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INDUSTRY STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

HY/T 0319-2021

Shellfish - Determination of lipophilic marine biotoxins - LC-MS/MS method

贝类 脂溶性海洋生物毒素的检测 液相色谱—串联质谱法

(English Translation)

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Foreword

This document is in accordance with the provisions of GB/T 1.1-2020 "Guidelines for Standardization Work Part 1: Structure and Drafting Rules of Standardization Documents" Drafting.

This document was proposed by the Ministry of Natural Resources of the People's Republic of China.

This document is under the jurisdiction of the National Marine Standardization Technical Committee (SAC/TC283).

Drafting organizations of this document. National Marine Environmental Monitoring Center, Institute of Oceanology, Chinese Academy of Sciences, Xiamen University, Chinese Academy of Fishery Sciences Yellow Sea Research Institute.

1 Scope

This document specifies the shellfish fat-soluble marine biological toxins Daejeon, Dinophyton, Saxitoxin, Ezo scallop toxin, Protodin Principles, operating procedures and

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result calculations of 11 toxin compounds such as acid toxins and cyclic imine toxins by liquid chromatography-tandem mass spectrometry.

This document is applicable to the detection of fat-soluble marine biological toxins in shellfish bodies.

2 Normative references

The content of the following documents constitutes an indispensable clause of this document through normative references in the text. Among them, dated quotations Only the version corresponding to the date is applicable to this document; for undated reference documents, the latest version (including all amendments) is applicable to this document.

GB/T 6682 Analyze laboratory water specifications and test methods.

3 Principle

Shellfish samples are extracted with methanol, hydrolyzed under alkaline conditions to release the esterified biological toxins, and purified with HLB solid-phase extraction column.

Chromatographic separation, tandem mass spectrometry determination, external standard curve method quantification.

4 Reagents and materials

4.1
Acetonitrile (CH3CN). chromatographically pure.

4.2
Methanol (CH3OH). chromatographically pure.

4.3
Formic acid (HCOOH).

4.4
Ammonium formate (HCOONH4).

4.5
Ammonia (NH3·H2O). concentration >= 25%.

4.6
Sodium hydroxide (NaOH).

4.7
Hydrochloric acid (HCL). concentration >=36%.

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4.8

Sodium hydroxide solution (2.5mol/L). accurately weigh 50g of sodium hydroxide, dilute to 500mL with water, and store at room temperature.

4.9

Hydrochloric acid solution (2.5mol/L). accurately measure 104.5mL of hydrochloric acid, dilute to 500mL with water, and store at room temperature.

4.10

Methanol solution (30%). Measure 30mL of methanol and add it to 70mL of water, mix well, at room temperature, and use it now.

4.11

Methanol solution (20%). Measure 20mL of methanol and add it to 80mL of water, mix well, at room temperature, and use it now.

4.12

Ammonia methanol solution (0.3%). Draw 0.3mL ammonia, dilute to 100mL with methanol, at room temperature, and use it now.

4.13

Fat-soluble marine biotoxin standards (see Appendix A) solutions. OA, DTX1, DTX2, PTX2, YTX, homoYTX, AZA1, the purity of AZA2, AZA3, GYM, SPX1 standard materials is >=97%, and it should be stored at -20°C in the dark.

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