



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

GB 23200.17-2016

Replacing NY/T 1649-2008

**Food safety national standard -- Determination of
Thiabendazole Residues in Fruits and Vegetables by Liquid
Chromatography**

水果蔬菜中噻菌灵残留量的测定 液相色谱法

(English Translation)

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Foreword

This standard replaces NY/T 1649-2008 "Determination of thiabendazole residues in fruits and vegetables by high performance liquid chromatography".

This standard and NY/T 1649-2008 compared to the main changes are as follows.

- the standard name has been modified to increase the national standard for food safety;
- modified in accordance with the format of the food safety standard.
- 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 Thiabendazole Residues in Fruits and

Vegetables by Liquid Chromatography

1 Scope

This standard specifies the method for the determination of thiabendazole residues in vegetables and fruits by high performance liquid chromatography (HPLC).

This standard applies to the determination of thiabendazole residues in vegetables and fruits.

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

Samples of thiabendazole in the sample after extraction, according to thiophanate in acidic conditions dissolved in water, alkaline conditions dissolved in the principle of ethyl acetate, Purification, and then by the reverse phase chromatography, UV detector 300nm detection, according to the retention time qualitative, external standard method quantitative.

4 Reagents and Materials

Unless otherwise stated, only reagents identified as analytical reagents and water at GB/T 6682 level were used in the analysis.

4.1 Reagents

4.1.1

Methanol (CH₃OH), chromatographic pure.

4.1.2

Ethyl acetate (CH₃COOC₂H₅).

4.1.3

Sodium chloride (NaCl).

4.1.4

anhydrous sodium sulfate (Na_2SO_4). 650 °C burning 4 h, the dryer to save.

4.2 Solution preparation

4.2.1

hydrochloric acid solution (0.1 mol/L). absorb 8.33 mL of hydrochloric acid, water volume to 1 L.

4.2.2

Sodium hydroxide solution (1.0 mol/L). Weigh 40 g of sodium hydroxide, dissolve in water and set to 1 L.

4.3 Standards

Thiabendazole (CAS 148-79-8). purity greater than 99%.

4.4 Standard solution preparation

Standard storage solution (100 mg/L). accurately weighed thymidazole 0.0100 g, dissolved in methanol, the volume to 100 mL, set 4 °C preservation, Valid for 3 months.

5 Instruments and equipment

5.1

High Performance Liquid Chromatograph with UV Detector.

5.2

Analysis of balance. 0.01 g and 0.1 mg.

5.3

Tissue crusher.

5.4

Rotary Evaporator.

5.5

Mechanical reciprocating oscillator.

5.6

Buchner funnel.

6 Sample preparation

Sampling samples of vegetables and fruit samples according to GB 2763-2014 Appendix A sampling, for the smaller individual samples, after sampling the whole for the individual large uniform sample can be split on the axis of symmetry or symmetry or cut into small pieces of post-processing; for slender, flat Flat or component content in the various parts of the sample can be cut in different parts of small pieces or cut into small pieces or processing; take the sample after the chopped, Fully mixed, with a quartile sampling or directly into the tissue crusher mashed into homogenate. The homogenate was placed in a polyethylene bottle at -16 ° C to -20 ° C Save under the file.

7.1 Extraction and purification

Weigh 10 g sample, accurate to 0.01 g, into a 250 mL stoppered flask, add 40 mL of methanol, homogenize 1 min, Shaking on the shaker for 20 min, the Buchner funnel was filtered and the residue was washed twice with the appropriate amount of methanol. The filtrate was combined in a 150 mL pear flask.

50 °C under reduced pressure evaporated to the remaining 5 mL ~ 10mL, with 20 mL hydrochloric acid solution into the 250 mL separatory funnel, add 20 mL of ethyl acetate vibration Swing, stand still, ethyl acetate layer and then 20 mL hydrochloric acid solution extraction time. The combined aqueous phase was adjusted to pH 8 to 9 with sodium hydroxide solution and 4 g was added Sodium chloride, into the 250 mL separatory funnel, with 40 mL of ethyl acetate were extracted 2 times, combined with ethyl acetate, dehydrated by anhydrous sodium sulfate, at 50 ° C under reduced pressure rotary evaporation near dry, the residue dissolved with mobile phase and volume to 5 mL, filtered through the 0.45µm filter to be tested.

7.2 Liquid Chromatographic Reference Conditions

Detector. UV detector.

Column. C18, 4.6 x 250 mm (5 µm) or equivalent.

Mobile phase. methanol water = 50 50.

Flow rate. 1.0 mL/min.

Detection wavelength. 300 nm.

Column temperature. room temperature.

Injection volume. 10 µL.