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SN

INDUSTRY STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

SN/T 3544-2013

Determinations of perfluorooctanoate(PFOA) and perfluorooctane sulfonate (PFOS)in food for export. Liquid chromatography-tandem mass spectrometry method

出口食品中全氟辛酸和全氟辛烷磺酸盐的测定 液相色谱-质谱/质谱法

(English Translation)

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Foreword

This standard is drafted in accordance with the rules given in GB/T 1.1-2009, GB/T.20001.4-2001 and SN/T 0001-1995.

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

The drafting unit of this standard. Hubei Entry - Exit Inspection and Quarantine Bureau of the People's Republic of China.

1 Scope

This standard specifies the content of perfluorooctanoic acid (PFOA) and perfluorooctane sulfonate (PFOS) in liquid chromatography-mass spectrometry/mass spectrometry Method.

This standard applies to fish, shrimp, eggs and other foods in the PFOA and PFOS content of the determination and confirmation.

2 Normative reference documents

The following documents are indispensable for the application of this standard. For dated references, only the dated edition applies to this article Pieces. For undated references, the latest edition (including all modifications) applies to this document.

GB/T 6682 Analytical laboratory water specifications and test methods

3 Method summary

PFOA and PFOS were extracted by solvent. The extract was purified by silica gel column chromatography and then purified by liquid chromatography-mass spectrometry Quantification by external standard method or internal standard method (ie, using isotope dilution technique, before adding 13C8-PFOA and 1,2,3,4-13C4-PFOS internal standard solution) to determine the content of PFOA and PFOS.

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4 Reagents and materials

Unless otherwise stated, the reagents used were of analytical grade and water was the primary water specified in GB/T 6682.

4.1
methanol. pure chromatography.

4.2
dichloromethane. chromatography pure.

4.3
Acetone. Chromatographic pure.

4.4
ammonia. excellent grade pure.

4.5
methyl tert-butyl ether (methyltert-butylether, MTBE).

4.6
tetrabutylammonium hydrogen sulfide (TBA).

4.7
ammonium acetate.

4.8
Sodium carbonate.

4.9
silica gel.

4.10
ammonium acetate solution (2mmol/L). accurately weighed 0.1542g ammonium acetate (4.7) dissolved in 1000mL water.

4.11
TBA solution (0.5mol/L, pH 10). accurately weighed 42.45gTBA (4.6) dissolved in 250mL water, and ammonia Water adjusted to pH 10.

4.12
sodium carbonate solution (0.25mol/L). accurately weighed 13.25g sodium carbonate (4.8) dissolved in 500mL of water.

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4.13
PFOA and PFOS single standard solution. concentration of 100μg/mL.

4.14 13C8-PFOA and 1,2,3,4-13C4-PFOS carbon 13 single standard solution. concentration of 50μg/mL.

4.15
PFOA and PFOS single standard stock solution. absorb a single standard solution (4.13), diluted with methanol (4.1), were prepared into concentrated

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