Supporting Information for

An Improved Procedure for the Synthesis of Benzimidazoles using Palladium Catalyzed Aryl-Amination Chemistry

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General Methods

Analytical TLC was performed on precoated glass backed plates (Macherey-Nagel silica gel 60Å) and visualised with ultraviolet light (254 nm). NMR spectra were recorded on a Bruker Avance™-400 spectrometer (¹H NMR at 400 MHz, ¹³C NMR at 100 MHz). Infrared spectra were recorded on a Nicolet Avatar 360 FT-IR spectrometer. HRMS were recorded on a Finnigan MAT 900S spectrometer, using electrospray ionisation. HPLC's were recorded on a Waters Alliance® 2695 system. A gradient elution method was used (first solvent system, S1): 0% MeCN/100% 0.08% aqueous formic acid to 20% MeCN/80% 0.08% aqueous formic acid over 5 min, then to 100% MeCN/0% 0.08% aqueous formic acid over a further 5 min, with a flow rate of 3 mL/min, on a Phenomenex Synergi 50 × 4.6 mm, 4 µm, MAX-RP 80R column. The same elution gradient was used with MeOH and 0.08% aqueous TFA (second solvent system, S2), using a Phenomenex Luna 30 × 4.6 mm, 3 µm, C18 column. A laboratory microwave

instrument using a monomodal cavity and sealed reaction vessels was used: a Smith Creator available from Personal Chemistry AB, Uppsala, Sweden. Melting points were determined on a Reichert Thermovar hot stage microscope and are uncorrected.

General Procedure

To a solution of amidine (0.26 mmol) in DME (90 μ L) in a microwave vial, was added PPh₃ (106 μ L, 0.2 M in DME, 8 mol%), NaOH (0.25 mL, 2.08 M in water, 2.0 equiv) and Pd₂(dba)₃ (53 μ L, 0.05 M in DME, 1 mol%). (The reactions were always carried out in a total volume of 0.5 mL of solvent.) The vial was sealed with a crimped cap and the reaction mixture was heated in the Personal Chemistry Smith Creator microwave instrument at 160°C. The reaction mixture was then diluted with EtOAc (15 mL), washed with brine (10 mL) and was extracted with EtOAc (2 $\times$ 10 mL). The organic layer was dried over MgSO₄, filtered and the solvent removed *in vacuo*. The residue was dissolved in DCM (5 mL) with washed Amberlyst 15 resin (250 mg) and stirred for 18 h. The resin was then washed with DCM (2 $\times$ 5 mL) and PhMe (2 $\times$ 5 mL), and treated with a solution of 50% Et₃N/DCM (5 mL) for 1 h. The resin was washed with DCM (5 mL), the washings were collected and the solvent was removed *in vacuo* to give the pure benzimidazole.

1,2-Dimethyl-5-nitrobenzimidazole (Table 3, Entry 1)

R_f = 0.53 (20% MeOH:EtOAc). mp 233-235°C, lit.¹ mp 231-232°C. ¹H NMR (CDCl₃, 400 MHz): δ 2.67 (3H, s), 3.80 (3H, s), 7.33 (1H, dd, *J* = 8.8), 8.21 (1H, dd, *J* = 2.2, 8.8), 8.59 (1H, d, *J* = 2.2). ¹³C NMR (CDCl₃, 100 MHz): δ 14.4, 30.7, 109.1, 116.0, 118.4, 140.3, 142.3, 143.9, 156.2. IR: 1615, 1508, 1440, 1304, 1259, 1061, 878, 816, 738 cm⁻¹. HRMS *m/z*: [M+H]⁺ calculated for C₉H₁₀N₃O₂, 192.0773; found: 192.0773. HPLC (S1: >98%, *t*_R = 2.16 min; S2: >98%, *t*_R = 2.80 min).

