

図 1-1 各農薬のピーク面積に対するキャピラリー電圧の影響(ESI(+))
(コーン電圧 25 V, アパーチャー1 電圧 5 V)

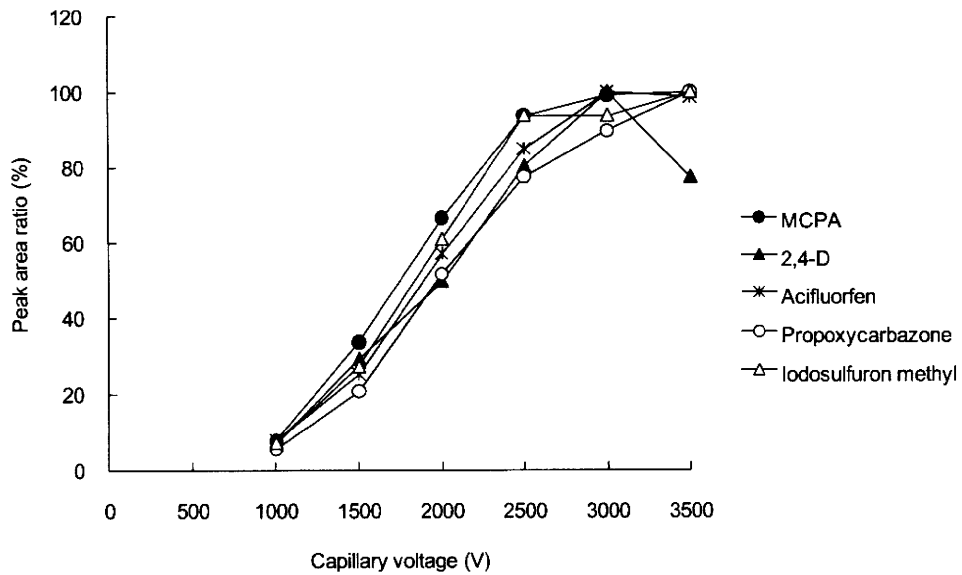


図 1-2 各農薬のピーク面積に対するキャピラリー電圧の影響(ESI(-))
(コーン電圧 25 V, アパーチャー1 電圧 5 V)

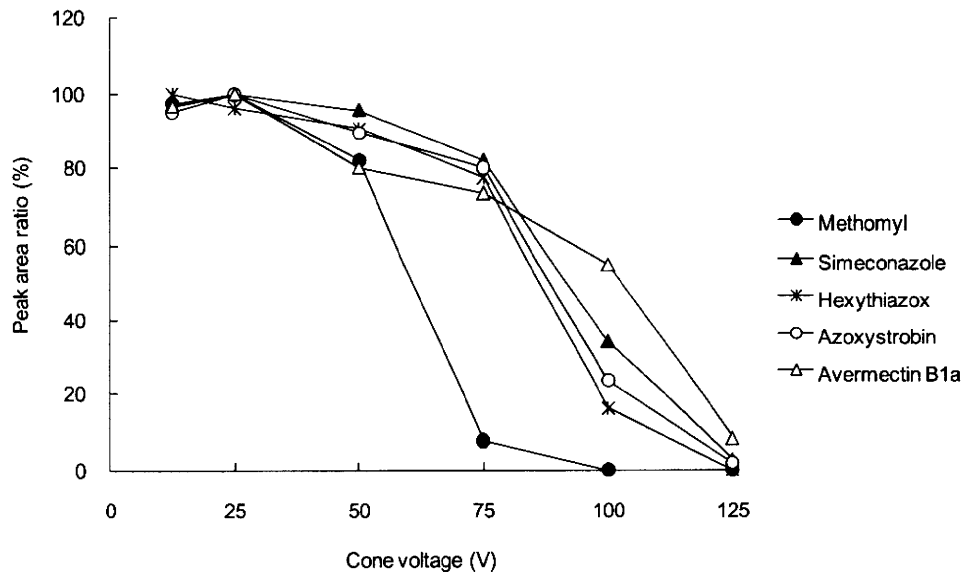


図 2-1 各農薬のピーク面積に対するコーン電圧の影響(ESI(+))
(キャピラリー電圧 3000 V, アパーチャー1 電圧 5 V)

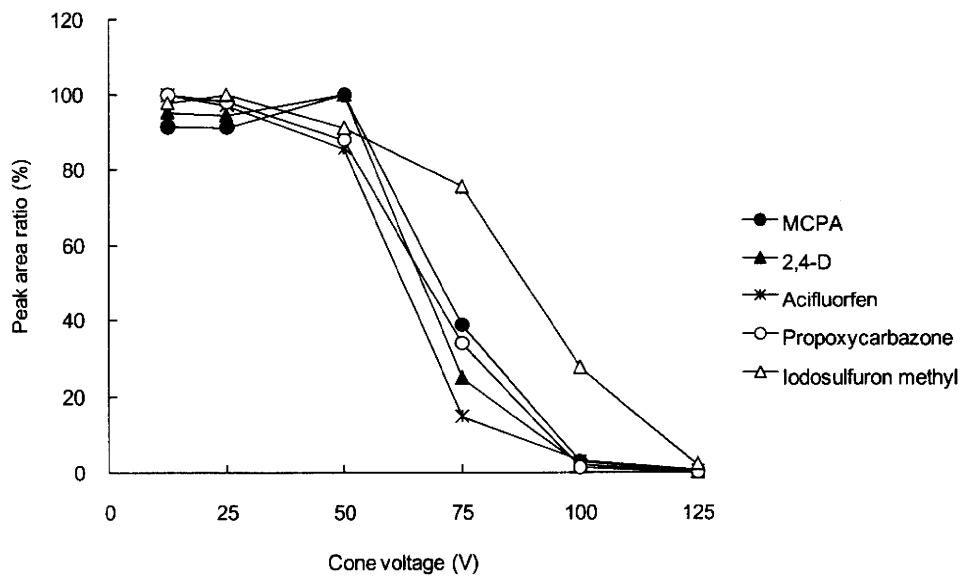


図 2-2 各農薬のピーク面積に対するコーン電圧の影響(ESI(-))
(キャピラリー電圧 3000 V, アパーチャー1 電圧 5 V)

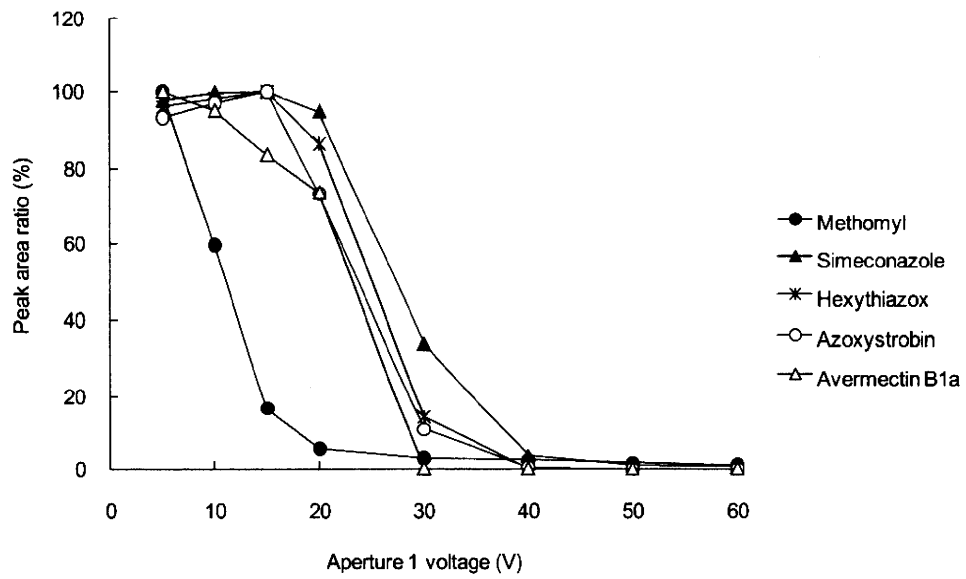


図 3-1 各農薬のピーク面積に対するアパーチャー1 電圧の影響(ESI(+))
(キャピラリー電圧 3000 V, コーン電圧 25 V)

