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SN

中华人民共和国出入境检验检疫行业标准

SN/T 0491—2019
代替 SN/T 0491—1995

出口植物源食品中苯氟磺胺残留量检测方法

Determination of dichlofluanid residues in foodstuffs origined from plant for export

2019-10-25 发布　　2020-05-01 实施

中华人民共和国海关总署　发布

前　言

本标准是按照 GB/T 1.1—2009 的要求进行编写。

本标准替代了 SN 0491—1995《出口粮谷中抑菌灵残留量检验方法》本标准与 SN 0491—1995 相比，主要技术变化如下：

——本标准增加了大米、小麦、大豆、玉米、梨、葡萄、番茄、黄瓜、马铃薯、蘑菇、干辣椒检验样品基质；

——增加了气相色谱-质谱确证部分；

——删除了抽样部分；

——优化了前处理方法；

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。

本标准由中华人民共和国海关总署提出并归口。

本标准起草单位：中华人民共和国石家庄海关

本标准主要起草人：李玮、王敬、艾连峰、马育松、陈瑞春、郭春海、张海超

本标准所代替标准的历次版本发布情况为：

——SN 0491—1995。

出口植物源食品中苯氟磺胺残留量检测方法

1 范围

本标准规定了出口植物源食品中苯氟磺胺残留量的气相色谱检测方法和气相色谱-质谱确证方法。

本标准适用于大米、小麦、大豆、玉米、糙米、梨、葡萄、马铃薯、番茄、黄瓜、蘑菇和干辣椒中苯氟磺胺残留量的测定。

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，仅所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

样品中的分析物用乙腈提取，提取液浓缩后经硅胶固相萃取柱净化后，供气相色谱测定及气相色谱-质谱确证，外标法定量。

4 试剂和材料

除另有规定外，试剂均为分析纯，水为 GB/T 6682 规定的一级水。

4.1 乙腈，高效液相色谱纯。

4.2 正己烷，高效液相色谱纯。

4.3 乙醚。

4.4 丙酮，高效液相色谱纯。

4.5 无水硫酸钠：650 ℃烘烤 4 h，在干燥器内冷却至室温，贮于密封瓶中备用。

4.6 正己烷-乙醚(3+47，V/V)：取 30 mL 正己烷(4.2)和 470 mL 乙醚(4.3)，混合均匀。

4.7 正己烷-乙醚(1+1，V/V)：取 100 mL 正己烷(4.2)和 100 mL 乙醚(4.3)，混合均匀。

4.8 标准品：苯氟磺胺(dichlofluanid，$C_9H_{11}Cl_2FN_2O_2S_2$，CAS NO.1085-98-9)，纯度大于等于 99%。

4.9 标准储备液：精确称取适量标准品(4.8)，用丙酮(4.4)配制成 1.0 mg/mL 的标准储备液，4 ℃下避光保存，保存期为 1 年。

4.10 标准中间工作液：取标准储备液(4.9)1.00 mL 至 100 mL 容量瓶中，用丙酮(4.4)定容至刻度，配制成标准中间工作液，浓度为 10.0 μg/mL，4 ℃下避光保存，保存期为 3 个月。

4.11 标准工作溶液：根据实验需要吸取适量的标准中间溶液(4.10)，用丙酮(4.4)稀释成适当浓度的标准工作溶液，现用现配。

4.12 硅胶固相萃取柱：500 mg，6 mL；或相当者。

4.13 微孔滤膜：0.22 μm，有机相型。

5 仪器和设备

5.1 气相色谱仪:配有电子捕获检测器(ECD)。
5.2 气相色谱串联质谱仪:配有电子轰击电离源(EI)。
5.3 组织捣碎机。
5.4 电子天平:感量分别为 0.01 g 和 0.1 mg。
5.5 离心机:转速不低于 5 000 r/mn。
5.6 均质器:转速不低于 15 000 r/min。
5.7 氮吹仪。
5.8 旋转蒸发仪。
5.9 固相萃取装置。
5.10 聚丙烯离心管:50 mL 和 15 mL,具塞。
5.11 10 mL 玻璃刻度试管。
5.12 鸡心瓶:120 mL。

6 样品制备和保存

6.1 制样要求

在制样的过程中,应防止样品受到污染或发生残留物含量的变化。

6.2 试样制备

6.2.1 梨、葡萄、马铃薯、番茄、黄瓜、蘑菇和干辣椒

取代表性样品 500 g,梨、番茄、黄瓜和干辣椒取全果去柄;葡萄取全果;马铃薯和蘑菇取整颗,用捣碎机将样品加工成浆状,混匀。试样均分为两份,装入洁净的容器内,密封并标明标记。于−18 ℃以下冷冻保存。

