



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

GB 23200.25-2016

Replacing SN/T 1115-2002

**Food safety national standard -- Determination of Herbicide
Residue in Fruits**

水果中噁草酮残留量的检测方法

(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**

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Foreword

This standard replaces SN/T 1115-2002 "Method for the determination of oxadiazone residues in fruit and vegetable".

This standard and SN/T 1115-2002, the main changes are as follows.

- Standard text format is modified to national standard text format for food safety;
- the name of the "import and export fruit" to "fruit";

- increase the "other food reference implementation" in the standard range.

This standard replaced the previous version of the standard release.

-SN/T 1115-2002.

National standards for food safety Determination of Herbicide Residue in Fruits

1 Scope

This standard specifies the sampling, sample preparation and gas chromatography-mass spectrometry and confirmation of the test of oxytetracycline residues in fruit law.

This standard applies to citrus, apple maltitol residues in the test, other food can refer to the implementation.

2 Normative reference documents

The following documents are indispensable for the application of this document. Note the date of the reference file, only the date of the date Apply to this document. 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 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.

3.1 Reagents

3.1.1

Benzene (C₆H₆). heavy distillation.

3.1.2

n-hexane (C₆H₁₄). re-distillation.

3.1.3

Sodium chloride (NaCl).

3.1.4

anhydrous sodium sulfate (Na₂SO₄). after 650 °C burning 4h, placed in the dryer in the spare.

3.2 Solution preparation

3.2.1

benzene - n-hexane solution (1 1). take 100 mL of benzene, add 100 mL of n-hexane, shake back.

3.2.2

benzene - n-hexane solution (2 1). take 200 mL of benzene, add 100 mL of n-hexane, shake back.

3.3 Standards

3.3.1

maloxione standard. purity $\geq 99\%$.

3.4 Standard solution preparation

3.4.1

maloxione stock solution. accurately weighed appropriate amount of maloxione standard, with a small amount of n-hexane dissolved, and n-hexane preparation into a concentration of 1000 $\mu\text{g/mL}$ standard stock solution. The standard stock solution is diluted to the appropriate concentration with n-hexane as needed Quasi working fluid.

3.5 Material

3.5.1

Activated carbon cartridge. SUPELLEAN ENVI-CARB cartridge, 125 mg, 3 mL or equivalent.

4 Instruments and equipment

4.1

Gas chromatograph with mass selective detector.

4.2 Analysis of balance. 0.01 g and 0.0001 g

4.3 solid phase extraction device with vacuum pump.

4.4 Centrifuge. 3000r/min.

4.5 Scroll Mixer.

4.6 centrifuge tube. 15mL.

4.7 scale tube. 15mL.

4.8 Micro-syringe. 10 μ L.

5.1 Preparation of the sample

Will be taken to take the original sample shrink 1kg, sampling site according to GB 2763 Appendix A, the organization of crusher mashed, are Divided into two, into a clean container, as a sample. Sealed and marked with mark.

5.2 Sample storage

The sample was stored at -18 ° C or lower.

Note: During sampling and sample preparation, it is necessary to prevent contamination of the sample or change in the content of the residue.

6 Analysis steps

The oxytetracycline residue in the sample was extracted with benzene-n-hexane and then purified by activated charcoal column.

Determination and confirmation of the gas chromatograph of the detector, and the external standard method.

6.1 Extraction

Accurately weigh 2.0 g homogeneous sample (accurate to 0.001g) in a 15 mL centrifuge tube, add 1 g of sodium chloride, mix and mixed with 2 mL of the benzene-n-hexane mixed solution on the mixer for 3 min at 2 500 r/min After centrifugation for 2 min, the supernatant was transferred to another 15 mL graduated tube and the residue was washed with 2 mL of benzene-n-hexane the solution was extracted twice, the combined extracts were added, and 1.0 g of anhydrous sodium sulfate was added to dryness.

6.2 Purification

The active carbon pellet was loaded with solid phase extraction on a vacuum filtration device, and the column was pre-leached with 1 ml x3 benzene to keep the flow rate 0.5 mL/min. Remove the eluent. The sample extract was added to a column and washed with a 1.5 mL? 3 benzene-n-hexane mixed solution the tubes were transferred to the pellet and the whole eluate was collected at 45 ° C. The air stream is blown to near and dissolved with n-hexane the residue was set at 0.50 mL for CC/MSD analysis.

6.3.1 Gas Chromatography - Mass Spectrometry Reference Conditions

A) Column. quartz capillary column HP-5,25 mx0.2 mm (inner diameter), film thickness 0.33 μ m, or equivalent;