Control of Olefin Geometry in Macrocyclic Ring-Closing Metathesis Using a Removable Silyl Group

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Contents:

Full reference 14b: Paone, D. V.; Shaw, A. W.; Nguyen, D. N.; Burgey, C. S.; Deng, J. Z.; Kane, S. A.; Koblan, K. S.; Salvatore, C. A.; Mosser, S. D.; Johnston, V. K.; Wong, B. K.; Miller-Stein, C. M.; Hershey, J. C.; Graham, S. L.; Vacca, J. P.; Williams, T. M. *J. Med. Chem.* **2007**, *50*, 5564-5567.

Full reference 22: Marcaurelle, L. A.; Comer, E.; Dandapani, S.; Duvall, J. R.; Gerard, B.; Kesavan, S.; Lee, M. D.; Liu, H.; Lowe, J. T.; Marie, J.-C.; Mulrooney, C. A.; Pandya, B. A.; Rowley, A.; Ryba, T. D.; Suh, B.-C.; Wei, J.; Young, D. W.; Akella, L. B.; Ross, N. T.; Zhang, Y.-L.; Fass, D. M.; Reis, S. A.; Zhao, W.-N.; Haggarty, S. J.; Palmer, M.; Foley, M. A. *J. Am. Chem. Soc.* **2010**, *132*, 16962-16976.

Material and Methods.

Except as otherwise noted, reactions were carried out under argon. All reaction solvents except acetone and pyridine were dispensed from a solvent purification system wherein solvents are passed through a packed activated alumina column. Acetone was Aldrich 99.5+% histological grade. Pyridine was Aldrich 99.8% histological grade. NMR spectra were recorded at 500 MHz using a Varian I-500 instrument. Chemical shifts for proton NMR spectra are reported in parts per million downfield from tetramethylsilane and were referenced to residual protonated solvent ($CHCl_3$: d 7.26, C_6H_6: d 7.15). Chemical shifts for carbon NMR spectra are reported in parts per million downfield from tetramethylsilane and referenced to protonated solvent ($CHCl_3$: d 77.0, C_6H_6: d 128.0). Data are represented as follows: chemical shift (multiplicity [bs = broad singlet, s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet], coupling constants in Hertz, integration). High-resolution mass spectra were obtained through the Harvard University mass spectrometry facility. Infrared spectra were obtained with a Nicolet IR100 FTIR from Thermo Scientific. Optical rotations were obtained using digital polarimeter Autopol IV (Rudolph research Analytical) with a 1 mL cell and a 1 dm path length. All reactions were magnetically stirred and monitored by thin-layer chromatography (TLC) using E. Merck silica gel 60 F254 precoated plates (0.25 mm). Flash chromatography was performed either with the indicated solvent on E. Merck silica gel 60 (230-400 mesh) or using a CombiFlash companion system (Teledyne ISCO, Inc.) with pre-packed FLASH silica gel columns (Teledyne ISCO, Inc.). SFC/MS chromatography was performed with a Berger analytic SFC (Waters ZQ Mass Spectometer) using CO_2 and isopropanol as the mobile phase and using a Chiralpak® AD-H column purchased from Chiral Technology Inc. (column length: 4.6x250mm, particle size: 5um). HPLC purification was performed on a Waters mass-directed autopurification system. The system consisted of 2767 injection/collection sample manager, a 2525 binary gradient high pressure LC pump, two 515 pumps to deliver makeup and dilution flow, a column fluidic organizer (CFO), a 2996 photodiode array detector, and a ZQ quadropole MS equipped with an electrospray interface. All of the instrumentation was controlled by MassLynx and FractionLynx software versions 4.1. All reagents were obtained from commercial sources and used without further purification.

Experimental Procedures.

A. General procedures for hydrosilylation, ring-closing metathesis, and protodesilylation.

Hydrosilylation: following the literature procedure,[1] to a solution of the alkyne substrate (1 equiv.) in DCM (0.5 M) was added the diethoxymethylsilane (1.1 equiv.). The flask was cooled to 0 °C and catalyst $[Cp^*Ru(MeCN)_3]PF_6$ (5 mol%) was added. The ice bath was immediately removed and the solution was stirred for 30 min. The resulting mixture was concentrated under reduced pressure and the residue was purified by silica gel column chromatography using Hexanes/EtOAc as eluent.

Ring-closing metathesis (RCM) of vinyl siloxane substrates: substrate (1 equiv.) was dissolved in anhydrous toluene (or other solvent when indicated) at a concentration of 2 mM under argon. 20 mol% catalyst **A** was added to the solution. High vacuum was applied to the reaction flask for 5 min and charged with argon. This operation cycle was repeated for 5 times. The reaction was then heated up to 35 ^oC and left for 12 hours. The resulting mixture was concentrated under reduced pressure and the residue was analyzed by ^1H NMR or purified by silica gel column chromatography using Hexanes/EtOAc as eluent.

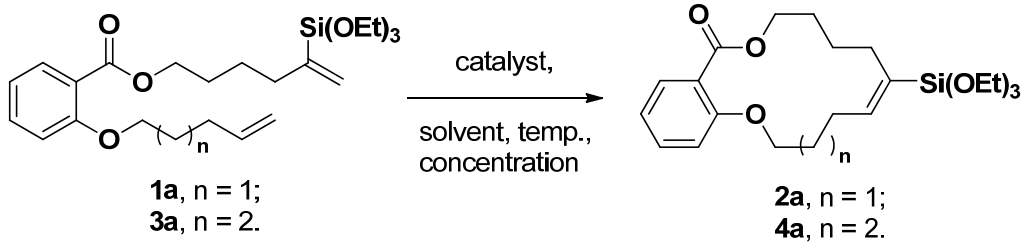
RCM of simple di-olefinic substrates: substrate (1 equiv.) was dissolved in anhydrous toluene (or other solvent when indicated) at a concentration of 2 mM under argon. 20 mol% catalyst **A** or 10 mol% Grubbs II, and 20 mol% 1,4-benzoquinone was added to the solution. High vacuum was applied to the reaction flask for 5 min and charged with argon. This operation cycle was repeated for 5 times. The reaction was then heated up to 35 ^oC and left for 12 hours. The resulting mixture was concentrated under reduced pressure and the residue was analyzed by ^1H NMR or purified by silica gel column chromatography using Hexanes/EtOAc as eluent.

Protodesilylation: adapted from the literature procedure,[2] the alkenyl siloxane product (1 equiv.) from the RCM reaction was dissolved in a anhydrous THF to a final concentration of 0.25 M. AgF (0.5 equiv.) was added to the solution immediately followed by acetic acid (1.5 equiv.) and TBAF (2.5 equiv., 1 M

solution in THF). The reaction was kept in dark and stirred for 2 hours. The resulting mixture was filtered with celite, concentrated under reduced pressure and the residue was purified by silica gel column chromatography using Hexanes/EtOAc as eluent.

B. Catalysts screening and reaction conditions optimization.

Scheme S1. RCM of model substrate for catalysts screening and reaction conditions optimization.

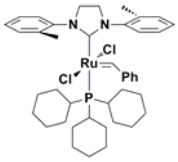
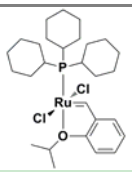
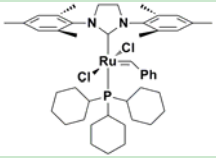
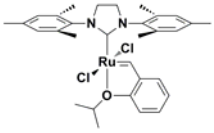
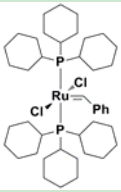
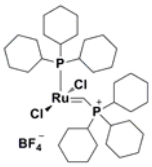
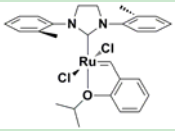
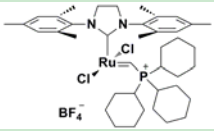
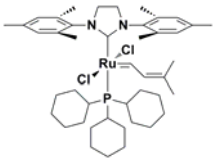
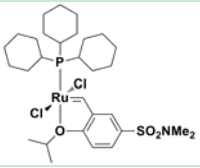
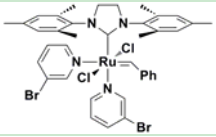
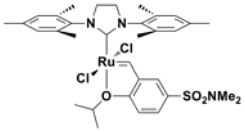
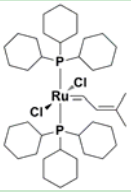
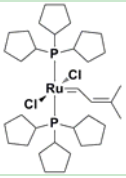
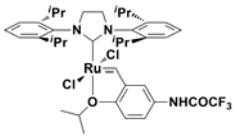
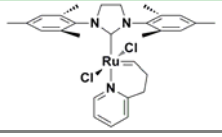


1a, n = 1;
3a, n = 2.

2a, n = 1;
4a, n = 2.

To a round-bottomed flask equipped with magnetic stir bar and armed with a condenser was added substrate **3a** (1.0 equiv.) in anhydrous dichloromethane (2 mM) under argon. The catalyst (0.2 equiv.) was then added and the reaction was refluxed for 18 hours. The mixture was cooled to room temperature, concentrated under reduced pressure. The conversion was analyzed by crude proton NMR study using $CDCl_3$ as solvent (**Table S1**). Representative NMR spectrum (olefinic proton area) of the RCM reaction of substrate **3a** with catalyst **A** was shown in **Figure S1**. The peak at 6.23 ppm (t) was the resonance of olefin proton within product **4a** (the overlap of product peak with one of the styrene olefin proton was corrected by subtracting integration of the other styrene olefin proton (6.42-6.39 ppm) from the integration of 6.26-6.20 ppm). Unreacted starting material, acyclic cross-dimmers, and the styrene derivative share the common moiety of vinylsiloxane which gives two terminal olefin proton peaks at 5.73 and 5.65 ppm. Integration for one of them and the corrected integration of desired product were then used for determination of the conversion of the reaction.

Table S1. Conversion of the RCM reaction of substrate **3a** with various catalysts to desired product.

Entry	Catalyst	Conversion to product (%)	Entry	Catalyst	Conversion to product (%)
A		19	**J**		< 2
B		< 2	**K**		< 2
C		< 2	**L**		< 2
D		3	**M**		< 2
E		< 2	**N**		< 2
F		< 2	**O**		< 2
G		< 2	**P**		< 2
H		< 2	**Q**		< 2
I		< 2			

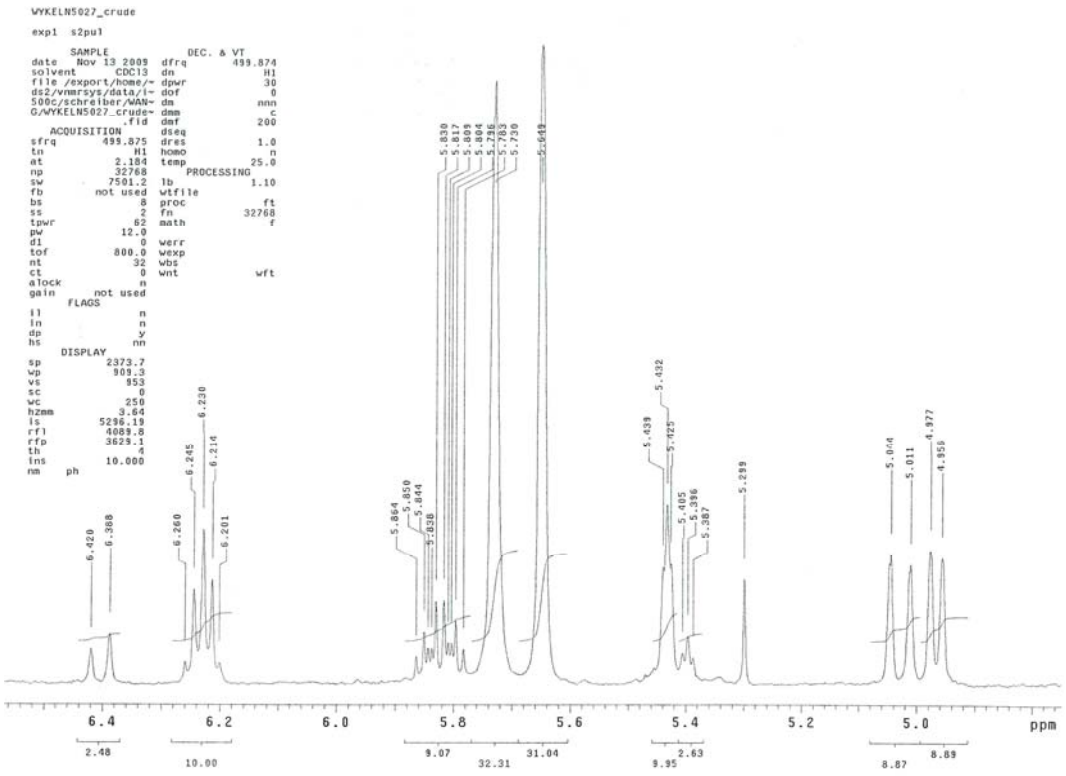


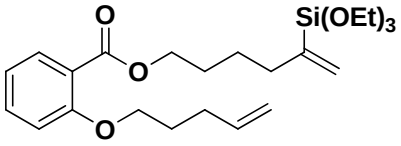
Figure S1. Representative crude proton NMR spectrum (olefinic proton area) of RCM reaction for catalysts screening, reaction condition optimization, and catalyst decomposition studies. Reaction condition: substrate **3** with catalyst **A**, DCM, reflux, 18 hours.

Reaction conditions for RCM of substrate **3a** were then optimized (Table **S2**). After varying solvents, temperature, and concentrations we found that optimal results (63%) were obtained using benzene or toluene as a solvent, temperatures of 35 °C and 20-mol% of catalyst.

Table S2. Optimization of reaction conditions.

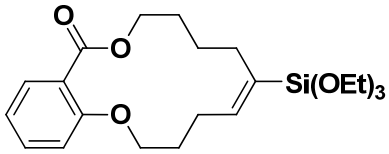
Entry	Catalyst	Solv.	Temp. (^{o}C)	Conc. (mM)	^{1}H NMR yield (%)[a]
1	**Grubbs I**	CH_2Cl_2	reflux	2	< 2
2	**Grubbs II**	CH_2Cl_2	reflux	2	3
3	**A**	CH_2Cl_2	reflux	2	19
4	**D**	CH_2Cl_2	reflux	2	3
5	**A**	$(CH_2Cl)_2$	50	2	15
6	**A**	C_6H_6	50	2	54
7	**A**	Ph-CH_3	50	2	50
8	**A**	C_6H_6	23	2	42
9	**A**	C_6H_6	30	2	63
10	**A**	C_6H_6	40	2	63
11	**A**	C_6H_6	60	2	45
12	**A**	C_6H_6	35	1	52
13	**A**	C_6H_6	35	5	39
14	**A**	C_6H_6	35	10	20
15	**A**	C_6H_6	35	20	12

[a] Yield calculated based on ^{1}H NMR analysis of reaction mixtures.



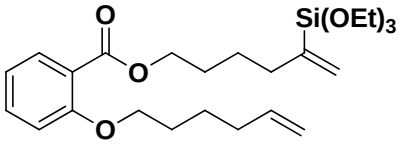
5-(Triethoxysilyl)hex-5-en-1-yl 2-(pent-4-en-1-yloxy)benzoate (1a)

Yield 72% (colorless oil); IR (neat, cm^{-1}) 3077, 2974, 2927, 2890, 2736, 1729, 1705, 1641, 1601, 1583, 1492, 1469, 1452, 1390, 1301, 1251, 1165, 1080, 1016, 958; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 7.78-7.76 (m, 1 H), 7.44-7.40 (m, 1 H), 6.97-6.93 (m, 2 H), 5.85 (ddt, *J* = 17.0, 10.5, 6.8 Hz, 1 H), 5.74-5.73 (m, 1 H), 5.65-5.65 (m, 1 H), 5.08-5.04 (m, 1 H), 4.99 (d, *J* = 10.0 Hz, 1 H), 4.30 (t, *J* = 6.8 Hz, 2 H), 4.04 (t, *J* = 6.5 Hz, 2 H), 3.82 (q, *J* = 6.8 Hz, 6 H), 2.28 (dt, *J* = 7.2, 7.2 Hz, 2 H), 2.21 (t, *J* = 7.8 Hz, 2 H), 1.93 (tt, *J* = 7.0, 7.0 Hz, 2 H), 1.76 (tt, *J* = 7.2, 7.2 Hz, 2 H), 1.65-1.59 (m, 2 H), 1.22 (t, *J* = 6.5 Hz, 9 H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ 166.6, 158.5, 143.3, 137.7, 133.1, 131.5, 129.4, 120.1, 120.0, 115.2, 113.1, 68.0, 64.8, 58.5, 35.6, 30.0, 28.5, 28.3, 25.1, 18.2; HRMS (ESI-TOF) calcd. for $C_{24}H_{38}O_6Si$ $[M+Na]^+$ 473.23299, found 473.23204.



(*E*)-6-(triethoxysilyl)-3,4,7,8,9,10-hexahydrobenzo[*b*][1,5]dioxacyclotetradecin-12(2*H*)-one (2a)

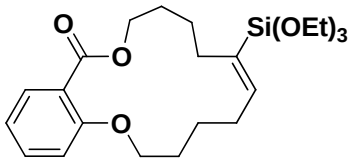
Yield 92% (pale yellow oil); IR (neat, cm^{-1}) 3076, 2972, 2927, 2735, 1705, 1602, 1582, 1491, 1453, 1387, 1302, 1252, 1166, 1128, 1080, 1025, 996, 958; ^{1}H-NMR (500 MHz, C_6D_6) δ 7.79-7.77 (m, 1 H), 7.44-7.41 (m, 1 H), 6.97 (dd, *J* = 7.5, 7.5 Hz, 1 H), 6.92 (d, *J* = 8.5 Hz, 1 H), 6.21 (t, *J* = 8.0 Hz, 1 H), 4.43 (t, *J* = 5.2 Hz, 2 H), 4.06 (t, *J* = 5.0 Hz, 2 H), 3.83 (q, *J* = 7.0 Hz, 6 H), 2.43-2.38 (m, 2 H), 2.23-2.19 (m, 2 H), 1.90-1.85 (m, 2 H), 1.83-1.78 (m, 2 H), 1.71-1.65 (m, 2 H), 1.24 (t, *J* = 6.8 Hz, 9 H); ^{13}C-NMR (125 MHz, C_6D_6) δ 168.1, 158.1, 145.2, 134.1, 132.9, 132.8, 122.1, 120.1, 112.1, 67.0, 63.5, 58.6, 30.1, 28.6, 27.7, 26.0, 25.5, 18.6; HRMS (ESI-TOF) calcd. for $C_{22}H_{34}O_6Si$ $[M+Na]^+$ 445.20169, found 445.20168.



5-(Triethoxysilyl)hex-5-en-1-yl 2-(hex-5-en-1-yloxy)benzoate (3a)

Yield 72% (colorless oil); IR (neat, cm^{-1}) 3076, 2974, 2929, 2736, 1729, 1705, 1641, 1601, 1583, 1491, 1452, 1389, 1301, 1249, 1165, 1079, 995, 958; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 7.76 (d, *J* = 8.0 Hz, 1H), 7.44-7.40 (m, 1 H), 6.96-6.93 (m, 2 H), 5.82 (ddt, *J* = 17.0, 10.5, 6.5 Hz, 1 H), 5.73-5.73 (m, 1 H), 5.65-5.65 (m, 1 H), 5.05-5.01 (m, 1 H), 4.97 (d, *J* = 10.5 Hz, 1 H), 4.30 (t, *J* = 6.8 Hz, 2 H), 4.03 (t, *J* = 6.2 Hz, 2 H), 3.82 (q, *J* = 6.8 Hz, 6 H), 2.21 (t, *J* = 7.5 Hz, 2 H), 2.13 (dt, *J* = 7.2, 7.2 Hz, 2 H), 1.84 (tt, *J* = 7.1, 7.1 Hz, 2 H), 1.76 (tt, *J* = 7.1, 7.1 Hz, 2 H), 1.64-1.57 (m, 4 H), 1.22 (t, *J* = 7.0 Hz, 9 H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ 166.6,

158.5, 143.3, 138.5, 133.1, 131.5, 129.4, 120.9, 119.9, 114.7, 113.0, 68.6, 64.8, 58.5, 35.5, 33.4, 28.6, 28.4, 25.2, 25.1, 18.2; HRMS (ESI-TOF) calcd. for $C_{25}H_{40}O_6Si$ $[M+Na]^+$ 487.24864, found 487.24889.



(*E*)-7-(Triethoxysilyl)-4,5,8,9,10,11-hexahydro-2*H*-benzo[*b*][1,5]dioxacyclopentadecin-13(3*H*)-one (4a)

Yield 60% (pale yellow oil); IR (neat, cm^{-1}) 2972, 2927, 1700, 1602, 1491, 1453, 1388, 1302, 1250, 1166, 1102, 1078, 1018, 958; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 7.75-7.74 (m, 1 H), 7.42-7.39 (m, 1 H), 6.96 (dd, *J* = 7.5, 7.5 Hz, 1 H), 6.91 (d, *J* = 8.5 Hz, 1 H), 6.23 (t, *J* = 7.5 Hz, 1 H), 4.40 (t, *J* = 5.5 Hz, 2 H), 4.07 (t, *J* = 5.0 Hz, 2 H), 3.80 (q, *J* = 6.8 Hz, 6 H), 2.27-2.21 (m, 4 H), 1.87-1.77 (m, 4 H), 1.68-1.58 (m, 4 H), 1.22 (t, *J* = 6.8 Hz, 9 H); ^{13}C-NMR (125 MHz, C_6D_6) δ 167.8, 158.2, 145.0, 134.9, 132.6, 132.2, 122.3, 120.1, 112.4, 68.1, 64.3, 58.6, 29.1, 28.9, 28.9, 28.7, 27.1, 26.9, 18.5; HRMS (ESI-TOF) calcd. for $C_{23}H_{36}O_6Si$ $[M+Na]^+$ 459.21734, found 459.21736.

C. Catalyst decomposition studies.

a) Reaction kinetics

To a round bottom flask equipped with magnetic stir bar and purged with argon, substrate **3a** (20.0 mg, 0.043 mmol) was added to 22 mL anhydrous benzene (2 mM). Next, catalyst **A** (6.8 mg, 0.009 mmol) was added to the resulting solution. The reaction was stirred at 23 °C and 50 °C respectively. Aliquots of 2 mL of the reaction mixture were taken at 0.5, 1, 1.5, 2, 3, 5, 7, 9, 12, and 24 hours, quenched with ethyl vinyl ether, concentrated and analyzed by proton NMR (**Table S3**). After 5 hours for the reaction at 50 °C, or 24 hours for the reaction at 23 °C, substrate **1a** was added to the reaction. No conversion of substrate **1a** was observed after another 18 hours for both cases.

Table S3. Conversion of the RCM reaction of substrate **3a** with catalyst **A** at different temperatures over time.

Reaction time (h)	Conversion to product (%) at 23 °C	Conversion to product (%) at 50 °C
0	0	0
0.5	1	26
1	3	35
1.5	5	38
2	7	40
3	11	42
5	18	42
7	24	-
9	29	-
12	34	-
24	36	-

b) Catalyst stability study without any substrate present

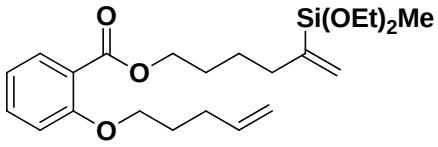
To a round bottom flask equipped with magnetic stir bar and purged with argon was added catalyst **A** (3.4 mg, 0.004 mmol) and 11 mL benzene. The reaction was carried out at 23 ^{o}C and 50 ^{o}C respectively. After 24 hours for the reaction at 23 ^{o}C or 5 hours for the reaction at 50 ^{o}C, substrate **1a** (9.7 mg, 0.021 mmol) was added to both reactions. After another 18 hours, 16% conversion of substrate **1a** was observed for the reaction at 23 ^{o}C and 72% conversion of substrate **1a** was observed for the reaction at 50 ^{o}C.

c) Catalyst stability study with simple diolefinic substrate (without siloxyl group)

To a round bottom flask, equipped with magnetic stir bar and purged with argon, was added substrate **32** (6.5 mg, 0.021 mmol) and 11 mL benzene. Next, catalyst **A** (3.4 mg, 0.004 mmol) was added to the resulting solution. The reaction was carried out at 50 ^{o}C. After 5.5 hours, substrate **1a** (9.7 mg, 0.021 mmol) was added to the reaction. No conversion of substrate **1a** was observed after another 18 hours.

D. Study of influence of silyl groups

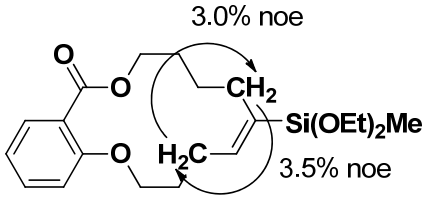
Different vinyl silane or vinyl siloxane substrates were synthesized following general procedure for hydrosilylation using the respective silanes. The RCM reaction was then performed following the general procedure for RCM.



5-(Diethoxy(methyl)silyl)hex-5-en-1-yl 2-(pent-4-en-1-yloxy)benzoate (1b, also as **19a)**

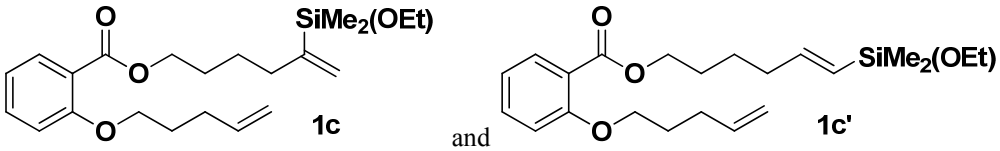
Yield 85% (colorless oil); IR (neat, cm^{-1}) 3077, 2972, 2943, 2879, 2763, 2735, 1728, 1705, 1641, 1601, 1583, 1491, 1452, 1389, 1301, 1253, 1164, 1130, 1103, 1079, 1016, 951; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 7.77 (d, *J* = 7.0 Hz, 1 H), 7.42 (dd, *J* = 7.2, 7.2 Hz, 1 H), 6.97-6.93 (m, 2 H), 5.85 (ddt, *J* = 17.2, 10.2, 7.0 Hz, 1 H), 5.69 (bs, 1 H), 5.57-5.56 (m, 1 H), 5.06 (d, *J* = 17.5 Hz, 1 H), 4.99 (d, *J* = 10.0 Hz, 1 H), 4.30 (t, *J* = 6.5 Hz, 2 H), 4.04 (t, *J* = 6.5 Hz, 2 H), 3.76 (q, *J* = 6.8 Hz, 4 H), 2.27 (dt, *J* = 7.0, 7.0 Hz, 2 H), 2.21 (t, *J* = 7.5 Hz, 2 H), 1.93 (tt, *J* = 6.9, 6.9 Hz, 2 H), 1.76 (tt, *J* = 7.2, 7.2 Hz, 2 H), 1.60 (tt, *J* = 7.6, 7.6 Hz, 2 H), 1.21 (t, *J* = 7.0 Hz, 6 H), 0.19 (s, 3 H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ 166.6, 158.4, 147.0, 137.7, 133.1, 131.5, 127.7, 120.8,

120.0, 115.2, 113.0, 67.9, 64.8, 58.2, 35.1, 30.0, 28.5, 28.3, 25.1, 18.3, -4.6; HRMS (ESI-TOF) calcd. for $C_{23}H_{36}O_5Si$ $[M+H]^+$ 421.24048, found 421.24067.

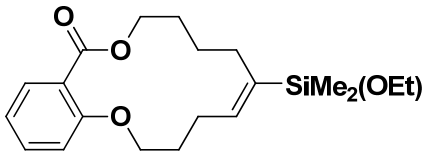


(*E*)-6-(Diethoxy(methyl)silyl)-3,4,7,8,9,10-hexahydrobenzo[*b*][1,5]dioxacyclotetradecin-12(2*H*)-one (2b, also as **19)**

Yield 95% (pale yellow oil); IR (neat, cm^{-1}) 3076, 2970, 2927, 2873, 1705, 1602, 1582, 1491, 1453, 1386, 1356, 1303, 1253, 1165, 1129, 1103, 1079, 1051, 1024, 995; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 7.79-7.77 (m, 1 H), 7.44-7.41 (m, 1 H), 6.97 (dd, *J* = 7.2, 7.2 Hz, 1 H), 6.92 (d, *J* = 8.0 Hz, 1 H), 6.11 (t, *J* = 8.0 Hz, 1 H), 4.43 (t, *J* = 5.2 Hz, 2 H), 4.06 (t, *J* = 5.0 Hz, 2 H), 3.77 (q, *J* = 7.0 Hz, 4 H), 2.40 (dt, *J* = 6.0, 6.0 Hz, 2 H), 2.21-2.18 (m, 2 H), 1.90-1.84 (m, 2 H), 1.83-1.78 (m, 2H), 1.69-1.62 (m, 2 H), 1.23 (t, *J* = 7.2 Hz, 6 H), 0.19 (s, 3 H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ 168.5, 157.6, 143.5, 136.8, 133.1, 132.2, 121.1, 120.1, 112.0, 67.4, 63.8, 58.2, 29.9, 28.4, 26.9, 25.7, 25.3, 18.3, -4.9; HRMS (ESI-TOF) calcd. for $C_{21}H_{32}O_5Si$ $[M+H]^+$ 393.20918, found 393.20943.

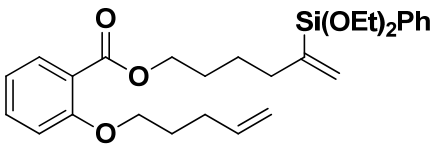


Hydrosilylation reaction gave rise to a 14.4:1 mixture of two regioisomers with the desired regioisomer **1c** being the major one. Yield 84% (colorless oil).



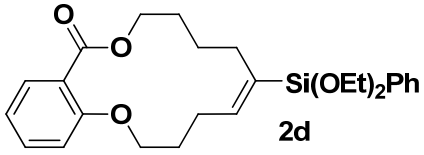
(*E*)-6-(Ethoxydimethylsilyl)-3,4,7,8,9,10-hexahydrobenzo[*b*][1,5]dioxacyclotetradecin-12(2*H*)-one (2c)

Yield 81% (pale yellow oil); IR (neat, cm^{-1}) 2959, 2926, 2865, 1704, 1602, 1491, 1453, 1386, 1303, 1250, 1164, 1131, 1102, 1080, 1049, 1023, 993; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 7.79-7.77 (m, 1 H), 7.44-7.41 (m, 1 H), 6.97 (dd, *J* = 7.5, 7.5 Hz, 1 H), 6.92 (d, *J* = 8.5 Hz, 1 H), 5.97 (t, *J* = 8.0 Hz, 1 H), 4.43 (t, *J* = 5.2 Hz, 2 H), 4.06 (t, *J* = 5.0 Hz, 2 H), 3.65 (q, *J* = 7.0 Hz, 2 H), 2.42-2.36 (m, 2 H), 2.22-2.18 (m, 2 H), 1.89-1.84 (m, 2 H), 1.83-1.78 (m, 2 H), 1.67-1.61 (m, 2 H), 1.19 (t, *J* = 7.0 Hz, 3 H), 0.19 (s, 6 H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ 168.5, 157.7, 141.7, 139.8, 133.1, 132.3, 121.1, 120.1, 112.1, 67.5, 63.8, 58.4, 30.1, 28.5, 27.2, 25.8, 25.5, 18.5, -2.4; HRMS (ESI-TOF) calcd. for $C_{20}H_{30}O_4Si$ $[M+Na]^+$ 385.18056, found 385.19580.



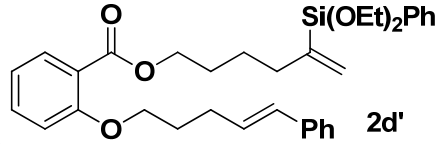
5-(Diethoxy(phenyl)silyl)hex-5-en-1-yl 2-(pent-4-en-1-yloxy)benzoate (1d)

Yield 91% (colorless oil); IR (neat, cm^{-1}) 3071, 2973, 2940, 2881, 1728, 1704, 1641, 1601, 1583, 1491, 1469, 1452, 1430, 1389, 1301, 1251, 1164, 1119, 1101, 1079, 1016, 952; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 7.76-7.74 (m, 1 H), 7.64-7.62 (m, 2 H), 7.44-7.33 (m, 4 H), 6.96-6.93 (m, 2 H), 5.88-5.79 (m, 2 H), 5.67-5.66 (m, 1 H), 5.06-5.03 (m, 1 H), 4.98 (d, *J* = 10.0 Hz, 1 H), 4.23 (t, *J* = 6.5 Hz, 2 H), 4.03 (t, *J* = 6.5 Hz, 2 H), 3.81 (q, *J* = 7.0 Hz, 4 H), 2.26 (dt, *J* = 7.2, 7.2 Hz, 2 H), 2.22 (t, *J* = 8.0 Hz, 2 H), 1.91 (tt, *J* = 6.9, 6.9 Hz, 2 H), 1.70 (tt, *J* = 7.2, 7.2 Hz, 2 H), 1.56 (tt, *J* = 7.6, 7.6 Hz, 2 H), 1.23 (t, *J* = 7.2 Hz, 6 H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ 166.6, 158.4, 145.5, 137.7, 134.6, 133.3, 133.1, 131.5, 130.0, 129.4, 127.7, 120.8, 120.0, 115.2, 113.1, 68.0, 64.8, 58.7, 35.2, 30.0, 28.4, 28.3, 25.1, 18.3; HRMS (ESI-TOF) calcd. for $C_{28}H_{38}O_5Si$ $[M+Na]^+$ 505.23807, found 505.24127.



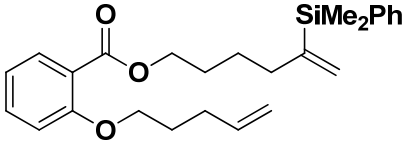
2d

and



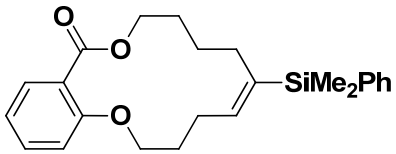
2d'

RCM reaction of compound **1d** gave rise to an inseparable mixture of product **2d** and styrene derivative **2d'** as well as acyclic dimer and unreacted starting material. The NMR yield was calculated to be 69% based on analysis of crude ^{1}H NMR spectrum.



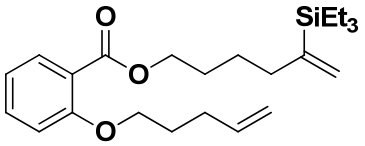
5-(Dimethyl(phenyl)silyl)hex-5-en-1-yl 2-(pent-4-en-1-yloxy)benzoate (1e)

Yield 93% (colorless oil); IR (neat, cm^{-1}) 3069, 2949, 1728, 1641, 1601, 1491, 1452, 1430, 1387, 1302, 1251, 1164, 1133, 1078, 1050, 1015; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 7.75-7.73 (m, 1 H), 7.51-7.49 (m, 2 H), 7.44-7.40 (m,1 H), 7.34-7.32 (m, 3 H), 6.97-6.93 (m, 2 H), 5.84 (ddt, *J* = 17.0, 10.0, 6.8 Hz, 1 H), 5.70-5.69 (m, 1 H), 5.42-5.42 (m, 1 H), 5.06-5.03 (m, 1 H), 4.99 (d, *J* = 10.5 Hz, 1 H), 4.21 (t, *J* = 7.0 Hz, 2 H), 4.03 (t, *J* = 6.5 Hz, 2 H), 2.26 (dt, *J* = 7.2, 7.2 Hz, 2 H), 2.17 (t, *J* = 7.5 Hz, 2 H), 1.91 (tt, *J* = 6.9, 6.9 Hz, 2 H), 1.68 (tt, *J* = 7.1, 7.1 Hz, 2 H), 1.52-1.46 (m, 2 H), 0.36 (s, 6 H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ 166.6, 158.4, 149.8, 138.2, 137.7, 133.8, 133.1, 131.5, 128.9, 127.7, 126.0, 120.8, 120.0, 115.2, 113.0, 67.9, 64.7, 35.4, 30.0, 28.4, 28.3, 25.1, -3.0; HRMS (ESI-TOF) calcd. for $C_{26}H_{34}O_3Si$ $[M+H]^+$ 423.23500, found 423.23601.



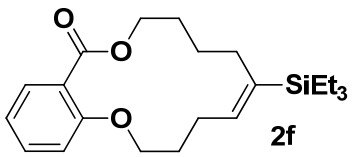
(*E*)-6-(Dimethyl(phenyl)silyl)-3,4,7,8,9,10-hexahydrobenzo[*b*][1,5]dioxacyclotetradecin-12(2*H*)-one (2e)

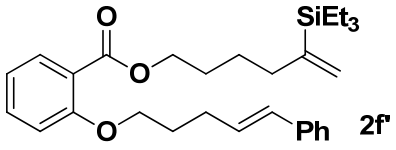
RCM reaction of the previous compound (**1e**) gave rise to an inseparable mixture of product and styrene derivative together with unreacted starting material. The NMR yield was calculated to be 71% based on analysis of crude ^{1}H NMR spectrum. After the first column chromatography to get rid of the unreacted starting materials, the mixture of product and styrene derivative was subjected to HPLC separation that gave rise to 25 mg pure product (54% yield) as pale yellow oil. HPLC condtions: compound was dissolved in a 1 ml volume of DMSO. The separation was executed on an XBridge 19x100 mm 5 µm columns at a flow rate of 44 ml/min. Aqueous mobile phase A consisted of 0.1% formic acid in water, and organic mobile phase B was 0.1% formic acid in acetonitrile. Purification fractions were immediately frozen at -50°C and lyophilized for 24hrs using the Genesis Virtis. After lyophilization the compound was transferred to a preweighed vial using dichloromethane. IR (neat, cm^{-1}) 3067, 2954, 2860, 1703, 1602, 1490, 1452, 1429, 1383, 1302, 1250, 1165, 1131, 1050, 1023, 992; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 7.78-7.76 (m, 1 H), 7.53-7.51 (m, 2 H), 7.44-7.40 (m, 1 H), 7.36-7.33 (m, 3 H), 6.97 (dd, *J* = 7.5, 7.5 Hz, 1 H), 6.92 (d, *J* = 7.5 Hz, 1 H), 5.91 (t, *J* = 8.0 Hz, 1 H), 4.38 (t, *J* = 5.2 Hz, 2 H), 4.06 (t, *J* = 5.0 Hz, 2 H), 2.42-2.37 (m, 2 H), 2.18-2.15 (m, 2 H), 1.89-1.84 (m, 2 H), 1.73-1.68 (m, 2 H), 1.59-1.52 (m, 2 H), 0.35 (s, 6 H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ 168.5, 157.7, 141.7, 139.4, 138.8, 134.0, 133.1, 132.2, 128.8, 127.7, 121.1, 120.0, 112.1, 67.5, 63.8, 30.1, 28.4, 28.2, 25.9, 25.7, -3.1; HRMS (ESI-TOF) calcd. for $C_{24}H_{30}O_3Si$ $[M+Na]^+$ 417.18564, found 417.18593.



5-(Triethylsilyl)hex-5-en-1-yl 2-(pent-4-en-1-yloxy)benzoate (1f)

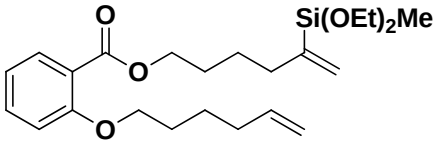
Yield 38% (colorless oil); IR (neat, cm^{-1}) 3077, 3048, 2951, 2911, 2875, 1729, 1704, 1641, 1601, 1582, 1491, 1453, 1416, 1385, 1301, 1250, 1164, 1133, 1078, 1050, 1013; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 7.78-7.76 (m, 1 H), 7.44-7.41 (m, 1 H), 6.97-6.94 (m, 2 H), 5.85 (ddt, *J* = 17.0, 10.0, 6.8 Hz, 1 H), 5.65-5.64 (m, 1 H), 5.32-5.31 (m, 1 H), 5.08-5.04 (m, 1 H), 4.99 (d, *J* = 10.0 Hz, 1 H), 4.30 (t, *J* = 6.5 Hz, 2 H), 4.04 (t, *J* = 6.5 Hz, 2 H), 2.27 (dt, *J* = 7.2, 7.2 Hz, 2 H), 2.14 (t, *J* = 7.8 Hz, 2 H), 1.93 (tt, *J* = 7.0, 7.0 Hz, 2 H), 1.76 (tt, *J* = 7.1, 7.1 Hz, 2 H), 1.60-1.54 (m, 2 H), 0.92 (t, *J* = 8.0 Hz, 6 H), 0.60 (q, *J* = 8.0 Hz, 9 H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ 166.6, 158.4, 148.5, 137.7, 133.1, 131.5, 125.3, 120.8, 120.0, 115.2, 113.1, 68.0, 64.8, 35.7, 30.0, 28.6, 28.3, 25.1, 7.3, 2.9; HRMS (ESI-TOF) calcd. for $C_{24}H_{38}O_3Si$ $[M+H]^+$ 403.26630, found 403.26630.



2f and 

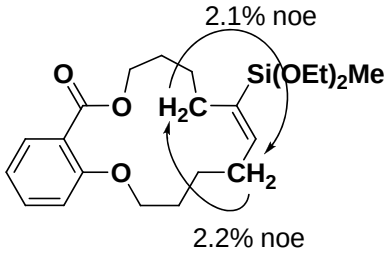
2f'

RCM reaction of compound **1f** gave rise to an inseparable mixture of product **2f** and styrene derivative **2f'**. Unreacted starting material and acyclic dimer were also observed. The NMR yield was calculated to be 10% based on analysis of crude ^{1}H NMR spectrum.



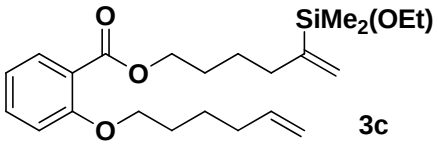
5-(Diethoxy(methyl)silyl)hex-5-en-1-yl 2-(hex-5-en-1-yloxy)benzoate (3b, also as **20a)**

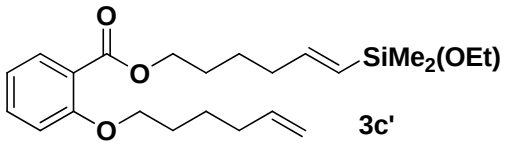
Yield 79% (colorless oil); IR (neat, cm^{-1}) 3076, 2972, 2940, 1729, 1705, 1641, 1601, 1491, 1452, 1389, 1301, 1252, 1164, 1103, 1079, 996, 951; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 7.78-7.76 (m, 1 H), 7.44-7.40 (m, 1 H), 6.96-6.93 (m, 2 H), 5.82 (ddt, *J* = 17.0, 10.5 , 6.5 Hz, 1 H), 5.69-5.69 (m, 1 H), 5.57-5.56 (m, 1 H), 5.05-5.01 (m, 1 H), 4.97 (d, *J* = 10.5 Hz, 1 H), 4.30 (t, *J* = 6.8 Hz, 2 H), 4.03 (t, *J* = 6.5 Hz, 2 H), 3.76 (q, *J* = 7.0 Hz, 4 H), 2.21 (t, *J* = 7.5 Hz, 2 H), 2.13 (dt, *J* = 7.2, 7.2 Hz, 2 H), 1.84 (tt, *J* = 7.1, 7.1 Hz, 2 H), 1.76 (tt, *J* = 7.2, 7.2 Hz, 2 H), 1.63-1.57 (m, 4 H), 1.21 (t, *J* = 7.0 Hz, 6 H), 0.19 (s, 3 H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ 166.6, 158.5, 147.1, 138.5, 133.1, 131.5, 127.6, 120.8, 119.9, 114.7, 113.0, 68.6, 64.8, 58.2, 35.1, 33.4, 28.6, 28.5, 25.2, 25.1, 18.3, -4.6; HRMS (ESI-TOF) calcd. for $C_{24}H_{38}O_5Si$ $[M+Na]^+$ 457.23807, found 457.24010.



(*E*)-7-(Diethoxy(methyl)silyl)-4,5,8,9,10,11-hexahydro-2*H*-benzo[*b*][1,5]dioxacyclopentadecin-13(3*H*)-one (4b, also as **20)**

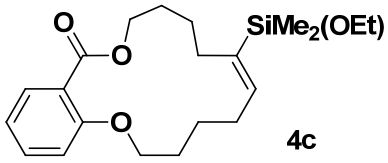
Yield 76% (pale yellow oil); IR (neat, cm^{-1}) 2969, 2928, 1700, 1602, 1491, 1452, 1387, 1302, 1251, 1165, 1130, 1103, 1078, 1016, 952; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 7.76-7.74 (m, 1 H), 7.42-7.39 (m, 1 H), 6.96 (dd, *J* = 7.5, 7.5 Hz, 1 H), 6.92 (d, *J* = 7.5 Hz, 1 H), 6.13 (t, *J* = 7.5 Hz, 1 H), 4.40 (t, *J* = 5.5 Hz, 2 H), 4.08 (t, *J* = 5.0 Hz, 2 H), 3.74 (q, *J* = 6.8 Hz, 4 H), 2.25-2.21 (m, 4 H), 1.87-1.76 (m, 4 H), 1.68-1.56 (m, 4 H), 1.21 (t, *J* = 6.8 Hz, 6 H), 0.17 (s, 3 H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ 168.2, 157.7, 143.5, 137.5, 132.9, 131.7, 121.2, 120.0, 112.3, 68.3, 64.6, 58.1, 28.9, 28.8, 28.6, 28.0, 26.9, 26.7, 18.3, -4.6; HRMS (ESI-TOF) calcd. for $C_{22}H_{34}O_5Si$ $[M+Na]^+$ 429.20677, found 429.20692.

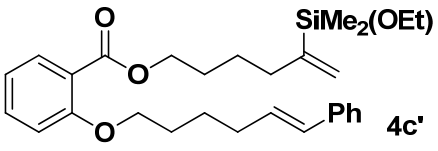


3c and 

3c'

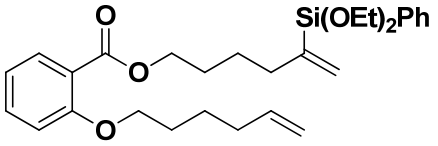
Hydrosilylation reaction gave rise to a 14.3:1 mixture of two regioisomers with the desired regio isomer **3c** being the major one. Yield 89% (colorless oil).



4c and 

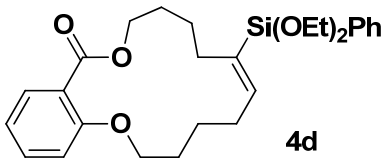
4c'

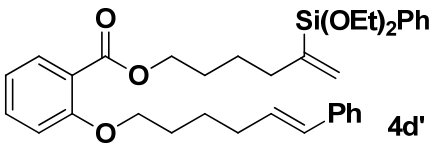
RCM reaction of the mixture **3c** and **3c'** gave rise to an inseparable mixture of product **4c** and styrene derivative **4c'** as well as acyclic dimer and unreacted starting material. The NMR yield was calculated to be 62% based on analysis of crude ^{1}H NMR spectrum.



5-(Diethoxy(phenyl)silyl)hex-5-en-1-yl 2-(hex-5-en-1-yloxy)benzoate (3d)

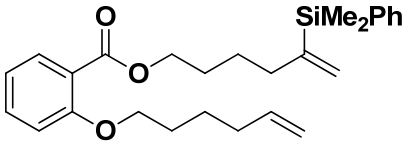
Yield 76% (colorless oil); IR (neat, cm^{-1}) 3071, 2973, 2938, 1729, 1704, 1640, 1601, 1583, 1491, 1470, 1453, 1430, 1389, 1301, 1250, 1164, 1119, 1102, 1079, 997, 952; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 7.75-7.73 (m, 1 H), 7.64-7.62 (m, 2 H), 7.44-7.33 (m, 4 H), 6.96-6.93 (m, 2 H), 5.85-5.77 (m, 2 H), 5.67-5.66 (m, 1 H), 5.04-5.00 (m, 1 H), 4.96 (d, *J* = 10.0 Hz, 1 H), 4.22 (t, *J* = 7.0 Hz, 2 H), 4.02 (t, *J* = 6.2 Hz, 2 H), 3.81 (q, *J* = 7.0 Hz, 4 H), 2.22 (t, *J* = 7.8 Hz, 2 H), 2.11 (dt, *J* = 5.5, 5.5 Hz, 2 H), 1.83 (tt, *J* = 7.0, 7.0 Hz, 2 H), 1.70 (tt, *J* = 7.2, 7.2 Hz, 2 H), 1.62-1.52 (m, 4 H), 1.23 (t, *J* = 7.2 Hz, 6 H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ 166.6, 158.5, 145.5, 138.5, 134.6, 133.3, 133.1, 131.5, 130.0, 129.4, 127.7, 120.9, 119.9, 114.7, 113.0, 68.6, 64.8, 58.7, 35.2, 33.4, 28.6, 28.4, 25.2, 25.1, 18.3; HRMS (ESI-TOF) calcd. for $C_{29}H_{40}O_5Si$ $[M+Na]^+$ 519.25372, found 519.25541.



4d and 

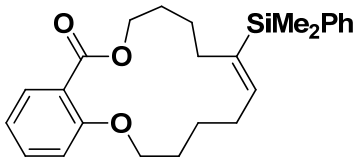
4d'

RCM reaction of compound **3d** gave rise to an inseparable mixture of product **4d** and styrene derivative **4d'** as well as acyclic dimers and unreacted starting material. The NMR yield was calculated to be 35% based on analysis of crude ^{1}H NMR spectrum.



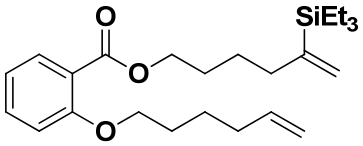
5-(Dimethyl(phenyl)silyl)hex-5-en-1-yl 2-(hex-5-en-1-yloxy)benzoate (3e)

Yield 74% (colorless oil); IR (neat, cm^{-1}) 3069, 2945, 1728, 1703, 1641, 1601, 1491, 1452, 1430, 1388, 1301, 1250, 1164, 1133, 1077, 1049, 996; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 7.75-7.73 (m, 1 H), 7.51-7.49 (m, 2 H), 7.44-7.40 (m, 1 H), 7.34-7.31 (m, 3 H), 6.96-6.93 (m, 2 H), 5.81 (ddt, *J* = 16.8, 10.2, 6.5 Hz, 1 H), 5.70-5.69 (m, 1 H), 5.43-5.42 (m, 1 H), 5.04-5.00 (m, 1 H), 4.96 (d, *J* = 9.5 Hz, 1 H), 4.21 (t, *J* = 6.5 Hz, 2 H), 4.02 (t, *J* = 6.2 Hz, 2 H), 2.17 (t, *J* = 7.8 Hz, 2 H), 2.11 (dt, *J* = 7.2, 7.2 Hz, 2 H), 1.83 (tt, *J* = 7.0, 7.0 Hz, 2 H), 1.67 (tt, *J* = 7.0, 7.0 Hz, 2 H), 1.58 (tt, *J* = 7.5, 7.5 Hz, 2 H), 1.52-1.46 (m, 2 H), 0.37 (s, 6 H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ 166.6, 158.5, 149.9, 138.5, 138.2, 133.8, 133.1, 131.5, 128.9, 127.7, 126.0, 120.8, 119.9, 114.7, 113.0, 68.6, 64.7, 35.4, 33.4, 28.6, 28.4, 25.2, 25.1, -3.0; HRMS (ESI-TOF) calcd. for $C_{27}H_{36}O_3Si$ $[M+H]^+$ 437.25065, found 437.25057.



(*E*)-7-(dimethyl(phenyl)silyl)-4,5,8,9,10,11-hexahydro-2*H*-benzo[*b*][1,5]dioxacyclopentadecin-13(3*H*)-one (4e)

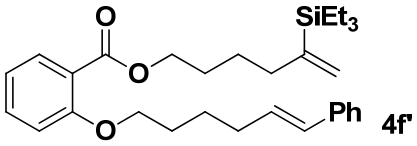
Yield 32% (pale yellow oil); IR (neat, cm^{-1}) 3067, 2952, 2859, 1698, 1601, 1490, 1452, 1429, 1383, 1302, 1249, 1165, 1132, 1108, 1049, 1015, 963; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 7.77-7.75 (m, 1 H), 7.51-7.49 (m, 2 H), 7.43-7.39 (m, 1 H), 7.34-7.33 (m, 3 H), 6.95 (dd, *J* = 7.8, 7.8 Hz, 1 H), 6.92 (d, *J* = 9.0 Hz, 1 H), 5.94 (t, *J* = 7.2 Hz, 1 H), 4.34 (t, *J* = 5.5 Hz, 2 H), 4.08 (t, *J* = 5.2 Hz, 2 H), 2.25-2.18 (m, 4 H), 1.87-1.82 (m, 2 H), 1.70-1.61 (m, 4 H), 1.46 (tt, *J* = 7.9 Hz, 2 H), 0.34 (s, 6 H); ^{13}C-NMR (125 MHz, C_6D_6) δ 168.2, 157.7, 142.2, 139.8, 139.0, 134.0, 133.0, 131.8, 128.8, 127.6, 121.1, 120.0, 112.3, 68.3, 64.6, 29.0, 28.9, 28.9, 28.8, 27.0, 27.0, -2.6; HRMS (ESI-TOF) calcd. for $C_{25}H_{32}O_3Si$ $[M+Na]^+$ 431.20129, found 431.20247.



5-(Triethylsilyl)hex-5-en-1-yl 2-(hex-5-en-1-yloxy)benzoate (3f)

Yield 56% (colorless oil); IR (neat, cm^{-1}) 3076, 3047, 2951, 2911, 2874, 1730, 1704, 1641, 1601, 1583, 1491, 1453, 1416, 1385, 1301, 1249, 1164, 1132, 1077, 1049, 1017, 959; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 7.78-7.76 (m, 1 H), 7.44-7.41 (m, 1 H), 6.97-6.94 (m, 2 H), 5.82 (ddt, *J* = 17.0, 10.0, 6.8 Hz, 1 H), 5.65-5.64 (m, 1 H), 5.32-5.31 (m, 1 H), 5.05-5.01 (m, 1 H), 4.98-4.96 (m, 1 H), 4.30 (t, *J* = 6.8 Hz, 2 H), 4.03 (t, *J* = 6.8 Hz, 2 H),

2.16-2.11 (m, 4 H), 1.85 (tt, *J* = 7.0, 7.0 Hz, 2 H), 1.76 (tt, *J* = 7.0, 7.0 Hz, 2 H), 1.63-1.54 (m, 4 H), 0.92 (t, *J* = 8.0 Hz, 6 H), 0.60 (q, *J* = 8.0 Hz, 9 H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ 166.7, 158.5, 148.6, 138.5, 133.1, 131.5, 125.3, 120.8, 120.0, 114.7, 113.1, 68.6, 64.8, 35.7, 33.4, 28.6, 28.6, 25.2, 25.1, 7.3, 2.9; HRMS (ESI-TOF) calcd. for $C_{25}H_{40}O_3Si$ $[M+Na]^+$ 439.26389, found 439.26459.

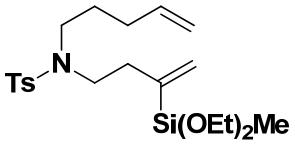


4f'

RCM reaction of compound **3f** gave rise to less than 2% product based on analysis of crude ^{1}H NMR spectrum. Styrene derivative **4f'**, unreacted starting material and acyclic dimer were observed.

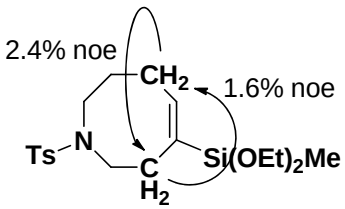
E. RCM of various vinylsiloxane substrates and protodesilylation of the alkenyl siloxane products.

Note on compound numbering: ring-closed alkenyl siloxanes are designated as the parent compound and numerated with just a number (ie. **5**), the **a**cyclic precursors are designated with an '**a**' following the parent compound number (ie. **5a**) and the desilylated cyclic compounds are designated with a '**b**' following the parent compound number (ie. **5b**).



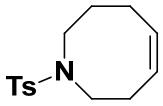
***N*-(3-(Diethoxy(methyl)silyl)but-3-enyl)-4-methyl-*N*-(pent-4-enyl)benzenesulfonamide (5a)**

Yield 70% (colorless oil); IR (neat, cm^{-1}) 3051, 2974, 2926, 2878, 1641, 1599, 1494, 1444, 1390, 1342, 1306, 1258, 1159, 1103, 1079, 955; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 7.70 (d, *J* = 7.8 Hz, 2 H), 7.28 (d, *J* = 7.8 Hz, 2 H), 5.77 (ddt, *J* = 17.2, 10.2, 6.5 Hz, 1 H), 5.70 (d, *J* = 1.2 Hz, 1 H), 5.58 (d, *J* = 1.2 Hz, 1 H), 5.01 (d, *J* = 17.2 Hz, 1 H), 4.97 (d, *J* = 10.2 Hz, 1 H), 3.74 (q, *J* = 7.0 Hz, 4 H), 3.22-3.19 (m, 2 H), 3.14 (t, *J* = 7.8 Hz, 2 H), 2.41 (s, 3 H), 2.35 (t, *J* = 8.0 Hz, 2 H), 2.06 (dt, *J* = 7.0, 7.0 Hz, 2 H), 1.69-1.63 (m, 2 H), 1.20 (t, *J* = 7.0 Hz, 6 H), 0.18 (s, 3 H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ 144.1, 142.9, 137.5, 137.2, 130.0, 129.5, 127.1, 115.2, 58.3, 48.0, 47.8, 35.1, 30.8, 27.7, 21.4, 18.3, -4.9; HRMS (ESI-TOF) calcd. for $C_{21}H_{35}NO_4SSi$ $[M+Na]^+$ 448.19483, found 448.19573.



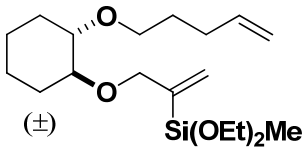
(*E*)-6-(Diethoxy(methyl)silyl)-1-tosyl-1,2,3,4,7,8-hexahydroazocine (5)

Yield 75% (pale yellow oil); IR (neat, cm^{-1}) 2972, 2926, 1615, 1455, 1389, 1338, 1292, 1257, 1158, 1079, 1050, 1017, 995; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 7.68 (d, *J* = 8.2 Hz, 2 H), 7.28 (d, *J* = 8.2 Hz, 2 H), 6.28 (t, *J* = 8.2 Hz, 1 H), 3.72 (q, *J* = 7.2 Hz, 4 H), 3.15 (bs, 2 H), 3.02 (t, *J* = 5.5 Hz, 2 H), 2.44 (t, *J* = 5.0 Hz, 2 H), 2.41 (s, 3 H), 2.32 (dt, *J* = 6.8, 6.8 Hz, 2 H), 1.79-1.74 (m, 2 H), 1.19 (t, *J* = 7.0 Hz, 6 H), 0.15 (s, 3 H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ 143.8, 142.8, 137.0, 137.0, 129.6, 126.8, 58.2, 50.8, 48.2, 29.2, 29.1, 24.8, 21.4, 18.3, -4.9; HRMS (ESI-TOF) calcd. for $C_{19}H_{31}NO_4SSi$ $[M+H]^+$ 398.18159, found 398.27160.



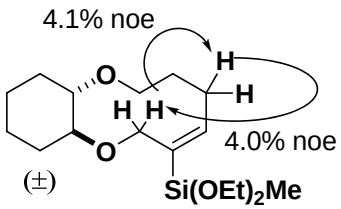
(*Z*)-1-tosyl-1,2,3,4,7,8-hexahydroazocine (5b)

Yield 72% (colorless oil); IR (neat, cm^{-1}) 3018, 2933, 2858, 1598, 1494, 1456, 1369, 1333, 1304, 1289, 1157, 1112, 1091, 1060, 1038, 991; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 7.67 (d, *J* = 8.5 Hz, 2 H), 7.27 (d, *J* = 8.5 Hz, 2 H), 5.74-5.66 (m, 2 H), 3.14 (t, *J* = 5.0 Hz, 2 H), 3.08 (t, *J* = 5.5 Hz, 2 H), 2.40 (s, 3 H), 2.31-2.28 (m, 2 H), 2.22 (dt, *J* = 6.9, 6.9 Hz, 2 H), 1.76-1.72 (m, 2 H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ 142.9, 136.9, 131.3, 129.5, 128.2, 126.8, 50.8, 48.2, 29.4, 28.1, 23.3, 21.4; HRMS (ESI-TOF) calcd. for $C_{14}H_{19}NO_2S$ $[M+H]^+$ 266.12093, found 266.12097.



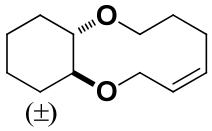
Diethoxy(methyl)(3-((1*S*,2*S*)-2-(pent-4-enyloxy)cyclohexyloxy)prop-1-en-2-yl)silane and its enantiomer (6a)

Yield 62% (colorless oil); IR (neat, cm^{-1}) 3077, 2974, 2934, 2865, 1641, 1449, 1390, 1366, 1295, 1257, 1164, 1104, 1083, 992, 951; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 6.00-5.98 (m, 1 H), 5.82 (dddd, *J* = 17.0, 10.0, 6.5, 6.5 Hz, 1 H), 5.64-5.63 (m, 1 H), 5.03-4.99 (m, 1 H), 4.96-4.93 (m, 1 H), 4.24-4.23 (m, 2 H), 3.77 (q, *J* = 7.0 Hz, 4 H), 3.59-3.50 (m, 2 H), 3.26-3.18 (m, 2 H), 2.14-2.10 (m, 2 H), 1.97-1.94 (m, 2 H), 1.68-1.62 (m, 4 H), 1.35-1.19 (m, 10 H), 0.22 (s, 3 H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ 144.8, 138.5, 126.9, 114.5, 81.1, 80.6, 72.4, 69.1, 58.3, 30.4, 29.8, 29.8, 29.5, 23.3, 23.3, 18.3, -4.3; HRMS (ESI-TOF) calcd. for $C_{19}H_{36}O_4Si$ $[M+Na]^+$ 379.22751, found 379.22440.



((8a*S*,12a*S*,*E*)-2,5,6,7,8a,9,10,11,12,12a-Decahydrobenzo[*b*][1,4]dioxecin-3-yl)diethoxy(methyl)silane and its enantiomer (6)

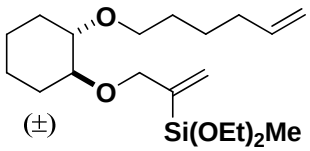
Yield 87% (pale yellow oil); IR (neat, cm^{-1}) 2972, 2932, 2862, 1615, 1451, 1390, 1364, 1256, 1165, 1113, 1082, 1009, 952; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 6.22 (dd, *J* = 10.2, 6.8 Hz, 1 H), 4.33 (d, *J* = 10.5 Hz, 1 H), 4.26 (d, *J* = 10.5 Hz, 1 H), 3.81-3.76 (m, 4 H), 3.72-3.68 (m, 1 H), 3.62-3.57 (m, 1 H), 3.22-3.17 (m, 1 H), 3.02-2.97 (m, 1 H), 2.68-2.60 (m, 1 H), 2.18-2.12 (m, 1 H), 2.00-1.98 (m, 1 H), 1.94-1.92 (m, 1 H), 1.90-1.82 (m, 1 H), 1.66-1.65 (m, 2 H), 1.54-1.48 (m, 1 H), 1.26-1.12 (m, 10 H), 0.21 (s, 3 H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ 146.8, 136.6, 83.2, 83.1, 67.6, 66.7, 58.3, 31.8, 31.8, 28.6, 25.1, 24.7, 24.5, 18.3, -4.4; HRMS (ESI-TOF) calcd. for $C_{17}H_{32}O_4Si$ $[M+Na]^+$ 351.19621, found 351.19793.



(±)

(8a*S*,12a*S*,*Z*)-2,3,4,7,8a,9,10,11,12,12a-decahydrobenzo[*b*][1,4]dioxecine and its enantiomer (6b)

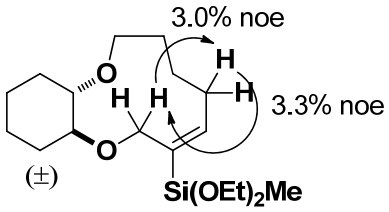
Yield 86% (colorless oil); IR (neat, cm^{-1}) 3012, 2930, 2858, 1451, 1360, 1315, 1239, 1206, 1117, 1086, 1051, 1026, 970; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 5.79 (ddd, *J* = 10.0, 10.0, 5.0 Hz, 1 H), 5.55 (ddd, *J* = 10.7, 10.7, 6.5 Hz, 1 H), 4.31 (dd, *J* = 10.5, 10.5 Hz, 1 H), 4.19 (dd, *J* = 10.7, 5.2 Hz, 1 H), 3.70-3.67 (m, 1 H), 3.53-3.49 (m, 1 H), 3.20-3.15 (m, 1 H), 2.96-2.92 (m, 1 H), 2.65-2.59 (m, 1 H), 1.94-1.80 (m, 4 H), 1.64-1.63 (m, 2 H), 1.43-1.37 (m, 1 H), 1.22-1.10 (m, 4 H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ 131.7, 128.8, 84.7, 83.2, 67.3, 66.9, 32.2, 31.6, 28.1, 24.6, 24.5, 22.6; HRMS (ESI-TOF) calcd. for $C_{12}H_{20}O_2$ $[M+H]^+$ 197.15361, found 197.15343.



(±)

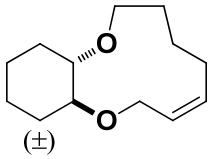
Diethoxy(3-((1*S*,2*S*)-2-(hex-5-enyloxy)cyclohexyloxy)prop-1-en-2-yl)(methyl)silane and its enantiomer (7a)

Yield 64% (colorless oil); IR (neat, cm^{-1}) 3076, 2974, 2934, 2863, 1641, 1451, 1390, 1366, 1295, 1257, 1164, 1104, 1083, 993, 951; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 5.99-5.98 (m, 1 H), 5.80 (dddd, *J* = 17.0, 10.5, 7.0, 7.0 Hz, 1 H), 5.64-5.62 (m, 1 H), 5.01-4.98 (m, 1 H), 4.94-4.92 (m, 1 H), 4.23-4.23 (m, 2 H), 3.77 (q, *J* = 7.0 Hz, 4 H), 3.58-3.49 (m, 2 H), 3.25-3.18 (m, 2 H), 2.08-2.04 (m, 2 H), 1.97-1.93 (m, 2 H), 1.65-1.62 (m, 2 H), 1.60-1.54 (m, 2 H), 1.48-1.42 (m, 2 H), 1.35-1.19 (m, 10 H), 0.22 (s, 3 H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ 144.8, 138.8, 126.9, 114.4, 81.1, 80.6, 72.4, 69.6, 58.3, 33.6, 29.8, 29.8, 25.6, 23.3, 23.3, 18.3, -4.3; HRMS (ESI-TOF) calcd. for $C_{20}H_{38}O_4Si$ $[M+Na]^+$ 393.24316, found 393.24372.



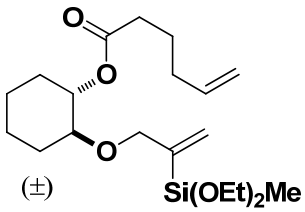
((9a*S*,13a*S*,*E*)-5,6,7,8,9a,10,11,12,13,13a-Decahydro-2*H*-benzo[*b*][1,4]dioxacycloundecin-3-yl)diethoxy(methyl)silane and its enantiomer (7)

Yield 36% (pale yellow oil); IR (neat, cm^{-1}) 2971, 2929, 2860, 1618, 1450, 1389, 1371, 1255, 1191, 1165, 1104, 1079, 1044, 1003, 951; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 6.23 (dd, *J* = 10.0, 6.0 Hz, 1 H), 4.24 (d, *J* = 10.2 Hz, 1 H), 4.14 (d, *J* = 10.2 Hz, 1 H), 3.85-3.82 (m, 1 H), 3.77 (q, *J* = 7.0 Hz, 4 H), 3.54-3.51 (m, 1 H), 3.14-3.09 (m, 1 H), 3.00-2.97 (m, 1 H), 2.66-2.58 (m, 1 H), 2.24-2.18 (m, 1 H), 2.10-2.08 (m, 1 H), 2.00-1.98 (m, 1 H), 1.75-1.66 (m, 4 H), 1.59-1.52 (m, 1 H), 1.44-1.39 (m, 1 H), 1.23-1.08 (m, 10 H), 0.19 (s, 3 H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ 149.7, 133.4, 84.1, 82.2, 71.9, 66.2, 58.2, 31.5, 31.1, 28.4, 27.2, 27.0, 24.5, 24.3, 18.3, -4.6; HRMS (ESI-TOF) calcd. for $C_{18}H_{34}O_4Si$ $[M+Na]^+$ 365.21186, found 365.21302.



(9a*S*,13a*S*,*Z*)-3,4,5,8,9a,10,11,12,13,13a-decahydro-2*H*-benzo[*b*][1,4]dioxacycloundecine and its enantiomer (7b)

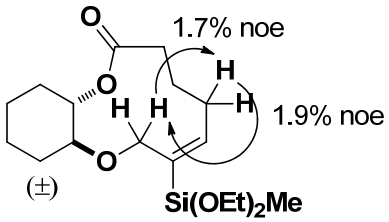
Yield 90% (colorless oil); IR (neat, cm^{-1}) 3012, 2930, 2858, 1450, 1370, 1312, 1243, 1188, 1130, 1102, 999; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 5.65 (ddd, *J* = 9.5, 9.5, 5.0 Hz, 1 H), 5.56 (ddd, *J* = 10.0, 10.0, 5.0 Hz, 1 H), 4.28 (dd, *J* = 10.0, 10.0 Hz, 1 H), 4.06 (dd, *J* = 10.0, 5.0 Hz, 1 H), 3.70 (dd, *J* = 10.0, 8.0 Hz, 1 H), 3.47 (dd, *J* = 11.5, 6.5 Hz, 1 H), 3.14 (ddd, *J* = 9.0, 9.0, 5.0 Hz, 1 H), 2.98 (ddd, *J* = 9.5, 9.5, 5.0 Hz, 1 H), 2.63-2.56 (m, 1 H), 2.04-2.00 (m, 3 H), 1.73-1.64 (m, 4 H), 1.51-1.45 (m, 1 H), 1.42-1.37 (m, 1 H), 1.19-1.07 (m, 4 H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ 135.0, 126.1, 84.3, 82.1, 71.0, 66.4, 31.9, 30.7, 28.2, 26.7, 26.1, 24.5, 24.2; HRMS (ESI-TOF) calcd. for $C_{13}H_{22}O_2$ $[M+H]^+$ 211.16926, found 211.16944.



(1*S*,2*S*)-2-((2-(Diethoxy(methyl)silyl)allyl)oxy)cyclohexyl hex-5-enoate and its enantiomer (8a)

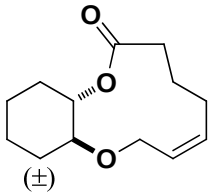
Yield 65% (colorless oil); IR (neat, cm^{-1}) 3077, 2973, 2938, 2866, 1736, 1641, 1452, 1389, 1365, 1254, 1168, 1103, 1080, 1009, 951; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 5.93-5.93 (m, 1 H), 5.78 (dddd, *J* = 17.0, 10.5, 6.5, 6.5

Hz, 1 H), 5.62-5.62 (m, 1 H), 5.04-5.00 (m, 1 H), 4.98 (d, *J* = 10.0 Hz, 1 H), 4.81 (ddd, *J* = 8.5, 8.5, 4.5 Hz, 1 H), 4.20 (d, *J* = 13.0 Hz, 1 H), 4.10 (d, *J* = 13.0 Hz, 1 H), 3.76 (q, *J* = 7.0 Hz, 4 H), 3.32 (ddd, *J* = 8.5, 8.5, 4.0 Hz, 1 H), 2.31 (dd, *J* = 8.0, 8.0 Hz, 2 H), 2.09 (ddd, *J* = 7.0, 7.0, 7.0 Hz, 2 H), 2.03-1.97 (m, 2 H), 1.76-1.64 (m, 4 H), 1.44-1.33 (m, 3 H), 1.30-1.20 (m, 7 H), 0.20 (s, 3 H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ 172.9, 144.4, 137.8, 127.1, 115.2, 78.7, 74.7, 72.0, 58.3, 58.3, 33.9, 33.0, 29.6, 29.5, 24.1, 23.1, 23.0, 18.3, -4.3; HRMS (ESI-TOF) calcd. for $C_{20}H_{36}O_5Si$ $[M+Na]^+$ 407.22242, found 407.22435.



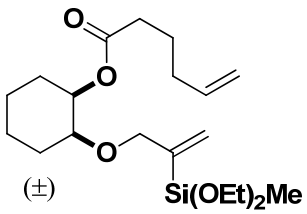
(9a*S*,13a*S*,*E*)-7-(Diethoxy(methyl)silyl)-3,4,5,8,9a,10,11,12,13,13a-decahydro-2*H*-benzo[*b*][1,4]dioxacycloundecin-2-one and its enantiomer (8)

Yield 43% (pale yellow oil); IR (neat, cm^{-1}) 2971, 2932, 2865, 1736, 1614, 1450, 1389, 1365, 1256, 1225, 1196, 1152, 1084, 1055, 983, 952; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 6.20-6.17 (m, 1 H), 4.75 (ddd, *J* = 10.0, 10.0, 5.0 Hz, 1 H), 4.22 (d, *J* = 12.8 Hz, 1 H), 3.99 (d, *J* = 12.8 Hz, 1 H), 3.81-3.73 (m, 4H), 3.21 (ddd, *J* = 10.0, 10.0, 4.5 Hz, 1 H), 2.85-2.76 (m, 1 H), 2.37-2.26 (m, 2 H), 2.16-2.12 (m, 2 H), 2.06-1.99 (m, 1 H), 1.95-1.93 (m, 1 H), 1.83-1.70 (m, 3 H), 1.34-1.16 (m, 10 H), 0.20 (s, 3 H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ 173.4, 150.2, 135.5, 79.4, 75.0, 63.3, 58.2, 58.2, 33.3, 30.6, 30.1, 27.1, 24.1, 23.6, 18.4, 18.3, -4.9; HRMS (ESI-TOF) calcd. for $C_{18}H_{32}O_5Si$ $[M+H]^+$ 357.20918, found 357.20950.



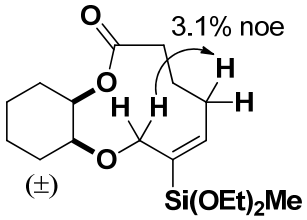
(9a*S*,13a*S*,*Z*)-3,4,5,8,9a,10,11,12,13,13a-decahydro-2*H*-benzo[*b*][1,4]dioxacycloundecin-2-one and its enantiomer (8b)

Yield 83% (colorless oil); IR (neat, cm^{-1}) 3011, 2936, 2862, 1735, 1451, 1364, 1322, 1217, 1153, 1087, 1032, 984; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 6.63-6.59 (m, 1 H), 5.54 (ddd, *J* = 10.5, 8.0, 8.0 Hz, 1 H), 4.73-4.68 (m, 1 H), 4.20 (dd, *J* = 13.2, 4.8 Hz, 1 H), 3.94 (dd, *J* = 13.2, 7.2 Hz, 1 H), 3.24-3.19 (m, 1 H), 2.50-2.43 (m, 1 H), 2.36-2.26 (m, 2 H), 2.16-2.03 (m, 2 H), 1.97-1.85 (m, 2 H), 1.82-1.67 (m, 3 H), 1.33-1.13 (m, 4 H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ 173.2, 134.6, 127.3, 80.1, 75.8, 64.3, 33.9, 30.9, 30.5, 25.9, 24.0, 23.9, 23.8; HRMS (ESI-TOF) calcd. for $C_{13}H_{20}O_3$ $[M+Na]^+$ 247.13047, found 247.13070.



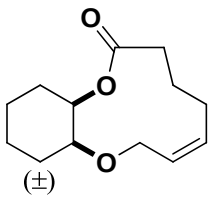
(1*R*,2*S*)-2-((2-(Diethoxy(methyl)silyl)allyl)oxy)cyclohexyl hex-5-enoate and its enantiomer (9a)

Yield 69% (colorless oil); IR (neat, cm^{-1}) 3077, 2973, 2939, 2869, 1733, 1641, 1449, 1388, 1364, 1255, 1170, 1104, 1082, 951; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 5.94-5.93 (m, 1 H), 5.78 (dddd, *J* = 16.8, 10.2, 6.8, 6.8 Hz, 1 H), 5.64-5.63 (m, 1 H), 5.08-5.07 (m, 1 H), 5.04-5.00 (m, 1 H), 4.98 (d, *J* = 10.0 Hz, 1 H), 4.14 (d, *J* = 13.0 Hz, 1 H), 4.10 (d, *J* = 13.0 Hz, 1 H), 3.79-3.75 (m, 4 H), 3.49-3.48 (m, 1 H), 2.34 (dd, *J* = 7.5, 7.5 Hz, 2 H), 2.09 (ddd, *J* = 7.0, 7.0, 7.0 Hz, 2 H), 1.93-1.88 (m, 1 H), 1.85-1.78 (m, 1 H), 1.76-1.65 (m, 3 H), 1.62-1.47 (m, 3 H), 1.43-1.29 (m, 2 H), 1.23-1.20 (m, 6 H), 0.22 (s, 3 H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ 173.1, 144.4, 137.8, 127.4, 115.2, 76.5, 71.6, 58.3, 33.9, 33.0, 27.8, 27.8, 24.2, 22.0, 21.8, 18.3, -4.3; HRMS (ESI-TOF) calcd. for $C_{20}H_{36}O_5Si$ $[M+Na]^+$ 407.22242, found 407.22426.



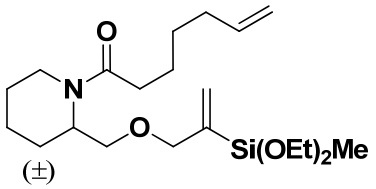
(9a*S*,13a*R*,*E*)-7-(Diethoxy(methyl)silyl)-3,4,5,8,9a,10,11,12,13,13a-decahydro-2*H*-benzo[*b*][1,4]dioxacycloundecin-2-one and its enantiomer (9)

Yield 36% (pale yellow oil); IR (neat, cm^{-1}) 2970, 2931, 2870, 1730, 1614, 1450, 1390, 1360, 1246, 1225, 1162, 1110, 1080, 1049, 949; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 6.24 (dd, *J* = 9.8, 6.2 Hz, 1 H), 4.67 (ddd, *J* = 11.0, 3.8, 3.8 Hz, 1 H), 4.29 (d, *J* = 11.5 Hz, 1 H), 3.89 (bs, 1 H), 3.86 (d, *J* = 11.5 Hz, 1 H), 3.80-3.75 (m, 4 H), 2.45 (ddd, *J* = 13.2, 8.2, 4.8 Hz, 1 H), 2.25-2.13 (m, 3 H), 1.94-1.88 (m, 2 H), 1.86-1.78 (m, 2 H), 1.72-1.70 (m, 1 H), 1.59-1.48 (m, 2 H), 1.42-1.17 (m, 9 H), 0.21 (s, 3 H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ 173.6, 147.9, 134.7, 75.6, 74.0, 65.8, 58.3, 34.7, 28.6, 27.6, 26.5, 24.9, 23.7, 19.9, 18.3, -4.6; HRMS (ESI-TOF) calcd. for $C_{18}H_{32}O_5Si$ $[M+H]^+$ 357.20918, found 357.21015.



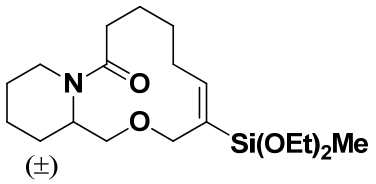
(9a*S*,13a*R*,*Z*)-3,4,5,8,9a,10,11,12,13,13a-decahydro-2*H*-benzo[*b*][1,4]dioxacycloundecin-2-one and its enantiomer (9b)

Yield 92% (colorless oil), inseparable mixture with styrene derivative; IR (neat, cm^{-1}) 3010, 2937, 2862, 1729, 1448, 1359, 1243, 1212, 1155, 1083, 1051, 1014; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 5.61-5.52 (m, 2 H), 4.66-4.62 (m, 1 H), 4.50 (dd, *J* = 12.2, 7.8 Hz, 1 H), 4.05 (bs, 1 H), 3.93 (dd, *J* = 12.2, 5.0 Hz, 1 H), 2.33-2.29 (m, 2 H), 2.24-2.10 (m, 2 H), 1.93-1.79 (m, 4 H), 1.74-1.72 (m, 1 H), 1.59-1.50 (m, 2 H), 1.47-1.27 (m 3 H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ 173.5, 133.7, 127.6, 75.5, 72.3, 64.8, 35.0, 30.1, 26.3, 26.1, 24.9, 24.1, 19.7; HRMS (ESI-TOF) calcd. for $C_{13}H_{20}O_3$ $[M+Na]^+$ 247.13047, found 247.13029.



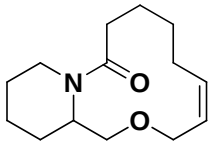
(±)-1-(2-(((2-(Diethoxy(methyl)silyl)allyl)oxy)methyl)piperidin-1-yl)hept-6-en-1-one (10a)

Yield 54% (pale yellow oil); IR (neat, cm^{-1}) 3075, 2973, 2930, 2865, 1644, 1425, 1390, 1365, 1257, 1166, 1103, 1081, 1029, 952; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 5.89 and 5.87 (pair of bs due to rotamers, 1 H), 5.80 (dddd, *J* = 17.0, 10.0, 6.8, 6.8 Hz, 1 H), 5.64 (bs, 1 H), 5.01-4.98 (m, 1 H), 4.93 (d, *J* = 10.0 Hz, 1 H), 4.57 and 3.66 (pair of d due to rotamers, *J* = 13.0 Hz, 1 H), 4.14-4.04 (m, 3 H), 3.76 (q, *J* = 6.8 Hz, 4 H), 3.61-3.46 (m, 2 H), 3.11 and 2.57 (pair of dd due to rotamers, *J* = 13.0, 13.0 Hz, 1 H), 2.43-2.29 (m, 2 H), 2.07 (ddd, *J* = 7.2, 7.2, 7.2 Hz, 2 H), 1.86-1.80 (m, 1 H), 1.71-1.49 (m, 6 H), 1.46-1.33 (m, 3 H), 1.21 (t, *J* = 7.0 Hz, 6 H), 0.20 (s, 3H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ 172.4 and 171.8 (due to rotamers), 144.0 and 143.8 (due to rotamers), 138.7, 127.7, 114.4, 74.0 and 73.8 (due to rotamers), 68.7 and 68.4 (due to rotamers), 58.3, 52.4, 46.8, 42.2, 37.0, 33.6 and 33.2 (due to rotamers), 33.6, 28.7, 26.5 and 25.9 (due to rotamers), 25.2 and 25.1 (due to rotamers), 24.9 and 24.8 (due to rotamers), 19.6 and 19.4 (due to rotamers), 18.3, -4.5; HRMS (ESI-TOF) calcd. for $C_{21}H_{39}NO_4Si$ $[M+H]^+$ 398.27211, found 398.27371.



(±)-(*E*)-4-(Diethoxy(methyl)silyl)-1,6,7,8,9,12,13,14,15,15a-decahydropyrido[2,1-*c*][1,4]oxaazacyclododecin-10(3*H*)-one (10)

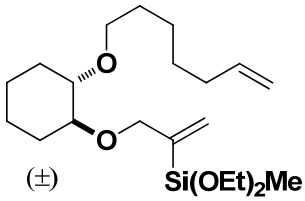
Yield 33% (colorless oil); IR (neat, cm^{-1}) 2970, 2928, 2865, 1634, 1444, 1389, 1366, 1256, 1165, 1106, 1079, 952; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 6.15 (dd, *J* = 9.5, 5.5 Hz, 1 H), 4.65 (d, *J* = 13.0 Hz, 1 H), 4.40 (bs, 1 H), 4.29 (d, *J* = 11.2 Hz, 1 H), 3.95 (d, *J* = 11.2 Hz, 1 H), 3.79 (dd, *J* = 9.8, 9.8 Hz, 1 H), 3.73 (q, *J* = 6.9 Hz, 4 H), 3.33 (dd, *J* = 11.0, 4.5 Hz, 1 H), 2.85-2.82 (m, 1 H), 2.59-2.55 (m, 2 H), 1.96-1.92 (m, 2 H), 1.74-1.58 (m, 6 H), 1.48-1.35 (m, 4 H), 1.19 (dd, *J* = 6.8, 6.8 Hz, 6 H), 0.16 (s, 3 H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ 173.7, 148.0, 132.9, 67.6, 67.4, 58.3 52.0, 36.6, 29.3, 27.3, 27.0, 26.9, 25.3, 23.8, 19.7, 18.3, 18.3, -4.6; HRMS (ESI-TOF) calcd. for $C_{19}H_{35}NO_4Si$ $[M+Na]^+$ 392.22276, found 392.22352.



(±)

(±)-(*Z*)-1,6,7,8,9,12,13,14,15,15a-decahydropyrido[2,1-*c*][1,4]oxaazacyclododecin-10(3*H*)-one (10b)

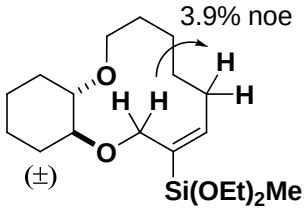
Yield 90% (colorless oil); IR (neat, cm^{-1}) 3010, 2934, 2861, 1631, 1444, 1419, 1367, 1327, 1266, 1125, 1078, 1029; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 5.59-5.49 (m, 2 H), 4.61-4.59 (m, 1 H), 4.36 (bs, 1 H), 4.24-4.20 (m, 1 H), 3.84-3.82 (m, 1 H), 3.76 (dd, *J* = 9.8, 9.8 Hz, 1 H), 3.43-3.40 (m, 1 H), 2.74 (bs, 1 H), 2.53 (dd, *J* = 12.2, 12.2 Hz, 1 H), 2.38 (bs, 1 H), 1.93-1.92 (m, 2 H), 1.73-1.60 (m, 6 H), 1.44-1.30 (m, 4 H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ 173.3, 134.9, 125.4, 67.3, 65.5, 51.1, 36.8, 29.5, 26.8, 26.4, 25.2, 25.1, 23.6, 19.4; HRMS (ESI-TOF) calcd. for $C_{14}H_{23}NO_2$ $[M+Na]^+$ 260.16210, found 260.16176.



(±)

Diethoxy(3-(((1S,2S)-2-(hept-6-en-1-yloxy)cyclohexyl)oxy)prop-1-en-2-yl)(methyl)silane and its enantiomer (11a)

Yield 59% (colorless oil); IR (neat, cm^{-1}) 3076, 2974, 2933, 2862, 1641, 1451, 1390, 1366, 1295, 1257, 1164, 1104, 1083, 994, 952; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 5.99-5.98 (m, 1 H), 5.80 (dddd, *J* = 17.0, 10.5, 6.8, 6.8 Hz, 1 H), 5.64-5.63 (m, 1 H), 5.01-4.97 (m, 1 H), 4.94-4.92 (m, 1 H), 4.23-4.23 (m, 2 H), 3.77 (q, *J* = 7.0 Hz, 4 H), 3.57-3.48 (m, 2 H), 3.25-3.18 (m, 2 H), 2.04 (ddd, *J* = 7.0, 7.0, 7.0 Hz, 2 H), 1.96-1.94 (m, 2 H), 1.65-1.62 (m, 2 H), 1.59-1.53 (m, 2 H), 1.41-1.19 (m, 14 H), 0.22 (s, 3 H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ 144.8, 139.0, 126.9, 114.2, 81.1, 80.7, 72.4, 69.8, 58.3, 33.7, 30.2, 29.8, 29.8, 28.8, 25.7, 23.3, 23.3, 18.3, -4.3; HRMS (ESI-TOF) calcd. for $C_{21}H_{40}O_4Si$ $[M+Na]^+$ 407.25881, found 407.25995.

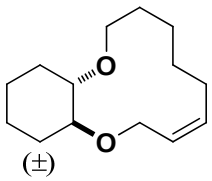


(±)

((10a*S*,14a*S*,*E*)-2,5,6,7,8,9,10a,11,12,13,14,14a-Dodecahydrobenzo[*b*][1,4]dioxacyclododecin-3-yl)diethoxy(methyl)silane and its enantiomer (11)

Yield 59% (pale yellow oil); IR (neat, cm^{-1}) 2970, 2930, 2859, 1616, 1450, 1389, 1365, 1254, 1166, 1135, 1111, 1083, 1024, 950; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 6.33 (dd, *J* = 8.8, 6.8 Hz, 1 H), 4.35 (d, *J* = 8.8 Hz, 1

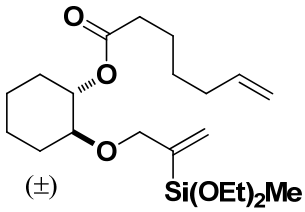
H), 4.07 (d, *J* = 8.8 Hz, 1 H), 3.92-3.89 (m, 1 H), 3.79-3.74 (m, 4 H), 3.16-3.12 (m, 1 H), 3.09-3.05 (m, 2 H), 2.62-2.55 (m, 1 H), 2.08-2.03 (m, 2 H), 2.00-1.94 (m, 1 H), 1.68-1.59 (m, 4 H), 1.56-1.46 (m, 4 H), 1.22-1.08 (m, 10 H), 0.19 (s, 3 H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ 149.7, 133.7, 84.2, 81.4, 70.0, 66.5, 58.2, 58.2, 31.2, 30.7, 27.7, 27.4, 27.2, 25.8, 24.5, 24.2, 18.3, -4.5; HRMS (ESI-TOF) calcd. for $C_{19}H_{36}O_4Si$ $[M+Na]^+$ 379.22751, found 379.22910.



(±)

(10a*S*,14a*S*,*Z*)-2,3,4,5,6,9,10a,11,12,13,14,14a-dodecahydrobenzo[*b*][1,4]dioxacyclododecine and its enantiomer (11b)

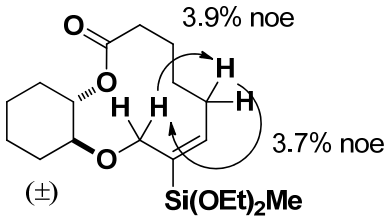
Yield 88% (colorless oil), inseparable mixture with styrene derivative; IR (neat, cm^{-1}) 3014, 2930, 2858, 1451, 1361, 1334, 1313, 1244, 1190, 1130, 1107, 1047, 983, 962; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 5.73-5.65 (m, 2 H), 4.47 (dd, *J* = 9.0, 9.0 Hz, 1 H), 3.96 (dd, *J* = 9.2, 4.2 Hz, 1 H), 3.93-3.90 (m, 1 H), 3.16-3.08 (m, 3 H), 2.45-2.38 (m, 1 H), 2.08-2.07 (m, 1 H), 2.00-1.98 (m, 1 H), 1.90-1.86 (m, 1 H), 1.70-1.60 (m, 3 H), 1.52-1.45 (m, 4 H), 1.28-1.13 (m, 5 H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ 135.4, 126.3, 84.8, 80.8, 69.6, 66.0, 31.5, 30.6, 28.1, 27.3, 24.9, 24.6, 24.1, 24.0; HRMS (ESI-TOF) calcd. for $C_{14}H_{24}O_2$ $[M+Na]^+$ 247.16685, found 247.16800.



(±)

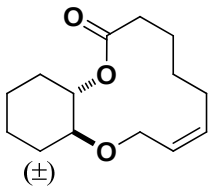
(1*S*,2*S*)-2-((2-(Diethoxy(methyl)silyl)allyl)oxy)cyclohexyl hept-6-enoate and its enantiomer (12a)

Yield 60% (colorless oil); IR (neat, cm^{-1}) 3075, 2973, 2937, 2865, 1736, 1641, 1452, 1389, 1257, 1166, 1103, 1080, 1008, 952; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 5.93-5.93 (m, 1 H), 5.79 (dddd, *J* = 17.0, 10.5, 6.5, 6.5 Hz, 1 H), 5.62-5.62 (m, 1 H), 5.02-4.98 (m, 1 H), 4.95 (d, *J* = 10.0 Hz, 1 H), 4.81 (ddd, *J* = 8.2, 8.2, 4.5 Hz, 1 H), 4.21 (d, *J* = 13.2 Hz, 1 H), 4.10 (d, *J* = 13.2 Hz, 1 H), 3.76 (q, *J* = 7.0 Hz, 4 H), 3.32 (ddd, *J* = 8.5, 8.5, 4.0 Hz, 1 H), 2.30 (dd, *J* = 8.0, 8.0 Hz, 2 H), 2.08-1.96 (m, 4 H), 1.71-1.61 (m, 4 H), 1.45-1.39 (m, 3 H), 1.34 (dd, *J* = 9.5, 9.5 Hz, 2 H), 1.30-1.20 (m, 7 H), 0.21 (s, 3 H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ 173.0, 144.4, 138.4, 127.1, 114.6, 78.7, 74.6, 72.0, 58.3, 58.3, 34.5, 33.4, 29.6, 29.5, 28.3, 24.4, 23.1, 23.0, 18.3, -4.3; HRMS (ESI-TOF) calcd. for $C_{21}H_{38}O_5Si$ $[M+Na]^+$ 421.23807, found 421.23885.



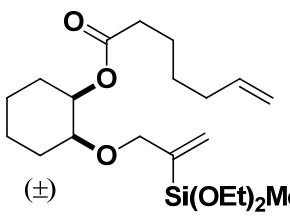
(10a*S*,14a*S*,*E*)-8-(Diethoxy(methyl)silyl)-3,4,5,6,10a,11,12,13,14,14a-decahydrobenzo[*b*][1,4]dioxacyclododecin-2(9*H*)-one and its enantiomer (12)

Yield 82% (pale yellow oil); IR (neat, cm^{-1}) 2971, 2936, 2866, 1734, 1617, 1451, 1389, 1360, 1338, 1256, 1225, 1189, 1150, 1104, 1082, 1036, 996, 950; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 6.37 (dd, *J* = 10.2, 5.8 Hz, 1 H), 4.61 (ddd, *J* = 10.2, 10.2, 4.5 Hz, 1 H), 4.10 (d, *J* = 9.0 Hz, 1 H), 4.04 (d, *J* = 9.0 Hz, 1 H), 3.76-3.71 (m, 4 H), 3.22 (ddd, *J* = 10.0, 10.0, 4.5 Hz, 1 H), 2.66 (dddd, *J* = 11.3, 11.3, 11.3, 4.0 Hz, 1 H), 2.47 (ddd, *J* = 12.5, 12.5, 4.5 Hz, 1 H), 2.35 (ddd, *J* = 13.2, 4.8, 4.8 Hz, 1 H), 2.16-2.15 (m, 1 H), 2.07-2.06 (m, 1 H), 1.96-1.90 (m, 1 H), 1.85-1.78 (m, 1 H), 1.74-1.69 (m, 2 H), 1.65-1.56 (m, 2 H), 1.33-1.18 (m, 11 H), 0.17 (s, 3 H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ 173.8, 151.1, 132.6, 80.4, 76.5, 65.4, 58.3, 58.2, 33.3, 31.0, 30.2, 27.9, 27.7, 24.9, 24.1, 24.0, 18.3, -4.9; HRMS (ESI-TOF) calcd. for $C_{19}H_{34}O_5Si$ $[M+H]^+$ 371.22483, found 371.22556.



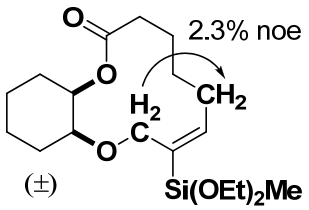
(10a*S*,14a*S*,*Z*)-3,4,5,6,10a,11,12,13,14,14a-decahydrobenzo[*b*][1,4]dioxacyclododecin-2(9*H*)-one and its enantiomer (12b)

Yield 91% (colorless oil); IR (neat, cm^{-1}) 3019, 2937, 2862, 1732, 1451, 1354, 1278, 1222, 1150, 1107, 1085, 1034, 989; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 5.84 (ddd, *J* = 10.0, 10.0, 5.5 Hz, 1 H), 5.65 (ddd, *J* = 10.0, 7.0, 7.0 Hz, 1 H), 4.62 (ddd, *J* = 10.0, 10.0, 5.0 Hz, 1 H), 4.06 (dd, *J* = 8.5, 8.5 Hz, 1 H), 3.98 (dd, *J* = 9.5, 6.5 Hz, 1 H), 3.22 (ddd, *J* = 10.0, 10.0, 4.0 Hz, 1 H), 2.55 (dddd, *J* = 11.5, 11.5, 11.5, 4.0 Hz, 1 H), 2.46 (ddd, *J* = 12.5, 12.5, 4.0 Hz, 1 H), 2.35 (ddd, *J* = 13.0, 5.0, 5.0 Hz, 1 H), 2.11-2.06 (m, 2 H), 1.94-1.87 (m, 1 H), 1.76-1.67 (m, 3 H), 1.65-1.55 (m, 2 H), 1.34-1.17 (m, 5 H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ 173.6, 138.1, 124.6, 80.6, 76.7, 65.2, 33.1, 30.9, 30.6, 28.2, 25.9, 24.6, 24.2, 23.9; HRMS (ESI-TOF) calcd. for $C_{14}H_{22}O_3$ $[M+Na]^+$ 261.14612, found 261.14610.



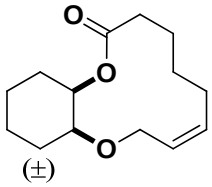
(1*R*,2*S*)-2-((2-(Diethoxy(methyl)silyl)allyl)oxy)cyclohexyl hept-6-enoate and its enantiomer (13a)

Yield 66% (colorless oil); IR (neat, cm^{-1}) 3076, 2972, 2938, 2866, 1734, 1641, 1449, 1388, 1364, 1257, 1169, 1104, 1081, 992, 951; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 5.94-5.93 (m, 1 H), 5.79 (dddd, *J* = 17.0, 10.5, 6.5, 6.5 Hz, 1 H), 5.64-5.63 (m, 1 H), 5.08-5.06 (m, 1 H), 5.02-4.98 (m, 1 H), 4.94 (d, *J* = 10.0 Hz, 1 H), 4.14 (d, *J* = 13.0 Hz, 1 H), 4.10 (d, *J* = 13.0 Hz, 1 H), 3.79-3.75 (m, 4 H), 3.49-3.48 (m, 1 H), 2.33 (dd, *J* = 7.2, 7.2 Hz, 2 H), 2.06 (ddd, *J* = 7.2, 7.2, 7.2 Hz, 2 H), 1.93-1.87 (m, 1 H), 1.85-1.78 (m, 1 H), 1.70-1.47 (m, 6 H), 1.46-1.29 (m, 4 H), 1.21 (dd, *J* = 7.0, 7.0 Hz, 6 H), 0.22 (s, 3 H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ 173.2, 144.4, 138.5, 127.4, 114.6, 76.5, 71.6, 58.3, 34.5, 33.4, 28.3, 27.8, 27.8, 24.5, 22.0, 21.9, 18.3, -4.3; HRMS (ESI-TOF) calcd. for $C_{21}H_{38}O_5Si$ $[M+Na]^+$ 421.23807, found 421.23931.



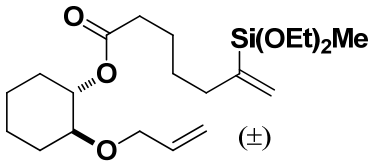
(10a*S*,14a*R*,*E*)-8-(Diethoxy(methyl)silyl)-3,4,5,6,10a,11,12,13,14,14a-decahydrobenzo[*b*][1,4]dioxacyclododecin-2(9*H*)-one and its enantiomer (13)

Yield 46% (pale yellow oil); IR (neat, cm^{-1}) 2970, 2935, 2864, 1730, 1616, 1449, 1389, 1353, 1256, 1224, 1156, 1105, 1079, 984, 952; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 6.26 (dd, *J* = 8.2, 6.8 Hz, 1 H), 5.02-5.00 (m, 1 H), 4.06-4.01 (m, 2 H), 3.78-3.73 (m, 4 H), 3.61-3.60 (m, 1 H), 2.40-2.28 (m, 2 H), 2.27-2.15 (m, 2 H), 1.96-1.90 (m, 1 H), 1.88-1.78 (m, 2 H), 1.75-1.68 (m, 1 H), 1.67-1.54 (m, 6 H), 1.40-1.29 (m, 2 H), 1.22-1.19 (m, 6 H), 0.18 (s, 3 H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ 174.3, 149.6, 133.3, 75.0, 71.9, 63.6, 58.3, 58.2, 34.9, 29.7, 28.6, 27.9, 27.7, 27.6, 24.4, 22.1, 21.7, 18.3, -4.8; HRMS (ESI-TOF) calcd. for $C_{19}H_{34}O_5Si$ $[M+Na]^+$ 393.20677, found 393.20690.



