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GC/MS/MS를 이용한 다종농약 다성분 분석시 분석체 보호제 D-sorbitol에 의한 매트릭스 효과 개선

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Reduced Matrix Effect by D-sorbitol as an Analyte Protectant in Multi-class Pesticide Multi-residue Analysis Using GC/MS/MS

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Abstract

The matrix effect of 235 pesticides in seven agricultural products of green pepper, mandarin orange, potato, perilla leaf, coriander, crown daisy, and chamnamul on simultaneous analysis of agricultural pesticide residues using GC/MS/MS were confirmed. D-sorbitol, as an analyte protectant(AP), was added in the final testing solution to reduce the matrix effect and to improve the reliability of the quantitative analysis. As D-sorbitol was added, the matrix effect %(%ME) of pesticides were decreased to about 1/10 in mandarin orange, about 1/3 in perilla leaf, coriander, green pepper, potato and chamnamul(*Pimpinella brachycarpa*). In addition, the repeatability and the linearity of calibration curves were increased with the addition of D-sorbitol. In most pesticides, the relative standard deviation %(%RSD, n = 3) of calibration curves were less than 10%, and the R^2 value were more than 0.99.

Key words : pesticide, GC/MS/MS, matrix effect, analyte protectant, D-sorbitol

서 론

MS/MS를 이용한 잔류농약 분석법은 한번에 여러 종류의 농약을 미량까지 분석할 수 있는 장점이 있어 최근 농산물의 잔류농약 분석에 많이 이용되고 있다. 그러나, 분석시료의 매트릭스 효과(matrix effect)에 의한 간섭현상이 문제가 된다. 특히, GC를 이용한 잔류농약 분석시 분석체(농약)가 분석과정 중 열에 의해 분해되거나, GC liner 또는 column의 활성부위와 상호작용하여 검출신호가 감소하거나 피크 tailing이 발생하여 감도와 재현성이 감소하기도 한다. 그런데, 농약과 농산물 시료 성분(매트릭스)이 공존할 경우, 매트릭스가 GC의 활성부위에 먼저 결합해 농약의 흡착 또는 분해를 막아 검출량이 증가하거나 감소할 수 있는데, 이러한 현상을 매트릭스 효과라고 한다(1~5). 또한 특정 농산물 시료 중에는 농약과 비슷한 product ion을 생성할 수 있는 물질이 존재하여 MS/MS 분석 결과에 영향을 미칠수도 있다.

이런 매트릭스 효과에 의한 분석상의 오차를 개선하기 위한 방법으로 시료와 동일한 무처리 시료의 추출물을 이용한 검량법(matrix-matched calibration)(4, 6), 통계기법을 활용한 검량선의 보정(7), 분석체 보호제(analyte protectant, AP)를 첨가하는 방법(8) 등이 연구되고 있다. 이들 중, AP는 3-ethoxy-1,2-propanediol, gulonolactone, sorbitol, shikimic acid 등이 많이 이용되고 있으며, 단독으로도 사용하지만 이들을 2~3가지 혼합하여 사용하기도 한다(9~11). 올리브유(12), 고춧잎 추출물(5) 등 천연물질도 연구되고 있다. AP는 일반적으로 최종 시험용액에 첨가하지만, GC carrier gas에 첨가하는 방법(13), 표준품과 시료를 주입하기에 앞서 AP를 먼저 주입하여 GC 주입구나 컬럼의 활성부위를 masking하는 방법(14) 등이 보고되었다.

본 연구는 235종 농약을 GC/MS/MS를 이용하여 식품공전 다종농약 다성분 분석법 제2법에 따라 분석할 경우의 주요 농산물별 매트릭스 효과를 측정하고, 여러 AP 중 가장 경제적이고, 손쉽게 이용할 수 있는 D-sorbitol을 이용하여 매트릭스 효과를 개선하고자 하였다.

재료 및 방법

1.농약 표준품 및 시약

농약 표준품은 AccuStandard(New Haven, CT, USA)로부터 구입하였으며, 약 500 μg/mL의 표준품을 혼합하여 표준용액을 만들고, acetone으로 적당 농도로 희석하여 -20°C 냉동고에 보관하며 한 달 이내로 사용하였다. Acetone은 Kanto chemical(Tokyo, Japan)로부터 구입하여 사용하였다.

분석체 보호제로 사용한 D-sorbitol은 Sigma-Aldrich(Steinheim, Germany)에서 구입하였다. D-sorbitol은 유기용매에 잘 녹지 않으므로 우선 소량의 증류수로 완전히 녹인 다음, acetone을 추가하여 100 mg/mL의 stock solution을 만들어 4°C에서 냉장보관하며 7일 이내로 사용하였다.

2. 기기분석 조건

GC-MS/MS 장비는 Thermo scientific사의 TSQ9000 모델을 사용하였고, 데이터 분석 소프트웨어는 Trace Finder 4.1을 사용하였다. 기기조건 및 MRM 조건은 각각 표 1과 2에 나타내었다.

3. 분석 시험용액의 제조

잔류농약이 검출되지 않은 농산물 시료 7종(고추, 감귤, 감자, 들깻잎, 쑥갓, 고수, 참나물)을 식품공전 제8. 일반시험법 7. 식품 중 잔류농약 분석법 7.1.2.2 다종농약 다성분 분석법에 따라 전처리하였다.

농산물 실험 재료 중 고추, 감귤, 감자는 '식품 등 시험법 마련 표준절차에 관한 가이드라인'에서 잔류농약 분석법 검증시 시험대상으로 권장하는 품목이며, 나머지 4종(들깻잎, 쑥갓, 고수, 참나물)은 다소비 농산물로 잔류농약 부적합 비율이 높으면서, 강한 휘발성 향미성분을 포함하고 있어 다른 농산물에 비해 복잡한 매트릭스 효과를 나타낼 것으로 생각되어 시험 대상으로 선정하였다.

이렇게 얻은 시료 추출액을 원래의 분석 농도보다 2배로 농축한 다음, 여기에 농약 표준용액을 적당 농도가 되도록 첨가한 후, acetone을 추가하

여 매트릭스 농도가 식품공전 시험법의 농도와 같아지도록 시험용액을 만들어 GC/MS/MS로 분석하였다.

대조군으로 순수한 acetone으로 희석한 표준용액을 함께 분석하였다.

25, 50, 100, 250 ng/mL의 농도로 각 농약별 검량선을 그리고, 일주일 간격으로 3회 반복 실험한 검량선의 평균기울기로부터 다음 식에 따라 matrix effect %(%ME)를 계산하였다. 시험용액 중 25~250 ng/mL의 농도는 식품공전 시험법에 따른 시료 전처리 과정의 농축배수를 감안하여 설정한 것으로, 농산물 시료 중의 농도로 환산하면 0.01~0.1 mg/kg에 해당한다.

$$\%ME = ((A-B)/B) \times 100$$

여기서 A는 시료 매트릭스를 포함한 추출액으로 제조한 표준물질 검량선의 기울기이고, B는 순수 유기용매(acetone)로 제조한 표준물질 검량선의 기울기이다(5).

D-sorbitol 첨가에 따른 개선 효과를 분석하기 위해 매트릭스 효과 분석 때와 같은 방법으로 표준용액 및 시험용액을 제조하고, 여기에 D-sorbitol을 10 μg/mL 농도로 첨가하여 GC/MS/MS로 분석하였다.

결과 및 고찰

1. 농약별, 농산물별 매트릭스 효과

235종 농약의 농산물별 매트릭스 효과는 Table 3과 같다. Chlorfluazuron을 제외하고는 모두 양의 값을 나타냈는데, 이는 농산물 분석시료 중의 성분이 GC의 활성부위에 먼저 결합하여 농약의 흡착 및 분해를 막아 피크의 강도가 증가했기 때문으로 생각된다. 7종의 농산물 중 참나물이 가장 큰 매트릭스 효과를 보였으며, 감귤, 깻잎, 쑥갓, 고수, 풋고추는 서로 비슷하였고, 감자가 가장 작았다.

박(5)은 QuEChERS EN법으로 전처리한 후 GC/MS/MS로 분석한 100종 농약의 5종의 농산물에 대한 매트릭스 효과를 보고하였는데, 그 중 감귤, 현미, 고추, 대두는 전반적으로 (+)의 매트릭스 효과를 보인 반면, 감자는 (-)의 매트릭스 효과가

Table 1. Instrumental conditions for multi-residue pesticide analysis

GC Conditions	
GC System	Thermo scientific Trace 1310
Column	TG-SQC, 15 m × 0.25 mm, 0.25 μm film
Oven program	Initial 70 °C, hold 3 min, then 15 °C/min to 160 °C, next 5 °C/min to 300 °C, and hold for 3 min
Injection volumn	2 μL(splitless)
Carrier Gas	Helium(1 mL/min)
Total running time	40 min
MS/MS Conditions	
MS/MS System	Thermo scientific TSQ9000
Ionization mode	EI mode
Transfer line temperature	280 °C
Ion source temperature	300 °C
Collision gas	Argon

더 많았다고 보고하였다. %ME의 크기는 본 연구보다는 작게 보고하였는데 이는 정제 등 시료 전처리 방법이나 기기분석 조건에서 온 차이로 생각된다. QuEChERS 전처리법도 사용하는 정제키트나 용매, 그리고 농약에 따라서 차이가 있는데, %ME는 대체적으로 -60~100% 수준으로 보고하였다(16~18).

김(7)은 GC를 이용한 정량분석시의 유기염소계 11종과 유기인계 6종 농약의 깻잎 등 12가지 농산물에 대한 %ME를 보고하였는데, 본 연구와 동일한 전처리법, 농산물(깻잎, 풋고추, 감자), 매트릭스 농도(2.5 g/mL), 농약 잔류 농도(50~100 ng/kg)에서의 %ME를 비교해보면, GC/MS/MS로 분석한 본 연구의 %ME는 김(7)이 GC-NPD나 GC-ECD를 이용한 분석한 것보다 procymidone, chlorfenapyr, endosulfan, bifenthrin, diazinon 및 kresoxim-methyl은 약 1~3배, fenitrothion과 EPN은 약 3~6배 높은 것이다. 이렇게 장비와 농약에 따라 %ME가 크게 차이가 나는 이유는 명확히 알 수 없으나, 매트릭스 효과에 영향을 주는 것으로 알려진 여러 요인들 중 분석장비와 관련된 부분, 예를 들어 liner나 column의 활성부위나 금속이온, ion source 등의 금속, 시료 주입구나 컬럼의 온도, 분석시간 등이 영향을 미쳤을 것으로 생각되며, 농약의 화학적 구조나 열안정성 등에 따라 그 영향의 정도가 달라질수 있을 것이다.

