SN

中华人民共和国出入境检验检疫行业标准

SN/T 0134—2010
代替 SN 0134—1992,SN 0490—1995,SN 0534—1996,SN 0582—1996

进出口食品中杀线威等12种氨基甲酸酯类农药残留量的检测方法 液相色谱-质谱/质谱法

Determination for pesticide residues of 12 kinds of carbamates including oxamyl in foods for import and export—LC-MS/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。

本标准代替了 SN 0134—1992《出口粮谷中甲萘威、克百威残留量检验方法》、SN 0490—1995《出口粮谷中异丙威残留量检验方法》、SN 0534—1996《出口粮谷中仲丁威残留量检验方法》、SN 0582—1996《出口粮谷及油籽中灭多威残留量检验方法》。本标准与 SN 0134—1992、SN 0490—1995、SN 0534—1996 和 SN 0582—1996 相比，主要技术变化如下：

——本标准扩大了检测样品基质至 13 种；

——增加了检测项目至 12 种；

——采用了液相色谱-质谱/质谱法；

——提高了方法灵敏度；

——增加了定性手段；

——优化了前处理方法。

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：中华人民共和国吉林出入境检验检疫局、中华人民共和国湖南出入境检验检疫局、中国检验检疫科学研究院。

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本标准所代替标准的历次版本发布情况为：

——ZBB 22017—1998、SN 0134—1992；

——SN 0490—1995；

——SN 0534—1996；

——SN 0582—1996。

进出口食品中杀线威等12种氨基甲酸酯类农药残留量的检测方法 液相色谱-质谱/质谱法

1 范围

本标准规定了食品中杀线威、灭多威、抗蚜威、涕灭威、速灭威、噁虫威、克百威、甲萘威、乙硫甲威、异丙威、乙霉威和仲丁威等12种氨基甲酸酯类农药残留量的液相色谱-质谱/质谱检测方法。

本标准适用于玉米、糙米、大麦、白菜、大葱、小麦、大豆、花生、苹果、柑橘、牛肝、鸡肾和蜂蜜中杀线威、灭多威、抗蚜威、涕灭威、速灭威、噁虫威、克百威、甲萘威、乙硫甲威、异丙威、乙霉威、仲丁威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，仅所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

试样用乙腈提取(蜂蜜用丙酮提取，二氯甲烷液-液分配)，乙腈饱和的正己烷液-液分配，经活性炭和氟罗里硅土固相柱净化后，液相色谱-质谱/质谱仪检测和确证，外标法定量。

4 试剂和材料

除另有规定外，所用试剂均为分析纯，水为GB/T 6682规定的一级水。

4.1 乙腈：残留级。

4.2 丙酮：残留级。

4.3 甲醇：高效液相色谱级。

4.4 正己烷：残留级。

4.5 二氯甲烷：残留级。

4.6 无水硫酸钠：经650 ℃灼烧4 h，储于密封容器中备用。

4.7 丙酮-正己烷(3+7，体积比)：量取30 mL丙酮和70 mL正己烷，混匀。

4.8 乙腈饱和的正己烷：取少量乙腈加入正己烷中，剧烈振摇，并继续加入乙腈至出现明显分层，静置备用。

4.9 杀线威、克百威、猛杀威、灭多威、甲萘威、速灭威、涕灭威、异丙威、乙硫甲威、抗蚜威、噁虫威、乙霉威等农药标准物质，纯度均≥98.5%。

4.10 标准储备溶液：分别准确称取适量的各种氨基甲酸酯标准物质(4.7)，用甲醇配制成浓度为100 μg/mL的标准储备溶液。该溶液于−18 ℃保存。

4.11 混合标准中间溶液：分别准确吸取适量的杀线威等12种氨基甲酸酯类农药标准储备溶液(4.8)，用甲醇配制成浓度为10 μg/mL的混合标准中间溶液。该溶液于－18 ℃保存。

4.12 混合标准工作溶液：标准工作溶液根据需要使用前吸取适量的混合标准中间溶液，用空白样品基质溶液配制成适当浓度的混合标准工作溶液。该溶液在0 ℃～4 ℃冰箱中保存，现用现配。

4.13 氟罗里硅土固相萃取柱：Florisil，1 000 mg，6 mL，或相当者。

4.14 活性炭固相萃取柱：Carbon，500 mg，6 mL，或相当者。

4.15 滤膜：有机滤膜，0.2 μm。

5 仪器与设备

5.1 液相色谱-质谱/质谱仪：配备电喷雾离子源(ESI)。

5.2 天平：感量0.1 mg和0.01 g。

5.3 组织捣碎机。

5.4 粉碎机。

5.5 均质器。

5.6 涡旋混合器。

5.7 离心机：4 000 r/min。

5.8 旋转蒸发器。

5.9 聚四氟乙烯离心管：50 mL。

5.10 锥形瓶：250 mL。

5.11 浓缩瓶：250 mL。

6 试样制备与保存

6.1 试样制备

6.1.1 玉米、糙米、大豆、花生

取代表性样品约500 g，用粉碎机粉碎，混匀，装入洁净容器，密封，标明标记。

6.1.2 柑桔、苹果、大葱、白菜、洋葱、大蒜

取代表性样品约500 g，将其可食用部分(不可用水洗)切碎后，用捣碎机将样品加工成浆状，混匀，装入洁净容器，密封，标明标记。

6.1.3 牛肝、鸡肾

牛肝取代表性样品约1 kg，鸡肾取代表性样品约100 g，经捣碎机充分捣碎均匀，装入洁净容器，密封，标明标记。

6.1.4 蜂蜜

取代表性样品约500 g，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。装入洁净容器，密封，标明标记。

6.2 试样保存

粮谷类、坚果类、蜂蜜试样于0 ℃～4 ℃保存；其他类试样于－18 ℃以下冷冻保存。在抽样及制样

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 测定步骤

7.1 提取

7.1.1 玉米、糙米、白菜、大葱、大麦、小麦、大豆、花生、苹果、柑橘、牛肝、鸡肾

称取试样 5 g(精确到 0.01 g)于 50 mL 离心管中，加入 20 mL 乙腈，均质提取 1 min，于 4 000 r/min 离心 3 min。将上清液倾入 50 mL 离心管中，残渣再用 10 mL 乙腈重复提取 1 次，合并上清液于 50 mL 离心管中，加入 10 mL 正己烷，旋涡混匀，弃去正己烷(大豆、花生再加入 10 mL 正己烷，旋涡混匀，弃去正己烷)。将乙腈层转入 250 mL 浓缩瓶中，于 40 ℃水浴中浓缩至近干，加 2 mL 丙酮-正己烷(3＋7)溶解。

7.1.2 蜂蜜

称取试样 15 g(精确到 0.01 g)于 250 mL 锥形瓶中，加入 30 mL 水，40 ℃水浴振荡 15 min。再加入 10 mL 丙酮，将其转入 250 mL 分液漏斗中，用 40 mL 二氯甲烷分数次洗涤锥形瓶，洗涤液移入另一分液漏斗，用力振摇 8 次，静置分层(小心排气)。下层有机相经无水硫酸钠脱水，收集于 250 mL 浓缩瓶中。再用 5 mL 丙酮和 40 mL 二氯甲烷振荡提取上层蜂蜜 1 min，重复两次，合并上述收集液，40 ℃水浴中浓缩至近干，加 2 mL 丙酮-正己烷(3＋7)溶解。

7.2 净化

自上而下将活性炭固相萃取柱(4.14)与氟罗里硅土固相萃取柱(4.13)串联连接，使用前用 20 mL 丙酮-正己烷(3＋7)预淋洗，弃去流出液。将 7.1.1 或 7.1.2 制备的样品提取液倾入柱中，再用 2 mL 丙酮-正己烷(3＋7)润洗浓缩瓶并倾入柱中。用 20 mL 丙酮-正己烷(3＋7)进行洗脱(流速不超过 2 mL/min)。收集全部洗脱液于 250 mL 浓缩瓶中，于 40 ℃水浴中浓缩至近干。用甲醇溶解并定容至 2.0 mL，过滤膜(4.15)，供液相色谱-质谱/质谱仪测定和确证。

7.3 测定

7.3.1 液相色谱条件

液相色谱条件如下：

a) 色谱柱：Zorbax C_{18}，150 mm×2.1 mm(内径)，5 μm，或相当者；

b) 柱温：40 ℃；

c) 流速：0.2 mL/min；

d) 进样量：5 μL；

e) 流动相及梯度洗脱条件见表 1。

表 1 流动相及梯度洗脱条件

时间 min	流速 μL/min	甲醇 %	水(1%甲酸) %	水 %
0.00	200	20.0	20.0	60.0
2.00	200	20.0	20.0	60.0
3.00	200	60.0	40.0	0
20.00	200	75.0	25.0	0
20.10	200	20.0	40.0	40.0
25.00	200	20.0	40.0	40.0

7.3.2 质谱条件

a) 电离方式:电喷雾电离(ESI);
b) 扫描方式:正离子扫描;
c) 检测方式:多反应监测(MRM);
d) 电喷雾电压:3.0 kV;
e) 雾化气、气帘气、辅助加热气、碰撞气均为高纯氮气,适用前应调节各气体流量以使质谱灵敏度达到检测要求;
f) 辅助气温度;
g) 定性离子对、定量离子对、采集时间、去簇电压及碰撞能量等参数参见附录 A。

7.3.3 液相色谱-质谱/质谱检测及确证

根据样液中被测物含量情况,选定浓度相近的标准工作溶液,标准工作溶液和待测样液中杀线威等 12 种氨基甲酸酯类农药的响应值均应在仪器检测的线性范围内。标准工作溶液与样液等体积参插进样测定。

标准溶液及样液均按 7.3.1 和 7.3.2 规定的条件进行测定,如果样液中与标准溶液相同的保留时间有峰出现,则对其进行确证。经确证分析被测物质量色谱峰保留时间与标准物质相一致,并且在扣除背景后的样品谱图中,所选择的离子均出现;同时所选择离子的丰度比与标准样物质相关离子的相对丰度一致,相似度在允许偏差之内(见表 2),被确证的样品可判定为阳性检出。杀线威等 12 种氨基甲酸酯类农药标准物质的液相色谱-质谱/质谱总离子流图参见附录 B 中图 B.1。

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

7.4 空白试验

除不称取试样外,均按上述步骤进行。

7.5 结果计算和表述

用色谱数据处理机或按式(1)计算试样中杀线威等 12 种氨基甲酸酯类农药残留量:

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

式中:

X_i ——试样中农药 i 残留量,单位为毫克每千克(mg/kg);

A_i ——样液中杀线威等 12 种氨基甲酸酯类农药的峰面积(或峰高);

c_i ——标准工作液中农药 i 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);

A_{is}——标准工作液中农药 i 的峰面积(或峰高);

m ——最终样液所代表的试样质量,单位为克(g)。

注:计算结果应扣除空白值。

8 测定低限和回收率

8.1 测定低限

本方法的测定低限参见附录C。

8.2 添加浓度范围及回收率

本方法添加浓度及回收率参见附录C中表C.1。

附 录 A
（资料性附录）
质 谱 条 件[1)]

质谱条件：

a） 电离方式：电喷雾电离（ESI）；

b） 电喷雾电压（IS）：5 500 V；

c） 雾化气压力（GS1）：20 psi；

d） 气帘气压力（CUR）：20 psi；

e） 辅助气流速（GS2）：30 psi；

f） 离子源温度（TEM）：550 ℃；

g） 碰撞气（CAD）：6 mL/min；

h） 碰撞池出口电压（CXP）：10 V；

i） 碰撞池入口电压（EP）：10 V；

j） 扫描方式：正离子扫描；

k） 检测方式：多反应监测（MRM）；

l） 质谱参数见表 A.1。

表 A.1 杀线威等 12 种氨基甲酸酯类农药的质谱参数（多反应监测条件）

被测物名称	英文名称	母离子 (m/z)	子离子 (m/z)	采集时间 ms	去簇电压 V	碰撞能量 eV	保留时间 min
杀线威	oxamyl	242.1	121.3	30	73	18	7.43
			72.3*	30	73	35	
灭多威	methomyl	163.0	106.1	30	58	12	9.28
			88.1*	30	58	12	
抗蚜威	pirimicarb	238.7	182.2	30	66	22	11.47
			72.2*	30	66	36	
涕灭威	aldicarb	208.1	116.2	30	55	10	12.28
			89.2*	30	55	21	
速灭威	metolcarb	166.2	109.1*	30	62	14	12.88
			94.1	30	62	42	
噁虫威	bendiocarb	224.4	167.1	30	65	13	13.37
			109.0*	30	65	25	
克百威	carbofuran	222.3	165.0	30	70	16	13.40
			123.2*	30	70	30	

1） 非商业声明：附录 A 所列参考质谱条件是在 API 4000 液相色谱-质谱/质谱仪上完成的，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。

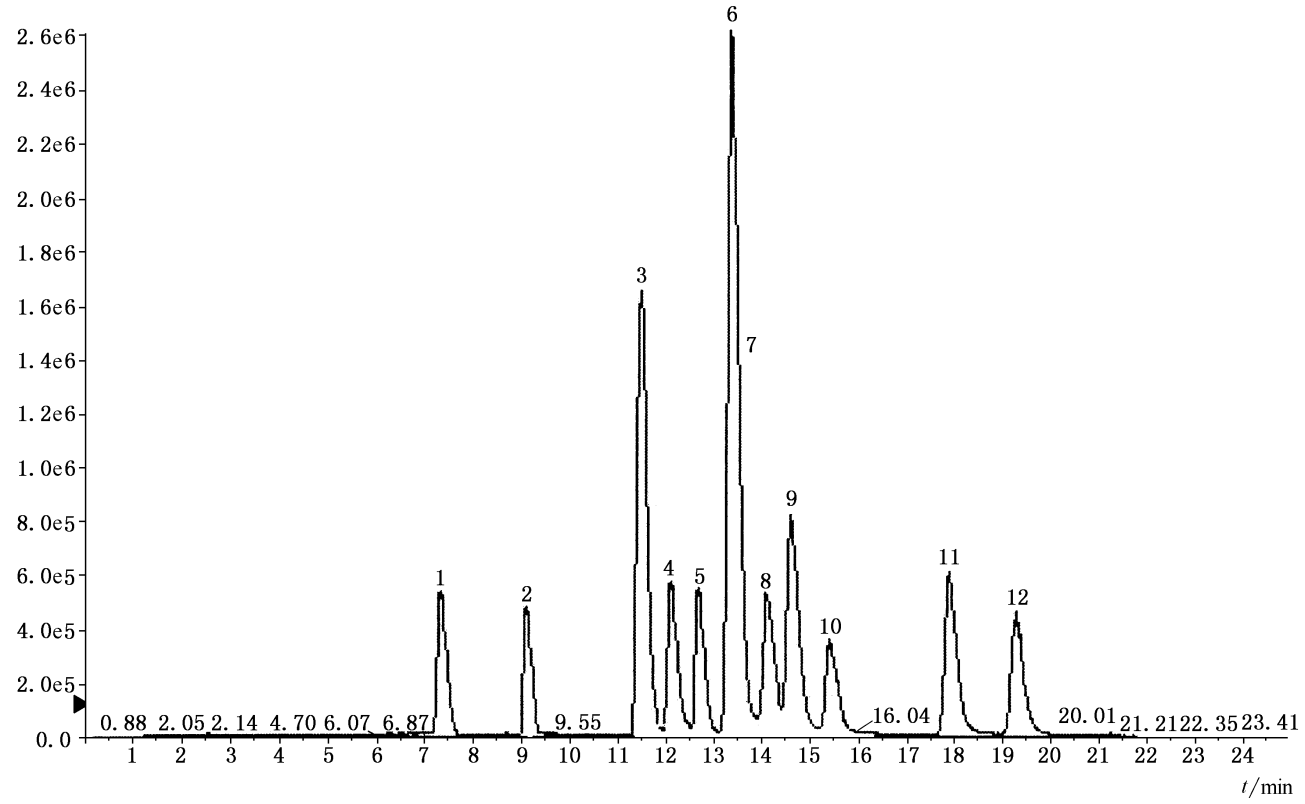
表 A.1（续）

被测物名称	英文名称	母离子（m/z）	子离子（m/z）	采集时间 ms	去簇电压 V	碰撞能量 eV	保留时间 min
甲萘威	carbaryl	202.2	145.2	30	55	12	14.15
			127.2*	30	55	38	
乙硫甲威	ethiofencarb	226.2	164.0*	30	53	11	14.61
			107.0	30	53	19	
异丙威	isoprocarb	194.2	137.2*	30	70	12	15.41
			95.3	30	70	20	
乙霉威	diethofencarb	268.3	180.0*	30	55	23	17.87
			152.0	30	55	30	
仲丁威	fenobucarb	208.4	151.2*	30	70	12	19.28
			109.1	30	70	21	

注 1：“*”的离子用于定量。

注 2：对于不同质谱仪器，仪器参数可能存在差异，测定前应将质谱参数优化到最佳。

附　录　B
（资料性附录）
杀线威等 12 种氨基甲酸酯类农药总离子流图



1——杀线威；　　7——克百威；
2——灭多威；　　8——甲萘威；
3——抗蚜威；　　9——乙硫甲威；
4——涕灭威；　　10——异丙威；
5——速灭威；　　11——乙霉威；
6——噁虫威；　　12——仲丁威。

图 B.1　杀线威等 12 种氨基甲酸酯类农药(0.1 μg/mL)总离子流

附 录 C
（资料性附录）
方法测定低限和确证低限及回收率范围

表 C.1 测定低限和确证低限及回收率范围

药品名称	样品名称	添加水平 mg/kg	测定和确证低限 mg/kg	回收率范围 %
杀线威	糙米	0.010	0.01	81.0～93.0
		0.100	0.01	82.5～94.9
		1.000	0.01	89.4～108.3
	玉米	0.010	0.01	84.0～98.0
		0.100	0.01	79.2～95.4
		1.000	0.01	81.6～99.7
	白菜	0.010	0.01	83.0～99.0
		0.100	0.01	84.3～97.0
		1.000	0.01	81.2～102.4
	大葱	0.010	0.01	80.0～97.0
		0.100	0.01	84.7～99.1
		1.000	0.01	81.9～92.4
	大麦	0.010	0.01	81.0～103.0
		0.100	0.01	76.8～95.7
		1.000	0.01	83.2～103.6
	小麦	0.010	0.01	86.0～102.0
		0.100	0.01	82.5～98.0
		1.000	0.01	88.0～108.4
	苹果	0.010	0.01	82.0～96.0
		0.100	0.01	86.3～98.7
		1.000	0.01	83.6～102.1
	柑橘	0.010	0.01	85.0～101.0
		0.100	0.01	87.2～110.0
		1.000	0.01	84.3～99.2
	大豆	0.010	0.01	80.0～93.0
		0.100	0.01	82.6～96.7
		1.000	0.01	83.1～102.6
	花生	0.010	0.01	79.0～95.0
		0.100	0.01	86.4～102.3
		1.000	0.01	79.2～96.1
	牛肝	0.010	0.01	86.0～105.0
		0.100	0.01	89.4～106.0
		1.000	0.01	84.3～99.6
	鸡肾	0.010	0.01	83.0～103.0
		0.100	0.01	81.0～96.1
		1.000	0.01	82.1～99.2
	蜂蜜	0.010	0.01	78.0～92.0
		0.100	0.01	82.6～99.7
		1.000	0.01	83.6～97.8

表 C.1（续）

药品名称	样品名称	添加水平 mg/kg	测定和确证低限 mg/kg	回收率范围 %
灭多威	糙米	0.010	0.01	86.0～100.0
		0.100	0.01	81.1～99.3
		1.000	0.01	86.8～102.7
	玉米	0.010	0.01	80.0～94.0
		0.100	0.01	85.7～97.6
		1.000	0.01	85.5～101.2
	白菜	0.010	0.01	80.0～97.0
		0.100	0.01	88.2～106.0
		1.000	0.01	88.5～100.4
	大葱	0.010	0.01	82.0～97.0
		0.100	0.01	79.6～98.2
		1.000	0.01	78.5～96.4
	大麦	0.010	0.01	81.0～97.0
		0.100	0.01	82.9～97.3
		1.000	0.01	82.4～103.7
	小麦	0.010	0.01	83.0～97.0
		0.100	0.01	85.2～106.0
		1.000	0.01	81.1～95.7
	苹果	0.010	0.01	80.0～94.0
		0.100	0.01	87.3～103.4
		1.000	0.01	81.3～99.6
	柑橘	0.010	0.01	78.0～95.0
		0.100	0.01	81.4～96.7
		1.000	0.01	80.2～99.3
	大豆	0.010	0.01	86.0～102.0
		0.100	0.01	82.3～95.7
		1.000	0.01	80.1～97.3
	花生	0.010	0.01	78.0～95.0
		0.100	0.01	77.8～95.3
		1.000	0.01	80.3～97.7
	牛肝	0.010	0.01	89.0～104.0
		0.100	0.01	79.5～96.0
		1.000	0.01	81.6～99.1
	鸡肾	0.010	0.01	79.0～95.0
		0.100	0.01	86.7～105.0
		1.000	0.01	88.6～100.3
	蜂蜜	0.010	0.01	78.0～92.0
		0.100	0.01	82.6～99.7
		1.000	0.01	83.6～97.8
抗蚜威	糙米	0.010	0.01	78.0～97.0
		0.100	0.01	81.9～98.0
		1.000	0.01	83.4～99.1
	玉米	0.010	0.01	78.0～96.0
		0.100	0.01	89.2～103.0
		1.000	0.01	83.6～97.1

