



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

GB 23200.110-2018

**National food safety standard -- Determination of
forchlorfenuron residues in plant-derived foods -- Liquid
chromatography-mass spectrometry**

食品安全国家标准 植物源性食品中氯吡脞残留量的测定 液相色谱-质谱联用法
(English Translation)

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1 Scope

This standard specifies the liquid chromatography-mass spectrometry/mass spectrometry (HPLC-MS/MS) method for the determination of clofenuron residues in plant-derived foods:

This standard is applicable to the determination of clofenuron 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 chlorfenuron in the sample was extracted with acetonitrile, purified by dispersive solid phase extraction, measured by liquid chromatography-mass spectrometry, and quantified by the external standard method:

4 Reagents and materials

Unless otherwise stated, only reagents confirmed to be of analytical grade and in compliance with Class 1 water specified in GB/T 6682:

4.1

Reagents

4.1.1

Formic acid (HCOOH, CAS number

64-18-6): chromatographically pure:

4.1.2

Acetonitrile (CH₃CN, CAS number

75-05-8): chromatographically pure:

4.1.3

Anhydrous magnesium sulfate (MgSO₄, CAS number

7487-88-9):

4.1.4

Sodium chloride (NaCl, CAS number

12125-02-9):

4.2

Solution preparation Formic acid solution (1:499, volume ratio): accurately absorb 1mL of formic acid and add 499mL of water, mix well, and then place it in ultrasonic for 15 minutes and set aside:

4.3

Standard products Chlofenuron standard (C₁₂H₁₀ClN₃O, CAS number

68157-60-8): purity ≥99.0%:

4.4

Preparation of standard solution

4.4.1

Chlorfenuron standard stock solution (100mg/L)

Accurately weigh 10mg (accurate to 0.1mg) of Chlorfenuron standard in a 50mL beaker, dissolve it in acetonitrile and transfer it to a 100mL volumetric flask, and dilute to volume with acetonitrile: Fill to the mark, mix well, and store in a freezer at -18°C away from light: The validity period is 6 months:

4.4.2

Chlorfenuron standard working solution (10 mg/L)

Accurately draw 1 mL of standard stock solution, add it to a 10 mL volumetric flask, dilute to volume with acetonitrile, and mix: Stored at 4°C, the validity period is 1 month:

4.5

Materials

4.5.1

N-propylethylenediamine (PSA), 40pm~60pm:

4.5.2

Graphitized carbon black (GCB), 38pm~120pm:

4.5.3

Octadecyl bonded silica gel (Cis), 50µm:

4.5.4

Filter membrane

0.22µm, organic system:

5 Instruments and equipment

5.1

Liquid chromatography-mass spectrometer

Equipped with electrospray ion source (ESI):

5.2

Analytical balance, sensitive to 0.01g and 0.0001g:

5.3

Centrifuge, the rotation speed is not less than 8000 r/min:

5.4

Vortex oscillator:

6 Preparation of specimens

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 or segments

can be cut from different parts for processing; the sample should be chopped into small pieces, mixed thoroughly, and 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 425µm 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 are stored at -18°C and below:

7 Analysis steps

7.1

Extraction

7.1.1

Vegetables, fruits, edible fungi, vegetable oils Weigh 10g (accurate to 0.01g) sample into a 50mL centrifuge tube, add 10mL acetonitrile, vortex and extract for 3 minutes; add 4g anhydrous magnesium sulfate and 1g sodium chloride to the centrifuge tube for vegetables, fruits, and edible fungi: , vegetable oil, add 5g sodium chloride to the centrifuge tube, vortex for 1 min; then centrifuge at 4000 r/min for 5 min, and take 1.5 mL of supernatant for purification:

7.1.2

Cereals, oilseeds, nuts Weigh 5g (accurate to 0.01g) of the sample into a 50mL centrifuge tube, add 10mL of water, and vortex for 30 seconds to completely wet the sample; then add 10mL of acetonitrile, vortex and extract for 3 minutes; add 5g of chlorine to the centrifuge tube: sodium, vortex for 1 min; centrifuge at 4000r/min for 5 min:

Take 1.5mL of supernatant for purification:

7.1.3

Tea and spices Weigh 2g (accurate to 0.01g) of the sample into a 50mL centrifuge tube, add 10mL of water, and vortex for 30 seconds to completely wet the sample; then add 10mL of acetonitrile, vortex and extract for 3 minutes; add 5g of chlorine to the centrifuge tube: sodium, vortex for 1 min; centrifuge at 4000r/min for 5 min:

Take 1.5mL of supernatant for purification:

7.2

Purification