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中华人民共和国出入境检验检疫行业标准

SN/T 4778—2017

出口花粉中链霉素和双氢链霉素的测定方法 液相色谱-质谱/质谱法

Determination of streptomycin and dihydrostreptomycin in pollen for export—LC-MS/MS method

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中华人民共和国
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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：中华人民共和国江苏出入境检验检疫局。

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出口花粉中链霉素和双氢链霉素的测定方法 液相色谱-质谱/质谱法

1 范围

本标准规定了出口花粉中链霉素和双氢链霉素的液相色谱-质谱/质谱法测定方法。

本标准适用于出口松花粉、玉米花粉、茶花粉、葵花粉、油菜花粉和杂花粉等中链霉素和双氢链霉素的测定和确证。

2 规范性文件

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

样品中的链霉素和双氢链霉素经提取溶液提取，三氯甲烷沉淀样品中的蛋白质，清液过 C_{18} 小柱净化后用液相色谱-质谱/质谱法测定。外标法定量。

4 试剂和材料

除非另有说明，在分析中仅使用确认为分析纯的试剂，水采用 GB/T 6682 中规定的一级水。

4.1 甲醇：高效液相色谱级。

4.2 磷酸。

4.3 叔丁基甲基醚。

4.4 甲酸：高效液相色谱级。

4.5 正己烷。

4.6 三氯甲烷。

4.7 乙酸铵：$CH_3CO_2NH_4$，高效液相色谱级。

4.8 庚烷磺酸钠($C_7H_{15}SO_3Na$)。

4.9 磷酸钠[$Na_3PO_4 \cdot (H_2O)_{12}$]。

4.10 提取溶液：准确称取 10.1 g 庚烷磺酸钠(4.8)，4.1 g 磷酸钠(4.9)，用水溶解并转移至 1 000 mL 容量瓶中，加 5 mL 磷酸(4.2)，用水定容至刻度。

4.11 叔丁基甲基醚-正己烷溶液(4+1，体积比)：准确量取 400 mL 叔丁基甲基醚(4.3)，加入 100 mL 正己烷(4.5)，混合均匀。

4.12 甲酸溶液(5‰，体积比)：准确量取 5 mL 甲酸(4.4)，加入至 1 000 mL 容量瓶中，用水定容至刻度。

4.13 0.02 mol/L 乙酸铵溶液：准确称取 1.54 g 乙酸铵(4.7)，用水溶解并转移至 1 000 mL 容量瓶中，用水定容至刻度。

4.14 甲醇水溶液(3+7,体积比):准确量取 300 mL 甲醇,加入 700 mL 水,混合均匀。

4.15 链霉素标准品[Streptomycin Sulfate,$(C_{21}H_{39}N_7O_{12})_2 \cdot (H_2SO_4)_3$,CAS No:3810-74-0]纯度大于或等于 98 %。

4.16 双氢链霉素标准品[Dihydrostreptomycin Sesquisulfate Hydrate,$(C_{21}H_{41}N_7O_{12})_2 \cdot (H_2SO_4)_3$,CAS No:5490-27-7]纯度大于或等于 99%。

4.17 标准储备液:分别准确称取适量的链霉素、双氢链霉素标准品,用甲醇水溶液(4.14)配制成浓度为 1 mg/mL 的标准储备溶液。0 ℃～4 ℃保存。

4.18 混合标准中间溶液:分别准确移取一定体积的链霉素、双氢链霉素标准储备液(4.17),用甲醇水溶液(4.14)稀释为 10 μg/mL,0 ℃～4 ℃保存。

