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NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

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National food safety standards Determination of virginiamycin M1 residues in edible tissues of pigs and poultry by liquid chromatography-tandem mass spectrometry

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600 μg/kg, and sebum tissue 50 μg/kg-800 μg/kg, the recovery rate was 70%-120%.

10.3 Precision

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 virginiamycin M1 residues in edible tissues of pigs and poultry

Liquid chromatography-tandem mass spectrometry

1 Scope

This document specifies the sample preparation and liquid chromatography-tandem mass spectrometry for detection of virginiamycin M1 residues in edible tissues of pigs and poultry.

method.

This document is applicable to the determination of virginiamycin M1 residues in muscle, liver, kidney and skin + fat of pigs and poultry (including chicken, duck and goose)

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,

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 virginiamycin M1 in the sample was extracted with acetonitrile, degreased with n-hexane, detected by liquid chromatography-tandem mass spectrometry, and quantified by external standard method.

5 Reagents and materials

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

5.2.1 Acetonitrile solution. Take 600mL of acetonitrile and 400mL of water, and mix them evenly.

5.2.2 0.1% formic acid solution. take 1mL of formic acid, dilute it with water to 1000mL, and mix well.

5.3 Standards

Virginiamycin M1 (Virginiamycin M1, C28H35N3O7, CAS number. 21411-53-0) content was >=90%.

5.4 Preparation of standard solution

5.4.1 Standard stock solution. Take 10 mg of virginiamycin M1 standard product, weigh it accurately, dissolve it with acetonitrile and dilute to volume.

200mL brown volumetric flask, prepared into a standard stock solution of virginiamycin M1 with a concentration of 50μg/mL. Store below 4°C, and the expiration date is

7d.

5.4.2 Standard intermediate solution. Accurately measure 0.1mL, 0.5mL, 1mL, 2mL and 3mL of standard stock solution, and put them into 50mL brown container

Diluted to the mark with acetonitrile to prepare vitamins with concentrations of 0.1 μg/mL, 0.5 μg/mL, 1 μg/mL, 2 μg/mL and 3 μg/mL.

Geniamycin M1 standard intermediate solution. Store below 4°C, with a validity period of 7 days.

6 Instruments and equipment

6.1 Liquid chromatography-tandem mass spectrometer. distribution of electrospray ionization source.

7.1 Preparation of samples

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

a) Take the homogeneous test sample as the test sample;

b) Take a homogeneous blank sample as a blank sample;

c) Take a homogeneous blank sample, add standard working solution of appropriate concentration, and add the sample as a blank.

7.2 Storage of samples

Store below -40°C and avoid repeated freezing and thawing.

8.1 Extraction

Take 2 g of the sample (accurate to +/-0.05 g), add 4 mL of acetonitrile (add 2 mL of water to the muscle tissue first), vortex for 2 min, and sonicate for 30 min.

Centrifuge at 4000r/min for 10min, take the supernatant and transfer it to a 15mL centrifuge tube with a stopper. Add 2mL of acetonitrile to the residue and repeat the extraction once, and combine

The supernatant was set aside to obtain the extract.

8.2 Purification

Take the above extract, add water to about 9.5mL, vortex and mix for 30s, add n-hexane 3mL, vortex for 30s, centrifuge at 4000r/min for 10min,

Discard the upper layer of n-hexane, and repeat the degreasing once. Transfer the lower layer solution to a 10mL graduated test tube, add water to 10mL, vortex for 30s, and prepare

Use. Take the spare solution (liver. take 2.0mL of the spare solution, add 4.0mL of acetonitrile solution, mix well; kidney, sebum. take 2.0mL of the spare solution, add acetonitrile

solution (6.0 mL, mixed), filtered, and determined by liquid chromatography-tandem mass spectrometry.

8.3.1 Preparation of poultry muscle and liver matrix matching standard curve

Accurately measure virginiamycin M1 standards with concentrations of 0.1 μg/mL, 0.5 μg/mL, 1 μg/mL, 2 μg/mL and 3 μg/mL

Add.200 μL of each intermediate solution to the blank tissue extract after degreasing