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# 中华人民共和国出入境检验检疫行业标准

SN/T 3862—2014

# 出口食品中沙蚕毒素类农药残留量的筛查测定　气相色谱法

**Determination of nereistoxinic pesticide residues in foodstuffs for export—GC method**

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# 前　言

本标准按照 GB/T 1.1—2009 给出的规则起草。

请注意本文件的某些内容可能涉及专利。本文件的发布机构不承担识别这些专利的责任。

本标准由国家认证认可监督管理委员会提出并归口。

本标准起草单位：中华人民共和国北京出入境检验检疫局、中国检验检疫科学研究院。

本标准主要起草人：王金花、卢晓宇、刘韦华、陈冬东、黄梅、王莹。

# 出口食品中沙蚕毒素类农药残留量的筛查测定　气相色谱法

## 1　范围

本标准规定了食品中杀螟丹、杀虫环、杀虫双、杀虫磺、杀虫单等沙蚕毒素类农药残留量的气相色谱筛查测定方法。

本标准适用于大米、玉米、马铃薯、菠菜、洋葱、橙子、樱桃、大豆、辣椒、蘑菇等食品中杀螟丹、杀虫环、杀虫双、杀虫磺、杀虫单等沙蚕毒素类农药残留量的筛查测定。

## 2　规范性引用文件

下列文件对于本文件的应用是必不可少的。凡是注日期的引用文件，仅注日期的版本适用于本文件。凡是不注日期的引用文件，其最新版本(包括所有的修改单)适用于本文件。

GB/T 6682　分析实验室用水规格和试验方法

## 3　方法提要

试样中残留的杀螟丹、杀虫环、杀虫双、杀虫磺、杀虫单等沙蚕毒素类农药采用含有1%L-半胱氨酸盐酸盐的0.1 mol/L盐酸振荡提取，在碱性条件下转化成沙蚕毒素，毛细管气相色谱柱进行分离，用配有火焰光度检测器(具硫滤光片)的气相色谱仪检测，外标法定量。

## 4　试剂和材料

除另有说明外，所用试剂均为分析纯，水为GB/T 6682中规定的二级水。

4.1　氨水。

4.2　浓盐酸。

4.3　甲酸。

4.4　正己烷：HPLC级。

4.5　氯化镍。

4.6　氯化钠。

4.7　L-半胱氨酸盐酸盐。

4.8　0.1 mol/L盐酸溶液：量取8.3 mL浓盐酸(4.2)，用水定容至1 L。

4.9　1%甲酸溶液：称取10 g(精确至0.1 g)甲酸(4.3)，用水定容至1 L，2 ℃～8 ℃下保存。

4.10　含有1%L-半胱氨酸盐酸盐的0.1 mol/L盐酸溶液：称取10 g(精确至0.1 g)L-半胱氨酸盐酸盐(4.7)，用0.1 mol/L盐酸(4.8)溶解，并定容至1 L，常温下保存半年。

4.11　2%氯化镍溶液：称取20 g(精确至0.1 g)氯化镍，用水定容至1 L，常温下保存半年。

4.12　农药标准品：杀螟丹盐酸盐、杀虫环草酸盐、杀虫双、杀虫磺、杀虫单和沙蚕毒素草酸盐的纯度均为99%±0.5%，相关信息参见附录A。

4.13　沙蚕毒素类农药标准储备溶液：称取适量沙蚕毒素类农药标准品至10 mL容量瓶，用1%甲酸溶

液(4.9)溶解并定容至刻度,配制成标准储备溶液,其中杀虫双和杀虫单由于在酸性条件下不稳定,直接用水定容即可,2 ℃～8 ℃保存。杀螟丹盐酸盐、杀虫环草酸盐和沙蚕毒素草酸盐称量时需按照相应的摩尔质量比例换算成杀螟丹、杀虫环和沙蚕毒素后称量。

4.14 沙蚕毒素类农药标准工作溶液:用正己烷将沙蚕毒素类农药标准储备液(4.13)稀释成适当浓度的标准工作液,2 ℃～8 ℃保存。

## 5 仪器和设备

5.1 气相色谱仪:配有硫滤光片的火焰光度检测器(FPD-S)。
5.2 离心机。
5.3 粉碎机。
5.4 组织捣碎机。
5.5 分析天平:感量 0.1 mg 和 0.01 g。
5.6 振荡器。
5.7 旋涡混匀器。
5.8 聚四氟乙烯离心管:50 mL 具塞带刻度。
5.9 微孔滤膜:0.45 μm,双相通用。

