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Stationary source emissions - Determination of the mass concentration of nitrogen oxides in flue gas - Performance characteristics of automated measuring systems

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Émissions de sources fixes - Détermination de la concentration en masse des oxydes d'azote dans les effluents gazeux - Caractéristiques de performance des systèmes de mesurage automatiques

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**Stationary source emissions —
Determination of the mass
concentration of nitrogen oxides
in flue gas — Performance
characteristics of automated
measuring systems**

*Émissions de sources fixes — Détermination de la concentration en
masse des oxydes d'azote dans les effluents gazeux — Caractéristiques
de performance des systèmes de mesurage automatiques*

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ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

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

This second edition cancels and replaces the first edition (ISO 10849:1996), which has been technically revised.

The main changes are as follows:

- the structure and the components have been updated to be similar to the latest editions of e.g. ISO 12039 (measurement of CO, CO₂ and O₂), ISO 17179 (measurement of NH₃), ISO 13199 (measurement of total VOC), ISO 25140 (measurement of CH₄), ISO 21258 (measurement of N₂O);
- [Clause 3](#) has been updated (addition or deletion and change in terms and definitions);
- a new analytical technique has been added (Fourier transform infrared spectroscopy) for measurement of NO and NO₂ or NO_x;
- the performance characteristics and criteria as well as QA/QC procedures have been changed to harmonize with latest ISO standards;
- examples of performance test results and the results of uncertainty calculation have been added for NO and NO₂ or NO_x measurement.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Nitrogen oxides are produced during most combustion processes. In fossil fuel combustion, nitrogen oxides are produced from nitrogen contained in the fuel and from the oxidation of nitrogen in the air used for combustion. The quantity of nitrogen oxides produced depends upon the nitrogen content of the fuel, the combustor design, and the combustor operating conditions.

In flue gases from conventional boiler combustion systems, the nitrogen oxides consist of approximately 95 % nitrogen monoxide (NO). The remaining oxide is predominantly nitrogen dioxide (NO₂) formed from the oxidation of NO when the flue gas temperature decreases. These two oxides (NO + NO₂) are generally designated as NO_x. It should be noted that in other processes the ratio of NO to NO₂, may be different and other nitrogen oxides may be present.

There are numerous ways of determining nitrogen oxides in the gases of combustion plants, both wet chemical/analytical methods and instrumental techniques.

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Stationary source emissions — Determination of the mass concentration of nitrogen oxides in flue gas — Performance characteristics of automated measuring systems

1 Scope

This document specifies a method for the determination of nitrogen oxides (NO_x) in flue gas of stationary sources and describes the fundamental structure and the key performance characteristics of automated measuring systems.

The method allows continuous monitoring with permanently installed measuring systems of NO_x emissions.

This document describes extractive systems and in situ (non-extractive) systems in connection with a range of analysers that operate using, for example, the following principles:

- chemiluminescence (CL);
- infrared absorption (NDIR);
- Fourier transform infrared (FTIR) spectroscopy;
- ultraviolet absorption (NDUV);
- differential optical absorption spectroscopy (DOAS);

Other equivalent instrumental methods such as laser spectroscopic techniques can be used provided they meet the minimum performance requirements specified in this document. The measuring system can be validated with reference materials, in accordance with this document, or comparable methods.

Automated measuring system (AMS) based on the principles listed above has been used successfully in this application for the measuring ranges as shown in [Annex F](#).

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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, *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*

3 Terms and definitions

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

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>

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— IEC Electropedia: available at <https://www.electropedia.org/>

3.1

analyser

analytical part in an extractive or in situ *automated measuring system* (3.2)

[SOURCE: ISO 12039:2019, 3.1]

3.2

automated measuring system**AMS**

measuring system interacting with the flue gas under investigation, returning an output signal proportional to the physical unit of the *measurand* (3.9) in unattended operation

[SOURCE: ISO 9169:2006, 2.1.2, modified — Note is removed]

Note 1 to entry: For the purposes of this document, an AMS is a system that can be attached to a duct or stack to continuously or intermittently measure the mass concentration of NO, NO₂ or NO_x passing through the duct.

3.3

in situ AMS

non-extractive system that measures the concentration directly in the duct or stack

Note 1 to entry: In situ systems measure either across the stack or duct or at a point within the duct or stack.

3.4

parallel measurements

measurements taken on the same duct in the same sampling plane for the same period of time with the AMS under test and with the reference method at points a short distance from each other, providing pairs of measured values

Note 1 to entry: See 3.21.