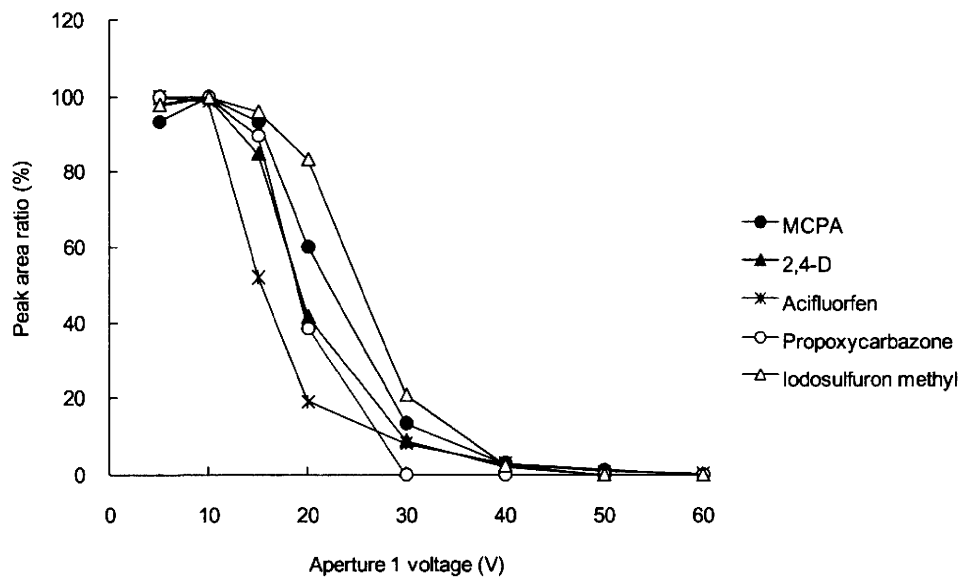


図 3-2 各農薬のピーク面積に対するアパーチャー1 電圧の影響(ESI(-))
(キャピラリー電圧 3000 V, コーン電圧 25 V)

表 1 検討に用いた農薬の定量イオン及び保持時間

Compound	Elemental composition ^{a)}	Retention time (min) ^{b)}	ESI (+)		ESI (-)		
			Type of molecular ion	<i>m/z</i> , calculated exact mass	Type of molecular ion	<i>m/z</i> , calculated exact mass	
Azamethiphos	C ₉ H ₁₀ ClN ₂ O ₅ PS	6.50	[M+H] ⁺	324.9809			
Acibenzolar-S-methyl	C ₈ H ₆ N ₂ OS ₂	8.34	[M+H] ⁺	210.9994			
Azoxystrobin	C ₂₂ H ₁₇ N ₃ O ₅	8.16	[M+H] ⁺	404.1241			
Anilofos	C ₁₃ H ₁₉ ClNO ₃ PS ₂	9.39	[M+H] ⁺	368.0305			
Aldicarb	C ₇ H ₁₄ N ₂ O ₂ S	5.94	[M+NH ₄] ⁺	208.1114			
Imidacloprid	C ₉ H ₁₀ ClN ₅ O ₂	4.57	[M+H] ⁺	256.0596			
Indanofan	C ₂₀ H ₁₇ ClO ₃	8.96	[M+H] ⁺	341.0939			
Epoxiconazole	C ₁₇ H ₁₃ ClFN ₃ O	8.96	[M+H] ⁺	330.0804			
Oxaziclofomefone	C ₂₀ H ₁₉ Cl ₂ NO ₂	10.09	[M+H] ⁺	376.0866			
Carpropamid	C ₁₅ H ₁₈ Cl ₃ NO	9.41	[M+H] ⁺	334.0527	[M-H] ⁻	332.0381	
Cumyluron	C ₁₇ H ₁₉ ClN ₂ O	8.69	[M+H] ⁺	303.1259			
Clothianidin	C ₆ H ₈ ClN ₅ O ₂ S	4.65	[M+H] ⁺	250.0160	[M-H] ⁻	248.0014	
Chloridazon	C ₁₀ H ₈ ClN ₃ O	5.09	[M+H] ⁺	222.0429	[M-H] ⁻	220.0283	
Chloroxuron	C ₁₅ H ₁₅ ClN ₂ O ₂	8.80	[M+H] ⁺	291.0895			
Simeconazole	C ₁₄ H ₂₀ FN ₃ OSi	8.94	[M+H] ⁺	294.1432			
Dimethomorph	C ₂₁ H ₂₂ ClNO ₄	8.31	8.58	[M+H] ⁺	388.1310		
Spinosyn A	C ₄₁ H ₆₅ NO ₁₀	11.20	[M+H] ⁺	732.4681			
Spinosyn D	C ₄₂ H ₆₇ NO ₁₀	11.41	[M+H] ⁺	746.4838			
Thiodicarb	C ₁₀ H ₁₈ N ₄ O ₄ S ₃	7.18	[M+H] ⁺	355.0563			
Tebuthiuron	C ₉ H ₁₆ N ₄ OS	6.90	[M+H] ⁺	229.1118			
Tebu fenozide	C ₂₂ H ₂₈ N ₂ O ₂	9.15	[M+H] ⁺	353.2224	[M-H] ⁻	351.2078	
Novaluron	C ₁₇ H ₉ ClF ₈ N ₂ O ₄	9.91			[M-H] ⁻	491.0050	
Pyrifthalid	C ₁₅ H ₁₄ N ₂ O ₄ S	8.05	[M+H] ⁺	319.0747			
Pirimicarb	C ₁₁ H ₁₈ N ₄ O ₂	7.39	[M+H] ⁺	239.1503			
Fenoxaprop-ethyl	C ₁₈ H ₁₆ ClNO ₅	10.07	[M+H] ⁺	362.0790			
Fenamidone	C ₁₇ H ₁₇ N ₃ OS	8.35	[M+H] ⁺	312.1165			
Benzofenap	C ₂₂ H ₂₀ Cl ₂ N ₂ O ₃	10.02	[M+H] ⁺	431.0924			
Methomyl	C ₅ H ₁₀ N ₂ O ₂ S	3.65	[M+H] ⁺	163.0536			
Mepanipyrim	C ₁₄ H ₁₃ N ₃	8.82	[M+H] ⁺	224.1182			
Lactofen	C ₁₉ H ₁₅ ClF ₃ NO ₇	10.17	[M+NH ₄] ⁺	479.0827			
Azinphos-methyl	C ₁₀ H ₁₂ N ₃ O ₃ PS ₂	7.95	[M+NH ₄] ⁺	335.0396			
Aldoxycarb	C ₇ H ₁₄ N ₂ O ₄ S	3.25	[M+NH ₄] ⁺	240.1013			
Iprovalicarb	C ₁₈ H ₂₈ N ₂ O ₃	8.78	[M+H] ⁺	321.2173			
Imazalil	C ₁₄ H ₁₄ Cl ₂ N ₂ O	9.42	[M+H] ⁺	297.0556			
Oxycarboxine	C ₁₂ H ₁₃ NO ₄ S	5.43	[M+NH ₄] ⁺	285.0904			
Carbaryl	C ₁₂ H ₁₁ NO ₂	7.05	[M+NH ₄] ⁺	219.1128			
Cloquintocet-mexyl	C ₁₈ H ₂₂ ClNO ₃	10.29	[M+H] ⁺	336.1361			
Clofentezine	C ₁₄ H ₈ Cl ₂ N ₄	9.64	[M+H] ⁺	303.0199			
Cyazofamid	C ₁₃ H ₁₃ ClN ₄ O ₂ S	9.02	[M+H] ⁺	325.0521			
Diuron	C ₉ H ₁₀ Cl ₂ N ₂ O	7.78	[M+H] ⁺	233.0243	[M-H] ⁻	231.0097	

表 1 検討に用いた農薬の定量イオン及び保持時間(続き)

