



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

GB 23200.82-2016

Replacing SN 0705-1997

**Food safety national standard -- Determination of ethephon
residues in meat and meat products**

肉及肉制品中乙烯利残留量的检测方法

(English Translation)

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Foreword

This standard replaces SN 0705-1997 "Method for the detection of ethephon residues in meat and meat products for import and export".

This standard compared with SN 0705-1997, 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 0705-1997.

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National standards for food safety Determination of ethephon residues in meat and meat products

1 Scope

This standard specifies sample preparation and gas chromatographic methods for the detection of ethephon residues in meat and meat products.

This standard applies to pork, beef, chicken and other residues in the detection of ethephon, 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 ethephon residue in the sample is extracted with methanol and the extract is frozen to remove the fat and wax, and then concentrated, and the sample is extracted with ether and derivatized by diazomethane, the ethephon derivative derived from dimethyl ethephon, with a nitrogen and phosphorus detector with gas chromatography, 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

Potassium hydroxide (KOH). Analytical purity.

4.1.2

Methanol (CH₃OH). Distillation.

4.1.3

N-methyl-N-nitroso-p-toluenesulfonamide (C₈H₁₀N₂O₃S).

4.1.4

anhydrous ethanol (CH₃CH₂OH).

4.1.5

anhydrous ether. (C₄H₁₀O). re-distillation.

4.1.6

anhydrous sodium sulfate (Na₂SO₄). 650 °C burning 4 h, placed in a sealed container in reserve.

4.2 Solution preparation

4.2.1

Methanol - Hydrochloric acid (38%) solution (90 10).

4.2.2

Diazomethane solution. 10 ml of aqueous solution of potassium hydroxide (0.6 g/mL), 35 mL of ethanol and 10 mL of ether the double mouth distilled bottle is placed in a water bath on a magnetic stirrer heating plate. Place the stir bar in the bottle, connect the dropping funnel and the high-performance condenser, and condense it After the series, two 125 mL flasks were used as receiving bottles. 10 mL of ether was placed in the second flask and the inlet tube was inserted into the ether level the following. Place the two receiving bottles in an ice bath. The water bath was heated to 70 ° C while stirring with a magnetic stirrer and dropping from the dropping funnel A solution of N-methyl-N-nitroso-p-toluenesulfonamide in diethyl ether (21.5 g/140 mL) was added. The time to complete the total solution was controlled at 20 min , When the distillate was pale yellow, stop the distillation. The liquids in the two receiving bottles were combined as a diazomethane solution. If it contains impurities too can be re-distilled. The solution is stored in a refrigerator and stored for one month.

4.4 Standard solution preparation

4.4.1

ethephon standard solution. accurately weighed the right amount of ethephon standard, with methanol prepared into a concentration of 1.0 mg/mL standard reserve Liquid, dilute the standard stock solution with methanol to prepare the standard working solution at the appropriate concentration. Note. should be used when preparing standard solutions

Polyethyleneware.

5 Instruments and equipment

5.1

Gas chromatography with nitrogen and phosphorus detector.

5.2

Analysis of balance. 0.01 g and 0.0001 g.

5.3

Quick Mixer.

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5.4

Centrifuge. 6000 r/min.

5.5

Polyethylene flask. 100 mL with stopper.

5.6

Polyethylene beaker. 250 mL.

5.7

Microinjector. 10 μ L or 5 μ L.

5.8

Ultrasonic bath.

6.1 Extraction

Weigh about 20 g of sample (accurate to 0.1 g) in a polyethylene beaker by adding 0.5 mL of methanol-hydrochloric acid (90 10) solution and 40 mL of methanol, Extraction in an ultrasonic bath for 5 min. After centrifugation in a centrifuge tube (6000 r/min) for 2 min, the supernatant was aspirated with 100 mL of polyethylene in a polyethylene flask. The residue was again extracted with 40 mL of methanol as described above and the combined extracts were pooled in the same polyethylene flask.

6.2 Purification

The polyethylene flask containing the methanol extract was frozen in a freezer at -18 ° C for 4 h. Removed quickly after the transfer into the centrifuge tube Heart (6000 r/min) 5 min. The supernatant was poured into a 250 mL heart flask treated with dimethyldichlorosilane. The remaining residue was washed with 2 mL Frozen methanol washed, the lotion