



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

GB 23200.87-2016

Replacing SN 0606-1996

**Food safety national standard -- Determination of thiabendazole
residues in milk and dairy products -- Fluorescence
spectrophotometric method**

乳及乳制品中噻菌灵残留量的测定 荧光分光光度法

(English Translation)

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Foreword

This standard replaces SN/T 0606-1996 "Test method for the determination of thiabendazole residues in milk and dairy products by fluorescence spectrophotometry".

Compared with SN/T 0606-1996, the main changes are as follows.

- Standard text format is modified to national standard text format for food safety;
- "Milk and dairy products" in the standard name "Export milk and dairy products";

- increase the "other food reference implementation" in the standard range.

This standard replaced the previous version of the standard release.

SN/T 0606-1996.

National standards for food safety Determination of thiabendazole residues in milk and dairy products - Fluorescence spectrophotometric method

1 Scope

This standard specifies sampling, sample preparation and fluorescence spectrophotometric methods for the determination of thiabendazole residues in milk and dairy products.

This standard applies to the detection of thiabendazole residues in fresh milk, 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

The fats in the sample were saponified with potassium hydroxide, and the thiabendazole was extracted with ethyl acetate. The extract of ethyl acetate was extracted with hydrochloric acid solution Determination of Fluorescence Spectrophotometry, Quantitative Determination of 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

Potassium hydroxide (KOH).

4.1.2

Hydrochloric acid (HCl).

4.1.3

Ethyl acetate (CH_3COOH).

4.2 Solution preparation

4.2.1 50% (m/V) potassium hydroxide solution. accurately weighed 50 g of potassium hydroxide solid, dissolved in water, cooled to 100 mL, Shaking back.

4.2.2 0.05% (m/V) potassium hydroxide solution. accurate removal of 50% potassium hydroxide solution (4.2.1) 1mL in 1000 mL volumetric flask, Water volume, shake back spare.

4.2.3 0.1 mol/L hydrochloric acid solution. accurate removal of 0.83 mL concentrated hydrochloric acid in a 100 mL volumetric flask, the volume of water, shake back.

4.3 Standards

4.3.1

Thiabendazole standard. purity $\geq 99\%$.

4.4 Standard solution preparation

4.4.1

Thiabendazole standard stock solution. accurately weighed the appropriate amount of thiabendazole standard, with hydrochloric acid solution dubbed the concentration of 0.100 mg/mL Standard stock solution. According to the need to use hydrochloric acid solution diluted to the appropriate concentration of standard working solution. Store in 4 °C refrigerator.

5 Instruments and equipment

5.1

fluorescence spectrophotometer.

5.2

Analysis of balance. 0.01 g and 0.0001 g.

5.3

Condensate tube.

5.4

Separate funnel. 125 mL.

5.5

Erlenmeyer flask. 100 mL, with a grinding mouth.

5.6

electric hot water bath.

5.7

volumetric flask. 10 mL, 100 mL, 1000 mL.

6.1 Preparation of the sample

The retrieved vials were thoroughly mixed and about 250 mL was taken as a sample. Into a clean container, sealed, marked mark.

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

6.2 Sample storage

Store the sample below -5 ° C.

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 Saponification

Weigh the sample 10 g (accurate to 0.1 g) in the conical flask, add 7 mL of potassium hydroxide solution, connected to the condenser, in the boiling water bath Reflux saponification 40 min, remove, fully cooled.

7.2 Extraction

The saponified solution was transferred to a separatory funnel and the Erlenmeyer flask was washed with 10 mL of water and the lotion was incorporated into the same separatory funnel. Add 15 mL of ethyl acetate Ester, gently shake 0.5 min, static stratification. The aqueous layer was transferred to another separatory funnel, extracted again with 15 mL of ethyl acetate, shaken vigorously for 1 min, Static stratification. The ethyl acetate extracts were combined.

7.3 Purification

The ethyl acetate extract was washed with 20 mL of potassium hydroxide solution, shaken vigorously for 1 min, and the layers were discarded and the aqueous layer was discarded. Add another 20 mL the potassium hydroxide solution was washed gently and the water was discarded. The ethyl acetate layer was extracted with 2 x 5 mL hydrochloric acid solution. The combined hydrochloric acid extract was added at 10 mL Volumetric flask, and with hydrochloric acid solution volume. Determination by fluorescence spectrophotometry.