SN

中华人民共和国出入境检验检疫行业标准

SN/T 2915—2011

出口食品中甲草胺、乙草胺、甲基吡恶磷等160种农药残留量的检测方法 气相色谱-质谱法

Determination of 160 pesticides residues including alachlor, acetochlor, azamethiphos in foodstuffs for 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：中国检验检疫科学研究院、中华人民共和国深圳出入境检验检疫局、中华人民共和国河北出入境检验检疫局、中华人民共和国云南出入境检验检疫局。

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出口食品中甲草胺、乙草胺、甲基吡恶磷等160种农药残留量的检测方法 气相色谱-质谱法

1 范围

本标准规定了食品中160种农药(参见附录A)残留量的气相色谱-质谱检测方法。

本标准适用于大米、糙米、大麦、小麦、玉米中160种农药残留量的测定。

2 方法提要

试样加水浸泡后用丙酮振荡提取,提取液经液-液分配法、凝胶渗透色谱法和固相萃取法净化,用气相色谱-质谱仪检测,标准曲线外标法定量。

3 试剂和材料

除另有说明,所有试剂均为分析纯,水为蒸馏水或相当纯度的水。

3.1 丙酮:色谱纯。

3.2 丙酮。

3.3 二氯甲烷。

3.4 环己烷。

3.5 乙酸乙酯。

3.6 正己烷。

3.7 氯化钠。

3.8 助滤剂:celite 545,或相当者。

3.9 无水硫酸钠:使用前在650 ℃灼烧4 h,贮于干燥器中,冷却后备用。

3.10 乙酸乙酯-环己烷(1+1,体积比):取等体积的乙酸乙酯和环己烷溶液混合均匀。

3.11 丙酮-正己烷(1+1,体积比):取等体积的丙酮(3.2)和正己烷混合均匀。

3.12 15%氯化钠水溶液(质量浓度):称取氯化钠15 g,用蒸馏水溶解并定容至100 mL。

3.13 以季胺盐强阴离子/乙二胺-*N*-丙基硅烷基化硅胶为填料的固相萃取柱:SAX/PSA,250 mg/250 mg,3 mL,或相当者。

3.14 农药标准物质:标准物质清单参见附录A,标准物质纯度≥95%。

3.15 农药标准溶液

3.15.1 标准储备溶液

准确称取各农药标准物质10 mg(精确至0.1 mg)置于10 mL容量瓶中,用丙酮(3.1)溶解定容,混合均匀,置于15 mL具塞并配有聚四氟乙烯密封垫的棕色样品瓶中,于−18 ℃保存。有效期3个月。

3.15.2 混合标准溶液

将160种农药进行分组(参见附录A),移取一定体积的标准储备溶液用丙酮(3.1)配制成10 mg/L的混合标准溶液,于0 ℃～4 ℃保存。有效期1个月。

3.15.3 基质混合标准工作溶液

分别移取一定体积的混合标准溶液，添加至经过6.1～6.2步骤净化后的空白样品基质溶液，混匀，用丙酮(3.1)定容至1.0 mL。基质混合标准工作溶液应现用现配。

4 仪器和设备

4.1 分析天平：感量为0.01 g。
4.2 分析天平：感量为0.1 mg。
4.3 样品粉碎机。
4.4 样品筛：40目(0.425 mm)。
4.5 振荡器。
4.6 抽滤装置。
4.7 旋转蒸发仪。
4.8 氮吹仪。
4.9 涡旋振荡器。
4.10 固相萃取装置。
4.11 凝胶渗透色谱仪：配凝胶色谱柱[Bio-beads S-X3，300 mm×25 mm(内径)]。
4.12 气相色谱-质谱仪：配有电子轰击电离源(EI)。

5 试样制备与保存

5.1 试样制备

取代表性试样500 g，用粉碎机粉碎，并使其全部通过420 μm的样品筛。混合均匀，装入洁净的容器内，密封并标识。

5.2 试样保存

试样于0 ℃～4 ℃避光保存。取样、制样及保存过程中应防止试样受到污染或者残留农药含量发生变化。

6 测定步骤

6.1 提取

称取试样20 g(精确至0.01 g)置于锥形瓶中，加入20 mL水放置30 min。加入80 mL丙酮(3.2)，置于振荡器上振荡提取30 min。将适量的助滤剂加入抽滤漏斗内，然后将试样及提取液转移至抽滤漏斗上，减压抽滤，再用3×5 mL丙酮(3.2)洗涤锥形瓶及残渣，合并提取液和洗涤液，于40 ℃水浴旋转浓缩至约20 mL。将上述溶液转移至分液漏斗中，依次向分液漏斗中加入50 mL的15%氯化钠溶液、50 mL二氯甲烷，置于振荡器上振荡5 min，静置分层后，二氯甲烷层溶液过无水硫酸钠脱水。再次加入50 mL二氯甲烷至分液漏斗中，重复上述操作。合并二氯甲烷层溶液，于40 ℃水浴中旋转浓缩至近干，再用氮气吹干。用环己烷-乙酸乙酯(3.10)定容至4.0 mL，待净化。

6.2 净化

6.2.1 凝胶渗透色谱净化

将6.1中待净化的溶液置于凝胶色谱仪上，进样体积为2.0 mL(相当于称取样品总量的一半)，以环己烷-乙酸乙酯作为流动相，流速为3.0 mL/min。弃去0 mL～20 mL淋洗液，收集21 mL～70 mL淋洗液，于40 ℃水浴旋转浓缩至近干，再用氮气吹干。用2 mL正己烷溶解残渣，待固相萃取净化。

6.2.2 固相萃取净化

将SAX/PSA固相萃取柱置于固相萃取装置上。分别用2 mL丙酮(3.2)、6 mL正己烷预淋洗。将6.2.1中待净化的溶液移入固相萃取柱，用2×1 mL正己烷洗涤容器并移入固相萃取柱上，弃去淋洗液，调节装置使淋洗液流速约为2 mL/min。再用5 mL丙酮-正己烷(3.11)溶液洗涤固相萃取柱，收集淋洗液。于40 ℃水浴旋转浓缩至近干，再用氮气吹干。用丙酮(3.1)定容至1.0 mL，供气相色谱-质谱测定。

6.3 测定

6.3.1 气相色谱-质谱条件

6.3.1.1 色谱柱：DB-5MS(30 m×0.25 mm×0.25 μm)石英毛细管柱或相当者。

6.3.1.2 色谱柱温度：本标准中被测的160农药采用2次进样完成测定，条件如下：

条件1：60 ℃保持2 min，以25 ℃/min升温至130 ℃，然后以2 ℃/min升温至220 ℃，再以20 ℃/min升温至300 ℃，保持6 min；

条件2：60 ℃保持2 min，以25 ℃/min升温至130 ℃，然后以4 ℃/min升温至180 ℃，再以6 ℃/min升温至300 ℃，保持12 min。

6.3.1.3 载气：氦气，纯度≥99.999%，流速1.0 mL/min。

6.3.1.4 进样口温度：250 ℃。

6.3.1.5 进样方式：脉冲不分流进样，脉冲压力137.9 kPa(20 psi)，保持1.5 min。

6.3.1.6 进样量：2 μL。

6.3.1.7 电子轰击电离源：70 eV。

6.3.1.8 离子源温度：230 ℃。

6.3.1.9 四极杆温度：150 ℃。

6.3.1.10 GC-MS接口温度：280 ℃。

6.3.1.11 溶剂延迟时间：在条件1下溶剂延迟时间为11 min，在条件2下溶剂延迟时间为9 min。

6.3.1.12 选择离子监测：每种农药选择2个～3个定性离子，其中包括1个定量离子，依据农药的保留时间分组检测。农药的保留时间、监测离子参见附录B。每组监测离子的分组时间和驻留时间参见附录B。

6.3.2 定性测定

进行样品测定时，如果样品质量色谱图中出现与标准物质保留时间一致的色谱峰，且在扣除背景后，该色谱峰对应的质谱图中出现所选择的离子(离子丰度比与标准物质离子丰度比的偏差满足残留分析要求)，则可以判断样品中存在这种农药。

6.3.3 定量测定

本标准采用基质混合标准工作溶液，外标法定量测定。基质混合标准工作溶液线性范围为

0.1 mg/kg～1.0 mg/kg。待测样品中各农药响应值应在基质混合标准工作溶液线性范围内。本标准中各标准物质的质量色谱图参见附录C。

6.4 平行试验

按照上述步骤对同一试样进行平行试验。

6.5 空白试验

除不称取试样外，均按照上述步骤进行。

7 结果计算

试样中农药残留量可由GC-MS数据处理软件计算，或按照式(1)计算：

$$X_i = \frac{A \times c \times V}{A_s \times m \times 1\,000} \quad \cdots\cdots(1)$$

式中：

X_i ——试样中农药残留量，单位为毫克每千克(mg/kg)；

A ——样液中农药定量离子色谱峰的峰面积；

c ——基质标准溶液中农药的浓度，单位为毫克每升(mg/L)；

V ——试样液最终定容体积，单位为毫升(mL)；

A_s ——基质标准溶液中农药定量离子色谱峰的峰面积；

m ——最终溶液所相当的试样质量，单位为克(g)。

注：计算结果应将空白值扣除。

8 测定低限、回收率

8.1 测定低限

采用本方法对大米、糙米、大麦、小麦和玉米5种食品中160种农药残留量进行测定，各种农药的测定低限为0.01 mg/kg。

8.2 回收率

采用本方法对大米、糙米、大麦、小麦和玉米5种食品进行添加回收实验，添加水平为0.01 mg/kg、0.05 mg/kg、0.1 mg/kg，160种农药在5种食品中的添加回收率参见附录D。

附 录 A
（资料性附录）
农药标准物质基本信息

表 A.1 农药中文名称、英文名称、CAS 编号、混合标准溶液分组

序号	中文名称	英文名称	CAS编号	混合标准溶液分组
1	乙滴涕	1,1-dichloro-2,2-bis(4-ethylphenyl)ethane	72-56-0	1
2	1-萘乙酰胺	2-(1-naphthyl)acetamide	86-86-2	1
3	乙草胺	acetochlor	34256-82-1	1
4	活化酯	acibenzoral-s-methyl	135158-54-2	1
5	甲草胺	alachlor	15972-60-8	1
6	甲基吡恶磷	azamethiphos	35575-96-3	1
7	燕麦灵	barban	101-27-9	1
8	苯霜灵	benalaxyl	71626-11-4	1
9	丙硫克百威	benfuracarb	82560-54-1	1
10	甲羧除草醚	bifenox	42576-02-3	1
11	联苯	biphenyl	92-52-4	1
12	联苯三唑醇	bitertanol	70585-36-3	1
13	烟酰胺	boscalid	188425-85-6	1
14	除草定	bromacil	314-40-9	1
15	溴硫磷	bromophos	2104-96-3	1
16	溴螨酯	bromopropylate	18181-80-1	1
17	丁草胺	butachlor	23184-66-9	1
18	唑草胺	cafenstrole	125306-83-4	1
19	双酰草胺	carbetamide	16118-49-3	1
20	唑草酮	carfentrazone-ethyl	128621-72-7	1
21	氯炔灵	chlorbufam	1967-16-4	2
22	氯丹	chlordane	57-74-9	2
23	氯氧磷	chlorethoxyfos	54593-83-8	2
24	乙酯杀螨醇	chlorobenzilate	510-15-6	2
25	地茂散	chloroneb	2675-77-6	2
26	氯苯胺灵	chlorpropham	101-21-3	2
27	乙菌利	chlozolinate	72391-46-9	2
28	吲哚酮草酯	cinidon-ethyl	142891-20-1	2
29	环庚草醚	cinmethylin	87818-31-3	2

表 A.1（续）

序号	中文名称	英文名称	CAS编号	混合标准溶液分组
30	异恶草松	clomazone	81777-89-1	2
31	氰草津	cyanazine	21725-46-2	2
32	杀螟腈	cyanophos	2636-26-2	2
33	氰氟草酯	cyhalofop-butyl	122008-85-9	2
34	环丙唑醇	cyproconazole	94361-06-5	2
35	嘧菌环胺	cyprodinil	121552-61-2	2
36	甲基内吸磷	demeton-s-methyl	919-86-8	2
37	除线磷	dichlofenthion	97-17-6	2
38	氯硝胺	dicloran	99-30-9	2
39	双氯氰菌胺	diclocymet	139920-32-4	2
40	三氯杀螨醇	dicofol	115-32-2	2
41	乙霉威	diethofencarb	87130-20-9	3
42	苯醚甲环唑	difenoconazole	119446-68-3	3
43	吡氟酰草胺	diflufenican	83164-33-4	3
44	异戊乙净	dimethametryn	22936-75-0	3
45	二甲吩草胺	dimethenamid	87674-68-8	3
46	噻节因	dimethipin	55290-64-7	3
47	烯唑醇	diniconazole	83657-24-3	3
48	乙拌磷砜	disulfoton sulfone	2497-6-5	3
49	氟硫草定	dithiopyr	97886-45-8	3
50	硫丹	endosulfan	115-29-7	3
51	乙硫磷	ethion	563-12-2	3
52	乙氧苯草胺	etobenzanid	79540-50-4	3
53	恶唑菌酮	famoxadone	131807-57-3	3
54	氯苯嘧啶醇	fenarimol	60168-88-9	3
55	腈苯唑	fenbuconazole	114369-43-6	3
56	氰菌胺	fenoxanil	115852-48-7	3
57	苯氧威	fenoxycarb	72490-01-8	3
58	甲氰菊酯	fenpropathrin	64257-84-7	3
59	芬螨酯	fenson	80-38-6	3
60	氟虫腈	fipronil	120068-37-3	3
61	麦草氟甲酯	flamprop-methyl	52756-25-9	4
62	咯菌腈	fludioxonil	131341-86-1	4

表 A.1（续）

序号	中文名称	英文名称	CAS编号	混合标准溶液分组
63	氟哒嗪草酯	flufenpyr-ethyl	188489-07-8	4
64	氟喹唑	fluquinconazole	136426-54-5	4
65	氟硅唑	flusilazole	85509-19-9	4
66	氟酰胺	flutolanil	66332-96-5	4
67	福拉比	furametpyr	123572-88-3	4
68	己唑醇	hexaconazole	79983-71-4	4
69	环嗪酮	hexazinone	51235-04-2	4
70	噻螨酮	hexythiazox	78587-05-0	4
71	抑霉唑	imazalil	35554-44-0	4
72	亚胺唑	imibenconazole	86598-92-7	4
73	茚草酮	indanofan	133220-30-1	4
74	辛酰碘苯腈	ioxynil-octanoate	3861-47-0	4
75	种菌唑	ipconazole	125225-28-7	4
76	异稻瘟净	iprobenfos	26087-47-8	4
77	异丙菌胺	iprovalicarb	140923-17-7	4
78	水胺硫磷	isocarbophos	24353-61-5	4
79	异柳磷	isofenphos	25311-71-1	4
80	氧异柳磷	isofenphos-oxon		4
81	异丙禾灵	isopropalin	33820-53-0	5
82	恶唑磷	isoxathion	18854-01-8	5
83	醚菌酯	kresoxim-methyl	143390-89-0	5
84	环草定	lenacil	2164/8/1	5
85	林丹	lindan	58-89-9	5
86	利谷隆	linuron	330-55-2	5
87	灭蚜磷	mecarbam	2595-54-2	5
88	苯噻酰草胺	mefenacet	73250-68-7	5
89	嘧菌胺	mepanipyrim	110235-47-7	5
90	灭锈胺	mepronil	55814-41-0	5
91	甲霜灵	metalaxyl	57837-19-1	5
92	杀扑磷	methidathion	950-37-8	5
93	烯虫酯	methoprene	40596-69-8	5
94	甲氧滴滴涕	methoxychlor	72-43-5	5
95	异丙甲草胺	metolachlor	51218-45-2	5

