A Tandem Metal Carbene Cyclization-Cycloaddition Approach to the Pseudolaric Acids

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(1'R,2S)-5-Benzylloxymethyl-2-[1'-(2-methoxyethoxymethoxy)ethyl]-5-hexenoic acid N, N'-methoxymethylamide (19). To a solution of acid 18 (40 mg, 0.109 mmol) in CH₂Cl₂ (3 mL) was added MeONHMe·HCl (14 mg, 0.144 mmol), *i*-Pr₂NEt (0.026 mL, 0.150 mmol), and DCC (25 mg, 0.121 mmol) successively at 0 °C. After stirring for 3 days at room temperature, the precipitates were filtered off. The organic filtrate was concentrated *in vacuo*. Further DCU precipitation in ether and filtration, followed by flash chromatography gave compound 19 (20 mg, 44% yield) as a colorless oil.

To a solution of acid 18 (40 mg, 0.109 mmol) in CH₂Cl₂ (3 mL) was added MeONHMe·HCl (14 mg, 0.144 mmol), Et₃N (0.052 mL, 0.375 mmol), and BOP (51 mg, 0.115 mmol) successively at room temperature. After stirring for 4 days, the mixture was diluted with CH₂Cl₂ (6 mL) and washed successively with 5% KHSO₄, 5% NaHCO₃ and saturated NaCl. The organic phase was dried over Na₂SO₄, filtered and concentrated *in vacuo* to give compound 19 (16 mg, 36% yield) as a colorless oil after flash chromatography. 19: *R_f* = 0.35 (50% Et₂O in hexane); IR (film, cm⁻¹): 2935, 2884, 1657, 1455, 1385, 1112, 1040, 908; ¹H NMR (300 MHz, CDCl₃) δ 7.34-7.25 (m, 5H), 5.06 (s, 1H), 4.95 (s, 1H), 4.79 (d, *J* = 7.1 Hz, 1H), 4.72 (d, *J* = 7.1 Hz, 1H), 4.47 (s, 2H), 3.95 (s, 2H), 3.90 (m, 1H), 3.69 (m, 2H), 3.67 (s, 3H), 3.53 (m, 2H), 3.37 (s, 3H), 3.18 (s, 3H), 3.04 (br m, 1H), 2.03 (m, 2H), 1.90 (t, *J* = 7.2 Hz, 2H), 1.19 (d, *J* = 6.2 Hz, 3H); ¹³C NMR (75 MHz, CDCl₃) δ 175.1 (br), 145.6, 138.4, 111.7, 94.4, 74.9, 73.1, 71.9, 71.8, 67.1, 61.3, 59.1, 46.7, 32.2, 30.9, 27.2, 18.4; LRMS (EI): *m/z* 409 (M⁺, 2), 378 (5), 349(9), 334 (14), 273 (28), 241 (25), 183 (23), 167 (29), 91 (100), 89 (65); HRMS (EI): Calcd for C₂₂H₃₅NO₆: 409.2464; Found 409.2452.

1-(1-Benzyl-2-hydroxyethyl)-1-[5-benzylloxymethyl-2'-(1'-hydroxyethyl)-5-hexenoyl]-3-methoxy-3-methylurea (20). To a stirred suspension of MeONHMeHCl (57 mg, 0.58 mmol) in CH₂Cl₂ (2 mL) was added 1.0 M Et₂AlCl in hexane (0.58 mL, 0.58 mmol) at 0 °C. The mixture was allowed to warm to room temperature over 1 h. A solution of compound 16 (83 mg, 0.19 mmol) in CH₂Cl₂ (2 mL) was added dropwise. After stirring for 5 h, phosphate buffer solution (2 mL) was added. The mixture was diluted with CHCl₃, and filtered through a Celite pad. The filtrate was extracted with CHCl₃. The combined organic layers were washed with brine, dried over MgSO₄ and concentrated. The residue was purified by flash chromatography to give compound 20 (29 mg, 32% yield) as a colorless oil. 20: *R_f* 0.15 (60% EtOAc in hexane); IR (film, cm⁻¹): 3512 (br), 2941, 2889, 1682, 1657, 1452, 1381, 1267, 1125, 1049; ¹H NMR (300 MHz, CDCl₃) δ 7.36-7.16 (m, 10H), 5.88 (br s, 1H), 5.07 (s, 1H), 4.97 (s, 1H), 4.48 (s, 2H), 4.42 (t, *J* = 8.1 Hz, 1H), 4.10 (m, 2H), 4.00 (m, 1H), 3.96 (s, 2H), 3.67 (s, 3H), 3.21 (m, 1H), 3.20 (s, 3H), 2.87 (m, 3H), 2.05 (m, 2H), 2.00-1.82 (m, 2H), 1.21 (d, *J* = 6.4 Hz, 3H); ¹³C NMR (75 MHz, CDCl₃) δ 159.4, 145.5, 138.4, 136.0, 129.0, 128.98, 128.4, 127.7, 127.6, 127.2, 111.9, 73.0, 71.9, 69.6, 68.4, 61.5, 53.8, 46.1, 41.4, 32.0, 31.1, 24.4, 20.6; LRMS (EI): *m/z* 433 (M⁺ – OMe – OH – OH, 3), 376 (21), 342 (12),

285 (17), 244 (27), 152 (29), 91 (100); HRMS (EI): Calcd for $C_{27}H_{33}N_2O_3$ ($M^+ - OMe - OH - OH$): 433.2491; Found 433.2496.

9-Benzyloxymethyl-1-diazo-6-(1-methyl-1-triethylsilanyloxyethyl)-dec-9-ene-2,5-dione (25).

Following the procedure for the preparation of substrate 12a, diazoketone 25 was similarly prepared in 69% yield from 8-benzyloxymethyl-5-(1-methyl-1-triethylsilanyloxyethyl)-4-oxonon-8-enoic acid (133.7mg, 0.289mmol), isobutyl chloroformate (0.037ml, 0.289 mmol) and diazomethane (2.6ml, 0.867mmol). 25: $R_f = 0.47$ (20% EtOAc in hexane); 1H NMR (300MHz, $CDCl_3$) δ 7.37-7.27 (m, 5H), 5.25 (br s, 1H), 5.07 (s, 1H), 4.94 (s, 1H), 4.48 (s, 2H), 3.92 (s, 2H), 3.01-2.93 (m, 1H), 2.85-2.77 (m, 1H), 2.72 (dd, $J = 2.3Hz, 11.3Hz$, 1H), 2.61-2.60 (br m, 1H), 2.44-2.40 (br m, 1H), 2.04-1.78 (m, 3H), 1.45-1.56 (m, 1H), 1.24 (s, 3H), 1.19 (s, 3H), 0.92 (t, $J = 8.0Hz$, 9H), 0.56 (q, $J = 8Hz$, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 212.5, 193.7, 145.4, 138.4, 128.4, 127.8, 127.6, 112.4, 77.3, 75.8, 72.8, 72.0, 62.6, 53.8, 41.3, 31.6, 29.2, 26.4, 26.2, 7.1, 6.7; IR (film, cm^{-1}): 2958, 2877, 2108, 1710, 1647, 1370, 1150, 1029; LRMS (EI) m/z 337 ($M^+ - Et$, 12), 309(19), 279(14), 251(53), 210(6); HRMS (EI): Calcd for $C_{25}H_{37}O_4Si$ ($M^+ - N_2$) 429.2461; Found 429.2454.

