Enantioselective Construction of Carbobicyclic Scaffolds

Douglass F. Taber, Yigang He, Ming Xu

Department of Chemistry and Biochemistry, University of Delaware, Newark, DE 19716

Supporting Information

Contents:

S-1 to S-5	Table of Contents
S-6	General Experimental
S-7	Epoxide 1a
S-8	Alcohol 2a , alcohol 2b
S-9	Alcohol 2c , alcohol 2d
S-10	Alcohol 2e
S-11	Alcohol 2f , benzoate 6b
S-12	(+) - Alcohol 2a
S-13	(+) - Diol 20
S-14	(-) - Diol 21
S-15	(-) - Benzyl ether 22
S-16	(-) - Aldehyde 23
S-17	(-) - Benzyl ether 24
S-18	(+) - Diol 25 , (-) - aldehyde 26
S-19	(-) - Ketone 27
S-20	(-) - Ketone 8 , tosylate 28
S-21	Aldehyde 9

S-22	Aldehyde 10
S-23	Benzenesulfonate 29
S-24	Methanesulfonate 13
S-25	Sulfone 11
S-26	Sulfone 12
S-27	Sulfone 3
S-28	Epoxide 30
S-29	Ketone 31
S-30	Ketal 14
S-31	Sulfone 15
S-32	Benzenesulfonate 32, methanesulfonate 16
S-33	Sulfone 17, benzenesulfonate 33
S-34	Methanesulfonate 18
S-35	Sulfone 19, references
S-36	^1H NMR of 1a
S-37	^{13}C NMR of 1a
S-38	^1H NMR of 2a
S-39	^{13}C NMR of 2a
S-40	HPLC of (+) - 2a
S-41	^1H NMR of 2b
S-42	^{13}C NMR of 2b
S-43	^1H NMR of 2c
S-44	^{13}C NMR of 2c

S-45	¹ H NMR of 2d
S-46	¹³ C NMR of 2d
S-47	¹ H NMR of 2f
S-48	¹³ C NMR of 2f
S-49	¹ H NMR of 6b
S-50	¹³ C NMR of 6b
S-51	¹ H NMR of 20
S-52	¹³ C NMR of 20
S-53	¹ H NMR of 21
S-54	¹³ C NMR of 21
S-55	¹ H NMR of 22
S-56	¹³ C NMR of 22
S-57	¹ H NMR of 23
S-58	¹³ C NMR of 23
S-59	¹ H NMR of 24
S-60	¹³ C NMR of 24
S-61	¹ H NMR of 25
S-62	¹³ C NMR of 25
S-63	¹ H NMR of 26
S-64	¹³ C NMR of 26
S-65	¹ H NMR of 27
S-66	¹³ C NMR of 27
S-67	¹ H NMR of 8

S-68	^{13}C NMR of 8
S-69	^1H NMR of 28
S-70	^{13}C NMR of 28
S-71	^1H NMR of 9
S-72	^{13}C NMR of 9
S-73	^1H NMR of 10
S-74	^{13}C NMR of 10
S-75	^1H NMR of 29
S-76	^{13}C NMR of 29
S-77	^1H NMR of 13
S-78	^{13}C NMR of 13
S-79	^1H NMR of 11
S-80	^{13}C NMR of 11
S-81	^1H NMR of 12
S-82	^{13}C NMR of 12
S-83	^1H NMR of 3
S-84	^{13}C NMR of 3
S-85	^1H NMR of 30
S-86	^{13}C NMR of 30
S-87	^1H NMR of 31
S-88	^{13}C NMR of 31
S-89	^1H NMR of 14
S-90	^{13}C NMR of 14

S-91	^1H NMR of 15
S-92	^{13}C NMR of 15
S-93	^1H NMR of 32
S-94	^{13}C NMR of 32
S-95	^1H NMR of 16
S-96	^{13}C NMR of 16
S-97	^1H NMR of 17
S-98	^{13}C NMR of 17
S-99	^1H NMR of 33
S-100	^{13}C NMR of 33
S-101	^1H NMR of 18
S-102	^{13}C NMR of 18
S-103	^1H NMR of 19
S-104	^{13}C NMR of 19

Experimental Section

General. ^1H and ^{13}C NMR were recorded as solutions in CDCl_3 at 400 and 100 (or 90) MHz, respectively. ^{13}C NMR multiplicities were determined with the aid of a JVERT sequence, differentiating the signals for methylene and quaternary carbons as "u" from methyl and methine carbons as "d". The infrared (IR) spectra were determined as neat oils. R_f values indicated refer to thin-layer chromatography on 2.5×10 cm, 250- μm analytical plates coated with silica gel GF and developed in the solvent indicated. Flash column chromatography was performed with Merck 35-60 μ silica gel following the procedure of Still.¹ Where specified, TLC mesh silica gel was employed, following the procedure described by Taber.² Solvents are referred as volume/volume mixture. PE is petroleum ether. EtOAc is ethyl acetate. MTBE is methyl tert-butyl ether. THF and diethyl ether were distilled from sodium/benzophenone under N_2 and used immediately. CH_2Cl_2 was distilled from CaH_2 under N_2 . All reactions were carried out in flame-dried glassware under a positive pressure of N_2 unless otherwise indicated. The alkenes were prepared by adding the corresponding ketone to the appropriate Grignard reagent or alkynyl lithium, followed by *p*-toluenesulfonic acid-catalyzed dehydration of the product alcohol.³ Known epoxides were prepared according to the literature.

General procedure for preparation of epoxides (1a, 1b, 1c)

The alkene (10 mmol), CH_2Cl_2 (80 mL) and saturated aqueous NaHCO_3 (80 mL) were combined, and the mixture was cooled in an ice water bath. mCPBA (70%, 2.70 g, 11.0 mmol) was added, and the resulting mixture was stirred vigorously at 0 °C for 1-2 h with monitoring by TLC. The layers were separated and the organic layer was partitioned between CH_2Cl_2 and, sequentially, 1 N aqueous NaOH and saturated aqueous NaCl. The

combined organic extract was dried (Na_2SO_4) and concentrated, and the residue was chromatographed.

Epoxide 1a

Clear oil (91% yield), TLC R_f = 0.71 (PE: MTBE, 9:1). IR (neat, cm^{-1}): 2928 (s), 2852 (m), 1598 (w), 1491 (m), 1443 (m); ^1H NMR (400 MHz, CDCl_3) δ 7.25-7.45 (m, 5H), 3.38 (dd, J = 6.8, 3.4 Hz, 1H), 1.37-2.30 (m, 10H); ^{13}C NMR (CDCl_3 , 100 MHz) δ u: 122.6, 90.6, 81.5, 54.7, 34.7, 31.3, 29.4, 24.9, 24.4; d: 132.0, 128.6, 128.4, 64.0; HRMS calcd for $\text{C}_{15}\text{H}_{16}\text{O}$ (M^+) 212.1201, found 212.1195.

Typical procedure for the ring opening of epoxides with Grignard reagents

To a solution of epoxide (2 mmol) in anhydrous THF (15 mL) was added dropwise a solution of Grignard reagent (8 mmol, if indicated, 10 mmol was used in some cases) over two minutes at 0 °C. The reaction mixture was warmed slowly to room temperature, then stirred overnight. The reaction mixture was partitioned between saturated aqueous NH_4Cl and ether. The combined organic extract was dried (Na_2SO_4) and concentrated, and the residue was chromatographed.

Alcohol 2a

Clear oil (83% yield), TLC R_f = 0.64 (PE: acetone, 3:1). IR (neat, cm^{-1}): 3434 (s), 2932 (s), 2240 (w), 1638 (w), 1597 (w), 1490 (m), 1442 (m); ^1H NMR (400 MHz, CDCl_3) δ 7.26-7.32 (m, 5H), 6.00 (m, 1H), 5.14 (d, J = 11.6 Hz, 1H), 5.13 (d, J = 15.2 Hz, 1H), 3.36 (dd, J = 9.2, 4.0 Hz, 1H), 1.30-2.80 (m, 13H); ^{13}C NMR (CDCl_3 , 100 MHz) δ u: 123.5, 118.2, 91.5, 86.1, 47.1, 44.4, 34.6, 33.7, 27.0, 23.0, 22.1; d: 135.2, 131.9, 128.4, 128.2, 78.3; HRMS calcd for $\text{C}_{18}\text{H}_{23}\text{O}$ ($\text{M}+\text{H}$) 255.1749, found 255.1743.

Alcohol 2b

Clear oil (88% yield), TLC R_f = 0.66 (PE: MTBE, 1:1). IR (neat, cm^{-1}): 3399 (s), 2933 (s), 2240 (w), 1639 (w), 1597 (w), 1490 (m), 1443 (m), 1050 (m); ^1H NMR (400 MHz, CDCl_3) δ 7.25-7.43 (m, 5H), 6.00 (m, 1H), 5.14 (d, J = 16.8 Hz, 1H), 5.13 (d, J = 9.6 Hz, 1H), 3.30 (br, 1H), 1.10-2.80 (m, 11H); ^{13}C NMR (CDCl_3 , 100 MHz) δ u: 123.6, 118.1, 91.1, 86.4, 44.8, 43.7, 35.5, 32.8, 24.9, 22.5; d: 134.9, 131.9, 128.4, 128.1, 75.3; HRMS calcd for $\text{C}_{17}\text{H}_{21}\text{O}$ ($\text{M}+\text{H}$) 241.1592, found 241.1582.

Alcohol 2c

Clear oil (88% yield), TLC R_f = 0.58 (PE: MTBE, 1:1). IR (neat, cm^{-1}): 3448 (s), 2965 (s), 2238 (w), 1640 (w), 1598 (w), 1490 (s), 1443 (m), 1092 (s), 914 (s); ^1H NMR (400 MHz, CDCl_3) δ 7.25-7.42 (m, 5H), 6.03 (m, 1H), 5.16 (m, 1H), 5.11 (m, 1H), 3.85 (dd, J = 11.2 Hz, 5.6 Hz, 1H), 1.55-2.44 (m, 9H); ^{13}C NMR (CDCl_3 , 100 MHz) δ u: 123.3, 117.7, 91.2, 85.9, 49.0, 42.5, 35.6, 32.2, 20.4; d: 135.0, 131.9, 128.4, 128.2, 79.2; HRMS calcd for $\text{C}_{16}\text{H}_{19}\text{O}$ ($\text{M}+\text{H}$) 227.1436, found 227.1434.

Alcohol 2d

Clear oil (84% yield), TLC R_f = 0.34 (PE:MTBE, 4:1). IR (neat, cm^{-1}): 3426 (s), 2932 (s), 2856 (m), 1491 (m), 1444 (m); ^1H NMR (400 MHz, CDCl_3) δ 7.20-7.43 (m, 10H), 3.31 (dt, J = 6.4 Hz, 10.4 Hz, 1H), 3.27 (d, J = 13.0 Hz, 1H), 2.83 (d, J = 13.0 Hz, 1H), 1.17-1.92 (m, 9H); ^{13}C NMR (CDCl_3 , 100 MHz) δ u: 137.4, 123.5, 91.3, 87.1, 45.9, 44.8, 35.0, 33.0, 24.8, 22.4; d: 131.8, 131.1, 128.4, 128.1, 127.9, 126.5, 74.8; HRMS calcd for $\text{C}_{21}\text{H}_{22}\text{O}$ (M^+) 290.1671, found 290.1683.

Alcohol 2e (2:1 ratio of two isomers)

Clear oil (85% yield), TLC R_f = 0.42 (PE:MTBE, 4:1). IR (neat, cm^{-1}): 3442 (s), 2933 (s), 2857 (m), 1490 (m), 1444 (m), 1052 (m); ^1H NMR (400 MHz, CDCl_3) δ major isomer: 7.23-7.44 (m, 5H), 5.80 (m, 1H), 5.10-5.20 (m, 2H), 3.49 (m, 1H), 2.88 (m, 1H), 1.50-1.91 (m, 9H), 1.21 (d, $J=6.8$ Hz, 3H); minor isomer: 7.23-7.44 (m, 5H), 6.15 (m, 1H), 5.01-5.14 (m, 2H), 3.55 (m, 1H), 2.68 (m, 1H), 1.50-1.91 (m, 9H), 1.12 (d, $J=6.8$ Hz, 3H); ^{13}C NMR (CDCl_3 , 100 MHz) δ (two isomers are reported together): u: 123.6, 116.7, 114.9, 113.5, 91.6, 91.3, 86.9, 86.2, 48.3, 47.8, 32.9, 32.3, 31.0, 29.4, 24.6, 24.4, 22.4, 22.3; d: 142.0, 139.4, 131.8, 131.7, 128.4, 128.3, 128.0, 127.9, 73.0, 72.4, 43.5, 42.5, 16.1, 14.4; HRMS calcd for $\text{C}_{18}\text{H}_{22}\text{O}$ (M^+) 254.1671, found 254.1659.

Alcohol 2f

Clear oil (76% yield), TLC R_f = 0.36 (PE:MTBE, 4:1). IR (neat, cm^{-1}): 3406 (s), 2933 (s), 2856 (m), 1643 (w), 1490 (m), 1443 (m), 1044 (m); ^1H NMR (400 MHz, CDCl_3) δ 7.39-7.43 (m, 2H), 7.26-7.31 (m, 3H), 4.91 (m, 1H), 4.82 (m, 1H), 3.29 (dt, $J=4.0$ Hz, 10.0 Hz, 1H), 2.78 (d, $J=13.2$ Hz, 1H), 2.19 (d, $J=13.2$ Hz, 1H), 1.96 (s, 3H), 1.16-2.00 (m, 9H); ^{13}C NMR (CDCl_3 , 100 MHz) δ u: 143.1, 123.6, 115.0, 91.8, 86.5, 47.5, 44.1, 35.6, 32.6, 24.8, 22.5; d: 131.7, 128.4, 128.0, 76.4, 24.7; HRMS calcd for $\text{C}_{18}\text{H}_{22}\text{O}$ (M^+) 254.1671, found 254.1675.

