainty is calculated with a coverage factor of $k = 2$, and with a level of confidence of 95 %.

Note 2 to entry: The expression overall uncertainty is sometimes used to express the expanded uncertainty.

3.33

uncertainty budget

calculation table combining all the sources of uncertainty according to EN ISO 14956 or ISO/IEC Guide 98-3 in order to calculate the combined uncertainty of the method at a specified value

4 Symbols and abbreviations

4.1 Symbols

For the purposes of this document, the following symbols apply.

$A(t_0)$	(result given by the analyser after adjustment at t_0 at span point – result given by the analyser after adjustment at t_0 at zero point) / (calibration gas concentration at span point – calibration gas concentration at zero point)
$B(t_0)$	result given by the analyser after adjustment at t_0 at zero point
C	measured volume concentration
C_{corr}	measured volume concentration corrected for drift
$\mathrm{Drift}(A)$	{[(result given by the analyser during the drift check at t_{end} at span point – result given by the analyser during the drift check at t_{end} at zero point) / (calibration gas concentration at span point – calibration gas concentration at zero point)] – $A(t_0)$} / ($t_{\mathrm{end}} - t_0$)
$\mathrm{Drift}(B)$	(result given by the analyser during the drift check at t_{end} at zero point – result given by the analyser after adjustment at t_0 at zero point) / ($t_{\mathrm{end}} - t_0$)
k	coverage factor
s_R	reproducibility standard deviation
$s_{r,\mathrm{limit}}$	maximum allowable repeatability standard deviation
t	time
t_0	time of adjustment
t_{end}	time of check for drift at the end of the measurement period
u	standard uncertainty
u_{c}	combined uncertainty
U	expanded uncertainty

4.2 Abbreviated terms

For the purposes of this document, the following abbreviated terms apply.

AM	alternative method
AMS	automated measuring system
P-AMS	portable automated measuring system
PTFE	polytetrafluoroethene
PFA	perfluoroalkoxy
SRM	standard reference method

5 Principle

5.1 General

This European Standard describes the standard reference method (SRM), based on the paramagnetism principle for sampling and determining oxygen concentration in flue gases emitted to atmosphere from ducts and stacks. The specific components and the requirements for the sampling system and the analyser are described in Clause 6 and Clause 7. A number of performance characteristics, together with associated performance criteria are specified for the analyser. These performance characteristics determined according to EN 15267-4:2017. and the expanded uncertainty of the method shall meet the performance criteria given in this European Standard. Requirements and recommendations for quality assurance and quality control are given in Clause 9 for measurements in the field.

5.2 Measuring principle

The paramagnetic method is based on the principle that oxygen molecules are strongly attracted to a magnetic field. This property, known as paramagnetism, can be used for the selective measurement of oxygen in flue gases where the other constituents are either slightly or non-paramagnetic. The magnetic susceptibility or degree of magnetisation produced in a gas sample by a magnetic field is inversely proportional to its absolute temperature. A gas sample containing oxygen, when exposed to the combined effect of a magnetic gradient in a confined space, shall be constrained to flow in the direction of the magnetic field. The magnitude of this flow, other factors being equal, is dependent on the oxygen concentration in the gas sample induced flow. A number of devices, described in Clause 6 have been developed to measure the paramagnetically induced flow (see in Annex B an example of assessment of compliance of paramagnetic method for oxygen with given uncertainty requirements).

Paramagnetic analysers are associated to an extractive sampling system and a gas conditioning system. A sample of gas is taken from the stack with a sampling probe and conveyed to the analyser through a sample gas line and suitable gas conditioning system. The values from the analyser are recorded and/or stored by means of electronic data processing systems.

6 Description of the measuring system

6.1 General

A volume of flue gas (see 9.2) is extracted from the emission source for a fixed period of time at a controlled flow rate. A filter removes the dust in the sampled volume before the sampled gas is conditioned and passed to the analyser.

Different sampling and conditioning configurations are available in order to avoid uncontrolled water vapour condensation in the measuring system.

Possible configurations are:

- configuration 1: removal of water vapour by condensation using a cooling system;
- configuration 2: removal of water vapour through elimination within a permeation drier.

Schematic diagrams of typical measuring systems are shown in Annex C.

They require that the user shall check that the dew point temperature or the outlet temperature of the cooling system is lower or equal to 4 °C at the inlet of the analyser. The user may correct the results for the remaining water content in order to report results on a dry basis (refer to the table of Annex B in EN 14790:2017).

It is important that all components of the sampling system upstream of the analyser are made of materials that do not react with or absorb oxygen.

Conditions and layout of the sampling and sample gas conditioning system contribute to the combined uncertainty of the measurement. In order to minimize this contribution to the combined measurement uncertainty, performance criteria for the sampling system and sampling conditions are given in 6.2 and Clause 7.

Some other sample gas conditioning systems may exist and could be acceptable, provided they fulfil the requirements of this European Standard and have been validated with success during the certification process. For example, some systems put gas in depression using a simple sonic nozzle in the collection probe in order to create a partial vacuum (between 50 hPa and 100 hPa absolute pressure) so that the head of collection and the sample gas line does not need to be heated and water vapour condensation is avoided.

6.2 Sampling and sample gas conditioning system

6.2.1 Sampling probe

In order to reach the measurement points in the measurement plane, probes of different lengths and inner diameters may be used. The design and configuration of the probe used shall ensure the residence time of the sample gas within the probe is minimised in order to reduce the response time of the measuring system.

NOTE 1 The probe can be marked before sampling in order to demonstrate that the measurement points in the measurement plane have been reached.

NOTE 2 A sealable connection can be installed on the probe in order to introduce test gases for adjustment.

6.2.2 Filter

The filter and filter holder shall be made of an inert material (e.g. ceramic or sinter metal filter with an appropriate pore size). It shall be heated above the water or acid dew point temperature, whichever is the greater. The particle filter shall be changed or cleaned periodically depending on the dust loading in the measurement plane.

NOTE Overloading of the particle filter can increase the pressure drop in the sample gas line.

6.2.3 Sample gas line

The sample gas line shall be heated up to the conditioning system. It shall be made of a suitable corrosion resistant material (e.g. stainless steel, borosilicate glass, ceramic or titanium; PTFE or PFA is only suitable for flue gas temperature lower than 200 °C).

6.2.4 Sample gas cooler or permeation drier

The sample-gas cooler or permeation drier shall be used before the gas enters the analyser in order to separate water vapour from the flue gas.

Due to ammonium salts deposition on the permeation tube, the permeation system shall not be used when the NH_3 concentration is outside the range specified by the manufacturer.

NOTE The measured oxygen concentration, given by these sampling configurations, can be considered to be dry. However, the user can correct the results for the remaining water (refer to the Table of Annex B in EN 14790:2017).

6.2.5 Sample gas pump

When a pump is not an integral part of the paramagnetic analyser, an external pump is necessary to draw the sample gas through the apparatus. It shall be capable of operating according to the specified flow requirements of the manufacturer of the analyser and pressure conditions required for the reaction chamber. The pump shall be resistant to corrosion and consistent with the requirements of the analyser to which it is connected. The whole sampling system associated to the analyser, including the pump, has to meet the criterion in Table 1 related to the influence of gas pressure.

NOTE The quantity of sample gas required can vary between 15 l/h and 500 I/h, depending upon the analyser and the expected response time.

6.2.6 Secondary filter

The secondary filter is used to separate fine dust, with a pore size of 1 µm to 2 µm. It may be made of glass-fibre, sintered ceramic, stainless steel or PTFE.

NOTE No additional secondary filter is necessary when they are part of the analyser itself.

6.2.7 Flow controller and flow meter

This apparatus sets the required sample gas flow. A corrosion resistant material shall be used. The sample gas flow rate into the instrument shall be maintained according to the analyser manufacturer's requirements. A controlled pressure drop across restrictors is usually employed to maintain flow rate control into the analyser.

