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中华人民共和国出入境检验检疫行业标准

SN/T 2581—2010

进出口食品中氟虫酰胺残留量的测定 液相色谱-质谱/质谱法

Determination of flubendiamide residues in food for import and export—LC-MS/MS

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食品伙伴网http://www.foodmate.net

前　言

本标准按照GB/T 1.1—2009给出的规则起草。

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嘉兴出入境检验检疫局、嘉兴学院、嘉兴市食品药品检验所负责起草。

本标准主要起草人：张萌萌、沈兵、李蕾、章展煌、欧贝丽、陈剑伟、奚奇辉、赖丽萍。

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进出口食品中氟虫酰胺残留量的测定 液相色谱-质谱/质谱法

1 范围

本标准规定了进出口食品中氟虫酰胺残留量测定的液相色谱-质谱/质谱分析方法。

本标准适用于葱、萝卜、西红柿、橙、大豆、苹果、茶、核桃、鱼、猪瘦肉、猪肝和牛奶中氟虫酰胺残留量的检测和确证。

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

试样中的氟虫酰胺残留用乙腈提取，提取液经石墨碳-氨基固相萃取柱或弗罗里硅土固相萃取柱净化，液相色谱-质谱/质谱测定，外标法定量。

4 试剂和材料

所有试剂除特殊注明外，均为分析纯，水为 GB/T 6682 规定的一级水。

4.1 乙腈：色谱纯。

4.2 甲苯：色谱纯。

4.3 正己烷：色谱纯。

4.4 丙酮：色谱纯。

4.5 乙腈饱和正己烷：向 250 mL 的分液漏斗中加入 150 mL 正己烷(4.3)和 50 mL 乙腈混匀，振荡 5 min，取上层备用。

4.6 乙腈-甲苯(3+1，体积比)：用量筒量取 300 mL 乙腈(4.1)和 100 mL 甲苯(4.2)，混匀备用。

4.7 丙酮-正己烷(1+1，体积比)：用量筒量取 200 mL 正己烷(4.3)和 200 mL 丙酮(4.4)，混匀备用。

4.8 乙腈-水(7+3，体积比)：用量筒量取 350 mL 乙腈(4.1)和 150 mL 水，混匀备用。

4.9 氯化钠。

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

4.11 氟虫酰胺(Flubendiamide)标准品：纯度大于等于 99%，CAS 号：272451-65-7，分子式：$C_{23}H_{22}F_7IN_2O_4S$，相对分子质量为 682.39。

4.12 氟虫酰胺标准储备溶液(100 μg/mL)：准确称取适量的氟虫酰胺标准物质，用乙腈配成浓度为 100 μg/mL 的标准储备液，该标准储备液置于 4 ℃冰箱中避光密封保存。

4.13 氟虫酰胺基质标准工作液：使用前根据需要将标准储备溶液用各种样本的空白基质稀释成适当浓度的标准工作液。基质标准工作液应现用现配。

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4.14 石墨碳-氨基固相萃取柱：500 mg/500 mg/6 mL或相当者。加样前先用5 mL乙腈-甲苯(4.6)预淋洗柱。

