INTERNATIONAL STANDARD

ISO 9169

Second edition
2006-07-01

Air quality — Definition and determination of performance characteristics of an automatic measuring system

Qualité de l'air — Définition et détermination de caractéristiques de performance d'un système automatique de mesurage

Reference number
ISO 9169:2006(E)

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Contents

Page

Foreword ... iv
Introduction ... v
1 Scope ... 1
2 Terms and definitions ... 1
2.1 General terms ... 1
2.2 Performance characteristics ... 5
3 Symbols and abbreviated terms ... 7
4 Terms of reference of the test programme ... 9
5 Performance characteristics ... 10
5.1 Basic requirements ... 10
5.2 Performance characteristics under laboratory conditions ... 11
5.3 Performance characteristics under field conditions ... 11
6 Test methods ... 11
6.1 General requirements ... 11
6.2 Requirements on the testing laboratory ... 12
6.3 Response time and minimum averaging time under stable laboratory conditions ... 12
6.4 Repeatability, lack of fit and limit of detection under stable laboratory conditions ... 14
6.5 Repeatability of the calibration method specified in the terms of reference under stable laboratory conditions ... 20
6.6 Drift under stable laboratory conditions ... 20
6.7 Sensitivity coefficients of interferent influence quantities under stable laboratory conditions ... 21
6.8 Sensitivity to external influence quantities under stable laboratory conditions ... 22
6.9 Standard deviation of paired measurements under field conditions ... 23
6.10 Drift under field conditions ... 24
6.11 Availability under field conditions ... 24
7 Test report ... 24
Annex A (informative) Examples ... 26
Bibliography ... 31

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.

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The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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.

ISO 9169 was prepared by Technical Committee ISO/TC 146, *Air quality*, Subcommittee SC 4, *General aspects*.

This second edition cancels and replaces the first edition (ISO 9169:1994), of which it constitutes a technical revision, and ISO 6879:1995.

Introduction

In this International Standard, automatic air quality measuring systems are considered as *black boxes* operated according to specified procedures as described in the terms of reference given by the client to the laboratory performing the tests aiming at determining performance characteristics selected by the client for each automatic measuring system.

This International Standard specifies definitions and methods to determine performance characteristics of automatic air quality measuring systems. This is done for most performance characteristics under steady laboratory conditions so as to have available data on clearly defined characteristics, based on specified conditions that can be adjusted and maintained in laboratory. This is also done under field conditions for a few performance characteristics for which field testing provide relevant additional information.

Air quality — Definition and determination of performance characteristics of an automatic measuring system

1 Scope

This International Standard provides definitions and specifies methods to determine performance characteristics of an identified automatic air quality measuring system. Tests are carried out under stable laboratory conditions or field conditions. The automatic measuring system is considered as a *black box* operated according to specified procedures.

This International Standard applies to measuring systems for which the following information is available:

— a description of the automatic measuring system providing the result of measurement in the physical unit of the measurand;

— operating procedures of the automatic measuring system including, where appropriate, the procedures of routine adjustment, routine verification and calibration;

— terms of reference for the test program specifying the client requirements and test conditions.

This International Standard applies to measuring systems for which it is possible to apply several reference materials with accepted values with known uncertainty for the measurand, within the range of application.

This International Standard does not specify the number of automatic measuring systems to be tested.

NOTE 1 The number of automatic measuring systems is specified by the client in the terms of reference.

NOTE 2 The list of performance characteristics in this document is limited. Additional performance characteristics can be specified by the client in the terms of reference, if appropriate.

2 Terms and definitions

For the purpose of this document, the following terms and definitions apply. Definitions taken from VIM[1] are generally kept identical. Some definitions have been adapted from VIM to take into account the specific wordings of the present International Standard.

2.1 General terms

2.1.1
measuring system
complete set of measuring instruments and other equipment with operating procedures to carry out specified air quality measurements

NOTE 1 Adapted from VIM:1993, 4.5.

NOTE 2 In general, a measuring system encompasses the different steps of the measurement process, such as taking the sample, the analytical quantification, etc.

2.1.2
automatic measuring system
AMS
measuring system interacting with the air under investigation, returning an output signal proportional to the physical unit of the measurand in unattended operation

NOTE The air under investigation includes, e.g. ambient air and emissions.

2.1.3
continuous automatic measuring system
automatic measuring system providing a continuous signal upon continuous interaction with the air mass under investigation

2.1.4
discontinuous automatic measuring system
automatic measuring system providing a series of discrete signals

NOTE Each discrete signal corresponds to the averaging time for field operation specified in the terms of reference.

2.1.5
adjustment
⟨automatic measuring system⟩ operation of bringing an automatic measuring system into a state of performance suitable for its use

NOTE 1 Adapted from VIM:1993, 4.30.

NOTE 2 Adjustment may be automatic, semi-automatic or manual.

2.1.6
primary result of measurement
result of measurement produced by an automatic measuring system measuring the measurand over the shortest period of time for which valid measurements can be obtained and used by the automatic measuring system to calculate the result of measurement over the specified averaging period for routine field operations of the automatic measurement system

NOTE For continuous automatic measurement systems, primary results of measurement are typically obtained for time periods of 1 s to 100 s, while the typical averaging time is 1 h in ambient air measurements and 30 min in emission measurements. For discontinuous automatic measuring systems, one primary measurement result is typically obtained for a cycle of a few minutes.

2.1.7
time interval for the primary measurement result
shortest period of time for which valid measurements can be obtained and used by the automatic measuring system to calculate the measurement result over the specified averaging period during routine functioning of the automatic measurement system

2.1.8
averaging time
minimum time interval equal to a stated multiple of the response time

NOTE See 6.3.1.

2.1.9
averaging time for field operation
time interval used by the automatic measuring system to produce routine results of measurement under normal (or envisaged) field operations

NOTE 1 Examples of averaging time for field operations are half an hour for emission measurements and one hour for ambient air measurements.

NOTE 2 The averaging time for field operation may be too long to be used during laboratory tests. Therefore, an **averaging time for laboratory test** (2.1.10) is defined and specified.

2.1.10
averaging time for laboratory test
time interval used for laboratory test and specified in such a way that

— the duration of the test is limited to minimize the possible drift effect during the test as well as the cost of the test;

— all conditions and influences may be considered as equal under steady laboratory conditions (e.g. insignificant drift effects);

— the number of primary results of measurement collected over the averaging time is equal to the number of primary results of measurement collected over the *intended* averaging time for routine field operations.

NOTE See 6.4.1.

2.1.11
measurand
particular quantity subject to measurement

[VIM:1993, 2.6]

NOTE In the field of air quality, the measurand is, e.g. the mass concentration of particulate matter or SO_2 in air.

2.1.12
interferent
interfering substance
substance present in the air mass under investigation, other than the measurand, that affects the response

2.1.13
influence quantity
quantity that is not the measurand but that affects the result of measurement (VIM:1993, 2.7), either an interferent influence quantity (i.e. the concentration of a substance in the air under investigation that is not the measurand), or an external influence quantity (i.e. a quantity that is not the measurand nor the concentration of a substance in the air mass under investigation)

EXAMPLE Examples include:

— presence of interfering gases in the flue gas matrix (interferent influence quantity);

— temperature of the surrounding air (external influence quantity);

— atmospheric pressure (external influence quantity); and

— pressure of the gas sample (external influence quantity).

2.1.14
reference material
RM
material or substance for which one or more properties are sufficiently homogeneous and well established to be used for the calibration and/or the validation of a measuring system

NOTE 1 Adapted from VIM:1993, 6.13 and ISO 11095:1996, 3.1.

NOTE 2 A reference material can be a pure or mixed gas, liquid or solid.

2.1.15
certified reference material
reference material, accompanied by a certificate, one or more of whose property values are certified by a procedure which establishes traceability to an accurate realization of the unit in which the property values are expressed, and for which each certified value is accompanied by an uncertainty at a stated level of confidence

[VIM:1993, 6.14]

2.1.16
basic state
specific state of an automatic measuring system for use as a base for the evaluation of actual states of the automatic measuring system

NOTE 1 Adapted from ISO 11843-1:1997, 2.

NOTE 2 The use of a zero reference gas may establish the basic state. Very often in the air quality field, "zero reference gas" is simply called "zero gas".

2.1.17
span level
level of concentration in the upper range of testing

NOTE Usually, span level is at 80 % of the intended upper limit of measurement.

2.1.18
calibration
set of operations that establish, under specified conditions, the relationship between the output of the automatic measuring system and the corresponding value given by the applicable reference

NOTE 1 Adapted from VIM:1993, 6.11 and ISO 11095:1996, Clause 4.

NOTE 2 The result of a calibration permits either the assignment of values of measurands to the indications or the determination of corrections with respect to indications.

NOTE 3 A calibration can also provide other metrological properties such as the effect of influence quantities.

NOTE 4 The result of a calibration should be recorded in a document, sometimes called a calibration certificate or a calibration report.

NOTE 5 Calibration as defined here is different from a simple check, i.e. checking without any adjustment that the automatic measuring system is still operating in the specified range. It is also different from a routine adjustment.

NOTE 6 The applicable reference can be a reference material (in the case of automatic ambient air quality measuring systems) or a standard reference method (in the case of automatic emission measuring systems).

