Supporting Information

Neighboring Group Participation of 9-Anthracenylmethyl Group in Glycosylation: Preparation of Unusual C-Glycosides

Suvarn S. Kulkarni, Yi-Hung Liu, and Shang-Cheng Hung*

Department of Chemistry, National Tsing Hua University, Hsinchu 300, Taiwan, and Institute of Chemistry & Genomics Research Center, Academia Sinica, Taipei 115, Taiwan

Contents	Pages
List of Contents.....	S1-S2
Figure 1. The ORTEP Drawing of Compound 21-exo	S3
General Procedures.....	S3.
Experimental Procedure and characterization data for compound 2	S4.
One pot preparation and characterization data of compound 3	S4.
General one-pot procedure for compounds 4-7	S5.
Characterization data of compounds 4-7	S6-S8.
General procedure for preparation of C-glycosides.....	S8.
Characterization data of C-glycosides 15-exo, 17-exo, 19-exo, 21-exo, 23-exo, 25-exo, 27-exo, 29-exo, 31-exo, 33-exo	S9-S15
¹ H NMR spectrum of compound 2	S16.
¹³ C NMR spectrum of compound 2	S17.
¹ H NMR spectrum of compound 3	S18.
¹³ C NMR spectrum of compound 3	S19.
¹ H NMR spectrum of compound 4	S20.
¹³ C NMR spectrum of compound 4	S21.
¹ H NMR spectrum of compound 5	S22.
¹³ C NMR spectrum of compound 5	S23.
¹ H NMR spectrum of compound 6	S24.
¹³ C NMR spectrum of compound 6	S25.
¹ H NMR spectrum of compound 7	S26.
¹³ C NMR spectrum of compound 7	S27.
¹ H NMR spectrum of compound 15-exo	S28.
¹³ C NMR spectrum of compound 15-exo	S29.

^1H - ^{13}C COSY (HMQC) spectrum of compound 15-<i>exo</i>	S30.
^1H - ^1H COSY spectrum of compound 15-<i>exo</i> showing H' assignments.....	S31.
^1H - ^1H COSY spectrum of compound 15-<i>exo</i> showing H2'-H3' correlation.....	S32.
^1H - ^1H COSY spectrum of compound 15-<i>exo</i> showing H assignments.....	S33.
^1H - ^1H COSY spectrum of compound 15-<i>exo</i> showing entire correlation.....	S34.
^1H - ^1H NOESY spectrum of compound 15-<i>exo</i>	S35.
^1H NMR spectrum of compound 17-<i>exo</i>	S36.
^{13}C NMR spectrum of compound 17-<i>exo</i>	S37.
^1H NMR spectrum of compound 19-<i>exo</i>	S38.
^{13}C NMR spectrum of compound 19-<i>exo</i>	S39.
^1H NMR spectrum of compound 21-<i>exo</i>	S40.
^{13}C NMR spectrum of compound 21-<i>exo</i>	S41.
^1H NMR spectrum of compound 23-<i>exo</i>	S42.
^{13}C NMR spectrum of compound 23-<i>exo</i>	S43.
^1H NMR spectrum of compound 25-<i>exo</i>	S44.
^{13}C NMR spectrum of compound 25-<i>exo</i>	S45.
^1H NMR spectrum of compound 27-<i>exo</i>	S46.
^{13}C NMR spectrum of compound 27-<i>exo</i>	S47.
^1H NMR spectrum of compound 29-<i>exo</i>	S48.
^{13}C NMR spectrum of compound 29-<i>exo</i>	S49.
^1H NMR spectrum of compound 31-<i>exo</i>	S50.
^{13}C NMR spectrum of compound 31-<i>exo</i>	S51.
^1H NMR spectrum of compound 33-<i>exo</i>	S52.
^{13}C NMR spectrum of compound 33-<i>exo</i>	S53.

§ **FIGURE 1.** The ORTEP Drawing of Compound **21-exo**

§ Experimental Section

General Procedures. All reactions were conducted in flame-dried glassware, under dry nitrogen atmosphere. Dichloromethane was purified and dried from a safe purification system containing activated Al_2O_3 . Acetonitrile was freshly distilled from calcium hydride under nitrogen. Anhydrous *N,N*-dimethylformamide, pyridine, and methanol were used as purchased. All reagents obtained from commercial sources were used without purification, unless otherwise mentioned. Flash column chromatography was carried out on Silica Gel 60 (230-400 mesh, E. Merck). TLC was performed on pre-coated glass plates of Silica Gel 60 F254 (0.25 mm, E. Merck); detection was executed by spraying with a solution of $\text{Ce}(\text{NH}_4)_2(\text{NO}_3)_6$ (0.5 g), $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24}$ (24 g) and H_2SO_4 (28 mL) in water (500 mL) and subsequent heating on a hot plate. Melting points were determined with a capillary apparatus and are uncorrected. Optical rotations were measured at 589 nm (Na) at $\sim 25^\circ\text{C}$. ^1H , ^{13}C NMR, DEPT, ^1H - ^1H COSY, ^1H - ^{13}C COSY, and NOESY spectra were recorded with 400 and 500 MHz instruments. Chemical shifts are in ppm from Me_4Si , generated from the CDCl_3 lock signal at δ 7.24. IR spectra were taken with a FT-IR spectrometer using NaCl plates. Mass spectra were obtained in FAB mode.

Compound 2. To a suspension of tetraol **1** (3.10 g, 10.80 mmol) and ($\pm$)-camphorsulfonic acid (CSA) (0.63 g, 2.7 mmol) in anhydrous acetonitrile (30 mL) was added benzaldehyde dimethyl acetal (1.95 mL, 13.0 mmol) in a dropwise manner at RT and the mixture was kept stirring overnight. The reaction was quenched with triethylamine (0.3 mL), the solvents were evaporated under reduced pressure, and the resulting residue was recrystallized in ethanol to get the 4,6-*O*-benzylidenated 2,3-diol as a white solid (3.64 g, 91%).

This diol (1.0 g, 2.71 mmol) was dissolved in DMF (10 mL) and the solution was stirred at ice-bath temperature. 60 % Sodium hydride in mineral oil (271 mg, 11.30 mmol) and benzyl bromide (0.71 mL, 5.96 mmol) were sequentially added to the solution, the ice-bath was removed, and the mixture was kept stirring overnight. Water (4 mL) was added to quench the reaction, and the mixture was extracted with EtOAc (3 x 30 mL). The combined organic layers were washed with brine, dried over MgSO_4 , filtered, and concentrated *in vacuo*. The residue was first rinsed with hexane (3 x 10 mL) to remove excess reagents and oil and further purified by recrystallization in ethanol to afford compound **2** as a white solid (1.35 g, 90%). $[\alpha]_D^{27} -32.3$ (c 1.4, CHCl_3); mp 136-137 $^\circ\text{C}$; IR (CHCl_3) ν 3071, 3035, 2872, 1602, 1496, 1454, 1379, 1365, 1170, 1092, 1028, 805, 745, 695 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3) δ 7.49-7.46 (m, 2H, ArH), 7.44 (d, $J = 8.0$ Hz, 2H, ArH), 7.43-7.39 (m, 2H, ArH), 7.39-7.24 (m, 11H, ArH), 7.11 (d, $J = 8.0$ Hz, 2H, ArH), 5.58 (s, 1H, benzylidene-H), 4.93, 4.78 (ABq, $J = 11.2$ Hz, 2H, PhCH_2), 4.87, 4.81 (ABq, $J = 10.3$ Hz, 2H, PhCH_2), 4.69 (d, $J = 9.8$ Hz, 1H, H-1), 4.38 (dd, $J = 5.0, 10.4$ Hz, 1H, H-6eq), 3.82 (t, $J = 8.4$ Hz, 1H, H-3), 3.80 (t, $J = 10.4$ Hz, 1H, H-6ax), 3.68 (t, $J = 9.5$ Hz, 1H, H-4), 3.48 (t, $J = 8.4, 9.8$ Hz, 1H, H-2), 3.45 (dt, $J = 5.0, 9.5$ Hz, 1H, H-5), 2.34 (s, 3H, ArCH_3); ^{13}C NMR (125 MHz, CDCl_3) δ 138.3 (C), 138.2 (C), 138.1 (C), 137.3 (C), 133.0 (CH), 129.7 (CH), 129.1 (C), 128.4 (CH), 128.23 (CH), 128.18 (CH), 128.1 (CH), 127.8 (CH), 127.7 (CH), 126.0 (CH), 101.1 (CH), 88.5 (CH), 83.0 (CH), 81.5 (CH), 80.4 (CH), 75.8 (CH_2), 75.3 (CH_2), 70.2 (CH), 68.7 (CH_2), 21.1 (ArCH_3); HRMS (FAB, M^+) calcd for $\text{C}_{34}\text{H}_{34}\text{O}_5\text{S}$ 554.2127, found 554.2124.

Compound 3. ($\pm$)-Camphorsulfonic acid (232 mg, 1.0 mmol) and benzaldehyde dimethyl

acetal (0.73 mL, 4.83 mmol) were sequentially added to a suspension of tetraol **1** (1.15 g, 4.02 mmol) in anhydrous acetonitrile (12 mL) at RT. The reaction mixture became clear at this stage, and the solution was stirred overnight. The reaction flask was immersed in an ice-bath, and dichloromethane (12 mL) and triethylamine (9.2 mL, 66 mmol) were consecutively added to the mixture. After 20 min, chlorotrimethylsilane (2.0 mL, 16 mmol) was slowly added to the reaction solution, the ice-bath was removed, and the reaction was kept stirring at room temperature for 8 h. The mixture was evaporated under reduced pressure, the residue was diluted with hexane (20 mL), and the salt was filtered followed by wash with hexane. The filtrate was concentrated *in vacuo*, and the resulting residue was recrystallized in ethanol to provide **3** as a white solid (1.50 g, 72%). $[\alpha]_D^{27}$ -48.0 (*c* 2.0, CHCl₃); mp 97-98 °C; IR (CHCl₃) ν 3035, 2964, 2893, 1492, 1454, 1383, 1312, 1248, 1145, 1092, 1028, 989, 875, 840, 751, 698 cm⁻¹; ¹H NMR (500 MHz, CDCl₃) δ 7.48-7.42 (m, 2H, ArH), 7.39 (d, *J* = 8.0 Hz, 2H, ArH), 7.37-7.32 (m, 3H, ArH), 7.11 (d, *J* = 8.0 Hz, 2H, ArH), 5.46 (s, 1H, benzylidene-H), 4.60 (d, *J* = 9.7 Hz, 1H, H-1), 4.30 (dd, *J* = 4.6, 10.4 Hz, 1H, H-6eq), 3.74 (t, *J* = 10.4 Hz, 1H, H-6ax), 3.71 (t, *J* = 7.8 Hz, 1H, H-3), 3.56 (dd, *J* = 7.8, 9.7 Hz, 1H, H-2), 3.44 (t, *J* = 9.7, 1H, H-4), 3.41 (dt, *J* = 4.6, 9.7 Hz, 1H, H-5), 2.33 (s, 3H, ArCH₃), 0.22 (s, 9H, Si(CH₃)₃), 0.06 (s, 9H, Si(CH₃)₃); ¹³C NMR (125 MHz, CDCl₃) δ 137.6 (C), 137.1 (C), 132.1 (C), 130.3 (C), 129.7 (CH), 129.1 (CH), 128.1 (CH), 126.4 (CH), 102.0 (CH), 90.5 (CH), 81.1 (CH), 77.3 (CH), 75.7 (CH), 70.2 (CH), 68.7 (CH₂), 21.1 (ArCH₃), 1.2 (Si(CH₃)₃), 0.9 (Si(CH₃)₃); HRMS (FAB, M⁺) calcd for C₂₆H₃₈O₅Si₂S 518.1979, found 518.1968.

General Procedure for One-pot O3-Benzylation and Subsequent O2-Arylmethylation

TMSOTf (5 μ L, 0.03 mmol) was added to a mixture of the bis-TMS ether **3** (0.1 g, 0.19 mmol), benzaldehyde (21 μ L, 0.20 mmol), and freshly dried 3Å molecular sieves (0.15 g) in dichloromethane (2 mL) at -78 °C under nitrogen. The solution was stirred at the same temperature for 1 h, triethylsilane (34 μ L, 0.21 mmol) was added to the mixture, and the reaction was kept stirring for another 3 h. A 1 M solution of tetra-*n*-butylammonium fluoride in THF (0.39 mL, 0.39 mmol) was added to the reaction mixture, and the resulting solution was further stirred for 1.5 h at RT. After the completion of desilylation, as indicated by TLC, the reaction flask was immersed in an ice-bath, and DMF (2 mL) and 60 % sodium hydride in mineral oil (23 mg, 0.96 mmol) were sequentially added to the solution. After 20 min, arylmethyl halide (0.39 mmol) was added to the mixture, the ice-bath was removed, and the whole solution was kept stirring overnight. Water (0.1 mL) was added to quench the reaction, the mixture was diluted with EtOAc, and the solution was filtered through a pad of celite. The organic phase was washed with water, dried over MgSO₄, filtered, and concentrated *in vacuo*. The residue was first rinsed with hexane (3 x 5 mL) to remove excess reagents and oil and further purified by flash column chromatography (EtOAc/Hex) or recrystallization to furnish the products **4-7** in 71-82% yields.

