



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

GB 23200.92-2016

Replacing SN/T 2445-2010

**Food safety national standard -- Determination of
pentachlorophenol residues in animal-derived foods by liquid
chromatography-mass spectrometry**

动物源性食品中五氯酚残留量的测定 液相色谱-质谱法

(English Translation)

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Foreword

This standard replaces SN/T 2445-2010 "Determination of Pentachlorophenol Residue in Foodstuffs for Import and Export Animal Liquid Chromatography-Mass Spectrometry/Mass Spectrometry law".

Compared with SN/T 2445-2010, the main changes are as follows.

- Standard text format is modified to national standard text format for food safety;

- standard name and "import and export animal food" to "animal food";
- increase the "other food reference implementation" in the standard range.

This standard replaced the previous version of the standard release.

-SN/T 2445-2010.

National standards for food safety Determination of pentachlorophenol residues in animal food by liquid chromatography - mass spectrometry

1 Scope

This standard specifies the preparation of pentachlorophenol residues in animal food and liquid chromatography-mass spectrometry.

This standard applies to pig liver, pig kidney, pork, milk, fish, shrimp, crab and other animal food in the determination of pentachlorophenol residues. Other food Goods can refer to the implementation.

2 Normative reference documents

The following documents are indispensable for the application of this document. 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 2763 National Standard for Food Safety - Maximum Residue Limit of Pesticides in Foodstuffs GB/T 6682 Analytical laboratory water specifications and test methods

3 Principle

The pentachlorophenol residue in the sample was extracted with alkaline aqueous acetonitrile and purified by MAX solid phase extraction column. After concentration and volume, the residue was purified by liquid chromatography-Mass spectrometer detection and confirmation, external standard method quantitative.

4 Reagents and materials

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

4.1 Reagents

4.1.1

Methanol (CH₃OH). Chromatographic Purification.

4.1.2

acetonitrile (CH₃CN). pure chromatography.

4.1.3

Triethylamine (C₆H₁₅N).

4.1.4

Formic acid (HCOOH). Chromatographic pure.

4.1.5

Ammonium acetate (CH₃COONH₄).

4.1.6

Ammonia (NH₃ · H₂O).

4.2 Solution preparation

4.2.1

acetonitrile - water solution (7 3). accurately measure 70mL acetonitrile and 30mL water, mix and shake.

4.2.2 5% triethylamine acetonitrile - water solution. accurately measure 50 mL of triethylamine, transferred into 1000 mL volumetric flask, with acetonitrile - water solution (7 3) Constant volume to the scale, mixed evenly.

4.2.3 5% ammonia solution. the amount of.200mL concentrated ammonia (25%), transferred into the 1000 mL volumetric flask, water volume to the scale, mixed Evenly.

4.2.4 4% formic acid methanol solution. the amount of 10 mL of formic acid, transferred into the 250 mL volumetric flask, with methanol volume to the scale, mixed evenly.

4.2.5 2% formic acid methanol aqueous solution. take a certain amount of 4% formic acid methanol solution and water volume mixed evenly.

4.2.6 5 mmol/L ammonium acetate aqueous solution. accurately weighed 0.3854 g ammonium acetate, dissolved in water, transferred to 1000 mL volumetric flask, set Capacity to scale, mixed evenly.

4.3 Standards

4.3.1

Pentachlorophenol sodium salt (CAS No.. 131-52-2). purity >=94.5%, or equivalent.

4.4 Standard solution preparation

4.4.1

Standard stock solution (200mg/L). Weigh the equivalent of 10 mg (accurate to 0.1 mg) pentachlorophenol standard, with a the alcohol was dissolved and set in a 50 mL brown volumetric flask. This stock solution can be stored for up to 12 months under -18 ° C.

4.4.2

standard working solution. standard working solution according to the need before use with blank sample matrix solution preparation. Store in 4 °C refrigerator.

4.5 Materials

4.5.1

Solid phase extraction (SPE) column. Oasis MAX, 60 mg, 3 mL, or equivalent. Use 5 mL of methanol and 5 mL of water before use activation.

4.5.2

Nitrogen. Purity $\geq 99.99\%$.

5 Instruments and equipment

5.1

Liquid Chromatography Series Quadrupole Mass Spectrometer. Distribution Spray Ion Source.

5.2

Analysis of balance. 0.01 g and 0.0001 g.

5.3

vortex mixer.

5.4

Centrifuge, 5 000 r/min.

5.5

Solid phase extraction vacuum device.

5.6

mechanical vacuum pump.

5.7

Tissue crusher.