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中华人民共和国出入境检验检疫行业标准

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进出口动物源性食品中敌百虫、敌敌畏、蝇毒磷残留量的检测方法 液相色谱-质谱/质谱法

Determination of trichlorfor, dichlorvos and coumaphos residues in foodstuffs of animal origin for import and export—LC-MS/MS method

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前　言

本标准的附录A、附录B和附录C均为资料性附录。

本标准由国家认证认可监督管理委员会提出并归口。

本标准由中华人民共和国重庆出入境检验检疫局、中华人民共和国湖南出入境检验检疫局和中华人民共和国浙江出入境检验检疫局负责起草。

本标准主要起草人：王国民、戴华、谢文、朱舸、王美玲、李贤良、李应国、张雷、周启明、王象贤。

本标准系首次发布的出入境检验检疫行业标准。

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进出口动物源性食品中敌百虫、敌敌畏、蝇毒磷残留量的检测方法 液相色谱-质谱/质谱法

1 范围

本标准规定了畜、禽分割肉、盐渍肠衣和蜂蜜中敌百虫、敌敌畏、蝇毒磷残留量的制样和液相色谱-质谱/质谱检验方法。

本标准适用于分割肉、盐渍肠衣和蜂蜜中敌百虫、敌敌畏、蝇毒磷残留量的测定。

2 试样的制备与保存

分割肉：取样品中有代表性的约500 g，用组织捣碎机捣碎，装入洁净容器作为试样，密封并做好标识，于－18℃冰箱内保存。

肠衣：取有代表性样品约100 g，用剪刀剪碎至2 mm以下，装入洁净容器作为试样，密封并做好标识，于－18℃冰箱内保存。

蜂蜜：取有代表性样品约500 g，搅拌均匀后装入洁净容器内密封并做好标识，于－18℃冰箱内保存。

制样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。

3 方法提要

试样中的敌百虫、敌敌畏、蝇毒磷用二氯甲烷或乙酸乙酯提取，提取液经浓缩、脱脂后，用液相色谱-质谱/质谱仪测定，外标峰面积法定量，子离子丰度比定性。

4 试剂和材料

所有试剂除特殊注明外，均为分析纯，水为二次蒸馏水。

4.1 乙腈：高效液相色谱级。

4.2 甲醇：高效液相色谱级。

4.3 乙酸乙酯。

4.4 二氯甲烷。

4.5 环己烷。

4.6 硫酸钠：650℃灼烧4 h，在干燥器内冷却至室温，贮于密封瓶中备用。

4.7 乙酸铵：优级纯或相当者。

4.8 50 mmol/L乙酸铵溶液：称取0.385 g乙酸铵溶于1 000 mL水中。

4.9 敌百虫(Trichlorfor，CAS号：52-68-6，分子式：$C_4H_8Cl_3O_4P$)标准品：纯度大于或等于98.0%。

4.10 敌敌畏(Dichlorvos，CAS号：62-73-7，分子式：$C_4H_7Cl_2O_4P$)标准品：纯度大于或等于97.6%。

4.11 蝇毒磷(Coumaphos，CAS号：56-72-4，分子式：$C_{14}H_{16}ClO_5PS$)标准品：纯度大于或等于99.9%。

4.12 标准储备液：分别准确称取适量敌百虫、敌敌畏、蝇毒磷标准品，用甲醇配制成浓度为100 μg/mL浓度的混合标准储备液。此溶液在0℃～4℃冰箱中避光保存，可使用3个月。

4.13 空白样品提取液：用不含敌百虫、敌敌畏、蝇毒磷残留的样品，按照6.1制备空白样品提取液。

4.14 标准工作溶液：根据需要用空白样品提取液将标准储备液稀释成50 ng/mL、100 ng/mL、

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200 ng/mL、500 ng/mL 的混合标准工作溶液。置于 0℃～4℃冰箱中避光保存，可使用 3 d。

5 仪器和设备

5.1 高效液相色谱-质谱/质谱仪：三重四极杆质谱检测器，配电喷雾离子源(ESI)或相当者。
5.2 超声波清洗器。
5.3 旋涡混匀器。
5.4 旋转蒸发仪。
5.5 离心机：5 000 r/min。
5.6 均质器。
5.7 0.45 μm 微孔滤膜。