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7b

200 727

[illegible]

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D1 1.0000000 sec

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10 NMR plot parameters

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7L

1H NMR (400 MHz, DMSO-d₆)
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8b

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1D NMR plot parameters

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S21

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10a

10b

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 P16 12.00 sec
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 PL18 0.00 dB
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 SFO26 300.135000 MHz
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 P29 12.00 sec
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 PL30 0.00 dB
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 P37 12.00 sec
 PL37 0.00 dB
 SFO37 300.135000 MHz
 ===== CHANNEL f38 =====
 NUC38 1H
 P38 12.00 sec
 PL38 0.00 dB
 SFO38 300.135000 MHz
 ===== CHANNEL f39 =====
 NUC39 1H
 P39 12.00 sec
 PL39 0.00 dB
 SFO39 300.135000 MHz
 ===== CHANNEL f40 =====
 NUC40 1H
 P40 12.00 sec
 PL40 0.00 dB
 SFO40 300.135000 MHz
 ===== CHANNEL f41 =====
 NUC41 1H
 P41 12.00 sec
 PL41 0.00 dB
 SFO41 300.135000 MHz
 ===== CHANNEL f42 =====
 NUC42 1H
 P42 12.00 sec
 PL42 0.00 dB
 SFO42 300.135000 MHz
 ===== CHANNEL f43 =====
 NUC43 1H
 P43 12.00 sec
 PL43 0.00 dB
 SFO43 300.135000 MHz
 ===== CHANNEL f44 =====
 NUC44 1H
 P44 12.00 sec
 PL44 0.00 dB
 SFO44 300.135000 MHz
 ===== CHANNEL f45 =====
 NUC45 1H
 P45 12.00 sec
 PL45 0.00 dB
 SFO45 300.135000 MHz
 ===== CHANNEL f46 =====
 NUC46 1H
 P46 12.00 sec
 PL46 0.00 dB
 SFO46 300.135000 MHz
 ===== CHANNEL f47 =====
 NUC47 1H
 P47 12.00 sec
 PL47 0.00 dB
 SFO47 300.135000 MHz
 ===== CHANNEL f48 =====
 NUC48 1H
 P48 12.00 sec
 PL48 0.00 dB
 SFO48 300.135000 MHz
 ===== CHANNEL f49 =====
 NUC49 1H
 P49 12.00 sec
 PL49 0.00 dB
 SFO49 300.135000 MHz
 ===== CHANNEL f50 =====
 NUC50 1H
 P50 12.00 sec
 PL50 0.00 dB
 SFO50 300.135000 MHz
 ===== CHANNEL f51 =====
 NUC51 1H
 P51 12.00 sec
 PL51 0.00 dB
 SFO51 300.135000 MHz
 ===== CHANNEL f52 =====
 NUC52 1H
 P52 12.00 sec
 PL52 0.00 dB
 SFO52 300.135000 MHz
 ===== CHANNEL f53 =====
 NUC53 1H
 P53 12.00 sec
 PL53 0.00 dB
 SFO53 300.135000 MHz
 ===== CHANNEL f54 =====
 NUC54 1H
 P54 12.00 sec
 PL54 0.00 dB
 SFO54 300.135000 MHz
 ===== CHANNEL f55 =====
 NUC55 1H
 P55 12.00 sec
 PL55 0.00 dB
 SFO55 300.135000 MHz
 ===== CHANNEL f56 =====
 NUC56 1H
 P56 12.00 sec
 PL56 0.00 dB
 SFO56 300.135000 MHz
 ===== CHANNEL f57 =====
 NUC57 1H
 P57 12.00 sec
 PL57 0.00 dB
 SFO57 300.135000 MHz
 ===== CHANNEL f58 =====
 NUC58 1H
 P58 12.00 sec
 PL58 0.00 dB
 SFO58 300.135000 MHz
 ===== CHANNEL f59 =====
 NUC59 1H
 P59 12.00 sec
 PL59 0.00 dB
 SFO59 300.135000 MHz
 ===== CHANNEL f60 =====
 NUC60 1H
 P60 12.00 sec
 PL60 0.00 dB
 SFO60 300.135000 MHz
 ===== CHANNEL f61 =====
 NUC61 1H
 P61 12.00 sec
 PL61 0.00 dB
 SFO61 300.135000 MHz
 ===== CHANNEL f62 =====
 NUC62 1H
 P62 12.00 sec
 PL62 0.00 dB
 SFO62 300.135000 MHz
 ===== CHANNEL f63 =====
 NUC63 1H
 P63 12.00 sec
 PL63 0.00 dB
 SFO63 300.135000 MHz
 ===== CHANNEL f64 =====
 NUC64 1H
 P64 12.00 sec
 PL64 0.00 dB
 SFO64 300.135000 MHz
 ===== CHANNEL f65 =====
 NUC65 1H
 P65 12.00 sec
 PL65 0.00 dB
 SFO65 300.135000 MHz
 ===== CHANNEL f66 =====
 NUC66 1H
 P66 12.00 sec
 PL66 0.00 dB
 SFO66 300.135000 MHz
 ===== CHANNEL f67 =====
 NUC67 1H
 P67 12.00 sec
 PL67 0.00 dB
 SFO67 300.135000 MHz
 ===== CHANNEL f68 =====
 NUC68 1H
 P68 12.00 sec
 PL68 0.00 dB
 SFO68 300.135000 MHz
 ===== CHANNEL f69 =====
 NUC69 1H
 P69 12.00 sec
 PL69 0.00 dB
 SFO69 300.135000 MHz
 ===== CHANNEL f70 =====
 NUC70 1H
 P70 12.00 sec
 PL70 0.00 dB
 SFO70 300.135000 MHz
 ===== CHANNEL f71 =====
 NUC71 1H
 P71 12.00 sec
 PL71 0.00 dB
 SFO71 300.135000 MHz
 ===== CHANNEL f72 =====
 NUC72 1H
 P72 12.00 sec
 PL72 0.00 dB
 SFO72 300.135000 MHz
 ===== CHANNEL f73 =====
 NUC73 1H
 P73 12.00 sec
 PL73 0.00 dB
 SFO73 300.135000 MHz
 ===== CHANNEL f74 =====
 NUC74 1H
 P74 12.00 sec
 PL74 0.00 dB
 SFO74 300.135000 MHz
 ===== CHANNEL f75 =====
 NUC75 1H
 P75 12.00 sec
 PL75 0.00 dB
 SFO75 300.135000 MHz
 ===== CHANNEL f76 =====
 NUC76 1H
 P76 12.00 sec
 PL76 0.00 dB
 SFO76 300.135000 MHz
 ===== CHANNEL f77 =====
 NUC77 1H
 P77 12.00 sec
 PL77 0.00 dB
 SFO77 300.135000 MHz
 ===== CHANNEL f78 =====
 NUC78 1H
 P78 12.00 sec
 PL78 0.00 dB
 SFO78 300.135000 MHz
 ===== CHANNEL f79 =====
 NUC79 1H
 P79 12.00 sec
 PL79 0.00 dB
 SFO79 300.135000 MHz
 ===== CHANNEL f80 =====
 NUC80 1H
 P80 12.00 sec
 PL80 0.00 dB
 SFO80 300.135000 MHz
 ===== CHANNEL f81 =====
 NUC81 1H
 P81 12.00 sec
 PL81 0.00 dB
 SFO81 300.135000 MHz
 ===== CHANNEL f82 =====
 NUC82 1H
 P82 12.00 sec
 PL82 0.00 dB
 SFO82 300.135000 MHz
 ===== CHANNEL f83 =====
 NUC83 1H
 P83 12.00 sec
 PL83 0.00 dB
 SFO83 300.135000 MHz
 ===== CHANNEL f84 =====
 NUC84 1H
 P84 12.00 sec
 PL84 0.00 dB
 SFO84 300.135000 MHz
 ===== CHANNEL f85 =====
 NUC85 1H
 P85 12.00 sec
 PL85 0.00 dB
 SFO85 300.135000 MHz
 ===== CHANNEL f86 =====
 NUC86 1H
 P86 12.00 sec
 PL86 0.00 dB
 SFO86 300.135000 MHz
 ===== CHANNEL f87 =====
 NUC87 1H
 P87 12.00 sec
 PL87 0.00 dB
 SFO87 300.135000 MHz
 ===== CHANNEL f88 =====
 NUC88 1H
 P88 12.00 sec
 PL88 0.00 dB
 SFO88 300.135000 MHz
 ===== CHANNEL f89 =====
 NUC89 1H
 P89 12.00 sec
 PL89 0.00 dB
 SFO89 300.135000 MHz
 ===== CHANNEL f90 =====
 NUC90 1H
 P90 12.00 sec
 PL90 0.00 dB
 SFO90 300.135000 MHz
 ===== CHANNEL f91 =====
 NUC91 1H
 P91 12.00 sec
 PL91 0.00 dB
 SFO91 300.135000 MHz
 ===== CHANNEL f92 =====
 NUC92 1H
 P92 12.00 sec
 PL92 0.00 dB
 SFO92 300.135000 MHz
 ===== CHANNEL f93 =====
 NUC93 1H
 P93 12.00 sec
 PL93 0.00 dB
 SFO93 300.135000 MHz
 ===== CHANNEL f94 =====
 NUC94 1H
 P94 12.00 sec
 PL94 0.00 dB
 SFO94 300.135000 MHz
 ===== CHANNEL f95 =====
 NUC95 1H
 P95 12.00 sec
 PL95 0.00 dB
 SFO95 300.135000 MHz
 ===== CHANNEL f96 =====
 NUC96 1H
 P96 12.00 sec
 PL96 0.00 dB
 SFO96 300.135000 MHz
 ===== CHANNEL f97 =====
 NUC97 1H
 P97 12.00 sec
 PL97 0.00 dB
 SFO97 300.135000 MHz
 ===== CHANNEL f98 =====
 NUC98 1H
 P98 12.00 sec
 PL98 0.00 dB
 SFO98 300.135000 MHz
 ===== CHANNEL f99 =====
 NUC99 1H
 P99 12.00 sec
 PL99 0.00 dB
 SFO99 300.135000 MHz
 ===== CHANNEL f100 =====
 NUC100 1H
 P100 12.00 sec
 PL100 0.00 dB
 SFO100 300.135000 MHz

