



作物中残留農薬の迅速一斉分析法

— GC/MS編 —

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AiSTI SCIENCE

目的

- ・ 農薬数255種における一律基準0.01ppmでの添加回収試験を行い、本手法を評価する。
- ・ 本手法では異常回収率の対策としてPEG共注入を取り入れているが、その効果を評価する。
- ・ SCANとSIMを組み合わせたOne-Injectionによる農薬数255種の一斉分析の検討を行う。

対象農薬

ポジティブリスト制 GC/MS対象農薬

製品名		農薬数	濃度	製品番号
農薬混合標準液	22	50種	10ppm	34021-96
農薬混合標準液	31	85種	10ppm	34030-96
農薬混合標準液	34	46種	10ppm	34033-96
農薬混合標準液	48	61種	10ppm	34049-96
農薬混合標準液	1111	13種	10ppm	49832-90

合計 255種

*いずれも関東化学社製

GC/MS条件

PTV Injector	LVI-S200(AiSTI Science); Stomach Insert
Injector Temp.	70°C-120°C/min-240°C(1min)-50°C/min-270°C(30min)
Auto Samplor	Agilent 7683; 50 µL Syringe
Injection Volume	25 µL
GC/MS	JMS-Q1000GC(JEOL)
Pre-column	Deactivated silica capillary tube, 0.25mm×0.3m
Column	ENV-5MS, 0.25mm i.d.×30m, df: 0.25µm
Column Oven Temp.	60°C(4min)-20°C/min-160°C-5°C/min-220°C-3°C/min-235°C-7°C/min-310°C(8min)
Inlet Mode	Solvent Vent Mode
Vent Flow	150 mL/min
Vent Press	70 kPa
Vent End Time	0.27 min
Purge Flow	50 mL/min
Purge Time	4 min
Gas Saver Flow	20 mL/min
Gas Saver Time	6 min
Detector Temp.	280°C
MS Method	SCAN; 50 - 450 m/z, SIM

胃袋型インサート



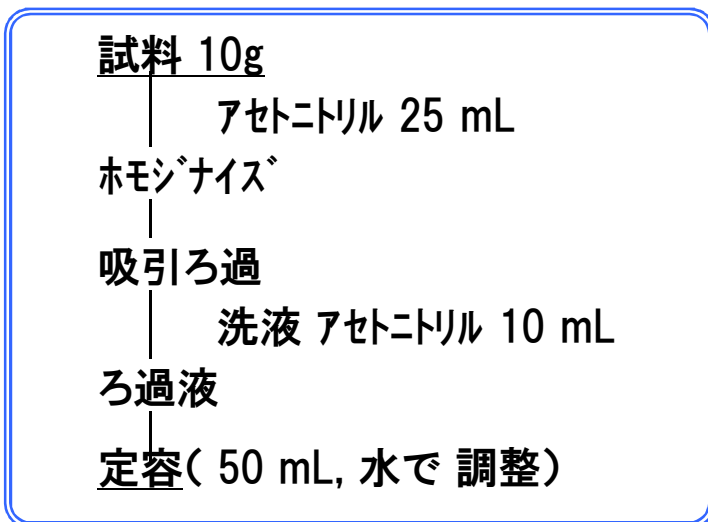
従来の
インサート



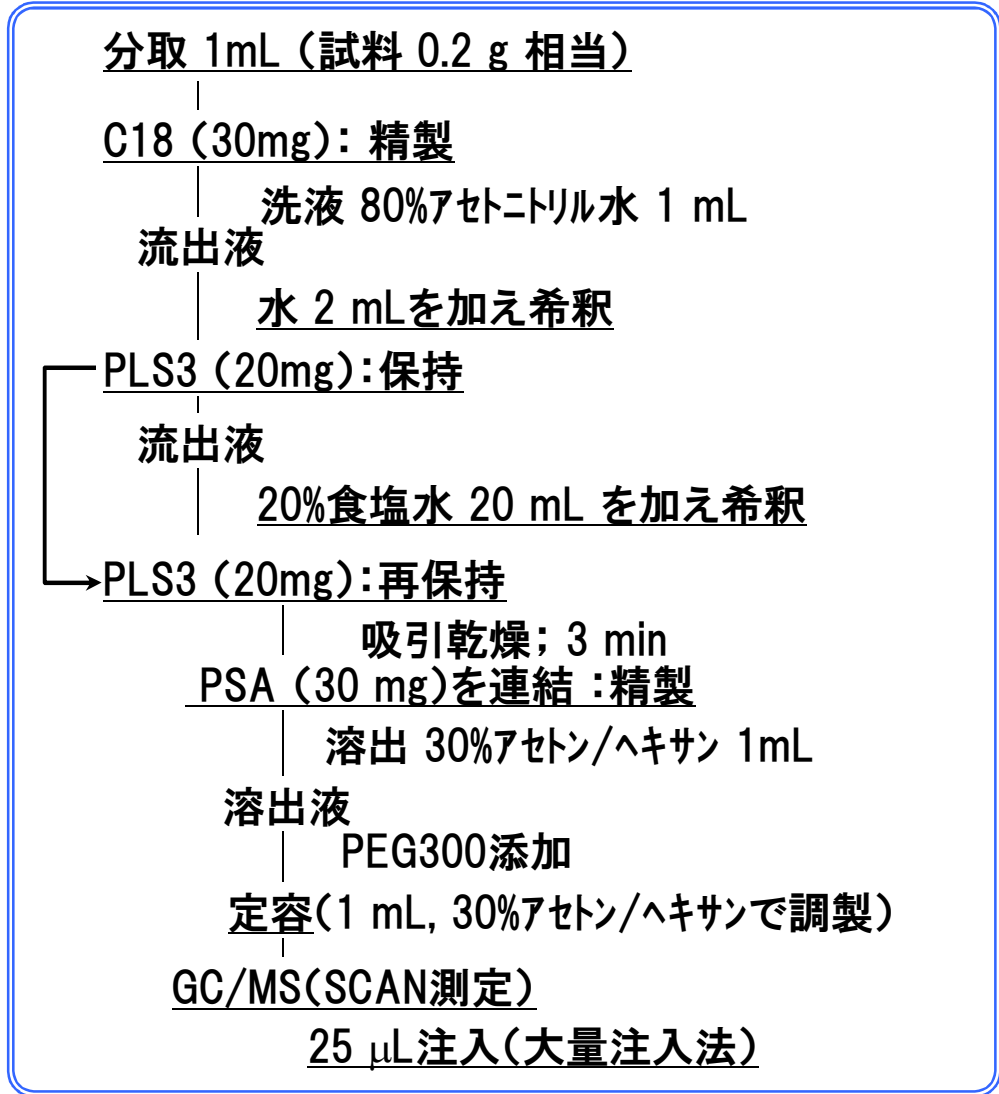
前処理フロー

前処理時間 15分/検体 60分/10検体

抽出法



分液ロートによる液液分配や
エバポレーターなどによる濃縮操作を省
略化



1. 抽出・ろ過

試料 10g

アセトニトリル 25 mL

ホモジナイズ

吸引ろ過

洗液 アセトニトリル 10 mL

ろ過液

定容

(ろ過液に水を加え 50 mL に定容)



吸引ろ過



定容

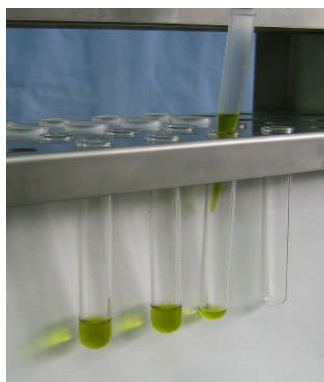
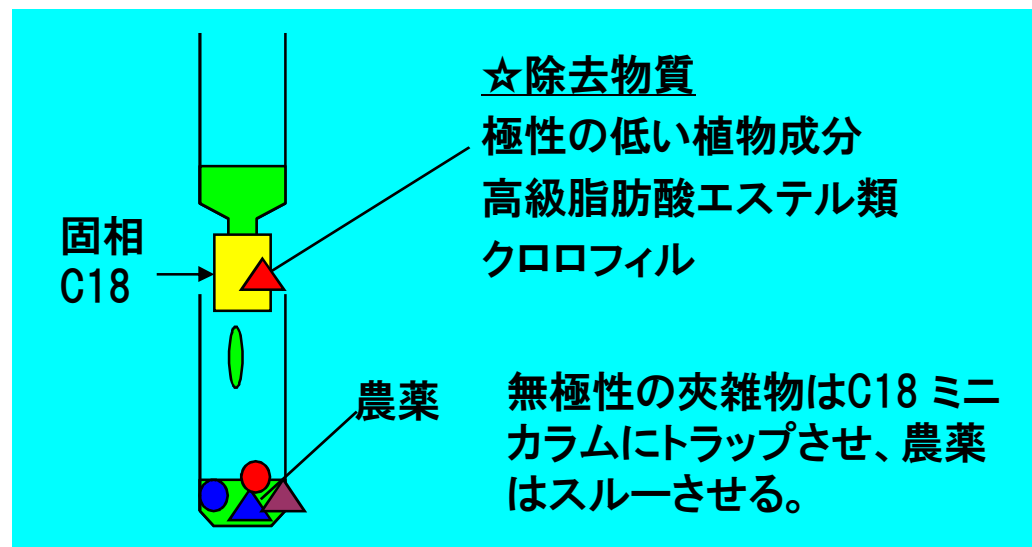
2. 分取&精製

分取 1mL (試料 0.2 g 相当)

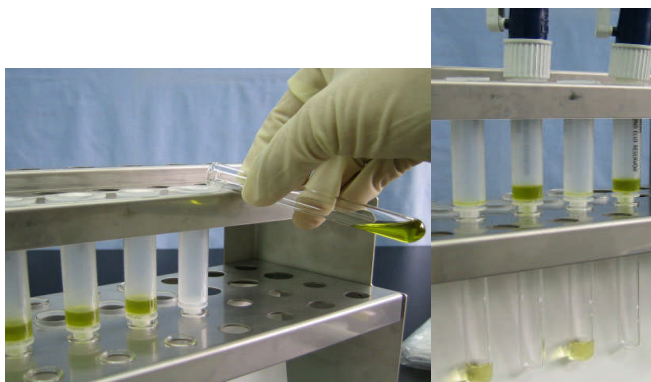
C18-30mg (精製)

洗液80%アセトニリル水 1 mL

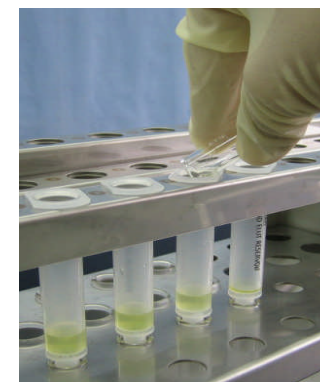
流出液



1 mL分取



C18 30mgに通液



洗液 1mLを通液

3. 保持 & 乾燥

流出液 (2 mL ; 75%)

水 2 mL を加え希釈 (約37%)

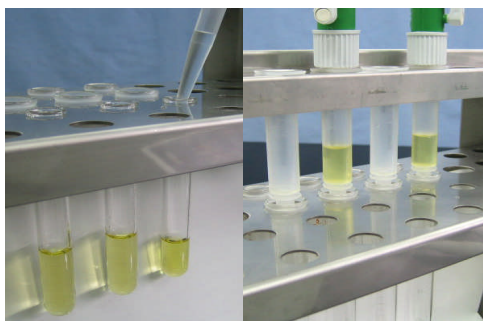
PLS3-20mg (保持)

流出液

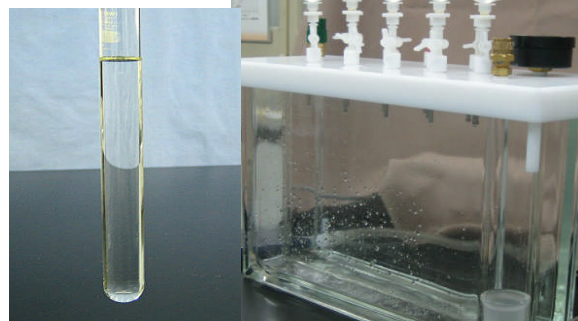
20%食塩水 20 mL を加え希釈 (6%)

PLS3-20mg (再保持)