(Table 1).

Compound 4. $[\alpha]_D^{26}$ -14.0 (*c* 2.0, CHCl₃); mp 133-134 °C; IR (CHCl₃) ν 3028, 2872, 1613, 1510, 1493, 1457, 1369, 1300, 1248, 1174, 1089, 1032, 809, 748, 699 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 7.49-7.44 (m, 2H, ArH), 7.42 (d, *J* = 8.0 Hz, 2H, ArH), 7.39-7.25 (m, 10H, ArH), 7.10 (d, *J* = 8.0 Hz, 2H, ArH), 6.87 (dd, *J* = 4.8, 9.4 Hz, 1H, ArH), 6.86 (d, *J* = 8.7 Hz, 1H, ArH), 5.56 (s, 1H, benzyldiene-H), 4.92, 4.78 (ABq, *J* = 11.2 Hz, 2H, PhCH₂), 4.79, 4.73 (ABq, *J* = 9.9 Hz, 2H, ArCH₂), 4.66 (d, *J* = 9.8 Hz, 1H, H-1), 4.36 (dd, *J* = 5.0, 10.5 Hz, 1H, H-6eq), 3.84-3.74 (m, 2H, H-6, H-3), 3.80 (s, 3H, OCH₃), 3.66 (t, *J* = 9.6 Hz, 1H, H-4), 3.46 (dd, *J* = 8.4, 9.8 Hz, 1H, H-2), 3.42 (dt, *J* = 5.0, 9.6 Hz, 1H, H-5), 2.33 (s, 3H, ArCH₃); ¹³C NMR (100 MHz, CDCl₃) δ 159.4 (C), 138.4 (C), 138.1 (C), 137.3 (C), 133.0 (CH), 130.3 (C), 129.9 (CH), 129.8 (CH), 129.2 (C), 128.9 (CH), 128.4 (CH), 128.2 (CH), 128.1 (CH), 127.7 (CH), 126.0 (CH), 113.8 (CH), 101.1 (CH), 88.6 (CH), 83.1 (CH), 81.5 (CH), 80.1 (CH), 75.5 (CH₂), 75.3 (CH₂), 70.2 (CH), 68.7 (CH₂), 55.3 (OCH₃), 21.1 (ArCH₃); HRMS (FAB, MH⁺) calcd for C₃₅H₃₇O₆S 585.2311, found 585.2308.

Compound 5. $[\alpha]_D^{27}$ -21.5 (*c* 3.3, CHCl₃); mp 159-160 °C; IR (CHCl₃) ν 3028, 2872, 1596, 1489, 1450, 1375, 1270, 1170, 1096, 1071, 1031, 982, 968, 776, 698 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 8.34 (d, *J* = 8.5 Hz, 1H, ArH), 7.85 (d, *J* = 8.7, 1H, ArH), 7.83 (d, *J* = 8.0, 1H, ArH), 7.52-7.46 (m, 6H, ArH), 7.46-7.42 (m, 2H, ArH), 7.42-7.31 (m, 5H, ArH), 7.30-7.24 (m, 3H, ArH), 7.12 (d, *J* = 7.9 Hz, 2H, ArH), 5.60 (s, 1H, benzyldiene-H), 5.42, 5.20 (ABq, *J* = 10.3 Hz, 2H, ArCH₂), 4.97, 4.77 (ABq, *J* = 11.2 Hz, 2H, PhCH₂), 4.74 (d, *J* = 9.8 Hz, 1H, H-1), 4.39 (dd, *J* = 5.0, 10.4 Hz, 1H, H-6eq), 3.85 (dd, *J* = 8.4, 9.6 Hz, 1H, H-3), 3.82 (t, *J* = 10.4 Hz, 1H, H-6ax), 3.74 (t, *J* = 9.6 Hz, 1H, H-4), 3.63 (dd, *J* = 8.4, 9.8 Hz, 1H, H-2), 3.45 (dt, *J* = 5.0, 9.6 Hz, 1H, H-5), 2.34 (s, 3H, ArCH₃); ¹³C NMR (100 MHz, CDCl₃) δ 138.4 (C), 138.2 (C), 137.2 (C), 133.77 (C), 133.74 (C), 132.9 (CH), 131.8 (C), 129.8 (CH), 129.3 (C), 128.9 (CH), 128.8 (CH), 128.4 (CH), 128.3 (CH), 128.2 (CH), 127.9 (CH), 127.6 (CH), 127.1 (CH), 126.2 (CH),

126.0 (CH), 125.7 (CH), 125.3 (CH), 124.6 (CH), 101.1 (CH), 88.7 (CH), 83.1 (CH), 81.5 (CH), 80.4 (CH), 75.2 (CH₂), 74.0 (CH₂), 70.1 (CH), 68.7 (CH₂), 21.1 (ArCH₃); HRMS (FAB, MH⁺) calcd for C₃₈H₃₇O₅S 605.2362, found 605.2363.

Compound 6. [α]_D²⁷ -4.7 (*c* 3.4, CHCl₃); mp 140-141 °C; IR (CHCl₃) ν 3028, 2866, 1599, 1492, 1450, 1369, 1270, 1170, 1088, 1028, 989, 812, 748, 698 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 7.86-7.78 (m, 4H, ArH), 7.55 (dd, *J* = 1.5, 8.4 Hz, 1H, ArH), 7.52-7.42 (m, 6H, ArH), 7.42-7.32 (m, 5H, ArH), 7.31-7.25 (m, 3H, ArH), 7.15 (d, *J* = 7.9 Hz, 2H, ArH), 5.59 (s, 1H, benzyldiene-H), 5.04, 4.98 (ABq, *J* = 10.5 Hz, 2H, ArCH₂), 4.96, 4.79 (ABq, *J* = 11.2 Hz, 2H, PhCH₂), 4.73 (d, *J* = 9.8 Hz, 1H, H-1), 4.39 (dd, *J* = 5.0, 10.4 Hz, 1H, H-6eq), 3.86 (dd, *J* = 8.5, 9.6 Hz, 1H, H-3), 3.82 (t, *J* = 10.4 Hz, 1H, H-6ax), 3.71 (t, *J* = 9.6 Hz, 1H, H-4), 3.55 (dd, *J* = 8.5, 9.8 Hz, 1H, H-2), 3.47 (dt, *J* = 5.0, 9.6 Hz, 1H, H-5), 2.32 (s, 3H, ArCH₃); ¹³C NMR (100 MHz, CDCl₃) δ 138.3 (C), 138.2 (C), 137.3 (C), 135.6 (C), 133.3 (C), 133.1 (C), 133.0 (CH), 129.8 (CH), 129.2 (C), 129.0 (CH), 128.4 (CH), 128.2 (CH), 128.10 (CH), 128.07 (CH), 127.95 (CH), 127.73 (CH), 127.67 (CH), 126.8 (CH), 126.2 (CH), 126.00 (CH), 125.97 (CH), 125.87 (CH), 101.1 (CH), 88.5 (CH), 83.1 (CH), 81.5 (CH), 80.4 (CH), 75.9 (CH₂), 75.3 (CH₂), 70.2 (CH), 68.7 (CH₂), 21.1 (ArCH₃); HRMS (FAB, MH⁺) calcd for C₃₈H₃₇O₅S 605.2362, found 605.2363.

Compound 7. [α]_D²⁷ -31.7 (*c* 1.5, CHCl₃); mp 197-198 °C; IR (CHCl₃) ν 3028, 2887, 1650, 1616, 1494, 1450, 1371, 1342, 1268, 1214, 1165, 1082, 1028, 989, 967, 731, 692 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 8.61 (d, *J* = 8.9 Hz, 2H, ANT-H), 8.44 (s, 1H, ANT-H), 7.97 (d, *J* = 8.4 Hz, 2H, ANT-H), 7.49 (d, *J* = 8.1 Hz, 2H, ArH), 7.45-7.25 (m, 12H, ArH), 7.24-7.22 (m, 2H, ArH), 7.12 (d, *J* = 8.1 Hz, 2H, ArH), 5.99, 5.73 (ABq, *J* = 9.8 Hz, 1H, ArCH₂), 5.58 (s, 1H, benzyldiene-H), 5.03, 4.80 (ABq, *J* = 11.7 Hz, 1H, PhCH₂), 4.75 (d, *J* = 8.9 Hz, 1H, H-1), 4.37

(dd, $J = 5.0, 10.5$ Hz, 1H, H-6a), 3.84-3.78 (m, 4H, H-2, H-3, H-4, H-6b), 3.48-3.38 (m, 1H, H-5), 2.34 (s, 3H, ArCH₃); ¹³C NMR (100 MHz, CDCl₃) δ 138.8 (C), 138.3 (C), 137.2 (C), 132.8 (CH), 131.5 (C), 131.1 (C), 129.9 (CH), 129.5 (C), 129.0 (CH), 128.8 (CH), 128.6 (CH), 128.5 (C), 128.3 (CH), 128.2 (CH), 127.4 (CH), 127.1 (CH), 126.2 (CH), 126.0 (CH), 125.0 (CH), 124.9 (CH), 101.2 (CH), 89.2 (CH), 82.8 (CH), 82.0 (CH), 80.6 (CH), 74.7 (CH₂), 69.9 (CH), 68.7 (CH₂), 68.2 (CH₂), 21.2 (CH₃); HRMS (FAB, MH⁺) calcd for C₄₂H₃₉O₅S 655.2518, found 655.2517. Anal. Calcd for C₄₂H₃₈O₅S: C, 77.04; H, 5.85. Found: C, 76.58; H, 5.88.

General Procedure for the Synthesis of Unusual C-Glycosides

A mixture of glycosyl donor **7** (1.1 equiv), acceptor (1 equiv) and freshly dried 3 Å MS (twice the amount of the donor), in dichloromethane (2 mL/100 mg donor) was stirred for 30 min at RT under nitrogen. A solution of DMTST (4 equiv) in dichloromethane (2 mL/ 100 mg scale) was added to the reaction in a dropwise manner at ice bath temperature. The mixture was stirred at 0°C throughout and upon completion (as per Table 2) was quenched with triethylamine, diluted with dichloromethane and filtered on a pad of celite. The organic layer was washed with water, dried over anhydrous MgSO₄, and concentrated *in vacuo* to give the crude product, which was purified by flash column chromatography (EA/Hexane) to provide the C-glycosides in good yields (Table 3).