## 6 试样制备和保存

### 6.1 通则

在制样的操作过程中,应防止样品受到污染或发生沙蚕毒素类农药残留含量的变化。

### 6.2 粮谷、豆类、食用菌

取代表性样品 500 g,用粉碎机粉碎。混匀,均分成两份作为试样,分装入洁净的盛样袋内,密闭,于 0 ℃～4 ℃保存。

### 6.3 水果、蔬菜

取代表性样品 1 000 g,将其可食用部分(不可用水洗涤)切碎后,用捣碎机将样品加工成浆状。混匀,均分成两份作为试样,分装入洁净的盛样袋内,密闭,于－18 ℃以下保存。

## 7 测定步骤

### 7.1 前处理

称取 5 g 试样(精确至 0.01 g)于 50 mL 离心管中,加入 10 mL 含有 1%L-半胱氨酸盐酸盐的 0.1 mol/L。盐酸溶液(4.10),振荡 20 min。依次加入 5 mL 氨水(4.1)和 2 mL 2%氯化镍溶液(4.11),经至少 2 500 r/min 旋涡混匀 1 min 后振荡 20 min。再加入 5 mL 正己烷(4.4)和约 2 g 氯化钠(4.6)使溶液达到饱和状态,经至少 2 500 r/min 旋涡混匀 1 min 后振荡 20 min,静置分层。在至少 8 000 r/min 下离心 10 min,取上清液,用 0.45 μm 微孔滤膜过滤后,供气相色谱仪测定。

### 7.2 标准曲线的绘制

进行样品分析前根据需要用正己烷稀释沙蚕毒素草酸盐标准储备溶液(4.13),配成适当浓度标准

作曲线，而且宜现用现配。本方法中，将标准储备溶液稀释至 20.0、50.0、100.0、200.0、500.0、800.0、1 000.0 ng/mL，以浓度与峰面积的平方根不过原点做标准工作曲线。

### 7.3 测定条件

7.3.1 色谱柱：DB-1701 石英弹性毛细管柱，30 m×0.32 mm(内径)，膜厚 0.25 μm，或性能相当者。

7.3.2 色谱柱温度：70 ℃保持 1 min，20 ℃/min 升温到 180 ℃保持 1.5 min，20 ℃/min 升温到 260 ℃保持 2 min。

7.3.3 进样口温度：250 ℃。

7.3.4 载气：氦气，纯度≥99.999%，恒压模式，压力 7.954 4 psi(1 psi=6.895 kPa，相当于恒流模式 1 mL/min)。

7.3.5 进样量：2 μL。

7.3.6 进样方式：不分流进样，开阀时间：0.75 min。

7.3.7 检测器：火焰光度检测器，具硫滤光片(FPD-S)，温度：250 ℃。

7.3.8 其他气体：氢气：60 mL/min；空气：70 mL/min；补充气：氮气，60 mL/min；隔垫吹扫：氦气，5 mL/min。

注：以上所列参数是在 Agilent GC 7890 A 气相色谱仪上完成的，仅为参考，并不涉及商业目的，鼓励标准使用者尝试采用不同厂家或型号的仪器。

### 7.4 气相色谱测定

根据样液中被测物含量情况，选取浓度相近的标准工作溶液。标准工作溶液和样液等体积穿插进样测定。标准工作溶液和样液中被测物的响应值均应在仪器检测的线性范围内。保留时间定性，标准曲线定量。在 7.3 给定的色谱条件下，沙蚕毒素的保留时间约为 7.9 min。标准品的色谱图参见附录 A 中图 A.1。

