

## Analysis of Residual Pesticides in Farm Products using LC-MS

Residual Pesticides in farm products are strictly regulated by the Food Sanitation Law. At present standards have been set for 217 types of pesticides for approximately 130 types of farm products. Of these, pesticides that are subject to thermal decomposition or highly volatile are analyzed using LC. However, the high percentage of interfering substances included in extracts from food products require highly selective detectors. This is why LC-MS has been attracting attention in this field.

Introduced here is an example of an LC-MS simultaneous analysis of 20 components of residual

pesticides on a farm product. Fig.1 shows the mass chromatogram in the scan mode. Highly sensitive analysis is possible with the conditions set during auto-tuning, eliminating the need of conditional optimization for each pesticide. In addition, the multi-sequence mode enables accurate qualification and quantitation of each component by performing mass chromatography at the proper mass number for each pesticide, whether it includes positive or negative ions.

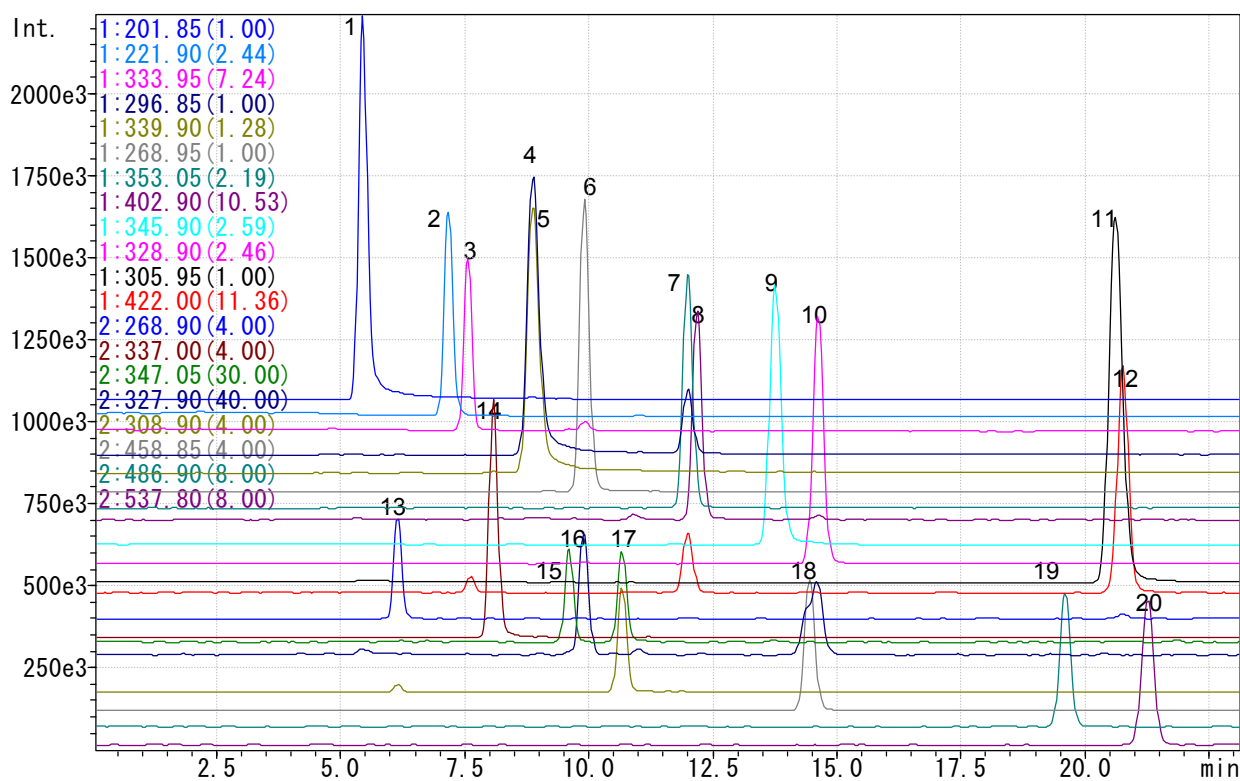


Fig. 1 Mass chromatograms of pesticides in foods

### ESI-Positive mode

1. thiabendazole	MW 201
2. methabenzthiazuron	MW 221
3. furametpyr	MW 333
4. imazalil	MW 296
5. etobenzanid	MW 339
6. dymron	MW 268
7. tebufenozide	MW 352
8. pyrazoxyfen	MW 402
9. triflumizole	MW 345
10. pencycuron	MW 328
11. buprofezin	MW 305
12. fenpyroximate	MW 421

### ESI-Negative mode

13. imibenconazole-debenzyl	MW 270
14. inabenfide	MW 338
15. myclobutanil	MW 288
16. iprodione metabolite	MW 329
17. diflubenzuron	MW 310
18. hexaflumuron	MW 460
19. flufenoxuron	MW 488
20. chlorfluazuron	MW 539

The SIM chromatograms and calibration curves (n=5) of pencycuron and hexaflumuron are shown in Figs. 2 and 3 respectively. Tables 1 and 2 show the reproducibility for these two components. As shown in this example,

components with similar of the same retention times, and different measurement modes (positive and negative ions) can be quantified at the same time.

