SN

中华人民共和国出入境检验检疫行业标准

SN/T 0147—2016
代替 SN 0147—1992

出口茶叶中六六六、滴滴涕残留量的检测方法

Determination of BHC and DDT in tea for export

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国家质量监督检验检疫总局　发布

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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 0147—1992《出口茶叶中六六六、滴滴涕残留量检验方法》。与 SN 0147—1992 相比，除编辑性修改外，主要技术变化如下：

——修改了样品提取步骤；

——将样品测定填充色谱柱改成毛细管色谱柱；

——增加了气相色谱质谱确证法；

——略去了抽样步骤。

请注意本文件的某些内容可能涉及专利。本文件的发布机构不承担识别这些专利的责任。

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：中华人民共和国上海出入境检验检疫局。

本标准主要起草人：时逸吟、杨惠琴、曲栗、盛永刚、樊彦莉、伊雄海、朱坚。

本标准所代替标准的历次版本发布情况为：

——SN 0147—1992。

出口茶叶中六六六、滴滴涕残留量的检测方法

1 范围

本标准规定了出口茶叶中六六六、滴滴涕残留量的检测和确证方法。

本标准适用于出口茶叶中六六六、滴滴涕残留量的检测和确证。

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、滴滴涕用丙酮-正己烷超声提取，提取液经浓硫酸磺化，用带有电子捕获检测器气相色谱测定，外标法定量，如有必要用气相色谱-质谱联用仪进行确证。

4 试剂材料

除非另有说明，所用试剂均为分析纯，水为 GB/T 6682 规定的一级水。

4.1 丙酮：色谱纯。

4.2 正己烷：色谱纯。

4.3 浓硫酸：优级纯。

4.4 无水硫酸钠：于 650 ℃下灼烧 4 h，冷却贮于密闭容器中备用。

4.5 丙酮-正己烷(1+1，体积比)溶液：量取 500 mL 丙酮和 500 mL 正己烷至 1 000 mL 试剂瓶中。

4.6 2%硫酸钠溶液：称取 20 g 无水硫酸钠溶解，用水定容至 1 000 mL。

4.7 农药标准物质：α-六六六(α-BHC，CAS 号：319-84-6)；β-六六六(β-BHC，CAS 号：319-85-7)；γ-六六六(γ-BHC，CAS 号：58-89-9)；δ-六六六(δ-BHC，CAS 号：319-86-8)；p,p'-滴滴滴(p,p'-DDD，CAS 号：72-54-8)；p,p'-滴滴伊(p,p'-DDE，CAS 号：72-55-9)；o,p'-滴滴涕(o,p'-DDT，CAS 号：789-02-6)；p,p'-滴滴涕(p,p'-DDT，CAS 号：50-29-3)，纯度均大于 98%。

4.8 标准储备液的配制：分别称取约 0.01 g(精确至 0.000 1 g)α-六六六、β-六六六、γ-六六六、δ-六六六、p,p'-滴滴滴、p,p-滴滴伊、o,p'-滴滴涕和 p,p'-滴滴涕的标准品于 10 mL 的容量瓶中，用正己烷配制成约 1 000 μg/mL 的标准储备溶液，低于 5 ℃避光冷藏保存。

4.9 混合标准中间溶液的配制：各移取上述的标准储备液 1.00 mL 至 100 mL 容量瓶中，用正己烷稀释至刻度，现配现用。

5 仪器与设备

5.1 气相色谱仪：配有电子捕获(ECD)检测器。

5.2 气相色谱-质谱联用仪:配有负化学电离源(NCI)。
5.3 超声提取仪。
5.4 涡漩混匀器。
5.5 氮吹仪。
5.6 塑料离心管:50 mL。
5.7 玻璃离心管:50 mL,具塞。
5.8 低速离心机:3 000 r/min。
5.9 天平:感量 0.000 1 g,0.01 g 各一台。

6 试样的制备与保存

6.1 试样制备

将样品全部磨碎,混匀,均分成两份试样,装入洁净的容器内,密封,标明标记。

6.2 试样保存

试样于常温状态下保存。在制样的操作过程中,应防止样品受到污染或发生残留物含量的变化。

7 分析步骤

7.1 提取

称取 2 g 样品(精确至 0.01 g)于 50 mL 塑料离心管中,加入 10.0 mL 蒸馏水,超声 15 分钟。在离心管中加入 10.0 mL 丙酮-正己烷(1+1,体积比)混合液,涡旋混匀 2 min,在 3 000 r/min 下离心 10 min。取出上清液于 50 mL 玻璃离心管中。残渣中再加入 10.0 mL 丙酮-正己烷(1+1,体积比),同上操作,合并有机层提取液。

7.2 净化

在上述提取液中加入约 1 mL 的浓硫酸,盖上盖子,轻轻振荡后静置,3 000 r/min 离心 3 min,将上层有机相转移到另一玻璃离心管中,继续加入 1 mL 浓硫酸,同上操作 2~3 次,直至浓硫酸层变颜色变浅,弃去下层硫酸溶液。在有机相中加入 10 mL 2%硫酸钠溶液,涡旋混匀后 3 000 r/min 离心 3 min,上层有机相经无水硫酸钠脱水后于 40 ℃氮吹浓缩至干,准确加入 1.0 mL 正己烷溶液供 GC 测定。

7.3 测定

7.3.1 气相色谱参考条件

色谱测定条件如下:

a) 色谱柱:DB-5 石英毛细管柱,长 30 m,内径 0.32 mm,膜厚 0.25 μm,或相当者;
b) 柱温:初始温度 50 ℃,保持 0.5 min,以 20 ℃/min 升温至 140 ℃,再以 11 ℃/min 升温至 275 ℃,保持 20 min;
c) 进样口温度:280 ℃;
d) 检测器温度:300 ℃;
e) 载气:氮气,纯度大于等于 99.999%:恒流模式;流速:0.6 mL/min;
f) 进样量:1.0 μL;不分流进样,0.6 min 开阀;
g) 尾吹气:氮气,60 mL/min。

7.3.2 气相色谱-质谱参考条件

气相色谱-质谱参考条件如下：

a) 色谱柱：DB-5 石英毛细管柱，长 30 m，内径 0.32 mm，膜厚 0.25 μm，或相当者；

b) 柱温：初始温度 50 ℃，保持 0.5 min，以 20 ℃/min 升温至 140 ℃，再以 11 ℃/min 升温至 275 ℃，保持 20 min；

c) 载气：氦气，纯度大于等于 99.999%；恒流模式；流速：1.0 mL/min；

d) 进样口温度：280 ℃；

e) 离子源温度：150 ℃；

f) GC-MS 接口温度：280 ℃；

g) 进样量：1.0 μL，不分流进样；

h) 电离方式：负化学电离(NCI)，反应气：甲烷；

i) 测定方式：选择离子监测模式(SIM)，详见表 1。

表 1 六六六和 DDT 的监测离子及其丰度比

名称	监测离子(m/z)/amu	监测离子丰度比/%
α-六六六	181*，219，217，145	100∶90∶70∶20
β-六六六	181*，219，217，145	100∶90∶70∶20
γ-六六六	181*，219，217，145	100∶90∶70∶20
δ-六六六	181*，219，217，145	100∶90∶70∶20
p,p'-DDE	246*，318，235，165，237	100∶80∶2∶0.5∶0.5
p,p'-DDD	235*，237，165，318，246	100∶65∶40∶2∶1
o,p'-DDT	235*，237，165，246，318	100∶65∶40∶10∶1
p,p'-DDT	235*，237，165，246，318	100∶65∶40∶10∶1
* 表示定量离子。		

7.3.3 气相色谱测定

7.3.3.1 标准曲线：采用混合标准储备液，分别配制成 0.005 μg/mL、0.01 μg/mL、0.02 μg/mL、0.05 μg/mL、0.20 μg/mL、0.50 μg/mL 标准溶液，直接进气相色谱测定，峰面积对浓度作线性回归曲线，计算相关系数。

7.3.3.2 根据样液中被测物含量情况，选取浓度相近的标准工作液。标准工作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。标准工作溶液和样液等体积穿插进样测定。标准品的色谱图参见附录 A。

