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中华人民共和国出入境检验检疫行业标准

SN/T 2149—2008

进出口食品中解草嗪、莎稗磷、二丙烯草胺等110种农药残留量的检测方法 气相色谱-质谱法

Determination of benoxacor, anilofos, allidochlor 110 pesticides residues in foodstuffs for import and export— GC-MS Method

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中华人民共和国
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前　言

本标准的附录A和附录E为规范性附录，附录B、附录C、附录D为资料性附录。

本标准由国家认证认可监督管理委员会提出并归口。

本标准起草单位：中国检验检疫科学研究院、中华人民共和国黑龙江出入境检验检疫局、中华人民共和国内蒙古出入境检验检疫局。

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本标准系首次发布的出入境检验检疫行业标准。

进出口食品中解草嗪、莎稗磷、二丙烯草胺等110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110种农药残留量检测的气相色谱-质谱检测方法。

本标准适用于大米、糙米、大麦、小麦、玉米中110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，然后依次通过液-液分配、凝胶渗透色谱和固相萃取对提取液进行净化，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，所用试剂均为分析纯，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 乙腈。

3.7 甲苯。

3.8 氯化钠。

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 氯化钠溶液：15%(质量浓度)，称取15 g氯化钠，溶于100 mL水中，摇匀备用。

3.11 环己烷-乙酸乙酯(1+1，体积比)。

3.12 乙腈-甲苯(3+1，体积比)。

3.13 农药标准物质：标准物质清单见附录A，标准物质纯度≥95%。

3.14 农药标准溶液

3.14.1 标准储备溶液

准确称取适量标准品(精确至0.1 mg)，用丙酮(3.1)溶解，配制成浓度为500 μg/mL的标准储备溶液，−18 ℃冷冻避光保存，有效期3个月。

3.14.2 混合标准中间溶液

将110种农药分成五个组，分组情况见附录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，4 ℃冷藏避光保存，有效期1个月。

3.14.3 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。

3.15 助滤剂：celite 545，或相当者。

3.16 以石墨化非多孔碳/酰胺丙基甲硅烷基化硅胶为填料的固相萃取柱：Envi-Carb/LC-NH_2，

500 mg/500 mg,6 mL,或相当者。

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 固相萃取装置。
4.4 样品粉碎机:配20目样品筛。
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 气相色谱-质谱仪:配有电子轰击电离源(EI)。
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)]。

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20目的样品筛。混和均匀,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。向抽滤装置内加入适量助滤剂,将试样及提取液转移至抽滤装置上,减压抽滤,再用3×5 mL丙酮(3.2)洗涤锥形瓶及试样残渣,合并提取液和洗涤液,于40 ℃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 15%(质量浓度)氯化钠水溶液,50 mL二氯甲烷,振荡5 min,静置后收集二氯甲烷层并过无水硫酸钠脱水。再加入50 mL二氯甲烷重复上述操作。合并经无水硫酸钠脱水的二氯甲烷,于40 ℃水浴旋转浓缩至近干,再用氮气吹干。用环己烷-乙酸乙酯定容至4.0 mL,待净化。

6.2 净化

6.2.1 凝胶渗透色谱净化

将6.1中待净化溶液置于凝胶色谱仪上,进样体积为2.0 mL(相当于一半称样量的样品),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乙腈-甲苯溶液溶解残渣。

6.2.2 固相萃取净化

将Envi-Carb/LC-NH_2固相萃取柱置于固相萃取装置上。用10 mL乙腈-甲苯溶液预淋洗。将经6.2.1净化的溶液移入固相萃取柱同时即开始收集淋洗液,用3×1 mL乙腈-甲苯溶液洗涤容器并移入固相萃取柱,并调节装置使淋洗液流速约为2 mL/min。再用20 mL乙腈-甲苯溶液洗涤固相萃取柱,合并淋洗液。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

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

b) 色谱柱温度：60 ℃保持 2 min，先以 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 10 min；

c) 载气：氦气，纯度≥99.999%，流速 1.0 mL/min；

d) 进样口温度：250 ℃；

e) 进样量：2 μL；

f) 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；

g) 电子轰击电离源：70 eV；

h) 离子源温度：230 ℃；

i) 四极杆温度：150 ℃；

j) GC-MS 接口温度：280 ℃；

k) 溶剂延迟时间：7 min；

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

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

为减少基质对定量结果的影响，本方法采用空白样品溶液配置基质混合标准工作溶液，外标法定量测定。样品中农药响应值应在基质混合标准工作溶液线性范围内。本方法标准物质在各类粮谷基质中选择离子监测 GC-MS 质量色谱图参见附录 D。

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-MSD 数据处理软件计算，或按照式(1)计算，(计算结果需将空白值扣除。)

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

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——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——最终溶液相当的试样质量，单位为克(g)。

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110种农药残留进行测定，各种农药的测定低限均为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，110种农药在5种食品中的添加回收率见附录E。

附　录　A
（规范性附录）
农药标准物质基本信息

表 A.1　农药标准物质基本信息

序号	中文通用名	英文通用名	CAS 编码	混合标准溶液分组
1	*烯丙菊酯*	Allethrin	584-79-2	A
2	二丙烯草胺	Allidochlor	93-71-0	A
3	莠灭净	Ametryn	834-12-8	A
4	莎稗磷	Anilofos	64249-01-0	A
5	莠去津	Atrazine	1912-24-9	A
6	氧环唑	Azaconazole	60207-31-0	A
7	保棉磷	Azinphos-methyl	86-50-0	A
8	乙丁氟灵	Benfluralin	1861-40-1	A
9	解草嗪	Benoxacor	98730-04-2	A
10	烟酰胺	Boscalid	188425-85-6	A
11	乙基溴硫磷	Bromophos-ethyl	4824-78-6	A
12	乙嘧酚磺酸酯	Bupirimate	41483-43-6	A
13	噻嗪酮	Buprofezin	69327-76-0	A
14	英拜除草剂	Butafenacil	134605-64-4	A
15	硫线磷	Cadusafos	95465-99-9	A
16	丁硫克百威	Carbosulfan	55285-14-8	A
17	萎锈灵	Carboxin	5234-68-4	A
18	杀螨醚	Chlorbenside	103-17-3	A
19	氯氧磷	Chlorethoxyfos	54593-83-8	A
20	虫螨腈	Chlorfenapyr	122453-73-0	A
21	杀螨酯	Chlorfenson	80-33-1	B
22	氯草敏	Chloridazon	1698-60-8	B
23	地茂散	Chlorneb	2675-77-6	B
24	氯酞酸甲酯	Chlorthal-dimethyl	1861-32-1	B
25	炔草酸	Clodinafop-propargyl	105512-06-9	B
26	氯甲酰草胺	clomeprop	84496-56-0	B
27	解草酯	Cloquintocet-mexyl	99607-70-2	B
28	杀螟腈	Cyanophos	2636-26-2	B
29	环草敌	Cycloate	1134-23-2	B
30	环氟菌胺	Cyflufenamid	180409-60-3	B

表 A.1（续）

序号	中文通用名	英文通用名	CAS 编码	混合标准溶液分组
31	燕麦敌	Diallate	2303-16-4	B
32	禾草灵	Diclofop-methyl	51338-27-3	B
33	百治磷	Dicrotophos	141-66-2	B
34	哌草丹	Dimepiperate	61432-55-1	B
35	乐果	Dimethoate	60-51-5	B
36	烯酰吗啉	Dimethomorph	110488-70-5	B
37	苯虫醚	Diofenolan	63837-33-2	B
38	敌恶磷	Dioxathion	78-34-2	B
39	双苯酰草胺	Diphenamid	957-51-7	B
40	乙拌磷	Disulfoton	298-04-4	B
41	硫丹硫酸盐	Endosulfan-sulfate	1031-07-8	C
42	氟环唑	Epoxiconazole	106325-08-0	C
43	S-氰戊菊酯	Esfenvalerate	66230-04-4	C
44	乙丁烯氟灵	Ethalfluralin	55283-68-6	C
45	乙硫磷	Ethion	563-12-2	C
46	乙氧呋草黄	Ethofumesate	26225-79-6	C
47	乙螨唑	Etoxazole	153233-91-1	C
48	咪唑菌酮	Fenamidone	161326-34-7	C
49	苯线磷	Fenamiphos	22224-92-6	C
50	苯线磷砜	Fenamiphos-sulfone	31972-44-8	C
51	腈苯唑	Fenbuconazole	114369-43-6	C
52	皮蝇磷	Fenchlorphos	299-84-3	C
53	嘧螨酯	Fluacrypyrim	229977-93-9	C
54	吡氟禾草灵	Fluazifop-butyl	69806-50-4	C
55	氟噻草胺	Flufenacet	142459-58-3	C
56	氟烯草酸	Flumiclorac pentyl	87546-18-7	C
57	丙炔氟草胺	Flumioxazin	103361-09-7	C
58	哒草氟/氟噻甲草酯	Fluthiacet-methyl	117337-19-6	C
59	粉唑醇	Flutriafol	76674-21-0	C
60	噻唑磷	Fosthiazate	98886-44-3	C
61	解草恶唑	Furilazole	121776-33-8	C
62	苄螨醚	Halfenprox	111872-58-3	C
63	氟吡禾灵	Haloxyfop-methyl	69806-40-2	C
64	七氯	Heptachlor	76-44-8	C

表 A.1（续）

序号	中文通用名	英文通用名	CAS 编码	混合标准溶液分组
65	六氯苯	Hexachlorobenzene	118-74-1	C
66	抑霉唑	Imazalil	35554-44-0	D
67	茚草酮	Indanofan	133220-30-1	D
68	茚虫威	Indoxacarb	144171-61-9	D
69	氯唑磷	Isazofos	42509-80-8	D
70	异丙威	isoprocarb	2631-40-5	D
71	稻瘟灵	Isoprothiolane	50512-35-1	D
72	双苯恶唑酸	Isoxadifen-ethyl	163520-33-0	D
73	乳氟禾草灵	Lactofen	77501-63-4	D
74	吡咯二酸二乙酯	Mefenpyr-diethyl	135590-91-9	D
75	嘧菌胺	Mepanipyrim	110235-47-7	D
76	甲霜灵	Metalaxyl	57837-19-1	D
77	虫螨畏	Methacrifos	62610-77-9	D
78	异丙甲草胺	metolachlor	51218-45-2	D
79	*速灭磷*	Mevinphos	26718-65-0	D
80	敌草胺	Napropamide	15299-99-7	D
81	三氯甲基吡啶	Nitrapyrin	1929-82-4	D
82	氟草敏	Norflurazon	27314-13-2	D
83	乙氧氟草醚	Oxyfluorfen	42874-03-3	D
84	二甲戊灵	pendimethalin	40487-42-1	D
85	*苯醚菊酯*	Phenothrin	26002-80-2	D
86	甲拌磷	Phorate	298-02-2	D
87	*磷胺*	Phosphamidon	13171-21-6	D
88	哌草磷	Piperophos	24151-93-7	D
89	茉莉酸诱导体	Prohydrojasmon	158474-72-7	D
90	毒草胺	Propachlor	1918-16-7	D
91	敌稗	Propanil	709-98-8	D
92	丙虫磷	Propaphos	7292-16-2	E
93	苯胺灵	Propham	122-42-9	E
94	吡菌磷	Pyrazophos	13457-18-6	E
95	吡丙醚	Pyriproxyfen	95737-68-1	E
96	咯喹酮	Pyroquilon	57369-32-1	E
97	五氯硝基苯	Quintozene	82-68-8	E
98	精喹禾灵	Quizalofop-ethyl	76578-14-8	E

