# Stationary source emissions — Guidance on the application of EN 14181:2004

ICS 13.040.40



# National foreword

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#### **English Version**

# Stationary source emissions - Guidance on the application of EN 14181:2004

Emissions de sources fixes - Guide d'application de l'EN 14181:2004

Emissionen aus stationären Quellen - Leitlinien zur Anwendung der EN 14181:2004

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# **Foreword**

This document (CEN/TR 15983:2010) has been prepared by Technical Committee CEN/TC 264 "Air quality", the secretariat of which is held by DIN.

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# Introduction

This CEN Technical Report provides supporting guidance on the application of EN 14181:2004. It is based on the growing experiences with EN 14181:2004 throughout the CEN member countries. EN 14181:2004 specifies three levels of quality assurance (QAL), known as QAL1, QAL2 and QAL3 as well as an Annual Surveillance Test (AST). This Technical Report explains the requirements of these levels of quality assurance to achieve a consistent application of EN 14181:2004.

## 1 Scope

This CEN Technical Report provides guidance for applying the European Standard EN 14181:2004.

This CEN Technical Report provides guidance only on applying the quality assurance levels QAL1, QAL2 and QAL3 as well as the Annual Surveillance Test (AST).

This CEN Technical Report is an informative document.

#### 2 Terms and definitions

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

#### 2.1

#### air quality characteristic

one of the quantifiable properties relating to an air mass under investigation, for example, concentration of a constituent

[EN 14181:2004, 3.1]

#### 2.2

#### automated measuring system

#### **AMS**

measuring system permanently installed on site for continuous monitoring of emissions

[EN 14181:2004, 3.2]

NOTE 1 An AMS is the automated application of a monitoring method, which is traceable to a reference method.

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

#### 2.3

#### calibration function

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

[EN 14181:2004, 3.3]

NOTE The calibration function is established during QAL2 on stack gases.

#### 2.4

#### competent authority

organisation which implements the requirements of EU Directives and regulates installations, which must comply with the requirements of applicable European Standards

[EN 15267-1:2009, 3.3]

#### 2 5

#### confidence interval (two-sided)

when  $T_1$  and  $T_2$  are two functions of the observed values such that,  $\theta$  being a population parameter to be estimated, the probability  $P_r(T_1 \le \theta \le T_2)$  is at least equal to  $(1 - \alpha)$  [where  $(1 - \alpha)$  is a fixed number, positive and less than 1], the interval between  $T_1$  and  $T_2$  is a two-sided  $(1 - \alpha)$  confidence interval for  $\theta$ 

[EN 14181:2004, 3.5]

#### 2.6

#### **CUSUM** chart

calculation procedure in which the amount of drift and change in precision is compared to the corresponding uncertainty components which are obtained during QAL1

[EN 14181:2004, 3.6]

#### 2.7

#### drift

monotonic change of the calibration function over stated period of unattended operation, which results in a change of the measured value

[EN 14181:2004, 3.7]

NOTE This refers to a change in the response of the AMS to a determinant which does not change.

#### 2.8

#### emission limit value

#### ELV

limit value related to the uncertainty requirement

[EN 14181:2004, 3.8]

NOTE For EU Directives it is the daily emission limit value that relates to the uncertainty requirement.

#### 2.9

#### extractive AMS

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

[EN 14181:2004, 3.9]

#### 2.10

#### instability

change in the measured value comprised of drift and dispersion resulting from the change in the calibration function over a stated period of unattended operation, for a given value of the air quality characteristic

- NOTE 1 Drift and dispersion specify the monotonic and stochastic change with time of the output signal, respectively.
- NOTE 2 This refers to a change in the response of the AMS to a determinant which does not change.
- NOTE 3 Adapted from EN 14181:2004, 3.10.

## 2.11

#### instrument reading

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

[EN 14181:2004, 3.11]

#### 2.12

#### intrinsic uncertainty

uncertainty component originating from the AMS itself, independent of the installation

#### 2.13

#### legislation

Directives, Acts, ordinances and regulations

[EN 14181:2004, 3.12]

#### 2.14

#### measurand

particular quantity subject to measurement

[EN 14181:2004, 3.13]

#### 2 15

#### measured value

estimated value of the air quality characteristic derived from an output signal

NOTE 1 This usually involves calculations related to the calibration process and conversion to required quantities.

NOTE 2 Adapted from EN 14181:2004, 3.14.

#### 2.16

#### non-extractive AMS

AMS having the detection unit in the gas stream or in a part of it

[EN 14181:2004, 3.15]

#### 2.17

#### outlier

observation that lies an abnormal distance from other values in a set of data, and therefore has a low probability of being a valid data point

#### 2.18

#### 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

[EN 14181:2004, 3.16]

#### 2.19

#### peripheral AMS or SRM

measuring system or SRM used to gather the data required to convert the measured values to standard reference conditions, i.e. AMS or SRM for moisture, temperature, pressure and oxygen

[EN 14181:2004, 3.17]

#### 2.20

#### precision

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

[EN 14181:2004, 3.18]

#### 2.21

#### reference material

material simulating a known concentration of the input parameter, by use of surrogates and traceable to national standards

NOTE Surrogates normally used are calibration gasses, gas cells, gratings or filters.

[EN 14181:2004, 3.19]

#### 2.22

#### response time

time interval between the instant 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

[EN 15267-3:2008, 3.31]

#### 2.23

#### span reading

instrument reading of the AMS for a simulation of the input parameter at a fixed elevated concentration

[EN 14181:2004, 3.21]

NOTE 1 This simulation is intended to test the measuring elements of the system, which contribute to its performance.

NOTE 2 The span reading is approximately 80 % of the measurement range.

#### 2.24

#### standard conditions

conditions as given in the EU-directives to which measured values have to be standardised to verify compliance with the emission limit values

[EN 14181:2004, 3.22]

#### 2.25

#### standard deviation

positive square root of: the mean squared deviation from the arithmetic mean divided by the number of degrees of freedom

NOTE The number of degrees of freedom is the number of measurements minus 1.

[EN 14181:2004, 3.23]

#### 2.26

#### standard reference method

#### **SRM**

method described and standardised to define an air quality characteristic, temporarily installed on site for verification purposes

NOTE Also known as a reference method.

[EN 14181:2004, 3.24]

#### 2.27

#### uncertainty

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

[EN 14181:2004, 3.25]

#### 2.28

#### variability

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

[EN 14181:2004, 3.26]

#### 2.29

## zero reading

instrument reading of the AMS on simulation of the input parameter at zero concentration, which tests the measuring elements of the AMS, that contribute to its performance

NOTE Adapted from EN 14181:2004, 3.23.

# 3 Symbols and abbreviations

# 3.1 Symbols

а	intercept of the calibration function
b	slope of the calibration function
C	mass concentration in milligrams per cubic metre (mg/m³)
$D_i$	difference between measured SRM value $y_i$ and calibrated AMS value $\hat{y}_i$
$\overline{D}$	average of $D_i$
E	emission limit value
$E_{\sf otm}$	extinction of the optical transmission monitor
i	index
$k_{V}$	test value for the variability test based on a $\chi^2$ -test, with a $\beta$ -value of 50 %, for ${\it N}$ numbers of paired measurements
L	control limit value
$L_{\sf mp}$	length of the measurement path in metres (m)
$m_0$	target value (chart centre line)
n	number of checks
N	number of paired samples in parallel measurements
P	percentage value
$R^2$	correlation coefficient
$s_{AMS}$	standard deviation of the AMS at zero and span level
$s_D$	standard deviation of the differences $D_i$ in parallel measurements
t <sub>0,95</sub>	students t-factor for a confidence level of 95 %
$x_i$	$\it i^{th}$ measured signal obtained with the AMS
$y_i$	$\it i^{th}$ measured result obtained with the SRM
$\hat{\mathcal{Y}}_i$	best estimate for the true value, calculated from the AMS measured signal $x_i$ by means of the calibration function
$\mathcal{Y}$ span	span value
Z	critical value in the Grubbs's test
$Z_i$	test value of $i^{th}$ data pair in the Grubbs's test
$z_i$	weighted average taking the past and the last check into account
λ	smoothing parameter

 $\mu$  average diameter of the grains in the stack in micrometres ( $\mu$ m)

 $\sigma_0$  uncertainty derived from requirements of legislation

#### 3.2 Abbreviations

AMS automated measuring system

ARL average run length

AST annual surveillance test

ELV emission limit value

EWMA exponentially weighted moving average

LCL lower control limit
NO<sub>x</sub> nitrogen oxides

QAL quality assurance level

SRM standard reference method

TOC total organic compounds

UCL upper control limit

## 4 General guidance on quality assurance and calibration

#### 4.1 General

The role of this Technical Report is to provide guidance on the application of the European Standards EN 14181:2004 on quality assurance of automated measuring systems used for monitoring stationary source emissions and EN 13284-2:2004 on automated measuring systems used for the determination of low range mass concentration of dust at stationary sources. Both European Standards are applicable to industrial plants falling under the European Directives for the incineration of waste (2000/76/EC) and large combustion plants (2001/80/EC), hence referred to as Directives in this Technical Report.