NOTE No additional flow controller or flow meter is necessary when they are part of the analyser itself.

6.3 Different variants of the paramagnetism principle

Several variants of application of the paramagnetism principle are available. Some of them are described below:

a) Thermo-magnetic

   Two separate chambers (reference and measuring chambers) are equipped with thermo-sensitive resistors, which form an assembly with a Wheatstone bridge. The measuring chamber is located in a magnetic field while the reference is not. When the concentration of oxygen increases, the flow in the measuring chamber is greater than the flow in the reference chamber. This creates a differential cooling effect on the resistors. The equilibrium of the Wheatstone bridge is restored by an increase of an electric current in the resistors and this current is proportional to the oxygen concentration.

b) Magneto-mechanic

In one measuring chamber, two permanent magnets create a non-homogenous magnetic field in which a very light float is suspended. When the gas is injected into the chamber, the oxygen molecules are attracted by the magnetic field. This in turn creates a differential partial pressure, which then alters the position of the float. An electromagnetic field is then applied to compensate for this change, while the strength of the applied compensatory field is proportional to the concentration of oxygen.

c) Magneto-pneumatic

The sampled gas and a reference gas circulate within an electromagnet. Each gas is subject to a modification of pressure that is proportional to the oxygen concentration. This pressure difference then alters the position of the membrane of a capacitor or alters the flow measured by a micro flow sensor. The variation in capacitance allows the conversion of the pressure signal to an electric signal, which in turn is proportional to the partial pressure of oxygen.

7 Performance characteristics of the SRM

Table 1 gives an overview of the performance characteristics of the whole measurement method including the analyser and the sampling and sample gas conditioning system. These performance characteristics shall be determined in a general performance test according to the test procedures described in EN 15267-4:2017 by an independent test laboratory accredited or recognized by the competent authorities for the implementation of test procedures of EN 15267-4:2017.

The independent test laboratory shall check the conformity of the analyser with its sampling and sample gas conditioning system to fulfil the performance criterion attached to each performance characteristic. The maximum allowable deviations as absolute values of the measured values are given as volume concentrations (volume fractions) or as percentages of the upper limit of the range.

Table 1 — Performance characteristics of the SRM and associated performance criteria

Performance characteristic	Performance criterion	Performance characteristic to be included in calculation of combined uncertainty
Response time	≤ 200 s	
Repeatability standard deviation at zero point	≤ 0,20 % [a]	X [b]
Repeatability standard deviation at span point	≤ 0,20 % [a]	X [b]
Reproducibility standard deviation	≤ 0,20 % [a]	X [b]
Lack of fit	≤ 0,30 % [a]	X
Short-term zero drift	≤ 0,20 % [a, c]	X
Short-term span drift	≤ 0,20 % [a, c]	X
Influence of ambient temperature change from 5 °C to 25 °C and from 40 °C to 20 °C at zero point	≤ 0,50 % [a, c]	X
Influence of ambient temperature change from 5 °C to 25 °C and from 40 °C to 20 °C at span point	≤ 0,50 % [a, c]	X
Influence of sample gas pressure at span point, for a pressure change Δp of 3 kPa	≤ 0,20 % [a]	X

Influence of sample gas flow on extractive P-AMS for a given specification by the manufacturer	≤ 0,20 % [a]	X
Influence of vibration	≤ 0,20 % [a]	
Influence of voltage, at −15 % below and at +10 % above nominal supply voltage	≤ 0,20 % [a]	X
Cross-sensitivity	≤ 0,40 % [a, d]	X
Leakage in the sample gas line and sample gas conditioning system	≤ 2,0 % of the measured value	X [e]

[a] Values are given as percentage values of oxygen volume concentration (volume fraction).

[b] The repeatability in the laboratory or the reproducibility in the field shall be used, whichever is greater. If the repeatability in the laboratory is used, only one of both values shall be included in the calculation: the first possibility is to choose the repeatability standard deviation got from laboratory tests corresponding to the closest concentration to the actual concentration in stack, or the higher (relative) standard deviation of repeatability independently of the concentration measured in stack.

[c] Consider either a combination of drift effect in the laboratory and effect of temperature in the laboratory or drift in the field whatever is the greatest, because drift in the field combines mainly intrinsic drift of the P-AMS and the drift due to temperature.

[d] The value of the maximum of algebraic sums of contributions to uncertainty producing positive effects on the result, (sum of contributions to uncertainty producing negative effects) shall be compared with the performance criterion.

[e] If the leak test is performed under severe conditions of depression, then leak can be considered as negligible in normal conditions of use.

8 Suitability of the measuring system for the measurement task

An uncertainty budget shall be established by the user to determine for which measurement range the analyser and its associated sampling and sample gas conditioning system fulfil the requirements for a maximum allowable expanded uncertainty.

The relative expanded uncertainty shall not exceed 6,0 % of the measured value expressed on dry basis or 0,3 % as a volume concentration.

The measurement range that could be covered by the measuring system can be extended if the user demonstrates that the uncertainty with the actual variation range of influence quantities and values of interferents at a particular plant is lower than the maximum allowable expanded uncertainty.

Table 1 indicates which performance characteristics have to be included in the calculation of the combined uncertainty.

The principle of calculation of the combined uncertainty is based on the law of propagation of uncertainty laid down in ISO/IEC Guide 98-3 (GUM):

— determine the standard uncertainties attached to the performance characteristics to be included in the calculation of the uncertainty budget according to ISO/IEC Guide 98-3;

— calculate the uncertainty budget by combining all the standard uncertainties according to ISO/IEC Guide 98-3, including the uncertainty of the calibration gas

— values of standard uncertainty that are less than 5 % of the maximum standard uncertainty may be neglected;

— calculate the combined uncertainty of the measured value,.

An example of an uncertainty budget is given in Annex B.

9 Field operation

9.1 Measurement planning

Emission measurements at a plant shall be carried out such that the results are representative for the emissions from this plant and comparable with results obtained for other comparable plants. Therefore, measurements shall be planned in accordance with EN 15259.

Before carrying out any measurements, the purpose of the sampling and the sampling procedures shall be discussed with the plant personnel concerned. The nature of the plant process, e.g. steady-state or cyclic, can affect the sampling programme. If the process can be performed in a steady-state, it is important that this is maintained during sampling.

Dates, starting times, duration of survey and sampling periods as well as plant operating conditions during these periods shall be agreed with the plant management.

If no suitable location exists in the plant, and/or that measurements have been carried out during non-steady-state conditions of the plant, which leads to an increase of the uncertainty of the measurements, it shall be stated in the measurement report.

9.2 Sampling strategy

9.2.1 General

Sampling requires a suitable measurement section and measurement plane.

The measurement plane shall be easily reached from convenient measurement ports and a safe working platform (see EN 15259).

9.2.2 Measurement section and measurement plane

The measurement section and measurement plane shall meet the requirements of EN 15259.

9.2.3 Minimum number and location of measurement points

It is necessary to ensure that the gas concentrations measured are representative of the average conditions inside the waste gas duct. Measurements may be performed at one representative measurement point or at any measurement point, if the corresponding requirements on the distribution of the oxygen volume concentration specified in 8.3 of EN 15259:2007 are fulfilled. In all other cases the measurements shall be performed as grid measurements. In that case, EN 15259 specifies the minimum number of measurement points to be used and the location in the measurement plane for circular and rectangular ducts.

9.2.4 Measurement ports and working platform

Measurement ports shall be provided for access to the measurement points selected in accordance with EN 15259.

Examples of suitable measurement ports are given in EN 15259.

For safety and practical reasons, the working platform shall comply with the requirements of EN 15259.