214.698
ppm

144.991
137.839
127.996
127.354
127.238
111.968
77.432
77.004
76.578
72.524
71.549
61.326
45.495
37.344
30.288
26.311
26.042
16.067

Current Data Parameters
 NAME: 11a
 EXPNO: 1
 PROCNO: 10
 F2 - Acquisition Parameters
 Date_ Time: 20170515 22:45
 INSTRUM: spect
 PULPROG: zgpg30
 SOLVENT: CDCl3
 NS: 13
 DS: 4
 SWH: 6472.639 Hz
 FIDRES: 0.090000 Hz
 AQ: 2.6642600 sec
 RG: 320
 GB: 200.1
 PC: 1.00
 DE: 1.00
 TE: 300.2 K
 D1: 4.50 sec
 D2: 1.00000000 sec
 P1: 9.00 usec
 P2: 300.1132500 sec
 SFO1: 101.253 MHz
 SFO2: 101.253 MHz
 SFO3: 101.253 MHz
 F2 - Processing parameters
 SI: 32768
 SF: 300.1360953 MHz
 DS: 4
 SW: 6472.639 Hz
 FIDRES: 0.090000 Hz
 AQ: 2.6642600 sec
 RG: 320
 GB: 200.1
 PC: 1.00
 DE: 1.00
 TE: 300.2 K
 D1: 4.50 sec
 D2: 1.00000000 sec
 P1: 9.00 usec
 P2: 300.1132500 sec
 SFO1: 101.253 MHz
 SFO2: 101.253 MHz
 SFO3: 101.253 MHz

11a

Current Data Parameters
 NAME July15-2000-sm
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20000715
 Time 10.43
 INSTRUM dpx300
 PROBD 5 mm Dual 13
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 6172.839 Hz
 FIDRES 0.188380 Hz
 AQ 2.6542580 sec
 RG 16
 DW 81.000 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec

===== CHANNEL f1 =====

NUC1 1H
 P1 9.00 usec
 PL1 -6.00 dB
 SF01 300.1318534 MHz

F2 - Processing parameters

SI 32768
 SF 300.1300105 MHz
 MDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters

CX 20.00 cm
 F1P 12.000 ppm
 F1 3601.56 Hz
 F2P -0.500 ppm
 F2 -150.07 Hz
 PPMCM 0.62500 ppm/cm
 HZCM 187.58125 Hz/cm