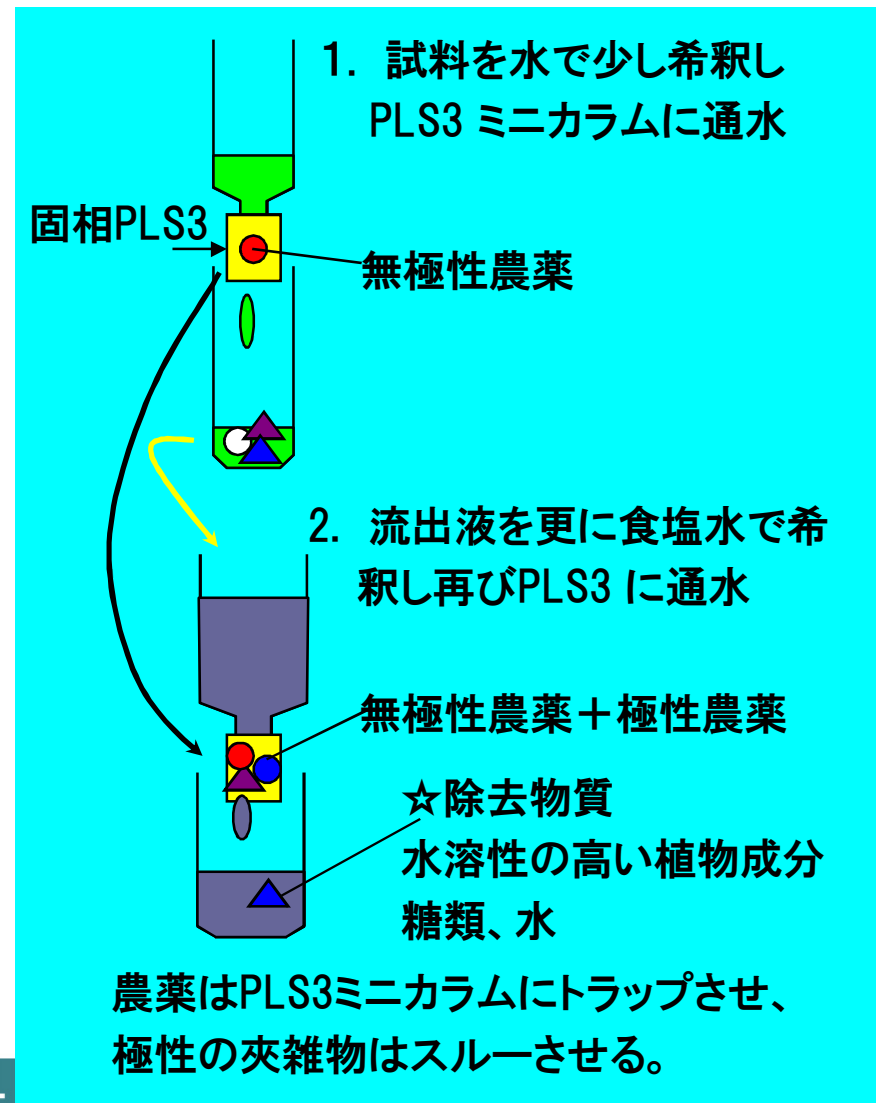
吸引乾燥 2min



水2mLを加えPLS3に保持



食塩水を加え再びPLS3に保持



4. 溶出 & 精製

連結 固相 PSA 30 mg (精製)

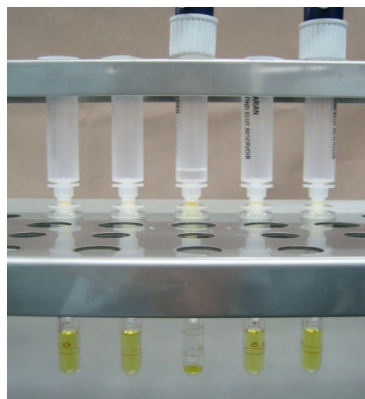
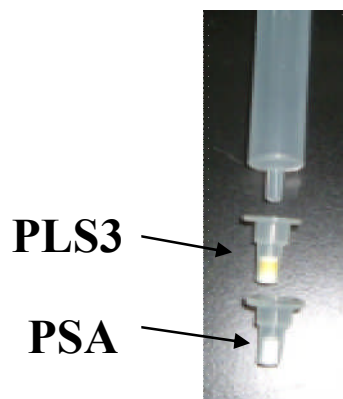
溶出 30%アセトン/ヘキサン 1mL

溶出液

0.2%PEG(300) 10 μ L 添加

定容 (1mL, 30%アセトン/ヘキサンで調製)

GC/MS(25 μ L 大量注入:SCAN測定)



PLS3に-PSAを連結し30%アセトン/ヘキサンで溶出



定容(1mL)

PSA

$$\text{Si-CH}_2\text{CH}_2\text{CH}_2\text{NCH}_2\text{CH}_2\text{NH}_2$$

H

固相PLS3

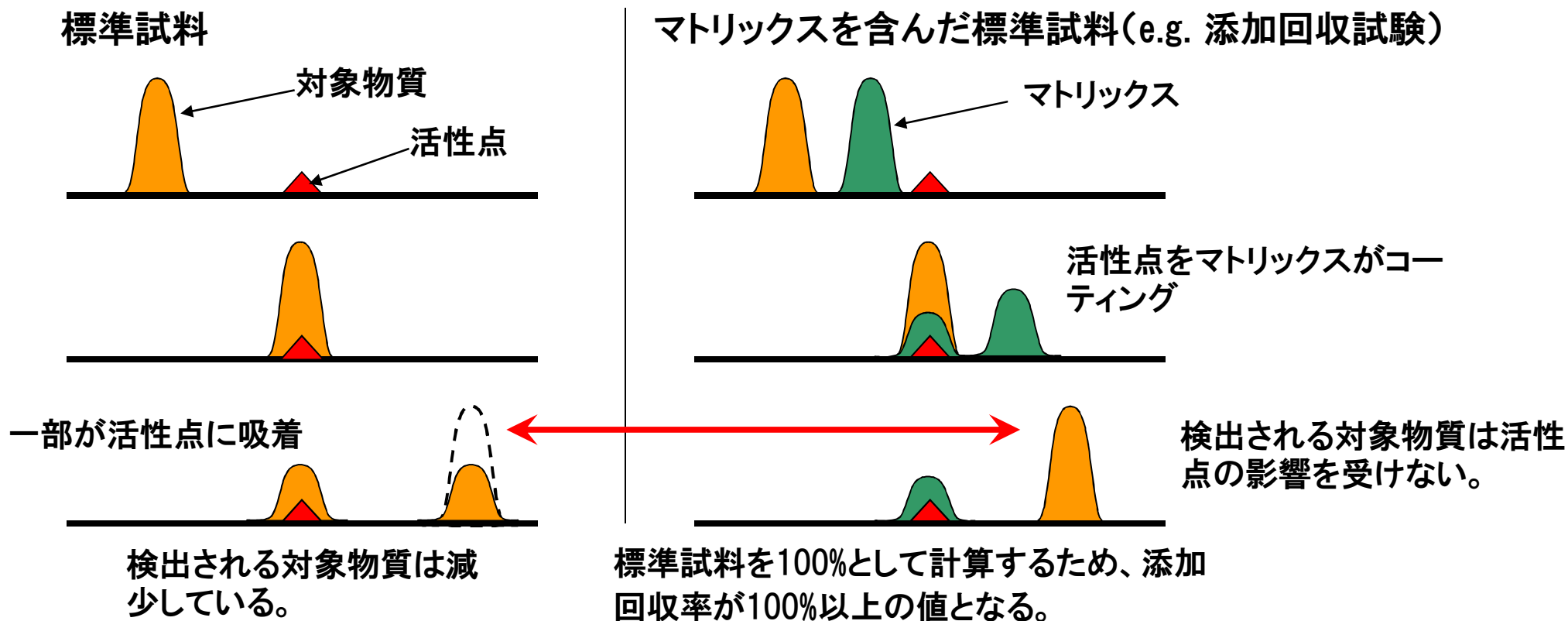
固相PSA

☆除去物質
高級脂肪酸
色素

PLS3ミニカラムから溶出させながら、イオン性の夾雑物は固相PSAミニカラムにトラップさせ、農薬はスルーさせる。

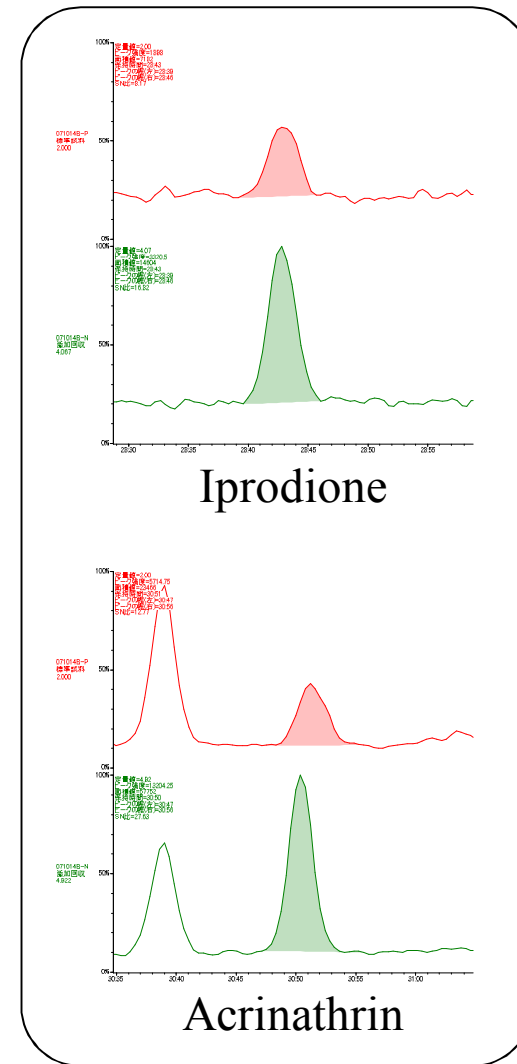
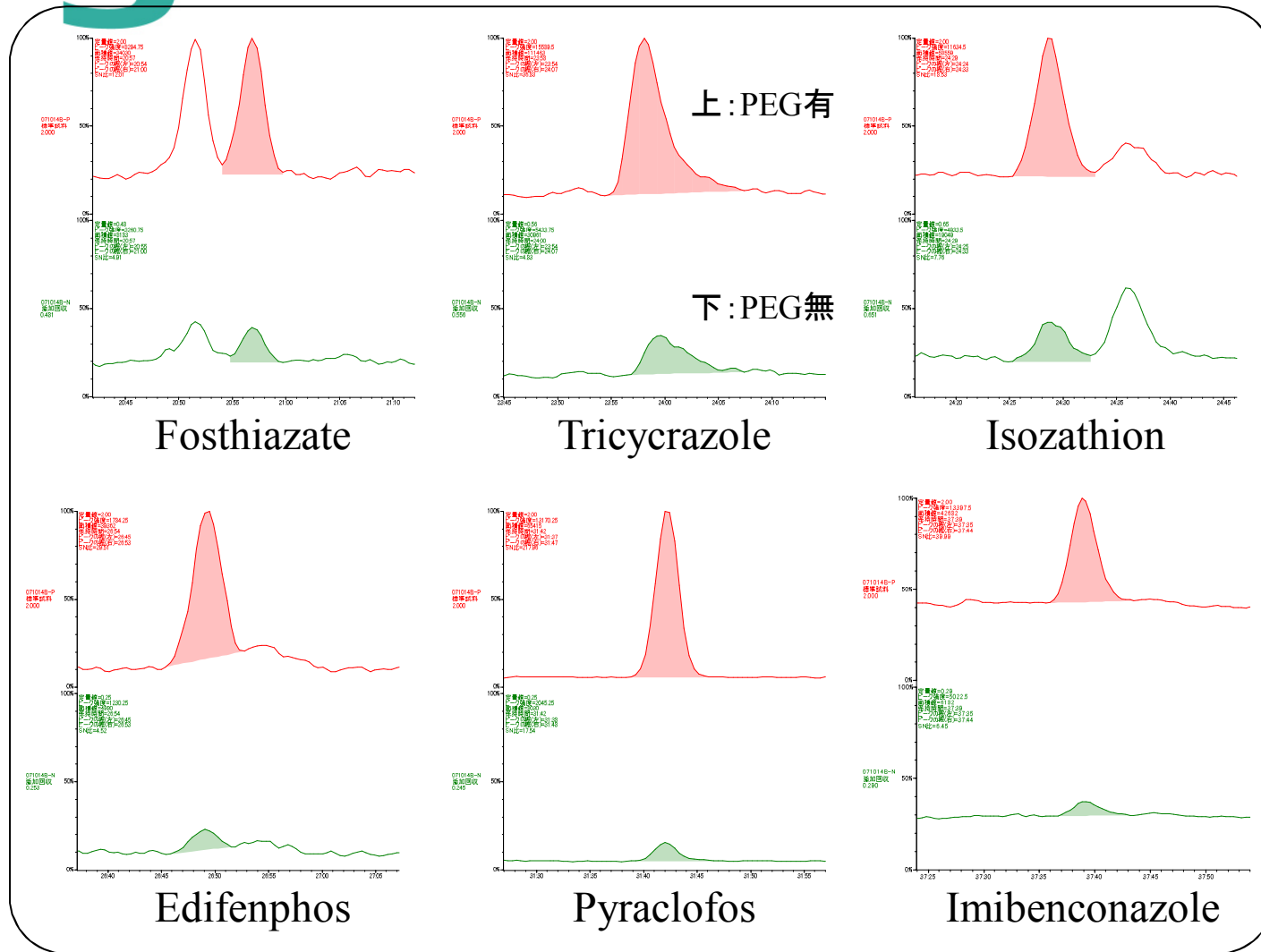
異常回収率の原因は？

この原因として注入口やカラムやイオン化室(MSの場合)などの活性点が異常回収率を引き起こしていると考えられる。

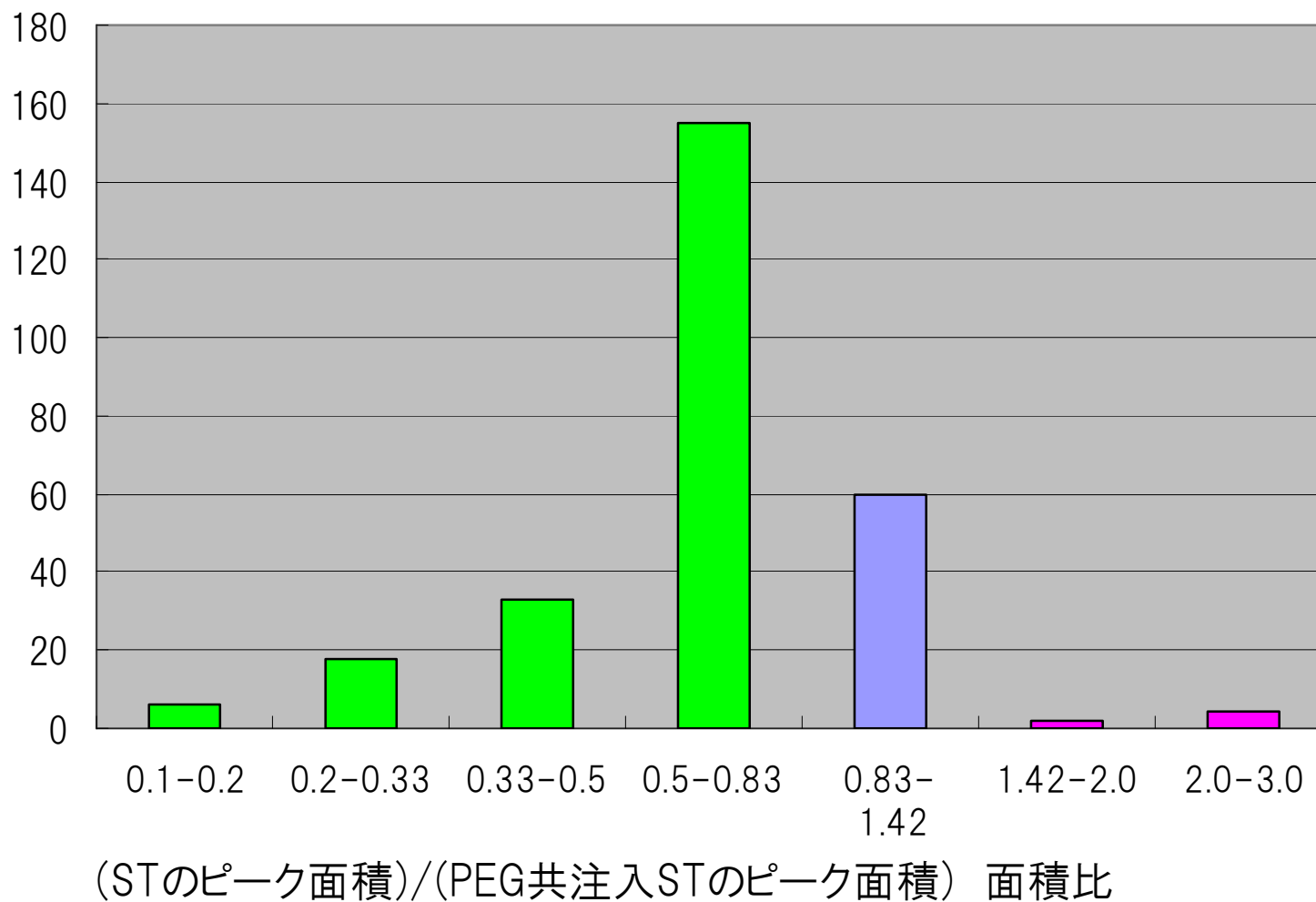


ポジティブ効果

ネガティブ効果

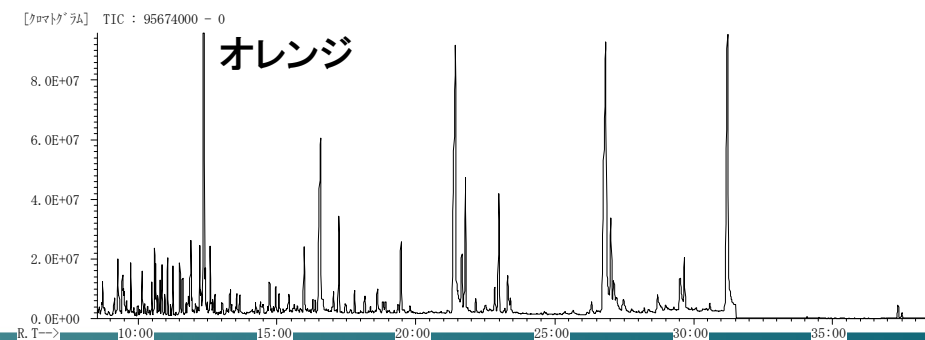
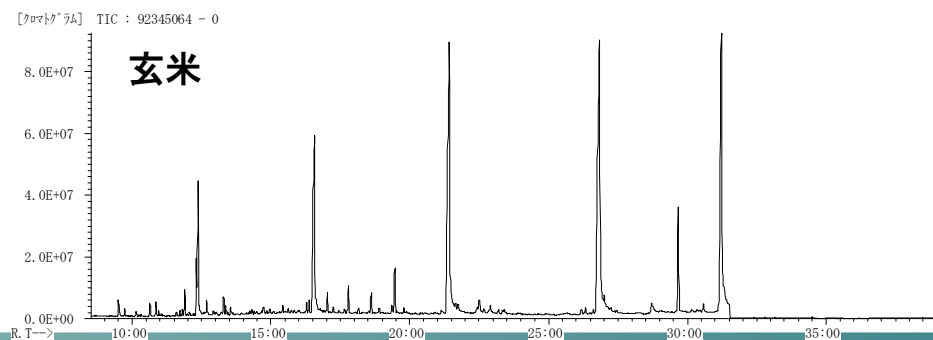
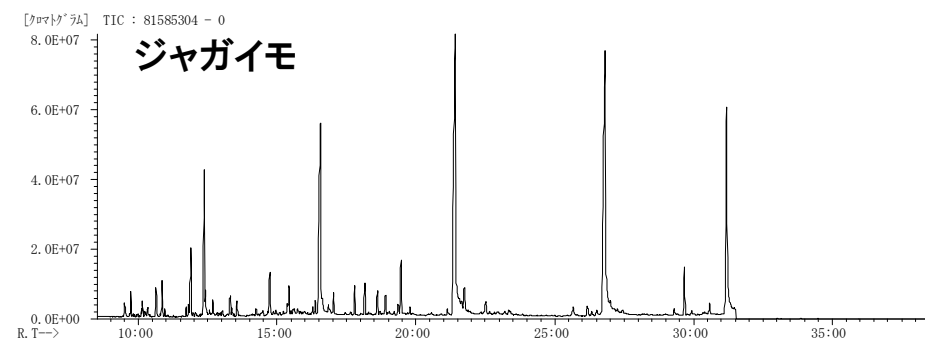
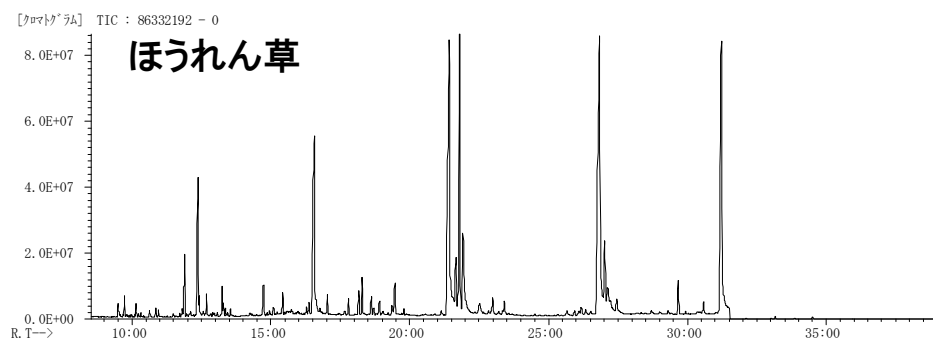
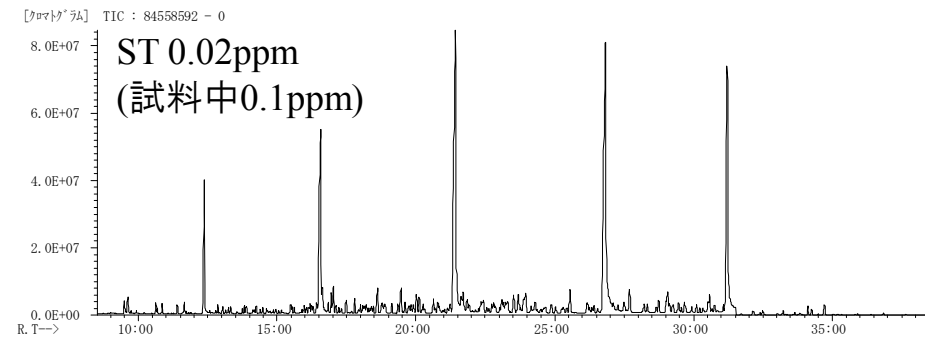
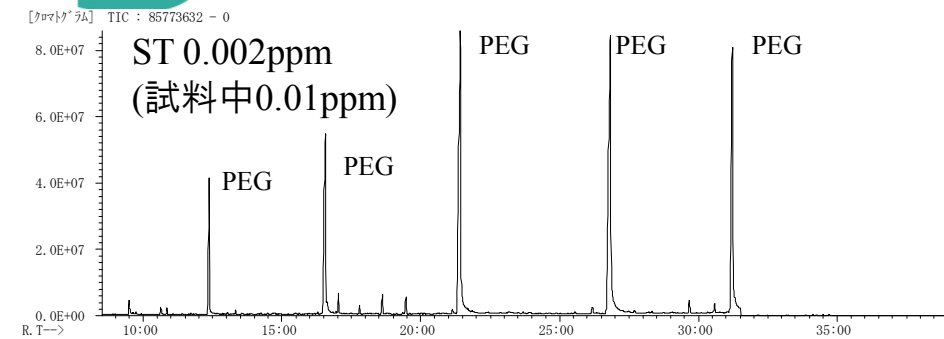


農薬数(成分)



