



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

GB 23200.99-2016

Replacing SN/T 2572-2010

**Food safety national standard -- Determination of various
carbamate pesticide residues in royal jelly by liquid
chromatography-mass spectrometry / mass spectrometry**

蜂王浆中多种氨基甲酸酯类农药残留量的测定 液相色谱-质谱质谱法

(English Translation)

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Foreword

This standard replaces SN/T 2572-2010 "Import and export royal jelly in a variety of carbamate pesticide residues detection method Liquid chromatography -Mass spectrometry/mass spectrometry".

Compared with SN/T 2572-2010, 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 2572-2010.

National standards for food safety Determination of various carbamate pesticide residues in royal jelly Liquid chromatography - mass spectrometry/mass spectrometry

1 Scope

This standard stipulates the royal jelly in the methyl thiouwei, evil worm Wei, isoprocab, carbamate, methomyl, carbofuran, pirimicarb, Zhong Dingwei residual Amount of liquid chromatography-mass spectrometry/mass spectrometry.

This standard applies to royal jelly in the methyl thiouwei, evil worm Wei, isoprene, carbazate, methomyl, carbofuran, pirimicarb, Zhong Dingwei residual the amount of the determination and confirmation, 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 samples were extracted with acetonitrile, purified by neutral alumina column chromatography, and analyzed by liquid chromatography-mass spectrometry/mass spectrometry.

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

Methanol (CH₄O). Chromatographic purity.

4.1.2

acetonitrile (C₂H₃N). pure chromatography.

4.1.3

Acetone (C₃H₆O). Chromatographic pure.

4.1.4

ether (C₄H₁₀O). chromatographic purity.

4.1.5

ammonium acetate (CH₃COONH₄). pure chromatography.

4.1.6

Sodium chloride (NaCl).

4.2 Solution preparation

4.2.1 0.005 mol/L ammonium acetate solution. Weigh 0.19 g ammonium acetate dissolved in the amount of water, and then set to 500 mL.

4.2.2

methanol - 0.005 mol/L ammonium acetate solution (6 + 4, V/V). take 600 mL of methanol, add 400 mL 0.005 mol/L B Ammonium acid solution, shake back spare.

4.2.3

acetone - ether solution (2 8, V/V). take 200 mL of acetone, add 800 mL of ether, shake back.

4.3 Standards

4.3.1

carbamate standard products. methyl thiourea, evil worm Wei, isoprove, carbaryl, carbofuran, pirimicarb, Zhong Dingwei purity $\geq 99.0\%$.

For more than 98.8%, see Appendix A.

4.4 Standard solution preparation

4.4.1

urethane standard stock solution. accurately weighed the amount of carbamate standard, dissolved in methanol, were prepared into a concentration of 200 g/mL standard stock solution, 4 °C refrigerator storage.

4.4.2

carbamate mixed working solution. according to need, respectively, take appropriate amount of liquid in the same volume bottle, with methanol - 0.005 mol/L Ammonium acetate solution was diluted to the appropriate concentration of working solution. Store in 4 °C

refrigerator.

4.4.3

Preparation of Carbamate Calibration Solution. The standard mixed solution was diluted with a blank sample solution treated with 7.1 and 7.2 to a concentration of 5.0, 10.0, 50.0, 100.0 g/L. Store in 4 °C refrigerator.

4.5 Materials

4.5.1

Neutral Alumina Solid Phase Extraction Column (500 mg, 3 mL). Rinse with 5 mL of ether before use.

5 Instruments and equipment

5.1

LC-Tandem Mass Spectrometer. Equipped with electrospray ion source and quadrupole mass analyzer.

5.2

Analysis of balance. 0.01 g and 0.0001 g.

5.3

Vortex Mixer.

5.4

Rotary Evaporator.

5.5

Centrifuge. speed greater than 5 000 r/min.

5.6

Solid phase extraction unit with vacuum pump.

5.7

covered plastic centrifuge tube. 50 mL.

6 Preparation and storage of samples

To replace the sample of about 500g, the sampling site according to GB 2763 Appendix A implementation, will be forced to stir evenly, into a clean container sealed, and mark the mark. Save below -18 ° C. During the operation of the sample preparation, the contamination of the sample or the change in the content of the residue should be prevented.