Supporting Information

for

Ligand-Controlled, Norbornene-Mediated, Regio- and Diastereoselective Rhodium-Catalyzed Intramolecular Alkene Hydrosilylation Reactions

Yuanda Hua, Hiep H. Nguyen, William R. Scaggs, and Junha Jeon*

Department of Chemistry and Biochemistry, University of Texas at Arlington
Arlington, TX 76019

*jjeon@uta.edu

Contents of Supporting Information page

Methods

Contents of Supporting Information S1

General Experimental S2-S3

Table of isolated compounds S4-S10

Preparation procedures and listings of characterization data for all key compounds S11-S52

NMR Spectra

Copies of ^{1}H and ^{13}C NMR spectra of each isolated compound S53-S181

General Experimental

^{1}H and ^{13}C NMR spectra were recorded on JOEL Eclipse Plus 500 (500 MHz) and JEOL ECX 300 (300 MHz) spectrometers. ^{1}H NMR chemical shifts in $CDCl_3$ are referenced to chloroform (7.26 ppm) and in PhH-d_6 to 7.16. Non-first order multiplets are identified as "nfom". ^{13}C NMR chemical shifts in $CDCl_3$ are referenced to chloroform (77.23 ppm). The following format was used to report peaks: chemical shift in ppm [multiplicity, coupling constant(s) in Hz, integral, and assignment]. ^{1}H NMR assignments are indicated by structure environment, e.g., CH_aH$_b$. Coupling constant analysis was guided by methods we have described elsewhere.[1] ^{1}H -NMR and ^{13}C NMR were processed with either iNMR or NUTS software program.

Infrared (IR) spectra were recorded on a Bruker Alpha-P FT-IR spectrometer using neat (for liquid compound) or a thin film from a concentrated DCM solution. Absorptions are reported in cm^{-1}.

MPLC refers to medium pressure liquid chromatography (25-200 psi) using hand-packed columns of Silasorb silica gel (20-45 μm, spherical, 70 Å pore size), a Waters HPLC pump, and a Waters R401 differential refractive index detector.

Electrospray ionization (ESI) mass spectrometry was performed on a Shimadzu LCMS IT-TOF. All HRMS data were recorded in the ESI mode. Samples were introduced as solutions in methylene chloride (DCM).

GCMS data were recorded either on a Varian 450-GC/Varian 240-MS System. The methods used are noted parenthetically: 5025015 refers to 2 min @ 50 °C – 20 °C/min – 3 min @ 250 °C (a 50 °C initial temperature that was held for 2 minutes followed by a 20 °C/min ramp to a final temperature of 250 °C that was held for 3 minutes for a total run time of 15 minutes). 5029017 refers to: 2 min @ 50 °C – 20 °C/min – 3 min @ 290 °C. 5029019 refers to: 2 min @ 50 °C – 20 °C/min – 5 min @ 290 °C. 5032021 refers to: 2 min @ 50 °C – 20 °C/min – 5 min @ 320 °C.

Reactions requiring anhydrous conditions were performed under an atmosphere of nitrogen or argon in flame or oven dried glassware. Anhydrous toluene and methylene chloride were distilled from CaH_2. Anhydrous THF and diethyl ether were distilled from sodium and benzophenone. Triethylamine and pyridine were distilled from KOH. DMF and DMSO were stored over 4Å molecular sieves. The concentrations of anionic solutions (e.g., *n*-BuLi and Grignards) were titered by spectroscopic (No-D NMR) methods.[2]

TLC experiments were performed on EMD Merck F254, 250 μm thickness.

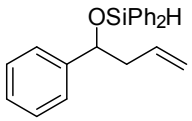
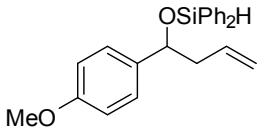
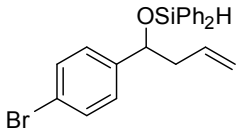
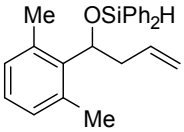
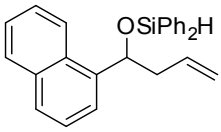
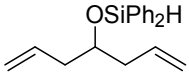
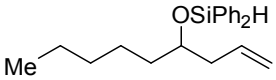
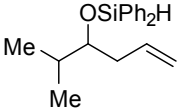
Abbreiviation: NBS = N-Bromosuccinimide, nbd = norbornadiene, nbe = norbornene, dpph = 1,6-bis(diphenylphosphino)hexane, BINAP = 2,2'-bis (diphenylphosphino)-1,1'-binaphthyl.

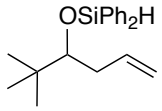
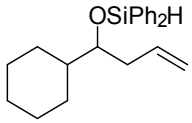
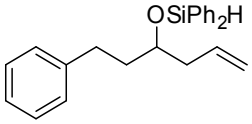
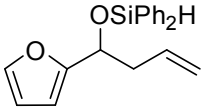
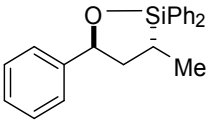
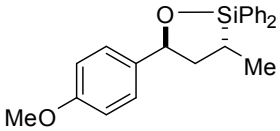
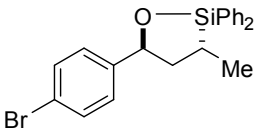
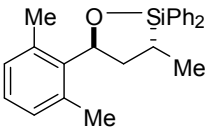
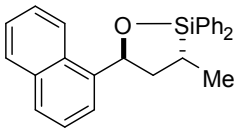
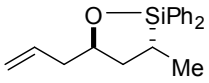
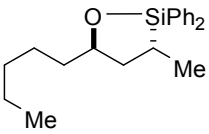
Supporting Information References

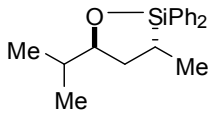
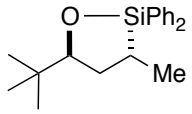
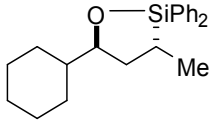
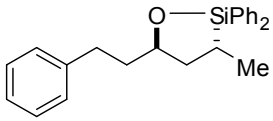
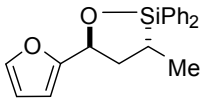
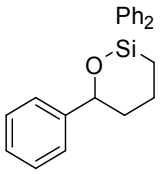
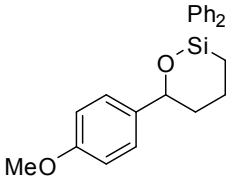
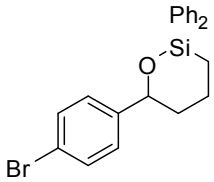
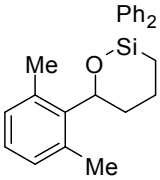
1. "A method for easily determining coupling constant (*J*) values: An addendum to 'A practical guide to first-order multiplet analysis in ^{1}H NMR spectroscopy'," Hoye, T. R.; Zhao, H. *J. Org. Chem.* **2002**, *67*, 4014-4016.
2. "No-D NMR spectroscopy as a convenient method for titering organolithium (RLi), RMgX, and LDA solutions," Hoye, T. R.; Eklov, B. M.; Voloshin, M. *Org. Lett.* **2004**, *6*, 2567-2570.

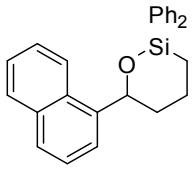
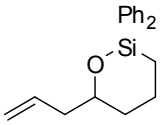
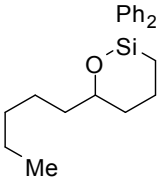
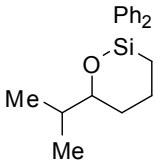
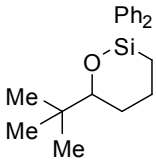
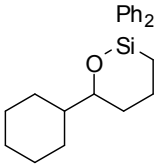
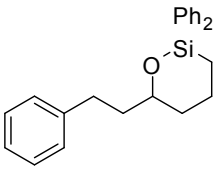
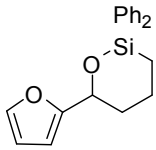
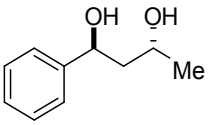
Table of Isolated Compounds

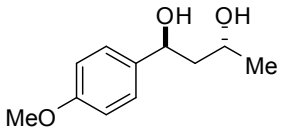
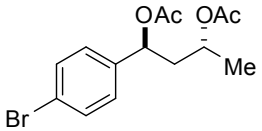
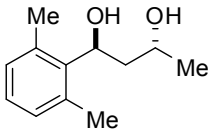
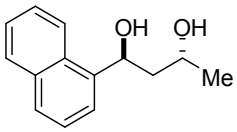
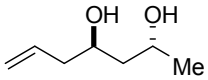
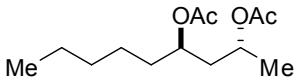
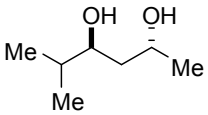
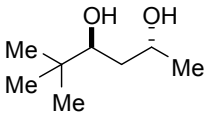
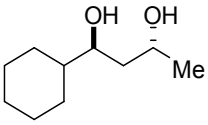
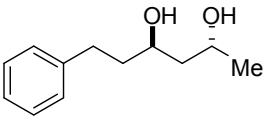
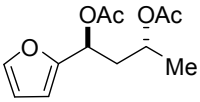
Structures not shown in the main manuscript but described here in the Supplementary Information (Master) and (NMR Spectra) documents are numbered with the form "***SI-##***".

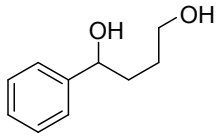
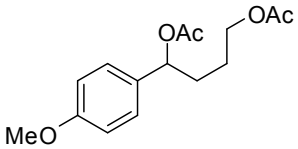
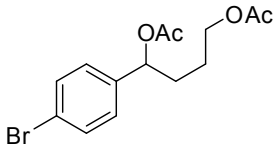
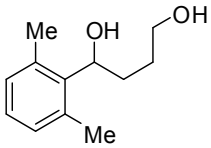
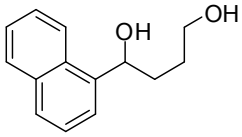
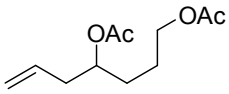
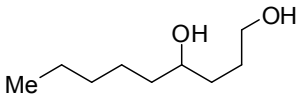
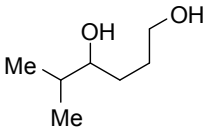
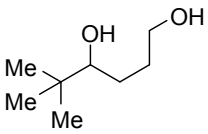
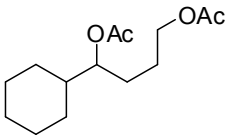
Structure	***Compound #***	***Procedure begins on page:***	***Compound characterization begins on page:***	***NMR spectra on page:***
	***1a***	11	11	53-54
	***1b***	11	12	55-56
	***1c***	11	12	57-58
	***1d***	11	13	59-60
	***1e***	11	13	61-62
	***1f***	11	14	63-64
	***1g***	11	14	65-66
	***1h***	11	15	67-68

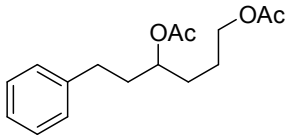
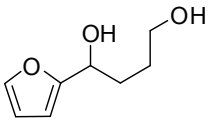
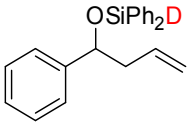
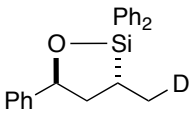
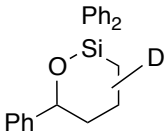
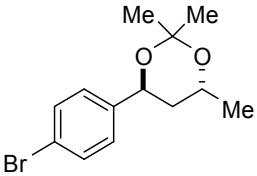
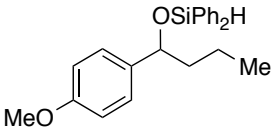
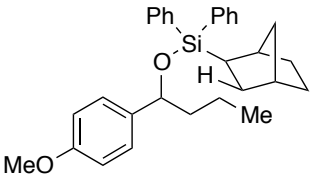
	***1i***	11	16	69-70
	***1j***	11	16	71-72
	***1k***	11	17	73-74
	***1l***	11	17	75-76
	*Trans-**2a***	18	19	77-78
	*Trans-**2b***	18	20	79-80
	*Trans-**2c***	18	20	81-82
	*Trans-**2d***	18	21	83-84
	*Trans-**2e***	18	21	85-86
	*Trans-**2f***	18	22	87-88
	*Trans-**2g***	18	23	89-90

	*Trans-**2h***	18	23	91-92
	*Trans-**2i***	18	24	93-94
	*Trans-**2j***	18	25	95-96
	*Trans-**2k***	18	25	97-98
	*Trans-**2l***	18	26	99-100
	***3a***	27	27	101-102
	***3b***	27	28	103-104
	***3c***	27	29	105-106
	***3d***	27	29	107-108

	***3e***	27	30	109-110
	***3f***	27	31	111-112
	***3g***	27	31	113-114
	***3h***	27	32	115-116
	***3i***	27	33	117-118
	***3j***	27	33	119-120
	***3k***	27	34	121-122
	***3l***	27	35	123-124
	*Anti-**4a***	36	36	125-126

	*Anti-**4b***	36	37	127-128
	*Anti-**4c**-diOAc*	36	37	129-130
	*Anti-**4d***	36	38	131-132
	*Anti-**4e***	36	38	133-134
	*Anti-**4f***	36	39	135-136
	*Anti-**4g**-diOAc*	36	39	137-138
	*Anti-**4h***	36	40	139-140
	*Anti-**4i***	36	40	141-142
	*Anti-**4j***	36	41	143-144
	*Anti-**4k***	36	41	145-146
	*Anti-**4l**-diOAc*	36	42	147-148

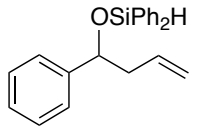
	***5a***	36	42	149-150
	***5b-diOAc***	36	43	151-152
	***5c-diOAc***	36	43	153-154
	***5d***	36	44	155-156
	***5e***	36	44	157-158
	***5f-diOAc***	36	45	159-160
	***5g***	36	45	161-162
	***5h***	36	46	163-164
	***5i***	36	46	165-166
	***5j-diOAc***	36	47	167-168

	***5k-diOAc***	36	47	169-170
	***5l***	36	48	171-172
	***1a-D***	11	48	173
	***Trans-2a-D***	18	49	174
	***3a-D***	27	49	175
	***SI-1***	50	50	176-177
	***6***	11	50	178-179
	***7***	18	51	180-181

General Procedure for Preparation of Homoallyl Silyl Ethers (1):[1]

Diphenylsilane (1.3 mL, 7.2 mmol) was dissolved with DCM (28 mL, 0.25 M), and the reaction mixture was cooled to 0 °C with an ice bath and stirred at 0 °C for 5 min. NBS (1.3 g, 7.2 mmol) was added to the mixture in a one portion, and the reaction mixture was warmed to rt and stirred for 1 h. During this process, the reaction mixture turned from organge to colorless. The reaction mixture was again cooled to 0 °C with an ice bath, and stirred at 0 °C for 5 min. Pyridine (0.63 mL, 7.8 mmol) and homoallyl alcohol (6.0 mmol) were added to the reaction mixture, which was warmed to rt and stirred for 1 h. The volatiles were removed *in vacuo*, and the resulting mixture was dissolved with pentane, filtered through a pad of Celite®, and concentrated *in vacuo* to afford the homoallyl silyl ether **1**, which was directly used for a subsequent reaction without further purification. For an analytical purpose, the product was purifed by MPLC (hexanes/EtOAc =80:1, 7 mL/min, retention time 5-10 min). Partial decomposition of the silyl ethers was observed during this MPLC separation.

(1-Phenylbut-3-enyloxy)diphenylsilane (1a)



1a

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.59 [dd, J = 8.0, 1.4 Hz, 2H, Si{$C_6(H_{ortho})_2H_4$}$_2$], 7.53 [dd, J = 8.0, 1.4 Hz, 2H, Si{$C_6(H_{ortho})_2H_4$}$_2$], 7.48-7.30 [m, 10H, *Ph*CH, Si{$C_6(H_{ortho})_2(H_{meta})_2H_{para}$}$_2$], 7.29-7.25 (nfom, 1H, *Ph*CH), 5.78 (dddd, J = 17.5, 10.5, 7.0, 7.0 Hz, 1H, CH_2=C*H*), 5.40 (s, 1H, Si*H*), 5.04 (dddd, J = 17.5, 2.2, 1.3, 1.3 Hz, 1H, $CH_{trans}H_{cis}$=CH), 5.03 (dddd, J = 10.5, 2.2, 1.3, 1.3 Hz, 1H, $CH_{trans}H_{cis}$=CH), 4.85 (dd, J = 7.2, 5.6 Hz, 1H, SiOC*H*CH_2), 2.65 (ddddd, J = 14.0, 7.2, 7.2, 1.3, 1.3 Hz, 1H, CH_aH_bCH=CH_2), and 2.53 (ddddd, J = 14.0, 7.0, 5.6, 1.3, 1.3 Hz, 1H, CH_aH_bCH=CH_2).

^{13}C NMR ($CDCl_3$, 125 MHz): δ 143.5, 135.0, 134.9, 134.8, 134.3, 134.1, 130.5, 130.4, 128.3, 128.15, 128.07, 127.5, 126.4, 117.6, 76.7, and 44.6.

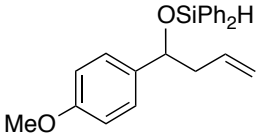
GC-MS (5029017): t_R=14.24 min, m/z 289 [(M-allyl)$^+$, 100], 259 (70), 211 (72), and 183 (90).

IR (neat): 3069 (w), 2127 (m), 1589 (w), 1428 (m), 811 (s), and 695 (s) cm^{-1}.

HRMS (ESI/TOF): Calcd for ($C_{22}H_{22}NaOSi$)$^+$: 353.1332. Found: 353.1329.

[1] Petit, M.; Chouraqui, G.; Aubert, C.; Malacria, M. *Org. Lett.* **2003**, *5*, 2037-2040.)

[1-(4-Methoxyphenyl)but-3-enyloxy]diphenylsilane (1b)



1b

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.62 [dd, J = 8.0, 1.4 Hz, 2H, Si{$C_6(H_{ortho})_2H_4$}$_2$], 7.56 [dd, J = 8.0, 1.4 Hz, 2H, Si{$C_6(H_{ortho})_2H_4$}$_2$], 7.47-7.33 [m, 6H, Si{$C_6(H_{ortho})_2(H_{meta})_2H_{para}$}$_2$], 7.23 (d, J = 8.7 Hz, 2H, *p*-MeO*Ph*), 6.85 (d, J = 8.7 Hz, 2H, *p*-MeO*Ph*), 5.77 (dddd, J = 17.2, 10.4, 7.0, 7.0 Hz, 1H, CH_2=C*H*), 5.38 (s, 1H, Si*H*), 5.05 (dddd, J = 17.2, 2.2, 1.2, 1.2 Hz, 1H, $CH_{trans}H_{cis}$=CH), 5.03 (dddd, J = 10.4, 2.2, 1.2, 1.2 Hz, 1H, $CH_{trans}H_{cis}$=CH), 4.08 (dd, J = 7.2, 5.7 Hz, 1H, SiOC*H*CH_2), 3.82(s, 3H, *Me*O), 2.65 (ddddd, J = 14.1, 7.2, 7.0, 1.2, 1.2 Hz, 1H, CH_aH_bCH=CH_2), and 2.51 (ddddd, J = 14.1, 7.0, 5.7, 1.2, 1.2 Hz, 1H, CH_aH_bCH=CH_2).

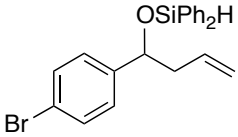
^{13}C NMR ($CDCl_3$, 125 MHz): δ 159.0, 135.7, 134.99, 134.95, 134.87, 134.4, 134.2, 130.5, 130.4, 128.12, 128.04, 127.7, 117.5, 113.7, 76.4, 55.4, and 44.6.

GC-MS (5032021): t_R=15.156 min, m/z 319 [(M-allyl)$^+$, 100], 197 (50), and 183 (60).

IR (neat): 3069 (w), 2126 (m), 1588 (w), 1511 (m), 1246 (m), 810 (s), and 696 (s) cm^{-1}.

HRMS (ESI/TOF): Calcd for ($C_{23}H_{24}NaO_2Si$)$^+$: 383.1438. Found: 383.1428.

[1-(4-Bromophenyl)but-3-enyloxy]diphenylsilane (1c)



1c

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.61 [dd, J = 8.0, 1.4 Hz, 2H, Si{$C_6(H_{ortho})_2H_4$}$_2$], 7.56 [dd, J = 8.0, 1.4 Hz, 2H, Si{$C_6(H_{ortho})_2H_4$}$_2$], 7.48-7.34 [m, 8H, *p*-Br*Ph*, Si{$C_6(H_{ortho})_2(H_{meta})_2H_{para}$}$_2$], 7.17 (d, J = 8.4 Hz, 2H, *p*-Br*Ph*), 5.74 (dddd, J = 17.3, 10.6, 7.0, 7.0 Hz, 1H, CH_2=C*H*), 5.39 (s, 1H, Si*H*), 5.04 (dddd, J = 10.5, 2.2, 1.2, 1.2 Hz, 1H, $CH_{trans}H_{cis}$=CH), 5.02 (dddd, J = 17.3, 2.2, 1.2, 1.2 Hz, 1H, $CH_{trans}H_{cis}$=CH), 4.80 (dd, J = 7.0, 5.7 Hz, 1H, SiOC*H*CH_2), 2.60 (ddddd, J = 14.0, 7.0, 7.0, 1.2, 1.2 Hz, 1H, CH_aH_bCH=CH_2), and 2.49(ddddd, J = 14.0, 7.0, 5.7, 1.2, 1.2 Hz, 1H, CH_aH_bCH=CH_2).

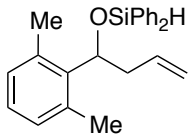
^{13}C NMR ($CDCl_3$, 125 MHz): δ 142.5, 135.0, 134.8, 134.2, 133.9, 133.8, 131.4, 130.63, 130.55, 128.21, 128.18, 128.15, 121.3, 118.0, 76.0, and 44.5.

GC-MS (5032021): t_R=15.424 min, m/z 467 [(M-allyl)$^+$, 80], 469 [(M-allyl)$^+$, isotope, 80], 245 (30), and 183 (100).

IR (neat): 3069 (w), 2349 (m), 1590 (w), 1428 (m), 1405 (m), 812 (s), and 695 (s) cm^{-1}.

HRMS (ESI/TOF): Calcd for ($C_{22}H_{21}BrNaOSi)^+$: 431.0437. Found: 431.0428.

[1-(2,6-Dimethylphenyl)but-3-enyloxy]diphenylsilane (1d)



1d

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.56 [dd, J = 8.0, 1.4 Hz, 2H, Si{$C_6(H_{ortho})_2H_4$}$_2$], 7.51 [dd, J = 8.0, 1.4 Hz, 2H, Si{$C_6(H_{ortho})_2H_4$}$_2$], 7.46-7.30 [m, 6H, Si{$C_6(H_{ortho})_2(H_{meta})_2H_{para}$}$_2$], 7.04 (dd, J = 7.5, 7.5 Hz, 1H, diMe-*Ph*), 6.94(br s, 2H, diMe-*Ph*), 5.80 (dddd, J = 17.2, 10.1, 7.0, 7.0 Hz, 1H, CH_2=C*H*), 5.30 (s, 1H, Si*H*), 5.20 (dd, J = 8.6, 5.9 Hz, 1H, SiOC*H*CH_2), 5.08 (dddd, J = 17.2, 2.1, 1.2, 1.2 Hz, 1H, C$H_{trans}H_{cis}$=CH), 5.03 (dddd, J = 10.1, 2.1, 1.2, 1.2 Hz, 1H, $CH_{trans}H_{cis}$=CH), 2.85 (ddddd, J = 14.1, 8.6, 7.0, 1.2, 1.2 Hz, 1H, C$H_a$$H_b$CH=$CH_2$), and 2.54 (ddddd, J = 14.1, 7.0, 5.9, 1.2, 1.2 Hz, 1H, $CH_a$$H_b$CH=$CH_2$).

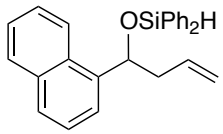
^{13}C NMR ($CDCl_3$, 125 MHz): δ 138.5, 135.5, 135.0, 134.8, 134.2 (2), 130.5, 130.4, 128.1 (2), 128.0 (2), 127.2, 117.2, 73.6, 42.1, and 20.9 (2).

GC-MS (5029017): t_R=14.694 min, m/z z 317 [(M-allyl)$^+$, 100], 239 (50), and 183 (60).

IR (neat): 3069 (w), 2126 (m), 1589 (w), 1428 (m), 811 (s), and 695 (s) cm^{-1}.

HRMS (ESI/TOF): Calcd for ($C_{24}H_{26}NaOSi)^+$: 381.1645. Found: 381.1638.

[1-(Naphthalen-1-yl)but-3-enyloxy]diphenylsilane (1e)



1e

^{1}H NMR ($CDCl_3$, 500 MHz): δ 8.04 (dd, J = 6.8, 2.4 Hz, 1H, *1-naph*), 7.90-7.88 (nfom, 1H, *1-naph*), 7.80 (d, J = 8.2 Hz, 1H, *1-naph*), 7.70 (d, J = 7.0 Hz, 1H, *1-naph*), 7.64 [dd, J = 8.0, 1.4 Hz, 2H, Si{$C_6(H_{ortho})_2H_4$}$_2$], 7.61 (dd, J = 8.0, 1.4 Hz, 1H, *1-naph*), 7.58 [dd, J = 8.0, 1.4 Hz, 2H, Si{$C_6(H_{ortho})_2H_4$}$_2$], 7.49-7.37 [m, 6H, Si{$C_6(H_{ortho})_2(H_{meta})_2H_{para}$}$_2$], 7.34-7.31 (m, 2H, *1-naph*), 5.90 (dddd, J = 17.2, 10.2, 7.0, 7.0 Hz, 1H, CH_2=C*H*), 5.64 (dd, J = 7.0, 5.1 Hz, 1H,

SiOC*H*CH_2), 5.44 (s, 1H, Si*H*), 5.08 (dddd, *J* = 17.2, 2.1, 1.2, 1.2 Hz, 1H, C$H_{trans}H_{cis}$=CH), 5.06 (dddd, *J* = 10.2, 2.1, 1.2, 1.2 Hz, 1H, $CH_{trans}H_{cis}$=CH), 2.80 (ddddd, *J* = 14.2, 7.0, 7.0, 1.2, 1.2 Hz, 1H, C$H_a$$H_b$CH=$CH_2$), and 2.76 (ddddd, *J* = 14.2, 7.0, 5.1, 1.2, 1.2 Hz, 1H, $CH_a$$H_b$CH=$CH_2$).