For simplicity, throughout this document, reference to EN 14181:2004 also refers to EN 13284-2:2004.

This Technical Report summarises the requirements of EN 14181:2004 and EN 13284-2:2004, and provides guidance on how to perform each of the required tasks.

#### 4.2 Regulatory framework and standards for monitoring

#### 4.2.1 Monitoring requirements in the Directives

The Directives prescribe the use of European Standards for monitoring emissions and calibration of automated measuring systems, or, if European Standards are not available, then the use of ISO, national or other equivalent international standards that provide data of a suitable quality. The standards for monitoring emissions are known as standard reference methods (SRM). Furthermore, the Directives specify overall performance requirements for continuous monitoring through uncertainty allowances expressed as a 95 % confidence interval. EN 14181:2004 presumes that the uncertainty of the AMS is expressed in the applicable Directives as half of the length of a 95 % confidence interval as a percentage P of the emission limit value E.

#### 4.2.2 Scope and structure of EN 14181:2004

EN 14181:2004 applies to AMS permanently installed at industrial plants regulated under the Directives. Also EN 14181:2004 applies to the AMS themselves and not the data recording systems used with AMS. The requirements for data acquisition and handling systems will be covered by a separate standard. The scope of EN 14181:2004 applies to complete AMS as defined by EN 15267-3, which includes not just the analyser, but also any sampling systems and other components required to analyse the stack gas and produce a measurement.

Although EN 14181:2004 was developed for application at industrial plants covered by the Directives, it can be applied to industrial plants covered by other EC laws, such as for other types of industrial plants regulated under the Directive 96/61/EC for Integrated Pollution Prevention and Control (IPPC). EN 14181:2004 specifies three quality assurance levels and an annual surveillance test. These are:

- QAL1 is a procedure to demonstrate that the AMS is suitable for the intended purpose before installation, by meeting required performance standards EN ISO 14956, and the uncertainty allowances specified in EU Directives. Since the publication of EN 14181:2004, CEN has published EN 15267-3 to apply EN ISO 14956 for new AMS.
- QAL2 includes a set of functional tests to check that the AMS has been installed appropriately, and that the AMS is operating correctly. The functional tests on the AMS are then followed by a procedure to calibrate the AMS, using standard reference methods and then verify whether it still meets the required uncertainty allowances, once installed. QAL2 establishes the traceability of the AMS measured values, to the applicable standard. This provides a demonstration of compliance with legally binding emission limit values.
- QAL3 is a procedure to maintain and demonstrate the required quality of the AMS during its normal operation by checking the zero and span readings.
- AST is a set of functional tests to check the correct operation of the AMS, followed by a procedure to
  evaluate the AMS to show that it continues to function correctly and the calibration function is still valid.

These quality assurance levels follow a logical sequence to demonstrate the suitability of the AMS, its correct installation, commissioning, and calibration, followed by procedures to ensure a continuing and correct operation (see Figure 1).

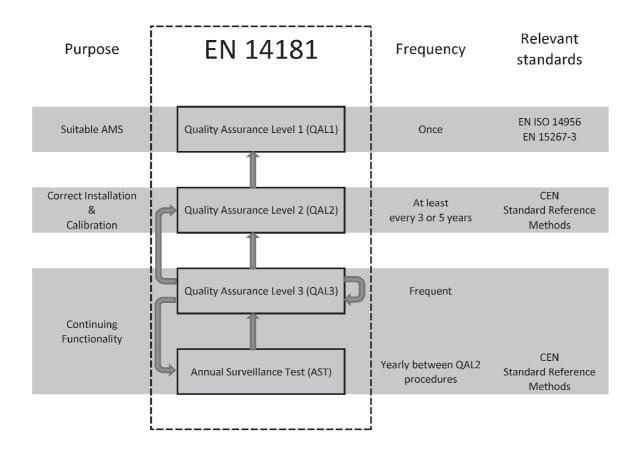


Figure 1 — Quality Assurance Levels from EN 14181

#### 4.3 Roles and responsibilities

The operator of an industrial plant regulated by the Directives has the overall responsibility to comply with the requirements of EN 14181:2004. The operator is responsible for organising the functional tests, according to the requirements of the competent authority. The AMS manufacturer, AMS supplier, the operator's own personnel, or a test laboratory may therefore perform the functional tests, depending on the requirements of the competent authority.

Although the operator is responsible for compliance with the requirements of EN 14181:2004, this standard specifies that the test laboratories which perform the parallel measurements, using standard reference methods, as required by QAL2 and the AST, shall be either accredited to EN ISO/IEC 17025, or shall otherwise meet the requirements of the competent authority. Therefore, EC member states may use national systems for approving testing laboratories for QAL2 and the AST.

#### 5 Application of QAL1

#### 5.1 General

The purpose of QAL1 is to show that an AMS is potentially suitable for its intended task, by demonstrating that the uncertainty at the emission limit value (ELV) will at least meet the uncertainty specified in the Directives. The ability of the AMS to meet this uncertainty in turn depends on:

 the ability of the AMS to capture all or most of the measurement peaks over a sufficiently wide measurement range, without compromising the required uncertainty at the ELV; and — the ability of the AMS to be sufficiency stable once installed, i.e. any drift and precision are acceptable within allowable specifications as defined by national or international standards.

## 5.2 AMS which are not yet installed at the plant

EN 14181:2004 refers to EN ISO 14956 when applying the requirements of QAL1. EN ISO 14956 describes a procedure to determine the uncertainty, using performance criteria and test data for an AMS. However, EN ISO 14956 only describes the mathematical procedure for determining the uncertainty. This standard does not describe exactly which performance criteria and data to use within the calculations, nor how to perform the tests to produce the data for the uncertainty calculations.

Therefore, CEN has published EN 15267-3, which specifies the performance criteria and test procedures for automated measuring systems for monitoring emissions from stationary sources in the framework of certification of automated measuring systems. This standard is both an application of EN ISO 14956, and a means of demonstrating compliance with the QAL1 requirements of EN 14181:2004. This means that, if AMS which have been tested to the requirements of EN 15267-3, and the results of these tests show that the uncertainty meets the requirements of the Directives, then the AMS meets the requirements for QAL1.

EN 15267-3 and the national standards which preceded it make allowances for the uncertainty contributions of the installation. For example, EN 15267-3 recommends that the uncertainty of the AMS, estimated using the procedure in EN ISO 14956, is not be more than 75 % of the uncertainty allowance specified in the Directives. This allows a margin of error for factors such as the uncertainty contributions from peripheral measurements and stack gas inhomogeneity.

There are many AMS which have been tested and certified in accordance with national standards which preceded EN 15267-3. In such cases, operators of industrial plants are advised to contact the competent authorities for guidance.

Annex D of EN 15267-3:2007 provides an example of an uncertainty determination for an AMS, applying EN ISO 14956 to determine the uncertainty.

## 5.3 AMS which are already installed at the plant

There can be cases where AMS are already installed at the plant, and where the AMS do not meet all of the requirements of EN 15267-3. However, the performance criteria specified within EN 15267-3 are set at a level which provides a margin of safety. This means that AMS which meet the requirements of EN 15267-3 are very likely to meet the uncertainty allowances specified in the Directives. Therefore, an AMS which does not meet the performance requirements of EN 15267-3 might still meet the uncertainty allowances specified within the Directives. In such cases, the operator can minimise the effects of influence factors which increase the uncertainty of the AMS once it is installed at the plant. For example, the AMS could be contained within a climate-controlled chamber, which could minimise the influence of variations in ambient temperature on the AMS.

Therefore if an AMS already installed at the plant does not meet the requirements of EN 15267-3, and hence the requirements of QAL1, the competent authorities in EC member states may decide what action is necessary. For example, the competent authority may state that if the AMS still meets the requirements of QAL2, QAL3 and the AST, then the operator may keep the AMS for the rest of its design life.

#### 6 Application of QAL2 and AST

#### 6.1 Tasks within QAL2 and AST

QAL2 requires operators to assure that AMS are installed in the correct location, that there is sufficient access to maintain, assess and control them, and to ensure that AMS are both calibrated and operating correctly. To this end, EN 14181:2004 specifies two parts to QAL2, which are:

- A set of functional tests and checks to ensure that the AMS has been installed correctly and is functioning at, or better than, the required performance levels; and that there are sufficient management-provisions in place for the management and maintenance of the AMS. EN 15259 describes a procedure to identify the best location for the sampling location of the AMS, in order to provide representative measurements.
- A set of repeated, parallel measurements using the SRM to verify whether the readings from the AMS are reliable, and to derive the calibration function. This includes a set of statistical operations and tests following the parallel measurements, to verify whether the AMS meets the uncertainty allowances specified in the EU Directives.

Although the scope of EN 14181:2004 excludes systems for data acquisition, it is good practice to check the data transfer from the AMS to the data acquisition and handling system during the QAL2 and AST.

