
**Stationary source emissions —
Determination of the mass concentration
of dinitrogen monoxide (N₂O) —
Reference method: Non-dispersive
infrared method**

*Émissions de sources fixes — Détermination de la concentration
massique de protoxyde d'azote (N₂O) — Méthode de référence:
Méthode infrarouge non dispersive*



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Contents

Page

Foreword	iv
Introduction.....	v
1 Scope	1
2 Normative references	1
3 Terms and definitions	1
4 Symbols and abbreviated terms	5
5 Principle.....	6
6 Description of the automated measuring equipment	6
6.1 General	6
6.2 Sampling line components.....	7
6.3 Analyser equipment	8
6.4 Responsibilities	8
7 Performance criteria and determination of the performance characteristics	9
7.1 Performance criteria.....	9
7.2 Determination of the performance characteristics and measurement uncertainty	10
7.3 Establishment of the uncertainty budget.....	10
8 Measurement procedure.....	11
8.1 Sampling location.....	11
8.2 Sampling point(s)	11
8.3 Choice of the measuring system	11
8.4 Setting of the analyser on site	12
9 Ongoing quality control	13
9.1 General	13
9.2 Frequency of checks	13
10 Evaluation of the method in the field	14
11 Expression of results	14
12 Test report	15
Annex A (informative) Schematic diagram of a typical analyser	16
Annex B (normative) Procedures for determination of the performance characteristics during the general performance test.....	17
Annex C (informative) Example of assessment of compliance of NDIR method for N ₂ O with requirements on emission measurements	20
Annex D (informative) Results of comparison tests.....	27
Annex E (informative) Leak test procedures	30
Bibliography	32

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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

ISO 21258 was prepared by Technical Committee ISO/TC 146, *Air quality*, Subcommittee SC 1, *Stationary source emissions*.

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Introduction

Dinitrogen monoxide (N_2O , also known as nitrous oxide) is an important greenhouse gas with a global warming potential 310 times that of carbon dioxide (CO_2). N_2O is of both natural and anthropogenic origin. Increased emissions of N_2O have been observed, for example, in the exhaust gas of combustion processes using nitrogenous fuels at temperatures below 900 °C, and in the reduction of NO_x using the selective non-catalytic reduction (SNCR) process, in particular when urea is used. There is considerable uncertainty over current N_2O emissions, which is reflected in the wide range of emission factors cited. The largest uncertainties are for emissions from natural and agricultural sources, which are difficult to measure accurately. In the past, emissions from stationary sources such as coal-fired plants and industry were overestimated due to a serious artefact in the grab-sampling methodology used to measure emissions. N_2O is involved in the EU emission trading scheme along with CO_2 and methane (CH_4).

Improved measurement techniques are helping to reduce uncertainties in emission estimates. Improved measurement techniques are also a prerequisite for accurate information on N_2O and its potential role in the enhanced greenhouse effect.

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Stationary source emissions — Determination of the mass concentration of dinitrogen monoxide (N₂O) — Reference method: Non-dispersive infrared method

1 Scope

This International Standard specifies a method for sampling, sample conditioning and determination of dinitrogen monoxide (N₂O) content in the flue gas emitted from ducts and stacks to atmosphere. It sets out the non-dispersive infrared (NDIR) analytical technique, including the sampling system and sample gas conditioning system.

This International Standard is a reference method for periodic monitoring and for calibration, adjustment or control of automatic monitoring systems permanently installed on a stack.

This reference method has been successfully tested on a sewage sludge incinerator where the N₂O concentration in the flue gas was up to about 200 mg/m³.

2 Normative references

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

ISO 9169:2006, *Air quality — Definition and determination of performance characteristics of an automatic measuring system*

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

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

3 Terms and definitions

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

3.1

influence quantity

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

[ISO/IEC Guide 98-3:2008, B.2.10]

3.2

interference

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

3.3

interferent interfering substance

substance present in the air mass under investigation, other than the measurand, that affects the response

[ISO 9169:2006, 2.1.12]

3.4

lack of fit

systematic deviation within the range of application between the measurement results obtained by applying the calibration function to the observed response of the measuring system measuring reference materials and the corresponding accepted value of such reference materials

NOTE 1 Lack of fit may be a function of the measurement result.

[ISO 9169:2006, 2.2.9]

NOTE 2 The expression “lack of fit” is often replaced in everyday language for linear relations by “linearity” or “deviation from linearity”.

3.5

measurand

particular quantity subject to measurement

[ISO/IEC Guide 98-3:2008, B.2.9]

3.6

performance characteristic

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

NOTE Performance characteristics can be described by values, tolerances, or ranges.

3.7

reference gas

gaseous mixture of stable composition used to calibrate the reference measuring system and which is traceable to national or international standards

3.8

reference method

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

3.9

repeatability in the laboratory

precision under repeatability conditions in the laboratory

NOTE 1 Repeatability can be expressed quantitatively in terms of the dispersion characteristics of the results. In this International Standard, the repeatability is expressed as a value with a level of confidence of 95 %.

NOTE 2 Adapted from ISO 3534-2:2006^[1], 3.3.5.

3.10

repeatability conditions in the laboratory

observation conditions where independent test results are obtained with the same method on identical test items in the same test or measuring facility by the same operator using the same equipment within short intervals of time in the laboratory

NOTE 1 Repeatability conditions in the laboratory include:

— the same measurement procedure at the same laboratory;

- the same operator;
- the same measuring instrument used under the same conditions;
- the same location;
- repetition over a short period of time.

NOTE 2 Adapted from ISO 3534-2:2006^[1], 3.3.6.

3.11

repeatability in the field

precision under repeatability conditions in the field

NOTE 1 Repeatability can be expressed quantitatively in terms of the dispersion characteristics of the results. In this International Standard the repeatability under field conditions is expressed as a value with a level of confidence of 95 %.

NOTE 2 Adapted from ISO 3534-2:2006^[1], 3.3.5.

3.12

repeatability conditions in the field

observation conditions where independent test results are obtained with the same method on identical test items in the same test or measuring facility by the same operator using the same equipment within short intervals of time in the field

NOTE 1 Repeatability conditions in the field include:

- the same measurement procedure;
- two sets of equipment, the performance of which fulfils the requirements of the reference method, used under the same conditions;
- the 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.

NOTE 2 Adapted from ISO 3534-2:2006^[1], 3.3.6.

3.13

reproducibility in the field

precision under reproducibility conditions in the field

NOTE 1 Reproducibility in the field can be expressed quantitatively in terms of the dispersion characteristics of the results. In this International Standard the reproducibility under field conditions is expressed as a value with a level of confidence of 95 %.

