



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

GB 23200.115-2018

**National food safety standard -- Determination of fipronil and its
metabolite residues in eggs -- Liquid chromatography -- Mass
spectrometry**

食品安全国家标准 鸡蛋中氟虫腈及其代谢物残留量的测定 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 fipronil and its metabolites residues in eggs:

This standard is applicable to the determination of fipronil and its metabolites residues in eggs:

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 sample was extracted with acetonitrile, and the extract was purified by dispersive solid-phase extraction, detected by liquid chromatography-mass spectrometry, and quantified by the external standard method:

4 Reagents and materials

Unless otherwise stated, only analytically pure reagents are used in the analysis, and the water is first-grade water specified in GB/T 6682:

4.1

Reagents

4.1.1

Acetonitrile (CH_3CN , CAS number

75-05-8): chromatographically pure:

4.1.2

Formic acid (HCOOH , CAS number

64-18-6): chromatographically pure:

4.1.3

Methanol (CH_3OH , CAS number

67-56-1): chromatographically pure:

4.1.4

Ammonium acetate ($\text{CH}_3\text{COONH}_4$, CAS number

631-61-8): chromatographically pure:

4.1.5

Anhydrous magnesium sulfate (MgSO_4 , CAS number

7487-88-9):

4.1.6

Sodium chloride (NaCl , CAS number

7647-14-5):

4.1.7

Anhydrous sodium sulfate (Na_2SO_4 , CAS number

7757-82-6):

4.2

Solution preparation

4.2.1

Formic acid solution (0.1%): Dilute 1mL of formic acid to 1000mL with water and shake well:

4.2.2

Ammonium acetate-formic acid solution (5mmol/L)

Weigh 0.3854g ammonium acetate, dissolve it with 0.1% formic acid solution and dilute it to 1000 mL, shake well:

4.3

Standard products for standard standards of fipronil and its metabolites, see Appendix A, purity $\geq 95\%$:

4.4

Preparation of standard solution

4.4.1

Standard stock solution (100mg/L)

Accurately weigh 10 ml each of fipronil, flufenoxuron, fipronil sulfone and fipronil sulfoxide (4:3) mg (accurate to 0.1 mg), dissolve and dilute to 100 mL with acetonitrile, shake well, and prepare a standard stock solution with a mass concentration of 100 mg/L: Store in the dark at -18°C and is valid for 1 year:

4.4.2

Mixed standard intermediate solution (1mg/L)

Accurately draw the standard reserves of fipronil, flufenoxuron, fipronil sulfone and fipronil sulfoxide respectively Dilute 1 mL of each solution (4:4:1) with acetonitrile to 100 mL, shake well, and prepare a 1 mg/L mixed standard intermediate solution: Store in the dark at 0°C to 4°C : The validity period is 1 month:

4.5

Materials

4.5.1

Ethylenediamine-N-propyl silanized silica gel (PSA)

40 μm ~ 60 μm :

4.5.2

Octadecylsilane bonded silica gel (Cg)

40 μm ~ 60 μm :

4.5.3

Microporous filter membrane (organic phase)

0.22 μm :

5 Instruments

5.1

Liquid chromatography-triple quadrupole mass spectrometer

Equipped with ESI source:

5.2

Analytical balance

Sensitive to 0.1mg and 0.01g:

5.3

Centrifuge

The rotation speed is not less than 5000 r/min:

5.4

Vortex oscillator:

5:5 oscillator:

5.6

Tissue homogenizer:

6 Sample preparation

6.1

Sample preparation Take 16 fresh eggs (about 1kg), wash and shell them, mix thoroughly with a tissue homogenizer, and put them into a polyethylene bottle:

6.2

Sample storage

Store the samples separately for testing and backup at -20°C ~ -16°C:

7 Analysis steps

7.1

Extraction

Accurately weigh 5g of the sample (accurate to 0.01g) and place it in a 50mL centrifuge tube, add 20mL of acetonitrile, vortex and mix for 1 min, shake and extract for 5 min, add 2g of sodium chloride and 6g of anhydrous sodium sulfate, vortex for 1 min: Centrifuge at 5000r/min for 5 minutes, and the supernatant needs to be purified: