



中华人民共和国出入境检验检疫行业标准

SN/T 4257—2015

出口食品中抗倒酯、脱叶磷、坐果安、 赤霉素残留量的测定 液相色谱-质谱/质谱法

Determination of trinexapac-ethyl, S,S,S-tributylphosphorotrithioate,
2-(3-Chlorophenoxy)propionic acid, gibberellin residues in foodstuffs
for export—LC-MS/MS method

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前 言

本标准按照 GB/T 1.1—2009 给出的规则起草。

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：中华人民共和国吉林出入境检验检疫局。

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出口食品中抗倒酯、脱叶磷、坐果安、 赤霉素残留量的测定 液相色谱-质谱/质谱法

1 范围

本标准规定了出口食品中抗倒酯、脱叶磷、坐果安、赤霉素残留量的液相色谱-质谱/质谱检测方法。本标准适用于苹果、菠菜、圆葱中抗倒酯、脱叶磷、坐果安、赤霉素残留量的测定和确证。

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

试样中残留的抗倒酯、脱叶磷、坐果安、赤霉素采用乙腈提取,HLB 固相萃取柱净化,液相色谱-质谱/质谱仪测定和确证,外标法定量。

4 试剂和材料

4.1 对试剂和材料的要求

除另有规定外,所用试剂均为分析纯,水为 GB/T 6682 规定的一级水。

4.2 试剂和材料

本方法所用的试剂和材料如下:

- 4.2.1 乙腈:HPLC 级。
- 4.2.2 甲醇:HPLC 级。
- 4.2.3 甲酸:HPLC 级。
- 4.2.4 氯化钠。
- 4.2.5 溶解液:甲醇-水(1+9,体积比)。
- 4.2.6 定容液:甲醇-水(1+1,体积比)。
- 4.2.7 标准品:标准品详细信息见附录 A 中表 A.1。
- 4.2.8 标准储备溶液:准确称取适量的抗倒酯、脱叶磷、坐果安、赤霉素标准物质,用乙腈将其配制成 100 $\mu\text{g}/\text{mL}$ 标准储备液,避光于 0 $^{\circ}\text{C}$ ~4 $^{\circ}\text{C}$ 保存。
- 4.2.9 标准中间液:用乙腈稀释标准储备溶液至 10 $\mu\text{g}/\text{mL}$,避光于 0 $^{\circ}\text{C}$ ~40 $^{\circ}\text{C}$ 保存。
- 4.2.10 标准工作溶液:根据检测要求临用前用空白样品提取液将标准中间液稀释成相应浓度的标准工作溶液。现用现配。

4.2.11 HLB 固相萃取柱(6 mL/500 mg):使用前先用 5 mL 甲醇、再用 5 mL 水依次活化。

4.2.12 微孔滤膜:0.22 μm 。

4.2.13 硅藻土:100 目~200 目。

5 仪器和设备

本方法所用的仪器设备如下:

5.1 液相色谱串联质谱仪,带电喷雾离子源(ESI)。

5.2 组织捣碎机。

5.3 旋转蒸发器。

5.4 高速均质器:18 000 r/min。

5.5 固相萃取仪(SPE)。

5.6 离心机:10 000 r/min。

5.7 涡旋混合器。

6 试样制备与保存

6.1 试样制备

取有代表性样品 500 g,将其切碎后,用组织捣碎机将样品加工成浆状,混匀,装入洁净的盛样容器内,密封并标明标记。

6.2 试样保存

试样于 0 $^{\circ}\text{C}$ ~4 $^{\circ}\text{C}$ 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。

7 测定步骤

7.1 提取

称取 20 g(精确至 0.01 g)均匀试样置于 100 mL 离心管中,加入 15 g 氯化钠、5 g 硅藻土和 10 mL 水,用涡旋混合器初步混匀。然后加入 50 mL 乙腈,于高速均质器上匀浆 2 min。将离心管以 5 000 r/min 离心 5 min,吸取上层提取液于 250 mL 心形瓶中。重复上述操作提取两次,合并提取液于 250 mL 心形瓶中。于 35 $^{\circ}\text{C}$ 下减压蒸干,准确加入甲醇+水(1+9,体积比)溶解液 10.0 mL,充分溶解残渣,供固相萃取仪净化。

