SLOVENSKI STANDARD

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Kakovost vode - Metode za več spojin - 2. del: Merila za kvantitativno določevanje organskih spojin z analizno metodo za več spojin (ISO/DIS 21253-2:2018)

Water quality - Multi-compound class methods - Part 2: Criteria for the quantitative determination of organic substances using a multi-compound class analytical method (ISO/DIS 21253-2:2018)

Wasserbeschaffenheit - Multi-Class-Verfahren - Teil 2: Anleitung für die Beurteilung eines analytischen Verfahrens für mehrere Gruppen organischer Stoffe (ISO/DIS 21253-2:2018)

Qualité de l'eau - Méthodes d'analyse de composés multi-classes - Partie 2: Critères pour la détermination quantitative de substances organiques avec une méthode d'analyse de composés multi-classess (ISO/DIS 21253-2:2018)

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Water quality — Multi-compound class methods —

Part 2:
Criteria for the quantitative determination of organic substances using a multi-compound class analytical method

Qualité de l'eau — Méthodes de composés multi-class —

Partie 2: Critères pour l'évaluation quantitative d'une méthode d'analyse de composés multi-class des substances organiques

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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.

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 documents 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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For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 147, *Water quality*, Subcommittee SC 2, *Physical, chemical and biochemical methods*.

A list of all parts in the ISO 21253- series can be found on the ISO website.

Water quality — Multi-compound class methods —

Part 2:
Criteria for the quantitative determination of organic substances using a multi-compound class analytical method

1 Scope

This document specifies the critical points to consider while developing an in-house mass spectrometry-based method for quantitative analysis of multi-group organic substances in the scope of physical-chemical analysis of water.

ISO/TS 13530 provides guidance on the initial characterization of the measurement performances. This document supplements ISO/TS 13530 by providing details on the following points:

- selection of the characterization-test matrix;
- internal standards and internal standard recoveries;
- analyte recovery;
- checking that performance levels do not degrade over time.

Chromatographic separation and identification of analytes are described in ISO/DIS 21253-1*.*

The estimation of measurement uncertainties is integral to the characterization of any quantitative method of analysis, but the process is not described here, as ISO 11352 applies. Similarly, the calibration protocols are not described here but set out in the ISO 8466- series of standards.

This document is not intended as a substitute for the currently applicable analytical standards dedicated to organic compounds but as a resource bringing additional characterization elements.

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 5667-3, *Water quality — Sampling — Part 3: Preservation and handling of water samples*

ISO 8466-1, *Water quality — Calibration and evaluation of analytical methods and estimation of performance characteristics — Part 1: Statistical evaluation of the linear calibration function*

ISO 8466-2, *Water quality — Calibration and evaluation of analytical methods and estimation of performance characteristics — Part 2: Calibration strategy for non-linear second-order calibration functions*

ISO 11352, *Water quality — Estimation of measurement uncertainty based on validation and quality control data*

ISO 12787, *Cosmetics — Analytical methods — Validation criteria for analytical results using chromatographic techniques*

ISO/TS 13530, *Water quality — Guidance on analytical quality control for chemical and physicochemical water analysis*

ISO/DIS 21253-1, *Water quality — Multi- compound class methods — Part 1: Criteria for the identification of target compounds by gas and liquid chromatography and mass spectrometry*

3 Terms and definitions

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

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

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

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

3.1
analyte
substance to be determined[SOURCE: ISO/TS 28581:2012,[5] 3.1]

3.2
blank
aliquot of reagent water (reagent blank) or of a matrix in which the analyte is absent (matrix blank) that is treated exactly as a sample through the complete analytical procedure including extraction, clean-up, identification and quantification including all the relevant reagents and materials

Note 1 to entry: It is crucial that the laboratory specifies which blank is considered.

3.3
isotope dilution
method using labelled (usually ^{13}C) internal standards to correct for losses during sample preparation and analysis

Note 1 to entry: Calculation of the ratio of characteristic signal intensity between molecule and labelled analogue makes it possible to calculate the quantitative ratio between compound and labelled compound and thereby to deduce compound concentration in the sample.

[SOURCE: ISO 17858:2007,[4] 3.1.9, modified — Note 1 to entry added]

3.4
scope of the analytical method
combination of the different types of matrix, substances and analyte concentration range covered, for which the analytical method applies

3.5
maximum allowed tolerance
upper and lower-bound extremes of acceptable range of a measurement result

Note 1 to entry: The maximum allowed tolerance can be imposed by normative or regulatory requirements or set by the customer, or by the user itself.

3.6
retention time standard
compound that is added to the sample (or to the sample extract) and to the external standard solution, and whose retention time is used to calculate the relative retention times of the target compounds

Note 1 to entry: The retention time standard may be exactly the same as the internal standard or standards.