7.3.4 气相色谱-质谱确证

根据样液中被测组分含量，选定浓度相近的标准工作溶液。其响应值均应在仪器检测的线性范围内。对标准工作溶液与样液等体积参插进样测定，以色谱峰面积按外标法定量。在相同实验条件下，试样中待测物质的保留时间与标准工作溶液中对应的保留时间偏差在±2.5%之内；并且被测样品与标准品的质谱图相似，所选择的全部监测离子均出现且丰度比也相一致，其允许偏差不超过表 2 规定的范围时，则可确定为样品中存在这种药物残留。α-六六六、β-六六六、γ-六六六、δ-六六六、p,p'-DDD、p,p'-

DDE、o,p'-DDT 和 p,p'-DDT 的选择离子监测总离子流图和全扫描质谱图参见附录 B。

表 2　定性确证时相对离子丰度的最大允许偏差

相对离子丰度	>50%	>20%至 50%	>10%至 20%	≤10%
允许的相对偏差	±20%	±25%	±30%	±50%

7.4　空白试验

除不加试样外，均按上述操作步骤进行。

8　结果计算和表述

用色谱处理软件中的外标法绘制标准曲线，试样中六六六、滴滴涕各组分残留量分别按式(1)计算。计算结果需扣除空白值。

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

式中：

X ——样品中六六六、滴滴涕残留量，单位为毫克每千克(mg/kg)；

c ——从标准曲线中得到六六六、滴滴涕标准工作溶液的浓度，单位为微克每毫升(μg/mL)；

V ——样品定容体积，单位为毫升(mL)；

m ——样品称样量，单位为克(g)。

9　测定低限、回收率

9.1　测定低限

本方法对所测定的六六六、滴滴涕残留量的测定低限均为：0.005 mg/kg。

9.2　回收率

茶叶添加浓度及回收率试验数据详见表 3。

表 3　茶叶添加浓度及回收率试验数据

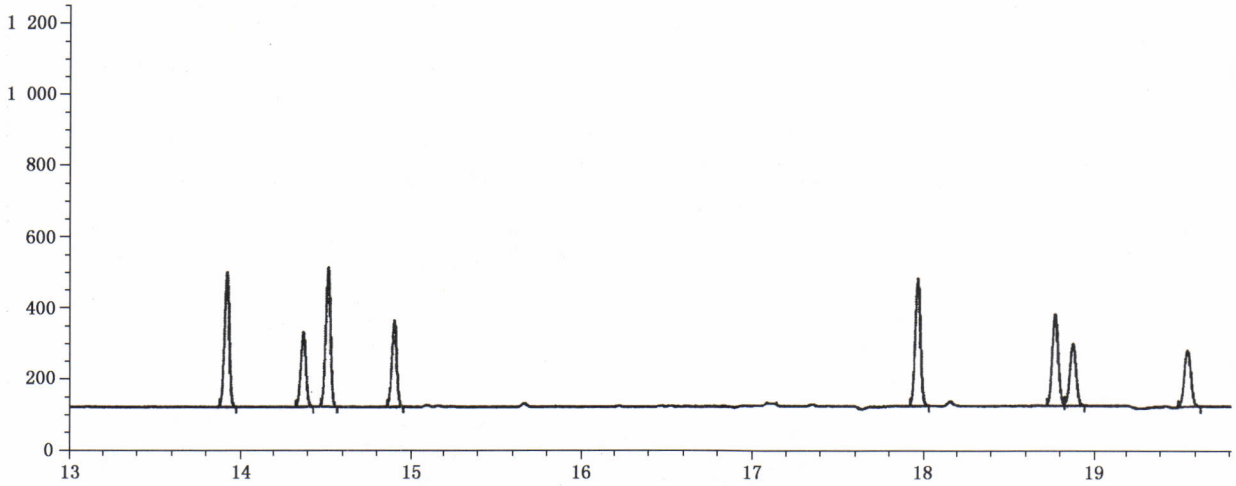
农药名称	添加浓度/(mg/kg)	红茶回收率/%	绿茶回收率/%	乌龙茶回收率/%
α-六六六	0.005	90.6～103	88.6～116	92.5～103
	0.010	84.0～100	89.6～104	89.8～102
	0.020	92.5～107	88.0～107	94.5～109
	0.200	89.5～97.0	88.0～101	93.0～107
β-六六六	0.005	94.8～104	88.8～113	93.2～102
	0.010	80.0～98.0	88.9～103	89.1～106
	0.020	85.5～105	88.5～102	94.5～109
	0.200	91.0～102	92.5～109	90.5～107
δ-六六六	0.005	93.2～102	89.8～112	94.6～103
	0.010	76.0～96.0	89.6～101	89.8～110
	0.020	89.5～105	89.5～106	90.5～107
	0.200	91.0～102	93.0～109	91.5～106

表 3（续）

农药名称	添加浓度/(mg/kg)	红茶回收率/%	绿茶回收率/%	乌龙茶回收率/%
γ-六六六	0.005	89.2～98.8	90.0～118	93.8～101
	0.010	80.0～94.0	87.9～110	89.8～106
	0.020	89.0～105	93.5～108	94.5～106
	0.200	93.0～104	90.5～105	88.0～106
p,*p*′-DDE	0.005	83.4～98.0	89.0～111	97.0～115
	0.010	84.0～96.0	87.9～110	90.6～103
	0.020	98.0～106	93.0～108	94.0～107
	0.200	88.0～107	90.5～105	91.0～106
p,*p*′-DDD	0.005	93.0～105	90.4～115	95.2～103
	0.010	80.0～96.0	89.9～103	96.9～113
	0.020	88.0～101	92.5～103	90.5～107
	0.200	90.5～100	91.5～104	94.5～106
o,*p*′-DDT	0.005	93.4～105	88.6～114	93.8～101
	0.010	78.0～100	88.8～100	90.1～106
	0.020	94.0～105	92.5～101	91.5～108
	0.200	92.0～103	90.0～104	91.5～106
p,*p*′-DDT	0.005	87.0～97.6	92.0～114	95.0～102
	0.010	80.0～94.0	90.1～107	89.9～106
	0.020	87.0～101	91.0～106	91.5～103
	0.200	89.0～101	91.5～103	90.5～103

附　录　A
（资料性附录）
六六六、DDT 标准品的气相色谱图

图 A.1 给出了六六六、DDT 标准品的气相色谱图。

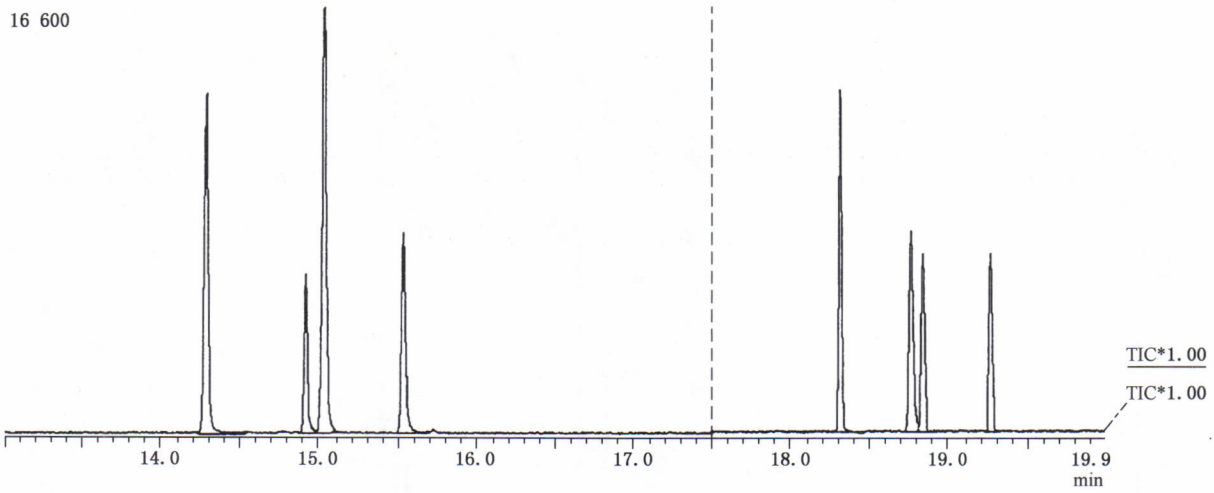


说明：图中曲线依次为 α-六六六、β-六六六、δ-六六六、γ-六六六、*p*，*p*′-DDE、*p*，*p*′-DDD、*o*，*p*′-DDT 和 *p*，*p*′-DDT。

