SN

中华人民共和国出入境检验检疫行业标准

SN/T 0663—2014
代替 SN 0663—1997

出口肉及肉制品中七氯和环氧七氯残留量测定

Determination of heptachlor, heptachlor epoxide residues in meats and meat products for export

2014-04-09 发布　　2014-11-01 实施

中华人民共和国
国家质量监督检验检疫总局　发布

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 0663—1997《出口肉及肉制品中七氯和环氧七氯残留量检验方法》。

本标准与 SN 0663—1997 相比主要修改如下：

——修改了标准名称：将“出口肉及肉制品中七氯和环氧七氯残留量检验方法”改为“出口肉及肉制品中七氯和环氧七氯残留量测定”；

——标准适用范围增加了牛肉、鸡肉、火腿肠、香肠；

——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：将层析柱改为商品化固相萃取柱，增加了凝胶渗透色谱(GPC)净化；

——增加了气相色谱-质谱确证。

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、汤国艳、郭平、魏远隆、李如坤、曾海龙、万建春、占春瑞、祝建新。

本标准所代替标准的历次版本发布情况为：

——SN 0663—1997。

出口肉及肉制品中七氯和环氧七氯残留量测定

1 范围

本标准规定了出口肉及肉制品中七氯和环氧七氯残留量的测定方法。

本标准适用于猪肉、牛肉、鸡肉、火腿肠、香肠中七氯和环氧七氯残留量的测定。

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

试样中残留的七氯和环氧七氯用丙酮-正己烷提取，提取液经凝胶渗透色谱(GPC)去除油脂，再经固相萃取柱净化后，供气相色谱测定及气相色谱-质谱确证，外标法定量。

4 试剂和材料

除特殊注明外，所有试剂均为分析纯，水为 GB/T 6682 规定的一级水。

4.1 丙酮：色谱级。

4.2 正己烷：色谱级。

4.3 环己烷：色谱级。

4.4 乙酸乙酯：色谱级。

4.5 无水硫酸钠：650 ℃灼烧 4 h，储于密封容器中备用。

4.6 丙酮-正己烷(2+8，体积比)：量取 20 mL 丙酮和 80 mL 正己烷，混匀。

4.7 乙酸乙酯-环己烷(1+1，体积比)：量取 500 mL 乙酸乙酯和 500 mL 环己烷，混匀。

4.8 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。

4.9 标准品：七氯，环氧七氯 A 和环氧七氯 B 的纯度均大于等于 98.0%，见附录 A 中表 A.1。

4.10 标准储备溶液：100 mg/L，分别准确称取 10 mg 七氯标准物质、环氧七氯 A、环氧七氯 B 于 100 mL 容量瓶中，分别用正己烷溶解并定容至刻度。4 ℃以下避光保存，有效期为 1 年。

4.11 标准混合溶液：分别移取适量标准储备溶液(4.10)用正己烷稀释为各组分浓度为 1 mg/L 的混合标准中间溶液。4 ℃避光保存。

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

4.13 微孔滤膜：0.45 μm，有机系。

5 仪器和设备

5.1 气相色谱仪：配电子捕获检测器(ECD)。

5.2 气相色谱质谱仪：配负化学电离源(NCI)。
5.3 天平：感量为 0.1 mg，0.01 g。
5.4 离心机：转速不低于 4 000 r/min。
5.5 均质器：转速不低于 15 000 r/min。
5.6 旋涡混匀器。
5.7 旋转蒸发仪。
5.8 固相萃取装置。
5.9 氮吹浓缩仪。
5.10 凝胶渗透色谱仪：配有单元泵、馏分收集器。

6 试样的制备与保存

6.1 试样制备

取代表性样品约 1 kg，取可食用部分，经捣碎机充分捣碎均匀，均分成两份，分别装入洁净容器内作为试样，密封，标明标记。

6.2 试样保存

试样于−18 ℃冰箱内密封保存。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 4 g 试样(精确至 0.01 g)于 50 mL 离心管中，加入 5 g 无水硫酸钠和 20 mL 丙酮-正己烷(4.6)，均质提取 1 min，以 4 000 r/min 离心 5 min，上清液过盛有无水硫酸钠的沙芯漏斗后收集到旋转浓缩瓶中。另取一 50 mL 离心管中，加入 20 mL 丙酮-正己烷(4.6)清洗刀头后，洗液倒入前一离心管中，涡旋提取残渣 2 min，以 4 000 r/min 离心 5 min，上清液过无水硫酸钠后合并于旋转浓缩瓶中，在 40 ℃下旋转蒸发至近干，加 4 mL 乙酸乙酯-环己烷(4.7)再浓缩至近干，如此重复 2 次，用少量乙酸乙酯-环己烷溶液(4.7)多次洗涤并转移至 10 mL 容量瓶中，定容至刻度后混匀，待净化。

