



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

GB 23200.81-2016

Replacing SN 0594-1996

**Food safety national standard -- Method for Detecting the
Residue of West Majin in Meat and Meat Products**

肉及肉制品中西玛津残留量的检测方法

(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 0594-1996 "Test Method for Residues of West Majin in Exported Meat and Meat Products".

Compared with SN/T 0594-1996, 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 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 0594-1996.

National standards for food safety Method for Detecting the Residue of West Majin in Meat and Meat Products

1 Scope

This standard specifies the method for the determination of mesazepam residues in meat and meat products by gas chromatography.

This standard applies to the test of residual residues of beef in the beef, 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 file. 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 sample was extracted with a dichloromethane-methanol mixed solution and the extract was purified by a Florisil column using gas chromatography with a nitrogen and phosphorus detector Instrument 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

anhydrous sodium sulfate (Na_2SO_4). 650 °C burning 4 h, set the dryer in the spare.

4.1.2

Florisil. for chromatography, 100-200 mesh, at 650 °C for 4 h, before drying at 130 °C for 5 h, after cooling stored in dense Closed container, spare.

4.2 Solution preparation

4.2.1

Acetone-petroleum ether mixed solution (15 85). Take 150 mL of acetone, add 850 mL of petroleum ether and shake well.

4.2.2

Dichloromethane - methanol mixed solution (9 1). take 900 mL of dichloromethane, add 100 mL of methanol, shake back.

4.3 Standards

4.3.1

Xi Ma Jin standard. purity $\geq 98\%$.

4.4 Standard solution preparation

4.4.1

Simaxin standard solution. accurately weighed the appropriate amount of Simazine standard, with acetone prepared into 100 $\mu\text{g/mL}$ standard stock solution, according to Need to re-use acetone diluted to the appropriate concentration of standard working solution.

5 Instruments and equipment

5.1

gas chromatograph, with nitrogen and phosphorus detector.

5.2

Analysis of balance. 0.01 g and 0.0001 g.

5.3

Rotary Evaporator.

5.4

Florisil column. glass column, 40 cm horizontal 1.5 cm (inner diameter), top-down filling 2 cm high anhydrous sodium sulfate, 10 g Florisil, 2 cm high anhydrous sodium sulfate. Pre-leaching with acetone-petroleum ether mixture (15 85) before use.

5.5

Ultrasonic water bath.

6.1 Preparation of the sample

A sample of about 1 kg was taken from all the samples taken. The sample was sampled according to GB 2763 Appendix A, Mix, are divided into two, respectively, into the clean container as a sample. Sealed and marked.

6.2 Sample storage

The sample was stored at -18 ° C or lower.

During sample and sample preparation, the sample should be protected from contamination or changes in the residue content.

7.1 Extraction

Weigh the sample about 30 g (accurate to 0.01 g). Add 30 g of anhydrous sodium sulfate, ground into powder, into the conical flask. Add 100 mL Dichloromethane - methanol (9 1) mixture, ultrasonic oscillation 20 min. Filtrate with a cloth funnel to retain the filtrate. The residue was then washed with 100 mL of dichloromethane Alkane-methanol (9 1) mixture, as described above. The filtrate was combined in a vial of rotary evaporator. And dried in a 50 ° C water bath under reduced pressure dry.

7.2 Purification

The residue was dissolved three times with 20 mL of acetone-petroleum ether (15 85) and the solution was poured into a florisil column. With 100 mL Acetone - petroleum ether (15 85), controlled flow rate of 2-3 drops per second, and the effluent was received in an evaporating flask of rotary evaporative liquid. Water at 60 ° C Bath in the bath to dry. With acetone to 5.0 mL, for GC determination.

7.3.1 Gas Chromatographic Reference Conditions

- A) Column. HP-1.5 mx0.53 mm (id), film thickness 2.65 m.
- B) Column temperature. 155 ° C.
- C) Inlet temperature. 240 ° C.
- D) Detector temperature. 250 ° C.
- E) Carrier gas. nitrogen, purity greater than or equal 99.99%, 40 mL/min.
- F) Hydrogen. 2 mL/min.
- G) air. 140 mL/min
- H) Injection volume. 2 µL.