Supporting Information

Epoxysilane Rearrangement Induced by an Carbanion Generated by Conjugate Addition of Enolates of Chloroacetate and α -Chloroacetamides: Formation of Functionalized Cyclopropane Derivatives

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Table of Contents

1-(<i>tert</i> -butyldimethylsilyl)-pent-1-en-3-one (8a)	S3
Methyl 2-(2-(<i>tert</i> -Butyldimethylsilyl)-1,2-epoxy)ethyl-(<i>E</i>)-2-butenolate (11a)	S3
Reaction of 11a with lithium thiophenoxide	S4
Ethyl 2-Methylene-6-(<i>tert</i> -butyldimethylsilyl)-5,6-epoxy-(<i>Z</i>)-3-hexenoate (13)	S4
Reaction of 13 with potassium enolate of ethyl chloroacetate	S4
Reaction of 13 with potassium enolate of ethyl chloroacetate in the presence of HMPA	S5
Derivatization of 14a	S5
Reaction of 13 with lithium enolate of α -chloroacetamide derivatives	S6
Reaction of 13 with potassium enolate of ethyl bromoacetate	S7
X-ray data for bis(<i>p</i> -bromobenzoate) derivative	S9
ORTEP drawing of bis(<i>p</i> -bromobenzoate) derivative	S10
¹ H NMR of 8a	S11
¹³ C NMR of 8a	S12
¹ H NMR of 11a	S13
¹³ C NMR of 11a	S14
¹ H NMR of 12	S15
¹³ C NMR of 12	S16
¹ H NMR of 13	S17
¹³ C NMR of 13	S18
¹ H NMR of 14a	S19
¹³ C NMR of 14a	S20
¹ H NMR of 14b	S21
¹³ C NMR of 14b	S22
¹ H NMR of diol derivative	S23
¹³ C NMR of diol derivative	S24
¹ H NMR of bis(<i>p</i> -bromobenzoate) derivative	S25
¹³ C NMR of bis(<i>p</i> -bromobenzoate) derivative	S26

¹ H NMR of 15a -----	S27
¹³ C NMR of 15a -----	S28
¹ H NMR of 15b -----	S29
¹³ C NMR of 15b -----	S30
¹ H NMR of 15c -----	S31
¹³ C NMR of 15c -----	S32
¹ H NMR of 15d -----	S33
¹³ C NMR of 15d -----	S34
¹ H NMR of 15e -----	S35
¹³ C NMR of 15e -----	S36
¹ H NMR of 21a -----	S37
¹³ C NMR of 21a -----	S38
¹ H NMR of 21b -----	S39
¹³ C NMR of 21b -----	S40

General. IR spectra were recorded on an FT spectrometer. ^1H NMR spectra were taken on 500 MHz spectrometers in CDCl_3 with reference to CHCl_3 (δ 7.26) or in C_6D_6 with reference to C_6H_6 (δ 7.20). ^{13}C NMR spectra were measured with either 125 MHz spectrometers in CDCl_3 with reference to the CDCl_3 triplet (δ 77.2) or in C_6D_6 with reference to C_6H_6 triplet (δ 128.0). Resonance patterns were described as s = singlet, d = doublet, t = triplet, and m = multiplet. The assignment of ^1H and ^{13}C NMR spectra is based on H-H decoupling and HMQC experiments. Mass spectrometry was electron impact mass spectra. Liquid chromatography under medium pressures (MPLC) was carried out using prepacked columns (22 mm x 100 mm, 5 μ silica gel). For routine chromatography, the following adsorbents were used: silica gel 60 of particle size 63-210 μm for column chromatography; precoated silica gel 60 F-254 plates for analytical thin-layer chromatography. All moisture sensitive reactions were performed under a positive pressure of nitrogen. Anhydrous MgSO_4 was used for drying all organic solvent extracts in workup, and the removal of the solvents was performed with a rotary evaporator. Dry solvents and reagents were obtained by using standard procedures.

1-(*tert*-butyldimethylsilyl)-pent-1-en-3-one (8a)

To a solution of 3-(*tert*-butyldimethylsilyl)-2,3-propene-1-ol (10 g, 58 mmol) in CH_2Cl_2 (58 mL) was added MnO_2 (75 g) and the reaction mixture was refluxed for 18 h. The mixture was filtered through a pad of Celite and washed with Et_2O , and the filtrate was concentrated. The residual oil was used for the next reaction without further purification.

A solution of the above compound in THF (37 mL) was added dropwise to a cooled (ice-water) solution of EtMgBr (1.01 M in Et_2O , 71 mL, 72 mmol) in THF (71 mL) over 10 min. The cooling bath was removed, and then the reaction mixture was stirred at room temperature for 20 min before addition of 1N HCl (70 mL) at 0 $^\circ\text{C}$. The mixture was filtered through a pad of Celite. The filtrate was separated, and the aqueous phase was extracted with Et_2O (50 mL x 3). Combined organic phases were successively washed with saturated aqueous NaHCO_3 solution (70 mL) and saturated brine (70 mL), dried, and concentrated. The residual oil was used in the next step without further purification.

To a solution of the above compound in CH_2Cl_2 (50 mL) was added MnO_2 (50 g) and the mixture was refluxed for 20 h. The mixture was filtered through a pad of Celite and washed with Et_2O . The filtrate was concentrated, and the residual oil was subjected to column chromatography (silica gel 60 g, hexane:AcOEt = 15:1) to give **8a** (7.53 g, 65%). Colorless oil, R_f = 0.36 (hexane: Et_2O = 5:1). IR (film) 1680, 834 cm^{-1} . ^1H NMR (CDCl_3) δ 0.05 (6H, s, SiMe_2), 0.86 (9H, s, *t*-Bu), 1.06 (3H, t, J = 7.3 Hz, H-5), 2.59 (2H, q, J = 7.3 Hz, H-4), 6.47 (1H, dd, J = 19.2, 0.7 Hz, H-2), 7.03 (1H, d, J = 19.2 Hz, H-1). ^{13}C NMR (125 MHz, CDCl_3) δ -6.32 (SiMe_2), 8.15 (C-5), 16.7 (CMe_3), 26.5 (*t*-Bu), 32.9 (C-4), 143.5 and 144.0 (C-1 and C-2), 200.5 (C-3). HRMS calcd for $\text{C}_7\text{H}_{13}\text{OSi}$ (M^+ - C_4H_9) 141.0736, found 141.0736.

Methyl 2-(2-(*tert*-Butyldimethylsilyl)-1,2-epoxy)ethyl-(*E*)-2-butenate (11a)

To a cooled (-80 $^\circ\text{C}$) solution of **8a** (7.53 g, 37.8 mmol) and Comins' reagent (16.3 g, 41.6 mmol) in THF (120 mL) was added dropwise KHMDS (0.62 M in toluene, 67 mL, 41.5 mmol), and the solution was warmed to -30 $^\circ\text{C}$ before addition of ice-water (100 mL). The reaction mixture was extracted with Et_2O (50 mL x 3). Combined organic phases were washed with saturated brine (100 mL), dried and concentrated. The residual oil was filtered through a short pad of silica gel (silica gel, 70g, elution with hexane: AcOEt = 20:1), and the filtrate was concentrated to give the crude product.

To a cooled (ice-water) solution of MeOH (60 mL) and Et_3N (30 mL) in THF (93 mL) was added $\text{Pd}(\text{OAc})_2$ (284 mg, 1.27 mmol) and PPh_3 (660 mg, 2.53 mmol). The mixture was stirred under a CO atmosphere for 10 min before addition of the above compound in THF (20 mL), and then stirred under a balloon of CO for 5 h. The solution was poured into ice-water (100 mL) and extracted with hexane (70 mL x 3). Combined organic phases were washed successively with water (70 mL) and saturated brine (70 mL), dried, and concentrated. The residual oil was filtered through a short pad of silica gel (silica gel, 60 g, elution with hexane: AcOEt = 20:1), and the filtrate was concentrated to give crude product which was used for next reaction.

To a cooled (ice-water) solution of the above product and Na_2HPO_4 (5.2 g, 36.6 mmol) in CH_2Cl_2 (101 mL) was added mCPBA (77% purity, 8.33 g, 36.6 mmol). After cooling bath was removed, the reaction mixture was stirred

at room temperature overnight. The mixture was diluted with aqueous K_2CO_3 solution (100 mL), separated, and the aqueous phase was extracted CH_2Cl_2 (50 mL x 3). Combined organic phases were washed successively with aqueous K_2CO_3 solution (100 mL) and saturated brine (100 mL), dried, and concentrated. The residual oil was subjected to column chromatography (silica gel 150 g, elution with hexane:AcOEt = 25:1) to give the title compound (2.89 g, 38%). Colorless clear oil, R_f = 0.52 (hexane:Et₂O = 5:1). IR (film) 1723, 1252 cm^{-1} . ¹H NMR (CDCl₃) δ 0.01 (6H, s, SiMe₂), 0.98 (9H, s, *t*-Bu), 1.97 (3H, d, J = 7.3 Hz, H-4), 2.25 (1H, dd, J = 3.7, 1.0 Hz, H-2'), 3.48 (1H, dt, J = 3.7, 1.0 Hz, H-1'), 3.72 (3H, s, CO₂Me), 6.98 (1H, q, J = 7.3 Hz, H-3). ¹³C NMR (125 MHz, CDCl₃) δ -8.40 and -8.23 (SiMe₂), 14.4 (C-4), 16.7 (CMe₃), 26.5 (*t*-Bu), 50.6 (C-2'), 51.6 (C-1'), 51.9 (CO₂Me), 130.6 (C-2), 141.8 (C-3), 166.9 (C-1). HRMS calcd for C₁₃H₂₄O₃Si 256.1495, found 256.1496.

Reaction of 11a with lithium thiophenoxide.

To a cooled (-80 °C) solution of PhSH (40 μ L, 0.39 mmol) in THF (1 mL) was added dropwise *n*-BuLi (2.25M in hexane, 175 μ L, 0.39 mmol) and the solution was stirred at the same temperature for 15 min. The mixture was added to a solution of **11a** (100 mg, 0.39 mmol) in THF (0.6 mL) via cannula and the reaction mixture was warmed to 0 °C over 1 h. The solution was diluted with 10% aqueous NH₄Cl solution (10 mL), and extracted with Et₂O (15 mL x 2). Combined organic phases were washed with saturated brine (20 mL), dried and concentrated. The residual oil was subjected to column chromatography (silica gel, 7 g, elution with hexane:Et₂O = 15:1) to give **12** (58 mg, 58%).

Colorless oil, R_f = 0.59 (hexane:Et₂O = 2:1). IR (film) 1720, 1184 cm^{-1} . ¹H NMR (CDCl₃) δ 0.16 (6H, s, SiMe₂), 0.92 (9H, s, *t*-Bu), 1.82 (3H, d, J = 7.3 Hz, H-4), 3.71 (3H, s, CO₂Me), 5.75 (1H, d, J = 11.9 Hz, H-1'), 6.60 (1H, q, J = 7.3 Hz, H-3), 7.06 (1H, d, J = 11.9 Hz, H-2'). ¹³C NMR (125 MHz, CDCl₃) δ -5.17 (SiMe₂), 14.5 (C-4), 18.4 (CMe₃), 25.7 (*t*-Bu), 51.6 (CO₂Me), 105.2 (C-1'), 128.6 (C-2), 134.4 (C-3), 146.3 (C-2'), 167.7 (C-1). HRMS calcd for C₁₃H₂₄O₃Si 256.1495, found 256.1498.

found 256.1498.

Ethyl 2-Methylene-6-(*tert*-butyldimethylsilyl)-5,6-epoxy-(*Z*)-3-hexenoate (**13**)

To a cooled (-80 °C) solution of (2-ethoxycarbonyl-2-propenyl)-triphenylphosphonium bromide (3.98 g, 8.72 mmol) in THF (49 mL) was added KHMDS (0.625 M in toluene, 14 mL, 8.72 mmol) and the reaction mixture was warmed to -25 °C over 2.5 h. To a cooled (-98 °C) solution of the mixture was added a solution of (2*R**,3*R**)-3-(*tert*-butyldimethylsilyl)-2,3-epoxypropanal (1.3 g, 6.98 mmol) in THF (5 mL), and the mixture was stirred below -90 °C for 15 min.

