



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

GB 23200.102-2016

Replacing SN/T 2573-2010

**Food safety national standard -- Determination of the content of
chlordimeform and its metabolites in royal jelly by gas
chromatography-mass spectrometry**

蜂王浆中杀虫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 2573-2010 "Import and export royal jelly in the detection of chlordimeform and its metabolites Residue Determination of gas chromatography -Mass spectrometry".

Compared with SN/T 2573-2010, 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 2573-2010.

National standards for food safety Determination of the content of chlordimeform and its metabolites in royal jelly by gas chromatography - mass spectrometry

1 Scope

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

This standard applies to the determination and confirmation of the residual amount of chlordimeform and its metabolites in royal jelly, 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 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 precipitated with trichloroacetic acid solution and extracted under basic conditions with n-hexane-acetone (1 1, v/v) mixed solvent.

The liquid was purified by gas chromatography - mass spectrometry and confirmed by external hexane - acetonitrile.

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). Chromatographic pure.

4.1.2

Acetone (C₃H₆O). Chromatographic pure.

4.1.3

n-hexane (C₆H₁₄). chromatographic purity.

4.1.4

Trichloroacetic acid (CCl₃COOH).

4.1.5

Sodium hydroxide (NaOH).

4.1.6

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

n-Hexane - Acetone (1:1, volume ratio) mixed solvent. take 100 mL of n-hexane, add 100 mL of acetone, shake back.

4.2.2

Trichloroacetic acid solution (5%). Weigh 5 g of trichloroacetic acid, add water and set to 100 mL.

4.2.3

Sodium hydroxide solution (2 mol/L). Weigh 8 g of sodium hydroxide into 100 mL of distilled water.

4.3 Standards

4.3.1

Chlordimeform, C₁₀H₁₃ClN₂, CAS No. 6164-98-3, molecular weight 196.68) Reference substance. pure Degree > 98.5%.

4.3.2 4-Chloro-2-methylaniline, C₇H₈ClN, CAS No. 95-69-2, molecular weight. 141.60) Standard substance. purity > 98.5%.

4.4 Standard solution preparation

4.4.1

Accurately weigh the appropriate amount of chlordimeform and 4-chloro-o-methylaniline standard substance, with a single standard storage solution with a concentration of 100 g/mL Preparation of liquid. According to the need to use acetone diluted mixed to the appropriate concentration of mixed 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 10000 r/min.

5.5

Rotary evaporator.

6 Preparation and storage of samples

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

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

7.1 Extraction

Weigh 2 g of sample (accurate to 0.01 g) in 50 mL centrifuge tube, add 10 mL of trichloroacetic acid solution vortex mixed for 1 min to 8 000 r/min Centrifuge 3 min. The upper aqueous phase was transferred to another 50 mL centrifuge tube, plus about 4 mL of sodium hydroxide solution adjusted to pH greater than 12. Add 5 g Sodium chloride and 20 mL of n-hexane-acetone (1 1, volume ratio) were mixed and vortexed for 1 min to transfer the upper organic phase into a concentrated flask.

The addition of 20 mL of n-hexane-acetone (1 1, volume ratio) in the aqueous phase was repeated and the extraction was repeated once. The upper organic phase was combined at 45 ° C the bath is concentrated under reduced pressure to near dryness, to be purified.

7.2 Purification

The residue in the concentrated flask was washed twice with 10 mL of acetonitrile and transferred to a 50 mL centrifuge tube. 10 ml of n-hexane was added and the vortex was mixed Min, centrifuged at 3 000 r/min for 3 min, the upper n-hexane phase was transferred