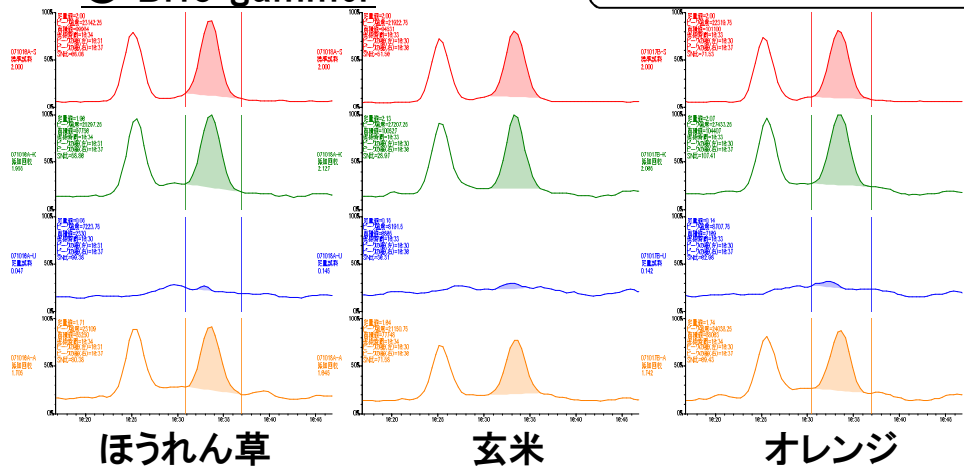
トータルイオンクロマトグラム比較

日本食品衛生学会 第
94回学術講演会

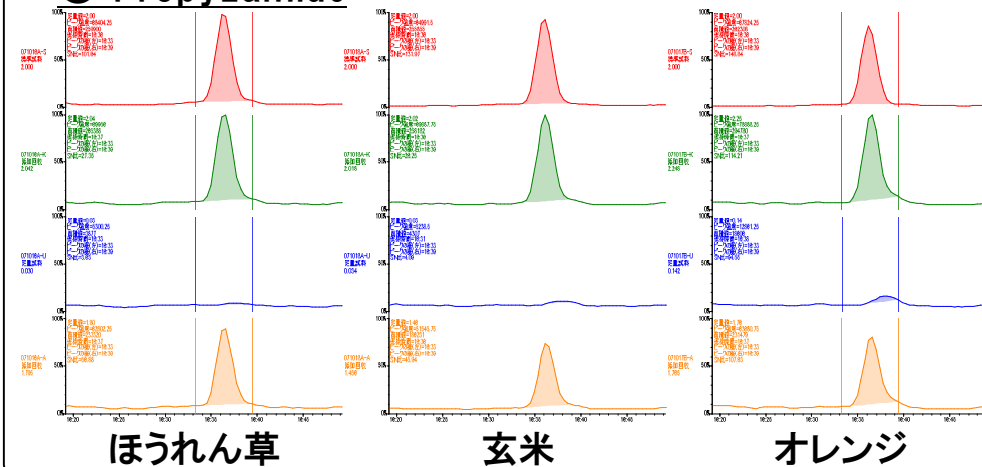


1. スタンダード
2. スパイク
3. 未知試料
4. 添加回収試験

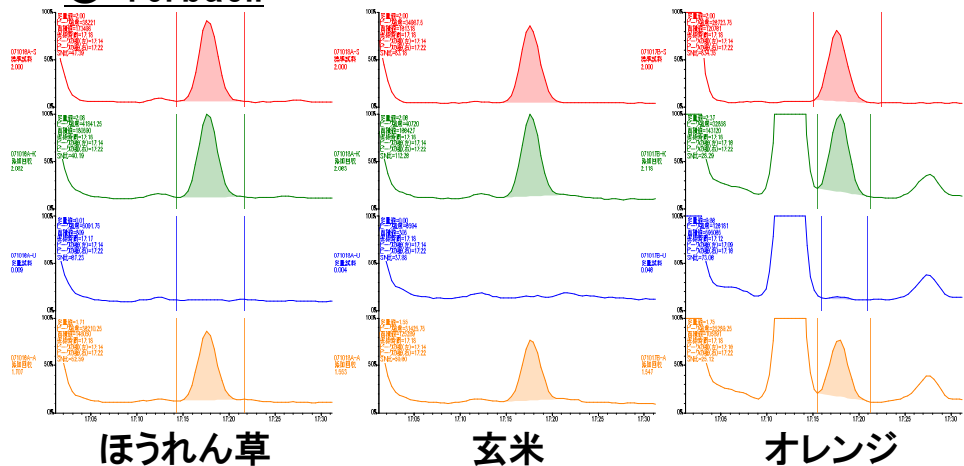
● BHC-gammer



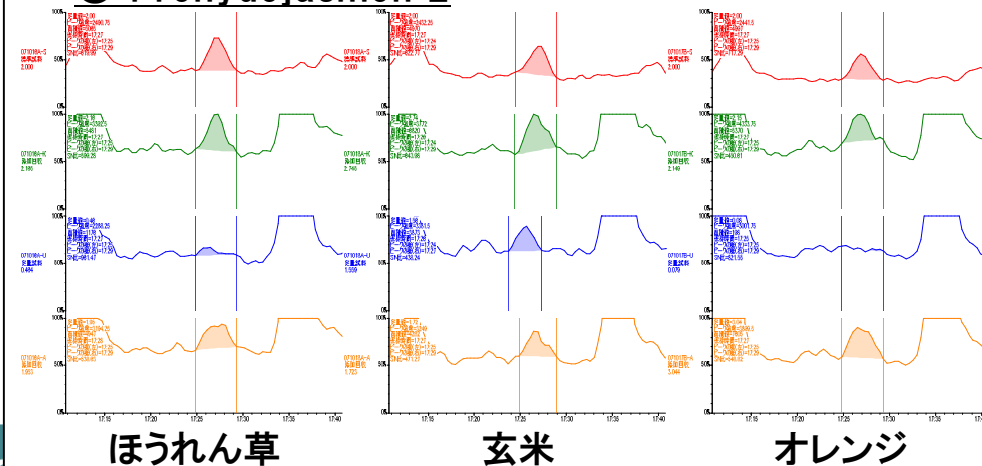
● Propyzamide



● Terbacil



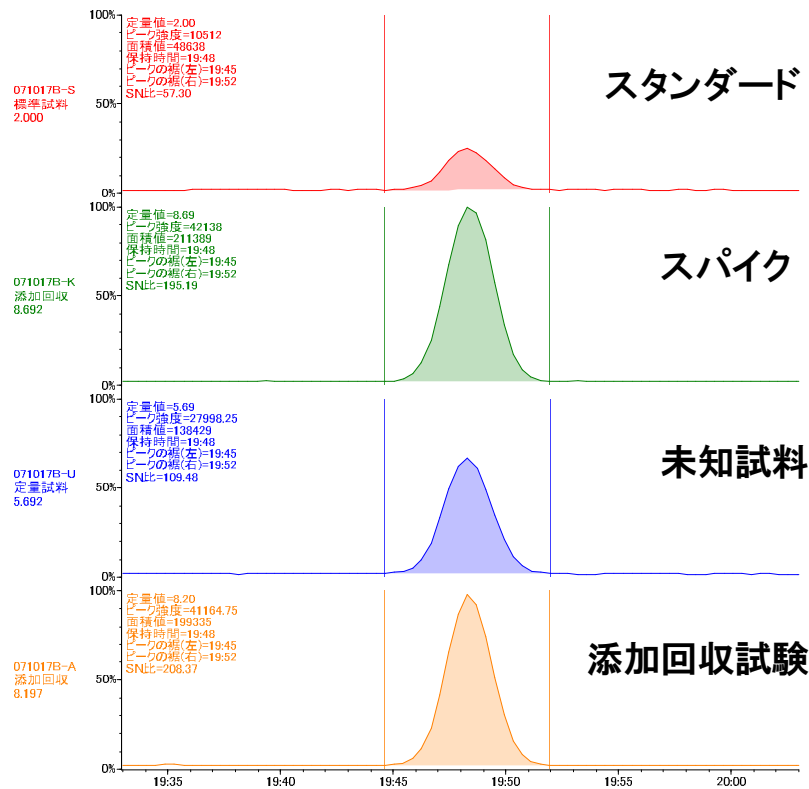
● Prohydrojasmon-2





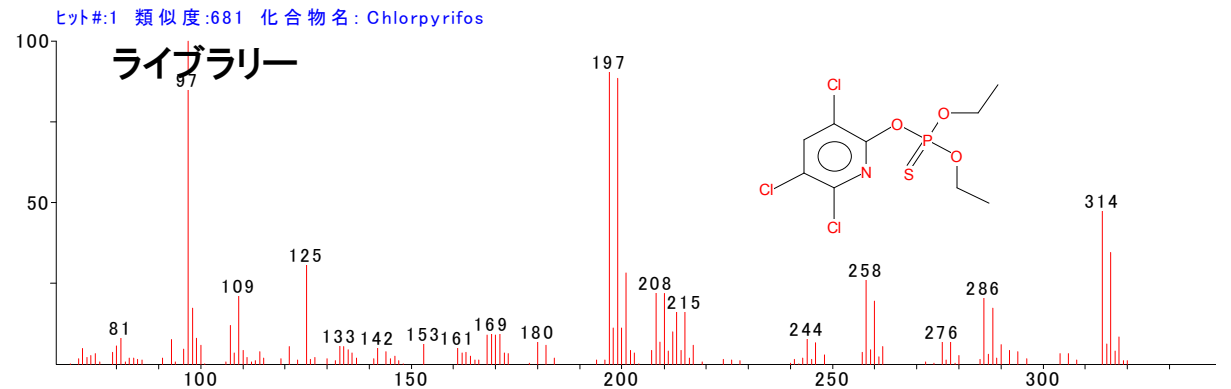
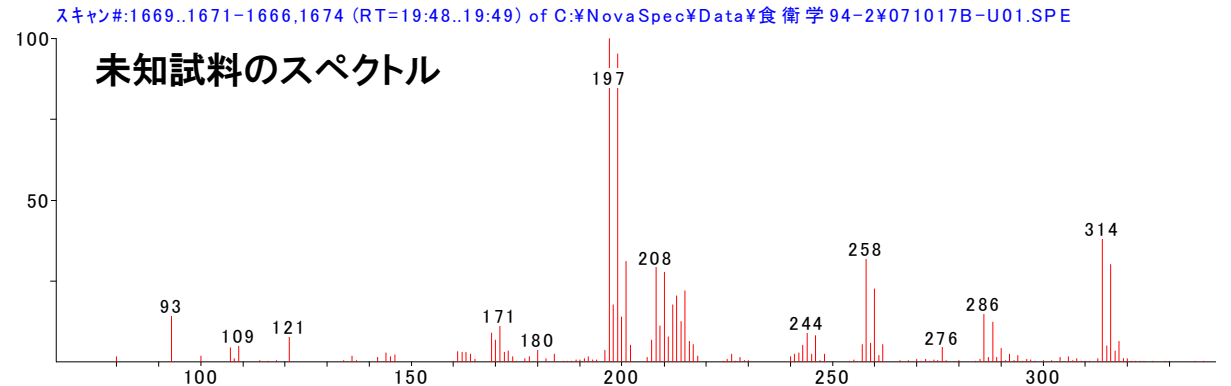
未知試料で検出された時の定性の確認として

