Waters® Alliance™ LC/MS System

Analysis of Pesticides (II)
Metribuzin & their metabolites in Rice

Jun Yonekubo, Nihon Waters, JAPAN

Analytical Conditions

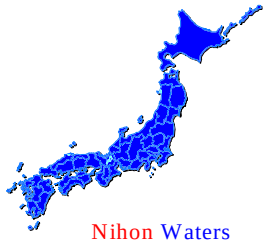
Chromatograms

Mass Spectra

Calibration

Quantitation

Key Words
Pesticides, sensitivity, calibration, electrospray



Nihon Waters
LC/MS Application Labo.

 ©1999 Waters Corporation

Analytical Conditions

Waters Alliance 2690 HPLC Pump Conditions

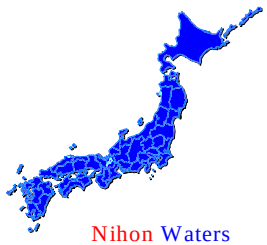
Column	Symmetry C18, 3.5 um, 2.1 x 150 mm
0.3% AcOH aq.	75.0
ACN	18.0
MeOH	7.0
Flow (ml/min)	0.30
Run Time (mins)	15.0
Column Temperature (ø C)	40.0

Waters ZMD MS Detector Conditions

MS
Ion Energy: 3.5 Volts
LM Resolution: 10.7
HM Resolution: 10.7
Multiplier: 650 Volts

Pressures Analyser Vacuum: 2.6e-5 mBar
Gas Flow: 3.4 lit/hr

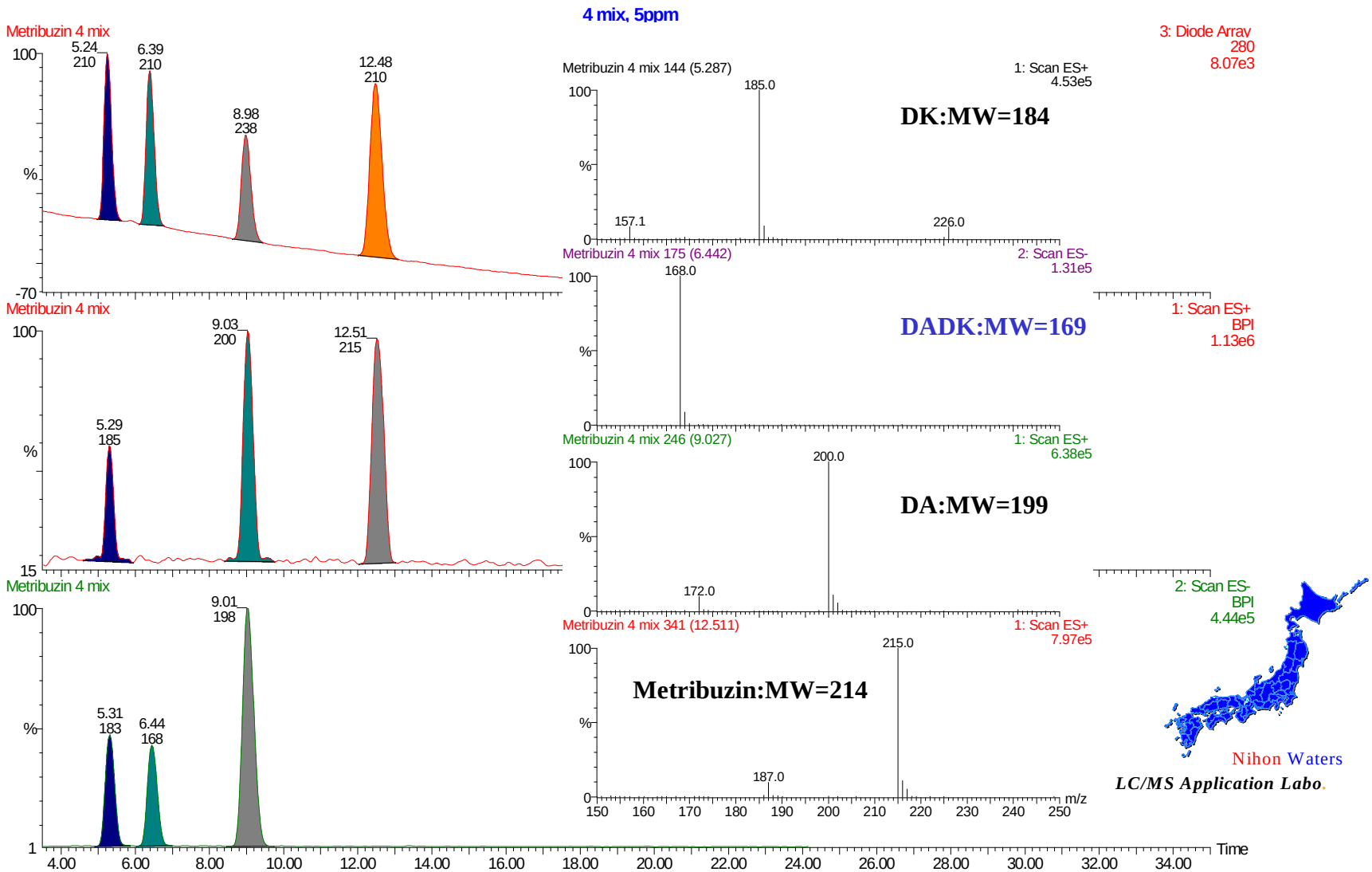
Ionization mode:	ES/APcI+	Ionization mode:	ES/APcI-
Function type:	Scan	Function type:	Scan
Mass range:	150 to 250	Mass range:	150 to 250
Cone Voltage:	35	Cone Voltage:	35