6 测定步骤

6.1 提取与净化

6.1.1 分割肉

a) 准确称取 5 g 均匀试样(精确到 0.01 g)于 50 mL 具塞试管中，加入 5 g 无水硫酸钠混匀，再加入 15 mL 二氯甲烷，用均质器(10 000 r/min)均质 2 min，4 000 r/min 离心 3 min，将有机相转移至 100 mL 梨形蒸馏瓶中，残渣再用 2×10 mL 二氯甲烷均质提取两次。

b) 离心合并有机相，于 40℃旋转蒸发至 2 mL，将样液转移至 5 mL 刻度试管中，并用少量二氯甲烷洗涤梨形蒸馏瓶，合并洗涤液到刻度试管中，室温通氮浓缩至干。定量加入 1.0 mL 乙腈溶解残渣，加入 2 mL 环已烷旋涡混匀 2 min 后，2 500 r/min 离心 3 min，将下层乙腈相过 0.45 μm微孔滤膜后，供液相色谱-质谱/质谱仪测定。

6.1.2 肠衣

准确称取 5 g 试样(精确到 0.01 g)于 50 mL 具塞离心瓶中，加入 2 g 无水硫酸钠混匀，再加入 15 mL二氯甲烷，盖上盖混匀，置于超声波清洗器中超声 30 min，冷却后将有机相过滤转移至 100 mL 梨形蒸馏瓶中，残渣再用 2×10 mL 用二氯甲烷混匀超声提取两次，离心合并有机相。接下来按照 6.1.1 中 b)的步骤进行。

6.1.3 蜂蜜

称取 5 g 试样(精确到 0.01 g)置于 50 mL 具塞离心管中，加 10 mL 水，加 25 mL 乙酸乙酯，于旋涡混合器上混匀 1 min，以 3 000 r/min 离心 5 min，将上层乙酸乙酯提取液收集于浓缩瓶中，残渣再加入 20 mL 乙酸乙酯，重复上述操作，合并乙酸乙酯提取溶液。在 50℃以下减压浓缩至约 3 mL 后转移至离心管，用 5 mL 乙酸乙酯分两次洗涤浓缩瓶，合并洗涤液于离心管中，用氮气吹干干。加 1.0 mL 乙腈溶解残渣，并过 0.45 μm 微孔滤膜后，供液相色谱-质谱/质谱仪测定。

6.2 测定

6.2.1 液相色谱-质谱/质谱条件

a) 色谱柱：柱填料为十八烷基硅烷键合相的色谱柱，4.6×150 mm，5 μm 或相当者；
b) 流动相：甲醇：50 mmol/L 乙酸铵，梯度洗脱程序参见附录 A；
c) 流速：0.50 mL/min 或根据仪器条件优化；
d) 柱温：40℃；
e) 进样量：10 μL；
f) 离子源：电喷雾离子源；
g) 扫描方式：正离子；
h) 检测方式：多反应监测(MRM)；
i) 质谱/质谱参考条件参见附录 B。

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6.2.2 定性、定量测定

按照6.2.1液相色谱-质谱/质谱条件测定样液和标准工作溶液，外标标准曲线法测定样液中的敌百虫、敌敌畏、蝇毒磷残留量。样品中待测物残留量应在标准曲线范围之内，如果残留量超出标准曲线范围，应用空白样品提取液(4.13)进行适当稀释。在上述色谱条件下，敌百虫、敌敌畏、蝇毒磷的质量色谱峰保留时间约为13.2 min，14.1 min，15.9 min。标准品的多反应监测色谱图参见附录C中的图C.1。

在相同的实验条件下，样品与标准工作液中待测物质的质量色谱峰相对保留时间在2.5%以内，并且在扣除背景后的样品质量色谱图中，所选择的离子对均出现，同时与标准品的相对丰度允许偏差不超过表1规定的范围，则可判断样品中存在对应的被测物。

表1 使用定性液相色谱-质谱/质谱时相对离子丰度最大容许误差

相对丰度(基峰)/(%)	液相色谱-质谱/质谱时相对离子丰度最大允许误差/(%)
>50	±20
大于20至小于等于50	±25
大于10至小于等于20	±30
小于等于10	±50

