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)

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 column tube of 12-13 mm in inner diameter packed with 500 mg of graphitized carbon black in the upper layer and 500 mg of ethylenediamine-*N*-propylsilanized silica gel in the lower layer, or a cartridge equivalent to the specified one in separation capability.

0.5 mol/L Phosphate buffer (pH 7.0): Weigh 52.7 g of dipotassium hydrogenphosphate (K_2HPO_4) and 30.2 g of potassium dihydrogenphosphate (KH_2PO_4), dissolve in about 500 mL of water, adjust pH to 7.0 with 1 mol/L sodium hydroxide or 1 mol/L hydrochloric acid, and add water to make a 1 L solution.

Reference standards of agricultural chemicals: Reference standards of known purities for each agricultural chemical. (When individual testing methods for each agricultural chemical designate purities of reference standards, follow the direction. If not, desirably use reference standards with a purity of not less than 95%.)

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 minutes. 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 a 20 mL aliquot of the extract

accurately, add 10 g of sodium chloride and 20 mL of 0.5 mol/L phosphate buffer (pH 7.0), and shake for 10 minutes. 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 to the cartridge and elute with 5 mL of acetonitrile. Collect the total eluates, concentrate the filtrate 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

Weigh 20.0 g of sample, 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 a 20 mL aliquot of the extract accurately, add 10 g of sodium chloride and 20 mL of 0.5 mol/L phosphate buffer (pH 7.0), and shake for 10 minutes. 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 minutes. 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 a 5 mL aliquot of the extract accurately, add 15 mL of acetonitrile. Add 10 g of sodium chloride and 20 mL of 0.5 mol/L phosphate buffer (pH 7.0), and shake for 10 minutes. 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 to the cartridge and elute with 5 mL of acetonitrile. Collect the total eluates, concentrate the filtrate 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

Add 10 mL of acetonitrile and toluene (3:1, v/v) to 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, elute with 20 mL of acetonitrile and toluene (3:1, v/v), collect the total eluate, and concentrate at below 40°C to 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) 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, elute with 20 mL of acetonitrile and toluene (3:1, v/v), collect the total eluate, and concentrate at below 40°C to 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 standard solutions by dissolving reference standard of each agricultural chemical in appropriate solvents, and prepare several diluted solutions at appropriate concentration range using methanol. 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 solution into LC-MS or LC-MS/MS, and calculate the concentrations of each agricultural chemical from the calibration curves made in **6**.

8. Confirmation

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

9. Measurement conditions

(Example)

Column: Octadecylsilanized silica gel, 2-2.1 mm in 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 of each agricultural chemical from sample with acetonitrile, dehydrating by salting out, purifying with an octadecylsilanized silica gel cartridge and a graphitized carbon black/aminopropylsilanized silica gel layered cartridge, quantifying and confirming using LC-MS or LC-MS/MS.

ii) Fruits and vegetables

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

iii) Tea leaves and hops

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

2) Notes

i) The tables in the appendix show agricultural chemicals to which the method can be applied and are listed in the order of the Japanese syllabary. Note that the analytes may include not only the parent compounds, but also their metabolites or other transformation products which may be inapplicable to this method. The isomers are listed by their retention times in the column of “Analytes”.

ii) This method does not ensure all simultaneous analysis using analytes listed in Tables 1 and 2. In advance, confirm that the interaction by the intended combination of analytes does not cause decomposition and interfere with measurement.

iii) Sodium phosphate can be used for the preparation of a phosphate buffer.

iv) If the quantity of sodium chloride (10 g) is too large to add to the acetonitrile extract, it may be reduced so long as saturation is achieved.

v) If an emulsion was formed in a salting-out process, centrifuge at 3,000 rpm for 5 minutes.

vi) Concentration and complete removal of the solvent should be performed under a gentle stream of nitrogen.

vii) Water may remain in the eluate after the clean-up with an octadecylsilanized silica gel cartridge. Water may also remain after the concentration of the salted-out acetonitrile layer.

In such a case, add about 5 mL of acetonitrile and concentrate at below 40°C.

viii) The sample may not be sufficiently purified with the above method for fruits and vegetables. In such a case, the octadecylsilanized silica gel cartridge can be used as in the case with grains, legumes, nuts, seeds, and teas.