Benzoate 6b

A solution of alcohol **2a** (0.226 g, 0.890 mmol) in 10 mL of anhydrous CH_2Cl_2 was added DMAP (0.217 g, 1.780 mmol) and 3,5-dinitrobenzoyl chloride (0.410 g, 1.780 mmol) sequentially at room temperature. The resulting mixture was stirred at room temperature

for 3 h, then partitioned between CH_2Cl_2 and, sequentially, saturated aqueous NH_4Cl and saturated aqueous NaCl . The CH_2Cl_2 extract was dried (Na_2SO_4) and concentrated, and the residue was chromatographed to give 0.386 g of benzoate **6b** (97% yield) as colorless crystals: mp = 113- 114 °C, TLC R_f = 0.55 (PE: MTBE, 5:1). IR (film, cm^{-1}): 2937 (m), 1728 (s), 1628 (w), 1548 (s), 1344 (s), 1276 (s), 1170 (m); ^1H NMR (400 MHz, CDCl_3) δ 9.23 (s, 3H), 7.33-7.56 (m, 5H), 5.97 (m, 1H), 5.07 (s, 1H), 4.98-5.04 (m, 2H), 1.61-2.53 (m, 12H); ^{13}C NMR (CDCl_3 , 100 MHz) δ u: 161.9, 148.9, 134.5, 123.5, 118.7, 90.9, 85.3, 45.0, 44.5, 34.7, 31.2, 26.7, 22.6, 22.1; d: 134.0, 132.0, 129.7, 128.6, 128.3, 122.6, 82.6; HRMS calcd for $\text{C}_{25}\text{H}_{24}\text{N}_2\text{O}_6$ (M^+) 448.1634, found 448.1646.

(+)-Alcohol 2a

To a 500 mL three-necked flask was added alkene **8** (2.94 g, 15.0 mmol), 150 mL of a 2:1 mixture of dimethoxymethane and acetonitrile, 90 mL of potassium carbonate-acetic acid buffer solution,⁴ tetrabutylammonium hydrogen sulfate (102 mg) and Shi's chiral ketone⁵ (1.35 g, 2.93 mmol). The flask was connected to two dropping funnels, one charged with a solution of oxone (13.8 g, 22.5 mmol) in 51 mL of aqueous 4×10^{-4} Na_2EDTA and the other charged with 51 mL of 1.47 M aqueous KOH. The two solutions were added dropwise at the same rate over 70 min to the cold reaction mixture which was stirred vigorously at 0 °C. After 2 h, ether (150 mL) was added. The biphasic mixture was

partitioned between ether and, sequentially, water and brine. The organic extract was dried (Na_2SO_4) and concentrated. The residue was dissolved in 50 mL of anhydrous THF. To this solution allylmagnesium chloride (32.6 mL, 2.0 M) was added dropwise over 10 min at 0 °C under N_2 . The reaction solution was warmed slowly from 0 °C to room temperature, then maintained at ambient temperature overnight. The reaction mixture was partitioned between saturated aqueous NH_4Cl and ether. The combined organic extract was dried over Na_2SO_4 and concentrated *in vacuo*. The residue was chromatographed to give 2.74 g of alcohol **2a** (72% yield from **7**) as a colorless oil, TLC R_f = 0.64 (PE: acetone, 3:1), $[\alpha]_D^{22}$ = +63.1 (c 1.7, THF); IR (neat, cm^{-1}): 3434 (s), 2932 (s), 2240 (w), 1638 (w), 1597 (w), 1490 (m), 1442 (m); ^1H NMR (400 MHz, CDCl_3) δ 7.26-7.32 (m, 5H), 6.00 (m, 1H), 5.14 (d, J = 11.6 Hz, 1H), 5.13 (d, J = 15.2 Hz, 1H), 3.36 (dd, J = 9.2, 4.0 Hz, 1H), 1.30-2.80 (m, 13H); ^{13}C NMR (CDCl_3 , 100 MHz) δ u: 123.5, 118.2, 91.5, 86.1, 47.1, 44.4, 34.6, 33.7, 27.0, 23.0, 22.1; d: 135.2, 131.9, 128.4, 128.2, 78.3; HRMS calcd for $\text{C}_{18}\text{H}_{23}\text{O}$ ($\text{M}+\text{H}$) 255.1749, found 255.1743.

(+)-Diol **20**

To alcohol **2a** (2.70 g, 10.6 mmol) was added 9-BBN in THF (0.5 M, 53.2 mL) slowly over 10 min under N_2 and stirring at 0 °C. The resulting solution was stirred at ambient temperature overnight. The reaction mixture was cooled in an ice water bath and aqueous

NaOH solution (3 N, 15 mL) was added, followed by addition of 30% H₂O₂ (15 mL) very carefully. After stirring at room temperature for 3 h, the reaction mixture was partitioned between brine and ether. The combined organic extract was dried over Na₂SO₄ and concentrated *in vacuo*. The residue was chromatographed to give 2.45 g of diol **21** (85% yield) as a colorless oil, TLC *R_f* = 0.5 (ether), [α]_D²² = +5.61 (c 0.82, THF). IR (neat, cm⁻¹): 3362 (s), 2933 (s), 2224 (w), 1490 (m), 1442 (m), 1052 (s); ¹H NMR (400 MHz, CDCl₃) δ 7.38-7.43 (m, 2H), 7.25-7.30 (m, 3H), 3.60-3.70 (m, 2H), 3.34 (dd, *J*=9.2 Hz, 3.0 Hz, 1H), 2.89 (br, 1H), 2.60 (br, 1H), 1.34-1.92 (m, 14 H); ¹³C NMR (CDCl₃, 100 MHz) δ u: 123.5, 92.0, 85.7, 63.1, 46.6, 35.8, 34.4, 33.8, 28.0, 26.9, 22.9, 22.2; d: 131.8, 128.3, 128.0, 78.2; HRMS calcd for C₁₈H₂₄O₂ (M⁺) 272.1776, found 272.1776.

(-)-Diol **21**

Diol **20** (1.73 g, 6.36 mmol) in pyridine (20 mL) was added palladium on BaSO₄ (677 mg). The resulting mixture then stirred under H₂ atmosphere (1 atm) for 23 h. Silica gel (4.0 g) was added and the mixture was concentrated. The residual solid was chromatographed to give 1.65 g of diol **21** (95% yield) as a colorless oil, TLC *R_f* = 0.5 (ether), [α]_D²² = -40.9 (c 0.85, THF). IR (neat, cm⁻¹): 3393 (s), 2924 (s), 1458 (m), 1030 (m); ¹H NMR (400 MHz, CDCl₃) δ 7.17-7.28 (m, 5H), 6.65 (d, *J*=13.2 Hz, 1H), 5.75 (d, *J*=13.2 Hz, 1H), 3.55 (d, *J*=8.4 Hz, 1H), 3.38-3.44 (m, 2H), 3.29 (s, 1H), 3.13 (s, 1H),

1.29-1.83 (m, 14H); ^{13}C NMR (CDCl_3 , 100 MHz) δ u: 139.4, 63.1, 48.1, 34.9, 34.2, 32.9, 27.6, 27.1, 23.0, 22.1; d: 136.3, 130.4, 128.4, 127.7, 126.5, 77.6; HRMS calcd for $\text{C}_{18}\text{H}_{26}\text{O}_2$ (M^+) 274.1933, found 274.1919.

(-)-Benzyl ether 22

To a stirred solution of diol **21** (1.34 g, 4.89 mmol) in dry THF (25 mL) was added tetrabutylammonium bromide (18 mg, 0.049 mmol) and 60% NaH (783 mg, 19.6 mmol). After stirring at room temperature under N_2 for 30 min, BnBr (1.45 mL, 12.2 mmol) was added. The resulting mixture was refluxed gently under N_2 for 8 h. The reaction was partitioned between water and ether. The combined organic extract was dried over Na_2SO_4 and concentrated *in vacuo*. The residue was chromatographed to give 1.89 g of benzyl ether **22** (85% yield) as a colorless oil, TLC R_f = 0.73 (PE:MTBE, 9:1), $[\alpha]_D^{22}$ = -25.9 (c 0.93, THF). IR (neat, cm^{-1}): 2929 (s), 2857 (s), 1494 (m), 1453 (s), 1361 (m), 1098 (s), 1070 (s); ^1H NMR (400 MHz, CDCl_3) δ 7.13-7.33 (m, 15H), 6.57 (d, J = 13.2 Hz, 1H), 5.85 (d, J = 13.2 Hz, 1H), 4.63 (d, J = 12.0 Hz, 1H), 4.40 (s, 2H), 4.36 (d, J = 12.0 Hz, 1H), 3.25 (d, J = 8.0 Hz, 1H), 3.18-3.21 (m, 2H), 1.12-1.81 (m, 14H); ^{13}C NMR (CDCl_3 , 100 MHz) δ u: 140.0, 139.3, 138.9, 72.9, 71.4, 71.0, 48.3, 35.2, 34.9, 28.5, 28.0, 25.0, 24.3, 22.4; d: 137.6, 129.0, 128.7, 128.5, 128.4, 127.8, 127.7, 127.6, 127.5, 127.4, 126.3, 86.0; HRMS calcd for $\text{C}_{32}\text{H}_{38}\text{O}_2\text{Na}$ ($\text{M} + \text{Na}$) 477.2770, found 477.2774.

(-)-Aldehyde 23

A solution of alkene **22** (1.60 g, 3.52 mmol) in CH_2Cl_2 (50 mL) was cooled to $-78\text{ }^\circ\text{C}$. After purging with O_2 for 2 min, O_3 was bubbled through the solution until the pale blue color persisted. The reaction was purged with N_2 for 5 min, then PPh_3 (1.38 g, 5.28 mmol) was added. The resulting solution was first stirred at $-78\text{ }^\circ\text{C}$ for 1 h, then room temperature for 1 h. Silica gel (4.0 g) was added and the mixture was concentrated. The residual solid was chromatographed to give 1.18 g of aldehyde **23** (88% yield) as a colorless oil, TLC $R_f = 0.54$ (PE:MTBE, 4:1), $[\alpha]_D^{22} = -14.0$ (c 0.8, THF). IR (neat, cm^{-1}): 2967 (s), 2858 (s), 1726 (s), 1453 (m), 1092 (s); ^1H NMR (400 MHz, CDCl_3) δ 9.54 (s, 1H), 7.25-7.36 (m, 10H), 4.60 (d, $J=11.6$ Hz, 1H), 4.46 (s, 2H), 4.30 (d, $J=11.6$ Hz, 1H), 3.66 (d, $J=7.2$ Hz, 1H), 3.38-3.43 (m, 2H), 1.36-2.04 (m, 14H); ^{13}C NMR (CDCl_3 , 100 MHz) δ u: 138.6, 138.4, 73.0, 70.8, 70.6, 56.3, 30.3, 29.3, 27.8, 27.2, 24.3, 22.8, 22.1; d: 205.6, 128.6, 128.5, 127.8, 127.7, 127.6, 127.5, 83.0; HRMS calcd for $\text{C}_{25}\text{H}_{32}\text{O}_3\text{Na}$ ($\text{M}+\text{Na}$) 403.2249, found 403.2251.

(-)-Benzyl ether **24**

To a 25 mL round bottom flask was added aldehyde **23** (424 mg, 1.12 mmol), ethylene glycol (10 mL), N_2H_4 (64%, 718 mg, 22.4 mmol) and ground KOH (86%, 1.46 g, 22.4 mmol). The flask was connected to a short-path distillation head. The reaction mixture was heated at 130 °C under N_2 for 2 h, then 210 °C for 3 h. After cooling down, the reaction mixture was partitioned between water and CH_2Cl_2 . The combined organic extract was dried over Na_2SO_4 and concentrated *in vacuo*. The residue was chromatographed to give 365 mg of benzyl ether **24** (89% yield) as a colorless oil, TLC $R_f = 0.67$ (PE:MTBE, 9:1), $[\alpha]_D^{22} = -31.6$ (c 0.7, THF). IR (neat, cm^{-1}): 2927 (s), 2854 (s), 1453 (m), 1098 (s); ^1H NMR (400 MHz, CDCl_3) δ 7.22-7.34 (m, 10H), 4.59 (d, $J=11.6$ Hz, 1H), 4.47 (s, 2H), 4.31 (d, $J=11.6$ Hz, 1H), 3.39 (dd, $J=7.6$ Hz, 6.4 Hz, 1H), 3.39 (dd, $J=6.4$ Hz, 5.6 Hz, 1H), 3.11 (dd, $J=6.8$ Hz, 3.8 Hz, 1H), 1.27-1.74 (m, 14H), 0.91 (s, 3H); ^{13}C NMR (CDCl_3 , 100 MHz) δ u: 139.5, 138.8, 73.0, 71.6, 71.3, 40.2, 36.8, 36.2, 29.6, 27.6, 25.5, 24.5, 22.3; d: 128.5, 128.3, 127.7, 127.6, 127.5, 127.3, 86.5, 22.1; HRMS calcd for $\text{C}_{25}\text{H}_{35}\text{O}_2$ ($\text{M}+\text{H}$) 367.2637, found 367.2629.

(+)-Diol **25**

To a solution of benzyl ether **24** (180 mg, 0.492 mmol) in absolute ethanol (5 mL) was added 10% Pd/C (20 mg). The mixture was stirred under H₂ atmosphere (1 atm) for 15 h. Silica gel (1.0 g) was added and the mixture was concentrated. The residual solid was chromatographed to give 84 mg of diol **25** (92% yield) as a colorless oil, TLC R_f = 0.44 (ether), $[\alpha]_D^{22}$ = +2.35 (c 0.85, THF). IR (neat, cm⁻¹): 3355 (s), 2928 (s), 1461 (m), 1019 (m); ¹H NMR (400 MHz, CDCl₃) δ 3.57-3.69 (m, 2H), 3.48 (dd, J =9.2 Hz, 2.4 Hz, 1H), 1.25-1.76 (m, 16H), 0.87 (s, 3H); ¹³C NMR (CDCl₃, 100 MHz) δ u: 63.9, 39.9, 36.7, 36.1, 33.5, 28.7, 27.1, 24.7, 22.1; d: 78.6, 20.5; HRMS calcd for C₁₁H₂₁O (M-OH) 169.1592, found 169.1590.

