

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

SN/T 4558-2016

Replacing SN/T 1990-2007; SN 0150-1992

**Determination of cyhexatin (azocyclotin) and fenbutatin oxide in
foods for export**

出口食品中三环锡（三唑锡）和苯丁锡含量的测定

(English Translation)

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and Quarantine**

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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 0150-1992 and SN/T 1990-2007.

This standard and SN 0150-1992 "Test Method for Triazolium Residues in Exported Fruits" and SN/T 1990-2007 "Import and Export Foods" the main technical changes of the method for the determination of triazolium tin and tricyclotin residues in gas chromatography-mass spectrometry are as follows.

---Standard name from "Test Method for Triazolium Residues in Exported Fruits" and "Residues of Triazosin and Tricyclic Tin in Import and Export Foods" Detection method gas chromatography-mass spectrometry is unified and integrated into the measurement of tricyclic tin (triazole tin) and phenbutyl tin in export foods.

set";

---Detecting target to unify tricyclotin and triazolyl, adding phenbutyl tin;

--- Detection method is divided into the first method. gas chromatography-mass spectrometry and second method. gas chromatography;

--- Expand the scope of application of standard test objects. from the original "citrus, banana, tea, apple fish, pig liver, etc." to "Ping Fruit, citrus, radish tea and honey, etc."

--- Use "ethyl magnesium bromide" as a chemical derivative reagent instead of the original "methyl magnesium iodide".

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

This standard was drafted. Qinhuangdao Entry-Exit Inspection and Quarantine Bureau of the People's Republic of China, Shaanxi Entry-Exit Inspection of the People's Republic of China Epidemic situation.

1 Scope

This standard specifies gas chromatography-mass spectrometry and gas chromatography for the content of tricyclic tin (triazole tin) and phenbutyl tin in exported foods.

This standard applies to tricyclic tin (triazolyl) in substrates such as apple, citrus, radish, chestnut, pork, pork liver, shellfish, rice, tea and honey.

And determination of phenyltin content.

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 Principle of the method

After the sample was digested with hydrochloric acid-tetrahydrofuran, the residual triclotin and phenbutyltin were extracted with n-hexane, and the extract was concentrated and then Derivatization of ethyl magnesium bromide, the derivative is purified by Florisil solid phase extraction column, and the eluent is concentrated and fixed for gas chromatography and gas.

Determination by phase chromatography-mass spectrometry and confirmation by external standard method.

First gas chromatography-mass spectrometry

4 Reagents and materials

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

4.1

Hydrochloric acid, excellent grade.

4.2

Tetrahydrofuran.

4.3

n-hexane, chromatographically pure.

4.4

Acetone, chromatographically pure.

4.5

Concentrated sulfuric acid, concentration 98%, rho is about 1.84.

4.6

anhydrous sodium sulfate. burned at 650 ° C for 4 h, cooled and stored in a sealed container for use.

4.7

Ethyl magnesium bromide tetrahydrofuran solution (1.0 mol/L). Ethyl magnesium bromide belongs to Grignard reagent, and the reagent reacts violently with water, which will seriously in the event of an explosion or fire, the storage conditions should be kept in a desiccant container, and the contact with water should be strictly prevented during the process of use.

Operate in the wind equipment. Further, the reagent is easily oxidized in the air to lower its derivatization efficiency, and thus it is dried and sealed.

4.8

Hydrochloric acid-tetrahydrofuran (1 20, by volume) solution. Take 5.0 mL of hydrochloric acid in a measuring cylinder, add to the reagent bottle, and then slowly add to it.

Add 100 mL of tetrahydrofuran solution and gently shake to stand.

4.9

n-hexane-acetone (99 1, by volume) solution. add 99 mL of n-hexane solution to the reagent bottle, then add 1 mL of C to it.

Ketone, shake gently and set aside.

4.10 0.5mol/L sulfuric acid solution. take 500mL deionized water, take 14.3mL concentrated sulfuric acid in the measuring cylinder, slowly add deionized water, mix