表 A.1（续）

序号	中文通用名	英文通用名	CAS 编码	混合标准溶液分组
99	硅氟唑	Simeconazole	149508-90-7	E
100	硫丙磷	Sulprofos	35400-43-2	E
101	丁噻隆	Tebuthiuron	34014-18-1	E
102	四氯硝基苯	Tecnazene	117-18-0	E
103	唑虫酰胺	Tolfenpyrad	129558-76-5	E
104	三唑酮	Triadimefon	43121-43-3	E
105	*三唑醇*	Triadimenol	55219-65-3	E
106	野麦畏	Triallate	2303-17-5	E
107	脱叶磷	Tribuphos	78-48-8	E
108	三环唑	Tricyclazole	41814-78-2	E
109	灭除威	XMC	2655-14-3	E
110	*苯酰菌胺*	Zoxamide	156052-68-5	E
注：第二栏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/MSD 测定的质谱条件

表 B.1 GC/MSD 测定的质谱条件

序号	中文通用名	英文通用名	保留时间/min	监测离子			
1	二丙烯草胺	Allidochlor	8.176	*132*	124	138	173
2	速灭磷	trans-Mevinphos	10.422	*192*	109	127	164
3	速灭磷	cis-Mevinphos	10.490	*192*	109	127	164
4	三氯甲基吡啶	Nitrapyrin	10.956	*196*	194	198	—
5	苯胺灵	Propham	11.176	*179*	93	137	—
6	虫螨畏	Methacrifos	11.968	*208*	125	180	—
7	地茂散	Chlorneb	12.153	*191*	206	208	—
8	丁噻隆	Tebuthiuron	12.480	*156*	157	171	—
9	异丙威	isoprocarb	12.790	*121*	91	136	—
10	灭除威	XMC	13.344	*122*	107	179	—
11	四氯硝基苯	Tecnazene	13.966	*261*	203	215	—
12	毒草胺	Propachlor	14.363	*120*	176	211	—
13	氯氧磷	Chlorethoxyfos	14.582	*299*	153	301	—
14	环草敌	Cycloate	15.089	*154*	83	215	—
15	乙丁烯氟灵	Ethalfluralin	15.372	*276*	316	292	—
16	百治磷	Dicrotophos	15.688	*127*	193	237	—
17	乙丁氟灵	Benfluralin	15.897	*292*	264	276	—
18	硫线磷	Cadusafos	16.236	*159*	214	270	—
19	燕麦敌	Diallate 1	16.390	*234*	128	236	—
20	甲拌磷	Phorate	16.398	*260*	75	231	—
21	六氯苯	Hexachlorobenzene	16.598	*284*	282	286	—
22	燕麦敌	Diallate 2	16.782	*234*	128	236	—
23	乐果	Dimethoate	17.125	*87*	125	229	—
24	解草恶唑	Furilazole	17.439	*220*	222	262	—
25	五氯硝基苯	Quintozene	17.740	*295*	237	249	—
26	莠去津	Atrazine	17.753	*200*	173	215	—
27	敌恶磷	Dioxathion	18.081	*270*	197	153	—
28	杀螟腈	Cyanophos	18.308	*243*	109	125	—
29	咯喹酮	Pyroquilon	18.462	*173*	130	144	—
30	磷胺	Phosphamidon 1	18.646	*127*	138	264	—

表 B.1（续）

序号	中文通用名	英文通用名	保留时间/min	监测离子			
31	乙拌磷	Disulfoton	19.062	*274*	88	142	—
32	茉莉酸诱导体	Prohydrojasmon	19.164	*83*	153	184	—
33	氯唑磷	Isazophos	19.181	*257*	161	285	—
34	野麦畏	Tri-allate	19.392	*268*	270	143	—
35	解草嗪	Benoxacor	19.770	*120*	259	261	—
36	磷胺	Phosphamidon 2	20.241	*127*	138	264	—
37	敌稗	Propanil	20.351	*163*	161	217	—
38	硅氟唑	Simeconazole	20.829	*121*	211	278	—
39	七氯	heptachlor	21.003	*272*	237	274	—
40	甲霜灵	Metalaxyl	21.148	*206*	220	234	—
41	莠灭净	Ametryn	21.179	*227*	185	212	—
42	皮蝇磷	Fenchlorphos	21.184	*285*	287	289	—
43	乙氧呋草黄	Ethofumesate	21.896	*207*	161	286	—
44	异丙甲草胺	metolachlor	22.268	*162*	238	240	—
45	氯酞酸甲酯	Chlorthal-dimethyl	22.506	*301*	299	332	—
46	氟噻草胺	Flufenacet	22.760	*151*	123	211	—
47	三唑酮	Triadimefon	22.772	*208*	181	210	—
48	双苯酰草胺	Diphenamid	23.191	*167*	165	239	—
49	噻唑磷	Fosthiazate 1	23.223	*195*	227	283	—
50	噻唑磷	Fosthiazate 2	23.313	*195*	227	283	—
51	二甲戊灵	pendimethalin	23.572	*252*	162	191	281
52	烯丙菊酯	Allethrin 1	23.991	*123*	107	136	—
53	烯丙菊酯	Allethrin 2	24.035	*123*	107	136	—
54	烯丙菊酯	Allethrin 3	24.185	*123*	107	136	—
55	烯丙菊酯	Allethrin 4	24.231	*123*	107	136	—
56	哌草丹	Dimepiperate	24.287	*119*	145	263	—
57	三唑醇	Triadimenol 1	24.297	*168*	112	128	—
58	苯酰菌胺	Zoxamide 1	24.378	*187*	242	—	—
59	三唑醇	Triadimenol 2	24.576	*168*	112	128	—
60	乙基溴硫磷	Bromophos-ethyl	24.649	*359*	303	357	—
61	杀螨醚	Chlorbenside	24.668	*125*	127	268	—
62	丙虫磷	Propaphos	24.823	*220*	262	304	—
63	氟吡禾灵	Haloxyfop-methyl	24.824	*316*	288	375	—
64	嘧菌胺	Mepanipyrim	25.256	*222*	221	223	—

表 B.1（续）

序号	中文通用名	英文通用名	保留时间/min	监测离子			
65	粉唑醇	Flutriafol	25.307	*123*	164	219	—
66	敌草胺	Napropamide	25.418	*271*	128	171	—
67	苯线磷	Fenamiphos	25.445	*303*	217	288	—
68	杀螨酯	Chlorfenson	25.540	*177*	302	304	—
69	抑霉唑	Imazalil	25.585	*215*	173	217	—
70	三环唑	Tricyclazole	25.591	*189*	161	162	—
71	稻瘟灵	Isoprothiolane	25.722	*204*	231	290	—
72	脱叶磷	Tribuphos	26.066	*169*	202	258	—
73	萎锈灵	Carboxin	26.194	*235*	143	—	—
74	噻嗪酮	Buprofezin	26.197	*105*	172	305	—
75	乙氧氟草醚	Oxyfluorfen	26.202	*252*	300	—	—
76	乙嘧酚磺酸酯	Bupirimate	26.209	*208*	273	—	—
77	氧环唑	Azaconazole	26.271	*217*	219	—	—
78	虫螨腈	Chlorfenapyr	26.497	*59*	247	408	—
79	环氟菌胺	Cyflufenamid	26.587	*118*	91	412	—
80	吡氟禾草灵	Fluazifop-butyl	26.866	*282*	254	383	—
81	乙硫磷	Ethion	27.361	*231*	153	384	—
82	嘧螨酯	Fluacrypyrim	27.695	*145*	189	204	—
83	硫丙磷	Sulprofos	27.866	*322*	156	280	—
84	双苯恶唑酸	Isoxadifen-ethyl	28.081	*204*	222	294	—
85	氟草敏	Norflurazon	28.210	*303*	145	173	—
86	硫丹硫酸盐	Endosulfan sulfate	28.334	*272*	387	422	—
87	苯虫醚	Diofenolan 1	28.386	*186*	225	300	—
88	氯草敏	Chloridazon	28.440	*221*	220	223	—
89	苯虫醚	Diofenolan 2	28.591	*186*	225	300	—
90	炔草酸	Clodinafop-propargyl	28.607	*266*	238	349	—
91	禾草灵	Diclofop-methyl	29.054	*253*	281	340	342
92	氟环唑	Epoxiconazole	29.409	*192*	165	194	—
93	苯酰菌胺	Zoxamide 2	29.442	*187*	189	258	—
94	吡咯二酸二乙酯	Mefenpyr-diethyl	29.529	*253*	255	299	—
95	苯线磷砜	Fenamiphos-sulfone	29.769	*320*	292	335	—
96	丁硫克百威	Carbosulfan	29.843	*160*	118	163	—
97	哌草磷	Piperophos	30.183	*122*	140	320	—
98	解草酯	Cloquintocet-mexyl	30.296	*192*	193	194	—