表 C.1（续）

药品名称	样品名称	添加水平 mg/kg	测定和确证低限 mg/kg	回收率范围 %
抗蚜威	白菜	0.010	0.01	76.0～93.0
		0.100	0.01	82.2～95.1
		1.000	0.01	81.5～96.3
	大葱	0.010	0.01	79.0～93.0
		0.100	0.01	83.4～97.6
		1.000	0.01	82.8～96.4
	大麦	0.010	0.01	76.0～92.0
		0.100	0.01	78.6～95.3
		1.000	0.01	84.8～103.7
	小麦	0.010	0.01	85.0～98.0
		0.100	0.01	78.6～95.8
		1.000	0.01	80.0～93.6
	苹果	0.010	0.01	80.0～95.0
		0.100	0.01	81.2～98.8
		1.000	0.01	88.4～100.9
	柑橘	0.010	0.01	81.0～94.0
		0.100	0.01	80.9～97.7
		1.000	0.01	82.6～96.1
	大豆	0.010	0.01	82.0～96.0
		0.100	0.01	84.0～107.0
		1.000	0.01	80.2～97.3
	花生	0.010	0.01	82.0～96.0
		0.100	0.01	84.3～97.3
		1.000	0.01	83.2～96.3
	牛肝	0.010	0.01	76.0～95.0
		0.100	0.01	80.5～96.4
		1.000	0.01	85.3～102.6
	鸡肾	0.010	0.01	82.0～95.0
		0.100	0.01	77.6～98.2
		1.000	0.01	81.2～96.2
	蜂蜜	0.010	0.01	76.0～93.0
		0.100	0.01	83.0～95.6
		1.000	0.01	85.3～101.5
涕灭威	糙米	0.010	0.01	81.0～95.0
		0.100	0.01	79.3～97.7
		1.000	0.01	80.1～97.7
	玉米	0.010	0.01	79.0～95.0
		0.100	0.01	78.8～94.0
		1.000	0.01	83.1～99.0

表 C.1（续）

药品名称	样品名称	添加水平 mg/kg	测定和确证低限 mg/kg	回收率范围 %
涕灭威	白菜	0.010	0.01	82.0～97.0
		0.100	0.01	78.6～91.5
		1.000	0.01	82.0～97.7
	大葱	0.010	0.01	81.0～97.0
		0.100	0.01	83.3～100.8
		1.000	0.01	79.8～93.5
	大麦	0.010	0.01	81.0～95.0
		0.100	0.01	81.1～95.6
		1.000	0.01	85.3～95.7
	小麦	0.010	0.01	77.0～95.0
		0.100	0.01	84.5～98.3
		1.000	0.01	83.6～95.8
	苹果	0.010	0.01	76.0～95.0
		0.100	0.01	81.9～98.8
		1.000	0.01	77.5～90.7
	柑橘	0.010	0.01	76.0～93.0
		0.100	0.01	81.9～98.0
		1.000	0.01	77.6～93.1
	大豆	0.010	0.01	80.0～97.0
		0.100	0.01	78.4～93.7
		1.000	0.01	85.8～100.4
	花生	0.010	0.01	82.0～109.0
		0.100	0.01	82.1～94.6
		1.000	0.01	78.7～93.1
	牛肝	0.010	0.01	80.0～97.0
		0.100	0.01	80.2～95.1
		1.000	0.01	82.3～96.1
	鸡肾	0.010	0.01	76.0～93.0
		0.100	0.01	82.3～96.7
		1.000	0.01	81.2～98.9
	蜂蜜	0.010	0.01	82.0～97.0
		0.100	0.01	82.6～97.2
		1.000	0.01	84.4～99.0
速灭威	糙米	0.010	0.01	76.0～93.0
		0.100	0.01	82.1～98.8
		1.000	0.01	81.1～95.8
	玉米	0.010	0.01	79.0～95.0
		0.100	0.01	81.9～98.8
		1.000	0.01	83.3～97.3
	白菜	0.010	0.01	86.0～106.0
		0.100	0.01	80.0～96.2
		1.000	0.01	78.6～92.1
	大葱	0.010	0.01	79.0～95.0
		0.100	0.01	80.1～95.4
		1.000	0.01	80.7～96.3

表 C.1（续）

药品名称	样品名称	添加水平 mg/kg	测定和确证低限 mg/kg	回收率范围 %
速灭威	大麦	0.010	0.01	81.0～97.0
		0.100	0.01	81.6～96.1
		1.000	0.01	76.1～97.1
	小麦	0.010	0.01	83.0～97.0
		0.100	0.01	81.9～97.8
		1.000	0.01	84.4～95.0
	苹果	0.010	0.01	77.0～92.0
		0.100	0.01	85.6～99.3
		1.000	0.01	85.3～98.9
	柑橘	0.010	0.01	81.0～99.0
		0.100	0.01	80.9～96.0
		1.000	0.01	82.9～97.5
	大豆	0.010	0.01	87.0～104.0
		0.100	0.01	81.2～96.9
		1.000	0.01	82.6～96.8
	花生	0.010	0.01	80.0～96.0
		0.100	0.01	81.2～96.5
		1.000	0.01	81.0～97.8
	牛肝	0.010	0.01	86.0～102.0
		0.100	0.01	81.7～95.4
		1.000	0.01	84.4～97.7
	鸡肾	0.010	0.01	80.0～94.0
		0.100	0.01	81.1～97.4
		1.000	0.01	81.2～95.9
	蜂蜜	0.010	0.01	78.0～95.0
		0.100	0.01	81.0～96.7
		1.000	0.01	80.0～93.7
噁虫威	糙米	0.010	0.01	86.0～103.0
		0.100	0.01	86.0～96.1
		1.000	0.01	82.6～95.2
	玉米	0.010	0.01	81.0～95.0
		0.100	0.01	82.5～96.4
		1.000	0.01	79.0～93.9
	白菜	0.010	0.01	86.0～101.0
		0.100	0.01	81.1～96.0
		1.000	0.01	83.6～97.8
	大葱	0.010	0.01	83.0～95.0
		0.100	0.01	80.8～96.4
		1.000	0.01	82.3～95.1
	大麦	0.010	0.01	82.0～96.0
		0.100	0.01	80.1～97.7
		1.000	0.01	85.2～103.6

表 C.1（续）

药品名称	样品名称	添加水平 mg/kg	测定和确证低限 mg/kg	回收率范围 %
噁虫威	小麦	0.010	0.01	85.0～99.0
		0.100	0.01	81.2～98.6
		1.000	0.01	84.4～96.1
	苹果	0.010	0.01	81.0～97.0
		0.100	0.01	81.2～99.7
		1.000	0.01	82.1～96.6
	柑橘	0.010	0.01	88.0～105.0
		0.100	0.01	78.6～97.8
		1.000	0.01	85.1～96.3
	大豆	0.010	0.01	82.0～97.0
		0.100	0.01	78.6～95.5
		1.000	0.01	85.3～97.5
	花生	0.010	0.01	78.0～95.0
		0.100	0.01	87.8～102.0
		1.000	0.01	81.2～97.4
	牛肝	0.010	0.01	76.0～95.0
		0.100	0.01	81.2～99.0
		1.000	0.01	84.5～98.8
	鸡肾	0.010	0.01	78.0～91.0
		0.100	0.01	86.4～98.6
		1.000	0.01	77.3～94.6
	蜂蜜	0.010	0.01	77.0～94.0
		0.100	0.01	83.9～99.8
		1.000	0.01	81.2～104.8
克百威	糙米	0.010	0.01	85.0～99.0
		0.100	0.01	78.6～94.4
		1.000	0.01	82.2～98.7
	玉米	0.010	0.01	85.0～105.0
		0.100	0.01	84.7～98.0
		1.000	0.01	80.3～96.8
	白菜	0.010	0.01	83.0～95.0
		0.100	0.01	80.5～109.0
		1.000	0.01	83.3～98.9
	大葱	0.010	0.01	86.0～100.0
		0.100	0.01	81.2～96.7
		1.000	0.01	78.7～95.5
	大麦	0.010	0.01	80.0～96.0
		0.100	0.01	82.6～94.8
		1.000	0.01	82.1～98.7
	小麦	0.010	0.01	79.0～96.0
		0.100	0.01	85.1～96.7
		1.000	0.01	81.3～97.9

表 C.1（续）

药品名称	样品名称	添加水平 mg/kg	测定和确证低限 mg/kg	回收率范围 %
克百威	苹果	0.010	0.01	79.0～89.0
		0.100	0.01	80.6～94.5
		1.000	0.01	81.6～98.7
	柑橘	0.010	0.01	83.0～106.0
		0.100	0.01	85.3～99.4
		1.000	0.01	79.7～91.3
	大豆	0.010	0.01	77.0～91.0
		0.100	0.01	77.8～95.6
		1.000	0.01	85.1～96.2
	花生	0.010	0.01	87.0～103.0
		0.100	0.01	84.5～99.7
		1.000	0.01	78.7～98.6
	牛肝	0.010	0.01	79.0～95.0
		0.100	0.01	89.0～112.0
		1.000	0.01	85.7～97.2
	鸡肾	0.010	0.01	85.0～99.0
		0.100	0.01	76.9～95.7
		1.000	0.01	82.3～98.8
	蜂蜜	0.010	0.01	81.0～95.0
		0.100	0.01	86.5～99.8
		1.000	0.01	81.4～95.6
甲萘威	糙米	0.010	0.01	81.0～102.0
		0.100	0.01	79.2～94.5
		1.000	0.01	83.7～100.0
	玉米	0.010	0.01	77.0～95.0
		0.100	0.01	82.4～98.0
		1.000	0.01	82.5～97.8
	白菜	0.010	0.01	80.0～94.0
		0.100	0.01	77.3～95.4
		1.000	0.01	78.6～93.4
	大葱	0.010	0.01	82.0～95.0
		0.100	0.01	83.4～98.3
		1.000	0.01	86.8～100.5
	大麦	0.010	0.01	82.0～106.0
		0.100	0.01	76.5～92.1
		1.000	0.01	79.6～95.3
	小麦	0.010	0.01	76.0～92.0
		0.100	0.01	82.5～106.5
		1.000	0.01	76.6～95.3
	苹果	0.010	0.01	82.0～99.0
		0.100	0.01	77.6～95.4
		1.000	0.01	82.0～99.9
	柑橘	0.010	0.01	87.0～104.0
		0.100	0.01	84.1～99.7
		1.000	0.01	80.4～95.1