AP로 D-sorbitol을 첨가한 경우 %ME가 대부분 감소하였는데, 감귤은 약 1/10 수준으로 감소하였으며, 깻잎, 고수, 풋고추, 감자, 참나물은 약 1/3 수준으로 감소하였다. 다만, 쑥갓은 AP 첨가에 따라 %ME가 감소한 농약도 많았지만, 증가한 농약도 많았다. 235종 농약 대부분 AP 첨가에 따라 %ME가 감소하였으나, lactofen은 풋고추를 제외하고는 나머지 6종 농산물에서 %ME가 크게 증가하였으며, chinomethionat도 7종 농산물 모두에서 %ME가 증가하였다.

그림 1은 AP 무첨가군과 첨가군의 각 농산물별 235종 농약의 %ME 분포를 나타낸 그림이다. AP를 첨가하지 않은 경우 500 이상의 %ME를 보인 농약이 다수 있었던 반면, AP을 첨가한 경우는 매트릭스 효과가 크게 감소하여 대부분 농약의 %ME가 0~300 사이에 분포함을 확인할 수 있었다.

2. 검량선의 반복 재현성 및 직선성

현재의 식품공전 다종농약 다성분 분석법은 유기용매에 녹인 외부표준물질로 작성한 검량선으로 정량을 하게되는데, 이때 검량선의 기울기는 표준용액 중에 존재하거나 또는 분석장비에 흡착된 물질로 인한 매트릭스 효과로 그 기울기가 달라질 수 있으며, 또한 GC injector 등 분석장비 자체가 갖는 반복오차 등에 의해서도 달라질 수 있다. 4-point로 작성한 1회 검량선의 기울기는 일내 반복성을, 일주일 간격으로 3회 반복 측정한 기울기의 상대표준편차는 일간 재현성을 포함하므로, 일주일 간격으로 3회 반복 측정한 검량선 기울기의 상대표준편차 %(relative standard deviation %, %RSD)를 구하여 AP 무첨가군과 첨가군의 반복 재현성을 비교하고자 하였다.

각 농산물별로 235종의 농약을 %RSD에 따라 0~10 미만, 10~20 미만, 20 이상, 이렇게 세 구역으로 나누어 비교한 결과, AP 무첨가군은 20 이상의 %RSD를 보인 농약이 다수였던 반면, AP 첨가군의 %RSD는 10 미만이 대부분을 차지하여, AP 첨가에 따라 분석값의 반복 재현성도 증가한 것을 확인할 수 있었다(그림 2).

그리고, AP 첨가에 따라 검량선의 직선성도 증가하였는데(표 4), AP 첨가시 검량선의 R^2값은 대부분이 0.99 이상의 양호한 직선성을 보였다. AP 무첨가 시료의 검량선의 R^2값은 0.99 미만이 많았는데, 검량선의 중간 농도에서 농도대비 감응비가 감소하여 직선성이 감소하였기 때문이다.

이렇게 AP 첨가시 검량선의 반복 재현성이나 직선성이 증가한 이유는 AP가 GC 장비의 활성부위를 masking하여 분석장비의 상태에 따른 영향을 더 적게 하기 때문으로 생각된다.

AP첨가에도 불구하고 azinphos-ethyl, chloridazon, lactofen, methoxychlor, pyraclofos, tolyfluanid, zoxamide 등 20여종의 농약은 %RSD가 20을 초과하였는데, 이들 농약 대부분은 검량선의 직선성도 낮게 나타난 것들이다. 즉, 검량선의 직선성이 떨어져 기울기의 반복오차가 크게 나타난 것으로 볼 수 있다.

Table 2. MRM conditions for multi-residue pesticide analysis by GC/MS/MS

Compound name	Isomers	Retention time	Quantifier			Qualifier		
			Precusor ion	Product ion	Collision energy	Precusor ion	Product ion	Collision energy
2,6-Diisoporpylnaphthalene		14.74	212	197	15	197	167	10
Aldrin		18.84	262.7	192.9	32	262.7	191	30
Allidochlor		9.01	132	56.1	8	138.1	95.9	6
Ametryne		17.55	227.1	58.1	12	227.1	170	10
Anilofos		27.35	225.9	184	6	225.9	157	14
Aspon		18.60	210.5	115	10	114.9	96.9	12
Atrazine		14.98	215.1	58.1	12	200	122.1	8
Azaconazole		22.59	217	172.9	14	218.9	174.9	14
Azinphos-ethyl		29.23	132	77	12	160	77	16
Benalaxyl		24.56	148.1	77	30	148.1	79	22
Benodanil		24.01	230.9	202.9	12	230.9	76	30
Benzoylprop-ethyl		26.24	105	77	12	105	51	28
BHC, Alpha		14.12	218.8	183	8	182.8	146.7	12
BHC, Beta		15.00	180.9	145	14	218.7	183	8
BHC, delta		16.13	218.8	182.9	8	182.8	146.7	14
BHC, gamma		15.29	180.9	145	14	218.7	183	8
Bifenox		27.40	172.9	137.9	16	341.1	281	12
Bifenthrin		26.85	181	165.9	10	181	179	12
Bromacil		18.19	204.8	187.8	12	204.8	162	14
Bromobutide		17.13	119.1	91.1	10	118.1	117.1	8
Bromophos-methyl (Bromophos)		19.48	330.8	315.8	14	328.9	313.8	14
Bromopropylate		26.78	340.8	185	14	184.9	75.5	30
Bupirimate		22.48	273.1	193.2	8	208.1	165	12
Butafenacil		30.92	331	180	16	180	124	16
Butralin		19.38	266.1	190.1	10	266.1	219.9	10
Butylate(Sutan)		10.55	156.1	57.1	8	146.1	90	8
Carbophenothion		24.66	157	45	12	199	142.9	10
Chinomethionat		20.99	234	206	8	206	148	12
Chlorbufam		15.00	223	127	12	223	53.1	16
Chlordane cis		21.44	372.8	265.8	20	376.6	268	20
Chlordane trans		20.91	372.8	265.8	20	376.6	268	20
Chlorethoxyfos		12.98	153	97	10	97	65	15
Chlorfenapyr		22.86	136.9	102	12	248.9	112	24
Chlorfenson		21.82	174.9	111	10	111	75	14
Chlorfluazuron		21.58	321	304	35	321	97	30
Chlorflurenol-methyl		20.68	215.1	152.1	22	152.1	151.1	16
Chloridazon		24.93	220.9	77	20	220	193	16
Chlorobenzilate		23.42	139	111	12	139	74.9	26
Chloroneb		11.49	206	190.9	12	190.9	113	14
Chloropropylate		23.42	139	111	12	251	139	14

Table 2. (*Continued*)

Compound name	Isomers	Retention time	Quantifier			Qualifier		
			Precusor ion	Product ion	Collision energy	Precusor ion	Product ion	Collision energy
Chlorpyrifos-methyl		17.14	285.9	93	20	286	270.8	10
Chlorthal-dimethyl (Dacthal)		18.90	300.7	222.9	22	300.7	272.9	12
Chlorthion		19.25	297	109	8	297	125.1	8
Chlorthiophos		23.84	324.9	269	12	296.9	268.9	8
Chlozolinate		20.16	331	259	8	259	187.9	12
Cinmethylin		17.72	105	77	20	123	81	10
Clomeprop		27.57	120.1	77.1	18	148.1	120.1	6
Cyanazine		18.85	198	91	10	198	157	8
Cyanophos		15.39	243	109	10	125	79	6
Cycloate		13.31	83.1	55.1	6	154.1	83.1	8
Cyhalofop butyl		28.43	256	120	10	256	91.1	24
Cyproconazole		22.97	222	125	20	222	82.1	10
DDD p,p		23.71	235	165.1	20	235	199	14
DDE p, p		22.25	246	176.1	28	317.8	246	20
DDT o,p		23.80	235	165.1	20	235	199.5	14
Demeton-O		12.87	88.1	59.8	6	89.1	61	8
Demeton-S (Disulfoton oxon)		14.60	114	81	14	170	114	8
Desmetryne		16.93	213.1	170.9	8	213.1	58.1	10
Diallate	1	14.28	234.1	150	18	235.8	152	18
Diallate	2	14.39	234.1	150	18	235.8	152	18
Diazinon		15.62	137.1	84.1	12	137.1	54.1	20
Dichlofenthion		16.95	279	223	12	222.9	205	12
Dichlormid		9.71	172	108.1	6	108.2	93	16
Dicloran(Bortran)		14.65	206	176	10	160	124.1	8
Dicofol		19.24	139	111	12	111	74.9	12
Diethatyl-ethyl		21.56	188.1	160.1	8	161.9	147.1	10
Diethofencarb		18.82	168	96.1	12	196	96	16
Diflufenican		25.71	266	246.1	10	394	266.1	12
Dimepiperate		20.47	119	91.1	10	145	112.1	8
Dimethachlor		16.95	134	105.1	12	197	148.1	10
Dimethenamid		16.97	230	154.1	10	154.1	111	10
Dimethoate		14.62	87	42.1	10	93	63	8
Dimethylvinphos		18.78	295	109	14	295	79	28
Diniconazole		23.53	268	232	10	268	136	34
Dinitramine		15.93	260.7	241	8	215.9	196	8
Dioxathion		15.25	125	97	6	96.9	65	16
Diphenamid		19.49	166.8	152	16	239.1	167.1	8
Diphenylamine		13.16	168.1	167.1	14	169.2	167.1	22

Table 2. (*Continued*)