表 B.1（续）

序号	中文通用名	英文通用名	保留时间/min	监测离子			
99	乙螨唑	Etoxazole	30.372	*300*	330	359	—
100	咪唑菌酮	Fenamidone	30.426	*238*	268	—	—
101	茚草酮	Indanofan	30.540	*139*	174	310	—
102	莎稗磷	Anilofos	30.582	*226*	125	184	—
103	氯甲酰草胺	Clomeprop	30.762	*288*	120	148	—
104	苯醚菊酯	Phenothrin 1	30.887	*123*	183	350	—
105	苯醚菊酯	Phenothrin 2	31.085	*123*	183	350	—
106	保棉磷	Azinphos-methyl	31.232	*77*	132	160	—
107	吡丙醚	Pyriproxyfen	31.472	*136*	96	226	—
108	乳氟禾草灵	Lactofen	31.836	*344*	346	461	—
109	吡菌磷	Pyrazophos	32.054	*221*	232	373	—
110	英拜除草剂	Butafenacil	33.659	*331*	180	333	—
111	腈苯唑	Fenbuconazole	33.906	*129*	125	198	—
112	烟酰胺	Boscalid	34.583	*140*	342	344	—
113	苄螨醚	Halfenprox	34.662	*265*	183	263	—
114	精喹禾灵	Quizalofop-ethyl	34.822	*299*	243	372	—
115	丙炔氟草胺	Flumioxazin	35.932	*354*	259	287	—
116	S-氰戊菊酯	Esfenvalerate	36.349	*167*	225	419	—
117	茚虫威	Indoxacarb	37.018	*203*	150	218	—
118	氟烯草酸	Flumiclorac pentyl	37.451	*423*	308	318	—
119	烯酰吗啉	Dimethomorph 1	37.670	*301*	303	387	—
120	唑虫酰胺	Tolfenpyrad	37.947	*383*	171	197	—
121	烯酰吗啉	Dimethomorph 2	38.202	*301*	303	387	—
122	哒草氟/氟噻甲草酯	Fluthiacet-methyl	39.341	*403*	405	282	84
注：监测离子中第一栏斜体为定量离子。							

附 录 C
（资料性附录）
农药选择离子监测分组和驻留时间参数

表 C.1 农药选择离子监测分组和驻留时间参数

组号	时间/min	选择离子(amu)	驻留时间/ms
1	7.00	124,132,138,173	100
2	10.00	109,127,164,192	100
3	10.80	93,137,179,194,196,198	70
4	11.60	125,180,191,206,208	90
5	12.37	91,121,136,156,157,171	70
6	13.00	107,122,179,203,215,261	70
7	14.15	120,153,176,211,299,301	70
8	14.70	83,154,167,168,169,215,276,292,316	50
9	15.52	127,193,237,264,276,292	70
10	16.05	75,128,159,214,231,234,236,260,270	50
11	16.50	128,234,236,282,284,286	70
12	16.96	87,125,220,222,229,262	70
13	17.60	153,173,197,200,215,237,249,270,295	50
14	18.18	109,125,127,130,138,144,173,264,243	50
15	18.85	83,88,142,153,161,184,257,274,285	50
16	19.28	120,143,259,261,268,270	70
17	20.00	127,138,161,163,217,264	70
18	20.60	121,211,237,272,274,278	70
19	21.07	185,206,212,220,227,234,285,287,289	50
20	21.50	161,162,207,238,240,286	70
21	22.38	123,151,181,208,210,211,299,301,302	50
22	23.00	165,167,195,227,239,283	70
23	23.44	162,191,252,281	100
24	23.80	107,112,119,123,128,136,145,168,187,242,263	40
25	24.48	112,125,127,128,168,268,303,357,359	50
26	24.75	220,262,288,304,316,375	70
27	25.05	123,164,219,221,222,223	70
28	25.37	128,171,217,271,288,303	70
29	25.50	161,162,173,177,189,215,217,302,304	50
30	25.66	169,202,204,231,258,290	70

表 C.1（续）

组号	时间/min	选择离子(amu)	驻留时间/ms
31	26.13	105,143,172,208,217,219,235,252,273,300,305	40
32	26.40	59,91,118,247,408,412	70
33	26.72	153,231,254,282,383,384	70
34	27.50	145,156,189,204,280,322	70
35	28.00	145,173,204,222,294,303	70
36	28.28	186,220,221,223,225,272,300,387,422	50
37	28.52	186,225,238,266,300,349	70
38	28.85	253,281,340,342	100
39	29.25	165,187,189,192,194,253	70
40	29.64	118,126,152,160,163,166,292,320,335	50
41	30.00	122,140,192,193,194,238,268,300,311,320,330,359	40
42	30.48	120,125,139,148,174,184,226,288,310	50
43	30.82	77,123,132,160,183,350	70
44	31.36	96,136,185,221,226,232,344,346,373,461	50
45	33.50	125,129,180,198,331,333	70
46	34.30	140,183,243,263,265,299,342,344,372	50
47	35.50	167,225,259,287,354,419	70
48	36.70	150,203,218,301,303,308,318,387,423	50
49	37.90	171,197,301,303,383,387	70
50	39.00	84,282,403,405	100

附　录　D
（资料性附录）
农药标准物质的全扫描质量色谱图

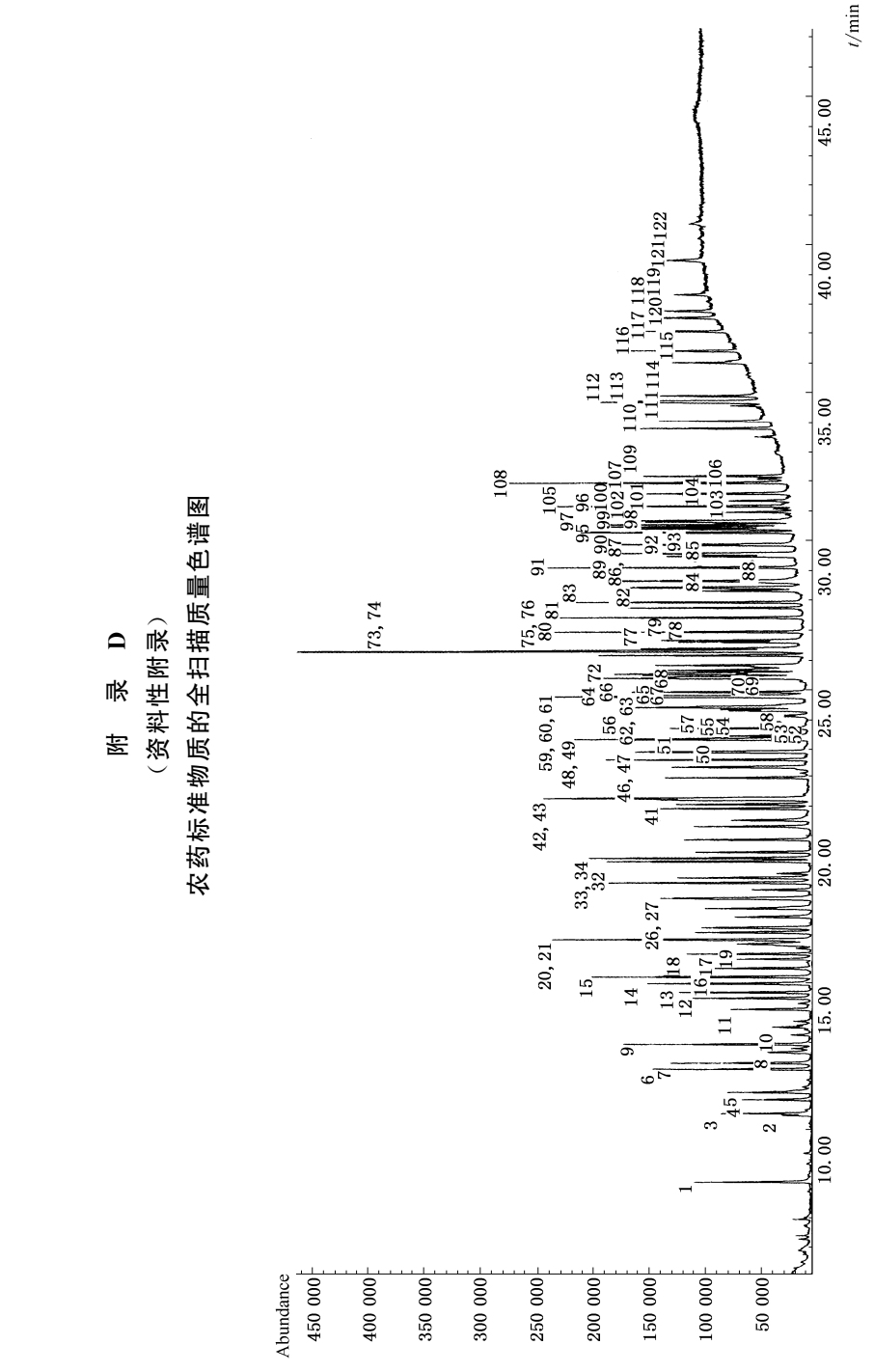

图 D.1　农药标准物质的全扫描质量色谱图

附 录 E
（规范性附录）
农药在大米、糙米、大麦、小麦及玉米中的添加回收率范围

表 E.1 农药在大米、糙米、大麦、小麦及玉米中的添加回收率范围

序号	中文通用名	添加回收率范围(添加水平为 0.01 mg/kg～0.1 mg/kg)/%				
		大米	糙米	小麦	大麦	玉米
1	烯丙菊酯	87.3～109.1	82.6～108.8	85.6～111.6	85.4～109.8	87.3～111.5
2	二丙烯草胺	80.0～105.9	81.5～107.8	80.1～104.1	81.2～104.8	81.7～103.8
3	莠灭净	87.4～106.3	87.5～109.9	86.7～105.7	86.2～106.6	87.1～107.8
4	莎稗磷	78.3～103.8	81.6～101.7	79.9～102.9	83.1～101.8	79.4～101.5
5	莠去津	88.1～106.3	80.5～102.3	88.9～107.9	86.4～110.3	86.8～107.9
6	氧环唑	85.5～109.5	88.2～109.3	85.2～109.7	85.3～111.8	85.9～111.3
7	保棉磷	83.0～104.4	72.3～104.0	85.4～109.9	83.4～104.5	85.7～107.1
8	乙丁氟灵	85.2～110.0	80.1～107.8	88.0～111.1	85.2～111.0	86.0～109.3
9	解草嗪	82.6～103.0	83.0～107.5	83.8～105.0	79.0～102.1	81.1～105.9
10	烟酰胺	86.6～108.1	84.9～102.9	85.9～109.9	85.3～107.9	85.1～108.7
11	乙基溴硫磷	85.6～108.5	80.6～109.2	85.6～110.6	85.3～109.9	86.5～108.1
12	乙嘧酚磺酸酯	86.8～109.0	85.2～105.3	86.7～104.8	92.4～107.8	86.4～106.2
13	噻嗪酮	80.5～97.6	80.7～99.6	80.0～100.0	80.2～99.7	82.8～98.7
14	英拜除草剂(暂定)	89.5～108.3	84.2～107.1	87.4～109.9	86.1～107.6	87.2～110.9
15	硫线磷	69.6～104.6	81.6～104.6	69.1～104.8	75.6～103.7	71.6～104.4
16	丁硫克百威	81.1～106.7	80.4～108.1	82.7～107.4	83.3～107.0	82.3～106.4
17	萎锈灵	83.6～101.2	82.3～101.6	82.3～103.0	78.1～103.6	79.4～101.8
18	杀螨醚	82.0～98.3	81.4～103.4	80.7～99.6	80.8～99.5	80.4～98.7