表 A.1（续）

序号	中文名称	英文名称	CAS编号	混合标准溶液分组
96	嗪草酮	metribuzin	21087-64-9	5
97	增效胺	MGK264	3547-33-9	5
98	禾草敌	molinate	2212-67-1	5
99	绿谷隆	monolinuron	1746-81-2	5
100	腈菌唑	myclobutanil	88671-89-0	5
101	酞菌酯	nitrothal-isopropyl	10552-74-6	6
102	九氯	nonachlor	3734-49-4	6
103	解草腈	oxabetrinil	74782-23-3	6
104	恶草酮	oxadiazon	19666-30-9	6
105	恶霜灵	oxadixyl	77732-09-3	6
106	氧化萎锈灵	oxycarboxin	5259-88-1	6
107	多效唑	paclobutrazol	76738-62-0	6
108	戊菌唑	penconazole	66246-88-6	6
109	二甲戊灵	pendimethalin	40487-42-1	6
110	苯醚菊酯	phenothrin	26002-80-2	6
111	伏杀硫磷	phosalone	2310-17-0	6
112	亚胺硫磷	phosmet	732-11-6	6
113	氟吡酰草胺	picolinafen	137641-05-5	6
114	稗草丹	piributicarb	88678-67-5	6
115	抗蚜威	pirimicarb	23103-98-2	6
116	烯丙苯噻唑	probenazole	27605-76-1	6
117	腐霉利	procymidone	32809-16-8	6
118	丙溴磷	profenofos	41198-08-7	6
119	茉莉酸诱导体	prohydrojasmon	158474-72-7	6
120	猛杀威	promecarb	2631-37-0	6
121	扑草净	prometryn	7287-19-6	7
122	炔螨特	propargite	2312-35-8	7
123	丙环唑	propiconazole	60207-90-1	7
124	残杀威	propoxur	114-26-1	7
125	炔苯酰草胺	propyzamide	23950-58-5	7
126	丙硫磷	prothiofos	34643-46-4	7
127	霸草灵	pyraflufen-ethyl	129630-19-9	7
128	哒螨灵	pyridaben	96489-71-3	7

表 A.1（续）

序号	中文名称	英文名称	CAS编号	混合标准溶液分组
129	哒嗪硫磷	pyridafenthion	119-12-0	7
130	啶虫丙醚	pyridalyl	179101-81-6	7
131	啶斑肟	pyrifenox	88283-41-4	7
132	环酯草醚	pyriftalid	135186-78-6	7
133	嘧霉胺	pyrimethanil	53112-28-0	7
134	嘧螨醚	pyrimidifen	105779-78-0	7
135	嘧草醚	pyriminobac-methyl	136191-64-5	7
136	喹氧灵	quinoxyfen	124495-18-7	7
137	蔬果磷	salithion	3811-49-2	7
138	硅醚菊酯	silafluofen	105024-66-6	7
139	西玛津	simazine	122-34-9	7
140	西草净	simetryn	1014-70-6	7
141	季酮甲螨酯	spiromesifen	283594-90-1	8
142	戊唑醇	tebuconazole	107534-96-3	8
143	吡螨胺	tebufenpyrad	119168-77-3	8
144	特草定	terbacil	5902-51-2	8
145	特丁净	terbutryn	886-50-0	8
146	杀虫威	tetrachlorvinphos	22248-79-9	8
147	四氟醚唑	tetraconazole	112281-77-3	8
148	三氯杀螨砜	tetradifon	116-29-0	8
149	噻吩草胺	thenylchlor	96491-05-3	8
150	噻草啶	thiazopyr	117718-60-2	8
151	噻呋灭	thifluzamide	130000-40-7	8
152	禾草丹	thiobencarb	28249-77-6	8
153	三甲苯草酮	tralkoxydim	87820-88-0	8
154	三环唑	tricyclazole	41814-78-2	8
155	布洛芬	trifloxystrobin	141517-21-7	8
156	氟菌唑	triflumizole	99387-89-0	8
157	杀铃脲	triflumuron	64628-44-0	8
158	氟乐灵	trifluralin	1582-09-8	8
159	单克素	uniconazole p	83657-17-4	8
160	乙烯菌核利	vinclozoline	50471-44-8	8

注：第二列中文通用名中斜体为有同分异构体的化合物。

附 录 B
（资料性附录）
GC-MS 测定的质谱条件

表 B.1 在程序升温条件 1 下，各种农药的保留时间和监测离子

序号	中文名称	英文名称	保留时间 min	监测离子		
1	杀铃脲	triflumuron	12.268	***139***	111	155
2	地茂散	chloroneb	13.889	***193***	208	206
3	残杀威	propoxur	17.462	***110***	111	152
4	甲基内吸磷	demeton-s-methyl	17.786	***88***	142	109
5	猛杀威	promecarb	20.98	***135***	150	136
6	氯硝胺	dichloran	21.822	***124***	206	176
7	西玛津	simazine	22.887	***201***	186	173
8	噻节因	dimethipin	23.094	***54***	118	210
9	异恶草松	clomazone	23.243	***204***	125	127
10	烯丙苯噻唑	probenazole(1)	23.581	***159***	130	117
11	炔苯酰草胺	propyzamide	24.632	***173***	175	145
12	嘧霉胺	pyrimethanil	25.078	***198***	199	
13	特草定	terbacil	26.09	***161***	163	117
14	异稻瘟净	iprobenfos	26.911	***204***	246	91
15	二甲吩草胺	dimethenamid	28.067	***230***	203	154
16	除线磷	dichlofenthion	28.18	***279***	251	281
17	乙草胺	acetochlor	28.581	***146***	234	162
18	乙烯菌核利	vinclozoline	29.094	***285***	212	198
19	甲草胺	alachlor	29.293	***160***	188	237
20	扑草净	prometryn	30.614	***184***	241	226
21	氟硫草定	dithiopyr	30.839	***354***	306	286
22	烯丙苯噻唑	probenazole(2)	31.57	***159***	130	117
23	特丁净	terbutryn	31.606	***226***	241	185
24	利谷隆	linuron	31.804	***61***	248	160
25	除草定	bromacil	32.193	***233***	231	207
26	噻草啶	thiazopyr	32.718	***327***	363	381
27	乙霉威	diethofencarb	33.747	***267***	225	151
28	三氯杀螨醇	dicofol	33.924	***251***	139	141

表 B.1（续）

序号	中文名称	英文名称	保留时间 min	监测离子		
29	异柳磷	isofenphos	34.122	***229***	201	314
30	溴硫磷	bromophos	34.513	***331***	333	329
31	酞菌酯	nitrothal-isopropyl	34.738	***236***	254	212
32	戊菌唑	penconazole	36.25	***248***	159	250
33	啶斑肟	pyrifenox(1)	36.605	***262***	187	171
34	异戊乙净	dimethametryn	36.707	***212***	255	240
35	氧异柳磷	isofenphos-oxon	36.807	***213***	255	121
36	腐霉利	procymidone	37.43	***283***	285	96
37	氯丹	chlordane(trans)	37.945	***373***	375	377
38	杀扑磷	methidathion	38.217	***145***	85	302
39	噻螨酮	hexythiazox	38.486	***227***	156	184
40	啶斑肟	pyrifenox(2)	38.861	***262***	187	171
41	硫丹	endosulfan(alfa)	38.95	***241***	265	339
42	氯丹	chlordane(cis)	39.044	***373***	375	377
43	杀虫威	tetrachlorvinphos	39.099	***329***	331	333
44	九氯	nonachlor(trans)	39.343	***409***	407	411
45	乙拌磷砜	disulfoton sulfone	39.588	***153***	213	185
46	嘧菌胺	mepanipyrim	40.155	***222***	223	207
47	丙硫磷	prothiofos	40.942	***309***	267	269
48	丙溴磷	profenofos	41.418	***339***	337	208
49	麦草氟甲酯	flamprop-methyl	42.267	***105***	276	230
50	恶草酮	oxadiazon	42.29	***258***	302	175
51	亚胺唑	imibenconazole	42.577	***235***	270	272
52	异丙菌胺	iprovalicarb(1)	42.842	***116***	158	
53	恶唑磷	isoxathion	43.703	***313***	177	134
54	异丙菌胺	iprovalicarb(2)	43.75	***116***	158	
55	硫丹	endosulfan(beta)	44.211	***241***	195	339
56	九氯	nonachlor(cis)	44.71	***409***	407	411
57	乙酯杀螨醇	chlorobenzilate	44.756	***251***	139	253
58	恶霜灵	oxadixyl	45.234	***163***	132	105
59	甲基吡恶磷	azamethiphos	47.283	***215***	183	125
60	苯霜灵	benalaxyl	47.47	***148***	206	325

表 B.1（续）

序号	中文名称	英文名称	保留时间 min	监测离子		
61	喹氧灵	quinoxyfen	47.999	***237***	272	307
62	唑草酮	carfentrazone-ethyl	48.186	***312***	330	340
63	环草定	lenacil	48.471	***153***	136	110
64	布洛芬	trifloxystrobin	49.052	***116***	131	222
65	己唑醇	hexazinone	49.156	***171***	252	128
66	炔螨特	propargite(1)	50.438	***135***	350	173
67	氧化萎锈灵	oxycarboxin	50.505	***175***	267	93
68	炔螨特	propargite(2)	50.531	***135***	350	173
69	哒嗪硫磷	pyridafenthion	51.47	***340***	199	188
70	亚胺硫磷	phosmet	51.527	***160***	161	133
71	溴螨酯	bromopropylate	51.813	***341***	183	339
72	氟吡酰草胺	picolinafen	51.963	***376***	238	239
73	苯氧威	fenoxycarb	52.072	***255***	186	116
74	甲氰菊酯	fenpropathrin	52.243	***181***	349	265
75	吡螨胺	tebufenpyrad	52.378	***318***	333	276
76	三氯杀螨砜	tetradifon	52.539	***356***	159	229
77	苯醚菊酯	phenothrin(1)	52.638	***123***	183	350
78	伏杀硫磷	phosalone	52.731	***182***	367	154
79	苯醚菊酯	phenothrin(2)	52.809	***123***	183	350
80	三甲苯草酮	tralkoxydim	53.136	***283***	226	268
81	辛酰碘苯腈	ioxynil-octanoate	53.38	***127***	226	243
82	环酯草醚	pyriftalid	53.437	***318***	274	303
83	丙硫克百威	benfuracarb	53.53	***190***	163	353
84	氟喹唑	fluquinconazole	54.2	***340***	341	342
85	哒螨灵	pyridaben	54.246	***117***	309	364
86	腈苯唑	fenbuconazole	54.615	***198***	129	125
87	嘧螨醚	pyrimidifen	55.873	***184***	186	185
88	恶唑菌酮	famoxadone	57.598	***330***	197	315
89	吲哚酮草酯	cinidon-ethyl	58.631	***330***	358	393

注：监测离子第一列内斜体加粗标记的离子为定量离子。

表 B.2　在程序升温条件 2 下，各种农药的保留时间和监测离子

序号	中文名称	英文名称	保留时间 min	监测离子		
1	联苯	biphenyl	9.907	***154***	153	152
2	禾草敌	molinate	13.020	***126***	187	98
3	氯氧磷	chlorethoxyfos	14.613	***299***	301	263
4	氯苯胺灵	chlorpropham	15.661	***127***	213	154
5	氟乐灵	trifluralin	15.817	***306***	264	290
6	蔬果磷	salithion	15.879	***216***	183	201
7	绿谷隆	monolinuron	17.768	***61***	214	126
8	氯炔灵	chlorbufam	17.944	***53***	223	164
9	林丹	lindan	18.100	***181***	183	217
10	杀螟腈	cyanophos	18.385	***109***	125	243
11	茉莉酸诱导体	prohydrojasmon	19.210	***83***	153	184
12	抗蚜威	pirimicarb	19.755	***166***	238	72
13	解草腈	oxabetrinil	19.791	***73***	103	
14	嗪草酮	metribuzin	20.647	***198***	199	144
15	活化酯	acibenzoral-s-methyl	21.088	***182***	181	153
16	西草净	simetryn	21.104	***213***	170	155
17	甲霜灵	metalaxyl	21.208	***206***	146	234
18	环庚草醚	cinmethylin	21.317	***105***	123	169
19	1-萘乙酰胺	2-(1-naphthyl)acetamide	21.690	***141***	142	185
20	异丙甲草胺	metolachlor	22.318	***238***	240	162
21	禾草丹	thiobencarb	22.484	***100***	72	257
22	氰草津	cyanazine	22.728	***225***	240	198
23	水胺硫磷	isocarbophos	22.904	***136***	230	289
24	四氟醚唑	tetraconazole	22.925	***336***	338	337
25	双酰草胺	carbetamide	23.034	***119***	120	236
26	芬螨酯	fenson	23.148	***77***	141	268
27	增效胺	mgk264(1)	23.325	***164***	177	210
28	异丙禾灵	isopripalin	23.475	***238***	264	280
29	二甲戊灵	pendimethalin	23.636	***252***	281	162
30	嘧菌环胺	cyprodinil	23.698	***224***	225	210
31	增效胺	mgk264(2)	23.776	***164***	177	210
32	氟虫腈	fipronil	23.932	***367***	369	351
33	乙菌利	chlozolinate	23.968	***259***	331	188

表 B.2（续）

序号	中文名称	英文名称	保留时间 min	监测离子		
34	灭蚜磷	mecarbam	24.150	***131***	159	329
35	双氯氰菌胺	diclocymet(1)	24.254	***277***	173	221
36	氟菌唑	triflumizole	24.477	***278***	206	287
37	烯虫酯	methoprene	24.679	***73***	109	107
38	双氯氰菌胺	diclocymet(2)	24.835	***277***	173	221
39	多效唑	paclobutrazol	25.006	***236***	167	238
40	丁草胺	butachlor	25.079	***176***	188	160
41	氟酰胺	flutolanil	25.654	***145***	173	323
42	抑霉唑	imazalil	25.655	***215***	217	173
43	已唑醇	hexaconazole	25.660	***214***	216	231
44	三环唑	tricyclazole	25.737	***189***	161	162
45	咯菌腈	fludioxonil	25.758	***248***	189	154
46	单克素	uniconazole p	26.028	***234***	236	235
47	噻呋灭	thifluzamide	26.116	***449***	447	427
48	腈菌唑	myclobutanil	26.168	***179***	181	288
49	燕麦灵	barban	26.184	***222***	224	141
50	氟硅唑	flusilazole	26.225	***233***	206	315
51	醚菌酯	kresoxim-methyl	26.334	***116***	131	206
52	环丙唑醇	cyproconazole(1)	26.656	***222***	224	139
53	环丙唑醇	cyproconazole(2)	26.708	***222***	224	139
54	氰菌胺	fenoxanil(1)	26.791	***189***	293	191
55	氰菌胺	fenoxanil(2)	26.827	***189***	293	191
56	乙滴涕	1,1-dichloro-2,2-bis(4-ethylphenyl)ethane	26.879	***223***	224	139
57	嘧草醚	pyriminobac-methyl(z)	27.107	***302***	256	230
58	氟哒嗪草酯	flufenpyr-ethyl	27.133	***408***	335	345
59	烯唑醇	diniconazole	27.237	***268***	270	232
60	乙硫磷	ethion	27.413	***231***	384	233
61	灭锈胺	mepronil	27.891	***119***	269	91
62	丙环唑	propiconazole(1)	28.373	***173***	259	261
63	嘧草醚	pyriminobac-methyl(e)	28.503	***302***	256	230
64	丙环唑	propiconazole(2)	28.570	***173***	259	261
65	霸草灵	pyraflufen-ethyl	28.638	***412***	349	339

