Highly Diastereoselective Synthesis of *trans*-2,5-Disubstituted Tetrahydrofurans

Supporting Information File

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S1	Index
S2	Experimental for <i>des</i> -acetyl 1b
S3	Experimental and data for <i>des</i> -acetyl 1b
S4	Experimental and data for 1b
S5	Data for 3 and Experimental for 5b
S6	Data for 5b and Experimental for 5c
S7	Experimental and data for 7
S8	Experimental and data for 8
S9	¹ H-NMR for <i>des</i> -acetyl 1b
S10	¹³ C-NMR fo <i>des</i> -acetyl 1b
S11	¹ H-NMR for 1b
S12	¹³ C-NMR for 1b
S13	¹ H-NMR for 3
S14	¹³ C-NMR for 3
S15	¹ H-NMR for 5b
S16	¹³ C-NMR for 5b
S17	¹ H-NMR for 5b + 6b
S18	¹ H-NMR for 5c
S19	¹³ C-NMR for 5c
S20	¹ H-NMR for 5c + 6c
S21	¹ H-NMR for 7
S22	¹³ C-NMR for 7
S23	¹ H-NMR for 8
S24	¹³ C-NMR for 8

General : Solvents were purified according to the procedures described. ^1H and ^{13}C NMR spectra were recorded at 200 MHz or 400 MHz. Chemical shifts (δ) were expressed in ppm with the protonated solvent as reference. Patterns were described according to Hoye *et al*¹. and coupling constants (J) were given in hertz (Hz). Flash chromatography was performed with silica gel 60 (9385 Merck), silica gel S (31607 Riedel-de-Haën), and silica gel 60H (7736 Merck). TLC was performed on plates coated with silica gel 60F₂₅₄ (554 Merck).

Preparation of (R)-1b :

Chlorhydrate of D-phenylalanine methyl ester : To a suspension of D-phenylalanine (33 g, 200 mmol) in freshly distilled methanol (250 mL) cooled at $-10\text{ }^{\circ}\text{C}$ under a nitrogen atmosphere, was added dropwise, over 10 min, SO_2Cl_2 (23.25 mL, 320 mmol). The solution was then heated under reflux for 2 h. After cooling to rt the solvent was evaporated and the white solid is dissolved in a minimum amount of MeOH, and precipitated by addition of ether. The precipitate is filtered and washed with ether to give the desired salt (37.84 g, 175 mmol) in 88 % yield.

(R)-2-amino-1,3,3-triphenylpropan-3-ol : The preceding salt (37.84 g, 175 mmol) was placed in ether (250 mL) and a 2 M aqueous NaOH solution (150 mL, 300 mmol) was added at rt. After 20 min of strong stirring, the amino ester was extracted with ether (3x150 mL). The combined ethereal solutions were dried over MgSO_4 , filtered, and the solvent evaporated leaving a white solid. The latter was poured into ether (80 mL) and added, through a dropping funnel, to a flask containing phenylmagnesium bromide (7 equivalents, 2.1 mol) at $0\text{ }^{\circ}\text{C}$ under a nitrogen atmosphere. The temperature was then brought to reflux of ether, and stirring

maintained 9 h. After cooling to rt, ice (250 g) was added and the heterogeneous mixture was filtered through celite. The filtrate was then extracted with EtOAc (5x300 mL), and the combined organic layers dried over MgSO₄, filtered, concentrated to give the crude product which is dissolved in ether and precipitated by addition of pentane, giving after filtration the amino alcohol (37 g, 122.5 mmol) in 70 % yield. Spectroscopic data in agreement with those published by C.S. Da *et al*².

