SN

中华人民共和国进出口商品检验行业标准

SN 0346—95

出口蔬菜中α-萘乙酸残留量检验方法

Method for the determination of α-naphthylacetic acid residues in vegetables for export

1995-05-29发布　　1995-11-01实施

中华人民共和国国家进出口商品检验局　发布

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1 主题内容与适用范围

本标准规定了出口蔬菜中α-萘乙酸残留量检验的抽样、制样和气相色谱测定方法。

本标准适用于出口速冻荷兰豆中α-萘乙酸残留量的检验。

2 抽样和制样

2.1 检验批

以不超过1 500件为一检验批。

同一检验批的商品应具有相同的特征，如包装、标记、产地、规格和等级等。

2.2 抽样数量

批量，件	最低抽样数，件
1～25	1
26～100	5
101～250	10
251～1 500	15

2.3 抽样方法

按2.2规定的抽样件数随机抽取，逐件开启，每件至少取500 g作为原始样品，原始样品总量不得少于2 kg。加封后，标明标记，及时送实验室。

2.4 试样制备

将所取原始样品缩分出1 kg，取可食部分，经组织捣碎，均分成两份，装入洁净容器内，作为试样，密封，并标明标记。

2.5 试样保存

将试样于－18℃以下冷冻保存。

注：在抽样和制样的操作过程中，必须防止样品受到污染或发生残留物含量的变化。

3 测定方法

3.1 方法提要

试样酸化后，以乙醚-石油醚提取，浓缩定容后用配有氢火焰离子化检测器的气相色谱仪测定，外标法定量。

3.2 试剂

所有试剂均为分析纯，不得含有干扰物质。

中华人民共和国国家进出口商品检验局1995-05-29批准　　1995-11-01实施

3.2.1 乙醚。

3.2.2 石油醚(蒸馏范围 30～60℃)。

3.2.3 乙醚-石油醚(4+1)。

3.2.4 无水硫酸钠:650℃灼烧 4 h,冷却后贮于密闭容器中。

3.2.5 盐酸。

3.2.6 盐酸溶液:1 mol/L。

3.2.7 α-萘乙酸标准品:纯度≥99.0%。

3.2.8 α-萘乙酸标准溶液:准确称取适量的α-萘乙酸标准品,用乙醚-石油醚(4+1)配成浓度为 0.10 mg/mL的标准储备液,根据需要再配成适当浓度的标准工作液。

3.3 仪器和设备

3.3.1 气相色谱仪并配有氢火焰离子化检测器。

3.3.2 高速组织捣碎机。

3.3.3 旋涡混匀器。

3.3.4 离心机:0～5 000 r/min。

3.3.5 多功能微量化样品处理仪或相当仪器。

3.3.6 离心管:50 mL。

3.3.7 K-D 浓缩瓶:20 mL(具 1 mL 尾管)。

3.3.8 微量注射器:10 μL。

3.4 测定步骤

3.4.1 提取

称取 15 g 试样(精确至 0.01 g)于 50 mL 离心管中,加入 2 mL 盐酸溶液(1 mol/L),混匀,加入 0.5 g无水硫酸钠,加入 10 mL 乙醚-石油醚(4+1),在旋涡混匀器上混匀 1 min,以 3 000 r/min 离心 2 min,醚层移至 K-D 浓缩瓶中。残渣再用 5 mL 乙醚-石油醚(4+1)以同样的步骤提取一次。合并有机相。置多功能微量化样品处理仪上,于 40℃通空气挥发,浓缩定容至 0.20 mL,供气相色谱测定。

