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NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB 31656.17-2022

National Food Safety Standard - Determination of Dithiocyanomethane Residues in Aquatic Products by Gas Chromatography

Issued on: September 20, 2022 **Implemented on: February 1, 2023**

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

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foreword

This document is in accordance with the provisions of GB/T 1.1-2020 "Guidelines for Standardization Work Part 1.Structure and Drafting Rules for Standardization Documents"

drafting.

This document is published for the first time.

National Food Safety Standards

Determination of Dithiocyanomethane Residues in Aquatic Products by Gas Chromatography

1 Scope

This document specifies the sample preparation and gas chromatography determination methods for the determination of dithiocyanomethane residues in aquatic products.

This document is applicable to the determination of dithiocyanomethane residues in edible tissues of fish, shrimp, crab, turtle and other aquatic products.

2 Normative references

The content in the following documents constitutes the essential provisions of this document through normative references in the text. Among them, the dated reference documents,

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

document.

GB/T 6682 Analytical laboratory water specifications and test methods

GB/T 30891-2014 Sampling Specifications for Aquatic Products

3 Terms and Definitions

This document does not have terms and definitions that need to be defined.

4 principles

The remaining dithiocyanomethane in the sample was extracted with a mixture of

dichloromethane and n-hexane, degreased, purified by a neutral alumina solid-phase extraction column, and gas

Determined by phase chromatography and quantified by external standard method.

5 Reagents and materials

Unless otherwise specified, all reagents are of analytical grade, and the water is first-class water in accordance with GB/T 6682.

5.2 Solution preparation

Dichloromethane-n-hexane (1.1, V/V). take 250mL of dichloromethane and n-hexane respectively, and mix well.

5.3 Standards

Methylenebisthiocyanate (Methylenebisthiocyanate, C3H2N2S2, CAS number. 6317-18-6), content >=99%.

5.4 Preparation of standard solution

5.4.1 Standard stock solution. Accurately weigh 10mg of dithiocyanomethane standard substance, dissolve it with an appropriate amount of acetonitrile and dilute to 50mL brown

The volumetric flask was prepared into a standard stock solution with a concentration of.200 μg/mL. Stored below -18°C, the validity period was 3 months.

5.4.2 Standard working solution. Accurately measure an appropriate amount of dithiocyanomethane standard stock solution, dilute it with dichloromethane, and prepare a concentration of

0.05μg/mL, 0.1μg/mL, 0.5μg/mL, 1.0μg/mL, 2.0μg/mL, 5.0μg/mL and 10.0μg/mL series standards

Working fluid. Ready to use.

5.5 Materials

Neutral alumina solid phase extraction column, packing 60mg/3mL, or equivalent.

7.1 Preparation of samples

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

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 the blank.

7.2 Storage of samples

Store frozen below -18°C.

8.1 Extraction

Take 5g of the sample (accurate to +/-0.05g), put it in a 50mL stoppered glass centrifuge tube, add dichloromethane-n-hexane (1.1, V/V)

15 mL, immediately vortexed for 1 min, ultrasonically extracted for 10 min, centrifuged at 4000 r/min for 5 min, and the supernatant was taken into a chicken heart bottle with a glass dropper.

Repeat the extraction once, combine the supernatants of the two times, mix well, and blow nitrogen at 40°C to 0.5mL-1.0mL, and set aside.

8.2 Purification and concentration

Take a neutral alumina solid-phase extraction column, add 1.0 g of anhydrous sodium sulfate to the upper layer, pass the spare liquid through the column and collect it, and use dichloromethane-n-hexane

(1.1, V/V) 2.0 mL washes 8.1 chicken heart bottles containing the extract, passes through the column, and collects them in 15 mL glass test tubes, and then washes them with dichloromethane.

8mL eluted small column, and collected in the same glass test tube, dried with nitrogen at 40°C, added dichloromethane-n-hexane (1.1, V/V) 1.0mL, vortexed

8.3 Preparation of standard curve

Precisely measure the appropriate amount of standard working solution for gas chromatography determination. Take the measured peak area as the ordinate, and the corresponding standard solution concentration as the abscissa

Coordinates, draw a standard curve. Find the regression equation and correlation coefficient.

8.4.1 Chromatographic conditions

a) Chromatographic column. HP-5MS quartz capillary column (30mx0.32mm, particle size 0.25μm), or equivalent;

b) Injection method. splitless injection;