

# Application News

Liquid Chromatography Mass Spectrometry

No.C80

## Analysis of Golf Course Pesticides Using Triple Quadrupole LC/MS/MS (LCMS-8030)

Many kinds of pesticides are used to maintain golf course turf, and because golf courses are often located in mountain corridors and near water sources, pollution of nearby fresh water sources and tap water from golf course waste water is of concern. With this increased concern over the environment, in 1990 Japan's Ministry of the Environment issued the "Provisional Guidance Indicators for the Prevention of Water Pollution from Pesticides Used in Golf Courses" (hereafter, Guidance Indicators), in which indicator values were specified for 21 pesticides. Then, a

The MRM chromatograms of 21 golf course pesticides are shown in Fig. 1, and calibration curves for typical golf course pesticides are shown in Fig. 2. Excellent linearity was obtained for all substances within the concentration range of 0.5 to 100 µg/L. Table 1 shows the parameters used in the MRM mode, in addition to

notification issued on September 29, 2010 increased the list to 72 pesticides and made it a requirement to conduct simultaneous analysis of the chemicals using higher sensitivity instruments.

Of those 72 pesticides, we conducted simultaneous analysis of 21 of the remaining 44 substances specified to be evaluated by LC/MS using the LCMS-8030, and in addition, we conducted monitor testing of golf course wastewater. The results are presented here.

the quantitation results. Six consecutive measurements were conducted for each compound at a concentration of 0.5 µg/L, and the area repeatability %RSD was less than 10 % in all cases. The quantitation limit (calculated with S/N = 10) ranged from 0.06 to 0.50 µg/L.

