



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

GB 23200.21-2016

Replacing SN/T 0350-2012

**Food safety national standard -- Determination of gibberellic
acid residues in fruits by liquid chromatography-mass
spectrometry / mass spectrometry**

水果中赤霉素残留量的测定 液相色谱-质谱 质谱法

(English Translation)

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Foreword

This standard replaces SN/T 0350-2012 "Determination of Gibberellin Residue in Import and Export Fruits by Liquid Chromatography-Mass Spectrometry/Mass Spectrometry".

Compared with SN/T 0350-2012, 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 fruit" to "fruit".
- increase the "other food reference implementation" in the standard range.

This standard replaced the previous version of the standard release.

-SN/T 0350-2012.

National standards for food safety Determination of gibberellic acid residues in fruits by liquid chromatography - mass spectrometry/mass spectrometry

1 Scope

This standard specifies the preparation of gibberellic acid residues in fruit and the method of liquid chromatography-mass spectrometry/mass spectrometry.

This standard applies to the import and export of apples, oranges, peaches, pears and grapes in the detection of gibberellic acid 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 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 residual gibberellic acid was extracted with acetonitrile, and the extract was purified by liquid-liquid distribution and then determined by liquid chromatography-mass spectrometry/mass spectrometry.

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

Acetonitrile (C₂H₃N). Chromatographic pure.

4.1.2

Methanol (CH₄O). Chromatographic purity.

4.1.3

Ethyl acetate (C₄H₈O₂). Chromatographic purity.

4.1.4

Formic acid (CH₂O₂). Chromatography grade.

4.1.5

Potassium dihydrogen phosphate (K₂HPO₄).

4.1.6

Sodium hydroxide (NaOH).

4.1.7

Sulfuric acid (H₂SO₄).

4.1.8

Sodium chloride (NaCl).

4.2 Solution preparation

4.2.1

sulfuric acid aqueous solution (pH 2.5). 1 drop of sulfuric acid into 100 mL of water, adjust the water pH of 2.5.

4.2.2

Phosphate Buffer Solution (pH 7). 6.7 g of potassium dihydrogen phosphate and 1.2 g of sodium hydroxide were dissolved in 1 L of water.

4.2.3 0.15% formic acid solution. Remove 0.15 mL of formic acid and dilute to 100 mL with water.

4.3 Standards

4.3.1

gibberellic acid standard (gibberellic acid, CAS NO. For 77-06-5, C₁₉H₂₂O₆). purity ≥ 98%.

4.4 Standard solution preparation

4.4.1

gibberellic acid standard stock solution. Weigh the appropriate amount of standard, dissolved in methanol, the solution concentration of 100 µg/mL. 0 °C ~ 4 °C refrigerated Light preservation. Valid for three months.

4.4.2

Standard working solution. Standard stock solution is diluted to 4, 5, 10, 100 and 150 ng/mL with a blank sample solution as required Standard working solution, equivalent to 8

μg/kg, 10 μg/kg, 20 μg/kg, 200 μg/kg, 300 μg/kg gibberellic acid in the sample. Pro Prepare before use.

4.5 Materials

4.5.1

Organic phase microporous membrane. 0.45 μm.

5 Instruments and equipment

5.1

Liquid Chromatography-Mass Spectrometry/Mass Spectrometer. Equipped with electrospray ion source.

5.2

Analysis of the balance. Analysis of balance. 0.01 g and 0.0001 g.

5.3

pH meter.

5.4

Rotary Evaporator.

5.5

vortex mixer.

5.6

Centrifuge. 4 000 r/min.

6 Preparation and storage of samples

A typical sample of about 500 g was taken from all the samples taken. The sampling site was carried out according to GB 2763 Appendix A, pulverized with a pulverizer, Mixed evenly, are divided into two copies, respectively, into the clean container as a sample, sealed, and marked mark. The sample was stored at -18 ° C.

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

7.1 Extraction

Weigh 5 g of sample (accurate to 0.01 g) in a 50 mL plastic centrifuge tube, add 25 mL of acetonitrile and 2 g of sodium chloride, vortex for 1 min, Centrifuged at 4 000 r/min for 5 min, the upper layer of acetonitrile extract transferred to the concentrated flask, the lower solution and then 20 mL of acetonitrile extraction time, and acetonitrile extract, in 45 °C