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Stationary source emissions — Greenhouse gases —

Part 1: Calibration of automated measuring systems

Émissions de sources fixes — Gaz à effet de serre —

Partie 1: Étalonnage des systèmes de mesurage automatiques

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Foreword

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

ISO 14385 consists of the following parts, under the general title *Stationary source emissions — Greenhouse gases*:

— *Part 1: Calibration of automated measuring systems*

— *Part 2: Ongoing quality control of automated measuring systems*

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Introduction

The measurement of greenhouse gas emissions (carbon dioxide, nitrous oxide, methane) in a framework of emission trading requires an equal and known quality of data.

This part of ISO 14385 describes the quality assurance procedures for calibration and ongoing quality control needed to ensure that automated measuring systems (AMS) installed to measure emissions of greenhouse gases to air are capable of meeting the uncertainty requirements on measured values specified, e.g. by legislation, competent authorities, or in an emission trade scheme.

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Stationary source emissions — Greenhouse gases —

Part 1:
Calibration of automated measuring systems

1 Scope

This part of ISO 14385 specifies the procedures for establishing quality assurance for automated measuring systems (AMS) installed on industrial plants for the determination of the concentration of greenhouse gases in flue and waste gas and other flue gas parameters.

This part of ISO 14385 specifies a procedure to calibrate the AMS and determine the variability of the measured values obtained by an AMS, which is suitable for the validation of an AMS following its installation.

This part of ISO 14385 is designed to be used after the AMS has been accepted according to the procedures specified in ISO 14956.

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 14385-2, *Stationary source emissions — Greenhouse gases — Part 2: Ongoing quality control of automated measuring systems*

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.

3.1
automated measuring system
AMS
measuring system permanently installed on site for continuous monitoring of emissions

Note 1 to entry: An AMS is a method which is traceable to a reference method.

Note 2 to entry: Apart from the analyser, an AMS includes facilities for taking samples (e.g. sample probe, sample gas lines, filters, flow meters, regulators, delivery pumps, blowers) and for sample conditioning (e.g. dust filter, water vapour removal devices, converters, diluters). This definition also includes testing and adjusting devices that are required for regular functional checks.

3.2
calibration function
linear relationship between the values of the SRM and the AMS with the assumption of a constant residual standard deviation

3.3
calibration gas
gas of known composition that can be used to check the response of the AMS

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3.4
competent authority
organization or organizations which implement the requirements of legislation and regulate installations which must comply with the requirements of legislation

3.5
confidence interval
interval estimator (T_1, T_2) for the parameter θ with the statistics T_1 and T_2 as interval limits and for which it holds that $P[T_1 < \theta < T_2] \geq 1 - \alpha$

Note 1 to entry: The two-sided 95 % confidence interval of a normal distribution is illustrated in Figure 1, where

$T_1 = \theta - 1{,}96\sigma_0$	is the lower 95 % confidence limit;
$T_2 = \theta + 1{,}96\sigma_0$	is the upper 95 % confidence limit;
$I = T_2 - T_1 = 2 \times 1{,}96 \times \sigma_0$	is the length of the 95 % confidence interval;
$\sigma_0 = I / (2 \times 1{,}96)$	is the standard deviation associated with a 95 % confidence interval;
n	is the number of observed values;
f	is the frequency;
m	is the measured value.

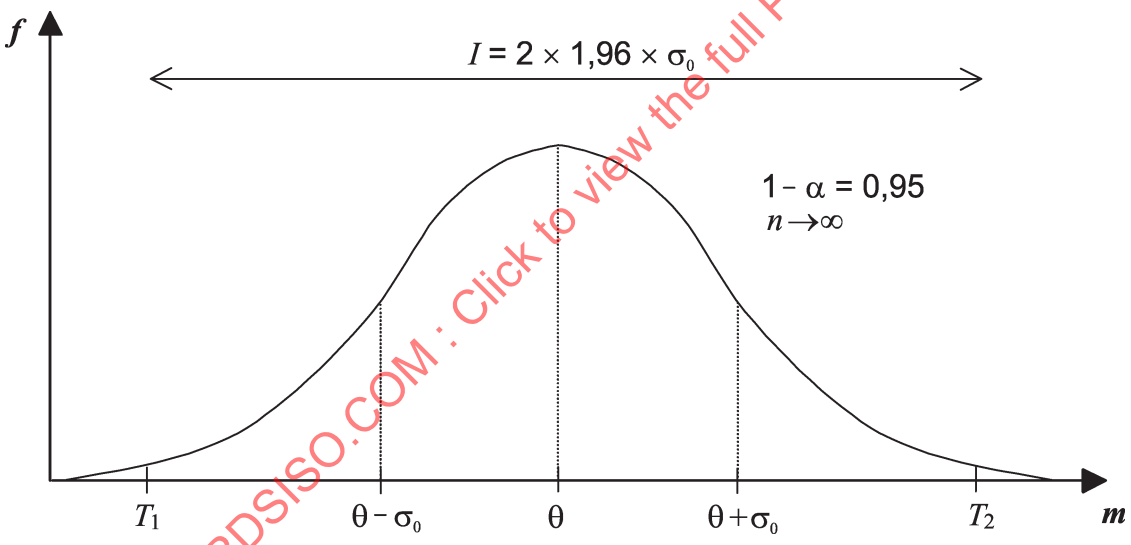

Figure 1 — Illustration of the 95 % confidence interval of a normal distribution

Note 2 to entry: In this part of ISO 14385, the standard deviation, σ_0, is estimated by parallel measurements with an SRM. It is assumed that the requirement for σ_0, presented in terms of an allowable uncertainty budget, i.e. variability is provided by the regulators. In the procedures of this part of ISO 14385, the premise is that the required variability is given as σ_0 itself, or as a quarter of the length of the full 95 % confidence interval.

[SOURCE: ISO 3534-1:2006, 1.28, modified: Figure 1 has been added. Notes 1 and 2 are different.]

3.6
drift
monotonic change of the calibration function over stated maintenance interval, which results in a change of the measured value

3.7
extractive AMS
AMS having the detection unit physically separated from the gas stream by means of a sampling system

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3.8
in-situ AMS
AMS having the detection unit in the gas stream, or in a part of it

3.9
instrument reading
indication of the measured value directly provided by the AMS without using the calibration function

3.10
legislation
directives, acts, ordinances, and regulations

3.11
low-level cluster
cluster of measurement values less than the maximum permissible uncertainty and between 0 % and 15 % of the lowest measuring range

3.12
measurand
particular quantity subject to measurement[5]

3.13
measured component
constituent of the waste gas for which a defined measurand is to be determined by measurement

3.14
measured value
estimated value of the measurand derived from an output signal

Note 1 to entry: This usually involves calculations related to the calibration process and conversion to required quantities

Note 2 to entry: A measured value is a short-term average. The averaging time can be, e.g. 10 min, 30 min, or 1 h.

3.15
period of unattended operation
maximum admissible interval of time for which the performance characteristics will remain within a predefined range without external servicing, e.g. refill, calibration, adjustment

Note 1 to entry: This is also known as the maintenance interval.

3.16
peripheral parameter
specified physical or chemical quantity which is needed for conversion of the AMS measured value to standard conditions

3.17
peripheral AMS
AMS used to gather the data required to convert the AMS measured value to standard conditions

Note 1 to entry: A peripheral AMS is used to measure water vapour, temperature, pressure, and oxygen.