NOTE 2 Adapted from ISO 3534-2:2006^[1], 3.3.10.

NOTE 3 Results are usually understood to be corrected results.

3.14

reproducibility conditions in the field

observation conditions where independent test results are obtained with the same method on identical test items in different test or measurement facilities with different operators using different equipment in the field

NOTE 1 Reproducibility conditions in the field include:

- the same measurement procedure;

- several sets of equipment, the performance of which fulfils the requirements of the reference method, used under the same conditions;
- the same location;
- implemented by several laboratories.

NOTE 2 Adapted from ISO 3534-2:2006^[1], 3.3.11.

3.15

residence time in the measuring system

time period for transportation of the sampled gas from the inlet of the probe to the inlet of the measurement cell

3.16

response time

time interval between the instant when a stimulus is subjected to a specified abrupt change and the instant when the response reaches and remains within specified limits around its final stable value, determined as the sum of the lag time and the rise time in the rising mode, and the sum of the lag time and the fall time in the falling mode

[ISO 9169:2006, 2.2.4]

3.17

span gas

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

NOTE This concentration is often chosen around 80 % of full scale.

3.18

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

[ISO/IEC Guide 98-3:2008, B.2.18]

3.19

standard uncertainty

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

[ISO/IEC Guide 98-3:2008, 2.3.1]

3.20

combined standard uncertainty

standard uncertainty of the result of a measurement when that result is obtained from the values of a number of other quantities, equal to the positive square root of a sum of terms, the terms being the variances or covariances of these other quantities weighted according to how the measurement result varies with changes in these quantities

[ISO/IEC Guide 98-3:2008, 2.3.4]

3.21

expanded uncertainty

quantity defining an interval around 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

[ISO/IEC Guide 98-3:2008, 2.3.5]

NOTE In this International Standard, the expanded uncertainty is calculated with a coverage factor of $k = 2$, and with a level of confidence of 95 %.

3.22**uncertainty budget**

list of sources of uncertainty and their associated standard uncertainties, compiled with a view to evaluating a combined standard uncertainty associated with a measurement result

[ISO/TS 21748:2004^[6], 3.13]

3.23**zero gas**

gas or gas mixture used to establish the zero point on a calibration curve within a given concentration range

[ISO 12039:2001^[4]]

4 Symbols and abbreviated terms

γ_a	measured concentration of N ₂ O at actual oxygen concentration
γ_d	concentration of N ₂ O on dry basis
γ_n	normalized concentration of N ₂ O
γ_w	measured concentration of N ₂ O on wet basis
$\bar{\gamma}$	grand mean of measured N ₂ O concentration
$\phi_{H_2O,m}$	measured water vapour content, as a percentage volume fraction
$\phi_{O_2,m}$	measured oxygen content, as a volume fraction, in the waste gas
$\phi_{O_2,ref}$	reference oxygen content, as a volume fraction
$C_{V,r}$	coefficient of variation of repeatability
$C_{V,R}$	coefficient of variation of reproducibility
$C_{V,u}$	coefficient of variation of the standard uncertainty
k	coverage factor
n	number of test results
PFA	perfluoroalkoxy
PTFE	polytetrafluoroethylene
QA	quality assurance
QC	quality control
s_j	standard deviation of level j
$s_{r,j}$	repeatability standard deviation
$s_{R,j}$	reproducibility standard deviation
u	standard uncertainty
$u(\gamma_{N_2O})$	combined uncertainty of the measured concentration of N ₂ O
$U(\gamma_{N_2O})$	expanded uncertainty of the measured concentration of N ₂ O

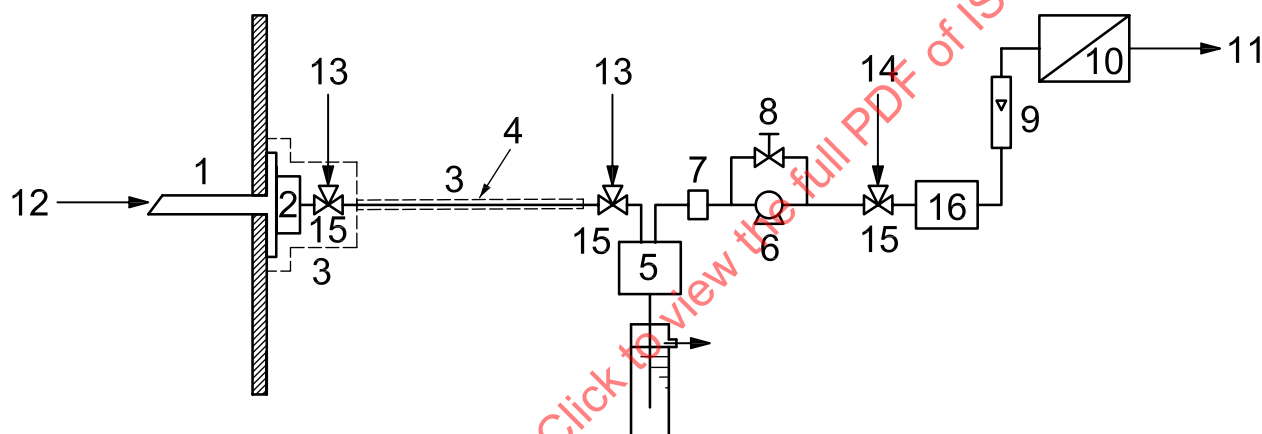
5 Principle

This International Standard describes a reference method for sampling, sample conditioning, and determining N_2O content in the flue gas emitted from ducts and stacks to atmosphere by means of a continuous analyser using non-dispersive infrared method. A number of performance characteristics with associated minimum performance criteria are given for the analyser and details of the uncertainty of the method are presented. Requirements and recommendations for quality assurance and quality control of field measurements are given.

6 Description of the automated measuring equipment

6.1 General

A representative volume of flue gas is extracted from the emission source for a fixed period of time at a controlled flow rate. Dust present in the volume sampled is removed by filtration before the sample is conditioned and passes to the analytical instrument. Figure 1 shows a typical arrangement of a complete measuring system for N_2O .



