



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

GB 23200.10-2016

Replacing GB/T 23200-2008

**Food safety national standard -- Determination of 488 pesticides and
related chemicals residues in mulberry, honeysuckle, medlar and lotus
leaf by gas chromatography-mass spectrometry**

桑枝、金银花、枸杞子和荷叶中488种农药及相关化学品残留量的测定 气相色谱-
质谱法

(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**

Table of Contents

Preface

1 Scope

2 Normative reference document

Principle

4 Reagents and materials

5 Instruments and equipment

6 Preparation and storage of sample

7 Analysis Step

Calculation and presentation of results

9 Precision

10% limit and recovery rate

Appendix A (informative) 488 pesticides and related chemicals Chinese and English names, methods Quantitative limits, grouping, solvent selection

Foreword

This standard replaces GB/T 23200-2008 "residues of 488 pesticides and related chemicals in mulberry, honeysuckle, medlar and lotus leaf Determination of gas chromatography - mass spectrometry".

This standard compared with GB/T 23200-2008, 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.

-GB/T 23200-2008.

National standards for food safety 818 kinds of pesticides and related chemical residues in mulberry, honeysuckle, medlar and lotus leaf Determination of gas chromatography - mass spectrometry

1 Scope

This standard specifies the residues of 488 pesticides and related chemicals (see Appendix A and Appendix F) residues of mulberry, honeysuckle, medlar and lotus leaf Quantitative Gas Chromatography - Mass Spectrometry.

This standard applies to the qualitative identification of 488 pesticides and related chemicals in mulberry, honeysuckle, medlar and lotus leaf, 431 pesticides and phase Quantitative determination of chemicals, 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 homogenate, salted out by centrifugation, solid phase extraction column, eluted with n-hexane-acetone and related chemicals.

Chromatography - mass spectrometry, internal 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

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

4.1.2

sodium chloride (NaCl, 7647-14-5). excellent grade pure.

4.1.3

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

4.1.4

Acetone (CH₃COCH₃, 67-64-1). Chromatographic pure.

4.1.5

n-Hexane (C₆H₁₄, 110-54-3). Chromatography pure, re-distillation.

4.1.6

Toluene C₇H₈, 108-88-3). Chromatographic Purification.

4.1.7

anhydrous sodium sulfate (Na_2SO_4 , 7757-82-6). analytical grade. 650 °C burning 4h, stored in a dryer, cooling and standby.

4.2 Standards

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

4.3.1 Standard stock solution

Respectively, to take appropriate (accurate to 0.1mg) a variety of pesticides and related chemical standards were 10mL volumetric flask, according to the standard Solvents Toluene, toluene-acetone mixture, dichloromethane and other solvents (solvents are used chromatographic pure) dissolved and fixed to the scale (solvent Select the reference to Appendix A), the standard solution from light 0 °C ~ 4 °C preservation, shelf life of one year.

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

According to the nature and retention time of pesticides and related chemicals, 488 kinds of pesticides and related chemicals are divided into A, B, C, D, E, F Six groups and determine their concentration in the mixed standard solution based on the response sensitivity of each pesticide and associated chemical on the instrument. This standard refer to Appendix A for the grouping of 488 pesticides and related chemicals and their mixed standard solution concentrations.

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 in a 100mL volumetric flask, with toluene volume to the scale. Mixed standard solution light 0 °C ~ 4 °C Save, save for one month.

4.3.3 Internal standard solution

Accurately weigh 3.5 mg Epoxy Heptachlor in a 100 mL volumetric flask and settle with a volume of toluene.

4.3.4 Matrix mixed standard working solution

A, B, C, D, E, F group of pesticides and related chemical matrix mixed standard working solution is 40 μL internal standard solution and a certain volume the mixed standard solutions of A, B, C, D, E and F were added to 1.0 mL of the sample blank matrix extract respectively, Standard working solutions A, B, C, D, E and F. Matrix mixed standard working solution should be used with the current distribution.

4.4 Materials

4.4.1

Solid phase extraction column. CleanertTPH1) 10 mL, 2.0 g, or equivalent.

4.4.2

Microporous membrane (nylon). 13mm x 0.2 μ m.

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

Rotary Evaporator.

5.4

Homogenizer. The maximum speed is 24 000 r/min.

5.5

Centrifuge. Maximum speed is 4.200 r/min.

5.6

chicken heart bottle. 150 mL.

5.7

Pipettes. 1 mL.

6.1 Preparation of the sample

Will be mulberry, honeysuckle, medlar and lotus leaf four kinds of Chinese herbal medicine grinding into fine powder, the sample all over 425 μ m standard mesh sieve, mix, the prepared samples are divided into two parts, packed in a clean container, sealed and marked.

7.1 Extraction

Respectively, to take honeysuckle, medlar sample 5 g or lotus leaf, mulberry sample 2.5 g (accurate to 0.01g) in 50mL centrifuge tube, plus Into 15 mL of acetonitrile, (wolfberry fruit samples need to add 5 mL of water), 15000 r/min homogenate extraction 1min, add 2 g sodium chloride, and then homogenate Extract 1min, 4200 r/min centrifugal 5 min, take all