SN

中华人民共和国出入境检验检疫行业标准

SN/T 0159—2012
代替 SN 0159—1992

出口水果中六六六、滴滴涕、艾氏剂、狄氏剂、七氯残留量测定 气相色谱法

Determination of BHC, DDT, aldrin, dieldrin and heptachlor residues in fruits for export—Gas chromatography method

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前　言

本标准按照 GB/T 1.1—2009 给出的规则起草。

本标准代替 SN 0159—1992《出口水果中艾氏剂、狄氏剂、七氯残留量检验方法》。

本标准与 SN 0159—1992 相比，除编辑性修改外主要技术性修改如下：

——修改了标准名称；将“检验方法”改为“测定”；名称中增加“六六六”、“滴滴涕”、“气相色谱法”；

——标准适用范围增加了脐橙、苹果；

——将原标准中“2 抽样和制样”修改为“6 试样的制备和保存”；

——修改原标准的样品前处理方法；将层析柱改为商品固相萃取柱（TPT）；

——将原标准中所使用填充色谱柱改为毛细管色谱柱。

本标准由国家认证认可监督管理委员会提出并归口。

本标准起草单位：中华人民共和国江西出入境检验检疫局。

本标准主要起草人：左海根、周平勇、占春瑞、李如坤、郭平、魏运隆、祝建新。

原标准于 1992 年首次发布，本次为第一次修订。

出口水果中六六六、滴滴涕、艾氏剂、狄氏剂、七氯残留量测定
气相色谱法

1 范围

本标准规定了出口水果中六六六、滴滴涕、艾氏剂、狄氏剂和七氯残留量的测定方法。

本标准适用于柑桔、脐橙、苹果中六六六、滴滴涕、艾氏剂、狄氏剂和七氯残留量的测定。

2 规范性引用文件

下列文件对于本文件的应用是必不可少的。凡是注日期的引用文件，仅注日期的版本适用于本文件。凡是不注日期的引用文件，其最新版本（包括所有的修改单）适用于本文件。

GB/T 6682 分析实验室用水规格和试验方法

3 方法提要

试样中残留的六六六、滴滴涕、艾氏剂、狄氏剂、七氯用丙酮-石油醚提取，经固相萃取柱净化后，用配有电子捕获检测器的气相色谱仪测定，内标法定量。

4 试剂和材料

除特殊注明外，所有试剂均为分析纯，水为符合GB/T 6682规定的一级水。

4.1 丙酮：HPLC级。

4.2 石油醚：沸程30 ℃～60 ℃。

4.3 正己烷：HPLC级。

4.4 乙酸乙酯：HPLC级。

4.5 丙酮-石油醚（1＋4，体积比）：量取20 mL丙酮和80 mL石油醚，混匀。

4.6 乙酸乙酯-正己烷（1＋9，体积比）：量取10 mL乙酸乙酯和90 mL正己烷，混匀。

4.7 有机氯农药标准品：艾氏剂、狄氏剂、七氯、α-六六六、β-六六六、γ-六六六、δ-六六六、p,p'-滴滴伊、o,p'-滴滴涕、p,p'-滴滴滴、p,p'-滴滴涕，浓度均为100 μg/mL，参见附录A中表A.1。

