Original: Japanese
Provisional translation

Multi-residue Method I for Agricultural Chemicals by LC−MS (Agricultural Products)

1. Analytes

Grains, legumes, nuts, seeds, fruits and vegetables, see Table 1.

Tea leaves and hops, see Table 2.

2. Application

Agricultural products

3. Instruments

Liquid chromatograph-mass spectrometer (LC-MS) or liquid chromatograph-tandem mass spectrometer (LC-MS/MS)

4. Reagents

Use the reagents listed in Section 3 of the General Rules, except for the following.

Graphitized carbon black/ethylenediamine-*N*-propylsilanized silica gel layered cartridge (500 mg/500 mg): A polyethylene tube of 12–13 mm in inside diameter packed with 500 mg each of graphitized carbon black in the upper layer and ethylenediamine-*N*-propylsilanized silica gel in the lower layer, or other cartridges with equal separation characteristics.

0.5 mol/L phosphate buffer solution (pH 7.0): Weigh 52.7 g of dipotassium hydrogen phosphate (K_2HPO_4) and 30.2 g of potassium dihydrogen phosphate (KH_2PO_4), dissolve in approximately 500 mL of water, adjust pH to 7.0 using 1 mol/L sodium hydroxide solution or 1 mol/L hydrochloric acid, and add water to make 1 L.

Reference standards for each agricultural chemical: Reference standards with known purity for each agricultural chemical. (When individual testing methods for each agricultural chemical indicate purity for reference standards, they should be followed. If not, it is preferable to use reference standards with a purity of 95% or higher.)

5. Procedure

1) Extraction

i) Grains, legumes, nuts and seeds

Add 20 mL of water to 10.0 g of sample and let stand for 30 min. Add 50 mL of acetonitrile, homogenize, and filter with suction. Add 20 mL of acetonitrile to the residue on the filter paper, homogenize, and filter with suction. Combine the resulting filtrates, and add acetonitrile to make exactly 100 mL. Take exactly a 20 mL aliquot of the solution, add 10 g of sodium chloride and 20 mL of 0.5 mol/L phosphate buffer solution

(pH 7.0), and shake for 10 min. Let stand, and discard the separated aqueous layer.

Inject 10 mL of acetonitrile into an octadecylsilanized silica gel cartridge (1,000 mg) and discard the effluent. Transfer the acetonitrile layer above to the cartridge and add another 5 mL of acetonitrile. Collect the total eluate, concentrate at below 40°C, and remove the solvent. Dissolve the residue in 2 mL of acetonitrile and toluene (3:1, v/v).

ii) Fruits and vegetables

Add 50 mL of acetonitrile to 20.0 g of sample, homogenize, and filter with suction. Add 20 mL of acetonitrile to the residue on the filter paper, homogenize, and filter with suction. Combine the resulting filtrates, and add acetonitrile to make exactly 100 mL. Take exactly a 20 mL aliquot of the solution, add 10 g of sodium chloride and 20 mL of 0.5 mol/L phosphate buffer solution (pH 7.0), and shake for 10 min. Let stand, and discard the separated aqueous layer. Concentrate the acetonitrile layer at below 40°C and remove the solvent. Dissolve the residue in 2 mL of acetonitrile and toluene (3:1, v/v).

iii) Tea leaves and hops

Add 20 mL of water to 5.00 g of sample and let stand for 30 min. Add 50 mL of acetonitrile, homogenize, and filter with suction. Add 20 mL of acetonitrile to the residue on the filter paper, homogenize, and filter with suction. Combine the resulting filtrates, and add acetonitrile to make exactly 100 mL. Take exactly a 5 mL aliquot of the solution and add 15 mL of acetonitrile. Then, add 10 g of sodium chloride and 20 mL of 0.5 mol/L phosphate buffer solution (pH 7.0), and shake for 10 min. Let stand, and discard the separated aqueous layer.

Add 10 mL of acetonitrile to an octadecylsilanized silica gel cartridge (1,000 mg) and discard the effluent. Transfer the acetonitrile layer above to the cartridge and add another 5 mL of acetonitrile. Collect the total eluate, concentrate at below 40°C and remove the solvent. Dissolve the residue in 2 mL of acetonitrile and toluene (3:1, v/v).

2) Clean-up

i) Grains, legumes, nuts, seeds, fruits and vegetables

Inject 10 mL of acetonitrile and toluene (3:1, v/v) into a graphitized carbon black/aminopropylsilanized silica gel layered cartridge (500 mg/500 mg) and discard the effluent. Transfer the solution obtained in 1) to the cartridge, add 20 mL of acetonitrile and toluene (3:1, v/v), concentrate the total eluate at below 40°C, and remove the solvent. Dissolve the residue in methanol to make exactly 4 mL, and use this solution as the test solution.

ii) Tea leaves and hops

Add 10 mL of acetonitrile and toluene (3:1, v/v) mixed solution to a graphitized carbon

black/ethylenediamine-*N*-propylsilanized silica gel layered cartridge (500 mg/500 mg) and discard the effluent. Transfer the solution obtained in 1) to the cartridge, add 20 mL of acetonitrile and toluene (3:1, v/v) mixed solution, concentrate the total eluate at below 40°C, and remove the solvent. Dissolve the residue in methanol to make exactly 1 mL, and use this solution as the test solution.

6. Calibration curve

Prepare stock standard solutions by dissolving the reference standards of each agricultural chemical in appropriate solvents. Mix each stock standard solution appropriately, and prepare several methanol solutions containing each agricultural chemical in an appropriate concentration range. Inject each standard solution into LC-MS or LC-MS/MS, and make calibration curves by peak-height or peak-area method.

7. Quantification

Inject the test solutions into LC-MS or LC-MS/MS, and calculate the concentrations of each agricultural chemical from the calibration curves prepared in **6**.

8. Confirmation

Confirm using LC-MS or LC-MS/MS.

9. Measurement conditions

(Example)

Column: Octadecylsilanized silica gel, 2–2.1 mm inside diameter, 150 mm in length and 3–3.5 μm in particle diameter

Column temperature: 40°C

Mobile phase: Control the gradient by mixing the mobile phases A and B as directed in the following table.

Mobile phase A: 5 mmol/L ammonium acetate solution

Mobile phase B: 5 mmol/L ammonium acetate-methanol solution

Time (min)	Mobile phase A (%)	Mobile phase B (%)
0	85	15
1	60	40
3.5	60	40
6	50	50
8	45	55
17.5	5	95
35	5	95

Ionization mode: ESI (+) and ESI (−)

Major monitoring ions (*m/z*): See Tables 1 and 2.