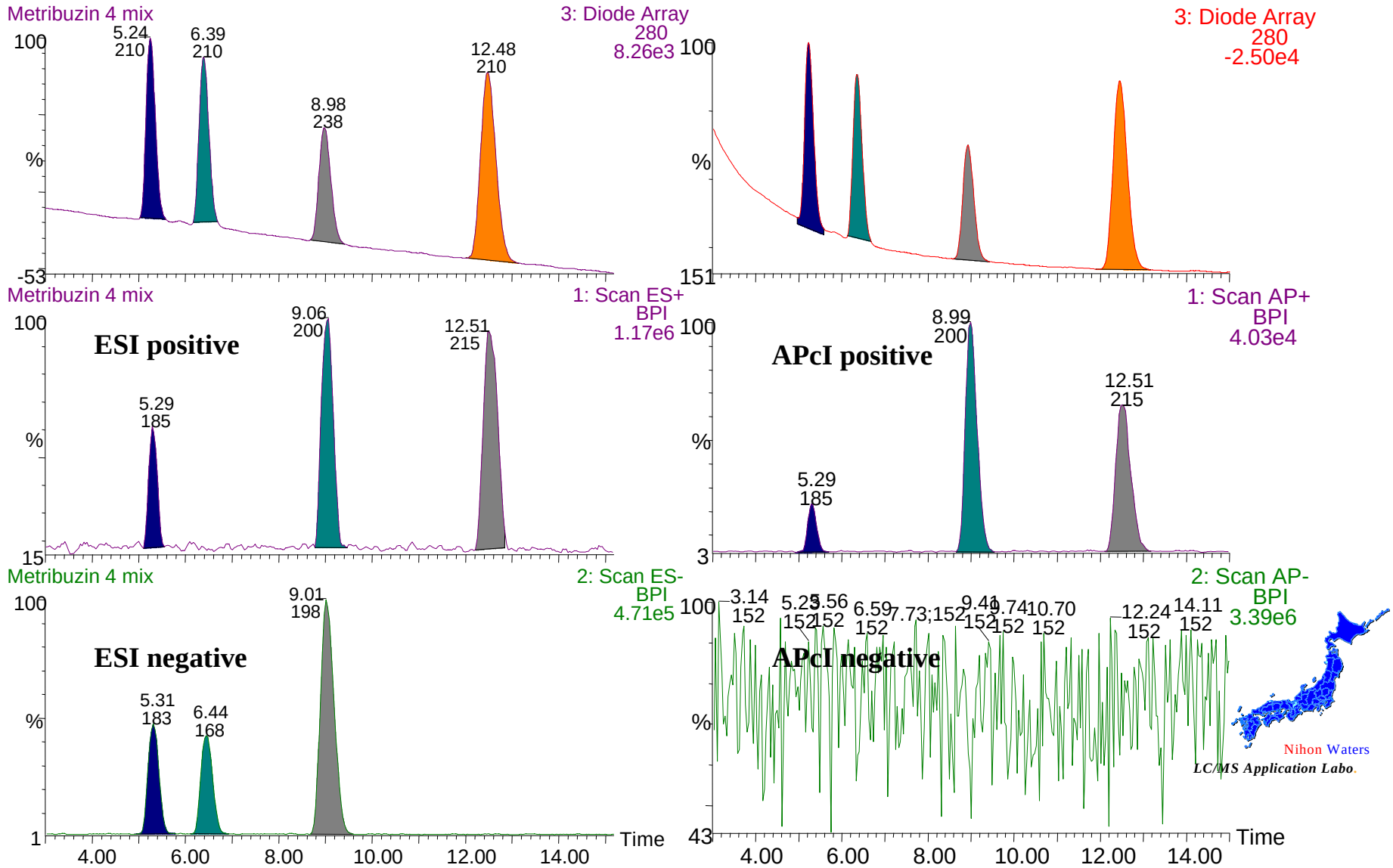
Nihon Waters
LC/MS Application Labo.

