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NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB 31658.10-2021

National food safety standard - Determination of carbamate insecticides residues in animal derived foods by liquid chromatography-tandem mass spectrometry method

Issued on: September 16, 2021 **Implemented on: February 1, 2022**

Issued by: National Health Commission of the People's Republic of China, State Administration for Market Regulation

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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 carbamate pesticides (carbofuran, 3-hydroxycarbofuran, aldicarb, aldicarb, etc:) in pig, cattle, sheep, chicken tissues (muscle, liver, kidney and fat), eggs and milk: Methiocarb sulfone, aldicarb sulfoxide, methomyl, methiocarb, methiocarb, methiocarb, methiocarb, methiocarb sulfone, methiocarb sulfoxide, pirimicarb, desmethylmethiocarb Sample preparation and liquid chromatography-tandem mass spectrometry method for detection of residues of ethylcarb, ethiobencarb, isoprocarb, carbaryl, dipiocarb, propoxur, secbutylcarb, fenoxycarb, and indoxacarb:

This document is applicable to the determination of carbamate pesticide residues in pig, cattle, sheep, chicken tissues (muscle, liver, kidney and fat), eggs and milk:

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

3Terms and Definitions

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

4 Principles

The target compound remaining in the sample was extracted with acetonitrile, degreased with n-hexane, enriched and purified by solid-phase extraction, measured by liquid chromatography-tandem 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 Methanol (CH2OH):

5:1:3 Formic acid (HCOOH): chromatographically pure:

5:1:4 n-hexane (C, Hμ)

5:1:5 Dichloromethane (CH2Cl2):

5:1:6 Sodium chloride (NaCl),

5:1:7 Anhydrous sodium sulfate (Na2SO4):

5:2 Solution preparation

5:2:1 Dichloromethane-methanol solution: Take 99 mL of dichloromethane and 1 mL of methanol, and mix well:

5:2:2 25% acetonitrile solution: Take 25 mL of acetonitrile and 75 mL of water, and mix well:

5:2:30:1% formic acid solution: Take 1mL of formic acid, dilute it with water to 1000mL, and mix well:

5:2:4 Saturated sodium chloride solution: Take 50g of sodium chloride, add 100mL of water, shake while adding, and let stand:

5:3 Standard products: carbofuran, 3-hydroxycarbofuran, aldicarb, aldicarb sulfone, aldicarb sulfoxide, methomyl, methiocarb, methiocarb, methiocarb, methiocarb, methiocarb Dimethiocarb sulfone, dipiocarb sulfoxide, pirimicarb, desmethylpipicarb, ethiobencarb, isoprocarb, carbaryl, dipiocarb, propoxur, butylcarb, fenoxycarb and indene Chongiocarb, the content is >=95%, see Appendix A:

5:4 Preparation of standard solution

5:4:1 Standard stock solution: Take about 10 mg of each carbamate pesticide standard, weigh it accurately, add an appropriate amount of acetonitrile to dissolve and dilute to a 10 mL brown volumetric flask, and prepare a solution with a concentration of 1000 μg/mL: Standard stock solution: Store in a dark place below -18 degrees C, valid for 6 months:

5:4:2 Mixed standard intermediate solution: Accurately pipette 1 mL of each standard stock solution into a 100 mL brown volumetric flask, dilute to the mark with acetonitrile, and prepare a mixed standard intermediate solution with a concentration of 10 μg/mL: Store in a dark place below -18 degrees C, valid for 6 months:

5:4:3 Mixed standard working solution: Accurately pipet an appropriate amount of the mixed standard intermediate solution, and dilute it with 25% acetonitrile solution to a concentration of 2 μg/L, 5 μg/L, 20 μg/L, 50 μg/L,:200 μg/L and 500 μg/L: L series mixed standard working fluid: Ready for use:

5:5 Materials

5:5:1 Amino solid-phase extraction column: 500 mg/6mL, or equivalent, activated with 4mL of dichloromethane-methanol solution before use:

5:5:2 Syringe filter (universal filter membrane): pore size 0:22μm, organic system:

6Instruments and equipment

6:1 Liquid chromatography tandem mass spectrometer: equipped with electrospray ion source:

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

6:3 Nitrogen blowing instrument:

6:4 Solid phase extraction device:

6:5 Centrifuge tube: polypropylene plastic centrifuge tube, 50 mL:

6:6 Centrifuge: 5000 r/min or above:

6:7 Vortex mixer:

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 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 the standard working solution of appropriate concentration, and add the sample as a blank:

7:2 Storage of samples

Store below -18 degrees C and conduct analysis and testing within 3 months:

8 Measurement steps

8:1 Extraction

Take 5g of the sample (accurate to +/-0:05g), put it in a 50mL plastic centrifuge tube, add 5g of anhydrous sodium sulfate and 15 mL of acetonitrile in sequence, vortex for 1 min, shake for 30 min, centrifuge at 5000 r/min for 5 min, and collect the supernatant solution, add 10 mL of acetonitrile to the residue, repeat the extraction once, combine the extracts, and set aside:

8:2 Purification

Take the reserve solution, add 10 mL of saturated sodium chloride solution and 10 mL of n-hexane, vortex for 1 min, and centrifuge at 5000 r/min for 3 min: Take the acetonitrile solution of the middle layer in a 50 mL plastic centrifuge tube, and blow with nitrogen in a 40°C water bath until it is nearly dry: Add 2 mL of dichloromethane-methanol solution to dissolve the residue, pass it through the activated amino solid-phase extraction column, collect the effluent, wash the 50 mL plastic centrifuge tube with 2 mL of dichloromethane-methanol solution, and pass through the column: Repeat once to collect all the effluent: liquid, blow with nitrogen in a 50°C water bath until it is nearly dry, dissolve the residue with 1:0 mL of 25% acetonitrile solution, vortex to mix, and filter with a 0:22 μm filter membrane for liquid chromatography-tandem mass spectrometry measurement:

8:3 Preparation of standard curve

Take a series of mixed standard working solutions of 2 μg/L, 5 μg/L, 20 pg/L, 50 pg/L,:200 pg/L and 500 μg/L, and inject samples for liquid chromatography-tandem mass spectrometry measurement: Using the quantitative ion pair peak area as the ordinate and the standard solution concentration as the abscissa, draw a standard curve and find the regression equation and correlation coefficient:

8:4 Determination

8:4:1 Liquid Chromatography Reference Conditions

a) Chromatographic column: Cis column (100 mmx2:1mm, 1:7μm), or equivalent;

b) Mobile phase: A is 0:1% formic acid solution, B is acetonitrile, gradient elution conditions are shown in Table 1; c) Flow rate: 0:3mL/min;

d) Column temperature: 35 degrees C;

e) Injection volume: 10 μL:

8:4:2 Mass spectrometry reference conditions

a) Ion source: electrospray ion source;

b) Scanning method: positive ion scanning;