■クロマトグラムの定性解析

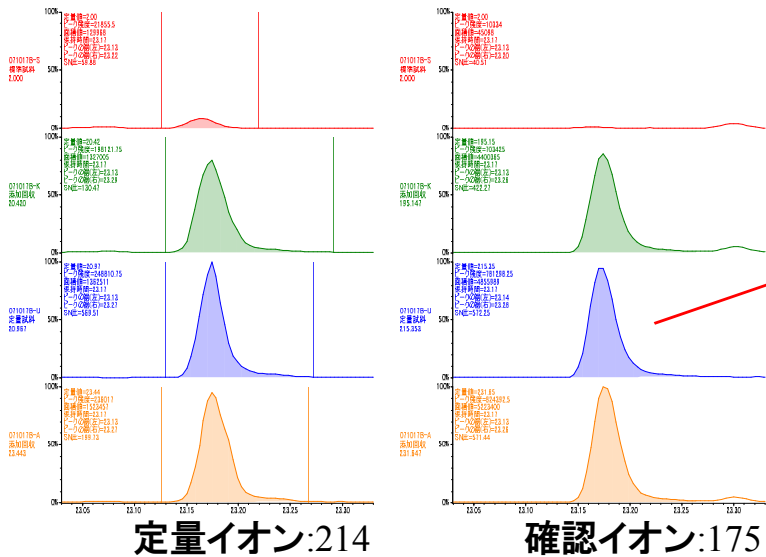


試料: オレンジ
農薬: クロルピリホス

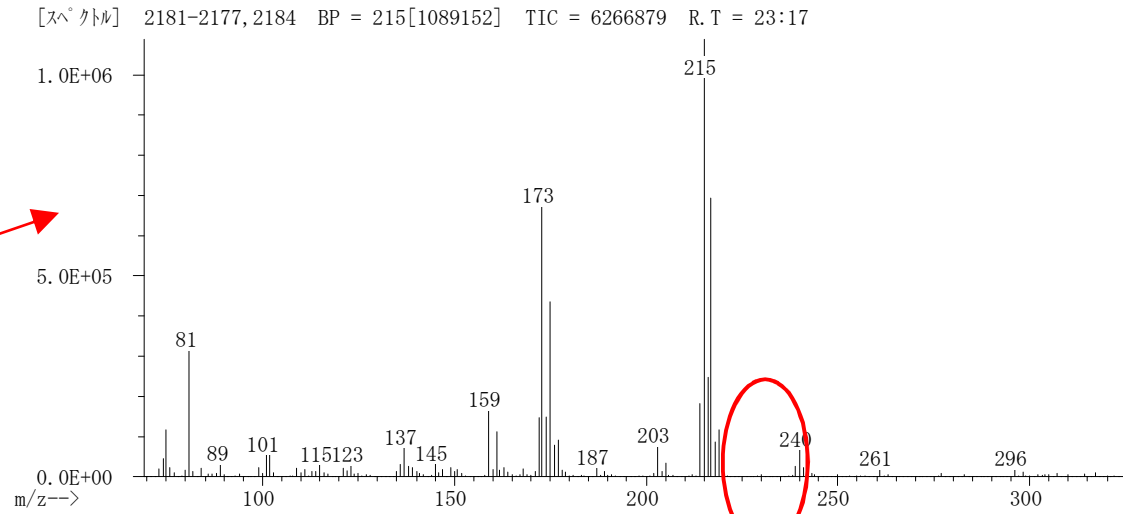
■スペクトルの検索



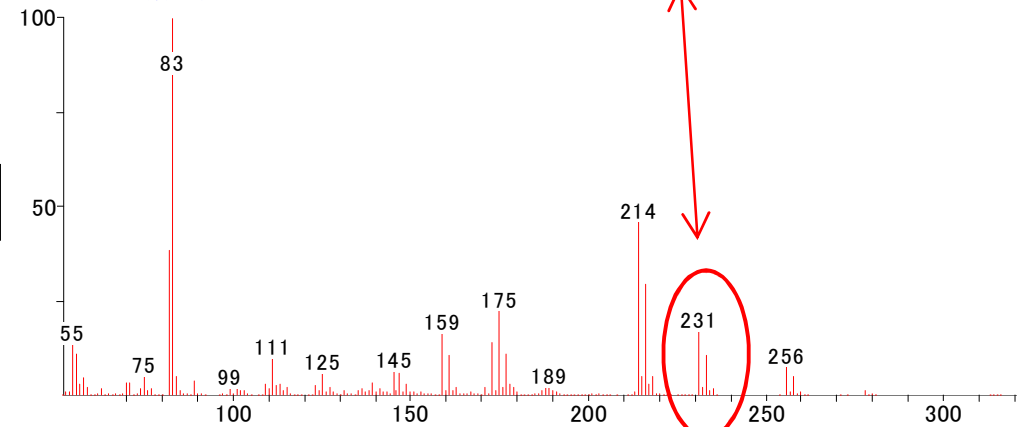
■クロマトグラムの定性解析



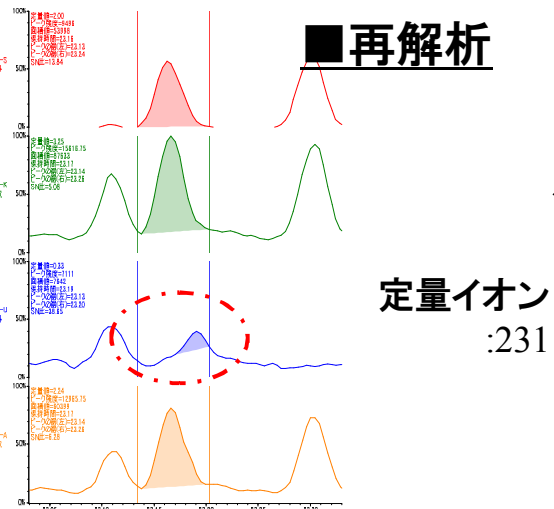
■スペクトルの検索



ヒット#:1 類似度:829 化合物名:Hexaconazole



■再解析

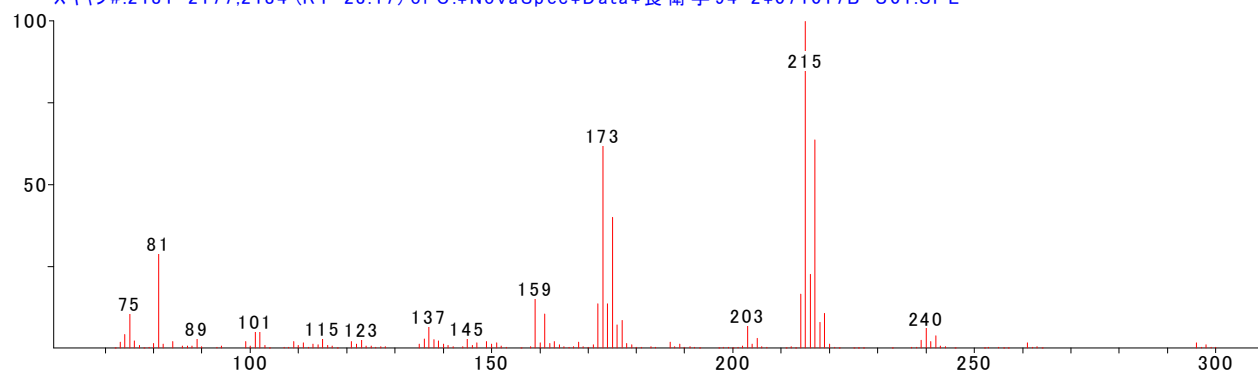


●SIM法の場合は誤認していたかも!?

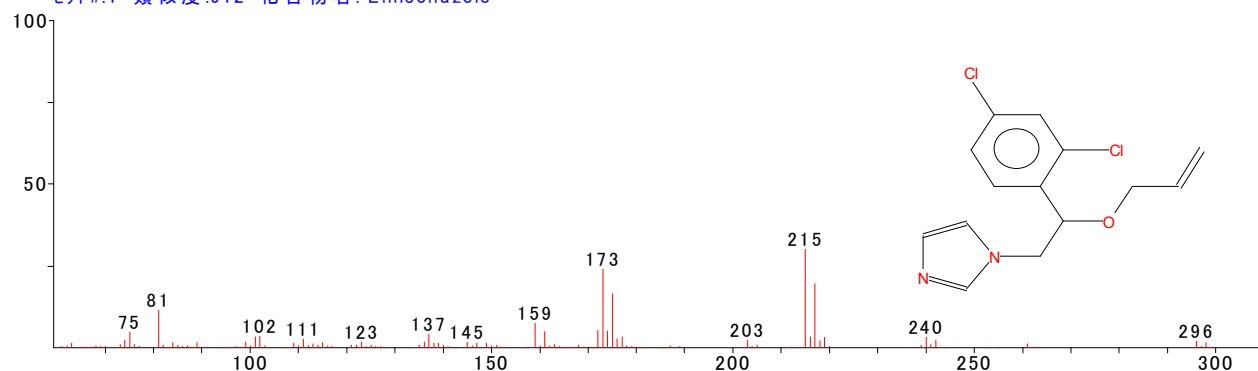


■スペクトルの検索

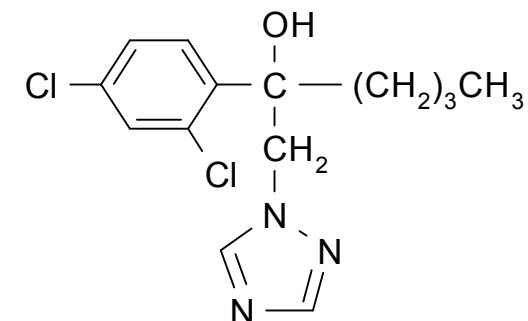
スキャン#:2181-2177,2184 (RT=23:17) of C:\NovaSpec\Data\食衛学94-2\071017B-U01.SPE



ヒット#:1 類似度:912 化合物名: Enilconazole

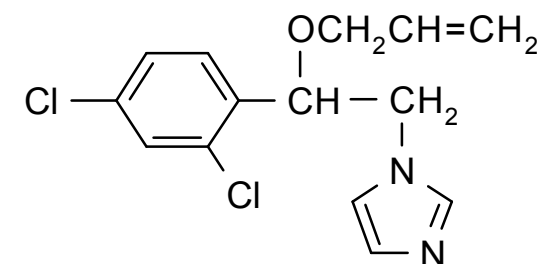


Hexaconazole



基準値: 0.02 ppm

Imazalil



基準値: 5 ppm

おそらくイマザリルが検出されている。ヘキサコナゾールと構造式も似通っているため間違えやすい。しかし基準値が全く異なるため注意が必要である！



添加回収試験①

No.	化合物名	ほうれん草				ジャガイモ		玄米		オレンジ(皮付)		
		0.1ppm(n=4)		0.01ppm(n=5)		0.01ppm(n=3)		0.01ppm(n=3)		0.01ppm(n=3)		
		REC.(%)	RSD(%)	REC.(%)	RSD(%)	REC.(%)	RSD(%)	REC.(%)	RSD(%)	REC.(%)	RSD(%)	
1	Methamidophos	ND	-	ND	-	ND	-	ND	-	ND	-	LogPow=-0.8
2	Dichlorvos	75.1	4.3	77.3	3.1	74.3	0.4	72.8	8.7	82.4	3.1	
3	EPTC	88.7	5.0	92.5	4.6	88.4	2.3	91.7	5.1	99.6	4.1	
4	Mevinphos	49.3	4.9	50.0	2.3	55.8	2.3	44.4	7.1	39.3	0.6	LogPow=0.13
5	Butylate	90.1	4.4	93.5	2.1	87.6	1.7	94.4	7.2	117.5	3.9	
6	Acephate	ND	-	ND	-	ND	-	ND	-	ND	-	LogPow=-0.89
7	Isoprocarbe	90.6	3.0	96.1	2.2	92.9	0.7	85.4	4.3	95.6	1.6	
8	XMC	100.5	3.1	107.7	1.6	96.0	1.4	91.2	5.2	94.1	3.0	
9	Tecnazene	93.0	3.6	96.9	5.5	97.8	3.8	89.6	5.3	104.6	3.4	
10	Fenobucarb	90.6	3.4	96.2	2.3	97.5	0.6	85.0	6.7	98.2	2.5	
11	Propoxur	92.0	3.3	93.6	2.7	97.6	1.8	92.5	4.3	95.2	0.7	
12	Propachlor	89.6	3.6	94.2	1.8	92.3	0.9	86.2	7.8	94.9	1.4	
13	Demeton-S-methyl	55.5	15.0	33.2	8.7	22.8	15.2	13.7	17.5	51.6	2.0	LogPow=1.3
14	Ethalfuralin	90.1	3.5	106.1	7.8	110.2	2.5	101.6	11.8	96.8	6.1	
15	Ethoprophos	93.8	2.9	98.8	2.9	93.0	1.8	86.0	7.7	97.9	2.5	
16	Trifluralin	90.2	4.4	100.6	3.8	105.0	1.4	97.2	4.4	103.4	2.9	
17	Benfluralin	91.7	3.7	99.8	3.5	105.5	1.0	97.9	4.6	104.9	4.2	
18	Chlorpropham	91.9	4.6	92.9	3.5	99.5	0.5	80.5	7.6	97.7	2.2	
19	Bendiocarb	99.3	3.6	91.3	7.4	96.9	4.9	117.4	3.1	105.3	3.5	
20	Cadusafos	92.4	2.2	97.9	7.5	86.3	6.6	89.2	7.5	97.8	1.6	
21	Monocrotophos	4.7	8.6	ND	-	ND	-	ND	-	ND	-	LogPow=-0.22
22	Phorate	74.2	15.7	55.4	1.8	30.0	14.8	36.7	3.8	67.9	3.2	P=S
23	BHC-alpha	93.4	3.7	101.1	3.0	101.7	3.3	84.9	8.4	94.9	3.6	
24	Hexachlorobenzene	78.6	4.2	85.1	2.9	89.4	0.4	79.6	4.5	76.8	3.4	
25	Thiometon	30.4	32.1	14.5	69.4	ND	-	ND	-	ND	-	P=S
26	Dimethoate	51.7	5.3	35.6	16.0	48.5	12.6	50.6	8.5	22.8	34.6	LogPow=0.7
27	Dicloran	91.5	5.8	64.5	6.3	86.5	7.3	65.2	3.3	ND	-	
28	Carbofuran	99.1	4.1	84.4	13.7	99.6	9.0	99.0	5.1	106.5	1.0	
29	Simazine	90.7	2.5	96.4	3.0	96.4	3.4	87.1	3.8	95.0	1.0	
30	Atrazine	92.9	3.3	93.1	1.3	96.7	1.1	88.9	5.3	101.7	1.3	
31	Chlorbufam	93.3	4.6	85.1	2.3	94.4	4.0	79.9	5.3	94.6	2.2	
32	Propazine	91.2	3.6	93.4	1.6	96.2	1.2	84.2	8.2	98.0	3.4	
33	Clomazone	94.3	3.4	99.4	8.2	95.4	4.0	81.7	5.9	91.2	5.0	
34	Quintozen	89.5	3.2	82.7	6.7	45.9	15.7	94.7	7.7	87.0	2.1	
35	Terbufos	83.0	11.6	62.5	3.9	36.9	7.2	46.1	1.8	83.0	4.8	P=S
36	BHC-beta	87.8	4.1	92.3	5.7	99.7	3.5	85.2	3.7	87.9	1.1	
37	Dimethipin	ND	-	ND	-	ND	-	ND	-	ND	-	LogPow=-0.17
38	Diazinon	83.2	2.0	76.2	4.5	68.3	2.2	72.2	7.4	69.6	2.8	P=S
39	BHC-gamma	85.1	3.8	87.0	5.7	87.6	1.5	79.6	6.5	79.1	1.7	
40	Propyzamide	90.2	3.5	89.2	3.4	86.0	1.1	77.0	7.0	81.0	0.6	



添加回収試験②

No.	化合物名	ほうれん草				ジャガイモ		玄米		オレンジ(皮付)	
		0.1ppm(n=4)		0.01ppm(n=5)		0.01ppm(n=3)		0.01ppm(n=3)		0.01ppm(n=3)	
		REC.(%)	RSD(%)	REC.(%)	RSD(%)	REC.(%)	RSD(%)	REC.(%)	RSD(%)	REC.(%)	RSD(%)
41	Cyanophos	92.0	3.8	92.5	3.0	92.7	1.4	79.6	8.9	86.8	2.6
42	Tefluthrine	88.3	3.4	90.5	1.6	92.1	0.8	89.4	2.1	87.6	2.4
43	Prohydrojasmon-1	89.2	2.5	88.2	1.6	84.8	2.8	84.1	7.9	87.0	5.7
44	Pyrimethanil	88.2	4.1	89.0	3.2	90.8	0.7	77.8	6.8	86.0	1.7
45	Pyroquilon	74.5	3.5	65.4	5.4	66.3	1.9	54.7	5.4	58.6	1.4
46	Isazophos	92.3	2.8	93.5	6.2	90.0	2.7	80.2	13.9	88.8	3.5
47	Etrimphos	88.5	3.9	92.1	5.6	91.0	1.7	83.1	6.9	92.3	1.6
48	Triallate	92.7	3.3	93.2	1.1	96.6	0.9	91.0	2.4	96.4	2.1
49	Terbacil	86.7	4.4	86.5	2.4	89.8	0.5	79.8	5.2	84.1	2.0
50	Prohydrojasmon-2	90.1	4.2	ND	-	ND	-	ND	-	ND	-
51	Pirimicarb	87.2	3.8	93.2	1.8	93.7	1.2	83.8	5.0	60.1	3.8
52	Iprobenfos	92.0	2.8	90.1	2.2	93.5	3.9	84.7	9.0	97.9	2.3
53	BHC-delta	96.7	4.2	107.5	10.0	104.8	7.1	113.8	4.7	81.4	13.6
54	Benoxacor	94.1	4.1	97.3	3.7	98.7	0.9	74.4	8.2	99.3	3.4
55	Ethiofencarb	45.6	15.4	24.2	10.1	16.5	19.5	11.3	35.6	35.7	2.5
56	Phosphamidon	88.0	4.7	85.3	3.4	87.9	4.1	88.3	5.8	84.5	4.2
57	Dichlofenthion	90.5	3.1	90.5	3.0	92.7	1.2	89.1	3.8	90.0	1.6
58	Dimethenamid	90.6	3.7	88.5	3.7	91.4	1.5	84.2	9.2	90.9	1.2
59	Benfuresate	89.7	3.6	91.5	2.1	97.3	1.9	81.7	9.0	87.7	1.7
60	Acetochlor	96.2	3.3	93.4	3.3	97.7	2.5	84.2	6.8	95.0	4.7
61	Spiroxamin-1	80.8	2.0	67.3	3.9	74.2	4.0	ND	-	51.2	6.2
62	Bromobutide	84.1	3.9	84.3	5.1	98.4	1.7	85.0	10.3	91.0	3.3
63	Chlorpyrifos-methyl	98.1	3.3	100.7	2.0	99.0	2.4	89.0	7.5	95.5	0.7
64	Propanil	92.3	3.9	90.2	4.8	100.0	2.9	79.3	6.4	96.9	1.0
65	Vinclozoline	93.0	3.7	92.5	3.2	98.6	0.6	85.6	9.2	101.6	2.1
66	Alachlor	91.3	3.6	96.4	3.7	96.1	0.2	84.2	8.2	92.9	2.3
67	Tolclofos-methyl	93.5	3.2	96.3	3.2	97.3	1.9	90.3	7.1	92.6	0.2
68	Methyl-parathion	88.9	5.0	46.6	10.1	114.5	12.5	46.2	4.2	27.0	5.1
69	Metalaxyl	90.0	3.2	90.5	2.8	96.8	1.5	87.5	4.1	96.2	1.2
70	Simetryn	89.9	3.6	92.1	3.4	94.3	0.2	84.7	3.7	90.2	1.9
71	Heptachlor	91.1	5.3	90.4	4.0	86.4	5.3	94.5	6.5	79.5	3.0
72	Ametryn	92.6	3.6	93.8	2.1	94.8	1.2	84.3	7.8	95.8	0.4
73	Prometryn	93.4	3.4	97.1	4.6	97.6	0.9	85.9	6.5	97.6	0.6
74	Carbaril	104.6	6.9	95.7	15.6	149.1	9.3	180.6	5.5	120.0	5.6
75	Spiroxamin-2	86.1	3.1	80.8	3.3	78.9	1.5	ND	-	52.9	2.9
76	Pirimiphos-methyl	90.4	3.4	88.0	4.2	91.1	4.0	84.1	8.6	95.7	4.1
77	Terbutryn	95.3	3.5	85.8	4.5	102.7	4.5	86.2	5.9	99.9	1.3
78	Phenitrothion	93.6	4.3	85.6	3.7	110.0	8.0	82.6	9.8	85.8	4.9
79	Ethofumesate	95.1	3.2	90.8	4.9	97.0	3.6	77.1	9.7	80.9	2.1
80	Methiocarb	103.0	4.7	83.4	12.1	115.9	11.0	121.0	1.6	106.9	5.5

LogPow=1.6

低感度

LogPow=2



添加回収試験③

No.	化合物名	ほうれん草				ジャガイモ		玄米		オレンジ(皮付)		
		0.1ppm(n=4)		0.01ppm(n=5)		0.01ppm(n=3)		0.01ppm(n=3)		0.01ppm(n=3)		
		REC.(%)	RSD(%)	REC.(%)	RSD(%)	REC.(%)	RSD(%)	REC.(%)	RSD(%)	REC.(%)	RSD(%)	
81	Malathion	95.3	3.4	92.1	3.5	104.8	1.9	90.6	7.1	97.8	0.5	
82	Esprocarb	92.5	3.0	92.4	2.3	103.2	1.8	92.6	4.0	97.4	1.4	
83	Dichlofluanid	81.8	34.6	29.5	17.1	60.3	15.4	49.5	22.4	88.2	14.1	
84	Bromacil	92.0	4.0	84.9	4.6	101.9	3.2	87.7	2.2	88.6	2.6	
85	Metolachlor	95.3	3.0	96.1	2.8	106.2	1.7	90.8	6.2	98.9	0.2	
86	Chlorpyrifos	91.9	3.0	92.7	4.1	105.3	3.2	90.6	5.6	127.8	0.6	
87	Fenpropemorph	80.2	4.6	80.5	6.7	84.5	2.2	19.1	24.2	88.0	1.5	
88	Diethofencarb	95.8	3.7	91.5	3.4	102.9	3.5	87.7	6.5	72.3	4.5	
89	Dimethylvinphos	89.7	3.7	92.0	4.5	98.0	4.2	91.0	6.3	99.5	2.1	
90	DCPA	90.4	3.3	92.6	2.6	96.7	1.0	86.2	7.7	94.3	0.5	
91	Thiobencarb	90.8	3.9	94.1	3.1	101.3	2.5	88.3	6.8	94.5	0.4	
92	Aldrin	84.5	3.2	75.7	1.9	83.8	4.5	83.7	3.2	81.2	8.1	
93	Fenthion	72.6	13.9	53.9	2.6	32.7	4.7	39.5	5.3	65.3	1.9	P=S
94	Isofenphos-oxon	95.1	3.4	92.0	4.9	109.9	0.7	94.5	3.8	104.3	0.2	
95	Quinoclamine	84.6	3.2	91.0	5.5	93.5	4.4	90.2	2.4	98.1	5.8	
96	Parathion	87.3	3.6	86.8	5.4	94.9	3.0	81.9	9.0	96.0	0.2	
97	Cyanazine	89.8	3.4	88.4	4.5	96.1	2.0	90.0	3.1	90.5	2.7	
98	Triadimefon	93.5	3.5	90.9	4.8	100.0	2.8	82.7	8.6	100.9	3.1	
99	Nitrothal-isopropyl	93.2	3.9	85.0	3.9	96.4	0.9	95.7	6.4	93.6	1.6	
100	Dichlorobenzophenone-4,4	91.3	3.4	83.0	4.4	100.3	2.6	83.9	5.8	90.9	2.1	
101	Bromofos-methyl	92.6	3.2	94.4	3.4	94.9	1.7	93.2	6.1	96.7	1.2	
102	Diphenamide	94.0	3.0	93.8	3.8	101.3	1.6	86.5	3.1	95.8	1.9	
103	Phthalide	93.6	3.9	93.9	4.5	100.8	4.1	83.9	8.7	94.3	1.1	
104	Fosthiazate-1	93.6	5.1	99.2	3.6	98.0	2.8	88.4	5.2	106.0	2.4	
105	Fosthiazate-2	94.5	5.0	99.1	5.4	100.3	4.6	96.7	5.1	104.2	4.6	
106	Chlorfenvinphos-E	93.5	2.1	99.1	5.4	65.8	7.4	70.1	9.6	82.8	11.6	
107	Pendimethalin	91.6	3.9	91.4	6.7	103.6	3.6	94.2	8.4	98.0	1.2	
108	Fipronil	91.3	5.8	91.6	5.1	100.4	0.9	82.0	11.7	92.9	1.7	
109	Allethrin-1,2	98.2	8.5	ND	-	ND	-	ND	-	ND	-	低感度
110	Isofenphos	91.7	4.1	88.4	5.3	84.6	13.9	79.5	11.1	84.5	1.7	
111	Dimethametryn	90.2	3.1	90.1	3.2	91.9	1.4	79.5	8.3	89.4	1.2	
112	Penconazole	87.5	3.0	88.4	2.8	90.2	1.8	78.7	6.7	84.3	2.2	
113	Chlorfenvinphos-Z	86.7	2.9	77.5	3.4	74.1	3.1	65.1	7.7	77.0	3.7	
114	Chlordane-oxy	88.1	4.0	81.6	2.6	87.3	1.5	81.8	5.0	79.9	3.1	
115	Allethrin-3,4	85.9	2.9	72.7	3.5	70.8	1.2	69.8	3.3	65.0	5.1	
116	Pyrifenox-Z	87.3	3.8	86.7	4.2	94.1	2.5	79.8	2.9	80.1	1.4	
117	Heptachlor-epoxide-cis	88.9	3.5	89.9	2.7	84.4	0.4	79.4	4.1	85.1	1.3	
118	Phenthoate	70.8	17.1	77.6	6.7	91.1	4.6	75.1	9.6	79.8	3.6	
119	Diclocymet-1	91.0	3.7	90.9	4.6	94.6	4.7	76.2	6.8	79.1	7.4	
120	Methoprene	42.6	2.6	ND	-	58.6	10.4	64.6	8.0	ND	-	低感度