Compound 15-*exo*. $[\alpha]_D^{28} +21.4$ (c 1, CHCl₃); mp 83-85 °C; IR (CHCl₃) ν 3028, 2905, 1640, 1489, 1452, 1368, 1211, 1096, 1069, 1028, 918, 745, 693 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 7.66 (d, $J = 7.7$ Hz, 1H, ArH), 7.46-7.40 (m, 3H, ArH), 7.39-7.32 (m, 7H, ArH), 7.32-7.26 (m, 13H, ArH), 7.26-7.22 (m, 3H, ArH), 7.22-7.19 (m, 4H, ArH), 7.17-7.12 (m, 2H, ArH), 5.27 (s, 1H, benzylidene-H), 5.20 (s, 1H, Ar₂CH-O), 4.91 (d, $J = 10.8$ Hz, 1H, PhCH₂), 4.88 (d, $J = 9.0$ Hz, 1H, CCH₂-O), 4.76 (d, $J = 10.8$ Hz, 1H, PhCH₂), 4.75 (d, $J = 11.1$ Hz, 1H, PhCH₂), 4.70 (d, $J = 12.0$ Hz, 1H, PhCH₂), 4.69 (d, $J = 3.4$ Hz, 1H, H-1'), 4.64, 4.60 (ABq, $J = 12.0$ Hz, 2H, PhCH₂), 4.56 (d, $J = 12.0$ Hz, 1H, PhCH₂), 4.53 (d, $J = 3.5$ Hz, 1H, H-1), 4.44 (d, $J = 11.1$ Hz, 1H, PhCH₂), 4.41 (d, $J = 9.0$ Hz, 1H, CCH₂-O), 4.33 (d, $J = 3.4$ Hz, 1H, H-2'), 4.04 (t, $J = 5.0$ Hz, 1H, H-6a'), 4.01 (d, $J = 7.0$ Hz, 1H, H-3'), 3.92 (t, $J = 9.3$ Hz, 1H, H-3), 3.77 (dd, $J = 5.0, 10.0$ Hz, 1H, H-5'), 3.75-3.70 (m, 1H, H-5), 3.66 (dd, $J = 1.7, 11.0$ Hz, 1H, H-6a), 3.59 (dd,

$J = 5.5, 11.0$ Hz, 1H, H-6b), 3.51 (dd, $J = 7.0, 10.0$ Hz, 1H, H-4'), 3.44 (dd, $J = 3.5, 9.3$ Hz, 1H, H-2), 3.37 (t, $J = 9.3$ Hz, 1H, H-4), 3.30 (s, 3H, OCH₃), 3.26 (t, $J = 10.0$ Hz, 1H, H-6b'); ¹³C NMR (100 MHz, CDCl₃) δ 141.8 (C), 138.7 (C), 138.3 (C), 138.2 (C), 137.9 (C), 137.6 (C), 137.5 (C), 136.7 (C), 136.3 (C), 130.0 (CH), 129.6 (CH), 128.9 (CH), 128.8 (CH), 128.40 (CH), 128.37 (CH), 128.3 (CH), 128.2 (CH), 128.1 (CH), 128.0 (CH), 127.9 (CH), 127.8 (CH), 127.6 (CH), 127.2 (CH), 127.1 (CH), 126.8 (CH), 126.6 (CH), 126.1 (CH), 101.0 (CH), 97.9 (CH), 81.9 (CH), 81.5 (CH), 80.94 (CH), 80.89 (CH), 80.1 (CH), 80.0 (CH), 78.5 (CH), 78.2 (CH), 75.7 (CH₂), 74.9 (CH₂), 73.4 (CH₂), 71.9 (CH₂), 71.5 (CH₂), 70.3 (CH), 70.0 (CH₂), 66.9 (CH₂), 64.6 (CH), 58.0 (C), 55.2 (CH₃); HRMS (FAB, MH⁺) calcd for C₆₃H₆₃O₁₁ 995.4370, found 995.4389.

Compound 17-exo. [α]_D²⁷ +17.7 (c 0.8, CHCl₃); IR (CHCl₃) ν 3025, 2912, 2866, 1653, 1453, 1372, 1215, 1098, 1074, 1054, 754, 700, 668 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 7.68 (d, $J = 7.7$ Hz, 1H, ArH), 7.47 (d, $J = 7.7$ Hz, 1H, ArH), 7.40-7.38 (m, 1H, ArH), 7.38-7.36 (m, 3H, ArH), 7.36-7.31 (m, 7H, ArH), 7.31-7.28 (m, 7H, ArH), 7.28-7.25 (m, 7H, ArH), 7.24-7.16 (m, 4H, ArH), 7.13 (dd, $J = 1.7, 7.7$ Hz, 2H, ArH), 5.12 (s, 1H, benzyldiene-H), 5.06 (s, 1H, Ar₂CH-O), 4.86 (d, $J = 9.3$ Hz, 1H, CCH₂-O), 4.78 (d, $J = 11.7$ Hz, 1H, PhCH₂), 4.75 (d, $J = 11.5$ Hz, 1H, PhCH₂), 4.72, 4.70 (ABq, $J = 12$ Hz, 2H, PhCH₂), 4.64, 4.61 (ABq, $J = 12.0$ Hz, 2H, PhCH₂), 4.61 (d, $J = 11.7$ Hz, 1H, PhCH₂), 4.58 (d, $J = 3.7$ Hz, 1H, H-1), 4.52 (d, $J = 3.3$ Hz, 1H, H-1'), 4.42 (d, $J = 9.3$ Hz, 1H, CCH₂-O), 4.37 (d, $J = 3.3$ Hz, 1H, H-2'), 4.15 (d, $J = 11.5$ Hz, 1H, PhCH₂), 4.04 (d, $J = 6.2$ Hz, 1H, H-3'), 4.01 (dd, $J = 5.0, 10.2$ Hz, 1H, H-6a'), 3.97 (dd, $J = 3.7, 9.7$ Hz, 1H, H-2), 3.86-3.75 (m, 4H, H-3, H-4, H-5, H-5'), 3.63 (dd, $J = 7.0, 8.7$ Hz, 1H, H-6a), 3.54-3.49 (m, 2H, H-4', H-6b), 3.26 (s, 3H, OCH₃), 3.15 (t, $J = 10.2$ Hz, 1H, H-6b'); ¹³C NMR (125 MHz, CDCl₃) δ 141.3 (C), 138.8 (C), 138.4 (C), 137.7 (C), 137.3 (C), 137.2 (C), 136.2 (C), 135.8 (C), 130.1 (CH), 129.8 (CH), 129.0 (CH), 128.9 (CH), 128.3 (CH), 128.3 (CH), 128.1 (CH), 128.0 (CH), 127.8 (CH), 127.6 (CH), 127.4 (CH), 127.0 (CH), 126.8 (CH), 126.6 (CH), 126.1 (CH), 101.1 (CH), 98.7 (CH), 81.5 (CH x 2), 80.4 (CH), 80.2 (CH), 79.0 (CH), 78.2 (CH), 76.2 (CH), 75.1 (CH), 74.8 (CH₂), 73.4 (CH₂), 72.9 (CH₂), 72.0 (CH₂), 71.6 (CH₂), 69.9 (CH₂), 69.4 (CH), 67.3 (CH₂), 64.4 (CH), 58.2 (C), 55.4 (CH₃); HRMS (FAB, MNa⁺) calcd for C₆₃H₆₂O₁₁Na 1017.4190, found 1017.4205.

Compound 19-*exo*. $[\alpha]_D^{27} +20.8$ (c 1.3, CHCl_3); IR (CHCl_3) ν 3029, 2905, 2867, 1603, 1496, 1454, 1369, 1211, 1095, 1070, 1027, 974, 750, 696 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 7.67 (d, $J = 7.2$ Hz, 1H, ArH), 7.45 (dd, $J = 1.4, 7.5$ Hz, 1H, ArH), 7.44-7.40 (m, 3H, ArH), 7.38 (dd, $J = 1.4, 7.1$ Hz, 1H, ArH), 7.36-7.31 (m, 6H, ArH), 7.31-7.29 (m, 3H, ArH), 7.28-7.21 (m, 15H, ArH), 7.19-7.16 (m, 2H, ArH), 7.07 (dt, $J = 0.9, 7.4$ Hz, 1H, ArH), 5.38 (s, 1H, $\text{Ar}_2\text{CH-O}$), 5.26 (s, 1H, benzylidene-H), 4.89 (d, $J = 9.2$ Hz, 1H, $\text{CCH}_2\text{-O}$), 4.79 (d, $J = 10.9$ Hz, 1H, PhCH_2), 4.73 (brs, 1H, H-1), 4.72 (d, $J = 3.2$ Hz, 1H, H-1'), 4.70, 4.69 (ABq, $J = 12.4$ Hz, 2H, PhCH_2), 4.66, 4.61 (ABq, $J = 12$ Hz, 2H, PhCH_2), 4.59 (s, 2H, PhCH_2), 4.47 (d, $J = 10.9$ Hz, 1H, PhCH_2), 4.41 (d, $J = 9.2$ Hz, 1H, $\text{CCH}_2\text{-O}$), 4.36 (dd, $J = 3.2$ Hz, 1H, H-2'), 4.01 (d, $J = 7.1$ Hz, 1H, H-3'), 4.00 (t, $J = 5.5$ Hz, 1H, H-6a'), 3.86-3.80 (m, 2H, H-3, H-5), 3.80-3.70 (m, 4H, H-6a, H-4', H-2, H-4), 3.67 (t, $J = 5.5$, 1H, H-6b), 3.53 (dd, $J = 7.0, 10.1$ Hz, 1H, H-5'), 3.27 (s, 3H, OCH_3), 3.19 (t, $J = 10.1$ Hz, 1H, H-6b'); ^{13}C NMR (125 MHz, CDCl_3) δ 141.9 (C), 138.7 (C), 138.6 (C), 138.4 (C), 138.0 (C), 137.6 (C), 137.4 (C), 137.1 (C), 136.4 (C), 130.3 (CH), 129.8 (CH), 128.9 (CH), 128.6 (CH), 128.3 (CH), 128.1 (CH), 127.9 (CH), 127.8 (CH), 127.6 (CH), 127.3 (CH), 127.0 (CH), 126.8 (CH), 126.5 (CH), 126.1 (CH), 101.0 (CH), 99.0 (CH), 81.5 (CH), 81.0 (CH), 80.8 (CH), 80.1 (CH), 79.4 (CH), 78.8 (CH), 75.3 (CH), 75.1 (CH), 74.9 (CH₂), 72.8 (CH₂), 72.2 (CH), 72.1 (CH₂), 72.0 (CH₂), 71.5 (CH₂), 70.0 (CH₂), 66.9 (CH₂), 64.5 (CH), 58.1 (C), 54.8 (CH₃); HRMS (FAB, MH^+) calcd for $\text{C}_{63}\text{H}_{63}\text{O}_{11}$ 995.4370, found 995.4365.

Compound 21-*exo*. $[\alpha]_D^{27} +2.5$ (c 1.8, CHCl_3); mp 210 – 211 $^\circ\text{C}$; IR (CHCl_3) ν 3023, 2908, 2866, 1645, 1482, 1453, 1375, 1215, 1095, 1064, 1025, 1000, 753, 696 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 7.78 (d, $J = 7.8$ Hz, 1H, ArH), 7.58-7.53 (m, 1H, ArH), 7.52-7.42 (m, 5H, ArH),

7.42-7.37 (m, 2H, ArH), 7.37-7.28 (m, 9H, ArH), 7.20-7.10 (m, 6H, ArH), 7.09-7.04 (m, 2H, ArH), 6.75-6.70 (m, 2H, ArH), 5.79 (s, 1H, Ar₂CH-O), 5.48 (s, 1H, benzyldiene-H), 5.45 (s, 1H, benzyldiene-H), 4.87 (d, $J = 9.2$ Hz, 1H, CCH₂-O), 4.72 (d, $J = 3.2$ Hz, 1H, H-1'), 4.65 (d, $J = 11.2$ Hz, 1H, PhCH₂), 4.43 (d, $J = 7.6$ Hz, 2H, H-1, CCH₂-O), 4.36-4.30 (m, 1H, H-6a), 4.35 (d, $J = 11.2$ Hz, 1H, PhCH₂), 4.33 (d, $J = 3.2$ Hz, 1H, H-2'), 4.27, 4.13 (ABq, $J = 12.6$ Hz, 2H, PhCH₂), 4.21 (dd, $J = 5.1, 10.6$ Hz, 1H, H-6a'), 3.93 (d, $J = 6.6$ Hz, 1H, H-3'), 3.87-3.78 (m, 2H, H-2, H-5'), 3.74 (t, $J = 10.0$ Hz, 1H, H-6b), 3.70-3.58 (m, 3H, H-3, H-4, H-4'), 3.58-3.48 (m, 1H, H-6b'), 3.56 (s, 3H, OCH₃), 3.37 (dt, $J = 4.9, 10.0$ Hz, 18.9 Hz, 1H, H-5); ¹³C NMR (100 MHz, CDCl₃) δ 141.7 (C), 138.7 (C), 137.7 (C), 137.5 (C), 137.3 (C), 137.2 (C), 137.1 (C), 136.6 (C), 130.2 (CH), 129.7 (CH), 128.89 (CH), 128.85 (CH), 128.7 (CH), 128.17 (CH), 128.13 (CH), 128.11 (CH), 127.6 (CH), 127.4 (CH), 127.34 (CH), 127.26 (CH), 127.23 (CH), 127.16 (CH), 127.0 (CH), 126.8 (CH), 126.1 (CH), 125.9 (CH), 105.1 (CH), 101.0 (CH x 2), 82.1 (CH), 81.6 (CH), 81.1 (CH), 80.4 (CH), 80.2 (CH), 80.1 (CH), 79.2 (CH), 77.7 (CH), 73.8 (CH₂), 71.5 (CH₂), 70.9 (CH₂), 70.2 (CH₂), 68.8 (CH₂), 65.5 (CH), 64.5 (CH), 58.2 (C), 57.0 (CH₃); HRMS (FAB, MH⁺) calcd for C₅₆H₅₅O₁₁ 903.3744, found 903.3741.