表 C.1（续）

药品名称	样品名称	添加水平 mg/kg	测定和确证低限 mg/kg	回收率范围 %
甲萘威	大豆	0.010	0.01	80.0～95.0
		0.100	0.01	82.6～95.3
		1.000	0.01	78.6～93.3
	花生	0.010	0.01	83.0～99.0
		0.100	0.01	82.5～107.4
		1.000	0.01	82.7～99.8
	牛肝	0.010	0.01	83.0～101.0
		0.100	0.01	79.3～98.5
		1.000	0.01	77.6～93.6
	鸡肾	0.010	0.01	82.0～97.0
		0.100	0.01	81.4～97.8
		1.000	0.01	82.4～96.7
	蜂蜜	0.010	0.01	81.0～96.0
		0.100	0.01	80.1～94.6
		1.000	0.01	83.5～98.7
乙硫甲威	糙米	0.010	0.01	84.0～97.0
		0.100	0.01	77.5～95.8
		1.000	0.01	81.4～95.5
	玉米	0.010	0.01	81.0～109.0
		0.100	0.01	81.5～99.3
		1.000	0.01	77.4～95.1
	白菜	0.010	0.01	82.0～94.0
		0.100	0.01	80.2～99.8
		1.000	0.01	88.6～100.2
	大葱	0.010	0.01	89.0～102.0
		0.100	0.01	83.6～96.1
		1.000	0.01	81.6～95.3
	大麦	0.010	0.01	80.0～96.0
		0.100	0.01	81.0～97.3
		1.000	0.01	83.3～96.5
	小麦	0.010	0.01	83.0～97.0
		0.100	0.01	83.6～97.6
		1.000	0.01	85.1～99.1
	苹果	0.010	0.01	81.0～96.0
		0.100	0.01	81.4～95.2
		1.000	0.01	82.2～94.2
	柑橘	0.010	0.01	79.0～91.0
		0.100	0.01	82.9～98.4
		1.000	0.01	81.6～93.5
	大豆	0.010	0.01	83.0～95.0
		0.100	0.01	83.8～97.1
		1.000	0.01	80.6～96.3

表 C.1（续）

药品名称	样品名称	添加水平 mg/kg	测定和确证低限 mg/kg	回收率范围 %
乙硫甲威	花生	0.010	0.01	82.0～96.0
		0.100	0.01	83.4～97.7
		1.000	0.01	81.4～98.6
	牛肝	0.010	0.01	81.0～95.0
		0.100	0.01	80.6～96.6
		1.000	0.01	81.2～95.3
	鸡肾	0.010	0.01	81.0～96.0
		0.100	0.01	81.1～94.5
		1.000	0.01	82.1～97.0
	蜂蜜	0.010	0.01	80.0～98.0
		0.100	0.01	81.2～96.0
		1.000	0.01	83.6～94.6
异丙威	糙米	0.010	0.01	81.0～95.0
		0.100	0.01	81.2～99.7
		1.000	0.01	80.1～98.1
	玉米	0.010	0.01	81.0～95.0
		0.100	0.01	80.1～96.3
		1.000	0.01	80.4～95.1
	白菜	0.010	0.01	82.0～95.0
		0.100	0.01	82.5～97.6
		1.000	0.01	80.0～96.1
	大葱	0.010	0.01	81.0～95.0
		0.100	0.01	84.9～104.0
		1.000	0.01	81.4～96.8
	大麦	0.010	0.01	81.0～95.0
		0.100	0.01	81.0～97.4
		1.000	0.01	81.3～97.6
	小麦	0.010	0.01	81.0～103.0
		0.100	0.01	81.4～97.5
		1.000	0.01	81.3～96.7
	苹果	0.010	0.01	80.0～95.0
		0.100	0.01	81.2～98.2
		1.000	0.01	80.7～96.6
	柑橘	0.010	0.01	79.0～96.0
		0.100	0.01	82.6～95.5
		1.000	0.01	80.7～96.1
	大豆	0.010	0.01	81.0～95.0
		0.100	0.01	80.3～91.2
		1.000	0.01	81.0～95.2
	花生	0.010	0.01	86.0～100.0
		0.100	0.01	83.7～97.6
		1.000	0.01	83.6～96.0

表 C.1（续）

药品名称	样品名称	添加水平 mg/kg	测定和确证低限 mg/kg	回收率范围 %
异丙威	牛肝	0.010	0.01	83.0～96.0
		0.100	0.01	80.2～96.5
		1.000	0.01	80.3～97.6
	鸡肾	0.010	0.01	81.0～96.0
		0.100	0.01	80.3～96.9
		1.000	0.01	80.1～95.5
	蜂蜜	0.010	0.01	81.0～103.0
		0.100	0.01	85.9～105.0
		1.000	0.01	80.2～95.6
乙霉威	糙米	0.010	0.01	80.0～101.0
		0.100	0.01	80.1～97.3
		1.000	0.01	80.9～96.7
	玉米	0.010	0.01	81.0～97.0
		0.100	0.01	81.1～97.5
		1.000	0.01	81.7～97.7
	白菜	0.010	0.01	86.0～101.0
		0.100	0.01	80.0～98.2
		1.000	0.01	80.8～95.7
	大葱	0.010	0.01	79.0～94.0
		0.100	0.01	80.5～102.1
		1.000	0.01	80.7～97.7
	大麦	0.010	0.01	84.0～103.0
		0.100	0.01	80.9～97.2
		1.000	0.01	77.9～95.0
	小麦	0.010	0.01	82.0～98.0
		0.100	0.01	80.2～98.4
		1.000	0.01	81.5～98.8
	苹果	0.010	0.01	85.0～104.0
		0.100	0.01	81.5～100.9
		1.000	0.01	79.1～96.9
	柑橘	0.010	0.01	80.0～100.0
		0.100	0.01	81.2～98.7
		1.000	0.01	83.6～95.9
	大豆	0.010	0.01	85.0～106.0
		0.100	0.01	81.7～93.5
		1.000	0.01	84.2～99.1
	花生	0.010	0.01	76.0～93.0
		0.100	0.01	80.6～98.0
		1.000	0.01	80.3～97.3
	牛肝	0.010	0.01	84.0～100.0
		0.100	0.01	82.5～103.6
		1.000	0.01	81.2～97.1
	鸡肾	0.010	0.01	84.0～102.0
		0.100	0.01	82.3～98.5
		1.000	0.01	80.4～96.6

表 C.1（续）

药品名称	样品名称	添加水平 mg/kg	测定和确证低限 mg/kg	回收率范围 %
乙霉威	蜂蜜	0.010	0.01	82.0～96.0
		0.100	0.01	83.7～99.2
		1.000	0.01	78.7～95.9
仲丁威	糙米	0.010	0.01	84.0～99.0
		0.100	0.01	80.9～98.7
		1.000	0.01	81.5～99.0
	玉米	0.010	0.01	85.0～96.0
		0.100	0.01	80.0～95.8
		1.000	0.01	82.3～95.6
	白菜	0.010	0.01	82.0～98.0
		0.100	0.01	82.7～97.5
		1.000	0.01	80.1～96.8
	大葱	0.010	0.01	82.0～96.0
		0.100	0.01	81.7～98.4
		1.000	0.01	82.6～96.7
	大麦	0.010	0.01	86.0～101.0
		0.100	0.01	86.9～97.8
		1.000	0.01	80.6～98.1
	小麦	0.010	0.01	84.0～96.0
		0.100	0.01	84.1～93.7
		1.000	0.01	80.5～96.3
	苹果	0.010	0.01	82.0～97.0
		0.100	0.01	82.9～98.4
		1.000	0.01	81.7～95.1
	柑橘	0.010	0.01	82.0～95.0
		0.100	0.01	82.4～97.0
		1.000	0.01	80.1～98.0
	大豆	0.010	0.01	81.0～97.0
		0.100	0.01	82.5～97.1
		1.000	0.01	80.2～98.3
	花生	0.010	0.01	81.0～99.0
		0.100	0.01	83.2～97.3
		1.000	0.01	85.1～103.1
	牛肝	0.010	0.01	82.0～104.0
		0.100	0.01	81.2～94.7
		1.000	0.01	82.0～97.5
	鸡肾	0.010	0.01	81.0～97.0
		0.100	0.01	81.0～96.3
		1.000	0.01	81.8～94.7
	蜂蜜	0.010	0.01	82.0～96.0
		0.100	0.01	81.7～96.4
		1.000	0.01	80.3～93.7

Foreword

This standard is drafted in accordance with GB/T 1.1—2009.

This standard reviews and combines four former SN standards including the Method for Determination of Carbaryl and Carbofuran Residues in Grain for Export (SN 0134—1992), the Method for the Determination of Isoprocarb Residues in Cereals for Export (SN 0490—1995), the Method for the Determination of Fenobucarb Residues in Cereals for Export (SN 0534—1996), and the Method for the Determination of Methomyl Residues in Cereals and Oil Seeds for Export (SN 0582—1996). Compared with the above mentioned four standards, this standard which broadens the matrix to 13 kinds and the tested compounds to 12 items uses liquid chromatography-tandem mass spectrometry (LC-MS/MS) with increased sensitivity, additional qualification and optimized pre-treatment method.

Attention is required to the certain contents of this text which might be related to some patents. This file is not responsible to identify these.

This part is proposed by and is under the charge of Certification and Accreditation Administration of tle People's Republic of china.