#### 6.2 Location and monitoring provisions for AMS

If an AMS is to give reliable results, then it is critical that the AMS is located in the correct position, such that it measures a representative sample of the emissions. Furthermore, the sampling ports for periodic monitoring also need to be located in a position which provides a representative sample, and allows a reliable comparison of sampled emissions with the emissions measured by the AMS. Therefore EN 14181:2004 requires operators to ensure that the AMS are installed in the correct location, and that there is sufficient access to assess, control and maintain them. EN 15259 provides guidance on the location of both AMS and sampling ports, as well as appropriate provisions for monitoring.

#### 6.3 Management system provisions for AMS

According to EN 14181:2004, the continued effective operation of AMS depends on the operator of the industrial plant having both the provisions and procedures in place to manage and maintain the AMS. Therefore, under A.4 of EN 14181:2004, there are requirements for documentation to support the management of the AMS. Such procedures can include specific provisions for AMS, covering:

- selection;
- maintenance and servicing;
- responsibilities and training of personnel;
- calibration, quality assurance checks and controls;
- records and data management;
- prevention of unauthorised adjustment of the AMS and its data recording devices; and
- maintaining availability by spares, contingencies and back-up monitoring.

A systematic approach to managing and maintaining the AMS, documented through procedures within an existing management system, meets the requirements of EN 14181:2004. Whilst operators can include these management system provisions within existing management systems certified to standards such as EN ISO 14001 and EN ISO 9001, more detailed guidance is available in a specific standard for measurement management systems, EN ISO 10012.

#### 6.4 Specific issues of the functional tests

#### 6.4.1 General

EN 14181:2004 requires a set of functional tests to be carried out as part of the QAL2 and the AST. The objective of the functional tests is to ensure that the AMS is working effectively, and that it is ready for parallel tests using the SRM. Annex A of EN 14181:2004 contains a detailed description of the functional tests and

related activities. There are some issues to consider when performing the functional tests, such as management system provisions for the AMS, zero and span checks, and the related linearity tests.

The test of lack-of-fit, response time and zero and span drift can be combined within one test. EN ISO 9169 describes a procedure to combine these tests. It is good practice to measure the response time for extractive systems for the analyser alone, and then the analyser plus the sampling system. This allows the determination of any losses in the sampling system, as well as the lag time within the sampling system.

#### 6.4.2 Zero and span checks

EN 14181:2004 requires zero and span checks. EN 15267-3 and EN ISO 9169, for example, describe procedures for these tests. Typically the zero and span checks require the use of reference materials. These tests require the following:

- Each AMS for gaseous compounds has an injection point for the test gases as close as possible to the sampling point in order to check the response time of the complete AMS. In addition to a check of the complete system, injecting the test gas directly into the analyser of the AMS allows a check of the losses in the sampling system, and the lag time caused by the sampling system.
- The output for the raw signal(s) is accessible and useable.

The lag times of the SRM and of the AMS, including the sampling systems, are needed to match the measurements from the AMS to those from the SRM, taking into account any delays due to sampling systems of the AMS and SRM. This applies to peripheral measurements, as well as the main measured components.

#### 6.4.3 Linearity test

The linearity test (lack-of-fit) is specified for the AST but not for the QAL2 test. However, it can be beneficial to perform this test during the QAL2 as well. For example, EN 14181:2004 permits the use of reference materials to extrapolate the calibration function up to the ELV, subject to certain conditions. There are also a number of issues to consider when planning and performing the linearity test.

It is good practice to use a test gas with an uncertainty which meets the requirements for test gases specified in EN 15267-3. Additionally, using a gas-mixing system to provide different concentrations of the test gas has a lower uncertainty than using different bottles of test gas for each required concentration.

In the case of HCl, HF and  $NH_3$ , for example, the test can take several hours and therefore compromise the availability of the AMS. Therefore, both process operators and test laboratories can consider alternative means of monitoring the process emissions whilst performing the test for linearity. Such alternative provisions can include hot-standby AMS or portable standby AMS.

If the tests for linearity take several hours, then the test laboratory and operator may wish to consult the competent authority about their position on maintenance operations, functional tests, and how these are seen to affect AMS availability. In cases where the time taken for the linearity test is unacceptably long, EN 15267-3 describes a procedure to shorten tests when justified and can provide data of a suitable quality.

If the AMS measures sampled source emissions which are heated and without moisture removed, then it is best practice to perform the test for linearity test using moist gases, using a combination of test gases and a moisture generator.

If the AMS measures moisture, then it is good practice to perform the linearity test for moisture.

#### 7 Calibration and validation of the AMS

#### 7.1 Standard reference methods

The effective application of QAL2 and AST depends on the robust application of standard reference methods. Accreditation to EN ISO/IEC 17025 for the applicable SRM is a means of achieving this aim. Therefore, many competent authorities in EC Member states require test laboratories to be accredited to EN ISO/IEC 17025 for the applicable SRM. However, EN ISO/IEC 17025 is a generic standard for the quality assurance of test laboratories and can apply to any type of laboratory which performs services for both testing and calibration. So CEN has produced a supplementary Technical Specification to EN ISO/IEC 17025, which is CEN/TS 15675. This Technical Specification elaborates on the requirements of EN ISO/IEC 17025, specifically for manual stack-emission monitoring. Therefore applying CEN/TS 15675 is likely to improve the quality of the application of the SRM, and therefore improve the quality of the QAL2 and AST procedures.

#### 7.2 Calibration using an SRM

#### 7.2.1 General

Figure 2 illustrates the principle of linear calibration using an SRM in which the SRM data is compared with the AMS data and is used to derive a calibration function. The AMS itself may have a bias in one direction or another, depending on gain of the AMS and its offset relative to zero. A calibration function, in its simplest form, can then be described by Equation (1):

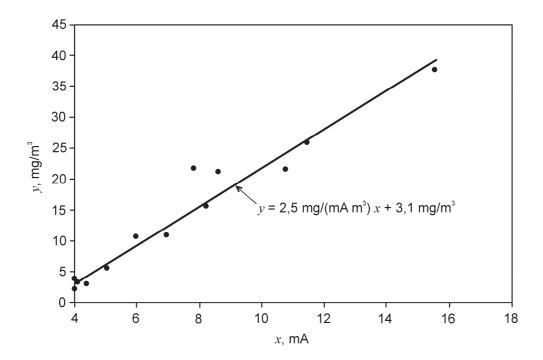
$$y_i = a + b x_i \tag{1}$$

where

- $x_i$  is the  $i^{th}$  measured signal obtained with the AMS, with i = 1 to N;
- $y_i$  is the  $i^{th}$  measured result obtained with the SRM, with i = 1 to N;
- *a* is the intercept of the calibration function;
- *b* is the slope of the calibration function.

There are two important factors to take into account when applying a calibration factor:

- The calibration function depends on linear responses to increasing concentrations of the measured components, for both the SRM and AMS. However, it is probable that there will be some imprecision in the agreement between the AMS and SRM measurements. This imprecision is caused by the uncertainties of both the SRM and AMS measurements. Therefore it is important to minimise the uncertainty of the SRM as far as practicable, as the uncertainty of the SRM could adversely affect both the calibration function and the outcome of the variability test. Many SRM have been developed to have an uncertainty which is typically half of the uncertainty required by the Directives. Applying CEN/TS 15675 helps to assure the lowest possible uncertainty in the SRM.
- EN 14181:2004 specifies that there shall be evidence to show that the AMS produces readings at or near zero, when the emissions are at or near zero.



#### Key

- x AMS measured signal, in milliamperes (mA)
- y SRM measured value, in milligrams per cubic metre (mg/m³)

Figure 2 — Principle of linear calibration using a SRM

#### 7.2.2 Spread of data

In order to meet the requirements of EN 14181:2004, QAL2 requires a set of data representing normal operating conditions. Furthermore, the data needs to cover as wide a range as possible. Some industrial processes can be varied in order to increase the emissions up to the ELV, in order to achieve a wide range of concentrations in the sampled emissions. However, it is the decision of the competent authority whether to allow adjustments to the industrial process, in order to artificially increase the emissions in order to provide a wider spread of data. Ideally, the QAL2 takes place at a time when the emissions are likely to be their highest and most varied; for example, when bag filters are replaced, emissions of particulate are temporarily higher and this produces an ideal time to measure a wider range of emissions.

#### 7.2.3 Number of data points

EN 14181:2004 specifies that the test laboratory needs to have at least 15 repetitions of the SRM for a QAL2, and at least five repetitions for an AST. Therefore EN 14181:2004 recommends that the test laboratory takes more than the minimum number of repetitions, because there is a significant chance that at least one result is an outlier. Therefore, it is better to have more data than is necessary, whereas a test laboratory which has just 15 pairs of data for the SRM and AMS can find that there is an insufficient number of data points if even one data point is an outlier. Therefore, it is better to take, for example, between 18 and 20 pairs of data, and then determine if any of these data points are invalid using a procedure for analysing for the presence of outliers (see 7.2.5 of this Technical Report).