4.15 弗罗里硅土固相萃取柱：1 000 mg/6 mL或相当者。加样前依次用2 mL丙酮(4.4)和4 mL正己烷(4.3)预淋洗柱。

4.16 微孔滤膜：0.2 μm，通用型。

5 仪器和设备

5.1 液相色谱-质谱/质谱仪：配电喷雾离子源(ESI)。

5.2 电子分析天平：感量分别为0.01 mg和0.01 g。

5.3 组织捣碎机。

5.4 均质器：10 000 r/min。

5.5 离心机：4 000 r/min。

5.6 聚四氟乙烯塑料离心管：50 mL，具塞。

5.7 旋转蒸发仪。

5.8 氮吹仪。

6 试样制备和保存

6.1 试样制备

6.1.1 茶叶、大豆、核桃

取代表性样品约500 g，经组织捣碎机粉碎并通过2.0 mm圆孔筛，混匀，装入洁净容器内密封，标明标记。

6.1.2 葱、萝卜、西红柿、橙、苹果、鱼、猪瘦肉、猪肝

取代表性样品约500 g，将其可食部分先切碎，经组织捣碎机充分捣碎均匀，装入洁净容器内密封，标明标记。

6.1.3 牛奶

取代表性样品约500 g，装入洁净容器内密封，标明标记。

6.2 试样保存

茶叶、粮谷、坚果类等试样于0 ℃～4 ℃保存；蔬菜、水果、畜、禽类等试样于－18 ℃冷冻保存。在制样过程中，应防止样品受到污染或发生氟虫酰胺残留量的变化。

7 测定步骤

7.1 提取

7.1.1 葱、萝卜、西红柿、橙、大豆、苹果、茶、核桃

大豆、核桃和茶叶：称取2 g(精确至0.01 g)试样于50 mL离心管中，加5 mL水放置0.5 h；葱、萝卜、西红柿、橙、苹果：称取2 g(精确至0.01 g)试样于50 mL离心管中。加入20 mL乙腈(4.1)，以

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10 000 r/min均质 1 min，加入 2 g 氯化钠(4.9)，摇匀，并于 4 000 r/min 离心 3 min。吸取上层有机相于浓缩瓶中，残渣中加入 15 mL 乙腈，重复提取一次，合并上层有机相，在 40 ℃水浴中减压浓缩至近干，待净化。

7.1.2 鱼、猪瘦肉、猪肝、牛奶

称取粉碎后试样 2 g(精确至 0.01 g)于 50 mL 离心管中，加入 2 g 无水硫酸钠(4.10)，20 mL 乙腈(4.1)和 10 mL 乙腈饱和正己烷(4.5)，以 10 000 r/min 均质 1 min，以 4 000 r/min 离心 3 min，吸取下层有机相于浓缩瓶中，残渣中加入 15 mL 乙腈(4.1)，重复提取一次，合并下层有机相，在 40 ℃水浴中减压浓缩至近干，待净化。

7.2 净化

7.2.1 葱、萝卜、西红柿、橙、大豆、苹果、茶叶、核桃

以 3×2 mL 乙腈-甲苯(4.6)洗涤浓缩瓶中残留物(7.1.1)并全部转入石墨碳-氨基固相萃取柱(4.14)中，然后用 15 mL 乙腈-甲苯(4.6)淋洗柱子，控制流速不超过 2.0 mL/min，收集全部流出液，于 40 ℃水浴中减压浓缩，氮吹至近干，用乙腈-水(4.8)溶解残渣并定容至 2.0 mL，过滤膜(4.16)，供液相色谱-质谱/质谱分析。

7.2.2 鱼、猪瘦肉、猪肝、牛奶

以 3×2 mL 正己烷(4.3)洗涤浓缩瓶中残留物(7.1.2)并全部转入弗罗里硅土固相萃取柱(4.15)中，待溶液全部流出固相萃取柱后加入 2 mL 正己烷(4.3)淋洗，弃去全部流出液。再以 10 mL 丙酮-正己烷(4.7)洗脱，控制流速不超过 2.0 mL/min，收集全部洗脱液，于 40 ℃水浴中减压浓缩，氮吹至近干，用乙腈-水(4.8)溶解残渣并定容至 2.0 mL，过滤膜(4.16)，供液相色谱-质谱/质谱分析。

7.3 测定

7.3.1 液相色谱条件

a) 色谱柱：C_{18}柱，2.1 mm×50 mm，1.7 μm 或相当者；

b) 流动相以及梯度洗脱程序：见表 1；

表 1 流动相梯度洗脱程序

时间/min	乙腈/%	水/%
0.00	60	40
2.00	100	0
3.00	60	40

c) 流速：0.20 mL/min；

d) 柱温：35 ℃；

e) 进样量：2 μL。

7.3.2 质谱条件

参见附录 A。

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7.3.3 液相色谱-质谱/质谱测定

根据样液中氟虫酰胺含量情况，选定峰面积相近的标准工作溶液。标准工作溶液和样液中氟虫酰胺响应值均应在仪器检测线性范围内。如果残留量超出标准曲线范围，应用乙腈将样品提取液(7.1)进行适当稀释。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，氟虫酰胺的保留时间约为 1.75 min。被测组分选择 1 个母离子，2 个以上子离子，在相同实验条件下，如果样品中待检测物质与标准溶液中对应的保留时间偏差在±2.5%之内，且样品谱图中各组分定性离子的相对丰度与浓度接近的标准溶液谱图中对应的定性离子的相对丰度进行比较，偏差不超过表 2 规定的范围，被确证的样品可判定为氟虫酰胺阳性检出。标准品的质谱图参见附录 B 中图 B.1、图 B.2。

