SLOVENSKI STANDARD
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Validacija okoljskih preskusnih metod

Validation of environmental test methods

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CEN GUIDE 13

Validation of environmental test methods

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Contents

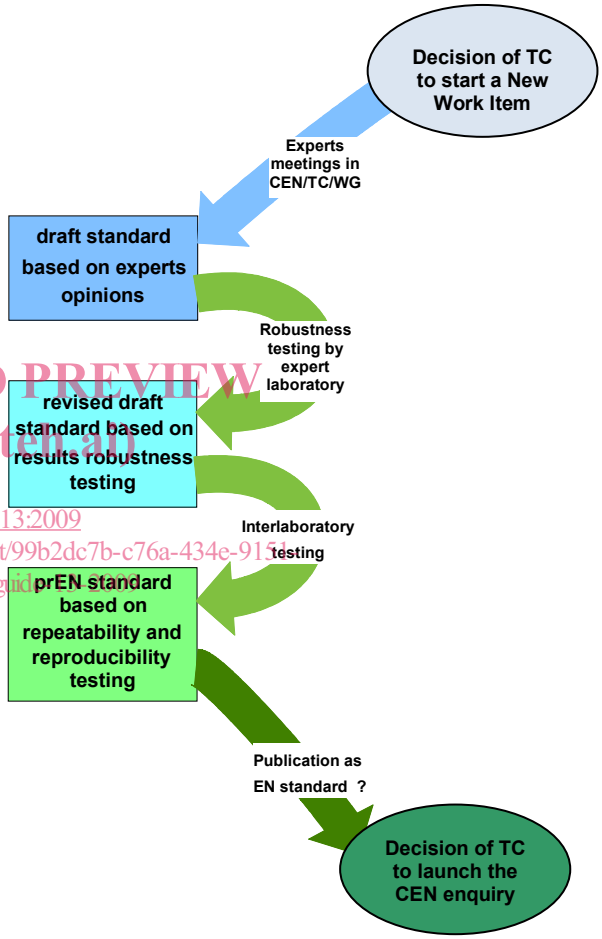
Foreword

This document has been produced by the CEN-SABE Environmental TCs Cooperation Team (ENV TCs) as a **policy document on validation tasks in the standardisation process of environmental test methods**.

The environmental TCs recognise that these **validation tasks are complex.** They consist of two main steps, the robustness testing and the inter-laboratory testing (determining repeatability and reproducibility), both interacting with the elaboration of the draft standards as shown in the flow chart. Furthermore, they apply to the different inter-related phases of encountered in environmental testing, typically sampling and production of laboratory sample, storage and transportation, extraction, analysis and reporting. Consequently, this document focuses on the 'why' and 'what' of validation tasks in direct relation to the different steps of the standardisation process. Given the policy aim of this document, it does not contain detailed procedures for performing the validation (such as number of laboratories, number of samples, etc.).

The environmental TCs recognise that the environmental test methods published as standards are very often used as reference methods in regulations and/or in contracts between several parties. Therefore, a **known quality** is considered as **vital prior to publishing** an environmental test method as a standard. Hence a general need for validation tasks interacts with the elaboration of the draft standards, and so there is also a general need to document the performed validation tasks and their results in the standard.

This document focuses on the validation tasks in the standardisation process of reference methods, being either the whole measurement process or one of its constituent parts.



Flow chart of the validation tasks in the standardisation process

Introduction

View of the Environmental TCs on validation

This paper is intended by the CEN-SABE Environmental TCs Cooperation Team to be a **policy document on validation**. It defines the view of the Environmental TCs on the role of validation in the process of the standardisation of environmental test methods.

Consequently, this document focuses on the 'why' and 'what' of validation in direct relation to the different steps of the standardisation process. Given the **policy aim** of the document, this document does not contain detailed procedures for performing the validation (such as number of laboratories, number of samples, etc.).

Uncertainty

The tests results in the environmental fields are often applied for the enforcement of regulation or for contract execution. In such legal situations it is vital that the associated uncertainties[1] in the tests' results are known.
The relation between the test result (TR) and the uncertainty (U) is generally presented as TR ± U. When the regulatory or contractual limit value is above TR + U or below TR – U, the conclusion is clear, respectively fulfilling or exceeding the limit value. If the limit value lies between these two boundaries, it is not possible to come to a clear conclusion.

This is an even bigger problem when the associated uncertainty is unknown, as, despite the test result itself, it is impossible to ascertain that the test result is really above or below the limit value.

Validation of an environmental test method is aimed at providing sound information on the uncertainty of the tests' results, and by that means providing the possibility to come to sound conclusions based on the standardised measurements (see in bibliography the IPPC-REF document on monitoring).