The drafters of this standard are the Jilin Entry-Exit Inspection & Quarantine Bureau, the Hunan Entry-Exit Inspection & Quarantine Bureau, and the Chinese Academy of Inspection and Quarantine.

Main drafters of this standard are Wang Mingtai, Mu Jun, Huang Zhiqiang, Qiu Yueming, Zhang Daihui, Zhou Xiao, Han Dachuan.

Determination for pesticide residues of 12 kinds of carbamates including oxamyl in foods for import and export—LC-MS/MS method

1 Scope

This standard specifies the determination method for residues of Oxamyl, Methomyl, Pirimicarb, Idicarb, Metolcarb, Bendiocarb, Carbofuran, Carbaryl, Ethiofencarb, Isoprocarb, Diethofencarb and Fenobucarb in foods for import and export by liquid chromatography coupled to tandem mass spectrometry (LC-MS/MS).

This standard is applicable to determination and confirmation for residues of Oxamyl, Methomyl, Pirimicarb, Idicarb, Metolcarb, Bendiocarb, Carbofuran, Carbaryl, Ethiofencarb, Isoprocarb, Diethofencarb and Fenobucarb in corn, brown rice, barley, Chinese cabbage, scallion, wheat, soybean, peanut, apple, orange, bovine liver, chicken kidney and honey.

2 Normative references

The items of the following listed standard become the items of this standard due to the quotation by this standard. The cited references with date would not apply to this standard if their amendment (not including corrected printing errors) or revision appear. However, it is encouraged to study if the newest edition of these references can be used. The newest edition is applicable to this standard if the references are not quoted with date.

GB/T 6682 Water for laboratory use—Specifications

3 Principle

Test sample is extracted with acetonitrile, employed liquid-liquid partition with *n*-hexane solution which is saturated with acetonitrile, cleaned up by solid phase extraction (SPE) column, and determined by LC-MS/MS with external standard method.

4 Reagents and materials

All the reagents used should be analytically pure unless otherwise specified. "Water" is the first level

water described by GB/T 6682.

4.1 Acetonitrile: Grade for residue analysis.

4.2 Acetone: Grade for residue analysis.

4.3 Methanol: Grade for residue analysis.

4.4 *n*-Hexane: Grade for residual analysis.

4.5 Dichloromethane: Grade for residual analysis.

4.6 Anhydrous sodium sulfate: Ignited at 650 ℃ for 4 h, and then stored in a tightly closed container.

4.7 Acetone-*n*-hexane (3+7, *V* / *V*): Volume 30 mL Acetone and 70 mL *n*-hexane, and mix to homogenous.

4.8 Acetonitrile-*n*-hexane.

4.9 12 kinds of carbamate standards including oxamyl etc. : Purity ≥98.5%.

4.10 Standard stock solution: Accurately weigh appropriate amount of carbamate standards and dissolve with methanol. Dilute with acetone to make a final concentration of 100 μg /mL stock solution. The solution is stored in a refrigerator at −18 ℃ and can be used for one year.

4.11 Standard working solution: Acquire suitable volume of the 12 kinds of carbamate standards stock solutions, and dilute the standard stock solution with methanol to make required concentration. The solution is stored in a refrigerator at −18 ℃ and can be used in three months.

4.12 Mix stand working solution: The solution is stored a refrigerator at 0 ℃～40 ℃.

4.13 Florisil SPE tube: Florisil, 1 000 mg, 6 mL.

4.14 Active carbon SPE tube: ENVI-Carb, 500 mg, 6 mL, or equivalent.

4.15 Membrane filter: 0.2 μm.

5 Apparatus and equipment

5.1 LC-MS /MS: Equipped with electrospray ionization (ESI).

5.2 Balance: Accurate to 0.1 mg and 0.01 g.

5.3 Organ blender.

5.4 Pulverizer.

5.5 Homogenizer.

5.6 Vortex mixer.

5.7 Centrifuge: 4 000 r/min.

5.8 Rotary vacuum evaporator.

5.9 Centrifuge tube: 50 mL.

5.10 Erlenmeyer Flask: 250 mL.

5.11 Concentrate bottle: 250 mL.

6 Preparation and storage of test sample

6.1 Preparation of test sample

6.1.1 Corn, brown-rice, soybear and peanut

Take approximately 500 g of representative sample. Smash thoroughly in a blender. Mix thoroughly. Put into clean containers. Seal and label them.

6.1.2 Orange, apple, scallion, Chinese cabbage, onion and garlic

Take approximately 500 g of representative sample. Collect the edible parts (do not wash with water) and cut into minces. Cut by a chopper into pulp. Mix thoroughly. Put into clean containers. Seal and label them.

6.1.3 Bovine liver and chicken kidney

Take approximately 500 g of representative sample. Collect the edible parts. Crush with a crusher. Mix thoroughly. Put into clean containers. Seal and label them.

6.1.4 Honey

Take approximately 500 g of representative sample. The non-crystallized sample is stirred to homoge-

neous status. The crystallized sample is warmed in one tightly sealed bottle in water-bath at no more than 60 ℃, mix thoroughly when all samples have melted then cool immediately to room temperature with caution of water loss. Take the prepared sample into two sample bottles, seal and label them.

6.2 Storage of test sample

Test samples of cereals & grains, nuts, tea, honey and chilli sauce shall be stored at temperature ranged from 0 ℃～4 ℃. Other samples shall be frozen and stored at a temperature below －18 ℃. In course of sampling and sample preparation, caution shall be taken to avoid contamination or any factors which may cause the change of residue content.

7 Procedure

7.1 Extraction

7.1.1 Corn, brown-rice, Chinese cabbage, scallion, barley, wheat, soybean, peanut, apple, orange bovine liver and chicken kidney

Weigh 5 g (accurate to 0.01) of the test sample into a 50 mL centrifuge tube. Add 20 mL of acetonitrile into the tube. Homogenize for 3 min. Centrifuge for 3 min at 4 000 r/min. Pour supernatant into a second 50 mL centrifuge tube. Extract the residue once more with 10 mL of acetonitrile. Combine the supernatants into the second 50 mL centrifuge tube. Add 10 mL of *n*-hexane. Vortex thoroughly. Discard upper layer of *n*-hexane away (for soybean and peanut, add 10 mL of *n*-hexane once more, vortex thoroughly, discard *n*-hexane away). Transfter the acetonitrile solution into one 250 mL concentrate bottle. Condense to nearly dryness by a rotary evaporator in water bath at 40 ℃. Add 2 mL acetone-*n*-hexane (3＋7) to dissolve the residue for further clean-up procedure.

7.1.2 Honey

Weigh 15 g (accurate to 0.01 g) of the test sample into a 250 mL Erlenmeyer Flask. Add 30 mL of water, shake for 15 min in 40 ℃ water bath. Add 10 mL of acetone, transfer into 250 mL seperatory funnel. Wash several times with CH_2Cl_2. Move the washings into another seperatory funnel (carefully exhaust air). Shake vigiously eight times, stand still and wait for layer seperation. The lower layer passes through Anhydrous sodium sulfate. Collect eluates into one 250 mL concentrate flask. Extract one minute firstly with 5 mL acetone and then with 40 mL CH_2Cl_2. Repeat the extraction. Collect and combine the two extracts. Condense to nearly dryness by a rotary evaporator in water bath at 40 ℃. Add 2 mL acetone-*n*-hexane (3＋7) to dissolve the residue for further clean-up procedure.

7.2 Cleaning-up

Couple one active carbon SPE tube (4.10) and one Florisil SPE tube (4.11) up to down. Condition the two tubes with 10 mL of acetone-*n*-hexane (3＋7) in advance. Discard the washings. Transfer the

extract solution (7.1.1 or 7.1.2) into the upper tube. Then wash the concetrate bottle with 2 mL of acetone-*n*-hexane (3 + 7) and elute the tubes with the washings. Collect all eluates into another 250 mL concentrate bottle. Condense to nearly dryness by a rotary evaporator in water bath at 40 ℃. Dissolve the residue and dilute exactly to 2.0 mL with acetone for the LC-MS /MS determination and confirmation.

7.3 Determination

7.3.1 LC operating condition

a) Chromatographic column: Zorbax C_{18}, 150 cm × 2.1 mm (i.d.), size 5 μm, or equivalent;

b) Column temperature: 40 ℃;

c) Flow rate: 0.2 mL /min;

d) Injection volume: 5 μL;

e) Mobile phase and gradient elution condition is found in table 1.

Table 1—Mobile phase and gradient elution condition

Time min	Flow rate μL/min	Methanol %	Water(1% formic acid) %	Water %
0.00	200	20.0	20.0	60.0
2.00	200	20.0	20.0	60.0
3.00	200	60.0	40.0	0
20.00	200	75.0	25.0	0
20.10	200	20.0	40.0	40.0
25.00	200	20.0	40.0	40.0

7.3.2 MS/MS condition

a) Ionization source: ESI;

b) Scan mode: positive ion mode;

c) Monitor mode: multiple reaction monitoring (MRM);

d) Electrospray voltage (IS): 3.0 kV;

e) Ion source gas, Curtain gas, auxiliary gas, and collision gas are all ultra pure nitrogen. Before usage all gas flow must be optimized to guarantee the required sensitivity of the MS detector;

f) Ion source gas temperature;

g) Qualification ion pairs, quantitation ion pairs, dwell time, and declustering potential (DP) etc. parameters are showed in Annex A.

7.3.3 LC-MS/MS determination and confirmation

According to the above mentioned LC-MS /MS operating condition, determine the sample solution and the working standard solution. Select the standard working solution with a similar concentration of the sample solution. The standard working solution should be injected in-between the injections of the sample solutions with one common volume. The responses of 12 kinds of carbamates in the standard working solution and in the sample solution should be within the linear range of the instrumental detection.

Determin the standard solution and the sample solution according to the conditions described in 7.3.1 and 7.3.2. If a respected peak of sample solution appears at the same retention time as that of the peak of the standard, comfirmation will be performed. After comfirmation, if the retention time of the tested sample is in accordance with the time of standards, all selected ions appear in the background subtracted chromatogram, furthermore the relative ionic abundance ration of sample and standard are accorded with each other, and the similarity of sample and standard is in the range of tolerance (see table 2), the positive result of the test sample can be derived.

The LC-MS /MS TIC chromatograph and SIM chromatograph of 12 kinds of carbamates including oxamyl etc. are shown as figure B.1 in Annex B.