(*R*)-4,4-diphenyl-5-benzyloxazolidin-2-thione : To a solution of (*R*)-2-amino-1,3,3-triphenylpropan-3-ol (21 g, 69.31 mmol) and Et₃N (24.15 mL, 173.28 mmol) in CH₂Cl₂ (250 mL) at 0 °C under a nitrogen atmosphere, was added thiophosgen (5.28 mL, 69.31 mmol) in CH₂Cl₂ (6 mL). Stirring was maintained 30 min at 0 °C, then a 10% aqueous NaHSO₄ solution (30 mL) was added. After separation of the two layers, aqueous solution was extracted with EtOAc. The combined organic layers were washed with 1 M aqueous NH₄OH solution, dried over MgSO₄, filtered and concentrated to give the desired product (21.10 g, 61.2 mmol) in 88 % yield, which is used directly for the next step. . [α]_D : +398 (c 1, CHCl₃). ¹H-NMR (400 MHz, CDCl₃)- δ ppm : 2.20 (dd, J = 1.3, 13.5 Hz, 1H); 2.67 (dd, J = 3.1, 13.9 Hz, 1H); 4.85 (dd, J = 3.2, 11.4 Hz, 1H); 7.11 (m, 3H); 7.35 (m, 10H); 7.53 (m, 2H). ¹³C-NMR (50 MHz, CDCl₃)- δ ppm : 38.9; 65.4; 95.5; 126.0; 126.6; 127.5; 128.4; 128.7; 128.8; 129.2; 136.1; 138.2; 141.0; 187.9. **ESI-MS** m/z : 713 (2M+Na, 53); 368 (M+Na⁺, 55); 308 (100). **IR** cm⁻¹ : 1495; 1450; 1200; 1165; 905; 755; 725; 695.

N-acetyl (*R*)-4,4-diphenyl-5-benzyloxazolidin-2-thione **1b**: To a suspension of NaH (0.41 g, 10.25 mmol) in anhydrous THF (20 mL), (*R*)-4,4-diphenyl-5-benzyloxazolidin-2-thione (3 g, 8.69 mmol) in THF (10 mL) was added at 0 °C. After 10 min of stirring at 0 °C, the solution was warmed to rt and acetyl chloride (0.74 mL, 10.4 mmol) was added dropwise. After 3 h, a saturated aqueous NaHCO₃ solution (15 mL) was added. The organic layer was extracted with CH₂Cl₂ (3x20 mL). The combined organic layers were dried over MgSO₄, filtered, concentrated to give a solid which was purified by flash chromatography (gradient of elution: cyclohexane:EtOAc 9:1 to 7:3) leading to (*R*)-**1b** (3 g, 77.4 mmol) in 89 % yield. $[\alpha]_D^{25}$: +263 (c 0.4, CHCl₃). ¹H-NMR (400 MHz, CDCl₃)- δ ppm : 2.63 (s, 3H); 2.74 (dd, *J* = 7.6, 14.1 Hz, 1H); 2.86 (dd, *J* = 5.9, 13.9 Hz, 1H); 5.88 (dd, *J* = 5.9, 7.4 Hz, 1H); 6.80 (m, 2H); 7.12 (m, 3H); 7.30 (m, 8H); 7.44 (m, 2H). ¹³C-NMR (50 MHz, CDCl₃)- δ ppm : 25.7; 36.3; 65.1; 92.5; 125.8; 126.4; 126.5; 128.1; 128.2; 128.3; 128.8; 129.0; 136.0; 136.9; 140.6; 170.3; 183.9. ESI-MS *m/z* : 410 (M+Na⁺, 100); 286 (38). IR cm⁻¹ : 1700; 1375; 1355; 1325; 1255; 1225; 1205; 1155; 960; 910; 730; 695.

N-(3*S*, 6*S*-epoxy-7-tertbutyldiphenylsilyloxyheptanoate)-4,4-diphenyl-(5*R*)-benzyloxazolidin-2-thione **3d**: To a cooled solution (at 0 °C) of N-acetyl 4,4-diphenyl-5-benzyloxazolidin-2-thione **1b** (0.580 g, 1.5 mmol) in CH₂Cl₂ (5 mL), TiCl₄ (0.16 mL, 1.5 mmol) was added. To this stirred solution was then added DIPEA (0.26 mL, 1.5 mmol) and stirring maintained 20 min before cooling to -20 °C. Pre-cooled CH₂Cl₂ (5 mL) solution (at -20 °C) of acetates **2c** (0.330 g, 1 mmol) was added dropwise. After 30 min, saturated aqueous NH₄Cl solution was added, and the reaction mixture extracted with EtOAc. The combined

