Multi-residue Method I for Agricultural Chemicals by LC-MS (Animal and Fishery Products)

1. Analytes

For muscle, fat, lever, kidney, fish and shellfish, see Table 1.

For milk, egg and honey, see Table 2.

2. Instruments

Liquid chromatograph-mass spectrometer (LC-MS)

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

3. Reagents

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

Reference standards of each agricultural chemical: Agricultural chemical which clearly shows its purity.

4. Procedure

1) Extraction

i) Muscle, fat, lever, kidney, fish and shellfish

For muscle, lever, kidney, fish and shellfish, weigh 20.0 g of sample. For fat, weigh 5.00 g of sample.

Add 20 mL of water to the sample, homogenize, add 100 mL of acetone/*n*-hexane (1:2, v/v), homogenize again, centrifuge at 2,500 rpm for 5 minutes, and collect the organic layer.

Add 50 mL of *n*-hexane to the residue, homogenize, and centrifuge at 2,500 rpm for 5 minutes. Combine the obtained organic layers, dehydrate with anhydrous sodium sulfate, and filter out the anhydrous sodium sulfate. Concentrate the filtrate at below 40°C, and remove the solvent. Weigh the extracted fat weight. Take all or some amounts of the extracted fat, and dissolve in acetone/cyclohexane (1:4, v/v) so that the amount applying to the gel permeation chromatography column (styrene-divinylbenzene copolymer column) corresponds to 5.00 g of sample. (If the extracted fat weight in 5.00 g of sample is more than 0.5 g, the amount applying to the column should be corresponded to 0.50 g of extracted fat).

ii) Milk, egg and honey

For milk and egg, weigh 20.0 g of sample. For honey, weigh 20.0 g of sample and dissolve in 20 mL of water.

Add 100 mL of acetonitrile to the sample, homogenize, centrifuge at 2,500 rpm for 5 minutes, and take the organic layer. Add 50 mL of acetonitrile to the residue, homogenize, and centrifuge at 2,500 rpm for 5 minutes. Combine the obtained organic layers, add 10 g of sodium chloride, and shake. Let stand and discard the separated aqueous layer. Dehydrate the acetonitrile layer with anhydrous sodium sulfate, and filter out the anhydrous

sodium sulfate. Concentrate the filtrate at below 40°C and remove the solvent. For milk and egg, dissolve the residue in acetone/cyclohexane (1:4, v/v) so that the amount applying to the gel permeation chromatography column (styrene-divinylbenzene copolymer column) corresponds to 5.00 g of sample. For honey, dissolve the residue in acetone/*n*-hexane (1:1, v/v) to make exactly 10 mL.

2) Clean-up

i) Muscle, fat, fish, shellfish, milk and egg

a) Gel permeation chromatography

Centrifuge the solution obtained in **1)** at 3,000 rpm for 5 minutes, transfer 5 mL of the supernatant to a gel permeation chromatography column (styrene-divinylbenzene copolymer column), and elute with acetone/cyclohexane (1:4, v/v). Collect the solution eluted from retention time of acrinathrin to the finish time of tricyclazole elution, concentrate at below 40°C, and remove the solvent. Dissolve the residue in 2 mL of acetone/*n*-hexane (1:1, v/v).

b) Ethylenediamine-*N*-propylsilanized silica gel column chromatography

Add 10 mL of acetone/*n*-hexane (1:1, v/v) to an ethylenediamine-*N*-propylsilanized silica gel cartridge (500 mg), and discard the effluent. Transfer the solution obtained in **a)** to the cartridge, elute with 20 mL of acetone/*n*-hexane (1:1, v/v), collect the total eluate, concentrate at below 40°C, and remove the solvent. Dissolve the residue in methanol to make exactly 5 mL (2.5 mL for fat), and use this solution as the test solution.

