Lewis Acid-Mediated Reactions of Alkyl Azides with α,β -Unsaturated Ketones

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Supporting Information

Experimental Section

^1H and ^{13}C -NMR Spectra of new compounds

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Alkyl azides were prepared from the corresponding bromides using NaN_3 . All the enones were purchased from Aldrich except one bicyclic enone¹ (entry 4, Table 2), which was prepared following literature precedent. Compounds **9**², **11**³, **15**³, **19**⁴, **20**⁴, and **21**^{5, 6} are known in the literature.

General Procedure. To an ice-cooled solution of enone (1 equiv) and alkyl azide (1.5 equiv) in CH_2Cl_2 was added TMSOTf (2 equiv) dropwise and the reaction mixture was allowed to warm to room temperature. After 10-12 h, the reaction was quenched with aq NaHCO_3 and extracted with CH_2Cl_2 . The combined organic layers were washed with water, brine, and dried (Na_2SO_4). The residue obtained after the evaporation of solvent was subjected to flash chromatography (1:1 EtOAc:hexane to EtOAc) to furnish product(s). Spectral data for all new compounds is given below.

1

2-(Benzylaminomethylene)cyclopentanone (1). Yield 68%; IR (neat) 3262, 1667, 1574 cm^{-1} ; MS (CI) m/e 202 ($\text{M}^+ + 1$), 91; HRMS calcd for $\text{C}_{13}\text{H}_{16}\text{NO}$: ($\text{M}^+ + 1$) 202.1232, found 202.1221; *Z* isomer: ^1H NMR (400 MHz, CDCl_3) δ 9.27 (br s, 1H), 7.40-7.25 (m, 5H), 6.68 (d, $J = 12.5$ Hz, 1H), 4.36 (d, $J = 6.1$ Hz, 2H), 2.53 (t, $J = 6.9$ Hz, 2H), 2.31 (m, 2H), 1.97-1.87 (m, 2H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 206.1, 147.3, 138.8, 129.3, 128.0, 127.6, 104.5, 52.7, 39.7, 27.9, 22.4. *E* isomer (diagnostic peaks only): ^1H NMR (400 MHz, CDCl_3) δ 4.71 (br s, 1H), 4.41 (d, $J = 5.9$ Hz, 2H), 2.44 (t, $J = 6.8$ Hz, 2H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 205.4, 142.6, 138.5, 107.1, 39.1, 25.8, 20.4.

2

2-(Phenethylaminomethylene)cyclopentanone (2). Yield 93%; IR (neat) 3263, 1667, 1574 cm^{-1} ; MS (CI) m/e 216 ($M^+ + 1$), 91; HRMS calcd for $\text{C}_{14}\text{H}_{18}\text{NO}$: ($M^+ + 1$) 216.1388, found 216.1385; *Z*-isomer: ^1H NMR (500 MHz, CDCl_3) δ 9.04 (br s, 1H), 7.35-7.18 (m, 5H), 6.52 (d, $J = 12.4$ Hz, 1H), 3.41 (q, $J = 6.9$ Hz, 2H), 2.85 (m, 2H), 2.47 (t, $J = 7.1$ Hz, 2H), 2.30 (m, 2H), 1.95-1.86 (m, 2H); ^{13}C NMR (125.75 MHz, CDCl_3) δ 205.1, 147.0, 138.1, 128.8, 128.7, 128.5, 103.3, 50.4, 39.1, 38.0, 27.4, 21.4. *E* isomer (diagnostic peaks only): ^1H NMR (500 MHz, CDCl_3) δ 4.50 (br s, 1H), 4.48 (q, $J = 6.7$ Hz, 2H), 2.91 (t, $J = 7.0$ Hz, 2H); ^{13}C NMR (125.75 MHz, CDCl_3) δ 204.7, 142.3, 137.8, 106.5.

3

2-(Hexylaminomethylene)cyclopentanone (3). Yield 83%; IR (neat) 3267, 1667, 1594 cm^{-1} ; MS (CI) m/e 196 ($M^+ + 1$), 124; HRMS calcd for $\text{C}_{12}\text{H}_{22}\text{NO}$: ($M^+ + 1$) 196.1701, found 196.1711; *Z* isomer: ^1H NMR (400 MHz, CDCl_3) δ 9.00 (br s, 1H), 6.62 (d, $J = 12.6$ Hz, 1H), 3.13 (q, $J = 6.6$ Hz, 2H), 2.48 (t, $J = 6.9$ Hz, 2H), 2.25 (m, 2H), 1.88 (m, 2H), 1.52 (m, 2H), 1.29 (m, 6H), 0.87 (t, $J = 6.0$ Hz, 3H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 205.2, 147.9, 103.2, 49.3, 39.5, 31.7, 31.6, 27.8, 26.5, 22.8, 22.3, 14.3. *E* isomer (diagnostic peaks only): ^1H NMR (400 MHz, CDCl_3) δ 7.25 (d, $J = 13.9$ Hz, 1H), 4.71 (br s, 1H), 4.41 (d, $J = 5.9$ Hz, 2H), 2.44 (t, $J = 6.8$ Hz, 2H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 204.9, 143.3, 138.5, 106.1, 48.8.

