



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

GB 23200.114-2018

**National food safety standard -- Determination of 208 pesticides
and their metabolite residues in plant-derived foods -- Gas
chromatography -- Mass spectrometry**

食品安全国家标准 植物源性食品中灭瘟素残留量的测定 液相色谱-质谱联用法

(English Translation)

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1 Scope

This standard specifies a liquid chromatography-mass spectrometry method for the determination of blasticidin-S residues in plant-derived foods:

This standard is applicable to the determination of blasticidin pesticide residues in plant-derived foods:

2 Normative reference documents

The following documents are essential for the application of this document: For dated references, only the dated version applies to this document:

For undated referenced documents, the latest version (including all amendments) applies to this document:

GB 2763 National Food Safety Standard Maximum Residue Limits of Pesticides in Foods
GB/T 6682 Analytical laboratory water specifications and test methods

3 Principles

The blasticidin in the sample was extracted with acetic acid aqueous solution, and after purification, it was detected by liquid chromatography-mass spectrometry and quantified by the external standard method:

4 Reagents and materials

Unless otherwise stated, only first-grade water confirmed as analytically pure reagents and in compliance with GB/T 6682 shall be used in the analysis:

4.1

Reagents

4.1.1

Acetic acid (CH_3COOH , CAS number

64-19-7); chromatographically pure:

4.1.2

Acetonitrile (CH_3CN , CAS number

75-05-8); chromatographically pure:

4.1.3

Formic acid (HCOOH , CAS number

64-18-6); chromatographically pure:

4.1.4

Ammonium formate (HCOONH_2 , CAS number

540-69-2):

4.1.5

Methanol (CH_3OH , CAS number

67-56-1); chromatographically pure:

4.2

Solution preparation

4.2.1

Acetonitrile solution (10 90, volume ratio)

Add 100 mL acetonitrile to 900 mL water and mix well:

4.2.2

Acetic acid-acetonitrile-water solution (2 10 88, volume ratio)

Add 2 mL acetic acid and 10 mL acetonitrile to 88 mL water and mix well:

4.2.3

Formic acid-methanol solution (10 90, volume ratio)

Add 100mL formic acid to 900mL methanol and mix well:

4:2:420mmol/L ammonium formate aqueous solution (pH=3:5): Weigh 0:385g ammonium acetate and dissolve it in an appropriate amount of water, and adjust the volume to 1000 mL, adjust pH to 3:5 with acetic acid:

4.3

Standard products Blasticide standard (C₁₇H₂sN₈O₈, CAS number

2079-00-7): purity $\geq 99.6\%$:

4.4

Preparation of standard solution

4.4.1

Standard stock solution (100mg/L)

Accurately weigh 10mg (accurate to 0.1mg) of blasticide standard into a 50mL beaker, and use Dissolve the acetonitrile solution (4:2:1) and transfer it to a 100mL volumetric flask, dilute to volume with acetonitrile, mix well, and store in a freezer at -18°C away from light: The validity period is 3 months:

4.4.2

Standard working solution (10 mg/L)

Take 1 mL of standard stock solution, add it to a 10 mL volumetric flask, dilute to volume with acetonitrile solution (4:2:1), and store in a freezer at -18°C away from light: The validity period is 1 month:

4.5

Materials

4.5.1

Solid phase extraction column

Weak cation exchange solid phase extraction cartridge (500mg/6mL, weak cation exchange column with carboxyl functional group):

4.5.2

Filter membrane

0.22μm, water system:

4.5.3

Octadecyl bonded silica gel (C)

40 μm ~ 60 μm:

5 Instruments and equipment

5.1

Liquid chromatography-tandem mass spectrometry

Equipped with electrospray ion source (ESI):

5.2

Analytical balance

Sensitivity 0:0001g and sensitivity 0:01g:

5.3

Tissue masher,

5.4

Centrifuge

The rotation speed is not less than 10000 r/min:

5.5

Rotary evaporator:

5.6

Vibration mixer (1200 times/min):

5.7

Vortex oscillator:

6 Sample preparation

Samples of vegetables, fruits and edible fungi should be taken in certain quantities in accordance with relevant standards, and the sampling locations should be in accordance with the provisions of GB 2763: For small individual samples, all are processed after sampling; for large individual and basically uniform samples, they can be divided or cut into small pieces on the axis or plane of symmetry and then processed; for slender, flat or component content in each part For samples with differences, small pieces can be cut from different parts or cut into small segments for processing; the sample should be chopped into small pieces, mixed thoroughly, sampled using the quartering method or directly put into a tissue masher and mashed into a homogenate: The homogenate was placed in a polyethylene container:

Take 500g of cereal samples, crush them so that they can all pass through a 425 μ m standard mesh sieve, and put them into polyethylene bottles or bags: Take 500g each of oil crops, tea leaves, nuts and spices samples, crush them and mix thoroughly, then put them into polyethylene bottles or bags:

Mix the vegetable oil evenly: Samples should be stored at temperatures below -18°C:

7 Analysis steps