

ISO 22104:2021 (E)

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

Contents

	Foreword
1	Scope
2	Normative references
3	Terms and definitions
4	Principle
5	Interferences
5.1	Biases
5.2	Limitations
6	Reagents and standards
6.1	General
6.2	Preparation of solutions
7	Apparatus
8	Sampling
9	Procedure
9.1	Preparation of samples
9.1.1	General
9.1.2	Preparation of method blank sample
9.1.3	Preparation of laboratory control spike sample
9.1.4	Preparation of calibration control sample
9.1.5	Preparation of calibration standard solutions
9.1.6	Preparation of drinking water and freshwater sample
9.1.7	Sample preparation procedure with freeze/thaw cycles
9.2	Instrumental analysis by LC-MS/MS procedure
9.2.1	Instrument set-up parameters
9.2.1.1	Typical liquid chromatograph (LC) parameters
9.2.1.2	Typical mass spectrometer (MS) conditions
9.3	Run processing and quality assurance
9.3.1	Run sequence
9.3.2	Run control operations / limits
9.3.2.1	Instrument check mix (high) solution
9.3.2.2	Low level method recovery (MR2) sample
9.3.2.3	Method blank measurements
9.3.2.4	Laboratory control spike sample measurements
9.3.2.5	Calibration control sample measurements
9.3.2.6	Replicate samples measurements
10	Calibration
11	Evaluation and calculation of results
11.1	Identification and calculations
11.2	Calibration curve equation determination
11.3	Internal standard calculation
11.4	Internal standard recovery calculation

12 **Expressing of results**

13 **Test report**

Annex A (informative) Use of high resolution mass spectrometry detectors (HRMS)

- A.1 Scope**
- A.2 Principle of the method**
- A.3 Interferences**
- A.4 Apparatus**
- A.5 Typical high resolution mass spectrometer conditions**
- A.6 Mass spectrometric acquisition**

Annex B (informative) Use of online solid phase extraction coupled to liquid chromatography for the automated analysis of microcystins

- B.1 Scope**
- B.2 Principle**
- B.3 Reagents and standards**
- B.3.1 General**
- B.3.2 Preparation of solutions**
- B.4 Apparatus**
- B.5 Procedure**
- B.5.1 Sample preparation procedure**
- B.5.2 Automated instrumental analysis by online solid phase extraction coupled to liquid chromatography**
- B.5.2.1 Online SPE chromatograph set-up parameters**
- B.5.2.2 Online SPE liquid chromatograph set-up procedure**
- B.5.2.3 Analysis procedure**
- B.5.3 Run processing and quality assurance**
- B.5.3.1 Run sequence**
- B.5.3.2 Run control operations / limits**
- B.5.3.3 Methanol injections**

Annex C (informative) Use of manual solid phase extraction prior to instrumental analysis for improved method detection limits

- C.1 Scope**
- C.2 Principle**
- C.3 Reagents and standards**
- C.3.1 General**
- C.3.2 Preparation of solutions**
- C.4 Apparatus**
- C.5 Sampling**
- C.6 Procedure**
- C.6.1 Preparing samples**
- C.6.1.1 General**
- C.6.1.2 Preparing method blank sample**
- C.6.1.3 Preparing laboratory control spike sample**
- C.6.1.4 Preparing calibration control sample**
- C.6.1.5 Preparing calibration standard solutions**
- C.6.1.6 Preparing drinking and fresh water samples**
- C.6.2 Sample preparation procedure**

Page count: 32