Injection volume: 5 μL

Expected retention time: See Tables 1 and 2.

10. Limit of quantification

See Tables 1 and 2.

11. Explanatory note

1) Outline of analytical method

i) Grains, legumes, nuts and seeds

The method consists of extracting each agricultural chemical from the sample using acetonitrile, dehydrating by salting out, cleaning up with an octadecylsilanized silica gel cartridge and a graphitized carbon black/aminopropylsilanized silica gel layered cartridge, and quantifying and confirming using LC-MS or LC-MS/MS.

ii) Fruits and vegetables

The method consists of extracting each agricultural chemical from the sample using acetonitrile, dehydrating by salting out, cleaning up with a graphitized carbon black/aminopropylsilanized silica gel layered cartridge, and quantifying and confirming using LC-MS or LC-MS/MS.

iii) Tea leaves and hops

The method consists of extracting each agricultural chemical from the sample using acetonitrile, dehydrating by salting out, cleaning up with an octadecylsilanized silica gel cartridge and a graphitized carbon black/ethylenediamine-*N*-propylsilanized silica gel layered cartridge, and quantifying and confirming using LC-MS or LC-MS/MS.

2) Notes

i) The table lists the analytes to which this method can be applied in Japanese syllabary order. However, note that some agricultural chemicals under regulations may contain compounds such as metabolites that cannot be applied to this method. In addition, isomers with different retention times are indicated separately in the “Analytes” column.

ii) This method does not ensure simultaneous analysis of all the analytes listed in the Tables. Since interactions between compounds may cause decomposition or interference with measurements, verifying these points before combining analytes is necessary.

iii) Sodium phosphate can be used to prepare phosphate buffer solutions.

iv) If the amount of sodium chloride (10 g) added to the acetonitrile extract is too large, it can be reduced but added enough to be fully saturated.

v) If an emulsion was formed during salting out, it is recommended to centrifuge at 3,000 rpm for 5 min.

vi) Concentration and complete removal of the solvent should be performed gently using a

nitrogen stream.

vii) If water remains when concentrating the eluate after clean-up using an octadecylsilanized silica gel cartridge or the acetonitrile layer after salting out, add about 5 mL of acetonitrile and concentrate at below 40°C.

viii) When clean-up is insufficient in the case of fruits and vegetables, clean-up using an octadecylsilanized silica gel cartridge may be performed as in the case of grains, legumes, nuts, seeds, and teas, if necessary.

ix) A graphitized carbon black/ethylenediamine-*N*-propylsilanized silica gel layered cartridge can be used for fruits, vegetables, grains, legumes, nuts, and seeds as well, if verified.

x) Depending on the sensitivity of LC-MS or LC-MS/MS, the test solution may be further diluted with methanol.

xi) The measurements should be performed immediately after preparing the test solutions since some agricultural chemicals are unstable, especially in methanol solutions. In addition, the solutions for the calibration curves should be prepared just before use.

xii) Matrix-matched standard solutions and standard addition methods may be required to obtain accurate measurement results.

xiii) Since the limit of quantification varies depending on the instrument used, the concentration rate, and the injection volume of the test solution, optimal conditions should be considered when necessary.

xiv) In LC-MS or LC-MS/MS measurements, it is recommended to wash the cartridge by increasing the methanol concentration in the mobile phase after the target analyte is eluted to reduce the influence of matrix carryover in the sample.

xv) Thiodicarb is changed to methomyl during sample preparation in some crops.

xvi) When a separate analytical method is indicated for teas other than powdered tea, it should be followed.

xvii) Food items used to develop the analytical method: brown rice, soybean, peanut, spinach, cabbage, potato, eggplant, orange, apple and tea (green tea, powdered tea, oolong tea and black tea).

12. Reference

Fillion, J., *et.al.*, Multiresidue method for the determination of residues of 251 pesticides in fruits and vegetables by gas chromatography/mass spectrometry and liquid chromatography with fluorescence detection, *Journal of AOAC International*, 83, 698–713, 2000.

13. Type

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Table 1. Multi-residue Method I for Agricultural Chemicals by LC-MS (Agricultural Products): grains, legumes, nuts, seeds, fruits and vegetables

