



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

GB 23200.86-2016

Replacing SN/T 3036-2011

**Food safety national standard -- Determination of residues of
organochlorine pesticides in milk and dairy products by gas
chromatography-mass spectrometry / mass spectrometry**

乳及乳制品中多种有机氯农药残留量的测定 气相色谱-质谱 质谱法

(English Translation)

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Foreword

This standard replaces SN/T 3036-2011 "Determination of residues of organochlorine pesticides in export milk and dairy products Gas chromatography-mass spectrometry /Mass spectrometry".

Compared with SN/T 3036-2011, the main changes are as follows.

- Standard text format is modified to national standard text format for food safety;
- the name and scope of the "export milk and dairy products" to "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 3036-2011.

National standards for food safety Determination of Residues of Organochlorine Pesticides in Milk and Dairy Products Gas chromatography - mass spectrometry/mass spectrometry

1 Scope

This standard specifies methods for the determination of multiple organochlorine pesticide residues in milk and dairy products by gas chromatography-mass spectrometry/mass spectrometry.

This standard applies to liquid milk, milk powder, yogurt (semi-solid), ice cream, milk and other milk and dairy products in a six six six, P, p'-DDT, o, p'-droplet, p, p'-droplet, o, P, p'-drop, Methotrexate, heptachlor, epoxidized heptachlor, aldrin, dieldrin, endrin, aldrin aldehydes, Trans-chlordane, chlorinated chlordane, alpha-endosulfan, beta-endosulfan, endosulfan sulfate, hexachlorobenzene, tetrachloronitrobenzene, pentachloronitrobenzene, pentachloroaniline, Methyl pentachlorophenyl sulfide, mirex and other 30 kinds of organochlorine pesticide residues in the determination and confirmation, 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 organochlorine pesticide residue in the sample was extracted with n-hexane-acetone (1 1, volume ratio) solution, and the extract was concentrated and gel permeated Chromatography and Florian silica column were determined by gas chromatography - mass spectrometry/mass spectrometry and quantified by external standard area method.

4 Reagents and materials

Unless otherwise specified, the reagents used are analytical grade and water is the primary water specified in GB/T 6682.

4.1 Reagents

4.1.1

n-hexane (C₆H₁₄). chromatographic purity.

4.1.2

Acetone (C₃H₆O). Chromatographic pure.

4.1.3

dichloromethane (C₂H₂Cl₂). chromatographic purity.

4.1.4

Cyclohexane (C₆H₁₂).

4.1.5

Ethyl acetate (C₄H₈O₂).

4.1.6

anhydrous sodium sulfate (Na₂SO₄). 650 °C burning 4 h, in the dryer to cool to room temperature, stored in a sealed bottle in reserve.

4.1.7

Sodium chloride (NaCl).

4.2 Solution preparation

4.2.1

Extract. Take appropriate n-hexane and acetone in a 1. 1 by volume ratio.

4.2.2

Gel permeation chromatography Eluent. Take appropriate amount of cyclohexane and ethyl acetate in a 1. 1 by volume ratio.

4.2.3

Solid Phase Extraction Eluent. Take appropriate n-hexane and dichloromethane in a volume ratio of 5.95.

4.3 Standards

4.3.1

Pesticide Reference Material. Purity >=95%.

4.4 Standard solution preparation

4.4.1

standard stock solution. accurately weighed the right amount of the standard material, with n-hexane prepared into a concentration of 100 µg/mL standard stock solution, the solution was stored at 0 ° C to 4 ° C in the dark.

4.4.2

standard intermediate solution. take appropriate amount of standard stock solution, prepared into a 2 µg/mL mixed standard working solution, the solution in the 0 °C ~ 4 °C dark preservation.

4.4.3

standard working solution. take appropriate amount of various standard stock solution, prepared into the appropriate concentration of mixed standard working solution. Standard work Liquid is now available.

4.5 Materials

4.5.1

Florisil Solid Phase Extraction Column. 1 g/6 mL, or equivalent, activated with 5 mL n-hexane prior to use.

4.5.2

Microporous membrane. 0.45 µm, organic.

5 Instruments and equipment

5.1

Gas Chromatography - Mass Spectrometry/Mass Spectrometer. Electronically Bombardive Source (EI).

5.2

Gel Permeation Chromatography.

5.3

Electronic balance. 0.01 g and 0.0001 g.

5.4

Vortex Mixer.

5.5

Centrifuge. Maximum speed up to 5 000 r/min.