2-Methyl-5-nitro-1-phenylbenzimidazole (Table 3, Entry 2)

R_f = 0.47 (80% EtOAc:*c*-hex). mp 172-174°C, lit.¹ 166-168°C. ¹H NMR (CDCl₃, 400 MHz): δ 2.55 (3H, s), 7.16 (1H, d, *J* = 8.9), 7.38 (2H, d, *J* = 6.9), 7.58-7.67 (3H, m), 7.13

¹ Joseph, L.; Julca, J. *J. Org. Chem.* **1962**, 27, 1101-1102.

(1H, dd, $J = 2.2, 8.9$), 8.63 (1H, d, $J = 2.2$). ^{13}C NMR (CDCl_3 , 100 MHz): δ 15.0, 110.2, 116.0, 119.0, 127.3, 130.1, 130.7, 135.3, 140.9, 142.4, 144.2, 155.9. IR: 1505, 1484, 1453, 1338, 1299, 1236, 1121, 1061, 1011 cm^{-1} . HRMS m/z : $[\text{M}+\text{H}]^+$ calculated for $\text{C}_{14}\text{H}_{12}\text{N}_3\text{O}_2$, 254.0930; found: 254.0929. HPLC (S1: >98%, $t_R = 6.92$ min; S2: >98%, $t_R = 8.33$ min).

1-Isopropyl-2-methyl-5-nitrobenzimidazole (Table 3, Entry 3)

$R_f = 0.33$ (90% EtOAc:*c*-hex). mp 223-224°C, lit.¹ 225-227°C. ^1H NMR (CDCl_3 , 400 MHz): δ 1.67 (6H, d, $J = 7.0$), 2.68 (3H, s), 4.72 (1H, heptet, $J = 7.0$), 7.52 (1H, d, $J = 9.0$), 8.15 (1H, dd, $J = 2.2, 9.0$), 8.58 (1H, d, $J = 2.2$). ^{13}C NMR (CDCl_3 , 100 MHz): δ 15.5, 21.8, 49.0, 111.0, 116.0, 118.0, 138.3, 143.0, 143.5, 155.3. IR: 1613, 1513, 1472, 1457, 1434, 1391, 1361, 1340, 1303, 1147, 1088, 882, 829, 743 cm^{-1} . HRMS m/z : $[\text{M}+\text{H}]^+$ calculated for $\text{C}_{11}\text{H}_{14}\text{N}_3\text{O}_2$, 220.1086; found: 220.1089. HPLC (S1: >98%, $t_R = 4.03$ min; S2: >98%, $t_R = 5.38$ min).

1,2-Dimethylbenzimidazole (Table 3, Entry 4)

$R_f = 0.39$ (10% MeOH:EtOAc). mp 106-109°C. ^1H NMR (CDCl_3 , 400 MHz): δ 2.54 (3H, s), 3.74 (3H, s), 7.15 (1H, dt, $J = 1.2, 7.3$), 7.20 (1H, dt, $J = 1.2, 7.3$), 7.47 (1H, d, $J = 7.3$), 7.52 (1H, d, $J = 7.3$). ^{13}C NMR ($\text{DMSO}-d_6$, 100 MHz): δ 13.9, 30.1, 110.1, 118.5, 121.6, 121.8, 136.3, 142.7, 152.6. IR: 1513, 1480, 1446, 1429, 1401, 1329, 1287, 1240, 1124, 1002 cm^{-1} . HRMS m/z : $[\text{M}+\text{H}]^+$ calculated for $\text{C}_9\text{H}_{11}\text{N}_2$, 147.0922; found: 147.0923. HPLC (S1: >98%, $t_R = 0.96$ min; S2: >98%, $t_R = 3.25$ min).

2-Methyl-1-phenylbenzimidazole (Table 3, Entry 5)

$R_f = 0.21$ (50% EtOAc:*c*-hex). mp 49-51°C. ^1H NMR (CDCl_3 , 400 MHz): δ 2.51 (3H, s), 7.12 (1H, d, $J = 7.9$), 7.19 (1H, dt, $J = 1.1, 7.1$), 7.27 (1H, dt, $J = 1.1, 7.1$), 7.37 (2H, m), 7.52 (1H, tt, $J = 1.3, 7.3$), 7.58 (2H, tt, $J = 1.3, 7.3$), 7.74 (1H, d, $J = 7.9$). ^{13}C NMR (CDCl_3 , 100 MHz): δ 14.8, 110.3, 119.4, 122.8, 123.0, 127.5, 129.2, 130.3, 136.5, 136.9, 143.0, 151.9. IR: 1596, 1498, 1455, 1390, 1370, 1324, 1285, 1245, 1015 cm^{-1} . HRMS m/z : $[\text{M}+\text{H}]^+$ calculated for $\text{C}_{14}\text{H}_{13}\text{N}_2$, 209.1079; found: 209.1078. HPLC (S1: >98%, $t_R = 3.08$ min; S2: >98%, $t_R = 6.76$ min).

1-Methyl-2-phenylbenzimidazole (Table 3, Entry 6)

R_f = 0.40 (50% EtOAc:*c*-hex). mp 92-95°C. ^1H NMR (CDCl_3 , 400 MHz): δ 3.88 (3H, s), 7.29-7.35 (2H, m), 7.41 (1H, m), 7.50-7.57 (3H, m), 7.75-7.79 (2H, m), 7.83 (1H, m). ^{13}C NMR (CDCl_3 , 100 MHz): δ 31.6, 109.6, 119.9, 122.4, 122.8, 128.7, 129.4, 129.7, 130.3, 136.6, 143.0, 153.8. IR: 1467, 1440, 1381, 1328, 1275, 1240, 1154, 1127, 1056, 1002, 925 cm^{-1} . HRMS m/z : $[\text{M}+\text{H}]^+$ calculated for $\text{C}_{14}\text{H}_{13}\text{N}_2$, 209.1079; found: 209.1079. HPLC (S1: >98%, t_R = 2.61 min; S2: >98%, t_R = 6.30 min).

1,2-Dimethyl-5-methoxybenzimidazole (Table 3, Entry 7)

R_f = 0.30 (10% MeOH:EtOAc). mp 130-132°C. ^1H NMR (CDCl_3 , 400 MHz): δ 2.58 (3H, s), 3.69 (3H, s), 3.85 (3H, s), 6.89 (1H, dd, J = 2.4, 8.7), 7.15 (1H, d, J = 8.7), 7.19 (1H, d, J = 2.4). ^{13}C NMR (CDCl_3 , 100 MHz): δ 14.2, 30.3, 56.2, 102.1, 109.5, 111.9, 130.8, 143.6, 152.4, 156.3. IR: 1509, 1488, 1436, 1394, 1338, 1273, 1195, 1151, 1133, 1097, 1026 cm^{-1} . HRMS m/z : $[\text{M}+\text{H}]^+$ calculated for $\text{C}_{10}\text{H}_{13}\text{N}_2\text{O}$, 177.1028; found: 177.1025. HPLC (S1: >98%, t_R = 1.41 min; S2: >98%, t_R = 4.31 min).

5-Methoxy-2-methyl-1-phenylbenzimidazole (Table 3, Entry 8)

R_f = 0.37 (80% EtOAc:*c*-hex). mp 91-94°C. ^1H NMR (CDCl_3 , 400 MHz): δ 2.49 (3H, s), 3.87 (3H, s), 6.84 (1H, dd, J = 2.4, 8.8), 7.01 (1H, d, J = 8.8), 7.24 (1H, d, J = 2.4), 7.36 (2H, m), 7.50 (1H, tt, J = 2.1, 7.3), 7.57 (2H, m). ^{13}C NMR (CDCl_3 , 100 MHz): δ 14.8, 56.3, 102.1, 110.6, 112.4, 127.3, 129.0, 130.2, 131.5, 136.6, 143.8, 152.1, 156.7. IR: 1594, 1500, 1481, 1434, 1392, 1336, 1272, 1195, 1148, 1111, 1027, 1010, 938 cm^{-1} . HRMS m/z : $[\text{M}+\text{H}]^+$ calculated for $\text{C}_{15}\text{H}_{15}\text{N}_2\text{O}$, 239.1184; found: 239.1183. HPLC (S1: >98%, t_R = 3.74 min; S2: >98%, t_R = 7.22 min).