3.18
peripheral SRM
SRM used to gather the data required to convert the SRM measured values to AMS or standard conditions

Note 1 to entry: A peripheral SRM is used to measure water vapour, temperature, pressure, and oxygen.

3.19
precision
closeness of agreement of results obtained from the AMS for successive zero readings and successive span readings at defined time intervals

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3.20
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 used are calibration gases, gas cells, gratings, or filters.

3.21
response time
t90
time interval between the instance of a sudden change in the value of the input quantity to an AMS and the time as from which the value of the output quantity is reliably maintained above 90 % of the correct value of the input quantity

Note 1 to entry: The response time is also referred to as the 90 % time.

3.22
span reading
instrument reading of the AMS for a simulation of the input parameter at a fixed elevated concentration. This simulation should test as much as possible all the measuring elements of the system, which contribute significantly to its performance.

Note 1 to entry: The span reading is approximately 80 % of the measured range.

3.23
standard conditions
conditions as given in legislation to which measured values have to be standardized

3.24
standard deviation
positive square root of the mean squared deviation from the arithmetic mean, divided by the degrees of freedom

Note 1 to entry: The number of degrees of freedom is the number of measurements minus 1.

3.25
Standard Reference Method
SRM
method described and standardised to define a measurand, temporarily conducted on site for verification purposes

Note 1 to entry: Also known as a reference method.

3.26
uncertainty
parameter associated with the result of a measurement that characterises the dispersion of the values that could reasonably be attributed to the measurand[5]

3.27
variability
standard deviation of the differences of parallel measurements between the SRM and AMS

3.28
zero reading
instrument reading of the AMS on simulation of the input parameter at zero concentration, which shall test as much as possible all the measuring elements of the AMS, that contribute significantly to its performance

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4 Symbols and abbreviations

4.1 Symbols

a	intercept of the calibration function
$\hat{a}$	best estimate of a
b	slope of the calibration function
$\hat{b}$	best estimate of b
D_i	difference between SRM value y_i and calibrated AMS measured value $\hat{y}_i$
$\overline{D}$	average of D_i
E	maximum value of measuring range
k_v	test value for variability (based on a χ^2-test, with a β-value of 50 %, for N numbers of paired measurements)
N	number of paired samples in parallel measurements
σ	standard deviation of the differences D_i in parallel measurements
$t_{0,95;\ N-1}$	value of the t distribution for a significance level of 95 % and a number of degrees of freedom of $N - 1$
u_{inst}	uncertainty due to instability (expressed as a standard deviation)
u_{temp}	uncertainty due to influence of temperature (expressed as a standard deviation)
u_{pres}	uncertainty due to influence of pressure (expressed as a standard deviation)
u_{volt}	uncertainty due to influence of voltage (expressed as a standard deviation)
u_{others}	any other uncertainty that can influence the zero and span reading (expressed as a standard deviation)
x_i	i^{th} measured signal obtained with the AMS at AMS measuring conditions
$\overline{x}$	average of AMS measured signals x_i
y_i	i^{th} measured value obtained with the SRM
$\overline{y}$	average of the SRM measured values y_i
$y_{i,s}$	SRM measured value y_i at standard conditions
$y_{s,min}$	lowest SRM measured value at standard conditions
$y_{s,max}$	highest SRM measured value at standard conditions
$\hat{y}_i$	best estimate for the "true value", calculated from the AMS measured signal x_i by means of the calibration function
$\hat{y}_{i,s}$	best estimate for the "true value", calculated from the AMS measured signal x_i at standard conditions
$\hat{y}_{s,max}$	best estimate for the "true value", calculated from the maximum value of the AMS measured signals x_i at standard conditions
Z	offset (the difference between the AMS zero reading and the zero)

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s_{AMS} standard deviation of the AMS used in ongoing quality control

α significance level

ε_i deviation between y_i and the expected value σ_0 standard deviation associated with the uncertainty derived from requirements of legislation

4.2 Abbreviations

AMS automated measuring system

AST annual surveillance test

QA quality assurance

SRM Standard Reference Method

5 Principle

5.1 General

An AMS to be used shall be proven suitable for its measuring task (parameter and composition of the flue gas) by use of the procedures specified in ISO 14956. Using this part of ISO 14385, it shall be proven that the total uncertainty of the results obtained from the AMS meets the specification for uncertainty stated in legislation or in requirements and specifications established in an international trading program. In ISO 14956, the total uncertainty required by the relevant regulations is calculated by summing all the relevant uncertainty components arising from the individual performance.

This part of ISO 14385 provides a procedure for the validation and calibration of an AMS. It consists of the determination of the calibration function and its variability, and a test of the variability of the measured values of the AMS compared with the uncertainty given by legislation or in requirements and specifications established in international trading programs. The tests are based on a number of parallel measurements performed with a Standard Reference Method (SRM). The variability of the measured values obtained with the AMS can then be evaluated against the maximum permissible uncertainty.

The tests are performed on AMS that have been correctly installed and commissioned.

The tests can be used to

a) establish a calibration function over a range of plant operating conditions and

b) calibrate the AMS and demonstrate that an AMS meets the required accuracy at a constant operating load.

The procedure is repeated periodically after a major change of plant operation, after a failure of the AMS, or as required by legislation.

5.2 Limitations

Figure 2 illustrates the components of the AMS covered by this part of ISO 14385.

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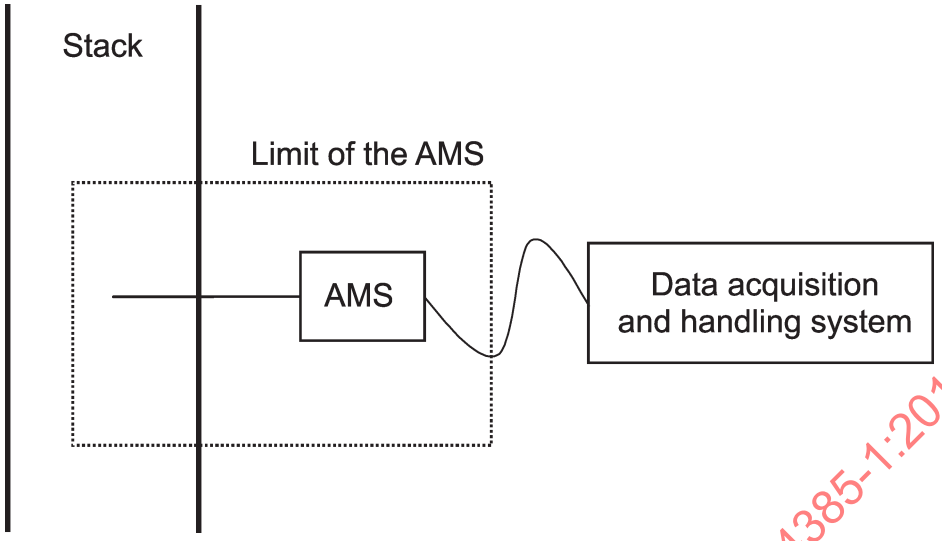

Figure 2 — Limits for the QA of the AMS excluding the data acquisition and handling system

NOTE 1 The influence of the uncertainty of the measurement results, which arise from the data acquisition recording and handling system of the AMS or of the plant system and its determination, are excluded from this part of ISO 14385.

