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NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB 31656.4-2021

National food safety standard - Determination of chlorpromazine residue in fishery products by liquid chromatography-tandem 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:

This document is published for the first time:

National food safety standards

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

1 Scope

This document specifies the sample preparation and liquid chromatography-tandem mass spectrometry determination methods for the detection of chlorpromazine residues in aquatic products:

This document is applicable to the detection of chlorpromazine residues in edible tissues of fish, shrimp and crabs:

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

GB/T 30891-2014 Sampling specifications for aquatic products

3 Terms and definitions

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

4 Principles

The remaining chlorpromazine in the sample was extracted with acetonitrile, purified with a mixed cationic solid-phase extraction column, and measured by liquid chromatography-tandem mass spectrometry, using the internal standard method:

Quantitative:

5 Reagents and materials

The reagents used below are of analytical grade unless otherwise noted, 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 (CH3OH): chromatographically pure:

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

5:1:4 Ammonium acetate (NH4C2H3O2):

5:1:5 Ammonia (NH3.H2O):

5:2 Standard products

5:3 Solution preparation

5:3:1 5% ammoniated methanol solution: Take 5 mL of ammonia water and 95 mL of methanol, and mix well:

5:3:2 2mmol/L ammonium acetate solution: Take 0:154g of ammonium acetate, add 1mL of formic acid, dissolve with appropriate amount of water and dilute to 1000mL:

5:3:3 30% methanol-ammonium acetate solution: Take 30 mL of methanol and 70 mL of 2 mmol/L ammonium acetate solution, and mix well:

5:4 Preparation of standard solution

5:4:1 Chlorpromazine standard stock solution (100 μg/mL): Take about 11 mg of chlorpromazine hydrochloride standard, weigh it accurately, add an appropriate amount of methanol to dissolve and dilute to a 100 mL volumetric flask, shake well, and you get : Store in the dark below -18 degrees C, valid for 6 months:

5:4:2 Internal standard stock solution (100 μg/mL): Take about 11 mg of chlorpromazine hydrochloride-D6 standard, weigh it accurately, add an appropriate amount of methanol to dissolve and dilute to a 100 mL volumetric flask, shake well, and you get : Store in the dark below -18 degrees C, valid for 6 months:

5:4:3 Chlorpromazine standard intermediate solution (500ng/mL): Accurately measure an appropriate amount of chlorpromazine standard stock solution and dilute it with methanol to make a concentration of

500ng/mL chlorpromazine standard intermediate solution: Store in a dark place below -18°C and is valid for 3 months:

5:4:4 Internal standard working solution (200ng/mL): Accurately measure an appropriate amount of the internal standard stock solution, dilute it with methanol to prepare a chlorpromazine-D6 internal standard working solution with a concentration of:200ng/mL: Store in the dark below -18°C ,valid for 3 months:

5:5 Materials

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

5:5:2 Aqueous polysulfone ether needle filter: 0:22μm:

6 Instruments and equipment

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

6:2 Balance: sensitivity 0:01g and 0:00001g:

6:3 Homogenizer:

6:4 Centrifuge: 6000r/min:

6:5 Rotary evaporator:

6:6 Vortex mixer:

6:7 Ultrasonic cleaner:

7 Measurement steps

7:1 Preparation of specimens

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

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

b) Take the blank sample after homogenization as the blank sample;

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

7:2 Storage of specimens

Stored below -18 degrees C, the validity period is 3 months:

8 Measurement steps

8:1 Extraction

Take 5g of the sample (accurate to +/-0:05g) in a 50mL centrifuge tube, add 50μL of internal standard working solution, add 15mL of acetonitrile, homogenize for 1 minute, and ultrasonic

Sonicate for 10 min, centrifuge at 4000 r/min for 7 min, transfer the supernatant to another 50 mL centrifuge tube; take another 10 mL of acetonitrile and clean the homogenizer for 30 s, pour the washing liquid into the residue, vortex and mix for 2 min, ultrasonic for 10 min, 4000 r/min Centrifuge for 7 minutes, combine the supernatant, centrifuge at 6000r/min for 5 minutes, take the supernatant and set aside:

8:2 Purification

Take the solid phase extraction column, activate it with 5 mL each of methanol and water, pass the supernatant through the column at a speed of about 1 drop per second, and elute with 5 mL of methanol:

Discard the effluent and drain it: Elute with 3 mL of 5% ammoniated methanol solution: Collect the eluate and blow dry with nitrogen at 40°C: Add 30% methanol-ethyl alcohol accurately:

Dissolve the residue in 1 mL of ammonium acid solution and filter it with an aqueous needle filter into an injection vial for liquid chromatography-tandem mass spectrometry measurement:

8:3 Preparation of matrix standard curve

Accurately measure appropriate amounts of 500ng/mL chlorpromazine standard intermediate solution and:200ng/mL internal standard working solution, blow dry with nitrogen at 40°C, and use blank matrix

The solution was prepared into a series of matrices with an isotope internal standard concentration of 10ng/mL and chlorpromazine concentrations of 2:5ng/mL, 5:0ng/mL,