4

2-(3-Phenyl-2-propenylmethylene)cyclopentanone (4). Yield 76%; IR (neat) 3255, 1668, 1568 cm^{-1} ; MS (EI) m/e 227 (M^+), 117, 91; HRMS calcd for $\text{C}_{15}\text{H}_{18}\text{NO}$: ($M^+ + 1$) 228.1388, found 228.1389; *Z* isomer: ^1H NMR (400 MHz, CDCl_3) δ 9.47 (br s, 1H), 7.38-7.24 (m, 5H), 6.67 (d, $J = 12.4$ Hz, 1H), 6.53 (d, $J = 15.8$ Hz, 1H), 6.22 (m, 1H), 3.94 (t, $J = 5.8$ Hz, 2H), 2.53 (t, $J = 7.0$ Hz, 2H), 2.32 (m, 2H), 1.95-1.88 (m, 2H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 206.0, 147.1, 136.7, 132.5, 129.0, 128.2, 126.9, 126.6, 104.5, 50.7, 39.7, 27.9, 22.4. *E* isomer (diagnostic peaks only): ^1H NMR (400 MHz, CDCl_3) δ 6.53 (d, $J = 15.8$ Hz, 1H), 6.18 (m, 1H), 4.67 (br s, 1H), 3.99 (t, $J = 6.0$ Hz, 2H), 2.45 (t, $J = 7.0$ Hz, 2H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 205.3, 142.6, 136.5, 107.2.

5

3-[(2-Oxocyclopentylidenemethyl)amino]propionic acid ethyl ester (5). Yield 69%; IR (neat) 3258, 1728, 1668, 1567 cm^{-1} ; MS (CI) m/e 212 ($M^+ + 1$), 124; HRMS calcd for $\text{C}_{11}\text{H}_{18}\text{NO}_3$: ($M^+ + 1$) 212.1287, found 212.1290; *Z* isomer: ^1H NMR (500 MHz, CDCl_3) δ 8.99 (br s, 1H), 6.62 (d, $J = 12.3$ Hz, 1H), 4.15 (m, 2H), 3.46 (m, 2H), 2.57-1.82 (series of m, 8H), 1.25 (t, $J = 7.1$ Hz, 3H); ^{13}C NMR (125.75 MHz, CDCl_3) δ 205.3, 171.0, 146.9, 103.7, 60.8, 44.3, 39.1, 36.0, 27.3, 21.8, 14.1. *E* isomer (diagnostic peaks only): ^1H NMR (500 MHz, CDCl_3) δ 7.21 (d, $J = 13.7$ Hz, 2H), 4.96 (br s, 1H); ^{13}C NMR (125.75 MHz, CDCl_3) δ 204.7, 171.7, 141.9, 138.5, 106.6.

6

2-[(4-Methoxybenzylamino)methylene]cyclopentanone (6). Yield 62%; IR (neat) 3265, 1667, 1574 cm^{-1} ; MS (CI) m/e 232 ($M^+ + 1$), 121; HRMS calcd for $\text{C}_{14}\text{H}_{18}\text{NO}_2$: ($M^+ + 1$) 232.1338, found 232.1333; *Z* isomer: ^1H NMR (400 MHz, CDCl_3) δ 9.22 (br s, 1H), 7.11 (d, $J = 8.5$ Hz, 2H), 6.82 (d, $J = 8.5$ Hz, 2H), 6.63 (d, $J = 12.5$ Hz, 1H), 4.23 (t, $J = 5.9$ Hz, 2H), 3.73 (s, 3H), 2.46 (t, $J = 7.0$ Hz, 2H), 2.21 (m, 2H), 1.86-1.79 (m, 2H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 205.7, 159.5, 147.3, 130.7, 129.1, 128.9, 114.5, 104.2, 52.1, 39.6, 27.9, 22.3. *E* isomer (diagnostic peaks only): ^1H NMR (400 MHz, CDCl_3) δ 7.28 (d, $J = 13.8$ Hz, 1H), 5.43 (br s, 1H), 4.23 (t, $J = 5.9$ Hz, 2H), 3.73 (s, 3H), 2.37 (t, $J = 6.8$ Hz, 2H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 205.1, 143.1, 106.7.