图 A.1　六六六、DDT 标准品的气相色谱图（0.005 μg/mL）

附　录　B
（资料性附录）
六六六、DDT 标准品气相色谱-质谱图

图 B.1～图 B.9 给出了六六六、DDT 标准品气相色谱-质谱图。



说明：图中曲线依次为 α-六六六、β-六六六、δ-六六六、γ-六六六、*p*，*p*′-DDE、*p*，*p*′-DDD、*o*，*p*′-DDT 和 *p*，*p*′-DDT。

图 B.1　六六六、DDT 标准品标准品选择离子监测总离子流图（0.20 μg/mL）

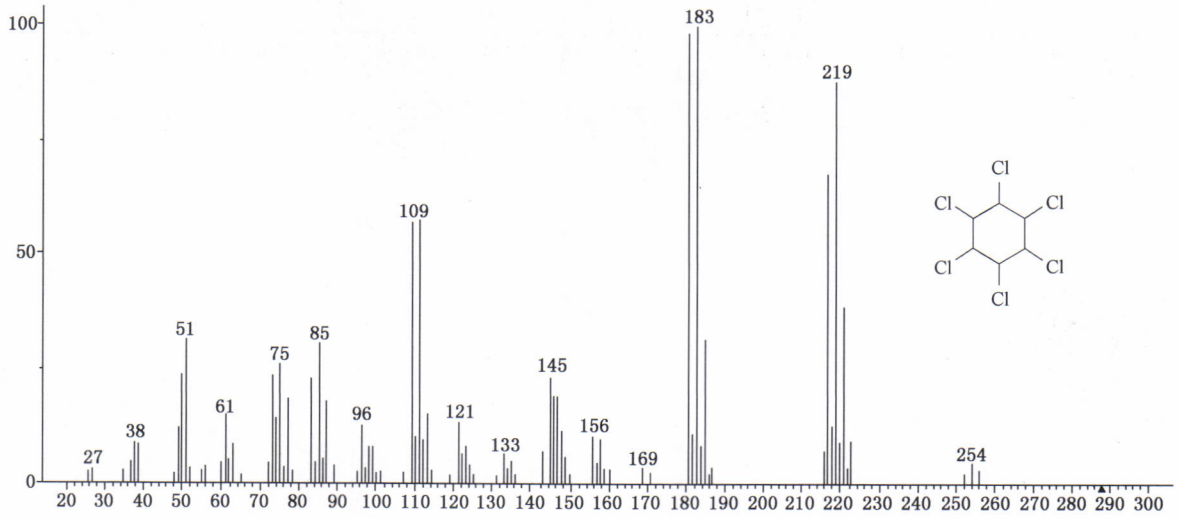


图 B.2　α-六六六全扫描质谱图

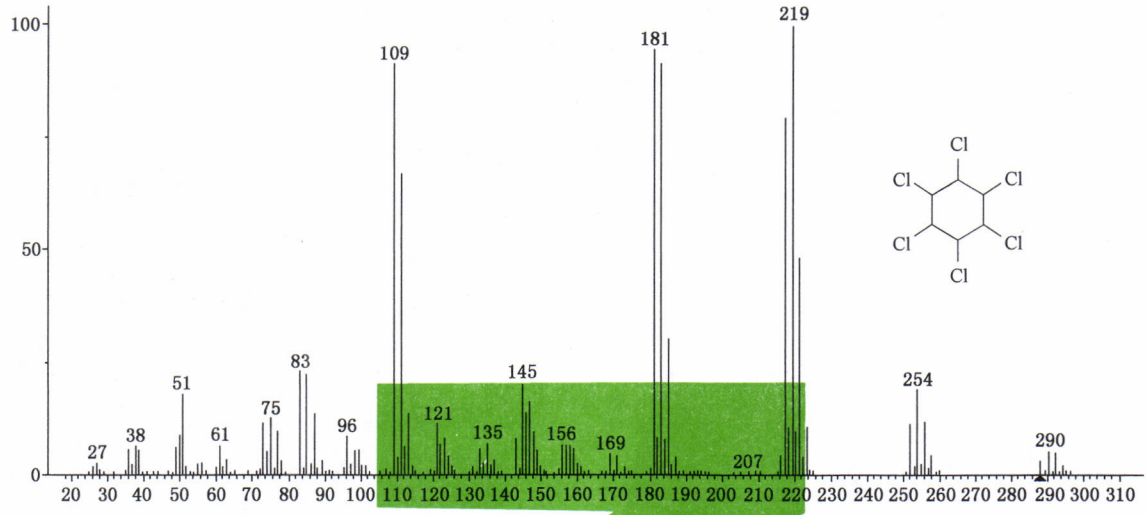


图 B.3 *β*-六六六全扫描质谱图

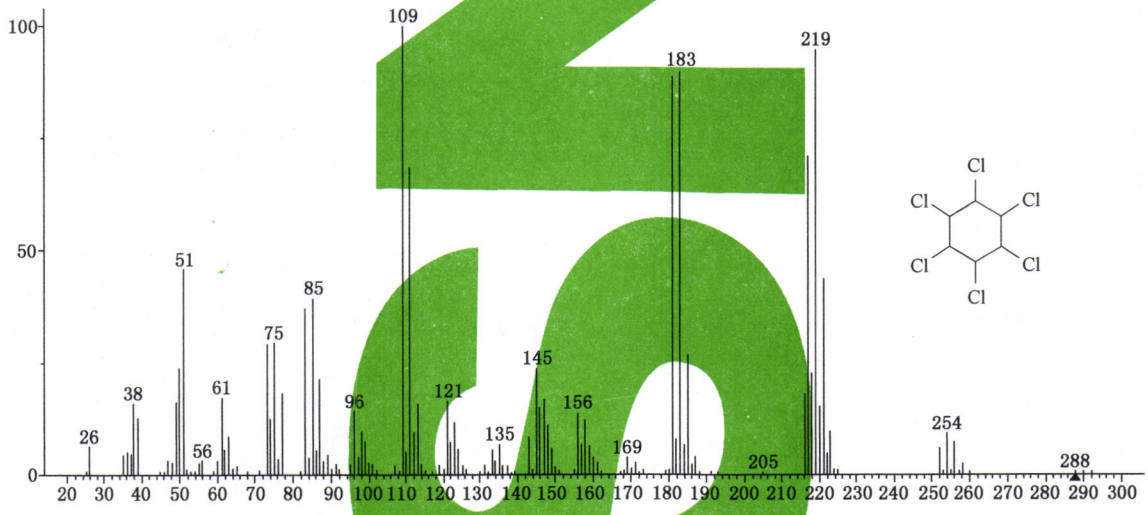


图 B.4 *δ*-六六六全扫描质谱图

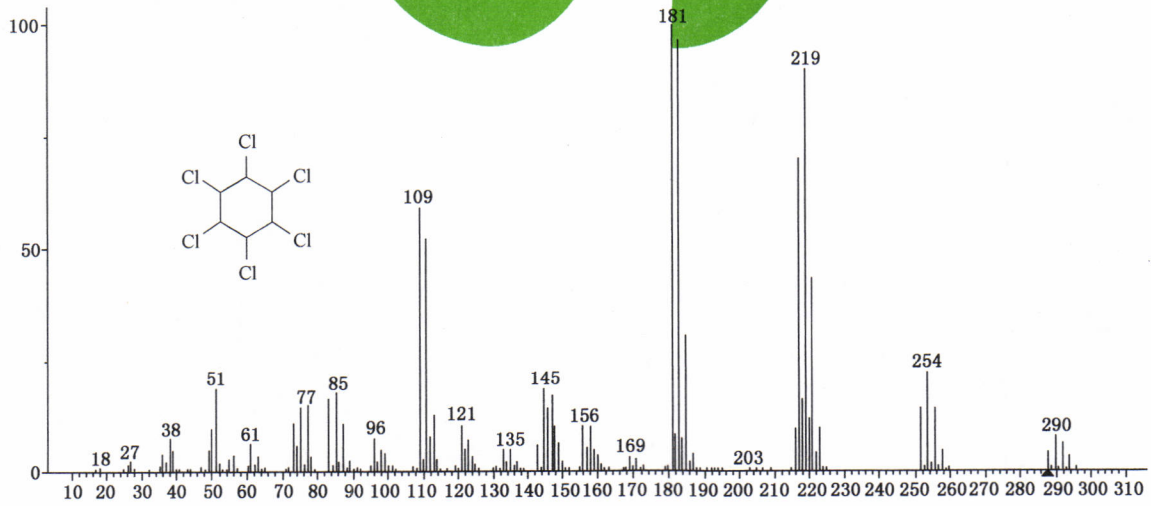


