Supporting Information

Silver mediated mild oxidation of nitrogen containing heteroaryl-2-methanols to esters

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I. General Methods and Materials

Analytical thin layer chromatography was carried out on silica pre-coated glass plates (Silica gel 60 F₂₅₄, 0.25 mm thickness) and visualized with UV light at 254 nm. Purification of all the compounds was carried out on Redisep 4 g silica flash column (Catalog No: 69-2203-304) using Biotage Isolera™ Flash Purification System. ¹H and ¹³C NMR spectra were recorded on Bruker 400-MHz Ultrashield Advance II 400 model. Chemical shifts (δ) are reported in parts per million (ppm) and with reference to tetramethylsilane (TMS) as internal standard. Liquid chromatography/mass spectrometry (LC/MS) data was obtained to verify molecular mass and analyze purity of products. The specifications of the LC/MS instrument are the following: electrospray (+) ionization, mass range of 100-1000 Da, 20V cone voltage, Acquity BEH C-18 column (2.1 x 100mm, 1.7 μ m), gradient mobile phase consisting of 5 mM ammonium acetate in water and acetonitrile, and a flow rate of 0.3 mL/min.

II. Characterization data of Synthesized Compounds:

Isopropyl 6-bromopicolinate (2a)

¹H NMR (400 MHz, CDCl₃): δ 8.01 (m, 1H), 7.64 (m, 2H), 5.27 (sep, 1H, *J* = 6.00 Hz), 1.38 (d, 6H, *J* = 6.40 Hz) ppm; **¹³C NMR (100 MHz, CDCl₃):** δ 163.35, 149.49, 142.30, 139.15, 131.61, 123.93, 70.14, 21.86 ppm; **LC-MS** Purity: 97.34%. **HRMS** calcd: 242.9895 found: 242.9892.

Isopropyl 5-bromopicolinate (2b)

¹H NMR (400 MHz, CDCl₃): δ 8.80 (d, 1H, *J* = 2.00 Hz), 7.96 (m, 2H), 5.32 (sep, 1H, *J* = 6.00 Hz), 1.40 (d, 6H, *J* = 6.00 Hz) ppm; **¹³C NMR (100 MHz, CDCl₃):** δ 164.11, 151.09, 146.96, 139.60, 126.24, 124.76, 69.94, 22.11 ppm; **LC-MS** Purity: 92.25%. **HRMS** calcd: 242.9895 found: 242.9891.

Isopropyl 4-bromopicolinate (2c)

¹H NMR (400 MHz, CDCl₃): δ 8.53 (d, 1H, *J* = 5.20 Hz), 8.23 (d, 1H, *J* = 1.60 Hz), 7.60 (dd, 1H, *J* = 1.60 Hz & 5.20 Hz), 5.30 (sep, 1H, *J* = 6.00 Hz), 1.39 (d, 6H, *J* = 6.00 Hz) ppm; **¹³C NMR (100 MHz, CDCl₃):** δ 163.58, 150.58, 149.74, 133.80, 129.93, 128.55, 70.17, 21.86 ppm; **LC-MS Purity:** 93.48%. **HRMS** calcd: 242.9895 found: 242.9890.

Isopropyl 4-methoxy-3,5-dimethylpicolinate (2d)

¹H NMR (400 MHz, CDCl₃): δ 8.32 (s, 1H), 5.29 (m, 1H), 3.76 (s, 3H), 2.41 (s, 3H), 2.25 (s, 3H), 1.40 (d, 6H, *J* = 6.00 Hz) ppm; **¹³C NMR (100 MHz, CDCl₃):** δ 166.31, 164.78, 149.79, 149.13, 128.95, 127.77, 69.37, 60.08, 21.95, 13.62, 12.06 ppm; **LC-MS Purity:** 99.53%. **HRMS** calcd: 223.1208 found: 223.1211.

Isopropyl 4-chloropicolinate (2e)

¹H NMR (400 MHz, CDCl₃): δ 8.64 (d, 1H, *J* = 5.20 Hz), 8.08 (d, 1H, *J* = 2.00 Hz), 7.44 (dd, 1H, *J* = 2.00 Hz & 5.20 Hz), 5.32 (sep, 1H, *J* = 6.40 Hz), 1.40 (d, 6H, *J* = 6.40 Hz) ppm; **¹³C NMR (100 MHz, CDCl₃):** δ 163.73, 150.79, 150.02, 145.34, 126.93, 125.63, 70.20, 21.89 ppm; **LC-MS Purity:** 99.74%. **HRMS** calcd: 199.0400 found: 199.0403.

Isopropyl 3-chloropicolinate (2f)

¹H NMR (400 MHz, CDCl₃): δ 8.56 (dd, 1H, *J* = 1.20 Hz & 4.40 Hz), 7.77 (dd, 1H, *J* = 1.20 Hz & 8.00 Hz), 7.34 (dd, 1H, *J* = 4.40 Hz & 8.00 Hz), 5.34 (m, 1H), 1.41 (d, 6H, *J* = 6.40 Hz) ppm; **¹³C NMR (100 MHz, CDCl₃):** δ 164.39, 148.88, 147.45, 138.55, 130.54, 126.07, 70.39, 21.84 ppm; **LC-MS** Purity: 98.50%. **HRMS** calcd: 199.0400 found: 199.0401.

