NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

GB 5009.205-2024

National food safety standard - Determination of toxic equivalents of dioxins and their analogues in food

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1 Scope

This Standard specifies the determination method of the contents of 17 kinds of 2,3,7,8-substituted polychlorodibenzo-p-dioxin, polychlorino-dibenzofura (PCDD/Fs) and 12 kinds of dioxin-like polychlorinated biphenyl (DL-PCBs) and their toxic equivalent (TEQ) in food (see Table A.1 in Appendix A).

Method I "Isotope dilution - gas chromatography - magnetic high-resolution mass spectrometry" applies to the determination of the content of 17 kinds of PCDD/Fs and

12 kinds of DLPCBs and their TEQ in food.

Method II "Isotope dilution - gas chromatography - triple quadrupole mass spectrometry" applies to the determination of the content of 17 kinds of PCDD/Fs and

12 kinds of DL-PCBs and its TEQ in meat and meat products, aquatic animals and their products, milk and dairy products, eggs and egg products, oils, and fats.

Method I - Isotope dilution - gas chromatography - magnetic high-resolution mass spectrometry

2 Principle

After the sample is extracted, purified and concentrated, measure by a gas chromatography - magnetic high-resolution mass spectrometer and quantify by the stable isotope dilution method; calculate the TEQ of PCDD/Fs and DL-PCBs in the sample by accumulating the toxic equivalency factor (TEF) of each target compound multiplied by the measured content.

3.1 Reagents

Unless otherwise specified, all the reagents in this method are analytical reagents, and the water is grade-1 water specified by GB/T 6682.

3.1.1 Acetone (C3H6O): pesticide residue grade.

it and place it on a shaker to shake until the silica gel is in a uniform flow state. Prepare it immediately before use.

3.2.3 Alkalized silica gel (33%, mass fraction): Weigh 100.0 g of active silica gel into

a 250 mL stoppered ground-rotatory flask; add 49.0 g of 1 mol/L sodium hydroxide solution; seal it and place it on a shaker to shake until the silica gel is in a uniform flow state. Prepare it immediately before use.

3.2.4 Silver nitrate silica gel: Weigh 10.0 g of silver nitrate into a 250 mL stoppered

ground-rotatory flask; add 40 mL of water to dissolve it; then, slowly add 90.0 g of active silica gel; seal it and place it on a shaker to shake until the silica gel is in a uniform flow state. Prepare it immediately before use.

3.2.5 Alkaline alumina: Take alkaline alumina and activate it at 600 °C for 24 hours.

Prepare it immediately before use.

3.2.6 Hydrous florisil (1%, mass fraction): Put an appropriate amount of florisil into a

Soxhlet extractor; use n-hexane: dichloromethane (1:1, volume ratio) to extract it for

24 hours; after evaporating, weigh 99.0 g; add 1.0 mL of water; mix evenly in a closed container (such as a stoppered glass flask or a stoppered glass conical flask); prepare immediately before use.

3.2.7 Mixed activated carbon: Weigh 9.0 g of activated carbon and 41.0 g of

diatomaceous earth for purification; mix evenly in a closed container (such as a stoppered glass flask or a stoppered glass conical flask); then, activate at 130 °C for 6 hours. Prepare immediately before use.

3.2.8 Anhydrous sodium sulfate: Take anhydrous sodium sulfate and burn it at 660 °C

for 6 hours. Prepare immediately before use.

3.3.1 Standard solution for separation degree check: solution containing natural

PCDD/Fs. For specific compounds and concentrations, see Table B.1 in Appendix B.

3.3.2 Standard solution for PCDD/Fs isotope labeled quantitative internal standard:

solution containing 15 kinds of 13C12-PCDD/Fs. See Table B.2 for specific compounds and concentrations.

3.3.3 Standard solution for PCDD/Fs isotope labeled recovery rate internal standard:

solution containing 13C12-1,2,3,4-TCDD and 13C12-1,2,3,7,8,9-HxCDD. See Table B.3 for specific concentrations.

3.3.4 Standard solution for PCDD/Fs calibration: standard solution containing natural

and isotope-labeled PCDD/Fs series calibration curve. See Table B.4 for specific compounds and concentrations.

3.3.5 Standard solution for DL-PCBs isotope labeled quantitative internal standard:

solution containing 12 kinds of 13C12-DL-PCBs. See Table B.5 for specific compounds and concentrations.

3.3.6 Standard solution for DL-PCBs isotope labeled recovery rate internal standard:

solution containing 13C12-PCB 70, 13C12-PCB 111 and 13C12-PCB 170. See Table B.6 for specific concentrations.

3.3.7 Standard solution for DL-PCBs calibration: standard solution containing natural

and isotope-labeled DL-PCBs series calibration curve. See Table B.7 for specific compounds and concentrations.

3.3.8 Standard solution for sensitivity check: Standard solution containing natural and

isotope-labeled PCDD/Fs series. See Table B.8 for specific compounds and concentrations.

Note: Store the standard solution at room temperature in the dark, where it has a shelf life of 6 years.

4.12 Muffle furnace: able to maintain constant temperature (+/-10 degrees C) within the range

of 200 degrees C ~ 700 degrees C.

4.13 Oven: able to maintain constant temperature (+/-5 degrees C) within the range of 105 degrees C ~

250 degrees C.

4.14 Glass chromatography column: equipped with polytetrafluoroethylene plunger, 150 mm (length) x 8 mm (inner diameter), 300 mm (length) x 15 mm (inner diameter).

4.15 Fully automatic sample purification system: equipped with acid-base composite

silica gel column, alkaline alumina column and activated carbon purification column.

4.16 Gel permeation chromatography column (GPC): glass column (inner diameter 15

mm ~ 20 mm), containing 50 g of S-X3 gel, or equivalent fully automatic GPC.

5.1.1 General food samples: After dry food is crushed evenly, weigh 10 g ~ 20 g

(accurate to 0.01 g) for later use; for milk powder, weigh 15 g (accurate to 0.01 g) for later use; after other samples are homogenized, weigh 50 g ~200 g (accurate to 0.01 g);

freeze-dried for later use.