6.2.2 大米、玉米、小麦、大豆和糙米

取有代表性样品 500 g,取整粒,用粉碎机粉碎,混匀。试样均分为两份,装入洁净的容器内,密封并标明标记。常温下保存。

7 测定步骤

7.1 提取

7.1.1 梨、葡萄、马铃薯、番茄、黄瓜和蘑菇

称取试样约 5 g(精确到 0.01 g)于 50 mL 离心管中,加入 10 g 无水硫酸钠(4.5),20 mL 乙腈(4.1),15 000 r/min 均质提取 2 min,4 000 r/min 离心 10 min,将上清液经装有 10 g 无水硫酸钠(4.5)的漏斗过滤至 120 mL 鸡心瓶中。再用 15 mL 乙腈重复以上提取过程,合并提取液于同一鸡心瓶中,于 45 ℃水浴浓缩至干,待净化。

7.1.2 大米、玉米、小麦、大豆、糙米和干辣椒

称取干辣椒试样约 2 g、大米、玉米、小麦、大豆和糙米试样约 5 g(精确到 0.01 g)于 50 mL 离心管

中，加 10 mL 水润湿样品，混匀 2 min，静置 30 min 后加入 10 g 无水硫酸钠(4.5)，20 mL 乙腈(4.1)，15 000 r/min 均质提取 2 min，4 000 r/min 离心 10 min，将上清液经装有 10 g 无水硫酸钠(4.5)的漏斗过滤至 120 mL 鸡心瓶中。再用 15 mL 乙腈重复以上提取过程，合并提取液于同一鸡心瓶中，于 45 ℃水浴浓缩至干，待净化。

7.2 净化

用 5 mL 正己烷(4.2)溶解 7.1 步骤中所获得的浓缩液。用 5 mL 正己烷(4.2)预淋洗硅胶固相萃取柱(4.12)，将样品提取液通过固相萃取柱，用 6 mL 正己烷-乙醚(4.6)溶液淋洗固相萃取柱，弃去全部流出液，再用 2 mL 正己烷-乙醚(4.7)溶液洗脱，收集全部流出液，于 45 ℃氮气吹干。用 2.0 mL 正己烷(4.2)溶解，过 0.22 μm 有机相微孔滤膜后，供气相色谱测定和气相色谱-质谱确证。

7.3 气相色谱测定

7.3.1 气相色谱参考条件

气相色谱条件如下：

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

b) 色谱柱升温程序：80 ℃(2 min)$\xrightarrow{30\ ℃/min}$200 ℃ $\xrightarrow{5\ ℃/min}$250 ℃(3 min)；

c) 进样口温度：250 ℃；

d) 检测器温度：300 ℃；

e) 载气：氮气，纯度 99.99%，流速：恒流模式 3 mL/min；

f) 进样方式：不分流；

g) 进样量：2.0 μL。

7.3.2 气相色谱测定

在仪器最佳工作条件下，气相色谱采用外标法定量。根据样液中被测物残留的含量情况，选定峰面积相近的标准工作溶液。标准工作溶液和样液中被测物的响应值应在仪器的线性范围内，若其响应值超过线性范围，用正己烷(4.2)稀释到合适浓度后分析。对标准工作溶液和样液等体积参差进样测定。上述色谱条件下，苯氟磺胺的参考保留时间约为 12.07 min，气相色谱图参见附录 A 图 A.1。

7.4 气相色谱-质谱确证

7.4.1 气相色谱-质谱参考条件

气相色谱-质谱条件如下：

a) 色谱柱：DB-5 MS(30 m×0.25 mm×0.25 μm)石英毛细管柱，或相当者；

b) 色谱柱温度程序：60 ℃保持 2 min，然后以 30 ℃/min 程序升温至 220 ℃，再以 5 ℃/min 升温至 250 ℃，保持 5 min；

c) 进样口温度：250 ℃；

d) 色谱-质谱接口温度：280 ℃；

e) 离子源温度：230 ℃；

f) 载气：氦气，纯度≥99.999%，流速 1.0 mL/min；

g) 进样方式：不分流进样，1.0 min 后打开分流阀和隔垫吹扫阀；

h) 电离方式：电子轰击源(EI)；

i) 进样量：1 μL；

j) 测定方式：选择离子监测方式；

k) 选择监测离子(m/z)：123、167、224、332(丰度比 100∶45∶30∶6)；

l) 溶剂延迟：6 min；

m) 电离能量：70 eV。

7.4.2 气相色谱质谱确证

经确证分析被测物色谱峰保留时间和标准品样品相一致，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，相似度在允许偏差之内(见表 1)，则可判定样品为苯氟磺胺阳性检出。苯氟磺胺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 A 中图 A.2 和 A.3。