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

x) Depending on the sensitivity of the LC-MS or LC-MS/MS, it may be necessary to dilute the test solution with methanol.

xi) Because some agricultural chemicals are particularly unstable in methanol, LC-MS/MS analysis should be performed immediately after preparation of a test solution. The standard solutions used to determine the calibration curves should be prepared just prior to use.

xii) Matrix-matched calibration or standard addition may be required to obtain accurate measurement results.

xiii) Because the limit of quantification differs depending on the instrument used, the concentration rate of the test solution, and the injection volume, it may be necessary to optimize the conditions.

xiv) The cartridge may be washed with a higher concentration of methanol of a mobile phase after the target analyte was eluted so that sample matrices may be prevented from being carried over and affecting a measurement with LC-MS or LC-MS/MS.

xv) Thiodicarb may be degraded to methomyl during pre-processing in some kinds of crops.

xvi) When another method is presented to assess tea (except for powder tea), follow the direction.

xvii) Food items used to develop the analytical method: brown rice, soya bean, peanut, spinach, cabbage, potato, aubergine, orange, apple and tea (green tea, powder 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

C

Table 1. Multi-residue Method I for Agricultural Chemicals by LC-MS (Agricultural Products): grains, legmes, 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
Azafendin	Azafendin	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*

Agricultural Chemicals	Analytes[1)]	RRT[2)]	Major monitoring ions (*m/z*) [3)]						Limit of quantification (mg/kg)[4)]
Carfentrazone-ethyl	Carfentrazone-ethyl	1.21	+412->366	+412->346					0.01*
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*
Di-allate	Di-allate	1.39	+270->128	+270->109	+270->86				0.01*

Agricultural Chemicals	Analytes[1)]	RRT[2)]	Major monitoring ions (*m/z*) [3)]						Limit of quantification (mg/kg)[4)]
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
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*
Tribuphos	Tribuphos	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	-493->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 Buthoxide	Piperonyl Buthoxide	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*

Agricultural Chemicals	Analytes[1)]	RRT[2)]	Major monitoring ions (*m/z*) [3)]						Limit of quantification (mg/kg)[4)]
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*
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*

Agricultural Chemicals	Analytes[1)]	RRT[2)]	Major monitoring ions (*m/z*) [3)]						Limit of quantification (mg/kg)[4)]
Permethrin	Permethrin (Isomer 1)	1.59	+410->183	+408->355	+408->183				0.01*
	Permethrin (Isomer 2)	1.65							
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*
Mandipropanid	Mandipropanid	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 analytes are listed in the order of the Japanese syllabary, and the isomers are listed by their retention times. Note that the maximum residue limits (MRLs) defined for some agricultural chemicals include not only the parent compounds, but also their metabolites or other transformation products which may be inapplicable to this method. All values are determined by measurement using LC-MS/MS.

2) Relative retention time (RRT) is the relative value to the retention time of isoxaflutole. The RRT above shows the average values obtained from laboratories.

3) The figures in “Monitoring ions” show [precursor ion → product ion] from LC-MS/MS measurement, and the code (+ or -) before the figures represents the ionization mode used (ESI (+) or ESI (-)). Each ion is listed in order of descending m/z.

4) Limit of quantification was regarded as 0.01 mg/kg (or the lowest spiked level) when the S/N of analyte peak obtained from a recovery test at a spiked level of 0.01 ppm (or the lowest spiked level) was not less than 10 in at least one sample. For anaylte which was not performed a recovery test at a spiked level of 0.01 ppm, the limit of quantification was estimated to be 0.01 mg/kg when the S/N of the analyte peak of matric-matched standard corresponding to 0.01 ppm in the sample was not less than 10 in at least sample, and indicated this in the table with "*".

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->228	+374->222	+374->194				0.01*
Pyraflufen-ethyl	Pyraflufen-ethyl	1.21	+415->341	+413->339	+413->261	+413->253			0.01*
Pyridaben	Pyridaben	1.45	+365->309	+365->147					0.01*

Agricultural Chemicals	Analytes[1)]	RRT[2)]	Major monitoring ions (*m/z*) [3)]						Limit of quantification (mg/kg)[4)]
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	+433->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 analytes are listed in the order of the Japanese syllabary, and the isomers are listed by their retention times. Note that the maximum residue limits (MRLs) defined for some agricultural chemicals include not only the parent compounds, but also their metabolites or other transformation products which may be inapplicable to this method. All values are determined by measurement using LC-MS/MS.

2) Relative retention time (RRT) is the relative value to the retention time of isoxaflutole. The RRT above shows the average values obtained from laboratories.

3) The figures in “Monitoring ions” show [precursor ion → product ion] from LC-MS/MS measurement, and the code (+ or -) before the figures represents the ionization mode used (ESI (+) or ESI (-)). Each ion is listed in order of descending m/z.

4) Limit of quantification was regarded as 0.01 mg/kg (or the lowest spiked level) when the S/N of analyte peak obtained from a recovery test at a spiked level of 0.01 ppm (or the lowest spiked level) was not less than 10 in at least one sample. For anaylte which was not performed a recovery test at a spiked level of 0.01 ppm, the limit of quantification was estimated to be 0.01 mg/kg when the S/N of the analyte peak of matric-matched standard corresponding to 0.01 ppm in the sample was not less than 10 in at least sample, and indicated this in the table with "*". For cycloprothrin, the limit of quantifiation was regarded as 0.5 mg/kg since the S/N of the analyte peak of matric-matched standard corresponding to 0.01 ppm in the sample was less than 10.