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中华人民共和国进出口商品检验行业标准

SN 0337－95

出口水果和蔬菜中克百威残留量检验方法

Method for the determination of carbofuran residues in fruits and 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

本标准规定了出口水果和蔬菜中克百威残留量的抽样、制样和气相色谱测定方法。

本标准适用于出口柑桔、荷兰豆中克百威残留量的检验。

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，经组织捣碎机捣碎，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，必需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

用盐酸溶液提取试样中克百威，用苯反提取，离心、脱水、浓缩、定容后，用配有氮磷检测器的气相色谱仪测定，外标法定量。

3.2 试剂和材料

3.2.1 苯：分析纯，用玻璃器皿重蒸馏，取 80～81℃馏分。

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3.2.2　无水硫酸钠：分析纯，650℃灼烧4 h，冷却后贮于密闭容器中。

3.2.3　盐酸：分析纯，1 mol/L。

3.2.4　克百威标准品：纯度≥99.8%。

3.2.5　克百威标准溶液：准确称取适量的克百威标准品，用苯配成浓度为1.00 mg/mL的标准贮备溶液，根据需要用苯配成适当浓度的标准工作溶液。

3.3　仪器和设备

3.3.1　气相色谱仪并配有氮磷检测器。

3.3.2　梨形瓶：具磨口塞，50 mL。

3.3.3　离心管：具磨口塞，50 mL。

3.3.4　涡流振荡器。

3.3.5　旋转蒸发器。

3.3.6　离心机。

3.3.7　高速组织捣碎机。

3.3.8　微量注射器：10 μL。

3.4　测定步骤

3.4.1　提取

称取试样5.0 g（精确到0.1 g）置于50 mL离心管内，加10 mL盐酸溶液（1 mol/L），用涡流振荡器振荡5 min后，加5 mL苯，振荡3 min。以3 000 r/min离心5 min，移出上层提取液。再按上述步骤分别用5 mL苯提取2次。合并苯提取液，经无水硫酸钠脱水，收集于梨形瓶内。在60 ℃水浴中减压旋转浓缩至约1 mL，用苯冲洗瓶壁后定容至2 mL，供气相色谱测定。

3.4.2　测定

3.4.2.1　色谱条件

- **a.**　色谱柱：石英毛细管柱，5 m×0.53 mm（内径），HP-1，2.65 μm（膜厚）；
- **b.**　色谱柱温度：160℃；
- **c.**　进样口温度：200℃；
- **d.**　检测器温度250℃；
- **e.**　氮气：纯度≥99.99%，10 mL/min；
- **f.**　氢气：3.0 mL/min；
- **g.**　空气：70 mL/min。

3.4.2.2　色谱测定

根据样液中克百威含量情况，选定峰高相近的标准工作溶液。标准工作溶液和样液中克百威的响应值均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，克百威保留时间约为1.5 min。

3.4.3　空白试验

除不加试样外，按上述操作步骤进行。

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克百威含量：

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

式中：X——试样中克百威含量，mg/kg；

h——样液中克百威的峰高，mm；

h_s——标准工作液中克百威的峰高，mm；

c——标准工作液中克百威的浓度，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 测定低限、回收率

4.1 测定低限

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：克百威添加浓度在 0.02～5.0 mg/kg 范围内，回收率为 91.5%～97.9%。

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。

本标准由中国进出口商品检验技术研究所、中华人民共和国厦门进出口商品检验局负责起草。

本标准主要起草人王超、周昱、庄无忌、庄宿燕。

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

SN 0337—95

Method for the determination of carbofuran residues in fruits and vegetables for export

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1 Scope and field of application

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

This standard is applicable to the determination of carbofuran residues in orange and green bean 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 packages 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 should 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. 1, 1995

3 Method of determination

3.1 Principle

After the sample is extracted by hydrochloric acid, the hydrochloric acid solution is re-extracted by benzene, then the organic phase is centrifuged, dehydrated, concentrated and diluted to definite volume, finally the extract is analyzed by GC with N-P detector, quantified by using external standard method.

3.2 Reagents and materials

3.2.1 Benzene: A. R., redistilled with all-glass apparatus, collect the distillate of 80—81℃。

3.2.2 Anhydrous sodium sulfate: A. R., ignite at 650℃ for 4 h, and keep in a tightly closed container after cooling.

3.2.3 Hydrochloric acid: A. R., 1 mol/L.

3.2.4 Standard of carbofuran: Purity≥99.8%.

3.2.5 Carbofuran standard solution:

Accurately weigh an adequate amount of standard carbofuran, dissolve in benzene and prepare a solution of 1.00 mg/mL as the standard stock solution, from which standard working solution of appropriate concentration are prepared according to requirement.

3.3 Apparatus and equipment

3.3.1 Gas chromatograph, equipped with N-P detector.

3.3.2 Pear-shaped flask(50 mL), with ground stopper.

3.3.3 Centrifugal tube(50 mL), with ground stopper.

3.3.4 Turbulent mixer.

3.3.5 Rotary evaporater.

3.3.6 Centrifuge.

3.3.7 High-speed blender.

3.3.8 Micro-syringe: 10 μL.

3.4 Procedure

3.4.1 Extraction

Weigh 5.0 g (accurate to 0.1 g)of the test sample into a 50 mL centrifugal tube, add 10 mL of 1 mol/L hydrochloric acid, and shake for 5 min by a turbulent mixer, then add 5 mL of benzene and shake for 3 min by the turbulent mixer, centrifuge 5 min at 3 000 r/min. Collect the benzene extract in a pear-shaped flask. Reextract twice each with 5 mL portions of benzene and centrifuge as above. Combine the extract and use anhydrous sodium sulfate to remove water. The combined solution is concentrated in the rotary evaporator under reduced pressure at 60℃ water bath to co 1 mL. The concentrated solution is transfered into a graduated test tube, and exactly diluted to 2 mL. The solution is used for gas chromatograph determination.

3.4.2 Determination

3.4.2.1 GC operating condition

a. Column: Fused silica capillary column, 5 m×0.53 mm(id), HP-1, 2.65 μm(film thickness);

b. Column temperature: 160℃;

c. Injection port temperature: 200℃;

d. Detector temperature: 250℃;

e. Nitrogen: Purity≥99.99%, 10 mL/min;

f. Hydrogen: 3.0 mL/min;

g. Air: 70 mL/min.

3.4.2.2 GC determination

According to the approximate concentration of carbofuran in the sample solution, select the standard working solution with similar peak height to that of sample solution. The responses of carbofuran in the standard solution and sample solution should be in the linear range of the instrumental detection. The standard working solution should be randomly injected in-between injections of the sample solution of equal volume. Under the above chromatographic condition, the retention time of carbofuran is about 1.5 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 of result

The calculation of result 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 carbofuran in test sample, mg/kg;

h——Peak height of carbofuran in the sample solution, mm;

h_s——Peak height of carbofuran in the standard working solution, mm;

c——Concentration of carbofuran in the standard working solution, μg/mL;

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

m——Mass of the 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 concentration of carbofuran is in the range of 0.02—5.0 mg/kg, the recovery is 91.5%—97.9%.

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 China Import & Export Commodity Inspection Technology Institute and Xiamen Import and Export Commodity Inspection Bureau of the People's Republic of China.

This standard was mainly drafted by Wang Chao, Zhou Yu, Zhuang Wuji, Zhuang Suyan.

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

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