(-)-Aldehyde **26**

To a stirred suspension of Dess-Martin reagent (349 mg, 0.82 mmol) in CH₂Cl₂ (2 mL) was added a solution of diol **25** (51 mg) in CH₂Cl₂ (2 mL) slowly over 5 min. The mixture was stirred at room temperature for 30 min. Silica gel (2.0 g) was added and the mixture was concentrated. The residual solid was chromatographed to give 46 mg of aldehyde **26** (92% yield) as a colorless oil, TLC R_f = 0.54 (PE:MTBE, 1:1), $[\alpha]_D^{22}$ = -29.3 (c 1.1, THF). IR (neat, cm⁻¹): 2928 (s), 2857 (m), 1723 (s), 1698 (s), 1458 (m); ¹H NMR

(400 MHz, CDCl₃) δ 9.75 (s, 1H), 2.37-2.69 (m, 4H), 1.28-1.92 (m, 10H), 1.04 (s, 3H);
¹³C NMR (CDCl₃, 100 MHz) δ u: 217.3, 50.1, 40.4, 39.2, 38.1, 31.2, 30.7, 26.7, 24.6; d:
 201.9, 21.5; HRMS calcd for C₁₁H₁₉O₂ (M+H) 183.1385, found 183.1379.

(-)-Ketone 27

To a stirred solution of ^tBuOK (56.8 mg, 0.506 mmol) in dry THF (2 mL) was added n-propyltriphenylphosphonium bromide (205 mg, 0.531 mmol) in two portions over 2 min at 0 °C. The resulting orange solution was stirred at 0 °C for 1 h, then room temperature for 30 min. The solution was cooled to 0 °C again, and aldehyde **26** (46 mg, 0.253 mmol) in dry THF (2 ml) was added slowly. The resulting solution was stirred at room temperature overnight. The reaction mixture was partitioned between saturated aqueous NH₄Cl and CH₂Cl₂. The combined organic extract was dried over Na₂SO₄ and concentrated *in vacuo*. The residue was chromatographed to give 45 mg of alkene **27** (86% yield) as a colorless oil, TLC *R_f* = 0.74 (PE:MTBE, 4:1), [α]_D²² = -35.2 (c 0.87, THF). IR (neat, cm⁻¹): 2930 (s), 1701 (s), 1458 (m); ¹H NMR (400 MHz, CDCl₃) δ 5.35 (m, 1H), 5.27 (m, 1H), 2.68 (m, 1H), 2.35 (m, 1H), 1.24-2.02 (m, 14H), 1.06 (s, 3H), 0.94 (t, *J*=7.2 Hz, 3H); ¹³C NMR (CDCl₃, 100 MHz) δ u: 217.9, 51.1, 40.4, 40.3, 38.0, 30.9, 26.9, 24.6, 22.1, 20.6; d: 132.2, 128.7, 21.5, 14.5; HRMS calcd for C₁₄H₂₅O (M+H) 209.1905, found 209.1903.

(-)-Ketone 8 ⁶

To a solution of alkene **27** (45 mg, 0.216 mmol) in absolute ethanol (4 mL) was added 10% Pd/C (10 mg). The mixture was stirred under H₂ atmosphere (1 atm) for 5 h. Silica gel (500 mg) was added and the mixture was concentrated. The residual solid was chromatographed to give 44 mg of ketone **8** (92% yield) as a colorless oil, $[\alpha]_D^{22} = -36.3$ (c 0.9, THF). IR (neat, cm⁻¹): 2929 (s), 2856 (m), 1701 (s), 1465 (m); ¹H NMR (400 MHz, CDCl₃) δ 2.67 (m, 1H), 2.33 (m, 1H), 1.18-1.83 (m, 18H), 1.03 (s, 3H), 0.87 (t, J=6.8 Hz, 3H); ¹³C NMR (CDCl₃, 100 MHz) δ u: 218.4, 51.1, 40.5, 40.4, 38.0, 31.9, 30.9, 30.1, 26.9, 24.6, 24.1, 22.8; d: 21.7, 14.3.

Tosylate 28

To a stirred solution of secondary alcohol **2a** (9.50 g, 37.4 mmol) in dry pyridine (80 mL) was added DMAP (0.457 g, 3.74 mmol) and tosyl chloride (14.3 g, 74.8 mmol). The resulting solution was stirred under N₂ at room temperature for 36 h. The reaction mixture was partitioned between ether and 5% HCl. The ether layer was dried over

Na₂SO₄ and concentrated *in vacuo*. The residue was chromatographed to give 13.9 g of tosylate **28** (91% yield) as a colorless oil, TLC *R_f* = 0.67 (PE:MTBE, 4:1). IR (neat, cm⁻¹): 2938 (s), 1640 (w), 1449 (m), 1363 (s), 1185 (s); ¹H NMR (400 MHz, CDCl₃) δ 7.80-7.84 (m, 2H), 7.36-7.40 (m, 2H), 7.25-7.29 (m, 5H), 5.95 (m, 1H), 5.08-5.16 (m, 2H), 4.48 (dd, *J*=10.0 Hz, 2.8 Hz, 1H), 2.61 (dd, *J*=14.0 Hz, 6.0 Hz, 1H), 2.39 (s, 3H), 2.13 (dd, *J*=14.0 Hz, 9.2 Hz, 1H), 1.40-1.97 (m, 10H); ¹³C NMR (CDCl₃, 100 MHz) δ u: 144.5, 134.8, 123.5, 118.7, 90.4, 85.2, 44.6, 43.6, 34.2, 31.5, 26.1, 22.0, 21.9; d: 134.0, 131.7, 129.7, 128.2, 127.9, 127.6, 88.3, 21.6; HRMS calcd for C₂₅H₂₈O₃NaS (M+Na) 431.1657, found 431.1656.

Aldehyde **9**

A solution of tosylate **28** (6.0 g, 14.7 mmol) in CH₂Cl₂ (200 mL) was cooled to -78 °C. After purging with O₂ for 3 min, O₃ then was bubbled through the solution until the pale blue color persisted. The reaction mixture was purged with N₂ for 5 min, then PPh₃ (5.01 g, 19.1 mmol) was added. The resulting solution was first stirred at -78 °C for 1 h, then room temperature for 1 h. Silica gel (20.0 g) was added and the mixture was concentrated. The residual solid was chromatographed to give 5.48 g of aldehyde **9** (91% yield) as a colorless oil, TLC *R_f* = 0.29 (PE:MTBE, 4:1). IR (neat, cm⁻¹): 2935 (s), 2864 (m), 1722 (s), 1598 (m), 1491 (m), 1443 (m), 1362 (s), 1175 (s); ¹H NMR (400 MHz,

CDCl₃) δ 9.82 (dd, $J=3.6$ Hz, 1.6 Hz, 1H), 7.80 (d, $J=6.4$ Hz, 2H), 7.28-7.40 (m, 7H), 4.48 (dd, $J=10.4$ Hz, 2.8 Hz, 1H), 2.79 (dd, $J=16$ Hz, 1.6 Hz, 1H), 2.42 (s, 3H), 2.38 (dd, $J=16$ Hz, 3.6 Hz, 1H), 1.45-2.17 (m, 10H); ¹³C NMR (CDCl₃, 100 MHz) δ u: 145.0, 134.2, 122.8, 88.1, 87.0, 52.1, 42.8, 36.2, 31.3, 25.7, 22.2, 21.8; d: 201.6, 131.7, 129.9, 128.4, 128.3, 127.8, 87.6, 21.7; HRMS calcd for C₂₄H₂₆O₄S (M⁺) 410.1552, found 410.1569.

Aldehyde 10

A solution of aldehyde **9** (4.1 g, 10 mmol) in 100 mL of a 1:1 mixture of dry THF and tert-butanol was heated to 45 °C under N₂, then a solution of ^tBuOK (1.46 g, 13 mmol) in 60 mL of a 1:1 mixture of dry THF and tert-butanol was added slowly over 10 min. The resulting solution was stirred at this temperature for 1.5 h. The reaction mixture was partitioned between water and ether. The combined organic extract was dried over Na₂SO₄ and concentrated *in vacuo*. The residue was chromatographed to give 2.1 g of aldehyde **10** (88% yield) as a colorless oil, TLC R_f = 0.68 (PE:MTBE, 4:1). IR (neat, cm⁻¹): 2925 (s), 2852 (m), 2221 (w), 1709 (s); ¹H NMR (400 MHz, CDCl₃) δ 9.21 (d, $J=6.8$ Hz, 1H), 7.35-7.40 (m, 2H), 7.25-7.29 (m, 3H), 2.41 (dd, $J=14.4$ Hz, 6.8 Hz, 1H), 2.29 (m, 1H), 2.08 (m, 1H), 1.91 (m, 1H), 1.08-1.84 (m, 8H); ¹³C NMR (CDCl₃, 100 MHz) δ

u: 123.1, 91.3, 80.2, 35.7, 31.9, 29.8, 28.1, 27.3, 26.6; d: 200.6, 131.7, 128.3, 128.1, 46.5, 34.4; HRMS calcd for $C_{17}H_{18}O$ (M^+) 238.1358, found 238.1352.

Benzenesulfonate **29**

To a stirred solution of alcohol **2a** (7.60 g, 29.9 mmol) in dry pyridine (40 mL) was added DMAP (0.365 g, 2.99 mmol) and benzenesulfonyl chloride (10.6 g, 59.8 mmol). The resulting solution was stirred under N_2 at room temperature for 40 h. The reaction mixture was partitioned between ether and 10% HCl. The ether layer was dried over Na_2SO_4 and concentrated *in vacuo*. The residue was chromatographed to give 10.8 g of benzenesulfonate **29** (92% yield) as a colorless oil, TLC R_f = 0.72 (PE:MTBE, 4:1). IR (neat, cm^{-1}): 2937 (s), 1640 (w), 1490 (w), 1448 (m), 1363 (s), 1185 (s); 1H NMR (400 MHz, $CDCl_3$) δ 7.91-7.94 (m, 2H), 7.23-7.60 (m, 8H), 5.91 (m, 1H), 5.05-5.13 (m, 2H), 4.47 (dd, $J=10.0$ Hz, 2.8 Hz, 1H), 1.46-2.59 (m, 12H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ u: 138.3, 123.9, 119.2, 90.7, 85.7, 45.1, 44.1, 34.6, 32.1, 26.6, 22.5, 22.4; d: 134.4, 134.0, 132.2, 129.6, 128.6, 128.4, 128.0, 89.1; HRMS calcd for $C_{24}H_{26}O_3S$ (M^+) 394.1603, found 394.1609.

Methanesulfonate 13

A solution of benzenesulfonate **29** (7.88 g, 20.0 mmol) in HPLC grade methanol (50 mL) and CH_2Cl_2 (50 mL) was cooled to $-78\text{ }^\circ\text{C}$. After purging with O_2 for 2 min, O_3 was bubbled through the solution until the pale blue color persisted. The reaction was purged with N_2 for 5 min, then NaBH_4 (1.50 g, 40.0 mmol) was added. After stirring at $-78\text{ }^\circ\text{C}$ for 10 min, the reaction mixture was cooled with an ice-water bath. After an additional 30 min, NaBH_4 (750 mg, 20.0 mmol) was added followed by stirring for another 30 min. The latter procedure was repeated once more. The reaction mixture was partitioned between water and CH_2Cl_2 . The combined organic extract was dried (Na_2SO_4) and concentrated, and the residual clear oil was dissolved in 150 mL of anhydrous CH_2Cl_2 . To this solution triethylamine (6.07 g, 60.0 mmol) was added, then methanesulfonyl chloride (3.44 g, 30.0 mmol) was added slowly at $0\text{ }^\circ\text{C}$. After stirring under N_2 at ambient temperature for 1 h, the reaction mixture was partitioned between saturated aqueous water and CH_2Cl_2 . The combined organic extract was dried (Na_2SO_4) and concentrated. The crude product was chromatographed to give 7.91 g of methanesulfonate **13** (83% yield) as a clear oil, TLC $R_f = 0.5$ (PE:MTBE, 1:2). IR (neat, cm^{-1}): 2939 (s), 1448 (m), 1357 (s), 1175 (s); ^1H NMR (400 MHz, CDCl_3) δ 7.93-7.95 (m, 2H), 7.28-7.66 (m, 8H), 4.46-4.50 (m, 3H), 3.00 (s, 3H), 1.42-2.28 (m, 12H); ^{13}C NMR (CDCl_3 , 100 MHz) δ u:

137.6, 122.9, 88.6, 86.3, 67.6, 44.1, 37.9, 35.7, 31.3, 26.1, 22.1; d: 133.9, 131.9, 129.4, 128.4, 127.7, 88.5, 37.6; HRMS calcd for $C_{24}H_{28}O_6NaS$ (M+Na) 499.1225, found 499.1242.

Sulfone 11

To a mixture of methanesulfonate **13** (160 mg, 0.338 mmol) in benzene (2 mL) and water (2 mL) was added benzenethiol (112 mg, 1.01 mmol), NaOH (135 mg, 3.38 mmol) and tetrabutylammonium bromide (11.0 mg, 0.0338 mmol). The mixture was stirred vigorously under N_2 at room temperature for 22 h. The reaction mixture was partitioned between water and CH_2Cl_2 . The combined CH_2Cl_2 extract was dried (Na_2SO_4), and mCPBA (70%, 332 mg, 1.35 mmol) was added to this solution. After 10 min at room temperature the reaction was partitioned between CH_2Cl_2 and, sequentially, saturated aqueous $Na_2S_2O_3$, saturated aqueous Na_2CO_3 and brine. The combined organic extract was dried (Na_2SO_4) and concentrated. The crude product was chromatographed to give 150 mg of sulfone **11** (85% yield) as a colorless oil, TLC R_f = 0.43 (PE:MTBE, 1:1). IR (neat, cm^{-1}): 2937 (s), 1447 (s), 1361 (m), 1306 (s), 1186 (s), 1145 (s), 1087 (m); 1H NMR (400 MHz, $CDCl_3$) δ 7.87-7.91 (m, 4H), 7.50-7.66 (m, 6H), 7.24-7.31 (m, 5H), 4.40 (dd, J =11.6 Hz, 2.4 Hz, 1H), 3.28-3.41 (m, 2H), 1.35-2.20 (m, 12H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ u: 138.9, 137.3, 122.8, 88.3, 86.5, 52.8, 44.6, 35.0, 32.0, 31.7, 26.0,

22.1, 22.0; d: 133.9, 133.8, 131.8, 129.4, 129.3, 128.4, 128.3, 128.2, 127.7, 87.8; HRMS calcd for $C_{29}H_{30}O_5NaS_2$ (M+Na) 545.1432, found 545.1454.