4.8 内标标准品：环氧七氯（Heptachlor epoxide，CAS号：1024-57-3），纯度≥99％。

4.9 内标标准储备液：准确称取适量环氧七氯标准品，用正己烷配制成100 μg/mL的标准储备液。0 ℃～4 ℃避光保存，有效期为1年。

4.10 混合标准中间溶液：移取适量标准品（4.7），用正己烷稀释为各组分浓度为5 μg/mL的混合标准中间溶液。0 ℃～4 ℃避光保存。

4.11 内标中间溶液：移取适量内标标准储备液，用正己烷稀释成浓度为1 μg/mL的内标中间溶液。0 ℃～4 ℃避光保存。

4.12 标准系列工作液：移取适量混合标准中间溶液（4.10），用正己烷稀释配制成适当浓度的标准系列

工作液。加入适量内标中间溶液(4.11)使标准系列工作液中内标物含量均为0.02 μg/mL。

4.13　固相萃取柱：Cleanert TPT(1 000 mg，6 mL)，或相当者。使用前用5 mL正己烷活化。

4.14　0.45 μm微孔滤膜：有机系。

5　仪器和设备

5.1　气相色谱仪，配微电子捕获检测器(μ-ECD)检测器。

5.2　天平：感量为0.1 mg，0.01 g。

5.3　振荡提取器。

5.4　涡旋混匀器。

5.5　旋转蒸发仪。

5.6　固相萃取装置。

5.7　氮吹仪。

6　试样的制备与保存

6.1　试样制备

取有代表性样品约500 g，不可水洗，取可食用部分将其切成小块，用食品料理机将样品匀浆，混合均匀，装入洁净容器内密封并做好标识。

6.2　试样保存

试样于－18 ℃冰箱内保存。在制样过程中，应防止样品受到污染或发生待测物残留量的变化。

7　测定步骤

7.1　提取

称取5.0 g试样(精确至0.01 g)于50 mL离心管中，加入20 mL丙酮-石油醚(4.5)，振荡提取20 min，并以4 000 r/min离心5 min，上清液收集到150 mL浓缩瓶中，残渣再加入20 mL石油醚，重复上述操作一次，合并2次上清液，于40 ℃下旋转蒸发浓缩至干，用2 mL正己烷溶解，待净化。

7.2　净化

将7.1步骤中所获得的提取液全部转移至活化好的TPT固相萃取柱上，用12 mL乙酸乙酯-正己烷(4.6)分三次洗脱，每次4 mL，收集全部流出液，40 ℃氮气吹干后，加入20 μL内标中间溶液，用正己烷1.0 mL溶解，涡旋混匀后，过0.45 μm微孔滤膜，待测定。

7.3　测定

7.3.1　气相色谱参考条件

气相色谱条件如下：

a)　色谱柱：DB-1701石英毛细管柱，30 m(长度)×0.25 mm(内径)×0.25 μm(膜厚)，或相当者。

b)　柱温程序：50 ℃(1 min)40 ℃/min190 ℃(8 min)15 ℃/min250 ℃(5 min)。

c)　进样口温度：240 ℃。

d)　检测器温度：300 ℃。

e） 载气：氮气，纯度≥99.999%；流速1.5 mL/min。

f） 不分流进样。

g） 进样量：1 μL。

7.3.2 气相色谱测定

根据样液中待测物含量情况，选定含量相近的标准系列工作溶液。标准系列工作溶液和样液中待测物的响应值均应在仪器检测的线性范围内。在上述色谱条件下*α*-六六六的保留时间约为9.53 min；*γ*-六六六的保留时间约为11.15 min；七氯的保留时间约为12.04 min；艾氏剂的保留时间约为13.31 min；*β*-六六六的保留时间约为14.47 min；*δ*-六六六的保留时间约为15.11 min；环氧七氯的保留时间约为15.63 min；*p*,*p*'-滴滴伊的保留时间约为16.59 min；狄氏剂的保留时间约为16.94 min；*o*,*p*'-滴滴涕的保留时间约为17.62 min；*p*,*p*'-滴滴滴的保留时间约为18.47 min；*p*,*p*'-滴滴涕的保留时间约为18.94 min。以标准物质色谱峰的保留时间定性。标准溶液的色谱图参见附录B中图B.1。

7.3.3 空白试验

除不加试样外，按上述测定步骤进行。

8 标准曲线的绘制

本标准采用内标法定量。吸取适量混合标准中间溶液（4.10），用正己烷稀释，并加入20 μL内标中间溶液，使得各标准系列工作液中内标浓度均为0.02 μg/mL，配制成适当浓度的标准系列工作液。以有机氯农药峰面积与内标物峰面积之比为纵坐标，有机氯浓度与内标物浓度比为横坐标绘制标准曲线，或用数据软件中的内标法，绘制标准曲线。

9 结果计算和表述

用色谱数据处理软件中的内标法，绘制标准曲线，按照式（1）计算样品中六六六、滴滴涕、艾氏剂、狄氏剂和七氯的残留量，计算结果应扣除空白值：

$$x_i = \frac{c_i}{m} \times V \qquad \cdots\cdots\cdots\cdots\cdots\cdots\cdots\cdots (1)$$