表 B.2（续）

序号	中文名称	英文名称	保留时间 min	监测离子		
66	噻吩草胺	thenylchlor	28.866	***127***	288	141
67	戊唑醇	tebuconazole	29.058	***125***	250	127
68	吡氟酰草胺	diflufenican	29.193	***266***	394	267
69	季酮甲螨酯	spiromesifen	29.608	***272***	254	370
70	稗草丹	piributicarb	29.681	***165***	181	108
71	甲氧滴滴涕	methoxychlor	30.381	***227***	228	274
72	茚草酮	indanofan	30.604	***174***	159	310
73	甲羧除草醚	bifenox	30.708	***341***	343	310
74	福拉比	furametpyr	30.864	***298***	157	291
75	苯噻酰草胺	mefenacet	31.606	***192***	298	120
76	氰氟草酯	cyhalofop-butyl	31.627	***256***	357	229
77	种菌唑	ipconazole	31.979	***333***	249	
78	氯苯嘧啶醇	fenarimol	32.099	***139***	251	330
79	联苯三唑醇	bitertanol(1)	33.048	***170***	268	171
80	联苯三唑醇	bitertanol(2)	33.235	***170***	268	171
81	唑草胺	cafenstrole	33.873	***100***	188	72
82	乙氧苯草胺	etobenzanid	33.873	***179***	149	121
83	烟酰胺	boscalid	34.672	***140***	342	344
84	啶虫丙醚	pyriproxyfen	35.207	***204***	164	
85	硅醚菊酯	silafluofen	35.388	***179***	286	258
86	苯醚甲环唑	difenoconazole(1)	36.789	***323***	265	267
87	苯醚甲环唑	difenoconazole(2)	36.898	***323***	265	267
注：监测离子第一列内斜体加粗标记的离子为定量离子。						

表 B.3　在程序升温条件 1 下，农药选择离子监测分组和驻留时间参数

序号	时间 min	选择离子 amu	驻留时间 ms
1	11.00	111,139,155	100
2	13.20	193,206,208	100
3	16.00	88,109,110,111,142,152	70
4	20.00	135,136,150	100
5	21.40	124,176,206	100
6	22.60	54,117,118,125,127,130,159,173,186,201,204,210	40
7	24.00	145,173,175,198,199	70

表 B.3（续）

序号	时间 min	选择离子 amu	驻留时间 ms
8	25.50	117,161,163	100
9	26.50	91,204,246	100
10	27.50	154,203,230,251,279,281	70
11	28.40	146,162,234	100
12	28.85	160,188,198,212,237,285	70
13	30.00	184,226,241,286,306,354	70
14	31.20	61,117,130,159,160,185,207,226,231,233,241,248	40
15	32.45	327,363,381	100
16	33.20	139,141,151,201,225,229,251,267,314	50
17	34.31	212,236,254,329,331,333	70
18	35.50	121,159,171,187,212,213,240,248,250,255,262	40
19	37.10	85,96,145,156,184,227,283,285,302,373,375,377	40
20	38.65	171,187,241,262,265,329,331,333,339,373,375,377	40
21	39.21	153,185,207,213,222,223,407,409,411	50
22	40.55	267,269,309	100
23	41.15	208,337,339	100
24	41.80	105,116,158,175,230,235,258,270,272,276,302	40
25	43.25	116,134,158,177,195,241,313,339,407,409,411	40
26	44.45	139,251,253	100
27	45.00	105,132,163	100
28	46.30	125,148,183,206,215,325	70
29	47.75	110,136,153,237,272,307,312,330,340	50
30	48.75	116,128,131,171,222,252	70
31	49.80	93,135,173,175,267,350	70
32	51.00	133,160,161,188,199,340	70
33	51.67	116,181,183,186,238,239,255,265,339,341,349,376	40
34	52.31	159,229,276,318,333,356	70
35	52.59	123,154,182,183,350,367	70
36	53.00	127,163,190,226,243,268,274,283,303,318,353	40
37	53.85	117,309,340,341,342,364	70
38	54.50	125,129,198	100
39	55.20	184,185,186	100
40	57.20	197,315,330	100
41	58.20	330,358,393	100

表 B.4　在程序升温条件 2 下，农药选择离子监测分组和驻留时间参数

序号	时间 min	选择离子 amu	驻留时间 ms
1	9.00	152,153,154	100
2	12.80	98,126,187	100
3	14.40	263,299,301	100
4	15.40	127,154,213	100
5	15.75	183,201,216,264,290,306	70
6	17.50	61,126,214	100
7	17.85	53,164,181,183,217,223	70
8	18.25	109,125,243	100
9	19.10	83,153,184	100
10	19.60	72,73,103,166,238	80
11	20.10	144,198,199	100
12	20.90	105,123,146,153,155,169,170,181,182,206,213,234	40
13	21.50	141,142,185	100
14	22.10	72,100,162,238,240,257	70
15	22.60	198,225,240	100
16	22.80	77,119,120,136,141,230,236,268,289,336,337,338	40
17	23.25	164,177,210,238,264,280	70
18	23.55	162,164,177,210,224,225,252,281	50
19	23.85	188,259,331,351,367,369	70
20	24.05	131,159,173,221,277,329	70
21	24.35	206,278,287	100
22	24.60	73,107,109,173,221,277	70
23	24.90	160,167,176,188,236,238	70
24	25.50	145,173,214,215,216,217,231,323	50
25	25.70	154,161,162,189,248	70
26	25.95	141,179,181,222,224,234,235,236,288,427,447,449	40
27	26.13	116,131,206,233,315	70
28	26.55	139,222,224	100
29	26.75	139,189,191,223,224,293	70
30	27.00	230,232,256,268,270,302,335,345,408	50
31	27.35	231,233,384	100
32	27.75	91,119,269	100
33	28.10	173,259,261	100

表 B.4（续）

序号	时间 min	选择离子 amu	驻留时间 ms
34	28.45	173,230,256,259,261,302,339,349,412	50
35	28.75	127,141,288	100
36	28.95	125,127,250,266,267,394	70
37	29.50	108,165,181,254,272,370	70
38	30.20	227,228,274	100
39	30.50	159,174,310,341,343	70
40	30.78	157,291,298	100
41	31.50	120,192,229,256,298,357	70
42	31.80	139,249,251,330,333	70
43	32.90	170,171,268	100
44	33.80	72,100,121,149,179,188	70
45	34.10	140,342,244	100
46	35.10	164,204	100
47	35.30	179,258,286	100
48	36.60	265,267,323	100

附 录 C
（资料性附录）
农药标准物质的质量色谱图



图 C.1 在程序升温条件 1 下，各种农药的质量色谱图

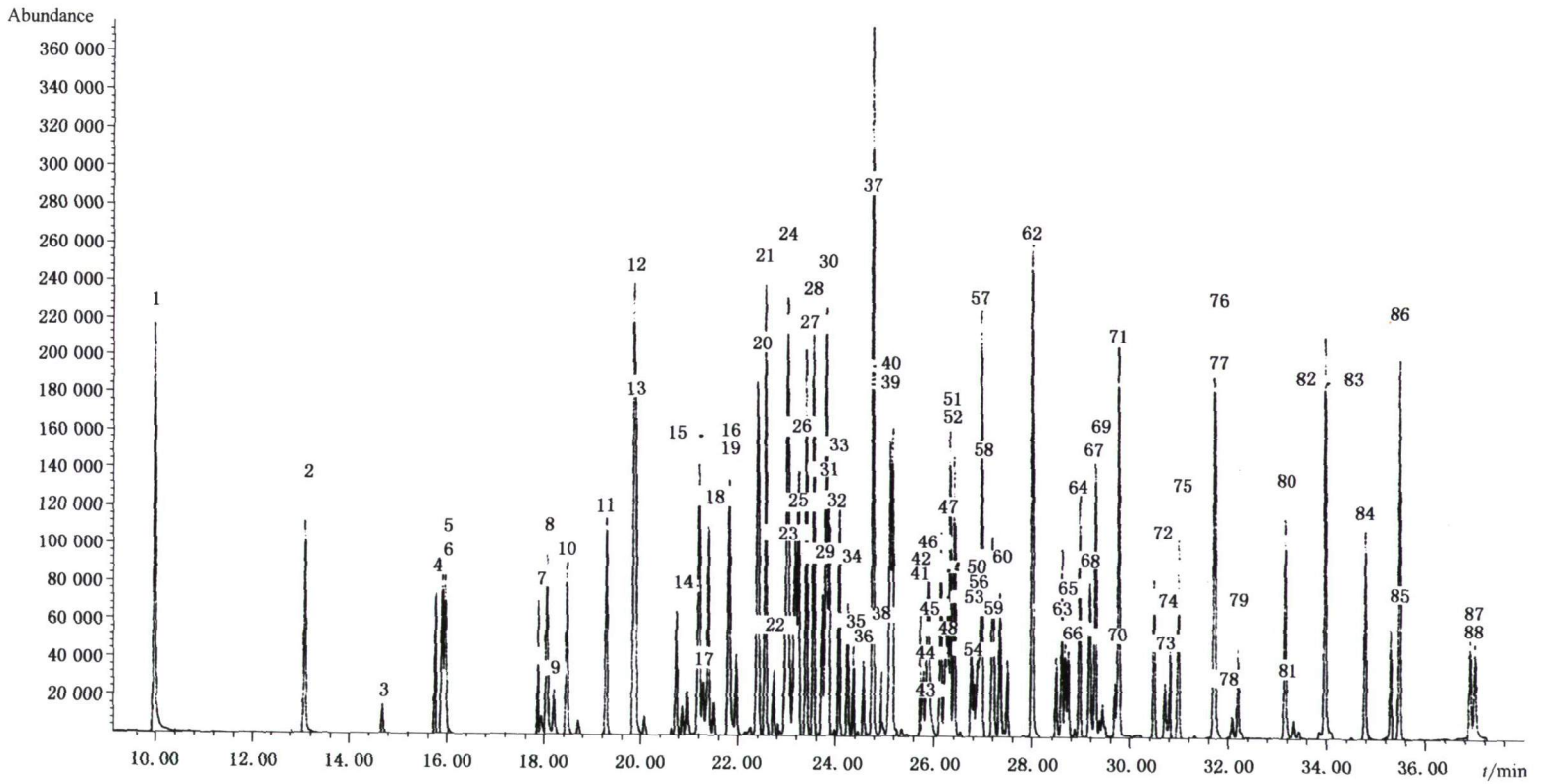


图 C.2　在程序升温条件 2 下，各种农药的质量色谱图

附 录 D
（资料性附录）
回收率范围

表 D.1 农药在大米、糙米、小麦、大麦及玉米中的添加回收率范围

序号	中文名称	英文名称	添加回收率范围 %				
			大米	糙米	小麦	大麦	玉米
1	乙滴涕	1,1-dichloro-2,2-bis(4-ethylphenyl)ethane	71.0～94.2	70.9～106.1	79.2～103.4	89.0～107.2	73.0～93.5
2	1-萘乙酰胺	2-(1-naphthyl)acetamide	74.3～97.4	83.0～106.6	74.3～97.8	71.4～95.3	73.6～100.2
3	乙草胺	acetochlor	83.2～107.9	82.2～107.1	86.7～109.4	87.0～94.9	85.1～93.2
4	活化酯	acibenzoral-s-methyl	76.0～108.4	72.6～102.5	81.2～94.6	81.9～93.6	79.0～106.6
5	甲草胺	alachlor	69.2～96.9	76.7～101.0	77.4～102.9	82.0～101.6	78.1～103.7
6	甲基吡恶磷	azamethiphos	85.4～104.0	84.3～109.1	81.9～106.3	72.0～95.5	71.6～96.2
7	燕麦灵	barban	77.7～98.2	76.1～96.3	84.4～99.7	72.4～97.9	85.3～96.4
8	苯霜灵	benalaxyl	73.9～100.8	87.2～94.9	88.3～103.1	84.1～100.8	87.6～107.7
9	丙硫克百威	benfuracarb	76.0～108.0	79.9～98.3	87.6～93.3	77.0～108.5	75.6～105.3
10	甲羧除草醚	bifenox	73.6～100.2	86.3～93.8	80.0～109.0	70.8～96.2	75.6～100.0
11	联苯	biphenyl	72.5～106.3	80.8～99.1	82.1～102.3	87.1～108.0	72.5～95.5
12	联苯三唑醇	bitertanol	78.1～97.7	86.2～106.0	80.9～101.2	75.5～102.8	86.1～94.2
13	烟酰胺	boscalid	72.2～94.5	85.0～105.2	73.0～109.6	87.1～107.8	78.8～96.7
14	除草定	bromacil	83.1～93.8	77.0～101.1	83.7～95.7	88.5～102.3	75.4～105.5
15	溴硫磷	bromophos	86.0～107.0	77.1～102.6	85.5～104.4	69.6～106.5	75.1～105.8
16	溴螨酯	bromopropylate	71.8～106.4	72.1～103.2	81.6～93.2	81.8～102.6	81.2～100.1
17	丁草胺	butachlor	88.2～107.5	79.0～100.0	75.1～99.2	76.1～108.2	76.9～93.6
18	唑草胺	cafenstrole	69.2～108.9	72.4～108.8	75.3～99.5	76.4～95.9	77.4～108.1
19	双酰草胺	carbetamide	84.1～105.1	70.5～106.2	70.1～100.5	69.5～94.1	71.0～105.0
20	唑草酮	carfentrazone-ethyl	77.1～95.7	81.0～100.7	71.5～107.4	76.6～96.4	74.4～108.1
21	氯炔灵	chlorbufam	78.5～96.7	70.8～102.4	84.9～105.5	69.7～108.4	84.0～104.2
22	氯丹	chlordane	88.5～101.3	74.6～103.1	71.3～97.8	78.3～100.2	70.1～96.6
23	氯氧磷	chlorethoxyfos	86.8～93.2	76.1～98.3	71.2～104.6	75.7～102.6	71.6～93.3
24	乙酯杀螨醇	chlorobenzilate	75.0～93.6	71.2～101.8	77.8～106.8	79.8～96.6	79.5～104.5
25	地茂散	chloroneb	83.1～101.4	71.5～107.1	69.1～100.5	71.5～101.3	80.0～96.8
26	氯苯胺灵	chlorpropham	78.2～98.7	88.5～103.0	73.7～107.9	88.3～109.3	77.1～99.7
27	乙菌利	chlozolinate	80.3～107.6	85.9～106.5	70.1～104.4	77.7～101.0	75.4～99.3