Compound 23-exo. [α]_D²³ -4.8 (c 1.8, CHCl₃); IR (CHCl₃) ν 3009, 2972, 2896, 1646, 1599, 1448, 1382, 1217, 1085, 759, 665 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 7.75 (d, $J = 7.8$ Hz, 1H, ArH), 7.48-7.44 (m, 2H, ArH), 7.44-7.36 (m, 9H, ArH), 7.36-7.31 (m, 3H, ArH), 7.31-7.25 (m, 2H, ArH), 7.22-7.15 (m, 6H, ArH), 7.12 (t, $J = 7.5$ Hz, 1H, ArH), 7.10-7.04 (m, 2H, ArH), 6.77-6.74 (m, 2H, ArH), 5.67 (s, 1H, Ar₂CH-O), 5.54 (s, 1H, benzyldiene-H), 5.42 (s, 1H, benzyldiene-H), 4.84 (d, $J = 9.2$ Hz, 1H, CCH₂-O), 4.76 (d, $J = 3.2$ Hz, 1H, H-1'), 4.62 (d, $J = 10.8$ Hz, 1H, PhCH₂), 4.41 (d, $J = 9.2$ Hz, 1H, CCH₂-O), 4.37 (d, $J = 7.4$ Hz, 1H, H-1), 4.37-4.30 (m, 2H, H-6a, H-2'), 4.30 (d, $J = 10.8$ Hz, 1H, PhCH₂), 4.28 (d, $J = 12.6$ Hz, 1H, PhCH₂), 4.17 (d, $J = 12.6$ Hz, 1H, PhCH₂), 4.12 (t, $J = 9.1$ Hz, 1H, H-3), 4.00 (dd, $J = 5.0, 10.5$ Hz, 1H, H-6a'), 3.91 (d, $J = 6.6$ Hz, 1H, H-3'), 3.76 (t, $J = 10.1$ Hz, 1H, H-6b), 3.75 (t, $J = 10.0$ Hz, 1H, H-5'), 3.68 (t, $J = 9.1$ Hz, 1H, H-4), 3.60 (dd, $J = 6.6, 10.0$ Hz, 1H, H-4'), 3.51-3.43 (m, 1H, H-5), 3.46 (s, 3H, OCH₃), 3.29 (t, $J = 10.5$ Hz, 1H, H-6b'), 3.26 (t, $J = 8.0$ Hz, 1H, H-2); ¹³C NMR (100 MHz, CDCl₃) δ 141.6 (C), 138.7 (C), 137.8 (C), 137.5 (C), 137.4 (C), 137.3 (C), 137.2 (C),

136.9 (C), 130.2 (CH), 129.6 (CH), 129.2 (CH), 128.8 (CH), 128.6 (CH), 128.3 (CH), 128.2 (CH), 128.1 (CH), 127.9 (CH), 127.6 (CH), 127.5 (CH), 127.4 (CH), 127.3 (CH), 127.2 (CH), 127.0 (CH), 126.9 (CH), 126.7 (CH), 126.4 (CH), 126.1 (CH), 105.6 (CH), 101.6 (CH), 100.9 (CH), 81.84 (CH), 81.75 (CH), 81.5 (CH), 81.1 (CH), 81.0 (CH), 80.1 (CH), 79.5 (CH), 77.8 (CH), 74.3 (CH₂), 71.3 (CH₂), 71.1 (CH₂), 70.1 (CH₂), 68.9 (CH₂), 65.9 (CH), 64.5 (CH), 58.2 (C), 57.3 (CH₃); HRMS (FAB, MH⁺) calcd for C₅₆H₅₅O₁₁ 903.3744, found 903.3741.

Compound 25-exo. $[\alpha]_D^{25} +42.5$ (*c* 1, CHCl₃); mp 98-99 °C; IR (CHCl₃) ν 3010, 2995, 2905, 1637, 1453, 1373, 1215, 1076, 1031, 759, 668 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 7.68 (d, *J* = 7.6 Hz, 1H, ArH), 7.50-7.40 (m, 5H, ArH), 7.39-7.25 (m, 15H, ArH), 7.24-7.20 (m, 2H, ArH), 5.52 (s, 1H, Ar₂CH-O), 5.45 (s, 1H, benzylidene-H), 4.91 (d, *J* = 9.0 Hz, 1H, CCH₂-O), 4.86 (d, *J* = 3.5 Hz, 1H, H-1'), 4.70-4.63 (m, 2H, PhCH₂), 4.62, 4.51 (ABq, *J* = 12.0 Hz, 2H, PhCH₂), 4.56 (d, *J* = 3.6 Hz, 1H, H-1), 4.41 (d, *J* = 9.0 Hz, 1H, CCH₂-O), 4.39 (d, *J* = 3.5 Hz, 1H, H-2'), 4.23 (dd, *J* = 5.5, 7.8 Hz, 1H, H-3), 4.19-4.08 (m, 3H, H-6a, H-4, H-4'), 4.05 (d, *J* = 7.2 Hz, 1H, H-3'), 3.84 (dt, *J* = 5.1, 9.8, 19.9 Hz, 1H, H-5), 3.73-3.64 (m, 3H, H-5', H-6a', H-6b'), 3.61 (dd, *J* = 3.6, 7.8 Hz, 1H, H-2), 3.44 (t, *J* = 9.8 Hz, 1H, H-6b), 1.28 (s, 3H, CCH₃), 1.22 (s, 3H, CCH₃); ¹³C NMR (100 MHz, CDCl₃) δ 141.9 (C), 138.2 (C), 138.0 (C), 137.6 (C), 137.5 (C), 136.4 (C), 135.9 (C), 130.0 (CH), 128.91 (CH), 128.87 (CH), 128.3 (CH), 128.20 (CH), 128.18 (CH), 127.76 (CH), 127.68 (CH), 127.64 (CH), 127.59 (CH), 127.55 (CH), 127.51 (CH), 127.3 (CH), 126.8 (CH), 126.7 (CH), 126.1 (CH), 109.2 (C), 101.2 (CH), 97.9 (CH), 81.12 (CH), 81.06 (CH x 2), 78.8 (CH), 78.1 (CH), 76.2 (CH), 73.8 (CH), 73.4 (CH₂), 73.2 (CH), 72.0 (CH₂), 71.9 (CH₂), 70.1 (CH₂), 69.6 (CH₂), 66.2 (CH), 64.8 (CH), 58.1 (C), 55.5 (CH₃), 27.9 (CH₃), 26.3 (CH₃); HRMS (FAB, MH⁺) calcd for C₅₂H₅₅O₁₁ 855.3744, found 855.3737.

Compound 27-exo. $[\alpha]_D^{25} +69.9$ (*c* 2.3, CHCl₃); mp 89-90 °C; IR (CHCl₃) ν 3019, 2971, 2896, 1721, 1646, 1603, 1448, 1373, 1212, 1099, 1028, 759, 665 cm⁻¹; ¹H NMR (500 MHz,

CDCl₃) δ 7.99-7.96 (m, 2H, ArH), 7.72 (d, J = 7.7 Hz, 1H, ArH), 7.54 (t, J = 7.4 Hz, 1H, ArH), 7.50 (d, J = 7.7 Hz, 1H, ArH), 7.48-7.32 (m, 13H, ArH), 7.32-7.24 (m, 5H, ArH), 7.16-7.12 (m, 3H, ArH), 7.00-6.96 (m, 2H, ArH), 5.48 (s, 1H, Ar₂CH-O), 5.47 (d, J = 1.8, Hz, 1H, H-1), 5.38 (s, 1H, benzylidene-H), 5.01 (dd, J = 1.8, 8.2 Hz, 1H, H-2), 4.88 (d, J = 9.4 Hz, 1H, CCH₂-O), 4.72, 4.64 (ABq, J = 11.9 Hz, 2H, PhCH₂), 4.48 (s, 2H, PhCH₂), 4.43 (d, J = 9.4 Hz, 1H, CCH₂-O), 4.42 (d, J = 3.0 Hz, 1H, H-1'), 4.36 (d, J = 3.0 Hz, 1H, H-2'), 4.21 (dd, J = 4.3, 8.2 Hz, 1H, H-4), 4.13 (t, J = 4.3 Hz, 1H, H-5), 4.08 (d, J = 6.5 Hz, 1H, H-3'), 4.02 (dd, J = 5.2, 10.6 Hz, 1H, H-6a), 3.97 (d, J = 7.7 Hz, 1H, H-6a'), 3.85 (t, J = 10.1 Hz, 1H, H-5'), 3.84 (t, J = 8.2 Hz, 1H, H-3), 3.64 (dd, J = 6.5, 10.1 Hz, 1H, H-4'), 3.56 (dd, J = 5.2, 6.9 Hz, 1H, H-6b), 3.14 (t, J = 10.1 Hz, 1H, H-6b'); ¹³C NMR (100 MHz, CDCl₃) δ 165.8 (C), 141.5 (C), 138.1 (C), 137.7 (C), 137.4 (C), 137.0 (C), 136.6 (C), 135.8 (C), 133.2 (CH), 129.8 (CH), 129.5 (C), 129.3 (CH), 129.1 (CH), 129.0 (CH), 128.42 (CH), 128.36 (CH), 128.20 (CH), 128.15 (CH), 128.0 (CH), 127.9 (CH), 127.8 (CH), 127.6 (CH), 127.5 (CH), 127.1 (CH), 126.10 (CH), 101.12 (CH), 99.3 (CH), 81.4 (CH), 81.2 (CH), 80.9 (CH), 80.3 (CH), 79.2 (CH), 79.0 (CH), 78.0 (CH), 76.8 (CH), 75.1 (CH₂), 73.8 (CH), 72.2 (CH₂), 71.7 (CH₂), 69.8 (CH₂), 65.4 (CH₂), 64.4 (CH), 58.1 (C); HRMS (FAB, MH⁺) calcd for C₅₅H₅₁O₁₁ 887.3431, found 887.3443.

Compound 29-exo. $[\alpha]_D^{26} +54.48$ (c 1.1, CHCl₃); IR (CHCl₃) ν 3049, 3018, 2908, 2103, 1721, 1646, 1601, 1495, 1482, 1451, 1373, 1272, 1215, 1119, 1099, 1072, 1051, 756, 699 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 7.97 (d, J = 8.0 Hz, 1H, ArH), 7.95 (d, J = 8.4 Hz, 1H, ArH), 7.72 (d, J = 8.0 Hz, 1H, ArH), 7.55 (t, J = 7.4 Hz, 1H, ArH), 7.52-7.38 (m, 7H, ArH), 7.38-7.34 (m, 5H, ArH), 7.34-7.25 (m, 10H, ArH), 7.13 (t, J = 7.4 Hz, 1H, ArH), 7.10 (t, J = 7.4 Hz, 1H, ArH), 5.69 (s, 1H, Ar₂CH-O), 5.45 (s, 1H, benzylidene-H), 4.84 (d, J = 9.3 Hz, 1H, CCH₂-O), 4.79 (d, J = 3.5 Hz, 1H, H-1), 4.71 (d, J = 11.2 Hz, 1H, PhCH₂), 4.65 (d, J = 3.4 Hz, 1H, H-1'), 4.64, 4.62 (ABq, J = 12.2 Hz, 2H, PhCH₂), 4.51 (d, J = 11.2 Hz, 1H, PhCH₂), 4.49 (d, J = 3.4 Hz, 1H, H-2'), 4.38 (d, J = 9.3 Hz, 1H, CCH₂-O), 4.32 (dd, J = 2.0, 12.1 Hz, 1H, H-6a), 4.14-4.03 (m, 4H, H6a', H6b, H-3', H-4), 3.94 (dd, J = 8.6, 10.0 Hz, 1H, H-3), 3.85-3.77 (m, 2H, H-4', H-5), 3.75 (dd, J = 6.5, 10.0 Hz, 1H, H-5'), 3.44 (dd, J = 3.5, 10.0 Hz, 1H, H-2), 3.37 (s, 3H, OCH₃), 3.36 (t, J = 10.0 Hz, 1H, H-6b'); ¹³C NMR (100 MHz, CDCl₃) δ 165.8 (C), 141.5 (C), 138.1 (C), 137.8 (C), 137.5 (C), 137.3 (C), 136.9 (C), 135.7 (C), 133.1 (CH), 130.1 (CH), 130.0 (CH), 129.6 (CH), 129.4 (CH), 129.3 (CH), 128.9 (CH), 128.4 (CH), 128.32 (CH), 128.30 (CH), 128.2 (CH), 128.0

(CH), 127.8 (CH), 127.66 (CH), 127.63 (CH), 127.5 (CH), 127.4 (CH), 127.3 (CH), 127.2 (CH), 126.1 (CH), 101.1 (CH), 98.4 (CH), 81.6 (CH), 81.5 (CH), 80.78 (CH), 80.77 (CH), 80.4 (CH), 78.2 (CH), 76.8 (CH), 75.0 (CH₂), 72.3 (CH₂), 72.0 (CH₂), 69.9 (CH₂), 69.1 (CH), 64.6 (CH), 64.3 (CH), 63.0 (CH₂), 57.8 (C), 55.2 (CH₃); HRMS (FAB, MH⁺) calcd for C₅₆H₅₄O₁₁N₃ 944.3758, found 944.3747.

