

Supporting Information

Toward the Total Synthesis of FR901483: Concise Synthesis of the Azatricyclic Skeleton

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Experimental Procedures

General Methods. Unless otherwise noted, solvents and reagents were reagent-grade and used without further purification. Methylene chloride (CH_2Cl_2), diisopropylamine ($i\text{-Pr}_2\text{NH}$) and triethylamine (Et_3N) were freshly distilled from CaH_2 . Tetrahydrofuran (THF) and ether (Et_2O) were dried by passage through two columns of activated neutral alumina. N,N -dimethylformamide (DMF) and toluene were passed through two columns of molecular sieves. Reactions were performed under a nitrogen atmosphere in oven-dried glassware. Reaction temperatures are reported as the temperatures of the bath surrounding the vessel. Nuclear magnetic resonance spectra were acquired at 400 MHz for ^1H and 125 MHz for ^{13}C in CDCl_3 unless otherwise indicated. Chemical shifts are reported in parts per million (ppm, δ), downfield from tetramethylsilane (TMS, $\delta = 0.00$ ppm) and are referenced to the residual solvent (CDCl_3 , $\delta = 7.24$ ppm). The abbreviations s, d, t, q, p, hex, hep, m and comp stand for the resonance multiplicities singlet, doublet, triplet, quartet, pentuplet, hexuplet, heptuplet, multiplet and complex (overlapping multiplets), respectively. Infrared (IR) spectra were recorded as films on sodium chloride plates and reported as wavenumbers (cm^{-1}). Thin-layer chromatography was performed on Merck Kieselgel 60 F₂₅₄ silica gel plates eluting with solvents indicated, visualized by 254 nm UV lamp and stained with ethanolic solution of anisaldehyde. Flash chromatography was performed with ICN Silitech 32-63 D 60A silica gel eluting with solvents as reported. The microwave reactions were performed using CEM Discover System, measuring the temperatures with the built-in thermometer that surrounds the reaction vessel. Compounds **8**¹ and **54**² were prepared according procedures reported in the literature.

(4-Oxooct-7-enyl)carbamic acid *tert*-butyl ester (10). 4-Bromobut-1-ene (28 μL , 0.28 mmol) was added dropwise to a solution of magnesium turnings (78 mg, 3.24 mmol) in THF (4 mL) at 25 °C, and the mixture was stirred for 20 min. An additional portion of 4-bromobut-1-ene (136 μL , 1.34 mmol) was added dropwise, and the mixture was stirred at 25 °C for 1 h. The mixture was then transferred via

cannula to a stirred solution of *N-tert*-butoxycarbonylpyrrolidin-2-one (**9**) (200 mg, 1.08 mmol) in THF (8 mL) at $-78\text{ }^{\circ}\text{C}$, and stirring was continued at $-78\text{ }^{\circ}\text{C}$ for 1.5 h. Isopropyl alcohol (4 mL) was added, and the cooling bath was removed. The mixture was partitioned between saturated aqueous NaHCO_3 (5 mL) and EtOAc (5 mL). The aqueous layer was extracted with EtOAc (2 x 10 mL), and the combined organic layers were washed with brine (10 mL), dried (MgSO_4) and concentrated *in vacuo*. The residue was purified by flash chromatography, eluting with EtOAc/hexanes (1:9) to give 256 mg (98%) of **10** as a clear oil. ^1H NMR δ 5.77 (ddt, $J = 16.8, 10.4, 6.4\text{ Hz}$, 1 H), 5.00 (dq, $J = 16.8, 1.6\text{ Hz}$, 1 H), 4.95 (dq, $J = 10.4, 1.6\text{ Hz}$, 1 H) 4.58 (br s, 1 H), 3.09 (br q, $J = 6.4\text{ Hz}$, 2 H), 2.49 (t, $J = 7.2\text{ Hz}$, 2 H) 2.43 (t, $J = 7.2\text{ Hz}$, 2 H), 2.31–2.28 (m, 2 H), 1.74 (p, $J = 7.2\text{ Hz}$, 2 H), 1.41 (s, 9 H); ^{13}C NMR δ 210.9, 156.2, 137.0, 115.7, 79.9, 41.9, 39.8, 39.1, 28.2, 27.9, 24.2; IR (neat) ν 3360, 2931, 1709, 1522, 1365, 1250, 1171, 828; mass spectrum (CI^+) m/z 242.1775 [$\text{C}_{13}\text{H}_{23}\text{O}_3\text{N} + \text{H}$ requires 242.1756].

2-(But-3-enyl)-1-pyrroline (11). A solution of trifluoroacetic acid (1.08 mL, 14.53 mmol) in CH_2Cl_2 (2 mL) was added to a solution of **10** (500 mg, 2.07 mmol) in CH_2Cl_2 (8 mL) at $0\text{ }^{\circ}\text{C}$. The mixture was stirred at $25\text{ }^{\circ}\text{C}$ for 18 h after which small portions of K_2CO_3 (s) were added at $0\text{ }^{\circ}\text{C}$ with stirring until the pH of the solution was slightly basic (by pH paper). The mixture was carefully concentrated under reduced pressure (300 mmHg) at $20\text{ }^{\circ}\text{C}$, and purified by flash chromatography (neutral alumina) eluting with pentane/ Et_2O (9:1) to give 240 mg (94%) of **11** as a clear oil. ^1H NMR δ 5.84 (ddt, $J = 16.8, 10.4, 6.8\text{ Hz}$, 1 H), 5.05 (dd, $J = 16.8, 1.2\text{ Hz}$, 1 H), 4.97 (dd, $J = 10.4, 1.2\text{ Hz}$, 1 H), 3.78 (tt, $J = 7.6, 1.2\text{ Hz}$, 2 H), 2.46–2.33 (comp, 6 H), 1.86 (p, $J = 7.6\text{ Hz}$, 2 H); This spectrum was consistent with that reported in the literature.³

[(*R*)-4-(*tert*-Butyldiphenylsilyloxy)]pyrrolidin-2-one (23). A solution of imidazole (100 mg, 1.48 mmol), TBDPS-Cl (271 μL , 1.04 mmol), and (*R*)-4-hydroxy-2-pyrrolidinone (100 mg, 0.99 mmol) in DMF (1.5 mL) was stirred for 0.5 h at $0\text{ }^{\circ}\text{C}$. The solution was poured into ice-cold water (5 mL), and the mixture was extracted with EtOAc (3 x 10 mL). The combined organic layers were washed with

brine (2 x 10 mL), dried (Na₂SO₄) and concentrated *in vacuo*. The residue was purified by flash chromatography eluting with MeOH/CH₂Cl₂ (2:98) to give 322 mg (96%) of **23** as a clear oil. ¹H NMR δ 7.62–7.60 (comp, 4 H), 7.45–7.35 (comp, 6 H), 5.81 (br s, 1 H), 4.54–4.48 (m, 1 H), 3.35 (dd, *J* = 6.0, 0.8 Hz, 1 H), 3.25 (dd, *J* = 4.0, 0.8 Hz, 1 H), 2.36 (d, *J* = 2.8 Hz, 1 H), 2.34 (d, *J* = 2.0 Hz, 1 H), 1.04 (s, 9 H). ¹³C NMR δ 175.2, 135.6, 133.1, 130.0, 127.8, 68.8, 51.1, 40.0, 26.8, 18.9; IR (neat) ν 3226, 2920, 1700, 1111, 703; mass spectrum (CI⁺) *m/z* 340.1657 [C₂₀H₂₅O₂N + H requires 340.1655].

[(*R*)-4-(*tert*-Butyldiphenylsilyloxy)-2-oxopyrrolidine-1-carboxylic acid *tert*-butyl ester (24). DMAP (166 mg, 1.36 mmol), Et₃N (379 μL, 2.72 mmol) and di-*tert*-butyldicarbonate (578 mg, 2.85 mmol) were added to a solution of **23** (923 mg, 2.72 mmol) in CH₃CN (30 mL) at 0 °C. The solution was stirred for 24 h at 25 °C whereupon water (50 mL) was added, and the mixture was extracted with EtOAc (3 x 100 mL). The combined organic layers were washed with saturated aqueous NaHCO₃ (100 mL) and brine (100 mL), dried (Na₂SO₄) and concentrated *in vacuo*. The residue was purified by flash chromatography eluting with EtOAc/hexanes (1:5) to give 1.12 g (94%) of **24** as a clear oil. ¹H NMR δ 7.64–7.62 (comp, 4 H), 7.42–7.35 (comp, 6 H), 4.33 (p, *J* = 5.2 Hz, 1 H), 3.65 (d, *J* = 5.2 Hz, 2 H), 2.50 (p, *J* = 5.2 Hz, 2 H), 1.49 (s, 9 H), 1.04 (s, 9 H); ¹³C NMR δ 171.9, 149.8, 135.5, 132.9, 130.0, 127.8, 78.9, 64.7, 54.9, 42.8, 27.9, 26.7, 18.9; IR (neat) ν 2932, 2858, 1787, 1714, 1472, 1317, 1152; mass spectrum (CI⁺) *m/z* 440.2252 [C₂₅H₃₃O₄NSi + H requires 440.2257].

[(*R*)-2-(*tert*-Butyldiphenylsilyloxy)-4-oxooct-7-enyl]carbamic acid *tert*-butyl ester (25). 4-Bromobut-1-ene (17 μL, 0.16 mmol) was added dropwise to a solution of magnesium turnings (48 mg, 1.96 mmol) in THF (1 mL) at 25 °C, and the mixture was stirred for 20 min, after which an additional portion of 4-bromobut-1-ene (83 μL, 0.82 mmol) was added dropwise. The mixture was stirred at 25 °C for 1 h, after which it was transferred via cannula to a stirred solution of **24** (228 mg, 0.49 mmol) in THF (3 mL) at –78 °C. The mixture was stirred at –78 °C for 4 h after which isopropyl alcohol (0.5 mL) was added and the cooling bath removed. The mixture was partitioned between saturated aqueous NaHCO₃

(10 mL) and ethyl acetate (10 mL). The aqueous layer was extracted with ethyl acetate (2 x 20 mL), and the combined organic layers were washed with brine (10 mL), dried (MgSO₄) and concentrated *in vacuo*. The residue was purified by flash chromatography eluting with EtOAc/hexanes (5:95) to give 150 mg (59%) of **25** as a clear oil. ¹H NMR (Cd₃CN) δ 7.64–7.62 (comp, 4 H), 7.44–7.35 (comp, 6 H), 5.77 (ddt, *J* = 16.4, 10.0, 6.4 Hz, 1 H), 4.96–4.89 (m, 2 H), 4.58 (br s, 1 H), 4.31–4.27 (m, 1 H), 3.21–3.15 (m, 1 H), 3.11–3.05 (m, 1 H), 2.53–2.46 (m, 2 H), 2.35–2.24 (m, 2 H), 2.19–2.14 (m, 2 H), 1.40 (s, 9 H), 1.02 (s, 9 H); ¹³C NMR (Cd₃CN) δ 207.7, 159.4, 138.0, 135.8, 133.4, 129.9, 127.8, 118.4, 79.1, 68.9, 45.6, 42.7, 38.4, 28.3, 27.4, 26.9, 19.2; IR (neat) ν 2931, 1716, 1506, 1365, 1249, 1169, 1111; mass spectrum (CI⁺) *m/z* 496.2888 [C₂₉H₄₁O₄NSi + H requires 496.2883].

(4-Oxo-oct-7-enyl)carbamic acid 1-ethylallyl ester (39). 4-Bromobut-1-ene (20 μL, 0.20 mmol) was added dropwise to a solution of magnesium turnings (48 mg, 2.0 mmol) in THF (1 mL) at 25 °C, and the mixture was stirred for 20 min, after which additional 4-bromobut-1-ene (85 μL, 0.80 mmol) was added dropwise. The mixture was stirred at 25 °C for 1 h, after which TMEDA (151 μL, 1.0 mmol) in THF (1 mL) was added, and the mixture was stirred for 10 min. The Grignard reagent (0.72 mL, 0.36 mmol, 0.5 M) thus prepared was transferred via a cannula to a stirred solution of **38** (36 mg, 0.18 mmol) in THF (1 mL) at –78 °C. The mixture was stirred at –78 °C for 2 h, whereupon isopropyl alcohol (0.3 mL) was added, and the cooling bath was removed. The mixture was partitioned between saturated aqueous NaHCO₃ (5 mL) and EtOAc (5 mL). The layers were separated, and the aqueous layer was extracted with EtOAc (2 x 10 mL). The combined organic layers were washed with brine (10 mL), dried (MgSO₄) and concentrated *in vacuo*. The residue was purified by flash chromatography eluting with EtOAc/hexanes (1:9) to give 41 mg (88%) of **39** as a clear oil. ¹H NMR δ 5.76–5.67 (comp, 2 H), 5.19 (dt, *J* = 17.2, 1.2 Hz, 1 H), 5.12 (d, *J* = 10.8 Hz, 1 H), 5.02–5.00 (m, 1 H), 4.94–4.92 (comp, 2 H), 4.74 (br s, 1 H), 3.11 (q, *J* = 6.4 Hz, 2 H), 2.44–2.38 (comp, 4 H), 2.26 (app q, *J* = 6.4 Hz, 2 H), 1.72 (p, *J* = 7.2 Hz, 2 H), 1.69–1.54 (m, 2 H), 0.85 (t, *J* = 7.2 Hz, 3 H); ¹³C NMR δ 209.7, 156.2, 136.9, 136.8,

116.2, 115.3, 76.3, 41.8, 40.3, 39.8, 27.7, 27.4, 23.8, 9.3; IR (neat) ν 3352, 2959, 1713, 1643, 1529, 1251, 922; mass spectrum (CI^+) m/z 254.1758 [$\text{C}_{14}\text{H}_{23}\text{O}_3\text{N} + \text{H}$ requires 254.1756].

(*R*)-4-(*tert*-Butyldiphenylsilyloxy)-2-oxopyrrolidine-1-carboxylic acid 1-ethylallyl ester (40). DMAP (204 mg, 1.67 mmol), Et_3N (350 μL , 2.51 mmol) and 1-ethylallyl chloroformate **36** (408 mg, 2.51 mmol) were added to a solution of **23** (568 mg, 1.67 mmol) in CH_3CN (8 mL) at 0 °C. The cooling bath was removed allowing the mixture to warm to room temperature, and then stirred at 80 °C for 16 h. Saturated NaHCO_3 (50 mL) was added, and the mixture was extracted with EtOAc (3 x 50 mL). The combined organic layers were washed with brine (100 mL), dried (Na_2SO_4), and concentrated *in vacuo*. The residue was purified by flash chromatography eluting with EtOAc /hexanes (5:95) to give 240 mg (36%) of **40** as a clear oil and 340 mg (60%) of the recovered starting material **23**. ^1H NMR δ 7.62–7.59 (m, 4 H), 7.43–7.35 (comp, 6 H), 5.78 (ddt, J = 17.2, 10.4, 6.0 Hz, 1 H), 5.29 (dt, J = 17.2, 1.2 Hz, 1 H), 5.20 (dd, J = 10.4, 1.2 Hz, 1 H), 5.19 (m, 1 H), 4.37 (p, J = 5.2 Hz, 1 H), 3.72–3.71 (m, 2 H), 2.53 (d, J = 5.2 Hz, 2 H), 1.74–1.67 (m, 2 H), 1.04 (s, 9 H), 0.91 (t, J = 7.2 Hz, 3 H). ^{13}C NMR δ 171.7, 150.7, 135.5, 132.8, 132.7, 130.0, 127.9, 117.6, 78.6, 64.8, 54.8, 42.7, 27.0, 26.6, 18.9, 9.2; IR (neat) ν 2931, 1789, 1713, 1350, 1288, 1112, 703; mass spectrum (CI^+) m/z 452.2255 [$\text{C}_{26}\text{H}_{33}\text{O}_4\text{NSi} + \text{H}$ requires 452.2257].

