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SN/T 5365—2022

出口植物源性食品中氟唑磺隆和氟吡磺隆残留量的测定　液相色谱-质谱/质谱法

Determination of flucarbazone-sodium and flucetosulfuron residues in foodstuffs of plant origin for export—LC-MS/MS method

2022-03-14 发布　　2022-10-01 实施

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前　言

本文件按照GB/T 1.1—2020《标准化工作导则　第1部分：标准化文件的结构和起草规则》的规定起草。

本文件由中华人民共和国海关总署提出并归口。

本文件起草单位：中华人民共和国伊宁海关技术中心、伊犁师范大学、中华人民共和国天津海关工业产品安全技术中心、中华人民共和国成都海关技术中心、中华人民共和国天津海关动植物与食品检测中心。

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出口植物源性食品中氟唑磺隆和氟吡磺隆残留量的测定　液相色谱-质谱/质谱法

1　范围

本文件规定了出口植物源性食品中氟唑磺隆和氟吡磺隆残留量的液相色谱-质谱/质谱测定方法。

本文件适用于菠菜、芹菜、土豆、西红柿、苹果、桃子、葡萄、糙米、小麦、核桃、大米等植物源性食品中氟唑磺隆和氟吡磺隆残留量的检测和确证。

2　规范性引用文件

下列文件中的内容通过文中的规范性引用而构成本文件必不可少的条款。其中，注日期的引用文件，仅该日期对应的版本适用于本文件；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 2763—2019　食品安全国家标准　食品中农药最大残留限量

GB/T 6682　分析实验室用水规格和试验方法

3　术语和定义

本文件没有需要界定的术语和定义。

4　方法提要

样品经乙腈-甲苯(90＋10)(*v*/*v*)提取、分散固相萃取净化后，液相色谱-质谱/质谱检测，外标法定量。

5　试剂和材料

除另有规定外，所用试剂均为分析纯，水为符合 GB/T 6682 规定的一级水。

5.1　乙腈：色谱纯。

5.2　乙酸铵：优级纯。

5.3　甲酸：优级纯。

5.4　甲苯：优级纯。

5.5　氯化钠。

5.6　0.1%甲酸-5 mmol/L 乙酸铵水溶液：准确移取 1.0 mL 甲酸(5.3)，用水定容至 1 000 mL；准确称取 0.375 g 乙酸铵(5.2)，加入至定容液中，混匀。

5.7　乙腈-甲苯(90＋10)(*v*/*v*)：取甲苯(5.4)100 mL，用乙腈(5.1)定容至 1 000 mL，混匀。

5.8　滤膜：0.22 μm，有机系。

5.9　十八烷基硅胶填料(C_{18} 填料)：40 μm。

5.10　石墨化碳黑(GCB)：120 mesh ～400 mesh。

5.11　氟唑磺隆标准物质：CAS 号 145026-88-6。
5.12　氟吡磺隆标准物质：CAS 号 412928-75-7。
5.13　标准储备溶液：准确称取 10 mg(精确至 0.1 mg)氟唑磺隆标准物质(5.11)和氟吡磺隆标准物质(5.12)，用乙腈分别配制成 1 000 μg/mL 的标准储备液，避光 4 ℃保存，有效期 6 个月。
5.14　中间混合标准溶液：分别吸取适量的标准储备液(5.13)，用乙腈稀释，配置浓度均为 10 μg/mL 的中间混合标准溶液，避光 4 ℃保存，有效期 2 个月。
5.15　基质空白溶液：将不同基质的阴性空白样品，按照 8.1 提取和 8.2 净化后，得到基质空白溶液。
5.16　基质匹配工作溶液：根据需要用基质空白溶液(5.15)稀释中间混合标准溶液(5.14)至合适浓度的基质匹配工作溶液，现配现用。

6　仪器和设备

6.1　液相色谱-质谱/质谱仪：配有电喷雾离子源(ESI)。
6.2　分析天平：感量分别为 0.1 mg 和 0.01 g。
6.3　组织捣碎机。
6.4　振荡器。
6.5　离心机：8 000 r/min。
6.6　离心管：50 mL 和 2 mL。
6.7　涡旋器。

7　试样制备与保存

蔬菜、水果的取样部位按照 GB 2763—2019 的规定执行，取样品 500 g，用组织捣碎机均质，充分混匀，均分为 2 份，装入洁净的容器，密封并标明标记，于－18 ℃以下冷冻保存。