7.2 净化

7.2.1 凝胶渗透色谱(GPC)净化

7.2.1.1 凝胶色谱条件

凝胶色谱条件如下：

a) 凝胶净化柱：Bio Beads S-X3，700 mm×25 mm(内径)，或相当者；
b) 流动相：乙酸乙酯-环己烷(1+1，体积比)；
c) 流速：4.7 mL/min；
d) 进样量：5.0 mL；
e) 预淋洗时间：0 min～22 min；
f) 收集时间：22 min～34 min。

7.2.1.2 凝胶色谱净化步骤

将 5 mL 待净化液按 7.2.1.1 规定的条件进行净化，合并馏份收集器中的收集液于 150 mL 浓缩瓶

中，于 40 ℃浓缩至近干，加入 2 mL 正己烷溶解残渣，待固相萃取净化。

7.2.2 固相萃取(SPE)净化

将待净化液转移至活化后的 Florisil 固相萃取柱上，用 12 mL 乙酸乙酯-正己烷(4.8)洗脱，收集全部流出液，40 ℃氮气吹干后，用 1.0 mL 正己烷涡旋溶解残渣，过有机相滤膜，供气相色谱测定和气相色谱-质谱确证。

7.3 测定

7.3.1 气相色谱条件

气相色谱条件如下：

a) 色谱柱：30 m×0.32 mm(内径)，膜厚 0.5 μm，DB-608 石英毛细管柱，或相当者；
b) 色谱柱温度：60 ℃保持 1 min，然后以 25 ℃/min 程序升温至 270 ℃，保持 5 min；
c) 进样口温度：240 ℃；
d) 进样方式：无分流进样；
e) 进样量：1 μL；
f) 载气：氦气，纯度≥99.999%，流速 1.5 mL/min；
g) 检测器：温度 300 ℃；尾吹气：氮气 60 mL/min。

7.3.2 气相色谱-质谱条件

气相色谱-质谱条件如下：

a) 色谱柱：30 m×0.25 mm(内径)，膜厚 0.25 μm，DB-1701 石英毛细管柱，或相当者；
b) 载气：氦气，纯度≥99.999%，流速 1.6 mL/min；
c) 色谱柱温度：60 ℃保持 1 min，然后以 20 ℃/min 程序升温至 180 ℃，保持 1 min，再以 10 ℃/min 程序升温至 240 ℃，保持 1 min；
d) 进样口温度：250 ℃；
e) 进样方式：无分流进样，0.75 min 后开阀；
f) 进样量：1 μL；
g) 电离方式：NCI；
h) 色谱-质谱接口温度：280 ℃；
i) 反应气：甲烷气，流量需调至响应值最佳状态；
j) 离子源温度：150 ℃；
k) 溶剂延迟：5 min；
l) 测定方式：选择离子监测方式；
m) 离子检测模式：选择监测离子(SIM)，监测离子及其丰度比见表 1。

表 1 选择离子和相对丰度

待测组分	七氯	环氧七氯 A	环氧七氯 B
选择离子 m/z	266，300(定量)，302，304	318，388(定量)，390，392	318(定量)，388，390，392
相对丰度/%	51∶100∶65∶26	12∶100∶98∶53	100∶61∶57∶30

7.3.3 气相色谱测定

吸取适量标准混合溶液(4.11)，用正己烷稀释配制成适当浓度的标准系列工作液。以待测组分响

应值为纵坐标、待测组分浓度为横坐标绘制标准曲线，采用外标法定量。根据样液中被测物含量情况，选定浓度相近的标准系列工作溶液。标准系列工作溶液和样液中被测物的仪器响应值均应在仪器检测的线性响应范围内，如果含量超过标准曲线范围，采用正己烷稀释到适当浓度后分析。在上述气相色谱条件下，七氯的保留时间约 10.48 min；环氧七氯 A 的保留时间约 11.89 min；环氧七氯 B 的保留时间约 11.71 min。标准溶液的色谱图参见附录 B 中图 B.1。

7.3.4 气相色谱质谱确证

根据样液中待测物含量情况，选定面积相近的标准工作溶液，测定标准工作溶液和样液中待测物的响应值应在仪器检测的线性范围内。在相同气相色谱-质谱条件下，试样待测液中和标准品的选择离子色谱峰在相同保留时间处出现，并且对应质谱碎片离子的质荷比与标准品一致，其相对丰度允许偏差小于20%，则可判定样品中存在对应的待测物。在上述气相色谱-质谱条件下，七氯的保留时间约 11.99 min，环氧七氯 A 的保留时间约 13.92 min，环氧七氯 B 的保留时间约 13.85 min，气相色谱-质谱选择离子色谱图参见附录 C.1。

7.3.5 空白试验

除不加试样外，按上述测定步骤进行。

7.4 结果计算和表述

用色谱数据处理器或按照式(1)计算样品中七氯和环氧七氯残留量。

$$X_i = \frac{c_i}{m} \times V \times \frac{1\,000}{1\,000} \qquad \cdots\cdots(1)$$

式中：

X_i ——试样中七氯、环氧七氯 A 和环氧七氯 B 的残留量，单位为毫克每千克(mg/kg)；

c_i ——样液中七氯、环氧七氯 A 和环氧七氯 B 的浓度，单位为微克每毫升(μg/mL)；

V ——样液最终定容体积，单位为毫升(mL)；

m ——最终样液代表的试样质量，单位为克(g)。

计算结果应扣除空白值。

8 测定低限、回收率

8.1 测定低限(LOQ)

本方法中七氯、环氧七氯 A 和环氧七氯 B 测定低限均为 0.01 mg/kg。

8.2 回收率

样品的添加浓度及回收率的实验数据见表 2。

表 2 样品的添加浓度及回收率范围(GC-ECD)

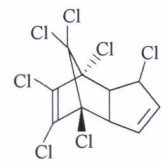
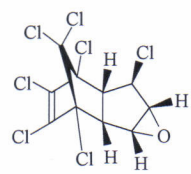
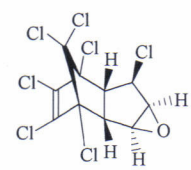
农药名称	添加浓度/(mg/kg)	猪肉 回收率/%	鸡肉 回收率/%	牛肉 回收率/%	火腿肠 回收率/%	香肠 回收率/%
七氯	0.01	100.9～109.7	90.3～108.1	99.5～109.0	103.7～109.5	91.3～98.2
	0.05	90.0～107.2	72.9～86.5	80.5～89.3	94.1～108.4	81.8～85.2
	0.10	86.4～109.6	89.9～109.5	91.2～100.0	104.1～109.9	80.2～93.4

表 2（续）

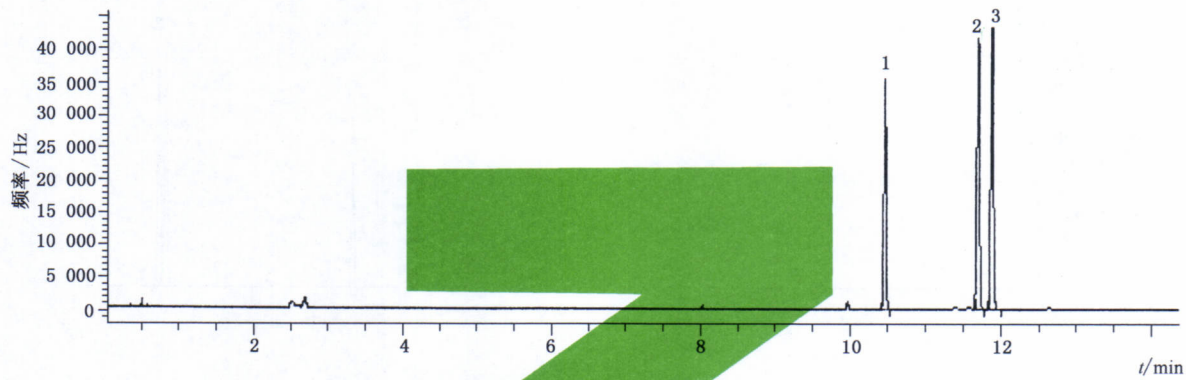
农药名称	添加浓度/(mg/kg)	猪肉 回收率/%	鸡肉 回收率/%	牛肉 回收率/%	火腿肠 回收率/%	香肠 回收率/%
环氧七氯 A	0.01	95.0～107.0	81.0～98.4	84.8～105.2	91.6～105.1	104.4～109.5
	0.05	86.3～101.5	70.9～95.3	80.7～87.9	89.1～98.4	85.8～88.7
	0.10	80.6～102.5	80.4～97.2	84.5～87.5	88.3～102.5	81.2～91.9
环氧七氯 B	0.01	100.7～109.1	99.4～108.7	101.9～107.8	98.6～108.6	90.2～100.0
	0.05	88.1～108.3	73.1～91.3	80.8～88.1	90.1～96.4	84.3～89.9
	0.10	87.6～98.5	81.2～101.1	85.2～89.7	88.9～100.7	80.5～89.9

附 录 A
（规范性附录）
农药种类表

表 A.1 农药种类表

序号	中文名	英文名	分子式	相对分子质量	CAS 号	结构式
1	七氯	heptachlor	$C_{10}H_5Cl_7$	373.35	76-44-8	
2	环氧七氯 A	heptachlor-endo-epoxide (isomer A)	$C_{10}H_5Cl_7O$	389.32	28044-83-9	
3	环氧七氯 B	heptachlor-exo-epoxide (isomer B)	$C_{10}H_5Cl_7O$	389.32	1024-57-3	