ii) Lever and kidney

a) Gel permeation chromatography

Centrifuge the solution obtained in **1)** at 3,000 rpm for 5 minutes, transfer 5 mL of the supernatant to a gel permeation chromatography column (styrene-divinylbenzene copolymer column), and elute with acetone/cyclohexane (1:4, v/v). Collect the fraction eluted from the retention time of acrinathrin to the finish time of acrinathrin elution (Fraction I), and the fraction obtained from the finish time of Fraction I to the finish time of tricyclazole elution (Fraction II).

b) Ethylenediamine-*N*-propylsilanized silica gel column chromatography

Add 10 mL of acetone/cyclohexane (1:4, v/v) to an ethylenediamine-*N*-propylsilanized silica gel cartridge (500 mg), and discard the effluent. Transfer the Fraction I to the cartridge, elute with 5 mL of acetone/cyclohexane (1:4, v/v), collect the total eluate, concentrate at below 40°C, and remove the solvent. Dissolve the residue in 1 mL of *n*-hexane.

c) Silica gel column chromatography

Add 10 mL of *n*-hexane to a silica gel cartridge (690 mg), and discard the effluent. Transfer the solution obtained in b) to the cartridge, add 10 mL of *n*-hexane, and discard the effluent.

Elute with 15 mL of ether/*n*-hexane (1:19, v/v), combine the eluate with Fraction II

obtained in **a)**, concentrate at below 40°C, and remove the solvent. Dissolve the residue in methanol to make exactly 5 mL, and use this solution as the test solution.

iii) Honey

Add 10 mL of acetone/*n*-hexane (1:1, v/v) to an ethylenediamine-*N*-propylsilanized silica gel cartridge (500 mg), and discard the effluent. Transfer 2.5 mL of acetone/*n*-hexane (1:1, v/v) obtained in **1)** to the cartridge, elute with 20 mL of acetone/*n*-hexane (1:1, v/v), collect the total eluate, concentrate at below 40°C, and remove the solvent. Dissolve the residue in methanol to make exactly 5 mL, and use this solution as the test solution.

5. Calibration curve

Prepare standard solutions (acetonitrile) of each agricultural chemical. Mix them, prepare several solutions (methanol) with appropriate concentration range. Inject 5 µL of each standard solution to LC-MS or LC-MS/MS, and make calibration curves by peak-height or peak-area method.

6. Quantification

Inject 5 µL of the test solution to LC-MS or LC-MS/MS, and calculate the concentration of each agricultural chemical from the calibration curves made in **5**.

7. Confirmation

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

8. Measurement conditions

LC-MS(/MS)

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
30	5	95
30	85	15

Flow rate: 0.20 mL/min

Ionization mode: ESI

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

Expected retention time: See Tables 1 or 2.

9. Limit of quantification

See Tables 1 or 2.

Note that these tables show examples of measurement limitation (ng).

10. Explanatory note

1) Outline of analytical method

The method consists of extraction of each agricultural chemical from sample with acetone/*n*-hexane (1:2, v/v) (acetonitrile for milk, egg and honey), clean-up with gel permeation chromatography and ethylenediamine-*N*-propylsilanized silica gel column chromatography (also clean-up with silica gel column chromatography for liver and kidney, omit gel permeation chromatography for honey), and quantification and confirmation using LC-MS or LC-MS/MS.

2) Notes

i) Tables 1 and 2 show analytes which are applicable to this method in the order of the Japanese syllabary. Agricultural chemicals may include chemicals like metabolites which are inapplicable to this method. Isomers having different retention time are listed separately in “Analytes”.

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

iii) If the amount of sodium chloride (10 g) is too much for addition to the acetonitrile extract, it may be reduced as far as it is enough for saturation.

iv) Concentration and removal of the solvent should be performed gently in nitrogen stream.

v) An example of the condition for gel permeation chromatography is shown below.

Column: Styrene-divinylbenzene copolymer column (20 mm in inside diameter and 300 mm in length) connected with styrene-divinylbenzene copolymer column (20 mm in inside diameter and 100 mm in length) as a guard column, or other column with equal characteristics.

Mobile phase: Acetone/cyclohexane (1:4, v/v)

Current speed: 5 mL/min

Column temperature: 40°C

Injection volume: 5 mL

Measuring wavelength: 254 mm

Collection range: Determine in advance using the following method.

