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

SN 0337-1995

**Determination of carbofuran and its metabolite residues in
foods of plant origin for export Liquid chromatography-mass
spectrometry / mass spectrometry**

(English Translation)

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Foreword

This standard was drafted in accordance with the rules given in GB/T 1.1-2009.

This standard replaces SN 0337-95 "Test Method for Carbofuran Residues in Exported Fruits and Vegetables".

Compared with SN 0337-95, the main technical changes of this standard are as follows.

--The name of the standard was revised from "Methods for the Inspection of Carbofuran Residues in Exported Fruits and Vegetables" to "Kebufuran in Exported Plant-derived Foods" Determination of residues of carbaryl and its metabolites by liquid chromatography-mass spectrometry/mass spectrometry"

--Increase the detection of carbofuran metabolite 3-hydroxy carbofuran;

--Expand the scope of application of standard testing;

- The sampling part has been deleted;

--Improved sample pretreatment technology;

--The instrument method was changed from gas chromatography to liquid chromatography-mass spectrometry/mass spectrometry.

--Reduce the limit of quantification, and give the recovery range of carbofuran and its

metabolites for different sample matrices.

1 Scope

This standard specifies the method for determining the residues of carbofuran and its metabolite 3-hydroxy carbofuran in exported plant-derived foods.

This standard applies to rice, wheat, soybean, peanut, onion, rape, spinach, tomato, cucumber, potato, orange, apple. Detection and confirmation of carbofuran and its metabolite 3-hydroxy carbofuran residues in fruits, bananas and tea.

2 Normative references

The following documents are indispensable for the application of this document. For dated reference documents, only the dated version applies to this article. For undated reference documents, the latest version (including all amendments) is applicable to this document.

GB/T 2763-2016 National Food Safety Standard Maximum Pesticide Residues in Food
GB/T 6682 Analytical laboratory water specifications and experimental methods

3 Principle

The sample was extracted by shaking with acidified acetonitrile, and the extract was purified by the QuEChERS method, and measured by liquid chromatography-mass spectrometry/mass spectrometry.

Determined and quantified by external standard method.

4 Reagents and materials

Unless otherwise specified, all reagents are of analytical grade, and the water is the first grade water specified in GB/T 6682.

4.1

Acetonitrile. Chromatographically pure.

4.2

Formic acid. chromatographically pure.

4.3

Acetic acid.

4.4

Sodium chloride.

4.5

Anhydrous magnesium sulfate.

4.6

Ethylenediamine-N-propyl silanized silica gel (PSA). 40 μm ~ 60 μm .

4.7

Octadecylsilane bonded silica gel (C18). 40 μm ~ 60 μm .

4.8

Graphitized carbon black (GCB). 40 μm ~ 120 μm .

4.9 0.5% acetic acid-acetonitrile solution. accurately measure 2.5 mL of acetic acid in a 500 mL volumetric flask, add acetonitrile (4.1) to constant volume, and mix well.

4.10 0.5% formic acid solution. accurately measure 2.5 mL of formic acid in a 500 mL volumetric flask, add first-grade water to the volume, and mix well.

4.11 0.1% formic acid solution. accurately measure 0.5 mL of formic acid into a 500 mL volumetric flask, add first-grade water to the volume, and mix well.

4.12

Acetonitrile-water solution (30 to 70, volume ratio). Take 30 mL of acetonitrile (4.1) and 70 mL of first-grade water and mix well.

4.13 0.1% formic acid-acetonitrile solution. accurately measure 0.5 mL of formic acid into a 500 mL volumetric flask, add acetonitrile (4.2) to a constant volume, and mix well.

4.14

Carbofuran standard substance. $\text{C}_{12}\text{H}_{15}\text{NO}_3$, CAS number. 1563-66-2, purity greater than or equal to 90%.

4.15 3-hydroxy carbofuran standard product. $\text{C}_{12}\text{H}_{15}\text{NO}_4$, CAS number. 16655-82-6, purity greater than or equal to 90%.

4.16

Carbofuran standard stock solution. accurately weigh 10 mg (accurate to 0.1mg) of carbofuran standard in a 10 mL volumetric flask, and use acetonitrile (4.2) Dissolve and dilute to a constant volume, prepare a standard stock solution with a concentration of about 1 mg/mL, and store in a refrigerator at 0 $^{\circ}\text{C}$ ~ 4 $^{\circ}\text{C}$ in the dark.

4.17 3-Hydroxy carbofuran standard stock solution. accurately weigh 10 mg (accurate to 0.1mg) of carbofuran standard into a 10 mL volumetric flask, and use Acetonitrile (4.1) is dissolved and the volume is adjusted to a standard stock solution with a concentration of about 1 mg/mL, and it is stored in a refrigerator at 0 $^{\circ}\text{C}$ ~ 4 $^{\circ}\text{C}$ in the dark.

4.18

Carbofuran standard intermediate solution. accurately draw an appropriate volume of Carbofuran standard stock solution and use acetonitrile (4.1) to prepare a concentration of 10.0 mg/L standard intermediate solution should be stored in a refrigerator at 0°C–4°C and protected from light.

4.19 3-hydroxy carbofuran standard intermediate solution. accurately draw an appropriate volume of 3-hydroxy carbofuran standard stock solution and prepare it with acetonitrile (4.1) Prepare a standard intermediate solution with a concentration of 10.0 mg/L and store it in a refrigerator at 0°C to 4°C in the dark.

4.20

Carbofuran and 3-hydroxy carbofuran mixed standard working fluid. take appropriate amount of carbofuran standard intermediate solution (4.18) and 3-hydroxy carbofuran (4.19) Standard intermediate solution, use acetonitrile-aqueous solution (4.12) to prepare a standard working solution, the working solution needs to be used on-the-spot.

5 Equipment

5.1

Liquid chromatography-mass spectrometer/mass spectrometer. equipped with electrospray ion source (ESI).

5.2

Electronic balance. Sensitivity 0.01 mg and 0.01 g.

5.3

Oscillator.

5.4

Vortex mixer.

5.5

Centrifuge. the maximum speed is not less than 10 000 r/min.

5.6

Nitrogen blowing concentrator.

5.7

High-speed centrifuge tube. specifications are 50 mL and 5 mL.