坚果样品去壳取全果 500 g，用组织捣碎机均质，充分混匀，均分为 2 份，装入洁净的容器，密封并标明标记，于 0 ℃～4 ℃条件下保存。

谷物类样品取代表性试验 500 g，粉碎后使其全部可通过 425 μm 的标准网筛，均分为 2 份，装入洁净的容器，密封并标明标记，于 0 ℃～4 ℃条件下保存。

8　测定步骤

8.1　提取

8.1.1　蔬菜、水果

称取 5 g 试样(精确至 0.01 g)于 50 mL 离心管中，加入 5 mL 水涡旋 30 s，加入 10 mL 乙腈-甲苯(90＋10)(*v*/*v*)，水平振荡 20 min，加入 4 g 氯化钠(5.5)，水平振荡 5 min，8 000 r/min 离心 5 min，取上清液 1.0 mL 于 2 mL 离心管，待净化。

8.1.2　谷物、坚果

称取 2.5 g 试样(精确至 0.01 g)于 50 mL 离心管中，加入 7 mL 水涡旋 30 s，加入 10 mL 乙腈-甲苯(90＋10)(*v*/*v*)，水平振荡 20 min，加入 4 g 氯化钠(5.5)，水平振荡 5 min，8 000 r/min 离心 5 min，取上清液 1.0 mL 于 2 mL 离心管，待净化。

8.2 净化

蔬菜、水果的待净化上清液中加入 C_{18} 填料(5.9)50 mg、石墨化碳黑 GCB(5.10)30 mg，涡旋 30 s，8 000 r/min 离心 2 min，上清液过 0.22 μm 滤膜，滤液待测。

谷物、坚果的待净化上清液中加入 C_{18} 填料(5.9) 50 mg，涡旋 30 s，8 000 r/min 离心 2 min，上清液过 0.22 μm 滤膜，滤液待测。

8.3 测定

8.3.1 液相色谱条件

液相色谱参考条件如下：

a) 色谱柱：C_{18} 柱，2.1 mm×100 mm，3 μm 或相当；

b) 流动相 A：0.1%甲酸-5 mmol/L 乙酸铵水溶液(5.6)；流动相 B：乙腈；洗脱梯度见表 1；

c) 柱温：30 ℃；

d) 进样体积：10 μL。

表 1 流动相及梯度洗脱条件

时间/min	流速/(μL/min)	流动相 A/%	流动相 B/%
0.00	250	90.0	10.0
1.00	250	90.0	10.0
1.50	250	50.0	50.0
5.50	250	10.0	90.0
5.60	250	90.0	10.0
10.00	250	90.0	10.0

8.3.2 质谱条件

质谱参考条件如下：

a) 电离方式：电喷雾电离(ESI)；

b) 干燥气温度：300 ℃；

c) 干燥气流量：9.0 L/min；

d) 鞘气温度：250 ℃；

e) 鞘气流速：11.0 L/min；

f) 毛细管电压：4 kV；

g) 扫描方式：正离子扫描。

定性离子对、定量离子对、碰撞能量、碎裂电压等条件见表 2。

表 2　化合物定性离子对、定量离子对、碰撞能量、碎裂电压、保留时间

序号	化合物	保留时间/min	定性离子/(m/z)	碎裂电压/V	碰撞能量/eV
1	氟唑磺隆	4.69	397.1/130.0*;397.1/115.0	80	10;58
2	氟吡磺隆	5.69	488.5/156.0*;488.5/273.0	120	14;18
注：带“*”为定量离子对。					

8.4　定性测定

按照上述条件测定样品和混合基质标准工作溶液，如果待测样品中化合物色谱峰的保留时间与标准溶液相比变化范围应在±2.5%之内，定性离子均出现且信噪比大于等于 3，定性离子对的相对丰度与浓度相当混合基质标准工作液的相对丰度一致，相对丰度偏差不超过表 3 的规定，可判断样品中存在相应的被测物。

