



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

GB 23200.16-2016

Replacing NY/T 1016-2006

**Food safety national standard -- Determination of ethephon
residues in fruits and vegetables -- Liquid chromatographic
method**

水果蔬菜中乙烯利残留量的测定 气相色谱法

(English Translation)

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Foreword

This standard replaces NY/T 1016-2006 "Determination of ethephon residues in vegetables by gas chromatography".

This standard and GB/T 5009.7-2008 compared to the main changes are as follows.

- modified in accordance with the format of food safety standards;
- normative reference documents to increase the GB 2763 "maximum pesticide residues in food" standard;

- the preparation of the sample to increase the sampling site requirements and refine the requirements of the preparation of the sample;

- Increased precision requirements.

National standards for food safety Determination of ethephon residues in fruits and vegetables - Gas chromatographic method

1 Scope

This standard specifies the determination of ethephon residues in fruits and vegetables.

This standard is applicable to the analysis of ethephon residues in fruits and vegetables.

2 Normative reference documents

The following documents are indispensable for the application of this document. For dated filing applications, only the dated edition applies to 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 ethephon from the sample with methanol and derivatization of dimethylethylenediethanoxide by diazomethane with a flame photometric detector (phosphor filter) the gas chromatograph was determined by external standard method.

4 Reagents and materials

Unless otherwise stated, only chromatographic pure reagents are used in the analysis, and water is the primary water specified in GB/T 6682.

4.1 Reagents

4.1.1

Potassium hydroxide (KOH).

4.1.2

Hydrochloric acid (HCl).

4.1.3

Methanol (CH₃OH).

4.1.4

anhydrous ether (C₂H₆O).

4.2 Solution preparation

Methanol - hydrochloric acid solution (90 10). take 90mL of methanol added to 10mL hydrochloric acid, and mix.

4.3 Standards

Ethephon standard, purity $\geq 95\%$.

4.4 Standard solution preparation

4.4.1

ethephon standard solution. accurately weighed the amount of ethephon standard, with methanol prepared into a mass concentration of 1000 mg/L standard storage Preparation of liquid. According to the need to dilute the standard stock solution with methanol to prepare the appropriate concentration of standard working fluid, the preparation process requires polyethylene containers.

4.4.2

Diazomethane solution. see Appendix A.

5 Instruments

5.1

gas chromatograph and equipped with flame photometric detector.

5.2

Ultrasonic cleaner.

5.3

Tissue crusher.

5.4

nitrogen dryers.

6.1 Preparation of the sample

The samples of vegetables and fruit samples shall be sampled according to the provisions of Appendix A of GB 2763, and the whole sample shall be processed for the smaller samples.

For a larger, substantially uniform sample, it can be divided or cut into small pieces on a

symmetry or symmetry plane; for slender, flat or component Samples in different parts of the sample can be cut in different parts of small pieces or cut into small pieces or processing; take the sample after its chopped, fully mixed Uniform, with a quartile sampling or directly into the tissue crusher mashed into homogenate, into the polyethylene bottle in the $-16\text{ }^{\circ}\text{C} \sim -20\text{ }^{\circ}\text{C}$ under the conditions of preservation.

6.2 Extraction

Weigh the sample 10 g (accurate to 0.01g) in the polyethylene beaker, add 0.5 mL of methanol - hydrochloric acid solution and 50 mL of methanol, ultrasound Shock extraction 5min, filtered in 100 mL of polyethylene volumetric flask, the residue and then 30 mL of methanol extraction time, combined with the extraction of polyethylene Capacity bottle, the volume of 100 mL.

6.3 Derivatization

Accurately absorb 10 mL of the above volume solution into the 15 mL polyethylene centrifuge tube, in a dry nitrogen flow on the $30\text{ }^{\circ}\text{C} \sim 35\text{ }^{\circ}\text{C}$ water bath Concentrated to about 1.5 mL, add 0.5 mL of methanol - hydrochloric acid solution and 8 mL of anhydrous ether, mixed well, placed 10 min, the supernatant Into another polyethylene centrifuge tube, the residual solution with 1 mL of anhydrous ether extraction 2 times, the extract into the above solution, at $30\text{ }^{\circ}\text{C} \sim 35\text{ }^{\circ}\text{C}$ Concentrate to about 1 mL in a water bath. In the fume hood, dropping the diazomethane solution into the concentrate until the yellow does not fade. Cover the claws, Placed for 15 min. Concentrated in a nitrogen stream at $30\text{ }^{\circ}\text{C}$ to $35\text{ }^{\circ}\text{C}$ to about 1 mL, diluted with ether to 2.00 mL, and analyzed by gas chromatography.

6.4 Derivation of standard solutions

Take the appropriate amount of ethephon standard working solution (10.0 mL with methanol), follow the sample extraction and derivatization steps (5.2 and 5.3) to operate.

6.5 Gas Chromatographic Reference Conditions

Column. FFAP 30 m x 0.32 mm (id) x 0.25 μm Elastic quartz capillary column or quite polar column.

Carrier gas. nitrogen, flow rate 2.5 mL/min, purity $\geq 99.999\%$;

Gas. hydrogen, flow rate 85 mL/min, purity $\geq 99.999\%$;

Gas combustion. air, flow 110 mL/min.

Inlet temperature. $240\text{ }^{\circ}\text{C}$;

Heating procedure. $120\text{ }^{\circ}\text{C}$ (1min) $40\text{ }^{\circ}\text{C}/\text{min}$ $230\text{ }^{\circ}\text{C}$ (2min);

Detector temperature $150\text{ }^{\circ}\text{C}$.