### 7.5 空白试验

除不加试样外，均按上述步骤进行。

## 8 结果计算和表述

气相色谱测定采用标准曲线法定量，标准曲线法定量结果按式(1)计算，计算结果应扣除空白值。

$$X = c \times \frac{V}{m} \times \frac{1\,000}{1\,000} \qquad \cdots\cdots(1)$$

式中：

$X$——试样中沙蚕毒素类农药的残留量，以沙蚕毒素总量计，单位为毫克每千克(mg/kg)；

$c$——从标准曲线上得到的沙蚕毒素溶液浓度，单位为微克每毫升(μg/mL)；

$V$——样品溶液定容体积，单位为毫升(mL)；

$m$——样品溶液所代表试样的质量，单位为克(g)。

## 9 测定低限、回收率和精密度

### 9.1 测定低限

本方法沙蚕毒素类农药总量的测定低限为 0.10 mg/kg。

### 9.2 回收率和精密度

采用本方法对样品进行添加回收试验。添加回收率和精密度参见附录 B。

# 附 录 A
（资料性附录）
沙蚕毒素标准品信息和标准溶液谱图

表 A.1 沙蚕毒素类农药标准品信息

| 中文名 | 英文名 | 分子式 | 相对分子质量 | CAS 号 |
|---|---|---|---|---|
| 杀螟丹盐酸盐 | cartap hydrochloride | $C_7H_{15}N_3O_2S_2 \cdot HCl$ | 273.81 | 22042-59-7 |
| 杀虫环草酸盐 | thiocyclam-hydrogen oxalate | $C_5H_{11}NS_3 \cdot C_2H_2O_4$ | 271.38 | 31895-22-4 |
| 杀虫双 | thiosultap-sodium | $C_5H_{11}NNa_2O_6S_4$ | 355.4 | 52207-48-4 |
| 杀虫磺 | bensultap | $C_{17}H_{21}NO_4S_4$ | 431.63 | 17606-31-4 |
| 杀虫单 | monosultap | $C_5H_{14}NNaO_7S_4$ | 351.39 | 29547-00-0 |
| 沙蚕毒素草酸盐 | nereistoxin oxalate | $C_5H_{11}NS_2 \cdot C_2H_2O_4$ | 239.31 | 1631-52-3 |