Agricultural Chemicals	Analytes[1)]	RRT[2)]	Major monitoring ions (*m/z*) [3)]						Limit of quantification (mg/kg)[4)]
EPN	EPN	1.42	+324→296	+324→157					0.01
XMC	XMC	0.91	+180→123	+180→108					0.01
Acrinathrin	Acrinathrin	1.55	+559→208	+559→181	–540→372	–540→300			0.01
Azafenidin	Azafenidin	1.00	+338→299	+338→264					0.01
Acibenzolar-*S*-methyl	Acibenzolar-*S*-methyl	1.10	+211→136	+211→91					0.01
Azinphos-methyl	Azinphos-methyl	1.04	+318→160	+318→132	+318→77				0.01
Acetamiprid	Acetamiprid	0.41	+223→126	+223→90	+223→56				0.01
Acetochlor	Acetochlor	1.23	+270→224	+270→148					0.01
Azoxystrobin	Azoxystrobin	1.08	+404→372	+404→344	+404→329				0.01*
Atrazine	Atrazine	0.97	+216→174	+216→104	+216→96				0.01
Anilofos	Anilofos	1.22	+368→199	+368→125					0.01
Abamectin	Avermectin B1a	1.58	+891→567	+891→305	+891→145	+891→95			0.01
	Avermectin B1b	1.63	+877→291	+877→145	+877→95	–857→551	–857→229		0.01
	8,9-*Z* avermectin B1a	1.61	+891→567	+891→305	+891→145	+891→95			0.01
Amisulbrom	Amisulbrom	1.35	+468→229	+468→108	+466→227	+466→108			0.01
Ametryn	Ametryn	1.11	+228→186	+228→96	+228→68				0.01
Alachlor	Alachlor	1.23	+270→238	+270→162					0.01
Aramite	Aramite	1.45	+352→255	+352→191	+352→91	+352→57			0.01
Aldicarb and Aldoxycarb	Aldicarb	0.64	+208→116	+208→115	+208→89				0.01*
	Aldoxycarb	0.32	+240→148	+240→86	+223→148	+223→86			0.01
Isouron	Isouron	0.76	+212→167	+212→72					0.01*
Isoxathion	Isoxathion	1.34	+341→105	+341→97					0.01*
Isoxaflutole	Isoxaflutole	1.00	+360→251	+360→220	+360→144				0.01
Isopyrazam	Isopyrazam (*syn, anti*)	1.40	+360→340	+360→320	+360→244				0.01
Isofenphos	Isofenphos	1.34	+346→245	+346→217					0.01
Isofenphos	Isofenphos-oxon	1.22	+330→229	+330→201					0.01
Isoprocarb	Isoprocarb	0.97	+194→137	+194→95					0.01
Isoprothiolane	Isoprothiolane	1.18	+291→231	+291→189					0.01
Ipfencarbazone	Ipfencarbazone	1.32	+427→198	+427→156					0.01
Iprodione	*N*-(3,5-dichlorophenyl)-3-isopropyl-2,4-dioxoimidazolidine-1-carboxamide (iprodione metabolite)	1.31	+330→143	+330→101	–330→141	–328→141	–328→99		0.01*
Iprovalicarb	Iprovalicarb	1.20	+321→203	+321→119					0.01
Iprobenfos	Iprobenfos	1.31	+289→205	+289→91					0.01
Imazalil	Imazalil	1.27	+299→161	+297→255	+297→159				0.01*
Imicyafos	Imicyafos	0.76	+305→235	+305→201					0.01
Imidacloprid	Imidacloprid	0.40	+256→209	+256→175					0.01*
Indanofan	Indanofan	1.23	+341→187	+341→175					0.01
Indoxacarb	Indoxacarb	1.38	+528→203	+528→150					0.01
Esprocarb	Esprocarb	1.49	+266→91	+266→71					0.01
Ethaboxam	Ethaboxam	0.94	+321→200	+321→183					0.01
Ethion	Ethion	1.46	+385→199	+385→143					0.01
Ethiprole	Ethiprole	1.13	+397→351	+397→255					0.01
Edifenphos	Edifenphos	1.30	+311→283	+311→111	+311→109				0.01
Etoxazole	Etoxazole	1.46	+360→304	+360→177	+360→141				0.01
Ethoprophos	Ethoprophos	1.26	+243→173	+243→131	+243→97				0.01
Etrimfos	Etrimfos	1.30	+293→265	+293→125					0.01
Epoxiconazole	Epoxiconazole	1.23	+330→141	+330→121	+330→101				0.01
Oxadiazon	Oxadiazon	1.50	+345→303	+345→220	+345→177				0.01
Oxadiargyl	Oxadiargyl	1.26	+358→341	+358→223	+358→151	+341→258	+341→223		0.01
Oxadixyl	Oxadixyl	0.69	+279→219	+279→133	+279→132				0.01*
Oxaziclomefone	Oxaziclomefone	1.42	+376→190	+376→161					0.01
Oxamyl	Oxamyl	0.32	+237→90	+237→72					0.01
Oxycarboxin	Oxycarboxin	0.54	+268→175	+268→147					0.01
Oxyfluorfen	Oxyfluorfen	1.47	+362→316	+362→237					0.01
Cadusafos	Cadusafos	1.37	+271→159	+271→131	+271→97				0.01
Cafenstrole	Cafenstrole	1.20	+351→100	+351→72					0.01
Carbaryl	Carbaryl	0.88	+202→145	+202→127					0.01*
Carfentrazone-ethyl	Carfentrazone-ethyl	1.21	+412→366	+412→346					0.01*

Agricultural Chemicals	Analytes[1)]	RRT[2)]	Major monitoring ions (*m/z*) [3)]						Limit of quantification (mg/kg)[4)]
Carpropamid	Carpropamid	1.26	+336→139	+336→103	+334→139	+334→103			0.01
Quinalphos	Quinalphos	1.28	+299→163	+299→147	+299→97				0.01
Carbofuran	Carbofuran	0.82	+222→165	+222→123					0.01*
	3- hydroxycarbofuran	0.48	+255→220	+255→163	+238→220	+238→181	+238→163		0.01*
Quizalofop	Quizalofop-ethyl	1.34	+373→299	+373→271	+373→91				0.01
	Quizalofop-P-tefuryl	1.36	+429→299	+429→85					0.01
Quinoxyfen	Quinoxyfen	1.49	+308→197	+308→162					0.01
Cumyluron	Cumyluron	1.16	+303→185	+303→125					0.01
Kresoxim-methyl	Kresoxim-methyl	1.29	+331→314	+331→116	+314→267	+314→222	+314→131	+314→116	0.01*
Cloquintocet-mexyl	Cloquintocet-mexyl	1.45	+336→238	+336→192					0.01
Clothianidin	Clothianidin	0.42	+250→169	+250→132					0.01*
Chromafenozide	Chromafenozide	1.21	+395→339	+395→175	+395→147	+395→91			0.01
Clomeprop	Clomeprop	1.44	+324→203	+324→148	+324→120				0.01
Chlorantraniliprole	Chlorantraniliprole	1.05	+484→453	+484→286	+484→112	+482→451	+482→284		0.01*
Chloridazon	Chloridazon	0.50	+222→104	+222→92	+222→77				0.01
Chlorpyrifos	Chlorpyrifos	1.48	+350→198	+350→97					0.01
Chlorpyrifos-methyl	Chlorpyrifos-methyl	1.37	+324→292	+324→125	+322→290	+322→125			0.01*
Chlorfenapyr	Chlorfenapyr	1.44	–349→268	–349→131	–349→81				0.01
Chlorfenvinphos	Chlorfenvinphos (*E*)	1.35	+361→155	+361→99	+359→170	+359→155	+359→127		0.01*
Chlorfenvinphos	Chlorfenvinphos (*Z*)	1.31	+359→155	+359→99					0.01*
Chlorbufam	Chlorbufam	1.10	+224→172	+224→154					0.01
Chlorpropham	Chlorpropham	1.17	+231→172	+214→172	+214→154				0.01
Chloroxuron	Chloroxuron	1.19	+291→218	+291→164	+291→72				0.01
Cyazofamid	Cyazofamid	1.20	+327→108	+325→261	+325→108				0.01
Cyanazine	Cyanazine	0.73	+241→214	+241→104	+241→96				0.01
Diuron	Diuron	1.01	+233→160	+233→72					0.01*
Diethofencarb	Diethofencarb	1.10	+268→226	+268→124					0.01
Cyenopyrafen	Cyenopyrafen	1.44	+394→310	+394→254					0.01
Cycloate	Cycloate	1.34	+216→154	+216→83					0.01
Diclocymet	Diclocymet (Isomer 1)	1.25	+313→173	+313→137	+313→102				0.01
	Diclocymet (Isomer 2)	1.28	+313→173	+313→137	+313→102				
Diclofop-methyl	Diclofop-methyl	1.45	+358→281	+358→120	+341→281	+341→120			0.01
Dithiopyr	Dithiopyr	1.40	+402→354	+402→272	+402→248				0.01
Cyhalofop-butyl	Cyhalofop-butyl	1.38	+375→256	+375→120	+358→256	+358→158			0.01
Difenoconazole	Difenoconazole (Isomer 1, 2)	1.36	+406→251	+406→111					0.01
Cyflufenamid	Cyflufenamid	1.33	+413→295	+413→241	+413→203				0.01
Diflufenican	Diflufenican	1.31	+395→266	+395→246	+395→238	–393→329	–393→272		0.002
Diflubenzuron	Diflubenzuron	1.18	+311→158	+311→141					0.01*
Cyflumetofen	Cyflumetofen	1.45	+465→173	+465→145	+448→249	+448→173	+448→145		0.01
Cyproconazole	Cyproconazole (Isomer 1)	1.17	+292→125	+292→70					0.01
	Cyproconazole (Isomer 2)	1.19							
Cyprodinil	Cyprodinil	1.28	+226→108	+226→93	+226→92				0.01
Cypermethrin	Cypermethrin	1.53	+435→193	+433→191	+416→191	+416→127			0.01*
Simazine	Simazine	0.80	+202→132	+202→124	+202→104	+202→96			0.01
Simeconazole	Simeconazole	1.19	+294→135	+294→73	+294→70				0.01
Dimethametryn	Dimethametryn	1.26	+256→186	+256→91	+256→68				0.01
Dimethirimol	Dimethirimol	0.94	+210→140	+210→71					0.01
Dimethenamid	Dimethenamid (*RS*)	1.14	+276→244	+276→168					0.01
Dimethoate	Dimethoate	0.42	+230→199	+230→125					0.01*
Dimethomorph	Dimethomorph (*E*)	1.14	+388→301	+388→165					0.01
	Dimethomorph (*Z*)	1.18	+388→301	+388→165					0.01
Cymoxanil	Cymoxanil	0.56	+199→128	+199→111					0.01*
Silafluofen	Silafluofen	1.67	+426→287	+426→168					0.01
Spinosad	Spinosyn A	1.55	+733→142	+733→98	+732→142	+732→98			0.01*
Spiroxamine	Spiroxamine	1.44	+298→144	+298→100					0.01
Spirodiclofen	Spirodiclofen	1.53	+411→313	+411→71					0.01
Zoxamide	Zoxamide	1.35	+336→187	+336→159					0.01
Terbacil	Terbacil	0.82	–215→159	–215→73					0.01
Diazinon	Diazinon	1.32	+305→169	+305→97					0.01*
Diallate	Diallate	1.39	+270→128	+270→109	+270→86				0.01*
Daimuron	Daimuron	1.14	+269→151	+269→119	+269→91				0.01
Thiacloprid	Thiacloprid	0.58	+255→128	+253→126	+253→90	+253→73			0.01