organic layers were dried over Na_2SO_4 , filtered and concentrated under vacuum. The crude mixture was then purified by flash chromatography on silica gel affording compound **3d** (0.550 g) in 84 % yield. $[\alpha]_{\text{D}} : +140$ (c 0.63, CHCl_3). **$^1\text{H-NMR}$** (400 MHz, CDCl_3)- δ ppm : 0.05 (s, 6H); 0.87 (s, 9H); 0.89 (t, $J = 6.7$ Hz, 3H); 1.27-1.43 (m, 7H); 1.64 (m, 1H); 1.83 (m, 1H); 1.97 (m, 1H); 2.75 (dd, $J = 7.9, 14.1$ Hz, 1H); 2.88 (dd, $J = 5.1, 14.1$ Hz, 1H); 3.20 (dd, $J = 7.3, 16.9$ Hz, 1H); 3.56 (m, 1H); 3.60 (dd, $J = 5.6, 17.0$ Hz, 1H); 3.89 (m, 1H); 4.23 (m, 1H); 5.85 (dd, $J = 5.2, 7.8$ Hz, 1H); 6.76 (m, 2H); 7.10 (m, 3H); 7.24 (m, 5H); 7.33 (m, 3H); 7.43 (m, 2H). **$^{13}\text{C-NMR}$** (50 MHz, CDCl_3)- δ ppm : -4.7; -4.2; 14.0; 18.2; 22.8; 26.0; 27.1; 27.9; 31.9; 32.3; 36.3; 43.1; 65.4; 74.7; 75.1; 81.5; 92.5; 125.8; 126.4; 126.5; 128.2; 128.3; 128.8; 129.0; 136.1; 136.8; 140.7; 171.2; 183.5. **ESI-MS** m/z : 680 ($\text{M}+\text{Na}^+$, 100); 615 (100). **IR** cm^{-1} : 1700; 1450; 1400; 1360; 1330; 1250; 1210; 1155; 1095; 1070; 970; 835; 775; 700.

(3*S*, 6*S*) Methyl 3,6-epoxy-7-tertbutyldiphenylsilyloxyheptanoate **5b**: To a cooled solution (at 0 °C) of N-acetyl 4,4-diphenyl-5-benzyloxazolidin-2-thione **1b** (0.580 g, 1.5 mmol) in CH_2Cl_2 (5 mL), TiCl_4 (0.165 mL, 1.5 mmol) was added. To this stirred solution was then added DIPEA (0.26 mL, 1.5 mmol) and stirring maintained 20 min before cooling to -20 °C. Pre-cooled CH_2Cl_2 (5 mL) solution (at -20 °C) of acetates **2b** (0.398 g, 1 mmol) was added dropwise. After 30 min, saturated aqueous NH_4Cl solution (8 mL) was added, and the reaction mixture extracted with EtOAc. The combined organic layers were dried over Na_2SO_4 , filtered and concentrated under vacuum. The crude mixture (0.856 g) was then poured into MeOH (5 mL), and K_2CO_3 (0.500 g, 3.6 mmol) added. After 20 min of stirring at rt, cyclohexane (5 mL) was added and the reaction mixture filtered through a short pad of

celite. The filtrate was washed with water (5 mL), dried over MgSO_4 , filtered and concentrated under vacuum. The crude mixture was purified by flash chromatography on silica gel (cyclohexane/ethyl acetate 8:2 to 7:3) to afford **5b** (0.225 g) in 55 % yield. $[\alpha]_D$: +7 (c 3, CHCl_3). **$^1\text{H-NMR}$** (400 MHz, CDCl_3)- δ ppm : 1.07 (s, 9H); 1.61 (m, 1H); 1.91 (m, 1H); 2.03 (m, 1H); 2.15 (m, 1H); 2.48 (dd, $J = 6.1, 15.0$ Hz, 1H); 2.62 (dd, $J = 7.2, 15.0$ Hz, 1H); 3.66 (m, 2H); 3.69 (s, 3H); 4.18 (m, 1H); 4.40 (quint, $J = 7.0$ Hz, 1H); 7.40 (m, 6H); 7.70 (m, 4H). **$^{13}\text{C-NMR}$** (50 MHz, CDCl_3)- δ ppm : 19.2; 26.8; 27.8; 31.7; 40.6; 51.5; 66.3; 75.6; 79.2; 127.6; 129.5; 133.6; 135.6; 171.6. **ESI-MS** m/z : 435 ($\text{M}+\text{Na}^+$, 100). **IR** cm^{-1} : 1740; 1430; 1110; 1075; 1005; 825; 740; 700.