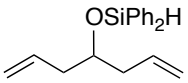
^{13}C NMR ($CDCl_3$, 125 MHz): δ 139.1, 135.1, 135.0, 134.9, 134.5, 134.2, 134.1, 133.9, 130.5, 130.44, 130.41, 130.37, 129.0, 128.14, 128.0, 125.9, 125.5, 124.3, 123.6, 117.5, 74.2, and 43.9.

GC-MS (5032021): t_R=16.205 min, m/z z 339 [(M-allyl)$^+$, 15], 309 (10), 261 (60), and 153 (100).

IR (neat): 3068 (w), 2128 (m), 1640 (w), 1589 (w), 1428 (m), 812 (s), and 695 (s) cm^{-1}.

HRMS (ESI/TOF): Calcd for ($C_{26}H_{24}NaOSi$)$^+$: 403.1489. Found: 403.1476.

(Hepta-1,6-dien-4-yloxy)diphenylsilane (1f)



1f

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.66 [dd, *J* = 8.0, 1.5 Hz, 4H, Si{C_6(H_{ortho})$_2$$H_4$}$_2$], 7.47-7.39 [m, 6H, Si{$C_6$($H_{ortho}$)$_2$($H_{meta}$)$_2$$H_{para}$}$_2$], 5.83 (dddd, *J* = 17.6, 10.6, 7.1, 7.1 Hz, 2H, CH_2=C*H*), 5.50 (s, 1H, Si*H*), 5.07 (dddd, *J* = 17.6, 2.1, 1.2, 1.2 Hz, 2H, C$H_{trans}H_{cis}$=CH), 5.06 (dddd, *J* = 9.7, 2.1, 1.2, 1.2 Hz, 2H, $CH_{trans}H_{cis}$=CH), 3.96 (dddd, *J* = 5.9, 5.9, 5.9, 5.9 Hz, 1H, SiOC*H*CH_2), and 2.35 (ddddd, *J* = 14.0, 7.1, 5.9, 1.2, 1.2 Hz, 2H, C$H_a$$H_b$CH=$CH_2$), and 2.32 (ddddd, *J* = 14.0, 7.1, 5.9, 1.2, 1.2 Hz, 2H, $CH_a$$H_b$CH=$CH_2$).

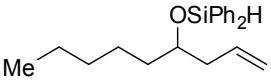
^{13}C NMR ($CDCl_3$, 125 MHz): δ 134.9 (2), 134.6, 130.5, 128.1, 117.6, 74.0, and 41.2.

GC-MS (5029017): t_R=12.574 min, m/z 253 [(M-allyl)$^+$, 55], 225 (10), 211 (60), and 183 (100).

IR (neat): 3070 (w), 2120 (m), 1641 (w), 1590 (w), 1428 (m), 812 (s), and 695 (s) cm^{-1}.

HRMS (ESI/TOF): Calcd for ($C_{19}H_{22}NaOSi$)$^+$: 317.1332. Found: 317.1314.

(Non-1-en-4-yloxy)diphenylsilane (1g)



1g

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.66-7.64 [nfom, 4H, Si{C_6(H_{ortho})$_2$$H_4$}$_2$], 7.46-7.38 [m, 6H, Si{$C_6$($H_{ortho}$)$_2$($H_{meta}$)$_2$$H_{para}$}$_2$], 5.82 (dddd, *J* = 17.3, 10.3, 7.1, 7.1 Hz, 1H, CH_2=C*H*), 5.48 (s, 1H, Si*H*), 5.04 (dddd, *J* = 17.3, 2.2, 1.2, 1.2 Hz, 2H, C$H_{trans}H_{cis}$=CH), 5.03 (dddd, *J* = 10.4, 2.2, 1.2, 1.2 Hz, 1H, $CH_{trans}H_{cis}$=CH), 3.88 (dddd, *J* = 7.1, 5.9, 5.9, 5.0 Hz, 1H, SiOC*H*CH_2), 2.33 (ddddd,

J = 15.0, 7.1, 5.9, 1.2, 1.2 Hz, 1H, CH_aH$_b$CH=CH$_2$), 2.28 (ddddd, J = 15.0, 7.1, 5.9, 1.2, 1.2 Hz, 1H, CH$_a$$H_b$CH=CH$_2$), 1.58-1.46 (m, 2H, SiOCHCH_aH$_b$C$_4$H$_9$, SiOCHCH$_a$$H_bC_4H_9$ or SiOCHCH$_2$CH_aH$_b$C$_3$H$_7$), 1.43-1.35 (nfom, 1H, SiOCHCH$_a$$H_bC_4H_9$ or SiOCHCH$_2$CH_aH$_b$C$_3$H$_7$), 1.32-1.16 (m, 5H, SiOCHCH$_2$CH$_a$$H_bCH_2CH_2$), and 0.85 (dd, J = 7.1, 7.1 Hz, 3H, CH_3).

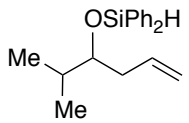
^{13}C NMR ($CDCl_3$, 125 MHz): δ 135.7, 135.0, 134.96, 130.4, 128.1, 117.2, 74.5, 41.8, 36.7, 32.0, 25.3, 22.8, and 14.2.

GC-MS (5029017): t_R=13.400 min, m/z 323 [(M-H)$^+$, 10], 283 [(M-allyl)$^+$, 75], 247 (30), 199 (30), and 183 (100).

IR (neat): 3070 (w), 2120 (m), 1641 (w), 1590 (w), 1465 (m), 811 (s), and 695 (s) cm^{-1}.

HRMS (ESI/TOF): Calcd for ($C_{21}H_{28}NaOSi$)$^+$: 347.1802. Found: 347.1801.

(2-Methylhex-5-en-3-yloxy)diphenylsilane (1h)



1h

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.67-7.65 [nfom, 4H, Si{C$_6$(H_{ortho})$_2$H$_4$}$_2$], 7.46-7.38 [m, 6H, Si{C$_6$(H_{ortho})$_2$H$_4$}$_2$], 5.81 (dddd, J = 17.2, 10.1, 7.1, 7.1 Hz, 1H, CH$_2$=CH), 5.49 (s, 1H, SiH), 5.05 (dddd, J = 17.2, 2.2, 1.2, 1.2 Hz, 1H, CH_{trans}H$_{cis}$=CH), 5.01 (dddd, J = 10.2, 2.2, 1.2, 1.2 Hz, 1H, CH$_{trans}$$H_{cis}$=CH), 3.67 (ddd, J = 6.6, 5.1, 5.1 Hz, 1H, SiOCHCH$_2$), 2.32 (ddddd, J = 14.2, 6.6, 6.6, 1.2, 1.2 Hz, 1H, CH_aH$_b$CH=CH$_2$), 2.28 (ddddd, J = 14.2, 6.8, 5.1, 1.2, 1.2 Hz, 1H, CH$_a$$H_b$CH=CH$_2$), 1.80 [qqd, J = 6.8, 6.8, 5.1 Hz, 1H, SiOCHCH(Me)$_2$], 0.93 (d, J = 6.8 Hz, 3H, CH_3), and 0.91 (d, J = 6.8 Hz, 3H, CH_3).

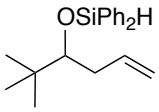
^{13}C NMR ($CDCl_3$, 125 MHz): δ 135.7, 135.00, 134.96, 130.4, 128.1, 117.0, 79.3, 38.7, 33.0, 19.1, and 17.5.

GC-MS (5029017): t_R=12.538 min, m/z 295 [(M-H)$^+$, 10], 255 [(M-^{i}Pr)$^+$, 20], 219 (70), and 183 (100).

IR (neat): 3069 (w), 2125 (m), 1640 (w), 1590 (w), 1467 (m), 812 (s), and 695 (s) cm^{-1}.

HRMS (ESI/TOF): Calcd for ($C_{19}H_{24}NaOSi$)$^+$: 319.1489. Found: 319.1487.

(2,2-Dimethylhex-5-en-3-yloxy)diphenylsilane (1i)



1i

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.65 [d, J = 7.4 Hz, 4H, Si{$C_6(H_{ortho})_2H_4\}_2$], 7.45-7.37 [m, 6H, Si{$C_6(H_{ortho})_2(H_{meta})_2H_{para}\}_2$], 5.76 (dddd, J = 17.1, 10.1, 7.1, 7.1 Hz, 1H, CH_2=C*H*), 5.52 (s, 1H, Si*H*), 5.00 (dddd, J = 17.2, 2.2, 1.1, 1.1 Hz, 1H, $CH_{trans}H_{cis}$=CH), 4.92 (dddd, J = 10.1, 2.2, 1.1, 1.1 Hz, 1H, $CH_{trans}H_{cis}$=CH), 3.49 (dd, J = 8.8, 2.9 Hz, 1H, SiOC*H*CH_2), 2.33 (ddddd, J = 14.3, 7.1, 2.9, 1.1, 1.1 Hz, 1H, CH_aH_bCH=CH_2), 2.24 (ddddd, J = 14.3, 8.8, 7.1, 1.1, 1.1 Hz, 1H, CH_aH_bCH=CH_2), and 0.91 [s, 9H, $C(CH_3)_3$].

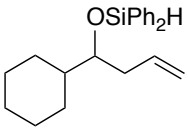
^{13}C NMR ($CDCl_3$, 125 MHz): δ 137.4, 135.23, 135.19, 130.3, 128.0, 116.8, 82.9, 37.5, 36.3, and 26.5.

GC-MS (5029017): t_R=12.785 min, m/z 309 [$(M-H)^+$, 10], 269 [$(M\text{-allyl})^+$, 100], 233 (70), and 183 (100).

IR (neat): 3069 (w), 2125 (m), 1640 (w), 1590 (w), 1428 (m), 808 (s), and 695 (s) cm^{-1}.

HRMS (ESI/TOF): Calcd for $(C_{20}H_{26}NaOSi)^+$: 333.1645. Found: 333.1638.

(1-Cyclohexylbut-3-enyloxy)diphenylsilane (1j)



1j

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.64 [app d, J = 6.9 Hz, 4H, Si{$C_6(H_{ortho})_2H_4\}_2$], 7.45-7.37 [m, 6H, Si{$C_6(H_{ortho})_2(H_{meta})_2H_{para}\}_2$], 5.79 (dddd, J = 17.2, 10.2, 7.1, 7.1 Hz, 1H, CH_2=C*H*), 5.46 (s, 1H, Si*H*), 5.02 (dddd, J = 17.2, 2.2, 1.3, 1.3 Hz, 1H, $CH_{trans}H_{cis}$=CH), 4.99 (dddd, J = 10.4, 2.2, 1.3, 1.3 Hz, 1H, $CH_{trans}H_{cis}$=CH), 3.63 (ddd, J = 5.6, 5.6, 5.6 Hz, 1H, SiOC*H*CH_2), 2.31 (ddddd, J = 14.3, 7.1, 5.6, 1.3, 1.3 Hz, 1H, CH_aH_bCH=CH_2), 2.28 (ddddd, J = 14.3, 7.1, 5.6, 1.3, 1.3 Hz, 1H, CH_aH_bCH=CH_2), 1.82 (ddddd, J = 14.2, 3.5, 3.5, 3.5, 1.9 Hz, 1H, *Cy*), 1.75-1.68 (m, 2H, *Cy*), 1.65-1.58 (m, 2H, *Cy*), 1.45 (ddddd, J = 11.7, 11.7, 5.3, 3.4, 3.4 Hz, 1H, *Cy*), and 1.19-1.02 (m, 5H, *Cy*).

^{13}C NMR ($CDCl_3$, 125 MHz): δ 135.6, 135.05, 135.01, 130.4, 128.1, 117.0, 78.7, 43.0, 38.6,

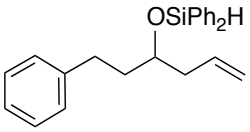
29.4, 28.2, 26.8, 26.6, and 26.4.

GC-MS (5029017): t_R=14.303 min, m/z 295 [(M-allyl)$^+$, 40], 255 [(M-Cy)$^+$, 70], 199 (70), and 183 (100).

IR (neat): 3069 (w), 2120 (m), 1640 (w), 1590 (w), 1428 (m), 813 (s), and 696 (s) cm^{-1}.

HRMS (ESI/TOF): Calcd for $(C_{22}H_{28}NaOSi)^+$: 359.1802. Found: 359.1792.

(1-Phenylhex-5-en-3-yloxy)diphenylsilane (1k)



1k

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.67-7.64 [m, 4H, Si{C_6(H_{ortho})$_2$$H_4$}$_2$], 7.47-7.38 [m, 6H, Si{$C_6$($H_{ortho}$)$_2$($H_{meta}$)$_2$$H_{para}$}$_2$], 7.25 (dd, J = 7.5, 7.5 Hz, 2H, *Ph-*$H_{meta}$$CH_2$), 7.16 (dddd, J = 7.5, 7.5, 1.5, 1.5 Hz, 1H, *Ph-*$H_{para}$$CH_2$), 7.08 (dd, J = 7.5, 1.5 Hz, 2H, *Ph-*$H_{ortho}$$CH_2$), 5.80 (dddd, J = 17.5, 10.4, 7.1, 7.1 Hz, 1H, CH_2=C*H*), 5.51 (s, 1H, Si*H*), 5.05 (dddd, J = 17.5, 2.2, 1.3, 1.3 Hz, 1H, C$H_{trans}$$H_{cis}$=CH), 5.03 (dddd, J = 10.4, 2.2, 1.3, 1.3 Hz, 1H, $CH_{trans}$$H_{cis}$=CH), 3.94 (dddd, J = 7.1, 5.9, 5.9, 4.6 Hz, 1H, SiOC*H*CH_2), 2.75 (ddd, J = 13.7, 10.0, 6.2 Hz, 1H, PhC$H_a$$H_b$), 2.58 (ddd, J = 13.7, 10.0, 6.4 Hz, 1H, Ph$CH_a$$H_b$), 2.37 (ddddd, J = 14.1, 7.1, 5.9, 1.3, 1.3 Hz, 2H, C$H_a$$H_b$CH=$CH_2$), 2.33 (ddddd, J = 14.1, 7.1, 5.9, 1.3, 1.3 Hz, 2H, $CH_a$$H_b$CH=$CH_2$), 1.86 (dddd, J = 13.8, 10.0, 7.1, 5.9 Hz, 1H, PhCH_2C$H_a$$H_b$), and 1.82 (dddd, J = 13.8, 10.0, 6.2, 4.6 Hz, 1H, Ph$CH_2$$CH_a$$H_b$).

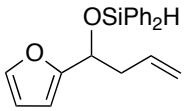
^{13}C NMR ($CDCl_3$, 125 MHz): δ 142.4, 134.99, 134.88, 134.65, 134.53, 130.53, 130.50, 128.62, 128.58, 128.52, 128.23, 128.18, 125.9, 117.6, 74.0, 41.7, 38.5, and 32.0.

GC-MS (5032021): t_R=15.578 min, m/z 357 [(M-H)$^+$, 2], 317 [(M-allyl)$^+$, 10], 199 (80), and 188 (100).

IR (neat): 3068 (w), 2125 (m), 1640 (w), 1590 (w), 1428 (m), 812 (s), and 695 (s) cm^{-1}.

HRMS (ESI/TOF): Calcd for $(C_{24}H_{26}NaOSi)^+$: 381.1645. Found: 381.1638.

[1-(Furan-2-yl)but-3-enyloxy]diphenylsilane (1l)



1l

This NMR includes 1,1,3,3-tetraphenyldisiloxane impurity (ca. 5%).

^{1}H NMR ($CDCl_3$, 500 MHz) of **1l**: δ 7.59 [dd, J = 8.0, 1.4 Hz, 2H, Si{$C_6(H_{ortho})_2H_4$}$_2$], 7.56 [dd, J = 8.0, 1.4 Hz, 2H, Si{$C_6(H_{ortho})_2H_4$}$_2$], 7.46-7.35 [m, 6H, Si{$C_6(H_{ortho})_2(H_{meta})_2H_{para}$}$_2$], 7.33 (dd, J = 1.8, 0.8 Hz, 1H, furanyl C5-*H*), 6.28 (dd, J = 3.2, 1.8 Hz, 1H, furanyl C4-*H*), 6.17 (d, J = 3.2 Hz, 1H, furanyl C3-*H*), 5.78 (dddd, J = 17.2, 10.2, 7.0, 7.0 Hz, 1H, CH_2=C*H*), 5.40 (s, 1H, Si*H*), 5.09 (dddd, J = 17.2, 2.2, 1.1, 1.1 Hz, 1H, C$H_{trans}$$H_{cis}$=CH), 5.05 (dddd, J = 10.2, 2.2, 1.1, 1.1 Hz, 1H, $CH_{trans}H_{cis}$=CH), 4.88 (dd, J = 6.7, 6.7 Hz, 1H, SiOC*H*CH_2), 2.73 (ddddd, J = 14.1, 7.0, 6.7, 1.1, 1.1 Hz, 1H, C$H_a$$H_b$CH=$CH_2$), and 2.67 (ddddd, J = 14.1, 7.0, 6.7, 1.1, 1.1 Hz, 1H, CH_aH_bCH=CH_2).

^{1}H NMR ($CDCl_3$, 500 MHz) of 1,1,3,3-Tetraphenyldisiloxane: δ 7.56 (dd, J = 7.5, 1.4 Hz, 8H, *Ph*), 7.42 (dddd, J = 7.5, 7.5, 1.4, 1.4 Hz, 4H, *Ph*), 7.35 (dd, J = 7.5, 7.5 Hz, 8H, *Ph*), and 5.60 (s, 2H, Si*H*).

^{13}C NMR ($CDCl_3$, 125 MHz) of **1l**: δ 155.4, 142.0, 135.0, 134.9, 134.5, 134.2, 130.5, 130.4, 128.16, 128.13, 128.06, 117.8, 110.1, 107.2, 69.9, and 40.7.

GC-MS (5029017): t_R=13.439 min, m/z 320 [M^+, 1], 279 [(M-allyl)$^+$, 100], 201 (80), and 183 (70).

IR (neat): 3069 (w), 2132 (m), 1590 (w), 1428 (m), 815 (s), and 696 (s) cm^{-1}.

HRMS (ESI/TOF): Calcd for $(C_{20}H_{20}NaO_2Si)^+$: 343.1125. Found: 343.1116.

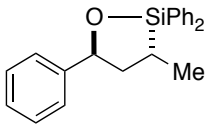
General Procedure for Preparation of 1,3-*trans*-Oxasilacyclopentanes (*trans-2*):

[Rh(nbd)Cl]$_2$ (4.6 mg, 0.010 mmol, 5 mol %) and *rac*-BINAP (18.7 mg, 0.015 mmol, 15 mol %) were added to a flame-dried, septum capped vial. The mixture was dissolved with THF (0.50 mL), which was stirred for 1 h at rt. Volatiles including norbornadiene (nbd) were removed *in vacuo*, and the resulting mixture was kept under vaccum for 0.5 h (the absence of nbd was confirmed by GC/MS analysis). Norbornene (3.7 mg, 0.040 mmol, 20 mol %) and THF (0.40 mL, 0.50 M) were added to the prepared catalyst, which was cooled to –78 °C. Homoallyl sily ether (0.2 mmol) dissolved in THF [0.40 mL for **1a**, **1b**, **1d**, **1h, 1i**, **1j** (total concentration: 0.25 M); 7.6 mL for **1c**, **1e**, **1f, 1g**, **1l** (total concentration: 0.025 M)] was added dropwise to the reaction mixture via a cannular. The septum on the vial was replaced by a screw cap with a Teflon liner, and the mixture was stirred at –78 °C for 2 h and the reaction vessel was then kept in a fridge (0 °C) for 24 h. The reaction mixture was warmed to rt and the volatiles were

removed *in vacuo*. The reaction progress was monitored by GC-MS spectrometry. The yield of the reaction was determined by NMR spectroscopy by an addition of DMF (16 μL, 0.20 mmol) as an internal standard. The regio- and diastereomeric ratios were determined by NMR spectroscopy. For an analytical purpose, the product was purifed by MPLC (hexanes/EtOAc =80:1, 5 mL/min, retention time 10-20 min). Partial decomposition of the product was observed during this MPLC separation.

Note: During this hydrosilylative cyclization, the formation of norbornane (via reduction of nbe) was not observed by GC/MS analysis [5029017 or 5032021 method], where the retention time of each bicyclic compound is 2.80 min (nbd), 2.96 min (nbe), and 3.29 min (norbornane).

***Trans*-3-Methyl-2,2,5-triphenyl-1,2-oxasilolane (*trans*-2a)**



*trans-**2a***

Regio- and Diastereomeric Ratios: 2:**3** = 89:11, *trans*-**2a**: *cis*-**2a** = 93:7.

NMR Yield: 81%.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.69 [dd, J = 7.9, 1.4 Hz, 2H, Si{$C_6(H_{ortho})_2H_4$}$_2$], 7.67 [dd, J = 7.9, 1.4 Hz, 2H, Si{$C_6(H_{ortho})_2H_4$}$_2$], 7.49-7.37 [m, 8H, *Ph*CH, Si{$C_6(H_{ortho})_2(H_{meta})_2H_{para}$}$_2$], 7.32 (dd, J = 7.5, 7.5 Hz, 2H, *Ph*CH), 7.26-7.24 [nfom, 1H, *Ph*CH], 5.41 (dd, J = 7.6, 5.8 Hz, 1H, SiOC*H*), 2.23 (ddd, J = 13.3, 7.6, 6.8 Hz, 1H, CH_aH_bCHMe), 2.18 (ddd, J = 13.3, 5.8, 5.1 Hz, 1H, CH_aH_bCHMe), 1.85 (qdd, J = 7.7, 6.8, 5.1 Hz, 1H, SiC*H*Me), and 1.09 (d, J = 7.7 Hz, 3H, SiCHCH_3).

^{13}C NMR ($CDCl_3$, 125 MHz): δ 144.7, 135.1, 134.9, 134.8, 133.1, 130.5, 130.3, 128.4, 128.2, 128.0, 127.2, 125.6, 82.4, 78.8, 44.3, 17.0, and 15.3.

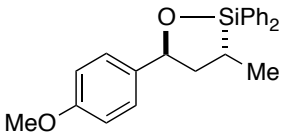
TLC: R_f = 0.25 in 20:1 hexanes: EtOAc.

GC-MS (5029017): t_R=15.599 min, m/z 329 [$(M-H)^+$, 10], 288 (100), 253 [$(M-Ph)^+$, 10], and 210 (100).

IR (neat): 3068 (w), 3025 (w), 1428 (m), 1117 (m), 698 (s), and 510 (s) cm^{-1}.

HRMS (ESI/TOF): Calcd for $(C_{22}H_{22}NaOSi)^+$: 353.1332. Found: 353.1327.

***Trans*-5-(4-Methoxyphenyl)-3-methyl-2,2-diphenyl-1,2-oxasilolane (*trans*-2b)**



*trans-**2b***

Regio- and Diastereomeric Ratios: 2:**3** = 86:14, *trans*-**2b**: *cis*-**2b** = 91:9.

NMR Yield: 75%.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.67 [dd, J = 7.9, 1.2 Hz, 2H, Si{$C_6(H_{ortho})_2H_4$}$_2$], 7.65 [dd, J = 7.8, 1.2 Hz, 2H, Si{$C_6(H_{ortho})_2H_4$}$_2$], 7.48-7.38 [m, 6H, Si{$C_6(H_{ortho})_2(H_{meta})_2H_{para}$}$_2$], 7.28 (d, J = 8.6 Hz, 2H, *p*-MeO*Ph*), 6.88 (d, J = 8.6 Hz, 2H, *p*-MeO*Ph*), 5.36 (dd, J = 7.9, 5.4 Hz, 1H, SiOC*H*CH_2), 3.79 (s, 3H, OCH_3), 2.20 (ddd, J = 13.4, 7.9, 7.0 Hz, 1H, C$H_a$$H_b$CHMe), 2.13 (ddd, J = 13.4, 5.4, 5.2 Hz, 1H, $CH_a$$H_b$CHMe), 1.83 (qdd, J =7.7, 7.0, 5.2 Hz, 1H, SiC*H*Me), and 1.08 (d, J =7.7 Hz, 3H, SiCHCH_3).

^{13}C NMR ($CDCl_3$, 125 MHz): δ 158.9, 136.8, 135.1, 135.0, 134.8, 133.2, 130.4, 130.2, 128.2, 128.0, 126.9, 113.9, 82.4, 78.5, 55.5, 44.3, 17.0, and 15.3.

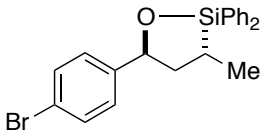
TLC: R_f = 0.20 in 20:1 hexanes: EtOAc.

GC-MS (5032021): t_R=16.378 min, m/z 317 (60), 287 (100), and 240 (40).

IR (neat): 3067 (w), 3047 (w), 1511 (m), 1243 (m), 1069 (s), 697 (s), and 510 (s) cm^{-1}.

HRMS (ESI/TOF): Calcd for $(C_{23}H_{24}NaO_2Si)^+$: 383.1438. Found: 383.1434.