2.1.19
expanded uncertainty
quantity defining an interval about the result of a measurement that may be expected to encompass a large fraction of the distribution of values that could reasonably be attributed to the measurand

[GUM:1995, 2.3.5]

NOTE The fraction may be viewed as the coverage probability or level of confidence of the interval.

2.1.20
linearity
maximum deviation between a linear calibration curve and the true value of the measurand, evaluated in practice as the maximum lack of fit within the measuring range

NOTE See 2.2.9.

2.1.21
memory effect
effect of previous values of the measurand on the current measurement results

2.1.22
parallel measurements
measurements by different measuring systems with sampling the same air over the same time period.

2.2 Performance characteristics

2.2.1
fall time
by convention, time taken for the output signal to pass from 90 % to 10 % of the initial output signal produced by a reference material applied to the automatic measuring system, when the application of this reference material is abruptly terminated to put the automatic measuring system in the basic state

NOTE For instruments where transient oscillations occur in approach to the final output signal, the 10 % of the initial output signal is considered as reached when the oscillations in the vicinity of the final output signal fall to less than 10 % of the initial output signal.

2.2.2
lag time
by convention, time taken for the output signal to reach 10 % of the final change in the output signal when a step function is applied by applying a reference material to the automatic measuring system initially in the basic state

2.2.3
rise time
by convention, time taken for the output signal to pass from 10 % to 90 % of the final change in the output signal when a reference material is abruptly applied to the automatic measuring system initially in the basic state.

NOTE For instruments where transient oscillations occur in approach to the final output signal, 90 % of the final change is considered as reached when the oscillations fall to less than 10 % of the final change in the output signal

2.2.4
response time
time interval between the instant when a stimulus is subjected to a specified abrupt change and the instant when the response reaches and remains within specified limits around its final stable value (VIM:1993, 5.17), determined as the sum of the lag time and the rise time in the rising mode, and the sum of the lag time and the fall time in the falling mode

2.2.5
repeatability
ability of an automatic measuring system to provide closely similar indications for repeated applications of the same measurand under the same conditions of measurement

NOTE 1 Adapted from VIM:1993, 5.27.

NOTE 2 These conditions include:

- same measurement procedure;
- same measuring equipment used under the same conditions;
- same location;
- repetition over a short period of time.

NOTE 3 Repeatability can be expressed quantitatively in terms of the dispersion characteristics of the measurement results of an automatic measuring system.

NOTE 4 The repeatability conditions selected in this International Standard are specified in Clause 6. These specifications are aimed at providing the user with an evaluation of the maximum difference that can be found, with 95 % statistical confidence, between two measurement results obtained:

— from the same automatic measuring system;

— operated according to the same operating procedures;

— at the same measurement location and under the conditions prevailing at this location (either laboratory or field);

— during a period of time short enough not to be sensitive to drift effects;

— at different times during the period of unattended operation.

2.2.6
reproducibility
closeness of the agreement between the results of measurements of the same measurand carried out under changed conditions of measurement

NOTE 1 Adapted from VIM:1993, 3.7.

NOTE 2 The changed conditions may include the measuring system but not the measurement procedure.

NOTE 3 Reproducibility may be expressed quantitatively in terms of the dispersion characteristics of the measurements results of an automatic measuring system.

NOTE 4 The reproducibility conditions selected in this International Standard are specified in Clause 6. These specifications are aimed at providing the user with an evaluation of the maximum difference that may be found, with 95 % statistical confidence, between two measurement results obtained:

— from two automatic measuring systems meeting the same specification;

— operated according to the same specified operating procedures and each system being calibrated with its own reference;

— at the same measurement location and under the conditions prevailing at this location (either laboratory or field);

— by parallel measurements during the same period of time;

— spread over the period of unattended operation.

2.2.7
availability
fraction of the total time that the automatic measuring system is operational and for which valid measuring data are available

2.2.8
drift
change over time of a metrological characteristic (generally an output quantity) of the measuring system

NOTE Adapted from VIM:1993, 5.16.

2.2.9
lack of fit
systematic deviation, within the range of application, between the accepted value of a reference material applied to the measuring system and the corresponding result of measurement produced by the measuring system

NOTE 1 Lack of fit can be a function of the result of measurement.

NOTE 2 Since bias is considered as too specific and too difficult to be determined experimentally, the concept of lack of fit is used in this document.

NOTE 3 See 2.1.20.

2.2.10
limit of detection
minimum value of the measurand for which the measuring system is not in the basic state, with a stated probability

NOTE The limit of detection, also referred to as capability of detection, is defined by reference to the applicable basic state. But it may be different from *zero*, for instance for oxygen measurement as well as when gas chromatographs are used.

2.2.11
period of unattended operation
maximum interval of time for which the performance characteristics remains within a predefined range without external servicing, e.g. refill, adjustment

3 Symbols and abbreviated terms

A	availability
A_i	intercept of the regression function applied in the drift test at the accepted value of the measurand for each reference material RM_i
B_i	drift at the accepted value of the measurand in reference material RM_i
C_i	accepted value of the measurand (concentration) in reference material RM_i
C_0	accepted value of the measurand (concentration) in reference material RM_0 of the basic state
E_i	residual (lack of fit) representing the difference between the accepted value C_i of RM_i and the average $\overline{Y}_i$ of the results of measurement obtained by the measuring system for this reference material
$E_{i,\mathrm{rel}}$	relative value of residual E_i (relative lack of fit)
IS	interfering substance in the sample applied to the measuring system
k	concentration number of the interferent substance; level number of the influence quantity
m	number of repetitions
n	number of measurements
p	number of reference materials
PI	external influence quantity applied to the measuring system

$r_{0,95}$	repeatability limit at 95 % confidence
R_i	correlation factor
RM	reference material
RM_i	ith reference material having an accepted value C_i of the measurand with a known uncertainty
RM_0	reference material for the basic state having an accepted value C_0 of the measurand with a known uncertainty
s_0	standard deviation associated with the average $\overline{Y}_0$
$s_{\mathrm{adj},i}$	standard deviation representing the variability of the method
s_{P}	standard deviation of paired measurements
s_r	standard deviation of repeatability
$s_{r,\mathrm{cal}}$	repeatability standard deviation of the calibration method
t	time
t_{av}	averaging time
$t_{\mathrm{av,min}}$	minimum averaging time
t_{fall}	fall time
t_{lag}	lag time
t_{resp}	response time
t_{rise}	rise time
t_{wait}	waiting time
$t_{\nu,\,0,95}$	Student's distribution at 95 % confidence, two-sided, number of degrees of freedom ν
$X_{\mathrm{PI},0}$	normal value of the external influence quantity applied to the automatic measuring system
$X_{\mathrm{PI},k}$	kth value of the external influence quantity applied to the automatic measuring system
$X_{\mathrm{IS},k}$	kth concentration of the interferent substance in the sample applied to the automatic measuring system
Y	result of measurement; output signal
Y_{D}	limit of detection
$Y_{i,j}$	jth result of measurement obtained by the automatic measuring system by measuring the measurand in RM_i over the averaging time

$Y_{i,j,k}$ jth result of measurement obtained by the automatic measuring system k by measuring the measurand in RM_i over the averaging time

$Y_i(X_{PI,0})$ result of measurement obtained by the automatic measuring system by measuring RM_i over the averaging time with the external influence quantity being set at the normal value $X_{PI,0}$

$Y_i(X_{PI,k})$ result of measurement obtained by the automatic measuring system by measuring RM_i over the averaging time with the external influence quantity being set at the value $X_{PI,k}$

$Y_i(X_{IS,0})$ result of measurement obtained by the automatic measuring system by measuring RM_i over the averaging time in the absence of the interferent substance

$Y_i(X_{IS,k})$ result of measurement obtained by the automatic measuring system by measuring RM_i over the averaging time with a concentration X_k of the interferent substance

$\bar{Y}_0$ average of the results of measurement $Y_{0,j}$ obtained by the automatic measuring system by measuring the measurand in RM_0 for basic state

$\bar{Y}_i$ average of the results of measurement $Y_{i,j}$

$\alpha_{i,k}$ sensitivity coefficient of an interferent substance at concentration number k with the measurand at concentration C_i

$\beta_{i,k}$ sensitivity coefficient of an external influence quantity at level number k with the measurand at concentration C_i

Γ intercept of the regression function applied in the lack of fit test

Δ slope of the regression function applied in the lack of fit test

4 Terms of reference of the test programme

For performing a test program according to this International Standard, the terms of reference are a key element to be agreed upon, before starting the test program, between the client and the laboratory which performs such test program. It specifies the client requirements and the corresponding test conditions. This shall include at least:

— a list of the performance characteristics to be determined, together with their associated data quality objectives;

— the measuring range over which the measuring system is to be tested;

— the averaging time for field operation over which measurements shall be obtained under normal (or envisaged) operation;

— the averaging time for laboratory tests over which measurements shall be obtained under stable laboratory conditions;

— the number of complete automatic measuring system(s) to be tested and providing – for the purpose of the test – the signal corresponding to the primary measurements results (as defined in 2.1.6).

Averaging time for field operation is the averaging time used by the automatic measuring system to produce routine measurement results under normal (or envisaged) operations, for example half an hour for air emission measurements and one hour for ambient air measurements. This averaging time for field operation can be too long to be used during laboratory tests. Therefore, an averaging time for laboratory test is chosen.