7 结果计算和表述

用数据处理软件中的外标法，或绘制标准曲线，按照式(1)计算样品中敌百虫、敌敌畏、蝇毒磷残留量。

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

式中：

X——试样中敌百虫、敌敌畏、蝇毒磷的残留量，单位为毫克每千克(mg/kg)；

c——由标准曲线而得的样液中敌百虫、敌敌畏、蝇毒磷的浓度，单位为纳克每毫升(ng/mL)；

V——样液最终定容体积，单位为毫升(mL)；

m——最终样液所代表的试样质量，单位为克(g)。

8 测定低限、回收率

8.1 测定低限

本方法敌百虫、敌敌畏、蝇毒磷的测定低限均为0.010 mg/kg。

8.2 回收率

8.2.1 肠衣中敌百虫添加浓度及其回收率数据：

添加浓度在0.010 mg/kg时，回收率在73.0%～94.0%之间；

添加浓度在0.020 mg/kg时，回收率在78.0%～95.0%之间；

添加浓度在0.040 mg/kg时，回收率在78.0%～95.0%之间。

8.2.2 肠衣中敌敌畏添加浓度及其回收率数据：

添加浓度在0.010 mg/kg时，回收率在76.0%～98.0%之间；

添加浓度在0.020 mg/kg时，回收率在78.0%～98.0%之间；

添加浓度在0.040 mg/kg时，回收率在86.0%～95.0%之间。

8.2.3 肠衣中蝇毒磷添加浓度及其回收率数据：

添加浓度在0.010 mg/kg时，回收率在75.0%～95.0%之间；

添加浓度在0.020 mg/kg时，回收率在80.0%～96.0%之间；

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添加浓度在0.040 mg/kg时，回收率在77.0%～92.0%之间。

8.2.4 分割肉中敌百虫添加浓度及其回收率数据：

添加浓度在0.010 mg/kg时，回收率在92.0%～115%之间；

添加浓度在0.020 mg/kg时，回收率在86.5%～113%之间；

添加浓度在0.040 mg/kg时，回收率在80.8%～101%之间。

8.2.5 分割肉中敌敌畏添加浓度及其回收率数据：

添加浓度在0.010 mg/kg时，回收率在93.0%～110%之间；

添加浓度在0.020 mg/kg时，回收率在88.5%～108%之间；

添加浓度在0.040 mg/kg时，回收率在84.3%～110%之间。

8.2.6 分割肉中蝇毒磷添加浓度及其回收率数据：

添加浓度在0.010 mg/kg时，回收率在91.0%～112%之间；

添加浓度在0.020 mg/kg时，回收率在79.0%～102%之间；

添加浓度在0.040 mg/kg时，回收率在73.9%～95.3%之间。

8.2.7 蜂蜜中敌百虫添加浓度及其回收率数据：

添加浓度在0.010 mg/kg时，回收率在70.0%～110%之间；

添加浓度在0.020 mg/kg时，回收率在79.0%～104%之间；

添加浓度在0.040 mg/kg时，回收率在80.0%～107%之间。

8.2.8 蜂蜜中敌敌畏添加浓度及其回收率数据：

添加浓度在0.010 mg/kg时，回收率在70.0%～92.0%之间；

添加浓度在0.020 mg/kg时，回收率在71.0%～91.0%之间；

添加浓度在0.040 mg/kg时，回收率在70.0%～96.0%之间。

8.2.9 蜂蜜中蝇毒磷添加浓度及其回收率数据：

添加浓度在0.010 mg/kg时，回收率在79.0%～110%之间；

添加浓度在0.020 mg/kg时，回收率在74.0%～105%之间；

添加浓度在0.040 mg/kg时，回收率在71.0%～104%之间。

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附 录 A
（资料性附录）
梯度洗脱程序

表 A.1 流动相梯度洗脱程序

时间/min	甲醇/(%)	50 mmol/L 乙酸铵/(%)
0	10.0	90.0
5.00	10.0	90.0
10.00	95.0	5.0
18.00	95.0	5.0
18.01	10.0	90.0
25.00	10.0	90.0

