



**NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

GB 5009.215-2016

**National food safety standard - Determination of Organotin in
Food**

食品安全国家标准 食品中有机锡的测定
(English Translation)

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1 Scope

This Standard specifies gas chromatography - pulse flame photometric detector detection method for organotin in food.

This Standard is applicable to the determination of dimethyltin, trimethyltin, monobutyltin, dibutyltin, tributyltin, phenyltin, diphenyltin and triphenyltin in samples like fish, shellfish, wine and soy sauce.

2 Principle

Respectively take monomethyltin as the internal standard of monosubstituted organotin; take tripropyltin as the internal standard of disubstituted organotin and trisubstituted organotin. Adopt internal standard method to quantify. In the sample, quantitatively add monomethyltin and tripropyltin internal standard. Through the assistance of ultrasound, extract organotin; use organic solvent to extract it. The extracted sample solution shall receive gel permeation chromatographic purification, pentyl Grignard reagent derivatization. The derivatized product shall receive Florisil purification. Adopt gas chromatography - pulse flame photometric detector (GC-PFPD) for determination.

3 Reagents and Materials

Unless it is otherwise stipulated, all reagents used in this Method shall be analytically pure. Water shall be Grade-1 water stipulated in GB/T 6682.

3.1 Reagents

3.1.1

N-hexane (n-C₆H₁₄)

Re-distilled.

3.1.2

Tetrahydrofuran (CH₂CH₂OCH₂CH₂)

Re-distilled.

3.1.3

Ethyl acetate (CH₃COOC₂H₅)

Re-distilled.

3.1.4

Cyclohexane (C₆H₁₂)

Re-distilled.

3.1.5

Methanol (CH₃OH).

3.1.6

Anhydrous ether (C₂H₅OC₂H₅).

3.3.1 Organotin standard substance and its standard solution: please refer to Appendix

A. The purity of dimethyltin and tributyltin is 95%, other than this, the purity of other standard substances shall all be more than 97%.

3.3.2 Internal standard: monomethyltin (MMT) and tripropyltin (TPrT) standard substance: purity > 98%.

3.4.1 Organotin standard stock solution: accurately weigh-take a proper amount of

organotin standard substance, then, place it in a 10 mL beaker. Add methanol - aqueous solution (3.2.4) to dissolve it, then, transfer it to a 10 mL volumetric flask. In addition, dilute it to the scale. Store it in the refrigerator at -20 °C.

3.4.2 Internal standard - standard stock solution: accurately weigh-take a proper

amount of the internal standard, then, place it in a 10 mL beaker. Add methanol - aqueous solution (3.2.4) to dissolve it, then, transfer it to a 10 mL volumetric flask. In addition, dilute it to the scale. Store it in the refrigerator at -20 °C.

3.4.3 Organotin standard and internal standard intermediate solution: measure-take a

proper amount of organotin standard or internal standard stock solution. Use methanol - aqueous solution (3.2.4) to dilute it by 100 times. Please refer to Appendix for the mass concentration. Store it in the refrigerator at -20 °C.

3.4.4 Organotin standard and internal standard working solution: measure-take a

proper amount of organotin standard or internal standard intermediate solution; place it in a 10 mL volumetric flask. Add methanol - aqueous solution (3.2.4); dilute to the scale. Please refer to Appendix A for the concentration. Store it in the refrigerator at -20 °C.

4 Instruments and Equipment

4.1

Gas chromatograph (GC)

Equipped with pulse flame photometric detector (PFPD), sulfur filter.

4.2 Chromatographic column: DB-1 capillary column or equivalent column; column

length: 30 m; membrane thickness: 0.25 µm; internal diameter: 0.25 mm.

4.3 Tissue homogenizer.

4.4 Oscillator.

4.5 Ultrasonic cleaner.

4.6 Rotary evaporator.

4.7 Nitrogen concentrator.

and drying may be adopted to make freeze-dried powder. Place it at a low temperature of -20 °C for storage.

5.2.1.1 Fish sample

Accurately weigh-take a proper amount of sample; add 0.15 g of ethylene diamine tetra-acetic acid and 5 mL of 20 g/L sodium chloride solution; shake it up. Add 50 μ L of internal standard working solution; add 15 mL of hydrobromic acid - tetrahydrofuran solution (1 + 20) (3.2.5); conduct ultrasound for 5 min. If the sample is wet, add 1 mL of saturated sodium chloride solution; shake it up. Then, add 50 μ L of internal standard working solution; add 15 mL of hydrobromic acid - tetrahydrofuran solution (1 + 20) (3.2.5); conduct ultrasound for 5 min.

5.2.1.2 Shellfish sample

Accurately weigh-take a proper amount of sample; add 5 mL of 20% sodium chloride solution; shake it up. Add 50 μ L of internal standard working solution; add 15 mL of hydrobromic acid - tetrahydrofuran solution (1 + 20) (3.2.5); conduct ultrasound for 5 min.

5.2.1.3 Liquid sample, for example, wine

Measure-take 10 mL of sample; add 2 g of sodium chloride; shake it up. Add 50 μ L of internal standard working solution; add 15 mL of hydrobromic acid - tetrahydrofuran solution (1 + 20) (3.2.5); conduct ultrasound for 5 min.

5.2.2 Purification of sample solution

Add 25 mL of n-hexane, which contains 0.03% tropolone, to the sample solution.

Conduct oscillating extraction for 40 min; conduct centrifugation for 10 min (at 3,000 r/min). Place it still for stratification. Absorb organic phase, then, transfer it to an eggplant-shaped bottle. Add 10 mL of n-hexane to the residue; conduct oscillating extraction for 10 min. Conduct centrifugation for 10 min (at 3,000 r/min); place it still for stratification; extract the organic phase. Combine them in the eggplant-shaped bottle; conduct rotary evaporation and concentration, till it reaches near dryness.

5.2.3.1 Filling of gel column: take polystyrene gel; use tetrahydrofuran - ethyl acetate

(1 + 1) solution to soak it overnight. Use glass wool to block the bottom of the glass chromatographic column whose internal diameter is 1.7 cm ~ 1.8 cm. Through wet process, add the previously soaked gel. The gel naturally settles. After it becomes stabilized, the column length is around 15 cm.

5.2.3.2 Purification: add 1 mL of tetrahydrofuran - ethyl acetate (1 + 1) solution to the a) Chromatographic column1: DB-1 column (or equivalent column); column length:

30 m; membrane thickness: 0.25 μ m; internal diameter: 0.25 mm.