
**Stationary source emissions —
Greenhouse gases —**

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

Émissions de sources fixes — Gaz à effet de serre —

*Partie 2: Contrôle qualité continu des systèmes de mesurage
automatiques*



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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).

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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 WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

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*

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 by legislation, competent authorities, or in an emission trade scheme.

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Stationary source emissions — Greenhouse gases —

Part 2:

Ongoing quality control of automated measuring systems

1 Scope

This part of ISO 14385 specifies 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 the following:

- a procedure to maintain and demonstrate the required quality of the measurement results during the normal operation of an AMS, by checking that the zero and span characteristics are consistent with those determined using the relevant procedure in ISO 14956;
- a procedure for the annual surveillance tests (AST) of the AMS in order to evaluate a) that it functions correctly and its performance remains valid and b) that its calibration function and variability remain as previously determined.

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

This part of ISO 14385 is restricted to quality assurance (QA) of the AMS and does not include QA of the data collection and recording system of the plant.

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 14385-1, *Stationary source emissions — Greenhouse gases — Part 1: Calibration 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 terms and definitions given in ISO 14385-1 and the following apply.

3.1

control chart

graphical presentation of the regular recording of the difference of the reading of an instrument or measuring system, when measuring the pollutant concentration in a gas with known concentration, and the nominal value of the pollutant concentration in that gas

4 Symbols and abbreviations

4.1 Symbols

D_i	difference between SRM value y_i and calibrated AMS measured value $\hat{y}_i$
$\bar{D}$	average of D_i
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
S_{AMS}	standard deviation of the AMS used in ongoing quality control
S_D	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
$\bar{x}$	average of AMS measured signals x_i
y_i	i th measured value obtained with the SRM
$\bar{y}$	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
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

EWMA chart	exponentially weighted moving average chart
QA	quality assurance
SRM	Standard Reference Method

5 Principle

5.1 General

An AMS to be used shall have been proven suitable for its measuring task (parameter and composition of the flue gas) by use of the procedures, as specified by 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.

NOTE It is advisable that uncertainty figures are provided by independent testing bodies.

This part of ISO 14385 provides two procedures.

- A procedure which is used to check drift and precision in order to demonstrate that the AMS is in control during its operation so that it continues to function within the required specifications for uncertainty. This is achieved by conducting periodic zero and span checks of the AMS, based on those used in the procedure for zero and span repeatability tests according to ISO 14956:2002, and then evaluating the results obtained using control charts. Zero and span adjustments or maintenance of the AMS can be necessary, depending on the results of this evaluation.
- A procedure which is used to evaluate whether the measured values obtained from the AMS still meet the maximum permissible uncertainty criteria, as demonstrated in the calibration procedure (ISO 14385-1). It also determines whether the calibration function obtained during the calibration procedure is still valid. The validity of the measured values obtained with the AMS is checked by means of a series of functional tests, as well as by the performance of a limited number of parallel measurements using an appropriate SRM.

5.2 Limitations

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

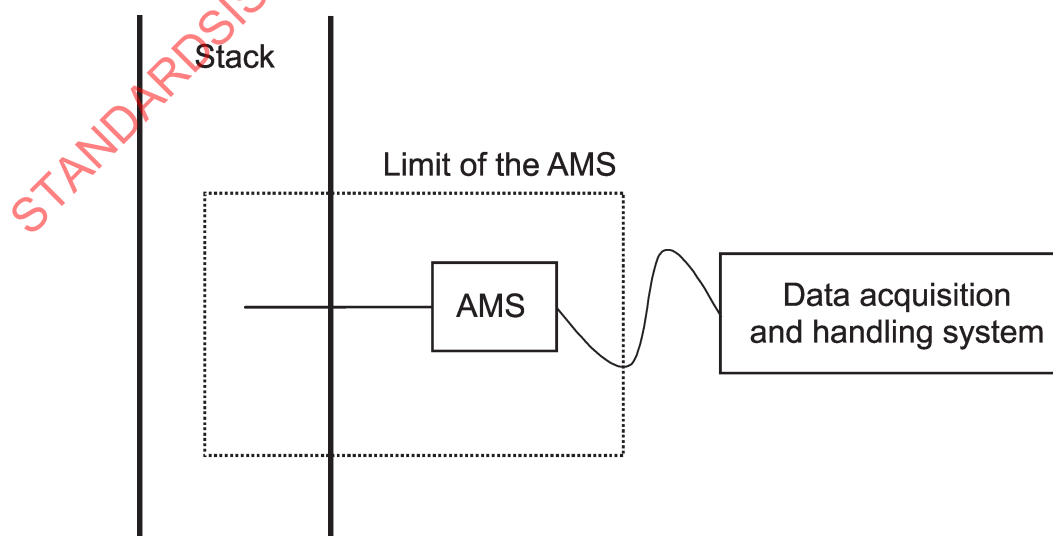


Figure 2 — Limits for the QA of the AMS excluding the data acquisition and handling system

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 AST procedures specified in this part of ISO 14385, by using an independent data collection system provided by the organization(s) carrying out the AST tests. 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 in a position where it measures a sample, which is 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-stream 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.

