Technical Report

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Air quality — Validation of air quality measurement methods in the standardization process

Qualité de l'air — La validation des méthodes de mesure de la qualité de l'air dans le processus de normalisation

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO document should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

ISO/IEC 17025 defines expectations for verification and validation of test methods. These expectations are based on definitions in ISO/IEC Guide 99 and aim to ensure that test methods are suitable for their intended use and that test results have a known, documented level of quality.

This document describes protocols that have been used within ISO/TC 146 and other technical committees on air quality to verify and validate measurement methods. It also establishes guidance intended to be used for method validation. This document seeks to establish a consistent framework for method validation within ISO/TC 146 and its subcommittees.

Air quality — Validation of air quality measurement methods in the standardization process

1 Scope

This document provides an overview of the validation of air quality measurement methods in the standardization process.

This document deals with robustness testing and interlaboratory testing as the two main steps of partial and full validation. It applies to the different inter-related elements of air quality measurement methods, covering e.g. sampling, sample preparation, storage and transportation of the sample, extraction, analysis or quantification of a measured component and reporting.

Consequently, this document focuses on the "why" and "what" of validation tasks in direct relation to the different steps of the standardization process. This document is focused on the validation tasks for measurement methods either for the whole measurement process or for one of its constituent parts.

Given the informative aim of this document, it does not contain detailed procedures for performing the validation tasks, such as number of laboratories, number of samples, etc.

This document is relevant to measurement methods in ISO/TC 146 and all of its subcommittees.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 4225, *Air quality — General aspects — Vocabulary*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 4225 and the following apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

3.1
verification
provision of objective evidence that a given item fulfils specified requirements

[SOURCE: ISO/IEC Guide 99:2007, 2.44]

3.2
validation
verification, where the specified requirements are adequate for an intended use

[SOURCE: ISO/IEC Guide 99:2007, 2.45]

3.3
method
measurement procedure
detailed description of a measurement according to one or more measurement principles and to a given measurement method, based on a measurement model and including any calculation to obtain a measurement result

[SOURCE: ISO/IEC Guide 99:2007, 2.6, modified — Notes to entry omitted]

3.4
measurement method
method of measurement
generic description of a logical organization of operations used in a measurement

[SOURCE: ISO/IEC Guide 99:2007, 2.5, modified — Note to entry omitted]

3.5
reference method
RM
measurement method taken as a reference, which gives the reference value of the measurand

Note 1 to entry: A reference method is fully described.

Note 2 to entry: A reference method can be a manual or an automated method.

3.6
alternative method
AM
measurement method which complies with the criteria given by the reference method

Note 1 to entry: An alternative method can consist of a simplification of the reference method.

Note 2 to entry: Alternative methods can be used if equivalence to the reference method has been demonstrated.

3.7
evaluation
<analytical chemistry> examination of validation data to determine suitability for intended use(s)

3.8
sensitivity
<analytical chemistry> change in instrument response which corresponds to a change in the measured quantity

3.9
selectivity
<analytical chemistry> extent to which the method can be used to determine particular analytes in mixtures or matrices without interferences from other components of similar behaviour

3.10
linearity
<analytical chemistry> ability to use a straight line to describe the relationship between a measurement result and concentration of the analyte of interest

3.11
robustness
<analytical chemistry> measure of the capacity of an analytical method to remain unaffected within specified limits by small, but deliberate variations in method parameters

3.12
repeatability
measurement repeatability
measurement precision under a set of repeatability conditions of measurement

[SOURCE: ISO/IEC Guide 99:2007, 2.21]

3.13
repeatability condition
repeatability condition of measurement
condition of measurement, out of a set of conditions that includes the same measurement procedure, same operators, same measuring system, same operating conditions and same location, and replicate measurements on the same or similar objects over a short period of time

[SOURCE: ISO/IEC Guide 99:2007, 2.20, modified — Notes to entry omitted]

3.14
reproducibility
measurement reproducibility
measurement precision under reproducibility conditions of measurement

[SOURCE: ISO/IEC Guide 99:2007, 2.25, modified — Note to entry omitted]

3.15
reproducibility condition
reproducibility condition of measurement
condition of measurement, out of a set of conditions that includes different locations, operators, measuring systems, and replicate measurements on the same or similar objects

Note 1 to entry: The different measuring systems can use different measurement procedures.