4.19 混合标准工作液:准确移取一定体积的混合标准中间溶液(4.18),用甲醇水溶液(4.14)稀释成 1.0 μg/mL,现用现配。

4.20 C_{18}小柱:500 mg,3 mL,或相当者。

4.21 微孔滤膜:水相,0.45 μm。

5 仪器和设备

5.1 液相色谱-串联质谱仪:配有电喷雾离子源(ESI)。

5.2 分析天平:感量分别为 0.01 g 和 0.01 mg。

5.3 高速离心机:8 000 r/min。

5.4 超声振荡器。

5.5 涡旋混合器。

5.6 氮吹仪。

5.7 具塞塑料离心管:50 mL。

5.8 SPE 固相萃取装置。

6 试样制备和保存

取有代表性的样品约 500 g,用磨碎机全部磨碎并通过 20 目筛,混匀,均分成两份作为试样,分别装入洁净的容器内,密封,标明标记,于常温下保存。

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 分析步骤

7.1 样品处理

7.1.1 提取

称取 2 g 试样(精确到 0.01 g)置于 50 mL 具塞塑料离心管中,加入提取溶液(4.10)20 mL,三氯甲烷 3 mL,于涡旋混匀器上混合 3 min,混合均匀后置于超声振荡器中超声 15 min,8 000 r/min 高速离心 5 min。取上层清液收集于塑料离心管中,供净化用。

7.1.2 净化

将 C_{18}小柱(预先用 5 mL 甲醇,5 mL 水活化)连接于 20 mL 塑料针筒下,将样液以 1 滴/s～2 滴/s 的流速全部通过 C_{18}小柱,在样液在小柱上还剩约 2 mL 左右时,加入 5 mL 水以 1 滴/s～2 滴/s 的流速

淋洗小柱，直至空气流经小柱，弃去流出液。去除针筒，加压抽干约 3 min 后加 3 mL 叔丁基甲基醚-正己烷溶液(4.11)以 1 滴/s～2 滴/s 的流速淋洗小柱，弃去流出液，加压抽干约 5 min 后加入 5 mL 甲醇以 1 滴/s 的流速洗脱于 10 mL 试管中。该洗脱液于 45 ℃下氮气吹干，加入 0.02 mol/L 乙酸铵溶液 1.0 mL 溶解残渣，充分涡旋混合后过 0.45 μm 水相滤膜(4.21)，供液相色谱-质谱/质谱仪测定。

7.1.3 基质标准溶液的制备

称取 5 份约 2 g 阴性试样(精确至 0.01 g)置于 50 mL 具塞塑料离心管中，分别加入标准品工作液(4.19)10 μL 、20 μL、50 μL、100 μL、200 μL，余下操作同 7.1.1～7.1.2。

7.2 测定

7.2.1 液相色谱条件

液相色谱条件如下：

a) 色谱柱：Protemix WCX-NP5 柱，2.1 mm×100 mm，5 μm 或相当者；

b) 流动相：5‰甲酸溶液(4.12)、0.02 mol/L 乙酸铵溶液(4.13)和甲醇，梯度洗脱；梯度参见附录 A 中表 A.1；

c) 流速：350 μL /min；

d) 进样量：25 μL；

e) 柱温：35 ℃。

7.2.2 质谱条件

质谱条件如下：

a) 离子化模式：电喷雾正离子模式(ESI+)；

b) 质谱扫描方式：多反应监测 (MRM)；

c) 其他参考质谱条件参见附录 B 中的表 B.1。

7.2.3 液相色谱-质谱/质谱测定

7.2.3.1 定性测定

在上述色谱-质谱条件下，如果样品中检测的色谱峰保留时间与相应的标准品相差在±2.5%以内，并且在扣除背景后所选择的两对离子对与浓度相当标准溶液的相对丰度一致，其相对丰度允许偏差不超过表 1 规定的范围，则可判定样品中存在对应的待测物。

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(%基峰)	>50%	>20%～50%	>10%～20%	≤10%
允许的相对误差	±20%	±25%	±30%	±50%

7.2.3.2 定量测定

采用基质提取标准曲线测定样液中的被测物的含量。样品中被测物量应在标准曲线范围之内，如果含量超出标准曲线范围，应适当稀释至标准曲线范围之内进行检测。标准溶液的多反应监测(MRM)色谱图参见附录 C 中的图 C.1。