附 录 B
（资料性附录）
API 4000 LC-MS/MS 系统检测敌百虫、敌敌畏、蝇毒磷参考条件

API 4000 LC-MS/MS 系统电喷雾离子源参考条件：

a) 窗帘气(CUR)：15.00 Pa；
b) 雾化气(GS1)：40.00 Pa；
c) 辅助加热气(GS2)：45.00 L/min；
d) 碰撞气(CAD)：7.00 Pa；
e) 离子源喷雾器电压(IS)：5 000.00 V；
f) 喷雾针温度：(TEM)：550.00℃；
g) 定性离子对、定量离子对、去簇电压、碰撞能量、碰撞室出口电压见表 B.1。

表 B.1 待测物定性离子对、定量离子对、去簇电压、碰撞能量和碰撞室出口电压

待测物	Q1 m/z	Q3 m/z	去簇电压/V	碰撞能量/V	碰撞室出口电压/V
敌百虫 Trichlorfon	259.0[a]	109.2	63.0	26.5	19.0
	256.9	221.2	63.0	16.0	13.0
敌敌畏 Dichlorvos	221.3[a]	109.2	63.0	25.5	19.0
	223.3	109.2	63.0	25.0	18.0
蝇毒磷 Coumaphos	363.3[a]	307.0	96.0	25.0	20.0
	363.3	227.0	96.0	36.0	13.0

[a] 为定量离子对。

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附 录 C
（资料性附录）
标准品多反应监测色谱图

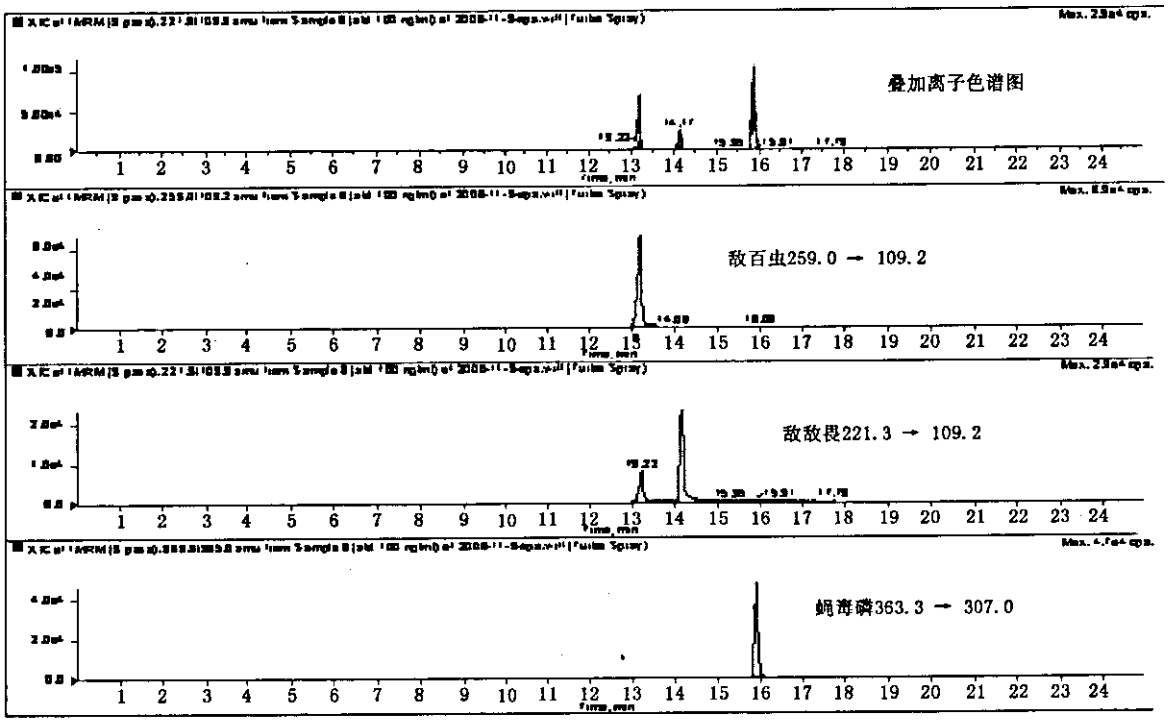


图 C.1 敌百虫、敌敌畏、蝇毒磷标准溶液多反应监测色谱图

1） 本标准所采用仪器设备及型号不涉及商业目的，鼓励标准使用者尝试不同厂家或型号的仪器。

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Foreword

Annex A, B and C of this standard are informative annexes.

