

ICS 67.050
CCS C 53

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

SN 0693-1997

**Determination of methoprene residues in foods of plant origin
for export
(English Translation)**

Issued on: January 1, 1970

Implemented on: July 1, 2020

Issued by: General Administration of Customs

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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 0693-1997 "method for the inspection of methoprene residues in grains for export".

Compared with SN 0693-1997, the main technical changes of this standard are as follows.

--The name of the standard was revised from "Methods for the Inspection of Methoprene Residues in Grain Exports" to "Methoprene Residues in Exported Plant-derived Foods" Measurement."

--The sampling part has been deleted.

--- Added gas chromatography-mass spectrometry as the first method. Compared with SN 0693-1997, this law expands the scope of application of standard testing.

It has improved the sample pretreatment technology and lowered the limit of quantification.

- SN 0693-1997 as the second method of this standard, that is, liquid chromatography-ultraviolet detector method.

--Reduce the limit of quantification, and for different detection objects, the recovery range of multiple levels of methoprene in different matrices is given.

1 Scope

The first method of this standard specifies the gas chromatography-mass spectrometry method for the determination of methoprene residues in foods of plant origin for export. Second law A liquid chromatographic method for the determination of methoprene residues in grains has been established.

The first method of this standard applies to rice, wheat, corn, oranges, grapes, tomatoes, potatoes, spinach, mushrooms, peanuts and tea Determination and confirmation of the residual amount of methoprene in leaves. The second method is suitable for the determination of methoprene residues in brown rice.

2 Normative references

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

GB/T 6682 Analytical laboratory water specifications and experimental methods Method 1 Gas Chromatography-Mass Spectrometry

3 Principle

The sample is extracted by shaking with acetonitrile, and the extract is purified by solid phase extraction, and detected by gas chromatography-mass spectrometry.

Time and selective ion qualitative, external standard method for quantification.

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.

4.2

Toluene. chromatographically pure.

4.3

n-hexane. chromatographically pure.

4.4

Acetone. chromatographically pure.

4.5

Sodium chloride.

4.6

Anhydrous sodium sulfate. Burn at 650 °C for 4 hours, store in a desiccator, and cool it for later use.

4.7

Acetone-n-hexane solution (37, volume ratio). Measure out 60 mL of acetone and 140 mL of n-hexane, and mix them evenly.

4.8

Acetonitrile-toluene solution (3 to 1, volume ratio). Measure out 60 mL of acetonitrile and 20 mL of toluene, and mix them evenly.

4.9

Methoprene standard material. C₁₉H₃₄O₃, CAS number 40596-69-8, purity greater than or equal to 99%.

4.10

Methoprene standard stock solution. accurately weigh 10 mg (accurate to 0.1mg) of methoprene standard substance (4.9) into a 10 mL volumetric flask, Dissolve with acetone (4.4) and dilute to the mark, prepare a standard stock solution with a concentration of 1 mg/mL, and store in a refrigerator at 0 °C ~ 4 °C in the dark.

4.11

Methoprene standard intermediate solution. accurately draw an appropriate volume of the methoprene standard stock solution (4.10), and prepare a concentrated solution with acetone (4.4).

The standard intermediate solution with a concentration of 10.0 mg/L should be stored in a refrigerator at 0 °C ~ 4 °C and protected from light.

4.12

Methoprene standard working solution. Prepare the standard intermediate solution of methoprene (4.11) with n-hexane to prepare the standard working solution. This working solution needs to be used and prepared.

4.13

Neutral alumina solid phase extraction column. 1 000 mg, 6 mL, or equivalent.

4.14

Graphitized carbon solid phase extraction column. 250 mg, 6 mL, or equivalent.

5 Equipment

5.1

Gas chromatography-mass spectrometer. equipped with electron impact source (EI).

5.2

Electronic balance. Sensitivity 0.01 mg and 0.01 g.

5.3

Vortex mixer.

5.4

Oscillator.

5.5

Centrifuge. the maximum speed is not less than 5 000 r/min.

5.6

Rotary evaporator.

5.7

Nitrogen blowing concentrator.

5.8

Low-speed centrifuge tube. 50 mL, with lid.

5.9

Sweetheart bottle.

6.1 Sample preparation

Grains, nuts and tea samples. Take a representative sample of about 500 g, crush them with a grinder and pass through a 20-mesh sieve; fruits, Vegetable and mushroom samples.