附 录 B
（资料性附录）
七氯和环氧七氯标准品气相色谱图



说明：

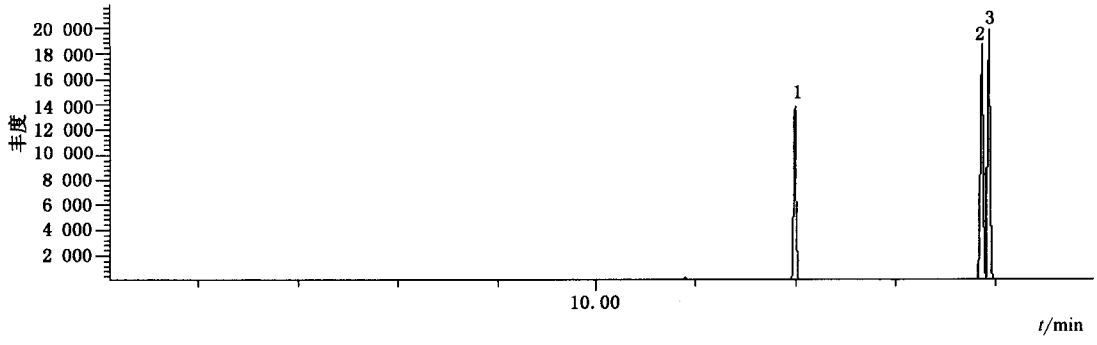
1——七氯；

2——环氧七氯 B；

3——环氧七氯 A。

图 B.1 七氯和环氧七氯标准品气相色谱图

附 录 C
（资料性附录）
七氯和环氧七氯标准品质谱图



说明：

1——七氯；

2——环氧七氯 B；

3——环氧七氯 A。

图 C.1 七氯和环氧七氯标准品选择离子色谱图

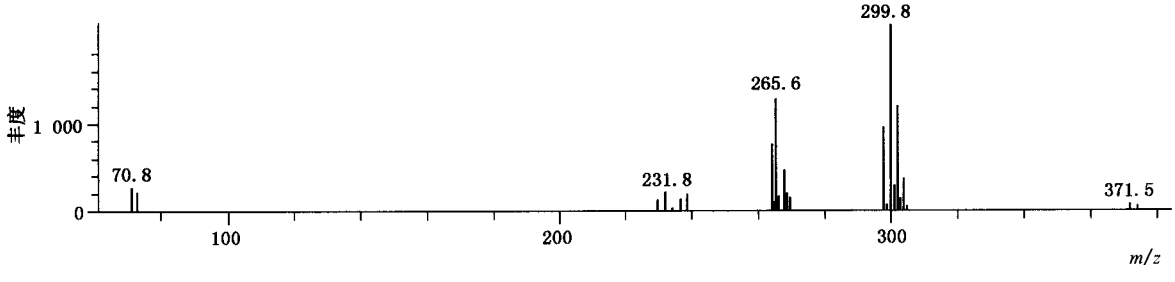


图 C.2 七氯标准品全扫描质谱图

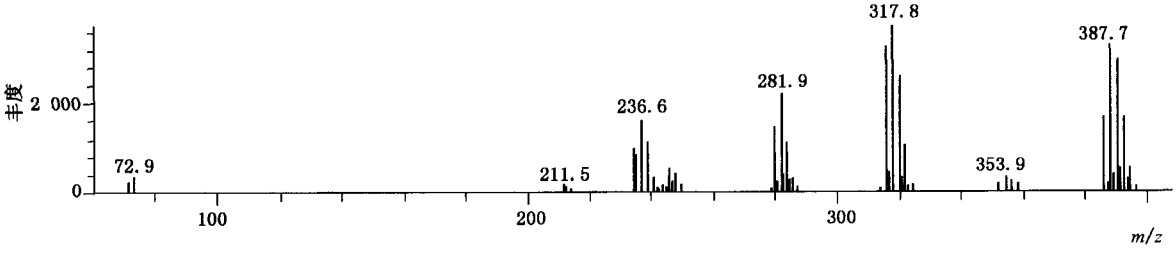


图 C.3 环氧七氯 B 标准品全扫描质谱图

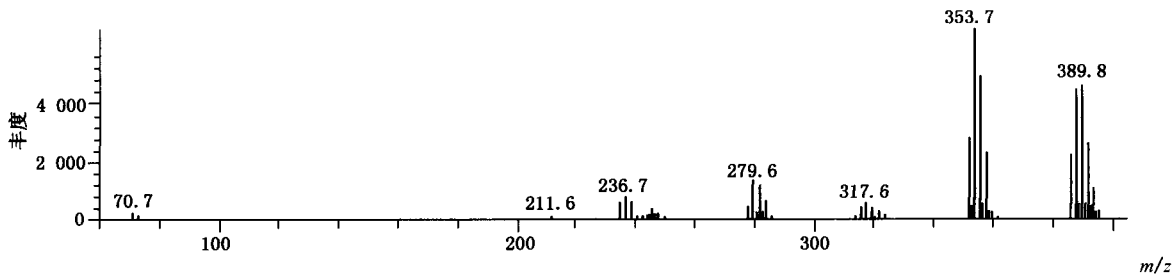


图 C.4　环氧七氯 A 标准品全扫描质谱图

Foreword

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

This standard replaces SN 0663—1997 Method for the determination of heptachlor and heptachlor epoxide residues in meats and meat products for export.