NOTE 2 The performance of the data collection and recording system can be as influential as the AMS performance in determining the quality of the results obtained from the whole measuring system/process. There are different requirements for data collection recording and presentation in different countries.

When conducting parallel measurements, the measured signals from the AMS are taken directly from the AMS (e.g. expressed as analogue or digital signal) during the calibration and annual surveillance test (AST) procedures specified in this part of ISO 14385 by using an independent data collection system provided by the organization(s) carrying out the calibration and AST tests, as specified in ISO 14385-2. All data shall be recorded in their uncorrected form (without corrections for, e.g. temperature and oxygen). A plant data collection system with quality control can additionally be used to collect the measured signal from the AMS.

5.3 Measurement site and installation

The AMS shall be installed in accordance with the requirements of the relevant national or international standards, as specified by legislation, competent authorities, or in emission trade scheme. Special attention shall be given to ensure that the AMS is readily accessible for regular maintenance and other necessary activities.

NOTE The AMS is intended to be positioned as far as practical so that it measures a sample representative of the stack gas composition.

All measurements shall be carried out on a suitable AMS and peripheral AMS installed within an appropriate working environment.

The working platform used to access the AMS shall readily allow parallel measurements to be performed using an SRM. The sampling ports for measurements with the SRM shall be placed as close as possible, but not more than three times the equivalent diameter up- or down-stream of the location of the AMS, in order to achieve comparable measurements between AMS and SRM.

It is necessary to have good access to the AMS to enable inspections to take place and also to minimize time taken to implement the quality assurance procedures of this part of ISO 14385. A clean, well-ventilated, and well-lit working space around the AMS is required to enable the staff to perform this work effectively. Suitable protection is required for the personnel and the equipment, if the working platform is exposed to the weather.

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5.4 Testing laboratories performing SRM measurements

The testing laboratories, which perform the measurements with the SRM, shall be accredited for this task according to ISO/IEC 17025 or shall be approved directly by the relevant competent authority.

6 Calibration and validation of the AMS

6.1 General

Testing shall cover the following items:

— installation of the AMS;

— functional test of the AMS;

— calibration of the AMS by means of parallel measurements with an SRM and, if necessary, in combination with calibration gases;

— validation of the AMS (determination of the variability of the AMS and the check of compliance with the maximum permissible uncertainty or determination of the relative uncertainty).

A calibration procedure shall be performed for all measurands at least every 5 years for every AMS and more frequently if so required by legislation, requirements, and specifications established in an international trading program or the competent authority.

Furthermore, a calibration procedure shall be performed for all the measurands influenced by

— any major change in plant operation (e.g. change in flue gas abatement system or change of fuel) or

— any major changes or repairs to the AMS, which will influence the results obtained significantly.

The results of the calibration procedure shall be reported within 6 months after the changes. During the period before a new calibration function has been established, the previous calibration function (where necessary with extrapolation) shall be used.

The measurement range shall be chosen to ensure the expected measurement values are between 25 % and 75 % of the maximum of this range.

6.2 Functional test

The requirements for installation and the measurement site as specified in 5.3 shall be checked.

If peripheral AMS is used to convert the measured values to other conditions, these AMS shall be subject of functional tests.

NOTE Since the AMS and SRM measured values are converted to other conditions by independently determined data sets of the peripheral parameters, the uncertainties in the peripheral parameters are attributed to the AMS of the air pollutant in the variability test.

The functional test before calibration shall be performed according to Annex A. The period between the functional test and the calibration shall be limited to 1 month.

The specific precautions to be taken should depend on the individual location.

6.3 Calibration and validation of multiple/complex measurement systems

Although the procedures in this part of ISO 14385 are primarily describing the calibration and validation of single instruments, the same procedures can be used for the calibration and validation of multiple/complex measurement systems. For instance, in many countries, emission limit values are expressed in concentrations at standard conditions (dry flue gas with temperature 273,15 K, pressure

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1013 hPa, and a specified oxygen concentration). In such a case, the measurement system consists of several analysers and measuring devices (peripheral AMS) (analyser for air-polluting compound, oxygen analyser, measuring devices for temperature, pressure, and water vapour).

If legislation or requirements and specifications established in an international trading program are requiring calibration and validation of concentrations of air-polluting compounds at standard conditions, two options are possible.

First, this can be realized by calibration of the results of the individual analysers and measuring devices using the measurement results of the appropriate reference methods for each of the components in the calculation (air-polluting compound, oxygen, temperature, pressure, and water vapour). The calibrated results are then used for conversion by calculation to concentrations at standard conditions.

Alternatively, the results of the individual analysers and measuring devices are converted to standard conditions and then calibrated to the converted results of the reference methods.

The standard deviation used in the validation procedure has to be calculated by using Formula (11) and on the basis of the normalized calibrated AMS values and the normalized SRM values.

6.4 Parallel measurements with an SRM

Parallel measurements shall be performed with the AMS and SRM in order to calibrate and/or validate the AMS by use of an independent method.

It is not sufficient to use reference materials alone to obtain the calibration functions and this is therefore not permitted. This is because these reference materials do not replicate sufficiently the matrix stack gas, they cannot be used to establish that the sampling point(s) of the AMS are representative, and they are not used with the sampling system in all cases. However, if there are limited variations in the results obtained in the AMS/SRM tests, and the measured concentrations are more than 20 % below the maximum value of the normal measuring range, an extrapolation of the calibration function to the highest annual value can be verified by the use of appropriate reference materials, taking into account the effects of interfering substances on the AMS, where appropriate.

If clear and distinct operating modes of the plant process are part of its normal operation (for example, changes of fuel), additional calibrations shall be performed and a calibration function established for each operational mode if the operation affects the calibration curve.

NOTE 1 It is recommended that a preliminary test be carried out in order to evaluate if a full calibration over the whole concentration range can be performed. Otherwise, a competent authority is intended to judge if, based on its experience, it is reasonable to establish one calibration function that covers all normal changes in the process.

In order to ensure that the calibration function is valid for the range of conditions within which the plant will operate, the concentrations during the calibration shall be varied as much as possible within the normal operations of the plant. This shall ensure that the calibration of the AMS is valid over as large a range as possible, and also that it covers most operational situations.

The test for variability shall be performed (see 6.5.6) for each calibration function, i.e. for each operating mode of the plant.

An SRM shall be used to measure the emissions through representative sampling in the duct, which is as close as possible to the AMS. The sampling of the AMS and SRM shall not influence the results of both measurement systems.

The presence of the equipment specified in the SRM shall not influence or disturb the AMS measurements.

For each calibration, a minimum of 15 valid parallel measurements shall be made with the plant operating normally. These measurements shall be uniformly spread both over at least 3 d and over each

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of the measuring days of normally 8 h to 10 h (e.g. not five measurements in the morning and none in the afternoon) and be performed within a period of 4 weeks.

NOTE 2 The required spread of a minimum of 15 valid measurements over 3 d is essential in minimising the effect of influences of the subsequent measurement results (i.e. to avoid auto-correlation between the calculated differences in the results of the AMS and SRM). The alternative of performing more measurements within a shorter time interval can lead to the establishment of an invalid calibration function.

NOTE 3 A minimum of 15 valid measurements can, in practice, require that more than 15 samples be taken, since some samples can be deemed to be invalid during subsequent analysis because of inadequate quality.