式中：

x_i ——试样中六六六、滴滴涕、狄氏剂、艾氏剂、七氯的残留量，单位为微克每千克（μg/kg）；

c_i ——标准曲线上查得六六六、滴滴涕、狄氏剂、艾氏剂、七氯的浓度，单位为纳克每毫升（ng/mL）；

V ——试样定容体积，单位为毫升（mL）；

m ——试样质量，单位为克（g）。

10 测定低限和回收率

10.1 测定低限

本方法对*α*-六六六、*γ*-六六六、七氯、艾氏剂、*δ*-六六六、*p*,*p*'-滴滴伊、狄氏剂、*β*-六六六、*o*,*p*'-滴滴涕、*p*,*p*'-滴滴滴和*p*,*p*'-滴滴涕的测定低限均为5 μg/kg。

10.2 添加浓度和回收率

样品的添加浓度及回收率的实验数据见表1。

表 1　样品的添加浓度及回收率的实验数据

农药名称	添加浓度 μg/kg	回收率 %		
		脐橙	柑桔	苹果
α-六六六	5	70.0～88.4	74.1～90.8	73.7～97.1
	10	68.3～76.8	70.7～78.9	70.9～81.7
	50	79.1～92.6	79.5～96.1	85.2～99.3
	200	82.5～87.0	85.2～90.3	88.4～97.2
β-六六六	5	83.5～101.9	79.7～97.4	81.0～97.9
	10	87.9～101.1	87.5～97.9	96.2～105.5
	50	77.2～86.7	77.0～90.2	80.2～95.3
	200	76.6～91.1	78.4～91.0	85.3～90.4
γ-六六六	5	72.9～91.9	88.9～92.9	82.3～95.0
	10	76.6～89.3	70.3～85.6	72.2～86.3
	50	87.4～97.2	82.6～100.9	85.0～102.1
	200	88.3～94.5	89.7～94.6	89.4～100.8
δ-六六六	5	64.9～87.4	84.2～98.3	75.2～99.5
	10	99.3～106.4	86.8～108.5	74.2～87.2
	50	93.0～107.9	91.8～107.7	89.4～108.3
	200	102.1～108.7	99.1～107.6	99.0～107.7
p,*p*'-滴滴伊	5	88.8～104.4	85.7～105.9	90.8～101.6
	10	93.9～106.9	96.5～102.2	92.1～103.6
	50	88.0～104.4	91.7～103.7	91.8～102.6
	200	96.5～105.4	95.3～109.5	98.4～103.5
o,*p*'-滴滴涕	5	67.8～102.5	70.9～84.0	85.2～99.4
	10	97.6～108.0	98.0～106.9	100.1～108.4
	50	95.0～106.5	89.5～109.5	95.0～105.1
	200	87.8～92.7	94.7～101.7	88.7～104.5
p,*p*'-滴滴滴	5	80.9～92.8	82.8～101.7	68.4～81.8
	10	89.8～106.6	96.8～107.9	99.8～108.4
	50	82.8～93.8	73.1～96.2	76.7～94.5
	200	88.9～94.3	83.4～93.4	78.8～102.4
p,*p*'-滴滴涕	5	89.0～103.5	91.5～102.2	85.2～99.4
	10	87.7～106.6	92.5～106.9	94.6～107.1
	50	85.4～106.4	87.1～105.9	86.9～97.2
	200	71.7～79.3	81.0～91.1	74.5～100.2

表 1（续）

农药名称	添加浓度 μg/kg	回收率 %		
		脐橙	柑桔	苹果
七氯	5	97.1～107.2	93.9～105.6	84.6～96.9
	10	71.7～95.3	73.7～96.1	78.1～86.9
	50	94.6～105.9	89.1～106.3	85.3～101.8
	200	80.1～89.7	90.1～97.5	81.8～91.3
狄氏剂	5	84.1～96.4	79.0～87.2	89.6～100.9
	10	97.3～107.9	98.3～103.5	98.7～107.1
	50	91.7～104.0	93.1～104.2	89.8～103.6
	200	94.9～105.6	96.0～108.8	97.8～103.2
艾氏剂	5	91.7～100.0	93.3～102.6	68.5～91.1
	10	79.2～97.2	81.7～96.0	82.5～94.7
	50	84.1～96.4	84.6～98.3	89.4～99.1
	200	88.2～92.7	88.1～95.7	88.9～96.0