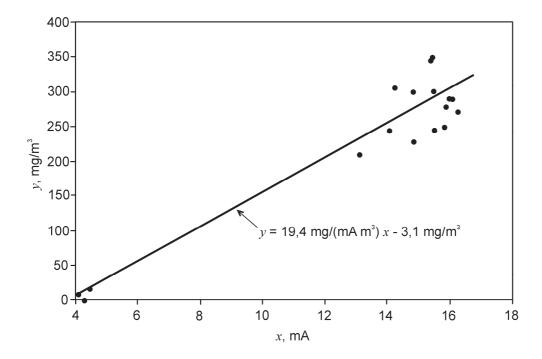
At the same time, if a test laboratory takes well over the minimum number of paired samples for the AMS and SRM, then all valid pairs of data need to be included in the QAL2 and AST calculations.

#### 7.2.4 Values near zero

As EN 14181:2004 specifies that there shall be evidence to show that the AMS produce readings at or near zero, when the emissions are at or near to zero, then measurements are needed at or near zero.

Ideally zero values are measured when the industrial plant is not producing emissions. If this is not possible, then the competent authorities can allow the test laboratory to use surrogate values.

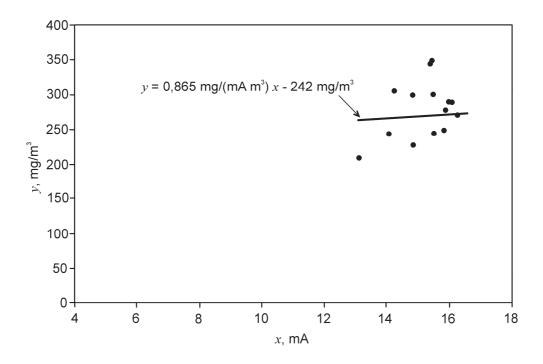
Figure 3 and Figure 4 illustrate the impact of clustered data points with and without measurements at zero. In Figure 3, there are three values near zero and the calibration function shows an acceptable agreement between the SRM and AMS values, with an approximate one to one mathematical relationship. However, Figure 4 shows that in the absence of zero values within a similar set of data, there is a completely different and incorrect calibration function. In such cases, EN 14181:2004 allows a forcing of the calibration line through zero if the difference between the highest and lowest measured SRM concentrations at standard conditions is smaller than 15 % of the ELV.



## Key

- x AMS measured signal, in milliamperes (mA)
- y SRM measured value, in milligrams per cubic metre (mg/m³)

Figure 3 — Example of an adequate spread of data for a set of QAL2 measurements



#### Key

- x AMS measured signal, in milliamperes (mA)
- y SRM measured value, in milligrams per cubic metre (mg/m³)

Figure 4 — Example of a set of QAL2 measurements without measurements near zero

#### 7.2.5 Invalid values and outliers

An outlier is defined as an invalid data point. Plotting AMS and SRM data on a graph shows whether there are any obvious outliers. There can be several causes of invalid data, such as:

- errors in the SRM;
- failures of an AMS or instrument used for SRM; or
- automatic zero and span functions of the AMS.

Invalid data caused by these influences can be avoided by correct application of the SRM, the check of the measuring systems and instruments before they are used in QAL2 and by switch-off of the automatic zero and span checks during QAL2.

Data can appear to be outliers without there being an obvious and immediately apparent reason. However, as EN 14181:2004 requires test laboratories to identify invalid data, this means that a test laboratory needs to have a systematic approach to identifying outliers. There are several tests for outliers, although test laboratories can choose any validated method. Most tests are based on the following principles:

- In any data set of paired samples,  $(\hat{y}_i, y_i)$  with i = 1 to N, the differences  $D_i$  between the paired samples are distributed normally.
- There is an average of the differences of the paired samples, and there is a standard deviation  $s_D$  of the differences.

- If the difference  $D_i$  between any pair of samples is greater or smaller than the average difference, by more than two standard deviations  $s_D$ , then there is a strong chance that the paired sample is an outlier.
- After a test for outliers, the outlier which deviates the most from the average is rejected, and then the outlier test is repeated.

Measurements above the ELV, however, can fail the outlier test because of signal-proportional uncertainty contributions which can increase the uncertainty at larger values of the measured signal from the AMS. In such situations, a visual inspection of the data can show whether such data points are true outliers.

Annex A shows an example of a procedure for determining outliers in a set of data.

#### 7.2.6 Decision on Method A or Method B

EN 14181:2004 applies ISO 11095, which is a standard for calibrating measuring devices that have a linear response to increasing values of the measurand. ISO 11095 in turn relies on the premise that a set of data is linear and have a sufficient spread over the measurement range. EN 14181:2004 defines this spread in terms of percentages of the ELV. This standard defines two situations based on the extent of the spread of data, and specifies two mathematical methods which allow the test laboratory to calculate the calibration function. These two mathematical methods are defined as Method A and Method B, respectively.

In order to determine whether to use Method A or Method B, the test laboratory uses the SRM data. EN 14181:2004 requires that this data is initially converted to values at standard conditions.

- Method A: If the spread of SRM data, defined as the difference from the largest SRM value to the smallest SRM value, is at least 15 % of the ELV, then Method A shall be used. This method is a standard linear regression.
- Method B: If the data is clustered, and the difference between the largest and smallest SRM values in the cluster is less than 15 % of the ELV, then Method B shall be used. In simple terms, this method calculates a ratio of the average of the paired SRM and AMS values, and then forces the average through zero. This results in a calibration function for clustered data.

Regardless of whether Method A or Method B is used to calculate the calibration function, EN 14181:2004 still requires data to show that the AMS reads zero when the emissions are zero. However, the potential for large errors is much greater with Method B, if this step is not performed.

#### 7.3 Low-level clusters

There are typically three types of patterns of emissions from industrial plants; in addition to the patterns of data described above and assessed using Method A or Method B, the emissions can be very low, clustered at or near to zero. Low-level clusters are often the result of highly controlled processes.

However, EN 14181:2004 was not developed to determine and apply calibration functions when the emissions are low. In this respect, low emissions are defined as concentrations which are at or below the 95 % confidence interval of the daily average ELV. In such cases, the uncertainties of both the SRM and AMS can undermine the accuracy of the calibration function. Therefore, if the emissions are low, then it is advisable to contact the competent authority for guidance on an alternative procedure. Annex B shows some possible approaches for dealing with low-level clusters, although these are outside the scope of EN 14181:2004.

Careful planning of the QAL2 and AST measurements can identify the optimum time for the SRM measurements, when the emissions are at their highest or most varied.

Modern abatement plant means that particulate emissions are often very low, which presents particular challenges during the QAL2 and AST tests.

The levels of particulate can be low, but not so low that it is impossible to determine a calibration function using either Method A or Method B. In such cases, it can be beneficial to reduce the number of samples taken

for the calibration function, whilst increasing the time taken for each sample. For example, EN 13284-2 allows the number of samples for the AST to be reduced from five to four or even three samples. However, EN 13284-2 requires the sampling time for each sample to be increased. As a guideline, if five samples are taken over an accumulated time of 7,5 h, then three samples would also be taken over an equal period of time, i.e. each sample would take 2,5 h.

When the levels of particulate are extremely low, then the calibration function might not be reliable because of the relative uncertainties of measurements at low levels. In such cases, it is advisable to contact the competent authority for guidance. Annex B of this Technical Report shows some alternative approaches for dealing with very low emissions.

#### 7.4 Peripheral AMS measurements

EN 14181:2004 specifies requirements for peripheral measurements. These are determinants which need to be measured but do not have performance characteristics assigned to them within the Directives. In EN 14181:2004, peripheral measurements are:

- oxygen;
- moisture:
- temperature; and
- stack gas pressure.

EN 15267-3 includes performance criteria and test procedures for oxygen and moisture. Therefore performance requirements equivalent to those required to QAL1 can be applied to these determinants, even though where the Directives do not include uncertainty allowances for oxygen and moisture.

Ordinarily, it is not necessary to determine a calibration function for oxygen and moisture. If the AMS for the main determinants passes the variability test, then this means that the oxygen and moisture measurements are sufficiently accurate. However, it is good practice to determine a calibration function for at least oxygen-measuring AMS.

If the AMS fails the variability test initially, then EN 14181:2004 states that the test can be repeated using the SRM measurements for the peripheral determinants when converting the data to standard conditions for both the AMS and SRM measurements. If the AMS then passes the variability test, then this means that the peripheral measurements are not sufficiently accurate or precise. Initially, EN 14181 requires the operator of the industrial plant to investigate the causes of the failure, and then repair or replace the AMS for peripheral measurements. In such cases, it may be necessary to determine a calibration function for the AMS for the peripheral measurements. Also functional tests for peripheral AMS would determine whether the AMS are ready for the parallel reference tests within QAL2 and the AST.