5.4 Testing laboratories performing SRM measurements

The testing laboratories, which perform the measurements with the SRM, shall have an accredited quality assurance system according to ISO/IEC 17025 or shall be approved directly by the relevant competent authority. They shall also have sufficient experience in performing the measurements using the appropriate SRM. The SRM used shall be an international or national standard to ensure the provision of data of an equivalent scientific quality.

6 Ongoing quality assurance during operation

6.1 General

An AMS can drift or become less precise during routine operation. Drift or instability can be due to, for example, changes in the AMS, such as contamination of an optical surface, a gradual failure of a component, or a blockage in a filter. Such changes cause systematic errors in the data from the AMS. On the other hand, AMS are also subject to short-term variations in stability and precision due to the influences of factors such as changes in ambient temperature. These variations cause random errors. The magnitude of the random errors is assessed during the certification process of the AMS.

After the acceptance and calibration of the AMS, further quality assurance and quality control procedures shall be followed so as to ensure that the measured values obtained with the AMS meet the stated or maximum permissible uncertainty on a continuous basis (also described as *ongoing quality control*). The implementation and performance of the procedures given in this part of ISO 14385 are the responsibility of the plant owner (i.e. the owner of the AMS). It is also the responsibility of the plant owner to ensure that the AMS is operating inside the valid calibration range (see 6.5). The procedures shall be implemented and be in place at the same time that the collection of emission data by means of the AMS is mandatory for reporting to the authorities. It is recommended, however, that these procedures commence as soon as possible after the installation of the AMS in order to gain as much information on the performance of the AMS as possible. This can begin before the AMS has to be calibrated with the SRM in order to fulfil the procedure requirements according to ISO 14385-1.

The instrument reading shall reflect the actual drifts in both zero and span readings. Negative instrument readings at zero level shall be recorded.

For some monitors, it is difficult to achieve a zero and span readings. In those situations, the supplier shall give instructions on how to achieve readings that reflect the actual drift in zero and span readings, as demonstrated in the procedures according to ISO 14956, and conforming to the definition of the zero reading.

6.2 Procedures to maintain ongoing quality

The aim of the procedure is to maintain and demonstrate the quality of the AMS, so that the requirements for the stated zero and span repeatability and drift values are met during ongoing operation and the AMS is maintained in the same operational condition as when installed. This shall be achieved by confirming that the drift and precision determined during the procedures according to ISO 14956 remain under control. A suitable methodology shall determine the combined drift and precision of the AMS.

The methodology shall identify whether an extra-maintenance (e.g. by the manufacturer) is necessary in order to adjust the AMS. The procedure uses control charts which plot the drifts (zero and span) against the time. In this procedure, reference materials are needed. The value of the reference material shall be known. The drift and precision components obtained from the procedure described in ISO 14956 and the uncertainty shall be combined and compared against the combined drift and precision obtained in the field.

Control charts require regular and ideally frequent measurements. The needed frequency of the ongoing quality control is at least the period of the maintenance interval. In order to extend a maintenance interval, some AMS suppliers developed automatic checks and adjustments which guarantee very limited drifts over time. Regular measurements at zero and reference points are the foundations of the procedure. Using control charts to show trends in the zero and reference point measurements show each measurement in context and can help prevent the operator from making adjustments to the AMS only when required.

A frequency of the ongoing quality of at least once every 2 weeks is recommended. Depending on the results of the zero and span checks, this frequency can be changed.

Therefore, ongoing quality control requires plant operators to have a procedure which describes the requirements for

- measuring zero and span values,
- plotting these values on control charts, and
- using the control charts to determine whether there are systematic errors, whether the random errors exceed the acceptable limits established by the implementation requirements in an international trading scheme.

The following sub-sections describe the following:

- choosing control charts;

- setting parameters for control charts;
- zero and span measurements;
- documentation and interpretation of the control charts.

6.3 Choosing control charts

6.3.1 General

Any type of control chart, manual or automated, can be used. Different charts have different advantages and can be more or less complicated to use, depending on the type of chart chosen. This part of ISO 14385 describes two types of chart: the Shewhart chart and the EWMA chart.

6.3.2 Shewhart chart

Shewhart charts simply plot the readings and test them against multiples of S_{AMS} . Its advantage is its simplicity; its disadvantage is that the approach is not as sensitive as other approaches such as EWMA charts. Furthermore, Shewhart charts cannot distinguish between systematic errors and random errors. Shewhart charts only indicate if the AMS has drifted or whether the precision has worsened. However, the Shewhart chart method is simple to set up and understand, and it is well suited for manual procedures.

[Annex D](#) describes in detail the procedure for Shewhart chart.

6.3.3 EWMA chart

Compared with the Shewhart chart, the exponentially weighted moving average (EWMA) chart is more appropriate for early detection of small- or medium-sized maladjustments. It keeps the graphical format of the Shewhart chart. This approach also implements only one decision rule. The approach also reduces the risks of unnecessary intervention due to the natural variability of the process.

