SN

中华人民共和国出入境检验检疫行业标准

SN/T 2147—2008

进出口食品中硫线磷残留量的检测方法

Determination of cadusafos residues in food for import and export

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前　言

本标准附录A和附录B均为资料性附录。

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：中华人民共和国山西出入境检验检疫局、中华人民共和国吉林出入境检验检疫局、中华人民共和国湖南出入境检验检疫局。

本标准主要起草人：薛平、连庚寅、王明泰、杜利君、张莹、康杰、苑利。

本标准系首次发布的出入境检验检疫行业标准。

进出口食品中硫线磷残留量的检测方法

1 范围

本标准规定了进出口食品中硫线磷残留量的气相色谱测定和气相色谱-质谱确证方法。

本标准适用于芦笋、土豆、大葱、苹果、桃、玉米、荞麦、茶叶、食醋、蜂蜜、核桃仁、兔肉、鸡肝和虾仁中硫线磷残留量的测定和确证。

2 方法提要

植物源性样品用乙酸乙酯-环己烷混合溶剂提取；动物源性样品和含油量高的植物源性样品用乙腈提取，提取液用凝胶色谱仪(GPC)或 C_{18} 固相萃取柱净化，供带有氮磷检测器的气相色谱仪测定，外标法定量，阳性样品用气相色谱-质谱法确证。

3 试剂和材料

除非另有规定，所用试剂均为分析纯，水为二次蒸馏水。

3.1 乙酸乙酯：农残级。

3.2 环己烷：农残级。

3.3 乙腈：色谱纯。

3.4 正己烷。

3.5 三氯甲烷。

3.6 无水硫酸钠：650 ℃灼烧 4 h，贮于密封容器中备用。

3.7 乙酸乙酯-环己烷(1+1，体积比)：500 mL 乙酸乙酯中加入 500 mL 环己烷，混匀。

3.8 乙酸乙酯-环己烷(1+2，体积比)：500 mL 乙酸乙酯中加入 500 mL 环己烷，混匀。

3.9 硫线磷标准物质(Cadusafos，$C_{10}H_{23}O_2PS_2$；CAS：95465-99-9，分子量：270.4)：纯度大于等于 99%。

3.10 硫线磷标准储备液(100 mg/L)：准确称取适量硫线磷标准物质，用乙酸乙酯溶解，配制成浓度为 100 mg/L 的标准储备液，该溶液在 0 ℃～4 ℃保存。

3.11 硫线磷标准工作液：根据需要再用乙酸乙酯稀释成适当浓度的标准工作溶液。标准工作液应现用现配。

3.12 C_{18} 固相萃取柱：60 mg，3 mL，或相当者。

3.13 微孔滤膜：0.45 μm。

4 仪器和设备

4.1 气相色谱仪：配有氮磷检测器。

4.2 气相色谱-质谱仪：配有电子轰击源(EI)。

4.3 分析天平：感量为 0.000 1 g。

4.4 凝胶色谱仪，配有单元泵，馏分收集器。

4.5 固相萃取装置。

4.6 离心机：4 000 r/min。

4.7 无水硫酸钠柱：7.5 cm×1.5 cm(内径)玻璃柱，内装 5 cm 高无水硫酸钠。

4.8 旋转蒸发器。

4.9 涡旋混合器。

4.10 超声波清洗器。

4.11 氮吹仪。

4.12 具塞离心管:50 mL,聚四氟乙烯。

5 样品置备与保存

5.1 样品制备

5.1.1 粮谷及茶叶类

将样品按四分法缩分至500 g,用磨碎机全部磨碎。混匀,均分成两份作为试样,分装入洁净的盛样瓶内,密闭,标明标记。

5.1.2 水果及蔬菜类

抽取水果或蔬菜样品500 g,或去壳、去籽、去皮、去茎、去根、去冠(不可用水洗涤),将其可食用部分切碎后,依次用食品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,标明标记。

5.1.3 肉及肉制品类、坚果类

从所取全部样品中取出有代表性样品约1 kg,取可食部分经捣碎机充分捣碎均匀,均分成两份,分别装入洁净容器内作为试样。密封并标明标记。

5.2 试样保存

粮谷类、茶叶类、坚果类、蜂蜜及蜂蜜制品类试样于0 ℃～4 ℃保存;其他类试样于－18 ℃以下冷冻保存。

在制样的操作过程中,应防止样品受到污染或发生残留物含量的变化。

6 测定步骤

6.1 提取

6.1.1 蔬菜、水果、粮谷

蔬菜、水果称取20 g试样(精确至0.1 g),粮谷称取10 g试样(精确至0.1 g),置于50 mL离心管中,加入20 mL乙酸乙酯-环己烷混合溶剂(3.7),涡旋振荡混匀30 s,超声提取20 min,4 000 r/min离心3 min,移取上层有机相,残留物再用20 mL乙酸乙酯-环己烷混合溶剂(3.7)重复提取一次,合并上层有机相,过无水硫酸钠玻璃柱(4.7),40 ℃下旋转浓缩至近干,准确加入10.0mL乙酸乙酯-环己烷(3.7)溶解残渣,并用0.45 μm滤膜过滤,待净化。