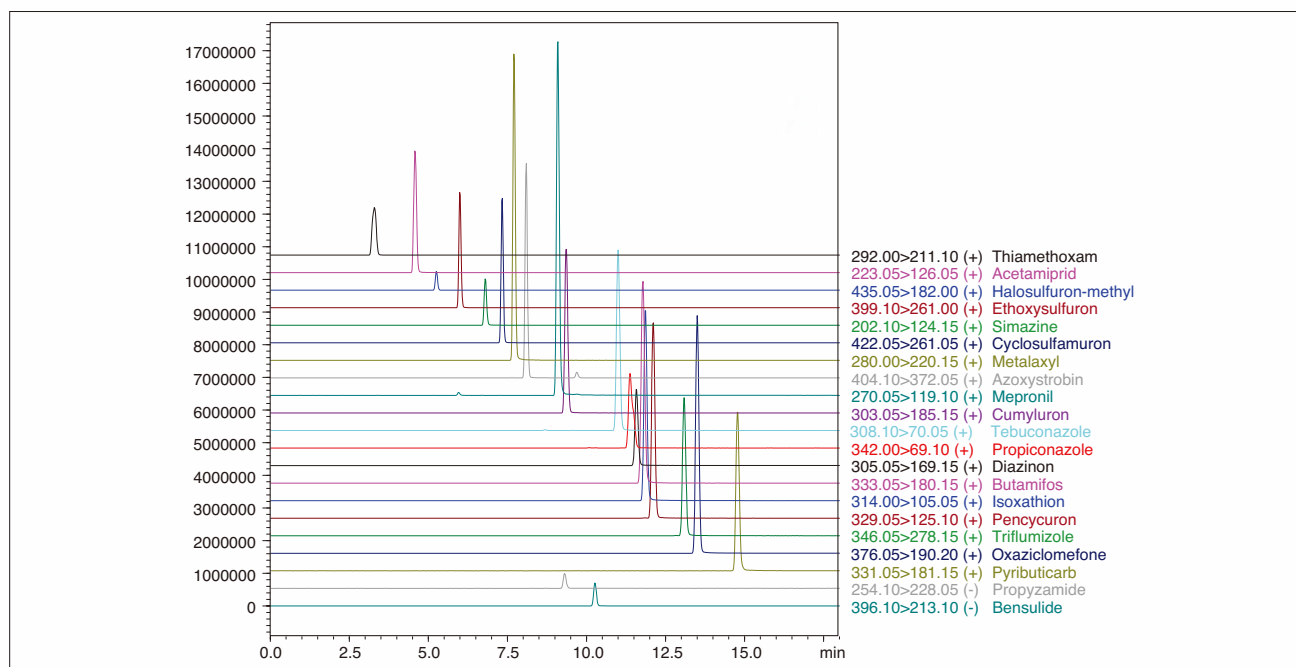


Fig. 1 Chromatograms of Pesticides Used on Golf Courses

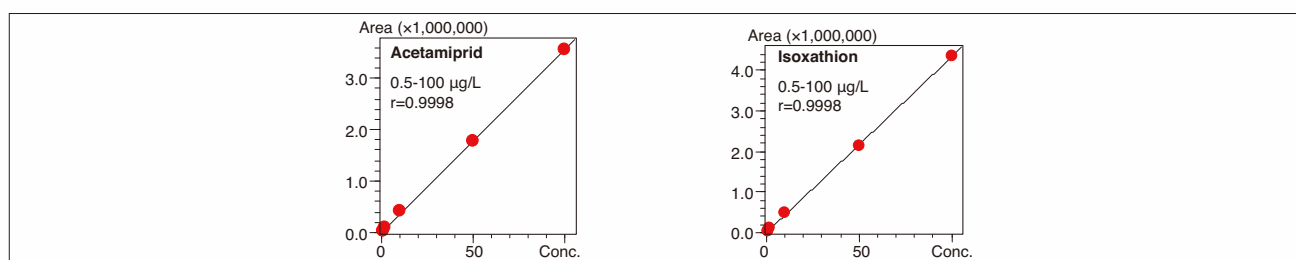


Fig. 2 Calibration Curves of Typical Golf Course Pesticides

Table 1 MRM Mode Parameters and Analysis Results for Each Pesticides

Compound	Polarity	Transition	CE (V)	%RSD (%)	S/N	LOQ (µg/L)	Compound	Polarity	Transition	CE (V)	%RSD (%)	S/N	LOQ (µg/L)
Thiamethoxam	+	292.00>211.10	-10	4.3	18	0.28	Bensulide	-	396.10>213.10	+10	6.7	13	0.38
Acetamiprid	+	223.05>126.05	-20	4.5	36	0.14	Tebuconazole	+	308.10>70.05	-25	4.2	18	0.28
Halosulfuron-methyl	+	435.05>182.10	-20	6.6	29	0.17	Propiconazole	+	342.00>69.10	-25	4.8	10	0.50
Ethoxysulfuron	+	399.10>261.00	-16	6.1	22	0.23	Diazinon	+	305.05>169.15	-20	6.9	12	0.42
Simazine	+	202.10>124.15	-20	6.3	11	0.45	Butamifos	+	333.05>180.15	-10	4.1	27	0.19
Cyclosulfamron	+	422.05>261.05	-20	2.9	83	0.06	Isoxathion	+	314.00>105.05	-15	3.9	18	0.28
Metalaxyl	+	280.00>220.15	-15	2.0	40	0.13	Pencycuron	+	329.05>125.10	-25	5.9	25	0.20
Azoxystrobin	+	404.10>372.05	-15	2.4	26	0.19	Triflumizole	+	346.05>278.15	-10	4.6	15	0.33
Mepronil	+	270.05>119.10	-25	5.9	13	0.38	Oxaziclomefone	+	376.05>190.20	-15	3.3	25	0.20
Propyzamide	-	254.10>228.05	+15	5.5	13	0.38	Pyributicarb	+	331.05>181.15	-15	1.9	16	0.31
Cumyluron	+	303.05>185.15	-15	4.3	12	0.42							

\* The %RSD, S/N and LOQ values are shown as the average of n = 6 measurements at 0.5 µg/L, respectively.

## ■ Analysis of Environmental Water

We conducted measurement of golf course wastewater. The collected sample was filtered through a 0.45 µm filter, and then analyzed.

Fig. 3 shows the MRM chromatograms of the sample solution (without added standard) and a sample solution spiked with 10 µg/L or 50 µg/L of pesticide standards (Simazine, Diazinon, Isoxathion and

Triflumizole, as examples). Pesticides were not detected in the wastewater used here. In the sample solution with the added pesticide standards, all of the substances were detected with excellent S/N ratios, without any interference from contaminating substances.