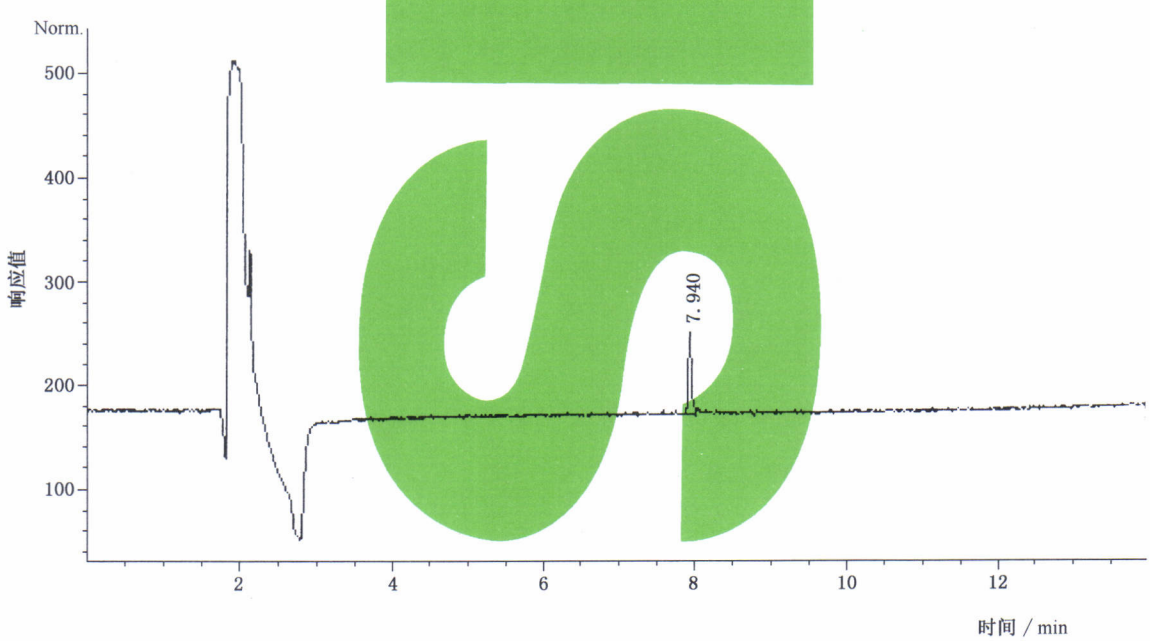


图 A.1 沙蚕毒素标准溶液（浓度为 0.10 μg/mL）气相色谱图

# 附 录 B
（资料性附录）
沙蚕毒素类农药在样品中的回收率范围和精密度

表 B.1 沙蚕毒素类农药在样品中的回收率范围和精密度（$n=12$）

| 样品名称 | 添加浓度/(mg/kg) | 回收率范围/% | 精密度/% |
|---|---|---|---|
| 大米 | 0.10 | 60.84～67.64 | 3.86 |
| | 0.20 | 60.71～81.64 | 12.13 |
| | 0.40 | 60.91～73.87 | 7.55 |
| 玉米 | 0.10 | 84.02～105.10 | 6.57 |
| | 0.20 | 60.71～76.54 | 6.63 |
| | 0.40 | 66.52～77.67 | 4.77 |
| 马铃薯 | 0.10 | 82.57～100.53 | 6.61 |
| | 0.20 | 68.22～75.99 | 3.64 |
| | 0.40 | 76.34～82.84 | 3.10 |
| 菠菜 | 0.10 | 69.40～114.29 | 19.47 |
| | 0.20 | 62.66～72.40 | 4.60 |
| | 3.00 | 62.35～72.53 | 5.10 |
| 洋葱 | 0.10 | 60.92～83.09 | 9.67 |
| | 0.20 | 82.09～93.59 | 4.37 |
| | 3.00 | 95.19～116.60 | 6.13 |
| 橙子 | 0.10 | 65.58～109.95 | 17.59 |
| | 0.20 | 69.10～84.62 | 7.59 |
| | 3.00 | 67.35～89.74 | 10.74 |
| 樱桃 | 0.10 | 84.24～101.67 | 6.05 |
| | 0.20 | 69.64～89.70 | 7.17 |
| | 3.00 | 77.29～98.45 | 10.25 |
| 大豆 | 0.10 | 67.27～104.23 | 14.21 |
| | 0.20 | 60.60～74.65 | 7.60 |
| | 3.00 | 68.87～87.36 | 10.82 |
| 辣椒 | 0.10 | 65.00～108.87 | 17.84 |
| | 0.20 | 78.81～90.85 | 4.22 |
| | 3.00 | 65.46～79.52 | 7.53 |
| 蘑菇 | 0.10 | 63.31～93.17 | 10.43 |
| | 0.20 | 85.61～118.43 | 9.75 |
| | 3.00 | 71.15～102.42 | 12.81 |

# Foreword

This standard was drafted in accordance with the rule of GB/T 1.1—2009.

Please note that some of the elements of this standard may involve patents, but the Standards Organization does not assume responsibility for identifying these patents.

This standard is proposed by and is under the charge of Certification and Accreditation Administration of the People's Republic of China.

This standard is drafted by Beijing Entry-Exit Inspection and Quarantine Bureau and Chinese Academy of Inspection and Quarantine.

This standard is mainly contributed by Wang Jinhua, Lu Xiaoyu, Liu Weihua, Chen DongDong, Huang Mei, and Wang Ying.

Note: This English version, a translation from the Chinese text, is only for reference.

# Determination of nereistoxinic pesticide residues in foodstuffs for export—GC method

## 1 Scope

This standard specifies the method to determinate of nereistoxin residues, including cartap, thiocyclam-hydrogenoxalate, thiosultap-sodium (bisultap), bensultap, monosultap, in foodsuffs for export by gas chromatograph (GC).

This standard is applicable to the qualitative determination of nereistoxin residues in rice, corn, potato, spinach, onion, orange, cherry, soy, pepper, mushroom.

## 2 Normative quoted documents

Following documents are necessary for the application of this present file. Only the version dated can be applied to the present documents, if not, the latest edition can do (including all the modified form).

GB/T 6682 Water for analytical laboratory use—Specification and test methods.

## 3 Principle

The nereistoxin residues from the above food samples are extracted with 0.1 mol/L hydrochloric acid containing 1% L-cysteine hydrochloride and transformed into nereistoxin under alkaline conditions. Then separated with capillary column, detected by GC equipped with flame photometric detector (with sulfur filter), and quantified by external standard method.