[Annex E](#) describes in detail the procedure for EWMA chart.

6.3.4 Built-in methods

An alternative to an external control chart is to use an instrument built-in method. Many instruments have a built-in check of zero and span points, and give alarm, if set limits are surpassed.

Some AMS equipped with automatic systems for zero and span checks do not ordinarily output the data for zero and span drift for plotting on control charts, even though the automatic systems are designed to achieve the same result as control charts, i.e. measuring drift and alerting the plant operator if the AMS has drifted out of control. Some systems also automatically adjust the zero and/or the span point in order. If a plant operator has such a system, it can be accepted as a method for ongoing quality control provided that an assessment of the total drifts and adjustments are possible during the AMS maintenance by the AMS supplier and that the information is also accessible to the operator and for third party auditing.

6.4 Setting parameters for control charts

6.4.1 Calculation of the standard deviation S_{AMS} using performance data

The standard deviation S_{AMS} shall be derived from the information obtained for the calculations according to ISO 14956. S_{AMS} shall be calculated considering actual plant conditions and not the test conditions during the procedures according to ISO 14956.

For example, during establishing performance characteristics of an instrument testing the influence of ambient temperature on the AMS could be defined in a range such as 5 °C to 40 °C. However, if the AMS

is kept in a climate controlled enclosure where the temperature varies from 18 °C to 23 °C, then the operator uses a temperature variation of 5 °C in the calculation for S_{AMS} .

S_{AMS} shall be calculated by

$$S_{AMS} = \sqrt{u_{inst}^2 + u_{temp}^2 + u_{volt}^2 + u_{pres}^2 + u_{others}^2} \quad (1)$$

where

u_{inst}	is the uncertainty from instability;
u_{temp}	is the uncertainty relating from variations in ambient temperature;
u_{volt}	is the uncertainty relating from variations in voltage;
u_{pres}	is the uncertainty relating from variations in ambient pressure;
u_{others}	is any other uncertainty that can influence the reading on zero and span reference material (e.g. dilution).

NOTE 1 S_{AMS} is expressed as a standard deviation; therefore, all above uncertainties are expressed as standard deviations. E.g. if the uncertainties are given as values at 95 % confidence, it is divided by the coverage factor ($k_s = 2$) for the correct calculation of S_{AMS} .

NOTE 2 It is advisable that values of uncertainties are provided by independent testing bodies.

If any of the above uncertainties are time dependent, this shall be taken into account. For example, if the uncertainty for instability is given as an upper and lower boundary such as a percentage value $\pm p$ per q days, then q equals the time between two readings for the control charts.

Examples of calculation of the standard deviation of the AMS at zero and span level are given in [Annex F](#).

6.4.2 Setting limits for control charts

The control charts required for ongoing quality control are a means of determining whether any zero and span readings are true outliers, rather than acceptable random variations. As the standard deviation S_{AMS} determines the positions of the warning and alarm limits, the value of S_{AMS} is a critical part of the control chart. In statistical terms, the purpose of S_{AMS} is to determine if there is a significant probability that a zero or span measurement is different from the target value. Therefore, S_{AMS} is usually chosen to represent one standard deviation of the acceptable variations in zero and span readings. Multiples of S_{AMS} can be chosen to represent statistical confidence intervals for the variations in zero and span readings.

6.4.3 Alternative approach for setting the control chart limits

Site data are necessary to calculate S_{AMS} . They are also needed to check whether the AMS is suitable for the monitoring purpose, i.e. to check that the uncertainty of the AMS after start-up at that particular site is lower than the maximum permissible uncertainty.

However, the collection of site data is sometimes complicated and several assumptions have to be made. Therefore, instead of using a S_{AMS} value including assumptions, some operators prefer a pragmatic and simpler approach consisting in using fixed limits for their control charts. The limits are then equal to the maximum permissible uncertainty.

Another pragmatic solution is to calculate S_{AMS} based on the specifications from performance testing. For example, an AMS can have easily met the requirements. If so, then the value of S_{AMS} based on data from test reports can result in relatively low warning and alarm limits. If the performance of the AMS falls slightly, then the control charts can direct the operator to perform more frequent maintenance than required, as some variations in performance could mean that the AMS still easily meets the uncertainty

allowances specified. A minimum value of 3 % of the measuring range shall be used for the standard deviation S_{AMS} . This avoids that the control charts wrongly indicate an out-of-range operation in case of very small standard deviations.

6.5 Zero and span measurements

6.5.1 General

Ongoing quality control requires the AMS to have a means to perform zero and span measurements. For some AMS, the use of test gases is not possible. In this case, surrogate material and/or a procedure developed by the AMS manufacturer must be used provided they are validated during a certification process.

To carry out zero and span checks internally in the AMS or in the data recording system, the AMS or the data recording systems have to be able to record both positive and negative values and record zero and span data results for a time period longer than one year to enable auditing of the data during the periodic check of the AST or during a new calibration procedure as described in ISO 14385-1.