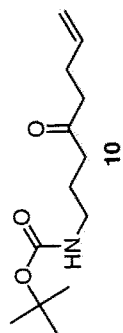
(*R*)-[2-(*tert*-Butyldiphenylsilyloxy)-4-oxo-oct-7-enyl]carbamic acid 1-ethylallyl ester (41). 4-Bromobut-1-ene (49 μL , 0.48 mmol) was added dropwise to a solution of magnesium turnings (88 mg, 3.60 mmol) in THF (1 mL) at 25 °C, and the mixture was stirred for 20 min. An additional portion of 4-bromobut-1-ene (140 μL , 1.32 mmol) was added dropwise and stirring continued for 1 h. TMEDA (272 μL , 1.80 mmol) in THF (1 mL) was then added, and the mixture was stirred for 10 min. The Grignard reagent was transferred via cannula to a stirred solution of **40** (416 mg, 0.92 mmol) in THF (1 mL) at –78 °C. The mixture was stirred at –78 °C for 4 h, whereupon isopropyl alcohol (0.2 mL) was added, and the cooling bath was removed. The mixture was partitioned between saturated NaHCO_3 (15 mL) and

EtOAc (15 mL). The layers were separated, and the aqueous layer was extracted with EtOAc (2 x 15 mL). The combined organic layers were washed with brine (15 mL), dried (MgSO₄) and concentrated *in vacuo*. The residue was purified by flash chromatography (neutral alumina) eluting with EtOAc/hexanes (3:97) to give 270 mg (58%) of **41** as a clear oil. ¹H NMR (CD₃CN) δ 7.70–7.64 (comp, 4 H), 7.48–7.40 (comp, 6 H), 5.89–5.68 (comp, 2 H), 5.47 (br s, 1 H), 5.21–5.10 (comp, 3 H), 4.97–4.89 (comp, 2 H), 4.27 (p, *J* = 5.2 Hz, 1 H), 3.19–3.11 (m, 2 H), 2.60–2.55 (m, 2 H), 2.32–2.29 (m, 2 H), 2.12–2.08 (m, 2 H), 1.60–1.55 (m, 2 H), 1.02 (s, 9 H), 0.91–0.85 (m, 3 H); ¹³C NMR (CD₃CN) δ 208.1, 157.0, 138.2, 137.5, 136.4, 134.3, 130.6, 128.4, 115.8, 115.0, 78.3, 69.0, 47.9, 42.6, 39.5, 33.7, 27.7, 27.0, 26.1, 19.0; IR (neat) ν 3397, 2932, 1716, 1701, 1511, 1244, 1112, 703; mass spectrum (CI⁺) *m/z* 508.2885 [C₃₀H₄₁O₄NSi + H requires 508.2883].

¹ The methyl ester was prepared by methylating (K₂CO₃, MeI, DMF 82% yield) the corresponding acid that was prepared according to: Armstrong, R. J.; Weiler, L. *Can. J. Chem.* **1983**, *61*, 2530–2539.

² Moreno-Dorado, F. J.; Guerra, F. M.; Manzano, F. L.; Aladro, F. J.; Jorge, Z. D.; massanet, G. M. *Tetrahedron Lett.* **2003**, *44*, 6691–6693.

³ Tehrani, K. A.; D'hooghe, M.; De Kimpe, N. *Tetrahedron* **2003**, *59*, 3099–3108.



ss-1-108

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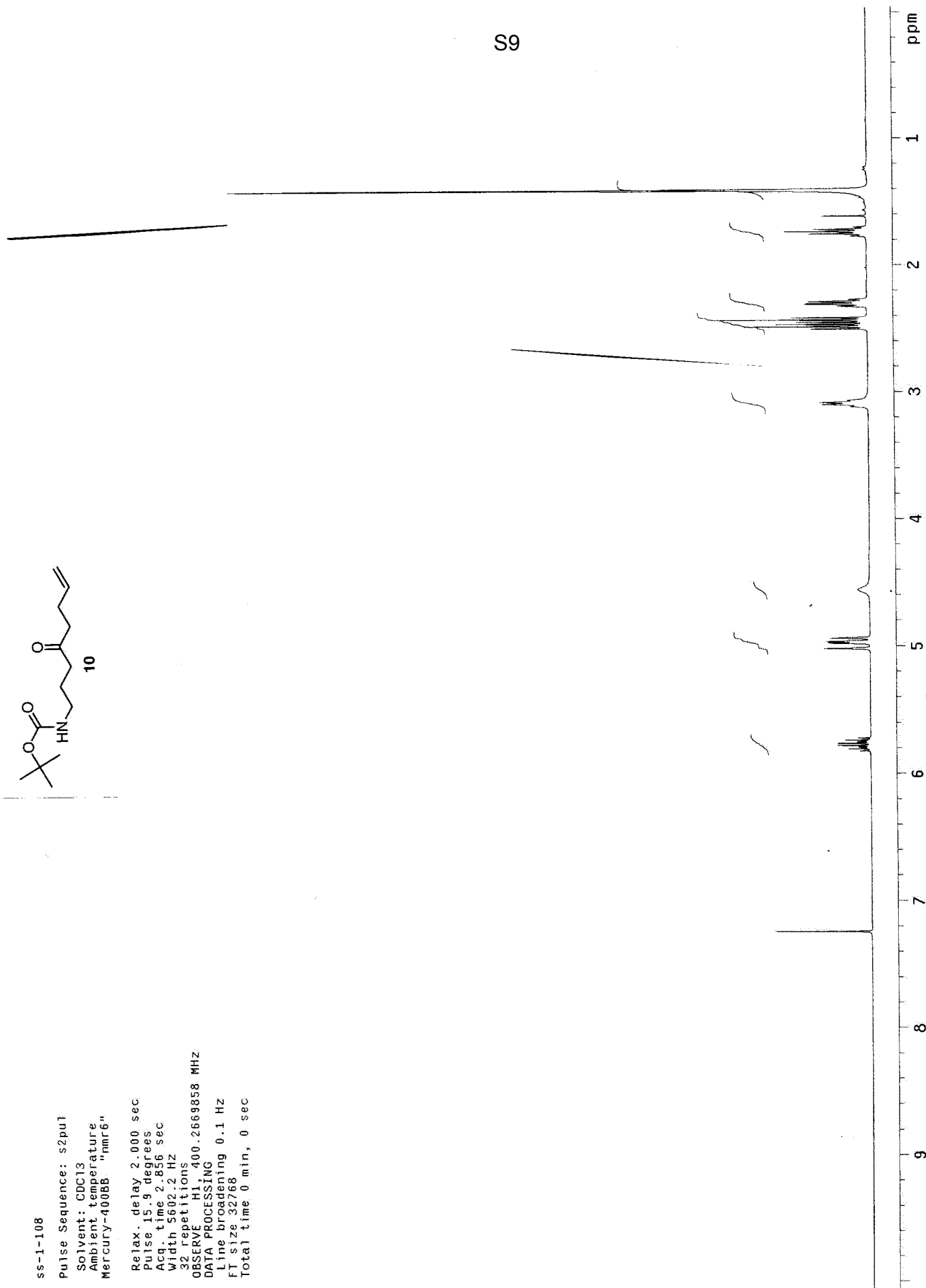
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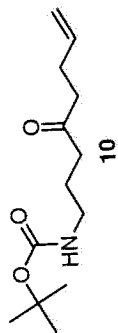
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11

ss-1-135

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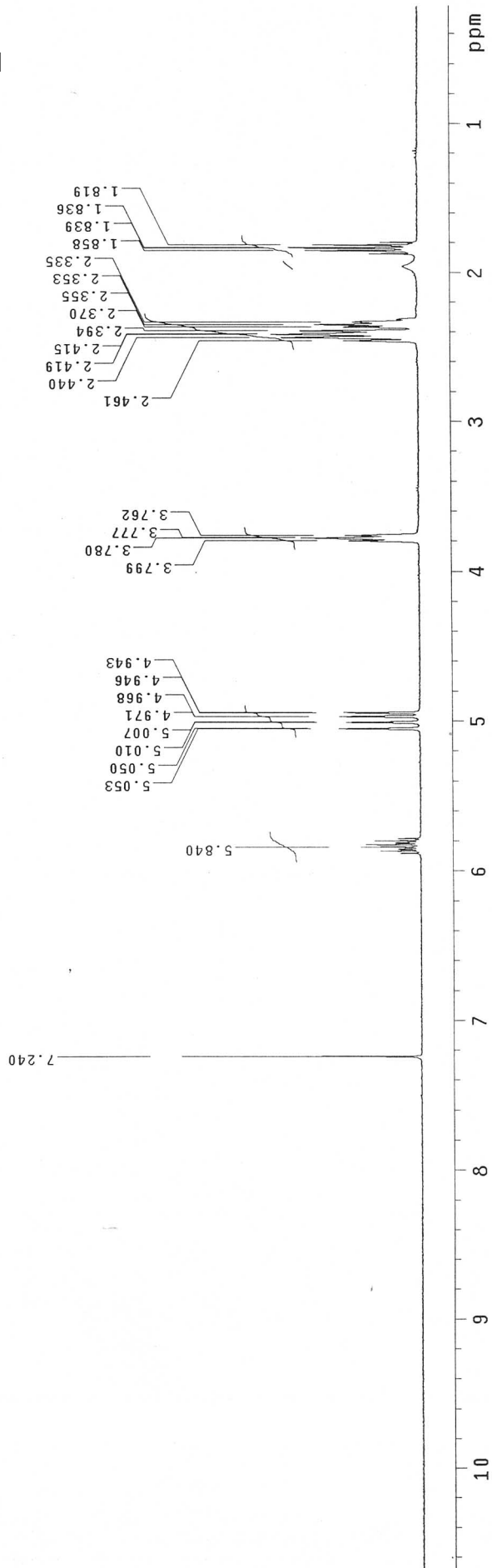
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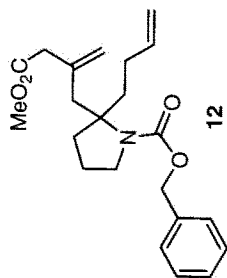
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S11

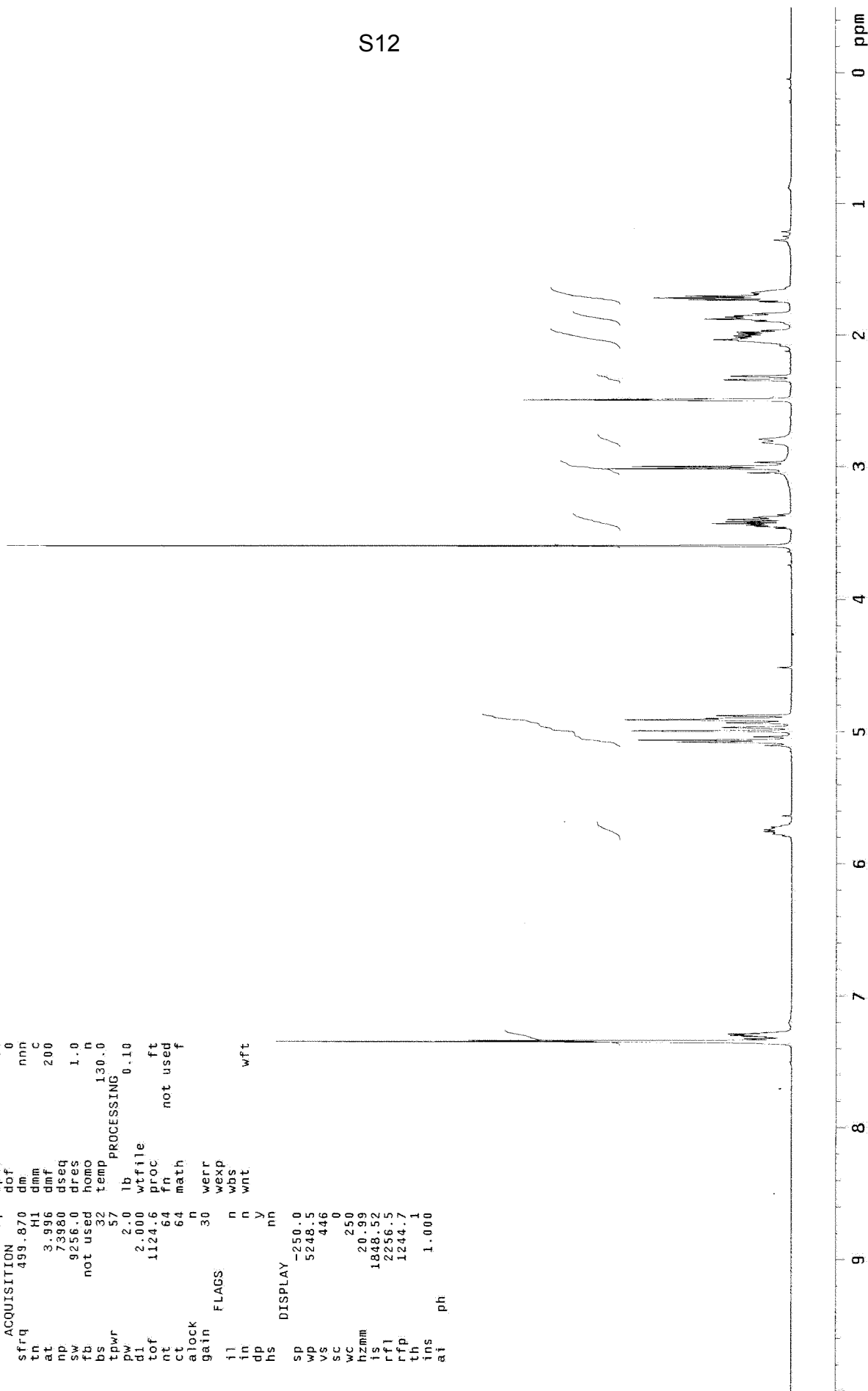


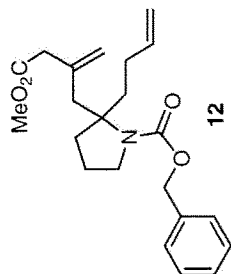


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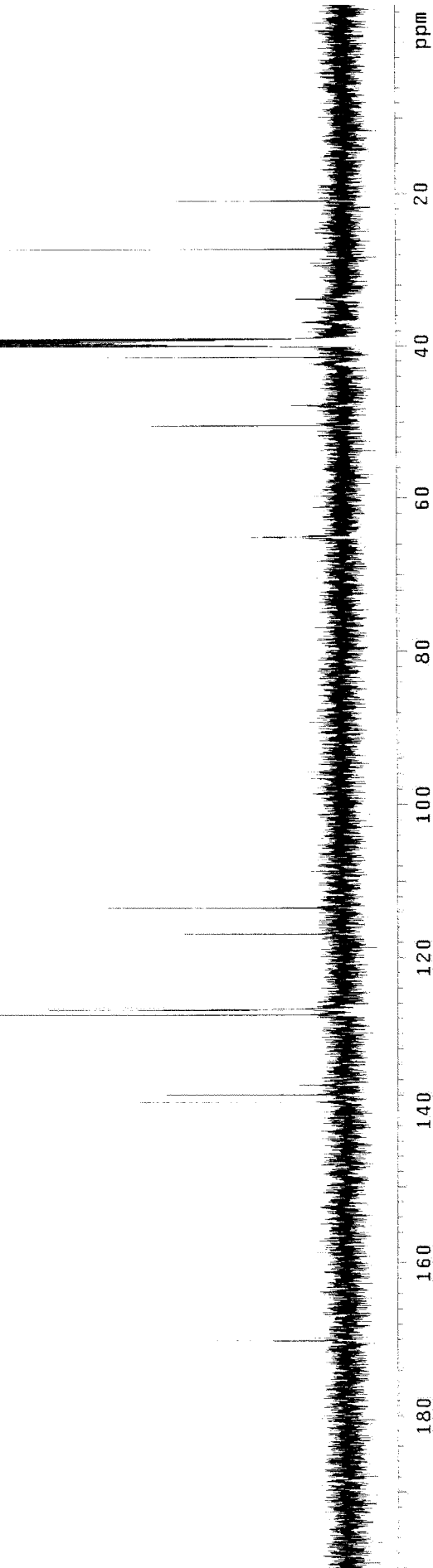


