



中华人民共和国出入境检验检疫行业标准

SN/T 2441—2010

进出口食品中涕灭威、涕灭威砜、 涕灭威亚砜残留量检测方法 液相色谱-质谱/质谱法

Determination of aldicarb, aldicarb-sulfone and aldicarb-sulfoxide
residues in food for import and export—LC-MS/MS method

2010-01-10 发布

2010-07-16 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

本标准的附录 A、附录 B 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。

本标准由中国检验检疫科学研究院负责起草。

本标准主要起草人：陈冬东、朱明达、蒋文彬、代汉慧、李晓娟、彭涛、李淑娟。

进出口食品中涕灭威、涕灭威砜、 涕灭威亚砜残留量检测方法 液相色谱-质谱/质谱法

1 范围

本标准规定了食品中涕灭威及其代谢产物涕灭威砜、涕灭威亚砜残留量的液相色谱-质谱/质谱检测和确证。

本标准适用于生姜、番茄、菠菜、大米、花生、大豆、杏仁、苹果、柑橘、茶叶、猪肝、鸡肉、牛奶中涕灭威、涕灭威砜、涕灭威亚砜的测定和确证。

2 方法提要

试样中残留的涕灭威、涕灭威砜、涕灭威亚砜用乙腈提取,经氨基 SPE 小柱净化后,用液相色谱-质谱/质谱仪检测,外标法定量。

3 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。

- 3.1 乙腈:HPLC 级。
- 3.2 甲醇。
- 3.3 二氯甲烷。
- 3.4 正己烷。
- 3.5 无水硫酸钠:650 ℃灼烧 4 h,贮于干燥器中,冷却后备用。
- 3.6 甲醇-二氯甲烷(1+99,体积比):量取 1 mL 甲醇和 99 mL 二氯甲烷,混匀。
- 3.7 0.1%甲酸溶液:量取 1 mL 甲酸,用水定容至 1 L 备用。
- 3.8 乙腈-0.1%甲酸水溶液(10+90,体积比):量取 10 mL 乙腈和 90 mL 0.1%甲酸溶液(3,7),混匀。
- 3.9 涕灭威标准品(aldicarb,CAS 编号 116-06-3):纯度大于等于 98%。
- 3.10 涕灭威砜标准品(aldicarb-sulfone,CAS 编号 1646-88-4):纯度大于等于 98%。
- 3.11 涕灭威亚砜标准品(aldicarb-sulfoxide,CAS 编号 1646-87-3):纯度大于等于 98%。
- 3.12 标准储备液:准确称取适量上述标准品,分别用甲醇配制成浓度为 100 μg/mL 的标准储备液,0 ℃~4 ℃避光保存。
- 3.13 空白样品提取液:用不含涕灭威、涕灭威砜、涕灭威亚砜的样品,按照 6.1 和 6.2 制备空白样品提取液。
- 3.14 基质工作溶液:根据需要用空白样品提取液将标准储备液稀释成 10.0 ng/mL、20.0 ng/mL、50.0 ng/mL、100 ng/mL、200 ng/mL 的标准工作溶液。置于 0 ℃~4 ℃避光保存。
- 3.15 氨基 SPE 柱:500 mg,3 mL,用 5 mL 甲醇-二氯甲烷(1+99,体积比)活化后备用。
- 3.16 0.45 μm 微孔滤膜:有机系。

4 仪器和设备

- 4.1 液相色谱-质谱/质谱仪,配电喷雾离子源(ESI)源。
- 4.2 均质器。

- 4.3 天平:感量为 0.1 mg,0.01 g。
- 4.4 振荡提取器。
- 4.5 旋转蒸发仪。
- 4.6 旋涡混匀器。
- 4.7 固相萃取装置。
- 4.8 氮吹仪。

5 试样的制备与保存

5.1 试样制备

5.1.1 大米、花生、大豆、杏仁、茶叶

取有代表性样品约 500 g,粉碎并使其全部通过孔径为 2.0 mm 的样品筛。混合均匀,装入洁净的容器内,密封并做好标识。

5.1.2 生姜、番茄、菠菜、苹果、柑橘、猪肝、鸡肉

取有代表性样品约 500 g,取可食用部分将其切成小块(不可水洗),用组织捣碎机将样品匀浆,混合均匀,装入洁净容器内密封并做好标识。

5.2 试样保存

粮谷、坚果类试样于 0 ℃~4 ℃冰箱内保存;水果、蔬菜、肉等试样于 -18 ℃冰箱内保存。

制样和样品保存过程中,应防止样品受到污染。

6 测定步骤

6.1 提取

称取 5 g 试样(精确至 0.01 g)于 50 mL 离心管中,加入 20 mL 乙腈,均质 2 min,振荡提取 20 min,上清液过无水硫酸钠收集到分液漏斗中,残渣再加入 20 mL 乙腈,重复上述操作一次,合并 2 次滤液,加入 20 mL 用乙腈饱和的正己烷,振荡 10 min,静置分层,弃去正己烷层,乙腈层于 40 ℃下旋转蒸发浓缩至干,用 2 mL 甲醇-二氯甲烷(1+99,体积比)(3.6)溶解,待净化。