[SOURCE: ISO/IEC Guide 99:2007, 2.24, modified — In Note 1 to entry, “may” changed to “can”; also, Note 2 to entry omitted]

4 Abbreviated terms

For the purposes of this document, the following abbreviated terms apply.

AAS	atomic absorption spectrometry
AM	alternative method
CRF	controlled release facility
FAIR	findable, accessible, interoperable and reusable
FID	flame ionisation detector
FTIR	Fourier-transform infrared
ICP	inductively coupled plasma
ICP-AES	inductively coupled plasma atomic emission spectrometry
ICP-MS	inductively coupled plasma mass spectrometry
LDAR	leak detection and repair
LIDAR	light detection and ranging
LOD	limit of detection

LOQ	limit of quantification
LV	limit value
MSD	mass selective detector
NPL	National Physical Laboratory
OELV	occupational exposure limit values
OGI	optical gas imaging
P-AMS	portable automated measuring systems
QA/QC	quality assurance and quality control
RDM	reverse dispersion modelling
RH	relative humidity
RM	reference method
SOF	solar occultation flux
ST-OELV	short-term occupational exposure limit values
STEL	short-term exposure limits
TDL	tuneable diode laser
TEM	transmission electron microscopy
TDLAS	tuneable diode laser absorption spectroscopy
TWA	time-weighted average concentration
UV	ultra-violet
VOC	volatile organic compounds
WP	work package

5 Purposes for validation

Validation is the process of defining an analytical requirement and confirming that the method under consideration has capabilities consistent with what the application requires in view of the measurement objective(s). This is often referred to as the method being fit for its intended purpose. An important part of this process is determining the uncertainty of the measurement results and whether this is suitable for the intended use, e.g. a maximum specific limit. It is also important to determine if it provides sufficient confidence in conclusions to be drawn from the measurement results.

Often, validation campaigns are designed to determine both an uncertainty associated with the entire measurement method and individual uncertainty components (i.e. various bias and precision terms) associated with individual components of the method. Either selectivity, limit of detection or limit of quantification, or a combination, can also be relevant. This supports the elaboration of the measurement method in terms of providing confidence that any maximum permissible uncertainty that is required for the entire method is fit for the intended application. It also supports the specification of any uncertainty requirement for individual components of the measurement method. Furthermore, validation campaigns confirm that reliable measurement results can be achieved by competent end users. Subsequently, this also serves to ensure that fit for purpose and achievable requirements are set by proficiency testing providers

(and equally importantly local competent authorities and national accreditation bodies) in assessing the competence of end users.

In the absence of full or partial validation, requirements in the measurement method are based on expert opinion. With no evidential data there is therefore a higher level of uncertainty at a given level of confidence that the measurement method has capabilities consistent with what the application requires, and that requirements placed upon end users by the measurement method are realistic and achievable.

6 Objectives for validation

The main objectives of validation are confirming that:

— the measurement method meets the requirements specified in the measurement objective;

— the measurement method specified in the standard is clearly described and practicable, or if collecting of improvement proposals is needed;

— the results are comparable when different testing laboratories or institutes use the measurement method for the same task.

NOTE Comparability can be quantified by e.g. repeatability and reproducibility.

Method validation is a process to verify that a measurement method fulfils specified requirements that are adequate for an intended use.[2] It is a series of actions, following development of a method but preceding routine implementation of the method, that demonstrates and documents the fitness of the method for the intended use.

The principal benefit of validation is to provide confidence that measurements made by using the measurement method provide data which can be relied upon by the end user(s) for making correct decisions in one or more of the following areas:

— process control;

— policy making;

— compliance with regulatory requirements (e.g. limit values for emissions or occupational exposure);

— suitability of modelling (e.g. atmospheric modelling).

