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NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB 31658.22-2022

National food safety standard - Determination of beta-agonists residues in animal derived food by liquid chromatography-tandem mass spectrometric method

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Table of Contents

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 22286-2008 "Determination of multiple beta-receptor agonist residues in foods of animal origin by liquid chromatography tandem

Mass spectrometry', GB/T 21313-2007 "Methods for the detection of beta-receptor agonist residues in food of animal origin liquid chromatography-mass spectrometry/mass spectrometry'.

Compared with GB/T 22286-2008, the main changes of this document are as follows.

a) Modify the text format to the national food safety standard text format;

b) Added testing organizations in the scope (see Chapter 1);

c) Increased the types of drugs in the scope (see Chapter 1);

d) The sensitivity is further improved. The detection limit of the analyte in the muscle, liver and kidney of pigs, cattle and sheep is 0.2 μg/kg, and the limit of quantification is

0.5 μg/kg.

Compared with GB/T 21313-2007, the main changes of this document are as follows.

a) Modify the text format to the national food safety standard text format;

b) Added testing organizations in the scope (see Chapter 1);

c) Added the category of drugs in the scope (see Chapter 1).

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

---GB/T 22968-2008, GB/T 21313-2007.

National Food Safety Standards

Determination of beta-receptor agonist residues in food of animal origin

Liquid chromatography-tandem mass spectrometry

1 Scope

This document specifies the sample preparation and liquid chromatography-tandem mass spectrometry method for the detection of beta-receptor agonist residues in animal foods.

This document applies to clenbuterol, ractopamine, albuterol, cimaterol, zilpaterol,

Chlorprenaline, terbutaline, sibuterol, mabuterol, bromobuterol, bambuterol, clemprorol, tobuterol, ritodrine, cloncerol, horse

Detection of single or mixture residues of 18 beta-receptor agonists including pentagon, clenbuterol and hydroxymethyl clenbuterol.

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 beta-receptor agonist in the test sample was enzymatically hydrolyzed and precipitated with perchloric acid, extracted with ethyl acetate and tert-butyl methyl ether, and purified by solid-phase extraction column.

It was determined by liquid chromatography-tandem mass spectrometry and quantified by internal standard method.

5.1 Reagents

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

5.2 Solution preparation

5.2.1 0.2mol/L ammonium acetate buffer solution. Take 15.4g of ammonium acetate,

dissolve it in 1000mL water, adjust the pH to 5.2.

5.2.2 0.1mol/L perchloric acid solution. Take 8.7mL of perchloric acid and dilute with water to 1000mL.

5.2.3 10mol/L sodium hydroxide solution. Weigh 40g of sodium hydroxide, dissolve it with an appropriate amount of water and cool it down, then dilute to 100mL with water.

5.2.4 2% formic acid solution. Take 2mL of formic acid and dilute with water to 100mL.

5.2.5 5% ammoniated methanol solution. Take 5mL ammonia water and dilute it to 100mL with methanol.

5.2.6 0.1% formic acid in acetonitrile solution. Take 1 mL of formic acid and dilute it to 1000 mL with acetonitrile.

5.2.7 0.1% formic acid solution. Take 1mL of formic acid and dilute it with water to 1000mL.

5.2.8 Methanol-0.1% formic acid solution (10+90, V/V). Take 10mL of methanol and 90mL of 0.1% formic acid solution, mix well.

5.3 Standards

5.3.1 beta-receptor agonists. clenbuterol, ractopamine, salbutamol, cimaterol, zilpaterol, clorprenaline, terbutaline, sibuterol

Tributerol, Mabuterol, Bromobuterol, Bambuterol, Clemprorol, Tobuterol, Ritodrine, Clenbuterol, Mapenterol, Clumpantrol, and Oxymetholone

Diclonbuterol, content >= 98.0%, see Appendix A.

5.3.2 Isotope internal standard. clenbuterol-D9, ractopamine hydrochloride-D6, salbutamol-D3, cimaterol-D7, zilpaterol-D7, chlorprena

Lin-D7, terbutaline-D9, sibuterol-D9, mabuterol hydrochloride-D9, bambuterol hydrochloride-D9, clemprorol-D7, tobuterol hydrochloride-D9

and hydroxymethylclenbuterol-D6, the content of which was >=98.0%. See Appendix A.

5.4 Preparation of standard solution

5.4.1 Mixed standard stock solution. Accurately weigh about 10 mg of clenbuterol and other standard products, dissolve in methanol and dilute to a 10 mL volumetric flask,

Prepare a standard stock solution with a concentration of 1.0 mg/mL, store it below -18°C, and have a validity period of 12 months.

5.4.2 Mixed internal standard stock solution. Accurately weigh about 10 mg of isotope internal standard such as clenbuterol-D9, dissolve it in methanol and dilute to 10 mL capacity.