Some AMS have been designed to perform automatic zero and span measurements. In order to fulfil the ongoing quality control requirements, the data from the zero and span measurements needs to be available to the operator.

The operator of an AMS should be aware that internal standards used to perform automatic zero and span measurements could fail.

6.5.2 Frequency of zero and span measurements

Operators have to plot zero and span data using control charts. The application of control charts requires regular and ideally frequent zero and span measurements. The maintenance interval defined during the performance testing of AMS is used as the minimum frequency for zero and span checks. However, the operator of the industrial plant can perform more frequent zero and span checks.

Manual zero and span adjustments are only performed if the control chart used indicates a need for manual adjustment.

The maximum allowable interval between zero and span measurements is known as the maintenance interval. The maintenance interval is specified by the manufacturer or determined during performance testing for approval to the requirements set by legislation. In most AMS, the maintenance interval is typically between 8 d and 1 month. Some AMS have much longer maintenance intervals; for example, from 3 to 6 months. The benefit of such AMS is that they have a proven long-term stability. Furthermore, as they do not require frequent span measurements, this means that the AMS have a higher availability for monitoring, as span measurements can be time consuming. This is most of the time achieved thanks to appropriate internal checks and adjustments during normal operation. When generating appropriate alarms, it can avoid the risk related to infrequent zero and span measurements of not detecting a systematic error in the AMS or an increase in random errors.

6.5.3 Extractive gas analysis systems

In simple terms, there are two ways to perform zero and span measurements on AMS with extractive sampling systems.

- Use of test gases: The exact concentration is not as important as the stability of the test gas. Nitrogen or ambient air without measurement components can be used as zero gas. If the sampling line serving the extractive AMS is relatively long, then the zero and span procedures can be time consuming and can consume relatively large amounts of gas and reduce the availability of the AMS. In this case, the test gas could be injected directly in front of the sampling conditioning system.
- Use of other reference materials, e.g. gas-filled cuvettes or filter devices within the AMS: The drawback of these reference materials is that they do not allow a check of the complete AMS, i.e. they

allow zero and span readings in the analyser alone and not through the complete sampling system. However, periodic manual checks by injecting gases through the sampling system can be used to verify the effectiveness of these reference materials for zero and span measurements.

6.5.4 *In situ* and cross-stack gas-monitoring AMS

There are two options for performing zero and span checks on *in situ* and cross-stack AMS.

- Use of test-gases: Cross-stack AMS can include a sintered tube which encloses the optical path of the AMS. Such tubes are similar to the sintered tubes which enclose the optical components of *in situ* AMS. In both cases, test gases can be used to perform zero and span checks. However, the sintered tubes have a relatively large volume when compared to the optical benches of most extractive AMS. This means that they have two drawbacks, a) the tubes require a large volume of gas for each test, and b) the time required can be longer than that required to perform zero and span checks on an extractive AMS.
- Use of gas-filled cuvettes and filters: If possible, it is good a practice to perform periodic manual checks by injecting gases through the sampling system, which can be used to verify the effectiveness of cuvettes for zero and span measurements.

6.5.5 Automatic zero and span checks

Many types of AMS now have built-in systems which automatically perform zero and span checks. Such systems can include extra functionality to meet all the requirements for ongoing quality control. However, some systems only warn the operator if the AMS drifts out of control and hence requires maintenance. If an AMS has an automatic system for zero and span checks, then these automatic systems are tested during performance testing.

6.5.6 Replacing gas bottles or other surrogates

When replacing gas bottles, differences in the concentrations of bottles can mislead an operative into believing that the AMS has drifted. This is because two gas bottles with seemingly identical contents can produce different readings in an AMS because of the uncertainty of the concentrations. This results in a step change in the AMS readings when one bottle is changed for another. Hence it is important to differentiate an account for such step changes, instead of mistaking such changes for drift. Therefore when changing gas bottles, the following procedure can be used:

- a) take at least three span readings with the current gas bottle, and then take an average of the readings;
- b) if the span readings using the current gas bottle show that the AMS has not drifted beyond the action limits since the last span readings, then go to d);
- c) if the AMS has drifted, then carry out any necessary actions to remedy the drift and proceed to d);
- d) take at least three span measurements using the replacement span-gas bottle, and then set a new baseline for the control-chart span-level using an average of the three measurements.

Sets of readings with an existing bottle, followed by an equal number of readings with a second bottle, establish the magnitude of any step-change.

6.6 Documentation of control charts

The control chart calculations shall be performed according to the requirements of this part of ISO 14385 and fully documented. If the zero and span checks are performed automatically with or without automatic adjustments, the check values shall be recorded in the plant computer or in a separate media (e.g. a spread sheet) in an auditable manner.