表 E.1（续）

序号	中文通用名	添加回收率范围(添加水平为 0.01 mg/kg～0.1 mg/kg)/%				
		大米	糙米	小麦	大麦	玉米
19	氯氧磷	82.7～99.9	71.2～101.6	86.5～107.3	80.8～101.1	85.5～101.7
20	虫螨腈	75.1～100.7	79.1～109.4	81.3～106.5	72.2～100.3	70.8～99.6
21	杀螨酯	80.4～103.0	84.8～103.3	80.1～109.7	83.7～102.6	78.0～103.0
22	氯草敏	83.6～106.2	75.3～104.6	87.1～109.2	82.5～104.6	82.8～109.7
23	地茂散(暂定)	80.8～107.1	82.2～106.1	78.9～104.0	81.0～106.4	80.3～107.8
24	氯酞酸甲酯	82.4～104.5	76.4～103.4	84.4～107.5	82.4～103.6	82.8～108.9
25	炔草酸	80.8～99.0	74.4～97.8	79.6～101.4	80.6～99.1	80.0～99.4
26	氯甲酰草胺	82.4～108.0	80.3～107.6	82.7～106.1	84.1～108.9	82.1～107.3
27	解草酯	82.6～105.2	81.0～103.0	82.9～107.6	82.5～104.3	79.5～105.1
28	杀螟腈	78.4～101.0	85.0～108.8	88.9～105.4	85.1～102.8	83.6～102.2
29	环草敌	82.2～102.0	84.3～103.8	80.3～106.3	82.7～104.9	79.3～104.4
30	环氟菌胺	83.4～104.9	79.6～103.8	87.5～109.4	82.4～104.0	82.1～103.7
31	燕麦敌	88.7～107.8	84.0～103.0	83.0～108.1	85.2～109.7	87.5～109.9
32	禾草灵	80.5～102.6	82.3～109.4	72.8～100.9	79.0～105.3	79.7～104.8
33	百治磷	81.6～103.1	80.3～106.4	83.2～104.4	83.6～108.2	80.5～105.8
34	哌草丹	81.0～101.7	83.9～109.3	80.1～101.0	83.9～100.8	82.2～101.7
35	乐果	83.3～106.6	78.5～105.2	82.7～101.0	85.9～107.3	82.3～109.5
36	烯酰吗啉	86.4～111.6	78.3～104.3	85.4～110.6	85.2～111.3	89.0～109.9
37	苯虫醚	80.6～109.5	83.1～102.4	84.6～99.8	84.6～107.5	80.8～102.6
38	敌恶磷	80.3～106.0	84.4～104.9	80.6～99.1	81.1～106.8	81.4～107.3
39	双苯酰草胺	78.3～102.4	87.3～105.1	85.2～109.7	78.9～103.6	79.4～100.2
40	乙拌磷	85.4～109.7	88.1～109.1	81.3～100.9	87.5～110.8	87.9～109.5

表 E.1（续）

序号	中文通用名	添加回收率范围(添加水平为 0.01 mg/kg～0.1 mg/kg)/%				
		大米	糙米	小麦	大麦	玉米
41	硫丹硫酸盐	82.8～103.0	84.0～105.7	82.8～105.5	82.9～107.6	85.8～106.2
42	氟环唑	80.3～105.9	86.4～110.9	81.7～107.9	81.1～105.1	81.4～101.6
43	S-氰戊菊酯	80.1～103.8	87.7～109.3	78.3～98.2	86.1～109.3	86.1～107.7
44	乙丁烯氟灵	86.2～110.3	88.0～109.4	85.9～104.4	85.1～109.8	87.2～111.1
45	乙硫磷	88.2～105.8	88.3～111.7	84.3～109.2	86.0～109.8	87.2～109.2
46	乙氧呋草黄	87.8～105.9	87.0～105.1	78.2～104.7	86.8～110.5	86.4～106.9
47	乙螨唑	86.0～108.9	85.1～109.9	86.3～104.2	86.7～108.9	86.7～106.4
48	咪唑菌酮	83.6～99.9	80.5～103.4	81.9～101.4	80.9～99.4	82.0～101.6
49	苯线磷	70.4～99.7	78.8～100.7	75.1～99.4	70.2～96.4	70.2～100.1
50	苯线磷砜	71.8～97.6	72.3～98.8	71.1～100.8	71.1～97.9	71.0～100.7
51	腈苯唑	83.4～104.0	80.2～105.8	79.4～103.7	78.9～103.9	83.4～103.6
52	皮蝇磷	80.0～100.3	81.1～101.5	81.1～101.8	82.3～100.2	81.8～100.3
53	嘧螨酯	82.0～101.8	81.0～100.4	83.5～100.9	82.6～102.0	81.8～101.3
54	吡氟禾草灵	76.1～98.2	76.2～109.8	73.9～101.0	75.1～100.4	74.7～99.1
55	氟噻草胺	80.0～105.5	81.4～101.9	80.5～104.6	80.8～103.4	81.7～105.8
56	氟烯草酸	80.2～105.1	80.1～103.1	80.6～102.9	81.2～105.5	80.6～105.4
57	丙炔氟草胺	72.4～97.8	82.9～108.5	75.4～97.6	77.4～97.3	72.9～98.1
58	哒草氟/氟噻甲草酯	84.5～105.3	84.5～110.1	81.9～106.3	81.6～103.7	80.2～106.5
59	粉唑醇	81.9～105.2	80.4～105.7	80.1～105.9	84.4～105.7	82.2～105.0
60	噻唑磷	80.8～100.0	79.4～101.5	80.2～99.8	80.5～99.4	80.1～99.5
61	解草恶唑	78.7～103.3	82.4～107.3	82.0～104.2	79.1～104.9	78.8～105.6
62	苄螨醚	75.6～99.1	80.0～109.1	71.7～99.1	71.7～100.9	72.3～100.3

表 E.1（续）

序号	中文通用名	添加回收率范围(添加水平为 0.01 mg/kg～0.1 mg/kg)/%				
		大米	糙米	小麦	大麦	玉米
63	氟吡禾灵	82.9～102.7	84.4～101.6	80.5～105.6	80.6～104.2	80.1～103.8
64	七氯	83.2～98.6	80.7～98.6	81.8～98.1	80.0～99.0	80.5～99.5
65	六氯苯	82.4～106.5	82.7～109.6	86.3～105.9	84.5～110.0	83.0～105.1
66	抑霉唑	86.3～110.3	86.4～107.3	86.0～107.1	86.5～107.0	87.6～110.8
67	茚草酮	86.0～108.0	86.9～104.2	87.0～108.1	89.6～107.1	88.5～110.3
68	茚虫威	88.1～108.7	81.3～98.9	86.4～107.2	86.4～110.8	90.6～109.5
69	氯唑磷	81.4～101.6	84.1～103.6	82.3～99.6	81.1～101.8	81.0～101.7
70	异丙威	86.4～105.6	86.1～111.7	86.8～109.6	86.0～106.3	86.1～106.0
71	稻瘟灵	80.3～105.2	80.3～108.7	85.1～106.9	80.1～107.8	80.5～107.2
72	双苯恶唑酸	82.6～103.1	81.1～103.9	80.8～102.2	81.2～102.3	82.4～103.9
73	乳氟禾草灵	80.2～100.0	80.9～101.1	81.2～99.7	81.1～98.9	82.4～100.0
74	吡咯二酸二乙酯	75.2～98.3	76.6～104.1	76.2～99.0	70.2～100.7	71.8～98.9
75	嘧菌胺	81.0～97.2	87.3～110.4	80.6～100.1	82.1～101.9	83.2～101.9
76	甲霜灵	82.6～105.7	84.5～108.3	82.7～105.9	79.6～105.3	78.2～100.6
77	虫螨畏	80.4～106.2	81.7～108.8	80.4～106.8	81.6～106.9	82.3～106.7
78	异丙甲草胺	87.6～110.0	86.7～108.9	88.9～108.5	86.5～110.1	86.6～106.9
79	速灭磷	83.1～101.0	81.9～105.2	85.6～102.7	82.0～102.6	78.4～103.0
80	敌草胺	80.3～106.8	84.2～102.7	81.3～106.9	87.0～104.4	82.6～105.9

表 E.1（续）

序号	中文通用名	添加回收率范围(添加水平为 0.01 mg/kg～0.1 mg/kg)/%				
		大米	糙米	小麦	大麦	玉米
81	三氯甲基吡啶	81.0～99.6	76.7～99.4	82.4～99.2	83.0～99.9	81.5～99.6
82	氟草敏	80.3～104.4	81.4～106.3	80.5～105.6	80.9～105.8	81.4～105.0
83	乙氧氟草醚	86.3～105.6	85.8～109.2	86.7～110.2	86.6～107.7	89.1～106.4
84	二甲戊灵	82.8～105.9	73.1～103.7	80.9～102.7	80.5～102.1	87.2～105.9
85	苯醚菊酯	84.0～107.8	75.4～104.6	83.8～103.7	85.1～108.7	81.3～105.1
86	甲拌磷	79.9～105.8	83.7～103.9	78.9～102.8	82.7～104.9	79.4～105.9
87	磷胺	86.3～109.6	83.2～107.9	82.0～105.7	82.8～109.7	83.0～107.8
88	哌草磷	83.9～100.5	82.1～107.7	82.0～100.6	80.0～100.9	80.0～101.9
89	茉莉酸诱导体	81.1～101.5	80.4～105.8	81.4～101.6	83.1～101.4	83.0～99.3
90	毒草胺	81.3～105.8	87.6～110.6	80.0～107.8	82.6～104.4	82.7～106.0
91	敌稗	81.8～107.2	82.6～107.2	80.2～102.2	80.0～107.5	81.2～107.6
92	丙虫磷	73.0～100.9	75.8～97.2	74.9～97.8	76.4～97.6	71.5～99.2
93	苯胺灵	84.3～109.0	81.9～105.0	85.6～107.6	81.9～104.3	85.2～104.2
94	吡菌磷	70.4～100.0	77.4～107.7	70.2～99.8	75.2～99.8	71.7～98.4
95	吡丙醚	74.6～98.6	80.9～104.7	71.9～97.6	72.9～99.6	73.0～101.0
96	咯喹酮	84.7～106.5	80.4～105.9	86.2～109.1	85.1～106.6	82.1～103.4
97	五氯硝基苯	81.0～102.5	79.1～101.9	78.5～102.7	82.6～102.2	80.3～102.8
98	精喹禾灵	80.6～104.4	81.1～100.9	78.7～103.0	80.6～101.2	83.5～105.9

