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Water quality — Determination of microcystins — Method using liquid chromatography and tandem mass spectrometry (LC-MS/MS)

Qualité de l'eau — Dosage des microcystines — Méthode par chromatographie en phase liquide couplée à la spectrométrie de masse en tandem (CL-SM/SM)

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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).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of 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 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*.

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.

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WARNING — Persons using this document should be familiar with normal laboratory practice. This document does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices.

IMPORTANT — It is absolutely essential that tests conducted in accordance with this document be carried out by suitably qualified staff.

1 Scope

This document specifies a method for the quantification of twelve microcystin variants (microcystin-LR, -LA, -YR, -RR, -LY, -WR, -HtyR, -HilR, -LW, -LF, [Dha^{7}]-microcystin-LR, and [Dha^{7}]-microcystin-RR) in drinking water and freshwater samples between 0,05 µg/l to 1,6 µg/l. The method can be used to determine further microcystins, provided that analytical conditions for chromatography and mass spectrometric detection has been tested and validated for each microcystin. Samples are analysed by LC-MS/MS using internal standard calibration.

This method is performance based. The laboratory is permitted to modify the method, e.g. increasing direct flow injection volume for low interference samples or diluting the samples to increase the upper working range limit, provided that all performance criteria in this method are met.

Detection of microcystins by high resolution mass spectrometry (HRMS) as an alternative for tandem mass spectrometry (MS/MS) is described in Annex A.

An alternative automated sample preparation method based on on-line solid phase extraction coupled to liquid chromatography is described in Annex B.

When instrumental sensitivity is not sufficient to reach the method detection limits by direct flow injection, a solid phase extraction clean-up and concentration step is described in Annex C.

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 3696, *Water for analytical laboratory use — Specification and test methods*

3 Terms and definitions

No terms and definitions are listed in this document.

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/