6.2 净化

将 6.1 步骤中所获得的提取液全部转移至活化好的氨基 SPE 柱中(3.15),用 5 mL 甲醇-二氯甲烷(1+99,体积比)洗脱,收集全部流出液,氮气吹干后,用 1 mL 乙腈-0.1% 甲酸水溶液(10+90,体积比)定容。过 0.45 μm 微孔滤膜,上液相色谱-质谱/质谱仪,待测定。

6.3 测定

6.3.1 液相色谱-质谱/质谱条件

- a) 液相色谱-质谱/质谱仪;
- b) 色谱柱:C₁₈柱 100 mm×2.1 mm(内径),3.5 μm,或相当者;
- c) 流动相:乙腈:0.1% 甲酸,梯度程序参见附录 A;
- d) 进样量:20 μL;
- e) 柱温:30 ℃;
- f) 流速:0.3 mL/min 或根据仪器条件优化;
- g) 离子源:电喷雾离子源;
- h) 扫描方式:正离子;
- i) 检测方式:多反应监测(MRM)。

6.3.2 定性、定量测定

按照 6.3.1 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涕灭威、涕灭威砜、涕灭威亚砜的残留量。样品中待测物涕灭威、涕灭威砜、涕灭威亚砜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(3.13)进行适当稀释。在上述色谱条件下,标准品的多反应监测色谱图参见附录B中的图B.1。在相同的试验条件下,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允许误差

相对丰度(基峰)/%	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

6.3.3 空自试验

空白样品按上述测定步骤进行。

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)计算样品中涕灭威、涕灭威砜和涕灭威亚砜的残留量。

式中：

X——试样中涕灭威、涕灭威砜、涕灭威亚砜的残留量,单位为毫克每千克(mg/kg);

c——由标准曲线而得的样液中涕灭威、涕灭威砜、涕灭威亚砜的浓度,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);

c_0 ——由标准曲线而得的空白试验中的涕灭威、涕灭威砜、涕灭威亚砜的浓度,单位为纳克每毫升(ng/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

本方法的测定低限为 0.002 mg/kg。

8.2 添加浓度和回收率

添加浓度和回收率数据见表 2。

表 2 不同基质中涕灭威、涕灭威砜、涕灭威亚砜添加浓度及回收率

样品名称	化合物	添加浓度/(μg/kg)	回收率/%
大米	涕灭威	2.0	66.3~77.6
		4.0	77.8~85.4
		10.0	82.4~89.3
	涕灭威砜	2.0	73.9~80.3
		4.0	83.5~88.7
		10.0	82.1~90.2

表 2 (续)

样品名称	化合物	添加浓度/($\mu\text{g}/\text{kg}$)	回收率/%
大米	涕灭威亚砜	2.0	78.6~84.8
		4.0	83.1~88.1
		10.0	86.5~92.1
生姜	涕灭威	2.0	63.5~72.3
		4.0	77.4~81.2
		10.0	80.3~86.4
	涕灭威砜	2.0	79.8~86.6
		4.0	80.5~85.4
		10.0	81.2~89.2
	涕灭威亚砜	2.0	77.8~89.4
		4.0	80.1~84.8
		10.0	84~92.2
菠菜	涕灭威	2.0	68.9~78.4
		4.0	76.1~84
		10.0	80.5~88.5
	涕灭威砜	2.0	77.2~89.7
		4.0	78.6~90.1
		10.0	81.5~95.7
	涕灭威亚砜	2.0	77~92.8
		4.0	78.2~96.0
		10.0	83.1~95.0
番茄	涕灭威	2.0	69.7~85.7
		4.0	73.8~87.9
		10.0	80.9~87.3
	涕灭威砜	2.0	75.6~85.6
		4.0	81.2~86.9
		10.0	80.2~89.8
	涕灭威亚砜	2.0	74.5~84.2
		4.0	80.5~87.4
		10.0	81.2~93.5
大豆	涕灭威	2.0	70.2~84.3
		4.0	70.1~84.0
		10.0	79.5~87.4
	涕灭威砜	2.0	81.2~87.4
		4.0	79.5~86.3

表 2 (续)