附　录　A

（资料性附录）

11 种有机氯农药种类表

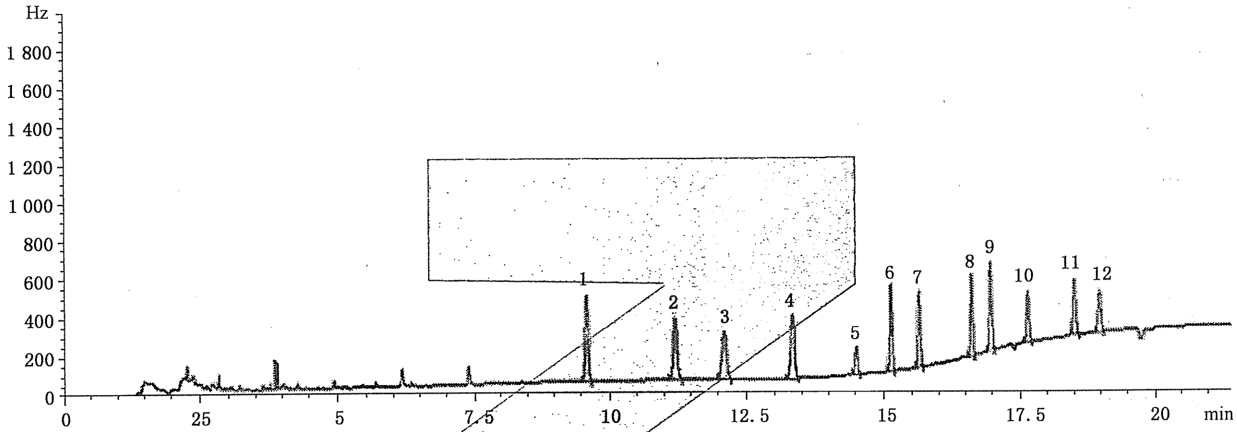
表 A.1　11 种有机氯农药种类表

农药中文名称	农药英文名称	化学分子式	相对分子质量	CAS 号	浓度	溶剂
α-六六六	α-BHC	$C_6H_6Cl_6$	290.83	319-84-6	100 μg/mL	正己烷
β-六六六	β-BHC	$C_6H_6Cl_6$	290.83	319-85-7	100 μg/mL	正己烷
γ-六六六	γ-BHC	$C_6H_6Cl_6$	290.83	58-89-9	100 μg/mL	正己烷
δ-六六六	δ-BHC	$C_6H_6Cl_6$	290.83	319-86-8	100 μg/mL	正己烷
p,*p*'-滴滴伊	*p*,*p*'-DDE	$C_{14}H_8Cl_4$	318.03	72-55-9	100 μg/mL	正己烷
o,*p*'-滴滴涕	*o*,*p*'-DDT	$C_{14}H_9Cl_5$	354.49	789-02-6	100 μg/mL	正己烷
p,*p*'-滴滴滴	*p*,*p*'-DDD	$C_{14}H_{10}Cl_4$	320.04	72-54-8	100 μg/mL	正己烷
p,*p*'-滴滴涕	*p*,*p*'-DDT	$C_{14}H_9Cl_5$	354.49	50-29-3	100 μg/mL	正己烷
七氯	Heptachlor	$C_{10}H_5Cl_7$	373.35	76-44-8	100 μg/mL	正己烷
艾氏剂	Aldrin	$C_{12}H_8Cl_6$	364.90	309-00-2	100 μg/mL	正己烷
狄氏剂	Dieldrin	$C_{12}H_8Cl_6O$	380.90	60-57-1	100 μg/mL	正己烷

附 录 B

（资料性附录）

六六六、滴滴涕、艾氏剂、狄氏剂和七氯标准品气相色谱图



1——α-六六六(9.53 min)；
2——γ-六六六(11.15 min)；
3——七氯(12.04 min)；
4——艾氏剂(13.31 min)；
5——β-六六六(14.47 min)；
6——δ-六六六(15.11 min)；
7——环氧七氯(15.63 min)；
8——*p*,*p*'-滴滴伊(16.59 min)；
9——狄氏剂(16.94 min)；
10——*o*,*p*'-滴滴涕(17.62 min)；
11——*p*,*p*'-滴滴滴(18.48 min)；
12——*p*,*p*'-滴滴涕(18.94 min)。

图 B.1 六六六、滴滴涕、艾氏剂、狄氏剂和七氯标准品气相色谱图

Foreword

This standard is drafted according to GB/T 1.1—2009.

