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Application News

No. C207A

Liquid Chromatograph Mass Spectrometry

Analysis of Residual Pesticides (No. 1: in Soybeans) Using Triple Quadrupole LC/MS/MS <LCMS™-8060>

With a recent increase in the number of regulated pesticides, more effective methods for simultaneous analysis of residual pesticides in food are required.

QuEChERS, which was introduced by the United States Department of Agriculture (USDA) in 2003, is known as a quick and simple pretreatment method and approved as an official method by AOAC and CEN. This method requires no special instruments for extraction of pesticides, but the contaminants that cannot be completely removed by means of purification procedures may affect accurate quantitative analysis. In such cases, sample dilution or review of the purification process is also required.

This article introduces an example of the analysis of 158 pesticides among those specified in the Multi-residue Method I and II for Agricultural Chemicals by LC-MS (Agricultural Products)[1)] by measuring these pesticides in the sample solutions pretreated using the QuEChERS method, resulting in good recovery.

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Sample Pretreatment

The soybean sample was pretreated using the QuEChERS method. The workflow of sample pretreatment is shown in Fig. 1. The concentration of samples extracted was 0.5 g/mL.

PL2005MIX-4, 5, 6, 7, 8, 9 and 10, mixtures of pesticides manufactured by Hayashi Pure Chemical Ind.,Ltd., were used as the standard samples. The matrix effect was identified using the matrix standard solution (10 ng/mL pesticide in the solution) made by adding each pesticide to the sample solution pretreated with the QuEChERS method to reach a concentration of 0.02 mg/kg in the soybean extract.

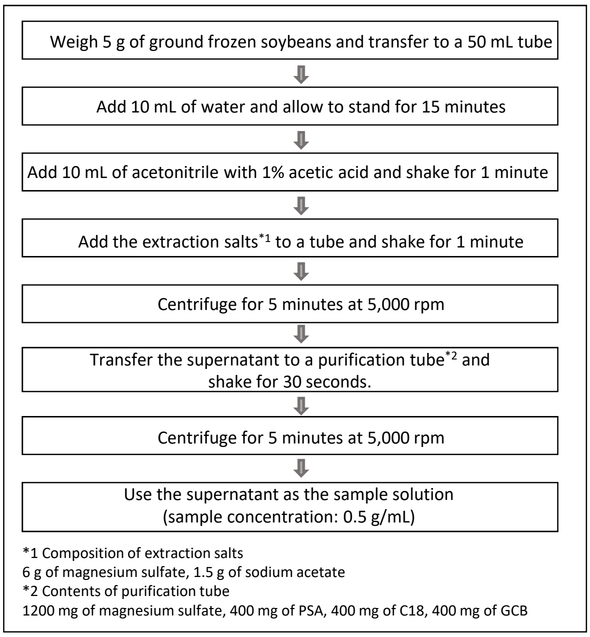

Fig. 1 Pretreatment Workflow

Analytical conditions

The analytical conditions for HPLC and MS are shown in Table 1.

Table 1. Analytical Conditions

[HPLC conditions] (Nexera™ X2)		
Column	:	Shim-pack Scepter™ C18-120 (100 mm x 2.1 mm I.D., 3 µm)
Mobile phases	:	A) 5 mM ammonium formate, 0.02% acetic acid in H_2O B) 5 mM ammonium formate, 0.02% acetic acid in MeOH
Gradient Program	:	B 5% (0-2 min) – B 50% (5 min) – B 97% (13-16 min) – B 5% (16.1-20 min)
Flow rate	:	0.3 mL/min
Column Temp.	:	40°C
Injection volume	:	1 µL
[MS conditions] (LCMS-8060)		
Ionization	:	ESI (Positive and negative mode)
Probe Voltage	:	+2.0 kV / -1.5 kV
Mode	:	MRM
Nebulizing gas flow	:	3.0 L/min
Drying gas flow	:	10.0 L/min
Heating gas flow	:	10.0 L/min
DL Temp.	:	200°C
Heat Block Temp.	:	300°C
Interface Temp.	:	200°C
Probe position	:	+2.0 mm

MRM Measurement of Matrix Standard Solution

Fig. 2 shows the MRM chromatogram of the matrix standard solution made by adding pesticide standard solution to the soybean extract.