6.1.2 茶叶

称取2.5 g试样(精确至0.1 g),置于50 mL离心管中,加入10 mL水,静置1 h,加入20 mL乙酸乙酯-环己烷混合溶剂(3.8)旋涡振荡混匀30 s,超声提取及随后步骤同6.1.1。

6.1.3 醋、蜂蜜

C_{18}固相萃取柱依次用2 mL正己烷(3.4),2 mL三氯甲烷(3.5),3 mL蒸馏水预淋洗。称取2.5 g试样(精确至0.1 g),加入5 mL水旋涡振荡混匀30 s进行提取,将提取液全部过已经预淋洗的固相萃取柱,流速控制在1滴/2 s,再用2 mL水淋洗柱子,弃去淋洗液,真空抽干2 min,用2.5 mL正己烷洗脱,洗脱液40 ℃下氮气吹干,用乙酸乙酯定容至1.0 mL,待测。

6.1.4 核桃仁、兔肉、鸡肝、虾

称取10 g试样(精确至0.1 g),置于50 mL离心管中,加入20 mL乙腈(3.3),使用均质器搅拌30 s,超声提取20 min,4 000 r/min离心3 min,收集上层有机相,残留物再用20 mL乙腈重复提取一次,合并上层有机相,过无水硫酸钠玻璃柱,收集液于40 ℃旋转浓缩至1 mL～2 mL时,加入5 mL环己烷-乙酸乙酯(3.7)再次旋转浓缩至近干,准确加入10 mL环己烷-乙酸乙酯(3.7)溶解残渣,并用

0.45 μm滤膜过滤，待净化。

6.2 凝胶色谱净化

6.2.1 凝胶色谱净化条件

a) 凝胶净化柱：Bio Beads S-X3，300 mm×20 mm(内径)，或相当者；

b) 流动相：环己烷-乙酸乙酯(3.7)；

c) 流速：4.7 mL/min；

d) 样品定量环：5 mL；

e) 收集时间：7.5 min～12.5 min。

6.2.2 凝胶色谱净化步骤

将10 mL待净化液按上述条件过凝胶色谱净化，收集7.5 min～12.5 min流出液，35 ℃下氮气吹至近干，用乙酸乙酯定容至1.0 mL，供气相色谱仪测定，气相色谱-质谱法确证。

6.3 气相色谱测定

6.3.1 气相色谱条件

a) 色谱柱：CP-1301石英毛细管柱，30 m×0.25 mm(内径)×0.25 μm，或性能相当者；

b) 色谱柱温度：50 ℃(1 min)$\xrightarrow{20\ ℃/min}$220 ℃；

c) 进样口温度：260 ℃；

d) 检测器温度：280 ℃；

e) 载气：氮气，纯度大于等于99.995%，柱流量1 mL/min；

f) 气体流量：氢气：3.0 mL/min，空气：150 mL/min，尾吹气：30 mL/min；

g) 恒流方式：1 pA；

h) 进样方式：无分流，1 min后打开分流阀；

i) 进样量：1 μL。

6.3.2 气相色谱测定

根据样液中硫线磷含量情况，选定峰面积相近的标准工作溶液，标准工作溶液和样液中硫线磷相应值均应在仪器检测线性范围内。标准工作溶液和样液等体积参插进样测定。在上述色谱条件下，硫线磷的保留时间约为13.0 min。标准品的色谱图参见附录A中图A.1。

标准溶液及样液均按6.3.1的条件进行测定，如果样液中与标准溶液相同的保留时间有峰出现，则对其进行气相色谱-质谱确证。

6.4 气相色谱-质谱确证

6.4.1 气相色谱-质谱条件

a) 色谱柱：HP-5MS石英毛细管柱，30 m×0.25 mm(内径)×0.25 μm，或性能相当者；

b) 色谱柱温度：50 ℃(1 min)$\xrightarrow{10\ ℃/min}$280 ℃(10 min)；

c) 进样口温度：250 ℃；

d) 色谱-质谱接口温度：280 ℃；

e) 电离方式：EI；

f) 电离能量：70 eV；

g) 载气：氦气，纯度大于等于99.999%，流速1 mL/min；

h) 进样方式：无分流，0.75 min后打开分流阀；

i) 进样量：1 μL；

j) 测定方式：选择离子监测；

k) 选择监测离子(m/z)：159、213、215、270；

l) 溶剂延迟：5.0 min。

6.4.2 气相色谱-质谱法确证

经确证分析被测物质量色谱峰保留时间与标准品样品相一致，并且在扣除背景后的样品谱图中，所选择的离子均出现；同时所选择离子的丰度比与标准样品相关离子的相对丰度一致，相似度在允许差之内（见表1），则可判定样品为硫线磷阳性检出。硫线磷标准物质的气相色谱-质谱选择离子色谱图和质谱图参见附录B中图B.1和图B.2。