This standard is replace of SN 0159—1992 Method for determination of aldrin,dieldrin and heptachlor residues in fruit for export.

Compared with SN 0159—1992,the main modifications of this standard are as follows:

—Modified the standard name: "Method for determination" to "Determination", added "BHC", "DDT",and "gas chromatography method";

—Enlarged the scope of application:"navel orange and apple";

—Modifled "2 sampling and preparation" in the original standard to"6 sample preparation and storage";

—Modified the sample preparation method:chromatography column to TPT SPE cartridge;

—Replaced the packed column with capillary column.

This standard was proposed by and is under the charge of the National Regulation Commission for Certification and Accreditation.

This standard was drafted by Jiangxi Entry-Exit Inspection and Quarantine Bureau of the People's Repubulie of China.

This standard was mainly drafted by Zuo Haigen,Zhou Pingyong,Zhan Chunrui,Li Rukun,Guo Ping,Wei Yuanlong,Zhu Jianxin.

SN 0159—1992 was proposed in 1992,and this standard is the first modification.

Determination of BHC, DDT, aldrin, dieldrin and heptachlor residues in fruits for export—Gas chromatography method

1 Scope

This standard specifies a method for the determination of BHC, DDT, aldrin, dieldrin and heptachlor residues in fruits for export.

This standard is applicable to the determination of BHC, DDT, aldrin, dieldrin and heptachlor residues in navel orange, mandarin and apple.

2 Normative references

The following documents is necessary for this standard. For dated references, only dated editions shall apply to this standard. For undated references, the latest edition of the normative document referred to applies.

GB/T 6682 Water for analytical laboratory use—Specification and test methods

3 Principle

The pesticide residues in the test samples are extracted with acetone-petroleum ether, then cleaned-up by a TPT SPE cartridge, and determined by gas chromatograph with electron capture detector (GC-ECD). Quantification is performed using internal standard method.

4 Reagents and materials

Unless otherwise specified, all the reagents used should be analytical grade.

4.1 Acetone: HPLC grade.

4.2 Petroleum ether: boiling range 30 ℃～60 ℃.

4.3 *n*-Hexane: HPLC grade.

4.4 Ethyl acetate: HPLC grade.

4.5 Acetone-Petroleum ether (1+4, *V*/*V*): Mix 20 mL of actone with 80 mL of petroleum ether.

4.6 Ethyl acetate-*n*-Hexane (1+9, *V*/*V*): Mix 10 mL of ethyl acetate with 90 mL of *n*-Hexane.

4.7 Standards of aldrin, dieldrin, heptachlor, α-BHC, β-BHC, γ-BHC, δ-BHC, *p*, *p*'-DDE, *o*, *p*'-DDT, *p*, *p*'-DDD, *p*, *p*'-DDT: 100 μg/mL. See annex A table A.1.

4.8 Internal standard: (Heptachlor-epoxide, CAS No. 1024-57-3, Purity≥99%).

4.9 Internal standard stock solution: Accurately weigh adequate amount of heptachlor epoxide, dissolve and dilute with *n*-Hexane to prepare a solution of 100 μg/mL as stock solution. Stored at 0 ℃ ~4 ℃ and away from light, this solution is valid for 1 year.

4.10 Intermediate mixed standard solution: Transfer appropriate amount of standard solutions (4.7), mix them together, and dilute with *n*-Hexane to prepare standard middle solution with each component of 5 μg/mL. The solution should be stored at 0 ℃ ~4 ℃ and away from light.

4.11 Internal standard intermediate solution: Transfer appropriate amount of internal standard stock solution, diluted with hexane to the concentration of 1 μg/mL internal standard intermediate solution. The solution should be stored at 0 ℃ ~4 ℃ and away from light.

4.12 Series of standard working solution: Transfer appropriate amount of mixture standard intermediate solution (4.10), dilute with hexane to prepare series of standard working solution, add appropriate amount of internal standard intermediate solution to prepare series of standard working solution with internal standard concentration of 0.02 μg/mL.

