



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

GB 23200.27-2016

Replacing SN/T 1734-2006

**Food safety national standard -- Determination of
4,6-dinitro-o-cresol Residue in Fruits by Gas Chromatography --
Mass Spectrometry**

水果中4, 6-二硝基邻甲酚残留量的测定 气相色谱-质谱法

(English Translation)

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Foreword

This standard replaces SN/T 1734-2006 "Test Method for Residue of 4,6-Dinitro-o-cresol in Import and Export Fruits Gas Chromatography-Mass Spectrometry law".

Compared with SN/T 1734-2006, the main changes are as follows.

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

This standard replaced the previous version of the standard release.

-SN/T 1734-2006.

National standards for food safety Determination of 4,6 - dinitro - o - cresol Residue in Fruits by Gas Chromatography - Mass Spectrometry

1 Scope

This standard specifies the method for the determination of 4,6-dinitro-o-cresol residues in fruit by gas chromatography-mass spectrometry.

This standard applies to the apple, pear 4,6 6-dinitro-o-cresol residue test, 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

Fruit sample 4,6-dinitro-o-cresol residue extracted with dichloromethane, blowing nitrogen to dry after acetonitrile dissolved, with N- (p-butyl dimethyl (MTBSTFA), which was determined by gas chromatography-mass spectrometry and quantified by 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). excellent grade pure.

4.1.2

acetonitrile (CH_3CN). pure chromatography.

4.1.3

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

4.1.4

Derivative reagent N- (tert-butyldimethylsilyl) -N-methyltrifluoroethylphthalide (MTBSTFA). 99% purity.

4.2 Standards

4.2.1 4,6-dinitro-o-cresol standard (C₇H₆N₂O₅, CAS No .. 534-52-1). purity 99%.

4.3 Standard solution preparation

4.3.1 4,6-dinitro-o-cresol standard stock solution. accurately weighed 10 mg 6,6-dinitro-o-cresol standard, dissolved in acetonitrile and prepared Into the concentration of 100 rmg/L of the standard stock solution, can be stored in the refrigerator 0 °C ~ 4 °C for 3 months.

4.3.2 4,6-dinitro-o-cresol standard working solution. according to the need for acetonitrile will be 4,6-dinitro-o-cresol standard stock solution diluted to the appropriate concentration Degree of standard working fluid, can be stored in the refrigerator 0 °C ~ 4 °C for 1 month.

5 Instruments and equipment

5.1

Gas Chromatography - Mass Spectrometer, with electron bombardment source.

5.2

Analysis of balance. 0.0001 g and 0.01 g.

5.3

Crusher.

5.4

Centrifuge..2000 r/min.

5.5

Scroll Mixer.

5.6

blowing nitrogen enrichment instrument.

5.7

incubator.

5.8

Polypropylene centrifuge tube. 15 mL, with plug.

6.1 Preparation of the sample

Replace the sample of about 500 g, the sampling site according to GB 2763 Appendix A implementation, (not available washing) chopped, with a crusher will sample the product is processed into a slurry, mixed, packed into a clean container, sealed and marked.

6.2 Sample storage

Samples stored at -5 ° C in the dark. During sample preparation, the sample should be protected from contamination or changes in the residue content.

7.1 Extraction

Weigh 1 g (accurate to 0.01 g) of the prepared homogeneous sample in a 15 mL centrifuge tube, add 3 g of anhydrous sodium sulfate and 5 mL of dichloromethane, Mix thoroughly for 3 min on a vortex mixer and centrifuge at 2 000 r/min for 5 min. Transfer the dichloromethane to another 15 mL centrifuge tube, with 5 mL of methylene chloride repeated extraction time, combined extract, 50 °C nitrogen blowing concentrated to near dry.

7.2 Derivatization

After drying in nitrogen, add 0.4 mL of acetonitrile to the centrifuge tube and add 100 µL of MTBSTFA, sealed at 80 ° C Derived 1 h. Standard working fluid also added 100 µL MTBSTFA, sealed, under the above conditions for derivatization. After derivatization 0.22 µm microporous membrane was determined by gas chromatography-mass spectrometry.

7.3.1 Gas Chromatography - Mass Spectrometry Reference Conditions

- A) Column. HP-5MS quartz capillary column. 30 m x 0.25 mm (inner diameter) x 0.25 µm film thickness, or equivalent.
- B) Column temperature. 100 ° C (1 min) 30 ° C/min 180 ° C (1 min) 50 ° C/min 205 ° C 10 ° C/min 260 ° C (20 min).
- C) Inlet temperature. 210 ° C.
- D) Transmission line temperature. 250 ° C.
- E) Carrier gas. helium, purity 99.999%, flow rate 1.0 mL/min.
- F) Injection volume. 1 µL.
- G) Injection method. no split injection, 1 min, open the diverter valve, split ratio of 20. 1.
- H) ionization mode. EI.
- I) Ionization energy. 70 eV.