表 E.1（续）

序号	中文通用名	添加回收率范围(添加水平为 0.01 mg/kg～0.1 mg/kg)/%				
		大米	糙米	小麦	大麦	玉米
99	硅氟唑	80.1～106.0	82.6～105.8	80.6～104.9	81.2～104.7	80.6～103.1
100	硫丙磷	86.1～106.5	83.3～99.2	82.6～109.1	87.1～108.1	84.9～105.1
101	丁噻隆	80.7～107.3	81.7～105.8	80.2～105.6	83.4～106.4	81.5～107.8
102	四氯硝基苯	83.0～97.8	81.2～103.0	78.5～100.9	78.5～101.5	81.3～102.1
103	唑虫酰胺	86.5～108.2	71.3～100.3	87.7～104.3	87.9～104.4	87.6～107.2
104	三唑酮	81.3～99.8	80.0～97.3	81.9～98.2	80.2～99.9	80.6～99.4
105	三唑醇	80.8～99.4	82.2～101.7	80.2～96.9	83.2～97.9	80.2～98.8
106	野麦畏	85.4～111.6	83.3～107.9	86.4～110.1	85.2～110.0	87.9～108.1
107	脱叶磷	80.6～101.6	82.1～100.5	80.7～101.9	79.9～102.3	79.2～102.1
108	三环唑	83.4～99.5	84.0～102.5	81.0～99.8	81.5～99.9	80.8～98.3
109	灭除威	82.8～101.1	80.1～105.7	80.5～99.9	81.5～101.4	81.9～101.4
110	苯酰菌胺	89.4～102.1	80.2～106.0	88.1～106.4	85.9～106.6	86.0～108.3

Foreword

Annex A and E are normative annex，annex B，annex C，annex D of this standard are informative annexes.

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 is drafted by Chinese Academy of Inspection and Quarantine，Heilongjiang Entry-Exit

Inspection and Quarantine Bureau of the People's Republic of China and Neimenggu Entry-Exit Inspection and Quarantine Bureau of the People's Republic of China.

Main drafters of this standard are：Li Shujuan，Li Gang，Chen Dongdong，Dai hanhui，Feng Fan，Feng Nan，Luo Wei，Kang Qinghe，Yang Changzhi，Li Gang，CaiHuixia，Shi Jing，Tang Yingzhang.

This standard is a professional standard for Entry-Exit inspection and quarantine of the People's Republic of China promulgated for the first time.

Determination of benoxacor，anilofos，allidochlor 110 pesticides residues in foodstuffs for import and export—GC-MS Method

1 Scope

This standard specifies the determination of 110 pesticides residues in foodstuffs by GC-MS.

This standard is applicable to the determination of 110 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 byliquid liquid partition and 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 pure“water”is distilled water.

3. 1 Acetone：HPLC grade.

3. 2 Acetone.

3. 3 Dichloromethane.

3. 4 Cyclohexane.

3. 5 Ethyl acetate.

3. 6 Acetonitrile.

3. 7 Toluene.

3. 8 Sodium chloride.

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

3.10 15% sodium chloride solution(W/V): 15 g sodium chloride is dissolved in water, then dilute to 100 mL.

3.11 Cyclohexane-Ethyl acetate: (1+1, V/V).

3.12 Acetonitrile-Toluene: (3+1, V/V).

3.13 Pesticides standards: purity≥95%. Other information is in annex A.

3.14 Pesticides standards solution

3.14.1 Standard stock solution

Accurately weigh up adequate amount of each standard(accurate to 0.1 mg), dissolve in Acetone(3.1) and prepare for solution of 500 mg/L as the standard stock solution, stored below −18 ℃, avoiding sunlight for three months.

3.14.2 Middle standard solution:

Divide 110 pestcides into 5 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 middle standard solution, stored below 4 ℃ avoiding sunlight for one month.

3.14.3 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.

3.15 Celite 545 or equivalent.

3.16 Envi-Carb/LC-NH_2 SPE colume: 500 mg/500 mg, 6 mL, or equivalent.

4 Apparatus and equipment

4.1 analyse balance: Accuracy to 0.01 g.

4.2 analyse balance: Accuracy to 0.1 mg.

4.3 SPE cartridge.

4.4 Sieve:20 mesh.

4.5 Shaker.

4.6 Pump and filtrate apparatus.

4.7 Rotary vacuum evaporator.

4.8 Nitrogen evaporator.

4.9 Vortex mixer.

4.10 GC-MS:EI source.

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

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 20 mesh sieve,and then divided into two equal portions. Each portion is placed in clean containers as the testsample,which is sealed and labeled. The test sample should be stored at 0 ℃～4 ℃ avoiding sunlight.

5.2 Sample storge

In 0 ℃～4 ℃ sample preparation,precautions should 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 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(3.15)is added. And wash the taper flask and extract with 3×5 mL acetone,combine filtrate. The filtrate is reduced to about 20 mL in rotary vacuum evaporator at 40 ℃,then is transfered to separating funnel. 50 mL 15% NaCl solution (3.10) and 50 mL dichloromethane 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 ℃, and blow it to dryness under nitrogen. The residue is reconstituted by 4.0 mL Cyclohexane-Ethyl acetate, and is ready for clean up.

6.2 Clean up

6.2.1 GPC clean up

Put the solution which is ready to clean up in 6.1 on the GPC, the injecton 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 rotory evaporator under 40 ℃ water. Then blow it to dryness completely with Nitrogen gas. Use 2 mL acetonitrile-toluene to resolve the residue.

6.2.2 SPE clean up

Put the Envi-Carb/LC-NH_2 columns on the SPE cartridge, use 10 mL acetonitrile-toluene to wash it. Transfer the solution purified by 6.2.1 into the column and collect outflow in same time. Use 3×1 mL acetonitrile-toluene to clean the container and also transfer it into the SPE column. Control the flow rate of outflow to 2 mL/min. Then use 20 mL acetonitrile-toluene to wash the SPE column and combine the outflow to 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

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

b) Oven temperature: 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 10 min;

c) Carrier gas: Helium, purity≥99.999%, flow rate: 1.0 mL/min;

d) Injection port temperature: 250 ℃;

e) Injection volume: 2 μL;

f) Injection mode: Pulsed splitless, pulsed pressure 137.9 kPa(20 Psi), hold 1.5 min;

g) EI source: 70 eV;

h) Ion source temperature:230 ℃;

i) Quadrupole temperature:150 ℃;

j) Interface temperature:280 ℃;

k) Solvent delay time:7 min;

l) 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 C.

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 pestcide response in the sample must in the linear range of the matrix mix standard working solution. The full-scan totle ion chromatogram of 110 pestcide is in annex D.

6.4 Parallel test

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

6.5 Blank test

Parallel 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 following formula(1),(The blank value should be subtracted from the result of calculation.):

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

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.

8 Limit of quantification and recovery

8.1 Limit of quantification

Use this method to determine 110 pestcides 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 110 pestcide is in annex E.

Annex A
（Normative）
Pesticides standards information

Table A. 1—**Information of 110 pesticides**

NO	Compound Name	CAS RN	Group
1	*Allethrin*	584-79-2	A
2	Allidochlor	93-71-0	A
3	Ametryn	834-12-8	A
4	Anilofos	64249-01-0	A
5	Atrazine	1912-24-9	A
6	Azaconazole	60207-31-0	A
7	Azinphos-methyl	86-50-0	A
8	Benfluralin	1861-40-1	A
9	Benoxacor	98730-04-2	A
10	Boscalid	188425-85-6	A
11	Bromophos-ethyl	4824-78-6	A
12	Bupirimate	41483-43-6	A
13	Buprofezin	69327-76-0	A
14	Butafenacil	134605-64-4	A
15	Cadusafos	95465-99-9	A
16	Carbosulfan	55285-14-8	A
17	Carboxin	5234-68-4	A
18	Chlorbenside	103-17-3	A
19	Chlorethoxyfos	54593-83-8	A
20	Chlorfenapyr	122453-73-0	A
21	Chlorfenson	80-33-1	B
22	Chloridazon	1698-60-8	B
23	Chlorneb	2675-77-6	B
24	Chlorthal-dimethyl	1861-32-1	B
25	Clodinafop-propargyl	105512-06-9	B
26	clomeprop	84496-56-0	B
27	Cloquintocet-mexyl	99607-70-2	B
28	Cyanophos	2636-26-2	B
29	Cycloate	1134-23-2	B
30	Cyflufenamid	180409-60-3	B
31	*Diallate*	2303-16-4	B

Table A. 1 (continue)

NO	Compound Name	CAS RN	Group
32	Diclofop-methyl	51338-27-3	B
33	Dicrotophos	141-66-2	B
34	Dimepiperate	61432-55-1	B
35	Dimethoate	60-51-5	B
36	*Dimethomorph*	110488-70-5	B
37	*Diofenolan*	63837-33-2	B
38	Dioxathion	78-34-2	B
39	Diphenamid	957-51-7	B
40	Disulfoton	298-04-4	B
41	Endosulfan-sulfate	1031-07-8	C
42	Epoxiconazole	106325-08-0	C
43	Esfenvalerate	66230-04-4	C
44	Ethalfluralin	55283-68-6	C
45	Ethion	563-12-2	C
46	Ethofumesate	26225-79-6	C
47	Etoxazole	153233-91-1	C
48	Fenamidone	161326-34-7	C
49	Fenamiphos	22224-92-6	C
50	Fenamiphos-sulfone	31972-44-8	C
51	Fenbuconazole	114369-43-6	C
52	Fenchlorphos	299-84-3	C
53	Fluacrypyrim	229977-93-9	C
54	Fluazifop-butyl	69806-50-4	C
55	Flufenacet	142459-58-3	C
56	Flumiclorac pentyl	87546-18-7	C
57	Flumioxazin	103361-09-7	C
58	Fluthiacet-methyl	117337-19-6	C
59	Flutriafol	76674-21-0	C
60	*Fosthiazate*	98886-44-3	C
61	Furilazole	121776-33-8	C
62	Halfenprox	111872-58-3	C
63	Haloxyfop-methyl	69806-40-2	C
64	Heptachlor	76-44-8	C
65	Hexachlorobenzene	118-74-1	C
66	Imazalil	35554-44-0	D

