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NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB 31659.3-2022

National Food Safety Standard Determination of Cephalosporin Residues in Milk and Milk Powder 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 replaces GB/T 22989-2008 "Determination of cefapirin, cephalexin, cefuroxime and cefquinome residues in milk and milk powder

Compared with GB/T 22989-2008, except for structural adjustment and editorial changes, the main changes are as follows.

--- The standard text format is changed to the national food safety standard text format;

--- Increase the detection of goat milk in the standard scope;

--- Increase the number of drug varieties in the standard range;

---Standard sensitivity is further improved.

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

---GB/T 22989-2008.

National Food Safety Standards

Determination of cephalosporin residues in milk and milk powder by liquid chromatography-tandem mass spectrometry

1 Scope

This document specifies cephalexin, cephradine, cefazolin, cefoperazone, cefacetonitrile, cefapirin, cephalosporins in milk, goat milk and milk powder.

Sample preparation and liquid chromatography-tandem mass spectrometry method for the detection of serotonin, cefquinoxime and cefotaxime residues.

This document is applicable to cephalexin, cephradine, cefazolin, cefoperazone, cefacetonitrile, cefapirin, cephalexin in milk, goat milk and milk powder

Determination of residues of serotonin, cefquinome and cefotaxime.

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 drug residues in the sample were extracted with phosphate buffer solution, purified by hydrophilic-lipophilic equilibrium solid-phase extraction column, and detected by liquid chromatography-tandem mass spectrometry.

determined and quantified by matrix calibration external standard method.

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 2.5mol/L sodium hydroxide solution. take 50g of sodium hydroxide, add water to dissolve and dilute to 500mL.

5.2.2 30% acetonitrile solution. Take 30mL of acetonitrile and dilute with water to 100mL.

5.2.3 0.05mol/L phosphate buffer solution (pH=8.5). take 6.8g of potassium dihydrogen phosphate, dissolve it in water and dilute it to 1000mL, and use

2.5 mol/L sodium hydroxide solution to adjust the pH to 8.5.

5.2.4 0.1% formic acid solution. Take 1mL of formic acid and dilute it with water to 1000mL.

5.2.5 0.1% formic acid solution-methanol (95.5). take 95mL of 0.1% formic acid solution and 5mL of methanol, and mix well.

5.3 Standards

Cefalexin, cephradine, cefazolin, cefoperazone, cefacetonitrile, cefapirin, deacetylcefapirin,

ceftaroning, cefquinone

Oxime and cefotaxime standard products, the content of which is >=95%, see Appendix A for details.

5.4 Preparation of standard solution

5.4.1 Standard stock solution. take 10 mg of each standard product, weigh them precisely, dissolve them in an appropriate amount with 30% acetonitrile solution and dilute to 25 mL.

A volumetric flask was prepared into a standard stock solution with a concentration of 400 μg/mL. It was stored at -18°C in the dark, and the validity period was 1 month.

5.4.2 Mixed standard stock solution. Accurately pipette 0.25mL of each standard stock solution into a 10mL volumetric flask, and dilute with 30% acetonitrile solution.

The solution was released to the mark, and prepared into a mixed standard stock solution with a concentration of 10 μg/mL. Stored in the dark at -18°C, the validity period was 7 days.

5.4.3 Mixed standard working solution. Accurately pipette an appropriate amount of mixed standard stock solution, and dilute it with 0.1% formic acid solution-methanol (95.5) to a concentration of

2.5μg/L, 5.0μg/L, 20μg/L, 100μg/L,.200μg/L and 500μg/L series mixed standard working solutions. Ready to use and prepare.

5.5 Materials

5.5.1 Solid-phase extraction column. hydrophilic-lipophilic balanced solid-phase extraction column, 500mg/6mL, or equivalent.

7.1 Preparation of samples

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

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 Storage of samples

Store below -18°C.

8.1 Extraction