Measurement methods contained in documents prepared by technical committees on air quality are intended for use in a variety of locations and with a variety of sampling and laboratory equipment. It is expected that a standard method, when properly used, provides consistent measurement results wherever it is used. Additionally, since ISO/IEC 17025 recommends the use of standard methods when available, it is incumbent for those who establish such methods (e.g. ISO/TC 146) to ensure a sufficient degree of validation to support their use in this manner.

Additional considerations are given in 8.2.

7 Types of validation

7.1 Validation of reference methods

Reference methods (RM) are measurement methods that have been validated and of which the quality of the measurement method is, given a specific field of application, fit for its intended purpose and accepted by experts and users. Knowledge on and documentation of the quality of the measurement method is essential to define it as a RM. Validation is therefore an essential step in the standardization process.

RM can be used as a legal reference in either legislation, regulation or in contracts between two or more parties, or a combination thereof. They need, therefore, to be self-supportive. RM are not necessarily of the

highest metrological quality. However, experts define a reference method as "reliable" and a good basis for decisions. In general, RM are "fit for purpose" in view of the measurement objective.

Validation of measurement methods is generally performed in two steps including performance characteristics relevant for the considered measurement method:

— robustness testing;

— interlaboratory testing.

As the first step is based on a first draft of the standard and each of the validation steps result in a revised draft standard, the implementation of validation in the standardization process normally relates to three different draft standards, the last one of which is published as a standard.

Cases can occur where the current state of the art is not sufficient for the efficient further development of the envisaged standard. In such a case, a so-called pre-normative research can be needed prior to any standardization with validation.

The robustness testing is generally performed by one or more competent laboratories which ideally already have experience with the new measurement method. The performance characteristics of the measurement method are determined through inter-laboratory experiments. Both steps are needed and contribute to the evaluation of the uncertainty of the measurement results.

The comparability of measurement results obtained by the standardized measurement method is ensured by metrological traceability of the measurement results by means of a documented unbroken chain of calibrations, each contributing to the measurement uncertainty, linking them to an appropriate reference.

Metrological traceability to the International System of Units (SI) can be achieved through:

— calibration provided by a competent laboratory; or

— certified reference materials with a stated metrological traceability to the SI units; or

— direct realization of the SI units.

When metrological traceability to the SI units is not technically possible, the metrological traceability can be ensured by use of an appropriate reference, e.g.

— reference materials without a stated metrological traceability to the SI units;

— results of RM, specified methods or consensus standards that are clearly described and accepted as providing measurement results fit for their intended use and ensured by suitable comparison.

Unfortunately, there is a clear disadvantage with certified reference materials as these are only available for a limited number of components and matrices and are, in general, so expensive that it is not financially possible to use certified reference materials for validation or routine checks. Therefore, it is important to specify in the standardized measurement method the appropriate reference.

The uncertainty of the measurement results obtained by the standardized measurement method consists of the uncertainty contributions resulting from the unbroken chain of calibrations. Therefore, it is important to specify in the standardized measurement method the minimum requirements for these uncertainty contributions.

In specific cases the introduction of a new RM results in the withdrawal of a previous RM. For example, due to the general application of new analytical instruments, the old RM is in practice no longer applicable. Validation of the new RM and cross-comparison of the results of both the old and the new RM allows the continuous use of data collected in the past (the "historical" data).

When a Technical Committee (TC) or Subcommittee (SC) starts a work item, this is generally given to a dedicated working group (WG). The aim of the work is to harmonize as far as possible the practices on a specific topic. Through a series of steps, consensus among the experts is achieved resulting in a first draft standard that, to the opinion of the experts, reflects the state of the art (in terms of standardization) and is assumed to be fit for purpose.

This first draft standard is used as a starting point for the validation work, or, if funds or means for validation are not sufficiently available, can be adopted by the TC or SC members and published as a Technical Specification (TS). When published as a TS, the document states that the measurement method has not been validated.