表 1 定性确证时相对离子丰度的最大允许误差

相对离子丰度/%	>50	>20～50	>10～20	≤10
允许的相对误差/%	±10	±15	±20	±50

7.5 空白试验

除不加试样外，按上述测定条件和步骤进行样品空白实验。

8 结果计算和表述

气相色谱法测定试样中苯氟磺胺残留量采用标准曲线法定量，标准曲线法定量结果按式(1)计算，计算结果需扣除空白值：

$$X_i = \frac{C_i \times V}{m} \quad \cdots\cdots(1)$$

式中：

X_i ——试样中苯氟磺胺的含量，单位为毫克每千克(mg/kg)；

C_i ——从标准工作曲线上得到的苯氟磺胺浓度，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)；

m ——最终样品溶液所代表试样的质量，单位为克(g)。

注：计算结果应扣除空白值。

9 定量限和回收率

9.1 定量限

本标准对大米、玉米、小麦、大豆、糙米、梨、葡萄、马铃薯、黄瓜、番茄和蘑菇的定量限为 0.01 mg/kg；干辣椒的定量限为 0.05 mg/kg。

9.2 回收率

在不同基质，不同添加水平的回收率范围见附录 B 表 B.1。

附　录　A
（资料性附录）
苯氟磺胺标准品色谱图

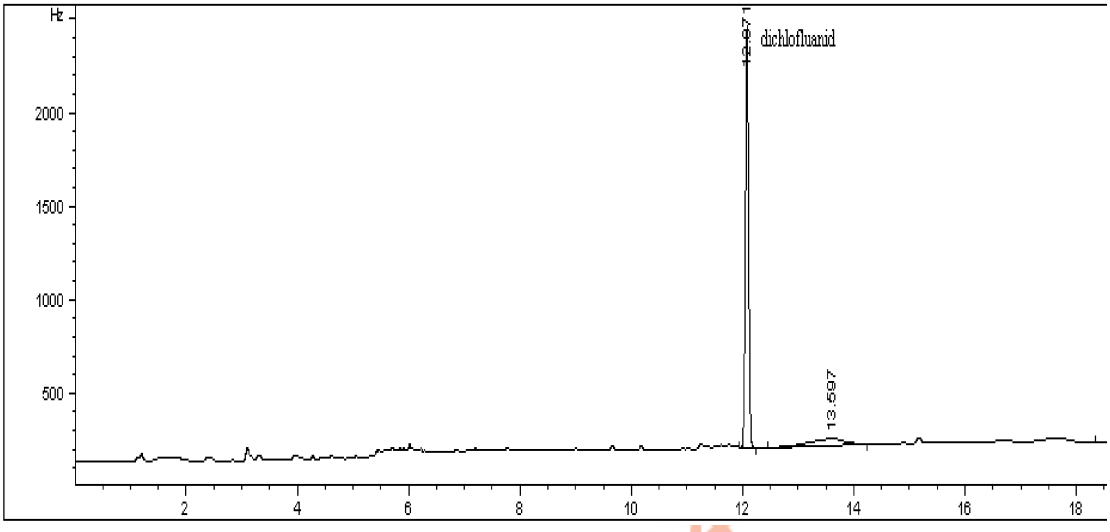

图 A.1　苯氟磺胺标准品气相色谱图（25 ng/mL）

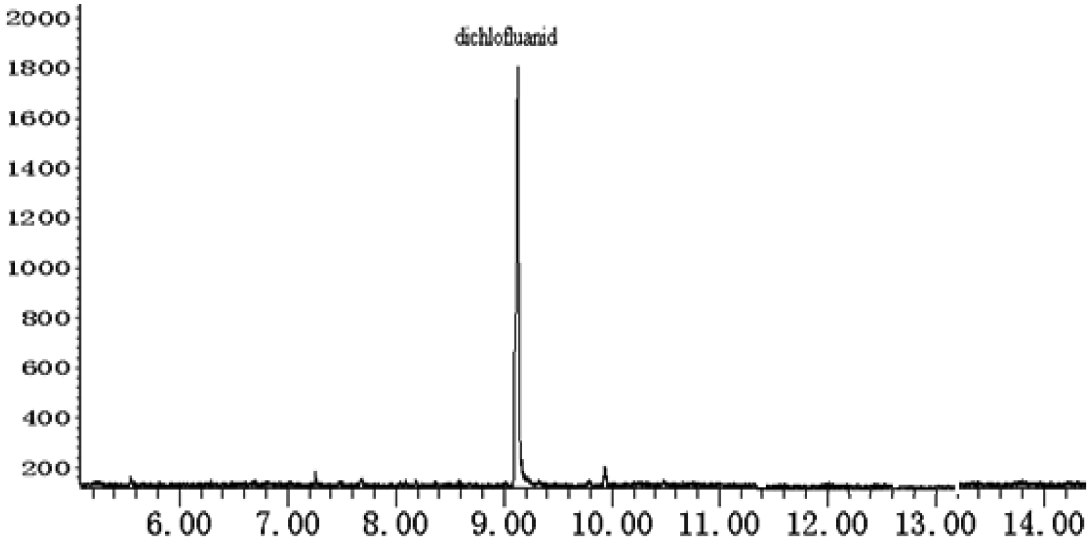

图 A.2　苯氟磺胺标准品气相色谱色谱选择离子色谱图（25 ng/mL）

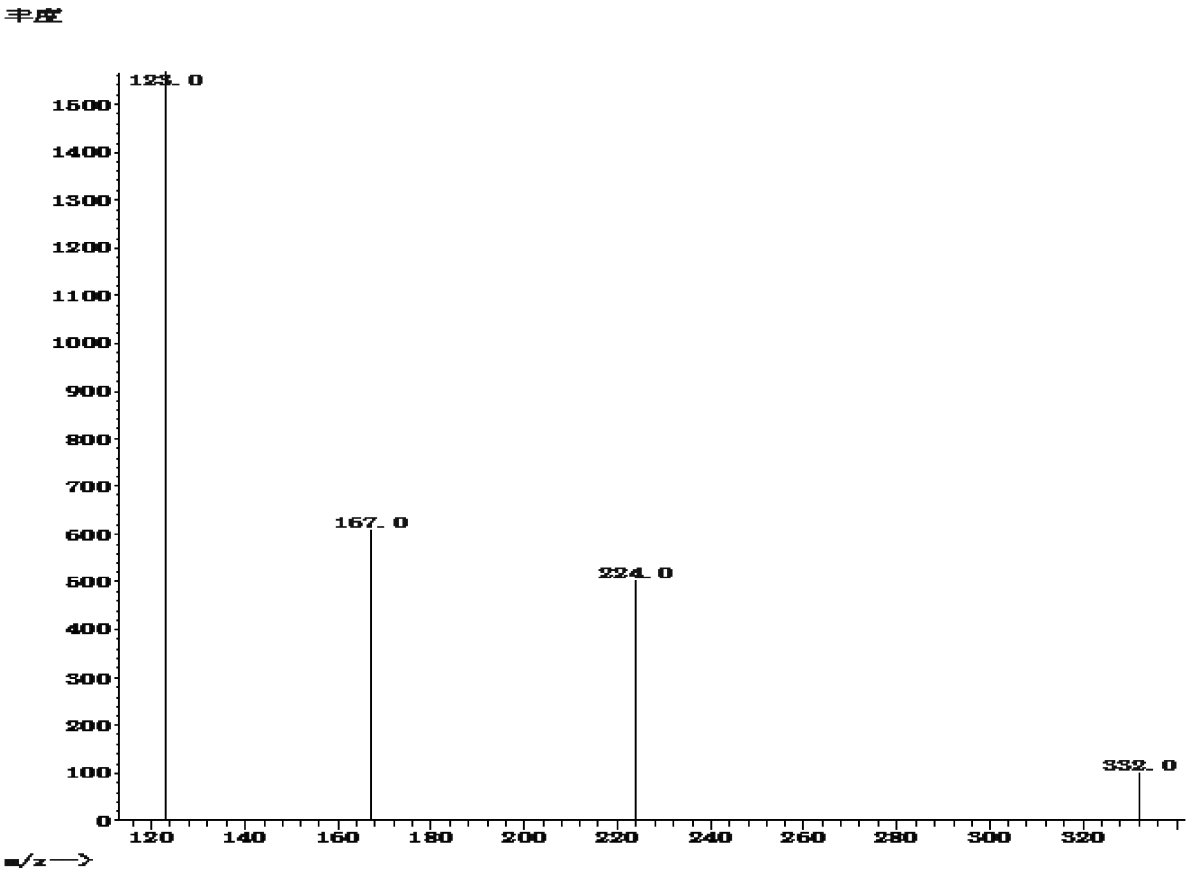

图 A.3　苯氟磺胺标准品选择离子质谱图(25 ng/mL)

附 录 B
（资料性附录）
回收率

表 B.1 样品的添加浓度及回收率数据

基质	添加量（mg/kg）	回收率范围（%）	基质	添加量（mg/kg）	回收率范围（%）
大米	0.01	76.0-105.0	糙米	0.01	79.0-99.0
	0.02	78.0-100.5		0.02	77.5-103.0
	0.1	84.2-100.4		0.1	84.9-103.4
小麦	0.01	77.0-103.0	马铃薯	0.01	79.0-103.0
	0.02	78.0-100.5		0.02	80.5-102.5