The solution was poured into 10% aqueous NH₄Cl solution (50 mL). Phases were separated and the aqueous phase was extracted with Et₂O (30 mL x 3). Combined organic phases were washed with saturated brine (50 mL), dried, and concentrated. The residual oil was subjected to column chromatography (silica gel 40 g, elution with hexane:Et₂O = 10:1) twice to give **13** (1.04 g, 50%). Yellow oil, R_f = 0.55 (hexane:AcOEt = 5:1). IR (film) 1724, 1263 cm^{-1} . ¹H NMR (CDCl₃) δ -0.05 and -0.03 (6H, s, SiMe₂), 0.94 (9H, s, *t*-Bu), 1.29 (3H, t, J = 7.1 Hz, CO₂CH₂CH₃), 2.25 (1H, d, J = 3.6 Hz, H-6), 3.47 (1H, dd, J = 8.5, 3.6 Hz, H-5), 4.22 (2H, q, J = 7.1 Hz, CO₂CH₂CH₃), 5.35 (1H, dd, J = 11.7, 8.5 Hz, H-4), 5.68 (1H, s, H-1'), 6.37 (1H, d, J = 1.6 Hz, H-1'), 6.42 (1H, d, J = 11.7 Hz, H-3). ¹³C NMR (125 MHz, CDCl₃) δ -8.34 and -8.00 (SiMe₂), 14.2 (CO₂CH₂CH₃), 16.7 (CMe₃), 25.6 (*t*-Bu), 50.9 (C-6), 51.7 (C-5), 61.2 (CO₂CH₂CH₃), 127.4 (C-1'), 129.1 (C-3), 134.2 (C-4), 136.3 (C-2), 166.1 (C-1). HRMS calcd for C₁₅H₂₆O₃Si 282.1651, found 282.1660.

Reaction of 13 with potassium enolate of ethyl chloroacetate

To a cooled (-80 °C) solution of KHMDS (0.61 M in toluene, 4.19 mL, 2.56 mmol) in THF (32 mL) was added dropwise ethyl chloroacetate (273 μ L, 2.56 mmol) and the solution was stirred at the same temperature for 15 min, and then cooled -98 °C. To this mixture was added a solution of **13** (556 mg, 1.97 mmol) in THF (7.4 mL) and the solution was stirred at the same temperature for 15 min. The mixture was diluted with 10% aqueous NH₄Cl solution (20 mL), and extracted with Et₂O (15 mL x 3). Combined organic phases were washed with saturated brine (20 mL), dried and concentrated. The residual oil was subjected to column chromatography (silica gel, 20 g, elution with hexane: Et₂O = 5:1) to give **14** (620 mg, 85%, **14a:14b** = 1:0.2).

Reaction of **13** with potassium enolate of ethyl chloroacetate in the presence of HMPA

To a cooled (-80 °C) solution of KHMDS (0.625 M in toluene, 1.65 mL, 1.08 mmol) and HMPA (130 μ L, 1.22 mmol) in THF (15 mL) was added dropwise ethyl chloroacetate (115 μ L, 1.08 mmol) and the solution was stirred at the same temperature for 15 min. To the mixture was added a solution of **13** (278 mg, 0.984 mmol) in THF (6 mL) and the mixture was stirred at the same temperature for 15 min. The mixture was diluted with 10% aqueous NH_4Cl solution (10 mL), and extracted with Et_2O (10 mL x 3). Combined organic phases were washed with saturated brine (20 mL), dried and concentrated. The residual oil was subjected to column chromatography (silica gel, 10 g, elution with hexane: Et_2O = 5:1) to give **14** (257 mg, 71%, **14a**:**14b** = 0.7:1). Separation of **14a** and **14b** was carried out using MPLC (hexane: Et_2O = 15:2).

14a (1,2-*cis*):

Colorless oil, R_f = 0.20 (hexane: Et_2O = 5:1). IR (film) 1737, 1176 cm^{-1} . ^1H NMR (CDCl_3) δ 0.16 and 0.16 (6H, s, SiMe_2), 0.91 (9H, s, *t*-Bu), 1.20 (3H, t, J = 7.1 Hz, $\text{CO}_2\text{CH}_2\text{CH}_3$), 1.23 (1H, m, H-3), 1.25 (3H, t, J = 7.1 Hz, $\text{CO}_2\text{CH}_2\text{CH}_3$), 1.95 (1H, m, H-1), 1.96 (1H, m, H-3), 4.04-4.17 (4H, m, $\text{CO}_2\text{CH}_2\text{CH}_3$), 5.29 (1H, d, J = 10.2 Hz, H-1'), 6.01 (1H, dd, J = 11.7, 10.2 Hz, H-2'), 6.15 (1H, dd, J = 11.8, 11.7 Hz, H-3'), 6.57 (1H, d, J = 11.8 Hz, H-4').

^{13}C NMR (125 MHz, CDCl_3) δ -5.19 (SiMe_2), 14.1 and 14.3 ($\text{CO}_2\text{CH}_2\text{CH}_3$), 18.3 (CMe_3), 20.2 (C-3), 25.6 (*t*-Bu), 29.0 (C-1), 32.9 (C-2), 60.9 and 61.2 ($\text{CO}_2\text{CH}_2\text{CH}_3$), 109.5 (C-3'), 121.2 (C-1'), 131.5 (C-2'), 147.7 (C-4'), 169.8 and 170.6 ($\text{CO}_2\text{CH}_2\text{CH}_3$). HRMS calcd for $\text{C}_{19}\text{H}_{32}\text{O}_5\text{Si}$ 368.2019, found 368.2020.

14b (1,2-*trans*):

Colorless oil, R_f = 0.20 (hexane: Et_2O = 5:1). IR (film) 1726, 1173 cm^{-1} . ^1H NMR (CDCl_3) δ 0.14 and 0.15 (6H, s, SiMe_2), 0.91 (9H, s, *t*-Bu), 1.23 (6H, m, $\text{CO}_2\text{CH}_2\text{CH}_3$), 1.56 (1H, dd, J = 6.4, 4.1 Hz, H-3), 1.77 (1H, dd, J = 8.6, 4.1 Hz, H-3), 2.53 (1H, dd, J = 8.6, 4.1 Hz, H-1), 4.05-4.18 (4H, m, $\text{CO}_2\text{CH}_2\text{CH}_3$), 5.15 (1H, d, J = 11.3 Hz, H-1'), 5.83 (1H, dd, J = 11.7, 11.6 Hz, H-3'), 6.13 (1H, dd, J = 11.6, 11.3 Hz, H-2'), 6.55 (1H, d, J = 11.7 Hz, H-4'). ^{13}C NMR (125 MHz, CDCl_3) δ -5.22 (SiMe_2), 14.2 and 14.3 ($\text{CO}_2\text{CH}_2\text{CH}_3$), 18.3 (CMe_3), 21.6 (C-3), 25.6 (*t*-Bu), 29.8 (C-1), 30.2 (C-2), 60.9 and 61.6 ($\text{CO}_2\text{CH}_2\text{CH}_3$), 109.4 (C-3'), 118.0 (C-1'), 131.9 (C-2'), 147.0 (C-4'), 169.6 and 172.3 ($\text{CO}_2\text{CH}_2\text{CH}_3$). HRMS calcd for $\text{C}_{19}\text{H}_{32}\text{O}_5\text{Si}$ 368.2019, found 368.2019.

Derivatization of **14a**

To a cooled (ice-water) suspension of LiAlH_4 (82 mg, 2.17 mmol) in THF (2.2 mL) was added a solution of **14a** (260 mg, 0.706 mmol) in THF (2 mL). The reaction mixture was stirred at the same temperature for 15 min. before addition of saturated aqueous Na_2SO_4 solution (2 mL). The mixture was filtered through a pad of Celite and washed with Et_2O . The filtrate was separated, and the aqueous phase was extracted with Et_2O (5 mL x 3). Combined organic phases were washed with

saturated brine (10 mL), dried, and concentrated. The residual oil was subjected to column chromatography (silica gel 6 g, elution with hexane:AcOEt = 2:1) to give diol derivative (148 mg, 74%). Colorless oil. R_f = 0.37 (hexane:AcOEt = 1:2). IR (film) 2941, 1176 cm^{-1} . ^1H NMR (CDCl_3) δ 0.16 (6H, s, SiMe_2), 0.57 (1H, dd, J = 5.3, 4.7 Hz, H-7), 0.80 (1H, dd, J = 8.3, 4.7 Hz, H-7), 0.91 (9H, s, *t*-Bu), 1.43 (1H, m, H-6), 3.33 (1H, m, H-2'), 3.37 (1H, d, J = 11.8 Hz, H-1'), 4.00 (1H, d, J = 11.8 Hz, H-1'), 4.11 (1H, m, H-2'), 5.38 (1H, d, J = 10.8 Hz, H-4), 5.96 (1H, dd, J = 11.3, 10.8 Hz, H-3), 6.22 (1H, dd, J = 11.7, 11.3 Hz, H-2), 6.54 (1H, d, J = 11.7 Hz, H-1). ^{13}C NMR (125 MHz, CDCl_3) δ -5.17 (SiMe_2), 17.5 (C-7), 18.4 (CMe_3), 25.7 (*t*-Bu), 26.0 (C-6), 26.5 (C-5), 63.9 (C-2'), 67.0 (C-1'), 109.5 (C-2), 127.4 (C-4), 129.1 (C-3), 146.7 (C-1). HRMS calcd for $\text{C}_{15}\text{H}_{26}\text{O}_2\text{Si}$ ($\text{M}^+ - \text{H}_2\text{O}$) 266.1702, found 266.1706.

To a solution of the above diol (50 mg, 0.176 mmol), 4-*N,N*-dimethylaminopyridine (60 mg, 0.49 mmol) in CH_2Cl_2 (3.5 mL) was added 4-bromobenzoyl chloride (116 mg, 0.527 mmol) and triethylamine (245 μ L, 1.76 mmol). After being stirred at room temperature for 1.5 h, the mixture was diluted with 10% aqueous NH_4Cl solution (10 mL) and extracted with Et_2O (10 mL x 2). Combined organic phases were successively washed with water (10 mL) and saturated brine (10 mL), dried, and concentrated. The residual oil was subjected to column chromatography (silica

gel 5 g, elution with hexane:AcOEt = 5:1) to give bis(*p*-bromobenzoate) derivative (95 mg, 83%) as colorless prisms.

R_f = 0.58 (hexane:AcOEt = 2:1). mp 80.5-81.0 °C (hexane). IR (KBr) 1720, 1270 cm^{-1} . ^1H NMR (CDCl_3) δ 0.14 and 0.15 (6H, s, SiMe_2), 0.77 (1H, dd, J = 5.3, 5.1 Hz, H-7), 0.89 (9H, s, *t*-Bu), 0.97 (1H, dd, J = 8.7, 5.1 Hz, H-7), 1.67 (1H, m, H-6), 4.20 (1H, dd, J = 12.0, 10.5 Hz, H-2'), 4.27 (1H, d, J = 11.7 Hz, H-1'), 4.51 (1H, d, J = 11.7 Hz, H-1'), 4.70 (1H, dd, J = 12.0, 5.7 Hz, H-2'), 5.38 (1H, d, J = 10.6 Hz, H-4), 5.99 (1H, dd, J = 11.5, 10.6 Hz, H-3), 6.32 (1H, dd, J = 11.7, 11.5 Hz, H-2), 6.56 (1H, d, J = 11.7 Hz, H-1), 7.25-7.39 and 7.71-7.77 (each 4H, m, ArH). ^{13}C NMR (125 MHz, CDCl_3) δ -5.20 and -5.14 (SiMe_2), 16.5 (C-7), 18.4 (CMe_3), 23.5 (C-6), 24.0 (C-5), 25.7 (*t*-Bu), 65.5 (C-2'), 67.7 (C-1'), 109.3 (C-2), 125.6 (C-4), 127.9, 128.0, 129.0 and 129.1 (Ph), 129.8 (C-3), 131.1, 131.2, 131.5 and 131.6 (Ph), 146.8 (C-1), 165.7 (CO_2). HRMS calcd for $\text{C}_{29}\text{H}_{34}\text{O}_5\text{Br}_2\text{Si}$ 648.0542, found 648.0525. Anal. Calcd for $\text{C}_{29}\text{H}_{34}$: C, 53.55; H, 5.27. Found: C, 53.69; H, 5.36.

