



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

GB 23200.98-2016

Replacing SN/T 2577-2010

**Food safety national standard -- Determination of Eleven
Organophosphorus Pesticide Residues in Royal Jelly by Gas
Chromatography**

蜂王浆中11种有机磷农药残留量的测定 气相色谱法

(English Translation)

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Foreword

This standard replaces SN/T 2577-2010 "Import and export royal jelly in 11 kinds of organophosphorus pesticide residues determination of gas chromatography." Compared with SN/T 2577-2010, the main changes are as follows.

- Standard text format is modified to national standard text format for food safety;
- standard name "import and export royal jelly" to "royal jelly";
- increase the "other food reference implementation" in the standard range.

This standard replaced the previous version of the standard release.

-SN/T 2577-2010.

National standards for food safety Determination of Eleven Organophosphorus Pesticide Residues in Royal Jelly by Gas Chromatography

1 Scope

This standard specifies the preparation of 11 kinds of organophosphorus pesticide residues in royal jelly and the determination of sample preparation and gas chromatography.

This standard applies to royal jelly in the dichlorvos, methamidophos, broken phosphorus, phorate, dimethoate, methyl parathion, malathion, parathion, Quetiapine, triazophos, phosphorus poison phosphorus pesticide residues detection, 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 organophosphorus pesticide was extracted from the sample with acetonitrile. The extract was purified by gel column chromatography using a gas chromatograph equipped with a flame photometric detector Quantitative and 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

Ethyl acetate (C₄H₈O₂). Chromatographic purity.

4.1.2

acetonitrile (CH₃CN). pure chromatography.

4.1.3

Cyclohexane (C₆H₁₂). Chromatographic Purification.

4.1.4

Acetone (C₃H₆O). Chromatographic pure.

4.1.5 Sodium chloride (NaCl)

4.1.6 anhydrous sodium sulfate (Na₂SO₄). 650 °C burning 4h, in the dryer to cool to room temperature, stored in a sealed bottle in reserve.

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 Evenly used. The

4.3 Standards

4.3.1

dichlorvos, methamidophos, methotrexate, phorate, dimethoate, methyl parathion, malathion, parathion, quinophos, triazophos, Flies poison phosphorus standard. purity >= 99%, see Appendix A Table A.1.

4.4 Standard solution preparation

4.4.1 11 kinds of organic phosphorus standard stock solution. Weigh the appropriate standard, respectively, with acetone dissolved to 100 mL, the solution concentration 100 g/mL, 1 °C ~ 4 °C refrigerator. Valid for 3 months.

4.4.2 11 kinds of organic phosphorus mixed standard working solution. before use as necessary with acetone diluted to the appropriate concentration, as a mixed standard working fluid.

1 °C ~ 4 °C refrigerator to save. Valid for 3 months.

4.4.3

Standard working solution concentration. 0.008 g/mL, 0.01 g/mL, 0.05 g/mL, 0.1 g/mL, 0.5 g/mL, Stored in the refrigerator at 4 °C.

4.5 Materials

4.5.1

Gel Purification Product Line (GPC). 400 mm x 15 mm (inner diameter); Filler. Bio-Beads, S-X3, 38 m-75 m Before the need to use the standard solution to do the first leaching

curve).

4.5.2

anhydrous sodium sulfate column. 80 mm x 40 mm (diameter) cylindrical funnel, the bottom pad 5 mm absorbent cotton, then install 50 mm anhydrous sodium sulfate.

5 Instruments and equipment

5.1

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

5.2

Analysis of balance. 0.0001 g and 0.01 g.

5.3 Rotary Evaporator

5.4 vortex mixer.

5.5 Centrifuge $\geq 6\,000$ r/min.

6 Preparation and storage of samples

Take 500 g representative royal jelly samples, the sampling site according to GB 2763 Appendix A implementation, thawed at room temperature, and other samples after all melted Stir the sample, the samples are divided into two copies, respectively, into the vial, sealed, and marked. One as a test sample and one at $-18\text{ }^{\circ}\text{C}$ save.

During the operation of the sample preparation, the contamination of the sample or the change in the content of the residue should be prevented.

7.1 Extraction

Weigh 5 g sample (accurate to 0.01 g) in a 50 mL plastic centrifuge tube, add 15 mL of water, mix for 5 min, add B Nitrile to 50 mL on a vortex mixer at 2 000 r/min, mix for 1 min, centrifuge at 6 000 r/min for 5 min, remove the supernatant the solution was placed in another 50 mL plastic centrifuge tube and 4 g of sodium chloride was added to the vortex mixer at 2 000 r/min for 1 min, Centrifuge at 6 000 r/min for 5 min. The upper layer of acetonitrile was passed through an anhydrous sodium sulphate column, collected in a concentrated flask, and then 5 mL of water and 20 ML of acetonitrile to the residue, repeat the above operation, combined acetonitrile extract, $40\text{ }^{\circ}\text{C}$ in the following water bath concentrated to near dry, to be purified.