Key

- 1 gas sampling probe
- 2 primary filter
- 3 heating (for use as necessary)
- 4 sampling line (heated as necessary)
- 5 sample cooler with condensate separator
- 6 sample pump
- 7 secondary filter
- 8 needle valve
- 9 flow meter
- 10 N_2O analyser
- 11 output
- 12 inlet for zero and span gas (preferably in front of the nozzle) to check the complete system
- 13 inlet for zero and span gas to check the conditioning system and N_2O analyser
- 14 inlet for zero and span gas to check the converter and N_2O analyser
- 15 valve
- 16 converter for CO oxidation

Figure 1 — Example of the installation of measuring devices

6.2 Components of the sampling apparatus

6.2.1 Sampling probe

The sampling probe shall be made of suitable, corrosion-resistant material, e.g. stainless steel. The probe shall be heated to avoid condensation occurring in its interior; it shall also be cooled by an air or water jacket when sampling very hot gases. Nonetheless, it shall not be cooled below the acid dew-point. The probe diameter shall be appropriately sized to provide a flow rate that meets the requirements of the analysers.

6.2.2 Primary filter

The filter shall be made of ceramic or sintered metal with 10 µm pore size. The filter shall be heated above the water or acid dew-point.

6.2.3 Sampling line

The sampling line shall be made of polytetrafluoroethylene (PTFE), perfluoroalkoxy (PFA) or stainless steel. The lines shall be operated 15 °C above the dew-point of condensable substances (generally the water or acid dew-point). The tube diameter should be appropriately sized to provide a flow rate that meets the requirements of the analysers, under selected line length and the degree of pressure drop in the line as well as the performance of the sampling pump used.

6.2.4 Sample cooler or permeation drier

The sample cooler or permeation drier shall be used to separate water vapour from the flue gas. The dew-point shall be sufficiently below the ambient temperature. A cooling temperature of 2 °C to 5 °C is suggested. Sufficient cooling is required for the volume of gas being sampled and the amount of water vapour that it contains. The cooler and the sample treatment procedure is important to prevent artefact formation of N₂O from NO₂ and SO₂ dissolved in the condensate, and thus minimize a source of error in the results.

6.2.5 Sampling pump

A gas-tight pump is used to withdraw a continuous sample from the duct through the sampling system. This may be a diaphragm pump, a metal bellows pump, an ejection pump or other pumps. The pump shall be constructed of corrosion-resistant material. The performance of the pump shall be such that it can supply the analyser with the gas flow required. In order to reduce the residence time in the sampling line and the risk of physicochemical transformation of the sample, the gas flow can be greater than that required for the analytical units.

6.2.6 Secondary filter

The secondary filter is needed to remove the remaining particulate material, in order to protect the pump and the analyser. A filter that retains particles greater than 1 µm is recommended. Acceptable materials are PTFE or borosilicate glass. The size of the filter shall be determined from the sample flow required and the manufacturer's data on the flow rate per unit area.

6.2.7 Flow controller and flow meter

The flow controller and flow meter are used to set the required flow. They shall be constructed of corrosion-resistant material.

6.2.8 Converter

The converter is an oxidation catalyst tube, which may be needed for pretreatment of the sample gas. The converter oxidizes CO in the sample gas into CO₂ which can be corrected for later, in order to decrease the influence of the interferent.

The converter uses a mixture of manganese and copper oxides which can oxidize CO to CO₂ at 120 °C. Since it has been confirmed that the decomposition of N₂O begins at temperatures higher than 300 °C, no effect of the converter on the measured N₂O concentration has been found. The converter efficiency shall be such that the performance criteria given in Table 1 shall be met.

6.3 Analyser equipment

The gas analysers use, as the measurement principle, the absorption of infrared radiation (IR) by the component measured in characteristic wavelength ranges. The analysers operate according to the non-dispersive IR method (NDIR), while the selectivity of measurement is achieved by the radiation detector which is filled with the component to be measured. A schematic diagram of a typical analyser is given in Figure A.1.

Special attention should be given to CO and CO₂ interference, since for detection of N₂O the absorption at around 4,5 µm is usually used, while CO has its absorption at 4,5 µm to 4,7 µm and CO₂ has its absorption at 4,3 µm. The CO interference can be excluded by using the converter (see 6.2.8). The CO₂ sensitivity requires determination with CO₂ test gases. During real operation, CO₂ requires simultaneous measurement to yield data for real-time correction of the N₂O readings. In many instruments, this CO₂ interference correction is done automatically through a CO₂ channel.

Since it can interfere with the measurement and lead to condensation in the analyser, water vapour present in the sampled gas is condensed in a gas cooler before the gas enters the analyser. Note that the presence of water droplets can affect the analysis of N₂O, since the solubility of N₂O in water is 1,2 g/l (at 20 °C, 101,3 kPa).

6.4 Responsibilities

6.4.1 General

The reference method shall comply with the performance criteria specified in Table 1. These performance criteria are allocated to the responsibilities specified in 6.4.2 to 6.4.4.

6.4.2 General performance test

The manufacturer of the measuring system shall prove, in a general performance test, that the relevant performance criteria listed in Table 1 are fulfilled by the instrument type.

6.4.3 Ongoing quality assurance and quality control in the laboratory

The user of the measuring system shall prove, during regular laboratory tests within the ongoing QC programme, that the relevant performance criteria listed in Table 1 are fulfilled.

6.4.4 Quality assurance during operation in the field

The user of the measuring system shall check, during field operation, that the relevant performance criteria listed in Table 1 are fulfilled.

Table 1 — Relevant performance criteria of the analyser and the measuring system criteria to be evaluated during the general performance test and by means of ongoing QA-QC in laboratory tests and during field operation

Performance characteristic	Performance criterion	Performance test	QA-QC in laboratory	Field operation	Term for evaluating uncertainty
Response time	≤ 200 s	×	×	×	
Detection limit	≤ 2 % of upper limit of the lowest measuring range used	×			×
Lack of fit	≤ 2 % of upper limit of the lowest measuring range used	×	×		×
Zero drift in 24 h	≤ 2 % of upper limit of the lowest measuring range used	×		×	×
Span drift in 24 h	≤ 2 % of upper limit of the lowest measuring range used	×		×	×
Sensitivity to atmospheric pressure	≤ 2 % of upper limit of the lowest measuring range used for 2 kPa	×			×
Sensitivity to sample volume flow or sample pressure	— ^a	×			×
Sensitivity to ambient temperature	≤ 2 % of upper limit of the lowest measuring range used per 10 °C	×			×
Sensitivity to electric voltage	≤ 2 % of the range per 10 V	×			×
Interferents ^b	≤ 6 % of upper limit of the lowest measuring range used	×	×		×
Losses and leakage in the sampling line and conditioning system	≤ 2 % of the measured value			×	×
Standard deviation of repeatability in laboratory at zero	≤ 2 % of upper limit of the lowest measuring range used	×	×		× ^c
Standard deviation of repeatability in laboratory at span level	≤ 2 % of upper limit of the lowest measuring range used	×	×		× ^c
^a The tested volume flow range or pressure is defined in the manufacturer's recommendations. ^b Interferents that shall be tested are at least those given in Table 2. ^c Only one of the two values shall be included in the calculation: the first possibility is to choose the repeatability standard deviation obtained from laboratory tests corresponding to the closest concentration to the actual concentration in stack, or the higher standard deviation of repeatability or coefficient of variation of repeatability independently of the concentration measured in stack.					