It is recommended that the averaging time for laboratory test is chosen such that:

a) the duration of a test not dedicated to drift is limited to minimize the possible drift effect during this test;

b) the number (see 6.4.1) of primary results of measurement collected over the test averaging time is equal to the number of primary measurement results collected over the averaging time for field operation;

c) the criteria given in 6.3.3 is fulfilled.

All conditions and influences should be kept under control and considered constant under stable laboratory conditions during the test. If this is not obtained, a), b) and c) should be reconsidered as well as the controlling equipment.

NOTE 1 Most of the performance characteristics are determined under stable laboratory conditions maintained during the tests. Some performance characteristics may also be determined in the field under field conditions as indicated in the following clauses.

NOTE 2 The terms of reference may specify – in accordance with the objectives of the test programme – stronger requirement(s) than specified in this International Standard, e.g. for the waiting time in 6.3.3, six response times instead of the specified value four response times.

NOTE 3 The number of complete automatic measuring system(s) to be tested is dictated by the objectives of the test programme. Certain certification schemes specify in their terms of reference that two identical automatic measuring systems are tested. For other applications, the terms of reference specify a test programme for a given automatic measurement system, e.g. determination/verification of specific performance characteristics of an automatic measuring system in view of regulatory periodic measurement, or of its installation in the field.

In the terms of reference, the client (operators and/or regulators) may require that the laboratory performing the test programme is accredited according to ISO/IEC 17025 for the present International Standard, in view of being confident – without auditing itself the laboratory – that the test programme is performed in accordance with the present International Standard under a recognized Quality Management System. This approach is often selected and is recommended for certification schemes of automatic measuring systems.

5 Performance characteristics

5.1 Basic requirements

Before starting the tests, the following elements shall be available:

— the information describing and defining the different parts of the automatic measuring system to be tested (see 2.1.2);

— the operating procedures of the automatic measuring system, including, where appropriate, the procedure of routine adjustment, routine check and calibration;

— the terms of reference for the test program specifying the client requirements and the corresponding test conditions as specified in Clause 5;

— the complete automatic measuring system(s) to be tested, as specified in the terms of reference. Such measuring system(s) shall provide the output signal corresponding to the averaging time for field operation under normal (or envisaged) operations as well as – for the purpose of the test – the signal corresponding to the primary measurements results (as defined in 2.1.6).

5.2 Performance characteristics under laboratory conditions

This International Standard provides procedures to determine the following performance characteristics under laboratory conditions as required in the terms of reference:

— lag time t_{lag}, rise time t_{rise}, fall time t_{fall}, response time t_{resp}, minimum averaging time $t_{\text{av,min}}$;

— repeatability standard deviation s_r;

— lack of fit E_i;

— limit of detection Y_{D};

— repeatability standard deviation of the calibration method specified in the terms of reference $s_{r,\text{cal}}$;

— drift B_i;

— sensitivity coefficients $\alpha_{i,k}$ of interferent influence quantities;

— sensitivity coefficients $\beta_{i,k}$ of external influence quantities.

5.3 Performance characteristics under field conditions

This International Standard provides procedures to determine the following performance characteristics under field conditions as required in the terms of reference:

— standard deviation of paired measurements s_{P};

— drift B_i;

— availability A.

6 Test methods

6.1 General requirements

The fitness for purpose of the testing facilities shall be verified. The characteristics of the testing facilities, especially the response time and repeatability, shall be determined and shall meet the test requirements. If this is not the case, either the test requirements shall be amended subject to client acceptance or the tests shall not be performed.

Unless otherwise specified in this International Standard, the expanded uncertainty of the reference material expressed with a level of confidence of 95 % shall be less than 10 % of its concentration.

NOTE 1 An expanded uncertainty of 10 % of the reference material defines an interval about the reference value C of $[(1 - 10\ \%) \times C; (1 + 10\ \%) \times C]$.

For all tests, the automatic measuring system shall be operated and adjusted according to the specifications of the operating procedures. The automatic measuring system shall be allowed to warm-up as specified in the operating procedures. The measuring system shall be tested under the conditions specified in the terms of reference.

In the test program, the first step to be performed shall be the determination of the response time of the automatic measuring system to be tested.

Automatic measuring system's indication shall not be blocked around the basic state. Alternatively, the threshold shall be shifted by a constant value which is subtracted from the indication. Negative values shall not be suppressed (live zero). For chromatography techniques, the detection limit cannot be determined by application of zero samples. In this case, and for automatic measuring system without any mobility around zero, C_0 (accepted value of the measurand in the reference material RM_0 for the basic state) can be specified as a low concentration.

Except for the tests under 6.7 and 6.8, specific influence quantities considered in the term of reference shall be maintained such that their effect on the performance characteristics is not significant during the tests.

Unless otherwise specified in the terms of reference, all data shall be reported except when a default or mis-operation has been identified. In such cases, for instance, a break or burnout of a component, an appropriate statement shall be made in the test report.

The automatic measuring system shall be subjected to the required test only when adjustment has been performed over the specified range of operation, including the basic state. Any calibration included in adjustment shall be performed according to the operating procedures of the automatic measuring system to be tested.

NOTE 2 Calibration at the basic state specified in the terms of reference is a very important step in the procedure, not only with respect to the basic state, but also in the evaluation of the limit of detection.

6.2 Requirements on the testing laboratory

Laboratories performing a test programme according to this International Standard shall have test facilities fulfilling the requirements of this International Standard and of the terms of reference as applicable for each performance characteristic included in the test programme.

6.3 Response time and minimum averaging time under stable laboratory conditions

6.3.1 Continuous automatic measuring system

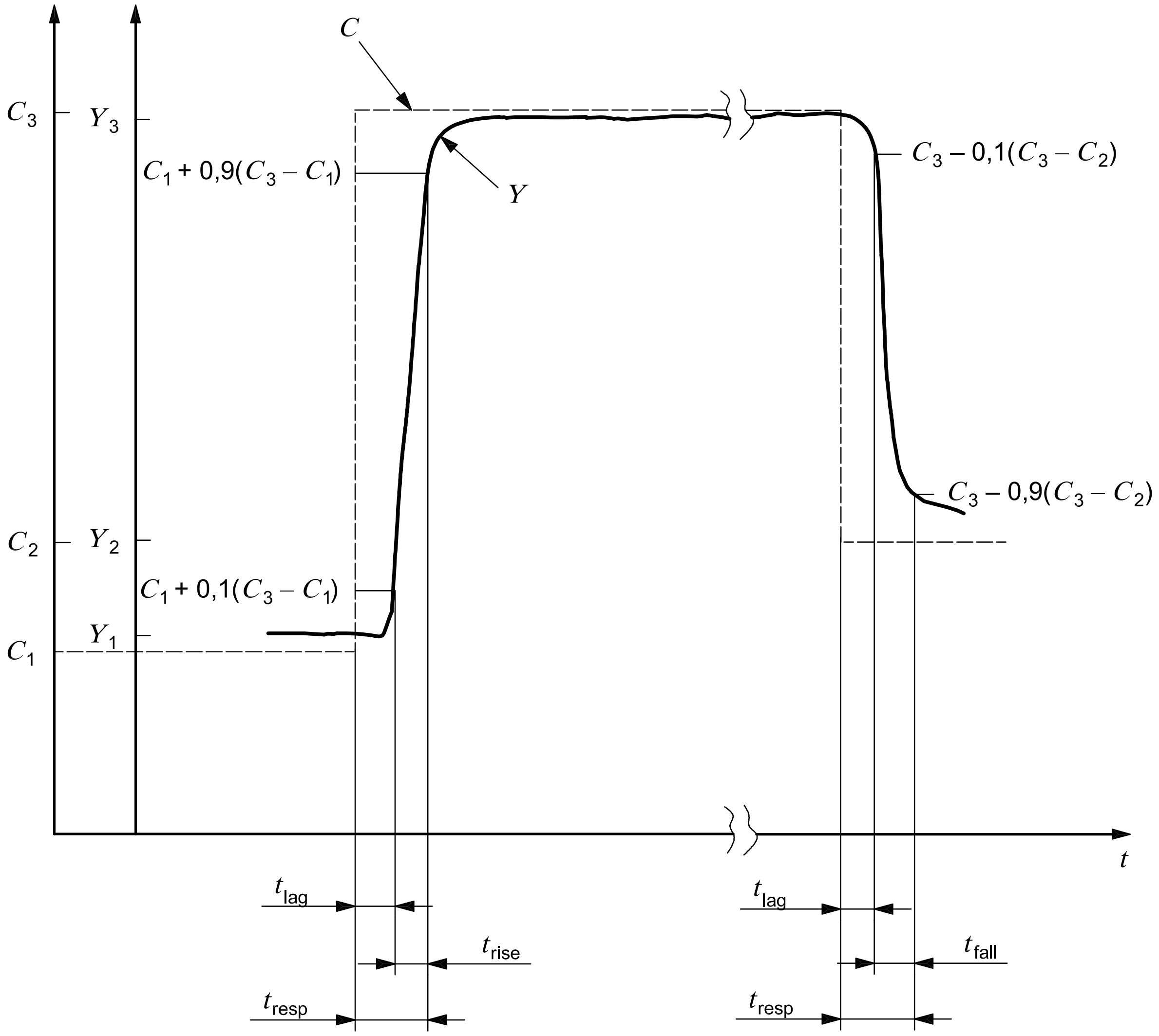
In order to establish response time, lag time, rise time and fall time, a step function of the measurand shall be input to the continuous automatic measuring system. Unless otherwise specified in the terms of reference, the step produced by the test facility shall have a rise time (between 10 % and 90 % of the step change) less than 10 % of the averaging time to be applied during the tests. The step change of the test facility shall correspond to at least 60 % of the measuring range (see Figure 1). If rise time and fall time differ, the longer one is to be taken for the computation of the response time.