表 2 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
允许的相对偏差/%	±20	±25	±30	±50

7.4 空白实验

除不称取样品外，均按上述测定条件和步骤进行。

8 结果计算和表述

用色谱数据处理机或按式(1)计算试样中氟虫酰胺的残留含量，计算结果需扣除空白值。

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

式中：

X ——试样中氟虫酰胺的残留量，单位为毫克每千克(mg/kg)；

A ——样液中氟虫酰胺的峰面积或峰高；

A_s——标准工作液中氟虫酰胺的峰面积或峰高；

c ——标准工作液中氟虫酰胺的浓度，单位为纳克每毫升(ng/mL)；

m ——最终样液所代表的试样质量，单位为克(g)；

V ——样液最终定容体积，单位为毫升(mL)。

9 测定低限、回收率

9.1 测定低限

本方法氟虫酰胺的测定低限为 0.005 mg·kg。

9.2 回收率

氟虫酰胺添加浓度及回收率的 LC-MS-MS 实验数据见表 3。

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表 3　不同基质的添加浓度、回收率

样品名称	添加浓度/(mg/kg)	回收率/%
葱	0.005	70.0～99.6
	1.50	88.5～98.1
	3.00	77.6～100.4
萝卜	0.005	68.0～95.6
	0.015	77.5～107.8
	0.030	90.9～108.7
西红柿	0.005	92.6～108.9
	0.300	68.0～86.0
	0.700	73.0～86.6
橙	0.005	61.8～95.3
	0.010	76.4～109.6
	0.020	78.6～110.0
大豆	0.005	68.4～91.2
	0.150	78.7～100.2
	0.300	68.4～95.3
苹果	0.005	71.7～88.4
	0.700	66.3～72.4
	1.000	91.1～94.9
茶	0.005	68.4～97.8
	20.00	75.4～91.1
	40.00	84.1～91.5
核桃	0.005	74.7～95.8
	0.030	81.7～100.7
	0.060	87.8～98.7
鱼	0.005	88.3～99.4
	0.010	79.6～109.6
	0.020	83.6～110.8
猪瘦肉	0.005	82.0～107.1
	0.050	66.6～83.4
	0.100	74.6～81.5
猪肝	0.005	70.8～96.1
	0.300	81.7～94.3
	0.600	84.3～101.0
牛奶	0.005	87.5～100.8
	0.100	74.8～89.6
	0.200	84.0～94.5

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附 录 A
（资料性附录）
氟虫酰胺液相色谱-质谱/质谱条件

氟虫酰胺液相色谱-质谱/质谱条件：

a） 离子源：电喷雾离子源（ESI），负离子扫描；

b） 检测方式：多反应选择离子检测（MRM）；

c） 电喷雾电压（IS）：2 500 V；

d） 雾化气、气帘气、辅助加热气、碰撞气均为高纯氮气及其他合适气体；使用前应调节各气体流量以使质谱灵敏度达到检测要求；

e） 辅助气温度（TEM）：250 ℃；

f） 离子源温度：110 ℃；

g） 定性离子对、定量离子对、采集时间、去簇电压及碰撞能量见表 A.1。

表 A.1 氟虫酰胺监测离子对、定量离子对、去簇电压及碰撞能

被测物名称	监测离子对（m/z）	定量离子对（m/z）	驻留时间/ms	去簇电压/V	碰撞能量/V
氟虫酰胺	681.1/254.1	681.1 254.1	500	−35	−30
	681.1/274.1				−20

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附 录 B
（资料性附录）
氟虫酰胺标准品 LC/MS/MS 质谱图