7.2 净化

取 5.0 mL 待净化液于已活化好的 HLB 固相萃取柱中,自然滴下,弃去流出液。用 10 mL 水淋洗,自然滴下,弃去。再用 5 mL 甲醇+水(1+9,体积比)溶解液淋洗,自然滴下,弃去。最后用 15 mL 甲醇洗脱(保持约 1 mL/min),收集流出液于 100 mL 心形瓶中,于 35 $^{\circ}\text{C}$ 下旋转浓缩至干,准确加入甲醇+水(1+1,体积比)2.0 mL 定容液充分溶解残渣,过 0.22 μm 有机滤膜后供液相色谱串联质谱(LC-MS/MS)测定。

7.3 测定

7.3.1 液相色谱条件

液相色谱参考条件如下：

- a) 色谱柱： C_{18} 柱， $3.5\text{ }\mu\text{m}$ ， $150\text{ mm}\times 2.1\text{ mm}$ (内径)，或相当者；
- b) 流动相：乙腈-0.1%甲酸溶液，梯度洗脱条件参见表 1；
- c) 流速： $200\text{ }\mu\text{L}/\text{min}$ ；
- d) 柱温：室温；
- e) 进样量： $10\text{ }\mu\text{L}$ 。

表 1 液相色谱的梯度洗脱条件

时间 min	流速 $\mu\text{L}/\text{min}$	0.1%甲酸水溶液 %	乙腈 %
0.0	200	95	5
1.5	200	95	5
2.0	200	5	95
10.0	200	0	100
15.0	200	0	100
15.10	200	95	5
30.00	200	95	5

7.3.2 质谱条件

质谱参考条件如下：

- a) 离子化模式：电喷雾电离正离子模式(ESI+)与负离子模式(ESI-)；
- b) 检测方式：多反应监测(MRM)；
- c) 分辨率：单位质量分辨率；
- d) 其他参考质谱条件：参见附录 B。

7.3.3 定性测定

在相同实验条件下，样液中被测化合物的保留时间，与标准溶液的保留时间偏差在 $\pm 2.5\%$ 之内；且样品中各组分定性离子的相对丰度与浓度接近的标准工作溶液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，则可判定为样品中存在对应的被测物。

表 2 定性时相对离子丰度的最大允许偏差

相对离子丰度/%	>50	$>20\sim 50$	$>10\sim 20$	≤ 10
允许的相对偏差/%	± 20	± 25	± 30	± 50

7.3.4 定量测定

在仪器最佳工作条件下，对标准工作溶液进样。用标准工作曲线按外标法定量，样品溶液中被测物

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的响应值均应在仪器测定的线性范围内。在上述色谱条件下 4 种激素类药物的参考保留时间:赤霉素 8.4 min、坐果安 9.6 min、抗倒酯 10.1 min、脱叶磷 14.8 min,4 种激素类标准溶液的多反应监测(MRM)色谱图参见附录 C。4 种激素类原药的分子式、相对分子质量、结构式见附录 A。

7.4 空白实验

除不加试样外,均按上述测定步骤进行。

8 结果计算和表述

用色谱数据处理机或按式(1)分别计算试样中被测化合物的含量:

$$X_i = \frac{A_i \times c_{si} \times V}{A_{si} \times m}$$

.....(1)

式中:

- X_i —— 试样中每种被测化合物的含量,单位为毫克每千克(mg/kg);
- A_i —— 样液中每种被测化合物的峰面积;
- c_{si} —— 标准工作液中每种化合物的浓度,单位为微克每毫升($\mu\text{g/mL}$);
- V —— 样液最终定容体积,单位为毫升(mL);
- A_{si} —— 标准工作液中每种化合物的峰面积;
- m —— 最终样液所代表的试样量,单位为克(g)。