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

Relative ionic abundance/%	>50	>20～50	>10～20	≤10
Allowed relative deviation/%	±20	±25	±30	±50

7.4 Blank test

Blank test will be conducted according to the procedures above without sample addition.

7.5 Calculation and expression of the result

Calculate the content of 12 kinds of carbamates including oxamyl etc. residue in the test sample by data processor or according to the followed formula (1).

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

Where

X_i —the ith pestcide residue content of 12 kinds of carbamates including oxamyl etc. in the test sample. Unit is milligram per kilogram, mg /kg;

A_i —the peak area (or height) of the ith pestcide residue of 12 kinds of carbamates including oxamyl etc. in the sample solution;

A_{is} —the peak area (or height) of the ith pestcide residue of 12 kinds of carbamates including oxamyl etc. in the standard solution;

c_i —the concentration of the ith pestcide residue of 12 kinds of carbamates including oxamyl etc. in the standard working solution. Unit is microgram per milliliter, μg /mL;

V —the final volume of the sample solution. Unit is milliliter, mL;

m —the corresponding mass of the test sample representing the final sample solution. Unit is gram, g.

Note: The result of calculation should be deducted with blank value.

8 Detection limit and recovery

8.1 Limit of determination

The limit of determination of the method is shown in table C.1.

8.2 Range of fortification and recovery

The ranges of fortification and recovery of this method are shown in table C.1.

Annex A
(Informative)
LC-MS/MS parameters[1)]

LC-MS /MS parameters

a) Ionization source:ESI;

b) Electrospray voltage (IS):5 500 V;

c) Ion source gas 1 (GS1):20 psi;

d) Curtain gas pressure (CUR):20 psi;

e) Ion source gas 2 (GS2):30 psi;

f) Temperature(TEM):550 ℃;

g) Collisionally activated dissociation gas (CAD):6 mL/min;

h) Collision cell exit potential CXP:10 V;

i) Entrance potential EP:10 V;

j) Scan mode:Positive ion scan;

k) Monitor mode:Multiple reaction monitoring (MRM);

l) MS/MS parameters see table A. 1.

Table A. 1 MS/MS parameters of 12 kinds of carbamates including oxamyl etc

Compound	Q1 (m/z)	Q3 (m/z)	Dwell time ms	declustering potential DP V	collision energy (CE) eV	Retention time min
oxamyl	242. 1	121. 3	30	73	18	7. 43
		72. 3*	30	73	35	

1) Non commercial statement:Parameters listed in Annex A1 were acquired from API4000 MS/MS. Here the listed companies are only for references without any commercial aims. User of this standards are encouraged to try various instruments from any companies.

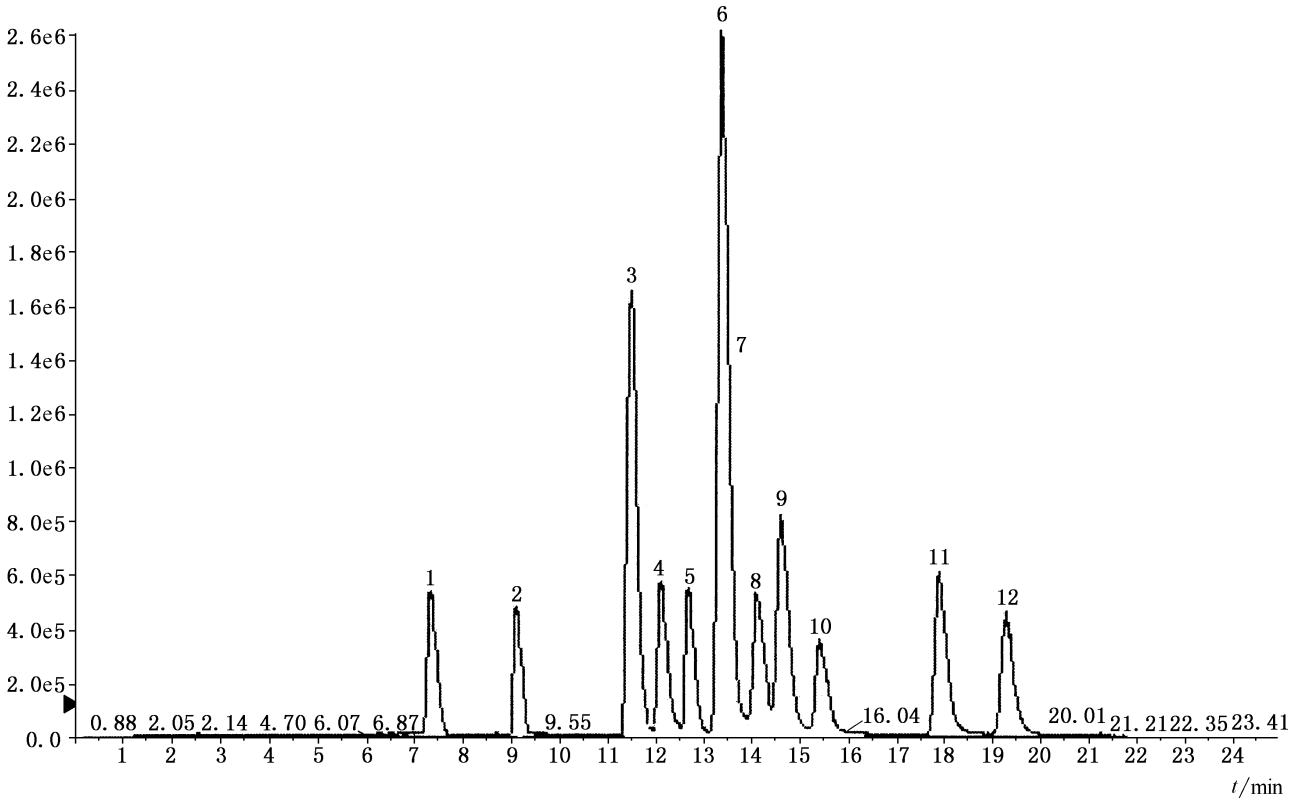
Table A.1（continued）

Compound	Q1 (m/z)	Q3 (m/z)	Dwell time ms	declustering potential DP V	collision energy (CE) eV	Retention time min
methomyl	163.0	106.1	30	58	12	9.28
		88.1*	30	58	12	
pirimicarb	238.7	182.2	30	66	22	11.47
		72.2*	30	66	36	
aldicarb	208.1	116.2	30	55	10	12.28
		89.2*	30	55	21	
metolcarb	166.2	109.1*	30	62	14	12.88
		94.1*	30	62	42	
bendiocarb	224.4	167.1	30	65	13	13.37
		109.0*	30	65	25	
carbofuran	222.3	165.0	30	70	16	13.40
		123.2*	30	70	30	
carbaryl	202.2	145.2	30	55	12	14.15
		127.2*	30	55	38	
ethiofencarb	226.2	164.0*	30	53	11	14.61
		107.0*	30	53	19	
Isoprocarb	194.2	137.2	30	70	12	15.41
		95.3*	30	70	20	
diethofencarb	268.3	180.0*	30	55	23	17.87
		152.0	30	55	30	
fenobucarb	208.4	151.2*	30	70	12	19.28
		109.1*	30	70	21	

Note 1：“*”are quantitation ions.

Note 2：To different MS instruments，their parameters which might vary shall be optimized before determination.

Annex B
（Informative）
TIC chromatograph of LC-MS/MS chromatograph of
12 carbamate standards including oxamyl etc.



1—Oxamyl;
2—Methomyl;
3—Pirimicarb;
4—Aldicarb;
5—Metolcarb;
6—Bendiocarb;
7 —Carbofuran;
8 —Carbaryl;
9 —Ethiofencarb;
10—Isoprocarb;
11—Diethofencarb;
12—Fenobucarb.

Figure B. 1—LC-MS/MS TIC chromatogram for 12 kinds of carbamate standards including oxamyl etc.

Annex C
（Informative）
The limit of determination and confirmation and the range of recovery of this method

Table C. 1—Limit of determination and confirmation and the range of recovery of this method

Compound	Sample	Fortified level mg/kg	Limit of determination and confirmation mg/kg	Range of recovery %
oxamyl	brown rice	0. 010	0. 01	81. 0～93. 0
		0. 100	0. 01	82. 5～94. 9
		1. 000	0. 01	89. 4～108. 3
	corn	0. 010	0. 01	84. 0～98. 0
		0. 100	0. 01	79. 2～95. 4
		1. 000	0. 01	81. 6～99. 7
	chinese cabbage	0. 010	0. 01	83. 0～99. 0
		0. 100	0. 01	84. 3～97. 0
		1. 000	0. 01	81. 2～102. 4
	scallion	0. 010	0. 01	80. 0～97. 0
		0. 100	0. 01	84. 7～99. 1
		1. 000	0. 01	81. 9～92. 4
	barley	0. 010	0. 01	81. 0～103. 0
		0. 100	0. 01	76. 8～95. 7
		1. 000	0. 01	83. 2～103. 6
	wheat	0. 010	0. 01	86. 0～102. 0
		0. 100	0. 01	82. 5～98. 0
		1. 000	0. 01	88. 0～108. 4
	apple	0. 010	0. 01	82. 0～96. 0
		0. 100	0. 01	86. 3～98. 7
		1. 000	0. 01	83. 6～102. 1
	orange	0. 010	0. 01	85. 0～101. 0
		0. 100	0. 01	87. 2～110. 0
		1. 000	0. 01	84. 3～99. 2
	soybean	0. 010	0. 01	80. 0～93. 0
		0. 100	0. 01	82. 6～96. 7
		1. 000	0. 01	83. 1～102. 6
	peanut	0. 010	0. 01	79. 0～95. 0
		0. 100	0. 01	86. 4～102. 3
		1. 000	0. 01	79. 2～96. 1
	bovine liver	0. 010	0. 01	86. 0～105. 0
		0. 100	0. 01	89. 4～106. 0
		1. 000	0. 01	84. 3～99. 6
	chicken kidney	0. 010	0. 01	83. 0～103. 0
		0. 100	0. 01	81. 0～96. 1
		1. 000	0. 01	82. 1～99. 2
	honey	0. 010	0. 01	78. 0～92. 0
		0. 100	0. 01	82. 6～99. 7
		1. 000	0. 01	83. 6～97. 8