4.13 SPE cartridge: Cleanert TPT (1 000 mg, 6 mL) or equivalent, activated with 5 mL *n*-hexane before use.

4.14 Membrane filter for organic: 0.45 μm.

5 Apparatus and equipments

5.1 Gas chromatograph: equipped with electron capture detector (ECD).

5.2 Electronic balances: accurate to 0.1 mg, 0.01 g.

5.3 Shaker.

5.4 Vortex mixer.

5.5 Rotary evaporator.

5.6 Solid phase extraction equipment.

5.7 Nitrogen evaporator.

6 Sample preparation and storage

6.1 Sample preparation

Take approximately 500 g of representative sample,The edible part is blended and homogenized in a high speed blender,And divided into two equal portions. Each portion is placed into a clean container as test sample,sealed and labeled.

6.2 Storage of test sample

The test samples should be stored below −18 ℃. 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.

7 Procedure

7.1 Extraction

Weigh 5.0 g (accurate to 0.01 g) of the test sample into a 50 mL centrifuge tube, add 20 mL of actone-Petroleum ether (4.5) shake for 20 min. then centrifuge for 5 min at 4 000 r/min, transfer the supernatant layer to a 150 mL concentration bottle, repeat the above procedure twice with 20 mL Petroleum ether and combine the organic phase, Evaporate the combined extract to dryness in a rotary evaporator with bath temperature below 40 ℃. Add 2 mL *n*-hexane to dissolve the residue for further cleanup.

7.2 Clean-up

Transfer the above solution (7.1) on SPE cartridge, elute the cartridge with 3×4 mL Ethyl acetate-*n*-Hexane (4.6), collect all the elute solution and blow to dryness with nitrogen evaporator, Add exactly 1 mL *n*-hexane and 20 μL internal standard intermediate solution, and dissolve the residue for GC determination.

7.3 Determination

7.3.1 GC operating conditions

GC operating conditions are as follows：

a) Chromatographic column：Fused silica capillary column，DB-1701 30 m (length) × 0.25 μm(i. d.) × 0.25 μm (film thickness) or equivalent.

b) Column Temperature：50 ℃ (1 min) 40 ℃/min190 ℃ (5 min) 15 ℃/min 250 ℃ (5 min).

c) Injection port temperature：240 ℃.

d) Detector temperature：300 ℃.

e) Carrier gas：Nitrogen (purity≥99.999%)，flow rate：1.5 mL/min.

f) Injection mode：splitless.

g) Injection volume：1 μL.

7.3.2 GC determination

According to the approximate concentrations of the pesticides in the sample solution，select the standard working solution with similar peak areas to those of the sample solution. The responses of the pesticides in the standard working solution and sample solution should be in the linear range of the instrumental detection. The standard working solution should be randomly injected in-between the injections of the sample solution of equal volume. Under the above GC operating condition，the retention time of α-BHC is about 9.53 min. the retention time of γ-BHC is about 11.15 min，the retention time of heptachlor is about 12.04 min，the retention time of aldrin is about 13.31 min，the retention time of β-BHC is about 14.47 min，the retention time of δ-BHC is about 15.11 min，the retention time of heptachlor epoxide is about 15.63 min，the retention time of *p*，*p*'-DDE is about 16.59 min，the retention time of dieldrin is about 16.94 min，the retention time of *o*，*p*'-DDT is about 17.62 min，the retention time of *p*，*p*'-DDD is about 18.48 min，the retention time of *p*，*p*'-DDT is about 18.94 min. Qualitation by the retention time of standard peak. The chromatogram of the standards see Figure B.1 in annex B.

7.3.3 Blank test

The operation of the blank test is the same as that described in the method of determination but with omission of sample addition.

8 Standard curves

Quantification is performed using internal standard method. transfer amount of mixture standard intermediate solution, dilute with hexane, Add 20 μL internal standard intermediate solution to preapre the internal standard concentration of 0.02 μg/mL in series of standard working solution. Make the ratio between the concentration of organochlorine pesticides and the internal standard as the x-coordinates, the ratio between the peak response of organochlorine pesticides and the internal standard as the y-coordinates, then draw the standard curves.