	0.1	81.8-105.4		0.1	86.9-102.2
玉米	0.01	75.0-103.0	梨	0.01	84.0-105.0
	0.02	78.2-101.0		0.02	84.0-108.0
	0.1	82.9-102.1		0.1	87.5-102.1
	5	83.8-101.4		5	86.6-100.2
大豆	0.01	75.0-104.0	葡萄	0.01	79.0-103.0
	0.02	74.0-98.5		0.02	79.5-101.0
	0.1	81.5-102.1		0.1	84.4-101.4
	0.2	84.0-106.0		15	85.2-100.3
番茄	0.01	79.0-107.0	蘑菇	0.01	76.0-108.0
	0.02	81.0-102.5		0.02	79.0-106.0
	0.1	84.9-103.1		0.1	81.2-102.1
	2	84.0-102.5		5	83.8-100.2
黄瓜	0.01	82.0-104.0	干辣椒	0.05	75.0-106.0
	0.02	82.5-106.0		0.1	80.9-100.5
	0.1	87.4-101.2		0.5	82.0-102.0
	5	87.8-100.4		20	81.6-100.1

Foreword

This standard was drafted according to GB/T 1.1—2009

This standard substitutes for the former standard of SN 0491—1995,《Method for the determination of dichlofluanid residues in cereals for export》.
Compared with the above mentioned standard, the changes mainly include:

—To add the matrix of rice, wheat, soybean, corn, pear, grape, tomato, cucumber, potato, mushroom and chilli;

—To add gas chromatography-mass spectrometry method validation;

—To deleted the sampling part;

—To optimize pre-treatment method.