For instruments where transient oscillations occur in approach to the final output signal, the final output signal is considered as reached when the oscillations fall to within 10 % of the input step.

NOTE A significant difference between the rise time and the fall time indicates memory effects, i.e. dependence of a result of measurement on the values of the measurand during previous measurement(s).

6.3.2 Discontinuous automatic measuring system

The minimum averaging time of a discontinuous automatic measuring system is determined by the maximum duration corresponding to the sampling time, filling time, accumulation time and analysis time, depending on the measurement method.



Key

C_i value of the measurand in RM_i

Y_i output signal obtained by measuring the measurand in RM_i

t_{lag} lag time

t_{rise} rise time

t_{fall} fall time

t_{resp} response time

Figure 1 — Illustration of the time characteristics of an automatic measuring system; Change of the output signal Y (output) after a change of the reference value C (input) as a function of time t

6.3.3 Waiting time and minimum averaging time

During laboratory tests, for each application of a reference material RM_i to an automatic measuring system, measurements shall not be undertaken immediately after the start of such application. The operator shall wait for a waiting time for laboratory tests equal to at least four times the response time of the automatic measuring system, unless otherwise specified in the terms of reference (see Figure 2).

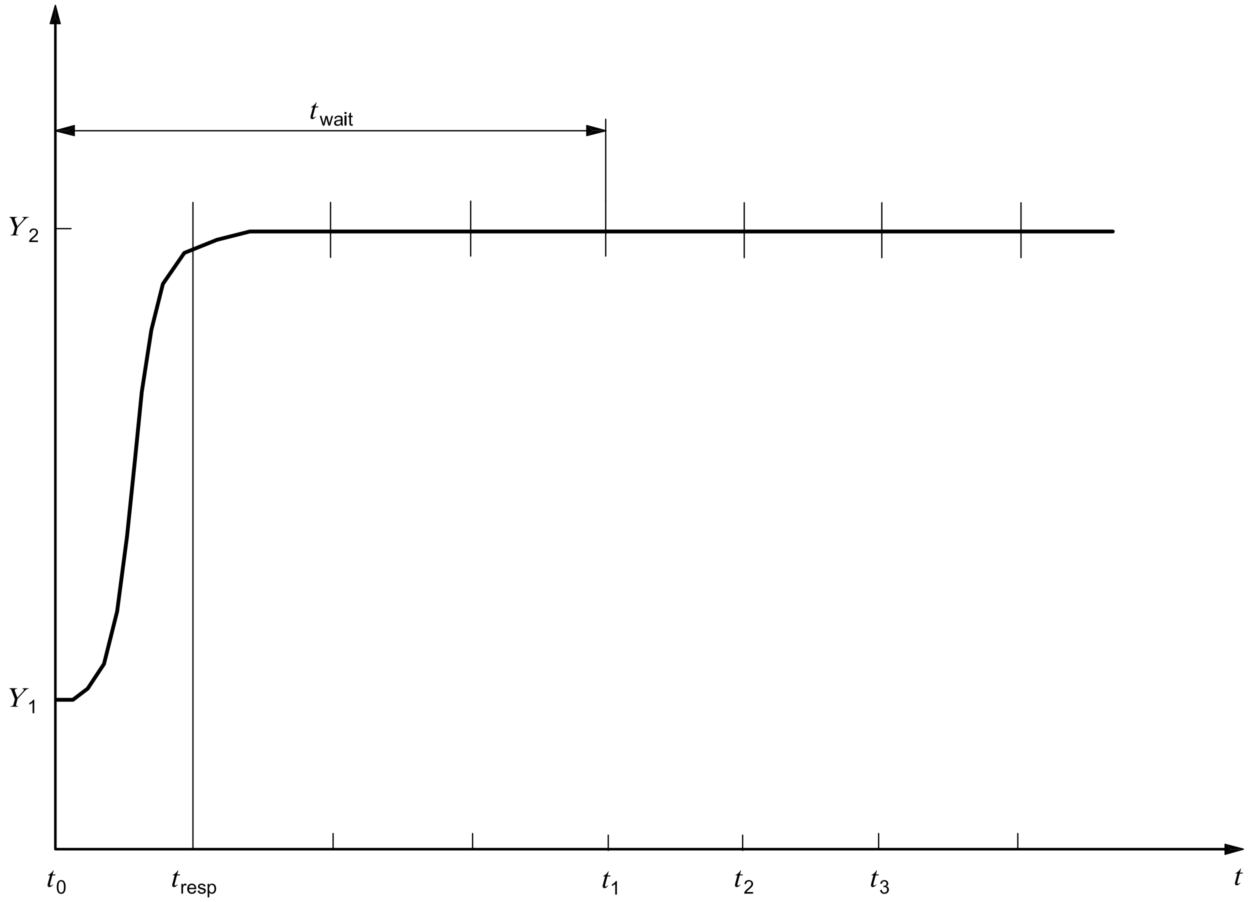


Figure 2 — Illustration of the waiting time t_{wait} after a change of the reference value C (input) where t_{resp} is the response time and t_1, t_2 and t_3 are the starting times of the first, second and third measurement

The minimum averaging time shall be equal to the response time, unless a larger time is specified in the terms of reference.

The averaging time for laboratory tests shall be greater or equal to the minimum averaging time.

6.4 Repeatability, lack of fit and limit of detection under stable laboratory conditions

6.4.1 Minimum requirements for test facilities and measurements

The laboratory shall provide the following reference materials for the measurements of the measurand with the automatic measuring system to be tested:

— a reference material RM_0 for testing the basic state having an accepted value C_0 of the measurand (for example, zero CO concentration for CO measurements or 20,9 % O_2 concentration for O_2 measurements);

— a set of at least four reference materials RM_1 to RM_4 having accepted values of the measurand C_1 to C_4, approximately equally distributed across the measuring range over which the automatic measuring system shall be tested.

The uncertainty of the reference material shall meet the test requirements as specified in the terms of reference.

During laboratory tests, for each application of a reference material RM_i to an automatic measuring system, measurements of the measurand shall not be undertaken immediately after the start of such application: the operator shall wait for a waiting time for laboratory tests equal to four times the response time of the automatic measuring system, unless otherwise specified in the terms of reference.

Measurements shall be performed over the averaging time for laboratory test and the results of measurement be taken to calculate the average response Y_i of the automatic measuring system for each RM_i:

— for continuous automatic measuring systems, the response Y_i is the average of at least 30 (unless otherwise specified in the terms of reference) primary results of measurement collected over the averaging time for laboratory test from the automatic measuring system;

— for non-continuous automatic measuring systems, the response Y_i is the average of all the primary results of measurement given by this measuring system for cycles occurring during the averaging time for laboratory test.

After each application of a RM_i to an automatic measuring system, it is not mandatory to return to the basic state, since each application of a RM_i to the automatic measuring system shall start with a waiting time during which the automatic measuring system and the bench providing this RM_i are stabilised as specified in this International Standard.

6.4.2 Requirements for the repeatability determination under stable laboratory conditions

Perform for each accepted value C_i at least 10 consecutive measurements providing results of measurement $Y_{i,j}$ obtained over the averaging time for laboratory test as specified in 6.4.1.

The concentration of the test gas provided by the bench facilities shall not vary during the test period by more than 25 % of the specified test criterion or of the expected repeatability.

NOTE This requirement can only be checked with a different analyser of sufficient quality, since the result of the check combines the behaviour of the bench and of the analyser.

The overall duration necessary to perform these tests shall be short enough not to be sensitive to drift effects. If the experiments are performed during several days, they shall be carried out on consecutive days. If a drift effect is found or suspected, then the procedure in 6.6 to quantify this drift shall be applied and assessed in the framework of the terms of reference.

If internal checks or adjustments at basic state or span level are performed during these tests, the repeatability includes the influence of variability of these adjustments. If this influence of the adjustment method specified in the operating procedures has to be known, a special experiment shall be carried out (see 6.5).

6.4.3 Requirements for the lack of fit determination under stable laboratory conditions

The test shall be performed with at least five different reference materials including the basic state. Perform for each RM with accepted value C_i at least 10 consecutive measurements. At each level, calculate from the 10 results of measurement the average $\overline{Y}_i$. The residuals E_i (lack of fit) shall be determined according to Equation (1):

$$E_i = C_i - \overline{Y}_i \qquad (1)$$

NOTE The requirement of at least 10 measurements limits the effect of repeatability.

If the expected uncertainty of the average $\overline{Y}_i$ is larger than 25 % of the criterion of lack of fit specified in the terms of reference, the number of measurements carried out for each RM shall be accordingly increased.

The concentration of the test gas provided by the bench facilities shall not vary during the test period by more than 25 % of the specified test criterion for lack of fit (i.e. a maximum variation of 0,5 %, if the criteria is 2 %). The matrix of the test gas shall be the same as the matrix of the reference gas used for calibration. The uncertainty in the ratios of the applied concentrations shall be less than 0,5 % with respect to each other.

