SN

中华人民共和国出入境检验检疫行业标准

SN/T 0127—2011
代替 SN 0127—1992、SN 0128—1992、SN 0129—1992、SN 0130—1992

进出口动物源性食品中六六六、滴滴涕和六氯苯残留量的检测方法 气相色谱-质谱法

Determination of BHCs, DDTs and HCB residues in foods of animal origin for import and export—GC-MS 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 0127—1992《出口乳及乳制品中六六六、滴滴涕残留量检验方法》、SN 0128—1992《出口蛋及蛋制品中六六六、滴滴涕的残留量检验方法》、SN 0129—1992《出口水产品中六六六、滴滴涕残留量的检验方法》、SN 0130—1992《出口蜂产品中六六六、滴滴涕残留量检验方法》。

本标准整合 SN/T 0127、SN/T 0128、SN/T 0129 和 SN/T 0130 等行业标准。

本标准与 SN 0127—1992、SN 0128—1992、SN 0129—1992 和 SN 0130—1992 相比，主要技术变化如下：

——扩大标准检测的适用基质范围；

——改进了样品前处理技术；

——用气相色谱-质谱法替代气相色谱法，并改用负化学电离(GC-MS/NCI)技术确证。

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：中华人民共和国江苏出入境检验检疫局。

本标准主要起草人：沈崇钰、沈伟健、赵增运、柳菡、吴斌、余可垚、桂茜雯、龚玉霞。

本标准所代替标准的历次版本发布情况为：

——SN 0127—1992；

——SN 0128—1992；

——SN 0129—1992；

——SN 0130—1992。

进出口动物源性食品中六六六、滴滴涕和六氯苯残留量的检测方法 气相色谱-质谱法

1 范围

本标准规定了鸡蛋、牛奶、芝士粉、鸡肝、鸡腿肉、牛肉、鲴鱼、河虾、蜂王浆和蜂蜜等动物源性食品中六六六、滴滴涕和六氯苯残留量的气相色谱-质谱检测方法。

本标准适用于鸡蛋、牛奶、芝士粉、鸡肝、鸡腿肉、牛肉、鲴鱼、河虾、蜂王浆和蜂蜜等食品中六六六、滴滴涕和六氯苯残留量的测定和确证。

2 方法提要

试样经正己烷或丙酮溶剂提取，磺化法净化，气相色谱-负化学离子源质谱法进行测定与确证，外标法定量。

3 试剂和材料

除另有规定外，所用试剂均为分析纯，水为蒸馏水。

3.1 正己烷：色谱纯。

3.2 丙酮：色谱纯。

3.3 浓硫酸。

3.4 无水硫酸钠：经 650 ℃灼烧 4 h，置于密闭容器中备用。

3.5 氯化钠。

3.6 硫酸钠水溶液(20 g/L)：将 20 g 无水硫酸钠溶于 1 000 mL 蒸馏水中。

3.7 农药标准品：六六六(α-BHC，CAS 编号：319-84-6；β-BHC，CAS 编号：319-85-7；γ-BHC，CAS 编号：58-89-9；δ-BHC，CAS 编号：319-86-8)、滴滴涕(*p*，*p*′-滴滴滴，*p*，*p*′-DDD，CAS 编号：1022-22-6；*p*，*p*′-滴滴伊，*p*，*p*′-DDE，CAS 编号：72-55-9；*o*，*p*′-滴滴涕，*o*，*p*′-DDT，CAS 编号：789-02-6 和 *p*，*p*′-滴滴涕，*p*，*p*′-DDT，CAS 编号：50-29-3)和六氯苯(hexachlorobenzene，CAS 编号：118-74-1)标准物质：纯度大于等于 98.0%。

3.8 各农药标准储备溶液：分别准确称取适量的农药标准品，用丙酮稀释配制成 100 μg/mL 的标准储备液，4 ℃下保存(有效期为 6 个月)。

3.9 农药混合标准工作液：根据需要，分别量取上述各标准储备液于同一容量瓶中，用正己烷稀释到刻度，配制成适当浓度的标准工作溶液，4 ℃下保存(有效期为 3 周)。