NOTE 4 The requirement that the measurements need to be uniformly spread over at least 3 d does not imply that the measurements need to be performed within three consecutive days.

If the calibration is not the first calibration being carried out on the AMS and the operator can prove that at least 95 % of the AMS measured values obtained since the last calibration or annual surveillance test (see ISO 14385-2) are less than the maximum permissible expanded uncertainty, the number of measurements can be reduced to five parallel measurements performed on 1 d. The results of these five measurements shall be used to check the validity of the existing calibration function. If the calibration function appears not to be valid, the number of parallel measurements shall be increased to 15 parallel measurements to calculate a new calibration function.

Examples of expanded uncertainty are given in Annex F.

A set of measurements is valid when all of the requirements below are fulfilled:

— the SRM measurements are performed according to the accepted standard;

— the time period of each AMS measured signal shall cover at least 90 % of the averaging time [excluding all of the measured signals which are above 100 % or below 0 % of the measuring range of the AMS, signals obtained during internal checks (auto calibration), and signals obtained during any other malfunctioning of the AMS].

During the parallel measurements with the AMS and SRM, each result is considered as a measurement pair (one AMS measured signal and one SRM measured value) and these shall cover the same time period.

The sampling time for each of the parallel measurements shall be at least 30 min, or at least four times the response time of the AMS, including the sampling system (as determined during the response time measurements carried out during the procedures according to ISO 14956), whichever is the greater. In general, the sampling time should equal the shortest averaging time, which is required by the legislation or as defined in an international trading program. The recording system shall have an averaging time significantly shorter than the response time of the AMS.

If the sampling time is shorter than 1 h, then the time interval between the start of each sample shall be longer than 1 h.

The results obtained from the SRM shall be expressed under the same conditions as measured by the AMS (e.g. conditions of pressure, temperature, etc.). In order to establish the calibration function and perform the variability test, all additional parameters and values included in the corrections to AMS conditions and standard conditions shall be obtained for each measurement pair.

EXAMPLE If the AMS measures N_2O in units of mg/m^3 in stack gas containing water vapour, then the SRM results are expressed in the same units (e.g. mg/m^3 in the stack gas with the same water vapour concentration).

The 15 parallel measurements can be performed in less than 3 d if

— at least 97 % of the validated half-hourly values obtained in the period since the last calibration were smaller than 30 % of the measurement range value specified for half-hourly values or

— at least 99 % of the validated half-hourly values obtained in the period since the last calibration do not deviate from the average of all validated half-hourly values by more than 5 %.

In these cases, the interval between the start of each sampling can be less than 1 h.

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In order to fulfil the requirement that the calibration of the AMS is valid over as large a range as possible, and that it covers most operational situations, parallel measurements over 3 d are generally required. However, this can require several manual SRM measurements of the water vapour concentration. If calibrated AMS measured values for water vapour are available, these can be used to convert the SRM data to dry or wet basis. When wet abatement techniques are used, the water vapour concentration is often nearly constant and extended measurement of the water vapour concentration is of little purpose. In those situations, conversion of SRM data to dry or wet basis as required can be carried out using calculated AMS water vapour measurements.

6.5 Procedure: calibration and validation of the AMS by means of parallel measurements

6.5.1 General

In this procedure, the calibration function of the AMS and its variability are determined by means of parallel measurements with an SRM. The variability of the measured values obtained with the AMS is then evaluated against the maximum permissible uncertainty.

The sequence of the tests to be carried out is shown in Figure 3.

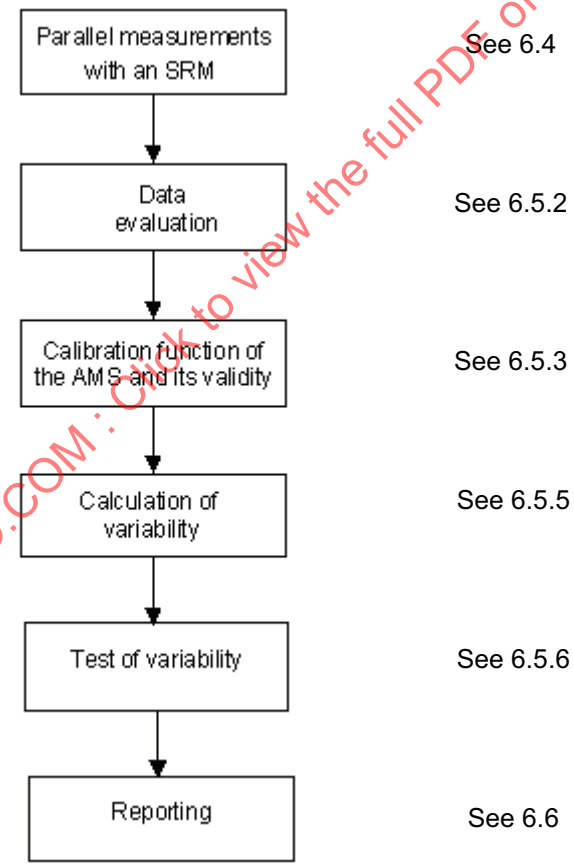

Figure 3 — Flow diagram for the calibration and variability tests

Examples of calculation of the calibration function and of the variability test are given in Annex D.

NOTE If change in fuel mixture is a part of the normal operation mode of the plant, it is recommended that the fuel mixture is varied during the parallel measurements.

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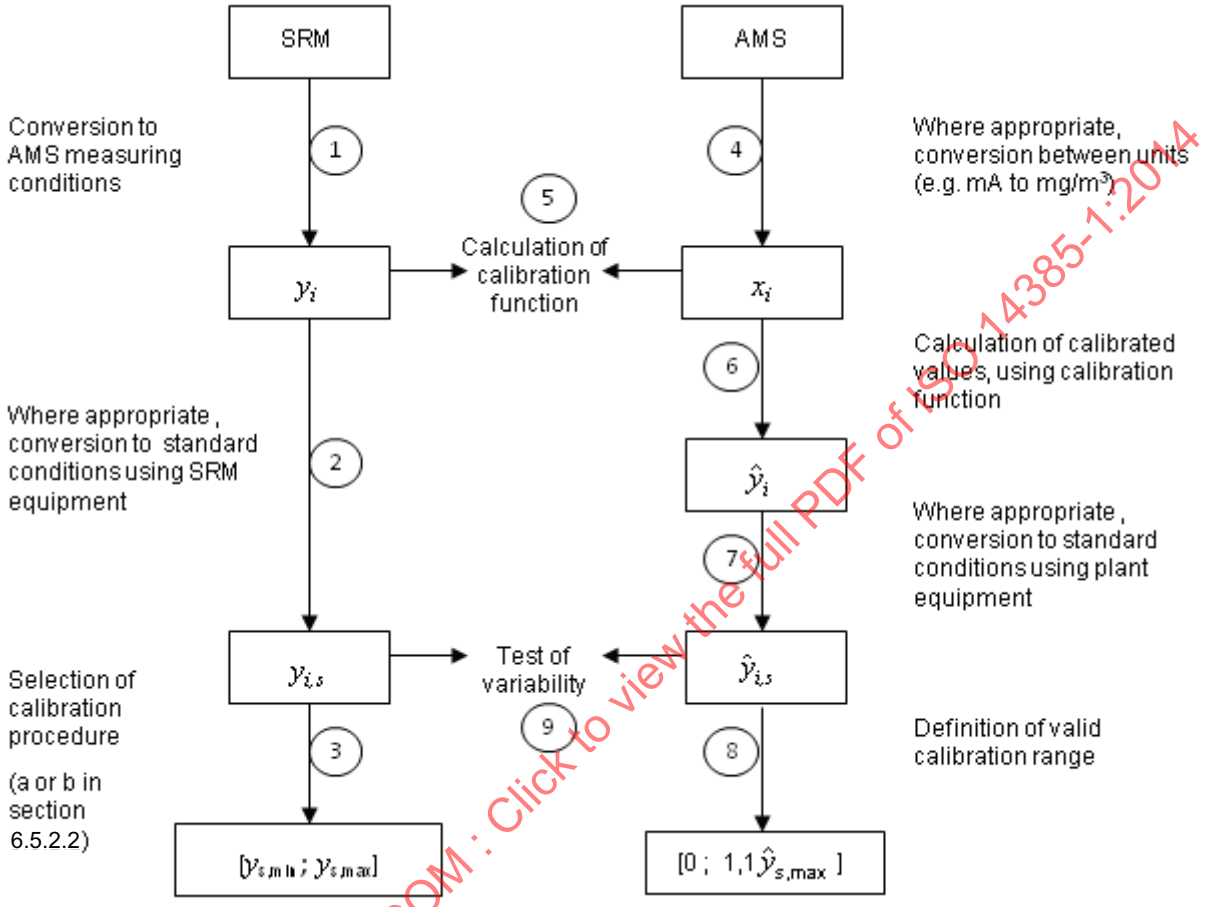
6.5.2 Data evaluation

6.5.2.1 Preparation of data

The steps for providing data required for establishing the calibration function and performing the test of variability are illustrated in Figure 4.



NOTE The figure in the circles indicates the sequence of the steps.

Figure 4 — Flow chart describing the steps in calibration procedure and test for variability

The AMS shall be calibrated at the condition of the exhaust gas as measured by the AMS. Therefore, the SRM values shall be converted to AMS measuring conditions, if necessary, giving SRM measured values, y_m, to be expressed in concentration units (e.g. mg/m^3).

The measured signals from the AMS, x_i can be either a signal in an electrical unit (e.g. mA or Volt) or in a concentration unit (e.g. mg/m^3).

NOTE For a non-extractive AMS that measures the gas directly, the calibration function reported shall be at the operating conditions. For an extractive AMS measuring at specified conditions, the calibration function is reported at these specified conditions.

The data sets obtained in the parallel measurements shall be checked for possible outliers (see Annex E). The method used to assess outliers and reasons for excluding outliers shall be given in the calibration report. Outliers shall be reported and identified in the calibration diagrams. This part of ISO 14385 requires at least 15 valid data points for a calibration function. If points are excluded, e.g. through the use of outlier tests, this requirement can be failed. It is therefore recommended that additional data points be taken, to allow for the exclusion of outliers. If this is not done, the calibration can be invalid.

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6.5.2.2 Establishing the calibration function

It is presupposed in this part of ISO 14385 that the calibration function is linear and has a constant residual standard deviation. The calibration function shall be described by Formula (1) [ISO 11095]:

$$y_i = a + b x_i + \varepsilon_i \quad (1)$$

where

x_i is the i^{th} result of the AMS; i = 1 to N; $N \geq 15$;

y_i is the i^{th} result of the SRM; i = 1 to N; $N \geq 15$;

ε_i is the deviation between y_i and the expected value;

a is the intercept of the calibration function;

b is the slope of the calibration function.

The general procedure [Formula (4) and (5)] requires a sufficient range of the measured concentrations to give a valid calibration of the AMS for the complete range of concentrations encountered during normal operation. As stated in 6.5, it is essential that the concentration range is as large as possible within the normal operation of the plant for a valid calibration function. However, at a large number of plants, it can be difficult under normal operating conditions to achieve a sufficiently large concentration range. In such cases, in which the concentration range (measured with the SRM) is less than the maximum permissible uncertainty, another (similar) procedure is given below (procedure b).

NOTE 1 If the concentration range is slightly bigger than maximum permissible uncertainty, and if Formulae (4) and (5) result in an inadequate calibration function (e.g. a function with negative slope), Formulae (6) and (7) can be used instead.

Formulae (2) and (3) shall be calculated:

$$\bar{x} = \frac{1}{N}\sum_{i=1}^{N} x_i \quad (2)$$

$$\bar{y} = \frac{1}{N}\sum_{i=1}^{N} y_i \quad (3)$$

The difference $(y_{s,max} - y_{s,min})$ between the highest and lowest measured SRM concentration at standard conditions shall be calculated.

a) If $(y_{s,max} - y_{s,min})$ is greater than or equal to the maximum permissible uncertainty, calculate:

$$\hat{b} = \frac{\sum_{i=1}^{N}(x_i - \bar{x})\left(y_i - \bar{y}\right)}{\sum_{i=1}^{N}(x_i - \bar{x})^2} \quad (4)$$

$$\hat{a} = \bar{y} - \hat{b}\bar{x} \quad (5)$$

b) If $(y_{s,max} - y_{s,min})$ is smaller than the maximum permissible uncertainty, calculate:

$$\hat{b} = \frac{\bar{y}}{\bar{x} - Z} \quad (6)$$

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$$\hat{a} = -\hat{b} \times Z \quad (7)$$

where the offset (Z) is the difference between the AMS zero reading and the zero.

NOTE 2 For several AMS, the offset is 4 mA.

For calculation b), it is essential that, prior to the parallel measurements, it is proven that the AMS gives a reading at, or below, detection limit (as demonstrated in the procedures according to ISO 14956) at a zero concentration. Before calibration is performed, it shall be proven that the AMS is commissioned satisfactorily, e.g. as specified by the AMS supplier and/or manufacturer. It shall also be shown and documented that the AMS gives a zero reading on a zero concentration (as stated in 6.2).

If the spread of the data is less than the maximum permissible uncertainty, a calibration function calculated as a linear regression function forced through the lower reference point (which is the zero point if the AMS reads zero) can be used, provided the functional test has proven that it is linear down to the lower reference point or zero.

The results shall be plotted on an x-y graph in order to show explicitly the calibration function and the valid calibration range.

6.5.2.3 Low-level clusters

There are typically three types of patterns of emissions from industrial plants; in addition to the patterns of data described in 6.5.2.2 and assessed using procedure a or procedure b, the emissions can be very low, clustered at, or near to zero. Low-level clusters are often the result of highly controlled processes, and are common for N_2O and CH_4 emissions from most combustion plants.

If $y_{s,max} - y_{s,min}$ is smaller than the maximum permissible uncertainty and $y_{s,min}$ is smaller than the maximum permissible uncertainty (low-level clusters), then the uncertainties of both the SRM and AMS can undermine the accuracy of the calibration function. Therefore, if the low-level condition is met, then it is advisable to contact the competent authority for guidance on an alternative procedure.

Alternative procedures include the following three options:

— Option 1: Performing a calibration as specified in Clause 6, applying procedure b, and accepting that the uncertainty of the measurements can introduce a significant calibration error.