Prepare 5 mg/L of acrinathrin/tricyckazole in mobile phase, transfer 5 mL of the solution to the gel permeation chromatography column, monitor the retention times at 254 mm, and confirm the elution position. Other appropriate methods, for example, taking eluates with proper intervals and measuring with LC-MS can be used.

a. Collection range for muscle, fat, fish, shellfish, milk and egg (Figure 1.)

From retention time of acrinathrin to finish time of tricyclazole elution

(Example) 58 to 165 mL (volume: 107 mL)

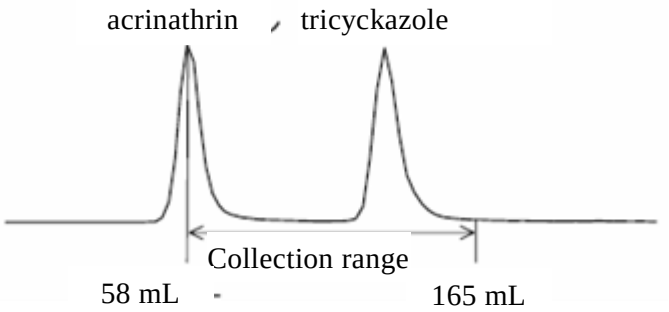

Figure 1. Collection range for muscle, fat, fish, shellfish, milk and egg

b. Collection range for liver and kidney (Figure 2.)

Fraction I: From retention time of acrinathrin to finish time of its elution

Fraction II: From finish time of Fraction I to finish time of tricyclazole elution

(Example) Fraction I: 58 to 65 mL (volume: 7 mL), Fraction II: 65 to 165 mL (volume: 100 mL)

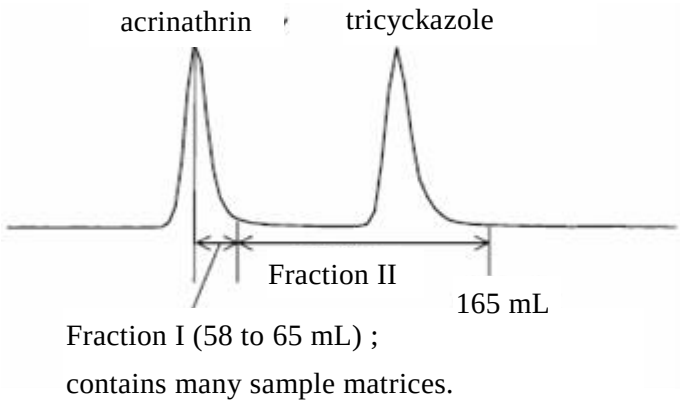

Figure 2. Collection range for liver and kidney

vi) Before measurement using cartridges, perform pretest on elution of each agricultural chemical and confirm the elution position under the use condition.

vii) For samples containing much fat, concentration rate of the test solution becomes low. If intended measurement sensitivity is not acquired, repeat the process after "**a)** gel permeation chromatography" using extracted fat, combine the test liquids, and use this solution as the test solution.

viii) Depends on the sensitivity of LC-MS or LC-MS/MS, dilute the test solution with methanol.

ix) Measurement should be performed immediately after preparation of the test solution. Some agricultural chemicals are unstable especially in methanol. Solutions for calibration curve should be prepared just before the use. Do not leave the test solution in auto sampler rack with normal temperature for a long time.

x) To gain accurate measured values, matrix-containing standard solution or standard addition

method may be required.

xi) Limit of quantification differs by the instrument to use, the concentration rate and the injection volume of the test solution. Consider optimum condition depending on the situation.