Isopropyl 5-methylpicolinate (2g)

¹H NMR (400 MHz, CDCl₃): δ 8.54 (d, 1H, *J* = 0.80 Hz), 7.97 (d, 1H, *J* = 8.40 Hz), 7.57 (dd, 1H, *J* = 1.60 Hz & 8.00 Hz), 5.29 (sep, 1H, *J* = 6.40 Hz), 1.38 (d, 6H, *J* = 6.40 Hz) ppm; **¹³C NMR (100 MHz, CDCl₃):** δ 164.78, 149.90, 148.66, 136.91, 126.67, 125.06, 69.56, and 21.86 ppm; **LC-MS** Purity: 97.42%. **HRMS** calcd: 179.0946 found: 179.0949.

Isopropyl isonicotinate (2i)

¹H NMR (400 MHz, CDCl₃): δ 8.75 (d, 1H, *J* = 4.80 Hz), 7.88 (d, 1H, *J* = 8.00 Hz), 7.84 (d, 1H, *J* = 5.20 Hz), 5.28 (m, 1H), 1.38 (d, 6H, *J* = 6.40 Hz) ppm; **¹³C NMR (100 MHz, CDCl₃):** δ 164.60, 150.50, 138.06, 136.90, 126.48, 122.84, 69.55, 21.84 ppm; **LC-MS** Purity: 98.31%. **HRMS** calcd: 165.0790 found: 165.0794.

Isopropyl pyrazine-2-carboxylate (2j)

¹H NMR (400 MHz, CDCl₃): δ 9.29 (d, 1H, *J* = 1.20 Hz), 8.74 (d, 1H, *J* = 2.40 Hz), 8.73 (d, 1H, *J* = 1.60 Hz), 5.39 (m, 1H), 1.43 (d, 6H, *J* = 6.40 Hz) ppm; **¹³C NMR (100 MHz, CDCl₃):** δ 163.60, 147.58, 146.39, 144.54, 144.05, 70.39, 21.95 ppm; **LC-MS** Purity: 99.49%. **HRMS** calcd: 166.0742 found: 166.0745.

Isopropyl imidazo[1,2-*a*]pyridine-2-carboxylate (2k)

¹H NMR (400 MHz, DMSO-*d*₆): δ 8.53 (m, 2H), 7.61 (d, 1H, *J* = 9.20 Hz), 7.32 (m, 1H), 6.98 (m, 1H), 5.14 (sep, 1H, *J* = 6.40 Hz), 1.31 (d, 6H, *J* = 6.00 Hz) ppm; **¹³C NMR (100 MHz, DMSO-*d*₆):** δ 162.23, 144.47, 135.80, 127.56, 126.81, 117.92, 117.81, 113.51, 67.54, 21.74 ppm; **LC-MS** Purity: 87.93%. **HRMS** calcd: 204.0899 found: 204.0903.

Isopropyl 1-methyl-1*H*-indazole-3-carboxylate (2l)

¹H NMR (400 MHz, CDCl₃): δ 8.20 (d, 1H, *J* = 8.40 Hz), 7.44 (d, 1H, *J* = 0.80 Hz), 7.43 (s, 1H), 7.30 (m, 1H), 5.40 (m, 1H), 4.16 (s, 3H), 1.46 (d, 6H, *J* = 6.40 Hz) ppm; **¹³C NMR (100 MHz, CDCl₃):** δ 162.43, 141.13, 135.28, 126.91, 123.71, 123.07, 122.43, 109.56, 68.64, 36.46, 22.17 ppm; **LC-MS Purity:** 93.40%. **HRMS** calcd: 218.1055 found: 218.1058.

Isopropyl 1-methyl-1*H*-imidazole-4-carboxylate (2m)

¹H NMR (400 MHz, CDCl₃): δ 7.51 (s, 1H), 7.39 (s, 1H), 5.19 (m, 1H), 3.68 (s, 3H), 1.30 (d, 6H, *J* = 6.00 Hz) ppm; **¹³C NMR (100 MHz, CDCl₃):** δ 162.43, 138.54, 134.54, 126.03, 67.79, 33.78, 21.96 ppm; **LC-MS Purity:** 72.27%. **HRMS** calcd: 168.0899 found: 168.0901.

Isopropyl quinoline-2-carboxylate (2n)

^1H NMR (400 MHz, CDCl_3): δ 8.30 (m, 2H), 8.15 (d, 1H, $J = 8.40$ Hz), 7.87 (d, 1H, $J = 8.00$ Hz), 7.78 (m, 1H), 7.64 (m, 1H), 5.39 (m, 1H), 1.47 (d, 6H, $J = 6.40$ Hz) ppm; **^{13}C NMR (100 MHz, CDCl_3):** δ 164.93, 148.71, 147.79, 137.23, 131.00, 130.20, 129.31, 128.55, 127.56, 121.07, 70.00, 22.01 ppm; **LC-MS** Purity: 85.38%. **HRMS** calcd: 215.0946 found: 215.0947.

Isopropyl 1-ethyl-1H-1,2,4-triazole-5-carboxylate (2o)

^1H NMR (400 MHz, CDCl_3): δ 7.94 (s, 1H), 5.30 (m, 1H), 4.64 (q, 2H, $J = 7.20$ Hz), 1.48 (t, 3H, $J = 7.20$ Hz), 1.42 (d, 6H, $J = 6.40$ Hz) ppm; **^{13}C NMR (100 MHz, CDCl_3):** δ 157.60, 150.75, 144.12, 70.79, 46.63, 21.83, 15.49 ppm; **LC-MS** Purity: 95.21%. **HRMS** calcd: 183.1008 found: 183.1010.