9.3 Choice of the measuring system

The following flue gas characteristics shall be determined, before the field campaign, so that an appropriate analyser and sampling configuration (including conditioning unit) can be adopted:

- flue gas moisture content and dew point;
- temperature of exhaust gases;

- dust loading;
- expected concentration of potentially interfering substances,

The sample gas line should be as short as possible to avoid long response times. If necessary, a bypass pump together with a heated filter appropriate to the dust loading shall be used.

The laboratory shall verify that the analyser is functioning in accordance with the required performance criteria fixed in this European Standard, and that the sample gas line and conditioning unit are in good operational conditions before conducting field measurements.

9.4 Setting of the measuring system on site

9.4.1 General

The complete measuring system, including the sampling system, the sample gas conditioning system and the analyser, shall be connected according to the manufacturer's instructions. The nozzle of the sampling probe placed at the measurement points in the duct (see 9.2).

The sample gas conditioning system, sampling probe, filter, connection tube and analyser shall be stabilized at the required temperature. At the same time, a constant pressure shall be achieved in the measuring chamber of the analyser.

After pre-heating, the flow passing through the sampling system and the analyser shall be adjusted to the chosen flow rate to be used during measurement.

When both the analyser and sampling system have been set-up, and before zero and span adjustments, the proper functioning of the analyser and sampling system shall be checked.

Any data recording and data processing system used shall be adapted to the measurement objective and to the response time of the measuring system; generally, data are recorded at least every 60 s.

9.4.2 Preliminary zero and span check and adjustments

9.4.2.1 Test gases

9.4.2.1.1 Zero gas

The zero gas shall be free of oxygen (oxygen volume concentration less than 0,05 %).

9.4.2.1.2 Span gases

The span gas used to adjust the analyser shall have a concentration of oxygen traceable to SI units. The expanded uncertainty on the analytical certificate of the span gas shall not exceed 2,0 %. Ambient air, the oxygen volume concentration of which is 20,9 %, may also be used for adjustment of the analyser (relative uncertainty 0,5 %). This air shall be clean and dried by passing through an appropriate agent (e.g. silica gel).

9.4.2.2 Adjustment of the analyser

At the beginning of the measurement period, zero and span gases or air shall be supplied to the analyser directly, without passing through the sampling system. Adjustments shall be made until the zero and span gas values are reached by the data sampling system and are stable:

- check and adjust the zero value;
- check and adjust the span value;
- in case of adjustments, check again zero to see if there is no significant change.

9.4.2.3 Check of the sampling system including the leak test

Before starting the measurement, check that there is no significant leakage in the sampling system by use of one of the two following procedures or any other relevant procedure validated during the general performance test (see Clause 7):

- Zero and span gases are supplied to the analyser through the sampling system, as close as possible to the nozzle (in front of the filter if possible). Differences between the readings at zero and at span point shall not exceed 2,0 % of the span value;

- Assemble the complete sampling system. Close the nozzle and switch on the pump. After reaching minimum pressure, read or measure the flow rate with an appropriate measuring device. The leak flow rate shall not exceed 2,0 % of the expected sample gas flow rate used during measurement.

NOTE The leak test can be carried out before the adjustment of the analyser.

9.4.3 Zero and span checks after measurement

At the end of the measurement period and at least once a day, zero and span checks shall be performed at the inlet of the analyser by supplying test gases. The results of these checks shall be documented. In case of deviation between checks after measurement and preliminary adjustments, values of deviation shall be indicated in the measurement report.

If the span or zero drifts are greater than 2,0 % of the span value, it is necessary to correct both for zero and span drifts (see in Annex D for a procedure of correction of data for drift effect).

The drift of zero and span shall be lower than 5,0 % of the span value; otherwise, the results shall be rejected.

The concentration C_{corr} corrected according to time t for the concentration C given by the analyser shall be calculated according to Formula (1):

$$C_{\mathrm{corr}} = \frac{C - \left(B\left(t_0\right) + \mathrm{Drift}\left(B\right) \times t\right)}{\left(A\left(t_0\right) + \mathrm{Drift}\left(A\right) \times t\right)} \quad (1)$$

where

$A(t_0)$ is (result given by the analyser after adjustment at t_0 at span point – result given by the analyser after adjustment at t_0 at zero point) / (calibration gas concentration at span point – calibration gas concentration at zero point);

$B(t_0)$ is the result given by the analyser after adjustment at t_0 at zero point;

Drift(A) is {[(result given by the analyser during the drift check at t_{end} at span point – result given by the analyser during the drift check at t_{end} at zero point) / (calibration gas concentration at span point – calibration gas concentration at zero point)] – $A(t_0)$} / ($t_{\mathrm{end}} - t_0$);

Drift(B) is (result given by the analyser during the drift check at t_{end} at zero point – result given by the analyser after adjustment at t_0 at zero point) / ($t_{\mathrm{end}} - t_0$);

$t_{\mathrm{end}} - t_0$ is the duration of the measurement period in minutes (duration between adjustment and check for drift at the end of the measurement period).

An example of a drift check and of a drift correction is given in Annex D.

10 Ongoing quality control

10.1 General

Quality control is critically important in order to ensure that the uncertainty of the measured values is kept within the stated limits during extended continuous monitoring periods in the field. This means that maintenance, as well as zero and span adjustment procedures, shall be followed, as they are essential for obtaining accurate and traceable data.

10.2 Frequency of checks

Table 2 shows the required minimum frequency of checks. The laboratory shall implement the relevant European Standards for the determination of performance characteristics.

Table 2 — Frequency of checks

Checks	Frequency	Action criteria
Cleaning or changing of particulate filters[a] at the sampling inlet and at the analyser inlet	Every campaign, if needed [a]	–
Leak test	Every campaign	As specified in 9.4.2.3
Zero and span adjustment	Every campaign	As specified in 9.4.2.2
Drift	Every campaign	As specified in 9.4.3
Regular maintenance of several parts of the analyser	As required by the manufacturer	As specified by the manufacturer
Lack of fit	At least every year and after repair of the analyser	As specified in Table 1

[a] The particle filter shall be changed periodically depending on the dust loading at the measurement site and the filter housing shall be cleaned. Overloading of the particle filter can increase the pressure drop in the sample gas line.

11 Expression of results

The measurement results shall be expressed as percentages by volume (volume concentration).

12 Equivalence of an alternative method

In order to show that an alternative method is equivalent to the standard reference method specified in this European Standard, follow the procedures described in EN 14793.

The maximum allowable repeatability standard deviation (see Formula (2)) and the reproducibility standard deviation (see Formula (3)) expressed as a volume concentration in percent for the standard reference method are:

$$s_{r,\mathrm{limit}}\left(C\right)=0{,}0119C+0{,}017\% \qquad (2)$$

$$s_{R}\left(C\right)=0{,}0136C-0{,}011\% \qquad (3)$$

where *C* is the measured volume concentration in percent.

13 Measurement report

The measurement report shall fulfil the requirements of EN 15259 and shall include the following information:

a) information about the personnel involved in the measurement;

b) description of the location of the measurement points in the measurement plane;

c) information about the analyser and description of the sampling and sample gas conditioning system;

d) operating range;

e) details of the quality and the concentration of the calibration gases used;

f) details on the adjustment performed before and after actual sampling

g) measurement results with sampling date, time and duration;

h) information on flue gas characteristics (temperature, velocity, moisture, pressure).

Annex A
(informative)

Validation of the method in the field

A.1 General

The method has been validated during six field tests, on waste incineration installations, co-incineration installations and large combustion plants. Each test was performed by at least four different European measuring teams originating from 10 countries.

A.2 Characteristics of installations

The following field tests were performed:

— 1st field test: INERIS bench-loop at Verneuil en Halatte (France); the bench-loop simulates combustion or waste incineration exhaust gases. Five teams took part in the 1st field test. Double measurements were not performed simultaneously but sequentially. Five different flue gas matrices were generated. Within each matrix, two sequential measurements were performed. Two additional sequential measurements were performed in flue gas matrices where the flue gas concentrations varied. There were a total of 12 measurements performed by all the teams.