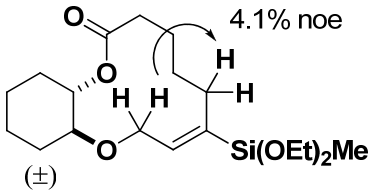
(10a*S*,14a*R*,*Z*)-3,4,5,6,10a,11,12,13,14,14a-decahydrobenzo[*b*][1,4]dioxacyclododecin-2(9*H*)-one and its enantiomer (13b)

Yield 97% (colorless oil); IR (neat, cm^{-1}) 2935, 2859, 1729, 1449, 1352, 1219, 1147, 1081, 1047; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 5.71 (ddd, *J* = 10.5, 8.0, 8.0 Hz, 1 H), 5.61 (ddd, *J* = 10.5, 6.5, 6.5 Hz, 1 H), 5.07-5.06 (m, 1 H), 4.09-4.00 (m, 2 H), 3.65-3.63 (m, 1 H), 2.43-2.38 (m, 1 H), 2.34-2.29 (m, 1 H), 2.26-2.18 (m, 1 H), 2.10-2.03 (m, 1 H), 1.99-1.94 (m, 1 H), 1.83-1.72 (m, 3 H), 1.71-1.50 (m, 6 H), 1.42-1.28 (m, 2 H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ 174.3, 136.3, 125.4, 74.3, 71.9, 62.8, 34.6, 28.7, 28.3, 27.5, 26.1, 23.9, 22.2, 21.6; HRMS (ESI-TOF) calcd. for $C_{14}H_{22}O_3$ $[M+Na]^+$ 261.14612, found 261.14045.



(1*S*,2*S*)-2-(Allyloxy)cyclohexyl 6-(diethoxy(methyl)silyl)hept-6-enoate and its enantiomer (14a)

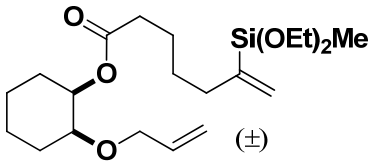
Yield 82% (colorless oil); IR (neat, cm^{-1}) 3075, 2973, 2937, 2865, 1736, 1641, 1452, 1389, 1364, 1257, 1166, 1102, 1080, 1008, 994, 952; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 5.91-5.84 (m, 1 H), 5.67 (bs, 1 H), 5.55-5.55 (m, 1 H), 5.25 (d, *J* = 17.5 Hz, 1 H), 5.13 (d, *J* = 10.0 Hz, 1 H), 4.80-4.76 (m, 1 H), 4.09 (dd, *J* = 12.8, 5.2 Hz, 1 H), 4.01 (dd, *J* = 12.8, 5.2 Hz, 1 H), 3.76 (q, *J* = 7.0 Hz, 4 H), 3.32-3.28 (m, 1 H), 2.31 (dd, *J* = 7.8, 7.8 Hz, 2 H), 2.16 (dd, *J* = 7.8, 7.8 Hz, 2 H), 2.00-1.97 (m, 2 H), 1.71-1.62 (m, 4 H), 1.52-1.46 (m, 2 H), 1.41-1.20 (m, 10 H), 0.19 (s, 3 H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ 173.0, 147.1, 135.4, 127.5, 116.2, 78.4, 74.7, 70.4, 58.2, 35.1, 34.6, 29.9, 29.7, 28.2, 24.9, 23.2, 23.2, 18.3, -4.6; HRMS (ESI-TOF) calcd. for $C_{21}H_{38}O_5Si$ $[M+Na]^+$ 421.23807, found 421.24013.



(10a*S*,14a*S*,*E*)-7-(Diethoxy(methyl)silyl)-3,4,5,6,10a,11,12,13,14,14a-decahydrobenzo[*b*][1,4]dioxacyclododecin-2(9*H*)-one and its enantiomer (14)

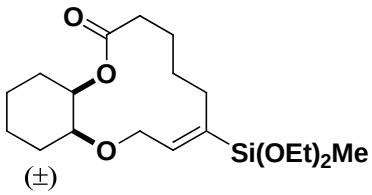
Yield 79% (pale yellow oil); IR (neat, cm^{-1}) 2971, 2937, 2866, 1733, 1450, 1390, 1353, 1339, 1256, 1227, 1146, 1103, 1081, 1036, 996, 951; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 6.21 (dd, *J* = 7.0, 7.0 Hz, 1H), 4.65-4.60 (m, 1 H), 4.20 (dd, *J* = 8.5, 8.5 Hz, 1 H), 3.98 (dd, *J* = 9.0, 6.0 Hz, 1 H), 3.76-3.72 (m, 4 H), 3.23 (ddd, *J* = 10.0, 10.0, 4.0 Hz, 1 H), 2.58 (ddd, *J* = 12.5, 12.5, 3.5 Hz, 1 H), 2.54-2.48 (m, 1 H), 2.34 (ddd, *J* = 13.0, 5.0, 5.0 Hz, 1 H), 2.10 (bs, 2 H), 1.95-1.87 (m, 2 H), 1.76-1.69 (m, 3 H), 1.64-1.57 (m, 1 H), 1.33-1.18 (m, 11 H), 0.18 (s, 3 H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ 173.7, 146.5, 136.5, 80.7, 77.1, 65.8, 58.2, 58.2, 33.4, 31.0, 30.7, 29.0, 27.8, 25.5, 24.3, 23.9, 18.3, 18.3, -4.7; HRMS (ESI-TOF) calcd. for $C_{19}H_{34}O_5Si$ $[M+H]^+$ 371.22483, found 371.22591.

Protodesilylation of **14** generated **12b** with 52% yield.



(1*R*,2*S*)-2-(Allyloxy)cyclohexyl 6-(diethoxy(methyl)silyl)hept-6-enoate and its enantiomer (15a)

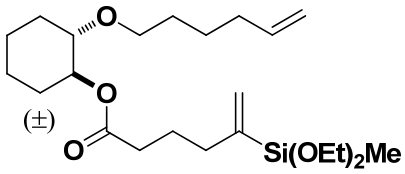
Yield 80% (colorless oil); IR (neat, cm^{-1}) 3051, 2971, 2938, 2866, 1733, 1449, 1388, 1365, 1257, 1238, 1168, 1104, 1081, 950; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 5.92-5.84 (m, 1 H), 5.68-5.67 (m, 1 H), 5.55-5.54 (m, 1 H), 5.28-5.24 (m, 1 H), 5.15-5.12 (m, 1 H), 5.09-5.08 (m, 1H), 4.05 (dd, *J* = 13.0, 5.8 Hz, 1H), 3.98 (dd, *J* = 13.2, 5.8 Hz, 1 H), 3.76 (q, *J* = 7.0 Hz, 4 H), 3.49-3.47 (m, 1 H), 2.35 (dd, *J* = 7.5, 7.5 Hz, 2 H), 2.16 (dd, *J* = 7.5, 7.5 Hz, 2 H), 1.91-1.86 (m, 1 H), 1.83-1.76 (m, 1 H), 1.71-1.62 (m, 3 H), 1.60-1.46 (m, 5 H), 1.43-1.29 (m, 2 H), 1.21 (dd, *J* = 7.0, 7.0 Hz, 6 H), 0.18 (s, 3 H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ 173.3, 174.1, 135.3, 127.5, 116.5, 76.0, 71.0, 69.7, 58.2, 35.1, 34.6, 28.2, 27.9, 27.8, 24.9, 22.1, 21.7, 18.3, -4.6; HRMS (ESI-TOF) calcd. for $C_{21}H_{38}O_5Si$ $[M+Na]^+$ 421.23807, found 421.23908.



(10a*S*,14a*R*,*E*)-7-(Diethoxy(methyl)silyl)-3,4,5,6,10a,11,12,13,14,14a-decahydrobenzo[*b*][1,4]dioxacyclododecin-2(9*H*)-one and its enantiomer (15)

Yield 14% (colorless oil); IR (neat, cm^{-1}) 2970, 2935, 2865, 1731, 1449, 1390, 1354, 1256, 1226, 1149, 1103, 1079, 986, 952; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 6.14 (dd, *J* = 6.5, 6.5 Hz, 1 H), 5.06-5.05 (m, 1H), 4.19 (dd, *J* = 10.8, 6.5 Hz, 1 H), 4.03 (dd, *J* = 10.8, 6.5 Hz, 1 H), 3.77-3.72 (m, 4 H), 3.60-3.59 (m, 1 H), 2.46-2.40 (m, 2 H), 2.34 (ddd, *J* = 13.0, 5.5, 5.5 Hz, 1 H), 2.08 (ddd, *J* = 12.2, 12.2, 5.0 Hz, 1 H), 1.98-1.92 (m, 1 H), 1.84-1.71 (m, 3 H), 1.68-1.54 (m, 5 H), 1.50-1.28 (m, 3 H), 1.20 (dd, *J* = 7.5, 7.5 Hz, 6 H), 0.18 (s, 3 H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ 174.0, 144.2, 137.3, 74.8, 72.3, 63.9, 58.2, 34.0, 29.7, 28.3, 28.1, 28.0, 27.4, 25.1, 22.3, 21.4, 18.3, -4.6; HRMS (ESI-TOF) calcd. for $C_{19}H_{34}O_5Si$ $[M+H]^+$ 371.22483, found 371.22603.

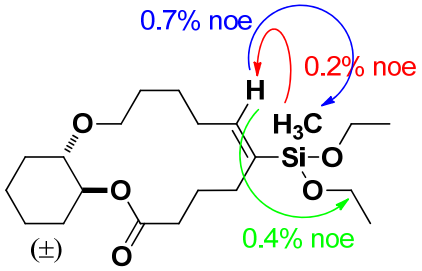
Protodesilylation of **15** generated **13b** with 54% yield.



(1*S*,2*S*)-2-(Hex-5-en-1-yloxy)cyclohexyl 5-(diethoxy(methyl)silyl)hex-5-enoate and its enantiomer (16a)

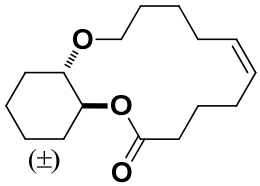
Yield 73% (colorless oil); IR (neat, cm^{-1}) 3076, 2972, 2938, 2866, 1735, 1641, 1452, 1389, 1256, 1165, 1109, 1082, 953; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 5.79 (dddd, *J* = 17.2, 10.2, 7.0, 7.0 Hz, 1 H), 5.69-5.69 (m, 1 H), 5.58-5.58 (m, 1 H), 5.01-4.97 (m, 1 H), 4.94 (d, *J* = 10.0 Hz, 1 H), 4.78-4.74 (m, 1 H), 3.76 (q, *J* = 7.0 Hz, 4 H), 3.56-3.52 (m, 1 H), 3.44-3.39 (m, 1 H), 3.24-3.19 (m, 1 H), 2.30 (dd, *J* = 7.5, 7.5 Hz, 2 H), 2.19 (dd, *J* = 7.5, 7.5 Hz, 2 H), 2.05 (ddd, *J* = 7.0, 7.0, 7.0 Hz, 2 H), 1.98-1.96 (m, 2 H), 1.82-1.76 (m, 2 H), 1.70-1.64 (m, 2 H), 1.56-1.50 (m, 2 H), 1.45-1.40 (m, 2 H), 1.36-1.31 (m, 3 H), 1.28-1.20 (m, 7 H), 0.20 (s, 3 H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ 172.9, 146.6, 138.8, 128.1, 114.4, 78.9, 74.6, 69.3, 58.2, 34.9, 34.2, 33.5, 29.8, 29.7,

29.6, 25.5, 24.1, 23.2, 18.3, -4.6; HRMS (ESI-TOF) calcd. for $C_{23}H_{42}O_5Si$ $[M+Na]^+$ 449.26937, found 449.27061.



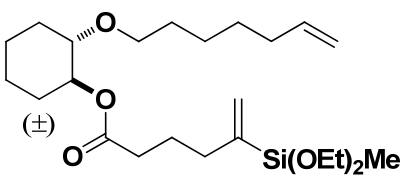
(12a*S*,16a*S*,*E*)-6-(Diethoxy(methyl)silyl)-4,5,8,9,10,11,12a,13,14,15,16,16a-dodecahydrobenzo[*b*][1,4]dioxacyclotetradecin-2(3*H*)-one and its enantiomer (16)

Yield 76% (pale yellow oil); IR (neat, cm^{-1}) 2930, 2865, 2733, 1731, 1612, 1452, 1389, 1367, 1338, 1293, 1252, 1212, 1191, 1165, 1110, 1080, 1020, 989, 951; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 6.08 (dd, *J* = 7.0, 7.0 Hz, 1H), 4.76 (ddd, *J* = 10.0, 10.0, 4.5 Hz, 1 H), 3.80-3.71 (m, 5 H), 3.28-3.22 (m, 2 H), 2.38-2.12 (m, 6 H), 2.07-1.99 (m, 2 H), 1.88-1.82 (m, 1 H), 1.74-1.52 (m, 6 H), 1.43-1.36 (m, 1 H), 1.32-1.18 (m, 10 H), 0.16 (s, 3 H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ 173.2, 145.5, 135.4, 79.8, 75.2, 67.7, 58.1, 33.4, 30.9, 29.6, 28.5, 28.3, 27.4, 27.1, 24.4, 24.1, 24.0, 18.3, -4.4; HRMS (ESI-TOF) calcd. for $C_{21}H_{38}O_5Si$ $[M+H]^+$ 399.25613, found 399.25752.



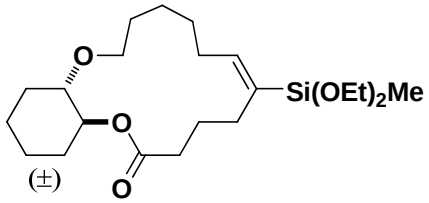
(12a*S*,16a*S*,*Z*)-4,5,8,9,10,11,12a,13,14,15,16,16a-dodecahydrobenzo[*b*][1,4]dioxacyclotetradecin-2(3*H*)-one and its enantiomer (16b)

Yield 60% (colorless oil), inseparable mixture with styrene; IR (neat, cm^{-1}) 3002, 2936, 2861, 1731, 1452, 1368, 1246, 1207, 1162, 1111, 1022; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 5.51 (ddd, *J* = 8.5, 8.5, 8.5 Hz, 1H), 5.24 (ddd, *J* = 10.0, 10.0, 6.5 Hz, 1H), 4.80 (ddd, *J* = 10.0, 10.0, 5.0 Hz, 1 H), 3.78-3.75 (m, 1 H), 3.25-3.20 (m, 2 H), 2.38-2.04 (m, 6 H), 1.99-1.83 (m, 3 H), 1.76-1.57 (m, 3 H), 1.55-1.37 (m, 4 H), 1.32-1.17 (m, 4 H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ 173.1, 131.6, 128.9, 79.8, 74.9, 66.9, 32.1, 30.9, 29.7, 28.3, 26.7, 26.6, 25.6, 24.1, 23.9, 23.7; HRMS (ESI-TOF) calcd. for $C_{16}H_{26}O_3$ $[M+Na]^+$ 289.17742, found 289.17804.



(1*S*,2*S*)-2-(Hept-6-en-1-yloxy)cyclohexyl 5-(diethoxy(methyl)silyl)hex-5-enoate and its enantiomer (17a)

Yield 79% (colorless oil); IR (neat, cm^{-1}) 3077, 2973, 2937, 2865, 1736, 1641, 1452, 1389, 1256, 1166, 1110, 1082, 995, 953; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 5.80 (dddd, *J* = 17.0, 10.0, 6.8, 6.8 Hz, 1 H), 5.69-5.69 (m, 1 H), 5.58-5.58 (m, 1 H), 5.01-4.97 (m, 1 H), 4.93 (d, *J* = 10.0 Hz, 1 H), 4.76 (ddd, *J* = 8.5, 8.5, 4.5 Hz, 1 H), 3.76 (q, *J* = 7.0 Hz, 4 H), 3.53 (ddd, *J* = 9.0, 6.5, 6.5 Hz, 1 H), 3.41 (ddd, *J* = 9.5, 7.0, 7.0 Hz, 1 H), 3.21 (ddd, *J* = 8.5, 8.5, 4.0 Hz, 1 H), 2.30 (dd, *J* = 7.2, 7.2 Hz, 2 H), 2.19 (dd, *J* = 7.8, 7.8 Hz, 2 H), 2.04 (ddd, *J* = 7.2, 7.2, 7.2 Hz, 2 H), 1.98-1.96 (m, 2 H), 1.83-1.77 (m, 2 H), 1.70-1.64 (m, 2 H), 1.55-1.49 (m, 2 H), 1.42-1.20 (m, 14 H), 0.20 (s, 3 H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ 172.9, 146.6, 138.9, 128.1, 114.2, 78.9, 74.6, 69.5, 58.2, 34.9, 34.3, 33.7, 30.0, 29.8, 29.7, 28.7, 25.6, 24.1, 23.2, 18.3, -4.6; HRMS (ESI-TOF) calcd. for $C_{24}H_{44}O_5Si$ $[M+H]^+$ 441.30308, found 441.30151.

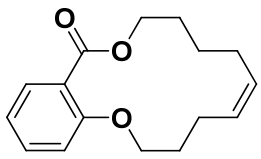


(13a*S*,17a*S*,*E*)-6-(Diethoxy(methyl)silyl)-3,4,5,8,9,10,11,12,13a,14,15,16,17,17a-tetradecahydro-2*H*-benzo[*b*][1,4]dioxacyclopentadecin-2-one and its enantiomer (17)

Yield 43% (pale yellow oil), *Z*:*E* = 8:92, *E* product was purified and characterized.; IR (neat, cm^{-1}) 2935, 2861, 1735, 1613, 1452, 1414, 1365, 1311, 1255, 1218, 1188, 1162, 1111, 1080, 1017, 988, 952; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 6.08 (dd, *J* = 10.5, 5.0 Hz, 1 H), 4.72 (ddd, *J* = 10.0, 10.0, 4.3 Hz, 1 H), 3.74 (q, *J* = 7.0Hz, 4 H), 3.68-3.65 (m, 1 H), 3.40-3.36 (m, 1 H), 3.15 (ddd, 9.5, 9.5, 4.5 Hz, 1 H), 2.55-2.49 (m, 1 H), 2.38-2.20 (m, 3 H), 2.12-2.07 (m, 2 H), 2.02-1.89 (m, 3 H), 1.72-1.62 (m, 4 H), 1.40-1.14 (m, 15 H), 0.17 (s, 3 H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ 173.3, 145.5, 135.3, 80.1, 76.4, 68.9, 58.1, 58.1, 32.4, 31.1, 31.0, 29.2, 28.8, 27.8, 27.3, 25.5, 24.2, 24.0, 23.5, 18.3, -4.3; HRMS (ESI-TOF) calcd. for $C_{22}H_{40}O_5Si$ $[M+H]^+$ 413.27178, found 413.27159.

Protodesilylation of **17** (mixture of *Z* and *E* isomers with a ratio of 8:92) gave rise to **17b** as an inseparable mixture of *Z* and *E* isomers with a ratio of 90:10 which is determined by ^{1}H NMR analysis.

19a and **19** reported earlier as **1b** and **2b**.

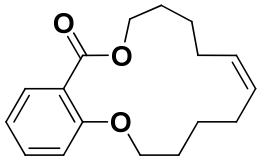


(*Z*)-3,4,7,8,9,10-Hexahydrobenzo[*b*][1,5]dioxacyclotetradecin-12(2*H*)-one (19b)

Protodesilylation of 91 mg **19** (0.22 mmol) followed by column chromatography (gradient 0 – 20% ethyl acetate/hexane) gave rise to 35 mg of the title compound. Yield 64% (colorless oil); IR (neat, cm^{-1}) 3009,

2935, 2865, 1703, 1601, 1581, 1490, 1453, 1384, 1354, 1302, 1250, 1165, 1132, 1097, 1049, 1015, 975; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 7.78-7.76 (m, 1 H), 7.44-7.40 (m, 1 H), 6.98-6.93 (m, 2 H), 5.68 (dt, *J* = 10.0, 8.2 Hz, 1 H), 5.48 (dt, *J* = 10.0, 8.2 Hz, 1 H), 4.43 (t, *J* = 6.0 Hz, 2 H), 4.09 (t, *J* = 5.2 Hz, 2 H), 2.29 (dt, *J* = 7.8, 7.8 Hz, 2 H), 2.13-2.08 (m, 2 H), 1.85-1.79 (m, 4 H), 1.69-1.63 (m, 2 H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ 168.5, 157.5, 133.1, 132.2, 130.1, 130.0, 121.3, 120.0, 112.2, 66.9, 63.8, 29.8, 27.6, 25.7, 25.4, 23.5; HRMS (ESI-TOF) calcd. for $C_{16}H_{20}O_3$ $[M+H]^+$ 261.14852, found 261.14455.

20a and **20** reported earlier as **3b** and **4b**.



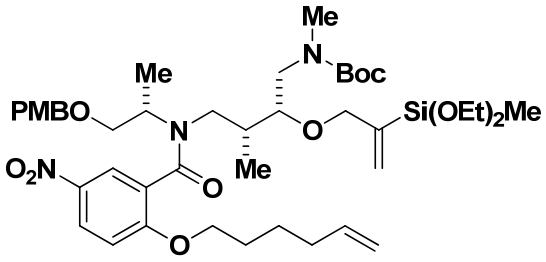
(*Z*)-4,5,8,9,10,11-Hexahydro-2*H*-benzo[*b*][1,5]dioxacyclopentadecin-13(3*H*)-one (20b)

Protodesilylation of **20** (60 mg, 0.14 mmol) followed by column chromatography (gradient 0 – 20% ethyl acetate/hexane) gave rise to 17 mg of the title compound. Yield 46% (colorless oil); IR (neat, cm^{-1}) 3007, 2936, 2862, 1698, 1601, 1491, 1452, 1384, 1300, 1249, 1164, 1131, 1097, 1050, 958; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 7.71-7.69 (m, 1 H), 7.42-7.38 (m, 1 H), 6.97-6.94 (m, 1 H), 6.91 (d, *J* = 8.0 Hz, 1 H), 5.55 (dt, *J* = 10.8, 7.4 Hz, 1 H), 5.50 (dt, *J* = 10.8, 7.4 Hz, 1 H), 4.40 (t, *J* = 6.0 Hz, 2 H), 4.04 (t, *J* = 5.2 Hz, 2 H), 2.14-2.09 (m, 4 H), 1.85-1.77 (m, 4 H), 1.65-1.59 (m, 2 H), 1.58-1.52 (m, 2 H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ 168.3, 157.6, 132.7, 131.2, 130.3, 129.8, 121.5, 120.0, 112.3, 68.3, 64.5, 28.6, 27.9, 27.1, 26.9, 26.4, 26.0; HRMS (ESI-TOF) calcd. for $C_{17}H_{22}O_3$ $[M+Na]^+$ 297.14612, found 297.14667.

Synthesis of compound 18 and 21

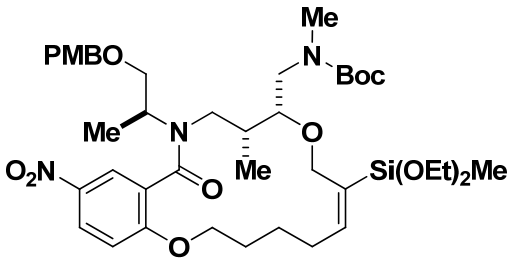
Following the reported procedure,[3] the alkyne substrates were synthesized. Hydrosilylation of the alkynes gave rise to the corresponding alkenyl siloxane **18a** and **21a**, which were subjected to the RCM reaction.

Note: The ^{1}H and ^{13}C NMR spectra of many of these compounds were extremely complicated owing to the various combinations of rotamers, and conformers. Efforts to completely coalesce the resonances through variable temperature NMR (up to 110 ^oC) were unsuccessful. Despite their complexity, all spectra are for single compounds that were larger than 95% pure by LC/MS.



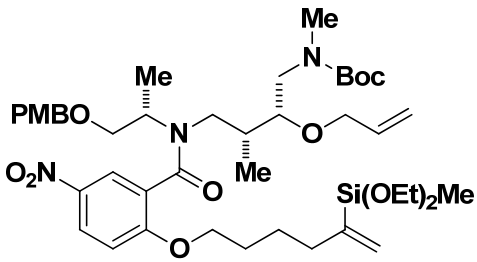
***tert*-Butyl (2*R*,3*R*)-2-(2-(diethoxy(methyl)silyl)allyloxy)-4-(2-(hex-5-enyloxy)-*N*-((*S*)-1-(4-methoxybenzyloxy)propan-2-yl)-5-nitrobenzamido)-3-methylbutyl(methyl)carbamate (18a)**

Yield 69% (pale yellow oil); IR (neat, cm^{-1}) 2973, 2932, 1693, 1640, 1612, 1588, 1516, 1458, 1391, 1365, 1341, 1272, 1251, 1160, 1078, 1036, 952; HRMS (ESI-TOF) calcd. for $C_{43}H_{67}N_3O_{11}Si$ $[M+Na]^+$ 852.44371, found 852.44396; $[\alpha]_D^{21}$ = -25.5 (c = 2.2, $CHCl_3$).



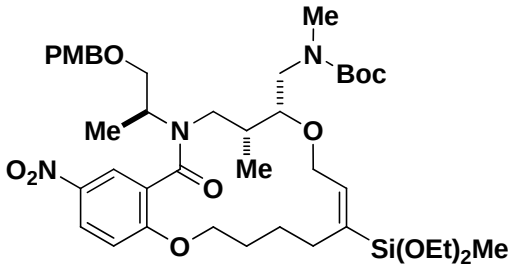
***tert*-Butyl ((10*R*,11*R*,*E*)-7-(diethoxy(methyl)silyl)-13-((*S*)-1-(4-methoxybenzyloxy)propan-2-yl)-11-methyl-16-nitro-14-oxo-2,3,4,5,8,10,11,12,13,14-decahydrobenzo[*b*][1,9,5]dioxaazacyclohexadecin-10-yl)methyl(methyl)carbamate (18)**

Z/*E* ratio is less than 1:99. Yield 47% (pale yellow oil); IR (neat, cm^{-1}) 2972, 2934, 1692, 1633, 1614, 1588, 1516, 1468, 1392, 1365, 1341, 1302, 1271, 1251, 1159, 1105, 1080, 1036, 1010, 986, 953; HRMS (ESI-TOF) calcd. for $C_{41}H_{63}N_3O_{11}Si$ $[M+Na]^+$ 824.41241, found 824.41263; $[\alpha]_D^{21}$ = -16.4 (c = 7.6, $CHCl_3$).



***tert*-Butyl ((2*R*,3*R*)-2-(allyloxy)-4-(2-((5-(diethoxy(methyl)silyl)hex-5-en-1-yl)oxy)-*N*-((*S*)-1-((4-methoxybenzyl)oxy)propan-2-yl)-5-nitrobenzamido)-3-methylbutyl)(methyl)carbamate (21a)**

Yield 69% (pale yellow oil); IR (neat, cm^{-1}) 2973, 2934, 1694, 1940, 1612, 1588, 1516, 1457, 1391, 1365, 1340, 1272, 1252, 1162, 1078, 1036, 952; HRMS (ESI-TOF) calcd. for $C_{43}H_{67}N_3O_{11}Si$ $[M+Na]^+$ 852.44371, found 852.44378; $[\alpha]_D^{20}$ = -32.3 (c = 2.4, $CHCl_3$).



***tert*-Butyl (((10*R*,11*R*,*E*)-6-(diethoxy(methyl)silyl)-13-((*S*)-1-((4-methoxybenzyl)oxy)propan-2-yl)-11-methyl-16-nitro-14-oxo-2,3,4,5,8,10,11,12,13,14-decahydrobenzo[*b*][1,9,5]dioxaazacyclohexadecin-10-yl)methyl)(methyl)carbamate (21)**

Z/*E* ratio is 14:86. Yield 44% (pale yellow oil); IR (neat, cm^{-1}) 2972, 2934, 1689, 1636, 1612, 1588, 1515, 1463, 1391, 1365, 1340, 1273, 1252, 1164, 1104, 1078; HRMS (ESI-TOF) calcd. for $C_{41}H_{63}N_3O_{11}Si$ $[M+H]^+$ 802.43046, found 802.42662; $[\alpha]_D^{22}$ = -29.8 (c = 3.2, $CHCl_3$).

The simple diolefinic substrate was synthesized and subjected to RCM reaction using catalyst **A**. A mixture of both stereoisomers was obtained. The *Z*/*E* ratio was analyzed to be 36:64 using SFC/MS chromatography (**Figure S2**, first trace). SFC: Chiralpak® AD-H column; 25% ^{i}PrOH, 75% sfCO2, 10 minutes run length, $t_R^{(Z)}$ = 3.77 min, area = 36%, $t_R^{(E)}$ = 5.12 min, area = 64%.

In order to confirm the geometry of the double bond within alkenyl siloxane products **18** and **21**, protodesilylation reaction was performed to generate the simple olefins. The *Z*/*E* ratio of desilylated product **18b** from compound **18** was larger than 99:1 (**Figure S2**, third trace, $t_R^{(Z)}$ = 3.70 min, area = 100%). Due to highly rotameric nature of compound **18b**, VT NMR was performed in C_6D_6 at 80 °C. The coupling constant was measured to be 10.5 Hz which is characteristic of *Z* olefin. Since the protodesilylation reaction is stereospecific, the configuration of compound **18** was *E*. The *Z*/*E* ratio of desilylated product from compound **21** was 86:14 (**Figure S2**, second trace, $t_R^{(Z)}$ = 3.83 min, area = 86%, $t_R^{(E)}$ = 5.33 min, area = 14%), which indicated that the *Z*/*E* ratio of compound **21** is 14:86. In both cases, the siloxyl group was able to overcome the intrinsic selectivity favoring the formation of the *E* olefin. However, the positions of the siloxyl group had different influences on the selectivity of the olefin geometry within the product.

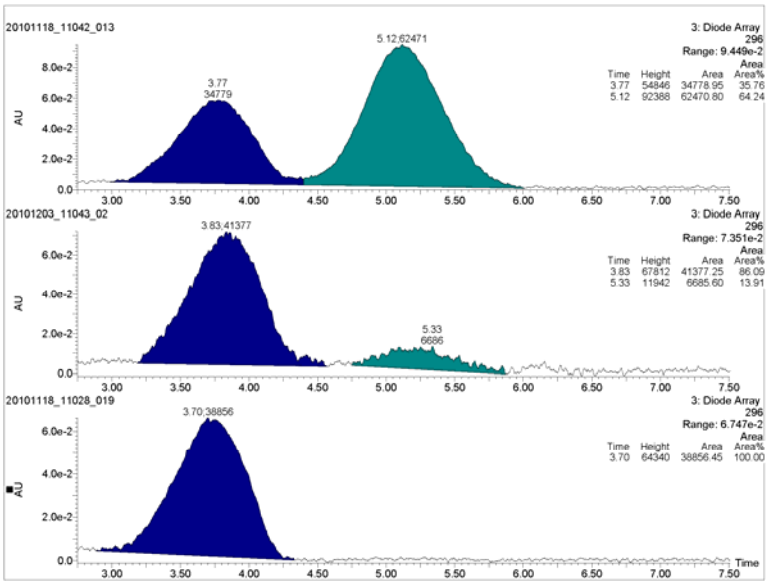
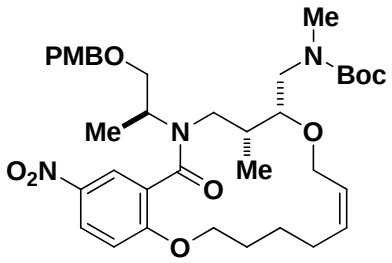


Figure S2. SFC/MS chromatography of olefin product from a) RCM of simple diolefinic substrate (first trace), b) protodesilylation of compound **21** (second trace), and c) protodesilylation of compound **18** (third trace).



***tert*-Butyl (((10*R*,11*R*,*Z*)-13-((*S*)-1-((4-methoxybenzyl)oxy)propan-2-yl)-11-methyl-16-nitro-14-oxo-2,3,4,5,8,10,11,12,13,14-decahydrobenzo[*b*][1,9,5]dioxaazacyclohexadecin-10-yl)methyl)(methyl)carbamate (18b)**

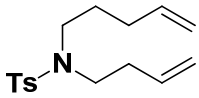
Yield 86% (pale yellow oil); IR (neat, cm^{-1}) 2936, 2862, 1690, 1633, 1588, 1515, 1464, 1392, 1366, 1341, 1272, 1250, 1159, 1104, 1036, 979; HRMS (ESI-TOF) calcd. for $C_{36}H_{51}N_3O_9$ $[M+Na]^+$ 692.35175, found 692.35064; $[\alpha]_D^{21}$ = -9.9 (c = 4.6, $CHCl_3$).

Protodesilylation of **21** generated **18b** with 60% yield

F. Influence of the silyl group on the specificity and stereoselectivity of RCM reactions.

Simple di-olefinic substrates were synthesized and subjected to two different reaction conditions: **I**, 20 mol% cat. **A**, 20 mol% 1,4-benzoquinone, toluene, 2 mM, 35 ^oC, 12 hours; **II**, 10 mol% Grubbs II, 20 mol% 1,4-benzoquinone, toluene, 2 mM, 35 ^oC, 12 hours. The reaction outcome was analyzed by proton NMR study of the crude mixture using $CDCl_3$ or C_6D_6 as solvent. Since the outcomes under both conditions are very similar, only expanded region of the proton NMR spectrum from condition **II** was shown here. The

resonance of the olefinic proton corresponding to the *cis* olefin was known from the protodesilylation of alkenyl siloxane intermediate. The resonance of the olefinic proton corresponding to the *trans* olefin was rigorously analyzed when the reaction is *trans* selective



***N*-(but-3-en-1-yl)-4-methyl-*N*-(pent-4-en-1-yl)benzenesulfonamide (22)**

IR (neat, cm^{-1}) 3077, 2977, 2929, 2869, 1641, 1599, 1494, 1458, 1340, 1158, 1091, 993, 958; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 7.68 (d, *J* = 8.0 Hz, 2 H), 7.28 (d, *J* = 8.0 Hz, 2 H), 5.80-5.66 (m, 2 H), 5.06-4.96 (m, 4 H), 3.16 (t, *J* = 7.5 Hz, 2 H), 3.11 (t, *J* = 7.5 Hz, 2 H), 2.41 (s, 3 H), 2.28 (dt, *J* = 7.3, 7.3 Hz, 2 H), 2.04 (dt, *J* = 7.1, 7.1 Hz, 2 H), 1.63 (tt, *J* = 7.5, 7.5 Hz, 2 H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ 143.0, 137.4, 136.9, 134.6, 129.6, 127.1, 117.0, 115.2, 47.9, 47.7, 33.3, 30.7, 27.8, 21.4; HRMS (ESI-TOF) calcd. for $C_{16}H_{23}NO_2S$ $[M+Na]^+$ 316.13417, found 316.13501.

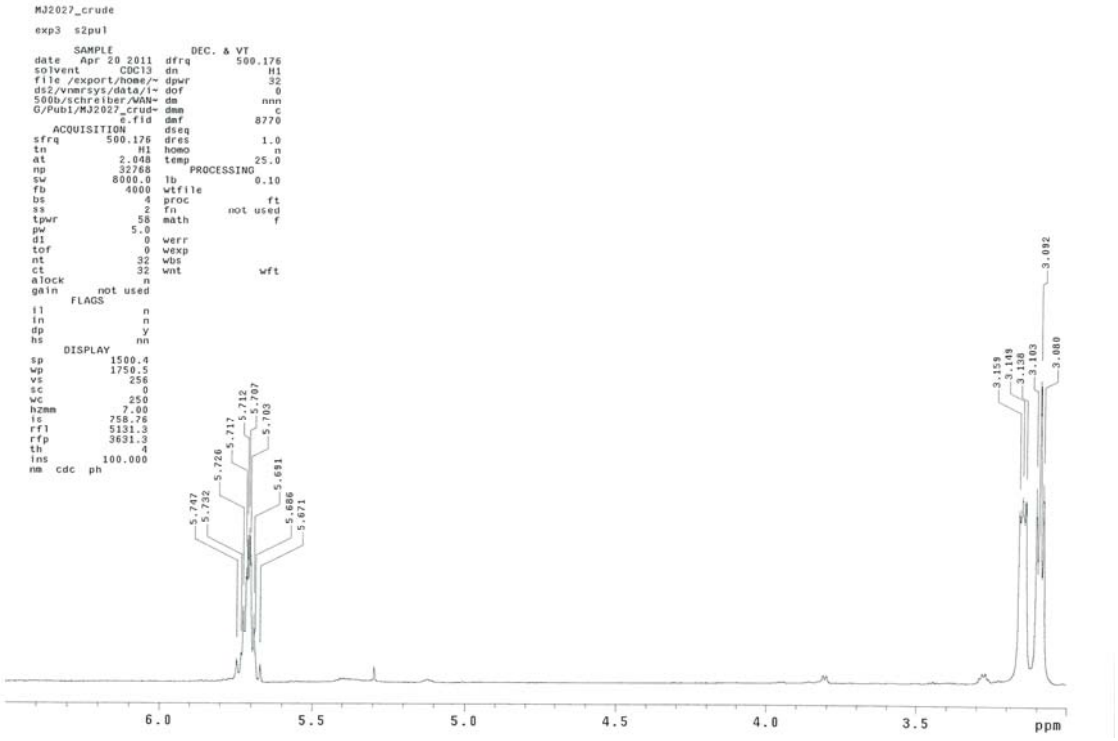
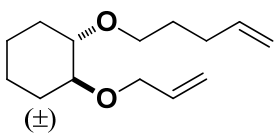


Figure S3. ^{1}H NMR spectrum (expansion of 3.0 to 6.5ppm) of reaction mixture of **22** under condition II.



(±)

(1*S*,2*S*)-1-(allyloxy)-2-(pent-4-en-1-yloxy)cyclohexane and its enantiomer (23)

IR (neat, cm^{-1}) 3078, 2934, 2861, 1642, 1450, 1366, 1315, 1271, 1243, 1208, 1161, 1106, 993; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 5.97-5.89 (m, 1 H), 5.86-5.78 (m, 1 H), 5.29-5.26 (m, 1 H), 5.14-5.12 (m, 1 H), 5.04-5.00 (m,

1 H), 4.96-4.94 (m, 1 H), 4.16-4.10 (m, 2 H), 3.60-3.52 (m, 2 H), 3.23-3.14 (m, 2 H), 2.15-2.11 (m, 2 H), 1.98-1.95 (m, 2 H), 1.68-1.62 (m, 4 H), 1.31-1.17 (m, 4 H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ 138.5, 135.8, 116.1, 114.5, 81.5, 80.8, 71.0, 69.2, 30.4, 30.2, 29.5, 23.6, 23.6; HRMS (ESI-TOF) calcd. for $C_{14}H_{24}O_2$ $[M+Na]^+$ 247.16685, found 247.16675.

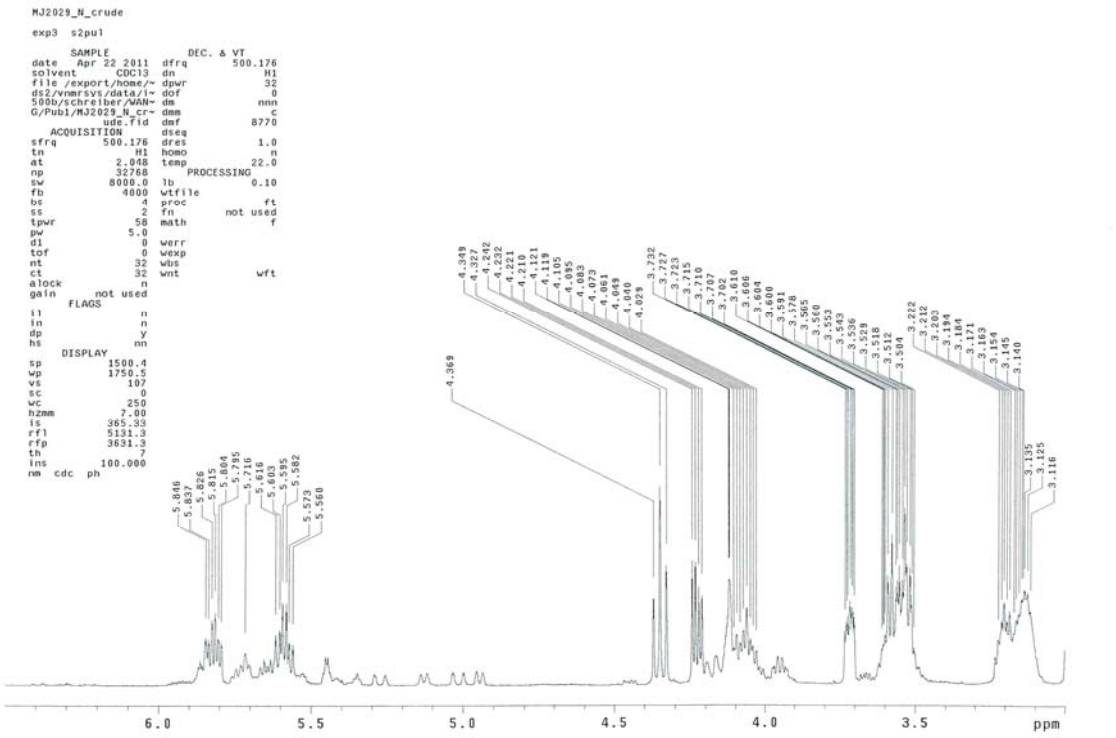
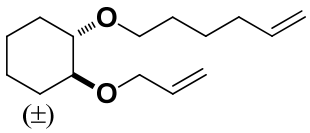


Figure S4. ^{1}H NMR spectrum (expansion of 3.0 to 6.5ppm) of reaction mixture of **23** under condition II.



(±)

(1*S*,2*S*)-1-(allyloxy)-2-(hex-5-en-1-yloxy)cyclohexane and its enantiomer (24)

IR (neat, cm^{-1}) 3077, 2933, 1642, 1450, 1366, 1244, 1098; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 5.97-5.89 (m, 1 H), 5.85-5.77 (m, 1 H), 5.29-5.26 (m, 1 H), 5.14-5.12 (m, 1 H), 5.01-4.98 (m, 1 H), 4.95-4.93 (m, 1 H), 4.16-4.08 (m, 2 H), 3.59-3.51 (m, 2 H), 3.22-3.14 (m, 2 H), 2.06 (ddd, *J* = 7.0, 7.0, 7.0 Hz, 2 H), 1.97-1.95 (m, 2 H), 1.64-1.55 (m, 4 H), 1.49-1.43 (m, 2 H), 1.31-1.17 (m, 4 H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ 138.9, 135.8, 116.1, 114.4, 81.5, 80.8, 71.1, 69.8, 33.4, 30.4, 30.2, 29.8, 25.5, 23.6, 23.6; HRMS (ESI-TOF) calcd. for $C_{15}H_{26}O_2$ $[M+Na]^+$ 261.18250, found 261.18388.

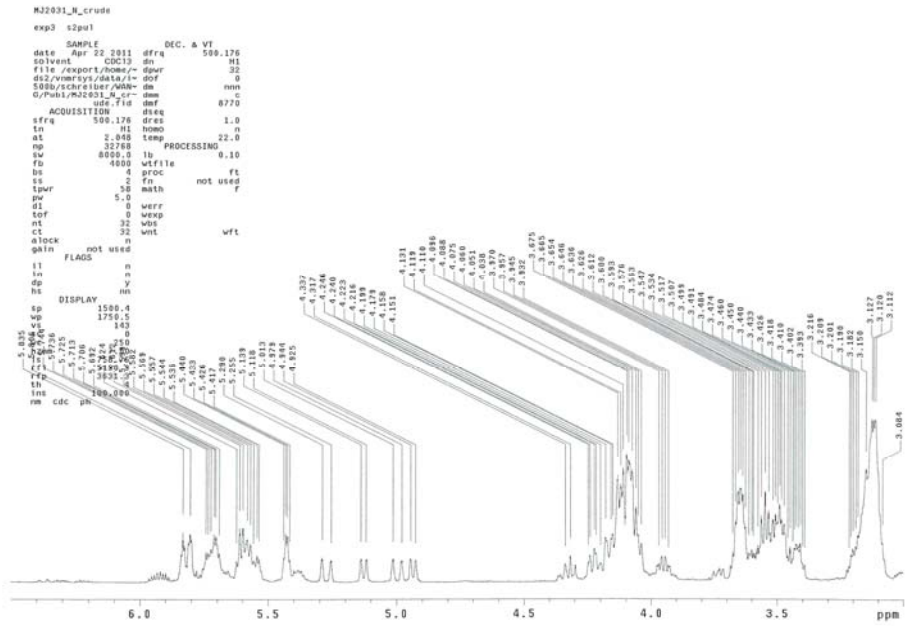


Figure S5. [1]H NMR spectrum (expansion of 3.0 to 6.5ppm) of reaction mixture of **24** under condition II.

The monocyclized *Z*-alkene compound **7b** (corresponding to what would be the monocyclized product of the RCM of **24**) obtained from protodesilylation of compound **7** was subjected to reaction condition II using second generation Grubbs catalyst. It was almost completely consumed to generate dimers and oligomers (**Figure S6**).

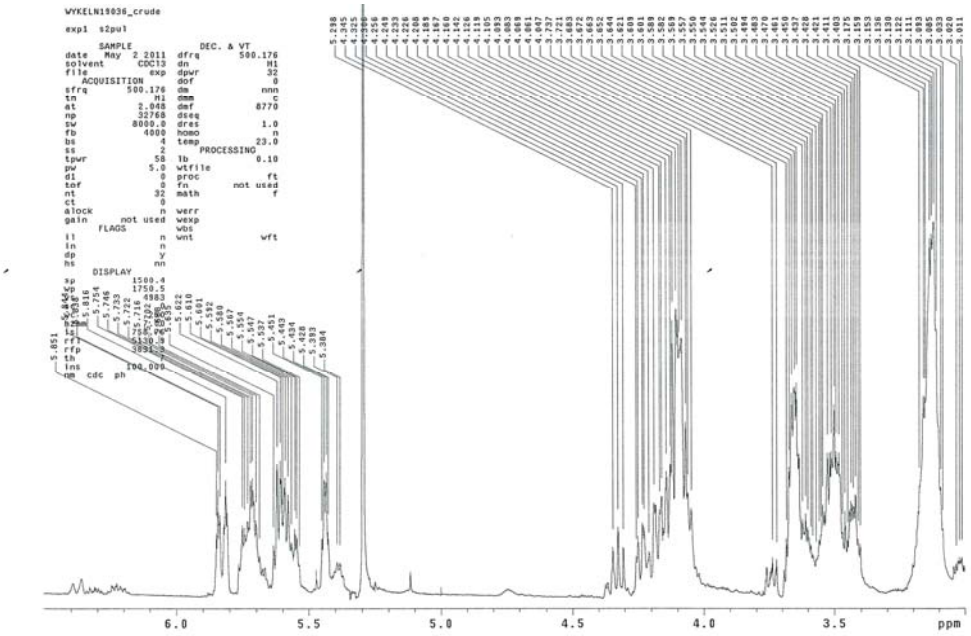
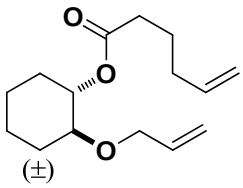


Figure S6. [1]H NMR spectrum (expansion of 3.0 to 6.5ppm) of reaction mixture of **7b** under condition II.



(±)

(1*S*,2*S*)-2-(allyloxy)cyclohexyl hex-5-enoate and its enantiomer (25)

IR (neat, cm^{-1}) 3078, 2938, 2863, 1734, 1642, 1453, 1367, 1246, 1175, 1101, 994; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 5.90-5.74 (m, 2 H), 5.26-5.23 (m, 1 H), 5.13-5.11 (m, 1 H), 5.04-4.96 (m, 2 H), 4.80-4.75 (m, 1 H), 4.10-4.06 (m, 1 H), 4.02-3.98 (m, 1 H), 3.29 (ddd, *J* = 8.5, 8.5, 4.0 Hz, 1 H), 2.31 (dd, *J* = 7.2, 7.2 Hz, 2 H), 2.09 (ddd, *J* = 7.0, 7.0, 7.0 Hz, 2 H), 2.00-1.97 (m, 2 H), 1.76-1.63 (m, 4 H), 1.40-1.20 (m, 4 H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ 172.9, 137.8, 135.3, 116.2, 115.2, 78.4, 74.8, 70.4, 33.9, 33.0, 29.9, 29.8, 24.1, 23.2; HRMS (ESI-TOF) calcd. for $C_{15}H_{24}O_3$ $[M+Na]^+$ 275.16177, found 275.16271.

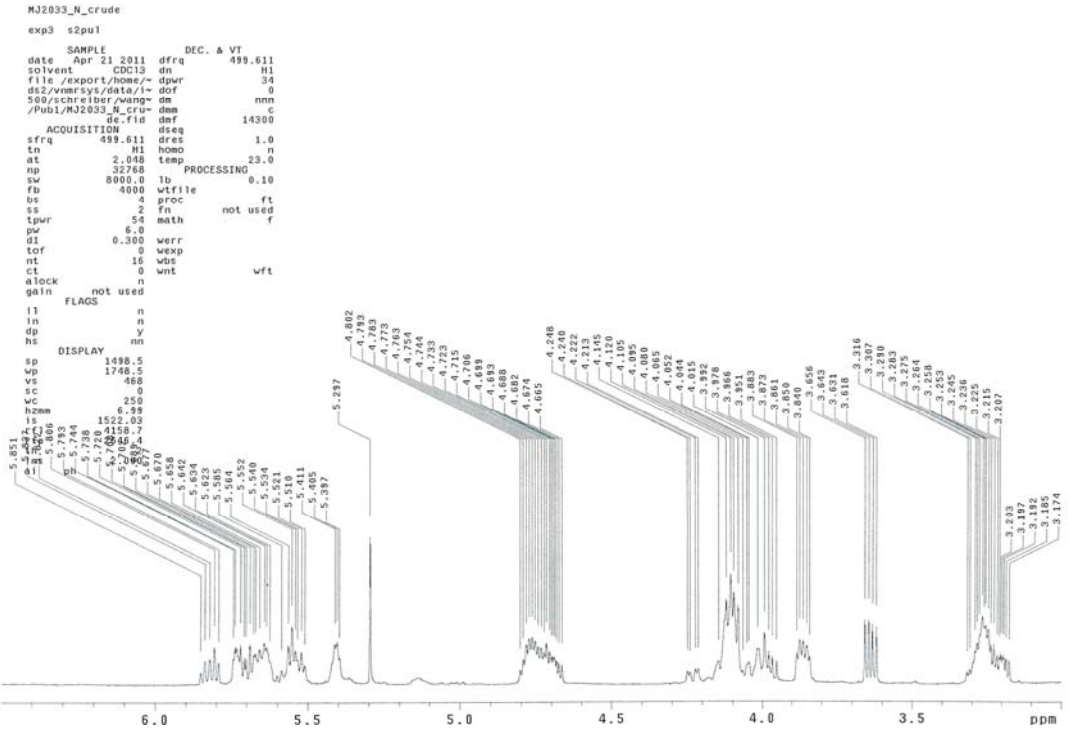


Figure S7. ^{1}H NMR spectrum (expansion of 3.0 to 6.5ppm) of reaction mixture of **25** under condition II.

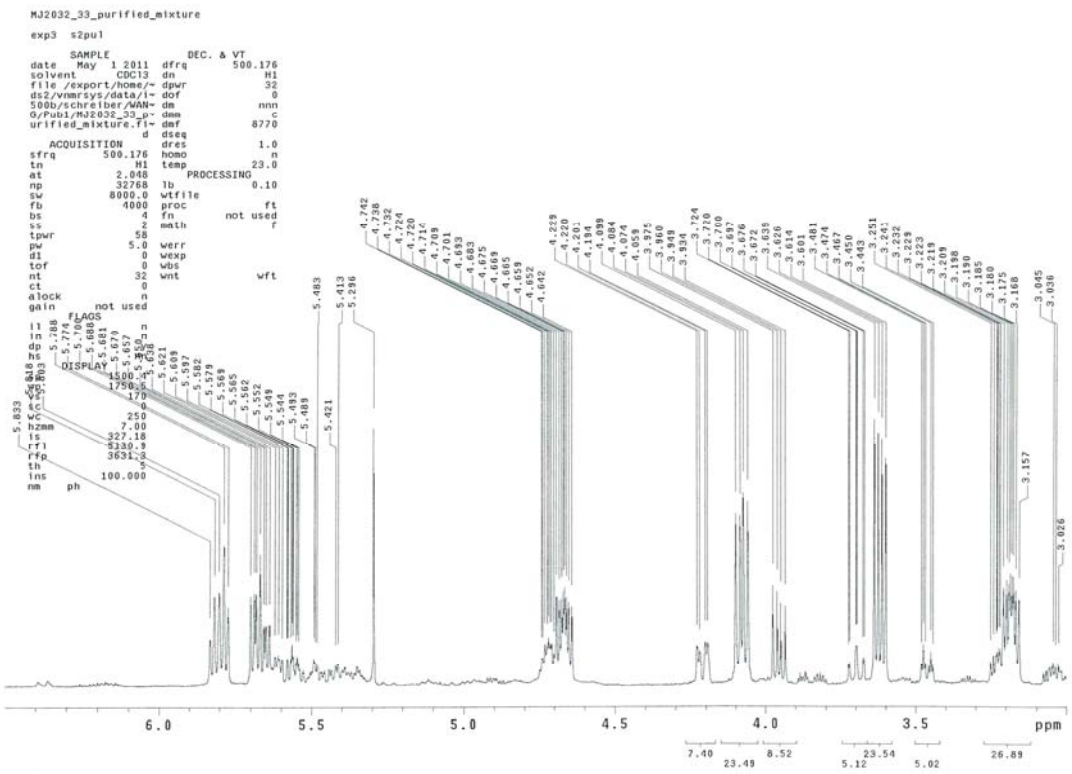
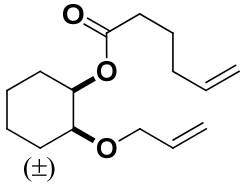


Figure S8. ^{1}H NMR spectrum of purified monocyclized product mixture from reaction of **25** for ratio determination.



(±)

(1*R*,2*S*)-2-(allyloxy)cyclohexyl hex-5-enoate and its enantiomer (26)

IR (neat, cm^{-1}) 3078, 2939, 2861, 1732, 1642, 1450, 1363, 1247, 1175, 1089, 994; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 5.91-5.83 (m, 1 H), 5.80-5.73 (m, 1 H), 5.27-5.23 (m, 1 H), 5.14-5.11 (m, 1 H), 5.10-5.08 (m, 1 H), 5.03-4.96 (m, 2 H), 4.04 (dd, *J* = 13.0, 5.5 Hz, 1 H), 3.97 (dd, *J* = 13.0, 5.7 Hz, 1 H), 3.47-3.46 (m, 1 H), 2.34 (dd, *J* = 7.5, 7.5 Hz, 2 H), 2.11-2.07 (m, 2 H), 1.90-1.85 (m, 1 H), 1.81-1.64 (m, 4 H), 1.60-1.46 (m, 3 H), 1.42-1.27 (m, 2 H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ 173.1, 137.8, 135.2, 116.5, 115.2, 76.0, 70.9, 69.6, 33.9, 33.0, 27.9, 27.8, 24.2, 22.1, 21.6; HRMS (ESI-TOF) calcd. for $C_{15}H_{24}O_3$ $[M+Na]^+$ 275.16177, found 275.16316.

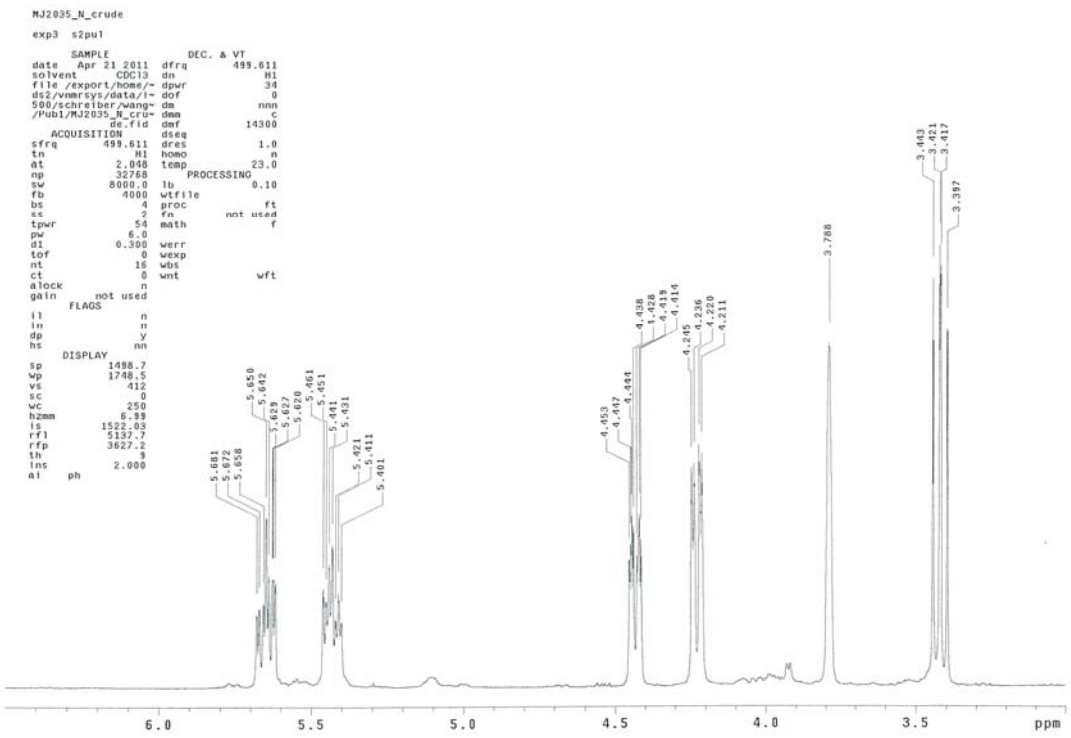
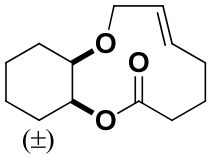
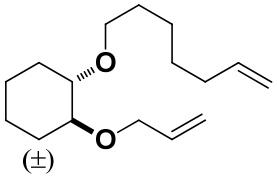


Figure S9. ^{1}H NMR spectrum (expansion of 3.0 to 6.5ppm) of reaction mixture of **26** under condition II (the major product was purifiable and is reported as **26b**).



(9a*R*,13a*S*,*E*)-3,4,5,8,9a,10,11,12,13,13a-decahydro-2*H*-benzo[*b*][1,4]dioxacycloundecin-2-one and its enantiomer (26b)

Yield 73% (colorless oil); IR (neat, cm^{-1}) 2934, 2858, 1725, 1443, 1363, 1256, 1210, 1159, 1139, 1109, 1089, 1073, 1047, 980; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 5.64 (ddd, *J* = 15.0, 10.5, 4.0 Hz, 1 H), 5.42 (ddd, *J* = 15.0, 10.2, 5.5 Hz, 1 H), 4.44-4.40 (m, 1 H), 4.23-4.20 (dd, *J* = 13.0, 4.2 Hz, 1 H), 3.77 (bs, 1 H), 3.40 (dd, *J* = 12.5, 11.0 Hz, 1 H), 2.38-2.29 (m, 2 H), 2.12-2.07 (m, 1 H), 1.99-1.70 (m, 6 H), 1.56-1.47 (m, 2 H), 1.41-1.23 (m, 3 H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ 174.9, 132.0, 132.0, 75.1, 74.9, 72.2, 34.4, 33.6, 30.6, 25.8, 24.5, 24.2, 19.4; HRMS (ESI-TOF) calcd. for $C_{13}H_{20}O_3$ $[M+H]^+$ 225.14852, found 225.16079.



(1*S*,2*S*)-1-(allyloxy)-2-(hept-6-en-1-yloxy)cyclohexane and its enantiomer (27)

IR (neat, cm^{-1}) 3077, 2932, 2859, 1642, 1451, 1365, 1314, 1270, 1244, 1208, 1161, 1107, 994; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 5.97-5.89 (m, 1 H), 5.84-5.76 (m, 1 H), 5.29-5.25 (m, 1 H), 5.14-5.12 (m, 1 H), 5.01-4.97 (m, 1 H), 4.94-4.91 (m, 1H), 4.16-4.08 (m, 2 H), 3.59-3.50 (m, 2 H), 3.22-3.13 (m, 2 H), 2.07-2.03 (m, 2 H), 1.96-1.95 (m, 2 H), 1.65-1.54 (m, 4 H), 1.44-1.33 (m, 4 H), 1.30-1.15(m, 4 H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ 139.0, 135.8, 116.0, 114.2, 81.6, 80.8, 71.1, 69.9, 33.7, 30.4, 30.2, 30.2, 28.8, 25.7, 23.6, 23.6; HRMS (ESI-TOF) calcd. for $C_{16}H_{28}O_2$ $[M+Na]^+$ 275.19815, found 275.19975.

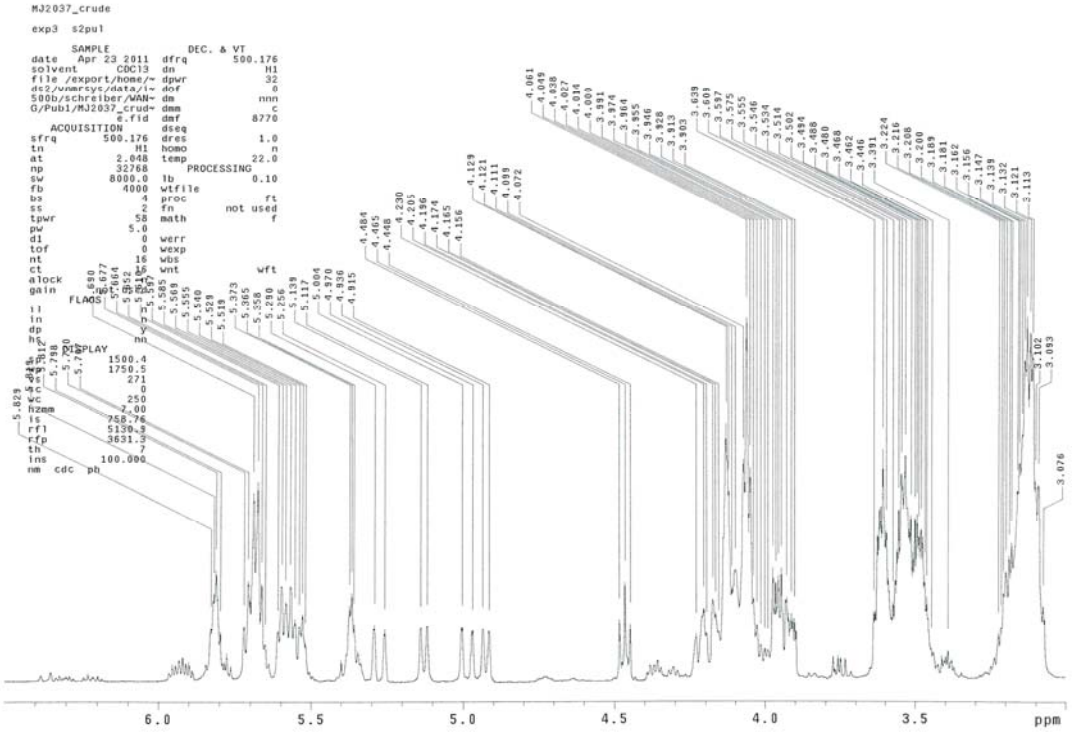
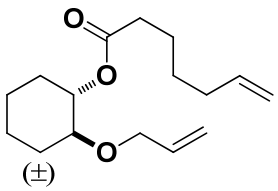


Figure S10. ^{1}H NMR spectrum (expansion of 3.0 to 6.5ppm) of reaction mixture of **27** under condition II.



(1*S*,2*S*)-2-(allyloxy)cyclohexyl hept-6-enoate and its enantiomer (28)

IR (neat, cm^{-1}) 3078, 2937, 2863, 1735, 1641, 1453, 1353, 1174, 1101, 994; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 5.90-5.74 (m, 2 H), 5.26-5.23 (m, 1 H), 5.13-5.11 (m, 1 H), 5.01-4.98 (m, 1 H), 4.95-4.93 (m, 1 H), 4.77 (ddd, *J* = 8.5, 8.5. 5.0 Hz, 1 H), 4.08 (dd, *J* = 7.8, 5.0 Hz, 1 H), 4.00 (dd, *J* = 7.8, 5.0 Hz, 1 H), 3.29 (ddd, *J* = 8.5, 8.5, 4.0 Hz, 1 H), 2.30 (dd, *J* = 7.2, 7.2 Hz, 2 H), 2.08-2.04 (m, 2 H), 1.99-1.97 (m, 2 H), 1.70-1.61 (m, 4 H), 1.46-1.20 (m, 6 H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ 173.0, 138.4, 135.3, 116.2, 114.6, 78.5, 74.8, 70.4, 34.5, 33.4, 29.9, 29.8, 28.3, 24.5, 23.2; HRMS (ESI-TOF) calcd. for $C_{16}H_{26}O_3$ $[M+Na]^+$ 289.17742, found 289.17766.

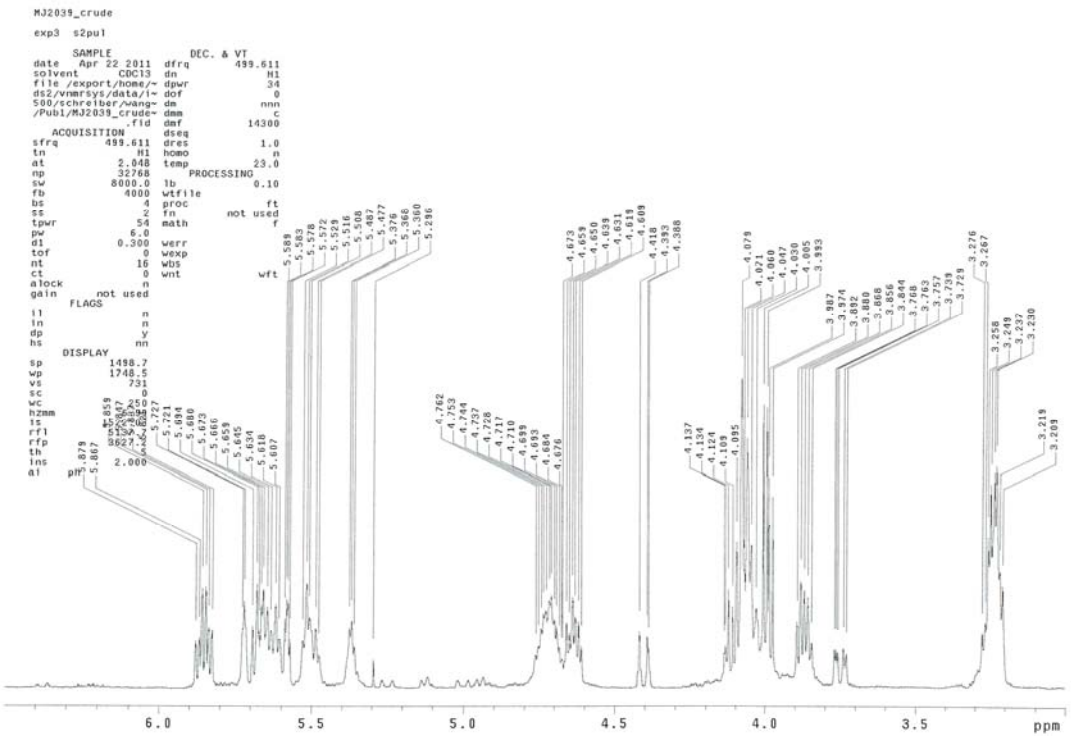
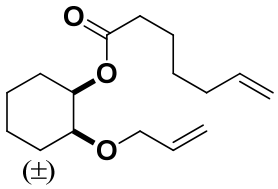


Figure S11. ^{1}H NMR spectrum (expansion of 3.0 to 6.5ppm) of reaction mixture of **28** under condition II.



(1*R*,2*S*)-2-(allyloxy)cyclohexyl hept-6-enoate and its enantiomer (29)

IR (neat, cm^{-1}) 3078, 2938, 2860, 1732, 1642, 1450, 1362, 1236, 1174, 1089, 994; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 5.91-5.75 (m, 2 H), 5.27-5.23 (m, 1 H), 5.14-5.08 (m, 2 H), 5.02-4.98 (m, 1 H), 4.95-4.93 (m, 1 H), 4.04 (dd, *J* = 8.0, 5.5 Hz, 1 H), 3.98 (dd, *J* = 8.0, 5.5 Hz, 1 H), 3.48-3.46 (m, 1 H), 2.34 (dd, *J* = 7.2, 7.2 Hz, 2 H), 2.08-2.04 (m, 2 H), 1.90-1.85 (m, 1 H), 1.81-1.75 (m, 1 H), 1.70-1.28 (m, 10 H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ 173.2, 138.5, 135.2, 116.5, 114.6, 76.0, 71.0, 69.6, 34.5, 33.4, 28.3, 27.9, 27.8, 24.5, 22.1, 21.7; HRMS (ESI-TOF) calcd. for $C_{16}H_{26}O_3$ $[M+Na]^+$ 289.17742, found 289.17867.

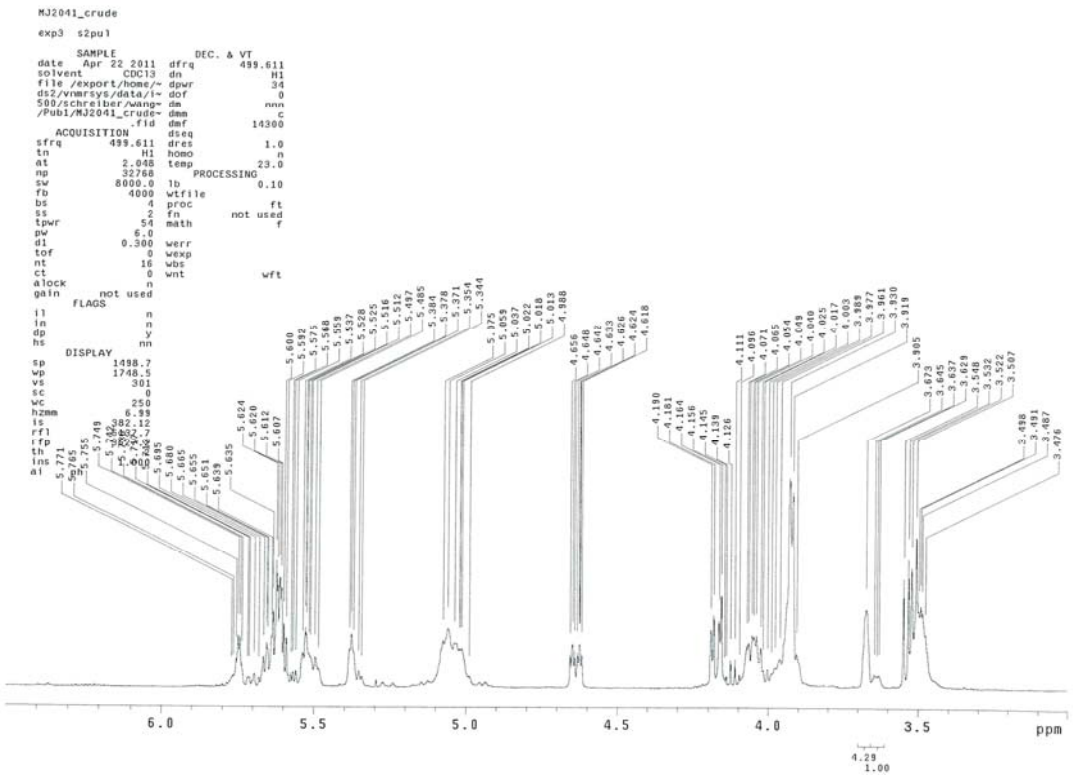


Figure S12. [1]H NMR spectrum (expansion of 3.0 to 6.5ppm) of reaction mixture of **29** under condition II.

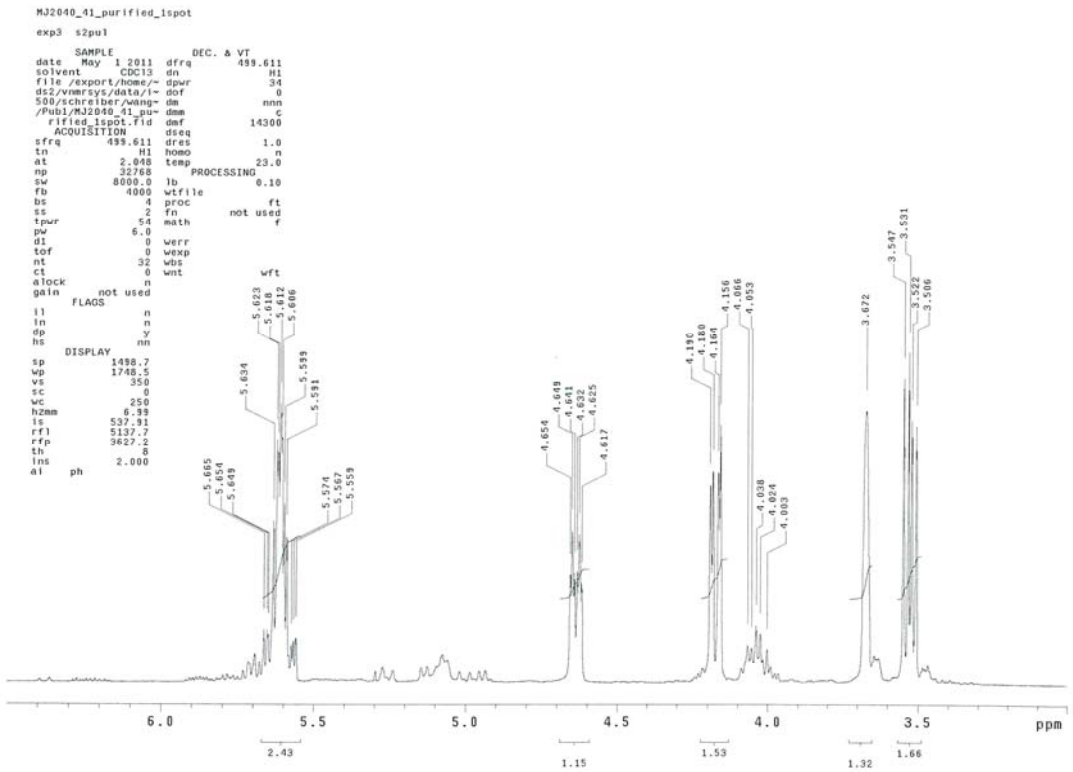
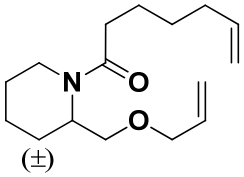


Figure S13. [1]H NMR spectrum of purified monocyclized product mixture from reaction of **29** for identification of *trans* isomer.



(±)-1-(2-((allyloxy)methyl)piperidin-1-yl)hept-6-en-1-one (30)

IR (neat, cm^{-1}) 3076, 2934, 2859, 1642, 1426, 1357, 1243, 1178, 1134, 1104, 1057, 1028, 992; The ^{1}H and ^{13}C NMR spectra of many of this compound was complicated owing to the combination of rotamers. HRMS (ESI-TOF) calcd. for $C_{16}H_{27}NO_2$ $[M+Na]^+$ 288.19340, found 288.19396.

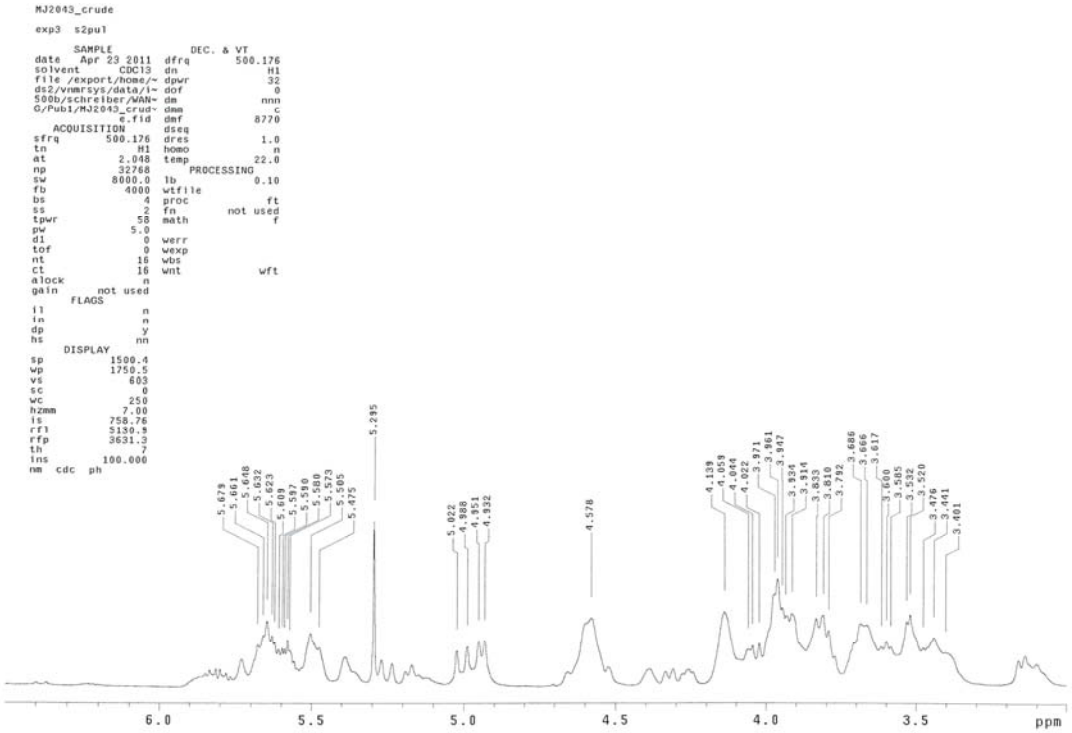
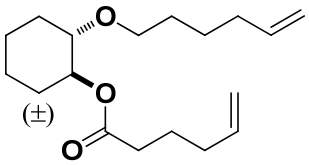


Figure S14. ^{1}H NMR spectrum (expansion of 3.0 to 6.5ppm) of reaction mixture of **30** under condition II.



(1*S*,2*S*)-2-(hex-5-en-1-yloxy)cyclohexyl hex-5-enoate and its enantiomer (31)

IR (neat, cm^{-1}) 3077, 2936, 2862, 1734, 1641, 1453, 1369, 1175, 1111; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 5.83-5.74 (m, 2 H), 5.04-4.92 (m, 4 H), 4.75 (ddd, *J* = 9.0, 9.0, 4.5 Hz, 1 H), 3.54 (ddd, *J* = 9.0, 6.5, 6.5 Hz, 1 H), 3.40 (ddd, *J* = 9.0, 6.5, 6.5 Hz, 1 H), 3.21 (ddd, *J* = 8.5, 8.5, 4.0 Hz, 1 H), 3.32-2.29 (m, 2 H), 2.11-2.02 (m, 4 H), 2.00-1.95 (m, 2 H), 1.76-1.63 (m, 4 H), 1.56-1.50 (m, 2 H), 1.46-1.38 (m, 2 H), 1.37-1.19(m, 4 H); ^{13}C-

NMR (125 MHz, $CDCl_3$) δ 172.9, 138.8, 137.8, 115.2, 114.4, 79.0, 74.7, 69.3, 33.9, 33.5, 33.0, 29.8, 29.6, 25.5, 24.2, 23.3; HRMS (ESI-TOF) calcd. for $C_{18}H_{30}O_3$ $[M+Na]^+$ 317.20872, found 317.20928.

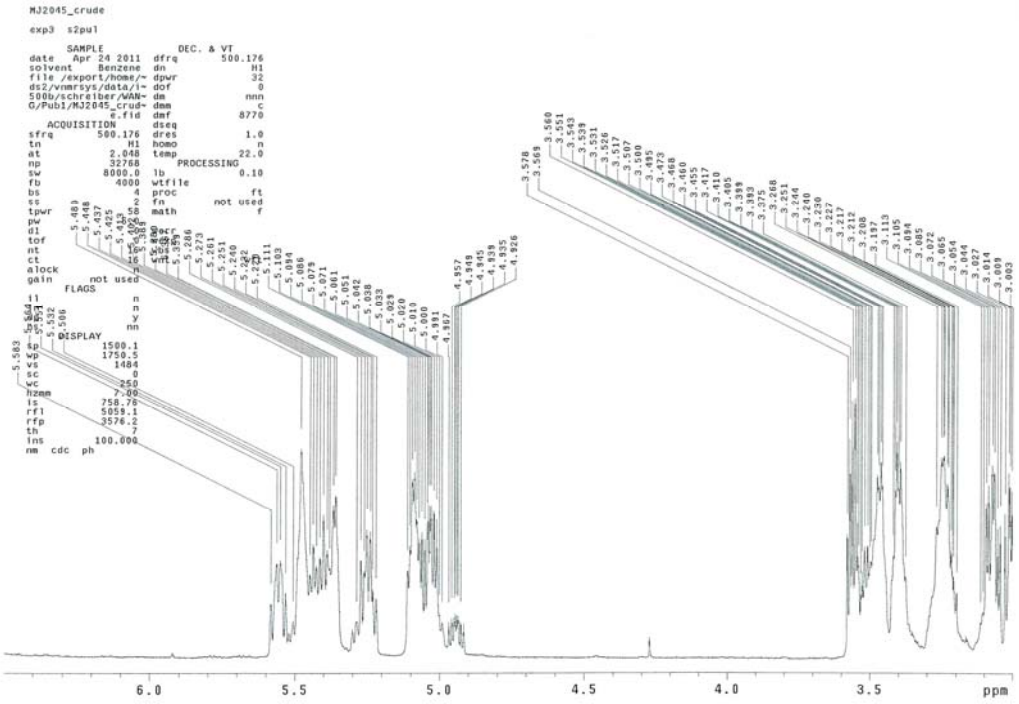


Figure S15. [1]H NMR spectrum (expansion of 3.0 to 6.5ppm) of reaction mixture of **31** under condition II.

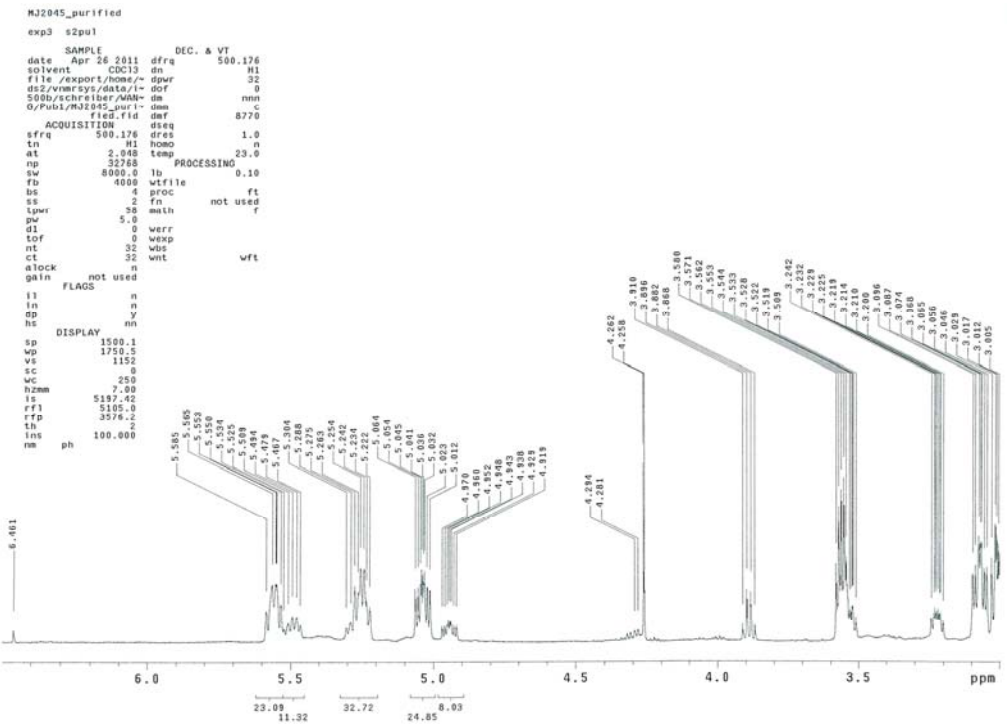
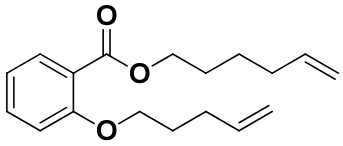


Figure S16. [1]H NMR spectrum of purified monocyclized product mixture from reaction of **31** for ratio determination.



Hex-5-en-1-yl 2-(pent-4-en-1-yloxy)benzoate (**32**)

IR (neat, cm^{-1}) 3077, 2976, 2940, 2870, 1728, 1704, 1641, 1601, 1583, 1491, 1469, 1452, 1416, 1386, 1302, 1251, 1164, 1133, 1080, 1049, 1013, 995; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 7.78-7.76 (m, 1 H), 7.44-7.41 (m, 1 H), 6.98-6.94 (m, 2 H), 5.89-5.77 (m, 2 H), 5.08-4.95 (m, 4 H), 4.30 (t, *J* = 7.0 Hz, 2 H), 4.04 (t, *J* = 6.5 Hz, 2 H), 2.30-2.25 (m, 2 H), 2.14-2.09 (m, 2 H), 1.96-1.90 (m, 2 H), 1.80-1.74 (m, 2 H), 1.58-1.52 (m, 2 H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ 166.7, 158.4, 138.4, 137.7, 133.1, 131.5, 120.9, 120.0, 115.2, 114.8, 113.1, 68.0, 64.7, 33.3, 30.0, 28.3, 28.2, 25.3; HRMS (ESI-TOF) calcd. for $C_{18}H_{24}O_3$ $[M+Na]^+$ 311.16177, found 311.16440.

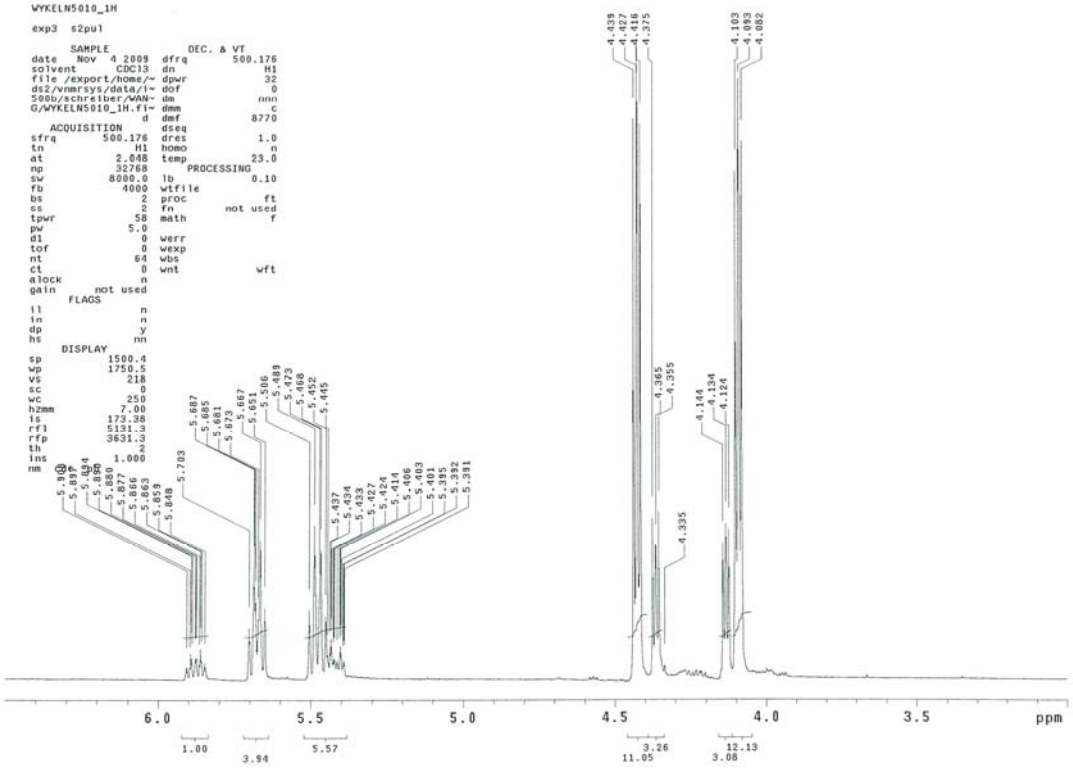
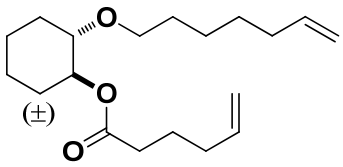


Figure S17. ^{1}H NMR spectrum (expansion of 3.0 to 6.5ppm) of reaction mixture of **32** under condition II.



(1*S*,2*S*)-2-(hept-6-en-1-yloxy)cyclohexyl hex-5-enoate and its enantiomer (33)

IR (neat, cm^{-1}) 3077, 2934, 2861, 1734, 1641, 1452, 1417, 1369, 1247, 1175, 1111, 1026, 994; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 5.83-5.74 (m, 2 H), 5.04-4.91 (m, 4 H), 4.75 (ddd, *J* = 9.0, 9.0, 4.5 Hz, 1 H), 3.53 (ddd, *J* = 9.5, 6.5, 6.5 Hz, 1 H), 3.39 (ddd, *J* = 9.0, 7.0, 7.0 Hz, 1 H), 3.20 (ddd, *J* = 9.0, 9.0, 4.0 Hz, 1 H), 2.32-2.29 (m, 2 H), 2.11-2.07 (m, 2 H), 2.05-2.01 (m, 2 H), 1.99-1.95 (m, 2 H), 1.76-1.63 (m, 4 H), 1.54-1.49 (m, 2 H), 1.41-1.19 (m, 8 H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ 172.9, 138.9, 137.8, 115.2, 114.2, 79.0, 74.8, 69.5, 33.9, 33.7, 33.0, 30.0, 29.8, 28.7, 25.6, 24.2, 23.3; HRMS (ESI-TOF) calcd. for $C_{19}H_{32}O_3$ $[M+H]^+$ 309.24242, found 309.24229.

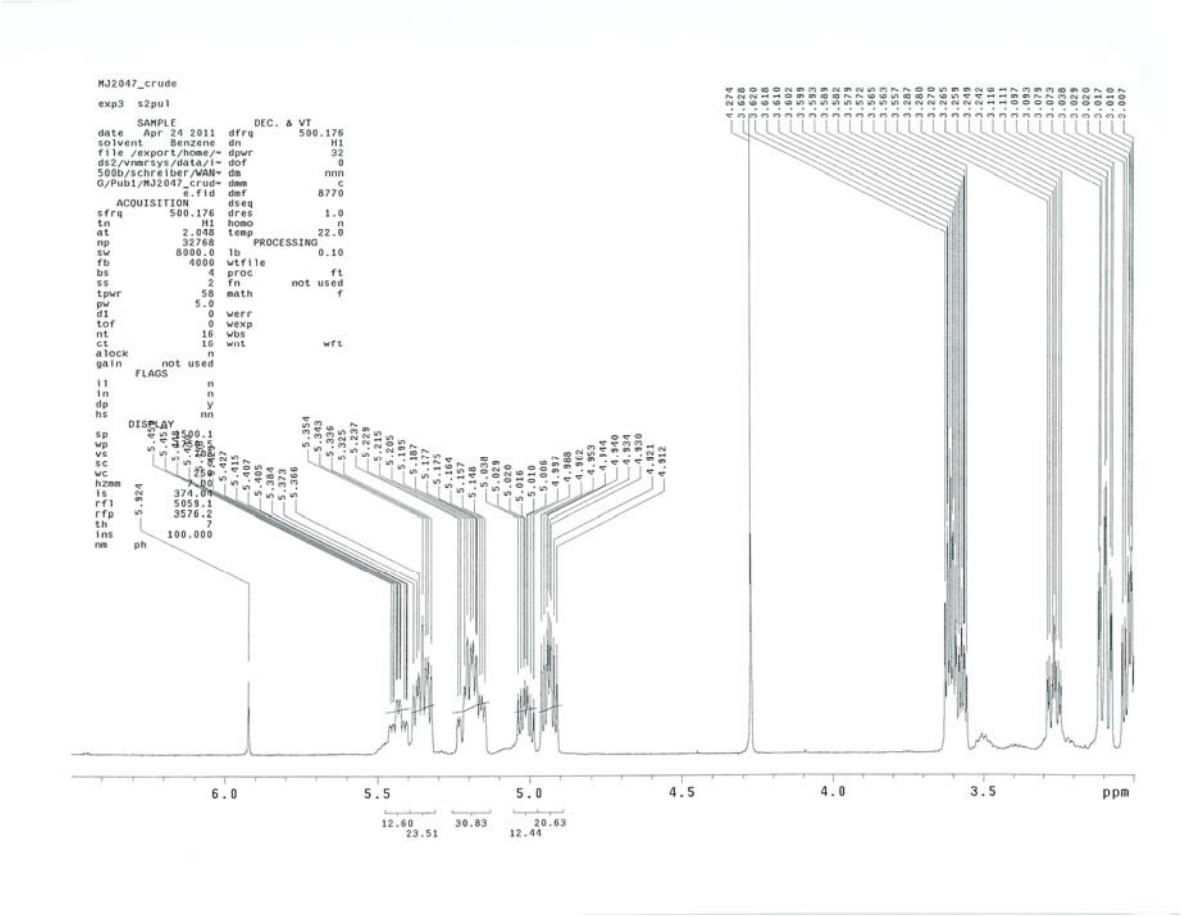
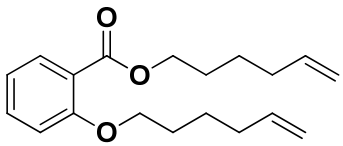


Figure S18. ^{1}H NMR spectrum (expansion of 3.0 to 6.5ppm) of reaction mixture of **33** under condition II.



Hex-5-en-1-yl 2-(hex-5-en-1-yloxy)benzoate (**34**)

IR (neat, cm^{-1}) 3076, 2937, 2862, 1728, 1704, 1640, 1601, 1583, 1491, 1469, 1453, 1386, 1302, 1250, 1164, 1133, 1079, 1049, 995, 953; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 7.78-7.76 (m, 1 H), 7.44-7.41 (m, 1 H), 6.97-6.94 (m, 2 H), 5.87-5.78 (m, 2 H), 5.05-5.01 (m, 2 H), 4.98-4.96 (m, 2 H), 4.30 (t, *J* = 6.8 Hz, 2 H), 4.03 (t, *J* = 6.5

Hz, 2 H), 2.15-2.10 (m, 4 H), 1.85 (tt, J = 7.1, 7.1 Hz, 2 H), 1.77 (tt, J = 7.2, 7.2 Hz, 2 H), 1.63-1.52 (m, 4 H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ 166.7, 158.4, 138.5, 138.4, 133.1, 131.5, 120.8, 120.0, 114.8, 114.7, 113.0, 68.6, 64.7, 33.4, 33.3, 28.6, 28.2, 25.3, 25.2; HRMS (ESI-TOF) calcd. for $C_{19}H_{26}O_3$ $[M+Na]^+$ 325.17742, found 325.17910.

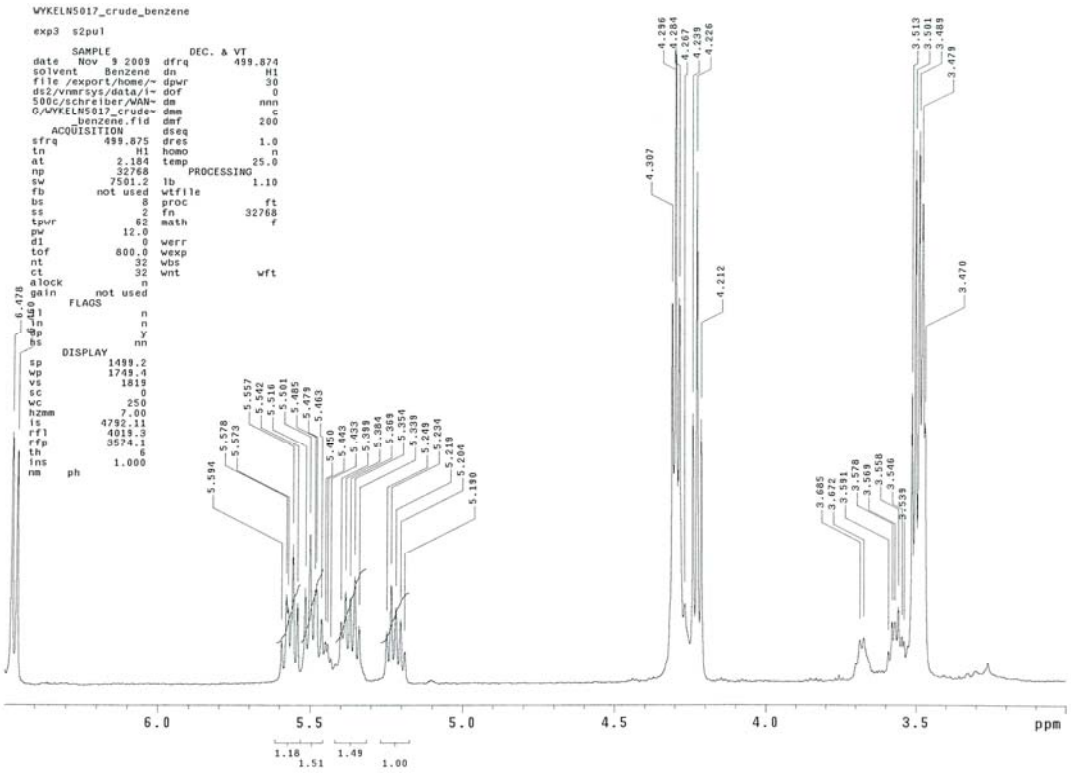


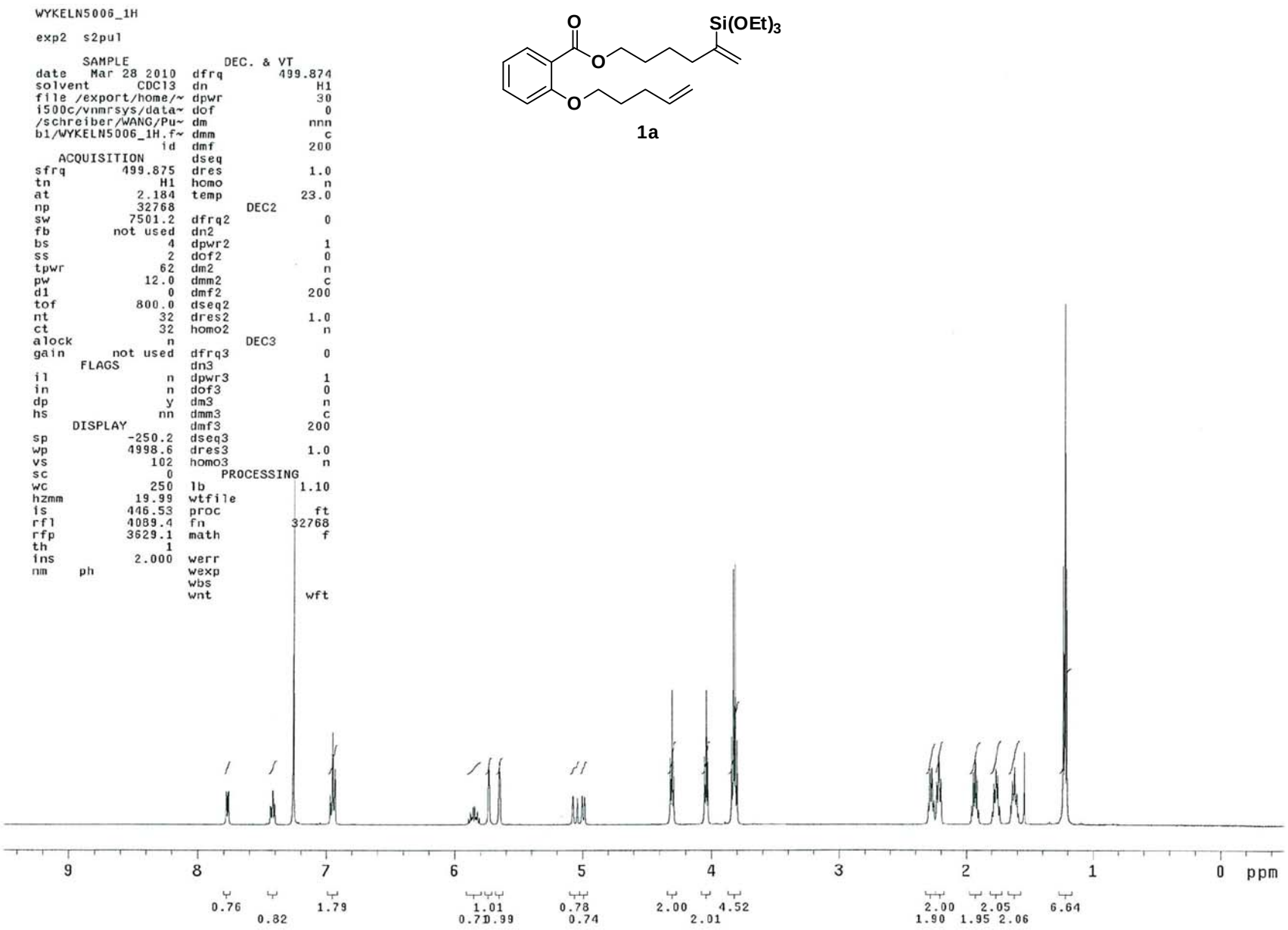
Figure S19. ^{1}H NMR spectrum (expansion of 3.0 to 6.5ppm) of reaction mixture of **34** under condition II.