Agricultural Chemicals	Analytes[1)]	RRT[2)]	Major monitoring ions (*m/z*)[3)]						Limit of quantification (mg/kg)[4)]
Tiadinil	Tiadinil	1.19	+268→101	–266→238	–266→71	–266→56			0.01
Thiabendazole	Thiabendazole	0.63	+202→175	+202→131					0.01*
Thiamethoxam	Thiamethoxam	0.36	+292→211	+292→181					0.01
Thiodicarb and Methomyl	Methomyl	0.40	+163→106	+163→88					0.01*
Thiobencarb	Thiobencarb	1.39	+258→125	+258→100	+258→89				0.01
Thifluzamide	Thifluzamide	1.26	+529→148	+529→107	+527→168	+527→148	–525→166	–525→125	0.01
Tetrachlorvinphos	Tetrachlorvinphos (*Z*)	1.24	+367→206	+367→127					0.01
Tetraconazole	Tetraconazole	1.17	+372→159	+372→70					0.01
Tebuconazole	Tebuconazole	1.29	+308→125	+308→70					0.01
Tebuthiuron	Tebuthiuron	0.83	+229→172	+229→116					0.01*
Tebufenozide	Tebufenozide	1.27	+353→297	+353→133	+353→105				0.01
Tebufenpyrad	Tebufenpyrad	1.43	+334→147	+334→145	+334→117				0.01
Teflubenzuron	Teflubenzuron	1.38	+381→158	+381→141					0.01*
Deltamethrin	Deltamethrin	1.54	+523→506	+523→281	+521→279	+504→279	+504→172		0.01*
Terbutryn	Terbutryn	1.27	+242→186	+242→91					0.01
Triadimenol	Triadimenol	1.21	+296→99	+296→70					0.01*
Triadimefon	Triadimefon	1.18	+294→197	+294→69					0.01*
Trichlamide	Trichlamide	1.29	+340→266	+340→121	–340→304	–340→119	–338→146	–338→117	0.01*
Tricyclazole	Tricyclazole	0.62	+190→163	+190→136					0.01*
Triticonazole	Triticonazole	1.18	+318→125	+318→70					0.01
Tridemorph	Tridemorph (Isomer 1, 2)	1.69	+299→130	+299→57	+298→130	+298→98			0.01*
Tribufos	Tribufos	1.62	+315→169	+315→113	+315→57				0.01
Triflumizole	Triflumizole	1.33	+346→278	+346→73					0.01*
	4-chloro-α, α, α-trifluoro-*N*-(1-amino-2-propoxyethylidene)-*o*-toluidine (Triflumizole metabolite)	1.18	+295→278	+295→215	+295→73	+295→72	+295→55		0.01*
Triflumuron	Triflumuron	1.34	+359→156	+359→139					0.01*
Trifloxystrobin	Trifloxystrobin	1.31	+409→186	+409→145					0.01
Triforine	Triforine (Isomer 1)	1.03	+437→392	+435→390	+435→215	+435→98			0.01*
	Triforine (Isomer 2)	1.06	+437→392	+435→390	+435→215	+435→98			0.01*
Tolfenpyrad	Tolfenpyrad	1.37	+384→197	+384→154	+384→145	+384→91			0.01
Naproanilide	Naproanilide	1.23	+292→171	+292→120					0.01
Napropamide	Napropamide	1.23	+272→171	+272→129					0.01
Novaluron	Novaluron	1.36	+493→158	+493→141	–491→471				0.01
Norflurazon	Norflurazon	1.03	+304→284	+304→160	+304→88				0.01
Barban	Barban	1.14	+275→178	+258→178	+258→143	+258→87			0.01
Paclobutrazol	Paclobutrazol	1.15	+294→125	+294→70					0.01
Parathion	Parathion	1.27	+309→236	+292→264	+292→236	+292→94			0.01
Bixafen	Bixafen	1.29	+414→394	+414→266	–412→280	–412→91			0.01
Picolinafen	Picolinafen	1.49	+377→238	+377→145					0.01
Bitertanol	Bitertanol	1.26	+338→269	+338→99	+338→70				0.01
Bifenthrin	Bifenthrin	1.63	+440→181	+440→166	+440→165				0.01
Piperonyl Butoxide	Piperonyl Butoxide	1.46	+356→177	+356→119					0.01*
Pyraclostrobin	Pyraclostrobin	1.29	+390→163	+388→194	+388→164	+388→163	+388→105		0.01
Pyraclonil	Pyraclonil	0.87	+315→276	+315→241	+315→169				0.01
Pyraclofos	Pyraclofos	1.34	+361→257	+361→138					0.01
Pyrazoxyfen	Pyrazoxyfen	1.31	+403→105	+403→91					0.01
Pyrazophos	Pyrazophos	1.27	+374→222	+374→194					0.01*
Pyrazolynate	Pyrazolynate	1.35	+439→173	+439→91					0.01*
Pyraflufen-ethyl	Pyraflufen-ethyl	1.33	+413→339	+413→253					0.01
Pyridaben	Pyridaben	1.50	+366→309	+366→147	+365→309	+365→147			0.01
Pyriftalid	Pyriftalid	1.07	+319→179	+319→139	+319→83				0.01
Pyributicarb	Pyributicarb	1.39	+331→190	+331→181	+331→133	+331→108			0.01
Pyriproxyfen	Pyriproxyfen	1.47	+322→227	+322→96	+322→78				0.01
Pirimicarb	Pirimicarb	0.94	+239→182	+239→72					0.01
Pyriminobac-methyl	Pyriminobac-methyl (*E*)	1.14	+362→330	+362→284					0.01
Pyriminobac-methyl	Pyriminobac-methyl (*Z*)	1.07	+362→330	+362→190	+362→174				0.01
Pirimiphos-methyl	Pirimiphos-methyl	1.35	+306→164	+306→108					0.01*
Pyrimethanil	Pyrimethanil	1.12	+200→107	+200→82	+200→77				0.01
Famoxadone	Famoxadone	1.24	+392→331	+392→238					0.01
Fenamiphos	Fenamiphos	1.25	+304→234	+304→217	+304→202				0.01*

