



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

GB 23200.90-2016

Replacing SN/T 3156-2012

**Food safety national standard -- Determination of various
carbamate pesticide residues in milk and dairy products by
liquid chromatography-mass spectrometry**

乳及乳制品中多种氨基甲酸酯类农药残留量的测定 液相色谱-质谱法

(English Translation)

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Foreword

This standard replaces SN/T 3156-2012 "Determination of various carbamate pesticide residues in milk and dairy products Liquid Chromatography - String Combined mass spectrometry".

Compared with SN/T 3156-2012, the main changes are as follows.

- Standard text format is modified to national standard text format for food safety;
- "Other food can be referred to in the standard range";

This standard replaced the previous version of the standard release.

-SN/T 3156-2012.

National standards for food safety Determination of various carbamate pesticide residues in milk and dairy products Liquid chromatography - mass spectrometry

1 Scope

This standard provides milk and dairy products in the kill line Wei, methomyl, pirimicarb, aldicaril, speed off Granville, evil worms, grams of Budweiser, Determination of 14 kinds of carbamate pesticide residues by flow cytometry-mass spectrometry Detection method.

This standard applies to pure milk, yogurt, milk powder, cheese and fruit milk in the kill line Wei, Dudu Wei, pirimicarb, aldicarb, Determination and confirmation of residual residues of carbamazepine, carbofuran, furosemide, isoprocarb, methacillin, sec.

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 samples were extracted with acetonitrile, and the extract was purified by solid phase extraction column and eluted with methanol. The samples were detected and confirmed by liquid chromatography-tandem mass spectrometry.

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

acetonitrile (C₂H₃N). residual grade.

4.1.2

Methanol (CH₄O). Residue.

4.1.3

formic acid (CH₂O₂). excellent grade pure.

4.1.4

methanol - water solution (3 2, V/V). accurate measurement of 60 mL of methanol and 40 mL of water, mixed with spare.

4.1.5 Anhydrous sodium sulfate (Na₂SO₄). analysis of pure, by 650

OC burning 4 h, stored in a sealed container in reserve.

4.1.6 Sodium chloride (NaCl). Analytical purity.

4.2 Standards

4.2.1

Pesticide standard substances. kill line Wei, methomyl, pirimicarb, aldicarb, speed off Granville, evil worm Granville, grams of Budweiser, carnoside, Isoprocabamate, methamidium, Zhong Dingwei, the killing of Granville and a sulfur Granville, purity $\geq$ 98.5%. Refer to Appendix A for reference material information.

4.3 Standard solution preparation

4.3.1

standard stock solution. accurately weighed appropriate amount of various carbamate standard material, with methanol prepared into a concentration of 100 μ g/mL of the standard stock solution, -18 oC preservation.

4.3.2

mixed standard intermediate solution. respectively, accurately absorb the appropriate amount of carbamate pesticide standard stock solution, with methanol prepared into the concentration of 5 μ g/mL mixed standard intermediate solution, -18 oC stored.

4.3.3

matrix mixed standard working solution. according to the need to absorb the appropriate amount of mixed standard intermediate solution, with a blank sample matrix solution prepared into appropriate when the concentration of the standard working solution mixed, 0 ~ 4 oC refrigerator to save, use before the preparation.

4.4 Materials

4.4.1

Microporous membrane. 13 mm x 0.22 μ m, organic.

4.4.2 C18 solid phase extraction column. ENVI

TM-181) 1 000 mg, 6 mL, or equivalent.

5 Instruments and equipment

5.1

Liquid Chromatography - Tandem Mass Spectrometer. Equipped with an electrospray ion source (ESI).

5.2

Balance. 0.01 g and 0.0001 g.

5.3

Homogenizer.

5.4

Centrifuge. 4 000 r/min.

5.5

Rotary evaporator.

5.6

Polypropylene centrifuge tube. 50 mL.

5.7

Flasks. 100 mL.

6 Sample preservation

Pure milk, yogurt, fruit milk, cheese is equal to 0 ~ 4 oC preservation, milk powder sealed at room temperature.

7.1.1 Pure milk, yogurt, fruit milk, cheese and so on

Weigh the sample 5 g (accurate to 0.01 g) in 50 mL centrifuge tube, add 20 mL of acetonitrile and 5 g of anhydrous sodium sulfate, Quality extraction 1 min, adding 2 g sodium chloride, oscillation, centrifugation at 4 000 r/min 3 min. Take the supernatant and remove 10 mL of the residue Acetonitrile repeated extraction 1 times, combined