



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

GB 23200.97-2016

Replacing SN/T 1593-2005

**Food safety national standard -- Determination of 5
organophosphorus pesticide residues in honey -- Gas
chromatographic method**

蜂蜜中5种有机磷农药残留量的测定 气相色谱法

(English Translation)

Issued on: December 18, 2016

Implemented on: June 18, 2017

**Issued by: National Health and Family Planning Commission of the
People 's Republic of China**

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Foreword

This standard replaces SN/T 1593-2005 "five kinds of organophosphorus pesticide residues in the import and export of honey - Gas Chromatography".

Compared with SN/T 1593-2005, the main changes are as follows.

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

This standard replaced the previous version of the standard release.

-SN/T 2572-2010.

National standards for food safety Determination of 5 organophosphorus pesticide residues in honey - Gas chromatographic method

1 Scope

This standard specifies the determination of trichlorfon, dolphin, chlorpyrifos, malathion, and phosphotoxic pesticide residues in honey by gas chromatography method.

This standard is applicable to the determination and confirmation of trichlorfon, dolphin, chlorpyrifos, malathion and phosphotoxic pesticide residues in honey.

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. 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 honey was diluted with water, the organophosphorus pesticide in the sample was extracted with ethyl acetate, concentrated at low temperature, and the gas phase was measured with a flame photometric detector. Determination of spectrometer, 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

Ethyl acetate (C₄H₈O₂). Chromatographic purity.

4.1.2

Sodium chloride (NaCl).

4.2 Standards

4.2.1

Organophosphorus standards. trichlorfon (CAS. 52-68-6), dolphin (fenchlorphos, CAS. 299-84-3), Chlorpyrifos, CAS. 2921-88-2, malathion, CAS. 121-75-5, coumaphos, CAS. 56-72-4) with a purity of > 97%.

4.3 Standard solution preparation

4.3.1

trichlorfon, dolphin phosphorus, chlorpyrifos, malathion, fly poison phosphorus standard stock solution. were weighed the appropriate standard, respectively, with B the ethyl acetate was dissolved in a volume of 100 mL and the solution concentration was 100 µL/mL and stored in a 4 ° C refrigerator. Before use, dilute with ethyl acetate as needed to the appropriate concentration, as a mixed standard working fluid.

5 Instruments and equipment

5.1

Gas Chromatograph with Flame Photometric Detector (Phosphor Filter 525 nm).

5.2

Analysis of balance. 0.01 g and 0.0001 g.

5.3

Rotary Evaporator.

5.4

Centrifuge. 4 000 r/min.

5.5

Mixer.

6 Preparation and storage of samples

Stirring of the un-crystallized sample is carried out evenly, and the sampling site is carried out according to GB 2763 Appendix A. For samples with crystalline precipitates, After the cap is tightened, it is warmed in a water bath that does not exceed 60 ° C. Once the sample has melted and stirred, it is rapidly cooled to room temperature. Must be thawed Pay attention to prevent moisture from evaporation. The prepared samples were divided into two separate sections, sealed into the vials, sealed and marked.

During the operation of the sample preparation, the contamination of the sample or the change in the content of the residue should be prevented. Store the sample at room temperature.

7.1 Extraction

Weigh 10 g homogeneous sample (accurate to 0.01 g), placed in 50 mL centrifuge tube, add 15 mL of water and 5 g of sodium chloride, mix, Then add 15 mL of ethyl acetate, mix on the mixer for 2 min, centrifuge 5 min (3 000 r/min), with a pointed nozzle to the upper

layer of acetic acid the ethyl ester solution was transferred to a 100 mL concentrated flask and the aqueous phase was added to 15 mL of ethyl acetate. The above procedure was repeated and the extracts were combined twice.

The evaporator was concentrated in a water bath at 40 ° C under reduced pressure to near dryness, dissolved in 8 mL of ethyl acetate and quantitatively transferred to a 10 mL centrifuge tube.

The following water bath with a gentle nitrogen flow to dry, accurately add 1 mL of ethyl acetate, mix, gas chromatographic determination.

7.3.1 Gas Chromatographic Reference Conditions

A) Column. quartz capillary column, DB-1701, 30 mx 0.25 mm (inner diameter) x 0.25 m (film thickness), or equivalent;

B) Carrier gas. nitrogen (purity greater than 99.999%); carrier gas flow rate 1.0 mL/min; tail gas flow rate. 30 mL/min; hydrogen flow rate.

75 mL/min, air flow rate. 100 mL/min;

C) Column temperature. initial temperature 50 ° C for 2 min, rise to 180 ° C at 15 ° C/min for 1 min, rise to 290 ° C at 8 ° C/min Keep for 10 min;

D) Inlet temperature. 250 ° C;

E) Detector temperature. 250 ° C;

F) Injection method. no split injection;

G) Injection volume. 2 µL;

H) Opening time. 1.5 min.

7.3.2 Chromatographic determination

According to the content of organophosphorus pesticides in the sample, the standard working solution with similar peak area is selected. Standard working solution and sample solution the response value of organophosphorus should be within the linear range of the instrument. Standard working solution and sample volume and other volume interspersed injection determination. In the above chromatographic conditions the retention time of trichlorfon was about 6.0 min, the retention time of flocculent phosphorus was about 17.8 min, the retention time of chlorpyrifos was about 18.5 min, the retention time of malathion was about 18.9 min, and the retention time of flocculent phosphorus was about 27.8 min. The chromatogram of the standard is shown in Appendix A.

Figure A.1.