#### 7.5 Establishing the calibration function and the test of variability

In carrying out the analysis of data, EN 14181 requires the following steps:

- tabulate the AMS and SRM data;
- express the SRM data and the AMS readings in the same conditions (i.e. either dry or humid, to the same temperature and pressure);
- plot the AMS data and SRM data together;
- assess whether there are any outliers and eliminate these; for example, if the AMS undergoes a regular zero and span operation, then such data will be invalid; Annex A shows a method for determining outliers;

- calculate the calibration function; as guidance, an indicator for a valid calibration function is a correlation coefficient of the regression line of at least  $R^2 = 0.90$ ;
- establish the valid calibration range;
- convert the data to calibrated and standardised values;
- carry out the variability test.

For an AMS measuring gas, the analogue signal recorded is converted into milligrams per cubic metre (mg/m³). No temperature correction is required to determine the calibration line or for the variability test. Depending on the type of device (in-situ or extractive) and how the sample is treated for the extractive AMS, concentration is expressed for dry or wet gas. If SRM and AMS concentrations are not expressed in the same moisture conditions, then the SRM values are converted to the conditions of the AMS to determine the calibration function, and all concentrations (SRM and AMS) are expressed at standard conditions (dry gas at 273 K, reference oxygen content) for the variability test.

For particulate AMS with beta-gauges, the result is given in dust concentration per normal cubic meter for wet gas. No temperature correction is required to determine the calibration function or for the variability test. If SRM and AMS concentrations are not expressed in the same moisture conditions, then the SRM values are converted to the conditions of the AMS to determine the calibration function, and all concentrations (SRM and AMS) are expressed for dry gas and in reference oxygen content for the variability test.

For particulate AMS with optical analyzers, the result is given in dust concentration in actual stack temperature and pressure conditions and for wet gas. If SRM and AMS concentrations are not expressed in the same temperature, pressure and moisture conditions, then the SRM values are converted to the conditions of the AMS to determine the calibration function, and the concentrations (SRM and AMS) are expressed in normal temperature and pressure conditions, for dry gas and in reference oxygen content for the variability test.

Some national regulators allow operators to determine the calibration function at standardized flue gas conditions (dry gas, 273 K, 101,3 kPa and at reference oxygen concentration). When using this approach, the peripheral measurements are included in the calibration function. However, this approach does not strictly comply with the procedure specified in EN 14181, which requires the AMS to be calibrated separately under the measurement conditions of the AMS.

If there are SRM measurements above the daily ELV, then the AMS can fail the variability test because of signal-proportional uncertainty contributions which can increase the uncertainty at larger values of the measured signal from the AMS. In such cases, one solution is to repeat the variability test excluding the data above the daily ELV. At least ten remaining data points are required for this procedure. Annex C shows the  $k_{\rm V}$  values for three to 30 data pairs.

#### 7.6 Data points outside the calibration range

#### 7.6.1 Extrapolating the calibration function using reference materials

If a calibration function does not extend as far as the ELV, then EN 14181:2004 allows the following procedures to extrapolate the calibration function:

- the calibration range of AMS is extended by 10 % of the highest SRM value to get the valid calibration range;
- the calibration function can be extrapolated further using reference materials, so long as the resulting data points are within the 95 % confidence interval about the calibration function (see Figure 5); however, EN 14181:2004 allows only a limited proportion of data points to exceed the valid calibration range derived during QAL2.

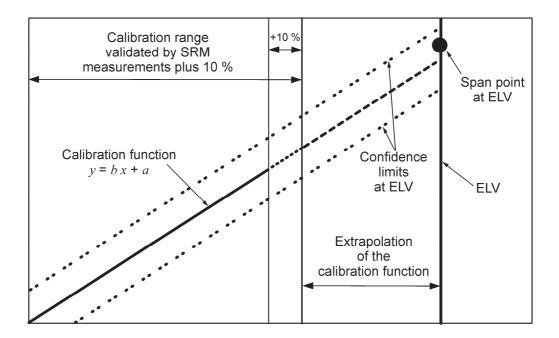


Figure 5 — Extrapolating the calibration range to the ELV

#### 7.6.2 Extending the calibration range using AST data

EN 14181:2004 allows the extension of the calibration range using AST data, subject to limiting conditions. If measurements from the AST verify the calibration function, and the highest SRM measurements are greater than the original calibration range, then the calibration range may be extended up to the value of the highest SRM measurement, but not more than 50 % of the ELV.

#### 7.7 Calibrating AMS for NO<sub>x</sub> and TOC

#### 7.7.1 Calibrating AMS for NO<sub>x</sub>

The Directives require operators to report emissions of oxides of nitrogen ( $NO_x$ ) as equivalent  $NO_2$ . However, AMS can measure and report  $NO_x$  in different ways, such as emissions of nitric oxide ( $NO_x$ ) in either milligrams per cubic metre ( $mg/m^3$ ), milliamperes ( $mA_x$ ) or millivolts ( $mV_x$ ), or emissions of both  $NO_x$  and  $NO_x$  in any of these output parameters, or as total  $NO_x$ . This means that in QAL2 and AST, EN 14181:2004 requires that test laboratories have to take into account the reported emissions of the AMS for  $NO_x$ .

For most applications, the emissions of  $NO_x$  are dominated by NO, so competent authorities could allow an operator to measure NO alone, and then use a conversion factor to report the measurements as  $NO_2$ . This conversion factor can be included in the calibration function. For example, if  $NO_x$  is made up of at least 95 % NO, then the value of  $NO_x$  is calculated from the NO measurements divided by 0,95.

Additionally, EN 14792 (SRM for  $NO_x$ ) specifies that test laboratories need to report the results as total  $NO_x$ , reported as  $NO_2$ . Therefore, when using EN 14792, the test laboratory has to take extra care in establishing exactly how the AMS both measures and reports emissions of  $NO_x$ , regardless of how the data acquisition and handling system reports  $NO_x$  emissions. This is because EN 14181:2004 requires the test laboratory to determine the calibration function for the measured component at the conditions measured by the AMS. Therefore, the test laboratory may need to convert the SRM results to AMS measuring conditions.

A QAL2 and AST procedure may therefore be made by comparing the AMS signal, measuring NO, with the SRM result, measuring  $NO_x$  or NO and  $NO_2$  individually, and calculate the corresponding  $NO_x$  calibration function.

For certain types of gas-turbines, the ratio of NO to  $NO_2$  can significantly change with the load, e.g. at full load 95 % of  $NO_x$  is NO and at a load of 25 % the NO is 20 % of the total  $NO_x$ .

If the AMS has a NO<sub>x</sub>-converter, then this requires a functional test to determine its continued efficiency. Such a test could be performed, for example, during the functional tests of QAL2 and AST.

#### 7.7.2 Calibrating AMS for TOC

According to the requirements of the Directives and EN 14181:2004, AMS for total organic compounds (TOC) are calibrated and verified using a SRM for TOC as specified in EN 12619. This SRM uses a flame ionisation detector to measure the response when carbon-hydrogen bonds are ionised in a flame. The emissions of organic compounds can vary in composition and proportion for any particular process, so it is ideal to measure the emissions over a wide range of concentrations and process conditions up to the ELV. In all other ways, the same principles which apply to other types of gas-monitoring AMS also apply to AMS which measure TOC, e.g. dealing with low levels of emissions, or extrapolating the calibration function.

## 7.8 Significant changes to operating conditions and fuels

Changes in operating conditions or fuels do not always require a second calibration function. Experience has shown that a single calibration function is often sufficient for a variety of operating conditions and fuels, provided that the AMS meets the uncertainty requirement. A second calibration function can be needed, for example, if the fuel is changed significantly.

A change of fuel is considered significant if it results in a change in the way that the AMS response (e.g. a change of cross-interference, particle-size distribution) or a change in the concentration profile in the stack, meaning that there is a need for a new calibration function for the applicable measured components.

It is advisable to contact the competent authority to determine whether the period of time using the alternative fuel is considered significant, and whether the change requires a second calibration function.

If operating conditions and fuels are changed after a QAL2, the significance for the calibration function and the variability can be checked by parallel measurements as described in the AST test. If this test shows that the calibration function is still valid, then no further action is needed. If the calibration function is no longer valid, then calibration functions for these different operating conditions and fuels need to be established.

#### 7.9 Significant changes to an AMS

If there is a change to the AMS, then a new calibration function is only required if the change is significant, and AMS response changes to the measured components. A significant change is defined as the following:

- a repair or replacement of a component or components of the AMS, where the repair or replacement could affect the calibration function;
- a replacement AMS, where the type is the same as the original AMS; or
- a replacement AMS, where the type differs from the original AMS.

In order to ensure that the AMS provides valid measured results after a significant change, EN 14181:2004 specifies that a QAL2 is required. This includes the functional tests. It is also important to note the QAL3 requirements specified in EN 14181:2004. However, it is also advisable to contact the competent authority in the first two cases above, as the competent authority may allow the following procedure:

- apply the existing calibration function for the original AMS;
- perform the procedures defined by the AST initially, including the functional tests;

- if the AST shows that the calibration function is still valid, then the competent authority may decide that a full QAL2 test is not required until the next scheduled QAL2;
- if the AST shows that the calibration function is not valid and the AMS requires a new calibration function, then a full QAL2 is required according to EN 14181:2004.

