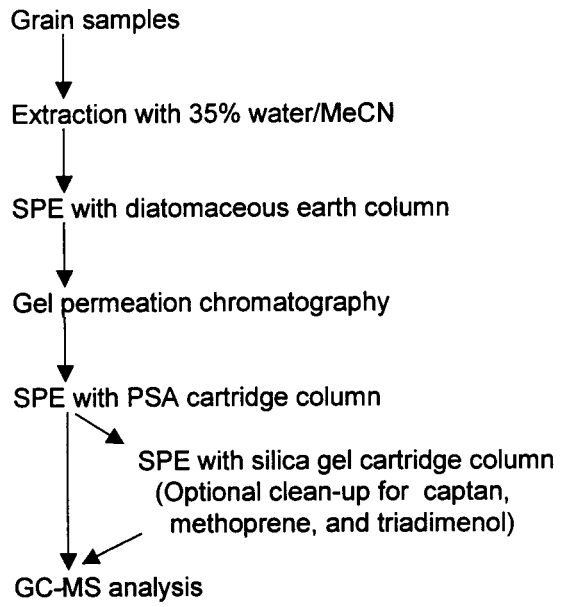


(a) SFE Method



(b) Solvent Extraction Method

**Scheme 1. 穀類試料の分析法の概略**

SPE: Solid phase extraction

表1. GC-MS (SIM)測定における農薬の保持時間( $t_r$ )及びモニターイオン( $m/z$ )