Compound name	Isomers	Retention time	Quantifier			Qualifier		
			Precusor ion	Product ion	Collision energy	Precusor ion	Product ion	Collision energy
Dithiopyr		17.75	354	306.1	8	306	286.1	8
Edifenphos		24.73	172.9	109	8	172.9	65.1	30
Endosulfan alpha		21.45	240.6	205.9	14	194.7	125	22
Endosulfan beta		23.48	194.7	159	8	240.6	205.8	12
Endosulfan sulfate		24.83	271.7	236.8	12	238.7	203.9	12
Endrin		23.11	262.8	192.9	30	245	173	22
EPN		26.63	169	141	8	169	77	22
Epoxiconazole		26.04	192	138	12	192	111	22
EPTC		9.76	128.1	43.1	10	189.1	128.1	6
Esprocarb		18.47	222.1	91.1	14	222.1	162.2	6
Etaconazole	1	23.63	172.9	144.9	14	172.9	109	24
Etaconazole	2	23.66	245	191	8	190.9	172.9	12
Ethalfluralin		13.42	276	202	14	315.9	276.1	8
Ethion		23.74	153	97	10	230.9	128.9	22
Ethofumesate		18.25	161.1	105.1	10	207.1	137.1	10
Ethoprop(Ethoprophos)		13.24	157.9	96.9	16	157.9	113.9	6
Etofenprox		32.55	163.1	135.1	10	163.1	107.1	16
Etoxazole		27.14	140.9	62.9	26	204	176.1	10
Etridiazole(Terrazole)		10.82	211	182.9	10	182.8	139.9	14
Etrimfos		16.15	167.9	153.1	6	153.1	56	16
Fenamidone		27.17	268	180.1	18	268	77.1	34
Fenchlorfos		17.64	124.9	79	6	124.9	47	12
Fenfuram		16.06	201.1	109	10	109	43	14
Fenitrothion		18.13	277	260	6	277	109	16
Fenobucarb		12.81	121.1	77	20	150.1	121.1	8
Fenoxanil		23.07	189	125.1	12	293.1	155	20
Fenoxycarb		26.96	116	88	8	116	44.1	16
Fenson		19.41	141	77	8	141	50.9	30
Fenthion		18.87	278	109	18	278	169	14
Fipronil		20.05	366.9	212.9	28	368.8	214.9	30
Flamprop-isopropyl		23.49	105	77	12	105	50.8	28
Fluchloralin		15.69	306	264	8	264	206.1	8
Fludioxonil		21.92	248	127	26	153.7	127	8
Flufenphyr-ethyl		23.44	408	345	12	408	373.1	8
Flumetralin		21.32	143	107	18	143	57	34
Flumiclorac-pentyl		35.32	308	280	10	318	260	15
Flumioxazin		33.53	354.1	326.1	8	354.1	312	8
Fluopyram		20.20	173	145	20	173	95	35
Fluorodifen		21.76	190	126	8	190	75	20
Flurochloridone		19.27	187.1	159.1	10	145	95	15
Flurtamone		27.75	333.2	120.1	12	198.9	157	16

Table 2. (*Continued*)

Compound name	Isomers	Retention time	Quantifier			Qualifier		
			Precusor ion	Product ion	Collision energy	Precusor ion	Product ion	Collision energy
Flusilazole		22.41	233	164.9	16	233	151.9	14
Flutolanil		21.83	173	145	14	173	95	28
Flutriafol		21.59	123	95	12	123	75	24
Fonofos		15.53	137	109	6	246	137	6
Fosthiazate		19.56	194.9	102.9	8	194.9	139	6
Fthalide		19.28	243	215	20	272	243	10
Halfenprox		31.94	263	115.1	18	264.8	237	12
Heptachlor		17.64	99.8	65	12	271.8	236.9	12
Heptachlor epoxide		20.16	352.8	262.9	16	262.9	192.9	30
Heptenophos		12.31	124	89	12	124	62.9	28
Hexachlorobenzene		14.41	283.8	248.8	18	283.8	213.8	30
Hexaconazole		21.89	231	175	10	213.9	159	18
Indanofan		27.29	159	103	15	174	131	15
Iprobenfos		16.44	203.9	91.1	8	91.1	65	16
Iprovalicarb	1	22.68	134.1	42	20	118.9	117.1	8
Iprovalicarb	2	22.75	118.9	117.1	8	134.1	42	20
Isazophos		16.00	161	119	8	161	146	6
Isofenphos		20.20	213	121	14	185	121	10
Isopropalin		19.69	280.1	238.2	8	280.1	180.2	10
Isoprothiolane		21.99	204	118	8	290	118	12
Kresoxim-methyl		22.53	116	89	14	130.9	130.1	10
Lactofen		28.81	343.9	223	14	223	131.8	20
Leptophos		28.00	171	77.1	18	171	51	38
Malathion		18.51	173.1	99	12	92.8	63	8
Mecarbam		20.31	159	131	6	131	42	12
Mefenacet		28.41	192	136	14	192	109	26
Mefenpyr-diethyl		26.17	299	253	10	253	189.3	22
Mepronil		24.24	119	91	12	119	65	22
Metconazole		27.42	125	89	16	125	99	18
Methidathion		20.78	145	85	6	85	58	5
Methoprotryne		22.61	256.1	212.1	12	256.1	170.1	20
Methoxychlor		27.05	227.1	169.1	22	227.1	141.1	32
Methyl pentachlorophenyl sulfide		18.34	295.8	263	15	295.8	246	20
Methyl trithion		23.35	125	47	15	157	75	40
Metolachlor		18.67	162.1	132.9	14	238.1	162.2	10
Metrafenone		29.32	393	362.7	16	393	346.9	20
Metribuzin		17.12	198	82.1	16	198	110	10
MGK-264	1	19.90	164	93.1	10	164	98.1	10
MGK-264	2	19.99	164	67.1	8	164	98	10
Molinate(Ordram)		12.00	126.1	55.1	12	187.1	126.1	6
Myclobutanil		22.37	179	125	14	179	90	28

Table 2. (*Continued*)

Compound name	Isomers	Retention time	Quantifier			Qualifier		
			Precusor ion	Product ion	Collision energy	Precusor ion	Product ion	Collision energy
Napropamide		21.71	128.2	72.1	6	100.1	72.1	6
Nitrapyrin		10.80	194	133	15	196	135	15
Nitrothal-isopropyl		19.31	236	194	8	194	148	10
Nuarimol		25.43	139	111	14	235	139	14
Ofurace		24.30	232.1	158.1	18	131.9	117	16
Oxadixyl		23.55	163.1	132.1	8	131.9	117	16
Paclobutrazol		21.19	236	125	12	125	89	18
Parathion(ethyl)		19.00	109	81	10	291	109	12
Parathion-methyl		17.33	263	109	12	263	79	25
Pebulate		10.92	128.1	57.1	8	161	128.1	10
Penconazole		20.08	248	157	22	248	192	12
Pentachloroaniline		16.69	264.8	193.6	18	264.8	202.8	20
Pentoxazone		28.01	284.8	70	10	286.8	70	10
Permethrin	1	30.51	183.1	168	12	183.1	153	12
Permethrin	2	30.51	183	168.1	10	183	153	14
Phenthoate		20.42	274	121	10	246	121	8
Phosalone		27.96	182	111	14	121.1	65	10
Phosmet		26.63	160	76.9	22	160	133	10
Phosphamidon		16.85	127	109	12	264.1	127	12
Picolinafen		26.89	238	145.1	22	145	95	12
Picoxystrobin		21.51	145.1	102.1	25	145.1	115.1	15
Pirimiphos-ethyl		19.52	304	168.1	12	318.1	166.1	12
Pirimiphos-methyl		17.86	290.1	233	8	290.1	125	20
Probenazole		15.21	159.1	130.1	8	130	77	25
Procymidone		20.56	95.9	67.1	8	95.9	53	16
Profenofos		22.10	336.9	266.9	12	296.7	268.9	10
Profluralin		15.31	318.1	199	15	330.2	69.1	20
Prometon		14.83	210.1	168.1	10	225	168.1	10
Pronamide(Propyzamide)		15.54	173	145	15	175	147	15
Propachlor		12.86	120	77	15	176.1	57.1	10
Propazine		15.09	214.1	172.1	8	229.1	58.1	12
Propetamphos		15.37	138	110	10	138	64	15
Propham		10.89	137	93	8	92.9	65.9	12
Propisochlor		17.38	162.1	120.1	12	162.1	144.1	8
Prothiofos		21.96	266.7	238.9	8	308.9	239	14
Pyracarbolid		19.52	125	43	20	125	55.1	12
Pyraclofos		29.57	194	138	18	139.2	96.9	6
Pyrazophos		29.05	221	193.1	8	231.9	204.1	10
Pyridaben		30.26	147.1	117.1	20	147.1	119.1	8
Pyrifenox	1	21.00	227	192.1	12	227	200	12
Pyrifenox	2	21.11	262	227	10	262	200	14

Table 2. (*Continued*)

