



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

GB 31659.4-2022

**National Food Safety Standard Determination of Abamectin
Drug Residues in Milk and Milk Powder by Liquid
Chromatography-Tandem Mass Spectrometry**

食品安全国家标准 奶及奶粉中阿维菌素类药物残留量的测定 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 for Standardization Documents" drafting.

This document replaces GB/T 22968-2008 "Ivermectin, Abamectin, Doramectin and Acetaminoabamectin in Milk and Milk Powder Determination of residues by liquid chromatography-tandem mass spectrometry", compared with GB/T 22968-2008, except for structural adjustments and editorial changes, the main The changes are as follows.

- The standard text format is changed to the national food safety standard text format;
- The detection of goat milk has been added to the scope of the standard;
- The standard determination substance is changed to the residual marker of each drug;
- Standard sensitivity is further improved.

The release status of previous versions of this document and the documents it replaces are as follows.

- GB/T 22968-2008.

1 Scope

This document specifies the sample preparation and liquid chromatography-tandem mass spectrometry method for the detection of avermectin drug residues in milk and milk powder.

This document is applicable to abamectin (calculated as avermectin B1a) and ivermectin (calculated as 22,23-dihydroabamectin) in milk, goat milk and milk powder B1a), doramectin and acetaminobamectin (calculated as acetaminobamectin B1a) single or multiple drug residue detection.

2 Normative references

The content in the following documents constitutes the essential provisions of this document through normative references in the text. Among them, the dated reference documents are only The version corresponding to the date applies to this document; for undated references, the latest version (including all amendments) applies to this document.

GB/T 6682 Analytical laboratory water specifications and test methods

3 Terms and Definitions

This document does not have terms and definitions that need to be defined.

4 Principles

The remaining avermectins in the test samples were extracted with acetonitrile, purified by C18 solid phase extraction column, determined by liquid chromatography-tandem mass spectrometry, and the matrix matched Quantification by standard solution external standard method.

5.1 Reagents

The reagents used in the following are all analytical reagents unless otherwise specified, and the water is the first-class water in accordance with the provisions of GB/T 6682.

5.1.1

Acetonitrile (CH₃CN). chromatographically pure.

5.1.2

Methanol (CH₃OH). chromatographically pure.

5.1.3

Formic acid (CH₂O₂). chromatographically pure.

5.1.4

Triethylamine (C₆H₁₅N).

5.1.5

Isooctane (C₈H₁₈).

5.2 Solution preparation

5.2.11 0.1% formic acid in acetonitrile solution. Take 1 mL of formic acid and dilute it to 1000 mL with acetonitrile.

5.2.2 0.1% formic acid solution. Take 1mL of formic acid and dilute it with water to 1000mL.

5.2.3

Rinse solution. take 30 mL of acetonitrile, 70 mL of water and 20 µL of triethylamine, and mix them evenly.

5.2.4 50% acetonitrile solution. Take 50mL of acetonitrile and dilute with water to 100mL.

5.3 Standards

Avermectin (Avermectin, C₄₈H₇₂O₁₄.C₄₇H₇₀O₁₄, CAS number. 71751-41-2), content ≥ 98.0%; ivermectin (Iver-mectin, C₄₈H₇₄O₁₄, CAS number. 70288-86-7), content ≥ 98.0%; doramectin (C₅₀H₇₄O₁₄, CAS number.

117704-25-3), content ≥ 94.3%; acetamidoabamectin (Eprinomectin, C₅₀H₇₅NO₁₄, CAS number. 123997-26-2), content ≥ 98.0%.

5.4 Preparation of standard solution

5.4.1

Standard stock solution of abamectin drugs. Accurately weigh abamectin (calculated as avermectin B1a), ivermectin (calculated as 22,23-dihydro Abamectin B1a), doramectin and acetamidoabamectin (calculated as acetamidoabamectin B1a) each about 10 mg standard, in different In the same 10mL volumetric flask, dissolve it with acetonitrile and adjust the volume to the mark to prepare avermectin B1a, 22,23-dihydro-alpha Vermectin B1a, doramectin and acetamidoabamectin B1a standard stock solution. Stored below -18 °C, valid for 12 months.

5.4.2

Standard working solution of abamectin drugs. Accurately measure 1 mg/mL of abamectin B1a, 22,23-dihydroabamectin B1a, dora 100 µL of standard stock solution of mycocin and acetamidobamectin B1a was diluted to the mark with acetonitrile in different 10mL volumetric flasks, and prepared into Abamectin B1a, 22,23-dihydroabamectin B1a, doramectin and acetamidoabamectin B1a standard lab with a concentration of 10 µg/mL As

solution. Store below -18°C, valid for 6 months.

5.5 Materials

5.5.1

C18 solid phase extraction column. 500mg/6mL, or equivalent.

5.5.2

Hydrophilic polytetrafluoroethylene microporous membrane. 0.22 µm.

6 Instruments and equipment

6.1

Liquid chromatography-tandem mass spectrometer. equipped with electrospray ionization source (ESI).

6.2

Analytical balance. Sensitivity 0.00001g and 0.01g.

6.3

Vortex mixer.

6.4

High speed centrifuge.

6.5

Nitrogen blowing instrument.

6.6

Horizontal oscillator.

7.1 Preparation of samples

Take an appropriate amount of fresh or thawed blank or test milk or goat milk and milk powder, and mix well.

- a) Take the homogenized test sample as the test sample;
- 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.