7

2-[(3-Chloropropylamino)methylene]cyclopentanone (7). Yield 77%; IR (neat) 3258, 1670, 1573 cm^{-1} ; MS (CI) m/e 212 ($M^+ + 1$), 188, 124, 96; HRMS calcd for $\text{C}_9\text{H}_{15}\text{NOCl}$: ($M^+ + 1$) 188.0842, found 188.0836; *Z* isomer: ^1H NMR (400 MHz, CDCl_3) δ 8.94 (br s, 1H), 6.65 (d, $J = 12.4$ Hz, 1H), 3.61 (t, $J = 6.0$ Hz, 2H), 3.37 (q, $J = 6.4$ Hz, 2H), 2.51 (t, $J = 7.1$ Hz, 2H), 2.30 (t, $J = 7.8$ Hz, 2H), 2.01 (quintet, $J = 6.0$ Hz, 2H), 1.89 (quintet, $J = 7.5$ Hz, 2H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 206.0, 147.7, 104.2, 45.8, 42.0, 39.7, 34.0, 27.9, 22.4. *E* isomer (diagnostic peaks only): ^1H NMR (400 MHz, CDCl_3) δ 7.25 (d, $J = 13.8$ Hz, 1H), 4.50 (br s, 1H), 3.61 (t, $J =$

6.3 Hz, 2H), 2.42 (t, $J = 7.7$ Hz, 2H), 2.30 (t, $J = 7.9$ Hz, 2H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 205.3, 142.5, 107.2, 46.9, 45.5, 39.1, 30.6, 25.7, 20.4.

2-(Cyclohexylaminomethylene)cyclopentanone (8). Yield 20%; IR (neat) 3262, 1664, 1578 cm^{-1} ; MS (EI) m/e 194 (M^+), 150, 55; HRMS calcd for $\text{C}_{12}\text{H}_{20}\text{NO}$: (M^++1) 194.1545, found 194.1537; *Z* isomer: ^1H NMR (500 MHz, CDCl_3) δ 9.08 (br s, 1H), 6.71 (d, $J = 12.7$ Hz, 1H), 3.01 (m, 1H), 2.50 (t, $J = 7.0$ Hz, 2H), 2.29 (t, $J = 7.8$ Hz, 2H), 1.96-1.85 (m, 6H), 1.39-1.24 (m, 6H); ^{13}C NMR (125.75 MHz, CDCl_3) δ 204.5, 145.4, 102.6, 56.4, 39.2, 34.1, 27.5, 25.2, 21.9, 19.4. *E* isomer (diagnostic peaks only): ^1H NMR (500 MHz, CDCl_3) δ 7.37 (d, $J = 14.1$ Hz, 1H), 4.42 (br s, 1H), 3.11 (m, 1H), 2.40 (t, $J = 6.6$ Hz, 2H), 2.29 (t, $J = 7.8$ Hz, 2H); ^{13}C NMR (125.75 MHz, CDCl_3) δ 204.5, 140.9, 105.8, 56.5, 38.6, 34.3, 25.3, 25.1, 24.6.

6-Benzyl-6-aza-bicyclo[3.1.0]hexan-2-one (10). Yield 33%; IR (neat) 1739 cm^{-1} ; MS (EI) m/e 188 (M^+), 91; HRMS calcd for $\text{C}_{12}\text{H}_{14}\text{NO}$: (M^++1) 188.1075, found 188.1068; ^1H NMR (400 MHz, CDCl_3) δ 7.36-7.25 (m, 5H), 3.56 (AB q, $J = 13.7$ Hz, $\Delta\nu = 58.4$ Hz, 2H), 2.74 (s, 1H), 2.39-2.21 (m, 3H), 2.03-1.95 (m, 2H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 212.6, 138.5, 128.9, 128.0, 127.7, 61.9, 47.4, 46.6, 33.3, 24.5.

2-(Benzylaminomethylene)indan-1,3-dione (12). Yield 49%; mp 198-199 °C; IR (neat) 3272, 1639, 1592 cm^{-1} ; MS (EI) m/e 264 (M^+), 91; HRMS calcd for $\text{C}_{17}\text{H}_{14}\text{NO}_2$: (M^++1) 264.1025, found 264.1029; ^1H NMR (400 MHz, CDCl_3) δ 9.49 (br s, 1H), 7.82-7.28 (m, 10H), 4.61 (d, $J = 6.1\text{ Hz}$, 2H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 194.1, 190.5, 152.2, 140.2, 135.9, 133.8, 133.7, 129.6, 129.0, 128.0, 122.3, 121.7, 105.2, 54.2.