No.	Pesticides	$t_r$ (min)	$m/z$	No.	Pesticides*	$t_r$ (min)	$m/z$
<b>Organochlorine (16)</b>				<b>Pyrethroid (12)</b>			
1	Aldrin	14.67	262.8	1	Acrinathrin	19.98	181.0
2	$\alpha$ -BHC	11.80	182.9	2	Cyfluthrin-1	21.18	226.0
3	$\beta$ -BHC	12.28	182.9		Cyfluthrin-2	21.27	226.0
4	$\delta$ -BHC	13.03	182.9		Cyfluthrin-3	21.33	226.0
5	$\gamma$ -BHC	12.47	182.9		Cyfluthrin-4	21.37	226.0
6	Captafol	18.45	79.0	3	Cyhalothrin-1	19.65	197.0
7	Captan	15.58	79.0		Cyhalothrin-2	19.83	197.0
8	Chlorobenzilate	17.13	250.9	4	Cypermethrin-1	21.51	162.9
9	p,p'-DDD	17.31	235.0		Cypermethrin-2	21.61	162.9
10	p,p'-DDE	16.51	245.9		Cypermethrin-3	21.67	162.9
11	o,p'-DDT	17.37	235.0		Cypermethrin-4	21.70	162.9
12	p,p'-DDT	18.02	235.0	5	Deltamethrin-1	23.14	181.0
13	Dieldrin	16.66	262.8		Deltamethrin-2	23.39	181.0
14	Endrin	17.07	262.8	6	Ethofenprox	21.85	163.0
15	Heptachlor	13.95	271.7	7	Fenvalerate-1	22.47	167.0
16	Heptachlor epoxide	15.42	352.8		Fenvalerate-2	22.71	167.0
<b>Carbamate (15)</b>				8	Flucythrinate-1	21.68	199.0
1	Aldicarb	3.74	115.0		Flucythrinate-2	21.87	199.0
2	Bendiocarb	11.39	151.0	9	Fluvalinate-1	22.59	250.0
3	Butylate	8.74	146.0		Fluvalinate-2	22.66	250.0
4	Carbaryl	13.87	144.0	10	Halfenprox	21.59	262.9
5	Chlorpropham	11.25	213.0	11	Permethrin-1	20.66	183.0
6	Diethofencarb	14.56	151.0		Permethrin-2	20.78	183.0
7	EPTC	7.97	86.0	12	Tefluthrin	12.91	177.0
8	Esprocarb	14.41	222.1	<b>Other (42)</b>			
9	Ethiofencarb	13.32	107.0	1	Acetamidiprid	18.76	152.0
10	Fenobucarb	10.69	121.0	2	Amitraz	19.83	293.2
11	Isoprocarb	9.92	121.0	3	Benalaxyl	17.74	148.1
12	Methiocarb	14.26	168.0	4	Benfuresate	13.47	163.0
13	Pirimicarb	13.13	166.1	5	Bitertanol-1	20.61	170.1
14	Propoxur	10.71	110.0		Bitertanol-2	20.71	170.1
15	Thiobencarb	14.59	100.1	6	Chinomethionat	15.90	233.9
<b>Organophosphate (43)</b>				7	Cyproconazole-1	16.90	222.0
1	Acephate	8.84	136.0		Cyproconazole-2	16.93	222.0
2	Azinphos-ethyl	20.21	160.0	8	Dichlofluanid	14.38	123.0
3	Azinphos-methyl	19.64	160.0	9	2,4-Dichloroaniline	7.78	160.9
4	Bromophos-ethyl	15.78	302.8	10	Difenoconazole-1	23.03	323.0
5	Butamifos	16.12	286.0		Difenoconazole-2	23.10	323.0
6	Cadusafos	11.58	158.9	11	Dimethipin	12.27	54.1
7	(E)-Chlorfenvinphos	15.18	266.9	12	Fenarimol	20.09	138.9
8	(Z)-Chlorfenvinphos	15.39	266.9	13	Flusilazole	16.60	233.0
9	Chlorpyrifos	14.54	198.9	14	Flutolanil	16.24	173.0
10	Chlorpyrifos-methyl	13.61	285.9	15	Fosthiazate-1	15.02	194.9
11	Diazinon	12.64	179.1		Fosthiazate-2	15.08	194.9
12	Dichlorvos	6.78	109.0	16	Imibenconazole	24.71	125.0
13	Dimethoate	12.03	87.0	17	Imibenconazole des benzyl type	16.68	83.0
14	(Z)-Dimethylvinphos	14.60	294.9	18	Iprodione	18.71	313.9
15	Dioxabenzofos	11.44	215.9	19	Iprodione metabolite	19.39	186.8
16	Disulfoton	12.89	88.0	20	Lenacil	17.94	153.0
17	Edifenphos	17.87	310.0	21	Mefenacet	19.78	192.0
18	EPN	18.92	157.0	22	Mepronil	17.53	119.1
19	Ethion	17.30	230.9	23	Methoprene	15.66	111.0
20	Ethoprophos	11.03	157.9	24	Metolachlor	14.51	162.1
21	Etrimfos	12.98	292.0	25	Metribuzin	13.62	198.0
22	Fenitrothion	14.24	277.0	26	Myclobutanil	16.57	179.0
23	Fensulfothion	17.16	292.0	27	Paclobutrazol	15.97	236.0
24	Fenthion	14.64	278.0	28	Pendimethalin	15.19	252.1
25	Isofenphos	15.36	213.0	29	Pretilachlor	16.35	238.1
26	Isofenphos oxon	14.71	228.9	30	Propiconazole-1	17.85	259.0
27	Malaoxon	13.66	127.0		Propiconazole-2	17.97	259.0
28	Malathion	14.39	173.1	31	Pyridaben	20.85	147.1
29	Methamidophos	6.66	94.0	32	(E)-Pyrifenox	15.89	261.9
30	Methidathion	15.79	144.9	33	(Z)-Pyrifenox	15.38	261.9
31	Parathion	14.70	291.0	34	Pyrimidifen	22.26	184.0
32	Parathion-methyl	13.75	262.9	35	Pyriproxyfen	19.70	136.0
33	Phenthoate	15.50	273.9	36	Siafluofen	22.00	258.0
34	Phosalone	19.54	181.9	37	Tebuconazole	18.28	125.0
35	Phosmet	18.88	160.0	38	Tebufenpyrad	19.15	333.1
36	Pirimiphos-methyl	14.16	290.0	39	Thenylchlor	18.18	127.0
37	Prothiofos	16.34	309.0	40	Triadimefon	14.76	57.1
38	Pyraclufos	20.34	360.0	41	Triadimenol	15.58	112.1
39	Quinalphos	15.52	146.0	42	Tricyclazole	16.45	188.9
40	Terbufos	12.54	231.0				
41	Thiometon	11.93	88.0				
42	Toiclofos-methyl	13.77	264.9				
43	Vamidothion	15.93	145.0				

\*異性体が存在する農薬については保持時間順に農薬名の後に番号をつけて示した。

表 2. SFE法の添加回収実験結果 (n = 3)