Request from CEN/SABE

Resolution 26/2004 of SABE taken on 19 October 2004 invites all environmental TCs to establish their own policy regarding the publication of validated or non-validated standards. The environmental TCs agreed that a commonly developed policy on validation would be preferable, therefore giving the lead for the development of this policy statement to the Environmental TC Cooperation Team.

In relation to its request, SABE wished to highlight two issues:

- there may be a financial liability if action is taken on the basis of a CEN document, and as such the uncertainty of the test result should be known;
- the subject of uncertainty influences the credibility of CEN.

Indeed, these two points are fully recognised by the environmental TCs.

Views within CEN

[1] Uncertainty: 'A parameter, associated with the result of a measurement that characterises the dispersion of the values that could reasonably be attributed to the measurand' (VIM and GUM).

When developing a policy view on validation, benefit should be taken of already established policies on validation within other CEN-sectors.

In general, the common view is that a test method can only be published as an EN when fully validated (first and second validation steps have been performed). It may happen that the results are considered by the WG expert as very poor and that they recommend to the TC to publish a TS instead. When no or only partial (e.g. first step) validation results are available at the time of completion of the CEN enquiry, the test method is to be published as a Technical Specification (TS).

When (partial) validation has been performed, the resulting performance characteristics are to be included in a separate section of the test method (specific clause or annex performance characteristics).

Consideration should be given to ENV 13005 Guide on Uncertainty in Measurement (GUM). It is to be noted in EN-ISO 17025 that 'General requirements for the competence of testing and calibration laboratories' requires that laboratories provide results with the associated uncertainties.

Major impact

There are different interpretations of the term ‘validation’, even within the environmental fields. However, there is a consensus that ‘validation’ is a key step in the standardisation process of environmental tests' methods.

Consequently, the definition of the term ‘validation’ has a major impact on the quality of the standards that describe a test method.

Defining a common policy on validation within the environmental sector of CEN also has, in turn, an impact on the work of the environmental TCs.

General principle within the environmental sector

The previously mentioned common view within CEN is embraced by the environmental TCs. This implies that only validated test methods can be published as EN standards. Test methods that are not or only partly validated are to be published as TSs. At the same time, the environmental TCs recognise the fact that not all standards are indeed test methods and, therefore, there might be a necessity to differentiate this general principle to some extent.

This policy document aims to clarify in which situations validation is to be considered as essential, and in which cases it is of less or of no importance. In addition, the validation activities during the different steps of the standardisation process are clarified.

Whenever there is a deviation from the general principle to publish validated standards, this should be a conscious decision of the involved TC.

Validation of the whole measurement[2] process

For the user / the customer of a standard or a series of standards, the reliability of the final overall result of a test is of major importance. That is the reliability that is obtained through all steps of the measurement process. Consequently, validation should not be just aimed at a single step of that procedure (like the analysis), but indeed should be aimed at quantifying the uncertainty that is associated with the full test procedure.

Depending on the matrix and the components that are to be assessed, this whole measurement process can be relatively simple or very complex. At least for part of the measurements in the environmental field, the whole

[2] i.e. sampling plan, taking of sample, sample pre-treatment in the field, packaging, storage and transportation, storage and conservation, sample pre-treatment, extraction, destruction, leaching, clean-up, analysis / quantification, data management, report

measurement process involving the appliance of a series of standards and full validation of the whole measurement process is not that simple. Therefore, this document starts with the validation of the individual steps of the whole measurement process, like the analytical determination of the content, and only after that will look at the validation of the whole measurement process.

1 Scope

This document provides guidance **on the validation tasks in the standardisation process of environmental test methods**.

It deals with the two main steps of such validation tasks, the robustness testing and the interlaboratory testing (determining the repeatability and reproducibility), both interacting with the elaboration of the draft standards as shown in the flow chart given in clause 2.1. It applies to the different inter-related phases of the environmental test methods, typically sampling and the production of a laboratory sample, storage and transportation of the laboratory sample, extraction, analysis or quantification of a test portion and finally reporting. Consequently, this document focuses on the 'why' and 'what' of validation tasks in direct relation to the different steps of the standardisation process. This document is focussed on the validation tasks in the standardisation process of reference methods either for the whole measurement process or for one of its constituent parts.

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

2 Validation of reference methods

2.1 General

In this document reference methods are test methods that have been validated and of which the quality of the test method is, given a specific field of application, accepted by experts and users. It might be the experts that state that the method is a reference method, but in general, the claim that it is a reference method is not made within the standardisation process. In order to have knowledge on the quality of the method and accepting that, information on the quality is essential in order to be defined as a reference method. Validation is therefore an essential step in the standardisation process from which this method originates.

Reference methods can be used as a legal reference in legislation/regulation and/or in contracts between two or more parties. They need, therefore, to be self supportive. Reference methods are not necessarily of the highest metrological quality, however, experts define a reference method as 'reliable' and a good basis for decisions. In general, reference methods are 'fit for purpose'.

Standardised reference methods are developed for 'common and repeated use'. They are not of the same nature as the utmost metrological quality that is required for Certified Reference Material developed in National Reference Laboratories (BAM, LNE, NPL….etc).

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

- robustness testing;
- interlaboratory testing (repeatability and reproducibility)

As the first step is based on a first draft of the standard and each of the validation steps will result in a revised draft standard, the implementation of validation in the standardisation process normally relates to three different draft standards, the last one of which will be published as an EN-standard. These steps are depicted in Figure 1.

It is to be noted that the actual state of the art may not be sufficient for the efficient further development of the envisaged standard. In such a case, a so-called pre-normative research may be needed prior to any standardisation with validation.

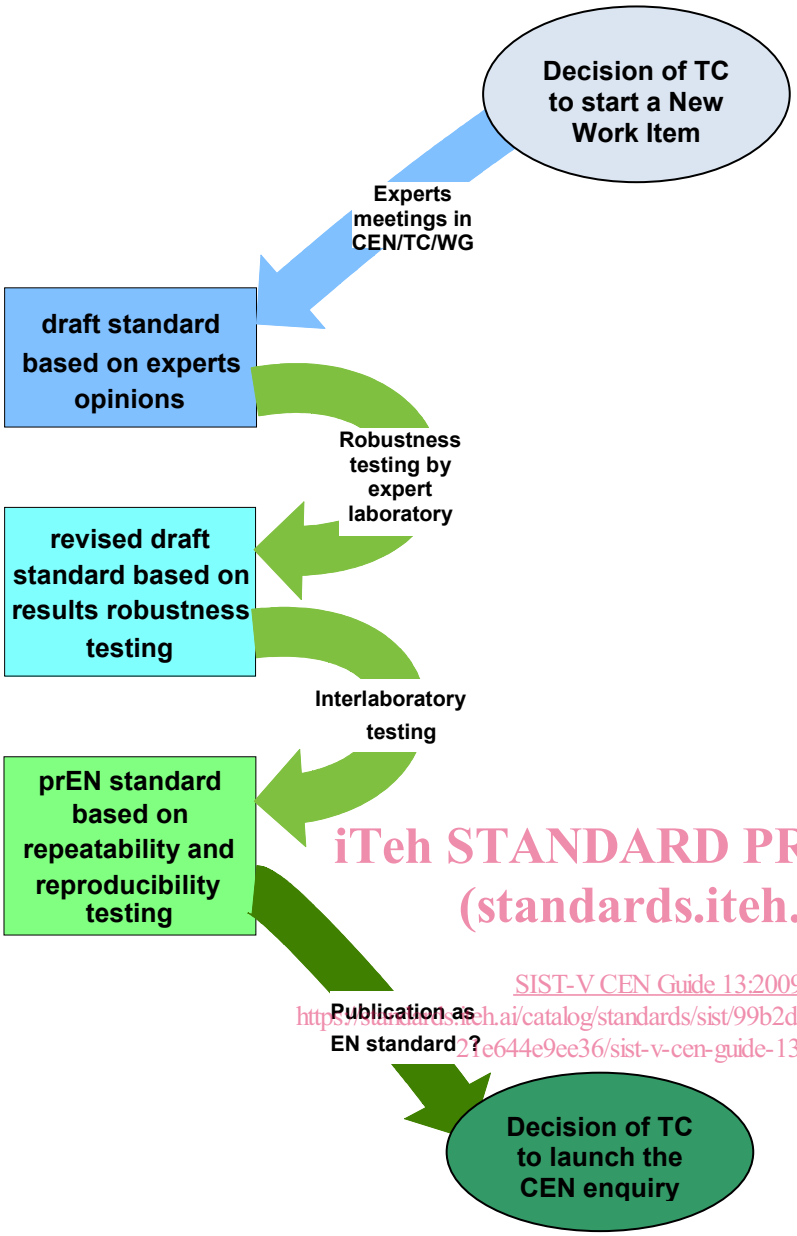


Figure 1: Flow chart of the validation tasks in the standardisation process

The robustness testing is generally performed in one highly competent laboratory which ideally already has experience with the new test method. The repeatability and reproducibility are determined through inter-laboratory experiments. Both steps are needed and contribute to the evaluation of the uncertainty of the test results.

In order to secure comparable data, the associated uncertainty of the test methods will often be based on traceability to SI units[3]. However, this is not, per definition, possible in all cases. Alternatively, the quantification of the uncertainty can also be based on the analysis of certified reference materials. Indeed, in the environmental

[3] e.g. weighing traceable to kg SI unit