



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

GB 23200.2-2016

Replacing SN/T 1737.2-2007

**Food safety national standard -- Methods for the determination of
herbicide residues -- Part 2: Determination of diphenyl ether herbicides
in grain and oilseeds by gas chromatography-mass spectrometry**

除草剂残留量检验方法 第2部分：气相色谱-质谱法测定 粮谷及油籽中二苯醚类除
草剂残留量

(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 1737.2-2007 "Methods for the determination of herbicide residues Part 2. Determination of grain and oil by gas chromatography/mass spectrometry the amount of diphenyl ether herbicide residues in seeds".

Compared with SN/T 1737.2-2007, the main changes are as follows.

- Standard text format is modified to national standard text format for food safety;
- increase the "other food reference implementation" in the standard range.

This standard replaced the previous version of the standard release.

-SN/T 1737.2-2007.

National standards for food safety Method for determination of herbicide residue Part 2.
Determination of diphenyl ethers in grain and oilseeds by gas chromatography - mass spectrometry Herbs Residue

1 Scope

This standard specifies the grain and oil seed in the ginger grass, methicillin ether, benzoyl ether, methylprednisolide, triflufen grass, Lecium grass Determination of 9 Diphenyl Ether Herbicide Residues by Gas Chromatography/Mass Spectrometry.

This standard applies to rice and soybeans in the nine kinds of diphenyl ether herbicide residues in the detection and confirmation, 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

The samples were extracted with acetonitrile, cleaned up by Florisil silica solid phase extraction column, gas chromatograph - mass spectrometer and 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

Acetone (CH_3COCH_3). Chromatographic pure.

4.1.2

acetonitrile (CH_3CN). excellent grade pure.

4.1.3

n-hexane (C_6H_{14}). chromatographic purity.

4.1.4

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

4.2 Standards

4.2.1

Diphenyl Ether Herbicide Standards. See Appendix A.

4.3 Standard solution preparation

4.3.1

standard reserve solution. accurately weighed the right amount of benzoin ether, methylprednisolone ether, trifluoro-nitrofen, Lactobenzene, Herbicide, B Oxyfloxacin, Fluorofenoxylate, respectively, dissolved in acetone and fixed capacity to brown volumetric flask, the concentration is equivalent to 1 000 mg/L, stock solution storage Below -18 ° C.

4.3.2

mixed standard intermediate solution. accurate absorption of appropriate amount of benzoin, methylprednisolone, triflufen grass, lactoferrin, herbicidal ether, Ethoxifluorfen, Efloxacillin standard stock solution in brown volumetric flask, fixed with acetone to the scale, a variety of diphenyl ether herbicides concentrated the degree of 10 mg/L, the intermediate solution stored below -18 °C.

4.3.3

Mixed standard working solution. Mix the standard intermediate solution with acetone as needed and dilute it with other diphenyl ether herbicides and other concentrations of cyclodextrin and methoxylated ether, with nine kinds of diphenyl ether herbicides mixed standard working solution, mixed standard work the solution is stored at 0 to 4 ° C.

4.4 Materials

4.4.1

Florisil Solid Phase Extraction Column. 500 mg, 3 mL.

5 Instruments and equipment

5.1

Gas Chromatography - Mass Spectrometer, with electron bombardment source.

5.2

Analysis of balance. 0.01 g and 0.0001 g. 5.3 Centrifuge.

5.4

blowing nitrogen enrichment instrument.

5.5

Solid phase extraction device.

5.6

Scroll Mixer.

6 Preparation and storage of samples

Respectively, representative of the rice and soy samples of about 500 g, the sample sampling site according to GB 2763 Appendix A implementation, full crushing, mixing, Into a clean container, sealed and marked with, stored at 4 °C cold storage.

7.1 Extraction

Weigh 5 g (accurate to 0.01 g) sample in 50 mL centrifuge tube, add 2 mL of water and 20 mL of acetonitrile, then add 2 g of anhydrous sodium sulfate, Mixed in a vortex mixer for 3 min, centrifuged at 5000 r/min for 5 min, transferred to 10 mL of acetonitrile in the upper layer to another 15 mL centrifuge tube.

40 ° C water bath blowing nitrogen to dry, with n-hexane volume to 1 mL.

7.2 Purification

A 1 cm high anhydrous sodium sulphate was filled above the Fryborough Silica Solid Phase Extraction Column packing, pre-leached with 6 mL of acetone and 6 mL of n-hexane, respectively.

The extract was transferred to a solid phase extraction column, the column was discarded at a rate of 1 d/s, the effluent was discarded, and 5 mL of n-hexane-acetone (60 40, V/V) Followed by elution at a flow rate of 1 d/s. The eluate was collected in a 15 mL centrifuge tube and concentrated in a 40 ° C water bath to dryness, dissolved with n-hexane and Constant volume to 1.0 mL, gas chromatographic - mass spectrometry.

7.3.1 Gas Chromatography - Mass Spectrometry Reference Conditions

A) Column. HP-5MSI quartz capillary column. 30 m x 0.25 mm (inner diameter), film thickness 0.25 µm, or equivalent.

B) Column temperature. 70 ° C (2 min) 50 ° C/min 180 ° C 10 ° C/min. 200 ° C 2 ° C/min 220 ° C 10 ° C/min 280 ° C (10 min).

C) Inlet temperature. 270 ° C.