 ó1999 Waters Corporation

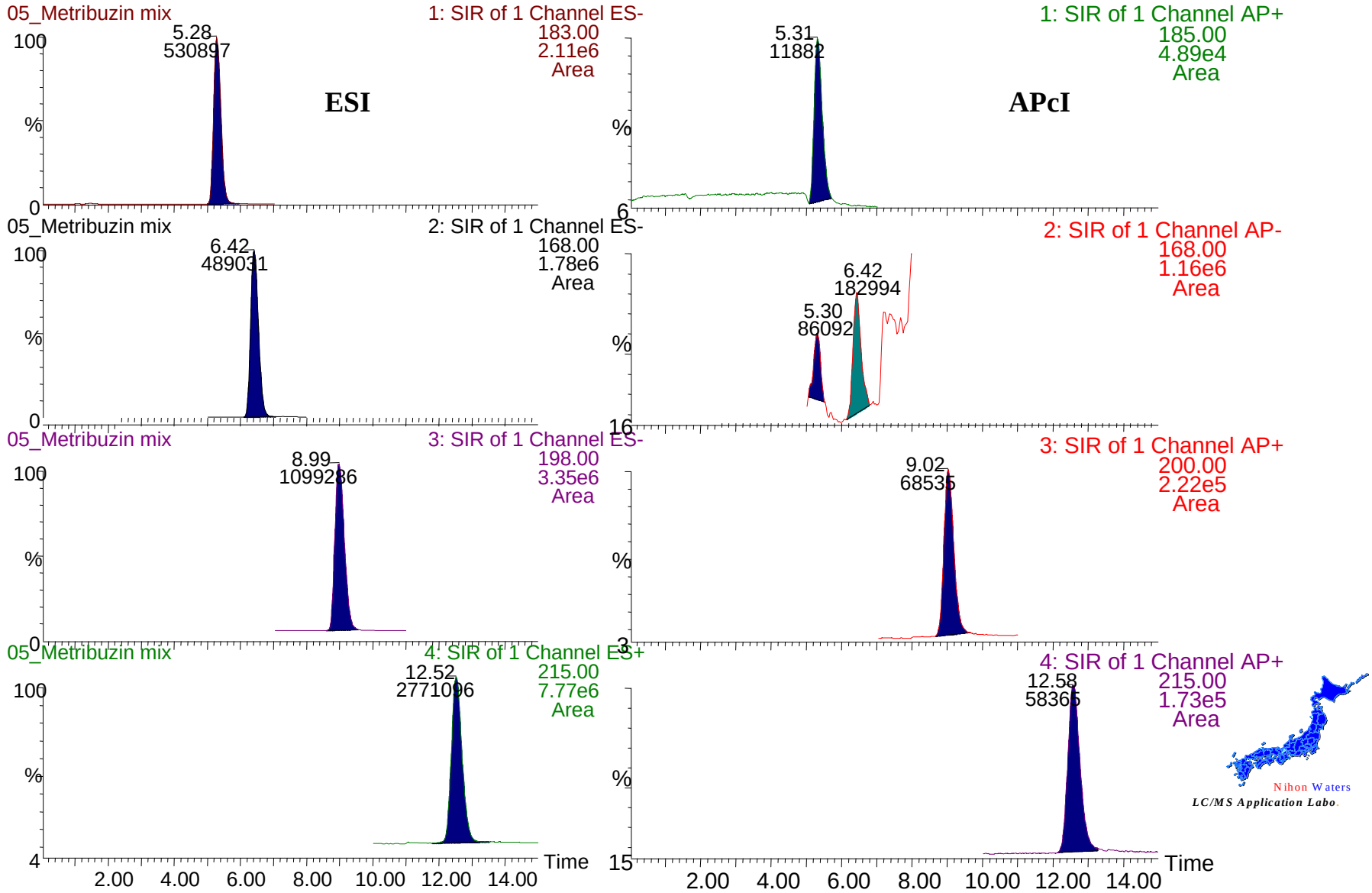
Metribuzin and their 3 metabolites (5ppm) standard mixture