样品名称	化合物	添加浓度/($\mu\text{g}/\text{kg}$)	回收率/%
大豆	涕灭威砜	10.0	82~89.3
		2.0	83.1~89.0
		4.0	82.1~89.8
		10.0	85.4~94.6
苹果	涕灭威	2.0	74.3~83.4
		4.0	79.3~85.9
		10.0	77.9~89.0
	涕灭威砜	2.0	77.4~87.7
		4.0	79.2~95.1
		10.0	84.9~94.3
	涕灭威亚砜	2.0	74.7~87.0
		4.0	78.6~86.5
		10.0	80.0~95.5
柑橘	涕灭威	2.0	71.2~83.7
		4.0	78.4~86.7
		10.0	79.8~88.4
	涕灭威砜	2.0	77.6~86.0
		4.0	76.4~85.8
		10.0	83.4~90.5
	涕灭威亚砜	2.0	81.2~95.7
		4.0	80.5~88.5
		10.0	81.4~94.6
茶叶	涕灭威	2.0	73.4~82.0
		4.0	78.9~87.0
		10.0	80.4~88.0
	涕灭威砜	2.0	76.2~82.7
		4.0	77.1~95.2
		10.0	81.8~88.5
	涕灭威亚砜	2.0	78.5~86
		4.0	77.0~89.7
		10.0	83.9~93.1
猪肝	涕灭威	2.0	74.1~83.1
		4.0	74.2~84.2
		10.0	79.8~88.4
	涕灭威砜	2.0	82.7~87.0

表 2 (续)

样品名称	化合物	添加浓度/(μg/kg)	回收率/%
猪肝	涕灭威砜	4.0	79.5~86.3
		10.0	84.5~90.5
	涕灭威亚砜	2.0	82.9~95.7
		4.0	82.1~89.2
		10.0	81.4~94.6
	涕灭威	2.0	71.2~85.7
		4.0	78.2~84.2
		10.0	79.8~88.4
	涕灭威砜	2.0	75.6~87.6
		4.0	82.1~86.9
		10.0	78.1~90.0
鸡肉	涕灭威亚砜	2.0	76.4~93.1
		4.0	82.4~85.2
		10.0	81.4~93.6
		2.0	65.9~78.6
		4.0	77.4~87.7
	涕灭威	10.0	80.9~87.3
		2.0	82.1~86.0
		4.0	83.4~86.0
		10.0	80.2~89.2
	涕灭威砜	2.0	77.3~89.4
		4.0	82.9~87.4
		10.0	81.2~92.2
花生	涕灭威	2.0	71.2~85.7
		4.0	78.2~81.8
		10.0	79.4~86.4
	涕灭威砜	2.0	75.1~87.6
		4.0	79.3~86.9
		10.0	78.1~90
	涕灭威亚砜	2.0	74.2~85.7
		4.0	81.7~88.6
		10.0	81.4~92.3
		2.0	68.9~80.8
		4.0	76.3~84.2
杏仁	涕灭威	10.0	79.8~88.5

表 2 (续)

样品名称	化合物	添加浓度/(μg/kg)	回收率/%
杏仁	涕灭威砜	2.0	77.3~86.2
		4.0	80.5~90.1
		10.0	81.9~95.7
	涕灭威亚砜	2.0	81.5~86.8
		4.0	78.2~93.8
		10.0	83.4~94.8

附录 A
(资料性附录)
液相色谱-串联质谱条件¹⁾

表 A.1 液相流动相梯度程序

时间/min	乙腈/%	0.1%甲酸水溶液/%
0.00	10	90
6.00	90	10
8.00	90	10
8.01	10	90
15.00	10	90

表 A.2 质谱条件

电离方式	ESI+
毛细管电压	4 000 V
源温度	100 ℃
去溶剂温度	300 ℃
锥孔气流	氮气, 10 L/h
碰撞气压	氮气, 206.85 kPa(30 psi)
监测模式	多反应监测(MRM)

表 A.3 多反应监测条件

化合物	母离子	子离子	驻留时间/s	锥孔电压/V	碰撞能量/eV
涕灭威	213	89 ^a	0.20	40	10
		98	0.20	40	5
涕灭威砜	223	86 ^a	0.20	40	10
		81	0.20	40	10
涕灭威亚砜	207	89	0.20	40	3
		132 ^a	0.20	40	2

^a 离子用于定量。

1) 非商业性声明:附录 A 所列参考质谱条件是在 Agilent6410 型液质联用仪上完成的,此处列出试验用仪器型号仅为提供参考,并不涉及商业目的,鼓励标准使用者尝试不同厂家或型号的仪器。