表 D.1（续）

序号	中文名称	英文名称	添加回收率范围 %				
			大米	糙米	小麦	大麦	玉米
28	吲哚酮草酯	cinidon-ethyl	86.5～100.8	80.0～107.2	74.1～96.7	78.7～105.4	77.7～103.7
29	环庚草醚	cinmethylin	77.6～93.6	76.0～102.5	84.5～97.8	73.7～102.0	78.1～102.7
30	异恶草松	clomazone	74.0～108.0	88.1～108.8	86.8～97.8	74.2～94.3	77.0～103.9
31	氰草津	cyanazine	77.3～95.5	70.4～105.3	88.2～97.7	85.2～97.2	84.1～105.5
32	杀螟腈	cyanophos	88.2～95.5	74.0～104.9	71.3～94.7	75.2～108.6	81.1～108.4
33	氰氟草酯	cyhalofop-butyl	74.9～106.0	84.0～107.4	79.3～106.0	75.1～102.7	83.4～96.0
34	环丙唑醇	cyproconazole	87.9～95.5	70.4～105.1	73.9～101.5	79.9～93.6	70.5～93.7
35	嘧菌环胺	cyprodinil	72.1～103.8	80.5～99.3	85.2～100.4	88.1～96.6	86.3～104.5
36	甲基内吸磷	demeton-s-methyl	92.8～95.4	82.5～94.6	77.2～95.3	86.6～109.3	76.9～100.0
37	除线磷	dichlofenthion	70.9～93.7	79.6～94.4	83.6～102.9	88.6～106.0	69.3～100.6
38	氯硝胺	dicloran	74.9～109.9	85.1～98.3	85.2～99.7	81.7～94.0	82.2～104.5
39	双氯氰菌胺	diclocymet	75.8～106.4	77.7～106.5	70.2～106.8	72.7～93.5	71.6～97.4
40	三氯杀螨醇	dicofol	74.6～105.1	88.9～95.8	79.3～106.6	87.9～96.3	75.9～94.8
41	乙霉威	diethofencarb	81.4～101.8	85.9～100.5	74.9～98.7	72.8～107.0	71.2～96.9
42	苯醚甲环唑	difenoconazole	71.1～109.3	76.9～102.1	69.7～95.8	88.4～103.7	78.6～108.3
43	吡氟酰草胺	diflufenican	85.7～102.8	77.5～108.7	87.6～97.2	81.6～109.8	79.2～98.4
44	异戊乙净	dimethametryn	84.2～104.8	86.1～94.0	72.7～102.3	70.5～102.4	77.0～103.2
45	二甲吩草胺	dimethenamid	79.3～97.3	69.9～107.5	86.2～99.1	83.6～104.3	75.1～93.5
46	噻节因	dimethipin	87.2～106.5	78.6～93.1	78.6～102.0	77.1～108.9	87.5～93.4
47	烯唑醇	diniconazole	78.0～108.5	80.1～97.7	71.0～107.7	75.8～109.5	73.6～96.5
48	乙拌磷砜	disulfoton sulfone	86.8～93.6	71.7～105.0	73.6～100.0	69.8～102.5	70.6～105.1
49	氟硫草定	dithiopyr	85.7～103.7	79.5～94.4	88.6～102.0	71.9～99.3	76.2～104.5
50	硫丹	endosulfan	71.3～106.1	76.8～106.4	82.0～109.6	83.6～109.1	72.1～97.0
51	乙硫磷	ethion	75.5～100.4	73.2～108.8	87.5～99.9	75.9～96.8	78.0～100.4
52	乙氧苯草胺	etobenzanid	83.3～100.0	81.9～102.2	85.0～105.5	73.6～99.3	70.0～93.1
53	恶唑菌酮	famoxadone	85.6～97.2	73.3～103.1	78.2～96.5	85.3～100.7	69.7～98.1
54	氯苯嘧啶醇	fenarimol	87.3～95.6	70.5～107.4	87.2～107.2	83.8～104.1	78.4～98.6
55	腈苯唑	fenbuconazole	82.0～94.7	83.0～109.6	73.7～106.2	75.5～105.7	72.4～107.5
56	氰菌胺	fenoxanil	75.7～101.2	72.0～108.6	85.7～109.3	87.3～93.9	71.9～94.3
57	苯氧威	fenoxycarb	81.6～101.2	75.7～94.6	72.2～102.8	73.4～95.4	74.8～98.8
58	甲氰菊酯	fenpropathrin	82.8～103.1	72.9～106.5	70.4～94.0	80.3～97.7	75.3～103.3
59	芬螨酯	fenson	70.6～94.5	81.4～93.6	78.7～106.4	71.5～108.7	74.1～95.9

表 D.1（续）

序号	中文名称	英文名称	添加回收率范围 %				
			大米	糙米	小麦	大麦	玉米
60	氟虫腈	fipronil	82.4～108.4	81.1～108.9	73.4～97.9	75.3～105.9	76.3～94.6
61	麦草氟甲酯	flamprop-methyl	84.1～96.0	77.7～108.4	74.1～100.5	80.2～109.3	77.0～107.8
62	咯菌腈	fludioxonil	70.3～102.0	79.1～104.8	80.2～98.9	84.6～95.2	69.5～105.3
63	氟吡嗪草酯	flufenpyr-ethyl	88.8～96.1	75.4～108.0	81.6～95.6	76.3～107.4	82.5～98.5
64	氟喹唑	fluquinconazole	84.6～110.0	81.0～108.1	78.6～106.0	77.8～96.6	87.4～106.5
65	氟硅唑	flusilazole	82.9～96.6	85.9～97.1	77.3～96.8	88.4～102.0	85.1～98.5
66	氟酰胺	flutolanil	77.5～96.1	74.5～105.5	82.7～98.5	69.1～95.1	88.6～103.9
67	福拉比	furametpry	77.1～106.1	76.7～102.5	89.0～105.2	87.8～98.7	75.8～96.7
68	己唑醇	hexaconazole	81.1～105.2	78.1～107.4	74.9～95.1	74.0～99.6	78.9～94.2
69	环嗪酮	hexazinone	70.8～97.4	81.6～97.1	78.2～93.5	79.5～99.3	69.8～98.1
70	噻螨酮	hexythiazox	72.1～108.1	80.5～105.1	75.9～108.7	87.4～99.0	70.3～105.5
71	抑霉唑	imazalil	73.8～94.9	72.5～100.9	82.7～103.8	82.7～108.0	72.7～99.6
72	亚胺唑	imibenconazole	76.2～101.9	87.8～106.5	71.5～105.9	87.7～109.0	72.7～101.2
73	茚草酮	indanofan	72.2～108.7	82.3～101.1	85.0～99.9	72.8～101.1	83.3～99.4
74	辛酰碘苯腈	ioxynil-octanoate	86.8～99.4	87.1～100.1	81.3～98.4	75.3～106.9	86.4～102.4
75	种菌唑	ipconazole	78.3～106.1	80.4～94.4	70.2～101.8	72.9～100.7	72.5～95.5
76	异稻瘟净	iprobenfos	87.3～109.8	84.9～95.9	82.8～93.1	84.1～93.8	82.1～103.5
77	异丙菌胺	iprovalicarb	83.1～102.6	72.8～94.9	78.5～105.1	82.5～99.3	75.4～104.3
78	水胺硫磷	isocarbophos	69.7～103.7	76.6～94.3	75.6～102.6	82.6～104.4	81.1～95.4
79	异柳磷	isofenphos	86.3～105.1	77.9～106.8	78.1～109.0	85.7～97.4	87.8～103.3
80	氧异柳磷	isofenphos-oxon	81.0～93.5	77.3～103.3	73.4～96.8	79.5～110.0	75.0～98.0
81	异丙禾灵	isopropalin	87.2～96.5	85.4～94.0	86.5～101.9	75.0～106.5	83.7～106.4
82	恶唑磷	isoxathion	72.8～102.2	73.3～106.3	82.1～104.0	77.4～93.2	88.5～97.9
83	醚菌酯	kresoxim-methyl	71.7～95.2	82.5～100.5	88.0～108.6	76.4～101.5	82.1～97.8
84	环草定	lenacil	88.3～109.7	82.6～100.6	71.4～94.5	74.5～98.1	81.8～93.6
85	林丹	lindan	86.4～105.1	73.0～93.9	80.5～96.5	83.1～100.1	70.9～100.5
86	利谷隆	linuron	73.6～104.1	77.5～93.2	87.3～109.6	84.3～97.0	88.8～96.9
87	灭蚜磷	mecarbam	69.8～108.3	79.6～103.3	77.1～98.8	74.0～106.4	74.8～105.4
88	苯噻酰草胺	mefenacet	74.5～104.0	77.1～100.7	83.7～106.7	80.7～102.4	74.7～109.9
89	嘧菌胺	mepanipyrim	79.3～105.4	69.5～98.9	82.2～99.5	85.3～96.1	83.3～103.0
90	灭锈胺	mepronil	83.4～98.1	75.9～101.0	85.9～108.5	82.6～94.4	78.7～94.9
91	甲霜灵	metalaxyl	88.5～103.6	88.9～108.7	78.8～96.3	75.2～102.7	69.4～94.9

表 D.1（续）

序号	中文名称	英文名称	添加回收率范围 %				
			大米	糙米	小麦	大麦	玉米
92	杀扑磷	methidathion	87.2～93.9	88.2～95.8	71.7～103.7	84.1～104.7	72.5～106.8
93	烯虫酯	methoprene	70.5～95.5	75.7～100.8	70.6～94.4	71.5～101.7	84.9～104.4
94	甲氧滴滴涕	methoxychlor	74.2～96.8	80.9～100.1	71.7～105.6	84.9～109.4	75.1～97.5
95	异丙甲草胺	metolachlor	77.4～104.5	77.1～108.5	86.5～98.1	76.1～107.1	82.9～98.1
96	嗪草酮	metribuzin	74.5～108.3	79.5～98.6	79.6～93.0	86.0～100.5	81.8～109.4
97	增效胺	MGK264	79.9～100.1	83.0～100.5	69.2～105.3	83.8～101.8	75.0～94.7
98	禾草敌	molinate	76.9～98.3	84.3～107.4	88.4～94.2	70.9～101.1	77.9～99.6
99	绿谷隆	monolinuron	71.1～105.6	86.1～100.9	76.3～109.3	88.6～106.4	84.6～99.9
100	腈菌唑	myclobutanil	74.1～96.0	81.4～102.4	76.4～99.7	71.2～108.4	77.8～96.0
101	酞菌酯	nitrothal-isopropyl	79.7～102.6	69.1～107.5	87.5～103.8	79.0～98.3	70.9～97.9
102	九氯	nonachlor	75.5～105.5	78.9～93.5	74.7～107.9	83.9～103.3	71.0～101.9
103	解草腈	oxabetrinil	76.9～96.3	70.2～97.0	87.9～101.1	74.2～96.2	69.3～94.7
104	恶草酮	oxadiazon	78.3～99.6	82.1～108.5	74.4～109.3	80.4～107.7	78.9～108.4
105	恶霜灵	oxadixyl	83.3～108.5	72.8～109.8	74.0～107.9	78.3～105.4	72.3～108.9
106	氧化萎锈灵	oxycarboxin	73.3～104.6	86.8～101.9	79.7～103.7	69.6～106.4	69.9～93.7
107	多效唑	paclobutrazol	87.0～94.8	76.6～93.7	80.6～94.3	80.5～106.2	69.0～94.7
108	戊菌唑	penconazole	83.2～105.0	85.4～100.8	82.6～99.2	81.8～97.0	72.6～108.9
109	二甲戊灵	pendimethalin	83.4～104.2	86.6～106.1	88.9～97.8	81.2～101.1	79.4～94.1
110	苯醚菊酯	phenothrin	78.2～107.4	83.3～93.7	69.9～105.4	83.7～99.0	77.5～106.4
111	伏杀硫磷	phosalone	87.9～106.4	75.2～107.3	70.5～104.1	87.0～101.2	84.6～98.1
112	亚胺硫磷	phosmet	86.4～103.2	73.6～105.5	75.2～96.0	78.4～99.9	81.5～100.3
113	氟吡酰草胺	picolinafen	84.9～94.3	82.9～109.1	74.5～101.9	84.1～102.5	85.3～95.2
114	稗草丹	piributicarb	82.5～108.7	76.8～93.8	73.4～109.8	86.2～107.5	79.5～106.6
115	抗蚜威	pirimicarb	71.8～96.8	87.9～106.6	80.9～107.7	70.9～98.4	72.1～93.0
116	烯丙苯噻唑	probenazole	79.6～99.0	71.8～101.1	74.2～108.7	79.3～99.5	75.2～104.8
117	腐霉利	procymidone	79.2～96.8	72.1～101.9	75.5～99.6	85.5～103.6	80.2～109.1
118	丙溴磷	profenofos	71.5～98.3	86.7～97.3	78.5～100.6	85.1～95.3	80.8～99.1
119	茉莉酸诱导体	prohydrojasmon	76.4～96.9	80.1～106.9	82.2～94.3	84.2～97.6	74.2～97.3
120	猛杀威	promecarb	78.7～99.4	82.1～96.3	88.7～96.9	88.9～103.6	74.8～101.3
121	扑草净	prometryn	86.8～102.3	88.0～94.4	84.2～94.0	77.1～106.1	86.5～95.2
122	炔螨特	propargite	69.1～103.6	70.2～102.0	82.5～105.8	85.5～103.1	78.2～96.1
123	丙环唑	propiconazole	76.6～103.2	77.2～96.8	83.5～107.8	88.3～98.0	80.0～104.1

表 D.1（续）

序号	中文名称	英文名称	添加回收率范围 %				
			大米	糙米	小麦	大麦	玉米
124	残杀威	propoxur	82.8～98.0	77.7～98.9	82.2～98.8	80.2～107.2	87.1～103.0
125	炔苯酰草胺	propyzamide	69.6～107.3	71.6～104.2	82.0～102.0	77.3～99.3	77.5～98.9
126	丙硫磷	prothiofos	87.0～97.6	73.6～104.7	77.3～100.9	70.1～95.5	69.3～95.5
127	霸草灵	pyraflufen-ethyl	86.6～103.7	72.2～106.3	79.4～95.3	80.8～96.1	75.6～96.2
128	哒螨灵	pyridaben	80.2～103.6	84.5～94.9	81.3～96.5	86.8～98.8	87.6～100.1
129	哒嗪硫磷	pyridafenthion	76.0～93.2	82.9～100.0	86.5～100.8	74.5～99.3	84.4～96.0
130	啶虫丙醚	pyridalyl	73.5～102.4	79.8～99.5	83.8～100.5	88.8～99.6	86.5～98.4
131	啶斑肟	pyrifenox	86.4～93.8	70.1～97.4	70.3～108.7	72.5～97.3	72.9～102.0
132	环酯草醚	pyriftalid	78.8～103.0	77.5～103.3	81.0～106.3	87.4～106.3	78.9～103.0
133	嘧霉胺	pyrimethanil	70.8～94.0	72.6～93.8	83.5～109.8	83.4～101.8	87.5～99.5
134	嘧螨醚	pyrimidifen	79.0～94.4	77.4～105.5	81.8～96.1	80.4～100.6	88.1～109.4
135	嘧草醚	pyriminobac-methyl	73.0～104.0	70.9～101.2	70.2～105.7	74.2～94.1	75.6～103.6
136	喹氧灵	quinoxyfen	82.3～107.7	87.1～96.3	71.1～93.0	82.4～96.9	70.6～105.9
137	蔬果磷	salithion	70.9～107.2	70.8～98.6	69.4～99.4	84.7～103.7	86.7～97.6
138	硅醚菊酯	silafluofen	82.5～107.8	87.5～100.9	86.7～94.1	85.3～107.3	81.6～100.9
139	西玛津	simazine	76.4～94.9	72.8～93.1	76.2～105.1	73.9～94.3	87.3～96.7
140	西草净	simetryn	86.6～101.1	75.5～106.4	69.3～101.7	85.2～107.0	81.0～105.3
141	季酮甲螨酯	spiromesifen	73.5～101.7	71.5～108.9	84.1～100.6	76.9～107.3	73.1～93.8
142	戊唑醇	tebuconazole	72.6～104.8	75.2～95.1	81.3～93.5	77.7～103.7	84.8～95.8
143	吡螨胺	tebufenpyrad	81.5～103.8	86.3～103.4	83.5～107.7	76.9～93.1	73.0～108.7
144	特草定	terbacil	83.0～107.3	75.0～100.5	84.2～108.6	82.6～99.1	75.6～102.6
145	特丁净	terbutryn	80.9～97.3	83.7～93.9	84.1～108.6	76.7～94.2	77.2～105.8
146	杀虫威	tetrachlorvinphos	76.9～103.1	80.7～98.2	81.8～109.6	71.4～98.7	75.9～98.7
147	四氟醚唑	tetraconazole	72.3～99.4	70.9～109.3	72.9～101.2	79.6～101.1	81.8～109.4
148	三氯杀螨砜	tetradifon	74.8～104.3	76.5～104.5	83.5～97.0	83.8～106.2	84.1～93.6
149	噻吩草胺	thenylchlor	74.5～100.6	70.9～109.7	87.6～106.1	80.0～104.4	81.5～101.2
150	噻草啶	thiazopyr	81.0～108.5	70.8～99.6	87.4～96.6	84.0～93.9	81.5～108.1
151	噻呋灭	thifluzamide	82.2～97.6	85.0～94.4	71.7～94.4	80.1～104.0	76.0～93.4
152	禾草丹	thiobencarb	87.3～109.8	77.7～109.2	80.2～93.6	88.0～105.4	85.6～96.6
153	三甲苯草酮	tralkoxydim	70.9～106.4	82.5～93.0	73.4～95.2	77.4～95.3	75.0～96.8
154	三环唑	tricyclazole	83.7～97.3	87.5～96.3	81.0～98.7	86.0～98.2	88.8～95.1
155	布洛芬	trifloxystrobin	82.2～98.0	79.0～99.8	85.4～107.1	69.9～99.5	71.6～109.8