Compound name	Isomers	Retention time	Quantifier			Qualifier		
			Precusor ion	Product ion	Collision energy	Precusor ion	Product ion	Collision energy
Pyrimidifen		33.32	184	169	18	185.8	171	18
Pyriminobac-methyl-E		24.95	302	256.1	16	302	229.8	16
Pyriminobac-methyl-Z		23.44	302	256.1	16	302	229.8	16
Quinalphos		20.45	146	118.1	10	157.1	102	22
Quinoxyfen		24.82	237	208	26	271.8	237.1	12
Quintozene		15.15	213.8	178.9	14	213.8	141.9	28
Secbumeton		16.04	196.1	85.1	10	196.1	68.7	20
Simeconazole		17.37	121	101	12	121	74.9	24
Spiroxamine	1	18.26	100.1	72.1	8	100.1	58	10
Spiroxamine	2	18.27	100.1	72.1	8	100.1	58	10
Sulfotep		13.78	202	145.9	10	322	202	10
Sulprofos		24.31	156	141	14	156	108	30
Tebufenpyrad		27.37	276.1	171	10	318.1	131.1	14
Tebupirimfos		16.39	261	137.1	15	233.8	126	10
Tefluthrin		16.09	177	127	14	177	137	16
Terbacil		15.96	160	117	8	161.2	144	12
Terbufos		15.41	230.9	128.9	22	230.9	203	8
Terbumeton		15.14	169.1	154.1	8	225.1	169.1	6
Terbuthylazine		15.41	229	173	5	214.1	132	10
Tetrachlorvinphos		21.17	328.9	109	18	330.8	109	18
Tetraconazole		19.13	100.9	51	10	336	204	28
Tetradifon		27.74	159	131	10	159	111	20
Tetramethrin	1	26.91	164	107.1	12	164	77.1	24
Tetramethrin	2	26.94	164	107.1	12	164	77.1	24
Tetrasul		24.22	251.9	181.9	32	251.9	173	34
Thiazopyr		18.60	327.1	277.1	26	306	291	10
Thifluzamide		22.35	194	166	10	166	125	15
Thiometon		14.47	88	60	6	158	125	5
Tolclofos-methyl		17.37	265	250	12	265	219.9	20
Tolfenpyrad		35.88	383.1	171.1	20	145	117	12
Tolyfluanid		20.18	137	91.1	18	238	137	10
Triadimefon		19.11	208	180.8	8	208	111	20
Triadimenol		20.56	168.2	70	10	128	65	18
Triazophos		24.26	161	134.1	8	162.1	119.1	12
Tribuphos		22.36	202	147	6	169.3	57.1	6
Triflumizole		20.66	206	179	14	206	186	8
Triflumuron		10.83	138.9	111	15	138.9	75	35
Trifluralin		13.65	306.1	264.1	8	306.1	206	10
Uniconazole		22.24	234	165	10	234	137	16
Vernolate		10.76	128.1	43.1	8	128.1	41.1	16
Vinclozolin		17.27	198	145	14	186.8	124	18
Zoxamide		26.09	186.9	159	14	258	187	10

Table 3. Matrix effect(%) of 235 pesticides analyzed without or with analyte protectant D-sorbitol in seven agricultural products

Compound	Matrix effect(%)													
	Mandarin orange		Perilla leaf		Crown daisy		Coriander		Green pepper		Potato		Chamnamul	
	without AP	with AP	without AP	with AP	without AP	with AP	without AP	with AP	without AP	with AP	without AP	with AP	without AP	with AP
2,6-Diisoporpylnaphthalene	193.9	-22.6	156.7	16.4	148.9	337.7	145.6	26.0	236.1	162.0	101.7	50.4	364.7	27.9
Aldrin	81.7	-11.7	60.5	2.6	62.2	287.0	49.3	2.0	186.1	130.8	78.7	48.9	289.0	3.5
Allidochlor	607.7	15.1	670.3	58.1	710.9	297.1	733.5	64.6	641.1	157.7	297.9	47.3	849.6	58.1
Ametryne	245.6	-29.9	268.6	29.8	292.1	328.1	300.9	33.4	390.6	143.8	160.2	44.8	597.3	23.6
Anilofos	2023.9	307.3	1974.4	745.8	2161.8	1267.6	2032.8	837.2	1501.3	329.8	614.0	183.6	1863.7	746.0
Aspon	214.9	2.5	244.0	75.7	263.0	530.1	260.1	88.2	318.4	210.0	120.0	56.3	555.1	94.8
Atrazine	305.4	-13.1	330.8	37.1	359.5	432.3	357.4	52.5	394.9	187.8	172.1	66.2	632.1	39.3
Azaconazole	267.5	-14.5	289.9	36.7	345.6	385.1	328.6	45.7	449.8	142.6	211.0	66.1	595.9	53.2
Azinphos-ethyl	2353.2	273.1	2437.3	1302.7	3174.6	2930.8	2149.2	1413.7	2282.4	417.9	729.7	203.9	2744.9	1271.1
Benalaxyl	256.2	-20.9	278.4	59.0	322.6	383.2	306.2	69.8	384.6	134.1	196.3	68.5	599.9	75.4
Benodanil	715.0	29.0	946.6	110.1	1103.9	687.6	1142.3	138.4	928.6	200.0	426.2	91.3	1443.5	128.6
Benzoylprop-ethyl	237.6	1.8	247.1	49.7	288.9	283.9	250.9	54.0	350.9	107.8	207.1	66.6	496.7	59.3
BHC, Alpha	223.4	-5.6	214.5	35.9	237.8	469.3	249.2	63.5	326.5	208.5	136.6	79.3	503.6	47.6
BHC, Beta	160.3	-10.9	156.0	11.6	188.9	470.4	175.2	39.0	319.7	214.4	131.1	87.4	494.0	41.5
BHC, delta	189.7	87.0	199.6	146.7	220.9	787.8	194.7	208.0	313.7	329.1	133.0	162.9	492.7	139.0
BHC, gamma	202.5	-0.6	192.5	25.1	233.0	503.3	215.6	64.7	332.0	215.2	144.6	95.3	522.7	64.5
Bifenox	581.7	114.8	664.1	386.8	875.4	1298.8	836.6	514.2	1116.6	378.4	527.1	227.5	1526.2	442.6
Bifenthrin	250.8	-7.4	229.7	68.4	246.2	422.9	236.7	78.4	375.2	163.6	205.3	88.0	556.9	82.0
Bromacil	620.8	-9.5	790.9	79.2	891.6	465.4	944.8	85.4	762.2	154.4	316.0	47.2	1249.7	78.6
Bromobutide	221.5	-6.9	150.5	13.4	225.2	298.8	114.1	-1.9	301.1	118.4	160.6	54.2	499.4	49.8
Bromophos-methyl (Bromophos)	218.9	4.8	182.5	31.9	210.8	574.5	197.4	43.3	413.9	198.3	162.7	76.9	643.0	43.8
Bromopropylate	201.7	-0.6	171.7	12.9	207.7	499.1	211.5	20.7	615.7	192.0	309.3	97.5	904.4	20.3
Bupirimate	207.8	-8.0	209.1	27.9	233.0	430.3	213.3	30.4	439.5	174.5	199.3	78.0	376.7	-3.6
Butafenacil	933.9	220.2	989.9	328.4	1143.1	1437.2	1216.0	400.0	1778.3	527.1	842.4	301.2	2564.2	335.9
Butralin	200.5	-7.4	182.6	59.1	215.4	593.5	202.6	68.8	428.2	209.0	173.7	86.3	754.7	66.6
Butylate (Sutan)	283.9	0.7	251.4	44.1	233.0	312.7	234.7	48.8	305.8	177.1	120.1	44.1	444.3	50.9
Carbophenothion	339.8	16.2	347.4	104.5	421.6	532.9	389.3	129.5	443.6	172.0	219.3	97.5	716.6	136.4
Chinomethionat	327.4	576.3	350.8	596.9	349.1	2069.8	381.8	1145.8	313.4	967.5	130.5	438.6	515.6	774.7
Chlorbufam	1485.0	18.4	2290.8	159.2	2855.5	899.3	2861.3	206.7	1573.7	229.6	574.3	89.6	3084.9	151.1
Chlordane cis	72.5	-17.9	34.4	-12.9	53.7	396.3	47.4	-8.8	259.6	175.4	110.2	67.2	385.3	-11.0
Chlordane trans	73.6	-14.6	38.9	-15.1	50.9	441.2	43.7	-12.9	262.0	192.1	106.7	72.7	394.0	-11.3
Chlorethoxyfos	340.5	-18.4	319.4	49.6	364.6	507.7	346.5	112.1	411.9	174.0	192.1	74.9	661.7	127.6
Chlorfenapyr	226.4	20.9	232.2	54.2	280.4	314.7	256.7	63.3	338.1	125.4	182.7	71.1	441.0	74.2

Table 3. (*Continued*)

