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# 中华人民共和国进出口商品检验行业标准

SN/T 0348.2—95

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## 出口茶叶中三氯杀螨醇残留量 检验方法 液相色谱法

Method for the determination of dicofol residues  
in tea for export  
—Liquid chromatography

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### 1 主题内容与适用范围

本标准规定了出口茶叶中三氯杀螨醇残留量检验的抽样、制样和高压液相色谱测定方法。  
本标准适用于出口茶叶中三氯杀螨醇残留量的检验。

### 2 抽样和制样

#### 2.1 检验批

以不超过 2 000 件为一检验批。

同一检验批的商品应具有相同的特征,如品种、包装、标记、产地、规格和等级等。

#### 2.2 抽样数量

批量,件	最低抽样数量,件
1~5	1
6~50	2
51~500	11
501~1 000	16
1 001~1 500	19
1 501~2 000	20

#### 2.3 抽样方法

按 2.2 规定的抽样件数从堆垛不同部位抽取样箱,逐件开启,用取样铲抽取样品,每箱至少取 500 g。将所取样品混合、充分拌匀,缩分出 500 g。装入清洁密封的样品筒内,加封后标明标记并及时送交实验室。

#### 2.4 试样制备

将取回样品全部磨碎使通过 20 目筛,用四分法匀分出二份,每份 50~100 g,作为试样,立即装入洁净容器内,密封并标明标记。

#### 2.5 试样保存

将试样于-5℃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残留物含量发生变化。

### 3 测定方法

#### 3.1 方法提要

以丙酮-正己烷混合液提取,蒸发去掉丙酮,磺化法净化,取正己烷相,蒸去正己烷,用甲醇溶解残渣。用配有紫外检测器的液相色谱仪测定,外标法定量。

### 3.2 试剂和材料

3.2.1 丙酮:分析纯,重蒸馏。

3.2.2 正己烷:分析纯,重蒸馏。

3.2.3 甲醇:紫外光谱纯。

3.2.4 浓硫酸:优级纯,含量98%。

3.2.5 三氯杀螨醇标准品:纯度 $\geq 95\%$ 。

3.2.6 三氯杀螨醇标准溶液:准确称取适量的标准品,用甲醇配成浓度为1.00 mg/mL的标准储备溶液,再根据需要用甲醇稀释成适当浓度的标准工作溶液。

### 3.3 仪器和设备

3.3.1 高压液相色谱仪配有紫外检测器。

3.3.2 微量进样器:50  $\mu$ L。

3.3.3 可调定量移液器。

3.3.4 旋涡混合器。

3.3.5 自动平衡离心机。

3.3.6 恒温水浴锅。

3.3.7 注射器:10 mL。

3.3.8 多功能微量化学样品处理仪或相当仪器。

3.3.9 试管:10 mL。

### 3.4 测定步骤

#### 3.4.1 提取

称取1.000 g试样于10 mL试管中,移入1.0 mL丙酮和2.0 mL正己烷。将试管放于旋涡混合器上剧烈旋混30 s。然后移入自动平衡离心机中,于3 000 r/min离心2 min。取出,将上清液小心移入10 mL试管中。于装有残渣的试管中再加入1.0 mL丙酮和2.0 mL正己烷;重复上述操作二次,即提取三次。将提取液并于同一试管中,得提取液约8 mL。

将盛有提取液的试管放入恒温水浴锅中,用60°C恒温蒸发、浓缩至约4 mL,取出。

#### 3.4.2 净化

在盛有浓缩提取液的试管中缓慢加入1.5 mL浓硫酸,轻轻摇动,然后置于旋涡混合器上稍作旋混。将试管放入离心机中,于3 500 r/min离心2 min。取出,用注射器将下层磺化液吸出并弃掉。于提取液试管中再加入1.5 mL浓硫酸,旋混30 s,于3 500 r/min离心2 min。取出,用移液器小心将上清液移入一干净试管中。在取出上清液后的试管中加入2 mL正己烷,旋混30 s,于3 500 r/min离心2 min。取出,用移液器将上清液小心移入同一试管中,备用。