— Option 2: Performing a limited number of measurements using the SRM, perhaps over 1 d instead of at least 3 d. The purpose of the SRM measurements is to ensure that the emissions are as low as the AMS shows. The AMS is then calibrated using surrogates, such as reference materials with a low uncertainty. This approach can have a high uncertainty, but again, this error will not be significant if the emissions remain well below the maximum value of the lowest measuring range.

— Option 3: As with option 2, but the SRM data are combined with the data produced from using reference materials, in order to derive a calibration function.

6.5.3 Calibration function of the AMS and its validity

The calibration function is given by Formula (8):

$$\hat{y}_i = \hat{a} + \hat{b}\, x_i \quad (8)$$

where

$\hat{y}_i$ is the calibrated value of the AMS;

$\hat{x}$ is the AMS measured signal

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Each measured signal $\hat{x}$ of the AMS shall be converted to a calibrated value $\hat{y}$ by means of the above calibration function.

NOTE 1 It is recommended that this calibration function is incorporated into the data processing system of the plant.

NOTE 2 $\hat{y}_i$ is the calibrated measurement result obtained from the AMS. In some legislation, the uncertainty value of a measurement is subtracted from that measurement before comparison with a limit value. That procedure is outside the scope of this part of ISO 14385. The calibrated result $\hat{y}_i$ of the AMS is without subtraction of the uncertainty.

The calibration function is valid when the plant is operated within the valid calibration range. This valid calibration range is defined as the calibration range from zero to $\hat{y}_{s,max}$, determined during the calibration procedure, plus an extension of 20 % of the calibration range beyond the highest value. This implies that only values in the valid calibration range are valid measured values.

For measurements outside the valid calibration range, however, the calibration curve shall be extrapolated in order to determine the concentration values, which exceed the valid calibration range.

If greater confidence in the performance of the AMS at the upper limit of the measuring range is required when the plant is emitting outside its calibration range determined above, reference materials at zero and at a concentration close to the upper limit of the measuring range shall be used, where available, as part of the calibration procedure to confirm the suitability of the linear extrapolation. In this case, calculate the deviation between the calibrated measured value of the AMS at zero and the upper limit of the measuring range and the corresponding SRM values. The deviation at the upper limit of the measuring range should be less than the uncertainty specified by legislation. The deviation at zero should be less than 10 % of the upper limit of the measuring range. If these criteria are not fulfilled then further investigations shall be performed to establish the reasons for this.

If the best estimate of the true value, $\hat{y}_{i,s}$, is outside the valid calibration range but below 50 % of the upper limit of the measuring range, then the competent authority can allow the plant to perform an AST instead of the calibration procedure according to this part of ISO 14385. If the AST demonstrates that the existing calibration function is valid beyond the calibration range, the competent authority can allow the plant to extend the calibration range up to the measured concentrations (but below 50 % of the upper limit of the measuring range) determined during the AST.

The existing calibration function shall be used until the new calibration function has been implemented.

Only calibrated values should be used when reporting to the authorities.

6.5.4 Extrapolating the calibration function using reference materials

As already stated in 6.5.3, the calibration function can be extrapolated with 10 % of the calibration range beyond the highest SRM value. Further extrapolation with the use of reference material to the highest annual measurement value is allowed if the following requirements are fulfilled:

— the difference between the value(s) of the reference material and the calibrated value has to be less than 75 % of the allowed uncertainty, expressed as the 95 % confidence interval;

— the difference between the zero value and the calibrated zero value has to be less than 10 % of the highest annual measurement value.

The results of a linearity check can be used to carry out the extrapolation. Interfering compounds have to be taken in consideration when extrapolating.

Figure 5 gives an example of the extrapolation.

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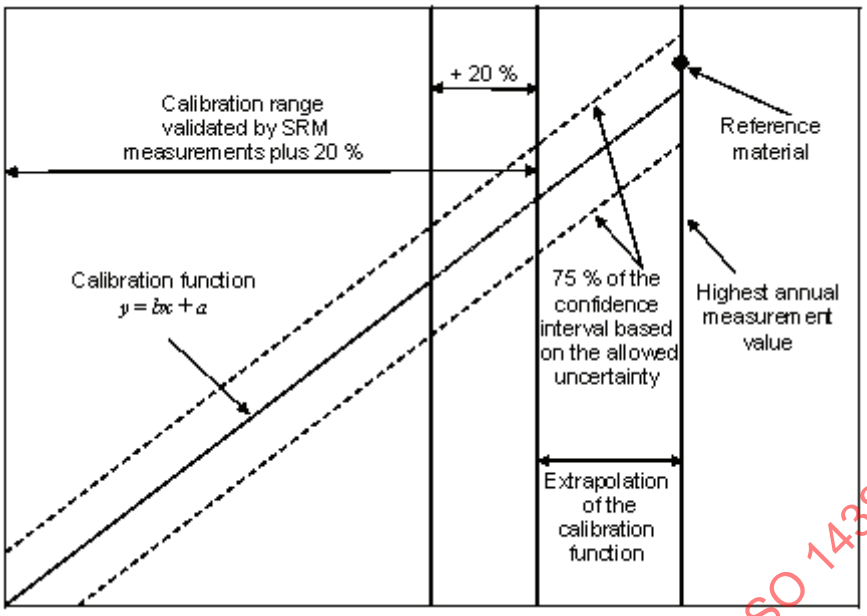

Figure 5 — Example of extrapolation of the calibration curve

6.5.5 Calculation of variability

Identify the stated or required maximum uncertainty for the measured values from the AMS. Verify the exact definition of this uncertainty (e.g. if it is expressed as a 95 % confidence interval, a standard deviation, or any other statistical formulation). If necessary, convert the required maximum uncertainty in terms of an absolute standard deviation σ_0.

In the case where the uncertainty is expressed at a level of confidence of 95 %, the value of an absolute standard deviation shall be determined by using a factor of 1,96 as the value for the coverage factor.

The variability test shall be performed on the measured values (calibrated values) of the AMS. Hence, for every parallel measurement, the AMS measured value $\hat{y}_i$ shall be calculated using the calibration function (see 6.5.3).

Where the requirements on the data quality are specified under standard conditions, the variability test shall be performed using concentrations under these conditions.

When calculating the variability, the peripheral parameters (e.g. water vapour content, temperature, and oxygen concentration) used to standardize the measurements shall be taken from the following:

a) the SRM instrumentation for normalizing the SRM results;

b) the plant instrumentation for normalizing the AMS results, or in case these do not exist, the default values used by the plant.

NOTE The purpose of this procedure is to ensure that the standardization procedure carried out in the plant's data recording and processing system is included in the variability test.

Examples of equations for the conversion of values are given in Annex D.

If the AMS fails the variability test due to error arising from measurements in the peripheral parameters obtained from the plant instrumentation, it is permissible to repeat the variability test using the parameters obtained by the test laboratory from their peripheral SRM. This is only permitted if measures are taken to correct the faulty plant instrumentation.