表1 定性确证时相对离子丰度的最大允许偏差

相对离子丰度/%	>50	>20～50	>10～20	≤10
允许相对偏差/%	±10	±15	±20	±50

6.5 空白试验

除不称取试样外，均按上述步骤进行。

7 结果计算和表述

用色谱数据处理机或按式(1)计算试样中硫线磷残留量：

$$X = \frac{A \cdot c_s \cdot V}{A_s \cdot m} \qquad \cdots\cdots (1)$$

式中：

X——试样中硫线磷残留量，单位为毫克每千克(mg/kg)；

A——样液中硫线磷的峰面积；

c_s——标准工作液中硫线磷的浓度，单位为微克每毫升(μg/mL)；

V——样液最终定容体积，单位为毫升(mL)；

A_s——标准工作液中硫线磷的峰面积；

m——最终样液所代表的试样质量，单位为克(g)。

注：计算结果应扣除空白值。

8 测定低限和回收率

8.1 测定低限

本标准对桃、苹果、芦笋、大葱、土豆、玉米、荞麦、虾仁和兔肉的测定低限为0.005 mg/kg；茶叶、食醋、蜂蜜、核桃仁和鸡肝的测定低限为0.01 mg/kg。

8.2 回收率

方法回收率见表2。

表2 检测低限及回收率范围

样品名称	添加浓度范围/(mg/kg)	回收率范围/%
桃	0.005～0.020	89.9～104.0
苹果	0.005～0.020	84.8～107.0
芦笋	0.005～0.020	89.6～102.0
大葱	0.005～0.020	87.0～98.1
土豆	0.005～0.020	87.8～92.5
玉米	0.005～0.020	92.8～105.0
荞麦	0.005～0.020	93.8～103.6
茶叶	0.01～0.050	93.0～99.0

表 2（续）

样品名称	添加浓度范围/(mg/kg)	回收率范围/%
蜂蜜	0.01～0.050	93.0～99.0
食醋	0.01～0.050	90.0～104.0
核桃仁	0.01～0.050	84.3～105.0
兔肉	0.005～0.020	93.5～110.0
鸡肝	0.01～0.050	90.7～99.0
虾仁	0.005～0.020	92.5～98.0

附　录　A
（资料性附录）
硫线磷标准物质气相色谱图

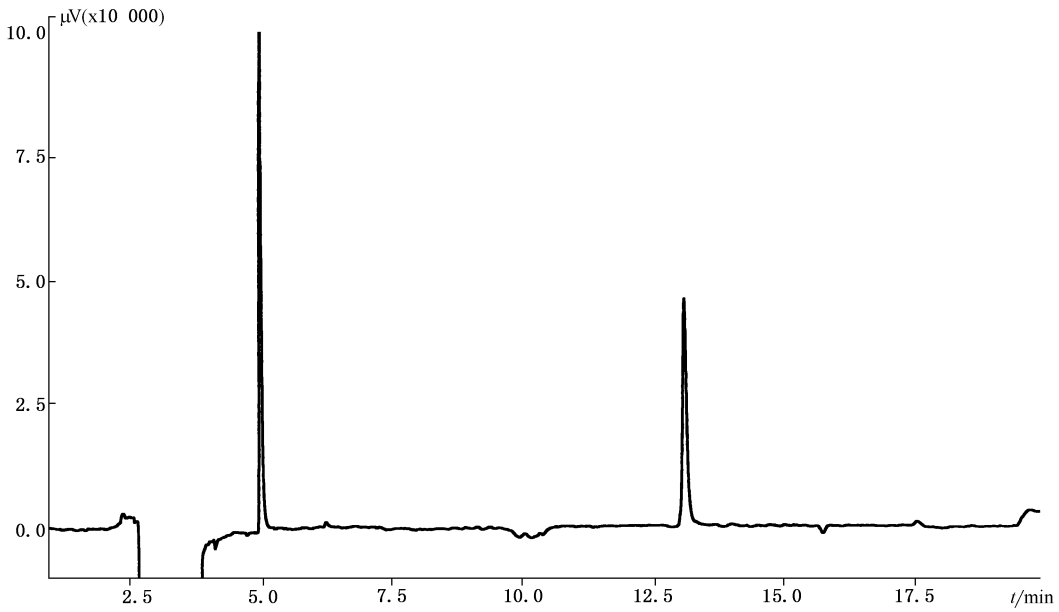

图 A.1　硫线磷标准物质气相色谱图

附　录　B
（资料性附录）
硫线磷标准物质气相色谱-质谱图

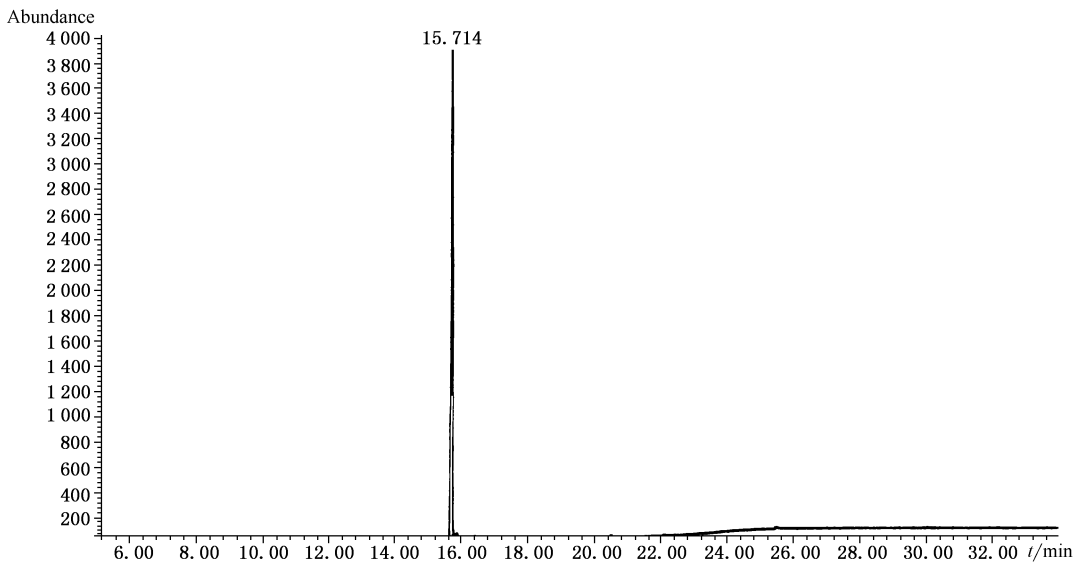

图 B.1　硫线磷标准物质(1.0 μg/mL)气相色谱-质谱选择离子色谱图

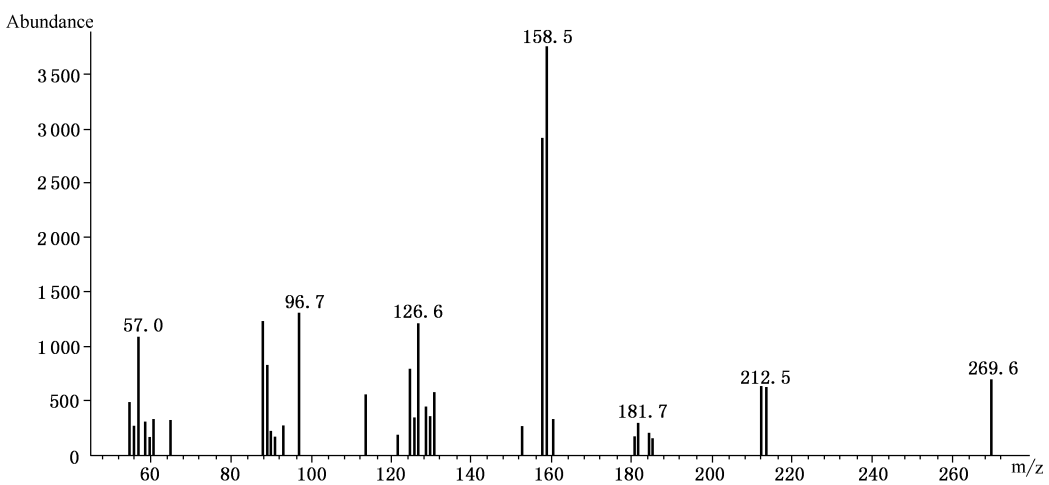

图 B.2　硫线磷标准物质质谱图

Foreword

Annex A and Annex B of this standard is an informative annex.

This standard was proposed by and is under the charge of the National Regulation Commission for Certification and Accreditation.

This standard was drafted by Shanxi Entry-Exit Inspection and Quarantine Bureau，Jilin Entry-Exit Inspection and Quarantine Bureau，Hunan Entry-Exit Inspection and Quarantine Bureau.

This main drafters of this standard are：Xue Ping，Lian Gengyin，Wang Mingtai，Du Lijun，Zhang Ying，Song Huan，Yuan Li，Pan Yali，Kang Jie.

This standard is an inspection and quarantine professional standard promulgated for the first time.

Determination of cadusafos residues in food for import and export

1 Scope

This standard specifies the methods of sample preparation, determination and confirmation of cadusafos residues in foods for import and export by GC and GC-MS.

This standard is applicable to the determination of cadusafos residues in asparagus, murphy, scallion, apple, peach, corn, buckwheat, tea leaf, vinegar, honey, walnut, chicken liver, rabbit meat and shrimp.

2 Principle

The cadusafos residues were extracted with ethyl acetate-cyclohexane or acetonitrile, purified by a gel permeation chromatography(GPC) or C_{18} SPE tubes, determinated by GC and confirmation by GC-MS, using external standard method.

3 Reagents and materials

All the reagents used should be analytically pure unless otherwise specified. "Water" is distilled water.

3.1 Ethyl acetate: Residues grade.

3.2 Cyclohexane: Residues grade.

3.3 Acetonitrile: HPLC grade.

3.4 Hexane.

3.5 Trichloromethane.

3.6 Anhydrous sodium sulfate: Ignited at 650 ℃ for 4 h, and kept in a tightly closed container.

3.7 Ethyl acetate + Cyclohexane(1 + 1, *V*/*V*).

3.8 Ethyl acetate + Cyclohexane(1 + 2, *V*/*V*).

3.9 Cadusafos standard(Cadusafos; $C_{10}H_{23}O_2PS_2$; CAS:95465-99-9, molecular weight:270.4): Purity ≥99%.

3.10 Standard stock solution: Accurately weigh a certain amount of cadusafos standard and dissolve it in a small volume of Ethyl acetate. Dilute with Ethyl acetate to make the standard stock solution of 100 mg/mL. It should be stored in a refrigerator at 0 ℃～4 ℃.

3.11 Standard working solution: Then dilute the standard stock solution with Ethyl acetate to the required concentration as the standard working solution. These solutions should be prepared before use.

3.12 C_{18} SPE tubes: 3 mL, 60 mg, or equivalent.

3.13 Film: 0.45 μm.

4 Apparatus and equipment

4.1 Gas Chromatography: Equipped with FTD.

4.2 Gas Chromatography: Equipped with electron ionization mass spectrometry.

4.3 Analytical balance: 0.000 1 g.

4.4 Gel permeation chromatograph equipped with isocratic pump and fraction collector.

4.5 Solid-extraction equipment.

4.6 Centrifuge: 4 000 r/min.

4.7 Anhydrous sodium sulfate column: 7.5 cm × 1.5 cm (i.d.) fuel, filled with 5 cm anhydrous sodium sulfate upon 5 mm absorbent cotton. Rotary vacuum evaporator.

4.8 Rotary vacuum evaporator.

4.9 Vortex mixer.

4.10 Ultrasonic machine.

4.11 Pressured gas blowing concentrator.

4.12 Stoppered plastic centrifuge tube: 50 mL.

5 Preparation and storage of test sample

5.1 Preparation of test sample

5.1.1 Cereals and tea

Quarter the sample to ca. 500 g, Grind thoroughly in a high speed blender. Mix thoroughly and divide into two equal portions as test sample. Place in clean containers, seal and label them.

5.1.2 Fruits and vegetables

The combined primary sample is reduced to ca. 500 g, from which shell, seed, peel, stem, root, and coronal has been removed (do not wash by water). Collect the edible parts, and blend and mix thoroughly in a high speed blender. Divide into two equal portions. Each portion is placed in a clean container, which is sealed and labeled, as the test sample.

5.1.3 Meat and meat products, nuts

Take representative approximately 1 kg of sample. The edible parts are collected, blended and homogenized. Divide into two equal portions. Each portion is placed in a clean container, which is sealed and labeled, as the test sample.

5.2 Storage of test samples

The test samples of cereals, tea, nuts, vinegar, honey and honey products should be stored between 0 ℃～4 ℃. The test samples of other one should be stored below －18 ℃.

While sampling and sample preparation, precaution must be taken to avoid contamination or any factors that may cause the change of residue content.

6 Procedure

6.1 Extraction

6.1.1 Vegetables, fruits and cereals

Weight 20 g(accurate to 0.1 g) of vegetable and fruits, 10 g(accurate to 0.1 g) of cereals into a 50 mL stoppered plastic centrifuge tube, then add 20 mL ethyl acetate + cyclohexane(3.7), stopper the tubes and vortex for 30 s and extract with ultrasonic machine for 20 min, and then centrifuge at 4 000 r/min for 3 min, transfer the supernatant to another clean tube, and repeat the extraction pro-

cedure with 20 mL ethyl acetate + cyclohexane(3.7)again. The supernatants are passed through a column of anhydrous sodium sulfate(4.7) to remove the water, collect the effluent into a 150 mL concentrate bottle and condense to nearly dry by a rotary evaporator with a 40 ℃ water bath. Dissolve the residue with 10.0 mL of ethyl acetate + cyclohexane (1 + 1), filter through 0.45 μm membrane filter and wait for purification.

6.1.2 Tea

Weight 2.5 g, (accurate to 0.1 g) of samples, into a 50 mL stoppered plastic centrifuge tube, add 10 mL distilled water, stand for 1 h. then add 20 mL Ethyl acetate + cyclohexane(3.8), stopper the tubes and vortex for 30 s and extract with ultrasonic machine and back procedure see 6.1.1.

6.1.3 Vinegar and honey

Rinse the C_{18} SPE column with 2 mL of *n*-hexane(3.4), 2 mL of trichloromethane(3.5) and 3 mL of distilled water before starting. Weight 2.5 g of samples into a plastic centrifuge tube, add 5 mL of distilled water and vortex for 30 s, Transfer the above solution into column, control the flow at 1 drop per 2 s. Then elute with 2 mL of distilled water, discard the rinse liquid, make vacuum for 2 min, add 2.5 mL of *n*-hexane, collect the eluates into a 1.0 mL centrifuge tube, then evaporated to dryness at 40 ℃ under a stream of nitrogen, and dissolved in 1 mL ethyl acetate for determination.

6.1.4 Walnut, rabbit meat, chicken liver and shrimp

Weight 10 g of sample into 50 mL stoppered plastic centrifuge tube, then add 20 mL of acetonitrile (3.3), stopper the tubes and vortex for 30 s and extract with ultrasonic machine for 20 min, and then centrifuge at 4 000 r/min for 3 min, transfer the supernatant to another clean tube, and repeat the extraction procedure with 20 mL of acetonitrile(3.3)again. The supernatants are passed through a column of anhydrous sodium sulfate(4.7)to remove the water, collect the effluent into a 150 mL concentrate bottle and condense to 1 mL～2 mL by a rotary evaporator with a 40 ℃ water bath, then add 5 mL ethyl acetate + cyclohexane(3.7) and condense again to nearly dry by a rotary evaporator with a 40 ℃ water bath, Dissolve the residue with 10 mL of ethyl acetate + cyclohexane (3.7), filter through 0.45 μm membrane filter and wait for purification.

6.2 GPC Cleaning-up

6.2.1 GPC operating condition

a) GPC column: 300 mm × 20 mm (i.d.), Bio Beads S-X3 or equivalent;

b) Mobile phase: Cyclohexane-ethyl acetate (3.7);

c) Flow rate: 4.7 mL/min;

d) Injection volume at sample loop:5 mL;

e) Time of collecting the eluate:7.5 min～12.5 min.

6.2.2 GPC Cleaning-up step

Transfer the waiting for purification solution acquired at 6.1 1.0 mL into the column of GPC, Cleaning-up with the parameters of section 6.2.1. The elution are collected into a clean tube and evaporated to dryness at 35 ℃ under a stream of nitrogen, and redissolved in 1 mL ethyl acetate for GC-FTD or GC-MS determination.

6.3 GC Determination

6.3.1 GC operation conditions

a) Column:CP-1301 30 m × 0.25 mm (i.d.) × 0.25 μm or equivalent;

b) Column temperature:50 ℃ (1 min) $\xrightarrow{20\ ℃/min}$ 220 ℃;

c) Injection port temperature:260 ℃;

d) FTD temperature:280 ℃;

e) Carrier gas:nitrogen(purity≥99.995%, flow rate:1 mL/min);

f) Gas flow rate:hydrogen:3.0 mL/min, air:150 mL/min, purge:30 mL/min;

g) Constant current:1 pA;

h) Injection mode:Splitless, open the valve after 1 min;

i) Injection volumn:1 μL.

6.3.2 GC determination

According to the approximate concentration of cadusafos residues in sample solution, select the standard working solution with similar peak area to that of the sample solution. The standard working solution should be randomly injected in between the injection of sample solution of equal volumn. Under the above GC conditions, the retention time of cadusafos is about 29.0 min. The chromatogram of the cadusafos standard is shown by Figure A.1 in annex A.

According to the operating condition assigned in 6.3.1, analyze the standard solution and sample solution, if there is a peak appeared at the same retention time for both of the sample solution and standard working solution, the GC-MS confirmation test should be conducted.