7.3 空白试验

除不称取试样外，均按上述步骤进行。

8 结果计算和表述

采用外标法定量，用色谱数据处理机或按式(1)计算样品中待测物残留量，计算结果需扣除空白值：

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

式中：

X ——试样中待测物残留量，单位为纳克每克(ng/g)；

A ——样液中待测物的峰面积；

A_s ——标准工作溶液中待测物的峰面积；

c ——标准工作溶液中待测物的浓度，单位为纳克每毫升(ng/mL)；

V ——样液最终定容体积，单位为毫升(mL)；

m ——最终样液代表的试样量，单位为克(g)。

9 测定低限与回收率

9.1 测定低限

本方法的测定低限为 10 μg/kg。

9.2 回收率

本标准方法分别以松花粉、玉米花粉、茶花粉、葵花粉、油菜花粉、杂花粉为空白样品基质，进行 3 个浓度水平的添加回收试验，每个浓度水平进行 6 次重复实验，测得各种基质链霉素和双氢链霉素的回收率范围参见附录 D。

附 录 A
（资料性附录）
液相色谱梯度洗脱程序

液相色谱梯度洗脱程序见表 A.1。

表 A.1 液相色谱梯度洗脱程序

时间/min	流速/(μL/min)	甲醇/%	0.02 mol/L 乙酸铵/%	5‰甲酸/%
0	350	20	80	0
1.00	350	20	80	0
2.00	350	100	0	0
2.50	350	100	0	0
2.60	350	20	0	80
3.50	350	20	0	80
3.60	350	20	80	0
5.00	350	20	80	0

附 录 B
（资料性附录）
质谱仪器参考条件[1)]

质谱条件如下：

a） 电离方式：ESI+；

b） 毛细管电压：2.5 kV；

c） 毛细管温度：350 ℃；

d） 加热源温度：500 ℃；

e） 气帘气：氮气，0.06 MPa；

f） 辅助气：氮气，5.0 L/h；

g） 碰撞气：氩气，0.2 Pa（1.5 mTor）；

h） 定性离子对、定量离子对、碰撞能量见表 B.1。

表 B.1 多反应监测条件

序号	测定物质	母离子（m/z）	子离子（m/z）	碰撞能量/eV
1	链霉素	614.3	263.2[a]	32
			582.3	21
2	双氢链霉素	584.3	263.3[a]	29
			246.1	32
[a] 为定量离子对。				

1） 非商业性声明：附录 B 所列参考质谱条件是在 Thermo Vantage 液质联用仪上完成的，此处列出试验用仪器型号仅为提供参考，并不涉及商业目的，鼓励标准使用者尝试不同厂家或型号的仪器。