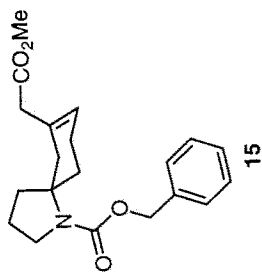
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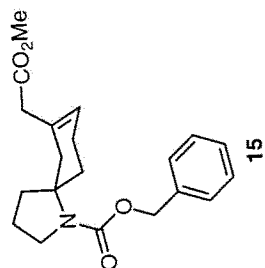


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pw	2.0	lb	0.10
dl	2.000	wtfile	
tof	1124.6	proc	ft
nt	64	fn	not used
ct	64	math	f
alock	n		
gain	30	werr	n
il	n	wexp	n
in	n	wbs	n
dp	y	wnt	wft
hs	nn		
DISPLAY			
sp	-250.0		
wp	5248.5		
vs	425		
sc	0		
wc	250		
hzmm	20.99		
is	3971.28		
rfl	2257.1		
rfp	1244.7		
th	7		
ins	100.000		
ai	ph		

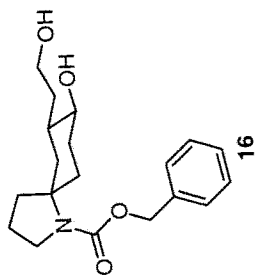
9 8 7 6 5 4 3 2 1 0 ppm



SS-1-240
temp=100c
exp4 s2pu1

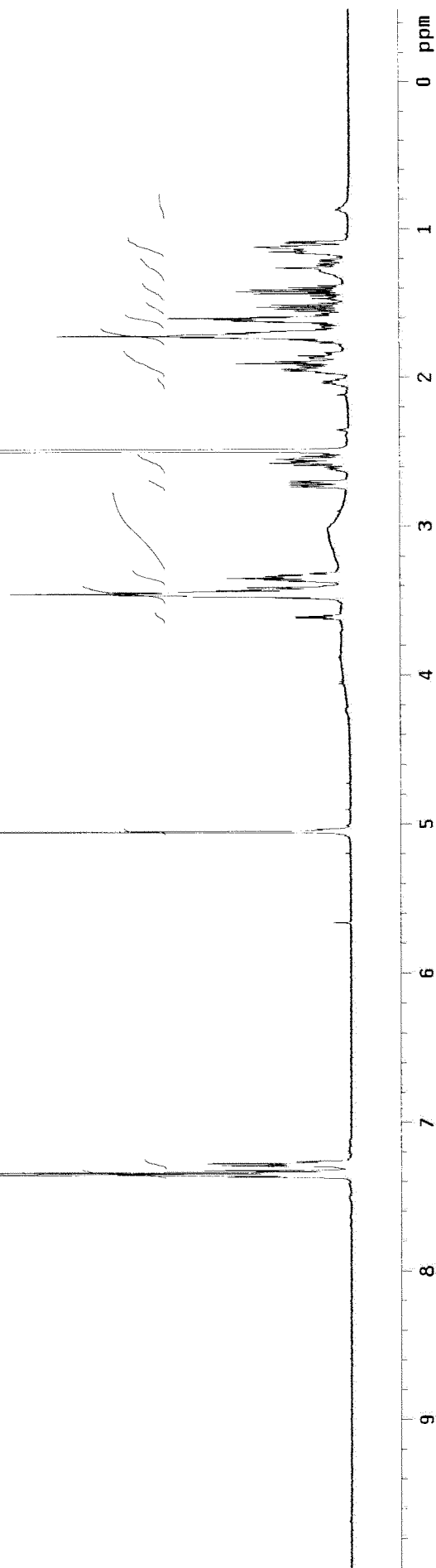
SAMPLE		DEC. & VT	
date	Jan 3 2005	dfrq	499.869
solvent	DMSO	dn	H1
file	exp	dpwr	37
ACQUISITION		dof	0
sfrq	125.706	dm	yyv
tn	1.278	dmm	w
at	85262	dmf	10582
np	33333.3	dseq	
sw	not used	dres	1.0
fb	64	homo	n
bs	53	temp	100.0
tpwr	3.0	PROCESSING	
pw	2.000	lb	1.00
dl	2198.1	wtfile	ft
tof	15000	proc	not used
nt	1858	fn	f
ct	n	math	
alock	60	werr	
gain	n	wexp	
il	n	wbs	
in	n	wnt	
dp	y		
hs	nn		
DISPLAY			
sp	-628.5		
wp	25766.5		
vs	2752		
sc	0		
wc	250		
hzm	103.07		
is	500.00		
rfl	7646.7		
rfl	4964.6		
th	1		
ins	100.000		
ai	cdc		
ph			

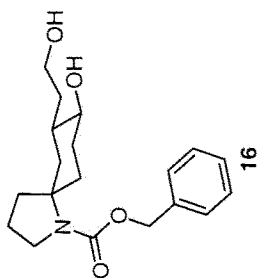
180 160 140 120 100 80 60 40 20 ppm



SS-1-231
temp = 100c
exp2 s2put

SAMPLE		DEC. & VT	
date	Dec 6 2004	dfrq	499.869
solvent	DMSO	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	499.870	dm	nnn
tn	H1	dmm	C
at	3.996	dmf	200
np	73980	dseq	1.0
sw	9256.0	dres	n
fb	not used	homo	100.0
bs	32	temp	100.0
tpwr	57	PROCESSING	
pw	2.0	lb	0.10
dl	2.000	wtfile	ft
tof	1124.6	proc	not used
nt	64	fn	f
ct	64	math	
alock	n	werr	
gain	30	wexp	
il	n	wbs	
in	n	wnt	
dp	y		
hs	nn		
DISPLAY			
sp	-250.0		
wp	5248.5		
vs	1301		
sc	0		
wc	250		
hzmm	20.99		
is	2688.66		
rfl	2256.8		
rfl	1244.7		
th	7		
ins	1.000		
ai	ph		



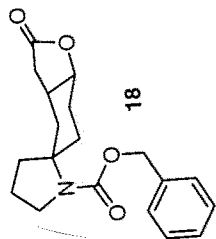


SS-1-231
temp = 100c

exp4 s2pu1

SAMPLE		DEC. & VT	
date	Dec 6 2004	dfrq	499.869
solvent	DMSO	dn	H1
file		dpwr	37
ACQUISITION		dof	0
sfrq	125.706	dm	yyy
tn	C13	dmm	w
at	1.279	dmf	10582
np	85262	dseq	
sw	33333.3	dres	1.0
fb	not used	homo	n
bs	64	temp	100.0
tpwr	53	PROCESSING	
pw	3.0	lb	1.00
d1	2.000	wtfile	
tof	2138.1	proc	ft
nt	2000	fn	not used
ct	2000	math	f
alock	n		
gain	60	werr	
FLAGS		wexp	
il	n	wbs	
in	n	wnt	
dp	y		
hs	nn		
DISPLAY			
sp	-628.5		
wp	25766.5		
vs	5242		
sc	0		
wc	250		
hzmm	103.07		
is	500.00		
rfl	7645.2		
rfl	4964.8		
th	68		
ins	100.000		
ai	cdc	ph	

180 160 140 120 100 80 60 40 20 ppm

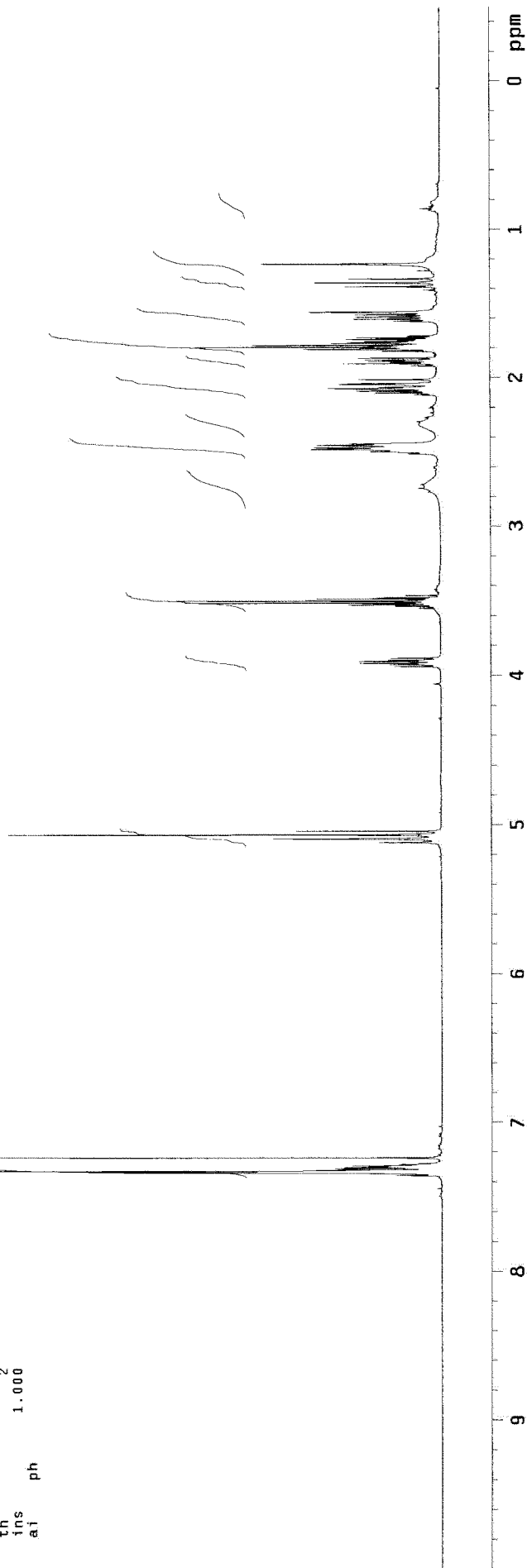


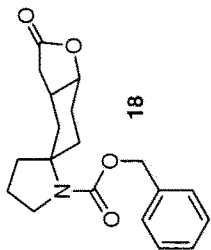
18

SS-1-265

expl s2pul

SAMPLE
 date Jan 10 2005
 solvent CDCl3
 file exp
 ACQUISITION
 sfrq 499.868
 tn H1
 at 3.996
 np 73980
 sw 9256.0
 fb not used
 bs 32
 tpwr 57
 pw 2.0
 d1 2.000
 tof 1124.6
 nt 64
 ct 64
 alock n
 gain 30
 FLAGS
 il n
 in n
 dp n
 hs y
 DEC. & VT
 dfrq 499.867
 dn H1
 dpwr 30
 dof 0
 dm nnn
 dmm C
 dmf 200
 dseq 1.0
 dres n
 homo 27.0
 temp 32
 PROCESSING
 lb 0.10
 wtfile
 proc ft
 fn not used
 math f
 werr n
 wexp n
 wbs n
 wnt y
 wft
 DISPLAY
 sp -250.0
 wp 5248.5
 vs 300
 sc 0
 wc 250
 hzmm 20.99
 is 2628.06
 rfl 4844.7
 rfp 3619.0
 th 2
 ins 1.000
 ai ph





18

SS-1-265
exp4 s2put

date	Jan 10 2005	DEC. & VT	499.867
solvent	CDCl3	dn	H1
file	exp	dpwr	37
ACQUISITION		dof	0
sfrq	125.705	dm	yyv
tn	1.279	dmm	w
at	85262	dmf	10582
np	33333.3	dseq	
sw	not used	dres	1.0
fb	not used	homo	27.0
bs	64	temp	27.0
tpwr	53	PROCESSING	
pw	3.0	lb	1.00
dl	2.000	wtfile	
tof	2198.1	proc	ft
nt	6000	fn	not used
ct	1299	math	f
alock	n	werr	
gain	60	wexp	
il	n	wbs	
in	n	wnt	
hs	nn		
DISPLAY			
sp	-628.6		
wp	25766.5		
vs	1808		
sc	0		
wc	250		
hzmm	103.07		
is	500.00		
rfl	12238.1		
rff	9678.2		
th	2		
ins	100.000		
ai	cdc		
ph			

180 160 140 120 100 80 60 40 20 ppm



19

SS-2-149
exp1 s2pu1

SAMPLE		DEC. & VT	
date	Jun 16 2005	dfrq	499.869
solvent	cd3od	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	499.870	dm	nnn
tn	H1	dmm	C
at	3.996	dmf	200
np	73880	dseq	
sw	9256.0	dres	1.0
fb	not used	homo	n
bs	32	temp	27.0
tpwr	57	PROCESSING	
pw	2.0	lb	0.10
d1	2.000	wtfile	
tof	1124.6	proc	ft
nt	64	fn	not used
ct	64	math	f
alock	n	werr	
gain	30	wexp	
FLAGS		wbs	
il	n	wnt	wft
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-250.1		
wp	5248.5		
vs	1151		
sc	0		
wc	250		
hzmm	20.99		
is	5030.16		
rfl	2667.7		
rff	1649.6		
th	3		
ins	1.000		
at			
	ph		