Compound	Matrix effect(%)													
	Mandarin orange		Perilla leaf		Crown daisy		Coriander		Green pepper		Potato		Chamnamul	
	without AP	with AP	without AP	with AP	without AP	with AP	without AP	with AP	without AP	with AP	without AP	with AP	without AP	with AP
Chlorfenson	286.9	-29.6	330.1	46.1	369.2	346.3	364.4	50.7	310.4	104.1	167.7	57.0	557.3	52.2
Chlorfluazuron	27.8	-55.4	-6.0	-71.8	-46.7	-70.4	45.1	-60.4	75.9	-17.7	22.0	5.4	23.7	-80.0
Chlorflurenol-methyl	337.7	-9.0	398.2	41.2	477.3	427.2	449.5	55.4	604.1	163.5	297.6	74.7	942.5	62.4
Chloridazon	3108.6	42.6	9838.8	196.9	9051.1	622.1	12443.3	208.9	2462.1	108.9	1019.6	47.8	3428.9	121.3
Chlorobenzilate	168.7	-15.9	173.9	21.7	205.8	282.1	197.6	27.6	346.8	109.3	181.7	52.4	496.5	29.6
Chloroneb	494.8	98.8	318.2	-64.0	566.4	1611.1	371.6	-19.6	855.2	482.2	395.9	120.9	1587.4	44.1
Chloropropylate	168.7	-15.9	173.9	21.7	205.8	282.1	197.6	27.6	346.8	109.3	181.7	52.4	496.5	29.6
Chlorpyrifos-methyl	279.1	1.5	142.9	19.8	185.6	485.8	76.6	-22.7	376.9	182.6	189.6	74.1	681.6	44.8
Chlorthal-dimethyl (Dacthal)	134.4	-15.0	127.2	3.0	141.3	387.8	139.7	9.2	310.2	155.8	125.6	49.1	492.4	8.4
Chlorthion	666.5	58.4	657.5	123.3	911.4	970.1	709.9	162.0	1217.1	229.2	411.1	100.4	1866.7	151.8
Chlorthiophos	205.9	-5.4	179.4	37.3	206.3	508.7	206.2	49.9	415.1	197.1	187.3	93.8	598.3	48.0
Chlozolinate	162.8	155.4	132.2	172.8	169.0	930.8	73.8	134.6	419.4	424.3	179.7	208.0	553.6	174.5
Cinmethylin	134.4	-16.8	132.1	20.1	130.9	191.6	129.4	21.5	173.8	85.4	89.1	38.2	269.5	18.3
Clomeprop	359.4	17.2	340.7	71.7	390.5	353.4	389.6	86.4	456.8	136.3	265.0	85.2	623.7	85.1
Cyanazine	749.1	26.2	999.3	161.8	1160.5	635.7	1140.2	188.2	858.1	195.9	358.8	85.2	1440.7	148.7
Cyanophos	477.1	4.1	536.6	60.2	586.5	610.3	367.8	14.7	617.4	208.3	259.3	77.7	998.7	70.3
Cycloate	220.1	5.9	199.9	26.9	195.3	295.7	210.3	29.6	243.3	142.2	115.4	49.9	400.6	29.3
Cyhalofop butyl	1267.3	12.3	1462.2	123.4	1684.5	779.3	1642.2	147.3	1270.8	270.9	624.0	141.1	1956.4	144.0
Cyproconazole	297.5	-13.2	313.0	62.6	354.0	497.3	359.0	59.9	500.0	197.5	236.1	87.0	562.5	51.2
DDD p,p	158.5	-5.8	173.4	52.6	183.8	500.1	188.6	67.6	333.8	194.0	157.2	103.9	532.1	76.4
DDE p, p	101.3	-27.7	82.0	17.8	89.5	346.1	84.7	17.1	221.4	135.7	93.8	58.3	355.9	20.3
DDT o,p	158.5	-5.8	173.4	52.6	183.8	500.1	188.6	67.6	333.8	194.0	157.2	103.9	532.1	76.4
Demeton-O	700.4	19.6	705.9	88.2	772.6	411.3	751.2	105.4	504.6	125.6	225.5	46.4	706.3	85.6
Demeton-S (Disulfoton oxon)	517.9	-3.1	539.7	46.9	608.1	329.5	615.5	68.6	531.5	122.4	272.6	50.1	772.3	50.0
Desmetryne	264.6	-27.0	331.4	34.1	296.4	325.1	344.9	35.7	390.0	146.2	173.2	44.8	596.9	24.7
Diallate	225.7	-14.5	183.0	15.4	191.5	360.6	233.8	36.6	322.3	164.2	134.6	45.7	507.0	15.8
Diazinon	218.0	-13.0	228.8	33.9	242.5	293.5	274.3	59.3	282.0	105.0	133.2	36.8	488.9	37.9
Dichlofenthion	170.1	-11.3	167.2	21.5	162.6	387.2	169.3	24.2	320.9	174.0	135.3	57.4	509.0	15.2
Dichlormid	551.9	19.2	600.4	74.1	684.4	494.2	657.4	94.7	699.5	243.2	290.4	78.8	974.9	103.6
Dicloran (Bortran)	405.0	-2.7	498.4	60.7	584.2	512.8	593.2	89.4	569.9	177.0	246.4	66.3	918.8	60.3
Dicofol	265.2	-38.3	249.5	8.4	279.7	198.2	285.0	23.8	230.1	51.9	108.0	14.5	409.0	26.0
Diethatyl-ethyl	254.2	1.6	287.3	89.6	316.7	472.7	321.3	95.2	447.2	186.2	204.4	87.7	657.4	95.7
Diethofencarb	550.8	-32.6	703.4	41.9	830.0	337.0	784.0	48.5	672.8	106.8	300.8	33.4	1163.1	48.4
Diflufenican	374.1	-0.1	384.8	57.9	459.2	560.1	451.1	73.7	684.9	194.2	340.2	101.2	1052.7	73.5

Table 3. (*Continued*)

Compound	Matrix effect(%)													
	Mandarin orange		Perilla leaf		Crown daisy		Coriander		Green pepper		Potato		Chamnamul	
	without AP	with AP	without AP	with AP	without AP	with AP	without AP	with AP	without AP	with AP	without AP	with AP	without AP	with AP
Dimepiperate	252.2	-4.4	251.8	60.5	278.4	339.2	270.9	74.7	290.2	101.2	139.2	47.6	462.9	74.2
Dimethachlor	275.1	-21.6	323.6	57.4	322.9	375.7	332.8	61.1	378.5	159.3	172.1	58.5	594.3	56.9
Dimethenamid	219.7	-23.5	239.7	36.1	228.0	389.8	255.6	39.2	351.8	162.6	153.6	56.1	588.9	31.8
Dimethoate	1360.0	42.0	1600.4	137.0	1862.7	603.7	1711.3	185.4	1151.9	159.0	445.3	71.3	1484.1	157.7
Dimethylvinphos	1160.4	71.6	903.8	159.4	991.2	646.8	833.2	149.2	1230.9	174.3	442.4	63.8	1497.8	131.4
Diniconazole	307.7	0.3	294.2	46.4	352.7	505.6	351.5	50.2	628.2	191.2	299.8	89.5	967.2	52.3
Dinitramine	261.8	8.0	294.3	44.0	345.3	639.7	359.0	57.8	527.4	271.9	222.6	65.3	909.5	56.0
Dioxathion	239.8	12.4	269.9	53.9	305.1	339.7	307.0	74.6	331.5	145.1	163.9	68.7	530.6	67.1
Diphenamid	241.2	-37.3	274.2	33.1	307.5	328.6	303.3	37.8	348.4	122.8	150.1	43.8	566.6	47.1
Diphenylamine	330.0	-18.9	404.3	39.4	432.3	352.8	438.3	40.9	391.6	153.9	166.1	41.0	642.1	38.0
Dithiopyr	149.9	-26.2	151.3	-11.7	177.9	389.2	176.3	-7.4	426.4	173.8	173.2	50.1	649.1	-14.3
Edifenphos	2100.2	366.2	1775.3	1210.4	1928.1	1637.2	1539.9	1244.9	1690.1	300.8	594.8	185.3	1991.3	1095.0
Endosulfan alpha	83.4	14.8	74.9	41.8	86.5	432.3	76.6	47.6	205.6	214.5	94.1	94.0	299.2	53.3
Endosulfan beta	113.3	172.2	103.6	264.5	117.3	843.3	110.2	342.4	244.7	441.7	117.3	241.1	342.3	307.2
Endosulfan sulfate	219.1	-3.2	219.9	53.6	230.6	551.9	235.7	65.1	372.1	227.3	173.7	85.2	522.2	74.2
Endrin	118.6	-12.0	80.0	18.3	108.4	509.7	94.5	42.8	278.1	172.5	129.3	95.6	397.9	39.7
EPN	615.1	63.9	714.4	356.2	968.3	1215.3	861.1	412.2	1043.5	338.1	450.9	200.3	1703.6	381.3
Epoxiconazole	486.7	-9.3	487.3	112.7	601.2	778.4	534.5	164.5	795.7	248.1	398.4	142.0	1119.7	171.6
EPTC	295.7	12.0	263.8	41.0	254.9	236.5	253.4	40.6	338.5	139.3	155.4	36.5	453.1	44.9
Esprocarb	197.5	-26.2	176.9	23.5	186.5	353.7	178.8	29.6	282.5	152.0	113.3	55.2	458.8	29.5
Etaconazole	234.3	-10.5	233.1	41.3	280.2	374.8	267.2	43.8	437.6	144.6	218.5	67.8	609.3	44.9
Ethalfluralin	230.9	-4.8	239.7	41.6	136.2	537.2	280.9	51.9	495.1	220.5	196.0	63.1	838.9	54.0
Ethion	328.6	21.6	328.6	104.7	391.8	567.1	378.5	119.6	479.2	200.7	222.5	100.3	743.1	120.6
Ethofumesate	257.0	-33.2	311.1	29.2	349.7	359.2	326.9	32.8	440.1	147.9	185.0	49.5	696.6	28.2
Ethoprop (Ethoprophos)	442.7	-8.2	457.3	56.3	474.5	367.0	476.1	66.3	432.9	148.0	193.4	45.1	663.5	65.2
Etofenprox	619.0	17.6	633.0	181.1	663.8	598.3	669.8	202.5	629.2	217.6	335.9	136.2	960.8	195.3
Etoxazole	332.7	-4.0	308.7	83.6	339.1	470.8	355.9	111.5	430.8	174.9	222.2	90.2	654.0	100.8
Etridiazole (Terrazole)	587.6	-26.3	399.5	-6.6	556.8	280.0	431.1	39.8	641.0	70.7	265.8	19.3	720.3	13.8
Etrimfos	319.4	3.1	331.2	60.9	364.0	487.6	370.4	82.9	364.4	171.3	157.0	65.2	650.9	70.3
Fenamidone	217.2	-11.7	227.1	25.4	274.2	416.1	266.2	36.2	505.2	170.6	262.0	87.4	706.5	33.2
Fenchlorfos	273.8	13.5	295.7	71.0	319.6	325.9	294.2	83.2	304.9	110.3	141.1	50.2	481.0	75.1
Fenfuram	453.5	-27.9	547.1	12.8	594.6	298.5	636.0	19.2	376.2	118.1	190.4	51.4	718.4	8.3
Fenitrothion	582.7	24.0	619.4	84.7	803.1	709.5	690.2	100.4	1003.7	203.6	357.7	66.9	1656.1	86.7
Fenobucarb	766.7	141.8	1030.7	331.2	1093.2	724.2	1109.7	384.5	671.4	220.5	318.2	116.5	1038.6	200.0

Table 3. (*Continued*)