(3*S*, 6*S*, 7*S*) Methyl 3,6-epoxy-7-tertbutyldimetylsilyloxyundecanoate **5c**: To a solution of **3d** (0.20 g, 0.30 mmol) in MeOH (5 mL), was added K_2CO_3 (0.100 g, 0.723 mmol). After 20 min of stirring at rt, cyclohexane (5 mL) was added and the reaction mixture filtered through a short pad of celite. The filtrate was washed with water (5 mL), dried over MgSO_4 , filtered and concentrated under vacuum. The crude mixture was purified by flash chromatography on silica gel (cyclohexane/ethyl acetate 9:1 to 7:3) to afford **5c** (0.092 g) in 88 % yield. $[\alpha]_D$: -6 (c 1.3, CHCl_3). **$^1\text{H-NMR}$** (400 MHz, CDCl_3)- δ ppm : 0.05 (s, 6H); 0.88 (br s, 12H); 1.31 (m, 6H); 1.55 (m, 2H); 1.89 (m, 1H); 2.10 (m, 1H); 2.43 (dd, $J = 6.3, 15.0$ Hz, 1H); 2.61 (dd, $J = 7.1, 15.0$ Hz, 1H); 3.55 (m, 1H); 3.67 (s, 3H); 3.92 (m, 1H); 4.32 (m, 1H). **$^{13}\text{C-NMR}$** (50 MHz, CDCl_3)- δ ppm : -4.8; -4.3; 14.0; 18.2; 22.8; 26.0; 27.6; 27.8; 32.2; 32.5; 40.7; 51.5; 75.0; 75.4; 81.9; 171.8. **ESI-MS** m/z : 353 ($\text{M}+\text{Na}-\text{CH}_3$, 100); 325 (13); 293 (75). **IR** cm^{-1} : 1740; 1380; 1140; 1090; 835; 775.

(3*S*, 6*S*, 7*S*) 3,6-epoxy-7-tertbutyldimetylsilyloxyundecan-1-ol **7**: To a suspension of LiBH₄ (0.025 g, 1.15 mmol) in THF (4 mL) under a nitrogen atmosphere, a solution of **3d** (0.150 g, 0.23 mmol) in THF (4 mL) was added at 0 °C, followed by MeOH (0.037 mL, 0.92 mmol). After 45 min of stirring at rt, a 1M aqueous solution of sodium and potassium tartrate (5 mL) was added. Water (5 mL) and Et₂O (5 mL) were then added. The two phases were separated, the organic layer extracted with EtOAc (3x 10 mL). The combined organic layers were dried over MgSO₄, filtered, and concentrated. The crude mixture was then purified by flash chromatography (cyclohexane/ethyl acetate 7:3) to afford **7** (0.60 g) in 83 % yield. $[\alpha]_D$: -4 (c 1.43, CHCl₃). ¹H-NMR (400 MHz, CDCl₃)- δ ppm : 4.11 (m, 1H); 3.97 (td, *J* = 6.2, 14.1 Hz, 1H); 3.77 (dd, *J* = 5.7, 5.1 Hz, 2H); 3.55 (m, 1H); 2.96 (br s, OH); 2.02 (m, 1H); 1.90 (m, 1H); 1.80-1.45 (m, 4H); 1.30 (m, 6H); 0.88 (br s, 12H); 0.06 (s, 3H); 0.05 (s, 3H). ¹³C-NMR (50 MHz, CDCl₃)- δ ppm : -4.5; -4.3; 14.0; 18.1; 22.9; 25.9; 27.4; 27.5; 32.5; 32.9; 37.3; 61.6; 74.8; 79.5; 81.6. ESI-MS *m/z* : 339 (M+Na⁺, 100). IR cm⁻¹ : 1460; 1250; 1060; 1020; 940; 835; 775.

(3*S*, 6*S*, 7*S*) 3,6-epoxy-7-tertbutyldimetylsilyloxyundecanal **8**: To a solution of **3d** (0.130 g, 0.197 mmol) in CH₂Cl₂ (5 mL), Dibal-H (0.20 mL, 0.197 mmol, of a 1M CH₂Cl₂ solution) was added at -78 °C, under a nitrogen atmosphere. After 40 min of stirring, a saturated aqueous NH₄Cl (8 mL) solution was added. The two phases were separated, and the organic layer extracted with EtOAc (3x 10 mL). The combined organic layers were dried over