表 3　定性时相对离子丰度的最大允许范围

相对离子丰度/%	>50	>20～50	10～20	≤10
允许的相对偏/%	±20	±25	±30	50

8.5　定量测定

根据试样中被测物的含量，选择浓度相近的基质匹配工作溶液，待测物的响应值在仪器检测的线性范围内，如果待测物含量超过标准曲线范围，应用基质空白溶液稀释到合适浓度后分析。在上述仪器条件下，氟唑磺隆和氟吡磺隆的多重反应监测色谱图参见附录 A 中图 A.1～图 A.2，保留时间亦可参照附录 A 中图 A.1～图 A.2。

8.6　空白试验

除不称取试样外，均按上述步骤操作。

9　结果计算和表述

试样中氟唑磺隆和氟吡磺隆的残留量按式(1)计算，计算结果需扣除空白值。

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

式中：

X　——试样中被测物的含量，单位为微克每千克(μg/kg)；

c　——待测组分相应值在标准曲线上计算得到的浓度，单位为纳克每毫升(ng/mL)；

V　——提取液的体积，单位为毫升(mL)；

m　——样品称样量，单位为克(g)；

1 000 ——换算系数。

计算结果保留两位有效数字。

10 定量限

本方法氟唑磺隆和氟吡磺隆的定量限均为 5.0 μg/kg。

11 回收率和精密度

本方法对波菜、芹菜、土豆、西红柿、苹果、桃子、葡萄、糙米、小麦、核桃、大米等基质进行添加回收率试验，不同添加浓度范围内氟唑磺隆和氟吡磺隆的回收率资料参见附录 B。