Table C.1（continued）

Compound	Sample	Fortified level mg/kg	Limit of determination and confirmation mg/kg	Range of recovery %
methomyl	brown rice	0.010	0.01	86.0～100.0
		0.100	0.01	81.1～99.3
		1.000	0.01	86.8～102.7
	corn	0.010	0.01	80.0～94.0
		0.100	0.01	85.7～97.6
		1.000	0.01	85.5～101.2
	chinese cabbage	0.010	0.01	80.0～97.0
		0.100	0.01	88.2～106.0
		1.000	0.01	88.5～100.4
	scallion	0.010	0.01	82.0～97.0
		0.100	0.01	79.6～98.2
		1.000	0.01	78.5～96.4
	barley	0.010	0.01	81.0～97.0
		0.100	0.01	82.9～97.3
		1.000	0.01	82.4～103.7
	wheat	0.010	0.01	83.0～97.0
		0.100	0.01	85.2～106.0
		1.000	0.01	81.1～95.7
	apple	0.010	0.01	80.0～94.0
		0.100	0.01	87.3～103.4
		1.000	0.01	81.3～99.6
	orange	0.010	0.01	78.0～95.0
		0.100	0.01	81.4～96.7
		1.000	0.01	80.2～99.3
	soybean	0.010	0.01	86.0～102.0
		0.100	0.01	82.3～95.7
		1.000	0.01	80.1～97.3
	peanut	0.010	0.01	78.0～95.0
		0.100	0.01	77.8～95.3
		1.000	0.01	80.3～97.7
	bovine liver	0.010	0.01	89.0～104.0
		0.100	0.01	79.5～96.0
		1.000	0.01	81.6～99.1
	chicken kidney	0.010	0.01	79.0～95.0
		0.100	0.01	86.7～105.0
		1.000	0.01	88.6～100.3
pirimicarb	brown rice	0.010	0.01	78.0～97.0
		0.100	0.01	81.9～98.0
		1.000	0.01	83.4～99.1
	corn	0.010	0.01	78.0～96.0
		0.100	0.01	89.2～103.0
		1.000	0.01	83.6～97.1

Table C.1（continued）

Compound	Sample	Fortified level mg/kg	Limit of determination and confirmation mg/kg	Range of recovery %
pirimicarb	chinese cabbage	0.010	0.01	76.0～93.0
		0.100	0.01	82.2～95.1
		1.000	0.01	81.5～96.3
	scallion	0.010	0.01	79.0～93.0
		0.100	0.01	83.4～97.6
		1.000	0.01	82.8～96.4
	barley	0.010	0.01	76.0～92.0
		0.100	0.01	78.6～95.3
		1.000	0.01	84.8～103.7
	wheat	0.010	0.01	85.0～98.0
		0.100	0.01	78.6～95.8
		1.000	0.01	80.0～93.6
	apple	0.010	0.01	80.0～95.0
		0.100	0.01	81.2～98.8
		1.000	0.01	88.4～100.9
	orange	0.010	0.01	81.0～94.0
		0.100	0.01	80.9～97.7
		1.000	0.01	82.6～96.1
	soybean	0.010	0.01	82.0～96.0
		0.100	0.01	84.0～107.0
		1.000	0.01	80.2～97.3
	peanut	0.010	0.01	82.0～96.0
		0.100	0.01	84.3～97.3
		1.000	0.01	83.2～96.3
	bovine liver	0.010	0.01	76.0～95.0
		0.100	0.01	80.5～96.4
		1.000	0.01	85.3～102.6
	chicken kidney	0.010	0.01	82.0～95.0
		0.100	0.01	77.6～98.2
		1.000	0.01	81.2～96.2
	honey	0.010	0.01	76.0～93.0
		0.100	0.01	83.0～95.6
		1.000	0.01	85.3～101.5
aldicarb	brown rice	0.010	0.01	81.0～95.0
		0.100	0.01	79.3～97.7
		1.000	0.01	80.1～97.7
	corn	0.010	0.01	79.0～95.0
		0.100	0.01	78.8～94.0
		1.000	0.01	83.1～99.0

Table C.1（continued）

Compound	Sample	Fortified level mg/kg	Limit of determination and confirmation mg/kg	Range of recovery %
aldicarb	chinese cabbage	0.010	0.01	82.0～97.0
		0.100	0.01	78.6～91.5
		1.000	0.01	82.0～97.7
	scallion	0.010	0.01	81.0～97.0
		0.100	0.01	83.3～100.8
		1.000	0.01	79.8～93.5
	barley	0.010	0.01	81.0～95.0
		0.100	0.01	81.1～95.6
		1.000	0.01	85.3～95.7
	wheat	0.010	0.01	77.0～95.0
		0.100	0.01	84.5～98.3
		1.000	0.01	83.6～95.8
	apple	0.010	0.01	76.0～95.0
		0.100	0.01	81.9～98.8
		1.000	0.01	77.5～90.7
	orange	0.010	0.01	76.0～93.0
		0.100	0.01	81.9～98.0
		1.000	0.01	77.6～93.1
	soybean	0.010	0.01	80.0～97.0
		0.100	0.01	78.4～93.7
		1.000	0.01	85.8～100.4
	peanut	0.010	0.01	82.0～109.0
		0.100	0.01	82.1～94.6
		1.000	0.01	78.7～93.1
	bovine liver	0.010	0.01	80.0～97.0
		0.100	0.01	80.2～95.1
		1.000	0.01	82.3～96.1
	chicken kidney	0.010	0.01	76.0～93.0
		0.100	0.01	82.3～96.7
		1.000	0.01	81.2～98.9
	honey	0.010	0.01	82.0～97.0
		0.100	0.01	82.6～97.2
		1.000	0.01	84.4～99.0
metolcarb	brown rice	0.010	0.01	76.0～93.0
		0.100	0.01	82.1～98.8
		1.000	0.01	81.1～95.8
	corn	0.010	0.01	79.0～95.0
		0.100	0.01	81.9～98.8
		1.000	0.01	83.3～97.3
	chinese cabbage	0.010	0.01	86.0～106.0
		0.100	0.01	80.0～96.2
		1.000	0.01	78.6～92.1
	scallion	0.010	0.01	79.0～95.0
		0.100	0.01	80.1～95.4
		1.000	0.01	80.7～96.3

Table C.1（continued）

Compound	Sample	Fortified level mg/kg	Limit of determination and confirmation mg/kg	Range of recovery %
metolcayb	barley	0.010	0.01	81.0～97.0
		0.100	0.01	81.6～96.1
		1.000	0.01	76.1～97.1
	wheat	0.010	0.01	83.0～97.0
		0.100	0.01	81.9～97.8
		1.000	0.01	84.4～95.0
	apple	0.010	0.01	77.0～92.0
		0.100	0.01	85.6～99.3
		1.000	0.01	85.3～98.9
	orange	0.010	0.01	81.0～99.0
		0.100	0.01	80.9～96.0
		1.000	0.01	82.9～97.5
	soybean	0.010	0.01	87.0～104.0
		0.100	0.0l	81.2～96.9
		1.000	0.01	82.6～96.8
	peanut	0.010	0.01	80.0～96.0
		0.100	0.01	81.2～96.5
		1.000	0.01	81.0～97.8
	bovine liver	0.010	0.01	86.0～102.0
		0.100	0.01	81.7～95.4
		1.000	0.01	84.4～97.7
	chicken kidney	0.010	0.01	80.0～94.0
		0.100	0.01	81.1～97.4
		1.000	0.01	81.2～95.9
	honey	0.010	0.01	78.0～95.0
		0.100	0.01	81.0～96.7
		1.000	0.01	80.0～93.7
bendiocarb	brown rice	0.010	0.01	86.0～103.0
		0.100	0.01	86.0～96.1
		1.000	0.01	82.6～95.2
	corn	0.010	0.01	81.0～95.0
		0.100	0.01	82.5～96.4
		1.000	0.01	79.0～93.9
	chinese cabbage	0.010	0.01	86.0～101.0
		0.100	0.01	81.1～96.0
		1.000	0.01	83.6～97.8
	scallion	0.010	0.01	83.0～95.0
		0.100	0.01	80.8～96.4
		1.000	0.01	82.3～95.1
	barley	0.010	0.01	82.0～96.0
		0.100	0.01	80.1～97.7
		1.000	0.01	85.2～103.6

Table C.1（continued）

Compound	Sample	Fortified level mg/kg	Limit of determination and confirmation mg/kg	Range of recovery %
bendiocarb	wheat	0.010	0.01	85.0～99.0
		0.100	0.01	81.2～98.6
		1.000	0.01	84.4～96.1
	apple	0.010	0.01	81.0～97.0
		0.100	0.01	81.2～99.7
		1.000	0.01	82.1～96.6
	orange	0.010	0.01	88.0～105.0
		0.100	0.01	78.6～97.8
		1.000	0.01	85.1～96.3
	soybean	0.010	0.01	82.0～97.0
		0.100	0.01	78.6～95.5
		1.000	0.01	85.3～97.5
	peanut	0.010	0.01	78.0～95.0
		0.100	0.01	87.8～102.0
		1.000	0.01	81.2～97.4
	bovine liver	0.010	0.01	76.0～95.0
		0.100	0.01	81.2～99.0
		1.000	0.01	84.5～98.8
	chicken kidney	0.010	0.01	78.0～91.0
		0.100	0.01	86.4～98.6
		1.000	0.01	77.3～94.6
	honey	0.010	0.01	77.0～94.0
		0.100	0.01	83.9～99.8
		1.000	0.01	81.2～104.8
carbofuran	brown rice	0.010	0.01	85.0～99.0
		0.100	0.01	78.6～94.4
		1.000	0.01	82.2～98.7
	corn	0.010	0.01	85.0～105.0
		0.100	0.01	84.7～98.0
		1.000	0.01	80.3～96.8
	chinese cabbage	0.010	0.01	83.0～95.0
		0.100	0.01	80.5～109.0
		1.000	0.01	83.3～98.9
	scallion	0.010	0.01	86.0～100.0
		0.100	0.01	81.2～96.7
		1.000	0.01	78.7～95.5
	barley	0.010	0.01	80.0～96.0
		0.100	0.01	82.6～94.8
		1.000	0.01	82.1～98.7
	wheat	0.010	0.01	79.0～96.0
		0.100	0.01	85.1～96.7
		1.000	0.01	81.3～97.9