11. References

None

12. Type

C

Table 1. Multi-residue Method I for Agricultural Chemicals by LC-MS (Animal and Fishery Products)

Agricultural Chemicals	Analytes	relative retention time (RRT)	Monitoring ions for LC-MS (*m/z*)				Monitoring ions for LC-MS/MS （*m/z*）						Limit of measurement (ng), S/N = 10	
			positive mode		negative mode		positive mode							
			Quantitative	Qualitative	Quantitative	Qualitative	Precursor		Product (quantification)		Product (qualification)		LC-MS	LC-MS/MS
Ametryn	Ametryn	1.09					228		***186***		96		—	0.0003
Aramite	Aramite	1.36					357		***191***		167	105	—	0.0005
Etoxazole	Etoxazole	1.41					360		***141***		304	177	—	0.0001
Epoxiconazole	Epoxiconazole	1.19					331	330	***121***		123	101	—	0.0002
Oxabetrinil	Oxabetrinil	1.08					233		***147***		87	77	—	0.0020
Carfentrazone-ethyl	Carfentrazone-ethyl	1.23					412		***346***		366		—	0.0004
Quizalofop-ethyl	Quizalofop-ethyl	1.34					374		***300***	***299***	272	91	—	0.0005
	Quizalofop-p-tefuryl	1.32					429		***85***		299		—	0.0008
Clethodim	Clethodim (isomer 1)	0.93					360		***164***		268	166	—	0.0060
	Clethodim (isomer 2)	1.05					360		***164***		268	166	—	0.0021
Cloquintocet-mexyl	Cloquintocet-mexyl	1.37					337	336	***239***	***238***	193	192	—	0.0002
Clodinafop-propargyl	Clodinafop-propargyl	1.23					350		***266***		238	91	—	0.0002
Clofentezine	Clofentezine	1.29					303		***138***		102		—	0.0005
Chlorbufam	Chlorbufam	1.08					224		***172***		154		—	0.0006
Chlorfluazuron	Chlorfluazuron	1.43					540		***383***		158		—	0.0007
Diflubenzuron	Diflubenzuron	1.21					311		***158***		141		—	0.0005
Ciprodinil	Ciprodinil	1.26					226		***108***	***93***	118	93	—	0.0021
Sethoxydim	Sethoxydim (isomer 1)	0.89					328		***178***		220	180	—	0.0432
	Sethoxydim (isomer 2)	1.11					328		***178***		220	180	—	0.0004
Di-allate	Di-allate	1.32					270		***86***		143	109	—	0.0088
Tebufenozide	Tebufenozide	1.21					353		***297***	***133***	297	133	—	0.0008
Teflubenzuron	Teflubenzuron	1.31			***-379***		381		***158***		141		0.004	0.0029
Triflumizole	Triflumizole	1.28	***346***				346		***278***		73		0.006	0.0003
	Triflumizole metabolite	1.12	***295***										0.003	—
Triflumuron	Triflumuron	1.23	***359***				359		***156***		139		0.057	0.0003
Trifloxystrobin	Trifloxystrobin	1.27	***409***				409		***186***		145		0.001	0.0002
Novaluron	Novaluron	1.28			***-491***		493		***158***		141		0.007	0.0012
Barban	Barban	1.11	***258***				258		***178***		143		0.212	0.0110
Pyraclostrobin	Pyraclostrobin	1.23	***388***				388		***194***		163		0.008	0.0004
Pyrazophos	Pyrazophos	1.24	***374***				374		***222***		194		0.020	0.0019
Pyrimethanil	Pyrimethanil	1.08	***200***				200		***107***		182		0.007	0.0059
Fenamidone	Fenamidone	1.08	***312***				312		***92***		236		0.003	0.0003
Fenpyroximate	Fenpyroximate (*E*)	1.37	***422***				422		***366***		135		0.005	0.0002
	Fenpyroximate (*Z*)	1.32	***422***				422		***366***		138		0.007	0.0003
Fenpropimorph	Fenpropimorph	1.44	***304***				304		***147***		130		0.001	0.0007
Butafenacil	Butafenacil	1.14	***492***				492		***331***		180		0.004	0.0003
Butroxydim	Butroxydim (isomer 1)	1.01					400		***138***		354		—	0.1006
	Butroxydim (isomer 2)	1.08					400		***138***		354		—	0.0003
Flamprop-methyl	Flamprop-methyl	1.13	***336***				336		***105***		77		0.007	0.0003
Fluazuron	Fluazuron	1.32			***-504***	-305	506		***158***		141		0.003	0.0004
Flufenacet	Flufenacet	1.15	***364***				364		***194***		152		0.022	0.0002
Propaquizafop	Propaquizafop	1.31	***444***				444		***100***		70		0.027	0.0007
Prometryn	Prometryn	1.15	***242***				242		***158***		200		0.005	0.0002
Hexythiazox	Hexythiazox	1.33	***353***				353		***228***		168		0.068	0.0009
Benalaxyl	Benalaxyl	1.22	***326***				326		***148***		294		0.002	0.0001
Benfuracarb	Benfuracarb	1.29	***411***				411		***195***		190		0.006	0.0002
Phoxim	Phoxim	1.23	***299***				299		***129***		77		0.007	0.0016
Boscalid	Boscalid	1.09	***343***	342			343		***307***		271		0.028	0.0008
Methoxyfenozide	Methoxyfenozide	1.11	***369***	313			369		***149***		91		0.015	0.0007
Mefenpyr-diethyl	Mefenpyr-diethyl	1.23	***373***	375			373		***327***		160		0.026	0.0002
Linuron	Linuron	1.07			***-247***	-249	249		***182***		160		0.010	0.0010
Lufenuron	Lufenuron	1.31			***-509***	-511	511		***141***		158		0.004	0.0013