9 Calculation and expression of result

Calculation the content of the multiple organochlorines residues in the test sample by GC data processor with Internal standards or according to the formula (1). The blank value should be subtracted from the above result of calculation:

$$x_i = \frac{c_i}{m} \times V \qquad \cdots\cdots (1)$$

Where:

x_i—the residue contents of mutiple organochlorines in the test sample, μg/kg;

c_i—the concentration of mutiple organochlorines in standard curve, ng/mL;

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

m—the mass of the test sample, g.

10 Limit of determination and recovery

10.1 Limits of determination

The limits of determination in fruits for BHC, DDT, aldrin, dieldrin and heptachlor are 5 μg/kg.

10.2 Recoveries

According to the experimental data, the fortifying concentrations of BHC, DDT, aldrin dieldrin and heptachlor in fruits and its corresponding recoveries are listed in table 1.

Table 1—The corresponding recoveries of fortified concentrations

Pesticides	Added/(μg/kg)	recoveries/%		
		Navel orange	Mandarin	Apple
α-BHC	5	70.0～88.4	74.1～90.8	73.7～97.1
	10	68.3～76.8	70.7～78.9	70.9～81.7
	50	79.1～92.6	79.5～96.1	85.2～99.3
	200	82.5～87.0	85.2～90.3	88.4～97.2
β-BHC	5	83.5～101.9	79.7～97.4	81.0～97.9
	10	87.9～101.1	87.5～97.9	96.2～105.5
	50	77.2～86.7	77.0～90.2	80.2～95.3
	200	76.6～91.1	78.4～91.0	85.3～90.4
γ-BHC	5	72.9～91.9	88.9～92.9	82.3～95.0
	10	76.6～89.3	70.3～85.6	72.2～86.3
	50	87.4～97.2	82.6～100.9	85.0～102.1
	200	88.3～94.5	89.7～94.6	89.4～1 00.8
δ-BHC	5	64.9～87.4	84.2～98.3	75.2～99.5
	10	99.3～106.4	86.8～108.5	74.2～87.2
	50	93.0～107.9	91.8～107.7	89.4～108.3
	200	102.1～108.7	99.1～107.6	99.0～107.7
p,*p*'-DDE	5	88.8～104.4	85.7～105.9	90.8～101.6
	10	93.9～106.9	96.5～102.2	92.1～103.6
	50	88.0～104.4	91.7～103.7	91.8～102.6
	200	96.5～105.4	95.3～109.5	98.4～103.5
o,*p*'-DDT	5	67.8～102.5	70.9～84.0	85.2～99.4
	10	97.6～108.0	98.0～106.9	100.1～108.4
	50	95.0～106.5	89.5～109.5	95.0～105.1
	200	87.8～92.7	94.7～101.7	88.7～104.5
p,*p*'-DDD	5	80.9～92.8	82.8～101.7	68.4～81.8
	10	89.8～106.6	96.8～107.9	99.8～108.4
	50	82.8～93.8	73.1～96.2	76.7～94.5
	200	88.9～94.3	83.4～93.4	78.8～102.4
p,*p*'-DDT	5	89.0～103.5	91.5～102.2	85.2～99.4
	10	87.7～106.6	92.5～106.9	94.6～107.1
	50	85.4～106.4	87.1～105.9	86.9～97.2
	200	71.7～79.3	81.0～91.1	74.5～100.2
Heptachlor	5	97.1～107.2	93.9～105.6	84.6～96.9
	10	71.7～95.3	73.7～96.1	78.1～86.9
	50	94.6～105.9	89.1～106.3	85.3～101.8
	200	80.1～89.7	90.1～97.5	81.8～91.3

Table 1 (continued)

