



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

GB 23200.84-2016

Replacing SN/T 0529-2013

**Food safety national standard -- Determination of Methanol --
DDT Residues in Meat by Gas Chromatography -- Mass
Spectrometry**

肉品中甲氧滴滴涕残留量的测定 气相色谱-质谱法

(English Translation)

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Foreword

This standard replaces SN/T 0529-2013 "Method for the determination of methotrexate residues in exported meat by gas chromatography/mass spectrometry".

Compared with SN/T 0529-2013, the main changes are as follows.

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

This standard replaced the previous version of the standard release.

-SN 0529-1996;

-SN/T 0529-2013.

National standards for food safety Determination of Methanol - DDT Residues in Meat by Gas Chromatography - Mass Spectrometry

1 Scope

This standard specifies the method of gas chromatography-mass spectrometry for the residual amount of methoxylated DDT in meat.

This standard applies to chicken, duck, pork, the determination of residual ozone droplets, other food can refer to the implementation of other food can participate According 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 samples were extracted with ethyl acetate-cyclohexane (1 1, volume ratio). The extract was concentrated by gel permeation chromatography (GPC) the system was purified and eluted with ethyl acetate-cyclohexane (1 1, by volume). The eluate was concentrated to dryness and constant volume. The gas chromatography-mass spectrometer Ion detection, external standard method.

4 Reagents and materials

Unless otherwise stated, all reagents are pesticide residues and water is a Class I water specified in GB/T 6682.

4.1 Reagents

4.1.1

Ethyl acetate (C₄H₈O₂).

4.1.2

Cyclohexane (C₆H₁₂).

4.1.3

n-hexane (C₆H₁₄).

4.1.4 Anhydrous sodium sulfate (Na₂SO₄). analytical grade, used before the 650

C burning 4 h, stored in a dryer, cooling and standby.

4.2 Solution preparation

4.2.1

Ethyl acetate - cyclohexane solution (1:1, volume ratio). take 200 mL of ethyl acetate, add 200 mL of cyclohexane, shake the standby.

4.3.1 Methoxychloride, C₁₆H₁₅Cl₃O₂, relative molecular weight. 345.66, CAS. 72-43-5). pure

Degrees $\geq$ 99%.

4.4 Standard solution preparation

4.4.1

Methanol DDT standard stock solution. accurately weighed the appropriate amount of methanoxazole standard, with n-hexane prepared into 100 μ g/mL Standard storage solution, dark in 0 $^{\circ}$ C ~ 4 $^{\circ}$ C preservation, shelf life of 6 months.

4.4.2

Methanol Drops Standard Working Solution. According to the testing requirements, absorb the appropriate amount of the above standard solution in the volumetric flask, Alkaline Dilute the standard working solution with appropriate mass concentration and keep it at 0 $^{\circ}$ C ~ 4 $^{\circ}$ C for 1 month.

5 Instruments and equipment

5.1

Gas Chromatography - Mass Spectrometer. Equipped with electron impact source (EI).

5.2

Tissue crusher.

5.3

Homogenizer.

5.4

Rotary Evaporator.

5.5

Analytical balance. 0.01 g and 0.0001 g.

5.6

Centrifuge.

5.7

Gel Permeation Chromatography System (GPC). A purification column containing a Biobeads S-X3 filler.

5.8

Scroll Mixer.

6.1 Preparation of the sample

Remove some of the representative samples from the original sample, the sampling site according to GB 2763 Appendix A implementation, into the high-speed tissue crusher Broken evenly. Fully mixed, with a four-point split out of not less than 500 g as a sample. Into a clean container, marked with a mark after sealing.

6.2 Sample storage

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

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

7.1 Extraction

Weigh 10 g (accurate to 0.01 g) sample in a 100 mL stoppered centrifuge tube, add 20 g of anhydrous sodium sulfate, add ethyl acetate-Cyclohexane (1 1, volume ratio) 40 mL, homogeneous 2 min, 4000 r/min centrifugation 5 min, the supernatant through the anhydrous sodium sulfate with a funnel, Collected in 100 mL rotary vials, the residue and then add 20 mL of ethyl acetate - cyclohexane (1 1, volume ratio), according to the above steps to extract a the combined extracts were concentrated to near dryness by rotary evaporation at 40 ° C.