Compound	Elemental composition ^{a)}	Retention time (min) ^{b)}	ESI (+)		ESI (-)	
			Type of molecular ion	<i>m/z</i> , calculated exact mass	Type of molecular ion	<i>m/z</i> , calculated exact mass
Cycloprothrin	C ₂₆ H ₂₁ Cl ₂ NO ₄	10.66	[M+NH ₄] ⁺	499.1186		
Cyprodinil	C ₁₄ H ₁₅ N ₃	9.42	[M+H] ⁺	226.1339		
Thiacloprid	C ₁₀ H ₉ ClN ₄ S	5.51	[M+H] ⁺	253.0309		
Thiamethoxam	C ₈ H ₁₀ ClN ₅ O ₃ S	3.87	[M+NH ₄] ⁺	309.0531		
Teflubenzuron	C ₁₄ H ₆ Cl ₂ F ₄ N ₂ O ₂	10.35			[M-H] ⁻	378.9670
Triflumuron	C ₁₅ H ₁₀ ClF ₃ N ₂ O ₃	9.59	[M+H] ⁺	359.0405	[M-H] ⁻	357.0259
Pyraclostrobin	C ₁₉ H ₁₈ ClN ₃ O ₄	9.53	[M+H] ⁺	388.1059		
Pyrazolynate	C ₁₉ H ₁₆ Cl ₂ N ₂ O ₄ S	9.69	[M+H] ⁺	439.0281		
Fenobucarb	C ₁₂ H ₁₇ NO ₂	8.20	[M+NH ₄] ⁺	225.1598		
(E)-Fenpyroximate	C ₂₄ H ₂₇ N ₃ O ₄	10.69	[M+H] ⁺	422.2074		
(Z)-Fenpyroximate	C ₂₄ H ₂₇ N ₃ O ₄	10.30	[M+H] ⁺	422.2074		
Flufenacet	C ₁₄ H ₁₃ F ₄ N ₃ O ₂ S	8.90	[M+H] ⁺	364.0737		
Flufenoxuron	C ₂₁ H ₁₁ ClF ₆ N ₂ O ₃	10.58	[M+H] ⁺	489.0435	[M-H] ⁻	487.0290
Fluridone	C ₁₉ H ₁₄ F ₃ NO	8.01	[M+H] ⁺	330.1100		
Hexythiazox	C ₁₇ H ₂₁ ClN ₂ O ₂ S	10.47	[M+H] ⁺	353.1085		
Boscalid	C ₁₈ H ₁₂ Cl ₂ N ₂ O	8.42	[M+H] ⁺	343.0399	[M-H] ⁻	341.0254
Methabenzthiazuron	C ₁₀ H ₁₁ N ₃ OS	7.53	[M+H] ⁺	222.0696		
Methoxyfenozide	C ₂₂ H ₂₈ N ₂ O ₃	8.57	[M+H] ⁺	369.2173	[M-H] ⁻	367.2027
Monolinuron	C ₉ H ₁₁ ClN ₂ O ₂	7.17	[M+H] ⁺	215.0582		
Azafenidin	C ₁₅ H ₁₃ Cl ₂ N ₃ O ₂	7.72	[M+H] ⁺	338.0458		
Aramite	C ₁₅ H ₂₃ ClO ₄ S	10.26	[M+NH ₄] ⁺	352.1344		
Isoxaflutole	C ₁₅ H ₁₂ F ₃ NO ₄ S	7.67			[M-H] ⁻	358.0366
Indoxacarb	C ₂₂ H ₁₇ ClF ₃ N ₃ O ₇	9.81	[M+H] ⁺	528.0780		
Oxamyl	C ₇ H ₁₃ N ₃ O ₃ S	3.37	[M+NH ₄] ⁺	237.1016		
Carbofuran	C ₁₂ H ₁₅ NO ₃	6.74	[M+H] ⁺	222.1125		
Quizalofop-ethyl	C ₁₉ H ₁₇ ClN ₂ O ₄	10.09	[M+H] ⁺	373.0950		
Chromafenozide	C ₂₄ H ₃₀ N ₂ O ₃	8.80	[M+H] ⁺	395.2329		
Clomeprop	C ₁₆ H ₁₅ Cl ₂ NO ₂	10.21	[M+H] ⁺	324.0553	[M-H] ⁻	322.0407
Cyflufenamid	C ₂₀ H ₁₇ F ₅ N ₂ O ₂	9.58	[M+H] ⁺	413.1283		
Diflubenzuron	C ₁₄ H ₉ ClF ₂ N ₂ O ₂	9.15	[M+H] ⁺	311.0393	[M-H] ⁻	309.0248
Dimethirimol	C ₁₁ H ₁₉ N ₃ O	7.44	[M+H] ⁺	210.1601		
Daimuron	C ₁₇ H ₂₀ N ₂ O	8.60	[M+H] ⁺	269.1648		
Thiabendazole	C ₁₀ H ₇ N ₃ S	6.15	[M+H] ⁺	202.0433		
Tetrachlorvinphos	C ₁₀ H ₉ Cl ₄ O ₄ P	9.24	[M+NH ₄] ⁺	383.9301		
Triticonazole	C ₁₇ H ₂₀ ClN ₃ O	8.92	[M+H] ⁺	318.1368		
Naproanilide	C ₁₉ H ₁₇ NO ₂	9.12	[M+H] ⁺	292.1332	[M-H] ⁻	290.1187
Fenoxycarb	C ₁₇ H ₁₉ NO ₄	9.20	[M+H] ⁺	302.1387		
Ferimzone	C ₁₅ H ₁₈ N ₄	8.34	[M+H] ⁺	255.1604		
Butafenacil	C ₂₀ H ₁₈ ClF ₃ N ₂ O ₆	8.75	[M+NH ₄] ⁺	492.1144		
Furathiocarb	C ₁₈ H ₂₆ N ₂ O ₅ S	10.14	[M+H] ⁺	383.1635		

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Compound	Elemental composition ^{a)}	Retention time (min) ^{b)}	ESI (+)		ESI (-)		
			Type of molecular ion	<i>m/z</i> , calculated exact mass	Type of molecular ion	<i>m/z</i> , calculated exact mass	
Furamctpyr	C ₁₇ H ₂₀ ClN ₃ O ₂	7.50	[M+H] ⁺	334.1317			
Propaquizafop	C ₂₂ H ₂₂ ClN ₃ O ₅	10.20	[M+H] ⁺	444.1321			
Pencycuron	C ₁₉ H ₂₁ ClN ₂ O	9.74	[M+H] ⁺	329.1415			
Bendiocarb	C ₁₁ H ₁₃ NO ₄	6.73	[M+NH ₄] ⁺	241.1183			
Pentoxazone	C ₁₇ H ₁₇ ClFNO ₄	10.08	[M+NH ₄] ⁺	371.1168			
Methiocarb	C ₁₁ H ₁₅ NO ₂ S	8.37	[M+NH ₄] ⁺	243.1162			
Linuron	C ₉ H ₁₀ Cl ₂ N ₂ O ₂	8.28	[M+H] ⁺	249.0192			
Avermectin Bla	C ₄₈ H ₇₂ O ₁₄	11.05	[M+NH ₄] ⁺	890.5260			
Oryzalin	C ₁₂ H ₁₈ N ₄ O ₆ S	9.02			[M-H] ⁻	345.0874	
Cycloate	C ₁₁ H ₂₁ NOS	9.87	[M+H] ⁺	216.1417			
Silafluofen	C ₂₅ H ₂₉ FO ₂ Si	11.91	[M+NH ₄] ⁺	426.2259			
Di-allate	C ₁₀ H ₁₇ Cl ₂ NOS	9.97	[M+H] ⁺	270.0481			
Tralkoxydim	C ₂₀ H ₂₇ NO ₃	7.57	8.18	[M+H] ⁺	330.2064	[M-H] ⁻	328.1918
Tridemorph	C ₁₉ H ₃₉ NO	11.80	12.09	[M+H] ⁺	298.3104		
Phenmedipham	C ₁₆ H ₁₆ N ₂ O ₄	7.96		[M+NH ₄] ⁺	318.1448		
Hexaflumuron	C ₁₆ H ₈ Cl ₂ F ₆ N ₂ O ₃	9.86				[M-H] ⁻	458.9743
Lu fenuron	C ₁₇ H ₈ Cl ₂ F ₈ N ₂ O ₃	10.26				[M-H] ⁻	508.9711
Azimsulfuron	C ₁₃ H ₁₆ N ₁₀ O ₅ S	4.58		[M+H] ⁺	425.1099	[M-H] ⁻	423.0953
Iodosulfuron-methyl	C ₁₄ H ₁₄ IN ₅ O ₆ S	6.03		[M+H] ⁺	507.9782	[M-H] ⁻	505.9637
Imazaquin	C ₁₇ H ₁₇ N ₃ O ₃	4.78		[M+H] ⁺	312.1343	[M-H] ⁻	310.1197
Ethametsulfuron-methyl	C ₁₅ H ₁₈ N ₆ O ₆ S	5.39		[M+H] ⁺	411.1081	[M-H] ⁻	409.0936
Ethoxysulfuron	C ₁₅ H ₁₈ N ₄ O ₇ S	6.56		[M+H] ⁺	399.0969	[M-H] ⁻	397.0823
Clodinafop	C ₁₄ H ₁₁ ClFNO ₄	7.17		[M+H] ⁺	312.0433	[M-H] ⁻	310.0288
Cloransulam-methyl	C ₁₅ H ₁₃ ClFN ₅ O ₅ S	6.50		[M+H] ⁺	430.0383	[M-H] ⁻	428.0237
Chlorimuron-ethyl	C ₁₅ H ₁₅ ClN ₄ O ₆ S	6.33		[M+H] ⁺	415.0474	[M-H] ⁻	413.0328
Chlorsulfuron	C ₁₂ H ₁₂ ClN ₅ O ₄ S	4.86		[M+H] ⁺	358.0371	[M-H] ⁻	356.0226
Diclosulam	C ₁₃ H ₁₀ Cl ₂ FN ₅ O ₃ S	6.67		[M+H] ⁺	405.9938	[M-H] ⁻	403.9793
Sulfentrazone	C ₁₁ H ₁₀ Cl ₂ F ₂ N ₄ O ₃ S	6.57		[M+NH ₄] ⁺	404.0157	[M-H] ⁻	384.9746
Sulfosulfuron	C ₁₆ H ₁₈ N ₆ O ₇ S ₂	5.11		[M+H] ⁺	471.0751		
Thifensulfuron-methyl	C ₁₂ H ₁₃ N ₅ O ₆ S ₂	4.40		[M+H] ⁺	388.0380	[M-H] ⁻	386.0234
Triasulfuron	C ₁₄ H ₁₆ ClN ₅ O ₅ S	5.23		[M+H] ⁺	402.0633	[M-H] ⁻	400.0488
Triflurosulfuron-methyl	C ₁₇ H ₁₉ F ₃ N ₆ O ₆ S	7.01		[M+H] ⁺	493.1112	[M-H] ⁻	491.0966
Trifloxysulfuron	C ₁₄ H ₁₄ F ₃ N ₅ O ₆ S	5.33		[M+H] ⁺	438.0690	[M-H] ⁻	436.0544
Haloxyfop	C ₁₅ H ₁₁ ClF ₃ NO ₄	8.24		[M+H] ⁺	362.0401	[M-H] ⁻	360.0256
Flumetsulam	C ₁₂ H ₉ F ₂ N ₅ O ₂ S	3.75		[M+H] ⁺	326.0518	[M-H] ⁻	324.0372
Florasulam	C ₁₂ H ₈ F ₃ N ₅ O ₃ S	4.79		[M+NH ₄] ⁺	377.0638	[M-H] ⁻	358.0227
Penoxsulam	C ₁₆ H ₁₄ F ₃ N ₅ O ₅ S	6.29		[M+H] ⁺	484.0709	[M-H] ⁻	482.0563
Forchlorfenuron	C ₁₂ H ₁₀ ClN ₃ O	7.74		[M+H] ⁺	248.0585	[M-H] ⁻	246.0440
2,4-D	C ₈ H ₆ Cl ₂ O ₃	6.18				[M-H] ⁻	218.9621
MCPA	C ₉ H ₉ ClO ₃	6.21				[M-H] ⁻	199.0167