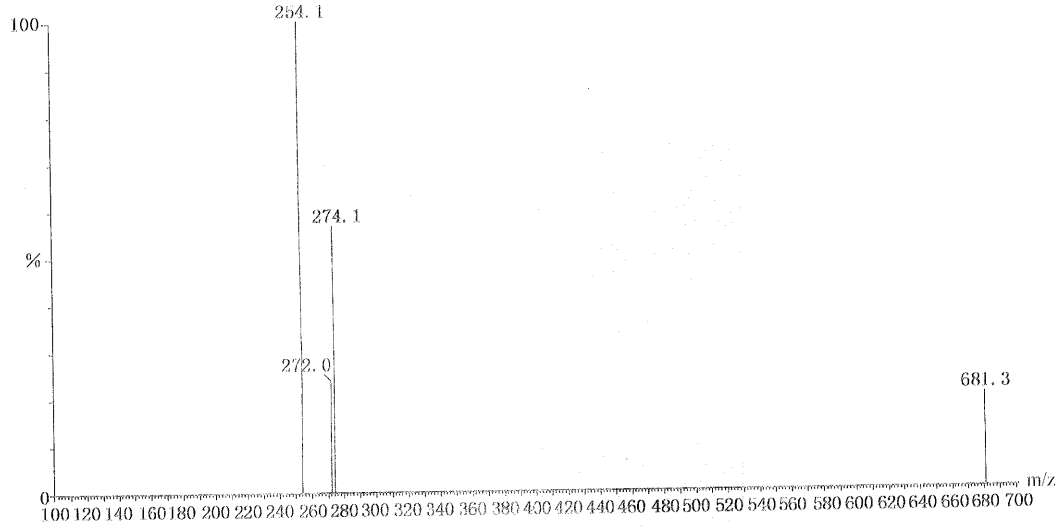


图 B.1 氟虫酰胺标准品的离子全扫描质谱图(浓度为 5 ng/mL)

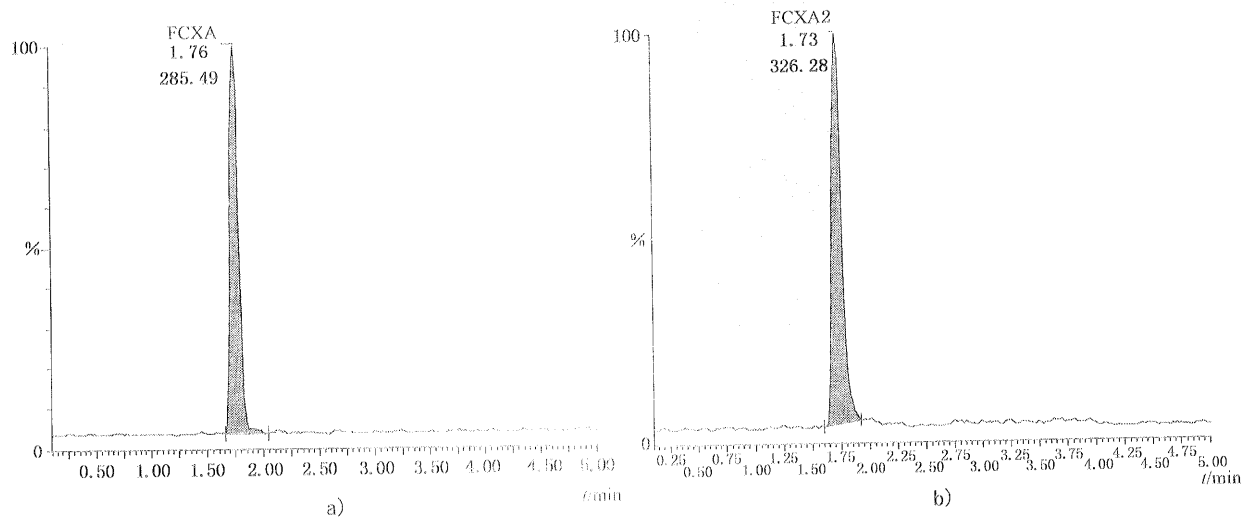


图 B.2 氟虫酰胺标准品的多反应监测(MRM)色谱图(浓度为 5 ng/mL)

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Foreword

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

The standard was drafted by Jiaxing Entry-Exit Inspection and Quarantine Bureau, Jiaxing Colledge and Jiaxing Institute For Food and Drug Control.