7 Performance criteria and determination of the performance characteristics

7.1 Performance criteria

Table 1 gives an overview of the relevant performance characteristics and performance criteria of the analyser and measurement system to be evaluated on three levels, during general performance test, by means of ongoing QA-QC during laboratory tests, and during field operation. In the rightmost column, values included in the calculation of the expanded uncertainty are indicated.

The upper limit of the lowest measuring range used should be set according to the application, so that the measurement values lie within 20 % to 80 % of the analyser range.

7.2 Determination of the performance characteristics and measurement uncertainty

7.2.1 Performance test

The performance characteristics of the method shall be determined during the general performance test. The values of the performance characteristics determined shall meet the performance criteria specified in Table 1. The procedures for the determination of these performance characteristics are specified in Annex B.

The ambient conditions applied during the general performance test have to be documented.

7.2.2 Ongoing quality control

The user shall check specific performance characteristics during ongoing operation of the measuring system with a frequency specified in Table 3.

The measurement uncertainty during field application shall be determined by the user of the measuring system in accordance with applicable national or International Standards. It can be determined by a direct or by an indirect approach for uncertainty estimation as described in ISO 20988^[5]. The uncertainty of the measured values under field operation is not only influenced by the performance characteristics of the analyser itself but also by uncertainty due to:

- a) sampling line and sample conditioning system;
- b) site specific conditions;
- c) reference gases used.

7.3 Establishment of the uncertainty budget

An uncertainty budget shall be established to determine if the analyser and its associated sampling system fulfils the requirements for a maximum allowable expanded uncertainty. This uncertainty budget shall be drawn up according to the procedures specified in ISO 14956 or ISO/IEC Guide 98-3 requirements, taking into account all the relevant characteristics included in calculation of expanded uncertainty given in Table 1.

The principle of calculation of the expanded uncertainty is based on the law on propagation of uncertainty laid down in ISO/IEC Guide 98-3 requirements as listed.

- Determine the standard uncertainties for each value included in the calculation of the budget uncertainty by means of laboratory and field tests, and in accordance with ISO/IEC Guide 98-3 requirements.
- Calculate the uncertainty budget by combining all the standard uncertainties in accordance with ISO 14956, including the uncertainty of the reference gas and taking variations range of influence quantities and interferences of the specific site conditions into account. These conditions are sometimes unknown. In this case, default values defined in Table 2 shall be applied.
- Values of standard uncertainty less than 5 % of the maximum standard uncertainty can be discarded.
- Calculate the expanded uncertainty.

Table 2 — Default ranges of influence quantities and interferences to be applied for the determination of the uncertainty budget

Influence quantities and interferences	Variations range and default concentrations of interferences
Atmospheric pressure	± 2 kPa
Sample volume flow variation	In accordance with the manufacturer's recommendations
Ambient temperature	± 15 °C
Power supply	± 10 % of nominal supply voltage
CO ₂	14 % volume
CO	300 mg/m ³
SO ₂	200 mg/m ³
NO	250 mg/m ³

NOTE An example of the evaluation of an uncertainty budget is given in Annex C.

8 Measurement procedure

8.1 Sampling location

The sampling location chosen for the measurement devices and sampling shall be of sufficient size and construction, that a representative emission measurement can be made that is suitable for the measurement task. In addition, the sampling location shall be chosen with regard to safety of the personnel, accessibility and availability of electrical power.

8.2 Sampling point(s)

It is necessary to ensure that the gas concentrations measured are representative of the average conditions inside the flue gas duct. Therefore, the sampling points shall be selected so that the sample is as representative as possible.

NOTE 1 The selection of sampling points for representative sampling is described, for example, in ISO 9096^[3] and EN 15259^[9].

If the flue gas is homogenous, sampling may be carried out at any point in the sampling plane. For larger ducts, this point may be situated close to the sampling port provided that there is no disturbance of the flow or concentration due to the influence of the sampling port.

NOTE 2 The homogeneity can be demonstrated with a continuous measurement of O₂ or CO₂ using a fixed and a moving probe covering the sampling plane. The method is described in EN 15259^[9].

8.3 Choice of the measuring system

To choose an appropriate analyser, sampling line and sample conditioning unit, the following characteristics of flue gases should be known before the field campaign:

- a) temperature of exhaust gases;
- b) humidity;
- c) dust loading;
- d) expected concentration range of N₂O;

- e) expected concentration of potentially interfering substances, including at least the four components listed in Table 2.

The full scale of the selected analyser shall be 150 % to 300 % of the expected concentration. The full scale should not be less than peak emission.

To avoid long response times, the sample line should be as short as possible. If necessary, a bypass pump should be used. If there is a high dust loading in the sample gas, then an appropriate heated filter shall be used.

Before conducting field measurements, the user shall verify that the analyser is functioning in accordance with the required performance criteria fixed in this International Standard, and that the sampling line and conditioning unit are in a good operational condition.

8.4 Setting of the analyser on site

8.4.1 General

The complete measuring system, including the conditioning unit, the sampling line, and the analyser, shall be connected according to the manufacturer's instructions. The nozzle of the probe is placed in the duct according to 8.2.

The conditioning unit, sampling probe, filter, connection tube and analyser shall be stabilised at the required temperature.

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. This flow shall be maintained at a constant level within ± 10 % of the nominal flow rate.

When both the instrument and sampling system have been set up, the proper functioning of the instrument and sampling system shall be checked. The results of these checks shall fulfil the requirements and limitations as set out by the manufacturer of the instrument, as well as the requirements (such as materials used, and so on) given by this International Standard. Compliance with the requirements of this International Standard shall be documented.

Any data recording, data processing and telemetry system used in conjunction with the measuring system shall be checked for proper functioning. If any components are changed, then these checks shall be repeated. All checks shall be documented. The time resolution of the data recording system used to calculate the mean values shall be less or equal to 10 s.

8.4.2 Preliminary zero and span check, and adjustments

8.4.2.1 Adjustment of the analyser

At the beginning of the measuring period, zero and span gases are supplied to the analyser directly, without passing through the sampling system. Adjustments, as listed, are made until the correct zero and span gas values are given by the data sampling system.

