



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

GB 23200.103-2016

Replacing SN/T 2574-2010

**Food safety national standard -- Determination of Residual
Amidamidine and Its Metabolites in Royal Jelly by Gas
Chromatography -- Mass Spectrometry**

蜂王浆中双甲脒及其代谢产物残留量的测定 气相色谱-质谱法

(English Translation)

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Foreword

This standard replaces SN/T 2574-2010 "Import and export royal jelly in the double amidine and its metabolites Residue detection method Gas chromatography -Mass spectrometry".

Compared with SN/T 2574-2010, 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 2574-2010.

National standards for food safety Determination of Residual Amidamidine and Its Metabolites in Royal Jelly by Gas Chromatography - Mass Spectrometry

1 Scope

This standard specifies the determination and confirmation of the method for the determination of amitraz and its metabolites in royal jelly by gas chromatography-mass spectrometry.

This standard applies to royal jelly in the double amidine and its metabolites residue 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 acid-hydrolyzed and alkalized with n-hexane-diethyl ether (2 1, volume ratio) mixed solvent extraction, acid, lye-liquid distribution purification, gas phase Chromatography - mass spectrometry and confirmation, 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

n-hexane (C₆H₁₄). chromatographic purity.

4.1.2

ether (C₄H₁₀O). pure chromatography.

4.1.3

Acetone (C₃H₆O). Chromatographic pure.

4.1.4

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

4.2 Solution preparation

4.2.1

hydrochloric acid solution (2.0 mol/L). with a pipette to remove 90 mL of hydrochloric acid, diluted with water to 500mL.

4.2.2

sodium hydroxide solution (10 mol/L). Weigh 40 g of sodium hydroxide, dissolved in water to 100mL.

4.3 Standards

4.3.1

Amitraz (amitraz, C₁₉H₂₃N₃, CAS No. 33089-61-1, molecular weight 293) Standard substance. purity > 99%.

4.3.2 2,4-Dimethyl aniline (C₈H₁₁N, CAS No. 95-68-1, molecular weight. 121) Reference substance.

Purity > 99%.

4.4 Standard solution preparation

4.4.1

Amitrazine standard solution. accurately weighed the appropriate amount of formamidine standard material, with acetone dubbed the concentration of 100 g/mL standard stock solution.

According to the need to use acetone diluted to the appropriate concentration of standard working fluid. Store in 4 °C refrigerator.

4.4.2 2,4-dimethylaniline standard solution. accurately weighed the appropriate amount of 2,4-dimethyl aniline standard material, with acetone dubbed the concentration of 100 g/mL standard stock solution. According to the need to use acetone diluted to the appropriate concentration of standard working fluid. Store in 4 °C refrigerator.

5 Instruments and equipment

5.1

Gas Chromatography-Mass Spectrometer. Equipped with an electron impact source (EI).

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

sets of thermostat. 50 °C ~.200 °C.

5.6

full glass reflow device.

6 Preparation and storage of samples

Approximately 500 g of the representative sample was added, stirred vigorously, placed in a clean container, sealed, and marked with the sample at -18C Frozen.

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

Note: The above sample sampling site according to GB 2763 Appendix A implementation.

7.1 Hydrolysis

Weigh 4 g of sample (accurate to 0.01 g) in a 100 mL flask and add 15 mL of hydrochloric acid solution (4.5) to mix on a vortex mixer.

Install the reflux condenser, heat reflux at 120 °C for 2 h, stop heating, continue to cool water at room temperature for 15min, move to the ice bath to cool 15min.

7.2 Extraction and purification

Transfer the hydrolyzate to a 50 mL stoppered centrifuge tube with 10 mL of n-hexane-diethyl ether (2 1, by volume) mixed solvent plus 4 mL of sodium hydroxide solution, vortex and mix well, cooled in ice bath for 5 min, centrifuged at 4000 r/min for 3 min. Transfer the upper organic phase to another A 50 mL stopper with a centrifuge tube. The aqueous phase was extracted twice with a mixed solvent of 2 x 10 mL n-hexane-diethyl ether (2 1, v/v). Merge the upper layer the organic phase. With 2 x 5 mL hydrochloric acid solution extraction 2 times, combined with hydrochloric acid extract and constant volume to 10mL, take 5mL hydrochloric acid extract in 10 mL with a plug in the centrifuge tube. Add 2 mL of n-hexane - acetone (2 1, volume ratio) mixed solvent, 2 mL of sodium hydroxide solution extraction 1, centrifugal Layer, upper organic phase for gas chromatography - mass