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NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB 31656.9-2021

National food safety standard - Determination of pendimethalin residue in aquatic products by liquid chromatography-tandem mass spectrometry 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:

This document is published for the first time:

National food safety standards

Determination of pendimethalin residues in aquatic products by liquid chromatography-tandem mass spectrometry

1 Scope

This document specifies the sample preparation and liquid chromatography-tandem mass spectrometry methods for detecting pendimethalin residues in aquatic products: This document is applicable to the detection of pendimethalin residues in edible tissues of fish, shrimps, crabs, shells, sea cucumbers, and turtles:

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 use in analytical laboratories GB/T 30891-2014 Specifications for sampling of aquatic products

3Terms and Definitions

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

4 Principles

The remaining pendimethalin in the sample was extracted with acetonitrile, purified with HLB solid-phase extraction column, lipid impurities were removed with Ci8 adsorbent, measured by liquid chromatography-tandem mass spectrometry, and quantified by the internal 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 (C2H3N): chromatographically pure:

5:1:2 Methanol (CH4O): chromatographically pure:

5:1:3 Formic acid (CH2O2): chromatographically pure:

5:1:4 n-hexane (C6H): chromatographically pure:

5:1:5 Acetone (C3H8O): chromatographically pure:

5:1:6 Sodium chloride (NaCl),

5:2 Solution preparation

5:2:1 n-hexane-acetone solution: Take 500 mL of n-hexane and 500 mL of acetone, and mix well:

5:2:2 Formic acid aqueous solution (0:1%): Take 1mL of formic acid and dilute with water to 1000mL:

5:3 Standard products

5:3:1 Pendimethalin (Ci3Hi9N3O4, CAS No:: 40487-42-1), content >=98:0%:

5:3:2 Pendimethalin-Ds, C3H14N3O4D5, CAS number: 1219803-39-0), content >=98:0%:

5:4 Preparation of standard solution

5:4:1 Standard stock solution (1mg/mL): Take about 10mg of pendimethalin standard, weigh it accurately, dissolve it in acetonitrile and dilute it to a 10mL volumetric flask, shake well, and it is ready: Store away from light at 4 degrees C, valid for 6 months:

5:4:2 Internal standard stock solution (1 mg/mL): Take about 10 mg of the deuterated pendimethalin standard, weigh it accurately, dissolve it with B and dilute it to a 10 mL volumetric flask, shake well, and it is ready: Store away from light at 4 degrees C, valid for 6 months:

5:4:3 Standard intermediate solution (1 μg/mL): Accurately transfer 0:1 mL of the pendimethalin standard stock solution into a 100 mL volumetric flask, dilute to the mark with acetonitrile, and prepare a standard intermediate solution with a concentration of 1 μg/mL: Store at 4°C away from light, valid for 2 weeks:

5:4:4 Internal standard intermediate solution (1 μg/mL): Accurately transfer 0:1 mL of the deuterated pendimethalin standard stock solution into a 100 mL volumetric flask, dilute to the mark with acetonitrile, and prepare an internal standard with a concentration of 1 μg/mL: Intermediate liquid: Store at 4°C away from light, valid for 2 weeks:

5:5 Materials

5:5:1 HLB solid phase extraction column: 60 mg/3mL, or equivalent:

5:5:2 Cs adsorbent: 40μm~60μm:

5:5:3 Microporous filter membrane: 0:22μm, resistant to organic reagents:

6Instruments and Equipment

6:1 Liquid chromatography-tandem mass spectrometer: equipped with electrospray ion source (ESI source):

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

6:3 Nitrogen blowing instrument:

6:4 Homogenizer:

6:5 Vortex mixer:

6:6 Centrifuge: 10000 r/min or above:

6:7 Ultrasonic oscillator:

6:8 Rotary evaporator:

6:9 Solid phase extraction device:

7 Preparation and preservation of samples

7:1 Preparation of samples

Prepare samples according to the requirements of Appendix B of GB/T 30891-2014:

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 a standard 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 5:0g of the sample (accurate to +/-0:02 g), add 50 µL of internal standard working solution, mix well, add 6 mL of water, 1 g of sodium chloride, and 20 mL of acetonitrile in sequence, vortex at 3000 r/min for 1 min, and ultrasonic extraction for 20 min, 4000 Centrifuge at r/min for 10 min: Take the supernatant, add 20 mL of acetonitrile to the residue and repeat the extraction once, and combine the supernatants: Concentrate to near dryness by rotary evaporation at 40°C, add 3 mL of acetonitrile to dissolve the residue, and set aside:

8:2 Purification

Activate the solid phase extraction column with 3 mL of acetonitrile, pass the reserve solution through the column, control the flow rate to not exceed 1 mL/min, elute with 3 mL of acetonitrile, and add 8 mL of n-hexane-acetone solution for elution: Collect the eluate, blow with nitrogen at 40°C until it is nearly dry, add 2 mL of acetonitrile accurately to reconstitute, add 0:5 g of C adsorbent, vortex at 3000 r/min for 1 min, centrifuge at 4000 r/min for 10 min, take the supernatant and 0:22µm filter membrane for liquid chromatography-tandem mass spectrometry measurement:

8:3 Preparation of standard curve

Accurately pipet appropriate amounts of standard working solution and internal standard working solution, dilute with acetonitrile to prepare pendimethalin concentration of 2:5 µg/L,

5:0µg/L, 10:0µg/L, 20:0 µg/L, 50:0µg/L, 100:0µg/L, deuterated pendimethalin concentration is 25:0µg/L series mixture 2

Standard working solution for determination by liquid chromatography-tandem mass spectrometer: Ready to use: Taking the peak area ratio of the test substance and the internal standard substance as the ordinate and the corresponding pendimethalin concentration as the abscissa, draw a standard working curve and find the regression equation and correlation coefficient:

8:4 Determination

8:4:1 Liquid Chromatography Reference Conditions

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

b) Column temperature: 40 degrees C;

c) Injection volume: 5pL;