- Adjust the zero value, if necessary.
- Adjust the span, if necessary.
- Finally, check zero again to see if there is no significant change (e.g. a change less than the standard deviation of the repeatability at zero). If there is any problem, repeat the procedure.

8.4.2.2 Check of the sampling system

Before starting the measurement, determine whether there is leakage in the sampling line by supplying zero gas and span gas to the analyser through the sampling system, as close as possible to the nozzle (in front of the filter if possible). Differences shall be less than 2 %.

NOTE Possible impurities in the sampling system can lead to memory effects due to adsorption on to or desorption from the surfaces.

Two procedures for checking the sampling line for leakage are given as examples in Annex E.

The measuring system is not suitable if this relative difference remains over 2 %, and the unit shall not be used.

8.4.3 Zero and span checks after measurement

At the end of the measuring period and at least once a day, zero and span checks shall be performed at the inlet of the sampling system. The information shall be documented. In case of deviation between checks after measurement and preliminary adjustments, values of deviation shall be indicated in the test report (Clause 12).

If the span or zero drift is larger than 2 % of the measured values, it is necessary to correct the measured values according to the span and drift values. The effect of span and zero drift may only be corrected if the drifts are lower than 10 % of the results. Otherwise, the results shall be rejected.

NOTE The drift is most likely to be proportional to time. In that case, the measured values can be corrected on the basis of a linear change of the zero and span point in time.

9 Ongoing quality control

9.1 General

Quality control is critically important in order to ensure that the uncertainty of the measured values for N₂O 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 quality data.

9.2 Frequency of checks

Table 3 shows the minimum required frequency of checks. The laboratory shall implement the relevant International Standards for determination of performance characteristics or procedures specified in Annex B.

Table 3 — Frequency of checks as reference method

Check	Frequency	Action criteria
Cleaning or changing of particulate filters ^a at the sampling inlet and at the analyser inlet	Every campaign if needed ^a	—
Regular maintenance of several parts of the analyser	As required by manufacturer	As required
The laboratory test, with the exception of the lack of fit	When the instrument is purchased or commissioned and after a major repair	As required
The lack of fit	At least every year and after repair of the instrument ^b	As required and when linearity exceeds ± 2 % of the range
Standard deviation of repeatability in laboratory at zero and at span level	At least every year and after repair of the instrument ^b	As required and when deviation exceeds ± 2 % of the range
^a The particle filter shall be changed periodically depending on the dust loading at the sampling site. During this filter change, the filter housing shall be cleaned. ^b Strongly dependent on site specific conditions.		

10 Evaluation of the method in the field

The uncertainty of measurement results obtained with this method has been evaluated on a sewage sludge incineration plant, determined with the participation of four testing teams and six analysers.

Details of the characteristics of the installations, the conditions during field tests, and the values of repeatability and reproducibility in the field are given in Annex D.

11 Expression of results

The signals from the analyser are converted to mass concentrations using appropriate conversion factors. The results of measurement shall be expressed as mass concentrations on a dry basis at 273 K and 101,3 kPa (standard conditions).

The measured N_2O concentration on a wet basis shall be corrected to that on a dry basis, γ_d , e.g. in milligrams per cubic metre, using Equation (1):

$$\gamma_d = \frac{100 \%}{100 \% - \varphi_{H_2O,m}} \gamma_w \quad (1)$$

where

γ_w measured concentration, e.g. in milligrams per cubic metre, of N_2O on the wet basis;

$\varphi_{H_2O,m}$ measured water vapour content, expressed as a percentage volume fraction.

The uncertainty of correction on the dry basis depends on the measured water vapour content and on the uncertainty of the measurement of water vapour content.

If necessary, the measured N_2O concentration shall be corrected to reference conditions of oxygen to give the normalised concentration, γ_n , e.g. in milligrams per cubic metre, using Equation (2):

$$\gamma_n = \frac{21\% - \varphi_{O_2,ref}}{21\% - \varphi_{O_2,m}} \gamma_a \quad (2)$$

where

γ_a is the measured concentration, e.g. in milligrams per cubic metre, of N_2O at actual oxygen concentration;

$\varphi_{O_2,m}$ is the measured oxygen content, expressed as a percentage volume fraction, in the waste gas;

$\varphi_{O_2,ref}$ is the reference oxygen content, expressed as a percentage volume fraction.

The uncertainty of correction to oxygen reference concentration depends on the measured concentration of oxygen and of the value of oxygen reference concentration.

12 Test report

The test report shall be in accordance with international or national regulations.

If not specified otherwise, the test report shall include at least the following information:

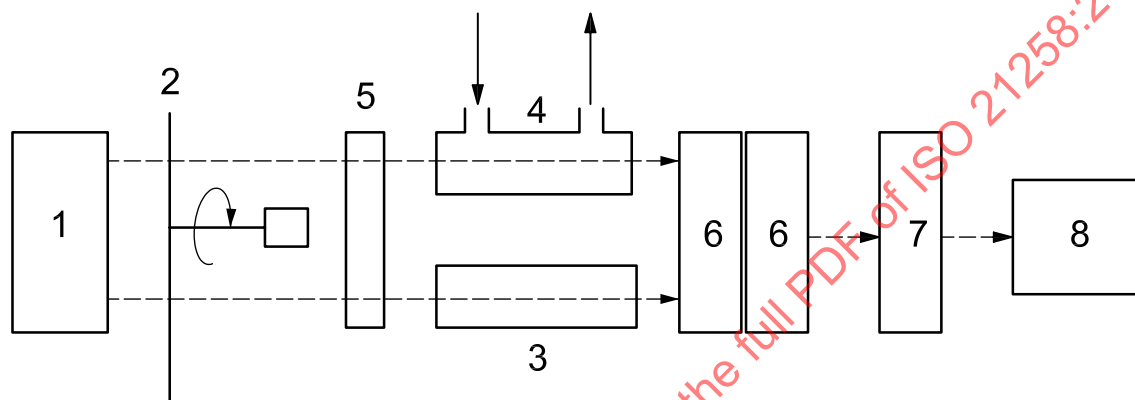
- a) reference to this International Standard (ISO 21258:2010);
- b) description of the purpose of tests;
- c) principle of gas sampling;
- d) information about the analyser and description of the sampling and conditioning line;
- e) identification of the analyser used, and certification of approval fulfilling the requirements for a reference method according to Table 1;
- f) operating range;
- g) details of the quality and the concentration of the reference gases used, details on the adjustment performed before and after actual sampling (at the inlet of the sampling line and at the inlet of the analyser);
- h) description of plant and process;
- i) identification of the sampling plane;
- j) actions taken to achieve representative samples;
- k) description of the location of the sampling point(s) in the sampling plane;
- l) description of the operating conditions of the plant process;
- m) changes in the plant operations during sampling;
- n) sampling date, time and duration;
- o) time averaging on relevant periods;
- p) measurement uncertainty;
- q) any deviations from this International Standard.

