



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

GB 23200.42-2016

Replacing SN/T 1017.9-2004

**Food safety national standard -- Detection Method of Residual
Fluoride in Grain**

粮谷中氟吡禾灵残留量的检测方法

(English Translation)

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Table of Contents

Foreword	
1 Scope	
2 Normative reference documents	
3 Principle	
4 Reagents and materials	
4.1 Reagents	
4.2 Solution preparation	
4.3 Standards	
4.4 Standard solution preparation	
4.5 Materials	
5 Instruments and equipment	
5.10 Analysis of balance. 0.01 g and 0.0001 g	
6 Preparation and storage of samples	
6.1 Preparation of the sample	
6.2 Sample storage	
7 Analysis steps	
7.1 Extraction and purification	
7.2 Derived	
7.3 Silica gel column purification	
7.4 Determination	
7.5 Blank test	
8 Results are calculated and expressed	
9 Precision	
10.1 Quantitation limits	
10.2 Recovery rate	

Foreword

This standard replaces SN/T 1017.9-2004 "Method for the determination of fluticillin residues in import and export grain".

This standard compared with SN/T 1017.9-2004, the main changes are as follows.

- Standard text format is modified to national standard text format for food safety;

- the name of the "import and export grain" to "grain".
- increase the "other food reference implementation" in the standard range.

This standard replaced the previous version of the standard release.

-SN/T 1017.9-2004.

National standards for food safety Detection Method of Residual Fluoride in Grain

1 Scope

This standard specifies the sampling, sample preparation and gas chromatographic methods for the determination of flupirtine residues in grain.

This standard applies to the determination of vanillafen residues in rice, and other foods may be referred to the implementation.

2 Normative reference documents

The following documents are indispensable for the application of this document. For dated references, only the date of the note applies this document. 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

Extraction of phosphoric acid and acetone fluoride in the sample of fluoride, the extract extracted with chloroform, and then through the liquid distribution purification. Then by five Fluorobromotoluene was purified and purified by silica gel column. The method was quantitatively determined by gas chromatograph with electron capture detector.

4 Reagents and materials

Unless otherwise specified, all reagents are of analytical grade and water is in accordance with the primary water specified in GB/T 6682.

4.1 Reagents

4.1.1

Trichloromethane (CHCl_3). Distillation.

4.1.2

anhydrous ether ($\text{C}_4\text{H}_{10}\text{O}$). re-distillation.

4.1.3

n-Hexane (C₆H₁₄). for pesticide residues analysis.

4.1.4

Acetone (C₃H₆O). re-distillation.

4.1.5

Dichloromethane (CH₂Cl₂). Distillation.

4.1.6

phosphoric acid (H₃PO₄).

4.1.7

Triethylamine (C₆H₁₅N).

4.1.8

Toluene (C₇H₈).

4.1.9

Hydrochloric acid (HCl).

4.1.10

anhydrous sodium sulfate (Na₂SO₄). anhydrous sodium sulfate at 650 °C for 4 hours, into the dryer standby.

4.2 Solution preparation

4.2.1

Phosphate solution. 1 g of sodium hydroxide and 17 g of disodium hydrogen phosphate, dissolved in water and diluted to 500 mL.

4.2.2

Pentafluorobromotoluene (PFBBR) Derivative Reagent. 100 µL Pentafluorobromotoluene was dissolved in 4.9 mL of toluene.

4.3 Standards

4.3.1

Fluprofen standard. content $\geq$ 99.5%.

4.4 Standard solution preparation

4.4.1

Fenfloxacine standard solution. accurately weighed the appropriate amount of Fenfen Wo Ling standard, with methanol added 0.1mg/mL of the stock solution, according to Need to be diluted to the appropriate concentration of standard working fluid.

4.5 Materials

4.5.1

silica gel column. 3 mL, before use in the top of the column loaded 1cm high anhydrous sodium sulfate, and with two grams of methanol-n-hexane (1585) Solution 3 mL pre-leaching.

5 Instruments and equipment

5.1

Gas Chromatograph with Electronic Capture Detector (ECD).

5.2

Quick Mixer.

5.3

Centrifuge. 4 000 r/min.

5.4

Multifunctional micro-chemical sample processor or other equivalent instruments.

5.5

with a plug centrifuge tube. 5 mL.

5.6

Centrifuge tube. 5 mL, 15 mL.

5.7

pointed mouth straw.

5.8

Trace Adjustable pipettes. 50 μ L, 200 μ L, 1 000 μ L.

5.9

Microinjector. 10 μ L.