图 B.5 *γ*-六六六全扫描质谱图

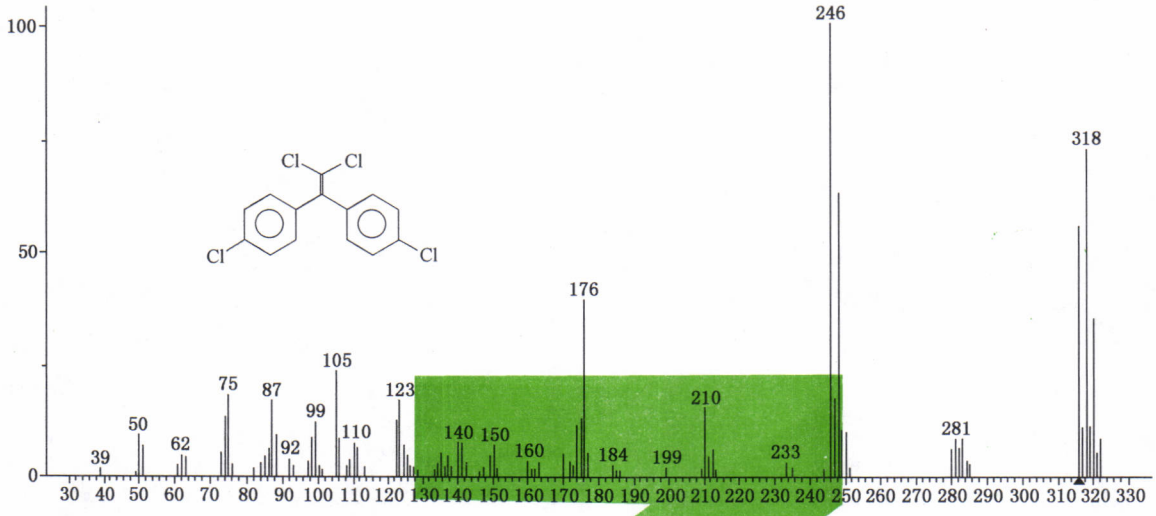


图 B.6　*p*,*p*′-DDE 全扫描质谱图

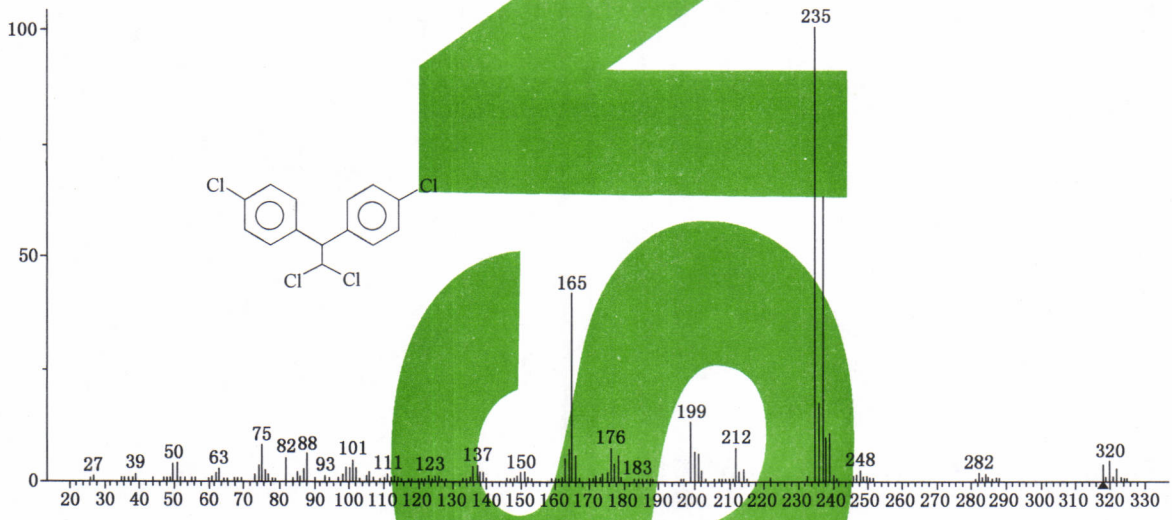


图 B.7　*p*,*p*′-DDD 全扫描质谱图

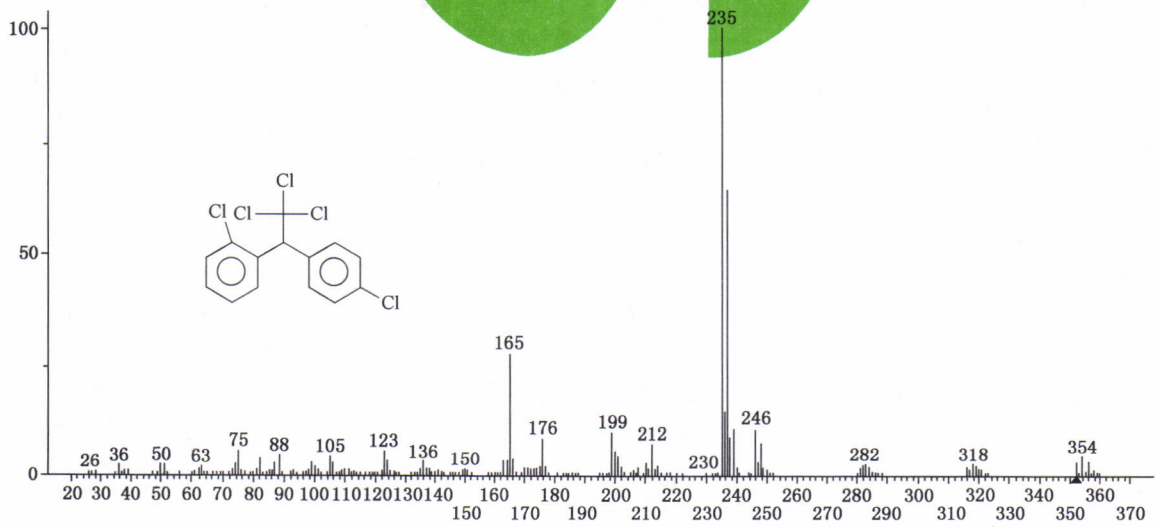


图 B.8　*o*,*p*′-DDT 全扫描质谱图

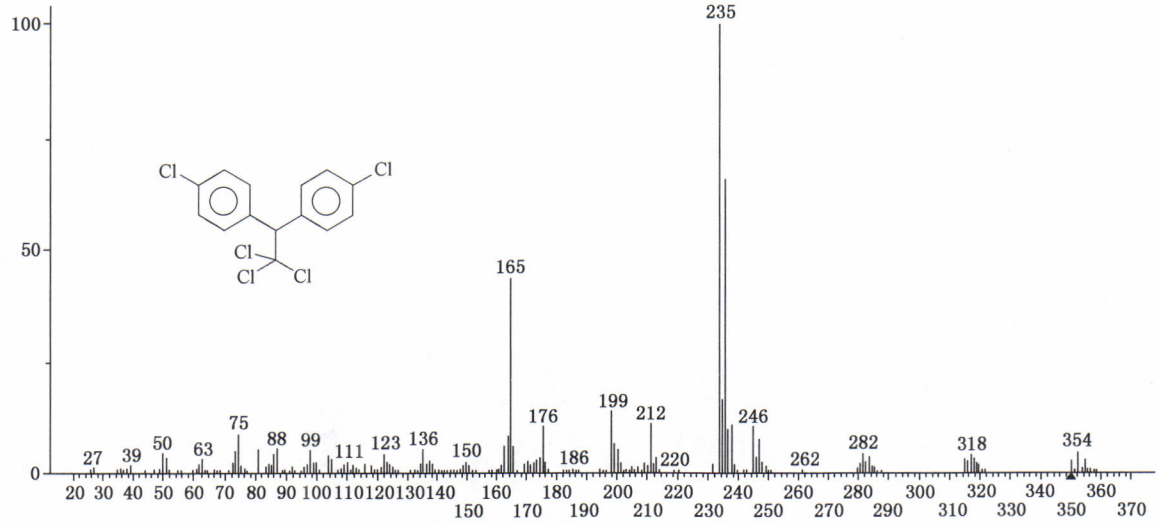


图 B.9　*p*，*p*′-DDT 全扫描质谱图

Foreword

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

SN 0147—1992 (Method for determination of BHC and DDT residues in tea for export) is replaced with this standard.