Sulfone 12

To a stirred solution of sulfone 11 (63.0 mg, 0.121 mmol) in dry THF (1 mL) was added $n\text{-BuLi}$ (71 μL , 1.80 M in hexane) under N_2 at -78°C . The reaction mixture was stirred at ambient temperature for 1h, then was partitioned between saturated aqueous NH_4Cl and ether. The combined organic extract was dried (Na_2SO_4) and concentrated. The crude product was chromatographed to give 36 mg of sulfone 12 (82% yield) as a colorless oil, TLC R_f = 0.58 (PE:MTBE, 1:1). IR (neat, cm^{-1}): 2925 (s), 2854 (m), 1446 (m), 1301 (s), 1149 (s); ^1H NMR (400 MHz, CDCl_3) δ 7.89-7.93 (m, 2H), 7.27-7.66 (m, 8H), 3.40 (m, 1H), 3.19 (m, 1H), 2.84 (dd, $J=12$ Hz, 9.2 Hz, 1H), 2.15 (dd, $J=12$ Hz, 8.8 Hz, 1H), 1.22-2.18 (m, 10H); ^{13}C NMR (CDCl_3 , 100 MHz) δ u: 138.9, 123.9, 95.4, 84.5, 36.8, 36.4, 33.7, 33.5, 32.2, 27.0, 26.2; d: 133.8, 131.8, 129.5, 128.6, 128.4, 128.0, 60.0, 47.2; HRMS calcd for $C_{23}H_{24}O_2S$ (M^+) 364.1497, found 364.1507.

Sulfone 3

To a stirred solution of methyl phenyl sulfone (145 mg, 0.930 mmol) in dry THF (1 mL) was added $n\text{BuLi}$ (0.37 mL, 2.5 M in hexane) at $-78\text{ }^{\circ}\text{C}$ under N_2 . After stirring at this temperature for 5 min, a solution of methanesulfonate **13** (400 mg, 0.840 mmol) in dry THF (2 mL) was added slowly. The resulting mixture then was stirred at ambient temperature for 19 h. The reaction was cooled to $-78\text{ }^{\circ}\text{C}$, and $n\text{BuLi}$ (0.37 mL, 2.5 M in hexane) was added slowly. After stirring at ambient temperature for 2 h, the reaction mixture was partitioned between saturated aqueous NH_4Cl and ether. The combined organic extract was dried (Na_2SO_4) and concentrated. The crude product was chromatographed to give 230 mg of sulfone **3** (72% yield) as a white solid: mp = $121\text{--}122\text{ }^{\circ}\text{C}$, TLC R_f = 0.85 (PE:MTBE, 1:2). IR (film, cm^{-1}): 2926 (s), 2854 (m), 1490 (s), 1446 (m), 1301 (s), 1147 (s), 1086 (m); ^1H NMR (400 MHz, CDCl_3) δ 7.90–7.95 (m, 2H), 7.47–7.53 (m, 3H), 7.24–7.34 (m, 5H), 3.13 (t d, $J=7.2\text{ Hz}, 10.8\text{ Hz}$, 1H), 2.78 (m, 1H), 2.27 (m, 1H), 2.02 (m, 1H), 1.23–1.97 (m, 12H); ^{13}C NMR (CDCl_3 , 100 MHz) δ u: 138.1, 123.9, 97.1, 82.5, 47.5, 41.2, 37.8, 33.6, 30.8, 29.2, 27.9, 25.6; d: 133.7, 131.8, 129.2, 129.1, 128.2, 127.7, 72.1, 52.1; HRMS calcd for $\text{C}_{24}\text{H}_{26}\text{O}_2\text{S}$ (M^+) 378.1654, found 378.1658.

Epoxide 30

To a stirred solution of benzenesulfonate **29** (9.1 g, 23.1 mmol) in CH_2Cl_2 (100 mL) was added mCPBA (70%, 6.82 g, 27.7 mmol). The resulting mixture was stirred at ambient temperature for 40 h. The reaction mixture then was partitioned between 2 N aqueous NaOH and ether. The combined organic extract was dried (Na_2SO_4) and concentrated. The crude product was chromatographed to give 9.0 g of epoxide **30** (95% yield) as a colorless oil, TLC R_f = 0.53 (PE:MTBE, 1:1). IR (neat, cm^{-1}): 2935 (s), 1448 (m), 1362 (s), 1185 (s); ^1H NMR (400 MHz, CDCl_3) δ major isomer: 7.27-7.60 (m, 10H), 4.60 (dd, J = 10.0 Hz, 2.4 Hz, 1H), 3.16 (m, 1H), 2.77 (t, J = 4.6 Hz, 1H), 2.46 (dd, J = 4.6 Hz, 2.4 Hz, 1H), 1.36-2.30 (m, 12H); minor isomer: 7.27-7.60 (m, 10H), 4.40 (dd, J = 10.4 Hz, 2.4 Hz, 1H), 3.25 (m, 1H), 2.74 (t, J = 4.4 Hz, 1H), 2.40 (dd, J = 4.4 Hz, 2.4 Hz, 1H), 1.36-2.30 (m, 12H); ^{13}C NMR (CDCl_3 , 100 MHz) δ (two isomers are reported together): u: 137.7, 137.6, 123.3, 89.0, 88.5, 85.9, 85.4, 47.4, 45.8, 45.1, 44.3, 42.2, 35.5, 34.8, 31.5, 31.2, 26.1, 22.2, 22.1, 22.0, 21.9; d: 133.7, 131.8, 129.3, 128.3, 128.2, 128.1, 128.0, 127.7, 89.3, 87.9, 49.8, 49.1; HRMS calcd for $\text{C}_{24}\text{H}_{26}\text{O}_4\text{S}$ (M^+) 410.1552, found 410.1547.

Ketone 31

To a stirred solution of methyl phenyl sulfone (1.22 g, 7.79 mmol) in 10 mL of dry THF was added ⁿBuLi (2.25 M, 3.46 mL) dropwise at -78 °C under N₂. After stirring for 5 min, a solution of BF₃·Et₂O (1.01 g, 7.14 mmol) in 1 mL of dry THF was added, followed by the addition of epoxide **30** (2.66 g, 6.49 mmol) in 5 mL of dry THF dropwise. The cold bath was removed, and the reaction mixture was stirred at ambient temperature for 70 min. The reaction mixture was partitioned between saturated aqueous NH₄Cl and ether. The combined organic extract was dried (Na₂SO₄) and concentrated. The crude product was chromatographed to give 2.64 g of secondary alcohol as a colorless oil. This alcohol was dissolved immediately in 40 mL of CH₂Cl₂, and to this solution Dess-Martin reagent (2.18 g, 5.15 mmol) was added. The resulting mixture was stirred at room temperature for 30 min. Silica gel (8.0 g) was added and the mixture was concentrated. The residual solid was chromatographed to give 2.47 g of ketone **31** (68% yield for two steps) as a colorless oil, TLC *R_f* = 0.47 (PE:MTBE, 1:2). IR (neat, cm⁻¹): 2932 (s), 1720 (s), 1447 (s), 1359 (s), 1308 (s), 1185 (s), 1150 (s); ¹H NMR (400 MHz, CDCl₃) δ 7.86-7.89 (m, 4H), 7.51-7.65 (m, 6H), 7.28-7.39 (m, 5H), 4.74 (dd, *J*=10.8 Hz, 3.2 Hz, 1H), 3.31-3.45 (m, 2H), 2.81-3.01 (m, 4H), 1.36-2.04 (m, 10H); ¹³C NMR (CDCl₃, 100 MHz) δ u: 203.7, 139.0, 137.5, 122.9, 89.1, 85.7, 50.9, 50.6, 43.8, 36.4,

34.0, 31.5, 25.5, 22.0, 21.6; d: 134.0, 133.9, 131.8, 129.5, 129.4, 128.4, 128.1, 127.8, 127.5, 87.0; HRMS calcd for $C_{31}H_{32}O_6NaS_2$ (M+Na) 587.1538, found 587.1539.

Ketal 14

A solution of ketone **31** (1.50 g, 2.66 mmol), dry ethylene glycol (1.65 g, 26.6 mmol), freshly-distilled triethylformate (1.18 g, 7.98 mmol), *p*-toluenesulfonic acid monohydrate (50.6 mg, 0.266 mmol) in dry THF (2.5 mL) was heated at 45 °C under N_2 for 39 h. The reaction mixture was partitioned between saturated aqueous $NaHCO_3$ and ether. The combined organic extract was dried (Na_2SO_4) and concentrated. The crude product was chromatographed to give 1.33 g of ketal **14** (82% yield) as a colorless oil, TLC R_f = 0.33 (PE:MTBE, 1:2). IR (neat, cm^{-1}): 2932 (s), 1447 (s), 1362 (s), 1307 (s), 1186 (s), 1148 (s); 1H NMR (400 MHz, $CDCl_3$) δ 7.89-7.92 (m, 2H), 7.71-7.74 (m, 2H), 7.48-7.63 (m, 4H), 7.30-7.42 (m, 7H), 4.43 (dd, $J=10.4$ Hz, 2.4 Hz, 1H), 3.74-3.90 (m, 4H), 3.09-3.25 (m, 2H), 1.38-2.36 (m, 14H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ u: 139.4, 138.1, 123.6, 110.0, 90.7, 85.7, 65.0, 64.7, 51.7, 44.6, 43.0, 34.7, 31.6, 31.1, 26.6, 22.4, 22.0; d: 133.7, 133.6, 132.0, 129.3, 128.5, 128.2, 128.1, 127.7, 89.9; HRMS calcd for $C_{33}H_{36}O_7NaS_2$ (M+Na) 631.1800, found 631.1796.

Sulfone 15

To a stirred solution of ketal **14** (555 mg, 0.913 mmol) in dry THF (10 mL) was added n BuLi (1.8 M, 0.56 mL) at $-78\text{ }^{\circ}\text{C}$ under N_2 . The cold bath was removed and the reaction mixture was stirred at ambient temperature for 1 h. The reaction was partitioned between saturated aqueous NH_4Cl and ether. The combined organic extract was dried (Na_2SO_4) and concentrated. The crude product was chromatographed to give 378 mg of sulfone **15** (92% yield) as a colorless oil, TLC $R_f = 0.54$ (PE:MTBE, 1:2). IR (neat, cm^{-1}): 2929 (s), 1490 (m), 1446 (s), 1304 (s), 1145 (s); ^1H NMR (400 MHz, CDCl_3) δ 7.83-7.91 (m, 2H), 7.43-7.52 (m, 3H), 7.25 (br, 5H), 3.71-3.92 (m, 4H), 3.26 (m, 1H), 2.28-2.45 (m, 2H), 1.26-2.03 (m, 13H); ^{13}C NMR (CDCl_3 , 100 MHz) δ u: 137.3, 123.5, 107.7, 96.0, 82.9, 64.5, 64.1, 45.6, 39.5, 39.4, 34.0, 30.0, 29.0, 27.3, 23.0; d: 133.7, 131.6, 129.3, 129.1, 128.1, 127.8, 64.0, 44.6; HRMS calcd for $\text{C}_{27}\text{H}_{30}\text{O}_4\text{S}$ (M^+) 450.1865, found 450.1865.

Benzenesulfonate 32

Same procedure as for the preparation of benzenesulfonate **29**. Clear oil (91% yield), TLC R_f = 0.62 (PE:MTBE, 3:1). IR (neat, cm^{-1}): 2940 (s), 1490 (w), 1448 (m), 1365 (s), 1186 (s); ^1H NMR (400 MHz, CDCl_3) δ 7.92-7.96 (m, 2H), 7.49-7.64 (m, 3H), 7.25-7.41 (m, 5H), 5.88 (m, 1H), 5.03-5.12 (m, 2H), 4.39 (dd, $J=11.2$ Hz, 4.4 Hz, 1H), 2.60 (dd, $J=13.6$ Hz, 5.2 Hz, 1H), 2.02 (dd, $J=13.6$ Hz, 8.4 Hz, 1H), 1.20-2.00 (m, 8H); ^{13}C NMR (CDCl_3 , 100 MHz) δ u: 138.1, 123.7, 118.7, 89.9, 85.8, 43.0, 42.5, 36.0, 29.9, 24.5, 21.9; d: 133.7, 133.5, 131.9, 129.3, 128.3, 128.1, 127.7, 86.3; HRMS calcd for $\text{C}_{23}\text{H}_{24}\text{O}_3\text{NaS}$ ($\text{M}+\text{Na}$) 403.1344, found 403.1333.

Methanesulfonate 16

Same procedure as for the preparation of methanesulfonate **13**. Clear oil (85% yield), TLC R_f = 0.14 (PE:MTBE, 1:1). IR (neat, cm^{-1}): 2940 (s), 1448 (m), 1357 (s), 1176 (s); ^1H NMR (400 MHz, CDCl_3) δ 7.94 (d, $J=7.2$ Hz, 2H), 7.65 (t, $J=7.2$ Hz, 1H), 7.53-7.57 (m, 2H), 7.26-7.41 (m, 5H), 4.44-4.53 (m, 2H), 4.39 (dd, $J=11.2$ Hz, 4.0 Hz, 1H), 2.99 (s,

3H), 1.26-2.31 (m, 10H); ^{13}C NMR (CDCl_3 , 100 MHz) δ u: 137.5, 122.8, 87.9, 86.9, 67.3, 41.8, 37.5, 36.9, 29.5, 24.2, 21.7; d: 133.9, 131.8, 129.4, 128.4, 128.3, 127.6, 86.2, 37.4; HRMS calcd for $\text{C}_{23}\text{H}_{26}\text{O}_6\text{NaS}_2$ ($\text{M}+\text{Na}$) 485.1069, found 485.1083.