Table A. 1（continue）

NO	Compound Name	CAS RN	Group
67	Indanofan	133220-30-1	D
68	Indoxacarb	144171-61-9	D
69	Isazofos	42509-80-8	D
70	isoprocarb	2631-40-5	D
71	Isoprothiolane	50512-35-1	D
72	Isoxadifen-ethyl	163520-33-0	D
73	Lactofen	77501-63-4	D
74	Mefenpyr-diethyl	135590-91-9	D
75	Mepanipyrim	110235-47-7	D
76	Metalaxyl	57837-19-1	D
77	Methacrifos	62610-77-9	D
78	metolachlor	51218-45-2	D
79	*Mevinphos*	26718-65-0	D
80	Napropamide	15299-99-7	D
81	Nitrapyrin	1929-82-4	D
82	Norflurazon	27314-13-2	D
83	Oxyfluorfen	42874-03-3	D
84	pendimethalin	40487-42-1	D
85	*Phenothrin*	26002-80-2	D
86	Phorate	298-02-2	D
87	*Phosphamidon*	13171-21-6	D
88	Piperophos	24151-93-7	D
89	Prohydrojasmon	158474-72-7	D
90	Propachlor	1918-16-7	D
91	Propanil	709-98-8	D
92	Propaphos	7292-16-2	E
93	Propham	122-42-9	E
94	Pyrazophos	13457-18-6	E
95	Pyriproxyfen	95737-68-1	E
96	Pyroquilon	57369-32-1	E
97	Quintozene	82-68-8	E
98	Quizalofop-ethyl	76578-14-8	E
99	Simeconazole	149508-90-7	E
100	Sulprofos	35400-43-2	E
101	Tebuthiuron	34014-18-1	E

Table A. 1（continue）

NO	Compound Name	CAS RN	Group
102	Tecnazene	117-18-0	E
103	Tolfenpyrad	129558-76-5	E
104	Triadimefon	43121-43-3	E
105	*Triadimenol*	55219-65-3	E
106	Triallate	2303-17-5	E
107	Tribuphos	78-48-8	E
108	Tricyclazole	41814-78-2	E
109	XMC	2655-14-3	E
110	*Zoxamide*	156052-68-5	E
Note：The italics of second line are the compouds with isomers.			

Annex B
(informative)
GC/MSD Condition

Table B. 1—**RT and Selected Ion**

NO	Compound Name	RT/min	Monitor ion			
1	Allidochlor	8. 176	*132*	124	138	173
2	trans-Mevinphos	10. 422	*192*	109	127	164
3	cis-Mevinphos	10. 490	*192*	109	127	164
4	Nitrapyrin	10. 956	*196*	194	198	—
5	Propham	11. 176	*179*	93	137	—
6	Methacrifos	11. 968	*208*	125	180	—
7	Chlorneb	12. 153	*191*	206	208	—
8	Tebuthiuron	12. 480	*156*	157	171	—
9	isoprocarb	12. 790	*121*	91	136	—
10	XMC	13. 344	*122*	107	179	—
11	Tecnazene	13. 966	*261*	203	215	—
12	Propachlor	14. 363	*120*	176	211	—
13	Chlorethoxyfos	14. 582	*299*	153	301	—
14	Cycloate	15. 089	*154*	83	215	—
15	Ethalfluralin	15. 372	*276*	316	292	—
16	Dicrotophos	15. 688	*127*	193	237	—
17	Benfluralin	15. 897	*292*	264	276	—
18	Cadusafos	16. 236	*159*	214	270	—
19	Di-allate 1	16. 390	*234*	128	236	—
20	Phorate	16. 398	*260*	75	231	—
21	Hexachlorobenzene	16. 598	*284*	282	286	—
22	Di-allate 2	16. 782	*234*	128	236	—
23	Dimethoate	17. 125	*87*	125	229	—
24	Furilazole	17. 439	*220*	222	262	—
25	Quintozene	17. 740	*295*	237	249	—
26	Atrazine	17. 753	*200*	173	215	—
27	Dioxathion	18. 081	*270*	197	153	—
28	Cyanophos	18. 308	*243*	109	125	—
29	Pyroquilon	18. 462	*173*	130	144	—
30	Phosphamidon 1	18. 646	*127*	138	264	—

Table B. 1（continue）

NO	Compound Name	RT/min	Monitor ion			
31	Disulfoton	19.062	*274*	88	142	—
32	Prohydrojasmon	19.164	*83*	153	184	—
33	Isazophos	19.181	*257*	161	285	—
34	Tri-allate	19.392	*268*	270	143	—
35	Benoxacor	19.770	*120*	259	261	—
36	Phosphamidon 2	20.241	*127*	138	264	—
37	Propanil	20.351	*163*	161	217	—
38	Simeconazole	20.829	*121*	211	278	—
39	heptachlor	21.003	*272*	237	274	—
40	Metalaxyl	21.148	*206*	220	234	—
41	Ametryn	21.179	*227*	185	212	—
42	Fenchlorphos	21.184	*285*	287	289	—
43	Ethofumesate	21.896	*207*	161	286	—
44	metolachlor	22.268	*162*	238	240	—
45	Chlorthal-dimethyl	22.506	*301*	299	332	—
46	Flufenacet	22.760	*151*	123	211	—
47	Triadimefon	22.772	*208*	181	210	—
48	Diphenamid	23.191	*167*	165	239	—
49	Fosthiazate 1	23.223	*195*	227	283	—
50	Fosthiazate 2	23.313	*195*	227	283	—
51	pendimethalin	23.572	*252*	162	191	281
52	Allethrin 1	23.991	*123*	107	136	—
53	Allethrin 2	24.035	*123*	107	136	—
54	Allethrin 3	24.185	*123*	107	136	—
55	Allethrin 4	24.231	*123*	107	136	—
56	Dimepiperate	24.287	*119*	145	263	—
57	Triadimenol 1	24.297	*168*	112	128	—
58	Zoxamide 1	24.378	*187*	242	—	—
59	Triadimenol 2	24.576	*168*	112	128	—
60	Bromophos-ethyl	24.649	*359*	303	357	—
61	Chlorbenside	24.668	*125*	127	268	—
62	Propaphos	24.823	*220*	262	304	—
63	Haloxyfop-methyl	24.824	*316*	288	375	—
64	Mepanipyrim	25.256	*222*	221	223	—

Table B. 1 (continue)

NO	Compound Name	RT/min	Monitor ion			
65	Flutriafol	25. 307	*123*	164	219	—
66	Napropamide	25. 418	*271*	128	171	—
67	Fenamiphos	25. 445	*303*	217	288	—
68	Chlorfenson	25. 540	*177*	302	304	—
69	Imazalil	25. 585	*215*	173	217	—
70	Tricyclazole	25. 591	*189*	161	162	—
71	Isoprothiolane	25. 722	*204*	231	290	—
72	Tribuphos	26. 066	*169*	202	258	—
73	Carboxin	26. 194	*235*	143	—	—
74	Buprofezin	26. 197	*105*	172	305	—
75	Oxyfluorfen	26. 202	*252*	300	—	—
76	Bupirimate	26. 209	*208*	273	—	—
77	Azaconazole	26. 271	*217*	219	—	—
78	Chlorfenapyr	26. 497	*59*	247	408	—
79	Cyflufenamid	26. 587	*118*	91	412	—
80	Fluazifop-butyl	26. 866	*282*	254	383	—
81	Ethion	27. 361	*231*	153	384	—
82	Fluacrypyrim	27. 695	*145*	189	204	—
83	Sulprofos	27. 866	*322*	156	280	—
84	Isoxadifen-ethyl	28. 081	*204*	222	294	—
85	Norflurazon	28. 210	*303*	145	173	—
86	Endosulfan sulfate	28. 334	*272*	387	422	—
87	Diofenolan 1	28. 386	*186*	225	300	—
88	Chloridazon	28. 440	*221*	220	223	—
89	Diofenolan 2	28. 591	*186*	225	300	—
90	Clodinafop-propargyl	28. 607	*266*	238	349	—
91	Diclofop-methyl	29. 054	*253*	281	340	342
92	Epoxiconazole	29. 409	*192*	165	194	—
93	Zoxamide 2	29. 442	*187*	189	258	—
94	Mefenpyr-diethyl	29. 529	*253*	255	299	—
95	Fenamiphos-sulfone	29. 769	*320*	292	335	—
96	Carbosulfan	29. 843	*160*	118	163	—
97	Piperophos	30. 183	*122*	140	320	—
98	Cloquintocet-mexyl	30. 296	*192*	193	194	—

Table B. 1（continue）

NO	Compound Name	RT/min	Monitor ion			
99	Etoxazole	30.372	*300*	330	359	—
100	Fenamidone	30.426	*238*	268	—	—
101	Indanofan	30.540	*139*	174	310	—
102	Anilofos	30.582	*226*	125	184	—
103	Clomeprop	30.762	*288*	120	148	—
104	Phenothrin 1	30.887	*123*	183	350	—
105	Phenothrin 2	31.085	*123*	183	350	—
106	Azinphos-methyl	31.232	*77*	132	160	—
107	Pyriproxyfen	31.472	*136*	96	226	—
108	Lactofen	31.836	*344*	346	461	—
109	Pyrazophos	32.054	*221*	232	373	—
110	Butafenacil	33.659	*331*	180	333	—
111	Fenbuconazole	33.906	*129*	125	198	—
112	Boscalid	34.583	*140*	342	344	—
113	Halfenprox	34.662	*265*	183	263	—
114	Quizalofop-ethyl	34.822	*299*	243	372	—
115	Flumioxazin	35.932	*354*	259	287	—
116	Esfenvalerate	36.349	*167*	225	419	—
117	Indoxacarb	37.018	*203*	150	218	—
118	Flumiclorac pentyl	37.451	*423*	308	318	—
119	Dimethomorph 1	37.670	*301*	303	387	—
120	Tolfenpyrad	37.947	*383*	171	197	—
121	Dimethomorph 2	38.202	*301*	303	387	—
122	Fluthiacet-methyl	39.341	*403*	405	282	84
Note：The first line of Monitor Ion is quantitative ion.						

