



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

GB 23200.96-2016

Replacing SN/T 0213.1-2011

**Food safety national standard -- Determination of the content of
chlordimeform and its metabolites in honey by liquid
chromatography-mass spectrometry / mass spectrometry**

蜂蜜中杀虫脒及其代谢产物残留量的测定 液相色谱-质谱 质谱法

(English Translation)

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Foreword

This standard replaces SN/T 0213.1-2011 "Determination of the content of chlordimeform and its metabolites in the export of honey by liquid chromatography-mass spectrometry Spectrum method".

Compared with SN/T 0213.1-2011, the main changes are as follows.

- Standard text format is modified to national standard text format for food safety;
- the standard name "export honey" to "honey";

- increase the "other food reference implementation" in the standard range.

This standard replaced the previous version of the standard release.

-SN/T 0213.3-1993

-SN/T 0213.1-2011.

National standards for food safety Determination of the content of chlordimeform and its metabolites in honey by liquid chromatography-mass spectrometry/mass spectrometry

1 Scope

This standard specifies a method for the determination of chlordimeform and its metabolite (4-chloro-o-toluidine) in honey by liquid chromatography-mass spectrometry/mass spectrometry.

This standard applies to honey (acacia honey, Vitex honey, honey honey, miscellaneous honey, wild honey, etc.) in the chlordimeform and its metabolite (4-chloro Toluidine) residue in liquid chromatography-mass spectrometry/mass spectrometry and other food samples.

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 sample was diluted with aqueous sodium hydroxide solution, purified by HLB solid phase extraction column, liquid chromatography-tandem mass spectrometer, external standard the amount.

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 (CH₃CN), high performance liquid chromatography pure.

4.1.2

methanol (CH₃OH), high performance liquid chromatography pure.

4.1.3

Sodium hydroxide (NaOH).

4.1.4

sulfuric acid (H₂SO₄).

4.1.5

Formic acid (HCOOH), purity $\geq 99\%$.

4.2 Solution preparation

4.2.1 0.02 mol/L sodium hydroxide solution. dissolved 800 mg sodium hydroxide in 1 L water, the use of a period of 1 month.

4.2.2 1 mol/L sulfuric acid solution. in the amount of water slowly into the 54.3 mL concentrated sulfuric acid, while stirring and stirring, diluted with water to 1 L, so that with a period of 3 months.

4.2.3 10 mmol/L sulfuric acid solution. in the amount of water slowly into 10 mL 1 mol/L sulfuric acid, while stirring and stirring, diluted with water to 1 L, Use for 1 month.

4.2.4

acetonitrile aqueous solution. acetonitrile. water (3 7, V/V).

4.2.5 0.1% formic acid solution. 1 mL formic acid dissolved in water and fixed to 1 L.

4.3 Standards

4.3.1

Pesticide standard (chlordimeform, C₁₀H₁₃ClN₂, CAS. 6164-98-3. purity $\geq 99\%$) and 4-chloro-o-toluidine standard (4-chloro-o-toluidine, C₇H₈ClN, CAS. 95-69-2. purity $\geq 99\%$).

4.4 Standard solution preparation

4.4.1

Preparation of standard stock solution. accurately weighed the appropriate amount of chlordimeform and 4-chloro o-toluidine standard, with acetonitrile prepared into a concentration of 1.0 Mg/mL standard stock solution. The solution was stored in a refrigerator at -18 ° C. Valid for 12 months.

4.4.2

Preparation of the standard intermediate solution. diluted the standard stock solution with acetonitrile to a final concentration of about 1.0 µg/mL, below 4 ° C.

Save, valid for 6 months.

4.4.3

matrix standard working solution preparation. according to need, when used to absorb a certain amount of standard intermediate solution, with the matrix blank solution with Mix the standard working solution with the appropriate concentration. Less than 4 °C dark storage, is now available with.

4.5 Materials

4.5.1

HLB solid phase extraction column (hydrophilic lipophilic balance column). 60 mg (filler. polystyrene-divinylbenzene-pyrrolidone), 3 mL or equivalent. Activate with 3 mL of methanol and 3 mL of water before use.

4.5.2

Microporous membrane. 0.22 µm, organic system.

5 Instruments and equipment

5.1

Liquid Chromatography - Tandem Mass Spectrometer. Equipped with electrospray ion source (ESI).

5.2

Electronic balance. 0.01g and 0.0001g respectively.

5.3

vortex mixer.

5.4

solid phase extraction device.

5.5

nitrogen blowing instrument.

5.6

Centrifuge tube. 15 mL.