



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

GB 23200.95-2016

Replacing SN 0691-1997

**Food safety national standard -- Detection method of
fenpropathrin residues in bee products**

蜂产品中氟胺氰菊酯残留量的检测方法

(English Translation)

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Foreword

This standard replaces SN 0691-1997 "Method for the determination of fenpropathrin residues in export bee products".

This standard compared with SN 0691-1997, the main changes are as follows.

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

This standard replaced the previous version of the standard release.

-SN 0691-1997.

National standards for food safety Detection method of fenpropathrin residues in bee products

1 Scope

This standard specifies the sampling, sample preparation and gas chromatographic method for the determination of fenpropathrin residues in export bee products.

This standard applies to the export of honey in the residual determination of fenpropathrin residues, 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 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

After the sample was alkalized, it was extracted with n-hexane-acetone, and the extract was purified by liquid-liquid partitioning with acetonitrile and n-hexane.

Into the acetonitrile layer. Acetonitrile extract and then evaporated to dry, with n-hexane dissolved residue, solution for gas chromatography determination, external standard method.

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

Acetone (C₃H₆O). Distillation.

4.1.2

Sodium hydroxide (NaOH) aqueous solution. 2 m ol/L.

4.1.3

N-Hexane (C₆H₁₄). Distillation.

4.1.4

anhydrous sodium sulfate (Na₂SO₄). 650 °C burning 4 h, stored in a sealed container for use.

4.2 Solution preparation

4.2.1

N-Hexane. Saturated with acetonitrile.

4.2.2

acetonitrile. saturated with n-hexane.

4.3 Standards

Fenvalerate standards. purity $\geq$ 97%.

4.4 Standard solution preparation

Accurately weigh the appropriate amount of fenpropathrin standards, with n-hexane dubbed the concentration of 20 µg/mL standard stock solution, and then need to use N-Hexane is formulated into a standard working solution at an appropriate concentration. Store in 4 °C refrigerator.

5 Instruments and equipment

5.1

Gas chromatograph with electronic capture detector.

5.2

Analysis of balance. 0.01g and 0.0001g.

5.3

Rotary Evaporator.

5.4

Oscillator.

6.1 Preparation of the sample

The samples will be thoroughly stirred, divided into two, into the sample bottle as a sample, sealed, marked mark. Such as retrieving samples in a knot when the crystal is precipitated, the vial is capped and placed in a water bath which does not exceed 40 ° C. To be all

melted after crystallization, open cover, stir well, are divided into two, sealed, and marked with the mark.

In the case of a cup of honey, all the cups of honey will be retrieved, poured together, fully stir well, and take out about 1 kg to represent the sample. And then 1 kg on behalf of the equipment After the samples were mixed, they were divided into two portions and placed in a sample bottle as a sample. Sealed, marked.

Note: The above sample sampling site according to GB 2763 Appendix A implementation.

6.2 Sample storage

Store the sample at room temperature.

Note: During sampling and sample preparation, it is necessary to prevent contamination of the sample or change in the content of the residue.

7.1 Extraction

Weigh about 20 g of sample (accurate to 0.1 g), add 20 mL of sodium hydroxide solution (2 mol/L) to a 125 mL stoppered flask, Oscillation causes the sample to dissolve. Pour into a 100 mL separatory funnel, add 20 mL of n-hexane and 10 mL of acetone, shake for 5 min, standing stratified. will the n-hexane layer was filtered into.200 mL of pear-shaped bottles and 20 mL of n-hexane and 10 mL of acetone were added to the residual night and shaken for 5 min.

Operation once. The n-hexane layer was combined with the pear-shaped flask and evaporated to 40 ° C and evaporated to dryness.

7.2 Purification

The pear-shaped bottle was rinsed with 30 mL of acetonitrile saturated n-hexane and 30 mL of n-hexane saturated acetonitrile and the residue was dissolved. The solution was transferred to 100 mL of the solution Funnel, intense oscillation 5 min, static stratification. Separate the acetonitrile layer into a.200 mL separatory funnel. Add 2 x 30 mL to n-hexane Hexane-saturated acetonitrile, repeat the above procedure. Consolidate the acetonitrile layer in the appendix to the separatory funnel. Add 50 mL of acetone to the separatory funnel and n-hexane, intense oscillation 5 min, standing stratification. The acetonitrile layer was taken in a.200 mL pear-shaped flask and evaporated at 40 ° C or below to just dry. The residue was dissolved in 5.0 mL of n-hexane and the solution was subjected to gas chromatography.