9 方法测定低限、回收率

9.1 测定低限

本方法的测定低限:脱叶磷为 0.002 mg/kg;抗倒酯、坐果安、赤霉素均为 0.01 mg/kg。

9.2 回收率

分别在苹果、菠菜、圆葱中添加抗倒酯、脱叶磷、坐果安、赤霉素药物,添加浓度及其回收率范围的实验数据见表 3。

表 3 三种食品中抗倒酯、脱叶磷、坐果安、赤霉素添加回收率范围

药物名称	添加浓度 mg/kg	回收率/%		
		苹果	菠菜	圆葱
抗倒酯	0.01	81.8~95.7	78.2~97.6	82.5~103.8
	0.05	80.9~96.5	78.1~98.7	83.6~97.2
	0.20	83.4~99.3	81.2~98.3	84.9~99.5
脱叶磷	0.002	50.8~80.9	54.1~83.6	51.7~80.4
	0.010	53.1~82.2	54.2~83.9	53.5~86.1
	0.050	53.3~85.2	54.1~85.0	52.5~83.6

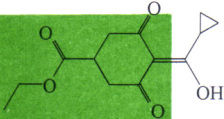
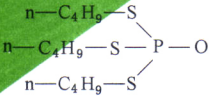
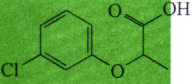
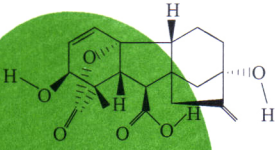
表 3 (续)

药物名称	添加浓度 mg/kg	回收率/%		
		苹果	菠菜	圆葱
坐果安	0.01	75.9~92.3	75.1~94.8	76.7~101.5
	0.05	78.4~96.7	77.5~96.2	76.3~98.5
	0.20	80.7~99.0	78.9~97.5	80.2~97.7
赤霉素	0.01	80.4~95.5	78.3~103.8	77.6~106.2
	0.05	82.9~96.6	78.6~97.5	79.9~96.9
	0.20	83.9~97.5	81.3~96.5	80.4~94.8

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附 录 A
(规范性附录)
抗倒酯、脱叶磷、坐果安、赤霉素原药信息

表 A.1 抗倒酯、脱叶磷、坐果安、赤霉素的分子式、相对分子质量、结构式、CAS 号

序号	药物名称	英文名称	分子式	结构式	相对分子质量	CAS 号
1	抗倒酯	Trinexapac-ethyl	$C_{13}H_{16}O_5$		252.27	95266-40-3
2	脱叶磷	S,S,S-tributylphosphorotrithioate	$C_{12}H_{27}OPS_3$		314.5	78-48-8
3	坐果安	2-(3-Chlorophenoxy)propionic acid	$C_9H_9ClO_3$		200.62	101-10-0
4	赤霉素	Gibberellin	$C_{19}H_{22}O_6$		346.38	77-06-5

附 录 B
(资料性附录)
质谱参考条件

表 B.1 参考质谱条件

质谱参数	正离子扫描参数值	负离子扫描参数值
喷雾电压 IS	5 500 V	—4 500 V
雾化气压力 GS1	50 Pa	50 Pa
气帘气压力 CUR	30 Pa	30 Pa
辅助气压力 GS2	50 Pa	50 Pa
离子源温度	550 °C	550 °C
碰撞气 CAD	6	6
碰撞室入口电压 EP	10 V	—10 V
碰撞室出口电压 CXP	14 V	—15 V