2-Benzyl-2H-benzo[c]azepine-1,5-dione (13). Yield 14%; mp 151-153 °C; IR (neat) 1681, 1593 cm^{-1} ; MS (EI) m/e 264 (M^+), 91; HRMS calcd for $\text{C}_{17}\text{H}_{14}\text{NO}_2$: (M^++1) 264.1025, found 264.1019; ^1H NMR (400 MHz, CDCl_3) δ 8.09 (dd, $J = 7.6, 14.7\text{ Hz}$, 2H), 7.74 (td, $J = 1.3, 7.5\text{ Hz}$, 1H), 7.64 (td, $J = 1.2, 7.6\text{ Hz}$, 1H) 7.42-7.28 (m, 5H), 6.24 (br s, 1H), 5.80 (s, 1H), 4.39 (d, $J = 5.7\text{ Hz}$, 2H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 183.5, 182.3, 148.1, 136.3, 135.2, 133.9, 132.5, 130.9, 129.4, 128.5, 128.1, 126.7, 126.6, 102.1, 47.2.

14

3-(Benzylaminomethylene)bicyclo[2.2.1]heptan-2-one (14). Yield 52%; IR (neat) 3294, 1682, 1614 cm^{-1} ; MS (EI) m/e 227 (M^+), 91; HRMS calcd for $\text{C}_{15}\text{H}_{18}\text{NO}$: (M^++1) 228.1388, found 228.1393; *Z*-isomer: ^1H NMR (400 MHz, CDCl_3) δ 7.98 (br s, 1H), 7.35-7.23 (m, 5H), 6.45 (d, $J = 12.5$ Hz, 1H), 4.28 (m, 2H), 2.88 (br s, 1H), 2.61 (m, 1H), 1.85-1.40 (m, 6H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 207.1, 142.7, 139.1, 129.1, 127.4, 127.5, 111.4, 52.4, 51.2, 42.2, 40.7, 30.5, 24.2. *E*-isomer (diagnostic peaks only): ^1H NMR (400 MHz, CDCl_3) δ 7.05 (d, $J = 13.2$ Hz, 1H), 5.02 (m, 1H), 4.32 (m, 1H), 3.07 (br s, 1H), 2.61 (m, 1H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 206.2, 138.4, 138.0, 113.2, 142.6, 136.5, 107.2.

16

1-[1-(Benzyliminomethyl)cyclopentyl]ethanone (16). (Selected peaks observed in the crude reaction mixture): ^1H NMR (500 MHz, CDCl_3) δ 7.74 (s, 1H), 4.63 (s, 1H), 2.14 (s, 3H); ^{13}C NMR (125.75 MHz, CDCl_3) δ 208.6, 167.1, 139.1.

1-Acetylcyclopentanecarbaldehyde (17). (Selected peaks observed in the crude reaction mixture): ^1H NMR (400 MHz, CDCl_3) δ 9.55 (s, 1H), 2.17 (s, 3H), 2.15 (s, 3H) ^{13}C NMR (125.75 MHz, CDCl_3) δ 200.0, 30.7, 27.8, 26.2.

2-(Benzyliminomethyl)-4-isopropenyl-2-methylcyclopentanone (18). Yield 74%; IR (neat) 1742, 1713, 1655 cm^{-1} ; MS (EI) m/e 256 ($\text{M}^+ + 1$), 174, 91; HRMS calcd for $\text{C}_{17}\text{H}_{22}\text{NO}$: ($\text{M}^+ + 1$) 256.1701, found 256.1710; Major isomer: ^1H NMR (400 MHz, CDCl_3) δ 7.60 (s, 1H), 7.43-7.24 (m, 5H), 4.79 (d, $J = 19.5$ Hz, 2H), 4.65 (AB q, $J = 14.0$ Hz, $\Delta\nu = 19.4$ Hz, 2H), 2.88 (m, 1H), 2.77 (m, 1H), 2.55 (ddd, $J = 2.2, 7.6, 14.0$ Hz, 1H), 2.20 (dd, $J = 11.6, 18.4$ Hz, 1H), 1.79 (s, 3H), 1.64 (m, 1H), 1.32 (s, 3H); ^{13}C NMR (100.6 MHz, CDCl_3) (diagnostic peaks only) δ 218.1, 198.4, 165.8, 109.9, 64.8, 43.2, 39.6, 21.4. Minor isomer (diagnostic peaks only): ^1H NMR (400 MHz, CDCl_3) δ 4.83 (d, $J = 24.7$ Hz, 2H), 1.84 (s, 3H), 1.38 (s, 3H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 110.5, 57.2, 43.4, 40.2, 36.6, 19.3.