附 录 C
（资料性附录）
标准溶液多反应监测(MRM)离子色谱图

链霉素和双氢链霉素多反应监测(MRM)离子色谱图见图 C.1。

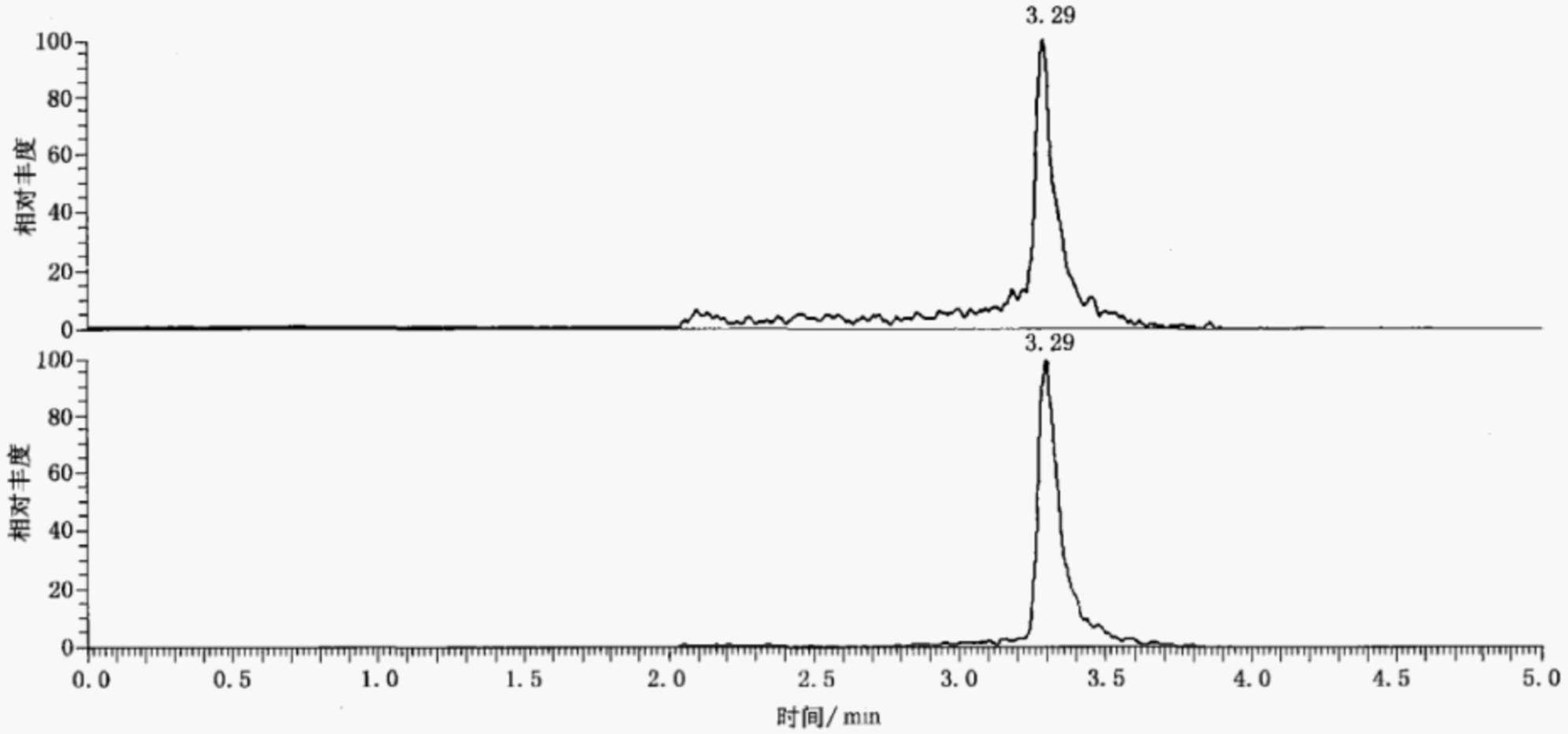

图 C.1 链霉素、双氢链霉素基质标准溶液的多反应监测(MRM)离子色谱图

附 录 D
（资料性附录）
回收率

回收率见表 D.1。

表 D.1 链霉素(STR)和双氢链霉素(DHS)实验回收率结果

基质	添加浓度 μg/kg	回收率范围 %	
		STR	DHS
松花粉	10	82.5～95.2	73.3～90.5
	20	87.5～96.5	82.5～106.0
	50	80.6～100.2	81.8～105.6
玉米花粉	10	76.4～93.7	79.6～95.6
	20	77.0～95.0	74.0～96.0
	50	83.2～102.0	85.4～101.8
茶花粉	10	77.5～99.4	74.1～90.9
	20	71.0～97.0	74.0～97.5
	50	95.4～104.8	87.2～106.4
葵花粉	10	70.5～84.4	72.0～90.2
	20	70.0～89.5	69.5～89.0
	50	77.6～98.6	71.8～89.8
油菜花粉	10	65.8～80.2	67.4～88.2
	20	67.5～86.0	67.5～89.5
	50	74.4～89.0	71.6～90.0
杂花粉	10	82.4～112.0	89.2～110.5
	20	80.0～107.5	92.0～107.5
	50	91.8～110.2	93.2～105.4

Foreword

This standard was drafted on the basis of the requirement of GB/T 1.1—2009.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. The document issuing organization shall not be held responsible for identifying any or all such patent rights.