Agricultural Chemicals	Analytes[1)]	RRT[2)]	Major monitoring ions (*m/z*)[3)]						Limit of quantification (mg/kg)[4)]
Fenarimol	Fenarimol	1.21	+331→268	+331→111	+331→81				0.01
Fenoxaprop-ethyl	Fenoxaprop-ethyl	1.41	+362→288	+362→91					0.01*
Fenoxycarb	Fenoxycarb	1.27	+302→116	+302→115	+302→88				0.01*
Fenobucarb	Fenobucarb	1.02	+208→152	+208→95					0.01
Ferimzone	Ferimzone (*E*)	1.13	+255→132	+255→91					0.01
	Ferimzone (*Z*)	1.06	+255→132	+255→124	+255→91				0.01
Fenamidone	Fenamidone	1.12	+312→236	+312→92					0.01
Fensulfothion	Fensulfothion	0.93	+309→281	+309→280	+309→173	+309→157			0.01
Phenthoate	Phenthoate	1.28	+321→247	+321→163	+321→135				0.01*
Fenpyrazamine	Fenpyrazamine	1.20	+332→272	+332→230	+332→216	+332→189			0.01
Fenpyroximate	Fenpyroximate (*E*)	1.48	+422→366	+422→214	+422→135				0.01*
	Fenpyroximate (*Z*)	1.42	+422→366	+422→214	+422→135				0.01*
Fenbuconazole	Fenbuconazole	1.24	+337→125	+337→70					0.01
Fenpropathrin	Fenpropathrin	1.51	+367→350	+367→125	+367→97	+350→125	+350→97		0.01
Fenpropimorph	Fenpropimorph	1.62	+305→147	+305→98	+304→147	+304→130			0.01*
Phenmedipham	Phenmedipham	1.06	+318→168	+318→136					0.01
Butachlor	Butachlor	1.40	+313→238	+313→162	+312→238	+312→162	+312→57		0.01
Butafenacil	Butafenacil	1.13	+492→331	+492→180					0.01
Buprofezin	Buprofezin	1.45	+306→201	+306→106	+306→57				0.01
Furathiocarb	Furathiocarb	1.37	+383→252	+383→195	+383→167				0.01*
Flamprop-methyl	Flamprop-methyl	1.18	+336→105	+336→77					0.01
Furametpyr	Furametpyr	0.96	+335→289	+335→157	+334→290	+334→157			0.01*
Fluazinam	Fluazinam	1.39	–463→416	–463→398					0.01
Fluopicolide	Fluopicolide	1.09	+385→175	+385→173	+383→173	+383→109			0.01
Fluometuron	Fluometuron	0.84	+233→160	+233→72	+233→46				0.01*
Fluquinconazole	Fluquinconazole	1.20	+376→349	+376→307	+376→108				0.01
Fludioxonil	Fludioxonil	1.14	–247→180	–247→126					0.01*
Flusilazole	Flusilazole	1.26	+316→247	+316→165					0.01
Flusulfamide	Flusulfamide	1.22	–413→349	–413→179	–413→171				0.01
Flutriafol	Flutriafol (Isomer 1)	0.86	+302→123	+302→109	+302→70				0.01
	Flutriafol (Isomer 2)	0.96	+302→123	+302→109	+302→70				0.01
Fluvalinate	Fluvalinate	1.57	+503→208	+503→181					0.01
Flufenacet	Flufenacet	1.19	+364→194	+364→152					0.01
Flufenoxuron	Flufenoxuron	1.45	+489→158	+489→141					0.01
Flubendiamide	Flubendiamide	1.20	–681→272	–681→254					0.01
Flumioxazin	Flumioxazin	0.98	+372→355	+372→327	+355→327	+355→299	+355→79		0.01
Flumiclorac-pentyl	Flumiclorac-pentyl	1.42	+441→354	+441→308	+424→354	+424→308			0.01
Fluridone	Fluridone	1.08	+330→310	+330→259					0.01
Prochloraz	Prochloraz	1.34	+378→310	+378→70	+376→308	+376→266	+376→70		0.01*
Prosulfocarb	Prosulfocarb	1.45	+252→128	+252→91	+252→86				0.01
Prothiofos	Prothiofos	1.55	+347→243	+345→241	+345→161	+345→133			0.01
Propaquizafop	Propaquizafop	1.44	+444→371	+444→163	+444→100	+444→70			0.01
Propanil	Propanil	1.11	+218→162	+218→127	–216→160	–216→124			0.01
Propargite	Propargite	1.50	+368→231	+368→175					0.01
Propiconazole	Propiconazole	1.31	+342→159	+342→69					0.01*
Propyzamide	Propyzamide	1.16	+256→190	+256→173					0.01*
Profenofos	Profenofos	1.42	+375→347	+375→305	+373→303	+373→128			0.01*
Propoxur	Propoxur	0.71	+210→168	+210→111					0.01*
Bromacil	Bromacil	0.78	+261→205	+261→188					0.01
Prometryn	Prometryn	1.22	+242→200	+242→158					0.01
Bromobutide	Bromobutide	1.22	+312→194	+312→119					0.01
	N-(α,α-dimethylbenzyl)-3,3-dimethylbutyramide (deBr-bromobutide)	1.15	+234→119	+234→116	+234→91				0.01
Hexaconazole	Hexaconazole	1.33	+314→159	+314→70					0.01*
Hexazinone	Hexazinone	0.80	+253→171	+253→71					0.01
Hexaflumuron	Hexaflumuron	1.32	–459→439	–459→175					0.01*
Hexythiazox	Hexythiazox	1.43	+353→228	+353→168	+353→116				0.01
Benalaxyl	Benalaxyl	1.27	+326→294	+326→208	+326→148	+326→91			0.01*
Permethrin	Permethrin (Isomer 1)	1.59	+410→183	+408→355	+408→183				0.01*
	Permethrin (Isomer 2)	1.65							

