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SN

INDUSTRY STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

SN/T 3543-2013

Determination of hexabromocyclododecane(HBCD) in food for export. Liquid chromatography-tandem mass spectrometry method

出口食品中六溴环十二烷的测定 液相色谱-质谱/质谱法

(English Translation)

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Foreword

Accordance with the standards GB/T 1.1-2009, GB/T 20001.4-2001 and rules of SN/T 0001-1995 given draft.

This standard is proposed and managed by the National Certification and Accreditation Administration Committee.

This standard was drafted. People's Republic of China Hubei Exit Inspection and Quarantine.

1 Scope

This standard specifies the food of three six HBCD isomers (alpha-HBCD, beta-HBCD and gamma-HBCD) content of liquid chromatography -Mass spectrometry/mass spectrometry.

This standard applies to fish, shrimp, eggs and other foods alpha-HBCD, beta-HBCD and gamma-HBCD Determination and confirmation.

2 Normative references

The following documents for the application of this standard is essential. For dated references, only the dated version suitable for use herein Member. For undated references, the latest edition (including any amendments) applies to this document.

Laboratory use specifications and test methods GB/T 6682 Analysis

3 Method summary

Three samples six HBCD isomers (alpha-HBCD, beta-HBCD and gamma-HBCD) by Soxhlet extraction, the extract by gel permeation chromatography Purification column, followed by liquid chromatography - mass spectrometry/mass spectrometer using the external standard or internal standard (that is, using isotope dilution technique, the sample extract Before taking added three kinds of deuterated six HBCD d18-alpha-HBCD,

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d18-beta-HBCD and the internal standard solution of d18-gamma-HBCD) Determination of Three hexa Cyclododecane isomer content.

4 Reagents and materials

Unless otherwise indicated, the reagents used were of analytical grade water as a water GB/T 6682 regulations.

4.1
hexane. chromatographically pure.

4.2
dichloromethane. chromatography.

4.3
Methanol. chromatographically pure.

4.4
acetonitrile. chromatography.

4.5
anhydrous sodium. 650 °C burning 4h, cooled in a desiccator to room temperature and stored in a sealed bottle aside.

4.6
n-hexane - methylene chloride solution (11). Take 200mL n-hexane (4.1), was added 200mL of methylene chloride (4.2) and mix.

4.7
alpha-HBCD, beta-HBCD and gamma-HBCD single standard solution. concentration was 100μg/mL.

4.8
d18-alpha-HBCD, d18-beta-HBCD and d18-gamma-HBCD deuterated single standard solution. concentration was 50μg/mL.

4.9
alpha-HBCD, beta-HBCD and gamma-HBCD single standard stock solution. draw a single standard solution (4.7), diluted with methanol (4.3), minutes Do not be formulated at a concentration of 1μg/mL standard stock solution.

4.10
internal standard mix solution I. lessons d18-alpha-HBCD, d18-beta-HBCD and d18-gamma-HBCD deuterated single standard solution (4.8), with methanol (4.3), and

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diluting the d18-alpha-HBCD, d18-beta-HBCD and d18-gamma-HBCD concentrations of 100ng/mL, 50ng/mL and the internal standard 50ng/mL in the mixed solution.

4.11

internal standard mix solution II. lessons internal standard mix solution I (4.10), diluted with methanol (4.3) to prepare a d18-alpha-HBCD, d18-beta-HBCD and d18-gamma-HBCD concentrations were 10ng/mL, internal standard 5ng/mL and 5ng/mL of the mixed solution.

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