表 D.1（续）

序号	中文名称	英文名称	添加回收率范围 %				
			大米	糙米	小麦	大麦	玉米
156	氟菌唑	triflumizole	85.4～100.3	77.8～104.1	71.8～99.2	70.5～99.1	74.5～93.3
157	杀铃脲	triflumuron	69.6～93.9	88.1～106.0	82.0～103.1	72.2～101.9	75.4～104.7
158	氟乐灵	trifluralin	73.4～103.9	72.5～101.8	73.5～105.8	88.5～97.3	71.5～104.2
159	单克素	uniconazole p	87.6～95.0	69.7～93.8	73.8～94.8	79.1～110.0	88.5～106.4
160	乙烯菌核利	vinclozoline	83.1～95.8	86.7～99.5	76.8～94.2	82.3～93.1	84.0～100.8

Foreword

This standard was drafted according to the principle of the GB/T 1. 1—2009.

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 Chinese Academy of Inspection and Quarantine, Shenzhen Entry-Exit Inspection and Quarantine Bureau of the People's Republic of China, Hebei Entry-Exit Inspection and Quarantine Bureau of the People's Republic of China, Yunnan Entry – Exit Inspection and Quarantine Bureau of the People's Republic of China.

Main drafters of this standard are Li Xiaojuan, Li Shujuan, Li Gang, Chen Dongdong, Dai Hanhui, Peng Tao, Wang Fengchi, Lan Fang, Peng Yunxia, Feng Nan, Feng Fan, Cai Huixia, Tang Yingzhang.

Determination of 160 pesticides residues including alachlor, acetochlor, azamethiphos in foodstuffs for export—GC-MS method

1 Scope

This standard specifies the determination of 160 pesticides (more information in annex A) residues in foodstuffs by GC-MS.

This standard is applicable to the determination of 160 pesticides residues in rice, brown rice, barley, wheat and corn.

2 Principle

Sample is soaked in water, and the residues in the test sample are extracted with acetone. After cleaned up by liquid liquid partition, GPC and solid phase extraction cartridge, the residues are then determined by GC-MS, and quantified by external standard method.

3 Reagents and materials

Unless otherwise specified, all regents used are A. R., and "water" is distilled water or equivalent pure water.

3.1 Acetone: HPLC grade.

3.2 Acetone.

3.3 Dichloromethane.

3.4 Cyclohexane.

3.5 Ethyl acetate.

3.6 Hexane.

3.7 Sodium chloride.

3.8 Celite 545,or equivalent.

3.9 Anhydrous sodium sulfate:Burn at 650 ℃ for 4 h and then store in a desiccator.

3.10 Cyclohexane-ethyl acetate(1+1,*V*/*V*).

3.11 Acetone-hexane(1+1,*V*/*V*).

3.12 15% sodium chloride solution(*m*/*V*):15 g sodium chloride is dissolved in water,then dilute to 100 mL.

3.13 SPE colume:SAX/PSA,250 mg/250 mg,3 mL,or equivalent.

3.14 Pesticides standards:Purity≥95%. Other information see annex A.

3.15 Pesticides standards solution

3.15.1 Standard stock solution

Accurately weigh 10 mg of each standard(accurate to 0.1 mg)into 10 mL volumetric flask, dissolve in acetone(3.1)and dilute to volume,transfer it into 15 mL stoppered brown sample bottle with teflon seal,stored below −18 ℃,avoiding sunlight for three months.

3.15.2 Mix standard solution

Divide 160 pestcides into groups,more information in annex A. Accurately transfer adequate standard stock solution and dilute it with acetone(3.1)to 10 mg/L as the mix standard solution,stored at 0 ℃～4 ℃ avoiding sunlight for one month.

3.15.3 Mix standard working solution with matrix

Transfer adequate mix standard solution to sample blank matrix solution purified by 6.1～6.2 step, vortex and fix in 1.0 mL with acetone(3.1)just before use.

4 Apparatus and equipment

4.1 Balance:Accuracy to 0.01 g.

4.2 Balance:Accuracy to 0.1 mg.

4.3 Disintegrator.

4.4 Sieve:0.425 mm.

4.5 Shaker.

4.6 Pump and filtrate apparatus.

4.7 Rotary vacuum evaporator.

4.8 Nitrogen evaporator.

4.9 Vortex mixer.

4.10 SPE cartridge.

4.11 GPC:Column [Bio-beads S-X3,300 mm×25 mm(i.d.)].

4.12 GC-MS: EI source.

5 Sample preparation and storage

5.1 Sample preparation

All primary sample is reduced to 500 g as the representative sample, which is comminuted and passed through a 420 μm sieve, and then divided into two equal portions. Each portion is placed in clean containers as the test sample, which is sealed and labeled.

5.2 Sample storage

The test sample shall be stored at 0 ℃~4 ℃ avoiding sunlight. In the course of sample preparation, precautions shall be taken to avoid contamination or any factors which may cause the change of residue content.

6 Procedure

6.1 Extraction

Weigh about 20 g(accurate to 0.01 g) test sample, then 20 mL water and 80 mL acetone (3.2) are added and vortex it. After 30 min, shake for 30 min. Transfer all the extract into pump and filtrate apparatus, in which appropriate Celite 545 is added. And wash the taper flask and extract with 3×5 mL acetone(3.2), combine filtrate. The filtrate is reduced to about 20 mL in rotary vacuum evaporator at 40 ℃, then is transfered to separating funnel. 50 mL NaCl solution and 50 mL dichlo-

romethane is added in sequence. After shaking for 5 min, be still to wait. Then collect the dichloromethane layer and dehydrate it by anhydrous sodium sulfate. Another 50 mL dichloromethane is added into the separating funnel; repeat the liquid liquid partition, and combine the dichloromethane layer. Evaporate the dichloromethane layer to nearly dryness in rotary vacuum evaporator under 40 ℃ water, and blow it to dryness under nitrogen. The residue is reconstituted by 4 mL cyclohexane-ethyl acetate(3.10), and is ready for cleanup.

6.2 Cleanup

6.2.1 GPC cleanup

Put the solution which is ready to clean up in 6.1 on the GPC, the injection volume is 2.0 mL(equivalent to half the weight). The mobile phase is cyclohexane-ethyl acetate and the flow rate is 3.0 mL/min, discard 0 mL～20 mL pour solution and collect 21 mL～70 mL pour solution. Evaporate the pour solution to almost dryness with the rotary evaporator under 40 ℃ water. Then blow it to dryness completely with nitrogen gas. Use 2 mL hexane to resolve the residue.

6.2.2 SPE cleanup

Put the SAX/PSA columns on the SPE cartridge, use 2 mL acetone(3.2) and 6 mL hexane to wash it. Transfer the solution purified by 6.2.1 into the column. Use 2×1 mL hexane to clean the container and also transfer it into the SPE column. Control the flow rate of outflow to 2 mL/min. Then use 5 mL acetone-hexane(3.11) to wash the SPE column and in same time collect the outflow. Then evaporate it to almost dryness under 40 ℃ water. Then blow it to dryness completely with nitrogen gas. Use acetone (3.1) to fix to 1.0 mL which is ready for being analysed by GC-MS.

6.3 Determination

6.3.1 GC-MC condition

6.3.1.1 Column: DB-5MS(30 m×0.25 mm×0.25 μm) capillary column or equivalent.

6.3.1.2 Oven temperature: All 160 pestcides will be analysed by two GC-MS methods.

Method 1: 60 ℃ hold 2 min, firstly rise to 130 ℃ at 25 ℃/min, secondly rise to 220 ℃ at 2 ℃/min, finally rise to 300 ℃ at 20 ℃/min, hold 6 min;

Method 2: 60 ℃ hold 2 min, firstly rise to 130 ℃ at 25 ℃/min, secondly rise to 180 ℃ at 4 ℃/min, finally rise to 300 ℃ at 6 ℃/min, hold 12 min.

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

6.3.1.4 Injection port temperature: 250 ℃.

6.3.1.5 Injection mode:Pulsed splitless,pulsed pressure 137.9 kPa(20 psi),hold 1.5 min.

6.3.1.6 Injection volume:2 μL.

6.3.1.7 EI source:70 eV.

6.3.1.8 Ion source temperature:230 ℃.

6.3.1.9 Quadrupole temperature:15 ℃.

6.3.1.10 Interface temperature:280 ℃.

6.3.1.11 Solvent delay time:11 min in method 1,9 min in method 2.

6.3.1.12 Select ion monitor:Select one quantitative ion and one to three qualitative ions,every pestcide is determined by groups according to their retention time. Retention time and quantitative ion and qualitative ions are in annex B,the beginning and stop time of every group and dwell time are in annex B.

6.3.2 Qualitative determination

During the sample determination,if retention time of some peak in sample mass spectrum is same to the standard,and all the selected ions are in the mass spectrum(the deviation between sample ion abundance ratio and the standards is qualified for need for the residue analysis),then we can determine the appearance of pestcide.

6.3.3 Quantitative determination

To reduce the influence of matrix,this method uses sample blank solution to make the matrix mix standard working solution and use external standard to quantitative. The linearity concentration range of the matrix mix standard working solution is between 0.1 mg/kg～1.0 mg/kg. The pestcide response in the sample must in the linear range of the matrix mix standard working solution. The full－scan totle ion chromatogram of 160 pestcides is in annex C.

6.4 Parallel test

Parallel test is conducted to the same sample according to the procedure above.

6.5 Blank test

Blank test is conducted according to the procedure above without sample addition.

7 Calculation and expression of the result

Calculate the residue content of pesticide in the test sample by the data processor of GC-MS, or according to the formula(1):

$$X_i = \frac{A \times c \times V}{A_s \times m \times 1\,000} \quad \cdots\cdots(1)$$

Where

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

A —the peak area of pesticide in the test sample solution;

c —the concentration of pesticide in the matrix standard working solution, mg/L;

V —the final volume of sample solution, mL;

A_s —the peak area of pesticide in the matrix standard working solution;

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

Note: The blank value should be subtracted from the result of calculation.

8 Limit of quantification and recovery

8.1 Limit of quantification

Use this method to determine 160 pesticides in rice, brown rice, barley, wheat and corn, and LOQ is 0.01 mg/kg.

8.2 Recovery

Use this method to do spiked test in rice, brown rice, barley, wheat and corn, the spiked level is 0.01 mg/kg, 0.05 mg/kg, 0.1 mg/kg, the recovery of 160 pesticides is in annex D.

Annex A
(Informative)
Pesticides standards information

Table A.1—Information of 160 pesticides

No.	Compound name	CAS No.	Group
1	1,1-Dichloro-2,2-bis(4-ethylphenyl)ethane	72-56-0	1
2	2-(1-Naphthyl)acetamide	86-86-2	1
3	Acetochlor	34256-82-1	1
4	Acibenzolar-s-methyl	135158-54-2	1
5	Alachlor	15972-60-8	1
6	Azamethiphos	35575-96-3	1
7	Barban	101-27-9	1
8	Benalaxyl	71626-11-4	1
9	Benfuracarb	82560-54-1	1
10	Bifenox	42576-02-3	1
11	Biphenyl	92-52-4	1
12	Bitertanol[a]	70585-36-3	1
13	Boscalid	188425-85-6	1
14	Bromacil	314-40-9	1
15	Bromophos	2104-96-3	1
16	Bromopropylate	18181-80-1	1
17	Butachlor	23184-66-9	1
18	Cafenstrole	125306-83-4	1
19	Carbetamide	16118-49-3	1
20	Carfentrazone-ethyl	128621-72-7	1
21	Chlorbufam	1967-16-4	2
22	Chlordane[a]	57-74-9	2
23	Chlorethoxyfos	54593-83-8	2
24	Chlorobenzilate	510-15-6	2
25	Chloroneb	2675-77-6	2
26	Chlorpropham	101-21-3	2
27	Chlozolinate	72391-46-9	2

Table A. 1 (continued)

No.	Compound name	CAS No.	Group
28	Cinidon-ethyl	142891-20-1	2
29	Cinmethylin	87818-31-3	2
30	Clomazone	81777-89-1	2
31	Cyanazine	21725-46-2	2
32	Cyanophos	2636-26-2	2
33	Cyhalofop-butyl	122008-85-9	2
34	Cyproconazole[a]	94361-06-5	2
35	Cyprodinil	121552-61-2	2
36	Demeton-s-methyl	919-86-8	2
37	Dichlofenthion	97-17-6	2
38	Dicloran	99-30-9	2
39	Diclocymet[a]	139920-32-4	2
40	Dicofol	115-32-2	2
41	Diethofencarb	87130-20-9	3
42	Difenoconazole[a]	119446-68-3	3
43	Diflufenican	83164-33-4	3
44	Dimethametryn	22936-75-0	3
45	Dimethenamid	87674-68-8	3
46	Dimethipin	55290-64-7	3
47	Diniconazole	83657-24-3	3
48	Disulfoton sulfone	2497-6-5	3
49	Dithiopyr	97886-45-8	3
50	Endosulfan[a]	115-29-7	3
51	Ethion	563-12-2	3
52	Etobenzanid	79540-50-4	3
53	Famoxadone	131807-57-3	3
54	Fenarimol	60168-88-9	3
55	Fenbuconazole	114369-43-6	3
56	Fenoxanil[a]	115852-48-7	3
57	Fenoxycarb	72490-01-8	3
58	Fenpropathrin	64257-84-7	3

Table A. 1 (continued)

No.	Compound name	CAS No.	Group
59	Fenson	80-38-6	3
60	Fipronil	120068-37-3	3
61	Flamprop-methyl	52756-25-9	4
62	Fludioxonil	131341-86-1	4
63	Flufenpyr-ethyl	188489-07-8	4
64	Fluquinconazole	136426-54-5	4
65	Flusilazole	85509-19-9	4
66	Flutolanil	66332-96-5	4
67	Furametpry	123572-88-3	4
68	Hexaconazole	79983-71-4	4
69	Hexazinone	51235-04 -2	4
70	Hexythiazox	78587-05-0	4
71	Imazalil	35554 -44 -0	4
72	Imibenconazole	86598-92-7	4
73	Indanofan	133220-30-1	4
74	Ioxynil-octanoate	3861-47-0	4
75	Ipconazole	125225-28-7	4
76	Iprobenfos	26087-47-8	4
77	Iprovalicarb[a]	140923-17-7	4
78	Isocarbophos	24353-61-5	4
79	Isofenphos	25311-71-1	4
80	Isofenphos-oxon		4
81	Isopropalin	33820-53-0	5
82	Isoxathion	18854-01-8	5
83	Kresoxim-methyl	143390-89-0	5
84	Lenacil	2164/8/1	5
85	Lindan	58-89-9	5
86	Linuron	330-55-2	5
87	Mecarbam	2595-54-2	5
88	Mefenacet	73250-68-7	5
89	Mepanipyrim	110235-47-7	5