References.

(1) (a) Trost, B. M.; Ball, Z. T. *J. Am. Chem. Soc.* **2005**, *127*, 17644-17655. (b) Trost, B. M.; Ball, Z. T. *J. Am. Chem. Soc.* **2001**, *123*, 12726-12727.

(2) (a) Furstner, A.; Radkowski, K. *Chem. Commun.* **2002**, 2182-2183. (b) Lacombe, F.; Radkowski, K.; Seidel, G.; Fürstner, A. *Tetrahedron* **2004**, *60*, 7315-7324.

(3) Marcaurelle, L. A.; Comer, E.; Dandapani, S.; Duvall, J. R.; Gerard, B.; Kesavan, S.; Lee, M. D.; Liu, H.; Lowe, J. T.; Marie, J.-C.; Mulrooney, C. A.; Pandya, B. A.; Rowley, A.; Ryba, T. D.; Suh, B.-C.; Wei, J.; Young, D. W.; Akella, L. B.; Ross, N. T.; Zhang, Y.-L.; Fass, D. M.; Reis, S. A.; Zhao, W.-N.; Haggarty, S. J.; Palmer, M.; Foley, M. A. *J. Am. Chem. Soc.* **2010**, *132*, 16962-16976.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gain not used
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dp y
hs nn
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8
7
6
5
4
3
2
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0
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0.99
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2.00
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2.05
1.95
2.06
6.64

1a

WYKELN5006_13C

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pw	4.2	dfrq2	0
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tof	2000.0	dpwr2	1
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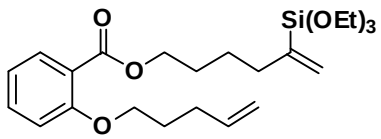

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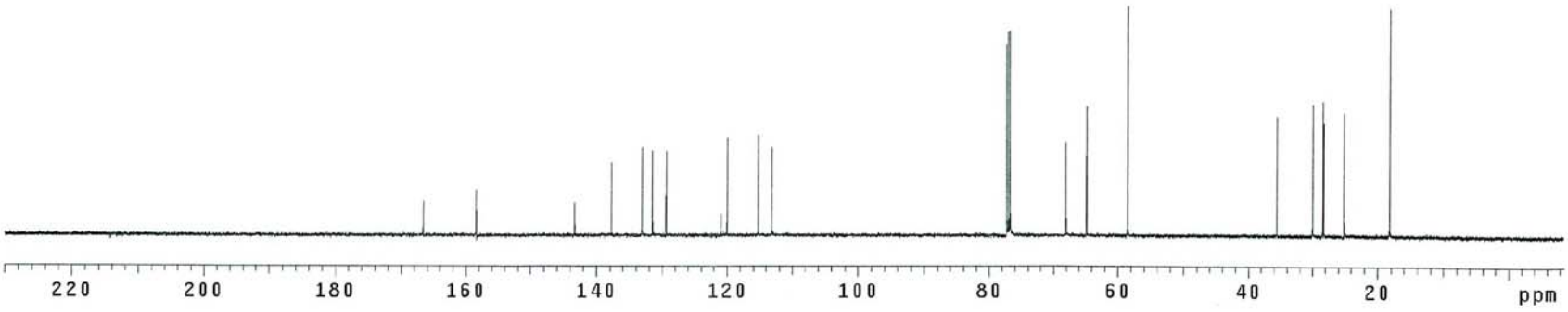

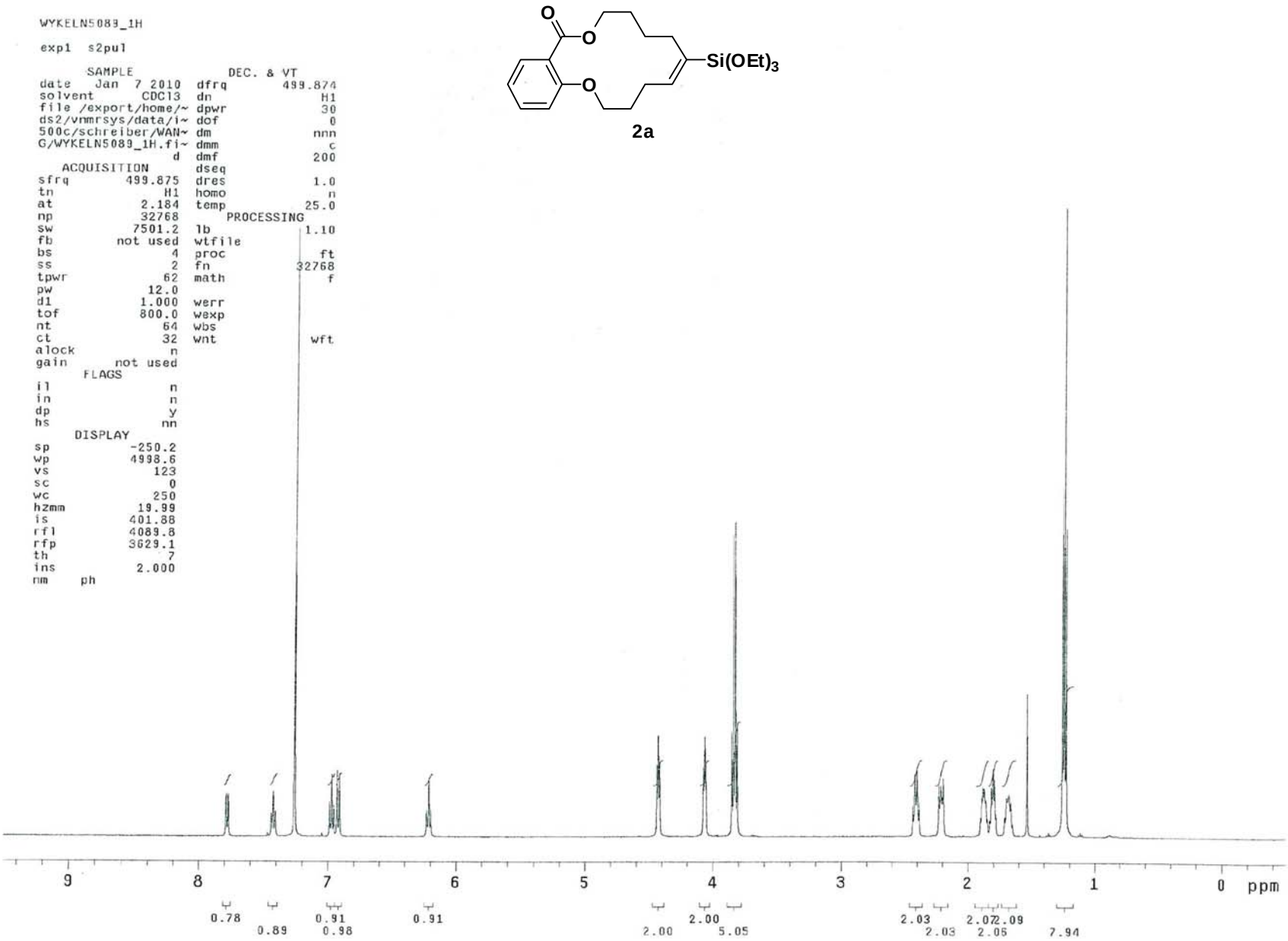
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dmf 200
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7
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5
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2.00
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2.03
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2.07
2.05
2.09
7.94

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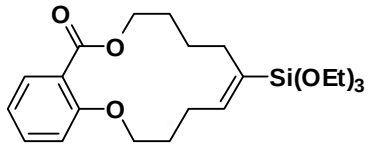

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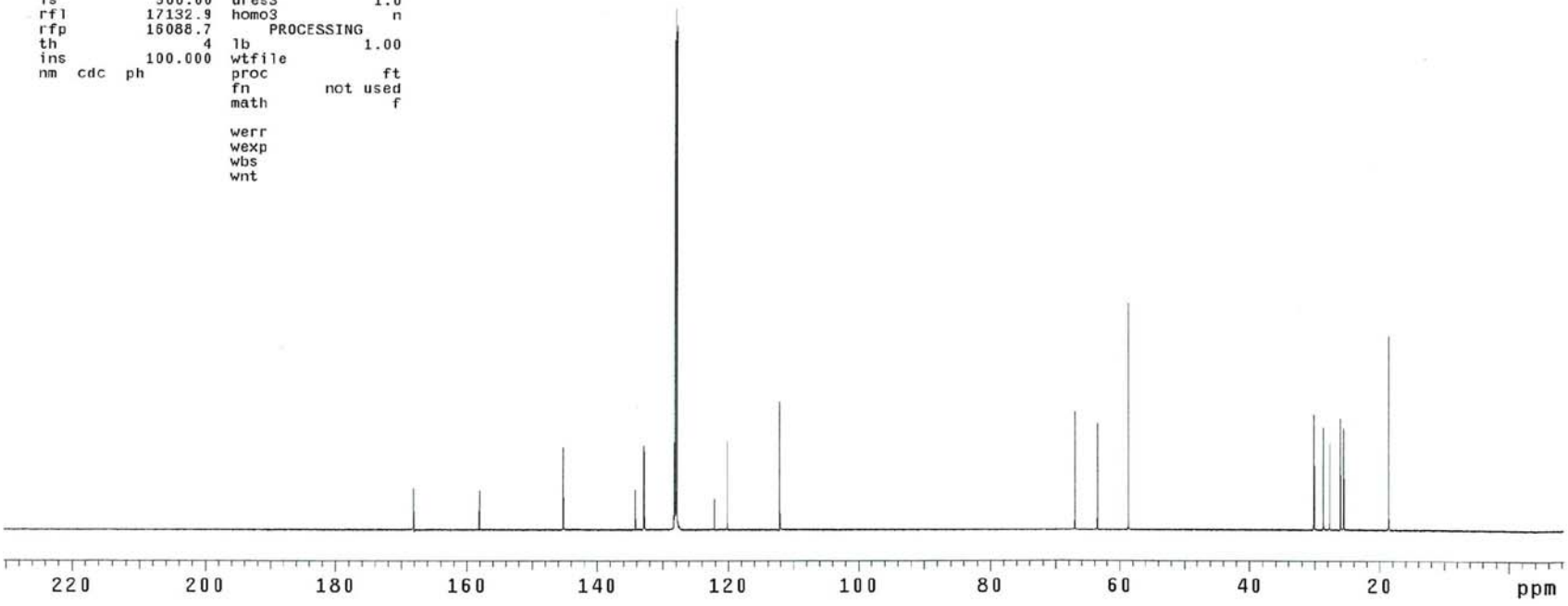

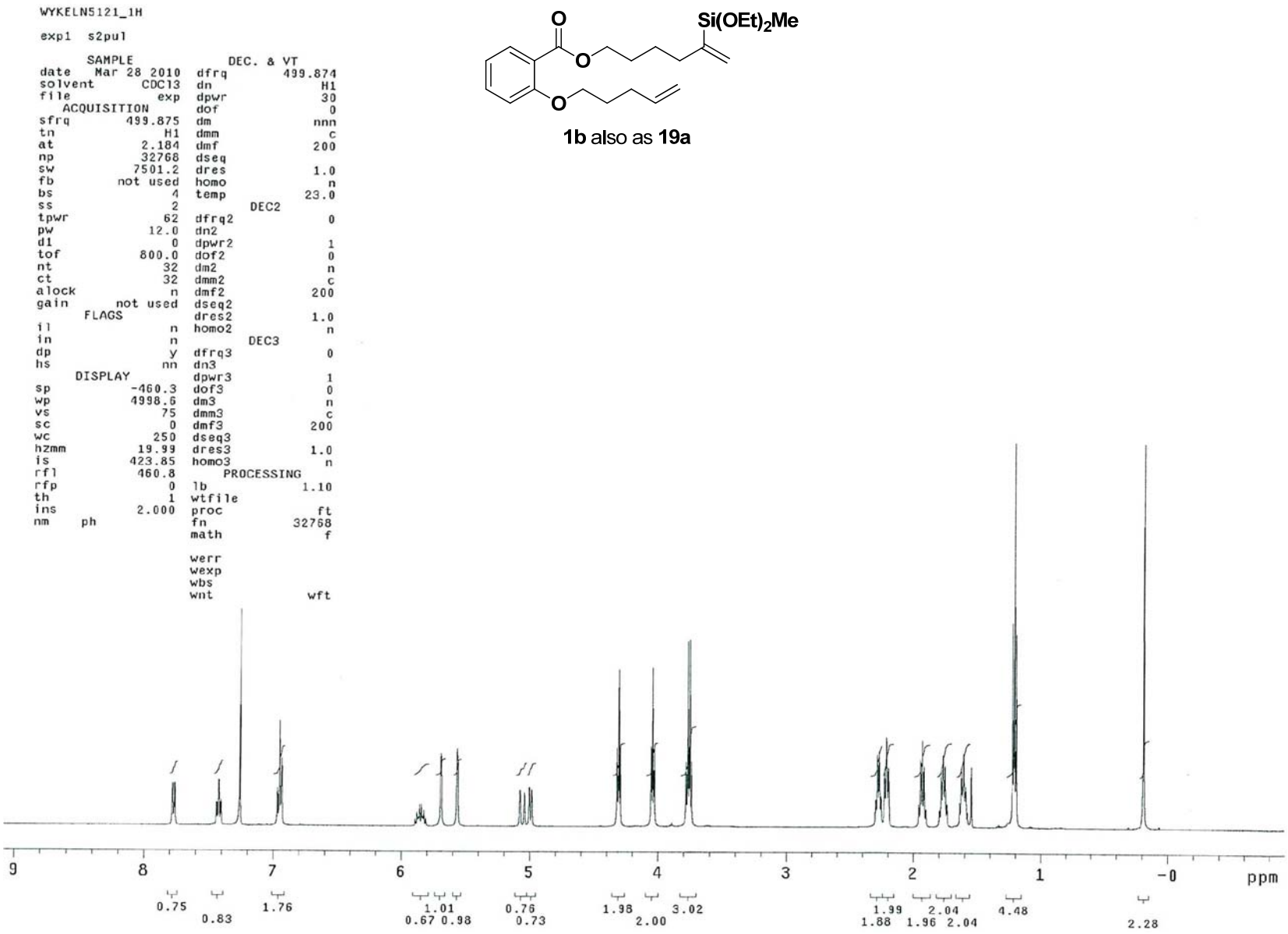

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WYKELN5121_13C

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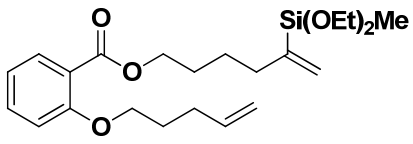

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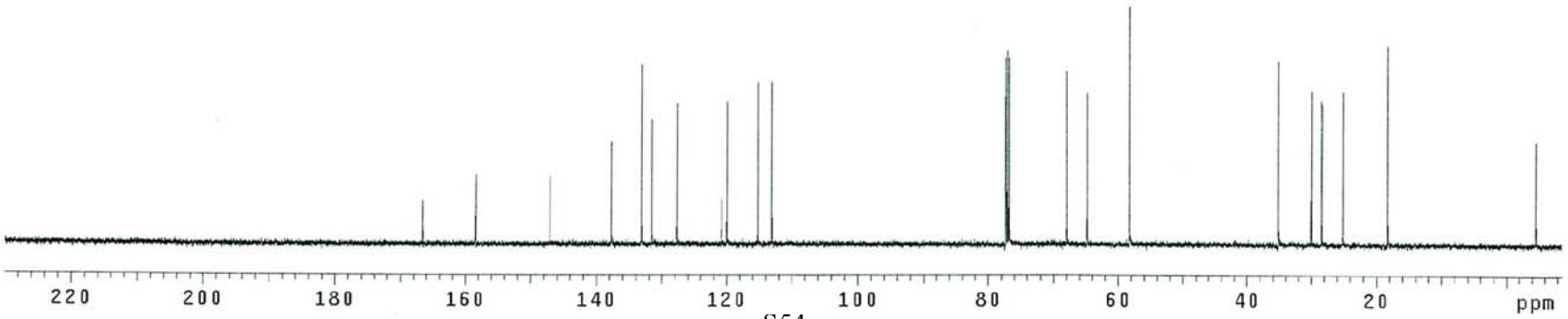

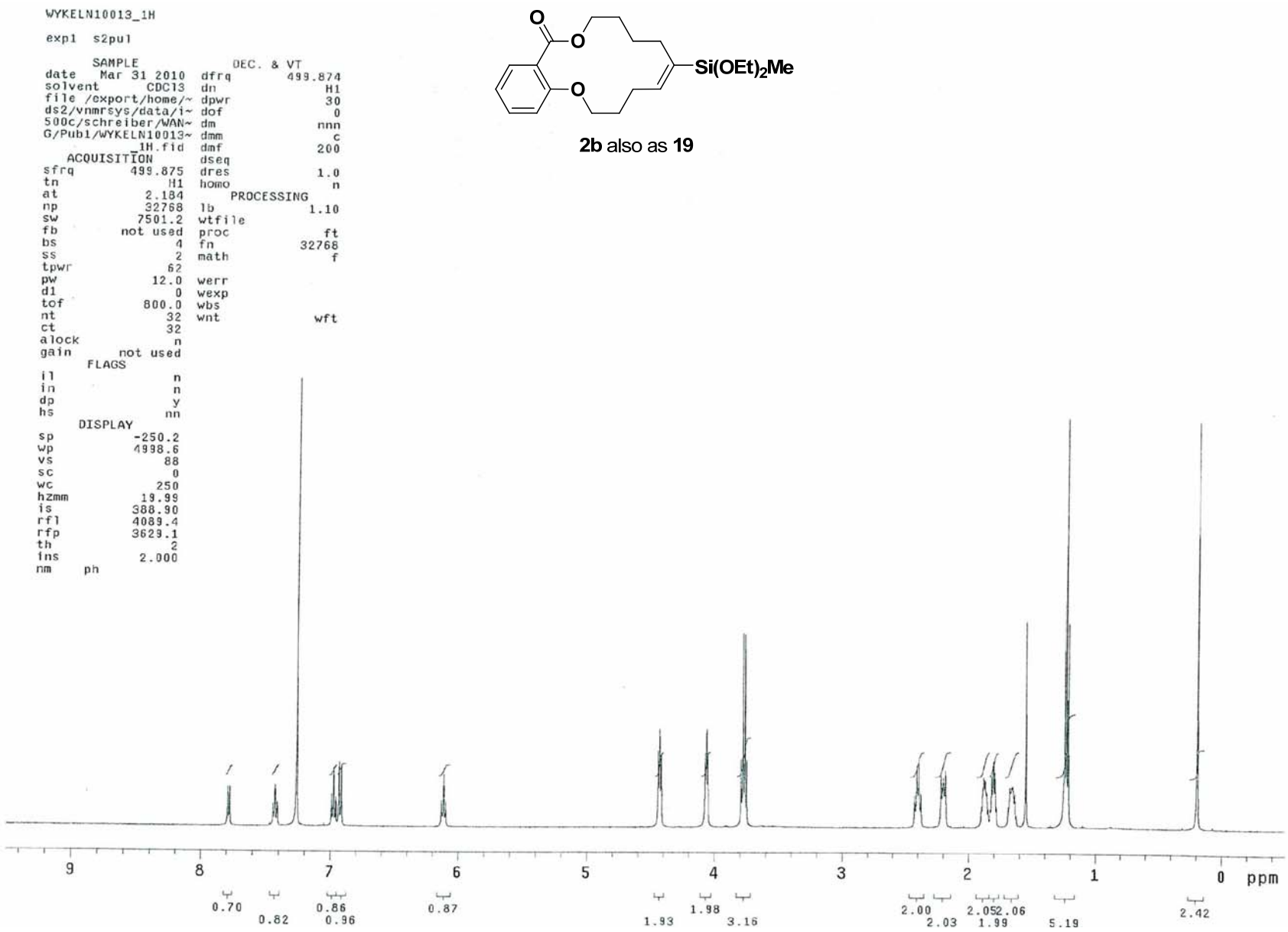
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8
7
6
5
4
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WYKELN10013_13C

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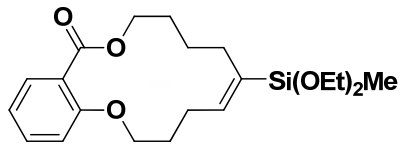

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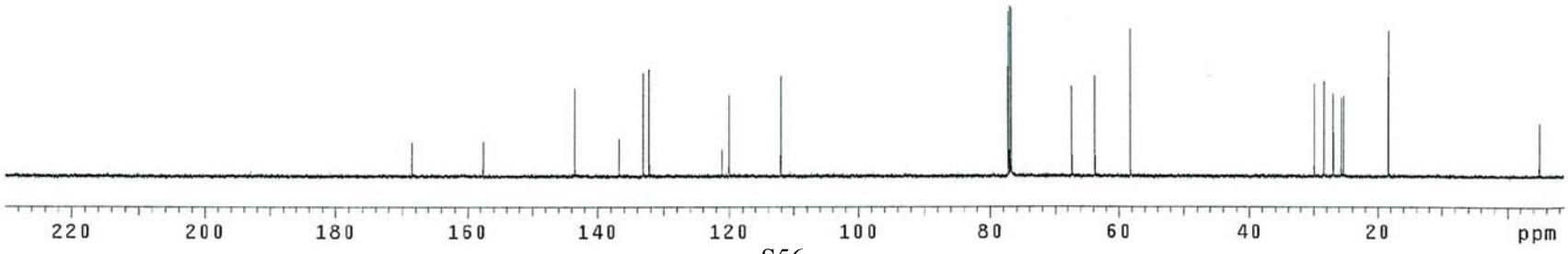

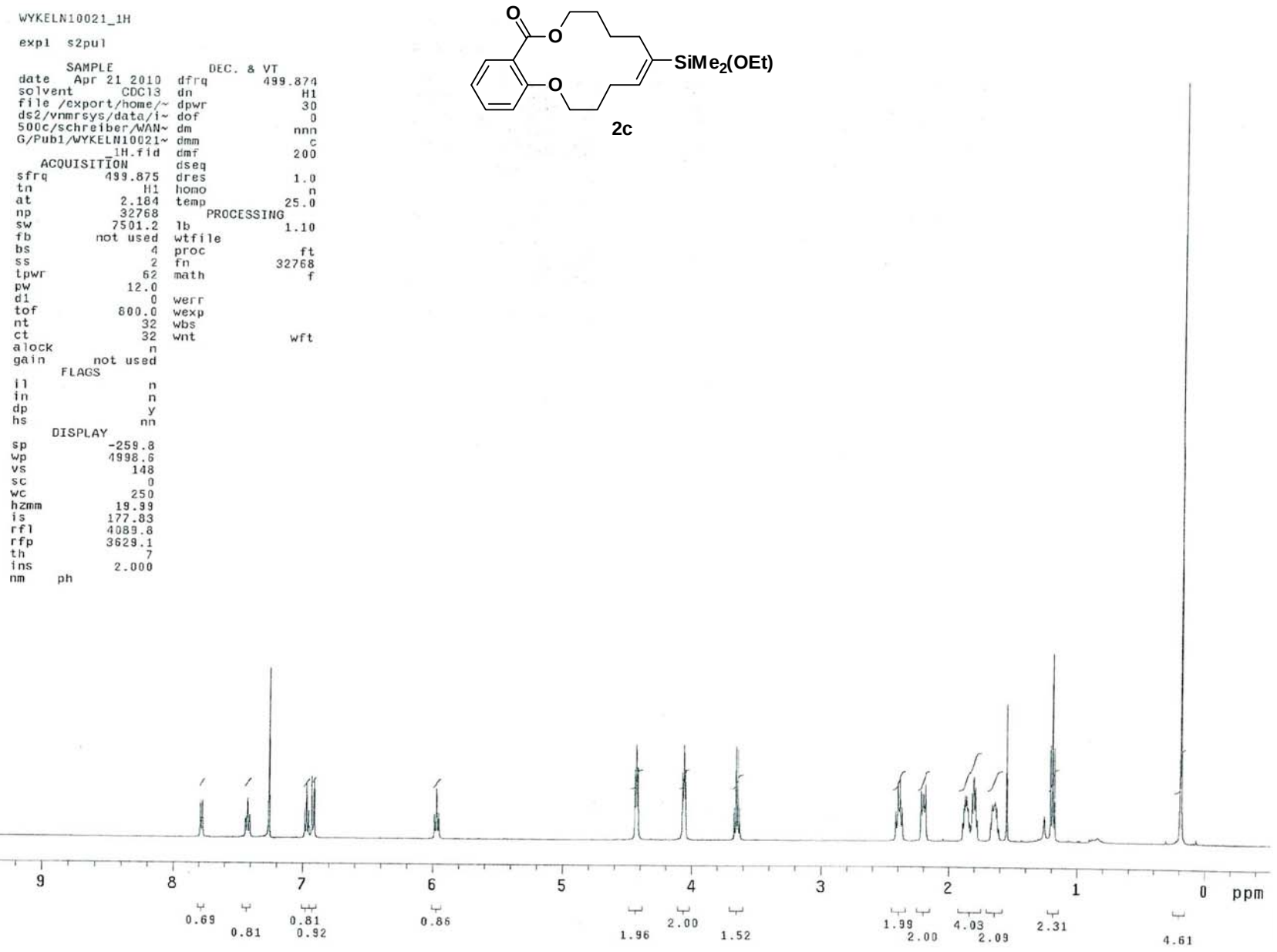
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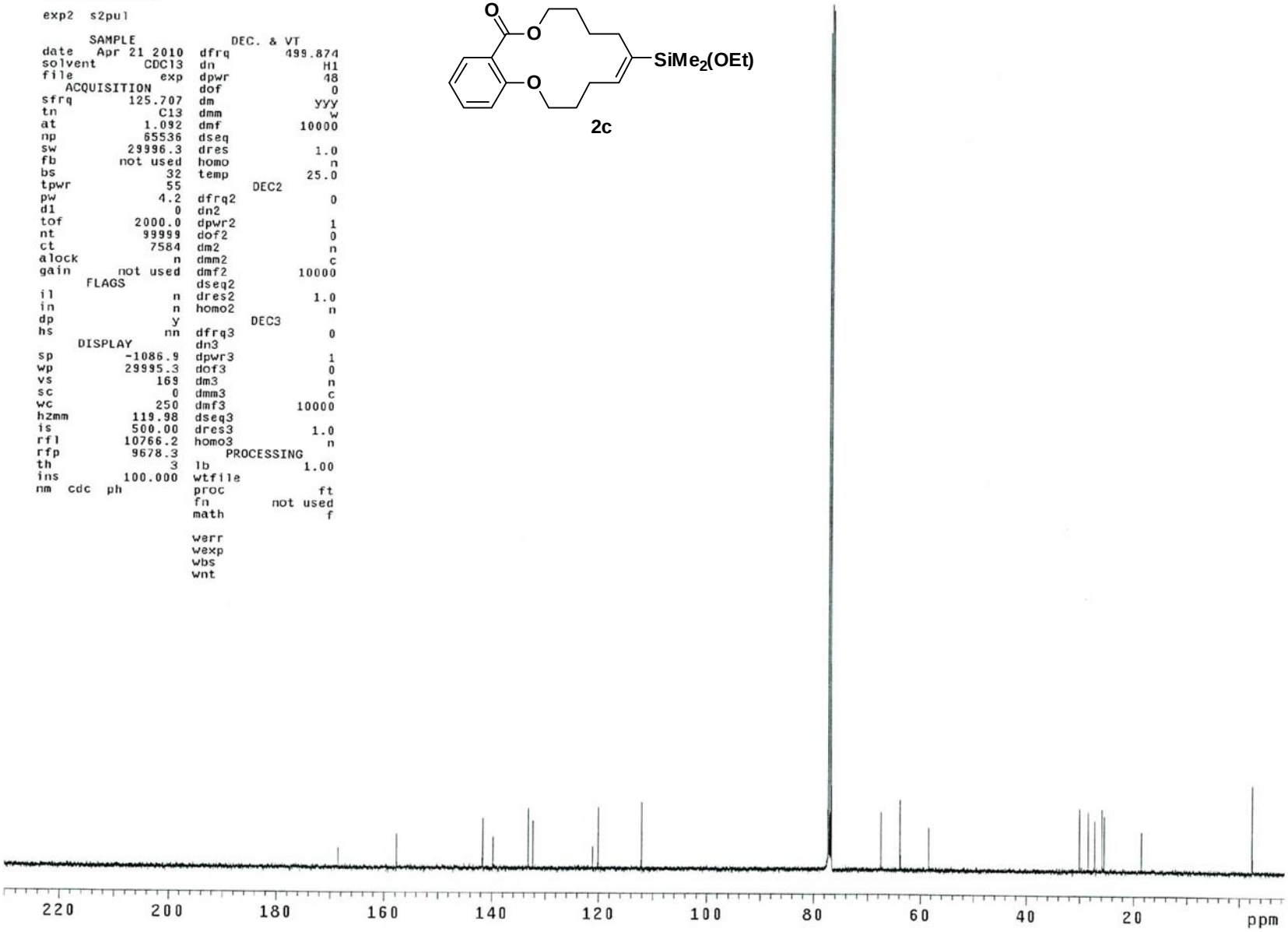
file /export/home/~
ds2/vnmrsys/data/i~
500c/schreiber/WAN~
G/Pub1/WYKELN10021~
_1H.fid
ACQUISITION
sfrq 499.875
tn H1
at 2.184
np 32768
sw 7501.2
fb not used
bs 4
ss 2
tpwr 62
pw 12.0
d1 0
tof 800.0
nt 32
ct 32
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -259.8
wp 4998.6
vs 148
sc 0
wc 250
hzmm 19.99
is 177.83
rfl 4089.8
rfp 3629.1
th 7
ins 2.000
nm ph
DEC. & VT
dfrq 499.874
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
temp 25.0
PROCESSING
lb 1.10
wtfile
proc ft
fn 32768
math f
werr
wexp
wbs
wnt wft
O
O
O
SiMe2(OEt)
2c
9
8
7
6
5
4
3
2
1
0
ppm
0.69
0.81
0.81
0.92
0.86
1.96
2.00
1.52
1.99
2.00
4.03
2.09
2.31
4.61

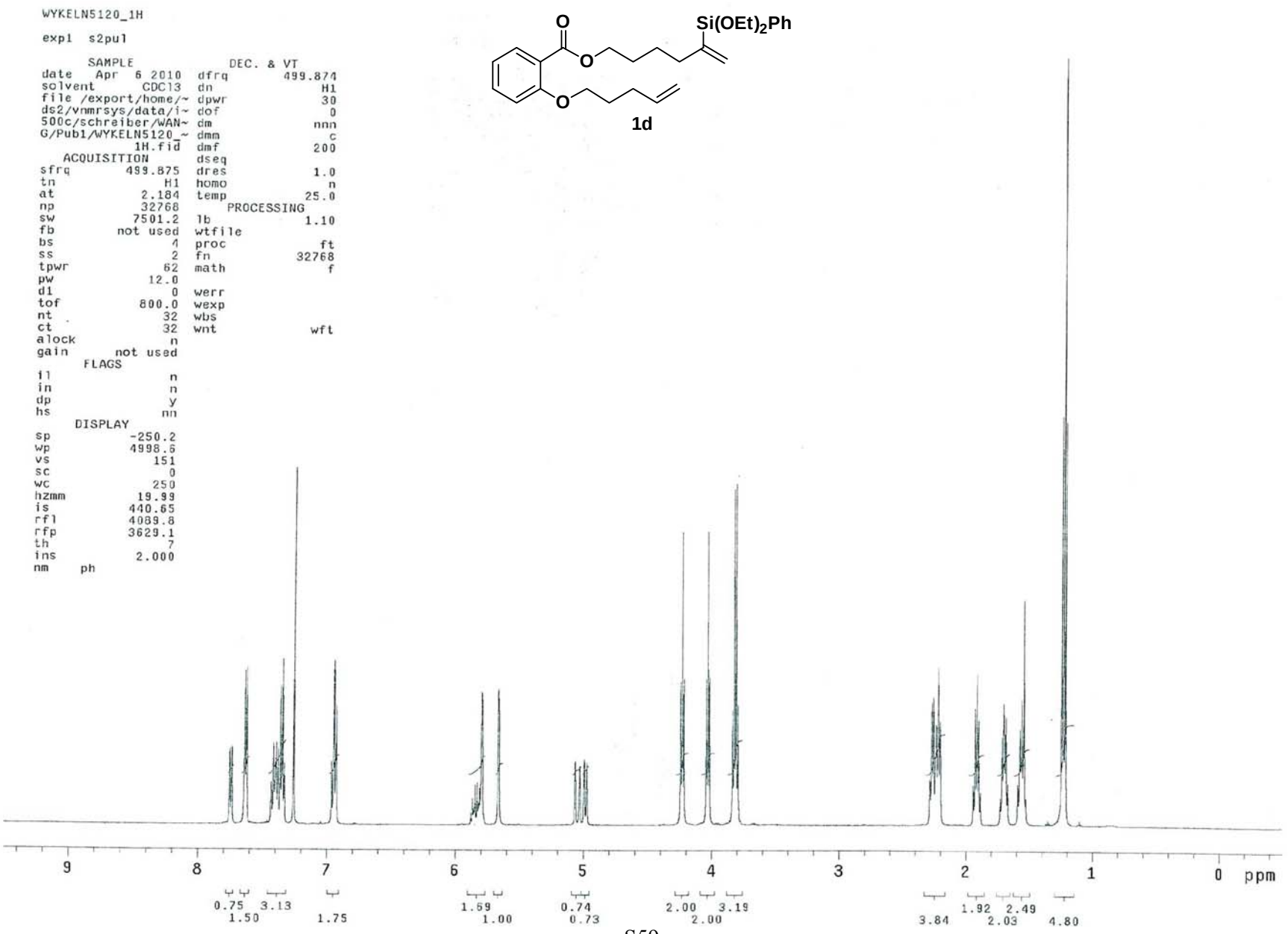
WYKELN10021_13C

exp2 s2pul

SAMPLE		DEC. & VT	
date	Apr 21 2010	dfrq	499.874
solvent	CDCl3	dn	H1
file	exp	dpwr	48
ACQUISITION		dof	0
sfrq	125.707	dm	yyy
tn	C13	dmm	w
at	1.092	dmf	10000
np	65536	dseq	
sw	29996.3	dres	1.0
fb	not used	homo	n
bs	32	temp	25.0
tpwr	55	DEC2	
pw	4.2	dfrq2	0
d1	0	dn2	
tof	2000.0	dpwr2	1
nt	99999	dof2	0
ct	7584	dm2	n
alock	n	dmm2	c
gain	not used	dmf2	10000
FLAGS		dseq2	
il	n	dres2	1.0
in	n	homo2	n
dp	y	DEC3	
hs	nn	dfrq3	0
DISPLAY		dn3	
sp	-1086.9	dpwr3	1
wp	29995.3	dof3	0
vs	169	dm3	n
sc	0	dmm3	c
wc	250	dmf3	10000
hzmm	119.98	dseq3	
is	500.00	dres3	1.0
rfl	10766.2	homo3	n
rfp	9678.3	PROCESSING	
th	3	lb	1.00
ins	100.000	wtfile	
nm cdc ph		proc	ft
		fn	not used
		math	f
		werr	
		wexp	
		wbs	
		wnt	

2c

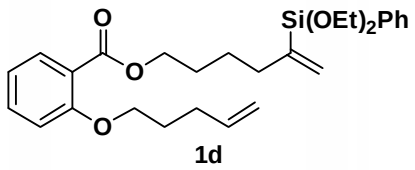



WYKELN5120_1H
expl s2pul
SAMPLE
date Apr 6 2010
solvent CDCl3
file /export/home/~
ds2/vnmrsys/data/i~
500c/schreiber/WAN~
G/Publ/WYKELN5120_~
1H.fid
ACQUISITION
sfrq 499.875
tn H1
at 2.184
np 32768
sw 7501.2
fb not used
bs 4
ss 2
tpwr 62
pw 12.0
d1 0
tof 800.0
nt 32
ct 32
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -250.2
wp 4998.6
vs 151
sc 0
wc 250
hzmm 19.99
is 440.65
rfl 4089.8
rfp 3629.1
th 7
ins 2.000
nm ph
DEC. & VT
dfrq 499.874
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
temp 25.0
PROCESSING
lb 1.10
wtfile
proc ft
fn 32768
math f
werr
wexp
wbs
wnt wft
O
O
O
Si(OEt)2Ph
1d
9
8
7
6
5
4
3
2
1
0 ppm
0.75 3.13
1.50
1.75
1.59
1.00
0.74
0.73
2.00 3.19
2.00
3.84
1.92 2.49
2.03
4.80

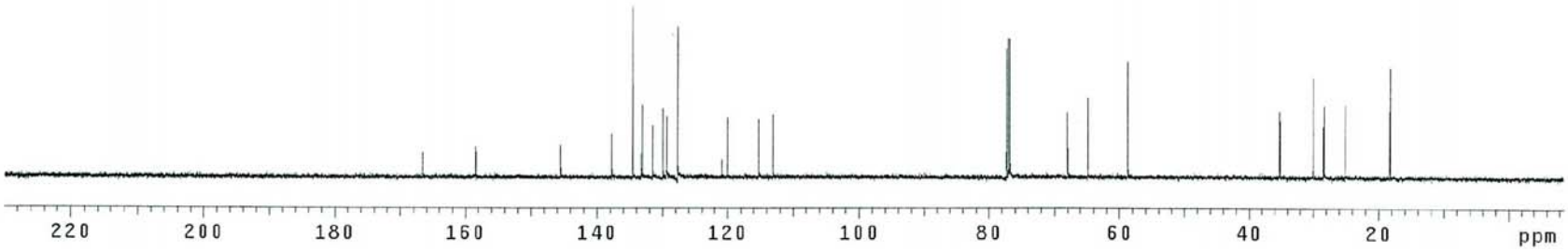
WYKELN5120_13C

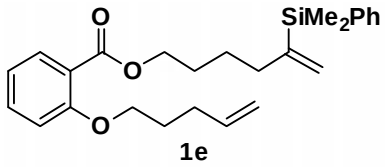
exp2 s2pul

SAMPLE		DEC. & VT	
date	Apr 6 2010	dfrq	499.874
solvent	CDCl3	dn	H1
file	/export/home/~	dpwr	48
	i500c/vnmrsys/data~	dof	0
	/schreiber/WANG/Pu~	dm	yyy
	b1/WYKELN5120_13C.~	dmm	w
	fid	dmf	10000
ACQUISITION		dseq	
sfrq	125.707	dres	1.0
tn	C13	homo	n
at	1.092	temp	25.0
np	65536	DEC2	
sw	29996.3	dfrq2	0
fb	not used	dn2	
bs	16	dpwr2	1
tpwr	55	dof2	0
pw	4.2	dm2	n
d1	0	dmm2	c
tof	2000.0	dmf2	10000
nt	99999	dseq2	
ct	192	dres2	1.0
alock	n	homo2	n
gain	not used	DEC3	
FLAGS		dfrq3	0
il	n	dn3	
in	n	dpwr3	1
dp	y	dof3	0
hs	nn	dm3	n
DISPLAY		dmm3	c
sp	-1089.7	dmf3	10000
wp	29995.3	dseq3	
vs	27	dres3	1.0
sc	0	homo3	n
wc	250	PROCESSING	
hzmm	119.98	lb	1.00
is	500.00	wtfile	
rfl	10768.9	proc	ft
rfp	9678.3	fn	not used
th	2	math	f
ins	100.000		
nm cdc ph		werr	
		wexp	
		wbs	
		wnt	



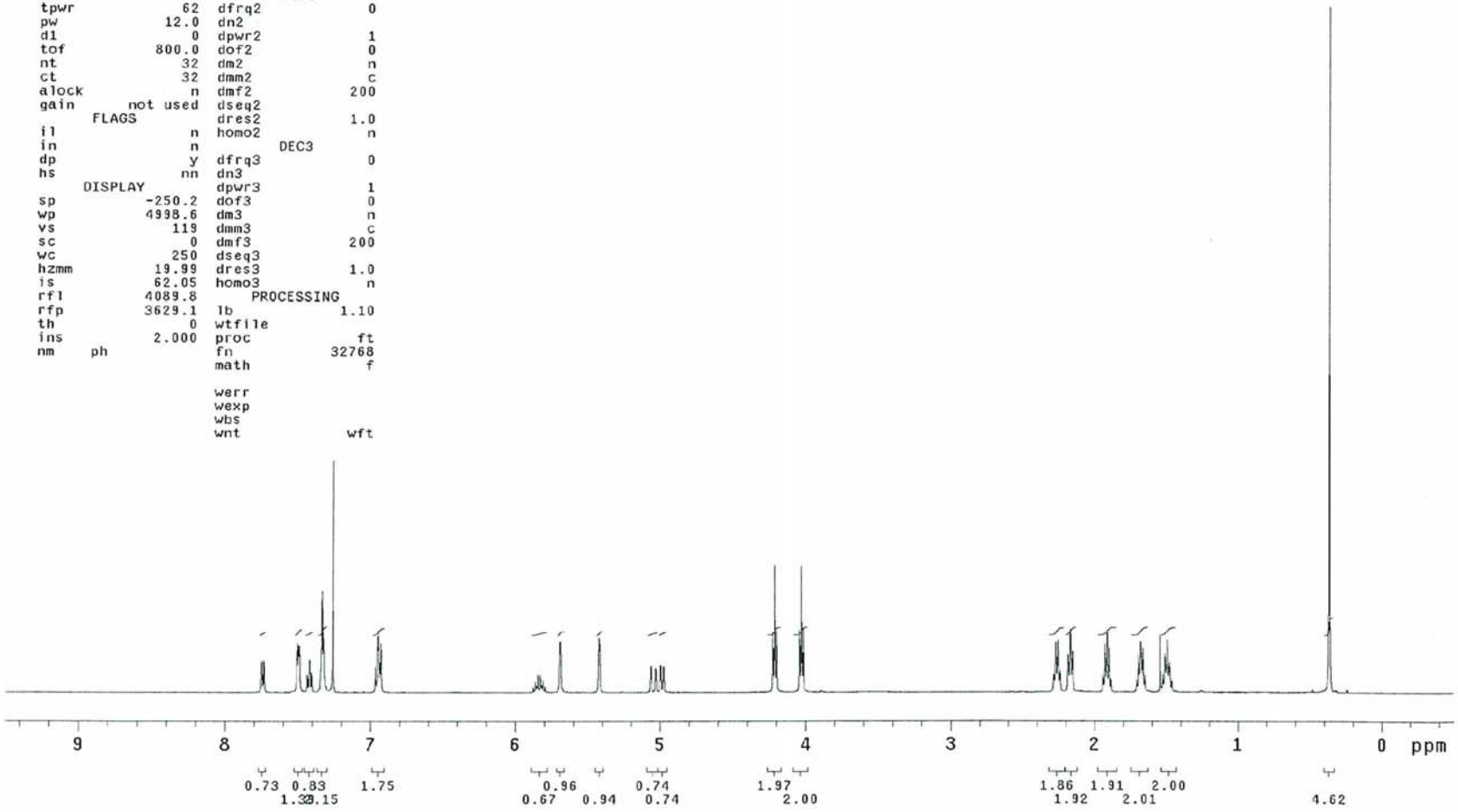
1d





1e

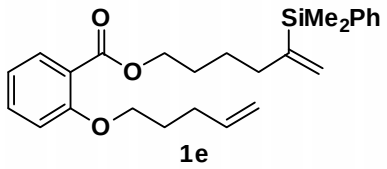
```
WYKELN5119_1H

exp1  s2pul

      SAMPLE
date    Mar 28 2010
solvent       CDCl3
file             exp
    ACQUISITION
sfrq         499.875
tn                H1
at             2.184
np             32768
sw            7501.2
fb         not used
bs                 4
ss                 2
tpwr              62
pw              12.0
d1                 0
tof            800.0
nt                32
ct                32
alock              n
gain       not used
      FLAGS
il                 n
in                 n
dp                 y
hs                nn
     DISPLAY
sp            -250.2
wp            4998.6
vs               119
sc                 0
wc               250
hzmm           19.99
is             62.05
rfl            4089.8
rfp            3629.1
th                 0
ins            2.000
nm     ph

      DEC. & VT
dfrq         499.874
dn                H1
dpwr              30
dof                0
dm               nnn
dmm                c
dmf              200
dseq
dres             1.0
homo               n
temp            23.0
      DEC2
dfrq2              0
dn2
dpwr2              1
dof2               0
dm2                n
dmm2               c
dmf2             200
dseq2
dres2            1.0
homo2              n
      DEC3
dfrq3              0
dn3
dpwr3              1
dof3               0
dm3                n
dmm3               c
dmf3             200
dseq3
dres3            1.0
homo3              n
    PROCESSING
lb              1.10
wtfile
proc              ft
fn             32768
math               f

werr
wexp
wbs
wnt             wft
```



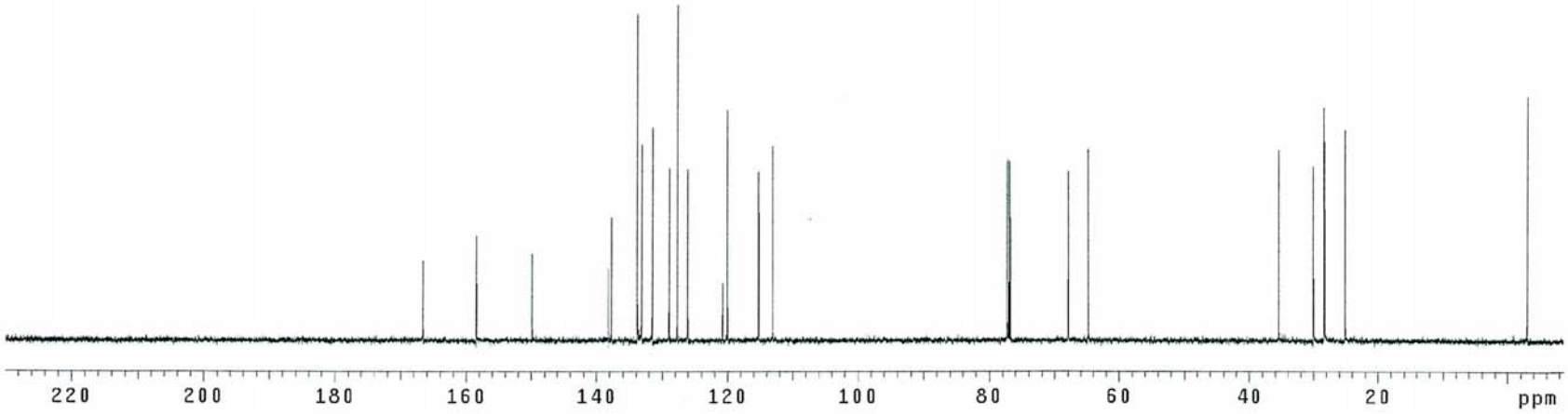
WYKELN5119_13C

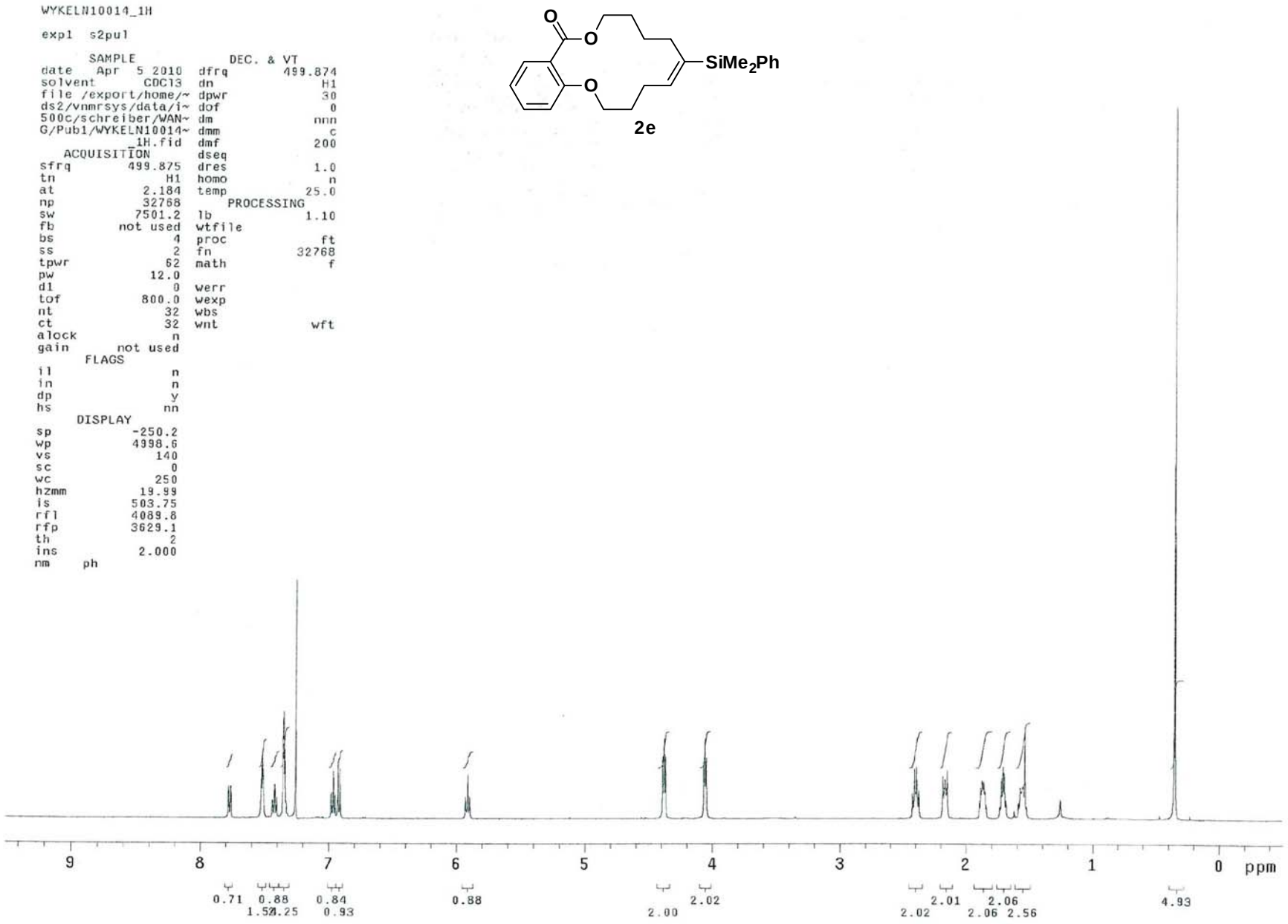
exp1 s2pul

SAMPLE		DEC. & VT	
date	Mar 28 2010	dfrq	499.874
solvent	CDCl3	dn	H1
file	exp	dpwr	48
ACQUISITION		dof	0
sfrq	125.707	dm	yyy
tn	C13	dmm	w
at	1.092	dmf	10000
np	65536	dseq	
sw	29996.3	dres	1.0
fb	not used	homo	n
bs	32	temp	23.0
tpwr	55	DEC2	
pw	4.2	dfrq2	0
d1	0	dn2	
tof	2000.0	dpwr2	1
nt	9999	dof2	0
ct	288	dm2	n
alock	n	dmm2	c
gain	not used	dmf2	10000
FLAGS		dseq2	
il	n	dres2	1.0
in	n	homo2	n
dp	y	DEC3	
hs	nn	dfrq3	0
DISPLAY		dn3	
sp	-1094.2	dpwr3	1
wp	29995.3	dof3	0
vs	54	dm3	n
sc	0	dmm3	c
wc	250	dmf3	10000
hzmm	119.98	dseq3	
is	500.00	dres3	1.0
rfl	10773.5	homo3	n
rfp	9678.3	PROCESSING	
th	7	lb	1.00
ins	100.000	wtfile	
nm cdc ph		proc	ft
		fn	not used
		math	f
		werr	
		wexp	
		wbs	
		wnt	



1e

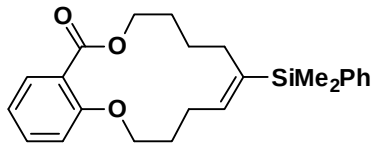



WYKELN10014_1H
expl s2pul
SAMPLE
date Apr 5 2010
solvent CDCl3
file /export/home/~
ds2/vnmrsys/data/i~
500c/schreiber/WAN~
G/Pub1/WYKELN10014~
_1H.fid
ACQUISITION
sfrq 499.875
tn H1
at 2.184
np 32768
sw 7501.2
fb not used
bs 4
ss 2
tpwr 62
pw 12.0
d1 0
tof 800.0
nt 32
ct 32
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -250.2
wp 4998.6
vs 140
sc 0
wc 250
hzmm 19.99
is 503.75
rfl 4089.8
rfp 3629.1
th 2
ins 2.000
nm ph
DEC. & VT
dfrq 499.874
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
temp 25.0
PROCESSING
lb 1.10
wtfile
proc ft
fn 32768
math f
werr
wexp
wbs
wnt wft
O
O
O
SiMe2Ph
2e
9
8
7
6
5
4
3
2
1
0
ppm
0.71
0.88
1.52
4.25
0.84
0.93
0.88
2.00
2.02
2.02
2.01
2.06
2.06
2.56
4.93

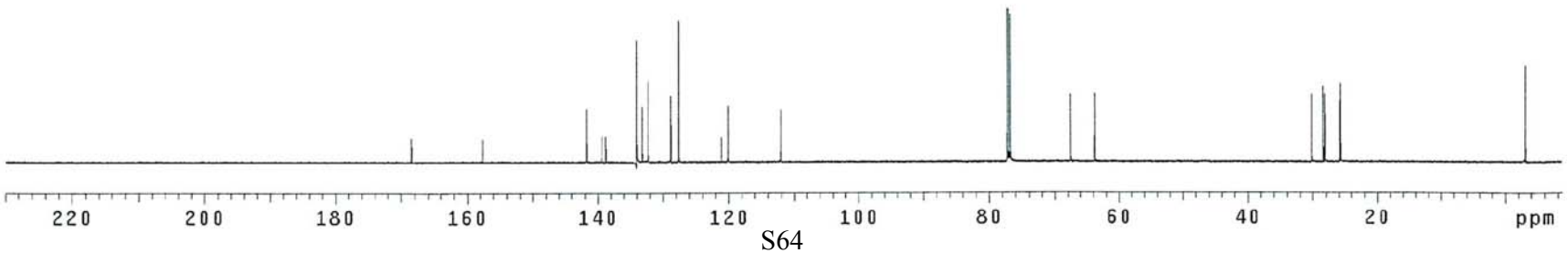
WYKELN10014_13C

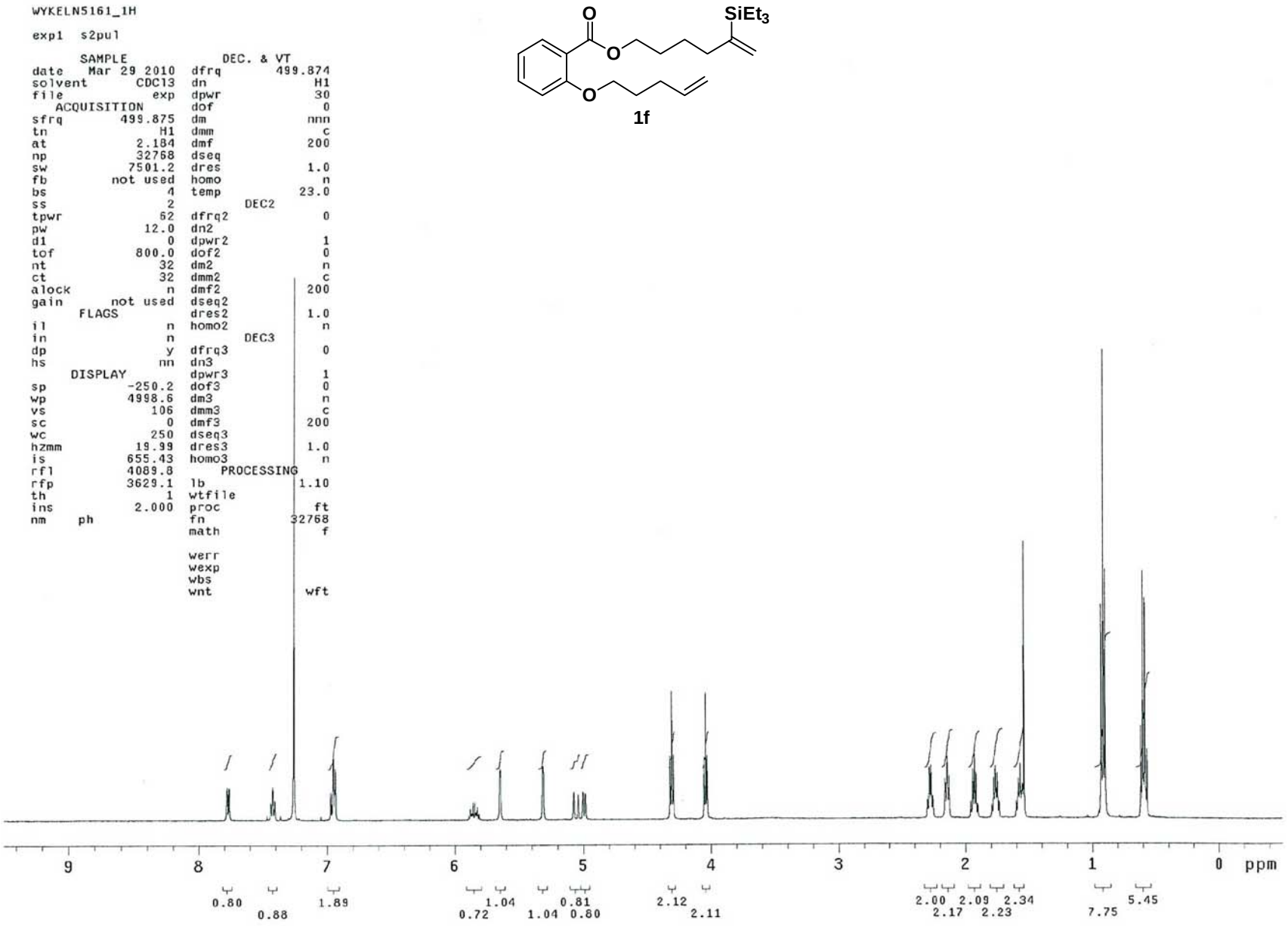
exp2 s2pul

SAMPLE		DEC. & VT	
date	Apr 2 2010	dfrq	499.874
solvent	CDCl3	dn	H1
file	exp	dpwr	48
ACQUISITION		dof	0
sfrq	125.707	dm	yyy
tn	C13	dmm	w
at	1.092	dmf	10000
np	65536	dseq	
sw	29996.3	dres	1.0
fb	not used	homo	n
bs	16	temp	25.0
tpwr	55	DEC2	
pw	4.2	dfrq2	0
d1	0	dn2	
tof	2000.0	dpwr2	1
nt	99999	dof2	0
ct	2128	dm2	n
alock	n	dmm2	c
gain	not used	dmf2	10000
FLAGS		dseq2	
il	n	dres2	1.0
in	n	homo2	n
dp	y	DEC3	
hs	nn	dfrq3	0
DISPLAY		dn3	
sp	-1088.7	dpwr3	1
wp	29995.3	dof3	0
vs	25	dm3	n
sc	0	dmm3	c
wc	250	dmf3	10000
hzmm	119.98	dseq3	
is	500.00	dres3	1.0
rfl	10768.0	homo3	n
rfp	9678.3	PROCESSING	
th	2	lb	1.00
ins	100.000	wtfile	
nm cdc ph		proc	ft
		fn	not used
		math	f
		werr	
		wexp	
		wbs	
		wnt	



2e

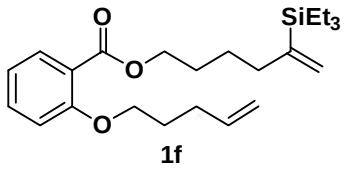



WYKELN5161_1H
exp1 s2pul
SAMPLE
date Mar 29 2010
solvent CDCl3
file exp
ACQUISITION
sfrq 499.875
tn H1
at 2.184
np 32768
sw 7501.2
fb not used
bs 4
ss 2
tpwr 62
pw 12.0
d1 0
tof 800.0
nt 32
ct 32
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -250.2
wp 4998.6
vs 106
sc 0
wc 250
hzmm 19.99
is 655.43
rfl 4089.8
rfp 3629.1
th 1
ins 2.000
nm ph
DEC. & VT
dfrq 499.874
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
temp 23.0
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 200
dseq2
dres2 1.0
homo2 n
DEC3
dfrq3 0
dn3
dpwr3 1
dof3 0
dm3 n
dmm3 c
dmf3 200
dseq3
dres3 1.0
homo3 n
PROCESSING
lb 1.10
wtfile
proc ft
fn 32768
math f
werr
wexp
wbs
wnt wft
O
O
O
SiEt3
1f
9
8
7
6
5
4
3
2
1
0 ppm
0.80
0.88
1.89
0.72
1.04
1.04
0.80
0.81
2.12
2.11
2.00
2.17
2.09
2.23
2.34
7.75
5.45

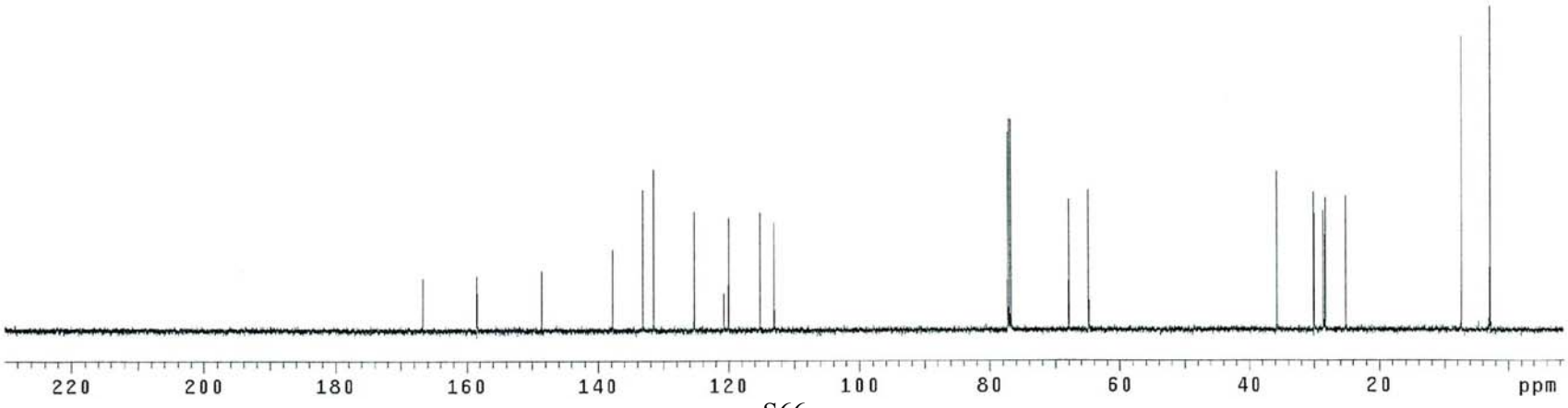
WYKELN5161_13C

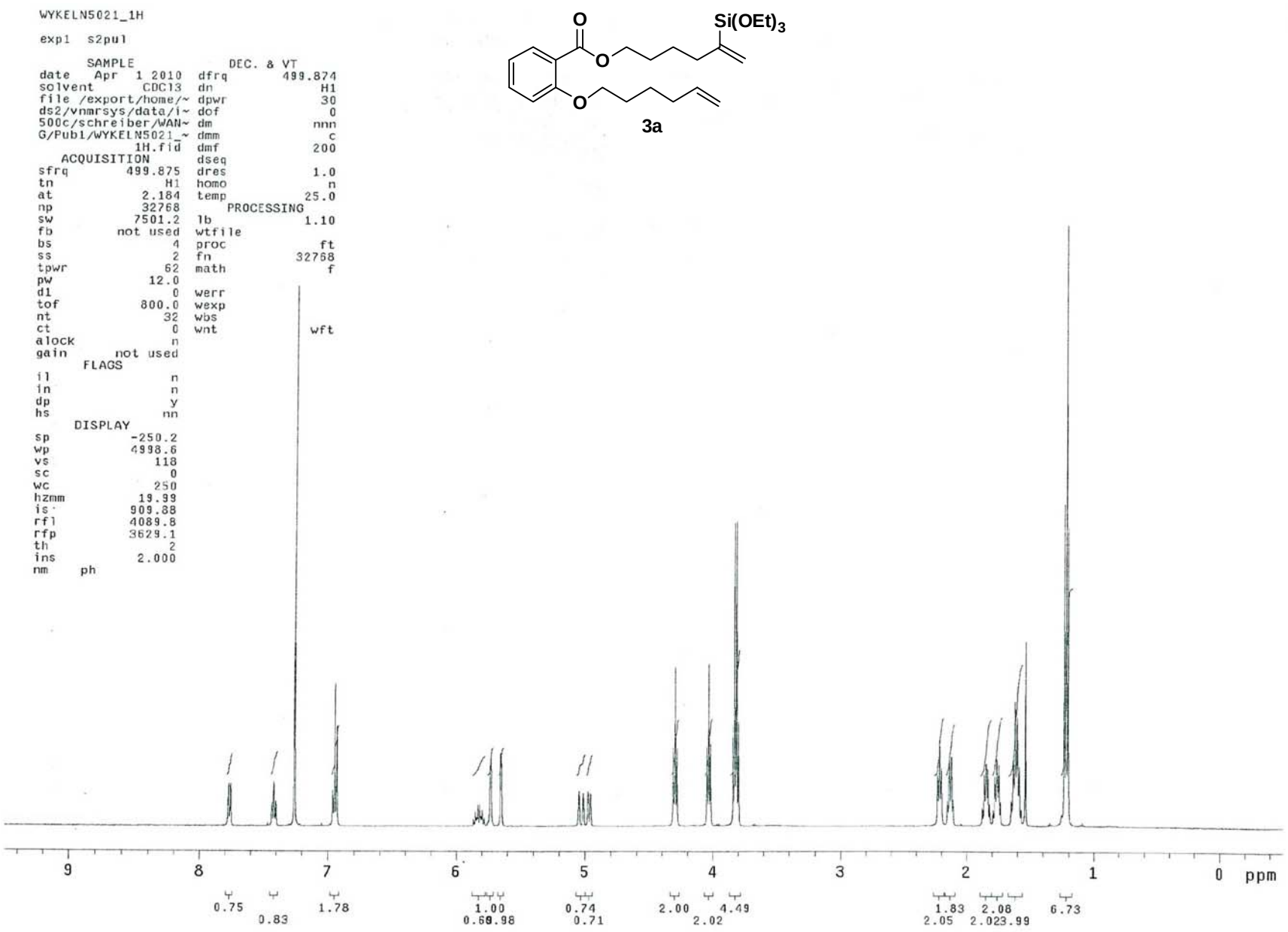
exp1 s2pul

SAMPLE		DEC. & VT	
date	Mar 29 2010	dfrq	499.874
solvent	CDCl3	dn	H1
file	exp	dpwr	48
ACQUISITION		dof	0
sfrq	125.707	dm	yyy
tn	C13	dmm	w
at	1.092	dmf	10000
np	65536	dseq	
sw	29996.3	dres	1.0
fb	not used	homo	n
bs	16	temp	23.0
tpwr	55	DEC2	
pw	4.2	dfrq2	0
d1	0	dn2	
tof	2000.0	dpwr2	1
nt	9999	dof2	0
ct	336	dm2	n
alock	n	dmm2	c
gain	not used	dmf2	10000
FLAGS		dseq2	
il	n	dres2	1.0
in	n	homo2	n
dp	y	DEC3	
hs	nn	dfrq3	0
DISPLAY		dn3	
sp	-1089.7	dpwr3	1
wp	29995.3	dof3	0
vs	52	dm3	n
sc	0	dmm3	c
wc	250	dmf3	10000
hzmm	119.98	dseq3	
is	500.00	dres3	1.0
rfl	10768.9	homo3	n
rfp	9678.3	PROCESSING	
th	3	lb	1.00
ins	100.000	wtfile	
nm cdc ph		proc	ft
		fn	not used
		math	f
		werr	
		wexp	
		wbs	
		wnt	



1f

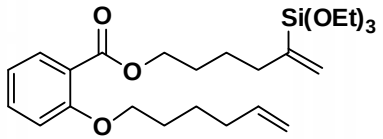



WYKELN5021_1H
exp1 s2pul
SAMPLE
date Apr 1 2010
solvent CDCl3
file /export/home/~
ds2/vnmrsys/data/i~
500c/schreiber/WAN~
G/Publ/WYKELN5021_~
1H.fid
ACQUISITION
sfrq 499.875
tn H1
at 2.184
np 32768
sw 7501.2
fb not used
bs 4
ss 2
tpwr 62
pw 12.0
d1 0
tof 800.0
nt 32
ct 0
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -250.2
wp 4998.6
vs 118
sc 0
wc 250
hzmm 19.99
is 909.88
rfl 4089.8
rfp 3629.1
th 2
ins 2.000
nm ph
DEC. & VT
dfrq 499.874
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
temp 25.0
PROCESSING
lb 1.10
wtfile
proc ft
fn 32768
math f
werr
wexp
wbs
wnt wft
O
Si(OEt)3
O
O
3a
9
8
7
6
5
4
3
2
1
0 ppm
0.75
0.83
1.78
1.00
0.69
0.98
0.74
0.71
2.00
4.49
2.02
1.83
2.05
2.08
2.02
3.99
6.73

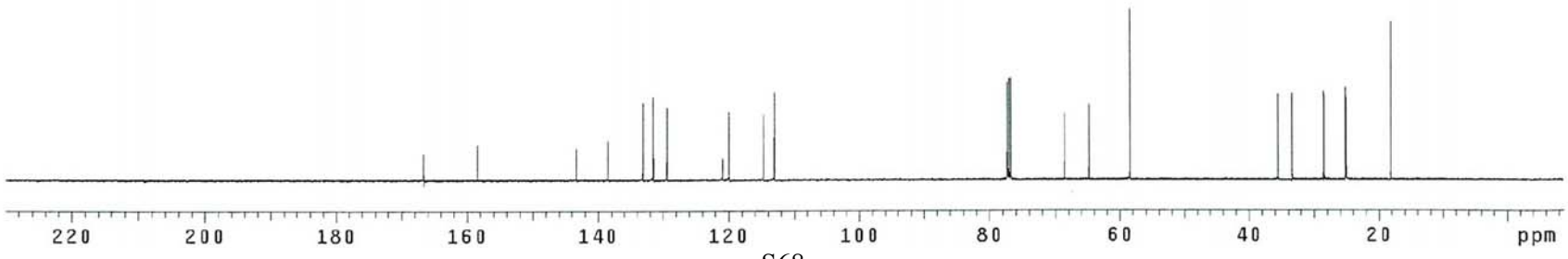
WYKELN5021_13C

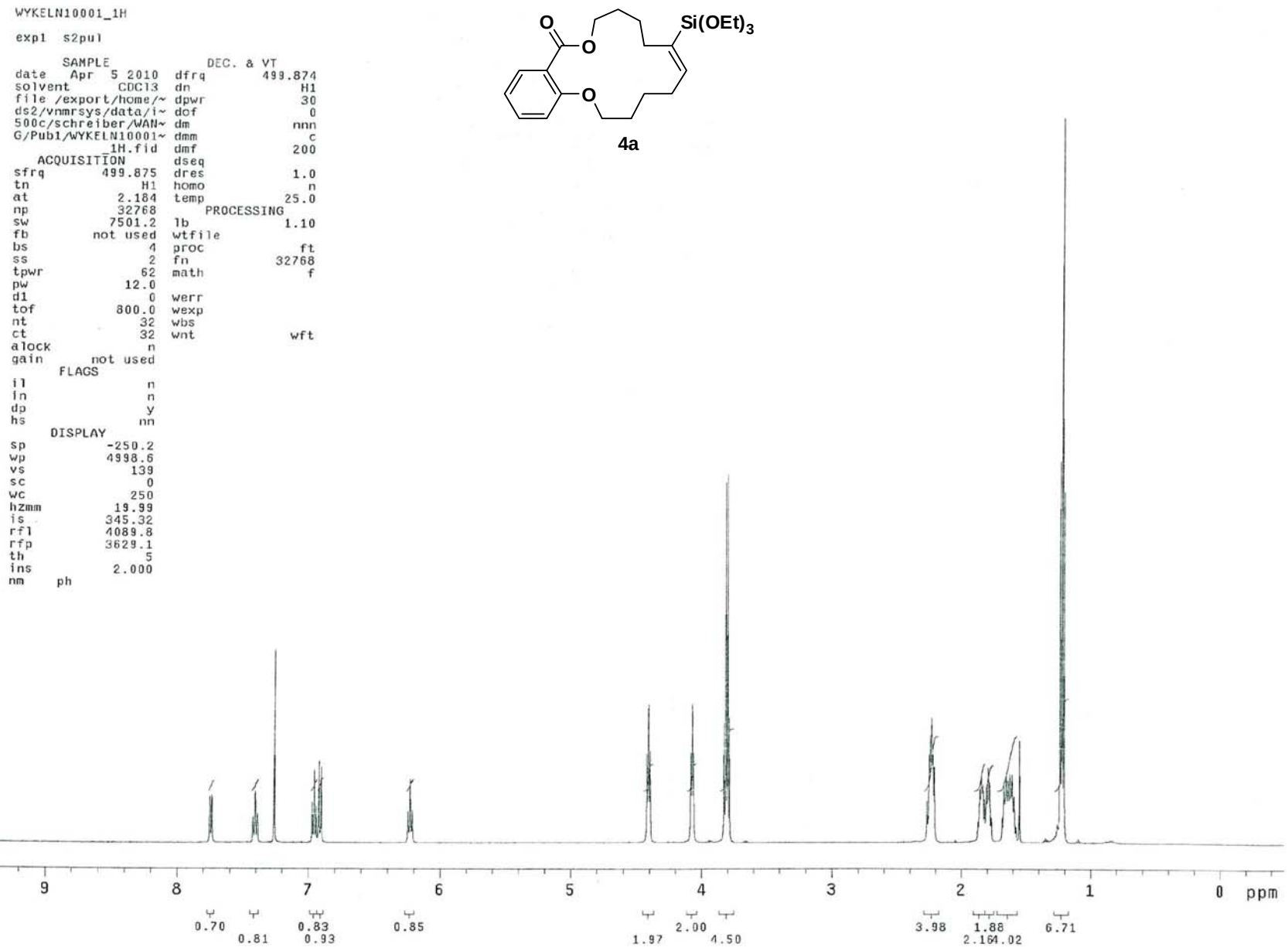
exp2 s2pul

SAMPLE		DEC. & VT	
date	Apr 1 2010	dfrq	499.874
solvent	CDCl3	dn	H1
file	exp	dpwr	48
ACQUISITION		dof	0
sfrq	125.707	dm	yyy
tn	C13	dmm	w
at	1.092	dmf	10000
np	65536	dseq	
sw	29996.3	dres	1.0
fb	not used	homo	n
bs	16	temp	25.0
tpwr	55	DEC2	
pw	4.2	dfrq2	0
d1	0	dn2	
tof	2000.0	dpwr2	1
nt	9999	dof2	0
ct	0	dm2	n
alock	n	dmm2	c
gain	not used	dmf2	10000
FLAGS		dseq2	
il	n	dres2	1.0
in	n	homo2	n
dp	y	DEC3	
hs	nn	dfrq3	0
DISPLAY		dn3	
sp	-1089.7	dpwr3	1
wp	29995.3	dof3	0
vs	27	dm3	n
sc	0	dmm3	c
wc	250	dmf3	10000
hzmm	119.98	dseq3	
is	500.00	dres3	1.0
rfl	10768.9	homo3	n
rfp	9678.3	PROCESSING	
th	2	lb	1.00
ins	100.000	wtfile	
nm cdc ph		proc	ft
		fn	not used
		math	f
		werr	
		wexp	
		wbs	
		wnt	



3a

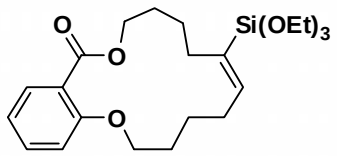



WYKELN10001_1H
expl s2pul
SAMPLE
date Apr 5 2010
solvent CDCl3
file /export/home/~
ds2/vnmrsys/data/i~
500c/schreiber/WAN~
G/Pub1/WYKELN10001~
_1H.fid
ACQUISITION
sfrq 499.875
tn H1
at 2.184
np 32768
sw 7501.2
fb not used
bs 4
ss 2
tpwr 62
pw 12.0
d1 0
tof 800.0
nt 32
ct 32
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -250.2
wp 4998.6
vs 139
sc 0
wc 250
hzmm 19.99
is 345.32
rfl 4089.8
rfp 3629.1
th 5
ins 2.000
nm ph
DEC. & VT
dfrq 499.874
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
temp 25.0
PROCESSING
lb 1.10
wtfile
proc ft
fn 32768
math f
werr
wexp
wbs
wnt wft
O
O
O
Si(OEt)3
4a
9
8
7
6
5
4
3
2
1
0 ppm
0.70
0.81
0.83
0.93
0.85
1.97
2.00
4.50
3.98
1.88
2.16
4.02
6.71

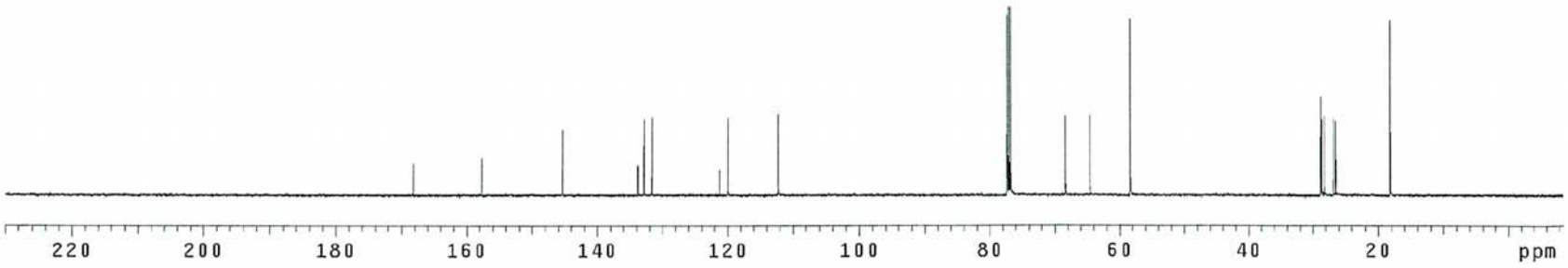
WYKELN10001_13C

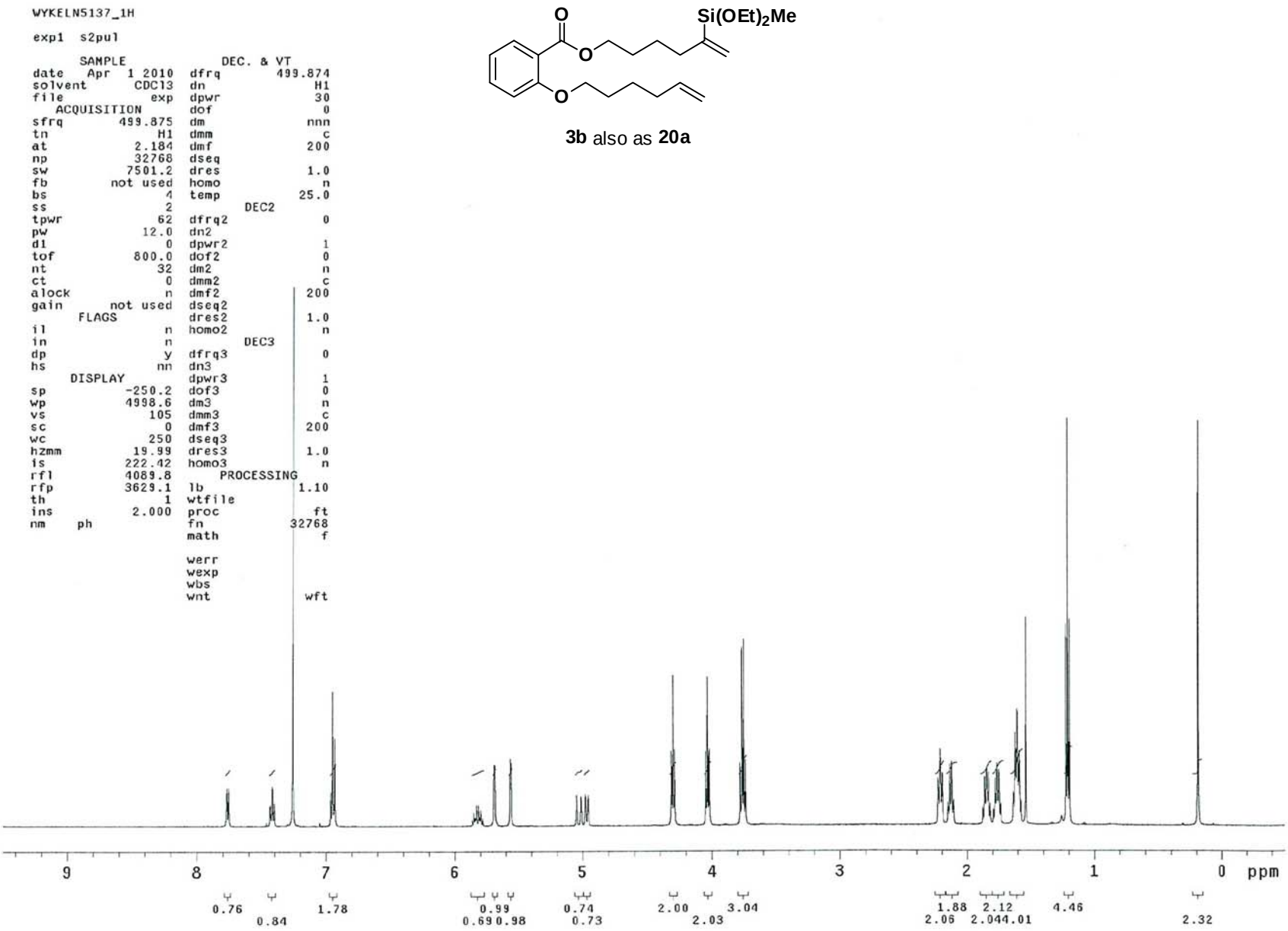
exp4 s2pul

SAMPLE		DEC. & VT	
date	Apr 5 2010	dfrq	499.874
solvent	CDCl3	dn	H1
file	exp	dpwr	48
ACQUISITION		dOf	0
sfrq	125.707	dm	yyy
tn	C13	dmm	w
at	1.092	dmf	10000
np	65536	dseq	
sw	29996.3	dres	1.0
fb	not used	homo	n
bs	16	temp	25.0
tpwr	55	DEC2	
pw	4.2	dfrq2	0
d1	0	dn2	
tof	2000.0	dpwr2	1
nt	99999	dOf2	0
ct	2112	dm2	n
alock	n	dmm2	c
gain	not used	dmf2	10000
FLAGS		dseq2	
il	n	dres2	1.0
in	n	homo2	n
dp	y	DEC3	
hs	nn	dfrq3	0
DISPLAY		dn3	
sp	-1088.7	dpwr3	1
wp	29995.3	dOf3	0
vs	30	dm3	n
sc	0	dmm3	c
wc	250	dmf3	10000
hzmm	119.98	dseq3	
is	500.00	dres3	1.0
rfl	10768.0	homo3	n
rfp	9678.3	PROCESSING	
th	3	lb	1.00
ins	100.000	wtfile	
nm cdc ph		proc	ft
		fn	not used
		math	f
		werr	
		wexp	
		wbs	
		wnt	



4a



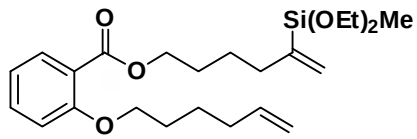


3b also as **20a**

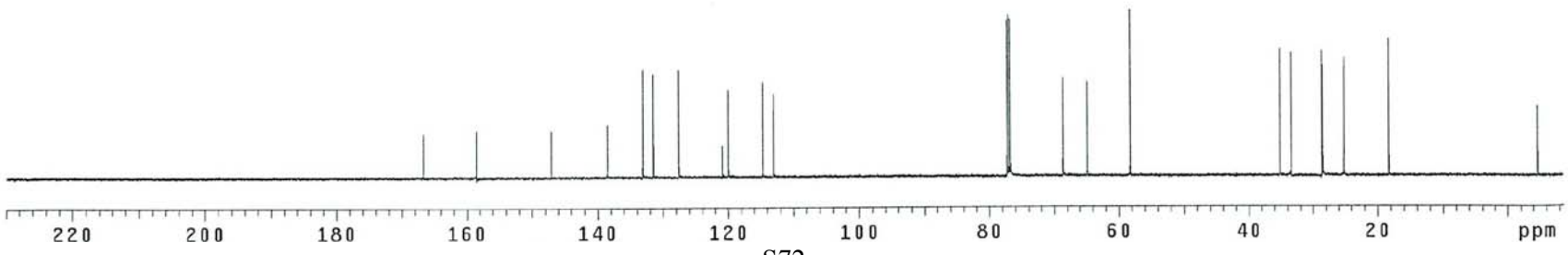
WYKELN5137_13C

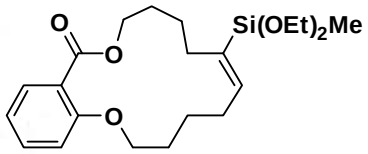
exp2 s2pul

SAMPLE		DEC. & VT	
date	Apr 1 2010	dfrq	499.874
solvent	CDCl3	dn	H1
file	exp	dpwr	48
ACQUISITION		dof	0
sfrq	125.707	dm	yyy
tn	C13	dmm	w
at	1.092	dmf	10000
np	65536	dseq	
sw	29996.3	dres	1.0
fb	not used	homo	n
bs	16	temp	25.0
tpwr	55	DEC2	
pw	4.2	dfrq2	0
d1	0	dn2	
tof	2000.0	dpwr2	1
nt	9999	dof2	0
ct	0	dm2	n
alock	n	dmm2	c
gain	not used	dmf2	10000
FLAGS		dseq2	
il	n	dres2	1.0
in	n	homo2	n
dp	y	DEC3	
hs	nn	dfrq3	0
DISPLAY		dn3	
sp	-1088.7	dpwr3	1
wp	29995.3	dof3	0
vs	27	dm3	n
sc	0	dmm3	c
wc	250	dmf3	10000
hzmm	119.98	dseq3	
is	500.00	dres3	1.0
rfl	10768.0	homo3	n
rfp	9678.3	PROCESSING	
th	2	lb	1.00
ins	100.000	wtfile	
nm cdc ph		proc	ft
		fn	not used
		math	f
		werr	
		wexp	
		wbs	
		wnt	



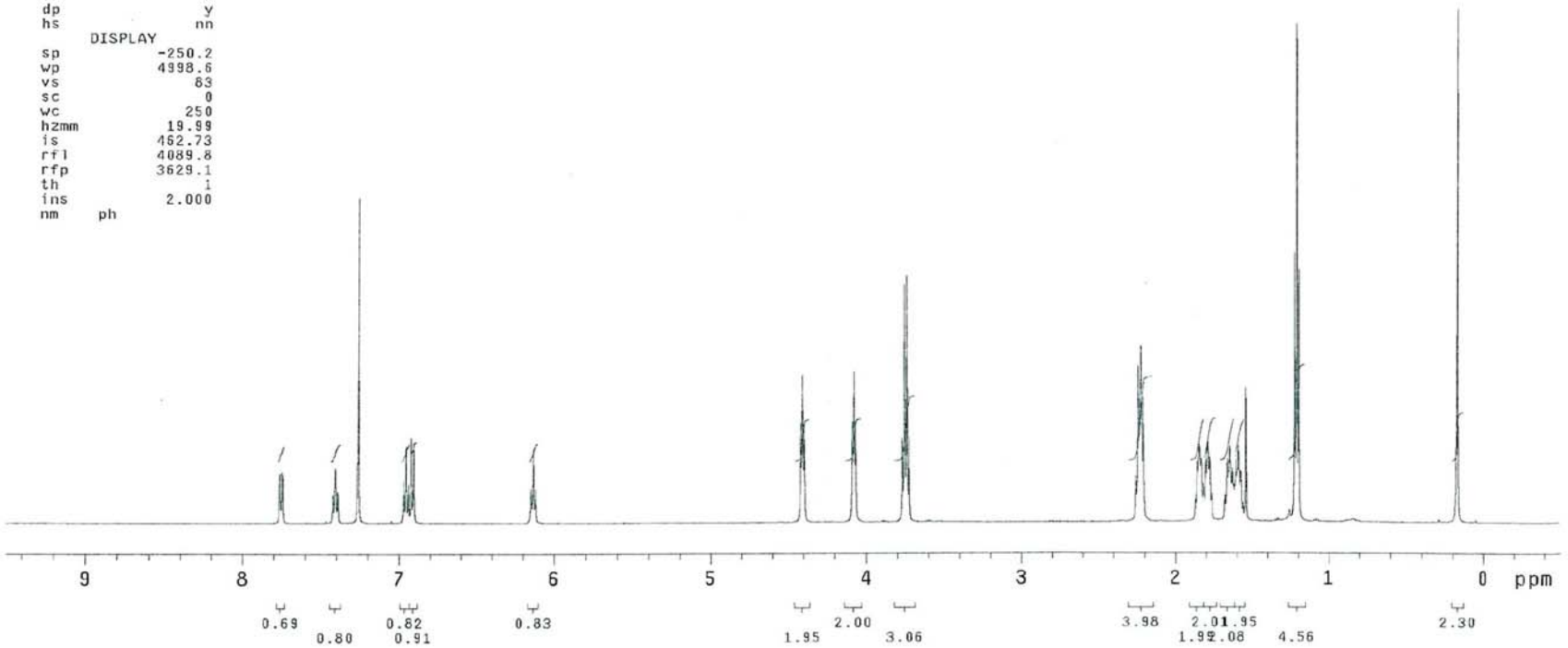
3b also as **20a**





4b also as **20**

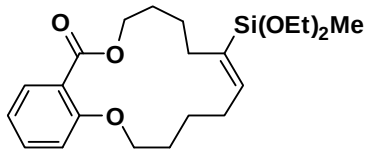
```
WYKELN10016_1H

exp1  s2pul

      SAMPLE
date    Apr  5 2010
solvent       CDCl3
file /export/home/~
ds2/vnmrsys/data/i~
500c/schreiber/WAN~
G/Pub1/WYKELN10016~
          _1H.fid
   ACQUISITION
sfrq        499.875
tn               H1
at            2.184
np            32768
sw           7501.2
fb        not used
bs                4
ss                2
tpwr             62
pw             12.0
d1                0
tof           800.0
nt               32
ct               32
alock             n
gain      not used
      FLAGS
il                n
in                n
dp                y
hs               nn
     DISPLAY
sp           -250.2
wp           4998.6
vs               63
sc                0
wc              250
hzmm          19.99
is           462.73
rfl          4089.8
rfp          3629.1
th                1
ins           2.000
nm    ph

      DEC. & VT
dfrq        499.874
dn               H1
dpwr             30
dof               0
dm              nnn
dmm               c
dmf             200
dseq
dres            1.0
homo              n
temp           25.0
     PROCESSING
lb              1.10
wtfile
proc              ft
fn             32768
math              f
werr
wexp
wbs
wnt             wft
```



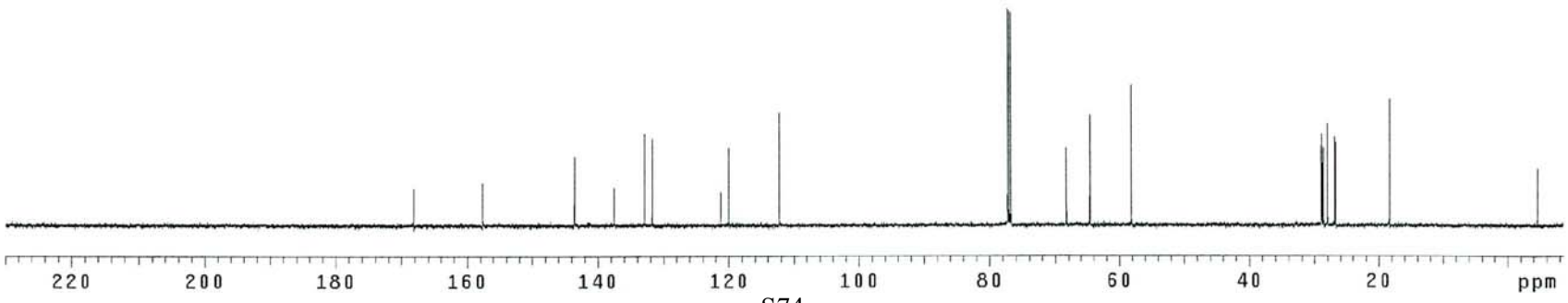
WYKELN10016_13C

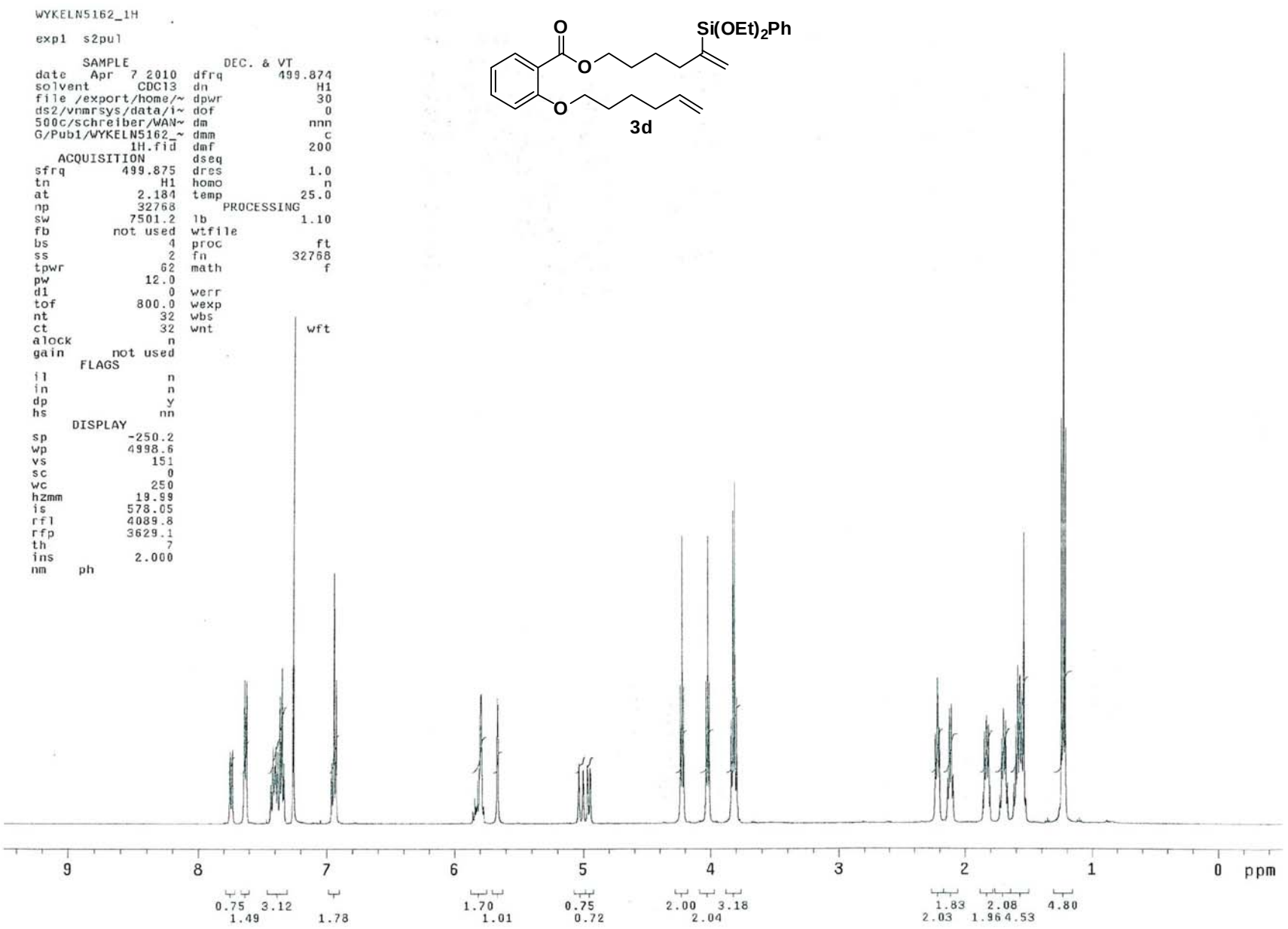
exp4 s2pul

SAMPLE		DEC. & VT	
date	Apr 5 2010	dfrq	499.874
solvent	CDCl3	dn	H1
file	exp	dpwr	48
ACQUISITION		dof	0
sfrq	125.707	dm	yyy
tn	C13	dmm	w
at	1.092	dmf	10000
np	65536	dseq	
sw	29996.3	dres	1.0
fb	not used	homo	n
bs	4	temp	25.0
tpwr	55	DEC2	
pw	4.2	dfrq2	0
d1	0	dn2	
tof	2000.0	dpwr2	1
nt	99999	dof2	0
ct	744	dm2	n
alock	n	dmm2	c
gain	not used	dmf2	10000
FLAGS		dseq2	
il	n	dres2	1.0
in	n	homo2	n
dp	y	DEC3	
hs	nn	dfrq3	0
DISPLAY		dn3	
sp	-1087.8	dpwr3	1
wp	29995.3	dof3	0
vs	35	dm3	n
sc	0	dmm3	c
wc	250	dmf3	10000
hzmm	119.98	dseq3	
is	500.00	dres3	1.0
rfl	10767.1	homo3	n
rfp	9678.3	PROCESSING	
th	2	lb	1.00
ins	100.000	wtfile	
nm cdc ph		proc	ft
		fn	not used
		math	f
		werr	
		wexp	
		wbs	
		wnt	



4b also as **20**

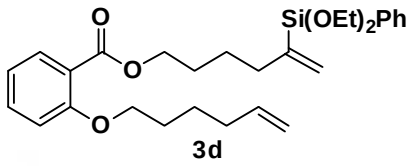



WYKELN5162_1H
exp1 s2pul
SAMPLE
date Apr 7 2010
solvent CDCl3
file /export/home/~
ds2/vnmrsys/data/i~
500c/schreiber/WAN~
G/Pub1/WYKELN5162_~
1H.fid
ACQUISITION
sfrq 499.875
tn H1
at 2.184
np 32768
sw 7501.2
fb not used
bs 4
ss 2
tpwr 62
pw 12.0
d1 0
tof 800.0
nt 32
ct 32
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -250.2
wp 4998.6
vs 151
sc 0
wc 250
hzmm 19.99
is 578.05
rfl 4089.8
rfp 3629.1
th 7
ins 2.000
nm ph
DEC. & VT
dfrq 499.874
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
temp 25.0
PROCESSING
lb 1.10
wtfile
proc ft
fn 32768
math f
werr
wexp
wbs
wnt wft
O
O
O
Si(OEt)2Ph
3d
9
8
7
6
5
4
3
2
1
0 ppm
0.75 3.12
1.49
1.78
1.70
1.01
0.75
0.72
2.00 3.18
2.04
1.83
2.03
2.08
1.96
4.80
4.53

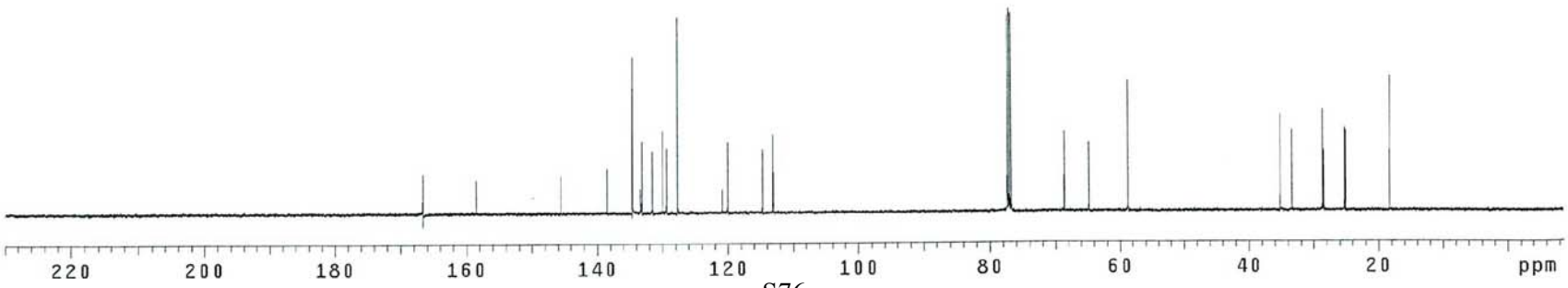
WYKELN5162_13C

exp2 s2pul

SAMPLE		DEC. & VT	
date	Apr 7 2010	dfrq	499.874
solvent	CDCl3	dn	H1
file	exp	dpwr	48
ACQUISITION		dof	0
sfrq	125.707	dm	yyy
tn	C13	dmm	w
at	1.092	dmf	10000
np	65536	dseq	
sw	29996.3	dres	1.0
fb	not used	homo	n
bs	16	temp	25.0
tpwr	55	DEC2	
pw	4.2	dfrq2	0
d1	0	dn2	
tof	2000.0	dpwr2	1
nt	99999	dof2	0
ct	1200	dm2	n
alock	n	dmm2	c
gain	not used	dmf2	10000
FLAGS		dseq2	
il	n	dres2	1.0
in	n	homo2	n
dp	y	DEC3	
hs	nn	dfrq3	0
DISPLAY		dn3	
sp	-1088.7	dpwr3	1
wp	29995.3	dof3	0
vs	33	dm3	n
sc	0	dmm3	c
wc	250	dmf3	10000
hzmm	119.98	dseq3	
is	500.00	dres3	1.0
rfl	10768.0	homo3	n
rfp	9678.3	PROCESSING	
th	3	lb	1.00
ins	100.000	wtfile	
nm cdc ph		proc	ft
		fn	not used
		math	f
		werr	
		wexp	
		wbs	
		wnt	



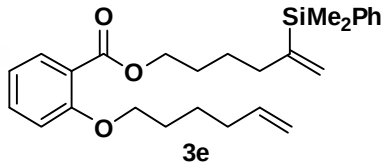
3d



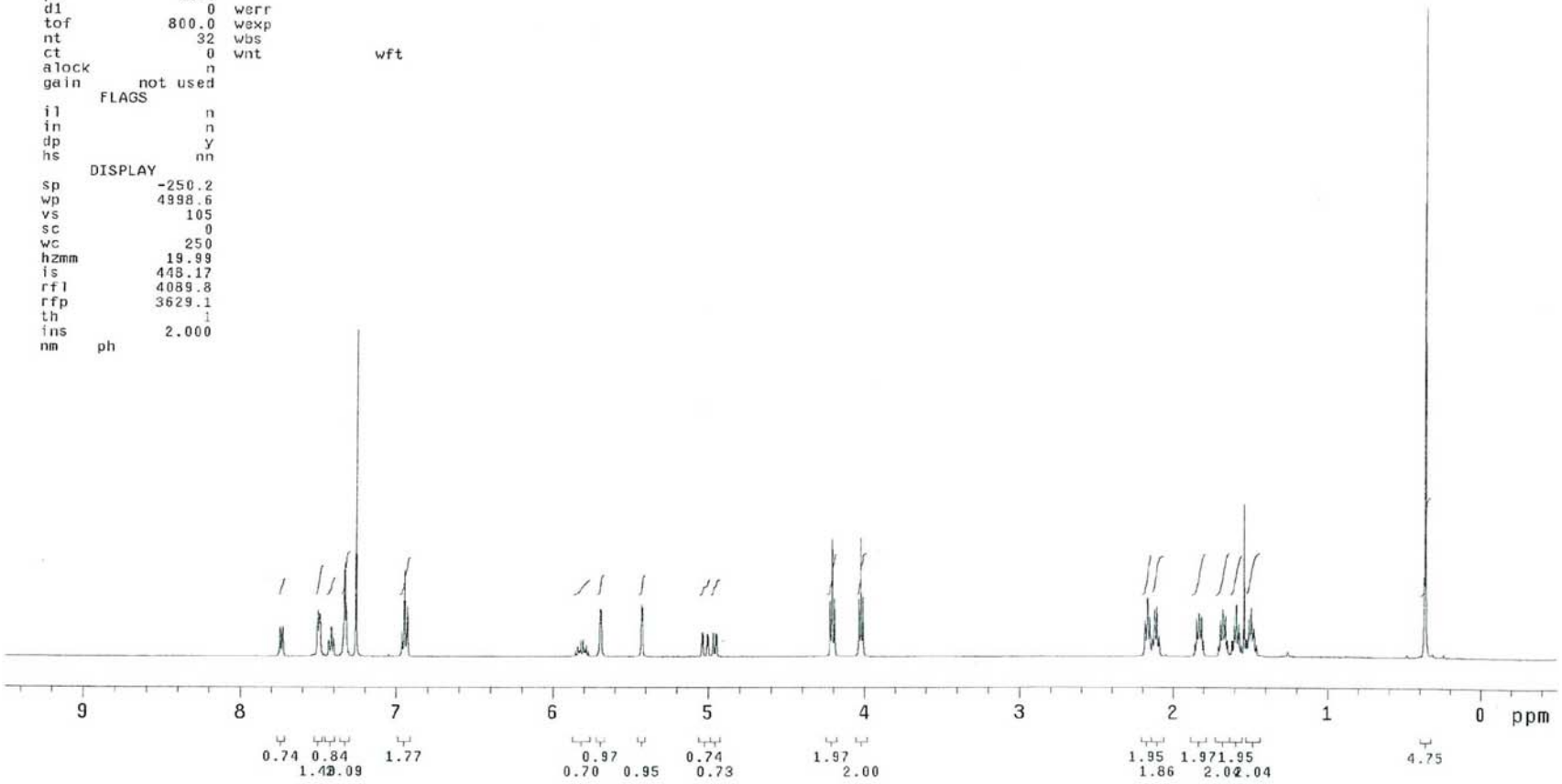
WYKELN5135_1H

exp1 s2pul

SAMPLE		DEC. & VT	
date	Apr 1 2010	dfrq	499.874
solvent	CDCl3	dn	H1
file	/export/home/~	dpwr	30
	ds2/vnmrsys/data/i~	dof	0
	500c/schreiber/WAN~	dm	nnn
	G/Pub1/WYKELN5135_~	dmm	c
	1H.fid	dmf	200
ACQUISITION		dseq	
sfrq	499.875	dres	1.0
tn	H1	homo	n
at	2.184	temp	25.0
np	32768	PROCESSING	
sw	7501.2	lb	1.10
fb	not used	wtfile	
bs	4	proc	ft
ss	2	fn	32768
tpwr	62	math	f
pw	12.0		
d1	0	werr	
tof	800.0	wexp	
nt	32	wbs	
ct	0	wnt	wft
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-250.2		
wp	4998.6		
vs	105		
sc	0		
wc	250		
hzmm	19.99		
is	448.17		
rfl	4089.8		
rfp	3629.1		
th	1		
ins	2.000		
nm	ph		



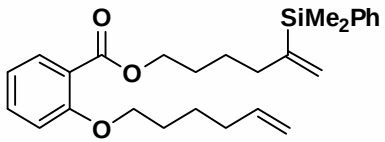
3e



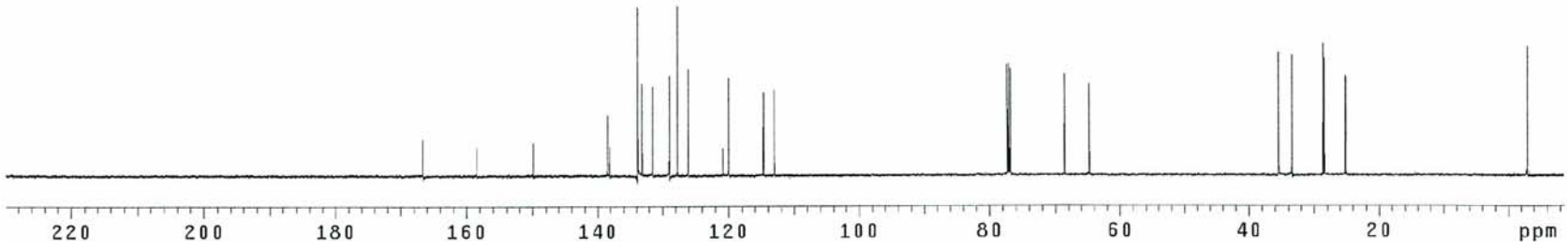
WYKELN5135_13C

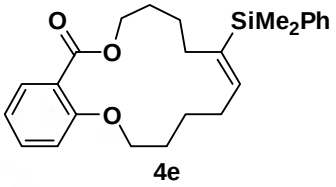
exp2 s2pul

SAMPLE		DEC. & VT	
date	Apr 1 2010	dfrq	499.874
solvent	CDCl3	dn	H1
file	exp	dpwr	48
ACQUISITION		dof	0
sfrq	125.707	dm	yyy
tn	C13	dmm	w
at	1.092	dmf	10000
np	65536	dseq	
sw	29996.3	dres	1.0
fb	not used	homo	n
bs	4	temp	25.0
tpwr	55	DEC2	
pw	4.2	dfrq2	0
dl	0	dn2	
tof	2000.0	dpwr2	1
nt	9999	dof2	0
ct	0	dm2	n
alock	n	dmm2	c
gain	not used	dmf2	10000
FLAGS		dseq2	
il	n	dres2	1.0
in	n	homo2	n
dp	y	DEC3	
hs	nn	dfrq3	0
DISPLAY		dn3	
sp	-1091.5	dpwr3	1
wp	29995.3	dof3	0
vs	27	dm3	n
sc	0	dmm3	c
wc	250	dmf3	10000
hzmm	119.98	dseq3	
is	500.00	dres3	1.0
rfl	10770.7	homo3	n
rfp	9678.3	PROCESSING	
th	2	lb	1.00
ins	100.000	wtfile	
nm cdc ph		proc	ft
		fn	not used
		math	f
		werr	
		wexp	
		wbs	
		wnt	



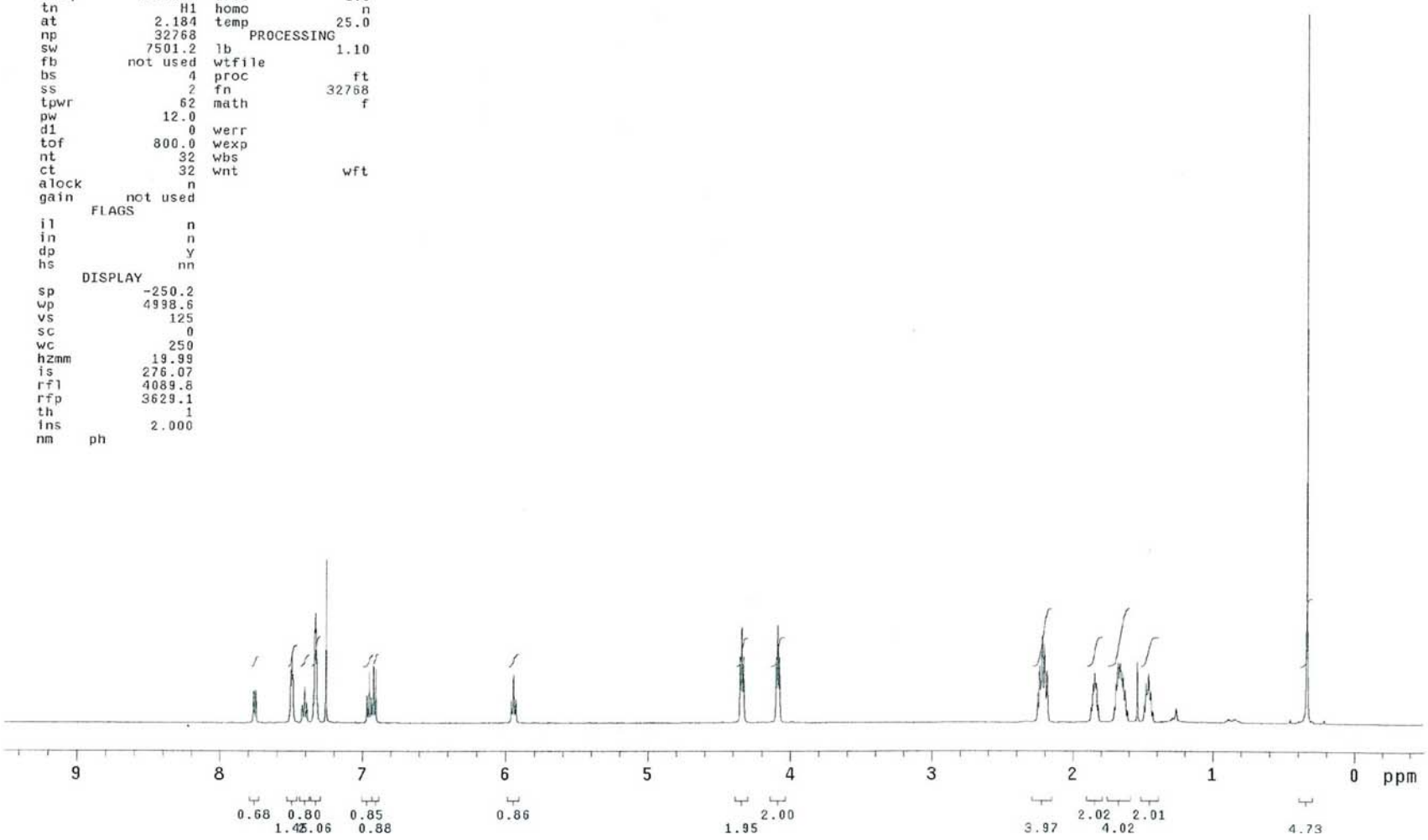
3e





4e

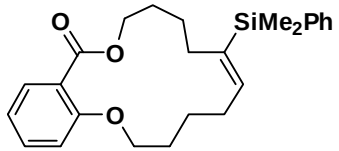
```
WYKELN10017_1H

exp1  s2pul

      SAMPLE
date    Apr  5 2010
solvent       CDCl3
file /export/home/~
ds2/vnmrsys/data/i~
500c/schreiber/WAN~
G/Pub1/WYKELN10017~
                _1H.fid
   ACQUISITION
sfrq         499.875
tn                H1
at             2.184
np             32768
sw            7501.2
fb         not used
bs                 4
ss                 2
tpwr              62
pw              12.0
d1                 0
tof            800.0
nt                32
ct                32
alock              n
gain       not used
      FLAGS
il                 n
in                 n
dp                 y
hs                nn
     DISPLAY
sp            -250.2
wp            4998.6
vs               125
sc                 0
wc               250
hzmm           19.99
is            276.07
rfl           4089.8
rfp           3629.1
th                 1
ins            2.000
nm     ph

      DEC. & VT
dfrq         499.874
dn                H1
dpwr              30
dof                0
dm               nnn
dmm                c
dmf              200
dseq
dres             1.0
homo               n
temp            25.0
     PROCESSING
lb               1.10
wtfile
proc               ft
fn              32768
math               f

werr
wexp
wbs
wnt              wft
```



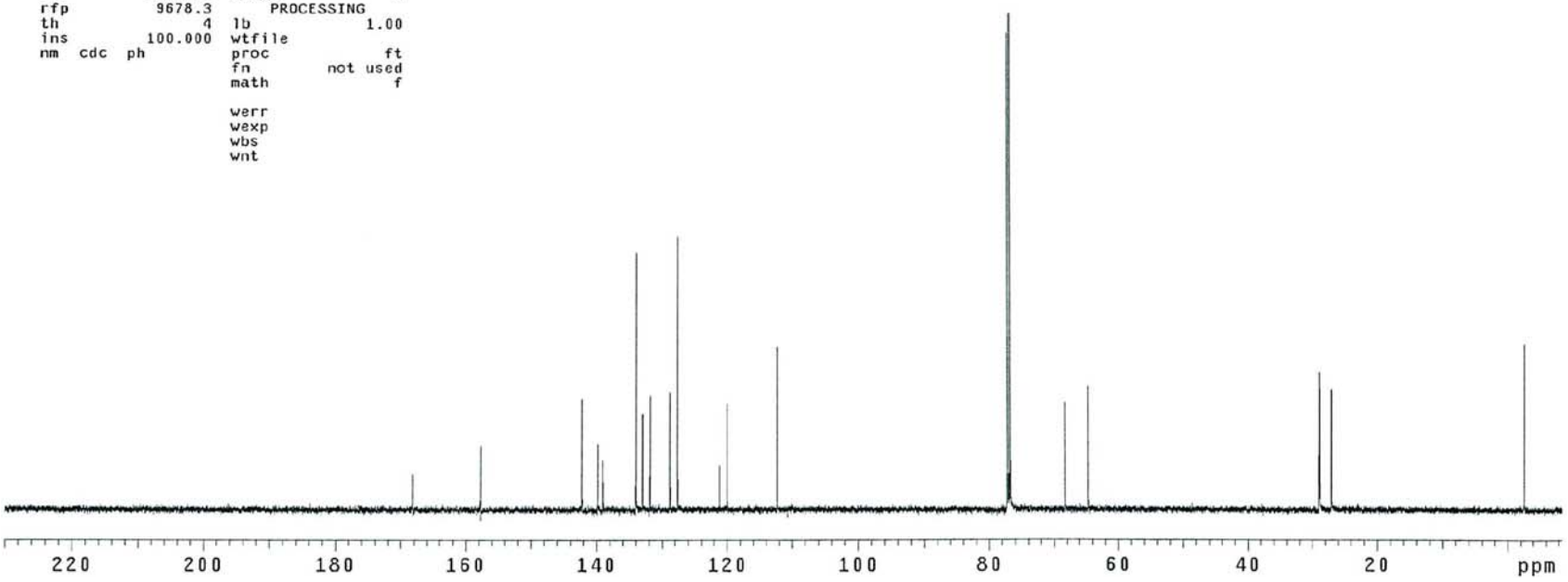
WYKELN10017_13C

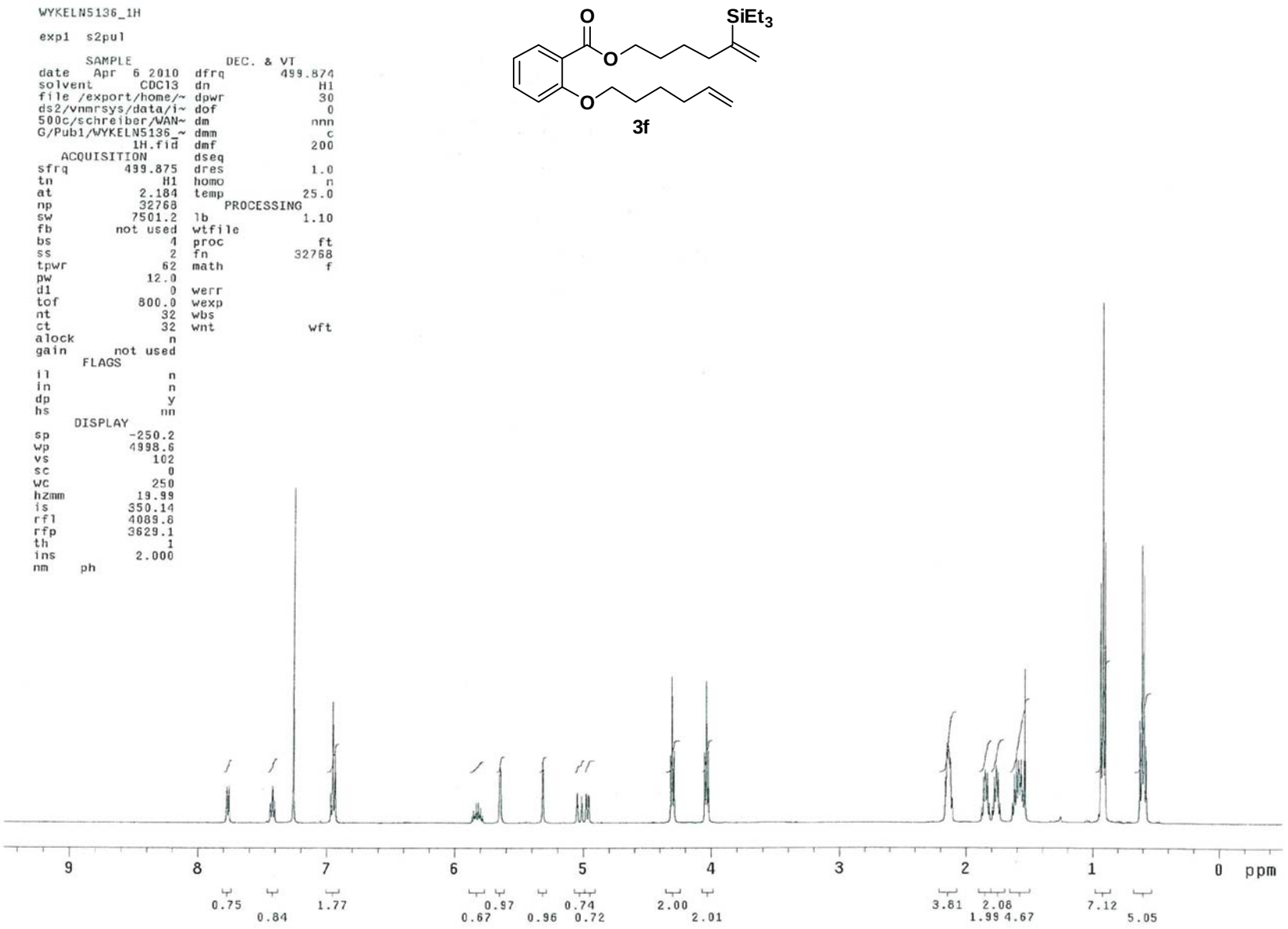
exp4 s2pul

SAMPLE		DEC. & VT	
date	Apr 5 2010	dfrq	499.874
solvent	CDCl3	dn	H1
file	exp	dpwr	48
ACQUISITION		dof	0
sfrq	125.707	dm	yyy
tn	C13	dmm	w
at	1.092	dmf	10000
np	65536	dseq	
sw	29996.3	dres	1.0
fb	not used	homo	n
bs	16	temp	25.0
tpwr	55	DEC2	
pw	4.2	dfrq2	0
d1	0	dn2	
tof	2000.0	dpwr2	1
nt	99999	dof2	0
ct	2048	dm2	n
alock	n	dmm2	c
gain	not used	dmf2	10000
FLAGS		dseq2	
il	n	dres2	1.0
in	n	homo2	n
dp	y	DEC3	
hs	nn	dfrq3	0
DISPLAY		dn3	
sp	-1087.8	dpwr3	1
wp	29995.3	dof3	0
vs	80	dm3	n
sc	0	dmm3	c
wc	250	dmf3	10000
hzmm	119.98	dseq3	
is	500.00	dres3	1.0
rfl	10767.1	homo3	n
rfp	9678.3	PROCESSING	
th	4	lb	1.00
ins	100.000	wtfile	
nm cdc ph		proc	ft
		fn	not used
		math	f
		werr	
		wexp	
		wbs	
		wnt	



4e

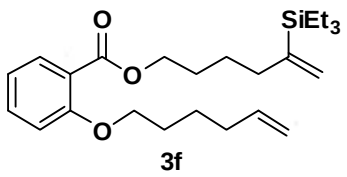



WYKELN5136_1H
exp1 s2pul
SAMPLE
date Apr 6 2010
solvent CDCl3
file /export/home/~
ds2/vnmrsys/data/i~
500c/schreiber/WAN~
G/Pub1/WYKELN5136_~
1H.fid
ACQUISITION
sfrq 499.875
tn H1
at 2.184
np 32768
sw 7501.2
fb not used
bs 4
ss 2
tpwr 62
pw 12.0
d1 0
tof 800.0
nt 32
ct 32
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -250.2
wp 4998.6
vs 102
sc 0
wc 250
hzmm 19.99
is 350.14
rfl 4089.8
rfp 3629.1
th 1
ins 2.000
nm ph
DEC. & VT
dfrq 499.874
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
temp 25.0
PROCESSING
lb 1.10
wtfile
proc ft
fn 32768
math f
werr
wexp
wbs
wnt wft
O
O
O
SiEt3
3f
9
8
7
6
5
4
3
2
1
0 ppm
0.75
0.84
1.77
0.67
0.97
0.96
0.74
0.72
2.00
2.01
3.81
2.08
1.99
4.67
7.12
5.05

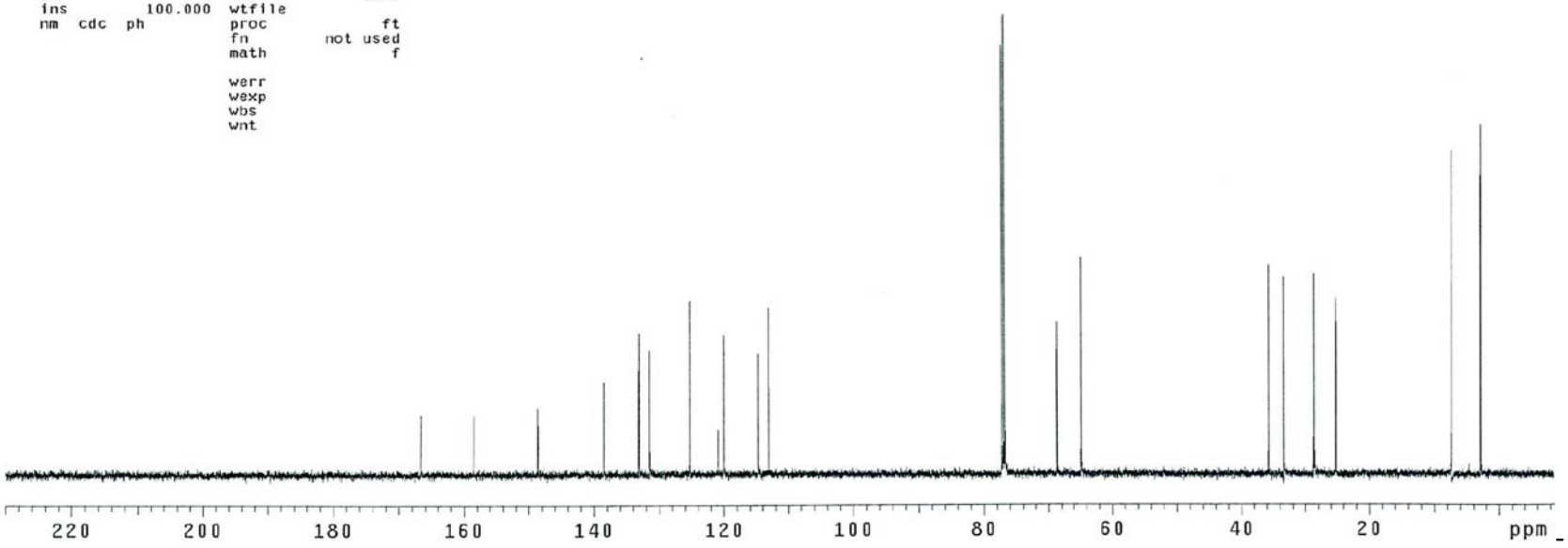
WYKELN5136_13C

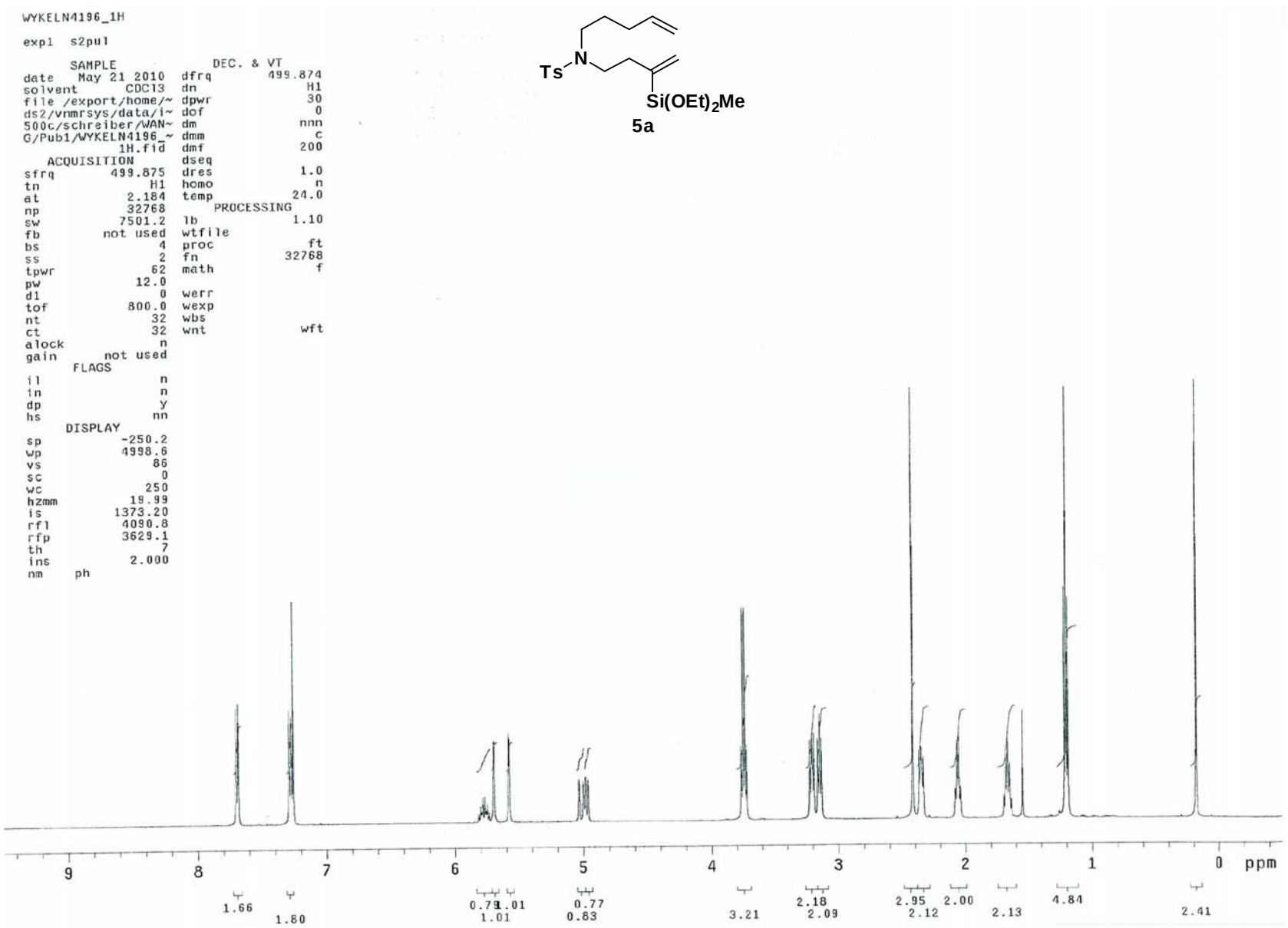
exp4 s2pul

SAMPLE		DEC. & VT	
date	Apr 6 2010	dfrq	499.874
solvent	CDCl3	dn	H1
file	exp	dpwr	48
ACQUISITION		dof	0
sfrq	125.707	dm	yyy
tn	C13	dmm	w
at	1.092	dmf	10000
np	65536	dseq	
sw	29995.3	dres	1.0
fb	not used	homo	n
bs	16	temp	25.0
tpwr	55	DEC2	
pw	4.2	dfrq2	0
d1	0	dn2	
tof	2000.0	dpwr2	1
nt	99999	dof2	0
ct	960	dm2	n
alock	n	dmm2	c
gain	not used	dmf2	10000
FLAGS		dseq2	
il	n	dres2	1.0
in	n	homo2	n
dp	y	DEC3	
hs	nn	dfrq3	0
DISPLAY		dn3	
sp	-1087.8	dpwr3	1
wp	29995.3	dof3	0
vs	75	dm3	n
sc	0	dmm3	c
wc	250	dmf3	10000
hzmm	119.98	dseq3	
is	500.00	dres3	1.0
rfl	10767.1	homo3	n
rfp	9678.3	PROCESSING	
th	6	lb	1.00
ins	100.000	wtfile	
nm cdc ph		proc	ft
		fn	not used
		math	f
		werr	
		wexp	
		wbs	
		wnt	



3f

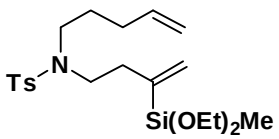



WYKELN4196_1H
expl s2pul
SAMPLE
date May 21 2010
solvent CDCl3
file /export/home/~
ds2/vnmrsys/data/i~
500c/schreiber/WAN~
G/Pub1/WYKELN4196_~
1H.fid
ACQUISITION
sfrq 499.875
tn H1
at 2.184
np 32768
sw 7501.2
fb not used
bs 4
ss 2
tpwr 62
pw 12.0
d1 0
tof 800.0
nt 32
ct 32
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -250.2
wp 4998.6
vs 86
sc 0
wc 250
hzmm 19.99
is 1373.20
rfl 4090.8
rfp 3629.1
th 7
ins 2.000
nm ph
DEC. & VT
dfrq 499.874
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
temp 24.0
PROCESSING
lb 1.10
wtfile
proc ft
fn 32768
math f
werr
wexp
wbs
wnt wft
Ts
N
Si(OEt)2Me
5a
9
8
7
6
5
4
3
2
1
0 ppm
1.66
1.80
0.79
1.01
1.01
0.77
0.83
3.21
2.18
2.09
2.95
2.12
2.00
2.13
4.84
2.41

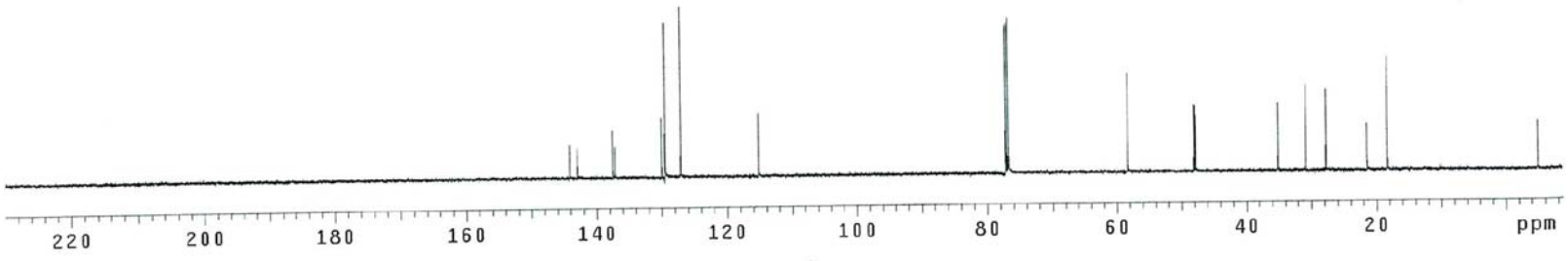
WYKELN4196_13C

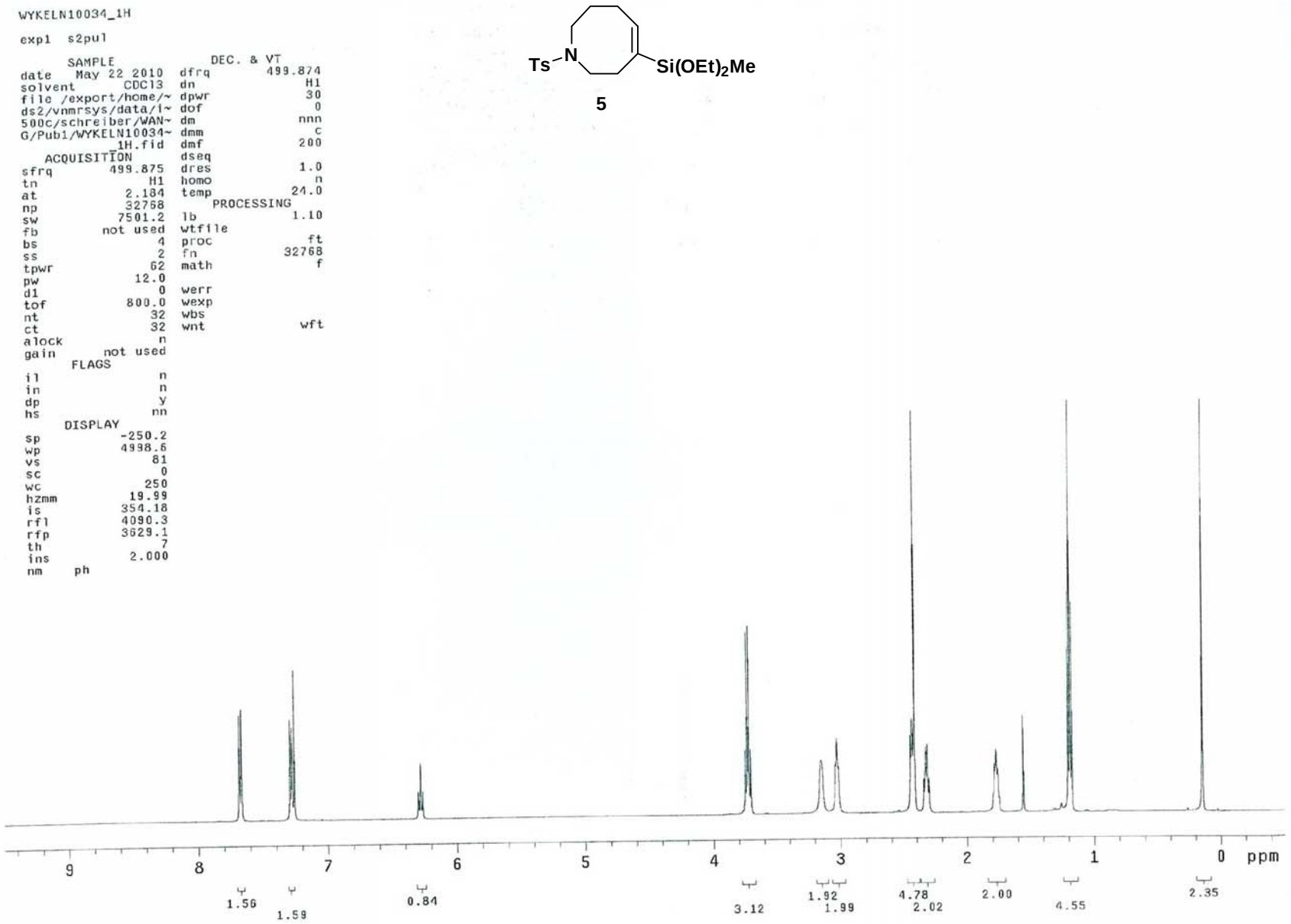
exp2 s2pul

SAMPLE		DEC. & VT	
date	May 21 2010	dfrq	499.874
solvent	CDCl3	dn	H1
file	exp	dpwr	48
ACQUISITION		dof	0
sfrq	125.707	dm	yyy
tn	C13	dmm	w
at	1.092	dmf	10000
np	65536	dseq	
sw	29996.3	dres	1.0
fb	not used	homo	n
bs	32	temp	24.0
tpwr	55	DEC2	
pw	4.2	dfrq2	0
d1	0	dn2	
tof	2000.0	dpwr2	1
nt	99999	dof2	0
ct	704	dm2	n
alock	n	dmm2	c
gain	not used	dmf2	10000
FLAGS		dseq2	
il	n	dres2	1.0
in	n	homo2	n
dp	y	DEC3	
hs	nn	dfrq3	0
DISPLAY		dn3	
sp	-1089.7	dpwr3	1
wp	29995.3	dof3	0
vs	27	dm3	n
sc	0	dmm3	c
wc	250	dmf3	10000
hzmm	119.98	dseq3	
is	500.00	dres3	1.0
rfl	10768.9	homo3	n
rfp	9678.3	PROCESSING	
th	3	lb	1.00
ins	100.000	wtfile	
nm cdc ph		proc	ft
		fn	not used
		math	f
		werr	
		wexp	
		wbs	
		wnt	



5a

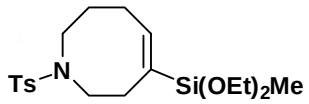



WYKELN10034_1H
expl s2pul
SAMPLE
date May 22 2010
solvent CDCl3
file /export/home/~
ds2/vnmrsys/data/i~
500c/schreiber/WAN~
G/Pub1/WYKELN10034~
1H.fid
ACQUISITION
sfrq 499.875
tn H1
at 2.184
np 32768
sw 7501.2
fb not used
bs 4
ss 2
tpwr 62
pw 12.0
d1 0
tof 800.0
nt 32
ct 32
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -250.2
wp 4998.6
vs 81
sc 0
wc 250
hzmm 19.99
is 354.18
rfl 4090.3
rfp 3629.1
th 7
ins 2.000
nm ph
DEC. & VT
dfrq 499.874
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
temp 24.0
PROCESSING
lb 1.10
wtfile
proc ft
fn 32768
math f
werr
wexp
wbs
wnt wft
Ts
N
Si(OEt)2Me
5
9
8
7
6
5
4
3
2
1
0 ppm
1.56
1.59
0.84
3.12
1.92
1.99
4.78
2.02
2.00
4.55
2.35

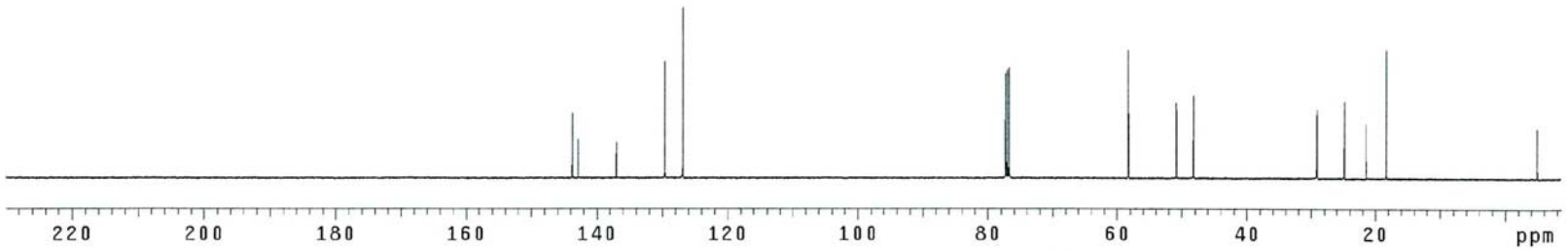
WYKELN10034_13C

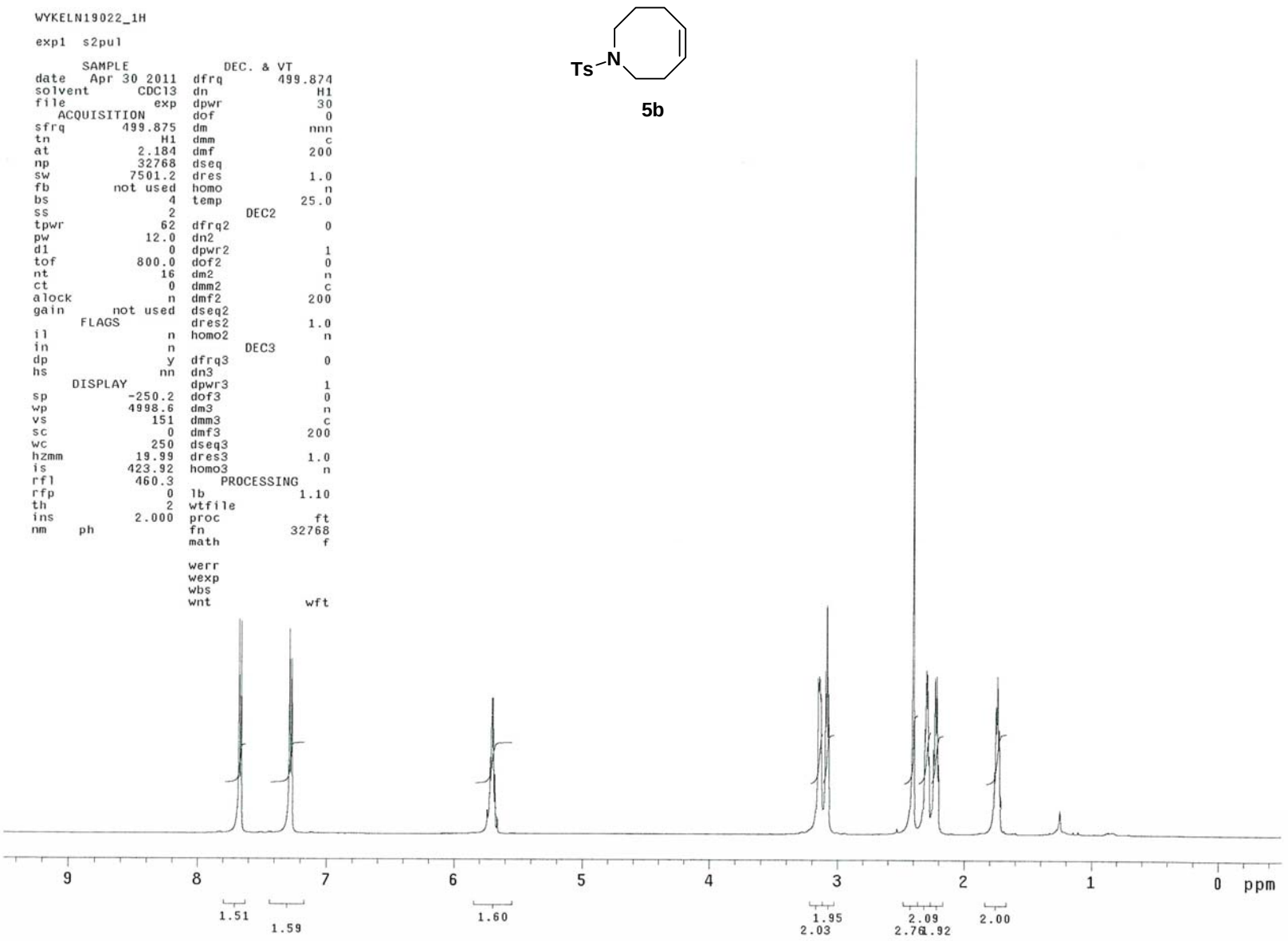
exp1 s2pul

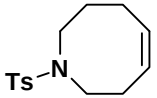
SAMPLE		DEC. & VT	
date	May 22 2010	dfrq	499.874
solvent	CDCl3	dn	H1
file	exp	dpwr	48
ACQUISITION		dof	0
sfrq	125.707	dm	yyy
tn	C13	dmm	w
at	1.092	dmf	10000
np	65536	dseq	
sw	29996.3	dres	1.0
fb	not used	homo	n
bs	32	temp	24.0
tpwr	55	DEC2	
pw	4.2	dfrq2	0
d1	0	dn2	
tof	2000.0	dpwr2	1
nt	99999	dof2	0
ct	1248	dm2	n
alock	n	dmm2	c
gain	not used	dmf2	10000
FLAGS		dseq2	
il	n	dres2	1.0
in	n	homo2	n
dp	y	DEC3	
hs	nn	dfrq3	0
DISPLAY		dn3	
sp	-1090.6	dpwr3	1
wp	29995.3	dof3	0
vs	27	dm3	n
sc	0	dmm3	c
wc	250	dmf3	10000
hzmm	119.98	dseq3	
is	500.00	dres3	1.0
rfl	10769.8	homo3	n
rfp	9678.3	PROCESSING	
th	2	lb	1.00
ins	100.000	wtfile	
nm cdc ph		proc	ft
		fn	not used
		math	f
		werr	
		wexp	
		wbs	
		wnt	



5

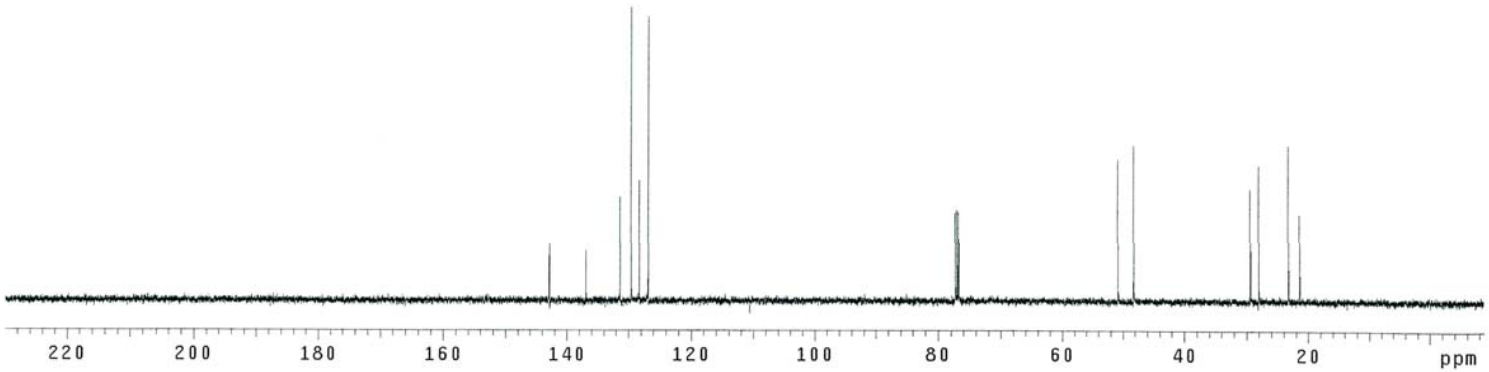


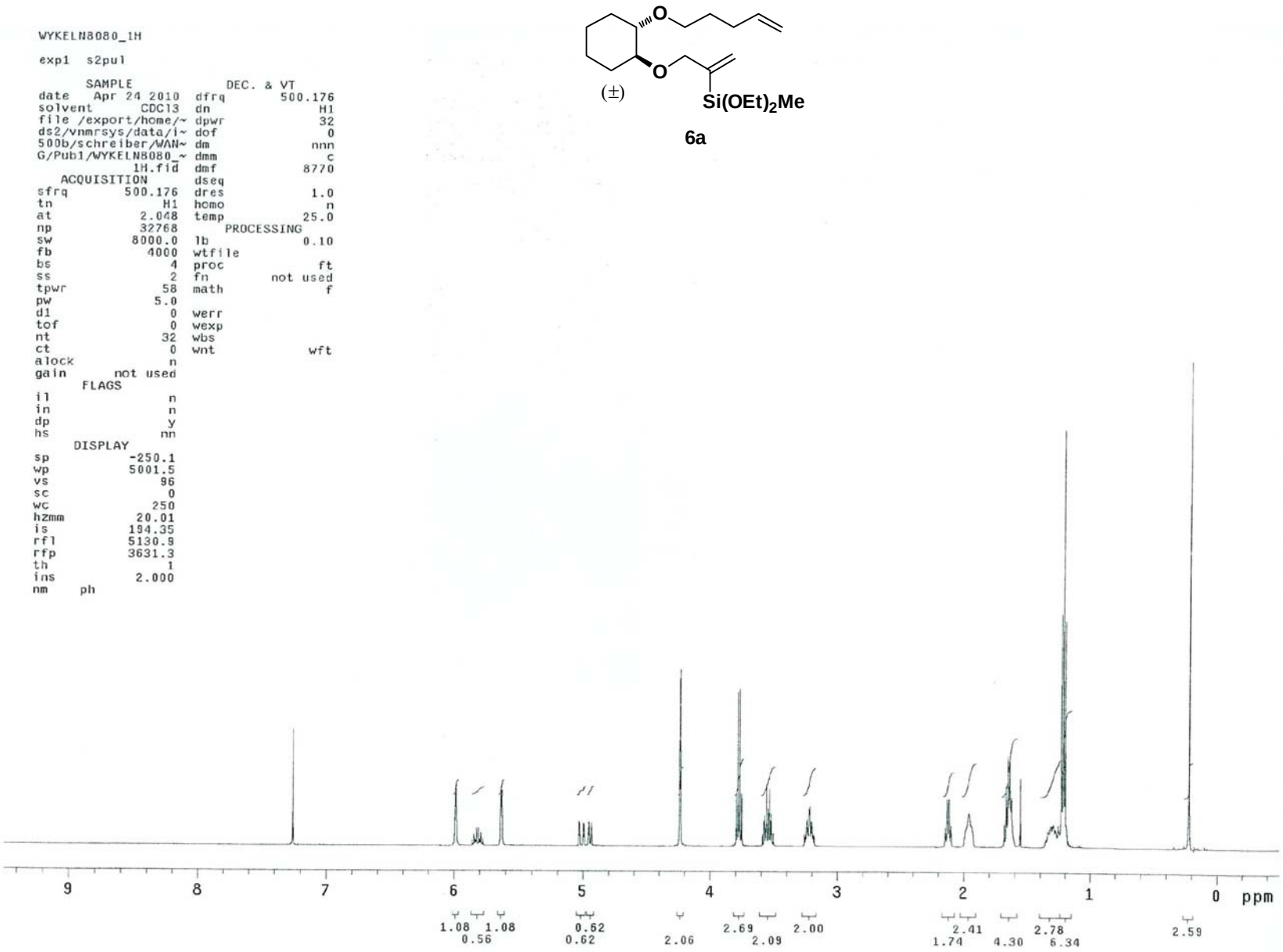

WYKELN19022_1H
exp1 s2pul
SAMPLE
date Apr 30 2011
solvent CDCl3
file exp
ACQUISITION
sfrq 499.875
tn H1
at 2.184
np 32768
sw 7501.2
fb not used
bs 4
ss 2
tpwr 62
pw 12.0
d1 0
tof 800.0
nt 16
ct 0
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -250.2
wp 4998.6
vs 151
sc 0
wc 250
hzmm 19.99
is 423.92
rfl 460.3
rfp 0
th 2
ins 2.000
nm ph
DEC. & VT
dfrq 499.874
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
temp 25.0
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 200
dseq2
dres2 1.0
homo2 n
DEC3
dfrq3 0
dn3
dpwr3 1
dof3 0
dm3 n
dmm3 c
dmf3 200
dseq3
dres3 1.0
homo3 n
PROCESSING
lb 1.10
wtfile
proc ft
fn 32768
math f
werr
wexp
wbs
wnt wft
Ts
N
5b
9
8
7
6
5
4
3
2
1
0
ppm
1.51
1.59
1.60
1.95
2.03
2.09
2.76
1.92
2.00



5b

```
WYKELN19022_13C

exp2  s2pul

      SAMPLE           DEC. & VT
date   Apr 30 2011  dfrq        499.874
solvent      CDCl3  dn               H1
file           exp  dpwr             48
   ACQUISITION      dof               0
sfrq       125.707  dm              yyy
tn             C13  dmm               w
at           1.092  dmf            8929
np           65536  dseq
sw         29996.3  dres             1.0
fb       not used   homo               n
bs             16   temp            25.0
tpwr           55         DEC2
pw            4.8   dfrq2              0
d1              0    dn2
tof         2000.0   dpwr2              1
nt            9999   dof2               0
ct               0   dm2                n
alock            n   dmm2               c
gain     not used    dmf2           10000
      FLAGS          dseq2
il               n   dres2            1.0
in               n   homo2              n
dp               y         DEC3
hs              nn   dfrq3              0
     DISPLAY         dn3
sp         -1094.2   dpwr3              1
wp         29995.3   dof3               0
vs              50   dm3                n
sc               0   dmm3               c
wc             250   dmf3           10000
hzmm        119.98   dseq3
is          500.00   dres3            1.0
rfl         1095.1   homo3              n
rfp              0        PROCESSING
th               6   lb              1.00
ins         100.000  wtfile
nm  cdc  ph          proc              ft
                     fn          not used
                     math               f

                     werr
                     wexp
                     wbs
                     wnt
```



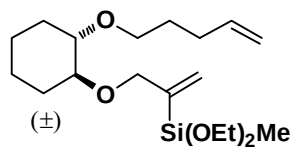

WYKELN8080_1H
exp1 s2pul
SAMPLE
date Apr 24 2010
solvent CDCl3
file /export/home/~
ds2/vnmrsys/data/i~
500b/schreiber/WAN~
G/Pub1/WYKELN8080_~
1H.fid
ACQUISITION
sfrq 500.176
tn H1
at 2.048
np 32768
sw 8000.0
fb 4000
bs 4
ss 2
tpwr 58
pw 5.0
d1 0
tof 0
nt 32
ct 0
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -250.1
wp 5001.5
vs 96
sc 0
wc 250
hzmm 20.01
is 194.35
rfl 5130.9
rfp 3631.3
th 1
ins 2.000
nm ph
DEC. & VT
dfrq 500.176
dn H1
dpwr 32
dof 0
dm nnn
dmm c
dmf 8770
dseq
dres 1.0
homo n
temp 25.0
PROCESSING
lb 0.10
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt wft
O
O
(±)
Si(OEt)2Me
9
8
7
6
5
4
3
2
1
0
ppm
1.08
1.08
0.56
0.62
0.62
2.06
2.69
2.09
2.00
2.41
1.74
4.30
2.78
6.34
2.59

6a

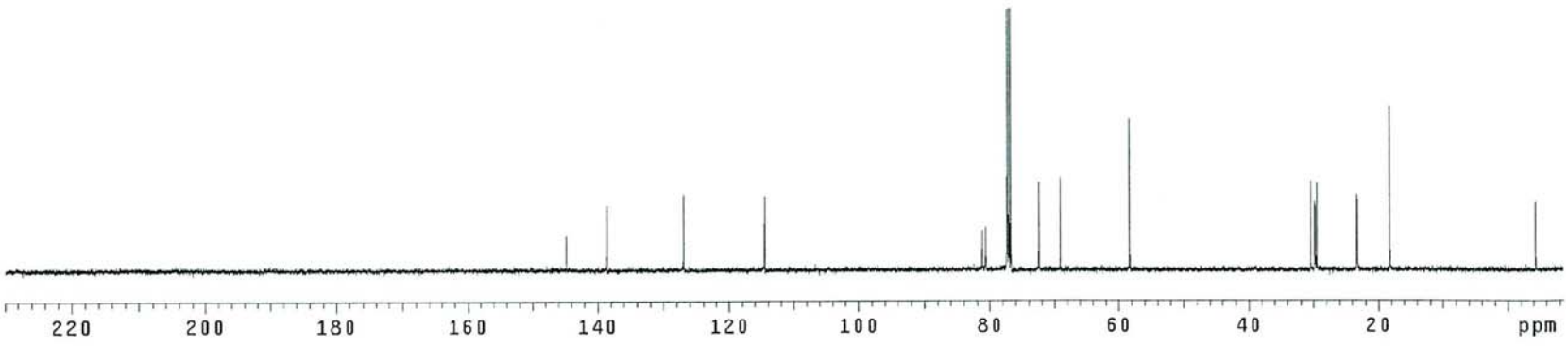
WYKELN8080_13C

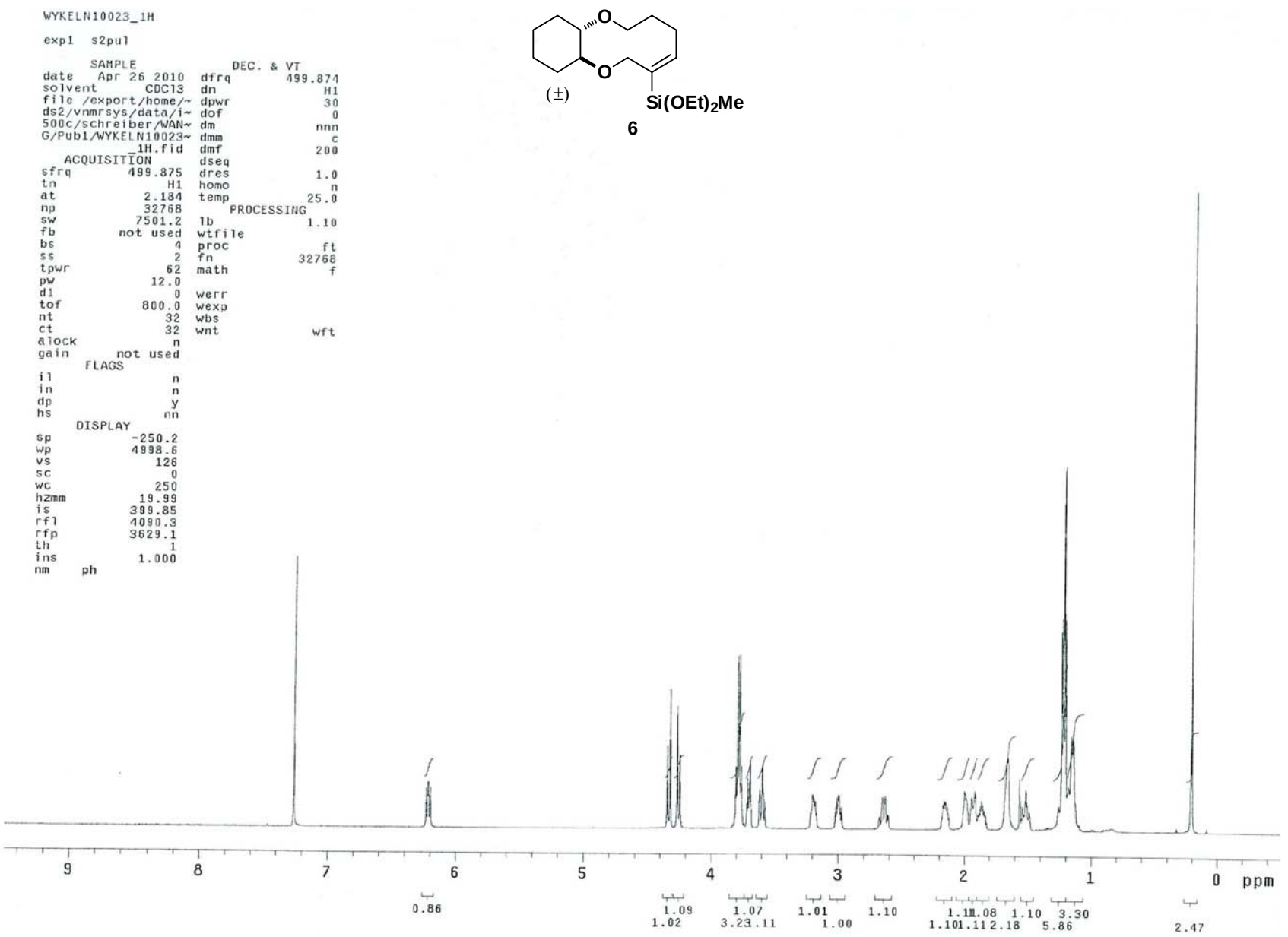
exp2 s2pul

SAMPLE		DEC. & VT	
date	Apr 25 2010	dfrq	499.874
solvent	CDCl3	dn	H1
file	exp	dpwr	48
ACQUISITION		dof	0
sfrq	125.707	dm	yyy
tn	C13	dmm	w
at	1.092	dmf	10000
np	65536	dseq	
sw	29996.3	dres	1.0
fb	not used	homo	n
bs	16	temp	25.0
tpwr	55	DEC2	
pw	2.0	dfrq2	0
d1	0	dn2	
tof	2000.0	dpwr2	1
nt	9999	dof2	0
ct	848	dm2	n
alock	n	dmm2	c
gain	not used	dmf2	10000
FLAGS		dseq2	
il	n	dres2	1.0
in	n	homo2	n
dp	y	DEC3	
hs	nn	dfrq3	0
DISPLAY		dn3	
sp	-1088.7	dpwr3	1
wp	29995.3	dof3	0
vs	42	dm3	n
sc	0	dmm3	c
wc	250	dmf3	10000
hzmm	119.98	dseq3	
is	500.00	dres3	1.0
rfl	10768.0	homo3	n
rfp	9678.3	PROCESSING	
th	3	lb	1.00
ins	100.000	wtfile	
nm cdc ph		proc	ft
		fn	not used
		math	f
		werr	
		wexp	
		wbs	
		wnt	



6a

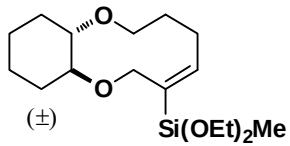



WYKELN10023_1H
expl s2pul
SAMPLE
date Apr 26 2010
solvent CDCl3
file /export/home/~
ds2/vnmrsys/data/i~
500c/schreiber/WAN~
G/Pub1/WYKELN10023~
_1H.fid
ACQUISITION
sfrq 499.875
tn H1
at 2.184
np 32768
sw 7501.2
fb not used
bs 4
ss 2
tpwr 62
pw 12.0
d1 0
tof 800.0
nt 32
ct 32
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -250.2
wp 4998.6
vs 126
sc 0
wc 250
hzmm 19.99
is 399.85
rfl 4090.3
rfp 3629.1
th 1
ins 1.000
nm ph
DEC. & VT
dfrq 499.874
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
temp 25.0
PROCESSING
lb 1.10
wtfile
proc ft
fn 32768
math f
werr
wexp
wbs
wnt wft
O
O
(±)
Si(OEt)2Me
6
9
8
7
6
5
4
3
2
1
0
ppm
0.86
1.09
1.02
1.07
3.23
1.11
1.01
1.00
1.10
1.10
1.08
1.11
2.18
1.10
3.30
5.86
2.47

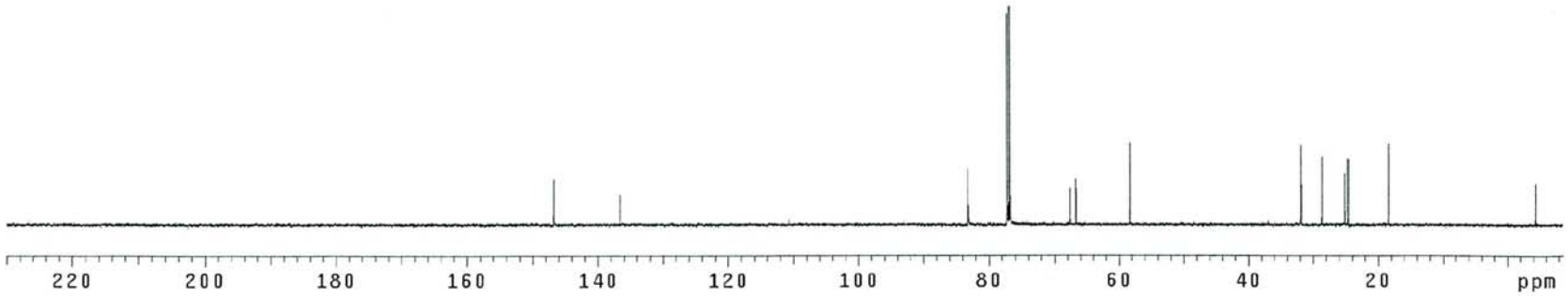
WYKELN10023_13C

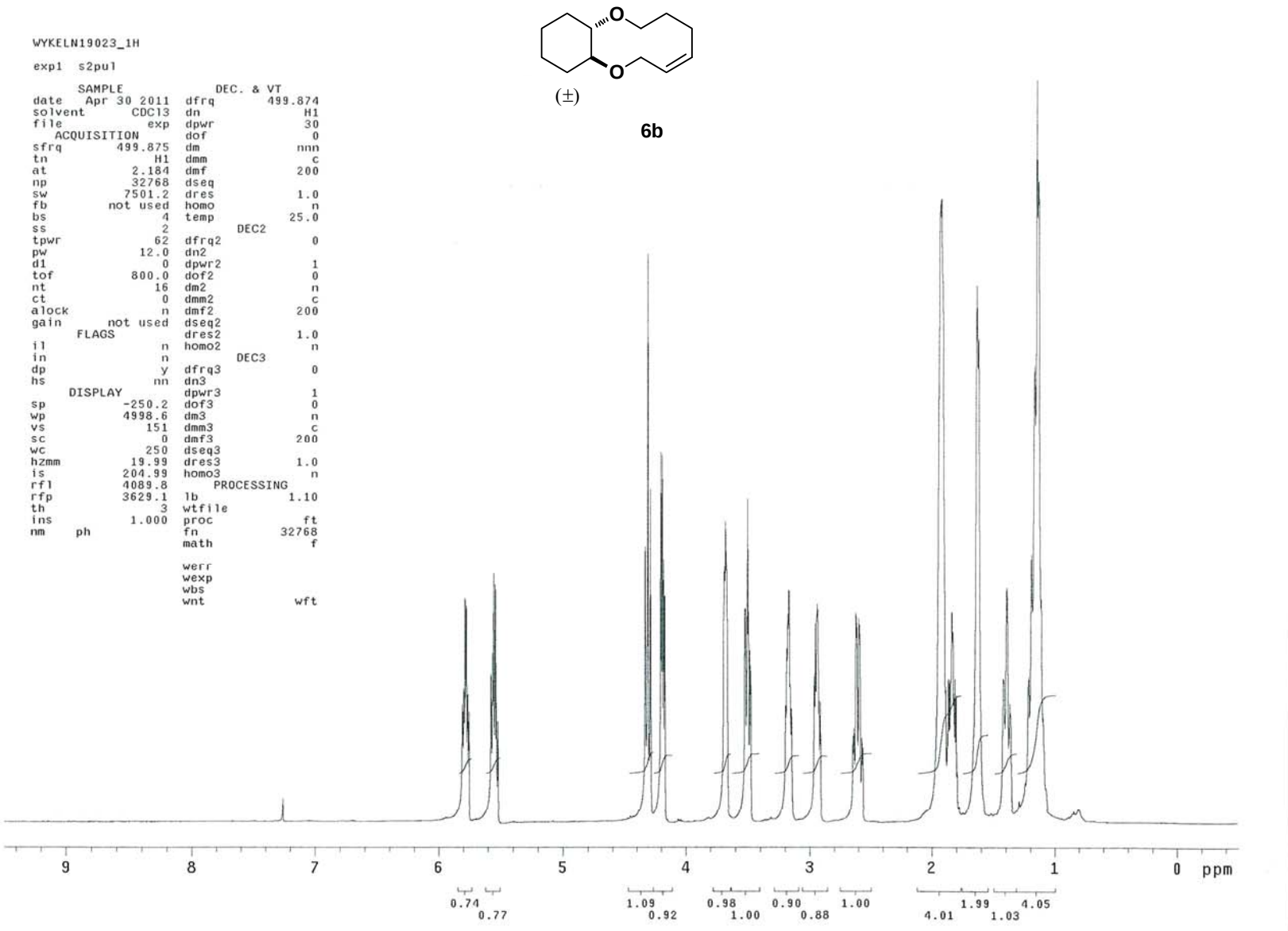
expl s2pul

SAMPLE		DEC. & VT	
date	Apr 26 2010	dfrq	499.874
solvent	CDCl3	dn	H1
file	exp	dpwr	48
ACQUISITION		dof	0
sfrq	125.707	dm	yyy
tn	C13	dmm	w
at	1.092	dmf	10000
np	65536	dseq	
sw	29996.3	dres	1.0
fb	not used	homo	n
bs	32	temp	25.0
tpwr	55	DEC2	
pw	4.2	dfrq2	0
d1	0	dn2	
tof	2000.0	dpwr2	1
nt	99999	dof2	0
ct	1248	dm2	n
alock	n	dmm2	c
gain	not used	dmf2	10000
FLAGS		dseq2	
il	n	dres2	1.0
in	n	homo2	n
dp	y	DEC3	
hs	nn	dfrq3	0
DISPLAY		dn3	
sp	-1086.9	dpwr3	1
wp	29995.3	dof3	0
vs	35	dm3	n
sc	0	dmm3	c
wc	250	dmf3	10000
hzmm	119.98	dseq3	
is	500.00	dres3	1.0
rfl	10766.2	homo3	n
rfp	9678.3	PROCESSING	
th	3	lb	1.00
ins	100.000	wtfile	
nm cdc ph		proc	ft
		fn	not used
		math	f
		werr	
		wexp	
		wbs	
		wnt	



6

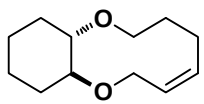



WYKELN19023_1H
exp1 s2pul
SAMPLE
date Apr 30 2011
solvent CDCl3
file exp
ACQUISITION
sfrq 499.875
tn H1
at 2.184
np 32768
sw 7501.2
fb not used
bs 4
ss 2
tpwr 62
pw 12.0
d1 0
tof 800.0
nt 16
ct 0
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -250.2
wp 4998.6
vs 151
sc 0
wc 250
hzmm 19.99
is 204.99
rfl 4089.8
rfp 3629.1
th 3
ins 1.000
nm ph
DEC. & VT
dfrq 499.874
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
temp 25.0
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 200
dseq2
dres2 1.0
homo2 n
DEC3
dfrq3 0
dn3
dpwr3 1
dof3 0
dm3 n
dmm3 c
dmf3 200
dseq3
dres3 1.0
homo3 n
PROCESSING
lb 1.10
wtfile
proc ft
fn 32768
math f
werr
wexp
wbs
wnt wft
O
O
(±)
6b
9
8
7
6
5
4
3
2
1
0 ppm
0.74
0.77
1.09
0.92
0.98
1.00
0.90
0.88
1.00
1.99
4.01
4.05
1.03

WYKELN19023_13C

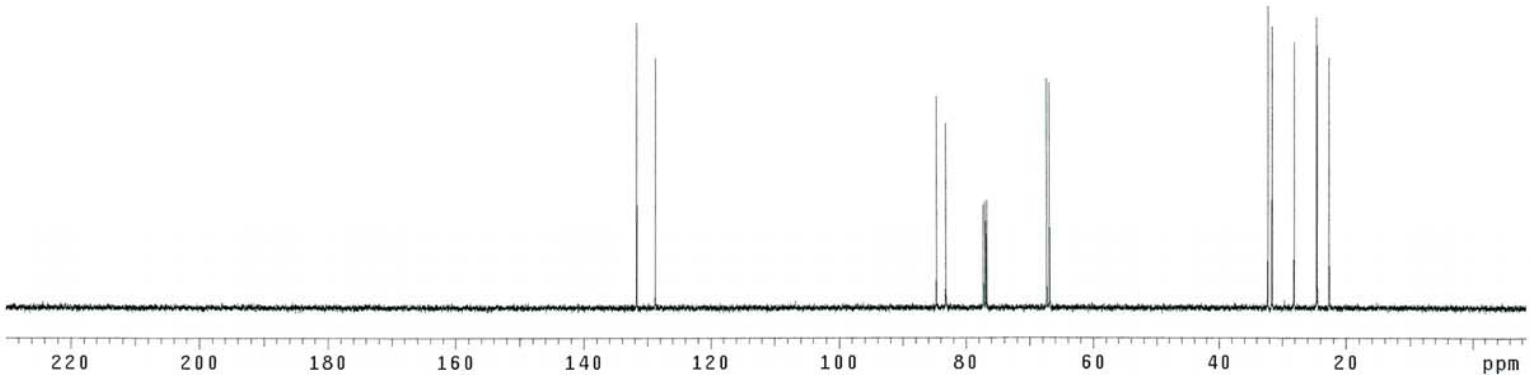
exp2 s2pul

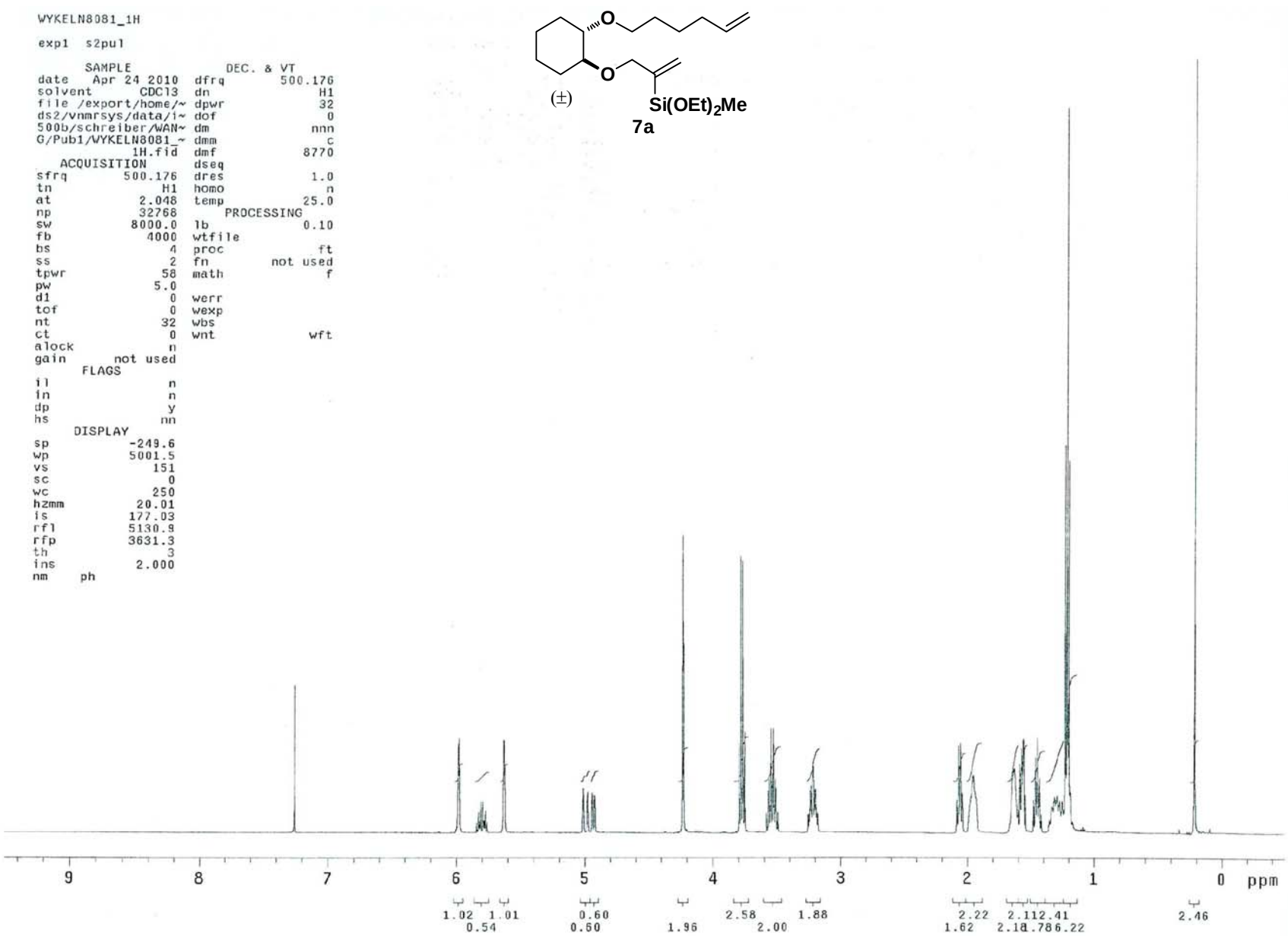
SAMPLE		DEC. & VT	
date	Apr 30 2011	dfrq	499.874
solvent	CDCl3	dn	H1
file	exp	dpwr	48
ACQUISITION		dof	0
sfrq	125.707	dm	yyy
tn	C13	dmm	w
at	1.092	dmf	8929
np	65536	dseq	
sw	29996.3	dres	1.0
fb	not used	homo	n
bs	16	temp	25.0
tpwr	55	DEC2	
pw	4.8	dfrq2	0
d1	0	dn2	
tof	2000.0	dpwr2	1
nt	9999	dof2	0
ct	0	dm2	n
alock	n	dmm2	c
gain	not used	dmf2	10000
FLAGS		dseq2	
il	n	dres2	1.0
in	n	homo2	n
dp	y	DEC3	
hs	nn	dfrq3	0
DISPLAY		dn3	
sp	-1093.3	dpwr3	1
wp	29995.3	dof3	0
vs	50	dm3	n
sc	0	dmm3	c
wc	250	dmf3	10000
hzmm	119.98	dseq3	
is	500.00	dres3	1.0
rfl	10772.6	homo3	n
rfp	9678.3	PROCESSING	
th	6	lb	1.00
ins	100.000	wtfile	
nm cdc ph		proc	ft
		fn	not used
		math	f
		werr	
		wexp	
		wbs	
		wnt	



(±)

6b

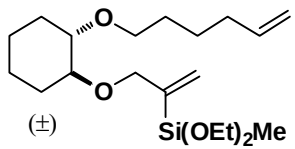



WYKELN8081_1H
exp1 s2pul
SAMPLE
date Apr 24 2010
solvent CDCl3
file /export/home/~
ds2/vnmrsys/data/i~
500b/schreiber/WAN~
G/Pub1/WYKELN8081_~
1H.fid
ACQUISITION
sfrq 500.176
tn H1
at 2.048
np 32768
sw 8000.0
fb 4000
bs 4
ss 2
tpwr 58
pw 5.0
d1 0
tof 0
nt 32
ct 0
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -249.6
wp 5001.5
vs 151
sc 0
wc 250
hzmm 20.01
is 177.03
rfl 5130.9
rfp 3631.3
th 3
ins 2.000
nm ph
DEC. & VT
dfrq 500.176
dn H1
dpwr 32
dof 0
dm nnn
dmm c
dmf 8770
dseq
dres 1.0
homo n
temp 25.0
PROCESSING
lb 0.10
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt wft
O
O
(±)
Si(OEt)2Me
7a
9
8
7
6
5
4
3
2
1
0
ppm
1.02
1.01
0.54
0.60
0.60
1.96
2.58
2.00
1.88
2.22
1.62
2.11
2.18
1.78
6.22
2.46

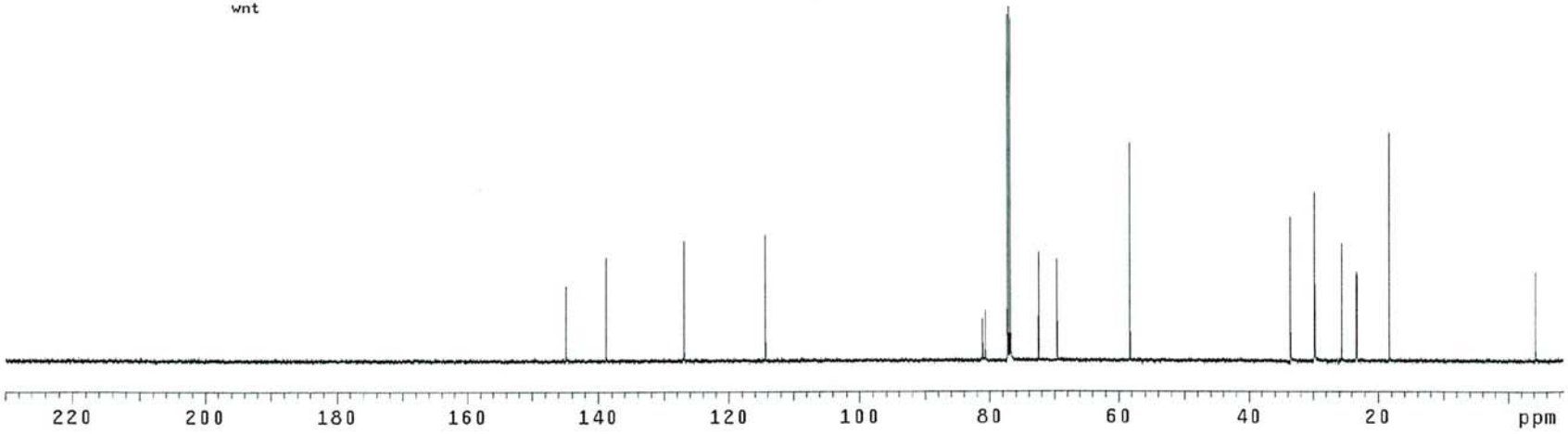
WYKELN8081_13C

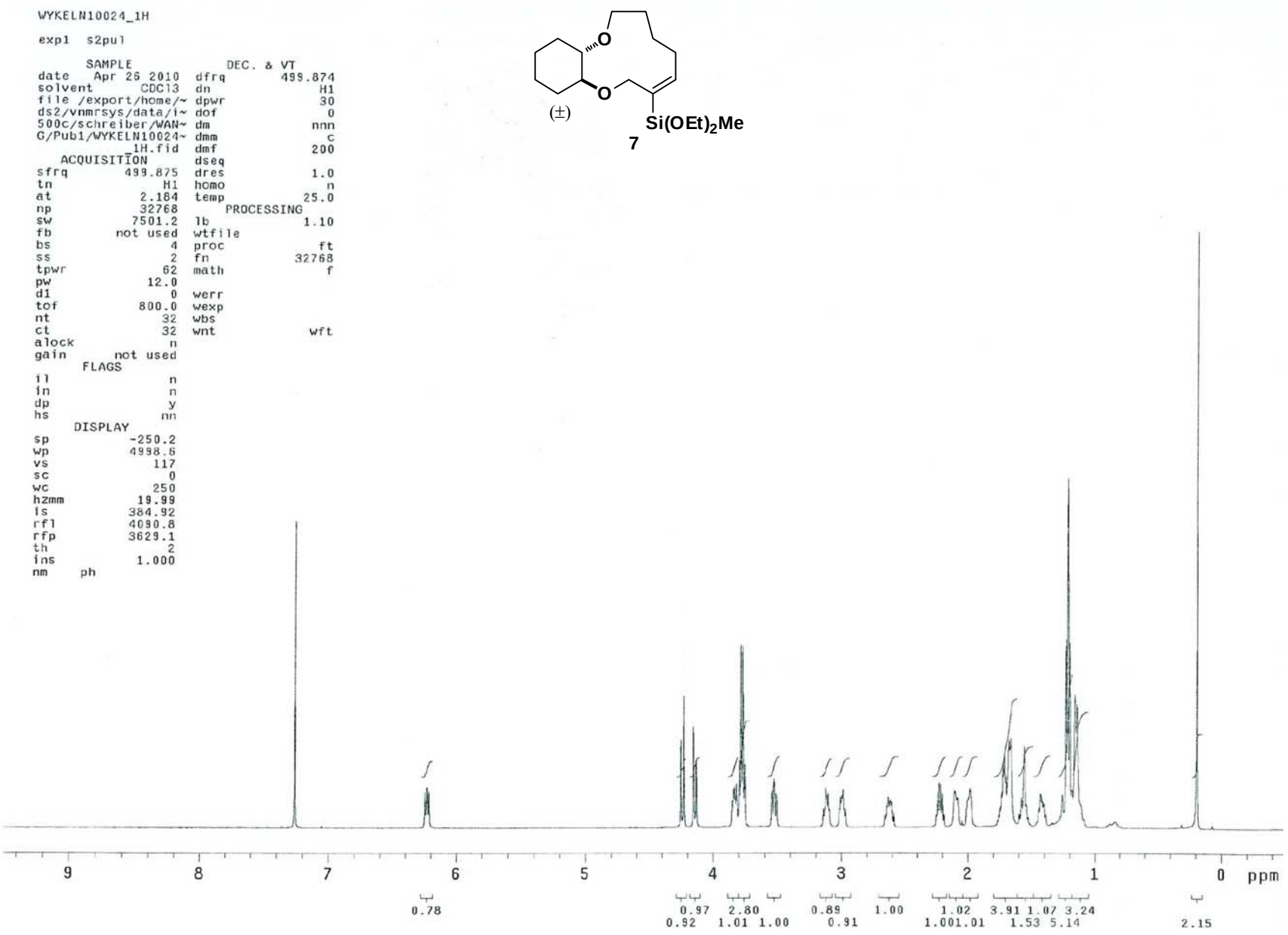
exp2 s2pul

SAMPLE		DEC. & VT	
date	Apr 25 2010	dfrq	499.874
solvent	CDCl3	dn	H1
file	exp	dpwr	48
ACQUISITION		dof	0
sfrq	125.707	dm	yyy
tn	C13	dmm	w
at	1.092	dmf	10000
np	65536	dseq	
sw	29996.3	dres	1.0
fb	not used	homo	n
bs	16	temp	25.0
tpwr	55	DEC2	
pw	2.0	dfrq2	0
d1	0	dn2	
tof	2000.0	dpwr2	1
nt	9999	dof2	0
ct	1520	dm2	n
alock	n	dmm2	c
gain	not used	dmf2	10000
FLAGS		dseq2	
il	n	dres2	1.0
in	n	homo2	n
dp	y	DEC3	
hs	nn	dfrq3	0
DISPLAY		dn3	
sp	-1087.8	dpwr3	1
wp	29995.3	dof3	0
vs	57	dm3	n
sc	0	dmm3	c
wc	250	dmf3	10000
hzmm	119.98	dseq3	
is	500.00	dres3	1.0
rfl	1088.7	homo3	n
rfp	0	PROCESSING	
th	3	lb	1.00
ins	100.000	wtfile	
nm cdc ph		proc	ft
		fn	not used
		math	f
		werr	
		wexp	
		wbs	
		wnt	



7a

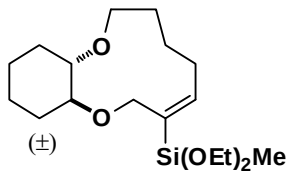



WYKELN10024_1H
exp1 s2pul
SAMPLE
date Apr 26 2010
solvent CDCl3
file /export/home/~
ds2/vnmrsys/data/i~
500c/schreiber/WAN~
G/Pub1/WYKELN10024~
1H.fid
ACQUISITION
sfrq 499.875
tn H1
at 2.184
np 32768
sw 7501.2
fb not used
bs 4
ss 2
tpwr 62
pw 12.0
d1 0
tof 800.0
nt 32
ct 32
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -250.2
wp 4998.6
vs 117
sc 0
wc 250
hzmm 19.99
is 384.92
rfl 4090.8
rfp 3629.1
th 2
ins 1.000
nm ph
DEC. & VT
dfrq 499.874
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
temp 25.0
PROCESSING
lb 1.10
wtfile
proc ft
fn 32768
math f
werr
wexp
wbs
wnt wft
(±)
Si(OEt)2Me
7
9
8
7
6
5
4
3
2
1
0
ppm
0.78
0.92
0.97
1.01
2.80
1.00
0.91
0.89
1.00
1.001.01
1.02
1.53
3.91
1.07
5.14
3.24
2.15

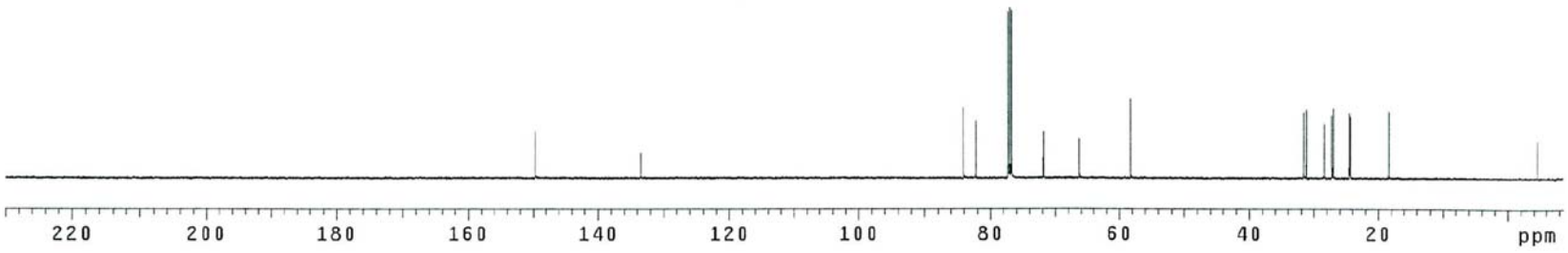
WYKELN10024_13C

exp1 s2pul

SAMPLE		DEC. & VT	
date	Apr 26 2010	dfrq	499.874
solvent	CDCl3	dn	H1
file	exp	dpwr	48
ACQUISITION		dof	0
sfrq	125.707	dm	yyy
tn	C13	dmm	w
at	1.092	dmf	10000
np	65536	dseq	
sw	29996.3	dres	1.0
fb	not used	homo	n
bs	32	temp	25.0
tpwr	55	DEC2	
pw	4.2	dfrq2	0
d1	0	dn2	
tof	2000.0	dpwr2	1
nt	99999	dof2	0
ct	1792	dm2	n
alock	n	dmm2	c
gain	not used	dmf2	10000
FLAGS		dseq2	
il	n	dres2	1.0
in	n	homo2	n
dp	y	DEC3	
hs	nn	dfrq3	0
DISPLAY		dn3	
sp	-1086.9	dpwr3	1
wp	29995.3	dof3	0
vs	27	dm3	n
sc	0	dmm3	c
wc	250	dmf3	10000
hzmm	119.98	dseq3	
is	500.00	dres3	1.0
rfl	10766.2	homo3	n
rfp	9678.3	PROCESSING	
th	2	lb	1.00
ins	100.000	wtfile	
nm cdc ph		proc	ft
		fn	not used
		math	f
		werr	
		wexp	
		wbs	
		wnt	



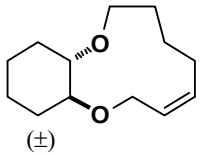
7



WYKELN19024_1H

exp3 s2pul

SAMPLE		DEC. & VT	
date	Apr 30 2011	dfrq	499.874
solvent	CDCl3	dn	H1
file	/export/home/~	dpwr	30
ds2/vnmrsys/data/i~		dof	0
500c/schreiber/WAN~		dm	nnn
G/Pub1/WYKELN19024~		dmm	c
	_1H.fid	dmf	200
ACQUISITION		dseq	
sfrq	499.875	dres	1.0
tn	H1	homo	n
at	2.184	temp	25.0
np	32768	PROCESSING	
sw	7501.2	lb	1.10
fb	not used	wtfile	
bs	4	proc	ft
ss	2	fn	32768
tpwr	62	math	f
pw	12.0		
d1	0	werr	
tof	800.0	wexp	
nt	16	wbs	
ct	0	wnt	wft
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-250.2		
wp	4998.6		
vs	57		
sc	0		
wc	250		
hzmm	19.99		
is	198.78		
rfl	460.8		
rfp	0		
th	4		
ins	1.000		
nm ph			



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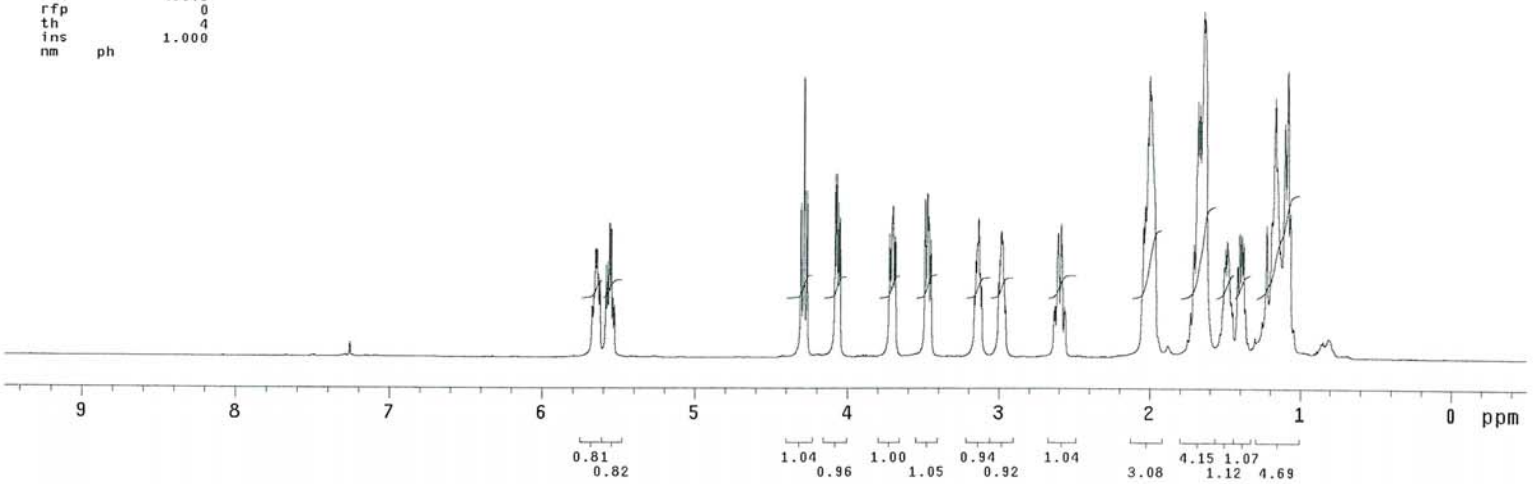
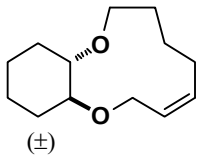
7b

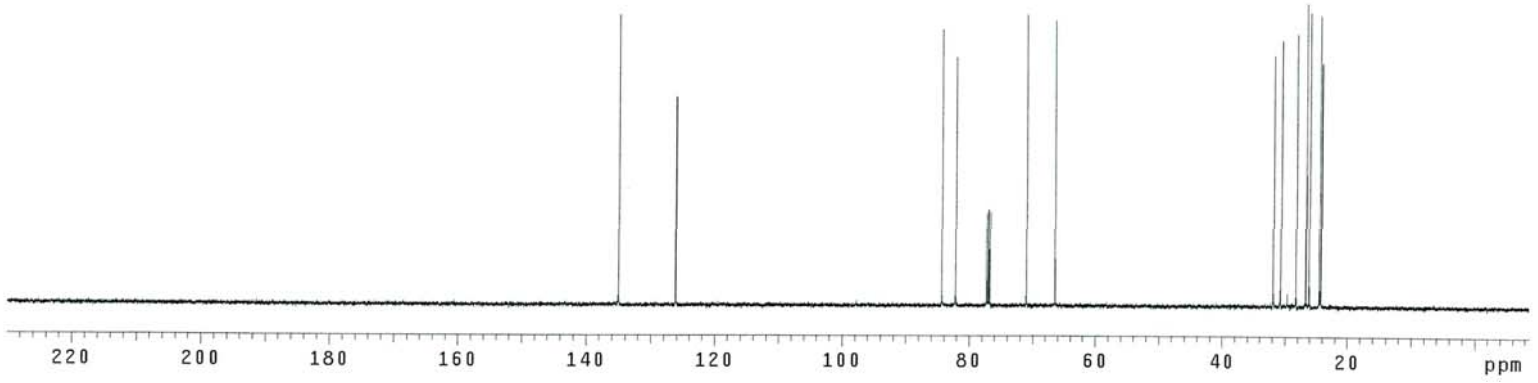


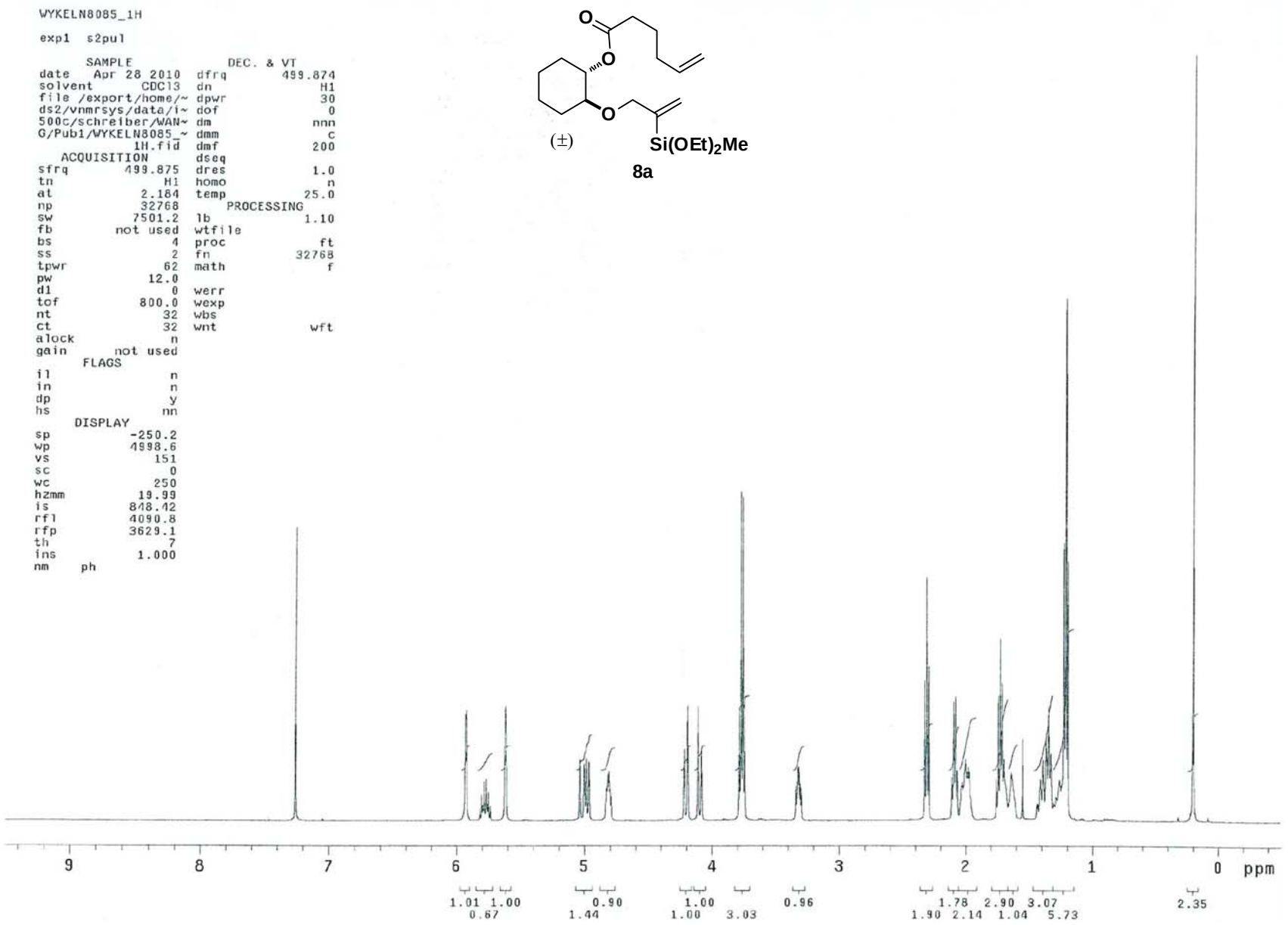
```
WYKELN19024_13C

exp3  s2pul

      SAMPLE
date    Apr 30 2011
solvent       CDCl3
file /export/home/~
ds2/vnmrsys/data/i~
500c/schreiber/WAN~
G/Pub1/WYKELN19024~
          _13C.fid
   ACQUISITION
sfrq        125.707
tn              C13
at              1.092
np              65536
sw          29996.3
fb         not used
bs                16
tpwr              55
pw               4.8
d1                 0
tof           2000.0
nt              9999
ct                 0
alock              n
gain       not used
      FLAGS
il                 n
in                 n
dp                 y
hs                nn
     DISPLAY
sp          -1092.4
wp          29995.3
vs                50
sc                 0
wc               250
hzmm          119.98
is            500.00
rfl          10771.7
rfp           9678.3
th                 9
ins          100.000
nm  cdc  ph

      DEC. & VT
dfrq        499.874
dn               H1
dpwr             48
dof               0
dm              yyy
dmm               w
dmf            8929
dseq
dres            1.0
homo              n
temp           25.0
    PROCESSING
lb              1.00
wtfile
proc              ft
fn         not used
math               f
werr
wexp
wbs
wnt
```