3.10 微孔滤膜：0.45 μm，有机相。

4 仪器和设备

4.1 气相色谱-质谱仪：配置负化学离子源(NCI)。

4.2 分析天平：感量为 0.1 mg 和 0.01 g。

4.3 旋转蒸发器。

4.4 组织捣碎机。

4.5 绞碎机。

4.6 均质器。

4.7 振荡器。

4.8 离心机，最大转速为 5 000 r/min。

4.9 涡旋器。

5 试样制备与保存

5.1 试样制备

5.1.1 鸡肝、鸡腿肉、牛肉、鲳鱼或河虾

取代表性样品 500 g，将其切碎后，依次用绞碎机将样品绞碎，混匀，均分成两份，分装入洁净容器内，密封并标明标记。

5.1.2 鸡蛋、芝士粉、牛奶、蜂王浆和蜂蜜

将样品搅拌均匀，分出 500 g 作为试样。制备好的试样均分成两份，分别装入样品瓶中，密封，并标明标记。

鸡蛋样品制备时应去壳。

对无结晶的蜂蜜样品将其搅拌均匀；对有结晶析出的蜂蜜样品，在密闭情况下，将样品瓶置于不超过 60 ℃的水浴中温热，振荡，待样品全部融化后搅匀，迅速冷却至室温，在融化时应注意防止水分挥发。

5.2 试样的保存

牛奶、芝士粉和蜂蜜于 0 ℃～4 ℃保存；鸡蛋、鸡肝、鸡腿肉、牛肉、鲳鱼、河虾和蜂王浆等试样于－18 ℃以下冷冻保存。

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。

6 测定步骤

6.1 提取

6.1.1 牛奶、鸡蛋、蜂蜜和蜂王浆等液态或半液态试样

称取 5 g 试样（精确至 0.01 g）于 50 mL 的离心管中，（分析蜂蜜和蜂王浆时应加入 3 mL 水溶解或稀释混匀）加 10 mL 丙酮，加入 6 g 氯化钠和 20 mL 正己烷，加盖涡旋 30 s，超声 15 min，2 000 r/min 离心 5 min，移取上清液于 125 mL 分液漏斗中，用 20 mL 正己烷按照上述步骤重复提取一次，合并提取液，待净化。

6.1.2 鸡肝、鸡腿肉、牛肉、鲳鱼、河虾和芝士粉等固态试样

称取 5 g 试样（精确至 0.01 g）于 250 mL 的具塞锥形瓶中，加入 30 mL 正己烷，加入 20 g 无水硫酸

钠，振荡提取 40 min 或静置过夜，过滤于 125 mL 分液漏斗中。再加入 20 mL 正己烷重复提取一次，合并提取液，待净化。

6.2 磺化净化

加 5 mL 浓硫酸于有提取液的分液漏斗内，轻轻地振摇。静置分层后，弃去酸液层。再按上述操作重复净化 2 次至酸液呈无色或淡黄色。静置分层后，弃去酸液。然后用硫酸钠水溶液 50 mL 洗涤提取溶液，振摇 1 min，静置分层。弃去水层，再重复洗涤一次。然后将有机相通过无水硫酸钠柱脱水，收集于 150 mL 平底烧瓶内，40 ℃下旋转蒸发至干，准确加入 1 mL 正己烷，待测定。

6.3 测定

6.3.1 气相色谱-质谱条件

6.3.1.1 色谱柱：DB-17ms 毛细管柱，30 m×0.25 mm(内径)，膜厚 0.25 μm，或相当者。

6.3.1.2 色谱柱温度：100 ℃ $\xrightarrow{30\ ℃/min}$ 210 ℃ $\xrightarrow{15\ ℃/min}$ 300 ℃(4.33 min)。

