



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

GB 23200.45-2016

Replacing SN/T 0528-2012

**Food safety national standard -- Determination of difluomide
residues in food by liquid chromatography-mass spectrometry**

食品中除虫脲残留量的测定 液相色谱-质谱法

(English Translation)

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Foreword

This standard replaces SN/T 0528-2012 "High performance liquid chromatography - mass spectrometry/mass spectrometry" for the detection of diflubes in export food.

Compared with SN/T 0528-2012, the main changes are as follows.

- Standard text format is modified to national standard text format for food safety;
- the name of the "export food" to "food";

- increase the "other food reference implementation" in the standard range.

This standard replaced the previous version of the standard release.

-SN 0528-1996;

-SN/T 0528-2012.

National standards for food safety Determination of difluomide residues in food by liquid chromatography - mass spectrometry

1 Scope

This standard specifies the method of liquid chromatography-mass spectrometry for the removal of diflubenzuron in food.

This standard applies to rice, corn, soybeans, wheat, peanuts (peanuts), oranges, apples, celery, onions, mushrooms (fresh), honey, Mushrooms (dry), tea, chicken, beef, pork, pig liver in the determination and removal of insecticides, other food can refer to the implementation.

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 file. 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 pyrethroids in the samples were extracted with acetonitrile and dispersed by solid phase extraction and purified by liquid chromatography-tandem mass spectrometry.

Quantitative.

4 Reagents and materials

Unless otherwise stated, the reagents used were of analytical grade and water was a primary water specified in GB/T 6682.

4.1 Reagents

4.1.1

Acetonitrile (CH₃CN). Chromatographically pure.

4.1.2

n-hexane (C₆H₁₄).

4.1.3

glacial acetic acid (CH₃COOH).

4.1.4

anhydrous sodium acetate (CH₃COONa).

4.1.5

sodium chloride (NaCl). 450 °C burning 4 h, sealed standby.

4.1.6

anhydrous sodium sulfate (Na₂SO₄). 650 °C burning 4 h, storage dryer in reserve.

4.1.7

anhydrous magnesium sulfate (MgSO₄). 650 °C burning 4 h, storage dryer in the spare.

4.1.8

Primary secondary amine (PSA). PSA filler or equivalent, particle size 40 μm.

4.1.9

Octadecyl silane bonded phase (C18). C18 filler or equivalent, particle size 50 μm.

4.2 Solution preparation

4.2.1

glacial acetic acid - acetonitrile solution (0.1 99.9, V/V). take 0.1 mL of glacial acetic acid by adding 99.9 mL of acetonitrile, mix and spare.

4.3 Standards

4.3.1

Pesticide standard. CAS No.. 35367-38-5, purity greater than or equal to 96%.

4.4 Standard solution preparation

4.4.1

Pesticide standard stock solution. accurately weighed appropriate amount of insecticides (accurate to 0.1 mg), with acetonitrile prepared into a concentration of 1.0 Mg/mL standard stock solution, set -18 °C refrigerator to save, the shelf life of 6 months.

4.4.2

Deicerurea standard working solution. according to the need to use acetonitrile standard stock solution diluted to the appropriate concentration of standard working fluid, temporary use with 4.4.3 0.005 mol/L ammonium acetate aqueous solution. Weigh 0.3850 g ammonium acetate, dissolved in water to 1 L, over 0.45 µm filter preparation use.

4.5 Materials

4.5.1

Microporous membrane. 0.22 µm, organic.

5 Instruments and equipment

5.1

High Performance Liquid Chromatography-Tandem Mass Spectrometer. Triple quadrupole tandem mass spectrometry with electrospray ionization source (ESI).

5.2

Analysis of balance. 0.01 g and 0.0001 g.

5.3

with a plug centrifuge tube. 50 mL, polypropylene capped plastic centrifuge tube or equivalent; 10 mL, glass with a plug tube or equivalent.

5.4

Scroll Mixer.

5.5

Ultrasonic cleaner.

5.6

Centrifuge.

5.7

sample grinder.

5.8

Tissue crusher.

5.9

Sample Sieve. A pore size of 2 mm.