The overall duration necessary to perform these tests shall be short enough not to be sensitive to drift effects. If the experiments have to be performed during several days, they shall be carried out on consecutive days. If a drift effect is found or suspected, then the procedure in 6.6 to quantify this drift shall be applied.

If internal checks or adjustments at basic state or span level are performed during these tests, the repeatability includes the influence of variability of these adjustments. If this influence of the adjustment method specified in the operating procedures shall be known, an additional experiment shall be carried out (see 6.5).

The residuals should be plotted versus the accepted values of the measurand in the RM. Such a plot is a powerful tool to detect deviations that may come from a lack of fit but these deviations may come from the adjustment proposed by the manufacturer. Thus, if the RM used for the adjustment of the instrument differs from the RM used by the laboratory to perform the lack of fit test, then the analyser can give a response different from what is given when the second RM material is used, deviation due to the uncertainty attached to the concentration of both RM.

The method to determine the lack of fit should be chosen depending on whether the linear regression function is used on the site to get the final result.

If the effect of lack of fit shall be determined independently of the problem of deviation due to an inaccurate adjustment, the following calculation procedure should be followed:

A linear regression function in the form of the following Equation (2) is calculated:

$$Y_{i,j} = \Gamma + \Delta \times C_i \tag{2}$$

where

C_i is the accepted value of the measurand (concentration) of the reference material RM_i, with i = 1 to p;

p is the number of reference materials RM_i ;

$Y_{i,j}$ is the jth individual response of the automatic measuring system at concentration C_i , with j = 1 to m;

Γ is the intercept of the regression function;

Δ is the slope of the regression function;

m is the number of repetition at concentration C_i .

For the regression, all measuring points (including zero) are taken into account. The coefficients of linear regression are calculated by Equations (3) and (4):

$$\Delta = \frac{\sum_{i=1}^{p}\sum_{j=1}^{m} C_i Y_{i,j} - \sum_{i=1}^{p}\sum_{j=1}^{m} Y_{i,j}\overline{C}}{m \times \sum_{i=1}^{p} (C_i - \overline{C})^2} \tag{3}$$

$$\Gamma = \overline{Y} - \Delta \times \overline{C} \tag{4}$$

where

$\overline{C}$ is the average value of the values C_i ; $\overline{C} = \frac{1}{p}\sum_{i=1}^{p} C_i$

$\overline{Y}$ is the average value of the values $Y_{i,j}$; $\overline{Y} = \frac{1}{m \cdot p}\sum_{i=1}^{p}\sum_{j=1}^{m} Y_{i,j}$

The residual of the average E_i at each concentration C_i (excluding the zero level) is calculated according to Equation (5):

$$E_i = \overline{Y_i} - (\Gamma + \Delta \times C_i) \quad (5)$$

where

$\overline{Y_i}$ is the average value of the values $Y_{i,j}$ at concentration C_i ; $\overline{Y_i} = \frac{1}{m}\sum_{j=1}^{m} Y_{i,j}$

Each relative residual $E_{i,\text{rel}}$ is calculated by Equation (6) and compared to the specification:

$$E_{i,\text{rel}} = \frac{E_{i,\text{rel}}}{\overline{Y_i}} \times 100\,\% \quad (6)$$

Each relative residual is compared to the specification. When one or more of the relative residuals does not fulfil the criterion, then the analyser does not pass the lack of fit test.

6.4.4 Requirements for the limit of detection determination under stable laboratory conditions

Perform for the accepted value C_0 at least 10 consecutive measurements each over the averaging time for laboratory tests. Calculate from the 10 results of measurement the average $\overline{Y}_0$ and the associated standard deviation s_0.

The concentration of the test gas provided by the bench facilities at the basic state shall not vary during the test period by more than 25 % of the criteria for the limit of detection.

The overall duration necessary to perform these tests shall be short enough not to be sensitive to drift effects. If the experiments shall be performed during several days, they shall be carried out on consecutive days. If a drift effect is found or suspected, then the procedure in 6.6 to quantify this drift shall be applied.

If internal checks or adjustments at basic state or span level are performed during these tests, the repeatability includes the influence of variability of these adjustments. If this influence of the adjustment method specified in the operating procedures shall be known, an experiment in accordance with 6.5 shall be carried out.

6.4.5 Combined test procedures and corresponding results

6.4.5.1 General

The test procedures provided in this section are informative as possible combined procedures when the three performance characteristics specified in 6.4.2, 6.4.3 and 6.4.4 shall be determined.

6.4.5.2 Experiments

The experiments are designed to determine simultaneously and under stable laboratory conditions the repeatability, lack of fit and limit of detection.

Reference materials RM_i are applied to the automatic measuring system with the following sequence of accepted values C_i of the measurand (concentration):

— C_0 = 0 % of the test range;

— C_3 = 80 % of the test range;

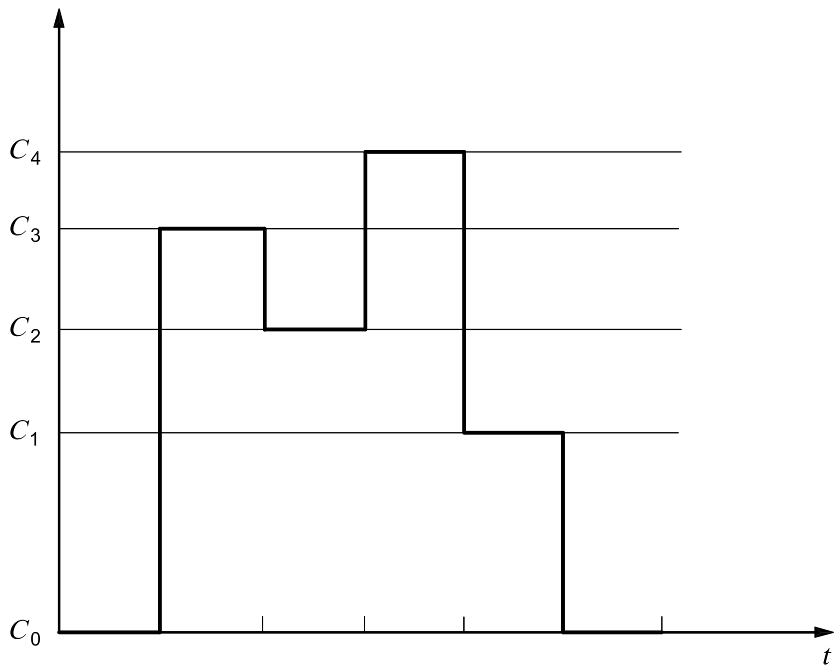
— C_2 = 60 % of the test range;

— C_4 = 95 % of the test range;

— C_1 = 40 % of the test range;

— C_0 = 0 % of the test range.

For each of the first five concentration steps (C_0, C_3, C_2, C_4, C_1), 10 consecutive measurements are performed. The duration of each measurement is the averaging time for laboratory tests. Three additional measurements are performed during a final application at C_0.



i =	0	3	2	4	1	0
C =	$C_{0,j}$	$C_{3,j}$	$C_{2,j}$	$C_{4,j}$	$C_{1,j}$	$C_{0,j}$
Y =	$Y_{0,j}$	$Y_{3,j}$	$Y_{2,j}$	$Y_{4,j}$	$Y_{1,j}$	$Y_{0,j}$
j =	1 to 10	1 to 10	1 to 10	1 to 10	1 to 10	11 to 13

Figure 3 — Illustration of the experiments of the combined tests; sequence of accepted values C of RM_i and results of measurement Y

The final step at C_0 provides additional information regarding a possible drift effect and more extended information on the critical basic state.

6.4.5.3 Repeatability under stable laboratory conditions

For each accepted value C_i (i = 1 to 4), the standard deviation of repeatability s_r and the repeatability limit at 95 % confidence (given as a $\pm r_{0,95}$ interval) shall be determined from the available 10 results of measurement $Y_{i,j}$ (j = 1 to 10).

An example is given in Annex A.

6.4.5.4 Lack of fit under stable laboratory conditions

For each accepted value C_i (i = 1 to 4), calculate the average $\overline{Y}_i$ from the results of measurement $Y_{i,j}$ and determine the residuals E_i by Equation (1).

The maximum deviation equal to the greatest E_i provides an indication on the maximum lack of fit.

Plot the residuals E_i versus the accepted values C_i in absolute values or preferably in relative values E_i/C_i versus C_i. The shape of the graph provides information on the type of lack of fit.

Examples are given in Annex A.

6.4.5.5 Limit of detection under stable laboratory conditions

The average $\overline{Y}_0$ and the associated standard deviation s_0 shall be calculated from the results of measurement $Y_{0,j}$.

In this experiment, the model equation is $Y = C$, where Y is the result of measurement obtained by the automatic measuring system and C the accepted value of the measurand in the RM. With the same confidence level, the limit of detection $Y_{\text{D, 0,95}}$ is estimated by Equation (7):

$$Y_{\text{D, 0,95}} = \overline{Y}_0 - C_0 + 2 \times t_{\nu,\text{ 0,95}} \times s_0 \qquad (7)$$

where

$\overline{Y}_0$ is the average of the values $Y_{0,j}$; j = 1 to 13

$t_{\nu,\text{ 0,95}}$ is the Student's factor at 95 % confidence, two-sided, number of degrees of freedom $\nu = n - 1$;

n the number of measurements for the determination of s_0.