6.3.1.3 进样口温度：300 ℃。

6.3.1.4 色谱-质谱接口温度：250 ℃。

6.3.1.5 载气：氦气，纯度大于等于 99.999%；流速，1.0 mL/min。

6.3.1.6 进样量：1 μL。

6.3.1.7 进样方式：不分流进样，1.5 min 后开阀。

6.3.1.8 电离方式：NCI。

6.3.1.9 离子源温度：150 ℃。

6.3.1.10 电子能量：70 eV。

6.3.1.11 反应气：甲烷，纯度大于等于 99.99%，反应气流速：2 mL/mim。

6.3.1.12 检测方式：分时段选择离子监测方式(SIM)：详见表 1。

表 1 目标农药的保留时间(窗口)和监测离子

农药	时间窗口 min	保留时间 min	STM 监测离子
六氯苯		5.16	250，282，284[a]，286
α-666		5.29	
γ-666	4.50～7.00	5.73	
β-666		5.95	253，255[a]，257，325
δ-666		6.31	
p，*p*′-DDE		7.74	281，316，318[a]，320
o，*p*′-DDT	7.00～END	8.40	35，246[a]，248，281
p，*p*′-DDD		8.46	248，250，318[a]，320
p，*p*′-DDT		8.81	35，248，281[a]，283

[a] 该离子为待测物的定量离子。

6.3.1.13 溶剂延迟时间：4.50 min。

6.3.2 气相色谱-质谱检测及确证

根据样液中待测物含量的情况，选定峰面积相近的标准工作溶液，对标准工作液和样液等体积参插进样。标准工作溶液和样液中待测农药的响应值均应在仪器的线性范围内。

进行样品测定时，如果检出的质量色谱峰保留时间与标准样品一致，并且在扣除背景后的样品谱图中，各定性离子的相对丰度与浓度接近的同样条件下得到的标准溶液谱图相比，最大允许相对偏差不超过表 2 中规定的范围，则可判断样品中存在对应的被测农药。在上述色谱条件下，每种农药的保留时间参见表 1。混合标准溶液的气相色谱-质谱总离子流色谱图参见图 A.1。

表 2 使用定性气相色谱-质谱时相对离子丰度最大容许误差

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

6.4 空白试验

除不加试样外，均按上述测定步骤进行。

6.5 结果计算和表述

用色谱数据处理机或按式(1)计算试样中待测农药的残留量：

$$X_i = \frac{A_{xi} \times c_{si} \times V_x}{A_{si} \times m} \quad \cdots\cdots(1)$$