Annex A

(informative)

Schematic diagram of a typical dual-beam type NDIR analyser

A schematic diagram of a typical configuration of a dual-beam type NDIR analyser is shown in Figure A.1. An optopneumatic receiver with analytical sensor consists of two kinds of detectors. One is the main detector for sample gas (N_2O) and for interfering substances. Another is the compensating detector, which detects the specific interfering substances such as CO_2 and CO . The signal from the second detector, used in combination with the main detector, corrects for the influence of interfering substances.



Key

- 1 IR radiation source
- 2 modulation unit (chopper)
- 3 reference cell
- 4 sample cell
- 5 filter cell or optical filter
- 6 optopneumatic receiver with analytical sensor
- 7 amplifier
- 8 display

Figure A.1 — Diagram of a dual-beam type NDIR analyser

Annex B

(normative)

Procedures for determination of the performance characteristics during the general performance test

B.1 General test conditions

Before any tests are performed, the warm-up time specified by the manufacturer shall be taken into account. If the warm-up time is not specified, a minimum of 2 h shall be taken.

During the test for each individual performance characteristic, the values of the following parameters shall be constant within the specified range stated below:

- surrounding temperature: ± 2 °C;
- sampling pressure: $\pm 0,2$ kPa;
- supply voltage: ± 1 % of the nominal line voltage (except for the voltage dependence test);
- sample flow rate: ± 1 % (except for the sensitivity test to sample volume flow).

For the determination of the various performance characteristics, reference gases with a known uncertainty of concentration shall be used.

B.2 Procedures

B.2.1 General

Performance characteristics are determined according to procedures described in ISO 9169, and according to the recommendations set out in B.2.2 to B.2.8.

B.2.2 Response time

The response time of the analyser shall be determined at the nominal sample flow rate specified by the manufacturer.

As the response time is strongly dependent on the sample flow rate, the sample flow rate shall be kept constant within (± 10 %) during the test.

The average of the four response times with an increasing concentration and the average of the four response times with a decreasing concentration shall be calculated. If the two response time averages differ, the longer one shall be taken.

B.2.3 Detection limit

The detection limit of the analyser is determined by performing at least 30 measurements at concentrations close to zero. The estimation of the limit of detection γ_{lod} is equal to:

$$\gamma_{\text{lod}} = \frac{2 s_0}{\gamma_{\text{mr}}} \quad (\text{B.1})$$

where

s_0 is the standard deviation of the series of measurements;

γ_{mr} is the measurement range of the analyser.

The duration of all measurements shall be short enough not to be sensitive to drift effects.

B.2.4 Lack of fit

Lack of fit of the analyser shall be determined by applying at least five concentrations of gas approximately evenly distributed across the range, according to the following sequence: $\gamma_0; \gamma_3; \gamma_2; \gamma_4; \gamma_1; \gamma_0$.

Lack of fit shall be determined by calculating the residual value for each concentration (difference between concentration of reference materials and the average of results of measurement). Each residual shall comply with the performance criteria given in Table 1.

B.2.5 Zero drift and span drift

Zero and span drifts shall be estimated by applying at least 10 sequences of zero and span level, evenly distributed on a period of 24 h, and calculating for each concentration the slope of a linear regression, in accordance with ISO 9169.

B.2.6 Sensitivity to influence quantities

An influence coefficient is determined for each influence quantity (temperature, pressure etc.). It is the ratio of the variation of the influence quantity versus the corresponding variation of the measurand.

The influence coefficient shall be determined by applying the measurand to the analyser when the external influence quantity is set at the "normal" value, and then when the influence quantity is set at a selected value.

The influence coefficient may vary with the concentration of the measurand.

Several determinations may be implemented, for instance at zero and at about 70 % to 80 % of the range.

B.2.7 Sensitivity to interferences

An interference coefficient is determined for each interferent. It is the ratio of the variation of the interferent versus the corresponding variation of the measurand.

The interference coefficient shall be determined by applying the measurand to the analyser without interferent, and then when the interferent is set at a selected value.

The interference coefficient may vary with the concentration of the measurand. Several determinations may be implemented, for instance at zero and about 70 % to 80 % of the range. If there is no internal CO₂ measurement and automatic recalculation, CO₂ has to be determined manually and the (linear) measured interference can be recalculated.

B.2.8 Repeatability at zero and at span level

To determine repeatability at zero and at span level, several measurements shall be repeated at zero and at a span concentration. Repeatability shall be calculated for both series of measurement (zero and span value) in accordance with ISO 9169.

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Annex C (informative)

Example of assessment of compliance of NDIR method for N₂O with requirements on emission measurements

C.1 Specific conditions in the site

See Table C.1

Table C.1 — Site specific conditions

Specific conditions	Value / range
Range of analyser (N ₂ O)	0 mg/m ³ to 400 mg/m ³
Studied concentration of N ₂ O	< 400 mg/m ³
Conditions on the field	
Sample volume flow	(60 ± 6) l/h
Temperature during adjustment	285 K
Fluctuations of ambient temperature during measurement	283 K to 308 K
Voltage variation	200 V to 220 V, or 100 V to 110 V
Atmospheric pressure during adjustment	100 kPa
Atmospheric pressure variation	< 1 kPa
CO concentration variations in the field	14 mg/m ³ to 40 mg/m ³
CO ₂ volume fraction variations in the field	10 % to 13 %
NO _x concentration variations in the field	13 mg/m ³ to 56 mg/m ³
SO ₂ concentration variations in the field	5 mg/m ³ to 15 mg/m ³
N ₂ O concentration of the reference gas	(320 ± 6,4) mg/m ³

C.2 Performance characteristics of the method — results of validation in the tests

See Table C.2

C.3 Calculation of standard uncertainty of measurement values given by the analyser

C.3.1 General

Model equations in this clause, as well as calculations of partial uncertainties, are related to the values measured by the analyser and expressed in milligrams per cubic metre.

