BS ISO 17179:2016

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

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## National foreword

This British Standard is the UK implementation of ISO 17179:2016.

The UK participation in its preparation was entrusted to Technical Committee EH/2/1, Stationary source emission.

A list of organizations represented on this committee can be obtained on request to its secretary.

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Published by BSI Standards Limited 2016

ISBN 978 0 580 84130 9

ICS 13.040.40

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This British Standard was published under the authority of the Standards Policy and Strategy Committee on 30 June 2016.

**Amendments/corrigenda issued since publication**

| Date | Text affected |
|---|---|

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# INTERNATIONAL STANDARD

# ISO 17179

First edition
2016-07-01

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

*Émission des sources fixes — Détermination de la concentration massique de l'ammoniac dans les gaz de combustion — Caractéristiques de performance des systèmes de mesure automatisés*

Reference number
ISO 17179:2016(E)

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# Foreword

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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).

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For an explanation on 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 the following URL: www.iso.org/iso/foreword.html.

The committee responsible for this document is ISO/TC 146, *Air quality*, Subcommittee SC 1, *Stationary source emissions*.


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# Introduction

Selective Catalytic Reduction (SCR) and Selective Non-Catalytic Reduction (SNCR) $NO_x$ control systems are used for emission control of $NO_x$ in flue gas from power generation plants, waste incinerators and others. The $NO_x$ reduction technologies require the injection of ammonia ($NH_3$) and/or urea into flue gas. The SCR system is designed to be operated at unreacted $NH_3$ in flue gas (or remained $NH_3$ in flue gas) as small as possible (typically below 2 mg/m$^3$ to 4 mg/m$^3$ $NH_3$ concentration) with more than 90 % $NO_x$ reduction efficiency. The standardization of a measurement method of $NH_3$ is thus strongly desired for efficient operation and maintenance of the $NO_x$ control systems and for minimization of environmental impacts due to ammonia and $NO_x$.

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

## 1 Scope

This International Standard specifies the fundamental structure and the most important performance characteristics of automated measuring systems for ammonia ($NH_3$) to be used on stationary source emissions, for example, combustion plants where SNCR/SCR $NO_x$ control systems (de$NO_x$ systems) are applied. The procedures to determine the performance characteristics are also specified. Furthermore, it describes methods and equipment to determine $NH_3$ in flue gases including the sampling system and sample gas conditioning system.

This International Standard describes extractive systems, based on direct and indirect measurement methods, and *in situ* systems, based on direct measurement methods, in connection with a range of analysers that operate using, for example, the following principles:

— ammonia conversion to, or reaction with NO, followed by chemiluminescence (CL) $NO_x$ difference measurement for ammonia (differential $NO_x$);

— ammonia conversion to, or reaction with NO, followed by non-dispersive ultraviolet (NDUV) spectroscopy $NO_x$ difference measurement for ammonia (differential $NO_x$);

— Fourier transform infrared (FTIR) spectroscopy;

— non-dispersive infrared (NDIR) spectroscopy with gas filter correlation (GFC);

— tuneable laser spectroscopy (TLS).

The method allows continuous monitoring with permanently installed measuring systems of $NH_3$ emissions, and is applicable to measurements of $NH_3$ in dry or wet flue gases, for process monitoring, long term monitoring of the performance of de$NO_x$ systems and/or emission monitoring.

Other equivalent instrumental methods can be used, provided they meet the minimum requirements proposed in this International Standard. The measuring system can be calibrated with certified gases, in accordance with this International Standard, or comparable methods.

The differential $NO_x$ technique using CL has been successfully tested on some power plants where the $NO_x$ concentration and $NH_3$ concentration in flue gas after de$NO_x$ systems are up to 50 mg (NO)/$m^3$ and 10 mg ($NH_3$)/$m^3$, respectively. AMS based on FTIR, NDIR with GFC and TLS has been used successfully in this application for measuring ranges as low as 10 mg ($NH_3$)/$m^3$.

## 2 Normative references

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

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*

ISO 20988, *Air quality — Guidelines for estimating measurement uncertainty*

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## 3 Terms and definitions

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

**3.1**
**analyser**
analytical part in an extractive or *in situ AMS* (3.3)

[SOURCE: ISO 12039:2001, 3.3]

**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* (3.11) in unattended operation

Note 1 to entry: In the sense of this International Standard, an AMS is a system that can be attached to a duct or stack to continuously or intermittently measure the mass concentration of $NH_3$ passing through the duct.

[SOURCE: ISO 9169:2006, 2.1.2, modified.]

**3.3**
***in situ* AMS**
non-extractive systems that measure 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**
**calibration of an automated measuring system**
procedure for establishing the statistical relationship between values of the *measurand* (3.11) indicated by the *automated measuring system* (3.2) and the corresponding values given by an independent method of measurement implemented simultaneously at the same measuring point

**3.5**
**efficiency of $NH_3$/NO**
efficiency of a converter which oxidizes $NH_3$ to NO

**3.6**
**efficiency of $NH_3/N_2$**
efficiency of a converter which reduces $NH_3$ to $N_2$

**3.7**
**influence quantity**
quantity that is not the *measurand* (3.11) but that affects the result of the measurement

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

**3.8**
**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* (3.11)

**3.9**
**interferent**
**interfering substance**
substance present in the air mass under investigation, other than the *measurand* (3.11), that affects the response

[SOURCE: ISO 9169:2006, 2.1.12]


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**3.10**
**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* (3.16) and the corresponding accepted value of such reference materials

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

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.]

**3.11**
**measurand**
particular quantity subject to measurement

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

**3.12**
**$NO_2$/NO converter efficiency**
efficiency with which the converter unit of a $NO_x$ analyser reduces $NO_2$ to NO

**3.13**
**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.

**3.14**
**period of unattended operation**
maximum interval of time for which the *performance characteristics* (3.13) remain within a predefined range without external servicing, e.g. refill, adjustment

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

[SOURCE: ISO 9169:2006, 2.2.11]

**3.15**
**reference gas**
gaseous mixture of stable composition used to calibrate the measuring system and which is traceable to national or international standards

**3.16**
**reference material**
**RM**
substance or mixture of substances with a known concentration within specified limits, or a device of known characteristics

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

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

**3.17**
**reference method**
measurement method taken as a reference by convention, which gives the accepted reference value of the *measurand* (3.11)

**3.18**
**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

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**3.19**
**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

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

[SOURCE: ISO 9169:2006, 2.2.4]

**3.20**
**span gas**
gas or gas mixture used to adjust and check the *span point* (3.21) on the response line of the measuring system

Note 1 to entry: This concentration is often chosen around 70 % to 80 % of full scale.

**3.21**
**span point**
value of the output quantity (measured signal) of the *automated measuring system* (3.2) for the purpose of calibration, adjustment, etc. that represents a correct measured value generated by *reference material* (3.16)

**3.22**
**standard uncertainty**
*uncertainty* (3.23) of the result of a measurement expressed as a standard deviation

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

**3.23**
**uncertainty (of measurement)**
parameter, associated with the result of a measurement, that characterizes the dispersion of the values that could reasonably be attributed to the *measurand* (3.11)

[SOURCE: ISO/IEC Guide 98-3:2008, 2.2.3]

**3.24**
**validation of an automated measuring system**
procedure to check the statistical relationship between values of the *measurand* (3.11) indicated by the *automated measuring system* (3.2) and the corresponding values given by an independent method of measurement implemented simultaneously at the same measuring point

**3.25**
**zero gas**
gas or gas mixture used to establish the *zero point* (3.26) on a calibration curve within a given concentration range

[SOURCE: ISO 12039:2001, 3.4.2]

**3.26**
**zero point**
specified value of the output quantity (measured signal) of the *AMS* (3.2) and which, in the absence of the measured component, represents the zero crossing of the calibration line


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## 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 |
| $u(\gamma_{NH_3})$ | Combined uncertainty of $NH_3$ mass concentration |
| $U(\gamma_{NH_3})$ | Expanded uncertainty of $NH_3$ mass concentration |
| $M_c$ | Molar mass of $NH_3$ (=17,031 g/mol) |
| $V_M$ | Molar volume (22,4 l/mol) |
| $\phi_{NH_3}$ | Volume fraction of $NH_3$ |
| $\gamma_{NH_3}$ | $NH_3$ mass concentration in mg/m$^3$ |
| $\gamma_S$ | $NH_3$ mass concentration at standard conditions in mg/m$^3$ (273,15 K; 101,325 kPa) |
| $\gamma_R$ | $NH_3$ mass concentration at reference conditions in mg/m$^3$ (273,15 K; 101,325 kPa; $O_2$ and $H_2O$ 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 |
| CL | Chemiluminescence |
| FTIR | Fourier transform infrared |
| GFC | Gas filter correlation |
| NDIR | Non-dispersive infrared |
| NDUV | Non-dispersive ultraviolet |
| QA | Quality assurance |
| QC | Quality control |
| SCR | Selective catalytic reduction |
| SNCR | Selective non-catalytic reduction |
| TLS | Tuneable laser spectroscopy |

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# 5 Principle

This International Standard describes automated measurement systems for sampling, sample conditioning, and determining $NH_3$ content in flue gas using instrumental methods (analysers).

There are two types of automated measuring systems:

— extractive systems;

— *in situ* systems.

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

*In situ* systems do not require any sample processing. For the installation of these systems, a representative place in the stack is to be chosen.