22

5-Benzylamino-hex-4-en-3-one (22). Yield 49%; IR (neat) 1709, 1614 cm^{-1} ; MS (CI) m/e 204 (M^++1), 174, 91; HRMS calcd for $\text{C}_{13}\text{H}_{18}\text{NO}$: (M^++1) 204.1388, found 204.1399; ^1H NMR (400 MHz, CDCl_3) δ 7.36-7.26 (m, 5H), 5.05 (s, 1H), 4.45 (d, $J = 6.3$ Hz, 2H), 2.30 (q, $J = 7.6$ Hz, 2H), 1.93 (s, 3H), 1.10 (t, $J = 7.5$ Hz, 3H); ^{13}C NMR (100.6 MHz, CDCl_3) δ 199.5, 163.5, 138.4, 129.2, 127.8, 127.2, 95.2, 47.2, 35.2, 19.4, 10.4.

References:

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1

Current Data Parameters
NAME dsr124h
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20020123
Time 14.02
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 4
DS 2
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 114
DW 104.400 usec
DE 4.50 usec
TE 300.0 K
D1 1.00000000 sec

===== CHANNEL f1 =====
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PL1 -6.00 dB
SFO1 400.1320007 MHz

F2 - Processing parameters
SI 16384
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
FIP 10.000 ppm
F1 4001.30 Hz
F2P -0.500 ppm
F2 -200.07 Hz
PPMCM 0.52500 ppm/cm
HZCM 210.06825 Hz/cm

1

Current Data Parameters
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EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
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TD 65536
SOLVENT CDCl3
NS 212
DS 2
SWH 23148.148 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 4096
DW 21.600 usec
DE 4.50 usec
TE 300.0 K
D1 0.05000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

===== CHANNEL f1 =====
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P1 12.30 usec
PL1 2.00 dB
SFO1 100.6232933 MHz

===== CHANNEL f2 =====
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NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 18.00 dB
PL13 18.00 dB
SFO2 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
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F1 22134.80 Hz
F2P 0.000 ppm
F2 0.00 Hz
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HZCM 1106.73999 Hz/cm

2

Current Data Parameters
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EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
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Time 16.52
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 4
DS 2
SWH 6009.615 Hz
FIDRES 0.183399 Hz
AQ 2.7264309 sec
RG 101.6
OW 83.200 usec
DE 5.00 usec
TE 300.0 K
D1 0.03000000 sec

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F2 - Processing parameters
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SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

F1 - Processing parameters
SI 1024
MC2 OF
SF 500.1300000 MHz
WDW GSINE
SSB 0
LB 0.30 Hz
GB 0

1D NMR plot parameters
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CY 8.00 cm
FIP 10.000 ppm
F1 5001.30 Hz
F2P -0.500 ppm
F2 -250.07 Hz
PPMCH 0.52500 ppm/cm
HZCN 262.56824 Hz/cm

2

ent Data Parameters
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EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
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Time 16.55
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 222
DS 4
SWH 31446.541 Hz
FIDRES 0.479836 Hz
AQ 1.040083 sec
RG 2048
OW 15.900 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

***** CHANNEL f1 *****
NUC1 13C
P1 6.50 usec
PL1 5.00 dB
SF01 125.7719472 MHz

***** CHANNEL f2 *****
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NUC2 1H
PCPD02 95.00 usec
PL2 -4.00 dB
PL12 19.00 dB
PL13 36.00 dB
SF02 500.1325000 MHz

F2 - Processing parameters
SI 65536
SF 125.7578008 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

F1 - Processing parameters
SI 1024
MC2 OF
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WDW no
SSB 0
LB 0.30 Hz
GB 0

1D NMR plot parameters
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CY 8.00 cm
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F1 27666.71 Hz
F2P 0.500 ppm
F2 0.00 Hz
PPMCH 11.00000 ppm/cm
HZCN 1381.32582 Hz/cm

3

Current Data Parameters
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EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
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INSTRUM drx400
PROBHD 5 mm Multinucl
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 4
DS 2
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 64
DW 104.400 usec
DE 4.50 usec
TE 300.0 K
D1 1.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 7.70 usec
PL1 -6.00 dB
SFO1 400.132007 MHz

F2 - Processing parameters
SI 16384
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
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CY 8.00 cm
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F2P -0.500 ppm
F2 -200.07 Hz
PPMCM 0.52500 ppm/cm
HZCM 210.06825 Hz/cm

3

Current Data Parameters
NAME dsr-III-18
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
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PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 130
DS 2
SWH 23148.148 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 32768
DW 21.600 usec
DE 4.50 usec
TE 300.0 K
D1 0.05000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 12.30 usec
PL1 2.00 dB
SFO1 100.6232933 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
MUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 18.00 dB
PL13 18.00 dB
SFO2 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
CY 9.00 cm
F1P 220.000 ppm
F1 22134.80 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 11.00000 ppm/cm
HZCM 1106.73999 Hz/cm