MgSO₄, filtered, concentrated and the crude mixture purified by flash chromatography over silica gel (cyclohexane/ethyl acetate 9:1, then 8:2) to afford **8** (0.056 g) in 90 % yield. $[\alpha]_D$: -2 (c 1.22, CHCl₃). **¹H-NMR** (400 MHz, CDCl₃)- δ ppm : 0.03 (s, 6H); 0.87 (br s, 12H); 1.30 (m, 5H); 1.44-1.58 (m, 2H); 1.70 (m, 1H); 1.92 (m, 1H); 2.12 (m, 1H); 2.53 (ddd, J = 1.6, 5.0, 16.1 Hz, 1H); 2.64 (ddd, J = 2.4, 7.3, 16.0 Hz, 1H); 3.54 (m, 1H); 3.94 (m, 1H); 4.35 (m, 1H); 9.79 (t, J = 2.1 Hz, 1H). **¹³C-NMR** (50 MHz, CDCl₃)- δ ppm : -4.7; -4.3; 14.0; 18.2; 22.8; 25.9; 27.6; 27.7; 32.5; 32.6; 49.5; 74.0; 75.0; 81.8; 201.3. **ESI-MS** m/z : 337 (M+Na⁺). **IR** cm⁻¹ : 1730; 1640; 1390; 1250; 1090; 1020; 940; 835; 775. Anal. Calcd. for C₁₇H₃₄O₃Si: C, 64.92; H, 10.90. Found: C, 64.98; H, 10.79.

References

- (1) Hoye, T. R.; Hanson, P. R.; Vyvyan J. R. *J. Org. Chem.* **1994**, *59*, 4096-4103
- (2) Da, C.-s.; Han, Z.-j.; Ni, M.; Yang, F.; Liu, D.-x.; Zhou, Y.-f.; Wang, R. *Tetrahedron Asym.* **2003**, *14*, 659-665

RMN ^1H (200MHz, CDCl_3) δ ppm :

SF: 200.13 MHz SW: 4132.23 Hz AQ: 7.93 seconds TD: 65536 points Scale units: ppm

RMN ^{13}C (50MHz, CDCl_3) δ ppm :

File:fid			
Threshold: 12.25			
FROM: 190.87 ppm	TO: 179.90 ppm		
Peak	Frequency	ppm	Intensity
1	9453.69	187.87	18.163
FROM: 143.72 ppm	TO: 129.87 ppm		
Peak	Frequency	ppm	Intensity
1	7094.51	140.58	20.543
2	6954.15	136.20	23.303
3	6846.08	136.05	28.735
FROM: 132.18 ppm	TO: 114.85 ppm		
Peak	Frequency	ppm	Intensity
1	6502.49	129.22	110.061
2	6482.94	128.93	151.945
3	6477.15	128.72	103.050
4	6462.23	128.42	145.849
5	6413.73	127.46	45.771
6	6369.93	126.59	96.863
7	6342.56	126.04	104.090
FROM: 98.49 ppm	TO: 89.45 ppm		
Peak	Frequency	ppm	Intensity
1	4804.06	95.47	19.413
FROM: 82.91 ppm	TO: 68.28 ppm		
Peak	Frequency	ppm	Intensity
1	3906.62	77.64	33.879
2	3874.44	77.00	38.718
3	3842.74	76.37	37.947
FROM: 70.78 ppm	TO: 50.00 ppm		
Peak	Frequency	ppm	Intensity
1	3290.01	65.38	42.028
FROM: 42.11 ppm	TO: 30.56 ppm		
Peak	Frequency	ppm	Intensity
1	1959.60	38.94	39.714

SF: 50.32 MHz SW: 12820.51 Hz AQ: 2.56 seconds TD: 65536 points Scale units: ppm

1b

RMN ^1H (200MHz, CDCl_3) δ ppm :

1b

RMN ^{13}C (50MHz, CDCl_3) δ ppm :

File: fid		Threshold: 11.73
FROM: 184.99 ppm	TO: 182.75 ppm	Intensity
Peak	Frequency	
1	9254.91	6.305
FROM: 172.28 ppm	TO: 169.32 ppm	Intensity
Peak	Frequency	
1	8570.61	12.422
FROM: 143.02 ppm	TO: 139.22 ppm	Intensity
Peak	Frequency	
1	7074.06	11.298
FROM: 137.43 ppm	TO: 133.82 ppm	Intensity
Peak	Frequency	
1	6843.63	10.361
FROM: 129.37 ppm	TO: 125.12 ppm	Intensity
Peak	Frequency	
1	6489.94	89.233
2	6482.51	134.545
3	6454.34	99.846
4	6451.21	70.119
5	6448.86	103.565
6	6365.91	48.468
7	6351.42	90.852
8	6327.57	99.351
FROM: 93.28 ppm	TO: 91.76 ppm	Intensity
Peak	Frequency	
1	4656.54	10.960
FROM: 78.58 ppm	TO: 75.15 ppm	Intensity
Peak	Frequency	
1	3906.90	14.154
2	3874.82	15.101
3	3842.73	14.287
FROM: 65.34 ppm	TO: 64.84 ppm	Intensity
Peak	Frequency	
1	1273.46	35.869
FROM: 39.53 ppm	TO: 34.63 ppm	Intensity
Peak	Frequency	
1	1827.40	56.436
FROM: 26.03 ppm	TO: 25.41 ppm	Intensity
Peak	Frequency	
1	1234.91	40.602