In the case of a replacement AMS which differs from the original type of AMS, a full QAL2 is required. EN 14181 also requires compliance with the QAL1 and QAL3 requirements when replacing AMS.

## 8 On-going surveillance and quality assurance of AMS (QAL3)

#### 8.1 The necessity for QAL3

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 deviations 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 deviations. However, the degree of these deviations can be acceptable. Therefore, QAL3 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 or random deviations, whether the
  deviations become too large, and to ignore the deviations if they lie between acceptable limits defined on
  the control charts.

Control charts require regular and ideally frequent measurements. Regular measurements at zero and reference points are the foundations of a QAL3 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.

The following subclauses describe the following:

- choosing control charts;
- zero and span measurements;
- setting parameters for control charts.

## 8.2 Choosing control charts

## 8.2.1 General

Any type of control chart, manual or automated, may be used according to EN 14181:2004. Different charts have different advantages and can be more or less complicated to use, depending on the type of chart chosen. EN 14181:2004 describes two types of chart, the simpler Shewhart charts and the more complicated CUSUM charts. This Technical Report also describes a third type of chart, the exponentially weighted moving average charts (EWMA).

Shewhart charts simply plot the readings and test them against multiples of  $s_{AMS}$ . Its advantage is the simplicity, its disadvantage is that the approach is not as sensitive as other approaches such as CUSUM or EWMA charts. Furthermore, Shewhart charts cannot distinguish between systematic errors and random errors. More complex charts, such as CUSUM or EWMA charts, do make this distinction. Such charts signify

either the requirement of readjustment of instrument amplification or zero point adjustment, and help operators identify the causes of either random or systematic errors.

#### 8.2.2 Shewhart charts and other simple control charts

Simple control charts such as Shewhart charts only indicate if the AMS has drifted or whether the precision has worsened. However, Shewhart charts do not distinguish between systematic deviations and loss of precision. However, the Shewhart chart method is simple to set up and understand, and it is well suited for manual procedures.

Annex D of this Technical Report shows an example of a Shewhart chart compared to an EWMA chart.

#### 8.2.3 EWMA chart

Compared with the Shewhart chart, the exponentially weighted moving average chart (EWMA) is more appropriate for early detection of small or medium-sized maladjustments, it is simpler and easier to use than the CUSUM chart, it is easier to set up and it keeps the graphical format of the Shewhart individuals 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 D of this Technical Report shows an example of a Shewhart chart compared to an EWMA chart.

#### 8.2.4 CUSUM charts

CUSUM charts are more complex to understand and set up. However, the benefit of this approach is that CUSUM charts can help distinguish between drift, and loss of precision. This means that the approach is better for predictive maintenance and for planning of the service work. It is mathematically more complex, and therefore mostly suited for automatic systems run on a personal computer. If combined with automatic logging of data from the instruments the whole process can be automated with considerable reduction in costs as a consequent.

Annex C of EN 14181:2004 describes in detail the procedure for CUSUM.

#### 8.2.5 Built-in methods

An alternative is to use an instrument built-in method, although it is advisable to seek guidance from the competent authority on the acceptability of this approach. Many instruments have a built-in check of zero and span points, and give alarm, if set limits are surpassed. The acceptability of this approach can depend on whether the competent authority requires the AMS to be able to provide data for zero and span values on request.

#### 8.3 Zero and span measurements

#### 8.3.1 General

QAL3 requires the AMS to have a means to perform zero and span measurements. If this is not possible with an AMS, then EN 14181:2004 requires operator of the industrial operator to describe a procedure which shows whether the AMS is stable, or whether there has been any drift. In other words, the manufacturer of the AMS needs to develop a surrogate zero and span procedure, in order to assist the plant operator to comply with the QAL3 requirements of EN 14181:2004.

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 QAL2.

Some AMS have been designed to perform automatic zero and span measurements. In order to fulfil the QAL3 requirements of EN 14181:2004, the data from the zero and span measurements needs to be available to the operator.

#### 8.3.2 Frequency of zero and span measurements

According to EN 14181:2004, operators have to plot zero and span data using control charts. The application of control charts require 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 may perform more frequent zero and span checks.

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

The maximum allowable interval between zero and span measurements is known as the maintenance interval. The maintenance interval is determined during performance testing for approval to the requirements of standards such as EN 15267-3 or for the schemes and programmes which preceded EN 15267-3. In most AMS, the maintenance interval is typically between eight days and one month. Some AMS have much longer maintenance intervals; for example, from three to six 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. On the other hand, infrequent zero and span measurements mean that there is a higher risk of an operator not detecting a systematic error in the AMS, or an increase in random errors.

EN 14181:2004 requires plant operators to archive the data from zero and span measurements by use of control charts. However, if an AMS does not require, for example, span measurements more frequently than every six months, then this does not generate enough data for produce control charts. Therefore, when an AMS has a long maintenance interval, the operator can do the following:

- perform span measurements more frequently, although this negates the point of having an AMS with a long maintenance interval;
- perform span measurements at the maintenance interval, but perform zero measurements much more frequently.

The advantage of frequent zero measurements is that they take much less time than span measurements, whilst zero measurements also detect drift in the AMS.

The CUSUM procedure defined in Annex C of EN 14181:2004 requires in principle weekly zero and span checks.

#### 8.3.3 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 can be used a zero gas. If the sampling line serving the extractive AMS is relatively long, then the zero and span procedures can be time consuming and consume relatively large amounts of gas.
- Use of gas-filled cuvettes within the AMS: The drawback of cuvettes is that they do not allow a check of the complete AMS, i.e. cuvettes 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 cuvettes for zero and span measurements.

#### 8.3.4 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, which are the following:

- 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, which are that (i) the tubes require a large volume of gas for each test, and (ii) 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 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.

#### 8.3.5 Particulate-monitoring AMS

There are several varieties of particulate-monitoring AMS, using a variety of techniques which respond to variations in concentrations of particulate matter. Regardless of the technique employed, zero and span readings are typically carried out using surrogates. These surrogates can consist of optical filters with a varying density, combined with mirrors.

Regardless of the technique employed in the particulate-monitoring AMS, EN 14181:2004 requires the AMS to have a means for testing the actual zero and span drift of the entire AMS, or a means of checking the AMS in an alternative manner, which also reflects the actual zero and span drift. Regardless of the method used, it is good practice for an operator to have a detailed description of the zero and span checks of the AMS. An AMS manufacturer can provide such a description and instructions on how to perform the checks.

#### 8.3.6 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 of QAL3. 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 according to EN 15267-3.

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 through 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. If a plant operator has such a system, then competent authorities may advise the operator if it is also necessary to plot control charts using zero and span data.

#### 8.3.7 Adjustments to span readings

Once the AMS has been calibrated and passed the QAL2 tests, the initial span readings are used to set the baseline for the control charts. In the past, it has been usual to make adjustments to the span setting if span checks show a difference between the original span level, and the most recent span check. However, this procedure is not recommended, unless the span readings are consistently outside the action limits set within the control charts. In other words, if the span readings are within the action and alarm limits on the control charts, then no action is required.

#### 8.3.8 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 the one bottle is changed for another. Hence it is important to differentiate and 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 five 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 five 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 five 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.

#### 8.4 Setting parameters for control charts

#### 8.4.1 Setting limits for control charts, except CUSUM charts

The control charts required for QAL3 are a means of determining whether any zero and span readings are true outliers, rather than acceptable random variations. As the standard deviation  $s_{\rm AMS}$  determines the positions of the warning and alarm limits, the value of  $s_{\rm AMS}$  is a critical part of the control chart. In statistical terms, the purpose of  $s_{\rm AMS}$  is to determine if there is a significant probability that a zero or span measurement is different from the target value. Therefore,  $s_{\rm AMS}$  is usually chosen to represent one standard deviation of the acceptable variations in zero and span readings. Multiples of  $s_{\rm AMS}$  can be chosen to represent statistical confidence intervals for the variations in zero and span readings.

#### 8.4.2 Calculation of $s_{AMS}$ using performance data

7.3 of EN 14181:2004 specifies a procedure for calculating  $s_{\text{AMS}}$ , whilst Annex F of EN 14181:2004 shows an example of this calculation. The calculation includes the influence factors which are likely to have the most significant influence on zero and span results.

This approach requires the following:

- understanding of the main influence factors which can cause random variations in zero and span readings;
- performance data for the AMS for each of the influence factors in the calculation.

The performance data are typically available from test reports for AMS tested according to EN 15267-3, or from test reports for performance testing schemes which preceded EN 15267-3. Alternatively, the plant operator can use the performance criteria for testing according to EN 15267-3.

Whilst this calculation uses performance data derived from QAL1, plant operators need to consider the actual conditions at the plant. For example, testing to EN 15267-3 for QAL1 tests the influence of ambient temperature on the AMS over defined ranges 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  $s_{\rm AMS}$ .