## 4 Reagents and materials

Unless otherwise specified, all reagents used should be Reagent for Residue analysis. Water used for preparing solutions must be in conformity with the requirements of second-level Water in GB/T 6682.

**4.1** Ammonia.

**4.2** Concentrated hydrochloric acid.

**4.3** Formic acid.

**4.4** *n*-Hexane: HPLC grade.

**4.5** Nickel chloride.

**4.6** Sodium chloride (NaCl).

**4.7** L-Cysteine hydrochloride.

**4.8** 0.1 mol/L hydrochloric acid solution: 8.3 mL concentrated hydrochloric acid(4.2) was dissolved and diluted by water to 1 L.

4.9 1% Formic acid: Accurately weigh 10 g (accurating to 0.1 g) formic acid (4.3), dissolved with water to 1 L, and store under 2 ℃～8 ℃.

**4.10** 0.1 mol/L hydrochloric acid containing 1% L-cysteine hydrochloride: Accurately weigh 10 g (accurating to 0.1 g) L-cysteine hydrochloride, dissolved with 0.1 mol/L hydrochloric acid (4.8) to 1 L, and store under room temperature for 6 months.

**4.11** 2% Nickel chloride: Accurately weigh 20 g (accurating to 0.1 g) nickel chloride, dissolved with water to 1 L, and store under room temperature for 6 months.

**4.12** Standards: cartap hydrochloride, thiocyclam-hydrogen oxalate, thiosultap-sodium, bensultap, monosultap, and nereistoxin oxalate, the purity = 99% ± 0.5 %. The details about the standards are showed in Annex A.

**4.13** Standard store solution: weigh an adequate amount of nereistoxinic pesticides (accurating to 0.1 mg) and dissolved with 1% formic acid (4.9) to 10 mL with the exception of thiosultap-sodium and monosultap with water because of their instability under acidic conditions. The solution should be stored under 2 ℃～8 ℃. Cartap hydrochloride, thiocyclam-hydrogen oxalate and nereistoxin oxalate should be scale into cartap, thiocyclam and nereistoxin in accordance with the corresponding molar mass before weighing.

**4.14** Standard working solution: according to the concentration required, a standard working solution is prepared from the stock solution (4.13) with *n*-hexane and should be stored under 2 ℃～8 ℃.

## 5 Apparatus and equipment

**5.1** GC: with flame photometric detector(with sulfur filter).

**5.2** Centrifuge.

**5.3** Grinder.

**5.4** Tissue-tearor.

**5.5** Analytical balance: 0.1 mg and 0.01 g.

**5.6** Shaker.

**5.7** Vortex mixter.

**5.8** Teflon centrifuge tube: 50 mL with plug and scale.

**5.9** Microporous membrane: 0.45 μm, dual phase.

## 6 Sample preparation and storage

### 6.1 Summary

In the course of sample preparation, precautions should be taken to avoid contamination or any factors which may cause the change of nereistoxinic pesticide residues content.

### 6.2 Cereals, legumes and edible fungus

The original sample is mixed from which a 500 g is taken for analysis. The sample is divided to two potions equally after grinded thoroughly. Then the sample is placed in a clean container to be used as the test sample. The container having the test sample is well sealed and labeled. The samples should be stored under 0 ℃～4 ℃.

### 6.3 Fruits and vegetables

One kg of the edible parts from the original sample without washing is blended thoroughly. The sample is then divided to two equal portions. The sample is placed in a clean container to be used as the test sample. The container having the test sample is well sealed and labeled. The test sample should be stored at −18 ℃.

## 7 Procedure

### 7.1 Pre-preparation of the sample

Weigh 5 g (accurating to 0.01 g) of the test sample, place the sample into 50 mL plastic centrifuge

tube, add 10 mL 0.1 mol/L hydrochloric acid containing 1% L-cysteine hydrochloride(4.10) and shake for 20 min. Add 5 mL ammonia (4.1) and 2 mL 2% nickel chloride solution ( 4.11), and shake for 20 min after homogenizing at a speed of 2 500 r/min for 1 min. And then add 5 mL *n*-hexan (4.4) and ca. 2 g NaCl (4.6) for saturating the solution. After homogenizing at 2 500 r/min for 1 min, shake for 20 min and place for stratification. After centrifuging at 8 000 r/min for 10 min, transfer the top clear layer to filtrate through the 0.45 μm microporous membrane filter for GC analysis.