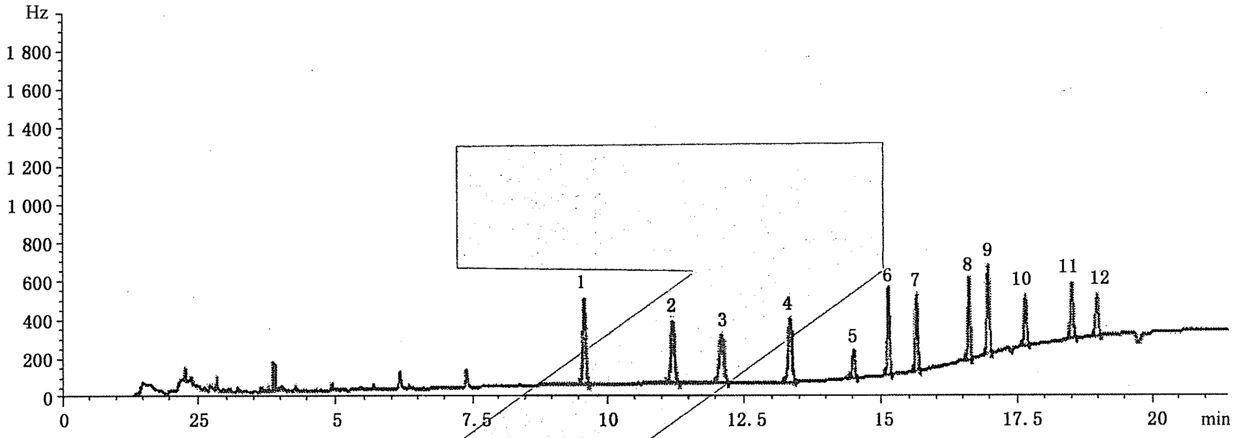
Pesticides	Added/(μg/kg)	recoveries/%		
		Navel orange	Mandarin	Apple
Dieldrin	5	84. 1～96. 4	79. 0～87. 2	89. 6～100. 9
	10	97. 3～107. 9	98. 3～103. 5	98. 7～107. 1
	50	91. 7～104. 0	93. 1～104. 2	89. 8～103. 6
	200	94. 9～105. 6	96. 0～108. 8	97. 8～103. 2
Aldrin	5	91. 7～100. 0	93. 3～102. 6	68. 5～91. 1
	10	79. 2～97. 2	81. 7～96. 0	82. 5～94. 7
	50	84. 1～96. 4	84. 6～98. 3	89. 4～99. 1
	200	88. 2～92. 7	88. 1～95. 7	88. 9～96. 0

Annex A
(Informative annex)
The information of 11 organochlorines

Table A. 1—The information of 11 organochlorines

Pesticides	Formula	molecular weight	CAS No	Concentration	Solvent
α-BHC	$C_6H_6Cl_6$	290. 83	319-84-6	100 μg/mL	Hexane
β-BHC	$C_6H_6Cl_6$	290. 83	319-85-7	100 μg/mL	Hexane
γ-BHC	$C_6H_6Cl_6$	290. 83	58-89-9	100 μg/mL	Hexane
δ-BHC	$C_6H_6Cl_6$	290. 83	319-86-8	100 μg/mL	Hexane
p,*p*'-DDE	$C_{14}H_8Cl_4$	318. 03	72-55-9	100 μg/mL	Hexane
o,*p*'-DDT	$C_{14}H_9Cl_5$	354. 49	789-02-6	100 μg/mL	Hexane
p,*p*'-DDD	$C_{14}H_{10}Cl_4$	320. 04	72-54-8	100 μg/mL	Hexane
p,*p*'-DDT	$C_{14}H_9Cl_5$	354. 49	50-29-3	100 μg/mL	Hexane
Heptachlor	$C_{10}H_5Cl_7$	373. 35	76-44-8	100 μg/mL	Hexane
Aldrin	$C_{12}H_8Cl_6$	364. 90	309-00-2	100 μg/mL	Hexane
Dieldrin	$C_{12}H_8Cl_6O$	380. 90	60-57-1	100 μg/mL	Hexane

Annex B
(Informative annex)
Chromatogram of the standards



1——α-BHC (9.53 min);
2——γ-BHC (11.15 min);
3——Heptachlor (12.04 min);
4——Aldrin (13.31 min);
5——β-BHC (14.47 min);
6——δ-BHC (15.11 min);
7——Heptachlor epoxide (15.63 min);
8——*p*,*p*'-DDE (16.59 min);
9——Dieldrin (16.94 min);
10——*o*,*p*'-DDT (17.62 min);
11——*p*,*p*'-DDD (18.48 min);
12——*p*,*p*'-DDT (18.94 min).

Figure B.1—Chromatogram of the BHC, DDT, aldrin, dieldrin and heptachlor standards