SN

中华人民共和国出入境检验检疫行业标准

SN/T 2114—2008

进出口水果和蔬菜中阿维菌素残留量检测方法　液相色谱法

Determination of abamectin residues in fruit and vegetable for import and export—LC method

2008-09-04 发布　　2009-03-16 实施

中华人民共和国
国家质量监督检验检疫总局　发布

前　言

本标准的附录A、附录B为资料性附录。

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山东出入境检验检疫局负责起草。

本标准起草人：李立、李金强、李佩暖。

本标准系首次发布的出入境检验检疫行业标准。

进出口水果和蔬菜中阿维菌素残留量检测方法　液相色谱法

1　范围

本标准规定了水果及蔬菜中阿维菌素检测的制样和液相色谱检测方法。

本标准适用于苹果及菠菜中阿维菌素残留量的检测。其他水果和蔬菜可以参照本标准。

2　试样制备与保存

2.1　试样制备

将所取样品缩分出 1 kg，取可食部分，经组织捣碎机捣碎，均分为两份，装入洁净容器内，作为试样密封并标明标记。

2.2　试样保存

将试样于－18 ℃以下保存。

在抽样和制样的操作过程中，应防止样品受到污染或发生残留物含量的变化。

3　方法提要

试样中的阿维菌素用丙酮提取，经浓缩后，用 SPE C_{18} 柱净化，并用甲醇洗脱。洗脱液经浓缩、定容、过滤后，用配有紫外检测器的高效液相色谱测定，外标法定量。

4　试剂和材料

除另有规定外，所有试剂均为分析纯，水为去离子水。

4.1　丙酮。

4.2　甲醇：色谱纯。

4.3　阿维菌素标准品：纯度≥96.0%。

4.4　阿维菌素标准储备液：称取 0.1 g(准确至 0.000 2 g)阿维菌素标准品于 100 mL 容量瓶中，用甲醇溶解并定容至刻度。配制成浓度为 1.0 mg/mL 的标准储备液。

4.5　阿维菌素标准工作液：根据需要移取适量的阿维菌素标准储备液，用甲醇稀释成适当浓度的标准工作液。标准工作液需每周配制一次。

5　仪器和设备

5.1　高效液相色谱仪：配有紫外检测器。

5.2　组织捣碎机。

5.3　振荡器。

5.4　旋转蒸发器。

5.5　固相萃取柱：SPE C_{18}　规格：60 mg/3 mL；使用前用 5 mL 甲醇和 5 mL 水活化。

6　测定步骤

6.1　提取

称取试样约 20 g(精确至 0.1 g)于 100 mL 具塞锥形瓶中，加入 50 mL 丙酮，于振荡器上振荡 0.5 h

用布氏漏斗抽滤，用 20 mL×2 丙酮洗涤锥形瓶及残渣。合并丙酮提取液，于 40 ℃水浴旋转蒸发至约 2 mL。

6.2 净化

将上述的浓缩提取液完全转入 SPE C_{18} 柱，再用 5mL 水淋洗，去掉淋洗液。最后用 5 mL 甲醇洗脱，收集洗脱液，用氮气吹至近干。准确加入 1.0 mL 甲醇溶解残渣，用 0.45 μm 滤膜过滤，滤液供液相色谱测定。外标法定量。

6.3 测定

6.3.1 高效液相色谱条件：

a) 色谱柱：ODS-C_{18} 反相柱，4.6 mm×125 mm；

b) 流动相：甲醇：水(90+10)；

c) 流速：1.0 mL/min；

d) 检测波长：245 nm；

e) 柱温：40 ℃；

f) 进样量：20 μL。

6.3.2 色谱测定

根据样液中阿维菌素含量情况，选定峰高相近的标准工作液，标准工作液和样液中阿维菌素响应值均应在仪器检测线性范围内，标准工作液和样液等体积参插进样。在上述色谱条件下，阿维菌素保留时间约为 5.3 min。