表 B.2 主要参考质谱参数

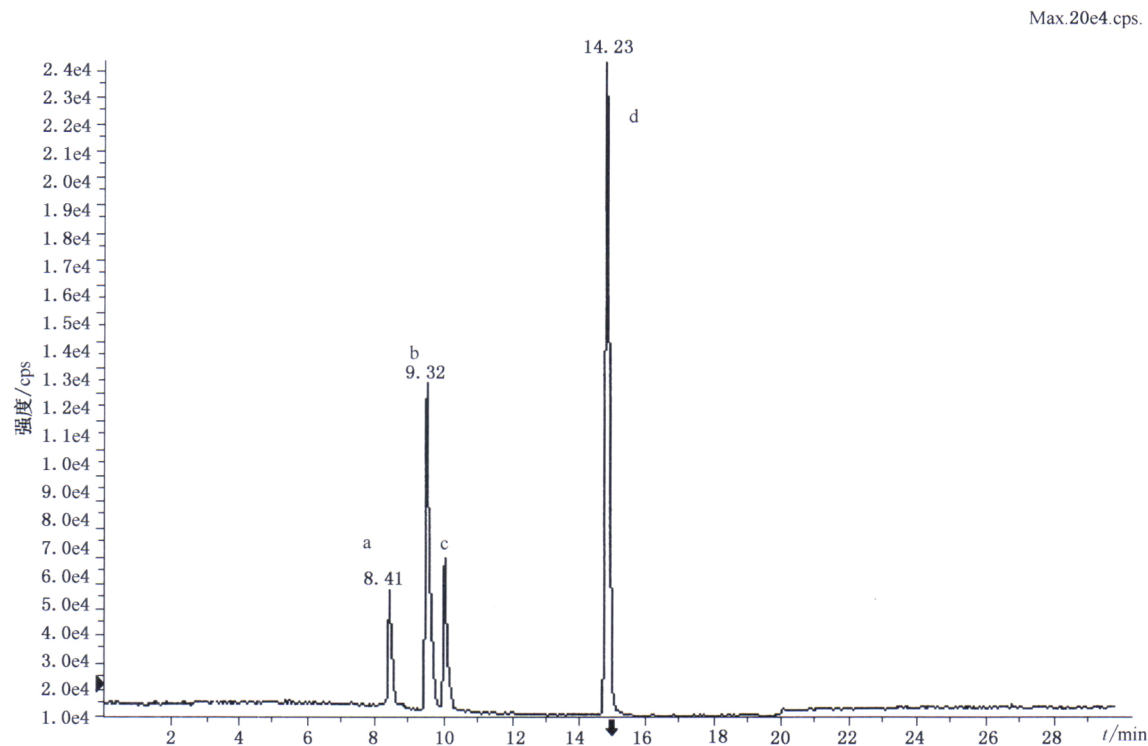
化合物	母离子 m/z	子离子 m/z	去簇电压 V	碰撞能量 V
赤霉素	345.2	143.1	—70	—36
		221.1	—70	—25
		239.1 *	—70	—23
坐果安	198.9	70.9	—47	—13
		126.3 *	—47	—17
抗倒酯	253.0	185.0	37	24
		207.0 *	37	17
脱叶磷	315.0	169.1 *	28	21
		225.1	28	16
		259.1	28	18

注：表中带 * 的离子为定量离子；对于不同质谱仪器，仪器参数可能存在差异，测定前应将质谱参数优化到最佳。

非商业性声明：所提供参数是在安捷伦 1 100 液相色谱仪和 API4000 质谱仪上完成的，此处列出试验用仪器型号仅是为了提供参考，并不涉及商业目的，鼓励标准使用者尝试不同厂家和型号的仪器。

附 录 C
(资料性附录)

抗倒酯、脱叶磷、坐果安、赤霉素标准溶液的多反应监测(MRM)色谱图



说明：
a——赤霉素；
b——坐果安；
c——抗倒酯；
d——脱叶磷。

图 C.1 抗倒酯、脱叶磷、坐果安、赤霉素 10 ng/mL 标准溶液的多反应监测(MRM)色谱图

Foreword

This standard was drafted in accordance with the requirements of GB/T 1.1—2009.

Please note that this standard some material may be involved in patent, this standard release mechanism does not assume the responsibility of identifying these patents.