Sulfone 17

Same procedure as for the preparation of sulfone 3. White solid: mp = 119-120°C (85% yield), TLC R_f = 0.59 (PE:MTBE, 1:1). IR (film, cm^{-1}): 2933 (s), 1489 (m), 1446 (s), 1302 (s), 1146 (s), 1086 (m); ^1H NMR (400 MHz, CDCl_3) δ 7.90-7.94 (m, 2H), 7.54-7.68 (m, 3H), 7.26-7.42 (m, 5H), 3.55 (m, 1H), 2.70 (m, 1H), 1.19-2.30 (m, 12H); ^{13}C NMR (CDCl_3 , 100 MHz) δ u: 138.8, 123.7, 94.1, 82.0, 42.6, 38.5, 32.6, 24.8, 24.0, 22.6, 20.4; d: 133.8, 131.8, 129.4, 128.7, 128.3, 127.9, 63.7, 45.9; HRMS calcd for $\text{C}_{23}\text{H}_{24}\text{O}_2\text{S}$ (M^+) 364.1497, found 364.1513.

Benzenesulfonate 33

Same procedure as for the preparation of benzenesulfonate 29. Clear oil (92% yield), TLC R_f = 0.65 (PE:MTBE, 3:1). IR (neat, cm^{-1}): 2976 (s), 1641 (w), 1490 (m), 1448 (m),

1362 (s), 1176 (s); ^1H NMR (400 MHz, CDCl_3) δ 7.89-7.93 (m, 2H), 7.38-7.56 (m, 3H), 7.25-7.35 (m, 5H), 5.85 (m, 1H), 5.01-5.08 (m, 2H), 4.61 (t, $J=5.6$ Hz, 1H), 2.27 (dd, $J=14.0$ Hz, 7.2 Hz, 1H), 2.10 (dd, $J=14.0$ Hz, 7.2 Hz, 1H), 1.61-2.02 (m, 6H); ^{13}C NMR (CDCl_3 , 100 MHz) δ u: 137.2, 123.5, 118.4, 90.2, 84.5, 47.1, 41.9, 35.5, 30.4, 20.1; d: 133.7, 133.6, 131.8, 129.1, 128.2, 128.0, 127.9, 87.5; HRMS calcd for $\text{C}_{22}\text{H}_{22}\text{O}_3\text{NaS}$ ($\text{M}+\text{Na}$) 389.1187, found 389.1207.

Methanesulfonate 18

Same procedure as for the preparation of methanesulfonate 13. Clear oil (84% yield), TLC R_f = 0.13 (PE:MTBE, 1:1). IR (neat, cm^{-1}): 2960 (s), 1449 (m), 1359 (s), 1175 (s); ^1H NMR (400 MHz, CDCl_3) δ 7.90-7.94 (m, 2H), 7.61 (m, 1H), 7.48-7.52 (m, 2H), 7.27-7.39 (m, 5H), 4.62 (t, $J=6.4$ Hz, 1H), 4.46 (t, $J=6.8$ Hz, 2H), 2.99 (s, 3H), 1.63-2.17 (m, 8H); ^{13}C NMR (CDCl_3 , 100 MHz) δ u: 136.8, 122.8, 88.0, 85.8, 67.6, 45.8, 36.4, 36.3, 29.1, 19.7; d: 133.8, 131.7, 129.3, 128.3, 127.7, 87.4, 37.2; HRMS calcd for $\text{C}_{22}\text{H}_{24}\text{O}_6\text{NaS}_2$ ($\text{M}+\text{Na}$) 471.0912, found 471.0893.

Sulfone 19

Same procedure as for the preparation of sulfone 3. Colorless crystals (86% yield): mp = 83-84°C (% yield), TLC R_f = 0.58 (PE:MTBE, 1:1). IR (neat, cm^{-1}): 2958 (s), 2868 (m), 2221 (w), 1490 (m), 1446 (s), 1302 (s), 1146 (s), 1086 (s); ^1H NMR (400 MHz, CDCl_3) δ 7.90-7.94 (m, 2H), 7.53-7.64 (m, 3H), 7.24-7.35 (m, 5H), 3.01-3.11 (m, 2H), 1.29-2.26 (m, 10H); ^{13}C NMR (CDCl_3 , 100 MHz) δ u: 138.5, 123.8, 96.6, 80.9, 49.3, 40.7, 39.6, 33.1, 28.8, 25.8; d: 133.8, 131.6, 129.3, 128.8, 128.2, 127.7, 71.9, 53.4; HRMS calcd for $\text{C}_{22}\text{H}_{22}\text{O}_2\text{S}$ (M^+) 350.1341, found 350.1348.

References:

1. Still, W. C.; Kahn, M.; Mitra, A. *J. Org. Chem.* **1978**, *43*, 2923.
2. Taber, D. F. *J. Org. Chem.* **1982**, *47*, 1351.
3. Davies, M. T.; Dobson, D. F.; Hayman, D. F.; Jackman, G. B.; Lester, M. G.; Petrow, V.; Stephenson, O.; Webb, A. A.; *Tetrahedron* **1962**, *18*, 751.
4. The buffer solution is prepared by adding 4.5 mL of glacial acetic acid to 1 L of a 0.1M solution of K_2CO_3 .
5. Tu, Y.; Frohn, M.; Wang, Z.; Shi, Y. *Org. Syn.* **2003**, *80*, 1.
6. Morisson V.; Barnier, J. P.; Blanco, L. *Tetrahedron* **1998**, *54*, 7749.

Current Data Parameters
 NAME b0283h
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20031023
 Time 8.16
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zg
 TD 32768
 SOLVENT CDCl3
 NS 8
 DS 0
 SMH 5592.841 Hz
 FIDRES 0.170680 Hz
 AQ 2.9295092 sec
 RG 20.2
 DW 89.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 5.0000000 sec

===== CHANNEL f1 ===
 NUC1 1H
 P1 9.00 usec
 PL1 0.00 dB
 SFO1 400.1320007 MHz

F2 - Processing parameters
 SI 16384
 SF 400.1300197 MHz
 WDW EM
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00

Current Data Parameters
 NAME b0283c13
 EXPNO 13
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20031023
 Time 8.22
 INSTRUM spect
 PROHD 5 mm QNP 1H
 PULPROG jvert
 TD 65536
 SOLVENT CDCl3
 NS 68
 DS 0
 SMH 27173.912 Hz
 FIDRES 0.414641 Hz
 AQ 1.2059124 sec
 RG 11585.2
 DW 18.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 6.00000000 sec
 D11 0.03000000 sec
 D13 0.00000300 sec
 D20 0.00700000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 10.50 usec
 P2 21.00 usec
 PL1 0.00 dB
 SFO1 100.6242995 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 P1 10.50 usec
 P2 21.00 usec
 PL1 0.00 dB
 SFO1 100.6242995 MHz

F2 - Processing parameters

SI 32768
 SF 100.6127584 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.00

140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

Current Data Parameters
 NAME b0110h101
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20030928
 Time 13.12
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zg
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 0
 SMH 5592.841 Hz
 FIDRES 0.170680 Hz
 AQ 2.9295092 sec
 RG 40.3
 DW 89.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 5.0000000 sec

===== CHANNEL f1 ===
 NUC1 1H
 P1 9.00 usec
 PL1 0.00 dB
 SFO1 400.1320007 MHz

F2 - Processing parameters
 SI 16384
 SF 400.1300180 MHz
 WDW EM
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00

135.208
131.927
128.453
128.169
123.487
118.234

91.547
86.149
78.252
77.544
77.227
76.909

47.055
44.361
34.577
33.679
27.008
22.974
22.121

Current Data Parameters
NAME b0110c1301
EXPNO 13
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030928
Time 13.18

INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG jvert
TD 65536
SOLVENT CDCl3
NS 751
DS 0
SWH 27173.912 Hz
FIDRES 0.414641 Hz
AQ 1.2059124 sec
RG 7298.2
DW 18.400 usec
DE 4.50 usec
TE 300.0 K
D1 6.00000000 sec
D11 0.03000000 sec
D13 0.00000300 sec
D20 0.00700000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 10.50 usec
F2 21.00 usec
PL1 0.00 dB
SFO1 100.6242995 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 95.00 usec
PL2 120.00 dB
PL12 18.00 dB
SFO2 400.1320007 MHz

F2 - Processing parameters
SI 32768
SF 100.6127534 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

HPLC Condition:

Column: Chiralcel OD
Chiral Technologies Inc.

Eluent: Hexane / Isopropanol (99 / 1)

Flow rate: 1.0 mL / min

Peak No.	Result	Ret. Time (min)	Area (Counts)
1	50.0668	33.713	11003448
2	49.9120	39.339	10969420

Racemate

Peak No.	Result	Ret. Time (min)	Area (Counts)
1	93.0117	33.494	18160704
2	6.8553	38.630	1338514

Reaction material

Current Data Parameters
 NAME b0146h101
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20031105
 Time 16.40
 INSTRUM spect
 PROBED 5 mm QNP 1H
 PULPROG zg
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 0
 SMH 5592.841 Hz
 FIDRES 0.170680 Hz
 AQ 2.9295092 sec
 RG 32
 INW 89.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 5.0000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 9.00 usec
 PL1 0.00 dB
 SFO1 400.1320007 MHz

F2 - Processing parameters
 SI 16384
 SF 400.1300191 MHz
 WDW EM
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00

134.897
 131.927
 128.432
 128.127
 123.560
 118.062
 91.111
 86.380
 77.550
 77.232
 76.915
 75.338
 44.756
 43.693
 35.468
 32.793
 24.853
 22.548

Current Data Parameters
 NAME B0146c1301
 EXPNO 13
 PROCNO 1
 F2 - Acquisition Parameters
 Date_ 20031105
 Time 16.47
 INSTRUM spect
 PROHD 5 mm QNP 1H
 PULPROG jvert
 TD 65536
 SOLVENT CDC13
 NS 280
 DS 0
 SMH 27173.912 Hz
 FIDRES 0.414641 Hz
 AQ 1.2059124 sec
 RG 9195.2
 DM 18.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 6.00000000 sec
 D11 0.03000000 sec
 D13 0.00000300 sec
 D20 0.00700000 sec
 ===== CHANNEL f1 =====
 NUC1 13C
 P1 10.50 usec
 P2 21.00 usec
 PL1 0.00 dB
 SFO1 100.6242995 MHz
 ===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 P1 10.00 usec
 P2 120.00 dB
 PL1 18.00 dB
 SFO2 400.1320007 MHz
 F2 - Processing parameters
 SI 32768
 SF 100.6127542 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.00

140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

Current Data Parameters
 NAME b0235h1
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20030924
 Time 8.35
 INSTRUM spect
 PROHD 5 mm QNP 1H
 PULPROG zg
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 0
 SMH 5592.841 Hz
 FIDRES 0.170680 Hz
 AQ 2.9295092 sec
 RG 20.2
 DW 89.400 use
 DE 4.50 use
 TE 300.0 K
 D1 5.0000000 sec

===== CHANNEL f1 ===
 NUC1 1H
 P1 9.00 use
 PL1 0.00 dB
 SFO1 400.1320007 MHz

F2 - Processing parameters
 SI 16384
 SF 400.1300201 MHz
 WDW EM
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00

160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 ppm

Current Data Parameters
 NAME b0411h101
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20040415
 Time 10.48
 INSTRUM spect
 PROBED 5 mm QNP 1H
 PULPROG zg
 TO 32768
 SOLVENT CDCl3
 NS 16
 DS 0
 SMH 5592.841 Hz
 FIDRES 0.170680 Hz
 AQ 2.9295092 sec
 RG 10.1
 DW 89.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 5.00000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 9.00 usec
 PL1 0.00 dB
 SFO1 400.1320007 MHz

F2 - Processing Parameters
 SI 16384
 SF 400.1300429 MHz
 WDW EM
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00

Current Data Parameters
 NAME b04111c1301
 EXPNO 13
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20040415
 Time 10.53
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG jvert
 TD 65536
 SOLVENT CDCl3
 NS 257
 DS 0
 SWH 27173.912 Hz
 FIDRES 0.414641 Hz
 AQ 1.2059124 sec
 RG 8192
 DW 18.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 6.00000000 sec
 D11 0.03000000 sec
 D13 0.00000300 sec
 D20 0.00700000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 10.50 usec
 P2 21.00 usec
 PL1 0.00 dB
 SFO1 100.6242995 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 95.00 usec
 PL2 120.00 dB
 PL12 18.00 dB
 SFO2 400.1320007 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127659 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.00

45.900
 44.841
 34.978
 32.985
 24.808
 22.436

91.260
 87.095
 74.843

137.449
 131.753
 131.117
 128.399
 128.085
 127.879
 126.520
 123.527

200 180 160 140 120 100 80 60 40 20 0 -20 -40 ppm

Current Data Parameters
 NAME b04129h1
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20040420
 Time 10.25
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zg
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 0
 SMH 5592.841 Hz
 FIDRES 0.170680 Hz
 AQ 2.9295092 sec
 RG 10.1
 DW 89.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 5.00000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 9.00 usec
 PL1 0.00 dB
 SFO1 400.1320007 MHz

F2 - Processing parameters
 SI 16384
 SF 400.1300235 MHz
 WDW EM
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00

Current Data Parameters
 NAME b04129c13
 EXPNO 13
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20040420
 Time 10.31
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG jvert
 TD 65536
 SOLVENT CDCl3
 NS 260
 DS 0
 SWH 27173.912 Hz
 FIDRES 0.414641 Hz
 AQ 1.2059124 sec
 RG 8192
 DW 18.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 6.00000000 sec
 D11 0.03000000 sec
 D13 0.00000300 sec
 D20 0.00700000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 10.50 usec
 P2 21.00 usec
 PL1 0.00 dB
 SF01 100.6242995 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCD2 95.00 usec
 PL2 120.00 dB
 PL12 18.00 dB
 SF02 400.1320007 MHz

F2 - Processing parameters

SI 32768
 SF 100.6127592 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.00

Current Data Parameters
 NAME b0113h1
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20031120
 Time 8.58
 INSTRUM spect
 PROHD 5 mm QNP 1H
 FULPROG zg
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 0
 SWH 5592.841 Hz
 FIDRES 0.170680 Hz
 AQ 2.9295092 sec
 RG 80.6
 DW 89.400 use
 DE 4.50 use
 TE 300.0 K
 D1 5.00000000 sec

===== CHANNEL f1 ===
 NUC1 1H
 P1 9.00 use
 PL1 0.00 dB
 SFO1 400.1320007 MHz

F2 - Processing parameters
 SI 16384
 SF 400.1300167 MHz
 WDW EM
 SSB 0
 LB 0
 GB 0
 PC 1.00

