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NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

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

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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 nitroimidazole drug residues in animal food

Liquid chromatography-tandem mass spectrometry

1 Scope

This document specifies the sample preparation and liquid chromatography for the detection of metronidazole, hydroxymetronidazole, demetonidazole and hydroxydemenidazole residues in food of animal origin.

Chromatography-tandem mass spectrometry method.

This document applies to the muscle, liver and kidney tissue of pigs, cattle, sheep and chickens for metronidazole, hydroxymetronidazole, demetonidazole and hydroxydemetrazol

Determination of azole residues.

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 nitroimidazoles in the sample were extracted with ethyl acetate, degreased by n-hexane liquid-liquid extraction, purified by solid-phase extraction column, and liquid chromatography-serial.

Mass spectrometry was used for detection, and matrix-matched internal standard method was used for quantification.

5 Reagents and materials

Unless otherwise specified, all reagents are of analytical grade, and the water is first-class water in accordance with GB/T 6682.

5.2 Solution preparation

5.2.11 0.1mol/L hydrochloric acid solution. take 8.3mL of concentrated hydrochloric acid, add water to dilute to 1L, and mix well.

5.2.2 2% ammonia solution. Take 2mL of ammonia water, dilute it with water to 100mL, mix well, and prepare immediately.

5.2.3 Eluent. Take 80 mL of methanol, add 15 mL of water and 5 mL of ammonia water, mix well, and prepare immediately after use.

5.2.4 0.1% formic acid aqueous solution. Take 500 mL of water, add 500 μL of formic acid, and mix well.

5.2.5 0.1% formic acid in acetonitrile solution. take 500 mL of acetonitrile, add 500 μL of formic acid, and mix well.

5.3 Standards

Metronidazole, demetonidazole, hydroxymetronidazole, hydroxydemetronidazole, metronidazole-D3, demetronidazole-D3, hydroxymetronidazole-D2, hydroxydemetronidazole

azole-D3, the contents were >=95%. See Appendix A for details.

5.4 Preparation of standard solution

5.4.1 Standard stock solution. take an appropriate amount of metronidazole, demetonidazole, hydroxymetronidazole, and hydroxydemenidazole standard (equivalent to

10mg), accurately weighed, dissolved in methanol and diluted to volume in a 10mL volumetric flask to prepare metronidazole and demetrazol with a concentration of 1mg/mL

azole, hydroxymetronidazole and hydroxydemenidazole standard stock solutions. Stored below -18 degrees C, valid for 6 months.

5.4.2 Internal standard stock solution. Take an appropriate amount of standard substances of metronidazole-D3, demetonidazole-D3, hydroxymetronidazole-D2 and hydroxydemetrinidazole-D3 (equivalent to

10 mg of each active ingredient), accurately weighed, dissolved in methanol and diluted to a volume in a 10 mL volumetric flask, to prepare methylnitrate with a concentration of 1 mg/mL

azole-D3, demetonidazole-D3, hydroxymetronidazole-D2 and hydroxymetronidazole-D3 internal standard stock solutions. Stored below -18 degrees C, valid for 6 months.

5.4.3 10μg/mL mixed standard working solution. Accurately measure 0.1mL of each standard stock solution into a 10mL volumetric flask, dilute with methanol

to the mark, and prepared into a mixed standard working solution with a concentration of 10 μg/mL. Stored below -18°C, the validity period is 6 months.

5.4.4 1μg/mL mixed standard working solution. accurately measure 10μg/mL mixed standard working solution 1mL into a 10mL volumetric flask,

Diluted with alcohol to the mark, and prepared a mixed standard working solution with a concentration of 1 μg/mL. Stored below -18°C, the validity period was 3 months.

5.4.5 0.1μg/mL mixed standard working solution. accurately measure 1mL of 1μg/mL mixed standard working solution into a 10mL volumetric flask,

Diluted with alcohol to the mark, and prepared a mixed standard working solution with a concentration of 0.1 μg/mL. Stored below -18°C, the validity period was 1 month.

5.4.6 10 μg/mL mixed internal standard working solution. Accurately measure 0.1 mL of each internal standard stock solution into a 10 mL volumetric flask, dilute with methanol

To the mark, it was prepared into a mixed internal standard working solution with a concentration of 10 μg/mL. Stored below -18°C, the validity period was 6 months.

5.4.7 1 μg/mL mixed internal standard working solution. accurately measure 1 mL of 10 μg/mL internal standard working solution in a 10 mL volumetric flask, dilute with methanol

The mixture was released to the mark, and prepared into a mixed internal standard working solution with a concentration of 1 μg/mL. Stored below -18°C, the validity period was 3 months.

5.5 Materials

5.5.1 Mixed strong cation exchange reversed-phase solid-phase extraction column. 60mg/3mL, or equivalent.