Reaction of **13** with lithium enolate of α -chloroacetamide derivatives

To a cooled (-80 °C) solution of LDA, prepared from diisopropylamine (198 μL , 1.41 mmol) and *n*-BuLi (583 μL , 1.41 mmol) in THF (3 mL), was added dropwise a solution of 2-chloro-*N,N*-diethylacetamide (217 mg, 1.41 mmol) in THF (1 mL). After being stirred at -80 °C for 20 min, the solution was added dropwise via cannula to a cooled (-80 °C) solution of **13** (265 mg, 0.938 mmol) in THF (5.4 mL). The reaction mixture was stirred at the same temperature for 15 min. The reaction mixture was diluted with 10% aqueous NH_4Cl solution (10 mL), and extracted with Et_2O (10 mL x 2). Combined organic phases were washed with saturated brine (15 mL), dried and concentrated. The residual oil was subjected to column chromatography (silica gel, 8 g, elution with hexane:AcOEt = 2:1) to give **15a** (371 mg, 100%)

15a:

Colorless oil, R_f = 0.31 (hexane:AcOEt = 2:1). IR (film) 1646, 1176 cm^{-1} . ^1H NMR (CDCl_3) δ 0.16 (6H, s, SiMe_2), 0.91 (9H, s, *t*-Bu), 1.08 (3H, t, J = 7.1 Hz, $\text{CON}(\text{CH}_2\text{CH}_3)_2$), 1.20 (3H, t, J = 7.1 Hz, $\text{CO}_2\text{CH}_2\text{CH}_3$), 1.21 (3H, t, J = 7.1 Hz, $\text{CON}(\text{CH}_2\text{CH}_3)_2$), 1.26 (1H, dd, J = 8.5, 4.6 Hz, H-3), 1.94 (1H, dd, J = 8.5, 6.8 Hz, H-1), 2.18 (1H, dd, J = 6.8, 4.6 Hz, H-3), 3.20-3.60 (4H, m, $\text{CON}(\text{CH}_2\text{CH}_3)_2$), 4.02-4.14 (2H, m, $\text{CO}_2\text{CH}_2\text{CH}_3$), 5.35 (1H, d, J = 10.1 Hz, H-1'), 6.01 (1H, dd, J = 11.4, 10.1 Hz, H-2'), 6.08 (1H, dd, J = 11.8, 11.4 Hz, H-3'), 6.56 (1H, d, J = 11.8 Hz, H-4'). ^{13}C NMR (125 MHz, CDCl_3) δ -5.24 and -5.19 (SiMe_2), 13.1 ($\text{CON}(\text{CH}_2\text{CH}_3)_2$), 14.1 and 14.5 ($\text{CO}_2\text{CH}_2\text{CH}_3$ and $\text{CON}(\text{CH}_2\text{CH}_3)_2$), 18.3 (CMe_3), 20.0 (C-3), 25.6 (*t*-Bu), 29.2 (C-1), 32.3 (C-2), 40.4 and 42.1 ($\text{CON}(\text{CH}_2\text{CH}_3)_2$), 60.9 ($\text{CO}_2\text{CH}_2\text{CH}_3$), 109.5 (C-3'), 122.2 (C-1'), 130.6 (C-2'), 147.3 (C-4'), 167.8 and 170.2 ($\text{CO}_2\text{CH}_2\text{CH}_3$ and $\text{CON}(\text{CH}_2\text{CH}_3)_2$). HRMS calcd for $\text{C}_{21}\text{H}_{37}\text{O}_4\text{NSi}$ 395.2492, found 395.2482.

15b:

Colorless oil, R_f = 0.21 (hexane:AcOEt = 2:1). IR (film) 1643, 1176 cm^{-1} . ^1H NMR (CDCl_3) δ -0.13 (6H, s, SiMe_2), 0.88 (9H, s, *t*-Bu), 0.97 (1H, d, J = 4.5 Hz, H-3), 1.04 (3H, t, J = 7.1 Hz, $\text{CON}(\text{CH}_2\text{CH}_3)_2$), 1.17 (3H, t, J = 7.1 Hz, $\text{CO}_2\text{CH}_2\text{CH}_3$), 1.18 (3H, t, J = 7.1 Hz, $\text{CON}(\text{CH}_2\text{CH}_3)_2$), 1.27 (3H, s, Me), 2.29 (1H, d, J = 4.5 Hz, H-3), 3.14-3.24 (2H, m, $\text{CON}(\text{CH}_2\text{CH}_3)_2$), 3.37-3.46 (1H, m, $\text{CON}(\text{CH}_2\text{CH}_3)_2$), 3.54-3.61 (1H, m, $\text{CON}(\text{CH}_2\text{CH}_3)_2$), 3.97-4.09 (2H, m, $\text{CON}(\text{CH}_2\text{CH}_3)_2$), 5.39 (1H, d, J = 10.7 Hz, H-1'), 5.93 (1H, dd, J = 11.9, 11.6 Hz, H-3'), 6.08 (1H, dd, J = 11.6, 10.7 Hz, H-2'), 6.54 (1H, d, J = 11.9 Hz, H-4'). ^{13}C NMR (125 MHz, CDCl_3) δ -5.22 (SiMe_2), 12.5 ($\text{CON}(\text{CH}_2\text{CH}_3)_2$), 14.0 and 14.1 ($\text{CO}_2\text{CH}_2\text{CH}_3$ and $\text{CON}(\text{CH}_2\text{CH}_3)_2$), 18.3 (CMe_3), 19.0 (Me), 25.6 (*t*-Bu), 27.8 (C-3), 33.0 and 35.3 (C-1 and C-2), 38.8 and 41.3 ($\text{CON}(\text{CH}_2\text{CH}_3)_2$), 61.0 ($\text{CO}_2\text{CH}_2\text{CH}_3$), 109.5 (C-3'), 119.5 (C-1'), 131.0 (C-2'), 147.0 (C-4'), 170.6 and 172.0 ($\text{CO}_2\text{CH}_2\text{CH}_3$ and $\text{CON}(\text{CH}_2\text{CH}_3)_2$). HRMS calcd for $\text{C}_{22}\text{H}_{39}\text{O}_4\text{NSi}$ 409.2648, found 409.2652.

15c:

Colorless oil, R_f = 0.31 (hexane:AcOEt = 2:1). IR (film) 1644, 1175 cm^{-1} . ^1H NMR (CDCl_3) δ 0.12 (6H, s, SiMe_2), 0.82 (3H, t, J = 7.1 Hz, $n\text{-Bu}$), 0.88 (9H, s, $t\text{-Bu}$), 0.96 (1H, d, J = 4.2 Hz, H-3), 1.03 (3H, t, J = 7.1 Hz, $\text{CON}(\text{CH}_2\text{CH}_3)_2$), 1.14-1.39 (12H, m, $n\text{-Bu}$, $\text{CO}_2\text{CH}_2\text{CH}_3$ and $\text{CON}(\text{CH}_2\text{CH}_3)_2$), 2.28 (1H, d, J = 4.2 Hz, H-3), 3.13-3.20 (1H, m, $\text{CON}(\text{CH}_2\text{CH}_3)_2$), 3.37-3.46 (2H, m, $\text{CON}(\text{CH}_2\text{CH}_3)_2$), 3.58-3.62 (1H, m, $\text{CON}(\text{CH}_2\text{CH}_3)_2$), 3.96-4.06 (2H, m, $\text{CO}_2\text{CH}_2\text{CH}_3$), 5.41 (1H, d, J = 10.8 Hz, H-1'), 5.90 (1H, dd, J = 11.7, 11.4 Hz, H-3'), 6.05 (1H, d, J = 11.4, 10.8 Hz, H-2'), 6.53 (1H, d, J = 11.7 Hz, H-4'). ^{13}C NMR (125 MHz, CDCl_3) δ -5.21 (SiMe_2), 12.5 ($\text{CON}(\text{CH}_2\text{CH}_3)_2$), 13.9, 14.0 and 14.1 ($\text{CO}_2\text{CH}_2\text{CH}_3$, $\text{CON}(\text{CH}_2\text{CH}_3)_2$ and $n\text{-Bu}$), 18.3 (CMe_3), 22.8 ($n\text{-Bu}$), 25.6 ($t\text{-Bu}$), 26.7 (C-3), 29.2 ($n\text{-Bu}$), 33.0 (C-1 or C-2), 38.8 ($\text{CON}(\text{CH}_2\text{CH}_3)_2$), 39.4 (C-1 or C-2), 41.3 ($\text{CON}(\text{CH}_2\text{CH}_3)_2$), 60.9 ($\text{CO}_2\text{CH}_2\text{CH}_3$), 109.3 (C-3'), 119.6 (C-1'), 130.6 (C-2'), 147.0 (C-4'), 169.2 and 171.2 ($\text{CO}_2\text{CH}_2\text{CH}_3$ and $\text{CON}(\text{CH}_2\text{CH}_3)_2$). HRMS calcd for $\text{C}_{25}\text{H}_{45}\text{O}_4\text{NSi}$ 451.3118, found 451.3116.

15d:

Colorless oil, R_f = 0.42 (hexane:AcOEt = 2:1). IR (film) 1642, 1174 cm^{-1} . ^1H NMR (CDCl_3) δ -0.14 (6H, s, SiMe_2), 0.89 (3H, t, J = 7.1 Hz, $\text{CON}(\text{CH}_2\text{CH}_3)_2$), 0.91 (9H, s, $t\text{-Bu}$), 1.02 (3H, t, J = 7.1 Hz, $\text{CON}(\text{CH}_2\text{CH}_3)_2$), 1.25 (3H, t, J = 7.1 Hz, $\text{CO}_2\text{CH}_2\text{CH}_3$), 1.88 (1H, d, J = 5.0 Hz, H-3), 2.55 (1H, d, J = 5.0 Hz, H-3), 3.20-3.33 and 3.35-3.50 (4H, m, $\text{CON}(\text{CH}_2\text{CH}_3)_2$), 4.13-4.19 (2H, m, $\text{CO}_2\text{CH}_2\text{CH}_3$), 4.82 (1H, d, J = 10.4 Hz, H-1'), 5.77 (1H, dd, J = 11.6, 10.4 Hz, H-2'), 5.88 (1H, dd, J = 11.6, 11.6 Hz, H-3'), 6.46 (1H, d, J = 11.6 Hz, H-4'), 7.22-7.36 (5H, m, Ph). ^{13}C NMR (125 MHz, CDCl_3) δ -5.22 and -5.17 (SiMe_2), 12.5 and 13.4 ($\text{CON}(\text{CH}_2\text{CH}_3)_2$), 14.2 ($\text{CO}_2\text{CH}_2\text{CH}_3$), 18.3 (CMe_3), 23.9 (C-3), 25.7 ($t\text{-Bu}$), 34.8 (C-1 or C-2), 39.5 and 41.5 ($\text{CON}(\text{CH}_2\text{CH}_3)_2$), 44.1 (C-1 or C-2), 61.4 ($\text{CO}_2\text{CH}_2\text{CH}_3$), 109.4 (C-3'), 121.0 (C-1'), 127.4, 128.4 and 128.5 (Ph), 129.6 (C-2'), 135.1 (Ph), 146.7 (C-4'), 168.6 and 172.3 ($\text{CO}_2\text{CH}_2\text{CH}_3$, $\text{CON}(\text{CH}_2\text{CH}_3)_2$). HRMS calcd for $\text{C}_{21}\text{H}_{41}\text{O}_4\text{NSi}$ 471.2805, found 471.2808.