附录 B
(资料性附录)
标准物质选择离子质量色谱图

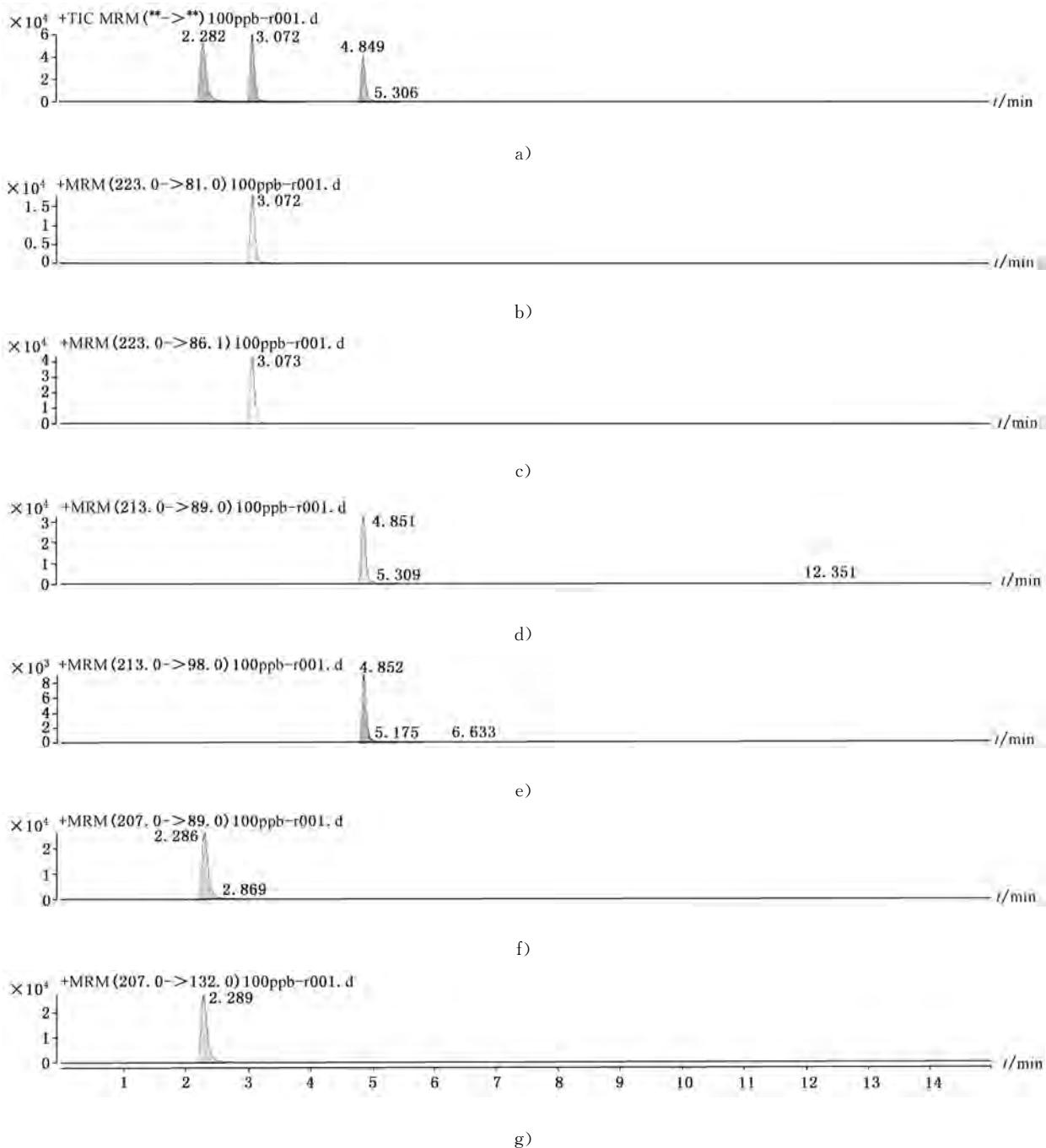


图 B.1 涕灭威及其代谢物标准品液相色谱-质谱/质谱多反应监测色谱图

Foreword

Annex A and B of this standard is an informative annex.

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

This standard was drafted by China Inspection and Quarantine Institute of Science and Technology.

This standard was mainly drafted by Chen Dongdong, Zhu Mingda, Jiang Wenbin, Dai Hanhui, Li Xiao-juan, Peng Tao, Li Shujuan.

Determination of aldicarb, aldicarb-sulfone and aldicarb-sulfoxide residues in food for import and export—LC-MS/MS method

1 Scope

This standard specifies the method of determination of aldicarb, aldicarb sulfoxide, aldicarb sulfone residue in food by HPLC-MS/MS.