附 录 A
（资料性）
氟唑磺隆和氟吡磺隆标准溶液的多反应监测（MRM）色谱图

氟唑磺隆和氟吡磺隆标准溶液的多反应监测（MRM）色谱图见图 A.1～图 A.2。

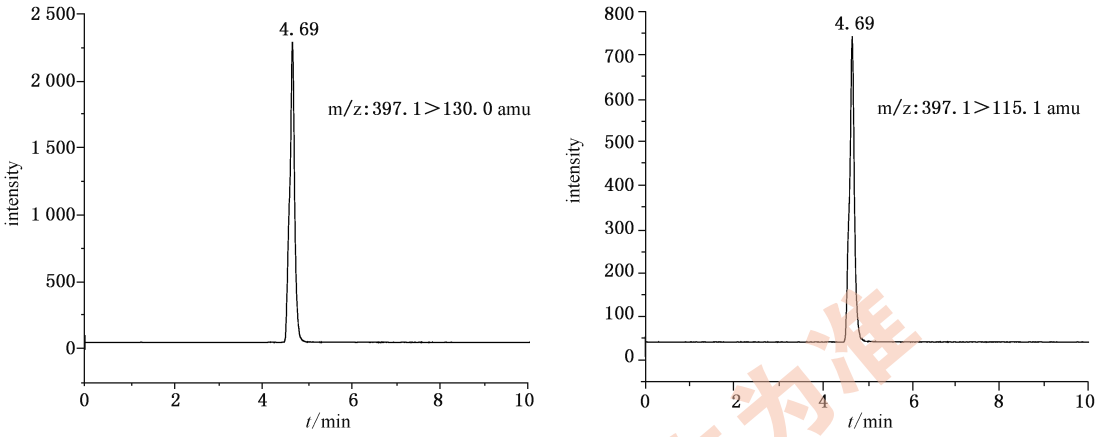

图 A.1 氟唑磺隆标准溶液（10 ng/mL）的反应监测（MRM）色谱图

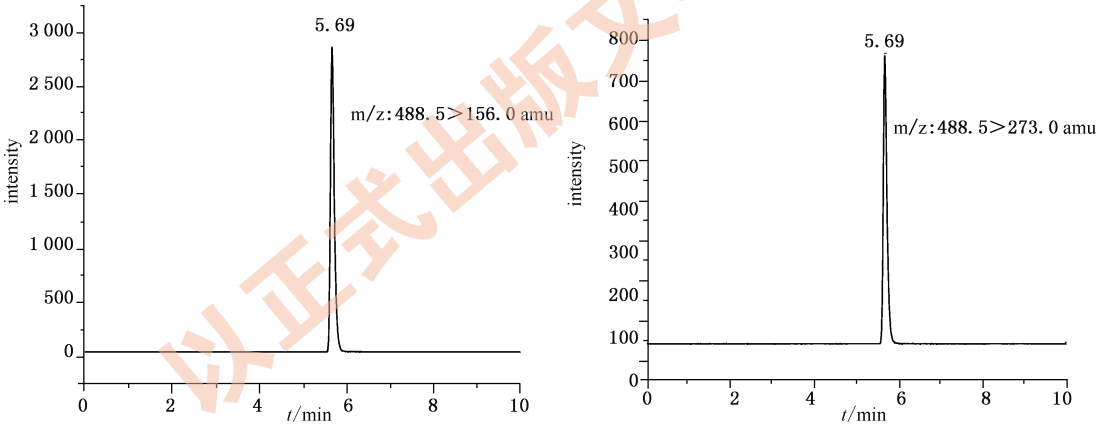

图 A.2 氟吡磺隆标准溶液（10 ng/mL）的反应监测（MRM）色谱图

附 录 B
（资料性）
添加回收率汇总表

氟唑磺隆和氟吡磺隆在波菜、芹菜、土豆、西红柿、苹果、桃子、葡萄、糙米、小麦、核桃、大米中的添加水平、平均回收率和相对标准偏差(RSD)参见表 B.1。

表 B.1 11 种基质中氟唑磺隆和氟吡磺隆的添加水平、平均回收率和相对标准偏差(RSD)(n=6)

基质种类	氟唑磺隆			氟吡磺隆		
	加标量 μg/kg	平均回收率 %	RSD %	加标量 μg/kg	平均回收率 %	RSD %
菠菜	5	83.80	9.6	5	81.57	6.6
	10	91.39	6.6	10	82.30	3.6
	20	95.62	5.7	20	103.48	13.0
	100	96.04	3.3	100	98.98	8.2
芹菜	5	82.53	10.1	5	92.73	12.1
	10	103.18	9.8	10	100.15	13.1
	20	100.62	5.8	20	98.55	11.6
	100	93.11	11.2	100	94.78	11.7
土豆	5	89.07	9.5	5	89.97	11.3
	10	93.47	9.0	10	96.22	7.9
	20	98.50	7.2	20	87.97	11.2
	100	101.18	12.9	100	97.21	6.6
西红柿	5	92.93	12.9	5	88.73	11.5
	10	104.38	3.5	10	90.02	9.8
	20	102.14	5.3	20	88.25	5.9
	100	107.00	4.3	100	91.84	6.4
苹果	5	87.43	13.2	5	84.50	10.0
	10	114.15	3.8	10	89.15	9.5
	20	103.99	7.1	20	89.70	6.3
	100	110.00	2.2	100	83.94	7.5
桃子	5	87.73	10.4	5	80.67	5.3
	10	106.80	9.0	10	78.70	6.4
	20	105.50	4.5	20	77.40	3.3
	100	112.04	5.8	100	92.39	2.5

表 B.1　11 种基质中氟唑磺隆和氟吡磺隆的添加水平、平均回收率和相对标准偏差(RSD)(n=6)(续)

基质种类	氟唑磺隆			氟吡磺隆		
	加标量 μg/kg	平均回收率 %	RSD %	加标量 μg/kg	平均回收率 %	RSD %
葡萄	5	91.30	13.5	5	82.57	9.3
	10	96.00	12.0	10	81.00	5.0
	20	107.85	4.7	20	18.11	12.9
	100	111.47	8.6	100	90.68	8.4
糙米	5	85.67	13.8	5	86.47	13.7
	10	95.53	13.1	10	91.87	12.8
	20	92.57	10.5	20	114.86	12.7
	100	87.48	2.9	100	105.11	12.8
小麦	5	88.30	14.8	5	86.53	13.0
	10	103.58	14.9	10	103.17	12.9
	20	92.57	10.5	20	88.58	6.2
	100	78.50	4.1	100	89.65	14.3
核桃	5	82.97	12.4	5	82.03	12.7
	10	92.45	14.4	10	94.08	14.7
	20	106.73	12.2	20	93.93	10.2
	100	90.62	2.7	100	84.20	14.8
大米	5	81.17	12.3	5	82.77	10.6
	10	93.90	14.2	10	95.57	13.1
	20	95.93	12.0	20	95.13	10.2
	100	93.60	6.8	100	84.21	9.1

Foreword

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

This standard was proposed by and was under the charge of General Administration of Customs of the People's Republic of China.

This standard was drafted by Yining Customs Technical Center, Yili Normal University, Tianjin Customs Industrial Product Safety Technical Center, Tianjin Customs Animal and plant and Food Inspection Center, Chengdu Customs Technical Center.

The main drafters of this standard are Li Fang, Su Youzhi, Fen Xinzhong, Xu Yingjie, Wang Xinglei, Zhou Lei, Liu Jun, Xiao Yabing, Lei Hongqin, Li Yanmei, Luo qiong, Yuan Xiaowen, Shang Shuang, Zhou Jun, Lu Ping.

Determination of flucarbazone-sodium and flucetosulfuron residues in foodstuffs of plant origin for export—LC-MS/MS method

1 Scope

This standard specifies the method of determination of flucarbazone-sodium and flucetosulfuron residues in foodstuffs of plant origin by LC-MS/MS method for export.

This standard is applicable to the determination and confirmation of flucarbazone-sodium and flucetosulfuron residues in spinach, celery, potatoes, tomatoes, apples, peaches, grapes, brown rice, wheat, walnuts and rice of plant origin foods.

2 Normative References

The following documents is necessary for this standard. For dated reference, only dated editions should apply to this standard. For undated reference, the latest edition of the normative document referred to applies.

GB 2763—2019 National food safety standards—Maximum pesticide residue limits for pesticides in food.

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

3 Terms and definitions

There is no term or definition to be defined in this document.

4 Principle

The test samples were extracted by acetonitrile-toluene (90+10) (*v*/*v*), purified by dispersion-solid phase extraction, detected by liquid chromatography-mass spectrometry (LC-MS/MS), and quantified by curve external standard method.

5 Reagents and materials

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

5.1 Acetonitrile: Chromatographic Grade.

5.2 Ammonium acetate: Guaranteed reagent Grade.

5.3 Formic acid: Guaranteed reagent Grade.

5.4 Toluene: Guaranteed reagent Grade.

5.5 Sodium chloride.

5.6 0.1% formic-5 mmol/L ammonium acetate solution: Dilute formic acid (5.3) 1 mL to 1 000 mL with water; accurately weigh 0.375 g ammonium acetate (5.2), add it to the constant volume solution, and mix well.

5.7 Acetonitrile-toluene (90+10) (*v*/*v*): Dilute toluene (5.4) 100 mL with acetonitrile (5.1) solution to 1 000 mL.

5.8 Filtration membrane: 0.22 μm, nylon membrane.

5.9 Octadecyl silica gel filler (C_{18} filler): 40 μm.

5.10 Graphitized carbon GCB: 120～400 mesh.

5.11 Flucarbazone-sodium standard substance: CAS 145026-88-6.

5.12 Flucetosulfuron standard substance: CAS 412928-75-7.

5.13 Standard reserve solution: 10 mg (accurate to 0.1 mg) flucarbazone-sodium standard substance (5.11) and flucetosulfuron standard substance (5.12) was accurately weighed and prepared into 1 000 μg/mL standard reserve solution with acetonitrile, respectively. The solution was stored at 4 ℃ away from light and was valid for 6 months.

5.14 Intermediate mixed standard solution: appropriate amount of standard reserve solution (5.13) was absorbed and diluted with acetonitrile. Intermediate mixed standard solution with concentration of 10 μg/mL was prepared and stored at 4 ℃ away from light for 2 months.

5.15 Blank matrix solution: blank matrix solution was obtained after the negative blank samples of

different substrates were extracted and purified according to 7.1 and 7.2.

5.16 Matrix matching working solution: Dilute the medium mixed standard solution (5.14) to the appropriate concentration of matrix matching working solution with matrix blank solution (5.15) as required, need compound at once when using.

6 Apparatus and equipment

6.1 Liquid chromatograhy with electrospray ionization mass spectrometry.

6.2 Balances(0.1 mg, 0.01 g).

6.3 Tissue blender.

6.4 Vibrator.

6.5 Centrifuge: 8 000 r /min.

6.6 Centrifuge tubes: 50 mL and 2 mL.

6.7 Vortex device.

7 Sample preparation and storage

The sampling parts of vegetables and fruits shall be carried out in accordance with the provisions of GB 2763—2019. 500 g of samples are taken, homogenized with a tissue mower, thoroughly mixed, and divided into 2 parts. The samples are put into a clean container, sealed and marked, and frozen under −18 ℃.

The whole nut sample was shelled 500 g, homogenized with a tissue mower, mixed thoroughly, divided into 2 parts, put into a clean container, sealed and marked, and stored at 0 ℃～4 ℃.