3.4.2 测定

3.4.2.1 色谱条件

a. 毛细管色谱柱:HP-1(dimethylpolysiloxane Gum)10 m×0.53 mm(内径)×2.65 μm 熔融石英制或相当的毛细管柱;

b. 柱温:165℃;

c. 进样口温度:230℃;

d. 检测器温度:250℃;

e. 载气、尾吹气:氮气(纯度≥99.99%),柱流速 8 mL/min,尾吹气流速 30 mL/min;

f. 氢气:40 mL/min;

g. 空气:400 mL/min。

3.4.2.2 色谱测定

根据样液中 α-萘乙酸含量情况,选择峰高相近的标准工作溶液。标准工作溶液和样液中 α-萘乙酸的响应值应在仪器检测线性范围内。对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,α-萘乙酸保留时间约为 2.4 min。

3.4.3 空白试验

除不加试样外,按上述测定步骤进行。

3.4.4 结果计算和表述

用色谱数据处理机或按下列公式计算试样中 α-萘乙酸含量:

$$X = \frac{h \cdot c \cdot V}{h_s \cdot m}$$

式中：X——试样中 α-萘乙酸残留量，mg/kg；

h——样液中 α-萘乙酸的峰高，mm；

h_s——标准工作溶液中 α-萘乙酸的峰高，mm；

c——标准工作液中 α-萘乙酸的浓度，μg/mL；

V——样液最终定容体积，mL；

m——称取的试样量，g。

注：计算结果需扣除空白值。

4 方法的测定低限、回收率

4.1 本方法的测定低限为 0.02 mg/kg。

4.2 回收率

回收率的实验数据：α-萘乙酸添加浓度在(0.02～5.0)mg/kg 范围内，回收率为 89.5%～103.2%。

附加说明：

本标准由中华人民共和国国家进出口商品检验局提出。

本标准由中华人民共和国厦门进出口商品检验局负责起草。

本标准主要起草人周昱、庄宿燕。

Professional Standard of the People's Republic of China for Import and Export Commodity Inspection

SN 0346—95

Method for the determination of α-naphthylacetic acid residues in vegetables for export

1 Scope and field of application

This standard specifies the methods of sampling, sample preparation and determination by gas chromatography (GC) of α-naphthylacetic acid residues in vegetables for export.

This standard is applicable to the determination of α-naphthylacetic acid residues in quick frozen Chinese pea pods for export.

2 Sampling and sample preparation

2.1 Inspection lot

The quantity of an inspection lot should not be more than 1 500 packages.

The characteristics of the cargo within the same inspection lot, such as packing, mark, origin, grade and specification, should be the same.

2.2 Quantity of sample taken

Number of package in each inspection lot	Minimum number of packages to be taken
1—25	1
26—100	5
101—250	10
251—1 500	15

2.3 Sampling procedure

A number of packages specified in 2.2 are taken at random and opened one by one. The sample weight taken as the primary sample from each package should be at least 500 grams. The total weight of all primary samples should not be less than 2 kg, which shall be sealed, labeled and sent to laboratory in time.

2.4 Preparation of test sample

The combined primary sample is reduced to 1 kg, the edible portions are blended, 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.

2.5 Storage of test sample

The test samples should be stored below −18℃.

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

Approved by the State Administration of Import and Export Commodity Inspection of the People's Republic of China on May. 29, 1995

Implemented from Nov. 01, 1995

3 Method of determination

3.1 Principle

After being acidified, the sample is extracted by diethyl ether-petroleum ether, then the extract is concentrated to a definite volume. Finally, the solution is analyzed by GC with flame ionization detector, using external standard method.

3.2 Reagents

All reagents should be of analytical grade and the presence of any interfering materials is not permissible.

3.2.1 Diethyl ether.

3.2.2 Petroleum ether(distillation range 30～60℃).

3.2.3 Diethyl ether-petroleum ether (4+1).

3.2.4 Anhydrous sodium sulfate: Ignite at 650℃ for 4 h, keep in a tightly closed container after cooling.

3.2.5 Hydrochloric acid.

3.2.6 Hydrochloric acid solution: 1 mol/L.

3.2.7 α-Naphthylacetic acid standard: Purity≥99.0%.