Annex C
(informative)
Groups of selected ion and Dewll time

Table C. 1—**Groups of selected ion and Dewll time**

Group	Time/min	Selected Ion(amu)	Dwell/ms
1	7.00	124,132,138,173	100
2	10.00	109,127,164,192	100
3	10.80	93,137,179,194,196,198	70
4	11.60	125,180,191,206,208	90
5	12.37	91,121,136,156,157,171	70
6	13.00	107,122,179,203,215,261	70
7	14.15	120,153,176,211,299,301	70
8	14.70	83,154,167,168,169,215,276,292,316	50
9	15.52	127,193,237,264,276,292	70
10	16.05	75,128,159,214,231,234,236,260,270	50
11	16.50	128,234,236,282,284,286	70
12	16.96	87,125,220,222,229,262	70
13	17.60	153,173,197,200,215,237,249,270,295	50
14	18.18	109,125,127,130,138,144,173,264,243	50
15	18.85	83,88,142,153,161,184,257,274,285	50
16	19.28	120,143,259,261,268,270	70
17	20.00	127,138,161,163,217,264	70
18	20.60	121,211,237,272,274,278	70
19	21.07	185,206,212,220,227,234,285,287,289	50
20	21.50	161,162,207,238,240,286	70
21	22.38	123,151,181,208,210,211,299,301,302	50
22	23.00	165,167,195,227,239,283	70
23	23.44	162,191,252,281	100
24	23.80	107,112,119,123,128,136,145,168,187,242,263	40
25	24.48	112,125,127,128,168,268,303,357,359	50
26	24.75	220,262,288,304,316,375	70
27	25.05	123,164,219,221,222,223	70
28	25.37	128,171,217,271,288,303	70
29	25.50	161,162,173,177,189,215,217,302,304	50
30	25.66	169,202,204,231,258,290	70

Table C.1 (continue)

Group	Time/min	Selected Ion(amu)	Dwell/ms
31	26.13	105,143,172,208,217,219,235,252,273,300,305	40
32	26.40	59,91,118,247,408,412	70
33	26.72	153,231,254,282,383,384	70
34	27.50	145,156,189,204,280,322	70
35	28.00	145,173,204,222,294,303	70
36	28.28	186,220,221,223,225,272,300,387,422	50
37	28.52	186,225,238,266,300,349	70
38	28.85	253,281,340,342	100
39	29.25	165,187,189,192,194,253	70
40	29.64	118,126,152,160,163,166,292,320,335	50
41	30.00	122,140,192,193,194,238,268,300,311,320,330,359	40
42	30.48	120,125,139,148,174,184,226,288,310	50
43	30.82	77,123,132,160,183,350	70
44	31.36	96,136,221,226,232,344,346,373,461	50
45	33.50	125,129,180,198,331,333	70
46	34.30	140,183,243,263,265,299,342,344,372	50
47	35.50	167,225,259,287,354,419	70
48	36.70	150,203,218,301,303,308,318,387,423	50
49	37.90	171,197,301,303,383,387	70
50	39.00	84,282,403,405	100

Annex D
（informative）
Chromatogram of 110 pestcides in Full scan mode

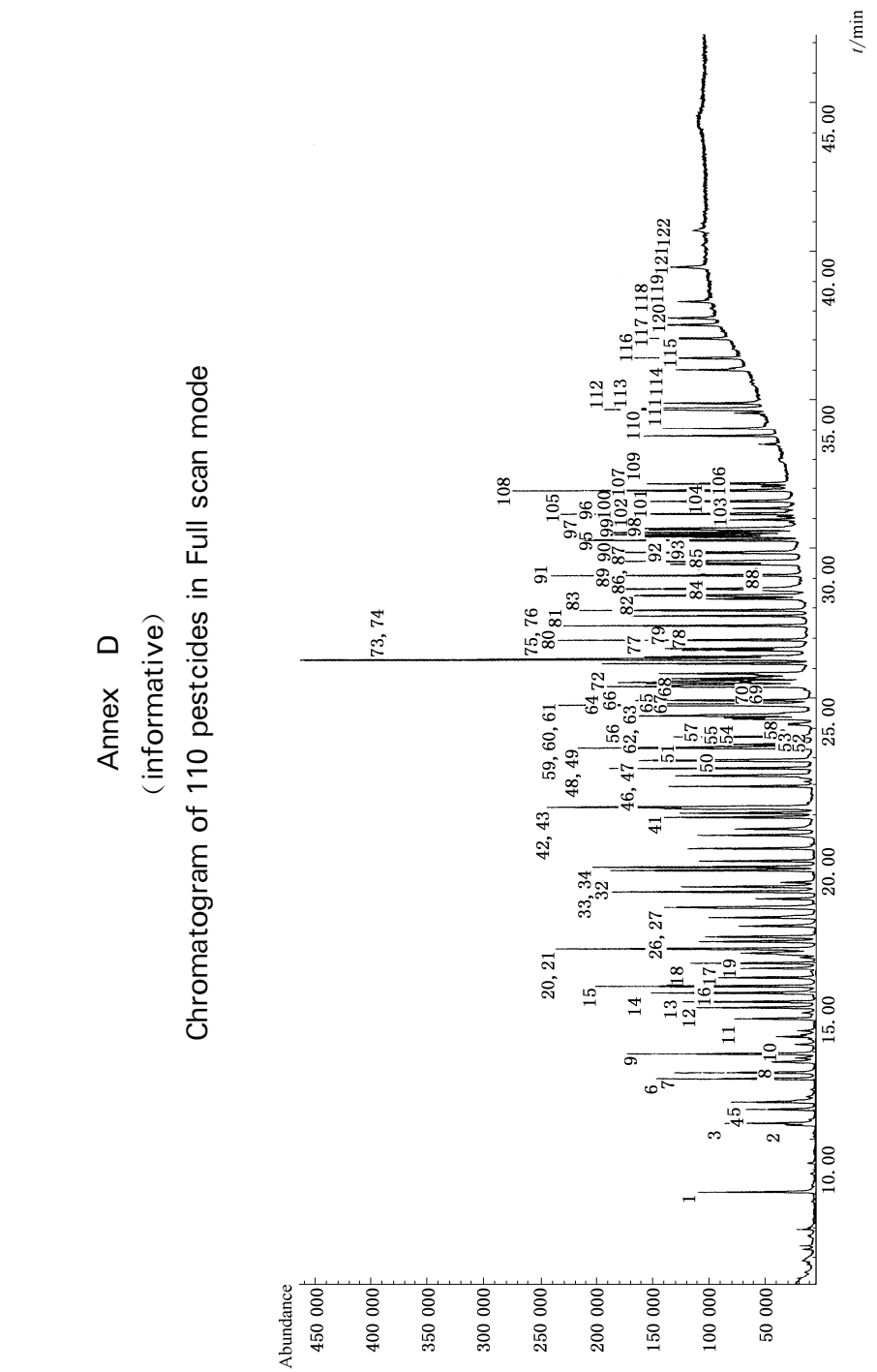

Figure D. 1—Chromatogram of 110 pestcides in Full scan mode

Annex E
(Normative)
Recovery range of 110 pestcides in rice, brown rice, barley, wheat and corn

Table E. 1—**Recovery range of 110 pestcides in rice, brown rice, barley, wheat and corn**

NO	Compound name	Range of recovery/%(spiked level is 0.01 mg/kg～0.1 mg/kg)				
		Rice	Brown rice	Wheat	Barley	Corn
1	Allethrin	87.3～109.1	82.6～108.8	85.6～111.6	85.4～109.8	87.3～111.5
2	Allidochlor	80.0～105.9	81.5～107.8	80.1～104.1	81.2～104.8	81.7～103.8
3	Ametryn	87.4～106.3	87.5～109.9	86.7～105.7	86.2～106.6	87.1～107.8
4	Anilofos	78.3～103.8	81.6～101.7	79.9～102.9	83.1～101.8	79.4～101.5
5	Atrazine	88.1～106.3	80.5～102.3	88.9～107.9	86.4～110.3	86.8～107.9
6	Azaconazole	85.5～109.5	88.2～109.3	85.2～109.7	85.3～111.8	85.9～111.3
7	Azinphos-methyl	83.0～104.4	72.3～104.0	85.4～109.9	83.4～104.5	85.7～107.1
8	Benfluralin	85.2～110.0	80.1～107.8	88.0～111.1	85.2～111.0	86.0～109.3
9	Benoxacor	82.6～103.0	83.0～107.5	83.8～105.0	79.0～102.1	81.1～105.9
10	Boscalid	86.6～108.1	84.9～102.9	85.9～109.9	85.3～107,9	85.1～108.7
11	Bromophos-ethyl	85.6～108.5	80.6～109.2	85.6～110.6	85.3～109.9	86.5～108.1
12	Bupirimate	86.8～109.0	85.2～105.3	86.7～104.8	92.4～107.8	86.4～106.2
13	Buprofezin	80.5～97.6	80.7～99.6	80.0～100.0	80.2～99.7	82.8～98.7
14	Butafenacil	89.5～108.3	84.2～107.1	87.4～109.9	86.1～107.6	87.2～110.9
15	Cadusafos	69.6～104.6	81.6～104.6	69.1～104.8	75.6～103.7	71.6～104.4
16	Carbosulfan	81.1～106.7	80.4～108.1	82.7～107.4	83.3～107.0	82.3～106.4
17	Carboxin	83.6～101.2	82.3～101.6	82.3～103.0	78.1～103.6	79.4～101.8
18	Chlorbenside	82.0～98.3	81.4～103.4	80.7～99.6	80.8～99.5	80.4～98.7

Table E. 1（continue）

NO	Compound name	Range of recovery/%（spiked level is 0.01 mg/kg～0.1 mg/kg）				
		Rice	Brown rice	Wheat	Barley	Corn
19	Chlorethoxyfos	82.7～99.9	71.2～101.6	86.5～107.3	80.8～101.1	85.5～101.7
20	Chlorfenapyr	75.1～100.7	79.1～109.4	81.3～106.5	72.2～100.3	70.8～99.6
21	Chlorfenson	80.4～103.0	84.8～103.3	80.1～109.7	83.7～102.6	78.0～103.0
22	Chloridazon	83.6～106.2	75.3～104.6	87.1～109.2	82.5～104.6	82.8～109.7
23	Chlorneb	80.8～107.1	82.2～106.1	78.9～104.0	81.0～106.4	80.3～107.8
24	Chlorthal-dimethyl	82.4～104.5	76.4～103.4	84.4～107.5	82.4～103.6	82.8～108.9
25	Clodinafop-propargyl	80.8～99.0	74.4～97.8	79.6～101.4	80.6～99.1	80.0～99.4
26	clomeprop	82.4～108.0	80.3～107.6	82.7～106.1	84.1～108.9	82.1～107.3
27	Cloquintocet-mexyl	82.6～105.2	81.0～103.0	82.9～107.6	82.5～104.3	79.5～105.1
28	Cyanophos	78.4～101.0	85.0～108.8	88.9～105.4	85.1～102.8	83.6～102.2
29	Cycloate	82.2～102.0	84.3～103.8	80.3～106.3	82.7～104.9	79.3～104.4
30	Cyflufenamid	83.4～104.9	79.6～103.8	87.5～109.4	82.4～104.0	82.1～103.7
31	Diallate	88.7～107.8	84.0～103.0	83.0～108.1	85.2～109.7	87.5～109.9
32	Diclofop-methyl	80.5～102.6	82.3～109.4	72.8～100.9	79.0～105.3	79.7～104.8
33	Dicrotophos	81.6～103.1	80.3～106.4	83.2～104.4	83.6～108.2	80.5～105.8
34	Dimepiperate	81.0～101.7	83.9～109.3	80.1～101.0	83.9～100.8	82.2～101.7
35	Dimethoate	83.3～106.6	78.5～105.2	82.7～101.0	85.9～107.3	82.3～109.5
36	Dimethomorph	86.4～111.6	78.3～104.3	85.4～110.6	85.2～111.3	89.0～109.9
37	Diofenolan	80.6～109.5	83.1～102.4	84.6～99.8	84.6～107.5	80.8～102.6
38	Dioxathion	80.3～106.0	84.4～104.9	80.6～99.1	81.1～106.8	81.4～107.3
39	Diphenamid	78.3～102.4	87.3～105.1	85.2～109.7	78.9～103.6	79.4～100.2
40	Disulfoton	85.4～109.7	88.1～109.1	81.3～100.9	87.5～110.8	87.9～109.5