161.869
148.864
134.500
134.042
131.967
129.742
128.596
128.323
123.468
122.573
118.654
90.925
85.284
82.603
77.545
77.228
76.910
45.041
44.542
34.723
31.248
26.744
22.642
22.127

Current Data Parameters
NAME 60113c13
EXPNO 13
PROCNO 1
F2 - Acquisition Parameters
Date_ 20031121
Time 8.10
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl₃
NS 6546
DS 0
SWH 27173.912 Hz
FIDRES 0.414641 Hz
AQ 1.2059124 sec
RG 8192
DQW 18.400 usec
DE 4.50 usec
TE 300.0 K
D1 3.00000000 sec
D11 0.00000000 sec
D13 0.00000000 sec
D20 0.00700000 sec

CHANNEL f1
NUC1 ¹³C
P1 10.50 usec
P2 21.00 usec
PL1 0.00 dB
SFO1 100.6242995 MHz

CHANNEL f2
CPDPRG2 waltz16
NUC2 ¹H
PCPD2 95.00 usec
PL2 120.00 dB
PL12 18.00 dB
SFO2 400.132007 MHz
F2 - Processing parameters
SI 32768
SF 100.617526 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

Current Data Parameters
 NAME b0411502h1
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20040512
 Time 8.04

INSTRUM spect

PROBHD 5 mm QNP 1H

PULPROG zg

TD 32768

SOLVENT CDCl3

AS 16

DS 0

SWH 5592.841 Hz

FIDRES 0.170680 Hz

AQ 2.9295092 sec

RG 10.1

DW 89.400 usec

DE 4.50 usec

TE 300.0 K

DI 5.0000000 sec

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

NUC1 1H

P1 9.00 usec

PL1 0.00 dB

SFO1 400.1320007 MHz

F2 - Processing parameters

SI 16384

SF 400.1300153 MHz

WDW EM

SSB 0

LB 0.00 Hz

GB 0

PC 1.00

123.470
 127.950
 128.307
 131.765
 92.009
 85.679
 78.162
 77.549
 77.230
 76.911
 63.093
 46.637
 35.776
 34.412
 33.783
 28.031
 26.889
 22.865
 22.198

Current Data Parameters
 NAME b0411502c13
 EXPNO 13
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20040512
 Time 8.10
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG jvert
 TD 65536
 SOLVENT CDCl3
 NS 461
 DS 0
 SMH 27173.912 Hz
 FIDRES 0.414641 Hz
 AQ 1.2059124 sec
 RG 10321.3
 DW 18.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 6.00000000 sec
 D11 0.03000000 sec
 D13 0.00000300 sec
 D20 0.00700000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 10.50 usec
 P2 21.00 usec
 PL1 0.00 dB
 SFO1 100.6242995 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 95.00 usec
 PL2 120.00 dB
 PL12 18.00 dB
 SFO2 400.1320007 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127659 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.00

Current Data Parameters
 NAME b04121hl
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20040415
 Time 10.13
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zg
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 0
 SWH 5592.841 Hz
 FIDRES 0.170680 Hz
 AQ 2.9295092 sec
 RG 5
 DW 89.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 5.0000000 sec

===== CHANNEL f1 ===
 NUC1 1H
 P1 9.00 usec
 PL1 0.00 dB
 SFO1 400.1320007 MHz

F2 - Processing parameters
 SI 16384
 SF 400.1300201 MHz
 WDW EM
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00

Handwritten notes:
 /s/s/ (near 3 ppm)
 / (near 6 ppm)
 / (near 7 ppm)

Current Data Parameters
 NAME b04121c13
 EXPNO 13
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20040415
 Time 10.18

INSTRUM spect
 PROBD 5 mm QNP 1H
 PULPROG jvert
 TD 65536
 SOLVENT CDCl3
 NS 140
 DS 0

SWH 27173.912 Hz
 FIDRES 0.414641 Hz
 AQ 1.2059124 sec
 RG 7298.2
 DW 18.400 usec
 DE 4.50 usec
 TE 300.0 K

D1 6.00000000 sec
 D11 0.03000000 sec
 D13 0.00000300 sec
 D20 0.00700000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 10.50 usec
 P2 21.00 usec
 PL1 0.00 dB
 SF01 100.6242995 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 95.00 usec
 PL2 120.00 dB
 PL12 18.00 dB
 SF02 400.1320007 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127741 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.00

Current Data Parameters
 NAME b04123hl
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters:

Date_ 20040414
 Time 14.15
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zg
 TD 32768
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 5592.841 Hz
 FIDRES 0.170680 Hz
 AQ 2.929592 sec
 RG 20.2
 DW 89.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 5.0000000 sec

===== CHANNEL f1 ===
 NUC1 1H
 P1 9.00 usec
 PL1 0.00 dB
 SFO1 400.1320007 MHz

F2 - Processing parameters
 SI 16384
 SF 400.1300146 MHz
 WDW EM
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00

Current Data Parameters
 NAME b04123c13
 EXPNO 13
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20040414
 Time 14.18

INSTRUM spect
 PROHD 5 mm QNP 1H
 PULPROG jvert
 TD 65536
 SOLVENT CDCl3
 NS 35
 DS 0
 SWH 27173.912 Hz
 FIDRES 0.414641 Hz
 AQ 1.2059124 sec
 RG 9195.2
 DW 18.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 6.00000000 sec
 D11 0.03000000 sec
 D13 0.00000300 sec
 D20 0.00700000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 10.50 usec
 P2 21.00 usec
 PL1 0.00 dB
 SF01 100.6242995 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCDP2 95.00 usec
 PL2 120.00 dB
 PL12 18.00 dB
 SF02 400.1320007 MHz

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

Current Data Parameters
 NAME b04127h1
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20040418
 Time 12.28

INSTRUM spect
 PROBED 5 mm QNP 1H
 PULPROG zg

TD 32768
 SOLVENT CDC13
 NS 16

DS 0
 SMH 5592.841 Hz
 FIDRES 0.170680 Hz

AQ 2.9295092 sec
 RG 20.2
 DW 89.400 usec

DE 4.50 usec
 TE 300.0 K
 D1 5.0000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 9.00 usec

PL1 0.00 dB
 SF01 400.1320007 MHz

F2 - Processing parameters
 SI 16384
 SF 400.1300232 MHz

WDW EM
 SSB 0
 LB 0.00 Hz

GB 0
 PC 1.00

Current Data Parameters
 NAME b04127c13
 EXPNO 13
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20040418
 Time 12.33
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG jvert
 TD 65536
 SOLVENT CDCl3
 NS 541
 DS 0
 SMH 27173.912 Hz
 FIDRES 0.414641 Hz
 AQ 1.2059124 sec
 RG 5792.6
 DW 18.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 6.00000000 sec
 D11 0.03000000 sec
 D13 0.00000300 sec
 D20 0.00700000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 10.50 usec
 P2 21.00 usec
 PL1 0.00 dB
 SFO1 100.624295 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 95.00 usec
 PL2 120.00 dB
 PL12 18.00 dB
 SFO2 400.1320007 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127559 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.00

Current Data Parameters
 NAME b04135h1
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20040422
 Time 8.08

INSTRUM spect
 PROBED 5 mm QNP 1H

PULPROG zg
 TD 32768

SOLVENT CDCl3
 NS 16

DS 0
 SMH 5592.841 Hz

FIDRES 0.170680 Hz
 AQ 2.9295092 sec

RG 10.1
 DW 89.400 usec

DE 4.50 usec
 TE 300.0 K

D1 5.0000000 sec

===== CHANNEL f1 =====
 NUC1 1H

P1 9.00 usec
 PL1 0.00 dB

SFO1 400.1320007 MHz

F2 - Processing parameters
 SI 16384

SP 400.1300532 MHz
 WDW EM

SSB 0
 LB 0.00 Hz

GB 0
 PC 1.00

Current Data Parameters
 NAME b04135c13
 EXPNO 13
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20040422
 Time 8.13
 INSTRUM spect
 PROHD 5 mm QNP 1H
 PULPROG jvert
 TD 65536
 SOLVENT CDCl3
 NS 236
 DS 0
 SWH 27173.912 Hz
 FIDRES 0.414641 Hz
 AQ 1.2059124 sec
 RG 7298.2
 DW 18.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 6.00000000 sec
 D11 0.03000000 sec
 D13 0.00000300 sec
 D20 0.00700000 sec

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

NUC1 13C
 P1 10.50 usec
 F2 21.00 usec
 PL1 0.00 dB
 SFO1 100.6242995 MHz

===== CHANNEL f2 =====

CPDPRG2 waltz16
 NUC2 1H
 P2 95.00 usec
 PL2 120.00 dB
 PL12 18.00 dB
 SFO2 400.1320007 MHz

F2 - Processing parameters

SI 32768
 SF 100.6127675 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.00

40.222
 36.801
 36.210
 29.565
 27.571
 25.491
 24.504
 22.288
 22.148

86.451
 77.545
 77.228
 76.909
 72.977
 71.636
 71.263

139.509
 138.822
 128.465
 128.306
 127.740
 127.581
 127.559
 127.299

Current Data Parameters
 NAME b04139h1
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20040425
 Time 11.02
 INSTRUM spect
 PROBD 5 mm QNP 1H
 PULPROG zg
 TD 32768
 SOLVENT CDC13
 NS 16
 DS 0
 SSW 5592.841 Hz
 FIDRES 0.170680 Hz
 AQ 2.9295092 sec
 RG 50.8
 LW 89.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 5.0000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 9.00 usec
 PL1 0.00 dB
 SFO1 400.1320007 MHz

F2 - Processing parameters
 SI 16384
 SF 400.1300133 MHz
 WDW EM
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00

ss

Current Data Parameters
 NAME 504139c13
 EXPNO 13
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20040423
 Time 14.16
 INSTRUM spect
 PROHD 5 mm QNP 1H
 PULPROG jvert
 TD 65536
 SOLVENT CDCl3
 NS 513
 DS 0
 SWH 27173.912 Hz
 FIDRES 0.414641 Hz
 AQ 1.2059124 sec
 RG 8192
 DW 18.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 6.00000000 sec
 D11 0.03000000 sec
 D13 0.00000300 sec
 D20 0.00700000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 10.50 usec
 P2 21.00 usec
 PL1 0.00 dB
 SF01 100.6242995 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 95.00 usec
 PL2 120.00 dB
 PL12 18.00 dB
 SF02 400.1320007 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127534 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.00

Current Data Parameters
 NAME b04145h1
 EXFNO 1
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20040426
 Time 8.00
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zg
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 0
 SMH 5592.841 Hz
 FIDRES 0.170680 Hz
 AQ 2.9295092 sec
 RG 20.2
 INW 89.400 use
 DE 4.50 use
 TE 300.0 K
 D1 5.00000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 9.00 use
 PL1 0.00 dB
 SFO1 400.1320007 MHz

F2 - Processing parameters
 SI 16384
 SF 400.1299982 MHz
 WDW EM
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00

21.533
 24.578
 26.713
 30.692
 31.212
 38.072
 39.242
 40.363
 50.074
 77.543
 77.225
 76.908

Current Data Parameters
 NAME b04153h1
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20040429
 Time 10.14
 INSTRUM spect
 PROBED 5 mm QNP 1H
 PULPROG zg
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 0
 SMH 5592.841 Hz
 FIDRES 0.170680 Hz
 AQ 2.9295092 sec
 RG 20.2
 DW 89.400 use
 DE 4.50 use
 TE 300.0 K
 D1 5.00000000 sec

===== CHANNEL f1 ===
 NUC1 1H
 P1 9.00 use
 PL1 0.00 dB
 SFO1 400.1320007 MHz

F2 - Processing parameters
 SI 16384
 SF 400.1300095 MHz
 WDW EM
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00

217.926

132.220
128.679

77.546
77.229
76.910

51.085

40.395
40.297
37.988
30.908
26.887
26.612
22.094
21.523
20.622
14.496

Current Data Parameters
NAME b04153c13
EXPNO 13
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040429
Time 10.19
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG jvert
TD 65536
SOLVENT CDC13
NS 389
DS 0
SWH 27173.912 Hz
FIDRES 0.414641 Hz
AQ 1.2059124 sec
RG 9195.2
DM 18.400 usec
DE 4.50 usec
TE 300.0 K
D1 6.00000000 sec
D11 0.03000000 sec
D13 0.0000300 sec
D20 0.00700000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 10.50 usec
P2 21.00 usec
PL1 0.00 dB
SFO1 100.6242995 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 95.00 usec
PL2 120.00 dB
PL12 18.00 dB
SFO2 400.1320007 MHz

F2 - Processing parameters
SI 32768
SF 100.6127534 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

Current Data Parameters
 NAME b04157h1
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20040501
 Time 9.42
 INSTRUM spect
 PROBED 5 mm QNP 1H
 PULPROG zg
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 0
 SMH 5592.841 Hz
 FIDRES 0.170680 Hz
 AQ 2.9295092 sec
 RG 20.2
 DW 89.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 5.0000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 9.00 usec
 PL1 0.00 dB
 SFO1 400.132007 MHz

F2 - Processing parameters
 SI 16384
 SF 400.1300105 MHz
 WDW EM
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00

Handwritten notes: several slanted lines and a checkmark-like symbol.