Compound	Matrix effect(%)													
	Mandarin orange		Perilla leaf		Crown daisy		Coriander		Green pepper		Potato		Chamnamul	
	without AP	with AP	without AP	with AP	without AP	with AP	without AP	with AP	without AP	with AP	without AP	with AP	without AP	with AP
Fenoxanil	1156.4	2.6	1673.6	281.8	1843.7	1094.8	1796.8	261.5	958.1	154.2	421.3	149.1	1513.3	159.4
Fenoxycarb	2928.6	41.8	5076.6	213.9	6585.6	692.3	5972.8	214.0	5274.4	173.8	2373.8	95.8	8776.6	233.7
Fenson	282.7	-28.3	326.0	27.7	365.2	262.2	357.9	35.4	331.3	86.0	153.3	31.0	551.3	35.8
Fenthion	277.4	-4.9	288.1	31.6	321.8	446.2	281.5	34.9	470.1	167.7	191.8	63.3	695.0	31.3
Fipronil	250.1	2.9	262.1	12.8	353.3	613.6	100.4	23.3	782.7	234.6	333.7	81.1	1255.3	35.1
Flamprop-isopropyl	196.8	-5.0	211.9	35.7	250.0	253.4	235.9	40.7	315.6	100.9	171.2	54.2	434.9	43.8
Fluchloralin	208.5	51.3	214.4	108.9	260.0	894.3	306.7	167.0	524.5	350.1	225.7	145.6	908.1	111.0
Fludioxonil	436.7	-21.2	537.1	37.4	542.8	387.3	593.4	41.0	617.5	137.0	289.5	60.1	858.8	30.6
Flufenphyr-ethyl	355.9	209.8	351.8	316.8	404.2	1524.1	400.9	363.6	870.9	566.3	404.9	292.4	1271.4	297.7
Flumetralin	252.1	89.2	306.6	307.3	224.7	655.9	356.7	341.0	444.8	256.4	213.0	144.0	755.0	338.7
Flumiclorac-pentyl	2980.4	1286.4	3491.5	3562.7	3820.0	6683.3	3702.4	4111.7	3223.5	1810.0	1442.2	890.7	5491.3	3374.5
Flumioxazin	2164.0	291.8	2372.8	567.5	2528.1	2413.4	2471.1	657.4	3062.3	706.1	1254.3	340.4	4928.6	578.4
Fluopyram	341.6	-27.9	397.2	32.0	460.4	368.5	1658.2	-10.2	507.5	122.0	246.7	53.3	744.1	47.6
Fluorodifen	589.1	201.9	751.4	806.3	1131.7	2278.7	920.0	978.8	1010.7	526.1	369.3	282.2	1639.5	887.1
Flurochloridone	427.2	58.7	464.7	191.2	607.8	853.1	200.8	262.2	673.4	257.1	281.5	144.4	1074.7	240.1
Flurtamone	1813.0	107.0	2147.5	158.5	2253.8	767.8	1920.3	159.8	1715.3	282.1	801.4	157.5	2557.2	152.7
Flusilazole	268.1	-19.6	313.4	48.4	359.0	423.8	340.3	49.9	504.5	162.0	236.2	67.9	543.3	44.8
Flutolanil	331.7	-21.5	416.4	59.7	491.6	410.0	462.5	63.7	495.2	134.1	239.4	70.5	804.4	69.2
Flutriafol	281.9	-2.4	372.7	44.9	427.7	272.7	410.4	49.2	519.3	102.0	270.8	53.9	746.8	48.6
Fonofos	264.4	-4.4	269.1	37.8	292.7	269.0	349.1	65.9	296.3	101.8	141.2	38.3	494.6	41.2
Fosthiazate	2170.2	136.4	1707.2	567.0	1846.8	910.7	1529.3	425.2	1747.8	158.7	553.3	73.5	2011.6	397.6
Fthalide	185.4	-12.5	197.3	15.1	229.1	346.3	217.4	21.6	327.7	131.5	137.9	53.5	497.7	13.8
Halfenprox	731.8	131.3	733.5	290.2	817.4	1416.0	803.6	343.5	1064.2	521.3	530.9	315.5	1710.2	411.5
Heptachlor	222.2	-23.2	157.7	16.3	212.8	482.2	178.0	69.7	389.4	129.0	163.0	48.9	614.8	59.3
Heptachlor epoxide	82.1	-16.2	50.9	-14.5	63.8	409.0	53.5	-17.2	280.6	179.0	121.3	73.2	390.1	0.1
Heptenophos	1229.7	26.3	1312.5	115.7	1440.3	409.0	959.1	75.2	943.1	136.9	431.7	53.1	1264.9	127.7
Hexachlorobenzene	71.9	-3.1	36.2	-8.0	36.2	407.6	48.5	10.4	201.4	198.1	73.0	58.9	319.3	-4.6
Hexaconazole	260.1	-18.4	275.2	48.7	320.0	439.0	300.4	50.4	448.3	160.4	218.2	76.3	742.4	51.2
Indanofan	455.9	24.2	441.3	225.0	484.8	997.6	395.6	202.7	467.2	256.2	225.3	143.4	672.4	225.0
Iprobenfos	724.9	-30.7	225.9	25.4	310.0	499.9	218.3	31.8	412.1	118.3	232.3	49.3	867.7	108.3
Iprovalicarb	393.4	-8.2	400.7	68.5	454.2	335.8	418.7	64.0	431.1	108.9	215.1	50.6	3626.3	154.9
Isazophos	289.9	-15.6	325.1	37.7	285.6	373.9	389.9	57.8	383.3	130.3	177.5	47.8	662.2	44.0
Isofenphos	233.3	-25.8	256.1	37.9	293.2	419.3	146.1	12.7	407.3	144.0	178.8	57.9	631.9	55.6
Isopropalin	159.4	-13.9	137.8	28.9	160.7	481.0	15.0	-28.4	373.6	190.4	160.3	73.4	624.7	33.4
Isoprothiolane	229.7	-35.0	246.7	41.5	288.7	372.6	271.8	42.9	379.2	141.8	171.1	62.9	599.6	47.6
Kresoxim-methyl	195.1	2.2	224.3	49.7	261.9	261.3	266.5	66.0	310.1	103.7	170.0	60.1	480.0	41.7

Table 3. (*Continued*)

Compound	Matrix effect(%)													
	Mandarin orange		Perilla leaf		Crown daisy		Coriander		Green pepper		Potato		Chamnamul	
	without AP	with AP	without AP	with AP	without AP	with AP	without AP	with AP	without AP	with AP	without AP	with AP	without AP	with AP
Lactofen	805.0	2785.5	874.1	5380.5	1201.6	6947.0	1159.3	7510.5	2041.1	1776.0	763.3	953.3	2740.2	5188.5
Leptophos	338.1	42.0	310.4	167.5	337.1	623.3	152.8	109.0	386.9	211.9	196.1	130.5	577.7	187.2
Malathion	853.6	194.3	1093.6	634.5	1173.9	1140.4	1140.6	722.3	837.6	319.1	347.1	166.9	1402.2	511.9
Mecarbam	507.9	169.6	565.0	402.8	662.5	1077.4	21049.4	817.9	585.8	338.4	263.0	186.5	858.4	369.6
Mefenacet	2198.3	33.7	2656.6	232.8	3190.9	868.9	3145.2	280.4	2041.6	243.7	929.1	121.6	2960.3	232.0
Mefenpyr-diethyl	269.4	59.7	243.8	120.6	295.4	768.1	236.9	128.9	612.1	319.1	313.1	181.1	881.5	132.6
Mepronil	575.0	11.9	666.4	87.3	765.9	382.6	762.5	108.1	624.0	134.3	333.4	71.1	890.3	109.3
Metconazole	363.9	18.7	371.8	83.6	437.3	335.3	429.5	101.4	506.0	122.6	310.3	76.1	691.2	97.2
Methidathion	848.4	70.2	932.7	239.4	1159.1	737.6	980.5	298.5	788.9	162.1	319.5	79.5	1174.5	302.9
Methoprotryne	264.9	-13.2	279.7	35.4	317.4	419.4	323.2	45.0	496.4	170.4	233.2	78.8	671.8	48.3
Methoxychlor	475.4	-77.4	230.3	28.7	503.9	906.7	299.8	307.8	866.4	87.1	450.7	126.4	1088.3	413.5
Methyl pentachlorophenyl sulfide	88.7	-16.3	50.0	-6.3	53.4	336.3	54.0	-2.9	205.3	163.0	77.9	57.0	312.7	-9.4
Methyl trithion	590.4	65.9	633.3	192.4	784.4	642.0	654.3	229.9	643.8	166.0	304.9	100.6	941.2	249.4
Metolachlor	211.6	-17.6	202.8	33.2	226.2	381.3	187.4	31.3	369.7	154.0	150.6	54.1	593.1	33.3
Metrafenone	215.2	-18.9	188.6	21.2	218.7	624.8	214.9	26.8	584.9	277.5	279.6	96.2	821.0	24.2
Metribuzin	311.2	-26.6	232.5	4.0	338.7	343.6	182.8	-18.2	433.5	143.6	213.9	53.8	729.8	36.3
MGK-264	159.2	-24.5	136.2	36.5	146.7	312.7	147.9	44.4	200.8	111.1	82.9	39.4	142.5	0.9
Molinate(Ordram)	262.4	-7.8	241.9	30.2	243.9	245.1	244.0	34.3	296.7	121.9	141.5	36.9	410.2	34.4
Myclobutanil	279.8	-22.9	315.9	49.8	363.7	354.9	345.1	53.7	448.6	137.1	216.5	60.2	659.4	56.8
Napropamide	268.9	-5.0	277.6	52.3	312.3	323.8	297.3	59.9	322.4	115.9	177.8	55.8	577.6	57.9
Nitrapyrin	737.1	-40.8	423.2	-33.6	722.3	322.7	508.8	0.4	694.4	42.6	268.4	9.6	914.0	6.6
Nitrothal-isopropyl	266.6	23.5	284.0	151.4	349.3	707.2	315.3	163.6	545.5	226.9	226.8	94.7	1000.7	169.7
Nuarimol	312.6	-3.5	341.3	58.5	351.2	351.3	379.0	70.3	525.2	137.2	288.4	79.2	726.4	67.2
Ofurace	498.6	60.7	545.2	219.2	620.7	837.6	585.3	267.5	771.2	305.3	361.6	174.4	1060.0	251.4
Oxadixyl	296.3	-13.7	312.7	35.4	363.2	346.9	348.9	42.7	536.5	150.6	270.7	74.4	673.1	45.7
Paclobutrazol	388.7	-19.3	448.6	55.8	521.1	442.7	494.1	62.1	675.2	162.9	308.6	64.7	1019.8	65.3
Parathion(ethyl)	374.6	6.1	463.2	72.6	594.2	328.8	543.6	86.6	596.6	99.6	290.4	50.5	953.0	84.6
Parathion-methyl	603.4	3.3	740.1	104.5	1027.8	707.0	888.4	141.5	978.0	195.7	355.5	69.5	1599.0	104.2
Pebulate	283.8	4.5	245.9	35.4	244.2	227.8	243.0	39.6	310.3	129.8	140.4	32.9	408.0	50.6
Penconazole	223.3	-25.6	221.7	24.5	254.6	410.0	240.4	26.2	411.3	154.4	176.4	57.2	576.2	41.1
Pentachloroaniline	142.2	-38.8	159.7	-1.7	161.7	323.7	162.0	3.1	253.6	148.5	109.1	32.1	448.0	-1.0
Pentoxazone	263.5	63.0	248.3	96.0	282.8	637.2	105.9	47.4	522.9	266.2	273.0	154.2	755.5	105.4
Permethrin	474.0	22.6	473.5	156.8	545.3	627.7	526.0	171.4	678.2	250.9	351.4	161.0	1023.9	178.1

