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NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB 31658.8-2021

National food safety standard - Determination of pyrethroid residues in animal derived food by gas chromatography-mass Spectrometric method

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Foreword

This document complies with the provisions of GB/T 1:1-2020 "Standardization Work Guidelines Part 1: Structure and Drafting Rules of Standardization Documents"

Drafting:

1 Scope

This document specifies the sample preparation and gas chromatography-mass spectrometry methods for the detection of pyrethroid drug residues in animal foods: This document applies to single or multiple versions of deltamethrin, bifenthrin, fluvalerate, fluvalinate, tefluthrin and fenvalerate in the muscle, fat and liver of cattle, sheep and pigs: Determination of drug residues:

2Normative reference documents

The contents of the following documents constitute essential provisions of this document through normative references in the text: Among them, for dated reference documents, only the version corresponding to the date applies to this document; for undated reference documents, the latest version (including all amendments) applies to this document:

GB/T 6682 Specifications and test methods for water used in analytical laboratories

3 Terms and definitions

There are no terms or definitions to be defined in this document:

4 principles

The remaining pyrethroid drugs in the sample were extracted with acetonitrile, purified with

a solid phase extraction column, measured by gas chromatography-mass spectrometry, and quantified by the external standard method:

5Reagents and Materials

Unless otherwise specified, all reagents are of analytical grade and the water is first-grade water that complies with GB/T 6682:

5:1 Reagents

5:1:1 Acetonitrile (CH3CN): chromatographically pure:

5:1:2 n-hexane (C5Hu): chromatographically pure:

5:1:3 Sodium chloride (NaCD):

5:1:4 Anhydrous sodium sulfate (Na2SO4):

5:1:5 Benzene (C6H6): chromatographically pure:

5:1:6 Acetone (CH3COCH3): chromatographically pure:

5:2 Standard products

Deltamethrin content >=99:7%, bifenthrin content >=98%, flumethrin content >=87:5%, fluvalinate content >=94%, tefluthrin content >=98%, fenvalerate content >=98% Ester content >=99%, see Appendix A:

5:3 Preparation of standard solution

5:3:1 Standard stock solution: Take 10 mg each of deltamethrin, bifenthrin, fluvalerate, fluvalinate, tefluthrin and fenvalerate standard products, weigh them accurately, and add benzene Dissolve an appropriate amount, dilute with acetone to a 100mL volumetric flask, shake well, and prepare deltamethrin, bifenthrin, fluvalerate, fluvalinate, sevfluthrin and fenvalerate Standard stock solution: Store at -18 degrees C, valid for 3 months:

5:3:210 μg/mL mixed standard working solution: Precisely measure 1 mL of each of the above standard stock solutions into a 10 mL volumetric flask, dilute to the mark with acetone, and prepare a mixed standard working solution with a concentration of 10 μg/mL: Store at 2 degrees C~8 degrees C, valid for 1 month:

5:3:31:0 μg/mL mixed standard working solution: Precisely measure 1 mL of the 10 μg/mL mixed standard working solution in a 10 mL volumetric flask, dilute to the mark with acetone, and prepare a mixed standard working solution with a concentration of 1:0 μg/mL: Store at 2 degrees C~8 degrees C, valid for 1 month:

5:4 Materials

Neutral alumina solid phase extraction column: 500 mg/6mL, or equivalent:

6Instruments and equipment

6:1 Gas Chromatograph-Mass Spectrometer: Equipped with electrospray ion source:

6:2 Analytical balance: sensitive to 0:00001 g and 0:01g:

6:3 High-speed centrifuge:

6:4 Vortex mixer:

6:5 Horizontal Oscillator:

6:6 Tissue homogenizer:

6:7 Solid phase extraction device:

6:8 Nitrogen blower:

7 Preparation and preservation of samples

7:1 Preparation of samples

Take an appropriate amount of fresh or thawed blank or test tissue, mince it, and homogenize it:

a) Take a homogeneous test sample as a test material;

b) Take a homogeneous blank sample as a blank sample;

c) Take a homogeneous blank sample, add standard working solution of appropriate concentration, and add the sample as a blank:

7:2 Storage of samples

Store below -18 degrees C:

8 Measurement steps

8:1 Extraction

Weigh 5g of the sample (accurate to +/-0:02g), put it in a 50mL polypropylene centrifuge tube, add 4g of sodium chloride and 25mL of acetonitrile, homogenize for 1 minute, shake for 15 minutes, centrifuge at 6000r/min for 5 minutes, and take the supernatant In another centrifuge tube, add 15 mL of acetonitrile to the residue and repeat the extraction once: Combine the supernatants, add 4 g of anhydrous sodium sulfate, shake, and centrifuge at 4°C and 1000 r/min for 10 min: Take the supernatant and set aside:

8:2 Purification

Take a neutral alumina solid-phase extraction column and activate it with acetonitrile twice, 5 mL each time: Pass the standby solution through the column and elute with 10 mL of acetonitrile: Collect all eluents, rotary evaporate to dryness at 50°C, add 1:0 mL of n-hexane to dissolve, filter, and use for gas chromatography-mass spectrometry

measurement:

8:3 Preparation of matrix matching standard curve

Precisely measure 0:01mL, 0:02mL, 0:05 mL of 1:0μg/mL mixed standard working solution, and 10μg/mL pyrethroid mixed standard:

Working solutions 0:01mL, 0:05mL and 0:10mL were added to 6 portions of the blank sample concentrate processed by the sample pretreatment step, and diluted to 1mL with n-hexane to obtain concentrations of 10ng/mL, 20ng/mL and 50ng/mL: mL, 100 ng/mL, 500 ng/mL, and 1000 ng/mL matrix-matched series standard solutions, filtered, and used for gas chromatography-mass spectrometry determination: Using the measured characteristic ion mass chromatographic peak area as the ordinate and the corresponding standard solution concentration as the abscissa, draw a matrix matching standard curve: Find the regression equation and correlation coefficient:

8:4 Determination

8:4:1 Gas Chromatography Reference Conditions

a) Chromatographic column: DB-1 (100% dimethylpolysiloxane) capillary column: 30mx0:25 mm, film thickness 0:25μm, or equivalent;

b) Chromatographic column temperature: starting column temperature is 70 degrees C, rising to 250 degrees C at 30 degrees C/min, rising to 274 degrees C at 3 degrees C/min, rising to 294 degrees C at 20 degrees C/min, rising to 30 degrees C/min at 30 degrees C/min: 300 degrees C, keep for 2 minutes;

c) Carrier gas: high purity helium, column flow rate 1:3mL/min;

d) Split: Splitless injection;

e) Injection volume: 1μL;

f) Inlet temperature: 280 degrees C;

g) Interface temperature: 270 degrees C:

8:4:2 Mass spectrometry reference conditions

a) Ion source: NCI source;

b) Reaction gas: CH4 (purity greater than 99:99%);

c) Ion source temperature: 150 degrees C;

d) Ionization voltage: 0:98 kV;

e) Quadrupole mass analyzer temperature: 150 degrees C;

f) Electron energy: 235 eV;