3.2.8 α-Naphthylacetic acid standard solution: Accurately weigh an adequate amount of α-naphthylacetic acid standard, dissolve in diethyl ether-petrolum ether (4 + 1) and prepare a solution of 0.10 mg/mL as the standard stock solution. According to the requirement, prepare a standard working solution of appropriate concentration.

3.3 Apparatus and equipment

3.3.1 Gas chromatograph, equipped with flame ionization detector.

3.3.2 High speed blender.

3.3.3 Vortex mixer.

3.3.4 Centrifuge: 0～5 000 r/min.

3.3.5 Multifunction sample treatment unit for microchemical method or equivalent.

3.3.6 Centrifuge tube: 50 mL.

3.3.7 K-D concentrating flask: 20 mL (with 1 mL end tube).

3.3.8 Micro syringe: 10 μL.

3.4 Procedure

3.4.1 Extraction

Weigh 15 g of the test sample(accurate to 0.01 g)into a 50 mL centrifuge tube, add 2 mL of 1 mol/L hydrochloric acid solution, mix well, add 0.5 g of anhydrous sodium sulfate, and 10 mL of diethyl ether-petroleum ether (4+1), mix intensely by a vortex mixer for 1 min, centrifuge for 2 min at 3 000 r/min, transfer the ether layer to a 20 mL K-D concentrating flask. Repeat the extraction in the same way with 5 mL of diethyl ether-petroleum ether (4+1), and then evaporate the combined organic phase on a multifunction sample treatment unit for microchemical method with air stream at 40℃, dilute exactly to 0.20 mL, the solution is used for gas chromatographic determination.

3.4.2 Determination

3.4.2.1 GC operating conditions.

a. Capillary column: HP-1 (dimethylpolysiloxane, Gum) 10 m×0.53 mm(id)×2.65 μm fused silica capillary column or equivalent;

b. Column temperature: 165℃;

c. Injection port temperature: 230℃;

d. Detector temperature: 250℃;

e. Carrier gas and make-up gas: Nitrogen, purity≥99.99%, flow rate: 8 mL/min, flow rate of make-up gas: 30 mL/min;

f. Hydrogen: 40 mL/min;

g. Air: 400 mL/min.

3.4.2.2 GC determination

According to the approximate concentration of α-naphthylacetic acid in the sample solution, select the standard working solution with similar peak height to that of sample solution. The responses of α-naphthylacetic acid 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 the sample solution of equal volume. Under the above chromatographic condition, the retention time of α-naphthylacetic acid is about 2.4 min.

3.4.3 Blank test

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

3.4.4 Calculation and expression of result

The calculation of α-naphthylacetic acid content in the test sample is carried out by GC data processor or according to the following formula:

$$X = \frac{h \cdot c \cdot V}{h_s \cdot m}$$

where

X——Residue content of α-naphthylacetic acid in test sample, mg/kg;

h——Peak height of α-naphthylacetic acid in the sample solution, mm;

h_s——Peak height of α-naphthylacetic acid in the standard working solution, mm;

c——Concentration of α-naphthylacetic acid in the standard working solution, μg/mL;

V——Final volume of test sample solution, mL;

m——Mass of test sample, g.

Note: The blank value should be subtracted from the above result of calculation.

4 Limit of determination and recovery

4.1 Limit of determination

The limit of determination of this method is 0.02 mg/kg.

4.2 Recovery

According to the experimental data, when the fortifying concentration of α-naphthylacetic acid is in the range of (0.02～5.0) mg/kg, the recovery is 89.5%～103.2%.

Additional explanations:

This standard was proposed by the State Administration of Import and Export Commodity Inspection of the People's Republic of China.

This standard was drafted by the Xiamen Import and Export Commodity Inspection Bureau of the People's Republic of China.

This standard was mainly drafted by Zhou Yu, Zhuang Shuyan.

Note: This English version, a translation from the Chinese text, is solely for guidance.