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NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB 31657.3-2022

National food safety standards-Determination of cephalosporin residues in bee products-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 22942-2008 "Residues of cefazolin, cefapirin, cephalexin, cefuronine and cefquinome in honey

Determination of liquid chromatography-tandem mass spectrometry'. Compared with GB/T 22942-2008, except for structural adjustments and editorial changes, the main changes

as follows.

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

--- The detection of royal jelly and royal jelly freeze-dried powder has been added to the scope of the standard;

--- 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 22942-2008.

National Food Safety Standards

Determination of cephalosporin residues in bee products by liquid chromatography-tandem mass spectrometry

1 Scope

This document specifies the cephalexin, cephradine, cefazolin, cefoperazone, cefacetonitrile, and cephalexin in honey, royal jelly and royal jelly freeze-dried powder.

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

This document is applicable to cephalexin, cephradine, cefazolin, cefoperazone, cefacetonitrile, cephalexin in honey, royal jelly and royal jelly freeze-dried powder

Detection of spirulina, cefuroxime, cefquinoxime and cefotaxime residues.

2 Normative references

The content in the following documents constitutes the essential clauses of this document through the normative references in the text. Among them, the dated references

For documents, only the version corresponding to the date is applicable to this document; for undated reference documents, the latest version (including all amendments) is applicable 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 residual cephalosporins in the sample were extracted by phosphate buffer solution, purified by hydrophilic-lipophilic equilibrium solid-phase extraction column, and liquid chromatography-tandem mass

Determination by spectrometry, quantification 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, dissolve it in water and dilute to 1000mL.

5.2.5 0.1% formic acid solution-methanol. Take 95 mL of 0.1% formic acid solution, add 5 mL of methanol, and mix well.

5.3 Standards

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

oxime and cefotaxime, the content of which was >=95.0%, see Appendix A for specific information.

5.4 Preparation of standard solution

5.4.1 Standard stock solution. Take 10 mg of each standard product, weigh them accurately, add an appropriate amount of 30% acetonitrile solution to dissolve and dilute to a 25 mL volumetric flask,

It was prepared into a solution with a concentration of 400 μg/mL. It was stored in the dark below -18 degrees C, 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, dilute with 30% acetonitrile solution

To the mark, it was prepared into a mixed standard stock solution with a concentration of 10 μg/mL. Stored in the dark below -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 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.

5.5 Materials

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

7.1 Preparation of samples

Take an appropriate amount of fresh or refrigerated blank or test bee products, if there is no crystallization, stir it evenly, if there is crystallization, place it at no more than 60°C

Warm in a water bath, oscillate, stir evenly after the sample is completely melted, and cool to room temperature. Royal jelly and royal jelly freeze-dried powder are taken out from the