Table A. 1 (continued)

No.	Compound name	CAS No.	Group
90	Mepronil	55814-41-0	5
91	Metalaxyl	57837-19-1	5
92	Methidathion	950-37-8	5
93	Methoprene	40596-69-8	5
94	Methoxychlor	72-43-5	5
95	Metolachlor	51218-45-2	5
96	Metribuzin	21087-64-9	5
97	MGK264[a]	3547-33-9	5
98	Molinate	2212-67-1	5
99	Monolinuron	1746-81-2	5
100	Myclobutanil	88671-89-0	5
101	Nitrothal-isopropyl	10552-74-6	6
102	Nonachlor[a]	3734-49-4	6
103	Oxabetrinil	74782-23-3	6
104	Oxadiazon	19666-30-9	6
105	Oxadixyl	77732-09-3	6
106	Oxycarboxin	5259-88-1	6
107	Paclobutrazol	76738-62-0	6
108	Penconazole	66246-88-6	6
109	Pendimethalin	40487-42-1	6
110	Phenothrin[a]	26002-80-2	6
111	Phosalone	2310-17-0	6
112	Phosmet	732-11-6	6
113	Picolinafen	137641-05-5	6
114	Piributicarb	88678-67-5	6
115	Pirimicarb	23103-98-2	6
116	Probenazole[a]	27605-76-1	6
117	Procymidone	32809-16-8	6
118	Profenofos	41198-08-7	6
119	Prohydrojasmon	158474-72-7	6
120	Promecarb	2631-37-0	6

Table A.1 (continued)

No.	Compound name	CAS No.	Group
121	Prometryn	7287-19-6	7
122	Propargite[a]	2312-35-8	7
123	Propiconazole[a]	60207-90-1	7
124	Propoxur	114-26-1	7
125	Propyzamide	23950-58-5	7
126	Prothiofos	34643-46-4	7
127	Pyraflufen-ethyl	129630-19-9	7
128	Pyridaben	96489-71-3	7
129	Pyridafenthion	119-12-0	7
130	Pyridalyl	179101-81-6	7
131	Pyrifenox[a]	88283-41-4	7
132	Pyriftalid	135186-78-6	7
133	Pyrimethanil	53112-28-0	7
134	Pyrimidifen	105779-78-0	7
135	Pyriminobac-methyl[a]	136191-64-5	7
136	Quinoxyfen	124495-18-7	7
137	Salithion	3811-49-2	7
138	Silafluofen	105024-66-6	7
139	Simazine	122-34-9	7
140	Simetryn	1014-70-6	7
141	Spiromesifen	283594-90-1	8
142	Tebuconazole	107534-96-3	8
143	Tebufenpyrad	119168-77-3	8
144	Terbacil	5902-51-2	8
145	Terbutryn	886-50-0	8
146	Tetrachlorvinphos	22248-79-9	8
147	Tetraconazole	112281-77-3	8
148	Tetradifon	116-29-0	8
149	Thenylchlor	96491-05-3	8
150	Thiazopyr	117718-60-2	8
151	Thifluzamide	130000-40-7	8

Table A. 1 (continued)

No.	Compound name	CAS No.	Group
152	Thiobencarb	28249-77-6	8
153	Tralkoxydim	87820-88-0	8
154	Tricyclazole	41814-78-2	8
155	Trifloxystrobin	141517-21-7	8
156	Triflumizole	99387-89-0	8
157	Triflumuron	64628-44-0	8
158	Trifluralin	1582-09-8	8
159	Uniconazole p	3657-17-4	8
160	Vinclozoline	50471-44-8	8
[a] The compound occurs as isomers.			

Annex B
(Informative)
GC-MS condition

Table B.1—Retention time and selected ion under method 1

No.	Compound name	RT min	Monitor ions		
1	Triflumuron	12.268	*139*	111	155
2	Chloroneb	13.889	*193*	208	206
3	Propoxur	17.462	*110*	111	152
4	Demeton-s-methyl	17.786	*88*	142	109
5	Promecarb	20.98	*135*	150	136
6	Dichloran	21.822	*124*	206	176
7	Simazine	22.887	*201*	186	173
8	Dimethipin	23.094	*54*	118	210
9	Clomazone	23.243	*204*	125	127
10	Probenazole(1)	23.521	*159*	130	117
11	Propyzamide	24.632	*173*	175	145
12	Pyrimethanil	25.078	*198*	199	
13	Terbacil	26.03	*161*	163	117
14	Iprobenfos	26.911	*204*	246	91
15	Dimethenamid	28.067	*230*	203	154
16	Dichlofenthion	28.18	*279*	251	281
17	Acetochlor	28.581	*146*	234	162
18	Vinclozoline	29.094	*285*	212	198
19	Alachlor	29.293	*160*	188	237
20	Prometryn	30.614	*184*	241	226
21	Dithiopyr	30.839	*354*	306	286
22	Probenazole(2)	31.57	*159*	130	117
23	Terbutryn	31.606	*226*	241	185
24	Linuron	31.804	*61*	248	160
25	Bromacil	32.193	*233*	231	207
26	Thiazopyr	32.718	*327*	363	381

Table B. 1 (continued)

No.	Compound name	RT min	Monitor ions		
27	Diethofencarb	33.747	*267*	225	151
28	Dicofol	33.924	*251*	139	141
29	Isofenphos	34.122	*229*	201	314
30	Bromophos	34.513	*331*	333	329
31	Nitrothal-isopropyl	34.738	*236*	254	212
32	Penconazole	36.25	*248*	159	250
33	Pyrifenox(1)	36.605	*262*	187	171
34	Dimethametryn	36.707	*212*	255	240
35	Isofenphos-oxon	36.807	*213*	255	121
36	Procymidone	37.43	*283*	285	96
37	Chlordane(trans)	37.945	*373*	375	377
38	Methidathion	38.217	*145*	85	302
39	Hexythiazox	38.486	*227*	156	184
40	Pyrifenox(2)	38.861	*262*	187	171
41	Endosulfan(alfa)	38.95	*241*	265	339
42	Chlordane(cis)	39.044	*373*	375	377
43	Tetrachlorvinphos	39.099	*329*	331	333
44	Nonachlor(trans)	39.343	*409*	407	411
45	Disulfoton sulfone	39.558	*153*	213	185
46	Mepanipyrim	40.155	*222*	223	207
47	Prothiofos	40.942	*309*	267	269
48	Profenofos	41.418	*339*	337	208
49	Flamprop-methyl	42.267	*105*	276	230
50	Oxadiazon	42.29	*258*	302	175
51	Imibenconazole	42.577	*235*	270	272
52	Iprovalicarb(1)	42.842	*116*	158	
53	Isoxathion	43.703	*313*	177	134
54	Iprovalicarb(2)	43.75	*116*	158	
55	Endosulfan(beta)	44.211	*241*	195	339
56	Nonachlor(cis)	44.71	*409*	407	411

Table B. 1 (continued)

No.	Compound name	RT min	Monitor ions		
57	Chlorobenzilate	44.756	*251*	139	253
58	Oxadixyl	45.234	*163*	132	105
59	Azamethiphos	47.283	*215*	183	125
60	Benalaxyl	47.47	*148*	206	325
61	Quinoxyfen	47.999	*237*	272	307
62	Carfentrazone-ethyl	48.186	*312*	330	340
63	Lenacil	48.471	*153*	136	110
64	Trifloxystrobin	49.052	*116*	131	222
65	Hexazinone	49.156	*171*	252	128
66	Propargite(1)	50.438	*135*	350	173
67	Oxycarboxin	50.505	*175*	267	93
68	Propargite(2)	50.531	*135*	350	173
69	Pyridafenthion	51.47	*340*	199	188
70	Phosmet	51.527	*160*	161	133
71	Bromopropylate	51.813	*341*	183	339
72	Picolinafen	51.963	*376*	238	239
73	Fenoxycarb	52.072	*255*	186	116
74	Fenpropathrin	52.243	*181*	349	265
75	Tebufenpyrad	52.378	*318*	333	276
76	Tetradifon	52.539	*356*	159	229
77	Phenothrin(1)	52.638	*123*	183	350
78	Phosalone	52.731	*182*	367	154
79	Phenothrin(2)	52.809	*123*	183	350
80	Tralkoxydim	53.136	*283*	226	268
81	Ioxynil-octanoate	53.38	*127*	226	243
82	Pyriftalid	53.437	*318*	274	303
83	Benfuracarb	53.53	*190*	163	353
84	Fluquinconazole	54.2	*340*	341	342
85	Pyridaben	54.246	*117*	309	364
86	Fenbuconazole	54.615	*198*	129	125

Table B. 1 (continued)

No.	Compound name	RT min	Monitor ions		
87	Pyrimidifen	55.873	*184*	186	185
88	Famoxadone	57.598	*330*	197	315
89	Cinidon-ethyl	58.631	*330*	358	393
Note: The first line of monitor ion is quantitative ion.					

Table B.2 Retention time and selected ion under method 2

No.	Compound name	RT min	Monitor ions		
1	Biphenyl	9.907	*154*	153	152
2	Molinate	13.020	*126*	187	98
3	Chlorethoxyfos	14.613	*299*	301	263
4	Chlorpropham	15.661	*127*	213	154
5	Trifluralin	15.817	*306*	264	290
6	Salithion	15.879	*216*	183	201
7	Monolinuron	17.768	*61*	214	126
8	Chlorbufam	17.944	*53*	223	164
9	Lindan	18.100	*181*	183	217
10	Cyanophos	18.385	*109*	125	243
11	Prohydrojasmon	19.210	*83*	153	184
12	Pirimicarb	19.755	*166*	238	72
13	Oxabetrinil	19.791	*73*	103	
14	Metribuzin	20.647	*198*	199	144
15	Acibenzoral-s-methyl	21.088	*182*	181	153
16	Simetryn	21.104	*213*	170	155
17	Metalaxyl	21.208	*206*	146	234
18	Cinmethylin	21.317	*105*	123	169
19	2-(1-naphthyl)acetamide	21.690	*141*	142	185
20	Metolachlor	22.318	*238*	240	162
21	Thiobencarb	22.484	*100*	72	257
22	Cyanazine	22.728	*225*	240	198
23	Isocarbophos	22.904	*136*	230	289

Table B. 2 (continued)

No.	Compound name	RT min	Monitor ions		
24	Tetraconazole	22.925	*336*	338	337
25	Carbetamide	23.034	*119*	120	236
26	Fenson	23.148	*77*	141	268
27	Mgk264(1)	23.325	*164*	177	210
28	Isopripalin	23.475	*238*	264	280
29	Pendimethalin	23.636	*252*	281	162
30	Cyprodinil	23.698	*224*	225	210
31	Mgk264(2)	23.776	*164*	177	210
32	Fipronil	23.932	*367*	369	351
33	Chlozolinate	23.968	*259*	331	188
34	Mecarbam	24.150	*131*	159	329
35	Diclocymet(1)	24.254	*277*	173	221
36	Triflumizole	24.477	*278*	206	287
37	Methoprene	24.679	*73*	109	107
38	Diclocymet(2)	24.835	*277*	173	221
39	Paclobutrazol	25.006	*236*	167	238
40	Butachlor	25.079	*176*	188	160
41	Flutolanil	25.654	*145*	173	323
42	Imazalil	25.655	*215*	217	173
43	Hexaconazole	25.660	*214*	216	231
44	Tricyclazole	25.737	*189*	161	162
45	Fludioxonil	25.758	*248*	189	154
46	Uniconazole p	26.028	*234*	236	235
47	Thifluzamide	26.116	*449*	447	427
48	Myclobutanil	26.168	*179*	181	288
49	Barban	26.184	*222*	224	141
50	Flusilazole	26.225	*233*	206	315
51	Kresoxim-methyl	26.334	*116*	131	206
52	Cyproconazole(1)	26.656	*222*	224	139
53	Cyproconazole(2)	26.708	*222*	224	139

Table B. 2 (continued)

No.	Compound name	RT min	Monitor ions		
54	Fenoxanil(1)	26.791	*189*	293	191
55	fenoxanil(2)	26.827	*189*	293	191
56	1,1-dichloro-2,2-bis (4-ethylphenyl)ethane	26.879	*223*	224	139
57	Pyriminobac-methyl(z)	27.107	*302*	256	230
58	Flufenpyr-ethyl	27.133	408	*335*	345
59	Diniconazole	27.237	*268*	270	232
60	Ethion	27.413	*231*	384	233
61	Mepronil	27.891	*119*	269	91
62	Propiconazole(1)	28.373	*173*	259	261
63	Pyriminobac-methyl(e)	28.503	*302*	256	230
64	Propiconazole(2)	28.570	*173*	259	261
65	Pyraflufen-ethyl	28.638	*412*	349	339
66	Thenylchlor	28.866	*127*	288	141
67	Tebuconazole	29.058	*125*	250	127
68	Diflufenican	29.193	*266*	394	267
69	Spiromesifen	29.608	*272*	254	370
70	Piributicarb	29.681	*165*	181	108
71	Methoxychlor	30.381	*227*	228	274
72	Indanofan	30.604	*174*	159	310
73	Bifenox	30.703	*341*	343	310
74	Furametpyr	30.864	*298*	157	291
75	Mefenacet	31.606	*192*	298	120
76	Cyhalofop-butyl	31.627	*256*	357	229
77	Ipconazole	31.979	*333*	249	
78	Fenarimol	32.099	*139*	251	330
79	Bitertanol(1)	33.048	*170*	268	171
80	Bitertanol(2)	33.235	*170*	268	171
81	Cafenstrole	33.873	*100*	188	172
82	Etobenzanid	33.873	*179*	149	121
83	Boscalid	34.672	*140*	342	344

Table B. 2 (continued)

No.	Compound name	RT min	Monitor ions		
84	Pyridalyl	35.207	*204*	164	
85	Silafluofen	35.388	*179*	286	258
86	Difenoconazole(1)	36.789	*323*	265	267
87	Difenoconazole(2)	36.893	*323*	265	267
Note: The first line of monitor ion is quantitative ion.					