Isopropyl benzo[d]thiazole-2-carboxylate (2p)

^1H NMR (400 MHz, CDCl_3): δ 8.25 (d, 1H, $J = 8.00$ Hz), 7.96 (d, 1H, $J = 7.60$ Hz), 7.54 (m, 2H), 5.40 (m, 1H), 1.47 (d, 6H, $J = 6.40$ Hz) ppm; **^{13}C NMR (100 MHz, CDCl_3):** δ

160.36, 159.17, 153.40, 136.88, 127.58, 127.13, 125.65, 122.16, 67.91, 21.93 ppm; **LC-MS**

Purity: 99.26%. **HRMS** calcd: 221.0510 found: 221.0514.

Isopropyl isoquinoline-1-carboxylate (2q)

¹H NMR (400 MHz, CDCl₃): δ 8.67 (d, 1H, J = 8.40 Hz), 8.63 (d, 1H, J = 8.00 Hz), 7.87 (d, 1H, J = 8.00 Hz), 7.80 (d, 1H, J = 5.60 Hz), 7.73 (t, 1H, J = 7.20 Hz), 7.67 (t, 1H, J = 7.20 Hz), 5.46 (m, 1H), 1.49 (d, 6H, J = 6.40 Hz) ppm; **¹³C NMR (100 MHz, CDCl₃):** δ 165.78, 149.80, 141.68, 137.04, 130.73, 128.70, 127.24, 126.61, 126.49, 123.87, 70.13, 22.00 ppm; **LC-MS** Purity: 99.73%. **HRMS** calcd: 215.0946 found: 215.0949.

Isopropyl isoquinoline-3-carboxylate (2r)

¹H NMR (400 MHz, CDCl₃): δ 9.33 (brs, 1H), 8.55 (brs, 1H), 8.03 (d, 1H, J = 8.00 Hz), 7.94 (d, 1H, J = 8.00 Hz), 7.73 (m, 2H), 5.38 (m, 1H), 1.44 (d, 6H, J = 6.40 Hz) ppm; **¹³C NMR (100 MHz, CDCl₃):** δ 165.40, 152.82, 142.20, 135.61, 131.23, 129.95, 129.61,

128.06, 127.84, 123.94, 69.50, 22.07 ppm; **LC-MS** Purity: 97.62%. **HRMS** calcd: 215.0946 found: 215.0948.

Isopropyl 1-methyl-1H-benzo[d]imidazole-2-carboxylate (2s)

¹H NMR (400 MHz, CDCl₃): δ 7.91 (d, 1H, J = 8.00 Hz), 7.41 (m, 2H), 7.32 (m, 1H), 5.36 (m, 1H), 4.14 (s, 3H), 1.46 (d, 6H, J = 6.40 Hz) ppm; **¹³C NMR (100 MHz, CDCl₃):** δ 159.92, 141.79, 141.42, 136.77, 125.42, 123.68, 122.08, 110.39, 70.29, 32.28, 21.92 ppm; **LC-MS** Purity: 99.75%. **HRMS** calcd: 218.1055 found: 218.1059.

J351-Z00589-001

CDCl3

2

Current Data Parameters
NAME J351-Z00589-001
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130118
Time 12.33
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 8223.685 Hz
FIDRES 0.250967 Hz
AQ 1.9923444 sec
RG 128
DW 60.800 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 16.00 usec
PL1 -2.80 dB
PL1W 20.58109283 W
SFO1 400.2624718 MHz

F2 - Processing parameters
SI 65536
SF 400.2600114 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

J351-Z00589-001

CDC13

2

Current Data Parameters
NAME J351-Z00589-001
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130118
Time 12.36
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1961
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.0000000 sec
D11 0.0300000 sec
TD0 1

==== CHANNEL f1 =====
NUC1 13C
P1 8.00 usec
PL1 -2.00 dB
PL1W 57.32743073 W
SFO1 100.6555216 MHz

==== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.80 dB
PL12 15.35 dB
PL13 19.00 dB
PL2W 20.58109283 W
PL12W 0.31511453 W
PL13W 0.13597794 W
SFO2 400.2616010 MHz

F2 - Processing parameters
SI 32768
SF 100.6454570 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Sample Name: J351-Z00589-001

Test Name: 5mM AA-3-5 MIN

18-Jan-2013 12:49:26

180113-J351-Z00589-001

JUBILANT CHEMSYS

UPLC-MS-02

2

1: Scan ES+
TIC
1.60e8

180113-J351-Z00589-001

4: Diode Array
214

S13

J351-Z00589-013

CDC13

Current Data Parameters
NAME J351-Z00589-013
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130130
Time 13.00
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 32892
SOLVENT CDC13
NS 8
DS 0
SWH 8223.685 Hz
FIDRES 0.250021 Hz
AQ 1.9998837 sec
RG 4
DW 60.800 usec
DE 6.50 usec
TE 296.0 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 15.50 usec
PL1 0.00 dB
PL1W 10.09778309 W
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 65536
SF 400.1300102 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

J351-Z00589-013

CDCl₃

Current Data Parameters
NAME J351-Z00589-013
EXNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130205
Time 10.08
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
DL 2.0000000 sec
D1 0.0300000 sec
TD 0

===== CHANNEL f1 =====
NUC1 13C
P1 8.00 usec
PL1 -2.00 dB
PL1W 57.32743073 W
SF01 100.6555216 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.80 dB
PL12 15.35 dB
PL13 19.00 dB
PL2W 20.58109283 W
PL12W 0.31511453 W
PL13W 0.13597794 W
SF02 400.2616010 MHz

F2 - Processing parameters
SI 32768
SF 100.6454507 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Sample Name: J351-Z00589-013
Test Type: MC-LCMS
Test Name: 5mM AA-3-5 MIN
Submitter: JUBILANT CHEMSYS
Run Date: 30-Jan-2013 12:24:47