1,2,7-Trimethylbenzimidazole (Table 3, Entry 9)

R_f = 0.40 (10% MeOH:EtOAc). mp 149-151°C. ^1H NMR (CDCl_3 , 400 MHz): δ 2.57 (3H, s), 2.73 (3H, s), 3.94 (3H, s), 6.93 (1H, d, J = 7.3), 7.07 (1H, t, J = 7.3), 7.51 (1H, d, J = 7.3). ^{13}C NMR (CDCl_3 , 100 MHz): δ 14.5, 19.0, 32.8, 117.4, 121.0, 122.1, 125.0, 134.6, 143.1, 152.4. IR: 1519, 1454, 1432, 1396, 1314, 1280, 1080, 785, 741 cm^{-1} . HRMS m/z :

$[M+H]^+$ calculated for $C_{10}H_{13}N_2$, 161.1079; found: 161.1079. HPLC (S1: >98%, t_R = 1.49 min; S2: >98%, t_R = 4.77 min).

2,7-Dimethyl-1-phenylbenzimidazole (Table 3, Entry 10)

R_f = 0.47 (80% EtOAc:*c*-hex). mp 112-117°C. 1H NMR ($CDCl_3$, 400 MHz): δ 1.90 (3H, s), 2.36 (3H, s), 6.92 (1H, d, J = 7.8), 7.15 (1H, t, J = 7.8), 7.33-7.38 (2H, m), 7.50-7.56 (3H, m), 7.60 (1H, d, J = 7.8). ^{13}C NMR ($CDCl_3$, 100 MHz): δ 14.3, 17.8, 117.0, 121.2, 122.1, 124.8, 128.8, 129.3, 134.9, 137.9, 142.9, 152.2. IR: 1594, 1523, 1496, 1452, 1414, 1383, 1310, 1270, 1201, 1008 cm^{-1} . HRMS m/z : $[M+H]^+$ calculated for $C_{15}H_{15}N_2$, 223.1235; found: 223.1236. HPLC (S1: >98%, t_R = 3.83 min; S2: >98%, t_R = 7.23 min).

Current Data Parameters
NAME E14575-29B
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20021126
Time 19.37
INSTRUM dpx400
PROBHD 5mm SEI 1H/13C
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 456.1
DM 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 6.90 usec
PL1 1.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300091 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 30.00 cm
F1P 11.000 ppm
F1 4401.43 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCM 0.40000 ppm/cm
HZCM 160.05200 Hz/cm

0.0700
0.0080
0.0000

1.6017

2.6647

3.8008

7.2615
7.3197
7.3319

8.1879
8.1932
8.2100
8.2153
8.5786
8.5840

144.414

wdw

14.414

Current Data Parameters
NAME E14537-938
EXPNO 5
PROCNO 1

F2 - Acquisition Parameters
Date_ 20020916
Time 23.47
INSTRUM dpx400
PROBHD 5mm SEI 1H/13C
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 24875.621 Hz
FIDRES 0.379572 Hz
AQ 1.3173236 sec
RG 4597.6
DW 20.100 usec
DE 6.00 usec
TE 300.0 K
D1 2.0000000 sec
D11 0.0300000 sec
D12 0.0002000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 12.80 usec
PL1 -6.00 dB
SF01 100.6237959 MHz

===== CHANNEL f2 =====
CPOPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.00 dB
PL12 24.00 dB
PL13 28.00 dB
SF02 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127290 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 215.000 ppm
F1 21631.74 Hz
F2P -5.000 ppm
F2 -503.06 Hz
PPMCM 11.00000 ppm/cm
HZCM 1106.73999 Hz/cm