NOTE The use of a spread sheet is very suitable for the calculation procedure given above. At the same time, the sheet can be used for providing traceable documentation on the performance of the AMS.^[3]

6.7 Check on validity of measured values

The requirement that measured values are within the valid calibration range shall be evaluated by the plant owner on a weekly basis (Monday to Sunday). A full new calibration (ISO 14385-1) shall be performed, reported, and implemented within 6 months, if any of the following conditions occur:

- >5 % of the number of AMS measured values calculated over this weekly period (based on normalized calibrated values) are outside the valid calibration range for more than 5 weeks in the period between two ASTs.
- >40 % of the number of AMS measured values calculated over this weekly period (based on normalized calibrated values) are outside the valid calibration range for one or more weeks.

NOTE ISO 14385-1 allows the extension of the valid calibration range using reference materials within specified constraints.

7 Annual surveillance test (AST)

7.1 Functional test

The first part of the AST is the functional test, which shall be performed according to [Annex A](#). The functional test shall be performed by an experienced testing laboratory, which has been recognized by the competent authority.

7.2 Parallel measurements with an SRM

During AST, at least five parallel measurements with an SRM shall be performed. This shall be carried out according to the procedure described in ISO 14385-1:2014, 6.4. The purpose of comparison measurements is to verify if the calibration function of the AMS is still valid and if the precision of the AMS is still within the required limits. If this is the case, and if these measurements include results outside the valid calibration range, the valid calibration range can be increased with use of these results.

The sequence of the test of the validity of the calibration function and the variability test is described in [Figure 3](#).

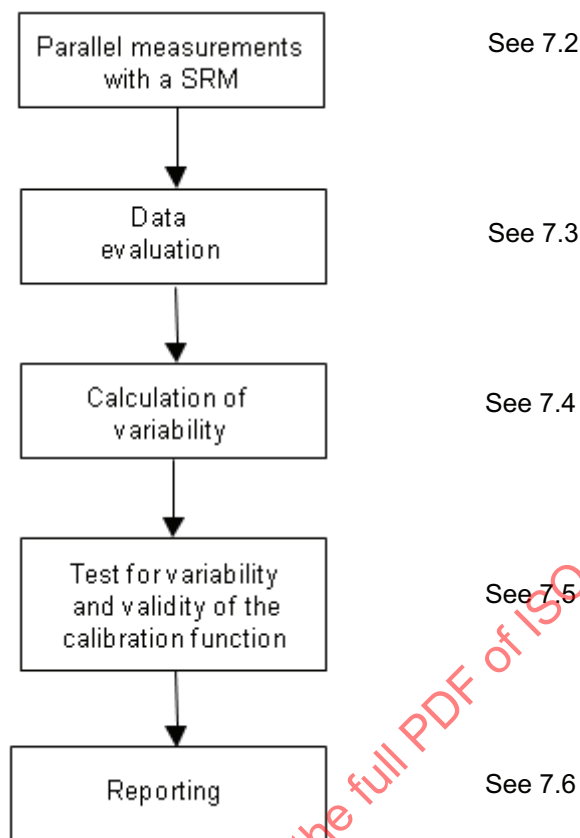


Figure 3 — Flow diagram for the calibration and variability test

Examples of calculation of the calibration function and performance of the variability test in the AST are given in [Annex F](#).

The evaluation shall be based on a minimum of five valid measurements within the calibration range. These measurements shall be uniformly spread over the whole measuring day (as described in [6.4](#)).

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

- the SRM measurements are performed according to the appropriate standard;
- the SRM measurements fulfil all the requirements given in the appropriate standard;
- the time period of each AMS measured signal is larger than 90 % of the averaging time [excluding the 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].

The sampling time per measurement shall be the same as used during the initial calibration as described in ISO 14385-1:2014, 6.4.

The sampling time for 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 in the procedures according to ISO 14956), whichever is the greater. In general, it is recommended that the sampling time used be the shortest averaging time, which is related to the legislation or to the requirements and specifications established in an international trading program.