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

Drafting Units of this standard: Jiangsu Import and Export Commodity Inspection Bureaus of the People's Republic of China.

The main drafters of this standard: Huang Juan, Liu Yah, Liu Han, Li Jingjing, Jiang Shah, Chen Huilan, Yin Yao, Zhang Xiaoyan, Ding Tao.

Determination of streptomycin and dihydrostreptomycin in pollen for export—LC-MS/MS method

1 Scope

The standard specifies the methods for determination of streptomycin and dihydrostreptomycin in pollen for export—LC/MS/MS method.

The standard is applicable to the determination of streptomycin and dihydrostreptomycin in pollen pini,corn pollen, camellia pollen, sunflower pollen, rape pollen, bee pollen and so on for export.

2 Quoted normative documents

The articles of the following documents have become the articles of this standard through the quotation. For the dated quoted documents, none of the revised contents (which does not include the content for correcting errors) or the revised editions after this standard is applicable to this standard. However, everybody who comes to an agreement through this standard is encouraged to study whether or not the latest editions of these documents can be used in this standard. For the quoted documents without date, their lasted editions are applicable to this standard.

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

3 Principle

The streptomycin and dihydrostreptomycin was extracted with extraction solution, deproteinized by chloroform, then cleaned up by a C18 solid phase extraction cartridge. The analytes were determined by high-performance liquid chromatography-tandem mass spectrometry. External standards were used to quantify.

4 Reagents and materials

Unless otherwise specified, all the reagent used should be analytical grade, "water" is the first grade water prescribed by GB/T 6682.

4.1 Methanol: HPLC grade.

4.2 Phosphate acid.

4.3 Tert-butyl methyl ether.

4.4 Formic acid: HPLC grade.

4.5 n-hexane.

4.6 Chloroform.

4.7 Ammonium acetate: $CH_3CO_2NH_4$, HPLC grade.

4.8 1-Heptane sulphonic acid sodium: $C_7H_{15}SO_3Na$, HPLC grade.

4.9 Sodium phosphate: $Na_3PO_4 \cdot (H_2O)_{12}$.

4.10 Extraction solution: dissolve 10.1 g of 1-Heptane sulphonic acid sodium (4.8), 4.1 g of sodium phosphate in 1 000 mL water, and then add 5 mL phosphate acid (4.2), mix homogeneously.

4.11 Tert-butyl methyl ether-n-hexane solution (4 + 1, *V*/*V*): add 400 mL tert-butyl methyl ether (4.3) into 100 mL n-hexane (4.5), mix adequately.

4.12 Formic acid solution (5‰, *V*/*V*): add 5 mL formic acid (4.4) into 1 000 mL water, mix adequately.

4.13 0.02 mol/L Ammonium acetate solution: dissolve 1.54 g of ammonium acetate (4.7) in 1 000 mL water, mix homogeneously.

4.14 Methanol solution (3 + 7, *V*/*V*): add 300 mL methanol into 700 mL water, mix adequately.

4.15 Standard of Streptomycin Sulfate: $(C_{21}H_{39}N_7O_{12})_2 \cdot (H_2SO_4)_3$, CAS No: 3810-74-0, purity ≥98%.

4.16 Standard of Dihydrostreptomycin Sesquisulfate Hydrate: $(C_{21}H_{41}N_7O_{12})_2 \cdot (H_2SO_4)_3$, GAS No: 5490-27-7, purity ≥99%.

4.17 Standard stock solution: accurately weigh appropriate standard(4.15, 4.16), dissolved with methanol solution (4.14), the concentration of solution is 1 mg/mL, the standard solution should be stored at 0 ℃ to 4 ℃.

4.18 Standard intermediate solution: Transfer an appropriate amount of standard stock solution (4.17), and diluted with methanol solution (4.14). The concentration of intermediate standard solution is 10 μg/mL, stored at 0 ℃ to 4 ℃.