Table 3. (*Continued*)

Compound	Matrix effect(%)													
	Mandarin orange		Perilla leaf		Crown daisy		Coriander		Green pepper		Potato		Chamnamul	
	without AP	with AP	without AP	with AP	without AP	with AP	without AP	with AP	without AP	with AP	without AP	with AP	without AP	with AP
Phenthoate	340.6	68.5	343.6	108.5	436.3	625.0	434.5	127.5	525.5	207.4	217.9	92.7	772.1	100.2
Phosalone	1334.2	984.3	1350.0	2277.1	1691.4	2955.5	976.2	2194.8	1285.9	765.2	582.7	480.4	1846.4	2362.4
Phosmet	2377.6	622.7	2359.9	1762.8	2825.9	2971.4	2359.4	2793.9	2166.8	666.4	749.6	339.5	2611.1	2034.4
Phosphamidon	2919.4	194.2	3154.1	785.1	3024.6	674.1	2727.1	687.1	2091.8	111.6	794.1	56.5	2884.0	596.9
Picolinafen	493.4	-1.3	513.5	81.2	591.4	556.0	468.9	79.2	637.0	183.1	323.7	89.2	1021.8	95.4
Picoxystrobin	236.1	-19.9	304.9	41.9	361.2	327.1	333.7	48.2	424.5	125.3	205.9	60.8	657.0	50.4
Pirimiphos-ethyl	143.9	-24.6	123.4	7.0	52.3	407.0	138.4	12.3	317.5	161.2	131.9	59.3	509.8	12.5
Pirimiphos-methyl	229.9	0.1	234.3	30.7	246.0	464.8	246.1	33.6	430.0	176.4	166.8	58.0	718.9	26.0
Probenazole	739.6	58.7	869.9	237.6	988.4	1271.3	1020.6	378.3	786.6	491.3	304.2	187.5	1244.2	245.6
Procymidone	184.8	-18.2	188.7	21.4	155.2	233.0	222.2	28.1	278.6	103.2	137.1	51.5	471.0	31.1
Profenofos	922.1	171.4	712.9	451.2	780.4	1394.0	644.3	443.5	1019.7	347.0	371.6	159.1	1374.6	403.9
Profluralin	180.4	25.3	157.6	34.1	182.7	624.7	199.0	43.8	451.9	259.7	197.1	96.1	793.3	35.9
Prometon	281.4	-31.6	308.0	26.5	319.8	386.2	351.6	40.2	380.4	166.4	166.3	49.5	618.0	29.4
Pronamide (Propyzamide)	255.7	-7.3	305.7	29.5	351.9	372.1	379.0	66.1	381.2	142.3	177.7	50.9	652.7	34.8
Propachlor	392.0	5.7	432.8	58.9	474.9	331.3	469.3	72.5	437.9	137.5	207.2	48.5	646.6	68.8
Propazine	252.7	-10.3	276.8	40.9	292.9	442.7	276.7	44.8	348.8	184.9	145.6	63.3	583.2	36.4
Propetamphos	308.2	-9.3	361.4	40.9	403.0	301.6	130.4	-30.4	394.8	106.3	197.1	40.7	656.3	46.7
Propham	577.8	-3.4	677.4	47.2	591.1	276.5	732.2	49.9	592.7	139.6	260.7	33.8	801.6	58.1
Propisochlor	242.7	-22.4	275.2	59.4	309.2	339.9	287.0	65.6	344.5	136.0	144.6	45.8	575.7	61.1
Prothiofos	149.7	8.5	128.1	29.7	136.0	432.5	136.3	33.7	263.2	172.3	117.6	73.5	420.4	34.6
Pyracarbolid	499.1	-21.5	621.2	44.7	687.4	258.9	416.8	24.0	452.1	75.5	222.4	31.4	718.4	50.5
Pyraclofos	7652.7	795.9	4567.4	4333.9	4918.8	5100.2	3092.8	3707.8	6926.1	687.8	1725.3	365.6	6906.6	3666.5
Pyrazophos	1344.9	134.2	1460.7	427.8	1679.0	1347.9	1562.1	496.6	1498.4	383.5	661.8	195.3	2141.4	475.3
Pyridaben	549.1	24.8	544.3	187.3	633.5	643.9	605.9	212.9	688.5	237.9	353.5	152.4	1086.1	214.0
Pyrifenox	249.6	-14.5	252.7	57.0	227.4	446.7	255.1	30.5	470.8	195.8	205.1	84.2	265.0	11.8
Pyrimidifen	1395.5	60.0	1583.5	336.9	1662.3	965.6	1584.7	366.4	1270.4	317.0	640.6	179.5	2107.1	386.0
Pyriminobac-methyl-E	260.5	-0.1	276.8	48.8	331.2	667.6	322.6	64.5	659.2	247.6	328.5	130.4	949.8	67.7
Pyriminobac-methyl-Z	215.4	-6.7	229.3	28.3	266.1	504.8	260.4	34.6	576.5	205.7	281.8	98.6	808.8	40.1
Quinalphos	308.4	2.8	333.0	76.3	382.8	398.7	386.4	96.2	371.7	122.4	169.7	57.4	574.5	96.2
Quinoxyfen	205.9	-6.4	194.4	43.7	208.2	482.7	213.3	55.6	369.2	184.7	171.6	91.0	540.1	57.6
Quintozene	172.8	-7.2	160.8	40.6	177.3	475.4	176.7	59.7	307.9	197.2	144.8	79.4	512.1	50.7
Secbumeton	298.3	-25.5	338.8	33.7	323.8	362.1	384.1	48.2	384.0	148.8	172.1	49.2	642.7	34.3

Table 3. (*Continued*)

Compound	Matrix effect(%)													
	Mandarin orange		Perilla leaf		Crown daisy		Coriander		Green pepper		Potato		Chamnamul	
	without AP	with AP	without AP	with AP	without AP	with AP	without AP	with AP	without AP	with AP	without AP	with AP	without AP	with AP
Simeconazole	304.4	-18.3	386.5	39.2	354.8	214.1	427.1	44.7	453.3	82.5	225.3	27.7	709.0	34.8
Spiroxamine	177.4	-47.5	181.0	56.7	192.5	201.7	171.7	61.5	71.8	76.4	8.3	27.5	145.8	48.5
Sulfotep	312.9	3.3	334.9	49.4	369.2	457.9	20.8	-59.2	470.2	195.4	195.3	60.0	747.2	42.7
Sulprofos	266.2	-13.2	266.9	64.6	306.9	428.9	297.3	80.8	358.1	146.6	175.4	76.8	533.8	75.7
Tebufenpyrad	275.8	-12.9	256.3	58.8	282.7	541.3	291.6	84.7	460.3	194.6	231.4	101.4	726.5	74.6
Tebupirimfos	178.4	-19.6	129.4	4.4	112.8	436.2	134.6	10.2	301.0	166.0	119.0	46.7	532.8	4.1
Tefluthrin	157.4	-24.7	145.9	14.3	152.5	310.8	163.5	26.8	239.8	123.4	113.7	46.8	413.7	15.6
Terbacil	850.2	-46.4	560.1	53.5	463.8	522.4	464.1	52.0	586.7	62.5	148.7	7.7	792.6	40.6
Terbufos	245.4	-5.0	237.0	44.2	245.9	461.8	159.8	12.2	360.4	178.4	157.4	62.1	612.9	52.8
Terbumeton	284.4	-24.6	310.8	34.0	314.3	345.6	338.3	44.6	363.0	153.6	163.1	51.7	577.5	43.9
Terbuthylazine	255.0	0.4	266.4	35.2	260.8	462.9	296.3	59.3	367.9	199.7	152.0	65.3	603.6	39.9
Tetrachlorvinphos	1193.0	139.4	914.2	278.9	963.7	910.5	780.5	258.0	1407.6	241.1	496.2	109.1	1584.9	258.4
Tetraconazole	214.7	-19.7	221.7	-5.0	269.0	368.7	273.1	-0.5	571.8	152.1	249.3	43.9	838.1	-4.3
Tetradifon	207.5	-21.0	195.9	47.7	228.0	351.1	217.4	63.4	315.7	122.9	160.8	70.9	480.7	51.5
Tetramethrin	597.5	180.9	629.8	445.2	713.9	1048.1	615.8	478.8	668.5	370.0	355.1	233.8	1038.9	462.2
Tetrasul	118.5	-25.1	93.2	20.2	97.1	393.6	99.1	30.7	218.7	148.4	102.2	68.3	348.7	31.8
Thiazopyr	168.3	-14.9	185.0	3.5	212.8	371.4	196.3	5.0	461.0	162.5	193.9	51.7	682.4	0.6
Thifluzamide	374.6	-1.7	464.8	55.1	542.8	406.8	518.2	60.3	679.6	149.6	330.2	72.4	1025.2	54.9
Thiometon	352.0	12.3	377.0	42.5	423.5	342.6	441.7	67.9	380.5	143.0	192.2	65.2	546.6	48.0
Tolclofos-methyl	202.9	-8.5	214.6	23.6	210.9	396.6	249.3	38.6	362.5	173.9	148.9	61.0	582.5	17.8
Tolfenpyrad	3090.5	173.0	3035.1	537.1	3304.2	1986.4	3209.9	581.6	2662.3	593.8	1132.8	282.5	4437.6	532.3
Tolyfluanid	623.0	99.9	635.3	285.9	825.8	1150.2	394.5	278.2	844.8	165.5	342.0	101.0	1333.5	490.8
Triadimefon	269.2	-29.0	309.8	24.3	345.5	339.3	334.8	30.5	428.4	132.5	193.3	42.6	675.1	29.4
Triadimenol	327.1	-16.7	368.9	38.1	377.3	331.3	412.5	48.2	544.0	128.8	269.3	56.1	829.2	48.1
Triazophos	966.4	53.0	1067.9	192.8	1306.2	670.9	1228.0	232.0	976.0	187.4	455.1	100.6	1393.5	220.0
Tribuphos	279.8	43.4	243.5	114.1	259.9	537.0	242.6	124.9	363.1	207.3	178.1	102.7	580.5	113.6
Triflumizole	260.7	-8.7	275.3	51.6	283.5	392.8	307.5	58.5	428.6	156.3	196.1	70.9	361.8	-11.0
Triflumuron	550.3	0.3	753.2	44.1	849.0	285.5	880.2	53.1	678.1	134.3	301.6	32.3	930.3	57.6
Trifluralin	192.1	25.1	169.3	18.7	203.2	513.4	139.6	-3.8	484.0	228.9	201.0	75.4	759.3	21.1
Uniconazole	371.9	-7.8	415.2	65.2	501.9	485.1	468.6	72.2	632.6	188.0	300.5	84.8	966.6	78.6
Vernolate	286.6	8.4	254.3	39.1	243.1	256.0	241.9	42.3	288.5	138.3	124.4	34.6	396.9	48.9
Vinclozolin	171.9	-20.8	225.2	23.0	113.3	228.1	223.0	25.4	285.0	116.8	141.0	45.8	434.8	19.3
Zoxamide	1587.2	693.5	2802.9	1996.2	2754.9	2631.6	2327.7	1976.8	2017.7	420.4	663.8	290.7	2310.1	1316.2
Average	526.1	50.3	577.4	188.3	647.3	663.9	696.2	218.9	662.3	214.5	285.7	99.0	988.6	191.5