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Compound	Elemental composition ^{a)}	Retention time (min) ^{b)}	ESI (+)		ESI (-)	
			Type of molecular ion	<i>m/z</i> , calculated exact mass	Type of molecular ion	<i>m/z</i> , calculated exact mass
MCPB	C ₁₁ H ₁₃ ClO ₃	7.53			[M-H] ⁻	227.0480
Ioxynil	C ₇ H ₃ I ₂ NO	5.99			[M-H] ⁻	369.8231
Acifluorfen	C ₁₄ H ₇ ClF ₃ NO ₅	7.97			[M-H] ⁻	359.9892
Cloprop	C ₉ H ₉ ClO ₃	5.49			[M-H] ⁻	199.0167
4-Chlorophenoxyacetic acid	C ₈ H ₇ ClO ₃	4.86			[M-H] ⁻	185.0011
Cyclanilide	C ₁₁ H ₉ Cl ₂ NO ₃	7.29			[M-H] ⁻	271.9887
Dichlorprop	C ₉ H ₈ Cl ₂ O ₃	6.90			[M-H] ⁻	232.9778
Gibberellic acid	C ₁₉ H ₂₂ O ₆	4.26			[M-H] ⁻	345.1344
Triclopyr	C ₇ H ₄ Cl ₃ NO ₃	6.61			[M-H] ⁻	253.9184
1-Naphthylacetic acid	C ₁₂ H ₁₀ O ₂	5.39	[M+NH ₄] ⁺	204.1019		
Fluroxypyr	C ₇ H ₅ Cl ₂ FN ₂ O ₃	4.15			[M-H] ⁻	252.9588
Bromoxynil	C ₇ H ₃ Br ₂ NO	4.89			[M-H] ⁻	275.8488
Fomesafen	C ₁₅ H ₁₀ ClF ₃ N ₂ O ₆ S	7.95	[M+NH ₄] ⁺	456.0238	[M-H] ⁻	436.9827
Mecoprop	C ₁₀ H ₁₁ ClO ₃	6.83			[M-H] ⁻	213.0324
Imazosulfuron	C ₁₄ H ₁₃ ClN ₆ O ₅ S	5.10	[M+H] ⁺	413.0429	[M-H] ⁻	411.0284
Cyclosulfamuron	C ₁₇ H ₁₉ N ₅ O ₆ S	7.22	[M+H] ⁺	422.1129	[M-H] ⁻	420.0983
Diclomezine	C ₁₁ H ₈ Cl ₂ N ₂ O	9.16	[M+H] ⁺	255.0086		
Cinosulfuron	C ₁₅ H ₁₉ N ₅ O ₇ S	4.74	[M+H] ⁺	414.1078	[M-H] ⁻	412.0932
Thidiazuron	C ₉ H ₈ N ₄ OS	6.73	[M+H] ⁺	221.0492	[M-H] ⁻	219.0346
Tribenuron-methyl	C ₁₅ H ₁₇ N ₅ O ₆ S	5.01	[M+H] ⁺	396.0972	[M-H] ⁻	394.0827
Naptalam	C ₁₈ H ₁₃ NO ₃	5.93	[M+H] ⁺	292.0968	[M-H] ⁻	290.0823
Halosulfuron-methyl	C ₁₃ H ₁₅ ClN ₆ O ₇ S	6.01	[M+H] ⁺	435.0484	[M-H] ⁻	433.0339
Pyrazosulfuron-ethyl	C ₁₄ H ₁₈ N ₆ O ₇ S	5.72	[M+H] ⁺	415.1030	[M-H] ⁻	413.0885
Fenhexamid	C ₁₄ H ₁₇ Cl ₂ NO ₂	8.79	[M+H] ⁺	302.0709	[M-H] ⁻	300.0564
Flazasulfuron	C ₁₃ H ₁₂ F ₃ N ₅ O ₅ S	4.98	[M+H] ⁺	408.0584	[M-H] ⁻	406.0438
Primisulfuron-methyl	C ₁₅ H ₁₂ F ₄ N ₄ O ₇ S	7.22	[M+H] ⁺	469.0436	[M-H] ⁻	467.0290
Fluazifop	C ₁₅ H ₁₂ F ₃ NO ₄	7.16	[M+H] ⁺	328.0791	[M-H] ⁻	326.0646
Prosulfuron	C ₁₅ H ₁₆ F ₃ N ₅ O ₄ S	6.83	[M+H] ⁺	420.0948	[M-H] ⁻	418.0802
Propoxycarbazone	C ₁₅ H ₁₈ N ₄ O ₇ S	5.31	[M+NH ₄] ⁺	416.1234	[M-H] ⁻	397.0823
Bensulfuron-methyl	C ₁₆ H ₁₈ N ₄ O ₇ S	6.73	[M+H] ⁺	411.0969	[M-H] ⁻	409.0823
Foramsulfuron	C ₁₇ H ₂₀ N ₆ O ₇ S	4.66	[M+H] ⁺	453.1187	[M-H] ⁻	451.1041
Mesosulfuron-methyl	C ₁₇ H ₂₁ N ₅ O ₉ S ₂	5.05	[M+H] ⁺	504.0853	[M-H] ⁻	502.0708
Metosulam	C ₁₄ H ₁₃ Cl ₂ N ₅ O ₄ S	6.24	[M+H] ⁺	418.0138	[M-H] ⁻	415.9993
Metsulfuron-methyl	C ₁₄ H ₁₅ N ₅ O ₆ S	4.41	[M+H] ⁺	382.0816	[M-H] ⁻	380.0670

a) Elemental composition correspond to the neutral molecule.

