



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

GB 23200.109-2018

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

食品安全国家标准 植物源性食品中二氯吡啶酸残留量的测定 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 glufosinate residues in plant-derived foods:

This standard is applicable to the determination of glufosinate ammonium 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 glufosinate ammonium in the sample was extracted with water and methanol, and then dispersed and purified by solid phase materials: The purification solution was mixed with 9-fluorenylmethyl chloroformate:

The derivative glufosinate-FMOC (glufosinate-FMOC) generated after the reaction of

(9-fluorenylmethyl chloroformate) was analyzed by liquid chromatography Detection by spectrometry-mass spectrometry and quantification by external standard method:

4 Reagents and materials

Unless otherwise stated, only analytical grade reagents and first-grade water specified in GB/T 6682 were used in the analysis:

4.1

Reagents

4.1.1

Acetonitrile (CH_3CN , CAS number 75-05-8): chromatographically pure:

4.1.2

Methanol (CH_3OH , CAS number 67-56-1): chromatographically pure:

4.1.3

Ammonium acetate ($\text{CH}_3\text{COONH}_4$, CAS number 631-61-8):

4.1.4

Sodium borate ($\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$, CAS number 1303-96-4):

4:1:5 9-Fluorenylmethyl chloroformate ($\text{C}_{19}\text{H}_{13}\text{ClO}_2$, CAS number: 28920-43-6): purity 99.0%, stored at $0^\circ\text{C} \sim 4^\circ\text{C}$:

4.2

Solution preparation

4.2.1

Borate buffer solution (50:0g/L, pH=9): Weigh 5g of sodium borate ($\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$), dissolve it in water and dilute to 100mL:

4.2.2

Acetonitrile solution of 9-fluorenylmethyl chloroformate (10:0g/L): Weigh 1g of 9-fluorenylmethyl chloroformate, dissolve it in acetonitrile and adjust the volume to 100mL:

4.2.3

Ammonium acetate aqueous solution (5 mmol/L)

Weigh 0.385g of ammonium acetate and dissolve it in an appropriate amount of water, and adjust the volume to 1000mL with water:

4.3

Standard products Glufosinate ammonium (C₃H₈N₃O₄P, CAS number

77182-82-2) standard product: purity $\geq 99.0\%$:

4.4

Preparation of standard solution

4.4.1

Glufosinate ammonium standard stock solution (100 mg/L)

Accurately weigh 10.0 mg of glufosinate ammonium standard solution in a 50 mL beaker, dissolve it in water, transfer it to a 100 mL volumetric flask, and dilute to volume with water: Stored in a refrigerator at 0°C~4°C, it is valid for 6 months:

4.4.2

Glufosinate ammonium standard intermediate solution (10 mg/L)

Pipette 5.0 mL of glufosinate ammonium standard stock solution into a 50 mL volumetric flask, and dilute to volume with water: Store in 0°C~4°C refrigerator, valid for 1 month:

4.5

Materials

4.5.1

Neutral alumina (Al₂O₃, CAS number

1344-28-1): 0.075mm~0.15mm:

4.5.2

Multi-walled carbon nanotubes (MWCNTs)

Particle size range 10nm~20nm; particle length 5 μ m~15 μ m; specific surface area (225 $\pm$ 25)m²/g:

4.5.3

Filter membrane

0.22 μ m, organic system:

5 Instruments and equipment

5.1

Liquid chromatography-mass spectrometer

Equipped with electrospray ion source (ESD):

5.2

Analytical balance

Sensitivity 0.0001g, sensitivity 0.01g:

5.3

Tissue mashing instrument:

5.4

Centrifuge

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

5.5

Vortex oscillator:

5.6

Constant temperature water bath:

6 Sample preparation

Samples of vegetables, fruits and edible fungi are taken in a certain amount according to relevant standards, and the sampling parts are 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 425 μ m standard mesh sieve, and put them into polyethylene bottles or bags: Take 500g each of oil crops, tea leaves, nuts and spice 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

7.1

Extraction

7.1.1

Vegetables, fruits and edible fungi Weigh 10g (accurate to 0.01g) of the sample into a 50mL centrifuge tube, refer to Appendix A to add water, and then add 10mL methanol (4:1:2), after vortexing for 2 minutes, centrifuge at 3800r/min for 5 minutes to be purified: