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GB 5009.205-2024

National Food Safety Standard

Determination of Toxic Equivalents of Dioxins and Their Analogues in Food

食品安全国家标准 食品中二噁英及其类似物毒性当量的测定

National Health Commission of the People's Republic of China
State Administration for Market Regulation

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National Standard of the People's Republic of China

GB 5009.205-2024
In Replacement of GB 5009.205-2013

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Foreword

This standard replaces GB 5009.205-2013 “National Food Safety Standard Determination of Toxic Equivalents of Dioxins and Their Analogues in Foods”. Compared with GB 5009.205-2013, the main changes of this standard are as follows.

——the second method “isotope dilution-gas chromatography-triple quadrupole mass spectrometry” has been added;

——the toxicity equivalence factor (TEF) established by the World Health Organization (WHO) in 1998 has been deleted;

——the expression and format of the first method “Isotope dilution-gas chromatography-magnetic high-resolution mass spectrometry” have been modified.

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1 Scope

This standard specifies the determination methods for the contents of 17 2,3,7,8-substituted polychlorinated dibenzodioxins and polychlorinated dibenzofurans (PCDD/Fs) and 12 dioxin-like polychlorinated biphenyls (DL-PCBs) in foods and their toxic equivalent quantities (TEQ) (see Table A.1 in Annex A).

The first method, “isotope dilution-gas chromatography-magnetic high-resolution mass spectrometry”, is applicable to the determination of the content and TEQ of 17 PCDD/Fs and 12 DL-PCBs in food.

The second method, “isotope dilution-gas chromatography-triple quadrupole mass spectrometry”, is applicable to the determination of the contents and TEQ of 17 PCDD/Fs and 12 DL-PCBs in meat and meat products, aquatic animals and their products, milk and dairy products, eggs and egg products, and oils and fats.

Method I: Isotope Dilution-Gas Chromatography-Magnetic High Resolution Mass Spectrometry

2 Principle

After extraction, purification and concentration, the samples were determined by gas chromatography-magnetic high-resolution mass spectrometry and quantified by the stable isotope dilution method; the TEQ of PCDD/Fs and DL-PCBs in the samples was calculated by multiplying the toxic equivalent factor (TEF) of each target compound with the measured content and then adding them up.

3 Reagents and materials

3.1 Reagents

Unless otherwise specified, all reagents used in this method were of analytical grade and the water was grade 1 water as specified in GB/T 6682.

3.1.1 Acetone (C_3H_6O) : pesticide residue level.

3.1.2 n-Hexane (C_6H_{14}): pesticide residue level.

3.1.3 Toluene (C_7H_8): pesticide residue level.

3.1.4 Cyclohexane (C_6H_{12}): pesticide residue level.

3.1.5 Dichloromethane (CH_2Cl_2): pesticide residue level.

3.1.6 Methanol (CH_3OH) : chromatographic grade.

3.1.7 n-Nonane (C_9H_{20}): ≥99%.

3.1.8 Ethyl acetate ($CH_3COOCH_2CH_3$): pesticide residue grade.

3.1.9 Anhydrous sodium sulfate (Na_2SO_4): high purity.

3.1.10 Concentrated sulfuric acid (H_2SO_4): high-grade purity.

3.1.11 Sodium hydroxide (NaOH): high purity.

3.1.12 Silver nitrate ($AgNO_3$): high purity.

3.1.13 Diatomaceous earth for extraction: bulk density 18.0 g /100 mL ~ 36.0 g/100 mL (such as EXtrelutNT, or equivalent products).

3.1.14 Gel chromatography filler : polystyrene gel , 38μm ~ 75μm (such as Bio-Beads S-X3 , or equivalent products).

3.1.15 Silica gel: 75μm ~250μm.

3.1.16 Basic alumina: 63μm ~200μm (such as EcoChrom™ MP Alumina B-Super I , or equivalent).

3.1.17 Florisil: 150μm ~ 250μm.

3.1.18 Activated carbon : 150μm ~ 180μm (such as Carbopack C , Supelco 10258, or equivalent).

3.1.19 Diatomaceous earth for purification: 26μm (such as Celite 545 AW, Supelco 20199-U, or equivalent).

3.2 Reagent preparation

3.2.1 Activated silica gel: The silica gel is cleaned with methanol and then with dichloromethane. After the solvent evaporates, it is baked at 600℃ for at least 12h and prepared before use.

3.2.2 Acidified silica gel (44%, mass fraction): Weigh 112.0 g of activated silica gel into a 250 mL stoppered ground-mouth rotating flask, add 88.0 g of concentrated sulfuric acid, seal the flask and oscillate on a shaker until the silica gel is in a uniform flowing state. Prepare it before use.

3.2.3 Alkalinized silica gel (33%, mass fraction): Weigh 100.0 g of activated silica gel into a 250 mL stoppered ground-mouth rotating flask, add 49.0 g of 1 mol/L sodium hydroxide solution, seal the flask and oscillate on a shaker until the silica gel is in a uniform flowing state. Prepare it before use.

3.2.4 Silver nitrate silica gel: Weigh 10.0g of silver nitrate into a 250mL stoppered ground -mouth rotating flask, add 40mL of water to dissolve it, then slowly add 90.0g of activated silica gel, seal it and place it on a shaker to oscillate until the silica gel is in a uniform flowing state. Prepare it before use.

3.2.5 Basic alumina: Activate basic alumina at 600℃ for 24h and prepare it before use.

3.2.6 Hydrous Florisil (1%, mass fraction): Take an appropriate amount of Florisil and place it into a Soxhlet extractor. Use n-hexane: dichloromethane (1:1, volume ratio) to extract for 24 h. After the solvent evaporates, weigh 99.0 g, add 1.0 mL of water, mix well in a sealed container (such as a stoppered glass flask or a stoppered glass conical flask), and prepare it before use.

3.2.7 Mixed activated carbon: weigh 9.0g activated carbon and 41.0g diatomaceous earth for purification, mix them evenly in a sealed container (such as a stoppered glass flask or a stoppered glass conical flask), then activate them at 130℃ for 6h and prepare them before use.

3.2.8 Anhydrous sodium sulfate: calcine anhydrous sodium sulfate at 660℃ for 6h and prepare it before use.

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