500 g representative grain samples were crushed to pass through 425 μm standard mesh sieve. The samples were divided into 2 parts and placed in a clean container, sealed and marked. The samples were stored at 0 ℃～4 ℃.

8 Procedure

8.1 Extraction

8.1.1 Vegetables and fruits

Weight 5 g (accurate to 0.01 g) of the test sample into 50 mL centrifuge tube, add 5 mL water ,vortex for 30 s , 10 mL acetonitrile-toluene (90 + 10) (*v*/*v*) were added and horizontally shocked for 20 min, Then 4 g sodium chloride (5.5) were added and horizontally oscillated for 5 min,centrifuged at 8 000 r/min for 5 min. Take 1 mL supernatant into 2 mL plugged centrifuge tube for purification.

8.1.2 Grains and nuts

Weight 2.5 g (accurate to 0.01 g) of the test sample into 50 mL centrifuge tube, add 7 mL water , vortex for 30 s , 10 mL acetonitrile-toluene (90 + 10) (*v*/*v*) were added and horizontally shocked for 20 min, Then 4 g sodium chloride (5.5) were added and horizontally oscillated for 5 min,centrifuged at 8 000 r/min for 5 min. Take 1 mL supernatant into 2 mL plugged centrifuge tube for purification.

8.2 Clean-up

For vegetables and fruits, 50 mg C_{18} filler (5.9) and 30 mg GCB (5.10) were added into the supernatant to be purified. The supernatant was vortex for 30 s and centrifuged at 8 000 r/min for 2 min. The supernatant was taken and filtered through the 0.22 μm nylon filter film. The filtrate is ready for LC-MS/MS analysis.

C_{18} filler (5.9) 50 mg were added into grain and nut supernatant to be purified, vortex for 30 s and centrifuged at 8 000 r/min for 2 min. The supernatant was taken and filtered through the 0.22 μm nylon filter film. The filtrate is ready for LC-MS/MS analysis.

The grain and nut supernatant to be purified was added with C_{18} filler (5.9) 50 mg, vortex 30 s, centrifugation at 8 000 r/min for 2 min, and the supernatant was filtered through a 0.22 μm membrane, and the filtrate was tested.

8.3 Determination

8.3.1 LC operating condition

Reference LC operating condition were as follows:

a) Column: C_{18}, 3 μm, 100 mm × 2.1 mm(i. d); or the equivalent;

b) Mobile phase A：0.1% formic acid-5 mmol/L ammonium acetate solution（5.6）；mobile phase B：acetonitrile；gradient of mobile phase see table 1；

c) Column temperature：30 ℃；

d) Injection volume：10 μL.

Table 1—Gradiet of mobile phase

time/min	Flow rate/（μL/min）	Moblie phase A/%	Moblie phase B/%
0.00	250	90.0	10.0
1.00	250	90.0	10.0
1.50	250	50.0	50.0
5.50	250	10.0	90.0
5.60	250	90.0	10.0
10.00	250	90.0	10.0

8.3.2 MS operating condition

Reference LC operating condition were as follows：

a) Ion source：ESI；

b) Drying temperature：300 ℃；

c) Dry gas flow rate：9.0 L /min；

d) Sheath gas temperature：250 ℃；

e) Sheath gas flow rate：11.0 L /min；

f) Capillary voltage：4 kV；

g) Scanning mode：positive ion scanning.

Quantitative and qualitative ion pair，collision energy，fragmentor voltage are shown in Table 2.

Table 2—Quantitative and qualitative ion pair, collision energy, fragmentor voltage, retention time

NO.	Compound	Retention time/min	Quantitative and qualitative ion pair/(m/z)	Fragmentor voltage/V	Collision energy/eV
1	flucarbazone-sodium	4.69	397.1/130.0* ;397.1/115.0	80	10;58
2	flucetosulfuron	5.69	488.5/156.0* ;488.5/273.0	120	14;18
* Quantitative ion pair					

8.4 Qualification test

In accordance with the above conditions for the determination of samples and the mixed matrix standard working solution, the retention time of compounds in the sample to be tested should be within ±2.5% compared with the standard solution. Qualitative ions were all present, and the S/N ≥3,The relative abundance of the qualitative ion pair is consistent with the relative abundance of the mixed matrix standard working, the deviation of the relative abundance does not exceed the provisions in Table 3, it can be judged that there are corresponding measured objects in the sample.