4.19 Working standard solution: Transfer an appropriate amount of standard intermediate solution

(4.18), and diluted with methanol solution (4.14). The concentration of solution is 1.0 μg/mL, prepared when in use.

4.20 C_{18} SPE: 500 mg, 3 mL, or equivalent.

4.21 Membrane: 0.45 μm.

5 Apparatus and equipment

5.1 LC/ESI-MS/MS (Liquid chromatography coupled with electrospray ionization triple quadrupole mass spectrometry).

5.2 Analytical balance(0.01 g and 0.01 mg).

5.3 High speed centrifuge: 8 000 r/min.

5.4 Ultrasonic oscillator.

5.5 Vortex mixer.

5.6 N_2-evaporatou.

5.7 Plastic centrifuge tube with cap: 50 mL.

5.8 SPE Vacuum Manifolds.

6 Sample preparation and storage

About 500 g representative samples should be taken from all samples, grinded and blended by tissue blender to produce homogeneous samples by passing through 20 mesh sieve. Put in suitable clean container. After being sealed and labeled, the samples should be stored at normal temperature.

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

7 Procedure

7.1 Prepration

7.1.1 Extraction

Weigh 2 g (accurtate to 0.01 g) sample in a 50 mL plastic centrifuge tube, add 20 mL extraction solu-

tion (4.10) and 3 mL Chloroform. Mix for 3 minutes on vortex mixer, then extract for 15 minutes on ultrasonic Oscillator, centrifuge sample at 8 000 r/min for 5 minutes. Remove the upper solution to a clean container.

7.1.2 Cleanup by SPE

Draw the above solution through C_{18} SPE cartridge (activated with 3 mL methanol and 3 mL water) at speed of 1－2 drop per minute until 2 mL solution left. Wash column with 5 mL water at the same speed, then elute column with 5 mL methanol after drying it about 5 minutes. The elute solution was dried by nitrogen gas at 45 ℃. The residue is dissolved by 1 mL 0.02 mol/L ammonium acetate solution, filtered by membrane of 0.45 μm (4.21) and determined by high-performance liquid chromatography-tandem mass spectrometry.

7.1.3 Matrix working standard solution

Weigh 5 of 2 g (accurtate to 0.01 g) blank sample in a 50 mL volumetric flask, add 10 μL, 20 μL, 50 μL, 100 μL, 200 μL working standard solution respectively. The following operation is as same as 7.1.1—7.1.2.

7.2 Determination

7.2.1 HPLC conditions

a) Column: Protemix WCX-NP5, 2.1 mm × 100 mm, 5 μm, or equivalent;

b) Mobile phase: 5‰ formic acid solution (4.12)、0.02 mol/L ammonium acetate solution(4.13) and methanol, gradient elution; Refer to Table A.1 in Annex A;

c) Flow rate: 350 μL/min;

d) Injector volume: 25 μL;

e) Column temperature: 35 ℃。

7.2.2 MS/MS condition

a) Ion model: Electrospray positive ion mode ESI(＋);

b) Monitor model: Multiple reaction monitoring (MRM);

c) The other conditions were listed as Table B.1 in Annex B.

7.2.3 LC-ESI-MS/MS determination

7.2.3.1 Qualitative determation

Under HPLC-LC/MS/MS conditions, If the rention time of sample chromatogram peaks are consistent with that of standard solution within ±2.5%. and the relative intensities of sample transitions shall correspond to those of standard solution transitions for confirmation, the permitted tolerance is within the tolerances listed in table 1, then the corresponding analyte must be present in sample.

Table 1—Maximum permitted tolerance for relative ion intensities while confirmation

Ralative intensity	>50%	>20%—50%	>10%—20%	≤10%
Permitted tolerance	±20%	±25%	±30%	±50%

7.2.3.2 Determination of quantitative

Under the optimized instrument working condition, different matrix extract solutions are injected into the instrument separately. Using peak area as y-axis, and concentration as x-axis, the concentration of analyte in the standard working solution and sample solution should be in the linear range of the instrumental detection, if not, the sample solution should be diluted before injected. Muti-response monitoring (MRM) chromatogram of standard working solution is shown in Figure C.1 in appendix C.