添加回収試験④

No.	化合物名	ほうれん草				ジャガイモ		玄米		オレンジ(皮付)	
		0.1ppm(n=4)		0.01ppm(n=5)		0.01ppm(n=3)		0.01ppm(n=3)		0.01ppm(n=3)	
		REC.(%)	RSD(%)	REC.(%)	RSD(%)	REC.(%)	RSD(%)	REC.(%)	RSD(%)	REC.(%)	RSD(%)
121	Quinalphos	89.3	2.7	83.0	7.8	94.8	9.5	67.4	15.6	82.8	3.2
122	Heptachlor-epoxide-trans	86.6	4.9	89.2	8.4	96.9	12.5	84.3	12.2	68.2	6.3
123	Procymidone	91.3	2.9	94.6	2.9	89.7	2.9	78.2	7.0	82.8	1.4
124	Triadimenol-1	76.5	6.5	ND	-	80.6	3.9	90.9	12.6	101.8	1.8
125	Dimepiperate	96.8	3.5	97.5	3.3	92.9	2.3	85.7	8.0	89.9	1.6
126	Captan	87.1	29.2	ND	-	ND	-	ND	-	ND	-
127	Triadimenol-2	95.5	3.7	100.2	5.4	87.1	0.5	76.7	11.6	88.7	6.3
128	Diclocymet-2	87.3	3.6	90.2	4.6	89.6	4.5	76.9	2.4	78.9	8.1
129	Chlordane-trans	89.4	3.6	92.2	0.9	94.1	1.4	88.2	5.7	85.1	1.8
130	Methidathion	94.1	3.8	96.5	3.5	93.0	2.0	82.3	7.4	86.6	1.5
131	Butachlor	90.1	2.5	90.8	4.8	90.3	1.2	84.2	10.8	85.8	4.0
132	Pyrifeno-E	92.1	3.8	95.2	4.7	107.1	12.6	95.9	11.4	99.8	6.3
133	Tetrachlorvinphos	90.2	5.1	92.2	5.0	101.7	4.9	86.5	6.0	96.5	3.0
134	Paclobutrazol	89.3	4.7	94.7	4.6	90.8	1.8	96.7	6.4	91.9	10.0
135	Quinomethionate	63.1	21.5	73.0	8.3	78.7	5.8	75.4	14.8	75.6	6.4
136	Fenothiocarb	96.5	3.0	94.2	4.5	97.8	2.4	88.0	5.6	95.9	3.7
137	Butamifos	91.8	3.1	83.0	6.3	93.8	2.6	83.2	7.8	92.0	2.7
138	Chlordane-cis	90.0	3.5	96.4	1.2	95.1	3.9	86.2	1.8	90.7	2.0
139	Endosulfan-alpha	83.8	3.8	ND	-	ND	-	ND	-	ND	-
140	Fenamiphos	81.3	8.6	68.0	4.7	57.1	3.3	49.0	2.9	81.6	2.0
141	Flutriafol	91.2	2.3	89.1	3.7	94.8	4.3	96.7	2.4	94.2	3.6
142	Napropamide	94.2	3.4	91.2	2.3	93.2	2.2	85.3	6.6	96.9	4.4
143	Flutolanil	91.9	4.1	94.2	4.6	95.4	1.6	83.8	7.3	86.6	3.5
144	Pretilachlor	93.8	3.8	98.2	4.4	96.2	8.1	98.1	10.0	88.6	2.1
145	Prothiofos	90.1	3.2	85.2	4.3	102.8	5.4	91.8	0.7	83.0	0.9
146	Hexaconazol	88.7	3.6	91.2	6.2	91.4	3.0	75.9	3.9	103.4	3.9
147	Metominostrobin-E	93.5	3.4	92.9	4.7	97.6	3.4	89.4	1.7	97.5	3.5
148	Isoxathion-ox	95.6	3.4	ND	-	ND	-	ND	-	ND	-
149	Profenofos	94.1	3.0	99.2	3.1	89.8	3.2	86.7	8.4	105.3	1.0
150	Isoprothiolane	92.2	2.9	93.9	3.3	95.7	1.1	80.3	8.0	96.2	3.4
151	Busan(TCMTB)	94.4	8.5	77.8	22.0	86.8	6.8	94.4	3.2	113.2	10.9
152	Thifluzamide	87.7	9.9	90.5	6.2	96.0	1.0	81.7	9.6	83.4	5.0
153	Fludioxonil	87.6	3.2	88.3	4.5	92.4	5.1	83.1	3.4	87.8	0.9
154	Oxadiazone	95.2	2.8	93.1	4.5	101.0	6.8	89.6	6.3	92.2	0.4
155	DDE-p,p'	84.2	3.5	90.6	3.3	91.8	1.0	84.2	4.0	83.7	0.2
156	DEF(tribuphos)	93.9	3.3	87.2	3.8	91.3	2.5	92.0	1.0	97.5	5.2
157	Flamprop-methyl	91.6	2.9	95.1	3.7	97.4	1.8	83.1	8.2	93.7	1.3
158	Uniconazole	88.8	3.8	91.1	3.5	93.9	0.8	85.6	7.5	97.4	1.1
159	Oxyfluorfen	92.0	3.7	88.7	2.7	100.2	4.6	98.4	7.8	89.2	4.1
160	Bupimate	93.5	1.5	91.5	3.4	94.3	9.9	82.8	4.0	81.9	5.3

低感度・分解

低感度

低感度



添加回収試験⑤

No.	化合物名	ほうれん草				ジャガイモ		玄米		オレンジ(皮付)	
		0.1ppm(n=4)		0.01ppm(n=5)		0.01ppm(n=3)		0.01ppm(n=3)		0.01ppm(n=3)	
		REC.(%)	RSD(%)	REC.(%)	RSD(%)	REC.(%)	RSD(%)	REC.(%)	RSD(%)	REC.(%)	RSD(%)
161	Buprofezin	91.7	3.2	94.2	1.9	94.5	3.8	90.6	9.5	94.7	2.7
162	Flusilazole	90.0	3.6	94.0	4.5	93.0	1.7	83.4	5.4	94.0	2.3
163	Myclobutanil	86.8	3.0	93.8	5.0	94.9	2.1	84.2	5.9	91.9	2.0
164	Kresoxim-methyl	89.1	3.7	94.0	2.3	97.0	1.0	88.3	7.2	93.2	1.5
165	Dieldrin	92.4	2.4	85.7	2.9	100.3	9.0	78.6	7.4	74.3	5.3
166	Tricyclazole	52.9	3.3	48.9	4.5	55.6	6.9	44.5	9.0	47.9	18.2
167	Metaminostrobin-Z	93.7	3.3	94.2	5.4	98.4	2.5	89.8	2.1	96.8	3.9
168	Imazametha-benzmthyl-es	99.0	4.7	96.8	7.0	87.0	11.0	88.3	1.2	86.7	3.6
169	Chlorfenapyr	90.0	3.8	99.5	9.2	89.5	14.2	81.8	8.1	84.6	15.8
170	Carboxin	46.1	15.9	32.6	5.5	27.9	9.8	19.6	5.2	50.5	1.8
171	Azaconazole	90.0	2.8	93.8	3.4	94.8	2.2	91.2	2.9	93.6	1.8
172	Imibenconazole=脱ベンジルイ	77.5	1.6	64.6	4.3	100.9	12.9	101.2	14.3	69.2	16.6
173	Isoxathion	103.3	4.5	94.8	6.8	92.1	8.1	97.1	3.9	137.0	3.8
174	Cyproconazole-1	91.8	3.5	94.0	6.4	98.2	4.3	95.3	3.6	95.6	1.9
175	Fenoxanil	91.2	3.5	92.2	4.8	101.3	1.9	79.5	9.8	100.1	2.6
176	Cyproconazole-2	91.8	3.9	92.6	4.2	102.1	5.1	90.7	4.7	99.4	2.7
177	Endrin	93.4	3.0	84.0	2.2	93.3	1.8	91.1	6.0	90.5	2.6
178	Pyriminobac-methyl-E	90.5	4.0	92.7	4.4	98.9	2.7	85.3	4.7	92.8	1.5
179	Chlorobenzilate	94.2	4.1	98.0	2.7	101.9	3.7	93.9	5.3	100.6	1.3
180	Fensulfothion	94.8	4.3	92.5	6.4	94.6	2.5	94.8	3.4	97.1	5.0
181	Ethion	100.7	3.5	99.0	3.8	105.4	2.3	101.8	6.4	104.4	2.7
182	Endosulfan-beta	89.8	4.0	72.8	2.9	84.5	6.4	80.6	17.4	80.5	3.9
183	DDD-p,p'+DDT-o,p'	87.8	3.9	95.5	2.4	100.9	2.0	94.8	5.0	96.2	1.8
184	Fluacrypyrim	94.1	3.5	95.6	2.8	97.6	4.4	87.4	8.5	100.0	0.8
185	Oxadixyl	83.2	3.4	84.0	5.9	85.7	2.5	83.0	3.9	71.1	3.7
186	Mepronil	87.7	4.0	115.3	11.5	122.2	11.3	32.5	4.6	34.2	5.9
187	Triazophos	94.2	3.6	99.6	7.3	93.6	2.9	81.5	6.9	82.2	5.0
188	Carfentrazone-ethyl	95.1	3.3	86.6	3.3	99.1	1.5	85.9	9.4	102.5	2.4
189	Benalaxyl	94.9	3.2	94.6	3.1	97.5	1.5	86.8	8.2	98.1	1.9
190	Trifloxystrobin	98.3	3.8	98.5	2.5	101.4	0.8	92.7	5.8	101.9	1.4
191	Propiconazole-1	88.2	3.1	82.4	1.9	84.8	1.4	73.5	8.7	74.0	6.6
192	Pyriminobac-methyl-Z	88.3	3.4	81.3	4.1	88.1	3.4	74.7	7.4	80.4	3.5
193	Edifenphos	80.0	5.4	96.2	4.8	95.5	3.2	75.6	10.4	76.0	2.5
194	Norflurazon	88.8	2.9	90.4	15.6	81.9	1.8	76.3	4.2	86.3	5.1
195	Quinoxifen	88.4	3.2	85.3	2.6	88.7	1.5	79.7	5.4	85.3	1.2
196	Pyraflufen-ethyl	89.8	2.8	88.7	3.7	87.6	2.3	79.3	9.3	91.5	2.6
197	Propiconazole-2	95.3	3.1	95.6	2.1	87.5	0.8	71.5	3.3	95.2	2.1
198	DDT-p,p'	93.6	5.3	118.7	5.2	97.3	5.8	81.6	7.5	82.7	0.4
199	Lenacil	91.8	2.5	90.5	5.3	90.5	1.4	85.7	0.5	84.8	2.9
200	Thenylchlor	90.2	5.0	96.6	5.5	82.9	1.2	81.6	2.3	91.8	1.1

Log-Pow=1.4

添加回収試験⑥

No.	化合物名	ほうれん草				ジャガイモ		玄米		オレンジ(皮付)	
		0.1ppm(n=4)		0.01ppm(n=5)		0.01ppm(n=3)		0.01ppm(n=3)		0.01ppm(n=3)	
		REC.(%)	RSD(%)	REC.(%)	RSD(%)	REC.(%)	RSD(%)	REC.(%)	RSD(%)	REC.(%)	RSD(%)
201	Hexazinone	82.3	3.5	89.3	4.4	84.8	1.3	74.4	5.2	81.3	2.2
202	Propargite(BPPS)-1	94.8	2.5	80.4	8.6	90.6	6.2	95.9	6.2	91.1	3.1
203	Propargite(BPPS)-2	89.2	3.5	103.1	3.2	98.8	0.9	95.6	6.3	93.1	8.2
204	Diflufenican	89.4	4.2	90.0	2.2	94.4	2.3	85.9	6.7	92.5	2.2
205	Tebuconazole	88.4	3.3	90.1	4.9	89.2	2.1	79.8	6.9	87.3	1.8
206	Diclofop-methyl	90.5	4.2	92.6	2.9	91.1	1.5	87.6	6.7	92.7	1.5
207	Mefenpyr-diethyl	96.3	3.4	97.6	3.1	96.7	2.5	86.7	7.9	98.3	1.0
208	Zoxamide	92.7	11.9	90.4	13.0	105.7	7.1	98.4	9.4	84.4	2.8
209	Captafol	80.6	7.6	ND	-	ND	-	ND	-	ND	-
210	Pyributicarb	91.9	4.6	92.8	3.4	76.7	2.6	79.4	4.9	91.7	2.0
211	Pyridafenthion	95.6	3.4	96.4	6.1	86.8	0.6	87.5	6.3	107.2	2.3
212	Iprodione	88.4	5.5	91.0	12.7	107.9	2.3	141.7	5.7	73.4	11.5
213	Bifenthrin	85.7	3.4	92.9	1.3	88.2	0.2	94.0	4.5	93.9	2.3
214	Piperophos	94.8	3.3	97.4	4.2	90.8	4.8	92.9	12.0	107.0	2.6
215	Bromopropylate	93.9	4.7	100.2	1.8	95.2	3.0	93.1	5.9	105.1	3.7
216	Picolinafen	91.7	4.9	98.3	1.4	94.9	1.2	94.8	6.8	105.0	0.5
217	EPN	85.8	3.8	92.8	5.2	93.9	3.6	88.2	6.1	81.0	9.1
218	Fosmet	89.3	4.7	97.8	9.9	97.4	3.3	109.7	4.9	101.5	3.0
219	Acetamipride	55.9	3.5	74.9	11.4	65.9	11.3	44.6	28.7	40.8	23.9
220	Etoxadole	90.9	3.6	95.2	2.6	94.9	2.8	93.3	8.2	99.7	3.7
221	Fenpropathrin	92.5	5.0	87.0	4.6	94.7	11.3	84.3	3.5	83.2	2.0
222	Methoxychlor	96.5	6.3	126.8	4.4	97.4	4.1	86.7	7.6	99.4	2.2
223	Tebuconazole	89.8	3.5	99.0	4.2	90.3	1.1	89.7	9.8	104.2	1.5
224	Fenamidone	93.1	4.2	97.5	4.6	95.9	3.2	81.8	9.0	95.3	1.3
225	Anilofos	93.3	3.8	94.8	7.1	92.0	4.3	97.7	9.8	116.8	1.7
226	Bifenox	92.5	3.8	72.1	6.3	91.8	5.8	90.7	10.9	104.5	11.2
227	Phenothrin1	94.4	2.9	78.7	8.9	78.0	6.2	83.7	7.8	91.2	6.2
228	Phenothrin2	93.4	3.0	99.2	5.3	94.4	1.9	98.0	7.3	97.9	1.5
229	Tetradifon	92.7	3.8	93.4	2.1	96.3	1.3	86.6	5.8	96.1	4.2
230	Phosalone	96.8	3.6	99.9	4.6	95.7	4.2	101.2	6.3	115.4	1.9
231	Cyhalothrin-1	93.7	1.8	90.1	3.6	89.4	3.0	95.0	8.0	90.3	4.8
232	Pyriproxyfen	93.4	3.9	98.0	1.9	98.4	2.6	93.9	6.5	100.8	1.3
233	Azinphos-Methyl	90.0	5.5	106.1	8.4	65.2	6.1	98.2	18.4	90.2	2.3
234	Cyhalofop-butyl	95.6	4.4	96.2	1.1	99.3	2.7	93.8	5.7	101.6	2.1
235	Cyhalothrin-2	93.3	4.3	85.0	4.7	88.1	4.8	91.0	6.3	101.3	5.7
236	Mefenacet	91.5	3.7	96.5	5.7	92.5	3.8	90.9	5.4	97.4	2.0
237	Acrinathrin	85.1	3.2	81.6	13.0	116.1	6.3	125.5	1.3	84.9	1.5
238	Pyrazophos	95.7	2.4	83.8	3.3	83.1	3.5	74.9	8.9	74.4	12.7
239	Fenarimol	87.1	3.9	86.4	5.8	88.8	0.9	75.7	5.4	70.8	5.2
240	Pyraclufos	88.4	5.0	99.1	5.4	93.3	1.3	91.8	7.6	109.5	1.8