式中：

X_i ——试样中待测农药残留量的含量，单位为毫克每千克(mg/kg)；

A_{xi}——样液中待测农药定量离子的峰面积；

c_{si} ——标准工作液中待测农药的浓度，单位为微克每毫升(μg/mL)；

V_x ——样液最后定容体积，单位为毫升(mL)；

A_{si}——标准工作液中待测农药定量离子的峰面积；

m ——最终样液所代表的试样质量，单位为克(g)。

注：计算结果需将空白值扣除。

7 测定低限和回收率

7.1 测定低限

本方法各种基质的六六六、滴滴涕和六氯苯检测限和定量限均分别为 0.002 mg/kg 和 0.010 mg/kg。

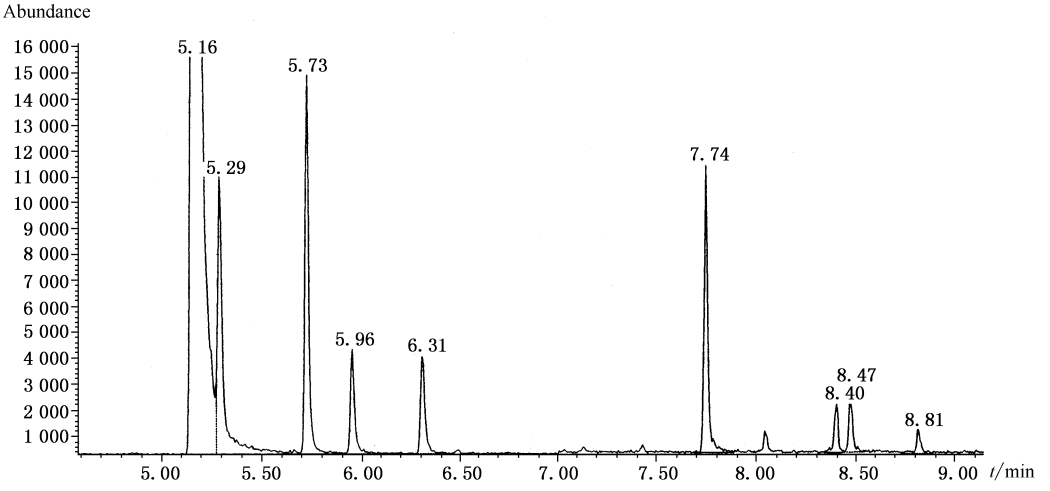
7.2 回收率

不同基质中添加浓度水平下的回收率范围参见表 3。

表 3　三个添加水平下食品中六六六、滴滴涕和六氯苯的回收率数据　%

样品名称	不同添加水平下所有农药的平均回收率范围		
	0.01 mg/kg	0.02 mg/kg	0.04 mg/kg
鸡蛋	103.7～120.0	96.3～110.9	94.4～120.3
牛奶	77.2～100.8	97.4～111.9	93.9～111.4
芝士粉	82.2～113.7	87.3～113.9	94.8～114.5
鸡肝	86.0～104.2	86.1～107.5	89.4～109.7
鸡腿肉	91.3～105.8	86.1～94.3	88.3～110.6
牛肉	82.7～93.8	83.3～94.0	88.6～99.6
鲳鱼	96.2～112.0	95.8～112.3	98.1～115.5
河虾	73.5～97.2	87.6～110.1	88.8～113.7
蜂王浆	81.7～103.7	74.8～102.6	79.5～104.3
蜂蜜	105.0～112.0	90.0～110.1	90.9～108.1

附 录 A
（资料性附录）
混合标准溶液 GC-MSD/NCI 总离子流色谱图



5.16 min——六氯苯；

5.29 min——α-666；

5.73——γ-666；

5.96——β-666；

6.31——δ-666；

7.74 min——*p*,*p*′-DDE；

8.40 min——*o*,*p*′-DDT；

8.47 min——*p*,*p*′-DDD；

8.81 min——*p*,*p*′-DDT。

图 A.1 混合标准溶液 GC-MS/NCI 总离子流色谱图

Foreword

This standard was drafted in accordance with the GB/T 1. 1—2009.

This standards replaced SN 0127—1992《Method for determination of BHC and DDT residues in milk and dairy product for export》、SN 0128—1992《Method for determination of BHC and DDT residues in egg and egg product for export》、SN 0129—1992《Method for determination of BHC and DDT residue in aquatic product for export》、SN 0310—1992《Method for determination of BHC and DDT residuers in honey product for export》.

The mainly differences between this standard，SN/T 0127，SN/T 0128，SN/T 129 and SN/T 130 are：

—expand the applicable matrix scope of the standard；

—develop the technique of sample preparation；

—GC-MS with the technique of negative ionization replace GC method.

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

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

This standard is mainly drafted by Shen Chongyu，Shen Weijian，Zao Zengyun，Liu Han，Wu Bin，Yu Keyao，Gui Qianwen，Gong Yuxia.

This standard replaced the previous version of the release of the standard as follows：

—SN 0127—1992；

—SN 0128—1992；

—SN 0129—1992；

—SN 0130—1992.

Determination of BHCs, DDTs and HCB residues in foods of animal origin for import and export—GC-MS method

1 Scope

This standard specifies preparation of test sample, determination and confirmation of BHCs, DDTs, and HCB residues by gas chromatography-negative chemical ionization mass spectrometry (GC-MS/NCI) in foodstuffs of animal origin, such as egg, milk, powdered cream, chicken liver, drumstick, beef, longsnout catfish, shrimp, royel jelly and bee honey, etc., for import and export.

This standard is applicable to the determination and confirmation of BHCs, DDTs, and HCB residues in foodstuffs of animal origin, such as egg, milk, powdered cream, chicken liver, drumstick, beef, longsnout catfish, shrimp, royel jelly and bee honey, etc.

2 Principle

BHCs, DDTs, and HCB residues are extracted with acetone or n-hexane from tested sample, following by a simple cleanup step with sulfuric acid. The aliquot is determined and confirmed by gas chromatography-negative chemical ionization mass spectrometry (GC-MS/NCI) using external standard method.

