



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

GB 23200.29-2016

Replacing SN/T 1977-2007

**Food safety national standard -- Determination of residual
residues of oxazabs in fruits and vegetables by liquid
chromatography**

水果和蔬菜中唑螨酯残留量的测定 液相色谱法

(English Translation)

Issued on: December 18, 2016

Implemented on: June 18, 2017

**Issued by: National Health and Family Planning Commission of the
People 's Republic of China**

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Foreword

This standard replaces SN/T 1977-2007 "High Performance Liquid Chromatography for the Detection of the Residual Potatoes in Fruit and Vegetables." Compared with SN/T 1977-2007, the main changes are as follows.

- Standard text format is modified to national standard text format for food safety;
- the name "export fruit and vegetables" to "fruit and vegetables";
- increase the "other food reference implementation" in the standard range.

This standard replaced the previous version of the standard release.

-SN/T 1977-2007.

National standards for food safety Determination of residual residues of oxazabs in fruits and vegetables by liquid chromatography

1 Scope

This standard specifies the method of high performance liquid chromatography for the determination of the residues of oxazolid in fruits and vegetables.

This standard applies to the citrus, cabbage, the determination of the residual amount of squalidomide; 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 methanol, n-hexane stripping, neutral alumina column, and detected by high performance liquid chromatography with UV detector.

Quantitative 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

Methanol (CH₃OH). Chromatographic Purification.

4.1.2

n-hexane (C₆H₁₄). pure chromatography.

4.1.3

Ethyl acetate (C₄H₈O₂). Chromatographic purity.

4.1.4 Anhydrous sodium sulfate (Na₂SO₄). 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

sodium chloride solution (10%). take 10 g of sodium chloride, the volume of water to 100 mL, shake back.

4.3 Standards

4.3.1

Streptomide standard (Fenpyroximate, CAS.NO .. 111812-58-9). purity >=99%.

4.4 Standard solution preparation

4.4.1

Streptomide standard stock solution. accurately weighed appropriate amount of azole mite ester standard, with acetonitrile prepared into a concentration of 100 µg/mL standard reserve liquid. According to the need to use acetonitrile diluted to the appropriate concentration of standard working fluid.

4.5 Materials

4.5.1

Neutral alumina solid phase extraction column. 1 000 mg, 3 mL.

5 Instruments and equipment

5.1

High Performance Liquid Chromatograph with UV Detector.

5.2

Analysis of balance. 0.01 g and 0.0001 g.

5.3

Homogenizer. 5000 r/min.

5.4

Centrifuge. 4000 r/min.

5.5

Rotary Evaporator.

5.6

nitrogen blowing instrument.

5.7

Scroll Mixer.

6.1 Preparation of the sample

To replace the sample of about 500 g, the sampling site according to GB 2763 Appendix A, the individual large samples need to be chopped, into the high-speed organization the crusher is mashed evenly, packed in a clean container, sealed and marked with a mark.

6.2 Sample storage

Samples were stored at 0 - 4 ° C. During the operation of the sample preparation, it is necessary to prevent contamination of the sample or change in the content of the residue.

7.1 Extraction

Weigh 15 g (accurate to 0.01 g) sample placed in 50 mL centrifuge tube, add 20 mL of methanol, high-speed homogenization 2 min. 4000 r/min Centrifuge 4 min, filter, add 20 mL of methanol residue, vortex 1 min, repeat centrifugal filtration operation, combined filtrate, to be purified.

7.2 Purification

The filtrate was transferred to a 250 mL separatory funnel, 40 mL of sodium chloride solution and 20 mL of n-hexane were added, vigorously shaken for 2 min, the layers were placed in a 100 mL stoppered flask. The aqueous layer was repeatedly extracted with 20 mL of n-hexane and the n-hexane layer was combined.

Add 2 g of anhydrous sodium sulfate, shake, after standing, the n-hexane layer into the 100 mL concentrated bottle, and then a small amount of n-hexane washing with a plug cone the bottles are washed twice and the washings are combined to the vials. In 40 ° C water bath rotation concentrated to near dry.

The flask was washed with 3 x 3 mL of n-hexane-ethyl acetate (28) and the solution was transferred to 5 mL of ethyl acetate pre-leached neutral oxygen in the aluminum solid phase extraction column, the flow rate was controlled by about 2 mL/min, and the whole effluent was collected. Dried in a nitrogen bath at 40 ° C and dissolved in the mobile phase Constant volume to 1.0 mL, over 0.45 µm filter for liquid chromatography analysis.