Instrument ID: UPLC-MS-03

300113-J351-Z00589-013

300113-J351-Z00589-013

300113-J351-Z00589-013

J351-Z00589-013

5mM AA-3-5 MIN

30-Jan-2013 12:24:47

300113-J351-Z00589-013 78 (2.101) Cm (78:79)

Instrument ID:UPLC-MS-03

2: Scan ES+
3.42e7

J351-Z00589-015

CDC13

Current Data Parameters
NAME J351-Z00589-015
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130130
Time 12.56
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 32892
SOLVENT CDC13
NS 8
DS 0
SWH 8223.685 Hz
FIDRES 0.250021 Hz
AQ 1.9998837 sec
RG 4
DW 60.800 usec
DE 6.50 usec
TE 296.1 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 15.50 usec
PL1 0.00 dB
PL1W 10.09778309 W
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 65536
SF 400.1300101 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

J351-Z00589-015

13C CDC13

2b

Current Data Parameters
NAME J351-Z00589-015
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130206
Time 8.55
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 256
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 8.00 usec
PL1 -2.00 dB
PL1W 57.32743073 W
SFO1 100.6555216 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.80 dB
PL12 15.35 dB
PL13 19.00 dB
PL2W 20.58109283 W

Sample Name: J351-Z00589-015
Test Type: MC-LCMS
Test Name: 5mM AA-3-5 MIN
Submitter: JUBILANT CHEMSYS
Run Date: 30-Jan-2013 12:15:49

Instrument ID: UPLC-MS-03

300113-J351-Z00589-015

3: Scan ES-
TIC
3.40e6

300113-J351-Z00589-015

2: Scan ES+
TIC
2.48e8

300113-J351-Z00589-015

4: Diode Array
214
Range: 1.022

J351-Z00589-015

5mM AA-3-5 MIN

30-Jan-2013 12:15:49

300113-J351-Z00589-015 77 (2.074)

Instrument ID:UPLC-MS-03

2b

2: Scan ES+
7.14e7

J351-Z00589-023-A

CDC13

2c

Current Data Parameters
NAME J351-Z00589-023-A
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130208
Time 17.46
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 32892
SOLVENT CDCl3
NS 8
DS 0
SWH 8223.685 Hz
FIDRES 0.250021 Hz
AQ 1.9998837 sec
RG 4
DW 60.800 usec
DE 6.50 usec
TE 294.4 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 15.50 usec
PL1 0.00 dB
PL1W 10.09778309 W
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 65536
SF 400.1300106 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

S23

J351-Z00589-023-A

CDCl₃ **2c**

Current Data Parameters
NAME J351-Z00589-023-A
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130211
Time 9.21
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl₃
NS 256
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.0000000 sec
D11 0.0300000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 8.00 usec
PL1 2.00 dB
PL1W 57.32743073 W
SFO1 100.6555216 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.80 dB
PL12 15.35 dB
PL13 19.00 dB
PL2W 20.58109283 W
PL12W 0.31511453 W
PL13W 0.13597794 W
SFO2 400.2616010 MHz

F2 - Processing parameters
SI 32768
SF 100.6454511 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Test Name:5mM AA-3-5 MIN

08-Feb-201318:08:21

080213-J351-Z00589-023-A 80 (2.067) Cm (80)

080213-J351-Z00589-023-A 78 (2.014) Cm (77:78)

080213-J351-Z00589-023-A 72 (1.858)

080213-J351-Z00589-023-A 60 (1.546)

J351-Z00589-019-A CDC13

2d

Current Data Parameters
NAME J351-Z00589-019-A
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130214
Time 10.51
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 32892
SOLVENT CDC13
NS 8
DS 0
SWH 8223.685 Hz
FIDRES 0.250021 Hz
AQ 1.9998837 sec
RG 128
DW 60.800 usec
DE 6.50 usec
TE 295.7 K
D1 1.0000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 15.50 usec
PL1 0.00 dB
PL1W 10.09778309 W
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 65536
SF 400.1300102 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

S27

J351-Z00589-019-A

CDCl₃ **2d**

Current Data Parameters
NAME J351-Z00589-019-A
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130215
Time 10.09
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl₃
NS 512
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 8.00 usec
PL1 -2.00 dB
PL1W 57.32743073 W
SFO1 100.6555216 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.80 dB
PL12 15.35 dB
PL13 19.00 dB
PL2W 20.58109283 W

Sample Name: J351-Z00589-019-A
Test Type: MC-LCMS
Test Name: 5mM AA-3-5 MIN
Submitter: JUBILANT CHEMSYS
Run Date: 14-Feb-2013 11:23:12

Instrument ID: UPLC-MS-03

140213-J351-Z00589-019-A

2: Scan ES-
TIC
1.28e8

140213-J351-Z00589-019-A

1: Scan ES+
TIC
2.25e8

140213-J351-Z00589-019-A

3: Diode Array
214
Range: 1.57

J351-Z00589-019-A

5mM AA-3-5 MIN

14-Feb-2013 11:23:12

Instrument ID:UPLC-MS-03

140213-J351-Z00589-019-A 109 (2.002) Cm (107:111)

2: Scan ES-
1.53e7

2d

140213-J351-Z00589-019-A 108 (1.975) Cm (106:111)

1: Scan ES+
5.89e7

J351-Z00589-029 CDC13

2e

Current Data Parameters
NAME J351-Z00589-029
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130225
Time 10.50
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 32892
SOLVENT CDCl3
NS 8
DS 0
SWH 8223.685 Hz
FIDRES 0.250021 Hz
AQ 1.9998837 sec
RG 128
DW 60.800 usec
DE 6.50 usec
TE 293.8 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 15.50 usec
PL1 0.00 dB
PL1W 10.09778309 W
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 65536
SF 400.1300104 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