b) Gradient method a

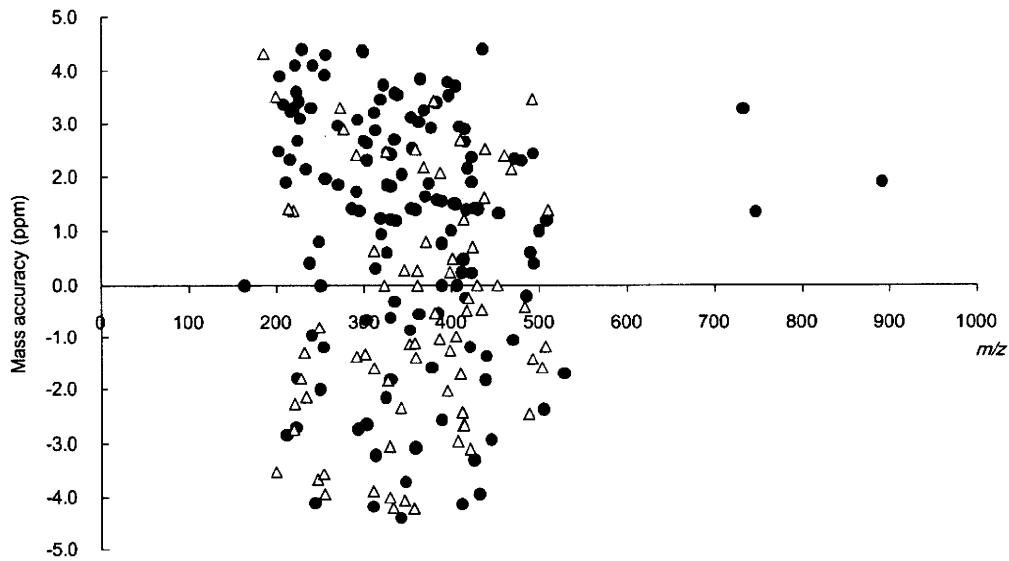


図4 定量イオンの質量精度

(混合標準溶液濃度: 0.1 $\mu\text{g/mL}$, ●: ESI(+), △: ESI(-))

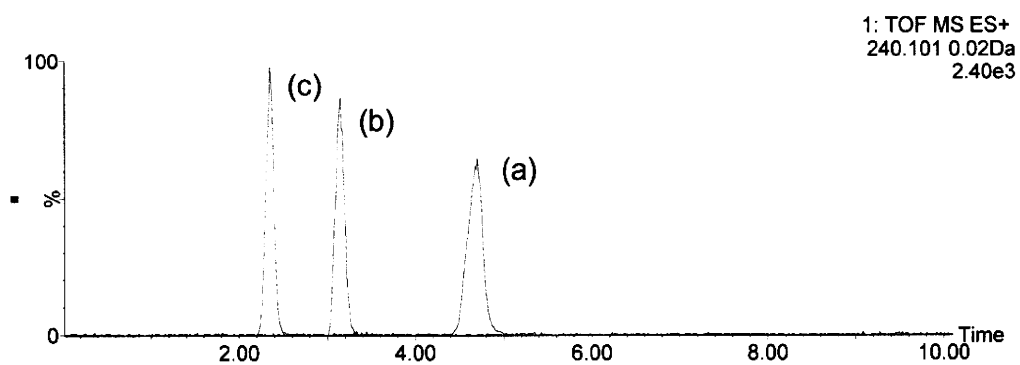


図5 アルドキシカルブの抽出イオンクロマトグラム (0.1 $\mu\text{g/mL}$, m/z 240.1012, ± 20 mDa)

(a) 流速 0.2 mL/min, グラジエント条件: 0 min (A:B=95:5) \rightarrow 15 min (A:B=5:95)

(b) 流速 0.3 mL/min, グラジエント条件: 0 min (A:B=95:5) \rightarrow 10 min (A:B=5:95)

(c) 流速 0.4 mL/min, グラジエント条件: 0 min (A:B=95:5) \rightarrow 7.5 min (A:B=5:95)

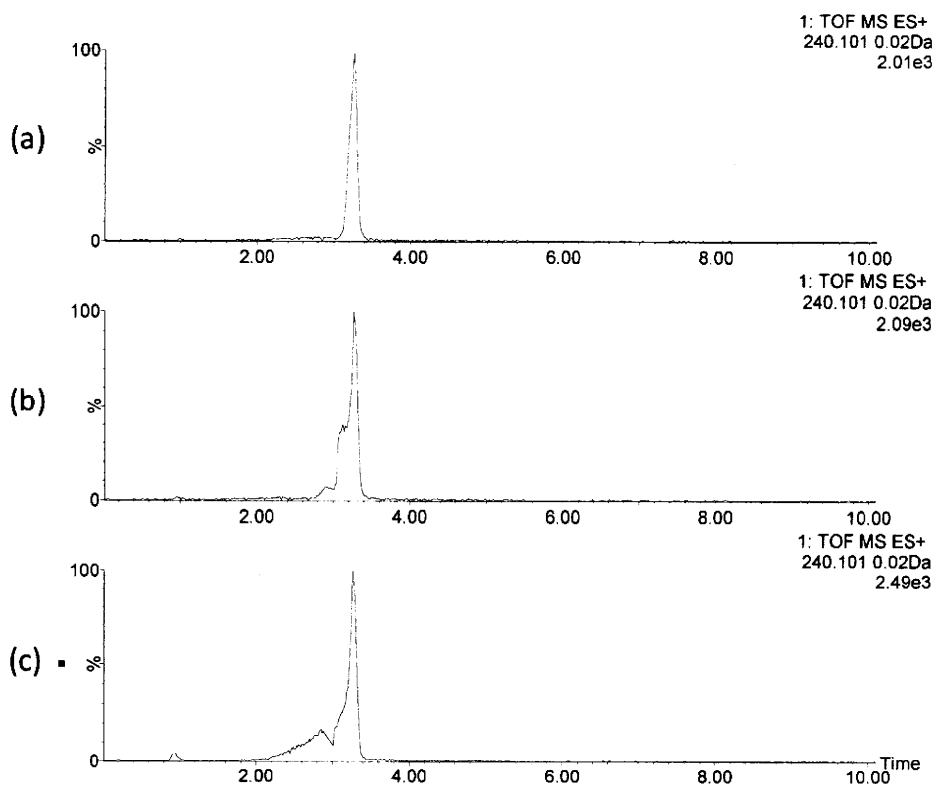


図 6 注入量によるアルドキシカルブのピーク形状の影響(0.1 $\mu\text{g/mL}$, m/z 240.1012, ± 20 mDa)

(a) 注入量 3 μL 、(b) 注入量 5 μL 、(c) 注入量 7 μL

表 2-1 食品マトリックスによる測定への影響及び定量限界(ESI(+))

Compound	Matrix effect ^{a)}				LOQ (mg/kg) ^{b)}	
	Soybean		Spinach		Soybean	Spinach
	0.1 (µg/mL)	0.01 (µg/mL)	0.1 (µg/mL)	0.01 (µg/mL)		
Azamethiphos	0.82	0.53	0.83	0.94	0.001	0.001
Acibenzolar-S-methyl	1.11	— ^b	1.09	— ^b	0.07	0.02
Azoxystrobin	1.03	0.98	0.97	1.03	0.001	0.001
Anilofos	1.07	1.01	0.95	1.11	0.001	0.001
Aldicarb	0.99	1.00	0.90	0.96	0.006	0.002
Imidacloprid	0.99	0.80	1.01	0.83	0.003	0.002
Indanofan	0.97	— ^b	1.03	— ^b	0.06	0.04
Epoxiconazole	1.03	0.97	0.96	0.97	0.001	0.001
Oxaziclomefone	1.02	1.00	1.00	0.96	0.001	0.001
Carpropamid	1.04	0.95	1.04	0.64	0.004	0.002
Cumyluron	1.03	0.92	0.97	1.00	0.001	0.001
Clothianidin	1.00	0.84	1.04	0.96	0.002	0.004
Chloridazon	0.98	1.01	0.98	0.97	0.001	0.001
Chloroxuron	1.02	0.99	1.00	0.91	0.001	0.001
Simeconazole	1.01	1.00	0.94	1.06	0.001	0.001
Dimethomorph	1.03	0.94	0.97	1.04	0.002	0.005
Spinosyn A	0.86	0.87	0.84	0.83	0.002	0.003
Spinosyn D	0.99	0.80	0.88	0.99	0.002	0.003
Thiodicarb	1.07	0.93	0.92	0.99	0.002	0.005
Tebuthiuron	1.01	0.99	0.87	0.95	0.001	0.002
Tebufenozide	1.03	1.07	1.00	1.04	0.001	0.001
Pyrifthalid	1.04	1.00	0.95	1.02	0.001	0.001
Pirimicarb	1.05	0.91	0.90	0.98	0.003	0.001
Fenoxaprop-ethyl	0.98	0.97	1.00	0.99	0.003	0.002
Fenamidone	1.01	0.95	0.96	0.98	0.001	0.001
Benzofenap	0.99	0.87	0.97	1.06	0.002	0.002
Methomyl	1.00	1.05	1.00	0.98	0.005	0.005
Mepanipyrim	1.06	1.01	0.95	0.99	0.001	0.001
Lactofen	0.98	0.92	1.03	1.04	0.002	0.001
Azinphos-methyl	1.02	1.07	0.93	1.01	0.001	0.001
Aldoxycarb	1.02	1.08	1.00	0.99	0.003	0.002
Iprovalicarb	1.04	1.00	1.00	1.05	0.001	0.002
Imazalil	1.07	1.01	0.99	0.99	0.001	0.001
Oxycarboxine	1.01	0.91	0.87	0.97	0.001	0.001
Carbaryl	1.01	1.04	0.92	0.95	0.003	0.002
Cloquintocet-mexyl	1.08	1.05	0.98	0.96	0.001	0.001
Clofentezine	1.01	0.93	1.05	0.65	0.007	0.003
Cyazofamid	0.94	0.92	1.00	0.97	0.006	0.001
Diuron	0.99	0.97	0.90	1.00	0.004	0.001
Cycloprothrin	0.89	0.83	1.03	0.84	0.004	0.003