Table C.1（continued）

Compound	Sample	Fortified level mg/kg	Limit of determination and confirmation mg/kg	Range of recovery %
carbofuran	apple	0.010	0.01	79.0～89.0
		0.100	0.01	80.6～94.5
		1.000	0.01	81.6～98.7
	orange	0.010	0.01	83.0～106.0
		0.100	0.01	85.3～99.4
		1.000	0.01	79.7～91.3
	soybean	0.010	0.01	77.0～91.0
		0.100	0.01	77.8～95.6
		1.000	0.01	85.1～96.2
	peanut	0.010	0.01	87.0～103.0
		0.100	0.01	84.5～99.7
		1.000	0.01	78.7～98.6
	bovine liver	0.010	0.01	79.0～95.0
		0.100	0.01	89.0～112.0
		1.000	0.01	85.7～97.2
	chicken kidney	0.010	0.01	85.0～99.0
		0.100	0.01	76.9～95.7
		1.000	0.01	82.3～98.8
	honey	0.010	0.01	81.0～95.0
		0.100	0.01	86.5～99.8
		1.000	0.01	81.4～95.6
carbaryl	brown rice	0.010	0.01	81.0～102.0
		0.100	0.01	79.2～94.5
		1.000	0.01	83.7～100.0
	corn	0.010	0.01	77.0～95.0
		0.100	0.01	82.4～98.0
		1.000	0.01	82.5～97.8
	chinese cabbage	0.010	0.01	80.0～94.0
		0.100	0.01	77.3～95.4
		1.000	0.01	78.6～93.4
	scallion	0.010	0.01	82.0～95.0
		0.100	0.01	83.4～98.3
		1.000	0.01	86.8～100.5
	barley	0.010	0.01	82.0～106.0
		0.100	0.01	76.5～92.1
		1.000	0.01	79.6～95.3
	wheat	0.010	0.01	76.0～92.0
		0.100	0.01	82.5～106.5
		1.000	0.01	76.6～95.3
	apple	0.010	0.01	82.0～99.0
		0.100	0.01	77.6～95.4
		1.000	0.01	82.0～99.9
	orange	0.010	0.01	87.0～104.0
		0.100	0.01	84.1～99.7
		1.000	0.01	80.4～95.1

Table C.1（continued）

Compound	Sample	Fortified level mg/kg	Limit of determination and confirmation mg/kg	Range of recovery %
carbaryl	soybean	0.010	0.01	80.0～95.0
		0.100	0.01	82.6～95.3
		1.000	0.01	78.6～93.3
	peanut	0.010	0.01	83.0～99.0
		0.100	0.01	82.5～107.4
		1.000	0.01	82.7～99.8
	bovine liver	0.010	0.01	83.0～101.0
		0.100	0.01	79.3～98.5
		1.000	0.01	77.6～93.6
	chicken kidney	0.010	0.01	82.0～97.0
		0.100	0.01	81.4～97.8
		1.000	0.01	82.4～96.7
	honey	0.010	0.01	81.0～96.0
		0.100	0.01	80.1～94.6
		1.000	0.01	83.5～98.7
ethiofencarb	brown rice	0.010	0.01	84.0～97.0
		0.100	0.01	77.5～95.8
		1.000	0.01	81.4～95.5
	corn	0.010	0.01	81.0～109.0
		0.100	0.01	81.5～99.3
		1.000	0.01	77.4～95.1
	chinese cabbage	0.010	0.01	82.0～94.0
		0.100	0.01	80.2～99.8
		1.000	0.01	88.6～100.2
	scallion	0.010	0.01	89.0～102.0
		0.100	0.01	83.6～96.1
		1.000	0.01	81.6～95.3
	barley	0.010	0.01	80.0～96.0
		0.100	0.01	81.0～97.3
		1.000	0.01	83.3～96.5
	wheat	0.010	0.01	83.0～97.0
		0.100	0.01	83.6～97.6
		1.000	0.01	85.1～99.1
	apple	0.010	0.01	81.0～96.0
		0.100	0.01	81.4～95.2
		1.000	0.01	82.2～94.2
	orange	0.010	0.01	79.0～91.0
		0.100	0.01	82.9～98.4
		1.000	0.01	81.6～93.5
	soybean	0.010	0.01	83.0～95.0
		0.100	0.01	83.8～97.1
		1.000	0.01	80.6～96.3

Table C.1（continued）

Compound	Sample	Fortified level mg/kg	Limit of determination and confirmation mg/kg	Range of recovery %
ethiofencarb	peanut	0.010	0.01	82.0～96.0
		0.100	0.01	83.4～97.7
		1.000	0.01	81.4～98.6
	bovine liver	0.010	0.01	81.0～95.0
		0.100	0.01	80.6～96.6
		1.000	0.01	81.2～95.3
	chicken kidney	0.010	0.01	81.0～96.0
		0.100	0.01	81.1～94.5
		1.000	0.01	82.1～97.0
	honey	0.010	0.01	80.0～98.0
		0.100	0.01	81.2～96.0
		1.000	0.01	83.6～94.6
isoprocarb	brown rice	0.010	0.01	81.0～95.0
		0.100	0.01	81.2～99.7
		1.000	0.01	80.1～98.1
	corn	0.010	0.01	81.0～95.0
		0.100	0.01	80.1～96.3
		1.000	0.01	80.4～95.1
	chinese cabbage	0.010	0.01	82.0～95.0
		0.100	0.01	82.5～97.6
		1.000	0.01	80.0～96.1
	scallion	0.010	0.01	81.0～95.0
		0.100	0.01	84.9～104.0
		1.000	0.01	81.4～96.8
	barley	0.010	0.01	81.0～95.0
		0.100	0.01	81.0～97.4
		1.000	0.01	81.3～97.6
	wheat	0.010	0.01	81.0～103.0
		0.100	0.01	81.4～97.5
		1.000	0.01	81.3～96.7
	apple	0.010	0.01	80.0～95.0
		0.100	0.01	81.2～98.2
		1.000	0.01	80.7～96.6
	orange	0.010	0.01	79.0～96.0
		0.100	0.01	82.6～95.5
		1.000	0.01	80.7～96.1
	soybean	0.010	0.01	81.0～95.0
		0.100	0.01	80.3～91.2
		1.000	0.01	81.0～95.2
	peanut	0.010	0.01	86.0～100.0
		0.100	0.01	83.7～97.6
		1.000	0.01	83.6～96.0

Table C.1（continued）

Compound	Sample	Fortified level mg/kg	Limit of determination and confirmation mg/kg	Range of recovery %
isoprocarb	bovine liver	0.010	0.01	83.0～96.0
		0.100	0.01	80.2～96.5
		1.000	0.01	80.3～97.6
	chicken kidney	0.010	0.01	81.0～96.0
		0.100	0.01	80.3～96.9
		1.000	0.01	80.1～95.5
	honey	0.010	0.01	81.0～103.0
		0.100	0.01	85.9～105.0
		1.000	0.01	80.2～95.6
diethofencarb	brown rice	0.010	0.01	80.0～101.0
		0.100	0.01	80.1～97.3
		1.000	0.01	80.9～96.7
	corn	0.010	0.01	81.0～97.0
		0.100	0.01	81.1～97.5
		1.000	0.01	81.7～97.7
	chinese cabbage	0.010	0.01	86.0～101.0
		0.100	0.01	80.0～98.2
		1.000	0.01	80.8～95.7
	scallion	0.010	0.01	79.0～94.0
		0.100	0.01	80.5～102.1
		1.000	0.01	80.7～97.7
	barley	0.010	0.01	84.0～103.0
		0.100	0.01	80.9～97.2
		1.000	0.01	77.9～95.0
	wheat	0.010	0.01	82.0～98.0
		0.100	0.01	80.2～98.4
		1.000	0.01	81.5～98.8
	apple	0.010	0.01	85.0～104.0
		0.100	0.01	81.5～100.9
		1.000	0.01	79.1～96.9
	orange	0.010	0.01	80.0～100.0
		0.100	0.01	81.2～98.7
		1.000	0.01	83.6～95.9
	soybean	0.010	0.01	85.0～106.0
		0.100	0.01	81.7～93.5
		1.000	0.01	84.2～99.1
	peanut	0.010	0.01	76.0～93.0
		0.100	0.01	80.6～98.0
		1.000	0.01	80.3～97.3
	bovine liver	0.010	0.01	84.0～100.0
		0.100	0.01	82.5～103.6
		1.000	0.01	81.2～97.1
	chicken kidney	0.010	0.01	84.0～102.0
		0.100	0.01	82.3～98.5
		1.000	0.01	80.4～96.6

Table C.1（continued）

Compound	Sample	Fortified level mg/kg	Limit of determination and confirmation mg/kg	Range of recovery %
diethofencarb	honey	0.010	0.01	82.0～96.0
		0.100	0.01	83.7～99.2
		1.000	0.01	78.7～95.9
fenobucarb	brown rice	0.010	0.01	84.0～99.0
		0.100	0.01	80.9～98.7
		1.000	0.01	81.5～99.0
	corn	0.010	0.01	85.0～96.0
		0.100	0.01	80.0～95.8
		1.000	0.01	82.3～95.6
	chinese cabbage	0.010	0.01	82.0～98.0
		0.100	0.01	82.7～97.5
		1.000	0.01	80.1～96.8
	scallion	0.010	0.01	82.0～96.0
		0.100	0.01	81.7～98.4
		1.000	0.01	82.6～96.7
	barley	0.010	0.01	86.0～101.0
		0.100	0.01	86.9～97.8
		1.000	0.01	80.6～98.1
	wheat	0.010	0.01	84.0～96.0
		0.100	0.01	84.1～93.7
		1.000	0.01	80.5～96.3
	apple	0.010	0.01	82.0～97.0
		0.100	0.01	82.9～98.4
		1.000	0.01	81.7～95.1
	orange	0.010	0.01	82.0～95.0
		0.100	0.01	82.4～97.0
		1.000	0.01	80.1～98.0
	soybean	0.010	0.01	81.0～97.0
		0.100	0.01	82.5～97.1
		1.000	0.01	80.2～98.3
	peanut	0.010	0.01	81.0～99.0
		0.100	0.01	83.2～97.3
		1.000	0.01	85.1～103.1
	bovine liver	0.010	0.01	82.0～104.0
		0.100	0.01	81.2～94.7
		1.000	0.01	82.0～97.5
	chicken kidney	0.010	0.01	81.0～97.0
		0.100	0.01	81.0～96.3
		1.000	0.01	81.8～94.7
	honey	0.010	0.01	82.0～96.0
		0.100	0.01	81.7～96.4
		1.000	0.01	80.3～93.7

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SN/T 0134—2010
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