15e:

Colorless oil, R_f = 0.48 (hexane:AcOEt = 2:1). IR (film) 1653, 1178 cm^{-1} . ^1H NMR (CDCl_3) δ 0.14 (6H, s, SiMe_2), 0.90 (9H, s, $t\text{-Bu}$), 1.06 (3H, t, J = 7.1 Hz, $\text{CON}(\text{CH}_2\text{CH}_3)_2$), 1.16-1.23 (6H, m, $\text{CO}_2\text{CH}_2\text{CH}_3$ and $\text{CON}(\text{CH}_2\text{CH}_3)_2$), 1.46 (1H, d, J = 6.4 Hz, H-3), 2.68 (1H, d, J = 6.4 Hz, H-3), 3.15-3.25 (2H, m, $\text{CON}(\text{CH}_2\text{CH}_3)_2$), 3.37-3.50 (1H, m, $\text{CON}(\text{CH}_2\text{CH}_3)_2$), 3.61-3.64 (1H, m, $\text{CON}(\text{CH}_2\text{CH}_3)_2$), 4.02-4.11 (2H, m, $\text{CO}_2\text{CH}_2\text{CH}_3$), 5.52 (1H, d, J = 10.7 Hz, H-1'), 5.83 (1H, dd, J = 11.6, 11.4 Hz, H-3'), 6.16 (1H, dd, J = 11.4, 10.7 Hz, H-2'), 6.59 (1H, d, J = 11.6 Hz, H-4'). ^{13}C NMR (125 MHz, CDCl_3) δ -5.20 (SiMe_2), 12.1 ($\text{CON}(\text{CH}_2\text{CH}_3)_2$), 13.5 and 14.1 ($\text{CO}_2\text{CH}_2\text{CH}_3$ and $\text{CON}(\text{CH}_2\text{CH}_3)_2$), 18.3 (CMe_3), 25.6 ($t\text{-Bu}$), 28.4 (C-3), 34.6 (C-2), 39.4 and 42.0 ($\text{CON}(\text{CH}_2\text{CH}_3)_2$), 50.7 (C-1), 61.7 ($\text{CO}_2\text{CH}_2\text{CH}_3$), 109.0 (C-3'), 118.6 (C-1'), 131.3 (C-2'), 147.8 (C-4'), 164.5 and 169.7 ($\text{CO}_2\text{CH}_2\text{CH}_3$, $\text{CON}(\text{CH}_2\text{CH}_3)_2$). HRMS calcd for $\text{C}_{21}\text{H}_{36}\text{O}_4\text{ClNSi}$ 429.2102, found 429.2102.

Reaction of 13 with potassium enolate of ethyl bromoacetate

To a cooled ($-80\text{ }^\circ\text{C}$) solution of KHMDS (0.59 M in toluene, 644 μL , 0.38 mmol) in THF (1.9 mL) was added dropwise ethyl bromoacetate (42 μL , 0.38 mmol) and the solution was stirred at the same temperature for 15 min. To the mixture was added a solution of **13** (80 mg, 0.28 mmol) in THF (0.9 mL) and the solution was stirred at the same temperature for 10 min. The mixture was diluted with 10% aqueous NH_4Cl solution (10 mL), and extracted with Et_2O (10 mL x 3). Combined organic phases were washed with saturated brine (10 mL), dried and concentrated. The residual oil was subjected to column chromatography (silica gel, 8 g, elution with hexane: Et_2O = 5:1) to give **21** (58.8 mg, 57%) and **14b** (12.4 mg, 12%). Separation of **21a** and **21b** was carried out using MPLC (hexane: Et_2O = 15:2).

21:

21a: Colorless oil. $R_f = 0.39$ (hexane:Et₂O = 3:1). IR (film) 1727, 1256 cm⁻¹. ¹H NMR (C₆D₆) δ -0.02 and 0.01 (6H, s, SiMe₂), 0.92 (3H, t, $J = 7.1$ Hz, CO₂CH₂CH₃), 0.93 (3H, t, $J = 7.1$ Hz, CO₂CH₂CH₃), 0.95 (9H, s, *t*-Bu), 1.69 (1H, dd, $J = 6.4, 3.9$ Hz, H-3), 1.74 (1H, dd, $J = 8.6, 3.9$ Hz, H-3), 2.19 (1H, d, $J = 3.3$ Hz, H-4'), 2.69 (1H, dd, $J = 8.6, 6.4$ Hz, H-1), 3.64 (1H, dd, $J = 8.7, 3.3$ Hz, H-3'), 3.82-4.00 (4H, m, CO₂CH₂CH₃), 5.46 (1H, dd, $J = 10.8, 8.7$ Hz, H-2'), 5.82

(1H, d, $J = 10.8$ Hz, H-1'). ¹³C NMR (125 MHz, C₆D₆) δ -8.55 and -8.30 (SiMe₂), 13.8 and 14.0 (CO₂CH₂CH₃), 16.6 (CMe₃), 21.8 (C-3), 26.4 (*t*-Bu), 29.7 (H-1), 29.8 (H-2), 50.3 (H-4'), 51.4 (H-3'), 60.5 and 61.4 (CO₂CH₂CH₃), 128.0 (C-1'), 136.9 (C-2'), 168.4 and 171.3 (CO₂CH₂CH₃). HRMS calcd for C₁₉H₃₂O₅Si 368.2019, found 368.2020.

21b: Colorless oil. $R_f = 0.39$ (hexane:Et₂O = 3:1). IR (film) 1728, 1256 cm⁻¹. ¹H NMR (C₆D₆) δ -0.03 and 0.01 (6H, s, SiMe₂), 0.89 (3H, t, $J = 7.1$ Hz, CO₂CH₂CH₃), 0.94 (9H, s, *t*-Bu), 1.01 (3H, t, $J = 7.1$ Hz, CO₂CH₂CH₃), 1.67-1.72 (2H, m, H-3), 2.19 (1H, d, $J = 3.1$ Hz, H-4'), 2.77 (1H, dd, $J = 8.5, 6.6$ Hz, H-1), 3.63 (1H, dd, $J = 8.5, 3.1$ Hz, H-3'), 3.80-4.11 (4H, m, CO₂CH₂CH₃), 5.45 (1H, dd, $J = 10.5, 8.5$ Hz, H-2'), 5.75 (1H, d, $J = 10.5$ Hz, H-1'). ¹³C NMR (125 MHz, C₆D₆) δ -8.53 and -8.20 (SiMe₂), 13.8 and 14.0 (CO₂CH₂CH₃), 16.6 (CMe₃), 21.2 (C-3), 26.4 (*t*-Bu), 29.8 (H-2), 30.0 (H-1), 50.0 (H-4'), 51.9 (H-3'), 61.0 and 61.4 (CO₂CH₂CH₃), 126.9 (C-1'), 137.5 (C-2'), 168.5 and 171.2 (CO₂CH₂CH₃). HRMS calcd for C₁₉H₃₂O₅Si 368.2019, found 356.2022.

((1*R,2*S**)-1-((1*Z*,3*E*)-4-(*tert*-butyldimethylsiloxy)buta-1,3-dienyl)cyclopropane-1,2-diyl)bis(methylene) bis(4-bromobenzoate)**

Empirical formula	C ₂₉ H ₃₄ Br ₂ O ₅ Si
Formula weight	650.47
Temperature	150 K
Wavelength	0.71073 Å
Crystal system	Monoclinic
Space group	P 21 / n
Unit cell dimensions	a = 11.4155(16) Å α = 90°. b = 20.035(3) Å β = 93.774(3)°. c = 25.895(4) Å γ = 90°.
Volume	5909.4(15) Å ³
Z	8
Density (calculated)	1.462 Mg/m ³
Absorption coefficient	2.820 mm ⁻¹
F(000)	2656
Crystal size	0.40 x 0.30 x 0.20 mm ³
Theta range for data collection	1.29 to 27.55°.
Index ranges	-14 ≤ h ≤ 10, -18 ≤ k ≤ 26, -30 ≤ l ≤ 33
Reflections collected	35442
Independent reflections	13525 [R(int) = 0.1019]
Completeness to theta = 27.55°	99.1 %
Absorption correction	Empirical
Max. and min. transmission	0.6024 and 0.3984
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	13525 / 0 / 671
Goodness-of-fit on F ²	0.927
Final R indices [I > 2σ(I)]	R1 = 0.0592, wR2 = 0.1240
R indices (all data)	R1 = 0.2047, wR2 = 0.1809
Largest diff. peak and hole	0.645 and -0.595 e.Å ⁻³

ORTEP Drawing of ((1*R**,2*S**)-1-((1*Z*,3*E*)-4-(*tert*-butyldimethylsiloxy)buta-1,3-dienyl)cyclopropane-1,2-diyl)bis(methylene) bis(4-bromobenzoate)

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X : parts per Million : 1H

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X : parts per Million : 13C

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X domain	=	1H
X offset	=	5 [ppm]
X freq	=	500.15991521 [MHz]
X sweep	=	7.50750751 [kHz]
X resolution	=	0.22911095 [Hz]
X acq duration	=	4.3646976 [s]
Digital filter	=	FALSE
Filter factor	=	1
Delay of start	=	1 [s]
Actual_start_time	=	17-JUN-2005 17:10
Acq delay	=	0.1314 [ms]
X90	=	7.4 [us]
Irr90	=	7.4 [us]
Tri90	=	10 [us]
Qua90	=	10 [us]
X90_hi	=	6 [us]
Irr90_hi	=	6 [us]
Tri90_hi	=	10 [us]
Qua90_hi	=	10 [us]
X90_lo	=	25 [us]
Irr90_lo	=	25 [us]
Tri90_lo	=	10 [us]
Qua90_lo	=	10 [us]
Spin_lock_90	=	1 [us]
Spin_lock_attn	=	29 [dB]
Deut_grad_shim_90	=	1 [us]
Deut_grad_shim_attn	=	10 [dB]
Adc card	=	16/1MHz/20
Field strength	=	11.7473579 [T]
Filter_mode	=	BUTTERWORTH
Filter_width	=	3.75375375 [kHz]
Recvr gain	=	6
Irr_code	=	192
Obs_pwidth	=	1 [us]
Obs_noise	=	WAUGH
Irr_pwidth	=	81 [us]
Irr_noise	=	WALTZ
Tri_pwidth	=	1 [us]
Tri_noise	=	WALTZ
Qua_pwidth	=	1 [us]
Qua_noise	=	WAUGH
Solvent	=	CHLOROFORM-D
Lock strength	=	1733
Lock_level	=	180
Lock_gain	=	19
Lock_osc_offset	=	70.3331 [kHz]
Lock_phase	=	240.7 [deg]
Lock_osc_state	=	2# OSC ON

----- ACQUISITION PARAMETERS -----	
File Name	= NO_IV_764B13C.3
Author	=
Sample ID	= 1
Content	= NO-IV-764B13C
Creation Date	= 17-JUN-2005 17:25
Revision Date	= 6-FEB-2006 09:51
Spec Site	= ECP500
Spec Type	= DELTA_NMR
Data Format	= 1D COMPLEX
Dimensions	= X
Dim Title	= 13C
Dim Size	= 32768
Dim Units	= [ppm]
Scans	= 250
Mod return	= 1
X_points	= 32768
X_prescans	= 4
X_domain	= 13C
X_offset	= 100[ppm]
X_freq	= 125.76529768[MHz]
X_sweep	= 31.44654088[kHz]
X_resolution	= 0.95967227[Hz]
Irr_domain	= 1H
Irr_offset	= 5[ppm]
Irr_freq	= 500.15991521[MHz]
X_acq_duration	= 1.04202224[s]
Digital_filter	= FALSE
Filter_factor	= 1
Delay_of_start	= 1[s]
Actual_start_time	= 17-JUN-2005 17:12
Acq_delay	= 27.9[us]
X90	= 23[us]
Irr90	= 7.4[us]
Tri90	= 10[us]
Qua90	= 10[us]
X90_hi	= 23.5[us]
Irr90_hi	= 6[us]
Tri90_hi	= 10[us]
Qua90_hi	= 10[us]
X90_lo	= 81[us]
Irr90_lo	= 25[us]
Tri90_lo	= 10[us]
Qua90_lo	= 10[us]
Spin_lock_90	= 1[us]
Spin_lock_attn	= 29[dB]
Deut_grad_shim_90	= 1[us]
Deut_grad_shim_attn	= 10[dB]
Adc_card	= 16/1MHz/20
Field_strength	= 11.7473579[T]
Filter_mode	= BUTTERWORTH
Filter_width	= 15.72327044[kHz]
Recvr_gain	= 20
Irr_code	= 192
Obs_pwidth	= 1[us]
Obs_noise	= WAUGH
Irr_pwidth	= 45[us]
Irr_noise	= WALTZ
Tri_pwidth	= 1[us]
Tri_noise	= WALTZ
Qua_pwidth	= 1[us]
Qua_noise	= WAUGH
Solvent	= CHLOROFORM-D
Lock_strength	= 1268
Lock_level	= 180
Lock_gain	= 19