**7.2** Preparation of standard curve

According to the concentration required, a appropriate concentration standard working curve is prepared from the nereistoxin oxalate standard stock solution (4.13) with *n*-hexane before sample analysis, and only by using. For the present method, the standard stock solution was diluted to 20.0, 50.0, 100.0, 200.0, 500.0, 800.0, and 1 000.0 ng /mL. The working-curve should be made with the concentration-the square root of peak area and not through the origin.

**7.3** Conditions of GC

**7.3.1** Column: DB-1701, 30 m×0.32 mm (i.d.)×0.25 μm (film thickness) or equivalent.

**7.3.2** Column temperature: 70 ℃, hold for 1 min;

Ramp to 180 ℃ at 20 ℃/min, hold for 1.5 min;

Ramp to 260 ℃ at 20 ℃/min, hold for 2 min.

**7.3.3** Iniector temperature: 250 ℃.

**7.3.4** Carrier gas: helium, purity≥99.999%, constant-voltage mode with pressure of 7.954 4 psi (1 psi=6.895 kPa,equal to constant-current mode with flow rate of 1.0 mL /min).

**7.3.5** Injection volumn: 2 μL.

**7.3.6** Injection mode: splitless, purge flow to split vent at: 0.75 min.

**7.3.7** Detector: FPD-S, temperature: 250 ℃.

**7.3.8** Other gas used: $H_2$ flow: 60 mL/min;air flow: 70 mL/min;makeup flow ($N_2$): 60 mL/min; septum purge flow (He): 5 mL/min.

Non-commercial statement: the equipments and their types Agilent GC 7890A involved in this standard method are not related to commercial aims, and the analysts are encouraged to use equipments of different corporation or different type.

### 7.4 Determination

Inject equal amount (2 μL) of the nereistoxin oxalate standard working solution and sample solution accurately into GC system, respectively. The sample solutions should be injected betweem the standard solutions. The response of the nereistoxin between the standard working solution and the sample solution should be matched and must be within the linear range of the detector. The retention time of nereistoxin is about 7.9 min under the condition as per section 7.3.The GC chromatogram of nereistoxin oxalate standard is showed in Annex A Figure. A. 1.

### 7.5 Blank test

The operation of the blank test is the same as that described in the method of determination,except without sample.

## 8 Calculation and expression of results

The quantitative determination of the residue content of nereistoxinic pesticides in samples is curried out by using standard curve method and the calculation is done by Chem Station software or according to the formula (1) (external standard method) and expressed in mg/kg. The blank value should be subtracted from the result of calculation.

$$X = c \times \frac{V}{m} \times \frac{1\,000}{1\,000} \qquad \cdots\cdots(1)$$

Where:

$X$ —the residue content of nereistoxinic pesticides in test sample, mg/kg;

$c$ —the concentration of nereistoxinic pesticides in the standard working solution, μg/mL;

$V$ —the volumn of nereistoxinic pesticides in sample solution, mL;

$m$ —the mass of test sample in the final sample solution, g.

## 9 Limit of determination and recovery

### 9.1 Limit of determination

The limit of determination for total nereistoxinic pesticide residuses using this method is 0.10 mg/kg.

### 9.2 Recovery and Precision

A spike test was carried out by this method.Recoveries and precision see annex B table B.1.

# Annex A
(Informative)
## Chromatogram of the nereistoxin standard

**Table A.1 Information about the nereistoxinic pesticide standards**

| Name | Molecular formula | Molecular weight | CAS.No. |
|---|---|---|---|
| Cartap hydrochloride | $C_7H_{15}N_3O_2S_2 \cdot HCl$ | 273.81 | 22042-59-7 |
| Thiocyclam-hydrogen oxalate | $C_5H_{11}NS_3 \cdot C_2H_2O_4$ | 271.38 | 31895-22-4 |
| Thiosultap-sodium | $C_5H_{11}NNa_2O_6S_4$ | 355.4 | 52207-48-4 |
| Bensultap | $C_{17}H_{21}NO_4S_4$ | 431.63 | 17606-31-4 |
| Monosultap | $C_5H_{14}NNaO_7S_4$ | 351.39 | 29547-00-0 |
| Nereistoxin oxalate | $C_5H_{11}NS_2 \cdot C_2H_2O_4$ | 239.31 | 1631-52-3 |