Table B.3—Groups of selected ion and dwell time under method 1

No.	Time min	Selected ion amu	Dwell ms
1	11.00	111,139,155	100
2	13.20	193,206,208	100
3	16.00	88,109,110,111,142,152	70
4	20.00	135,136,150	100
5	21.40	124,176,206	100
6	22.60	54,117,118,125,127,130,159,173,186,201,204,210	40
7	24.00	145,173,175,198,199	70
8	25.50	117,161,163	100
9	26.50	91,204,246	100
10	27.50	154,203,230,251,279,281	70
11	28.40	146,162,234	100
12	28.85	160,188,198,212,237,285	70
13	30.00	184,226,241,286,306,354	70
14	31.20	61,117,130,159,160,185,207,226,231,233,241,248	40
15	32.45	327,363,381	100
16	33.20	139,141,151,201,225,229,251,267,314	50
17	34.31	212,236,254,329,331,333	70
18	35.50	121,159,171,187,212,213,240,248,250,255,262	40
19	37.10	85,96,145,156,184,227,283,285,302,373,375,377	40
20	38.65	171,187,241,262,265,329,331,333,339,373,375,377	40
21	39.21	153,185,207,213,222,223,407,409,411	50
22	40.55	267,269,309	100
23	41.15	208,337,339	100
24	41.80	105,116,158,175,230,235,258,270,272,276,302	40

Table B.3 (continued)

No.	Time min	Selected ion amu	Dwell ms
25	43.25	116,134,158,177,195,241,313,339,407,409,411	40
26	44.45	139,251,253	100
27	45.00	105,132,163	100
28	46.30	125,148,183,206,215,325	70
29	47.75	110,136,153,237,272,307,312,330,340	50
30	48.75	116,128,131,171,222,252	70
31	49.80	93,135,173,175,267,350	70
32	51.00	133,160,161,188,199,340	70
33	51.67	116,181,183,186,238,239,255,265,339,341,349,376	40
34	52.31	159,229,276,318,333,356	70
35	52.59	123,154,182,183,350,367	70
36	53.00	127,163,190,226,243,268,274,283,303,318,353	40
37	53.85	117,309,340,341,342,364	70
38	54.50	125,129,198	100
39	55.20	184,185,186	100
40	57.20	197,315,330	100
41	58.20	330,358,393	100

Table B.4—Groups of selected ion and dwell time under method 2

No.	Time min	Selected ion amu	Dwell ms
1	9.00	152,153,154	100
2	12.80	98,126,187	100
3	14.40	263,299,301	100
4	15.40	127,154,213	100
5	15.75	183,201,216,264,290,306	70
6	17.50	61,126,214	100
7	17.85	53,164,181,183,217,223	70
8	18.25	109,125,243	100
9	19.10	83,153,184	100
10	19.60	72,73,103,166,238	80
11	20.10	144,198,199	100
12	20.90	105,123,146,153,155,169,170,181,182,206,213,234	40

Table B. 4 (continued)

No.	Time min	Selected ion amu	Dwell ms
13	21.50	141,142,185	100
14	22.10	72,100,162,238,240,257	70
15	22.60	198,225,240	100
16	22.80	77,119,120,136,141,230,236,268,289,336,337,338	40
17	23.25	164,177,210,238,264,280	70
18	23.55	162,164,177,210,224,225,252,281	50
19	23.85	188,259,331,351,367,369	70
20	24.05	131,159,173,221,277,329	70
21	24.35	206.278,287	100
22	24.60	73,107,109,173,221,277	70
23	24.90	160,167,176,188,236,238	70
24	25.50	145,173,214,215,216,217,231,323	50
25	25.70	154,161,162,189,248	70
26	25.95	141,179,181,222,224,234,235,236,288,427,447,449	40
27	26.13	116,131,206,233,315	70
28	26.55	139,222,224	100
29	26.75	139,189,191,223,224,293	70
30	27.00	230,232,256,268,270,302,335,345,408	50
31	27.35	231,233,384	100
32	27.75	91,119,269	100
33	28.10	173,259,261	100
34	28.45	173,230,256,259,261,302,339,349,412	50
35	28.75	127,141,288	100
36	28.95	125,127,250,266,267,394	70
37	29.50	108,165,181,254,272,370	70
38	30.20	227,228,274	100
39	30.50	159,174,310,341,343	70
40	30.78	157,291,298	100
41	31.50	120,192,229,256,298,357	70
42	31.80	139,249,251,330,333	70

Table B. 4 (continued)

No.	Time min	Selected ion amu	Dwell ms
43	32.90	170,171,268	100
44	33.80	72,100,121,149,179,188	70
45	34.10	140,342,244	100
46	35.10	164,204	100
47	35.30	179,258,286	100
48	36.60	265,267,323	100

Annex C
(Informative)
Chromatogram of 160 pestcides in full scan mode

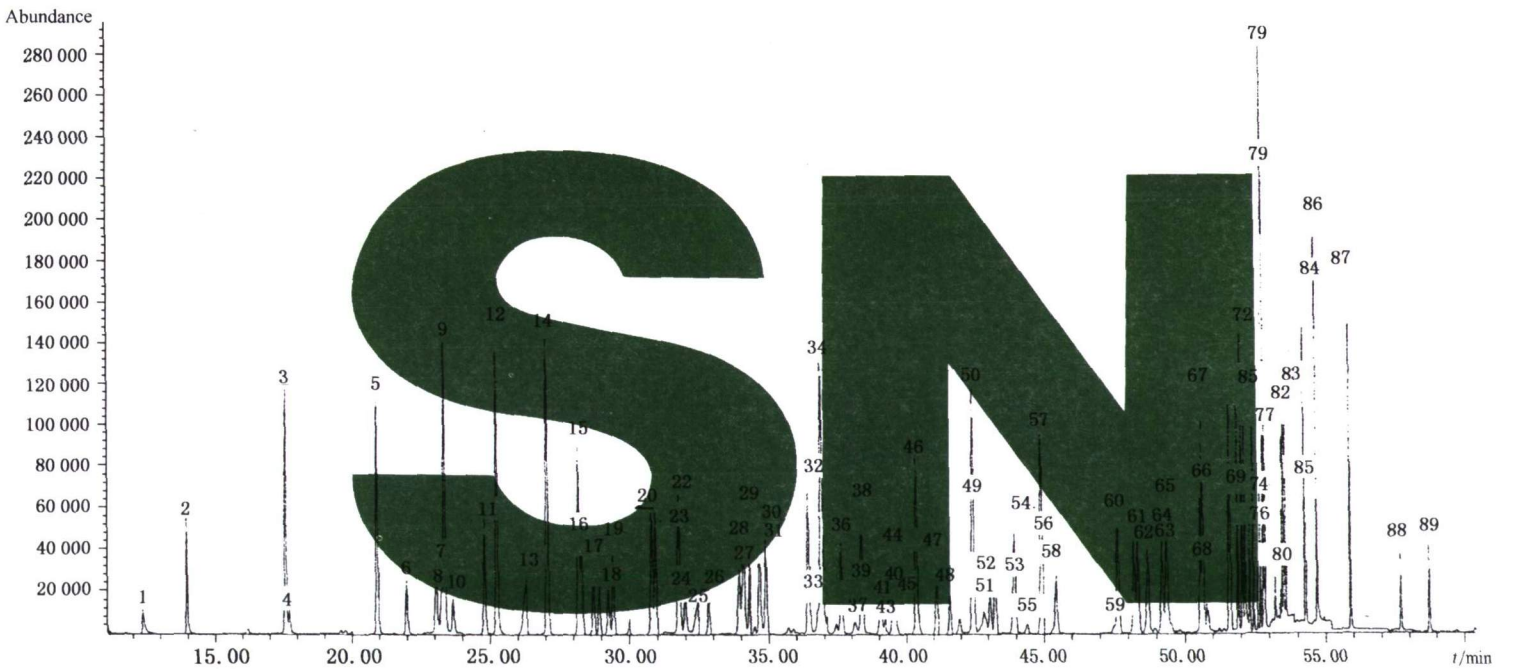


Figure C. 1—Chromatogram of pestcides under method 1

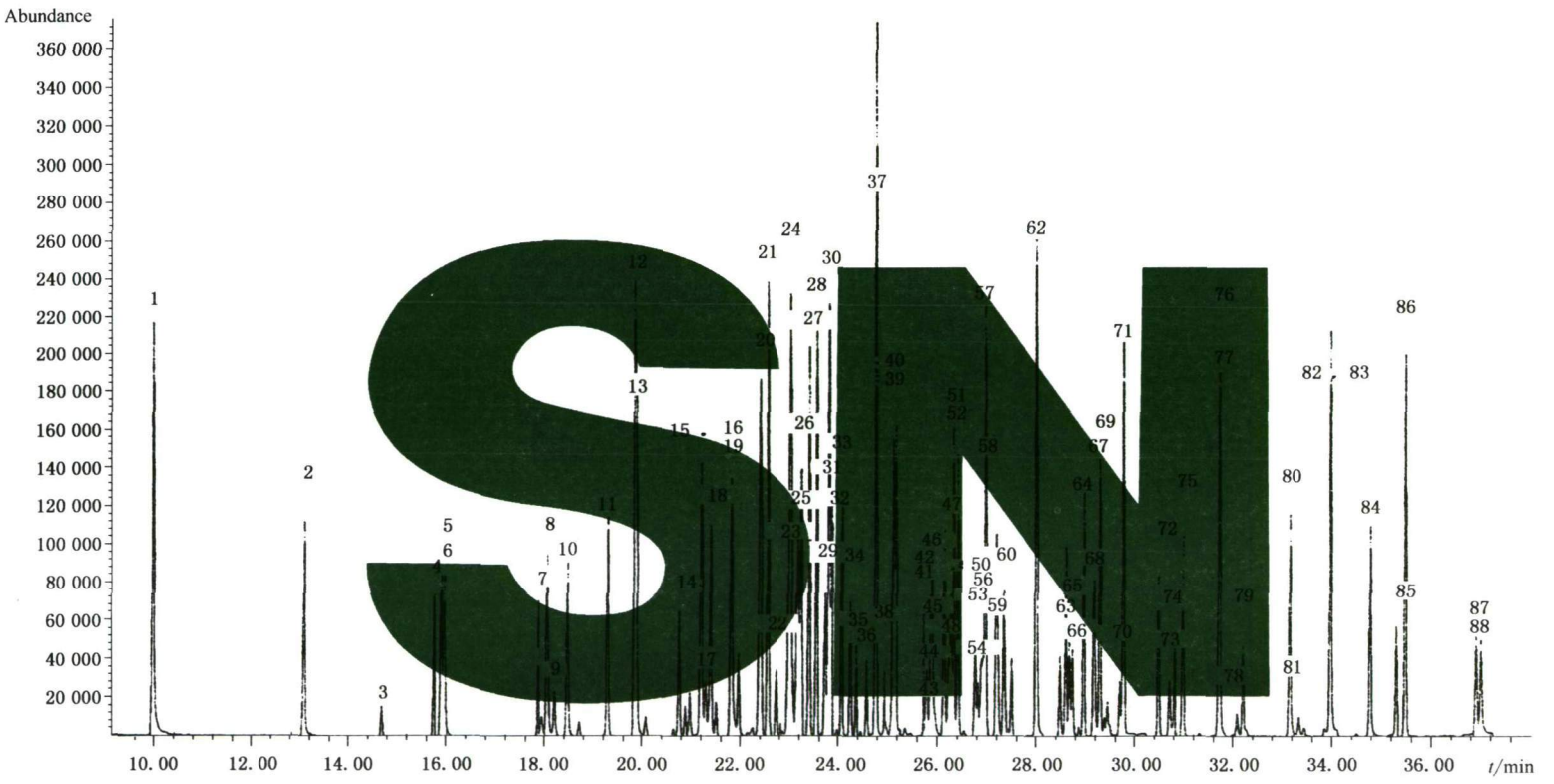


Figure C. 2—Chromatogram of pestcides under method 2

Annex D
(Informative)
Recovery range

Table D.1—Recovery range of 160 pestcides

No.	Compound name	Range of recovery %				
		Rice	Brown rice	Wheat	Barley	Corn
1	1,1-dichloro-2,2-bis(4-ethylphenyl)ethane	71.0～94.2	70.9～106.1	79.2～103.4	89.0～107.2	73.0～93.5
2	2-(1-naphthyl)acetamide	74.3～97.4	83.0～106.6	74.3～97.8	71.4～95.3	73.6～100.2
3	Acetochlor	83.2～107.9	82.2～107.1	86.7～109.4	87.0～94.9	85.1～93.2
4	Acibenzoral-s-methyl	76.0～108.4	72.6～102.5	81.2～94.6	81.9～93.6	79.0～106.6
5	Alachlor	69.2～96.9	76.7～101.0	77.4～102.9	82.0～101.6	78.1～103.7
6	Azamethiphos	85.4～104.0	84.3～109.1	81.9～106.3	72.0～95.5	71.6～96.2
7	Barban	77.7～98.2	76.1～96.3	84.4～99.7	72.4～97.9	85.3～96.4
8	Benalaxyl	73.9～100.8	87.2～94.9	88.3～103.1	84.1～100.8	87.6～107.7
9	Benfuracarb	76.0～108.0	79.9～98.3	87.6～93.3	77.0～108.5	75.6～105.3
10	Bifenox	73.6～100.2	86.3～93.8	80.0～109.0	70.8～96.2	75.6～100.0
11	Biphenyl	72.5～106.3	80.8～99.1	82.1～102.3	87.1～108.0	72.5～95.5
12	Bitertanol	78.1～97.7	86.2～106.0	80.9～101.2	75.5～102.8	86.1～94.2
13	Boscalid	72.2～94.5	85.0～105.2	73.0～109.6	87.1～107.8	78.8～96.7
14	Bromacil	83.1～93.8	77.0～101.1	83.7～95.7	88.5～102.3	75.4～105.5
15	Bromophos	86.0～107.0	77.1～102.6	85.5～104.4	69.6～106.5	75.1～105.8
16	Bromopropylate	71.8～106.4	72.1～103.2	81.6～93.2	81.8～102.6	81.2～100.1
17	Butachlor	88.2～107.5	79.0～100.0	75.1～99.2	76.1～108.2	76.9～93.6
18	Cafenstrole	69.2～108.9	72.4～108.8	75.3～99.5	76.4～95.9	77.4～108.1
19	Carbetamide	84.1～105.1	70.5～106.2	70.1～100.5	69.5～94.1	71.0～105.0
20	Carfentrazone-ethyl	77.1～95.7	81.0～100.7	71.5～107.4	76.6～96.4	74.4～108.1
21	Chlorbufam	78.5～96.7	70.8～102.4	84.9～105.5	69.7～108.4	84.0～104.2
22	Chlordane	88.5～101.3	74.6～103.1	71.3～97.8	78.3～100.2	70.1～96.6
23	Chlorethoxyfos	86.8～93.2	76.1～98.3	71.2～104.6	75.7～102.6	71.6～93.3
24	Chlorobenzilate	75.0～93.6	71.2～101.8	77.8～106.8	79.8～96.6	79.5～104.5
25	Chloroneb	83.1～101.4	71.5～107.1	69.1～100.5	71.5～101.3	80.0～96.8
26	Chlorpropham	78.2～98.7	88.5～103.0	73.7～107.9	88.3～109.3	77.1～99.7
27	Chlozolinate	80.3～107.6	85.9～106.5	70.1～104.4	77.7～101.0	75.4～99.3

Table D. 1 (continued)