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

This standard is under the charged of Certification and Accreditation Administration of the People's Republic of China.

This standard was drafted by Jilin Entry-Exit Inspection and Quarantine Bureau of P.R.China.

This main draftsman of standard: Rong Hui, Mu Jun, Hu Tingting, Kang Mingqin, Zhang Qi, Zhou Xiao, Ma Shumin, Wang Anying.

Determination of trinexapac-ethyl, S,S,S-tributylphosphorotrithioate, 2-(3-Chlorophenoxy)propionic acid, gibberellin residues in foodstuffs for export—LC-MS/MS method

1 Scope

This standard specifies the testing method of s,s,s-tributylphosphorotrithioate, trinexapac, gibberellin, 2-(3-chlorophenoxy)propionic acid residues in foods for import and export by liquid chromatography-mass-mass spectrometry (LC/MS/MS).

This standard is applicable to the determination and confirmation of residual content of trinexapac, gibberellin, s,s,s-tributylphosphorotrithioate, 2-(3-chlorophenoxy)propionic acid in apple, spinach, onion.

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 analytical laboratory use—Specification and test methods.

3 Principle

The test samples are extracted with acetonitrile. The extract is cleaned up by HLB (solid phase extraction-SPE). LC/MS/MS is applied for determination and confirmation with quantitation by external standard method.

4 Reagents and materials

4.1 Requirement

Unless otherwise specified, all the reagents used should be analytically pure, and "water" is GB/T 6682 distilled water.

4.2 Reagents and materials

The reagents used in the methods and materials are as follows:

4.2.1 Acetonitrile; HPLC grade.

4.2.2 Methanol; HPLC grade.

4.2.3 Formic acid; HPLC grade.

4.2.4 Sodium chloride.

4.2.5 Dissolved liquid; Methanol-Water (1+9, V/V).

4.2.6 Diluted liquid; Methanol-Water (1+1, V/V).

4.2.7 Standards: Standard details see Annex A Table A.1.

4.2.8 Standard stock solutions: Accurately weigh an adequate amount of trinexapac, gibberellin, s,s,s-tributylphosphorotrithioate, 2-(3-chlorophenoxy) propionic acid standards. Make 100 $\mu\text{g/mL}$ of standard stock solution with acetonitrile which is stored in a dark place of 0 $^{\circ}\text{C}$ ~4 $^{\circ}\text{C}$.

4.2.9 Standard intermediate working solutions: Dilute the standard stock solution with acetonitrile to 10 $\mu\text{g/mL}$ with acetonitrile which is stored in a dark place of 0 $^{\circ}\text{C}$ ~4 $^{\circ}\text{C}$.

4.2.10 Standard working solutions: Dilute the standard intermediate work solution with extract of blank sample to required concentration to acquire the related standard working solution on site.

4.2.11 HLB SPE cartridge (6 mL/500 mg); conditioned with 5 mL acetonitrile and then 5 mL water.

4.2.12 Micro-pore filter film; for organic; 0.22 μm .

4.2.13 Diatomite; 100~200 mesh.

5 Apparatus and equipments

The apparatus and equipment used in the method are as follows:

5.1 LC-MS/MS; equipped with electro-spray ion source (ESI).

5.2 Blender.

5.3 Rotary vacuum evaporator.

5.4 Homogenizer: 18 000 r/min.

5.5 Automatic SPE system.

5.6 Centrifuge: 10 000 r/min.

5.7 Vortex mixer.

6 Preparation and test samples

6.1 Preparation of sample

Take a representative sample 500 g, it chopped, mashed machine using tissue samples processed into paste, mix, containing sample into a clean container, sealed and marked with tags.

6.2 Storage of sample

Samples at 0 °C ~ 4 °C preservation, the sample preparation procedure, it shall occur to prevent sample contamination or residue content changes.