Table C.2 — Performance characteristics

Performance characteristic for NDIR method	Performance criteria	Results of tests at laboratory and during field operation
Response time	200 s	90 s
Detection limit	±2 % of the range	±1,5 % of the range
Lack of fit	±2 % of the range	±2 % of the range
Zero drift in 24 h	±2 % of the range	±1 % of the range
Span drift in 24 h	±2 % of the range	±1 % of the range
Sensitivity to the sample volume flow	2 % of the range	1 % of the range
Sensitivity to atmospheric pressure	2 % of the range per 2 kPa	2 % of the range per 2 kPa
Sensitivity to ambient temperature	2 % of the range per 10 K	1 % of the range per 10 K
Sensitivity to electric voltage at span level	2 % of the range per 10 V	1 % of the range per 10 V
Interferents	Total ≤ ±6 % of the range CO ₂ at 14 % CO at 300 mg/m ³ NO ₂ at 40 mg/m ³ NO at 320 mg/m ³ SO ₂ at 200 mg/m ³	Total ≤ ±6 % of the range 2,8 % 2,3 % 0,4 % 0,1 % 0,1 %
Loss and leakage in the sampling line and conditioning system	2 % of the measured value	2 % of the measured value
Standard deviation of repeatability in laboratory at zero	1 % of the range	0,2 % of the range
Standard deviation of repeatability in laboratory at span level	2 % of the range	0,2 % of the range

C.3.2 Elements required for the uncertainty determinations

The concentration of N₂O, γ_{N_2O} , e.g. in milligrams per cubic metre, is equal to the concentration given by the analyser plus corrections due to deviations associated with influence quantities and to the performance characteristics of the analyser. Equation (C.1) shows a corresponding statistical model equation used for the uncertainty estimation.

$$\gamma_{N_2O} = \gamma_{\text{read}} + \delta\gamma_{\text{lof}} + \delta\gamma_{\text{d,z}} + \delta\gamma_{\text{d,s}} + \delta\gamma_{\text{rep}} + \delta\gamma_{\text{adj}} + \delta\gamma_i + \sum_{j=1}^p \delta\gamma_{\text{inf},j} \quad (\text{C.1})$$

where

- γ_{read} is the concentration of N₂O given by the analyser;
- $\delta\gamma_{\text{lof}}$ is the correction for lack of fit;
- $\delta\gamma_{\text{d,z}}$ is the correction for zero drift;
- $\delta\gamma_{\text{d,s}}$ is the correction for span drift;
- $\delta\gamma_{\text{rep}}$ is the correction for repeatability of the measurement;
- $\delta\gamma_{\text{adj}}$ is the correction for adjustment;

$\delta\gamma_i$ is the correction for interferents (e.g. CO, CO₂, NO_x, SO₂);

$\delta\gamma_{inf,j}$ is the correction for influence quantities (e.g. ambient temperature, atmospheric pressure, sample volume flow, voltage).

The corrections are specific to each analyser. Sometimes the value of a correction is zero. For example, influence parameters can increase or decrease during the measurement period and are generally not monitored. Therefore, it can be considered that the best correction to be applied is zero.

Whether the corrections are equal to zero or not, the uncertainties associated with these corrections shall be taken into account in the calculation of the uncertainty. Since the corrections shown in Equation (C.1) are non-correlated, the combined standard uncertainty associated with the N₂O concentration measurement, $u(\gamma_{N_2O})$, is given as the square root of the sum of the squares of the individual uncertainty contributions, u_i :

$$u(\gamma_{N_2O}) = \sqrt{u_i^2} = \sqrt{u_{read}^2 + u_{lof}^2 + u_{d,z}^2 + u_{d,s}^2 + u_{rep}^2 + u_{adj}^2 + u_i^2 + \sum_{j=1}^p u_{inf,j}^2} \quad (C.2)$$

where

u_{read} is the uncertainty of the reading of the analyser;

u_{lof} is the uncertainty contribution due to lack of fit;

$u_{d,z}$ is the uncertainty contribution due to zero drift;

$u_{d,s}$ is the uncertainty contribution due to span drift;

u_{rep} is the uncertainty contribution due to repeatability of the measurement;

u_{adj} is the uncertainty contribution due to adjustment;

u_i is the uncertainty contribution due to interferents;

$u_{inf,j}$ is the uncertainty contribution due to influence quantities.

The expanded uncertainty $U(\gamma_{N_2O})$ is determined using Equation (C.3):

$$U(\gamma_{N_2O}) = 1,96 u(\gamma_{N_2O}) \quad (C.3)$$

For Equation (C.2), the uncertainties are determined from test data, where the probability distribution of values is rectangular for most parameters and normal for a few others. The coverage factor 1,96 may be used since the number of measurements used to determine the uncertainty contribution and the associated number of degrees of freedom is sufficiently high or a rectangular distribution is assumed.

In the case of rectangular distributions, the standard uncertainties for each performance characteristic are calculated in accordance with ISO 14956 by Equation (C.4):

$$u_i = \sqrt{\frac{(x_{i,\max} - x_{i,\text{adj}})^2 + (x_{i,\min} - x_{i,\text{adj}}) \times (x_{i,\max} - x_{i,\text{adj}}) + (x_{i,\min} - x_{i,\text{adj}})^2}{3}} \quad (\text{C.4})$$

where

- $x_{i,\min}$ is the minimum value of the average reading influenced by performance characteristic i during the performance test;
- $x_{i,\max}$ is the maximum value of the average reading influenced by performance characteristic i during the performance test;
- $x_{i,\text{adj}}$ is the value of the average reading with the influence quantity at its nominal value during the performance test.

Equation (C.4) can be simplified in the following three cases:

- if the value $x_{i,\text{adj}}$ is at the centre of the interval bounded by the maximum value $x_{i,\max}$ and the minimum value $x_{i,\min}$ of all values x_i , then the standard uncertainty u_i is given by Equation (C.5):

$$u_i = \frac{(x_{i,\max} - x_{i,\min})}{\sqrt{12}} \quad (\text{C.5})$$

- if the absolute values of the measured deviation above and below the central value are equal (see Equation (C.6)), then the standard uncertainty u_i is given by Equation (C.7):

$$|x_{i,\max} - x_{i,\text{adj}}| = |x_{i,\min} - x_{i,\text{adj}}| = \Delta x_i \quad (\text{C.6})$$

$$u_i = \frac{\Delta x_i}{\sqrt{3}} \quad (\text{C.7})$$

- if the value of $x_{i,\text{adj}}$ is the same as either $x_{i,\min}$ or $x_{i,\max}$, then the standard uncertainty u_i is given by Equation (C.8):

$$u_i = \frac{(x_{i,\max} - x_{i,\min})}{\sqrt{3}} \quad (\text{C.8})$$

C.3.3 Individual uncertainty contributions

The uncertainty related to the reading of the concentration is due to the resolution of the analyser and of the data acquisition. The uncertainty can be considered as negligible.