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For each data set (minimum 15 pairs) for a given calibration function, the following parameters shall be calculated, where $y_{i,s}$ is the SRM value at standard conditions and $\hat{y}_{i,s}$ is the calibrated AMS value (the best estimate for the "true value"), calculated from the AMS measured signal x_i at standard conditions:

$$D_i = y_{i,s} - \hat{y}_{i,s} \tag{9}$$

$$\bar{D} = \frac{1}{N}\sum_{i=1}^{N} D_i \tag{10}$$

$$\sigma = \sqrt{\frac{1}{N}\sum_{i=1}^{N}(D_i)^2} \tag{11}$$

6.5.6 Test of variability

The AMS passes the variability test when

$$\sigma \leq \sigma_0 \, k_v \tag{12}$$

Values for k_v which shall be applied for different number of parallel measurements are given in Table 1.

Table 1 — k_v values

Number of parallel measurements	k_v
3	0,832 6
4	0,888 1
5	0,916 1
6	0,932 9
7	0,944 1
8	0,952 1
9	0,958 1
10	0,962 9
11	0,966 5
12	0,969 5
13	0,972 1
14	0,974 2
15	0,976 1
15	0,976 1
16	0,977 7
17	0,979 1
18	0,980 3
19	0,981 4
20	0,982 4
25	0,986 1
30	0,988 5

NOTE 1 The k_v values are the test values from a χ^2 -test, with a β-value of 50 %.

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NOTE 2 The variability obtained includes uncertainty components associated with the repeatability's of both the AMS and the SRM, but not the overall uncertainty of the SRM (therefore, an imprecise implementation of the SRM can result in an apparent poorer variability of the AMS and could result in its false rejection during the variability test). The procedure for determination of variability is not in accordance with the GUM[6].

NOTE 3 This method implies that the quality of the application of the SRM will influence the result of the test. However, it is the result which determines a pass or failure and that, in some cases, a better application of the SRM could change the result from fail to pass.

6.6 Report

The report shall contain at least the following information:

a) a description of the plant and its sampling location(s);

b) a description of the operating condition of the plant, and the fuel(s) used at the plant during the tests;

c) the names of the testing laboratory and of the personnel conducting the tests;

d) details of the ISO/IEC 17025 accreditation of the testing laboratory;

e) a description of the AMS used, including the measurands covered, its principle, type, operating range, and its location;

f) a description of the SRM used, its principle, type, operating range, repeatability and/or measurement uncertainty, and its ISO reference number where appropriate;

g) dates and times of the parallel measurements;

h) detailed data of all the measured values obtained from the AMS and the SRM, averaged over relevant periods;

i) method used to assess outliers and reasons for excluding outliers;

j) the calibration function and the valid calibration range including all data used for calculating the calibration function and performing the variability test;

k) the x-y plot of parallel measurements expressed in mg/m^3, including the valid calibration range;

l) any deviation from the procedures described in this part of ISO 14385 (i.e. ISO 14385-1) and their possible influence on the results obtained presented;

m) the results of the last functional test (see Annex A).

7 Documentation

Every occurrence affecting the AMS during its life span shall be documented. The AMS shall be assigned a registration number and a file specific to the AMS containing all the relevant information shall be drawn up and updated by the person in charge of the AMS.

The AMS documentation shall include all relevant diagrams and can include photographs of the sampling system and AMS when they were installed and commissioned.

See Annex C.

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Annex A
(normative)

Functional test of AMS

A.1 General

Table A.1 specifies the individual steps of the functional test of AMS to be performed during the calibration procedure and AST for extractive and non-extractive AMS.

Table A.1 — Specification of individual steps of the functional test

Activity	Extractive AMS	Non-extractive AMS
Alignment and cleanliness		X
Sampling system	X	
Documentation and records	X	X
Serviceability	X	X
Leak test	X	
Zero and span check	X	X
Linearity	X	X
Interferences	X	X
Response time	X	X
Report	X	X

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A.2 Alignment and cleanliness

A visual inspection, with reference to the AMS manuals, shall be carried out on the following when applicable:

— internal control of analyser;

— cleanliness of the optical components;

— flushing air supply;

— obstructions in the optical path.

After reassembly at the measurement location, at least the following shall be checked:

— alignment of the measuring instrument;

— contamination control (internal control of optical surfaces);

— flushing air supply.

A.3 Sampling system

A visual inspection of the sampling system shall be performed, noting the condition of the following components, when fitted:

— sampling probe;

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— gas conditioning systems;

— pumps;

— all connections;

— sample lines;

— power supplies;

— filters.

The sampling system shall be in good condition and free of any visible faults which would decrease the quality of data.

A.4 Documentation and records

The following documentation shall be controlled, readily accessible, and up to date:

— a plan of the AMS;

— all manuals (maintenance, users, etc.);

— log books to document possible malfunctions and action taken;

— service reports;

— management system procedures for maintenance, calibration, and training;

— training records;

— maintenance schedules;

— auditing plans and records.

A.5 Serviceability

There shall be provisions for the effective management and maintenance of the AMS in order to ensure the maintenance of the quality of data. Such provisions include at least the following:

— safe and clean working environment with sufficient space and weather protections;

— easy and safe access to the AMS itself;

— adequate supplies of calibration materials, tools, and spare parts.

In order to conduct the tests effectively, in addition to the requirements for testing the AMS, and the requirements for the sampling location and the working platform which are required for performing the calibration procedure and the procedures according to ISO 14385-2, facilities shall be provided to introduce the reference materials, both at the inlet of the sampling line (where present) and at the inlet of the analyser.

A.6 Leak test

Leak testing shall be performed according to the AMS manuals. The test shall cover the entire sampling system.

A.7 Zero and span check

Reference zero and span materials shall be used to verify the corresponding readings of the AMS.

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In case of non-extractive AMS, zero and span checks shall be performed on a waste gas free reference path before and after the readjustment and after re-assembly of the AMS at the measurement location.

NOTE For some monitors, it is difficult to achieve a zero reading. In those situations, the AMS can be removed from the stack and zeroed using a test bench or similar. As an alternative, a measuring path, which enables this zero test to be carried out, can be installed in the stack.

A.8 Linearity

The linearity of the analyser's response shall be checked using five different reference materials, including a zero concentration.

In case of gaseous reference materials, these four reference materials can be obtained from different gas cylinders or can be prepared by means of a calibrated dilution system from one single gas concentration.

The reference material concentrations shall be selected such that the measured values are at approximately 20 %, 40 %, 60 %, and 80 % of the measuring range. It is necessary to know the values of the ratios of their concentrations precisely enough so that an incorrect failure of the linearity test does not occur. The dry test reference material shall be applied to the inlet of the AMS.

The individual analysers are tested using the following concentrations applied in a randomized sequence:

— reference material with zero concentration;

— reference material concentration approximately 20 % of the measuring range;

— reference material concentration approximately 40 % of the measuring range;

— reference material concentration approximately 60 % of the measuring range;

— reference material concentration approximately 80 % of the measuring range;

— reference material with zero concentration.

After each change in concentration, the first instrument reading shall be taken after a time period equal to at least three times the response time of the AMS. At each reference material concentration, at least three readings shall be made. The time period between the start of each of the three readings shall be separated by at least four times the response time.

NOTE 1 This procedure means that the quality of the reference material may influence the result of the tests. However, it is the result that leads to a pass or failure in the test. In some cases, a reference material with a higher quality may change the result from fail to pass.

NOTE 2 Special care is intended to be taken when handling HCl or HF in dry gases. For example, particular surface reactions in tubing can result in very long response time, which is not representative for the response time for humid gases.

NOTE 3 Where no other method is possible, the linearity can also be performed with the aid of reference materials such as grating filters or gas filters.