NOTE 1 The limit of detection may also be determined by ISO 11843-2 and is not significantly different from the result given by the above simplified procedure, provided that the repeatability of the calibration remains negligible.

NOTE 2 Different factors may result in a higher limit of detection under field conditions due to e. g. interferences, repeatability of calibration.

6.4.6 Analysis of results

Unless otherwise specified in the terms of reference, all data shall be reported except when a default or mis-operation has been identified. In such cases, for instance, a break or burnout of a component, an appropriate statement shall be made in the test report.

6.5 Repeatability of the calibration method specified in the terms of reference under stable laboratory conditions

Perform the calibration method specified in the terms of reference. Then apply the reference materials RM_0 and RM_4 to the automatic measuring system. The operator shall wait for a waiting time of four times the response time of the automatic measuring system, unless otherwise specified in the terms of reference. The results of measurement $Y_{0,j}$ and $Y_{4,j}$ are obtained over the averaging time as specified in 6.4.1. Repeat this procedure 10 times. Calculate, from the results of measurement $Y_{0,j}$ and $Y_{4,j}$ (j = 1 to 10), the averages $\overline{Y}_0$ and $\overline{Y}_4$. For both accepted values C_0 and C_4, calculate the residuals by Equation (1).

E_i represents the lack of fit on the results of measurement due to the adjustment method proposed by the operating procedures.

The standard deviation $s_{\text{adj},i}$ of the $Y_{i,j}$ population represents the variability caused by repeated application of the method of calibration specified in the terms of reference.

Plot the residuals versus the accepted values C_0 and C_4.

The concentration of the test gas provided by the bench facilities shall not vary during the test period by more than 25 % of the corresponding RM accepted value.

6.6 Drift under stable laboratory conditions

Perform a number of at least 10 replicated measurements equally spread over a time period and at concentrations specified in the terms of reference. Under laboratory conditions, it is recommended to determine the drift over a short term period. Under field conditions, drift determination over a long term period is recommended, as specified in 6.10. Automatic adjustments shall be performed as specified in the terms of reference and reported.

The acceptable drift as specified in the terms of reference should correspond to a duration longer than the duration required for the individual tests.

The concentration of the test gas provided by the bench facilities shall not vary during the test period by more than 25 % of the specified value for drift.

For each concentration C_i, the response $Y_{i,j}$ of the measuring system is given by Equation (8):

$$Y_{i,j} = A_i + B_i \times t_j \tag{8}$$

with

$$B_i = \frac{n\sum_{j=1}^{n} t_j Y_{i,j} - \left(\sum_{j=1}^{n} t_j\right)\left(\sum_{j=1}^{n} Y_{i,j}\right)}{n\sum_{j=1}^{n} t_j^2 - \left(\sum_{j=1}^{n} t_j\right)^2} \tag{9}$$

$$A_i = \frac{\sum_{j=1}^{n} Y_{i,j} - B_i \sum_{j=1}^{n} t_j}{n} \tag{10}$$

where

n is the number of measurements;

t_j is the jth time interval.

Calculate for each concentration C_i, the drift B_i and the correlation factor R_i specified by Equation (11):

$$R_i = \frac{n\sum_{j=1}^{n} t_j Y_{i,j} - \left(\sum_{j=1}^{n} t_j\right)\left(\sum_{j=1}^{n} Y_{i,j}\right)}{\sqrt{\left[n\sum_{j=1}^{n} t_j^2 - \left(\sum_{j=1}^{n} t_j\right)^2\right]\left[n\sum_{j=1}^{n} Y_{i,j}^2 - \left(\sum_{j=1}^{n} Y_{i,j}\right)^2\right]}} \tag{11}$$

6.7 Sensitivity coefficients of interferent influence quantities under stable laboratory conditions

6.7.1 General

Interference is expressed by the sensitivity coefficient $\alpha_{i,k}$ of the interferent substance (see Equation 12):

$$\alpha_{i,k} = \frac{Y_i(X_{\mathrm{IS},k}) - Y_i(X_{\mathrm{IS},0})}{X_{\mathrm{IS},k}} \tag{12}$$

where

- $Y_i(X_{\mathrm{IS},0})$ is the result of measurement obtained by the automatic measuring system by measuring RM_i over the averaging time in the absence of the interferent substance
- $Y_i(X_{\mathrm{IS},k})$ is the result of measurement obtained by the automatic measuring system by measuring RM_i over the averaging time with a concentration $X_{\mathrm{IS},k}$ of the interferent substance
- $X_{\mathrm{IS},k}$ is the kth concentration of the interferent substance in the sample applied to the automatic measuring system
- k is the concentration number of the interferent substance

NOTE 1 A quenching effect (due to condensation) can occur. A linear interpolation is then not possible and testing is necessary at least at C_0 and C_4.

NOTE 2 Humidity of the air sample can be an influence quantity.

6.7.2 Test procedure

First, apply a sample of the measurand at concentration C_i to the automatic measuring system. The result of measurement is $Y_{i,0}$.

Then, apply a sample of the measurand at the same concentration C_i and the interferent substance at concentration X_k to the automatic measuring system. The measurement result is $Y_{i,k}$.

NOTE 1 The test concentrations are specified in the terms of reference (see 5.1), in relation with the future conditions of use of the automatic measuring system. In general, medium concentrations are specified for the measurand and high but realistic concentrations for interferent substances.

NOTE 2 The sensitivity coefficients may vary with the concentrations specified for the measurand. Several determinations may be implemented, for instance at C_0 and C_4.

The concentration of the test gas provided by the bench facilities shall not vary during the test period by more than 25 % of the repeatability. The concentration of the interferent influence quantity provided by the facilities shall not vary during the test period by more than 10 % of the interferent quantity. The test gas to be used shall have an uncertainty of the concentration of the interferent influence quantity lower than 5 %.

6.8 Sensitivity to external influence quantities under stable laboratory conditions

6.8.1 General

An influence quantity is a quantity that is neither the measurand nor the concentration of a substance in the air mass under investigation, but that affects the result of measurement.

Each influence quantity shall be dealt with in the tests, according to the requirements of the terms of reference, in order to estimate its influence on the result of measurement of the automatic measuring system:

- sample flow-rate, pressure, temperature;
- temperature of the surrounding air;
- humidity of the surrounding air;
- voltage and frequency of electric supply network, power cut;
- vibrations;
- shocks;
- electrostatic discharge, electromagnetic interference, etc.

The variations of an external influence quantity induce variations of the response of the automatic measuring system. This influence is expressed by the sensitivity coefficient of the external influence quantity given by Equation (13):

$$\beta_{i,k} = \frac{Y_i(X_{\mathrm{PI},k}) - Y_i(X_{\mathrm{PI},0})}{X_{\mathrm{PI},k} - X_{\mathrm{PI},0}} \tag{13}$$

where

$Y_i(X_{\mathrm{PI},0})$ is the result of measurement obtained by the automatic measuring system by measuring RM_i over the averaging time with the external influence quantity being set at the normal value $X_{\mathrm{PI},0}$;

$Y_i(X_{\mathrm{PI},k})$ is the result of measurement obtained by the automatic measuring system by measuring RM_i over the averaging time with the external influence quantity being set at the value $X_{\mathrm{PI},k}$;

$X_{\mathrm{PI},k}$ is the kth value of the external influence quantity applied to the automatic measuring system;

k is the level number of the influence quantity.

NOTE 1 For several external influence quantities, the data to be determined may be only qualitative information, for instance for vibration, the equipment still functions or still meet the performance criteria.

NOTE 2 For several external influence quantities, reference to applicable IEC standards may be specified.

6.8.2 Test procedure

First, apply a sample of concentration C_i to the automatic measuring system. The external influence quantity is set at the normal value $X_{\mathrm{PI},0}$. All the other external influence quantities are maintained at their normal values. The measurement result is $Y_i(X_{\mathrm{PI},0})$.

Then, apply a sample of the same concentration C_i to the automatic measuring system. The external influence quantity is set at the value $X_{\mathrm{PI},k}$. All the other external influence quantities are maintained at their normal values. The measurement result is $Y_i(X_{\mathrm{PI},k})$.

NOTE 1 The test concentrations are specified in the terms of reference (see 5.1), in relation with the future conditions of use of the automatic measuring system. In general, medium concentrations are specified for the measurand and high but realistic variations for the external influence quantity.

NOTE 2 The sensitivity coefficients may vary with the values specified for the measurand. Several determinations may be implemented, for instance at C_0 and C_4.

Samples shall never undergo any modification when passing through the measuring system, e.g. condensation of water vapour shall be avoided.

The concentration of the test gas provided by the bench facilities shall not vary during the test period by more than 25 % of the specified repeatability. The value of the external influence quantity provided by the facilities shall not vary during the test period by more than 10 % of the range of variation of the influence quantity.

6.9 Standard deviation of paired measurements under field conditions

6.9.1 Minimum requirements for the test facilities and for taking the measurements

Paired measurements are performed under field conditions in order to obtain a measure of reproducibility.