This standard was proposed by and was under the charge of National Regulatory Commission for Certification and Accreditation of the People's Republic of China.

The standard was drafted by Chongqing Entry-Exit Inspection and Quarantine Bureau of the People's Republic of China, Hunan Entry-Exit Inspection and Quarantine Bureau of the People's Republic of China, and Zhejiang Entry-Exit Inspection and Quarantine Bureau of the People's Republic of China.

This standard was mainly drafted by Wang Guoming, Dai Hua, Xie Wen, Zhu Ge, Wang Meiling, Li Xianliang, Li Yingguo, Zhang Lei, Zhou Qiming and Wang Xiangxian.

This standard is a professional standard for entry-exit inspection and quarantine promulgated for the first time.

Note: This English version, a translation from the Chinese text, is only for reference.

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Determination of trichlorfor, dichlorvos and coumaphos residues in foodstuffs of animal origin —LC-MS/MS method

1 Scope

This standard specifies the methods of sample preparation and qualified and quantified determination by liquid chromatography-mass spectrometry of trichloffor, dichlorvos and coumaphos residues in animal muscle, salted casing and honey.

This standard is applicable to the determination of trichloffor, dichlorvos and coumaphos residues in animal muscle, salted casing and honey.

2 Sample preparation and storage

Muscle: About 500 g representative samples should be taken from all samples, then grinded and blended by a tissue blender to produce homogenous samples, put in suitable clean container. After being sealed and labeled, the samples should be stored at below −18℃.

Casing: About 100 g representative samples should be taken from all samples, then grinded and blended by a forfex, put in suitable clean container. After being sealed and labeled, the samples should be stored at below −18℃.

Honey: About 500 g representative samples should be taken from all samples, then mix round to produce homogenous samples, put in suitable clean container. After being sealed and labeled, the samples should be stored at below −18℃.

In the course of sampling and sample preparation, precaution should be taken to avoid contamination or any factor that may cause the change of residue content.

3 Principle

The residues of trichlorfor, dichlorvos and coumaphos residues in the test sample are extracted with Ethyl Acetate or dichloromethane. After being concentrated and reconstituted, the residues are determined by liquid chromatography-mass spectrometry, quantified by external standard method.

4 Reagents and materials

Unless specified, all reagents should be of analytical grade; "Water" is the distilled water.

4.1 Acetonitrile: HPLC grade.

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4.2 Methanol:HPLC grade.

4.3 Ethyl Acetate.

4.4 Dichloromethane.

4.5 Cyclohexane.

4.6 Sodium sulfate:Ignite at 650℃ for 4 h,cool to room temperature in a desiccator and store in sealed container.

4.7 Ammonium acetate:GR.

4.8 50 mmol/L ammonium acetate:Weigh 0.385 g ammonium acetate,dissolved in 1 000 mL water.

4.9 Trichlorfor (CAS NO:52-68-6 formula:$C_4H_8Cl_3O_4P$),purity≥98%.

4.10 Dichlorvos (CAS NO:62-73-7 formula:$C_4H_7Cl_2O_4P$),purity≥97.6%.

4.11 Coumaphos (CAS NO:56-72-4,formula:$C_{14}H_{16}ClO_5PS$),purity≥99.9%.

4.12 Standard stock solution:Accurately weigh trichlorfor,dichlorvos and coumaphos,dissloved with methanol,and mix to homogeneity. The concentration of the solution is 100 g/mL,can be preserved at the temperature 0℃～4℃ for three months.

4.13 Blank matrix extract solution:according to 6.1,extract the test sample without trichlorfor,dichlorvos and coumaphos.

4.14 Standard working solution:according to the requirement,dilute mixed standard solution with blank matrix extract solution . The concentration of the solution is 50 ng/mL,100 ng/mL,200 ng/mL and 500 ng/mL. It can be preserved at the temperature 0℃～4℃ for three days.

