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中华人民共和国进出口商品检验行业标准

SN 0338—95

出口水果中敌菌丹残留量检验方法

Method for the determination of captafol
residues in fruits for export

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1 主题内容和适用范围

本标准规定了出口水果中敌菌丹残留量检验的抽样、制样和气相色谱测定方法。
本标准适用于出口苹果、菠萝中敌菌丹残留量的检验。

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

以丙酮提取,再用石油醚抽提丙酮-水溶液,提取液经脱水、浓缩、定容后,用配有电子俘获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 石油醚:分析纯,重蒸馏,收集 65~75℃馏分。

3.2.2 丙酮:分析纯,重蒸馏。

3.2.3 无水硫酸钠:分析纯,650℃灼烧4 h,冷却后贮于密闭容器中。

3.2.4 敌菌丹标准品:纯度≥95%。

3.2.5 敌菌丹标准溶液:准确称取适量的敌菌丹标准品,用适量的丙酮预溶,再用石油醚配成浓度为1.000 mg/mL的标准贮备溶液,根据需要再配成适当浓度的标准工作溶液。

3.3 仪器和设备

3.3.1 气相色谱仪并配有电子俘获检测器。

3.3.2 多功能微量样品处理仪,或相当者。

3.3.3 旋涡振荡器。

3.3.4 离心机。

3.3.5 全玻璃系统蒸馏装置。

3.3.6 离心管:具磨口塞,10 mL,25 mL。

3.3.7 无水硫酸钠柱:筒形漏斗,3 cm(内径),内装2 cm高的无水硫酸钠。

3.3.8 精密可调体积移液管:5 000 μL。

3.3.9 微量注射器:10 μL。

3.4 测定步骤

3.4.1 提取

称取试样约2.0 g(精确到0.1 g)于10 mL离心管中,加3 mL丙酮,振荡2 min,离心(3 000 r/min)1 min,吸出上层清液,置于25 mL离心管中。再用2 mL丙酮重复提取一次,合并提取液。往提取液中加入10 mL蒸馏水,每次用3 mL石油醚萃取三次,合并萃取液。用4 mL蒸馏水洗涤二次,弃去水相。石油醚相经无水硫酸钠柱脱水,并用少量石油醚洗涤无水硫酸钠柱,流出液收集于25 mL离心管内,于50℃用空气吹干,用石油醚定容为1.0 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 SE-30,25 m×0.53 mm(内径);
- b. 色谱柱温度:185℃;
- c. 进样口温度:240℃;
- d. 检测器温度:280℃;
- e. 氮气:纯度≥99.99%,10 mL/min。

3.4.2.2 色谱测定

根据样液中敌菌丹含量情况,选定峰高相近的标准工作溶液。标准工作溶液和样液中敌菌丹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,敌菌丹保留时间约为8.2 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:

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

式中: X——试样中敌菌丹残留量,mg/kg;

h——样液中敌菌丹的峰高,mm;

h_s——标准工作溶液中敌菌丹的峰高,mm;

c——标准工作溶液中敌菌丹的浓度,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 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: 敌菌丹浓度在 0.02~0.20 mg/kg 范围, 回收率为 89.5%~114.8%。

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北京进出口商品检验局负责起草。

本标准主要起草人胡文炬、王晓强、张军。

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

SN 0338—95

Method for the determination of captafol residues in fruits for export

1 Scope and field of application

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

This standard is applicable to the determination of captafol residues in fruits, such as apple, pineapple 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 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°C .

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.

3 Method of determination

3.1 Principle

The sample is extracted by acetone, the acetone-water solution is extracted by petroleum ether. After the removal of water, the solution is concentrated and diluted to a definite volume, the solution is analyzed by GC with electron-capture detector, using external standard method.

3.2 Reagents and materials

3.2.1 Petroleum ether; A. R., redistill and collect the distillate of 65—75°C.

3.2.2 Acetone; A. R., redistilled.

3.2.3 Anhydrous sodium sulfate; A. R., ignite at 650°C for 4 h, and keep in a tightly closed container after cooling.

3.2.4 Captafol standard; Purity $\geq 95\%$.

3.2.5 Captafol standard solution;

Accurately weigh an adequate amount of captafol standard, first dissolve in an adequate amount of acetone, then prepare a solution of 1.000 mg/mL with petroleum ether 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 electron-capture detector.

3.3.2 Multifunction sample treatment unit for microchemical method or equivalent apparatus.

3.3.3 Vortex shaker.

3.3.4 Centrifuge.

3.3.5 All-glass distillation system.

3.3.6 Centrifuge tube, 10 mL, 25 mL, with ground stopper.

3.3.7 Column of anhydrous sodium sulfate; A cylindrical funnel 3 cm(id), filled with 2 cm height of anhydrous sodium sulfate.

3.3.8 Accurate volume-adjustable pipet; 5 000 μL .

3.3.9 Micro syringe; 10 μL .

3.4 Procedure

3.4.1 Extraction

Weigh ca 2.0 g of the test sample (accurate to 0.1 g) into a 10 mL centrifuge tube, add 3 mL of acetone, stopper and shake for 2 min by a vortex shaker, centrifugalize (3 000 r/min) for 1 min, the upper clear extract is transferred into a 25 mL centrifuge tube. Repeat the procedure with 2 mL of acetone, combine the extracts. Add 10 mL of distilled water into the acetone extract, extract 3 times with petroleum ether, 3 mL each time. Combine petroleum ether extracts, wash 2 times with 4 mL of distilled water, discard the water phase. Pass the petroleum phase through the column of anhydrous sodium sulfate to remove water, and rinse the column with a small amount of petroleum ether, collect the solution and rinsings into a 25 mL centrifuge tube. Blow dry with air at 50°C in a multifunction sample treatment unit (or equivalent), dilute quantitatively to 1.0 mL with petroleum ether. The solution is used for gas chromatographic determination.

3.4.2 Determination

3.4.2.1 GC operating condition

- Column; Fused silica capillary column, SE-30, 25 m \times 0.53 mm(id);
- Column temperature; 185°C;

- c. Injection port temperature; 240℃;
- d. Detector temperature; 280℃;
- e. Nitrogen; Purity ≥ 99.99%, 10 mL/min.

3.4.2.2 GC determination

According to the approximate concentration of captafol in the sample solution, select the standard working solution with similar peak height to that of sample solution. The responses of captafol in the standard 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 sample solution of equal volume. Under the above chromatographic condition, the retention time of captafol is about 8.2 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 the sample.

3.4.4 Calculation and expression of the result

The calculation of the content of the captafol is carried out by a GC data processor or according to the following formula:

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

where

- X —Content of captafol in test sample, mg/kg;
- h —Peak height of captafol in the sample solution, mm;
- h_s —Peak height of captafol in the standard working solution, mm;
- c —Concentration of captafol 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 captafol is in the range of 0.02—0.20 mg/kg, the recovery is 89.5%—114.8%.

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 Beijing Import and Export Commodity Inspection Bureau of the People's Republic of China.

This standard was mainly drafted by Hu Wenju, Wang Xiaoqiang, Zhang Jun.

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

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