Agricultural Chemicals	Analytes[1)]	RRT[2)]	Major monitoring ions (*m/z*) [3)]						Limit of quantification (mg/kg)[4)]
Penconazole	Penconazole	1.29	+284→159	+284→70					0.01*
Pencycuron	Pencycuron	1.36	+329→218	+329→125	+329→89				0.01
Bensulide	Bensulide	1.22	+398→356	+398→314	+398→158				0.01*
Benzofenap	Benzofenap	1.36	+433→105	+431→119	+431→105				0.01
Bendiocarb	Bendiocarb	0.82	+224→167	+224→109					0.01
Benthiavalicarb-isopropyl	Benthiavalicarb-isopropyl	1.12	+382→180	+382→116	+382→72				0.01
Penthiopyrad	Penthiopyrad	1.22	+360→276	+360→256	+360→177				0.01
Pentoxazone	Pentoxazone	1.35	+371→286	+371→186	+354→286	+354→186			0.01
Penflufen	Penflufen	1.31	+318→141	+318→234					0.01
Phoxim	Phoxim	1.34	+299→129	+299→77					0.01*
Phosalone	Phosalone	1.33	+368→182	+368→111					0.01
Boscalid	Boscalid	1.11	+345→307	+343→307	+343→140				0.01
Fosthiazate	Fosthiazate	0.92	+284→228	+284→104					0.01
Phosphamidon	Phosphamidon	0.71	+300→174	+300→127					0.01
Phorate	Phorate	1.34	+263→75	+261→199	+261→75				0.01*
Malathion	Malathion	1.21	+331→285	+331→127	+331→99				0.01*
Mandipropamid	Mandipropamid	1.12	+412→356	+412→328	+412→204	+412→125			0.01
Milbemectin	Milbemectin	1.49	+551→337	+551→240	+546→511	+546→493			0.01
Metaflumizone	Metaflumizone (*E*)	1.43	+507→178	+507→116	–505→302	–505→285	–505→117		0.01
Metaflumizone	Metaflumizone (*Z*)	1.41	+507→287	+507→178	–505→302	–505→116			0.01
Metaflumizone	Metaflumizone metabolite D	1.20	–288→273	–288→145	–288→142				0.01
Methabenzthiazuron	Methabenzthiazuron	0.96	+222→165	+222→150					0.01
Metalaxyl and Mefenoxam	Metalaxyl	0.92	+280→220	+280→192	+280→160				0.01*
	Mefenoxam	0.98	+281→192	+281→160	+280→220	+280→192			0.01*
Methiocarb	Methiocarb	1.12	+226→169	+226→121					0.01*
	Methiocarb sulfoxide	0.50	+242→185	+242→170	+242→122				0.01*
	Methiocarb sulfone	0.43	+258→201	+258→122	+258→107				0.01
Methidathion	Methidathion	1.04	+320→145	+320→85	+303→145	+303→85			0.01*
Methoxyfenozide	Methoxyfenozide	1.09	+369→149	+369→91					0.01
Metconazole	Metconazole (*cis*)	1.33	+320→125	+320→70					0.01
Metconazole	Metconazole (*trans*)	1.33	+320→125	+320→70					0.01
Metolachlor	Metolachlor (*RS*)	1.24	+284→252	+284→176					0.01
Mepanipyrim	Mepanipyrim	1.14	+224→106	+224→77					0.01
Mefenacet	Mefenacet	1.21	+299→148	+299→120					0.01
Mefenpyr-diethyl	Mefenpyr-diethyl	1.32	+373→327	+373→160	+373→133				0.01
Mepronil	Mepronil	1.18	+270→228	+270→119	+270→91				0.01
Monocrotophos	Monocrotophos	0.37	+224→193	+224→127	+224→98				0.01
Monolinuron	Monolinuron	0.90	+215→148	+215→126					0.01*
Lactofen	Lactofen	1.39	+479→344	+479→223					0.01
Linuron	Linuron	1.08	+251→162	+249→182	+249→160				0.01*
Lufenuron	Lufenuron	1.40	+511→158	+511→141	–509→339	–509→326	–509→175		0.01

1) The table lists the analytes to which this method can be applied in Japanese syllabary order. However, note that some agricultural chemicals under regulations may contain compounds such as metabolites that cannot be applied to this method. In addition, isomers with different retention times are indicated separately in the “Analytes” column. Moreover, the table is based on results from LC-MS/MS measurements.