***Trans*-5-(4-Bromophenyl)-3-methyl-2,2-diphenyl-1,2-oxasilolane (*trans*-2c)**



*trans-**2c***

Regio- and Diastereomeric Ratios: 2:**3** = 84:16, *trans*-**2c**: *cis*-**2c** = 83:17.

NMR Yield: 67%.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.66 [ddd, J = 7.3, 7.3, 1.2 Hz, 2H, Si{$C_6(H_{ortho})_2H_4$}$_2$], 7.49-7.40 [m, 8H, Si{$C_6(H_{ortho})_2(H_{meta})_2H_{para}$}$_2$, *p*-Br*Ph*], 7.24 (d, J = 6.6, 1.8 Hz, 2H, *p*-Br*Ph*), 5.37 (dd, J = 6.9, 6.9 Hz, 1H, SiOC*H*CH_2), 2.18 (ddd, J = 13.4, 6.9, 5.6 Hz, 1H, C$H_a$$H_b$CHMe), 2.15 (ddd, J = 13.4, 7.6, 6.9 Hz, 1H, $CH_a$$H_b$CHMe), 1.83 (qdd, J = 7.6, 7.6, 5.6 Hz, 1H, SiC*H*Me), and 1.08 (d, J = 7.6 Hz, 3H, SiCHCH_3).

^{13}C NMR ($CDCl_3$, 125 MHz): δ 143.8, 135.1, 134.8, 134.6, 132.8, 131.5, 130.6, 130.4, 128.3, 128.1, 127.3, 120.9, 78.1, 44.2, 16.9, and 15.2.

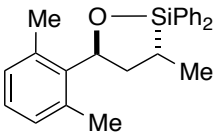
TLC: R_f = 0.25 in 20:1 hexanes: EtOAc.

GC-MS (5032021): t_R=16.756 min, m/z 368 (30), 366 (30), 287 (100), and 181 (60).

IR (neat): 3068 (w), 3047 (w), 2925 (m), 1453 (m), 1241 (m), 698 (s), and 514 (s) cm^{-1}.

HRMS (ESI/TOF): Calcd for $(C_{22}H_{21}BrNaOSi)^+$: 431.0437. Found: 431.0447.

***Trans*-5-(2,6-Dimethylphenyl)-3-methyl-2,2-diphenyl-1,2-oxasilolane (*trans*-2d)**



*trans-**2d***

Regio- and Diastereomeric Ratios: 2:**3** = 85:15, *trans*-**2d**: *cis*-**2d** = 97:3.

NMR Yield: 80%.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.69 [dd, J = 7.7, 0.9 Hz, 2H, $Si\{C_6(H_{ortho})_2H_3\}_2$], 7.64 [dd, J = 7.4 , 0.9 Hz, 2H, $Si\{C_6(H_{ortho})_2H_3\}_2$], 7.44-7.40 [m, 6H, $Si\{C_6(H_{ortho})_2(H_{meta})_2H_{para}\}_2$], 7.05 [t, J = 7.5 Hz, 1H, $Me_2(C_6H_2H_{para})$], 6.98 [d, J = 7.5 Hz, 2H, $Me_2(C_6H_2H_{para})$], 5.75 (dd, J = 11.8, 4.1 Hz, 1H, SiOC*H*), 2.52 (ddd, J = 13.5, 11.8, 7.6 Hz, 1H, CH_aH_bCHMe), 2.34 [s, 6H, $(CH_3)_2C_6H_3$], 2.09 (app pent, J = 7.8 Hz, 1H, $SiCHCH_3$), 1.94 (ddd, J = 13.5, 4.1, 1.0 Hz, 1H, CH_aH_bCHMe), and 1.34 (d, J = 7.2 Hz, 3H, $SiCHCH_3$).

^{13}C NMR ($CDCl_3$, 125 MHz): δ 137.8, 136.2, 135.0, 134.9, 134.7, 134.5, 133.4, 130.5, 130.2, 129.6, 128.2, 128.1, 128.0, 127.2, 76.7, 40.7, 21.2 (2), 16.5, and 16.2.

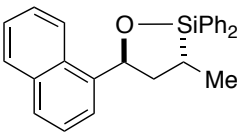
TLC: R_f = 0.25 in 20:1 hexanes: EtOAc.

GC-MS (5032021): t_R=18.444 min, m/z 380 (M^+, 15), 338 (100), 260 (90), 215 (50), and 167 (30).

IR (neat): 3068 (w), 3048 (w), 2943 (m), 1377 (m), 1265 (m), 696 (s), and 497 (s) cm^{-1}.

HRMS (ESI/TOF): Calcd for $(C_{24}H_{26}NaOSi)^+$: 381.1645. Found: 381.1630.

***Trans*-3-Methyl-5-(naphthalen-1-yl)-2,2-diphenyl-1,2-oxasilolane (*trans*-2e)**



*trans-**2e***

This NMR includes *cis*-**2e** (ca. 3.6%).

Regio- and Diastereomeric Ratios: 2:**3** = 86:14, *trans*-**2e**: *cis*-**2e** = 79:21.

NMR Yield: 62%.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 8.03 (app d, *J* = 8.0 Hz, 1H, *1-naph*), 7.89 (app d, *J* = 7.5 Hz, 1H, *1-naph*), 7.76 (m, 3H, *1-naph*), 7.72-7.69 [m, 2H, Si{$C_6(H_{ortho})_2H_3$}$_2$], 7.67 (d, *J* = 7.0 Hz, 1H, *1-naph*), 7.53-7.40 [m, 8H, Si*Ph-H*], 7.40 (d, *J* = 8.0 Hz, 1H, *1-naph*), 6.23 (app t, *J* = 6.3 Hz, 1H, SiOC*H*), 2.46 (dddd, *J* = 13.4, 5.7, 5.7, 0.7 Hz, 1H, C$H_a$$H_b$CHMe), 2.35 (dddd, *J* = 13.4, 7.0, 7.0, 0.7 Hz, 1H, $CH_a$$H_b$CHMe), 1.88 (app sextet, *J* = 7.0 Hz, 1H, SiC*H*CH_3), and 1.15 (d, *J* = 7.6 Hz, 3H, SiCHCH_3).

^{13}C NMR ($CDCl_3$, 125 MHz): δ 140.2, 135.2, 135.0, 134.7, 133.9, 133.2, 130.6, 130.34, 130.26, 129.1, 128.3, 128.1, 127.6, 125.9, 125.8, 125.5, 123.3, 122.4, 76.2, 43.2, 16.7, and 15.1.

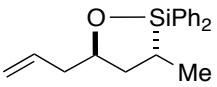
TLC: R_f = 0.25 in 20:1 hexanes: EtOAc.

GC-MS (5032021): t_R=15.599 min, m/z 329 [$(M-H)^+$, 10], 288 (100), 253 [$(M-Ph)^+$, 10], and 210 (100).

IR (neat): 3068 (w), 3048 (w), 2924 (m), 1452 (m), 1263 (m), 698 (s), and 512 (s) cm^{-1}.

HRMS (ESI/TOF): Calcd for $(C_{26}H_{24}NaOSi)^+$: 403.1489. Found: 403.1482.

***Trans*-5-Allyl-3-methyl-2,2-diphenyl-1,2-oxasilolane (*trans*-2f)**



*trans-**2f***

Regio- and Diastereomeric Ratios: 2:**3** = 90:10, *trans*-**2f**: *cis*-**2f** = 93:7.

NMR Yield: 76%.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.64 [dd, *J* =7.9, 1.2 Hz, 2H, Si{$C_6(H_{ortho})_2H_3$}$_2$], 7.59 [dd, *J* =7.6, 1.3 Hz, 2H, Si{$C_6(H_{ortho})_2H_3$}$_2$], 7.43-7.36 [m, 6H, Si{$C_6(H_{ortho})_2(H_{meta})_2H_{para}$}$_2$], 5.90 (dddd, *J* = 17.1, 10.2, 7.0, 7.0 Hz, 1H, CH_2=C*H*CH_2), 5.09 (dddd, *J* = 17.1, 2.1, 1.2, 1.2 Hz, 2H, C$H_{trans}$$H_{cis}$=CH), 5.05 (dddd, *J* = 10.2, 2.1, 1.2, 1.2 Hz, 2H, $CH_{trans}$$H_{cis}$=CH), 4.40(dddd, *J* = 6.5, 6.5, 6.5, 5.2 Hz, 1H, SiOC*H*), 2.47 (ddddd, *J* = 13.9, 6.5, 6.5, 1.2, 1.2 Hz, 1H, CH_2=CHC$H_a$$H_b$), 2.30 (ddddd, *J* = 13.9, 6.5, 6.5, 1.2, 1.2 Hz, 1H, CH_2=C*H*$CH_a$$H_b$), 2.00 (ddd, *J* = 13.2, 7.0, 6.8 Hz, 1H, $CH_a$$H_b$CHMe$SiPh_2$), 1.84 (ddd, *J* = 13.2, 6.1, 5.2 Hz, 1H, $CH_a$$H_b$CHMe$SiPh_2$), 1.74 (qdd, *J* = 7.5, 7.5, 6.0 Hz, 1H, SiC*H*CH_3), and 1.03 (d, *J* = 7.5 Hz, 3H, SiCHCH_3).

^{13}C NMR ($CDCl_3$, 125 MHz): δ 135.44, 135.39, 135.1, 134.6, 133.3, 130.3, 130.2, 128.1, 128.0,

127.9, 117.1, 77.1, 42.4, 40.3, 16.8, and 15.4.

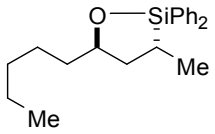
TLC: R_f = 0.25 in 20:1 hexanes: EtOAc.

GC-MS (5029017): t_R=13.243 min, m/z 253 [(M-allyl)$^+$, 100], 216 (18), 199 (50), and 175 (90).

IR (neat): 3069 (w), 3049 (w), 2924 (m), 1428 (m), 1116 (m), 632 (s), and 501 (s) cm^{-1}.

HRMS (ESI/TOF): Calcd for $(C_{19}H_{22}NaOSi)^+$: 317.1332. Found: 317.1327.

***T*rans-3-Methyl-5-pentyl-2,2-diphenyl-1,2-oxasilolane (*trans*-2g)**



*trans-**2g***

Regio- and Diastereomeric Ratios: 2:**3** = 74:26, *trans*-**2g**: *cis*-**2g** = 82:18.

NMR Yield: 58%.

This NMR includes unidentified impurities (ca. 4%).

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.65-7.63 [nfom, 2H, Si{$C_6(H_{ortho})_2H_3$}$_2$], 7.61-7.59 [nfom, 2H, Si{$C_6(H_{ortho})_2H_3$}$_2$], 7.44-7.36 [m, 6H, Si{$C_6(H_{ortho})_2(H_{meta})_2H_{para}$}$_2$], 4.33 (app pent, J = 6.0 Hz, 1H, SiOC*H*), 1.95 (ddd, J = 13.4, 7.1, 5.9 Hz, 1H, $CH_aH_bCHMeSiPh_2$), 1.85 (ddd, J = 13.4, 5.9, 5.6 Hz, 1H, $CH_aH_bCHMeSiPh_2$), 1.74 (qdd, J = 7.6, 7.1, 5.6 Hz, 1H, SiC*H*Me), 1.71-1.66 (m, 2H, CH_2), 1.54-1.46 (m, 2H, CH_2), 1.36-1.28 (m, 5H, CH_2), 1.03 (d, J = 7.6 Hz, 3H, SiCHCH_3), 0.90 (dd, J = 6.2, 6.2 Hz, 3H, CH_2CH_3).

^{13}C NMR ($CDCl_3$, 125 MHz): δ 135.7, 135.1, 134.6, 133.5, 130.2, 130.1, 128.1, 127.9, 77.8, 41.0, 38.0, 32.1, 26.0, 22.9, 17.0, 15.5, and 14.3.

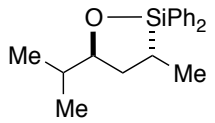
TLC: R_f = 0.25 in 20:1 hexanes: EtOAc.

GC-MS (5029017): t_R=14.065 min, m/z 253 [(M-penty)$^+$, 100], 199 (30), and 175 (50).

IR (neat): 3068 (w), 3049 (w), 2926 (m), 1456 (m), 1263 (m), 696 (s), and 446 (s) cm^{-1}.

HRMS (ESI/TOF): Calcd for $(C_{21}H_{28}NaOSi)^+$: 347.1802. Found: 347.1794.

***Trans*-5-Isopropyl-3-methyl-2,2-diphenyl-1,2-oxasilolane (*trans*-2h)**



*trans-**2h***

Regio- and Diastereomeric Ratios: 2:**3** = 84:16, *trans*-**2h**: *cis*-**2h** = 86:14.

NMR Yield: 70%.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.66-7.61 [m, 4H, Si{$C_6(H_{ortho})_2H_3$}$_2$], 7.44-7.38 [m, 6H, Si{$C_6(H_{ortho})_2H_3$}$_2$], 3.99 (ddd, *J* = 7.9, 7.3, 5.0 Hz, 1H, SiOC*H*), 2.01 (ddd, *J* = 13.2, 7.9, 7.4 Hz, 1H, $CH_aH_bCHMeSiPh_2$), 1.78 (ddd, *J* = 13.2, 5.0, 5.0 Hz, 1H, $CH_aH_bCHMeSiPh_2$), 1.72 (qqd, *J* = 7.3, 6.6, 6.6 Hz, 1H, C*H*Me_2), 1.74 (qdd, *J* = 7.6, 7.4, 5.0 Hz, 1H, SiC*H*Me), 1.04 [d, *J* = 6.6 Hz, 3H, $(CH_3)_2CH$], and 1.01 (d, *J* = 7.6 Hz, 3H, SiCHCH_3), and 0.91 [d, *J* = 6.6 Hz, 3H, $(CH_3)_2CH$].

^{13}C NMR ($CDCl_3$, 125 MHz): δ 135.7, 135.1, 134.6, 133.7, 130.14, 130.06, 128.1, 127.9, 82.9, 38.2, 34.6, 19.5, 18.7, 17.1, and 15.8.

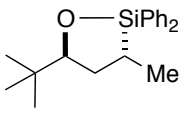
TLC: R_f = 0.25 in 20:1 hexanes: EtOAc.

GC-MS (5029017): t_R=13.062 min, m/z 253 [(M-^{i}Pr)$^+$, 100], 199 (45), and 175 (70).

IR (neat): 3068 (w), 2925 (m), 1453 (m), 1117 (m), 697 (s), and 499 (s) cm^{-1}.

HRMS (ESI/TOF): Calcd for $(C_{19}H_{24}NaOSi)^+$: 319.1489. Found: 319.1473.

***Trans*-5-*tert*-Butyl-3-methyl-2,2-diphenyl-1,2-oxasilolane (*trans*-2i)**



*trans-**2i***

Regio- and Diastereomeric Ratios: 2:**3** = 75:25, *trans*-**2i** : *cis*-**2i** = 88:12.

NMR Yield: 62%.

This NMR includes *cis*-**2e** (ca. 6.5%).

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.64 [dd, *J* = 7.8, 1.5 Hz, 2H, Si{$C_6(H_{ortho})_2H_3$}$_2$], 7.61 [dd, *J* = 7.8, 1.5 Hz, 2H, Si{$C_6(H_{ortho})_2H_3$}$_2$], 7.45-7.37 [m, 6H, Si{$C_6(H_{ortho})_2(H_{meta})_2H_{para}$}$_2$], 3.98 (dd, *J* = 9.9, 4.8 Hz, 1H, SiOC*H*), 2.07 (ddd, *J* = 13.1, 9.9, 7.7 Hz, 1H, $CH_aH_bCHMeSiPh_2$), 1.75 (qdd, *J* = 7.7, 7.7, 3.1Hz, 1H, SiC*H*Me), 1.71 (ddd, *J* = 13.1, 4.8, 3.1 Hz, 1H, $CH_aH_bCHMeSiPh_2$), 1.01 (d, *J* = 7.7 Hz, 3H, SiCHCH_3), and 0.96 [s, 9H, $C(CH_3)_3$].

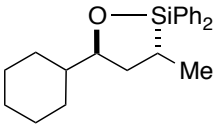
^{13}C NMR ($CDCl_3$, 125 MHz): δ 135.6, 135.1, 134.6, 133.9, 130.1, 130.0, 128.0, 127.9, 85.1, 36.0, 35.0, 26.2, 17.3, and 16.1.

GC-MS (5029017): t_R=13.203 min, m/z 295 [(M-Me)$^+$, 5], 253 [(M-^{t}Bu)$^+$, 100], 199 (70), and 175 (60).

IR (neat): 3069 (w), 2952 (m), 1456 (m), 1261 (m), 697 (s), and 500 (s) cm^{-1}.

HRMS (ESI/TOF): Calcd for $(C_{20}H_{26}NaOSi)^+$: 333.1645. Found: 333.1620.

***Trans*-5-Cyclohexyl-3-methyl-2,2-diphenyl-1,2-oxasilolane (*trans*-2j)**



*trans-**2j***

Regio- and Diastereomeric Ratios: 2:**3** = 81:19, *trans*-**2j**: *cis*-**2j** = 87:13.

NMR Yield: 65%.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.62 [dd, *J* =7.8, 1.5 Hz, 2H, $Si\{C_6(H_{ortho})_2H_3\}_2$], 7.58 [dd, *J* =7.8, 1.5 Hz, 2H, $Si\{C_6(H_{ortho})_2H_3\}_2$], 7.44-7.35 [m, 6H, $Si\{C_6(H_{ortho})_2(H_{meta})_2H_{para}\}_2$], 4.01 (ddd, *J* = 7.7, 7.3, 4.9 Hz, 1H, SiOC*H*), 2.06 (ddddd, *J* = 10.3, 7.7, 3.4, 3.4, 2.4 Hz, 1H, *Cy*), 2.03 (ddd, *J* = 13.1, 7.7, 7.3 Hz, 1H, $CH_aH_bCHMeSiPh_2$), 1.77 (ddd, *J* = 13.1, 4.9, 3.5 Hz, 1H, $CH_aH_bCHMeSiPh_2$), 1.79-1.66 (m, 5H, *Cy*), 1.43 (qdd, *J* = 7.7, 7.7, 3.5 Hz, 1H, SiC*H*Me), 1.27-1.11(m, 3H, *Cy*), 1.08-0.97 (m, 2H, *Cy*), and 1.00 (d, *J* = 7.7 Hz, 3H, $SiCHCH_3$).

^{13}C NMR ($CDCl_3$, 125 MHz): δ 135.7, 135.1, 134.6, 133.7, 130.1, 130.0, 128.1, 127.9, 82.0, 44.4, 38.3, 30.0, 29.1, 26.8, 26.4, 26.3, 17.0, and 15.8.

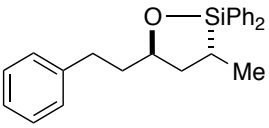
TLC: R_f = 0.25 in 20:1 hexanes: EtOAc.

GC-MS (5029017): t_R=15.177 min, m/z 253 [$(M-Cy)^+$, 100], 199 (30), and 175 (50).

IR (neat): 3068 (w), 2922 (m), 1428 (m), 1118 (m), 699 (s), and 513 (s) cm^{-1}.

HRMS (ESI/TOF): Calcd for $(C_{22}H_{28}NaOSi)^+$: 359.1802. Found: 359.1796.

***Trans*-3-Methyl-5-phenethyl-2,2-diphenyl-1,2-oxasilolane (*trans*-2k)**



*trans-**2k***

Regio- and Diastereomeric Ratios: 2:**3** = 76:24, *trans*-**2k**: *cis*-**2k** = 90:10.

NMR Yield: 56%.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.68 [dd, *J* =7.8, 1.5 Hz, 2H, $Si\{C_6(H_{ortho})_2H_3\}_2$], 7.62 [dd, *J* =7.8, 1.5 Hz, 2H, $Si\{C_6(H_{ortho})_2H_3\}_2$], 7.47-7.38 [m, 6H, $Si\{C_6(H_{ortho})_2(H_{meta})_2H_{para}\}_2$], 7.32-7.28 (m, 2H, *Ph*CH_2], 7.22-7.18 (m, 3H, *Ph*CH_2), 4.37 (dddd, *J* = 8.1, 6.7, 5.1, 5.1 Hz, 1H, SiOC*H*CH_2), 2.88 (ddd, *J* = 13.7, 10.2, 5.3 Hz, 1H, $PhCH_aH_b$), 2.76 (ddd, *J* = 13.7, 10.2, 6.5 Hz, 1H, $PhCH_aH_b$),

2.01 (ddd, J = 13.1, 7.5, 6.7 Hz, 1H, CH_aH$_b$CHMe), 1.97 (dddd, J = 13.3, 10.2, 8.1, 5.3 Hz, 1H, PhCH$_2$CH_aH$_b$), 1.89 (ddd, J = 13.1, 6.5, 5.1 Hz, 1H, CH$_a$$H_b$CHMe), 1.82 (dddd, J = 13.3, 10.2, 6.5, 5.1 Hz, 1H, PhCH$_2$CH$_a$$H_b$), 1.76 (qdd, J = 7.5, 7.5, 6.5 Hz, 1H, SiC*H*Me), and 1.00 (d, J = 7.5 Hz, 3H, SiCHCH_3).

^{13}C NMR ($CDCl_3$, 125 MHz): δ 142.6, 135.6, 135.1, 134.6, 133.4, 130.26, 130.16, 128.7, 128.5, 128.2, 128.0, 125.9, 76.9, 41.0, 39.8, 32.7, 17.0, and 15.5.

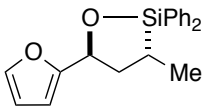
TLC: R_f = 0.25 in 20:1 hexanes: EtOAc.

GC-MS (5032021): t_R=15.962 min, m/z 357 [(M-H)$^+$, 1], 280 (8), 254 [(M-PhCH$_2$CH$_2$)$^+$, 20], and 199 (100).

IR (neat): 3068 (w), 2923 (m), 1494 (m), 1118 (m), 695 (s), and 510 (s) cm^{-1}.

HRMS (ESI/TOF): Calcd for ($C_{24}H_{26}NaOSi$)$^+$: 381.1645. Found: 381.1640.

***Trans*-5-(Furan-2-yl)-3-methyl-2,2-diphenyl-1,2-oxasilolane (*trans*-2l)**



*trans-**2l***

Regio- and Diastereomeric Ratios: 2:**3** = 82:18, *trans*-**2l**: *cis*-**2l** = 80:20.

NMR Yield: 59%.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.65 [dd, J = 8.0, 1.4 Hz, 2H, Si{C$_6$(H$_{ortho}$)$_2$H$_3$}$_2$], 7.61 [dd, J = 8.0, 1.4 Hz, 2H, Si{C$_6$(H$_{ortho}$)$_2$H$_3$}$_2$], 7.46-7.35 [m, 7H, furanyl C5-*H*, Si{C$_6$(H$_{ortho}$)$_2$$H_3$}$_2$], 6.30 (dd, J = 3.2, 1.8 Hz, 1H, furanyl C4-*H*), 6.22 (ddd, J = 3.2, 0.7, 0.7 Hz, 1H, furanyl C3-*H*), 5.40 [dd, J =5.9, 5.9 Hz, 1H, SiOC*H*], 2.50 (ddd, J = 13.2, 7.2, 5.9 Hz, 1H, CH_aH$_b$CHMe), 2.06 (ddd, J = 13.2, 7.6, 5.9 Hz, 1H, CH$_a$$H_b$CHMe), 1.89 (dqd, J = 7.6, 7.5, 7.2 Hz 1H, Me$_2$C*H*), 1.08 (d, J = 7.5 Hz, 3H, SiCHCH_3).

^{13}C NMR ($CDCl_3$, 125 MHz): δ 156.7, 142.1, 135.1, 134.9, 134.5, 132.9, 130.4, 130.3, 128.1, 128.0, 110.3, 106.4, 72.5, 39.6, 16.9, and 14.9.

TLC: R_f = 0.25 in 20:1 hexanes: EtOAc.

GC-MS (5029017): t_R=14.341 min, m/z 320 [M$^+$, 2], 278 (100), 260 (50), and 200 (35).

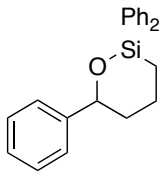
IR (neat): 3069 (w), 2925 (m), 1462 (m), 1116 (s), 700 (s), and 508 (m) cm^{-1}.

HRMS (ESI/TOF): Calcd for ($C_{20}H_{20}NaO_2Si$)$^+$: 343.1125. Found: 343.1117.

General Procedure for Preparation of Oxasilacyclohexane (3):

$[Rh(nbd)Cl]_2$ (2.3 mg, 5.0 μmol, 2.5 mol %) and dpph (9.1 mg, 0.020 mmol, 10 mol %) were added to a flame-dried, septum capped vial. The mixture was dissolved with THF (0.50 mL), which was stirred for 1 h at rt. Volatiles including norbornadiene (nbd) were removed *in vacuo*, and the resulting mixture was kept under vaccum for 0.5 h (the absence of nbd was confirmed by GC/MS analysis). Norbornene (3.7 mg, 0.04 mmol, 20 mol %) and THF (0.40 mL, 0.50 M) were added to the prepared catalyst. Homoallyl sily ether (0.20 mmol) dissolved in THF (0.40 mL, 0.50 M) was added dropwise to the reaction mixture via a cannular. The septum on the vial was replaced by a screw cap with a Teflon liner, and the mixture was stirred at rt for 24 h and the volatiles were removed *in vacuo*. The reaction progress was monitored by GC-MS spectrometry. The yield of the reaction was determined by the NMR spectroscopy by an addition of DMF (16 μL, 0.2 mmol) as an internal standard. The regio- and diastereomeric ratios were determined by the NMR spectroscopy. For an analytical purpose, the product was purifed by MPLC (hexanes/EtOAc =80:1, 5 mL/min, retention time 10-20 min). Partial decomposition of the product was observed during this MPLC separation.

2,2,6-Triphenyl-1,2-oxasilinane (3a)



3a

Regioisomeric Ratio: 2:**3** = 4:96.