J351-Z00589-029

CDC13

Current Data Parameters
NAME J351-Z00589-029
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130227
Time 9.07
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.0000000 sec
D11 0.0300000 sec
TD0 1

==== CHANNEL f1 =====
NUC1 13C
P1 8.00 usec
PL1 -2.00 dB
PL1W 57.32743073 W
SFO1 100.6555216 MHz

==== CHANNEL f2 =====
CHOPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.80 dB
PL12 15.35 dB
PL13 19.00 dB
PL2W 20.58109283 W
PL12W 0.31511453 W
PL13W 0.13597794 W
SFO2 400.2616010 MHz

F2 - Processing parameters
SI 32768
SF 100.6454490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

250213-J351-Z00589-029 78 (2.023) Cm (78:79)

2: Scan ES+
7.42e7

J351-Z00589-031

CDCl3

2f

Current Data Parameters
NAME J351-Z00589-031
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130228
Time 15.41
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 32892
SOLVENT CDCl3
NS 8
DS 0
SWH 8223.685 Hz
FIDRES 0.250021 Hz
AQ 1.9998837 sec
RG 128
DW 60.800 usec
DE 6.50 usec
TE 295.9 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 15.50 usec
PL1 0.00 dB
PL1W 10.09778309 W
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 65536
SF 400.1300101 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

J351-Z00589-031

CDCl₃

2f

Current Data Parameters
NAME J351-Z00589-031
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130301
Time 19.53
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 12288
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 32800
DW 20.800 usec
DE 6.50 usec
TE 295.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

==== CHANNEL f1 =====
NUC1 13C
P1 7.50 usec
PL1 -3.00 dB
PL1W 71.16858673 W
SFO1 100.623333 MHz

==== CHANNEL f2 =====
CHOPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 14.26 dB
PL13 18.00 dB
PL2W 10.09778309 W
PL12W 0.37863961 W
PL13W 0.16003908 W
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127566 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Sample Name: J351-Z00589-031

JUBILANT CHEMSYS LTD

UPLC-MS-05

Test Name: 5mM AA-3-5 MIN

28-Feb-2013 15:03:37

280213-J351-Z00589-031

3: Scan ES-
TIC
6.23e6

280213-J351-Z00589-031

2: Scan ES+
TIC
3.77e8

280213-J351-Z00589-031

4: Diode Array
214
Range: 6.51e-1

280213-J351-Z00589-031 76 (1.971) Cm (75:77)

2: Scan ES+
4.38e7

2f

J351-Z00589-025

CDC13

Current Data Parameters
NAME J351-Z00589-025
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130211
Time 13.11
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 32892
SOLVENT CDCl3
NS 8
DS 0
SWH 8223.685 Hz
FIDRES 0.250021 Hz
AQ 1.9998837 sec
RG 4
DW 60.800 usec
DE 6.50 usec
TE 295.4 K
D1 1.0000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 15.50 usec
PL1 0.00 dB
PL1W 10.09778309 W
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 65536
SF 400.1300103 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

S39

J351-Z00589-025

CDC13

2g

Current Data Parameters
NAME J351-Z00589-025
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130212
Time 9.23
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 200
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.0000000 sec
D11 0.0300000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 8.00 usec
PL1 2.00 dB
PL1W 57.32743073 W
SFO1 100.6555216 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.80 dB
PL12 15.35 dB
PL13 19.00 dB
PL2W 20.58109283 W
PL12W 0.31511453 W
PL13W 0.13597794 W
SFO2 400.2616010 MHz

F2 - Processing parameters
SI 32768
SF 100.6454500 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

3: Scan ES-
TIC
4.49e5

2: Scan ES+
TIC
1.68e8

4: Diode Array
214
Range: 1.418

Time	Height	Area	Area%
1.39	42950	1031.28	2.58
1.75	1329416	38997.49	97.42

J351-Z00589-009

CDC13

2i

Current Data Parameters
NAME J351-Z00589-009
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130124
Time 17.32
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDC13
NS 8
DS 0
SWH 8223.685 Hz
FIDRES 0.250967 Hz
AQ 1.9923444 sec
RG 128
DW 60.800 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 16.00 usec
PL1 -2.80 dB
PL1W 20.58109283 W
SFO1 400.2624718 MHz

F2 - Processing parameters
SI 65536
SF 400.2600115 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

J351-Z00589-009

CDCl₃ **2i**

Current Data Parameters
NAME J351-Z00589-009
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130128
Time 20.15
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl₃
NS 13250
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 32800
DW 20.800 usec
DE 6.50 usec
TE 296.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 ¹³C
P1 7.50 usec
PL1 -3.00 dB
PL1W 71.16858673 W
SFO1 100.6228298 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 ¹H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 14.26 dB
PL13 18.00 dB
PL2W 10.09778309 W

S44

Sample Name: J351-Z00589-009
Test Type: MC-LCMS
Test Name: 5mM AA-3-5 MIN
Submitter: JUBILANT CHEMSYS
Run Date: 22-Jan-2013 16:47:01

Instrument ID: UPLC-MS-03

220113-J351-Z00589-009

1: Scan ES+
TIC
5.72e7

2i

220113-J351-Z00589-009

4: Diode Array
214
Range: 7.089e-1

S45

J351-Z00589-009

5mM AA-3-5 MIN

22-Jan-2013 16:47:01

Instrument ID:UPLC-MS-03

220113-J351-Z00589-009 82 (2.201) Cm (81:82)

1: Scan ES+
8.99e5

2i

220113-J351-Z00589-009 69 (1.849) Cm (69:71)