This standard is applicable for the determination and confirmation of aldicarb, aldicarb sulfoxide, aldicarb sulfone residue in ginger, tomato, broccoli, rice, peanut, soybeans, almonds, apples, citrus, tea, liver, chicken, milk.

2 Principle

Aldicarb, aldicarb sulfoxide, aldicarb sulfone residue in sample is extracted with acetonitrile, the extraction is passed through SPE column to purify, MRM mode monitoring and determined by HPLC-MS/MS, quantified by external standard method.

3 Reagent and materials

Unless specifically mentioned, all reagents used should be analytical grade. “water” is GB/T 6682 water.

3.1 Acetonitrile: HPLC grade.

3.2 Methanol.

3.3 Dichloromethane.

3.4 N-hexane.

3.5 Anhydrous sodium sulfate; Ignited at 650 °C for 4 h, and stored in desiccator.

3.6 Methanol + Dichloromethane (1 + 99, V/V); Add 1 mL methanol into 99 mL dichloromethane, mix adequately.

- 3.7 0.1% Formic acid solution: Add 1 mL Formic acid then diluted to 1 L with water.
- 3.8 Acetonitrile-0.1% formic acid solution (10 + 90, V/V): Add 10 mL acetonitrile into 90 mL 0.1% formic acid solution, mix adequately.
- 3.9 Aldicarb standard (Aldicarb, CAS No. 116-06-3): Purity greater than or equal to 98%.
- 3.10 Aldicarb sulfone standard (Aldicarb-sulfone, CAS No. 1646-88-4): Purity greater than or equal to 98%.
- 3.11 Aldicarb sulfone standard (Aldicarb-sulfoxide, CAS No. 1646-87-3): Purity greater than or equal to 98%.
- 3.12 Stock standard solution: Accurately weight an adequate amount of standard dissolved with methanol of 100 $\mu\text{g}/\text{mL}$. Stored the stock standard solution avoid light at the temperature 0 $^{\circ}\text{C}$ ~ 4 $^{\circ}\text{C}$.
- 3.13 Blank matrix extract solution: Extract the test samples which there are no aldicarb, aldicarb sulfoxide, aldicarb sulfone residue according to the 6.1 and 6.2 to prepare the blank matrix extract solution.
- 3.14 Standard working solution: According to the requirement, dilute standard solution with blank matrix extract solution. The concentration of the solution is 10.0 ng/mL , 20.0 ng/mL , 50.0 ng/mL , 100 ng/mL , 200 ng/mL . Stored the standard working solution avoid light at the temperature 0 $^{\circ}\text{C}$ ~ 4 $^{\circ}\text{C}$.
- 3.15 Amido SPE column: 500 mg, 3 mL, or equivalent.
- 3.16 0.45 μm Millipore.

4 Apparatus and equipment

- 4.1 Liquid chromatography-mass spectrometer equipment, triple quadrupole mass spectrometry detector, equipped with electrospray (ESI) ion source, or equivalent.
- 4.2 Homogenizer.
- 4.3 Balance, sensitivity: 0.01 g and 0.1 mg.
- 4.4 Shaker.

4.5 Rotary evaporator.

4.6 Vortex oscillator.

4.7 SPE vacuum container.

4.8 Nitrogen evaporator.

5 Sample preparation and storage

5.1 Sample preparation

5.1.1 Rice, peanut, soybean, almond, tea: Representative sample 500 g, after homogenization using heterogeneous devices, put into sealed clean containers and labeled, -18 °C in refrigerator to preserve.

5.1.2 Ginger, tomatoes, broccoli, apple, citru, pig liver, chicken: Representative sample 500 g, use organize broken machine to broke, then put into sealed clean containers and labeled, -18 °C in refrigerator to preserve.

5.2 Sample storage

The test samples of cereals and nuts should be stored in 0 °C ~4 °C, avoiding from the light; the other samples should be stored below -18 °C, avoiding from the light.

In the course of sample preparation and sample storage, precaution must be taken to avoid contamination.

6 Procedure

6.1 Extraction

5 g (accurating to 0.01 g) of test sample is weighed and put into a 50 mL centrifuge tubes, add 20 mL acetonitrile, homogenize the sample for 2 min and shake the tube for 20 min. Transfer the whole extract solution through anhydrous sodium sulfate into separatory funnel. The extraction is repeated with 20 mL of acetonitrile, combine the extract solution to the sub-liquid funnel, intermixed, then add 20 mL acetonitrile saturated with *n*-hexane, shake for 10 min, wipe off hexane, transfer acetonitrile into the conical flask and evaporate to dry in a water bath under 40 °C. The residue is redissolved in 2 mL methanol + dichloromethane (1+99, *V/V*) (3.6).

