

Application Data Sheet

No. 40

LC-MS

Liquid Chromatograph Mass Spectrometer

Simultaneous Analysis of 36 Veterinary Drugs using Triple Quadrupole LC/MS/MS

The Japanese Ministry of Health, Labor, and Welfare (MHLW) has established an analytical standard for each veterinary drug found in food. Instrumentation measuring these standards must have sensitivity exceeding 1 ng / mL per analyte in order to detect trace levels of these drugs in food. Additionally, because the MHLW has established many compounds as residue standards, instrumentation used for these analyses must have the capability to make simultaneous measurements with sensitivity, specificity, and speed.

This report illustrates the simultaneous analysis of 36 veterinary drugs measured in 15 minutes using the Shimadzu LCMS-8050 Ultra Fast Triple Quadrupole Mass Spectrometer, featuring ultrafast polarity switching. The polarity switching speed of the LCMS-8050 is just 5 milliseconds.

Combination of the Nexera X2 LC and the LCMS-8050 provides incomparably fast run times without compromising the quality of the results.

■ HPLC conditions (Nexera X2)

Column	: YMC-Triart C18 (Manufactured by YMC) (2.0 mmI.D. × 150 mmL., 1.9 μm)
Mobile phase A	: 0.1% formic acid in water
Mobile phase B	: Acetonitrile
Time program	: 1% B. (0.00 min) →10% B. (0.10 min) →80% B. (11.00 min-12.00 min) →1% B. (12.01 -15.00min)
Flow rate	: 0.4 mL/min
Injection volume	: 5 μL
Column oven	: 40 °C

■ MS conditions (LCMS-8050)

Ionization	: ESI (Positive / Negative)
Nebulizing gas flow	: 2.0 L/min
Drying gas flow	: 10.0 L/min
Heating gas flow	: 10.0 L/min
Interface temperature	: 300 °C
DL temperature	: 200 °C
Heat block temperature	: 200 °C
Interface voltage	: +0.5, +1.5, +5.0kV (Positive) -1.0, -4.0kV (Negative)

* Interface voltage was optimized for each compound

Compound	Polarity	Precursor (m/z)	Product (m/z)	Area reproducibility (1 ng/mL)	Compound	Polarity	Precursor (m/z)	Product (m/z)	Area reproducibility (1 ng/mL)
Dexamethasone	+	393.30	337.30	10.3%	Bromacil	+	261.00	205.00	0.6%
Clopidol	+	191.70	101.10	2.2%	Diaveridine	+	261.15	123.10	0.9%
Enrofloxacin	+	360.00	316.20	4.1%	Famphur	+	326.00	93.10	0.9%
Flubendazole	+	314.10	282.05	3.2%	Josamycin	+	828.55	173.95	2.4%
Flumequine	+	262.10	244.05	0.9%	Meloxicam	+	352.10	115.20	1.2%
Mebendazole	+	296.10	264.00	2.1%	Menbutone	+	259.25	241.10	2.8%
Nalidixic acid	+	233.10	215.05	1.5%	Oxibendazole	+	250.00	176.30	0.5%
Orbifloxacin	+	396.00	352.20	6.8%	Sulfaethoxyipyridazine	+	295.15	156.05	1.6%
Oxolinic acid	+	261.90	160.00	4.2%	Valemuline	+	565.40	263.20	3.7%
Sarafloxacin	+	386.00	299.10	9.3%	Cafoperzone	+	646.40	143.30	9.4%
Tylosin	+	916.50	174.10	2.2%	Difloxacin	+	400.10	356.20	2.2%
Warfarin	+	309.05	163.00	0.9%	Methylprednisolone	+	375.20	161.20	3.6%
Ciprofloxacin	+	331.90	288.20	11.7%	Nafcillin	+	415.10	199.10	1.3%
Thiabendazole	+	202.10	175.00	2.5%	Ofloxacin	+	362.10	148.30	12.4%
Levamisole	+	205.10	178.20	3.4%	Phenoxyethylpenicillin	-	349.10	208.30	1.1%
Sulfachloropyridazine	+	285.10	156.10	1.2%	Thiamphenicol	-	353.80	185.10	11.3%
5-Hydroxythiabendazole	+	218.10	191.20	1.7%	Florfenicol	-	356.10	185.30	5.4%
Benzocaine	+	166.10	138.20	8.4%	Clorsulon	-	377.90	342.20	2.2%