1: Scan ES+
1.03e7

J351-Z00589-021 CDC13

2j

Current Data Parameters
NAME J351-Z00589-021
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130208
Time 17.50
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 32892
SOLVENT CDCl3
NS 8
DS 0
SWH 8223.685 Hz
FIDRES 0.250021 Hz
AQ 1.9998837 sec
RG 4
DW 60.800 usec
DE 6.50 usec
TE 294.4 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 15.50 usec
PL1 0.00 dB
PL1W 10.09778309 W
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 65536
SF 400.1300101 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

J351-Z00589-021

CDCl₃ **2j**

Current Data Parameters
NAME J351-Z00589-021
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130211
Time 9.44
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.0000000 sec
D11 0.0300000 sec
TD0 1

==== CHANNEL f1 =====
NUC1 13C
P1 8.00 usec
PL1 2.00 dB
PL1W 57.32743073 W
SFO1 100.6555216 MHz

==== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.80 dB
PL12 15.35 dB
PL13 19.00 dB
PL2W 20.58109283 W
PL12W 0.31511453 W
PL13W 0.13597794 W
SFO2 400.2616010 MHz

F2 - Processing parameters
SI 32768
SF 100.6454450 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Sample Name: J351-Z00589-021
Test Type: MC-LCMS
Test Name: 5mM AA-3-5 MIN
Submitter: JUBILANT CHEMSYS
Run Date: 08-Feb-2013 17:45:31

Instrument ID: UPLC-MS-03

080213-J351-Z00589-021

080213-J351-Z00589-021

080213-J351-Z00589-021

J351-Z00589-021

5mM AA-3-5 MIN

08-Feb-2013 17:45:31

Instrument ID:UPLC-MS-03

080213-J351-Z00589-021 79 (1.442) Cm (78:80)

1: Scan ES+
1.42e6

080213-J351-Z00589-021 67 (1.222) Cm (66:69)

1: Scan ES+
5.78e6

J351-Z00589-027-A

DMSO

2k

Current Data Parameters
NAME J351-Z00589-027-A
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130211
Time 13.14
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 32892
SOLVENT DMSO
NS 8
DS 0
SWH 8223.685 Hz
FIDRES 0.250021 Hz
AQ 1.9998837 sec
RG 4
DW 60.800 usec
DE 6.50 usec
TE 295.5 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 15.50 usec
PL1 0.00 dB
PL1W 10.09778309 W
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 65536
SF 400.1300040 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

S51

J351-Z00589-027-A

DMSO

2k

Current Data Parameters
NAME J351-Z00589-027-A
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130214
Time 8.55
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 300
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.0000000 sec
D11 0.0300000 sec
TD0 1

==== CHANNEL f1 =====
NUC1 13C
P1 8.00 usec
PL1 -2.00 dB
PL1W 57.32743073 W
SFO1 100.6555216 MHz

==== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.80 dB
PL12 15.35 dB
PL13 19.00 dB
PL2W 20.58109283 W
PL12W 0.31511453 W
PL13W 0.13597794 W
SFO2 400.2616010 MHz

F2 - Processing parameters
SI 32768
SF 100.6455045 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

S52

Test Name:5mM AA-3-5 MIN

11-Feb-201312:33:01

110213-J351-Z00589-027-A 52 (1.409) Cm (50:52)

1: Scan ES+
5.98e6

110213-J351-Z00589-027-A 56 (1.519) Cm (55:56)

1: Scan ES+
2.88e7

J351-Z00589-037

CDCl3

Current Data Parameters
NAME J351-Z00589-037
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130514
Time 18.32
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 8223.685 Hz
FIDRES 0.250967 Hz
AQ 1.9923444 sec
RG 128
DW 60.800 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 16.00 usec
PL1 -2.80 dB
PL1W 20.58109283 W
SFO1 400.2624718 MHz

F2 - Processing parameters
SI 65536
SF 400.2600112 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

J351-Z00589-037

CDCl₃ **2I**

Current Data Parameters
NAME J351-Z00589-037
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130514
Time 18.35
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl₃
NS 256
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 8.00 usec
PL1 -2.00 dB
PL1W 57.32743073 W
SFO1 100.6555216 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.80 dB
PL12 15.35 dB
PL13 19.00 dB
PL2W 20.58109283 W

Sample Name: J351-Z00589-037
Test Type: MC-LCMS
Test Name: 5mM AA-3-5 MIN
Submitter: JUBILANT CHEMSYS
Run Date: 14-May-2013 18:14:24

Instrument ID: UPLC-MS-03

140513-J351-Z00589-037

J351-Z00589-037

5mM AA-3-5 MIN

14-May-2013 18:14:24

140513-J351-Z00589-037 118 (2.159) Cm (118:120)

Instrument ID:UPLC-MS-03

1: Scan ES+
6.92e7

J351-Z00589-051

CDC13

Current Data Parameters
NAME J351-Z00589-051
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130606
Time 16.05
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 32892
SOLVENT CDCl3
NS 8
DS 0
SWH 8223.685 Hz
FIDRES 0.250021 Hz
AQ 1.9998837 sec
RG 128
DW 60.800 usec
DE 6.50 usec
TE 296.2 K
D1 1.00000000 sec
TD0 1

==== CHANNEL f1 =====
NUC1 1H
P1 15.50 usec
PL1 0.00 dB
PL1W 10.09778309 W
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 65536
SF 400.1300094 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

J351-Z00589-051

CDCl₃

Current Data Parameters
NAME J351-Z00589-051
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130608
Time 9.04
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl₃
NS 432
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 8.00 usec
PL1 -2.00 dB
PL1W 57.32743073 W
SFO1 100.6555216 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.80 dB
PL12 15.35 dB
PL13 19.00 dB
PL2W 20.58109283 W
PL12W 0.31511453 W
PL13W 0.13597794 W
SFO2 400.2616010 MHz