标准色谱图参见附录 A，标准品紫外光谱图参见附录 B。

6.4 空白试验

除不加试样外，均按照上述测定步骤进行。

7 结果计算与表述

用色谱数据处理机，或按式(1)计算试样中阿维菌素残留量，计算结果需扣除空白值：

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

式中：

X——试样中阿维菌素残留量，单位为毫克每千克(mg/kg)；

h——样液中阿维菌素峰高，单位为毫米(mm)；

h_s——标准工作液中阿维菌素峰高，单位为毫米(mm)；

c——标准工作液中阿维菌素浓度，单位为毫克每升(mg/L)；

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)。

8 测定低限和回收率

8.1 测定低限

本方法的测定低限为 0.01 mg/kg。

8.2 回收率

苹果样品中添加阿维菌素的浓度和回收率的实验数据：

——在 0.01 mg/kg 时，回收率为 82.5%；

——在 0.05 mg/kg 时，回收率为 87.5%；

——在0.50 mg/kg时，回收率为95.0%。

菠菜中添加阿维菌素的浓度和回收率的实验数据：

——在0.01 mg/kg时，回收率为83.0%；

——在0.05 mg/kg时，回收率为89.0%；

——在0.50 mg/kg时，回收率为97.0%。

附 录 A
（资料性附录）
阿维菌素标准品液相色谱图

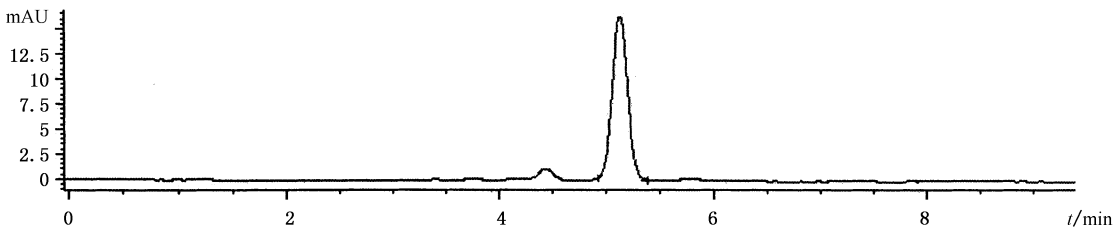


图 A.1 阿维菌素标准品液相色谱图

附 录 B
（资料性附录）
阿维菌素标准品紫外吸收光谱图

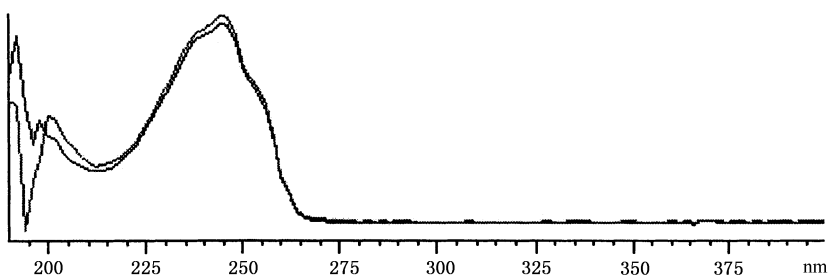


图 B.1 阿维菌素标准紫外吸收光谱图

Foreword

Annex A，Annex B of this standard are informative annexes.

This standard was proposed by and is under the charge of the Certification and Accreditation of the people's Republic of China. This standard was drafted by Shandong Entey-Exit Inspection and Quarantine Bureau of the people's Republic of China.

The main drafters of this standard are LiLi，Li jinQiang，Li peinuan.

This standard is a professional standard for inspection and quarantine promulgated for the first time.

Determination of abamectin residues in fruit and vegetable for import and export—LC method

1 Scope

This standard specifies the methods of sample preparation and determination of abamectin residues by high Performance liquid chromatography in fruit and vegetable.

This standard is applicable to determination of abamectin residuce in apple and spinach.

2 Sample preparation and storage

2.1 Preparation of test sample

The combined primary sanple is reduced to 1 kg, the edible portions are blended in a blender, and then divided into two equal portions. Each portion is placed in a clean container as the test sample, which is then sealed and labeled.

2.2 Storage of test sample

The test sample should be stored below −18 ℃.

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

3 Principle

The abamectin residues in the test sample are extracted by acetone, and then concentrated and cleaned up by solid phase extraction C_{18} cartridge. Elute with methanol, and the eluate is concentrated, made up to volume, and filtered. Determination is made by high performance liquid chromatography equipped with UV detector, using external standard method.

4 Reagents and materials

Unless otherwise specified, the reagents should be analytically pure, "water" is distilled water.

4.1 Acetone.

4.2 Methanol: HPLC grade.

4.3 Abamectin standard: Purity≥96.0%.

4.4 Abamectin standard stock solution: Weigh 0.1 g (accurate to 0.000 2 g) of abamectin standard into a 100 mL volumetric flask, dissolve with methanol, mix throughly. The concentration of the standard stock solution is 1.0 mg/mL.

4.5 Abamectin standard working solution: According to the requirement prepare standard working solution of appropriate concentration by diluting the standard stock solution with methanol. Prepare the standard working solution fleshly every week.