Current Data Parameters
 NAME E13808-21B
 EXPNO 2
 PROCNO 1
 F2 - Acquisition Parameters
 Date_ 20030611
 Time 17.59
 INSTRUM dpx400
 PROBRD 5mm SEI 1H/13C
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQC 3.9584243 sec
 RG 645.1
 DM 60.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec
 ===== CHANNEL f1 =====
 NUC1 1H
 P1 6.90 usec
 PL1 1.00 dB
 SFO1 400.1324710 MHz
 F2 - Processing parameters
 SI 32768
 SF 400.1300078 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00
 ID NMR plot parameters
 CX 30.00 cm
 FL1 11.070 ppm
 F1 4429.48 Hz
 F2P -0.970 ppm
 F2 -387.97 Hz
 PPMCH 0.40132 ppm/cm
 HZCM 160.58162 Hz/cm

Current Data Parameters
NAME E1380B-218
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030612
Time 22.19
INSTRUM dpx400
PROBHD 5mm SEI 1H/13C
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 1024
DS 4
SWH 24875.621 Hz
FIDRES 0.379572 Hz
AQ 1.3173235 sec
RG 4096
DM 20.100 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
D12 0.00002000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 14.90 usec
PL1 -6.00 dB
SF01 100.6237959 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.00 dB
PL12 24.00 dB
PL13 28.00 dB
SF02 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127290 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 215.000 ppm
F1 21631.74 Hz
F2P -5.000 ppm
F2 -503.06 Hz
PPMCM 11.00000 ppm/cm
HZCM 1106.73999 Hz/cm

Current Data Parameters
 NAME E14575-35A
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20021127
 Time_ 18.41
 INSTRUM dpx400
 PROHD 5mm SEI 1H/13C
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.9584243 sec
 RG 362
 DW 60.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 6.90 usec
 PL1 1.00 dB
 SFO1 400.1324710 MHz

F2 - Processing parameters
 SI 32768
 SF 400.1300091 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 30.00 cm
 F1P 11.000 ppm
 F1 4401.43 Hz
 F2P -1.000 ppm
 F2 -400.13 Hz
 PPMCM 0.40000 ppm/cm
 HZCM 160.05200 Hz/cm

Current Data Parameters
NAME E14537-698
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20020828
Time 20.34
INSTRUM dpx400
PROBHD 5mm SEI 1H/13C
PULPROG zgpg30
TO 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 24875.621 Hz
FIDRES 0.379572 Hz
AQ 1.3173236 sec
RG 2560.3
DM 20.100 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
D12 0.00020000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 12.80 usec
PL1 -6.00 dB
SF01 100.6237959 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.00 dB
PL12 24.00 dB
PL13 28.00 dB
SF02 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127290 MHz
WDM EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 215.000 ppm
F1 21631.74 Hz
F2P -5.000 ppm
F2 -503.06 Hz
PPMCM 11.00000 ppm/cm
HZCM 1106.73999 Hz/cm

1.6729

Current Data Parameters
 NAME E14575-27B
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20021122
 Time 18.35
 INSTRUM dpx400
 PROHD 5mm SEI 1H/13C
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.9584243 sec
 RG 645.1
 DM 60.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 6.90 usec
 PL1 1.00 dB
 SFO1 400.1324710 MHz

F2 - Processing parameters
 SI 32768
 SF 400.1300098 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 30.00 cm
 F1P 11.000 ppm
 F1 4401.43 Hz
 F2P -1.000 ppm
 F2 -400.13 Hz
 PPMCM 0.40000 ppm/cm
 HZCM 160.05200 Hz/cm

Current Data Parameters
 NAME EL4575-37B
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20021128
 Time 19.33
 INSTRUM dpx400
 PROBD 5mm SEI 1H/13C
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.9584243 sec
 RG 80.6
 DW 60.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 6.90 usec
 PL1 1.00 dB
 SF01 400.1324710 MHz