Table 3—Maximum permitted tolerance of relative ion intensities for qualification

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

8.5 Quantification test

According to the content of the tested object in the sample, the standard solution of matrix with similar concentration should be selected. The response value of the tested object should be within the linear range of the instrument detection. If the content of the tested object exceeds the range of the standard curve, the matrix blank sample solution should be diluted to the appropriate concentration for analysis. Under the above conditions, the monitoring chromatograms of multiple reactions of flucarbazone-sodium and flucetosulfuron are shown in Figure A.1～A.2 in Annex A, and also the retention time.

8.6 Blank test

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

9 Calculation and expression of the result

The residual amounts of flucarbazone-sodium and flucetosulfuron in the samples were calculated according to Formula (1), Blank values should be deducted from the calculation results.

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

Type:

X The content of the measured object in the sample in micrograms per kilogram, (μg/kg);

c The concentration of the measured component solution obtained from the standard working curve, (ng/mL);

V The volume of the extract, (mL);

m The corresponding mass of the test sample, (g);

1 000 Conversion factor.

The calculation results retain two significant digits.

10 Limit of determination

The limit of detection (LOD) of flucarbazone-sodium and flucetosulfuron were 5.0 μg/kg.

11 Recovery and precision

The addition recovery test was conducted on the matrix of spinach, celery, potato, tomato, apple, peach, grape, brown rice, wheat, rice, walnut and so on. The recovery data of different matrixs with different addition levels were shown in Annex B.

Appendix A
(Informative)
MRM chromatogram of flucarbazone-sodium and flucetosulfuron

MRM chromatogram of flucarbazone－sodium and flucetosulfuron see Figure A.1～Figure A.2.

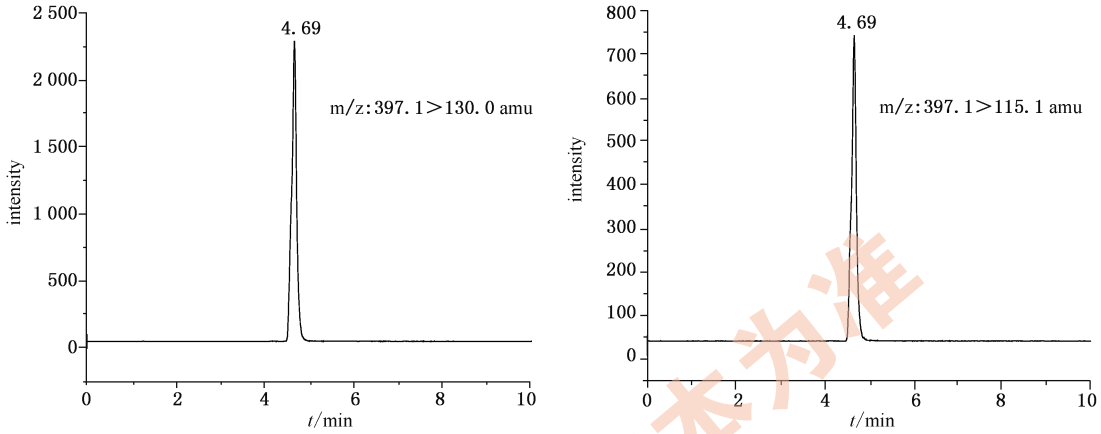

Figure A.1 MRM chromatogram of flucarbazone-sodium(10 ng/mL)