—Compared with SN 0147—1992, the modifications are below:

—modified method for the sample pretreatments.

—changed the packing column to capillary column.

—added the ascertainment experiments by GC-MS.

—skipped the sampling procedure.

Some parts of the standard may have relationship with some patents. The release department has no responsibility to recognize these patents.

This standard was proposed by and is under the charge of the Certification and Accreditation Administration of the People's Republic of China.

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

This standard was mainly drafted by Shi yiyin, Yang huiqin, Qu li, Sheng yonggang, Fan yanli, Yi xionghai, Zhu jian.

This standard replaced the following standards:

—SN 0147—1992.

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

Determination of BHC and DDT in tea for export

1 Scope

The standard specifies the method of determination and confirmation BHC and DDT residues in tea. This standard is applicable to the determination and confirmation of BHC and DDT residues in tea.

2 Normative references

The following document 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 is referred for application.GB/T 6682 analyzes the water-using specification in lab and testing methods.

3 Principle

BHC and DDT residues are extracted with acetone and hexane, and then cleaned up by sulfonane method. The residual content is determined by GC with ECD detector and quantified by external standard.

4 Reagents and materials

Unless otherwise specified, all the reagents used should be analytical grade,"water"is distilled water described by GB/T 6682.

4.1 Acetone: HPLC grade.

4.2 N-hexane: HPLC grade.

4.3 Concentrated sulfuric acid: Guaranteed reagent.

4.4 Anhydrous sodium sulfate: ignite at 650 ℃ for 4 h, then cool to room temperature and keep in a tightly closed container.

4.5 Acetone-hexane (1+1,v/v): combine 500 mL acetone and 500 mL n-hexane.

4.6 2% Sodium sulfate solution: weigh 20 g sodium sulfate, dissolve and add water to 1 000 mL.

4.7 Standard substance:

α-BHC: CAS code 319-84-6, formula: $C_6H_6Cl_6$.

β-BHC: CAS code 319-85-7, formula: $C_6H_6Cl_6$.

δ-BHC: CAS code 319-86-8, formula: $C_6H_6Cl_6$.

γ-BHC: CAS code 58-89-9, formula: $C_6H_6Cl_6$.

p,*p*′-DDE: CAS code 72-55-9, formula: $C_{14}H_8Cl_4$.

p,*p*′-DDD: CAS code 72-54-8, formula: $C_{14}H_{10}Cl_4$.

o,*p*′-DDT: CAS code 789-02-6, formula: $C_{14}H_9Cl_5$.

p,*p*′-DDT: CAS code 50-29-3, formula: $C_{14}H_9Cl_5$.

4.8 Stock standard solution: accurately weigh 0.01 ± 0.000 1 g of α-BHC, β-BHC, δ-BHC, γ-BHC, *p*,*p*′-DDE, *p*,*p*′-DDD, *o*,*p*′-DDT and *p*,*p*′-DDT standard into a 10 mL volumetric flask and dilute to the volume with acetone. Mix well. These solutions are stock solutions of 1 000 μg/mL in hexane, and can be used for one year when stored under 5 ℃.

4.9 Intermediate standard solution: dilute the stock standard solution with hexane to 10.0 μg/mL when necessary.

5 Apparatus and equipment

5.1 GC with ECD.

5.2 GC-MS with NCI.

5.3 Ultrasonic extractor.

5.4 Vortex mixer.

5.5 Nitrogen evaporator.

5.6 Polypropylene centrifuge tube: 50 mL with graduation.

5.7 Glass centrifuge tube: 50 mL with graduation.

5.8 Low speed centrifuge: 3 000 r/min.

5.9 Balance: scale 0.000 1 g and 0.001 g.

6 Preparation and storage of test sample

6.1 Preparation of test samples

Smash the representative sample thoroughly by a crusher. Mix thoroughly. Put in clean containers. Seal and label them.

6.2 Storage of test samples

The test samples should be stored at room temperature.

While sampling and preparing sample, please avoid contamination or any factors that may change residue content.

7 Procedure

7.1 Extraction

Weigh 2 g (±0.01g) of the sample into a 50 mL polypropylene centrifuge tube, add 10.0 mL of distilled water mixed, extracted for 15 min in the ultrasonic extraction instrument. Add 10.0 mL of acetone-hexane (1+1, v/v) into the polypropylene centrifuge tube, blend for 2 min with vortex mixer, centrifuge for 10 min at 3 000 r/min. Transfer the supernatant to a new glass centrifuge tube, extract the residue with 10.0 mL acetone-hexane (1+1, v/v) once more, and combine the organic supernatant.

7.2 Clean up

1 mL concentrated sulfuric acid was added into the supernatant, sealed with gap, shaking gently and then centrifuged at 3 000 r/min for 3 min. Then transfer the upper organic layer to another glass centrifuge tube, add 1 mL concentrated sulfuric acid, repeat the sulfonation for 2～3 times until the lower layer turns to be clear. Discard the lower inorganic layer, and add 10 mL 2% sodium sulfate solution, shaking gently and then centrifuged at 3 000 r/min for 3 min. Let the upper layer through the anhydrous sodium sulfate and evaporate to dryness with gentle nitrogen evaporator at 40 ℃. Add exactly 1.0 mL hexane to dissolve the residue and for GC determination.

7.3 Determination

7.3.1 GC condition

GC condition is as follows:

a) Colum (analysis): DB-5 30 m × 0.32 mm(i,d) × 0.25 μm(film thickness), or equivalent.

b) Column temperature: 50 ℃ (0.5 min)—20 ℃/min—140 ℃ —11℃/min—275 ℃ (20 min).

c) Injection port temperature: 280 ℃.

d) GC temperature:300 ℃.

e) Carrier gas: Nitrogen, purity≥99.999%, Constant flow, Flow rate: 12 cm/sec.

f) Injection volume: 1.0 μL, Splitless, Open the valve at 0.6 min.

g) Make up gas: Nitrogen, 60 mL/min.

7.3.2 GC-MS condition

GC-MS condition is as follows:

a) Colum (analysis):DB-5 30 m × 0.32 mm(i,d) × 0.25 μm (film thickness), or equivalent.

b) Column temperature:50 ℃ (0.5 min)—20 ℃/min—140℃—11 ℃/min—275 ℃ (20 min).

c) Carrier gas: Helium, purity≥99.999%, Constant flow, Flow rate: 1.0 mL/sec.

d) Injection port temperature: 280 ℃.

e) Ion source temperature: 150 ℃.

f) GC-MS interface temperature: 280 ℃.

g) Injection volume: 1.0 μL, splitless, open the valve at 0.6 min.

h) Electron ionization mode: NCI, reacting gas:methane.

i) Selected monitoring ion (m/z): Table 1.

Table 1 Monitor ions and relative intensity for α-BHC, β-BHC, δ-BHC, γ-BHC, p, p′-DDE, p, p′-DDD, o, p′-DDT and p, p′-DDT.

Name of pesticides	Monitor ion(m/z)/amu	Relative intensity/%
α-BHC	181*, 219, 217, 145	100 : 90 : 70 : 20
β-BHC	181*, 219, 217, 145	100 : 90 : 70 : 20
γ-BHC	181*, 219, 217, 145	100 : 90 : 70 : 20
δ-BHC	181*, 219, 217, 145	100 : 90 : 70 : 20
p, p′-DDE	246*, 318, 235, 165, 237	100 : 80 : 2 : 0.5 : 0.5
p, p′-DDD	235*, 237, 165, 318, 246	100 : 65 : 40 : 12 : 1
o, p′-DDT	235*, 237, 165, 246, 318	100 : 65 : 40 : 10 : 1
p, p′-DDT	235*, 237, 165, 246, 318	100 : 65 : 40 : 10 : 1
* stands for quantitation ion		

7.3.3 GC determination

7.3.3.1 Standard curves: prepare mixed standard solution, whose concentrations are 0.005 μg/mL, 0.01 μg/mL, 0.02 μg/mL, 0.05 μg/mL, 0.20 μg/mL, 0.50 μg/mL. Establishing the standard curves by peak areas on GC responses against the concentrations, and then calculating the correlation coefficients.

7.3.3.2 Use the standard working solutions based on estimating the concentration of analyses in the sample. The responses of analyses in the standard working solution and sample solutions should be within 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 operation condition, the chromatograms of BHC and DDT are in Annex A.