Table E. 1 (continue)

NO	Compound name	Range of recovery/%(spiked level is 0.01 mg/kg～0.1 mg/kg)				
		Rice	Brown rice	Wheat	Barley	Corn
41	Endosulfan-sulfate	82.8～103.0	84.0～105.7	82.8～105.5	82.9～107.6	85.8～106.2
42	Epoxiconazole	80.3～105.9	86.4～110.9	81.7～107.9	81.1～105.1	81.4～101.6
43	Esfenvalerate	80.1～103.8	87.7～109.3	78.3～98.2	86.1～109.3	86.1～107.7
44	Ethalfluralin	86.2～110.3	88.0～109.4	85.9～104.4	85.1～109.8	87.2～111.1
45	Ethion	88.2～105.8	88.3～111.7	84.3～109.2	86.0～109.8	87.2～109.2
46	Ethofumesate	87.8～105.9	87.0～105.1	78.2～104.7	86.8～110.5	86.4～106.9
47	Etoxazole	86.0～108.9	85.1～109.9	86.3～104.2	86.7～108.9	86.7～106.4
48	Fenamidone	83.6～99.9	80.5～103.4	81.9～101.4	80.9～99.4	82.0～101.6
49	Fenamiphos	70.4～99.7	78.8～100.7	75.1～99.4	70.2～96.4	70.2～100.1
50	Fenamiphos-sulfone	71.8～97.6	72.3～98.8	71.1～100.8	71.1～97.9	71.0～100.7
51	Fenbuconazole	83.4～104.0	80.2～105.8	79.4～103.7	78.9～103.9	83.4～103.6
52	Fenchlorphos	80.0～100.3	81.1～101.5	81.1～101.8	82.3～100.2	81.8～100.3
53	Fluacrypyrim	82.0～101.8	81.0～100.4	83.5～100.9	82.6～102.0	81.8～101.3
54	Fluazifop-butyl	76.1～98.2	76.2～109.8	73.9～101.0	75.1～100.4	74.7～99.1
55	Flufenacet	80.0～105.5	81.4～101.9	80.5～104.6	80.8～103.4	81.7～105.8
56	Flumiclorac pentyl	80.2～105.1	80.1～103.1	80.6～102.9	81.2～105.5	80.6～105.4
57	Flumioxazin	72.4～97.8	82.9～108.5	75.4～97.6	77.4～97.3	72.9～98.1
58	Fluthiacet-methyl	84.5～105.3	84.5～110.1	81.9～106.3	81.6～103.7	80.2～106.5
59	Flutriafol	81.9～105.2	80.4～105.7	80.1～105.9	84.4～105.7	82.2～105.0
60	Fosthiazate	80.8～100.0	79.4～101.5	80.2～99.8	80.5～99.4	80.1～99.5
61	Furilazole	78.7～103.3	82.4～107.3	82.0～104.2	79.1～104.9	78.8～105.6
62	Halfenprox	75.6～99.1	80.0～109.1	71.7～99.1	71.7～100.9	72.3～100.3

Table E.1 (continue)

NO	Compound name	Range of recovery/%(spiked level is 0.01 mg/kg～0.1 mg/kg)				
		Rice	Brown rice	Wheat	Barley	Corn
63	Haloxyfop-methyl	82.9～102.7	84.4～101.6	80.5～105.6	80.6-104.2	80.1～103.8
64	Heptachlor	83.2～98.6	80.7～98.6	81.8～98.1	80.0～99.0	80.5～99.5
65	Hexachlorobenzene	82.4～106.5	82.7～109.6	86.3～105.9	84.5～110.0	83.0～105.1
66	Imazalil	86.3～110.3	86.4～107.3	86.0～107.1	86.5～107.0	87.6～110.8
67	Indanofan	86.0～108.0	86.9～104.2	87.0～108.1	89.6～107.1	88.5～110.3
68	Indoxacarb	88.1～108.7	81.3～98.9	86.4～107.2	86.4～110.8	90.6～109.5
69	Isazofos	81.4～101.6	84.1～103.6	82.3～99.6	81.1～101.8	81.0～101.7
70	Isoprocarb	86.4～105.6	86.1～111.7	86.8～109.6	86.0～106.3	86.1～106.0
71	Isoprothiolane	80.3～105.2	80.3～108.7	85.1～106.9	80.1～107.8	80.5～107.2
72	Isoxadifen-ethyl	82.6～103.1	81.1～103.9	80.8～102.2	81.2～102.3	82.4～103.9
73	Lactofen	80.2～100.0	80.9～101.1	81.2～99.7	81.1～98.9	82.4～100.0
74	Mefenpyr-diethyl	75.2～98.3	76.6～104.1	76.2～99.0	70.2～100.7	71.8～98.9
75	Mepanipyrim	81.0～97.2	87.3～110.4	80.6～100.1	82.1～101.9	83.2～101.9
76	Metalaxyl	82.6～105.7	84.5～108.3	82.7～105.9	79.6～105.3	78.2～100.6
77	Methacrifos	80.4～106.2	81.7～108.8	80.4～106.8	81.6～106.9	82.3～106.7
78	Metolachlor	87.6～110.0	86.7～108.9	88.9～108.5	86.5～110.1	86.6～106.9
79	Mevinphos	83.1～101.0	81.9～105.2	85.6～102.7	82.0～102.6	78.4～103.0
80	Napropamide	80.3～106.8	84.2～102.7	81.3～106.9	87.0～104.4	82.6～105.9

Table E. 1 (continue)

NO	Compound name	Range of recovery/%(spiked level is 0.01 mg/kg～0.1 mg/kg)				
		Rice	Brown rice	Wheat	Barley	Corn
81	Nitrapyrin	81.0～99.6	76.7～99.4	82.4～99.2	83.0～99.9	81.5～99.6
82	Norflurazon	80.3～104.4	81.4～106.3	80.5～105.6	80.9～105.8	81.4～105.0
83	Oxyfluorfen	86.3～105.6	85.8～109.2	86.7～110.2	86.6～107.7	89.1～106.4
84	Pendimethalin	82.8～105.9	73.1～103.7	80.9～102.7	80.5～102.1	87.2～105.9
85	Phenothrin	84.0～107.8	75.4～104.6	83.8～103.7	85.1～108.7	81.3～105.1
86	Phorate	79.9～105.8	83.7～103.9	78.9～102.8	82.7～104.9	79.4～105.9
87	Phosphamidon	86.3～109.6	83.2～107.9	82.0～105.7	82.8～109.7	83.0～107.8
88	Piperophos	83.9～100.5	82.1～107.7	82.0～100.6	80.0～100.9	80.0～101.9
89	Prohydrojasmon	81.1～101.5	80.4～105.8	81.4～101.6	83.1～101.4	83.0～99.3
90	Propachlor	81.3～105.8	87.6～110.6	80.0～107.8	82.6～104.4	82.7～106.0
91	Propanil	81.8～107.2	82.6～107.2	80.2～102.2	80.0～107.5	81.2～107.6
92	Propaphos	73.0～100.9	75.8～97.2	74.9～97.8	76.4～97.6	71.5～99.2
93	Propham	84.3～109.0	81.9～105.0	85.6～107.6	81.9～104.3	85.2～104.2
94	Pyrazophos	70.4～100.0	77.4～107.7	70.2～99.8	75.2～99.8	71.7～98.4
95	Pyriproxyfen	74.6～98.6	80.9～104.7	71.9～97.6	72.9～99.6	73.0～101.0
96	Pyroquilon	84.7～106.5	80.4～105.9	86.2～109.1	85.1～106.6	82.1～103.4
97	Quintozene	81.0～102.5	79.1～101.9	78.5～102.7	82.6～102.2	80.3～102.8
98	Quizalofop-ethyl	80.6～104.4	81.1～100.9	78.7～103.0	80.6～101.2	83.5～105.9

Table E. 1（continue）

NO	Compound name	Range of recovery/%（spiked level is 0.01 mg/kg～0.1 mg/kg）				
		Rice	Brown rice	Wheat	Barley	Corn
99	Simeconazole	80.1～106.0	82.6～105.8	80.6～104.9	81.2～104.7	80.6～103.1
100	Sulprofos	86.1～106.5	83.3～99.2	82.6～109.1	87.1～108.1	84.9～105.1
101	Tebuthiuron	80.7～107.3	81.7～105.8	80.2～105.6	83.4～106.4	81.5～107.8
102	Tecnazene	83.0～97.8	81.2～103.0	78.5～100.9	78.5～101.5	81.3～102.1
103	Tolfenpyrad	86.5～108.2	71.3～100.3	87.7～104.3	87.9～104.4	87.6～107.2
104	Triadimefon	81.3～99.8	80.0～97.3	81.9～98.2	80.2～99.9	80.6～99.4
105	Triadimenol	80.8～99.4	82.2～101.7	80.2～96.9	83.2～97.9	80.2～98.8
106	Triallate	85.4～111.6	83.3～107.9	86.4～110.1	85.2～110.0	87.9～108.1
107	Tribuphos	80.6～101.6	82.1～100.5	80.7～101.9	79.9～102.3	79.2～102.1
108	Tricyclazole	83.4～99.5	84.0～102.5	81.0～99.8	81.5～99.9	80.8～98.3
109	XMC	82.8～101.1	80.1～105.7	80.5～99.9	81.5～101.4	81.9～101.4
110	Zoxamide	89.4～102.1	80.2～106.0	88.1～106.4	85.9～106.6	86.0～108.3