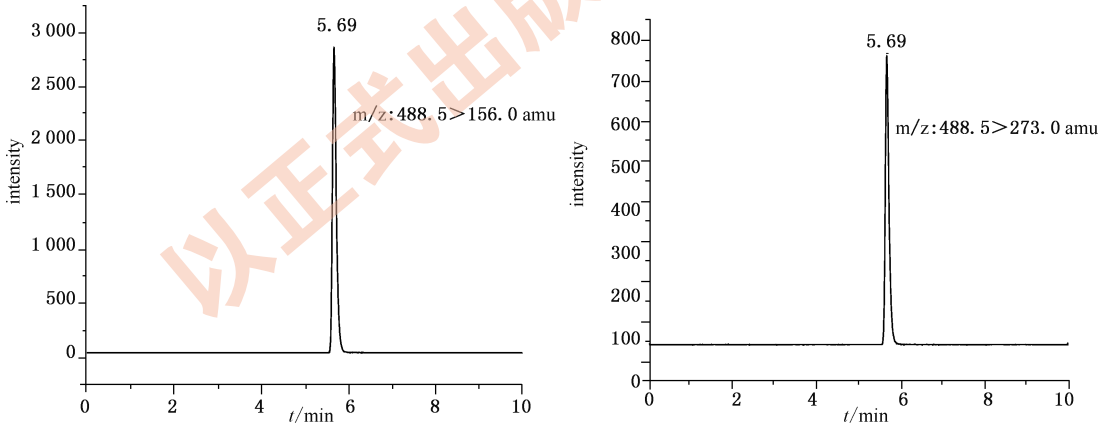

Figure A.2 MRM chromatogram of flucetosulfuron(10 ng/mL)

Appendix B
(Informative)
Azithromycin and its internal standard chromatogram

Spiked level, average recovery and relative standard deviation (RSD) of flucarbazone-sodium and flucetosulfuron in spinach, celery, potato, tomato, apple, peach, grape, brown rice, wheat, walnut and rice see Table B.1.

Table B.1—Spiked level, average recovery and relative standard deviation (RSD) of flucarbazone-sodium and flucetosulfuron in 11 kinds of matrix(n=6)

Matrix	Flucarbazone-sodium			Flucetosulfuron		
	Spiked level μg/kg	Average recovery %	RSD %	Spiked level μg/kg	Average recovery %	RSD %
spinach	5	83.80	9.6	5	81.57	6.6
	10	91.39	6.6	10	82.30	3.6
	20	95.62	5.7	20	103.48	13.0
	100	96.04	3.3	100	98.98	8.2
celery	5	82.53	10.1	5	92.73	12.1
	10	103.18	9.8	10	100.15	13.1
	20	100.62	5.8	20	98.55	11.6
	100	93.11	11.2	100	94.78	11.7
potato	5	89.07	9.5	5	89.97	11.3
	10	93.47	9.0	10	96.22	7.9
	20	98.50	7.2	20	87.97	11.2
	100	101.18	12.9	100	97.21	6.6
tomato	5	92.93	12.9	5	88.73	11.5
	10	104.38	3.5	10	90.02	9.8
	20	102.14	5.3	20	88.25	5.9
	100	107.00	4.3	100	91.84	6.4
apple	5	87.43	13.2	5	84.50	10.0
	10	114.15	3.8	10	89.15	9.5
	20	103.99	7.1	20	89.70	6.3
	100	110.00	2.2	100	83.94	7.5
peach	5	87.73	10.4	5	80.67	5.3
	10	106.80	9.0	10	78.70	6.4
	20	105.50	4.5	20	77.40	3.3
	100	112.04	5.8	100	92.39	2.5

Table B.1—Spiked level, average recovery and relative standard deviation (RSD) of flucarbazone-sodium and flucetosulfuron in 11 kinds of matrix (n=6) (Continued)

Matrix	Flucarbazone-sodium			Flucetosulfuron		
	Spiked level μg/kg	Average recovery %	RSD %	Spiked level (μg/kg)	Average recovery %	RSD %
grape	5	91.30	13.5	5	82.57	9.3
	10	96.00	12.0	10	81.00	5.0
	20	107.85	4.7	20	18.11	12.9
	100	111.47	8.6	100	90.68	8.4
brown rice	5	85.67	13.8	5	86.47	13.7
	10	95.53	13.1	10	91.87	12.8
	20	92.57	10.5	20	114.86	12.7
	100	87.48	2.9	100	105.11	12.8
wheat	5	88.30	14.8	5	82.03	12.7
	10	103.58	14.9	10	94.08	14.7
	20	92.57	10.5	20	93.93	10.2
	100	78.50	4.1	100	84.20	14.8
walnut	5	82.97	12.4	5	82.77	10.6
	10	92.45	14.4	10	95.57	13.1
	20	106.73	12.2	20	95.13	10.2
	100	90.62	2.7	100	84.21	9.1
rice	5	81.17	12.3	5	86.53	13.0
	10	93.90	14.2	10	103.17	12.9
	20	95.93	12.0	20	88.58	6.2
	100	93.60	6.8	100	89.65	14.3

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