3 Reagents and materials

Unless otherwise specified, all the reagents used should be analytical grade. "Water" is redistilled water.

3.1 *n*-Hexane: HPLC Grade.

3.2 Acetone: HPLC Grade.

3.3 Concentrated sulfuric acid.

3.4 Anhydrous sodium sulfate: Roasted at 650 ℃ for 4 h, and stored in a tightly closed container.

3.5 Sodium chloride.

3.6 Sodium sulfate solution (20 g/L): Weight 20 g anhydrous sodium sulfate to 1 000 mL water.

3.7 Pesticide standard: α-BHC, CAS-319-84-6; β-BHC, CAS No.: 319-85-7; γ-BHC, CAS No.: 58-89-9; δ-BHC, CAS No.: 319-86-8; *p*, *p*′-DDD, CAS No.: 1022-22-6; *p*, *p*′-DDE, CAS No.: 72-55-9; *o*, *p*′-DDT, CAS No.: 789-02-6; *p*, *p*′-DDT, CAS No.: 50-29-3; hexachlorobenzene, CAS No.: 118-74-1; Purity ≥98.0%.

3.8 Standard stock solution: Accurately weight an adequate amount of each standard and dissolve in a small volume of acetone respectively. Dilute with acetone to form a standard stock solution of 100 μg/mL in concentration (Be stored below 4 ℃ for 6 months).

3.9 Mixture standard stock and working solution: accurately transfer 1.0 mL each stock standard solution into 10 mL volumetric flask, then make up to graduation with acetone, prepare a solution of 10 μg/mL as the mixture standard stock solution, stored below 4 ℃ for three month, Then dilute the mixture standard stock solution with acetone to the required concentration as the standard working solution (Be stored below 4 ℃ for 3 weeks only).

3.10 0.45 μm organic phase fiber.

4 Apparatus and equipment

4.1 Gas chromotograph/msss spectrometry (MSD) equipped with negative chemical ionization.

4.2 Balances (0.1 mg, 0.01 g).

4.3 Rotatory evaporator.

4.4 Tissue triturator.

4.5 Grinding machine.

4.6 Homogenizer.

4.7 Oscillator.

4.8 Centrifuge: (max 5 000 r/min).

4.9 Vortex mixer.

5 Sample preparation and storage

5.1 Preparation of test sample

5.1.1 Chicken liver, drumstick, beef, longsnout catfish and shrimp, etc.

About 500 g representative samples should be taken from all samples, the edible parts are cut into mince and homogenized by a high speed tissue triturator. The mixed primary sample is divided into two equal portion. Each portion is put into one clean sample bottle which is sealed and labled.

5.1.2 Egg, milk, powdered cream, bee products, such as royel jelly and bee honey, etc.

About 500 g representative samples should be taken from all samples, and the sample that is not crystallized shall be stirred well to make homogeneous. If the sample is crystallized, it must be warmed in a water-bath below 60 ℃ with the sample bottle covered tightly, mix thoroughly when all sample has melted, then cool immediately to room temperature. In the course of melting the sample, precautions must be taken to avoid evaporation of water from the sample. Then divide the pulp into two equal portions. Each portion is put in a clean container which is sealed and labled.

5.2 Storage of test sample

The test samples of milk, powdered cream and bee honey should be stored 0 ℃～4 ℃. The test samples of chicken liver, drumstick, beef, longsnout catfish, shrimp, egg and royel jelly 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.

6 Procedure

6.1 Extraction

6.1.1 Milk, egg, bee honey and royal jelly, etc.

Weigh ca 5 g of the test sample (accurate to 0.01 g) into 50 mL centrifuge tube (For bee honey and royal jelly sample, add 3 mL water homogenized), add 10 mL acetone, 6 g sodium chloride and 20 mL *n*-hexane, homogenize for 30 s with the vortex mixer, and extract with ultersonic machine for 15 min, and then centrifuge at 2 000 r/min for 5 min. Transfer the upper solvent layer to 125 mL separator. The residue is extracted with 20 mL *n*-hexane again, and combine the extracts into the same separator, and waiting for cleanup operation.