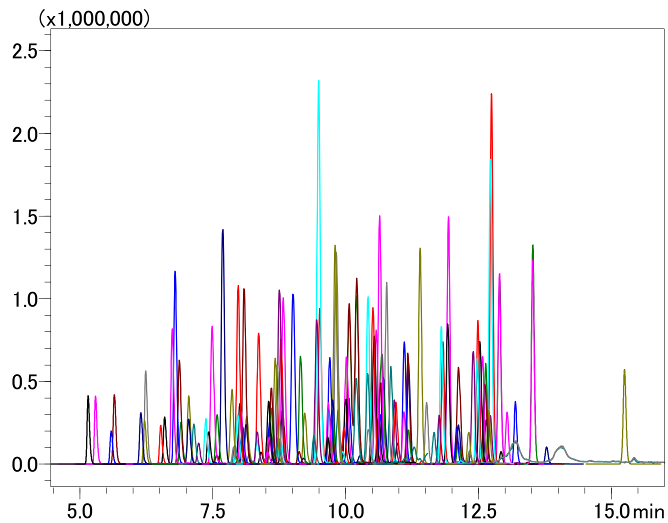

Fig. 2 Example of Peak Detections of 158 Pesticides (Soybean Extract Added to 10 ng/mL Standard Solution)

Recovery

The recovery and peak area repeatabilities (n=6) of the matrix standard solutions for 158 pesticides were determined. The results of determination are shown in Table 2. Details of the recovery are shown in Fig. 3.

The recovery for 156 of 158 pesticides were in the range of 70 to 120%. Even in the test solution containing a high concentration of sample, 98.7% of these pesticides were not significantly affected by the matrix, resulting in good recovery and repeatabilities.