5 Apparantus and equipment

5.1 Liquid chromatography-mass spectrometry,equipped with electrospray ion source and triquadruple mass spectrometer.

5.2 Ultrasonic extractor.

5.3 Mechanical shaker.

5.4 Vortex mixer.

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5.5 Centrifuge:5 000 r/min.

5.6 Homogenizer.

5.7 Membrane filter:0.45 μm.

6 procedure

6.1 Extract and Clean-up

6.1.1 Muscle

a) Accurately weigh 5 g of the test sample (accurate to 0.01 g) into a 50 mL polypropylene centrifuge tube with cap,add 5 g anhydrous sodium sulfate and 15 mL of dichloromethane,homogenize for 2 min (10 000 r/min),then centrifuge at 4 000 r/min for 3 min. Transfer the supematant to a 100 mL evaporated flask, the residue was homogenized and extracted with 2 × 10 mL dichloromethane.

b) After centrifuging, combine the supernatant to the evaporated flask. Evaporate the dichloromethane to about 2 mL with rotary evaporator below 40℃, remove the residues to a 5 mL centrifuge tube,wash evaporated flask with little dichloromethane,combine the dichloromethane solution to graduated test tube,evaporate the extract to dryness on N-Evap at room temperature. The residue is dissolved with 1.0 mL of acetonitrile and 2 mL of cyclohexane. Votex and centrifuge at 2 500 r/ min for 3 min. Filter the nether layer through a 0.45 μm membrane into HPLC vials. The solution is ready for LC-MS/MS determination.

6.1.2 Casing

Accurately weigh 5 g of the test sample (accurate to 0.01 g) into a 50 mL polypropylene centrifuge tube with cap,add 5 g anhydrous sodium sulfate,add 15 mL of dichloromethane and vortex,then extract in ultrasonic extractor for 30 min. After cooling,transfer the supematant to a 100 mL evaporated flask,the residue was vortexed and extracted with 2 × 10 mL dichloromethane. After centrifuging, combine the supernatant to the evaporated flask. Then,deal with the sample according to the step of 6.1.1b).

6.1.3 Honey

Accurately weigh 5 g of the test sample (accurate to 0.01 g) into a 50mL polypropylene centrifuge tube with cap,add 10 mL water and 25 mL ethyl acetate,shake for 1 min on a mechanical shaker at 2 000 r/min,then centrifuge at 3 000 r/min for 5 min. Transfer the supernatant to a evaporated flask, the residues was re-extracted with 20 mL ethyl acetate as the above procedure,combine the supernatant,evaporated to about 3 mL with rotary evaporator in a water-bath below 50℃, remove the residue to a new centrifuge tube,wash the evaporated flask twice use 5 mL ethyl acetate,combine the eluate,evaporate the eluate to dryness on N-Evap,dissolved by 1.0 mL of methanol,Filter through a

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0.45 μm membrane into HPLC vials. The solution is ready for LC-MS/MS determination.

6.2 Determination

6.2.1 LC-MS/MS operation condition

a) LC column: C_{18}, 4.6 × 150 mm, 5 μm (or other conformable column);
b) Mobile phase: methanol; 50 mmol/L ammonium acetate, the elution gradient is listed in Annex A;
c) Flow rate: 0.50 mL/min;
d) Column temperature: 40℃;
e) Injector volume: 10 μL;
f) Ion source: ESI;
g) Scanning model: positive ion;
h) Monitoring model: multiple reaction monitoring (MRM);
i) Referenced conditions seen Annex B.

6.2.2 Qualification and quantification test

According to the method of 6.2.1, detect the residues of trichlorfor, dichlorvos and coumaphos in the test sample solution, the standard working solution. The response of trichlorfor, dichlorvos and coumaphos should be in the linear range of the instrumental detection. If the response is out of the linear range, the sample should be diluted with the blank matrix extract solution (4.13) to suitable concertration. Under the above chromatograph conditions, the reference retention time of trichlorfor, dichlorvos and coumaphos is about 13.2 min, 14.1 min, 15.9 min. Reconstituted ion chromatogram of standard working solution is listed in Annex C.

Under the same conditions of experiment, the retention time of the unknown sample is the same as the standard working solution; the qualification ions for every compound must be found. 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 1.