2) The relative retention times (RRT) are relative to the isoxaflutole retention time (11–19 min) and are shown as the mean value of the laboratories studied.

3) The “Major monitoring ions” shows [precursor ion → product ion] in LC-MS/MS measurements, and the signs (+ or –) before the numbers indicate the ionization mode [ESI (+) or ESI (–)] in ESI measurements. Also, each ion is listed in descending order of numbers.

Table 2. Multi-residue Method I for Agricultural Chemicals by LC-MS (Agricultural Products): tea leaves and hops

Agricultural Chemicals	Analytes[1)]	RRT[2)]	Major monitoring ions (*m/z*)[3)]						Limit of quantification (mg/kg)[4)]
XMC	XMC	0.94	+180→123	+180→108	+180→107				0.01*
Acetamiprid	Acetamiprid	0.57	+223→126	+223→90	+223→56				0.01*
Azoxystrobin	Azoxystrobin	1.09	+404→372	+404→344	+404→329				0.01*
Atrazine	Atrazine	1.01	+216→174	+216→96					0.01*
Isoxathion	Isoxathion	1.28	+314→170	+314→105	+314→97				0.01*
Iprovalicarb	Iprovalicarb	1.15	+321→203	+321→119	+321→91				0.01
Imidacloprid	Imidacloprid	0.49	+256→209	+256→175					0.01*
Imibenconazole	Imibenconazole	1.33	+413→171	+413→125	+411→342	+411→171	+411→125		0.01*
Indoxacarb	Indoxacarb	1.28	+528→203	+528→150					0.01
Ethion	Ethion	1.36	+385→199	+385→143	+385→97				0.01*
Ethiprole	Ethiprole	1.07	+397→351	+397→255					0.01*
Etoxazole	Etoxazole	1.40	+360→304	+360→177	+360→141	+360→113			0.01*
Etofenprox	Etofenprox	1.52	+394→177	+394→135	+394→107				0.01*
Oxaziclomefone	Oxaziclomefone	1.32	+376→190	+376→161					0.01
Carfentrazone-ethyl	Carfentrazone-ethyl	1.19	+412→366	+412→346					0.01*
Carbofuran	Carbofuran	0.85	+222→165	+222→123					0.01*
Quizalofop	Quizalofop-ethyl	1.31	+373→299	+373→91					0.01
Quinalphos	Quinalphos	1.25	+299→163	+299→146	+299→97				0.01*
Cumyluron	Cumyluron	1.14	+303→185	+303→125					0.01
Kresoxim-methyl	Kresoxim-methyl	1.23	+314→206	+314→131	+314→116	+267→235	+267→207		0.01*
Cloquintocet-mexyl	Cloquintocet-mexyl	1.33	+336→238	+336→192	+336→179				0.01
Clodinafop-propargyl	Clodinafop-propargyl	1.18	+350→266	+350→91					0.01*
Clothianidin	Clothianidin	0.50	+250→169	+250→132					0.01*
Clofentezine	Clofentezine	1.31	+303→138	+303→102					0.01*
Clomazone	Clomazone	1.03	+240→125	+240→89					0.01*
Chromafenozide	Chromafenozide	1.17	+395→339	+395→175	+395→147				0.01*
Chlorpyrifos	Chlorpyrifos	1.38	+352→200	+350→198	+350→97				0.01*
Chlorpyrifos-methyl	Chlorpyrifos-methyl	1.28	+322→290	+322→125					0.01*
Chloroxuron	Chloroxuron	1.11	+291→218	+291→164	+291→72	+291→46			0.01*
Cyazofamid	Cyazofamid	1.18	+325→261	+325→108	+325→44				0.01
Dioxathion	Dioxathion	1.32	+474→271	+474→97					0.01*
Cycloprothrin	Cycloprothrin	1.40	+499→499	+499→257	+499→229	+499→181			0.5
Difenoconazole	Difenoconazole	1.27	+406→251	+406→111					0.01*
Difenzoquat	Difenzoquat	0.59	+249→130	+249→77					0.01*
Diflubenzuron	Diflubenzuron	1.19	+311→158	+311→141					0.01*
Simeconazole	Simeconazole	1.15	+294→135	+294→73	+294→70				0.01*
Dimethoate	Dimethoate	0.56	+230→199	+230→125					0.01*
Dimethomorph	Dimethomorph (*E*)	1.10	+388→301	+388→165					0.01
	Dimethomorph (*Z*)	1.12	+388→301	+388→165					0.01
Spinosad	Spinosyn A	1.52	+732→142	+732→98					0.01*
Spinosad	Spinosyn D	1.57	+747→142	+747→98					0.01*
Spiromesifen	Spiromesifen	1.38	+388→273	+388→255	+371→273	+371→255	+273→255	+273→187	0.01*
Diazinon	Diazinon	1.24	+305→169	+305→153	+305→97				0.01*
Daimuron	Daimuron	1.09	+269→151	+269→91					0.01
Thiacloprid	Thiacloprid	0.65	+253→126	+253→90					0.01*
Thiamethoxam	Thiamethoxam	0.39	+292→211	+292→181	+292→132				0.01*
Tetrachlorvinphos	Tetrachlorvinphos	1.20	+367→206	+367→127	+365→127				0.01
Tetraconazole	Tetraconazole	1.15	+372→159	+372→70					0.01*
Tebuconazole	Tebuconazole	1.21	+308→125	+308→70					0.01*
Tebuthiuron	Tebuthiuron	0.87	+229→172	+229→116					0.01*
Teflubenzuron	Teflubenzuron	1.35	+381→158	+381→141	–379→339	–379→196			0.01*
Triadimenol	Triadimenol	1.13	+296→99	+296→70	+296→43				0.01*
Triadimefon	Triadimefon	1.09	+294→197	+294→69					0.01*
Triflumizole	Triflumizole	1.30	+346→278	+346→73	+346→42				0.01*
Trifloxystrobin	Trifloxystrobin	1.30	+409→186	+409→206	+409→145				0.01*
Tolfenpyrad	Tolfenpyrad	1.35	+384→197	+384→145	+384→117	+384→91			0.01*
Parathion	Parathion	1.19	+292→264	+292→236	+292→140				0.01*
Bitertanol	Bitertanol	1.23	+338→148	+338→99	+338→70				0.01*
Pyrazophos	Pyrazophos	1.27	+374→238	+374→222	+374→194				0.01*
Pyraflufen-ethyl	Pyraflufen-ethyl	1.21	+415→341	+413→339	+413→261	+413→253			0.01*