The main drafters of this standard are Zhang Mengmeng, Shen Bin, Li Lei, Zhang Zhanhuang, Ou Beili, Chen Jianwei, Xi Qihui, Lai Liping.

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Determination of flubendiamide residues in food for import and export—LC-MS/MS

1 Scope

This standard specifies the method of determination of flubendiamide residues in food for import and export by LC-MS/MS method.

This standard is applicable to the determination and confirmation of flubendiamide residues in shallot、radish、tomato、orange、soybean、apple、tea、walnut、fish、pork、pig liver、milk and so on…

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this National Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based in this National Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies.

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

3 Principle

The flubendiamide residue in the test sample is extracted with acetonitrile. The extracts are cleaned up by Envi-Carb/LC-NH_2 or LC-Florisil SPE cartridge. Determination and confirmation made by LC-MS/MS using external standard method.

4 Reagents and materials

Unless specified, all reagents should be of analytical pure; "water" is the first grade water prescribed by GB/T 6682.

4.1 Acetonitrile: Chromatogram grade.

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4.2 Toluene: Chromatogram grade.

4.3 *n*-hexane: Chromatogram grade.

4.4 Acetone: Chromatogram grade.

4.5 *n*-hexane saturated by acetonitrile: Add 150 mL *n*-hexane(4.3) and 50 mL acetonitrile (4.1) to pear shape separatory funnel, shake for 5 min, pipette the upper solution which is ready for use.

4.6 Acetonitrile-toluene(3+1, *V*/*V*): Measure 300 mL acetonitrile (4.1) and 100 mL toluene (4.2) by measuring cylinder, mixed and ready for use.

4.7 Acetone-*n*-hexane(1+1, *V*/*V*): Measure 200 mL aetone (4.4) and 200 mL *n*-hexane (4.5) by measuring cylinder, mixed and ready for use.

4.8 Acetonitrile-water (7+3, *V*/*V*): Measure 350 mL acetonitrile (4.1) and 150 mL water by measuring cylinder, mixed and ready for use.

4.9 Sodium chloride: Analytic grade.

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

4.11 Flubendiamide standard chemicals: Purity ≥ 99%, CAS number: 272451-65-7, formular: $C_{23}H_{22}F_7IN_2O_4S$, molecular weight: 682.39.

4.12 Flubendiamide standard stock solution(100 μg/mL): Accurately weigh adequate amount of flubendiamide standards, dissolve with acetonitrile and prepare a solution of 100 μg/mL as standard stock solution. The standard stock solution should be stored at the temperature 4 ℃ and protecting from light.

4.13 Flubendiamide standard working solution: According to the requirement, pipette adequate amount of standard stock solution(4.12), and dilute with different solution extracted from blank sample to prepare appropriate concentration standard working solutions. These solutions should be prepared before use.

4.14 Envi-Carb/LC-NH_2 SPE cartridge: 500 mg/500 mg, 5 mL or equivalent. Before use, condition the Envi-Carb /LC-NH_2 SPE cartridge with 5 mL of acetonitrile- toluene (4.6).

4.15 LC-Florisil SPE cartridge: 1 000 mg, 5 mL or equivalent. Before use, condition the LC-Florisil SPE cartridge with 2 mL acetone (4.4) and 4 mL *n*-hexane (4.3) in turn.

4.16 Syringe drireven filter: 0.2 μm.

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5 Apparatus and equipment

5.1 LC-MS/MS spectrometry, equipped with ESI.

5.2 Electronic baiance, spectively sensitivity at 0.01 mg and 0.01 g.

5.3 Tissue blender.

5.4 Vortex mixer (10 000 r/min).

5.5 Centrifuge (4 000 r/min).

5.6 Centrifuge tube, polytetrafluoroethylene, 50 mL with stopper.

5.7 Rotary vacuum evaporator.

5.8 Nitrogen evaporator.

6 Preparation and storage of test sample procedures

6.1 Sample preparation

6.1.1 Tea、soybean and walnut

Take about 500 g of representative sample. Ground in a pulverizer for granule with size of 2.0 mm. Keep the prepared sample into a clean container, seal and label.