#### 3.4.3 浓缩

将盛有正己烷提取液的试管,于50°C空气流下,使正己烷蒸干。准确加入1.00 mL甲醇以溶解残留物,所得溶液即为待测液。

#### 3.4.4 测定

##### 3.4.4.1 色谱条件

- a. 色谱柱:150 mm $\times$ 3.9 mm(内径)不锈钢柱,内填10  $\mu$ m C<sub>18</sub>键合固定相;
- b. 流动相:甲醇-水(90+10);
- c. 测定波长:230 nm;
- d. 流速:0.5 mL/min;
- e. 测定温度:室温。

### 3.4.4.2 色谱测定

根据样液中三氯杀螨醇含量情况,选定峰面积相近的标准工作溶液。标准工作溶液和样液中的三氯杀螨醇响应值均应在仪器检测线性范围内。对标准工作液和样液等体积参插进样测定。在上述色谱条件下,三氯杀螨醇保留时间约为 5.5 min。

### 3.4.5 空白试验

除不加试样外,按上述测定步骤进行。

### 3.4.6 结果计算和表述

用色谱数据处理机或按下列公式计算:

$$X = \frac{A \cdot c \cdot V}{A_s \cdot m}$$

式中:  $X$ ——试样中三氯杀螨醇残留量,mg/kg;

$A$ ——样液中三氯杀螨醇面积,mm<sup>2</sup>;

$A_s$ ——标准工作液中三氯杀螨醇峰面积,mm<sup>2</sup>;

$c$ ——标准工作液中三氯杀螨醇浓度,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

本方法测定低限为 0.1 mg/kg。

### 4.2 回收率

回收率的实验数据:三氯杀螨醇浓度在 0.1~4.0 mg/kg 范围内,回收率为 79.4%~95.0%。

### 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重庆进出口商品检验局负责起草。

本标准主要起草人刘尧志、张纯勇。

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

SN/T 0348.2-95

**Method for the determination of dicofol residues  
in tea for export  
—Liquid chromatography**

**1 Scope and field of application**

This standard specifies the methods of sampling, sample preparation and determination by high pressure liquid chromatography of dicofol residues in tea for export.

This standard is applicable to the determination of dicofol residues in tea for export.

**2 Sampling and sample preparation**

**2.1 Inspection lot**

The quantity of an inspection lot should not be more than 2 000 packages.

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

**2.2 Quantity of the sample taken**

Number of packages in each inspection lot	Minimum number of packages to be taken
1—5	1
6—50	2
51—500	11
501—1 000	16
1 001—1 500	19
1 501—2 000	20

**2.3 Sampling procedure**

A number of packages specified in 2.2 are taken from different locations of the stack and opened one by one. About 500 g of sample tea are taken with a sampling shovel from each package. The samples taken are fully mixed, reduced to 500 g, placed in a clean sample can, sealed, labeled and sent to laboratory in time.

**2.4 Preparation of test sample**

All the sample is ground and let pass through a 20-mesh sieve and reduced to two portions (50—100 g each portion) as the test samples by quartering. The samples should be immediately placed in a clean container, sealed and labelled.

**2.5 Storage of test sample**

The test samples should be stored below  $-5^{\circ}\text{C}$ .

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

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

### 3 Method of determination

#### 3.1 Principle

The sample is extracted with a mixture of acetone and n-hexane, the extract is evaporated to remove the acetone, and cleaned up with sulfonation. Take the n-hexane phase, then evaporate to dryness. Dissolve the residue in methanol. Finally, the solution is analyzed by LC with a UV detector, using external standard method.

#### 3.2 Reagents and materials

3.2.1 Acetone; AR, redistilled.

3.2.2 n-Hexane; AR, redistilled.

3.2.3 Methanol; UV spectrophotometric grade.

3.2.4 Sulphuric acid; GR, purity  $\geq 98\%$ .

3.2.5 Dicofol standard; Purity  $\geq 95\%$ .

3.2.6 Dicofol standard solution; Accurately weigh an adequate amount of dicofol standard, dissolve in methanol and prepare a solution of 1.00 mg/mL as the standard stock solution. Then, as required, dilute it with methanol to appropriate concentration to serve as the standard working solution.