Attention is required to the certain contents of this text which might be related to some patents. This file is not responsible to identify these.

This standard was proposed by and is under the charge of General Administrations of Customs of th P.R.C.
This standard was drafted by Shijiazhuang Castoms of the People's Republic of China.

Main drafters of this standard are: Li Wei、Wang Jing、Ai Lianfeng、Ma Yusong、Chen Ruichun、Guo Chunhai、Zhang Haichao

This standard replaces the previous version of the release of the standard as following:

—SN 0491—1995.

Note: This English version, a translation from the Chinese text, is solely for guidance.

Determination of dichlofluanid residues in foodstuffs origined from plant for export

1 Scope

This standard specifies the determination and confirmation of dichlofluanid residues by gas chromatography and confirmation by gas chromatography-mass spectrometry in foodstuffs origined from plant for export.

This standard is applicable to the determination and confirmation of dichlofluanid residues in rice, wheat, soybean, corn, cereal, pear, grape, potato, tomato, cucumber, mushroom and chilli for export.

2 Normative references

The items of the following listed standard become the items of this standard due to the quotation by this standard. The cited references with date would not apply to this standard if their amendment (not including corrected printing errors) or revision appear. However, it is encouraged to study if the newest edition of these references can be used. The newest edition is applicable to this standard if the references are not quoted with date.

GB/T 6682 water for laboratory use-Specifications

3 Principle

The residues in the test sample are extracted with acetonitrile. After concentrated, the solution is cleaned up with SPE cartridge of Silica Gel, the residues are then determined by gas chromatography and confirmation by gas chromatography-mass spectrometry, using external standard method.

4 Reagents and materials

Unless specifically noted, all reagents used should be of analytically grade; "water" is the first level water described by GB/T 6682.

4.1 Acetonitrile: HPLC grade.

4.2 n-Hexane: HPLC grade.

4.3 Ether.

4.4 Acetone: HPLC grade.

4.5 Anhydrous sodium sulfate: Ignite at 650 ℃ for 4 h, and keep in a tightly closed container after cool.

4.6 n-Hexane-ether (3 + 47, v/v): transfer 30 mL n-Hexane (4.2) into 470 mL ether (4.3), mix them.

4.7 n-Hexane-ether (1 + 1, v/v): transfer 100 mL n-Hexane (4.2) into 100 mL ether (4.3), mix them.

4.8 Standard of dichlofluanid: Molecular formula $C_9H_{11}Cl_2FN_2O_2S_2$, GAS NO. 1085-98-9, purity is no less than 99%.

4.9 Stock standard solution: Accurately weigh an adequate amount of standard (4.8), dissolve in acetone (4.4) and prepare a solution of 1.0 mg/mL. The solution should be stored at the temperature 4 ℃ avoiding light and stable for one year.

4.10 Middle working solution: diluted 1 mL stock standard solution (4.9) with acetone to 100 mL and the concentration of the middle solution is 10.0 μg/mL. The solution should be stored at the temperature 4 ℃ avoiding light and stable for three months.

4.11 Standard working solution: Then dilute middle working standard solution (4.10) with acetone (4.4) to the required concentration as the standard working solution.

4.12 Silica Gel cartridge: 500 mg, 6 mL, or equivalent.

4.13 Membrane filter: 0.22 μm, organic type.

5 Apparatus and equipment

5.1 Gas chromatography, equipped with electron capture detector (ECD).

5.2 Gas chromatography-mass spectrometry, equipped with electro-Impact source (EI).

5.3 Organ blender.

5.4 Balance: accuracy to 0.01 g and 0.1 mg.

5.5 Centrifuge, speed of no less than 5 000 r/min.

5.6 Homogenizer, speed of no less than 15 000 r/min.

5.7 N_2 evaporator.

5.8 Rotary vacuum evaporator.

5.9 Solid-phase extraction.

5.10 Centrifuge tube with cap: 50 mL and 15 mL.

5.11 Glass centrifuge tube: 10 mL.

5.12 Concentrate bottle: 120 mL.

6 Preparation and storage of test sample

6.1 Requirement

In the course of sample preparation, precaution must be taken avoid the contamination or any factors which may cause the charge of residue content.

6.2 Preparation of test sample

6.2.1 Pear, grape, potato, tomato, cucumber, mushroom and chilli

Take approximately 500 g of representative sample, take the whole pear, tomato, cucumber and chilli without handle; take the whole grape, potato and mushroom. Cut into minces and crush with a crusher into pulp and mix thoroughly, divide into two equal portions and then place in clean containers, which should be stored at −18 ℃.

6.2.2 Rice, corn, wheat, soybean and cereal

Take approximately 500 g of representative sample, take the whole grain of rice, corn, wheat, soybean and cereal, smash thoroughly by a pulverizer, mix thoroughly, divide into two equal portions and then place in clean containers, which is sealed and labeled.