Current Data Parameters
 Name: 12a
 EXPNO: 1
 PROCNO: 1
 F2 - Acquisition Parameters
 Date_: 08/10/09
 Time: 14:45
 INSTRUM: spect
 PULPROG: zgpg30
 FIDRES: 5.00 Hz
 TO LINDO: 32768
 COC13: 32768
 SOLVENT: CDCl3
 DS: 2
 SFO: 6172.859 Hz
 ZPRES: 2.000000 Hz
 A2: 2.000000 Hz
 RE: 61
 DE: 4.00 usec
 TE: 300.0 K
 D1: 1.0000000 sec
 DE: 4.00 usec
 SFO1: 300.1318534 MHz
 F1: -5.00 dB
 F2 - Processing parameters
 SI: 300.1300007 MHz
 SF: 300.1300007 MHz
 EQ: 0
 SFO: 0
 LB: 0.30 Hz
 GB: 0
 PC: 1.00
 G3: 90.00 Hz
 G4: 90.00 Hz
 F1P: 9.197 GHz
 F2P: 250.1318534 MHz
 F3P: -0.703 GHz
 F4P: -210.00 MHz
 SFO2: 0.0000000 MHz
 H2O2: 115.1900000 MHz

12a

0.00

wdw

```

MNUF      SGNON
OBNUC      1H
OFR        270.05 MHz
OBSET      112.00 KHz
OBFIN      5800.0 Hz
PW1        5.0 us
DEADT      108.6 us
PREDL      0.2000 ms
IWT        1.0 sec
POINT      16384
SPO        16384
TIMES      16
DUMMY      1
FREQU      5402.4 Hz
FLT        2700 Hz
DELAY      74.1 us
ACQTM      3.033 sec
PD         1.500 sec
ADBIT      16.00
RGAIN      16
BF         0.30 Hz
T1         0.00
T2         0.00
T3         90.00
T4         100.00
EXMOD      SGNON
EXPCM      SGNON:Single.Coupled:PW1-AC
IRNUC      1H
IFR        270.05 MHz
IRSET      112.00 KHz
IRFIN      5800.0 Hz
IRFPW      60 us
IRATN      511
DFILE      C:\WINNMR95\COMMON\_DEFAULT
SF         TH5
LKSET      64.60 KHz
LKFIN      59.0 Hz
LKLEV      190
LGAIN      21
LKPHS      113
LKSIG      188
CSPED      11 Hz
FILDG      FIDF
  
```

12b

13a

13a

14a

14a

```

Current Data Parameters
NAME rx390n14
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20010328
Time 8.36
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 823
DS 2
SWH 25125.629 Hz
FIDRES 0.766773 Hz
AQ 0.652132 sec
RG 1149.4
DN 19.500 usec
DE 6.00 usec
TE 300.2 K
D1 3.0000000 sec
d11 0.0300000 sec
d12 0.0000000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 6.20 usec
PL1 -5.00 dB
SFO1 100.6237864 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
P2 100.00 usec
PL2 120.00 dB
PL12 14.00 dB
PL13 14.00 dB
SFO2 400.1318005 MHz

F2 - Processing parameters
SI 32768
SF 100.612053 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 0.70

1D NMR plot parameters
GX 25.00 cm
CY 5.00 cm
FID 240.000 cm
F1 244.700 Hz
F2 -10.000 ppm
F2 -1005.13 Hz
PPMCN 10.00000 ppm/cm
HZCN 1005.13762 Hz/cm

```

Current Data Parameters
NAME xenobius
EXPNO 3
PROCNO 1
F2 Acquisition Parameters
Date_ 20051023
Time 4:02
INSTnm dr-6500
PROBHD BB0 5mm
PULPROG zgpg30
TD 32768
SFOVENT CQCL3
NS 1428
DS 4
SWH 31446.541 Hz
FIDRES 0.856672 Hz
AQ 0.5210612 sec
RG 4096
AQ 15.500 usec
DE 1.500 usec
TE 300.0 K
U1 3.0000000 sec
G11 0.0300000 sec
----- CHANNEL f1 -----
NUC1 13C
P1 1.50 usec
PL1 0.00 dB
SC1 125.771524 MHz
----- CHANNEL f2 -----
CPDPRG2 waltz16
NUC2 1H
P2PRG2 100.00 usec
PL2 -6.00 dB
PC12 17.00 dB
SFO2 500.137900 MHz
F2 - Processing parameters
SI 32768
SF 500.137900 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
LA 25.00 dB
LB 20.00 dB
GB 0.00 dB
F1 270M 71 MHz
F2 500.137900 MHz
P2 1.50 usec
RG 4096
WDW EM
SSB 0
LB 1.00 Hz
GB 0.00 dB
PC 1.00

14b

Current Data Parameters
 NAME: 2415
 EXPNO: 1
 PROCNO: 1
 F2 - Acquisition Parameters
 Date_: 19980808
 Time: 15.32
 INSTRUM: spect
 PROBHD: 5 mm BBO 13C/1H
 PULPROG: zgpg30
 TO PROG: zgpg30
 SOLVENT: CDCl3
 DS: 4
 AS: 2
 SFO: 612.839 MHz
 AQ: 2.5642580 sec
 FIDRES: 0.083000 Hz
 RM: 81.000 usec
 DE: 4.00 usec
 TE: 300.2 K
 D1: 1.00000000 sec
 P1: 9.00 usec
 PL1: 0.00 dB
 SFO2: 300.136261 MHz
 MZC1: -5.00 dB
 F2 - Processing parameters
 SI: 32768
 SF: 300.130094 MHz
 DSF: 16384
 ASFO: 0.000000 MHz
 LB: 0.30 Hz
 GB: 0
 PC: 1.00
 LO MD 0101 parameters
 CB: 26.25 cm
 F1: 117.17 MHz
 F2: 270.17 MHz
 F3: 1.000 MHz
 WDS: 0.000000 MHz
 CH1: 300.130094 MHz
 CH2: 300.130094 MHz

Current Data Parameters
NAME
EXPNO 10
PROCNO 1
Date_ Time
19.10
19.10
PROBHD 5 mm QNP1 125/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2
DS 2
SWH 5175.899 Hz
AQ 0.05000000 sec
RG 81.000 usec
DE 1.100000000 usec
TE 300.2 K
F1 9.40 usec
SFO1 360.1326800 MHz
PC 1.00
13 NMR Data Parameters
C1 25.25 usec
F1 12.50 usec
SFO1 101.6251250 MHz
NUC1 13C
NUC2 1H
NUC3 13C
PROMCH 0.41518 sec/chan
AQDN 120.75518 Hz/chan

Current Data Parameters
NAME: p-05-1
EXPNO: 2
PROCNO: 1
F2 - Acquisition Parameters
Date_ Time: 23.36
Time: 23.36
INSTRUM: spect
PROBHD: 5 mm QNP 1H/13
PULPROG: zgpg30
TD: 65536
SOLVENT: CDCl3
NS: 41
DS: 4
SWH: 6172.879 Hz
FIDRES: 0.08636 Hz
AQ: 2.544628 sec
RG: 57
AQ: 0.000000 sec
DE: 1.0000000 Hz
TE: 300.2 K
D1: 1.00000000 sec
d11: 0.00000000 sec
DE: 4.50 MHz
SFO1: 300.131029 MHz
SFO2: 101.626119 MHz
NUC1: 13C
NUC2: 1H
F2 - Processing parameters
SI: 32768
SF: 300.130050 MHz
WDW: EM
SSB: 0
LB: 0.30 Hz
GB: 0
PC: 1.00
SC: 300.130050 MHz
CT: 26.25 Hz
F1P: 9.058 ppm
F2P: -0.007 ppm
F2: -74.4728 Hz
NUC1R: 13C
NUC1F: 112.45480 Hz