Compound 31-exo. [α]_D²⁵ +32.9 (*c* 2.3, CHCl₃); mp 90-91 °C; IR (CHCl₃) ν 3065, 2982, 1602, 1494, 1480, 1453, 1371, 1215, 1119, 1072, 848, 752, 698, 668 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 7.71 (d, *J* = 7.7 Hz, 1H, ArH), 7.53-7.44 (m, 5H, ArH), 7.44-7.33 (m, 5H, ArH), 7.33-7.25 (m, 6H, ArH), 7.25-7.20 (m, 1H, ArH), 5.68 (d, *J* = 3.6 Hz, 1H, H-1), 5.49 (s, 1H, benzyldiene-H), 5.45 (s, 1H, Ar₂CH-O), 4.86 (d, *J* = 9.3 Hz, 1H, CCH₂-O), 4.73, 4.65 (ABq, *J* = 11.9 Hz, 1H, PhCH₂), 4.43 (d, *J* = 9.3 Hz, 1H, CCH₂-O), 4.41 (d, *J* = 3.4 Hz, 1H, H-1'), 4.37 (d, *J* = 7.4 Hz, 1H, H-4), 4.36 (d, *J* = 3.4 Hz, 1H, H-2'), 4.24 (td, *J* = 7.4, 6.2 Hz, 1H, H-5), 4.17 (dd, *J* = 5.0, 10.0 Hz, 1H, H6a'), 4.13 (dd, *J* = 3.0, 7.6 Hz, 1H, H-3), 4.08 (d, *J* = 6.6 Hz, 1H, H-3'), 4.03 (d, *J* = 3.6 Hz, 1H, H-2), 3.94 (dd, *J* = 6.2, 8.6 Hz, 1H, H-6a), 3.92 (dd, *J* = 6.2, 8.6 Hz, 1H, H-6b), 3.82 (dt, *J* = 5.0, 10.0 Hz, 1H, H-5'), 3.69 (dd, *J* = 6.6, 10.0 Hz, 1H, H-4'), 3.50 (t, *J* = 10.0 Hz, 1H, H-6b'), 1.46 (s, 3H, CCH₃), 1.32 (s, 3H, CCH₃); ¹³C NMR (125 MHz, CDCl₃) δ 141.6 (C), 137.8 (C), 137.4 (C), 137.0 (C), 136.3 (C), 135.5 (C), 129.9 (CH), 129.4 (CH), 129.2 (CH), 129.0 (CH), 128.3 (CH), 128.2 (CH), 127.77 (CH), 127.73 (CH), 127.68 (CH), 127.5 (CH), 127.3 (CH), 127.2 (CH), 126.9 (CH), 126.1 (CH), 111.8 (C), 108.9 (C), 105.1 (CH), 101.1 (CH), 83.5 (CH), 81.7 (CH), 81.4 (CH), 81.1 (CH), 81.0 (CH), 80.4 (CH), 80.2 (CH), 78.1 (CH), 72.5 (CH), 72.1 (CH₂), 71.7 (CH₂), 70.0 (CH₂), 67.2 (CH₂), 64.4 (CH), 57.8 (C), 26.9 (CH₃), 26.8 (CH₃), 26.1 (CH₃), 25.5 (CH₃); HRMS (FAB, MH⁺) calcd for C₆₃H₆₂O₁₁ 791.3431, found 791.3430.

Compound 33-*exo*. $[\alpha]_D^{25} +32.1$ (*c* 2.3, CHCl₃); IR (CHCl₃) ν 3019, 1632, 1212, 1047, 760, 670 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 7.68 (d, *J* = 8.2 Hz, 1H, ArH), 7.48-7.44 (m, 2H, ArH), 7.42 (d, *J* = 8.2 Hz, 1H, ArH), 7.39-7.31 (m, 6H, ArH), 7.31-7.21 (m, 18H, ArH), 7.21-7.12 (m, 4H, ArH), 7.09 (dd, *J* = 6.6, 7.4 Hz, 1H, ArH), 5.70 (s, 1H, Ar₂CH-O), 5.45 (s, 1H, benzylidene-H), 4.86 (d, *J* = 9.2 Hz, 1H, CCH₂-O), 4.80 (d, *J* = 11.4 Hz, 1H, PhCH₂), 4.67 (d, *J* = 11.7 Hz, 1H, PhCH₂), 4.65 (d, *J* = 11.9 Hz, 1H, PhCH₂), 4.63 (d, *J* = 3.4 Hz, 1H, H-1'), 4.58 (d, *J* = 11.7 Hz, 1H, PhCH₂), 4.57 (d, *J* = 3.4 Hz, 1H, H-1), 4.52 (d, *J* = 11.9 Hz, 1H, PhCH₂), 4.44 (d, *J* = 11.4 Hz, 1H, PhCH₂), 4.43 (d, *J* = 3.4 Hz, 1H, H-2'), 4.42 (d, *J* = 12.3 Hz, 1H, PhCH₂), 4.37 (d, *J* = 9.2 Hz, 1H, CCH₂-O), 4.28 (d, *J* = 12.3 Hz, 1H, PhCH₂), 4.10-4.02 (m, 3H, H-4, H-3', H-6a), 3.88 (t, *J* = 9.4 Hz, 1H, H-3), 3.81 (dt, *J* = 5.0, 9.9 Hz, 1H, H-5'), 3.74 (dd, *J* = 6.6, 10.1 Hz, 1H, H-5), 3.65-3.60 (m, 2H, H-6b, H-6a'), 3.57 (dd, *J* = 3.4, 9.4 Hz, 1H, H-2), 3.39-3.32 (m, 2H, H-6b', H-4'), 3.30 (s, 3H, OCH₃); ¹³C NMR (100 MHz, CDCl₃) δ 141.6 (C), 139.4 (C), 138.3 (C), 138.1 (C), 137.7 (C), 137.51 (C), 137.47 (C), 137.0 (C), 136.6 (C), 130.3 (CH), 129.5 (CH), 128.9 (CH), 128.7 (CH), 128.4 (CH), 128.30 (CH), 128.26 (CH), 128.20 (CH), 128.16 (CH), 128.01 (CH), 127.98 (CH), 127.86 (CH), 127.7 (CH), 127.5 (CH), 127.4 (CH), 127.3 (CH), 127.2 (CH), 127.06 (CH), 127.04 (CH), 126.7 (CH), 126.1 (CH), 101.0 (CH), 97.5 (CH), 82.1 (CH), 81.4 (CH), 81.0 (CH), 80.9 (CH), 80.7 (CH), 80.6 (CH), 78.1 (CH), 76.7 (CH), 75.0 (CH₂), 73.32 (CH₂), 73.27 (CH₂), 72.1 (CH₂), 71.8 (CH₂), 70.0 (CH), 69.9 (CH₂), 68.9 (CH₂), 64.6 (CH), 57.9 (C), 55.0 (CH₃); HRMS (FAB, M⁺) calcd for C₆₃H₆₂O₁₁ 994.4292, found 994.4296.

Current Data Parameters
NAME sskdiObn
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20041017
Time 9.07
INSTRUM spect
PROBHD 5 mm QNP 1H/15
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6510.417 Hz
FIDRES 0.198682 Hz
AQ 2.5166323 sec
RG 90.5
DE 76.800 usec
TE 296.5 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCMRK 0.01500000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 8.00 usec
PL1 -2.00 dB
SFO1 500.2027511 MHz

F2 - Processing parameters
SI 16384
SF 500.2000209 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

- 7.4021
- 7.3947
- 7.3878
- 7.3849
- 7.3762
- 7.3663
- 7.3624
- 7.3571
- 7.3526
- 7.3375
- 7.3224
- 7.3116
- 7.3064
- 7.2999
- 7.2946
- 7.2886
- 7.2745
- 7.2650
- 7.2631
- 7.2529
- 7.2497
- 7.2399
- 7.1220
- 7.1062
- 5.5798
- 4.9466
- 4.9243
- 4.8809
- 4.8604
- 4.8218
- 4.8013
- 4.7852
- 4.7629
- 4.7009
- 4.6813
- 4.3935
- 4.3834
- 4.3725
- 4.3625
- 3.8430
- 3.8244
- 3.8166
- 3.8074
- 3.7960
- 3.7754
- 3.7007
- 3.6819
- 3.6632
- 3.4999
- 3.4829
- 3.4808
- 3.4713
- 3.4634
- 3.4517
- 3.4419
- 3.4326
- 3.4226
- 2.3381
- 1.5343
- 0.0757

138.2858
138.1532
138.0759
137.2466
132.9987
129.7438
129.1153
128.9352
128.3641
128.2256
128.1779
128.0940
127.8276
127.7337
125.9609

101.0828

88.4798
83.0288
81.4558
80.3753
77.2527
76.9988
76.7446
75.8169
75.2830
70.1761
68.6942

21.1164

Current Data Parameters
NAME sakdiObn
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20041017
Time 10.11
INSTRUM spect
PROBHD 5 mm QNP 1H/15
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1536
DS 2
SWH 34013.609 Hz
FIDRES 1.035013 Hz
AQ 0.481396 s
RG 2048
DW 14.700 u
DE 6.00 u
TE 298.2 K
D1 3.0000000 s
d11 0.0300000 s
ICREST 0.0000000 s
ICMRK 0.0150000 s
===== CHANNEL f1 =====
NUC1 13C
P0 8.00 u
PL1 3.00 dB
SFO1 125.789571 MHz
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 u
PL2 0.00 dB
PL12 14.89 dB
SFO2 500.2020008 MHz
F2 - Processing parameter

Current Data Parameters
NAME skk277r500
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20041017
Time 13.10
INSTRUM spect
PROBHD 5 mm QNP 1H/15
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6510.417 Hz
FIDRES 0.198682 Hz
AQ 2.5166323 sec
RG 35.9
DE 76.800 usec
TE 296.1 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 8.00 usec
PL1 -2.00 dB
SFO1 500.2027511 MHz

F2 - Processing parameters
SI 16384
SF 500.2000205 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

137.6233
137.0720
132.0479
130.3058
129.6636
129.0838
128.1271
126.4121

102.0097

90.5248

81.0701
77.2474
76.9970
76.7430
75.7437
70.1585
68.7247

21.0932

1.1864
0.8932

Current Data Parameters
NAME ssk2775500
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20041017
Time 13.20
INSTRUM spect
PROBHD 5 mm QNP 1H/15
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1536
DS 2
SWH 34013.605 H
FIDRES 1.036013 H
AQ 0.481396 s
RG 2048
DW 14.700 u
DE 6.00 u
TE 297.3 K
D1 3.0000000 s
d11 0.0300000 s
ICREST 0.0000000 s
MCWK 0.0150000 s

===== CHANNEL f1 =====
NUC1 13C
P0 8.00 u
PL1 3.00 d
SFO1 125.7898571 M

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 u
PL2 0.00 d
PL12 14.89 d
SFO2 500.2020008 M

F2 - Processing parameter

7.3702
7.3617
7.3575
7.3544
7.3477
7.3436
7.3287
7.3248
7.3199
7.3132
7.3032
7.2977
7.2931
7.2912
7.2870
7.2781
7.2735
7.2689
7.2597
7.2573
7.2401
7.1141
7.0943
6.8802
6.8732
6.8681
6.8566
6.8515
6.8444
5.5617
4.9371
4.9091
4.8038
4.7803
4.7533
4.7390
4.7143
4.6739
4.6495
4.3750
4.3624
4.3488
4.3362
3.8146
3.8014
3.7959
3.7763
3.7704
3.7506
3.6851
3.6617
3.6383
3.4799
3.4590
3.4555
3.4509
3.4379
3.4347
3.4261
3.4142
3.4023
3.3898
2.3284
1.5298

1.952
2.046
9.553
1.972
1.944

1.000

1.043
3.155
1.032
1.030

5.132
1.051
2.113

3.095

Current Data Parameters
NAME ssk244r
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040612
Time 17.06
INSTRUM spect
PROBHD 5 mm Multinu
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 256
DM 78.000 usec
DE 111.43 usec
TE 300.0 K
HLL 3 dB
D1 1.00000000 sec
P1 6.00 usec
SFO1 400.1319596 MHz
NUCLEUS 1H

F2 - Processing parameters
SI 32768
SF 400.1300177 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

159.3913

138.3735
138.1283
137.2634
132.9510
130.2666
129.8977
129.7535
129.2173
128.9405
128.3758
128.2343
128.0740
127.7340
125.9736