218.357

77.543
77.226
76.908

51.127
40.477
40.436
37.972
31.887
30.942
30.098
26.894
24.646
24.131
22.800
21.699

Current Data Parameters
NAME b04157c13
EXPNO 13
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040501
Time 9.47

INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG jvert
TD 65536
SOLVENT CDCl3
NS 315
DS 0
SWH 27173.912 Hz
FIDRES 0.414641 Hz
AQ 1.2059124 sec
RG 7298.2
DW 18.400 usec
DE 4.50 usec
TE 300.0 K
D1 6.00000000 sec
D11 0.03000000 sec
D13 0.00000300 sec
D20 0.00700000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 10.50 usec
P2 21.00 usec
PL1 0.00 dB
SFO1 100.6242995 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 95.00 usec
PL2 120.00 dB
PL12 18.00 dB
SFO2 400.1320007 MHz

F2 - Processing parameters
SI 32768
SF 100.6127526 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

220 200 180 160 140 120 100 80 60 40 20 0 -20 ppm

Current Data Parameters
 NAME b02131h1
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20031109
 Time 11.47

INSTRUM spect
 PROBRD 5 mm QNP 1H
 PULPROG zg
 TD 32768
 SOLVENT CDCl3
 NS 8
 DS 0
 SMH 5592.841 Hz
 FIDRES 0.170680 Hz
 AQ 2.9295092 sec
 RG 5
 DM 89.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 5.0000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 9.00 usec
 PL1 0.00 dB
 SFO1 400.1320007 MHz

F2 - Processing parameters
 SI 16384
 SF 400.1300170 MHz
 WDW EM
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00

Current Data Parameters
 NAME b02131c13
 EXPNO 13
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20031109
 Time 12.00
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG jvert
 TD 65536
 SOLVENT CDCl3
 NS 104
 DS 0
 SWH 27173.912 Hz
 FIDRES 0.414641 Hz
 AQ 1.2059124 sec
 RG 9195.2
 DW 18.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 6.00000000 sec
 D11 0.03000000 sec
 D13 0.00000300 sec
 D20 0.00700000 sec

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

NUC1 13C
 P1 10.50 usec
 P2 21.00 usec
 PL1 0.00 dB
 SFO1 100.6242995 MHz

===== CHANNEL f2 =====

CPDPRG2 waltz16
 NUC2 1H
 PCPD2 95.00 usec
 PL2 120.00 dB
 PL12 18.00 dB
 SFO2 400.1320007 MHz

F2 - Processing parameters

SI 32768
 SF 100.6127758 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.00

44.622
 43.578
 34.163
 31.545
 26.128
 22.024
 21.971
 21.601

90.378
 88.346
 85.160

144.496
 134.845
 134.036
 131.671
 129.733
 128.168
 127.873
 127.573
 123.522
 118.654

Current Data Parameters
 NAME b0226101h1
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20040505
 Time 15.15

INSTRUM spect

PROBHD 5 mm QNP 1H

PULPROG zg

TD 32768

SOLVENT CDCl3

NS 16

DS 0

SMH 5592.841 Hz

FIDRES 0.170680 Hz

AQ 2.9295092 sec

RG 10.1

DW 89.400 usec

DE 4.50 usec

TE 300.0 K

D1 5.0000000 sec

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

NUC1 1H

P1 9.00 usec

PL1 0.00 dB

SFO1 400.1320007 MHz

F2 - Processing parameters

SI 16384

SF 400.1300143 MHz

WDW EM

SSB 0

LB 0.00 Hz

GB 0

PC 1.00

Current Data Parameters
 NAME b0226101c13
 EXPNO 13
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20040505
 Time 15.22
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG jvert
 TD 65536
 SOLVENT CDCl3
 NS 200
 DS 0
 SWH 27173.912 Hz
 FIDRES 0.414641 Hz
 AQ 1.2059124 sec
 RG 10321.3
 DW 18.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 6.00000000 sec
 D11 0.03000000 sec
 D13 0.00000300 sec
 D20 0.00700000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 10.50 usec
 P2 21.00 usec
 PL1 0.00 dB
 SFO1 100.6242995 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 95.00 usec
 PL2 120.00 dB
 PL12 18.00 dB
 SFO2 400.1320007 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127683 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.00

21.718
 21.833
 22.157
 25.665
 31.113
 36.214
 42.752
 52.107

76.914
 77.231
 77.551
 86.990
 87.603
 88.144

122.753
 127.772
 128.337
 128.424
 129.940
 131.746
 134.184
 144.991

201.587

220 200 180 160 140 120 100 80 60 40 20 -20 ppm

Current Data Parameters
 NAME b02263h1
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20031229
 Time 16.21
 INSTRUM spect
 PROBD 5 mm QNP 1H
 PULPROG zg
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 0
 SWH 5592.841 Hz
 FIDRES 0.170680 Hz
 AQ 2.9295092 sec
 RG 10.1
 DE 89.400 usec
 TE 4.50 usec
 D1 300.0 K
 D1 5.0000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 9.00 usec
 PL1 0.00 dB
 SFO1 400.1320007 MHz

F2 - Processing parameters
 SI 16384
 SF 400.1300173 MHz
 WDW EM
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00

200.564

131.726
128.320
128.124
128.066
123.122

91.252

80.183
77.550
77.231
76.913

46.459
35.697
34.253
31.905
29.756
28.099
27.277
26.583

Current Data Parameters
NAME b02263c13
EXPNO 13
PROCNO 1

F2 - Acquisition Parameters

Date_ 20031229
Time 16.28
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG jvert
TD 65536
SOLVENT CDCl3
NS 190
DS 0
SMH 27173.912 Hz
FIDRES 0.414641 Hz
AQ 1.2059124 sec
RG 9195.2
DW 18.400 usec
DE 4.50 usec
TE 300.0 K
D1 6.00000000 sec
D11 0.03000000 sec
D13 0.0000300 sec
D20 0.00700000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 10.50 usec
P2 21.00 usec
PL1 0.00 dB
SFO1 100.6242995 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 95.00 usec
PL2 120.00 dB
PL12 18.00 dB
SFO2 400.1320007 MHz

F2 - Processing parameters
SI 32768
SF 100.6127659 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

Current Data Parameters
 NAME b0229501h1
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20040427
 Time 8.49
 INSTRUM spect
 PROBD 5 mm QNP 1H
 PULPROG zg
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 0
 SMH 5592.841 Hz
 FIDRES 0.170680 Hz
 AQ 2.9295092 sec
 RG 5
 DW 89.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 5.00000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 9.00 usec
 PL1 0.00 dB
 SFO1 400.1320007 MHz

F2 - Processing parameters
 SI 16384
 SF 400.1300279 MHz
 WDW EM
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00

Current Data Parameters
 NAME b0229501c13
 EXPNO 13
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20040427
 Time 8.19
 INSTRUM spect
 PROHD 5 mm QNP 1H
 PULPROG jvert
 TD 65536
 SOLVENT CDCl3
 NS 228
 DS 0
 SWH 27173.912 Hz
 FIDRES 0.414641 Hz
 AQ 1.2059124 sec
 RG 8192
 DW 18.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 6.00000000 sec
 D11 0.03000000 sec
 D13 0.00000300 sec
 D20 0.00700000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 10.50 usec
 P2 21.00 usec
 PL1 0.00 dB
 SF01 100.6242995 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCDP2 95.00 usec
 PL2 120.00 dB
 PL12 18.00 dB
 SFO2 400.1320007 MHz

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

45.093
 44.058
 34.572
 32.063
 26.568
 22.461
 22.420

90.736
 89.128
 85.674
 77.967
 77.648
 77.330

138.257
 134.401
 134.007
 132.150
 129.602
 128.649
 128.368
 127.998
 123.944
 119.180

Current Data Parameters
 NAME b03193h101
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20040212
 Time 15.29
 INSTRUM spect
 PROBD 5 mm QNP 1H
 PULPROG zg
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 0
 SWH 5592.841 Hz
 FIDRES 0.170680 Hz
 AQ 2.9295092 sec
 RG 20.2
 DW 89.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 5.00000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 9.00 usec
 PL1 0.00 dB
 SFO1 400.1320007 MHz

F2 - Processing parameters
 SI 16384
 SF 400.1300163 MHz
 WDW EM
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00

Handwritten notes: *ssll*

Handwritten notes: *ssll*

Current Data Parameters
 NAME b03193c1301
 EXPNO 13
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20040212
 Time 15.33
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG jvert
 TD 65536
 SOLVENT CDCl3
 NS 257
 DS 0
 SWH 27173.912 Hz
 FIDRES 0.414641 Hz
 AQ 1.2059124 sec
 RG 7298.2
 DW 18.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 6.00000000 sec
 D11 0.03000000 sec
 D13 0.00000300 sec
 D20 0.00700000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 10.50 usec
 P2 21.00 usec
 PL1 0.00 dB
 SFO1 100.6242995 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 P1 95.00 usec
 P2 120.00 dB
 PL2 18.00 dB
 SFO2 400.1320007 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127592 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.00

Current Data Parameters
 NAME b040101h1
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20040512
 Time 15.19
 INSTRUM spect
 PROHD 5 mm QNP 1H
 PULPROG zg
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 0
 SWH 5592.841 Hz
 FIDRES 0.170680 Hz
 AQ 2.9295092 sec
 RG 20.2
 DW 89.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 5.0000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 9.00 usec
 PL1 0.00 dB
 SFO1 400.1320007 MHz

F2 - Processing parameters
 SI 16384
 SF 400.1300167 MHz
 WDW EM
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00

11

138.921
137.328
133.890
133.852
131.826
129.436
129.396
128.431
128.350
128.275
127.714
122.777

88.316
87.756
86.538
77.544
77.227
76.909

52.848
44.616
35.021
31.993
31.659
26.046
22.097
22.002

Current Data Parameters
NAME b040101c13
EXPNO 13
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040512
Time 15.30
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG jvert
TD 65536
SOLVENT CDCl3
NS 342
DS 0
SWH 27173.912 Hz
FIDRES 0.414641 Hz
AQ 1.2059124 sec
RG 10321.3
DW 18.400 usec
DE 4.50 usec
TE 300.0 K
D1 6.0000000 sec
D11 0.0300000 sec
D13 0.0000300 sec
D20 0.0070000 sec

Current Data Parameters
 NAME b040901hl
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20040512
 Time 15.13

INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zg
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 0
 SWH 5592.841 Hz
 FIDRES 0.170680 Hz
 AQ 2.9295092 sec
 RG 90.5
 LW 89.400 use
 DE 4.50 use
 TE 300.0 K
 D1 5.0000000 sec

===== CHANNEL f1 ===
 NUC1 1H
 P1 9.00 use
 PL1 0.00 dB
 SFO1 400.1320007 MHz

F2 - Processing parameters
 SI 16384
 SF 400.1300177 MHz
 WDW EM
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00

123.882
 128.003
 128.380
 128.631
 129.468
 131.828
 133.828
 138.902
 95.440
 84.482
 77.588
 77.235
 76.882
 59.994
 47.206
 36.839
 36.421
 33.692
 33.543
 32.154
 27.042
 26.198

Current Data Parameters
 NAME B0409c13
 EXPNO 13
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20040307
 Time 9.47
 INSTRUM spect
 PROHD 5 mm QNP 1H
 PULPROG jmod.sb
 TD 65536
 SOLVENT CDCl3
 NS 11678
 DS 0
 SWH 21834.061 Hz
 FIDRES 0.333161 Hz
 AQ 1.5008202 sec
 RG 16384
 DW 22.900 usec
 DE 28.62 usec
 TE 300.0 K
 FI 8.00 usec
 CNST2 145.0000000
 CNST11 1.0000000
 D20 0.0068966 sec
 HL1 13 dB
 D1 5.00000000 sec
 CPDPRG waltz16
 F31 100.00 usec
 D13 0.0000040 sec
 P2 16.0 usec
 SFO1 90.5648644 MHz
 NUCLEUS 13C

F2 - Processing parameters
 SI 65536
 SF 90.5557376 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 4.00

Current Data Parameters
 NAME b0320302h1
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20040511
 Time 15.19
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zg
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 0
 SWH 5592.841 Hz
 FIDRES 0.170680 Hz
 AQ 2.9295092 sec
 RG 35.9
 ZW 89.400 use
 DE 4.50 use
 TE 300.0 K
 D1 5.0000000 sec

===== CHANNEL f1 ===
 NUC1 1H
 P1 9.00 use
 PL1 0.00 dB
 SFO1 400.1320007 MHz

F2 - Processing parameters
 SI 16384
 SF 400.1300184 MHz
 WDW EM
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00

}}
 }}
 }}

Current Data Parameters
 NAME b03203c13
 EXPNO 13
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20040209
 Time 14.43

INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG jvert
 TD 65536
 SOLVENT CDCl3
 NS 207
 DS 0
 SWH 27173.912 Hz
 FIDRES 0.414641 Hz
 AQ 1.2059124 sec
 RG 11585.2
 DW 18.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 6.0000000 sec
 D11 0.0300000 sec
 D13 0.0000300 sec
 D20 0.0070000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 10.50 usec
 P2 21.00 usec
 PL1 0.00 dB
 SFO1 100.6242995 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 95.00 usec
 PL2 120.00 dB
 PL12 18.00 dB
 SFO2 400.1320007 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127584 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.00

Current Data Parameters
 NAME b0321701h1
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20040428
 Time 8.08

INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zg
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 0
 SWH 5592.841 Hz
 FIDRES 0.170680 Hz
 AQ 2.9295092 sec
 RG 10.1
 DW 89.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 5.0000000 sec

===== CHANNEL f1 ===
 NUC1 1H
 P1 9.00 usec
 PL1 0.00 dB
 SFO1 400.1320007 MHz

F2 - Processing parameters
 SI 16384
 SF 400.1300177 MHz
 EM 0
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00

Current Data Parameters
 NAME b0321701c13
 EXPNO 13
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20040428
 Time 8.13

INSTRUM spect
 PROBD 5 mm QNP 1H
 PULPROG jvert
 TD 65536
 SOLVENT CDCl3
 NS 377
 DS 0
 SMH 27173.912 Hz
 FIDRES 0.414641 Hz
 AQ 1.2059124 sec
 RG 9195.2
 DW 18.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 6.00000000 sec
 D11 0.03000000 sec
 D13 0.00000300 sec
 D20 0.00700000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 10.50 usec
 P2 21.00 usec
 PL1 0.00 dB
 SFO1 100.6242995 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 95.00 usec
 PL2 120.00 dB
 PL12 18.00 dB
 SFO2 400.1320007 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127650 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.00

Current Data Parameters
 NAME b0403h1
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20040504
 Time 9.19
 INSTRUM spect
 PROHD 5 mm QNP 1H
 PULPROG zg
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 0
 SWH 5592.841 Hz
 FIDRES 0.170680 Hz
 AQ 2.9295092 sec
 RG 20.2
 DW 89.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 5.0000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 9.00 usec
 PL1 0.00 dB
 SFO1 400.1320007 MHz

F2 - Processing parameters
 SI 16384
 SF 400.1300143 MHz
 WDW EM
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00