6.2 Clean up

Transfer the extract solution of 6.1 to the amido SPE column (3.8), wash. Elution by methanol + dichloromethane (1+99, V/V) (3.6), collect the outflow and eluate. Nitrogen evaporator to dryness, add 1 mL mobile phase, through 0.45 μm millipore ready for HPLC-MS/MS determination.

6.3 Determination

6.3.1 LC-MS/MS operation condition

- a) Liquid chromatography-tandem mass spectrometry;
- b) LC column: C₁₈, 100 mm × 2.1 (i. d.) mm, 3.5 μm ;
- c) Mobile phase: Acetonitrile, formic acid solution (3.7). Gradient program, see annex A;
- d) Injector volume: 20 μL ;
- e) Column temperature: 30 °C;
- f) Flow rate: 0.3 mL/min. Or under instrument conditions optimize;
- g) Ion source: ESI ion source;
- h) Scan mode: Positive ion;
- i) Detect mode: Multiple reaction monitoring (MRM).

6.3.2 Qualitative and quantitative determination

According to the method of 6.3.1, detect the residues of aldicarb, aldicarb sulfoxide, aldicarb sulfone in the test sample solution, the standard working solution. The response of aldicarb, aldicarb sulfoxide, aldicarb sulfone 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 (3.13) to suitable concentration. Under the above chromatograph conditions, reconstituted ion chromatogram of standard working solution is listed in annex B figure B.1. 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/%	Permitted tolerances/%
>50	± 20
>20 to 50	± 25
>10 to 20	± 30
≤10	± 50

6.4 Blank test

The operation of blank test is the same as above, except without sample.

7 Calculation and expression of result

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

Where:

X—the residue content of aldicarb, aldicarb sulfoxide, aldicarb sulfone in the test sample, mg/kg;

c—the concentration of aldicarb, aldicarb sulfoxide, aldicarb sulfone in the test sample calculated by calibration curve, ng/mL;

V —the final volume of sample solution, mL;

c_0 —the concentration of aldicarb, aldicarb sulfoxide, aldicarb sulfone in the blank test calculated by calibration curve, ng/mL;

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

8 Limit of determination and recovery

8.1 Limit of determination

The limit of determination of this method is 0.002 mg/kg.

8.2 Add concentration and recovery

Add concentration and recovery see table 2.

Table 2—Recovery

Sample	Compound	Add concentration/($\mu\text{g}/\text{kg}$)	Recovery/%
rice	aldicarb	2.0	66.3~77.6
		4.0	77.8~85.4
		10.0	82.4~89.3
	aldicarb sulfoxide	2.0	73.9~80.3
		4.0	83.5~88.7
		10.0	82.1~90.2
	aldicarb sulfone	2.0	78.6~84.8
		4.0	83.1~88.1
		10.0	86.5~92.1
ginger	aldicarb	2.0	63.5~72.3
		4.0	77.4~81.2
		10.0	80.3~86.4
	aldicarb sulfoxide	2.0	79.8~86.6
		4.0	80.5~85.4
		10.0	81.2~89.2
	aldicarb sulfone	2.0	77.8~89.4
		4.0	80.1~84.8
		10.0	84~92.2
broccoli	aldicarb	2.0	68.9~78.4
		4.0	76.1~84
		10.0	80.5~88.5
	aldicarb sulfoxide	2.0	77.2~89.7
		4.0	78.6~90.1
		10.0	81.5~95.7
	aldicarb sulfone	2.0	77~92.8
		4.0	78.2~96.0
		10.0	83.1~95.0
tomato	aldicarb	2.0	69.7~85.7
		4.0	73.8~87.9
		10.0	80.9~87.3
	aldicarb sulfoxide	2.0	75.6~85.6
		4.0	81.2~86.9
		10.0	80.2~89.8
	aldicarb sulfone	2.0	74.5~84.2
		4.0	80.5~87.4

Table 2 (continued)

Sample	Compound	Add concentration/($\mu\text{g}/\text{kg}$)	Recovery/%
tomato	aldicarb sulfone	10.0	81.2~93.5
soybean	aldicarb	2.0	70.2~84.3
		4.0	70.1~84.0
		10.0	79.5~87.4
		2.0	81.2~87.4
	aldicarb sulfoxide	4.0	79.5~86.3
		10.0	82~89.3
		2.0	83.1~89.0
	aldicarb sulfone	4.0	82.1~89.8
		10.0	85.4~94.6
apple	aldicarb	2.0	74.3~83.4
		4.0	79.3~85.9
		10.0	77.9~89.0
	aldicarb sulfoxide	2.0	77.4~87.7
		4.0	79.2~95.1
		10.0	84.9~94.3
	aldicarb sulfone	2.0	74.7~87.0
		4.0	78.6~86.5
		10.0	80.0~95.5
citrus	aldicarb	2.0	71.2~83.7
		4.0	78.4~86.7
		10.0	79.8~88.4
	aldicarb sulfoxide	2.0	77.6~86.0
		4.0	76.4~85.8
		10.0	83.4~90.5
	aldicarb sulfone	2.0	81.2~95.7
		4.0	80.5~88.5
		10.0	81.4~94.6
tea	aldicarb	2.0	73.4~82.0
		4.0	78.9~87.0
		10.0	80.4~88.0
	aldicarb sulfoxide	2.0	76.2~82.7
		4.0	77.1~95.2
		10.0	81.8~88.5
	aldicarb sulfone	2.0	78.5~86