9 8 7 6 5 4 3 2 1 0 ppm



19

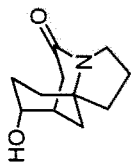
SS-2-149

exp4 s2pul

SAMPLE		DEC. & VT	
date	Jun 16 2005	dfrq	499.869
solvent	cd3od	dn	H1
file	exp	dpwr	37
ACQUISITION		dof	0
sfrq	125.706	dm	YVY
tn	C13	dmm	W
at	1.279	dmf	10582
np	85262	dseq	
sw	33333.3	dres	1.0
fb	not used	homo	n
bs	64	temp	27.0
tpwr	53	PROCESSING	
pw	3.0	lb	1.00
d1	2.000	wtfile	
tof	2198.1	proc	ft
nt	10000	fn	not used
ct	10000	math	f
alock	n	werr	
gain	60	wexp	
FLAGS		wbs	
il	n	wnt	
in	n		
dp	Y		
hs	mn		
DISPLAY			
sp	-628.7		
wp	28280.1		
vs	8453		
sc	0		
wc	250		
hzm	113.12		
is	500.00		
rfl	8538.8		
rff	6158.9		
th	5		
ins	100.000		
ai	cdc		
	ph		

S21

200 180 160 140 120 100 80 60 40 20 ppm



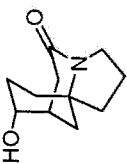
20

SS-2-62-c6d6
 exp1 s2pu1
 SAMPLE 6 2005
 date Apr 6 2005
 solvent Benzene
 file exp
 ACQUISITION
 sfrq 499.868
 tn H1
 at 3.996 dm
 np 73980 dmf
 sw 9256.0 dres
 fb not used
 bs 32
 temp 27.0
 tpwr 57
 pw 2.0 lb
 dl 2.000 wtfile
 tof 1124.6 proc
 nt 64 fn
 ct 64 math
 alock n
 gain 30
 flags
 il n
 in n
 dp n
 hs y
 sp -249.9
 wp 5248.5
 vs 435
 sc 0
 wc 250
 hzmm 20.99
 ts 7020.77
 rfl 999.3
 rfp 0
 th 5
 ins 1.000
 af ph

DEC. & VT
 dfrq 499.867
 dn H1
 dpwr 30
 dof 0
 dm nnn
 dmm C
 dmf 200
 dres 1.0
 homo n
 temp 27.0
 PROCESSING
 lb 0.10
 wtfile
 proc ft
 fn not used
 math f
 werr
 wexp
 wbs
 wnt wft

DISPLAY
 sp -249.9
 wp 5248.5
 vs 435
 sc 0
 wc 250
 hzmm 20.99
 ts 7020.77
 rfl 999.3
 rfp 0
 th 5
 ins 1.000
 af ph

9 8 7 6 5 4 3 2 1 0 ppm



20

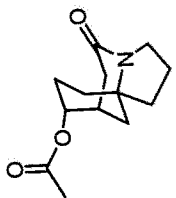
S23

ss-2-62-c6d6
exp4 s2pul

date	6 2005	DEC. & VT	dfrq	499.867
solvent	Benzene	dn	H1	
file	exp	dpwr	37	
ACQUISITION		dof	0	
sfrq	125.705	dm	yvy	
tn	C13	dmm	w	
at	1.279	dmf	10582	
np	85262	dseq		
sw	33333.3	dres	1.0	
fb	not used	homo	n	
bs	64	temp	27.0	
tpwr	53	PROCESSING		
pw	3.0	lb	1.00	
di	2.000	wtfile		
tof	2198.1	proc	ft	
nt	15000	fn	not used	
ct	15000	math	f	
alock	n	werr		
gain	60	wexp		
il	n	wbs		
in	n	wnt		
dp	v			
hs	nn			
DISPLAY				
sp	-628.6			
wp	25766.5			
vs	3940			
sc	0			
wc	250			
hzm	103.07			
is	500.00			
rfl	18604.0			
rffp	16088.4			
th	1			
ins	100.000			
ai	ph			

180 160 140 120 100 80 60 40 20 ppm

JW

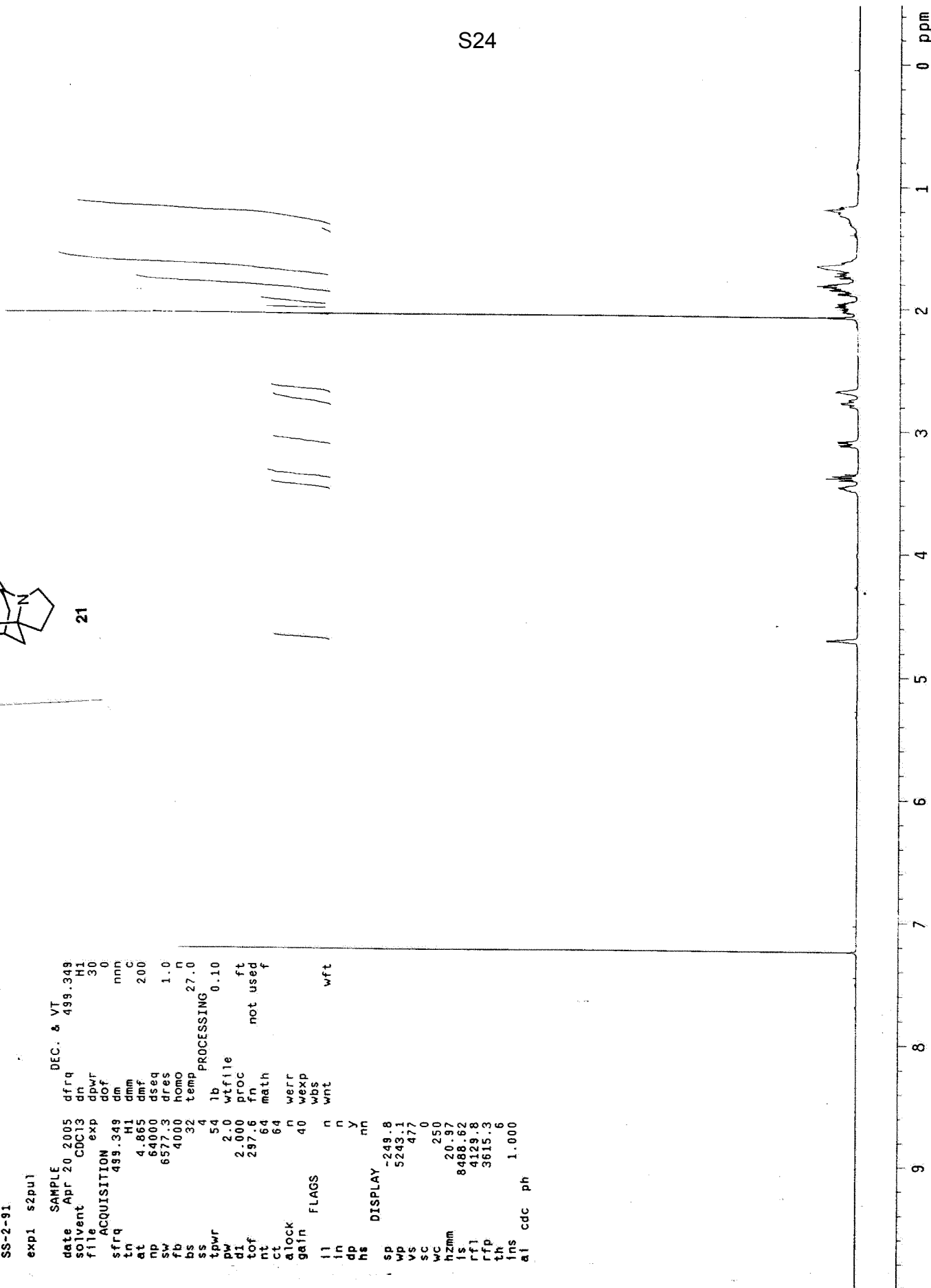


21

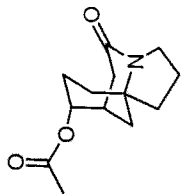
SS-2-91

exp1 s2pul

SAMPLE DEC. & VT
 date Apr 20 2005 dfrq 499.349
 solvent CDCl3 dn H1
 file ACQUISITION exp 30
 sfrq 499.349 dm nnn
 tn 4.865 dm c
 at 64000 dmf 200
 np 6577.3 dres 1.0
 sw 4000 homo n
 fb 32 temp 27.0
 bs 4
 ss 54 lb 0.10
 tpwr 2.0 wtfile
 pw 2.000 proc ft
 di 297.6 fn not used
 nt 64 math
 ct 64
 alock n werr
 gain 40 wexp
 flags n wnt wft
 ll n
 in y
 dp nn
 hs
 DISPLAY
 sp -249.8
 wp 5243.1
 vs 477
 sc 0
 wc 250
 hzmm 20.97
 ls 8488.62
 rf1 4129.8
 rfp 3615.3
 th 6
 lns 1.000
 al cdc ph



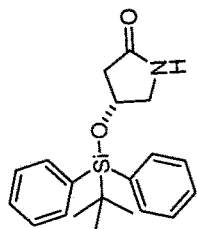
9 8 7 6 5 4 3 2 1 0 ppm



21

SS-2-91
exp4 s2pul
SAMPLE DEC. & VT
date Apr 21 2005 dfrq 499.349
solvent CDC13 dn H1
file /export/home/~44
space/data/1maybed~ -500.0
K-fid dm yy
ACQUISITION dmm 11000
sfreq 125.580
tn C13 dseq
at 1.073 dres 1.0
np 107792 homo n
sw 50219.7 temp 27.0
fb 28000
bs 64 lb 2.00
ss 84 wtfile
tpwr 84 proc ft
pw 3.0 fn not used
d1 2.000 math f
tof 6904.3 werr
nt 17000 wexp
ct 100 wbs
alock n
gain 50 wnt
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -627.9
wp 25739.4
vs 2813
sc 0
wc 250
hzm 102.96
is 90000.00
rf1 15974.4
rfp 9668.2
th 15
ins 100.000
nm cdc ph

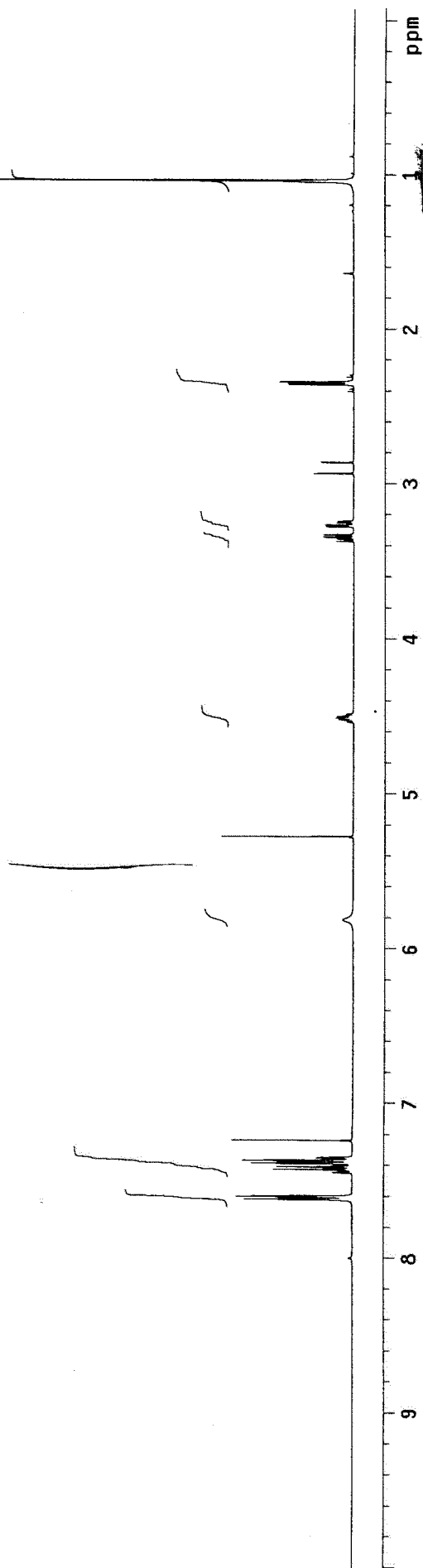
180 160 140 120 100 80 60 40 20 ppm

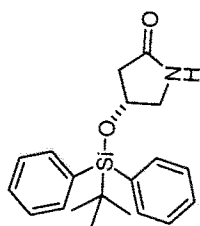


23

ss-1-282
 exp1 stdih
 SAMPLE
 date Jan 16 2005
 solvent CDC13
 file
 ACQUISITION
 sfrq 400.269
 tn H1
 at 2.856
 np 32000
 sw 5602.2
 fb 3200
 bs 16
 tpwr 58
 pw 2.0
 d1 2.000
 tof 169.9
 nt 32
 ct 32
 alock not used
 gain not used
 flags
 it n
 in n
 dp y
 sp -30.9
 wp 4040.9
 vs 168
 sc 0
 wc 250
 hzmm 16.18
 ls 301.56
 rfl 3519.4
 rfp 2897.9
 th 20
 ins 100.000
 nm cdc ph

DEC. & VT
 dfrq 400.269
 dn H1
 dpwr 30
 dof 0
 dm nnn
 dmf 200
 lb PROCESSING
 wf file 0.10
 proc ft
 fn not used
 werr
 wexp
 wbs
 wnt



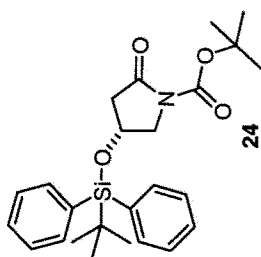


23

ss-1-282
 exp1 std13c
 SAMPLE
 date Jan 16 2005
 solvent CDCl3
 file exp
 ACQUISITION
 sfrq 100.658
 tn C13
 at 1.280
 np 64484
 sw 25188.9
 fb 13800
 bs 16
 tpwr 60
 pw 3.0
 d1 2.000
 tof 1539.5
 nt 200
 ct 200
 alock not used
 gain not used
 FLAGS
 il n
 in n
 dp y
 DISPLAY
 sp -141.9
 wp 20289.1
 vs 160
 sc 0
 wc 250
 hzmm 81.08
 is 500.00
 rfl 9301.6
 rfp 7749.8
 th 100.000
 ins cdc
 nm ph

DEC. & VT
 dfrq 400.269
 dn H1
 dpwr 38
 dof 0
 yvy
 9300
 PROCESSING
 lb 1.00
 wfile
 proc ft
 fn not used
 werr
 wexp
 whs
 wnt

180 160 140 120 100 80 60 40 20 ppm



exp1 Proton

date	Nov 14 2006	temp	27.0
solvent	cdcl3	gain	not used
file	/export/home/~	spin	20
space/data/mart	in ~	hst	0.008
ss-2-143_s2pul_H1	~	pw90	13.800
		alpha	6.600