A calculation of  $s_{AMS}$  is an uncertainty calculation and therefore an estimate. Over time, a plant operator could find that the value of  $s_{AMS}$  is set too high or too low, and therefore needs optimising. Therefore, setting control chart action limits is an iterative process. In some CEN Member states, a pragmatic solution has been found to apply a drift specification based on the specifications from performance testing. Experience has shown that this approach takes into account the uncertainty factors which can influence drift and changes in precision.

For example, an AMS can have easily met the requirements of EN 15267-3 during performance testing. If so, then the value of  $s_{\text{AMS}}$  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 in the Directives.

# **Annex A** (informative)

# An example of a procedure for determining outliers

Grubbs's test is a simple test and applies when the standard deviation and mean of a sample of data points is known. The procedure relies on the probability that valid data is likely to lie within two standard deviations of the mean, and applies Equation (A.1).

$$Z_i = \frac{\left| \overline{D} - D_i \right|}{s_D} \tag{A.1}$$

Grubbs's test is applied to the data in Table A.1. The difference  $D_i$  between the pairs of SRM and AMS data is calculated. The mean and standard deviations of the differences are then used to determine the Z value required by the Grubbs's test for each data pair.

Table A.1 — Pairs of data assessed using the Grubbs's outlier test

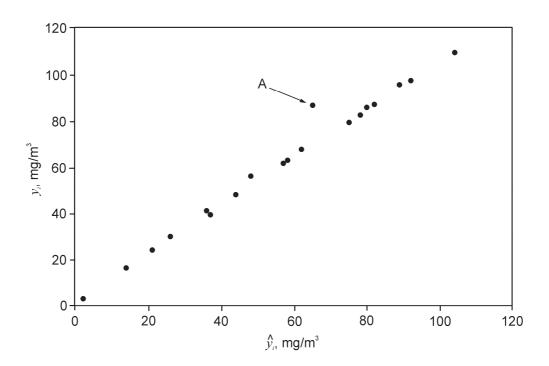
Data pair	AMS value	SRM value	Difference	Z value
i	$\hat{\mathcal{Y}}_i$	$\mathcal{Y}_i$	$D_i$	$Z_i$
	mg/m³	mg/m³	mg/m³	
1	21,0	24,1	3,1	0,62
2	36,0	41,2	5,2	0,12
3	37,0	39,6	2,6	0,72
4	2,0	3,2	1,2	1,05
5	48,0	56,3	8,3	- 0,60
6	57,0	62,0	5,0	0,16
7	58,0	63,2	5,2	0,12
8	78,0	82,6	4,6	0,26
9	65,0	87,0	22,0	- 3,79
10	89,0	96,2	7,2	- 0,35
11	82,0	87,5	5,5	0,05
12	75,0	79,8	4,8	0,21
14	44,0	47,9	3,9	0,42
13	62,0	67,9	5,9	- 0,05
14	92,0	98,1	6,1	- 0,09
15	26,0	29,8	3,8	0,44
16	14,0	16,5	2,5	0,74
17	80,0	86,0	6,0	- 0,07
18	104,0	110,2	6,2	- 0,12
		Mean	5,7	
		Standard deviation	4,3	

The  $Z_i$  value is then compared to a critical Z value (see Table A.2). If the  $Z_i$  value for any pair of data is more than the critical value, then there is a 95 % probability that the data point is an outlier, and the data point can be eliminated.

Table A.2 — Critical Z values

Number of data pairs	Critical Z value		
3	1,15		
4	1,48		
5	1,71		
6	1,89		
7	2,02		
8	2,13		
9	2,21		
10	2,29		
11	2,34		
12	2,41		
13	2,46		
14	2,51		
15	2,55		
16	2,59		
17	2,62		
18	2,65		
19	2,68		
20	2,71		
21	2,73		
22	2,76		
23	2,78		
24	2,8		
25	2,82		
26	2,84		
27	2,86		
28	2,88		
29	2,89		
30	2,91		

It can be seen from Grubbs's test that data point number nine is an outlier, because the Z value is above the critical Z value. This can also be seen by plotting the data (see Figure A.1).



# Key

- A outlier
- $\hat{y}_i$  AMS measured value, in milligrams per cubic metre (mg/m³)
- $y_i$  SRM measured value, in milligrams per cubic metre (mg/m³)

Figure A.1 — Raw data plotted showing the outlier

# Annex B

(informative)

# Alternative approaches for dealing with low-level clusters of emissions

## **B.1 Background**

EN 14181:2004 was developed for calibrating and verifying AMS when the emissions are high enough to allow the calculation of a valid and reliable calibration function. The calibration function is considered as valid and reliable when the uncertainty of the measurements is less than the measurements themselves. However, if the emissions are very low, then the uncertainty of the measurements can be too large to derive a reliable calibration function. EN 14181:2004 does not include provisions for such situations. Therefore, if the emissions are very low, then it is advisable to contact the competent authority for guidance on an appropriate procedure. The following subclauses describe some approaches which have been applied by some competent authorities, although there are no specified rules as to which procedure shall be followed.

#### **B.2** Definition of low emissions

The emissions can be considered as low, if the highest result from the SRM is not more than the 95 % confidence interval of the daily average emission limit value, as specified in the Directives.

The uncertainty allowance for total organic carbon (TOC) specified in the Directive for the incineration of waste is 30 % of the daily average ELV. The daily average ELV is 10 mg/m³. Therefore, the 95 % confidence interval is 3 mg/m³. When examining the typical emissions from the industrial plant over a year, for example, the emissions are regarded as low if the highest value does not exceed 3 mg/m³. In other words, all of the emissions values are within the uncertainty allowance.

## **B.3 Alternative procedures for gas-monitoring AMS**

The alternative procedures for gas-monitoring AMS include the following four options:

- Option 1: Performing a QAL2 as specified in EN 14181:2004, applying Method B, and accepting that the uncertainty of the measurements can introduce a significant calibration error at the ELV. However, this error will not be significant if the emissions remain well below the ELV.
- Option 2: Performing a QAL2 as specified in EN 14181:2004, but increasing the sampling time for each measurement using SRMs, and therefore reducing the number of samples. This will decrease the uncertainty of each measurement. Method B is applied to calculate a calibration function, although there can still be a significant error at the ELV. However, this error will not be significant if the emissions remain well below the ELV.
- Option 3: Performing a limited number of measurements using the SRM, perhaps over one day instead of at least three days. 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 ELV.
- Option 4: As with Option 3, but the SRM data is combined with the data produced from using reference materials, in order to derive a calibration function.

### **B.4 Particulate Monitors**

The same four options can apply to calibrating particulate monitors. However, there are not any equivalent surrogates for particulate monitors, whereas test gases can be used as surrogates for gas-monitoring AMS. Therefore, an alternative approach may be needed instead of Options 3 and 4 above.

When the emissions are very low, the absence of surrogates mean that it may be impossible to calibrate particulate monitoring AMS, even with a high uncertainty. Some competent authorities have therefore applied the following two procedures, in addition to Options 1 and 2 above.

- A reduced number of SRM measurements to confirm the emissions are low: A reduced number of SRM measurements are taken over longer periods of time. The SRM measurements are then used to confirm that the emissions are as low as the AMS shows. The AMS are then set up to respond to process changes which would indicate an increase in emissions, although the outputs from the AMS are considered as qualitative rather than quantitative.
- Using SRM to confirm compliance, followed by an alternative calibration procedure: The procedure is the same as above, except that the AMS is calibrated using an alternative procedure, which in turn depends on the AMS technique. The following sub-section describes three alterative methods for calibration.

Specific examples for particulate monitors include:

- Techniques directly dealing with mass: AMS using techniques which measure particulate mass either directly or by inference – such as beta-ray monitors or micro-balance systems – can be calibrated using known masses of particulate matter.
- Optical transmission monitors: Based on optical physics, theoretical calibration functions can be calculated for optical transmission monitors. The accuracy of this approach depends on an accurate estimation of the average grain size. It can be possible that the manufacturer of the AMS can supply that information. The following Equation (B.1) can be used to calculate a calibration function:

$$C = 833 E_{\text{otm}} \frac{\mu}{L_{\text{mp}}}$$
 (B.1)

where

C is the mass concentration in milligrams per cubic metre (mg/m $^3$ );

 $E_{\text{otm}}$  is the extinction of the optical transmission monitor;

 $\mu$  is the average diameter of the grains in the stack in micrometres ( $\mu$ m);

 $L_{mp}$  is the length of the measurement path in metres (m).

Scattered-light monitors: It is not yet possible to calculate a theoretical calibration function for AMS which use this technique. However, an alternative is to apply a calibration function from a similar plant. Again is the grain size is critical, and since this is not identical from plant to plant, the calibration curve will again only be indicative, and only be used reliably as long as the concentration is low and steady. A test laboratory might be able to use its experience from other plants with the same type of AMS and a similar process. However, the uncertainties can still be high and under such conditions, it is advisable to regard the measurements from the AMS as qualitative, rather than quantitative monitor.