6.1.2 Chicken liver、drumstick、beef, longsnout catfish, shrimp, and powdered cream, etc.

Weigh ca 5 g of the test sample (accurate to 0.01 g) into 250 mL stoppered conical flask, add 30 mL *n*-hexane, 20 g sodium acetate anhydrous, vibrating for 40 min in the oscillator. Filter the extract into a 125 mL separator. The residue is extracted with 20 mL *n*-hexane again, filter and combine the extracts into the same separator, and waiting for cleanup operation.

6.2 Cleanup

Add 5 mL concentrated sulfuric acid to the separator, and shake for minutes. After statically separate into two layers, lay the acid layer aside, and duplicate the step to no color in the acid layer, and discard the acid layer again. Wash the organic layer with 50 mL sulfuric acid solution, and shake for 1 min, and wash again after separate into two layer and discard the water layer. Then collect organic layer into 150 mL Bunsen flask after dehydration with anhydrous sodium sulfate column, and concentrate to dry with Rotary Evaporator at 40 ℃. Add 1 mL *n*-hexane, and wash, and solute, and filter with 0.45 μm organic phase fiber.

6.3 Determination

6.3.1 GC-MSD operation conditions

6.3.1.1 Column: DB-17 ms fused quartz capillary column, 30 m × 0.25 mm (i. d.), film thickness 0.25 μm, or the equivalent.

6.3.1.2 Column temperature: 100 ℃ $\xrightarrow{30\ ℃/min}$ 210 ℃ $\xrightarrow{15\ ℃/min}$ 300 ℃ (4.33 min).

6.3.1.3 Inlet temperature: 300 ℃.

6.3.1.4 Interface temperature: 250 ℃.

6.3.1.5 Carrier gas: Helium, purity ≥99.999%, flow rate = 1.0 mL/min.

6.3.1.6 Injection volumn: 1 μL.

6.3.1.7 Injection mode: splitless, purge after 1.5 min.

6.3.1.8 Ionization mode: NCI.

6.3.1.9 Ionization source temperature: 150 ℃.

6.3.1.10 Electron energy: 70 eV.

6.3.1.11 Reagent gas: methane, purity ≥99.99%, flow rate: 2 mL/min.

6.3.1.12 Acquisition mode: Selected Ion Monitoring (SIM) mode (see table 1).

Table 1—RT(window)and mornitoring ion of pesticide

Pesticide	RT Window(min)	RT(min)	SIM Ion
Hexachlorobenzene		5.16	250,282,284[a],286
α-666		5.29	
γ-666	4.50～7.00	5.73	
β-666		5.95	253,255[a],257,325
δ-666		6.31	
p,*p*′-DDE		7.74	281,316,318[a],320
o,*p*′-DDT	7.00～END	8.40	35,246[a],248,281
p,*p*′-DDD		8.46	248,250,318[a],320
p,*p*′-DDT		8.81	35,248,281[a],283
[a] the qualitative ion of the pesticide.			

6.3.1.13 Solvent delay time: 4.50 min.

6.3.2 GC-MS determination and confirmation

According to the approximate concentration of the pesticide in the sample solution, select the standard working solution with similar concentration of the sample solution. The standard working solution should be injected in-between the injections of the sample solution with one common volume. The response of BHCs, DDTs, and HCB in the standard working solution and sample solution should be within the linear range of the instrument detection.

If there is a peak appeared at the same retention time for both of sample solution and standard working solution, and the qualification ions for every compound must be found, and for the same analysis batch and the same compound, the variation range of the ion ratio between the two daughter ions for the unknown sample and the standard working solution at the similar concentration cannot be out of range of table 2.

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

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

Under the above GC-MSD operating conditions, the retention time of BHCs, DDTs, and HCB peaks are listed in table 1. The GC-MS total ion chromatogram of mixture standard solution are shown respectively by figure A.1 in annex A.

