



**NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

GB 31656.10-2021

**National food safety standard - Determination of metaldehyde
residues in aquatic products by liquid chromatography-tandem
mass spectrometry method**

食品安全国家标准 水产品中四聚乙醛残留量的测定 液相色谱-串联质谱法
(English Translation)

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Foreword

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

This document is published for the first time:

National food safety standards Determination of metaldehyde residues in aquatic products by liquid chromatography-tandem mass spectrometry

1 Scope

This document specifies the sample preparation and liquid chromatography-tandem mass spectrometry methods for detecting metaldehyde residues in aquatic products:

This document is applicable to the detection of metaldehyde residues in edible tissues of fish and shrimp:

2 Normative reference documents

The contents of the following documents constitute essential provisions of this document through normative citations in the text: Among them, the cited documents with dates are:

Only the version corresponding to the date applies to this document; for undated referenced documents, the latest version (including all amendments) applies to this document:

document:

GB/T 6682 Specifications and test methods for water used in analytical laboratories GB/T 30891-2014 Sampling specifications for aquatic products

3 Terms and definitions

There are no terms or definitions that need to be defined in this document:

4 Principles

The metaldehyde in the sample was extracted by acetonitrile homogenate, and the extract was salted out and purified using N-propylethylenediamine and C18 packing: The liquid chromatography G series Detection by mass spectrometry and quantification by internal standard method:

5 Reagents and materials

Unless otherwise specified, all reagents are of analytical grade and the water is first-grade water that complies with GB/T 6682:

5.1

Reagents

5.1.1

Acetonitrile (CH₃CN)

Chromatographically pure:

5.1.2

Formic acid (HCOOH)

Chromatographically pure:

5.1.3

Sodium chloride (NaCl):

5.1.4

Anhydrous magnesium sulfate (MgSO₄):

5.2

Preparation of solution 5:2:1 0.1% formic acid solution: Take 1mL of formic acid, add water to dissolve and dilute to 1000mL, and mix well:

5:2.250% acetonitrile solution: Pipette 100mL of acetonitrile, add 100mL of water, and shake well:

5.3

Standard products Metaldehyde and metaldehyde-D16 standard products, the content is $\geq 95\%$, see Appendix A:

5.4

Preparation of standard solution

5.4.1

Standard stock solution (100 $\mu\text{g/mL}$)

Take an appropriate amount of metaldehyde and metaldehyde-D16 (equivalent to 10 mg of active ingredient) and weigh them accurately:

Dissolve and dilute with acetonitrile respectively to a 100mL volumetric flask, shake well, and get it: Store below -18°C , valid for 6 months:

5.4.2

Standard intermediate solution (5.0 $\mu\text{g/mL}$): Precisely measure 0.5mL each of metaldehyde and metaldehyde-D16 standard stock solution into 10mL respectively:

In a volumetric flask, dilute with acetonitrile to the mark, shake well, and get it: Store in a dark place at 2°C to 8°C , and is valid for 1 month:

5.4.3

Standard working solution (0.025 $\mu\text{g/mL}$): Precisely measure 0.5 mL of metaldehyde and metaldehyde-D16 standard intermediate solutions respectively:

In a 10mL volumetric flask, dilute to the mark with acetonitrile, shake well, and get it: Ready to use:

5.5

Materials

5.5.1

N-propylethylenediamine (PSA) filler, 40 mesh to 60 mesh or equivalent:

5.5.2

C filler, 40 mesh to 60 mesh or equivalent:

6 Instruments and equipment

6.1

Liquid chromatography-tandem mass spectrometer

Equipped with automatic sampler and electrospray ion source:

6.2

Analytical balance

Sensitive to 0.01g and 0.00001g:

6.3

Homogenizer,

6.4

Centrifuge tubes

Polypropylene plastic centrifuge tubes, 10 mL and 50 mL:

6.5

Centrifuge

10000 r/min or above:

6.6

Vortex mixer:

7 Preparation and preservation of samples

7.1

Preparation of samples Prepare samples according to the requirements of Appendix B of GB/T 30891-2014:

- a) Take the homogenized test material as the test material;
- b) Take the homogenized blank sample as the blank sample;
- c) Take the homogenized blank sample, add the standard working solution of appropriate concentration, and add the sample as a blank:

7.2

Storage of samples Store below -18°C and conduct analysis and testing within 3 months:

8 Measurement steps

8.1

Extraction

Take 5 g of the sample (accurate to ± 0.05 g), put it in a 50 mL centrifuge tube, add 100 μ L of metaldehyde-Ds standard working solution (0.25 μ g/mL), then add 2.0 mL of water and 10.0 mL of acetonitrile in sequence, and stir at 10000 r/min speed for 1 minute, add 2g of sodium chloride, cover and vortex for 1 minute, then centrifuge at 5000r/min for 2 minutes, collect the supernatant and set aside: