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中华人民共和国出入境检验检疫行业标准

SN/T 2234—2008

进出口食品中丙溴磷残留量检测方法 气相色谱法和气相色谱-质谱法

Determination of profenofos residue in food for import and export—GC and GC-MS method

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前　言

本标准的附录A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。

本标准由中华人民共和国天津出入境检验检疫局、中华人民共和国深圳出入境检验检疫局、中华人民共和国陕西出入境检验检疫局、中华人民共和国重庆出入境检验检疫局负责起草。

本标准主要起草人：王云凤、葛宝坤、林安清、常春艳、陈其勇、高建会、肖亚兵、蓝芳、李建华、王国民、吴卫东、何强、张雷。

本标准系首次发布的出入境检验检疫行业标准。

进出口食品中丙溴磷残留量检测方法 气相色谱法和气相色谱-质谱法

1 范围

本标准规定了食品中丙溴磷残留量测定的制样、气相色谱测定和气相色谱-质谱测定及确证方法。

本标准适用于西兰花、甘蓝、胡萝卜、生姜、苹果、梨、花生、茶叶、牛奶、蜂蜜、大米、大豆、鸡肉、兔肉、鱼肉、虾肉中丙溴磷残留量的测定和确证。

2 原理

试样经乙腈提取，串联的丙基乙二胺键合硅胶固相萃取柱和石墨化碳黑固相萃取柱净化，气相色谱、气相色谱-质谱确证测定，外标法定量。

3 试剂和材料

除另有规定外，所有试剂均为分析纯，水为二次蒸馏水。

3.1 丙酮：残留级。

3.2 乙腈：残留级。

3.3 正己烷：残留级。

3.4 乙酸乙酯：残留级。

3.5 无水硫酸钠：650 ℃灼烧 4 h，置入干燥器中冷却，备用。

3.6 氯化钠。

3.7 乙酸乙酯-正己烷混合溶液(1+4，体积比)：取 200 mL 乙酸乙酯，加入 800 mL 正己烷，摇匀备用。

3.8 氢氧化钠(2 mol/L)：称取 8.0 g 氢氧化钠溶于 100 mL 水中。

3.9 磷酸盐缓冲溶液(0.5 mol/L，pH7.0)：称取 52.7 g 磷酸氢二钾和 30.2 g 磷酸二氢钾，加水 1 L 溶解，用 2 mol/L 氢氧化钠调 pH 值至 7.0。

3.10 丙溴磷标准品：(Profenofos，$C_{11}H_{15}BrClO_3PS$，CAS 号：41198-08-7)纯度大于 97%。

3.11 丙溴磷标准溶液的制备：准确称取适量标准品，用丙酮溶解，配置成浓度为 1.0 mg/mL 的标准储备液。根据需要再用丙酮稀释成适当浓度的标准工作溶液。保存在 4 ℃冰箱中。

3.12 石墨化碳黑固相萃取柱：Envi-Carb 3 mL，500 mg，或相当者。

3.13 丙基乙二胺键合硅胶固相萃取柱：Primary Secondary Amine，PSA3 mL，500 mg，或相当者。

4 仪器与设备

4.1 气相色谱-质谱仪：配有电子轰击电离源(EI)。

4.2 气相色谱仪：配电子捕获检测器(ECD)。

4.3 固相萃取装置。

4.4 均质器。

4.5 旋转蒸发器。

4.6 氮气浓缩仪。

4.7 100 mL 具塞量筒。

4.8 离心机。

5 试样制备与保存

5.1 试样制备

5.1.1 粮谷

取有代表性样品500 g,用磨碎机全部磨碎并通过2.0 mm圆孔筛。混匀,均分成两份作为试样,分别装入洁净的容器内,密闭,标明标记。

5.1.2 水果和蔬菜

取有代表性样品500 g,将其可食部分切碎后,依次用食品捣碎机将样品加工成浆状,混匀,装入洁净的容器内,密闭,标明标记。

5.1.3 肉及肉制品

取有代表性样品500 g,取可食部分经捣碎机充分捣碎均匀,装入洁净的容器内,密闭,标明标记。

5.1.4 蜂蜜及蜂制品

取有代表性样品500 g,对无结晶的蜂蜜样品将其搅拌均匀;对有结晶析出的蜂蜜样品,在密闭情况下,将样品瓶移至不超过60 ℃的水浴中温热,振荡,待样品全部溶化后搅匀,迅速冷却至室温,在溶化时注意防止水分挥发,装入洁净的容器内,密闭,标明标记。

5.1.5 茶叶

取有代表性样品量500 g,用磨碎机全部磨碎并通过2.0 mm圆孔筛。混匀,装入洁净的容器内,密闭,标明标记。

5.1.6 坚果

取有代表性样品500 g,用磨碎机全部磨碎。混匀,均分成两份作为试样,分别装入洁净的容器内,密闭,标明标记。

5.1.7 水产品

取有代表性样品500 g,取可食部分经捣碎机充分捣碎均匀,装入洁挣的容器内,密闭,标明标记。

5.2 试样保存

粮谷类试样于0 ℃～4 ℃保存;水果和蔬菜及其他类试样于－18 ℃以下冷冻保存。在抽样及制样的操作过程中,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、牛奶样品的提取

除牛奶取样10 g外,其余样品称取5 g(精确至0.01 g),于100 mL离心管中,加入20 mL乙腈,均质1 min,4 000 r/min离心5 min,将上清液转入已加入20 g氯化钠(3.6)、50 mL磷酸缓冲溶液(3.9)的100 mL具塞量筒内,残渣再加入20 mL乙腈均质、离心,转移上清液至100 mL具塞量筒内,盖上塞子剧烈振摇5 min,静置分层,取上层乙腈过无水硫酸钠(3.5)后,40 ℃旋转蒸发近干,残渣用2 mL乙酸乙酯-正己烷(3.7)溶解。

6.1.2 大米、大豆、茶、花生、蜂蜜样品的提取

称取样品5 g(精确至0.01 g),于100 mL离心管中,先加10 mL水浸泡30 min,蜂蜜加10 mL水,40 ℃振摇15 min,加入20 mL乙腈,均质1 min,4 000 r/min离心5 min,以下步骤按6.1.1操作。

6.1.3 鲫鱼、虾、兔肉、鸡肉等样品的提取

取样5 g(精确至0.01 g),于100 mL离心管中,加入20 mL乙腈,均质1 min,离心5 min,将上清液转入已加入20 g氯化钠(3.6)、50 mL磷酸缓冲溶液(3.9)的100 mL具塞量筒内,残渣再加入20 mL乙腈均质、离心,转移上清液至100 mL具塞量筒内,盖上塞子剧烈振摇5 min,静置分层,取上层乙腈于

另一个 100 mL 具塞量筒内，加 20 mL 正己烷，振摇分层，去掉上层正己烷，乙腈过无水硫酸钠(3.5)后，40 ℃旋转蒸发近干，残渣用 2 mL 乙酸乙酯-正己烷(3.7)溶解。

6.2 净化

将 PSA 萃取柱连接在 Envi-Carb 小柱下端，用 6 mL 乙酸乙酯-正己烷混合溶液(3.7)活化，转移提取液过串联柱，控制流速在小于 2 mL/min，然后再用 3×2 mL 乙酸乙酯-正己烷混合溶液(3.7)洗涤鸡心瓶，并将洗涤液过串联柱，收集全部流出液，在 40 ℃氮气流下吹至近干，1 mL 丙酮溶解，供 GC 或 GC-MS 测定。

6.3 测定

6.3.1 气相色谱条件

a) 色谱柱：DB-17MS 石英毛细管柱，30 m×0.25 mm(内径)×0.25 μm，或相当者；

b) 升温条件：150 ℃保持 2 min，然后以 6 ℃/min 的程序升温至 270 ℃，保持 25 min；

c) 进样口温度 200 ℃，检测器温度 300 ℃；

d) 载气：氮气，纯度大于 99.999%；

e) 流速：1 mL/min；

f) 进样方式：分流进样，分流比：10∶1；

g) 进样量：1.0 μL。

6.3.2 气相色谱-质谱条件

a) 色谱柱：DB-1701 石英毛细管柱，30 m×0.25 mm(内径)×0.15 μm 或相当者；

b) 色谱柱温度：40 ℃保持 1 min，然后以 30 ℃/min 的程序升温至 130 ℃，再以 5 ℃/min 升至 250 ℃，保持 5 min；

c) 载气：氦气，纯度大于 99.999%；

d) 进样口温度：280 ℃；

e) 流速为 1.0 mL/min；

f) 进样量：1 μL；

g) 进样方式：无分流进样，0.75 min 后打开分流阀；

h) 电子轰击源：70 eV；

i) 离子源温度：180 ℃；

j) 接口温度：280 ℃；

k) 监测离子(m/z)：268，296，338，373。

6.3.3 定量测定

根据样品中丙溴磷含量情况，选定峰面积相近的标准工作溶液。标准工作溶液和样液中丙溴磷响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行测定。在上述色谱条件下，丙溴磷在气相色谱上的保留时间为 19.28 min，色谱图参见附录 A 中图 A.1。丙溴磷在气相色谱-质谱上的保留时间为 25.05 min。标准物质的总离子流色谱图参见附录 A 中图 A.2，全扫描质谱图参见附录 A 中图 A.3。

丙溴磷的定性离子(m/z)：268，296，373，定量离子(m/z)：338，其监测离子(m/z)的丰度比是 268∶296∶338∶373＝76∶42∶100∶43。在扣除背景后的样品质谱图中，若所选择的离子均出现，经过对比所选择离子的丰度比与标准品对应离子的丰度比，其值在允许范围内(允许范围见表 1)则可判断样品中存在对应的被测物。

表 1 定性确证时相对离子丰度最大允许误差

相对丰度/%	>50	>20～50	>10～20	<10
GC/MS 时相对离子丰度最大允许误差/%	±10	±15	±20	±50

6.4 空白实验

除不加试样外，均按上述测定步骤进行。

6.5 结果计算

按式(1)计算试样中丙溴磷的含量：

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

式中：

X——试样中丙溴磷的含量，单位为毫克每千克(mg/kg)；

A——试样中丙溴磷的色谱峰面积；

c_s——标准工作溶液中丙溴磷的浓度，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——标准工作溶液中丙溴磷的色谱峰面积；

m——最终样液所代表的试样量，单位为克(g)。

7 测定低限、回收率

7.1 测定低限

本方法的测定低限为 0.01 mg/kg。

7.2 回收率

回收率数据见表 2。

表 2 不同样品中丙溴磷农药的添加浓度和回收率范围

样品名称	添加浓度/(mg/kg)	回收率/%	
		GC	GC-MS
甘蓝	0.01	69.0～89.0	76.0～92.0
	0.02	78.0～92.0	79.0～92.0
	0.05	78.0～96.0	76.0～92.0
西兰花	0.01	74.0～85.0	69.0～88.0
	0.02	82.5～90.0	74.0～87.0
	0.05	76.0～96.0	76.0～94.0
胡萝卜	0.01	69.0～85.0	75.0～86.0
	0.02	76.0～88.0	74.0～86.0
	0.05	78.0～96.0	76.0～92.0
生姜	0.01	68.0～85.0	76.0～88.0
	0.02	84.0～92.0	78.0～87.5
	0.05	72.0～90.0	76.0～84.0
梨	0.01	69.0～92.0	74.0～85.0
	0.02	77.0～89.0	77.0～92.0
	0.05	76.0～96.0	79.0～92.0
苹果	0.01	72.0～93.0	70.0～96.0
	0.02	76.0～89.0	71.0～87.0
	0.05	78.0～96.0	76.0～94.0

表 2（续）

样品名称	添加浓度/(mg/kg)	回收率/%	
		GC	GC-MS
大米	0.01	69.0～85.0	70.0～87.0
	0.02	84.0～94.5	77.0～83.0
	0.05	78.0～96.0	76.0～96.0
大豆	0.01	69.0～82.0	76.0～86.0
	0.02	79.5～90.0	74.0～86.0
	0.05	78.0～94.0	76.0～96.0
蜂蜜	0.01	76.0～90.0	69.0～86.0
	0.02	77.0～87.0	75.0～89.0
	0.05	76.0～98.0	76.0～92.0
花生	0.01	68.0～82.0	75.0～89.0
	0.02	77.0～87.0	77.0～86.0
	0.05	74.0～86.0	74.0～96.0
牛奶	0.01	75.0～86.0	74.0～92.0
	0.02	81.0～92.0	74.0～91.5
	0.05	80.0～94.0	76.0～94.0
茶	0.01	68.0～86.0	72.0～88.0
	0.02	77.0～89.0	73.5～89.0
	0.05	72.0～88.0	76.0～96.0
鲫鱼	0.01	68.0～86.0	75.0～87.0
	0.02	78.0～87.0	74.5～83.0
	0.05	74.0～90.0	76.0～92.0
虾	0.01	74.0～88.0	69.0～87.0
	0.02	75.0～86.0	79.0～91.0
	0.05	74.0～88.0	76.0～92.0
鸡肉	0.01	68.0～85.0	76.0～87.0
	0.02	78.0～84.0	73.0～87.5
	0.05	76.0～90.0	76.0～88.0
兔肉	0.01	69.0～84.0	75.0～86.0
	0.02	76.5～87.0	73.5～83.0
	0.05	76.0～88.0	70.0～92.0

附 录 A
（资料性附录）
丙溴磷标准品的气相色谱图和气相色谱-质谱图

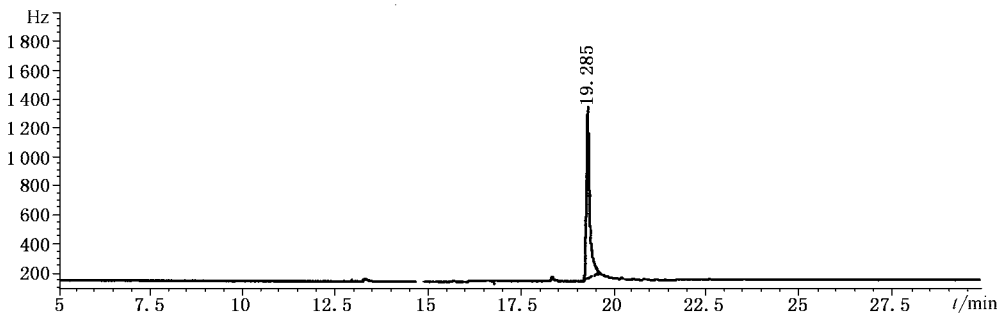

图 A.1 丙溴磷标准品的气相色谱图

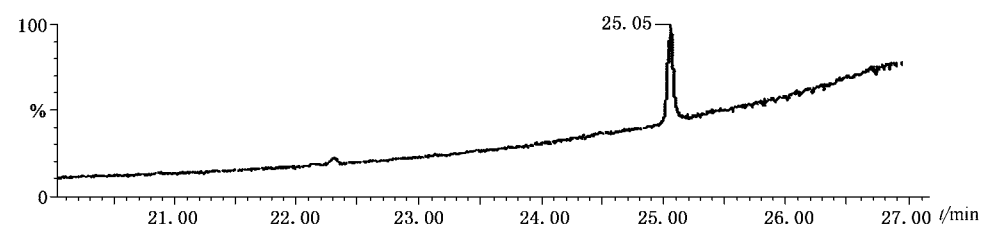

图 A.2 丙溴磷标准品气相色谱-质谱总离子流色谱图

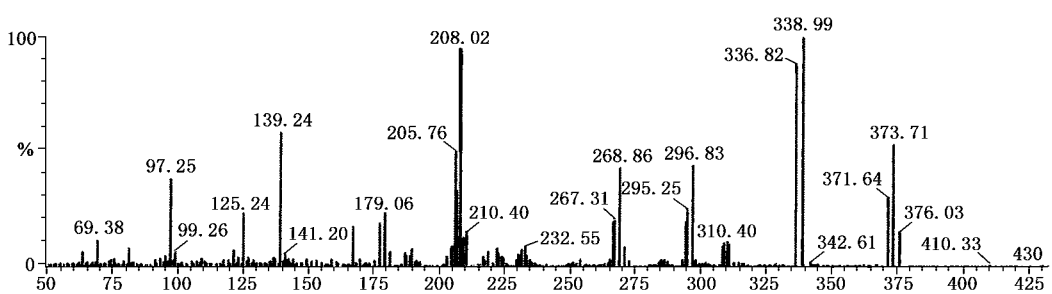

图 A.3 丙溴磷标准品的全扫描气相色谱-质谱图

Foreword

Annex A of this standard is an informative annex.

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

This standard was drafted by Tianjin Entry-Exit Inspection and Quarantine Bureau, Shenzhen Entry-Exit Inspection and Quarantine Bureau, Shanxi Entry-Exit Inspection and Quarantine Bureau, Chongqing Entry-Exit Inspection and Quarantine Bureau.

This standard was mainly drafted by Wang yunfeng, Ge baokun, Lin anqing, Chang chunyan, Chen qiyong, Gao jianhui, Xiao yabin, Lan fang, Li jianhua, Wang guomin, Wu weidong, He qiang, Zhang lei.

This standard is a professional standard for entry-exit inspection and quarantine promulgated for the first time.

Determination of profenofos residue in food for import and export—GC and GC-MS method

1 Scope

This standard specifies the sample prepration, the method of determination and confirmation of profenofos residues in food by gas chromatography and gas chromatography-mass spectrum.

This standard is applicable for the determination and confirmation of profenofos residues in broccoli, cabbage, carrot, ginger, apple, pear, peanut, tea, milk, honey, rice, soybean, chicken, rabbit meat, crucian and shrimp.

2 Principle

The residus of profenofos in the test samples are extracted with acetonitrile and cleaned up with PSA and Envi-Carb solid phase extraction column. Determined by GC, GC-MS, using external method.

3 Reagents and materials

Unless otherwise specified, all reagent used should be analytical grade, "water" is the distilled water.

3.1 Acetone: grade for residue analysis.

3.2 Acetonitrile: grade for residue analysis.

3.3 *n*-hexane: grade for residue analysis.

3.4 Ethyl acetate: grade for residue analysis.

3.5 Sodium sulfate: Ignite at 650 ℃ for 4 h, cool to room temperature and store in desiccator container.

3.6 Sodium chloride.

3.7 Ethyl acetate-*n*-Hexane mixed solution(1+4, *V*/*V*): Mix 200 mL of ethyl acetate with 800 mL *n*-Hexane.

3.8 Sodium hydroxide(2 mol/L): Dissolve 8.0 g sodium hydroxide in 100 mL water.

3.9 Phosphate buffer solution(0.5 mol/L,pH 7.0):Dissolve 52.7g dipotassium hydrogenphosphate (K_2HPO_4) and 30.2 g potassium dihydrogenphosphate(KH_2PO_4)in approximately 1 L of water. Adjust the pH of the solution to 7.0 with 2 mol/L sodium hydroxide.

3.10 Profenofos standard:($C_{11}H_{15}BrClO_3PS$,CAS Number:41198-08-7)Purity large than 97%.

3.11 Standard stock solution:Accurately weigh appropriate amount of phosphate standard and dissolve with a little volume of acetone followed by a further dilution to the final concentration of 1.0 mg/mL. Then dilute the standard stock solution with acetone to make standard working solution of required concentration and stored at 4 ℃.

3.12 Envi-Carb solid phase extraction:500 mg,3 mL or equivalent.

3.13 Primary secondary amine(PSA)solid phase extraction:500 mg,3 mL or equivalent.

4 Apparatus and equipment

4.1 Gas chromatography combined with electron ionization mass spectrometry.

4.2 Gas chromatography combined with electron capture detector.

4.3 Solid phase extraction equipment.

4.4 Homogenizer.

4.5 Rotation evaporator.

4.6 Nitrogen evaporator with heated bath.

4.7 100 mL mixing cylinder with stopper.

4.8 Centrifuge.

5 Sample preparation and storage

5.1 Sample preparation

5.1.1 Cereal

Representative sample,about 500 g,which is crushed and passed through a 2.0 mm mesh sieve. The sample is mixed and divided into two equal portions and placed in a clean container,which is sealed

and labeled.

5.1.2 Fruit and vegetable:

Representative sample, about 500 g. Edible part is minced and prepare to be starchy with a blender. The sample is mixed and placed in a clean container, which is sealed and labeled.

5.1.3 Meat and meat product

Representative sample, about 500 g. Edible part is minced. The sample is mixed and placed in a clean container, which is sealed and labeled.

5.1.4 Honey

Representative sample, about 500 g. The sample which is not crystallized shall be stirred well to make homogeneous. If the sample is crystallized, it must be warmed in a water-bath below 60 ℃ with the sample bottle covered tightly, mix thoroughly when all sample has melted, then cool immediately to room temperature. If the course of sample melting, precautions must be taken to avoid evaporation of water from the sample. Place in a clean container, which is labeled and sealed.

5.1.5 Tea

Representative sample, about 500 g, which is crushed and passed through a 2.0 mm mesh sieve. The sample is mixed and placed in a clean container, which is sealed and labeled.

5.1.6 Nuts

Representative sample, about 500 g, which is crushed and passed through a 20 mesh sieve. The sample is mixed and divided into two equal portions and placed in a clean container, which is sealed and labeled.

5.1.7 Aquatic product

Representative sample, about 500 g. Edible part is minced. The sample is mixed and placed in a clean container, which is sealed and labeled.

5.2 Storage of test sample

The test samples of cereals should be stored at the temperature ranged from 0 ℃～4 ℃. The test samples of fruit, vegetable and other samples should be frozen and stored at the temperature below −18 ℃. In course of sampling and sample preparation, it must be taken to avoid contamination or any factors which may cause the change of residue content.

6 Precedure

6.1 Extraction

6.1.1 Extraction of vegetable, fruit, creamery

Weigh 5 g of the test sample (broccoli, cabbage, carrot, ginger, apple, pear) or 10 g creamery into 100 mLcentrifuge, add 20 mL acetonitrile. Homogenize for 1 min, centrifuge at 4 000 r/min for 5 minute, taking the upper liquid into 100 mL mixing cylinder with stopper, add 20 mL acetonitrile to centrifuge, homogenize and centrifuge again, combine acetonitrile solution to cylinder, Add 20 g sodium chloride(3.6)and 50 mL of phosphate buffer solution(3.9)into mixing cylinder with stopper. Shake 5 min sharply with stopper and stand still for layering. Collect the acetonitrile layer through sodium sulfate and evaporate to dryness at 40 ℃, then dissolved the residue with 2 mL ethyl acetate-n-Hexane mixed solution(3.7).

6.1.2 Extraction of rice, soybean, tea, peanut, honey

Weigh 5 g of the test sample(rice, soybean, tea, peanut,)into 100 mL centrifuge, add 10 mL water, for 30 min before adding acetonitrile, for honey, add 10 mL water and shake for 15 min at 40 ℃, add 20 mL acetonitrile to centrifuge, the follow procedure is same as 6.1.1.

6.1.3 Extraction of crucian, shrimp, rabbit meat, chicken

Weigh 5 g of the test sample into 100 mL centrifuge, add 20 mL acetonitrile. Homogenize for 1 min, centrifuge at 4 000 r/min for 5 minute, taking the upper liquid into 100 mL mixing cylinder with stopper. , add 20 mL acetonitrile to centrifuge, homogenize and centrifuge again, combine acetonitrile solution to cylinder, Add 20 g sodium chloride and 50 mL of phosphate buffer solution into mixing cylinder with stopper. Shake 5 min sharply with stopper and stand still for layering. Collect the acetonitrile layer to another 100 mL mixing cylinder, add 20mL *n*-hexane, shake and collect the acetonitrile layer through Sodium sulfate and evaporate to dryness at 40 ℃, then dissolved the residue with 2 mL ethyl acetate-n-Hexane mixed solution(3.7).

6.2 Clean up

Set the Envi-Carb SPE column below the PSA SPE column and put it on the solid phase extract vaccum manifold and mechanical pump. Wash the cartridge with 6 mL ethyl acetate-n-Hexane mixed solution(3.7), transfer the sample extraction into the cartridge, keeping flow speed under 2 mL/min. Wash the flask with 3×2 mL ethyl acetate-n-Hexane mixed solution(3.7), and transfer the wash solution into cartridge, collect the eluates and blow it dryness with nitrogen at 40 ℃, dissolve the residue and dilute exactly to 1.0 mL with acetone for GC or GC-MS determination.

6.3 Determination

6.3.1 GC operation conditions

a) Column: DB-17 MS fused quartz capillary column, 30 m × 0.25 mm (i.d.), film thickness 0.25 μm, or the equivalent;

b) Column temperature: 150 ℃ for 2 min, ramp at 6 ℃/min to 270 ℃, hold for 25 min;

c) Injection port temperature: 200 ℃, detection temperature: 300 ℃;

d) Carrier gas: N_2 (purity > 99.999%);

e) Flow rate: 1 mL/min;

f) Injection mode: Split injection, spilt ratio is 10 : 1;

g) Injection volumn: 1.0 μL.

6.3.2 GC-MS operating conditions

a) Column: DB-1701, 30 mx0.25 mm(i.d.)x0.15 μm or equivalent;

b) Temperature program: 40 ℃ (keep 1 min), 30 ℃/min to 130 ℃, 5 ℃/min to 250 ℃ (keep 5 min);

c) Carrier gas: He (purity > 99.999%);

d) Injection temperature: 280 ℃;

e) Flow rate of carrier gas: 1.0 mL/min;

f) Injection volume: 1 μL;

g) Injection mode: splitless, purge after 0.75 min;

h) Electron mode: EI, 70 eV;

i) Ion source temperature: 180 ℃;

j) Interface temperature: 280 ℃;

k) Monitor ion(m/z): 268, 296, 338, 373.

6.3.3 determination and confirmation

According to the approximate concentration of profenofos in the test sample solution, select the

standard working solution with similar peak area to that of sample solution. The responses of profenofos in the standard working solution and sample solution should be in the linear range of the instrumental detection. The standard working solution should be injected randomly in between the injections of sample solution of equal volume. Under the above GC conditions, the retention time of profenofos is about 19. 28 min. See FigA. 1 in annex A. The retention time of profenofos is about 25. 05 min under GC-MS conditions. Under the above GC/MS operating conditions, the total ion chromatogram and mass spectrum of the profenofos standard see FigA. 2 and FigA. 3 in annex A.

The ion for qualification are 268, 296, 373 and ion for quantification is 338 of profenofos. The monitoring ions ratio are 268 ∶ 296 ∶ 338 ∶ 373 = 76 ∶ 42 ∶ 100 ∶ 43. According to the GC/MS operating conditions, and subtracted from background compensation, selected ions are all present and the relative ion abundance of the selected ions according with that of the calibration standard, at comparable concentrations, within the tolerances(seen table 1).

Table 1—Maximum permitted tolerances for relative ion abundance while confirmation

Relative bundance/%	>50	>20～50	>10～20	<10
Permitted tolerances/%	±10	±15	±20	±50

6.4 Blank test

The operation of the blank test is the same as that described in the method of determination, but with omission of sample addition.

6.5 Calculation of the result

Calculation the content of profenofos residue in the test sample according the formula(1):

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

Where:

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

A—the peak area of profenofos in the test Sample;

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

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

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

m—Mass of test sample, g.

7 Limit of determination and recovery

7.1 Limit of determination

The limit of determination of this method is 0. 01 mg/kg.

7.2 Recovery

Range of recoveries are listed in table 2.

Table 2—The fortified levels and the range of recoveries of profenofos in the test sample

Sample name	fortified levels/(mg/kg)	Recoveries/%	
		GC	GC-MS
Cabbage	0.01	69.0～89.0	76.0～92.0
	0.02	78.0～92.0	79.0～92.0
	0.05	78.0～96.0	76.0～92.0
Broccoli	0.01	74.0～85.0	69.0～88.0
	0.02	82.5～90.0	74.0～87.0
	0.05	76.0～96.0	76.0～94.0
Carrot	0.01	69.0～85.0	75.0～86.0
	0.02	76.0～88.0	74.0～86.0
	0.05	78.0～96.0	76.0～92.0
Ginger	0.01	68.0～85.0	76.0～88.0
	0.02	84.0～92.0	78.0～87.5
	0.05	72.0～90.0	76.0～84.0
Pear	0.01	69.0～92.0	74.0～85.0
	0.02	77.0～89.0	77.0～92.0
	0.05	76.0～96.0	79.0～92.0
Apple	0.01	72.0～93.0	70.0～96.0
	0.02	76.0～89.0	71.0～87.0
	0.05	78.0～96.0	76.0～94.0
Meat	0.01	69.0～85.0	70.0～87.0
	0.02	84.0～94.5	77.0～83.0
	0.05	78.0～96.0	76.0～96.0
Soybean	0.01	69.0～82.0	76.0～86.0
	0.02	79.5～90.0	74.0～86.0
	0.05	78.0～94.0	76.0～96.0
Honey	0.01	76.0～90.0	69.0～86.0
	0.02	77.0～87.0	75.0～89.0
	0.05	76.0～98.0	76.0～92.0
Peanut	0.01	68.0～82.0	75.0～89.0
	0.02	77.0～87.0	77.0～86.0
	0.05	74.0～86.0	74.0～96.0

Table 2（Continued）

Sample name	fortified levels/（mg/kg）	Recoveries/%	
		GC	GC-MS
Milk	0.01	75.0～86.0	74.0～92.0
	0.02	81.0～92.0	74.0～91.5
	0.05	80.0～94.0	76.0～94.0
Tea	0.01	68.0～86.0	72.0～88.0
	0.02	77.0～89.0	73.5～89.0
	0.05	72.0～88.0	76.0～96.0
Crucian	0.01	68.0～86.0	75.0～87.0
	0.02	78.0～87.0	74.5～83.0
	0.05	74.0～90.0	76.0～92.0
Shrimp	0.01	74.0～88.0	69.0～87.0
	0.02	75.0～86.0	79.0～91.0
	0.05	74.0～88.0	76.0～92.0
Chicken	0.01	68.0～85.0	76.0～87.0
	0.02	78.0～84.0	73.0～87.5
	0.05	76.0～90.0	76.0～88.0
Rabbit meat	0.01	69.0～84.0	75.0～86.0
	0.02	76.5～87.0	73.5～83.0
	0.05	76.0～88.0	70.0～92.0

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Annex A

(informative)

GC and GC-MS chromatogram of the profenofos standard

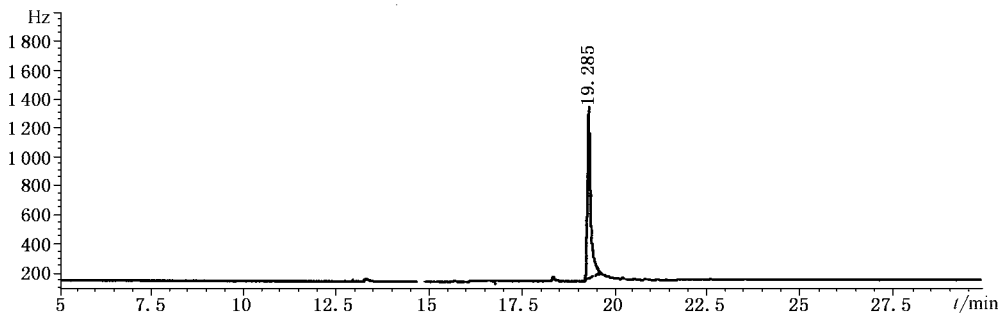

Figure A. 1—GC chromatograph of profenofos standard

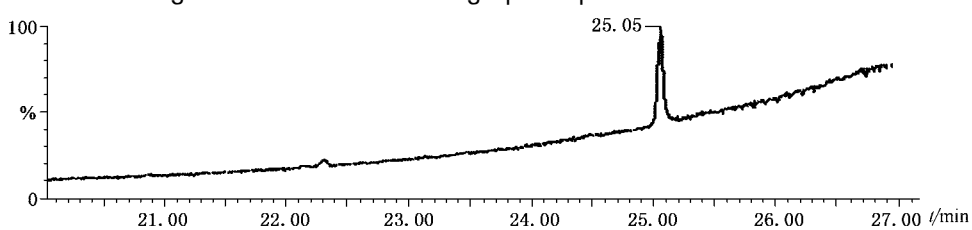

Figure A. 2—GC-MS sim chromatograph of profenofos standard

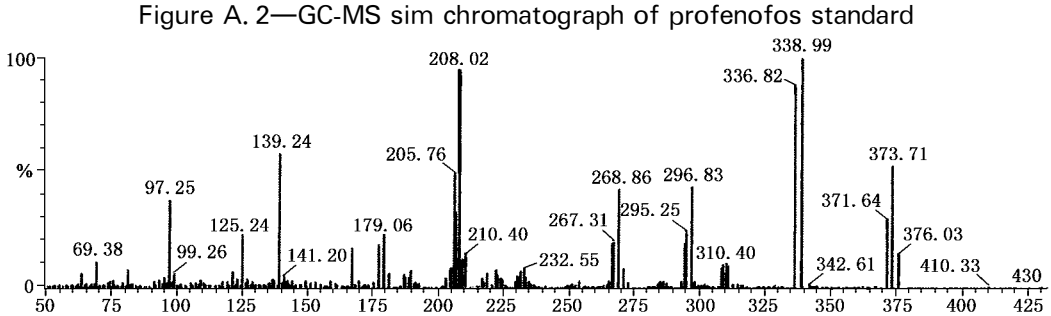

Figure A. 3—GC-MS scan chromatograph of profenofos standard