No.	Pesticide	Recovery (%)							
		Rice		Wheat		Corn		Buckwheat	
		Mean	RSD <sup>1</sup>	Mean	RSD	Mean	RSD	Mean	RSD
<b>Organochlorine (16)</b>									
1	Aldrin	90.0	3.3	90.2	5.0	93.7	1.9	87.5	0.3
2	$\alpha$ -BHC	90.8	2.7	90.2	5.2	93.1	2.0	89.8	1.2
3	$\beta$ -BHC	89.5	2.3	89.1	4.3	90.9	3.3	90.7	1.3
4	$\delta$ -BHC	88.9	3.2	90.6	5.3	94.3	1.7	94.4	2.5
5	$\gamma$ -BHC	89.5	2.1	92.6	6.5	96.9	1.7	92.9	0.9
6	Captafol	74.3	2.0	64.8	6.1	85.6	6.8	77.5	6.7
7	Captan	78.7	3.3	55.7	6.3	73.0	4.6	91.0	0.5
8	Chlorobenzilate	90.4	2.9	94.9	6.0	101.0	4.9	102.5	1.3
9	p,p'-DDD	88.1	2.5	91.9	4.5	99.5	3.3	99.0	0.2
10	p,p'-DDE	90.6	2.3	93.1	5.9	98.8	2.3	94.5	0.6
11	o,p'-DDT	88.6	2.6	90.6	5.8	96.8	3.8	95.6	0.6
12	p,p'-DDT	93.4	3.1	93.8	4.5	98.3	3.5	95.9	1.1
13	Dieldrin	87.7	3.7	91.2	6.0	96.2	3.5	92.0	0.8
14	Endrin	90.3	3.9	92.5	6.2	95.0	3.6	100.3	0.6
15	Heptachlor	90.9	2.8	92.4	6.4	94.1	2.8	87.5	1.2
16	Heptachlor epoxide	88.3	3.6	89.9	6.4	93.9	2.7	88.6	1.1
<b>Pyrethroid (8)</b>									
2	Cyfluthrin	89.2	6.2	93.0	3.8	98.1	4.6	94.0	1.7
3	Cyhalothrin	93.4	5.3	97.5	3.1	97.7	3.7	97.1	0.5
4	Cypermethrin	90.8	2.6	91.4	3.0	83.2	0.8	91.6	5.2
5	Deltamethrin	84.2	2.1	89.0	2.7	93.9	3.4	90.8	2.5
7	Fenvalerate	90.9	1.7	89.9	2.3	94.9	3.7	97.3	1.9
8	Flucythrinate	88.4	3.5	88.2	2.3	91.6	3.7	95.1	2.9
9	Fluvalinate	87.7	2.1	85.3	1.8	89.8	1.9	88.1	2.4
11	Permethrin	100.1	5.2	93.2	1.9	100.8	2.3	96.6	1.7
<b>Carbamate (11)</b>									
2	Bendiocarb	92.0	1.0	89.7	5.9	91.1	3.3	89.7	0.9
5	Chlorpropham	92.1	1.8	92.9	5.4	97.4	4.3	92.1	0.6
6	Diethofencarb	89.2	1.3	93.3	6.8	101.6	6.3	98.6	1.1
8	Espirocarb	89.3	1.5	91.8	5.6	98.4	2.7	92.9	0.8
9	Ethiofencarb	91.1	2.1	93.0	5.4	95.2	4.2	75.8	4.1
10	Fenobucarb	94.6	2.3	91.9	5.8	92.1	4.1	89.8	1.9
11	Isoprocab	91.2	2.0	90.4	6.0	90.9	3.6	87.5	3.3
12	Methiocarb	90.2	3.1	92.9	6.3	96.9	4.6	90.2	0.9
13	Pirimicarb	92.3	1.9	91.5	5.7	96.1	3.4	87.3	0.8
14	Propoxur	90.8	0.9	90.2	6.0	90.0	4.2	88.3	1.8
15	Thiobencarb	90.8	1.6	94.1	4.3	98.7	3.7	96.4	1.7
<b>Organophosphate (33)</b>									
1	Acephate	27.3	6.3	8.5	4.9	14.2	5.2	4.6	8.2
2	Azinphos-ethyl	91.4	6.6	96.9	3.4	102.7	4.3	101.1	1.2
3	Azinphos-methyl	98.3	9.6	102.6	4.3	105.1	5.1	97.0	1.6
4	Bromophos-ethyl	92.9	3.1	94.4	6.2	104.8	4.2	83.4	1.9
7	(E)-Chlorfenvinphos	90.5	1.5	93.7	6.0	98.6	7.2	95.9	1.0
8	(Z)-Chlorfenvinphos	83.7	3.1	85.0	6.5	97.9	6.0	94.4	0.9
9	Chlorpyrifos	92.1	3.8	94.7	6.1	101.4	3.4	92.6	0.9
10	Chlorpyrifos-methyl	91.1	1.7	93.5	5.9	96.8	3.1	90.8	0.8
11	Diazinon	96.2	3.8	97.6	2.9	101.6	1.7	91.4	1.2
12	Dichlorvos	79.9	4.1	74.1	10.3	87.1	1.7	79.3	4.9
13	Dimethoate	74.8	3.5	73.4	6.5	64.7	8.2	67.5	1.2
15	Dioxabenzofos	89.7	2.5	88.7	5.5	91.8	3.2	87.5	1.3
16	Disulfoton	90.4	1.0	91.4	5.8	97.8	3.6	83.3	2.1
17	Edifenphos	91.6	7.2	91.7	4.7	101.1	5.6	101.8	0.1
18	EPN	91.3	7.1	94.6	4.7	95.5	7.3	100.4	2.3
20	Ethoprophos	91.2	1.1	91.7	4.8	93.2	3.9	88.8	2.0
21	Etrimfos	91.1	2.6	93.3	5.1	97.9	2.6	90.3	1.0
22	Fenitrothion	89.2	3.3	91.9	6.9	96.8	6.2	92.1	1.3
23	Fensulfotlion	97.3	5.6	100.4	6.1	95.8	6.0	104.8	2.8
24	Fenthion	89.1	2.8	90.1	5.8	97.9	3.9	86.5	0.6

表 2. (続き)

No.	Pesticide	Recovery (%)							
		Rice		Wheat		Corn		Buckwheat	
		Mean	RSD	Mean	RSD	Mean	RSD	Mean	RSD
28	Malathion	92.5	1.6	96.3	7.7	110.4	4.0	98.4	0.6
29	Methamidophos	ND <sup>2</sup>		ND		ND		ND	
30	Methidathion	90.8	2.7	92.9	5.8	104.3	2.7	100.7	1.6
31	Parathion	95.3	4.1	91.8	7.0	91.3	3.4	86.9	5.9
32	Parathion-methyl	88.5	1.0	91.5	6.1	98.6	2.3	91.2	1.2
33	Phenthoate	91.9	2.3	91.6	6.3	98.7	5.1	94.0	1.0
34	Phosalone	92.2	6.1	96.5	4.6	97.5	2.9	103.3	1.1
35	Phosmet	88.5	7.6	90.1	4.6	92.5	5.3	100.8	1.1
36	Pirimiphos-methyl	91.4	2.0	92.3	4.9	103.8	2.8	94.4	0.6
37	Prothiofos	93.6	3.8	95.7	5.4	101.0	3.3	96.4	0.6
39	Quinalphos	92.2	3.1	92.0	6.0	101.9	3.8	94.7	1.1
40	Terbufos	89.9	1.4	90.7	5.9	97.6	3.7	88.3	1.5
41	Thiometon	89.6	0.8	90.2	5.6	88.5	2.2	88.0	2.3
	<b>Other (19)</b>								
2	Amitraz	12.8	35.8	10.2	3.7	13.6	5.1	19.2	35.9
3	Benalaxyl	75.6	5.0	72.5	5.4	102.5	3.3	99.9	1.0
5	Bitertanol	119.1	2.5	113.4	1.6	97.2	4.5	109.6	3.8
6	Chinomethionat	36.5	6.7	38.8	8.9	41.2	6.5	44.6	2.0
8	Dichlofluanid	81.6	8.8	59.9	3.4	87.8	3.9	95.2	2.0
11	Dimethipin	77.0	0.9	74.7	6.4	74.5	3.8	74.2	1.0
14	Flutolanil	98.8	7.6	97.9	5.2	88.3	4.9	100.7	3.4
20	Lenacil	70.9	0.5	69.3	6.5	15.2	5.6	46.9	3.6
21	Mefenacet	94.5	4.7	100.8	4.2	96.7	3.3	94.0	1.2
22	Mepronil	93.0	5.4	96.2	4.1	100.5	5.1	100.8	1.3
23	Methoprene	96.4	1.2	105.6	3.2	91.3	3.1	101.8	5.6
25	Metribuzin	78.8	1.6	80.2	7.7	84.1	4.0	73.7	1.0
26	Myclobutanil	87.7	3.5	88.6	6.4	92.2	2.8	100.1	2.2
28	Pendimethalin	88.9	1.2	92.9	7.0	97.5	5.7	94.4	0.3
29	Pretilachlor	94.2	2.5	96.7	6.1	103.5	3.5	104.1	1.4
30	Propiconazole	97.1	5.2	93.2	3.5	96.8	4.8	102.0	4.1
31	Pyridaben	83.1	4.4	85.9	4.6	98.6	8.1	94.5	3.6
40	Triadimefon	93.2	2.5	90.3	6.2	100.1	3.5	90.7	0.7
41	Triadimenol	89.8	3.9	95.4	3.1	100.4	5.1	94.6	1.2

<sup>1</sup> RSD, relative standard deviation; <sup>2</sup> ND, not detected.