3.5

independent reading

reading that is not influenced by a previous individual reading by separating two individual readings by at least four response times

3.6

interference**cross-sensitivity**

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

3.7

interferent**interfering substance**

substance present in the air mass under investigation, other than the *measurand* (3.9), that affects the response of AMS (3.2)

3.8

lack-of-fit

systematic deviation within the range of application, between the accepted value of a reference material applied to the measuring system and the corresponding result of measurement produced by the measuring system

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

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

[SOURCE: ISO 9169:2006, 2.2.9, modified — Note 2 is removed.]

3.9**measurand**

particular quantity subject to measurement

[SOURCE: ISO/IEC Guide 98-3:2008, B.2.9, modified— Example and Note is removed.]

3.10**NO₂/NO converter efficiency**

ratio with which the converter device of a NO_x analyser reduces NO₂ to NO

3.11**performance characteristic**

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

Note 1 to entry: Performance characteristics can be described by values, tolerances, or ranges.

3.12**period of unattended operation**

maximum interval of time for which the performance characteristics remain within a predefined range without external servicing, e.g. refill, adjustment

[SOURCE: ISO 9169:2006, 2.2.11]

Note 1 to entry: The period of unattended operation is often called maintenance interval.

3.13**reference material**

substance or mixture of substances with a known concentration within specified limits, or a device of known characteristics

Note 1 to entry: Normally calibration gases, gas cells, gratings or filters are used.

[SOURCE: ISO 14385-1:2014, 3.20]

3.14**reference method**

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

Note 1 to entry: See [3.4](#).

3.15**transport 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 instrument

3.16**response time**

time interval between the instant when a stimulus is subjected to bring about 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

[SOURCE: ISO 9169:2006, 2.2.4]

Note 1 to entry: Lag time, rise time and fall time are defined in ISO 9169:2006.

3.17**span gas**

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

Note 1 to entry: The concentration is often chosen around 70 % to 90 % of full scale.

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3.18

span point

value of the output quantity (measured signal) of the automated measuring system for the purpose of calibration, adjustment, etc. that represents a correct measured value generated by reference gas

3.19

standard uncertainty

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

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

3.20

uncertainty

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

[SOURCE: ISO/IEC Guide 98-3: 2008, 2.2.3 modified — Note 1, 2 and 3 removed.]

3.21

validation of an automated measuring system

procedure to check the statistical relationship between values of the measurand indicated by the automated measuring system and the corresponding values given by parallel measurements implemented simultaneously at the same measuring point

3.22

zero gas

gas or gas mixture used to establish the *zero point* (3.23) on a calibration curve within a given concentration range

[SOURCE: ISO 12039:2019, 3.20]

3.23

zero point specified value of the output quantity (measured signal) of the AMS and which, in the absence of the measured component, represents the zero crossing of the calibration line

4 Symbols and abbreviated terms

e_i	Residual (lack-of-fit) at level i
K	Coverage factor
N	Number of measurements
s_r	Standard deviation of repeatability
$u(\gamma_X)$	Combined uncertainty of X (NO or NO ₂) mass concentration
$U(\gamma_X)$	Expanded uncertainty of X (NO or NO ₂) mass concentration
M_x	Molar mass of X (NO or NO ₂ , g/mol)
V_M	Molar volume (22,4 l/mol at standard conditions, 273,15 K; 101,325 kPa)
φ_X	Volume fraction of X (NO or NO ₂)
γ_X	X (NO or NO ₂) mass concentration at standard conditions in mg/m ³ (273,15 K; 101,325 kPa)
γ_R	NO or NO ₂ mass concentration at reference conditions in mg/m ³ (273,15 K; 101,325 kPa; H ₂ O corrected)

\bar{x}	Average of the measured values x_i
x_i	i th measured value
\bar{x}_i	Average of the measured value at level i
\hat{x}_i	Value estimated by the regression line at level i
AMS	Automated measuring system
FTIR	Fourier transform infrared
NDIR	Non-dispersive infrared
NDUV	Non-dispersive ultraviolet
DOAS	Differential optical absorption spectroscopy
QA	Quality assurance
QC	Quality control

5 Principle

This document describes automated measurement systems for sampling, sample conditioning, and determining NO and NO₂ or NO_x content in flue gas using instrumental methods (analysers).

There are two types of automated measuring systems:

- extractive systems;
- in situ systems.

With extractive systems, a representative sample of gas is taken from the stack with a sampling probe and conveyed to the analyser through the sample line and sample gas conditioning system.

In situ systems do not require any sampling transfers out of the stack. For the installation of these systems, a representative place in the stack is to be chosen.

The systems described in this document measure NO and NO₂ or NO_x concentrations using instrumental methods that shall meet the minimum performance specifications given.

In most of the cases, it is considered that only NO is measured, because the NO₂ content is much smaller than NO. However, in some cases NO₂ may exist in large quantities and shall be taken into account, either by direct measurement or by using a converter of NO₂ to NO. The sampling is more complex.