Table 1—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 Calculation and expression of result

Calculating the content of trichlorfor, dichlorvos and coumaphos residue concentration in the sample is carried out by LC/MS/MS data processor or according to the formula (1):

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

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where

X—the residue content of trichlorfor,dichlorvos and coumaphos in the test sample,mg/kg;

c—the concentration of trichlorfor,dichlorvos and coumaphos in the test sample calculated by calibration curve,ng/mL;

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

V—the final volume of sample solution,mL.

8 Limit of quantification and recovery

8.1 Limit of quantification

The limit of quantification for trichlorfor,dichlorvos and coumaphos is 0.010 mg/kg.

8.2 Recovery

8.2.1 According to the experimental date,the fortifying concentration of trichlorfor in casing and its corresponding recoveries are:

0.010 mg/kg,the recovery is 73.0%～94.0%;
0.020 mg/kg,the recovery is 78.0%～95.0%;
0.040 mg/kg,the recovery is 78.0%～95.0%.

8.2.2 According to the experimental date,the fortifying concentration of dichlorvos in casing and its corresponding recoveries are:

0.010 mg/kg,the recovery is 76.0%～98.0%;
0.020 mg/kg,the recovery is 78.0%～98.0%;
0.040 mg/kg,the recovery is 86.0%～95.0%.

8.2.3 According to the experimental date,the fortifying concentration of coumaphos in casing and its corresponding recoveries are:

0.010 mg/kg,the recovery is 75.0%～95.0%;
0.020 mg/kg,the recovery is 80.0%～96.0%;
0.040 mg/kg,the recovery is 77.0%～92.0%.

8.2.4 According to the experimental date,the fortifying concentration of trichlorfor in musle and its corresponding recoveries are:

0.010 mg/kg,the recovery is 92.0%～115%;
0.020 mg/kg,the recovery is 86.5%～113%;
0.040 mg/kg,the recovery is 80.8%～101%.

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8.2.5 According to the experimental date, the fortifying concentration of dichlorvos in musle and its corresponding recoveries are:

0.010 mg/kg, the recovery is 93.0%～110%;
0.020 mg/kg, the recovery is 88.5% ～108%;
0.040 mg/kg, the recovery is 84.3%～110%.

8.2.6 According to the experimental date, the fortifying concentration of coumaphos in musle and its corresponding recoveries are:

0.010 mg/kg, the recovery is 91.0%～112%;
0.020 mg/kg, the recovery is 79.0%～102%;
0.040 mg/kg, the recovery is 73.9%～95.3%.

8.2.7 According to the experimental date, the fortifying concentration of trichlorfor in honey and its corresponding recoveries are:

0.010 mg/kg, the recovery is 70.0%～110%;
0.020 mg/kg, the recovery is 79.0%～104%;
0.040 mg/kg, the recovery is 80.0%～107%.

8.2.8 According to the experimental date, the fortifying concentration of dichlorvos in honey and its corresponding recoveries are:

0.010 mg/kg, the recovery is 70.0%～92.0%;
0.020 mg/kg, the recovery is 71.0%～91.0%;
0.040 mg/kg, the recovery is 70.0%～96.0%.

8.2.9 According to the experimental date, the fortifying concentration of coumaphos in honey and its corresponding recoveries are:

0.010 mg/kg, the recovery is 79.0%～110%;
0.020 mg/kg, the recovery is 74.0%～105%;
0.040 mg/kg, the recovery is 71.0%～104%.

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Annex A
(informative)
The reference conditions of the elution gradient program

Table A. 1—The elution gradient

Time/min	Methanol/(%)	50 mmol/L ammonium acetate/(%)
0	10.0	90.0
5.00	10.0	90.0
10.00	95.0	5.0
18.00	95.0	5.0
18.01	10.0	90.0
25.00	10.0	90.0

Annex B
(informative)
Referenced conditions for analysing trichlorfor, dichlorvos and coumafos by API 4 000 LC-MS/MS system

Referenced conditions for electrospray ionization source of API 4 000 LC/MS/MS system:

a) Curtain gas (CUR): 15.00 Pa;
b) Nebulizer gas (GS1): 40.00 Pa;
c) Auxiliary gas (GS2): 45.00 L/min;
d) Collision gas (CAD): 7.00 Pa;
e) Ion spray voltage(IS): 5 000.00 V;
f) Probe temperature: 550.00℃;
g) Transitions for confirmation and quantification, DP, CE, CXP see Table B. 1.

