



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

GB 23200.9-2016

Replacing GB/T 19649-2006

**Food safety national standard -- Determination of residues of
475 pesticides and related chemicals in grain -- Gas
chromatographic-mass spectrometry**

粮谷中475种农药及相关化学品残留量的测定 气相色谱-质谱法

(English Translation)

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Foreword

This standard replaces GB/T 19649-2006 "Determination of 475 pesticides and related chemical residues in grain" Gas chromatography-mass spectrometry " this standard compared with GB/T 19649-2006, 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This standard has been replaced by the previous version of the release.

-GB/T 19649-2006.

National standards for food safety Determination of 475 pesticides and related chemical residues in grain Gas chromatography - mass spectrometry

1 Scope

This standard specifies the remaining amount of 475 pesticides and related chemicals (see Appendix A) for barley, wheat, oat, rice and maize.

Determination of Chromatography - Mass Spectrometry.

This standard applies to the determination of 475 pesticides and related chemical residues

in barley, wheat, oats, rice and maize. Other grain can refer to the implementation of 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 sample was extracted with acetonitrile in an accelerated solvent extraction instrument. The extract was purified by solid phase extraction column and washed with acetonitrile-toluene solution (3 1) De-pesticide and related chemicals, with gas chromatography - mass spectrometry.

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

Acetonitrile (CH₃CN, 75-05-8). Chromatographic Purification.

4.1.2

diatomaceous earth (SiO₂, 61790-53-2). excellent grade pure.

4.1.3

anhydrous sodium sulfate (Na₂SO₄, 7757-82-6). analytical grade. Before burning at 650 °C for 4h, stored in a dryer, cooling and standby.

4.1.4

toluene C₇H₈, 108-88-3). excellent grade pure.

4.1.5

Acetone (CH₃COCH₃, 67-64-1). Analytical pure, re-distilled.

4.1.6

dichloromethane (CH₂Cl₂, 75-09-2). chromatographic purity.

4.2 Standards

Pesticide and Related Chemical Reference Material. Purity $\geq 95\%$, see Appendix A.

4.3.1 Standard stock solution

Accurately weighed 5mg ~ 10mg (accurate to 0.1mg) pesticides and related chemicals in the standard 10mL volumetric flask, according to the standard the solubility of the quasi-material and the determination of the need to select toluene, - acetone mixture, dichloromethane and other solvents dissolved and constant volume to the scale (solvent selection See Appendix A).

4.3.2 Mixing standard solutions (mixed standard solutions A, B, C, D and E)

According to the nature of pesticides and related chemicals and retention time, will be 475 kinds of pesticides and related chemicals into A, B, C, D, E five and the concentration of each pesticide and associated chemical in the instrument to determine the concentration in the mixed standard solution. This is the standard 475 groups of pesticides and related chemicals and their mixed standard solution concentrations are given in Appendix A, Depending on the group number of each pesticide and associated chemical, the concentration of the standard solution and the concentration of the standard stock solution, Pesticide and related chemicals Standard stock solution (4.3.1) in a 100 mL volumetric flask, fixed to the mark with toluene. Mixed standard solution to avoid Light 4 °C preservation, shelf life of one month.

4.3.3 Internal standard solution

Accurately weighed 3.5mg epoxy heptachlor in 100mL volumetric flask, with toluene volume to the scale.

4.3.4 Matrix mixed standard working solution

A, B, C, D, E group of pesticides and related chemical matrix mixed standard working solution is 40 μ L standard solution (4.3.3) and a certain Volume of the mixed standard solution were added to 1.0 mL of the sample blank matrix extract, mix, dubbed the matrix mixed standard working solution A, B, C, D and E. Matrix mixed standard working solution should be used with the current distribution.

4.4 Materials

4.5.1 Envi-18 Column 1). 12 mL, 2 g or equivalent.

4.5.2 Envi-Carb Column 1). 6 mL, 0.5 g or equivalent.

4.5.3 Sep-Pak NH₂ column

2). 3 mL, 0.5 g or equivalent.

5 Instruments and equipment

5.1

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

5.2

Analysis of balance. 0.01 g and 0.0001 g.

5.3

Accelerated solvent extraction. equipped with 34 mL extraction tank.

5.4

nitrogen dryers.

5.5

Pear bottles..200mL.

5.6

pipettes. 1 mL.

6.1 Preparation of the sample

Grain samples taken according to GB 5491 were crushed by pulverizer. All samples were passed through a standard screen of 425 μ m, and the samples were prepared.

Divided into two, packed into a clean sample container, sealed and marked.

7.1 Extraction

Weigh 10 g of sample (accurate to 0.01 g) and mix with 10 g of diatomaceous earth and move into a 34 mL extraction cell of an accelerated solvent extractor.

10.34MPa pressure, 80 °C under the conditions of heating 5min, with acetonitrile static extraction 3min, cycle 2 times, and then use the pool volume of 60% of the B Nitrile (20.4 mL) was rinsed and purged with nitrogen for 100 seconds. After extraction, the extract was mixed and the samples with little oil content were mixed Take a half of the volume of the extract (equivalent to 5 g of the sample), and take a quarter of the volume of the extract from the larger oil sample (equivalent to 2.5g sample volume), to be purified.