



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

GB 23200.74-2016

Replacing SN/T 2387-2009

**Food safety national standard -- Determination of
Jinggangmycin Residue in Food by Liquid Chromatography --
Mass Spectrometry / Mass Spectrometry**

食品中井冈霉素残留量的测定 液相色谱-质谱质谱法

(English Translation)

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Table of Contents

Foreword	
1 Scope	
2 Normative reference documents	
3 Principle	
4 Reagents and materials	
4.1 Reagents	
4.2 Solution preparation	
4.3 Standards	
4.4 Standard solution preparation	
4.5 Materials	
5 Instruments and equipment	
6 Sample preparation and storage	
6.1 Sample requirements	
6.2 Tea and cereals	
6.3 Vegetables and fruits	
6.4 Nuts	
6.5 Animal muscle, liver, fish, shrimp	
7 Analysis steps	
7.1 Extraction	
7.2 Purification	
7.3 Determination	
7.4 Blank test	
8 Results are calculated and expressed	
9 Precision	
10.1 Quantitation limits	
10.2 Recovery rate	

Foreword

This standard replaces SN/T 2387-2009 "Determination of Jinglyngmycin Residue in Import and Export Food by Liquid Chromatography-Mass Spectrometry/Mass Spectrometry".

Compared with SN/T 2387-2009, the main changes are as follows.

- Standard text format is modified to national standard text format for food safety;
- the name of the "import and export food" to "food";
- "liquid chromatography - mass spectrometry/mass spectrometry" in the standard name to "liquid chromatography - tandem mass spectrometry";
- increase the "other food reference implementation" in the standard range.

This standard replaced the previous version of the standard release.

-SN/T 2387-2009.

National standards for food safety Determination of Jinglyngmycin Residue in Food by Liquid Chromatography - Mass Spectrometry/Mass Spectrometry

1 Scope

This standard specifies the method for the determination of Jinglyngmycin residues in food by liquid chromatography-mass spectrometry/mass spectrometry.

This standard applies to rice, cabbage, onions, carrots, tomatoes, cucumbers, spinach, fungus, pear, lemon, almonds, tea, pork, Pig liver, tilapia, shrimp in the detection and confirmation of Jinglyngmycin 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 document.

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 of Jinglyngmycin residues in methanol aqueous solution extraction, by HLB solid phase extraction column or ethyl acetate liquid extraction and purification, with liquid chromatography -Mass spectrometry/mass spectrometer detection and confirmation, external standard method quantitative.

4 Reagents and materials

Unless otherwise specified, the reagents used in this method are pure liquid chromatography, water in line with GB/T 6682 in the provisions of a water.

4.1 Reagents

4.1.1

Ammonium acetate ($\text{CH}_3\text{COONH}_4$, CAS number. 631-61-8).

4.1.2

Methanol (CH_3OH , CAS No. 67-56-1).

4.1.3

Acetonitrile (CH_3CN , CAS No. 75-05-8).

4.1.4

Ethyl acetate ($\text{CH}_3\text{COOCH}_2\text{CH}_3$, CAS number. 141-78-6).

4.1.5

glacial acetic acid (CH_3COOH , CAS number. 64-19-7).

4.2 Solution preparation

4.2.1

methanol solution (9 1). the amount of 90 mL of methanol by adding 10 mL of water, mix.

4.2.2 5 mmol/L ammonium acetate buffer solution. Weigh 0.385 g of ammonium acetate, dissolved in water and fixed to 1 000 mL, adjusted with glacial acetic acid PH to 4.5 ± 0.1 .

4.3 Standards

4.3.1 Jinglyngmycin standard (Validamycin A, molecular formula. $\text{C}_{20}\text{H}_{35}\text{NO}_{13}$, CAS. 37248-47-8). purity $\geq 91.0\%$.

4.4 Standard solution preparation

4.4.1

Jinglyngmycin standard stock solution. Weigh 2 mg (accurate to 0.1 mg) the standard of Jinglyngmycin, dissolved in water and set to 25 mL of a brown volumetric flask to prepare a standard stock solution at a concentration of 80 mg/L.

4.4.2

Jinglyngmycin matrix standard working solution. according to the need, with blank samples in accordance with the sample treatment steps to get the extract, preparation is not with the

concentration of the matrix standard solution, is now available.

4.5 Materials

4.5.1

HLB column. 6 mL / .200 mg or equivalent; activated with 6 mL of methanol and 6 mL of water before use.

4.5.2

Filtration. 0.22 m, biphasic.

5 Instruments and equipment

5.1

Liquid Chromatography - Mass Spectrometry/Mass Spectrometer. Atmospheric Pressure Chemical Ionization Source (APCI).

5.2

Analysis of balance. 0.01 g and 0.0001 g.

5.3

vortex mixer.

5.4

Homogenizer.

5.5

Centrifuge. speed greater than 3 000 r/min.

5.6

nitrogen blowing concentrator.

5.7

Centrifuge tube. 50 mL Teflon centrifuge tube and 50 mL stopper glass centrifuge tube.

5.8

scale tube. 30 mL, the smallest scale of 0.1 mL.

5.9

pH meter. the amount of 0.1.

6.1 Sample requirements

During sample preparation, the sample should be protected from contamination or changes in residue content.