SF: 50.32 MHz SW: 12820.51 Hz AQ: 2.56 seconds TD: 65536 points Scale units: ppm

RMN ^1H (400MHz, CDCl_3) δ ppm:

3d

RMN ^{13}C (50MHz, CDCl_3) δ ppm :

File:f1d		Threshold: 12.81	
FROM: 185.23 ppm TO: 134.49 ppm		Intensity	
Peak	Frequency	Peak	Frequency
1	9233.09	1	183.49
2	8612.94	2	171.16
3	7080.45	3	140.71
4	6886.28	4	136.85
5	6848.46	5	136.10
FROM: 130.13 ppm TO: 124.83 ppm		Intensity	
Peak	Frequency	Peak	Frequency
1	6489.62	1	128.97
2	6482.11	2	128.82
3	6433.54	3	128.25
4	6377.52	4	126.54
5	6367.81	5	126.45
6	6331.82	6	125.83
FROM: 93.25 ppm TO: 91.12 ppm		Intensity	
Peak	Frequency	Peak	Frequency
1	4655.41	1	92.52
FROM: 81.90 ppm TO: 80.70 ppm		Intensity	
Peak	Frequency	Peak	Frequency
1	4100.25	1	81.48
FROM: 78.64 ppm TO: 75.07 ppm		Intensity	
Peak	Frequency	Peak	Frequency
1	3906.61	1	77.64
2	3874.39	2	76.37
3	3842.75	3	75.05
FROM: 75.60 ppm TO: 74.05 ppm		Intensity	
Peak	Frequency	Peak	Frequency
1	3778.19	1	75.08
2	3750.17	2	74.73
FROM: 56.10 ppm TO: 64.85 ppm		Intensity	
Peak	Frequency	Peak	Frequency
1	3288.70	1	65.36
FROM: 45.71 ppm TO: 26.39 ppm		Intensity	
Peak	Frequency	Peak	Frequency
1	2167.46	1	44.701
2	1825.06	2	43.07
3	1623.61	3	36.27
4	1605.67	4	32.27
5	1501.64	5	31.92
6	1362.59	6	27.08
FROM: 26.54 ppm TO: 24.92 ppm		Intensity	
Peak	Frequency	Peak	Frequency
1	1362.59	1	27.08
FROM: 24.26 ppm TO: 16.83 ppm		Intensity	
Peak	Frequency	Peak	Frequency
1	1119.81	1	22.85
FROM: 14.43 ppm TO: -5.46 ppm		Intensity	
Peak	Frequency	Peak	Frequency
1	106.41	1	14.04
2	-210.15	2	-4.18
3	-234.77	3	-4.67
FROM: 14.43 ppm TO: -5.46 ppm		Intensity	
Peak	Frequency	Peak	Frequency
1	106.41	1	14.04
2	-210.15	2	-4.18
3	-234.77	3	-4.67
FROM: 14.43 ppm TO: -5.46 ppm		Intensity	
Peak	Frequency	Peak	Frequency
1	106.41	1	14.04
2	-210.15	2	-4.18
3	-234.77	3	-4.67

SF: 50.32 MHz 175 150 125 100 75 50 25 0

SW: 12820.51 Hz AQ: 2.56 seconds TD: 65536 points Scale units: ppm

RMN ^1H (400MHz, CDCl_3) δ ppm :

RMN ^{13}C (50MHz, CDCl_3) δ ppm :