7 Determining procedure

7.1 Extraction

Weigh 20 g (accurate to 0.01 g) homogeneous sample placed 100 mL centrifuge tube, add 15 g NaCl, 5 g of diatomaceous earth and 10 mL of water, and mix with a vortex mixer initially. Then 50 mL of acetonitrile, homogenized at high speed homogenizer on 2 min. The tube to 5 000 r/min centrifugation 5 min, draw the upper extract in 250 mL heart-shaped bottle. Repeat extracted twice in 250 mL combined extracts heart-shaped flask. Evaporated under reduced pressure at 35 °C, accuracy methanol + water (1 + 9, V/V) dissolving liquid 10.0 mL, fully dissolve the residue purified for solid-phase extraction device.

7.2 Cleaning-up

Take 5.0 mL liquid to be purified in an activated good HLB solid phase extraction column, the natural drop, discard the effluent. Rinse with 10 mL of water, natural dripping discarded. Then 5 mL methanol + water (1 + 9, V/V) solution bath for natural dripping discarded. Finally eluted with 15 mL of methanol (for approximately 1 mL / min), collected in 100 mL of effluent heart-shaped flask is rotated at 35 °C was concentrated to dryness, accurate methanol + water (1 + 1, V/V) 2.0 mL constant

volume solution to dissolve the residue, after over 0.22 μm membrane for organic liquid chromatography tandem mass spectrometry (LC-MS/MS) determination.

7.3 Determination

7.3.1 LC operating condition

Reference HPLC conditions were as follows:

- a) LC column: C_{18} , 3.5 μm , 150 mm \times 2.1 mm (i.d.), or equivalent;
- b) Mobile phase: acetonitrile-0.1% formic acid solution (see Table 1);
- c) Flow rate: 200 $\mu\text{L}/\text{min}$;
- d) Column temperature: environmental temperature;
- e) Injector volume: 10 μL .

Table 1—LC gradient eluting conditions

Time min	Flow rate $\mu\text{L}/\text{min}$	0.1% Formic acid %	Acetonitrile %
0.0	200	95	5
1.5	200	95	5
2.0	200	5	95
10.0	200	0	100
15.0	200	0	100
15.10	200	95	5
30.00	200	95	5

7.3.2 MS/MS operating condition

Reference MS/MS operating conditions were as follows:

- a) Ion source: ESI+ and ESI-;
- b) Monitoring model: multiple reaction monitoring (MRM);
- c) Resolution: unit mass resolution;
- d) Other parameters: shown in Annex B.

7.3.3 Qualification

Under the same operating conditions, if the difference of the retention times between compounds in the sample and those in the standard solution shall be in-between $\pm 2.5\%$; the permitted tolerances of the ion pairs relative intensities shall not exceed the ranges listed in Table 2, the corresponding analytes in sample could be judged as existence.

Table 2—Maximum permitted tolerances of relative ion intensities while confirmation

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

7.3.4 Quantitation

Inject the standard solutions when the instrument is under optimized conditions. Quantitate the analytes with external standard method while the responding values of analytes in sample solutions are in the linear range. Under the described chromatographic conditions, the retention times of four phytohormones are gibberellin 8.4 min, s, s, s – tributylphosphorotrithioate 14.8 min, 2 – (3 – chlorophenoxy)propionic acid 9.6 min, and trinexapac 10.1 min. The MRM chromatogram of four phytohormones is shown in Annex C. The formulas, molecule weights, and structures of the four compounds are listed in Annex A.

7.4 Blank test

Undergo the blank test according to the above procedures excluding the sample.

8 Calculation and report of the result

Calculate the content of analytes in sample by chromatographic station or according to the followed formula:

$$X_i = \frac{A_i \times c_{si} \times V}{A_{si} \times m} \dots\dots\dots (1)$$

where

X_i —the content of every analyte in the sample, milligram per kilogram, mg/kg;

A_i —the peak area of every analyte in the sample solution;

c_{si} —the concentration of every analyte in the standard working solution, microgram per milliliter, $\mu\text{g/mL}$;

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

A_{si} —the peak area of every analyte in the standard working solution;

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

The result of calculation should be deducted with blank value.