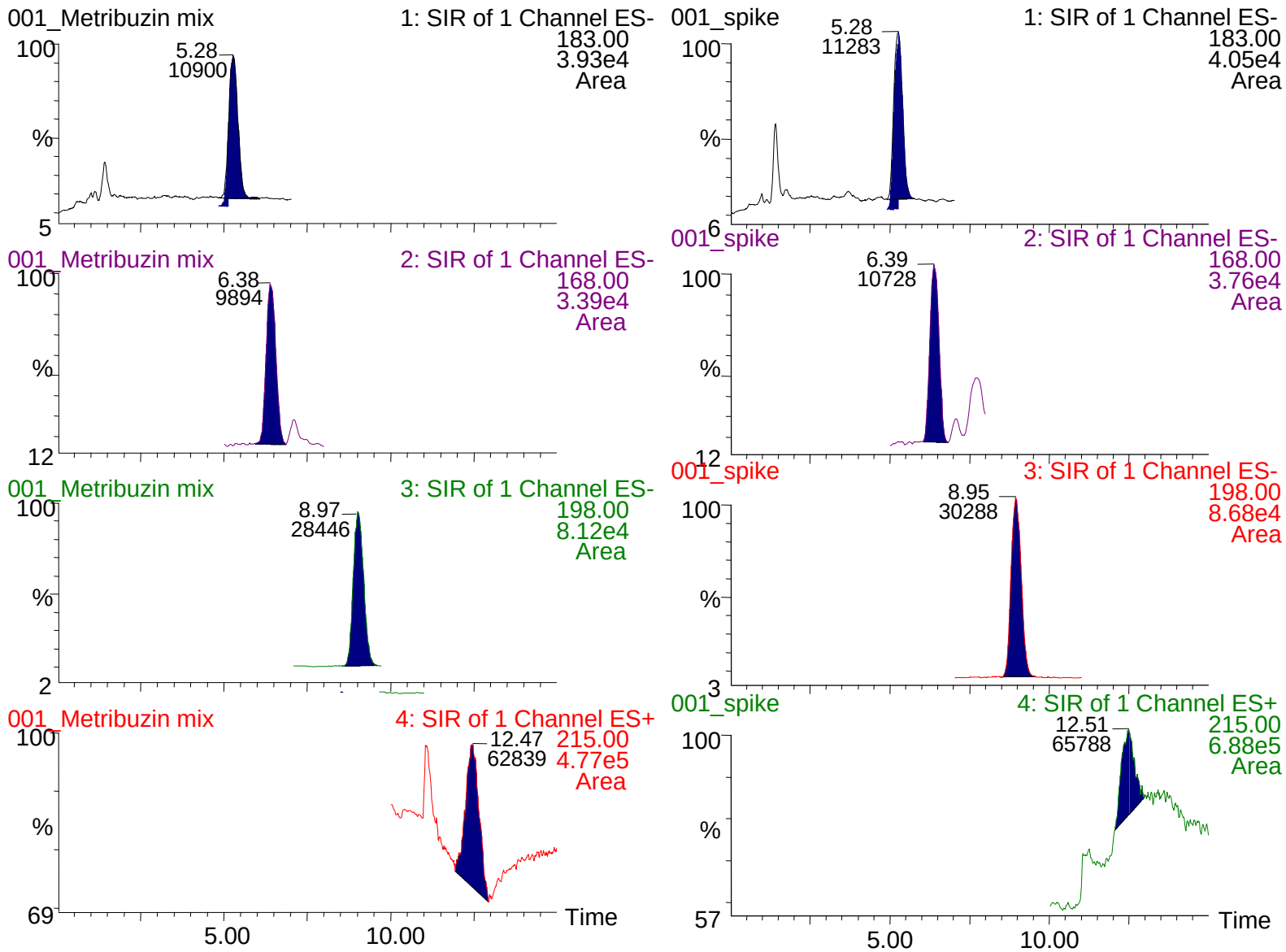
ESI (left) and APcI (right) detection of 5ppm std.mixture.

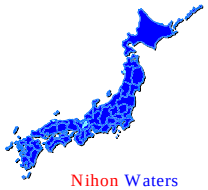


ESI (left) and APcI (right) detection of 0.5ppm standard mixture



10ppb standard mixture (left) and Spiked Rice Sample (right)