Current	State	Parameters	MCs
1	EXPLO	EXPLO	1
2	EXPLO	EXPLO	1
F2 - Acquisition parameters			
3	EXPLO	EXPLO	1
4	EXPLO	EXPLO	1
5	EXPLO	EXPLO	1
6	EXPLO	EXPLO	1
7	EXPLO	EXPLO	1
8	EXPLO	EXPLO	1
9	EXPLO	EXPLO	1
10	EXPLO	EXPLO	1
11	EXPLO	EXPLO	1
12	EXPLO	EXPLO	1
13	EXPLO	EXPLO	1
14	EXPLO	EXPLO	1
15	EXPLO	EXPLO	1
16	EXPLO	EXPLO	1
17	EXPLO	EXPLO	1
18	EXPLO	EXPLO	1
19	EXPLO	EXPLO	1
20	EXPLO	EXPLO	1
21	EXPLO	EXPLO	1
22	EXPLO	EXPLO	1
23	EXPLO	EXPLO	1
24	EXPLO	EXPLO	1
25	EXPLO	EXPLO	1
26	EXPLO	EXPLO	1
27	EXPLO	EXPLO	1
28	EXPLO	EXPLO	1
29	EXPLO	EXPLO	1
30	EXPLO	EXPLO	1
31	EXPLO	EXPLO	1
32	EXPLO	EXPLO	1
33	EXPLO	EXPLO	1
34	EXPLO	EXPLO	1
35	EXPLO	EXPLO	1
36	EXPLO	EXPLO	1
37	EXPLO	EXPLO	1
38	EXPLO	EXPLO	1
39	EXPLO	EXPLO	1
40	EXPLO	EXPLO	1
41	EXPLO	EXPLO	1
42	EXPLO	EXPLO	1
43	EXPLO	EXPLO	1
44	EXPLO	EXPLO	1
45	EXPLO	EXPLO	1
46	EXPLO	EXPLO	1
47	EXPLO	EXPLO	1
48	EXPLO	EXPLO	1
49	EXPLO	EXPLO	1
50	EXPLO	EXPLO	1
51	EXPLO	EXPLO	1
52	EXPLO	EXPLO	1
53	EXPLO	EXPLO	1
54	EXPLO	EXPLO	1
55	EXPLO	EXPLO	1
56	EXPLO	EXPLO	1
57	EXPLO	EXPLO	1
58	EXPLO	EXPLO	1
59	EXPLO	EXPLO	1
60	EXPLO	EXPLO	1
61	EXPLO	EXPLO	1
62	EXPLO	EXPLO	1
63	EXPLO	EXPLO	1
64	EXPLO	EXPLO	1
65	EXPLO	EXPLO	1
66	EXPLO	EXPLO	1
67	EXPLO	EXPLO	1
68	EXPLO	EXPLO	1
69	EXPLO	EXPLO	1
70	EXPLO	EXPLO	1
71	EXPLO	EXPLO	1
72	EXPLO	EXPLO	1
73	EXPLO	EXPLO	1
74	EXPLO	EXPLO	1
75	EXPLO	EXPLO	1
76	EXPLO	EXPLO	1
77	EXPLO	EXPLO	1
78	EXPLO	EXPLO	1
79	EXPLO	EXPLO	1
80	EXPLO	EXPLO	1
81	EXPLO	EXPLO	1
82	EXPLO	EXPLO	1
83	EXPLO	EXPLO	1
84	EXPLO	EXPLO	1
85	EXPLO	EXPLO	1
86	EXPLO	EXPLO	1
87	EXPLO	EXPLO	1
88	EXPLO	EXPLO	1
89	EXPLO	EXPLO	1
90	EXPLO	EXPLO	1
91	EXPLO	EXPLO	1
92	EXPLO	EXPLO	1
93	EXPLO	EXPLO	1
94	EXPLO	EXPLO	1
95	EXPLO	EXPLO	1
96	EXPLO	EXPLO	1
97	EXPLO	EXPLO	1
98	EXPLO	EXPLO	1
99	EXPLO	EXPLO	1
100	EXPLO	EXPLO	1
F2 - Acquisition parameters			
101	EXPLO	EXPLO	1
102	EXPLO	EXPLO	1
103	EXPLO	EXPLO	1
104	EXPLO	EXPLO</

19

1D ¹H NMR parameters
 Name: 19
 ExpNO: 1
 F2: 500.135 MHz
 F1: 125.761 MHz
 AQ: 0.163 s
 SFO: 500.135 MHz
 SF: 125.761 MHz
 PC: 0.70
 LB: 1.00 Hz
 GB: 0
 CB: 0.00 Hz
 F2 - Processing parameters
 SF: 500.135 MHz
 LB: 1.00 Hz
 GB: 0
 CB: 0.00 Hz
 F1 - Processing parameters
 SF: 125.761 MHz
 LB: 1.00 Hz
 GB: 0
 CB: 0.00 Hz
 F2 - Processing parameters
 SF: 500.135 MHz
 LB: 1.00 Hz
 GB: 0
 CB: 0.00 Hz
 F1 - Processing parameters
 SF: 125.761 MHz
 LB: 1.00 Hz
 GB: 0
 CB: 0.00 Hz