X : parts per Million : 13C

X : parts per Million : 1H

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----- ACQUISITION PARAMETERS -----	
File Name	= NO_IV_770D.4
Author	=
Sample ID	= 1
Content	= NO-IV-770D
Creation Date	= 30-JUN-2005 18:14
Revision Date	= 6-FEB-2006 09:39
Spec Site	= ECP500
Spec Type	= DELTA NMR
Data Format	= 1D COMPLEX
Dimensions	= 1H
Dim Title	= X
Dim Size	= 32768
Dim Units	= [ppm]
Scans	= 4
Med return	= 1
X points	= 32768
X prescans	= 0
X domain	= 1H
X_offset	= 5[ppm]
X_freq	= 500.15991521[MHz]
X_sweep	= 70.50750751[kHz]
X_resolution	= 0.22911095[HHz]
X_acq duration	= 4.3646976[s]
Digital filter	= FALSE
Filter factor	= 1
Delay_of_start	= 1[s]
Actual_start_time	= 30-JUN-2005 18:13
Actual_delay	= 0.1314[ms]
X90	= 7.4[us]
Ir90	= 7.4[us]
Tr190	= 10[us]
Qua90	= 10[us]
X90 hi	= 6[us]
Ir90 hi	= 6[us]
Tr190 hi	= 10[us]
Qua90 hi	= 10[us]
X90 lo	= 25[us]
Ir90 lo	= 25[us]
Tr190 lo	= 10[us]
Qua90 lo	= 10[us]
Spin_lock_90	= 1[us]
Spin_lock_attn	= 29[db]
Deut_grad_shim_90	= 1[us]
Deut_grad_shim_attn	= 10[db]
Acq card	= 16/1MHz/20
Field strength	= 11.7473579[T]
Filterz_mode	= BUTTERWORTH
Filter_width	= 3.75375375[kHz]
Recvr_gain	= 6
Irr_code	= 192
Obs_pwidth	= 1[us]
Obs_noise	= WAUGH
Irr_pwidth	= 45[us]
Irr_noise	= WALTZ
Tri_pwidth	= 1[us]
Tri_noise	= WALTZ
Qua_pwidth	= 1[us]
Qua_noise	= WAUGH
Solvent	= CHLOROFORM-D
Lock_strength	= 1239
Lock_level	= 180
Lock_gain	= 18
Lock_osc_offset	= 70.3331[kHz]
Lock_phase	= 240.7[deg]
Lock_osc state	= 2H OSC ON

---- ACQUISITION PARAMETERS ----	
File Name	= NO_IV_770D13C.3
Author	=
Sample ID	= 1
Content	= NO-IV-770D13C
Creation Date	= 30-JUN-2005 18:26
Revision Date	= 6-FEB-2006 09:41
Spec Site	= ECP500
Spec Type	= DELTA_NMR
Data Format	= 1D COMPLEX
Dimensions	= X
Dim Title	= 13C
Dim Size	= 32768
Dim Units	= [ppm]
Scans	= 200
Mod return	= 1
X_points	= 32768
X_prescans	= 4
X_domain	= 13C
X_offset	= 100[ppm]
X_freq	= 125.76529768[MHz]
X_sweep	= 31.44654088[kHz]
X_resolution	= 0.95967227[Hz]
Irr_domain	= 1H
Irr_offset	= 5[ppm]
Irr_freq	= 500.15991521[MHz]
X_acq_duration	= 1.0420224[s]
Digital_filter	= FALSE
Filter_factor	= 1
Delay_of_start	= 1[s]
Actual_start_time	= 30-JUN-2005 18:15
Acq_delay	= 27.9[us]
X90	= 23[us]
Irr90	= 7.4[us]
Tri90	= 10[us]
Qua90	= 10[us]
X90_hi	= 23.5[us]
Irr90_hi	= 6[us]
Tri90_hi	= 10[us]
Qua90_hi	= 10[us]
X90_lo	= 81[us]
Irr90_lo	= 25[us]
Tri90_lo	= 10[us]
Qua90_lo	= 10[us]
Spin_lock_90	= 1[us]
Spin_lock_attn	= 29[dB]
Deut_grad_shim_90	= 1[us]
Deut_grad_shim_attn	= 10[dB]
Adc_card	= 16/1MHz/20
Field_strength	= 11.7473579[T]
Filter_mode	= BUTTERWORTH
Filter_width	= 15.72327044[kHz]
Recvr_gain	= 20
Irr_code	= 192
Obs_pwidth	= 1[us]
Obs_noise	= WAUGH
Irr_pwidth	= 45[us]
Irr_noise	= WALTZ
Tri_pwidth	= 1[us]
Tri_noise	= WALTZ
Qua_pwidth	= 1[us]
Qua_noise	= WAUGH
Solvent	= CHLOROFORM-D
Lock_strength	= 871
Lock_level	= 180
Lock_gain	= 18

X : parts per Million : 13C

---- ACQUISITION PARAMETERS ----	
File Name	= NO_III_606B13c.3
Author	=
Sample ID	= S#438027
Content	= Single Pulse with
Creation Date	= 17-JAN-2005 13:07
Revision Date	= 6-FEB-2006 11:16
Spec Site	= ECP500
Spec Type	= DELTA_NMR
Data Format	= 1D COMPLEX
Dimensions	= X
Dim Title	= 13C
Dim Size	= 32768
Dim Units	= [ppm]
Scans	= 1
Mod return	= 32768
X_points	= 4
X_prescans	= 13C
X_domain	= 100[ppm]
X_offset	= 125.76529768[MHz]
X_freq	= 31.44654088[kHz]
X_sweep	= 0.95967227[Hz]
X_resolution	= 1H
X_domain	= 5[ppm]
Irr_offset	= 500.15991521[MHz]
Irr_freq	= 1.04202224[s]
X_acq_duration	= FALSE
Digital_filter	= 1
Filter_factor	= 1[s]
Delay_of_start	= 17-JAN-2005 12:39
Actual_start_time	= 27.9[us]
Acq_delay	= 23[us]
X90	= 7.4[us]
Irr90	= 10[us]
Tri90	= 10[us]
Qua90	= 23.5[us]
X90_hi	= 6[us]
Irr90_hi	= 10[us]
Tri90_hi	= 10[us]
Qua90_hi	= 81[us]
X90_lo	= 25[us]
Irr90_lo	= 10[us]
Tri90_lo	= 10[us]
Qua90_lo	= 1[us]
Spin_lock_90	= 29[us]
Spin_lock_attn	= 29[us]
Deut_grad_shim_90	= 10[us]
Deut_grad_shim_attn	= 10[us]
Adc_card	= 16/1MHz/20
Field_strength	= 11.7473579[T]
Filter_mode	= BUTTERWORTH
Filter_width	= 15.72327044[kHz]
Recvr_gain	= 20
Irr_code	= 192
Obs_pwidth	= 1[us]
Obs_noise	= WAUGH
Irr_pwidth	= 45[us]
Irr_noise	= WALTZ
Tri_pwidth	= 1[us]
Tri_noise	= WALTZ
Qua_pwidth	= 1[us]
Qua_noise	= WAUGH
Solvent	= CHLOROFORM-D
Lock_strength	= 1023.0
Lock_level	= 180
Lock_gain	= 18

---- ACQUISITION PARAMETERS ----	
File Name	= NO_III_606A.4
Author	=
Sample ID	= 1
Content	= NO-III-606A
Creation Date	= 17-JAN-2005 13:54
Revision Date	= 6-FEB-2006 11:20
Spec Site	= ECP500
Spec Type	= DELTA_NMR
Data Format	= 1D COMPLEX
Dimensions	= X
Dim Title	= 1H
Dim Size	= 32768
Dim Units	= [ppm]
Scans	= 4
Mod return	= 1
X_points	= 32768
X_prescans	= 0
X_domain	= 1H
X_offset	= 5[ppm]
X_freq	= 500.15991521[MHz]
X_sweep	= 7.50750751[kHz]
X_resolution	= 0.22911095[Hz]
Digital filter	= 4.3646976[s]
Filter factor	= FALSE
Delay of start	= 1
Actual_start_time	= 1[s]
Acq_delay	= 17-JAN-2005 13:53
X90	= 0.1314[ms]
Irr90	= 7.4[us]
Tri90	= 10[us]
Qua90	= 10[us]
X90_hi	= 6[us]
Irr90_hi	= 6[us]
Tri90_hi	= 10[us]
Qua90_hi	= 10[us]
X90_lo	= 25[us]
Irr90_lo	= 25[us]
Tri90_lo	= 10[us]
Qua90_lo	= 10[us]
Spin_lock_90	= 1[us]
Spin_lock_attn	= 29[dB]
Deut_grad_shim_90	= 1[us]
Deut_grad_shim_attn	= 10[dB]
Adc card	= 16/1MHz/20
Field strength	= 11.7473579[T]
Filter_mode	= BUTTERWORTH
Filter_width	= 3.75375375[kHz]
Recvr_gain	= 13
Irr_code	= 192
Obs_pwidth	= 1[us]
Obs_noise	= WAUGH
Irr_pwidth	= 81[us]
Irr_noise	= WALTZ
Tri_pwidth	= 1[us]
Tri_noise	= WALTZ
Qua_pwidth	= 1[us]
Qua_noise	= WAUGH
Solvent	= CHLOROFORM-D
Lock_strength	= 1485
Lock_level	= 180
Lock_gain	= 18
Lock_osc_offset	= 70.3331[kHz]
Lock_phase	= 240.7[deg]
Lock_osc_state	= 2H OSC ON

---- ACQUISITION PARAMETERS ----	
File Name	= NO_III_606A13C.3
Author	=
Sample ID	= S#483531
Content	= Single Pulse with
Creation Date	= 17-JAN-2005 14:23
Revision Date	= 6-FEB-2006 11:23
Spec Site	= ECP500
Spec Type	= DELTA_NMR
Data Format	= 1D COMPLEX
Dimensions	= X
Dim Title	= 13C
Dim Size	= 32768
Dim Units	= [ppm]
Scans	= 1
Mod return	= 32768
X_points	= 4
X_prescans	= 13C
X_domain	= 100[ppm]
X_offset	= 125.76529768[MHz]
X_freq	= 31.44654088[kHz]
X_sweep	= 0.95967227[Hz]
X_resolution	= 1H
X_domain	= 5[ppm]
Irr_offset	= 500.15991521[MHz]
Irr_freq	= 1.04202224[s]
X_acq_duration	= FALSE
Digital_filter	= 1
Filter_factor	= 1[s]
Delay_of_start	= 17-JAN-2005 13:55
Actual_start_time	= 27.9[us]
Acq_delay	= 23[us]
X90	= 7.4[us]
Irr90	= 10[us]
Tri90	= 10[us]
Qua90	= 23.5[us]
X90_hi	= 6[us]
Irr90_hi	= 10[us]
Tri90_hi	= 10[us]
Qua90_hi	= 81[us]
X90_lo	= 25[us]
Irr90_lo	= 10[us]
Tri90_lo	= 10[us]
Qua90_lo	= 1[us]
Spin_lock_90	= 29[dB]
Spin_lock_attn	= 1[us]
Deut_grad_shim_90	= 10[dB]
Deut_grad_shim_attn	= 16/1MHz/20
Adc_card	= 11.7473579[T]
Field_strength	= BUTTERWORTH
Filter_mode	= 15.72327044[kHz]
Filter_width	= 20
Recvr_gain	= 192
Irr_code	= 1[us]
Obs_pwidth	= WAUGH
Obs_noise	= 45[us]
Irr_pwidth	= WALTZ
Irr_noise	= 1[us]
Tri_pwidth	= WALTZ
Tri_noise	= 1[us]
Qua_pwidth	= WAUGH
Qua_noise	= CHLOROFORM-D
Solvent	= 1142
Lock_strength	= 180
Lock_level	= 18
Lock_gain	=