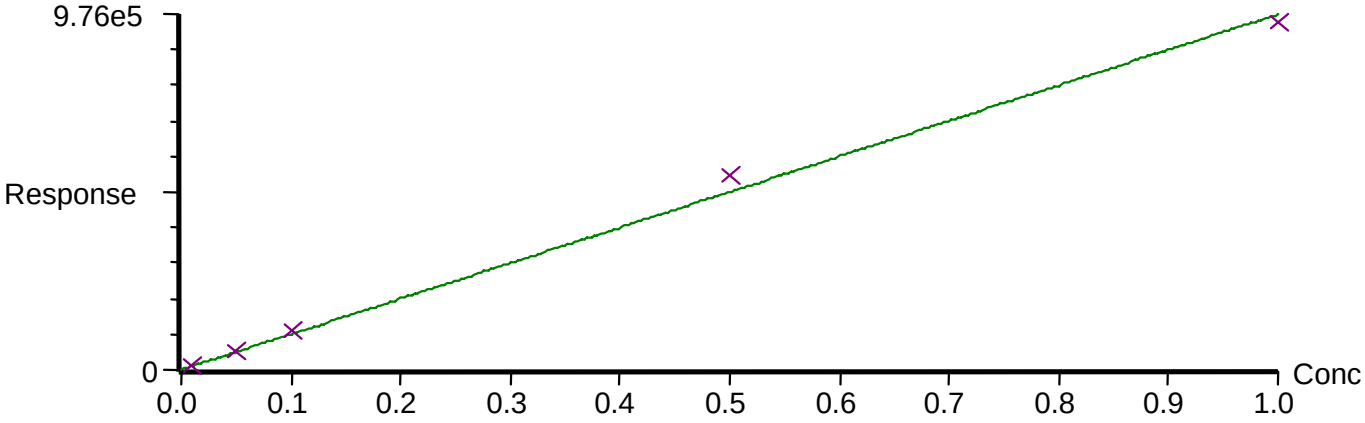




Nihon Waters
LC/MS Application Labo.

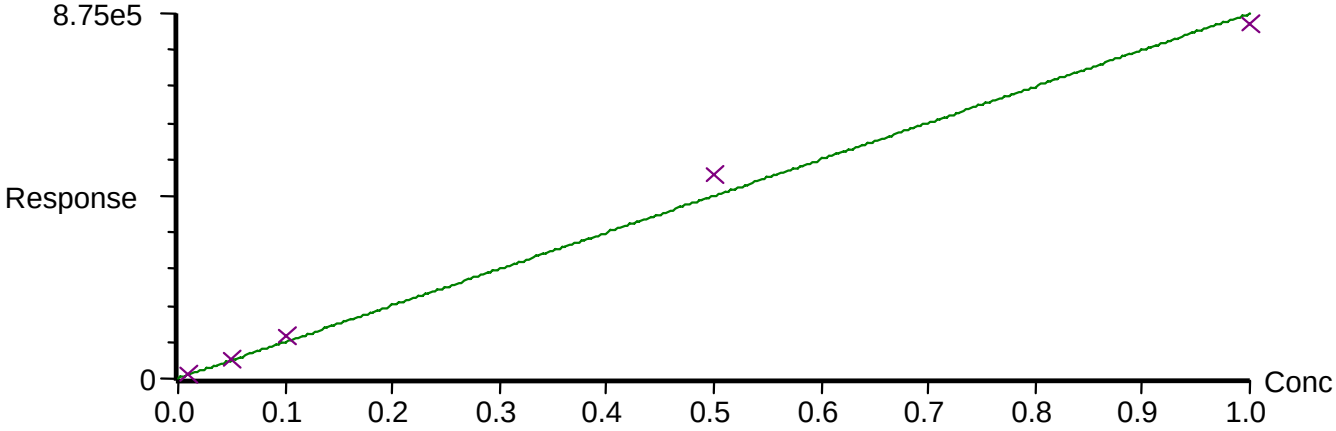
Compound 1 name: DK

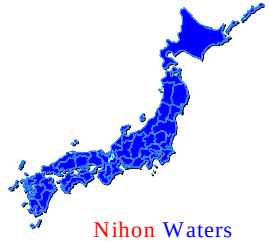
Coefficient of Determination: 0.996118
Calibration curve: 975607 * x + 0
Response type: External Std, Area
Curve type: Linear, Origin: Force, Weighting: Null, Axis trans: None



Compound 2 name: DADK

Coefficient of Determination: 0.992975
Calibration curve: 874956 * x + 0
Response type: External Std, Area
Curve type: Linear, Origin: Force, Weighting: Null, Axis trans: None



