



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

GB 23200.80-2016

Replacing SN/T 0709-2012

**Food safety national standard -- Determination of Dithiophos
Residue in Meat and Meat Products**

肉及肉制品中双硫磷残留量的检测方法

(English Translation)

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Foreword

This standard replaces SN 0709-1997 "Method for the determination of dithiophos residues in meat and meat products".

Compared with SN 0709-1997, the main changes are as follows.

- Standard text format is modified to national standard text format for food safety;
- the name of the "export of meat and meat products" to "meat and meat products";
- "Other food can be referred to in the standard range";
- the sampling step was removed;
- homemade Florian column to commercial column.

This standard replaced the previous version of the standard release.

-SN 0709-1997.

National standards for food safety Determination of the content of dithiophos in meat and meat products

1 Scope

This standard specifies the method for the determination of dithiophos in meat and meat products by gas chromatography-mass spectrometry.

This standard applies to the determination of the amount of dithiophos in export pork, and other foodstuffs can be used for reference.

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 file. 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 residual bisulfophosphamide in the sample was extracted with acetonitrile-methanol mixture (4 1, v/v), the extract was extracted with chloroform, Shrink to dry, the residue was dissolved with n-hexane, the solution was purified by florisil silica column. Eluting with a mixture of ethyl acetate-n-hexane (3 7, v/v) the eluate was concentrated and quantified with n-hexane. The solution was subjected to gas chromatography-mass spectrometry and quantified by external standard method.

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

acetonitrile (C₂H₃N). pesticide residues.

4.1.2

Methanol (CH₃O). Pesticide level.

4.1.3

Trichloromethane (CHCl_3). Pesticide level.

4.1.4

ethyl acetate ($\text{C}_4\text{H}_8\text{O}_2$). pesticide residues.

4.1.5

n-Hexane (C_6H_{14}). Pesticide level.

4.1.6

anhydrous sodium sulfate (Na_2SO_4). 650 °C burning 4 h, stored in a sealed container after cooling and standby.

4.2 Solution preparation

4.2.1

Sodium Sulfate Solution. 5% aqueous solution.

4.2.2

acetonitrile - methanol mixture (4 1, v/v). take 400 mL of acetonitrile, add 100 mL of methanol, shake back.

4.2.3

Ethyl acetate-n-hexane mixture (3 7, v/v). Take 300 mL of ethyl acetate, add 700 mL of n-hexane and shake well.

4.4 Standard solution preparation

4.4.1

Dithiophosphoric acid standard solution. accurately weighed the appropriate amount of bisphosphorus standard, with a small amount of benzene dissolved, and then with n-hexane prepared into the concentration of 2.0 mg/mL standard stock solution, stored in the refrigerator. If necessary, dilute with n-hexane to the standard working solution at the appropriate concentration.

4.5 Materials

4.5.1

Florisil Column. 4 g.

5 Instruments and equipment

5.1

Gas Chromatography-Mass Spectrometer, with electron bombardment ion source (EI source).

5.2

Analysis of balance. 0.01 g and 0.0001 g.

5.3

Centrifuge.

5.4

Crusher.

5.5

Rotary evaporator.

5.6

Microinjector. 10 μ L.

5.7

Oscillator.

6.1 Preparation of the sample

To replace the sample of about 1000 g, crushed by grinding, mixing, are divided into two, respectively, into the clean container as a sample, sealed, and mark the mark.

6.2 Sample storage

The sample was stored at -18 ° C or lower. During the operation of the sample preparation, it is necessary to prevent contamination of the sample or change in the content of the residue.

7.1 Extraction

Weigh about 10 g of sample (accurate to 0.1 g) in a conical flask and add 50 mL of acetonitrile-methanol mixture (4:1, v/v) for 30 min.

Centrifuge at 4000 rpm for 15 min, remove the supernatant in a separatory funnel. And then three times with 3 x 25 mL methanol washing the sample three times, wash with the former the supernatant was combined in the same separatory funnel.