Compared with SN 0663—1997, the main modifications of this standard are as follows:

——Modified the standard name: from "Method for the determination of heptachlor and heptachlor epoxide residues in meats and meat products for export" to "Determination of heptachlor, heptachlor epoxide residues in meats and meat products for export".

——Enlarged the scope of application: "beef, chicken, ham sausage and sausage".

——Modified "2 sampling and preparation" in the original standard to "6 sample preparation and storage".

——Modified the sample preparation method: chromatography column to Florisil SPE cartridge, added the GPC cleanup step.

——Add GC-MS confirmation method.

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 Repubulic of China.

This standard was mainly drafted by: Zuo Haigen, Tang Guoyan, Guo Ping, Wei Yuanlong, Li rukun, Zeng Hailong, Wan Jianchun, Zhan Chunrui, Zhu Jianxin.

SN 0663—1997 was proposed in 1997, and this standard is the first modification.

Determination of heptachlor, heptachlor epoxide residues in meats and meat products for export

1 Scope

This standard specifies a method for the determination of heptachlor and heptachlor epoxide residues in meats and meat products for export.

This standard is applicable to the determination of heptachlor and heptachlor epoxide residues in pork, beef, chicken, ham sausage and sausage.

2 Normative references

The following document is essential to the application of this standard. For dated references, it applies to only date edition. 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 heptachlor and heptachlor epoxide residues in the test samples are extracted with acetone-*n*-hexane, then cleaned-up by GPC and Florisil SPE cartridge, determination is made by GC-FPD and confirmation by GC-MS.

4 Reagents and materials

Unless specifically mentioned, all the reagents used should be analytical grade, water should be the first grade water specified in GB/T 6682.

4.1 Acetone: Chromatographic grade.

4.2 *n*-Hexane: Chromatographic grade.

4.3 Cyclohexane: Chromatographic grade.

4.4 Ethyl acetate: Chromatographic grade.

4.5 Anhydrous sodium sulfate: Ignite at 650 ℃ for 4 h, and keep in a tightly closed container.

4.6 Acetone-*n*-hexane(2+8, *V*/*V*): Mix 20 mL of actone with 80 mL of *n*-hexane.

4.7 Ethyl acetate-cyclohexane(1+1, *V*/*V*): Mix 500 mL of ethyl acetate with 500 mL of cyclohexane.

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

4.9 Standards of heptachlor, heptachlor epoxide A and heptachlor epoxide B, purify≥98.0%. See Table A.1 in Annex A.

4.10 Standard stock solutions: 100 mg/L, accurately weigh 10 mg heptachlor, heptachlor epoxide A and heptachlor epoxide B in 100 mL volumetric flasks, dissolve and dilute with *n*-hexane. Stored below 4 ℃ and away from light, these solutions are valid for 1 year.

4.11 Intermediate mixed standard solution: Transfer appropriate amount of standard solutions (4.10), mix them together, and dilute with *n*-hexane to prepare standard middle solution with each component of 1 mg/L. The solution should be stored at 4 ℃ and away from light.

4.12 SPE cartridge: Florisil SPE tube(1 000 mg, 6 mL) or equivalent, activated with 5 mL *n*-hexane before use.

4.13 Membrane filter for organic: 0.45 μm.

5 Apparatus and equipments

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

5.2 Gas chromatograph-mass spectrometry: equipped with negative chemical ionization source (NCI).

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

5.4 Centrifuge: ≥4 000 r/min.

5.5 Homogenizer: ≥15 000 r/min.

5.6 Vortex mixer.

5.7 Rotary evaporator.

5.8 Solid phase extraction equipment.

5.9 Nitrogen evaporator.

5.10 Gel permeation chromatograph(GPC).

6 Sample preparation and storage

6.1 Sample preparation

Take approximately 1 kg 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 4 g(accurate to 0.01 g) of the test sample into a 50 mL centrifuge tube, add 5 g anhydrous sodium sulfate, 20 mL of acetone-*n*-hexane(4.6) homogenize for 1 min. then centrifuge for 5 min at 4 000 r/min, transfer the supernatant layer to a round-bottomed flask through anhydrous sodium sulfate layer. Take another 50 mL centrifuge tube, add 20 mL acetone-*n*-hexane(4.6) to wash the homogenizer blender, and transfer to the former 50 mL centrifuge tube, votex for 2 min, centrifuge for 5 min at 4 000 r/min, and combine the upper layer to the round-bottomed flask through anhydrous sodium sulfate layer. Evaporate the combined extract to nearly dryness in a rotary evaporator with bath temperature below 40 ℃, add 4 mL ethyl acetate-cyclohexane(4.7) and evaporate to dryness, repeat it twice. Add acetate-cyclohexane(4.7) to dissolve residues and transfer to 10 mL volumetric flask, repeat it twice and dilute to 10 mL, prepare for the following clean-up procedure.