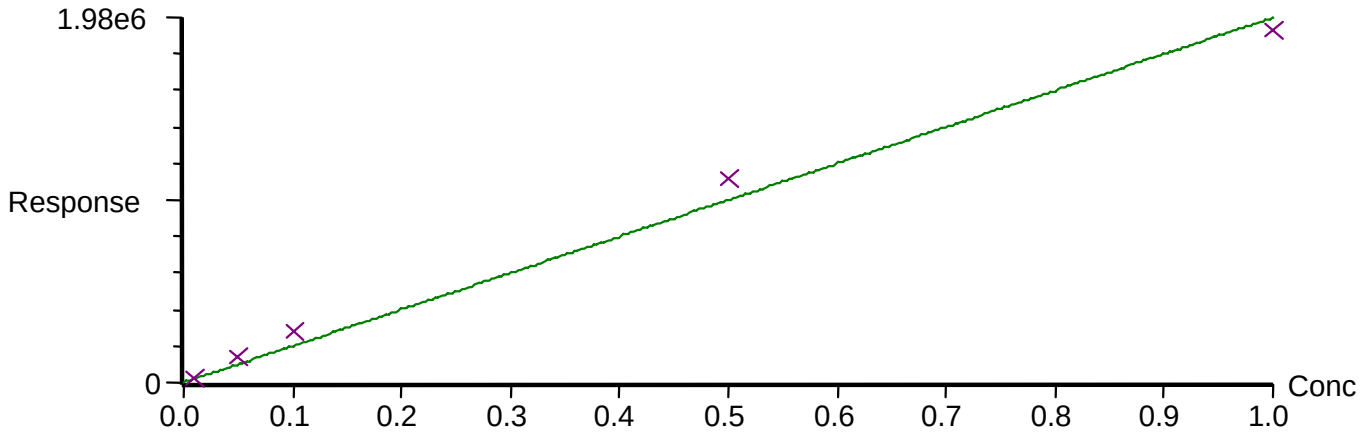


Nihon Waters
LC/MS Application Labo.

 ©1999 Waters Corporation

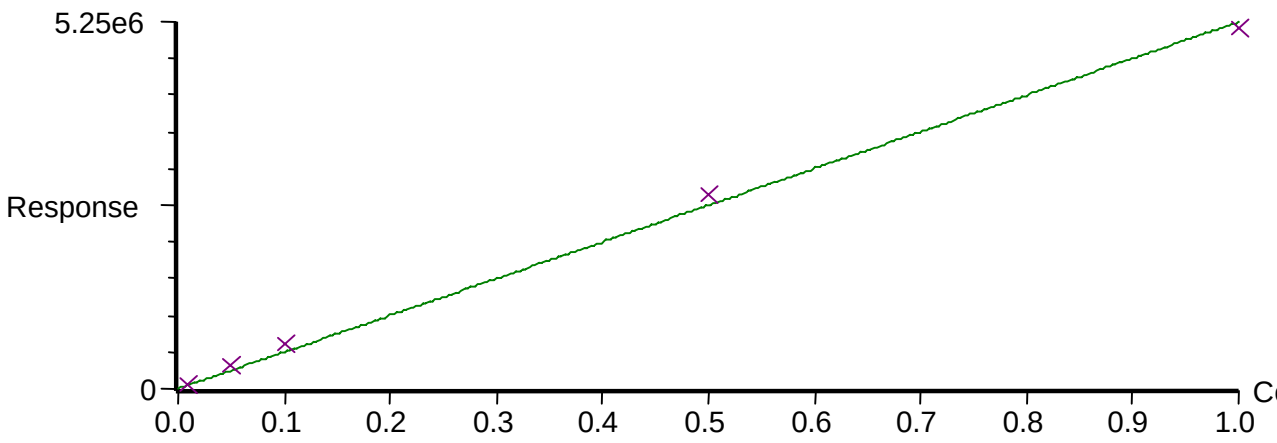
Compound 3 name: DA

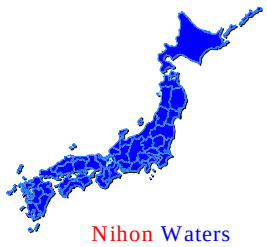
Coefficient of Determination: 0.990900
Calibration curve: 1.97741e6 * x + 0
Response type: External Std, Area
Curve type: Linear, Origin: Force, Weighting: Null, Axis trans: None



Compound 4 name: Metribuzin

Coefficient of Determination: 0.997558
Calibration curve: 5.25398e6 * x + 0
Response type: External Std, Area
Curve type: Linear, Origin: Force, Weighting: Null, Axis trans: None





Nihon Waters
LC/MS Application Labo.