7 Procedure

7.1 Extraction

7.1.1 Pear, grape, potato, tomato, cucumber and mushroom

Weigh 5 g (accurate to 0.01g) in a 50 mL centrifuge tube, add 10 g anhydrous sodium sulfate (4.5) and 20 mL acetonitrile (4.1) to the sample and then homogenize it for 2 min (15 000 r/min), and then centrifuge the sample solution for 10 min at 4 000 r/min. The supematants are passed through a filler of 10 g anhydrous sodium sulfate (4.5), collect the effluent into a 120 mL condense bottle.Repeat the extraction procedure with 15 mL acetonitrile again. Evaporate the combined extract to nearly dry by a rotary evaporator with a bath temperature below 45 ℃ and wait for purification.

7.1.2 Rice, corn, wheat, soybean, cereal and chilli

For chilli, weigh 2 g (accurate to 0.01 g) of the test sample into a 50 mL centrifuge tube. For rice, com, wheat, soybean, and cereal, weigh 5 g (accurate to 0.01 g) of the test sample into a 50 mL centrifuge tube. Add 10 mL deionized water to soak the sample, mix for 2 min on vortex mixer and allow to stand for 30 min. Add 10 g anhydrous sodium sulfate (4.5) and 20 mL acetonitrile (4.1) to the sample and then homogenize it for 2 min (15 000 r/min), and then centrifuge the sample solution for 10 min at 4 000 r/min. The supernatants are passed through a filler of 10 g anhydrous sodium sulfate (4.5), collect the effluent into a 120 mL condense bottle. Repeat the extraction procedure with 20 mL acetonitrile again. Evaporate the combined extract to nearly dry by a rotary evaporator with a bath temperature below 45 ℃ and wait for purification.

7.2 Cleaning-up

Add 5 mL n-hexane (4.2) to dissolve the residue for further clean-up procedure. And then rinse the Silica Gel column(4.12) with 5 mL of n-hexane (4.2) before use. Transfer the solution into the conditioned column. Wash the column with 6 mL n-hexane-ether (4.6). Discard the washings, and then elute with 2 mL n-hexane-ether (4.7). Collect the residue and blow to dryness under a nitrogen flow in a water bath below 45 ℃. Residues are dissolved with 2 mL n-hexane (4.2). Then the solution is passed through 0.22 μm filter and ready for analysis.

7.3 Determination

7.3.1 GC operating conditions

GC operating conditions:

a) Capilary column: DB-1701, 30 m×0.25 mm(i.d.)×0.25 μm(film thickness) or equivalent;

b) Column oven temperature procedure: 80 ℃ (2 min) $\xrightarrow{30\ ℃/min}$ 200 ℃ 5 $\xrightarrow{℃/min}$ 250 ℃ (3 min);

c) Injection temperature: 250 ℃;

d) Detector temperature: 300 ℃;

e) Carrier gas: Nitrogen, purity 99.99%; Carrier gas flow rate: constant code 3 mL/min;

f) Injection mode: Splitless;

g) Injection volume: 2.0 μL。

7.3.2 GC determination

Under the best conditions of the apparatus, inject series of standard working solutions separately. According to the approximate concentration of analyte in sample solution, select the standard working solution with similar responses to that of sample solution. The responses of the analyte in the standard working solution and the sample solution should be within the linear range of the instrument detection. Under the above operating condition, the retention time of dichlofluanid is 12.07 min, and the chromatogram of the standard can be found in Figure A. 1 in annex A.

7.4 GC-MS confirmation

7.4.1 GC operating conditions

GC operating conditions:

a) Capilary column: DB-5 MS, 30 m × 0.25 mm(i.d.) × 0.25 μm(film thickness) or equivalent;

b) Column oven temperature procedure: 60 ℃ (2 min) $\xrightarrow{30\ ℃/min}$ 200 ℃ $\xrightarrow{5\ ℃/min}$ 250 ℃ (5 min);

c) Injection temperature: 250 ℃;

d) Transfer temperature: 280 ℃;

e) Ion source temperature: 230 ℃;

f) Carrier gas: Helium, purity 99.99%; Carrier gas flow rate: 1.0 mL/min;

g) Injection mode: Splitless, purge on after 1.0 min;

h) Ionization mode: EI;

i) Injection volume: 1 μL;

j) Determination mode: SIM mode;

k) Selected monitoring ion (m/z): 123,167,224,332 (relative intensity 100∶45∶30∶6);

l) Solvent delay time: 6 min;

m) Electron energy: 70 eV.

7.4.2 GC-MS confirmation

Under above determination conditions, if the retention time of sample chromatogram peaks are consistent with the standard, 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). The corresponding analyte could be confirmed. Under the above operating condition, the chromatogram and mass spectrum of the standard can be found in Figure A.2 and Figure A.3 in annex A.

Table 1—Maximum permitted tolerance for ion intensities using a range of mass spectrometric techniques

Relative intensity(of base peak)/(%)	>50	>20～50	>10～20	≤10
Permitted tolerance(EI-GC-MS)/(%)	±10	±15	±20	±50

7.5 Blank test

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

8 Calculation an expression of result

Calculation the content of dichlofluanid residues in test sample by data processor or according to formula (1)

$$X_i = \frac{C_i \times V}{m} \qquad \cdots\cdots(1)$$

where:

X_i —the residue content of dichlofluanid in test sample, mg/kg;

C_i —the concentration of dichlofluanid in the standard working solution, μg/ml;

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

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

note: the blank value shall be subtracted from the result of calculation.