NMR Yield: 89%.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.72 [dd, J = 8.0, 1.4 Hz, 2H, $Si\{C_6(H_{ortho})_2H_3\}_2$], 7.63 [dd, J = 8.0, 1.4 Hz, 2H, $Si\{C_6(H_{ortho})_2H_3\}_2$], 7.46-7.34 [m, 10H, *Ph*CH, $Si\{C_6(H_{ortho})_2H_3\}_2$], 7.27-7.24 (m, 1H, *Ph*CH), 5.01 (dd, J = 10.9, 2 Hz, 1H, *H6*), 2.25 (ddddd, J = 13.4, 5.8, 3.9, 3.9, 3.9 Hz, 1H, $H4_{equitorial}$), 1.97 (ddddd, J = 13.4, 3.9, 3.9, 2, 1.5 Hz, 1H, $H5_{equitorial}$), 1.89 (ddddd, J = 13.4, 13.4, 13.4, 3.9, 3.9 Hz, 1H, $H4_{axial}$), 1.68 (dddd, J = 13.4, 13.4, 10.9, 3.9 Hz, 1H, $H5_{axial}$), 1.38 (dddd, J = 14.8, 3.9, 3.9, 1.5 Hz, 1H, $H3_{equitorial}$), and 1.12 (ddd, J = 14.8, 13.6, 5.8 Hz, 1H, $H3_{axial}$).

^{13}C NMR ($CDCl_3$, 125 MHz): δ 145.3, 136.1, 134.8, 134.4, 134.2, 130.0, 129.9, 128.2, 128.1, 127.8, 127.0, 125.3, 77.1, 39.0, 22.3, and 10.4.

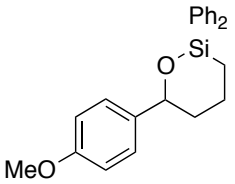
TLC: R_f = 0.25 in 20:1 hexanes: EtOAc.

GC-MS (5029017): t_R=15.599 min, m/z 329 [$(M-H)^+$, 40], 287 (20), 252 [$(M-Ph)^+$, 100], and 181 (70).

IR (neat): 3067 (w), 2925 (m), 1428 (m), 1187 (m), 695 (s), and 503 (s) cm^{-1}.

HRMS (ESI/TOF): Calcd for $(C_{22}H_{22}NaOSi)^+$: 353.1332. Found: 353.1316.

6-(4-Methoxyphenyl)-2,2-diphenyl-1,2-oxasilinane (3b)



3b

Regioisomeric Ratio: 2:**3** = 6:94.

NMR Yield: 85%.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.71 [dd, *J* = 8.0, 1.4 Hz, 2H, $Si\{C_6(H_{ortho})_2H_3\}_2$], 7.63 [dd, *J* = 8.0, 1.4 Hz, 2H, $Si\{C_6(H_{ortho})_2H_3\}_2$], 7.47-7.34 [m, 8H, *p*-MeO*Ph*, $Si\{C_6(H_{ortho})_2H_3\}_2$], 6.88 [d, *J* = 8.7 Hz, 2H, *p*-MeO*Ph*], 4.95 (dd, *J* = 10.8, 1.4 Hz, 1H, *H6*), 3.80 (s, 3H, OCH_3), 2.23 (ddddd, *J* = 13.6, 5.8, 3.9, 3.9, 2.5 Hz, 1H, $H4_{equitorial}$), 1.92 (ddddd, *J* = 13.6, 3.9, 2.5, 1.4, 1.4 Hz, 1H, $H5_{equitorial}$), 1.87 (ddddd, *J* = 13.6, 13.6, 13.6, 3.4, 2.5 Hz, 1H, $H4_{axial}$), 1.67 (dddd, *J* = 13.6, 13.6, 10.8, 2.5 Hz, 1H, $H5_{axial}$),1.36 (dddd, *J* = 14.8, 3.9, 3.4, 1.4 Hz, 1H, $H3_{equitorial}$), and 1.11 (ddd, *J* = 14.8, 13.6, 5.8 Hz, 1H, $H3_{axial}$).

^{13}C NMR ($CDCl_3$, 125 MHz): δ 158.6, 137.7, 136.1, 134.9, 134.4, 134.2, 129.94, 129.88, 128.0, 127.8, 126.5, 113.6, 77.5 (overlap with $CDCl_3$), 55.3, 39.0, 22.3, and 10.4.

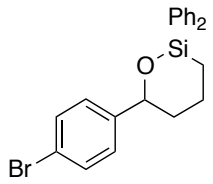
TLC: R_f = 0.20 in 20:1 hexanes:EtOAc.

GC-MS (5032021): t_R=16.672 min, m/z 359 [$(M-H)^+$, 30], 331 [$(M-MeO)^+$, 10], 282 (100), and 181 (40).

IR (neat): 3067 (w), 3047 (w), 1511 (m), 1243 (m), 1069 (s), 697 (s), and 510 (s) cm^{-1}.

HRMS (ESI/TOF): Calcd for $(C_{23}H_{24}NaO_2Si)^+$: 383.1438. Found: 383.1415.

6-(4-Bromophenyl)-2,2-diphenyl-1,2-oxasilinane (3c)



3c

Regioisomeric Ratio: 2:**3** = 7:93.

NMR Yield: 88%.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.73 [dd, *J* = 8.0, 1.4 Hz, 2H, Si{$C_6(H_{ortho})_2H_3$}$_2$], 7. 65 [dd, *J* = 8.0, 1.4 Hz, 2H, Si{$C_6(H_{ortho})_2H_3$}$_2$], 7.48-7.37 [m, 8H, *p*-Br*Ph*, Si{$C_6(H_{ortho})_2H_3$}$_2$], 7.33 (d, *J* = 8.4 Hz, 2H, *p*-Br*Ph*), 4.97 (dd, *J* = 11.0, 2.0 Hz, 1H, *H6*), 2.27 (ddddd, *J* = 13.6, 5.8, 4.1, 3.9, 2.8 Hz, 1H, $H4_{equitorial}$), 1.95 (ddddd, *J* = 13.6, 4.1, 2.5, 2.0, 1.5 Hz, 1H, $H5_{equitorial}$), 1.91 (ddddd, *J* = 13.6, 13.6, 13.6, 3.6, 2.5 Hz, 1H, $H4_{axial}$), 1.66 (dddd, *J* = 13.6, 13.6, 11.0, 2.8 Hz, 1H, $H5_{axial}$), 1.40 (dddd, *J* = 14.9, 3.9, 3.6, 1.5 Hz, 1H, $H3_{equitorial}$), and 1.14 (ddd, *J* = 14.9, 13.6, 5.8 Hz, 1H, $H3_{axial}$).

^{13}C NMR ($CDCl_3$, 125 MHz): δ 144.5, 136.0, 134.8, 134.6, 134.4, 131.4, 130.3, 130.2, 128.3, 128.0, 127.3, 120.8, 76.6, 39.1, 22.4, and 10.5.

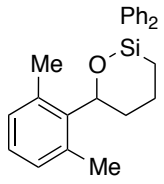
TLC: R_f = 0.25 in 20:1 hexanes: EtOAc.

GC-MS (5032021): t_R=17.114 min, m/z 332 [$(M\text{-}Ph)^+$, 100], 330 [$(M\text{-}Ph)^+$, isotpote, 100], 287 (60), 251 (65), and 181 (70).

IR (neat): 3068 (w), 2925 (m), 1511 (m), 1245 (m), 943 (m), 697 (s), and 512 (s) cm^{-1}.

HRMS (ESI/TOF): Calcd for $(C_{22}H_{21}BrNaOSi)^+$: 431.0437. Found: 431.0418.

6-(2,6-Dimethylphenyl)-2,2-diphenyl-1,2-oxasilinane (3d)



3d

Regioisomeric Ratio: 2:**3** = 7:93.

NMR Yield: 87%.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.73 [dd, *J* = 8.0, 1.4 Hz, 2H, Si{$C_6(H_{ortho})_2H_3$}$_2$], 7.65 [dd, *J* = 8.0, 1.4 Hz, 2H, Si{$C_6(H_{ortho})_2H_3$}$_2$], 7.49-7.32 [m, 6H, Si{$C_6(H_{ortho})_2H_3$}$_2$], 7.05 (dd, *J* = 7.1,

7.1Hz, 1H, diMe*Ph*), 6.99 (d, *J* = 7.1 Hz, 2H, diMe*Ph*), 5.44 (dd, *J* = 11.6, 5.0 Hz, 1H, *H6*), 2.45 [s, 6H, $(CH_3)_2C_6H_3$], 2.28 (ddddd, *J* = 13.6, 5.5, 5.2, 2.8, 2.8 Hz, 1H, $H4_{equitorial}$), 2.15 (dddd, *J* =13.6, 5.0, 2.8, 2.8, 1.5 Hz, 1H, $H5_{equitorial}$), 1.84 (ddddd, *J* = 13.6, 13.6, 13.6, 3.7, 2.8 Hz, 1H, $H4_{axial}$), 1.70 (dddd, *J* = 13.6, 13.6, 11.6, 2.8 Hz, 1H, $H5_{axial}$), 1.37 (dddd, *J* = 14.8, 5.2, 3.7, 1.5 Hz, 1H, $H3_{equitorial}$), and 1.20 (ddd, *J* = 14.8, 13.6, 5.5 Hz, 1H, $H3_{axial}$).

^{13}C NMR ($CDCl_3$, 125 MHz): δ 139.9, 135.9, 134.8, 134.6, 134.48, 134.46, 130.2, 130.1, 128.3, 128.01, 127.97, 127.1, 75.2, 33.8, 23.1, 21.2 (2), and 10.5.

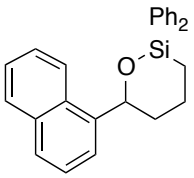
TLC: R_f = 0.25 in 20:1 hexanes: EtOAc.

GC-MS (5032021): t_R=16.139 min, m/z 358 (M^+, 10), 301 (100), 280 [$(M\text{-}Ph)^+$, 95], 265 (55), and 199 (90).

IR (neat): 3068 (w), 2923 (m), 1428 (m), 1193 (m), 695 (s), and 499 (s) cm^{-1}.

HRMS (ESI/TOF): Calcd for $(C_{24}H_{26}NaOSi)^+$: 381.1645. Found: 381.1637.

6-(Naphthalen-1-yl)-2,2-diphenyl-1,2-oxasilinane (3e)



3e

Regioisomeric Ratio: 2:**3** = 3:97.

NMR Yield: 87%.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.95-7.92 (nfom, 1H, *1-naph*), 7.89 (d, *J* =7.2 Hz, 1H, *1-naph*), 7.88-7.86 (nfom, 1H, *1-naph*), 7.78-7.75 [m, 3H, *1-naph*, $Si\{C_6(H_{ortho})_2H_3\}_2$], 7.68 [dd, *J* = 7.5, 1.4 Hz, 2H, $Si\{C_6(H_{ortho})_2H_3\}_2$], 7.52(dd, *J* = 7.6, 7.6 Hz, 1H, *1-naph*), 7.48-7.38 [m, 8H, *1-naph*, $Si\{C_6(H_{ortho})_2H_3\}_2$], 5.74 (d, *J* = 10.8, 2.0 Hz, 1H, *H6*), 2.30 (ddddd, *J* = 13.6, 5.6, 4.2, 4.2, 2.5 Hz, 1H, $H4_{equitorial}$), 2.22 (ddddd, *J* =13.6, 4.2, 2.5, 2.0, 1.5 Hz, 1H, $H5_{equitorial}$), 2.03 (ddddd, *J* = 13.6, 13.6, 13.6, 3.6, 2.5 Hz, 1H, $H4_{axial}$), 1.77 (dddd, *J* = 13.6, 13.6, 10.8, 2.5 Hz, 1H, $H5_{axial}$), 1.46 (dddd, *J* = 14.8, 4.2, 3.6, 1.5 Hz, 1H, $H3_{equitorial}$), and 1.21 (ddd, *J* = 14.8, 13.6, 5.6 Hz, 1H, $H3_{axial}$).

^{13}C NMR ($CDCl_3$, 125 MHz): δ 141.0, 136.3, 134.9, 134.7, 134.5, 133.8, 130.25, 130.22, 130.0, 129.1, 128.4, 128.1, 127.6, 126.0, 125.8, 125.4, 123.2, 123.0, 74.5, 38.5, 22.8, and 10.8.

TLC: R_f = 0.25 in 20:1 hexanes: EtOAc.

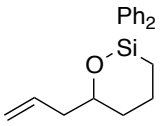
GC-MS (5032021): t_R=18.682 min, m/z 380 (M^+, 45), 338 (90), 302 (100), 260 (65), 215 (50),

and 181 (30).

IR (neat): 3067 (w), 2923 (m), 1454 (m), 1186 (m), 698 (s), and 503 (s) cm^{-1}.

HRMS (ESI/TOF): Calcd for $(C_{26}H_{24}NaOSi)^+$: 403.1489. Found: 403.1479.

6-Allyl-2,2-diphenyl-1,2-oxasilinane (3f)



3f

Regioisomeric Ratio: 2:**3** = 4:96.

NMR Yield: 83%.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.66 [dd, J = 8.0, 1.4 Hz, 2H, $Si\{C_6(H_{ortho})_2H_3\}_2$], 7.55 [dd, J = 8.0, 1.4 Hz, 2H, $Si\{C_6(H_{ortho})_2H_3\}_2$], 7.45-7.31 [m, 6H, $Si\{C_6(H_{ortho})_2H_3\}_2$], 5.93 (dddd, J = 17.2, 10.2, 7.0, 7.0 Hz, 1H, CH_2=C$H$$CH_2$), 5.08 (dddd, J = 17.2, 2.2, 1.3, 1.3 Hz, 1H, C$H_{trans}$$H_{cis}$=$CHCH_2$), 5.06 (dddd, J = 10.2, 2.2, 1.3, 1.3 Hz, 1H, $CH_{trans}$$H_{cis}$=$CHCH_2$), 4.40 (dddd, J = 10.4, 6.1, 6.1, 2.0 Hz, 1H, H_6), 2.40 (ddddd, J = 13.6, 7.0, 6.1, 1.3, 1.3 Hz, 1H, CH_2=CHC$H_a$$H_b$), 2.28 (ddddd, J = 13.6, 7.0, 6.1, 1.3, 1.3 Hz, 1H, CH_2=C$H$$CH_a$$H_b$), 2.15 (ddddd, J = 13.6, 5.8, 4.9, 4.0, 2.8 Hz, 1H, $H4_{equitorial}$), 1.74 (ddddd, J = 13.6, 4.0, 2.6, 2.0, 1.7 Hz, 1H, $H5_{equitorial}$), 1.69 (ddddd, J = 13.6, 13.6, 13.6, 4.0, 2.6 Hz, 1H, $H4_{axial}$), 1.45 (dddd, J = 13.6, 13.6, 10.4, 2.8 Hz, 1H, $H5_{axial}$), 1.27 (dddd, J = 14.8, 4.9, 4.0, 1.7 Hz, 1H, $H3_{equitorial}$), and 1.05 (ddd, J = 14.8, 13.6, 5.8 Hz, 1H, $H3_{axial}$).

^{13}C NMR ($CDCl_3$, 125 MHz): δ 136.5, 135.6, 135.5, 134.6, 134.4, 130.09, 129.98, 128.2, 128.0, 116.7, 75.1, 43.4, 35.2, 21.8, and 10.6.

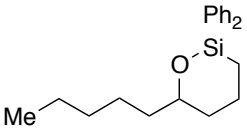
TLC: R_f = 0.25 in 20:1 hexanes: EtOAc.

GC-MS (5029017): t_R=13.421 min, m/z 253 [$(M\text{-allyl})^+$, 100], 225 (33), 199 (20), and 175 (30).

IR (neat): 3068 (w), 2926 (m), 1641 (w), 1486(m), 1117 (s), 696 (s), and 475 (s) cm^{-1}.

HRMS (ESI/TOF): Calcd for $(C_{19}H_{22}NaOSi)^+$: 317.1332. Found: 317.1338.

6-Pentyl-2,2-diphenyl-1,2-oxasilinane (3g)



3g

Regioisomeric Ratio: 2:**3** = 3:97.

NMR Yield: 85%.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.69 [dd, J = 8.0, 1.4 Hz, 2H, Si{C_6(H_{ortho})$_2$$H_3$}$_2$], 7.65 [dd, J = 8.0, 1.4 Hz, 2H, Si{C_6(H_{ortho})$_2$$H_3$}$_2$], 7.46-7.32 [m, 6H, Si{$C_6$($H_{ortho}$)$_2$$H_3$}$_2$], 3.91 (dddd, J = 10.3, 7.5, 4.5, 1.9 Hz, 1H, *H6*), 2.18-2.13 (nfom, Hz, 1H, $H4_{equitorial}$), 1.72-1.68 (m, 2H, $H5_{equitorial}$ and CH_2), 1.67-1.59 (m, 1H, $H4_{axial}$), 1.57-1.39 (m, 4H, $H5_{axial}$, CH_2), 1.38-1.26 (m, 5H, CH_2, $H3_{equitorial}$), 1.07 (ddd, J= 14.8, 13.2, 5.7 Hz, 1H, $H3_{axial}$), and 0.93 (dd, J = 7.0, 7.0 Hz, 3H, CH_3).

^{13}C NMR ($CDCl_3$, 125 MHz): δ 136.7, 135.7, 134.5, 134.4, 130.0, 129.9, 128.2, 128.0, 75.6, 38.9, 35.9, 32.1, 25.4, 22.9, 22.0, 14.3, and 10.7.

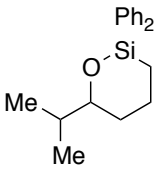
TLC: R_f = 0.25 in 20:1 hexanes: EtOAc.

GC-MS (5029017): t_R=14.222 min, m/z 253 [(M-penty)$^+$, 100], 225 (55), 199 (30), and 175 (50).

IR (neat): 3068 (w), 2926 (m), 1428 (m), 1187 (m), 696 (s), and 501 (m) cm^{-1}.

HRMS (ESI/TOF): Calcd for ($C_{21}H_{28}NaOSi$)$^+$: 347.1802. Found: 347.1806.

6-Isopropyl-2,2-diphenyl-1,2-oxasilinane (3h)



3h

Regioisomeric Ratio: 2:**3** = 6:94.

NMR Yield: 85%.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.67 [dd, J = 8.0, 1.4 Hz, 2H, Si{C_6(H_{ortho})$_2$$H_3$}$_2$], 7.55 [dd, J = 8.0, 1.4 Hz, 2H, Si{C_6(H_{ortho})$_2$$H_3$}$_2$], 7.44-7.32 [m, 6H, Si{$C_6$($H_{ortho}$)$_2$$H_3$}$_2$], 3.66 (ddd, J = 10.8, 5.3, 1.8 Hz, 1H, *H6*), 2.17 (ddddd, J = 13.6, 5.8, 4.4, 4.0, 2.8 Hz, 1H, $H4_{equitorial}$), 1.73 [qqd, J = 6.8, 6.8, 5.3 Hz, 1H, *CH*(CH_3)$_2$], 1.73 (ddddd, J = 13.6, 4.4, 2.5, 1.8, 1.5 Hz, 1H, $H5_{equitorial}$), 1.65 (ddddd, J = 13.6, 13.6, 13.6, 4.0, 2.5 Hz, 1H, $H4_{axial}$), 1.44 (dddd, J = 13.6, 13.6, 10.8, 2.8 Hz, 1H, $H5_{axial}$), 1.27 (dddd, J = 14.8, 4.0, 4.0, 1.5 Hz, 1H, $H3_{equitorial}$), 1.00 [d, J = 6.8 Hz, 3H, CH(CH_3)$_2$], 0.96 [d, J = 6.8 Hz, 3H, CH(CH_3)$_2$], and 0.89 (ddd, J = 14.8, 13.6, 5.8 Hz, 1H, $H3_{axial}$).

^{13}C NMR ($CDCl_3$, 125 MHz): δ 137.0, 135.8, 134.5, 134.4, 130.0, 129.9, 128.2, 128.0, 80.3, 35.1, 32.4, 22.2, 18.9, 18.0, and 10.9.

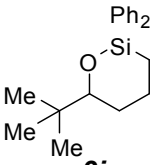
TLC: R_f = 0.25 in 20:1 hexanes: EtOAc.

GC-MS (5029017): t_R=13.278 min, m/z 253 [(M-^{i}Pr)$^+$, 100], 225 (55), 199 (25), and 175 (60).

IR (neat): 3068 (w), 2928 (m), 1468 (m), 1183 (m), 697 (s), and 539 (m) cm^{-1}.

HRMS (ESI/TOF): Calcd for $(C_{19}H_{24}NaOSi)^+$: 319.1489. Found: 319.1476.

6-*tert*-Butyl-2,2-diphenyl-1,2-oxasilinane (3i)



3i

Regioisomeric Ratio: 2:**3** = 10:90.

NMR Yield: 86%.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.68 [dd, J = 8.0, 1.4 Hz, 2H, Si{C_6(H_{ortho})$_2$H$_3$}$_2$], 7. [dd, J = 8.0, 1.4 Hz, 2H, Si{C_6(H_{ortho})$_2$H$_3$}$_2$], 7.45-7.33 [m, 6H, Si{C_6(H$_{ortho}$)$_2$$H_3$}$_2$], 3.53 (dd, J = 11.1, 1.8 Hz, 1H, *H6*), 2.20 (ddddd, J = 13.6, 5.8, 4.5, 3.6, 2.5 Hz, 1H, $H4_{equitorial}$), 1.76 (ddddd, J = 13.6, 4.5, 2.5, 1.8, 1.5 Hz, 1H, $H5_{equitorial}$), 1.63 (ddddd, J = 13.6, 13.6, 11.0, 3.6, 2.5 Hz, 1H, $H4_{axial}$), 1.39 (dddd, J = 13.6, 13.6, 11.1, 2.5 Hz, 1H, $H5_{axial}$), 1.28 (dddd, J = 14.7, 3.6, 3.6, 1.5 Hz, 1H, $H3_{equitorial}$), 0.97 [s, 9H, C(CH_3)$_3$], and 0.96 (dddd, J = 14.7, 11.0, 5.8 Hz, 1H, $H3_{axial}$).

^{13}C NMR ($CDCl_3$, 125 MHz): δ 137.2, 135.9, 134.5, 134.3, 129.90, 129.86, 128.2, 127.9, 83.1, 35.8, 29.7, 26.0, 22.5, and 11.0.

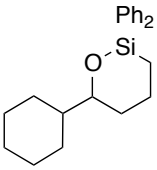
TLC: R_f = 0.25 in 20:1 hexanes: EtOAc.

GC-MS (5029017): t_R=13.434 min, m/z 295 [(M-Me)$^+$, 2], 253 [(M-^{t}Bu)$^+$, 100], 225 (60), and 175 (50).

IR (neat): 3068 (w), 2926 (m), 1478 (m), 1187 (m), 696 (s), and 529 (s) cm^{-1}.

HRMS (ESI/TOF): Calcd for $(C_{20}H_{26}NaOSi)^+$: 333.1645. Found: 333.1652.

6-Cyclohexyl-2,2-diphenyl-1,2-oxasilinane (3j)



3j

Regioisomeric Ratio: 2:**3** = 6:94.

NMR Yield: 86%.

This NMR includes unidentified impurities (ca. 5%).

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.68 [dd, *J* = 8.0, 1.4 Hz, 2H, Si{C_6(*H*$_{ortho}$)$_2$H$_3$}$_2$], 7.56 [dd, *J* = 8.0, 1.4 Hz, 2H, Si{C_6(*H*$_{ortho}$)$_2$H$_3$}$_2$], 7.45-7.33 [m, 6H, Si{C_6(H$_{ortho}$)$_2$*H$_3$*}$_2$], 3.66 (ddd, *J* = 10.7, 5.5, 1.4 Hz, 1H, *H6*), 2.17 (ddddd, *J* = 13.6, 4.1, 4.1, 3,6, 2.5 Hz, 1H, *H4*$_{equitorial}$), 1.99 (app d, *J* = 12.4 Hz, 1H, *Cy*), 1.79-1.60 (m, 6H, *H4*$_{axial}$, *H5*$_{equitorial,}$ and *Cy*), 1.46 (dddd, *J* = 13.6, 13.6, 10.7, 2.5 Hz, 1H, *H5*$_{axial}$), 1.47-1.41(m, 1H, *Cy*), 1.28-1.10 (m, 6H, *H3*$_{equitorial,}$ and *Cy*), and 1.01 (ddd, *J* = 14.6, 13.6, 5.6 Hz, 1H, *H3*$_{axial}$).

^{13}C NMR ($CDCl_3$, 125 MHz): δ 136.9, 135.8, 134.5, 134.4, 130.0, 129.9, 128.2, 127.9, 79.8, 45.0, 32.5, 29.2, 28.5, 26.9, 26.6, 26.5, 22.3, and 10.9.

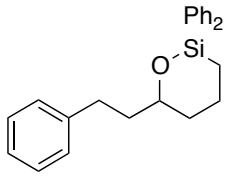
TLC: R_f = 0.25 in 20:1 hexanes: EtOAc.

GC-MS (5029017): t_R=15.470 min, m/z 253 [(M-Cy)$^+$, 100], 225 (40), and 175 (48).

IR (neat): 3067 (w), 2922 (m), 1486 (m), 1188 (m), 696 (s), and 521 (s) cm^{-1}.

HRMS (ESI/TOF): Calcd for ($C_{22}H_{28}NaOSi$)$^+$: 359.1802. Found: 359.1799.

6-Phenylethyl-2,2-diphenyl-1,2-oxasilinane (3k)



3k

Regioisomeric Ratio: 2:**3** = 10:90.