F2 - Processing parameters
SI 32768
SF 100.6454541 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

SampleName: J351-Z00589-051

Vial No : P1-C-07

Inj Date : 6. Jun. 2013

Inj Time: 3:44:06 PM

Method : C:\CHEM32\1\METHODS\ACQ-5MAA-3-5MIN.M

System Name: UHPLC-MS-1

Peak No	RT min	Area	Area %
1	0.243	5.423	0.38
2	0.277	213.969	15.13
3	0.356	87.266	6.17
4	0.444	36.733	2.60
5	0.948	15.451	1.09
6	1.065	5.542	0.39
7	1.085	14.847	1.05
8	1.252	1022.231	72.27
9	1.455	4.363	0.31
10	1.905	8.615	0.61

MS Spectrum

J351-Z00589-033

CDC13

2n

Current Data Parameters
NAME J351-Z00589-033
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130513
Time 18.09
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 32892
SOLVENT CDC13
NS 8
DS 0
SWH 8223.685 Hz
FIDRES 0.250021 Hz
AQ 1.9998837 sec
RG 128
DW 60.800 usec
DE 6.50 usec
TE 298.6 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 15.50 usec
PL1 0.00 dB
PL1W 10.09778309 W
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 65536
SF 400.1300096 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

J351-Z00589-033

CDC13

2n

Current Data Parameters
NAME J351-Z00589-033
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130515
Time 14.36
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 264
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 8.00 usec
PL1 -2.00 dB
PL1W 57.32743073 W
SFO1 100.6555216 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.80 dB
PL12 15.35 dB
PL13 19.00 dB
PL2W 20.58109283 W

Sample Name: J351-Z00589-033
Test Type: MC-LCMS
Test Name: 5mM AA-3-5 MIN
Submitter: JUBILANT CHEMSYS
Run Date: 13-May-2013 18:23:02

Instrument ID: UPLC-MS-03

130513-J351-Z00589-033

2: Scan ES-
TIC
4.95e6

130513-J351-Z00589-033

1: Scan ES+
TIC
2.14e8

130513-J351-Z00589-033

3: Diode Array
214
Range: 8.861e-1

Time	Height	Area	Area%
1.58	27037	913.75	2.68
1.74	60058	2077.79	6.08
2.12	872197	29160.81	85.38
2.32	7383	192.00	0.56
2.36	25805	926.17	2.71
2.38	18440	386.11	1.13
2.43	6257	166.42	0.49
2.57	11821	331.54	0.97

J351-Z00589-033

5mM AA-3-5 MIN

13-May-2013 18:23:02

Instrument ID: UPLC-MS-03

2n

1: Scan ES+
1.82e7

130513-J351-Z00589-033 117 (2.141) Cm (117:122)

1: Scan ES+
6.32e7

130513-J351-Z00589-033 96 (1.755)

1: Scan ES+
5.40e7

J351-Z00589-041

CDC13

Current Data Parameters
NAME J351-Z00589-041
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130527
Time 20.04
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 32892
SOLVENT CDC13
NS 8
DS 0
SWH 8223.685 Hz
FIDRES 0.250021 Hz
AQ 1.9998837 sec
RG 2
DW 60.800 usec
DE 6.50 usec
TE 296.7 K
D1 1.0000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 15.50 usec
PL1 0.00 dB
PL1W 10.09778309 W
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 65536
SF 400.1300096 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

J351-Z00589-041

CDC13

2o

Current Data Parameters
NAME J351-Z00589-041
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130528
Time 14.22
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 1024
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 8.00 usec
PL1 -2.00 dB
PL1W 57.32743073 W
SFO1 100.6555216 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.80 dB
PL12 15.35 dB
PL13 19.00 dB
PL2W 20.58109283 W
PL12W 0.31511453 W
PL13W 0.13597794 W
SFO2 400.2616010 MHz

F2 - Processing parameters
SI 32768
SF 100.6454444 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

S68

Test Name:5mM AA-3-5 MIN

27-May-201319:40:45

270513-J351-Z00589-041 88 (2.405) Cm (87:88)

2: Scan ES+
7.55e6

2o

270513-J351-Z00589-041 77 (2.102) Cm (77:78)

2: Scan ES+
6.74e6

270513-J351-Z00589-041 75 (2.047)

2: Scan ES+
1.42e6

270513-J351-Z00589-041 57 (1.551) Cm (56:57)

2: Scan ES+
8.94e7

J351-Z00589-043

CDCl₃ **2p**

Current Data Parameters
NAME J351-Z00589-043
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130527
Time 20.02
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 32892
SOLVENT CDCl₃
NS 8
DS 0
SWH 8223.685 Hz
FIDRES 0.250021 Hz
AQ 1.9998837 sec
RG 2
DW 60.800 usec
DE 6.50 usec
TE 296.7 K
D1 1.0000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 15.50 usec
PL1 0.00 dB
PL1W 10.09778309 W
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 65536
SF 400.130093 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

J351-Z00589-043

CDC13

2p

Current Data Parameters
NAME J351-Z00589-043
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130528
Time 12.36
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 8.00 usec
PL1 -2.00 dB
PL1W 57.32743073 W
SFO1 100.6555216 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.80 dB
PL12 15.35 dB
PL13 19.00 dB
PL2W 20.58109283 W
PL12W 0.31511453 W
PL13W 0.13597794 W
SFO2 400.2616010 MHz

F2 - Processing parameters
SI 32768
SF 100.6454449 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