表 3. SFE 法の添加回収実験結果のまとめ

Recovery	Rice	Wheat	Corn	Buckwheat
>70%	83	79	81	81
50 - 70%	0	4	1	1
<50%	4	4	5	5

数字は農薬数を示す.

表4. SFE法と溶媒抽出法の小麦からの回収率の比較

No.	Pesticide	Recovery (%), n = 3				No.	Pesticide	Recovery (%), n = 3			
		SFE		Solvent extraction				SFE		Solvent extraction	
		Mean	RSD*	Mean	RSD			Mean	RSD	Mean	RSD
<b>Organochlorine (16)</b>					<b>Organophosphate (cont.)</b>						
1	Aldrin	96.9	3.1	75.4	1.5	21	Etrinfos	100.2	3.3	93.8	5.1
2	α-BHC	93.3	6.8	77.2	2.7	22	Fenitrothion	106.2	5.1	105.4	8.0
3	β-BHC	101.7	4.9	88.0	5.2	23	Fensulfthion	90.1	2.8	83.0	4.4
4	δ-BHC	93.5	2.8	93.0	3.4	24	Fenthion	102.4	4.0	85.6	8.3
5	γ-BHC	97.2	3.6	83.0	0.7	25	Isofenphos	106.8	2.8	96.9	5.3
6	Captafol	9.9	92	23.3	12.7	26	Isofenphos oxon	101.8	5.4	102.8	7.3
7	Captan	19.8	26	18.9	13.6	27	Malaoxon	97.9	3.2	116.7	4.4
8	Chlorobenzilate	105.4	4.6	101.5	6.3	28	Malathion	99.6	3.4	101.2	9.2
9	p,p'-DDD	102.2	2.0	92.1	6.0	29	Methamidophos	17.4	8.0	90.9	3.0
10	p,p'-DDE	104.0	2.1	84.9	4.5	30	Methidathion	104.6	4.3	106.2	6.7
11	o,p'-DDT	101.9	1.7	87.1	4.4	31	Parathion	95.4	1.6	105.3	3.9
12	p,p'-DDT	105.3	5.0	91.1	5.1	32	Parathion-methyl	110.6	5.1	116.6	8.7
13	Dieldrin	100.3	0.7	88.5	2.6	33	Phenthoate	105.3	3.9	104.5	6.8
14	Endrin	106.2	2.9	96.4	4.0	34	Phosalone	97.7	2.8	88.4	5.1
15	Heptachlor	97.7	2.3	77.1	2.8	35	Phosmet	94.6	3.1	87.9	4.2
16	Heptachlor epoxide	98.2	1.3	88.0	3.6	36	Pirimiphos-methyl	104.6	3.3	96.3	4.3
<b>Pyrethroid (12)</b>					<b>Other (42)</b>						
1	Acrinathrin	87.4	5.3	18.6	3.6	37	Prothiofos	99.6	4.2	95.1	5.2
2	Cyfluthrin	91.3	1.1	86.6	5.4	38	Pyraclufos	96.9	3.2	96.7	6.1
3	Cyhalothrin	99.0	2.5	86.1	6.3	39	Quinalphos	104.6	3.7	96.7	6.2
4	Cypermethrin	87.3	1.7	87.5	1.1	40	Terbufos	98.7	3.4	69.2	7.9
5	Deltamethrin	83.9	8.8	88.3	4.1	41	Thiometon	92.8	4.2	50.4	13
6	Ethofenprox	94.2	1.3	85.7	3.3	42	Toiclofos-methyl	99.1	4.0	90.8	3.5
7	Fenvalerate	85.9	5.0	82.5	3.9	43	Vamidothion	64.9	4.3	68.3	16
8	Flucythrinate	85.4	1.0	84.1	3.2	1	Acetamiprid	17.7	5.7	89.4	0.9
9	Fluvalinate	76.3	8.4	81.8	1.8	2	Amitraz	14.1	16.3	22.3	91
10	Halfeprox	98.0	2.4	83.7	5.1	3	Benalaxyl	100.0	1.1	90.7	8.2
11	Permethrin	95.6	1.6	91.7	5.1	4	Benfuresate	99.5	3.3	92.1	5.1
12	Tefluthrin	100.5	2.4	87.9	3.2	5	Bitertanol	87.4	5.9	104.0	2.3
<b>Carbamate (15)</b>					<b>Other (42)</b>						
1	Aldicarb	80.6	6.3	72.2	12	6	Chinomethionat	17.5	28.6	32.3	12
2	Bendiocarb	94.3	5.1	94.8	4.9	7	Cyproconazole	98.0	3.8	96.7	6.8