4

Current Data Parameters
NAME dsr-III-17
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030721
Time 13.17
INSTRUM drx400
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 4
DS 2
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 64
DW 104.400 usec
DE 4.50 usec
TE 300.0 K
D1 1.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 7.70 usec
PL1 -6.00 dB
SFO1 400.1320007 MHz

F2 - Processing parameters
SI 16384
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 6.00 cm
FIP 11.000 ppm
F1 4401.43 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCM 0.60000 ppm/cm
HZCM 240.07800 Hz/cm

4

Current Data Parameters
NAME dsr-III-17
EXPNO 5
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030721
Time 13.21
INSTRUM drx400
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 230
DS 2
SWH 23148.148 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 32768
DW 21.600 usec
DE 4.50 usec
TE 300.0 K
D1 0.05000000 sec
d11 0.03000000 sec
d12 0.0002000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 12.30 usec
PL1 2.00 dB
SFO1 100.6232933 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 18.00 dB
PL13 18.00 dB
SFO2 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
CY 12.50 cm
FIP 220.000 ppm
F1 22134.80 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 11.00000 ppm/cm
HZCM 1106.73999 Hz/cm

5

Current Data Parameters
NAME dsr-II-2968
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030619
Time 17.03
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6009.615 Hz
FIDRES 0.183399 Hz
AQ 2.7264309 sec
RG 80.5
DW 83.200 usec
DE 6.00 usec
TE 300.0 K
D1 0.03000000 sec

***** CHANNEL f1 *****
NUC1 1H
P1 7.70 usec
PL1 -4.00 dB
SFO1 500.1320005 MHz

F2 - Processing parameters
SI 32768
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

F1 - Processing parameters
SI 1024
MC2 OF
SF 500.1300000 MHz
WDW OSLINE
SSB 0
LB 0.30 Hz
GB 0

1D NMR plot parameters
CX 20.00 cm
CY 8.00 cm
FIP 10.000 ppm
F1 5001.30 Hz
F2P -0.500 ppm
F2 -250.07 Hz
PPMCM 0.52500 ppm/cm
HZCM 262.56824 Hz/cm

5

Current Data Parameters
NAME dsr-II-2968
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030619
Time 17.08
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 282
DS 4
SWH 31446.541 Hz
FIDRES 0.479836 Hz
AQ 1.0420893 sec
RG 2048
DW 15.900 usec
DE 6.00 usec
TE 300.0 K
D1 0.18000000 sec
d11 0.03000000 sec
d12 0.00020000 sec

***** CHANNEL f1 *****
NUC1 13C
P1 6.50 usec
PL1 5.00 dB
SFO1 125.7719472 MHz

***** CHANNEL f2 *****
CPDPRG2 waltz16
NUC2 1H
POPRG2 95.00 usec
PL2 -4.00 dB
PL12 19.00 dB
PL13 30.00 dB
SFO2 500.1325000 MHz

F2 - Processing parameters
SI 65536
SF 125.7579808 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

F1 - Processing parameters
SI 1024
MC2 OF
SF 500.1300000 MHz
WDW no
SSB 0
LB 0.30 Hz
GB 0

1D NMR plot parameters
CX 20.00 cm
CY 8.00 cm
FIP 220.500 ppm
F1 27666.71 Hz
F2 0.000 ppm
F2P 11.00000 ppm/cm
PPMCM 1383.33582 Hz/cm

6

Current Data Parameters
NAME dsr-III-19
EXPNO 1
PROCNO 2

F2 - Acquisition Parameters
Date_ 20030721
Time 13.10
INSTRUM drx400
PROBHD 5 mm Multinucl
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 4
DS 2
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 35.9
DW 104.400 usec
DE 4.50 usec
TE 300.0 K
D1 1.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 7.70 usec
PL1 -6.00 dB
SFO1 400.1320007 MHz

F2 - Processing parameters
SI 16384
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 15.00 cm
FIP 10.000 ppm
P1 4001.30 Hz
F2P -0.500 ppm
F2 -200.07 Hz
PPMCH 0.52500 ppm/cm
HZCM 210.06825 Hz/cm

6

Current Data Parameters
NAME dsr-III-19
EXPNO 2
PROCNO 2

F2 - Acquisition Parameters
Date_ 20030721
Time 13.14
INSTRUM drx400
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 130
DS 2
SWH 23148.148 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 32768
DW 21.600 usec
DE 4.50 usec
TE 300.0 K
D1 0.05000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 12.30 usec
PL1 2.00 dB
SFO1 100.6232933 MHz

===== CHANNEL f2 =====
CPOPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 18.00 dB
PL13 18.00 dB
SFO2 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
CY 12.50 cm
FIP 220.000 ppm
P1 22134.80 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCH 11.00000 ppm/cm
HZCM 1106.73999 Hz/cm