Current Data Parameters
NAME: 8441-2
EXPNO: 1
PROCNO: 1

F2 - Acquisition Parameters

Date_: 990117

Time: 17.40

INSTRUM: spect

PROBHD: 5 mm QNP 1H/13

PULPROG: zgpg30

TD: 65536

SF: 400.136260

WDW: EM

SSB: 0

LB: 3.00

GB: 0

PC: 1.00

AD: 2.0000000

RG: 327.5

IN: 81.000

DE: 1.0000000

TE: 300.2

JE: 1.0000000

SE: 1.0000000

SI: 500.130700

RG: 327.5

IN: 81.000

DE: 1.0000000

TE: 300.2

JE: 1.0000000

SE: 1.0000000

SI: 500.130700

RG: 327.5

IN: 81.000

DE: 1.0000000

TE: 300.2

JE: 1.0000000

SE: 1.0000000

SI: 500.130700

RG: 327.5

IN: 81.000

DE: 1.0000000

TE: 300.2

JE: 1.0000000

SE: 1.0000000

SI: 500.130700

RG: 327.5

IN: 81.000

DE: 1.0000000

TE: 300.2

JE: 1.0000000

SE: 1.0000000

SI: 500.130700

RG: 327.5

IN: 81.000

DE: 1.0000000

TE: 300.2

JE: 1.0000000

SE: 1.0000000

SI: 500.130700

RG: 327.5

IN: 81.000

DE: 1.0000000

TE: 300.2

JE: 1.0000000

SE: 1.0000000

SI: 500.130700

RG: 327.5

IN: 81.000

DE: 1.0000000

TE: 300.2

JE: 1.0000000

SE: 1.0000000

SI: 500.130700

RG: 327.5

IN: 81.000

DE: 1.0000000

TE: 300.2

JE: 1.0000000

SE: 1.0000000

SI: 500.130700

RG: 327.5

IN: 81.000

DE: 1.0000000

TE: 300.2

JE: 1.0000000

SE: 1.0000000

SI: 500.130700

RG: 327.5

IN: 81.000

DE: 1.0000000

TE: 300.2

JE: 1.0000000

SE: 1.0000000

20

055

21

Current Data Parameters
NAME: 4473
PROCNO: 1
F2 - Acquisition Parameters
DATE_: 200811
INSTRUM: spect
PROBHD: 5 mm QNP 1H/13
PULPROG: zgpg30
PCPDPRG: zgpg30
AQ: 0.22000000
RG: 655
SI: 32768
SF: 500.1360000 MHz
FIDRES: 0.1772328 Hz
AQRES: 0.1666666 Hz
RGRES: 2.0000000 Hz
RG: 655
TE: 300.2 K
F1: 120.9000000 MHz
F2: 100.6281250 MHz
SF2: 500.1360000 MHz
PC1: 300.1360000 MHz
PC2: 100.6281250 MHz
PC3: 400.1460000 MHz
PC4: 100.6281250 MHz
PC5: 100.6281250 MHz
PC6: 100.6281250 MHz
PC7: 100.6281250 MHz
PC8: 100.6281250 MHz
PC9: 100.6281250 MHz
PC10: 100.6281250 MHz
PC11: 100.6281250 MHz
PC12: 100.6281250 MHz
PC13: 100.6281250 MHz
PC14: 100.6281250 MHz
PC15: 100.6281250 MHz
PC16: 100.6281250 MHz
PC17: 100.6281250 MHz
PC18: 100.6281250 MHz
PC19: 100.6281250 MHz
PC20: 100.6281250 MHz
PC21: 100.6281250 MHz
PC22: 100.6281250 MHz
PC23: 100.6281250 MHz
PC24: 100.6281250 MHz
PC25: 100.6281250 MHz
PC26: 100.6281250 MHz
PC27: 100.6281250 MHz
PC28: 100.6281250 MHz
PC29: 100.6281250 MHz
PC30: 100.6281250 MHz
PC31: 100.6281250 MHz
PC32: 100.6281250 MHz
PC33: 100.6281250 MHz
PC34: 100.6281250 MHz
PC35: 100.6281250 MHz
PC36: 100.6281250 MHz
PC37: 100.6281250 MHz
PC38: 100.6281250 MHz
PC39: 100.6281250 MHz
PC40: 100.6281250 MHz
PC41: 100.6281250 MHz
PC42: 100.6281250 MHz
PC43: 100.6281250 MHz
PC44: 100.6281250 MHz
PC45: 100.6281250 MHz
PC46: 100.6281250 MHz
PC47: 100.6281250 MHz
PC48: 100.6281250 MHz
PC49: 100.6281250 MHz
PC50: 100.6281250 MHz
PC51: 100.6281250 MHz
PC52: 100.6281250 MHz
PC53: 100.6281250 MHz
PC54: 100.6281250 MHz
PC55: 100.6281250 MHz
PC56: 100.6281250 MHz
PC57: 100.6281250 MHz
PC58: 100.6281250 MHz
PC59: 100.6281250 MHz
PC60: 100.6281250 MHz
PC61: 100.6281250 MHz
PC62: 100.6281250 MHz
PC63: 100.6281250 MHz
PC64: 100.6281250 MHz
PC65: 100.6281250 MHz
PC66: 100.6281250 MHz
PC67: 100.6281250 MHz
PC68: 100.6281250 MHz
PC69: 100.6281250 MHz
PC70: 100.6281250 MHz
PC71: 100.6281250 MHz
PC72: 100.6281250 MHz
PC73: 100.6281250 MHz
PC74: 100.6281250 MHz
PC75: 100.6281250 MHz
PC76: 100.6281250 MHz
PC77: 100.6281250 MHz
PC78: 100.6281250 MHz
PC79: 100.6281250 MHz
PC80: 100.6281250 MHz
PC81: 100.6281250 MHz
PC82: 100.6281250 MHz
PC83: 100.6281250 MHz
PC84: 100.6281250 MHz
PC85: 100.6281250 MHz
PC86: 100.6281250 MHz
PC87: 100.6281250 MHz
PC88: 100.6281250 MHz
PC89: 100.6281250 MHz
PC90: 100.6281250 MHz
PC91: 100.6281250 MHz
PC92: 100.6281250 MHz
PC93: 100.6281250 MHz
PC94: 100.6281250 MHz
PC95: 100.6281250 MHz
PC96: 100.6281250 MHz
PC97: 100.6281250 MHz
PC98: 100.6281250 MHz
PC99: 100.6281250 MHz
PC100: 100.6281250 MHz

[illegible]

21

Current Data Parameters
 NAME: 1
 INSTRUM: 1
 F2 - Acquisition Parameters
 DATE_: 200001
 TIME_: 09:30
 INSTRUM: 5 mm 1H/13C NMR
 PULPROG: zgpg30
 TO: 2570
 FIDRES: 0.00018
 SFO: 600.13177
 AQRES: 0.000060
 RG: 327.5
 DE: 4.00
 TE: 300.2
 P1: 1.000000
 P2: 0.100000
 P3: 0.100000
 P4: 0.100000
 P5: 0.100000
 P6: 0.100000
 P7: 0.100000
 P8: 0.100000
 P9: 0.100000
 P10: 0.100000
 P11: 0.100000
 P12: 0.100000
 P13: 0.100000
 P14: 0.100000
 P15: 0.100000
 P16: 0.100000
 P17: 0.100000
 P18: 0.100000
 P19: 0.100000
 P20: 0.100000
 P21: 0.100000
 P22: 0.100000
 P23: 0.100000
 P24: 0.100000
 P25: 0.100000
 P26: 0.100000
 P27: 0.100000
 P28: 0.100000
 P29: 0.100000
 P30: 0.100000
 P31: 0.100000
 P32: 0.100000
 P33: 0.100000
 P34: 0.100000
 P35: 0.100000
 P36: 0.100000
 P37: 0.100000
 P38: 0.100000
 P39: 0.100000
 P40: 0.100000
 P41: 0.100000
 P42: 0.100000
 P43: 0.100000
 P44: 0.100000
 P45: 0.100000
 P46: 0.100000
 P47: 0.100000
 P48: 0.100000
 P49: 0.100000
 P50: 0.100000
 P51: 0.100000
 P52: 0.100000
 P53: 0.100000
 P54: 0.100000
 P55: 0.100000
 P56: 0.100000
 P57: 0.100000
 P58: 0.100000
 P59: 0.100000
 P60: 0.100000
 P61: 0.100000
 P62: 0.100000
 P63: 0.100000
 P64: 0.100000
 P65: 0.100000
 P66: 0.100000
 P67: 0.100000
 P68: 0.100000
 P69: 0.100000
 P70: 0.100000
 P71: 0.100000
 P72: 0.100000
 P73: 0.100000
 P74: 0.100000
 P75: 0.100000
 P76: 0.100000
 P77: 0.100000
 P78: 0.100000
 P79: 0.100000
 P80: 0.100000
 P81: 0.100000
 P82: 0.100000
 P83: 0.100000
 P84: 0.100000
 P85: 0.100000
 P86: 0.100000
 P87: 0.100000
 P88: 0.100000
 P89: 0.100000
 P90: 0.100000
 P91: 0.100000
 P92: 0.100000
 P93: 0.100000
 P94: 0.100000
 P95: 0.100000
 P96: 0.100000
 P97: 0.100000
 P98: 0.100000
 P99: 0.100000
 P100: 0.100000