ACQUISITION

sw	6410.3	il	n
at	4.049	in	n
np	51906	dp	y
fb	4000	hs	nn
bs	32		
ss	2	lb	0.10
dl	2.000	fn	65536
nt	16		
gt	16	sp	-112.0

PROCESSING

wp	4205.9
rfl	3705.0
rff	2894.6
rp	-118.0
lp	-25.4

DISPLAY

WC	250
SC	0
VS	161
TH	9
AI	
CD	
PH	

TRANSMITTER

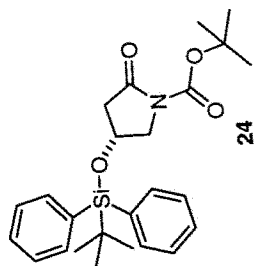
H1	
rf1	
rfp	
rp	
lp	

DECOUPLER

WC	250
SC	0
VS	161
TH	9
AI	
CD	
PH	

29412

10 9 8 7 6 5 4 3 2 1 ppm



exp1 Carbon

date	Nov 14 2006	temp	27.0
solvent	cdcl3	gain	30
file	/mnt/home_dir~	spin	20
s/space/data/mart~	hst	0.008	
n_ss-2-143_s2pul_C~	pw90	9.700	
13.fid	alfa	10.000	

ACQUISITION

sw	24509.8	fl	n
at	1.300	in	n
np	63750	dp	y
fb	17000	hs	nn
bs	64		
d1	2.000	lb	1.00
nt	256	fn	not used
ct	256		

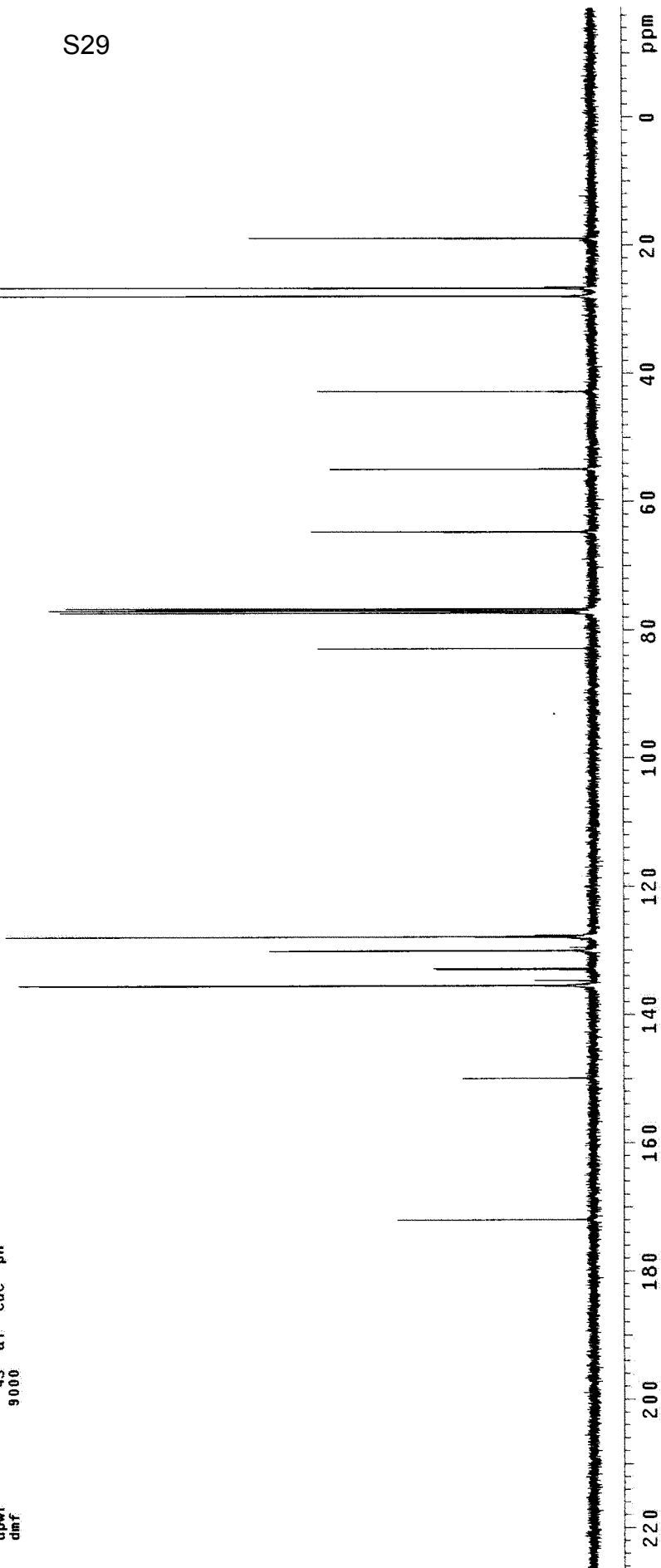
PROCESSING

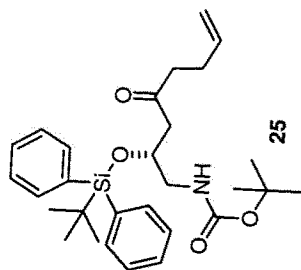
tn	-1731.5
sfreq	100.542
tof	1042.7
tpwr	54
pw	3.233

DISPLAY

dn	H1	WC	250
dof	0	SC	0
dm	YYV	VS	36462
dmm	W	TH	4
dpwr	43	AI	
dmf	9000	cdc	ph

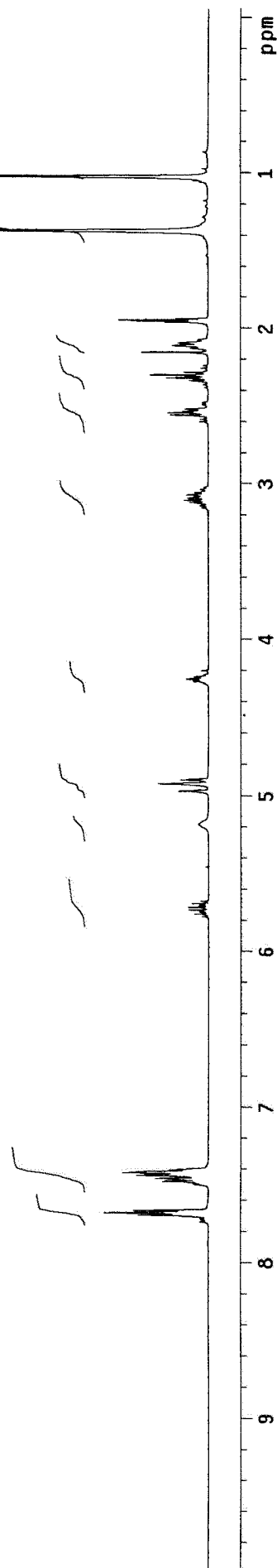
PLOT

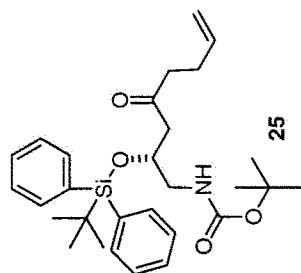




ss-2-148
exp2 std1h

SAMPLE		DEC. & VT	
date	Nov 15 2006	dfrq	400.271
solvent	cd3cn	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	400.271	dm	nnn
tn	H1	dm	C
at	2.856	dmf	200
np	32000	PROCESSING 0.10	
sw	5602.2	lb	wtfile
fb	not used	16	proc
bs	16	fn	not used
tpwr	58		
pw	2.0		
d1	2.000	werr	
tof	169.9	wexp	
nt	8	wbs	
ct	8	wnt	
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-23.4		
wp	4014.0		
vs	147		
sc	0		
wc	250		
hzmm	16.06		
ls	242.43		
rfl	1399.5		
rffp	780.5		
th	20		
ins	1.000		
nm	cdc		
ph			



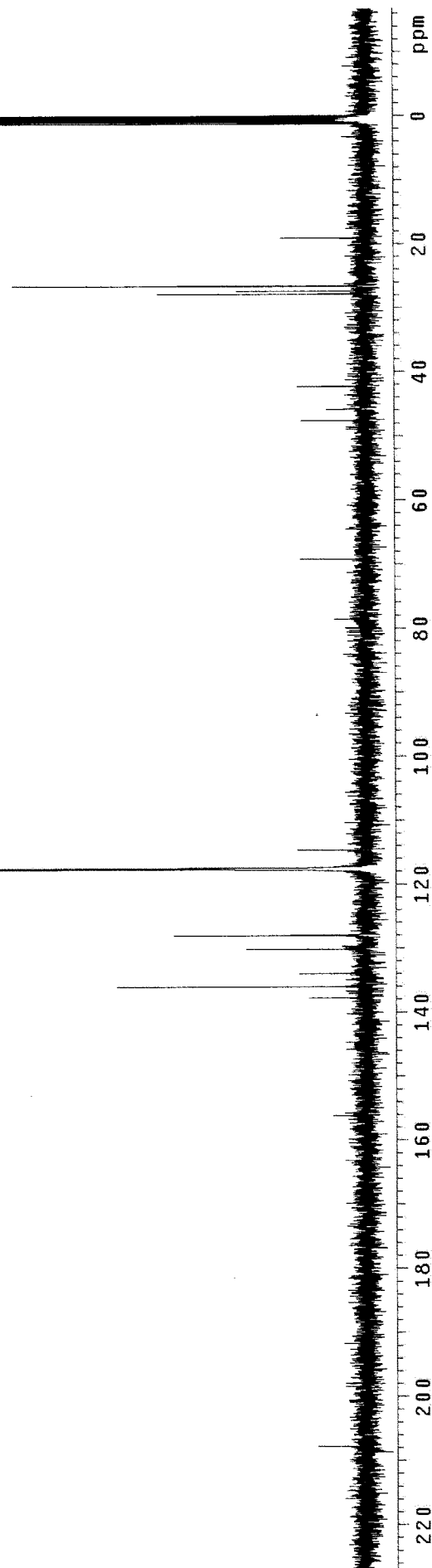


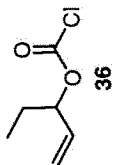
Automation directory: /export/home/staffw/vnmrsys/data/auto_2006.11.15
 File: martin_ss-2-148_s2pul_01
 Sample id: S_20061115_15_01
 Sample: ss-2-148

Pulse Sequence: s2pul

Solvent: cd3cn
 Temp: 27.0 C / 300.1 K
 Sample #15, Operator: martin
 File: martin_ss-2-148_s2pul_01
 VNMR5-400 "nmrrobo"

Relax. delay 2.000 sec
 Pulse 30.0 degrees
 Acq. time 1.300 sec
 Width 24509.8 Hz
 256 Repetitions
 OBSERVE C13, 100.5314798 MHZ
 DECOUPLE H1, 399.8088258 MHZ
 Power 43 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 65536
 Total time 14 min, 7 sec





ss-4-170

exp2 std1h

SAMPLE DEC. & VT
 date 1 2007 dfrq 400.269
 solvent Benzene dn H1
 file exp dpwr 30
 ACQUISITION dof 0
 sfrq 400.269 dm nnn
 tn H1 C
 at 2.856 dmm 200
 mp 32000 dmf
 sw 5602.2 lb PROCESSING 0.10
 fb not used wtfile ft
 bs 16 proc not used
 tpwr 58 fn
 pw 2.0 werr
 d1 2.000 wexp
 tof 169.9 wbs
 nt 16 wnt
 ct 16
 alock n
 gain not used
 FLAG n
 il n
 in n
 dp y
 DISPLAY
 sp -63.7
 wp 4102.2
 vs 147
 sc 0
 wc 250
 hzmm 16.41
 is 301.56
 rfl 3462.4
 rfp 2861.9
 th 20
 ins 1.000
 nm cdc ph

PDM

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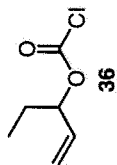
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S33

```

expi Carbon
SAMPLE
date Feb 1 2007 temp 27.0
solvent c6d6 gain 30
file /export/home/~ spin 20
space/data/martin_~ hst 0.008
ss-4-170_s2pul_C13~ pw90 9.500
-fid alfa 10.000
ACQUISITION
sw 24509.8 il n
at 1.300 in n
np 63750 dp y
fb 17000 hs nn
bs 64
d1 2.000 lb 1.00
nt 256 fn not used
ct 256 DISPLAY
tn TRANSMITTER C13 SP -175.3
sfrq 100.542 wf 18461.6
tof 1042.7 rfl 14555.7
tpwr 55 rfp 12868.0
pw 3.167 lp -139.1
DECOUPLER H1 wc 250 -187.2
dn 0 sc 0
dof 0
dm yyv vs 10178
dmm w th cdc ph
dpwr 44 ai
dmf 9700
  
```

SPECIAL

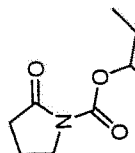
FLAGS

PROCESSING

DISPLAY

PLOT

160 140 120 100 80 60 40 20 ppm



38

exp1 Proton

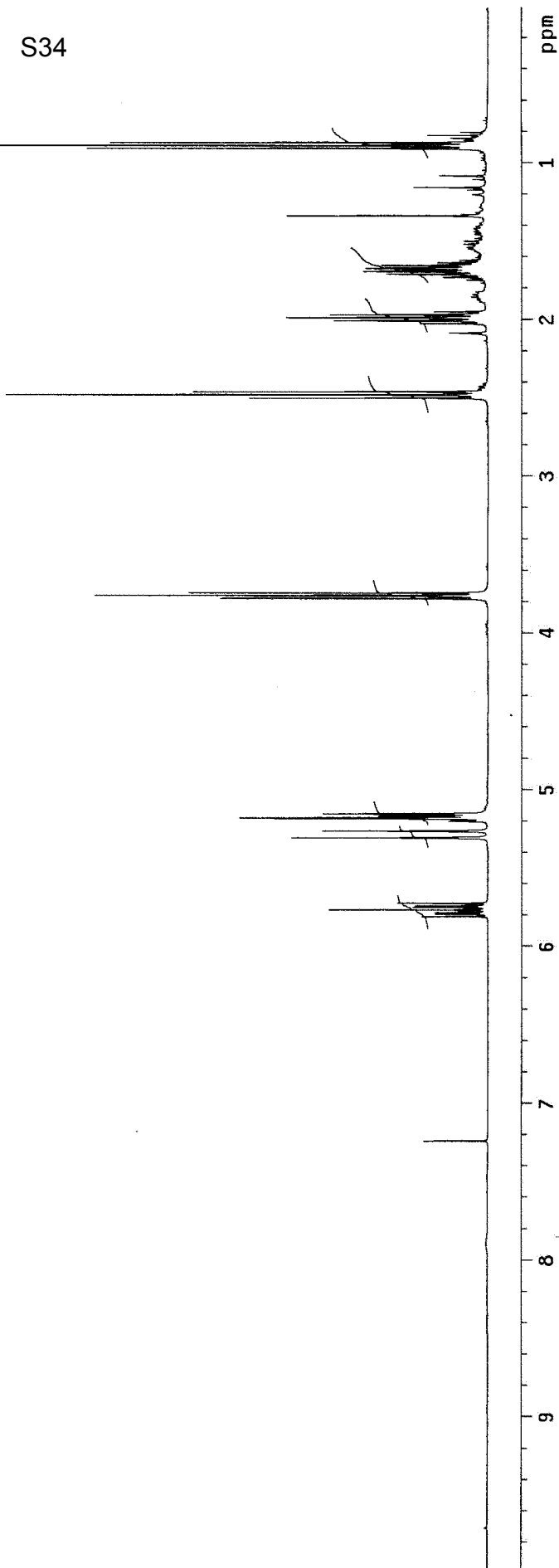
SAMPLE date Feb 3 2007 temp 27.0
 solvent cdc13 gain not used
 file /export/home/~ spin 20
 space/data/martin~ hst 0.008
 ss-4-171_s2pu_H1~ pw90 13.900
 ss-4-171_s2pu_H1~ fid alfa 6.600