— 2nd field test: waste incinerator in Denmark. Four teams took part to the field test and performed double measurements simultaneously. A total of 16 measurements was performed by all the teams:

— 3rd field test: waste incinerator in Italy. Four teams took part to the field test. Two pairs of two teams performed double measurements simultaneously and the four teams performed single measurements simultaneously. A total of six double measurements was performed by each pair of two teams while a total of 12 single measurements was performed by all teams.

— 4th field test: co-incinerator combined heat and power installation in Sweden. The fluidised bed boilers operate on fuel mixes of wood chips, demolition waste, peat and coal. Two pairs of two teams performed double measurements simultaneously and the four teams performed single measurements simultaneously. A total of six double measurements was performed by each pair of two teams while a total of 12 single measurements was performed by all the teams.

— 5th field test: co-incinerator cement plant in Germany. The fuel could be coal, heavy oil and secondary fuel (e.g. paper, plastics, textiles, and tires). Four teams took part to the field test and performed double measurements simultaneously. A total of 16 double measurements was performed by all the teams.

— 6th field test: coal fired power plant in Germany. Four teams performed their double measurements simultaneously. The total amount of double measurements performed by all teams was 12.

An overview of the flue gas characteristics is given in Table A.1.

Table A.1 — Flue gas characteristics during field tests

Field test	Installation	Fuel	Flue gas characteristics						
			T °C	O_2 %	NO_x mg/m³	SO_2 mg/m³	CO mg/m³	H_2O %	PM mg/m³
1	Power plant [a]	Natural gas	< 150	3 to 13	10 to 1 300	10 to 2 000	20 to 400	10 to 21	< 1
2	Waste incinerator	Municipal waste	90 to 110	8 to 11	180 to 250	25 to 250	5 to 15	13 to 19	1 to 5
3	Waste incinerator	Municipal waste	85 to 105	16 to 18	61 to 78	5 to 50	0 to 2	8 to 12	1 to 5
4	Co-incinerator	Wood, waste, coal	70 to 80	4 to 6	4 to 70	0 to 10	50 to 150	8 to 12	0 to 20
5	Co-incinerator	Coal, oil, waste	140 to 170	4 to 6	440 to 1060	60 to 170	260 to 740	23 to 26	5 to 10
6	Power plant	Coal	130 to 140	8,9 to 9,2	110 to 140	1 000 to 1 130	3 to 6	5,5 to 8	< 50

[a] Bench-loop: flue gas simulation.

A.3 Repeatability and reproducibility in the field

A.3.1 General

Repeatability standard deviation s_r and reproducibility standard deviation s_R are determined from data obtained during inter-laboratory tests at the plants described in A.2.

Repeatability standard deviation s_r (see Formula (A.1)) and repeatability in the field r (see Formula (A.2)) are calculated according to ISO 5725-2 and ISO 5725-6 from the results of the double measurements implemented by the same laboratory (see Table A.2):

$$s_\mathrm{r} = \sqrt{\frac{\sum \left(x_i - \overline{x} \right)^2}{n-1}} \tag{A.1}$$

$$r = \sqrt{2}\, t_{0{,}95;\mathrm{n}-1}\, s_\mathrm{r} \tag{A.2}$$

where

s_r is the repeatability standard deviation;

x_i is the i^{th} measured signal;

$\overline{x}$ is the average of the measured signals x_i;

$t_{0{,}95;n-1}$ is the student factor for a level of confidence of 95 % and a degree of freedom of n–1 with n the number of double measurements;

r is the repeatability in the field.

Reproducibility standard deviation s_R used to estimate the expanded uncertainty U (see Formula (A.3)) and reproducibility in the field R (see Formula (A.4)) are calculated according to ISO 5725-2 from the results of parallel measurements performed simultaneously by several laboratories (see Table A.3):

$$U = t_{0,95;np-1}\, s_R \tag{A.3}$$

$$R = \sqrt{2}\, t_{0,95;np-1}\, s_R \tag{A.4}$$

where

U	is an estimate of the expanded uncertainty;
s_R	is the reproducibility standard deviation;
$t_{0,95;np-1}$	is the student factor for a level of confidence of 95 % and a degree of freedom of np–1 with n the number of measurements and p the number of laboratories;
R	is the reproducibility in the field.

A.3.2 Repeatability

Table A.2 — Repeatability in the field

Field test	**Volume concentration**		**Number of teams**	**Number of double measurements**	**Repeatability standard deviation** s_r	**Repeatability** r	
	Range %	Average %			%	absolute %	relative %
1A		11,2	5	2	0,018	0,058	0,5
1B		12,6	5	2	0,018	0,058	0,5
1C		9,2	5	2	0,017	0,054	0,6
1D		6,1	5	2	0,015	0,048	0,8
1E		3,1	5	2	0,013	0,042	1,4
1F	6,8 to 7,2	7,1	5	2	0,016	0,050	0,7
2	8,5 to 10,5	9,2	4	16	0,09	0,28	3,1
3	16,4 to 17,7	17,0	4	12	0,19	0,58	3,4
4	4,5 to 5,3	4,8	4	12	0,04	0,13	2,7
5	4,3 to 6,0	4,9	3	12	0,05	0,14	2,9
6	8,9 to 9,2	9,1	4	12	0,08	0,24	2,7

The following functions expressed as volume concentrations in percent were determined:

$$s_r(C) = 0{,}0119C + 0{,}01\% \tag{A.5}$$

$$s_{r,\text{limit}}(C) = 0{,}0143C + 0{,}012\% \tag{A.6}$$

$$r(C) = 0{,}0337C + 0{,}028\% \tag{A.7}$$

where *C* is the oxygen volume concentration expressed in percent.

A.3.3 Reproducibility

Table A.3 — Reproducibility in the field

Field test	**Volume concentration**		**Number of teams**	**Number of double measurements**	**Reproducibility standard deviation** s_R	**Estimate of expanded uncertainty** U	**Reproducibility** R	
	Range %	Average %			%	%	absolute %	relative %
1A		11,2	5	2	0,058	0,13	0,18	1,6
1B		12,6	5	2	0,06	0,13	0,19	1,5
1C		9,2	5	2	0,06	0,13	0,19	2,1
1D		6,1	5	2	0,041	0,093	0,13	2,1
1E		3,1	5	2	0,027	0,061	0,09	2,8
1F	6,8 to 7,2	7,1	5	2	0,12	0,27	0,39	5,4
2	8,5 to 10,5	9,3	4	16	0,11	0,23	0,33	3,5
3	16,4 to 17,7	17	4	12	0,22	0,49	0,69	4,1
4	4,7 to 5,2	5	4	12	0,13	0,29	0,41	8,2
5	4,3 to 6,0	4,9	3	12	0,06	0,13	0,18	3,8
6	8,9 to 9,2	9,1	4	12	0,12	0,26	0,37	4,1

The following functions expressed as volume concentrations in percent were determined:

$$s_R(C) = 0{,}0136\,C - 0{,}011\% \tag{A.8}$$

$$U(C) = 0{,}029\,C - 0{,}022\% \tag{A.9}$$

$$R(C) = 0{,}0385\,C - 0{,}031\% \tag{A.10}$$

where *C* is the oxygen volume concentration expressed in percent.

Annex B

(informative)

Example of assessment of compliance of paramagnetic method for oxygen with given uncertainty requirements

B.1 General

This informative annex gives an example of the calculation of the uncertainty budget established for configuration 1 to demonstrate compliance with given uncertainty requirements.

