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Stationary source emissions — Determination of the mass concentration of dinitrogen monoxide (N_2O) — Reference method: Non-dispersive infrared method

Émissions de sources fixes — Détermination de la concentration massique de protoxyde d'azote (N_2O) — Méthode de référence: Méthode infrarouge non dispersive

Reference number
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Foreword

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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.

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

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.

Stationary source emissions — Determination of the mass concentration of dinitrogen monoxide (N_2O) — Reference method: Non-dispersive infrared method

1 Scope

This International Standard specifies a method for sampling, sample conditioning and determination of dinitrogen monoxide (N_2O) 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 N_2O concentration in the flue gas was up to about 200 mg/m^3.

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_2O at actual oxygen concentration
γ_d	concentration of N_2O on dry basis
γ_n	normalized concentration of N_2O
γ_w	measured concentration of N_2O on wet basis
$\bar{\gamma}$	grand mean of measured N_2O concentration
$\varphi_{H_2O,m}$	measured water vapour content, as a percentage volume fraction
$\varphi_{O_2,m}$	measured oxygen content, as a volume fraction, in the waste gas
$\varphi_{O_2,ref}$	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_2O
$U(\gamma_{N_2O})$	expanded uncertainty of the measured concentration of N_2O

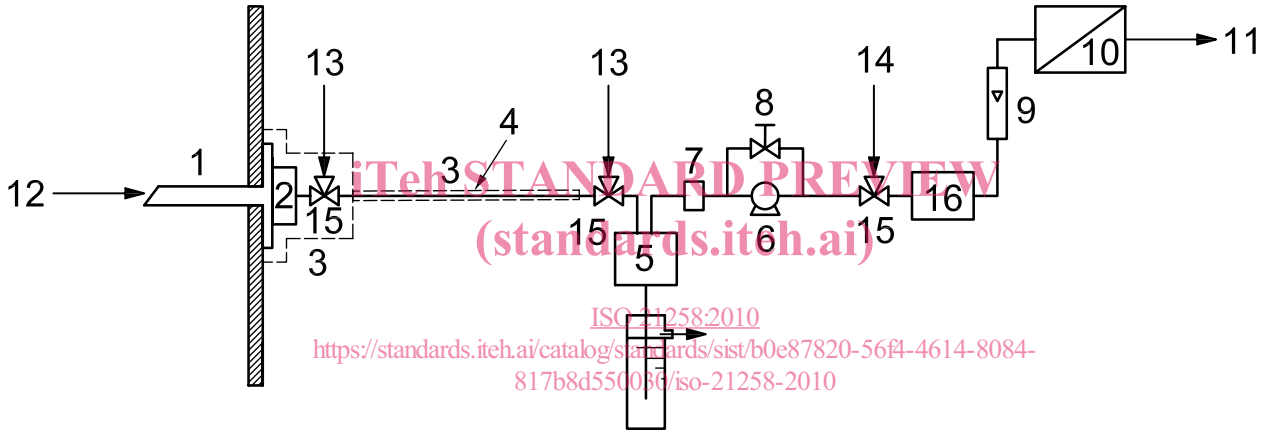
5 Principle

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 N_2O from NO_2 and SO_2 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 CO_2 which can be corrected for later, in order to decrease the influence of the interferent.