9 Limit of detection (LOD) and recovery of the method

9.1 LOD

The LOD of this method: S,S,S-tributylphosphorotrithioate is 0.002 mg/kg; others is 0.01 mg/kg.

9.2 Recovery

Respectively, apples, spinach, onion added Trinexapac, defoliation phosphorus, fruit Ann, GA drug concentration and recoveries adding the experimental data are shown in Table 3.

Table 3—The fortification levels and their recoveries in three food matrixs

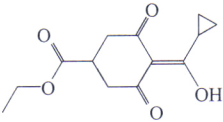
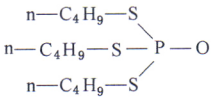
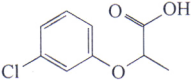
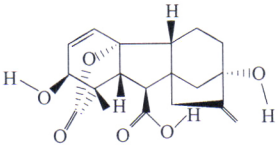
Compounds	Fortification Levels mg/kg	Recoveries/%		
		Apple	Spinach	Onion
Trinexapac	0.01	81.8~95.7	78.2~97.6	82.5~103.8
	0.05	80.9~96.5	78.1~98.7	83.6~97.2
	0.20	83.4~99.3	81.2~98.3	84.9~99.5
S,S,S-tributylphosphorotrithioate	0.002	50.8~80.9	54.1~83.6	51.7~80.4
	0.010	53.1~82.2	54.2~83.9	83.5~86.1
	0.050	53.3~85.2	54.1~85.0	52.5~83.6
2-(3-Chlorophenoxy)propionic acid	0.01	75.9~92.3	75.1~94.8	76.7~101.5
	0.05	78.4~96.7	77.5~96.2	76.3~98.5
	0.20	80.7~99.0	78.9~97.5	80.2~97.7
Gibberellin	0.01	80.4~95.5	78.3~103.8	77.6~106.2
	0.05	82.9~96.6	78.6~97.5	79.9~96.9
	0.20	83.9~97.5	81.3~96.5	80.4~94.8

Annex A

(Normative Annex)

**Trinexapac-ethyl, S, S, S-tributylphosphorotrithioate, 2-(3-Chlorophenoxy) propionic acid, gibberellin
original drug information**

Table A.1—Trinexapac-ethyl, S, S, S-tributylphosphorotrithioate, 2-(3-Chlorophenoxy) propionic acid, gibberellin molecular formula, molecular weight, structure, CAS number

No.	English name	Molecular formula	Structure	Molecular weight	CAS No.
1	Trinexapac-ethyl	$C_{13}H_{16}O_5$		252.27	95266-40-3
2	S, S, S-tributylphosphorotrithioate	$C_{12}H_{27}OPS_3$		314.5	78-48-8
3	2-(3-Chlorophenoxy) propionic acid	$C_9H_9ClO_3$		200.62	101-10-0
4	Gibberellin	$C_{19}H_{22}O_6$		346.38	77-06-5

Annex B
(Informative Annex)

Table B.1—MS conditions

MS conditions	ESI +	ESI –
Ion spray voltage(IS)	5 500 V	– 4 500 V
Nebulizer(GS1)	50 Pa	50 Pa
Curtain gas(CUR)	30 Pa	30 Pa
Auxiliary gas(GS2)	50 Pa	50 Pa
Ion temperature	550 ℃	550 ℃
Collision gas(CAD)	6	6
entrance potential(EP)	10 V	– 10 V
collision exit potential(CXP)	14 V	– 15 V