F2 - Processing parameters
 SI 32768
 SF 400.1300095 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 30.00 cm
 FIP 11.000 ppm
 F1 4401.43 Hz
 F2p -1.000 ppm
 F2 -400.13 Hz
 PPMCM 0.40000 ppm/cm
 HZCM 160.05200 Hz/cm

Current Data Parameters
 NAME E14575-7B
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20021104
 Time 12.02
 INSTRUM dpx400
 PROBD 5mm SEI 1H/13C
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQC 3.9584243 sec
 RG 228.1
 DW 60.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 6.90 usec
 PL1 1.00 dB
 SF01 400.1324710 MHz

F2 - Processing parameters
 SI 32768
 SF 400.1300106 MHz
 WDW EM
 SS 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 30.00 cm
 FL1 11.025 ppm
 F1 4411.56 Hz
 F2P -1.004 ppm
 F2 -401.73 Hz
 PPM0X 0.40098 ppm/cm
 HZCM 160.44304 Hz/cm

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3.87639

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 7.52087
 7.51850
 7.39586
 7.39321
 7.33716
 7.33206
 7.32276
 7.31364
 7.25752

Current Data Parameters
NAME E14575-7B
EXPNO 7
PROCNO 1

F2 - Acquisition Parameters
Date_ 20021105
Time 0.41
INSTRUM dpx400
PROBHD 5mm SEI 1H/13C
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 24875.621 Hz
FIDRES 0.379572 Hz
AQ 1.3173236 sec
RG 2580.3
DM 20.100 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
D12 0.0002000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 12.80 usec
PL1 -6.00 dB
SF01 100.6237959 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.00 dB
PL12 24.00 dB
PL13 26.00 dB
SF02 400.1315005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127689 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
FIP 215.000 ppm
F1 21631.75 Hz
F2P -5.000 ppm
F2 -503.06 Hz
PPMCM 11.00000 ppm/cm
HZCM 1106.74048 Hz/cm

Current Data Parameters
 NAME E14575-119B
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20030522
 Time 12.39
 INSTRUM dxt400
 PROBED 5mm SEI IH/13C
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.9584243 sec
 RG 228.1
 DW 60.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 6.90 usec
 PL1 1.00 dB
 SFO1 400.1324710 MHz

F2 - Processing parameters
 SI 32768
 SF 400.1300084 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 30.00 cm
 F1P 11.000 ppm
 F1 4401.43 Hz
 F2P -1.000 ppm
 F2 -400.13 Hz
 PPMCM 0.40000 ppm/cm
 HZCM 160.05200 Hz/cm

Current Data Parameters
 NAME E13808-20B
 EXPNO 1
 PROCNO 1
 F2 - Acquisition Parameters
 Date_ 20030611
 Time 13.19
 INSTRUM dpx400
 PROBD 5mm SEI IH/13C
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.9584243 sec
 RG 1625.5
 DW 60.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec
 ===== CHANNEL f1 =====
 NUC1 1H
 P1 6.90 usec
 PL1 1.00 dB
 SF01 400.1324710 MHz
 F2 - Processing parameters
 SI 32768
 SF 400.1300093 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00
 ID NMR plot parameters
 CX 30.00 cm
 FLIP 10.993 ppm
 F1 4398.49 Hz
 F2P -1.022 ppm
 F2 -409.12 Hz
 PPMCM 0.40050 ppm/cm
 HZCM 160.25389 Hz/cm

Current Data Parameters
NAME E1380B-20B
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030612
Time 5.52
INSTRUM dpx400
PROBHD 5mm SEI 1H/13C
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 24875.621 Hz
FIDRES 0.379572 Hz
AQ 1.3173236 sec
RG 3251
DM 20.100 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
D12 0.00002000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 14.90 usec
PL1 -6.00 dB
SF01 100.6237959 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.00 dB
PL12 24.00 dB
PL13 28.00 dB
SF02 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127290 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 215.000 ppm
F1 21631.74 Hz
F2P -5.000 ppm
F2 -503.06 Hz
PPMCM 11.00000 ppm/cm
HZCM 1106.73999 Hz/cm