5 Apparatus and equipment

5.1 High performance liquid chromatograph, equipped with UV detector.

5.2 High-speed blender.

5.3 Shaker.

5.4 Rotary evaporator.

5.5 Cleanup column: SPE C_{18} cartridge, 60 mg/3 mL activated with 5 mL of methanol and rinsed with 5 mL of water.

6 Procedure

6.1 Extraction

Weight ca 20 g (accurate to 0.1 g) into a 100 mL conical flask with stopper, add 50 mL of acetone, shake for 0.5 h with shaker, the extract is filtered by suction through a buchner funnel, wash the residue and the flask with 2×20 mL acetone, combine acetone and evaporated to ca 2 mL with rotary evaporator under 40 ℃.

6.2 Cleanup

Transfer the concentrated extract into a SPE C_{18} cartridge, the effluent is discarded and rinsing with 5 mL of water. Then elute with 5 mL of methanol, collect the eluate and nearly to dryness with a stream of nitrogen. Make up to 1.0 mL with methanol. Filter the solution with the filter membrane of

0.45 μm. The filtrate is used for HPLC determination.

6.3 Determination

6.3.1 HPLC operating condition:

a) HPLC column: ODS C_{18}, 4.6 mm × 125 mm;

b) Mobile phase: Methanol : water (90 + 10);

c) Flow rate: 1.0 mL/min;

d) Wavelength: 245 nm;

e) Column temperature: 40 ℃;

f) Injection volume: 20 μL.

6.3.2 HPLC determination

According to the approximate concentration of abamectin in the sample solution, select the standard working solution with similar peak height to that of the sample solution . The responses of abamectin in the standard working solution and sample solution should be in the linear range of the instrumental detection. The standard solution should be injected in-between the injections of the sample solution of equal volume. Under the above operating condition, the retention time of abamectin is 5.3 min.

For HPLC chromatogram of the standard, see figure in annex A and for photodiode array spectrogram of the standard, see figue in annex B.

6.4 Blank test

The operation of the blank test is the same as that described in the method of determination, but without addition of sample.

7 Calculation and expression of the result

Calculate the content of abamectin residues in the test sample by date processor or according to the formula (1), the blank volue shoule be subtracted from the above result of calculation.

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

where:

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

h—the peak height of abamectin in the sample solution, mm;

h_s—the peak height of abamectin in the standard solution, mm;

c—the concentration of abametion in the standard working solution, μg/mL;

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

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

8 Limit of determination and recovery

8.1 Limit of determination

The limit of determination of this method is 0.01 mg/kg.

8.2 Recovery

According to the experimental data, the fortifying concentrations of abamectin in apple and its corresponding recoveries are:

—0.01 mg/kg, the recovery 82.5%;

—0.05 mg/kg, the recovery 87.5%;

—0.50 mg/kg, the recovery 95.0%.

The fortifying concentrations of abamectin in spinach and its corresponding recoveries are:

—0.01 mg/kg, the recovery 83.0%;

—0.05 mg/kg, the recovery 89.0%;

—0.50 mg/kg, the recovery 97.0%.

Annex A
(informative)
Chromatogram of the standard

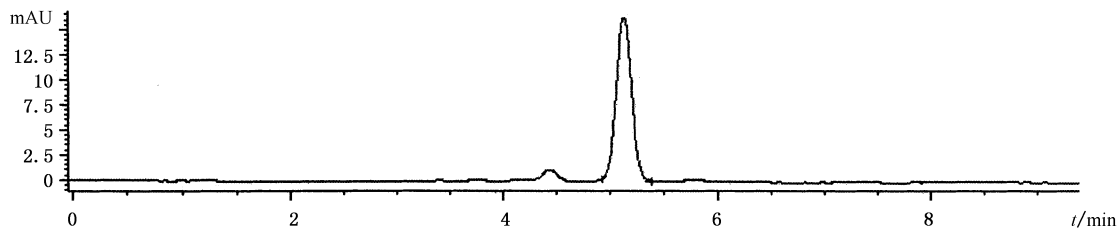


Figure A. 1—Liquid chromatogram of abamectin standard

Annex B
(informative)
spectrogram of the standard

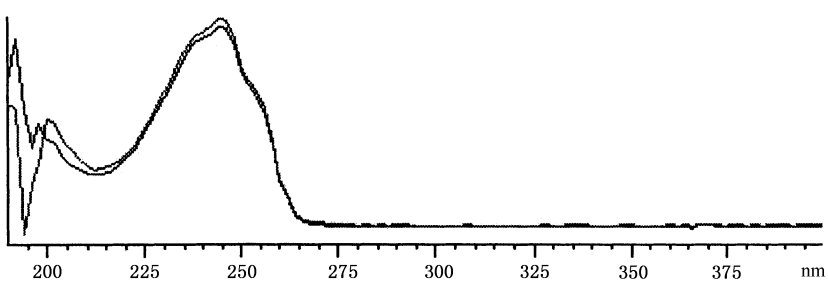


Figure B. 1—Spectrogram of abamectin standard