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NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB 31658.24-2022

National food safety standards-Determination of seduromycin residues in animal foods by liquid chromatography-tandem mass spectrometry

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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 is published for the first time.

National Food Safety Standards

Determination of seduromycin residues in food of animal origin

Liquid chromatography-tandem mass spectrometry

1 Scope

This document specifies the ultra-high performance liquid chromatography-tandem mass spectrometry method for the determination of seduromycin in food of animal origin.

This document is applicable to the determination of sedumecin residues in chicken muscle and liver tissue.

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,

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

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 residual seduramycin in the sample was extracted with acetonitrile, purified by solid-phase extraction column, detected by ultra-high performance liquid

chromatography-tandem mass spectrometry, and external standard method.

Quantitative.

5 Reagents and materials

Unless otherwise specified, all reagents are of analytical grade; water is grade-1 water in accordance with GB/T 6682.

5.2 Solution preparation

5.2.1 50% acetonitrile solution. take 50mL of acetonitrile, dissolve in water and dilute to 100mL, mix well.

5.2.2 80% dichloromethane methanol solution. Take 80mL of dichloromethane, add 20mL of methanol, and mix well.

5.3 Standards

Seduramicin (Semduramicin, molecular formula. C45H75O16, CAS number. 113378-31-7), content >=94.3%.

5.4 Preparation of standard solution

5.4.1 Standard stock solution. Accurately weigh the reference substance equivalent to 10 mg of seduromycin, dissolve it with acetonitrile and dilute it in a 10 mL volumetric flask,

Prepare seduramycin standard stock solution with a concentration of 1 mg/mL, store it below -18 degrees C, and have a validity period of 3 months.

5.4.2 10 μg/mL seduromycin standard working solution. Accurately measure 0.1 mL of the standard stock solution into a 10 mL volumetric flask, add 50% acetonitrile

The aqueous solution was diluted to the mark, and prepared into 10 μg/mL seduromycin standard working solution. Stored at 2°C~8°C, the validity period was 1 month.

5.4.3 1 μg/mL seduamycin standard working solution. accurately measure 1.0 mL of 10 μg/mL seduamycin standard working solution in a volume of 10 mL

Dilute to the mark with 50% acetonitrile aqueous solution to prepare 1 μg/mL seduromycin standard working solution. Store at 2°C~8°C, valid for 1

month.

5.5 Materials

5.5.1 Solid phase extraction column. graphitized carbon black amino solid phase extraction column. 500mg/6mL.

6 Instruments and equipment

6.1 Ultra-high performance liquid chromatography-tandem mass spectrometer. with electrospray ionization source.

7.1 Sample preparation

Take an appropriate amount of fresh or thawed blank or test tissue, mince it, and homogenize it.

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.

7.2 Sample storage

Store below -20°C.

8.1 Extraction

Take 2 g of the sample (accurate to +/-0.05 g), add 12 mL of acetonitrile to a 50 mL centrifuge tube, vortex and mix for 1 min, shake for 10 min,

Centrifuge at 5000r/min for 5min (liver at 8000r/min), take the supernatant, add acetonitrile 12mL to repeat the extraction once, combine the two extracts,

spare.

8.2 Purification

The extraction column was activated with 5 mL of dichloromethane and 5 mL of acetonitrile in sequence, and the spare solution was taken and passed through the column at a controlled flow rate of 2 mL/min to 3 mL/min.

Drained for 10 min, eluted with 6 mL of 80% dichloromethane methanol solution, collected the eluate, and dried with nitrogen in a water bath at 40°C. Dissolved with 50% acetonitrile in water

The solution was dissolved and diluted to 2.0 mL, filtered, and determined by ultra-high performance liquid chromatography-tandem mass spectrometry.

8.3 Preparation of matrix-matched standard curve

Precisely measure 20 μL, 40 μL and 100 μL of 1 μg/mL sedumecin standard working solution, 10 μg/mL sedumecin standard working solution