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Guideline for the validation of physico-chemical analytical methods

Lignes directrices pour la validation des méthodes d'analyse physico-chimiques

Anleitung zur Validierung physikalisch-chemischer Analysenverfahren

This Technical Specification (CEN/TS) was approved by CEN on 9 November 2020 for provisional application.

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European foreword

This document (CEN/TS 16800:2020) has been prepared by Technical Committee CEN/TC 444 "Environmental characterization of solid matrices", the secretariat of which is held by NEN.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN shall not be held responsible for identifying any or all such patent rights.

This document supersedes CEN/TS 16800:2015.

The main changes compared to the previous edition are listed below:

— the scope has been extended from water only to water and environmental solid matrices, thus the document has been modified accordingly;

— a protocol for spiking of solid matrices has been added in an informative annex.

According to the CEN/CENELEC Internal Regulations, the national standards organisations of the following countries are bound to announce this Technical Specification: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Republic of North Macedonia, Romania, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

Introduction

Environmental monitoring of chemical substances is increasingly carried out within a European framework, and there is concern about the comparability of data at the European level. Methods used for the monitoring of substances with recent interest have often not been properly validated either in-house (i.e. within a single laboratory) or at the international level.

These issues may be addressed by adopting a harmonized approach towards method development and validation. The main objective of this document is to provide a common European approach to the validation of chemical methods for the respective monitoring of chemical substances in a broad range of matrices. Although the development of this approach was triggered by the needs for monitoring of emerging pollutants, it is of general nature and can be applied to the measurement of the concentration of a wide range of substances in a variety of matrices.

This guidance considers the different requirements for the level of method maturity and validation at different stages of the investigation or regulation of chemical substances.

This protocol will guide the user through the following steps:

— classification of existing methods with respect to their status towards validation, and the selection of the appropriate validation approach;

— development of a method to extend its application; for example, if a method for determining a required target compound in a selected matrix is available, but is not suitable for the same compound in a different matrix of interest;

— the validation procedures to be undertaken to effectively demonstrate the validation status of a selected method according to the approach adopted.

To agree on the use of one method, or several similar methods, in a trans-border or a multi-metrological context, and allow comparison of the results reported by several data producers on the same location (water quality measured on both bank of the same river, or soil composition measured on both sides of a border, or continuity of quality assessment of waste after measurement provider, e.g.), the procedure of establishing the LOQ of the measurement method must be clearly published.

The LOQ may result of:

— statistical evaluation of repeated measurements of a blank sample or a sample with a low concentration of the compound of interest (LOQ);

— experimental verification with a spike matched matrix that the LOQ meets accuracy validation criteria (LOQ-V).

Many (national and international) standards currently contain in their scope a statement like “this method is applicable from a concentration level of xx µg/l or yy mg/kg dry matter”, without any statement how this concentration level was established. When the limit of quantification is evaluated (LOQ) or verified (LOQ-V) using the procedure of this guideline, there is a possibility that it does not meet the lower limit of the claimed range.

Also, the LOQ and LOQ-V might be different depending on the analytical method. Therefore, if criteria are set to the LOQ of a method, it is necessary to clarify if LOQ or LOQ-V is meant.

1 Scope

This document describes an approach for the validation of physico-chemical analytical methods for environmental solid matrices and water.

The guidance in this document addresses the initial description of the method and two different validation approaches, in increasing order of complexity. These are:

a) method development, if the method is developed by the laboratory, or conditions of adoption, if the method is a standardized protocol adopted by the laboratory;

b) validation at the level of single laboratories (within-laboratory validation);

c) method validation at the level of several laboratories (between-laboratory or inter-laboratory validation), with a focus on methods that are sufficiently mature and robust to be applied not only by a few expert laboratories but by laboratories operating at the routine level.

The concept is strictly hierarchical, i.e. a method shall fulfil all criteria of within-laboratory validation before it can enter the validation protocol of the between-laboratory.

This document is applicable to the validation of a broad range of quantitative physico-chemical test methods for the analysis of water (including drinking water, surface water, groundwater, waste water, marine water), and of solid environmental matrices, such as soil, sludge, liquid and solid waste, sediment and biota. It is intended for standardized protocols adopted by a laboratory, and either for test methods aiming at substances that have recently become of interest or for test methods applying recently developed technologies.

The minimal requirements that are indispensable for the characterization of the fitness for the intended purpose of an analytical method are: selectivity, precision, trueness, performances characteristics and measurement uncertainty. The aim of validation is to prove that these requirements are met.

In this document after the definitions (Clause 3) and description of the principles (Clause 4) a toolbox is given describing the relevant performance characteristics in the validation process.

Clause 7 and 8 focus on the within laboratory validation process (V1) and Clause 9 on the interlaboratory validation process (V2). Clause 7 and 8 describe largely the same processes, but differ in approach for establishing the LOQ.

Reporting of the results of the validation studies is addressed in Clause 10.

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/IEC Guide 99:2007, *International vocabulary of metrology — Basic and general concepts and associated terms (VIM)*