113.8155

101.0909

88.5715

83.0807
81.4557
80.1333
77.3185
77.0005
76.6835
75.4958
75.2995
70.1844
68.7078

55.2949

21.1261

Current Data Parameters
NAME sk242a
EXPNO 5
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040510
Time 2.06
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 6144
DS 4
SWH 23980.814 H
FIDRES 0.731836 H
AQ 0.683628 s
RG 7298.2
DW 20.850 u
DE 6.00 u
TE 298.9 K
D1 3.0000000 s
D11 0.0300000 s
MCREST 0.0000000 s
MCWRK 0.0150000 s

===== CHANNEL f1 =====
NUC1 13C
P0 5.00 u
PL1 -2.00 d
SFO1 100.628298 M

===== CHANNEL f2 =====
CDDPRG2 waltz16
NUC2 1H
PCPD2 95.00 u
PL2 -2.00 d
SFO2 400.1316005 M

F2 - Processing parameter

7.4057
7.3948
7.3840
7.3803
7.3718
7.3675
7.3647
7.3579
7.3538
7.3462
7.3414
7.3381
7.3302
7.3218
7.3166
7.3085
7.2847
7.2812
7.2759
7.2729
7.2707
7.2646
7.2589
7.2539
7.2407
7.1371
7.1174
5.5953
5.4337
5.4081
5.2146
5.1889
4.9831
4.9550
4.7835
4.7554
4.7499
4.7254
4.4084
4.3958
4.3822
4.3697
3.8751
3.8542
3.8518
3.8460
3.8310
3.8204
3.7946
3.7653
3.7420
3.7186
3.6562
3.6355
3.6318
3.6111
3.4830
3.4704
3.4582
3.4467
3.4347
3.4221
2.3504
2.3415

0.926
0.926
1.014
5.710
1.805
5.540
2.799
2.057

0.998
1.000
0.965
1.020
1.989
1.001

2.050
1.004
0.988
1.036

3.006

Current Data Parameters
NAME ssk281R
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20041010
Time_ 21.28
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 16384
FIDRES 0.292314 Hz
AQ 1.7105396 sec
RG 161.3
DE 104.400 usec
DM 6.00 usec
TE 298.4 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

==== CHANNEL f1 =====
NUC1 1H
P1 10.30 usec
PL1 -2.00 dB
SFO1 400.1320424 MHz

F2 - Processing parameters
SI 16384
SF 400.1300163 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

138.3961
138.2050
137.2355
133.7692
133.7423
132.9278
131.8028
129.7860
129.3075
128.9446
128.7581
128.4295
128.3360
128.2297
127.8655
127.6391
127.0580
126.1938
125.9756
125.7288
125.3090
124.5701

101.1148

88.7421
83.1250
81.5390
80.4382
77.3209
77.0035
76.6862
75.1963
73.9841
70.1036
68.7160

21.1380

Current Data Parameters
NAME SSK281R
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20041011
Time 0.42
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 2048
DS 4
SWH 23980.814 Hz
FIDRES 0.731836 Hz
AQ 0.6832628 s
RG 1149.4
DW 20.850 u
DE 6.00 u
TE 299.1 K
D1 3.00000000 s
D11 0.03000000 s
ICREST 0.00000000 s
PCMRK 0.01500000 s

===== CHANNEL f1 =====
NUC1 13C
P0 5.00 u
PL1 -2.00 d
SFO1 100.628298 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 95.00 u
PL2 -2.00 d
PL12 16.50 d
SFO2 400.1316005 MHz

F2 - Processing parameter

7.3061
7.3021
7.2896
7.2873
7.2817
7.2793
7.2765
7.2711
7.2649
7.2559
7.2399
7.2086
7.1250
7.1053
5.7919
5.6132
5.5923
5.3877
5.1175
5.0998
5.0890
5.0716
5.0522
5.0259
5.0042
4.9862
4.9754
4.9598
4.9474
4.9260
4.9179
4.8921
4.8652
4.8541
4.8289
4.8077
4.7926
4.7797
4.7605
4.7478
4.7360
4.7234
4.7086
4.6960
4.6842
4.6325
4.6086
4.4130
4.4005
4.3868
4.3743
3.9380
3.9148
3.9051
3.8844
3.8614
3.8399
3.8232
3.8110
3.7853
3.7433
3.7315
3.7201
3.7081

3.890
1.045
6.147
5.133
2.736
2.014
1.023
1.011
2.015
1.011
1.000
1.025
2.071
1.054
0.993
1.083
3.065

Current Data Parameters
NAME ssk279R
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20041010
Time 15.23
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 16384
FIDRES 0.292314 Hz
AQ 1.7105396 sec
RG 161.3
DE 104.400 usec
DM 6.00 usec
TE 298.1 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRR 0.01500000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 10.30 usec
PL1 -2.00 dB
SFO1 400.1320424 MHz

F2 - Processing parameters
SI 16384
SF 400.1300165 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

101.1096

21.1203

Current Data Parameters
NAME ssk279R
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20041010
Time 18.36
INSTRUM spect
PROBHD 5 mm DUL 13041
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 2048
DS 4
SWH 23980.814
FIDRES 0.731836
AQ 0.683262
RG 1149.4
DM 20.850
DE 6.00
TE 298.8
D1 3.00000000
D11 0.03000000
ICREST 0.00000000
MCMRK 0.01500000

===== CHANNEL f1 =====
NUC1 13C
P0 5.00
PL1 -2.00
SFO1 100.628298

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 95.00
PL2 -2.00
PL12 16.50
SFO2 400.1316005

F2 - Processing parameters

7.9595
7.5086
7.4884
7.4680
7.4434
7.4149
7.4059
7.4001
7.3773
7.3694
7.3649
7.3487
7.3378
7.3286
7.3263
7.3179
7.3121
7.2978
7.2783
7.2668
7.2640
7.2401
7.2283
7.1634
7.1334
7.1133
6.0071
5.9825
5.7466
5.7220
5.5802
5.0443
5.0151
4.8083
4.7791
4.7599
4.7376
4.3888
4.3762
4.3625
4.3501
3.8481
3.8310
3.8231
3.8147
3.7976
3.4551
3.4425
3.4308
3.4189
2.3418
1.5215

Current Data Parameters
NAME ssk204r2-H1
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20031028
Time_ 22.35
INSTRUM spect
PROBHD 5 mm Multinu
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 64
DS 2
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 1024
DW 78.000 usec
DE 11.43 usec
TE 300.0 K
HL1 3 dB
P1 1.00000000 sec
SFO1 400.1319596 MHz
NUCLEUS 1H

F2 - Processing parameters
SI 32768
SF 400.1300177 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

138.7483
138.2475
137.1537
132.7746
131.4854
131.1265
129.8465
129.5302
128.9542
128.8153
128.6296
128.4790
128.2853
128.2237
127.3771
127.1381
126.1681
125.9819
124.9976
124.9245

101.1847

89.1967
82.8181
81.9402
80.5458
77.3178
77.2036
77.0000
76.6823
74.7148
69.8948
68.7201
68.2392

21.1590

Current Data Parameters
NAME sak204r2
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20031027
Time 0.07
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 4096
DS 4
SWH 23980.814 H
FIDRES 0.731836 H
AQ 0.683628 s
RG 2896.3
DW 20.850 u
DE 6.00 u
TE 298.0 K
D1 3.0000000 s
d11 0.0300000 s
MCREST 0.0000000 s
MCWRR 0.0150000 s

===== CHANNEL f1 =====
NUC1 13C
P0 5.00 u
PL1 -2.00 d
SFO1 100.628298 M

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
POPD2 95.00 u
PL12 -2.00 d
PL12 16.50 d
SFO2 400.1316005 M

F2 - Processing parameters
SI 32768
SF 100.612772 M
RG 0
SSB 0
IB 1.00 H
GB 0
PC 1.40

1.031
3.264
6.648
12.932
3.061
2.066

1.000
1.007
2.058
2.197
2.090
2.778
1.458
2.063
1.035
2.090
1.167
2.091
1.031
1.090
1.082
1.063
1.160
3.034
1.136

7.2101
7.2093
7.2053
7.2044
7.2034
7.2024
7.2014
7.2005
7.1994
7.1984
7.1965
7.1956
7.1926
7.1887
7.1560
7.1497
7.1447
7.1426
7.1408
7.1368
7.1322
6.9761
5.2719
5.2027
4.9258
4.8989
4.8781
4.7736
4.7684
4.7463
4.7409
4.7200
4.7022
4.6933
4.6900
4.6561
4.6261
4.6061
4.5844
4.5760
4.5543
4.5364
4.5278
4.4536
4.4256
4.4016
4.3422
4.3337
4.0529
4.0401
4.0262
4.0178
4.0005
3.9503
3.9269
3.9038
3.7901
3.7775
3.7651
3.7517
3.7402
3.7357
3.7267
3.7106

Current Data Parameters
NAME ssk163pure
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20030517
Time 3.40
INSTRUM spect
PROBHD 5 mm Multinu
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 64
DS 2
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 1024
DW 78.000 usec
DE 11.43 usec
TE 300.0 K
H1 3 dB
NUCLEUS 1H
SFO1 400.1319596 MHz
P1 1.00000000 sec
PC 1.00
F2 - Processing parameters
SI 32768
SF 400.1300174 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

- 141.7600
- 138.7161
- 138.3022
- 138.1490
- 137.8967
- 137.5718
- 137.4851
- 136.6756
- 136.2565
- 130.0039
- 129.5605
- 128.9229
- 128.7541
- 128.4043
- 128.3724
- 128.2880
- 128.1899
- 128.0712
- 127.9943
- 127.8467
- 127.7677
- 127.5906
- 127.1922
- 127.0958
- 126.7585
- 126.5615
- 126.1062
- 101.0355
- 97.8727
- 81.9209
- 81.4948
- 80.9413
- 80.8855
- 80.1440
- 79.9516
- 78.4906
- 78.2039
- 77.2549
- 77.0010
- 76.7469
- 75.7321
- 74.9179
- 73.3766
- 71.9132
- 71.4552
- 70.2943
- 69.9841
- 66.9059
- 64.5535
- 58.0230
- 55.2443

Current Data Parameters
NAME schung052203
EXPTNO 35
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030523
Time 19.40
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 331
DS 4
SWH 32679.738 H
FIDRES 0.997306 H
AQ 0.5014004 s
RG 5160.6
DE 15.300 u
TE 323.0 K
D11 0.03000000 s
PL12 15.00 d
CPDPRG2 waltz16
PCPD2 94.00 u
SFO2 500.1325326 M
NUC2 1H
PL2 3.00 d
D1 3.00000000 s
P0 4.00 u
SFO1 125.7715724 M
NUC1 13C
PL1 3.00 d

F2 - Processing Parameters
SI 32768
SF 125.7577931 M
WDW EM
SSB 0
GB 0
PC 1.40

Current Data Parameters
 NAME schung052203
 EXENO 31
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20030523
 time 5.26
 INSTRUM spect
 PROBD 5 mm tx12 ih/1
 PULPROG zg
 TD 16384
 FIDRES 1.484404 sec
 AQ 114
 RG 90.600 usec
 DW 15.50 usec
 DE 280.0 K
 D1 3.00000000 sec
 P1 4.00 usec
 SFO1 500.1322463 MHz
 NUC1 1H
 PL1 0.00 dB

F2 - Processing parameters
 SI 16384
 SF 500.1300252 MHz
 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00

Current Data Parameters
NAME schung052203
EXPNO 32
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030523
Time 5.27
INSTRUM spect
PROBHD 5 mm tx12 1h/1
PULPROG cosyg
TD 1024
SOLVENT CDCl3
NS 2
DS 16
SWH 4990.020 Hz
FIDRES 4.873066 Hz
AQ 0.1026548 sec
RG 724.1
DW 100.200 usec
DE 15.50 usec
TE 300.0 K
D1 2.0000000 sec
P1 8.10 usec
SFO1 500.1321506 MHz
NUC1 1H
PL1 0.00 dB
D0 0.00000300 sec
P16 1000.00 usec
D16 0.00030000 sec
P0 4.00 usec
D13 0.00000300 sec
IN0 0.00020040 sec

F1 - Acquisition Parameters
ND0 1
TD 256
SF01 500.1322 MHz
FIDRES 19.492266 Hz
SW 9.977 ppm
FMODE undefined