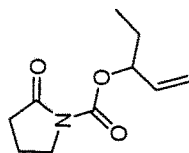
ACQUISITION
 sw 6410.3 il n
 at 4.048 tn n
 np 51906 dp y
 fb 4000 hs nn
 bs 32
 ss 2 lb 0.10
 d1 2.000 fn 65536
 nt 32
 ct 32 sp 2.5

TRANSMITTER H1
 tn 3987.2
 sfrq 398.807 rfl 3708.3
 tof 399.5 rfp 2894.6
 tpwr 61 lp 56.1
 pw 4.633 plot -27.6

DECOUPLER WC 250
 dn C13 SC 0
 dof 0 vs 174
 dm nnn th 50
 dmm C al cdc ph
 dpwr 35
 dmf 29412

S34



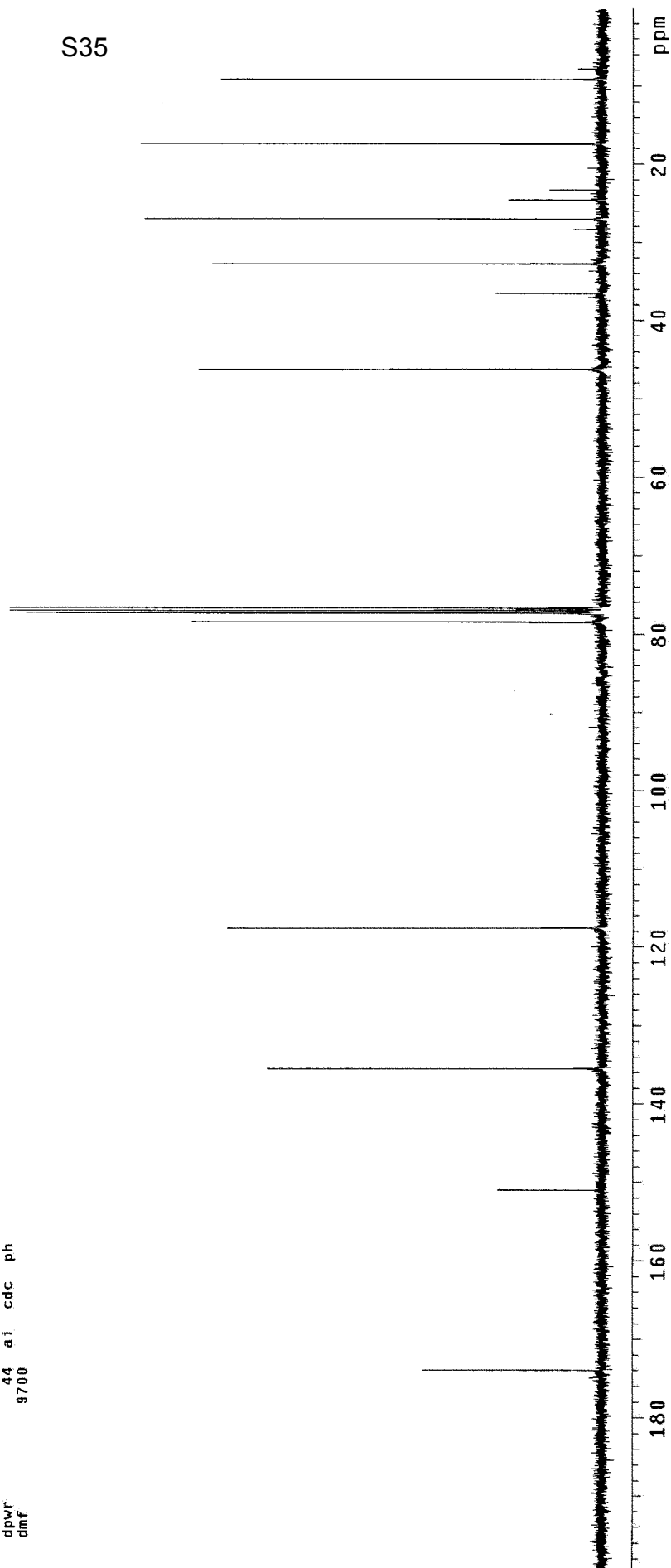


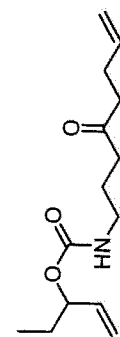
38

expi Carbon

SAMPLE		SPECIAL		27.0
date	Feb 3 2007	temp		
solvent	cdcl3	gain	30	
file	/export/home/~	spin	20	
space/data/martin_	hst	0.008		
ss-4-171_s2pul_C13~	pw90	9.500		
	.fid	alpha	10.000	

ACQUISITION		FLAGS	
sw	24509.8	il	n
at	1.300	in	n
np	63750	dp	y
fb	17000	hs	nn
bs	64		
d1	2.000	lb	1.00
nt	256	fn	not used
ct	256		
TRANSMITTER		DISPLAY	
tn	C13	sp	6.8
tn	20037.6		
sfrq	100.542	rf1	9471.7
tof	1042.7	rfp	7740.9
tpwr	55	rp	-110.7
pw	3.167	lp	-188.6
DECOUPLER		PLOT	
dn	H1	wc	250
dof	0	sc	0
dm	yyy	vs	39970
dmm	w	th	9
dpwr	44	ai	cdc
dmf	9700	ph	

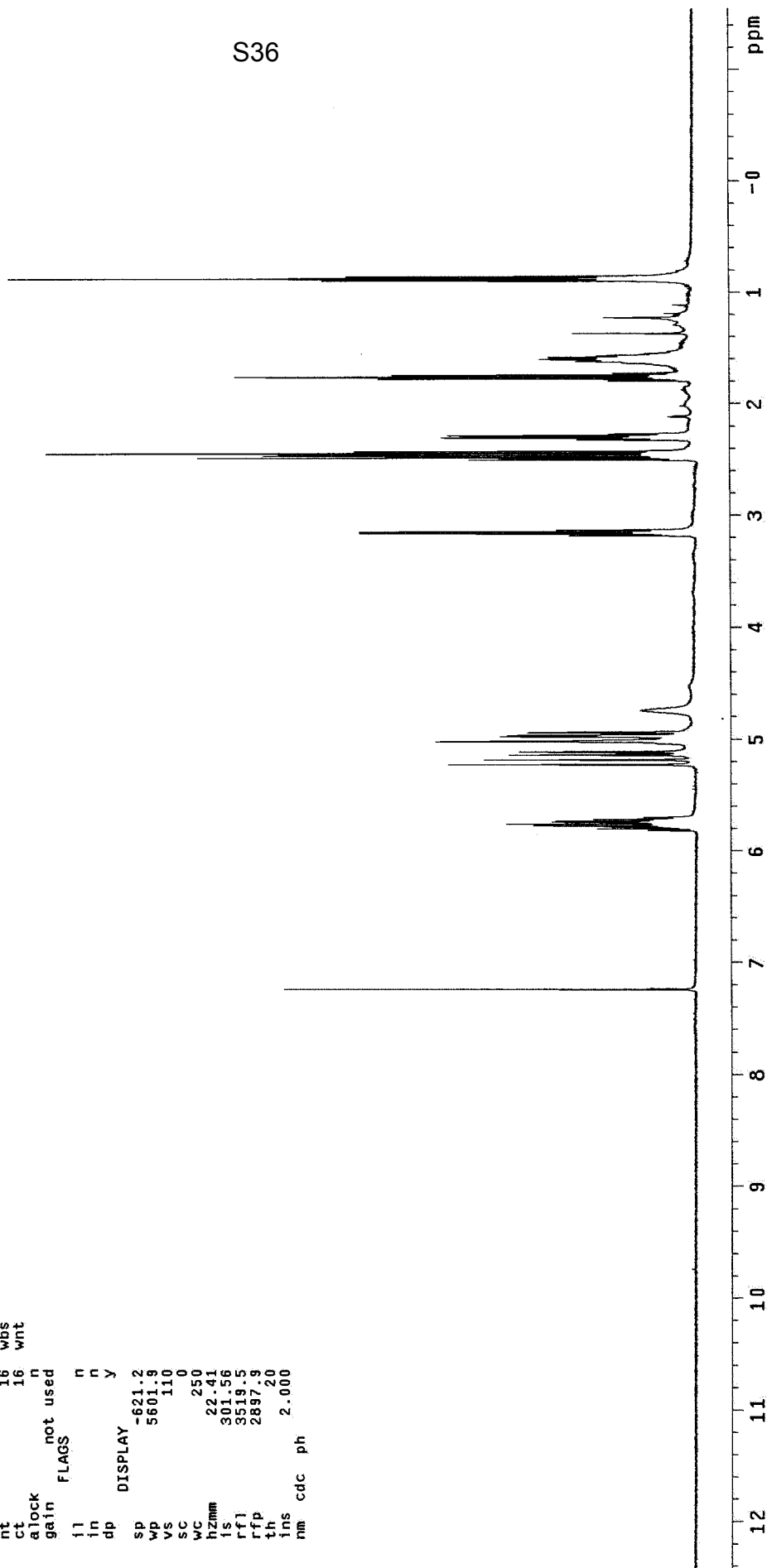


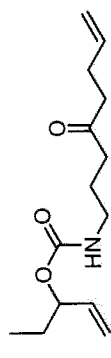


39

ss-3-159
exp2 std1h

date	Feb 9 2007	DEC. & VT	400.269
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	400.269	dm	nnn
tn	2.856	dmm	C
at	32000	dmf	200
np	5602.2	lb	PROCESSING 0.10
sw	not used	wtfile	
fb	16	proc	ft
bs	58	fn	not used
tpwr	2.0		
pw	2.000	werr	
d1	168.9	wexp	
tof	16	wbs	
nt	16	wnt	
ct	not used		
alock	not used		
gain	n		
fl	n		
in	n		
dp	y		
sp	-521.2		
wp	5601.9		
vs	110		
sc	0		
wc	250		
hzmh	22.41		
ls	301.56		
rfl	3519.5		
rffp	2897.9		
th	2.000		
ins	2.000		
nm	cdc	ph	





39

exp1 Carbon

date	Feb 5 2007	temp	27.0
solvent	cdc13	gain	30
file	/mnt/home/dlr~	spin	20
s/space	/data/marti~	hst	0.008
n_ss-3-159_s2pul1_C~	pw90		9.500
13.fid	alfa	10.000	

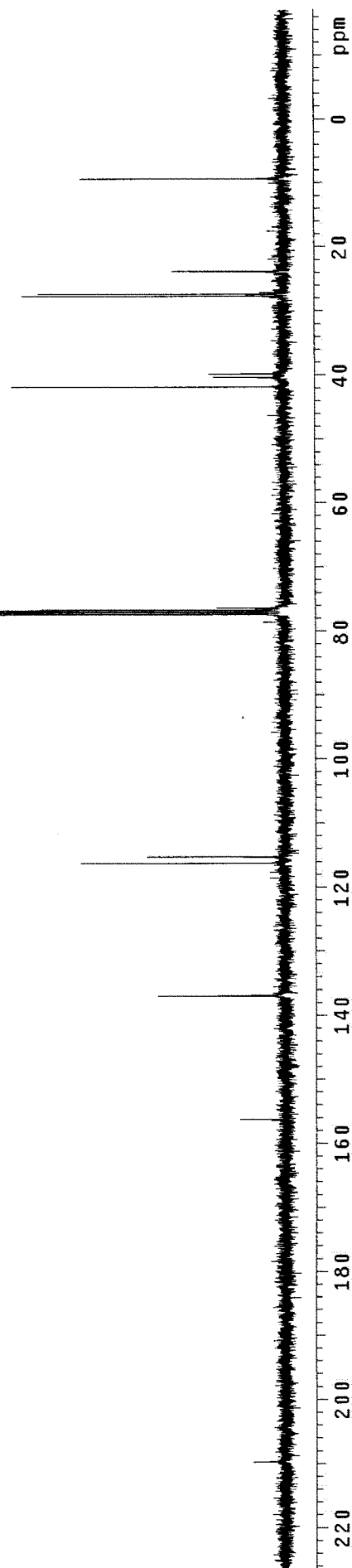
sw	24509.8	il	n
at	1.300	in	n
np	63750	dp	y
fb	17000	hs	nn
bs	64		
d1	2.000	lb	1.00
nt	256	fn	not used
ct	256		