Agricultural Chemicals	Analytes[1)]	RRT[2)]	Major monitoring ions (*m/z*) [3)]						Limit of quantification (mg/kg)[4)]
Pyridaben	Pyridaben	1.45	+365→309	+365→147					0.01*
Pyriftalid	Pyriftalid	1.09	+319→179	+319→139	+319→83	+319→82			0.01
Pyriproxyfen	Pyriproxyfen	1.39	+322→227	+322→185	+322→96	+322→77			0.01*
Pirimicarb	Pirimicarb	0.97	+239→182	+239→72					0.01
Pyrimidifen	Pyrimidifen	1.38	+378→184	+378→150					0.01*
Pirimiphos-methyl	Pirimiphos-methyl	1.29	+306→164	+306→108					0.01*
Fenamiphos	Fenamiphos	1.16	+304→234	+304→217	+304→202				0.01*
Fenoxaprop-ethyl	Fenoxaprop-ethyl	1.30	+362→288	+362→119	+362→91	+362→77			0.01
Fenobucarb	Fenobucarb	1.02	+208→152	+208→95					0.01*
Ferimzone	Ferimzone (*Z*)	1.10	+255→132	+255→91					0.01
Fenamidone	Fenamidone	1.05	+312→236	+312→92					0.01
Phenthoate	Phenthoate	1.20	+321→247	+321→163	+321→135	+321→79			0.01*
Fenpyroximate	Fenpyroximate (*E*)	1.43	+422→366	+422→138	+422→135				0.01*
Fenpyroximate	Fenpyroximate (*Z*)	1.37	+422→366	+422→138	+422→135				0.01*
Fenbuconazole	Fenbuconazole	1.17	+337→125	+337→70					0.01*
Fenpropathrin	Fenpropathrin	1.40	+367→125	+350→125	+350→97				0.01*
Fenpropimorph	Fenpropimorph	1.50	+304→147	+304→130	+304→117	+304→98			0.01*
Phenmedipham	Phenmedipham	1.04	+318→168	+318→136	+301→168	+301→136	+168→136	+168→93	0.01
Butafenacil	Butafenacil	1.11	+492→349	+492→331	+492→180				0.01
Buprofezin	Buprofezin	1.34	+306→201	+306→116	+306→57				0.01*
Fluometuron	Fluometuron	0.90	+233→160	+233→72	+233→46				0.01*
Flufenacet	Flufenacet	1.16	+364→194	+364→152					0.01
Flufenoxuron	Flufenoxuron	1.37	+489→158	+489→141					0.01*
Flubendiamide	Flubendiamide	1.19	–681→254	–681→274					0.01*
Fluridone	Fluridone	1.04	+330→310	+330→309	+330→259				0.01
Prochloraz	Prochloraz	1.23	+378→310	+376→308	+376→70				0.01*
Prothiofos	Prothiofos	1.46	+347→243	+345→269	+345→241	+345→133			0.01*
Propaquizafop	Propaquizafop	1.34	+444→371	+444→163	+444→100	+444→56			0.01
Propargite	Propargite	1.38	+368→231	+368→175	+231→175	+231→57			0.01*
Propiconazole	Propiconazole	1.23	+342→159	+342→69					0.01*
Propyzamide	Propyzamide	1.09	+256→190	+256→173					0.01*
Profenofos	Profenofos	1.31	+375→305	+375→96	+373→345	+373→303	+373→128		0.01*
Propoxur	Propoxur	0.80	+210→168	+210→111					0.01*
Hexaconazole	Hexaconazole	1.21	+316→70	+314→159	+314→70				0.01*
Hexythiazox	Hexythiazox	1.37	+353→228	+353→168					0.01*
Benalaxyl	Benalaxyl	1.21	+326→148	+326→91					0.01*
Pencycuron	Pencycuron	1.24	+329→218	+329→125	+329→89				0.01
Benzofenap	Benzofenap	1.31	+431→119	+431→105					0.01
Bendiocarb	Bendiocarb	0.81	+224→167	+224→109					0.01
Phosalone	Phosalone	1.26	+368→322	+368→182	+368→111				0.01*
Boscalid	Boscalid	1.10	+343→307	+343→271	+343→139				0.01
Phosphamidon	Phosphamidon	0.73	+300→174	+300→127					0.01*
Malathion	Malathion	1.13	+331→127	+331→99					0.01*
Myclobutanil	Myclobutanil	1.12	+289→125	+289→70					0.01*
Methiocarb	Methiocarb	1.09	+226→169	+226→121					0.01
Methidathion	Methidathion	1.02	+303→145	+303→85	+303→84				0.01*
Methoxyfenozide	Methoxyfenozide	1.14	+369→313	+369→149					0.01*
Monolinuron	Monolinuron	0.95	+215→148	+215→126	+215→99				0.01*
Lactofen	Lactofen	1.32	+479→344	+479→223	+462→344	+462→223			0.01
Linuron	Linuron	1.06	+249→182	+249→160	+249→133				0.01*
Lufenuron	Lufenuron	1.35	+511→158	+511→141	–509→326	–509→175			0.01*

1) The table lists the analytes to which this method can be applied in Japanese syllabary order. However, note that some agricultural chemicals under regulations may contain compounds such as metabolites that cannot be applied to this method. In addition, isomers with different retention times are indicated separately in the "Analytes" column. Moreover, the table is based on results from LC-MS/MS measurements.

2) The relative retention times (RRT) are relative to the isoxaflutole retention time (11–19 min) and are shown as the mean value of the laboratories studied.

3) The "Major monitoring ions" shows [precursor ion → product ion] in LC-MS/MS measurements, and the signs (+ or –) before the numbers indicate the ionization mode [ESI (+) or ESI (–)] in ESI measurements. Also, each ion is listed in descending order of numbers.

4) Limit of quantification was regarded as 0.01 ppm (or the lowest spiked concentration) when the S/N of the analyte peak obtained from a recovery test at a spiked level of 0.01 ppm (or the lowest spiked concentration) was more than 10 in at least one sample. In cases where there were no results of a spike recovery test at a spiked concentration of 0.01 ppm and the S/N of the analyte peak equivalent to 0.01 ppm in the sample using matrix-matched standard solutions was more than 10 in at least one food sample, the estimated limit of quantification was regarded at 0.01 mg/kg and indicated with an asterisk (*). For cycloprothrin, limit of quantification was regarded as 0.5 mg/kg, a spiked concentration, since the S/N ratio for the matrix standard solutions equivalent to 0.01 ppm in the sample was less than 10.