9 Limit of determination and recovery

9.1 Limit of determination

The limit of dichlofluanid pesticide is 0.01 mg/kg for rice, corn, wheat, soybean, cereal, pear, grape, potato, cucumber, tomato and mushroom, and the limit of dichlofluanid pesticide is 0.05 mg/kg for chilli.

9.2 Recovery

The ranges of recovery in different matrix at four different levels are showed in Table B.1 in annex B.

Annex A
（Informative）
Gas chromatogram of dichlofluanid standard

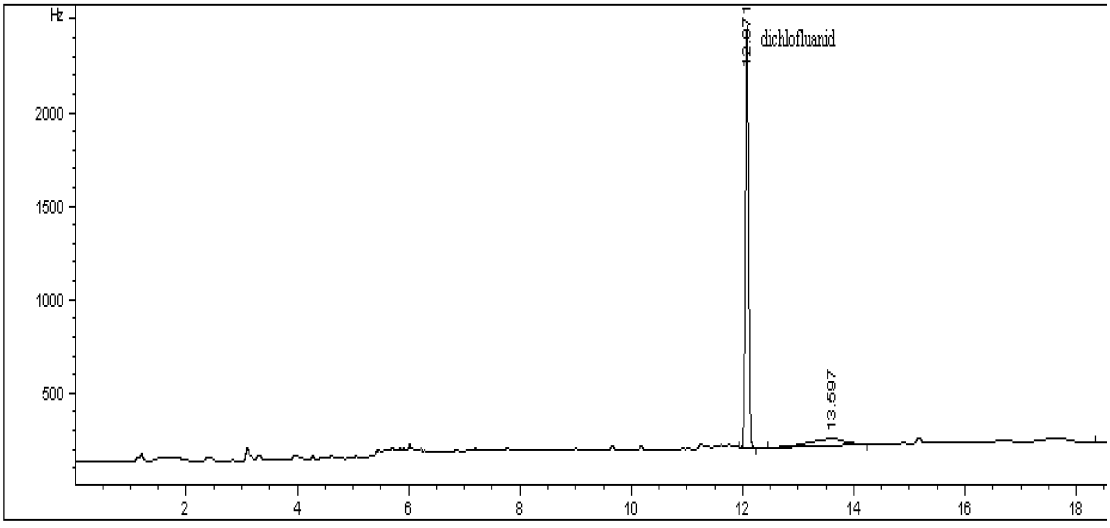

Fig.A.1　GC chromatogram of dichlofluanid standard at 25 ng/mL

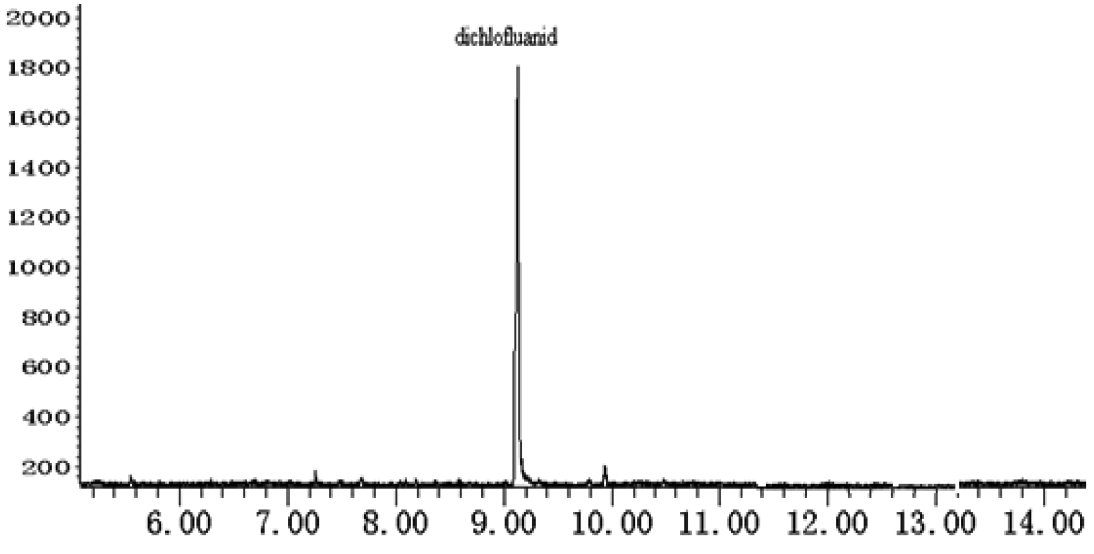

Fig.A.2　GC-MS（SIM）chromatogram of dichlofluanid standard at 25 ng/mL

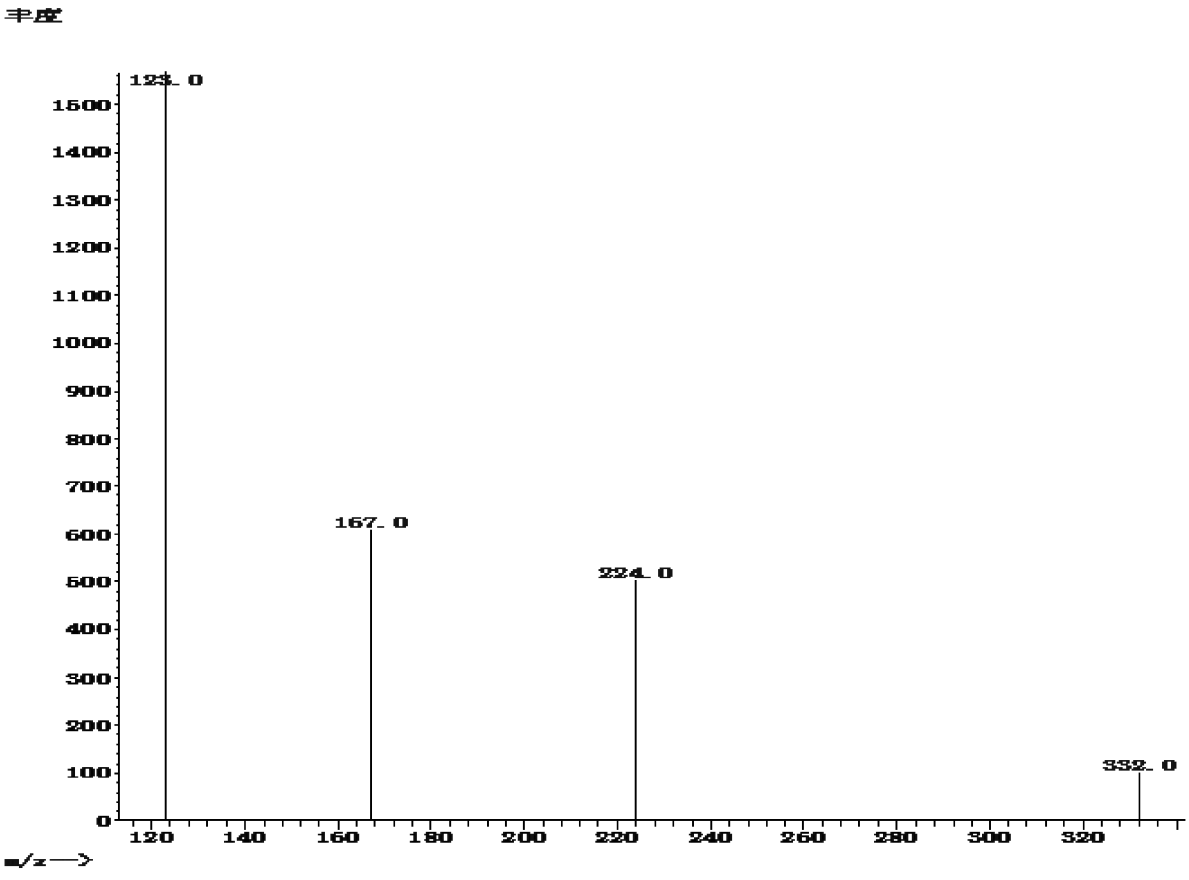

Fig.A.3 SIM mass spectrogram of dichlofluanid standard at 25 ng/mL

Annex B
（Informative）
Recovery ranges

Table B.1 Fortifying concentrations and recovery experimental data