[illegible]

\$55

Current Data Parameters
NAME: 23
EXPNO: 1
PROCNO: 1
P2 - Acquisition Parameters
Date_Time: 20070611 11:37
Time: 11:37
INSTRUM: spect
PROBHD: 5 mm QNP 152/11
PULPROG: zgpg30
SOLVENT: CDCl3
D1: 1.50
D2: 0.05
D3: 0.05
SFO: 612.829 MHz
F2: 100.626130 MHz
AQ: 0.58560000 sec
RG: 61.716
DE: 4.50 uSVC
D1: 1.0000000 sec
P1: 9.86 uSVC
SFO: 500.1360000 MHz
SFO: 200.1320000 MHz
PC: 1.00
P2 - Processing parameters
SI: 32768
SF: 500.1360000 MHz
WDW: EM
SSB: 0
LB: 0.30 Hz
GB: 0
PC: 1.00
SFO: 500.1360000 MHz
F2: 100.626130 MHz
F1: 200.1320000 MHz
F0: 0.0000000 MHz
P2: 1.0000000 sec
P1: 0.0000000 sec
P0: 0.0000000 sec
RG: 61.716
DE: 4.50 uSVC
SFO: 500.1360000 MHz
SFO: 200.1320000 MHz
PC: 1.00

23

Current Data Parameters
NAME: 245C
PROCNO: 1

F2 - Acquisition Parameters

Date: SS0915
Time: 16:45
INSTRUM: spect
PROBHD: 5 mm
PULPROG: zgpg30
TD: 65536
SOLVENT: CDCl3
NS: 144
DS: 2
SWH: 19607.844 Hz
FIDRES: 0.59592 Hz
AQ: 0.32000000 sec
RG: 1.2000000
GB: 11.000000
PC: 300.000000 sec
PR: 0.03000000 sec
DE: 1.00000000
TE: 300.2 K
D1: 1.00000000 sec
DELTA: 0.00000000 sec
CHRGPRG: mw1215
P3: 300.000000 MHz
P2: 100.000000 MHz
NUC1: 13C
NUC2: 1H
PL1: -6.00 dB
PL2: -6.00 dB
PL3: 19.00 dB
PL4: 19.00 dB
DE: 4.50 dB
SFO1: 75.478721 MHz
SFO2: 101.626181 MHz
MAG1: 13C
MAG2: 1H
PC: 0.70

F2 - Processing parameters

SI: 32768
SF: 75.46377250 MHz
WDW: EM
SSB: 0
LB: 1.00 Hz
GB: 0
PC: 0.70
F2 - NMR plot parameters
C2: 245.000000 cm
F1P: 245.000000 ppm
F1: 88112.80 Hz
F2P: 154.000000 ppm
F2: -754.66 Hz
PRACH: 10.000000 ppm/cm
HZCM: 754.67469 Hz/cm

210.274
ppm

177.943

145.224
138.206
132.510
128.767
128.314
127.668
127.532
112.356

94.447
93.866
78.038
77.754
77.497
77.072
76.648
76.429
73.992
72.848
72.742
71.915
71.656
67.071
58.960
56.354
39.294
33.754
30.970
29.636
27.547
25.884
19.913
17.121

23

24a

0.000
1.026
24.592
27.214
31.230
33.760
37.889
41.998

54.857
61.492
72.537
73.393
76.601
77.024
77.225
77.446
82.400
93.275

127.648
127.752
128.357
137.930

209.474
211.709
211.709

Current Data Parameters
NAME 4.135a
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20010223
Time 14:35
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 65536
FIDRES 0.39000
SOLVENT DMS-D6
NS 25440
DS 4
SWH 22075.736 Hz
F2 500.136000 MHz
AQRES 0.860000 MHz
RG 11395.2
RG 0.725984 sec
DE 25.000 uV
TE 300.0 K
FID 1.00000000 sec
d11 0.03000000 sec
d12 0.00000000 sec
----- CHANNEL f1 -----
NUC1 13C
P1 1.00 sec
PL1 -6.00 dB
SFO1 75.4767751 MHz
----- CHANNEL f2 -----
CPDPRG2 waltz16
PULPROG zgpg30
TD 65536
FIDRES 0.39000
P2 100.00 uV
PL2 -6.00 dB
PL3 -6.00 dB
PL4 -6.00 dB
PL5 -6.00 dB
PL6 -6.00 dB
SFO2 300.1310005 MHz
F2 - Processing parameters
SI 32768
SF 500.136000 MHz
WDW EM
SSB 0
GB 0
PC 0.70
F2 500.136000 MHz
F1 25.000 MHz
FIP 25.000 MHz
F1 17357.58 Hz
F2 17357.58 Hz
WDW EM
SSB 0
PC 0.70
PUNCH 9.000000 cm/s
H2O 724.45048 Hz/cm

Integral

ppm

Current Data Parameters
NAME: r1998ch217a
EXPNO: 10
PROCNO: 1

F2 - Acquisition Parameters
Date_ : 20021120
Time : 09:21:40
INSTRUM : gnu300
PROBHD : 5 mm QNP 1H/1
PULPROG : zgpg30
TD : 32768
SOLVENT : CDCl3
OS : 0
SHE : 6172.839 Hz
F2 - Processing parameters
SI : 300.1300713 MHz
SF : 300.1300713 MHz
WDW : EM
SSB : 0
LB : 0.30 Hz
GB : 0
PC : 1.00
SD MEAS g101 parameters
CA : 26.25 cm
CF : 0.15 Hz
F1 : 270.131 Hz
F2 : 0.500 MHz
PCPDG : 0.15007 Hz
H2OCH : 108.11648 Hz/Hz