表 2-1 食品マトリックスによる測定への影響及び定量限界(ESI(+)) (続き)

Compound	Matrix effect ^{a)}				LOQ (mg/kg) ^{b)}	
	Soybean		Spinach		Soybean	Spinach
	0.1 (µg/mL)	0.01 (µg/mL)	0.1 (µg/mL)	0.01 (µg/mL)		
Cyprodinil	1.08	1.05	0.95	0.97	0.001	0.002
Thiacloprid	1.00	0.99	0.92	0.96	0.003	0.002
Thiamethoxam	1.00	1.02	1.00	0.88	0.002	0.002
Triflumuron	1.02	0.97	1.10	0.93	0.003	0.003
Pyraclostrobin	1.08	1.07	0.98	0.99	0.001	0.001
Pyrazolynate	0.92	0.70	0.90	0.85	0.005	0.001
Fenobucarb	0.96	1.10	0.98	0.99	0.002	0.003
(E)-Fenpyroximate	0.96	0.97	0.95	1.06	0.001	0.001
(Z)-Fenpyroximate	1.03	0.99	0.95	0.97	0.001	0.001
Flufenacet	1.02	1.01	0.98	0.95	0.001	0.001
Flufenoxuron	1.01	1.04	— ^c	— ^c	0.001	— ^c
Fluridone	1.02	1.03	0.91	0.99	0.001	0.001
Hexythiazox	0.90	0.88	0.84	0.90	0.003	0.002
Boscalid	1.02	0.97	1.02	0.95	0.003	0.003
Methabenzthiazuron	1.05	1.01	0.90	1.07	0.004	0.004
Methoxyfenozide	1.02	0.92	0.98	1.00	0.001	0.001
Monolinuron	0.89	1.03	0.91	0.95	0.005	0.003
Azafenidin	1.00	0.95	0.92	1.02	0.002	0.001
Aramite	0.93	0.98	1.04	0.98	0.002	0.002
Indoxacarb	0.95	0.83	1.16	1.02	0.005	0.002
Oxamyl	1.03	1.06	1.04	0.95	0.009	0.008
Carbofuran	1.01	1.02	0.90	0.99	0.008	0.01
Quizalofop-ethyl	0.96	0.97	0.99	1.01	0.003	0.003
Chromafenozide	1.04	0.96	1.00	1.01	0.001	0.002
Clomeprop	0.98	0.97	0.83	0.98	0.003	0.002
Cyflufenamid	1.02	0.89	1.02	1.01	0.001	0.001
Diflubenzuron	1.05	1.14	0.98	0.80	0.005	0.003
Dimethirimol	1.06	1.01	0.90	0.99	0.003	0.001
Daimuron	0.99	0.96	0.97	1.02	0.001	0.001
Thiabendazole	1.00	1.00	0.88	1.02	0.001	0.001
Tetrachlorvinphos	1.04	1.03	0.93	1.01	0.002	0.001
Triticonazole	1.00	0.98	0.95	1.04	0.001	0.002
Naproanilide	1.01	0.89	0.99	0.95	0.002	0.002
Fenoxycarb	1.03	0.99	0.99	1.01	0.003	0.003
Ferimzone	0.99	0.99	0.96	1.00	0.002	0.001
Butafenacil	1.02	0.98	0.98	0.84	0.001	0.001
Furathiocarb	1.00	1.03	0.99	0.99	0.001	0.001
Furametpyr	1.05	1.01	0.90	0.98	0.001	0.001
Propaquizafop	1.02	1.02	0.98	0.96	0.001	0.001
Pencycuron	1.05	0.94	0.96	1.01	0.002	0.001

表 2-1 食品マトリックスによる測定への影響及び定量限界(ESI(+)) (続き)

Compound	Matrix effect ^{a)}				LOQ (mg/kg) ^{b)}	
	Soybean		Spinach		Soybean	Spinach
	0.1 (µg/mL)	0.01 (µg/mL)	0.1 (µg/mL)	0.01 (µg/mL)		
Bendiocarb	0.99	1.01	0.90	0.99	0.005	0.002
Pentoxazone	0.91	— ^b	0.92	— ^b	0.07	0.08
Methiocarb	1.01	0.97	0.95	1.05	0.005	0.005
Linuron	1.15	0.93	1.04	1.06	0.005	0.003
Avermectin B1a	0.89	— ^b	0.98	— ^b	0.02	0.02
Cycloate	1.01	0.99	1.19	0.96	0.004	0.002
Silafluofen	0.84	1.02	1.04	1.02	0.005	0.003
Di-allate	0.96	0.89	1.07	0.74	0.008	0.01
Tralkoxydim	1.00	— ^b	0.88	— ^b	0.02	0.02
Tridemorph	0.98	1.02	1.01	0.98	0.004	0.004
Phenmedipham	1.09	1.07	0.97	1.00	0.002	0.002
Azimsulfuron	0.98	0.94	0.97	0.94	0.001	0.001
Iodosulfuron-methyl	0.94	0.93	0.86	0.96	0.002	0.003
Imazaquin	1.00	1.00	1.01	0.96	0.001	0.001
Ethametsulfuron-methyl	0.97	0.97	0.91	0.99	0.001	0.001
Ethoxysulfuron	1.01	0.99	0.90	0.98	0.001	0.001
Clodinafop	1.08	0.90	0.95	0.96	0.004	0.005
Cloransulam-methyl	1.03	1.01	0.91	0.73	0.003	0.001
Chlorimuron-ethyl	1.00	0.97	0.90	0.98	0.001	0.001
Chlorsulfuron	1.01	0.82	0.96	0.91	0.001	0.001
Diclosulam	1.10	1.04	0.97	1.00	0.01	0.002
Sulfentrazone	1.03	1.04	0.92	1.28	0.002	0.001
Sulfosulfuron	1.07	0.93	0.93	1.02	0.007	0.002
Thifensulfuron-methyl	1.01	0.92	0.97	0.95	0.001	0.001
Triasulfuron	0.94	0.83	0.63	0.57	0.001	0.001
Triflusulfuron-methyl	1.03	1.00	0.90	0.92	0.001	0.001
Trifloxysulfuron	1.00	0.95	0.84	0.87	0.001	0.001
Haloxyfop	1.10	0.98	1.00	1.22	0.007	0.005
Flumetsulam	0.97	0.97	1.01	0.95	0.003	0.001
Florasulam	1.06	0.92	0.99	0.91	0.003	0.001
Penoxsulam	1.02	1.00	0.95	0.96	0.001	0.001
Forchlorfenuron	1.02	0.93	0.85	1.01	0.002	0.002
1-Naphthylacetic acid	0.92	0.73	0.89	0.70	0.006	0.002
Fomesafen	1.00	0.91	0.98	1.02	0.004	0.001
Imazosulfuron	0.97	0.93	0.85	0.96	0.001	0.001
Cyclosulfamuron	1.03	0.98	0.93	1.00	0.001	0.001
Diclomezine	0.99	0.85	1.02	1.08	0.005	0.003
Cinosulfuron	1.00	0.98	1.00	1.00	0.001	0.001
Thidiazuron	0.89	0.77	0.90	0.89	0.001	0.002
Tribenuron-methyl	1.06	1.14	0.88	0.98	0.002	0.001

表 2-1 食品マトリックスによる測定への影響及び定量限界(ESI(+)) (続き)

Compound	Matrix effect ^{a)}				LOQ (mg/kg) ^{b)}	
	Soybean		Spinach		Soybean	Spinach
	0.1 (µg/mL)	0.01 (µg/mL)	0.1 (µg/mL)	0.01 (µg/mL)		
Naptalam	0.97	0.97	0.89	0.91	0.004	0.002
Halosulfuron-methyl	0.99	0.95	0.88	0.94	0.001	0.001
Pyrazosulfuron-ethyl	0.94	0.86	0.87	1.00	0.002	0.001
Fenhexamid	0.97	1.01	1.00	1.04	0.002	0.001
Flazasulfuron	0.96	0.90	0.92	0.89	0.001	0.001
Primisulfuron-methyl	1.07	0.89	0.92	0.98	0.003	0.001
Fluazifop	1.06	0.97	0.95	0.98	0.002	0.001
Prosulfuron	1.00	0.96	0.87	0.96	0.001	0.001
Propoxy carbazone	1.03	0.83	0.81	0.48	0.001	0.001
Bensulfuron-methyl	1.00	0.95	0.89	0.95	0.001	0.001
Foramsulfuron	0.97	0.92	0.98	0.85	0.002	0.002
Mesosulfuron-methyl	0.92	0.94	0.82	0.57	0.002	0.001
Metosulam	1.03	0.96	0.88	0.97	0.002	0.001
Metsulfuron-methyl	1.00	0.81	0.96	0.71	0.001	0.002

a) Peak area ratio of matrix standard to that of standard in solvent.

b) S/N=10

c) S/N<10

d) Found in blank sample.

