



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

GB 23200.104-2016

Replacing SN/T 1392-2004

**Food safety national standard -- Meat and meat products --
Determination of 2-methyl-4-chloro and 2-methyl-4-chlorobutyric acid
residues by liquid chromatography-mass spectrometry**

肉及肉制品中2甲4氯及2甲4氯丁酸残留量的测定 液相色谱-质谱法

(English Translation)

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Foreword

This standard replaces SN/T 1392-2015 "Method for the determination of 2-methyl-4-chloro and 2-chlorobutyric acid residues in meat and meat products for liquid chromatography /Tandem mass spectrometry".

This standard compared with SN/T 1392-2015, the main changes are as follows.

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

This standard replaced the previous version of the standard release.

-SN/T 1392-2004;

-SN/T 1392-2015.

National standards for food safety Determination of 2 - 4 - chloro and 2 - 4 - chlorobutyric acid residues in meat and meat products Liquid chromatography - mass spectrometry

1 Scope

This standard specifies the method for the determination of 2-methylchloride and 2-methylchlorobutyric acid residues in meat and meat products by liquid chromatography-mass spectrometry.

This standard applies to the export of frozen beef, fish, pork, chicken, beef canned 2 a 4 chlorine and 2 a 4 chlorobutyric acid residues Detection, 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

Under acidic conditions, the residual 2-methyl-4-chloro, 2-methyl-4-chlorobutyric acid in the sample was extracted with methylene chloride, and the extract was replaced with solvent.

Chromatography - mass spectrometry - 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

dichloromethane (CH_2Cl_2). chromatographic purity.

4.1.2

Methanol (CH₃OH). Chromatographic Purification.

4.1.3

Formic acid (HCOOH). Chromatographic pure.

4.1.4

concentrated sulfuric acid (H₂SO₄). excellent grade pure.

4.1.5

sodium chloride. excellent grade pure.

4.2 Solution preparation

4.2.1

Sulfuric acid - water (19, V/V). take 100 mL concentrated sulfuric acid, add 900mL of water, shake back.

4.2.2 0.1% formic acid aqueous solution. take 1 mL of formic acid, the volume of water to 1000 mL.

4.3 Standards

2 a 4 chlorobutyric acid standard. purity $\geq$ 99%, CAS. 94-82-6.

4.4 Standard solution preparation

4.4.1 0.1 mg/mL standard stock solution. accurately weighed the appropriate amount of 2 a 4 chlorine, 2 a 4 chlorobutyric acid standard, with methanol prepared into the concentration Prepare the solution for 0.1 mg/mL standard. Store in 4 °C refrigerator.

4.4.2

mixed standard working fluid. the use of the corresponding matrix solution prepared with the appropriate concentration of the standard working solution, stored in 4 °C refrigerator.

5 Instruments and equipment

5.1

Ultra High Pressure Liquid Chromatography - Tandem Mass Spectrometer with Electrospray Ion Source (ESI).

5.2

high-speed homogenizer. not less than 12000 r/min.

5.3

Centrifuge. not less than 4000 r/min.

5.4

Analytical balance. 0.01 g and 0.0001 g.

5.5

nitrogen blowing instrument.

5.6

Scroll Mixer.

6.1 Preparation of the sample

Take a representative sample 500g, sample sampling site according to GB 2763 Appendix A implementation, crushing machine, and then ground into fine powder Shaped, packed into a clean glass container, sealed and marked.

6.2 Sample storage

The samples were cryopreserved at -18 ° C.

7.1 Extraction and purification

Weigh the sample 4 g (accurate to 0.01 g) in a 50 mL centrifuge tube, add 3 mL of methanol, 1 mL of sulfuric acid-water, 2 g of sodium chloride and 20 mL Dichloromethane. In the high-speed homogenizer on the homogeneous extraction 2 min, centrifugation, take the supernatant in another centrifuge tube, standing, take the next layer of dichloromethane Layer 10 mL. Repeat the extraction once, merge the two extracts, at 40°C nitrogen blowing near dry, with methanol volume to 2 mL, vortex, membrane, for Determination of Ultra - High Pressure Liquid Chromatography by Tandem Mass Spectrometry.

7.2.1 Liquid Chromatographic Reference Conditions

- A) Column. C18, 1.8 μm, 2.1 x 50 mm or equivalent;
- B) 0.1% formic acid aqueous solution of methanol, gradient elution see Table 1;
- C) Flow rate. 0.5 mL/min;
- D) Injection volume. 5 μL;
- E) Column temperature. room temperature;

Table 1 Liquid Chromatography Gradient Elution Procedure

Time (min) 0.1% Formic acid Aqueous solution (%) Methanol (%)