(±)

7b

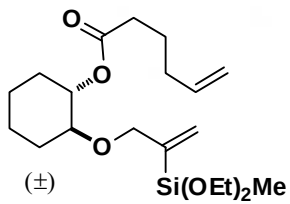



WYKELN8085_1H
exp1 s2pul
SAMPLE
date Apr 28 2010
solvent CDCl3
file /export/home/~
ds2/vnmrsys/data/i~
500c/schreiber/WAN~
G/Pub1/WYKELN8085_~
1H.fid
ACQUISITION
sfrq 499.875
tn H1
at 2.184
np 32768
sw 7501.2
fb not used
bs 4
ss 2
tpwr 62
pw 12.0
d1 0
tof 800.0
nt 32
ct 32
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -250.2
wp 4998.6
vs 151
sc 0
wc 250
hzmm 19.99
is 848.42
rfl 4090.8
rfp 3629.1
th 7
ins 1.000
nm ph
DEC. & VT
dfrq 499.874
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
temp 25.0
PROCESSING
lb 1.10
wtfile
proc ft
fn 32768
math f
werr
wexp
wbs
wnt wft
(±)
Si(OEt)2Me
8a
9
8
7
6
5
4
3
2
1
0 ppm
1.01 1.00
0.67
0.90
1.44
1.00
3.03
0.96
1.78 2.90 3.07
1.90 2.14 1.04 5.73
2.35

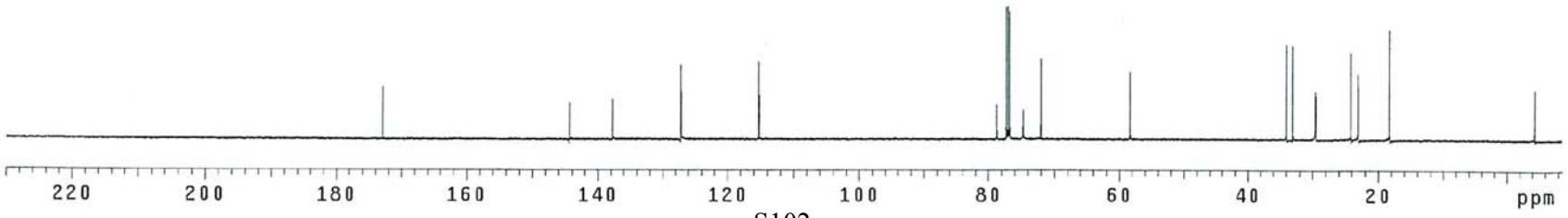
WYKELN8085_13C

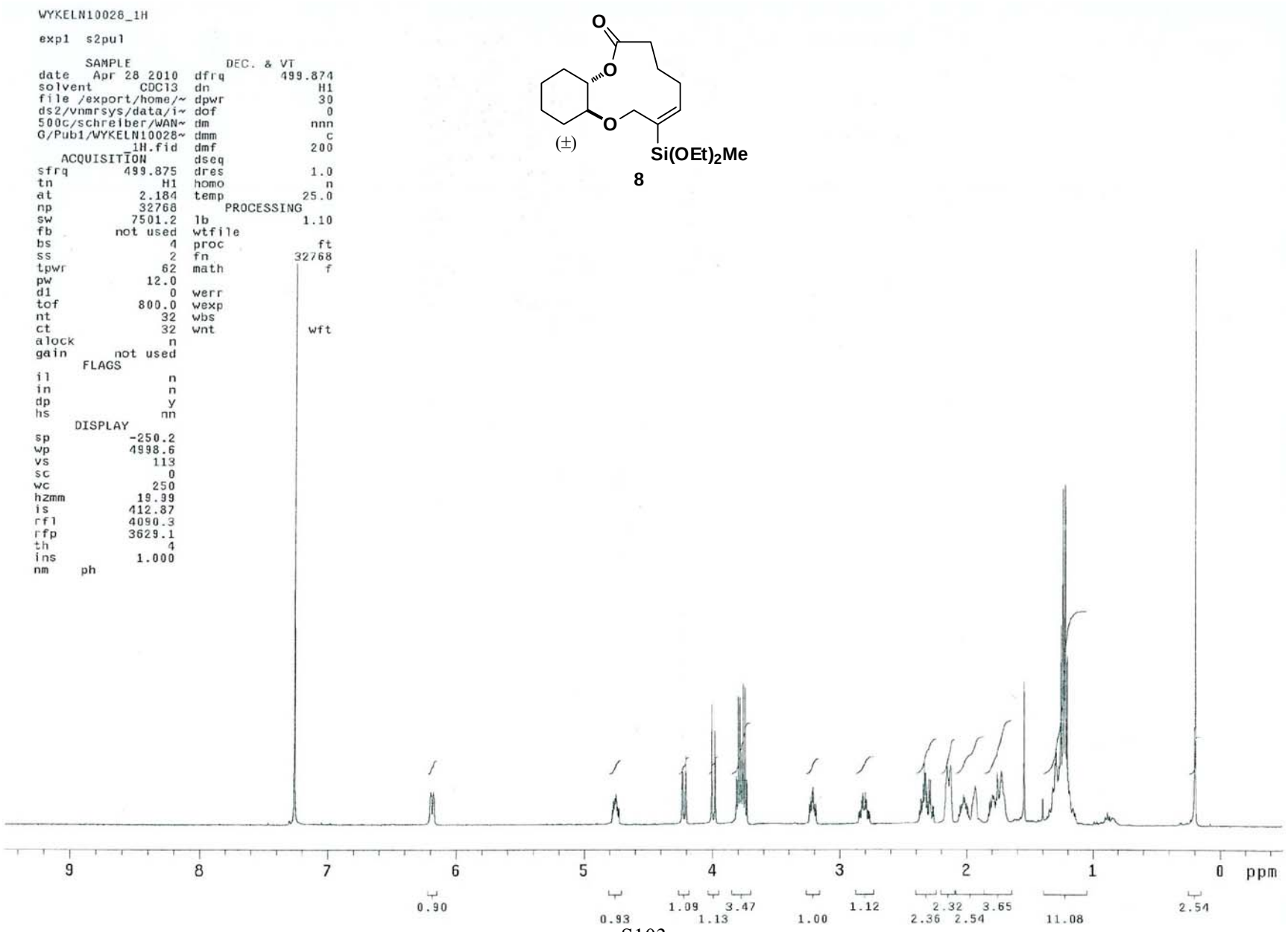
exp3 s2pul

SAMPLE		DEC. & VT	
date	Apr 28 2010	dfrq	499.874
solvent	CDCl3	dn	H1
file	exp	dpwr	48
ACQUISITION		dof	0
sfrq	125.707	dm	yyy
tn	C13	dmm	w
at	1.092	dmf	10000
np	65536	dseq	
sw	29996.3	dres	1.0
fb	not used	homo	n
bs	32	temp	25.0
tpwr	55	DEC2	
pw	4.2	dfrq2	0
d1	0	dn2	
tof	2000.0	dpwr2	1
nt	99999	dof2	0
ct	1024	dm2	n
alock	n	dmm2	c
gain	not used	dmf2	10000
FLAGS		dseq2	
il	n	dres2	1.0
in	n	homo2	n
dp	y	DEC3	
hs	nn	dfrq3	0
DISPLAY		dn3	
sp	-1087.8	dpwr3	1
wp	29995.3	dof3	0
vs	21	dm3	n
sc	0	dmm3	c
wc	250	dmf3	10000
hzmm	119.98	dseq3	
is	500.00	dres3	1.0
rfl	10767.1	homo3	n
rfp	9678.3	PROCESSING	
th	3	lb	1.00
ins	100.000	wtfile	
nm cdc ph		proc	ft
		fn	not used
		math	f
		werr	
		wexp	
		wbs	
		wnt	



8a

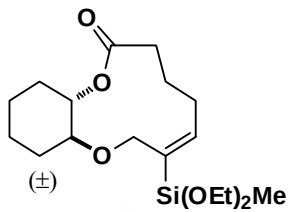



WYKELN10028_1H
expl s2pul
SAMPLE
date Apr 28 2010
solvent CDCl3
file /export/home/~
ds2/vnmrsys/data/i~
500c/schreiber/WAN~
G/Pub1/WYKELN10028~
1H.fid
ACQUISITION
sfrq 499.875
tn H1
at 2.184
np 32768
sw 7501.2
fb not used
bs 4
ss 2
tpwr 62
pw 12.0
d1 0
tof 800.0
nt 32
ct 32
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -250.2
wp 4998.6
vs 113
sc 0
wc 250
hzmm 19.99
is 412.87
rfl 4090.3
rfp 3629.1
th 4
ins 1.000
nm ph
DEC. & VT
dfrq 499.874
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
temp 25.0
PROCESSING
lb 1.10
wtfile
proc ft
fn 32768
math f
werr
wexp
wbs
wnt wft
O
O
O
(±)
Si(OEt)2Me
8
9
8
7
6
5
4
3
2
1
0
ppm
0.90
0.93
1.09
1.13
3.47
1.00
1.12
2.36
2.32
2.54
3.65
11.08
2.54

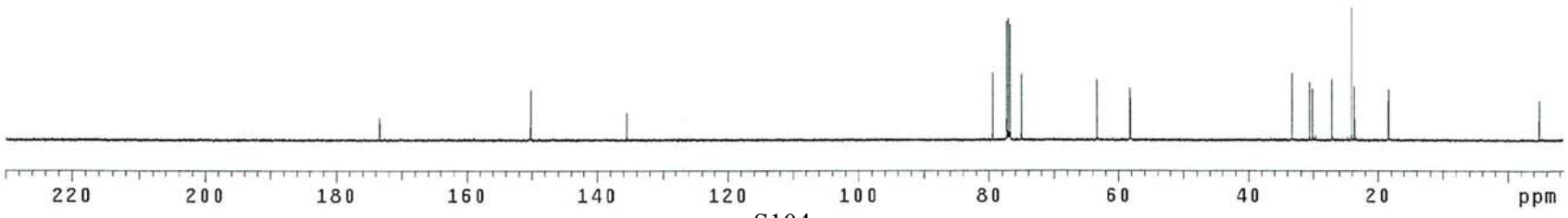
WYKELN10028_13C

exp3 s2pul

SAMPLE		DEC. & VT	
date	Apr 28 2010	dfrq	499.874
solvent	CDCl3	dn	H1
file	exp	dpwr	48
ACQUISITION		dof	0
sfrq	125.707	dm	yyy
tn	C13	dmm	w
at	1.092	dmf	10000
np	65536	dseq	
sw	29996.3	dres	1.0
fb	not used	homo	n
bs	32	temp	25.0
tpwr	55	DEC2	
pw	4.2	dfrq2	0
d1	0	dn2	
tof	2000.0	dpwr2	1
nt	99999	dof2	0
ct	1088	dm2	n
alock	n	dmm2	c
gain	not used	dmf2	10000
FLAGS		dseq2	
il	n	dres2	1.0
in	n	homo2	n
dp	y	DEC3	
hs	nn	dfrq3	0
DISPLAY		dn3	
sp	-1087.8	dpwr3	1
wp	29995.3	dof3	0
vs	21	dm3	n
sc	0	dmm3	c
wc	250	dmf3	10000
hzmm	119.98	dseq3	
is	500.00	dres3	1.0
rfl	10767.1	homo3	n
rfp	9678.3	PROCESSING	
th	5	lb	1.00
ins	100.000	wtfile	
nm cdc ph		proc	ft
		fn	not used
		math	f
		werr	
		wexp	
		wbs	
		wnt	



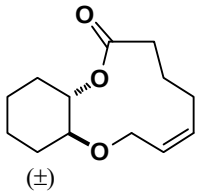
8



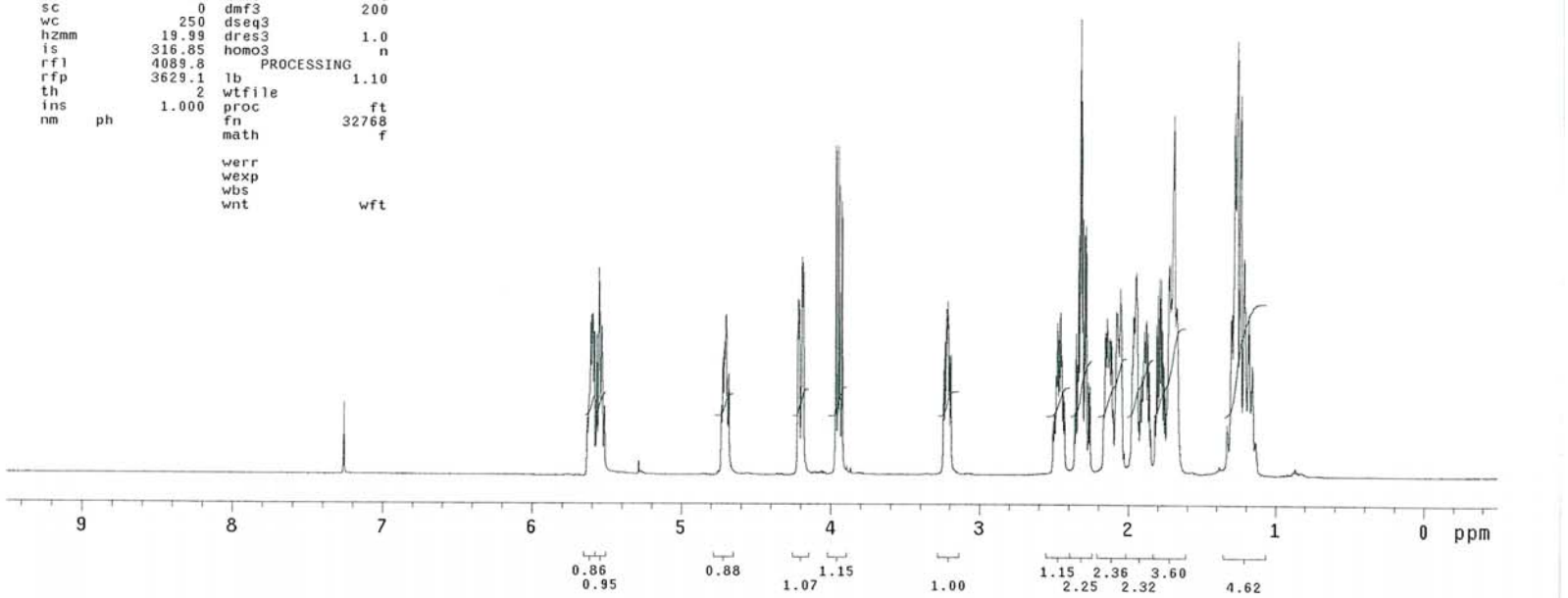
WYKELN19028_1H

exp1 s2pul

SAMPLE		DEC. & VT	
date	Apr 30 2011	dfrq	499.874
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	499.875	dm	nnn
tn	H1	dmm	c
at	2.184	dmf	200
np	32768	dseq	
sw	7501.2	dres	1.0
fb	not used	homo	n
bs	4	temp	25.0
ss	2	DEC2	
tpwr	62	dfrq2	0
pw	12.0	dn2	
d1	0	dpwr2	1
tof	800.0	dof2	0
nt	16	dm2	n
ct	0	dmm2	c
alock	n	dmf2	200
gain	not used	dseq2	
FLAGS		dres2	1.0
il	n	homo2	n
in	n	DEC3	
dp	y	dfrq3	0
hs	nn	dn3	
DISPLAY		dpwr3	1
sp	-250.2	dof3	0
wp	4998.6	dm3	n
vs	77	dmm3	c
sc	0	dmf3	200
wc	250	dseq3	
hzmm	19.99	dres3	1.0
is	316.85	homo3	n
rfl	4089.8	PROCESSING	
rfp	3629.1	lb	1.10
th	2	wtfile	
ins	1.000	proc	ft
nm ph		fn	32768
		math	f
		werr	
		wexp	
		wbs	
		wnt	wft



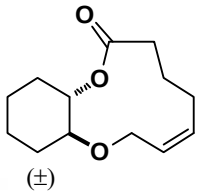
8b



WYKELN19028_13C

exp2 s2pul

SAMPLE		DEC. & VT	
date	Apr 30 2011	dfrq	499.874
solvent	CDCl3	dn	H1
file	exp	dpwr	48
ACQUISITION		dof	0
sfrq	125.707	dm	yyy
tn	C13	dmm	w
at	1.092	dmf	8929
np	65536	dseq	
sw	29996.3	dres	1.0
fb	not used	homo	n
bs	16	temp	25.0
tpwr	55	DEC2	
pw	4.8	dfrq2	0
d1	0	dn2	
tof	2000.0	dpwr2	1
nt	9999	dof2	0
ct	0	dm2	n
alock	n	dmm2	c
gain	not used	dmf2	10000
FLAGS		dseq2	
il	n	dres2	1.0
in	n	homo2	n
dp	y	DEC3	
hs	nn	dfrq3	0
DISPLAY		dn3	
sp	-1094.2	dpwr3	1
wp	29995.3	dof3	0
vs	30	dm3	n
sc	0	dmm3	c
wc	250	dmf3	10000
hzmm	119.98	dseq3	
is	500.00	dres3	1.0
rfl	10773.5	homo3	n
rfp	9678.3	PROCESSING	
th	6	lb	1.00
ins	100.000	wtfile	
nm cdc ph		proc	ft
		fn	not used
		math	f
		werr	
		wexp	
		wbs	
		wnt	



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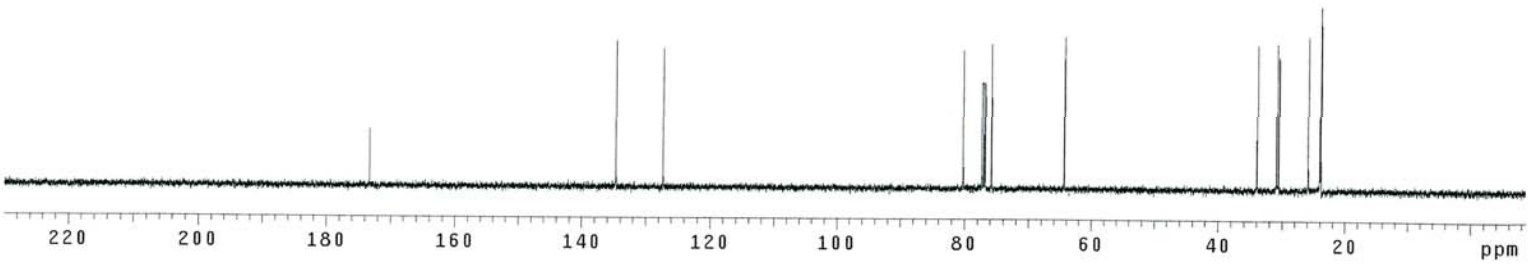
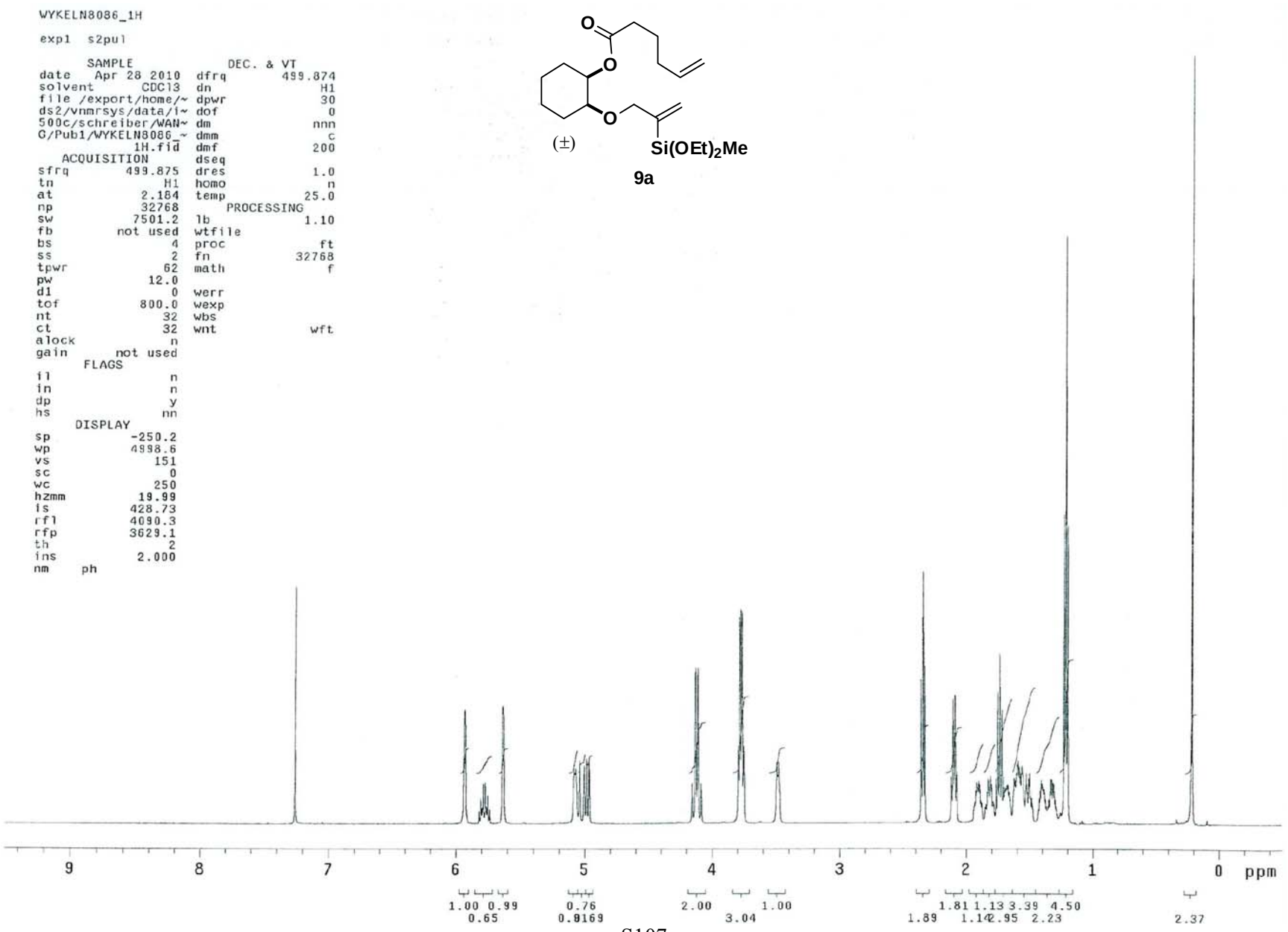
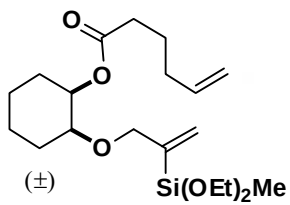



WYKELN8086_1H
expl s2pul
SAMPLE
date Apr 28 2010
solvent CDCl3
file /export/home/~
ds2/vnmrsys/data/i~
500c/schreiber/WAN~
G/Pub1/WYKELN8086_~
1H.fid
ACQUISITION
sfrq 499.875
tn H1
at 2.184
np 32768
sw 7501.2
fb not used
bs 4
ss 2
tpwr 62
pw 12.0
d1 0
tof 800.0
nt 32
ct 32
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -250.2
wp 4998.6
vs 151
sc 0
wc 250
hzmm 19.99
is 428.73
rfl 4090.3
rfp 3629.1
th 2
ins 2.000
nm ph
DEC. & VT
dfrq 499.874
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
temp 25.0
PROCESSING
lb 1.10
wtfile
proc ft
fn 32768
math f
werr
wexp
wbs
wnt wft
(±)
Si(OEt)2Me
9a
9
8
7
6
5
4
3
2
1
0
ppm
1.00 0.99
0.65
0.76
0.8169
2.00
3.04
1.00
1.89
1.81 1.13 3.39 4.50
1.14 2.95 2.23
2.37

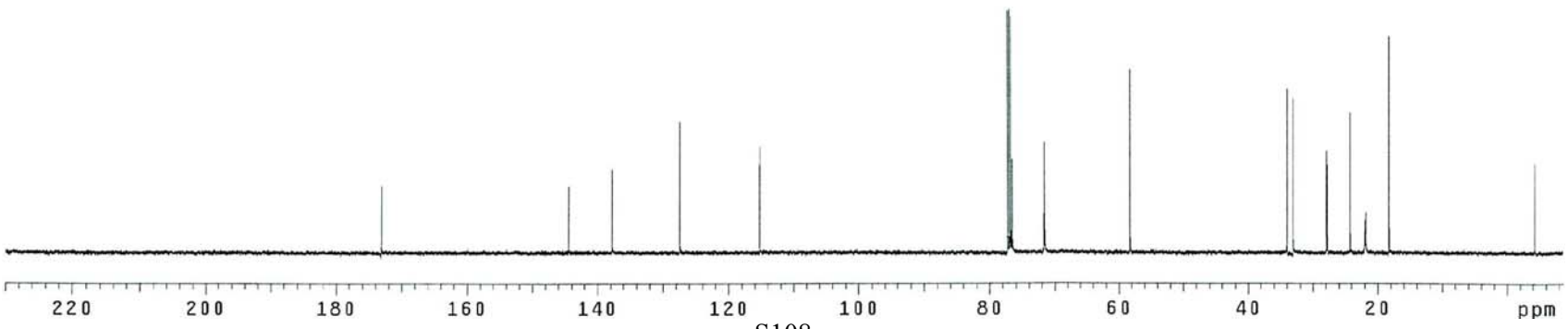
WYKELN8086_13C

exp3 s2pul

SAMPLE		DEC. & VT	
date	Apr 28 2010	dfrq	499.874
solvent	CDCl3	dn	H1
file	exp	dpwr	48
ACQUISITION		dof	0
sfrq	125.707	dm	yyy
tn	C13	dmm	w
at	1.092	dmf	10000
np	65536	dseq	
sw	29996.3	dres	1.0
fb	not used	homo	n
bs	32	temp	25.0
tpwr	55	DEC2	
pw	4.2	dfrq2	0
d1	0	dn2	
tof	2000.0	dpwr2	1
nt	99999	dof2	0
ct	896	dm2	n
alock	n	dmm2	c
gain	not used	dmf2	10000
FLAGS		dseq2	
il	n	dres2	1.0
in	n	homo2	n
dp	y	DEC3	
hs	nn	dfrq3	0
DISPLAY		dn3	
sp	-1087.8	dpwr3	1
wp	29995.3	dof3	0
vs	39	dm3	n
sc	0	dmm3	c
wc	250	dmf3	10000
hzmm	119.98	dseq3	
is	500.00	dres3	1.0
rfl	1088.7	homo3	n
rfp	0	PROCESSING	
th	4	lb	1.00
ins	100.000	wtfile	
nm cdc ph		proc	ft
		fn	not used
		math	f
		werr	
		wexp	
		wbs	
		wnt	



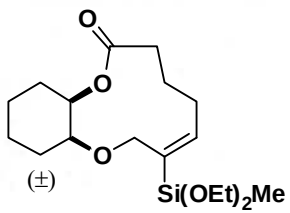
9a



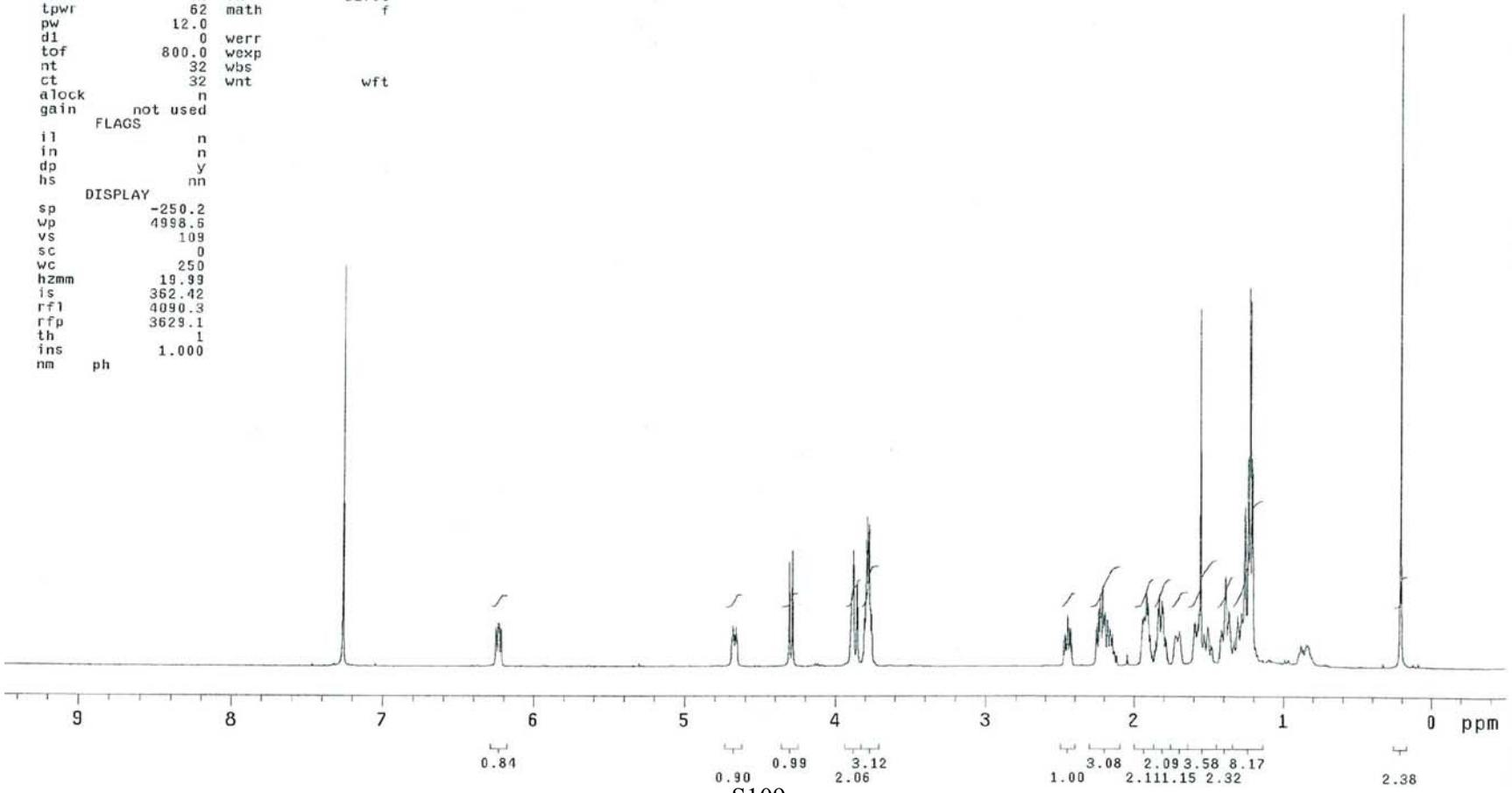
WYKELN10029_1H

exp1 s2pul

SAMPLE		DEC. & VT	
date	Apr 28 2010	dfrq	499.874
solvent	CDCl3	dn	H1
file	/export/home/~	dpwr	30
	ds2/vnmrsys/data/i~	dof	0
	500c/schreiber/WAN~	dm	nnn
	G/Pub1/WYKELN10029~	dmm	c
	_1H.fid	dmf	200
ACQUISITION		dseq	
sfrq	499.875	dres	1.0
tn	H1	homo	n
at	2.184	temp	25.0
np	32768	PROCESSING	
sw	7501.2	lb	1.10
fb	not used	wtfile	
bs	4	proc	ft
ss	2	fn	32768
tpwr	62	math	f
pw	12.0		
d1	0	werr	
tof	800.0	wexp	
nt	32	wbs	
ct	32	wnt	wft
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-250.2		
wp	4998.6		
vs	109		
sc	0		
wc	250		
hzmm	19.99		
is	362.42		
rfl	4090.3		
rfp	3629.1		
th	1		
ins	1.000		
nm	ph		



9

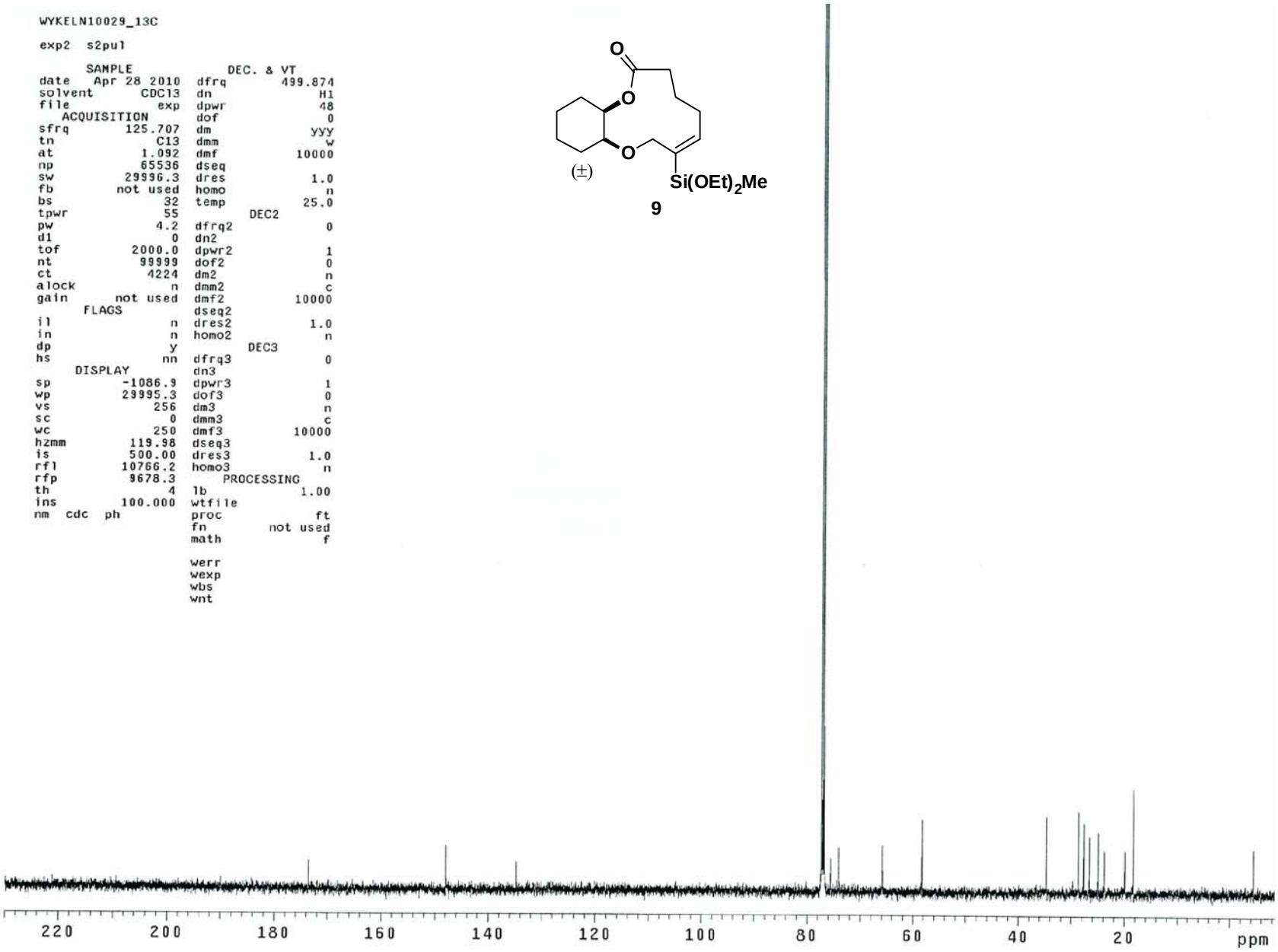


WYKELN10029_13C

exp2 s2pul

SAMPLE		DEC. & VT	
date	Apr 28 2010	dfrq	499.874
solvent	CDCl3	dn	H1
file	exp	dpwr	48
ACQUISITION		dof	0
sfrq	125.707	dm	yyy
tn	C13	dmm	w
at	1.092	dmf	10000
np	65536	dseq	
sw	29996.3	dres	1.0
fb	not used	homo	n
bs	32	temp	25.0
tpwr	55	DEC2	
pw	4.2	dfrq2	0
d1	0	dn2	
tof	2000.0	dpwr2	1
nt	99999	dof2	0
ct	4224	dm2	n
alock	n	dmm2	c
gain	not used	dmf2	10000
FLAGS		dseq2	
il	n	dres2	1.0
in	n	homo2	n
dp	y	DEC3	
hs	nn	dfrq3	0
DISPLAY		dn3	
sp	-1086.9	dpwr3	1
wp	29995.3	dof3	0
vs	256	dm3	n
sc	0	dmm3	c
wc	250	dmf3	10000
hzmm	119.98	dseq3	
is	500.00	dres3	1.0
rfl	10766.2	homo3	n
rfp	9678.3	PROCESSING	
th	4	lb	1.00
ins	100.000	wtfile	
nm cdc ph		proc	ft
		fn	not used
		math	f
		werr	
		wexp	
		wbs	
		wnt	

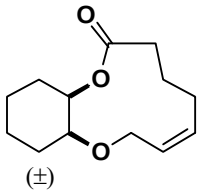
9



WYKELN19029_1H

exp1 s2pul

SAMPLE		DEC. & VT	
date	Apr 30 2011	dfrq	499.874
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	499.875	dm	nnn
tn	H1	dmm	c
at	2.184	dmf	200
np	32768	dseq	
sw	7501.2	dres	1.0
fb	not used	homo	n
bs	4	temp	25.0
ss	2	DEC2	
tpwr	62	dfrq2	0
pw	12.0	dn2	
d1	0	dpwr2	1
tof	800.0	dof2	0
nt	16	dm2	n
ct	16	dmm2	c
alock	n	dmf2	200
gain	not used	dseq2	
FLAGS		dres2	1.0
il	n	homo2	n
in	n	DEC3	
dp	y	dfrq3	0
hs	nn	dn3	
DISPLAY		dpwr3	1
sp	-250.2	dof3	0
wp	4998.6	dm3	n
vs	76	dmm3	c
sc	0	dmf3	200
wc	250	dseq3	
hzmm	19.99	dres3	1.0
is	307.76	homo3	n
rfl	4089.4	PROCESSING	
rfp	3629.1	lb	1.10
th	7	wtfile	
ins	1.000	proc	ft
nm ph		fn	32768
		math	f
		werr	
		wexp	
		wbs	
		wnt	wft



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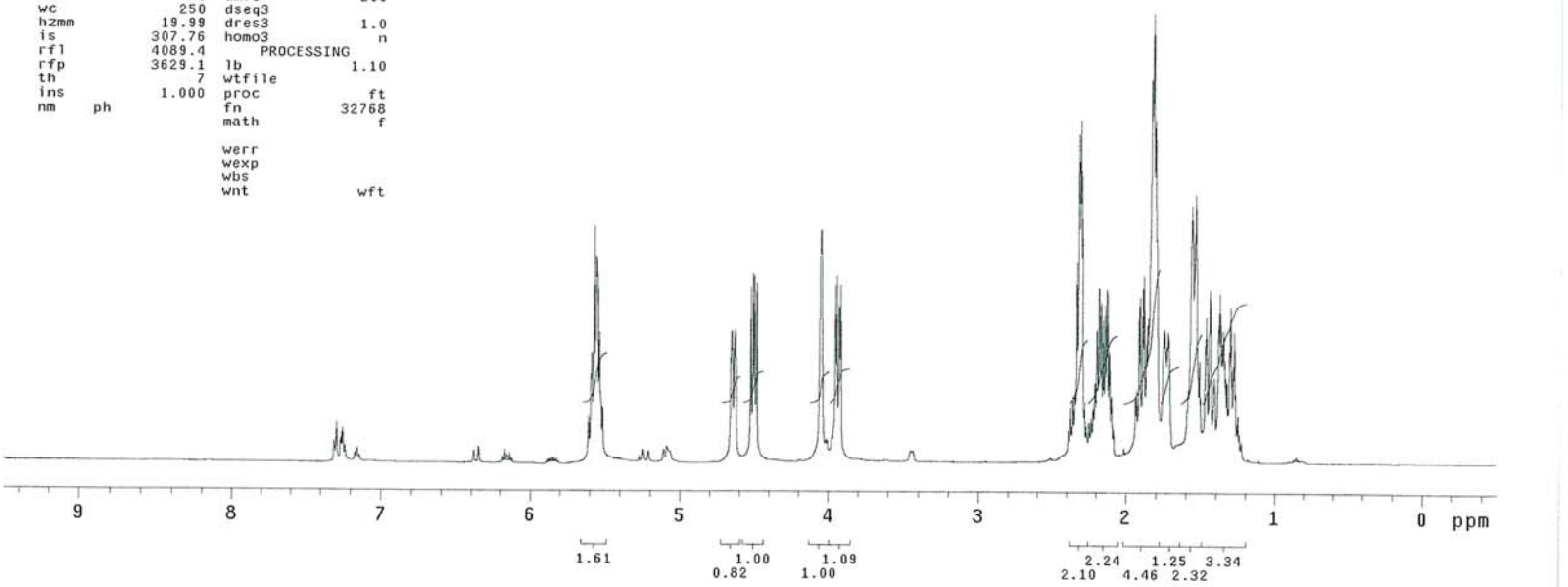
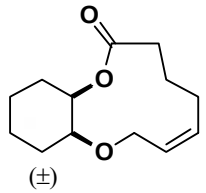


WYKELN19029_13C

exp2 s2pul

SAMPLE		DEC. & VT	
date	Apr 30 2011	dfrq	499.874
solvent	CDCl3	dn	H1
file	exp	dpwr	48
ACQUISITION		dof	0
sfrq	125.707	dm	yyy
tn	C13	dmm	w
at	1.092	dmf	8929
np	65536	dseq	
sw	29996.3	dres	1.0
fb	not used	homo	n
bs	16	temp	25.0
tpwr	55	DEC2	
pw	4.8	dfrq2	0
d1	0	dn2	
tof	2000.0	dpwr2	1
nt	9999	dof2	0
ct	400	dm2	n
alock	n	dmm2	c
gain	not used	dmf2	10000
FLAGS		dseq2	
il	n	dres2	1.0
in	n	homo2	n
dp	y	DEC3	
hs	nn	dfrq3	0
DISPLAY		dn3	
sp	-1094.2	dpwr3	1
wp	29995.3	dof3	0
vs	30	dm3	n
sc	0	dmm3	c
wc	250	dmf3	10000
hzmm	119.98	dseq3	
is	500.00	dres3	1.0
rfl	10773.5	homo3	n
rfp	9678.3	PROCESSING	
th	6	lb	1.00
ins	100.000	wtfile	
nm cdc ph		proc	ft
		fn	not used
		math	f
		werr	
		wexp	
		wbs	
		wnt	



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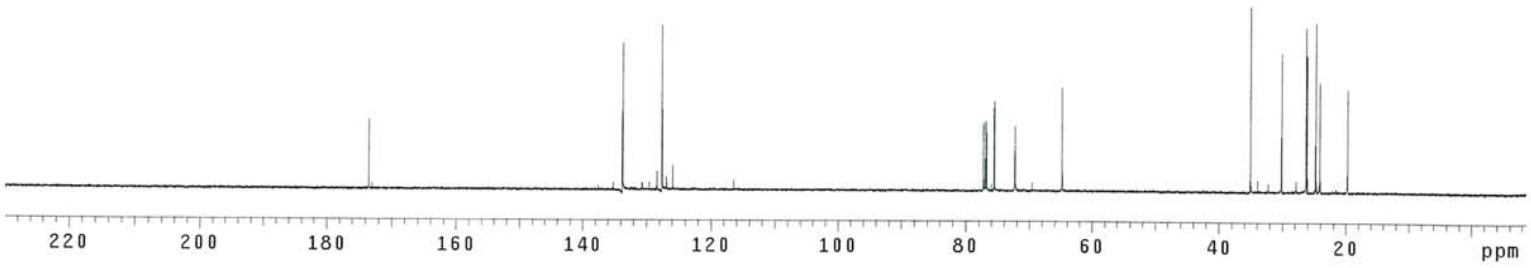
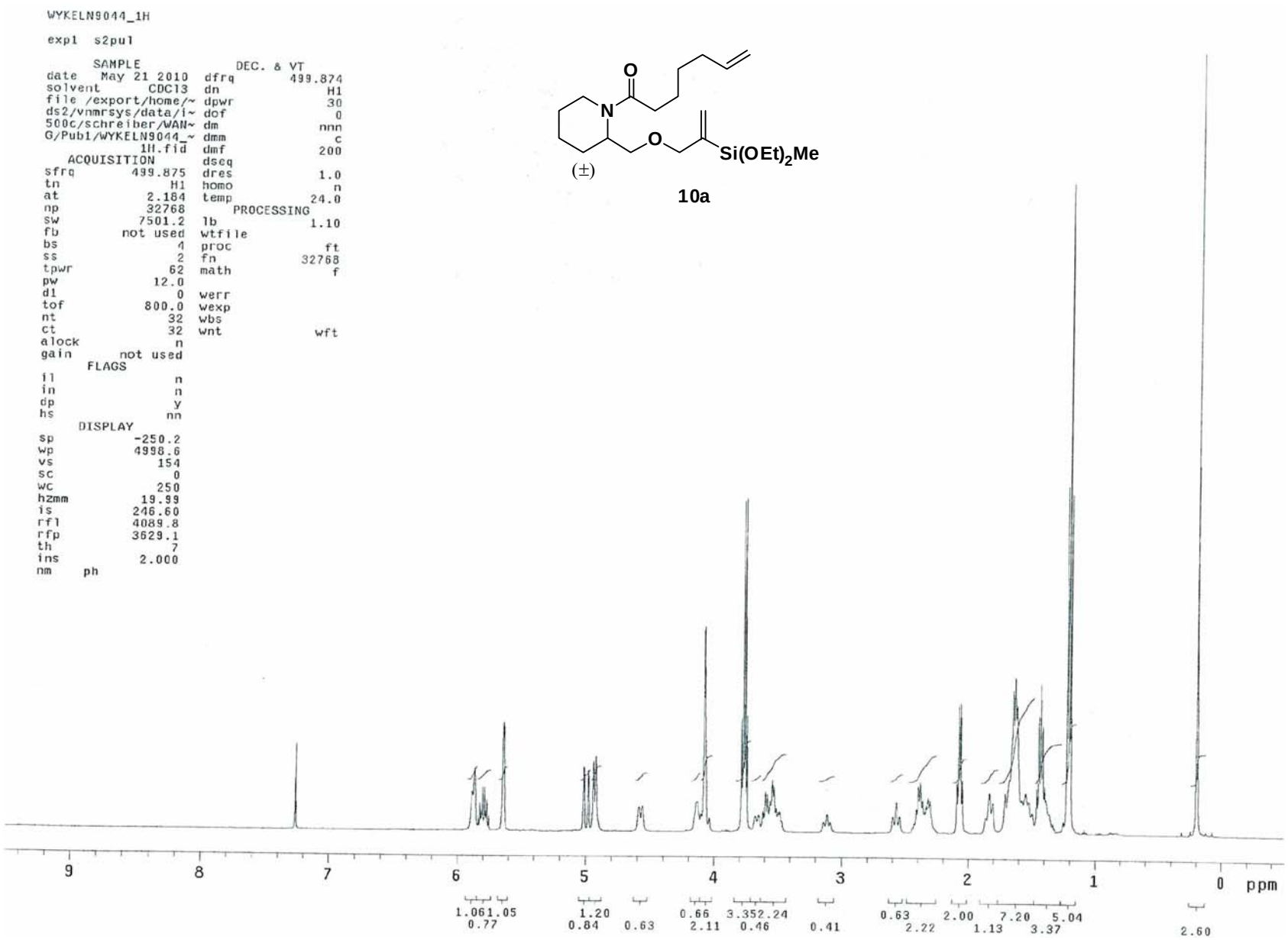
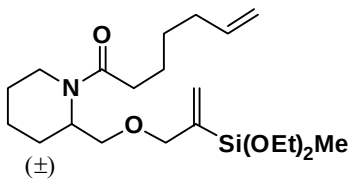



WYKELN9044_1H
expl s2pul
SAMPLE
date May 21 2010
solvent CDCl3
file /export/home/~
ds2/vnmrsys/data/i~
500c/schreiber/WAN~
G/Pub1/WYKELN9044_~
1H.fid
ACQUISITION
sfrq 499.875
tn H1
at 2.184
np 32768
sw 7501.2
fb not used
bs 4
ss 2
tpwr 62
pw 12.0
d1 0
tof 800.0
nt 32
ct 32
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -250.2
wp 4998.6
vs 154
sc 0
wc 250
hzmm 19.99
is 246.60
rfl 4089.8
rfp 3629.1
th 7
ins 2.000
nm ph
DEC. & VT
dfrq 499.874
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
temp 24.0
PROCESSING
lb 1.10
wtfile
proc ft
fn 32768
math f
werr
wexp
wbs
wnt wft
O
N
O
Si(OEt)2Me
(±)
10a
9
8
7
6
5
4
3
2
1
0 ppm
1.06 1.05
0.77
1.20
0.84
0.63
0.66
2.11
3.35 2.24
0.46
0.41
0.63
2.22
2.00
7.20
1.13
5.04
3.37
2.60

WYKELN9044_13C_2

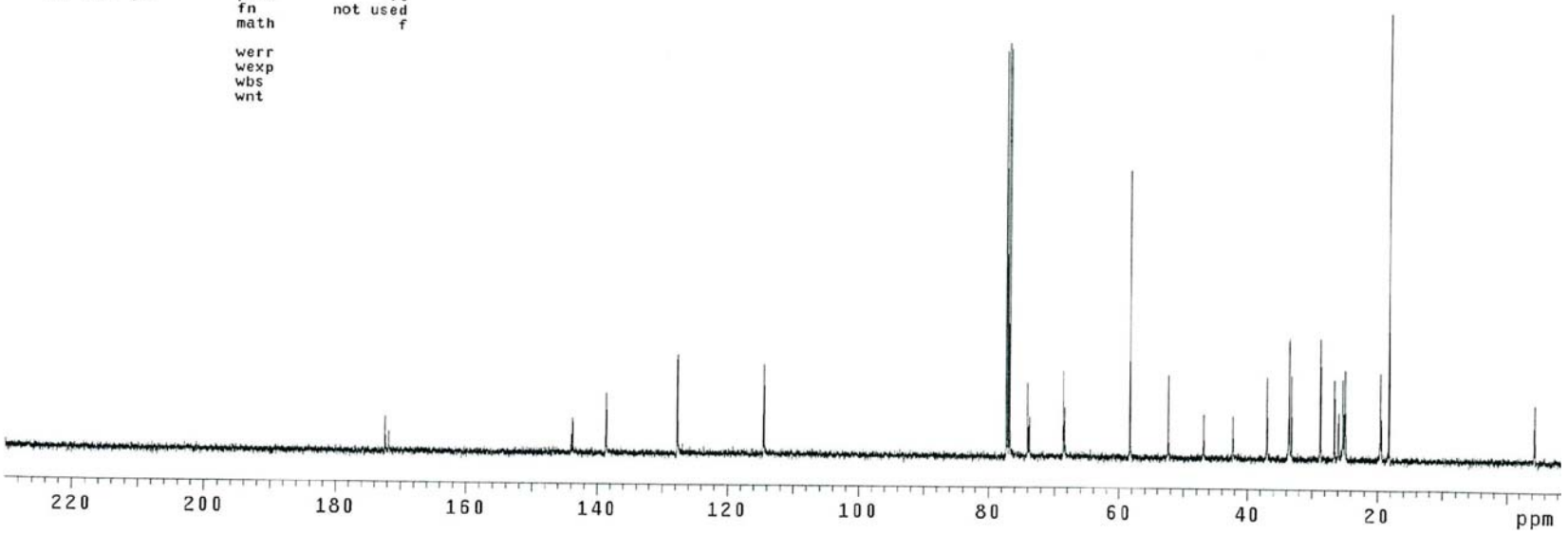
exp1 s2pul

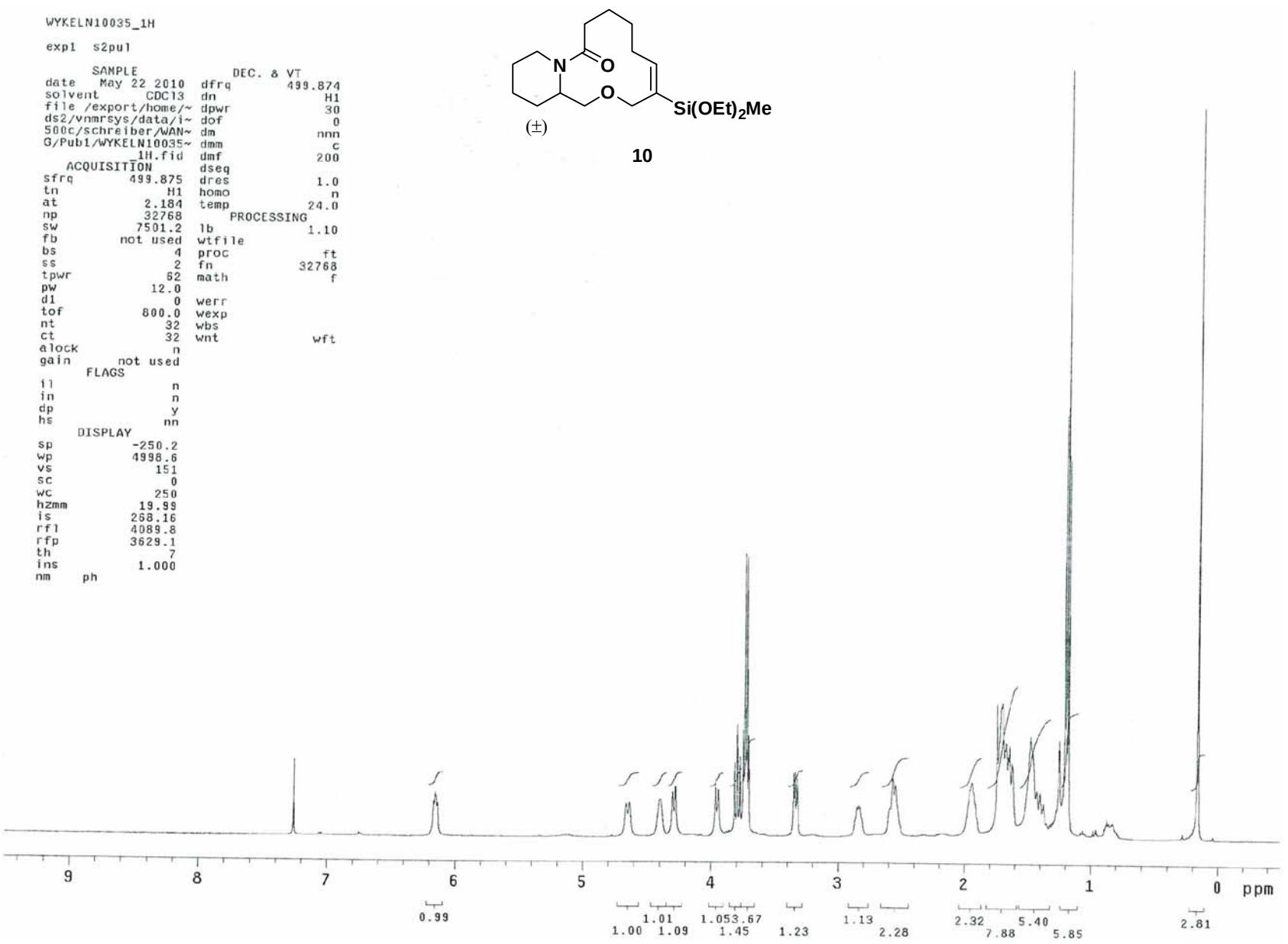
SAMPLE		DEC. & VT	
date	May 21 2010	dfrq	499.874
solvent	CDCl3	dn	H1
file	exp	dpwr	48
ACQUISITION		dof	0
sfrq	125.707	dm	yyy
tn	C13	dmm	w
at	1.092	dmf	10000
np	65536	dseq	
sw	29996.3	dres	1.0
fb	not used	homo	n
bs	32	temp	24.0
tpwr	55	DEC2	
pw	4.2	dfrq2	0
d1	0	dn2	
tof	2000.0	dpwr2	1
nt	99999	dof2	0
ct	1408	dm2	n
alock	n	dmm2	c
gain	not used	dmf2	10000
FLAGS		dseq2	
il	n	dres2	1.0
in	n	homo2	n
dp	y	DEC3	
hs	nn	dfrq3	0
DISPLAY		dn3	
sp	-1091.5	dpwr3	1
wp	29995.3	dof3	0
vs	72	dm3	n
sc	0	dmm3	c
wc	250	dmf3	10000
hzmm	119.98	dseq3	
is	500.00	dres3	1.0
rfl	10770.7	homo3	n
rfp	9678.3	PROCESSING	
th	3	lb	1.00
ins	100.000	wtfile	
nm cdc ph		proc	ft
		fn	not used
		math	f
		werr	
		wexp	
		wbs	
		wnt	



(±)

10a

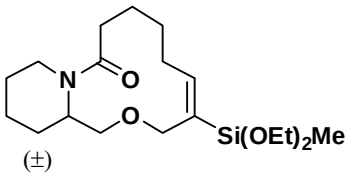



WYKELN10035_1H
exp1 s2pul
SAMPLE
date May 22 2010
solvent CDCl3
file /export/home/~
ds2/vnmrsys/data/i~
500c/schreiber/WAN~
G/Pub1/WYKELN10035~
_1H.fid
ACQUISITION
sfrq 499.875
tn H1
at 2.184
np 32768
sw 7501.2
fb not used
bs 4
ss 2
tpwr 52
pw 12.0
d1 0
tof 800.0
nt 32
ct 32
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -250.2
wp 4998.6
vs 151
sc 0
wc 250
hzmm 19.99
is 268.16
rfl 4089.8
rfp 3629.1
th 7
ins 1.000
nm ph
DEC. & VT
dfrq 499.874
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
temp 24.0
PROCESSING
lb 1.10
wtfile
proc ft
fn 32768
math f
werr
wexp
wbs
wnt wft
N
O
O
Si(OEt)2Me
(±)
10
9
8
7
6
5
4
3
2
1
0 ppm
0.99
1.00
1.01
1.09
1.053.67
1.45
1.23
1.13
2.28
2.32
7.88
5.40
5.85
2.81

WYKELN10035_13C

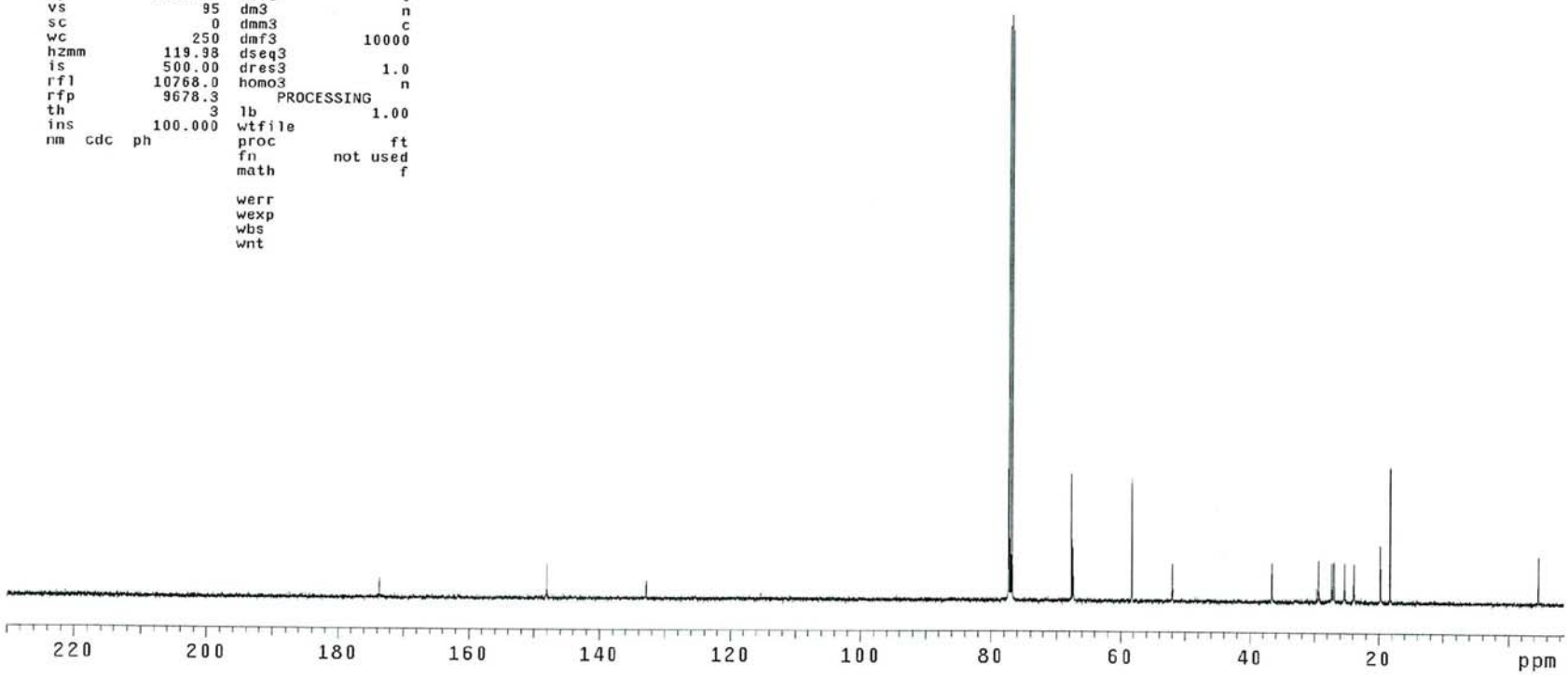
exp3 s2pul

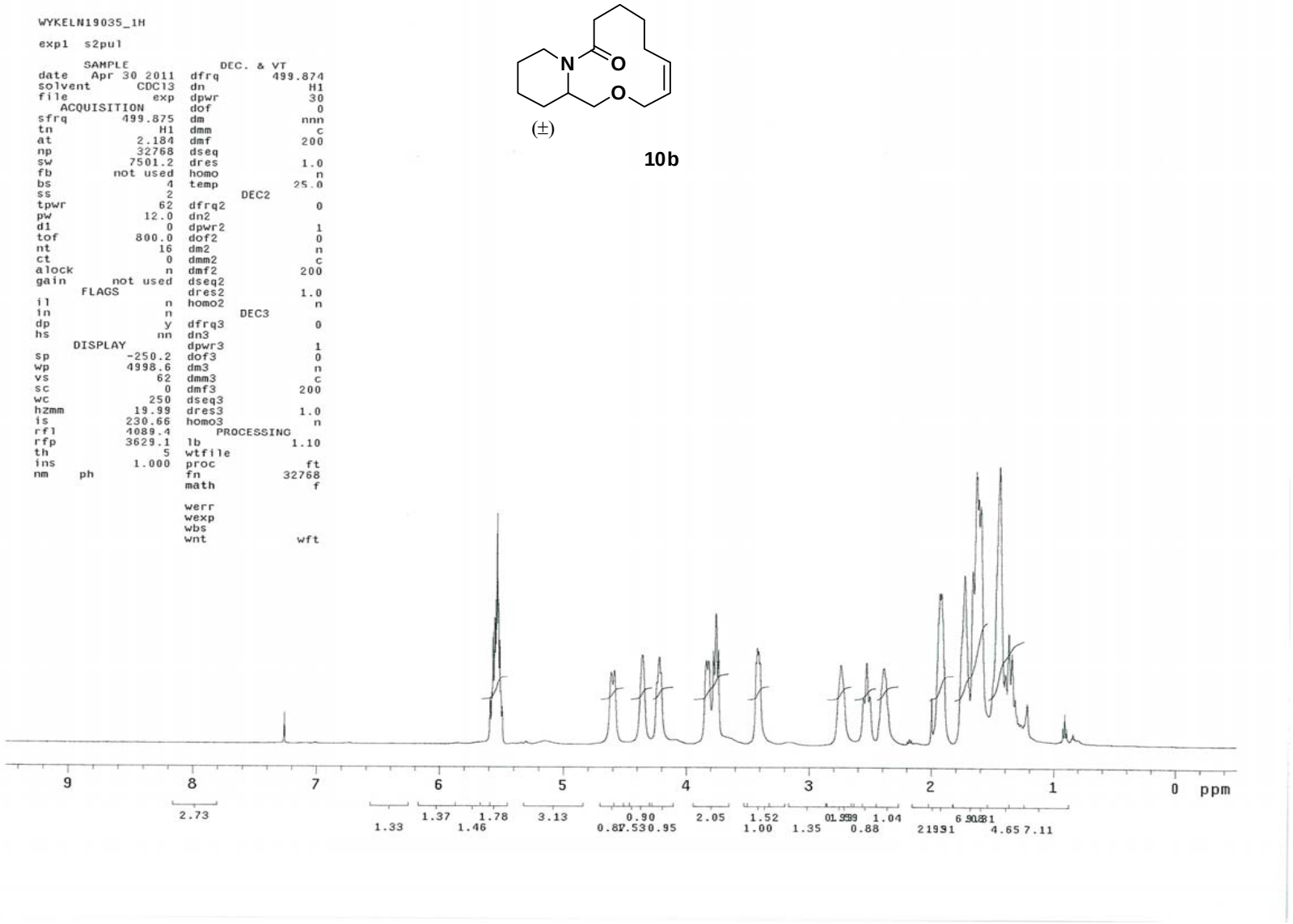
SAMPLE		DEC. & VT	
date	May 22 2010	dfrq	499.874
solvent	CDCl3	dn	H1
file	exp	dpwr	48
ACQUISITION		dof	0
sfrq	125.707	dm	yyy
tn	C13	dmm	w
at	1.092	dmf	10000
np	65536	dseq	
sw	29996.3	dres	1.0
fb	not used	homo	n
bs	32	temp	24.0
tpwr	55	DEC2	
pw	4.2	dfrq2	0
d1	0	dn2	
tof	2000.0	dpwr2	1
nt	99999	dof2	0
ct	3200	dm2	n
alock	n	dmm2	c
gain	not used	dmf2	10000
FLAGS		dseq2	
il	n	dres2	1.0
in	n	homo2	n
dp	y	DEC3	
hs	nn	dfrq3	0
DISPLAY		dn3	
sp	-1088.7	dpwr3	1
wp	29995.3	dof3	0
vs	95	dm3	n
sc	0	dmm3	c
wc	250	dmf3	10000
hzmm	119.98	dseq3	
is	500.00	dres3	1.0
rfl	10768.0	homo3	n
rfp	9678.3	PROCESSING	
th	3	lb	1.00
ins	100.000	wtfile	
nm cdc ph		proc	ft
		fn	not used
		math	f
		werr	
		wexp	
		wbs	
		wnt	



(±)

10




WYKELN19035_1H
exp1 s2pul
SAMPLE
date Apr 30 2011
solvent CDCl3
file exp
ACQUISITION
sfrq 499.875
tn H1
at 2.184
np 32768
sw 7501.2
fb not used
bs 4
ss 2
tpwr 62
pw 12.0
d1 0
tof 800.0
nt 16
ct 0
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -250.2
wp 4998.6
vs 62
sc 0
wc 250
hzmm 19.99
is 230.66
rfl 4089.4
rfp 3629.1
th 5
ins 1.000
nm ph
DEC. & VT
dfrq 499.874
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
temp 25.0
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 200
dseq2
dres2 1.0
homo2 n
DEC3
dfrq3 0
dn3
dpwr3 1
dof3 0
dm3 n
dmm3 c
dmf3 200
dseq3
dres3 1.0
homo3 n
PROCESSING
lb 1.10
wtfile
proc ft
fn 32768
math f
werr
wexp
wbs
wnt wft
N
O
O
9
8
7
6
5
4
3
2
1
0
ppm
2.73
1.33
1.37
1.78
1.46
3.13
0.90
0.87
1.53
0.95
2.05
1.52
1.00
1.35
0.88
1.04
4.65
7.11

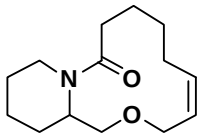
(±)

10b

WYKELN19035_13C

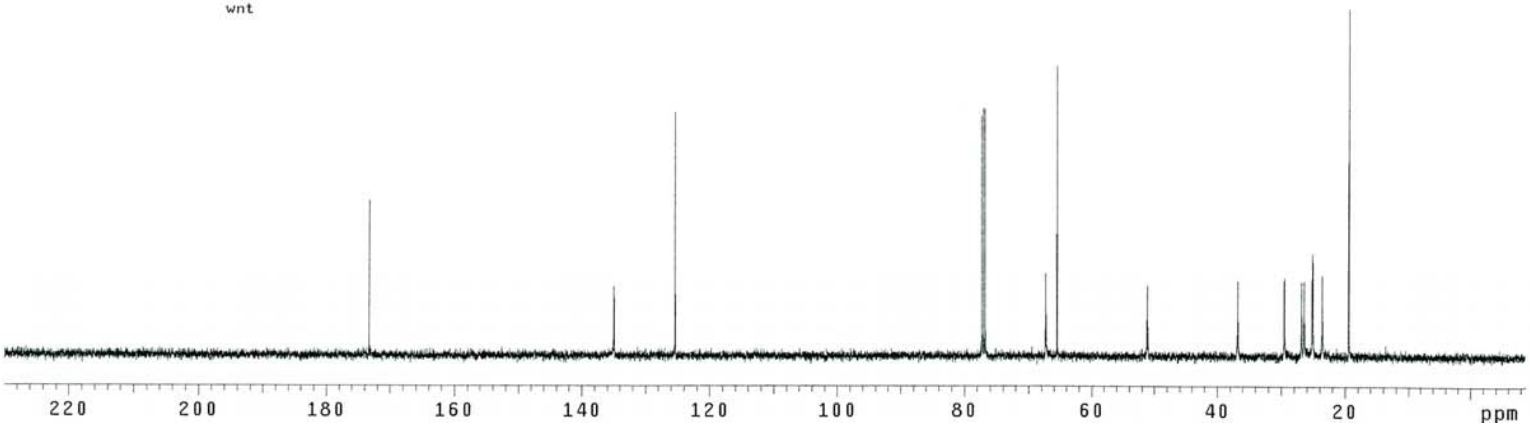
exp2 s2pul

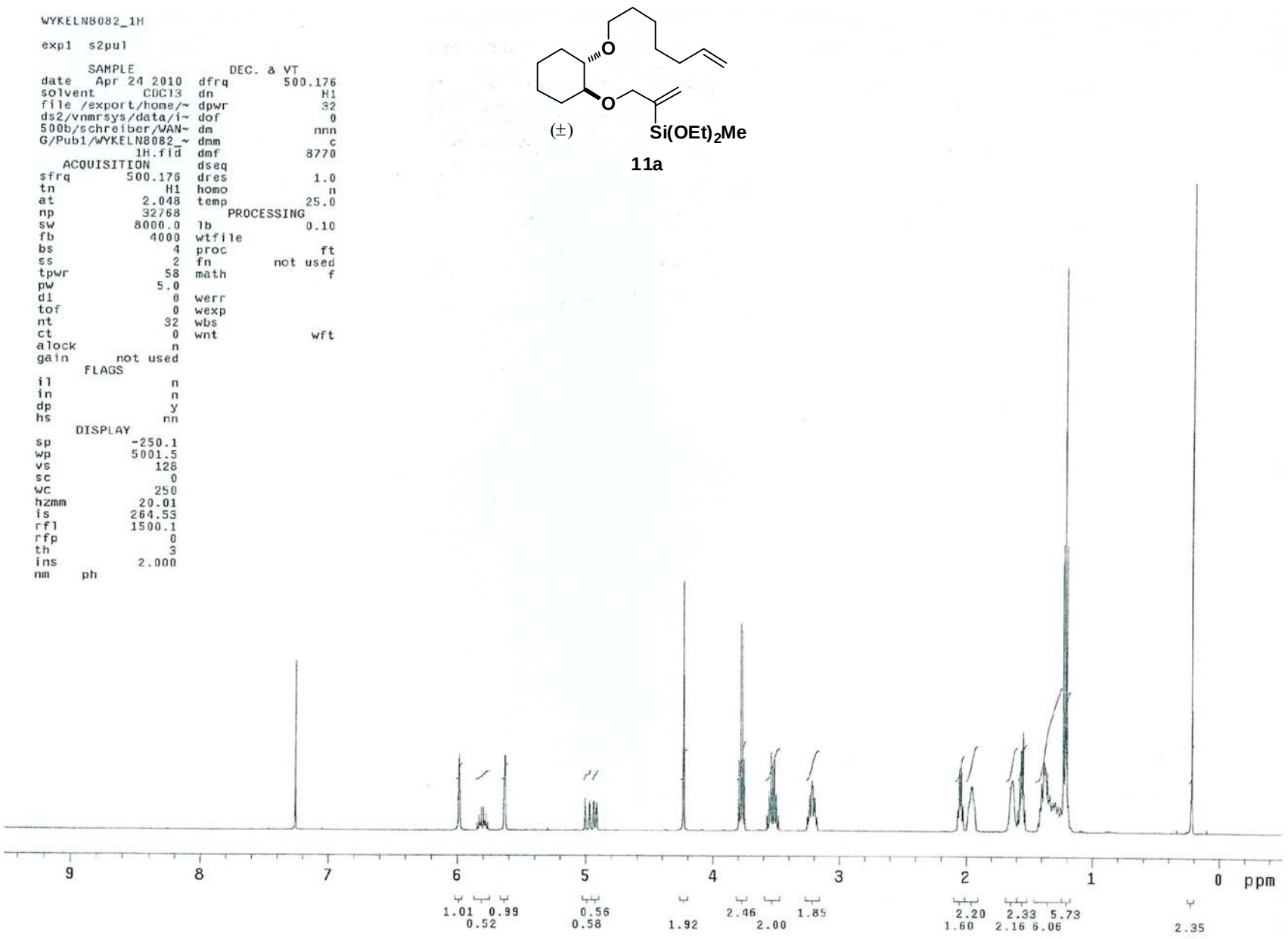
SAMPLE		DEC. & VT	
date	Apr 30 2011	dfrq	499.874
solvent	CDCl3	dn	H1
file	exp	dpwr	48
ACQUISITION		dof	0
sfrq	125.707	dm	yyy
tn	C13	dmm	w
at	1.092	dmf	8929
np	65536	dseq	
sw	29996.3	dres	1.0
fb	not used	homo	n
bs	16	temp	25.0
tpwr	55	DEC2	
pw	4.8	dfrq2	0
d1	0	dn2	
tof	2000.0	dpwr2	1
nt	9999	dof2	0
ct	0	dm2	n
alock	n	dmm2	c
gain	not used	dmf2	10000
FLAGS		dseq2	
il	n	dres2	1.0
in	n	homo2	n
dp	y	DEC3	
hs	nn	dfrq3	0
DISPLAY		dn3	
sp	-1097.0	dpwr3	1
wp	29995.3	dof3	0
vs	57	dm3	n
sc	0	dmm3	c
wc	250	dmf3	10000
hzmm	119.98	dseq3	
is	500.00	dres3	1.0
rfl	10776.2	homo3	n
rfp	9678.3	PROCESSING	
th	5	lb	1.00
ins	100.000	wtfile	
nm cdc ph		proc	ft
		fn	not used
		math	f
		werr	
		wexp	
		wbs	
		wnt	



(±)

10b

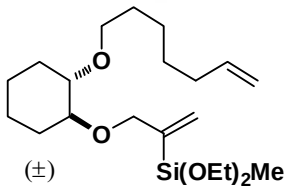



WYKELN8082_1H
exp1 s2pul
SAMPLE
date Apr 24 2010
solvent CDCl3
file /export/home/~
ds2/vnmrsys/data/i~
500b/schreiber/VAN~
G/Pub1/WYKELN8082_~
1H.fid
ACQUISITION
sfrq 500.176
tn H1
at 2.048
np 32768
sw 8000.0
fb 4000
bs 4
ss 2
tpwr 58
pw 5.0
d1 0
tof 0
nt 32
ct 0
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -250.1
wp 5001.5
vs 128
sc 0
wc 250
hzmm 20.01
is 264.53
rfl 1500.1
rfp 0
th 3
ins 2.000
nm ph
DEC. & VT
dfrq 500.176
dn H1
dpwr 32
dof 0
dm nnn
dmm c
dmf 8770
dseq
dres 1.0
homo n
temp 25.0
PROCESSING
lb 0.10
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt wft
O
O
(±)
Si(OEt)2Me
11a
9
8
7
6
5
4
3
2
1
0 ppm
1.01 0.99
0.52
0.56
0.58
1.92
2.46
2.00
1.85
2.20
1.60
2.33 5.73
2.16 6.06
2.35

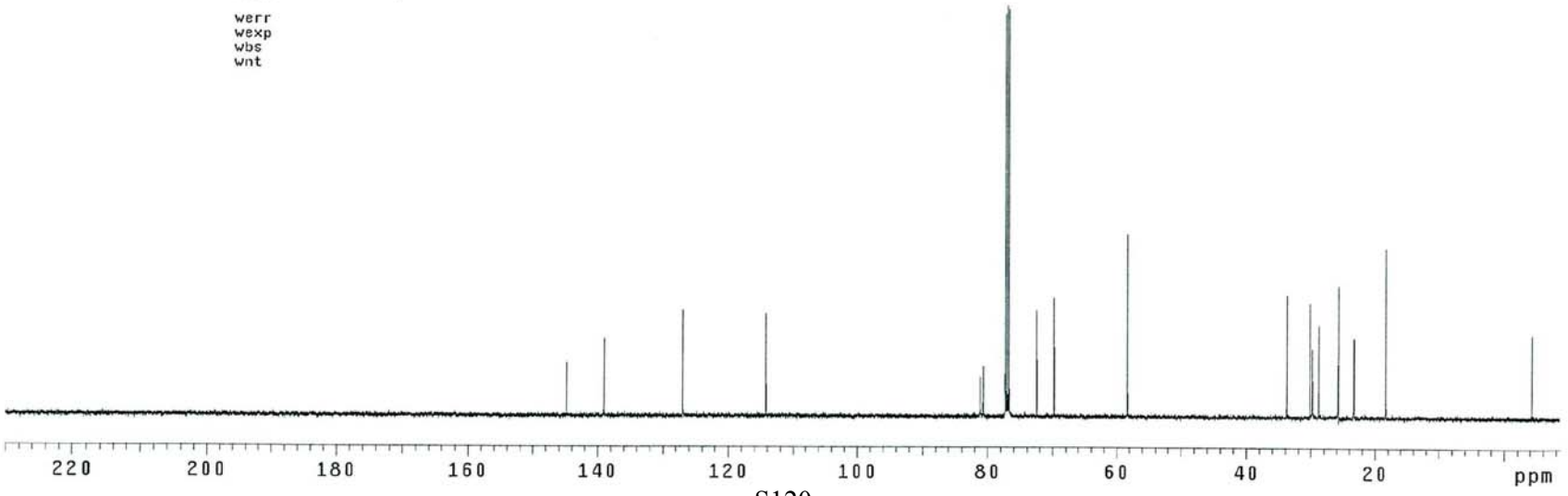
WYKELN8082_13C

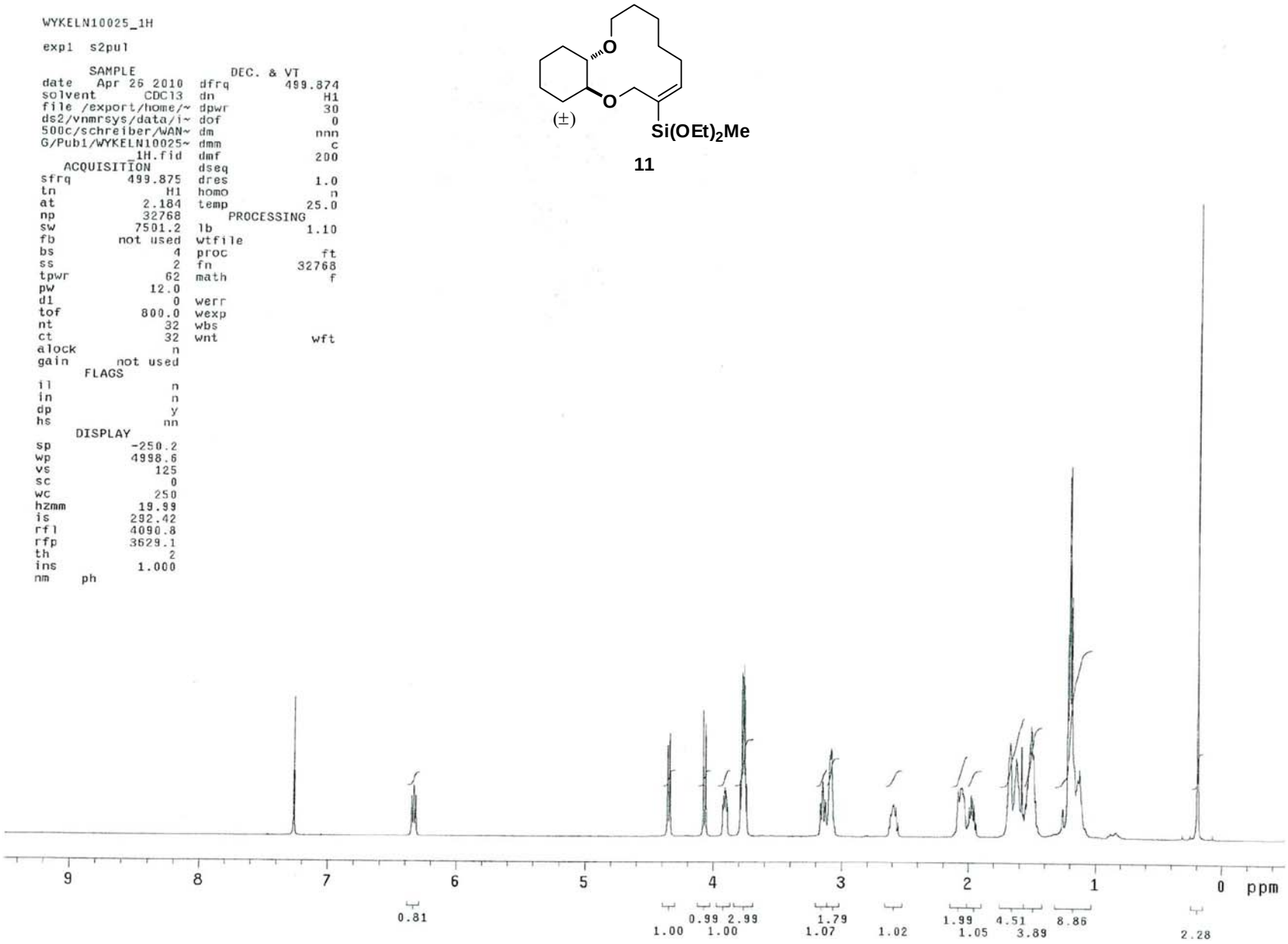
exp2 s2pul

SAMPLE		DEC. & VT	
date	Apr 25 2010	dfrq	499.874
solvent	CDCl3	dn	H1
file	exp	dpwr	48
ACQUISITION		dof	0
sfrq	125.707	dm	yyy
tn	C13	dmm	w
at	1.092	dmf	10000
np	65536	dseq	
sw	29996.3	dres	1.0
fb	not used	homo	n
bs	16	temp	25.0
tpwr	55	DEC2	
pw	2.0	dfrq2	0
d1	0	dn2	
tof	2000.0	dpwr2	1
nt	9999	dof2	0
ct	1056	dm2	n
alock	n	dmm2	c
gain	not used	dmf2	10000
FLAGS		dseq2	
il	n	dres2	1.0
in	n	homo2	n
dp	y	DEC3	
hs	nn	dfrq3	0
DISPLAY		dn3	
sp	-1087.8	dpwr3	1
wp	29995.3	dof3	0
vs	67	dm3	n
sc	0	dmm3	c
wc	250	dmf3	10000
hzmm	119.98	dseq3	
is	500.00	dres3	1.0
rfl	1088.7	homo3	n
rfp	0	PROCESSING	
th	4	lb	1.00
ins	100.000	wtfile	
nm cdc ph		proc	ft
		fn	not used
		math	f
		werr	
		wexp	
		wbs	
		wnt	



11a

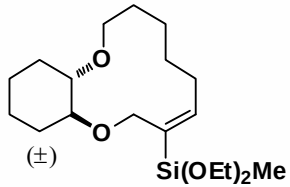



WYKELN10025_1H
expl s2pul
SAMPLE
date Apr 26 2010
solvent CDCl3
file /export/home/~
ds2/vnmrsys/data/i~
500c/schreiber/WAN~
G/Pub1/WYKELN10025~
1H.fid
ACQUISITION
sfrq 499.875
tn H1
at 2.184
np 32768
sw 7501.2
fb not used
bs 4
ss 2
tpwr 62
pw 12.0
d1 0
tof 800.0
nt 32
ct 32
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -250.2
wp 4998.6
vs 125
sc 0
wc 250
hzmm 19.99
is 292.42
rfl 4090.8
rfp 3629.1
th 2
ins 1.000
nm ph
DEC. & VT
dfrq 499.874
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
temp 25.0
PROCESSING
lb 1.10
wtfile
proc ft
fn 32768
math f
werr
wexp
wbs
wnt wft
O
O
(±)
Si(OEt)2Me
11
9
8
7
6
5
4
3
2
1
0 ppm
0.81
1.00
0.99
1.00
2.99
1.79
1.07
1.02
1.99
1.05
4.51
3.89
8.86
2.28

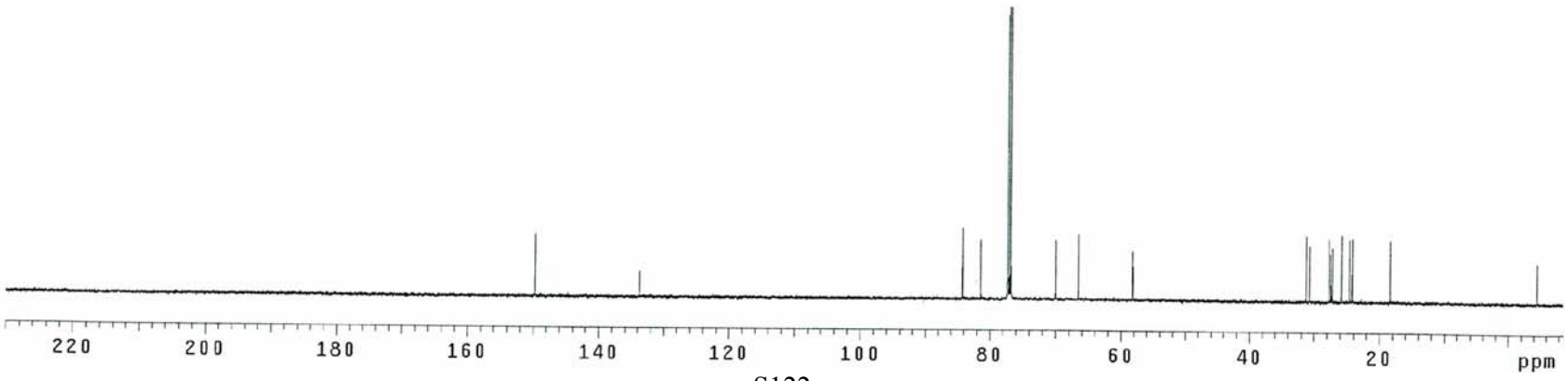
WYKELN10025_13C

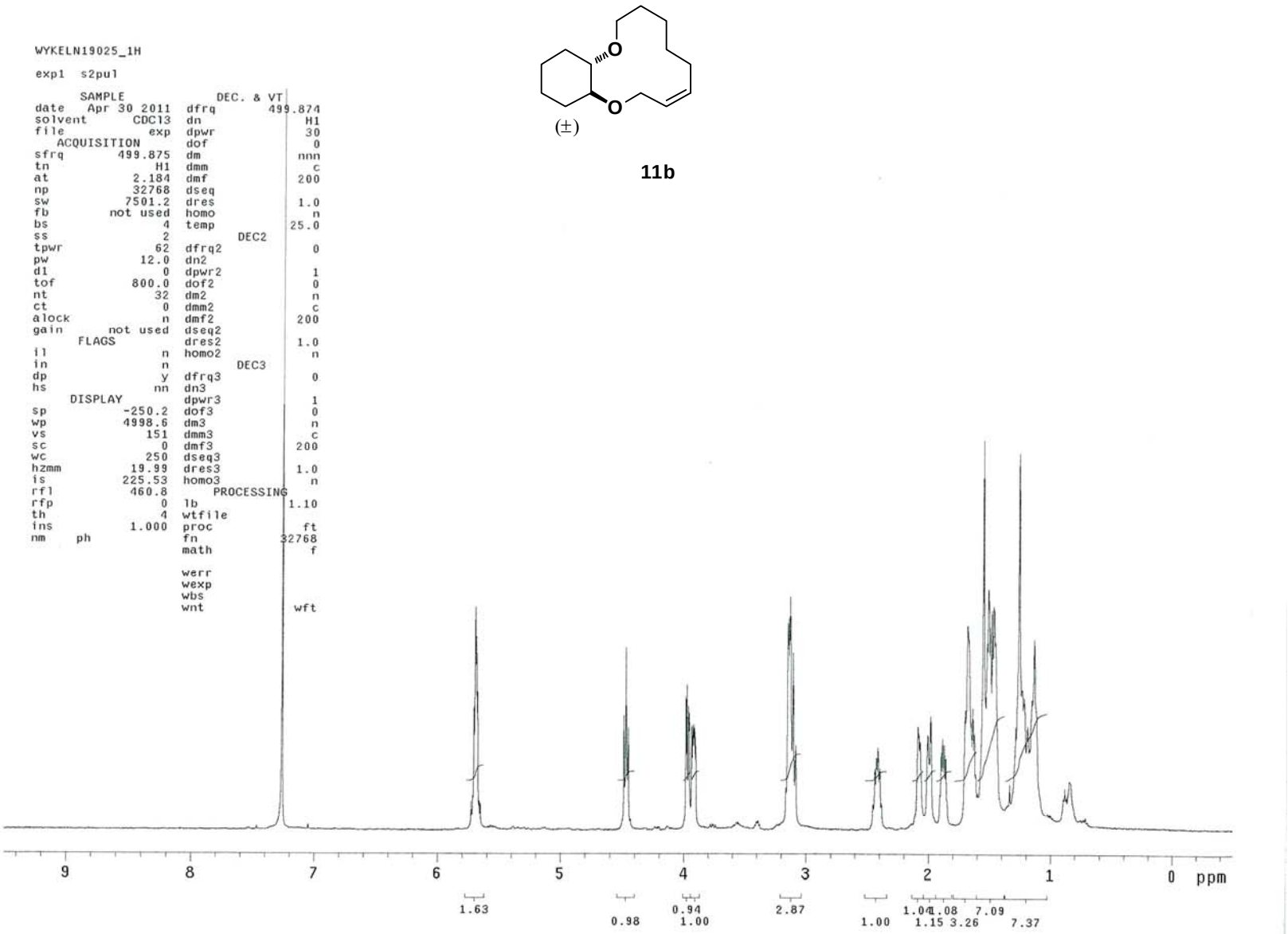
exp1 s2pul

SAMPLE		DEC. & VT	
date	Apr 26 2010	dfrq	499.874
solvent	CDCl3	dn	H1
file	exp	dpwr	48
ACQUISITION		dof	0
sfrq	125.707	dm	yyy
tn	C13	dmm	w
at	1.092	dmf	10000
np	65536	dseq	
sw	29996.3	dres	1.0
fb	not used	homo	n
bs	32	temp	25.0
tpwr	55	DEC2	
pw	4.2	dfrq2	0
d1	0	dn2	
tof	2000.0	dpwr2	1
nt	99999	dof2	0
ct	2016	dm2	n
alock	n	dmm2	c
gain	not used	dmf2	10000
FLAGS		dseq2	
il	n	dres2	1.0
in	n	homo2	n
dp	y	DEC3	
hs	nn	dfrq3	0
DISPLAY		dn3	
sp	-1086.9	dpwr3	1
wp	29995.3	dof3	0
vs	47	dm3	n
sc	0	dmm3	c
wc	250	dmf3	10000
hzmm	119.98	dseq3	
is	500.00	dres3	1.0
rfl	10766.2	homo3	n
rfp	9678.3	PROCESSING	
th	3	lb	1.00
ins	100.000	wtfile	
nm cdc ph		proc	ft
		fn	not used
		math	f
		werr	
		wexp	
		wbs	
		wnt	



11




WYKELN19025_1H
exp1 s2pul
SAMPLE
date Apr 30 2011
solvent CDCl3
file exp
ACQUISITION
sfrq 499.875
tn H1
at 2.184
np 32768
sw 7501.2
fb not used
bs 4
ss 2
tpwr 62
pw 12.0
d1 0
tof 800.0
nt 32
ct 0
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -250.2
wp 4998.6
vs 151
sc 0
wc 250
hzmm 19.99
is 225.53
rfl 460.8
rfp 0
th 4
ins 1.000
nm ph
DEC. & VT
dfrq 499.874
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
temp 25.0
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 200
dseq2
dres2 1.0
homo2 n
DEC3
dfrq3 0
dn3
dpwr3 1
dof3 0
dm3 n
dmm3 c
dmf3 200
dseq3
dres3 1.0
homo3 n
PROCESSING
lb 1.10
wtfile
proc ft
fn 32768
math f
werr
wexp
wbs
wnt wft
9
8
7
6
5
4
3
2
1
0 ppm
1.63
0.98
0.94
1.00
2.87
1.00
1.04
1.15
1.08
3.26
7.09
7.37

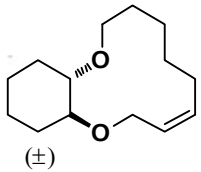
(±)

11b

WYKELN19025_13C

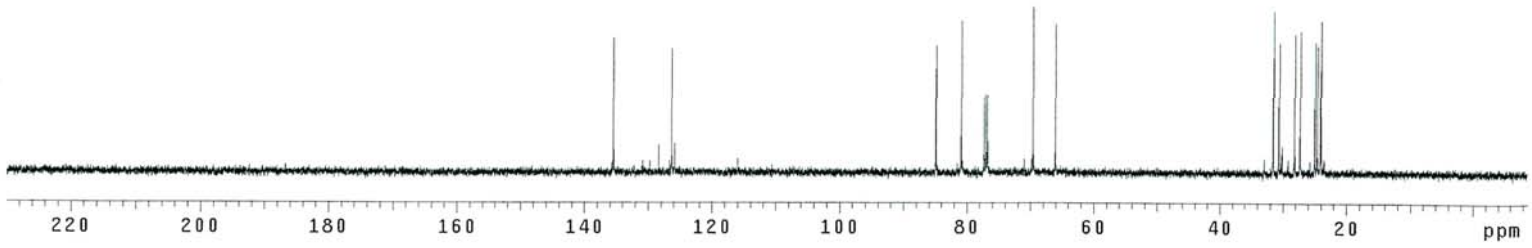
exp2 s2pul

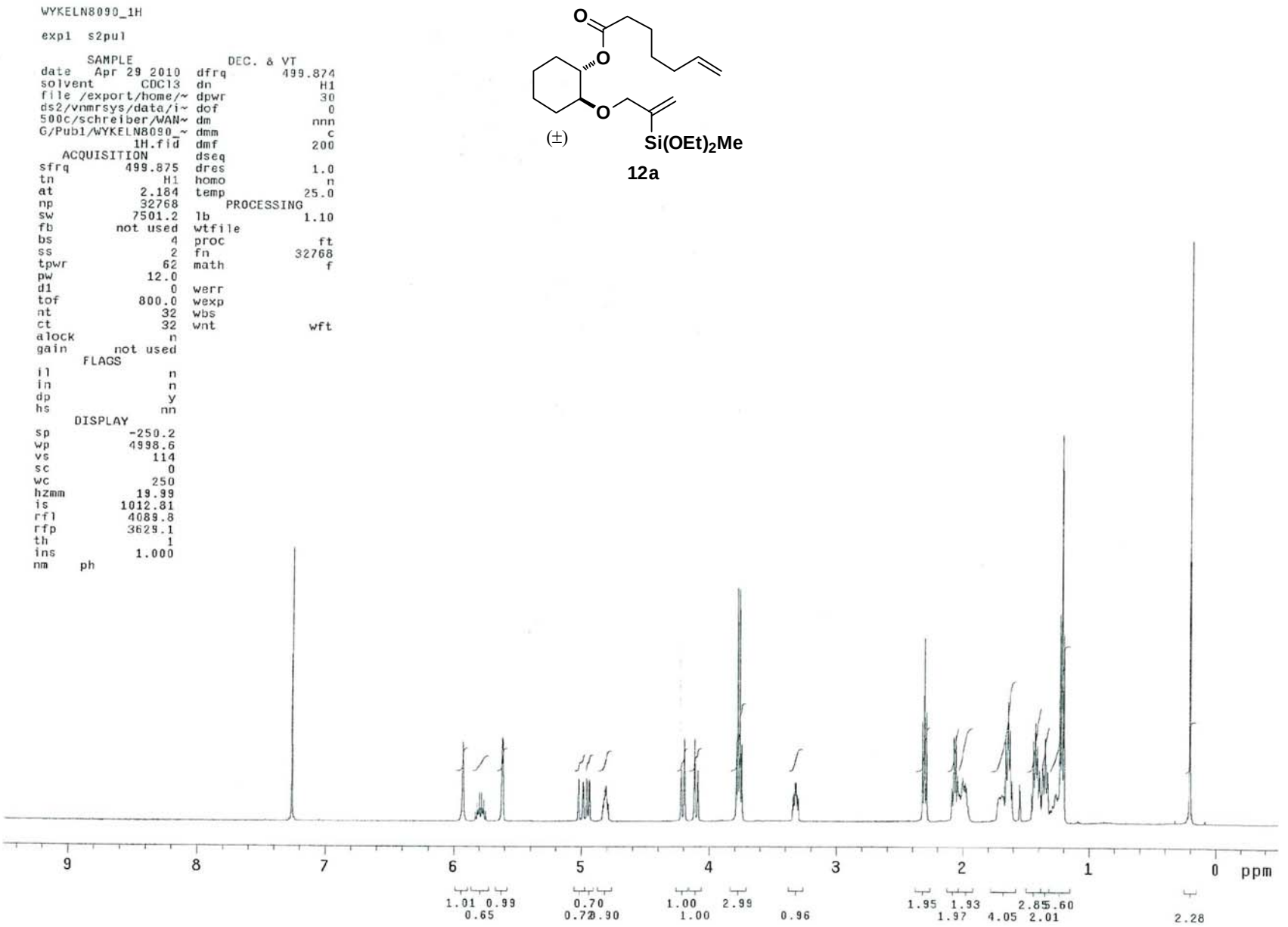
SAMPLE		DEC. & VT	
date	Apr 30 2011	dfrq	499.874
solvent	CDCl3	dn	H1
file	exp	dpwr	48
ACQUISITION		dof	0
sfrq	125.707	dm	yyy
tn	C13	dmm	w
at	1.092	dmf	8929
np	65536	dseq	
sw	29996.3	dres	1.0
fb	not used	homo	n
bs	16	temp	25.0
tpwr	55	DEC2	
pw	4.8	dfrq2	0
d1	0	dn2	
tof	2000.0	dpwr2	1
nt	9999	dof2	0
ct	0	dm2	n
alock	n	dmm2	c
gain	not used	dmf2	10000
FLAGS		dseq2	
il	n	dres2	1.0
in	n	homo2	n
dp	y	DEC3	
hs	nn	dfrq3	0
DISPLAY		dn3	
sp	-1095.1	dpwr3	1
wp	29995.3	dof3	0
vs	27	dm3	n
sc	0	dmm3	c
wc	250	dmf3	10000
hzmm	119.98	dseq3	
is	500.00	dres3	1.0
rfl	10774.4	homo3	n
rfp	9678.3	PROCESSING	
th	10	lb	1.00
ins	100.000	wtfile	
nm cdc ph		proc	ft
		fn	not used
		math	f
		werr	
		wexp	
		wbs	
		wnt	



(±)