表 2-2 食品マトリックスによる測定への影響及び定量限界(ESI(-))

Compound	Matrix effect ^{a)}				LOQ (mg/kg) ^{b)}	
	Soybean		Spinach		Soybean	Spinach
	0.1 (µg/mL)	0.01 (µg/mL)	0.1 (µg/mL)	0.01 (µg/mL)		
Carpropamid	1.19	— ^c	1.15	— ^c	0.03	0.03
Clothianidin	0.99	0.90	1.02	1.13	0.002	0.001
Chloridazon	1.08	— ^c	1.10	— ^c	0.02	0.02
Tebufenozide	1.13	1.07	1.10	1.19	0.004	0.006
Novaluron	1.06	0.92	1.08	1.16	0.001	0.001
Diuron	1.16	0.93	1.17	1.14	0.002	0.003
Teflubenzuron	1.07	0.83	1.10	1.11	0.002	0.001
Triflumuron	1.13	0.95	1.14	1.19	0.001	0.001
Flufenoxuron	1.07	1.04	— ^d	— ^d	0.002	— ^d
Boscalid	1.20	0.98	1.19	1.17	0.006	0.006
Methoxyfenozide	1.17	1.06	1.19	1.09	0.008	0.007
Isoxaflutole	1.12	0.82	1.10	0.93	0.004	0.004
Clomeprop	1.17	— ^c	1.19	— ^c	0.02	0.03
Diflubenzuron	1.12	0.99	1.13	0.98	0.002	0.001
Naproanilide	1.04	— ^c	1.05	— ^c	0.02	0.02
Oryzalin	1.14	1.00	1.04	1.13	0.007	0.001
Tralkoxydim	1.10	0.89	1.05	0.97	0.007	0.007
Hexaflumuron	1.10	0.92	1.01	1.10	0.001	0.001
Lufenuron	1.10	1.05	1.03	1.20	0.001	0.001
Azimsulfuron	1.07	— ^c	0.93	— ^c	0.03	0.03
Iodosulfuron-methyl	1.04	1.04	0.85	1.09	0.008	0.009
Imazaquin	1.05	0.98	0.88	1.00	0.006	0.006
Ethametsulfuron-methyl	1.11	— ^c	1.07	— ^c	0.03	0.03
Ethoxysulfuron	1.08	— ^c	0.95	— ^c	0.02	0.02
Clodinafop	1.14	0.82	1.07	1.15	0.01	0.01
Cloransulam-methyl	0.98	1.10	0.88	1.10	0.005	0.01
Chlorimuron-ethyl	1.03	0.84	0.94	0.83	0.004	0.009
Chlorsulfuron	1.08	— ^c	0.94	— ^c	0.03	0.02
Diclosulam	0.93	0.90	0.98	1.20	0.007	0.008
Sulfentrazone	1.04	1.18	1.02	1.25	0.008	0.008
Thifensulfuron-methyl	1.01	— ^c	0.93	— ^c	0.02	0.02
Triasulfuron	1.10	— ^c	0.94	— ^c	0.02	0.02
Triflusulfuron-methyl	1.13	— ^c	1.03	— ^c	0.02	0.02
Trifloxysulfuron	1.14	— ^c	0.96	— ^c	0.02	0.02
Haloxypop	0.98	— ^c	0.81	— ^c	0.02	0.02
Flumetsulam	1.15	— ^c	0.93	— ^c	0.04	0.02
Florasulam	0.98	0.86	0.91	1.09	0.006	0.006
Penoxsulam	0.92	1.04	0.92	1.13	0.005	0.003
Forchlorfenuron	0.93	0.94	1.08	1.11	0.001	0.001
2,4-D	1.06	1.03	0.96	1.05	0.008	0.008

表 2-2 食品マトリックスによる測定への影響及び定量限界(ESI(-)) (続き)

Compound	Matrix effect ^{a)}				LOQ (mg/kg) ^{b)}	
	Soybean		Spinach		Soybean	Spinach
	0.1 (µg/mL)	0.01 (µg/mL)	0.1 (µg/mL)	0.01 (µg/mL)		
MCPA	1.07	0.98	0.96	1.16	0.007	0.007
MCPB	1.19	— ^c	1.12	— ^c	0.07	0.03
Ioxynil	1.02	0.96	0.98	1.18	0.001	0.001
Acifluorfen	1.07	0.99	1.05	1.09	0.002	0.002
Cloprop	1.13	1.12	0.95	1.19	0.009	0.009
4-Chlorophenoxyacetic acid	1.05	— ^c	0.90	— ^c	0.02	0.02
Cyclanilide	1.10	0.96	1.03	1.19	0.001	0.001
Dichlorprop	1.07	0.92	1.06	1.30	0.005	0.005
Gibberellic acid	0.96	— ^c	0.82	— ^c	0.09	0.08
Triclopyr	1.07	— ^c	1.05	— ^c	0.02	0.02
Fluroxypyr	1.01	— ^c	0.85	— ^c	0.08	0.04
Bromoxynil	1.05	0.98	0.93	1.18	0.001	0.001
Fomesafen	1.15	0.96	1.07	1.14	0.001	0.002
Mecoprop	1.06	0.84	1.02	1.09	0.009	0.008
Imazosulfuron	1.09	0.93	0.94	1.08	0.005	0.004
Cyclosulfamuron	1.10	0.92	1.03	1.13	0.004	0.004
Cinosulfuron	0.95	— ^c	0.90	— ^c	0.03	0.03
Thidiazuron	1.01	0.90	1.02	1.33	0.001	0.001
Tribenuron-methyl	0.96	— ^c	0.97	— ^c	0.03	0.02
Naptalam	1.10	0.95	0.89	1.10	0.006	0.005
Halosulfuron-methyl	1.11	0.81	0.94	0.93	0.005	0.004
Pyrazosulfuron-ethyl	1.09	— ^c	0.91	— ^c	0.02	0.02
Fenhexamid	1.08	0.93	0.94	1.12	0.005	0.007
Flazasulfuron	1.05	— ^c	0.97	— ^c	0.02	0.02
Primisulfuron-methyl	1.18	1.05	1.05	1.29	0.001	0.001
Fluazifop	1.13	0.81	1.14	1.20	0.009	0.01
Prosulfuron	1.10	1.08	1.04	1.13	0.004	0.003
Propoxycarbazone	1.03	— ^c	1.05	— ^c	0.03	0.02
Bensulfuron-methyl	1.09	— ^c	1.04	— ^c	0.03	0.02
Foramsulfuron	1.08	— ^c	1.02	— ^c	0.02	0.02
Mesosulfuron-methyl	0.98	— ^c	1.04	— ^c	0.02	0.02
Metosulam	1.15	— ^c	0.86	— ^c	0.04	0.04
Metsulfuron-methyl	1.08	— ^c	0.85	— ^c	0.02	0.02

a) Peak area ratio of matrix standard to that of standard in solvent.

b) S/N=10

c) S/N<10

d) Found in blank sample.