7.3.4 GC-MS determination

Determination the standard working solutions and sample solutions based on condition above. If the retention time of the analyses are the same to that of the external standards when subtraction the background, in the same time the ion relative abundance of the compounds from samples are similar to that of the standards' at the deviation as Table 2, it can be inferred that is positive sample. The TIC and SCAN chromatograms of α-BHC, β-BHC, δ-BHC, γ-BHC, p, p′-DDE, p, p′-DDD, o, p′-DDT and p, p′-DDT are in Annex B.

Table 2 Maximum permitted tolerances for relative ion intensities while confirmation.

Relative intensity	>50%	>20% to 50%	>10% to 20%	≤10%
Permitted tolerances	±20%	±25%	±30%	±50%

7.4 Blank test

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

8 Calculation and expression of result

According to the external calibration curve from the chromatograph software, calculate the content of BHC and DDT residue in the test sample according to the formula (1). The blank value should be subtracted from the result of calculation above.

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

where:

X —the residue content of BHC and DDT in the test sample, mg/kg;

c —the concentration of BHC and DDT in the standard working solution, μg/mL;

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

m —the corresponding mass of test sample in the final sample solution, g.

9 Limit of quantitation (LOQ) and recovery

9.1 Limit of quantitation

The limit of quantitation (LOQ) of the method is 0.005 mg/kg.

9.2 Recovery

Spike levels of tea and recovery data are listed in Table 3.

Table 3 Recovery range of α-BHC, β-BHC, δ-BHC, γ-BHC, *p*, *p*′-DDE, *p*, *p*′-DDD, *o*, *p*′-DDT and *p*, *p*′-DDT in black tea, green tea and woloong tea by GC method.

Name	Spike level	Recovery of black tea/%	Recovery of black tea/%	Recovery of woloong tea/%
α-BHC	0.005	90.6～103	88.6～116	92.5～103
	0.010	84.0～100	89.6～104	89.8～102
	0.020	92.5～107	88.0～107	94.5～109
	0.200	89.5～97.0	88.0～101	93.0～107
β-BHC	0.005	94.8～104	88.8～113	93.2～102
	0.010	80.0～98.0	88.9～103	89.1～106
	0.020	85.5～105	88.5～102	94.5～109
	0.200	91.0～102	92.5～109	90.5～107
δ-BHC	0.005	93.2～102	89.8～112	94.6～103
	0.010	76.0～96.0	89.6～101	89.8～110
	0.020	89.5～105	89.5～106	90.5～107
	0.200	91.0～102	93.0～109	91.5～106
γ-BHC	0.005	89.2～98.8	90.0～118	93.8～101
	0.010	80.0～94.0	87.9～110	89.8～106
	0.020	89.0～105	93.5～108	94.5～106
	0.200	93.0～104	90.5～105	88.0～106
p, *p*′-DDE	0.005	83.4～98.0	89.0～111	97.0～115
	0.010	84.0～96.0	87.9～110	90.6～103
	0.020	98.0～106	93.0～108	94.0～107
	0.200	88.0～107	90.5～105	91.0～106
p, *p*′-DDD	0.005	93.0～105	90.4～115	95.2～103
	0.010	80.0～96.0	89.9～103	96.9～113
	0.020	88.0～101	92.5～103	90.5～107
	0.200	90.5～100	91.5～104	94.5～106
o, *p*′-DDT	0.005	93.4～105	88.6～114	93.8～101
	0.010	78.0～100	88.8～100	90.1～106
	0.020	94.0～105	92.5～101	91.5～108
	0.200	92.0～103	90.0～104	91.5～106
p, *p*′-DDT	0.005	87.0～97.6	92.0～114	95.0～102
	0.010	80.0～94.0	90.1～107	89.9～106
	0.020	87.0～101	91.0～106	91.5～103
	0.200	89.0～101	91.5～103	90.5～103

Annex A
(informative annex)
GC chromatogram of BHC and DDT standard

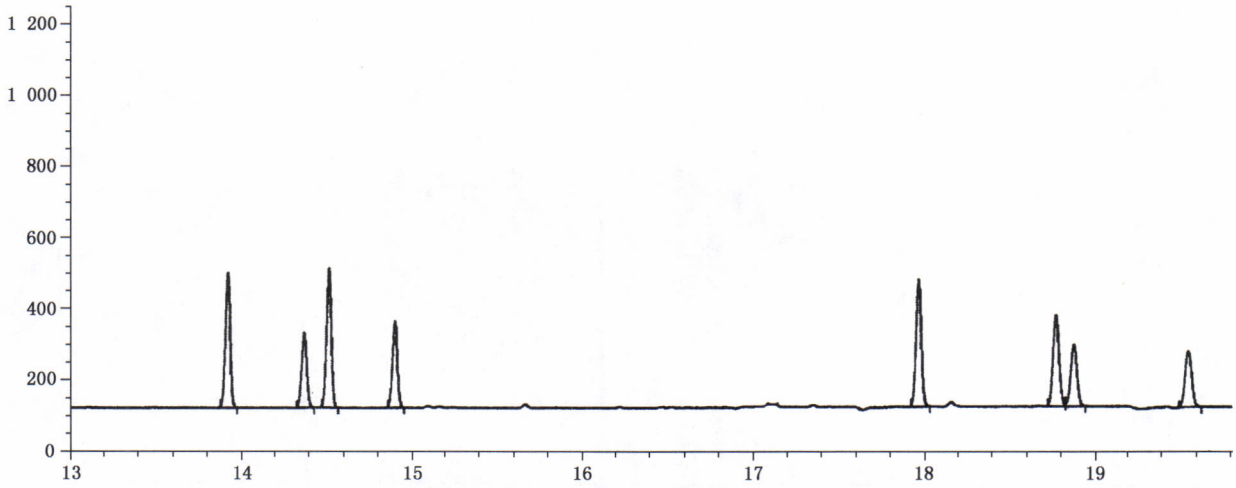


Figure A.1 GC chromatogram of BHC and DDT standard(0.005 μg/mL)

Followed by α-BHC、β-BHC、δ-BHC、γ-BHC、Heptachlor、*p*,*p*′-DDE、*p*,*p*′-DDD、*o*,*p*′-DDT and *p*,*p*′-DDT

Annex B
(Informative annex)
TIC and SCAN chromatograms of BHC and DDT

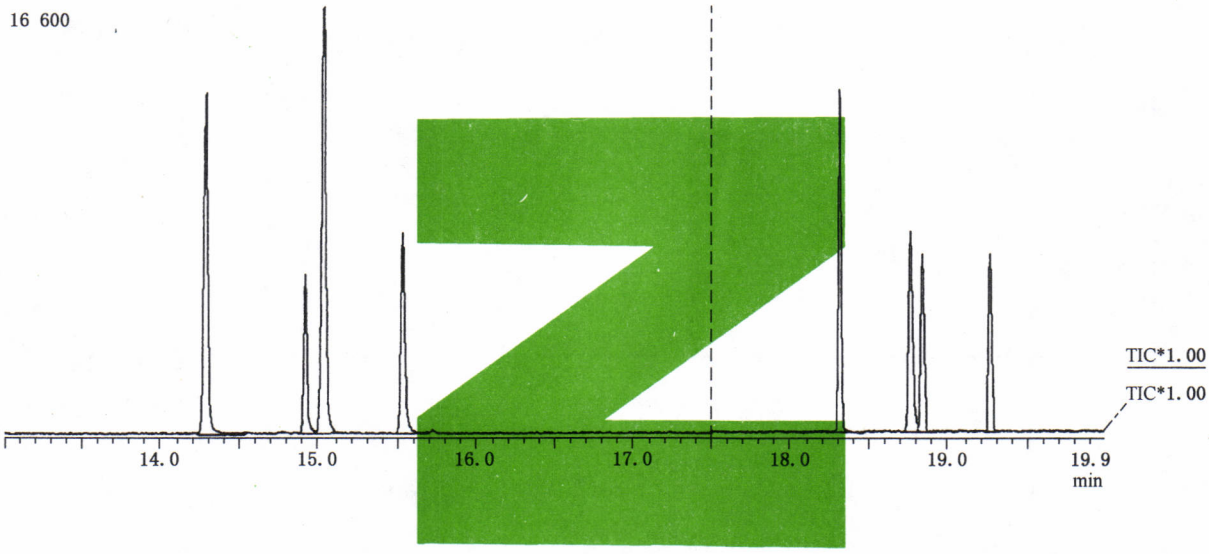


Figure B.1 TIC of BHC and DDT standard on SIM mode(0.20 μg/mL)