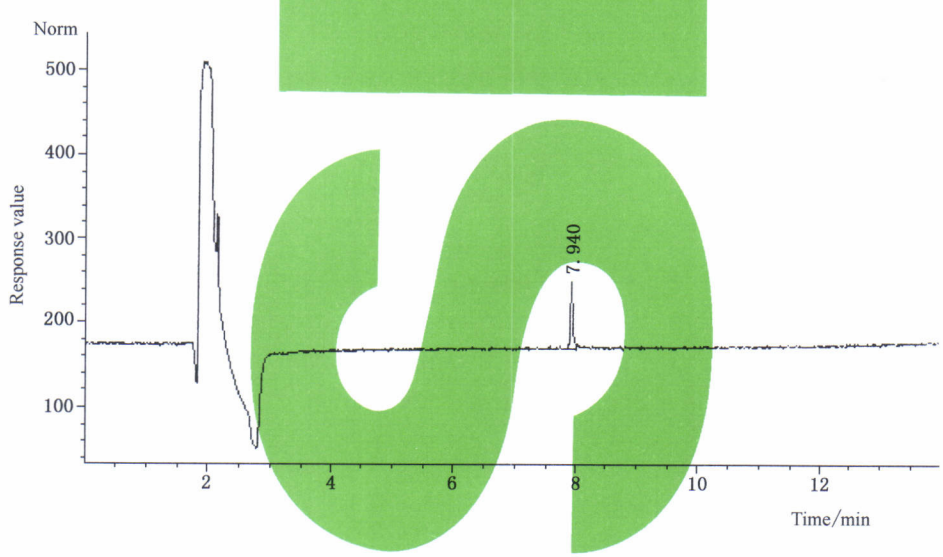


**Figure A.1 GC chromatogram of nereistoxin standard (concentration at 0.10 μg/mL)**

## Annex B
## (Informative)
## Recovery and Precision

**Table B.1 Recovery range and precision(*RSD*) of nereistoxin in different foodstuff samples ($n = 12$)**

| Sample | Fortifying concentration/(mg/kg) | Recovery range/% | *RSD*/% |
|---|---|---|---|
| Rice | 0.10 | 60.84～67.64 | 3.86 |
| | 0.20 | 60.71～81.64 | 12.13 |
| | 0.40 | 60.91～73.87 | 7.55 |
| Corn | 0.10 | 84.02～105.10 | 6.57 |
| | 0.20 | 60.71～76.54 | 6.63 |
| | 0.40 | 66.52～77.67 | 4.77 |
| Potato | 0.10 | 82.57～100.53 | 6.61 |
| | 0.20 | 68.22～75.99 | 3.64 |
| | 0.40 | 76.34～82.84 | 3.10 |
| Spinach | 0.10 | 69.40～114.29 | 19.47 |
| | 0.20 | 62.66～72.40 | 4.60 |
| | 3.00 | 62.35～72.53 | 5.10 |
| Onion | 0.10 | 60.92～83.09 | 9.67 |
| | 0.20 | 82.09～93.59 | 4.37 |
| | 3.00 | 95.19～116.60 | 6.13 |
| Orange | 0.10 | 65.58～109.95 | 17.59 |
| | 0.20 | 69.10～84.62 | 7.59 |
| | 3.00 | 67.35～89.74 | 10.74 |
| Cherry | 0.10 | 84.24～101.67 | 6.05 |
| | 0.20 | 69.64～89.70 | 7.17 |
| | 3.00 | 77.29～98.45 | 10.25 |
| Soy | 0.10 | 67.27～104.23 | 14.21 |
| | 0.20 | 60.60～74.65 | 7.60 |
| | 3.00 | 68.87～87.36 | 10.82 |
| Pepper | 0.10 | 65.00～108.87 | 17.84 |
| | 0.20 | 78.81～90.85 | 4.22 |
| | 3.00 | 65.46～79.52 | 7.53 |
| Mushroom | 0.10 | 63.31～93.17 | 10.43 |
| | 0.20 | 85.61～118.43 | 9.75 |
| | 3.00 | 71.15～102.42 | 12.81 |