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

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

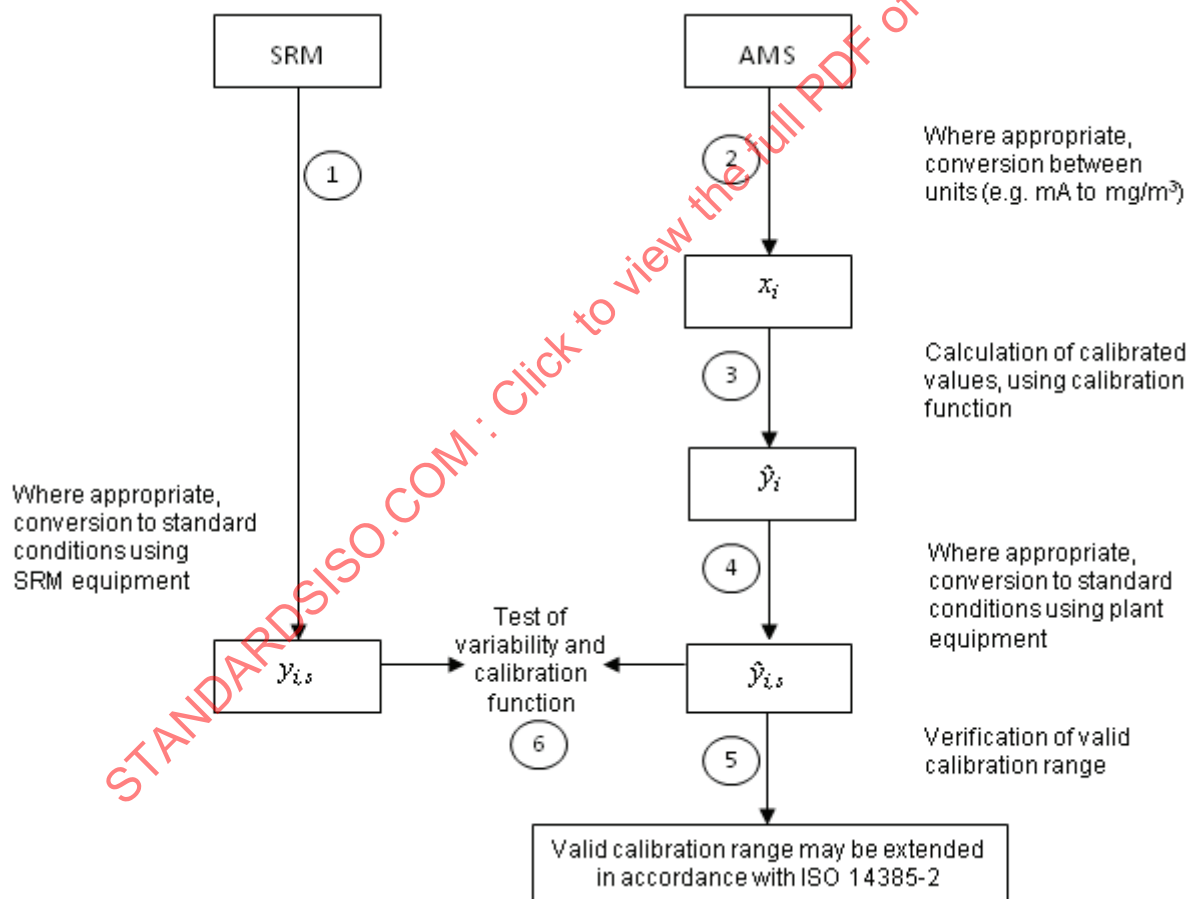
The results obtained from the SRM shall be expressed in the same conditions as the uncorrected results obtained from the AMS.

7.3 Data evaluation

The steps for providing data required for performing the variability test and to test the calibration function are illustrated in [Figure 4](#).

The data sets obtained in the parallel measurements shall be checked for possible outliers. 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 five valid data points for the test on the validity of the 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 test can be invalid.



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

Figure 4 — Flow chart describing the steps in the test for variability and calibration function and verification of the valid calibration range

Calculate the AMS measured values $\hat{Y}_i$ (calibrated values) from the AMS measured signals X_i using the established calibration function (calculated according to [6.5](#)) and use the peripheral AMS equipment to convert $\hat{Y}_i$ to standard conditions and to calculate $\hat{Y}_{i,s}$.

Verify that the measured values of the AMS are within the valid calibration range.

The results from the comparative measurements (AST) shall not be used together with the measurements from the most recent calibration to determine a new calibration function (procedures according to ISO 14956) but they can be used to extend the valid calibration range.

7.4 Calculation of variability

Identify the uncertainty required by the legislation, σ_0 , using the same procedure as in 6.3.

Calculate for all data sets:

$$D_i = y_{i,S} - \hat{y}_{i,S} \quad (2)$$

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

$$s_D = \sqrt{\frac{1}{N-1} \sum_{i=1}^N (D_i - \bar{D})^2} \quad (4)$$

7.5 Test of variability and validity of the calibration function

The variability of the measured values of the AMS is accepted if the following inequality is fulfilled:

$$s_D \leq 1,5\sigma_0 k_v \quad (5)$$

Values of k_v for different numbers of parallel measurements are given in Table 1.

Table 1 — k_v values and students t -value

Number of parallel measurements	k_v	$t_{0,95}(N-1)$
5	0,916 1	2,132
6	0,932 9	2,015
7	0,944 1	1,943
8	0,952 1	1,895

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

NOTE 2 The variability obtained includes within it 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 a false failure of the variability test). The procedure for determination of uncertainty is not in accordance with the GUM [7].

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.

The calibration of the AMS is accepted if

$$|D| \leq t_{0,95}(N-1) \frac{s_D}{\sqrt{N}} + \sigma_0 \quad (6)$$

If either of the two above tests fails, the causes shall be identified and rectified. Subsequent new parallel measurements according ISO 14385 shall be performed, reported, and implemented within 6 months. If necessary, the supplier shall be contacted in order to maintain the AMS before the next calibration.