7.2 Clean-up

7.2.1 GPC Clean-up

7.2.1.1 GPC operating conditions

The GPC operating conditions are as follows:

a) GPC column:Bio Beads S-X3,700 mm × 25 mm(i.d.),or equivalent;

b) Mobile phase:Cyclohexane-ethyl acetate(1+1,*V*/*V*);

c) Flow rate:4.7 mL/min;

d) Injection volume:5.0 mL;

e) Time for rinsing:0 min~22 min;

f) Time for collecting the eluate:22 min~34 min.

7.2.1.2 GPC clean-up operating

Transfer the above 5 mL solution into an GPC column,proceed as section 7.2.1.1.Combined the elutes in a 150 mL concentrate bottle,evaporate to dryness in a rotary evaporator with a bath temperature at 40 ℃.dissolve the residue with 2 mL of *n*-hexane for furthermore cleanup.

7.2.2 SPE Clean-up

Transfer the above solution into the preconditioned florisil column. Wash the column with 12 mL ethyl acetate-*n*-hexane(4.8),collect all the elutes and nitrogen blow to dryness in nitrogen blowing device with a bath temperature at 40 ℃.Dissolve the residue and dilute exactly to 1 mL *n*-hexane for GC determination and GC-MS confirmation.

7.3 Determination

7.3.1 GC operating conditions

GC operating conditions are as following:

a) Chromatographic column:DB-608 Fused silica capillary column,30 m × 0.32 μm(i.d.) × 0.5 μm (film thickness) or equivalent;

b) Column Temperature:60 ℃ for 1 min,25 ℃/min ramp to 270 ℃,held for 5 min;

c) Injection port temperature:240 ℃;

d) Injection mode:splitless;

e) Injection volume:1 μL;

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

g) Detector:temperature,300 ℃.Makeup gas(nitrogen),60 mL/min.

7.3.2 GC-MS operating conditions

GC-MS operating conditions are as following:

a) Chromatographic column:DB-1701 silica capillary column,30 m × 0.25 mm(i.d.) × 0.25 μm(film thicknes),or equivalent;

b) Carrier gas:Helium(purity≥99.999%),flow rate:1.6 mL/min;

c) Column temperature:60 ℃ for 1 min,20 ℃/min ramp to 180 ℃,held for 1 min,finally 10 ℃/min ramp to 240 ℃,and held for 1 min;

d) Injector port temperature:250 ℃;

e) Injection mode:Splitless,purge on after 0.75 min;

f) Injection volume:1 μL;

g) Ionization mode:NCI;

h) Interface temperature:280 ℃;

i) Reaction gas:methane,adjust the flow rate to appropriate flow to get the maximum response;

j) Ionization souce:150 ℃;

k) Solvent delay:5 min;

l) Determination mode:SIM mode;

m) Determination mode:Selected monitoring ion(m/z):see Table 1.

Table 1 Selective ions and relative abundance

Compounds	Heptachlor	Heptachlor-endo-epoxide (Isomer A)	Heptachlor-exo-epoxide (Isomer B)
Selected ions m/z	266,300(quantitative ion), 302,304	318,388(quantitative ion), 390,392	318(quantitative ion), 388,390,392
Relative abundance/%	51 : 100 : 65 : 26	12 : 100 : 98 : 53	100 : 61 : 57 : 30

7.3.3 GC determination

Transfer appropriate amount of standard solutions(4.11),and dilute with *n*-hexane to prepare standard series working solutions.The standard linear was carried out by plotting the chromatographic response for each target compounds as y-axis and the final concentrations of each target compounds as x-axis.The result was calculated by external standard methods.According to the approximate concentrations of the compounds in the sample solution,select the standard working solution with similar peak areas to those of the sample solution.The responses of the target compounds in the standard working solution and sample solution should be in the linear range of the instrumental detection. Under the above GC operation condition,the retention time of heptachlor is 10.48 min,heptachlor epoxide A 11.89 min,and heptachlor epoxide B 11.71 min.the standard solution see Figure B.1 in Annex B.

7.3.4 GC-MS confirmation

According to the approximate concentration of the pesticide in the sample solution,select the standard working solution with similar peak height to that of the sample solution.The responses of each compounds in the standard working solution and sample solution should be within the linear range of the instrumental detection.Under the above GC-MS operation condition,the retention time of heptachlor is 11.99 min,heptachlor epoxide A 13.92 min,and heptachlor epoxide B 13.85 min,the SIM chromatogram and full scan spectra of standard solution see Figure C.1 in Annex C.