3	Butylate	110.7	3.2	32.0	19	8	Dichlofluanid	25.7	16.5	25.5	3.4
4	Carbaryl	95.1	4.7	97.4	6.7	9	2,4-Dichloroaniline	95.5	7.4	57.7	8.3
5	Chlorpropham	98.2	3.7	89.2	5.8	10	Difenoconazole	88.2	2.5	93.2	1.7
6	Diethofencarb	91.3	3.0	85.2	5.5	11	Dimethipin	84.0	3.3	93.8	7.6
7	EPTC	111.4	3.3	28.9	8.8	12	Fenarimol	88.3	1.4	86.9	6.3
8	Esprocarb	100.2	3.1	92.3	3.6	13	Flusilazole	100.7	3.2	95.2	6.5
9	Ethiofencarb	95.1	5.7	18.2	21	14	Flutolanil	106.7	4.4	100.4	8.9
10	Fenobucarb	94.9	3.5	87.9	4.3	15	Fosthiazate	98.2	2.2	98.6	7.0
11	Isoprocarb	90.3	2.8	82.2	2.3	16	Imibenconazole	96.9	3.4	93.7	4.7
12	Methiocarb	103.9	3.6	93.6	6.6	17	Imibenconazole des benzyl type	78.8	2.4	98.6	7.6
13	Pinmicarb	96.9	3.1	100.6	5.5	18	Iprodione	92.3	4.7	83.6	3.5
14	Propoxur	93.3	3.9	97.1	4.0	19	Iprodione metabolite	90.3	2.1	92.9	8.6
15	Thiobencarb	101.9	2.5	92.0	4.8	20	Lenacil	77.3	2.0	95.0	6.2
<b>Organophosphate (43)</b>					<b>Other (42)</b>						
1	Acephate	46.1	6.7	97.4	5.6	21	Mefenacet	91.0	1.9	90.7	5.0
2	Azinphos-ethyl	94.8	2.9	87.9	5.4	22	Mepronil	99.8	4.0	94.0	9.0
3	Azinphos-methyl	99.6	2.6	105.0	4.6	23	Methoprene	106.0	5.1	98.7	8.1
4	Bromophos-ethyl	108.2	4.7	97.1	5.1	24	Metolachlor	106.3	3.1	99.7	6.5
5	Butamifos	114.2	5.5	98.6	0.7	25	Metribuzin	78.8	1.3	91.6	4.4
6	Cadusafos	101.3	4.7	84.3	3.9	26	Myclobutanil	100.9	3.4	94.5	6.7
7	(E)-Chlorfenvinphos	103.4	5.1	98.8	6.4	27	Paclbutrazol	102.5	4.2	96.4	7.6
8	(Z)-Chlorfenvinphos	105.1	3.1	97.9	7.3	28	Pendimethalin	104.5	3.0	98.5	6.9
9	Chlorpyrifos	98.4	4.8	92.7	4.9	29	Pretilachlor	112.9	2.6	98.4	7.2
10	Chlorpyrifos-methyl	98.3	2.0	106.6	2.9	30	Propiconazole	103.4	3.4	92.4	2.9
11	Diazinon	100.1	3.2	96.7	7.5	31	Pyridaben	98.3	0.9	86.9	3.9
12	Dichlorvos	74.3	6.4	60.6	11	32	(E)-Pyrifenox	101.3	5.6	106.8	0.9
13	Dimethoate	87.4	6.4	105.0	4.8	33	(Z)-Pyrifenox	102.9	3.3	108.4	8.0
14	(Z)-Dimethylvinphos	105.9	4.3	104.3	7.0	34	Pyrimidifen	88.8	0.2	85.1	2.8
15	Dioxabenzofos	90.4	3.9	83.4	3.6	35	Pyriproxyfen	97.8	3.3	90.5	6.5
16	Disulfoton	95.2	3.7	46.6	11	36	Silaflofen	91.3	2.1	75.3	5.5
17	Edifenphos	102.3	2.8	94.2	3.3	37	Tebuconazole	93.8	3.0	88.0	3.8
18	EPN	99.6	1.2	89.0	5.3	38	Tebuconazole	98.6	4.5	89.7	5.5
19	Ethion	103.8	1.7	96.4	6.8	39	Thenylchlor	104.7	4.3	92.7	2.4
20	Ethoprophos	96.8	4.4	83.5	2.2	40	Triadimefon	110.7	1.1	102.4	2.6
						41	Triadimenol	103.4	3.8	99.6	3.1
						42	Tricyclazole	10.8	8.9	100.8	6.0

\* RSD, relative standard deviation.