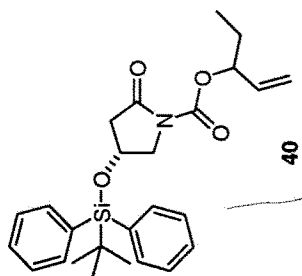
tn	TRANSMITTER	C13	sp
sfrq	100.542	wp	-1724.8
tof	1042.7	rfl	24509.1
tpwr	55	rfp	9466.4
		rp	7740.9
		lp	-114.5
			-190.0

pw	DECOUPLER	H1	pl
dn		WC	250
dof		0	SC
dm		YV	VS
dmm		W	TH
dpwr		44	AI
dmf		9700	

cds	ph
cdc	13

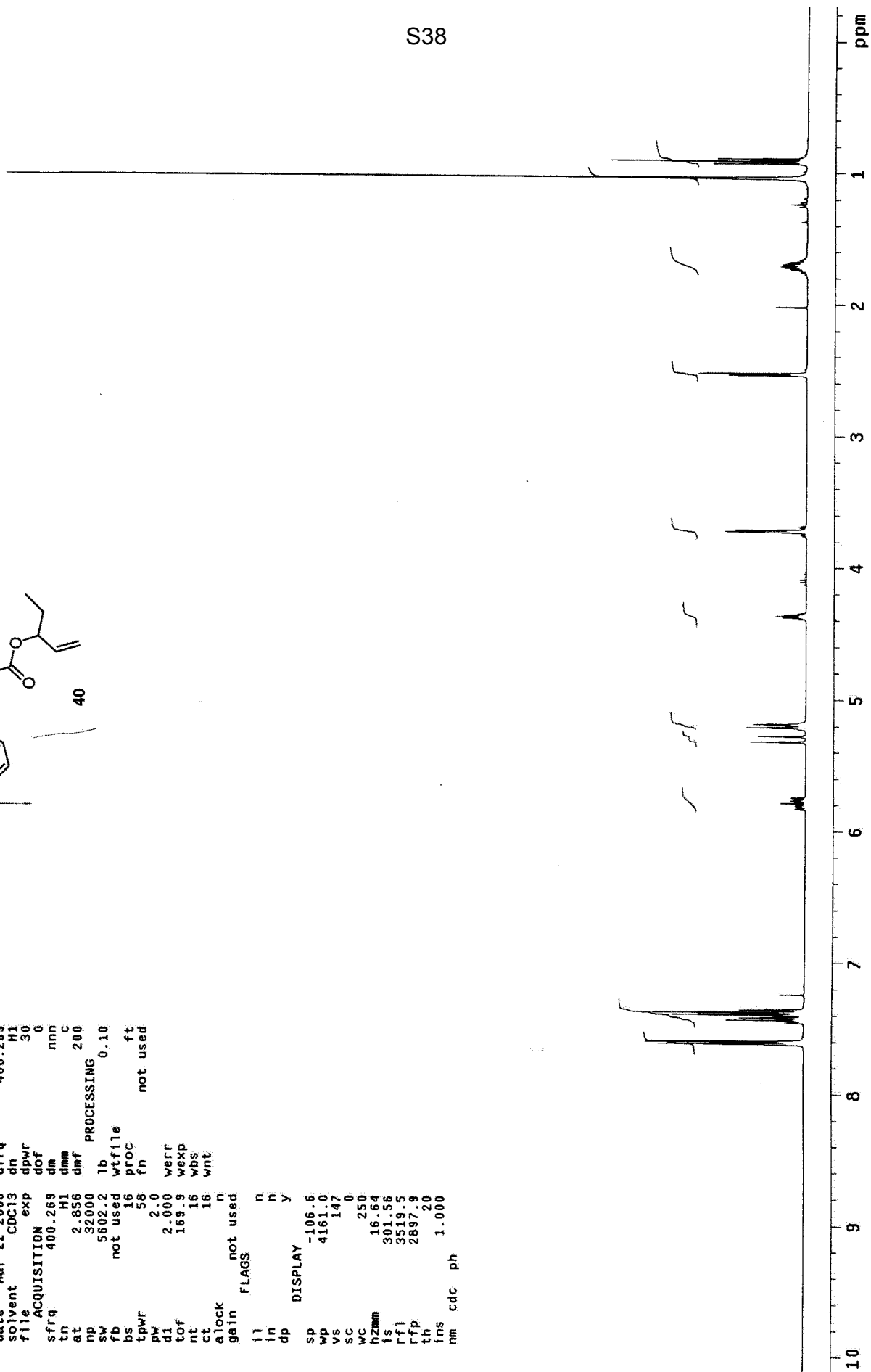
S37

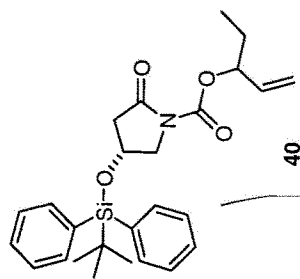




ss-3-161
exp2 stdih

SAMPLE		DEC. & VT	
date	Mar 22 2006	dfrq	400.269
solvent	CDC13	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	400.269	dm	nnn
tn	H1	dmm	C
at	2.856	dmf	200
np	32000	PROCESSING 0.10	
sw	5602.2	lb	not used
fb	not used	wf	ft
bs	16	proc	not used
tpwr	58	fn	
pw	2.0	werr	
d1	2.000	wexp	
tof	169.9	wbs	
nt	16	wnt	
ct	16		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-106.6		
wp	4161.0		
vs	147		
sc	0		
wc	250		
h2mm	16.64		
ls	301.56		
rfl	3519.5		
rfp	2897.8		
th	20		
ins	1.000		
nm	cdc		
ph			



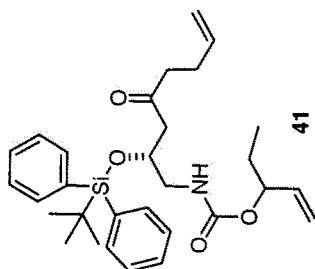


ss-3-164
exp2 std13c

date	Mar 22 2006	DEC. & VI	400.269
solvent	CDCl3	dn	H1
file	exp	dpwr	38
ACQUISITION		dof	0
sfrq	100.658	dm	YVY
tn	1.280	dmm	w
at	64484	dmf	9300
np	25188.9	lb	PROCESSING
sw	13800	wtfile	1.00
fb	16	proc	ft
bs	60	fn	not used
tpwr	3.0	werr	
pw	2.000	wexp	
d1	1539.5	wbs	
nt	64	wnt	
ct	64		
alock	n		
gain	not used		
il	n		
in	n		
dp	y		
sp	-336.2		
wp	21683.6		
vs	130		
sc	0		
wc	250		
hzmm	86.73		
is	500.00		
rfl	9309.8		
rfp	7749.8		
th	20		
ins	100.000		
nm	cdc		
ph			

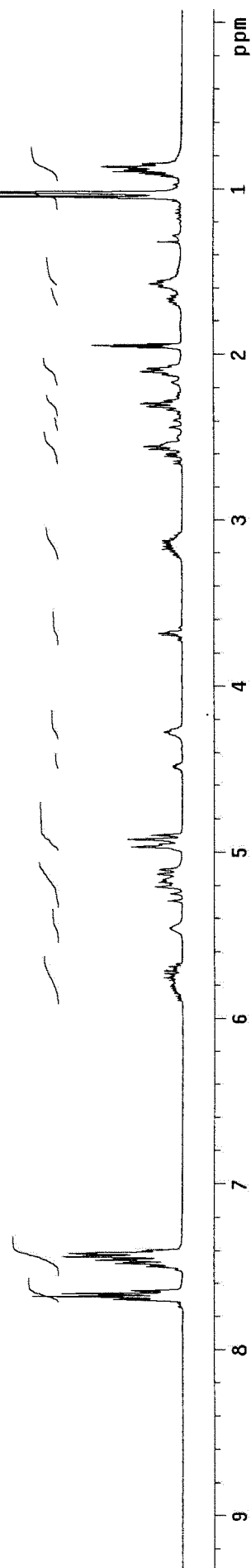
200 180 160 140 120 100 80 60 40 20 ppm

S40



ss-3-279
exp2 stdlh

SAMPLE		DEC. & VT	
date	Nov 15 2006	dfrq	400.271
solvent	cd3cn	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	400.271	dm	nnn
tn	H1	dmm	C
at	2.856	dmf	200
np	32000	PROCESSING	
sw	5602.2	lb	0.10
fb	not used	wtfile	ft
bs	16	proc	not used
tpwr	58	fn	
pw	2.0	werr	
d1	2.000	wexp	
tof	169.9	wbs	
nt	8	wnt	
ct	8		
alock	n		
gain	not used		
FLAGS		il	n
		in	n
		dp	y
DISPLAY		sp	-30.5
		wp	3764.0
		vs	158
		sc	0
		wc	250
		hzmm	15.06
		is	224.98
		rfl	1399.5
		rfp	780.5
		th	20
		ins	1.000
nm	cdc	ph	



exp1 Carbon

date Nov 15 2006 temp 27.0
 solvent cd3cn gain 30
 file /export/home/~ martin~ hst 20
 space/data/martin~ hst 0.008
 ss-3-279_s2pul_C13~ pw90 9.700
 .fid alfa 10.000

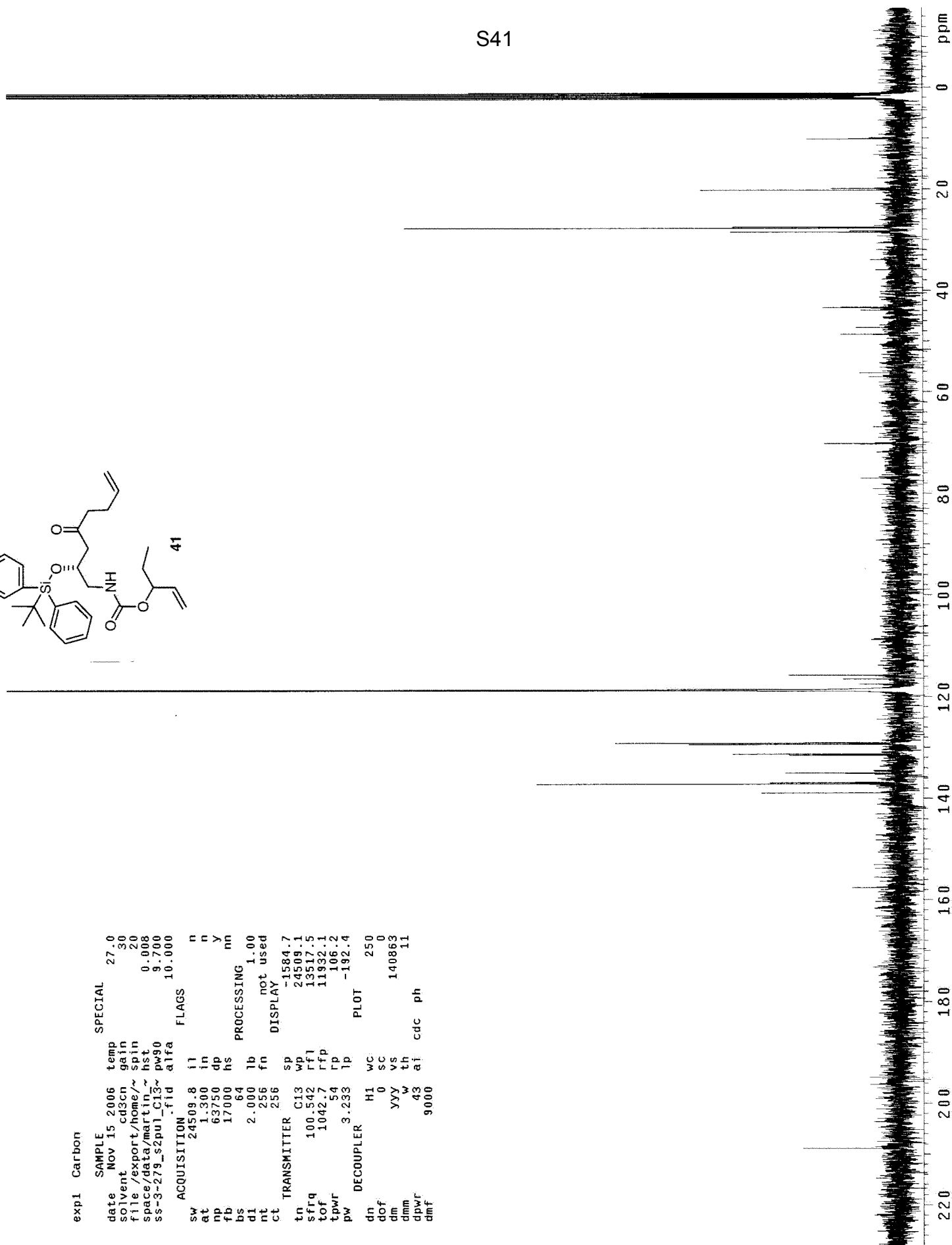
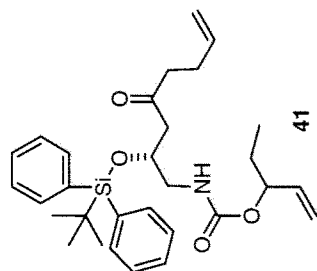
ACQUISITION
 sw 24509.8 il n
 at 1.300 in n
 np 63750 dp y
 fb 17000 hs nn
 bs 64
 d1 2.000 lb 1.00
 nt 256 fn not used
 ct 256

PROCESSING
 not used
 DISPLAY
 -1584.7
 24509.1
 13517.5
 11932.1
 106.2
 -192.4

TRANSMITTER C13
 tn wp
 sfrq 100.542 rfl
 tof 1042.7 rfp
 tpwr 54 rp
 pw 3.233 lp

DECOUPLER
 dn H1 wc 250
 dof 0 sc 0
 dm yy vs 140863
 dmm w th 11
 dpwr 43 ai
 dmf 9000

PLOT
 cdc ph





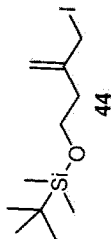
ppm



ss-3-229

exp2 std13c

SAMPLE		DEC. & VT	
date	Jun 2 2006	dfrq	400.269
solvent	CDCl3	dn	H1
file	exp	dpwr	38
ACQUISITION		dof	0
sfrq	100.658	dm	VVY
tn	C13	dmm	w
at	1.280	dmf	9300
np	64484	lb	PROCESSING
sw	25188.9	wtfile	1.00
fb	13800	fn	ft
bs	16	proc	not used
tpwr	60	fn	
pw	3.0	werr	
d1	2.000	wexp	
tof	1539.5	wbs	
nt	32	wnt	
ct	32		
alock	not used		
gain	not used		
il	n		
in	n		
dp	y		
DISPLAY			
sp	-1121.8		
wp	19634.3		
vs	99		
sc	0		
wc	250		
hzm	78.54		
ls	500.00		
rfl	9302.1		
rfl	7749.8		
th	20		
ins	100.000		
nm	cdc		
ph			



77.321
77.000
76.687

25.897

18.251

37.223

-5.333

144.303

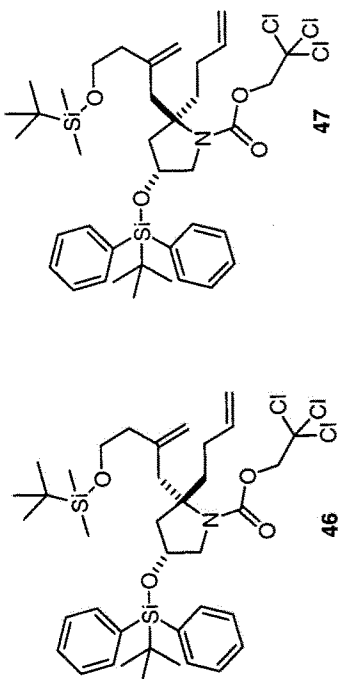
S43

180 160 140 120 100 80 60 40 20 0 ppm

SS-3-195
temp=100C
ss3195_h1_100C

exp1 s2pul

SAMPLE		DEC. & VT	
date	May 3 2006	dfrq	499.869
solvent	DMSO	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	499.870	dm	nnn
tn	H1	dmm	C
at	3.996	dmf	200
np	73980	dseq	
sw	9256.0	dres	1.0
fb	not used	homo	n
bs	32	temp	100.0
tpwr	57	PROCESSING	
pw	2.0	lb	0.10
d1	2.000	wtfile	
tof	1124.6	proc	ft
nt	64	fn	not used
ct	64	math	f
alock	n	werr	
gain	30	wexp	
il	n	wbs	
in	n	wnt	
dp	y		
hs	nn		
DISPLAY		sp	-250.0
wp	5248.5	vs	423
sc	0	sc	0
wc	250	hzmm	20.99
is	3133.75	rfl	2257.0
th	1244.7	ins	1.000
ai		ph	