7.3.5 Blank test

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

7.4 Calculation and expression of result

Calculation the content of the compound residues in the test sample by GC data processor or according to Formula(1):

$$X_i = \frac{c_i}{m} \times V \times \frac{1\,000}{1\,000} \qquad \cdots\cdots(1)$$

Where:

X_i —the residue contents of heptachlor, heptachlor epoxide A and heptachlor epoxide B in the test sample, mg/kg;

c_i —the concentration of heptachlor, heptachlor epoxide A and heptachlor epoxide B in the sample solution, μg/mL;

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

m —the mass of the test sample, g.

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

8 Limit of determination and recovery

8.1 Limits of determination

The limits of determination for heptachlor, heptachlor epoxide A and heptachlor epoxide B are 0.01 mg/kg.

8.2 Recoveries

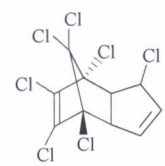
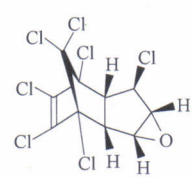
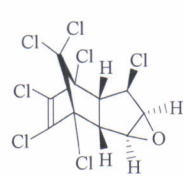
According to the experimental data, the fortifying concentrations of heptachlor, heptachlor epoxide A and heptachlor epoxide B and its corresponding recoveries are listed in Table 2.

Table 2 The corresponding recoveries of fortified concentrations(GC-ECD)

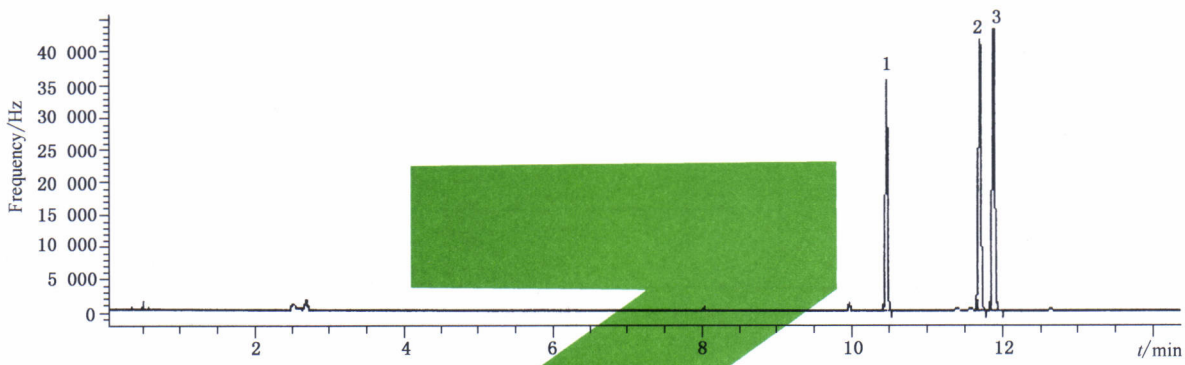
Name	Added/ (mg/kg)	Pork recoveries/%	Chicken recoveries/%	Beef recoveries/%	Ham sausage recoveries/%	Sausage recoveries/%
Heptachlor	0.01	100.9~109.7	90.3~108.1	99.5~109.0	103.7~109.5	91.3~98.2
	0.05	90.0~107.2	72.9~86.5	80.5~89.3	94.1~108.4	81.8~85.2
	0.10	86.4~109.6	89.9~109.5	91.2~100.0	104.1~109.9	80.22~93.4
Heptachlor epoxide A	0.01	95.0~107.0	81.0~98.4	84.8~105.2	91.6~105.1	104.4~109.5
	0.05	86.3~101.5	70.9~95.3	80.7~87.9	89.1~98.4	85.8~88.7
	0.10	80.6~102.5	80.4~97.2	84.5~87.5	88.3~102.5	81.2~91.9
Heptachlor epoxide B	0.01	100.7~109.1	99.4~108.7	101.9~107.8	98.6~108.6	90.2~100.0
	0.05	88.1~108.3	73.1~91.3	80.8~88.1	90.1~96.4	84.3~89.9
	0.10	87.6~98.5	81.2~101.1	85.2~89.7	88.9~100.7	80.5~89.9

Annex A
(Normative annex)
The information of pesticides

Table A.1 The information of pesticides

No.	Name	Formula	Molecular weight	CAS.No	Structure
1	Heptachlor	$C_{10}H_5Cl_7$	373.35	76-44-8	
2	Heptachlor-endo-epoxide (Isomer A)	$C_{10}H_5Cl_7O$	389.32	28044-83-9	
3	Heptachlor-exo-epoxide (Isomer B)	$C_{10}H_5Cl_7O$	389.32	1024-57-3	

Annex B
(Informative annex)
GC chromatogram of the standards



Instruction:

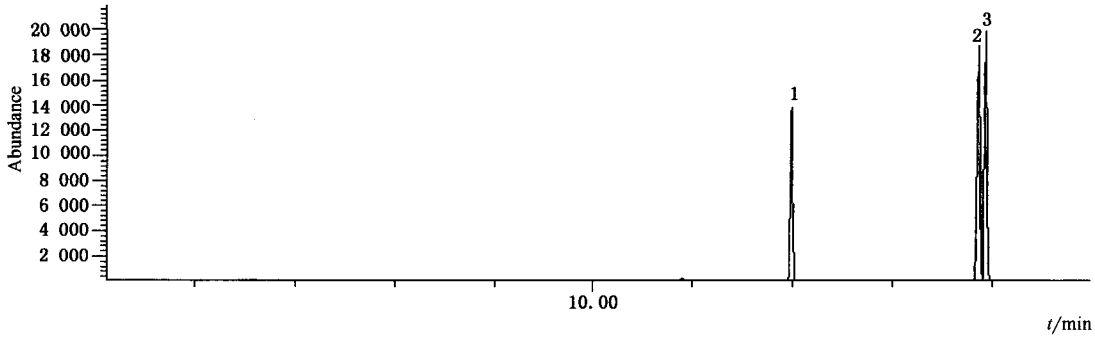
1—Heptachlor;

2—Heptachlor-exo-epoxide(Isomer B);

3—Heptachlor-endo-epoxide(Isomer A).

Figure B.1 Chromatogram of the pesticide standards

Annex C
(Informative annex)
GC-MS chromatogram of the standards



Instruction:

1—Heptachlor;

2—Heptachlor-exo-epoxide(Isomer B);

3—Heptachlor-endo-epoxide(Isomer A).

Figure C.1 SIM Chromatogram of the pesticide standards

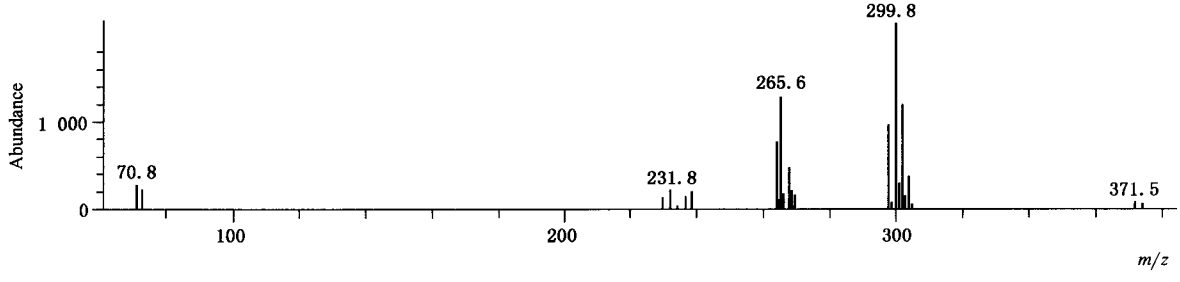


Figure C.2 Full scan of heptachlor standard

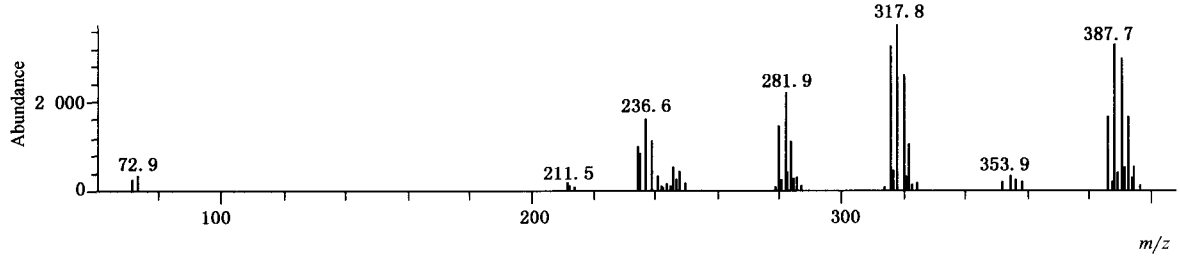


Figure C.3 Full scan of heptachlor-exo-epoxide(Isomer B) standard

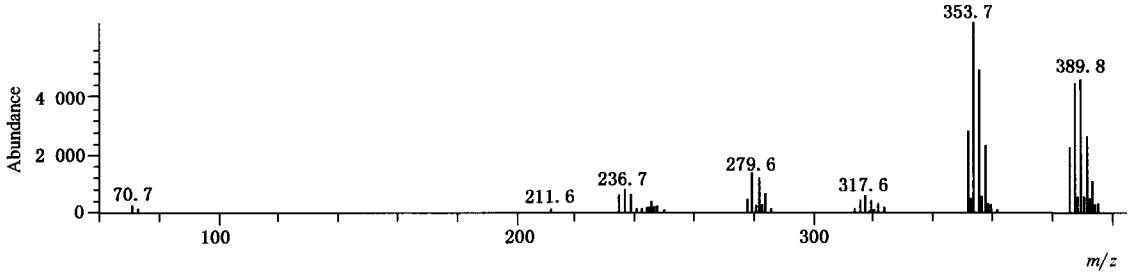


Figure C.4 Full scan of heptachlor-endo-epoxide(Isomer A) standards