表5. 小麦からの添加回収実験で低回収率の農薬

a) SFE法と溶媒抽出法の両方で低回収率の農薬

OC: captafol, captan

OT: amitraz, chinomethionat, dichlofluanid

b) SFE法で低回収の農薬

OP: acephate, methamidophos

OT: acetamiprid, tricyclazole

c) 溶媒抽出法で低回収率の農薬

PY: acrinathrin

CA: butylate, EPTC, ethiofencarb

OP: disulfoton, thiometon

OT: 2,4-dichloroaniline

OC = organochlorine; PY = pyrethroid; CA = carbamate;  
OP = organophosphate; OT = other.

表6. SFE法と溶媒抽出法の測定値の比較

Sample	Pesticide	Pesticide residue (ng/g), n=3			Sample	Pesticide	Pesticide residue (ng/g), n=3		
		SFE		Ratios <sup>2</sup>			SFE		Ratios <sup>2</sup>
		Mean (RSD <sup>1</sup> )	Solvent extraction Mean (RSD)				Mean (RSD)	Solvent extraction Mean (RSD)	
Rice No.1	Chlorpyrifos-methyl	3 (3.3)	3 (5.6)	1.02	Barley No.2	Chlorpyrifos-methyl	53 (7.3)	52 (4.2)	1.03
	Fenitrothion	4 (6.3)	4 (4.7)	1.04		Malathion	42 (7.5)	42 (2.1)	0.99
	Fenobucarb	0.3 (4.6)	1 (7.9)	0.35	Barley No.3	Chlorpyrifos-methyl	32 (1.2)	29 (5.0)	1.09
	Pirimiphos-methyl	1 (8.7)	1 (5.0)	0.97		Malathion	103 (1.2)	111 (4.0)	0.93
Rice No.2						p,p'-DDT	2 (5.5)	3 (5.1)	0.96
	Fenitrothion	2 (6.0)	2 (3.5)	0.91	Barley No.4	Carbaryl	3 (7.1)	9 (1.0)	0.34
	Fenobucarb	128 (5.3)	275 (1.3)	0.46		Chlorpyrifos	3 (7.2)	3 (0.2)	0.93
	p,p'-DDE	1 (4.9)	1 (9.3)	0.98		Chlorpyrifos-methyl	156 (4.5)	141 (2.6)	1.10
	p,p'-DDT	2 (6.6)	2 (5.7)	0.96		Dichlorvos	3 (4.7)	59 (8.3)	0.04
Rice No.3	Fenobucarb	3 (4.3)	6 (5.2)	0.42		Fenitrothion	628 (4.7)	733 (1.6)	0.86
Rice No.4						Methoprene	56 (2.5)	53 (6.3)	1.06
	Fenobucarb	19 (3.8)	39 (4.6)	0.48		Permethrin	13 (4.8)	12 (4.5)	1.04
Rice No.5						Pirimiphos-methyl	95 (2.0)	85 (4.4)	1.12
	Fenitrothion	9 (0.4)	11 (3.1)	0.88	Barley No.5	Carbaryl	17 (6.7)	40 (4.8)	0.43
Wheat No.1	Chlorpyrifos-methyl	13 (6.1)	11 (4.6)	1.16		Chlorpyrifos-methyl	13 (9.9)	14 (3.3)	0.98
	Malathion	430 (4.0)	432 (2.1)	1.00		Dichlorvos	7.7 (6.7)	85 (24)	0.09
	p,p'-DDE	2 (4.2)	1 (2.4)	1.09		Fenitrothion	528 (4.6)	844 (3.6)	0.63
	p,p'-DDT	4 (5.1)	4 (1.6)	1.13		Malathion	3 (7.6)	3 (3.6)	1.10
Wheat No.2						Pirimiphos-Me	19 (0.7)	19 (7.7)	0.98
	Chlorpyrifos-methyl	87 (3.3)	77 (5.3)	1.12	Barley No.6	Chlorpyrifos	7 (9.3)	7 (7.5)	1.01
	Fenitrothion	3 (1.5)	3 (2.4)	1.00		Chlorpyrifos-methyl	162 (4.6)	157 (7.3)	1.03
	γ-BHC	3 (7.1)	3 (3.9)	1.04		Dichlorvos	1 (7.7)	13 (9.6)	0.10
	Malathion	548 (6.2)	531 (4.8)	1.03		Fenitrothion	7 (7.2)	11 (14)	0.61
	p,p'-DDE	1 (3.7)	1 (9.6)	1.00		Malathion	6 (1.7)	7 (1.1)	0.93
	p,p'-DDT	2 (6.6)	2 (7.4)	1.00		Methoprene	24 (6.1)	23 (1.5)	1.01
Wheat No.3						Pirimiphos-methyl	38 (7.7)	36 (5.0)	1.05
	Bendiocarb	5 (8.7)	6 (8.3)	0.88	Corn No.1	Chlorpyrifos-methyl	1 (2.7)	1 (5.5)	1.07
	Chlorpyrifos-methyl	164 (4.1)	163 (8.6)	1.01		Malathion	86 (1.7)	108 (7.3)	0.79
	Fenitrothion	2 (6.1)	2 (5.9)	0.95		Pirimiphos-methyl	11 (3.5)	11 (1.4)	1.08
	Malathion	11 (8.7)	12 (7.4)	0.94	Corn No.2	Chlorpyrifos-methyl	3 (1.3)	3 (9.7)	1.06
Wheat No.4						Fenitrothion	15 (5.3)	20 (4.9)	0.75
	Chlorpyrifos-methyl	281 (8.9)	251 (1.6)	1.12		Pirimiphos-methyl	4 (4.3)	4 (8.2)	1.08
	Malathion	681 (3.8)	668 (2.7)	1.02	Corn No.3	Malathion	5 (1.8)	8 (1.8)	0.61
Wheat No.5					Corn No.4	Malathion	21 (7.4)	28 (6.9)	0.74
	Chlorpyrifos	3 (3.3)	3 (8.5)	1.00		Pirimiphos-methyl	5 (8.6)	5 (0.3)	1.06
	Chlorpyrifos-methyl	740 (1.2)	701 (3.2)	1.06	Corn No.5	Malathion	3 (7.4)	5 (4.1)	0.66
	Malathion	1129 (5.0)	1087 (2.7)	1.04		Pirimiphos-methyl	17 (1.0)	15 (1.5)	1.15
Wheat No.6									
	Chlorpyrifos	4 (1.8)	4 (1.5)	1.01					
	Chlorpyrifos-methyl	677 (3.9)	644 (1.0)	1.05					
	Malathion	663 (6.2)	669 (6.0)	0.99					
Barley No.1									
	Chlorpyrifos-methyl	90 (4.4)	80 (10)	1.13					
	Fenitrothion	1721 (2.2)	1873 (15)	0.92					
	Malathion	3 (5.1)	3 (7.8)	0.95					
	Methoprene	180 (5.1)	179 (4.7)	1.01					
	Pirimiphos-methyl	1 (3.0)	1 (12)	1.06					