File: ffd		Threshold: 4.11	
FROM: 172.19 ppm	TO: 171.00 ppm		
Peak 1	Frequency 171.59	Intensity 11.027	
FROM: 136.45 ppm	TO: 134.46 ppm		
Peak 1	Frequency 135.56	Intensity 107.385	
FROM: 134.32 ppm	TO: 132.89 ppm		
Peak 1	Frequency 133.59	Intensity 16.022	
FROM: 130.65 ppm	TO: 128.95 ppm		
Peak 1	Frequency 129.51	Intensity 56.400	
FROM: 128.70 ppm	TO: 126.06 ppm		
Peak 1	Frequency 127.56	Intensity 133.753	
FROM: 79.97 ppm	TO: 78.54 ppm		
Peak 1	Frequency 79.20	Intensity 33.210	
FROM: 78.16 ppm	TO: 75.73 ppm		
Peak 1	Frequency 77.63	Intensity 8.655	
Peak 2	Frequency 77.00	Intensity 8.946	
Peak 3	Frequency 76.36	Intensity 8.494	
FROM: 75.89 ppm	TO: 74.71 ppm		
Peak 1	Frequency 75.56	Intensity 34.794	
FROM: 66.93 ppm	TO: 65.25 ppm		
Peak 1	Frequency 66.29	Intensity 31.429	
FROM: 51.88 ppm	TO: 51.07 ppm		
Peak 1	Frequency 51.45	Intensity 24.387	
FROM: 41.49 ppm	TO: 26.97 ppm		
Peak 1	Frequency 40.58	Intensity 37.479	
Peak 2	Frequency 31.69	Intensity 31.067	
Peak 3	Frequency 27.76	Intensity 32.613	
FROM: 27.47 ppm	TO: 25.87 ppm		
Peak 1	Frequency 26.75	Intensity 69.060	
FROM: 20.04 ppm	TO: 18.26 ppm		
Peak 1	Frequency 19.16	Intensity 26.872	

SF: 50.32 MHz SW: 12820.51 Hz AQ: 2.56 seconds TD: 65536 points Scale units: ppm

6b

77:23

minor

RMN ¹H (400MHz, CDCl₃) δppm:

8.0 7.5
SF: 400.13 MHz

SW: 3333.33 HZ

AQ: 4.92 seconds

TD: 32768 points

Scale units: ppm

RMN ^1H (400MHz, CDCl_3) δ ppm :

SF: 400.13 MHz SW: 8250.83 Hz AQ: 3.97 seconds TD: 65536 points Scale units: ppm

RMN ^{13}C (50MHz, CDCl_3) δ ppm :

File: f1d
Threshold: 16.69

FROM: 171.94 ppm	TO: 171.59 ppm	Intensity
Peak	Frequency	ppm
1	9643.09	171.76
FROM: 82.41 ppm	TO: 81.46 ppm	Intensity
Peak	Frequency	ppm
1	4120.90	81.89
FROM: 78.15 ppm	TO: 75.92 ppm	Intensity
Peak	Frequency	ppm
1	3906.25	77.63
2	3874.04	76.99
3	3842.35	76.36
FROM: 75.56 ppm	TO: 74.45 ppm	Intensity
Peak	Frequency	ppm
1	3793.84	75.35
2	3773.51	74.99
FROM: 51.74 ppm	TO: 51.21 ppm	Intensity
Peak	Frequency	ppm
1	2591.18	51.49
FROM: 41.12 ppm	TO: 31.48 ppm	Intensity
Peak	Frequency	ppm
1	2045.34	40.65
2	1634.16	32.48
3	1620.45	32.20
FROM: 28.29 ppm	TO: 27.22 ppm	Intensity
Peak	Frequency	ppm
1	1397.09	27.76
2	1387.35	27.57
FROM: 26.52 ppm	TO: 24.81 ppm	Intensity
Peak	Frequency	ppm
1	1306.28	25.96
FROM: 23.71 ppm	TO: 22.14 ppm	Intensity
Peak	Frequency	ppm
1	1149.37	22.84
FROM: 18.60 ppm	TO: 17.92 ppm	Intensity
Peak	Frequency	ppm
1	918.20	18.25
FROM: 17.45 ppm	TO: 16.63 ppm	Intensity
Peak	Frequency	ppm
1	857.56	17.04
FROM: 14.78 ppm	TO: 13.41 ppm	Intensity
Peak	Frequency	ppm
1	704.95	14.01
FROM: -3.67 ppm	TO: -5.18 ppm	Intensity
Peak	Frequency	ppm
1	215.69	-4.29
2	239.84	-4.77
		32.298

SF: 50.32 MHz

SW: 12820.51 Hz

AQ: 2.56 seconds

TD: 65536 points

Scale units: ppm

60:40

RMN ^1H (400MHz, CDCl_3) δ ppm:

AQ: 4.88 seconds

SF: 400.13 MHz
 SW: 3360.22 Hz
 AQ: 4.88 seconds
 TD: 32768 points
 Scale units: ppm