The linearity shall be calculated and tested using the procedure as given in Annex B. If the AMS does not pass this test, then the problem shall be identified and solved.

A.9 Interferences

A test shall be undertaken if the process gases to be monitored contain components that are known interferences.

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A.10 Response time

The response time of the AMS shall be checked. This can be performed, if appropriate, by injecting the reference material at the end of the sampling probe. The response time shall not exceed the measured value that has been identified during the procedures according to ISO 14956.

A.11 Report

The results of the functional test shall be reported. Any faults shall be recorded. If the faults are judged to have an effect on the quality of data, then the operator shall carry out the necessary corrective and preventive action.

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Annex B
(normative)

Test of linearity

B.1 Description of the test procedure

In this test procedure, a regression line is established between the instrument readings of the AMS (*Y*-values) and the reference material values (*X*-values) during the linearity test performed in A.8. In the next step, the average of AMS readings at each concentration level is calculated. Then the deviation (residual) of this average to the regression line is calculated.

B.2 Establishment of the regression line

A linear regression for the function in Formula (B.1) is established:

$$Y_i = A' + B\left(X_i - X_z\right) \tag{B.1}$$

For the calculation, all the measuring points are taken into account. The total number of measuring points n is equal to the number of concentration levels (of which there are five, including zero) times the number of repetitions (these are the results of at least three readings) at a particular concentration level. In total, n is at least 18 as at zero in total at least six repetitions are made.

The coefficient A' is obtained by Formula (B.2):

$$A' = \frac{1}{n}\sum_{i=1}^{n} Y_i \tag{B.2}$$

where

A' is the average value of the *Y*-values, i.e. the average of the AMS instrument readings;

Y is the individual AMS instrument reading;

n is the number of measuring points (at least 18).

The coefficient B is obtained by Formula (B.3):

$$B = \frac{\sum_{i=1}^{n} Y_i\left(X_i - X_z\right)}{\sum_{i=1}^{n}\left(X_i - X_z\right)^2} \tag{B.3}$$

where

X_z is the average of the *X*-values, i.e. the average of the reference material concentrations;

X_i is the individual value of the reference material concentration.

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Secondly, the function $Y_i = A' + B\,(X_i - X_Z)$ is converted to $Y_i = A + B\,X_i$ through the calculation of A according to Equation (B.4):

$$A = A' - BX_Z \tag{B.4}$$

B.3 Calculation of the residuals of the average concentrations

The residuals of the average concentration at each concentration level to the regression line are calculated as follows:

Calculate at each concentration level the average of the AMS readings at one and the same concentration level c:

$$\bar{Y}_c = \frac{1}{m_c}\sum_{i=1}^{m_c} Y_{c,i} \tag{B.5}$$

where

$\bar{Y}_c$ is the average Y-value (AMS reading) at concentration level c;

$Y_{c,i}$ is the individual Y-value (AMS reading) at concentration level c;

m_c is the number of repetitions at one at the same concentration level c.

Calculate the residual d_c of each average according to Formula (B.6):

$$d_c = \bar{Y}_c - (A + Bc) \tag{B.6}$$

where

c is the concentration level.

Convert d_c in concentration units to a relative unit $d_{c,\mathrm{rel}}$ by dividing d_c by the upper limit c_u of the range used in the linearity test (see A.8) according to Formula (B.7):

$$d_{c,\mathrm{rel}} = \frac{d_c}{c_\mathrm{u}}\,100\,\% \tag{B.7}$$

B.4 Test of the residuals

Test each residual according to

$$d_{c,\mathrm{rel}} < 5\,\% \tag{B.8}$$

All residuals shall pass this test.

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Annex C
(normative)

Documentation

C.1 Principle

Every event significantly affecting the AMS during its life span should be documented. A file specific to the AMS and containing all the relevant information should be drawn up and updated, under the responsibility of the person in charge of the AMS.

C.2 Setup of the AMS file

The file should be set up as soon as the AMS has been received. It contains at least the following elements, for example, in the form of sheets:

— an identification sheet;

— a follow-up sheet;

— a procedure for calibration and verification (it can be the manufacturer's instructions in the national language or a specific internal procedure);

— reports for all verifications, calibrations, and interventions.

The following elements can also be included in the file:

— certificate of delivery;

— manufacturer's instructions for operation and maintenance.

A registration number should be assigned to the AMS, indicated in the identification attached to the analyser itself, in order to identify it more easily.

C.3 Management of the AMS file

Proof of the qualification of the person in charge of the AMS should be given (initial training, professional training, or on-the-job training). The person in charge of the AMS should make sure that the file is kept up to date and that calibration and maintenance operations are carried out when necessary. Record of the maintenance operations should be kept. After the AMS is decommissioned, it can be necessary, according to national legislation, to retain the records for a given period of time in order to ensure the documentation of past results.

C.4 Composition of the AMS file

C.4.1 Identification record

This record should be produced after the delivery of the AMS and should indicate the following:

— the type and designation of the AMS and its identification;

— the name of the manufacturer and, if applicable, the supplier;

— the location;

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— the expiry date of the guarantee;

— the date of delivery and of putting into service, optionally the date and number of the order form;

— references for the operation, calibration, verification, and preventive maintenance procedures.

C.4.2 Follow-up record

The follow-up record should be kept up to date under the responsibility of the person in charge of the AMS. Every event affecting the AMS should be recorded, indicating the date, the nature of the event, the element of the AMS concerned, observations and/or results, the name of the intervening person and his/her identification (signature or initials). Examples of events, which affect the AMS, include installation and commissioning, calibration, verification, preventive maintenance, malfunction, corrective maintenance, modification, and decommissioning.

C.4.3 Verification report

The verification report or form should be completed by the user of the AMS or a designated individual, following each verification, according to the procedure and to a frequency specified beforehand.

C.4.4 Calibration report

The individual responsible for this task should complete the report following each calibration exercise, following routine calibrations according to the relevant procedure and to a frequency specified beforehand, when an applicable non-conformity has been identified, following a verification or an intervention. The calibration procedure ensures that the result is linked to a certified standard.

C.4.5 Intervention report

Any intervention should be documented and filed under the responsibility of the person in charge of the AMS. The intervention can be carried out, for example, by the manufacturer, by the user, or by the maintenance department.

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Annex D
(informative)

Example of calculation of the calibration function

D.1 General

The purpose of this procedure is to perform a calibration. The procedures are carried out as described in Clause 6.

The following example illustrates how to perform the tests for determining the calibration function.

D.2 Example – N_2O analyser

Table D.1 — General

Parameter	N_2O
AMS method	NDIR
SRM method	Automated, ISO 21258
Measurement range	50 mg/m^3

In this example, 18 parallel measurements have been taken over three days, evenly distributed. The results are given in the Table D.2:

Table D.2 — Results of parallel measurements

Sample number	SRM value mg/m^3	AMS value mg/m^3
	y	x
1	27,6	26,8
2	28,8	29,0
3	34,0	33,5
4	31,3	33,0
5	30,6	31,8
6	28,0	28,6
7	39,1	39,1
8	36,3	38,6
9	33,2	34,0
10	40,7	39,7
11	37,5	39,5
12	36,8	37,2
13	19,4	18,5
14	20,1	20,5
15	19,8	19,3
16	21,7	22,1

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