



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

GB 23200.73-2016

Replacing SN/T 3264-2012

**Food safety national standard -- Determination of rotenone and
azadirachtin residues in food by liquid chromatography-mass
spectrometry / mass spectrometry**

食品中鱼藤酮和印楝素残留量的测定 液相色谱-质谱 质谱法

(English Translation)

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Foreword

This standard replaces SN/T 3264-2012 "Detection methods of rotenone and azadirachtin residues in export food by liquid chromatography-mass spectrometry Spectrum method".

Compared with SN/T 3264-2012, the main changes are as follows.

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

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

This standard replaced the previous version of the standard release.

-SN/T 3264-2012.

National standards for food safety Determination of rotenone and azadirachtin residues in food by liquid chromatography - mass spectrometry/mass spectrometry

1 Scope

This standard specifies methods for the determination of rotenone and azadirachtin residues in food by liquid chromatography-mass spectrometry/mass spectrometry.

This standard applies to rice, cauliflower, apple, fungus, tea, honey, liver, fish, shrimp, chicken, milk rotenone and azadirachtin residues in the determination and confirmation, 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 rotenone and azadirachtin in the sample were extracted with acetonitrile, and the extract was salted out by sodium chloride and degreased with n-hexane. The polystyrene-Dimethylbenzene-pyrrolidone polymer filler solid phase extraction column purification, liquid chromatography - mass spectrometry/mass spectrometry detection and confirmation, external standard statutory 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). Chromatographically pure.

4.1.2

n-hexane (C₆H₁₀). pure chromatography.

4.1.3

Methanol (CH₃OH).

4.1.4

Formic acid (HCOOH). Chromatographic pure.

4.1.5

Sodium chloride (NaCl).

4.1.6

Ammonium acetate (CH₃COONH₄).

4.1.7

Sodium bicarbonate (NaHCO₃).

4.2 Solution preparation

4.2.1

Saturated sodium bicarbonate solution. Weigh a certain amount of sodium bicarbonate dissolved in water to saturation.

4.2.2 5mmol/L ammonium acetate buffer. Weigh 0.38 g of ammonium acetate dissolved in 800 mL of water, add 2 mL of formic acid, the water volume to 1000 ML.

4.3 Standards

4.3.1

Reference substance (rotenone. English name Rotenone, molecular formula C₂₃H₂₂O₆, CAS No. 83-79-4, molecular weight 394.42;

Azadirachtin. English name Azadirachtin, molecular formula C₃₅H₄₄O₁₆, CAS No.11141-17-6, molecular weight 720.71). purity ≥98%.

4.4 Standard solution preparation

4.4.1

rotenone and azadirachtin standard stock solution (100 mg/L). accurately weighed 0.0100 g of rotenone and azadirachtin standard substance, with a the alcohol was dissolved and set to 100 mL, and the standard stock was kept at 4 ° C for no more than one month.

4.4.2

rotenone and azadirachtin standard working solution. according to the need to take appropriate standard stock solution, 20% acetonitrile aqueous solution diluted to the appropriate concentration of the standard working fluid, temporary with the distribution.

4.5 Materials

4.5.1

Solid phase extraction column of polystyrene-divinylbenzene-pyrrolidone polymer filler. 60 mg, 3 mL. Use 3 mL before use Methanol, 3 mL water pretreatment.

4.5.2

Filtration. 0.22 μm , organic.

5 Instruments and equipment

5.1

Liquid Chromatography-Mass Spectrometry/Mass Spectrometer, Distribution Spray (ESI) Source.

5.2

Analysis of balance. 0.01 g and 0.0001 g.

5.3

Centrifuge. 4 500 r/min with 50 mL stoppered plastic centrifuge tube.

5.4

pulverizer.

5.5

Tissue crusher.

5.6

Scroll Mixer.

5.7

Ultrasonic Cleaner.

5.8

solid phase extraction device.

5.9

nitrogen blowing instrument.