----- ACQUISITION PARAMETERS -----	
File Name	= NO_IV_763B.3
Author	= 1
Sample ID	= NO-IV-763B
Content	= 16-JUN-2005 18:19
Creation Date	
Revision Date	= 22-FEB-2006 15:17
Spec Site	= ECP500
Spec Type	= DELTA NMR
Data Format	= 1H COMPLEX
Dimensions	= X
Dim Title	= 1H
Dim Size	= 32768
Dim Units	= [ppm]
Scans	= 4
Mod return	= 1
X points	= 32768
X prescans	= 0
X domain	= 1H
X offset	= 5[ppm]
X freq	= 500.15991521[MHz]
X sweep	= 7.50750751[kHz]
X resolution	= 0.22911095[Hz]
X acq duration	= 4.3646976[s]
Digital filter	= FALSE
Filter factor	= 1
Delay_of_start	= 1[s]
Actual_start_time	= 16-JUN-2005 18:18
Acq delay	= 0.1314[ms]
X90	= 7.4[us]
Irr90	= 7.4[us]
Tri90	= 10[us]
Qua90	= 10[us]
X90 hi	= 6[us]
Irr90 hi	= 6[us]
Tri90 hi	= 10[us]
Qua90 hi	= 10[us]
X90 lo	= 25[us]
Irr90 lo	= 25[us]
Tri90 lo	= 10[us]
Qua90 lo	= 10[us]
Spin_lock_90	= 1[us]
Spin_lock_attn	= 29[dB]
Deut_grad_shim_90	= 1[us]
Deut_grad_shim_attn	= 10[dB]
Adc card	= 16/1MHz/20
Field strength	= 11.7473579[T]
Filter_mode	= BUTTERWORTH
Filter_width	= 3.75375375[kHz]
Recvr gain	= 11
Irr code	= 192
Obs pwidth	= 1[us]
Obs noise	= WAUGH
Irr_pwidth	= 45[us]
Irr_noise	= WALTZ
Tri_pwidth	= 1[us]
Tri_noise	= WALTZ
Qua pwidth	= 1[us]
Qua noise	= WAUGH
Solvent	= CHLOROFORM-D
Lock strength	= 1219
Lock_level	= 180
Lock gain	= 18
Lock osc_offset	= 70.3331[kHz]
Lock phase	= 240.7[deg]
Lock osc state	= 2H OSC ON

---- ACQUISITION PARAMETERS ----	
File Name	= NO_IV_763B13C.2
Author	=
Sample ID	= 1
Content	= NO-IV-763B13C
Creation Date	= 16-JUN-2005 18:46
Revision Date	= 22-FEB-2006 15:19
Spec Site	= ECP500
Spec Type	= DELTA_NMR
Data Format	= 1D COMPLEX
Dimensions	= X
Dim Title	= 13C
Dim Size	= 32768
Dim Units	= [ppm]
Scans	= 500
Mod return	= 1
X_points	= 32768
X_prescans	= 4
X_domain	= 13C
X_offset	= 100[ppm]
X_freq	= 125.76529768[MHz]
X_sweep	= 31.44654088[kHz]
X_resolution	= 0.95967227[Hz]
Irr_domain	= 1H
Irr_offset	= 5[ppm]
Irr_freq	= 500.15991521[MHz]
X_acq_duration	= 1.04202224[s]
Digital_filter	= FALSE
Filter_factor	= 1
Delay_of_start	= 1[s]
Actual_start_time	= 16-JUN-2005 18:20
Acq_delay	= 27.9[us]
X90	= 23[us]
Irr90	= 7.4[us]
Tri90	= 10[us]
Qua90	= 10[us]
X90_hi	= 23.5[us]
Irr90_hi	= 6[us]
Tri90_hi	= 10[us]
Qua90_hi	= 10[us]
X90_lo	= 81[us]
Irr90_lo	= 25[us]
Tri90_lo	= 10[us]
Qua90_lo	= 10[us]
Spin_lock_90	= 1[us]
Spin_lock_attn	= 29[dB]
Deut_grad_shim_90	= 1[us]
Deut_grad_shim_attn	= 10[dB]
Adc_card	= 16/1MHz/20
Field_strength	= 11.7473579[T]
Filter_mode	= BUTTERWORTH
Filter_width	= 15.72327044[kHz]
Recvr_gain	= 20
Irr_code	= 192
Obs_pwidth	= 1[us]
Obs_noise	= WAUGH
Irr_pwidth	= 45[us]
Irr_noise	= WALTZ
Tri_pwidth	= 1[us]
Tri_noise	= WALTZ
Qua_pwidth	= 1[us]
Qua_noise	= WAUGH
Solvent	= CHLOROFORM-D
Lock_strength	= 1081
Lock_level	= 180
Lock_gain	= 18

X : parts per Million : 13C

----- ACQUISITION PARAMETERS -----	
File Name	= NO_IV_773B.4
Author	=
Sample ID	= 1
Content	= NO-IV-773B
Creation Date	= 15-JUL-2005 09:19
Revision Date	= 6-FEB-2006 09:32
Spec Site	= ECP500
Spec Type	= DELTA_NMR
Data Format	= 1D COMPLEX
Dimensions	= X
Dim Title	= 1H
Dim Size	= 32768
Dim Units	= [ppm]
Scans	= 4
Mod return	= 1
X_points	= 32768
X_prescans	= 0
X_domain	= 1H
X_offset	= 5[ppm]
X_freq	= 500.15991521[MHz]
X_sweep	= 7.50750751[kHz]
X_resolution	= 0.22911095[Hz]
Digital filter	= 4.3646976[s]
Filter factor	= FALSE
Delay of start	= 1
Actual_start_time	= 1[s]
Acq_delay	= 15-JUL-2005 09:18
X90	= 0.1314[ms]
Irr90	= 7.4[us]
Tri90	= 10[us]
Qua90	= 10[us]
X90_hi	= 6[us]
Irr90_hi	= 6[us]
Tri90_hi	= 10[us]
Qua90_hi	= 10[us]
X90_lo	= 25[us]
Irr90_lo	= 25[us]
Tri90_lo	= 10[us]
Qua90_lo	= 10[us]
Spin_lock_90	= 1[us]
Spin_lock_attn	= 29[dB]
Deut_grad_shim_90	= 1[us]
Deut_grad_shim_attn	= 10[dB]
Adc card	= 16/1MHz/20
Field strength	= 11.7473579[T]
Filter_mode	= BUTTERWORTH
Filter_width	= 3.75375375[kHz]
Recvr_gain	= 10
Irr_code	= 192
Obs_pwidth	= 1[us]
Obs_noise	= WAUGH
Irr_pwidth	= 45[us]
Irr_noise	= WALTZ
Tri_pwidth	= 1[us]
Tri_noise	= WALTZ
Qua_pwidth	= 1[us]
Qua_noise	= WAUGH
Solvent	= CHLOROFORM-D
Lock_strength	= 1978
Lock_level	= 180
Lock_gain	= 20
Lock_osc_offset	= 70.3331[kHz]
Lock_phase	= 240.7[deg]
Lock_osc_state	= 2H OSC ON

X : parts per Million : 1H

0 1.0 2.0 3.0 4.0 5.0 6.0 7.0 8.0

(Millions)

---- ACQUISITION PARAMETERS ----	
File Name	= NO_IV_773B13C.4
Author	=
Sample ID	= 1
Content	= NO-IV-773B13C
Creation Date	= 15-JUL-2005 09:46
Revision Date	= 6-FEB-2006 09:35
Spec Site	= ECP500
Spec Type	= DELTA_NMR
Data Format	= 1D COMPLEX
Dimensions	= X
Dim Title	= 13C
Dim Size	= 32768
Dim Units	= [ppm]
Scans	= 500
Mod return	= 1
X_points	= 32768
X_prescans	= 4
X_domain	= 13C
X_offset	= 100[ppm]
X_freq	= 125.76529768[MHz]
X_sweep	= 31.44654088[kHz]
X_resolution	= 0.95967227[Hz]
X_domain	= 1H
Irr_offset	= 5[ppm]
Irr_freq	= 500.15991521[MHz]
X_acq_duration	= 1.0420224[s]
Digital_filter	= FALSE
Filter_factor	= 1
Delay_of_start	= 1[s]
Actual_start_time	= 15-JUL-2005 09:20
Acq_delay	= 27.9[us]
X90	= 23[us]
Irr90	= 7.4[us]
Tri90	= 10[us]
Qua90	= 10[us]
X90_hi	= 23.5[us]
Irr90_hi	= 6[us]
Tri90_hi	= 10[us]
Qua90_hi	= 10[us]
X90_lo	= 81[us]
Irr90_lo	= 25[us]
Tri90_lo	= 10[us]
Qua90_lo	= 10[us]
Spin_lock_90	= 1[us]
Spin_lock_attn	= 29[dB]
Deut_grad_shim_90	= 1[us]
Deut_grad_shim_attn	= 10[dB]
Adc_card	= 16/1MHz/20
Field_strength	= 11.7473579[T]
Filter_mode	= BUTTERWORTH
Filter_width	= 15.72327044[kHz]
Recvr_gain	= 20
Irr_code	= 192
Obs_pwidth	= 1[us]
Obs_noise	= WAUGH
Irr_pwidth	= 45[us]
Irr_noise	= WALTZ
Tri_pwidth	= 1[us]
Tri_noise	= WALTZ
Qua_pwidth	= 1[us]
Qua_noise	= WAUGH
Solvent	= CHLOROFORM-D
Lock_strength	= 1571
Lock_level	= 180
Lock_gain	= 20

```

---- ACQUISITION PARAMETERS ----
File Name      = NO_V_863B13C.5
Author         = 
Sample ID      = 1
Content        = NO-V-863B13C
Creation Date   = 19-OCT-2005 09:45
Revision Date  = 6-FEB-2006 08:56
Spec Site      = ECP500

Spec Type      = DELTA_NMR
Data Format     = 1D COMPLEX
Dimensions     = X
Dim Title      = 13C
Dim Size       = 32768
Dim Units      = [ppm]
Scans          = 300
Mod return     = 1
X_points       = 32768
X_prescans     = 4
X_domain       = 13C
X_offset       = 100[ppm]
X_freq         = 125.76529768[MHz]
X_sweep        = 31.44654088[kHz]
X_resolution   = 0.95967227[Hz]
Irr_domain     = 1H
Irr_offset     = 5[ppm]
Irr_freq       = 500.15991521[MHz]
X_acq_duration = 1.04202224[s]
Digital_filter = FALSE
Filter_factor   = 1
Delay_of_start  = 1[s]
Actual_start_time = 19-OCT-2005 09:30
Acq_delay      = 27.9[us]
X90            = 23[us]
Irr90          = 7.4[us]
Tri90          = 10[us]
X90_hi         = 23[us]
Irr90_hi       = 6[us]
Tri90_hi       = 10[us]
Qua90_hi       = 10[us]
X90_lo         = 0.165[ms]
Irr90_lo       = 25[us]
Tri90_lo       = 10[us]
Qua90_lo       = 10[us]
Spin_lock_90   = 1[us]
Spin_lock_attn = 29[dB]
Deut_grad_shim_90 = 1[us]
Deut_grad_shim_attn = 10[dB]
Adc_card       = 16/1MHz/20
Field_strength = 11.7473579[T]
Filter_mode     = BUTTERWORTH
Filter_width    = 15.72327044[kHz]
Recvr_gain     = 20
Irr_code       = 192
Obs_pwidth     = 1[us]
Obs_noise      = WAUGH
Irr_pwidth     = 45[us]
Irr_noise      = WALTZ
Tri_pwidth     = 1[us]
Tri_noise      = WALTZ
Qua_pwidth     = 1[us]
Qua_noise      = WAUGH
Solvent        = CHLOROFORM-D
Lock_strength  = 799
Lock_level     = 180
Lock_gain      = 18
  