RMN ^1H (400MHz, CDCl_3) δ ppm :

RMN ^{13}C (50MHz, CDCl_3) δ ppm :

File:fid
Threshold: 11.46

FROM: 82.28 ppm TO: 78.41 ppm	Intensity
Peak Frequency ppm	
1 4106.94 81.62	64.062
2 4024.47 79.54	58.431
FROM: 77.94 ppm TO: 75.71 ppm	
Peak Frequency ppm	
1 3906.57 77.00	77.00
2 3879.46 76.17	76.17
3 3842.80 74.78	74.78
FROM: 76.20 ppm TO: 73.40 ppm	
Peak Frequency ppm	
1 61.93 ppm TO: 61.14 ppm	61.14
FROM: 61.93 ppm TO: 61.14 ppm	
Peak Frequency ppm	
1 3105.62 61.72	61.72
FROM: 37.40 ppm TO: 26.97 ppm	
Peak Frequency ppm	
1 2684.43 32.60	32.60
2 1635.01 32.49	32.49
3 1384.99 27.52	27.52
4 1378.37 27.39	27.39
FROM: 26.88 ppm TO: 24.33 ppm	
Peak Frequency ppm	
1 1303.97 25.91	25.91
FROM: 23.27 ppm TO: 22.40 ppm	
Peak Frequency ppm	
1 1154.16 22.34	22.34
FROM: 18.29 ppm TO: 18.01 ppm	
Peak Frequency ppm	
1 913.52 18.16	18.16
FROM: 14.19 ppm TO: 13.63 ppm	
Peak Frequency ppm	
1 905.00 14.01	14.01
FROM: -3.99 ppm TO: -5.04 ppm	
Peak Frequency ppm	
1 -217.94 -4.33	-4.33
2 -227.32 -4.52	-4.52
FROM: 14.19 ppm TO: 13.63 ppm	
Peak Frequency ppm	
1 905.00 14.01	14.01
FROM: -3.99 ppm TO: -5.04 ppm	
Peak Frequency ppm	
1 -217.94 -4.33	-4.33
2 -227.32 -4.52	-4.52

SF: 50.32 MHz SW: 12820.51 Hz AQ: 2.56 seconds ID: 65536 points Scale units: ppm

RMN ^1H (400MHz, CDCl_3) δ ppm :

RMN ^1H (400MHz, CDCl_3) δ ppm :

File: f1d
Threshold: 10.59

FROM: 201.84 ppm	TO: 200.56 ppm	Intensity
Peak	Frequency	ppm
1	10128.23	27.534
FROM: 82.52 ppm	TO: 80.91 ppm	Intensity
Peak	Frequency	ppm
1	4118.24	63.531
FROM: 78.18 ppm	TO: 75.92 ppm	Intensity
Peak	Frequency	ppm
1	3906.56	77.63
2	3874.76	77.00
3	3842.76	76.37
FROM: 75.41 ppm	TO: 73.42 ppm	Intensity
Peak	Frequency	ppm
1	3771.52	74.35
2	3723.41	73.39
FROM: 49.88 ppm	TO: 49.09 ppm	Intensity
Peak	Frequency	ppm
1	2491.51	49.51
FROM: 33.09 ppm	TO: 31.77 ppm	Intensity
Peak	Frequency	ppm
1	1642.32	32.64
2	1633.82	32.47
FROM: 28.87 ppm	TO: 26.39 ppm	Intensity
Peak	Frequency	ppm
1	1351.97	27.66
2	1388.08	27.59
FROM: 26.85 ppm	TO: 24.15 ppm	Intensity
Peak	Frequency	ppm
1	1303.19	25.90
FROM: 23.18 ppm	TO: 22.04 ppm	Intensity
Peak	Frequency	ppm
1	1147.08	22.80
FROM: 18.70 ppm	TO: 17.48 ppm	Intensity
Peak	Frequency	ppm
1	915.03	18.18
FROM: 15.17 ppm	TO: 12.97 ppm	Intensity
Peak	Frequency	ppm
1	702.20	13.95
FROM: -3.46 ppm	TO: -5.40 ppm	Intensity
Peak	Frequency	ppm
1	-216.50	-4.30
2	-236.09	-4.69

SF: 50.32 MHz
SW: 12820.51 Hz
AQ: 2.56 seconds
TD: 65536 points
Scale units: ppm