NMR Yield: 78%.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.65 [dd, *J* = 8.0, 1.4 Hz, 2H, Si{C_6(*H*$_{ortho}$)$_2$H$_3$}$_2$], 7.58 [dd, *J* = 8.0, 1.4 Hz, 2H, Si{C_6(*H*$_{ortho}$)$_2$H$_3$}$_2$], 7.45-7.33 [m, 6H, Si{C_6(H$_{ortho}$)$_2$*H$_3$*}$_2$], 7.27-7.23 (m, 3H, *Ph*CH$_2$), 7.20-7.15 (m, 2H, *Ph*CH$_2$), 3.92 (dddd, *J* = 10.5, 8.5, 3.7, 2.1 Hz, 1H, *H6*), 2.90 (ddd, *J* = 14.1, 9.8, 5.1 Hz, 1H, PhC*H$_a$*H$_b$), 2.74 (ddd, *J* = 14.1, 9.8, 6.9 Hz, 1H, PhCH$_a$*H$_b$*), 2.13 (ddddd, *J* = 13.6, 5.8, 4.4, 3.9, 3.3 Hz, 1H, *H4*$_{equitorial}$), 1.91 (dddd, *J* = 13.6, 9.8, 8.5, 5.1 Hz, 1H, PhCH$_2$C*Ha*Hb), 1.78 (dddd, *J* = 13.6, 9.8, 6.9, 3.7 Hz, 1H, PhCH$_2$CH$_a$*H$_b$*), 1.71 (ddddd, *J* = 13.6, 4.4, 2.5, 2.1, 1.5 Hz, 1H, *H5*$_{equitorial}$), 1.68 (ddddd, *J* = 13.6, 13.6, 13.6, 3.9, 2.5 Hz, 1H, *H4*$_{axial}$), 1.48 (dddd, *J* = 13.6, 13.6, 10.5, 3.3 Hz, 1H, *H5*$_{axial}$), 1.26 (dddd, *J* = 14.8, 3.9, 3.9, 1.5 Hz, 1H, *H3*$_{equitorial}$), and 1.10 (ddd, *J* = 14.8, 13.6, 5.8 Hz, 1H, *H3*$_{axial}$).

^{13}C NMR ($CDCl_3$, 125 MHz): δ 142.8, 136.7, 135.7, 134.6, 134.4, 130.1, 129.9, 128.8, 128.4, 128.2, 128.0, 125.8, 74.6, 40.5, 36.1, 31.9, 21.9, and 10.7.

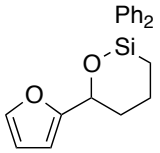
TLC: R_f = 0.25 in 20:1 hexanes: EtOAc.

GC-MS (5032021): t_R=16.154 min, m/z 358 [M^+, 1], 280 (30), 254 [$(M\text{-}PhCH_2CH_2)^+$, 90], and 199 (100).

IR (neat): 3067 (w), 2923 (m), 1495 (m), 1185 (m), 695 (s), and 537 (s) cm^{-1}.

HRMS (ESI/TOF): Calcd for $(C_{24}H_{26}NaOSi)^+$: 381.1645. Found: 381.1629.

6-(2-Furanyl)-2,2-diphenyl-1,2-oxasilinane (3l)



3l

Regioisomeric Ratio: 2:**3** = 3:97.

NMR Yield: 87%.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.73 [dd, *J* = 8.0, 1.4 Hz, 2H, $Si\{C_6(H_{ortho})_2H_3\}_2$], 7.56 [dd, *J* = 8.0, 1.4 Hz, 2H, $Si\{C_6(H_{ortho})_2H_3\}_2$], 7.48-7.31 [m, 7H, $Si\{C_6(H_{ortho})_2H_3\}_2$, one *H* of *furanyl*], 6.32 (dd, *J* = 3.1, 2.0 Hz, 1H, *furanyl*), 6.27 (dd, *J* = 3.1, 0.5 Hz, 1H, *furanyl*), 5.03 (dd, *J* = 10.4, 2.4 Hz, 1H, *H6*), 2.24 (ddddd, *J* = 12.9, 5.8, 4.7, 4.3, 2.4 Hz, 1H, $H4_{equitorial}$), 2.03 (ddddd, *J* =12.9, 4.7, 2.8, 2.4, 1.5 Hz, 1H, $H5_{equitorial}$), 1.92 (dddd, *J* = 12.9, 12.9, 10.5, 2.4 Hz, 1H, $H5_{axial}$), 1.82 (ddddd, *J* = 13.2, 12.9, 12.9, 3.7, 2.8 Hz, 1H, $H4_{axial}$), 1.34 (dddd, *J* = 14.8, 4.3, 3.7, 1.5 Hz, 1H, $H3_{equitorial}$), and 1.15 (ddd, *J* = 14.8, 13.2, 5.8 Hz, 1H, $H3_{axial}$).

^{13}C NMR ($CDCl_3$, 125 MHz): δ 157.4, 141.7, 135.8, 134.8, 134.65, 134.57, 130.24, 130.21, 128.35, 128.00, 110.2, 105.3, 71.1, 34.6, 21.8, and 10.6.

TLC: R_f = 0.25 in 20:1 hexanes: EtOAc.

GC-MS (5029017): t_R=14.590 min, m/z 320 [M^+, 35], 291 (20), 242 (100), and 199 (25).

IR (neat): 3068 (w), 2926 (m), 1486 (m), 1151 (m), 696 (s), and 505 (m) cm^{-1}.

HRMS (ESI/TOF): Calcd for $(C_{20}H_{20}NaO_2Si)^+$: 343.1125. Found: 343.1092.

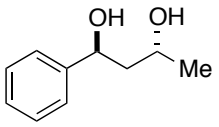
General Procedure for oxidation of 1,3-trans-oxasilacyclopentane and oxasilacyclohexane (*anti*-4 and 5):

CsOH (120 mg, 0.80 mmol), *tert*-butylhydroperoxide (0.60 mL, 5.0 M in decane, 3.0 mmol) and DMF (2.0 mL, 0.1 M) were added to a vial, and tetrabutylammonium fluoride (TBAF, 1.0 M in THF solution, 1.0 mmol) was added to the mixuture. Then either 1,3-*trans*-oxasilacyclopentane (0.20 mmol) or oxasilacyclohexane (0.20 mmol) was added to the mixture, which was stirred at rt for 48h. The reaction mixture was quenched with sodium thiosulfate and stirred for 10 min. The mixture was extract with etheyl acetate (20 mL×4), and the combined organic layer was washed with aqueous HCl (1.0 M, 20 mL), saturated aqueous sodium bicarbonate (20 mL), brine (20 mL), and dried over anhydrous sodium sulfate. Solvent was removed under reduced pressure and the crude mixture was purified by MPLC to afford *anti*-1,3-diols (hexanes/EtOAc =1:1.5, 5 mL/min, retention time 10-20 min) or 1,4-diols (hexanes/EtOAc =1:4, 7 mL/min, retention time 10-20 min).

General Procedure for bisacetylation of *anti*-1,3-diol and 1,4-diol (*anti*-4-diOAc and 5-diOAc):

Crude diol from oxidative desilylation reaction (0.20 mmol), 4-dimethylaminopyridine (5 mg), triethylamine (0.11 mL, 0.80 mmol) and CH_2Cl_2 (0.2 M, 1 mL) were added to the vial, and acetic anhydride (75 uL, 0.80 mmol) was added to the mixture, which was stirred at rt for 12 h. Volatiles including acetic anhydride were removed *in vacuo*, and the resulting crude mixture was purified by MPLC to afford 1,3-diacetate (hexanes/EtOAc = 10:1, 7 mL/min, retention time 10-15 min) or 1,4-diacetate (hexanes/EtOAc =5:1, 7 mL/min, retention time 10-15 min).

***Anti*-1-Phenylbutane-1,3-diol (*anti*-4a)**



*anti-**4a***

Yield: 23.9 mg, 72%.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.37-7.30 (nfom, 4H, *Ph*), 7.27-7.24 (m, 1H, *Ph*), 5.06 (dd, *J* = 7.4, 3.9 Hz, 1H, PhC*H*), 4.03 (dqd, *J* = 8.3, 6.4, 3.1 Hz, 1H, CH_3C*H*), 3.04 (br, 1H, O*H*), 2.42 (br, 1H, O*H*), 1.87 [ddd, *J* = 14.5, 7.4, 3.1 Hz, 1H, PhCH(OH)C$H_a$$H_b$], 1.87 [ddd, *J* = 14.5, 8.3, 3.9 Hz, 1H, PhCH(OH)$CH_a$$H_b$], and 1.24 (d, *J* = 6.4 Hz, 3H, CH_3).

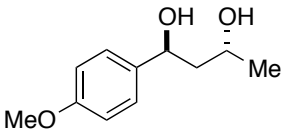
^{13}C NMR ($CDCl_3$, 125 MHz): δ 144.6, 128.6, 127.4, 125.8, 71.8, 65.5, 46.3, and 23.6.

TLC: R_f = 0.2 in 1:1 hexanes: EtOAc.

IR (neat): 3335 (br), 3063 (w), 2930 (m), 1452 (w), 1047 (m), 699 (s), and 550 (m) cm^{-1}.

HRMS (ESI/TOF): Calcd for $(C_{10}H_{14}O_2Na)^+$: 189.0886. Found: 189.0856.

1-(4-Methoxyphenyl)butane-*anti*-1,3-diol (*anti*-4b)



*anti-**4b***

Yield: 24 mg, 61%.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.26 (d, *J* = 8.6 Hz, 2H, *Ar*), 7.25 (d, *J* = 8.6 Hz, 2H, *Ar*), 4.97 (dd, *J* = 8.0, 3.5 Hz, 1H, ArC*H*), 4.04 (dqd, *J* = 8.3, 6.4, 3.1 Hz, 1H, CH_3C*H*), 3.79 (s, 3H, OCH_3), 3.01 (br, 2H, O*H*), 1.88 [ddd, *J* = 14.4, 8.0, 3.1 Hz, 1H, ArCH(OH)C$H_a$$H_b$], 1.79 [ddd, *J* = 14.4, 8.3, 3.5 Hz, 1H, ArCH(OH)C$H_a$$H_b$], and 1.22 (d, *J* = 6.4 Hz, 3H, CH_3).

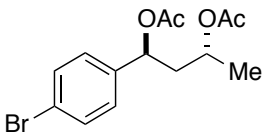
^{13}C NMR ($CDCl_3$, 125 MHz): δ 159.0, 136.8, 127.4, 114.0, 71.5, 65.5, 55.5, 46.3, and 23.6.

TLC: R_f = 0.2 in 1:1 hexanes: EtOAc.

IR (neat): 3354 (br), 3063 (w), 2932 (m), 1512 (m), 1244 (s), 1033 (m), and 558 (m) cm^{-1}.

HRMS (ESI/TOF): Calcd for $(C_{11}H_{16}O_3Na)^+$: 219.0992. Found: 219.0955.

1-(4-Bromophenyl)butane-*anti*-1,3-diyl diacetate (*anti*-4c-diOAc)



*anti-**4c**-diOAc*

Yield: 39.4 mg, 60%.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.47 (d, *J* = 8.5 Hz, 2H, *Ar*), 7.18 (dd, *J* = 8.5 Hz, 2H, *Ar*), 5.74 [dd, *J* = 7.1, 7.1 Hz, 1H, ArC*H*(OAc)], 4.78 [dqd, *J* = 7.9, 6.3, 5.1 Hz, 1H, CH_3C*H*(OAc)], 2.27 [ddd, *J* = 14.2, 7.9, 7.1 Hz, 1H, ArCH(OAc)C$H_a$$H_b$], 2.06 (s, 3H, Ac), 1.99 (s, 3H, Ac), 1.90 [ddd, *J* = 14.2, 7.1, 5.1 Hz, 1H, ArCH(OAc)C$H_a$$H_b$], and 1.23 [d, *J* = 6.3 Hz, 3H, CH_3CH(OAc)].

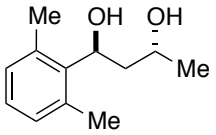
^{13}C NMR ($CDCl_3$, 125 MHz): δ 170.6, 170.2, 132.0, 128.5, 127.5, 122.4, 72.7, 67.9, 42.2, 21.44, 21.40, and 20.3.

TLC: R_f = 0.5 in 5:1 hexanes: EtOAc.

IR (neat): 3057 (w), 2927 (w), 1732 (s), 1593 (m), 1226 (s), 1009 (s), and 819 (m) cm^{-1}.

HRMS (ESI/TOF): Calcd for $(C_{14}H_{17}BrO_4Na)^+$: 351.0202. 351.0187.

1-(2,6-Dimethylphenyl)butane-*anti*-1,3-diol (*anti*-4d)



*anti-**4d***

Yield: 28.7 mg, 74%.

^{1}H NMR [$CDCl_3$, 500 MHz]: δ 7.05 (dd, *J* = 8.3, 6.6 Hz, 1H, Ar-*H*), 6.99 (app d, *J* = 7.5 Hz, 2H, Ar-*H*), 5.54 (dd, *J* = 10.7, 3.3 Hz, 1H, ArC*H*), 4.20-4.14 (dqd, *J* = 7.7, 6.3, 3.0 Hz, 1H, CH_3C*H*), 2.44 [s, 6H, $(CH_3)_2$Ar], 2.38-2.14 (br s, 2H, O*H*), 2.27 [ddd, *J* = 14.6, 10.7, 3.0 Hz, ArCH(OH)C$H_a$$H_b$], 1.58 [ddd, *J* = 14.6, 7.7, 3.3 Hz, ArCH(OH)C$H_a$$H_b$], 1.57-1.52 [m, 1H, ArCH(OH)C$H_a$$H_b$], and 1.32 (d, *J* = 6.3 Hz, CH_3).

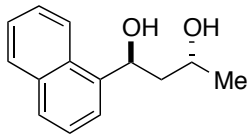
^{13}C NMR ($CDCl_3$, 125 MHz): δ 139.8, 136.0, 129.6, 127.2, 68.4, 65.8, 42.9, 23.4, and 20.9 (2).

TLC: R_f = 0.2 in 1:1 hexanes: EtOAc.

IR (neat): 3358 (br), 3021 (w), 2926 (m), 1453 (w), 1075 (s), 768 (s), and 520 (m) cm^{-1}.

HRMS (ESI/TOF): Calcd for $(C_{12}H_{18}O_2Na)^+$: 217.1199. Found: 217.1155.

1-(Naphthalen-1-yl)butane-*anti*-1,3-diol (*anti*-4e)



*anti-**4e***

Yield: 23.8 mg, 55%.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 8.08 (app d, *J* = 8.5 Hz, 1H, *1-naph*), 7.88 (app d, *J* = 7.8 Hz, 1H, *1-naph*), 7.79 (app d, *J* = 8.2 Hz, 1H, *1-naph*), 7.71 (ddd, *J* = 7.2, 1.1, 0.8 Hz, 1H, *1-naph*), 7.53 (dd, *J* = 6.8, 1.5 Hz, 1H, *1-naph*), 7.51-7.45 (m, 2H, *1-naph*), 5.75 (ddd, *J* = 8.8, 4.1 Hz, 1H, ArC*H*), 4.33 (dqd, *J* = 8.0, 6.2, 3.3 Hz, 1H, CH_3C*H*), 3.21 (br s, 1H, O*H*), 3.01 (br s, 1H, O*H*), 2.07-1.95 [m, 2H, ArCH(OH)C$H_a$$H_b$], and 1.27 (d, *J* = 6.2 Hz, 3H, CH_3).

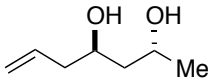
^{13}C NMR ($CDCl_3$, 125 MHz): δ 140.1, 134.0, 130.2, 129.2, 128.2, 126.3, 125.8, 125.7, 123.1, 123.0, 72.4, 69.4, 46.4, and 24.3.

TLC: R_f = 0.2 in 1:1 hexanes: EtOAc.

IR (neat): 3348 (br), 3051 (w), 2967 (m), 1164 (s), 702 (s), and 432 (m) cm^{-1}.

HRMS (ESI/TOF): Calcd for $(C_{14}H_{16}O_2Na)^+$: 239.1043. Found: 239.0999.

Hept-6-ene-*anti*-2,4-diol (*anti*-4f)



*anti-**4f***

Yield: 17.7 mg, 68%.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 5.82 (dddd, J = 17.4, 9.6, 7.5, 6.8 Hz, 1H, CH_2=C*H*CH_2), 5.14 (dddd, J = 17.4, 2.1, 1.2, 1.2 Hz, 1H, $CH_{cis}H_{trans}$=CH), 5.13 (dddd, J = 9.6, 2.1, 1.2, 1.2 Hz, 1H, C$H_{cis}H_{trans}$=CH), 4.16 (qdd, J = 6.3, 6.3, 4.7 Hz, 1H, CH_3C*H*), 4.00 (dddd, J = 7.5, 5.8, 5.4, 4.7 Hz, 1H, CH_2=CHCH_2C*H*), 2.45 (br, 2H, 2O*H*), 2.29 (ddddd, J = 13.6, 6.8, 5.4, 1.2, 1.2 Hz, 1H, CH_2=CHC$H_a$$H_b$), 2.25 (ddddd, J = 13.6, 7.5, 7.5, 1.2, 1.2 Hz, 1H, CH_2=CH$CH_a$$H_b$), 1.64 [ddd, J = 14.8, 6.3, 4.7 Hz, 1H, CH_3CH(OH)C$H_a$$H_b$], 1.61 [dddd, J = 14.8, 5.8, 4.7 Hz, 1H, CH_3CH(OH)$CH_a$$H_b$], and 1.24 (d, J = 6.3 Hz, 3H, CH_3).

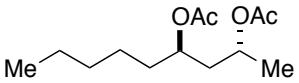
^{13}C NMR ($CDCl_3$, 125 MHz): δ 134.8, 118.5, 68.3, 65.6, 43.7, 42.2, and 23.7.

TLC: R_f = 0.2 in 1:1 hexanes: EtOAc.

IR (neat): 3376 (br), 2923 (s), 1729 (w), 1554 (m), 1097 (s), and 700 (s) cm^{-1}.

HRMS (ESI/TOF): Calcd for $(C_7H_{14}O_2Na)^+$: 153.0886. Found: 153.0893.

Nonane-*anti*-2,4-diyl diacetate (*anti*-4g-diOAc)



*anti-**4g**-diOAc*

Yield: 24.2 mg, 50%.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 4.98-4.88 [m, 2H, CH_3C*H*(OAc), CH_2C*H*(OAc)CH_2], 2.009 (s, 3H, Ac), 2.007 (s, 3H, Ac), 1.77 [ddd, J = 14.5, 9.9, 3.4 Hz, 1H, CH_2CH(OAc)C$H_a$$H_b$], 1.68 [ddd, J = 14.5, 9.8, 3.5 Hz, 1H, CH_2CH(OAc)$CH_a$$H_b$], 1.55-1.44 (m, 2H, CH_2), 1.32-1.23 (m, 6H, CH_2), 1.21 [d, J = 6.3 Hz, 3H, CH_3C*H*(OAc)], and 0.87 (dd, J = 7.1, 7.1 Hz, 3H, C$H_3$$CH_2$).

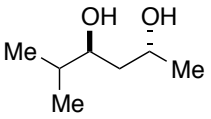
^{13}C NMR ($CDCl_3$, 125 MHz): δ 170.9 (2), 70.5, 67.2, 40.6, 34.9, 31.9, 25.0, 22.7, 21.4, 21.3, 20.7, and 14.2.

TLC: R_f = 0.5 in 5:1 hexanes: EtOAc.

IR (neat): 2932 (w), 1735 (s), 1459 (m), 1236 (s), 1020 (m), 925 (m), and 606 (m) cm^{-1}.

HRMS (ESI/TOF): Calcd for $(C_{13}H_{24}O_4Na)^+$: 267.1567. Found: 267.1569.

5-Methylhexane-*anti*-2,4-diol (*anti*-4h)



*anti-**4h***

Yield: 16.4 mg, 62%.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 4.16 [dqd, J = 7.9, 6.3, 3.1Hz, 1H, $CH_3CH(OH)$], 3.67 [ddd, J = 9.2, 6.3, 2.6 Hz, 1H, ^{i}PrC*H*(OH)], 2.32 (br, 2H, O*H*), 1.69 [qqd, J = 6.8, 6.8, 6.3 Hz, 1H, $(CH_3)_2CH$], 1.64 [ddd, J = 14.5, 9.2, 3.1 Hz, 1H, ^{i}PrCH(OH)CH_aH$_b$], 1.54 (ddd, J = 14.5, 7.9, 2.6 Hz, 1H, ^{i}PrCH(OH)CH$_a$$H_b$], 1.25 (d, J = 6.3 Hz, 3H, CH_3CH(OH)], 0.94 (d, J = 6.8 Hz, 3H, CH$(CH_3)_2$], and 0.90 [d, J = 6.8 Hz, 3H, CH$(CH_3)_2$].

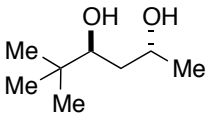
^{13}C NMR ($CDCl_3$, 125 MHz): δ 74.1, 65.9, 41.2, 34.0, 23.7, 18.8, and 18.1.

TLC: R_f = 0.2 in 1:1 hexanes: EtOAc.

IR (neat): 3376 (br), 2923 (s), 1459 (m), 1077 (s), 638 (m), and 514 (m) cm^{-1}.

HRMS (ESI/TOF): Calcd for $(C_7H_{16}O_2Na)^+$: 155.1043. Found: 155.0868.

5,5-Dimethylhexane-*anti*-2,4-diol (*anti*-4i)



*anti-**4i***

Yield: 13.2 mg, 45%.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 4.15 [qdd, J = 6.8, 6.5, 3.6 Hz, 1H, $CH_3CH(OH)$], 3.60 (dd, J = 10.0, 3.0 Hz, 1H, ^{t}BuC*H*(OH)], 2.21 (br, 2H, O*H*), 1.57 [ddd, J = 14.4, 10.0, 3.6 Hz, 1H, ^{t}BuCH(OH)CH_aH$_b$], 1.52 [ddd, J = 14.4, 6.5, 3.0 Hz, 1H, ^{t}BuCH(OH)CH$_a$$H_b$], 1.27 [d, J = 6.8 Hz, 3H, CH_3CH(OH)], and 0.90 [s, 9H, C(CH_3)$_3$].

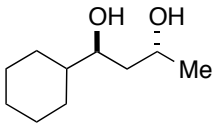
^{13}C NMR ($CDCl_3$, 125 MHz): δ 76.2, 66.1, 39.0, 34.8, 25.8, and 23.5.

TLC: R_f = 0.2 in 1:1 hexanes: EtOAc.

IR (neat): 3427 (br), 2918 (s), 1708 (s), 1363 (m), 1074 (s), 737 (m), and 469 (m) cm^{-1}.

HRMS (ESI/TOF): Calcd for $(C_8H_{18}O_2Na)^+$: 169.1199. Found: 169.1170.

4-Cyclohexyl-*anti*-2,4-diol (*anti*-4j)



*anti-**4j***

Yield: 18.8 mg, 55%.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 4.14 [dqd, *J* = 8.9, 6.3, 3.2 Hz, 1H, $CH_3CH(OH)$], 3.66 (dddd, *J* = 9.2, 6.4, 2.6, 1.1 Hz, 1H, CyC*H*(OH)], 2.61 (br, 2H, O*H*), 1.87 (ddddd, *J* = 14.4, 3.5, 3.5, 3.5, 1.7 Hz, 1H, *Cy*), 1.78-1.72 (m, 2H, *Cy*), 1.67-1.61(m, 3H, CyCH(OH)CH_aH_b, *Cy*), 1.54 [dddd, *J* = 14.6, 8.0, 2.8, 1.2 Hz, 1H, *Cy*], 1.39-1.32 (m, 1H, *Cy*), 1.23 [dd, *J* = 6.3, 1.0 Hz, 3H, CH_3CH(OH)], 1.27-1.08 (m, 3H, *Cy*), and 0.98 (ddddd, *J* = 11.8, 11.8, 11.8, 7.8, 3.4 Hz, 2H, *Cy*).

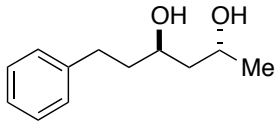
^{13}C NMR ($CDCl_3$, 125 MHz): δ 73.5, 65.8, 43.8, 41.2, 29.2, 28.6, 26.7, 26.4, 26.3, and 23.7.

TLC: R_f = 0.2 in 1:1 hexanes: EtOAc.

IR (neat): 3347 (br), 2923 (s), 1449 (s), 1065 (s), 737 (m), and 539 (m) cm^{-1}.

HRMS (ESI/TOF): Calcd for $(10_8H_{20}O_2Na)^+$: 195.1356. Found: 195.1303.

6-Phenyl-*anti*-2,4-diol (*anti*-4k)



*anti-**4k***

Yield: 19.4 mg, 50%.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.28 (dd, *J* = 7.7, 7.7 Hz, 2H, Ph-H_{meta}), 7.21-7.18 (m, 3H, *Ph*), 4.17 [qdd, *J* = 6.3, 6.3, 4.0 Hz, 1H, $CH_3CH(OH)$], 3.97 [dddd, *J* = 8.2, 6.7, 4.6, 4.2 Hz, 1H, $PhCH_2CH_2CH(OH)$], 2.96 (br, 2H, O*H*), 2.80 (ddd, *J* = 13.8, 10.0, 5.6 Hz, 1H, PhC*Ha*Hb), 2.67 (ddd, *J* = 13.8, 9.8, 6.4 Hz, 1H, PhCHaH_b), 1.86 (ddddd, *J* = 14.7, 9.8, 8.2, 5.6, 1.1 Hz, 1H, $PhCH_2CH_aH_b$), 1.77 (ddddd, *J* = 14.7, 10.0, 6.4, 4.6, 1.1 Hz, 1H, $PhCH_2CHaH_b$), 1.64 (ddd, *J* = 14.5, 6.7, 4.0 Hz, 1H, $CH_3CH(OH)CH_aH_b$], 1.61 [ddd, *J* = 14.5, 6.3, 4.2 Hz, 1H, $CH_3CH(OH)CH_aH_b$], and 1.23 [dd, *J* = 6.3, 1.1 Hz, 3H, CH_3CH(OH)].

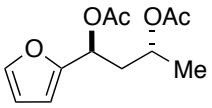
^{13}C NMR ($CDCl_3$, 125 MHz): δ 142.2, 128.60, 128.58, 126.0, 68.9, 65.6, 44.2, 39.2, 32.4, and 23.7.