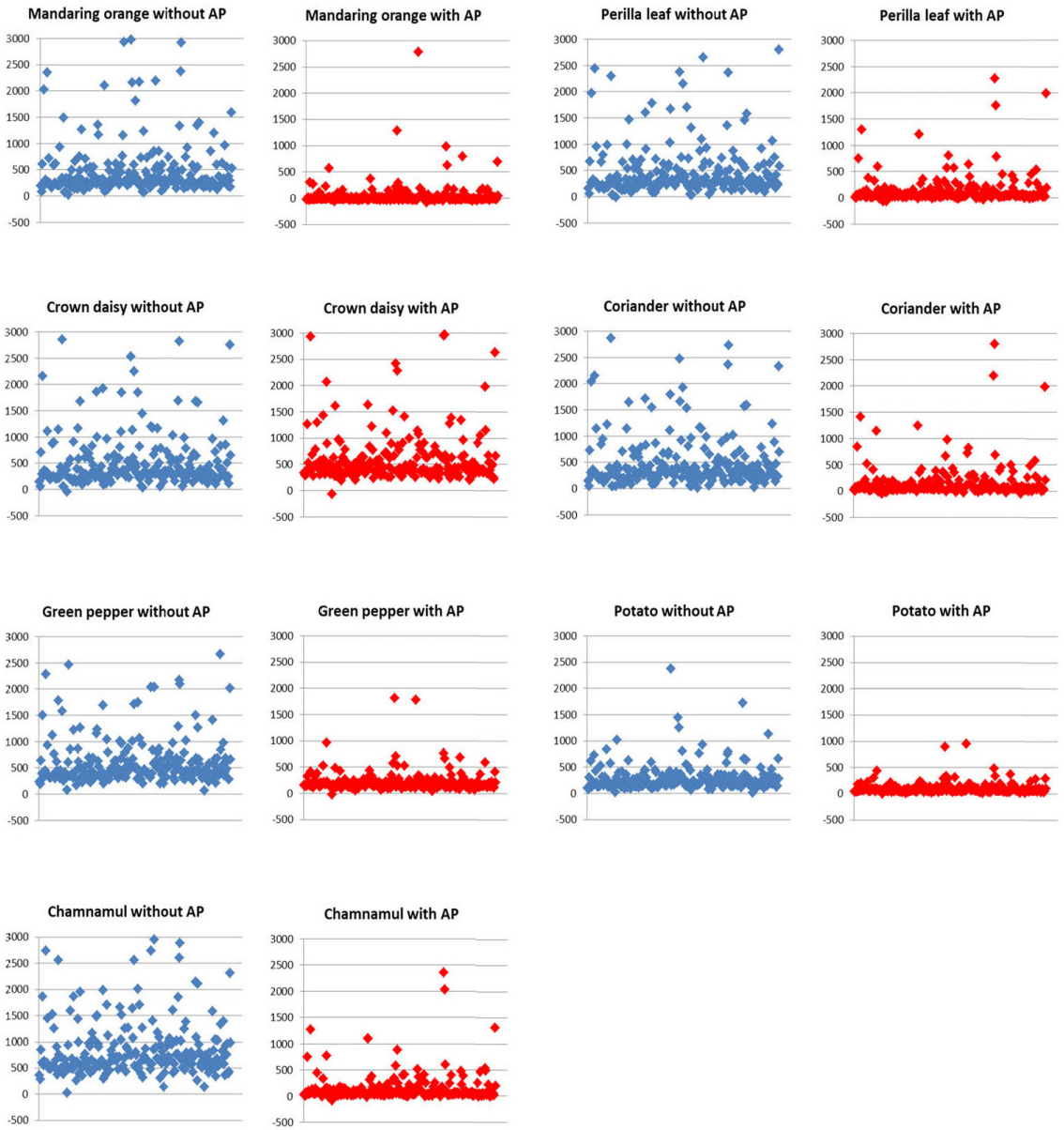

Fig. 1. Distribution plot of matrix effect(%) of 235 pesticides analyzed without or with the analyte protectant(AP) D-sorbitol in seven agricultural products. y-axis is matrix effect(%).

Chloroneb는 깻잎에서, mecabam은 고수에서, 그리고 iprovalicarb는 참나물에서 특히 큰 %RSD를 보였는데, 이는 해당 농산물 매트릭스가 농약과 유사한 머무름 시간에 같은 크기의 product ion을 생성하여 나타나는 것으로 추측되며, 분석조건에 대한 추가적인 검토가 필요할 것으로 보인다.

결 론

GC/MS/MS를 이용한 다종농약 다성분 분석시 고추, 감귤, 감자, 들깻잎, 쑥갓, 고수, 참나물 7종의 농산물에 대한 235종 농약의 매트릭스 효과를 확인하고, 시험용액에 분석체 보호제로 D-

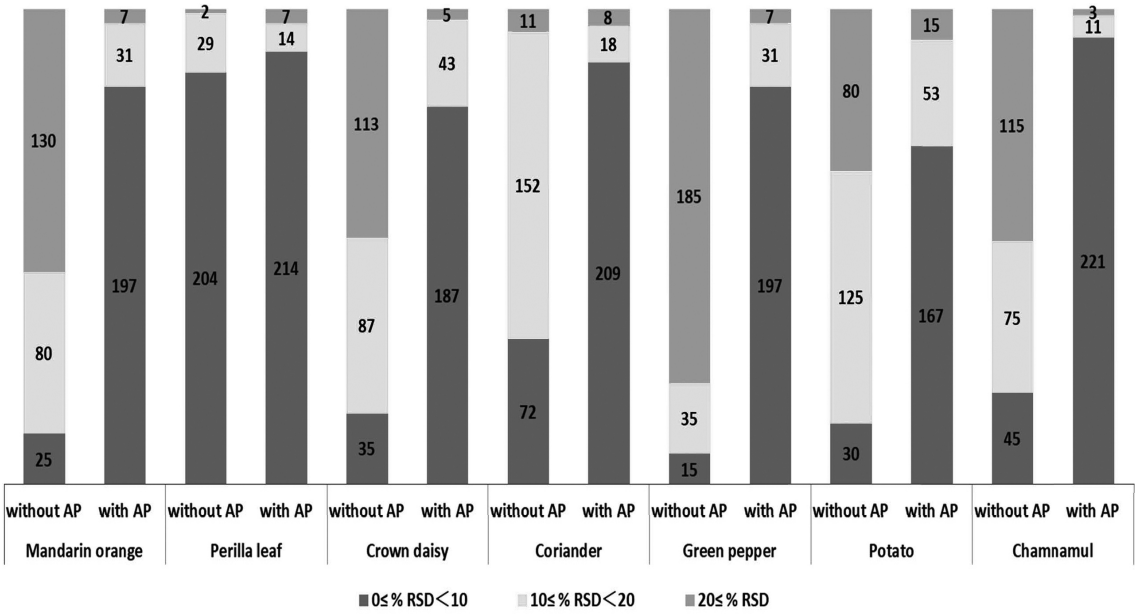

Fig. 2. The number of pesticides classfied into their %RSD(n = 3, weekly interval) of calibration curve slope without or with the analyte protectant(AP) D-sorbitol in seven agricultural products.

Table 4. Comparison of calibration curve linearity of 235 pesticides without or with the analyte protectant(AP) D-sorbitol

Linearity of calibration curve (25~250ng/mL)	No. of pesticides													
	Mandarin orange		Perilla leaf		Crown daisy		Coriander		Green pepper		Potato		Chamnamul	
	Without AP	With AP	Without AP	With AP	Without AP	With AP	Without AP	With AP	Without AP	With AP	Without AP	With AP	Without AP	With AP
0.99 ≤R^2	139	234	64	233	58	230	49	230	25	227	53	180	70	232
0.98≤R^2<0.99	79	0	86	1	95	1	93	2	54	7	81	48	105	1
0.97≤R^2<0.98	15	0	64	0	79	3	85	1	68	0	55	5	53	0
R2<0.97	2	1	21	1	3	1	8	2	88	1	46	2	7	2

sorbitol을 첨가하여 매트릭스 효과를 개선하여 분석값의 신뢰도를 향상시키고자 하였다. D-sorbitol을 첨가한 경우, 그렇지 않은 경우보다 matrix effect %가 대부분 감소하여, 감귤은 약 1/10로, 깻잎, 고수, 풋고추, 감자, 참나물은 약 1/3로 감소하였다. 또한, D-sorbitol 첨가에 따라 분석값의 반복 재현성 및 검량선의 직선성도 증가하였는데, 3회 반복 측정한 235종 농약 대부분 검량선의 %RSD는 10 미만이었고, R^2값는 0.99 이상이었다.

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