The following procedure for calculating the measurement uncertainty is based on the law of propagation of uncertainty as described in EN ISO 14956 or ISO/IEC Guide 98-3 (GUM). The individual standard uncertainties, the combined standard uncertainty and the expanded uncertainty are determined according to the requirements of EN ISO 14956 or ISO/IEC Guide 98-3.

B.2 Elements required for the uncertainty determinations

B.2.1 Model equation

In the first step, the model equation is established. The model equation describes the mathematical relationship between the measurand and all the parameters that influence the result of measurement. These parameters are called input quantities. It is necessary to clearly define the measurand and the input quantities.

The model function is used to calculate the result of measurement on the basis of the values assigned to the input quantities and to obtain the combined uncertainty of the result of measurement by application of the law of propagation of uncertainty.

The model equation for the oxygen volume concentration C_{O2} can be generally expressed as a sum of individual contributions C_i as given by Formula (B.1):

$$C_{\mathrm{O2}} = \sum_{i=1}^{N} C_i \tag{B.1}$$

The individual contributions C_i represents the volume concentration indicated by the analyser and additional corrections due to deviations caused by the performance characteristics of the analyser and the influence quantities such as given by Table B.1.

Table B.1 — Parameters and signal and uncertainty contributions

Number i	**Parameter**	**Signal contribution** C_i	**Uncertainty contribution** u_i
1	volume concentration indicated by the analyser	C_read	u_read
2	repeatability	C_r	$u_\mathrm{r} = s_\mathrm{r}$
3	lack of fit	C_lof	u_lof
4	short-term zero drift	$C_\mathrm{d,z}$	$u_\mathrm{d,z}$
5	short-term span drift	$C_\mathrm{d,s}$	$u_\mathrm{d,s}$
6	cross-sensitivity (interference)	C_i	u_i
7	influence of ambient temperature at zero [a]	$C_\mathrm{t,z}$	$u_\mathrm{t,z}$
8	influence of ambient temperature at span [a]	$C_\mathrm{t,s}$	$u_\mathrm{t,s}$
9	influence of sample gas pressure	C_p	u_p
10	influence of sample gas flow	C_f	u_f
11	influence of supply voltage	C_v	u_v
12	adjustment (calibration gas)	C_adj	u_adj

[a] Choose the most appropriate of both according to the level of concentration measured.

B.2.2 Combined uncertainty

The combined uncertainty $u_\mathrm{c}\left(C_\mathrm{O2}\right)$ of the oxygen volume concentration C_{O2} is obtained by application of the law of propagation of uncertainty on Formula (B.1) which leads to a quadratic summation of the uncertainty contributions u_i listed in Table B.1 as given by Formula (B.2):

$$u_\mathrm{c}\left(C_\mathrm{O2}\right)=\sqrt{\sum_{i=1}^{N}\left[\left(\frac{\partial C_\mathrm{O2}}{\partial C_i}\right)^2 u^2\left(C_i\right)\right]}=\sqrt{\sum_{i=1}^{N}u^2\left(C_i\right)}=\sqrt{\sum_{i=1}^{N}u_i^2} \qquad \text{(B.2)}$$

The uncertainty contributions u_i are quantified on the basis of available performance characteristics of the measurement system, data from the dispersion of repeated measurements, data describing the range of influence quantities or data provided in calibration certificates. If an uncertainty contribution is not expressed as a standard uncertainty (standard deviation), a conversion to a standard uncertainty is required.

B.2.3 Expanded uncertainty

In general, the uncertainty associated to a result of measurement is expressed as an expanded uncertainty which corresponds to the combined uncertainty multiplied by a coverage factor k. Since most of the values of the uncertainty components u_i are determined from test data, where the probability distribution of values is rectangular for most parameters and a normal distribution for a few parameters, a factor $k = 2{,}0$ is used for a level of confidence of approximately 95 % since the number of measurements to determine the uncertainty contributions and the associated number of degrees of freedom is sufficiently high or a rectangular distribution is assumed.

The expanded uncertainty $U\left(C_\mathrm{O2}\right)$ of the oxygen volume concentration C_O2 is determined using Formula (B.3):

$$U\left(C_{\text{O2}}\right) = 2{,}0\,u_{\text{c}}\left(C_{\text{O2}}\right) \tag{B.3}$$

B.2.4 Determination of uncertainty contributions in case of rectangular distributions

In the case of rectangular distributions the standard uncertainty u_i is calculated according to EN ISO 14956 by Formula (B.4):

$$u_i = \sqrt{\frac{\left(C_{i,\max} - C_{i,\text{adj}}\right)^2 + \left(C_{i,\min} - C_{i,\text{adj}}\right)\left(C_{i,\max} - C_{i,\text{adj}}\right) + \left(C_{i,\min} - C_{i,\text{adj}}\right)^2}{3}} \tag{B.4}$$

where

$C_{i,\min}$ is the minimum value of the average reading influenced by parameter *i*;

$C_{i,\max}$ is the maximum value of the average reading influenced by parameter *i*;

$C_{i,\text{adj}}$ is the value of the average reading during adjustment.

Formula (B.4) can be simplified in the following cases:

— if the value $C_{i,\text{adj}}$ is at the centre of the interval bounded by the maximum value $C_{i,\max}$ and the minimum value $C_{i,\min}$ of all values C_i, then the standard uncertainty u_i is given by Formula (B.5):

$$u_i = \frac{\left(C_{i,\max} - C_{i,\min}\right)}{\sqrt{12}} \tag{B.5}$$

If the absolute value of the deviation above and below the central value is expressed by ΔC_i (see Formula (B.6)), then the standard uncertainty u_i is given by Formula (B.7):

$$\left|C_{i,\max} - C_{i,\text{adj}}\right| = \left|C_{i,\min} - C_{i,\text{adj}}\right| = \Delta C_i \tag{B.6}$$

$$u_i = \frac{\Delta C_i}{\sqrt{3}} \tag{B.7}$$

— if the value of $C_{i,\text{adj}}$ is the same as either $C_{i,\min}$ or $C_{i,\max}$, then the standard uncertainty u_i is given by Formula (B.8):

$$u_i = \frac{\left(C_{i,\max} - C_{i,\min}\right)}{\sqrt{3}} \tag{B.8}$$

B.2.5 Determination of uncertainty contributions by use of sensitivity coefficients

The contribution C_i to the measured value caused by a parameter *i* can be calculated by use of the value X_i of the parameter and a corresponding sensitivity coefficient b_i of this parameter as shown by Formula (B.9):

$$C_i = b_i\,X_i \tag{B.9}$$

The contribution of a variation of the parameter to the total uncertainty of the measured values can be calculated from the range of values of the parameter in the considered application and the sensitivity coefficient of this parameter determined in the laboratory test of the analyser by use of Formula (B.10):

$$u_i = \left|b_i\right| u\left(X_i\right) \tag{B.10}$$

where

u_i is the uncertainty contribution to the total uncertainty of the measured values caused by a variation of the parameter *i*;

b_i is the sensitivity coefficient of the parameter *i*;

$u(X_i)$ is the standard uncertainty due to variation of the parameter *i*.

The variation of the parameter *i* can be converted to a standard uncertainty by use of Formulae (B.4) to (B.8).

B.3 Example of an uncertainty calculation

B.3.1 Site specific conditions

Table B.2 gives the specific conditions at the site, that is to say the values and the variation ranges of the influence quantities used in this example.

Table B.2 — Site specific conditions and values or ranges of influence parameters applied for the example

Specific condition	Value or range of influence parameters
Range of the oxygen analyser	0 % to 25 %
Mean oxygen volume concentration	12 %
Conditions in the field:	
ambient temperature during adjustment	285 K
fluctuations of ambient temperature during the measurement	283 K to 308 K
atmospheric pressure during adjustment	99 kPa
atmospheric pressure variation	99 kPa to 100 kPa
sample gas flow variation	60 l/h ± 5 l/h
voltage variation	230 V ± 10 V [a]
NO mass concentration variations	100 mg/m^3 to 150 mg/m^3
NO_2 mass concentration variations	5 mg/m^3 to 7,5 mg/m^3
CO_2 volume concentration variations	8 % to 15 %
Calibration gas (volume concentration of oxygen in nitrogen, without interferents)	20,0 % ± 0,2 % [b]

[a] This corresponds to an uncertainty of approximately 5 %.