Measurement results are then taken to calculate the average response Y_i over the averaging time for field operation of the automatic measuring system:

— for continuous automatic measuring system, the response Y_i is the average of at least 30 (unless otherwise specified in the terms of reference) primary results of measurement collected over the intended averaging time for field operation;

— for non-continuous automatic measuring system, the response $\overline{Y}_i$ is the average of all the primary results of measurement given by this measuring system for cycles occurring during the averaging time for field test.

6.9.2 Test procedure

The variability under field conditions obtained from the differences of paired measurements provides an evaluation of the maximum difference that may be found, with 95 % statistical confidence, between two results of measurement obtained:

— from two automatic measuring systems meeting the same specification;

— operated according to the same specified operating procedures and each system being calibrated with the same reference;

— at the same measurement location and under the field conditions prevailing at this location;

— by parallel measurements during the same period of time;

— distributed over the period of unattended operation (unless otherwise specified in the term of reference).

NOTE Such variability averages possible variations during the period of unattended operation. Alternatively it may be specified in the term of reference that this variability under field conditions should be determined at different periods of time, especially at the beginning and at the end of the period of unattended operation.

Adjust both automated measuring systems to read the same at the beginning of the test. Otherwise a bias term is included in the result. For each date, obtain at least 10 pairs of results of measurement $Y_{i,j,1}$ (system 1) and $Y_{i,j,2}$ (system 2) over the averaging time for field operation as specified in 6.4.1.

If internal checks or adjustments at basic state or span level are performed during these tests, the standard deviation includes the influence of the variability of these adjustments. If the influence of the adjustment method specified in the operating procedures has to be known, a special experiment shall be carried out (see 6.10).

Calculate the standard deviation of paired measurements according to Equation (14):

$$s_\text{P} = \sqrt{\sum \frac{\left(Y_{i,j,1} - Y_{i,j,2}\right)^2}{2n}} \qquad (14)$$

6.10 Drift under field conditions

Perform at least 10 replicated measurements of a reference material at the beginning and the end of the period of unattended operation, at least at C_0 and C_4. Under field conditions, it is recommended to determine the drift over a long period. Under laboratory conditions, drift determination over a short period is recommended as specified in 6.6. Automatic adjustments shall be performed as specified in the terms of reference and reported.

The concentration of the reference material shall have an uncertainty less than 25 % of the specified test criterion for drift.

For each concentration C_i, the response $Y_{i,j}$ of the measuring system is given by Equation (8).

Calculate for each concentration C_i, the drift B_i given by Equation (9) and the parameter R_i specified by Equation (11).

6.11 Availability under field conditions

During the field test, notes shall be taken to record any period of time during which the automatic measuring system is not operational and of any period of time during which the automatic measuring system is producing invalid measuring results.

From these records, calculate the availability A as the fraction of the total time that the automatic measuring system was operational and for which valid measuring data are available.

7 Test report

The test report shall at least include the following information:

— complete terms of reference of the test program;

— a reference to this International Standard (ISO 9169:2006);

— description and definition of the automatic measuring system under investigation;

— operating procedure of the automatic measuring system under investigation;

— description of the test facilities including its performance characteristics as determined or verified for this test program;

— description of the tests performed to determine the required performance characteristics of the automatic measuring system under investigation;

— test conditions not specified in the term of reference and justification of choices being made;

— tests results and determination of the required performance characteristics of the automatic measuring system under investigation;

— concentrations for which the performance characteristics have been determined, because performance characteristics may depend on the level of concentration.

Annex A
(informative)

Examples

A.1 General

The following test results have been obtained in accordance with this International Standard in the course of laboratory tests on a continuous automatic measuring system. They are provided in this informative annex to illustrate the determination of some performance characteristics as specified in this standard.

A.2 Repeatability and limit of detection

Tables A.1 and A.2 list the measurement results obtained for three reference materials repeated ten times (n = 10). The average and standard deviation are thereafter calculated for each RM (accepted values C_0, C_1 and C_7). The repeatability is then calculated for the three RM as well as the limit of detection.

Table A.1 — Measurement results obtained for three reference materials

C_i mg/m^3	$Y_{i,1}$ mg/m^3	$Y_{i,2}$ mg/m^3	$Y_{i,3}$ mg/m^3	$Y_{i,4}$ mg/m^3	$Y_{i,5}$ mg/m^3	$Y_{i,6}$ mg/m^3
0,00	0,06	0,08	0,06	0,07	0,06	0,06
77,57	78,81	78,82	78,31	78,31	77,80	77,80
352,12	348,3	348,5	349,1	349,3	349,0	348,5

Table A.2 — Measurement results obtained for three reference materials

C_i mg/m^3	$Y_{i,7}$ mg/m^3	$Y_{i,8}$ mg/m^3	$Y_{i,9}$ mg/m^3	$Y_{i,10}$ mg/m^3	$\overline{Y}_i$ mg/m^3	s_i mg/m^3
0,00	0,07	0,07	0,08	0,06	0,067	0,008 2
77,57	78,52	78,13	78,64	78,59	78,373	0,373 3
352,12	348,7	348,6	349,0	348,5	348,75	0,327 4

Repeatability at C_0: 0,008 2 mg/m^3

Repeatability at C_1: 0,373 3 mg/m^3

Repeatability at C_7: 0,327 4 mg/m^3

Limit of detection: 0,099 9 mg/m^3

A.3 Lack of fit

For the lack of fit, Table A.3 provides the average of repeated measurements versus the accepted reference values. Residuals are calculated in absolute value as well as in relative values. Relative residuals are plotted versus concentrations in Figure A.1.

Table A.3 — Results of lack of fit test

C_i mg/m^3	$\overline{Y}_i$ mg/m^3	E_i mg/m^3	$E_{i,\text{rel}}$ %
0,00	0,06	−0,06	−0,02
47,29	49,43	−2,14	−0,54
77,57	80,37	−2,80	−0,70
134,2	138,95	−4,75	−1,19
214,33	216,46	−2,13	−0,53
249,75	249,3	0,45	0,11
302,13	300,88	1,25	0,31
352,12	349,12	3,00	0,75
400,06	395,34	4,72	1,18

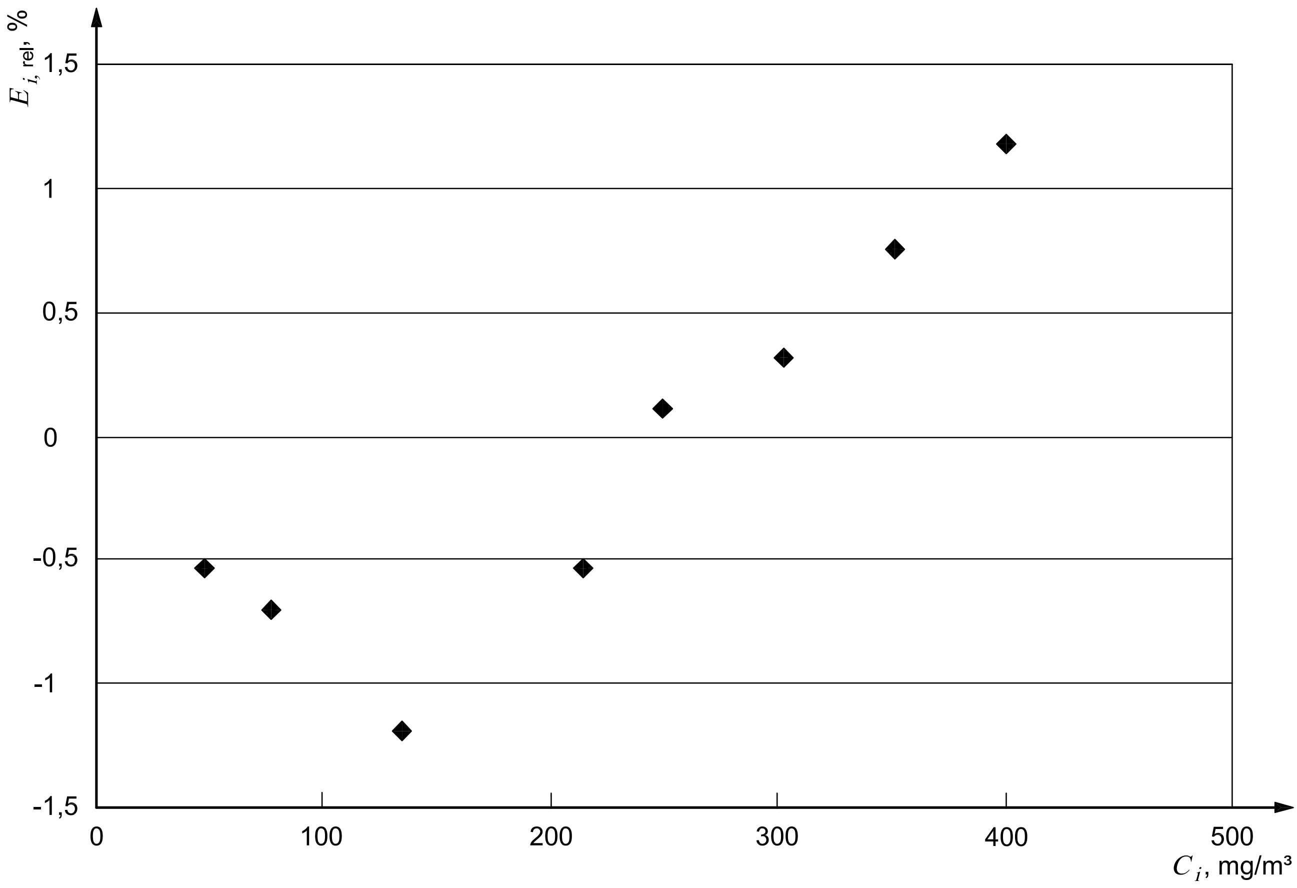


Figure A.1 — Relative residuals $E_{i,\text{rel}}$ versus concentrations C_i

The maximum deviation (residual) is 1,2 % of the range. Lack of fit for C_3 and C_8 is present, if the lack of fit criteria is less than 1 %. If the criterion is 2 %, there is no lack of fit.