F2 - Processing parameters
SI 1024
SF 500.1300280 MHz
WDW SSB
LB 0
GB 0
PC 1.00

F1 - Processing parameters
SI 1024
MC2 OF
SF 500.1300913 MHz
WDW SSB
LB 0
GB 0

Current Data Parameters
NAME schung052203
EXPNO 32
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030523
Time 5.27
INSTRUM spect
PROBHD 5 mm txi2 1h/1
PULPROG cosyg
TD 1024
SOLVENT CDCl3
NS 2
DS 16
SWH 4990.020 Hz
FIDRES 4.873066 Hz
AQ 0.1026548 sec
RG 724.1
DE 100.200 usec
TE 300.0 K
D1 2.00000000 sec
P1 8.10 usec
SFO1 500.1321506 MHz
NUC1 1H
PL1 0.00 dB
DO 0.00000300 sec
P16 1000.00 usec
D16 0.00030000 sec
P0 4.00 usec
D13 0.00000300 sec
IN0 0.00020040 sec

F1 - Acquisition Parameters
ND0 1
TD 256
SFO1 500.1322 MHz
FIDRES 19.492266 Hz
SW 9.977 ppm
FMODE undefined

F2 - Processing parameters
SI 1024
SF 500.1300280 MHz
WDW SSB
LB 0
GB 0
PC 1.00

F1 - Processing parameters
SI 1024
MC2 OF
SF 500.1300913 MHz
WDW SSB
LB 0
GB 0

Current Data Parameters
NAME schung052203
EXPNO 32
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030523
Time 5.27
INSTRUM spect
PROBHD 5 mm txi2 1h/1
PULPROG cosyg
TD 1024
SOLVENT CDCl3
NS 2
DS 16
SWH 4990.020 Hz
FIDRES 4.873066 Hz
AQ 0.1026548 sec
RG 724.1
DW 100.200 usec
DE 15.50 usec
TE 300.0 K
D1 2.0000000 sec
P1 8.10 usec
SFO1 500.1321506 MHz
NUC1 1H
PL1 0.00 dB
D0 0.00000300 sec
P16 1000.00 usec
D16 0.0030000 sec
P0 4.00 usec
D13 0.00000300 sec
IN0 0.00020040 sec

F1 - Acquisition Parameters
ND0 1
TD 256
SFO1 500.1322 MHz
FIDRES 19.492266 Hz
SW 9.977 ppm
FMODE undefined

F2 - Processing parameters
SI 1024
SF 500.1300280 MHz
WDW QSINE
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

F1 - Processing parameters
SI 1024
MC2 OF
SF 500.1300913 MHz
WDW QSINE
SSB 0
LB 0.00 Hz
GB 0

Current Data Parameters
NAME schung052203
EXPNO 32
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030523
Time 5.27
INSTRUM spect
PROBHD 5 mm tx12 1h/1
PULPROG cosyg
TD 1024
SOLVENT CDCl3
NS 2
DS 16
SWH 4990.020 Hz
FIDRES 4.873066 Hz
AQ 0.1026548 sec
RG 724.1
DE 100.200 usec
TE 300.0 K
D1 2.0000000 sec
P1 8.10 usec
SFO1 500.1321506 MHz
NUC1 1H
PL1 0.00 dB
DO 0.00000300 sec
P16 1000.00 usec
D16 0.00030000 sec
P0 4.00 usec
D13 0.00000300 sec
IN0 0.00020040 sec

F1 - Acquisition Parameters
ND0 1
TD 256
SF01 500.1322 MHz
FIDRES 19.492266 Hz
SW 9.977 ppm
FMODE undefined

F2 - Processing parameters
SI 1024
SF 500.1300913 MHz
WDW QSINE
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

F1 - Processing parameters
SI 1024
MC2 OF
SF 500.1300913 MHz
WDW QSINE
SSB 0
LB 0.00 Hz
GB 0

Current Data Parameters
NAME schung052203
EXPNO 31
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030523
Time 5.26
INSTRUM spect
PROBHD 5 mm tx12 1h/1
PULPROG zg
TD 16384
SOLVENT D2O
NS 16
DS 0
SWH 5518.764 Hz
FIDRES 0.336839 Hz
AQ 1.484404 sec
RG 114
DW 90.600 usec
DE 15.50 usec
TE 280.0 K
D1 3.00000000 sec
P1 4.00 usec
SFO1 500.1322463 MHz
NUC1 1H
PL1 0.00 dB

F2 - Processing parameters
SI 16384
SF 500.1300252 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

141.3433
138.7752
138.4219
137.7238
137.3446
137.1993
136.1859
135.7777
130.0846
129.8042
129.0042
128.9109
128.3353
128.2686
128.1196
127.9618
127.7672
127.6290
127.4181
127.0410
126.8288
126.6397
126.0937

101.0556
98.6994

81.4854
80.3592
80.1515
79.0304
78.2370
77.2540
77.0003
76.7463
76.2370
76.1707
75.1774
75.0889
74.8193
73.4188
72.9268
72.0288
71.5916
69.8552
69.3485
67.2842
64.3771
58.2402
55.4242

Current Data Parameters
NAME ssK231b2-500
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040403
Time 16.43
INSTRUM spect
PROBHD 5 mm QNP 1H/15
PULPROG zgpgdc
TD 32768
SOLVENT CDCl3
NS 4096
DS 2
SWH 34013.605 H
FIDRES 1.038013 H
AQ 0.4817396 s
RG 574.7
DW 14.700 u
DE 6.00 u
TE 296.7 K
D1 3.00000000 s
d11 0.03000000 s
MCREST 0.00000000 s
MCWRK 0.01500000 s

===== CHANNEL f1 =====
NUC1 13C
P0 8.00 u
P11 3.00 d
SFO1 125.789571 M

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 u
PL2 0.00 d
PL12 14.89 d
SFO2 500.202008 M

F2 - Processing parameter

7.2422
7.2398
7.2368
7.2336
7.2297
7.2266
7.2240
7.2174
7.2141
7.2062
7.2024
7.1967
7.1862
7.1802
7.1754
7.1711
7.1670
7.1627
7.0981
7.0958
7.0795
7.0773
7.0610
7.0587
5.3829
5.2567
4.9058
4.8827
4.8024
4.7753
4.7255
4.7197
4.7012
4.6936
4.6796
4.6625
4.6495
4.6230
4.5895
4.4832
4.4561
4.4255
4.4028
4.3687
4.3672
4.3603
4.3587
4.0271
4.0145
4.0089
4.0009
3.9880
3.8622
3.8516
3.8449
3.8211
3.7877
3.7681
3.7633
3.7457
3.7338
3.7202
3.7003
3.6902

1.049
1.267
3.439
1.251
6.537
3.254
15.254
2.090
1.133

1.009
1.000
1.020
1.054
4.559
1.717
2.278
1.069
1.034
1.058
2.054
1.970
5.380
1.091
2.982
1.070

Current Data Parameters
NAME: ssk224-b
EXPNO: 1
PROCNO: 1
F2 - Acquisition Parameters
Date_: 20040308
Time_: 17.19
INSTRUM: spect
PROBHD: 5 mm Multinu
PULPROG: zg
TD: 32768
SOLVENT: CDCl3
NS: 64
DS: 2
SWH: 6410.256 Hz
FIDRES: 0.195625 Hz
AQ: 2.5559540 sec
RG: 512
DW: 78.000 usec
DE: 11.43 usec
TE: 300.0 K
HL1: 3 dB
P1: 1.00000000 sec
SFO1: 400.1319596 MHz
NUCLEUS: 1H
F2 - Processing parameters
SI: 32768
SF: 400.1300176 MHz
WDW: no
SSB: 0
LB: 0.00 Hz
GB: 0
PC: 1.00

141.8896
138.6878
138.5617
138.3844
137.9635
137.5676
137.4073
137.0547
136.3884
130.2692
129.7500
128.8638
128.6256
128.3251
128.1392
127.8810
127.7939
127.5492
127.2921
126.9771
126.7973
126.5217
126.1057

100.9841
99.0144

81.5277
80.9893
80.8154
80.1413
79.4183
78.7674
77.2554
77.0016
76.7476
75.2936
74.9297
72.8000
72.2215
72.1389
71.9770
71.4582
69.9540
66.8997
64.5121
58.0709
54.8008

Current Data Parameters
NAME
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040309
Time 14.42
INSTRUM spect
PROBHD 5 mm QNP 1H/15
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 2048
DS 2
SWH 34013.605 H
FIDRES 1.038013 H
AQ 0.4817396 s
RG 2048
DW 14.700 u
DE 6.00 u
TE 299.1 K
D1 3.0000000 s
d11 0.0300000 s
MCREST 0.0000000 s
MCWRR 0.0150000 s

===== CHANNEL f1 =====
NUC1 13C
P0 8.00 u
PL1 3.00 d
SFO1 125.7898571 M

===== CHANNEL f2 =====
CDDPRG2 waltz16
NUC2 1H
PCPD2 90.00 u
PL2 0.00 d
PL12 14.89 d
SFO2 500.2020008 M

F2 - Processing parameter

141.6642
138.7101
137.7429
137.5018
137.3214
137.2296
137.0663
136.5948
130.2259
129.7246
128.8896
128.8531
128.6667
128.1721
128.1325
128.1075
127.5756
127.4230
127.3440
127.2649
127.2315
127.1586
127.0365
126.7963
126.0612
125.9060
105.1127
101.0060
82.1141
81.6414
81.0706
80.3471
80.1832
80.0725
79.2261
77.6742
77.3185
77.2036
77.0006
76.6831
73.8128
71.5358
70.9344
70.1528
68.8145
65.4940
64.4888
58.2029
56.9789

Current Data Parameters
NAME SSK206AK
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters

Date_ 20031102
Time 23:54
INSTRUM spect
PROBHD 5 mm DUL
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 4096
DS 4
SWH 23980.814 H
FIDRES 0.731836 H
AQ 0.6832628 s
RG 724.1
DE 20.850 u
TE 298.0 K
D1 3.0000000 s
D11 0.0300000 s
MCREST 0.0000000 s
MCMRK 0.0150000 s

===== CHANNEL f1 =====
NUC1 13C
P0 5.00 u
PL1 -2.00 d
SFO1 100.628298 M

S41

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 95.00 u
PL2 -2.00 d
PL12 16.50 d
SFO2 400.1316005 M

F2 - Processing parameters
SI 32768
SF 100.612729 M
WDW EM
SSB 0
LB 1.00 H
GB 0
PC 1.40

7.2741
7.2552
7.2524
7.2397
7.2026
7.1961
7.1853
7.1791
7.1711
7.1684
7.1401
7.1216
7.1029
7.0798
7.0750
7.0658
7.0566
6.7715
6.7666
6.7575
6.7485
5.6719
5.5444
5.4217
4.8598
4.8367
4.7651
4.7571
4.6319
4.6048
4.4289
4.4062
4.3856
4.3670
4.3556
4.3431
4.3340
4.3103
4.3068
4.2790
4.1842
4.1527
4.1422
4.1199
4.0977
4.0200
4.0074
3.9934
3.9808
3.9263
3.9097
3.7924
3.7671
3.7546
3.7416
3.7054
3.6821
3.6588
3.6255
3.6088
3.6003
3.5836
3.4805

0.962
1.874
9.307
3.014
2.537
5.770
1.194
1.982
1.919

0.962
1.004
1.000
1.029
1.025
1.026
0.994
5.010
1.017
1.057
1.022
0.989
2.117
1.085
1.171
4.077
2.044

Current Data Parameters
NAME: ssk205b-pure
EXPNO: 1
PROCNO: 1

F2 - Acquisition Parameters
Date_: 20031027
Time: 4.41
INSTRUM: spect
PROBHD: 5 mm DUL 13C-1
PULPROG: zg30
TD: 16384
SOLVENT: CDCl3
NS: 32
DS: 2
SWH: 4789.272 Hz
FIDRES: 0.292314 Hz
AQ: 1.7105396 sec
RG: 181
DW: 104.400 usec
DE: 6.00 usec
TE: 298.0 K
D1: 1.00000000 sec
MCREST: 0.00000000 sec
MCWRK: 0.01500000 sec

===== CHANNEL f1 =====
NUC1: 1H
P1: 10.30 usec
PL1: -2.00 dB
SFO1: 400.1320424 MHz

F2 - Processing parameters
SI: 16384
SF: 400.1300174 MHz
WDW: no
SSB: 0
LB: 0.00 Hz
GB: 0
PC: 1.00

141.5967
138.6773
137.7720
137.5295
137.3629
137.3072
137.2005
136.8991
136.1760
130.6856
130.2397
129.5751
129.1578
128.8355
128.5499
128.2725
128.1625
128.1267
127.9396
127.5650
127.4913
127.3509
127.3034
127.2249
127.0385
126.9192
126.7251
126.4013
126.0661
105.6003
101.5951
100.9227
81.8363
81.7461
81.4855
81.1121
81.0021
80.0783
79.4474
77.7591
77.3206
77.2064
77.0031
76.6859
74.2563
71.2534
71.0558
70.0752
68.8889
65.9217
64.5040
58.1582
57.2845
47.1557