7.3 Blank test

Blank sample is treated according to the above procedure.

8 Calculation and expression of result

External standard is used for quantify, calculation the content of analyte in the test sample by LC-MS/MS ChemStations or according to the formula (1). The blank value should be subtracted from the above result of calculation:

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

where:

X —the analyte content in the test sample, ng/g;

A —the peak area of analyte in sample solution;

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

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

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

m —mass of test sample of final sample solution, g.

9 Limit of quantification and recovery

9.1 Limit of quantification

10 μg/kg.

9.2 Recovery

Pollen pini, corn pollen, camellia pollen, sunflower pollen, rape pollen, bee pollen are prepared for the recovery test by adding three concentrations level which containing quantification limit. Each fortifying concentration test repeatly 6 times, and the recovery range is listed in Annex D.

Annex A
(informative annex)
Liquid chromatography gradient elution conditions

Gradient elution program of HPLC is listed in Table A.1.

Table A.1—Gradient elution program of HPLC

Time/min	Flow rate/(μL/min)	Methanol/%	0.02 mol/L ammonium acetate/%	5‰ formic acid solution/%
0	350	20	80	0
1.00	350	20	80	0
2.00	350	100	0	0
2.50	350	100	0	0
2.60	350	20	0	80
3.50	350	20	0	80
3.60	350	20	80	0
5.00	350	20	80	0

Annex B
(informative annex)
MS/MS condition[1)]

Referenced conditions are as follows:

a) Ionization mode: ESI+;

b) Ion spray voltage: 2.5 kV;

c) Capillary Temperature: 350 ℃;

d) Vaporizer Temperature: 500 ℃;

e) Curtain gas: Nitrogen, 0.06 MPa;

f) Auxiliary gas: Nitrogen, 5.0 L/h;

g) Collision Pressure: Argon, 0.2 Pa (1.5 mTor);

h) Qualitative ion pairs, quantitative ion pairs, collision energies are listed in Table B.1.

Table B.1—MS/MS condition

Serial	Compound	Parent ion(m/z)	Daughter ions(m/z)	Collision energies/eV
1	STR	614.3	263.2[a]	32
			582.3	21
2	DHS	584.3	263.3[a]	29
			246.1	32
[a] is Quantitative ion pair.				

1) Non-commercial statement: Parameters listed in annex A are accomplished by Thermo Vantage LC-MS/MS. The equipment and its type involved in the standard method is only for reference and not related to commercial aims, and the analysts are encouraged to use equipments of different corporation or different type.

Annex C
(informative annex)
MRM chromatogram of standard working solution

MRM chromatogram of the STR and DHS are shown in Figure C.1.