6.1.2 Shallot、radish、tomato、orange、apple、tea、fish、pork、pig liver

Take about 500 g of representative sample. The sample can eat, and ground in a blender. Keep the prepared sample into a clean container, seal and label.

6.1.3 Milk

Take about 500 g of representative sample. Keep the prepared sample into clean containers, seal and label.

6.2 Storage

Tea、soybean、walnut products are stored at 0 ℃～4 ℃ in refrigerator. Shallot、radish、tomato、orange、apple、fish、pork、pig liver、milk products are stored at －18 ℃ in refrigerator.

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In the course of sample preparation, precautions shall be taken to avoid contamination or any factors that may cause the change of residue content.

7 Procedure of Determination

7.1 Extraction

7.1.1 Shallot、radish、tomato、orange、soybean、apple、tea、walnut、fish、pork、pigliver、milk

Soybean、tea and walnut: weigh an aliquot of 2 g (accurately, 0.01 g) sample into a 50 mL capped centrifuge tube, add 5 mL water, stand for 0.5 h.

Shallot、radish、tomato、orange、apple: weigh an aliquot of 2 g (accurately, 0.01 g) sample into a 50 mL capped centrifuge tube.

Add 20 mL acetonitrile (4.1) into centrifuge tube, vortex for 1 min at 10 000 r/min, then add 2 g sodium chloride (4.9), mixed and centrifuge at 4 000 r/min for 3 min. Transfer the supernatant into another tube. Repeat the extraction of the residues in the same way with 15 mL acetonitrile (4.1), combined the solution. The solution evaporate to nearly dryness in a water bath 40 ℃. It is ready for SPE column to clean up.

7.1.2 Fish、pork、pig liver、milk

Weigh an aliquot of 2 g (accurately, 0.01 g) sample into a 50 mL capped centrifuge tube, add 2 g anhydrous sodium sulfate (4.10), 20 mL acetonitrile (4.1) and 10 mL *n*-hexane saturated by acetonitrile (4.5). Then vortex for 1 min at 10 000 r/min, and centrifuge at 4 000 r/min for 3 min. Transfer the lower solution into another tube. Repeat the extraction of the residues in the same way with 15 mL acetonitrile (4.1), combined the solution. The solution evaporate to nearly dryness in a water bath 40 ℃. It is ready for SPE column to clean up.

7.2 Cleanup

7.2.1 Shallot、radish、tomato、orange、soybean、apple、tea、walnut

Draw the extractant (7.1.1) with 2 mL acetonitrile-toluene (4.6) through Envi-Carb/LC-NH_2 SPE cartridge (4.14), repeat twice. Wash the tube with 15 mL acetonitrile-toluene (4.6), flow rate is 2 mL/min, collect elution. The solution evaporate to nearly dryness in a water bath below 40 ℃. Dissolve the residue and dilute to 2.0 mL acetonitrile-water (4.8). Filter the solution with a 0.20 μm filter (4.16). The final solution is ready for analysis.

7.2.2 fish、pork、pig liver、milk

Draw the extractant (7.1.2) with 2 mL *n*-hexane (4.3) through LC-Florisil SPE cartridge (4.15),

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repeat twice. Wash the tube with 2 mL *n*-hexane (4.3), discard elution. Wash the tube with 10 mL *n*-hexane-Acetone(4.7), flow rate is 2 mL/min, collect elution. The solution evaporate to nearly dryness in a water bath below 40 ℃. Dissolve the residue and dilute to 1.0 mL acetonitrile-water (4.8). Filter the solution with a 0.20 μm filter (4.16). The final solution is ready for analysis.