TLC: R_f = 0.2 in 1:1 hexanes: EtOAc.

IR (neat): 3347 (br), 3061 (w), 2930 (m), 1453 (s), 1075 (m), 698 (s), and 480 (m) cm^{-1}.

HRMS (ESI/TOF): Calcd for $(C_{12}H_{18}O_2Na)^+$: 217.1199. Found: 217.1151.

1-(Furan-2-yl)-*buta*-1,3-diyl diacetate (*anti*-4l-*diO*Ac)



*anti-**4l**-diOAc*

Yield: 24.4 mg, 51%.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.38 (app s, 1H, *Ar*), 6.34-6.32 (m, 2H, *Ar*), 5.95 [dd, *J* = 9.8, 4.6 Hz, 1H, ArC*H*(OAc)], 5.01 [dqd, *J* = 9.8, 6.3, 3.6 Hz, 1H, CH_3C*H*(OAc)], 2.24 [ddd, *J* = 14.4, 9.8, 3.6 Hz, 1H, ArCH(OAc)C$H_a$$H_b$], 2.12 [ddd, *J* = 14.4, 9.8, 4.6 Hz, 1H, ArCH(OAc)$CH_a$$H_b$], 2.04 (s, 3H, Ac), 2.00 (s, 3H, Ac), and 1.26 [d, *J* = 6.3 Hz, 3H, CH_3CH(OAc)].

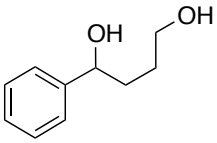
^{13}C NMR ($CDCl_3$, 125 MHz): δ 170.8, 170.4, 152.4, 142.8, 110.5, 108.7, 70.0, 65.0, 38.7, 21.4, 21.2, and 20.5.

TLC: R_f = 0.5 in 5:1 hexanes: EtOAc.

IR (neat): 3123 (w), 2934 (w), 1734 (s), 1224 (s), 1011 (m), 742 (m), and 600 (m) cm^{-1}.

HRMS (ESI/TOF): Calcd for $(C_{12}H_{16}O_5Na)^+$: 263.0890. Found: 263.0876.

1-Phenylbutane-1,4-diol (5a)



5a

Yield: 24.5 mg, 74%.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.37-7.30 (m, 4H, Ph), 7.27-7.23 (m, 1H, Ph), 4.71 [dd, *J* = 6.3, 6.3 Hz, 1H, PhC*H*(OH)], 3.68 [ddd, *J* = 10.6, 6.3, 5.5 Hz, 1H, CH_2C(OH)$H_a$$H_b$], 3.64 [ddd, *J* = 10.6, 7.0, 5.4 Hz, 1H, CH_2C(OH)$H_a$$H_b$], 2.73 (br, 2H, O*H*), 1.83 [app dd, *J* = 13.6, 7.0 Hz, 2H, PhC*H*(OH)C$H_a$$H_b$], 1.69 [ddddd, *J* = 14.2, 6.9, 6.9, 6.9, 5.5 Hz, 1H, PhCH(OH)CH_2C$H_a$$H_b$], and 1.63 [ddddd, *J* = 14.2, 7.4, 7.4, 6.3, 5.4 Hz, 1H, PhCH(OH)$CH_2$$CH_a$$H_b$].

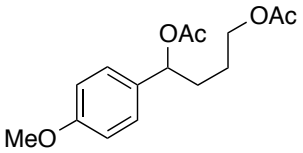
^{13}C NMR ($CDCl_3$, 125 MHz): δ 144.9, 128.6, 127.7, 126.0, 74.6, 63.0, 36.5, and 29.4

TLC: R_f = 0.2 in 1:4 hexanes: EtOAc.

IR (neat): 3326 (br), 3029 (w), 2931 (s), 1452 (s), 1026 (s), 698 (s), and 553 (m) cm^{-1}.

HRMS (ESI/TOF): Calcd for $(C_{10}H_{14}O_2Na)^+$: 189.0886. Found: 189.0854.

1-(4-Methoxyphenyl)butane-1,4-diyl diacetate (5b-diOAc)



***5b**-diOAc*

Yield: 38.4 mg, 69%.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.26 (d, *J* = 8.6 Hz, 2H, *Ar*), 6.87 (d, *J* = 8.6 Hz, 2H, *Ar*), 5.70 [dd, *J* = 7.7, 6.4 Hz, 1H, ArC*H*(OAc)], 4.05 [ddd, *J* = 11.0, 6.5, 6.5 Hz, 1H, $CH_2C(OAc)H_aH_b$], 3.63 [ddd, *J* = 11.0, 6.5, 6.5 Hz, 1H, $CH_2C(OAc)H_aH_b$], 3.79 (s, 3H, OCH_3), 2.05 (s, 3H, Ac), 2.03 (s, 3H, Ac), 1.97 [dddd, *J* = 13.6, 10.4, 7.7, 5.2 Hz, 1H, $ArCH(OAc)CH_aH_b$], 1.82 [dddd, *J* = 13.6, 10.4, 6.4, 5.5 Hz, 1H, $ArCH(OAc)CH_aH_b$], 1.66 [ddddd, *J* = 13.4, 10.4, 6.5, 6.5, 5.2 Hz, 1H, $ArCH(OAc)CH_2CH_aH_b$], and 1.55 [ddddd, *J* = 13.4, 10.4, 6.5, 6.5, 5.5 Hz, 1H, $ArCH(OAc)CH_2CH_aH_b$].

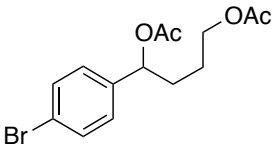
^{13}C NMR ($CDCl_3$, 125 MHz): δ 171.3, 170.6, 159.6, 132.5, 128.1, 114.1, 75.5, 64.2, 55.5, 32.8, 25.1, 21.5, and 21.2.

TLC: R_f = 0.5 in 5:1 hexanes: EtOAc.

IR (neat): 3057 (w), 2927 (w), 1733 (s), 1514 (m), 1229 (s), 1030 (s), 830 (s), 7301(m), and 555 (m) cm^{-1}.

HRMS (ESI/TOF): Calcd for $(C_{15}H_{20}O_5Na)^+$: 303.1203. Found: 303.1201.

1-(4-Bromophenyl)butane-1,4-diyl diacetate (5c-diOAc)



***5c**-diOAc*

Yield: 47.9 mg, 73%.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.47 (d, *J* = 8.4 Hz, 2H, *Ar*), 7.20 (d, *J* = 8.4 Hz, 2H, *Ar*), 5.16 (dd, *J* = 7.8, 6.0 Hz, 1H, ArC*H*), 4.06 [ddd, *J* = 13.3, 6.5, 6.5 Hz, 1H, $CH_2C(OAc)H_aH_b$], 4.04 [ddd, *J* = 13.3, 6.5, 6.5 Hz, 1H, $CH_2C(OAc)H_aH_b$], 2.07 (s, 3H, $COCH_3$), 2.04 (s, 3H, $COCH_3$), 1.94 [dddd, *J* = 13.8, 10.3, 7.8, 5.2 Hz, 1H, $ArCH(OAc)CH_aH_b$], 1.81 [dddd, *J* = 13.8, 10.3, 6.0, 5.7 Hz, 1H, $ArCH(OAc)CH_aH_b$], 1.66 [ddddd, *J* = 13.6, 10.3, 6.5, 6.5, 5.2 Hz, 1H,

ArCH(OAc)$CH_2CH_aH_b$], and 1.57 [ddddd, J = 13.6, 10.3, 6.5, 6.5, 5.7 Hz, 1H, ArCH(OAc)$CH_2CH_aH_b$].

^{13}C NMR ($CDCl_3$, 125 MHz): δ 171.2, 170.4, 139.5, 131.9, 128.4, 122.1, 75.0, 64.0, 32.9, 24.9, 21.4, and 21.1.

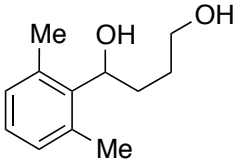
TLC: R_f = 0.3 in 5:1 hexanes: EtOAc.

GC-MS (5029017): t_R=13.042 min, m/z 287 [$(M\text{-}Ac)^+$, 22], 285 [$(M\text{-}Ac)^+$, 22], 227 [$(M\text{-}AcOCH_2CH_2CH_2)^+$, 100], 225 [$(M\text{-}AcOCH_2CH_2CH_2)^+$, 100], and 185 (60).

IR (neat): 3029 (w), 2927 (s), 1733 (s), 1226 (s), 1009 (m), 820 (s), and 718 (m) cm^{-1}.

HRMS (ESI/TOF): Calcd for $(C_{14}H_{17}BrO_4Na)^+$: 351.0202. Found: 351.0190.

1-(2,6-Dimethylphenyl)butane-1,4-diol (5d)



5d

Yield: 31.4 mg, 81%.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.04 (dd, J = 8.3, 6.5 Hz, 1H, Ar-*H*), 6.97 (app d, J = 7.4 Hz, 2H, Ar-*H*), 5.21 (dd, J = 9.7, 4.1 Hz, 1H, ArC*H*), 3.76 [ddd, J = 10.8, 6.3, 5.4 Hz, 1H, $CH_2C(OH)H_aH_b$], 3.72 [ddd, J = 10.8, 7.1, 5.3 Hz, 1H, $CH_2C(OH)H_aH_b$], 2.45 [s, 6H, $Ar(CH_3)_2$], 2.15 (br s, 2H, O*H*), 2.13-2.05 [m, 1H, $CH(OH)CH_2$ or $CH(OH)CH_2CH_2$], 1.86-1.68 [m, 3H, $CH(OH)CH_2$ and $CH(OH)CH_2CH_2$].

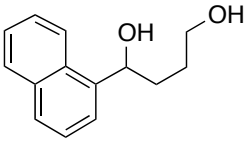
^{13}C NMR ($CDCl_3$, 125 MHz): δ 140.0, 136.1, 129.6, 127.2, 71.9, 63.0, 32.9, 30.5, and 21.0 (2).

TLC: R_f = 0.2 in 1:4 hexanes: EtOAc.

IR (neat): 3347 (br), 3065 (w), 2929 (w), 1467 (m), 1174 (s), 1043 (s), and 769 (s) cm^{-1}.

HRMS (ESI/TOF): Calcd for $(C_{12}H_{18}O_2Na)^+$: 217.1199. Found: 217.1179.

1-(Naphthalen-1-yl)butane-1,4-diol (5e)



5e

Yield: 36.3 mg, 84%.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 8.04 (d, J = 7.8 Hz, 1H, *Ar*), 7.86-7.85 (nfo m, 1H, *Ar*), 7.75 (d,

J = 8.1 Hz, 1H, *Ar*), 7.64 (ddd, J = 7.1, 1.0, 1.0 Hz, 1H, *Ar*), 7.51-7.43 (m, 3H, *Ar*), 5.47 [dd, J = 8.4, 3.6 Hz, 1H, ArC*H*(OH)], 3.70 [ddd, J = 10.6, 6.0, 5.5 Hz, 1H, $CH_2C(OH)H_aH_b$], 3.65 [ddd, J = 10.6, 6.9, 5.5 Hz, 1H, $CH_2C(OH)H_aH_b$], 3.10 (br, 2H, O*H*), 2.06 [dddd, J = 14.3, 7.0, 7.0, 3.6 Hz, 1H, ArC*H*(OH)CH_aH_b], 1.91 [dddd, J = 14.3, 8.4, 7.4, 6.6 Hz, 1H, ArCH(OH)CH_aH_b], and 1.81-1.69 [m, 2H, ArCH(OH)CH_2CH_aH_b].

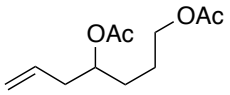
^{13}C NMR ($CDCl_3$, 125 MHz): δ 140.5, 133.9, 130.4, 129.1, 128.0, 126.2, 125.67, 125.63, 123.2, 122.9, 71.1, 63.0, 35.7, and 29.7.

TLC: R_f = 0.2 in 1:4 hexanes: EtOAc.

IR (neat): 3292 (br), 3049 (w), 2938 (w), 1105 (m), 1080 (m), and 837 (s) cm^{-1}.

HRMS (ESI/TOF): Calcd for $(C_{14}H_{16}O_2Na)^+$: 239.1043. Found: 239.0996.

Hept-6-ene-1,4-diyl diacetate (5f-diOAc)



***5f**-diOAc*

Yield: 27.5 mg, 65%.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 5.74 (dddd, J = 17.3, 10.2, 7.1, 7.1 Hz, 1H, CH_2=C*H*CH_2), 5.09 (dddd, J = 17.3, 2.1, 1.2, 1.2 Hz, 1H, $CH_{cis}H_{trans}$=CH), 5.06 (dddd, J = 10.2, 2.1, 1.2, 1.2 Hz, 1H, C$H_{cis}H_{trans}$=CH), 4.94[dddd, J = 7.4, 6.1, 6.1, 6.1 Hz, 1H, CH_2=CHCH_2C*H*(OAc)], 4.05[app dd, J = 6.3, 6.3 Hz, 2H, CH_2CH_2CH_aH_b(OAc)], 2.33-2.30 (m, 2H, CH_2=CHCH_2), 2.044 (s, 3H, Ac), 2.040 (s, 3H, Ac), and 1.70-1.57 [m, 4H, CH(OAc)CH_2C$H_2$$CH_2$(OAc)].

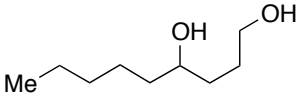
^{13}C NMR ($CDCl_3$, 125 MHz): δ 171.3, 170.9, 133.6, 118.1, 72.9, 64.3, 38.8, 30.2, 24.8, 21.4, and 21.2.

TLC: R_f = 0.5 in 5:1 hexanes: EtOAc.

IR (neat): 2923 (s), 2853 (s), 1784 (s), 1508 (m), 1364 (m), 1240 (s), and 1196 (m) cm^{-1}.

HRMS (ESI/TOF): Calcd for $(C_{11}H_{18}O_4Na)^+$: 237.1097. Found: 237.1087.

Nonane-1,4-diol (5g)



5g

Yield: 24.1 mg, 75%.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 3.69-3.59 [m, 2H, $CH_2C(OH)HCH_2$, $CH_2C(OH)H_aH_b$], 3.68 [ddd, J = 10.6, 5.2, 5.2 Hz, 1H, $CH_2C(OH)H_aH_b$], 2.86 (br, 2H, O*H*), 1.71-1.57 (m, 3H, CH_2), 1.49-1.38 (m, 4H, CH_2), 1.34-1.22 (m, 5H, CH_2), and 0.88 (dd, J = 7.0, 7.0 Hz, 3H, CH_3CH_2).

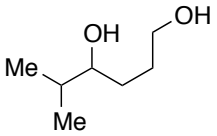
^{13}C NMR ($CDCl_3$, 125 MHz): δ 72.1, 63.2, 37.8, 34.6, 32.1, 29.3, 25.6, 22.8, and 14.2.

TLC: R_f = 0.2 in 1:4 hexanes: EtOAc.

IR (neat): 3320 (br), 2926 (s), 1457 (m), 1051 (s), and 1009 (s) cm^{-1}.

HRMS (ESI/TOF): Calcd for $(C_9H_{20}O_2Na)^+$: 183.1356. Found: 183.1313.

5-Methylhexane-1,4-diol (5h)



5h

Yield: 17.7 mg, 67%.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 3.71 [ddd, J = 10.8, 5.6, 5.6 Hz, 1H, $CH_2C(OH)H_aH_b$], 3.65 [ddd, J = 10.8, 6.0, 6.0 Hz, 1H, $CH_2C(OH)H_aH_b$], 3.39 (ddd, J = 9.1, 5.3, 2.6 Hz, 1H, $(CH_3)_2CHCH(OH)$], 2.34(br, 2H, O*H*), 1.73-1.61 [m, 4H, $CH(OH)CH_aH_b$ and $CH(OH)CH_2CH_2$, $(CH_3)_2CH$], 1.44 [dddd, J = 13.8, 9.1, 7.0, 7.0 Hz, 1H, $CH(OH)CH_aH_b$], 0.92 [d, J = 6.8 Hz, 3H, $CH(CH_3)_2$], and 0.91 [d, J = 6.8 Hz, 3H, $CH(CH_3)_2$].

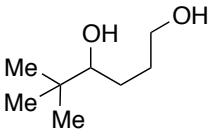
^{13}C NMR ($CDCl_3$, 125 MHz): δ 77.0, 63.3, 34.0, 31.3, 29.8, 18.9, and 17.6.

TLC: R_f = 0.2 in 1:4 hexanes: EtOAc.

IR (neat): 3347 (br), 2925 (s), 1457 (m), 1089 (s), and 1023 (s) cm^{-1}.

HRMS (ESI/TOF): Calcd for $(C_7H_{16}O_2Na)^+$: 155.1043. Found: 155.0858.

5,5-Dimethylhexane-1,4-diol (5i)



5i

Yield: 20.7 mg, 71%.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 3.73 [ddd, J = 10.8, 5.3, 5.3 Hz, 1H, $CH_2C(OH)H_aH_b$], 3.66 [ddd, J = 10.8, 7.1, 4.5 Hz, 1H, $CH_2C(OH)H_aH_b$], 3.23 [dd, J = 10.5, 1.5 Hz, 1H, $(CH_3)_3CCH(OH)$], 2.25 (br, 2H, O*H*), 1.78-1.67 [m, 3H, $CH(OH)CH_2$ and $CH(OH)CH_2CH_2$], 1.39-1.27 [m, 1H,

CH(OH)CH_2 or CH(OH)CH_2CH_2], and 0.90 [s, 9H, C(CH_3)$_3$].

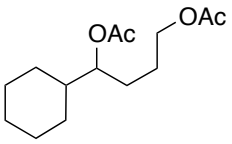
^{13}C NMR ($CDCl_3$, 125 MHz): δ 80.4, 63.3, 35.2, 30.7, 28.6, and 25.9.

TLC: R_f = 0.2 in 1:4 hexanes: EtOAc.

IR (neat): 3348 (br), 2926 (s), 1466 (m), 1057 (s), 974 (s), and 737 (s) cm^{-1}.

HRMS (ESI/TOF): Calcd for $(C_8H_{18}O_2Na)^+$: 169.1199. Found: 169.1190.

1-Cyclohexylbutane-1,4-diyl diacetate (5j-diOAc)



***5j**-diOAc*

Yield: 38.4 mg, 75%.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 4.75 [ddd, *J* = 10.6, 5.6, 3.4Hz, 1H, CyC*H*(OAc)CH_2], 4.04 [app dd, *J* = 6.1, 6.1 Hz, 2H, CH_2C(OAc)H_aH_b], 2.05 (s, 3H, Ac), 2.04 (s, 3H, Ac), 1.76-1.52 (m, 9H, *Cy,* CH_2), 1.51-1.43 [m, 1H, Cy or CyCH(OAc)CH_2], 1.25-1.16 (m, 2H, *Cy,* CH_2), 1.11 (ddddd, *J* = 12.5, 12.5, 12.5, 3.3, 3.3 Hz, 1H, *Cy*), and 1.03-0.94 (m, 2H, *Cy*).

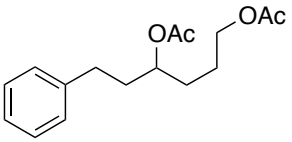
^{13}C NMR ($CDCl_3$, 125 MHz): δ 171.3, 171.2, 77.5, 64.5, 41.4, 29.1, 28.4, 27.8, 26.5, 26.3, 26.2, 25.0, 21.3, and 21.2.

TLC: R_f = 0.5 in 5:1 hexanes: EtOAc.

IR (neat): 2927 (m), 2854 (m), 1732 (s), 1367 (m), 1228 (s), 1019 (s), and 632 (s) cm^{-1}.

HRMS (ESI/TOF): Calcd for $(C_{14}H_{24}O_4Na)^+$: 279.1567. Found: 279.1565.

6-Phenylhexane-1,4-diyl diacetate (5k-diOAc)



***5k**-diOAc*

Yield: 38.9 mg, 70%.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.28 (dd, *J* =7.4, 7.4 Hz, 2H, *Ph*), 7.20-7.16 (m, 3H, *Ph*), 4.96 [dddd, *J* = 7.9, 6.2, 6.2, 4.6 Hz, 1H, PhCH_2CH_2C*H*(OAc)], 4.08-4.01 [m, 2H, CH(OAc)CH_2CH_2C(OAc)H_aH_b], 2.66 (ddd, *J* = 13.8, 10.0, 6.0 Hz, 1H, PhC$H_a$$H_b$), 2.60 (ddd, *J* = 13.8, 9.8, 6.4 Hz, 1H, Ph$CH_a$$H_b$), 2.041 [s, 3H, C(O)C$H_3$], 2.038 [s, 3H, C(O)C$H_3$], 1.91 (dddd, *J* = 14.0, 9.8, 7.9, 6.0 Hz, 1H, PhCH_2C$H_a$$H_b$), 1.84 (ddddd, *J* = 14.0, 10.0, 6.4, 4.6

Hz, 1H, $PhCH_2CHaH_b$), and 1.69-1.61 [m, 4H, $CH(OAc)CH_2CH_2CH_2(OAc)$].

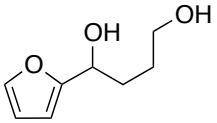
^{13}C NMR ($CDCl_3$, 125 MHz): δ 171.3, 171.0, 141.6, 128.6, 128.5, 126.2, 73.4, 64.3, 36.0, 31.9, 30.8, 24.7, 21.4, and 21.1.

TLC: R_f = 0.5 in 5:1 hexanes: EtOAc.

IR (neat): 3027 (w), 2954 (w), 1732 (s), 1367 (m), 1230 (s), 1023 (m), and 699 (m) cm^{-1}.

HRMS (ESI/TOF): Calcd for $(C_{16}H_{22}O_4Na)^+$: 301.1410. Found: 301.1412.

1-(Furan-2-yl)butane-1,4-diol (5l)



5l

Yield: 25.1 mg, 80%.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.37 (dd, J = 1.8, 0.8 Hz, 1H, *Ar-H5*), 6.33 (dd, J = 3.2, 1.8 Hz, 1H, *Ar-H4*), 6.24 (ddd, J = 3.2, 0.8, 0.8 Hz, 1H, *Ar-H3*), 4.74 [dd, J = 7.8, 5.1 Hz, 1H, $ArCH(OH)$], 3.72 [ddd, J = 12.8, 6.4, 5.8 Hz, 1H, $CH_2C(OH)H_aH_b$], 3.69 [ddd, J = 12.8, 6.6, 5.6 Hz, 1H, $CH_2C(OH)H_aH_b$], 2.21 (br, 2H, O*H*), 2.04-1.91 [m, 2H, $CH(OH)CH_2$ and/or $CH(OH)CH_2CH_2$], and 1.77-1.65 [m, 2H, $CH(OH)CH_2$ and/or $CH(OH)CH_2CH_2$].

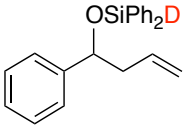
^{13}C NMR ($CDCl_3$, 125 MHz): δ 156.9, 142.1, 110.4, 106.0, 67.9, 63.0, 32.9, and 29.0.

TLC: R_f = 0.2 in 1:4 hexanes: EtOAc.

IR (neat): 3379 (br), 3057 (w), 2926 (w), 1437 (m), 1023 (s), and 699 (s) cm^{-1}.

HRMS (ESI/TOF): Calcd for $(C_8H_{12}O_3Na)^+$: 179.0679. Found: 179.0662.

Diphenyl(1-phenylbut-3-enyloxy)silane (1a-D)



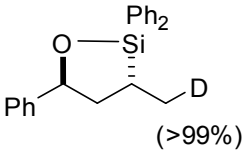
1a-D

For ^{1}H and ^{13}C NMR, see **1a** (page SI-11).

IR (neat): 3068 (w), 2908 (w), 1546 (m), 1428 (m), 1112 (m), 1062 (m), 915 (m), 695 (s), and 599 (s) cm^{-1}.

HRMS (ESI/TOF): Calcd for $(C_{22}H_{21}DOSiNa)^{+}$: 354.1395. Found: 354.1321.

***Trans*-3-Methyl-2,2,5-triphenyl-1,2-oxasilolane (*trans*-2a-D)**



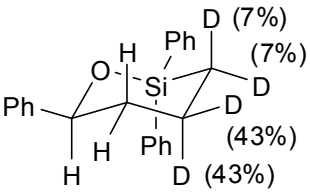
trans-2a-D

For ^{1}H and ^{13}C NMR, see *trans*-**2a** (page SI-19).

IR (neat): 3068 (w), 2921 (w), 1428 (m), 1111 (m), 1065 (m), 695 (s), and 497 (s) cm^{-1}.

HRMS (ESI/TOF): Calcd for $(C_{22}H_{21}DOSiNa)^{+}$: 354.1395. Found: 354.1330.

2,2,6-Triphenyl-1,2-oxasilinane (3a-D)



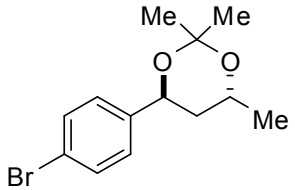
3a-D

For ^{1}H and ^{13}C NMR, see **3a** (page SI-27).

IR (neat): 3068 (w), 2924 (w), 1428 (m), 1117 (m), 1062 (m), 696 (s), and 604 (s) cm^{-1}.

HRMS (ESI/TOF): Calcd for $(C_{22}H_{21}DOSiNa)^{+}$: 354.1395. Found: 354.1333.

Trans-4-(4-Bromophenyl)-2,2,6-trimethyl-1,3-dioxane (SI-1)[2]



SI-1

Diol *trans*-**5c** (10 mg, 0.041 mmol), dimethoxypropane (15.1 µL, 0.13 mmol), camphorsulfonic acid (0.95 mg, 0.0041 mmol) was dissolved with DCM (250 µL, 0.16 M). The reaction mixture was stirred at rt for 12 h. Solvent was removed under reduced pressure and the crude mixture was purified by MPLC (hexanes/EtOAc =7.5:1, 6 mL/min, retention time 7 min) to afford acetonide **SI-1** (10.7 mg, 92%).