Table 2 (continued)

Sample	Compound	Add concentration/($\mu\text{g}/\text{kg}$)	Recovery/%
tea	aldicarb sulfone	4.0	77.0~89.7
		10.0	83.9~93.1
liver	aldicarb	2.0	74.1~83.1
		4.0	74.2~84.2
		10.0	79.8~88.4
	aldicarb sulfoxide	2.0	82.7~87.0
		4.0	79.5~86.3
		10.0	84.5~90.5
	aldicarb sulfone	2.0	82.9~95.7
		4.0	82.1~89.2
		10.0	81.4~94.6
chicken	aldicarb	2.0	71.2~85.7
		4.0	78.2~84.2
		10.0	79.8~88.4
	aldicarb sulfoxide	2.0	75.6~87.6
		4.0	82.1~86.9
		10.0	78.1~90.0
	aldicarb sulfone	2.0	76.4~93.1
		4.0	82.4~85.2
		10.0	81.4~93.6
milk	aldicarb	2.0	65.9~78.6
		4.0	77.4~87.7
		10.0	80.9~87.3
	aldicarb sulfoxide	2.0	82.1~86.0
		4.0	83.4~86.0
		10.0	80.2~89.2
	aldicarb sulfone	2.0	77.3~89.4
		4.0	82.9~87.4
		10.0	81.2~92.2
peanut	aldicarb	2.0	71.2~85.7
		4.0	78.2~81.8
		10.0	79.4~86.4
	aldicarb sulfoxide	2.0	75.1~87.6
		4.0	79.3~86.9
		10.0	78.1~90

Table 2 (continued)

Sample	Compound	Add concentration/($\mu\text{g}/\text{kg}$)	Recovery/%
peanut	aldicarb sulfone	2. 0	74. 2~85. 7
		4. 0	81. 7~88. 6
		10. 0	81. 4~92. 3
almond	aldicarb	2. 0	68. 9~80. 8
		4. 0	76. 3~84. 2
		10. 0	79. 8~88. 5
	aldicarb sulfoxide	2. 0	77. 3~86. 2
		4. 0	80. 5~90. 1
		10. 0	81. 9~95. 7
	aldicarb sulfone	2. 0	81. 5~86. 8
		4. 0	78. 2~93. 8
		10. 0	83. 4~94. 8

Annex A
(Information)
Condition¹⁾

Table A. 1—Liquid mobile phase gradient program¹⁾

Time/min	Acetonitrile/%	0. 1% Formic acid solution/%
0. 00	10	90
6. 00	90	10
8. 00	90	10
8. 01	10	90
15. 00	10	90

Table A. 2—The condition parameters of the mass detector

Ion source	ESI +
Capillary voltage	4. 0 kV
Source temperature	100 °C
Desolvation temperature	300 °C
Cone gas flow	Nitrogen, 10 L/h
Collision gas pressure	Nitrogen, 206. 85 kPa(30 psi)
Scanning model	multiple reaction monitor (MRM)

Table A. 3—MRM condition

Compound	Precursor ion (m/z)	Production (m/z)	Dwell time/s	Cone voltage/V	Collision energy/eV
aldicarb	213	89 ^a	0. 20	40	10
		98	0. 20	40	5
Aldicarb sulfone	223	86 ^a	0. 20	40	10
		81	0. 20	40	10
aldicarb sulfoxide	207	89	0. 20	40	3
		132 ^a	0. 20	40	2

^a quantified ion pair.

1) Non-commercial statement: the reference mass parameters in annex A are accomplished by agilent 6410 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.

Annex B
(Information)

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

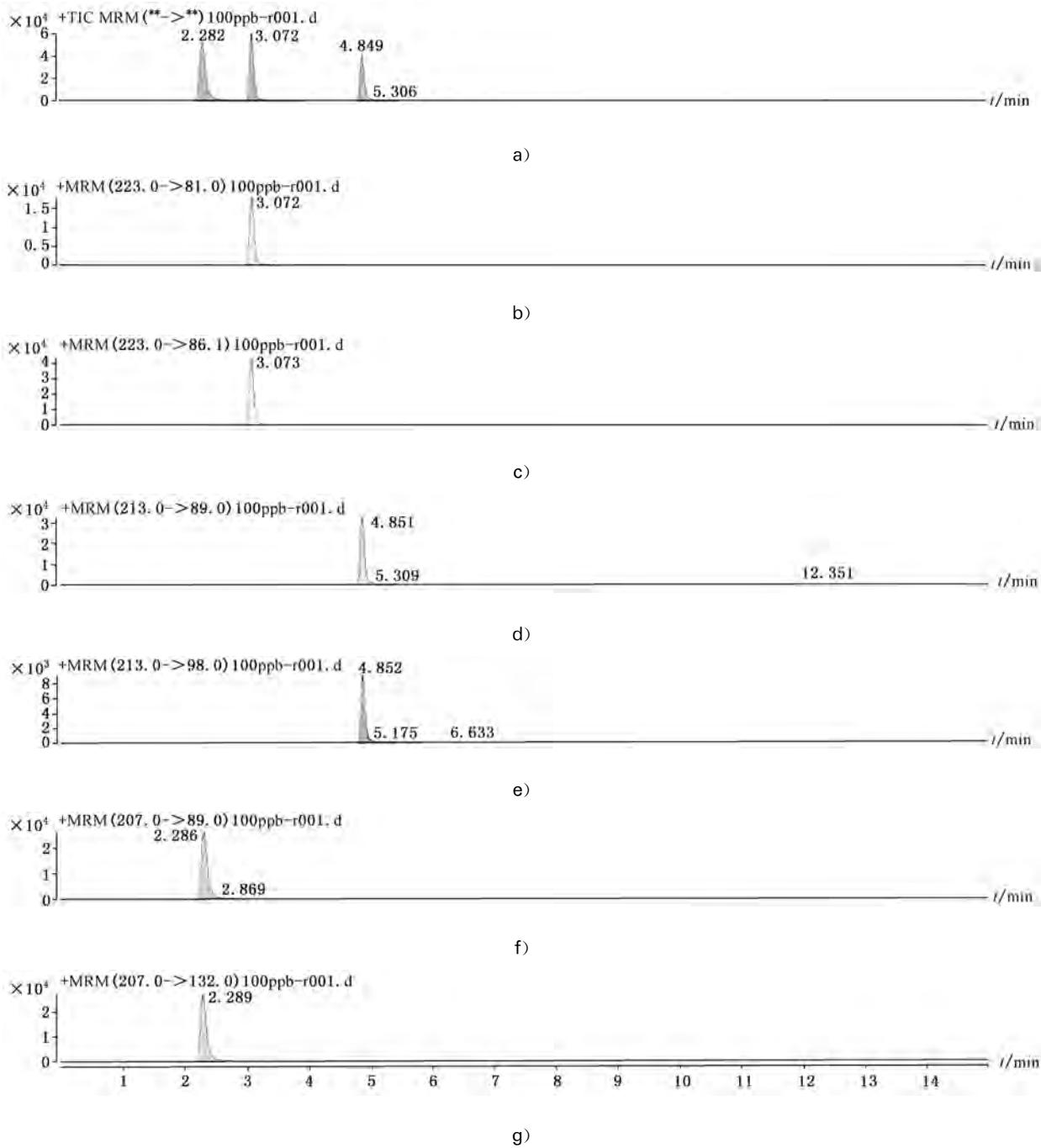


Figure B. 1—Aldicarb and its metabolites in standard LC-MS/MS multiple reaction monitoring chromatogram

SN/T 2441—2010

中华人民共和国出入境检验检疫
行业标准
进出口食品中涕灭威、涕灭威砜、
涕灭威亚砜残留量检测方法
液相色谱-质谱/质谱法

SN/T 2441—2010

*

中国标准出版社出版
北京复兴门外三里河北街 16 号
邮政编码：100045

网址 www.spc.net.cn
电话：68523946 68517548

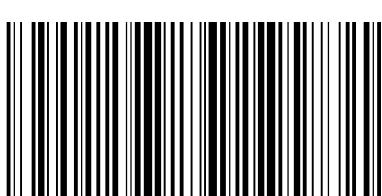
中国标准出版社秦皇岛印刷厂印刷

*

开本 880×1230 1/16 印张 1.75 字数 38 千字
2010 年 5 月第一版 2010 年 5 月第一次印刷
印数 1—1 600

*

书号：155066 · 2-20728 定价 27.00 元



SN/T 2441-2010