The uncertainty contribution due to lack of fit is determined from the maximum deviation $\delta\gamma_{\text{of,max}}$ between the measured value and the value given through the linear regression achieved during the laboratory test. With the assumption that the lack of fit has an equal probability to take any value included in the interval $[-\delta\gamma_{\text{of,max}}; +\delta\gamma_{\text{of,max}}]$, the standard uncertainty is calculated by application of a rectangular probability distribution.

For the zero and the span drift, it is assumed that the drift has the same probability to take a value included in the intervals $[-\delta\gamma_{\text{d,z}}; +\delta\gamma_{\text{d,z}}]$ and $[-\delta\gamma_{\text{d,s}}; +\delta\gamma_{\text{d,s}}]$. The standard uncertainties are calculated by application of a rectangular probability distribution.

The standard uncertainty due to the repeatability is equal to the standard deviation of repeatability calculated from the results of the repetitions of the measurements. Several tests can be carried out under different concentrations (at least at zero and at span level). Only one of these values is included in the uncertainty estimation. The value can be selected, e.g. on the basis of the intended measurement range, i.e. as the standard deviation of repeatability corresponding to the closest concentration measured in the stack, or as the highest standard deviation of repeatability regardless of the concentration measured in the stack.

The uncertainty contribution due to adjustment is calculated from the uncertainty of the calibration gas. In general, the uncertainty given by the manufacturer is the expanded uncertainty. If the expanded uncertainty U_{cal} of the calibration gas is expressed as a percentage value, the standard uncertainty due to adjustment at the measurement range γ_{mr} is given by Equation (C.9):

$$u_{\text{adj}} = \frac{U_{\text{cal}}}{2} \gamma_{\text{mr}} \quad (\text{C.9})$$

The influence quantities, such as ambient temperature, atmospheric pressure, sample volume flow, and voltage, are tested for one value of the parameter and the effects of the influence quantities are assumed to be proportional to the value of the parameter. The correction of the effect of an influence quantity j , $\delta\gamma_{\text{inf},j}$, is also proportional to its variation:

$$\delta\gamma_{\text{inf},j} = b_j \delta x_j \quad (\text{C.10})$$

where

δx_j is the change in the influence quantity j ;

b_j is the sensitivity coefficient of influence quantity j .

The standard uncertainty due to influence quantity j is given by Equation (C.11):

$$u_{\text{inf},j} = b_j u(x_j) \quad (\text{C.11})$$

where $u(x_j)$ is the uncertainty associated with the variation of influence quantity j .

The sensitivity coefficient of an influence quantity is determined during the laboratory tests. The standard uncertainty associated with the variation of the influence quantity can be estimated on the basis of a rectangular distribution for the values x_j of the influence quantity.

The case of the interferences is similar to the case of influence quantities. The influence of interferences is tested for one concentration of an interferent and is likely to be proportional to the value of the parameter, i.e. the correction of the effect of an interferent is proportional to its variation.

C.3.4 Results of uncertainty calculation

Table C.3 — Results of uncertainty calculation

Performance characteristic	Standard uncertainty	Value of standard uncertainty at upper limit of the lowest measuring range used mg/m ³
Lack of fit	u_{lof}	$\frac{(2/100) \times 400}{\sqrt{3}} = 4,62$
Zero drift	$u_{\text{d,z}}$	$\frac{(1/100) \times 400}{\sqrt{3}} = 2,31$
Span drift	$u_{\text{d,s}}$	$\frac{(1/100) \times 400}{\sqrt{3}} = 2,31$
Standard deviation of repeatability in laboratory at zero	u_{rep}	$(0,2/100) \times 400 = 0,8$
Uncertainty of reference gas	u_{adj}	$\frac{(2/100) \times 320}{2} = 3,2$
Interferents	$u_{\text{i,CO}_2}$	$\frac{2,8}{14} \times \frac{14}{\sqrt{3}} = 1,62$
	$u_{\text{i,CO}}$	$\frac{2,3}{300} \times \frac{300}{\sqrt{3}} = 1,33$
	$u_{\text{i,NO}_2}$	$\frac{0,4}{40} \times \frac{40}{\sqrt{3}} = 0,23$
	$u_{\text{i,NO}}$	$\frac{0,1}{320} \times \frac{320}{\sqrt{3}} = 0,06$
	$u_{\text{i,SO}_2}$	$\frac{0,1}{200} \times \frac{200}{\sqrt{3}} = 0,06$
Sensitivity to the sample volume flow	$u_{\text{inf,svf}}$	$\frac{(1/100) \times 400}{\sqrt{3}} = 2,31$
Sensitivity to atmospheric pressure	$u_{\text{inf,ap}}$	$\frac{(2/100) \times 400}{\sqrt{3}} = 4,62$
Sensitivity to ambient temperature	$u_{\text{inf,at}}$	$\frac{(1/100)}{10} \times 400 \times \sqrt{\frac{(23)^2 + (23)(-2) + (-2)^2}{3}} = 5,04$
Sensitivity to electric voltage at span level	$u_{\text{inf,v}}$	$\frac{(1/100)}{10} \times 400 \times \frac{(220 - 200)}{\sqrt{3}} = 4,62$

C.3.5 Combined uncertainty

The combined uncertainty, $u(\gamma_{\text{N}_2\text{O}})$, in milligrams per cubic metre, is calculated according to Equation (C.12):

$$u(\gamma_{\text{N}_2\text{O}}) = \sqrt{u_i^2} = 10,5 \quad (\text{C.12})$$

C.3.6 Expanded uncertainty

The expanded uncertainty, $U(\chi_{\text{N}_2\text{O}})$, in milligrams per cubic metre, is calculated according to Equation (C.13):

$$\begin{aligned} U(\chi_{\text{N}_2\text{O}}) &= 1,96 \, u(\chi_{\text{N}_2\text{O}}) \\ &= 20,6 \end{aligned} \quad (\text{C.13})$$

C.4 Evaluation of the compliance with the required measurement quality

The sum of the effects of interferences with a positive impact is obtained as:

$$S = 2,8 \% + 2,3 \% + 0,4 \% + 0,1 \% + 0,1 \% = 5,7 \% \quad (\text{C.14})$$

This sum is smaller than the performance criterion of 6 % of the range.

The combined standard uncertainty and the expanded uncertainty obtained here are 10,5 mg/m³ and 20,6 mg/m³, respectively, which are lower than 18,1 mg/m³ and 36,2 mg/m³, respectively, which are calculated by using the values of performance criteria in Table 1.

The value of the performance characteristics obtained from the general performance test, laboratory test and test during field operation comply with requirements.

Conclusion: the measurement method fulfils the requirements.