



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

GB 23200.39-2016

Replacing SN/T 3139-2012

**Food safety national standard -- Determination of thiamethoxam and its
metabolite thiamethoxam residues in food by liquid
chromatography-mass spectrometry / mass spectrometry**

食品中噻虫嗪及其代谢物噻虫胺残留量的测定 液相色谱-质谱 质谱法

(English Translation)

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Foreword

This standard replaces SN/T 3139-2012 "Determination of thiamethoxam and its metabolite thiamethoxam residues in export agricultural products. Liquid Chromatography-Mass Spectrometry / Mass spectrometry".

Compared with SN/T 3139-2012, the main changes are as follows.

- Standard text format is modified to national standard text format for food safety;
- the name of the "export of agricultural products" to "food";
- increase the "other food reference implementation" in the standard range.

This standard replaced the previous version of the standard release.

-SN/T 3139-2012.

National standards for food safety Determination of thiamethoxam and its metabolite thiamethoxam residues in food Liquid chromatography - mass spectrometry/mass spectrometry

1 Scope

This standard specifies the method of liquid chromatography-mass spectrometry/mass spectrometry for the determination of thiamethoxam and thiamethoxam residues in food.

This standard applies to rice, soybeans, chestnuts, spinach, oil, vegetables, onions, eggplant, potatoes, citrus, mushrooms, tea and other plant sources Sexual products and chicken liver, pork, milk and other animal-derived products of thiamethoxam, thiophanate residues in the detection and confirmation, other food can refer to the Executive Row.

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 thiamethoxam and thiamethoxam residues in the samples were extracted by 0.1% acetic acid-acetonitrile, and purified by matrix dispersion solid phase extraction.

Determination by liquid chromatography - mass spectrometry/mass spectrometry and external standard method.

4 Reagents and materials

Unless otherwise stated, the reagents used are chromatographic grade and the water is in accordance with the primary water specified in GB/T 6682.

4.1 Reagents

4.1.1

Acetonitrile (CH₃CN).

4.1.2

Ammonium acetate (CH₃COONH₄).

4.1.3

acetic acid (CH_3COOH). analytical grade.

4.1.4

Formic acid (HCOOH).

4.1.5

Methanol (CH_3OH).

4.1.6

n-hexane (C_6H_{14}).

4.1.7

anhydrous magnesium sulfate (MgSO_4). analytical grade.

4.1.8

anhydrous sodium sulfate (Na_2SO_4). analytical grade.

4.1.9

N-propyl ethylenediamine (PSA) adsorbent. Particle size $40\ \mu\text{m} \sim 60\ \mu\text{m}$.

4.1.10

Graphite Carbon Black Adsorbent (GCB). Particle size $120\ \mu\text{m} \sim 400\ \mu\text{m}$.

4.1.11

Octadecylsilane (ODS) Bonded Adsorbent (C18). Particle size $40\ \mu\text{m}$ to $60\ \mu\text{m}$.

4.1.12

matrix dispersion solid phase extractant.

A) for rice, eggplant, onion, potato, citrus sample. 50 mg PSA, 150 mg MgSO_4 ;

B) for spinach, rapeseed sample. 50 mg PSA, 150 mg MgSO_4 , 10 mg GCB;

C) for chestnuts, mushrooms, milk samples. 50 mg PSA, 150 mg MgSO_4 , 50 mg C18;

D) for tea, soybean samples. 50 mg PSA, 150 mg MgSO_4 , 50 mg C18, 10 mg GCB;

E) for chicken liver, pork samples. 50 mg PSA, 150 mg MgSO_4 , 50 mg C18.

4.2 Solution preparation

4.2.1 0.1% acetic acid - acetonitrile solution. take 1 mL of acetic acid, add acetonitrile fixed to 1000 mL, mix.

4.2.2 10 mmol/L ammonium acetate solution (containing 0.1% formic acid). accurately weighed 0.16 g of ammonium acetate in a 200 mL volumetric flask, A small amount of

water dissolved after adding 200 µL formic acid, and then the water volume to the scale, mixing, is now with the distribution.

4.3 Standards

4.3.1

thiamethoxam. CAS No. 153719-23-4, molecular formula C₈H₁₀ClN₅O₃S, molecular weight 291.7, pure Degrees >= 99%.

4.3.2

clothianidin. CAS No. 205510-53-8, molecular formula C₆H₈ClN₅O₂S, molecular weight 250.2, pure Degrees >= 99%.

4.4 Standard solution preparation

4.4.1

thiamethasone standard stock solution. accurately weighed the appropriate amount of thiamethasone standard (accurate to 0.01 mg), dissolved in acetonitrile, preparation A standard stock solution of 1000 µg/mL was stored at -18 ° C.

4.4.2

Seamethamine standard stock solution. accurately weighed the appropriate amount of thiamethoxamine standard (accurate to 0.01 mg), dissolved in acetonitrile, preparation A standard stock solution of 1000 µg/mL was stored at -18 ° C.

4.4.3

thiamethoxam and xylidazole mixed standard working solution. according to the need to take appropriate amount of thiamethasone standard solution and the same solution the solution was diluted with acetonitrile to a standard working solution of 1 µg/mL and stored at -18 ° C.

4.4.4

matrix blank solution. the negative samples of different substrates were treated in accordance with the "extraction and purification" solution obtained after the solution.

4.4.5

matrix standard working fluid. according to the need to absorb the appropriate amount of mixed standard working solution, diluted with a matrix blank solution to the appropriate concentration of the standard working fluid, is now available with.

4.5 Materials