Table B. 1—Transitions for confirmation and quantification, DP, CE, CXP

Target analyte	Q1 m/z	Q3 m/z	DP/V	CE/V	CXP/V
Trichlorfon	259.0[a]	109.2	63.0	26.5	19.0
	256.9	221.2	63.0	16.0	13.0
Dichlorvos	221.3[a]	109.2	63.0	25.5	19.0
	223.3	109.2	63.0	25.0	18.0
Coumaph	363.3[a]	307.0	96.0	25.0	20.0
	363.3	227.0	96.0	36.0	13.0

[a] Quantitation ion pair.

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Annex C
(informative)
LC/MS/MS extracting ion current(XIC)of+MRM chromatograms for the mixture standard

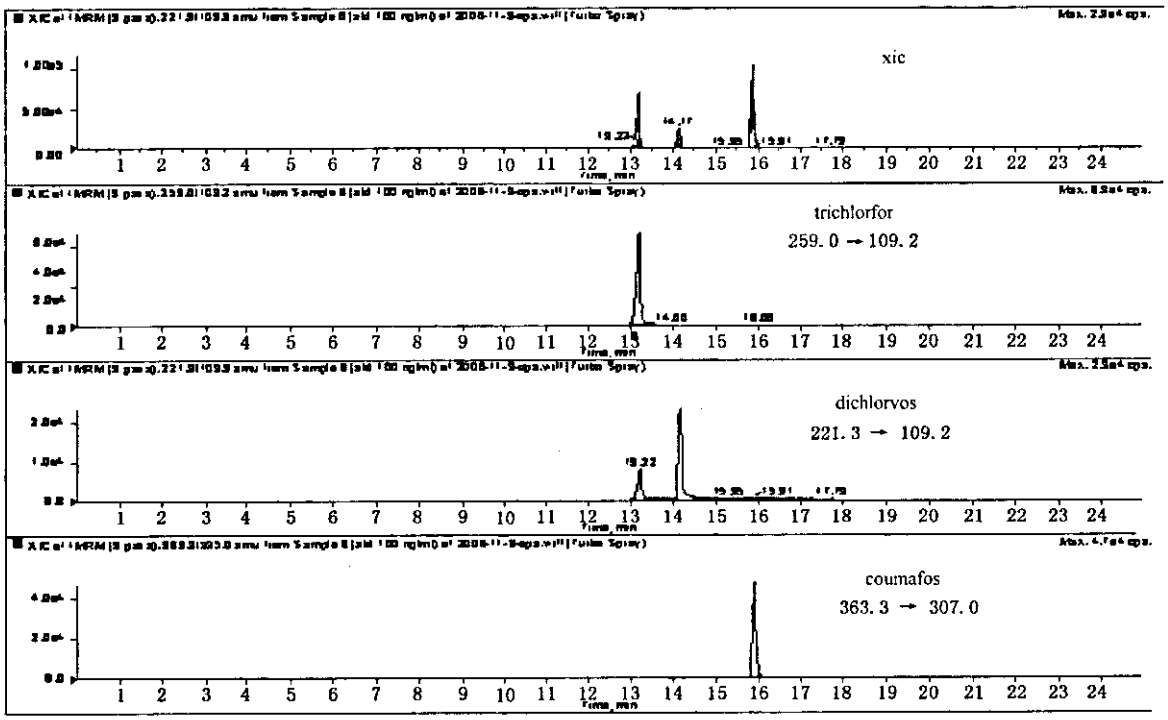


Figure C.1—LC/MS/MS extracting ion current(XIC)of+MRM chromatograms for a mixture of trichlorfor,dichlorvos and coumafos standard

1) the reference mass parameters in Annex A are accomplished by API 4000 LC/MS/MS,the equipment and its type involved in the standard method is only for reference and not related to any commercial aim,and the analysts are encouraged to use equipments of different corporation or different type.

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