No.	Compound name	Range of recovery %				
		Rice	Brown rice	Wheat	Barley	Corn
28	Cinidon-ethyl	86.5～100.8	80.0～107.2	74.1～96.7	78.7～105.4	77.7～103.7
29	Cinmethylin	77.6～93.6	76.0～102.5	84.5～97.8	73.7～102.0	78.1～102.7
30	Clomazone	74.0～108.0	88.1～108.8	86.8～97.8	74.2～94.3	77.0～103.9
31	Cyanazine	77.3～95.5	70.4～105.3	88.2～97.7	85.2～97.2	84.1～105.5
32	Cyanophos	88.2～95.5	74.0～104.9	71.3～94.7	75.2～108.6	81.1～108.4
33	Cyhalofop-butyl	74.9～106.0	84.0～107.4	79.3～106.0	75.1～102.7	83.4～96.0
34	Cyproconazole	87.9～95.5	70.4～105.1	73.9～101.5	79.9～93.6	70.5～93.7
35	Cyprodinil	72.1～103.8	80.5～99.3	85.2～100.4	88.1～96.6	86.3～104.5
36	Demeton-s-methyl	82.8～95.4	82.5～94.6	77.2～95.3	86.6～109.3	76.9～100.0
37	Dichlofenthion	70.9～93.7	79.6～94.4	83.6～102.9	88.6～106.0	69.3～100.6
38	Dicloran	74.9～109.9	85.1～98.3	85.2～99.7	81.7～94.0	82.2～104.5
39	Diclocymet	75.8～106.4	77.7～106.5	70.2～106.8	72.7～93.5	71.6～97.4
40	Dicofol	74.6～105.1	88.9～95.8	79.3～106.6	87.9～96.3	75.9～94.8
41	Diethofencarb	81.4～101.8	85.9～100.5	74.9～98.7	72.8～107.0	71.2～96.9
42	Difenoconazole	71.1～109.3	76.9～102.1	69.7～95.8	88.4～103.7	78.6～108.3
43	Diflufenican	85.7～102.8	77.5～108.7	87.6～97.2	81.6～109.8	79.2～98.4
44	Dimethametryn	84.2～104.8	86.1～94.0	72.7～102.3	70.5～102.4	77.0～103.2
45	Dimethenamid	79.3～97.3	69.9～107.5	86.2～99.1	83.6～104.3	75.1～93.5
46	Dimethipin	87.2～106.5	78.6～93.1	78.6～102.0	77.1～108.9	87.5～93.4
47	Diniconazole	78.0～108.5	80.1～97.7	71.0～107.7	75.8～109.5	73.6～96.5
48	Disulfoton sulfone	86.8～93.6	71.7～105.0	73.6～100.0	69.8～102.5	70.6～105.1
49	Dithiopyr	85.7～103.7	79.5～94.4	88.6～102.0	71.9～99.3	76.2～104.5
50	Endosulfan	71.3～106.1	76.8～106.4	82.0～109.6	83.6～109.1	72.1～97.0
51	Ethion	75.5～100.4	73.2～108.8	87.5～99.9	75.9～96.8	78.0～100.4
52	Etobenzanid	83.3～100.0	81.9～102.2	85.0～105.5	73.6～99.3	70.0～93.1
53	Famoxadone	85.6～97.2	73.3～103.1	78.2～96.5	85.3～100.7	69.7～98.1
54	Fenarimol	87.3～95.6	70.5～107.4	87.2～107.2	83.8～104.1	78.4～98.6
55	Fenbuconazole	82.0～94.7	83.0～109.6	73.7～106.2	75.5～105.7	72.4～107.5
56	Fenoxanil	75.7～101.2	72.0～108.6	85.7～109.3	87.3～93.9	71.9～94.3
57	Fenoxycarb	81.6～101.2	75.7～94.6	72.2～102.8	73.4～95.4	74.8～98.8
58	Fenpropathrin	82.8～103.1	72.9～106.5	70.4～94.0	80.3～97.7	75.3～103.3
59	Fenson	70.6～94.5	81.4～93.6	78.7～106.4	71.5～108.7	74.1～95.9
60	Fipronil	82.4～108.4	81.1～108.9	73.4～97.9	75.3～105.9	76.3～94.6

Table D. 1 (continued)

No.	Compound name	Range of recovery %				
		Rice	Brown rice	Wheat	Barley	Corn
61	Flamprop-methyl	84.1～96.0	77.7～108.4	74.1～100.5	80.2～109.3	77.0～107.8
62	Fludioxonil	70.3～102.0	79.1～104.8	80.2～98.9	84.6～95.2	69.5～105.3
63	Flufenpyr-ethyl	88.8～96.1	75.4～108.0	81.6～95.6	76.3～107.4	82.5～98.5
64	Fluquinconazole	84.6～110.0	81.0～108.1	78.6～106.0	77.8～96.6	87.4～106.5
65	Flusilazole	82.9～96.6	85.9～97.1	77.3～98.8	88.4～102.0	85.1～98.5
66	Flutolanil	77.5～96.1	74.5～105.5	82.7～98.5	69.1～95.1	88.6～103.9
67	Furametpry	77.1～106.1	76.7～102.5	89.0～105.2	87.8～98.7	75.8～96.7
68	Hexaconazole	81.1～105.2	78.1～107.4	74.9～95.1	74.0～99.6	78.9～94.2
69	Hexazinone	70.8～97.4	81.6～97.1	79.2～93.5	79.5～99.3	69.8～98.1
70	Hexythiazox	72.1～108.1	80.5～105.1	75.9～108.7	87.4～99.0	70.3～105.5
71	Imazalil	73.8～94.9	72.5～100.9	82.7～103.8	82.7～108.0	72.7～99.6
72	Imibenconazole	76.2～101.9	87.8～106.5	71.5～105.9	87.7～109.0	72.7～101.2
73	Indanofan	72.2～108.7	82.3～101.1	85.0～99.9	72.8～101.1	83.3～99.4
74	Ioxynil-octanoate	86.8～99.4	87.1～100.1	81.3～98.4	75.3～106.9	86.4～102.4
75	Ipconazole	78.3～106.1	80.4～94.4	70.2～101.8	72.9～100.7	72.5～95.5
76	Iprobenfos	87.3～109.8	84.8～95.9	82.8～93.1	84.1～93.8	82.1～103.5
77	Iprovalicarb	83.1～102.6	72.8～94.9	78.5～105.1	82.5～99.3	75.4～104.3
78	Isocarbophos	69.7～103.7	76.6～94.3	75.6～102.6	82.6～104.4	81.1～95.4
79	Isofenphos	86.3～105.1	77.9～106.8	78.1～109.0	85.7～97.4	87.8～103.3
80	Isofenphos-oxon	81.0～93.5	77.3～103.3	73.4～96.8	79.5～110.0	75.0～98.0
81	Isopropalin	87.2～96.5	85.4～94.0	86.5～101.9	75.0～106.5	83.7～106.4
82	Isoxathion	72.8～102.2	73.3～106.3	82.1～104.0	77.4～93.2	88.5～97.9
83	Kresoxim-methyl	71.7～95.2	82.5～100.5	88.0～108.6	76.4～101.5	82.1～97.8
84	Lenacil	88.3～109.7	82.6～100.6	71.4～94.5	74.5～98.1	81.8～93.6
85	Lindan	86.4～105.1	73.0～93.9	80.5～96.5	83.1～100.1	70.9～100.5
86	Linuron	73.6～104.1	77.5～93.2	87.3～109.6	84.3～97.0	88.8～96.9
87	Mecarbam	69.8～108.3	79.6～103.3	77.1～98.8	74.0～106.4	74.8～105.4
88	Mefenacet	74.5～104.0	77.1～100.7	83.7～106.7	80.7～102.4	74.7～109.9
89	Mepanipyrim	79.3～105.4	69.5～98.9	82.2～99.5	85.3～96.1	83.3～103.0
90	Mepronil	83.4～98.1	75.9～101.0	85.9～108.5	82.6～94.4	78.7～94.9
91	Metalaxyl	88.5～103.6	88.9～108.7	78.8～96.3	75.2～102.7	69.4～94.9
92	Methidathion	87.2～93.9	88.2～95.8	71.7～103.7	84.1～104.7	72.5～106.8
93	Methoprene	70.5～95.5	75.7～100.8	70.6～94.4	71.5～101.7	84.9～104.4

Table D. 1 (continued)

No.	Compound name	Range of recovery %				
		Rice	Brown rice	Wheat	Barley	Corn
94	Methoxychlor	74.2～96.8	80.9～100.1	71.7～105.6	84.9～109.4	75.1～97.5
95	Metolachlor	77.4～104.5	77.1～108.5	86.5～98.1	76.1～107.1	82.9～98.1
96	Metribuzin	74.5～108.3	79.5～98.6	79.6～93.0	86.0～100.5	81.8～109.4
97	MGK264	79.9～100.1	83.0～100.5	69.2～105.3	83.8～101.8	75.0～94.7
98	Molinate	76.9～98.3	84.3～107.4	89.4～94.2	70.9～101.1	77.9～99.6
99	Monolinuron	71.1～105.6	86.1～100.9	76.3～109.3	88.6～106.4	84.6～99.9
100	Myclobutanil	74.1～96.0	81.4～102.4	78.4～99.7	71.2～108.4	77.8～96.0
101	Nitrothal-isopropyl	79.7～102.6	69.1～107.5	87.5～103.8	79.0～98.3	70.9～97.9
102	Nonachlor	75.5～105.5	78.9～93.5	74.7～107.9	83.9～103.3	71.0～101.9
103	Oxabetrinil	76.9～96.3	70.2～97.6	87.9～101.1	74.2～96.2	69.3～94.7
104	Oxadiazon	78.3～99.6	82.1～108.5	74.4～109.3	80.4～107.7	78.9～108.4
105	Oxadixyl	83.3～108.5	72.8～109.8	74.0～107.9	78.3～105.4	72.3～108.9
106	Oxycarboxin	73.3～104.6	86.8～101.9	79.7～103.7	69.6～106.4	69.9～93.7
107	Paclobutrazol	87.0～94.8	76.6～93.7	80.6～94.3	80.5～106.2	69.0～94.7
108	Penconazole	83.2～105.0	85.4～100.8	82.6～99.2	81.8～97.0	72.6～108.9
109	Pendimethalin	83.4～104.2	86.6～106.1	88.9～97.8	81.2～101.1	79.4～94.1
110	Phenothrin	78.2～107.4	88.3～93.7	69.9～105.4	83.7～99.0	77.5～106.4
111	Phosalone	87.9～106.4	75.2～107.3	70.6～104.1	87.0～101.2	84.6～98.1
112	Phosmet	86.4～103.2	73.6～105.5	75.2～96.0	78.4～99.9	81.5～100.3
113	Picolinafen	84.9～94.3	82.9～109.1	74.5～101.9	84.1～102.5	85.3～95.2
114	Piributicarb	82.5～108.7	78.8～93.8	73.4～103.8	86.2～107.5	79.5～106.6
115	Pirimicarb	71.8～96.8	87.9～106.6	80.9～107.7	70.9～98.4	72.1～93.0
116	Probenazole	79.6～98.0	71.8～101.1	74.2～108.7	79.3～99.5	75.2～104.8
117	Procymidone	79.2～96.8	72.1～101.9	75.5～99.6	85.5～103.6	80.2～109.1
118	Profenofos	71.5～98.3	86.7～97.3	78.5～100.6	85.1～95.3	80.8～99.1
119	Prohydrojasmon	76.4～96.9	80.1～106.9	82.2～94.3	84.2～97.6	74.2～97.3
120	Promecarb	78.7～99.4	82.1～96.3	88.7～96.9	88.9～103.6	74.8～101.3
121	Prometryn	86.8～102.3	88.0～94.4	84.2～94.0	77.1～106.1	86.5～95.2
122	Propargite	69.1～103.6	70.2～102.0	82.5～105.8	85.5～103.1	78.2～96.1
123	Propiconazole	76.6～103.2	77.2～96.8	83.5～107.8	88.3～98.0	80.0～104.1
124	Propoxur	82.8～98.0	77.7～98.9	82.2～98.8	80.2～107.2	87.1～103.0
125	Propyzamide	69.6～107.3	71.6～104.2	82.0～102.0	77.3～99.3	77.5～98.9
126	Prothiofos	87.0～97.6	73.6～104.7	77.3～100.9	70.1～95.5	69.3～95.5

Table D. 1 (continued)

No.	Compound name	Range of recovery %				
		Rice	Brown rice	Wheat	Barley	Corn
127	Pyraflufen-ethyl	86.6～103.7	72.2～106.3	79.4～95.3	80.8～96.1	75.6～96.2
128	Pyridaben	80.2～103.6	84.5～94.9	81.3～96.5	86.8～98.8	87.6～100.1
129	Pyridafenthion	76.0～93.2	82.9～100.0	86.5～100.8	74.5～99.3	84.4～96.0
130	Pyridalyl	73.5～102.4	79.8～99.5	83.8～100.5	88.8～99.6	86.5～98.4
131	Pyrifenox	86.4～93.8	70.1～97.4	70.3～108.7	72.5～97.3	72.9～102.0
132	Pyriftalid	78.8～103.0	77.5～103.3	81.0～106.3	87.4～106.3	78.9～103.0
133	Pyrimethanil	70.8～94.0	72.6～93.8	83.5～109.8	83.4～101.8	87.5～99.5
134	Pyrimidifen	79.0～94.4	77.4～105.5	81.8～96.1	80.4～100.6	88.1～109.4
135	Pyriminobac-methyl	73.0～104.0	70.9～101.2	70.2～105.7	74.2～94.1	75.6～103.6
136	Quinoxyfen	82.3～107.7	87.1～96.3	71.1～93.0	82.4～96.9	70.6～105.9
137	Salithion	70.9～107.2	70.8～98.6	69.4～99.4	84.7～103.7	86.7～97.6
138	Silafluofen	82.5～107.8	87.5～100.9	86.7～94.1	85.3～107.3	81.6～100.9
139	Simazine	76.4～94.9	72.8～93.1	76.2～105.1	73.9～94.3	87.3～96.7
140	Simetryn	86.6～101.1	75.5～106.4	69.3～101.7	85.2～107.0	81.0～105.3
141	Spiromesifen	73.5～101.7	71.5～108.9	84.1～100.6	76.9～107.3	73.1～93.8
142	Tebuconazole	72.6～104.8	75.2～95.1	81.3～93.5	77.7～103.7	84.8～95.8
143	Tebufenpyrad	81.5～103.8	86.3～103.4	83.5～107.7	76.9～93.1	73.0～108.7
144	Terbacil	83.0～107.3	75.0～100.5	84.2～108.6	82.6～99.1	75.6～102.6
145	Terbutryn	80.9～97.3	83.7～93.9	84.1～108.6	76.7～94.2	77.2～105.8
146	Tetrachlorvinphos	76.9～103.1	80.7～98.2	81.8～109.6	71.4～98.7	75.9～98.7
147	Tetraconazole	72.3～99.4	70.9～109.3	72.9～101.2	79.6～101.1	81.8～109.4
148	Tetradifon	74.8～104.3	76.5～104.5	83.5～97.0	83.8～106.2	84.1～93.6
149	Thenylchlor	74.5～100.6	70.9～109.7	87.6～106.1	80.0～104.4	81.5～101.2
150	Thiazopyr	81.0～108.5	70.8～99.6	87.4～96.6	84.0～93.9	81.5～108.1
151	Thifluzamide	82.2～97.6	85.0～94.4	71.7～94.4	80.1～104.0	76.0～93.4
152	Thiobencarb	87.3～109.8	77.7～109.2	80.2～93.6	88.0～105.4	85.6～96.6
153	Tralkoxydim	70.9～106.4	82.5～93.0	73.4～95.2	77.4～95.3	75.0～96.8
154	Tricyclazole	83.7～97.3	87.5～96.3	81.0～98.7	86.0～98.2	88.8～95.1
155	Trifloxystrobin	82.2～98.0	79.0～99.8	85.4～107.1	69.9～99.5	71.6～109.8

Table D. 1 (continued)

No.	Compound name	Range of recovery %				
		Rice	Brown rice	Wheat	Barley	Corn
156	Triflumizole	85.4～100.3	77.8～104.1	71.8～99.2	70.5～99.1	74.5～93.3
157	Triflumuron	69.6～93.9	88.1～106.0	82.0～103.1	72.2～101.9	75.4～104.7
158	Trifluralin	73.4～103.9	72.5～101.8	73.5～105.8	88.5～97.3	71.5～104.2
159	Uniconazole p	87.6～95.0	69.7～93.8	73.8～94.8	79.1～110.0	88.5～106.4
160	Vinclozoline	83.1～95.8	86.7～99.5	76.8～94.2	82.3～93.1	84.0～100.8