```

X : parts per Million : ¹³C

----- ACQUISITION PARAMETERS -----
File Name = NO_V 885A.4
Author = 1
Sample ID = NO V 885A
Content = 9-NOV-2005 11:08
Creation Date
Revision Date = 6-FEB-2006 08:37
Spec Site = ECP500
Spec Type = DELTA_NMR
Data Format = 1D COMPLEX
Dimensions = X
Dim Title = 1H
Dim Size = 32768
Dim Units = [ppm]
Scans = 4
Mod return = 1
X_points = 32768
X_prescans = 0
X_domain = 1H
X_offset = 5[ppm]
X_freq = 500.15991521[MHz]
X_sweep = 7.50750751[kHz]
X_resolution = 0.22911095[Hz]
Digital filter = 4.3646976[s]
Filter factor = FALSE
Delay of start = 1
Actual_start_time = 1[s]
Acq_delay = 9-NOV-2005 11:07
X90 = 0.1314[ms]
Irr90 = 7.4[us]
Tri90 = 10[us]
Qua90 = 10[us]
X90_hi = 6[us]
Irr90_hi = 6[us]
Tri90_hi = 10[us]
Qua90_hi = 10[us]
X90_lo = 25[us]
Irr90_lo = 25[us]
Tri90_lo = 10[us]
Qua90_lo = 10[us]
Spin_lock_90 = 1[us]
Spin_lock_attn = 29[dB]
Deut_grad_shim_90 = 1[us]
Deut_grad_shim_attn = 10[dB]
Adc card = 16/1MHz/20
Field strength = 11.7473579[T]
Filter_mode = BUTTERWORTH
Filter_width = 3.75375375[kHz]
Recvr_gain = 8
Irr_code = 192
Obs_pwidth = 1[us]
Irr_pwidth = 0.165[ms]
Tri_pwidth = 1[us]
Qua_pwidth = 1[us]
Solvant = WAUGH
Lock_strength = 1714
Lock_level = 180
Lock_gain = 19
Lock_osc_offset = 70.3331[kHz]
Lock_phase = 240.7[deg]
Lock_osc_state = 2H OSC ON

X : parts per Million : 1H

0

1.0

2.0

3.0

4.0

5.0

6.0

Millions

0

30.0

28.0

26.0

24.0

22.0

20.0

18.0

16.0

14.0

12.0

10.0

8.0

6.0

4.0

2.0

0

30.0

28.0

26.0

24.0

22.0

20.0

18.0

16.0

14.0

12.0

10.0

8.0

6.0

4.0

2.0

0

30.0

28.0

26.0

24.0

22.0

20.0

18.0

16.0

14.0

12.0

10.0

8.0

6.0

4.0

2.0

0

30.0

28.0

26.0

24.0

22.0

20.0

18.0

16.0

14.0

12.0

10.0

8.0

6.0

4.0

2.0

0

30.0

28.0

26.0

24.0

22.0

20.0

18.0

16.0

14.0

12.0

10.0

8.0

6.0

4.0

2.0

0

30.0

28.0

26.0

24.0

22.0

20.0

18.0

16.0

14.0

12.0

10.0

8.0

6.0

4.0

2.0

0

30.0

28.0

26.0

24.0

22.0

20.0

18.0

16.0

14.0

12.0

10.0

8.0

6.0

4.0

2.0

0

30.0

28.0

26.0

24.0

22.0

20.0

18.0

16.0

14.0

12.0

10.0

8.0

6.0

4.0

2.0

0

30.0

28.0

26.0

24.0

22.0

20.0

18.0

16.0

14.0

12.0

10.0

8.0

6.0

4.0

2.0

0

30.0

28.0

26.0

24.0

22.0

20.0

18.0

16.0

14.0

12.0

10.0

8.0

6.0

4.0

2.0

0

30.0

28.0

26.0

24.0

22.0

20.0

18.0

16.0

14.0

12.0

10.0

8.0

6.0

4.0

2.0

0

30.0

28.0

26.0

24.0

22.0

20.0

18.0

16.0

14.0

12.0

10.0

8.0

6.0

4.0

2.0

0

30.0

28.0

26.0

24.0

22.0

20.0

18.0

16.0

14.0

12.0

10.0

8.0

6.0

4.0

2.0

0

30.0

28.0

26.0

24.0

22.0

20.0

18.0

16.0

14.0

12.0

10.0

8.0

6.0

4.0

2.0

0

30.0

28.0

26.0

24.0

22.0

20.0

18.0

16.0

14.0

12.0

10.0

8.0

6.0

4.0

2.0

0

30.0

28.0

26.0

24.0

22.0

20.0

18.0

16.0

14.0

12.0

10.0

8.0

6.0

4.0

2.0

0

30.0

28.0

26.0

24.0

22.0

20.0

18.0

16.0

14.0

12.0

10.0

8.0

6.0

4.0

2.0

0

30.0

28.0

26.0

24.0

22.0

20.0

18.0

16.0

14.0

12.0

10.0

8.0

6.0

4.0

2.0

0

30.0

28.0

26.0

```

---- ACQUISITION PARAMETERS ----
File Name      = NO_V 885A13C.3
Author         = 
Sample ID      = 1
Content        = NO-V-885A13C
Creation Date   = 9-NOV-2005 11:25
Revision Date  = 6-FEB-2006 08:40
Spec Site      = ECP500

Spec Type      = DELTA_NMR
Data Format     = 1D COMPLEX
Dimensions     = X
Dim Title      = 13C
Dim Size       = 32768
Dim Units      = [ppm]
Scans          = 300
Mod return     = 1
X_points       = 32768
X_prescans     = 4
X_domain       = 13C
X_offset       = 100[ppm]
X_freq         = 125.76529768[MHz]
X_sweep        = 31.44654088[kHz]
X_resolution   = 0.95967227[Hz]
Irr_domain     = 1H
Irr_offset     = 5[ppm]
Irr_freq       = 500.15991521[MHz]
X_acq_duration = 1.0420224[s]
Digital_filter = FALSE
Filter_factor   = 1
Delay_of_start  = 1[s]
Actual_start_time = 9-NOV-2005 11:09
Acq_delay       = 27.9[us]
X90             = 23[us]
Irr90           = 7.4[us]
Tri90           = 10[us]
Qua90           = 10[us]
X90_hi          = 23[us]
Irr90_hi        = 6[us]
Tri90_hi        = 10[us]
Qua90_hi        = 10[us]
X90_lo          = 0.165[ms]
Irr90_lo        = 25[us]
Tri90_lo        = 10[us]
Qua90_lo        = 10[us]
Spin_lock_90    = 1[us]
Spin_lock_attn  = 29[dB]
Deut_grad_shim_90 = 1[us]
Deut_grad_shim_attn = 10[dB]
Adc_card        = 16/1MHz/20
Field_strength  = 11.7473579[T]
Filter_mode     = BUTTERWORTH
Filter_width    = 15.72327044[kHz]
Recvr_gain      = 20
Irr_code        = 192
Obs_pwidth      = 1[us]
Obs_noise       = WAUGH
Irr_pwidth      = 45[us]
Irr_noise       = WALTZ
Tri_pwidth      = 1[us]
Tri_noise       = WALTZ
Qua_pwidth      = 1[us]
Qua_noise       = WAUGH
Solvent         = CHLOROFORM-D
Lock_strength    = 1278
Lock_level       = 180
Lock_gain        = 19
  
```

---- ACQUISITION PARAMETERS ----	
File Name	= NO_V_912A13C.3
Author	=
Sample ID	= 1
Content	= NO-V-912A13C
Creation Date	= 29-NOV-2005 11:52
Revision Date	= 6-FEB-2006 08:26
Spec Site	= ECP500
Spec Type	= DELTA_NMR
Data Format	= 1D COMPLEX
Dimensions	= X
Dim Title	= 13C
Dim Size	= 32768
Dim Units	= [ppm]
Scans	= 500
Mod return	= 1
X_points	= 32768
X_prescans	= 4
X_domain	= 13C
X_offset	= 100[ppm]
X_freq	= 125.76529768[MHz]
X_sweep	= 31.44654088[kHz]
X_resolution	= 0.95967227[Hz]
Irr_domain	= 1H
Irr_offset	= 5[ppm]
Irr_freq	= 500.15991521[MHz]
X_acq_duration	= 1.0420224[s]
Digital_filter	= FALSE
Filter_factor	= 1
Delay_of_start	= 1[s]
Actual_start_time	= 29-NOV-2005 11:26
Acq_delay	= 27.9[us]
X90	= 23[us]
Irr90	= 7.4[us]
Tri90	= 10[us]
Qua90	= 10[us]
X90_hi	= 23[us]
Irr90_hi	= 6[us]
Tri90_hi	= 10[us]
Qua90_hi	= 10[us]
X90_lo	= 0.165[ms]
Irr90_lo	= 25[us]
Tri90_lo	= 10[us]
Qua90_lo	= 10[us]
Spin_lock_90	= 1[us]
Spin_lock_attn	= 29[dB]
Deut_grad_shim_90	= 1[us]
Deut_grad_shim_attn	= 10[dB]
Adc_card	= 16/1MHz/20
Field_strength	= 11.7473579[T]
Filter_mode	= BUTTERWORTH
Filter_width	= 15.72327044[kHz]
Recvr_gain	= 20
Irr_code	= 192
Obs_pwidth	= 1[us]
Obs_noise	= WAUGH
Irr_pwidth	= 45[us]
Irr_noise	= WALTZ
Tri_pwidth	= 1[us]
Tri_noise	= WALTZ
Qua_pwidth	= 1[us]
Qua_noise	= WAUGH
Solvent	= CHLOROFORM-D
Lock_strength	= 944
Lock_level	= 180
Lock_gain	= 18

<div><div>JEOL</div><div><div>E</div><div>3</div></div></div>	
----- ACQUISITION PARAMETERS -----	
File Name	= NO_V_880A.4
Author	=
Sample ID	= 1
Content	= NO-V-880A
Creation Date	= 4-NOV-2005 11:56
Revision Date	= 6-FEB-2006 08:44
Spec Site	= ECP500
Spec Type	= DELTA_NMR
Data Format	= 1D COMPLEX
Dimensions	= X
Dim Title	= 1H
Dim Size	= 32768
Dim Units	= [Ppm]
Scans	= 4
Mod return	= 1
X_points	= 32768
X_prescans	= 0
X_domain	= 1H
X_offset	= 5[ppm]
X_freq	= 500.15991521[MHz]
X_sweep	= 7.50750751[kHz]
X_resolution	= 0.22911095[Hz]
Digital filter	= 4.3646976[s]
Filter factor	= FALSE
Delay of start	= 1
Actual_start_time	= 1[s]
Acq_delay	= 4-NOV-2005 11:55
X90	= 0.1314[ms]
Irr90	= 7.4[us]
Tri90	= 10[us]
Qua90	= 10[us]
X90_hi	= 10[us]
Irr90_hi	= 6[us]
Tri90_hi	= 10[us]
Qua90_hi	= 10[us]
X90_lo	= 10[us]
Irr90_lo	= 25[us]
Tri90_lo	= 50[us]
Qua90_lo	= 10[us]
Spin_lock_90	= 1[us]
Spin_lock_attn	= 29[us]
Deut_grad_shim_90	= 1[us]
Deut_grad_shim_attn	= 10[us]
Adc card	= 16/1MHz/20
Field strength	= 11.7473579[T]
Filter_mode	= BUTTERWORTH
Filter_width	= 3.75375375[kHz]
Recvr_gain	= 12
Irr_code	= 192
Obs_pwidth	= 1[us]
Obs_noise	= WAUGH
Irr_pwidth	= 45[us]
Irr_noise	= WALTZ
Tri_pwidth	= 1[us]
Tri_noise	= WALTZ
Qua_pwidth	= 1[us]
Qua_noise	= WAUGH
Solvent	= CHLOROFORM-D
Lock_strength	= 1380
Lock_level	= 180
Lock_gain	= 18
Lock_osc_offset	= 70.3331[kHz]
Lock_phase	= 240.7[deg]
Lock_osc_state	= 2H OSC ON

----- ACQUISITION PARAMETERS -----
File Name = NO_V_880A13C.3
Author =
Sample ID = 1
Content = NO-V-880A13C
Creation Date = 4-NOV-2005 15:50
Revision Date = 6-FEB-2006 08:47
Spec Site = ECP500