11b

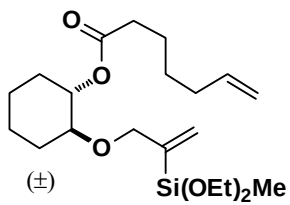



WYKELN8090_1H
expl s2pul
SAMPLE
date Apr 29 2010
solvent CDCl3
file /export/home/~
ds2/vnmrsys/data/i~
500c/schreiber/WAN~
G/Pub1/WYKELN8090_~
1H.fid
ACQUISITION
sfrq 499.875
tn H1
at 2.184
np 32768
sw 7501.2
fb not used
bs 4
ss 2
tpwr 62
pw 12.0
d1 0
tof 800.0
nt 32
ct 32
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -250.2
wp 4998.6
vs 114
sc 0
wc 250
hzmm 19.99
is 1012.81
rfl 4089.8
rfp 3629.1
th 1
ins 1.000
nm ph
DEC. & VT
dfrq 499.874
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
temp 25.0
PROCESSING
lb 1.10
wtfile
proc ft
fn 32768
math f
werr
wexp
wbs
wnt wft
O
O
O
(±)
Si(OEt)2Me
12a
9
8
7
6
5
4
3
2
1
0 ppm
1.01 0.99
0.65
0.70
0.72 0.90
1.00
1.00
2.99
0.96
1.95 1.93
1.97
4.05
2.85 5.60
2.01
2.28

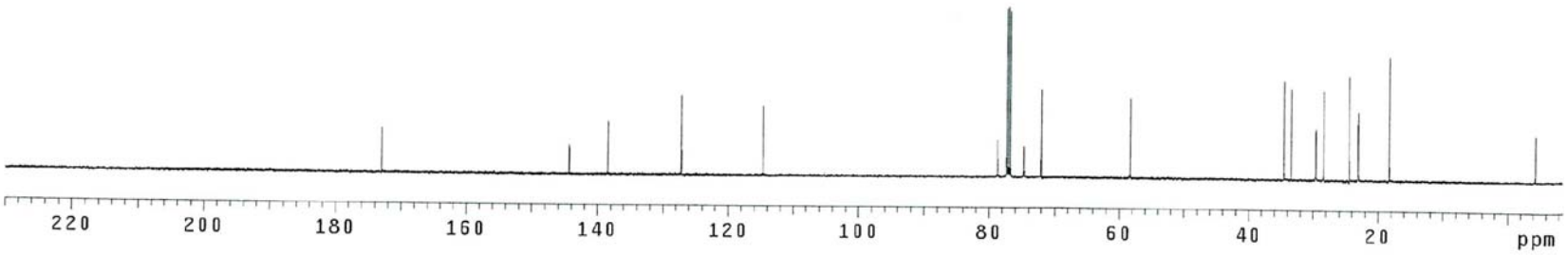
WYKELN8090_13C

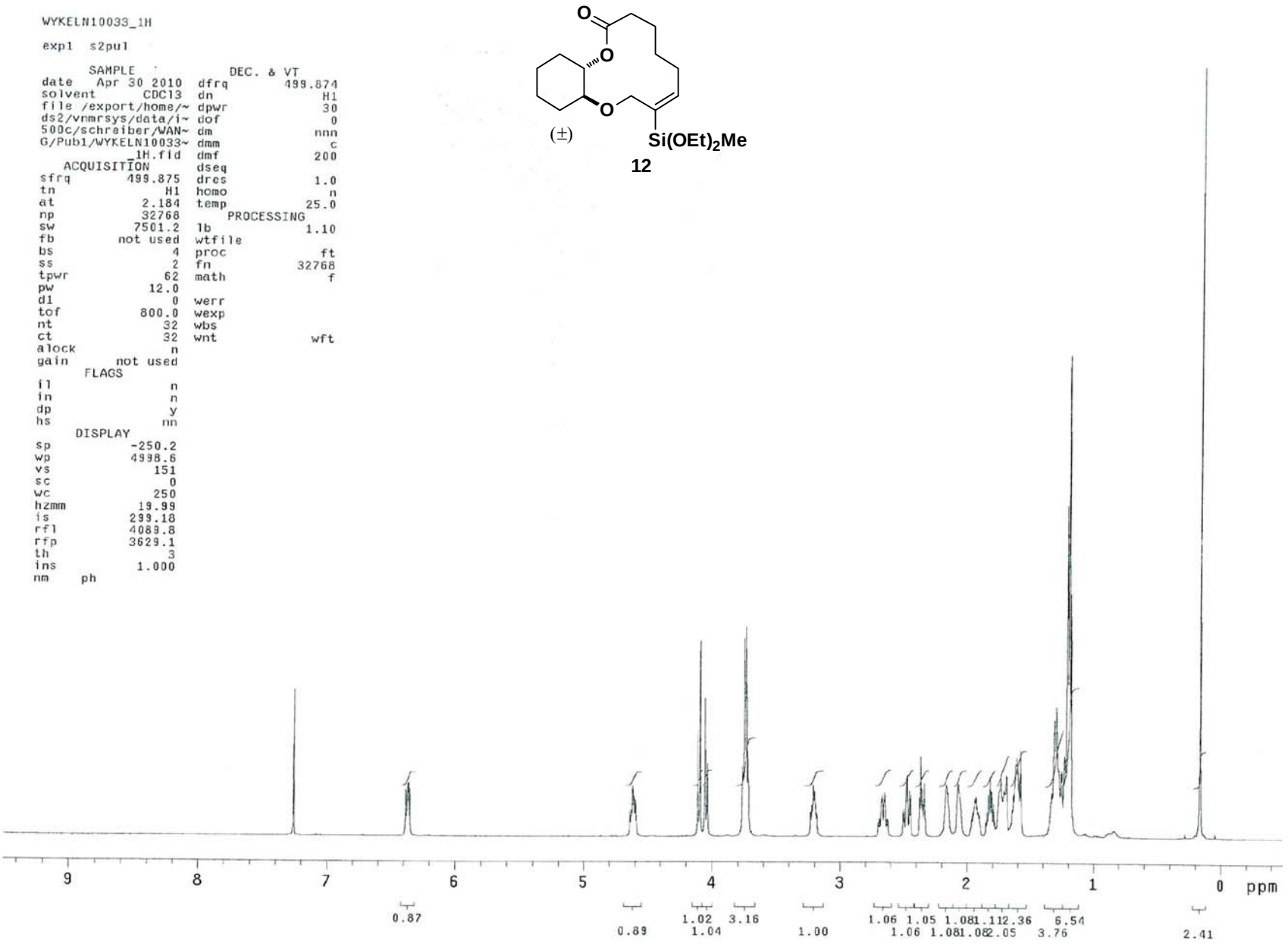
exp2 s2pul

SAMPLE		DEC. & VT	
date	Apr 29 2010	dfrq	499.874
solvent	CDCl3	dn	H1
file	exp	dpwr	48
ACQUISITION		dof	0
sfrq	125.707	dm	yyy
tn	C13	dmm	w
at	1.092	dmf	10000
np	65536	dseq	
sw	29996.3	dres	1.0
fb	not used	homo	n
bs	32	temp	25.0
tpwr	55	DEC2	
pw	4.2	dfrq2	0
d1	0	dn2	
tof	2000.0	dpwr2	1
nt	99999	dof2	0
ct	1280	dm2	n
alock	n	dmm2	c
gain	not used	dmf2	10000
FLAGS		dseq2	
il	n	dres2	1.0
in	n	homo2	n
dp	y	DEC3	
hs	nn	dfrq3	0
DISPLAY		dn3	
sp	-1087.8	dpwr3	1
wp	29995.3	dof3	0
vs	27	dm3	n
sc	0	dmm3	c
wc	250	dmf3	10000
hzmm	119.98	dseq3	
is	500.00	dres3	1.0
rfl	10767.1	homo3	n
rfp	9678.3	PROCESSING	
th	3	lb	1.00
ins	100.000	wtfile	
nm cdc ph		proc	ft
		fn	not used
		math	f
		werr	
		wexp	
		wbs	
		wnt	



12a

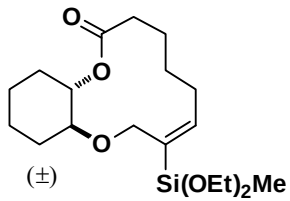



WYKELN10033_1H
exp1 s2pul
SAMPLE
date Apr 30 2010
solvent CDCl3
file /export/home/~
ds2/vnmrsys/data/i~
500c/schreiber/WAN~
G/Pub1/WYKELN10033~
_1H.fid
ACQUISITION
sfrq 499.875
tn H1
at 2.184
np 32768
sw 7501.2
fb not used
bs 4
ss 2
tpwr 62
pw 12.0
d1 0
tof 800.0
nt 32
ct 32
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -250.2
wp 4998.6
vs 151
sc 0
wc 250
hzmm 19.99
is 299.18
rfl 4089.8
rfp 3629.1
th 3
ins 1.000
nm ph
DEC. & VT
dfrq 499.874
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
temp 25.0
PROCESSING
lb 1.10
wtfile
proc ft
fn 32768
math f
werr
wexp
wbs
wnt wft
O
O
O
(±)
Si(OEt)2Me
12
9
8
7
6
5
4
3
2
1
0 ppm
0.87
0.89
1.02
1.04
3.16
1.00
1.06
1.06
1.05
1.08
1.08
1.11
2.05
2.36
3.76
6.54
2.41

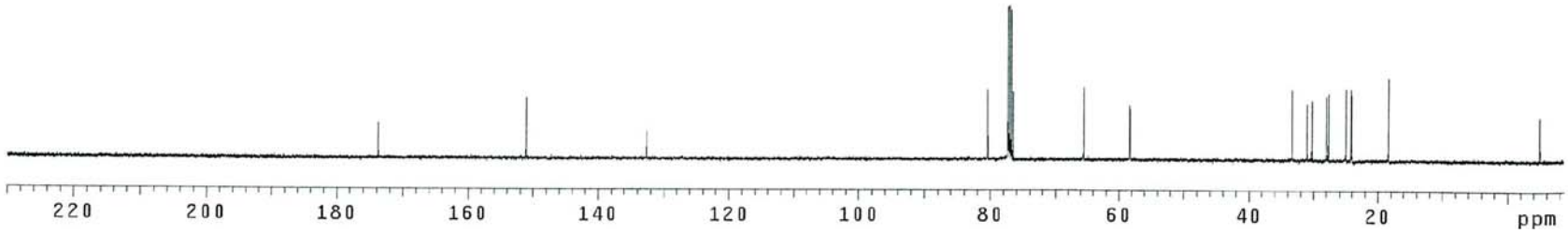
WYKELN10033_13C

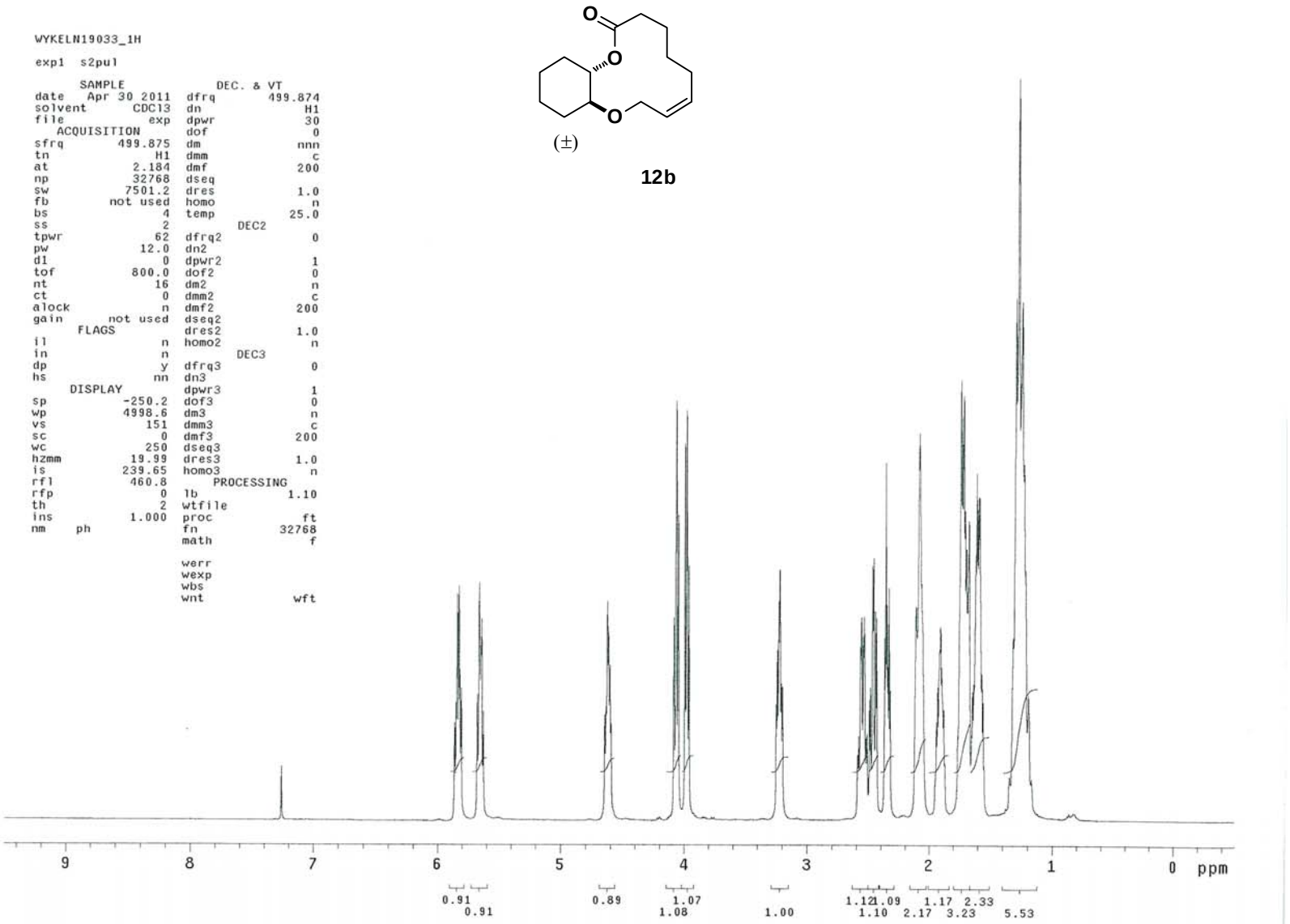
exp3 s2pul

SAMPLE		DEC. & VT	
date	Apr 30 2010	dfrq	499.874
solvent	CDCl3	dn	H1
file	exp	dpwr	48
ACQUISITION		dof	0
sfrq	125.707	dm	yyy
tn	C13	dmm	w
at	1.092	dmf	10000
np	65536	dseq	
sw	29996.3	dres	1.0
fb	not used	homo	n
bs	32	temp	25.0
tpwr	55	DEC2	
pw	4.2	dfrq2	0
d1	0	dn2	
tof	2000.0	dpwr2	1
nt	99999	dof2	0
ct	800	dm2	n
alock	n	dmm2	c
gain	not used	dmf2	10000
FLAGS		dseq2	
il	n	dres2	1.0
in	n	homo2	n
dp	y	DEC3	
hs	nn	dfrq3	0
DISPLAY		dn3	
sp	-1087.8	dpwr3	1
wp	29995.3	dof3	0
vs	25	dm3	n
sc	0	dmm3	c
wc	250	dmf3	10000
hzmm	119.98	dseq3	
is	500.00	dres3	1.0
rfl	10767.1	homo3	n
rfp	9678.3	PROCESSING	
th	3	lb	1.00
ins	100.000	wtfile	
nm cdc ph		proc	ft
		fn	not used
		math	f
		werr	
		wexp	
		wbs	
		wnt	

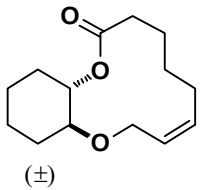


12



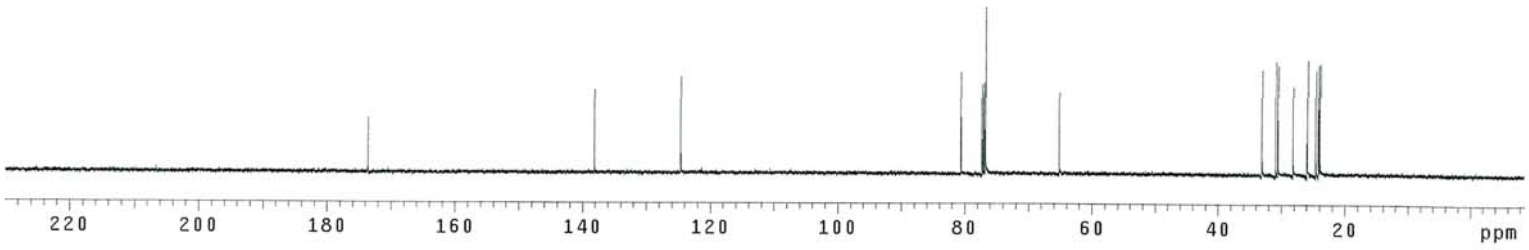

WYKELN19033_1H
exp1 s2pul
SAMPLE
date Apr 30 2011
solvent CDCl3
file exp
ACQUISITION
sfrq 499.875
tn H1
at 2.184
np 32768
sw 7501.2
fb not used
bs 4
ss 2
tpwr 62
pw 12.0
d1 0
tof 800.0
nt 16
ct 0
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -250.2
wp 4998.6
vs 151
sc 0
wc 250
hzmm 19.99
is 239.65
rfl 460.8
rfp 0
th 2
ins 1.000
nm ph
DEC. & VT
dfrq 499.874
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
temp 25.0
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 200
dseq2
dres2 1.0
homo2 n
DEC3
dfrq3 0
dn3
dpwr3 1
dof3 0
dm3 n
dmm3 c
dmf3 200
dseq3
dres3 1.0
homo3 n
PROCESSING
lb 1.10
wtfile
proc ft
fn 32768
math f
werr
wexp
wbs
wnt wft
O
O
O
(±)
12b
9
8
7
6
5
4
3
2
1
0 ppm
0.91
0.91
0.89
1.07
1.08
1.00
1.12
1.10
1.09
1.17
2.17
2.33
3.23
5.53

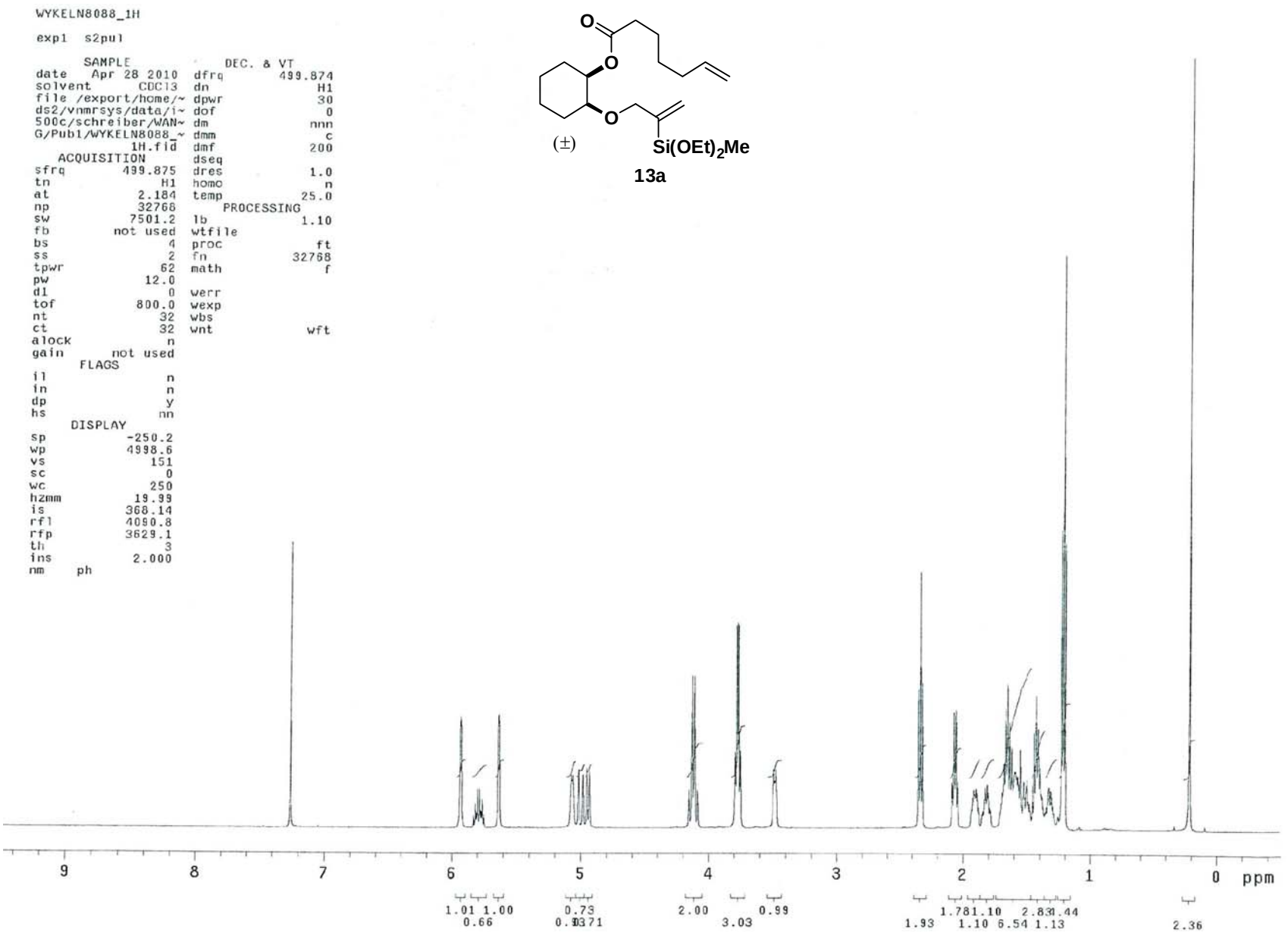
```
WYKELN19033_13C

exp2  s2pul

        SAMPLE
date    Apr 30 2011
solvent       CDCl3
file             exp
   ACQUISITION
sfrq         125.707
tn               C13
at             1.092
np             65536
sw           29996.3
fb         not used
bs               16
tpwr             55
pw              4.8
d1                0
tof          2000.0
nt             9999
ct                0
alock             n
gain       not used
      FLAGS
il                n
in                n
dp                y
hs               nn
     DISPLAY
sp           -1092.4
wp           29995.3
vs                27
sc                 0
wc               250
hzmm          119.98
is            500.00
rfl          10771.7
rfp           9678.3
th                 4
ins          100.000
nm  cdc  ph

        DEC. & VT
dfrq         499.874
dn                H1
dpwr              48
dof                0
dm               yyy
dmm                w
dmf             8929
dseq
dres             1.0
homo               n
temp            25.0
      DEC2
dfrq2              0
dn2
dpwr2              1
dof2               0
dm2                n
dmm2               c
dmf2           10000
dseq2
dres2            1.0
homo2              n
      DEC3
dfrq3              0
dn3
dpwr3              1
dof3               0
dm3                n
dmm3               c
dmf3           10000
dseq3
dres3            1.0
homo3              n
    PROCESSING
lb              1.00
wtfile
proc               ft
fn           not used
math               f

werr
wexp
wbs
wnt
```



(±)

12b

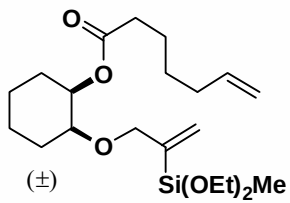



WYKELN8088_1H
exp1 s2pul
SAMPLE
date Apr 28 2010
solvent CDCl3
file /export/home/~
ds2/vnmrsys/data/i~
500c/schreiber/WAN~
G/Pub1/WYKELN8088_~
1H.fid
ACQUISITION
sfrq 499.875
tn H1
at 2.184
np 32768
sw 7501.2
fb not used
bs 4
ss 2
tpwr 62
pw 12.0
d1 0
tof 800.0
nt 32
ct 32
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -250.2
wp 4998.6
vs 151
sc 0
wc 250
hzmm 19.99
is 368.14
rfl 4090.8
rfp 3629.1
th 3
ins 2.000
nm ph
DEC. & VT
dfrq 499.874
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
temp 25.0
PROCESSING
lb 1.10
wtfile
proc ft
fn 32768
math f
werr
wexp
wbs
wnt wft
O
O
O
(±)
Si(OEt)2Me
13a
9
8
7
6
5
4
3
2
1
0 ppm
1.01 1.00
0.66
0.73
2.00
0.99
3.03
1.93
1.78 1.10
1.10 6.54
2.83 1.44
1.13
2.36

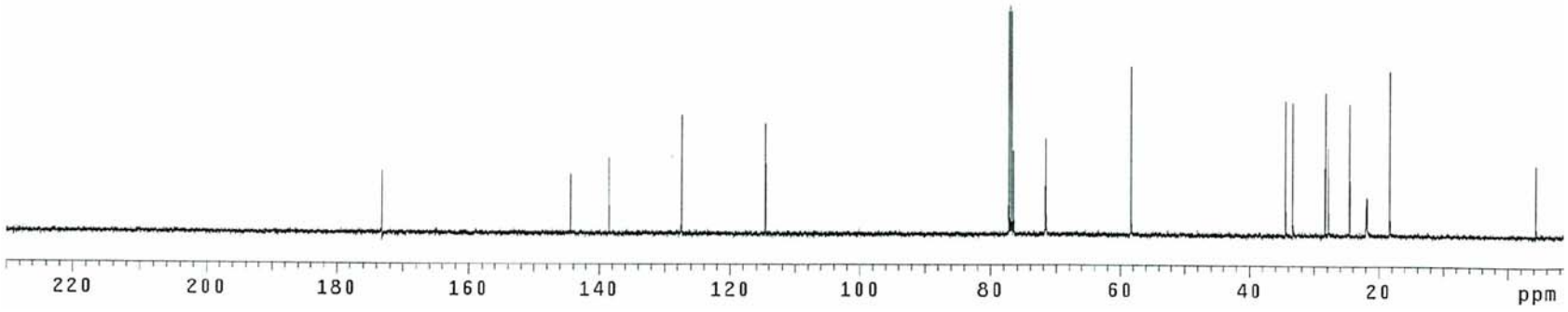
WYKELN8088_13C

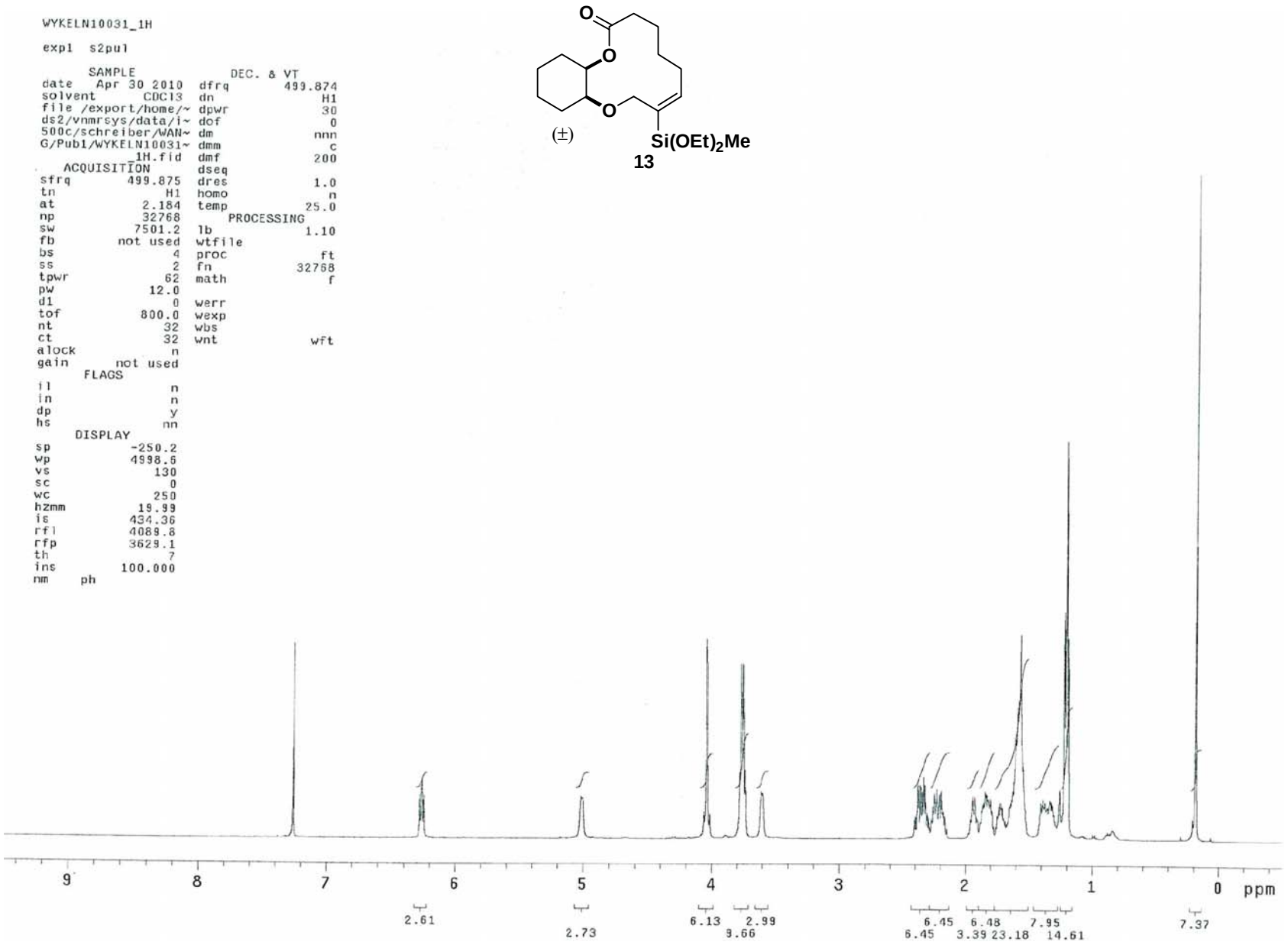
exp3 s2pul

SAMPLE		DEC. & VT	
date	Apr 28 2010	dfrq	499.874
solvent	CDCl3	dn	H1
file	exp	dpwr	48
ACQUISITION		dof	0
sfrq	125.707	dm	yyy
tn	C13	dmm	w
at	1.092	dmf	10000
np	65536	dseq	
sw	29996.3	dres	1.0
fb	not used	homo	n
bs	32	temp	25.0
tpwr	55	DEC2	
pw	4.2	dfrq2	0
d1	0	dn2	
tof	2000.0	dpwr2	1
nt	99999	dof2	0
ct	736	dm2	n
alock	n	dmm2	c
gain	not used	dmf2	10000
FLAGS		dseq2	
fl	n	dres2	1.0
in	n	homo2	n
dp	y	DEC3	
hs	nn	dfrq3	0
DISPLAY		dn3	
sp	-1087.8	dpwr3	1
wp	29995.3	dof3	0
vs	37	dm3	n
sc	0	dmm3	c
wc	250	dmf3	10000
hzmm	119.98	dseq3	
is	500.00	dres3	1.0
rfl	1088.7	homo3	n
rfp	0	PROCESSING	
th	2	lb	1.00
ins	100.000	wtfile	
nm cdc ph		proc	ft
		fn	not used
		math	f
		werr	
		wexp	
		wbs	
		wnt	



13a

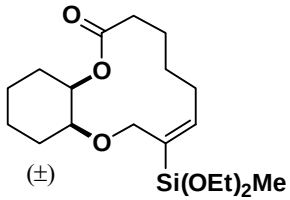



WYKELN10031_1H
expl s2pul
SAMPLE
date Apr 30 2010
solvent CDCl3
file /export/home/~
ds2/vnmrsys/data/i~
500c/schreiber/WAN~
G/Publ/WYKELN10031~
_1H.fid
ACQUISITION
sfrq 499.875
tn H1
at 2.184
np 32768
sw 7501.2
fb not used
bs 4
ss 2
tpwr 62
pw 12.0
d1 0
tof 800.0
nt 32
ct 32
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -250.2
wp 4998.6
vs 130
sc 0
wc 250
hzmm 19.99
is 434.36
rfl 4089.8
rfp 3629.1
th 7
ins 100.000
nm ph
DEC. & VT
dfrq 499.874
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
temp 25.0
PROCESSING
lb 1.10
wtfile
proc ft
fn 32768
math f
werr
wexp
wbs
wnt wft
O
O
O
(±)
Si(OEt)2Me
13
9
8
7
6
5
4
3
2
1
0 ppm
2.61
2.73
6.13
2.99
9.66
6.45
6.48
7.95
6.45
3.39
23.18
14.61
7.37

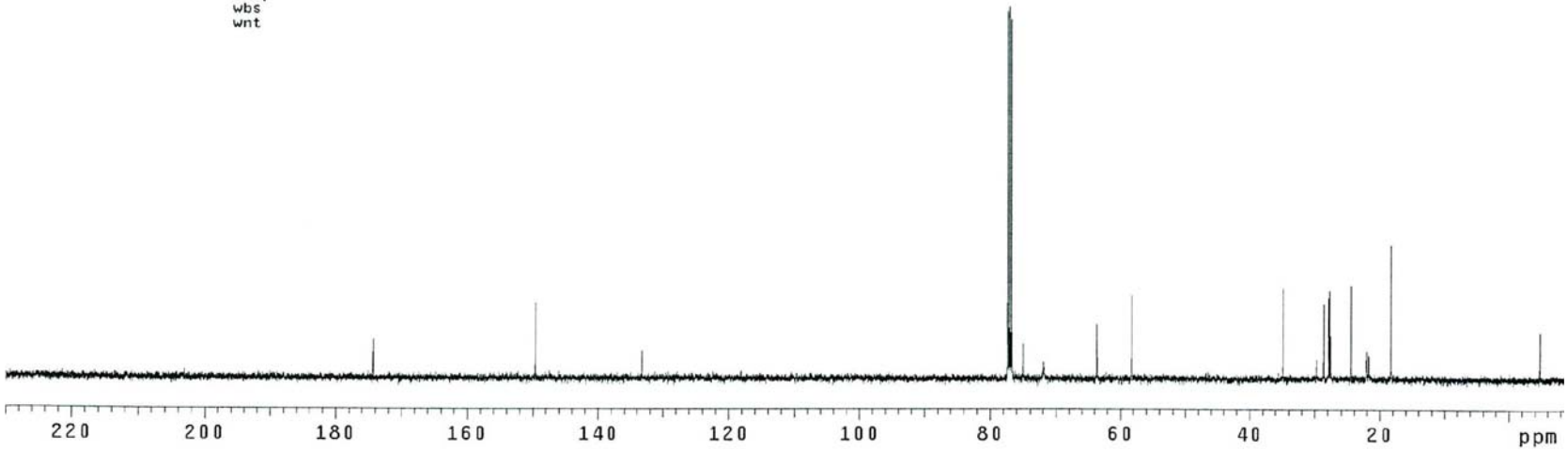
WYKELN10031_13C

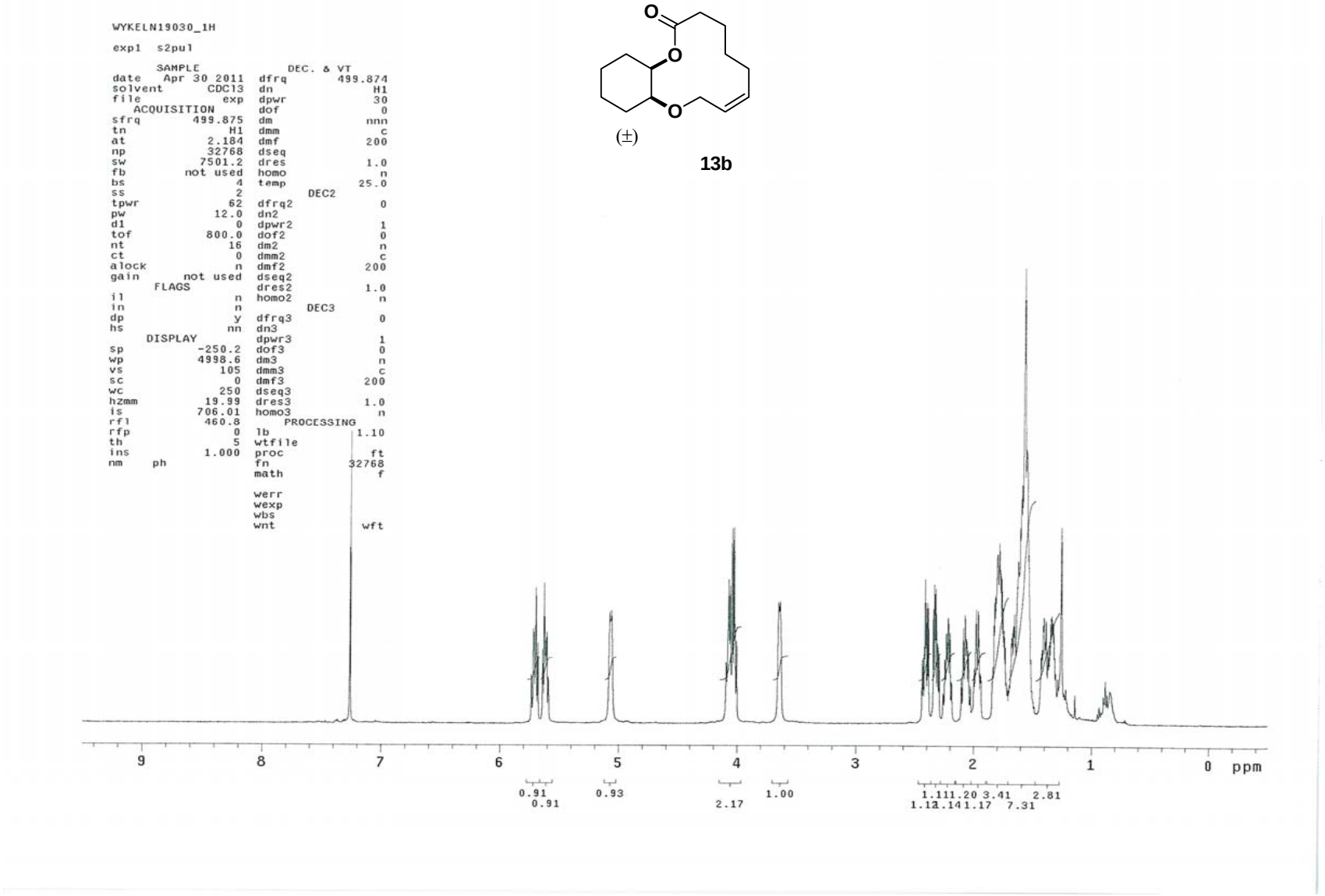
exp3 s2pul

SAMPLE		DEC. & VT	
date	Apr 30 2010	dfrq	499.874
solvent	CDCl3	dn	H1
file	exp	dpwr	48
ACQUISITION		dof	0
sfrq	125.707	dm	yyy
tn	C13	dmm	w
at	1.092	dmf	10000
np	65536	dseq	
sw	29996.3	dres	1.0
fb	not used	homo	n
bs	32	temp	25.0
tpwr	55	DEC2	
pw	4.2	dfrq2	0
d1	0	dn2	
tof	2000.0	dpwr2	1
nt	99999	dof2	0
ct	1856	dm2	n
alock	n	dmm2	c
gain	not used	dmf2	10000
FLAGS		dseq2	
il	n	dres2	1.0
in	n	homo2	n
dp	y	DEC3	
hs	nn	dfrq3	0
DISPLAY		dn3	
sp	-1086.9	dpwr3	1
wp	29995.3	dof3	0
vs	60	dm3	n
sc	0	dmm3	c
wc	250	dmf3	10000
hzmm	119.98	dseq3	
is	500.00	dres3	1.0
rfl	1087.8	homo3	n
rfp	0	PROCESSING	
th	2	lb	1.00
ins	100.000	wtfile	
nm cdc ph		proc	ft
		fn	not used
		math	f
		werr	
		wexp	
		wbs	
		wnt	



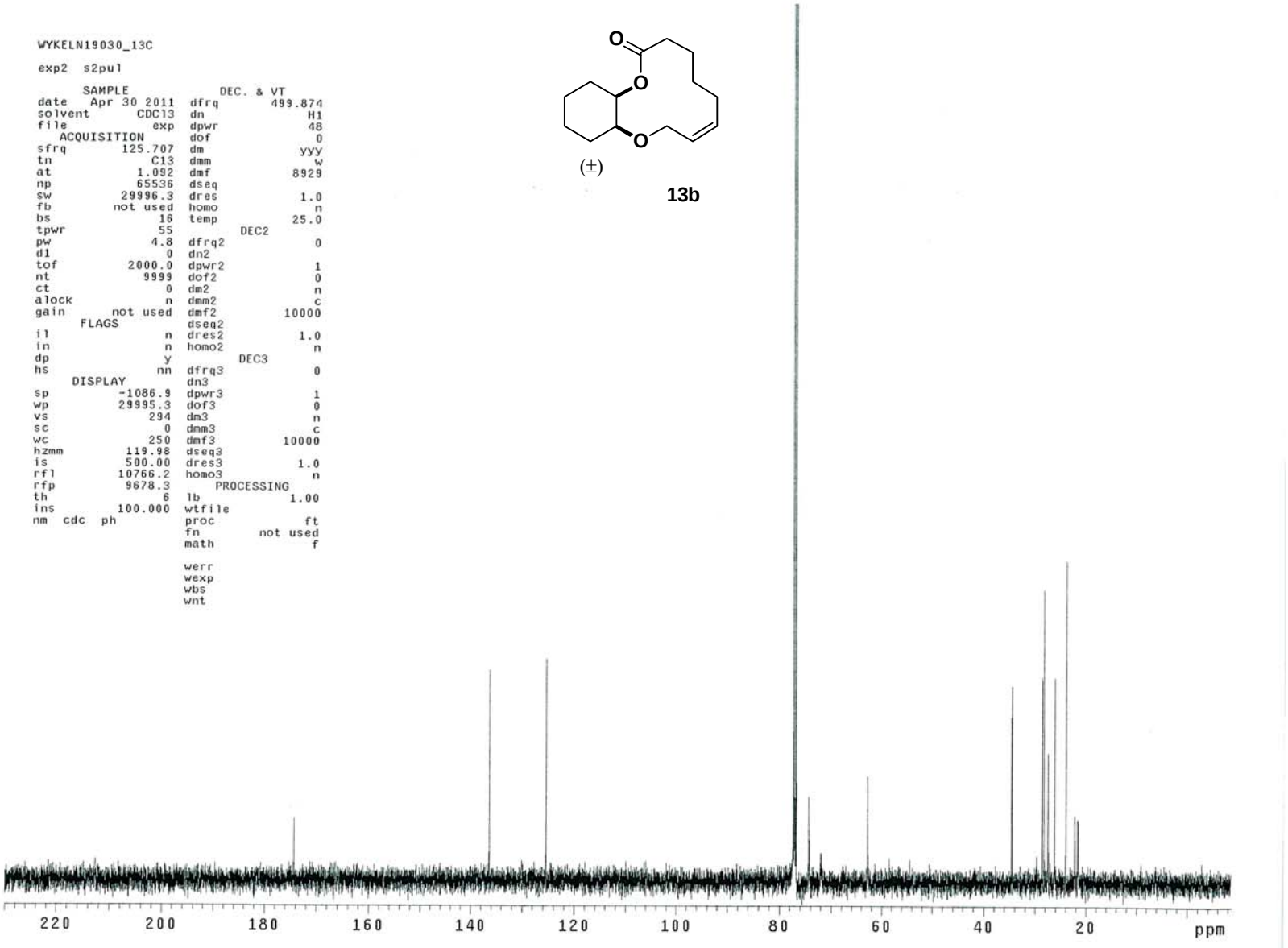
13

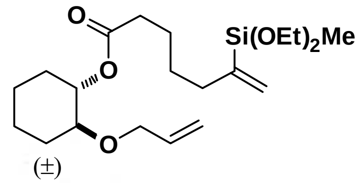



WYKELN19030_1H
exp1 s2pul
SAMPLE
date Apr 30 2011
solvent CDCl3
file exp
ACQUISITION
sfrq 499.875
tn H1
at 2.184
np 32768
sw 7501.2
fb not used
bs 4
ss 2
tpwr 62
pw 12.0
d1 0
tof 800.0
nt 16
ct 0
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -250.2
wp 4998.6
vs 105
sc 0
wc 250
hzmm 19.99
is 706.01
rfl 460.8
rfp 0
th 5
ins 1.000
nm ph
DEC. & VT
dfrq 499.874
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
temp 25.0
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 200
dseq2
dres2 1.0
homo2 n
DEC3
dfrq3 0
dn3
dpwr3 1
dof3 0
dm3 n
dmm3 c
dmf3 200
dseq3
dres3 1.0
homo3 n
PROCESSING
lb 1.10
wtfile
proc ft
fn 32768
math f
werr
wexp
wbs
wnt wft
9
8
7
6
5
4
3
2
1
0 ppm
0.91
0.91
0.93
2.17
1.00
1.11
1.12
1.20
1.14
3.41
1.17
2.81
7.31

(±)

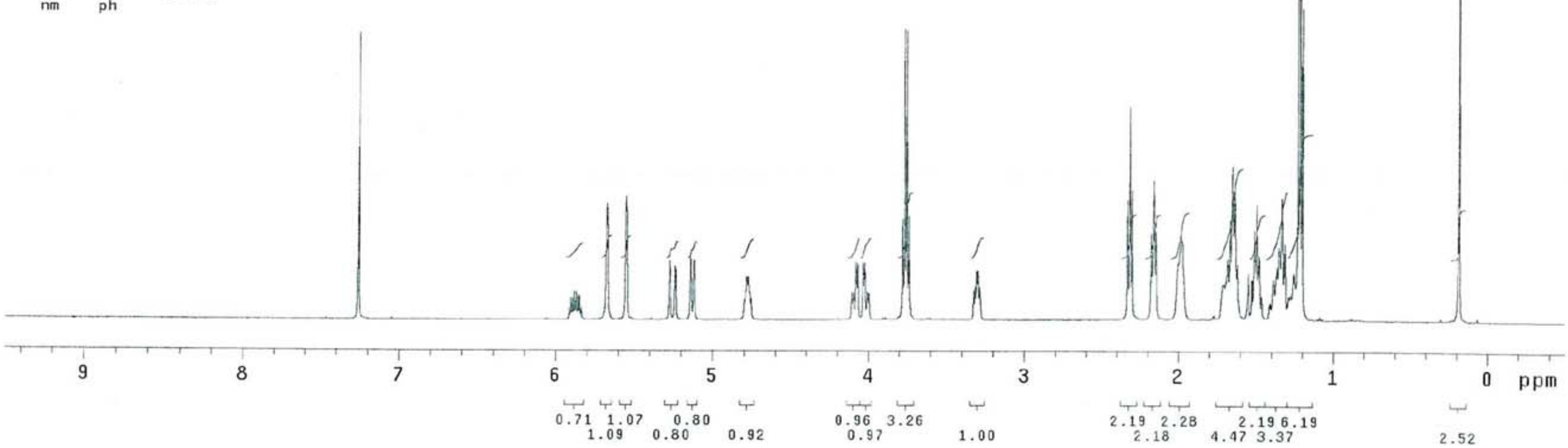
13b


WYKELN19030_13C
exp2 s2pul
SAMPLE
date Apr 30 2011
solvent CDCl3
file exp
ACQUISITION
sfrq 125.707
tn C13
at 1.092
np 65536
sw 29996.3
fb not used
bs 16
tpwr 55
pw 4.8
d1 0
tof 2000.0
nt 9999
ct 0
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -1086.9
wp 29995.3
vs 294
sc 0
wc 250
hzmm 119.98
is 500.00
rfl 10766.2
rfp 9678.3
th 6
ins 100.000
nm cdc ph
DEC. & VT
dfrq 499.874
dn H1
dpwr 48
dof 0
dm yyy
dmm w
dmf 8929
dseq
dres 1.0
homo n
temp 25.0
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 10000
dseq2
dres2 1.0
homo2 n
DEC3
dfrq3 0
dn3
dpwr3 1
dof3 0
dm3 n
dmm3 c
dmf3 10000
dseq3
dres3 1.0
homo3 n
PROCESSING
lb 1.00
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
O
O
O
(±)
13b
220
200
180
160
140
120
100
80
60
40
20
ppm



14a

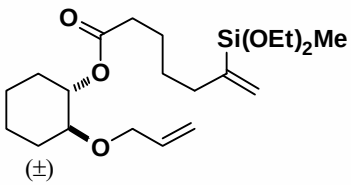
```
WYKELN8089_1H

exp1  s2pul

      SAMPLE                DEC. & VT
date   Apr 29 2010   dfrq       499.874
solvent      CDCl3   dn              H1
file /export/home/~  dpwr            30
ds2/vnmrsys/data/i~  dof              0
500c/schreiber/WAN~  dm             nnn
G/Pub1/WYKELN8089_~  dmm              c
            1H.fid   dmf            200
   ACQUISITION       dseq
sfrq       499.875   dres           1.0
tn              H1   homo             n
at           2.184   temp          25.0
np           32768         PROCESSING
sw          7501.2   lb            1.10
fb        not used   wtfile
bs               4   proc            ft
ss               2   fn           32768
tpwr            62   math             f
pw            12.0
d1               0   werr
tof          800.0   wexp
nt              32   wbs
ct              32   wnt            wft
alock            n
gain      not used
      FLAGS
il               n
in               n
dp               y
hs              nn
     DISPLAY
sp          -250.2
wp          4998.6
vs             102
sc               0
wc             250
hzmm         19.99
is          741.30
rfl         4089.8
rfp         3629.1
th               1
ins          1.000
nm    ph
```



WYKELN8089_13C

exp2 s2pul

SAMPLE		DEC. & VT	
date	Apr 29 2010	dfrq	499.874
solvent	CDCl3	dn	H1
file	exp	dpwr	48
ACQUISITION		dof	0
sfrq	125.707	dm	yyy
tn	C13	dmm	w
at	1.092	dmf	10000
np	65536	dseq	
sw	29996.3	dres	1.0
fb	not used	homo	n
bs	32	temp	25.0
tpwr	55	DEC2	
pw	4.2	dfrq2	0
d1	0	dn2	
tof	2000.0	dpwr2	1
nt	99999	dof2	0
ct	928	dm2	n
alock	n	dmm2	c
gain	not used	dmf2	10000
FLAGS		dseq2	
il	n	dres2	1.0
in	n	homo2	n
dp	y	DEC3	
hs	nn	dfrq3	0
DISPLAY		dn3	
sp	-1087.8	dpwr3	1
wp	29995.3	dof3	0
vs	49	dm3	n
sc	0	dmm3	c
wc	250	dmf3	10000
hzmm	119.98	dseq3	
is	500.00	dres3	1.0
rfl	10767.1	homo3	n
rfp	9678.3	PROCESSING	
th	4	lb	1.00
ins	100.000	wtfile	
nm cdc ph		proc	ft
		fn	not used
		math	f
		werr	
		wexp	
		wbs	
		wnt	



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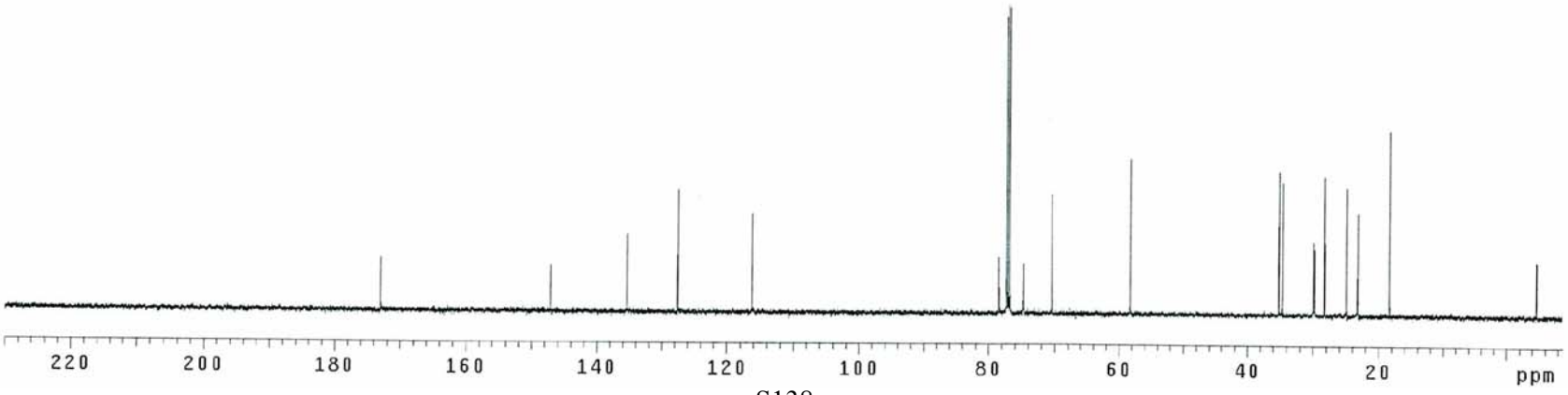
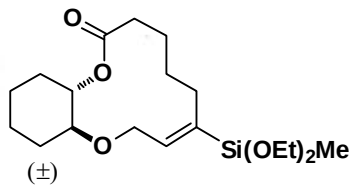
14a



WYKELN10032_1H

exp1 s2pul

SAMPLE		DEC. & VT	
date	Apr 30 2010	dfrq	499.874
solvent	CDCl3	dn	H1
file	/export/home/~	dpwr	30
	ds2/vnmrsys/data/i~	dof	0
	500c/schreiber/WAN~	dm	nnn
	G/Pub1/WYKELN10032~	dmm	c
	1H.fid	dmf	200
ACQUISITION		dseq	
sfrq	499.875	dres	1.0
tn	H1	homo	n
at	2.184	temp	25.0
np	32768	PROCESSING	
sw	7501.2	lb	1.10
fb	not used	wtfile	
bs	4	proc	ft
ss	2	fn	32768
tpwr	52	math	f
pw	12.0		
d1	0	werr	
tof	800.0	wexp	
nt	32	wbs	
ct	32	wnt	wft
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-250.2		
wp	4998.6		
vs	100		
sc	0		
wc	250		
hzmm	19.99		
is	598.03		
rfl	4089.8		
rfp	3629.1		
th	1		
ins	1.000		
nm	ph		



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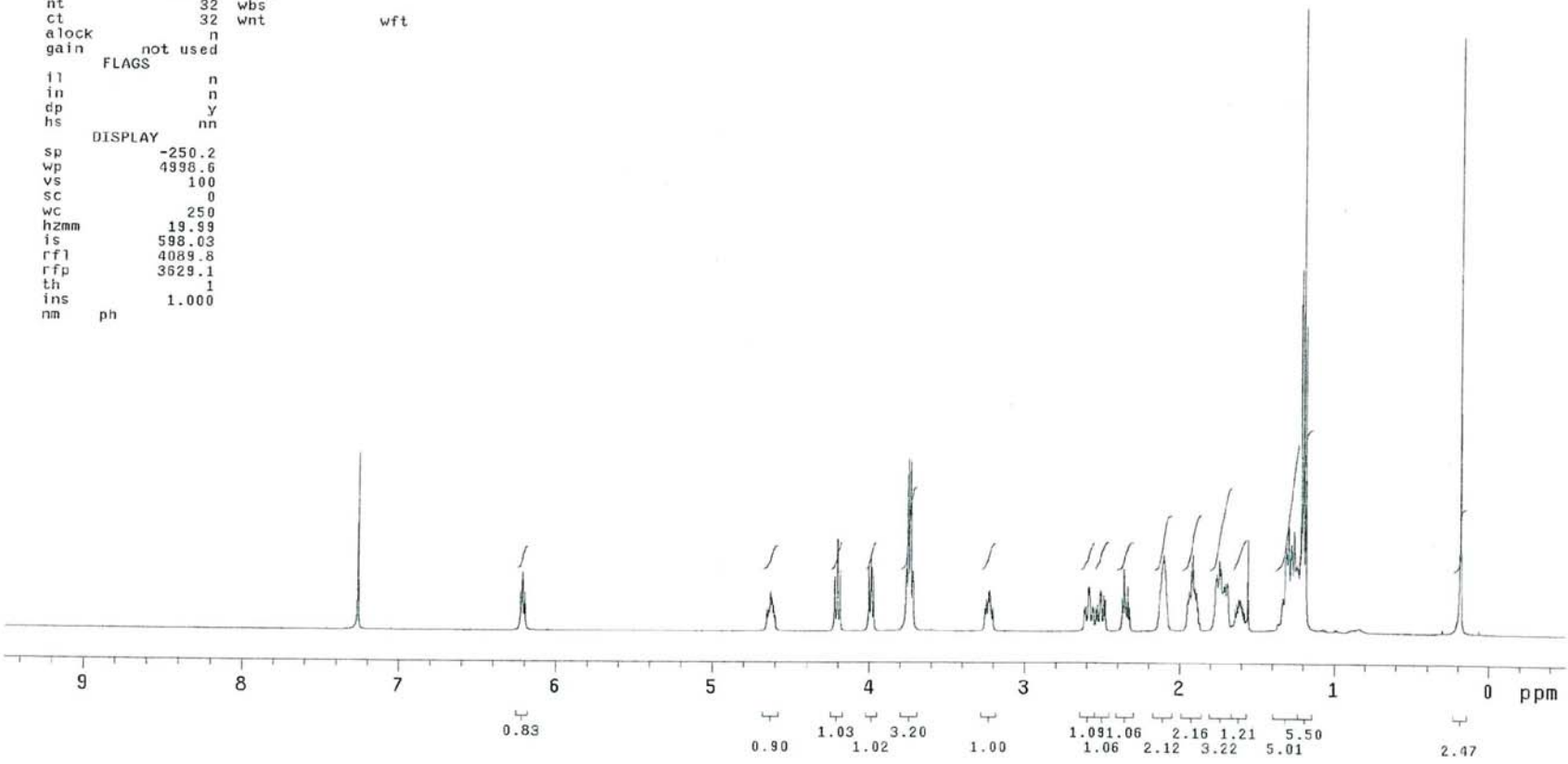
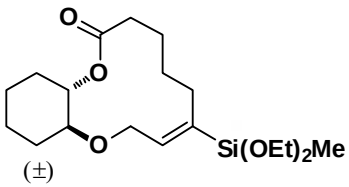
14



WYKELN10032_13C

exp3 s2pul

SAMPLE		DEC. & VT	
date	Apr 30 2010	dfrq	499.874
solvent	CDCl3	dn	H1
file	exp	dpwr	48
ACQUISITION		dof	0
sfrq	125.707	dm	yyy
tn	C13	dmm	w
at	1.092	dmf	10000
np	65536	dseq	
sw	29996.3	dres	1.0
fb	not used	homo	n
bs	32	temp	25.0
tpwr	55	DEC2	
pw	4.2	dfrq2	0
d1	0	dn2	
tof	2000.0	dpwr2	1
nt	99999	dof2	0
ct	1216	dm2	n
alock	n	dmm2	c
gain	not used	dmf2	10000
FLAGS		dseq2	
il	n	dres2	1.0
in	n	homo2	n
dp	y	DEC3	
hs	nn	dfrq3	0
DISPLAY		dn3	
sp	-1090.6	dpwr3	1
wp	29995.3	dof3	0
vs	18	dm3	n
sc	0	dmm3	c
wc	250	dmf3	10000
hzmm	119.98	dseq3	
is	500.00	dres3	1.0
rfl	10769.8	homo3	n
rfp	9678.3	PROCESSING	
th	4	lb	1.00
ins	100.000	wtfile	
nm cdc ph		proc	ft
		fn	not used
		math	f
		werr	
		wexp	
		wbs	
		wnt	



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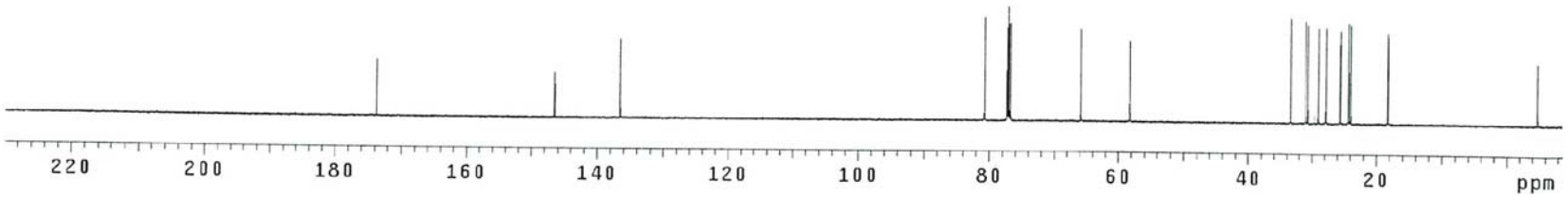
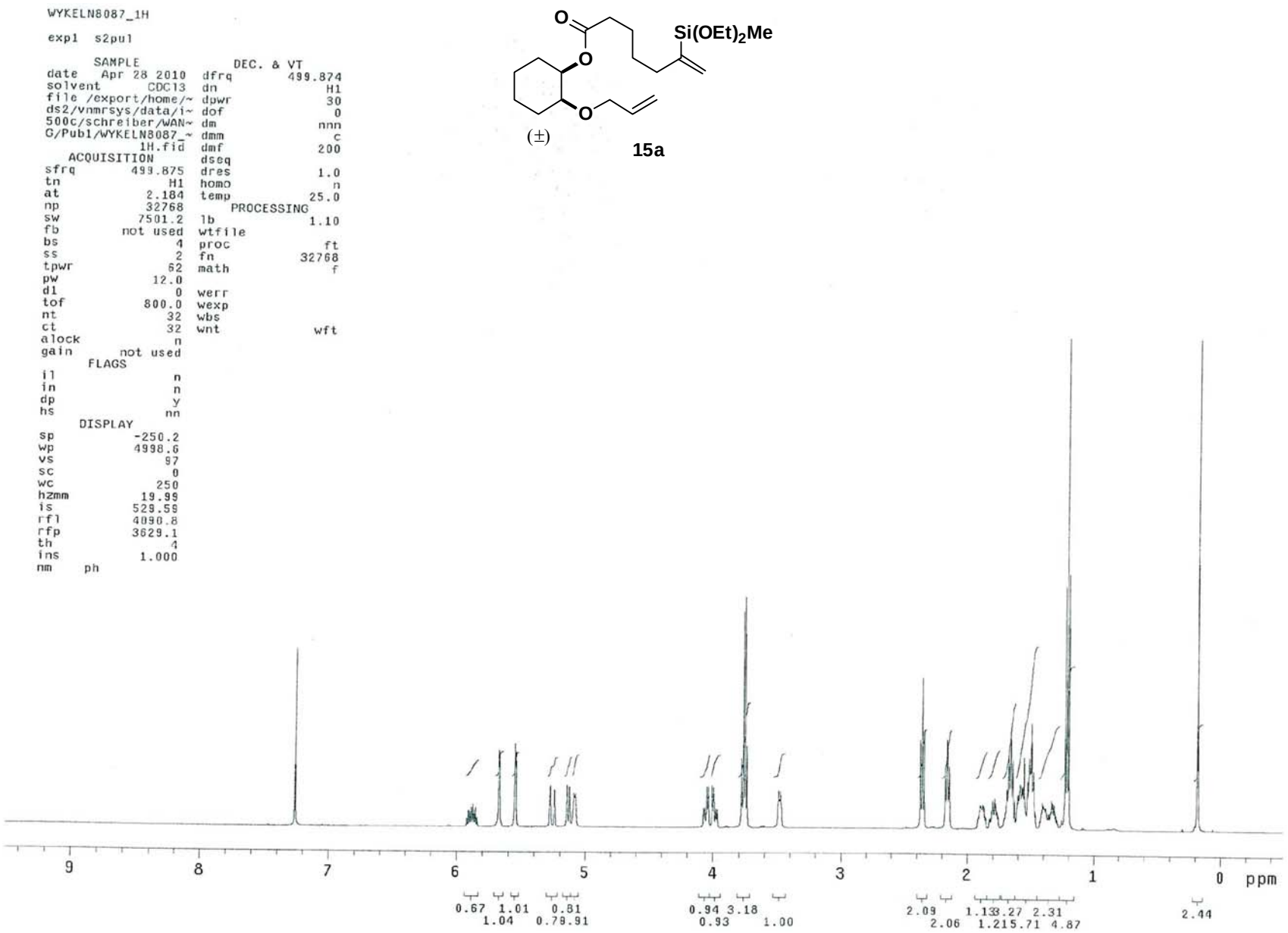
14

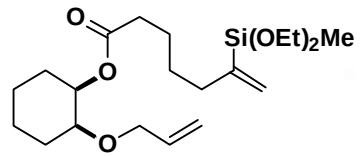



WYKELN8087_1H
expl s2pul
SAMPLE
date Apr 28 2010
solvent CDCl3
file /export/home/~
ds2/vnmrsys/data/i~
500c/schreiber/WAN~
G/Pub1/WYKELN8087_~
1H.fid
ACQUISITION
sfrq 499.875
tn H1
at 2.184
np 32768
sw 7501.2
fb not used
bs 4
ss 2
tpwr 52
pw 12.0
d1 0
tof 800.0
nt 32
ct 32
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -250.2
wp 4998.6
vs 97
sc 0
wc 250
hzmm 19.99
is 529.59
rfl 4090.8
rfp 3629.1
th 4
ins 1.000
nm ph
DEC. & VT
dfrq 499.874
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
temp 25.0
PROCESSING
lb 1.10
wtfile
proc ft
fn 32768
math f
werr
wexp
wbs
wnt wft
O
O
O
Si(OEt)2Me
(±)
15a
9
8
7
6
5
4
3
2
1
0
ppm
0.67
1.01
0.81
1.04
0.79
0.91
0.94
3.18
0.93
1.00
2.09
1.13
3.27
2.31
2.06
1.21
5.71
4.87
2.44

WYKELN8087_13C

exp3 s2pul

SAMPLE		DEC. & VT	
date	Apr 28 2010	dfrq	499.874
solvent	CDCl3	dn	H1
file	exp	dpwr	48
ACQUISITION		dof	0
sfrq	125.707	dm	yyy
tn	C13	dmm	w
at	1.092	dmf	10000
np	65536	dseq	
sw	29996.3	dres	1.0
fb	not used	homo	n
bs	32	temp	25.0
tpwr	55	DEC2	
pw	4.2	dfrq2	0
d1	0	dn2	
tof	2000.0	dpwr2	1
nt	99999	dof2	0
ct	896	dm2	n
alock	n	dmm2	c
gain	not used	dmf2	10000
FLAGS		dseq2	
il	n	dres2	1.0
in	n	homo2	n
dp	y	DEC3	
hs	nn	dfrq3	0
DISPLAY		dn3	
sp	-1087.8	dpwr3	1
wp	29995.3	dof3	0
vs	37	dm3	n
sc	0	dmm3	c
wc	250	dmf3	10000
hzmm	119.98	dseq3	
is	500.00	dres3	1.0
rfl	1088.7	homo3	n
rfp	0	PROCESSING	
th	2	lb	1.00
ins	100.000	wtfile	
nm cdc ph		proc	ft
		fn	not used
		math	f
		werr	
		wexp	
		wbs	
		wnt	



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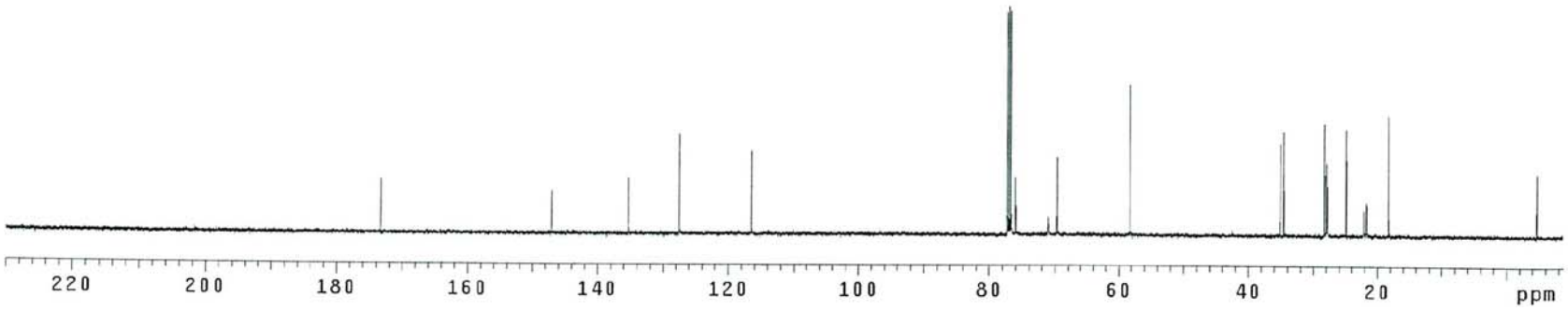
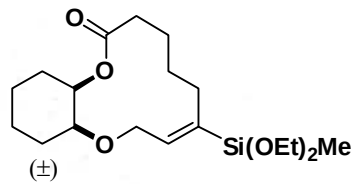
15a



WYKELN10030_1H

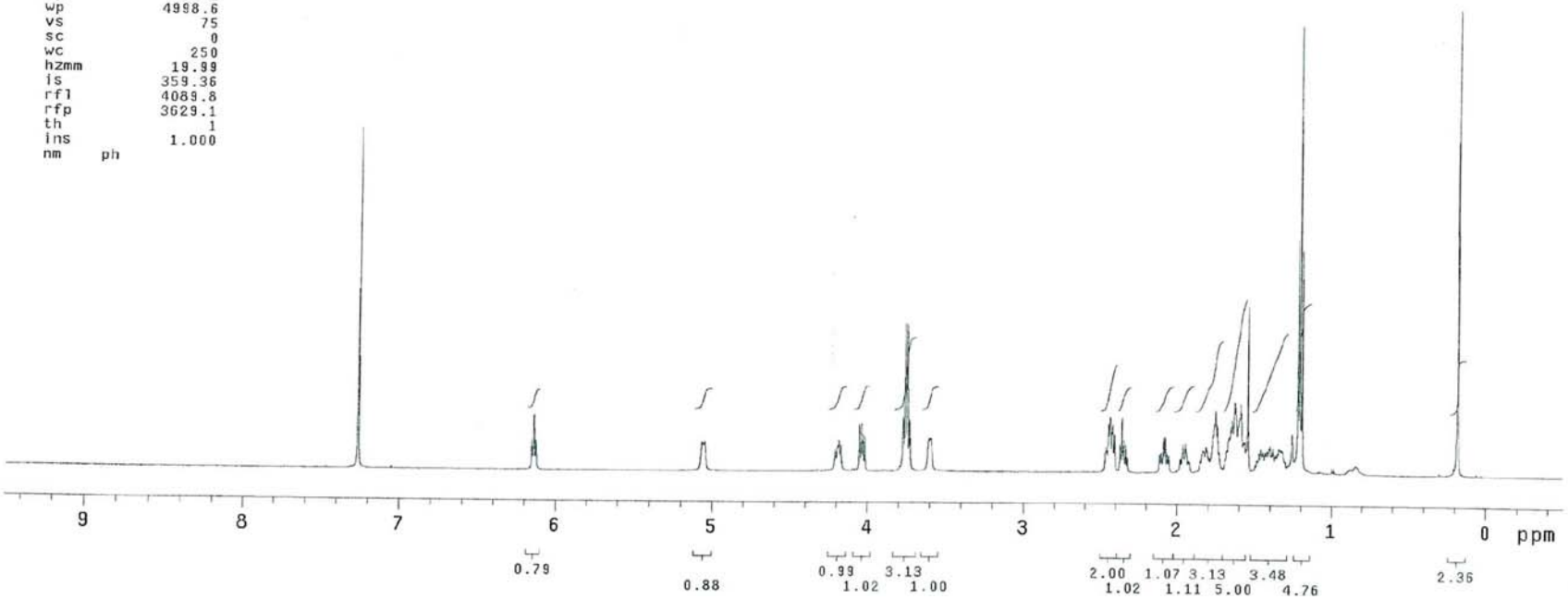
expl s2pul

SAMPLE		DEC. & VT	
date	Apr 30 2010	dfrq	499.874
solvent	CDCl3	dn	H1
file	/export/home/~	dpwr	30
	ds2/vnmrsys/data/i~	dof	0
	500c/schreiber/WAN~	dm	nnn
	G/Pub1/WYKELN10030~	dmm	c
	1H.fid	dmf	200
ACQUISITION		dseq	
sfrq	499.875	dres	1.0
tn	H1	homo	n
at	2.184	temp	25.0
np	32768	PROCESSING	
sw	7501.2	lb	1.10
fb	not used	wtfile	
bs	4	proc	ft
ss	2	fn	32768
tpwr	62	math	f
pw	12.0		
d1	0	werr	
tof	800.0	wexp	
nt	32	wbs	
ct	32	wnt	wft
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-250.2		
wp	4998.6		
vs	75		
sc	0		
wc	250		
hzmm	19.99		
is	359.36		
rfl	4089.8		
rfp	3629.1		
th	1		
ins	1.000		
nm	ph		



(±)

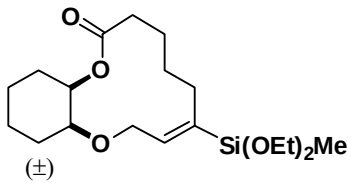
15



WYKELN10030_13C

exp3 s2pul

SAMPLE		DEC. & VT	
date	Apr 30 2010	dfrq	499.874
solvent	CDCl3	dn	H1
file	exp	dpwr	48
ACQUISITION		dof	0
sfrq	125.707	dm	yyy
tn	C13	dmm	w
at	1.092	dmf	10000
np	65536	dseq	
sw	29996.3	dres	1.0
fb	not used	homo	n
bs	32	temp	25.0
tpwr	55	DEC2	
pw	4.2	dfrq2	0
d1	0	dn2	
tof	2000.0	dpwr2	1
nt	99999	dof2	0
ct	1632	dm2	n
alock	n	dmm2	c
gain	not used	dmf2	10000
FLAGS		dseq2	
il	n	dres2	1.0
in	n	homo2	n
dp	y	DEC3	
hs	nn	dfrq3	0
DISPLAY		dn3	
sp	-1086.9	dpwr3	1
wp	29995.3	dof3	0
vs	97	dm3	n
sc	0	dmm3	c
wc	250	dmf3	10000
hzmm	119.98	dseq3	
is	500.00	dres3	1.0
rfl	10766.2	homo3	n
rfp	9678.3	PROCESSING	
th	2	lb	1.00
ins	100.000	wtfile	
nm cdc ph		proc	ft
		fn	not used
		math	f
		werr	
		wexp	
		wbs	
		wnt	



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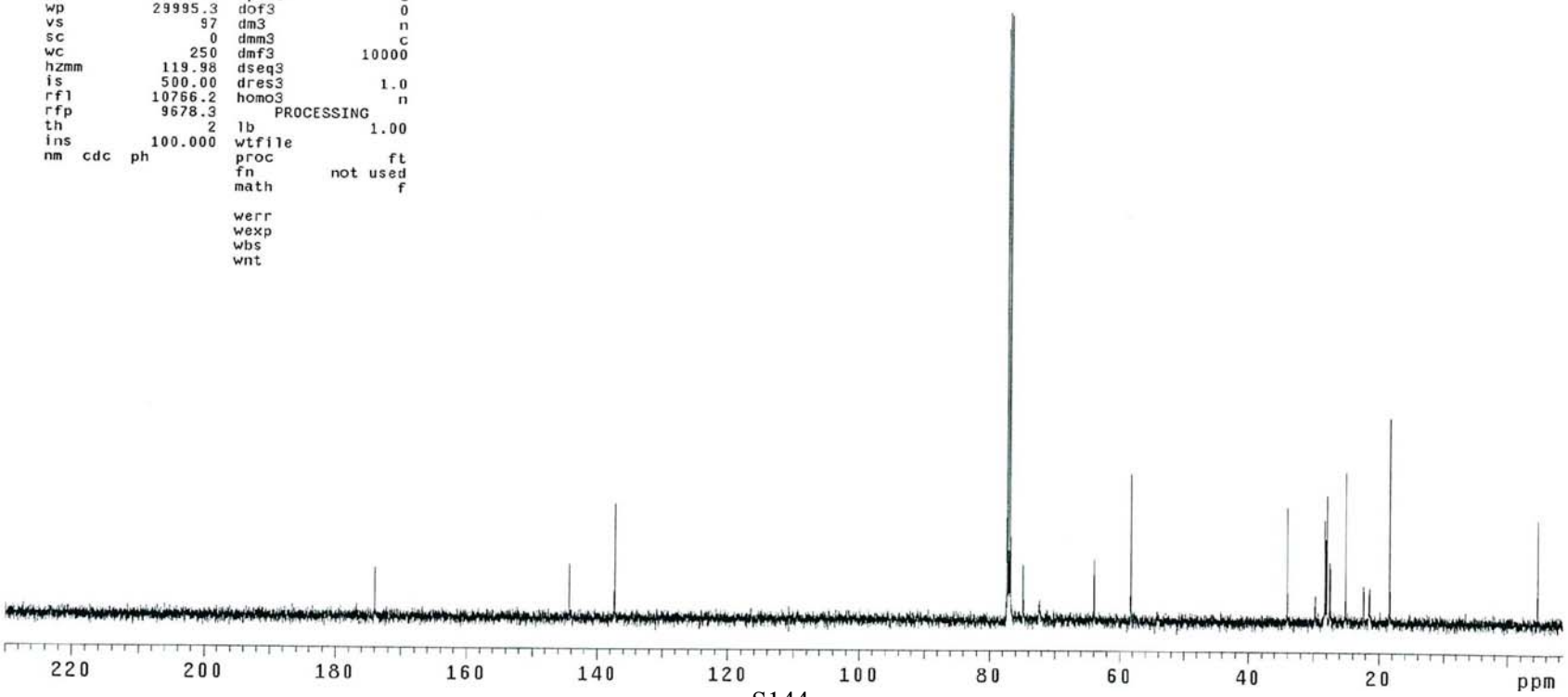
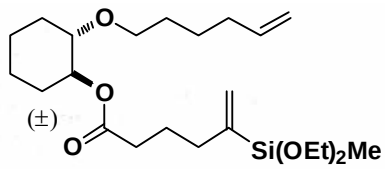
15



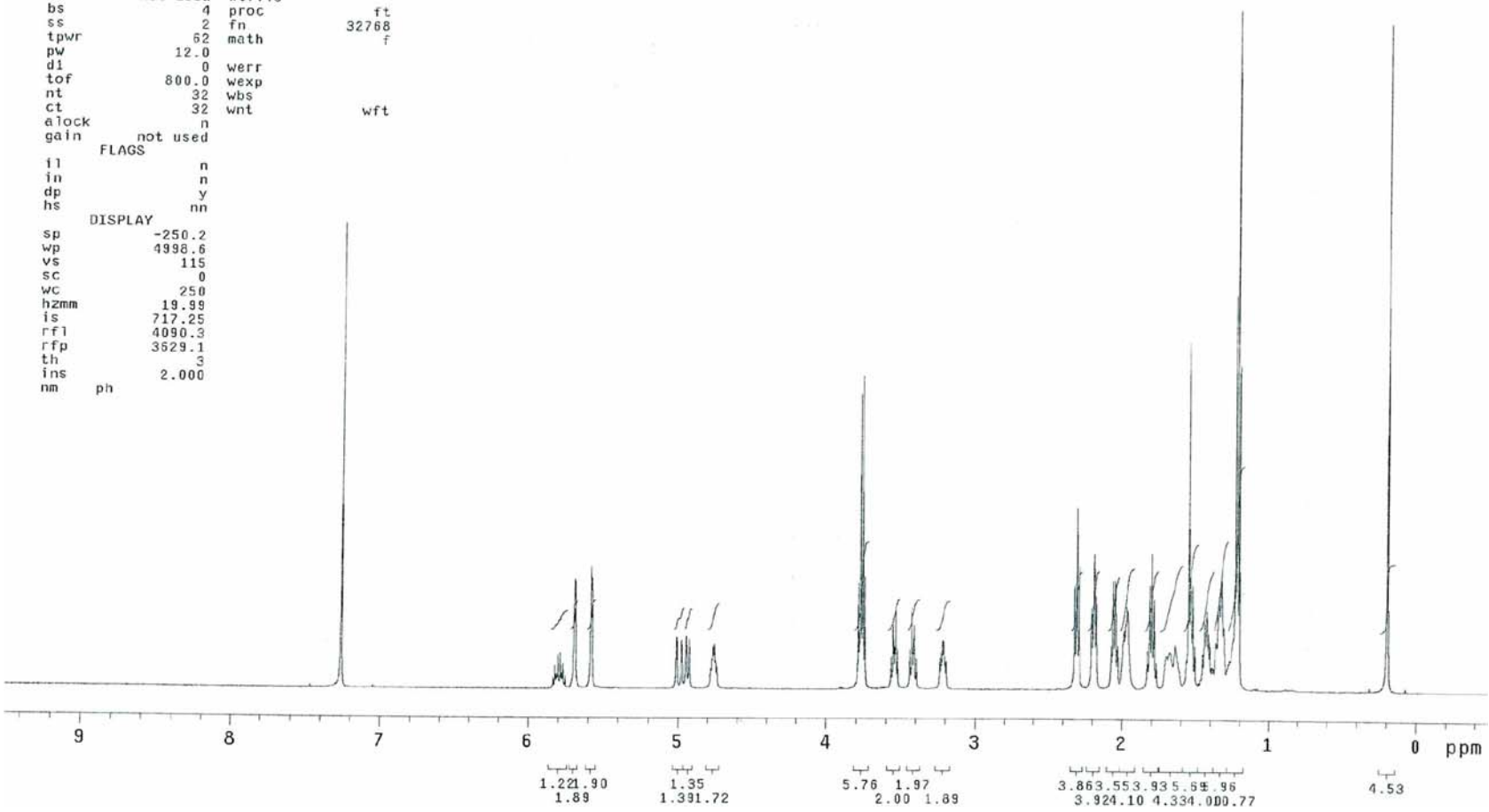
WYKELN8083_1H

expl s2pul

SAMPLE		DEC. & VT	
date	Apr 25 2010	dfrq	499.874
solvent	CDCl3	dn	H1
file	/export/home/~	dpwr	30
	ds2/vnmrsys/data/i~	dof	0
	500c/schreiber/WAN~	dm	nnn
	G/Pub1/WYKELN8083_~	dmm	c
	1H.fid	dmf	200
ACQUISITION		dseq	
sfrq	499.875	dres	1.0
tn	H1	homo	n
at	2.184	temp	25.0
np	32768	PROCESSING	
sw	7501.2	lb	1.10
fb	not used	wtfile	
bs	4	proc	ft
ss	2	fn	32768
tpwr	62	math	f
pw	12.0		
d1	0	werr	
tof	800.0	wexp	
nt	32	wbs	
ct	32	wnt	wft
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-250.2		
wp	4998.6		
vs	115		
sc	0		
wc	250		
hzmm	19.99		
is	717.25		
rfl	4090.3		
rfp	3529.1		
th	3		
ins	2.000		
nm	ph		



16a

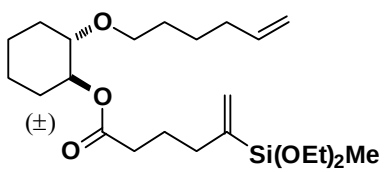


WYKELN8083_13C

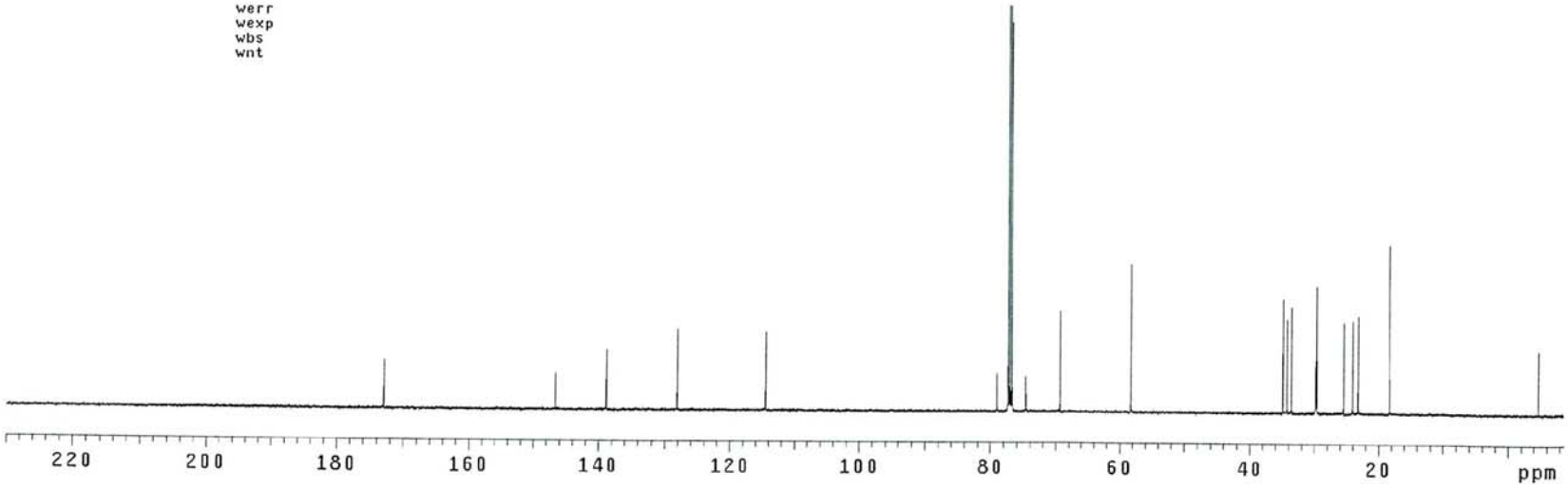
exp2 s2pul

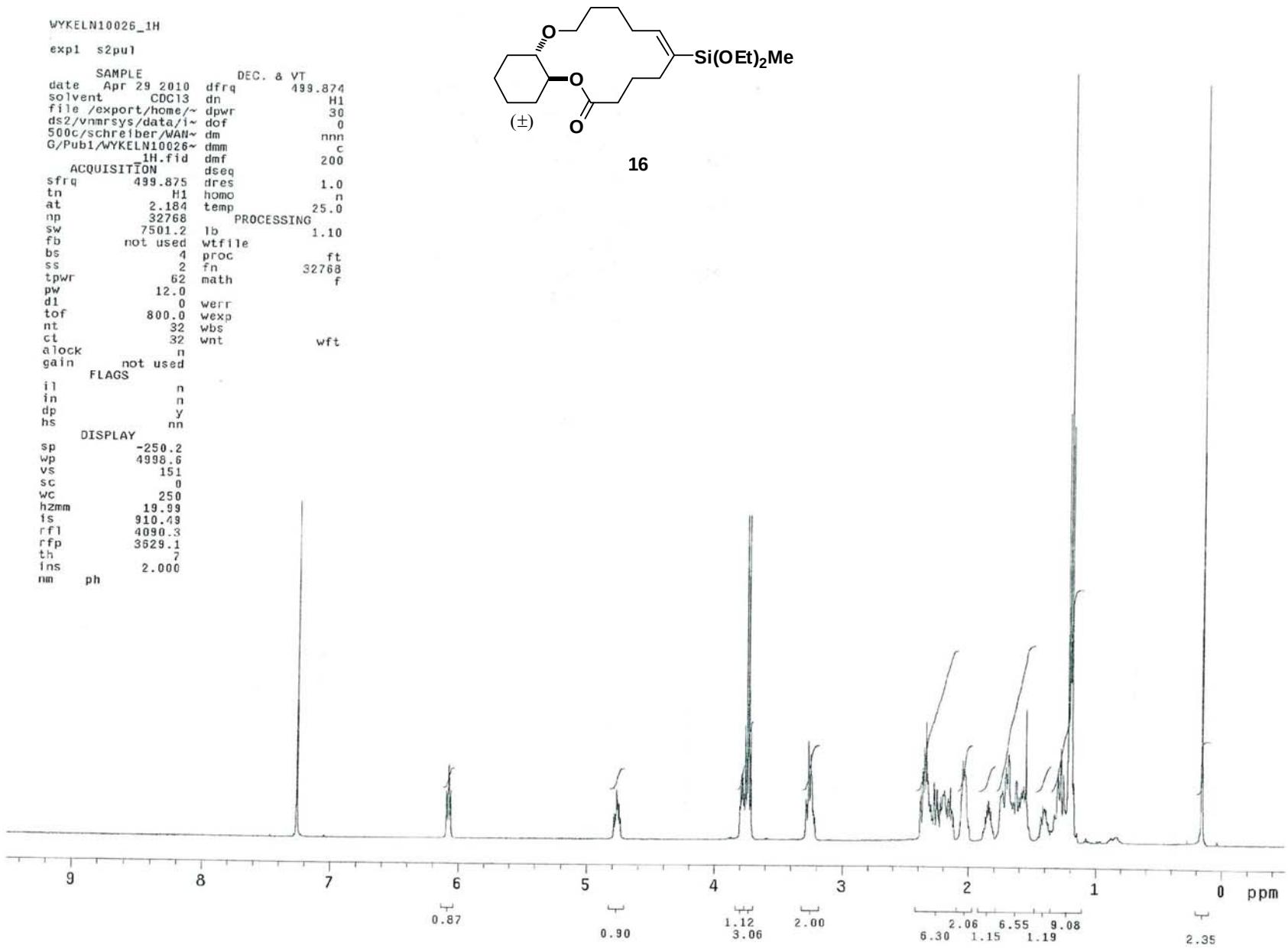
SAMPLE	
date	Apr 25 2010
solvent	CDCl3
file	exp
ACQUISITION	
sfrq	125.707
tn	C13
at	1.092
np	65536
sw	29996.3
fb	not used
bs	16
tpwr	55
pw	2.0
d1	0
tof	2000.0
nt	9999
ct	2848
alock	n
gain	not used
FLAGS	
il	n
in	n
dp	y
hs	nn
DISPLAY	
sp	-1087.8
wp	29995.3
vs	65
sc	0
wc	250
hzmm	119.98
is	500.00
rfl	10767.1
rfp	9678.3
th	2
ins	100.000
nm	cdc ph

DEC. & VT	
dfrq	499.874
dn	H1
dpwr	48
dof	0
dm	yyy
dmm	w
dmf	10000
dseq	
dres	1.0
homo	n
temp	25.0
DEC2	
dfrq2	0
dn2	
dpwr2	1
dof2	0
dm2	n
dmm2	c
dmf2	10000
dseq2	
dres2	1.0
homo2	n
DEC3	
dfrq3	0
dn3	
dpwr3	1
dof3	0
dm3	n
dmm3	c
dmf3	10000
dseq3	
dres3	1.0
homo3	n
PROCESSING	
lb	1.00
wtfile	
proc	ft
fn	not used
math	f
werr	
wexp	
wbs	
wnt	



16a

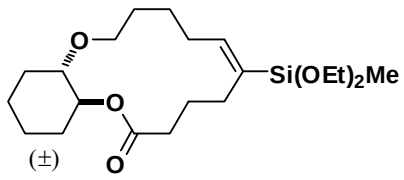



WYKELN10026_1H
expl s2pul
SAMPLE
date Apr 29 2010
solvent CDCl3
file /export/home/~
ds2/vnmrsys/data/i~
500c/schreiber/WAN~
G/Pub1/WYKELN10026~
1H.fid
ACQUISITION
sfrq 499.875
tn H1
at 2.184
np 32768
sw 7501.2
fb not used
bs 4
ss 2
tpwr 62
pw 12.0
d1 0
tof 800.0
nt 32
ct 32
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -250.2
wp 4998.6
vs 151
sc 0
wc 250
hzmm 19.99
is 910.49
rfl 4090.3
rfp 3629.1
th 7
ins 2.000
nm ph
DEC. & VT
dfrq 499.874
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
temp 25.0
PROCESSING
lb 1.10
wtfile
proc ft
fn 32768
math f
werr
wexp
wbs
wnt wft
O
O
O
Si(OEt)2Me
(±)
16
9
8
7
6
5
4
3
2
1
0 ppm
0.87
0.90
1.12
3.06
2.00
6.30
2.06
1.15
6.55
1.19
9.08
2.35

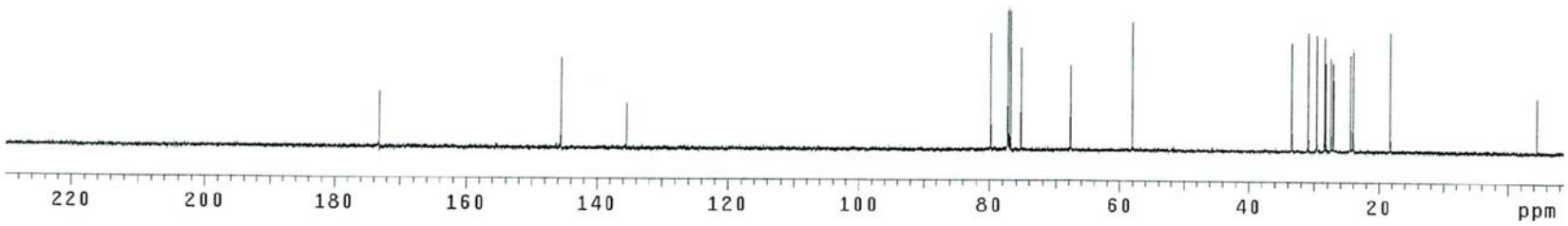
WYKELN10026_13C

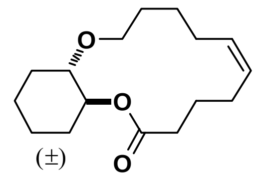
exp2 s2pul

SAMPLE		DEC. & VT	
date	Apr 29 2010	dfrq	499.874
solvent	CDCl3	dn	H1
file	exp	dpwr	48
ACQUISITION		dof	0
sfrq	125.707	dm	yyy
tn	C13	dmm	w
at	1.092	dmf	10000
np	65536	dseq	
sw	29996.3	dres	1.0
fb	not used	homo	n
bs	32	temp	25.0
tpwr	55	DEC2	
pw	4.2	dfrq2	0
d1	0	dn2	
tof	2000.0	dpwr2	1
nt	99999	dof2	0
ct	256	dm2	n
alock	n	dmm2	c
gain	not used	dmf2	10000
FLAGS		dseq2	
il	n	dres2	1.0
in	n	homo2	n
dp	y	DEC3	
hs	nn	dfrq3	0
DISPLAY		dn3	
sp	-1089.7	dpwr3	1
wp	29995.3	dof3	0
vs	23	dm3	n
sc	0	dmm3	c
wc	250	dmf3	10000
hzmm	119.98	dseq3	
is	500.00	dres3	1.0
rfl	10768.9	homo3	n
rfp	9678.3	PROCESSING	
th	5	lb	1.00
ins	100.000	wtfile	
nm cdc ph		proc	ft
		fn	not used
		math	f
		werr	
		wexp	
		wbs	
		wnt	



16

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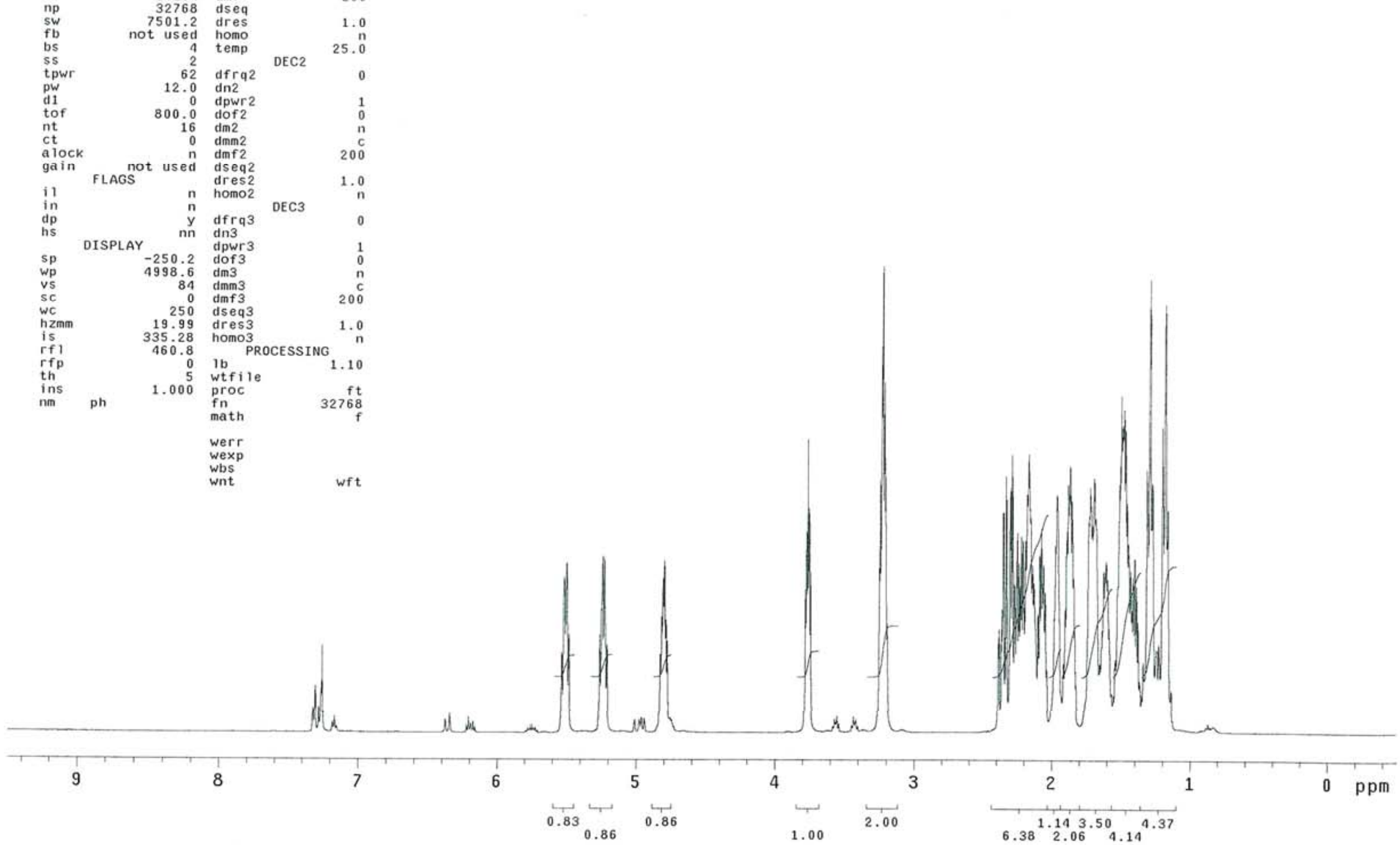
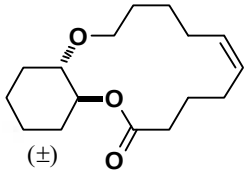

WYKELN19026_1H
exp1 s2pul
SAMPLE
date Apr 30 2011
solvent CDCl3
file exp
ACQUISITION
sfrq 499.875
tn H1
at 2.184
np 32768
sw 7501.2
fb not used
bs 4
ss 2
tpwr 62
pw 12.0
d1 0
tof 800.0
nt 16
ct 0
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -250.2
wp 4998.6
vs 84
sc 0
wc 250
hzmm 19.99
is 335.28
rfl 460.8
rfp 0
th 5
ins 1.000
nm ph
DEC. & VT
dfrq 499.874
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
temp 25.0
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 200
dseq2
dres2 1.0
homo2 n
DEC3
dfrq3 0
dn3
dpwr3 1
dof3 0
dm3 n
dmm3 c
dmf3 200
dseq3
dres3 1.0
homo3 n
PROCESSING
lb 1.10
wtfile
proc ft
fn 32768
math f
werr
wexp
wbs
wnt wft
9 8 7 6 5 4 3 2 1 0 ppm
0.83 0.86 0.86 1.00 2.00 6.38 1.14 2.06 3.50 4.14 4.37

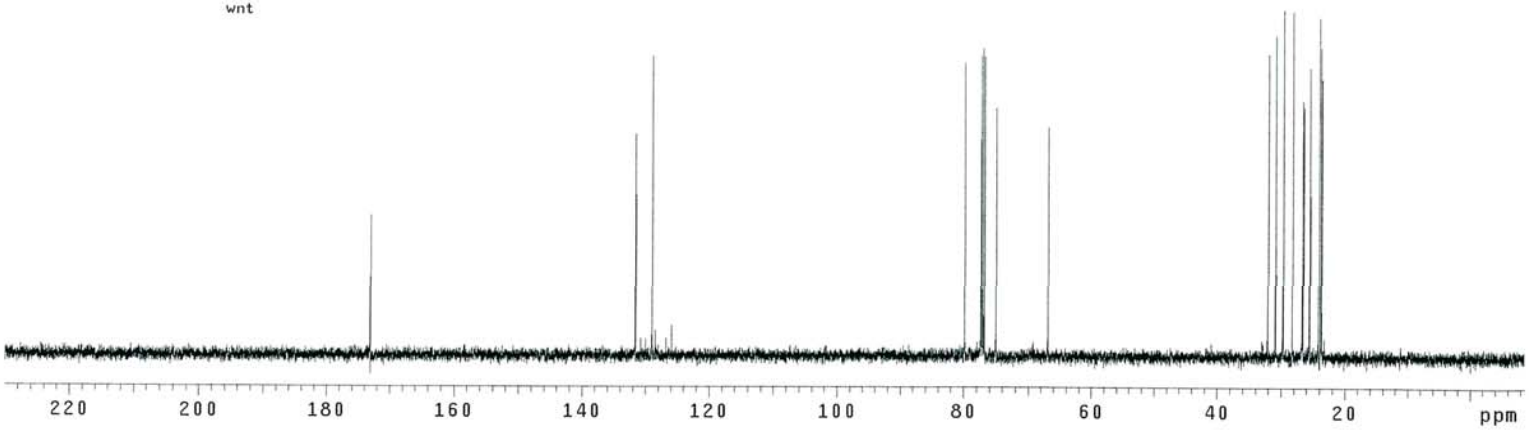
WYKELN19026_13C

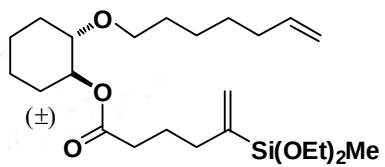
exp2 s2pul

SAMPLE		DEC. & VT	
date	Apr 30 2011	dfrq	499.874
solvent	CDCl3	dn	H1
file	exp	dpwr	48
ACQUISITION		dof	0
sfrq	125.707	dm	yyy
tn	C13	dmm	w
at	1.092	dmf	8929
np	65536	dseq	
sw	29996.3	dres	1.0
fb	not used	homo	n
bs	16	temp	25.0
tpwr	55	DEC2	
pw	4.8	dfrq2	0
d1	0	dn2	
tof	2000.0	dpwr2	1
nt	9999	dof2	0
ct	0	dm2	n
alock	n	dmm2	c
gain	not used	dmf2	10000
FLAGS		dseq2	
il	n	dres2	1.0
in	n	homo2	n
dp	y	DEC3	
hs	nn	dfrq3	0
DISPLAY		dn3	
sp	-1090.6	dpwr3	1
wp	29995.3	dof3	0
vs	57	dm3	n
sc	0	dmm3	c
wc	250	dmf3	10000
hzmm	119.98	dseq3	
is	500.00	dres3	1.0
rfl	10769.8	homo3	n
rfp	9678.3	PROCESSING	
th	15	lb	1.00
ins	100.000	wtfile	
nm cdc ph		proc	ft
		fn	not used
		math	f
		werr	
		wexp	
		wbs	
		wnt	