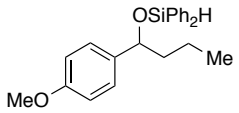
^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.46 (d, J = 8.5 Hz, 2H, *Ar*), 7.23 (d, J = 8.5 Hz, 2H, *Ar*), 4.84 (dd, J = 9.7, 6.2 Hz, 1H, ArC*H*), 4.10 (dqd, J = 8.7, 6.3, 6.3 Hz, 1H, CH_3C*H*), 1.95 (ddd, J = 13.0, 9.7, 6.3 Hz, 1H, $CH_3CHCH_aH_b$), 1.86 (ddd, J = 13.0, 8.7, 6.2 Hz, 1H, $CH_3CHCH_aH_b$), 1.44 [s, 6H, $C(CH_3)_2$], and 1.26 (d, J = 6.3 Hz, 3H, $CHCH_3$).

^{13}C NMR ($CDCl_3$, 125 MHz): δ 141.9, 131.7, 127.9, 121.3, 101.0, 68.1, 63.1, 41.6, 25.5, 25.2, and 22.0.

IR (neat): 3123 (w), 2984 (w), 1489 (s), 1222 (s), 1174 (s), 1130 (s), 811 (s), and 523 (m) cm^{-1}.

HRMS (ESI/TOF): Calcd for $(C_{13}H_{17}BrO_2+H)^+$: 285.0485. Found: 285.0443.

[1-(4-methoxyphenyl)butoxy]diphenylsilane (6)



6

For the preparation of the silyl ether **6**, see page S11.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.61 [dd, J = 8.0, 1.4 Hz, 2H, $Si\{C_6(H_{ortho})_2H_4\}_2$], 7.54 [dd, J = 8.0, 1.4 Hz, 2H, $Si\{C_6(H_{ortho})_2H_4\}_2$], 7.47-7.32 [m, 6H, $Si\{C_6(H_{ortho})_2(H_{meta})_2H_{para}\}_2$], 7.21 (d, J = 8.7 Hz, 2H, *p*-MeO*Ph*), 6.84 (d, J = 8.7 Hz, 2H, *p*-MeO*Ph*), 5.34 (s, 1H, Si*H*), 4.74 (dd, J = 7.0, 5.9 Hz, 1H, SiOC*H*CH_2), 3.81 (s, 3H, *Me*O), 1.88 (dddd, J = 13.5, 10.1, 7.0, 5.2 Hz, 1H,

---

[2] Rychnovsky, S. D.; Rogers, B.; Yang, G. *J. Org. Chem.* **1993**, *58*, 3511-3515.

SiOCHCH_aH$_b$), 1.68 (dddd, J = 13.5, 10.1, 5.9, 5.6 Hz, 1H, SiOCHCH$_a$$H_b$), 1.41 (ddqd, J = 14.9, 10.1, 7.4, 5.2 Hz, 1H, SiOCHCH$_2$CH_aH$_b$), 1.28 (ddqd, J = 14.9, 10.1, 7.4, 5.6 Hz, 1H, SiOCHCH$_2$CH$_a$$H_b$), and 0.87 (dd, J = 7.4, 7.4 Hz, 3H, CH$_2$CH_3).

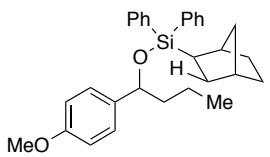
^{13}C NMR ($CDCl_3$, 125 MHz): δ 159.9, 136.5, 134.96, 134.86, 134.6, 134.4, 130.4, 130.3, 128.1, 128.0, 127.7, 113.7, 76.5, 55.4, 42.4, 19.1, and 14.1.

GC-MS (5032021): t_R=14.763 min, m/z 319 [(M-propyl)$^+$, 100], 361 [(M-H)$^+$,10], 197 (30), and 183 (30).

IR (neat): 3069 (w), 3049 (w), 2125 (m), 1588 (w), 1511 (m), 1245 (m), 811 (s), 731 (s), and 696 (s) cm^{-1}.

HRMS (ESI/TOF): Calcd for ($C_{23}H_{26}NaO_2Si$)$^+$: 385.1594. Found: 385.1596.

Bicyclo[2.2.1]heptan-2-yl[1-(4-methoxyphenyl)butoxy]diphenylsilane (7)



7

For the preparation of the 2-silylnorbornane **7**, see page S18. However, the reaction was carried out at either 23 °C or 80 °C, and either 20 mol % or 1.2 equiv of nbe was added.

<u>**7** was isolated in ca. 1:1 of diastereomers.</u>

^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.55 [dd, J = 8.0, 1.4 Hz, 2H, Si{C$_6$(H_{ortho})$_2$H$_4$}$_2$], 7.53 [dd, J = 8.0, 1.4 Hz, 2H, Si{C$_6$(H_{ortho})$_2$H$_4$}$_2$], 7.474 [dd, J = 8.0, 1.4 Hz, 2H, Si{C$_6$(H_{ortho})$_2$H$_4$}$_2$], 7.466 [dd, J = 8.0, 1.4 Hz, 2H, Si{C$_6$(H_{ortho})$_2$H$_4$}$_2$], 7.41-7.36 [m, 6H, Si{C$_6$(H$_{ortho}$)$_2$(H_{meta})$_2$$H_{para}$}$_2$], 7.34-7.28 [m, 6H, Si{C$_6$(H$_{ortho}$)$_2$(H_{meta})$_2$$H_{para}$}$_2$], 7.14 (d, J = 8.7 Hz, 2H, *p*-MeO*Ph*), 7.10 (d, J = 8.7 Hz, 2H, *p*-MeO*Ph*), 6.78 (d, J = 8.7 Hz, 2H, *p*-MeO*Ph*), 6.76 (d, J = 8.7 Hz, 2H, *p*-MeO*Ph*), 4.60 (dd, J = 6.3, 6.3 Hz, 1H, SiOC*H*CH$_2$), 4.56 (dd, J = 6.3, 6.3 Hz, 1H, SiOC*H*CH$_2$), 3.791 (s, 3H, *Me*O), 3.787 (s, 3H, *Me*O), 2.37 (d, J = 2.9 Hz, 1H, *H1*-norbornyl), 2.17 (d, J = 3.2 Hz, 1H, *H1*-norbornyl), 2.08 (dd, J = 4.0, 4.0 Hz, 1H, *H4*-norbornyl), 2.04 (dd, J = 4.0, 4.0 Hz, 1H, *H4*-norbornyl), 1.74-1.67 (m, 2H, SiOCHCH_2 or norbornyl), 1.63-1.41 (m, 9H, SiOCHCH$_2$CH$_2$ or norbornyl), 1.40-1.33 (m, 1H, SiOCHCH$_2$CH_2 or norbornyl), 1.31-1.01 (m, 10H, SiOCHCH$_2$CH_2 or norbornyl), 0.85 (app d, J = 9.3 Hz, 1H, norbornyl), 0.78 (app d, J = 9.4 Hz, 1H, norbornyl), 0.73 (dd, J = 7.6, 7.6 Hz, 3H, CH$_2$CH_3), 0.72 (dd, J = 7.6, 7.6 Hz, 3H, CH$_2$CH_3), 0.70 (app d, J = 9.4 Hz, 1H, norbornyl), and 0.64 (d, J = 9.4 Hz, 1H, norbornyl).

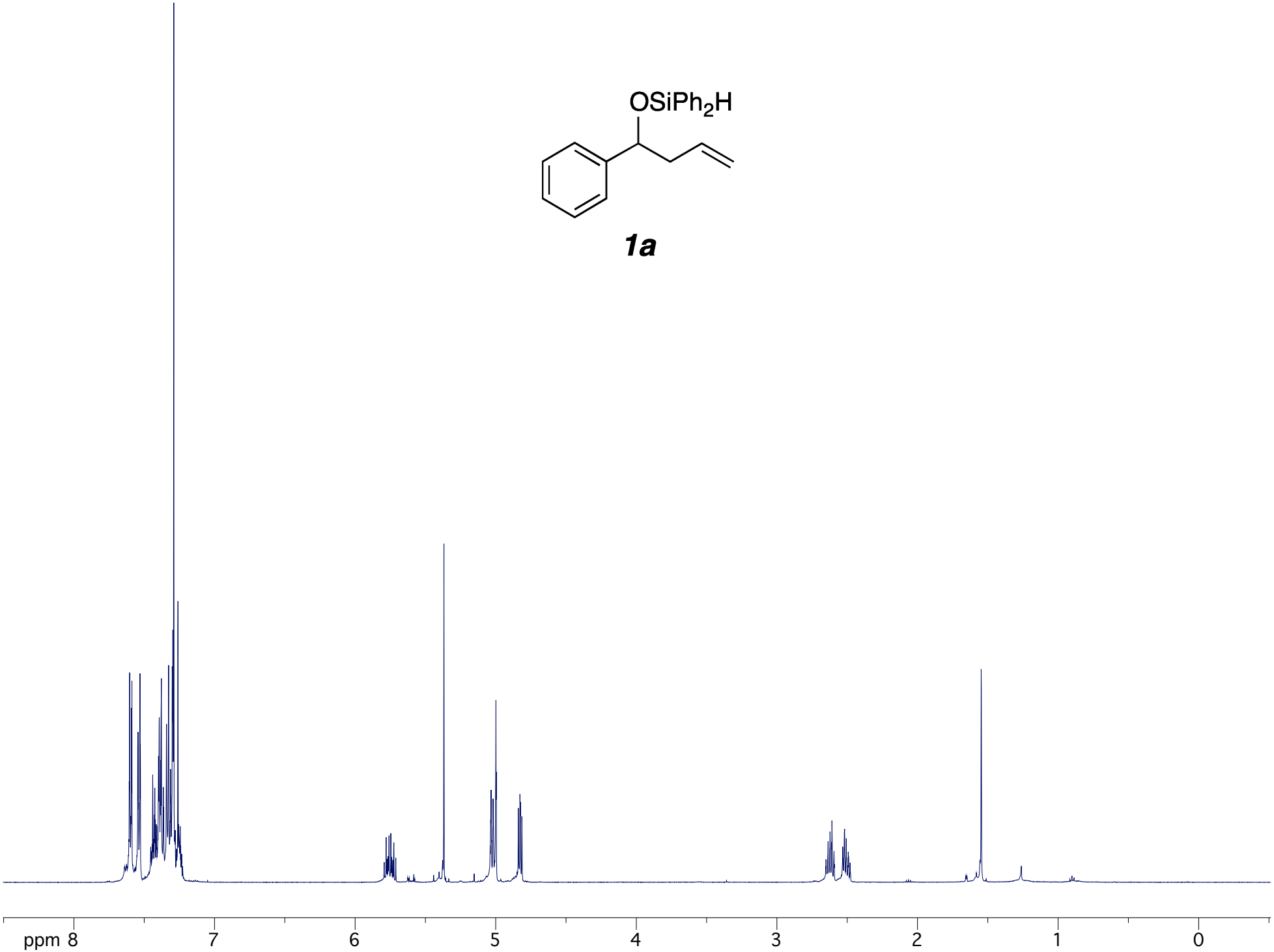
^{13}C NMR ($CDCl_3$, 125 MHz): δ 158.73, 158.66, 137.49, 137.37, 135.73, 135.69, 135.59 (2), 135.47, 135.42, 135.3, 135.1, 129.75, 129.71, 129.68 (2), 128.0 (2), 127.9 (2), 127.68, 127.65, 113.5, 113.4, 75.26, 75.23, 55.47, 55.44, 42.71, 42.67, 37.99, 37.83, 37.67, 37.59, 37.03, 36.89, 34.43, 34.22, 32.70, 32.51, 29.26 (2), 27.19, 27.10, 18.74, 18.67, 14.14, and 14.10.

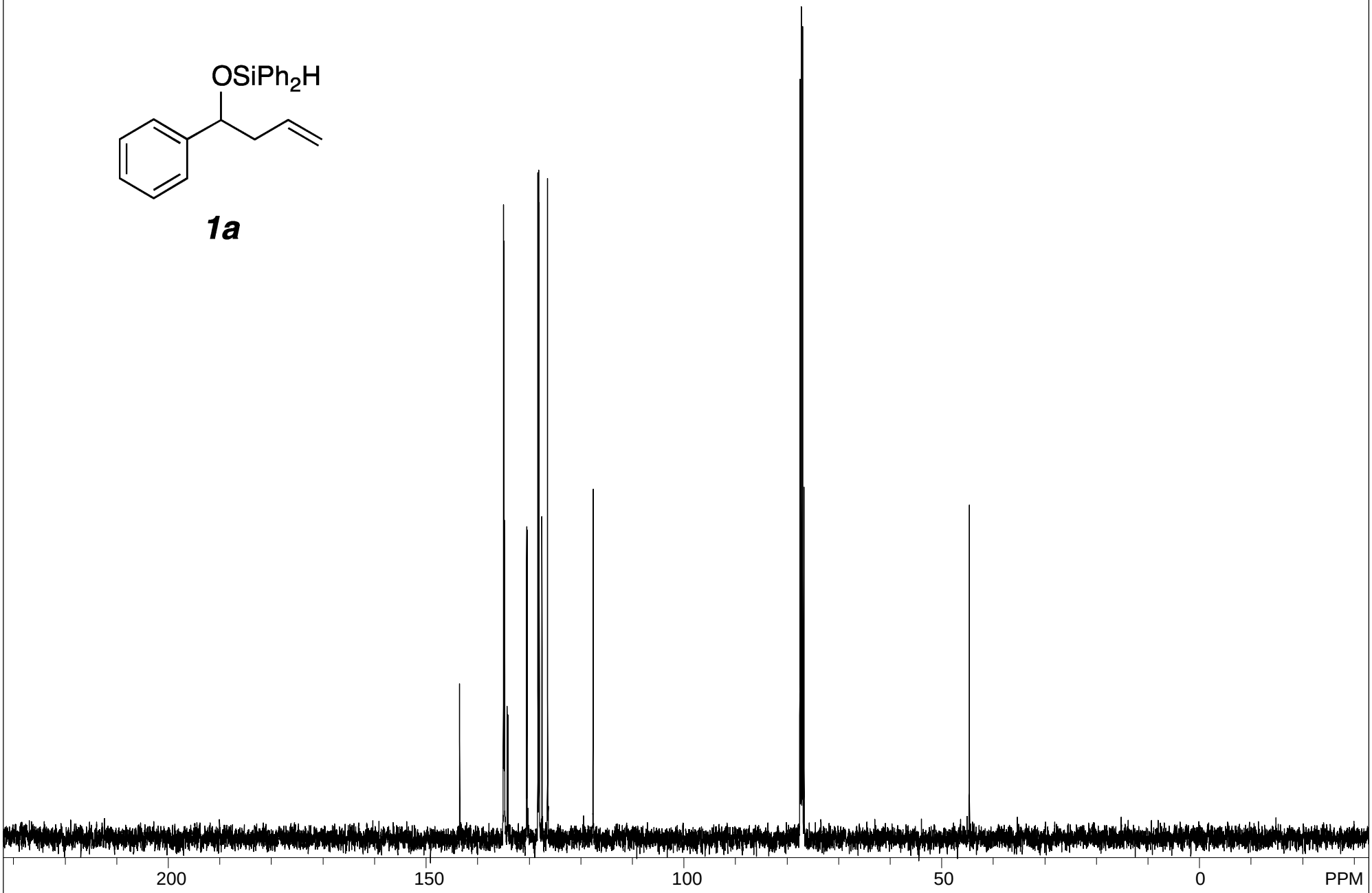
GC-MS (5032021): t_R=16.908 min, m/z 413 [(M-propyl)$^+$, 100], 455 [(M-H)$^+$, 8], 379 (50), 199 (80), and 183 (30).

IR (neat): 3068 (w), 3048 (w), 2126 (m), 1588 (w), 1511 (m), 1246 (m), 1036 (m), 699 (s), and 503 (s) cm^{-1}.

HRMS (ESI/TOF): Calcd for $(C_{30}H_{36}NaO_2Si)^+$: 479.2377. Found: 479.2371.

1a

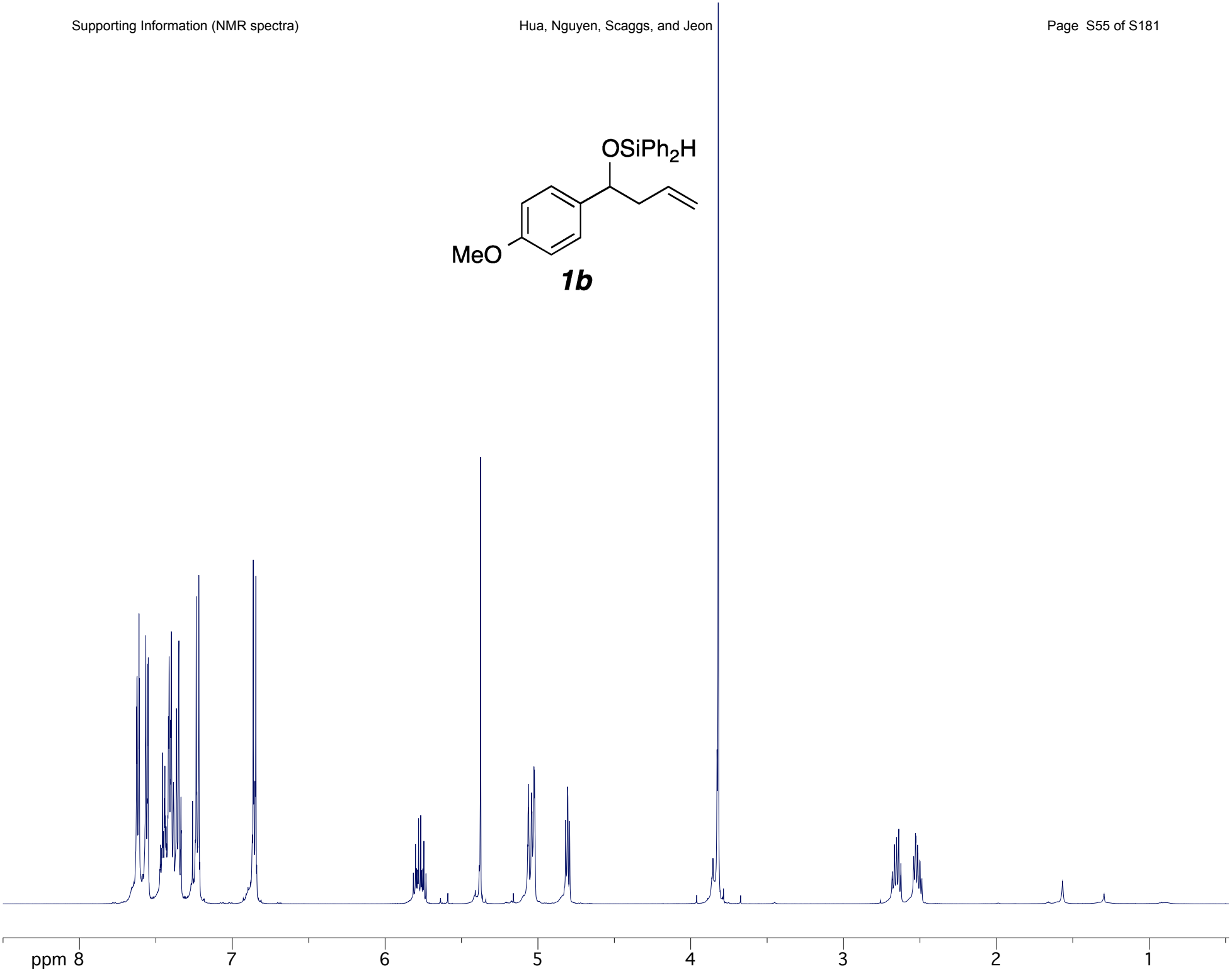




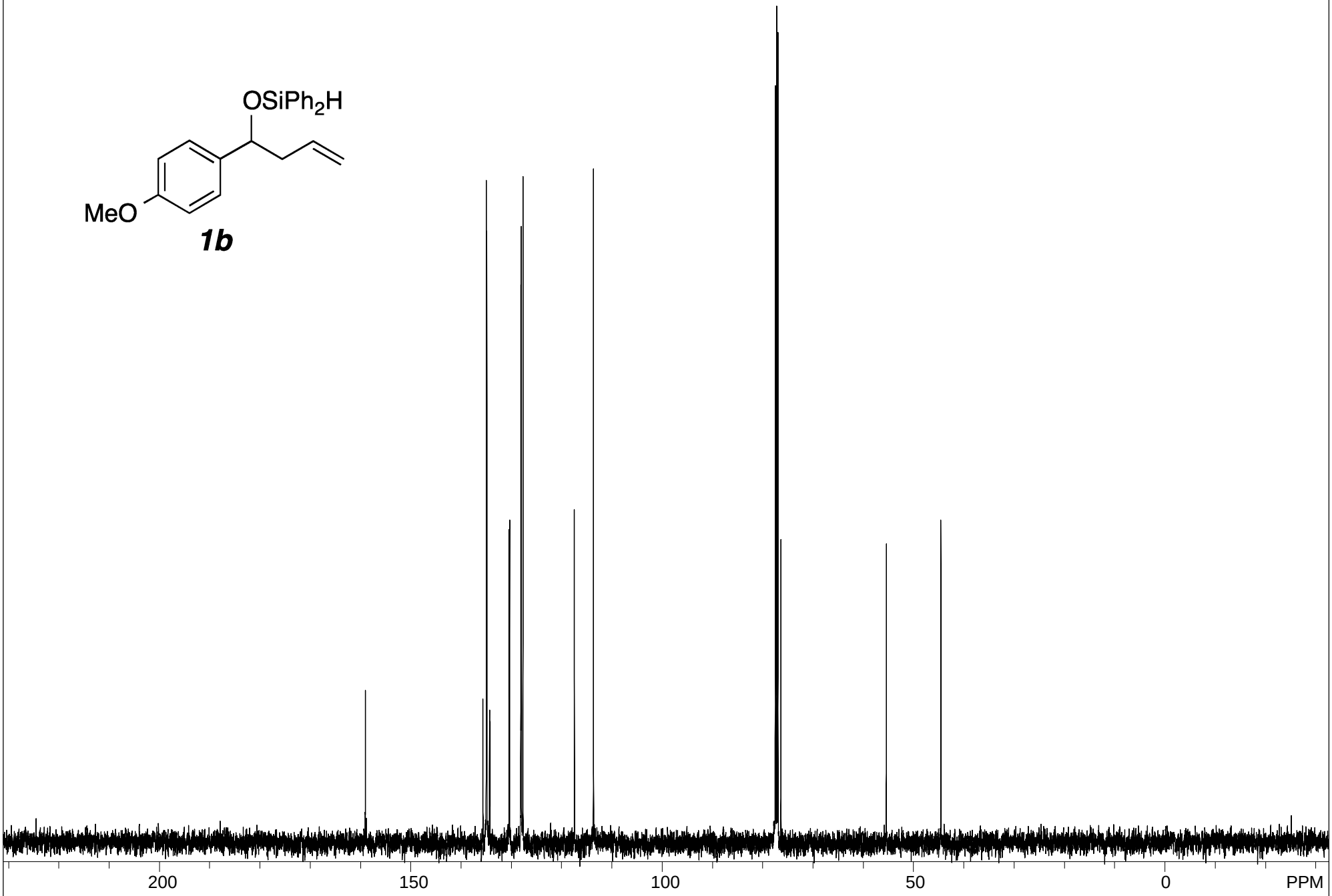
S#757732 (single pulse decoupled gated NOE) USER: delta -- DATE: Feb 14, 2013 11:30:32

F1: 125.778	F2: 500.162	SW1: 39308		OF1: 12587.8		PTS1d: 32768	
EX: single_pulse_dec		PW: 3.6 us, 30deg	PD: 2.0 sec	NA: 72	LB: 0.0	Nuts - $YH_silyether_Ph_M3_13C-2 3_1a.jdf	