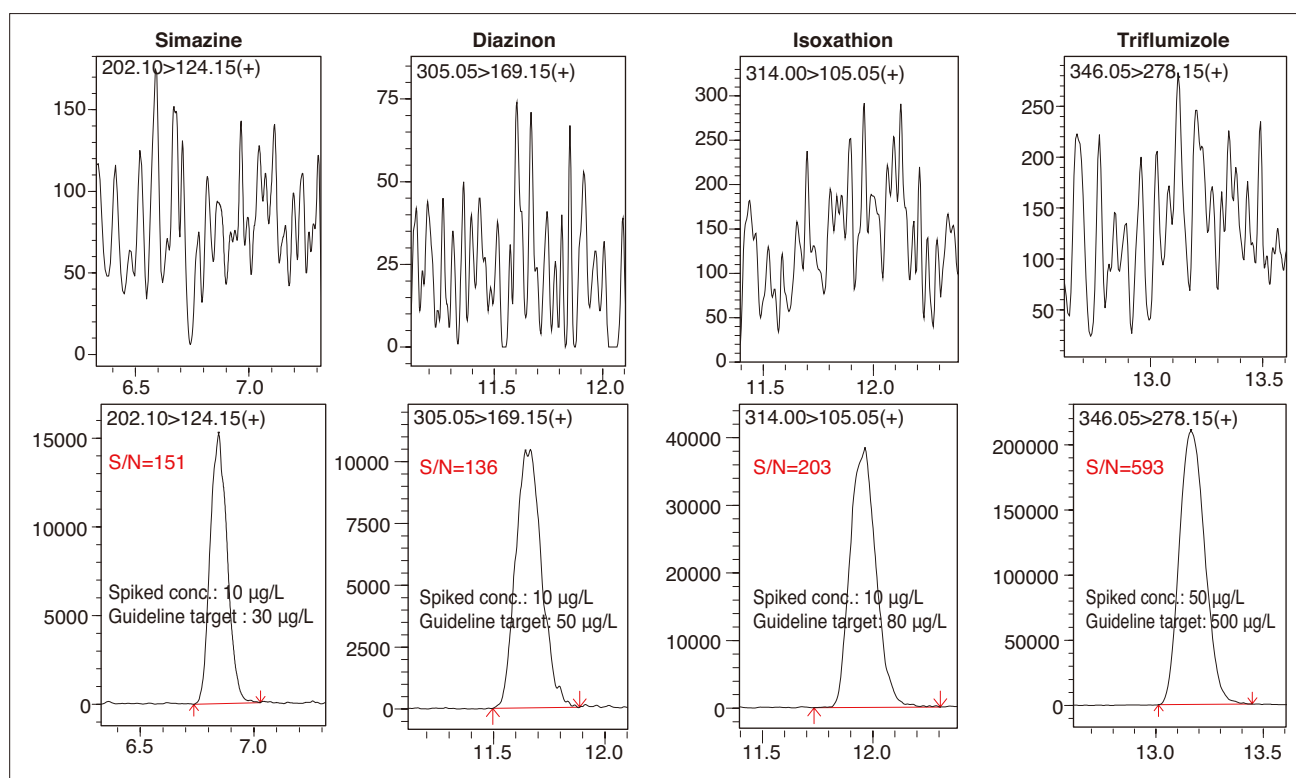


Fig. 3 MRM Measurement Results for Golf Course Wastewater (Upper: Blank, Lower: Spiked with standards at 10 µg/L or 50 µg/L)

Table 2 Analytical Conditions

Column	: Shim-pack FC-ODS (2.0 mm I.D. × 150 mm L., 3 µm)	Probe Voltage	: +4.5 kV (ESI-positive mode), -3.5 kV (ESI-negative mode)
Mobile Phase A	: 5 mmol/L Ammonium acetate - water	Nebulizing Gas Flow	: 1.5 L/min
Mobile Phase B	: 5 mmol/L Ammonium acetate - methanol	Drying Gas Flow	: 10 L/min
Gradient Program	: 30 %B (0 min) – 70 %B (5 min) – 90 %B (15-20 min) – 30 %B (20.01-25 min)	DL Temperature	: 250 °C
Flow Rate	: 0.2 mL/min	BH Temperature	: 400 °C
Injection Volume	: 2 µL	DL Voltage/Q-array Voltage	: Using default values
Column Temperature	: 40 °C		



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