7.6 AST report

The AST report shall contain at least the following information:

- a) a description of the plant and sampling location;
- b) a description of the AMS used, including the measurands covered, its principle, type, operation range, and its location;
- c) a description of the SRM used, its principle, type, operation range, repeatability and/or measurement uncertainty, and its EN or ISO reference number where appropriate;
- d) the dates and times of the parallel measurements;
- e) detailed data of all the measured values obtained from the AMS and the SRM, averaged over the relevant periods;
- f) method used to assess outliers and reasons for excluding outliers;
- g) the test results for validity of the precision and calibration;
- h) any deviation from the procedures described in this part of ISO 14385 (i.e. ISO 14385-2) and the possible influence on the obtained result(s);
- i) the results of the AST functional test ([Annex A](#)).

8 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 informative [Annex C](#).

Annex A (normative)

AST functional test of AMS

A.1 General

[Table A.1](#) specifies the individual steps of the functional test of AMS to be performed during AST for extractive and non-extractive AMS.

Table A.1 — Specification of individual steps of the functional test to be performed during AST

Activity	AST	
	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
Zero and span drift (audit)	X	X
Response time	X	X
Report	X	X

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 re-assembly 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;
- 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 this part of ISO 14385, 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.

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.

A.8 Linearity

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

The reference material with zero concentration, as well as the reference materials with four different concentrations, shall have a verifiable quality.

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 can 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 can change the result from fail to pass.

NOTE 2 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.

A.10 Response time

The response time of the AMS shall be checked. This can be performed, if appropriate, by feeding of 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:

$$Y_i = A' + B(X_i - X_z) \quad (\text{B.1})$$

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):

$$A' = \frac{1}{n} \sum_{i=1}^n Y_i \quad (\text{B.2})$$

where

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

Y_i 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):

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

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.

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 Formula (B.4):

$$A = A' - B X_z \quad (\text{B.4})$$

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 :

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

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):

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

Convert d_c in concentration units to a relative unit $d_{c,\text{rel}}$ by dividing d_c by the upper limit c_u of the measuring range:

$$d_{c,\text{rel}} = \frac{d_c}{c_u} 100 \% \quad (\text{B.7})$$

B.4 Test of the residuals

Test each residual according to

$$d_{c,\text{rel}} < 5 \% \quad (\text{B.8})$$

All residuals shall pass this test.

Annex C **(informative)**

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 Setting-up 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;

- 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 designation 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

Annex D (informative)

Shewhart control charts

Under the ongoing quality control procedures, the operator regularly checks the response of the AMS to zero and span reference materials. If these readings are repeated over a sufficiently short period of time, and the AMS has not had a chance to drift, then the actual readings will be due to variations in precision and allowable effects of influence quantities. Over a period of time, as the operator collects more data, there is only a very small chance that the readings will change by more than three standard deviations, unless the AMS has truly drifted. The purpose of control charts is to plot such trends and give an indication of actual or forthcoming drift.

The user either calculates or determines the standard deviation S_{AMS} for the operation of the instrument under anticipated stack conditions and then uses multiples of this standard deviation to set warning levels and alarm (or intervention) levels. Two control charts are needed, one for zero drift and one for span drift.

The results are presented as a function of time. The values shown by the AMS can be expressed as an absolute value or as the difference between the reading and the expected value of the reference material. Two control charts are needed, one for zero drift and one for span drift. [Figure D.1](#) shows an example template for span drift.

The target values on the two charts are the average value of zero and span readings $\bar{y}_z$ and $\bar{y}_s$ established during the initial steps in the ongoing quality control. This should be carried out immediately after the procedures according to ISO 14385-1 have been completed. The associated standard deviations $S_{AMS(zero)}$ and $S_{AMS(span)}$ are used to calculate the levels which will trigger an alarm and possible intervention.

The upper and lower warning levels are given by

$$\bar{y}_s + \frac{2S_{AMS(span)}}{\sqrt{n}} \text{ and } \bar{y}_s - \frac{2S_{AMS(span)}}{\sqrt{n}} \quad (D.1)$$

The upper and lower alarm limits are given by

$$\bar{y}_s + \frac{3S_{AMS(span)}}{\sqrt{n}} \text{ and } \bar{y}_s - \frac{3S_{AMS(span)}}{\sqrt{n}} \quad (D.2)$$

where n is the number of consecutive repetitions of the test carried out (n should be at least 10 for the initial steps in operating control charts but can be equal to 1 in the case of a repeat use control charts). Once the control chart is set up, the results of the zero and span tests (averages of the n readings on the AMS) are placed on the chart in order to detect drift and/or changes in precision that require intervention by the operator, e.g. maintenance of the AMS and possible rejection of the results since the previous tests. The operator shall intervene when any of the following happens:

- one or more data points are beyond one of the upper alarm limits;
- three consecutive data points are beyond one of the warning limits;
- four points among 5 consecutive ones are beyond $\bar{y}_s \pm \frac{S_{AMS}}{\sqrt{n}}$ i.e. half the alarm levels for span;
- eight consecutive points are on the same side of the mean;
- six consecutive points are either increasing or decreasing.