7.3 Determination

7.3.1 LC operating conditions:

a) Column: Symmetry C_{18}, 2.1 mm×50 mm, 1.7 μm or equivalent;

b) Mobile phase and Gradient elution, as table 1;

Table 1--Gradient elution program

Time/min	Acetonitrile/%	Water/%
0.00	60	40
2.00	100	0
3.00	60	40

c) Flow rate: 0.20 mL/min;

d) Column temperature: 35 ℃;

e) Injection volume: 2 μL.

7.3.2 MS operation conditions.

Seeing in annex A.

7.3.3 LC-MS/MS determination

According to approximate concentration of analyte, select the standard working solution with similar response to that of sample solution. The responses of flubendiamide 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. At the above LC-MS/MS conditions, the retention time is 1.75 min. The qualitative ions for each analyst include one precursor ion and two product ions at least. Under the same determination conditions, The retention time of the analyte in the sample shall match that of the calibration standard within the tolerances 2.5%. The relative intensities of the detected ions of each analyst, shall correspond to those of the calibration standard at comparable concentrations, within the tolerances shown in table 2, then the corresponding analyte must be present in the sample. For LC-MS/MS chromatogram (MRM) of flubendiamide standards, see figure B.1 and B.2 in annex B.

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Table 2 Maximum permitted tolerances for relative ion intensities while confirmation

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

7.4 Blank test

The operation of the blank test is the same as the procedure that described above, but with omission of sample addition.

8 Calculation of result

The calculation of flubendiamide concentration in the sample is according to the formula (1):

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

Where

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

A —peak area of flubendiamide in the sample solution;

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

c —the concentration of flubendiamide in the standard working solution, ng/mL;

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

V —the final volume of sample solution, mL.

9 Limit of quantification and recovery

9.1 Limit of quantification

The limit of quantification for flubendiamide is 0.005 mg/kg.

9.2 Recovery

According to the experimental data, the fortified concentrations of flubendiamide and their corresponding recoveries are shown in table 3.

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Table 3—recovery of flubendiamide

Sample	Spiking/(mg/kg)	Recovery/%
shallot	0.005	70.0～99.6
	1.50	88.5～98.1
	3.00	77.6～100.4
radish	0.005	68.0～95.6
	0.015	77.5～107.8
	0.030	90.9～108.7
tomato	0.005	92.6～108.9
	0.300	68.0～86.0
	0.700	73.0～86.6
orange	0.005	61.8～95.3
	0.010	76.4～109.6
	0.020	78.6～110.0
soybean	0.005	68.4～91.2
	0.150	78.7～100.2
	0.300	68.4～95.3
apple	0.005	71.7～88.4
	0.700	66.3～72.4
	1.000	91.1～94.9
tea	0.005	68.4～97.8
	20.00	75.4～91.1
	40.00	84.1～91.5
walnut	0.005	74.7～95.8
	0.030	81.7～100.7
	0.060	87.8～98.7
fish	0.005	88.3～99.4
	0.010	79.6～109.6
	0.020	83.6～110.8
pork	0.005	82.0～107.1
	0.050	66.6～83.4
	0.100	74.6～81.5
pig liver	0.005	70.8～96.1
	0.300	81.7～94.3
	0.600	84.3～101.0
milk	0.005	87.5～100.8
	0.100	74.8～89.6
	0.200	84.0～94.5

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Annex A
(Informative)
LC-MS/MS operation conditions

A.1 LC-MS/MS operation conditions

a) Ion source: ESI, negative ionisation mode.

b) Scan mode: multiple reaction monitoring (MRM).

c) Ion spray voltage (IS): 2 500 V.

d) The other reference mass conditions are adjusted so that the sensitiveness is satisfactional.

e) Probe temperature (TEM): 250 ℃.

f) Source Temperature: 110 ℃.

g) Monitoring ion pairs and reference collision voltage as table A.1.