[b] This corresponds to a relative uncertainty of 1 %.

B.3.2 Performance characteristics

Table B.3 gives the performance characteristics of the method used in this example. These parameters can have an influence on the response of the analyser and include the metrological performance of the analyser and the effect of influence quantities (environmental conditions like ambient temperature, voltage, pressure and chemical interferents).

Table B.3 — Performance characteristics, applied for the example

Performance characteristic	Performance criterion	Results of laboratory and field tests
Response time	≤ 200 s	120 s
Repeatability standard deviation in the laboratory at span	≤ 0,20 % [a]	0,10 % [a]
Lack of fit	≤ 0,30 % [a]	0,12 % [a]
Short-term zero drift	≤ 0,20 % [a]	0,10 % [a]
Short-term span drift	≤ 0,20 % [a]	0,10 % [a]
Influence of ambient temperature change from 5 °C to 25 °C and from 40 °C to 20 °C at zero point	≤ 0,50 % [a]	0,18 % [a]
Influence of ambient temperature change from 5 °C to 25 °C and from 40 °C to 20 °C at span point	≤ 0,50 % [a]	0,40 % [a]
Influence of sample gas pressure at span point, for a pressure change Δp of 3 kPa	≤ 0,20 % [a]	0,20 % [a]
Influence of sample gas flow on extractive P-AMS for flow change of 10 l/h	≤ 0,20 % [a]	0,20 % [a]
Influence of voltage, for a voltage change of 10 V	≤ 0,20 % [a]	0,08 % [a]
Cross-sensitivity	≤ 0,40 % [a]	–
NO (300 mg/m^3)	–	0,05 % [a]
NO_2 (30 mg/m^3)	–	0,02 % [a]
CO_2 (10 %)	–	–0,003 % [a]
Adjustment with calibration gases		1,0 % of the measured value

[a] expressed as volume concentration

B.3.3 Determination of the uncertainty contributions

The relevant uncertainty contributions are determined as follows:

a) Volume concentration indicated by the analyser

The uncertainty u_{read} related to the reading of the volume concentration is due to the resolution of the analyser and of the data acquisition. It can be considered as negligible.

b) Repeatability

The standard uncertainty u_r due to repeatability is equal to the repeatability standard deviation s_r calculated from the results of the repetitions of the measurements.

Several tests can be carried out at different concentrations but only one of the values is included in the calculation of the uncertainty budget e.g.

— the repeatability standard deviation corresponding to the closest concentration measured in stack;

- the highest (relative) repeatability standard deviation whatever is the concentration measured in stack.

c) Lack of fit

If $C_{\mathrm{lof,max}}$ is the maximum deviation between measured values and the corresponding values given by the linear regression achieved during the laboratory test, then it can be assumed that the lack of fit has an equal probability to take any value in the interval [$-C_{\mathrm{lof,max}}$; $+C_{\mathrm{lof,max}}$]. The standard uncertainty u_{lof} is calculated by application of a rectangular probability distribution according to Formula (B.11):

$$u_{\mathrm{lof}} = \frac{C_{\mathrm{lof,max}}}{\sqrt{3}} \tag{B.11}$$

d) Short-term zero drift

It can be assumed that the zero drift $C_{\mathrm{d,z}}$ has an equal probability to take any value in the interval [$-C_{\mathrm{d,z}}$; $+C_{\mathrm{d,z}}$]. The standard uncertainty $u_{\mathrm{d,z}}$ is calculated by application of a rectangular probability distribution according to Formula (B.12):

$$u_{\mathrm{d,z}} = \frac{C_{\mathrm{d,z}}}{\sqrt{3}} \tag{B.12}$$

e) Short-term span drift

It can be assumed that the span drift $C_{\mathrm{d,s}}$ has an equal probability to take any value in the interval [$-C_{\mathrm{d,s}}$; $+C_{\mathrm{d,s}}$]. The standard uncertainty $u_{\mathrm{d,s}}$ is calculated by application of a rectangular probability distribution according to Formula (B.13):

$$u_{\mathrm{d,s}} = \frac{C_{\mathrm{d,s}}}{\sqrt{3}} \tag{B.13}$$

f) Cross-sensitivity (interference)

Particularly with chemical components, deviations created by different interferents occur at the same time in the same proportion, i.e. the standard uncertainties of those substances are correlated. To avoid underestimation of additive effects and overestimation of effects by compensation, EN ISO 14956 recommends to determine the sum of all standard uncertainties of interferents with a positive impact on the measured value and the sum of all standard uncertainties of interferents with a negative impact on the measured value and to retain the highest sum as the representative value for all interferents.

Cross-sensitivity is tested in the laboratory test for one concentration of an interferent and is supposed to be proportional to the value of the interferent. The correction $C_{\mathrm{i},j}$ of the cross-sensitivity of an interferent j is also proportional to its variation $X_{\mathrm{i},j}$:

$$C_{\mathrm{i},j} = b_{\mathrm{i},j}\, X_{\mathrm{i},j} \tag{B.14}$$

where $b_{\mathrm{i},j}$ is the (constant) sensitivity coefficient of interferent j determined in the laboratory test.

In general, the concentration of the interferent in the calibration gas used for adjustment of the analyser is equal to zero.

If the maximum deviation $C_{\mathrm{ip},j}$ of the measured value caused by interferent j or the maximum value $X_{\mathrm{ip},j}$ of interferent j with a positive impact on the measured value are known only, then it can be assumed that a deviation caused by this interferent has an equal probability to take any value in the interval between zero and the maximum value. In this case the corresponding standard uncertainty $u_{\mathrm{ip},j}$ is given by Formula (B.15):

$$u_{\mathrm{ip},j} = \frac{C_{\mathrm{ip},j}}{\sqrt{3}} = \left| b_{\mathrm{ip},j} \right| \frac{X_{\mathrm{ip},j}}{\sqrt{3}} \tag{B.15}$$

If the value $X_{\mathrm{ip},j,\mathrm{adj}}$ during the adjustment of the analyser and the minimum and maximum value, $X_{\mathrm{ip},j,\mathrm{min}}$ and $X_{\mathrm{ip},j,\mathrm{max}}$, during the measurement period are known, then the standard uncertainty of interferent j with a positive impact on the measured value can be calculated on the basis of Formulae (B.4) and (B.10) by use of Formula (B.16):

$$u_{\mathrm{ip},j} = \left| b_{\mathrm{ip},j} \right| \sqrt{\frac{\left(X_{\mathrm{ip},j,\mathrm{max}} - X_{\mathrm{ip},j,\mathrm{adj}} \right)^2 + \left(X_{\mathrm{ip},j,\mathrm{min}} - X_{\mathrm{ip},j,\mathrm{adj}} \right)\left(X_{\mathrm{ip},j,\mathrm{max}} - X_{\mathrm{ip},j,\mathrm{adj}} \right) + \left(X_{\mathrm{ip},j,\mathrm{min}} - X_{\mathrm{ip},j,\mathrm{adj}} \right)^2}{3}} \tag{B.16}$$

If the value $X_{\mathrm{ip},j,\mathrm{adj}}$ during the adjustment of the analyser is zero, then the standard uncertainty of interferent j with a positive impact on the measured value is given by Formula (B.17):

$$u_{\mathrm{ip},j} = \left| b_{\mathrm{ip},j} \right| \sqrt{\frac{\left(X_{\mathrm{ip},j,\mathrm{max}} \right)^2 + \left(X_{\mathrm{ip},j,\mathrm{min}} \right)\left(X_{\mathrm{ip},j,\mathrm{max}} \right) + \left(X_{\mathrm{ip},j,\mathrm{min}} \right)^2}{3}} \tag{B.17}$$