Current Data Parameters
 NAME E14575-32B
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20021127
 Time 19.38
 INSTRUM dpx400
 PROBED 5mm SEI 1H/13C
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.958423 sec
 RG 574.7
 DW 60.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 6.90 usec
 PL1 1.00 dB
 SFO1 400.1324710 MHz

F2 - Processing parameters
 SI 32768
 SF 400.1300100 MHz
 WDW EM
 SS 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 30.00 cm
 F1P 11.000 ppm
 F1 4401.43 Hz
 F2P -1.000 ppm
 F2 -400.13 Hz
 PPMCM 0.40000 ppm/cm
 HZCM 160.05200 Hz/cm

0.0095
 0.0081
 0.0000
 -0.0082

1.6129

2.7266
 2.5825
 2.5730

3.9523
 3.9620

7.5170
 7.4960
 7.2690
 7.2591
 7.0927
 7.0739
 7.0544
 6.9340
 6.9158

Current Data Parameters
NAME E14537-140B
EXPNO 7
PROCNO 1

F2 - Acquisition Parameters
Date_ 20021021
Time 23.08
INSTRUM dpx400
PROBHD 5mm SEI 1H/13C
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 24875.621 Hz
FIDRES 0.379572 Hz
AQ 1.3173236 sec
RG 6502
DM 20.100 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
D12 0.0002000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 12.80 usec
PL1 -6.00 dB
SF01 100.6237959 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.00 dB
PL12 24.00 dB
PL13 28.00 dB
SF02 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127290 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

10 NMR plot parameters
CX 20.00 cm
F1P 215.000 ppm
F1 21631.74 Hz
F2P -5.000 ppm
F2 -503.06 Hz
PPMCM 11.00000 ppm/cm
HZCM 1106.73999 Hz/cm

Current Data Parameters
 NAME E14575-34B
 EXPNO 1
 PROCNO 1
 F2 - Acquisition Parameters
 Date_ 20021127
 Time 19.53
 INSTRUM dpx400
 PROBD 5mm SEI IH/13C
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.9584243 sec
 RG 362
 DW 60.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 6.30 usec
 PL1 1.00 dB
 SF01 400.1324710 MHz
 F2 - Processing parameters
 SI 32768
 SF 400.1300103 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00
 1D NMR plot parameters
 CX 30.00 cm
 F1P 11.000 ppm
 F1 4401.43 Hz
 F2P -1.000 ppm
 F2 -400.13 Hz
 PPMCM 0.40000 ppm/cm
 HZCM 160.05200 Hz/cm

Current Data Parameters	
NAME	E14575-34B
EXPNO	2
PROCNO	1
F2 - Acquisition Parameters	
Date_	20030523
Time	4.54
INSTRUM	gdx400
PROBHD	5mm SEI 1H/13C
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	8192
DS	4
SWH	24875.621 Hz
FIDRES	0.379572 Hz
AQ	1.3173236 sec
RG	5160.6
DW	20.100 usec
DE	6.00 usec
TE	300.0 K
D1	2.00000000 sec
D11	0.03000000 sec
D12	0.00002000 sec
===== CHANNEL f1 =====	
NUC1	13C
P1	14.90 usec
PL1	-6.00 dB
SFO1	100.6237959 MHz
===== CHANNEL f2 =====	
CPDPRG2	waltz16
NUC2	1H
PCPD2	100.00 usec
PL2	1.00 dB
PL12	24.00 dB
PL13	28.00 dB
SFO2	400.1316003 MHz
F2 - Processing parameters	
SI	32768
SF	100.6127675 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40
10 NMR plot parameters	
CX	20.00 cm
F1P	215.000 ppm
F1	21631.75 Hz
F2P	-5.000 ppm
F2	-503.06 Hz
PPM0	11.00000 ppm/cm
HZCM	1106.74048 Hz/cm