Spec Type = DELTA_NMR
Data Format = 1D COMPLEX
Dimensions = X
Dim Title = 13C
Dim Size = 32768
Dim Units = [ppm]
Scans = 250
Mod return = 1
X_points = 32768
X_prescans = 4
X_domain = 13C
X_offset = 100[ppm]
X_freq = 125.76529768[MHz]
X_sweep = 31.44654088[kHz]
X_resolution = 0.95967227[Hz]
X_domain = 1H
Irr_domain = 5[ppm]
Irr_offset = 500.15991521[MHz]
Irr_freq = 1.04202224[s]
X_acq_duration = FALSE
Digital_filter = 1
Filter_factor = 1
Delay_of_start = 1[s]
Actual_start_time = 4-NOV-2005 15:37
Acq_delay = 27.9[us]
X90 = 23[us]
Irr90 = 7.4[us]
Tri90 = 10[us]
Qua90 = 23[us]
X90_hi = 6[us]
Irr90_hi = 10[us]
Tri90_lo = 0.165[ms]
Irr90_lo = 25[us]
Tri90_lo = 10[us]
Qua90_lo = 10[us]
Spin_lock_90 = 1[us]
Spin_lock_attn = 29[dB]
Deut_grad_shim_90 = 1[us]
Deut_grad_shim_attn = 10[dB]
Adc_card = 16/1MHz/20
Field_strength = 11.7473579[T]
Filter_mode = BUTTERWORTH
Filter_width = 15.72327044[kHz]
Recvr_gain = 20
Irr_code = 192
Obs_pwidth = 1[us]
Obs_noise = WAUGH
Irr_pwidth = 45[us]
Irr_noise = WALTZ
Tri_pwidth = 1[us]
Tri_noise = WALTZ
Qua_pwidth = 1[us]
Qua_noise = WAUGH

Lock_strength = CHLOROFORM-D
Lock_level = 875
Lock_gain = 180
Lock_level = 18

```

---- ACQUISITION PARAMETERS ----
File Name      = NO_V_909A13C.3
Author         =
Sample ID      = 1
Content        = NO-V-909A13C
Creation Date   = 26-NOV-2005 12:03

Revision Date  = 6-FEB-2006 08:32
Spec Site      = ECP500

Spec Type      = DELTA_NMR
Data Format     = 1D COMPLEX
Dimensions     = X
Dim Title      = 13C
Dim Size       = 32768
Dim Units      = [ppm]
Scans          = 500
Mod return     = 1
X_points       = 32768
X_prescans     = 4
X_domain       = 13C
X_offset       = 100[ppm]
X_freq         = 125.76529768[MHz]
X_sweep        = 31.44654088[kHz]
X_resolution   = 0.95967227[Hz]
X_domain       = 1H
Irr_offset     = 5[ppm]
Irr_freq       = 500.15991521[MHz]
X_acq_duration = 1.04202224[s]
Digital_filter = FALSE
Filter_factor   = 1
Delay_of_start  = 1[s]
Actual_start_time = 26-NOV-2005 11:37
Acq_delay      = 27.9[us]
X90            = 23[us]
Irr90          = 7.4[us]
Tri90          = 10[us]
Qua90         = 10[us]
X90_hi        = 23[us]
Irr90_hi      = 6[us]
Tri90_hi      = 10[us]
Qua90_hi      = 10[us]
X90_lo        = 0.165[ms]
Irr90_lo      = 25[us]
Tri90_lo      = 10[us]
Qua90_lo      = 10[us]
Spin_lock_90  = 1[us]
Spin_lock_attn = 29[dB]
Deut_grad_shim_90 = 1[us]
Deut_grad_shim_attn = 10[dB]
Aqc_card      = 16/1MHz/20
Field_strength = 11.7473579[T]
Filter_mode    = BUTTERWORTH
Filter_width   = 15.72327044[kHz]
Recvr_gain    = 20
Irr_code      = 192
Obs_pwidth    = 1[us]
Obs_noise     = WAUGH
Irr_pwidth    = 45[us]
Irr_noise     = WALTZ
Tri_pwidth    = 1[us]
Tri_noise     = WALTZ
Qua_pwidth    = 1[us]
Qua_noise     = WAUGH
Solvent       = CHLOROFORM-D
Lock_strength  = 907
Lock_level    = 180
Lock_gain     = 18
  
```

X : parts per Million : 13C

----- ACQUISITION PARAMETERS -----
File Name = NO_III_609A1h.3
Author = 1
Sample ID = NO-III-609A1h
Content = 21-JAN-2005 20:22
Creation Date
Revision Date = 14-FEB-2006 07:36
Spec Site = ECP500
Spec Type = DELTA_NMR
Data Format = 1D COMPLEX
Dimensions = X
Dim Title = 1H
Dim Size = 32768
Dim Units = [ppm]
Scans = 16
Mod return = 1
X_points = 32768
X_prescans = 0
X_domain = 1H
X_offset = 5[ppm]
X_freq = 500.15991521[MHz]
X_sweep = 7.50750751[kHz]
X_resolution = 0.22911095[Hz]
Digital filter = 4.3646976[s]
Filter factor = FALSE
Delay of start = 1
Actual_start_time = 1[s]
Acq_delay = 21-JAN-2005 20:20
X90 = 0.1314[ms]
Irr90 = 7.4[us]
Tri90 = 10[us]
Qua90 = 10[us]
X90_hi = 10[us]
Irr90_hi = 10[us]
Qua90_hi = 10[us]
X90_lo = 25[us]
Irr90_lo = 50[us]
Tri90_lo = 10[us]
Qua90_lo = 10[us]
Spin_lock_90 = 1[us]
Spin_lock_attn = 29[dB]
Deut_grad_shim_90 = 1[us]
Deut_grad_shim_attn = 10[dB]
Adc card = 16/1MHz/20
Field strength = 11.7473579[T]
Filter_mode = BUTTERWORTH
Filter_width = 3.75375375[kHz]
Recvr_gain = 14
Irr_code = 192
Obs_pwidth = 1[us]
Irr_pwidth = 81[us]
Tri_pwidth = 1[us]
Qua_pwidth = 1[us]
Solvnt = WAUGH
Lock_strength = BENZENE-D6
Lock_level = 1172
Lock_gain = 160
Lock_osc_offset = 14
Lock_phase = 70.3297[kHz]
Lock_osc_state = 240.7[deg]
= 2H OSC ON

----- ACQUISITION PARAMETERS -----
File Name = NO_III_609A13c.2
Author = S#716786
Sample ID = NO_III_609A13c
Content = 22-JAN-2005 08:06
Creation Date
Revision Date = 14-FEB-2006 07:39
Spec Site = ECP500

Spec Type = DELTA_NMR
Data Format = 1D COMPLEX
Dimensions = X
Dim Title = 13C
Dim Size = 32768
Dim Units = [ppm]
Scans = 1
Mod_return = 32768
X_points = 4
X_prescans = 13C
X_domain = 100[ppm]
X_offset = 125.76529768[MHz]
X_freq = 31.44654088[kHz]
X_sweep = 0.95967227[Hz]
X_resolution = 1H
Irr_domain = 5[ppm]
Irr_offset = 500.15991521[MHz]
Irr_freq = 1.04202224[s]
X_acq_duration = FALSE
Digital_filter = 1
Filter_factor = 1
Delay_of_start = 1[s]
Actual_start_time = 22-JAN-2005 08:06
Acq_delay = 27.9[us]
X90 = 23[us]
Irr90 = 7.4[us]
Tri90 = 10[us]
Qua90 = 23.5[us]
X90_hi = 6[us]
Irr90_hi = 10[us]
Tri90_hi = 10[us]
Qua90_hi = 81[us]
X90_lo = 25[us]
Irr90_lo = 10[us]
Tri90_lo = 10[us]
Qua90_lo = 1[us]
Spin_lock_90 = 29[us]
Spin_lock_attn = 1[us]
Deut_grad_shim_90 = 10[us]
Deut_grad_shim_attn = 10[us]
ADC_card = 16/1MHz/20
Field_strength = 11.7473579[T]
Filter_mode = BUTTERWORTH
Filter_width = 15.72327044[kHz]
Recvr_gain = 30
Irr_code = 192
Obs_pwidth = 1[us]
Obs_noise = WAUGH
Irr_pwidth = 45[us]
Irr_noise = WALTZ
Tri_pwidth = 1[us]
Tri_noise = WALTZ
Qua_pwidth = 1[us]
Qua_noise = WAUGH

Solvent = BENZENE-D6
Lock_strength = 616
Lock_level = 160
Lock_gain = 14

X : parts per Million : 13C

----- ACQUISITION PARAMETERS -----
File Name = NO_III_609A1h.3
Author = 1
Sample ID = NO-III-609A1h
Content = 21-JAN-2005 20:22
Creation Date
Revision Date = 14-FEB-2006 07:36
Spec Site = ECP500
Spec Type = DELTA_NMR
Data Format = 1D COMPLEX
Dimensions = X
Dim Title = 1H
Dim Size = 32768
Dim Units = [ppm]
Scans = 16
Mod return = 1
X_points = 32768
X_prescans = 0
X_domain = 1H
X_offset = 5[ppm]
X_freq = 500.15991521[MHz]
X_sweep = 7.50750751[kHz]
X_resolution = 0.22911095[Hz]
Digital filter = 4.3646976[s]
Filter factor = FALSE
Delay of start = 1
Actual_start_time = 1[s]
Acq_delay = 21-JAN-2005 20:20
X90 = 0.1314[ms]
Irr90 = 7.4[us]
Tri90 = 10[us]
Qua90 = 10[us]
X90_hi = 10[us]
Irr90_hi = 10[us]
Qua90_hi = 10[us]
X90_lo = 25[us]
Irr90_lo = 50[us]
Qua90_lo = 10[us]
Spin_lock_90 = 1[us]
Spin_lock_attn = 29[dB]
Deut_grad_shim_90 = 1[us]
Deut_grad_shim_attn = 10[dB]
Adc card = 16/1MHz/20
Field strength = 11.7473579[T]
Filter_mode = BUTTERWORTH
Filter_width = 3.75375375[kHz]
Recvr_gain = 14
Irr_code = 192
Obs_pwidth = 1[us]
Irr_pwidth = 81[us]
Tri_pwidth = 1[us]
Qua_pwidth = 1[us]
Solvnt = WAUGH
Lock_strength = BENZENE-D6
Lock_level = 1172
Lock_gain = 160
Lock_osc_offset = 14
Lock_phase = 70.3297[kHz]
Lock_osc_state = 240.7[deg]
= 2H OSC ON

X : parts per Million : 13C

----- ACQUISITION PARAMETERS -----	
File Name	= NO_III_609B13c.3
Author	=
Sample ID	= S#293830
Content	= NO III_609B13C
Creation Date	= 22-JAN-2005 13:57
Revision Date	= 14-FEB-2006 07:47
Spec Site	= ECP500
Spec Type	= DELTA NMR
Data Format	= 1D COMPLEX
Dimensions	= X
Dim Title	= 13C
Dim Size	= 32768
Dim Units	= [ppm]
Scans	= 1707
Med return	= 1
X points	= 32768
X prescans	= 4
X domain	= 13C
X_offset	= 100[ppm]
X_freq	= 125.76529768[MHz]
X_resolution	= 31.44654088[kHz]
X_resolution	= 0.95967227[Hz]
Irr domain	= 1H
Irr_offset	= 5[ppm]
Irr_freq	= 500.15991521[MHz]
X_acq_duration	= 1.0420224[s]
Digital_filter	= FALSE
Filter_factor	= 1
Delay of start	= 1[s]
Actual_start_time	= 22-JAN-2005 13:57
Actual_delay	= 27.9[us]
X90	= 23[us]
Irr90	= 7.4[us]
Trt90	= 10[us]
Qua90	= 10[us]
X90_hi	= 23.5[us]
Irr90_hi	= 6[us]
Trt90_hi	= 10[us]
Qua90_hi	= 10[us]
X90_lo	= 81[us]
Irr90_lo	= 25[us]
Trt90_lo	= 10[us]
Qua90_lo	= 10[us]
Spin_lock_90	= 1[us]
Spin_lock_attn	= 29[db]
Deut_grad_shim_90	= 1[us]
Deut_grad_shim_attn	= 10[db]
Acq_card	= 16/1MHz/20
Field strength	= 11.7473579[T]
Filter mode	= BUTTERWORTH
Filter width	= 15.72327044[kHz]
Recvr_gain	= 30
Irr_code	= 192
Obs_width	= 1[us]
Obs_noise	= WAUGH
Irr_width	= 45[us]
Irr_noise	= WALTZ
Tri_width	= 1[us]
Tri_noise	= WALTZ
Qua_width	= 1[us]
Qua_noise	= WAUGH
Solvent	= BENZENE-D6
Lock strength	= 921
Lock level	= 160
Lock gain	= 14