#### 3.3 Apparatus and equipment

3.3.1 High pressure liquid chromatograph, equipped with UV detector.

3.3.2 Micro-injector; 50  $\mu$ L.

3.3.3 Volume adjustable pipet.

3.3.4 Vortex mixer.

3.3.5 Auto-balanced centrifuge.

3.3.6 Constant temperature water-bath.

3.3.7 Syringe; 10 mL.

3.3.8 Multi-functional micro-chemical sample processor or equivalent apparatus.

3.3.9 Test-tube; 10 mL.

#### 3.4 Procedure

##### 3.4.1 Extraction

Weigh 1.000 g of the test sample into a 10 mL test-tube. Add 1.0 mL of acetone and 2.0 mL of n-hexane. Vigorously shake the test-tube on a vortex mixer for 30 s, centrifugalize in an auto-balanced centrifuge at 3 000 r/min for 2 min. Take out the test tube and carefully transfer the supernatant solution to another 10 mL test-tube. Add 1.0 mL of acetone and 2.0 mL of n-hexane to the test-tube with the residue, repeat the above operations twice and combine the extracts in the same 10 mL test-tube, with the total extract of ca 8 mL.

Place the test-tube with the extract in a constant temperature water-bath, concentrate it to ca 4 mL by evaporation at the constant temperature of 60°C before taking it out.

##### 3.4.2 Cleanup

Gradually add 1.5 mL of con.  $H_2SO_4$  to the test-tube with the concentrated extract, shake and rotate gently for a little while in a vortex mixer. Centrifugalize at 3 500 r/min for 2 min and take it out. Remove and discard the lower sulfonated solution with a syringe. Add 1.5 mL of conc.  $H_2SO_4$  to the same test tube, rotate it for 30 s and centrifugalize at 3 500 r/min for 2 min and take it out. Carefully

pipet the supernatant solution to a clean test-tube. Add 2 mL of n-hexane to the test-tube from which the supernatant solution has been removed, rotate the test-tube for 30 s, centrifugalize it at 3 500 r/min for 2 min and take it out. Pipet the supernatant solution and combine it in the same clean testtube.

### 3.4.3 Concentrating

Evaporate the n-hexane in the test-tube with an air current at 50°C to dryness. Add exactly 1.0 mL of methanol to dissolve the residues and the solution is used for determination.

### 3.4.4 Determination

#### 3.4.4.1 LC operating condition

- a. Column;Stainless steel,150 mm×3.9 mm(id),packed with 10 μm C<sub>18</sub> cross-linked (stationary) phase;
- b. Mobile phase;Methanol-water(90+10);
- c. Wave length;230 nm;
- d. Flow rate;0.5 mL/min;
- e. Temperature;Room temperature.

#### 3.4.4.2 LC determination

According to the approximate concentration of dicofol in the sample solution,select the standard working solution with similar peak area to that of the sample solution. The responses of dicofol 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 the sample solution of equal volume. Under the above chromatographic condition, the retention time of dicofol is about 5.5 min.

### 3.4.5 Blank test

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

### 3.4.6 Calculation and expression of result

The calculation of result is carried out by an LC data processor or according to the following formula:

$$X = \frac{A \cdot c \cdot V}{A_s \cdot m}$$

where

- X—Residue content of dicofol in the test sample,mg/kg;  
 A—Peak area of dicofol in the sample solution,mm<sup>2</sup>;  
 A<sub>s</sub>—Peak area of dicofol in the standard working solution,mm<sup>2</sup>;  
 c—Concentration of dicofol 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 oalculation.

## 4 Limit of determination and recovery

### 4.1 Limit of determination

The limit of determination of this method is 0.1 mg/kg.

#### 4.2 Recovery

According to the experimental data, when the concentration of dicofol is in the range of 0.1—4.0 mg/kg, the recovery is 79.4%—95.0%.

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#### **Additional explanations:**

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

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

This standard was mainly drafted by Liu Yaozhi and Zhang Chonyong.

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

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