<sup>1</sup> RSD, relative standard deviation; <sup>2</sup> Ratios = (Results of SFE method)/(Results of solvent extraction method)

表7. 農薬別のSFE法と溶媒抽出法の比較

Pesticides	Ratios <sup>1</sup> (Mean ± SD <sup>2</sup> )	Recovery (%)		Solubility in water (mg/L)
		SFE <sup>3</sup>	Solvent <sup>4</sup>	
Dichlorvos	0.08 ± 0.03	74	61	10000
Fenobucarb	0.43 ± 0.06	95	88	610
Malathion	0.91 ± 0.15	100	101	145
Carbaryl	0.38 ± 0.06	95	97	120
Bendiocarb	0.88	94	95	40
Fenitrothion	0.85 ± 0.15	106	105	30
γ-BHC	1.04	97	83	7.5
Pirimiphos-methyl	1.06 ± 0.06	105	96	5
Chlorpyrifos-methyl	1.07 ± 0.05	98	107	4
Chlorpyrifos	0.99 ± 0.04	98	93	2
Methoprene	1.03 ± 0.03	106	99	1.4
Permethrin	1.04	96	92	0.2
p,p'-DDE	1.02 ± 0.06	104	85	0.006
p,p'-DDT	1.01 ± 0.08	105	91	0.003

<sup>1</sup> Ratios = (Results of SFE method)/(Results of solvent extraction method); <sup>2</sup> SD, standard deviation; <sup>3</sup> SFE method; <sup>4</sup> Solvent extraction method.