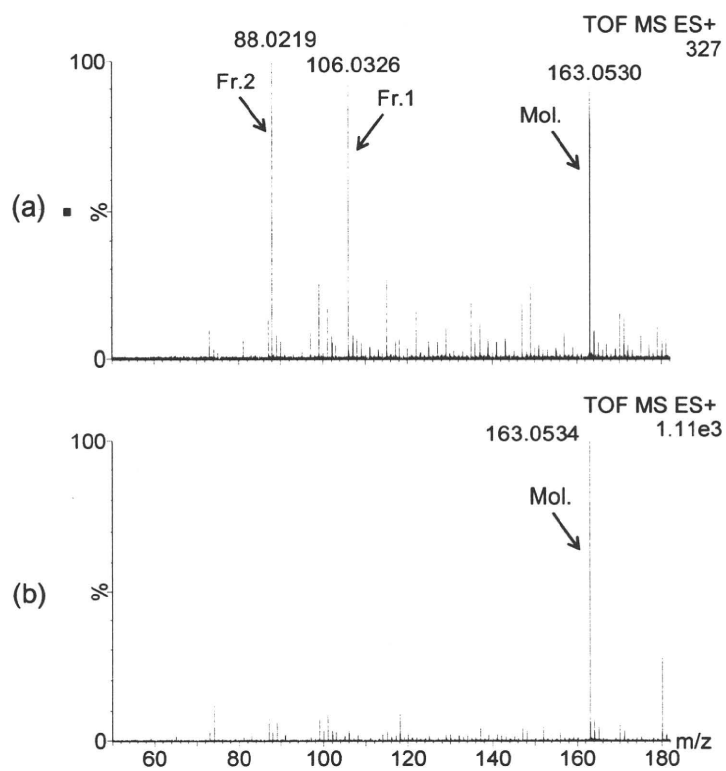


図 7-1 メソミルのマスペクトル

アパーチャー1 電圧 (a) 15 V, (b) 5 V

Mol.: $[M+H]^+$ (calcd. m/z 163.0536), Fr.1: $[C_3H_8NOS]^+$ (calcd. m/z 106.0321),

Fr.2: $[C_3H_6NS]^+$ (calcd. m/z 88.0215)

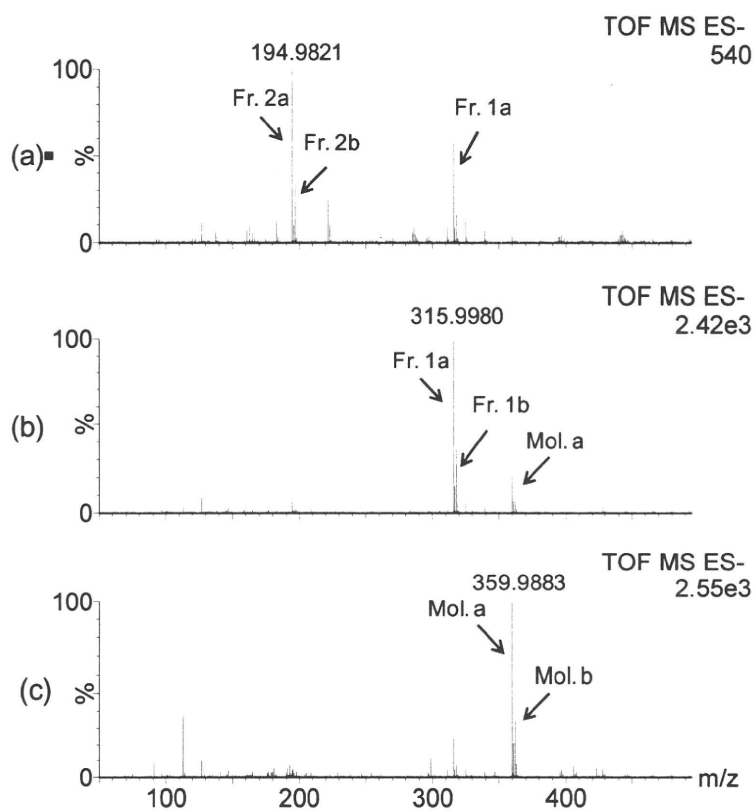


図 7-2 アシフルオルフェンのマススペクトル

アパーチャー1 電圧 (a) 50 V, (b) 20 V, (c) 5 V

Mol.a: $[M-H]^-$ (calcd. m/z 359.9892), Mol.b: $[C_{14}H_6^{37}ClF_3NO_5]^-$ (calcd. m/z 361.9863), Fr.1a: $[C_{13}H_6ClF_3NO_3]^-$ (calcd. m/z 315.9994), Fr.1b: $[C_{13}H_6^{37}ClF_3NO_3]^-$ (calcd. m/z 317.9964), Fr.2a: $[C_7H_3ClF_3O]^-$ (calcd. m/z 194.9830), Fr.2b: $[C_7H_3^{37}ClF_3O]^-$ (calcd. m/z 196.9801)

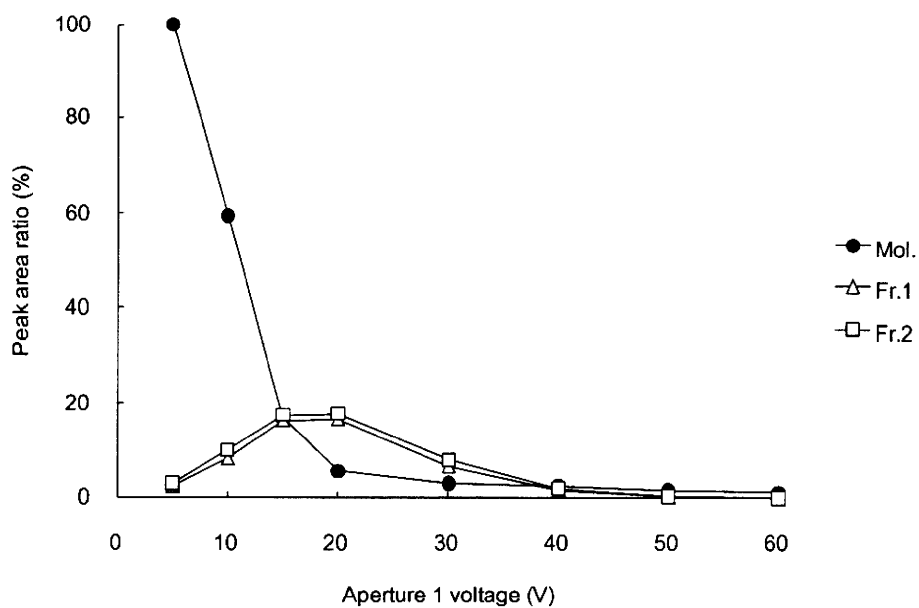


図 8-1 メソミル及びそのフラグメントイオンのピーク面積に対するアパーチャー1 電圧の影響
 (アパーチャー1 電圧 5 V での定量イオンのピーク面積を 100 としたときの同位体イオン及び
 主なフラグメントイオンのピーク面積比(%))

Mol.: $[M+H]^+$, Fr.1: $[C_3H_8NOS]^+$, Fr.2: $[C_3H_6NS]^+$

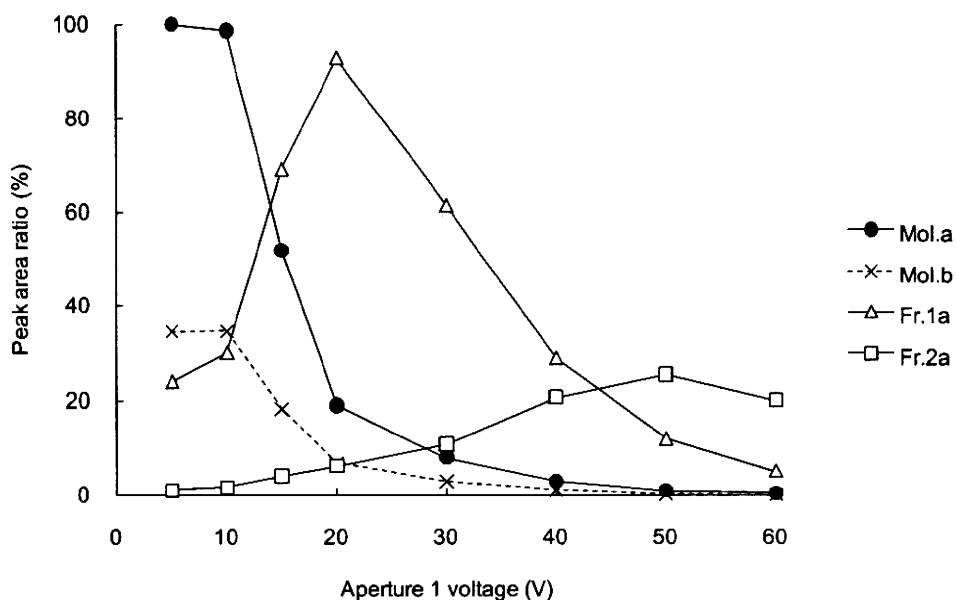


図 8-2 アシフルオルフェン及びそのフラグメントイオンのピーク面積に対するアパーチャー1電圧の影響

(アパーチャー1 電圧 5 V での定量イオンのピーク面積を 100 としたときの同位体イオン及び主なフラグメントイオンのピーク面積比(%))

Mol.a: $[M-H]^-$, Mol.b: $[C_{14}H_6^{37}ClF_3NO_5]^-$, Fr.1a: $[C_{13}H_6ClF_3NO_3]^-$,

Fr.2a: $[C_7H_3ClF_3O]^-$

Ⅱ. 分担研究報告

2. 畜水産物中残留動物用医薬品及び農薬の 包括的スクリーニング分析法の開発

研究分担者 坂井隆敏