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NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB 31658.19-2022

National Food Safety Standard Determination of Atropine, Scopolamine, Anisodamine, Lidocaine, and Procaine Residues in Animal Foods-Liquid Chromatography-Tandem Mass Spectrometry

Issued on: September 20, 2022 **Implemented on: February 1, 2023**

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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 is published for the first time.

National Food Safety Standards

Atropine, scopolamine, anisodamine, lidocaine, procaine in animal food

Determination of residues by liquid chromatography-tandem mass spectrometry

1 Scope

This document specifies the sample preparation and solutions for the detection of atropine, scopolamine, anisodamine, lidocaine, and procaine residues in animal foods.

Phase chromatography-tandem mass spectrometry detection method.

This document applies to atropine, scopolamine, anisodamine, lidocaine, and procaine in muscle, liver, kidney, and fat of pigs, cattle, and sheep

Determination of single or multiple drug 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 analyte remaining in the sample was extracted by phosphate buffer solution, purified by PEP-2 solid phase extraction column, determined by liquid chromatography-tandem mass spectrometry, and external standard method.

Quantitative.

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 Potassium dihydrogen phosphate buffer solution. Take 13.6g of potassium dihydrogen phosphate, add 900mL of water to dissolve, adjust the pH to

4.0+/-0.05, dilute with water to 1000mL, and mix well.

5.2.2 0.2mol/L dipotassium hydrogen phosphate solution. Take 22.8g of dipotassium hydrogen phosphate, add water to dissolve and dilute to 500mL, and mix well.

5.2.3 5% methanol solution. Take 5mL of methanol, add water to dilute to 100mL, and mix well.

5.2.4 30% acetonitrile solution. take 30mL of acetonitrile, add water to dilute to 100mL, and mix well.

5.2.5 0.1% formic acid solution. take 0.5mL of formic acid, add water to dilute to 500mL, and mix well.

5.3 Standards

Atropine (Atropine, C17H23NO3, CAS number. 51-55-8), scopolamine (Scopolamine, C17H21NO4, CAS number. 51-

34-3), anisodamine (Anisodamine, C17H23NO4, CAS number. 55869-99-3), lidocaine (Lidocaine C14H22N2O, CAS

No.. 137-58-6), procaine (Procaine, C13H20N2O2, CAS No.. 59-46-1), the content is >=99%. The standard can also be the corresponding salt.

5.4 Preparation of standard solution

5.4.1 Standard stock solution. Take the appropriate amount of atropine, scopolamine, anisodamine, lidocaine and procaine

About 10 mg), accurately weighed, add methanol to dissolve and dilute to 10 mL volumetric flask, and prepare a standard concentration of 1 mg/mL

Stock solution. Stored at -18°C in the dark, valid for 6 months.

5.4.2 Mixed standard intermediate solution. Accurately measure 1mL of each standard stock solution, put it in a 100mL volumetric flask, dilute it to the mark with methanol, and prepare

The mixed standard working solution with a concentration of 10 μg/mL was stored at -18°C in the dark, and the validity period was 1 month.

5.4.3 series of standard working solutions. accurately measure an appropriate amount of mixed standard intermediate solution, dilute with 30% acetonitrile solution, and prepare the concentrations of

1ng/mL, 2ng/mL, 5ng/mL, 10ng/mL, 20ng/mL, 50ng/mL and 100ng/mL series of standard solutions. Ready to prepare

Now in use.

5.5 Materials

1) The PEP-2 solid-phase extraction column listed here is for reference only and does not involve commercial purposes. Standard users are encouraged to try to use different manufacturers or models of solid-phase extraction columns.

5.5.1 PEP-2 solid phase extraction column 1). The filler is functionalized polystyrene/divinylbenzene, 60mg/3mL, or equivalent.

7.1 Preparation of samples

Take an appropriate amount of fresh or thawed blank or test tissue, mince it, and make it homogeneous.

a) Take the homogenized test sample as the test material;

b) Take the homogenized blank sample as the blank sample;

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

7.2 Storage of samples

Store below -18°C.

8.1 Extraction

Take 2 g of the test material (accurate to +/-0.05 g) into a 50 mL centrifuge tube, and add