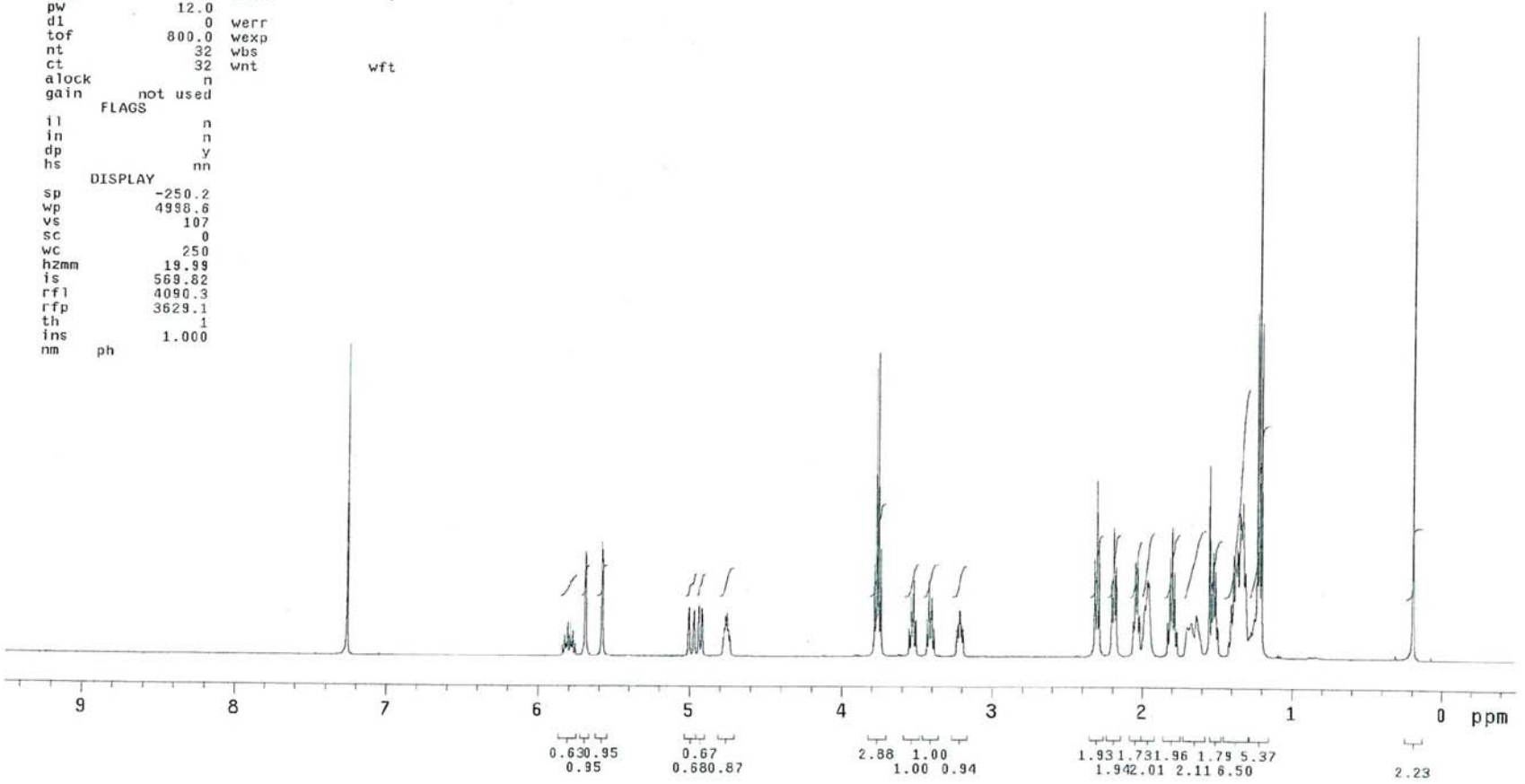
16b





17a

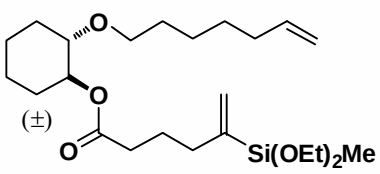

```
WYKELN8084_1H

expl  s2pul

      SAMPLE
date    Apr 25 2010
solvent       CDCl3
file /export/home/~
ds2/vnmrsys/data/i~
500c/schreiber/WAN~
G/Pub1/WYKELN8084_~
              1H.fid
   ACQUISITION
sfrq         499.875
tn               H1
at             2.184
np             32768
sw            7501.2
fb         not used
bs                 4
ss                 2
tpwr              62
pw              12.0
d1                 0
tof            800.0
nt                32
ct                32
alock              n
gain       not used
      FLAGS
il                 n
in                 n
dp                 y
hs                nn
     DISPLAY
sp            -250.2
wp            4998.6
vs               107
sc                 0
wc               250
hzmm           19.99
is            569.82
rfl           4090.3
rfp           3629.1
th                 1
ins            1.000
nm    ph

          DEC. & VT
dfrq          499.874
dn                 H1
dpwr               30
dof                 0
dm                nnn
dmm                 c
dmf               200
dseq
dres              1.0
homo                n
temp             25.0
     PROCESSING
lb                1.10
wtfile
proc                ft
fn               32768
math                f

werr
wexp
wbs
wnt               wft
```

WYKELN8084_13C

exp2 s2pul

SAMPLE		DEC. & VT	
date	Apr 25 2010	dfrq	499.874
solvent	CDCl3	dn	H1
file	exp	dpwr	48
ACQUISITION		dof	0
sfrq	125.707	dm	yyy
tn	C13	dmm	w
at	1.092	dmf	10000
np	65536	dseq	
sw	29996.3	dres	1.0
fb	not used	homo	n
bs	16	temp	25.0
tpwr	55	DEC2	
pw	2.0	dfrq2	0
d1	0	dn2	
tof	2000.0	dpwr2	1
nt	9999	dof2	0
ct	7248	dm2	n
alock	n	dmm2	c
gain	not used	dmf2	10000
FLAGS		dseq2	
il	n	dres2	1.0
in	n	homo2	n
dp	y	DEC3	
hs	nn	dfrq3	0
DISPLAY		dn3	
sp	-1087.8	dpwr3	1
wp	29995.3	dof3	0
vs	64	dm3	n
sc	0	dmm3	c
wc	250	dmf3	10000
hzmm	119.98	dseq3	
is	500.00	dres3	1.0
rfl	1088.7	homo3	n
rfp	0	PROCESSING	
th	4	lb	1.00
ins	100.000	wtfile	
nm cdc ph		proc	ft
		fn	not used
		math	f
		werr	
		wexp	
		wbs	
		wnt	



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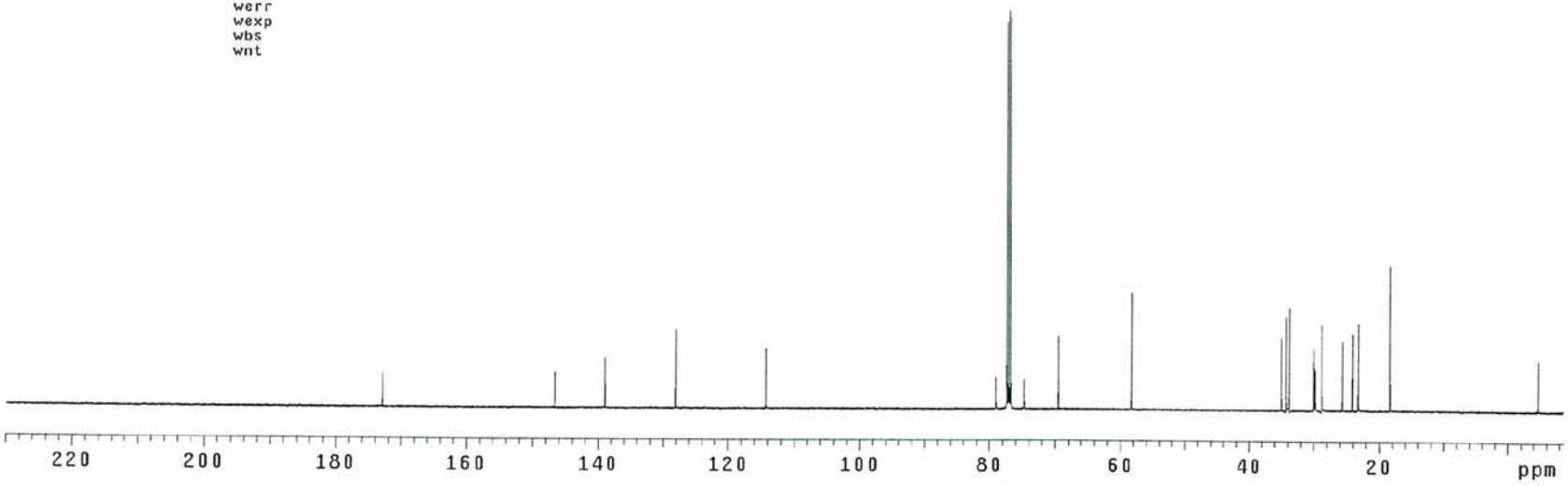
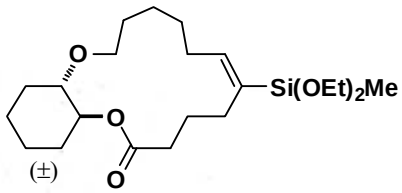


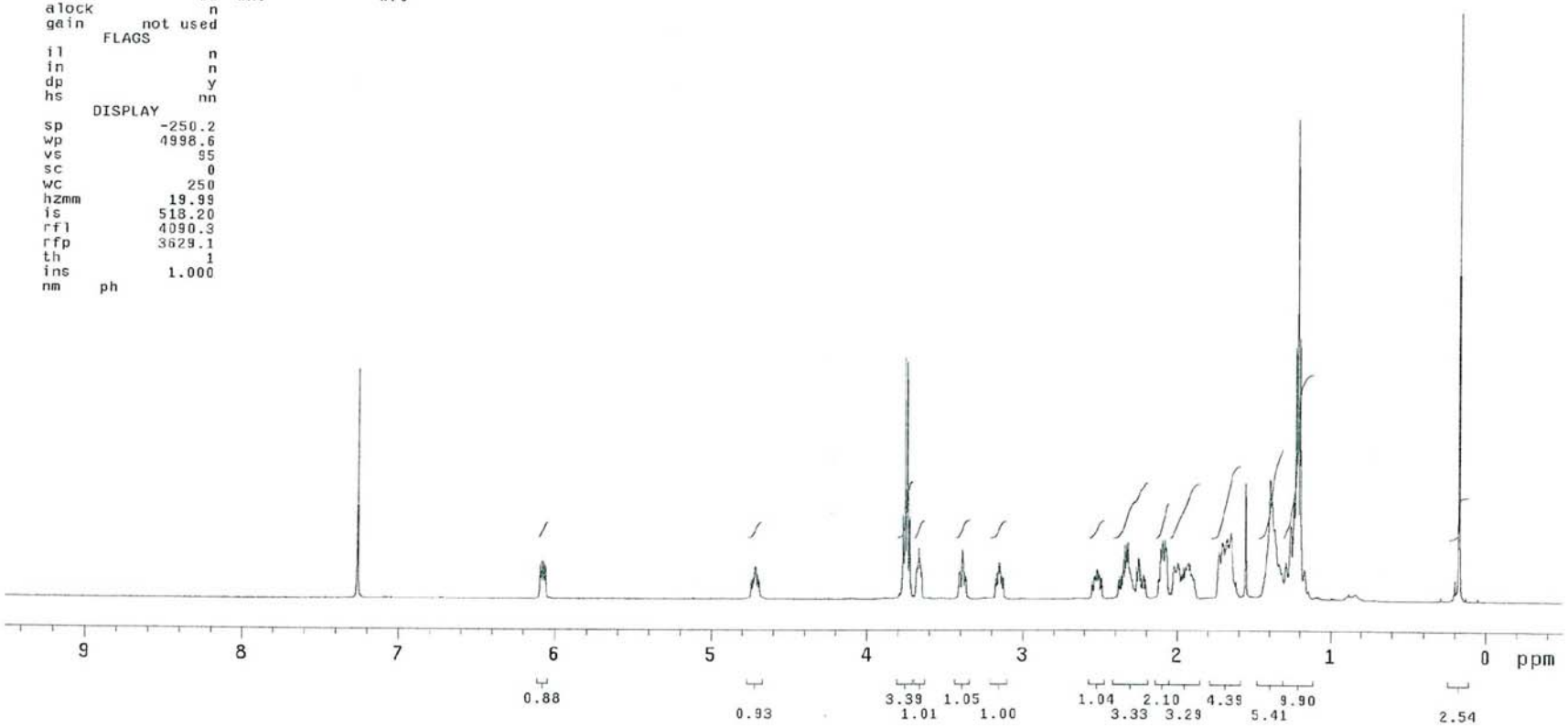
WYKELN10027_1H

exp1 s2pul

SAMPLE		DEC. & VT	
date	Apr 28 2010	dfrq	499.874
solvent	CDCl3	dn	H1
file	/export/home/~	dpwr	30
	ds2/vnmrsys/data/i~	dof	0
	500c/schreiber/WAN~	dm	nnn
	G/Pub1/WYKELN10027~	dmm	c
	1H.fid	dmf	200
ACQUISITION		dseq	
sfrq	499.875	dres	1.0
tn	H1	homo	n
at	2.184	temp	25.0
np	32768	PROCESSING	
sw	7501.2	lb	1.10
fb	not used	wtfile	
bs	4	proc	ft
ss	2	fn	32768
tpwr	62	math	f
pw	12.0		
d1	0	werr	
tof	800.0	wexp	
nt	32	wbs	
ct	32	wnt	wft
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-250.2		
wp	4998.6		
vs	95		
sc	0		
wc	250		
hzmm	19.99		
is	518.20		
rfl	4090.3		
rfp	3629.1		
th	1		
ins	1.000		
nm	ph		



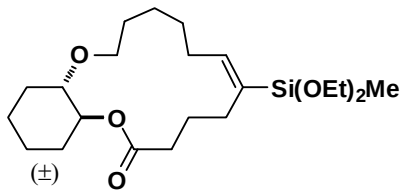
17



WYKELN10027_13C

exp3 s2pul

SAMPLE		DEC. & VT	
date	Apr 28 2010	dfrq	499.874
solvent	CDC13	dn	H1
file	exp	dpwr	48
ACQUISITION		dof	0
sfrq	125.707	dm	yyy
tn	C13	dmm	w
at	1.092	dmf	10000
np	65536	dseq	
sw	29996.3	dres	1.0
fb	not used	homo	n
bs	32	temp	25.0
tpwr	55	DEC2	
pw	4.2	dfrq2	0
d1	0	dn2	
tof	2000.0	dpwr2	1
nt	99999	dof2	0
ct	928	dm2	n
alock	n	dmm2	c
gain	not used	dmf2	10000
FLAGS		dseq2	
il	n	dres2	1.0
in	n	homo2	n
dp	y	DEC3	
hs	nn	dfrq3	0
DISPLAY		dn3	
sp	-1086.9	dpwr3	1
wp	29995.3	dof3	0
vs	58	dm3	n
sc	0	dmm3	c
wc	250	dmf3	10000
hzmm	119.98	dseq3	
is	500.00	dres3	1.0
rfl	10766.2	homo3	n
rfp	9678.3	PROCESSING	
th	5	lb	1.00
ins	100.000	wtfile	
nm cdc ph		proc	ft
		fn	not used
		math	f
		werr	
		wexp	
		wbs	
		wnt	



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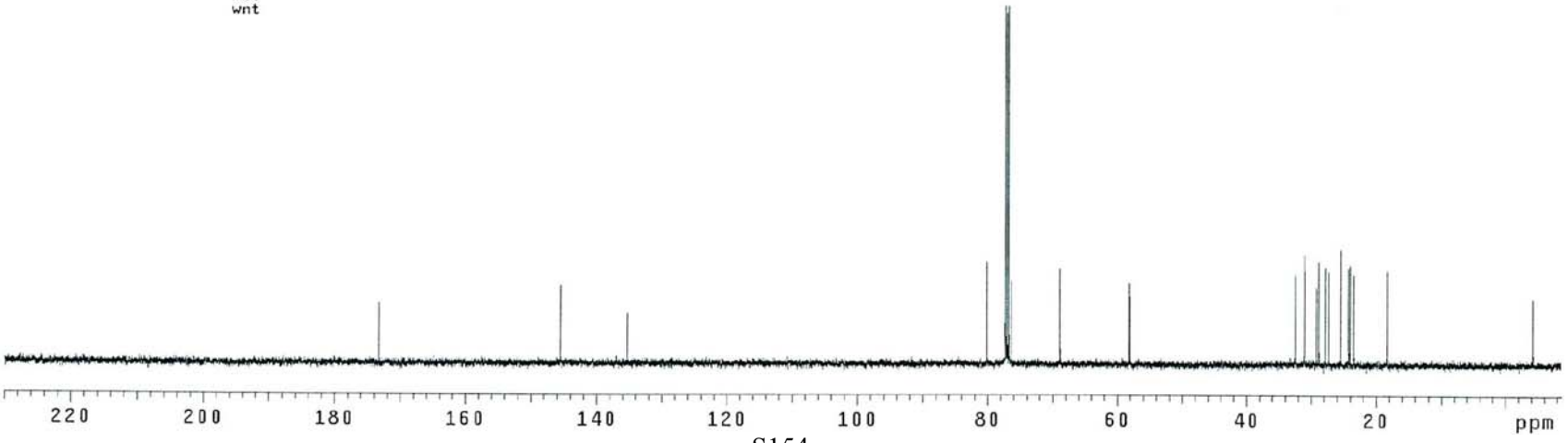
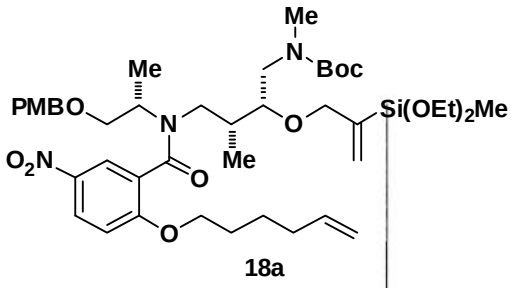


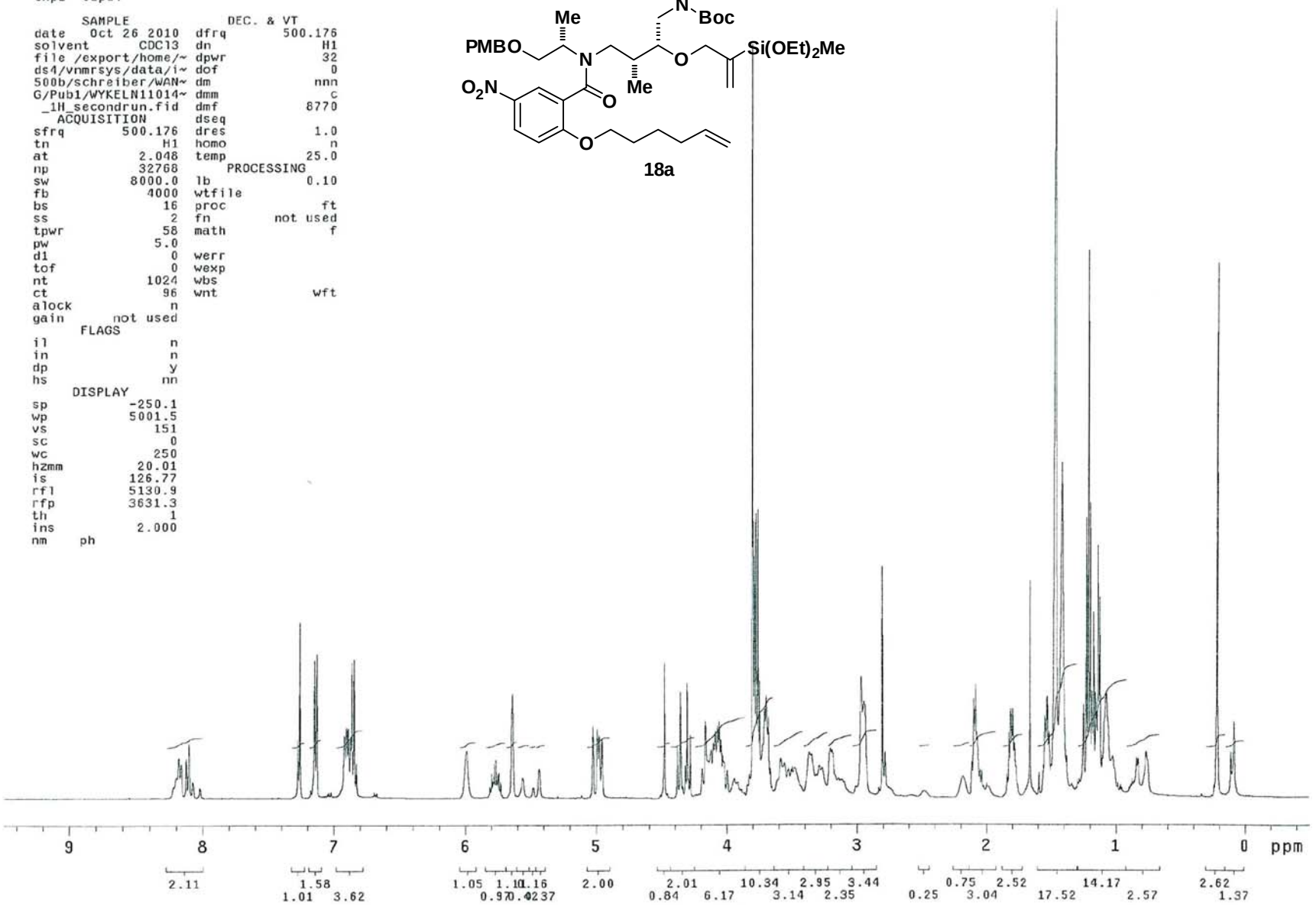
WYKELN11014_1H

exp2 s2pul

SAMPLE		DEC. & VT	
date	Oct 26 2010	dfrq	500.176
solvent	CDCl3	dn	H1
file	/export/home/~	dpwr	32
	ds4/vnmrsys/data/i~	dof	0
	500b/schreiber/WAN~	dm	nnn
	G/Pub1/WYKELN11014~	dmm	c
	_1H_secondrun.fid	dmf	8770
ACQUISITION		dseq	
sfrq	500.176	dres	1.0
tn	H1	homo	n
at	2.048	temp	25.0
np	32768	PROCESSING	
sw	8000.0	lb	0.10
fb	4000	wtfile	
bs	16	proc	ft
ss	2	fn	not used
tpwr	58	math	f
pw	5.0		
d1	0	werr	
tof	0	wexp	
nt	1024	wbs	
ct	96	wnt	wft
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-250.1		
wp	5001.5		
vs	151		
sc	0		
wc	250		
hzmm	20.01		
is	126.77		
rfl	5130.9		
rfp	3631.3		
th	1		
ins	2.000		
nm	ph		



18a

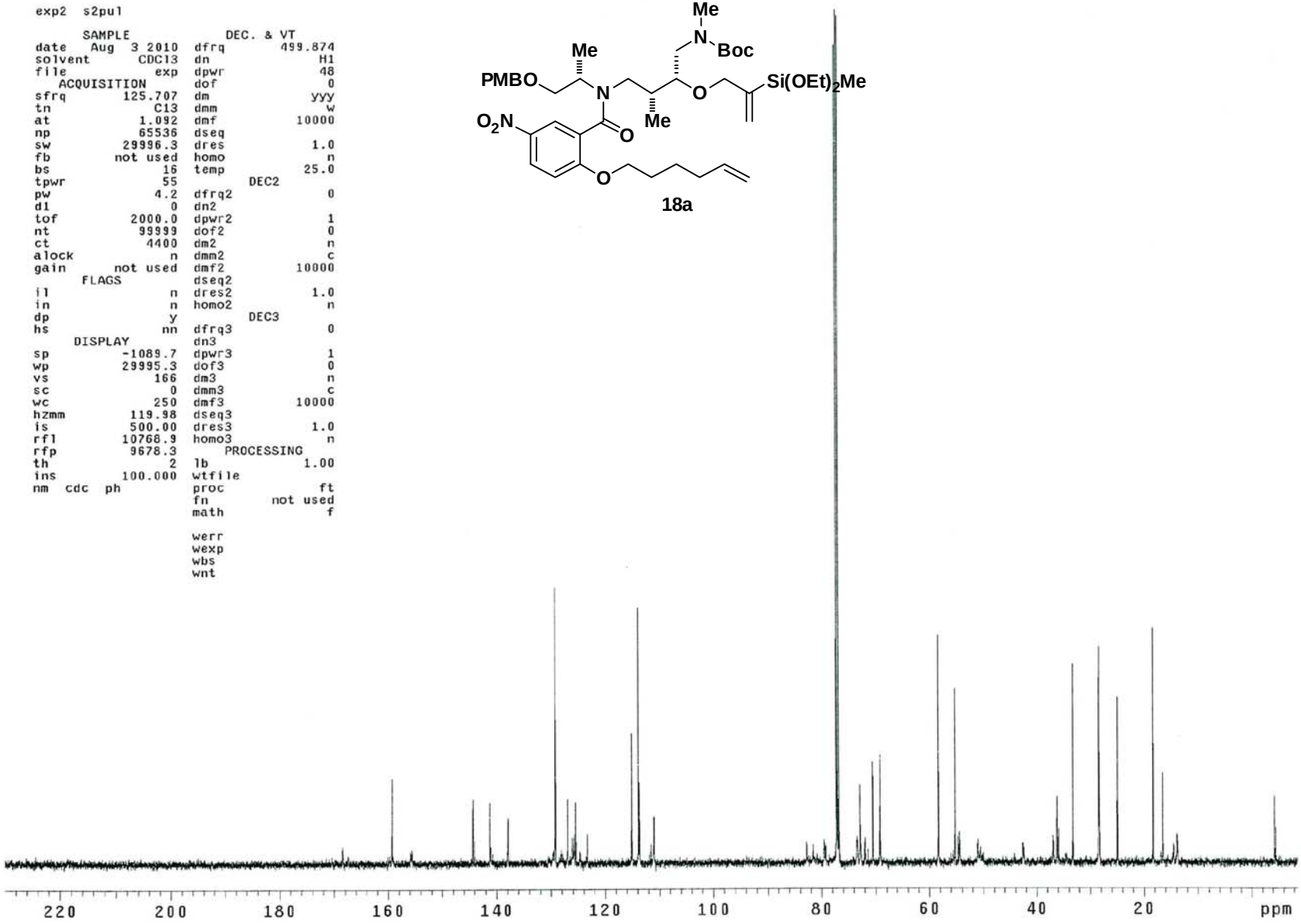


WYKELN11014_13C

exp2 s2pul

SAMPLE		DEC. & VT	
date	Aug 3 2010	dfrq	499.874
solvent	CDCl3	dn	H1
file	exp	dpwr	48
ACQUISITION		dof	0
sfrq	125.707	dm	yyy
tn	C13	dmm	w
at	1.092	dmf	10000
np	65536	dseq	
sw	29996.3	dres	1.0
fb	not used	homo	n
bs	16	temp	25.0
tpwr	55	DEC2	
pw	4.2	dfrq2	0
d1	0	dn2	
tof	2000.0	dpwr2	1
nt	99999	dof2	0
ct	4400	dm2	n
alock	n	dmm2	c
gain	not used	dmf2	10000
FLAGS		dseq2	
il	n	dres2	1.0
in	n	homo2	n
dp	y	DEC3	
hs	nn	dfrq3	0
DISPLAY		dn3	
sp	-1089.7	dpwr3	1
wp	29995.3	dof3	0
vs	166	dm3	n
sc	0	dmm3	c
wc	250	dmf3	10000
hzmm	119.98	dseq3	
is	500.00	dres3	1.0
rfl	10768.9	homo3	n
rfp	9678.3	PROCESSING	
th	2	lb	1.00
ins	100.000	wtfile	
nm cdc ph		proc	ft
		fn	not used
		math	f
		werr	
		wexp	
		wbs	
		wnt	

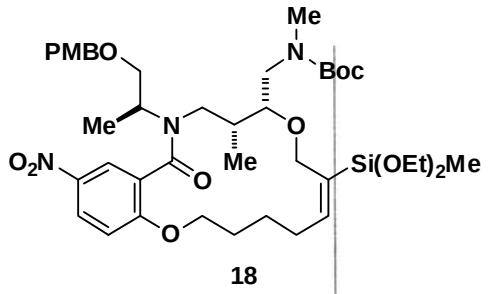
18a



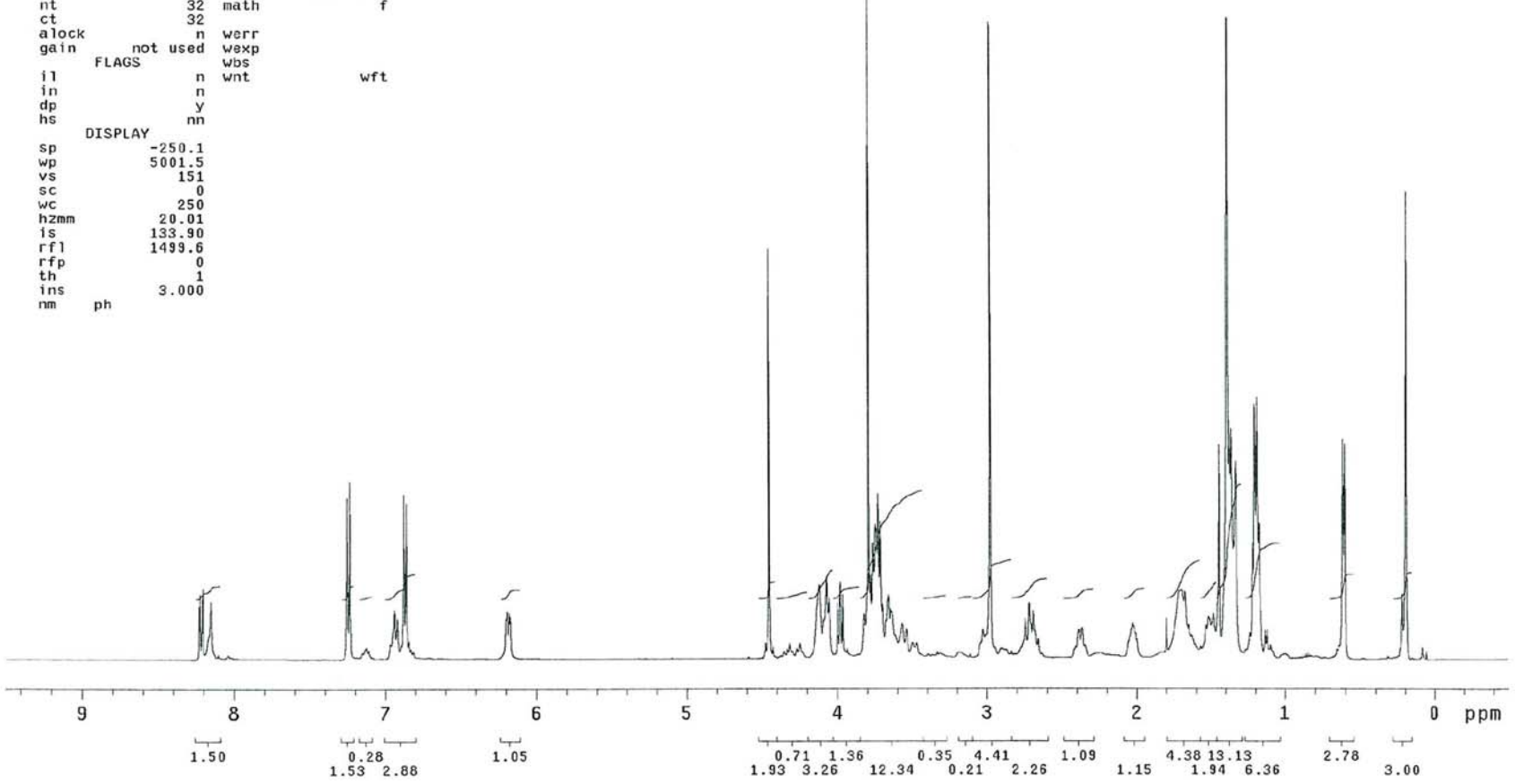
WYKELN11017_1H

exp5 s2pul

SAMPLE		DEC. & VT	
date	Oct 27 2010	dfrq	500.176
solvent	CDCl3	dn	H1
file	exp	dpwr	32
ACQUISITION		dof	0
sfrq	500.176	dm	nnn
tn	H1	dmm	c
at	2.048	dmf	8770
np	32768	dseq	
sw	8000.0	dres	1.0
fb	4000	homo	n
bs	4	temp	25.0
ss	2	PROCESSING	
tpwr	58	lb	0.10
pw	5.0	wtfile	
d1	0	proc	ft
tof	0	fn	not used
nt	32	math	f
ct	32		
alock	n	werr	
gain	not used	wexp	
FLAGS		wbs	
il	n	wnt	wft
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-250.1		
wp	5001.5		
vs	151		
sc	0		
wc	250		
hzmm	20.01		
is	133.90		
rfl	1499.6		
rfp	0		
th	1		
ins	3.000		
nm	ph		



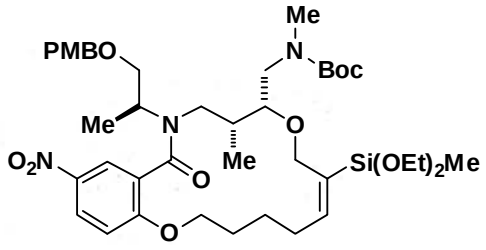
18



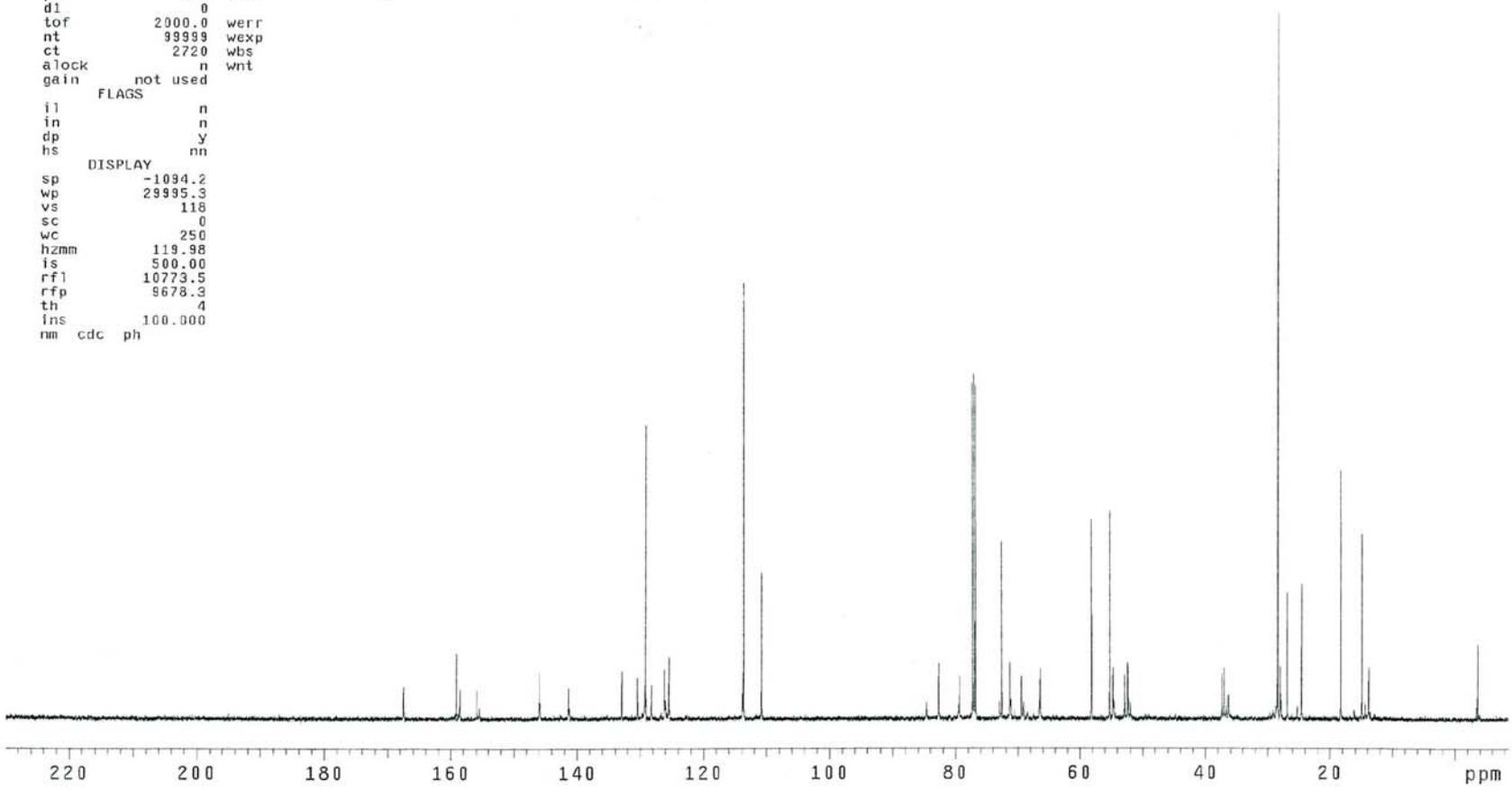
WYKELN11017_13C

exp1 s2pul

SAMPLE		DEC. & VT	
date	Oct 28 2010	dfrq	499.874
solvent	CDC13	dn	H1
file	/export/home/~	dpwr	48
ds2/vnmrsys/data/i~		dof	0
500c/schreiber/WAN~		dm	yyy
G/Pub1/WYKELN11017~		dmm	w
	13C.fid	dmf	9180
ACQUISITION		dseq	
sfrq	125.707	dres	1.0
tn	C13	homo	n
at	1.092	temp	25.0
np	65536	PROCESSING	
sw	29996.3	lb	1.00
fb	not used	wtfile	
bs	16	proc	ft
tpwr	55	fn	not used
pw	4.8	math	f
d1	0		
tof	2000.0	werr	
nt	99999	wexp	
ct	2720	wbs	
alock	n	wnt	
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-1094.2		
wp	29995.3		
vs	118		
sc	0		
wc	250		
hzmm	119.98		
is	500.00		
rfl	10773.5		
rfp	9678.3		
th	4		
ins	100.000		
nm cdc ph			



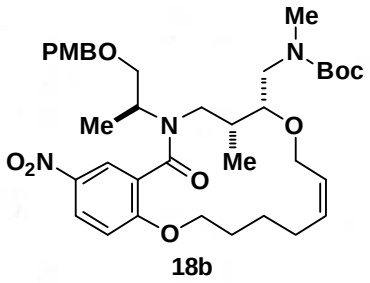
18



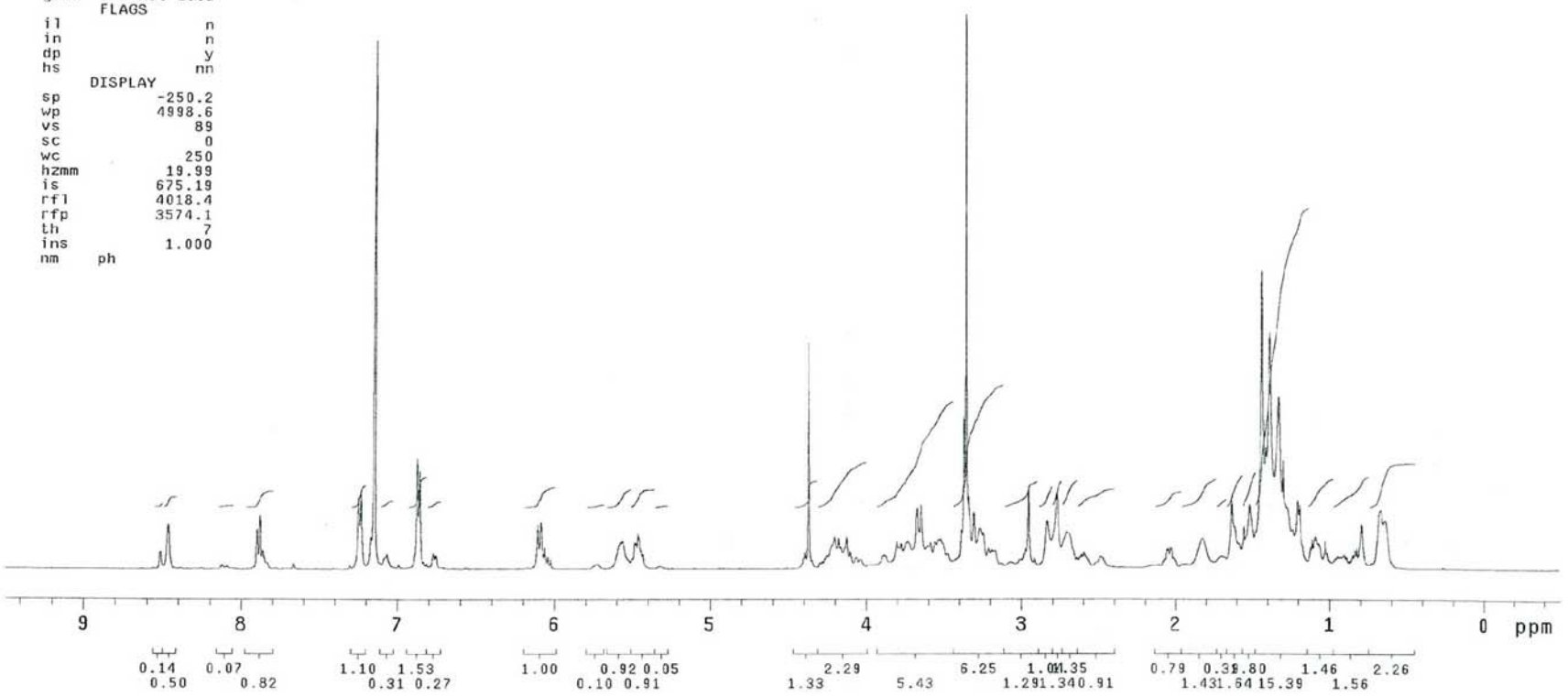
WYKELN11028_1H_Benzene

exp1 s2pul

SAMPLE		DEC. & VT	
date	Aug 6 2010	dfrq	499.874
solvent	Benzene	dn	H1
file	/export/home/~	dpwr	30
	ds2/vnmrsys/data/i~	dof	0
	500c/schreiber/WAN~	dm	nnn
	G/Pub1/WYKELN11028~	dmm	c
	1H.fid	dmf	200
ACQUISITION		dseq	
sfrq	499.875	dres	1.0
tn	H1	homo	n
at	2.184	temp	25.0
np	32768	PROCESSING	
sw	7501.2	lb	1.10
fb	not used	wtfile	
bs	4	proc	ft
ss	2	fn	32768
tpwr	62	math	f
pw	12.0		
d1	0	werr	
tof	800.0	wexp	
nt	32	wbs	
ct	16	wnt	wft
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-250.2		
wp	4998.6		
vs	89		
sc	0		
wc	250		
hzmm	19.99		
is	675.19		
rfl	4018.4		
rfp	3574.1		
th	7		
ins	1.000		
nm	ph		



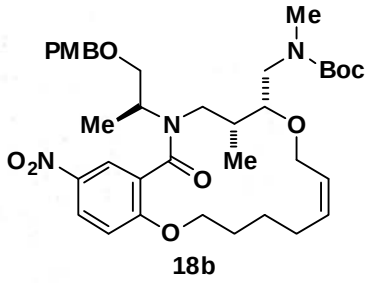
18b



WYKELN11028_1H_C6D6_80C

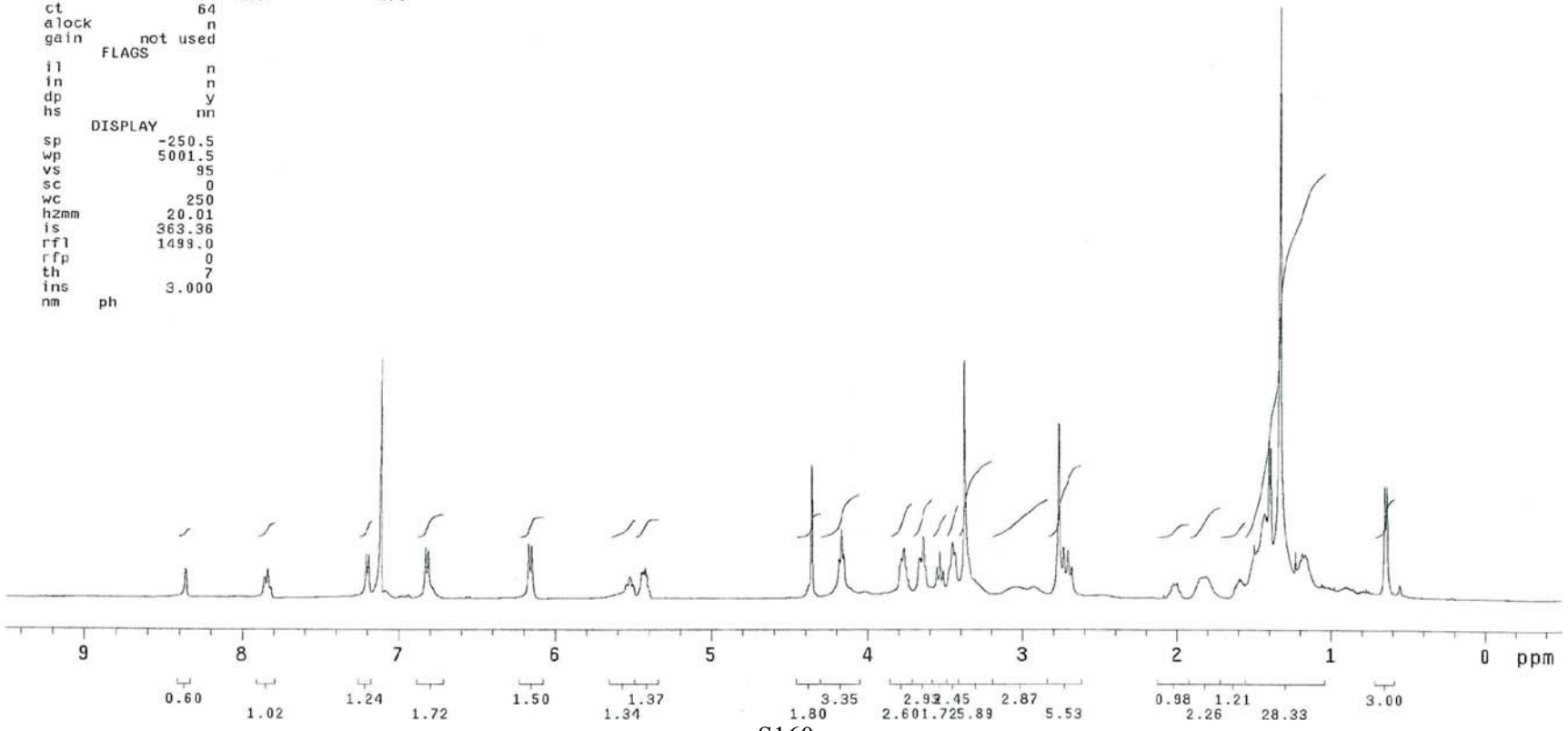
exp1 s2pul

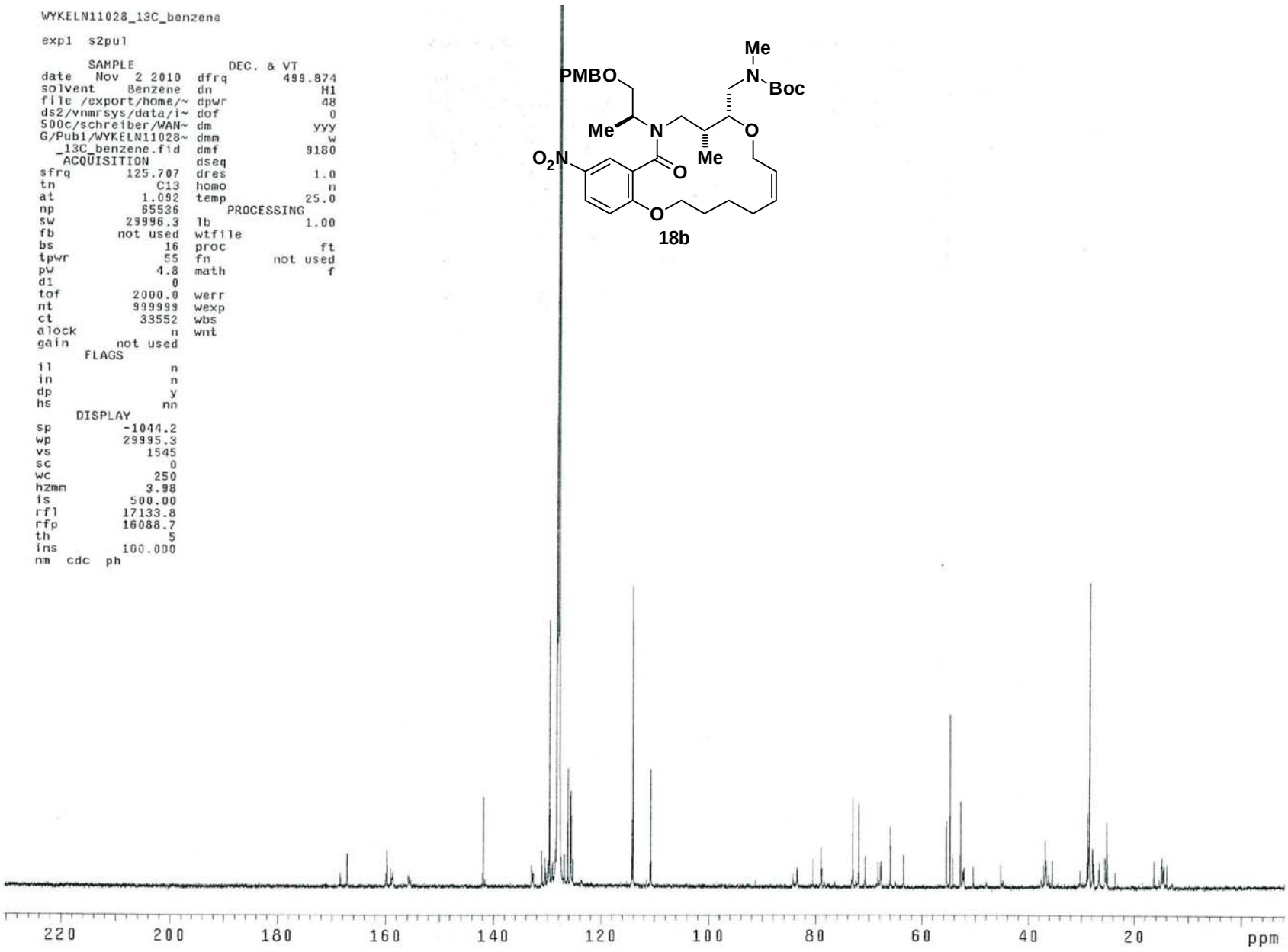
SAMPLE		DEC. & VT	
date	Oct 30 2010	dfrq	500.176
solvent	Benzene	dn	H1
file	/export/home/~	dpwr	32
	ds2/vnmrsys/data/i~	dof	0
	500b/schreiber/WAN~	dm	nnn
	G/Pub1/WYKELN11028~	dmm	c
	fromdesilylation~	dmf	8770
	1H_benzene_80C.fid	dseq	
ACQUISITION		dres	1.0
sfrq	500.176	homo	n
tn	H1	temp	80.0
at	2.048	PROCESSING	
np	32768	lb	0.10
sw	8000.0	wtfile	
fb	4000	proc	ft
bs	4	fn	not used
ss	2	math	f
tpwr	58		
pw	5.0	werr	
d1	0	wexp	
tof	0	wbs	
nt	64	wnt	wft
ct	64		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-250.5		
wp	5001.5		
vs	95		
sc	0		
wc	250		
hzmm	20.01		
is	363.36		
rfl	1499.0		
rfp	0		
th	7		
ins	3.000		
nm	ph		



18b

at 80 °C in C_6D_6

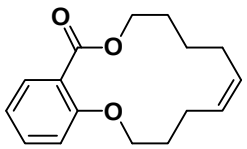



WYKELN11028_13C_benzene
exp1 s2pul
SAMPLE
date Nov 2 2010
solvent Benzene
file /export/home/~
ds2/vnmrsys/data/i~
500c/schreiber/WAN~
G/Pub1/WYKELN11028~
_13C_benzene.fid
ACQUISITION
sfrq 125.707
tn C13
at 1.092
np 65536
sw 29996.3
fb not used
bs 16
tpwr 55
pw 4.8
d1 0
tof 2000.0
nt 999999
ct 33552
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -1044.2
wp 29995.3
vs 1545
sc 0
wc 250
hzmm 3.98
is 500.00
rfl 17133.8
rfp 16088.7
th 5
ins 100.000
nm cdc ph
DEC. & VT
dfrq 499.874
dn H1
dpwr 48
dof 0
dm yyy
dmm w
dmf 9180
dseq
dres 1.0
homo n
temp 25.0
PROCESSING
lb 1.00
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
PMBO
Me
N
Boc
Me
N
O
O2N
Me
O
O
18b
220
200
180
160
140
120
100
80
60
40
20
ppm

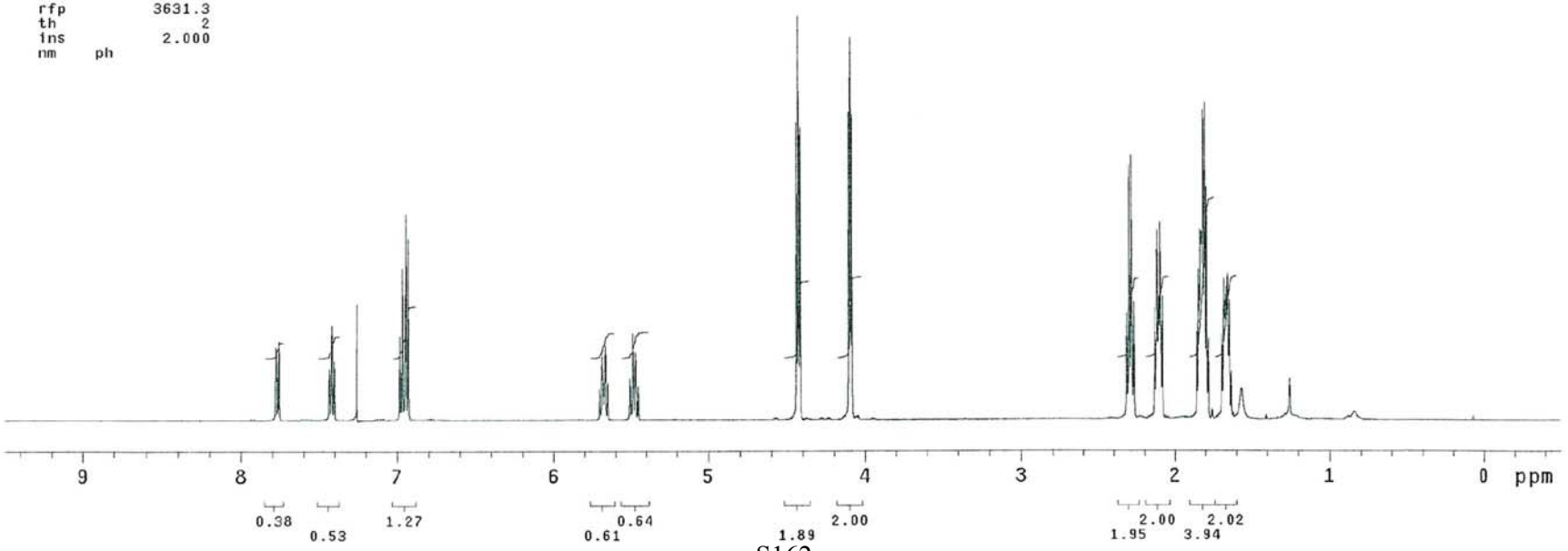
WYKELN10039_1H

expl s2pul

SAMPLE		DEC. & VT	
date	Nov 17 2010	dfrq	500.176
solvent	CDCl3	dn	H1
file	exp	dpwr	32
ACQUISITION		dof	0
sfrq	500.176	dm	nnn
tn	H1	dmm	c
at	2.048	dmf	8770
np	32768	dseq	
sw	8000.0	dres	1.0
fb	4000	homo	n
bs	4	temp	24.0
ss	2	PROCESSING	
tpwr	58	lb	0.10
pw	5.0	wtfile	
d1	0	proc	ft
tof	0	fn	not used
nt	32	math	f
ct	32	werr	
alock	n	wexp	
gain	not used	wbs	
FLAGS		wnt	wft
il	n		
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-250.1		
wp	5001.5		
vs	65		
sc	0		
wc	250		
hzmm	20.01		
is	220.05		
rfl	5127.4		
rfp	3631.3		
th	2		
ins	2.000		
nm	ph		



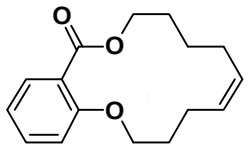
19b



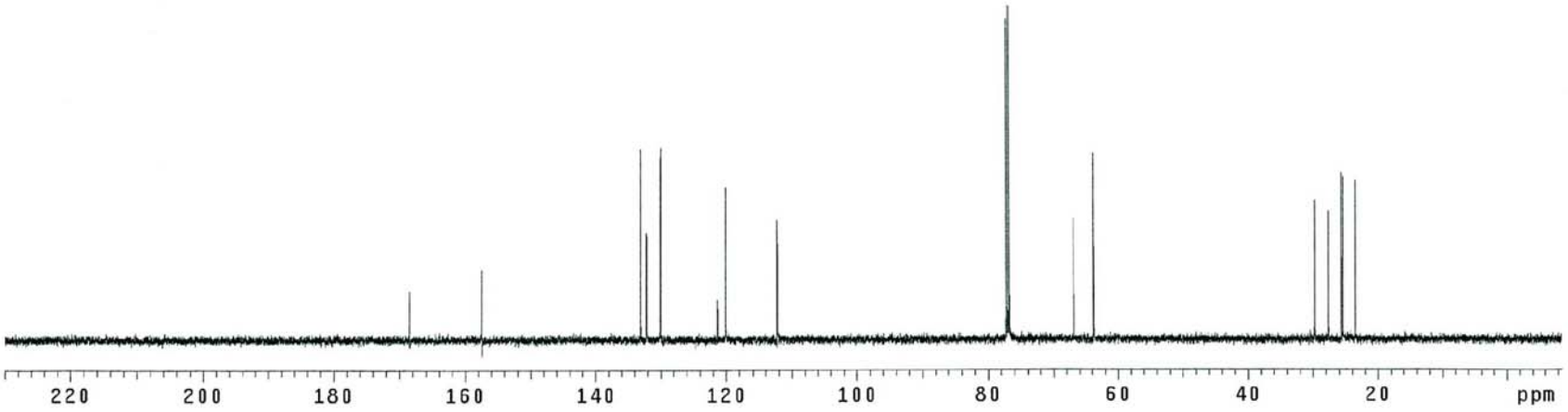
WYKELN10039_13C

exp4 s2pul

SAMPLE		DEC. & VT	
date	Nov 17 2010	dfrq	499.874
solvent	CDCl3	dn	H1
file	exp	dpwr	48
ACQUISITION		dof	0
sfrq	125.707	dm	yyy
tn	C13	dmm	w
at	1.092	dmf	9180
np	65536	dseq	
sw	29996.3	dres	1.0
fb	not used	homo	n
bs	16	temp	25.0
tpwr	55	DEC2	
pw	4.8	dfrq2	0
d1	0	dn2	
tof	2000.0	dpwr2	1
nt	99999	dof2	0
ct	1104	dm2	n
alock	n	dmm2	c
gain	not used	dmf2	10000
FLAGS		dseq2	
il	n	dres2	1.0
in	n	homo2	n
dp	y	DEC3	
hs	nn	dfrq3	0
DISPLAY		dn3	
sp	-1086.9	dpwr3	1
wp	29995.3	dof3	0
vs	54	dm3	n
sc	0	dmm3	c
wc	250	dmf3	10000
hzmm	5.10	dseq3	
is	500.00	dres3	1.0
rfl	10766.2	homo3	n
rfp	9678.3	PROCESSING	
th	3	lb	1.00
ins	100.000	wtfile	
nm cdc ph		proc	ft
		fn	not used
		math	f
		werr	
		wexp	
		wbs	
		wnt	



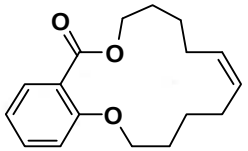
19b



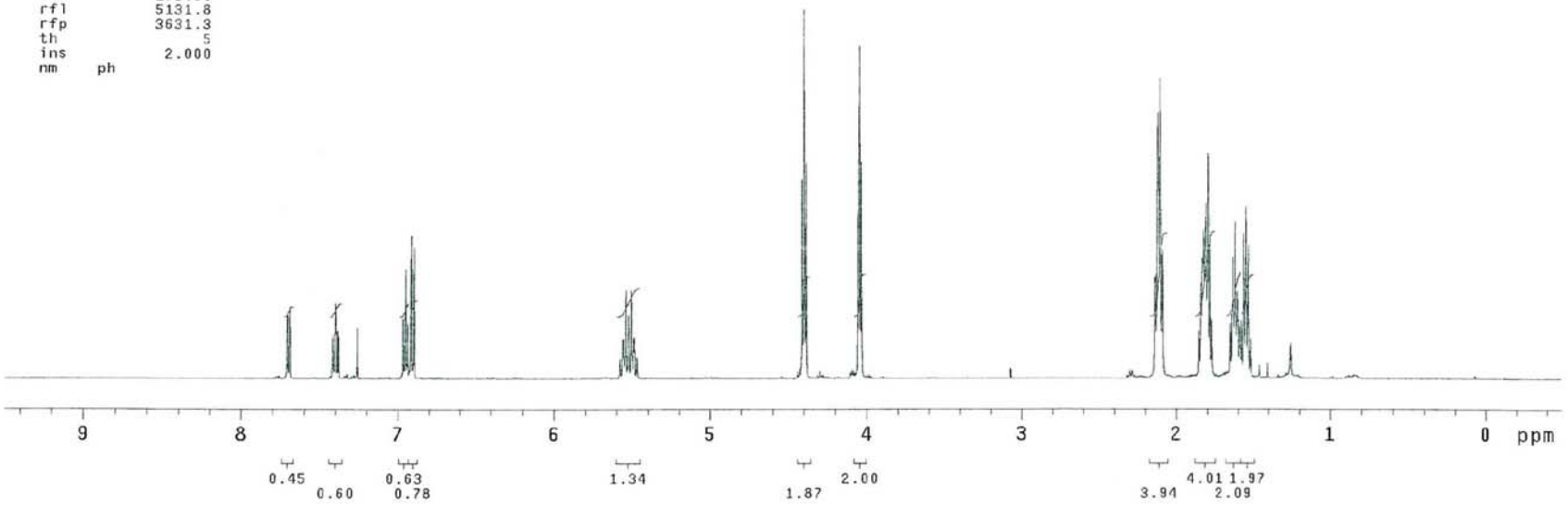
WYKELN10002_1H

exp1 s2pul

SAMPLE		DEC. & VT	
date	Nov 17 2010	dfrq	500.176
solvent	CDCl3	dn	H1
file	/export/home/~	dpwr	32
	ds2/vnmrsys/data/i~	dof	0
	500b/schreiber/WAN~	dm	nnn
	G/Pub1/WYKELN10002~	dmm	c
	_1H.fid	dmf	8770
ACQUISITION		dseq	
sfrq	500.176	dres	1.0
tn	H1	homo	n
at	2.048	temp	24.0
np	32768	PROCESSING	
sw	8000.0	lb	0.10
fb	4000	wtfile	
bs	4	proc	ft
ss	2	fn	not used
tpwr	58	math	f
pw	5.0		
d1	0	werr	
tof	0	wexp	
nt	32	wbs	
ct	32	wnt	wft
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-250.1		
wp	5001.5		
vs	59		
sc	0		
wc	250		
hzmm	20.01		
is	173.80		
rfl	5131.8		
rfp	3631.3		
th	5		
ins	2.000		
nm	ph		



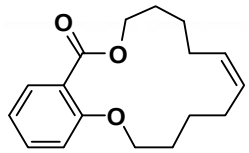
20b



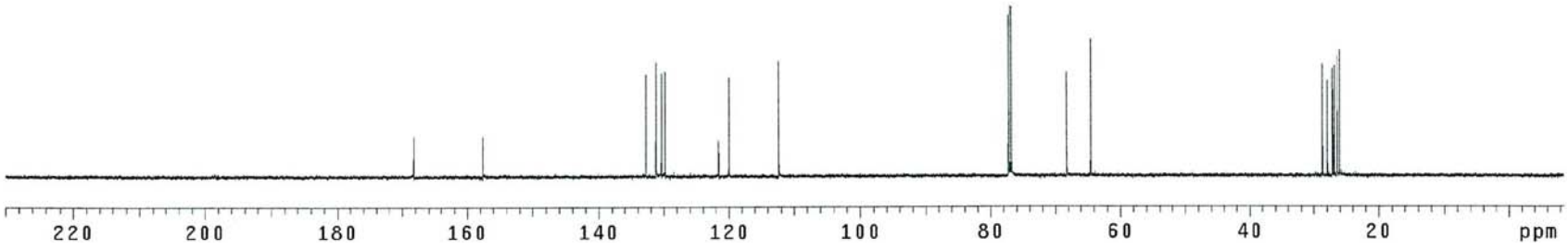
WYKELN10002_13C

exp3 s2pul

SAMPLE		DEC. & VT	
date	Nov 17 2010	dfrq	499.874
solvent	CDCl3	dn	H1
file	exp	dpwr	48
ACQUISITION		dof	0
sfrq	125.707	dm	yyy
tn	C13	dmm	w
at	1.092	dmf	9180
np	65536	dseq	
sw	29996.3	dres	1.0
fb	not used	homo	n
bs	16	temp	25.0
tpwr	55	DEC2	
pw	4.8	dfrq2	0
d1	0	dn2	
tof	2000.0	dpwr2	1
nt	999999	dof2	0
ct	1440	dm2	n
alock	n	dmm2	c
gain	not used	dmf2	10000
FLAGS		dseq2	
il	n	dres2	1.0
in	n	homo2	n
dp	y	DEC3	
hs	nn	dfrq3	0
DISPLAY		dn3	
sp	-1088.7	dpwr3	1
wp	29995.3	dof3	0
vs	27	dm3	n
sc	0	dmm3	c
wc	250	dmf3	10000
hzmm	119.98	dseq3	
is	500.00	dres3	1.0
rfl	10768.0	homo3	n
rfp	9678.3	PROCESSING	
th	4	lb	1.00
ins	100.000	wtfile	
nm cdc ph		proc	ft
		fn	not used
		math	f
		werr	
		wexp	
		wbs	
		wnt	



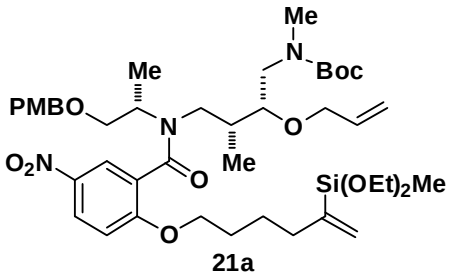
20b



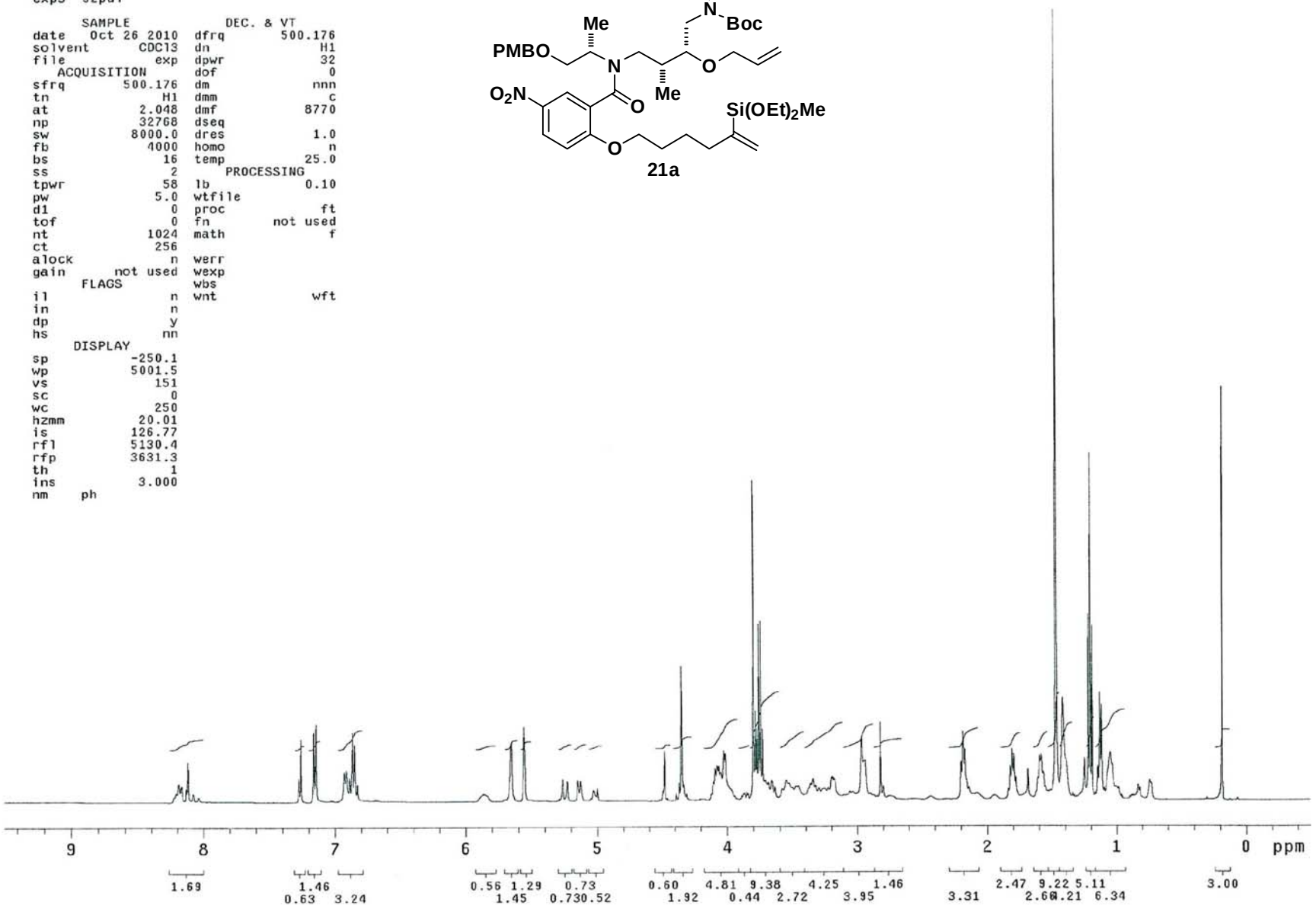
WYKELN11015_1H

exp3 s2pul

SAMPLE		DEC. & VT	
date	Oct 26 2010	dfrq	500.176
solvent	CDCl3	dn	H1
file	exp	dpwr	32
ACQUISITION		dof	0
sfrq	500.176	dm	nnn
tn	H1	dmm	c
at	2.048	dmf	8770
np	32768	dseq	
sw	8000.0	dres	1.0
fb	4000	homo	n
bs	16	temp	25.0
ss	2	PROCESSING	
tpwr	58	lb	0.10
pw	5.0	wtfile	
d1	0	proc	ft
tof	0	fn	not used
nt	1024	math	f
ct	256		
alock	n	werr	
gain	not used	wexp	
FLAGS		wbs	
il	n	wnt	wft
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-250.1		
wp	5001.5		
vs	151		
sc	0		
wc	250		
hzmm	20.01		
is	126.77		
rfl	5130.4		
rfp	3631.3		
th	1		
ins	3.000		
nm	ph		



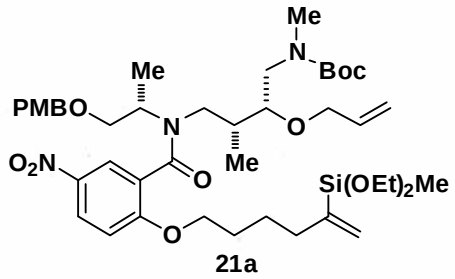
21a



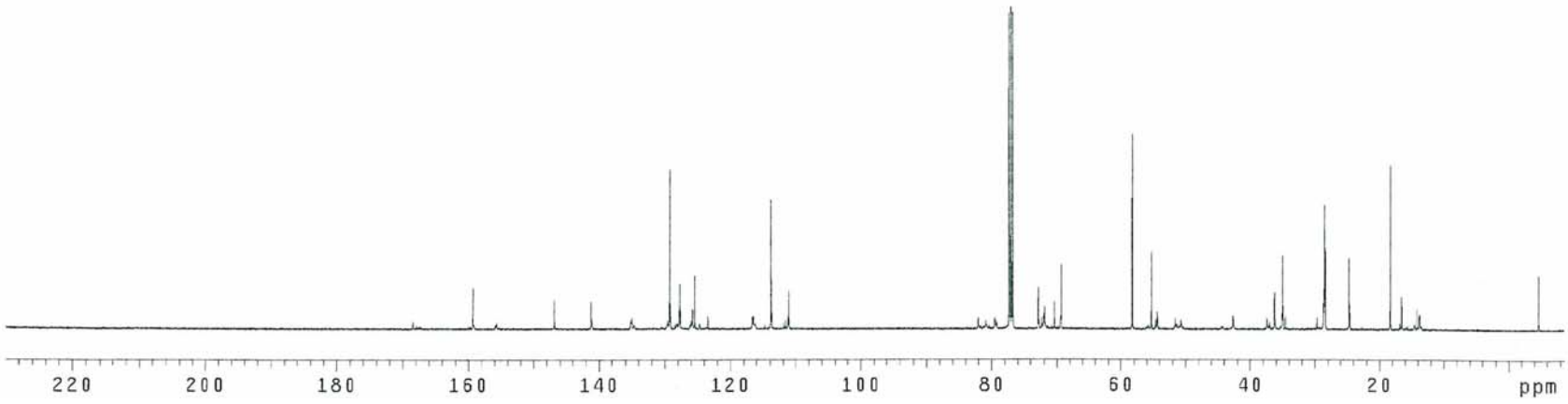
WYKELN11015_13C

exp1 s2pul

SAMPLE		DEC. & VT	
date	Oct 26 2010	dfrq	499.874
solvent	CDCl3	dn	H1
file	/export/home/~	dpwr	48
	ds2/vnmrsys/data/i~	dof	0
	500c/schreiber/WAN~	dm	yyy
	G/Pub1/WYKELN11015~	dmm	w
	13C.fid	dmf	9180
ACQUISITION		dseq	
sfrq	125.707	dres	1.0
tn	C13	homo	n
at	1.092	temp	25.0
np	65536	PROCESSING	
sw	29996.3	lb	1.00
fb	not used	wtfile	
bs	16	proc	ft
tpwr	55	fn	not used
pw	4.8	math	f
d1	0		
tof	2000.0	werr	
nt	999999	wexp	
ct	36144	wbs	
alock	n	wnt	
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-1090.6		
wp	29995.3		
vs	52		
sc	0		
wc	250		
hzmm	119.98		
is	500.00		
rfl	10769.8		
rfp	9678.3		
th	68		
ins	100.000		
nm cdc ph			



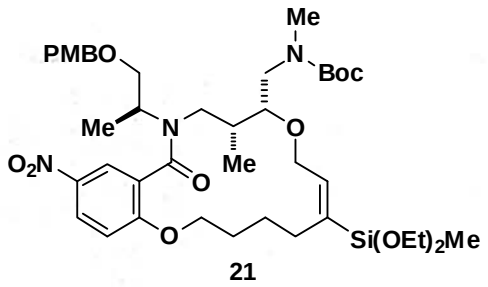
21a



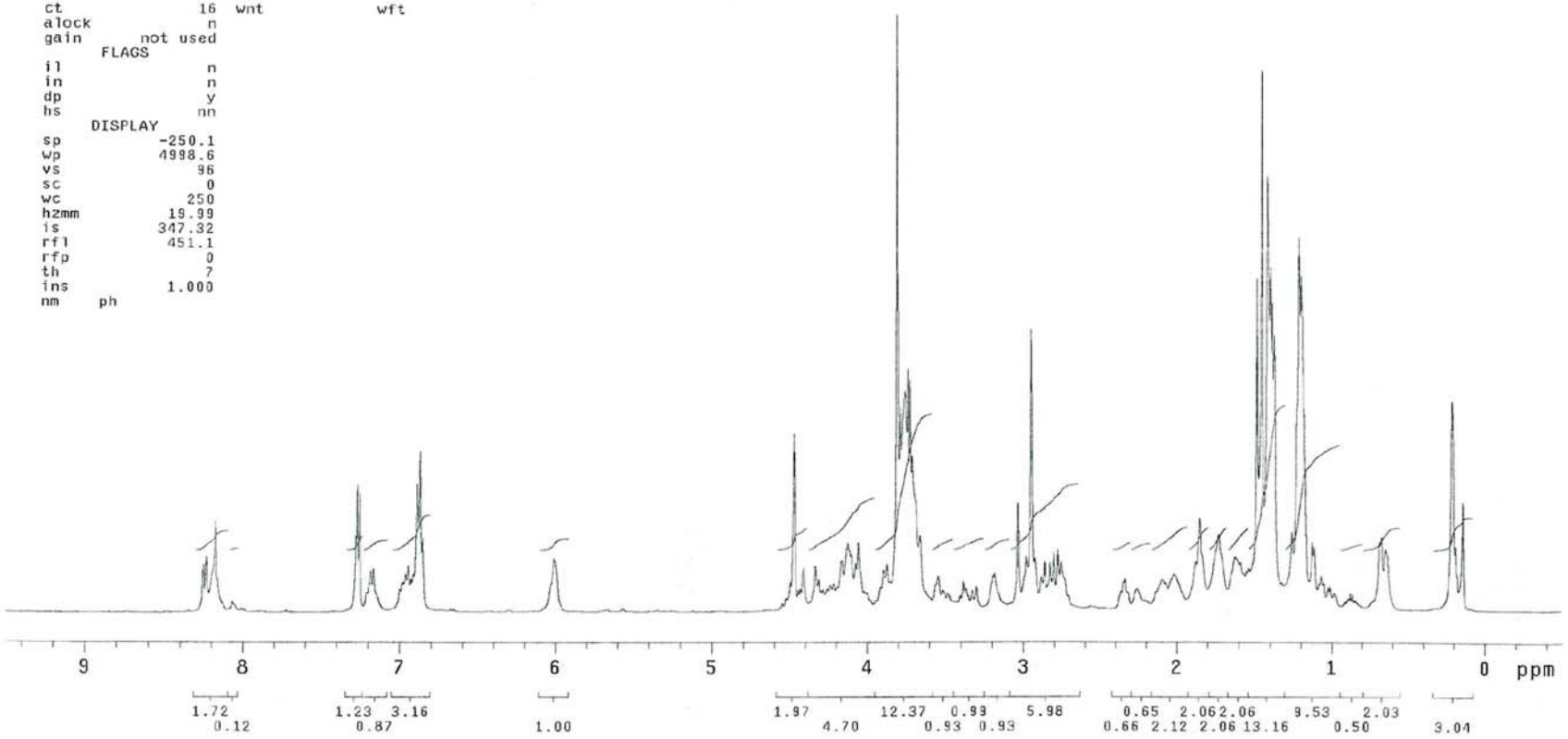
WYKELN11018_1H

exp1 s2pul

SAMPLE		DEC. & VT	
date	Oct 27 2010	dfrq	499.874
solvent	CDCl3	dn	H1
file	/export/home/~	dpwr	30
	ds2/vnmrsys/data/i~	dof	0
	500c/schreiber/WAN~	dm	nnn
	G/Pub1/WYKELN11018~	dmm	c
	_1H.fid	dmf	200
ACQUISITION		dseq	
sfrq	499.875	dres	1.0
tn	H1	homo	n
at	2.184	temp	25.0
np	32768	PROCESSING	
sw	7501.2	lb	1.10
fb	not used	wtfile	
bs	8	proc	ft
ss	2	fn	32768
tpwr	62	math	f
pw	12.0		
d1	0	werr	
tof	800.0	wexp	
nt	16	wbs	
ct	16	wnt	wft
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-250.1		
wp	4998.6		
vs	96		
sc	0		
wc	250		
hzmm	19.99		
is	347.32		
rfl	451.1		
rfp	0		
th	7		
ins	1.000		
nm	ph		



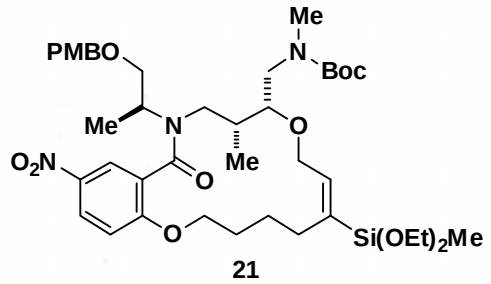
21



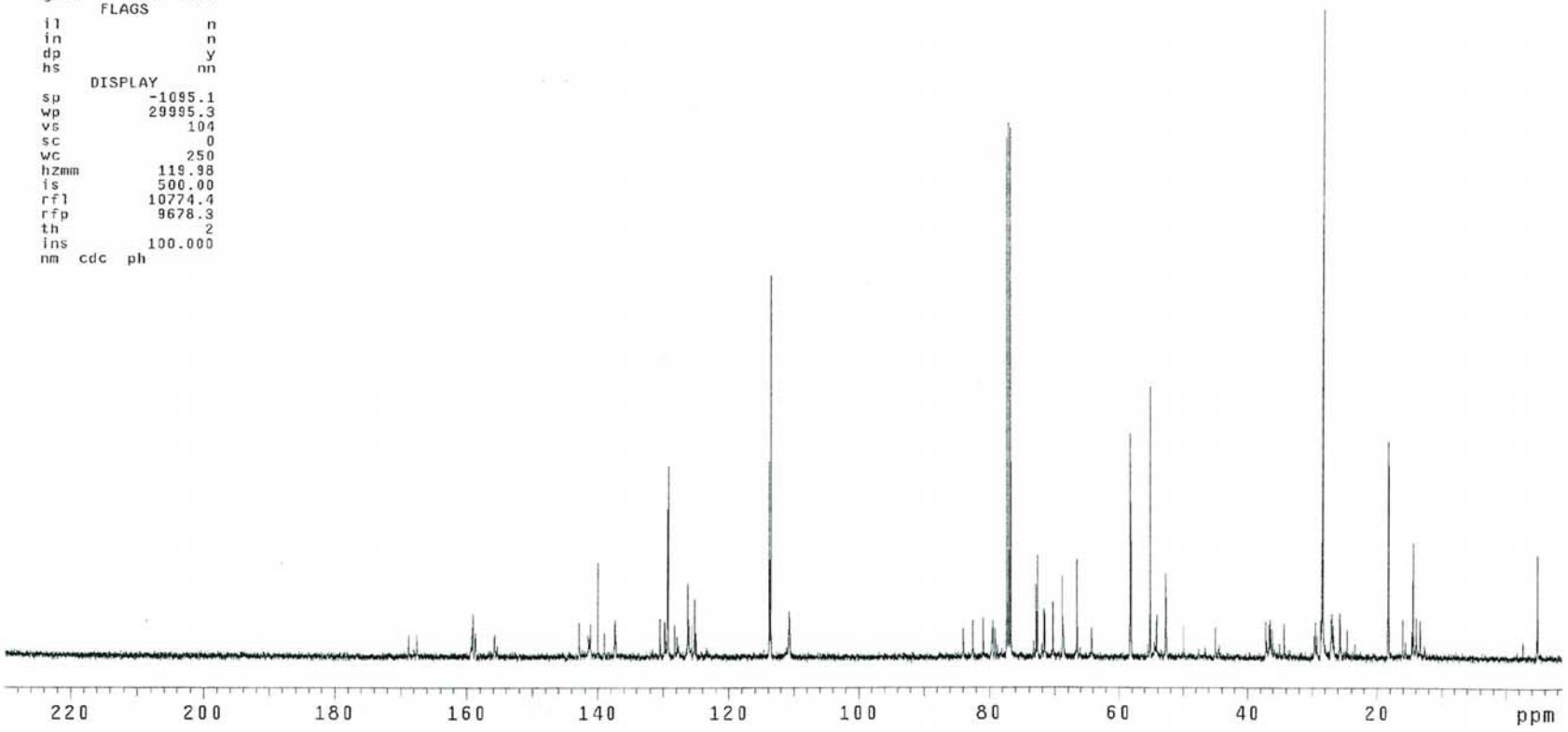
WYKELN11018_13C

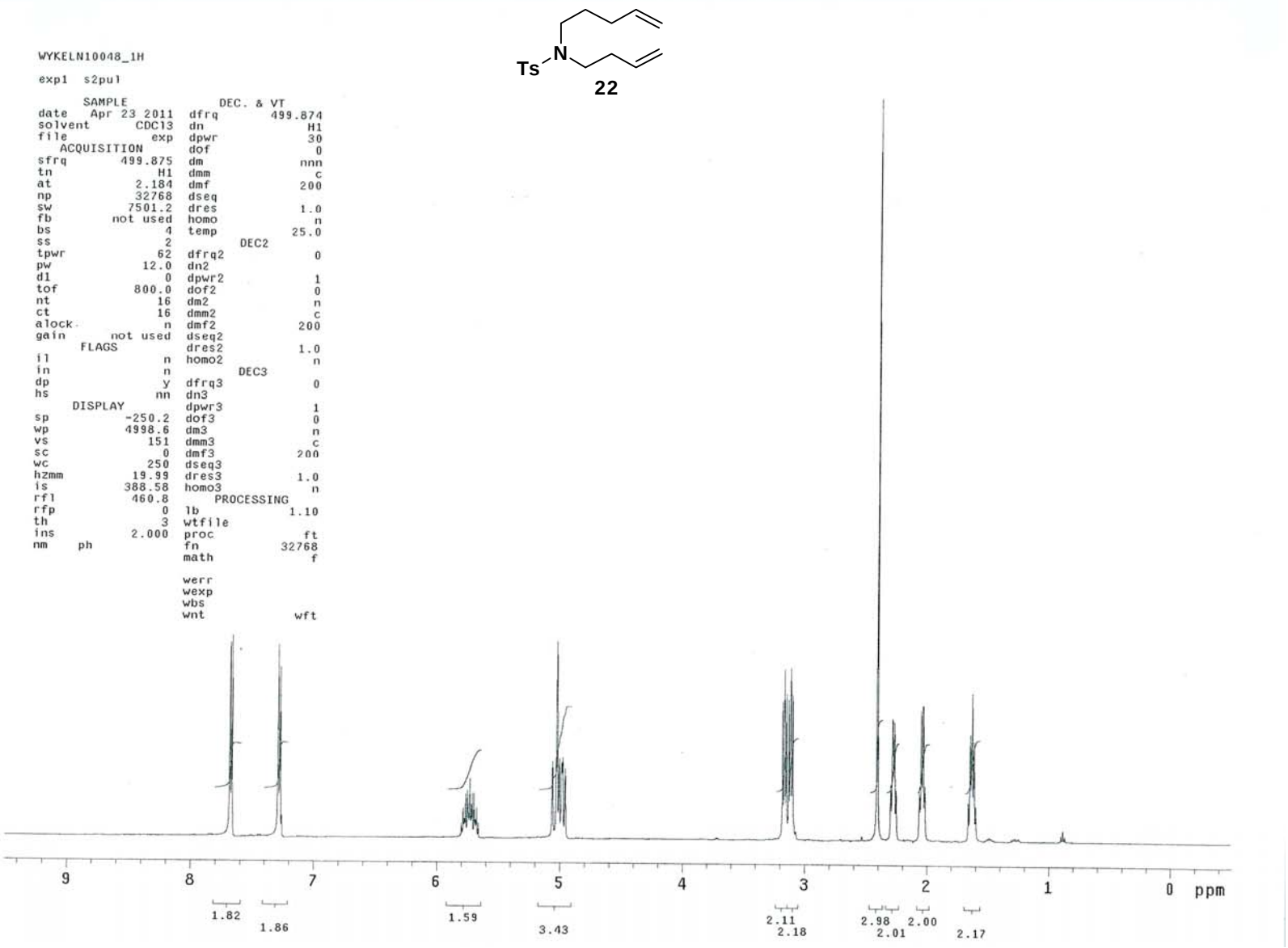
exp1 s2pul

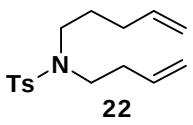
SAMPLE		DEC. & VT	
date	Oct 27 2010	dfrq	499.874
solvent	CDCl3	dn	H1
file	/export/home/~	dpwr	48
ds2/vnmrsys/data/i~		dof	0
500c/schreiber/WAN~		dm	yyy
G/Pub1/WYKELN11018~		dmm	w
	_13C.fid	dmf	9180
ACQUISITION		dseq	
sfrq	125.707	dres	1.0
tn	C13	homo	n
at	1.092	temp	25.0
np	65536	PROCESSING	
sw	29996.3	lb	1.00
fb	not used	wtfile	
bs	16	proc	ft
tpwr	55	fn	not used
pw	4.8	math	f
d1	0		
tof	2000.0	werr	
nt	999999	wexp	
ct	3104	wbs	
alock	n	wnt	
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-1095.1		
wp	29995.3		
vs	104		
sc	0		
wc	250		
hzmm	119.98		
is	500.00		
rfl	10774.4		
rfp	9678.3		
th	2		
ins	100.000		
nm cdc ph			



21




WYKELN10048_1H
exp1 s2pul
SAMPLE
date Apr 23 2011
solvent CDCl3
file exp
ACQUISITION
sfrq 499.875
tn H1
at 2.184
np 32768
sw 7501.2
fb not used
bs 4
ss 2
tpwr 62
pw 12.0
d1 0
tof 800.0
nt 16
ct 16
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -250.2
wp 4998.6
vs 151
sc 0
wc 250
hzmm 19.99
is 388.58
rfl 460.8
rfp 0
th 3
ins 2.000
nm ph
DEC. & VT
dfrq 499.874
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
temp 25.0
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 200
dseq2
dres2 1.0
homo2 n
DEC3
dfrq3 0
dn3
dpwr3 1
dof3 0
dm3 n
dmm3 c
dmf3 200
dseq3
dres3 1.0
homo3 n
PROCESSING
lb 1.10
wtfile
proc ft
fn 32768
math f
werr
wexp
wbs
wnt wft
Ts
N
22
9
8
7
6
5
4
3
2
1
0 ppm
1.82
1.86
1.59
3.43
2.11
2.18
2.98
2.01
2.00
2.17

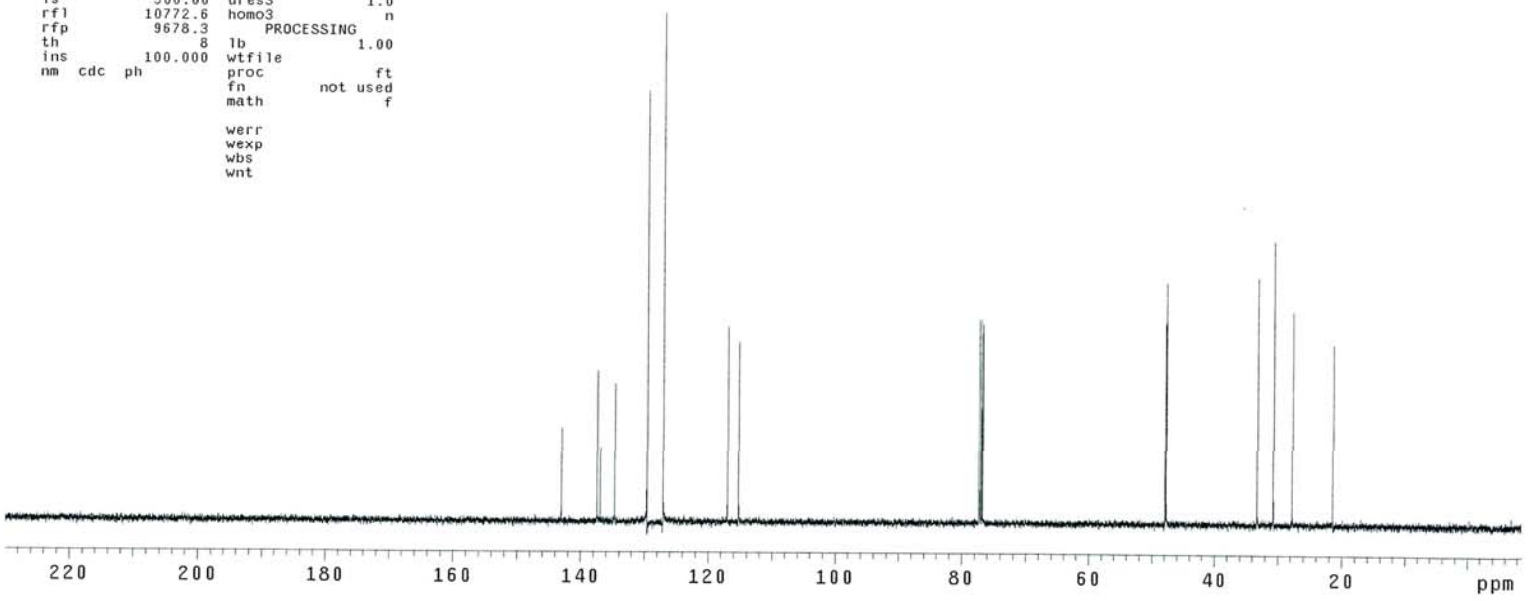


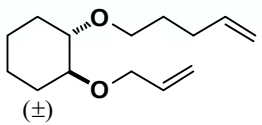
22

WYKELN10048_13C

exp2 s2pul

SAMPLE		DEC. & VT	
date	Apr 23 2011	dfrq	499.874
solvent	CDCl3	dn	H1
file	exp	dpwr	48
ACQUISITION		dof	0
sfrq	125.707	dm	yyy
tn	C13	dmm	w
at	1.092	dmf	8929
np	65536	dseq	
sw	29996.3	dres	1.0
fb	not used	homo	n
bs	16	temp	25.0
tpwr	55	DEC2	
pw	4.8	dfrq2	0
d1	0	dn2	
tof	2000.0	dpwr2	1
nt	9999	dof2	0
ct	144	dm2	n
alock	n	dmm2	c
gain	not used	dmf2	10000
FLAGS		dseq2	
il	n	dres2	1.0
in	n	homo2	n
dp	y	DEC3	
hs	nn	dfrq3	0
DISPLAY		dn3	
sp	-1093.3	dpwr3	1
wp	29995.3	dof3	0
vs	84	dm3	n
sc	0	dmm3	c
wc	250	dmf3	10000
hzmm	119.98	dseq3	
is	500.00	dres3	1.0
rfl	10772.6	homo3	n
rfp	9678.3	PROCESSING	
th	8	lb	1.00
ins	100.000	wtfile	
nm cdc ph		proc	ft
		fn	not used
		math	f
		werr	
		wexp	
		wbs	
		wnt	





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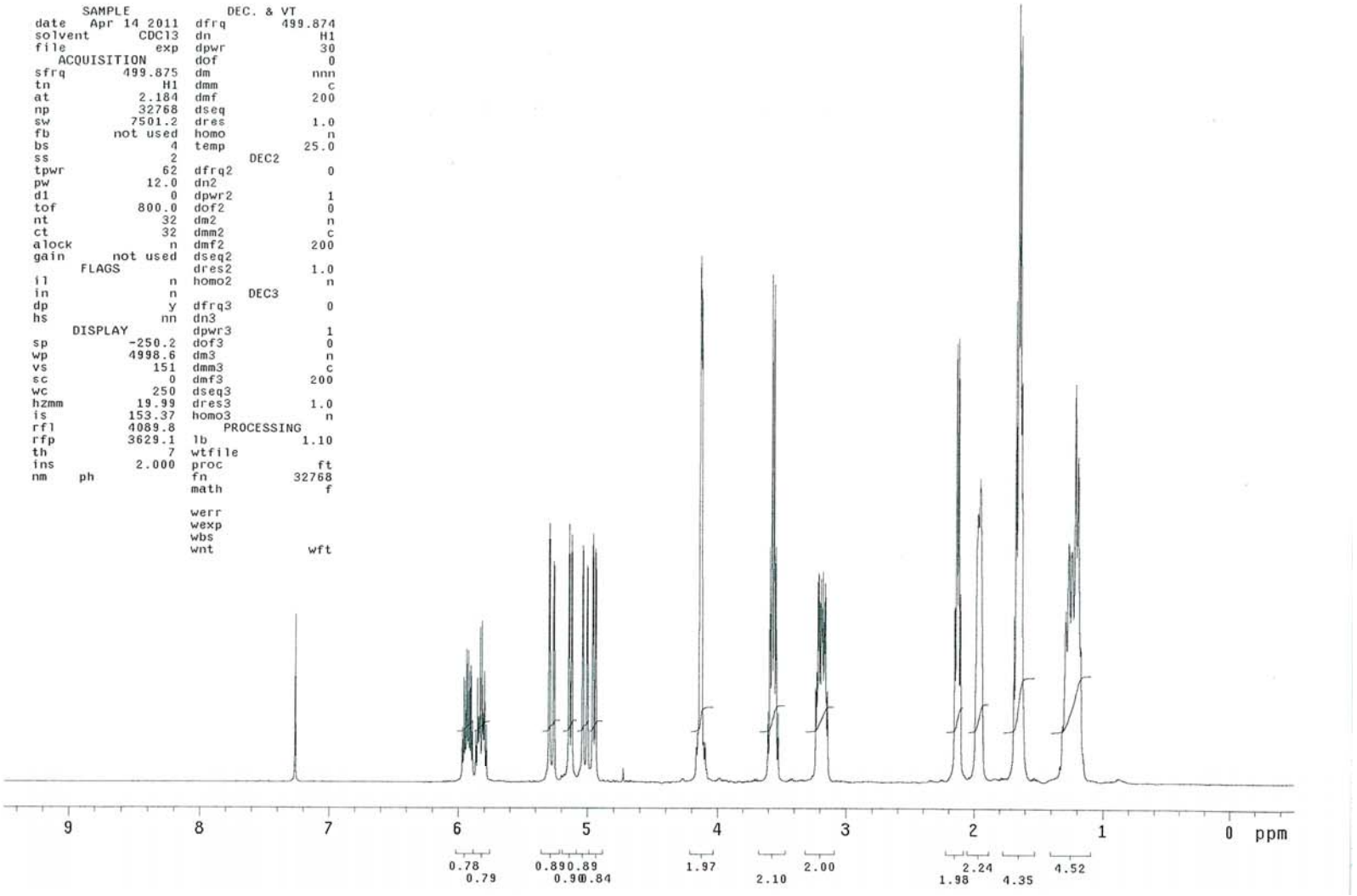
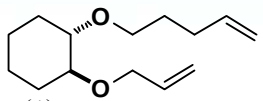
23