Followed by α-BHC、β-BHC, δ-BHC, γ-BHC, Heptachlor, *p*, *p*′-DDE、*p*, *p*′-DDD、*o*, *p*′-DDT and *p*, *p*′-DDT

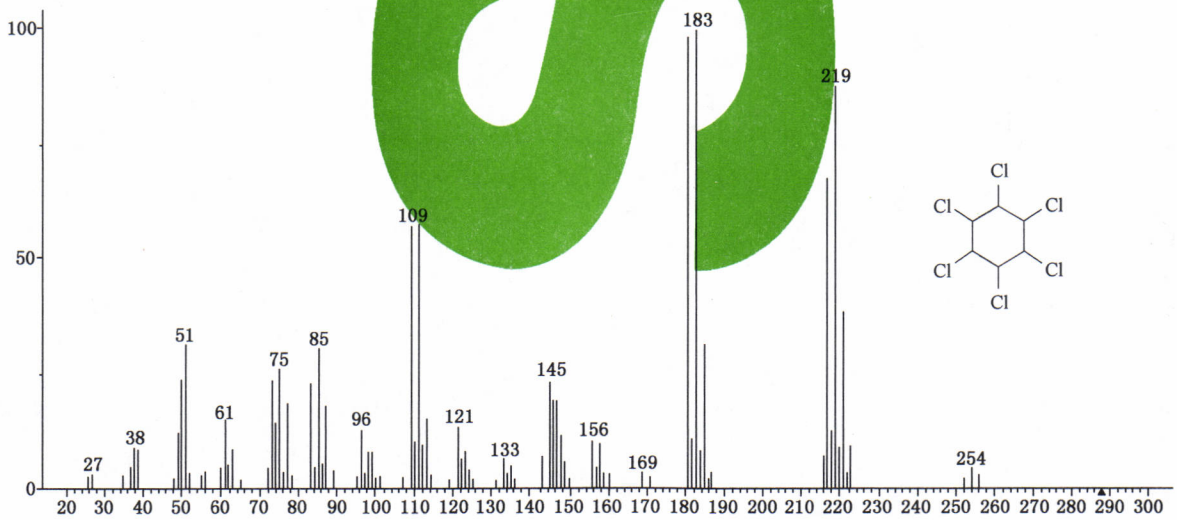


Figure B.2 SCAN chromatogram of α-BHC

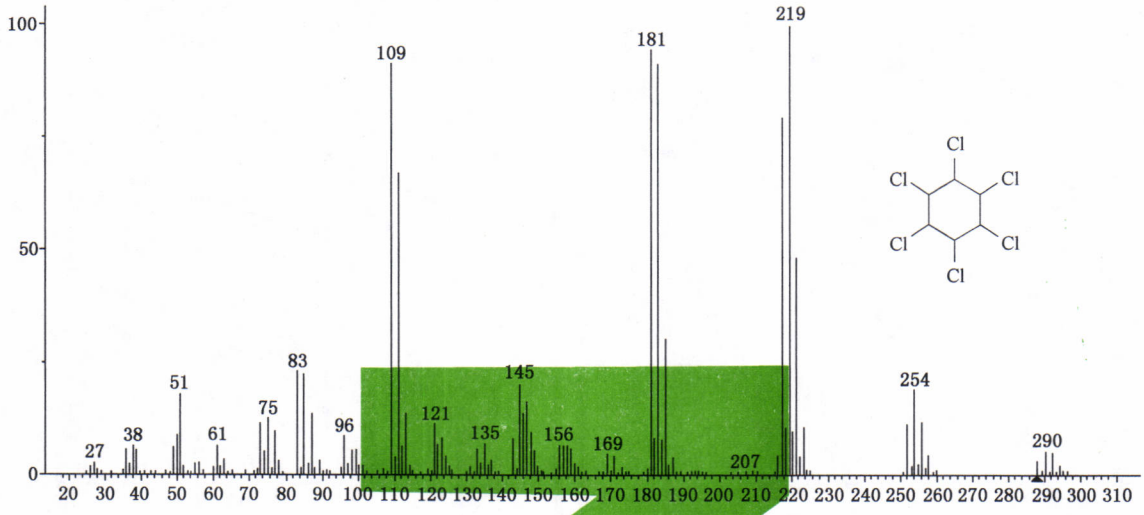


Figure B.3 SCAN chromatogram of β-BHC

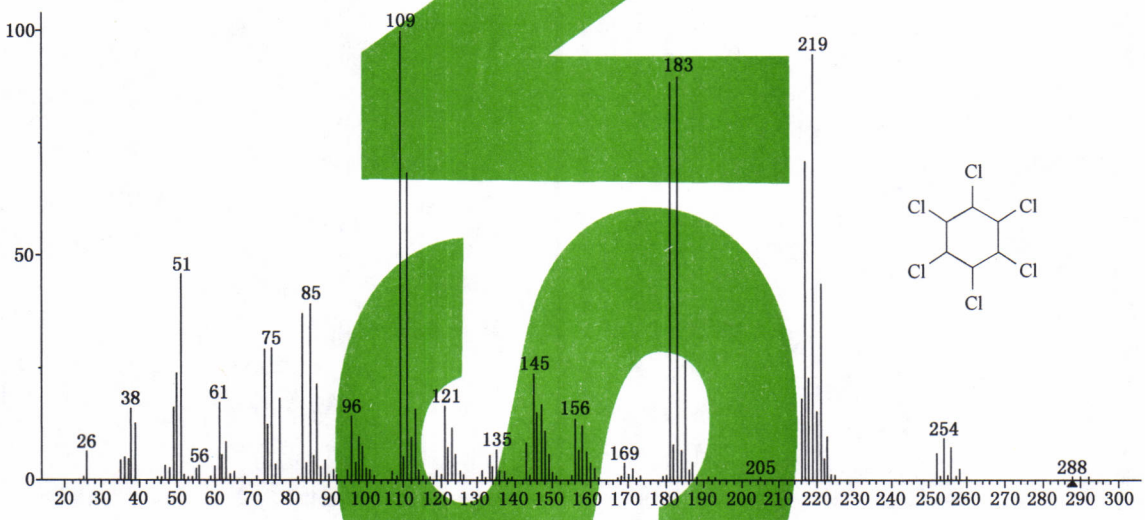


Figure B.4 SCAN chromatogram of δ-BHC

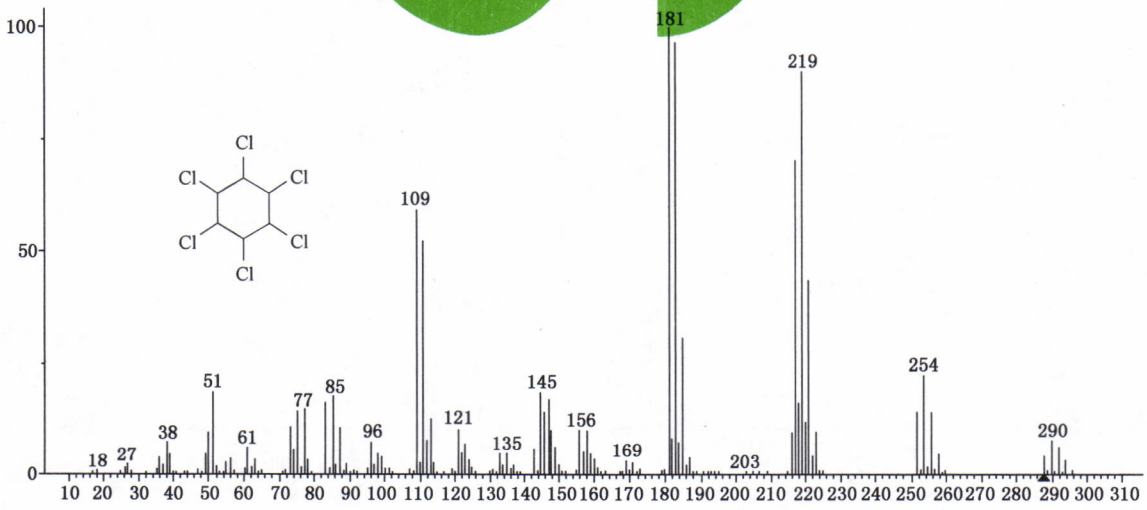


Figure B.5 SCAN chromatogram of γ-BHC

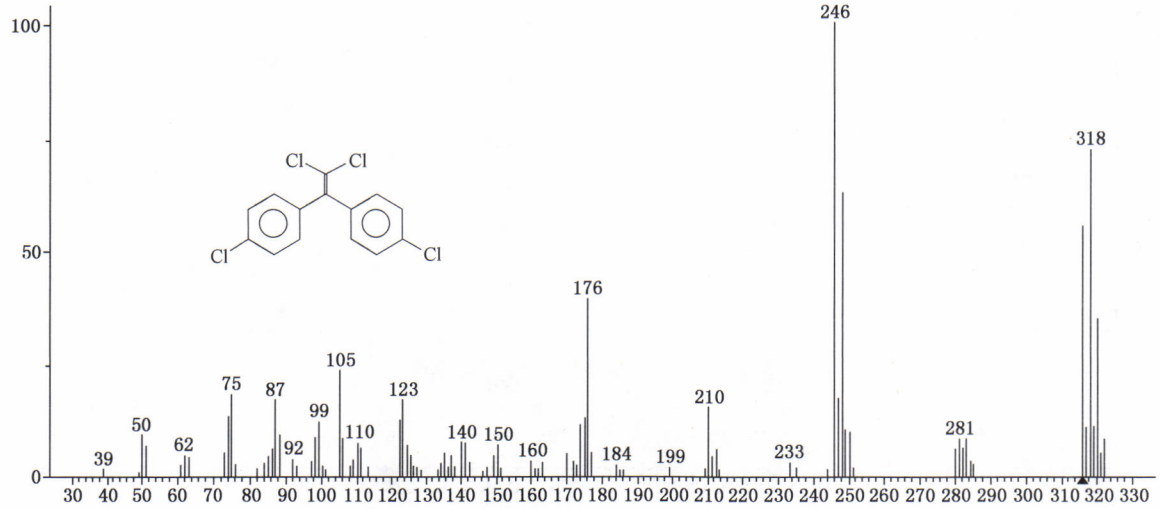


Figure B.6 SCAN chromatogram of *p*, *p*′-DDE

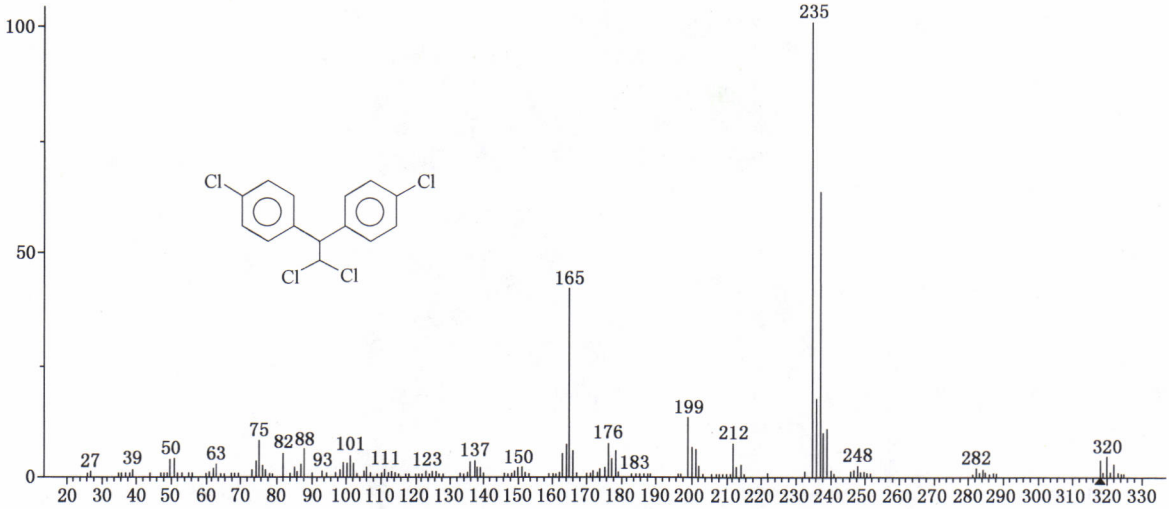


Figure B.7 SCAN chromatogram of *p*, *p*′-DDD

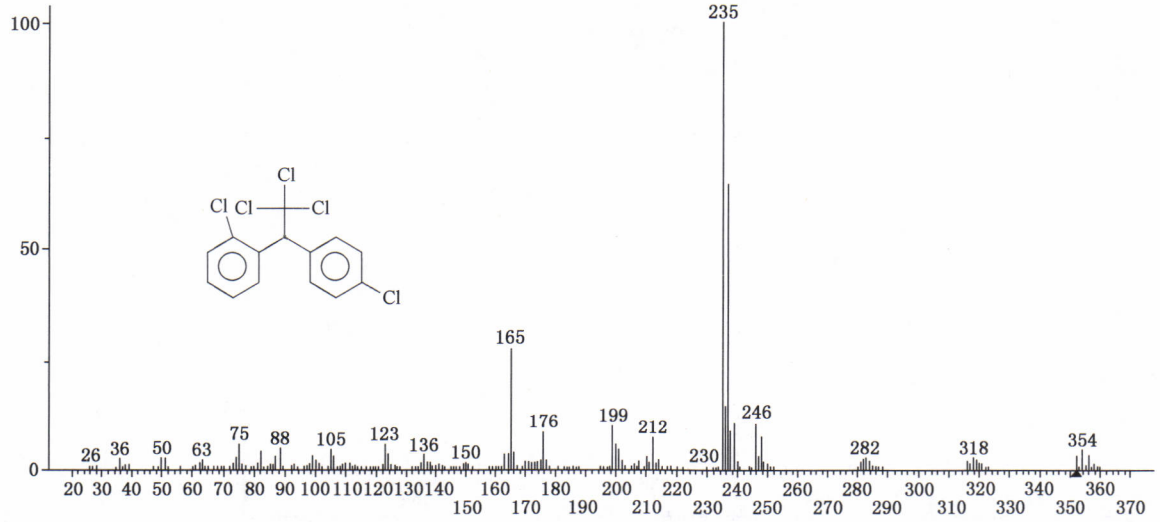


Figure B.8 SCAN chromatogram of *o*, *p*′-DDT

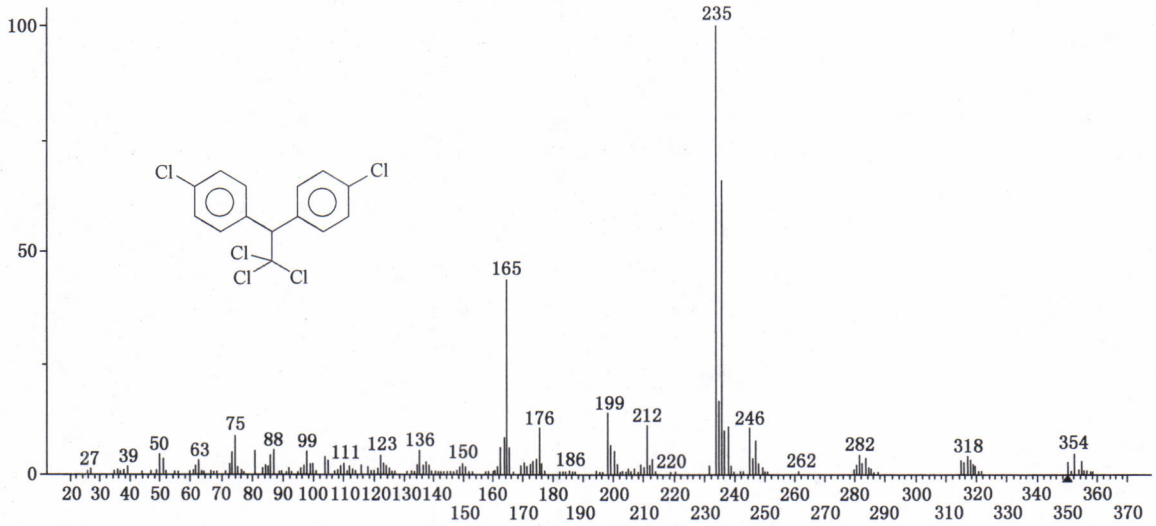


Figure B.9 SCAN chromatogram of *p*, *p*′-DDT

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