



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

GB 23200.49-2016

Replacing SN/T 1975-2007

**Food safety national standard -- Determination of permethine
residues in food by gas chromatography-mass spectrometry**

食品中苯醚甲环唑残留量的测定 气相色谱-质谱法

(English Translation)

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Foreword

This standard replaces SN/T 1975-2007 "Method for the determination of benzofenazole residues in food for import and export by gas chromatography-mass spectrometry".

Compared with SN/T 1975-2007, 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 food" to "food";
- increase the "other food reference implementation" in the standard range.

This standard replaced the previous version of the standard release.

-SN/T 1975-2007.

National standards for food safety Determination of permethine residues in food by gas chromatography - mass spectrometry

1 Scope

This standard specifies the preparation and determination of difenoconazole in food by gas chromatography-mass spectrometry.

This standard applies to peas, basil, carrots, spinach, rice, soybeans, Chinese medicine, tea, almonds, grapefruit, pineapple, strawberry, Soy sauce, vinegar, royal jelly dry powder, pork, beef, chicken, eel and lobster and other foods in the determination of the amount of phenylethylenediazole residues Confirmed that 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 sample was treated with ethyl acetate, and the mixture was treated with tandem activated carbon and neutral alumina double column method or Florisil single column method the extraction was confirmed by gas chromatography - mass spectrometry and quantification.

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

Ethyl acetate (C₄H₈O₂). Chromatographic purity.

4.1.2

n-hexane (C₆H₁₄). pure chromatography.

4.1.3

Acetone (C₃H₆O). Chromatographic pure.

4.1.4

anhydrous sodium sulfate (Na_2SO_4). 650 °C burning 4 h, placed in a closed container for use.

4.2 Solution preparation

4.2.1

N-Hexane Acetone (3 2, V/V) Mixing Solvent. Take 300 mL of n-hexane, add.200 mL of acetone and mix well.

4.3.1 Phenylethylenediamine standard substance (Difenoconazole, $\text{C}_{19}\text{H}_{17}\text{Cl}_2\text{N}_3\text{O}_3$, CAS

5.3 Solid phase extraction device with vacuum pump.

5.4 Tissue crusher.

5.5 Crusher.

5.6 Centrifuge.

5.7 Rotary Evaporator.

6.1.1 Vegetables or fruits

500 g of the sample was taken and the mixture was chopped and the sample was processed into a slurry by a pulverizer. Mix, are divided into two as a test Sample, into the clean container, sealed and marked.

6.1.2 Tea, nuts and cereals

500 g of the representative sample was taken, pulverized by a pulverizer and passed through a 2.0 mm round hole sieve. Mix well, into a clean container, sealed and marked Mark.

6.1.3 Meat and meat products

After picking 500 g of the sample, it was chopped and the sample was processed into a slurry by a mashing machine, and the mixture was packed into a clean bag Inside, sealed and marked.

6.1.4 Spices

Replace the sample 500 g, mix, sub-packed into a clean container, sealed and marked.

6.1.5 Bee products

Take a representative sample volume of 500g, the crystallization of honey samples will be evenly stirred; on the crystallization of honey samples, in the closed the case, the sample bottle placed in not more than 60 °C in the water bath in the warm, shaking, until the sample all melted and stir well, quickly cooled to room temperature, in the melting must pay attention to prevent moisture evaporation. The prepared samples were divided into two separate sections, which were sealed in a vial and sealed Remember.

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

6.2 Sample storage

Tea, bee products, condiments and cereals and other samples stored at 4 °C; fruits and vegetables and meat and meat products such as samples at -18 °C the following frozen storage.

During the operation of the sample preparation, the sample should be protected from contamination or changes in the content of the residue.

7.1 Extraction

For samples with low water content or higher fat content (tea, rice, beans, meat and meat products, bee products, etc.), Take 5 g homogeneous sample (accurate to 0.01 g). For samples with high water content (vegetables, fruits, soy sauce and vinegar spices, etc.), Take 10 g homogeneous sample (accurate to 0.01 g). Place the weighed sample in a 250 mL stoppered flask with 50 mL of ethyl acetate Into 15 g of anhydrous sodium sulfate, placed in the oscillator for 40 min, filtered in 150 mL concentrated bottle. Add 20 mL of ethyl acetate to repeat the extract was extracted once and the extract was concentrated at 40 ° C under reduced pressure to dryness. With 3 mL of n-hexane dissolved, to be purified.

7.2.1 High pigmented samples

The activated carbon column and the neutral alumina column in series, followed by 5 mL of acetone, 5 mL of n-hexane activation, the n-hexane extraction solution Column, after the end and then 3 mL n-hexane cleaning bottle and column, to maintain the droplet flow rate of about 2 mL/min, remove the filtrate, dry, with 5 mL N-hexane acetone (3 2, V/V), and the eluate was collected in 10 mL of small tubes and dried in a water bath at 40 ° C, 1.0 mL with n-hexane, and 0.45 µm organic phase filter, gas chromatography-mass spectrometry and confirmation.