WYKELN8091_1H
exp2 s2pul
SAMPLE
date Apr 14 2011
solvent CDCl3
file exp
ACQUISITION
sfrq 499.875
tn H1
at 2.184
np 32768
sw 7501.2
fb not used
bs 4
ss 2
tpwr 62
pw 12.0
d1 0
tof 800.0
nt 32
ct 32
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -250.2
wp 4998.6
vs 151
sc 0
wc 250
hzmm 19.99
is 153.37
rfl 4089.8
rfp 3629.1
th 7
ins 2.000
nm ph
DEC. & VT
dfrq 499.874
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
temp 25.0
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 200
dseq2
dres2 1.0
homo2 n
DEC3
dfrq3 0
dn3
dpwr3 1
dof3 0
dm3 n
dmm3 c
dmf3 200
dseq3
dres3 1.0
homo3 n
PROCESSING
lb 1.10
wtfile
proc ft
fn 32768
math f
werr
wexp
wbs
wnt wft
9
8
7
6
5
4
3
2
1
0 ppm
0.78
0.79
0.89
0.90
0.89
0.84
1.97
2.10
2.00
2.24
1.98
4.35
4.52



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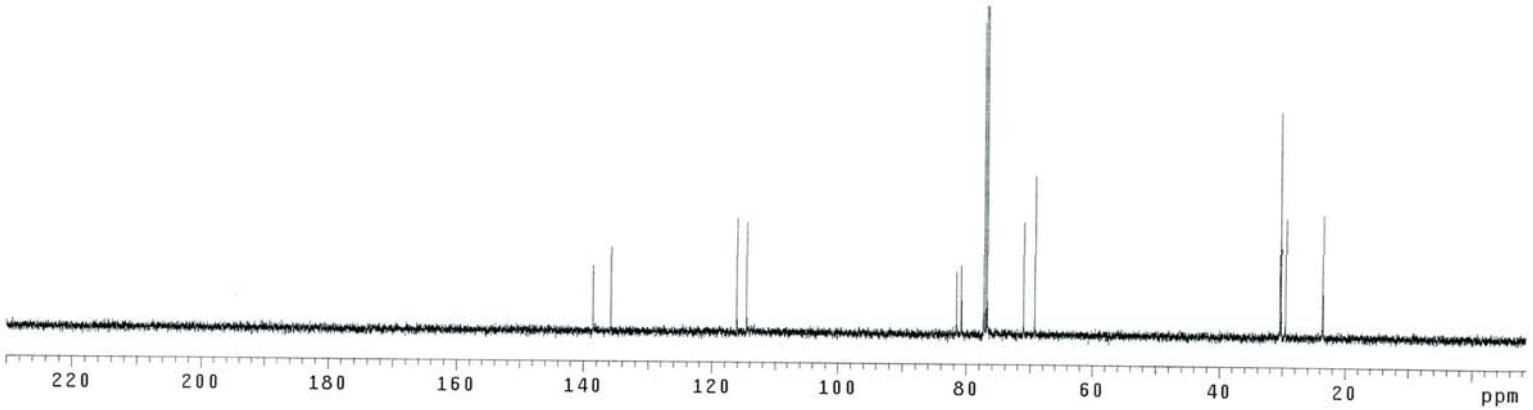
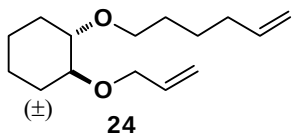
23

```
WYKELN8091_13C

exp3  s2pul

      SAMPLE
date    Apr 14 2011
solvent       CDCl3
file            exp
   ACQUISITION
sfrq        125.707
tn              C13
at              1.092
np              65536
sw           29996.3
fb          not used
bs                16
tpwr              55
pw               4.8
d1                0
tof            2000.0
nt               9999
ct                208
alock               n
gain        not used
      FLAGS
il                  n
in                  n
dp                  y
hs                 nn
    DISPLAY
sp           -1087.8
wp           29995.3
vs                54
sc                 0
wc               250
hzmm          119.98
is            500.00
rfl          10767.1
rfp           9678.3
th                 5
ins          100.000
nm  cdc  ph

      DEC. & VT
dfrq         499.874
dn                H1
dpwr              48
dof                0
dm               yyy
dmm                w
dmf             8929
dseq
dres             1.0
homo               n
temp            25.0
      DEC2
dfrq2              0
dn2
dpwr2              1
dof2               0
dm2                n
dmm2               c
dmf2           10000
dseq2
dres2            1.0
homo2              n
      DEC3
dfrq3              0
dn3
dpwr3              1
dof3               0
dm3                n
dmm3               c
dmf3           10000
dseq3
dres3            1.0
homo3              n
     PROCESSING
lb              1.00
wtfile
proc               ft
fn          not used
math               f

werr
wexp
wbs
wnt
```





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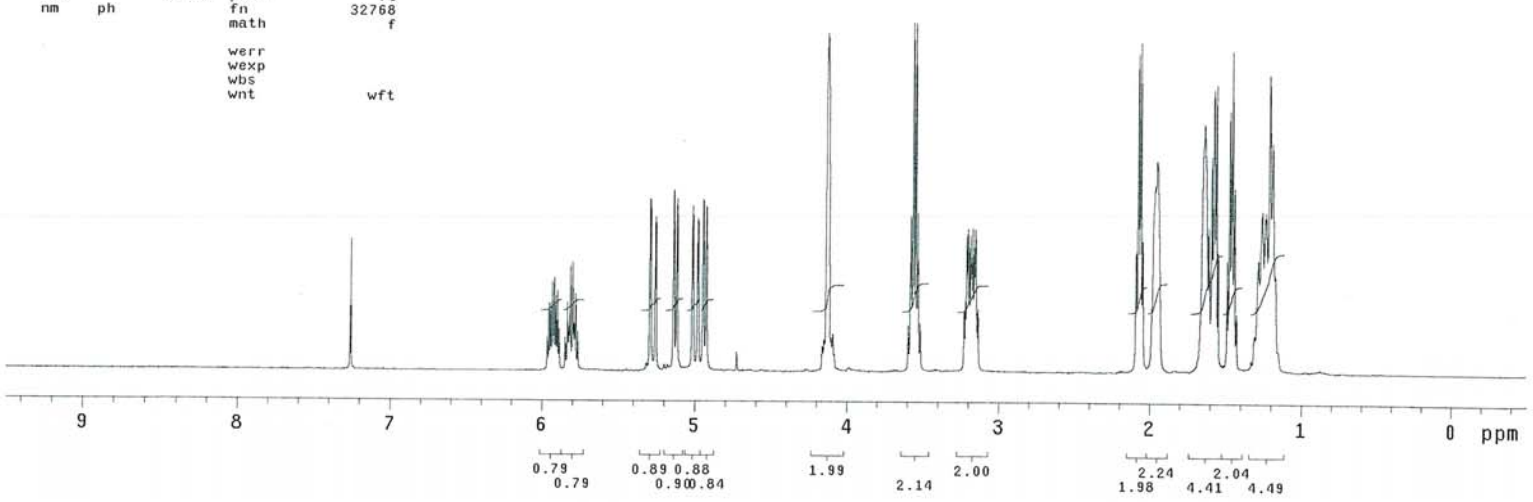
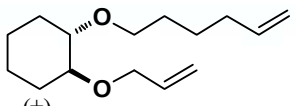
24

```
WYKELN8092_1H

exp2  s2pul

      SAMPLE
date    Apr 14 2011
solvent       CDCl3
file             exp
   ACQUISITION
sfrq         499.875
tn                H1
at            2.184
np            32768
sw           7501.2
fb         not used
bs                4
ss                2
tpwr             62
pw             12.0
d1                0
tof           800.0
nt               16
ct               16
alock             n
gain       not used
      FLAGS
il                n
in                n
dp                y
hs               nn
     DISPLAY
sp           -250.2
wp           4998.6
vs               58
sc                0
wc              250
hzmm          19.99
is           153.37
rfl          4090.3
rfp          3629.1
th                5
ins           2.000
nm     ph

      DEC. & VT
dfrq         499.874
dn                H1
dpwr              30
dof                0
dm               nnn
dmm                c
dmf              200
dseq
dres             1.0
homo               n
temp            25.0
     DEC2
dfrq2              0
dn2
dpwr2              1
dof2               0
dm2                n
dmm2               c
dmf2             200
dseq2
dres2            1.0
homo2              n
     DEC3
dfrq3              0
dn3
dpwr3              1
dof3               0
dm3                n
dmm3               c
dmf3             200
dseq3
dres3            1.0
homo3              n
     PROCESSING
lb              1.10
wtfile
proc              ft
fn             32768
math               f

werr
wexp
wbs
wnt              wft
```





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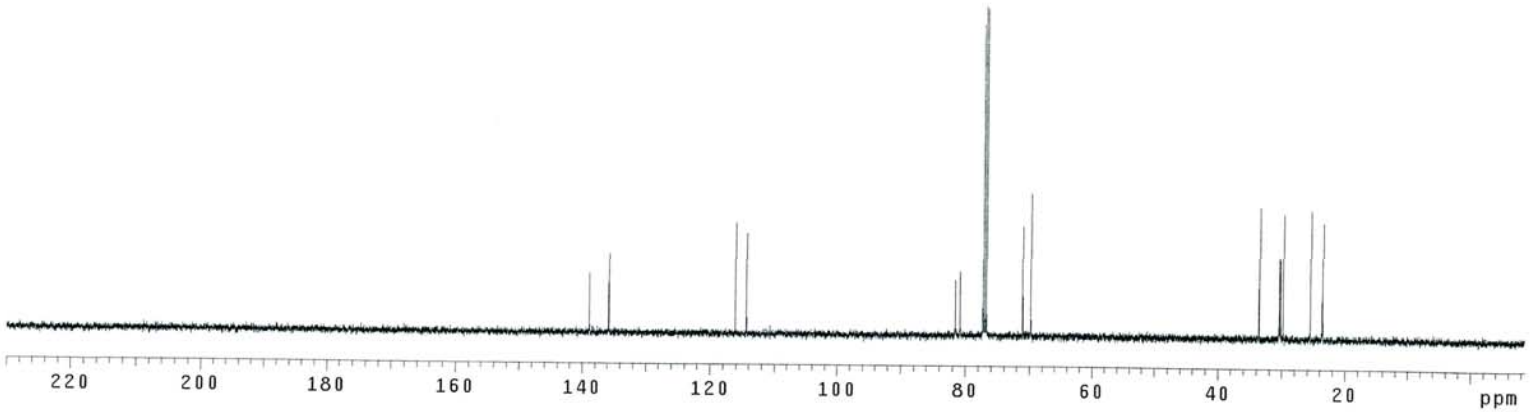
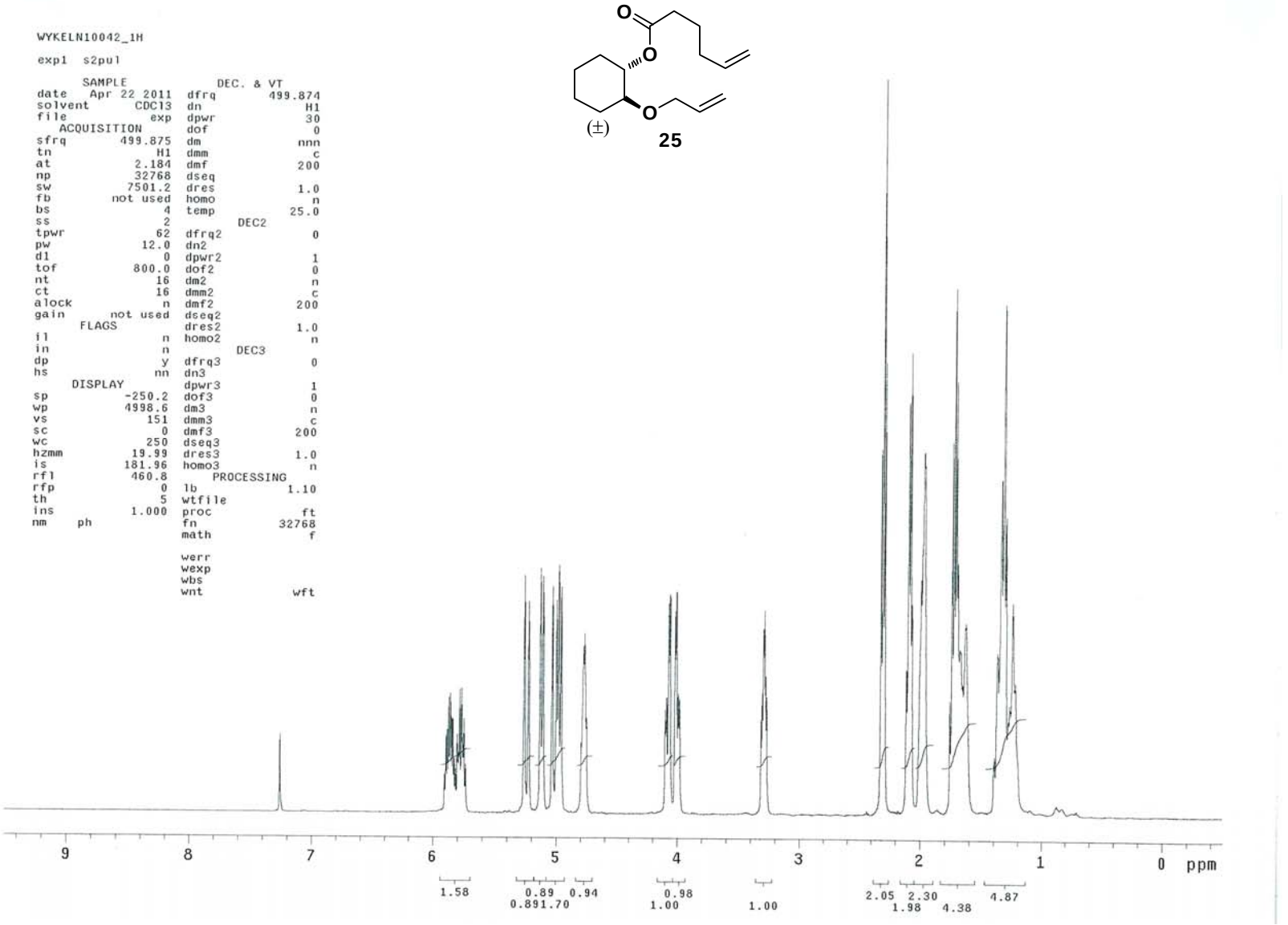
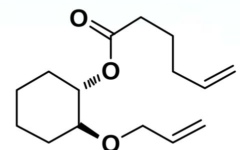
24

```
WYKELN8092_13C

exp3  s2pul

      SAMPLE
date      Apr 14 2011
solvent        CDCl3
file             exp
   ACQUISITION
sfrq         125.707
tn               C13
at             1.092
np             65536
sw           29996.3
fb         not used
bs               16
tpwr             55
pw              4.8
d1                0
tof          2000.0
nt             9999
ct              320
alock             n
gain       not used
      FLAGS
il                n
in                n
dp                y
hs               nn
    DISPLAY
sp           -1087.8
wp           29995.3
vs                54
sc                 0
wc               250
hzmm          119.98
is            500.00
rfl         10767.1
rfp          9678.3
th                 5
ins          100.000
nm  cdc  ph

      DEC. & VT
dfrq         499.874
dn                H1
dpwr              48
dof                0
dm               yyy
dmm                w
dmf             8929
dseq
dres             1.0
homo               n
temp            25.0
      DEC2
dfrq2              0
dn2
dpwr2              1
dof2               0
dm2                n
dmm2               c
dmf2           10000
dseq2
dres2            1.0
homo2              n
      DEC3
dfrq3              0
dn3
dpwr3              1
dof3               0
dm3                n
dmm3               c
dmf3           10000
dseq3
dres3            1.0
homo3              n
    PROCESSING
lb              1.00
wtfile
proc               ft
fn          not used
math               f

werr
wexp
wbs
wnt
```




WYKELN10042_1H
exp1 s2pul
SAMPLE
date Apr 22 2011
solvent CDCl3
file exp
ACQUISITION
sfrq 499.875
tn H1
at 2.184
np 32768
sw 7501.2
fb not used
bs 4
ss 2
tpwr 62
pw 12.0
d1 0
tof 800.0
nt 16
ct 16
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -250.2
wp 4998.6
vs 151
sc 0
wc 250
hzmm 19.99
is 181.96
rfl 460.8
rfp 0
th 5
ins 1.000
nm ph
DEC. & VT
dfrq 499.874
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
temp 25.0
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 200
dseq2
dres2 1.0
homo2 n
DEC3
dfrq3 0
dn3
dpwr3 1
dof3 0
dm3 n
dmm3 c
dmf3 200
dseq3
dres3 1.0
homo3 n
PROCESSING
lb 1.10
wtfile
proc ft
fn 32768
math f
werr
wexp
wbs
wnt wft
O
O
O
(±)
25
9
8
7
6
5
4
3
2
1
0 ppm
1.58
0.89
0.94
0.891.70
0.98
1.00
1.00
2.05
2.30
1.98
4.38
4.87

```
WYKELN10042_13C

exp2  s2pul

     SAMPLE
date    Apr 22 2011
solvent       CDCl3
file            exp
   ACQUISITION
sfrq        125.707
tn              C13
at            1.092
np            65536
sw          29996.3
fb         not used
bs               16
tpwr             55
pw              4.8
d1                0
tof          2000.0
nt             9999
ct              192
alock             n
gain       not used
     FLAGS
il                n
in                n
dp                y
hs               nn
    DISPLAY
sp          -1089.7
wp          29995.3
vs               47
sc                0
wc              250
hzmm         119.98
is           500.00
rfl         10768.9
rfp          9678.3
th                3
ins         100.000
nm  cdc  ph

     DEC. & VT
dfrq        499.874
dn               H1
dpwr             48
dof               0
dm              yyy
dmm               w
dmf            8929
dseq
dres            1.0
homo              n
temp           25.0
       DEC2
dfrq2             0
dn2
dpwr2             1
dof2              0
dm2               n
dmm2              c
dmf2          10000
dseq2
dres2           1.0
homo2             n
       DEC3
dfrq3             0
dn3
dpwr3             1
dof3              0
dm3               n
dmm3              c
dmf3          10000
dseq3
dres3           1.0
homo3             n
    PROCESSING
lb             1.00
wtfile
proc             ft
fn         not used
math              f

werr
wexp
wbs
wnt
```



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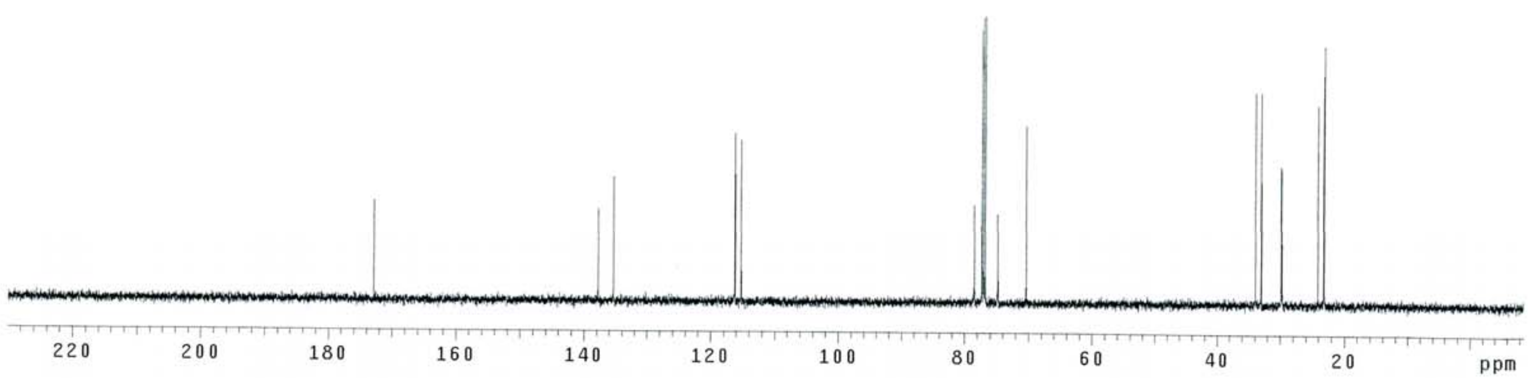
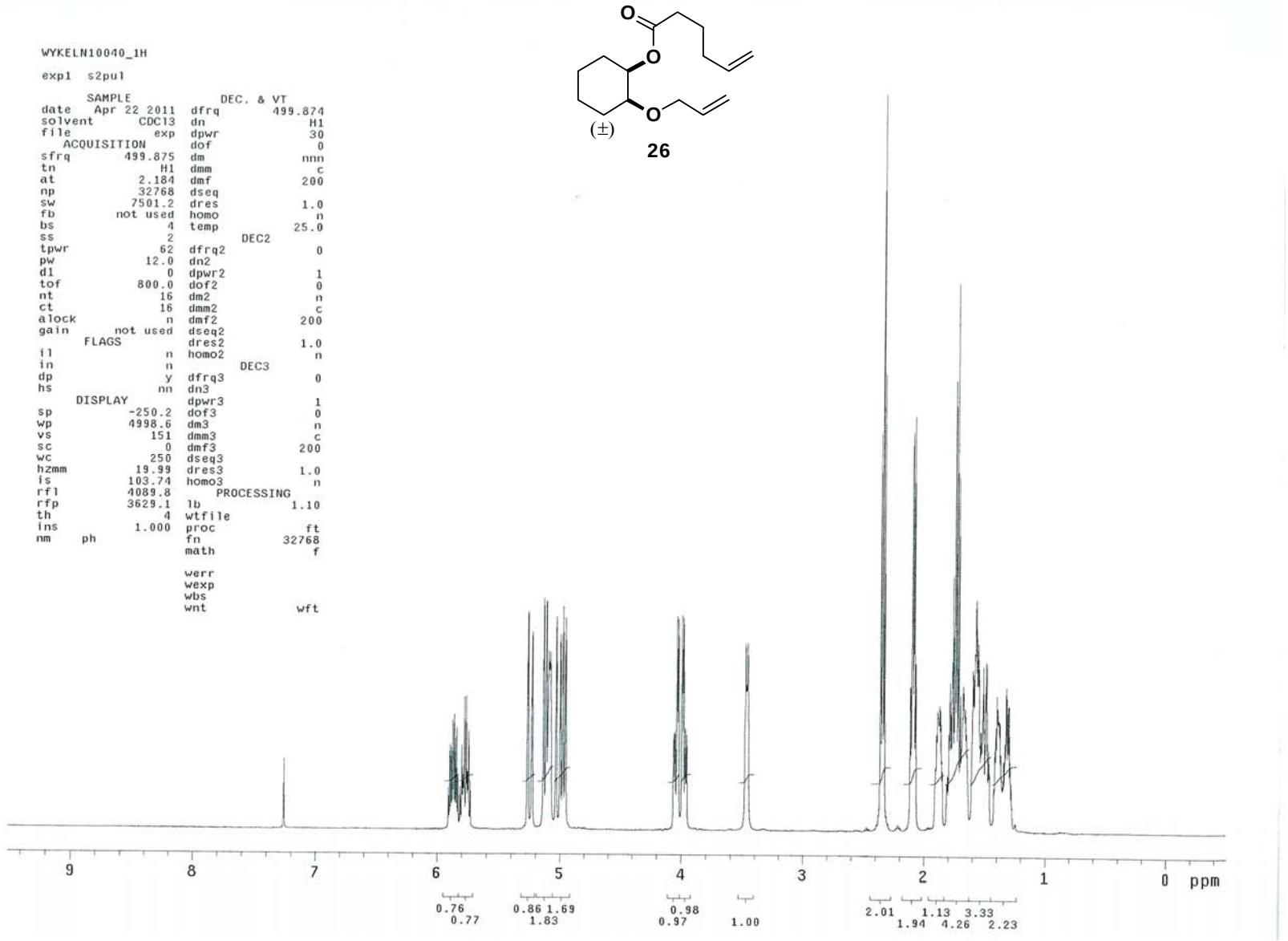
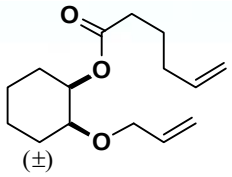
25




WYKELN10040_1H
exp1 s2pul
SAMPLE
date Apr 22 2011
solvent CDCl3
file exp
ACQUISITION
sfrq 499.875
tn H1
at 2.184
np 32768
sw 7501.2
fb not used
bs 4
ss 2
tpwr 62
pw 12.0
d1 0
tof 800.0
nt 16
ct 16
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -250.2
wp 4998.6
vs 151
sc 0
wc 250
hzmm 19.99
is 103.74
rfl 4089.8
rfp 3629.1
th 4
ins 1.000
nm ph
DEC. & VT
dfrq 499.874
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
temp 25.0
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 200
dseq2
dres2 1.0
homo2 n
DEC3
dfrq3 0
dn3
dpwr3 1
dof3 0
dm3 n
dmm3 c
dmf3 200
dseq3
dres3 1.0
homo3 n
PROCESSING
lb 1.10
wtfile
proc ft
fn 32768
math f
werr
wexp
wbs
wnt wft
O
O
O
(±)
26
9
8
7
6
5
4
3
2
1
0 ppm
0.76
0.77
0.86 1.69
1.83
0.98
0.97
1.00
2.01
1.94
1.13 3.33
4.26 2.23



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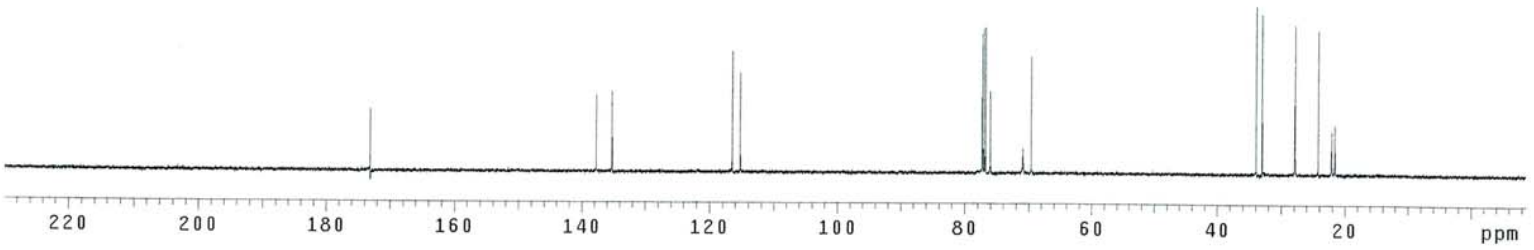
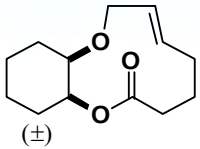
26

WYKELN10040_13C

exp2 s2pul

SAMPLE		DEC. & VT	
date	Apr 22 2011	dfrq	499.874
solvent	CDCl3	dn	H1
file	exp	dpwr	48
ACQUISITION		dof	0
sfrq	125.707	dm	yyy
tn	C13	dmm	w
at	1.092	dmf	8929
np	65536	dseq	
sw	29996.3	dres	1.0
fb	not used	homo	n
bs	16	temp	25.0
tpwr	55	DEC2	
pw	4.8	dfrq2	0
d1	0	dn2	
tof	2000.0	dpwr2	1
nt	99999	dof2	0
ct	288	dm2	n
alock	n	dmm2	c
gain	not used	dmf2	10000
FLAGS		dseq2	
il	n	dres2	1.0
in	n	homo2	n
dp	y	DEC3	
hs	nn	dfrq3	0
DISPLAY		dn3	
sp	-1090.6	dpwr3	1
wp	29995.3	dof3	0
vs	27	dm3	n
sc	0	dmm3	c
wc	250	dmf3	10000
hzmm	119.98	dseq3	
is	500.00	dres3	1.0
rfl	10769.8	homo3	n
rfp	9678.3	PROCESSING	
th	2	lb	1.00
ins	100.000	wtfile	
nm cdc ph		proc	ft
		fn	not used
		math	f
		werr	
		wexp	
		wbs	
		wnt	





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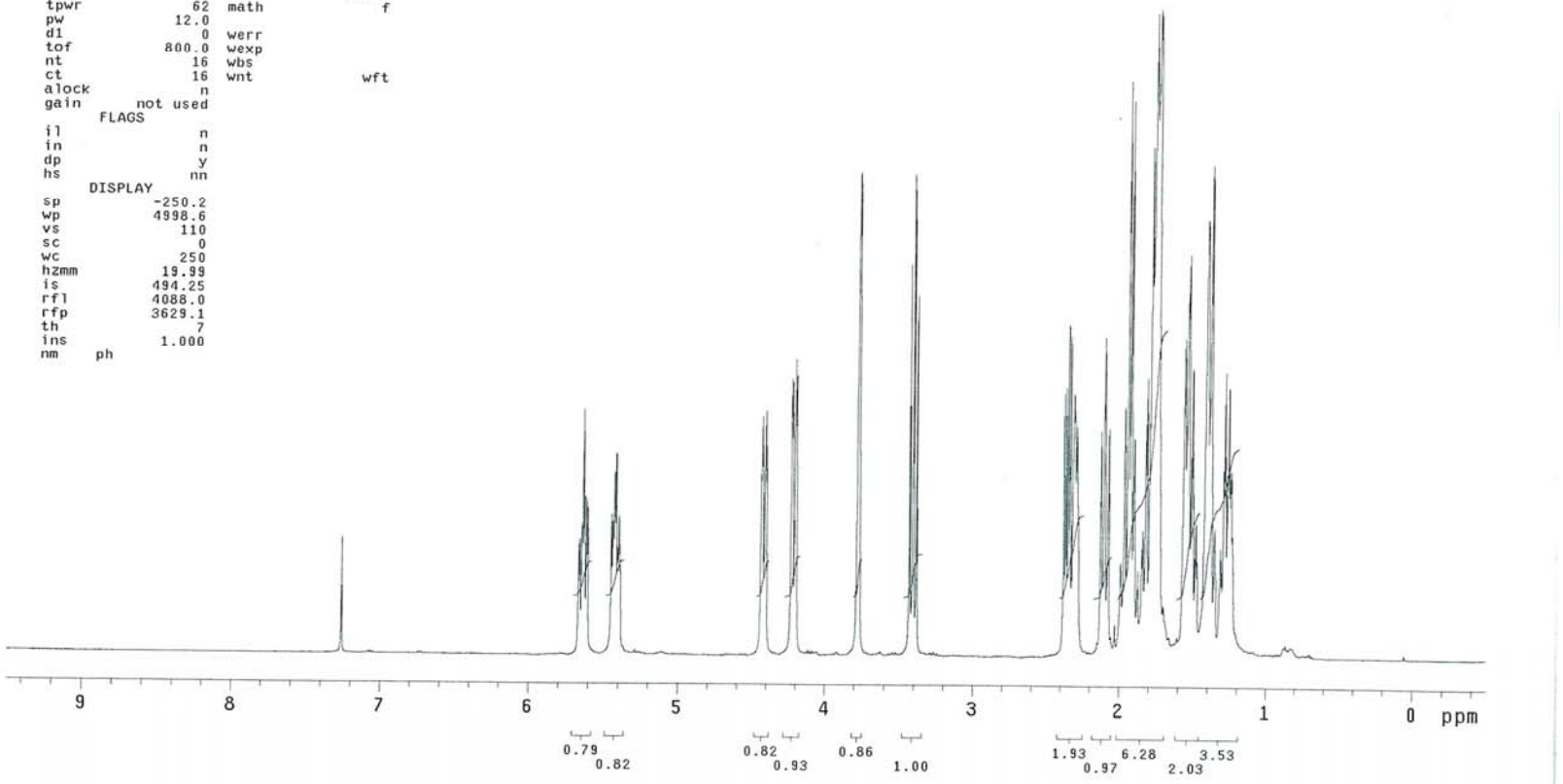
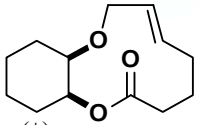
26b

S/N = 351

exp3 s2pul

SAMPLE		DEC. & VT	
date	Apr 22 2011	dfrq	499.874
solvent	CDCl3	dn	H1
file	/export/home/~	dpwr	30
	ds2/vnmrsys/data/i~	dof	0
	500c/schreiber/mji~	dm	nnn
	menez/MJ2034-35.fi~	dmm	c
	d	dmf	200
ACQUISITION		dseq	
sfrq	499.875	dres	1.0
tn	H1	homo	n
at	2.184	temp	25.0
np	32768	**PROCESSING**	
sw	7501.2	lb	1.10
fb	not used	wtfile	
bs	8	proc	ft
ss	2	fn	32768
tpwr	62	math	f
pw	12.0		
d1	0	werr	
tof	800.0	wexp	
nt	16	wbs	
ct	16	wnt	wft
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-250.2		
wp	4998.6		
vs	110		
sc	0		
wc	250		
hzmm	19.99		
is	494.25		
rfl	4088.0		
rfp	3629.1		
th	7		
ins	1.000		
nm	ph		





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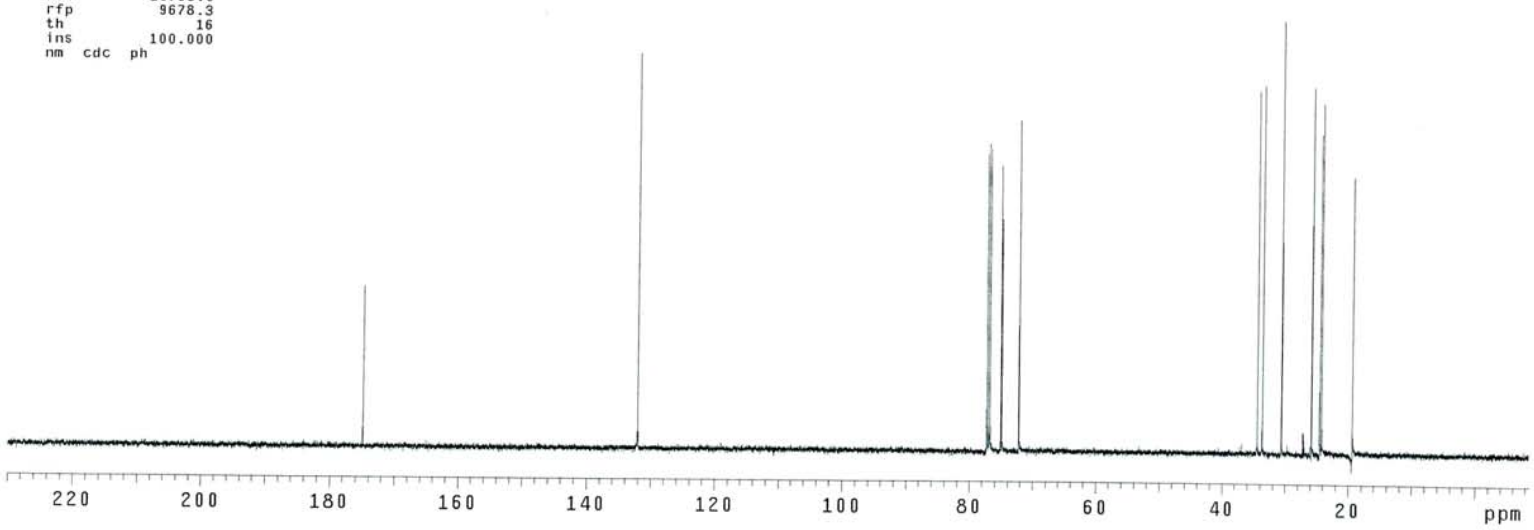
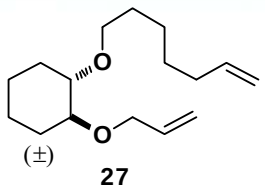
26b

STANDARD CARBON PARAMETERS

exp3 s2pul

SAMPLE		DEC. & VT	
date	Apr 22 2011	dfrq	499.874
solvent	CDCl3	dn	H1
file	/export/home/~	dpwr	48
ds2/vnmrsys/data/i~		dof	0
500c/schreiber/mji~		dm	yyy
menez/MJ2034-35_C1~		dmm	w
	3.fid	dmf	8929
ACQUISITION		dseq	
sfrq	125.707	dres	1.0
tn	C13	homo	n
at	1.092	temp	25.0
np	65536	PROCESSING	
sw	29996.3	lb	1.00
fb	not used	wtfile	
bs	32	proc	ft
tpwr	55	fn	not used
pw	4.8	math	f
d1	0		
tof	2000.0	werr	
nt	1024	wexp	
ct	742	wbs	
alock	n	wnt	
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-1088.7		
wp	29995.3		
vs	71		
sc	0		
wc	250		
hzmm	119.98		
is	500.00		
rfl	10768.0		
rfp	9678.3		
th	16		
ins	100.000		
nm cdc ph			





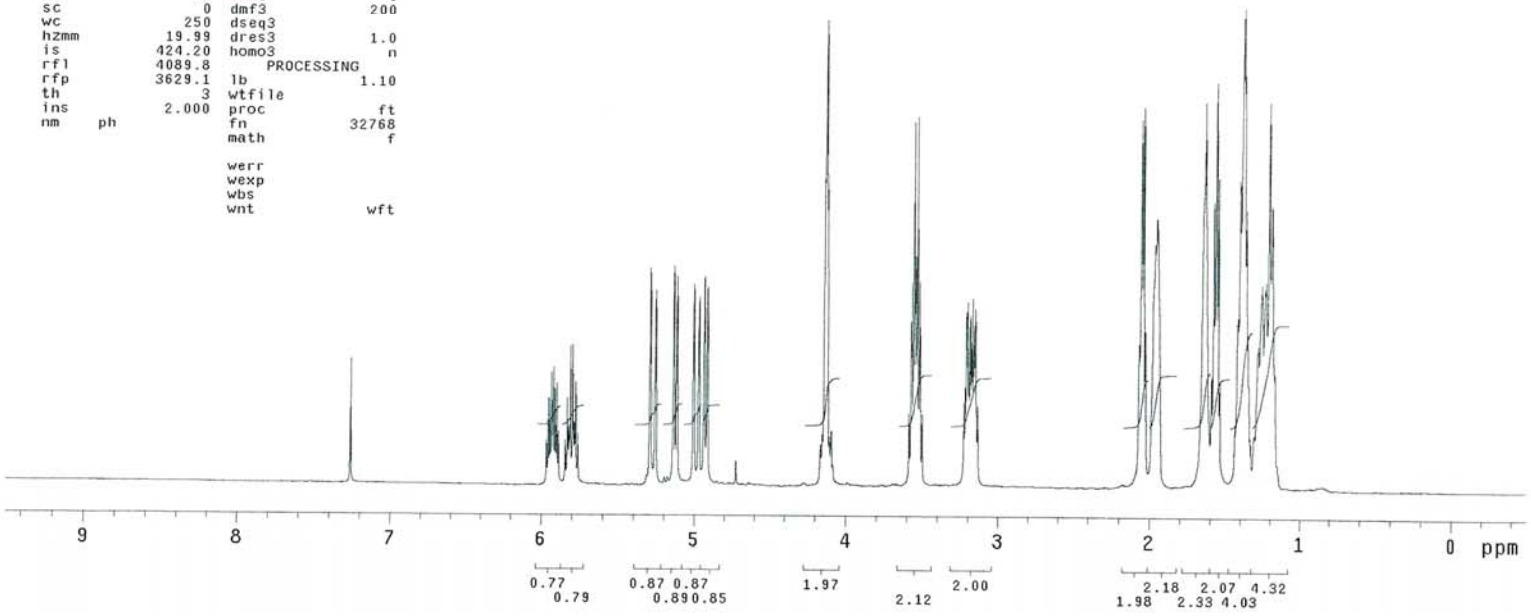
(±)

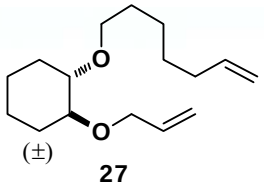
27

WYKELN8093_1H

exp2 s2pul

SAMPLE		DEC. & VT	
date	Apr 14 2011	dfrq	499.874
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	499.875	dm	nnn
tn	H1	dmm	c
at	2.184	dmf	200
np	32768	dseq	
sw	7501.2	dres	1.0
fb	not used	homo	n
bs	4	temp	25.0
ss	2	DEC2	
tpwr	62	dfrq2	0
pw	12.0	dn2	
d1	0	dpwr2	1
tof	800.0	dof2	0
nt	16	dm2	n
ct	16	dmm2	c
alock	n	dmf2	200
gain	not used	dseq2	
FLAGS		dres2	1.0
il	n	homo2	n
in	n	DEC3	
dp	y	dfrq3	0
hs	nn	dn3	
DISPLAY		dpwr3	1
sp	-250.2	dof3	0
wp	4998.6	dm3	n
vs	79	dmm3	c
sc	0	dmf3	200
wc	250	dseq3	
hzmm	19.99	dres3	1.0
is	424.20	homo3	n
rfl	4089.8	PROCESSING	
rfp	3629.1	lb	1.10
th	3	wtfile	
ins	2.000	proc	ft
nm ph		fn	32768
		math	f
		werr	
		wexp	
		wbs	
		wnt	wft

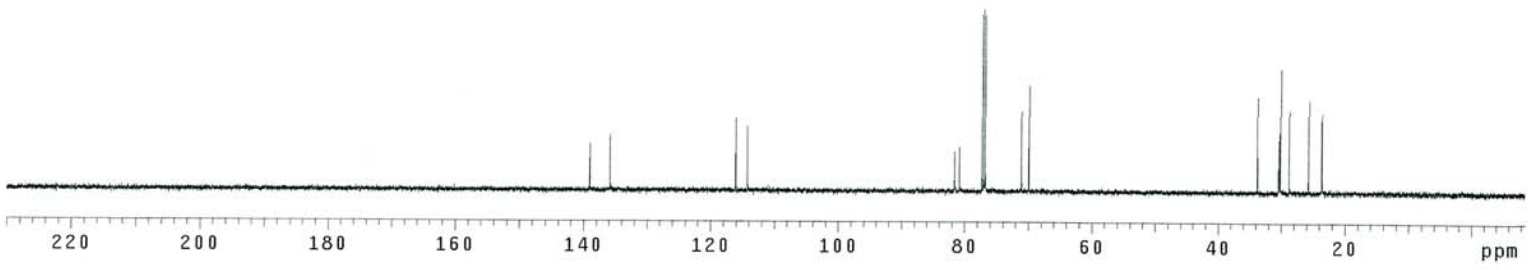


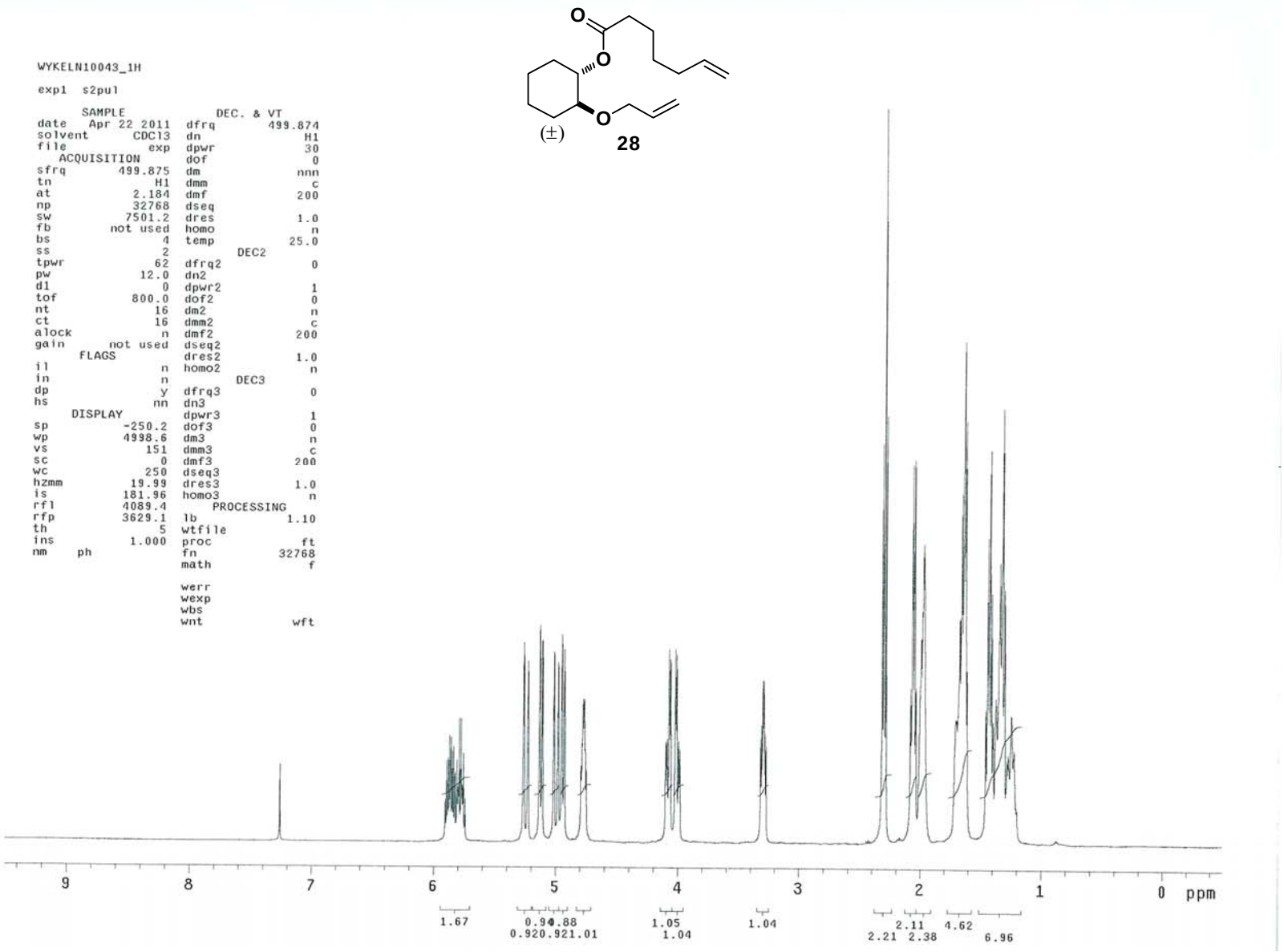


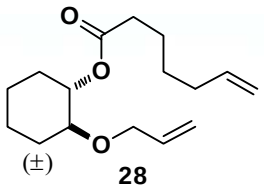
(±)

27

```
WYKELN8093_13C

exp3  s2pul

      SAMPLE
date    Apr 14 2011
solvent       CDCl3
file            exp
   ACQUISITION
sfrq        125.707
tn              C13
at              1.092
np              65536
sw           29996.3
fb          not used
bs                16
tpwr              55
pw               4.8
d1                 0
tof           2000.0
nt                9999
ct                176
alock               n
gain        not used
      FLAGS
il                  n
in                  n
dp                  y
hs                 nn
     DISPLAY
sp            -1087.8
wp           29995.3
vs                30
sc                 0
wc               250
hzmm          119.98
is            500.00
rfl          10767.1
rfp           9678.3
th                 5
ins          100.000
nm  cdc  ph

      DEC. & VT
dfrq         499.874
dn                H1
dpwr              48
dof                0
dm               yyy
dmm                w
dmf             8929
dseq
dres             1.0
homo               n
temp            25.0
      DEC2
dfrq2              0
dn2
dpwr2              1
dof2               0
dm2                n
dmm2               c
dmf2           10000
dseq2
dres2            1.0
homo2              n
      DEC3
dfrq3              0
dn3
dpwr3              1
dof3               0
dm3                n
dmm3               c
dmf3           10000
dseq3
dres3            1.0
homo3              n
     PROCESSING
lb              1.00
wtfile
proc               ft
fn          not used
math               f

werr
wexp
wbs
wnt
```

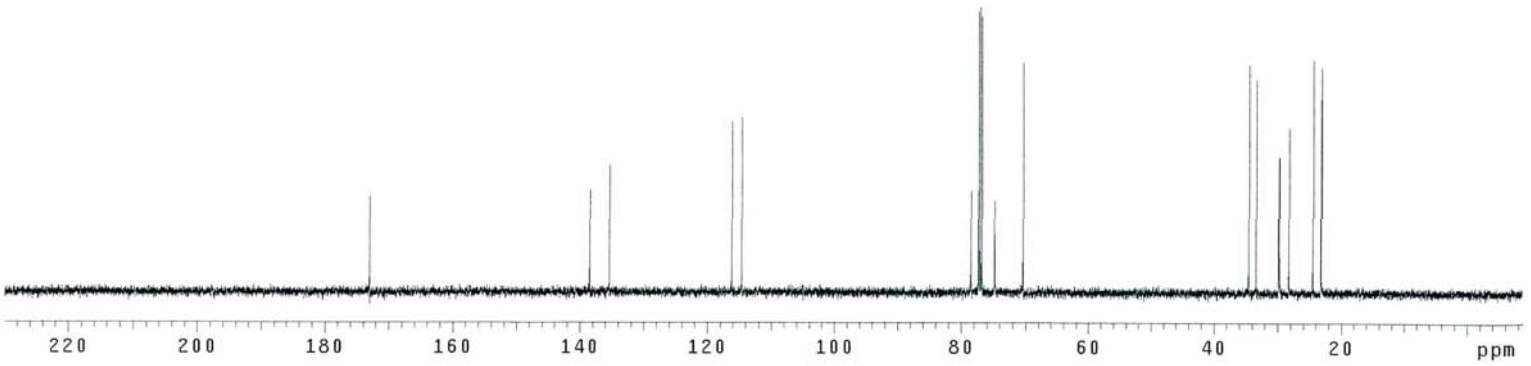


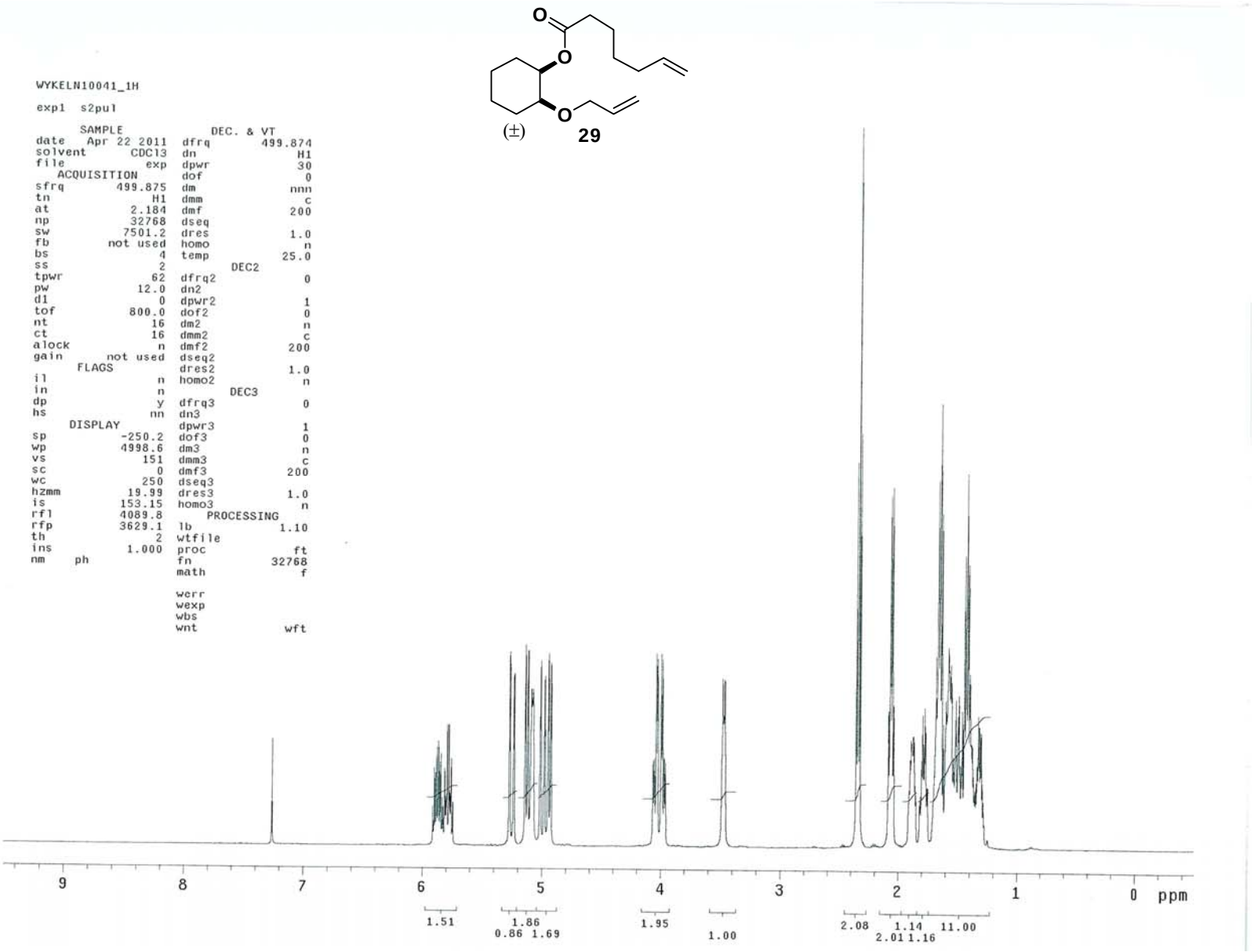

WYKELN10043_1H
exp1 s2pul
SAMPLE
date Apr 22 2011
solvent CDCl3
file exp
ACQUISITION
sfrq 499.875
tn H1
at 2.184
np 32768
sw 7501.2
fb not used
bs 4
ss 2
tpwr 62
pw 12.0
d1 0
tof 800.0
nt 16
ct 16
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -250.2
wp 4998.6
vs 151
sc 0
wc 250
hzmm 19.99
is 181.96
rfl 4089.4
rfp 3629.1
th 5
ins 1.000
nm ph
DEC. & VT
dfrq 499.874
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
temp 25.0
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 200
dseq2
dres2 1.0
homo2 n
DEC3
dfrq3 0
dn3
dpwr3 1
dof3 0
dm3 n
dmm3 c
dmf3 200
dseq3
dres3 1.0
homo3 n
PROCESSING
lb 1.10
Wtfile
proc ft
fn 32768
math f
werr
wexp
wbs
wnt wft
(±)
28
9
8
7
6
5
4
3
2
1
0 ppm
1.67
0.94 0.88
0.92 0.92 1.01
1.05
1.04
1.04
2.11
2.21 2.38
4.62
6.96

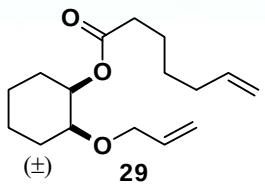


(±) **28**

```
WYKELN10043_13C

exp2  s2pul

      SAMPLE           DEC. & VT
date   Apr 22 2011  dfrq        499.874
solvent      CDCl3  dn               H1
file           exp  dpwr             48
   ACQUISITION      dof               0
sfrq       125.707  dm              yyy
tn              C13 dmm               w
at            1.092 dmf            8929
np            65536 dseq
sw          29996.3 dres             1.0
fb        not used  homo              n
bs              16  temp           25.0
tpwr            55        DEC2
pw             4.8  dfrq2             0
d1               0  dn2
tof         2000.0  dpwr2             1
nt            9999  dof2              0
ct             192  dm2               n
alock            n  dmm2              c
gain     not used   dmf2          10000
       FLAGS        dseq2
il               n  dres2           1.0
in               n  homo2             n
dp               y        DEC3
hs              nn  dfrq3             0
     DISPLAY        dn3
sp         -1089.7  dpwr3             1
wp         29995.3  dof3              0
vs              47  dm3               n
sc               0  dmm3              c
wc             250  dmf3          10000
hzmm        119.98  dseq3
is          500.00  dres3          1.0
rfl         1090.6  homo3             n
rfp              0      PROCESSING
th               3  lb             1.00
ins        100.000  wtfile
nm  cdc  ph         proc              ft
                    fn         not used
                    math              f

                    werr
                    wexp
                    wbs
                    wnt
```

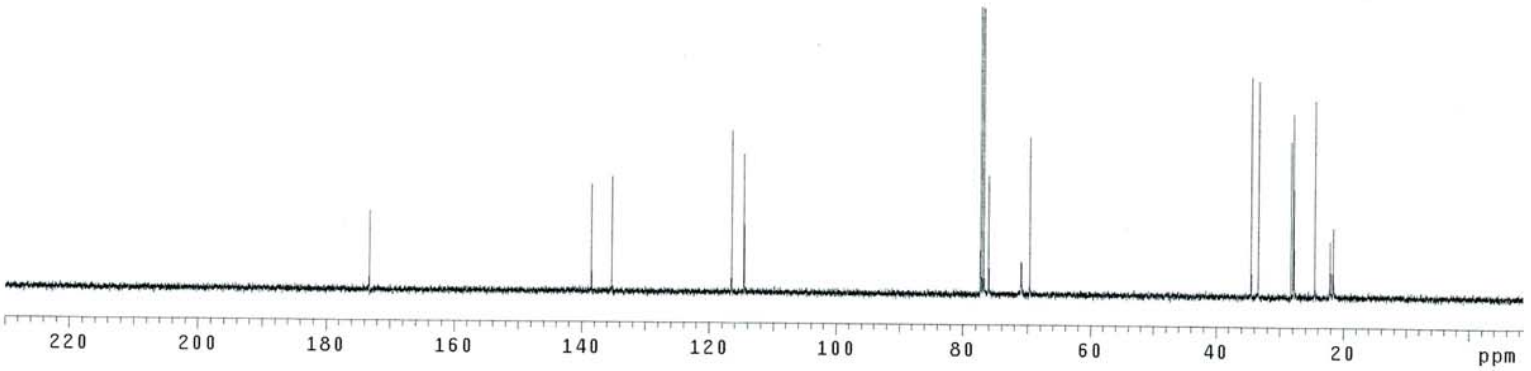


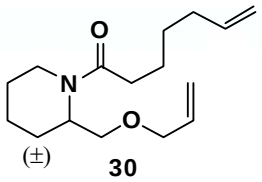

WYKELN10041_1H
exp1 s2pul
SAMPLE
date Apr 22 2011
solvent CDCl3
file exp
ACQUISITION
sfrq 499.875
tn H1
at 2.184
np 32768
sw 7501.2
fb not used
bs 4
ss 2
tpwr 62
pw 12.0
d1 0
tof 800.0
nt 16
ct 16
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -250.2
wp 4998.6
vs 151
sc 0
wc 250
hzmm 19.99
is 153.15
rfl 4089.8
rfp 3629.1
th 2
ins 1.000
nm ph
DEC. & VT
dfrq 499.874
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
temp 25.0
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 200
dseq2
dres2 1.0
homo2 n
DEC3
dfrq3 0
dn3
dpwr3 1
dof3 0
dm3 n
dmm3 c
dmf3 200
dseq3
dres3 1.0
homo3 n
PROCESSING
lb 1.10
wtfile
proc ft
fn 32768
math f
werr
wexp
wbs
wnt wft
O
O
O
(±)
29
9
8
7
6
5
4
3
2
1
0 ppm
1.51
1.86
0.86 1.69
1.95
1.00
2.08
1.14
2.01 1.16
11.00



29

```
WYKELN10041_13C

exp2  s2pul

      SAMPLE
date    Apr 22 2011
solvent       CDCl3
file             exp
   ACQUISITION
sfrq         125.707
tn               C13
at             1.092
np             65536
sw           29996.3
fb           not used
bs                16
tpwr              55
pw               4.8
d1                 0
tof           2000.0
nt                32
ct               384
alock              n
gain         not used
      FLAGS
il                 n
in                 n
dp                 y
hs                nn
    DISPLAY
sp           -1088.7
wp           29995.3
vs                47
sc                 0
wc               250
hzmm          119.98
is            500.00
rfl          10768.0
rfp           9678.3
th                 3
ins          100.000
nm  cdc  ph

      DEC. & VT
dfrq         499.874
dn                H1
dpwr              48
dof                0
dm               yyy
dmm                w
dmf             8929
dseq
dres             1.0
homo               n
temp            25.0
     DEC2
dfrq2              0
dn2
dpwr2              1
dof2               0
dm2                n
dmm2               c
dmf2           10000
dseq2
dres2            1.0
homo2              n
     DEC3
dfrq3              0
dn3
dpwr3              1
dof3               0
dm3                n
dmm3               c
dmf3           10000
dseq3
dres3            1.0
homo3              n
    PROCESSING
lb              1.00
wtfile
proc               ft
fn           not used
math               f

werr
wexp
wbs
wnt
```



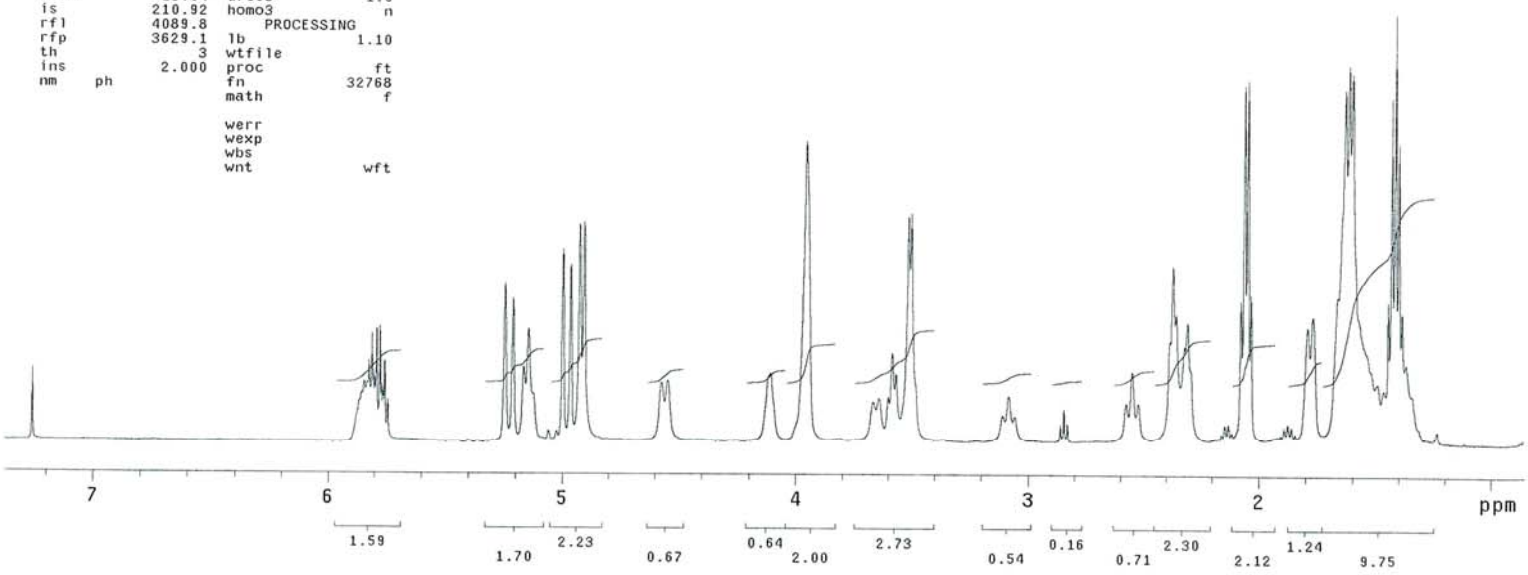


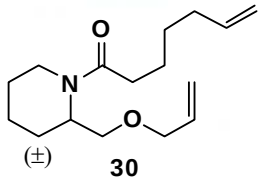
(±) **30**

WYKELN10052_1H

exp1 s2pul

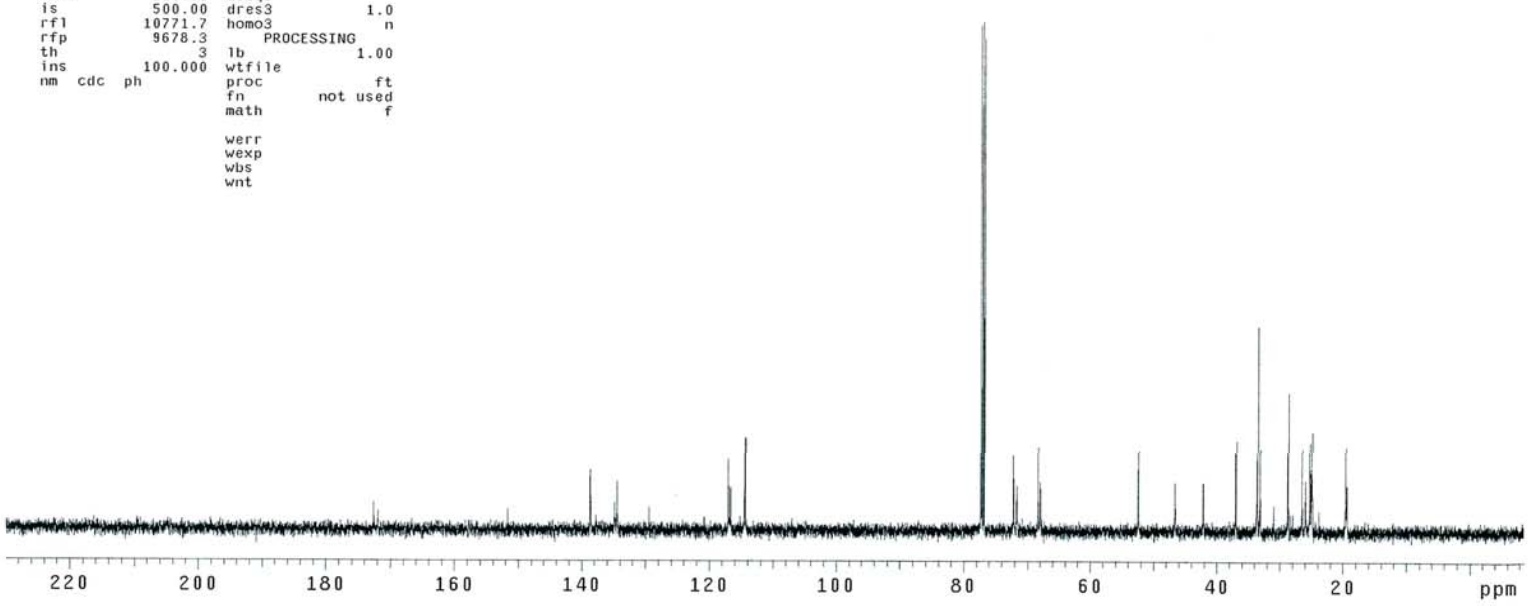
SAMPLE		DEC. & VT	
date	Apr 23 2011	dfrq	499.874
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	499.875	dm	nnn
tn	H1	dmm	c
at	2.184	dmf	200
np	32768	dseq	
sw	7501.2	dres	1.0
fb	not used	homo	n
bs	4	temp	25.0
ss	2	DEC2	
tpwr	62	dfrq2	0
pw	12.0	dn2	
d1	0	dpwr2	1
tof	800.0	dof2	0
nt	16	dm2	n
ct	16	dmm2	c
alock	n	dmf2	200
gain	not used	dseq2	
FLAGS		dres2	1.0
il	n	homo2	n
in	n	DEC3	
dp	y	dfrq3	0
hs	nn	dn3	
DISPLAY		dpwr3	1
sp	428.3	dof3	0
wp	3258.9	dm3	n
vs	71	dmm3	c
sc	0	dmf3	200
wc	250	dseq3	
hzmm	13.04	dres3	1.0
is	210.92	homo3	n
rfl	4089.8	PROCESSING	
rfp	3629.1	lb	1.10
th	3	wtfile	
ins	2.000	proc	ft
nm ph		fn	32768
		math	f
		werr	
		wexp	
		wbs	
		wnt	wft

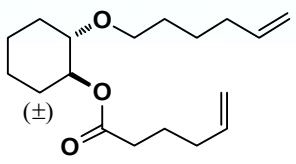




(±) **30**

```
WYKELN10052_13C

exp2  s2pul

      SAMPLE
date    Apr 23 2011
solvent       CDCl3
file            exp
   ACQUISITION
sfrq        125.707
tn              C13
at              1.092
np              65536
sw           29996.3
fb          not used
bs                16
tpwr              55
pw               4.8
d1                0
tof            2000.0
nt              99999
ct                288
alock               n
gain        not used
      FLAGS
il                  n
in                  n
dp                  y
hs                 nn
     DISPLAY
sp            -1092.4
wp            29995.3
vs                 84
sc                  0
wc                250
hzmm           119.98
is             500.00
rfl           10771.7
rfp            9678.3
th                  3
ins           100.000
nm  cdc  ph

      DEC. & VT
dfrq          499.874
dn                 H1
dpwr               48
dof                 0
dm                yyy
dmm                 w
dmf              8929
dseq
dres              1.0
homo                n
temp             25.0
      DEC2
dfrq2               0
dn2
dpwr2               1
dof2                0
dm2                 n
dmm2                c
dmf2            10000
dseq2
dres2             1.0
homo2               n
      DEC3
dfrq3               0
dn3
dpwr3               1
dof3                0
dm3                 n
dmm3                c
dmf3            10000
dseq3
dres3             1.0
homo3               n
     PROCESSING
lb               1.00
wtfile
proc               ft
fn          not used
math                f

werr
wexp
wbs
wnt
```





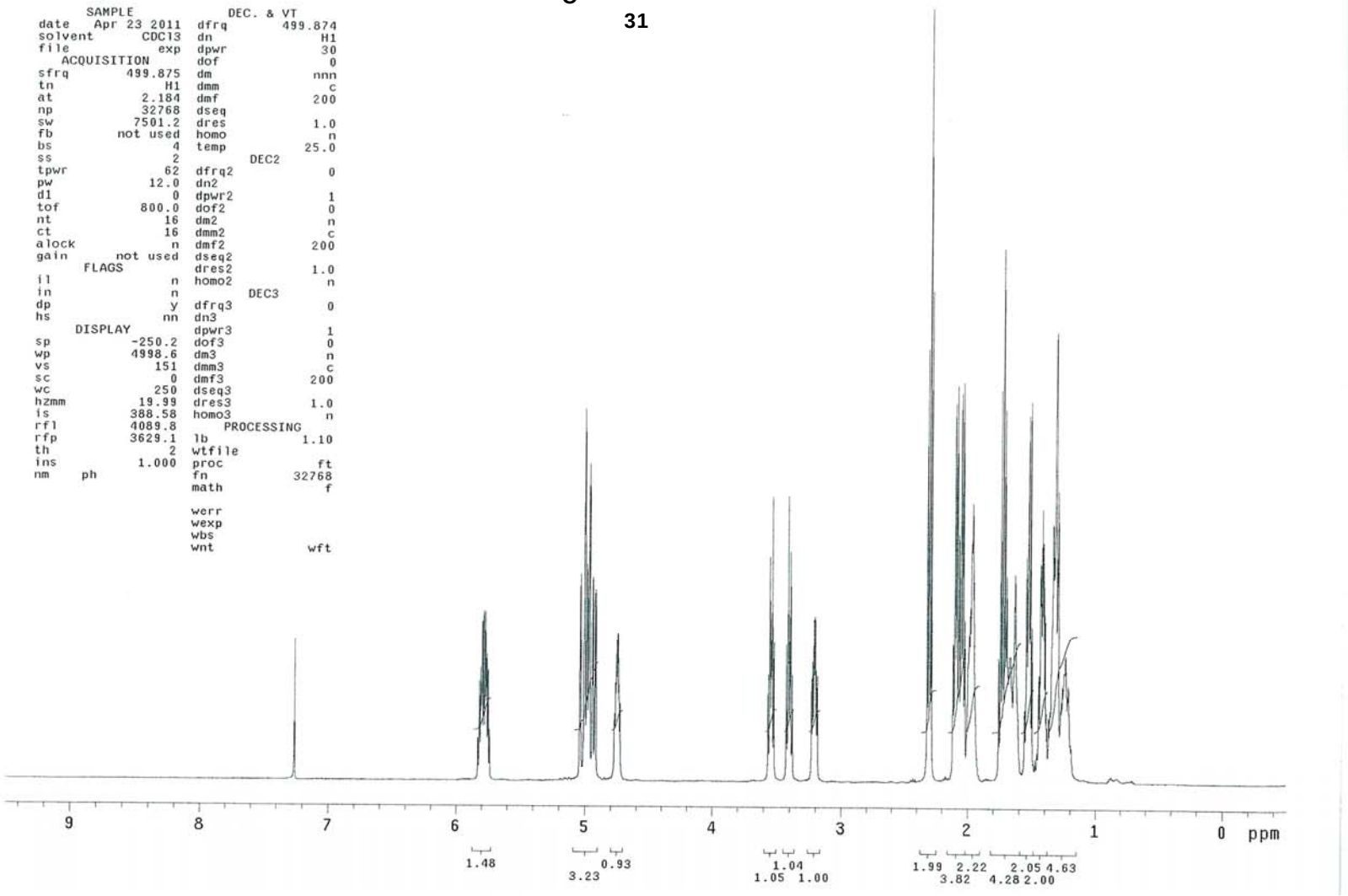
(±)

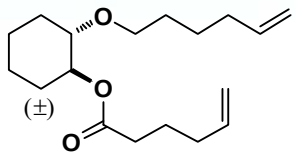
31

WYKELN10044_1H

exp1 s2pul

SAMPLE		DEC. & VT	
date	Apr 23 2011	dfrq	499.874
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	499.875	dm	nnn
tn	H1	dmm	c
at	2.184	dmf	200
np	32768	dseq	
sw	7501.2	dres	1.0
fb	not used	homo	n
bs	4	temp	25.0
ss	2	DEC2	
tpwr	62	dfrq2	0
pw	12.0	dn2	
d1	0	dpwr2	1
tof	800.0	dof2	0
nt	16	dm2	n
ct	16	dmm2	c
alock	n	dmf2	200
gain	not used	dseq2	
FLAGS		dres2	1.0
il	n	homo2	n
in	n	DEC3	
dp	y	dfrq3	0
hs	nn	dn3	
DISPLAY		dpwr3	1
sp	-250.2	dof3	0
wp	4998.6	dm3	n
vs	151	dmm3	c
sc	0	dmf3	200
wc	250	dseq3	
hzmm	19.99	dres3	1.0
is	388.58	homo3	n
rfl	4089.8	PROCESSING	
rfp	3629.1	lb	1.10
th	2	wtfile	
ins	1.000	proc	ft
nm ph		fn	32768
		math	f
		werr	
		wexp	
		wbs	
		wnt	wft



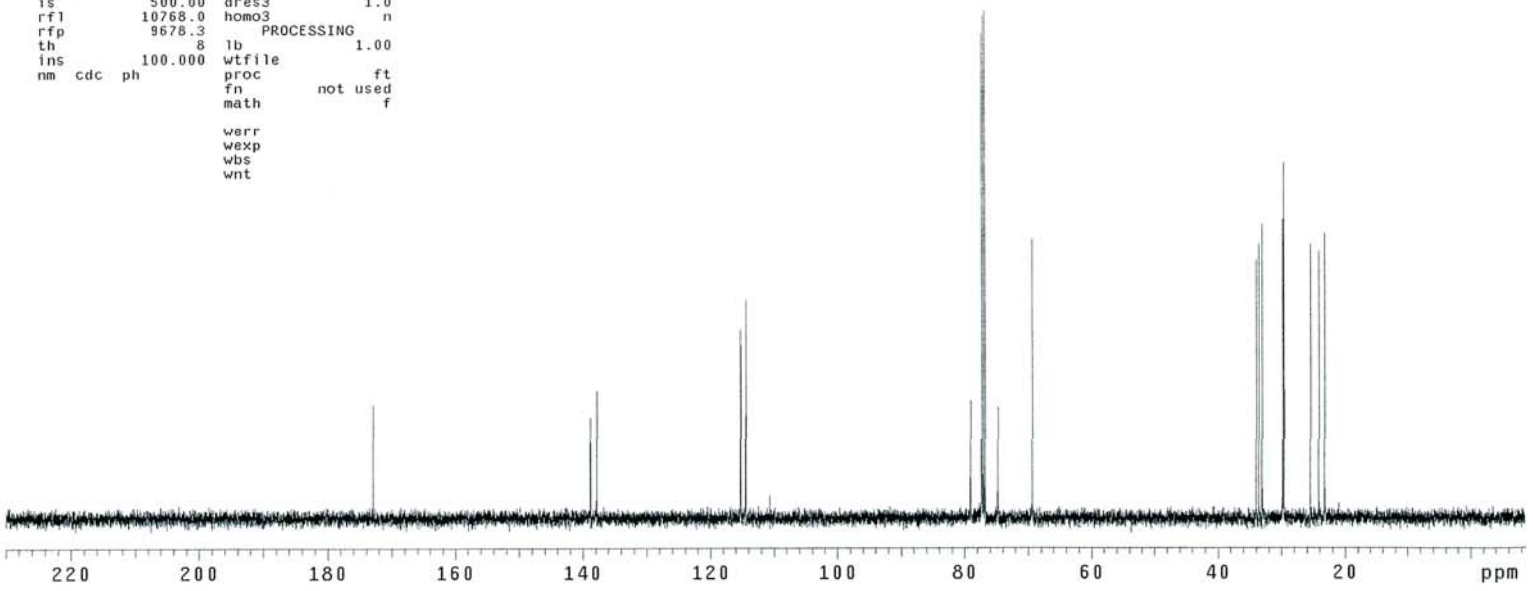


31

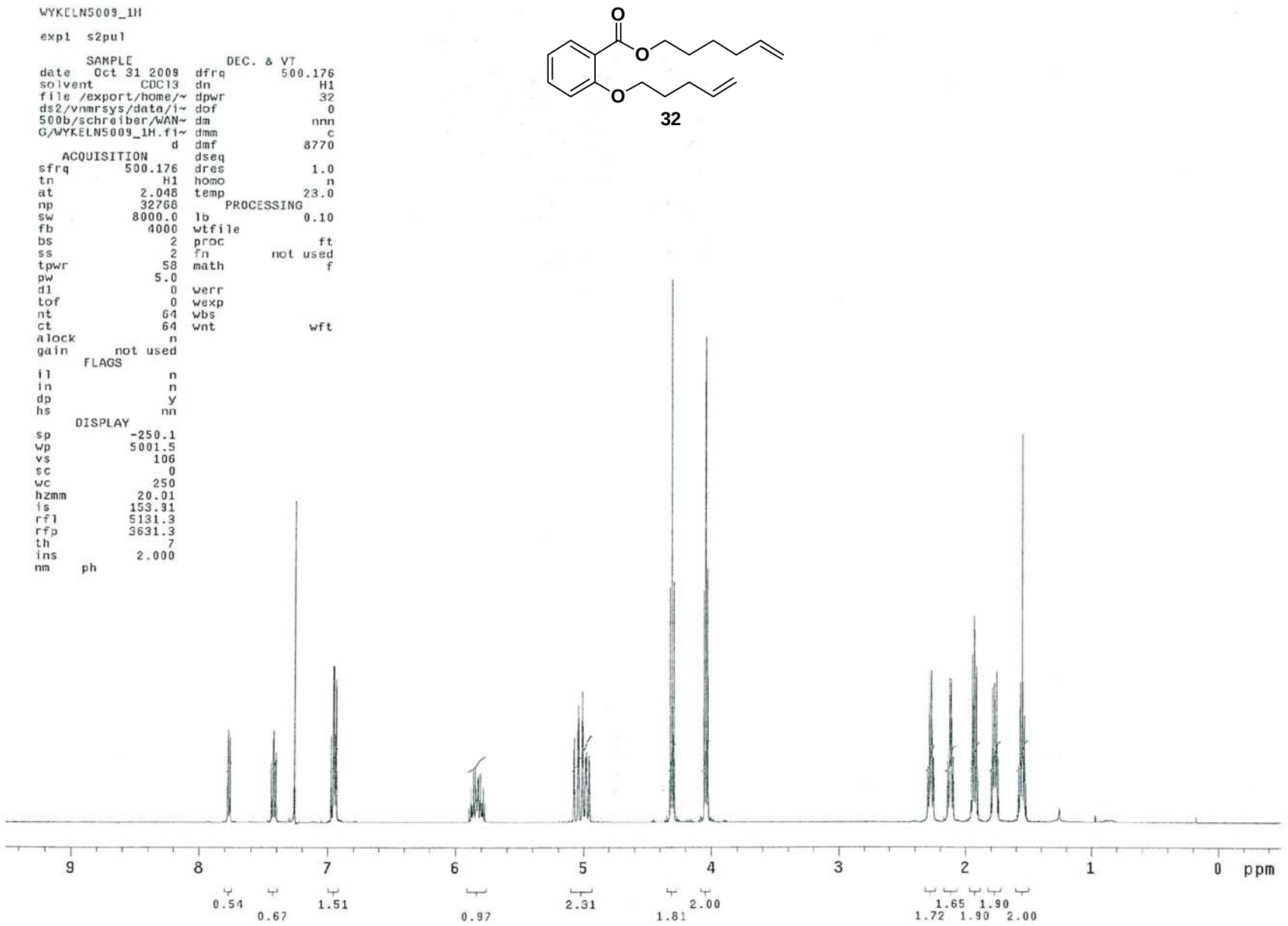
WYKELN10044_13C

exp2 s2pul

SAMPLE		DEC. & VT	
date	Apr 23 2011	dfrq	499.874
solvent	CDCl3	dn	H1
file	exp	dpwr	48
ACQUISITION		dof	0
sfrq	125.707	dm	yyy
tn	C13	dmm	w
at	1.092	dmf	8929
np	65536	dseq	
sw	29996.3	dres	1.0
fb	not used	homo	n
bs	16	temp	25.0
tpwr	55	DEC2	
pw	4.8	dfrq2	0
d1	0	dn2	
tof	2000.0	dpwr2	1
nt	9999	dof2	0
ct	192	dm2	n
alock	n	dmm2	c
gain	not used	dmf2	10000
FLAGS		dseq2	
il	n	dres2	1.0
in	n	homo2	n
dp	y	DEC3	
hs	nn	dfrq3	0
DISPLAY		dn3	
sp	-1088.7	dpwr3	1
wp	29995.3	dof3	0
vs	84	dm3	n
sc	0	dmm3	c
wc	250	dmf3	10000
hzmm	119.98	dseq3	
is	500.00	dres3	1.0
rfl	10768.0	homo3	n
rfp	9678.3	PROCESSING	
th	8	lb	1.00
ins	100.000	wtfile	
nm cdc ph		proc	ft
		fn	not used
		math	f
		werr	
		wexp	
		wbs	
		wnt	



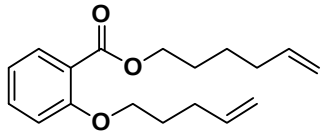
32


WYKELN5009_1H
exp1 s2pul
SAMPLE
date Oct 31 2009
solvent CDCl3
file /export/home/~
ds2/vnmrsys/data/i~
500b/schreiber/WAN~
G/WYKELN5009_1H.fi~
d
ACQUISITION
sfrq 500.176
tn H1
at 2.048
np 32768
sw 8000.0
fb 4000
bs 2
ss 2
tpwr 58
pw 5.0
d1 0
tof 0
nt 64
ct 64
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -250.1
wp 5001.5
vs 106
sc 0
wc 250
hzmm 20.01
is 153.91
rfl 5131.3
rfp 3631.3
th 7
ins 2.000
nm ph
DEC. & VT
dfrq 500.176
dn H1
dpwr 32
dof 0
dm nnn
dmm c
dmf 8770
dseq
dres 1.0
homo n
temp 23.0
PROCESSING
lb 0.10
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt wft
9
8
7
6
5
4
3
2
1
0 ppm
0.54
0.67
1.51
0.97
2.31
1.81
2.00
1.65
1.72
1.90
1.90
2.00

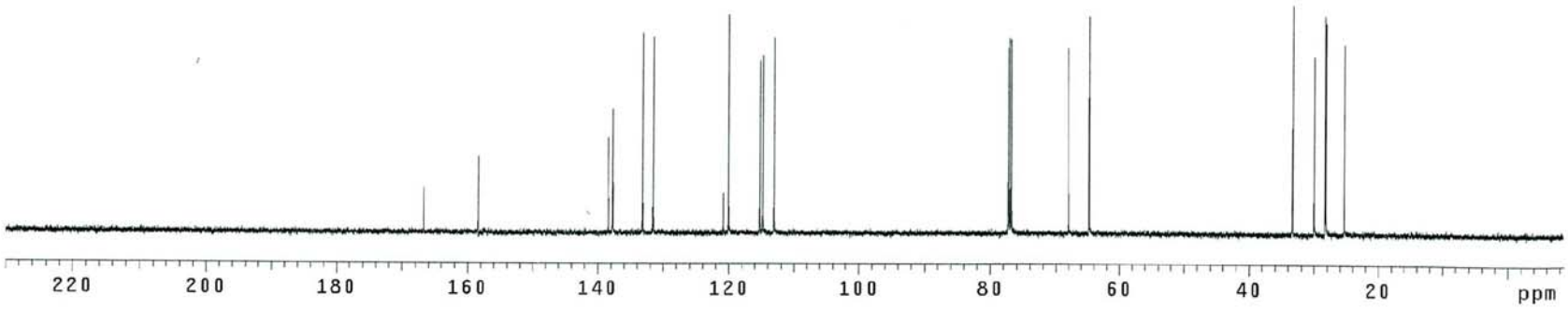
WYKELN5009_13C

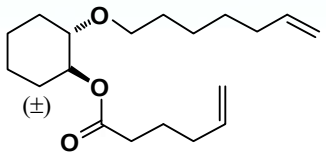
exp2 s2pul

SAMPLE		DEC. & VT	
date	Oct 31 2009	dfrq	499.874
solvent	CDCl3	dn	H1
file	exp	dpwr	49
ACQUISITION		dof	0
sfrq	125.707	dm	yyy
tn	C13	dmm	w
at	1.092	dmf	12000
np	65536	dseq	
sw	29996.3	dres	1.0
fb	not used	homo	n
bs	8	temp	25.0
tpwr	55	DEC2	
pw	4.2	dfrq2	0
d1	0	dn2	
tof	2000.0	dpwr2	1
nt	9999	dof2	0
ct	1368	dm2	n
alock	n	dmm2	c
gain	not used	dmf2	10000
FLAGS		dseq2	
il	n	dres2	1.0
in	n	homo2	n
dp	y	DEC3	
hs	nn	dfrq3	0
DISPLAY		dn3	
sp	-1088.7	dpwr3	1
wp	29995.3	dof3	0
vs	37	dm3	n
sc	0	dmm3	c
wc	250	dmf3	10000
hzmm	119.98	dseq3	
is	500.00	dres3	1.0
rfl	10768.0	homo3	n
rfp	9678.3	PROCESSING	
th	2	lb	1.00
ins	100.000	wtfile	
nm cdc ph		proc	ft
		fn	not used
		math	f
		werr	
		wexp	
		wbs	
		wnt	



32



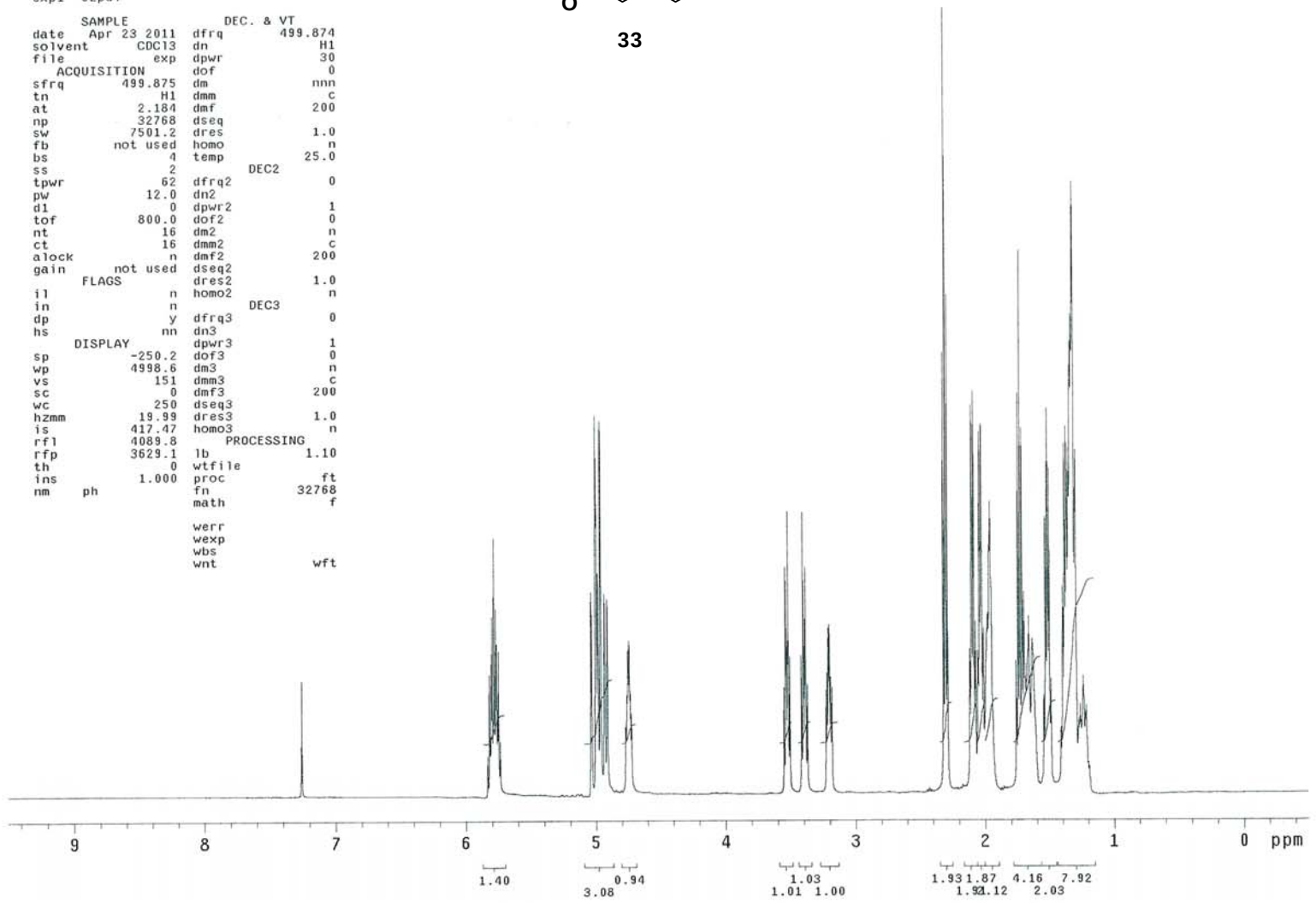


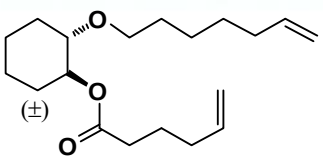
33

WYKELN10045_1H

exp1 s2pul

SAMPLE		DEC. & VT	
date	Apr 23 2011	dfrq	499.874
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	499.875	dm	nnn
tn	H1	dmm	c
at	2.184	dmf	200
np	32768	dseq	
sw	7501.2	dres	1.0
fb	not used	homo	n
bs	4	temp	25.0
ss	2	DEC2	
tpwr	62	dfrq2	0
pw	12.0	dn2	
d1	0	dpwr2	1
tof	800.0	dof2	0
nt	16	dm2	n
ct	16	dmm2	c
alock	n	dmf2	200
gain	not used	dseq2	
FLAGS		dres2	1.0
il	n	homo2	n
in	n	DEC3	
dp	y	dfrq3	0
hs	nn	dn3	
DISPLAY		dpwr3	1
sp	-250.2	dof3	0
wp	4998.6	dm3	n
vs	151	dmm3	c
sc	0	dmf3	200
wc	250	dseq3	
hzmm	19.99	dres3	1.0
is	417.47	homo3	n
rfl	4089.8	PROCESSING	
rfp	3629.1	lb	1.10
th	0	wtfile	
ins	1.000	proc	ft
nm ph		fn	32768
		math	f
		werr	
		wexp	
		wbs	
		wnt	wft



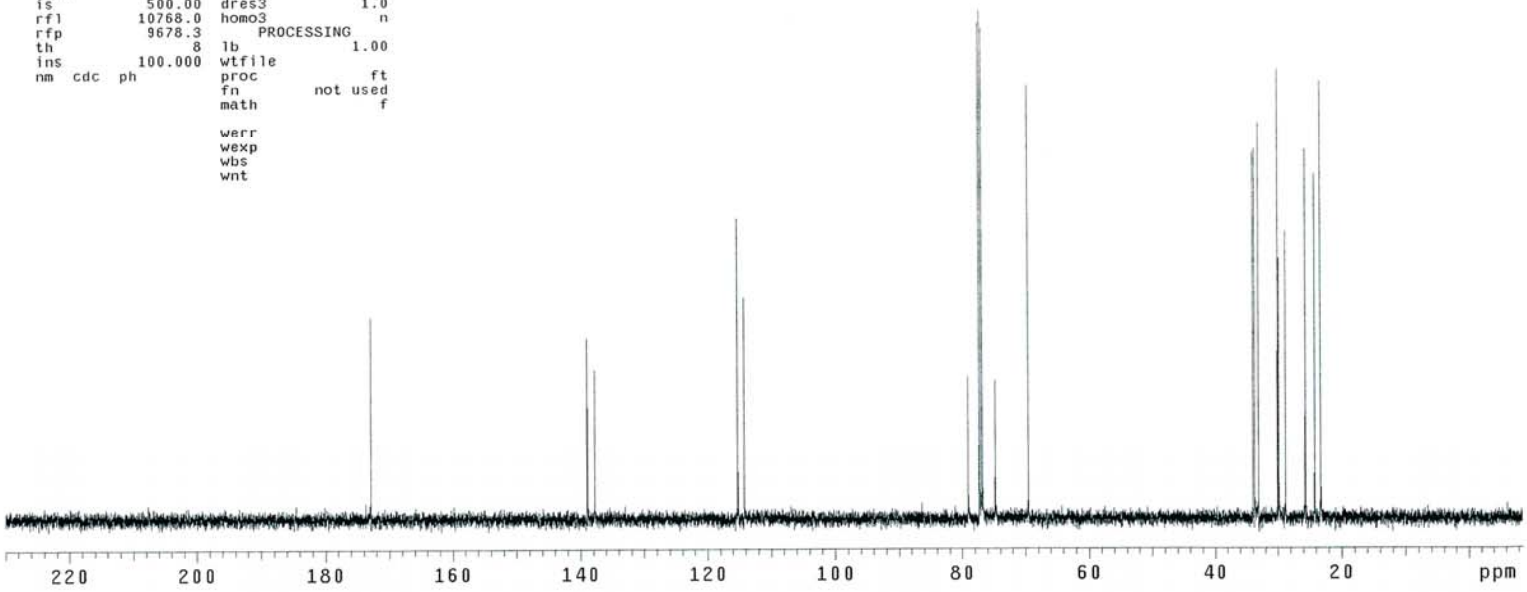


33

WYKELN10045_13C

exp2 s2pul

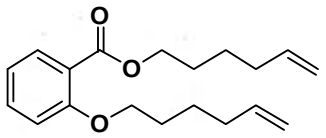
SAMPLE		DEC. & VT	
date	Apr 23 2011	dfrq	499.874
solvent	CDCl3	dn	H1
file	exp	dpwr	48
ACQUISITION		dof	0
sfrq	125.707	dm	yyy
tn	C13	dmm	w
at	1.092	dmf	8929
np	65536	dseq	
sw	29996.3	dres	1.0
fb	not used	homo	n
bs	16	temp	25.0
tpwr	55	DEC2	
pw	4.8	dfrq2	0
d1	0	dn2	
tof	2000.0	dpwr2	1
nt	9999	dof2	0
ct	160	dm2	n
alock	n	dmm2	c
gain	not used	dmf2	10000
FLAGS		dseq2	
il	n	dres2	1.0
in	n	homo2	n
dp	y	DEC3	
hs	nn	dfrq3	0
DISPLAY		dn3	
sp	-1088.7	dpwr3	1
wp	29995.3	dof3	0
vs	84	dm3	n
sc	0	dmm3	c
wc	250	dmf3	10000
hzmm	119.98	dseq3	
is	500.00	dres3	1.0
rfl	10768.0	homo3	n
rfp	9678.3	PROCESSING	
th	8	lb	1.00
ins	100.000	wtfile	
nm cdc ph		proc	ft
		fn	not used
		math	f
		werr	
		wexp	
		wbs	
		wnt	



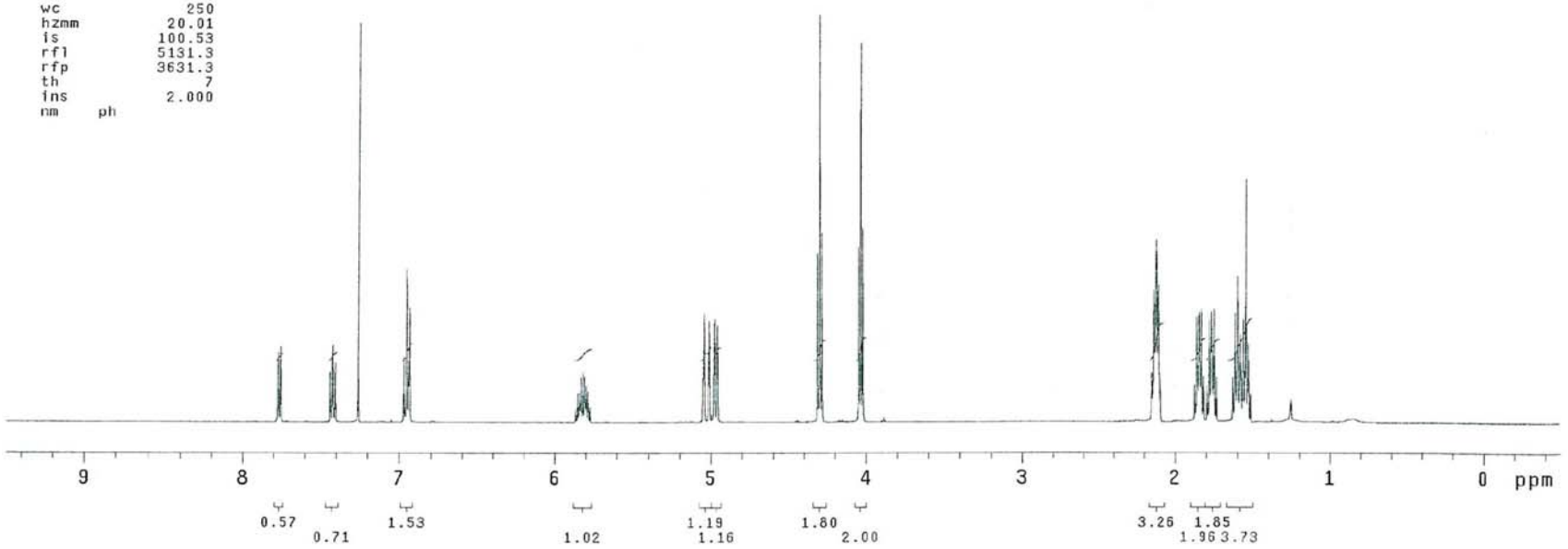
WYKELN5011_byProduct_1H

expl s2pul

SAMPLE		DEC. & VT	
date	Nov 6 2009	dfrq	500.176
solvent	CDCl3	dn	H1
file	/export/home/~	dpwr	32
	ds2/vnmrsys/data/i~	dof	0
	500b/schreiber/WAN~	dm	nnn
	G/WYKELN5011_byPro~	dmm	c
	duct_1H.fid	dmf	8770
ACQUISITION		dseq	
sfrq	500.176	dres	1.0
tn	H1	homo	n
at	2.048	temp	23.0
np	32768	PROCESSING	
sw	8000.0	lb	0.10
fb	4000	wtfile	
bs	8	proc	ft
ss	2	fn	not used
tpwr	58	math	f
pw	5.0		
d1	0	werr	
tof	0	wexp	
nt	64	wbs	
ct	64	wnt	wft
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-250.1		
wp	5001.5		
vs	65		
sc	0		
wc	250		
hzmm	20.01		
is	100.53		
rfl	5131.3		
rfp	3631.3		
th	7		
ins	2.000		
nm	ph		



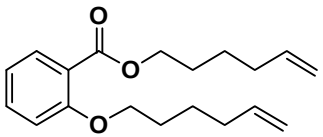
34



WYKELN5011_byProduct_13C

exp2 s2pul

SAMPLE		DEC. & VT	
date	Apr 30 2010	dfrq	499.874
solvent	CDCl3	dn	H1
file	/export/home/~	dpwr	48
	i500c/vnmrsys/data~	dof	0
	/schreiber/WANG/Pu~	dm	yyy
	b1/WYKELN5011_byPr~	dmm	w
	oduct_13C.fid	dmf	10000
ACQUISITION		dseq	
sfrq	125.707	dres	1.0
tn	C13	homo	n
at	1.092	temp	25.0
np	65536	DEC2	
sw	29996.3	dfrq2	0
fb	not used	dn2	
bs	32	dpwr2	1
tpwr	55	dof2	0
pw	4.2	dm2	n
d1	0	dmm2	c
tof	2000.0	dmf2	10000
nt	99999	dseq2	
ct	896	dres2	1.0
alock	n	homo2	n
gain	not used	DEC3	
FLAGS		dfrq3	0
il	n	dn3	
in	n	dpwr3	1
dp	y	dof3	0
hs	nn	dm3	n
DISPLAY		dmm3	c
sp	-1088.7	dmf3	10000
wp	29995.3	dseq3	
vs	27	dres3	1.0
sc	0	homo3	n
wc	250	PROCESSING	
hzmm	5.34	lb	1.00
is	500.00	wtfile	
rfl	10768.0	proc	ft
rfp	9678.3	fn	not used
th	3	math	f
ins	100.000		
nm cdc ph		werr	
		wexp	
		wbs	
		wnt	



34