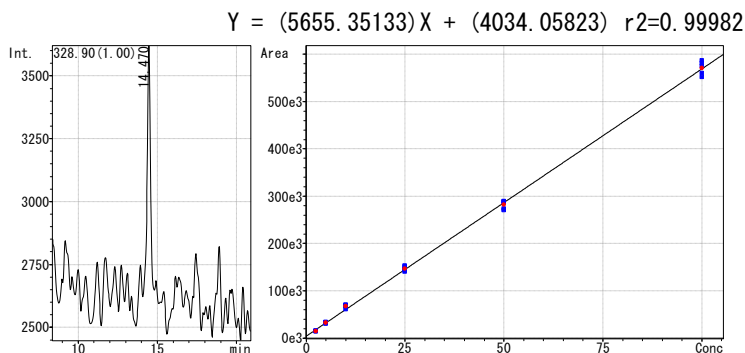
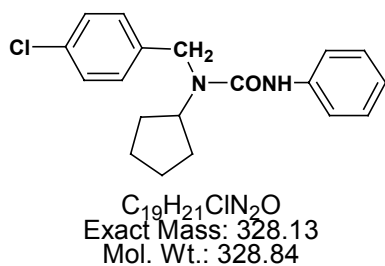


Fig. 2 SIM chromatogram (12.5pg) and calibration curve (12.5-500pg) of pencycuron

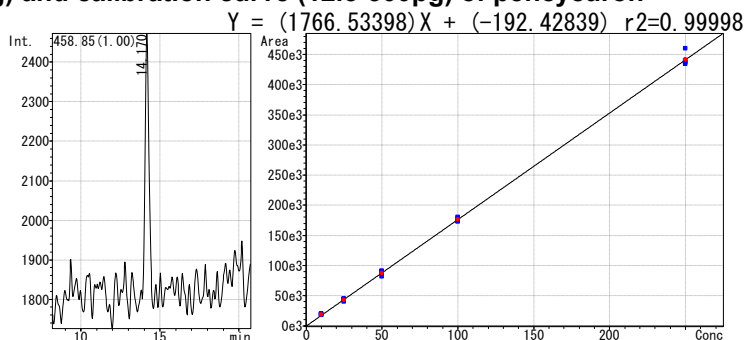
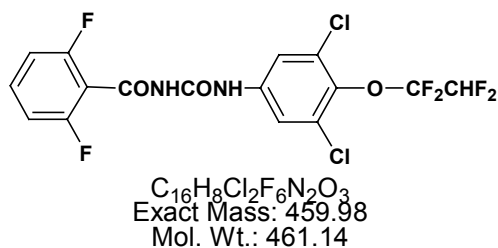


Fig. 3 SIM chromatogram (25pg) and calibration curve (25-1250pg) of hexaflumuron

Table 1 Reproducibility of pencycuron

	1	2	3	4	5	Average	SD	CV
12.5pg	13917	14526	14018	13948	15251	14332.00	570.19163	3.98 %
25pg	33710	33710	30242	30793	31331	31957.20	1645.7532	5.15 %
50pg	67238	68996	69932	66772	61325	66852.60	3346.1268	5.01 %
125pg	150565	145253	144468	152698	140439	146684.60	4929.2189	3.36 %
250pg	289289	287762	270265	288482	273127	281785.00	9281.106	3.29 %
500pg	586968	581783	560675	575145	551669	571248.00	14734.456	2.58 %

Table 2 Reproducibility of hexaflumuron

	1	2	3	4	5	Average	SD	CV
25pg	11153	8984	9859	9766	9007	9753.80	883.07401	9.05 %
50pg	18690	19229	20473	18580	17762	18946.80	1001.644	5.29 %
125pg	42881	43726	40001	44840	43646	43018.80	1825.9509	4.24 %
250pg	81842	89280	90536	86530	91154	87868.40	3808.3029	4.33 %
500pg	174911	177600	180001	174125	172789	175885.20	2894.5713	1.65 %
1250pg	437000	437627	439882	434111	460184	441760.80	10502.585	2.38 %

\* This data has been acquired with the cooperation of Dr. Kimihiko Yoshii and Dr. Yasuhide Tonogai from the Osaka branch of the National Institute of Health Sciences. We hereby express our appreciation for their cooperation.

Table 3 Analytical conditions for LC-MS

Column	: Shimadzu Shim-pack VP-ODS (2.0 mmI.D. x 150 mm)	
Mobile phase A	: water	Mobile phase B : acetonitrile
Gradient program	: 20%B - 60%B(0.03min) - 80%B(20min) - 100%B(20.01-30min) - 20%B(30.01-40min)	
Flow rate	: 0.2 mL/min	
Injection volume	: 5 uL	Column temperature : 40 degree C
Probe voltage	: +4.5 kV (ESI-Positive mode), -3.0 kV (ESI-Negative mode)	
CDL temperature	: 200 degree C	Block Heater temperature : 200 degree C
Nebulizing gas flow	: 4.5 L/min	
CDL voltage	: +0 V (ESI-Positive mode), +0 V (ESI-Negative mode)	
Q-array DC voltage	: scan mode	Q-array RF : scan mode
Scan range	: m/z 50 - 650 (1.5 sec/scan)	

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