

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

SN 0602-1996

Replacing SN 0602-1996

**Determination of pyrazoxyfen residue in plant-originated
foodstuffs for export--LC-MS/MS method
(English Translation)**

Issued on: August 23, 2016

Implemented on: March 1, 2017

**Issued by: The General Administration of Quality Supervision, Inspection
and Quarantine**

Table of Contents

- Foreword
- 1 Scope
- 2 Normative references
- 3 Method summary
- 4 Reagents and materials

Foreword

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

This standard replaces SN 0602-1996 "Test Method for Pizolazole Residues in Export Grains". This standard is related to SN 0602-1996 in addition to the editorial changes, the main technical changes are as follows.

- The standard scope of application extends from brown rice to rice, brown rice, corn, red beans, cabbage, carrots, and bananas;
- Change the pizozofen to the generic name benzyloxazole;
- Pretreatment to acidic acetonitrile extraction, N-propyl ethylene diamine (PSA), octadecyl silane (C18) purification;
- The measuring instrument changed from liquid chromatography to liquid chromatography-mass spectrometry/mass spectrometry;
- Sampling steps were omitted.

This standard is proposed and managed by the National Certification and Accreditation Administration.

This standard was drafted. Tianjin Entry-Exit Inspection and Quarantine Bureau of the People's Republic of China.

1 Scope

This standard specifies the ultra-high performance liquid chromatography-mass spectrometry/mass spectrometry method for the determination of benzylidene residues in exported foods.

This standard applies to the determination of benzylidene residues in exported rice, brown rice, corn, red beans, cabbage, carrots and bananas.

2 Normative references

The following documents are indispensable for the application of this document. For dated references, only dated versions apply to this article.

Pieces. For undated references, the latest edition (including all amendments) applies to this document.

GB/T 6682 Analytical laboratory water specifications and test methods

3 Method summary

The residual benzoxazole in the sample is extracted with acetonitrile solution containing 1% glacial acetic acid, and the extract is passed through N-propyl ethylenediamine (PSA), octadecyl group.

The silane (C18) was purified by ultra performance liquid chromatography-mass spectrometry/mass spectrometry and quantified by external standard method.

4 Reagents and materials

Unless otherwise specified, the reagents used were of analytical grade and the water was the first grade water specified in GB/T 6682.

4.1

Acetonitrile. chromatographically pure.

4.2

Glacial acetic acid. chromatographically pure.

4.3

Formic acid. chromatographically pure.

4.4

Methanol. Chromatographically pure.

4.5

Anhydrous magnesium sulfate. Analytically pure, burned at 650 ° C for 4 h, stored in a desiccator.

4.6

Sodium chloride. Analytical grade.

4.7 0.1% formic acid solution. Dissolve 1 mL of formic acid in water and dilute to 1 L.

4.8

Extraction solvent (1% glacial acetic acid in acetonitrile). Add 1 mL of glacial acetic acid (4.2) to 99 mL of acetonitrile (4.1).

4.9

benzoxoyfen standard. CAS number 71561-11-0, purity greater than 98%.

4.10

Standard stock solution. Accurately weigh the appropriate amount of benzylidene standard (4.9) to the nearest 0.1mg, dissolve, dilute and dilute to acetonitrile 10 mL was prepared as a standard stock solution at a concentration of 1 mg/mL. The solution can be stored for 6 months in the dark at 0 °C ~ 4 °C.

4.11

Standard intermediate solution. Dilute the mixed standard stock solution (4.10) as needed, and prepare a standard intermediate solution of appropriate concentration.

This solution can be stored for 1 month in the dark at 0 °C ~ 4 °C.

4.12

Blank sample matrix solution. Weigh a homogeneous blank sample and follow the steps 7.1 and 7.2.

4.13

Standard working solution. Pipette an appropriate amount of standard intermediate solution (4.11) and dilute with blank sample substrate solution (4.12) to make the solution Standard working solution, ready to use.

4.14

N-propyl ethylenediamine (PSA) adsorbent. 40µm~100µm.

4.15

Octadecylsilane (C18) adsorbent. 40µm~100µm.