低感度・分解

LogPow=0.8

添加回収試験⑦

No.	化合物名	ほうれん草				ジャガイモ		玄米		オレンジ(皮付)		
		0.1ppm(n=4)		0.01ppm(n=5)		0.01ppm(n=3)		0.01ppm(n=3)		0.01ppm(n=3)		
		REC.(%)	RSD(%)	REC.(%)	RSD(%)	REC.(%)	RSD(%)	REC.(%)	RSD(%)	REC.(%)	RSD(%)	
241	Spirodiclofen	77.5	4.2	83.8	3.1	89.8	4.3	93.4	7.8	33.1	12.0	
242	Oryzalin	73.2	8.5	ND	-	ND	-	ND	-	ND	-	低感度
243	Bitertanol-1	92.6	4.7	96.1	5.8	84.2	1.4	91.1	6.6	93.4	1.9	
244	Permethrin-cis	90.7	3.5	88.1	2.2	88.0	2.1	85.8	8.8	89.3	1.9	
245	Bitertanol-2	95.5	5.1	96.0	7.1	81.7	2.5	98.8	9.4	96.3	2.7	
246	Permethrin-trans	91.7	3.8	96.1	1.4	92.7	1.3	98.8	6.7	92.1	2.7	
247	Pyridaben	94.1	3.5	98.0	2.4	91.6	0.9	94.7	6.7	98.4	1.5	
248	Fluquinconazole	89.3	3.8	92.5	7.3	91.6	2.7	91.1	6.5	90.6	2.0	
249	Cafenstrole	90.3	4.5	93.1	6.8	105.3	7.2	107.7	6.3	110.6	3.1	
250	Cyfluthrin-1	84.9	1.3	88.6	5.4	79.6	21.8	85.9	17.4	96.4	27.2	低感度
251	Cyfluthrin-2	79.5	10.3	68.0	6.5	106.9	5.4	54.8	31.6	90.8	2.5	
252	Fenbuconazole	89.0	2.9	94.1	4.6	88.0	2.0	85.8	5.3	81.1	7.4	
253	Cyfluthrin-3	89.5	3.1	80.9	4.3	100.7	0.6	91.6	10.9	95.3	5.4	
254	Cyfluthrin-4	92.3	4.4	83.4	8.4	91.7	1.1	92.3	8.7	99.5	3.7	
255	Cypermethrin-1	89.9	4.2	87.6	6.5	88.5	5.3	103.9	19.1	76.1	4.0	
256	Halfenprox	78.8	3.2	86.7	1.8	83.9	1.8	81.6	8.7	79.9	1.2	
257	Flucythrinate-1	88.9	4.9	91.4	6.0	94.8	1.9	93.1	6.6	89.8	5.6	
258	Cypermethrin-2	91.5	4.0	90.5	7.0	88.7	1.5	95.8	9.1	96.4	7.8	
259	Cypermethrin-3	77.6	3.3	10.9	7.5	35.1	0.6	36.9	6.4	23.2	5.0	低感度
260	Cypermethrin-4	88.1	5.5	79.6	8.5	84.5	18.4	88.0	6.9	101.1	8.5	
261	Etofenprox	88.4	2.9	90.9	1.5	92.1	0.8	86.2	5.8	90.8	1.4	
262	Flucythrinate-2	88.5	5.3	89.1	5.5	95.5	2.7	96.1	5.8	91.0	3.6	
263	Silafluofen	59.1	4.0	71.1	3.1	66.4	0.8	61.7	8.4	63.5	2.2	LogPow=8
264	Fluridone	85.7	3.6	88.9	6.6	85.3	0.9	82.4	5.1	97.0	4.0	
265	Pyrimidifen	88.0	3.6	91.1	2.7	87.8	0.9	83.2	8.0	95.9	0.7	
266	Fenvalerate-1	82.1	4.9	85.3	4.5	93.5	1.6	92.8	6.3	82.0	5.7	
267	Fluvalinate-1	86.1	7.4	87.5	17.4	107.0	2.1	125.3	5.4	71.5	2.7	
268	Fluvalinate-2	88.2	7.3	88.8	17.9	109.7	4.1	123.8	5.1	73.1	0.9	
269	Flumioxazin	88.3	6.8	88.1	7.8	87.9	2.8	92.1	5.7	102.0	4.2	
270	Fenvalerate-2	90.0	6.1	91.3	5.6	90.6	1.0	107.4	6.4	88.0	3.1	
271	Difenoconazole-1	87.2	4.3	91.1	6.5	87.3	2.7	87.2	3.9	87.1	2.9	
272	Difenoconazole-2	85.7	5.9	92.5	7.6	86.0	3.3	84.1	5.4	88.1	4.9	
273	Deltamethrin	81.7	4.7	83.1	15.0	107.6	12.7	107.6	10.7	65.3	5.8	
274	Flumiclorac-pentyl	103.8	4.5	103.2	6.4	101.3	0.9	110.9	6.6	116.0	1.2	
275	Tolfenpyrad	91.9	4.3	90.9	3.0	90.8	1.2	89.6	6.1	95.6	0.7	
276	Imibenconazole	88.0	9.9	94.1	6.3	90.5	3.4	97.1	8.2	74.4	7.1	
277	Fluthiacet-methyl	97.3	10.3	95.4	11.7	100.6	2.6	110.8	2.7	107.4	2.7	

回収率分布

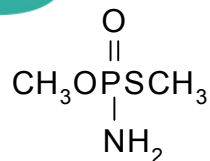
作物	ほうれん草		ジャガイモ	玄米	オレンジ
	0.1ppm	0.01ppm	0.01ppm	0.01ppm	0.01ppm
試料中濃度	0.1ppm	0.01ppm	0.01ppm	0.01ppm	0.01ppm
REC(%)					
ND, 0-30	3	17	16	18	17
30-50	5	5	6	11	6
50-70	6	10	11	9	14
70-120	264	245	243	234	239
120-	0	1	2	6	2
合計	278	278	278	278	278

(単位:成分)

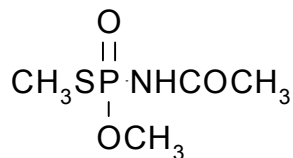
対応策

農薬名	物性	LC/MSMS
Acephate	LogPow=-0.89	◎
Acetamipride	LogPow=0.8	○
Carboxin	LogPow=2.2, Pka<0.5	?
Demeton-S-methyl	LogPow=1.3	△
Dichlofluanid		?
Dimethipin	LogPow=-0.17	×
Dimethoate	LogPow=0.7	○
Ethiofencarb	LogPow=2	○
Methamidophos	LogPow=-0.8	◎
Mevinphos	LogPow=0.13	△
Monocrotophos	LogPow=-0.22	○
Oryzalin	Pka=9.4	?
Phorate	P=S	△
Pyroquilon	LogPow=1.6	?
Thiometon	LogPow=3.15, P=S	?
Tricyclazole	LogPow=1.4	?

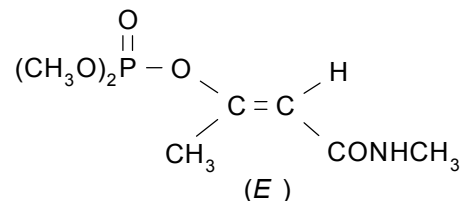
回収率の低い農薬



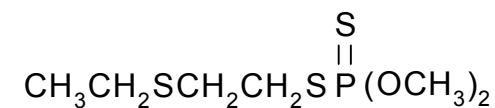
Methamidophos



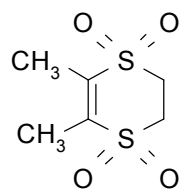
Acephate



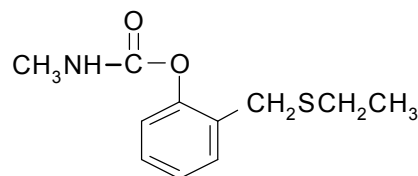
Monocrotophos



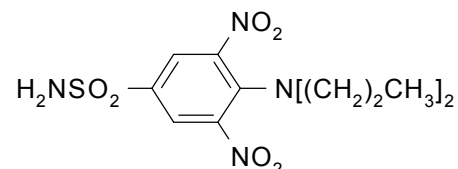
Thiometon



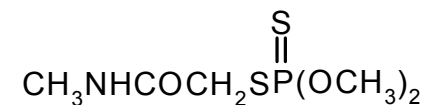
Dimethipin



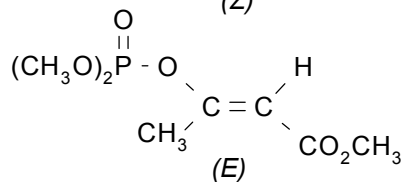
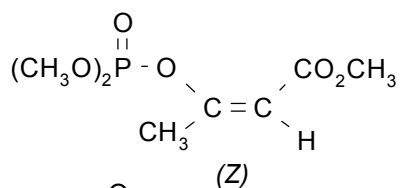
Ethiofencarb



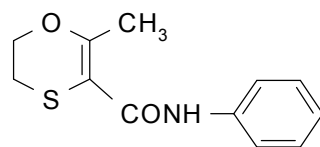
Oryzalin



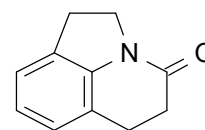
Dimethoate



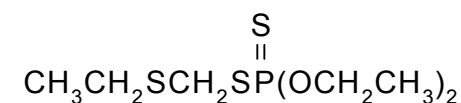
Mevinphos



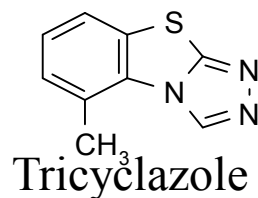
Carboxin



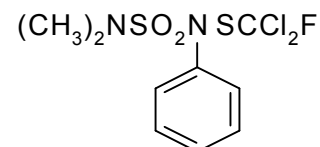
Pyroquilon



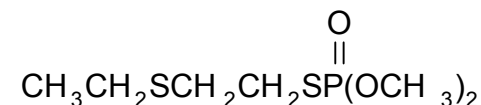
Phorate



Tricyclazole



Dichlofluanid



Dimethon-S-methyl