・The analytes are listed in the order of the Japanese syllabary, and the isomers are listed by their retention times.

・Relative retention time (RRT) is the relative value when Isoxaflutole (retention time: 15-18 min.) is 1. The RRT above shows the average of values which obtained from two laboratories.

・Bold italic figures in the "Monitoring Ions" mean quantitative ions and the other means qualitative ions.

・Figures in the "Limit of measurement" are the values at S/N = 10 when a standard solution is injected into LC/MS or LC-MS/MS. For LC/MS, figures were obtained from one laboratory. For LC-MS/MS, some figures were obtained from two laboratories. In that case, lower one was adopted.

・When 5 μL of a test solution prepared by the described method is injected into LC-MS(/MS), 0.05 ng of a sample (except fat)[*1] or 0.0125 ng of a sample (fat)[*2] corresponds to 0.01 ppm.

*1 When a test solution (final volume: 5 mL) was prepared using an amount corresponds to 5 g of sample.

*2 When a test solution (final volume: 2.5 mL) was prepared using an amount corresponds to 0.625 g of sample (an amount of a sample corresponds to 0.5 g of fat when the sample contains 80% of fat.

Table 2. Multi-residue Method I for Agricultural Chemicals by LC-MS (Animal and Fishery Products)

Agricultural Chemicals	Analytes	Relative retention time(RRT)	Monitoring ions for LC-MS (*m/z*)				Monitoring ions for LC-MS/MS （*m/z*）						Limit of measurement (ng), S/N = 10	
			positive mode		negative mode		positive mode							
			Quantitative	Qualitative	Quantitative	Qualitative	Precursor		Product (quantification)		Product (qualification)		LC-MS	LC-MS/MS
Azamethiphos	Azamethiphos	0.79					325		***183***		139	112	—	0.0005
Amitraz	Amitraz	1.45					294		***163***		253	122	—	0.0003
Ametryn	Ametryn	1.09					228		***186***		96		—	0.0003
Aramite	Aramite	1.36					357		***191***		167	105	—	0.0005
Aldicarb	Aldicarb	0.70					213	208	***116***	***89***	116	89	—	0.0128
Aldoxycarb	Aldoxycarb	0.37					223		***86***		148	76	—	0.0089
Etoxazole	Etoxazole	1.41					360		***141***		304	177	—	0.0001
Epoxiconazole	Epoxiconazole	1.19					331	330	***121***		123	101	—	0.0002
Oxabetrinil	Oxabetrinil	1.08					233		***147***		87	77	—	0.0020
Oxamyl	Oxamyl	0.39					237		***90***	***72***	90	72	—	0.0085
Oxydemeton-methyl	Oxydemeton-methyl	0.40					247		***169***		109	105	—	0.0009
Carbaryl	Carbaryl	0.89					202		***145***	***127***	146	117	—	0.0023
Carfentrazone-ethyl	Carfentrazone-ethyl	1.23					412		***346***		366		—	0.0004
Carbetamide	Carbetamide	0.75					237		***192***	***118***	120	118	—	0.0054
Carbendazim	Carbendazim	0.63					192		***160***		132		—	0.0011
Carbosulfan	Carbosulfan	1.50					382	381	***118***		160		—	0.0005
Carbofuran	Carbofuran	0.86	***222***				222		***165***		123		0.008	0.0005
	3-Hydroxy-carbofuran	0.54					238		***163***		181		—	0.0027