7

Current Data Parameters
NAME wj4-63-2
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030801
Time 13.04
INSTRUM drx400
PROBHD 5 mm Multinucl
PULPROG zg30
TD 32768
SOLVENT CDC13
NS 16
DS 2
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 114
DW 104.400 usec
DE 4.50 usec
TE 300.0 K
D1 1.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 7.70 usec
PL1 -6.00 dB
SFO1 400.1320007 MHz

F2 - Processing parameters
SI 16384
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 0.00 cm
F1P 9.500 ppm
F1 3801.24 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPHMC 0.47500 ppm/cm
HZCM 190.06175 Hz/cm

7

Current Data Parameters
NAME wj4-63-2
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030801
Time 13.07
INSTRUM drx400
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 290
DS 2
SWH 23148.148 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 32768
DW 21.600 usec
DE 4.50 usec
TE 300.0 K
D1 0.05000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 12.30 usec
PL1 2.00 dB
SFO1 100.6232933 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 18.00 dB
PL13 18.00 dB
SFO2 400.1316005 MHz

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

1D NMR plot parameters
CX 20.00 cm
CY 0.00 cm
F1P 220.036 ppm
F1 22138.41 Hz
F2P -0.500 ppm
F2 -50.31 Hz
PPHMC 11.02680 ppm/cm
HZCM 1109.43604 Hz/cm

8

Current Data Parameters

NAME wj4-39
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters

Date_ 20030804
Time 13.49
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TO 32768
SOLVENT CDCl3
NS 16
DS 2
SMH 6009.615 Hz
FIQRES 0.183399 Hz
AQ 2.7264309 sec
RG 80.6
OW 83.200 usec
DE 6.00 usec
TE 300.0 K
D1 0.03000000 sec

***** CHANNEL f1 *****

NUC1 1H
P1 7.70 usec
PL1 -4.00 dB
SFO1 500.1320005 MHz

F2 - Processing parameters

SF 32768
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

F1 - Processing parameters

SF 1024
MC2 OF
SF 500.1300000 MHz
WDW OSINE
SSB 0
LB 0.30 Hz
GB 0

1D NMR plot parameters

CX 20.00 cm
CY 0.00 cm
FIP 9.500 ppm
F1 4751.23 Hz
F2P -0.500 ppm
F2 -250.07 Hz
PPMCH 0.50000 ppm/cm
HZCM 250.06500 Hz/cm

8

Current Data Parameters

NAME wj4-39
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters

Date_ 20030804
Time 13.53
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TO 65536
SOLVENT CDCl3
NS 383
DS 4
SMH 31446.541 Hz
FIQRES 0.479836 Hz
AQ 1.6420883 sec
RG 2048
OW 15.900 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
D11 0.03000000 sec
D12 0.00002000 sec

***** CHANNEL f1 *****

NUC1 13C
P1 6.50 usec
PL1 5.00 dB
SFO1 125.7718472 MHz

***** CHANNEL f2 *****

CPDPRG2 waltz16
MC2 1H
PCPD2 95.00 usec
PL2 -4.00 dB
PL12 19.00 dB
PL13 30.00 dB
SFO2 500.1325000 MHz

F2 - Processing parameters

SF 65536
SF 125.7570000 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

F1 - Processing parameters

SF 1024
MC2 OF
SF 500.1300000 MHz
WDW no
SSB 0
LB 0.30 Hz
GB 0

1D NMR plot parameters

CX 20.00 cm
CY 0.00 cm
FIP 220.000 ppm
F1 27866.71 Hz
F2P -0.500 ppm
F2 -62.88 Hz
PPMCH 11.02500 ppm/cm
HZCM 1386.47974 Hz/cm

10

10

Current Data Parameters
NAME dsrhl
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20020522
Time 9.40
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 4
DS 2
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 203.2
DW 104.400 usec
DE 4.50 usec
TE 300.0 K
D1 1.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 7.70 usec
PL1 -6.00 dB
SFO1 400.1320007 MHz

F2 - Processing parameters
SI 16384
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P -0.500 ppm
F2 -200.07 Hz
PPMCM 0.52500 ppm/cm
HZCM 210.06825 Hz/cm

Current Data Parameters
NAME dsrcl3
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20020522
Time 17.27
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 81
DS 2
SWH 23148.148 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 32768
DW 21.600 usec
DE 4.50 usec
TE 300.0 K
D1 0.05000000 sec
d12 0.03000000 sec
d12 0.00002000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 12.30 usec
PL1 2.00 dB
SFO1 100.6232933 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 18.00 dB
PL13 18.00 dB
SFO2 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 220.000 ppm
F1 22134.80 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 11.00000 ppm/cm
HZCM 1106.73999 Hz/cm

