



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

GB 23200.91-2016

Replacing SN/T 1776-2006

**Food safety national standard -- Determination of 9
organophosphorus pesticide residues in animal-derived foods
-- Gas chromatographic method**

动物源性食品中9种有机磷农药残留量的测定 气相色谱法

(English Translation)

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Table of Contents

Foreword	
1 Scope	
2 Normative reference documents	
3 Principle	
4 Reagents and materials	
4.1 Reagents	
4.2 Solution preparation	
4.3 Standards	
4.4 Standard solution preparation	
5 Instruments and equipment	
6 Preparation and storage of samples	
7 Analysis steps	
7.1 Extraction	
7.2 Purification	
7.3 Determination	
7.4 Blank experiment	
8 Results are calculated and expressed	
9 Precision	
10.1 Quantitation limits	
10.2 Recovery rate	

Foreword

This standard replaces SN/T 1776-2006 "Method for the determination of nine organophosphorus pesticide residues in foodstuffs of imported and exported animals.

This standard compared with SN/T 1776-2006, the main changes are as follows.

- Standard text format is modified to national standard text format for food safety;
- the name of the "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 1776-2006.

National standards for food safety Determination of 9 organophosphorus pesticide residues in animal - derived foods - Gas chromatographic method

1 Scope

This standard specifies the preparation of the import and export of ham and pickled fish (?) in the 9 kinds of organophosphorus pesticide residue test sample preparation and gas chromatography detection side law.

This standard applies to ham and pickled fish (?) in the dichlorvos, methamidophos, acephate, methyl parathion, malathion, p-Thiophosphate, quinophosphoric acid, halophosphate, triazophos pesticide residues in the detection of other food 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 sample was extracted with acetonitrile and chromatographed on a gel column. The sample was quantitated by a gas chromatograph equipped with a flame photometric detector.

4 Reagents and materials

Unless otherwise specified, all reagents are of analytical grade and water is in accordance with the primary water specified in GB/T 6682.

4.1 Reagents

4.1.1

Ethyl acetate (C₄H₈O₂). Chromatographic purity.

4.1.2

acetonitrile (C₂H₃N). pure chromatography.

4.1.3

Cyclohexane (C₆H₁₂). Chromatographic Purification.

4.2 Solution preparation

4.2.1

GPC eluent. ethyl acetate cyclohexane (1:1, volume ratio). take 100 mL of ethyl acetate, add 100 mL of cyclohexane, shake spare.

4.3 Standards

4.3.1

Organic phosphorus standard. dichlorvos (CAS. 62-73-7), methamidophos, CAS.

10265-92-6), acephate (CAS. 30560-19-1), methyl parathion methyl (CAS.

298-00-0), malathion (CAS. 121-75-5), parathion (CAS. 56-38-2), quinophos (Quinalphos, CAS. 1776-83-6), methidathion (CAS. 950-37-8), triazophos, CAS.

24017-47-8), purity $\geq 99\%$.

4.4 Standard solution preparation

4.4.1

dichlorvos, methamidophos, acephate, methyl parathion, malathion, parathion, quinophosphoric acid, methamphetamine, triazolam Quasi-reserve solution. Weigh the appropriate standard, respectively, with acetone dissolved to 100 mL, the solution concentration of 100 g/mL. Before use According to the need to use acetone diluted to the appropriate concentration, as a mixed standard working fluid. Store in 4 °C refrigerator.

5 Instruments and equipment

5.1

Gas Chromatograph with Flame Photometric Detector (Phosphor Filter 525 nm).

5.2

Analysis of balance. 0.01 g and 0.0001 g.

5.3

Rotary Evaporator 5.4 Gel Purified Finished Product Column (GPC). 25 mmID x 400 mm; Filler. Bio-Beads, S-X3,38 m-75 m (need to do the elution curve before use).

5.5

constant temperature oscillator.

5.6

Mixer.

6 Preparation and storage of samples

A typical sample of about 500 g was taken from all ham or pickled fish samples. The sampling site was carried out according to GB 2763 Appendix A, Crushed with a pulverizer, mixed evenly, are divided into two, respectively, into the clean container as a sample. Sealed and marked with mark.

During sample and sample preparation, contamination of the sample or changes in the residue content should be prevented. Store the sample at -18 ° C ice Save under the box.

7.1 Extraction

Weigh 20 g of the pulverized homogeneous sample (accurate to 0.01 g) in a 100 mL stoppered flask, add 50 mL of acetonitrile at 30 ° C $\pm$ 2 ° C oscillator, shake 2 h, filter, with a small amount of acetonitrile washing residue several times, combined filtrate, set below 40 ° C water bath under reduced pressure to Near dry.

7.2 Purification

Residue with 4.2.1 solvent volume to 10 mL, mix 2 min, centrifuge 5 min, 3000r/min. Take 5.0 mL supernatant over GPC Column at a flow rate of 5 mL/min, eluted with 4.2.1 solvent, discarded the first 100 mL of eluent, collected 100 mL to 165 mL of eluent, 40 ° C below the water bath under reduced pressure to near dry, add 8 mL of ethyl acetate dissolved, quantitative transfer to 10 mL centrifuge tube, below 40 ° C water Bath with a gentle nitrogen flow to dry, ham samples accurately add 1.0 mL of acetone, aquatic products (pickled products) to accurately add 5.0 mL of acetone, Mixing, gas chromatographic determination.

7.3.1 Gas Chromatographic Reference Conditions

A) Column. quartz capillary column, DB-1701, 30 m x 0.53 mm (inner diameter) x 1.0 μ m (film thickness), or equivalent;

B) Carrier gas. nitrogen (purity greater than 99.999%); carrier gas flow rate 10 mL/min; tail gas flow rate. 30 mL/min;

75 mL/min, air flow rate. 100 mL/min;

C) Column temperature. initial temperature 150 ° C for 2 min, rise to 270 ° C at 8 ° C/min for 18 min;

D) Inlet temperature. 250 ° C;

E) Detector temperature. 250 ° C;

F) Injection method. no split injection;