The sum of all standard uncertainties of interferents with a positive impact on the measured value is calculated by Formula (B.18):

$$u_{\mathrm{ip}} = \sum_{j=1}^{p} u_{\mathrm{ip},j} \tag{B.18}$$

The standard uncertainties $u_{\mathrm{in},j}$ and the sum u_{in} of all standard uncertainties of interferents with a negative impact on the measured value are calculated in the same manner as the uncertainties of interferents with a positive impact:

$$u_{\mathrm{in}} = \sum_{j=1}^{n} u_{\mathrm{in},j} \tag{B.19}$$

The standard uncertainty u_{i} due to cross-sensitivity caused by correlated interferents is the maximum value of u_{ip} and u_{in}:

$$u_{\mathrm{i}} = \max\left(u_{\mathrm{ip}} ; u_{\mathrm{in}} \right) \tag{B.20}$$

Uncorrelated interferents are treated individually.

g) Influence quantities

Influence quantities such as ambient temperature, atmospheric pressure, sample gas flow and supply voltage are tested in the laboratory test for one value of the quantity and the effects of the influence quantities are supposed to be proportional to the value of the quantity. The correction C_i of the effect of an influence quantity i is also proportional to its variation X_i (see Formula (B.21):

$$C_i = b_i X_i \tag{B.21}$$

where b_i is the (constant) sensitivity coefficient of influence quantity i determined in the laboratory test.

The calculation of the standard uncertainty associated with the correction of deviations caused by variations of influence quantities depends on the value $X_{i,\mathrm{adj}}$ of the influence quantity during the adjustment of the analyser and the minimum and maximum value, $X_{i,\mathrm{min}}$ and $X_{i,\mathrm{max}}$, of the influence quantity during the measurement period. The uncertainty can be calculated by use of Formulae (B.4) to (B.10) either by use of the sensitivity coefficient and the deviations of the values of the influence quantity or directly from the deviations of the measured values.

h) Adjustment

The standard uncertainty u_{adj} is calculated from the uncertainty of the calibration gas. In general, the uncertainty given by manufacturer is an expanded uncertainty U_{cal}. For a level of confidence of 95 % the standard uncertainty u_{adj} is approximately given by Formula (B.22):

$$u_{\mathrm{adj}} = \frac{U_{\mathrm{cal}}}{2{,}0} \tag{B.22}$$

If the expanded uncertainty is expressed as a relative uncertainty $U_{\mathrm{cal,rel}}$ in form of a percentage value, the standard uncertainty of the adjustment at the oxygen volume concentration C_{O2} is given by Formula (B.23):

$$u_{\mathrm{adj}} = \frac{U_{\mathrm{cal,rel}}\, C_{\mathrm{O2}}}{2{,}0} \tag{B.23}$$

B.3.4 Results of uncertainty calculation

B.3.4.1 Standard uncertainties

Table B.4 presents the results of the uncertainty calculation based on the data presented in Table B.2 and Table B.3.

NOTE 1 In Table B.4 the absolute percentage value of the volume concentrations are presented without the unit % since the unit is extracted and presented in the table header. In case of relative percentage values such as “2 % of the range” the unit % is presented to reflect the performance criteria and test results presented in Table B.3.

NOTE 2 The unit % is identical to the factor 0,01, i.e. 2,0 % = 2,0 × 0,01 = 0,020.

Table B.4 — Results of uncertainty calculation

Parameter	Standard uncertainty	Value of standard uncertainty expressed as volume concentration in %
Repeatability standard deviation at span	u_r	$0{,}1\,\% \times 20{,}0 = 0{,}020$
Lack of fit	u_lof	$\frac{0{,}12}{\sqrt{3}} = 0{,}069$
Short-term zero drift	$u_\mathrm{d,z}$	$\frac{0{,}1}{\sqrt{3}} = 0{,}058$
Short-term span drift	$u_\mathrm{d,s}$	$\frac{0{,}1}{\sqrt{3}} = 0{,}058$
Influence of ambient temperature	$u_\mathrm{t,s}$	$\left\lvert\frac{0{,}40}{20}\right\rvert \times \sqrt{\frac{(308-285)^2 + (308-285)(283-285) + (283-285)^2}{3}} = 0{,}254$
Influence of sample gas pressure	u_p	$\left\lvert\frac{0{,}2}{3{,}0}\right\rvert \times \frac{(100-99)}{\sqrt{3}} = 0{,}038$
Influence of sample gas flow	u_f	$\left\lvert\frac{0{,}2}{10{,}0}\right\rvert \times \frac{5{,}0}{\sqrt{3}} = 0{,}058$
Influence of supply voltage	u_v	$\left\lvert\frac{0{,}08}{10}\right\rvert \times \frac{10}{\sqrt{3}} = 0{,}046$
Interferent NO	$u_\mathrm{i,NO}$	$\left\lvert\frac{0{,}05}{300}\right\rvert \times \sqrt{\frac{150^2 + 150 \times 100 + 100^2}{3}} = 0{,}021$
Interferent NO_2	$u_\mathrm{i,NO2}$	$\left\lvert\frac{0{,}02}{30}\right\rvert \times \sqrt{\frac{7{,}5^2 + 7{,}5 \times 5 + 5^2}{3}} = 0{,}004$
Interferent CO_2	$u_\mathrm{i,CO2}$	$\left\lvert\frac{-0{,}003}{10}\right\rvert \times \sqrt{\frac{8^2 + 8 \times 15 + 15^2}{3}} = \lvert -0{,}004 \rvert$
Uncertainty of calibration gas	u_adj	$\frac{1\,\% \times 12{,}0}{2{,}0} = 0{,}060$

B.3.4.2 Combined uncertainty

The sum of the standard uncertainties of interferents with a positive impact on the measured value is greater than the sum of all standard uncertainties of interferents with a negative impact on the measured value:

$$u_\mathrm{i} = \max\left(u_\mathrm{ip}\,; u_\mathrm{in}\right) = 0{,}025\,\%$$

According to Formula (B.2) the combined uncertainty of the oxygen volume concentration is given by:

$$u_\mathrm{c}\left(C_{\mathrm{O2}}\right)=\sqrt{\sum_{i=1}^{N}u_i^2}=\sqrt{u_\mathrm{r}^2+u_\mathrm{lof}^2+u_\mathrm{d,z}^2+u_\mathrm{d,s}^2+u_\mathrm{i}^2+u_\mathrm{tz}^2+u_\mathrm{p}^2+u_\mathrm{f}^2+u_\mathrm{v}^2+u_\mathrm{adj}^2}$$
$$=\sqrt{0{,}020^2+0{,}069^2+0{,}058^2+0{,}058^2+0{,}021^2+0{,}254^2+0{,}038^2+0{,}058^2+0{,}046^2+0{,}060^2}\,\%$$
$$=\sqrt{0{,}0874}\,\%=0{,}296\,\%$$

B.3.4.3 Expanded uncertainty

The expanded uncertainty of the oxygen volume concentration for a level of confidence of approximately 95 % (k = 2) is given by:

$$U\left(C_{\mathrm{O2}}\right)=0{,}59\,\%$$

The relative expanded uncertainty U_rel of the oxygen volume concentration for a level of confidence of approximately 95 % (k = 2) at an oxygen volume concentration of 12 % is given by:

$$U_\mathrm{rel}\left(C_{\mathrm{O2}}\right)=\frac{0{,}59\,\%}{12\,\%}=0{,}049=4{,}9\,\%$$