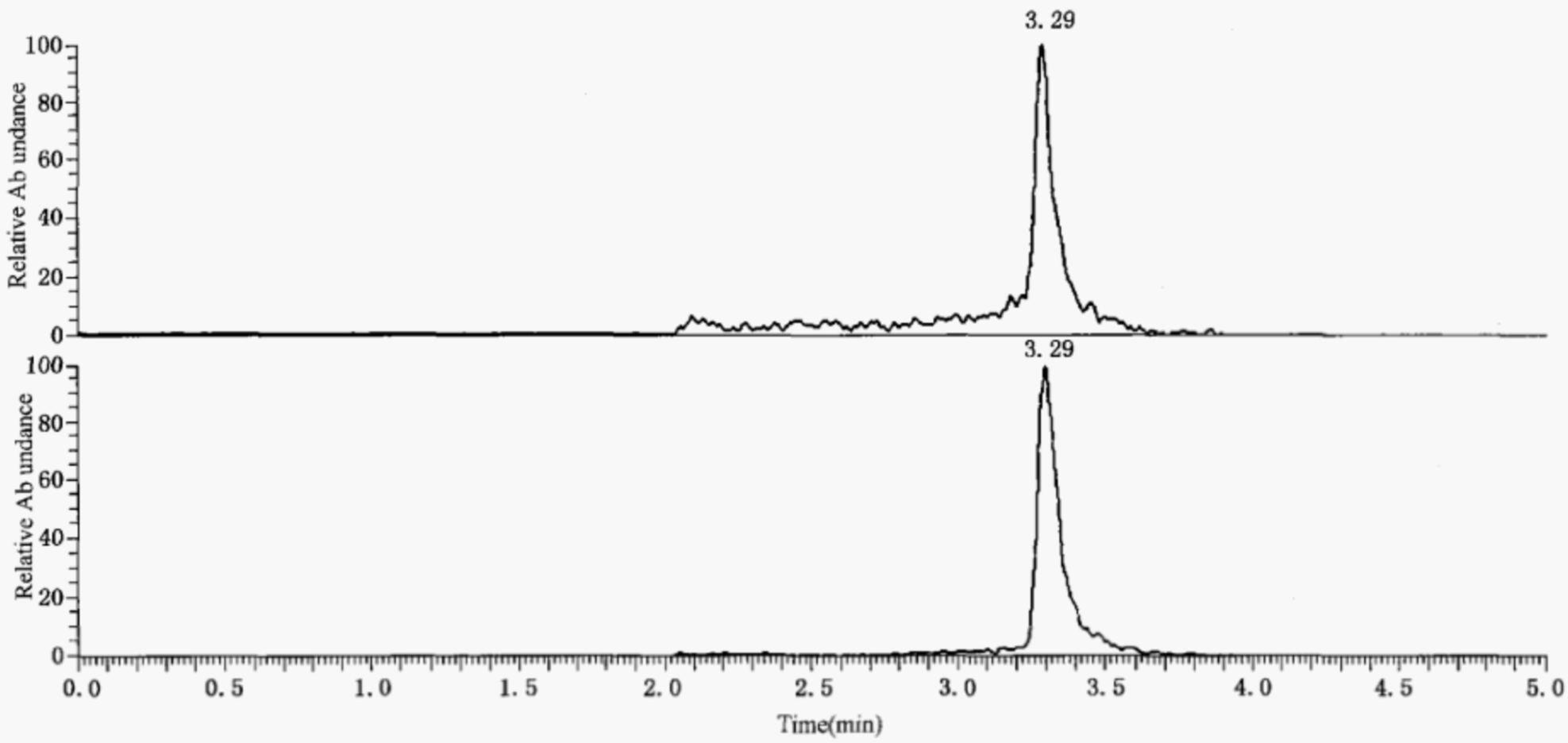

图 C.1—MRM chromatogram of STR and DHS

Annex D
(informative annex)
Recovery results

Recovery results are listed in Table D.1.

Table D.1—Recovery results

Matrix	Spiked level μg/kg	Recovery range %	
		STR	DHS
pollen pini	10	82.5—95.2	73.3—90.5
	20	87.5—96.5	82.5—106.0
	50	80.6—100.2	81.8—105.6
corn pollen	10	76.4—93.7	79.6—95.6
	20	77.0—95.0	74.0—96.0
	50	83.2—102.0	85.4—101.8
camellia pollen	10	77.5—99.4	74.1—90.9
	20	71.0—97.0	74.0—97.5
	50	95.4—104.8	87.2—106.4
sunflower pollen	10	70.5—84.4	72.0—90.2
	20	70.0—89.5	69.5—89.0
	50	77.6—98.6	71.8—89.8
rape pollen	10	65.8—80.2	67.4—88.2
	20	67.5—86.0	67.5—89.5
	50	74.4—89.0	71.6—90.0
bee pollen	10	82.4—112.0	89.2—110.5
	20	80.0—107.5	92.0—107.5
	50	91.8—110.2	93.2—105.4

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