Quizalofop-ethyl	Quizalofop-ethyl	1.34					374		***300***	***299***	272	91	—	0.0005
	Quizalofop-p-tefuryl	1.32					429		***85***		299		—	0.0008
Clethodim	Clethodim (isomer 1)	0.93					360		***164***		268	166	—	0.0060
	Clethodim (isomer 2)	1.05					360		***164***		268	166	—	0.0021
Cloquintocet-mexyl	Cloquintocet-mexyl	1.37					337	336	***239***	***238***	193	192	—	0.0002
Clodinafop-propargyl	Clodinafop-propargyl	1.23					350		***266***		238	91	—	0.0002
Clothianidin	Clothianidin	0.49					250		***169***		132		—	0.0022
Clofentezine	Clofentezine	1.29					303		***138***		102		—	0.0005
Chloridazon	Chloridazon	0.56					222		***104***		92		—	0.0017
Chlorbufam	Chlorbufam	1.08					224		***172***		154		—	0.0006
Chlorfluazuron	Chlorfluazuron	1.43					540		***383***		158		—	0.0007
Diuron	Diuron	1.00					233		***72***		233		—	0.0091
Diflubenzuron	Diflubenzuron	1.21					311		***158***		141		—	0.0005
Cyprodinil	Cyprodinil	1.26					226		***108***	***93***	118	93	—	0.0021
Dimethomorph	Dimethomorph (*E*)	1.10					388		***301***		165		—	0.0005
	Dimethomorph (*Z*)	1.13					388		***301***		165		—	0.0002
Cymoxanil	Cymoxanil	0.60					199		***128***		111		—	0.0024
Sethoxydim	Sethoxydim (isomer 1)	0.89					328		***178***		220	180	—	0.0432
	Sethoxydim (isomer 2)	1.11					328		***178***		220	180	—	0.0004
Di-allate	Di-allate	1.32					270		***86***		143	109	—	0.0088
Thiacloprid	Thiacloprid	0.62					253		***126***		90		—	0.0011
Thiabendazole	Thiabendazole	0.74					202		***175***		131		—	0.0005
Thiamethoxam	Thiamethoxam	0.42					292		***211***		246	181	—	0.0017
Tebuthiuron	Tebuthiuron	0.86					229		***172***		116		—	0.0003
Tebufenozide	Tebufenozide	1.21					353		***297***	***133***	297	133	—	0.0008
Tepraloxydim	Tepraloxydim (isomer 1)	0.57					342		***250***		166		—	0.0449
	Tepraloxydim (isomer 2)	0.79	***342***				342		***250***		166		0.080	0.0037
Triflumizole	Triflumizole	1.28	***346***				346		***278***		73		0.006	0.0003
	Triflumizole metabolite	1.12	***295***										0.003	—
Triflumuron	Triflumuron	1.23	***359***				359		***156***		139		0.057	0.0003
Trifloxystrobin	Trifloxystrobin	1.27	***409***				409		***186***		145		0.001	0.0002
Triforine	Triforine (isomer 1)	1.03	***390***	392			435		***390***		97		0.061	0.0163
	Triforine (isomer 2)	1.05	***390***	392			435		***390***		97		—	0.0390
Novaluron	Novaluron	1.28			***-491***		493		***158***		141		0.007	0.0012
Barban	Barban	1.11	***258***				258		***178***		143		0.212	0.0110
Parbendazole	Parbendazole	1.16	***248***				248		***216***		173		0.025	0.0001
Pymetrozine	Pymetrozine	0.48	***218***				218		***105***		79		0.007	0.0004
Pyraclostrobin	Pyraclostrobin	1.23	***388***				388		***194***		163		0.008	0.0004
Pyrazophos	Pyrazophos	1.24	***374***				374		***222***		194		0.020	0.0019
Pyrimethanil	Pyrimethanil	1.08	***200***				200		***107***		182		0.007	0.0059