A.4 Drift

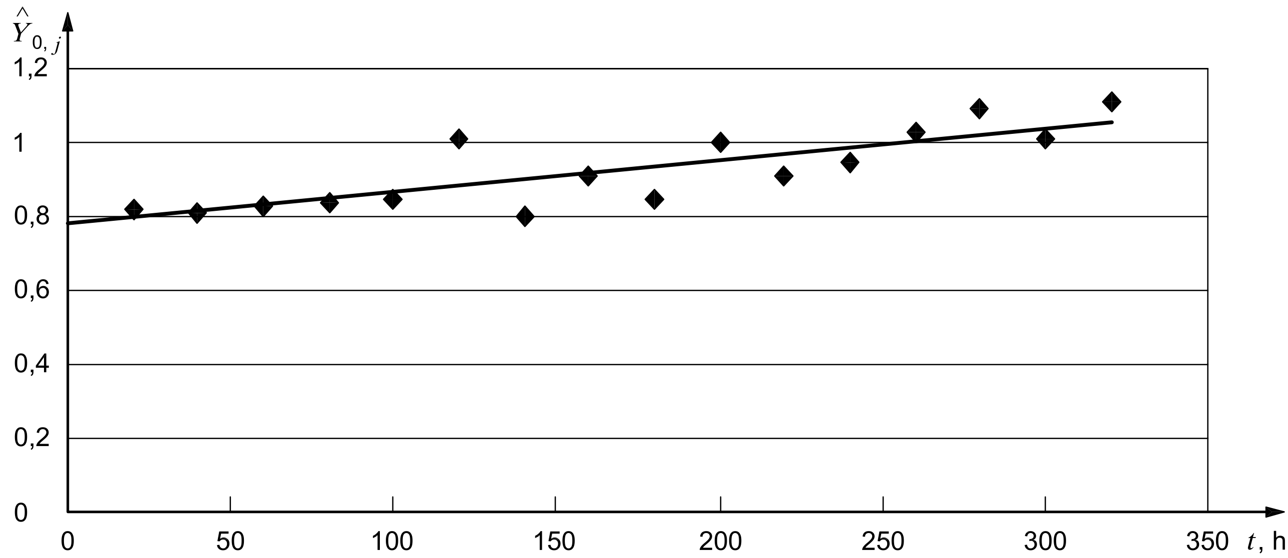
Tables A.4 and A.5 provide test results for drift and calculations resulting in Figure A.2 (for C_0) and Figure A.3 (for C_4) which show the graphical trend and the linear regression determined on the basis of the results.

Table A.4 — Results of drift test

n	t	$Y_{0,j}$	$Y_{4,j}$	$(t-\bar{t})^2$	$t\,Y_{0,j}$	$t\,Y_{4,j}$	t^2
1	0	0,80	210,29	25 600	0,0	0,0	0
2	20	0,82	209,78	19 600	16,4	4 195,6	400
3	40	0,81	210,79	14 400	32,4	8 431,6	1 600
4	60	0,83	210,29	10 000	49,8	12 617,4	3 600
5	80	0,84	209,79	6 400	67,2	16 783,2	6 400
6	100	0,85	209,78	3 600	85,0	20 978,0	10 000
7	120	1,01	211,79	1 600	121,2	25 414,8	14 400
8	140	0,80	211,29	400	112,0	29 580,6	19 600
9	160	0,91	212,29	0	145,6	33 966,4	25 600
10	180	0,85	210,28	400	153,0	37 850,4	32 400
11	200	1,00	211,79	1 600	200,0	42 358,0	40 000
12	220	0,91	209,78	3 600	200,2	46 151,6	48 400
13	240	0,95	211,28	6 400	228,0	50 707,2	57 600
14	260	1,03	209,28	10 000	267,8	54 412,8	67 600
15	280	1,09	210,28	14 400	305,2	58 878,4	78 400
16	300	1,01	209,78	19 600	303,0	62 934,0	90 000
17	320	1,11	210,29	25 600	355,5	67 292,8	102 400
Sum	2 720	15,62	3 578,85	163 200	2 642,3	572 552,8	598 400

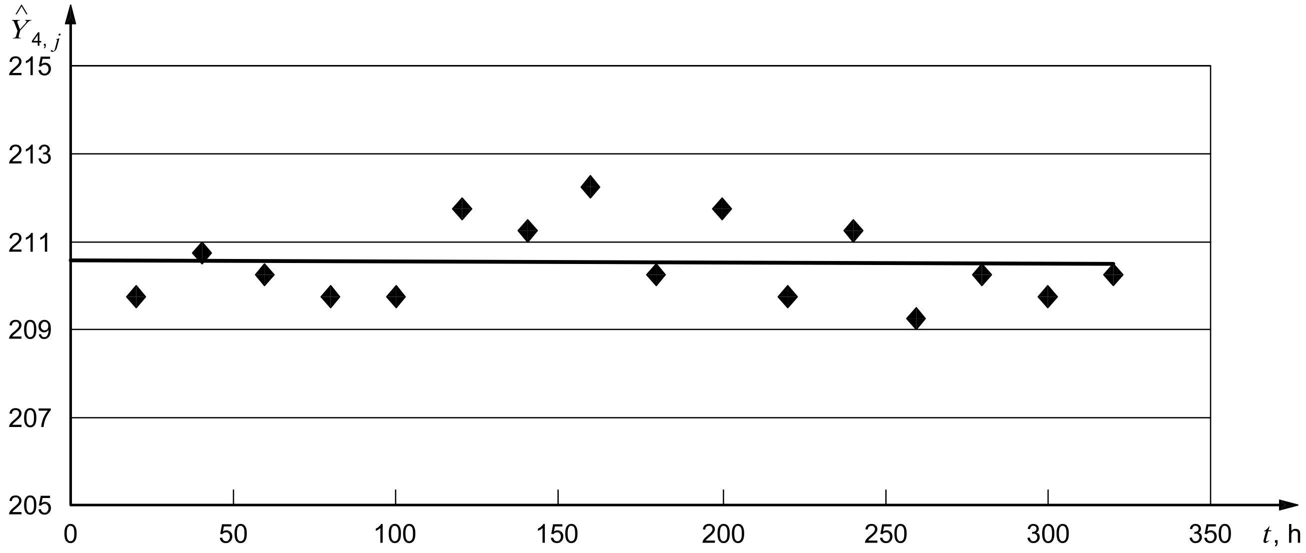
Table A.5 — Results of drift test

n	t	$\left(Y_{0,j}\right)^2$	$\left(Y_{4,j}\right)^2$	$\hat{Y}_{0,j}$	$\left(Y_{0,j}-\hat{Y}_{0,j}\right)^2$	$\hat{Y}_{4,j}$	$\left(Y_{4,j}-\hat{Y}_{4,j}\right)^2$
1	0	0,640 0	44 221,9	0,779	0,000 5	210,58	0,085 6
2	20	0,672 4	44 007,7	0,796	0,000 6	210,59	0,656 5
3	40	0,656 1	44 432,4	0,814	0,000 0	210,60	0,036 9
4	60	0,688 9	44 221,9	0,832	0,000 0	210,61	0,099 7
5	80	0,705 6	44 011,8	0,849	0,000 1	210,61	0,678 1
6	100	0,722 5	44 007,7	0,867	0,000 3	210,62	0,707 7
7	120	1,020 1	44 855,0	0,884	0,015 8	210,63	1,348 0
8	140	0,640 0	44 643,5	0,902	0,010 4	210,64	0,426 8
9	160	0,828 1	45 067,0	0,920	0,000 1	210,64	2,707 8
10	180	0,722 5	44 217,7	0,937	0,007 6	210,65	0,138 5
11	200	1,000 0	44 855,0	0,955	0,002 0	210,66	1,277 0
12	220	0,828 1	44 007,6	0,972	0,003 9	210,67	0,788 0
13	240	0,902 5	44 639,2	0,990	0,001 6	210,68	0,365 5
14	260	1,060 9	43 798,1	1,008	0,000 5	210,68	1,969 0
15	280	1,188 1	44 217,7	1,025	0,004 2	210,69	0,168 9
16	300	1,020 1	44 007,7	1,043	0,001 1	210,70	0,844 0
17	320	1,234 3	44 221,9	1,060	0,002 5	210,71	0,173 4
Sum	2 720	14,530 2	753 433,7	15,632	0,051 0	3 580,96	12,471 2



$$\hat{Y}_{0,j} = 0{,}000\ 88t + 0{,}778\ 73$$

Figure A.2 — Result of drift test at concentration C_0



$\hat{Y}_{4,j} = 0{,}000\ 387\ 3t + 210{,}582\ 5$

Figure A.3 — Result of drift test at concentration C_4

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