6.4 GC-MS confirmation

6.4.1 GC-MS operation conditions

a) Chromatographic column:30 m×0.25 mm (i.d.),0.25 μm film thickness,and HP-5MS silica capillary column or equivalent;

b) Column temperature:50 ℃ (1 min) $\xrightarrow{10\ ℃/min}$ 280 ℃ (10 min);

c) Injection port temperature:250 ℃;

d) Interface temperature:280 ℃;

e) Electron ionization mode:EI;

f) Ionization energy:70 eV;

g) Carrier gas:Helium,purity≥99.999%,flow rate 1 mL/min;

h) Injection mode:Splitless,open the valve after 0.75 min;

i) Injection volume:1 μL;

j) Determination mode:SIM;

k) Selected monitoring ions (m/z): 159,213,215,270;

l) Solvent protection delay:5.0 min.

6.4.2 GC-MS confirmation

If the retention times of sample chromatogram peaks are consistent with the standard,and after subtracted background noise,the relative intensity ratios of each qualitative ions are also consistent with similar concentration standard,and the similarity degree of their relative abundance ratio in permitted tolerance(see table 1),we can confirm that there are corresponding analyte in the sample. The GC-MS selected ion chromatogram and mass spectrum of the cadusafos standard are shown respectively by figure B.1 and figure B.2 in annex B.

Table 1 —Maximum permitted tolerance for relative ion intensities of confirmation

Relative intensity/%	>50	>20～50	>10～20	≤10
Permitted relative tolerances/%	±20	±25	±30	±50

6.5 Blank test

Blank test will be conducted according to the procedures above without sample addition.

7 Calculation and expression of the result

Calculate the content of cadusafos residue in the test sample by GC processor or according to the formula(1):

$$X = \frac{A \cdot c_s \cdot V}{A_s \cdot m} \quad \cdots\cdots (1)$$

where:

X—the residue content of cadusafos in the test sample, mg/kg;

A—the peak area of cadusafos in the sample solution;

c_s—the concentration of cadusafos in the standard working solution, μg/mL;

V—the final volume of the sample solution, mL;

A_s—the peak area of cadusafos in the standard working solution;

m—the corresponding mass of the test sample representing the final sample solution, g.

8 Limit of determination and recovery

8.1 Limit of determination

The limit of determination of peach, apple, asparagus, scallion, murphy, corn, buckwheat, Rabbit meat and shrimp are 0.005 mg/kg, the limit of determination of walnut, honey, chicken liver, vinegar and tea leaf are 0.01 mg/kg.

8.2 Recovery

The recovery of this method see table 2.

Table 2 —The recovery of this method

Name of test sample	Added consistence/(mg/kg)	Recovery/%
peach	0.005～0.020	89.9～104.0
apple	0.005～0.020	84.8～107.0
asparagus	0.005～0.020	89.6～102.0
scallion	0.005～0.020	87.0～98.1
murphy	0.005～0.020	87.8～92.5

Table 2（continue）

Name of test sample	Added consistence/（mg/kg）	Recovery/%
corn	0.005～0.020	92.8～105.0
buckwheat	0.005～0.020	93.8～103.6
tea leaf	0.01～0.050	93.0～99.0
honey	0.01～0.050	93.0～99.0
vinegar	0.01～0.050	90.0～104.0
walnut	0.01～0.050	84.3～105.0
rabbit meat	0.005～0.020	93.5～110.0
chicken liver	0.01～0.050	90.7～99.0
shrimp	0.005～0.020	92.5～98.0