# **Annex C** (informative)

# $k_{\rm v}$ values

Table C.1 —  $k_v$  values

Number of parallel measurements	$k_{v}(N)$	$t_{0,95}(N-1)$
3	0,832 6	2,920
4	0,888 1	2,353
5	0,916 1	2,132
6	0,932 9	2,015
7	0,944 1	1,943
8	0,952 1	1,895
9	0,958 1	1,860
10	0,962 9	1,833
11	0,966 5	1,812
12	0,969 5	1,796
13	0,972 1	1,782
14	0,974 2	1,771
15	0,976 1	1,761
16	0,977 7	1,753
17	0,979 1	1,746
18	0,980 3	1,740
19	0,981 4	1,734
20	0,982 4	1,729
25	0,986 1	1,711
30	0,988 5	1,701

Ordinarily between three and eight pairs of data are required for an AST, and at least 15 measurements for a QAL2. If there are more than eight pairs of data for an AST, or more than 30 for a QAL2, then it is advisable to choose the applicable  $k_v$  and  $t_{0.95}$  values for the next lowest value. For example, if there are 37 pairs of data points, then  $k_v$  and  $t_{0.95}$  values for 30 data pairs can be safely used.

# Annex D (informative)

## **Shewhart and EWMA control charts**

#### D.1 Shewhart control charts

Shewhart charts are the simplest types of control chart, and can either plot the recorded span value, or the actual difference between the original span value and subsequent span values, i.e. the drift, whether this drift consists of random variations, or systematic changes. Shewhart control charts contain a centre line and control limits (lower and upper), as follows:

- the chart centre line which is equal to the target value  $m_0$ ;
- action limits at  $m_0 \pm 2 s_{AMS}$ .

Once the limits for the control charts have been determined (one for zero and one for the span of the reference material, using the procedure described in 7.3 of EN 14181:2004, together with the example in Annex F), the results from the AMS readings are indicated on each chart to detect the drifts and/or changes in accuracy requiring the operator to take action. Such action can include AMS adjustment, or maintenance and potential elimination of the AMS measurements since the previous QAL3 test.

When using Shewhart control charts, the decision is taken based on the last result. This chart rapidly detects drift which is large, but is less efficient at detecting small amounts of drift. A drift of two standard deviations  $(2 s_{AMS})$ , for example, has only a 16 % chance of being detected in a single check.

On such a chart, the decision rules for taking action on the AMS could be as follows (in decreasing order of importance):

- three consecutive data points are beyond one of the action limits;
- four out of five consecutive points are beyond the action limits;
- eight consecutive points are on the same side of the centre line;
- six consecutive points are either increasing or decreasing (a trend).

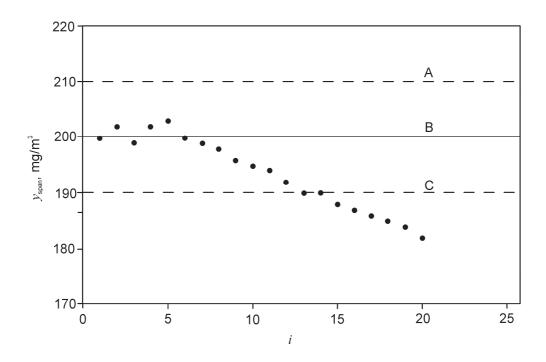
The following example shows a series of span measurements for NO, with 20 checks for the span value at regular intervals (see Table D.1). Absolute values and relative values with the target value are shown, whilst in this example,  $s_{AMS}$  would be calculated using the procedure in 7.3 of EN 14181:2004. For the sake of example, the value of  $s_{AMS}$  is given as 5 mg/m<sup>3</sup>.

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

Span-point number	Span value	Deviation from the baseline
	mg/m³	mg/m³
1	200	0
2	202	2
3	199	<b>–</b> 1
4	202	2
5	203	3
6	200	0
7	199	<b>–</b> 1
8	198	-2
9	196	-4
10	195	<b>-</b> 5
11	194	<b>-</b> 6
12	192	-8
13	190	<b>– 10</b>
14	190	<b>– 10</b>
15	188	<b>– 12</b>
16	187	<b>– 13</b>
17	186	<b>– 14</b>
18	185	<b>–</b> 15
19	184	<b>–</b> 16
20	182	<b>–</b> 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 ten 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.

CUSUM charts, whilst difficult to set up, would be more sensitive to both changes in precision, and systematic changes.



#### Key

 $y_{\rm span}$  span value, in milligrams per cubic metre (mg/m³)

*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

## D.2 Exponentially weighted moving average charts

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

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

Exponential weighted moving average (EWMA) charts or CUSUM improve the efficiency of detection by using results of measurements previous to the last check, whereas Shewhart charts do not. Annex C of EN 14181:2004 describes the procedure for setting up CUSUM charts. This Technical Report 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;

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- is simpler and easier to use than the CUSUM chart;
- is easier to set up and keeps the graphical format of the Shewhart chart;
- implements only one decision rule;
- reduces the risks due to the natural variability of the process and helps to avoid useless questioning as to the random changes in this process.

This chart requires three preparatory steps:

- selecting the decision criteria: n = 1;  $\delta \sqrt{n} = 1$ ;  $\sigma_0 = s_{AMS}$ ; ARL, maximum ARL; L and  $\lambda$ ;
- determining weighting of the past values with the last reading:

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

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;

and  $0 < \lambda < 1$ ;

— determining the control limits:

$$UCL = m_0 + L \frac{\sigma_0}{\sqrt{n}} \sqrt{\frac{\lambda}{2 - \lambda}}$$
 (D.2)

$$LCL = m_0 - L \frac{\sigma_0}{\sqrt{n}} \sqrt{\frac{\lambda}{2 - \lambda}}$$
 (D.3)

The control limit value L and the smoothing parameter  $\lambda$  are selected so as to obtain an Average Run Length (ARL) that is set as a quality objective. This ARL is the average number of successive checks required to detect a  $\delta$  maladjustment that can be a false alarm if the process is not maladjusted. The Maximum Average Run Length (Max ARL) is the maximum number of successive checks required to detect a  $\delta$  drift, if the process is maladjusted.

If  $\lambda$  nears 0, this will take the past more into account and detect small drifts, but sudden major drifts are less easily detected.

If  $\lambda$  nears 1, this will take the past less into account and responsiveness to sudden major drifts are greater, but small drifts will be less easily detected.

If a slow drift is expected, select a  $\lambda$  near 0,25, but if sudden changes are expected then select  $\lambda$  near 0,5.

To simplify the choice of EWMA chart decision criteria, where n=1 and  $\sigma_0=s_{\rm AMS}$ , let  $\lambda=0.35$  and L=2.944 5, which produces an ARL of 11,7 (12) and a maximum ARL of 29. In comparison, the Shewhart chart with  $\lambda=1$  and L=3 produces an ARL of 370 for  $\delta\sqrt{n}=0$ , or 44 for a  $\delta\sqrt{n}=1$  with a maximum ARL of 130.

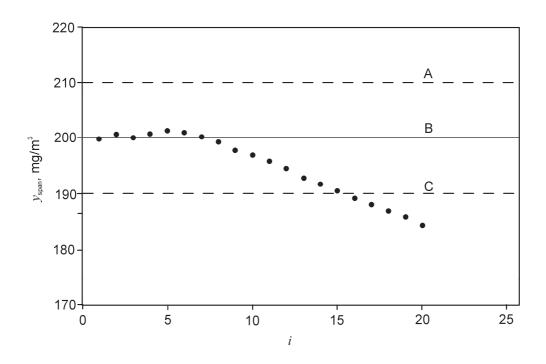
This means that for the same set of data from an AMS, a Shewhart chart will require 44 successive checks to detect a problem compared with only 12 for the EWMA chart. An estimated 130 checks is required for the Shewhart chart compared with a maximum of 29 for the EWMA chart.

Table D.2 shows the values of the span checks which were used in the example of a Shewhart chart, together with the weighted, smoothed values.

Table D.2 — Raw and calculated EWMA data

Span check number	Span value	EWMA value
number	mg/m³	mg/m³
1	200	200,0
2	202	200,7
3	199	200,2
4	202	200,8
5	203	201,5
6	200	201,0
7	199	200,3
8	198	199,5
9	196	198,0
10	195	197,1
11	194	196,0
12	192	194,6
13	190	193,0
14	190	191,9
15	188	190,6
16	187	189,3
17	186	188,2
18	185	187,1
19	184	186,0
20	182	184,6

Figure D.2 shows the EWMA chart. When compared to the Shewhart chart, acceptable random variations are smoothed, whilst the systematic changes are much clearer to see.



# Key

 $y_{\rm span}~$  span value, in milligrams per cubic metre (mg/m³)

i number of span check, with i = 1 to n

A upper action limit

B baseline

C lower action limit

Figure D.2 — Example of an EWMA chart

# **Bibliography**

This Technical Report cites, by dated or undated reference, provisions from other publications. These references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this Technical Report only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies.

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