203.740

138.987
137.456
134.000
133.914
131.848
129.516
129.388
128.430
128.110
127.762
127.482
122.948

89.114
86.972
85.727
77.552
77.234
76.916

50.901
50.594
43.826
36.422
33.965
31.485
25.544
21.979
21.581

Current Data Parameters
NAME b0403c13
EXPNO 13
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040504
Time 9.25

INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG jvert
TD 65536
SOLVENT CDCl3
NS 355
DS 0
SWH 27173.912 Hz
FIDRES 0.414641 Hz
AQ 1.2059124 sec
RG 5792.6
DM 18.400 usec
DE 4.50 usec
TE 300.0 K
D1 6.00000000 sec
D11 0.03000000 sec
D13 0.00000300 sec
D20 0.00700000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 10.50 usec
P2 21.00 usec
PL1 0.00 dB
SF01 100.6242995 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 95.00 usec
PL2 120.00 dB
PL12 18.00 dB
SF02 400.1320007 MHz

F2 - Processing parameters
SI 32768
SF 100.6127592 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

Current Data Parameters
 NAME b03255h1
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20040225
 Time 8.03

INSTRUM spect
 PROBHD 5 mm Multinu
 PULPROG zg
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 0

SWH 5592.841 Hz
 FIDRES 0.170680 Hz
 AQ 2.9295092 sec
 RG 20.2
 DW 89.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 5.0000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 9.00 usec
 PL1 0.00 dB
 SFO1 400.1320007 MHz

F2 - Processing parameters
 SI 16384
 SF 400.1300150 MHz
 WDW EM
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00

139.401
 138.138
 133.669
 133.625
 131.955
 129.318
 128.459
 128.222
 128.196
 127.740
 123.564
 109.988
 90.706
 89.862
 85.665
 77.582
 77.229
 76.876
 65.005
 64.666
 51.682
 44.639
 43.029
 34.667
 31.581
 31.086
 26.565
 22.439
 21.985

Current Data Parameters
 NAME b03255c13
 EXPNO 13
 PROCNO 1
 F2 - Acquisition Parameters
 Date_ 20040503
 Time 8.42
 INSTRUM spect
 PROHD 5 mm QNP 1H
 PULPROG jmod.sb
 TD 65536
 SOLVENT CDCl3
 NS 1030
 DS 0
 SMH 21834.061 Hz
 FIDRES 0.333161 Hz
 AQ 1.5008202 sec
 RG 16384
 DE 22.900 usec
 TE 28.62 usec
 P1 300.0 K
 P1 8.00 usec
 CNST2 145.0000000
 CNST11 1.0000000
 D20 0.0068966 sec
 HL1 13 dB
 D1 5.0000000 sec
 CPDPRG waltz16
 P31 100.00 usec
 D13 0.0000040 sec
 P2 16.0 usec
 SFO1 90.5648644 MHz
 NUCLEUS 13C
 F2 - Processing parameters
 SI 65536
 SF 90.5557409 MHz
 MDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 4.00

Current Data Parameters
 NAME B03249hl
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20040223
 Time 9.42
 INSTRUM spect
 PROBHD 5 mm Multinu
 PULPROG zg
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 0
 SMH 5592.841 Hz
 FIDRES 0.170680 Hz
 AQ 2.9295092 sec
 RG 20.2
 DW 89.400 use
 DE 4.50 use
 TE 300.0 K
 D1 5.00000000 sec

===== CHANNEL f1 ===
 NUC1 1H
 P1 9.00 use
 PL1 0.00 dB
 SFO1 400.1320007 MHz

F2 - Processing parameters
 SI 16384
 SF 400.1300133 MHz
 WDW EM
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00

Current Data Parameters
 NAME b03249c13
 EXPNO 13
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20040501
 Time 10.33
 INSTRUM spect
 PROBD 5 mm QNP 1H
 PULPROG jvert
 TD 65536
 SOLVENT CDC13
 NS 340
 DS 0
 SWH 27173.912 Hz
 FIDRES 0.414641 Hz
 AQ 1.2059124 sec
 RG 9195.2
 DW 18.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 6.00000000 sec
 D11 0.03000000 sec
 D13 0.00000300 sec
 D20 0.00700000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 10.50 usec
 P2 21.00 usec
 PL1 0.00 dB
 SFO1 100.6242995 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 95.00 usec
 PL2 120.00 dB
 PL12 18.00 dB
 SFO2 400.1320007 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127659 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.00

137.293
 133.733
 131.624
 129.256
 129.137
 128.092
 127.778
 123.525
 107.677
 95.981
 82.942
 77.546
 77.228
 76.909
 64.521
 64.053
 64.032
 45.563
 44.623
 39.498
 39.381
 34.041
 29.980
 28.983
 27.334
 23.044

Current Data Parameters
 NAME b0449h1
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20040319
 Time 14.10
 INSTRUM spect
 PROBHD 5 mm Multinu
 PULPROG zg
 TD 32768
 SOLVENT CDC13
 NS 16
 DS 0
 SWH 5592.841 Hz
 FIDRES 0.170680 Hz
 AQ 2.9295092 sec
 RG 20.2
 DW 89.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 5.0000000 sec

===== CHANNEL f1 ===
 NUC1 1H
 P1 9.00 usec
 PL1 0.00 dB
 SFO1 400.1320007 MHz

F2 - Processing parameters
 SI 16384
 SF 400.1300214 MHz
 WDW EM
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00

Current Data Parameters
 NAME b0449c13
 EXPNO 13
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20040517
 Time 15.13
 INSTRUM spect
 PROHD 5 mm QNP 1H
 PULPROG jvert
 TD 65536
 SOLVENT CDCl3
 NS 565
 DS 0
 SWH 27173.912 Hz
 FIDRES 0.414641 Hz
 AQ 1.2059124 sec
 RG 9195.2
 DW 18.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 6.0000000 sec
 D11 0.0300000 sec
 D13 0.0000030 sec
 D20 0.0070000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 10.50 usec
 P2 21.00 usec
 PL1 0.00 dB
 SFO1 100.6242995 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 95.00 usec
 PL2 120.00 dB
 PL12 18.00 dB
 SFO2 400.1320007 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127542 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.00

42.999
 42.496
 35.965
 29.853
 24.505
 21.876

89.940
 86.298
 85.794
 77.549
 77.231
 76.913

138.060
 133.679
 133.517
 131.916
 129.296
 128.347
 128.076
 127.743
 123.665
 118.737

Current Data Parameters
 NAME b0459hl
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters:

Date_ 20040322
 Time 8.34
 INSTRUM spect
 PROBHD 5 mm Multinu
 PULPROG zg
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 0
 SMH 5592.841 Hz
 FIDRES 0.170680 Hz
 AQ 2.9295092 sec
 RG 10.1
 DW 89.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 5.0000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 9.00 usec
 PL1 0.00 dB
 SFO1 400.1320007 MHz

F2 - Processing parameters
 SI 16384
 SF 400.1300139 MHz
 WDW EM
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00

Handwritten notes:
 f
 f
 f

Current Data Parameters
 NAME b0459c13
 EXPNO 13
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20040322
 Time 14.15

INSTRUM spect
 PROHD 5 mm Multinu
 PULPROG jvert
 TD 65536
 SOLVENT CDCl3
 NS 233
 DS 0
 SWH 27173.912 Hz
 FIDRES 0.414641 Hz
 AQ 1.2059124 sec
 RG 11585.2
 DW 18.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 6.00000000 sec
 D11 0.03000000 sec
 D13 0.00000300 sec
 D20 0.00700000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 10.50 usec
 P2 21.00 usec
 PL1 0.00 dB
 SFO1 100.6242995 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 P1 95.00 usec
 P2 120.00 dB
 PL1 18.00 dB
 SFO2 400.1320007 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127634 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.00

41.788
 37.533
 37.478
 36.919
 29.540
 24.220
 21.733

87.868
 86.908
 86.236
 77.546
 77.229
 76.910
 67.340

137.531
 133.938
 131.847
 129.412
 128.432
 128.405
 127.656
 122.859

Current Data Parameters
 NAME b0461hl
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20040321
 Time 11.04
 INSTRUM spect
 PROBEID 5 mm Multinu
 PULPROG zg
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 0
 SWH 5592.841 Hz
 FIDRES 0.170680 Hz
 AQ 2.9295092 sec
 RG 20.2
 DW 89.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 5.0000000 sec

===== CHANNEL f1 ===
 NUC1 1H
 P1 9.00 usec
 PL1 0.00 dB
 SF01 400.1320007 MHz

F2 - Processing parameters
 SI 16384
 SF 400.1300167 MHz
 WDW EM
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00

Current Data Parameters
 NAME b0461c13
 EXPNO 13
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20040321
 Time 11.08
 INSTRUM spect
 PROBHD 5 mm Multinu
 PULPROG jvert
 TD 65536
 SOLVENT CDCl3
 NS 231
 DS 0
 SWH 27173.912 Hz
 FIDRES 0.414641 Hz
 AQ 1.2059124 sec
 RG 10321.3
 DW 18.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 6.00000000 sec
 D11 0.03000000 sec
 D13 0.00000300 sec
 D20 0.00700000 sec

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

NUC1 13C
 P1 10.50 usec
 P2 21.00 usec
 PFI 0.00 dB
 SFO1 100.6242995 MHz

===== CHANNEL f2 =====

CPDPRG2 waltz16
 NUC2 1H
 PCPD2 95.00 usec
 PL2 120.00 dB
 PL12 18.00 dB
 SFO2 400.1320007 MHz

F2 - Processing parameters

SI 32768
 SF 100.6127601 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.00

138.843
 133.843
 131.766
 129.429
 128.736
 128.340
 127.887
 123.692
 94.112
 82.038
 77.549
 77.230
 76.912
 63.739
 45.878
 42.557
 38.486
 32.639
 24.774
 23.966
 22.645
 20.432

200 180 160 140 120 100 80 60 40 20 0 -20 -40 ppm

Current Data Parameters
 NAME b0463h1
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20040322
 Time 14.46
 INSTRUM spect
 PROBRID 5 mm Multinu
 PULPROG zg
 TD 32768
 SOLVENT CDCl3
 NS 8
 DS 0
 SMH 5592.841 Hz
 FIDRES 0.170680 Hz
 AQ 2.9295092 sec
 RG 10.1
 DW 89.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 5.0000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 9.00 usec
 PL1 0.00 dB
 SFO1 400.1320007 MHz

F2 - Processing parameters
 SI 16384
 SF 400.1300242 MHz
 WDW EM
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00

Current Data Parameters
 NAME b0463c13
 EXPNO 13
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20040322
 Time 14.52
 INSTRUM spect
 PROBHD 5 mm Multinu
 PULPROG jvert
 TD 65536
 SOLVENT CDCl3
 NS 68
 DS 0
 SMH 27173.912 Hz
 FIDRES 0.414641 Hz
 AQ 1.2059124 sec
 RG 2048
 DW 18.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 6.00000000 sec
 D11 0.03000000 sec
 D13 0.00000300 sec
 D20 0.00700000 sec

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

NUC1 13C
 P1 10.50 usec
 P2 21.00 usec
 PL1 0.00 dB
 SFO1 100.6242995 MHz

===== CHANNEL f2 =====

CPDPRG2 waltz16
 NUC2 1H
 PCDP2 95.00 usec
 PL2 120.00 dB
 PL12 18.00 dB
 SFO2 400.1320007 MHz

F2 - Processing parameters

SI 32768
 SF 100.6127683 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.00

Current Data Parameters
 NAME b0469hl
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20040324
 Time 8.12
 INSTRUM spect
 PROHD 5 mm Multinu
 PULPROG zg
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 0
 SWH 5592.841 Hz
 FIDRES 0.170680 Hz
 AQ 2.9295092 sec
 RG 10.1
 DW 89.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 5.0000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 9.00 usec
 PL1 0.00 dB
 SFO1 400.1320007 MHz

F2 - Processing parameters
 SI 16384
 SF 400.1300115 MHz
 WDW EM
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00

Integrations: 1.952, 1.014, 2.043, 2.014, 2.033, 2.989, 1.000, 1.980, 2.023, 4.119, 2.051

Current Data Parameters
 NAME b0469c13
 EXPNO 13
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20040324
 Time 8.16
 INSTRUM spect
 PROBHD 5 mm Multinu
 PULPROG jvert
 TD 65536
 SOLVENT CDC13
 NS 130
 DS 0
 SWH 27173.912 Hz
 FIDRES 0.414641 Hz
 AQ 1.2059124 sec
 RG 9195.2
 DW 18.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 6.00000000 sec
 D11 0.03000000 sec
 D13 0.00000300 sec
 D20 0.00700000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 10.50 usec
 P2 21.00 usec
 PL1 0.00 dB
 SFO1 100.6242995 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 95.00 usec
 PL2 120.00 dB
 PL12 18.00 dB
 SFO2 400.1320007 MHz

F2 - Processing parameters

SI 32768
 SF 100.6127758 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.00

Current Data Parameters
 NAME b0473h1
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20040326
 Time 8.04
 INSTRUM spect
 PROBD 5 mm Multinu
 PULPROG zg
 TD 32768
 SOLVENT CDCl3
 NS 16
 DS 0
 SWH 5592.841 Hz
 FIDRES 0.170680 Hz
 AQ 2.9295092 sec
 RG 10.1
 DW 89.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 5.0000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 9.00 usec
 PL1 0.00 dB
 SFO1 400.132007 MHz

F2 - Processing parameters
 SI 16384
 SF 400.1300160 MHz
 WDW EM
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00

Current Data Parameters
 NAME b0473c13
 EXPNO 13
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20040326
 Time 8.09
 INSTRUM spect
 PROHD 5 mm Multinu
 PULPROG jvert
 TD 65536
 SOLVENT CDCl3
 NS 130
 DS 0
 SMH 27173.912 Hz
 FIDRES 0.414641 Hz
 AQ 1.2059124 sec
 RG 9195.2
 DW 18.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 6.00000000 sec
 D11 0.03000000 sec
 D13 0.00000300 sec
 D20 0.00700000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 10.50 usec
 P2 21.00 usec
 PL1 0.00 dB
 SFO1 100.6242995 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 95.00 usec
 PL2 120.00 dB
 PL12 18.00 dB
 SFO2 400.1320007 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127659 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.00