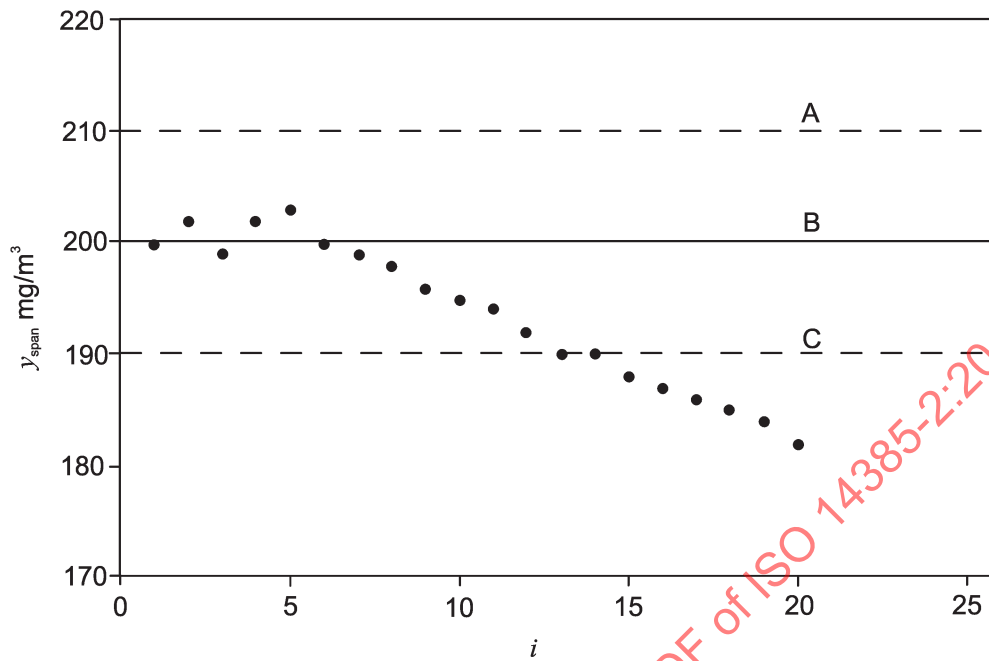
Example

The following example shows a series of span measurements for N₂O, with 20 checks for the span value at regular intervals (see [Table D.1](#)). Absolute values and relative values with the target value are shown, while in this example, S_{AMS} would be calculated using the procedure in [6.4.1](#) of. For the sake of example, the value of S_{AMS} is given as 5 mg/m³.

Table D.1 — Raw and relative data for Shewhart control charts

Span-point number	Span value mg/m ³	Deviation from the base- line mg/m ³
1	200	0
2	202	2
3	199	-1
4	202	2
5	203	3
6	200	0
7	199	-1
8	198	-2
9	196	-4
10	195	-5
11	195	-6
12	192	-8
13	190	-10
14	190	-10
15	188	-12
16	187	-13
17	186	-14
18	185	-15
19	184	-16
20	182	-18

[Figure D.1](#) shows a plot of the span measurements in sequence, together with the deviation from the baseline. The results show the random variations quite clearly over the first 10 measurements, although it is not clear whether there is a systematic change until over 15 measurements have been taken, even though there appears to be a trend of drift.

**Key** y_{span} span value, in milligrams per cubic metre (mg/m^3) i number of span check, with $i = 1$ to n

A upper action limit

B baseline

C lower action limit

Figure D.1 — Example of a Shewhart chart

Annex E (informative)

Exponentially weighted moving average (EWMA) charts

Two solutions are possible to improve the efficiency in detecting a slow and gradual change in the precision and accuracy of the AMS due to drift. The options are to

- increase the number of checks, since efficiency increases with n , but this produces a cost that increases proportionally to $\sqrt{n}$; however, increasing the number of checks also decreases the availability of the AMS and
- take previous results into account.

Exponential weighted moving average (EWMA) charts improve the efficiency of detection by using results of measurements previous to the last check, whereas Shewhart charts do not. This Annex shows an example of an EWMA chart.

Compared with the Shewhart chart, the EWMA chart

- is more appropriate for early detection of small or medium-sized drift,
- is easier to set up and keeps the graphical format of the Shewhart chart, and
- implements only one decision rule.

The EMWA control chart requires three preparatory steps:

- selecting the control chart parameters:
 - m_0 the centre line;
 - $s_0 = s_{AMS}$ the process standard deviation;
 - n the number of checks ($n = 1$ for one zero or span check);
 - δ the shift to be detected as soon as possible as a multiple of s_0 ;
 - $ARL(0)$ the average run length corresponding to a risk of a false alarm;
 - $ARL(\delta)$ the average run length to detect a shift δ ;
 - λ the smoothing parameter that determines the depth of memory of the EWMA;
 - K a constant for setting the control limits.
- determining weighting of the past values with the last reading:

$$z_i = \lambda x_i + (1 - \lambda) z_{i-1} \quad (E.1)$$

where

z_i is the weighted average taking the past and the last check into account;

x_i is the AMS reading for the last check;