Table 1: MRM transition and area reproducibility at 1 ng / mL

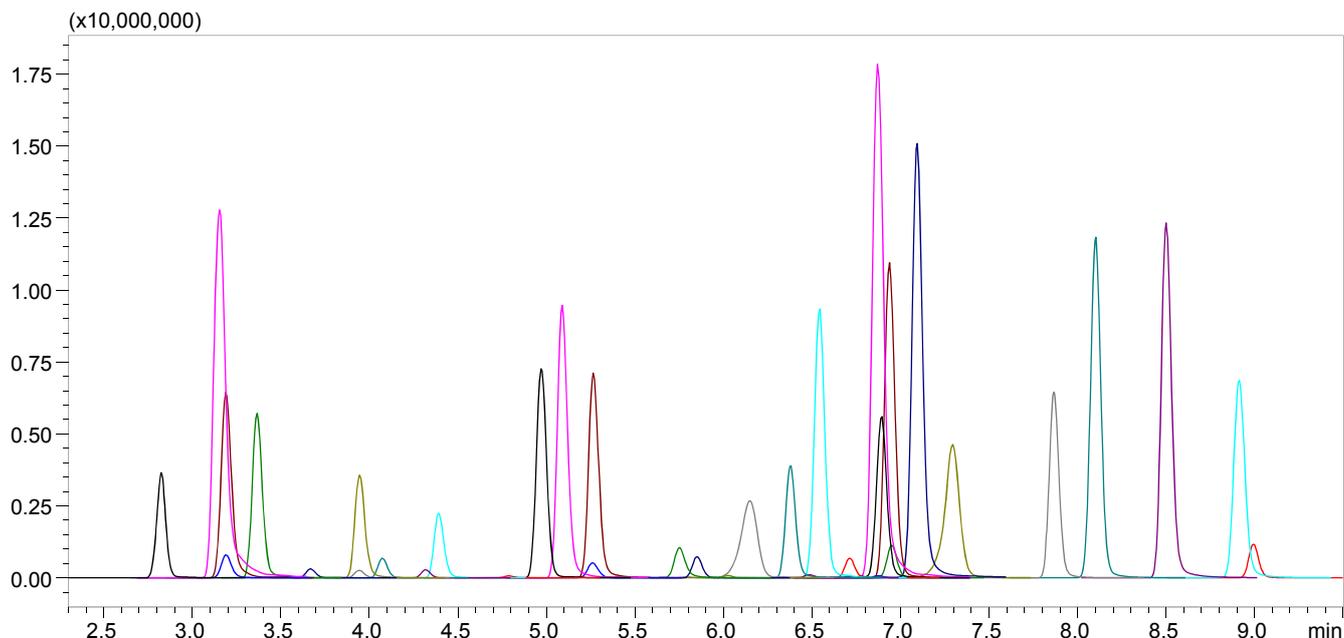


Fig. 1: Representative MRM chromatograms for 36 veterinary drugs at 0.5 µg / mL

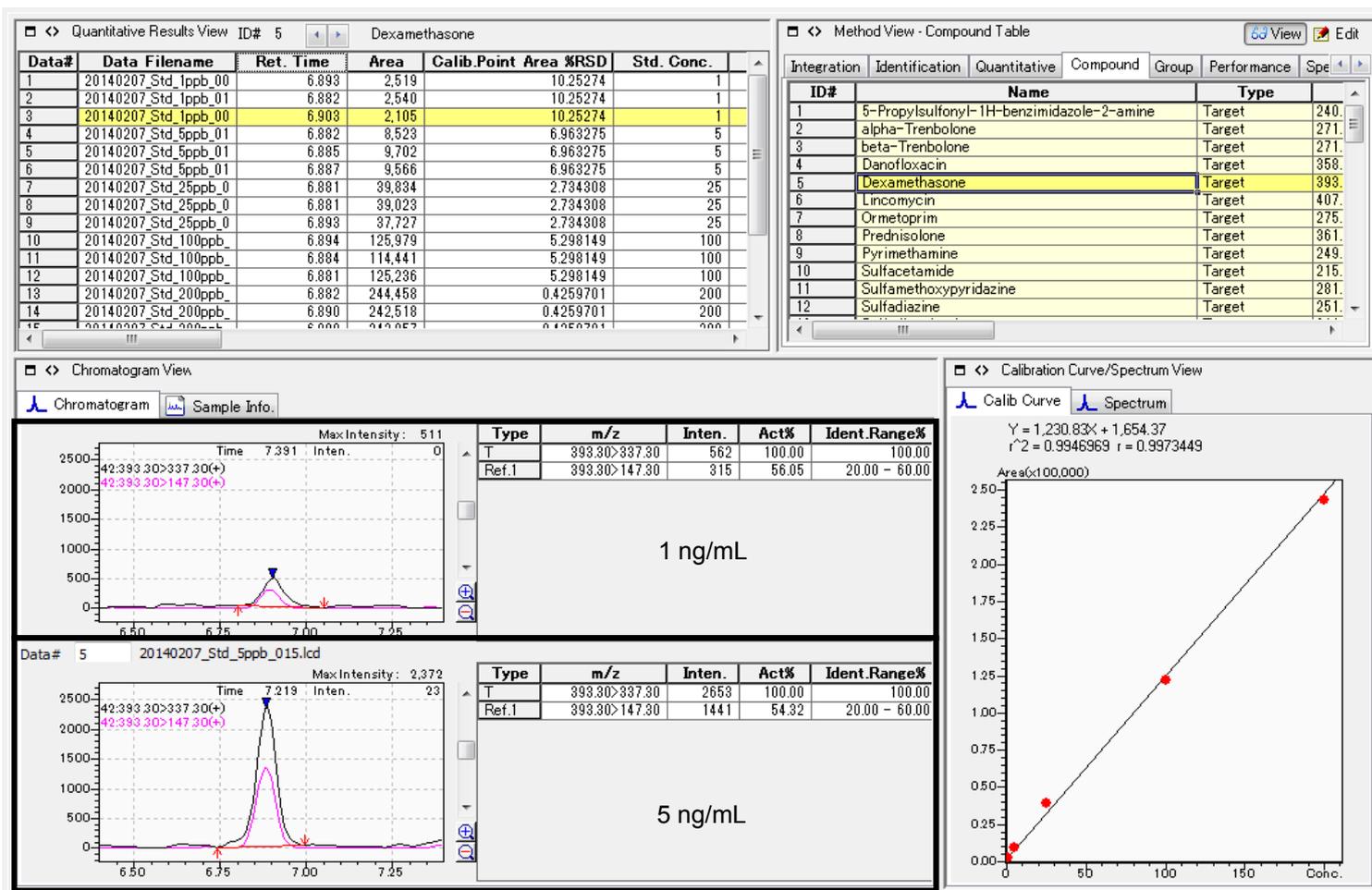


Fig. 2: Results of Dexamethasone

Area %RSDs of these 36 veterinary drugs are less than 20% at 1 ng / mL and all compounds show excellent linearity (R²), greater than 0.99. Results above are from Dexamethasone.