***** CHANNEL f1 *****
NUC1 : 1H
P1 : 9.80 uSec
PL1 : 0.00 dB
SFO1 : 300.1300713 MHz

Current Data Parameters
NAME 26a
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20081115
Time 16.10
INSTRUM spect
PROBHD 5mm
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 16
DS 1
SWH 7485.030 Hz
FIDRES 0.722625 Hz
AQ 2.1588250 sec
RG 1024
DM 65.800 uSsec
DE 4.50 uSsec
TE 300.0 K
D1 1.00000000 sec
===== CHANNEL f1 =====
NUC1 1H
P1 10.00 uSsec
PL1 -6.00 dB
SFO1 500.1315202 MHz
F2 - Processing parameters
SI 32768
SF 500.1300239 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
U0 NMR plot parameters
AQ 2.1588250 sec
FID 10.500 dB
F1 5251.37 Hz
F2 -0.5000 ppm
F2 250.07 Hz
NUC1 1H
AQCN 220.07122 Hz/cm

Current Data Parameters
 Name: 26a
 EXPNO: 3
 PROCNO: 1
 F2 - Acquisition Parameters
 Date_ : 20061110
 Time : 6.39
 INSTRUM: gpc500
 PROBHD: BBO 5mm
 PULPROG: zgpg30
 SOLVENT: CDCl3
 NS: 709
 DS: 4
 SH: 31446.541 Hz
 FIDRES: 0.959672 Hz
 AQ: 0.8210512 sec
 RG: 1032.5
 DE: 1.900 usec
 TE: 300.0 K
 D1: 3.00000000 sec
 d11: 0.03000000 sec
 ===== CHANNEL f1 =====
 NUC1: 13C
 P1: 8.20 usec
 PL1: 0.00 dB
 SFO1: 125.770204 MHz
 ===== CHANNEL f2 =====
 NUC2: 1H
 P2: 12.00 usec
 PL2: -6.00 dB
 SFO2: 500.1315000 MHz
 F2 - Processing parameters
 SI: 32768
 SF: 125.7577984 MHz
 DS: 4
 SSF: 0
 LB: 1.00 Hz
 GB: 0
 PC: 1.00
 1D NMR plot parameters
 CX: 25.00 cm
 FIP: 220.000 dBm
 F1P: 125.7577984 MHz
 F2P: 500.1315000 MHz
 PRNCH: 9.000000 ppm/cm
 HZCH: 1131.85007 Hz/cm

Current Data Parameters
 NAME az25chr11
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20001114
 Time 2.57
 INSTRUM drx500
 PROBHD BBO 5mm
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 8
 DS 1
 SWH 7485.036 Hz
 FIDRES 0.180524 Hz
 AQ 2.188924 sec
 RG 456 1
 DN 66.800 USEC
 DE 4.50 USEC
 TE 300.0 K
 D1 1.00000000 sec

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

NUC1 H
 P1 10.00 USEC
 PL1 -6.00 dB
 SFO1 500.1324006 MHz

F2 - Processing parameters

SI 32768
 SF 500.130436 MHz
 MD 0
 SS 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 25.00 cm
 F1P 10.500 ppm
 F1 5231.37 Hz
 F2P -0.500 ppm
 F2 -250.07 Hz
 PRGCM 0.44000 ppm/cm
 RECN 250.05721 Hz/cx

26b

Current Data Parameters
 EXPNO 1
 PROCNO 1
 F2 - Acquisition Parameters
 Da 300.134
 Time 3.07
 INSTRUM gpr-500
 PROBNM BBO 5mm
 PULPROG zgpg30
 TO 32768
 SOLVENT CDCl3
 NS 350
 SWH 31446.541 Hz
 FIDRES 0.559612 Hz
 AQ 0.5210612 sec
 RG 778.5
 DN 15.500 uSec
 DE 4.50 uSec
 TE 300.2 K
 D1 3.00000000 sec
 D11 0.03000000 sec
 ***** CHANNEL f1 *****
 NUC1 13C
 P1 120.00 uSec
 PL1 0.00 dB
 SFO1 125.761524 MHz
 ***** CHANNEL f2 *****
 NUC2 1H
 P2 100.00 uSec
 PL2 -6.00 dB
 SFO2 500.1314500 MHz
 F2 - Processing parameters
 SI 32768
 SF 125.7577854 MHz
 WHW 64
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.00
 10 MHz plot parameters
 CX 25.00 cm
 F1 2706.51 um
 F1 2706.51
 F2 500.1314500 um
 F2 -528.79 Hz
 SFOCH 9.000000000 MHz/cm
 NUCN 1131.65007 Hz/cm

29a + 29b

30

NAME: 30-13C-NMR-2
 EXPNO: 2
 PROCNO: 1
 F2 - Acquisition Parameters
 Date_Time: 20130627
 Time: 11:03
 File: 30-13C-NMR-2
 INSTRUM: spect
 P1: 12.00
 FIDRES: 0.090333
 AQ: 0.000100
 RG: 327.50
 DQ: 0.000000
 SFO: 125.760
 A2: 1.00
 F2 - Processing parameters
 Date_Time: 20130627
 Time: 11:03
 File: 30-13C-NMR-2
 INSTRUM: spect
 P1: 12.00
 FIDRES: 0.090333
 AQ: 0.000100
 RG: 327.50
 DQ: 0.000000
 SFO: 125.760
 A2: 1.00
 F2 - Processing parameters
 Date_Time: 20130627
 Time: 11:03
 File: 30-13C-NMR-2
 INSTRUM: spect
 P1: 12.00
 FIDRES: 0.090333
 AQ: 0.000100
 RG: 327.50
 DQ: 0.000000
 SFO: 125.760
 A2: 1.00

31

Current Data Parameters
NAME: 31
EXPNO: 1
PROCNO: 1
F2 - Acquisition Parameters
Date_ : 20110522
Time: 11.55
INSTRUM: spect
PROBHD: 5 mm QNP 1H/13
PULPROG: zgpg30
TD: 65536
SFO: 500.135098
AQ: 0.080000
RG: 327.50
WDW: EM
SSB: 0
LB: 3.00
GB: 0
PC: 1.00
F3 - Processing parameters
SI: 32768
SF: 500.135098 MHz
WDW: EM
SSB: 0
LB: 3.00
GB: 0
PC: 1.00
F4 - 13C NMR Parameters
SI: 32768
SF: 125.760150 MHz
WDW: EM
SSB: 0
LB: 3.00
GB: 0
PC: 1.00
F5 - 1H NMR Parameters
SI: 32768
SF: 500.135098 MHz
WDW: EM
SSB: 0
LB: 3.00
GB: 0
PC: 1.00
F6 - 13C NMR Parameters
SI: 32768
SF: 125.760150 MHz
WDW: EM
SSB: 0
LB: 3.00
GB: 0
PC: 1.00

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