CCS X 20

NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB 31656.8-2021

National food safety standard - Determination of organophosphorus pesticide residues in fishery products by liquid chromatography-tandem mass spectrometry

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

Table of Contents

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 organophosphorus drug residues in aquatic products by liquid chromatography-tandem mass spectrometry

1 Scope

This document stipulates phoxim, palamiphos, fenthion, malathion, diazinon, trichlorfon, dichlorvos, picoline, and fly poison in aquatic products

Sample preparation and liquid chromatography-tandem mass spectrometry determination methods for phosphorus residue detection:

This document applies to phoxim, palamiphos, fenthion, malathion, diazinon, trichlorfon, dichlorvos, picolinate and Detection of phosphorus residues:

2 Normative reference documents

The contents of the following documents constitute essential provisions of this document through normative citations in the text: Among them, the cited documents with dates are:

Only the version corresponding to the date applies to this document; for undated referenced documents, the latest version (including all amendments) applies to this document:

document:

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

3 Terms and definitions

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

4 Principles

The residues of organophosphorus drugs in the sample were extracted by acidified acetonitrile, purified by PSA adsorbent, and measured by liquid chromatography-tandem mass spectrometry with internal standards:

Legal quantity:

5:1 Reagents

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

5:1:2 Acetic acid (CH3COOH): superior grade pure:

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

5:1:4 Anhydrous sodium acetate (CH3COONa): superior grade pure:

5:2 Solution preparation

5:2:1 Stable volume solution: Take 150 mL of acetonitrile, add 350 mL of water and 0:5 mL of formic acid, and mix well:

5:2:2 1% acetic acid acetonitrile: Take 990mL of acetonitrile, add 10mL of acetic acid, and mix:

5:3 Standard products

5:3:1 Phim, pamiphos, fenthion, malathion, diazinon, trichlorfon, dichlorvos, picolinate and pyritin: the contents are all >=98:0%, see Appendix A for details:

5:3:2 Fenthion-Ds, malathion-Do, diazinon-Do, dichlorvos-Ds, picolinate-Ds and pyritin-Do: the concentrations are all 100 mg/L, see the appendix for details A:

5:4 Preparation of standard solution

5:4:1 Standard stock solution: Under light-proof conditions, take appropriate amounts of organophosphorus drug standards (equivalent to 10 mg of each component to be tested), weigh them accurately, and add acetonitrile (5:1: 1) Dissolve appropriate amount and dilute to volume: Prepare standard stock solutions with a concentration of 1 mg/mL: Stored below -18 degrees C, valid for 6 months:

5:4:2 Mixed standard intermediate solution: Measure an appropriate amount of the

standard stock solution (5:4:1), dilute it with acetonitrile (5:1:1), and prepare the concentrations as follows: 10 mg/L malathion and 10 mg malathion: /L, diazinon 10 mg/L, trichlorfon 10mg/L, dichlorvos 10mg/L, picolinate 10 mg/L; phoxim 20 mg/L, fenthion 20mg/L and pyritin 20 mg/L mixed standard intermediate solution: Store at 0 degrees C~4 degrees C away from light, valid for 1 month:

5:4:3 Mixed standard working solution: Accurately measure an appropriate amount of the mixed standard intermediate solution (5:4:2), dilute it with acetonitrile (5:1:1), and prepare the concentrations as follows: 1 mg/L of paminephos and 1 mg/L of malathion: of Mix standard working fluid: Store at 0 degrees C~4 degrees C away from light, valid for 1 week:

5:4:4 Mix the internal standard intermediate solution: accurately draw the internal standard standard material solution (5:3:2), dilute it with acetonitrile (5:1:1) to prepare malathion-D, diazinon-Do, dichlorvos-D and formazan: Pyridinophos-D; both are 5 mg/L, Fenthion-D6 and Mythophos-Do are both 10 mg/L mixed internal standard intermediate solutions, stored at 0 degrees C~4 degrees C in the dark, and are valid for 1 month:

5:4:5 Mix the internal standard working solution: accurately absorb the internal standard intermediate solution (5:4:4), dilute it with acetonitrile (5:1:1) to prepare malathion-Do, diazinon-Do, dichlorvos-Ds and methyl Pyridinophos-Da0:5 mg/L, Fenthion-D: Mixed internal standard working solution with Phytophen-D1mg/L, store in the dark at 0 degrees C~4 degrees C, valid for 2 weeks to 3 weeks:

5:5 Materials

5:5:1 PSA (N-propylethylenediamine) adsorbent: particle diameter is 40 μm ~ 60 μm:

5:5:2 PVDF (polyvinylidene fluoride) filter membrane: pore size is 0:22μm:

6Instruments and Equipment

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

6:2 Electronic balance: sensitivity 0:00001g and 0:01g:

6:3 Homogenizer:

6:4 Centrifuge: the rotation speed is not less than 8000 r/min:

6:5 ultrasonic cleaner,

6:6 Vortex mixer:

6:7 Chlorine 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 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:

8 Measurement steps

8:1 Extraction

Take 5 g of sample (accurate to +/-0:02 g), add 100 μL of mixed internal standard working solution (5:4:5), 25:0 mL of 1% acetic acid acetonitrile (5:2:2), and no

Add 1g sodium acetate (5:1:4), vortex and mix for 2 minutes, sonicate for 30 minutes, and centrifuge at 8000 r/min for 10 minutes; take the supernatant and set aside:

8:2 Purification

Add 500 mg of PSA adsorbent to the reserve solution, vortex for 1 min, and centrifuge at 8000 r/min for 10 min; take 5:0 mL of the supernatant, blow dry with nitrogen at 45°C, and accurately measure 1:0 mL of constant volume solution (5:2:1) Dissolve the residue, add:200 mg of PSA adsorbent, vortex for 1 min, and centrifuge at 5000 r/min for 10 min: The supernatant is passed through the membrane and used for liquid chromatography-tandem mass spectrometry: During the experiment, care should be taken to avoid direct sunlight: After sample pre-processing,

After completion, it should be tested on the machine in time:

8:3 Preparation of standard curve

Accurately measure an appropriate amount of mixed standard working solution (5:4:3) and 20 μL of mixed internal standard working solution (5:4:5), and dilute it with constant volume solution (5:2:1) into pamiphos, malathion, diazinon, The concentrations of trichlorfon, dichlorvos and picolinate are 10μg/L, 20μg/L, 50μg/L, 100μg/L,:200pg/L and 500μg/L respectively, and the concentrations of phoxim, fenthion and pyritin are respectively It is a series of standard solutions of 20 μg/L, 40 μg/L, 100 μg/L,:200 μg/L, 400 μg/L, and 1000 μg/L, for liquid chromatography-tandem mass spectrometry determination: Draw a standard curve using the ratio of the peak area of each standard to the peak area of the internal standard as the ordinate and the concentration of each standard solution as the abscissa:

8:4 Determination