Table A.1—Monitoring ion pairs and reference collision voltage

Compound	Monitorions (m/z)	Quantitation ion (m/z)	Dwell/ ms	Cone Volt/ V	Col. Energy/ V
flubendiamide	681.1/254.1	681.1/254.1	500	−35	−30
	681.1/274.1				−20

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Annex B
(Informative)
LC-MS/MS chromatogram of flubendiamide standards

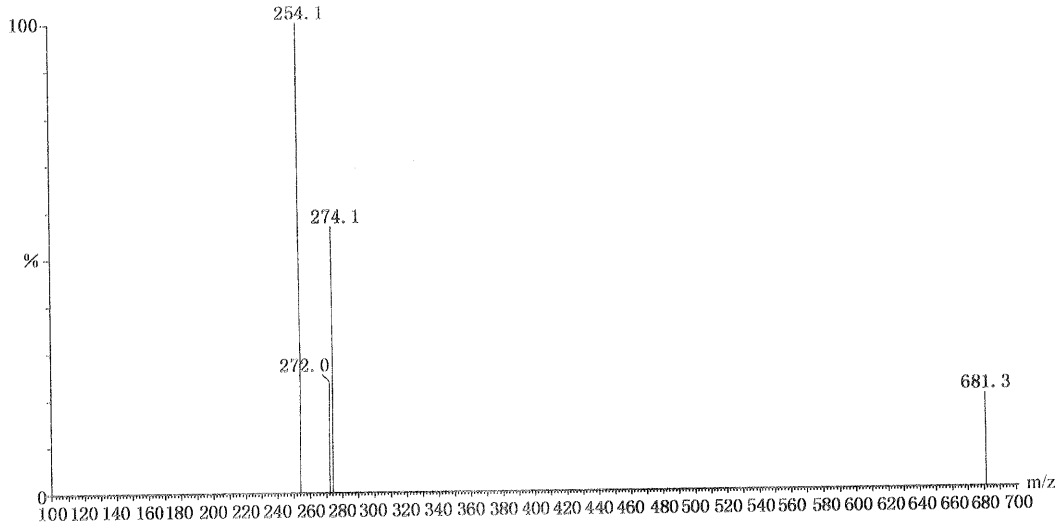


Figure B.1—LC-MS/MS chromatogram of flubendiamide

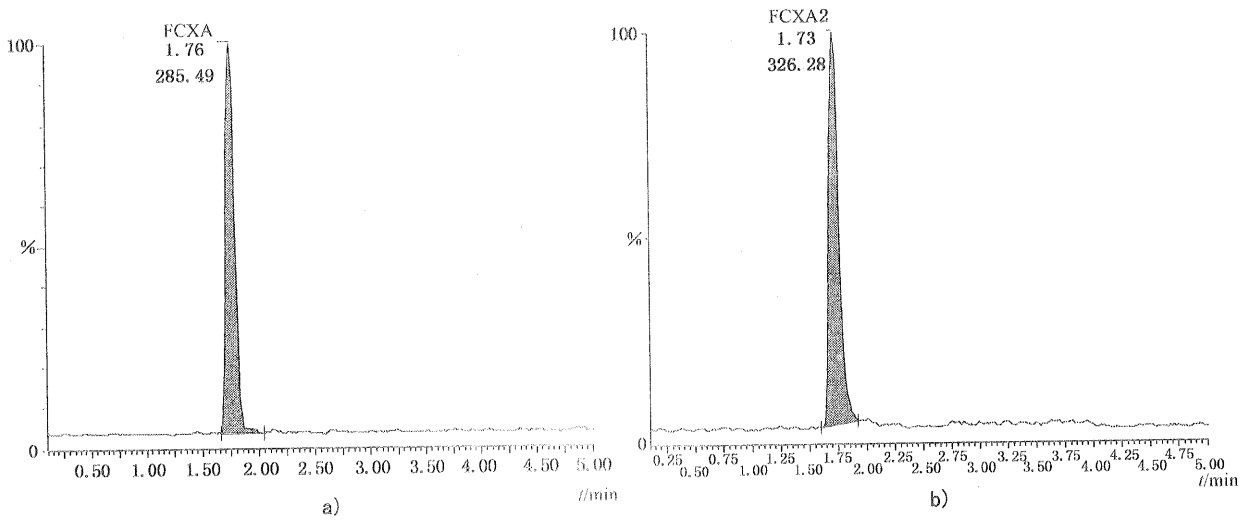


Figure B.2—Extracting ion current of MRM chromatograms
for working standard solution of flubendiamide (5 ng/mL)

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