S44

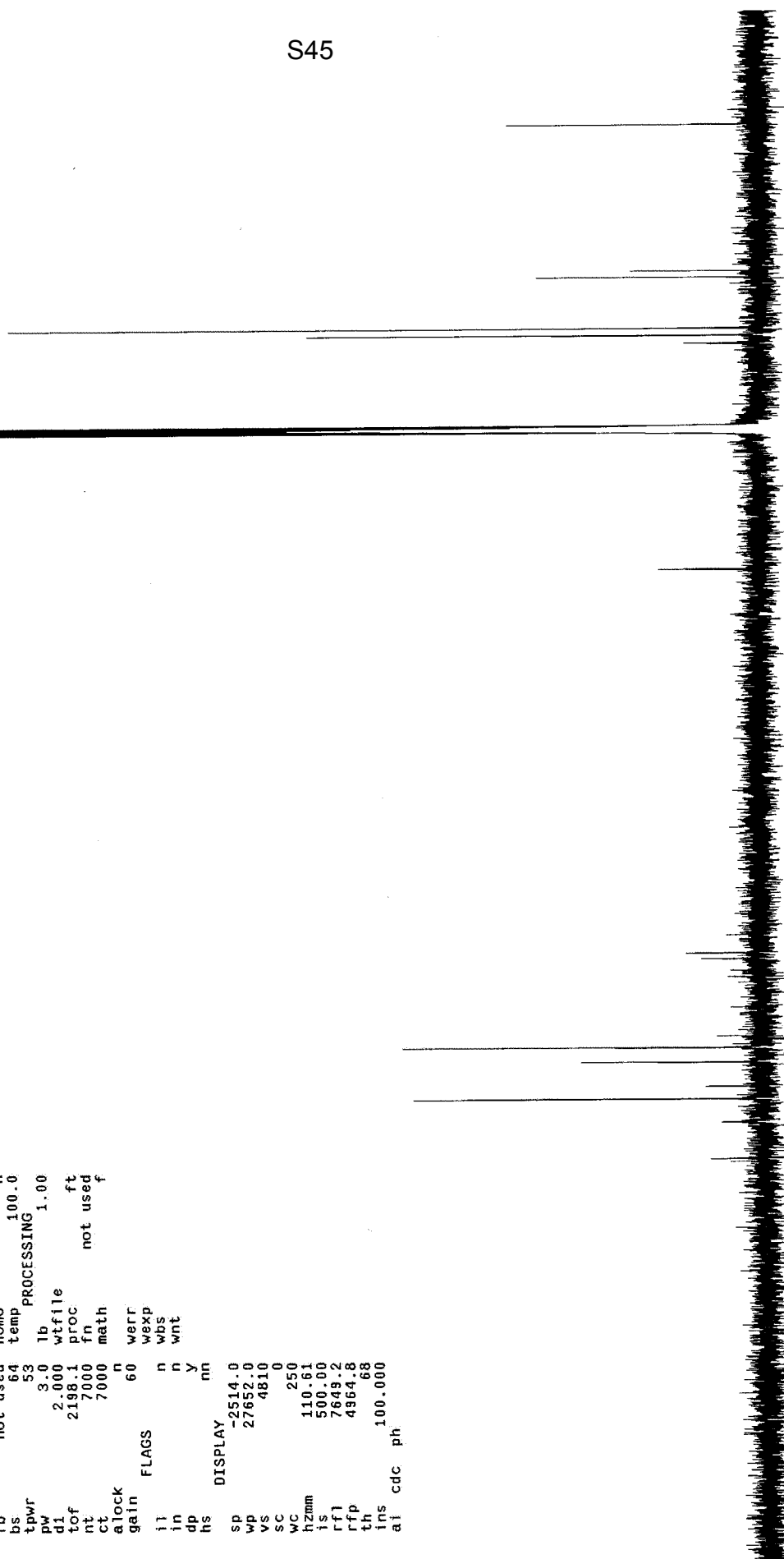
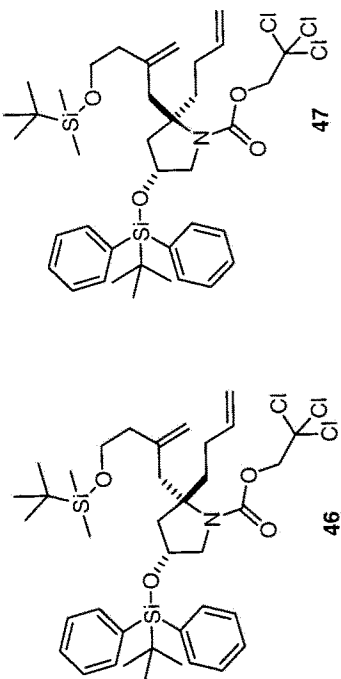
9 8 7 6 5 4 3 2 1 0 ppm

S45

ppm

SS-3-195
temp=100c
ss3195_c13_100c
exp4 s2pul

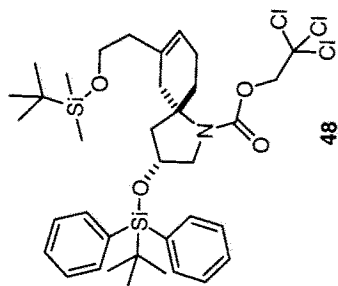
SAMPLE DEC. & VT
date May 3 2006 dfrq 499.869
solvent DMSO dn H1
file exp 37
ACQUISITION
sfrq 125.706 dm VVY
tn 1.278 dm 10582
at 85262 dseq
np 33333.3 dres 1.0
sw not used homo n
fb temp 100.0
bs 64
tpwr 53
pw 3.0 lb
d1 2.000 wfile 1.00
tof 2198.1 proc ft
nt 7000 fn not used
ct 7000 math f
alock n
gain 60 werr
FLAGS n wbs
il n
in n
dp y
hs nm
DISPLAY
sp -2514.0
wp 27652.0
vs 4810
sc 0
wc 250
hzmm 110.61
is 500.00
rfl 7649.2
rfp 4964.8
th 68
ins 100.000
ai cdc ph



SS-4-30-1-PREP
ss4301prep_h1

exp1 s2pul

SAMPLE		DEC. & VT	
date	Mar 23 2007	dfrq	499.867
solvent	CDC13	dn	H1
file	/export/home/~	dpwr	30
space/data/ss4301p	~	dof	0
rep	h1.fid	dm	nnn
ACQUISITION		dmm	C
sfrq	499.868	dmf	200
tn	H1	dseq	1.0
at	3.996	dres	1.0
np	73980	homo	n
sw	9256.0	temp	27.0
fb	not used	PROCESSING	
bs	32	lb	0.10
tpwr	57	wfifile	
pw	11.0	proc	ft
dl	2.000	fn	not used
tof	1124.6	math	f
nt	128	werr	
ct	128	wexp	n
alock	n	wbs	30
gain	30	wnt	wft
FLAGS			
il	n		
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-175.1		
wp	5162.3		
vs	1646		
sc	0		
wc	250		
hzmm	20.65		
ls	3411.71		
rfl	4644.5		
rfl	3619.0		
th	7		
ins	1.000		
ai			
ph			



S46

ppm

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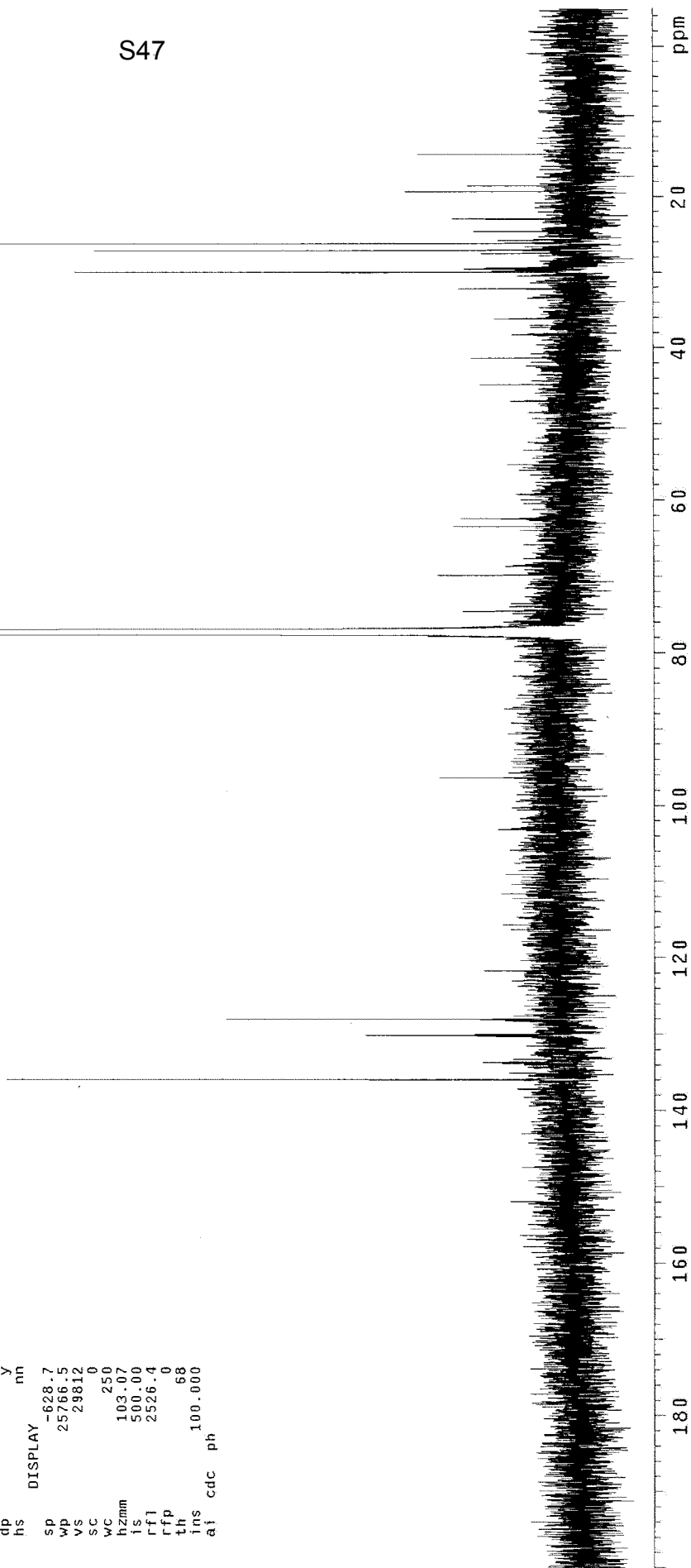
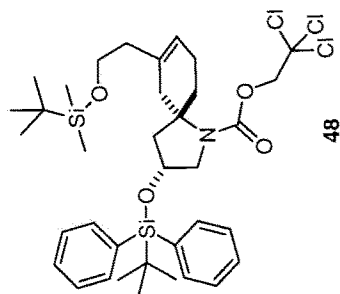
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SS-4-30-1-PREP
ss4301prep_c13_shigem
exp4 s2pul

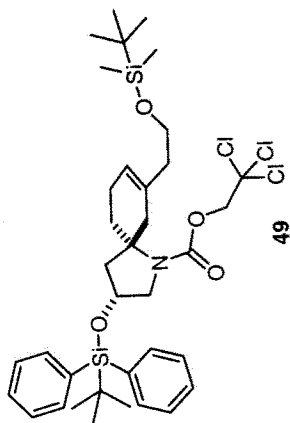
date	Mar 30 2007	DEC. & VT	
solvent	CDCl3	dfrq	499.867
file	exp	dn	H1
ACQUISITION		dpwr	37
sfrq	125.705	dof	0
tn	C13	dm	yyv
at	1.279	dmm	w
np	85262	dseq	10582
sw	33333.3	dres	1.0
fb	not used	homo	n
bs	64	temp	27.0
tpwr	53	PROCESSING	
pw	3.0	lb	1.00
d1	2.000	wtfile	
tof	2198.1	proc	ft
nt	60000	fn	not used
ct	60000	math	f
alock	n		
gain	60	werr	
il	n	wexp	
in	n	wbs	
dp	n	wnt	
hs	nn		
DISPLAY			
sp	-628.7		
wp	25766.5		
vs	29812		
sc	0		
wc	250		
hzm	103.07		
is	500.00		
rfl	2526.4		
rff	0		
th	68		
ins	100.000		
ai	cdc	ph	



SS-4-30-2
ss4302_h1

exp1 s2pu1

SAMPLE DEC. & VI
date Mar 27 2007 dfrq 499.867
solvent CDC13 dn H1
file /export/home/~ dpwr 30
space/data/ss4302_~ dof 0
h1_al.fid dm nmn
ACQUISITION dmm c
sfrq 499.868 dmf 200
tn H1 dseq
at 3.996 dres 1.0
np 73980 homo n
sw 9256.0 temp 27.0
fb not used lb PROCESSING 0.10
bs 32 wtfile ft
tpwr 57 proc
pw 11.0 fn not used
d1 2.000 math f
tof 1124.6
nt 512
ct 512 werr
alock n wexp
gain 30 wbs
il n wnt
in n
dp y
hs nn
DISPLAY
sp -163.1
wp 5174.5
vs 1103
sc 0
wc 250
h2mm 20.70
ls 3573.52
rfl 4644.7
rfp 3619.0
th 1
ins 1.000
ai ph



S48

ppm

1

2

3

4

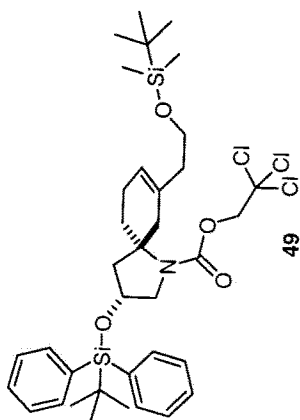
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SS-4-30-RCM-2

exp4 s2pul

SAMPLE DEC. & VT
 date Sep 11 2006 dfrq 499.349
 solvent CDCl3 dn H1
 file exp dpwr 44
 ACQUISITION dof -500.0
 sfrq 125.580 dm VVY
 tn C13 dmm W
 at 1.073 dmf 11000
 np 107792 dseq
 sw 50219.7 dres 1.0
 fb 28000 homo n
 bs 64 temp 27.0
 ss 128 PROCESSING
 tpwr 54 lb 2.00
 pw 3.0 wtfile
 d1 2.000 proc ft
 tof 6904.3 fn not used
 nt 17000 math f
 ct 17000
 alock n werr
 gain 50 wexp
 flags n wbs
 il n wnt
 in n
 dp Y
 hs mn
 DISPLAY
 sp -1256.1
 wp 26367.3
 vs 1943
 sc 0
 wc 250
 hzmm 105.47
 is 90000.00
 rfl 15974.8
 rfp 3668.2
 th 1
 ins 100.000
 nm cdc ph

180 160 140 120 100 80 60 40 20 0 ppm