



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

GB 23200.101-2016

Replacing SN/T 2571-2010

**Food safety national standard -- Determination of residues of
acaricides in royal jelly by gas chromatography -- Mass
spectrometry**

蜂王浆中多种杀螨剂残留量的测定 气相色谱-质谱法

(English Translation)

Issued on: December 18, 2016

Implemented on: June 18, 2017

**Issued by: National Health and Family Planning Commission of the
People 's Republic of China**

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Foreword

This standard replaces SN/T 2571-2010 "Import and export royal jelly in a variety of acaricide residues detection method of gas chromatography - mass spectrometry." Compared with SN/T 2571-2010, 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 2571-2010.

National standards for food safety Determination of residues of acaricides in royal jelly by gas chromatography - mass spectrometry

1 Scope

This standard provides the royal jelly in the mite ether, methyl grams of mites, acaricides, lei mites, ethyl keto mite, brom mite, dicofol, Determination and Confirmation of Residue of Pyridenopsis by Gas Chromatography - Mass Spectrometry.

This standard applies to royal jelly in the acaricidal ether, methyl grams of mites, acaricides, lei acarics, ethyl methicillin, bromide mites, dicofol, Determination and confirmation of residues of pyridaben residues, 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 residue of the acaricide in the sample was extracted with a mixed solvent of n-hexane acetone (1 1, v/v), purged with a Florisil column, and the gas phase Spectral - mass spectrometry - negative chemical source determination, 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

ether (C₄H₁₀O). pure chromatography.

4.1.2

n-hexane (C₆H₁₄). pure chromatography.

4.1.3

Acetone (C₃H₆O). Chromatographic pure.

4.1.4

Sodium chloride (NaCl).

4.1.5

anhydrous sodium sulfate (Na_2SO_4). 650 °C burning 4h, in the dryer to cool to room temperature, stored in a sealed bottle in reserve.

4.2 Standards

4.2.1

acaricidal mite, methyl acaricides,, deer mites, ethyl methicillin, bromocarbide, dicofol, Purity $\geq 99\%$, see Appendix A.

4.3 Standard solution preparation

4.3.1

acaricides, methyl methicillin, acaricides, lemi mite, ethyl methicillin, bromide mite, dicofol, Solution. Weigh the appropriate amount of standard material accurately, with n-hexane dubbed the concentration of 100 $\mu\text{L}/\text{mL}$ standard stock solution. Store in 4 °C refrigerator.

4.3.2

acaricides, methicillin mites, acaricides, lei mites, ethyl methicillin, bromocarbide, dicofolidone, pyridaben standard work Liquid. Dilute the mixture with n-hexane to the appropriate concentration of the standard working solution. Store in 4 °C refrigerator.

4.4 Materials

4.4.1

Florisil Solid Phase Extraction Column. (1 g, 6 mL) or equivalent. Use a 10 mm high anhydrous sodium sulphate layer in the front column with 5 mL N - hexane elution activated solid phase extraction.

5 Instruments and equipment

5.1

Gas Chromatography-Mass Spectrometer. Negative Chemical Source (NCI).

5.2

Analysis of balance. 0.01 g and 0.0001 g.

5.3

vortex mixer.

5.4

Centrifuge. speed greater than 5 000 r/min.

5.5

nitrogen blowing instrument.

5.6

Rotary evaporator.

6 Preparation and storage of samples

Take about 500g of the sample, the sampling site according to GB 2763 Appendix A implementation, will be forced to stir evenly, into a clean container, Sealed and marked with the mark, and the sample was stored at -18 ° C.

During sample and sample preparation, contamination of the sample or changes in the residue content should be prevented.

7.1 Extraction

Weigh 2 g sample (accurate to 0.01 g) in 50 mL centrifuge tube, add 10 mL water vortex mixed 1 min, standing 5 min. Join 20 mL of n-hexane acetone (1 1, volume ratio) mixed solvent, vortex mixed for 1 min, centrifuged at 4000 r/min for 3 min, the upper organic Phase transferred into the concentration bottle. Add 10 mL of n-hexane acetone (1 1, volume ratio) mixed solvent, repeat the extraction time, combined the upper organic phase, in 45 °C below the water bath under reduced pressure to near dry, to be purified.

7.2 Purification

The residue in the concentrated flask was washed twice with 3 mL of n-hexane and transferred to a Florisil solid phase extraction column. With 5 mL of n-hexane the effluent was discarded and eluted with 10 mL of n-hexane + ether (8 + 2, v/v) mixed solvent. The eluate was collected in 10 mL of glass off Tube in the 45 °C water bath with nitrogen blowing instrument slowly blowing to near dry, with n-hexane dissolved and set to 0.5mL, gas chromatography-mass spectrometry.

7.3.1 Gas Chromatography - Mass Spectrometry Reference Conditions

- A) Column. DB-5ms quartz capillary column, 30 m x 0.25 mm (id) x 0.25 µm, or equivalent;
- B) column temperature. 50 °C for 1 min, at 20 °C/min heating rate rose to 220 °C, keep 1 min, the temperature was raised to 250 ° C at a rate of 5 ° C/min and raised to 280 ° C at a rate of 20 ° C/min for 8 min.
- C) Inlet temperature. 250 ° C;