Agricultural Chemicals	Analytes	Relative retention time(RRT)	Monitoring ions for LC-MS (*m/z*)				Monitoring ions for LC-MS/MS （*m/z*）						Limit of measurement (ng), S/N = 10	
			positive mode		negative mode		positive mode							
			Quantitative	Qualitative	Quantitative	Qualitative	Precursor		Product (quantification)		Product (qualification)		LC-MS	LC-MS/MS
Fenamidone	Fenamidone	1.08	***312***				312		***92***		236		0.003	0.0003
Fenpyroximate	Fenpyroximate (*E*)	1.37	***422***				422		***366***		135		0.005	0.0002
	Fenpyroximate (*Z*)	1.32	***422***				422		***366***		138		0.007	0.0003
Fenpropimorph	Fenpropimorph	1.44	***304***				304		***147***		130		0.001	0.0007
Phenmedipham	Phenmedipham	1.04	***301***	318			318		***168***		136		0.029	0.0002
Butafenacil	Butafenacil	1.14	***492***				492		***331***		180		0.004	0.0003
Butroxydim	Butroxydim (isomer 1)	1.01					400		***138***		354		–	0.1006
	Butroxydim (isomer 2)	1.08					400		***138***		354		–	0.0003
Flamprop-methyl	Flamprop-methyl	1.13	***336***				336		***105***		77		0.007	0.0003
Fluridone	Fluridone	1.05	***330***				330		***310***		309		0.002	0.0001
Propaquizafop	Propaquizafop	1.31	***444***				444		***100***		70		0.027	0.0007
Bromacil	Bromacil	0.85	***263***	205			261		***205***		188		0.042	0.0017
Prometryn	Prometryn	1.15	***242***				242		***158***		200		0.005	0.0002
Hexythiazox	Hexythiazox	1.33	***353***				353		***228***		168		0.068	0.0009
Benalaxyl	Benalaxyl	1.22	***326***				326		***148***		294		0.002	0.0001
Boscalid	Boscalid	1.09	***343***	342			343		***307***		271		0.028	0.0008
Methoxyfenozide	Methoxyfenozide	1.11	***369***	313			369		***149***		91		0.015	0.0007
Mefenpyr-diethyl	Mefenpyr-diethyl	1.23	***373***	375			373		***327***		160		0.026	0.0002
Monolinuron	Monolinuron	0.92	***215***				215		***126***		148		0.008	0.0024
Linuron	Linuron	1.07			***-247***	-249	249		***182***		160		0.010	0.0010
Lufenuron	Lufenuron	1.31			***-509***	-511	511		***141***		158		0.004	0.0013

・The analytes are listed in the order of the Japanese syllabary, and the isomers are listed by their retention times.

・Relative retention time (RRT) is the relative value when Isoxaflutole (retention time: 15-18 min.) is 1. The RRT above shows the average of values which obtained from two laboratories.

・Bold italic figures in the "Monitoring Ions" means quantitative ions and the other means qualitative ions.

・Figures in the "Limit of measurement" are the value at S/N = 10 when a standard solution is injected into LC/MS or LC-MS/MS. For LC/MS, figures were obtained from one laboratory. For LC-MS/MS, some figures were obtained from two laboratories. In that case, lower one was adopted.

・When 5 μL of a test solution prepared by the described method is injected into LC-MS(/MS), 0.05 ng of a sample[*1] corresponds to 0.01 ppm.

*1 When a test solution (final volume: 5 mL) was prepared using an amount corresponds to 5 g of sample.