Annex A
（informative）
Chromatogram of the cadusafos standard derivative

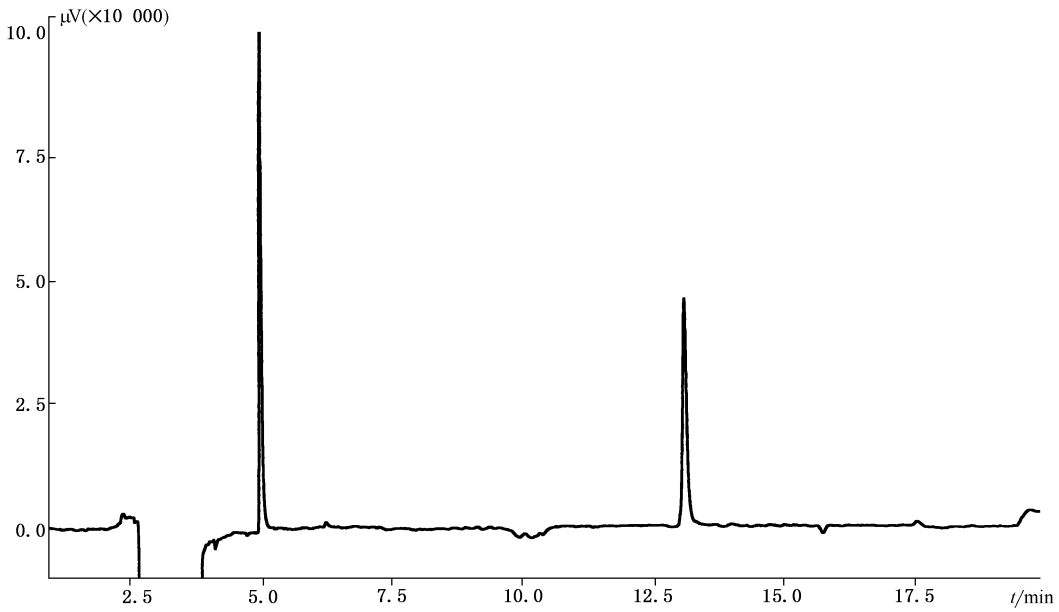

Figure A.1—Chromatogram of the cadusafos standard

Annex B
（informative）
GC-MS selected ion chromatogram and mass spectrum of the cadusafos standard

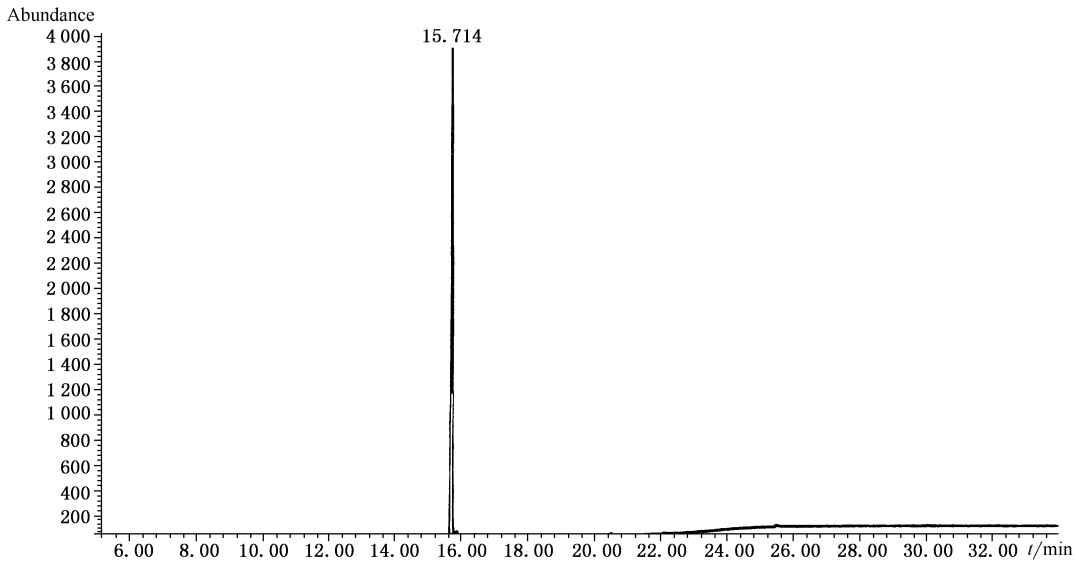

Figure B.1—GC-MS selected ion chromatogram of the cadusafos standard（1.0 μg/mL）

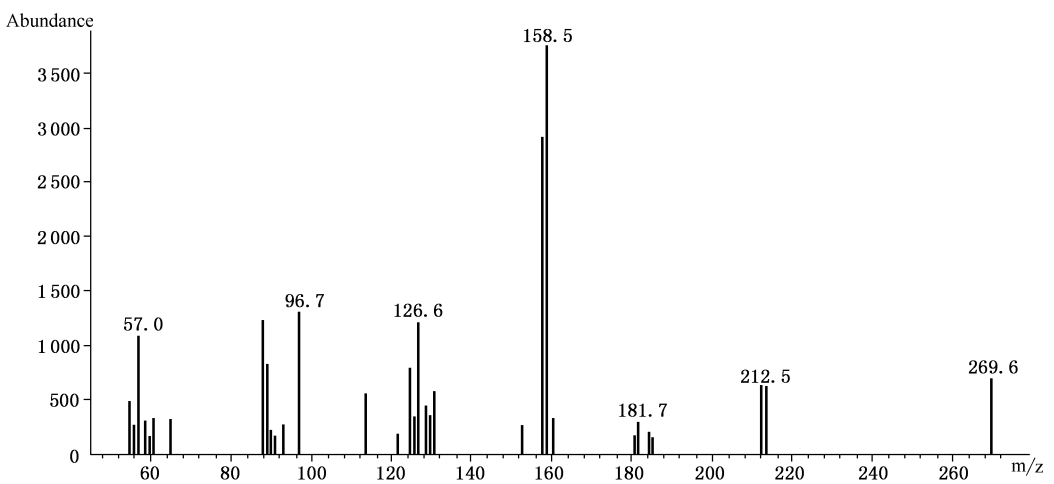

Figure B.2—Gas chromatogram and mass spectrum of the cadusafos standard

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