Current Data Parameters
NAME ssf205b-pure
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 2001027
Time 11.54
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpgcg
TD 32768
SOLVENT CDCl3
NS 3072
DS 4
SWH 23980.814
FIDRES 0.731836
AQ 0.6832628
RG 2896.3
DW 20.850
DE 6.00
TE 298.0
D1 3.00000000
D11 0.03000000
MCREST 0.00000000
MCWRE 0.01500000

===== CHANNEL f1 =====
NUC1 13C
P0 5.00
PL1 -2.00
SFO1 100.6228298

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 95.00
PL2 -2.00
PL12 16.50
SFO2 400.1316005

F2 - Processing parameters
SI 32768
SF 100.6127722
WDW EM
SSB 0
LB 1.00
GB 0
PC 1.40

Current Data Parameters
NAME ssk212c
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20031125
Time 16.07
INSTRUM spect
PROBHD 5 mm Multinu
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 64
DS 2
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 1024
DW 78.000 usec
DE 111.43 usec
TE 300.0 K
HL1 3 dB
D1 1.00000000 sec
P1 6.00 usec
SF01 400.1319596 MHz
NUCLEUS 1H

F2 - Processing parameters
SI 32768
SF 400.1300177 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

- 141.9193
- 138.2320
- 138.0092
- 137.6050
- 137.5400
- 136.3637
- 135.8376
- 129.9918
- 128.9042
- 128.8694
- 128.3384
- 128.2020
- 128.1829
- 127.7583
- 127.6828
- 127.6360
- 127.5869
- 127.5534
- 127.5127
- 127.3140
- 126.7710
- 126.6618
- 126.1276
- 109.1788
- 101.1808
- 97.8451
- 81.1216
- 81.0578
- 78.8040
- 78.0565
- 77.3169
- 77.2019
- 76.9991
- 76.6813
- 76.2071
- 73.7867
- 73.4214
- 73.2094
- 71.9831
- 71.8912
- 70.1108
- 69.6006
- 66.1549
- 64.7793
- 58.1066
- 55.5256
- 27.9408
- 26.2968

Current Data Parameters
NAME SSK-212G
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20031201
Time 7.36
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 5120
DS 4
SWH 23980.814 H
FIDRES 0.731836 H
AQ 0.6832628 s
RG 2896.3
DW 20.850 u
DE 6.00 u
TE 298.0 K
D1 3.0000000 s
d11 0.0300000 s
MCREST 0.0000000 s
MCWRR 0.0150000 s

===== CHANNEL f1 =====
NUC1 13C
P0 5.00 u
PL1 -2.00 d
SFO1 100.628298 M

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 95.00 u
PL2 -2.00 d
PL12 16.50 d
SFO2 400.1316005 M

F2 - Processing parameters
SI 32768
SF 100.6127716 M
WDW EM
SSB 0
LB 1.00 H
GB 0
PC 1.40

Current Data Parameters
 NAME: ssk219b500
 EXPNO: 1
 PROCNO: 1

F2 - Acquisition Parameters
 Date_: 20031226
 Time: 13.20
 INSTRUM: spect
 PROBHD: 5 mm QNP 1H/15
 PULPROG: zg
 TD: 32768
 SOLVENT: CDCl3
 NS: 32
 DS: 2
 SWH: 6510.417 Hz
 FIDRES: 0.198682 Hz
 AQ: 2.5166323 sec
 RG: 406.4
 DW: 76.800 usec
 DE: 6.00 usec
 TE: 298.1 K
 D1: 1.00000000 sec
 MCREST: 0.00000000 sec
 MCWRR: 0.01500000 sec

===== CHANNEL f1 =====
 NUC1: 1H
 P1: 8.00 usec
 PL1: -2.00 dB
 SFO1: 500.2027511 MHz

F2 - Processing parameters
 SI: 16384
 SF: 500.2000197 MHz
 WDW: no
 SSB: 0
 LB: 0.00 Hz
 GB: 0
 PC: 1.00

Current Data Parameters
NAME sk219b
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20031227
Time_ 9.02

INSTRUM spect
PROBHD 5 mm Multinu
PULPROG zgpgdc
TD 32768
SOLVENT CDCl3
NS 4096
DS 2

SWH 31250.000 Hz
FIDRES 0.953674 Hz
AQ 0.5243380 sec
RG 32768
DE 16.000 usec
TE 300.0 K

CPDPRG waltz16
P31 100.00 usec
S2 28 dB
H11 3 dB
D1 5.00 sec
P0 100.6273592 MHz

NUCLEUS 13C

F2 - Processing parameters
SI 32768
SF 100.6127732 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

Current Data Parameters
NAME: ssk223-30p
EXPNO: 1
PROCNO: 1

F2 - Acquisition Parameters
Date_: 20040403
Time: 4.22
INSTRUM: spect
PROBHD: 5 mm DUL 13C-1
PULPROG: zg30
TD: 16384
SOLVENT: CDCl₃
NS: 8
DS: 2
SWH: 4789.272 Hz
FIDRES: 0.292314 Hz
AQ: 1.7105396 sec
RG: 256
DM: 104.400 usec
DE: 6.00 usec
TE: 298.0 K
D1: 1.00000000 sec
MCREST: 0.00000000 sec
MCWRK: 0.01500000 sec

==== CHANNEL f1 =====
NUC1: ¹H
P1: 10.30 usec
PL1: -2.00 dB
SFO1: 400.1320424 MHz

F2 - Processing parameters
SI: 16384
SF: 400.1300178 MHz
WDW: no
SSB: 0
LB: 0.00 Hz
GB: 0
PC: 1.00

165.8057

141.4744
138.1155
137.8156
137.4738
137.3154
136.8929
135.7025
133.0534
130.1344
130.0079
129.6209
129.3449
129.2579
128.9204
128.4261
128.3211
128.3044
128.1846
128.0021
127.7506
127.6618
127.6342
127.4661
127.3574
127.2709
127.1593
126.0955

101.0793
98.4258

81.5972
81.5011
80.7822
80.7665
80.3893
78.1917
77.3177
77.2046
76.9999
76.7695
76.6821
75.0368
72.2512
71.9959
69.8877
69.0489
64.6321
64.2997
62.9641
57.8336
55.2303

Current Data Parameters
NAME ssk223-30p
EXNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040403
Time 17.19
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 4480
DS 4
SWH 23980.814
FIDRES 0.731836
AQ 0.683628
RG 2896.3
DW 20.850
DE 6.00
TE 298.0
D1 3.00000000
D1.1 0.03000000
MCOREST 0.00000000
MCRMK 0.01500000

===== CHANNEL f1 =====
NUC1 13C
P0 5.00
PL1 -2.00
SFO1 100.628298

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 95.00
PL2 2.00
PL12 16.50
SFO2 400.1316005

F2 - Processing Parameters

7.3127
7.3045
7.2983
7.2899
7.2825
7.2770
7.2692
7.2618
7.2546
7.2494
7.2401
7.2308
7.2279
7.2177
7.2143
7.2118
5.6931
5.6841
5.4851
5.4682
4.8776
4.8544
4.7484
4.7186
4.6590
4.6292
4.4488
4.4254
4.4212
4.4126
4.3734
4.3627
4.3547
4.2682
4.2532
4.2500
4.2346
4.2194
4.1889
4.1762
4.1624
4.1506
4.1437
4.1323
4.1248
4.0929
4.0763
4.0367
4.0277
3.9700
3.9545
3.9484
3.9330
3.9261
3.9113
3.9044
3.8897
3.8552
3.8425
3.8303
3.8175
3.8054
3.7928
3.7093

1.059
5.352
5.265
6.138
0.440

0.981
1.000
1.013

1.028
2.140
2.108
2.098
1.076
2.128
1.045
1.039
2.096
1.078
1.068
1.050

6.160
3.257
3.019

Current Data Parameters
NAME: ssk252p
EXPNO: 1
PROCNO: 1
F2 - Acquisition Parameters
Date_: 20040630
Time: 3.24
INSTRUM: spect
PROBHD: 5 mm Multinu
PULPROG: zg
TD: 32768
SOLVENT: CDCl3
NS: 16
DS: 2
SWH: 6410.256 Hz
FIDRES: 0.195625 Hz
AQ: 2.5559540 sec
RG: 256
DW: 78.000 usec
DE: 11.43 usec
TE: 300.0 K
HL1: 3 dB
D1: 1.00000000 sec
P1: 6.00 usec
SFO1: 400.1319596 MHz
NUCLEUS: 1H
F2 - Processing parameters
SI: 32768
SF: 400.1300176 MHz
WDW: no
SSB: 0
LB: 0.00 Hz
GB: 0
PC: 1.00

141.5910
137.7999
137.4125
137.0434
136.3176
135.5059
129.8804
129.3617
129.2325
128.9613
128.3426
128.2109
127.7691
127.7312
127.6782
127.5009
127.3006
127.1840
126.9279
126.1112
111.7963
108.9127
105.0762
101.1431
83.4967
81.6481
81.4432
81.0801
81.0020
80.4252
80.1686
78.1206
77.2561
77.0019
76.7480
72.4547
72.1317
71.7171
69.9585
67.2341
64.4091
57.8403
26.8556
26.7890
26.0750
25.5229

Current Data Parameters
NAME ssk252p500
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20040704
Time_ 18.18
INSTRUM spect
PROBHD 5 mm QNP 1H/15
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 7168
DS 2
SWH 34013.605 H
FIDRES 1.035013 H
AQ 0.481396 s
RG 406.4
DW 14.700 u
DE 6.00 u
TE 282.9 K
D1 3.0000000 s
d11 0.0300000 s
ICREST 0.0000000 s
ICMRK 0.0150000 s

===== CHANNEL f1 =====
NUC1 13C
P0 8.00 u
PL1 3.00 d
SFO1 125.7898571 M

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 u
PL2 0.00 d
PL12 14.89 d
SFO2 500.2020008 M

F2 - Processing parameter

7.2457
7.2426
7.2399
7.2377
7.2320
7.2261
7.2217
7.2200
7.2160
7.2146
7.2072
7.1983
7.1953
7.1741
7.1699
7.1553
7.1397
7.1372
7.1141
7.0976
7.0955
7.0771
5.7019
5.4547
4.8716
4.8486
4.8144
4.7858
4.6869
4.6665
4.6573
4.6368
4.6285
4.6043
4.5769
4.5753
4.5685
4.5383
4.5085
4.4457
4.4337
4.4251
4.4173
4.3940
4.3865
4.3638
4.2969
4.2666
4.0962
4.0831
4.0670
4.0567
4.0321
3.9060
3.8827
3.8611
3.8254
3.8124
3.8008
3.7574
3.7408
3.7321
3.7156

1.105
2.075
1.280
5.767
20.751
4.135
1.183

1.000
1.010
1.046
1.093
3.161
2.167
1.092
4.185
1.114
3.087
1.074
1.128
1.072
3.145
2.093
3.027

Current Data Parameters
NAME: ssk218pure
EXPNO: 1
PROCNO: 1

F2 - Acquisition Parameters
Date_: 20040116
Time: 4.17
INSTRUM: spect
PROBHD: 5 mm DUL 13C-1
PULPROG: zg30
TD: 2930
FIDRES: 0.292314 Hz
RG: 1.7105396 sec
DE: 181
TE: 298.0 K
D1: 1.00000000 sec
MCREST: 0.00000000 sec
MCWRK: 0.01500000 sec

===== CHANNEL f1 =====
NUC1: 1H
P1: 10.30 usec
PL1: -2.00 dB
SFO1: 400.1320424 MHz

F2 - Processing parameters
SI: 16384
SF: 400.1300171 MHz
WDW: no
SSB: 0
LB: 0.00 Hz
GB: 0
PC: 1.00

9
8
7
6
5
4
3
2
1
0 ppm

Current Data Parameters	
NAME	ssk218bute
EXPO	4
PROCO	1
F2 - Acquisition Parameters	
Time	20040116
Date	13.29
INSTRM	
PROHD	5 mm DUL
PULPROG	13C-1
TD	2904d
	3768d
SOLVENT	CDCl3
NS	40936
DS	4
SWH	23960.8144
FIDRES	0.7318361
AQ	0.6683628
RG	2896.3
DM	20.8530
DE	6.00
TE	298.0
D1	3.00000000
D11	0.03000000
MCRST	0.00000000
MOMR	0.01500000
===== CHANNEL f1 =====	
NUC1	13C
F0	5.00
PD1	-3.00
SFO1	100.6282298
===== CHANNEL f2 =====	
CPDPRG2	waltz16
NUC2	1H
PCPDZ	95.00
PD2	-2.00
PD12	16.50
SFO2	400.1316005
F2 - Processing Parameters	