sample	fortified level mg/kg	range of recovery %	sample	fortified level mg/kg	range of recovery %
rice	0.01	76.0-105.0	cereal	0.01	79.0-99.0
	0.02	78.0-100.5		0.02	77.5-103.0
	0.1	84.2-100.4		0.1	84.9-103.4
wheat	0.01	77.0-103.0	potato	0.01	79.0-103.0
	0.02	78.0-100.5		0.02	80.5-102.5
	0.1	81.8-105.4		0.1	86.9-102.2
corn	0.01	75.0-103.0	pear	0.01	84.0-105.0
	0.02	78.2-101.0		0.02	84.0-108.0
	0.1	82.9-102.1		0.1	87.5-102.1
	5	83.8-101.4		5	86.6-100.2
soybean	0.01	75.0-104.0	grape	0.01	79.0-103.0
	0.02	74.0-98.5		0.02	79.5-101.0
	0.1	81.5-102.1		0.1	84.4-101.4
	0.2	84.0-106.0		15	85.2-100.3
tomato	0.01	79.0-107.0	mushroom	0.01	76.0-108.0
	0.02	81.0-102.5		0.02	79.0-106.0
	0.1	84.9-103.1		0.1	81.2-102.1
	2	84.0-102.5		5	83.8-100.2
cucumber	0.01	82.0-104.0	chilli	0.05	75.0-106.0
	0.02	82.5-106.0		0.1	80.9-100.5
	0.1	87.4-101.2		0.5	82.0-102.0
	5	87.8-100.4		20	81.6-100.1