13

Current Data Parameters
NAME dsrhl
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20020520
Time 9.36
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 4
DS 2
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 128
DW 104.400 usec
DE 4.50 usec
TE 300.0 K
D1 1.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 7.70 usec
PL1 -6.00 dB
SFO1 400.1320007 MHz

F2 - Processing parameters
SI 16384
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P -0.500 ppm
F2 -200.07 Hz
PPMCM 0.52500 ppm/cm
HZCM 210.06825 Hz/cm

13

Current Data Parameters
NAME dsrhl
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20020520
Time 9.40
INSTRUM drx400
PROBHD 5 mm Multinu
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 183
DS 2
SWH 23148.148 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 32768
DW 21.600 usec
DE 4.50 usec
TE 300.0 K
d11 0.05000000 sec
d12 0.03000000 sec
d12 0.00002000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 12.30 usec
PL1 2.00 dB
SFO1 100.6232933 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 18.00 dB
PL13 18.00 dB
SFO2 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 220.000 ppm
F1 22134.80 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 11.00000 ppm/cm
HZCM 1106.73999 Hz/cm

14

Current Data Parameters
NAME dsr-III-16
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030718
Time 15.22
INSTRUM drx400
PROBHD 5 mm Multinucl
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 2
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 50.8
DW 104.400 usec
DE 4.50 usec
TE 300.0 K
D1 1.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 7.70 usec
PL1 -6.00 dB
SFO1 400.1320007 MHz

F2 - Processing parameters
SI 16384
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 6.00 cm
F1P 11.000 ppm
F1 4401.43 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCM 0.60000 ppm/cm
HZCM 240.07800 Hz/cm

14

Current Data Parameters
NAME dsr-III-16
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030718
Time 15.26
INSTRUM drx400
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 124
DS 2
SWH 23148.148 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 32768
DW 21.600 usec
DE 4.50 usec
TE 300.0 K
D1 0.05000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 12.30 usec
PL1 2.00 dB
SFO1 100.6232933 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
P12 0.00 dB
PL12 18.00 dB
PL13 18.00 dB
SFO2 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
CY 12.50 cm
F1P 220.000 ppm
F1 22134.80 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 11.00000 ppm/cm
HZCM 1106.73999 Hz/cm

Current Data Parameters
NAME wj4-26.crude
EXFNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030619
Time 16.36
INSTRUM drx400
PROBHD 5 mm Multinucl
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 64
DW 104.400 usec
DE 4.50 usec
TE 300.0 K
D1 1.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 7.70 usec
PL1 -6.00 dB
SFO1 400.1320007 MHz

F2 - Processing parameters
SI 16384
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 0.00 cm
F1P 10.985 ppm
F1 4395.28 Hz
F2P -0.500 ppm
F2 -200.07 Hz
PPMCM 0.57423 ppm/cm
HZCM 229.76746 Hz/cm

Current Data Parameters
NAME wj4-26.crude
EXFNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030619
Time 16.40
INSTRUM drx400
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 197
DS 2
SWH 23148.148 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 32768
DW 21.600 usec
DE 4.50 usec
TE 300.0 K
D1 0.05000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 12.30 usec
PL1 2.00 dB
SFO1 100.6232933 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
F12 0.00 dB
PL12 18.00 dB
PL13 18.00 dB
SFO2 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
CY 0.00 cm
F1P 220.036 ppm
F1 22138.41 Hz
F2P -0.500 ppm
F2 -50.31 Hz
PPMCM 11.02680 ppm/cm
HZCM 1109.43604 Hz/cm

22

Current Data Parameters
NAME wj4-9,7-15-2
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030529
Time 16.36
INSTRUM drx400
PROBHD 5 mm Multinucl
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 64
DW 104.400 usec
DE 4.50 usec
TE 300.0 K
D1 1.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 7.70 usec
PL1 -6.00 dB
SFO1 400.1320007 MHz

F2 - Processing parameters
SI 16384
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 0.00 cm
FIP 8.000 ppm
F1 3201.04 Hz
F2P -0.500 ppm
F2 -200.07 Hz
PPMCH 0.42500 ppm/cm
HZCM 170.05525 Hz/cm

22

Current Data Parameters
NAME wj4-9,7-15-2
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030529
Time 16.39
INSTRUM drx400
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 94
DS 2
SWH 23148.148 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 32768
DW 21.600 usec
DE 4.50 usec
TE 300.0 K
D1 0.05000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 12.30 usec
PL1 2.00 dB
SFO1 100.6232933 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 18.00 dB
PL13 18.00 dB
SFO2 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
CY 0.00 cm
FIP 220.036 ppm
F1 22138.41 Hz
F2P -50.31 Hz
PPMCH 11.02680 ppm/cm
HZCM 1109.43604 Hz/cm