In general, at least the following different levels of validation can be distinguished:

— full validation by sufficient funding, which means that robustness testing and inter-laboratory testing (e.g. repeatability and reproducibility) as well as evaluation of the uncertainty are acceptably performed;

— partial validation in cases where results of a full validation are not available due to lack of funding, but a set of validation data provided e.g. by at least one member country or one testing laboratory, on the basis of current investigations being available and that expert assessment justifies the validity of these data;

— validation on the basis of existing historical data provided e.g. by at least one member country where the measurement method has been validated on a national level and expert assessment justifies the validity of these data.

7.2 Validation of alternative methods

In specific cases it can be necessary to standardize an alternative method (AM) to the already existing RM, e.g. if a simplification of the RM is desirable or a different measurement technique is available. The scope of the AM can be limited in comparison with the scope of the reference method, but is covered by the scope of RM.

With respect to validation, an AM is treated as outlined in 7.1 for the RM, but often with a limited scope in comparison with the RM. In addition, the equivalence of the AM with the RM is demonstrated for the scope of the AM. The three steps for demonstration of equivalence are:

— description of the AM and setting of the field of application;

— determination of the performance characteristics of the AM and calculation of the expanded uncertainty where appropriate and check of compliance with the maximum expanded uncertainty allowed for the RM;

— check of repeatability and lack of systematic deviation of the AM in comparison with the RM.

Guidance on demonstration of equivalence is given in EN 14793 for stationary source emission measurement methods and in the "Guide to the demonstration of equivalence of ambient air monitoring methods" for ambient air quality measurement methods[5].

The field of application of an AM can partially or completely cover the field of application of the RM. However, if it covers the fields of application of several RM (horizontal method), several evaluations of each RM are needed, e.g. in the case of multi-component measurement methods like FTIR.

The definition of the field of application depends entirely upon the body specifying the AM and the knowledge acquired during the development of the method. It is sometimes preferable to segment a field of application rather than to attempt to validate an overly general method. In this case, a validation file for each field of application is compiled.

7.3 Validation in the absence of certified reference materials

While metrological traceability of a measurement method to the SI is typically accomplished by use of certified reference materials, in some situations a certified reference material might not be available. In these situations, alternative approaches can include one or more of the following:

— inter-laboratory studies;

— recovery experiments using spiked samples;

— comparison with results obtained from another measurement method for the same measurand.

These alternatives do not necessarily establish metrological traceability.

8 Objective, design and documentation of a validation study

8.1 General

A validation study generally includes, at a minimum, the following aspects:

— objective(s) of the validation study (8.2);

— design of the validation study (8.3);

— documentation of the validation study (8.4).

NOTE Guidance on design and documentation of the validation study is also given in Eurachem documents.

8.2 Objective of a validation study

8.2.1 General

The initial step in the validation process is the analysis of the measurement objective (measurement task) and the associated measurement method to identify the specific elements and associated requirements of the measurement method. The validation concerns the following elements of the measurement method, for example:

— sampling;

— sample preparation including e.g. sample transfer and storage;

— analysis;

— performance characteristics;

— determination of the result of measurement and the associated measurement uncertainty;

— documentation and reporting.

8.2.2 Purpose(s) for performing the measurement

Because the objective of validation is to demonstrate fitness for purpose in view of the measurement objective, it is necessary to establish the purpose(s) for performing the measurement so that validation activities can be properly designed to establish that the measurement method is fit for the intended purpose(s). If the measurement method is to be fit for a single, narrow purpose, validation activities can be more narrowly defined. If, however, the purposes are broad or numerous, or both, validation activities also need to be sufficiently broad to address the full range of purposes for which the measurement method is intended to be suitable. For example, validation activities for a measurement method intended to measure a single air pollutant might not need to be as extensive as for a method intended to measure concentration of multiple metals in an air sample.

8.2.3 Representative sampling

It is important to develop sampling strategies that result in samples that reflect the properties of interest of the overall population being sampled. If this is not accomplished, even a reference method might not be able to produce reliable results for decision making. Method validation needs to include consideration for collecting representative samples.

8.2.4 Sample preparation

For many measurement methods, some preparation of the sample is necessary in advance of analysis.