Table B.2—MS paramters

Compounds	Parents ions <i>m/z</i>	Daughter ions <i>m/z</i>	Declustering potential voltage(DP) V	collision gas energy(CE) V
Gibberellin	345.2	143.1	– 70	– 36
		239.1 *	– 70	– 23
		221.1	– 70	– 25
2-(3-Chlorophenoxy) propionic acid	198.9	126.3 *	– 47	– 17
		70.9	– 47	– 13
Trinexapac	253.0	207.0 *	37	17
		185.0	37	24
S,S,S-tributylphos phorotrithioate	315.0	169.1 *	28	21
		225.1	28	16
		259.1	28	18

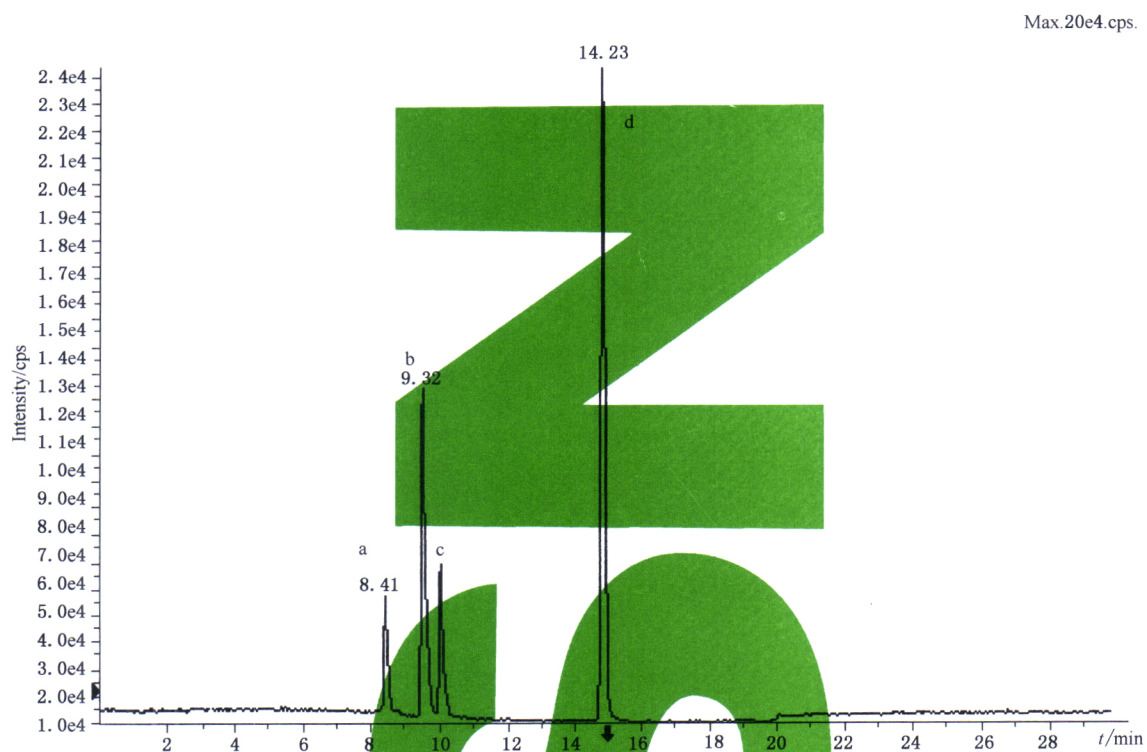
Note: The ions with * are quantitative. From different instruments, the parameters might vary. Optimization is recommended before determination.

Non-commercial statement: provide a parameter is completed in the Agilent 1100 liquid chromatograph and API4000 mass spectrometer, here is a list of test instrument type is only for reference, does not involve commercial purposes, to encourage users to try different types of instruments.

Annex C

(Informative Annex)

The MRM chromatogram of Gibberellin, 2-(3-Chlorophenoxy)propionic acid, Trinexapac, S, S, S-tributylphosphorotrithioate



Illustrate:

a—Gibberellin;

b—2-(3-Chlorophenoxy)propionic acid;

c—Trinexapac;

d—S, S, S-tributylphosphorotrithioate.

Figure C.1—MRM Chromatogram of the four phytohormones of 10 ng/mL standard solution