Quantify Compound Summary Report (I)

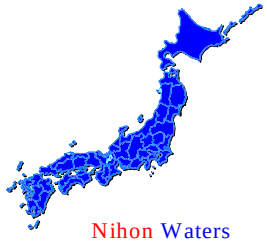
Compound 1: DK

Name	Type	Std Conc*	RT	Area	Conc.*	BS/Conc.*
001_Metribuzin mix	Standard	0.01	5.279	10900	0.01	0.01
005_Metribuzin mix	Standard	0.05	5.280	52009	0.05	0.05
01_Metribuzin mix	Standard	0.10	5.280	109806	0.11	0.11
05_Metribuzin mix	Standard	0.50	5.280	530897	0.54	0.54
1_Metribuzin mix	Standard	1.00	5.246	952662	0.98	0.98
Control	Analyte		5.008	448	0.00	0.00
001_spike	Analyte		5.280	11283	0.01	0.01
005_spike	Analyte		5.279	56061	0.06	0.06

Compound 2: DADK

Name	Type	Std Conc*	RT	Area	Conc.*	BS/Conc.*
001_Metribuzin mix	Standard	0.01	6.385	9894	0.01	0.01
005_Metribuzin mix	Standard	0.05	6.419	48219	0.06	0.06
01_Metribuzin mix	Standard	0.10	6.419	102653	0.12	0.12
05_Metribuzin mix	Standard	0.50	6.419	489031	0.56	0.56
1_Metribuzin mix	Standard	1.00	6.385	847429	0.97	0.97
Control	Analyte		6.216	593	0.00	0.00
001_spike	Analyte		6.386	10728	0.01	0.01
005_spike	Analyte		6.418	56317	0.06	0.06

* ppm



Nihon Waters
LC/MS Application Labo.

Quantify Compound Summary Report (II)

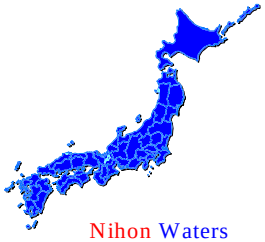
Compound 3: DA

Name	Type	Std Conc*	RT	Area	Conc.*	BS/Conc.*
001_Metribuzin mix	Standard	0.01	8.969	28446	0.01	0.01
005_Metribuzin mix	Standard	0.05	9.021	134903	0.07	0.07
01_Metribuzin mix	Standard	0.10	9.004	273264	0.14	0.14
05_Metribuzin mix	Standard	0.50	8.986	1099286	0.56	0.56
1_Metribuzin mix	Standard	1.00	8.953	1912679	0.97	0.97
Control	Analyte		8.767	1797	0.00	0.00
001_spike	Analyte		8.953	30288	0.02	0.01
005_spike	Analyte		8.986	146491	0.07	0.07

Compound 4: Metribuzin

Name	Type	Std Conc*	RT	Area	Conc.*	BS/Conc.*
001_Metribuzin mix	Standard	0.01	12.471	62839	0.01	0.01
005_Metribuzin mix	Standard	0.05	12.490	324447	0.06	0.06
01_Metribuzin mix	Standard	0.10	12.474	642414	0.12	0.12
05_Metribuzin mix	Standard	0.50	12.524	2771096	0.53	0.53
1_Metribuzin mix	Standard	1.00	12.440	5167030	0.98	0.98
Control	Analyte		12.338	22315	0.00	0.00
001_spike	Analyte		12.507	65788	0.01	0.01
005_spike	Analyte		12.489	342316	0.07	0.07

* ppm



Nihon Waters
LC/MS Application Labo.