



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

GB 23200.32-2016

Replacing SN/T 1989-2007

**Food safety national standard -- Determination of butyryl
hydrazine residues in foodstuffs by gas chromatography-mass
spectrometry**

食品中丁酰肼残留量的测定 气相色谱-质谱法

(English Translation)

Issued on: December 18, 2016

Implemented on: June 18, 2017

**Issued by: National Health and Family Planning Commission of the
People 's Republic of China**

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Foreword

This standard replaces SN/T 1989-2007 "Determination of Butyryl Hydrazide Residue in Import and Export Food by Gas Chromatography-Mass Spectrometry".

Compared with SN/T 1989-2007, the main changes are as follows.

- Standard text format is modified to national standard text format for food safety;
- the name of the "export food" to "food";
- increase the "other food reference implementation" in the standard range.

This standard replaced the previous version of the standard release.

-SN/T 1989-2007.

National standards for food safety Determination of butyryl hydrazine residues in foodstuffs by gas chromatography - mass spectrometry

1 Scope

This standard specifies the method for the determination of butyryl hydrazide residues in food by gas chromatography-mass spectrometry.

This standard applies to the determination of the residual amount of butyryl hydrazide in peanuts, rice, soybeans, parsley, apples, fish, chicken, tea and honey.

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 unreacted hydrazide residue in the sample was extracted with water, purified by steam distillation, salicylaldehyde derivatization, silica gel solid phase extraction column, and purified by gas chromatography - mass spectrometer for determination, external standard method quantitative.

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

n-hexane (C₆H₁₄). chromatographic purity.

4.1.2

Sodium hydroxide (NaOH).

4.1.3

Sodium chloride (NaCl).

4.1.4

glacial acetic acid (C₂H₄O₂).

4.1.5

Salicylaldehyde (C₇H₆O₂).

4.1.6

Ethyl acetate (C₄H₈O₂). Chromatographic purity.

4.2 Solution preparation

4.2.1

Ethyl acetate n-hexane solution (3 97, V/V). Take 30 mL of ethyl acetate, add 970 mL of n-hexane and shake well.

4.2.2

acetic acid solution (10%, v/v). Remove 10 mL of glacial acetic acid by adding 90 mL of water and mix well.

4.2.3

Sodium hydroxide solution (50%, m/v). Weigh 500 g of sodium hydroxide gradually dissolved in 1 L of water, cooled and mixed thoroughly.

4.3 Standards

4.3.1 1,1-dimethyl hydrazine, C₂H₈N₂, CAS. 57-14-7). purity $\geq$ 98%.

4.4 Standard solution preparation

4.4.1 1,1-dimethyl hydrazine reserve solution. accurately weighed the appropriate amount of 1,1-dimethyl hydrazine standard 0.1 g (accurate to 0.1 mg), Was added to a small beaker containing 2 mL of acetic acid solution and transferred to 1 L brown volumetric flask with a standard concentration of 100 μ g/mL.

Preparation of liquid. The standard stock solution is diluted with water to the standard working solution at the appropriate concentration. The stock solution was stored at 4 ° C in the dark. each 6 months to prepare once.

5 Instruments and equipment

5.1

Gas chromatograph with mass selective detector.

5.2

Analysis of balance. 0.01 g and 0.0001 g.

5.3

steam distillation unit.

5.4

Centrifuge. 5 000 r/min.

5.5

Rotary Evaporator.

5.6

Oscillator.

5.7

Ultrasonic instrument.

5.8

Solid phase extraction column. silica gel, 6 mL, 1 000 mg, or equivalent.

5.9

Erlenmeyer flask. 250 mL.

5.10

plug test tube. 25 mL.

6.1.1 Fruits and vegetables

Take apples, parsley and other representative samples of about 500 g, chopped, with a crusher to sample processed into a slurry. Mix well into a clean capacity Inside, sealed and marked. -18 °C below the preservation.

6.1.2 Grains and nuts

Take about 500 g of representative samples of rice, soybeans, peanuts, etc., crushed with a pulverizer and passed through a 20 mesh round hole. Mix well into the clean Net inside the container, sealed and marked. 4 °C below the preservation.