J351-Z00589-047

CDC13

2q

Current Data Parameters
NAME J351-Z00589-047
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130603
Time 15.29
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDC13
NS 8
DS 0
SWH 8223.685 Hz
FIDRES 0.250967 Hz
AQ 1.9923444 sec
RG 322
DW 60.800 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 16.00 usec
PL1 -2.80 dB
PL1W 20.58109283 W
SFO1 400.2624718 MHz

F2 - Processing parameters
SI 65536
SF 400.2600112 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

S75

J351-Z00589-047

CDC13

2q

Current Data Parameters
NAME J351-Z00589-047 C-13
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130603
Time 18.00
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 1552
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

==== CHANNEL f1 =====
NUC1 13C
P1 8.00 usec
PL1 -2.00 dB
PL1W 57.32743073 W
SFO1 100.6555216 MHz

==== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.80 dB
PL12 15.35 dB
PL13 19.00 dB
PL2W 20.58109283 W
PL12W 0.31511453 W
PL13W 0.13597794 W
SFO2 400.2616010 MHz

F2 - Processing parameters
SI 32768
SF 100.6454453 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

S76

Sample Name: J351-Z00589-047

JUBILANT CHEMSYS LTD

UPLC-MS-05

Test Name: 5mM AA-3-5 MIN

03-Jun-2013 16:09:56

030613-J351-Z00589-047

2q

3: Scan ES-
TIC
5.89e6

030613-J351-Z00589-047

2: Scan ES+
TIC
2.85e8

030613-J351-Z00589-047

4: Diode Array
214
Range: 1.754

Sample Name: J351-Z00589-047 JUBILANT CHEMSYS LTD

UPLC-MS-05

Test Name: 5mM AA-3-5 MIN

03-Jun-2013 16:09:56

030613-J351-Z00589-047 82 (2.136) Cm (82:83)

3: Scan ES-
9.37e4

030613-J351-Z00589-047 82 (2.127) Cm (82:83)

2: Scan ES+
8.41e7

J351-Z00589-049

CDCl₃ **2r**

Current Data Parameters
NAME J351-Z00589-049
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130604
Time 15.19
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl₃
NS 8
DS 0
SWH 8223.685 Hz
FIDRES 0.250967 Hz
AQ 1.9923444 sec
RG 203
DW 60.800 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 16.00 usec
PL1 -2.80 dB
PL1W 20.58109283 W
SFO1 400.2624718 MHz

F2 - Processing parameters
SI 65536
SF 400.2600112 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

J351-Z00589-049

CDCl₃ **2r**

Current Data Parameters
NAME J351-Z00589-049
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130604
Time 19.33
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl₃
NS 9216
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 8.00 usec
PL1 -2.00 dB
PL1W 57.32743073 W
SFO1 100.6555216 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.80 dB
PL12 15.35 dB
PL13 19.00 dB
PL2W 20.58109283 W
PL12W 0.31511453 W
PL13W 0.13597794 W
SFO2 400.2616010 MHz

F2 - Processing parameters
SI 32768
SF 100.6454450 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

S80

Sample Name: J351-Z00589-049
Test Type: MC-LCMS
Test Name: 5mM AA-3-5 MIN
Submitter: JUBILANT CHEMSYS
Run Date: 04-Jun-2013 15:52:32

Instrument ID: UPLC-MS-03

040613-J351-Z00589-049

2r

040613-J351-Z00589-049

J351-Z00589-049

5mM AA-3-5 MIN

04-Jun-2013 15:52:32

040613-J351-Z00589-049 111 (2.031) Cm (111:113)

Instrument ID:UPLC-MS-03

1: Scan ES+
8.21e7

040613-J351-Z00589-049 102 (1.865)

1: Scan ES+
7.05e6

040613-J351-Z00589-049 88 (1.608)

1: Scan ES+
1.08e7

J351-Z00589-053

CDCl₃ **2s**

Current Data Parameters
NAME J351-Z00589-053
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130604
Time 10.13
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl₃
NS 8
DS 0
SWH 8223.685 Hz
FIDRES 0.250967 Hz
AQ 1.9923444 sec
RG 228
DW 60.800 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

==== CHANNEL f1 =====
NUC1 1H
P1 16.00 usec
PL1 -2.80 dB
PL1W 20.58109283 W
SFO1 400.2624718 MHz

F2 - Processing parameters
SI 65536
SF 400.2600113 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

S83

J351-Z00589-053

CDCl₃ **2s**

Current Data Parameters
NAME J351-Z00589-053
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130604
Time 17.57
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl₃
NS 1024
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.0000000 sec
D11 0.0300000 sec
TD0 1

==== CHANNEL f1 =====
NUC1 13C
P1 8.00 usec
PL1 -2.00 dB
PL1W 57.32743073 W
SF01 100.6555216 MHz

==== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.80 dB
PL12 15.35 dB
PL13 19.00 dB
PL2W 20.58109283 W

Sample Name: J351-Z00589-053
Test Type: MC-LCMS
Test Name: 5mM AA-3-5 MIN
Submitter: JUBILANT CHEMSYS
Run Date: 04-Jun-2013 10:50:42

Instrument ID: UPLC-MS-03

040613-J351-Z00589-053

040613-J351-Z00589-053

J351-Z00589-053
5mM AA-3-5 MIN
04-Jun-2013 10:50:42

Instrument ID:UPLC-MS-03

040613-J351-Z00589-053 120 (2.196) Cm (118:121-106:110)

1: Scan ES+
4.20e6

2s

040613-J351-Z00589-053 107 (1.957) Cm (106:110)

1: Scan ES+
6.88e7