B.3.4.4 Evaluation of the compliance with the required measurement quality

The performance criterion on cross-sensitivity is met for the sum of interferents with a positive impact on the measured values as well as for the sum of interferents with a negative impact:

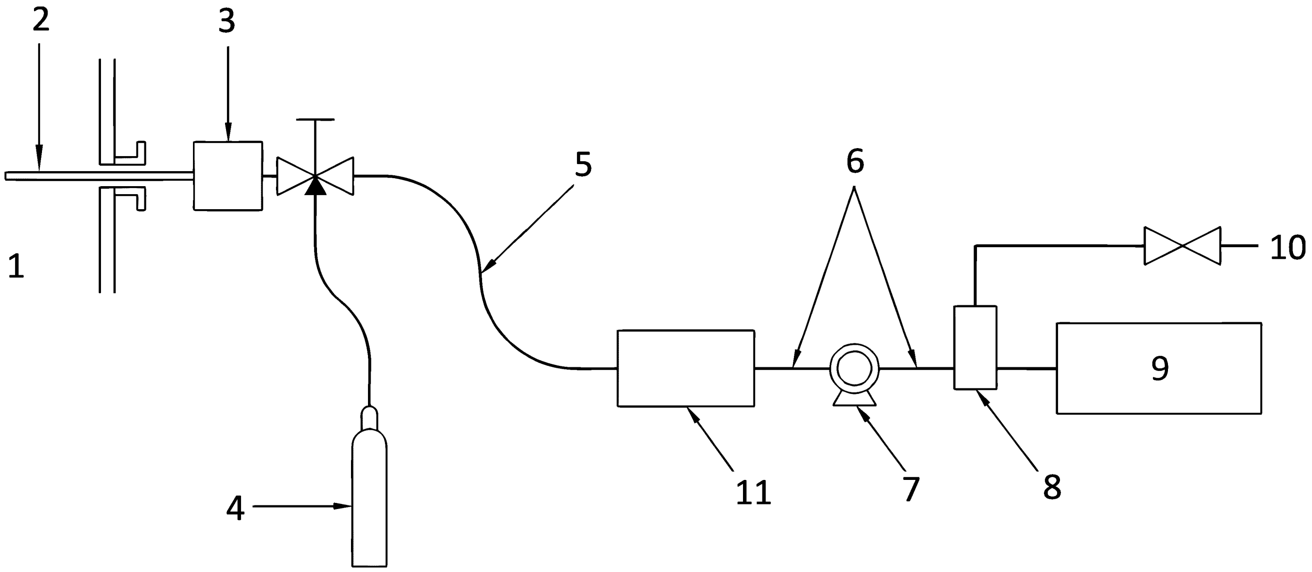
$$C_\mathrm{ip}=0{,}025\,\%<0{,}2\,\%$$

$$C_\mathrm{in}=0{,}004\,\%<0{,}2\,\%$$

All values of the performance characteristics obtained in the laboratory and field tests comply with the performance criteria. Therefore, the measurement method fulfils the requirements.

Annex C
(informative)

Schematic diagram of the measuring system



Key

1	stack
2	sampling probe
3	heated filter
4	calibration gas
5	heated sample gas line
6	sample gas transport line (PTFE)
7	pump
8	sample gas manifold
9	gas analyser
10	sample by-pass vent
11	conditioning system: configuration 1: condenser with a cooling system configuration 2: permeation drier

Figure C.1 — Schematic diagram of the measuring system

Annex D
(informative)

Example of correction of data from drift effect

Table D.1 shows a spreadsheet example based on the correction procedure given in 9.4.3.

Table D.1 —Spreadsheet example of correction of data from drift effect

	B	C	D	E	F	G	H	I
	Input data							
3		concentration unit		%				
4			calibration gas concentration	concentration given by the analyser				
5				adjustment at t_0 (before measurement)	check at t_f (end of measurement)			
6		span point	9,00	8,98	9,00		🟧	:to be filled
7		zero point	0	0,03	0,01		🟨	: list choice
8		time		10:00:00	15:00:00			
9		duration (h)		5:00:00 (F8-E8)				
10		duration (min)		300 (HOUR(E9)*60+ MINUTE(E9))				
11								
12								
13	Calculation of zero and span drift							
14				adjustment	check	deviation		
15		A: span point		0,99444 (E6-E7)/(D6-D7)	0,99889 (F6-F7)/(D6-D7)	0,00444 (F15-E15)		
16		B_{corr}: zero point corrected of span		0,03017 (E7/E15)	0,01001 (F7/F15)	−0,02016 (F16-E16)		
17								
18		drift at zero point	−0,22 % ((G16)/D6)					

19		drift at span point	0,44 % ((D6*G15)/D6)					
20								
21	*If the drift at zero or at the span point is greater than 2 % of the selected span point*							
22								
23	Equation to calculate the concentration C_{corr} corrected according to time t for the concentration C given by the analyser							
24								
25				value at t_0	drift per min			
26		A (span point)		0,994444 (E15)	0,00001481 (G15/E10)			
27		B (zero point not corrected of span)		0,030000 (E7)	−0,00006667 ((F7-E7)/E10)			
28								
29	$C_{\mathrm{corr}} = [C - (B(t_0) + \mathrm{Drift}(B) \times t)] / [A(t_0) + \mathrm{Drift}(A) \times t]$							
30	$C_{\mathrm{corr}} = [C - (\mathrm{E27} + \mathrm{F27} \times t)] / [\mathrm{E26} + \mathrm{F26} \times t]$							
31	:							
32	➔ C_{corr} =	$(C{-}(0{,}030000000{-}0{,}00006667{*}t))/(0{,}994444{+}0{,}00001481{*}t)$						
33	*To apply the formula in a calculation file:* *– copy cell C32 above;* *– in the calculation file click on “edition – special paste – values” in the first cell dedicated to a corrected value;* *– insert before formula “=” then the concentration value measured and replace “t” by its value in min.*							
34								
35								

Annex E
(informative)

Significant technical changes

Table E.1 — Significant technical changes

Clause	**Technical change**
1	Directive 2000/76/EC has been replaced by Directive 2010/75/EU.
2	Normative reference to EN 13284–1 has been replaced by EN 15259 related to requirements for measurement sections and sites and for the measurement objective, plan and report. Normative reference to EN 15267–4 related to performance criteria and test procedures for portable automated measuring systems for monitoring emissions from stationary sources has been added.
3	Definitions have been reviewed taking into account EN 15259 definitions and new version of VIM (2012). Detection limit is no more considered in the list of definition and in performance characteristics (repeatability at zero is more suitable).
4	Symbols and abbreviations used in the main section of the document have been added.
6.3	This clause corresponds to the previous Clause 6.
7	The performance characteristics shall be determined in a general performance test according to the test procedures described in EN 15267–4.
9.2	For determination of homogeneity reference to the EN 15259 has been added.
9.4.2.1	The test gases shall have concentrations traceable to SI units.
9.4.3	Equation to calculate the concentration corrected when drift occurs has been added.
12	Expression of results has been modified to be in line with EN 15259.
13	According to the new rules fixed in EN 14793, $s_{r,\text{limit}}$ has been recalculated.
Annex A	An estimate of the uncertainty calculated through the determination of reproducibility has been added and replaced the expression “reproducibility confidence interval”.
Annex C	The presentation of the calculation of the uncertainty budget has been improved.
Annex D	Typing errors of the table to determine the drift have been corrected.

Bibliography

[1] Directive 2010/75/EU of the European Parliament and of the Council of 24 November 2010 on industrial emissions (integrated pollution prevention and control)

[2] EN 14790:2017, *Stationary source emissions – Determination of the water vapour in ducts — Standard reference method*

[3] 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*

[4] ISO 5725-6:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 6: Use in practice of accuracy values*

[5] JCGM 200:2012, *International vocabulary of metrology – Basic and general concepts and associated terms (VIM)*

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