6.4 Blank test

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

6.5 Calculation and expression of the result

Calculate the content of BHCs, DDTs, and HCB residue in the test sample by GC-MSD data processor or using the formula (1):

$$X_i = \frac{A_{xi} \times c_{si} \times V_x}{A_{si} \times m} \quad \cdots\cdots (1)$$

Where

X_i —the residue content of triadimenol in the test sample, mg/kg;

A_{xi}—the total area of quantitative ion for two triadimenol peaks in the sample solution;

c_{si} —the concentration of triadimenol in the standard working solution, μg/mL;

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

A_{si}—the total area of quantitative ion for two triadimenol peaks in the standard working solution;

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

7 LOD, LOQ and recovery

7.1 LOD and LOQ

LOD and LOQ of this method is 0.002 mg/kg and 0.010 mg/kg, respectively.

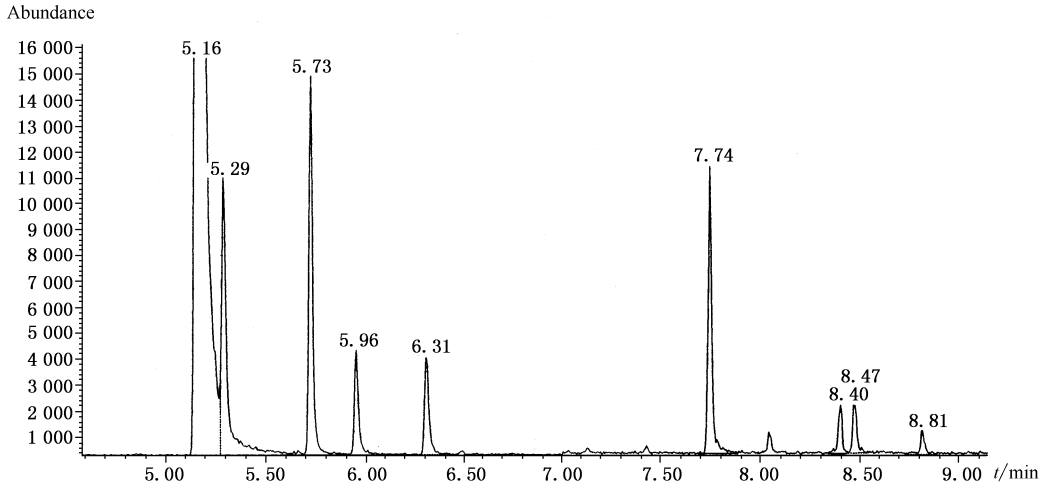
7.2 Recovery

Recovery range data of BHCs, DDTs, and HCB at three spiked levels in different foodstuff are listed in table 3.

Table 3—Recovery and RSD data of BHCs，DDTs，and HCB at three spiked levels in foods %

Food type	Recovery range data of all pesticides at three spiked level		
	0.01 mg/kg	0.02 mg/kg	0.04 mg/kg
egg	103.7～120.0	96.3～110.9	94.4～120.3
milk	77.2～100.8	97.4～111.9	93.9～111.4
zhishi Powder	82.2～113.7	87.3～113.9	94.8～114.5
chicken liver	86.0～104.2	86.1～107.5	89.4～109.7
chicken	91.3～105.8	86.1～94.3	88.3～110.6
beef	82.7～93.8	83.3～94.0	88.6～99.6
longsnout catfish	96.2～112.0	95.8～112.3	98.1～115.5
shrimp	73.5～97.2	87.6～110.1	88.8～113.7
rayal jelly	81.7～103.7	74.8～102.6	79.5～104.3
bee honey	105.0～112.0	90.0～110.1	90.9～108.1

Annex A
(Informative)
GC-MSD/NCI selected ion chromatogram of the mixture standard solution



5. 16 min—Hexachlorobenzene;

5. 29 min—α-666;

5. 73—γ-666;

5. 96—β-666;

6. 31—δ-666;

7. 74 min—*p*,*p*′-DDE;

8. 40 min—*o*,*p*′-DDT;

8. 47 min—*p*,*p*′-DDD;

8. 81 min—*p*,*p*′-DDT.

Figure A. 1—GC-MS/NCI selected ion chromatogram of the mixture standard solution