The systems described in this International Standard measure $NH_3$ concentration using instrumental methods that shall meet the minimum performance specifications given.

# 6 Description of the automated measuring systems

## 6.1 Sampling and sample gas conditioning systems

Since ammonia ($NH_3$) is condensable and highly reactive component, there are many opportunities for loss of sample integrity due to condensation, deposition or other loss of ammonium compounds in the sample transport system.

The details of the sampling and sample gas conditioning systems for each of automated measuring systems are described in Annex A, Annex B, and Annex C.

## 6.2 Analyser equipment

Examples of the typical analytical methods available are described in Annex A ($NO/NO_x$ CL technique and NDUV technique), Annex B (FTIR and NDIR with GFC technique) and Annex C (TLS technique).

Instruments that use these techniques shall meet the performance characteristics as described in Clause 7.

# 7 Performance characteristics

## 7.1 Performance criteria

Table 1 gives the performance characteristics and performance criteria of the analyser and measurement system to be evaluated during general 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.


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**Table 1 — Main performance characteristics and criteria of AMS for measurement of ammonia**

| Performance characteristic | Performance criterion | Test procedure |
|---|---|---|
| Response time | ≤400 s[a]<br>≤900 s[b] | E.2 |
| Standard deviation of repeatability in laboratory at zero point | ≤2 % of the upper limit of the lowest measuring range used | E.3.2 |
| Standard deviation of repeatability ($NH_3$) in laboratory at span point | ≤2 % of the upper limit of the lowest measuring range used | E.3.3 |
| Lack-of-fit | ≤ ±2 % of the upper limit of the lowest measuring range used | E.4 |
| Zero drift within 24 h | ≤ ±2 % of the upper limit of the lowest measuring range used | E.10 |
| Span drift within 24 h | ≤ ±2 % of the upper limit of the lowest measuring range used | E.10 |
| Sensitivity to sample gas pressure, for a pressure change of 2 kPa | ≤ ±3 % of the upper limit of the lowest measuring range used | E.13 |
| Zero drift within the period of unattended operation | ≤ ±3 % of the upper limit of the lowest measuring range used | E.11 |
| Span drift within the period of unattended operation | ≤ ±3 % of the upper limit of the lowest measuring range used | E.11 |
| Sensitivity to ambient temperature, for a change of 10 K in the temperature range specified by the manufacturer | ≤ ±3 % of the upper limit of the lowest measuring range used | E.14 |
| Sensitivity to electric voltage in the voltage range specified by the manufacturer | ≤ ±2 % of the upper limit of the lowest measuring range used per 10 V[c] | E.15 |
| Cross-sensitivity | ≤4 % of the upper limit of the lowest measuring range used | E.5 |
| $NO_2$/NO converter efficiency, if applicable | ≥95 % | E.6 |
| $NH_3$/NO converter efficiency, if applicable | ≥90 % | E.7 |
| $NH_3$/$N_2$ converter efficiency, if applicable | ≥95 % | E.7 |
| Losses and leakage in the sampling line and conditioning system | ≤2 % of the measured value | E.8 for losses and E.9 for leakage |
| Excursion of the measurement beam of cross-stack *in situ* AMS | ≤2 % of the measured value of the lowest measuring range used | E.12 |

[a] For emission monitoring from $deNO_x$ systems.

[b] For long term monitoring of changes in the $deNO_x$ systems, especially the reduction of catalyst activity.

[c] In the case of a nominal supply voltage of 200 V.

The measuring range is defined by two values of the measurand, or quantity to be supplied, within which the limits of uncertainty of the measuring instrument are specified. The upper limit of the lowest measuring range used should be set suitable to the application, such that the measurement values lie within 20 % to 80 % of the measuring range.

## 7.2 Determination of the performance characteristics

### 7.2.1 Performance test

The performance characteristics of the AMS 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 described in Annex E.

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

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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 general performance test and shall meet the level of uncertainty appropriate for the intended use.

#### 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 (non-regulatory application), 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 calibration gases used.

## 8 Measurement procedure

### 8.1 General

The AMS shall be operated according to the manufacturer's instructions.

The QA/QC procedures specified in Clause 9 shall be strictly observed.

During the measurement, the ambient conditions should be in the ranges applied during the general performance test.

### 8.2 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 of the flue gas;

— water vapour content of the flue gas;

— dust load of the flue gas;

— expected concentration range of $NH_3$;

— expected concentration of potentially interfering substances.

To avoid long response times and memory effects, the sampling 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, an appropriate heated filter shall be used.

Before monitoring emissions, the user shall verify that the necessary QA/QC procedures have been performed.

NOTE Information on QA/QC procedures is provided in ISO 14385-1[3] and ISO 14385-2.[4]


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