[SOURCE: ISO 15680:2003,[2] 3.4]

3.7
calibration curve
expression of the relation between indication and corresponding measured quantity value calibration curve[SOURCE: ISO/IEC Guide 99:2007(en),[6] 4.31]

3.8
limit of detection
lowest amount or concentration of an analyte in a test sample that can be reliably distinguished from the absence of that substance

3.9
limit of quantification of the method (LoQ)
smallest quantity of an analyte in a sample that may be assayed quantitatively under the experimental conditions described in the method to a specified level of accuracy

3.10
analytical method
unambiguously written procedure describing all details required to carry out the analysis of the determinand or parameter, namely: scope and field of application, principle and/or reactions, definitions, reagents, apparatus, analytical procedures, calculations and presentation of results, performance data and test report[SOURCE: ISO/TS 16489:2006,[3] 3.3]

3.11
recovery (or relative revovery)
extent to which a known, added quantity of determinand in a sample can be measured by an analytical systemRecovery is calculated from the difference between results obtained from a spiked and an unspiked aliquot of sample and is usually expressed as a percentage.[SOURCE: ISO 5667-14:2014,[1] 3.8]

3.12
relative retention time
ratio between the retention time of the target compound and the retention time of the retention-time calibration standard[SOURCE: ISO 15680:2004,[2] 3.5]

3.14
injection standard
compound added to the sample and the calibration standard solution and used to verify that instrument-based step of the analytical method runs properlyThe following terms and definitions are indispensable for the application of this document.

3.15
substances group
analytes pre-classified into groups that share similar patterns of analytical behaviour in response to extraction mode and analysis

3.16
internal standard
compound added in a known amount to the sample from the beginning of the protocol and enabling analytical coverage throughout the procedure, and that is used to correct for losses during sample preparation and analysis by accounting for all-system matrix effects (recoveries, ionization effect, variability of the detector response of the instrument for example)

Note 1 to entry: Determining the ratio of characteristic molecule signal intensity to the internal standard makes it possible to calculate the quantitative ratio between molecule and internal standard and thereby to deduce molecule concentration in the sample. An identical amount is also added to the calibration standard solutions.

3.17
Yield (or absolute recovery)
amount of analyte added in the test sample corrected by the relative recovery of the internal standard (analyte-to-internal standard ratio)

Note 1 to entry: Yield is a value that accounts for both sample matrix effect and compound recovery.

4 Principle

This document specifies the critical points to be considered when developing a mass spectrometry-based method for the analysis of multi-class organic substances in water samples. The critical points addressed here involve:

- selection of the matrix;
- internal standards and internal standard recoveries;
- analyte recovery.

This document then goes on to propose a rating-scale method for analytical protocols dedicated to huge numbers of compounds, making it possible to give the method a reliability score for each compound. When a review of these critical points concludes that the method can reliably quantify the target analyte or analytes, then the initial characterization of the measurement performances of the method can be started.

This document concludes by proposing quality assurance criteria that should be made routine to ensure that performance levels do not degrade over time.

Should the method's initial list of reliably quantifiable analytes be expanded, then verify the analogue transitions, retention times, LoQ performances and accuracy. Should the method's performances for the previously characterized analytes be modified, then the characterization of the method shall be updated to integrate the new performance levels achieved.

For each substance, the method can only be claimed as reliable for those matrices that were effectively tested during the characterization process.

Should one or more purification steps need to be carried out, if substances are present that are liable to create interferences in the chromatogram or contaminate the analytical system, then the measured yield for each substance shall account for the purification step(s).

5 Selection of the matrix

The method shall be characterized on a matrix that is representative of the claimed scope, as described e.g. in CEN/TS 16800[7] or Eurachem Guide.[8]

The water matrix is characterized by its source (marine water, waste water, groundwater, drinking water, etc.) and its physical-chemical properties (SPM, TOC, conductivity, pH, major ions, etc.). These parameters shall be identified and recorded.

The representative matrix can be natural or synthetic. Laboratory-grade water (distilled water, deionized water) is not representative of any natural water.

In the absence of any real analyte-free matrix (especially for the characterization of the LoQ), then the natural matrix can be diluted (with drinking water or laboratory-grade water, e.g.) in order to lower the concentration of organic compounds, taking care to appropriately adjust the modified properties (SPM, TOC, conductivity, pH, etc.) as specified in ISO/DIS 21253-1, 7.1.

6 Sample preservation prior to analysis

In the absence of normative requirements expressly covering the analytes, holding time and sample preservation mode shall be defined and verified end-to-end from collection of the sample through to completion of all analysis-related operations. The holding time defined commences at the date of sample collection.

Sample preservation integrity tests shall be conducted on the representative matrix (Clause 5) and, where appropriate, in presence of the preservative. They shall be conducted at least one level at or near 10 × LoQ or at the regulatory value, with field/laboratory duplicates; ISO 5667-3 provides a