6 Description of the automated measuring systems

6.1 Sampling and sample gas conditioning systems

Sampling and sample gas conditioning systems for extractive and in situ methods shall conform to ISO 10396.

In extractive sampling, these gases are conditioned to remove aerosols, particulate matter and other interfering substances before being conveyed to the instruments. Three kinds of extractive systems as well as non-extractive systems are described in ISO 10396:

- a) Cold-dry,
- b) Hot-wet and

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- c) Dilution. In non-extractive sampling, the measurements are made in situ; therefore, no sample conditioning is required.

The details of the extractive sampling and sample gas conditioning systems are described in [Annex A](#) and two kinds of in situ system are illustrated in [Annex B](#).

6.2 Analyser equipment

Examples of the typical analytical methods available are described in [Annex A](#) and [Annex B](#).

The NO₂ to NO converter is necessary if NO₂ is measured with an NO analyser (only required in combination with extractive systems). The details of the converter and the test method for the performance characteristics are described in [Annex C](#).

AMS shall meet the performance characteristics as described in [Clause 7](#).

7 Performance characteristics and criteria

7.1 Performance criteria

[Table 1](#) gives the performance characteristics and performance criteria of the analyser and measurement system to be evaluated during performance test, by means of ongoing QA/QC in the laboratory and during field operation. Test procedures for the performance test are specified in [Annex E](#).

Table 1 — Performance characteristics and criteria of AMS for measurement of NO and NO₂

Performance characteristic	Performance criterion	Test procedure
Response time	≤ 200 s	E.2
Standard deviation of repeatability in laboratory at zero point	≤ 2,0 % of the upper limit of the lowest measuring range used	E.3.2
Standard deviation of repeatability (NO or NO ₂) in laboratory at span point	≤ 2,0 % of the upper limit of the lowest measuring range used	E.3.3
Lack-of-fit	≤ 2,0 % of the upper limit of the lowest measuring range used	E.4
Zero drift within 24 h	≤ 2,0 % of the upper limit of the lowest measuring range used	E.5
Span drift within 24 h	≤ 2,0 % of the upper limit of the lowest measuring range used	E.5
Zero drift within the period of unattended operation	≤ 3,0 % of the upper limit of the lowest measuring range used	E.6
Span drift within the period of unattended operation	≤ 3,0 % of the upper limit of the lowest measuring range used	E.6
Sensitivity to ambient temperature, for a change of 20 K in the temperature range specified by the manufacturer	≤ 5,0 % of the upper limit of the lowest measuring range used	E.7
Sensitivity to sample gas pressure, for a pressure change of 3 kPa	≤ 2,0 % of the upper limit of the lowest measuring range used	E.8
Sensitivity to sample gas flow for an extractive AMS	≤ 2,0 % of the upper limit of the lowest measuring range used	E.9
Sensitivity to electric voltage in the range -15 % below or +10 % above from the nominal voltage stated by the manufacturer	≤ 2,0 % of the upper limit of the lowest measuring range used	E.10
Cross-sensitivity	≤ 4,0 % of the upper limit of the lowest measuring range used	E.11

Table 1 (continued)

Performance characteristic	Performance criterion	Test procedure
Losses and leakage in the sampling line and conditioning system	$\leq 2,0$ % of the measured value	E.12 and E.13
Excursion of the measurement beam of cross-stack in situ AMS	≤ 2 % of the measured value of the lowest measuring range used	E.14
NO ₂ /NO converter efficiency if applicable	$\geq 95,0$ %	Annex C

7.2 Determination of the performance characteristics

7.2.1 Performance test

The performance characteristics of the AMS shall be determined during the 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 E](#).

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

The measurement uncertainty of the AMS measured values shall be calculated in accordance with ISO 14956 on the basis of the performance characteristics determined during the performance test and shall meet the level of uncertainty appropriate for the intended use. These characteristics may be determined either by the manufacturer or by the user.

7.2.2 Ongoing quality control

The user shall check specific performance characteristics during ongoing operation of the measuring system with a periodicity specified in [Table 2](#).

The measurement uncertainty during field application shall be determined by the user of the measuring system in accordance with applicable international or national standards. For process monitoring, the level of uncertainty shall be appropriate for the intended use. It can be determined by a direct or an indirect approach for uncertainty estimation as described in ISO 20988. 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 contributions due to:

- the sampling line and conditioning system,
- the site specific conditions, and
- the reference materials used.

8 Selection and installation procedure

8.1 Choice of the measuring system

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

- ambient temperature range;
- temperature range of the flue gas;
- water vapour content of the flue gas;
- dust loading of the flue gas;
- expected concentration range of NO, NO₂ or NO_x;