

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

SN 0491-1995

Replacing SN 0491-1995

**Method for detection of phenflusulfamine residues in foods of
plant origin for export
(English Translation)**

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Foreword

This standard was prepared in accordance with the requirements of GB/T 1.1-2009.

This standard replaces SN 0491-1995 "Test Method for Bacteriophos Residues in Grains for Export" This standard is in line with SN 4901-1995 the main technical changes are as follows.

--- This standard adds rice, wheat, soybean, corn, pear, grape, tomato, cucumber, potato, mushroom, dried pepper test samples Matrix

--- Added confirmation of gas chromatography-mass spectrometry;

--- deleted the sampling part;

--- Optimized the pre-processing method;

Please note that some elements of this document may involve patents. The issuer of this document is not responsible for identifying these patents.

This standard is proposed and managed by the General Administration of Customs of the People's Republic of China.

This standard was drafted. Shijiazhuang Customs, People's Republic of China

1 Scope

This standard specifies the gas chromatographic detection method and gas chromatography-mass spectrometry confirmation method of benzylsulfonamide residues in foods of plant origin for export.

This standard applies to benzylsulfonamide in rice, wheat, soybean, corn, brown rice, pear, grape, potato, tomato, cucumber, mushroom and dried pepper.

Determination of residual amount.

2 Normative references

The following documents are essential for the application of this document. For dated references, only the dated version applies to this document.

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

GB/T 6682 Analytical laboratory water specifications and test methods

3 Method summary

The analytes in the sample were extracted with acetonitrile. The extract was concentrated and purified by a silica gel solid-phase extraction column for gas chromatography and gas chromatography.

Confirmed by spectrum-mass spectrometry and quantified by external standard method.

4 Reagents and materials

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

4.1

Acetonitrile, pure by high performance liquid chromatography.

4.2

n-hexane, pure by high performance liquid chromatography.

4.3

Ether.

4.4

Acetone, pure by high performance liquid chromatography.

4.5

Anhydrous sodium sulfate. Bake at 650 ° C for 4h, cool to room temperature in a desiccator, and store in a sealed bottle for later use.

4.6

N-hexane-ether (3 + 47, V/V). Take 30mL of n-hexane (4.2) and 470mL of ether (4.3) and mix well.

4.7

N-hexane-ether (1 + 1, V/V). Take 100mL of n-hexane (4.2) and 100mL of ether (4.3) and mix well.

4.8

Standard product. phenylfluorosulfanil (dichlofluid, C₉H₁₁Cl₂FN₂O₂S₂, CASN O. 1085-98-9), with a purity of 99% or more.

4.9

Standard stock solution. Accurately weigh an appropriate amount of standard product (4.8), and prepare 1.0 mg/mL standard stock solution with acetone (4.4). Avoid at 4 ° C.

Light preservation, storage period is 1 year.

4.10

Standard intermediate working solution. Take standard stock solution (4.9) from 1,000mL to 100mL volumetric flask, make up to the mark with acetone (4.4), prepare a standard intermediate working solution with a concentration of 10.0 µg/mL and store it at 4 ° C in the dark, with a shelf life of 3 months.

4.11

Standard working solution. Absorb an appropriate amount of standard intermediate solution (4.10) according to the needs of the experiment, and dilute with acetone (4.4) to a standard of appropriate concentration.

Quasi-working solution, ready to use.

4.12

Silica gel solid phase extraction column. 500mg, 6mL; or equivalent.

4.13

Microporous membrane. 0.22 μm , organic phase type.

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5 Instruments and equipment

5.1

Gas chromatograph. Equipped with an electronic capture detector (ECD).

5.2 Gas tandem mass spectrometer. Equipped with electron impact ionization source (EI)

5.3 Organization of mashers.

5.4 Electronic balance. Sensitivity is 0.01g and 0.1mg respectively.

5.5 Centrifuge. The rotation speed is not less than 5000r/min.

5.6 Homogenizer. The rotation speed is not less than 1500r/min.

5.7 Nitrogen blowing instrument.

5.8 Rotary evaporator.

5.9 Solid-phase extraction device.

5.10 Polypropylene centrifuge tubes. 50mL and 15mL, with stoppers.

5.11 10mL glass graduated test tube.

5.12 Chicken heart bottle. 120mL.

6.1 Sample preparation requirements

During sample preparation, the sample should be protected from contamination or changes in the content of residues.

6.2.1 Pear, grape, potato, tomato, cucumber, mushroom and dried pepper

Take a representative sample of 500g, pear, tomato, cucumber and dried pepper to take the whole fruit and remove the handle; grapes to take the whole fruit. potatoes and mushrooms take the whole one, use a masher to process the sample into a slurry and mix. The samples were divided into two parts, put into clean containers, sealed and marked. Below -18 °C Cryopreserved.