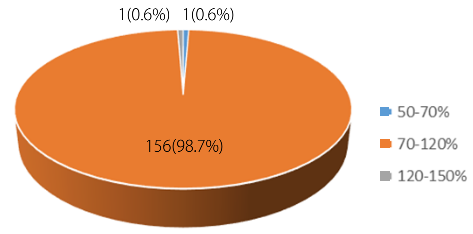

Fig. 3 Details of Recovery

Table 2. Recovery and Peak Area Repeatability of Sample Solutions

Compound name	Recovery(%)	%RSD	Compound name	Recovery(%)	%RSD	Compound name	Recovery(%)	%RSD
1-Naphthaleneacetic Acid	76.3	8.32	Dymuron	91.8	5.36	Methoxyfenozide	92.0	6.35
2,4-D	97.3	4.66	Epoxiconazole	90.0	1.87	Metosulam	102.7	7.44
4-Chlorophenoxyacetic acid	76.5	5.34	Ethametsulfuron-methyl	95.3	6.52	Metsulfuron-methyl	96.5	5.54
Abamectin B1a	93.5	1.37	Ethoxysulfuron	101.2	4.22	Monolinuron	96.0	2.52
Acibenzolar-S-methyl	90.0	8.84	Fenamidone	92.8	3.74	Naproanilide	91.3	5.53
Acifluorfen	88.7	7.50	Fenhexamid	92.0	6.06	Naptalam	95.9	9.97
Aldicarb	92.8	4.24	Fenobucarb	96.5	4.83	Novaluron	90.9	6.44
Aldoxycarb	96.5	1.15	Fenoxaprop-ethyl	88.0	2.68	Oryzalin	90.5	9.95
Anilofos	93.3	3.08	Fenoxycarb	92.9	3.94	Oxamyl	93.6	2.71
Aramite	95.8	3.88	Fenpyroximate E	93.4	3.11	Oxaziclomefone	89.5	4.16
Azamethiphos	93.5	4.68	Fenpyroximate Z	93.9	2.90	Oxycarboxin	96.3	3.21
Azimsulfuron	84.9	8.46	Ferimzone(E)	95.2	2.66	Pencycuron	95.8	3.89
Azinphos-methyl	95.2	3.91	Ferimzone(Z)	96.9	2.08	Penoxsulam	99.9	2.92
Azoxystrobin	93.6	6.70	Flazasulfuron	97.3	4.79	Pentoxazone	79.9	9.20
Bendiocarb	99.2	2.14	Florasulam	97.6	7.50	Phenmedipham	95.5	1.35
Bensulfuron-methyl	97.9	5.57	Fluazifop	94.1	6.73	Pirimicarb	94.6	5.96
Benzofenap	97.4	1.42	Flufenacet	95.3	4.66	Primisulfuron-methyl	95.0	3.81
Boscalid	98.0	2.53	Flufenoxuron	93.1	7.23	Propaquizafop	93.6	3.13
Bromoxynil	92.7	8.96	Flumetsulam	101.8	6.45	Propoxycarbazone	142.5	8.39
Butafenacil	99.0	2.63	Fluridone	93.6	2.22	Prosulfuron	99.6	6.03
Carbaryl(NAC)	98.5	5.77	Fluroxypyr	91.2	8.65	Pyraclostrobin	96.5	4.62
Carbofuran	93.5	5.35	Fomesafen	103.4	4.83	Pyrazolynate	93.7	2.96
Carpropamid	94.4	3.72	Foramsulfuron	115.1	8.65	Pyrazosulfuron-ethyl	96.8	8.99
Chloridazon	92.1	2.82	Forchlorfenuron	92.0	5.85	Pyriftalid	95.7	4.52
Chlorimuron-ethyl	101.8	7.76	Furametpyr	97.5	3.02	Quizalofop-ethyl	81.4	1.31
Chloroxuron	95.5	5.95	Furathiocarb	93.5	1.76	Silafluofen	84.9	6.25
Chlorsulfuron	96.9	5.36	Gibberellic acid	63.5	10.61	Simeconazole	95.1	2.63
Chromafenozide	95.1	1.65	Halosulfuron-methyl	80.2	7.45	SpinosynA	100.9	6.04
Cinosulfuron	98.4	9.43	Haloxyfop	82.7	7.70	SpinosynD	105.9	4.00
Clodinafop acid	91.9	8.00	Haloxyfop	85.0	8.78	Sulfentrazone	86.4	7.29
Clofentezine	84.7	4.06	Hexaflumuron	96.4	7.41	Sulfosulfuron	97.4	7.18
Clomeprop	87.6	3.77	Hexythiazox	93.8	3.31	Tebufenozide	95.3	5.07
Cloprop	97.8	9.36	Imazalil	106.8	3.52	Tebuthiuron	91.6	4.51
Cloquintocet-mexyl	97.8	3.64	Imazaquin	95.5	4.25	Teflubenzuron	87.9	7.57
Cloransulam-methyl	101.9	6.16	Imazosulfuron	94.0	5.67	Tetrachlorvinphos	94.2	3.80
Clothianidin	85.9	5.42	Imidacloprid	89.9	1.12	Thiabendazole	94.0	3.67
Cumyluron	98.5	2.16	Indanofan	94.3	3.07	Thiacloprid	94.3	1.85
Cyazofamid	95.7	1.39	Indoxacarb	99.9	4.16	Thiamethoxam	96.0	1.72
Cyclanilide	96.8	4.10	Iodosulfuron-methyl	93.0	7.59	Thidiazuron	82.8	7.17
Cycloate	94.9	3.31	Ioxynil	98.8	7.08	Thifensulfuron-methyl	96.6	6.43
Cycloprothrin	72.6	5.13	Iprovalicarb	95.6	3.46	Thiodicarb	95.8	2.97
Cyclosulfamuron	96.8	5.74	Isoxaflutole	92.8	6.43	Tralkoxydim 1	104.0	5.25
Cyflufenamid	91.9	1.72	Lactofen	90.5	2.10	Tralkoxydim 2	93.9	4.25
Cyprodinil	94.6	3.10	Linuron	95.4	3.54	Triasulfuron	96.8	3.77
Diallate	94.1	4.05	Lufenuron	93.2	4.48	Tribenuron-methyl	94.1	7.77
Dichlorprop	97.5	9.08	MCPA	96.1	4.16	Triclopyr	94.5	7.21
Diclomezine	100.7	8.89	MCPB	86.6	2.15	Tridemorph 1	97.5	4.28
Diclosulam	95.9	2.23	Mecoprop+Mecoprop-P	85.2	2.79	Tridemorph 2	96.3	2.18
Diflubenzuron	87.4	3.15	Mepanipyrim	94.5	3.97	Trifloxysulfuron	96.3	7.75
Dimethirimol	94.7	3.20	Mesosulfuron-methyl	95.1	3.50	Triflumuron	92.9	3.70
Dimethomorph(E)	98.1	2.86	Methabenzthiazuron	96.6	2.11	Triflusulfuron-methyl	99.5	5.49
Dimethomorph(Z)	98.1	2.86	Methiocarb	95.0	4.16	Triticonazole	94.2	2.68
Diuron	96.6	2.34	Methomyl	97.8	1.44			

1) Ministry of Health, Labour and Welfare: Testing Method of Agricultural Chemical Residues in Food, Feed Additives or Components of Animal Pharmaceuticals (PFSB/DFS Notification No. 1129002)P

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