1b

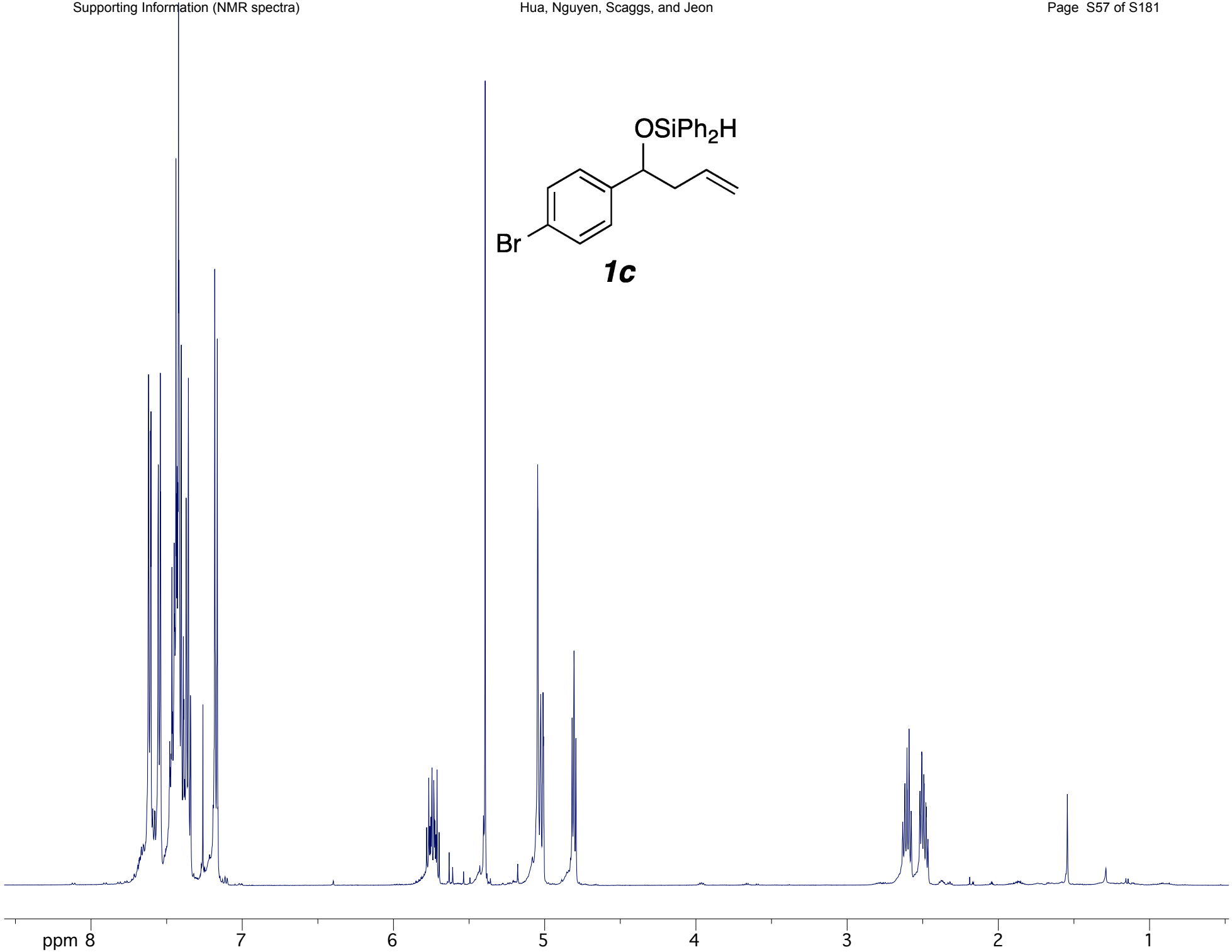


1b

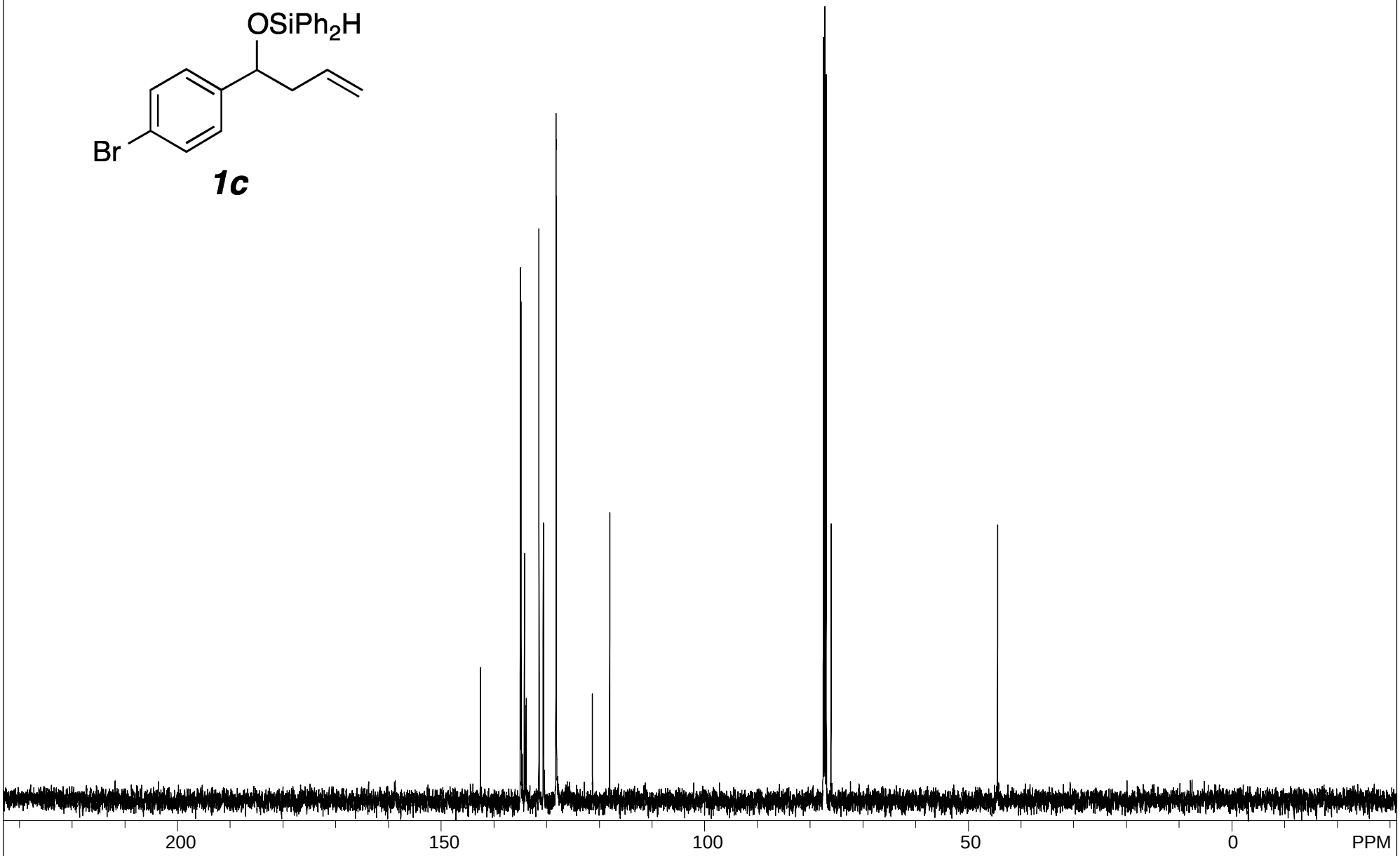


S#406833 (single pulse decoupled gated NOE) USER: delta -- DATE: Feb 15, 2013 01:44:31

F1: 125.778	F2: 500.162	SW1: 39308		OF1: 12586.6		PTS1d: 32768	
EX: single_pulse_dec		PW: 3.6 us, 30deg	PD: 2.0 sec	NA: 46	LB: 0.0	Nuts - $YH_silyether_pMeO_M1_13C-2_1b.jdf	

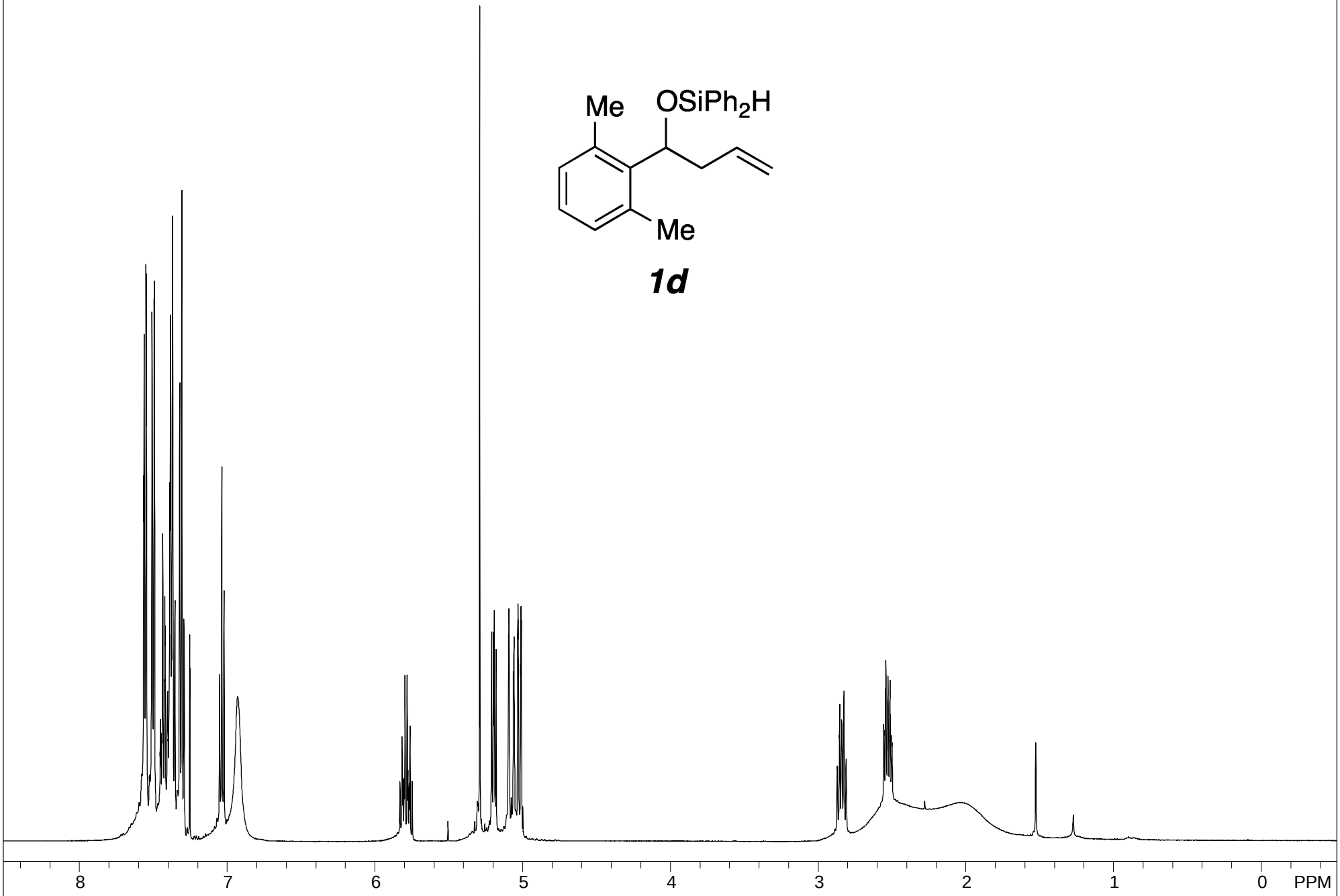

OSiPh2H
Br
1c
ppm 8
7
6
5
4
3
2
1

1c



S#586710 (single pulse decoupled gated NOE) USER: delta -- DATE: Feb 15, 2013 06:44:19

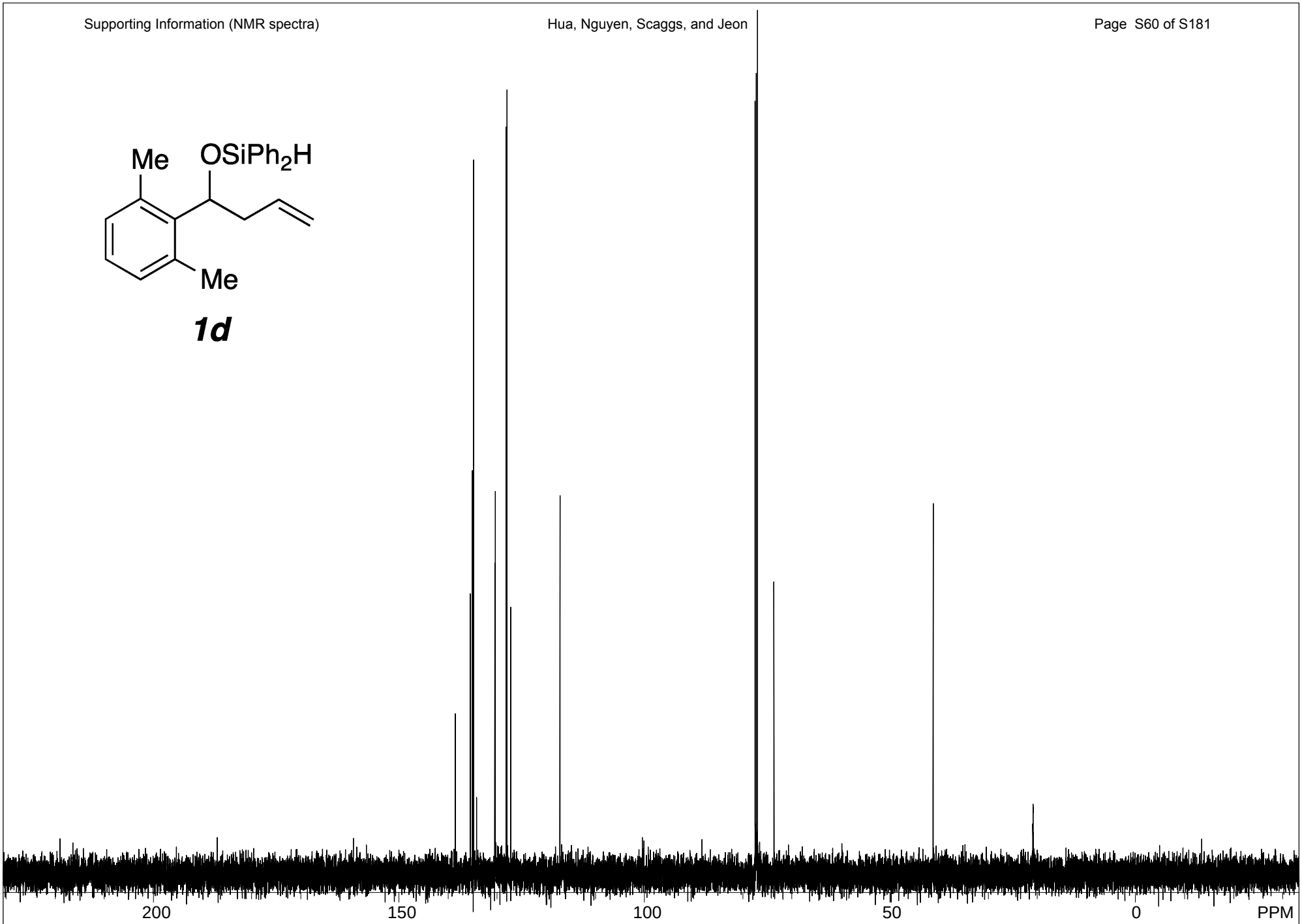
F1: 125.778	F2: 500.162	SW1: 39308		OF1: 12587.8		PTS1d: 32768	
EX: single_pulse_dec		PW: 3.6 us, 30deg	PD: 2.0 sec	NA: 46	LB: 0.0	Nuts - $YH_silyether_pBr_M2_13C-2.jdf	



S#786664 (single_pulse) USER: delta -- DATE: Mar 8, 2013 12:17:37

F1: 500.162	F2: 500.162	SW1: 9384		OF1: 2500.8		PTS1d: 16384 , 16384
EX: single_pulse.ex2		PW: 6.3 us, 45deg	PD: 5.0 sec	NA: 14	LB: 0.0	Nuts - YH_silyether_diMe_M2_1H-1_1d_1H.jdf

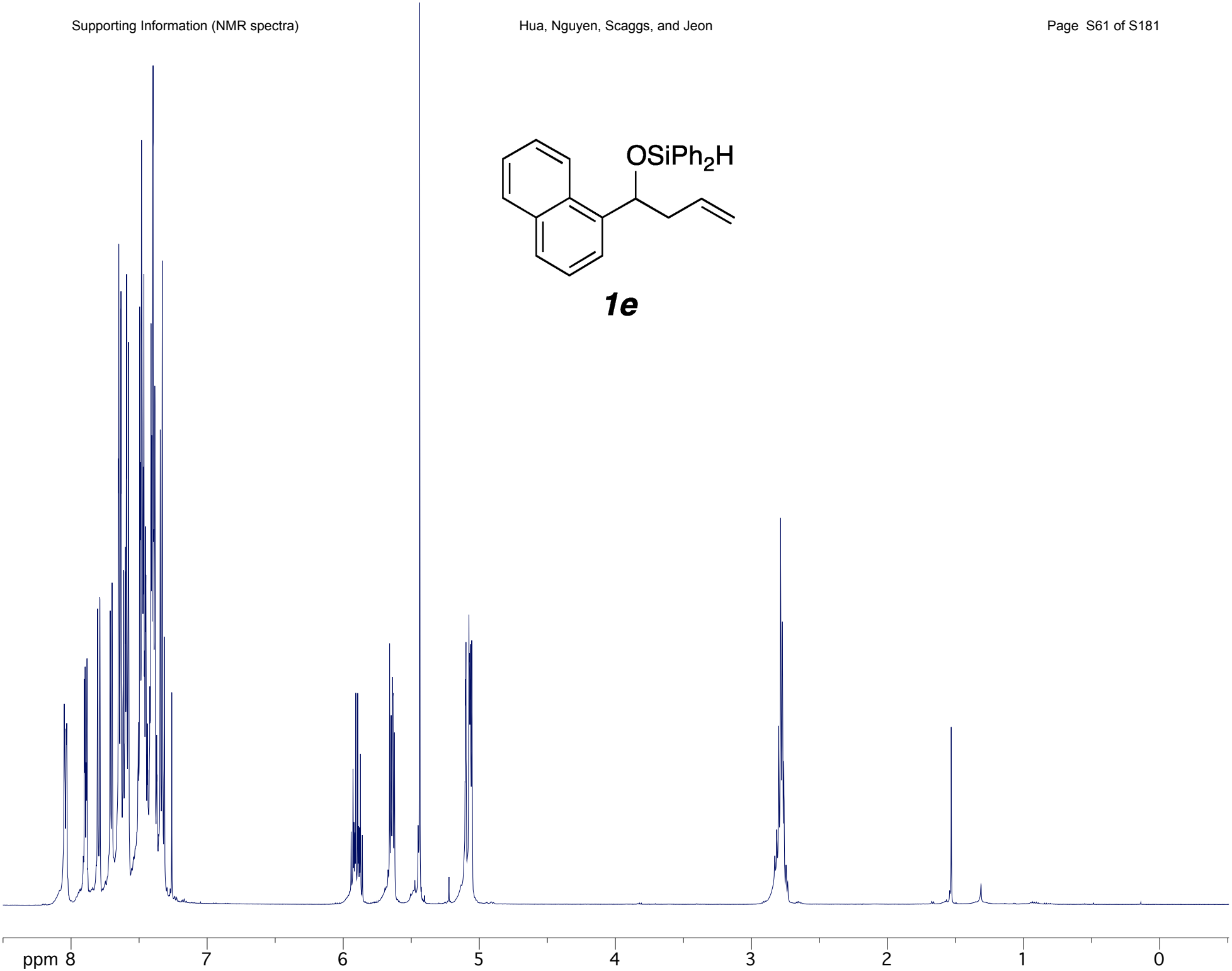
1d



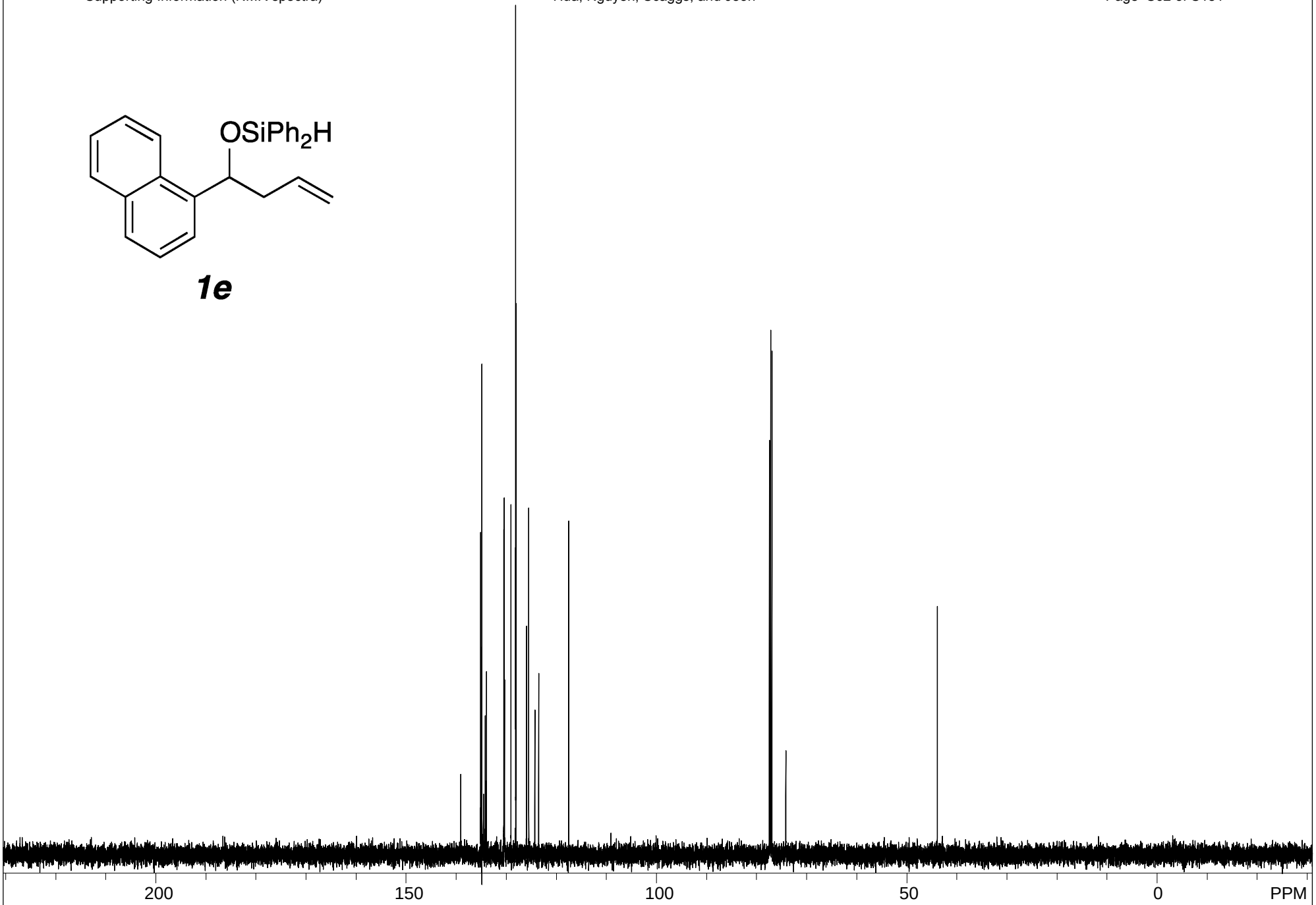
S#427394 (single pulse decoupled gated NOE) USER: delta -- DATE: Feb 15, 2013 02:19:11

F1: 125.778	F2: 500.162	SW1: 39308		OF1: 12589.0		PTS1d: 32768 , 32768
EX: single_pulse_dec		PW: 3.6 us, 30deg	PD: 2.0 sec	NA: 55	LB: 0.0	Nuts - YH_silyether_diMe_M1_13C-1_1d.jdf

1e

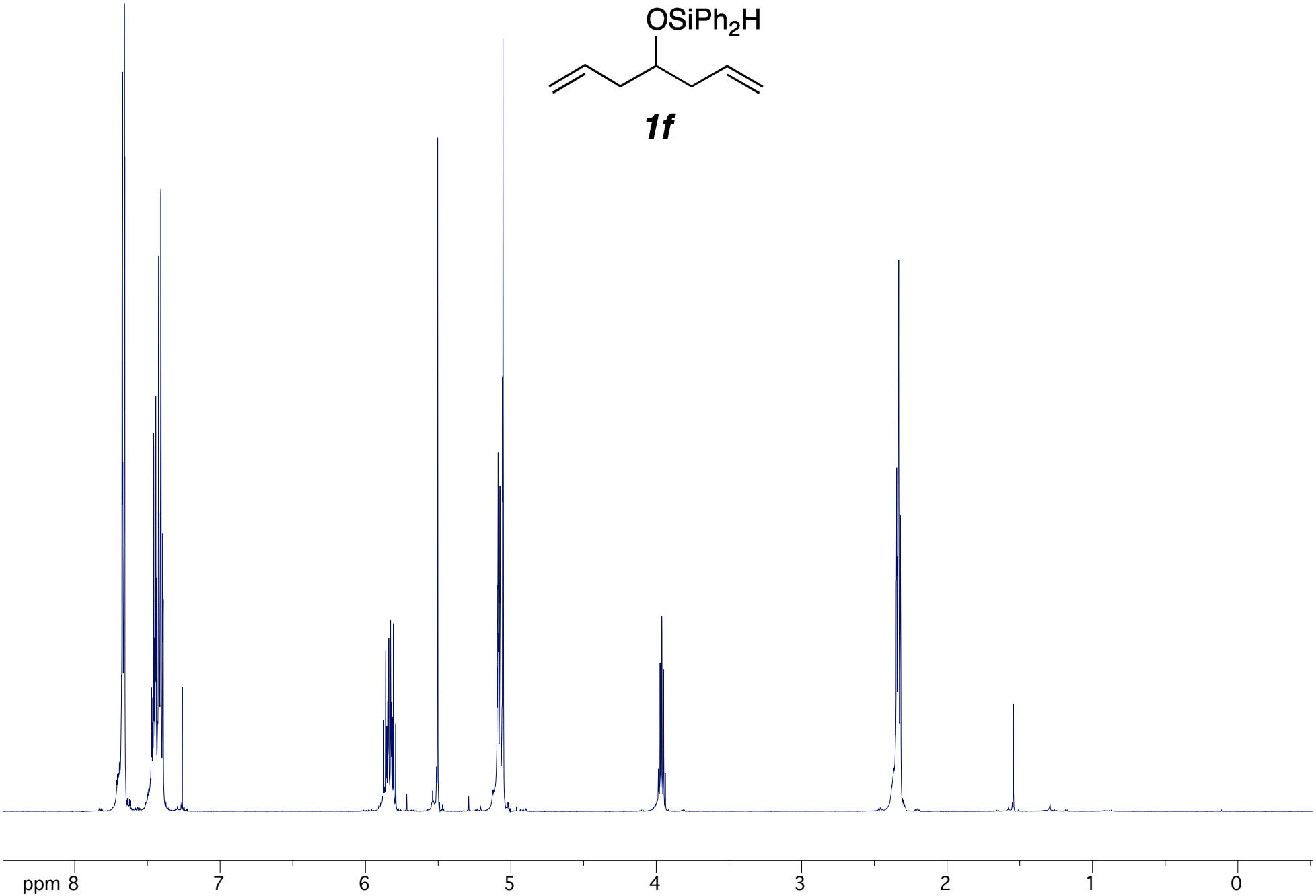


1e

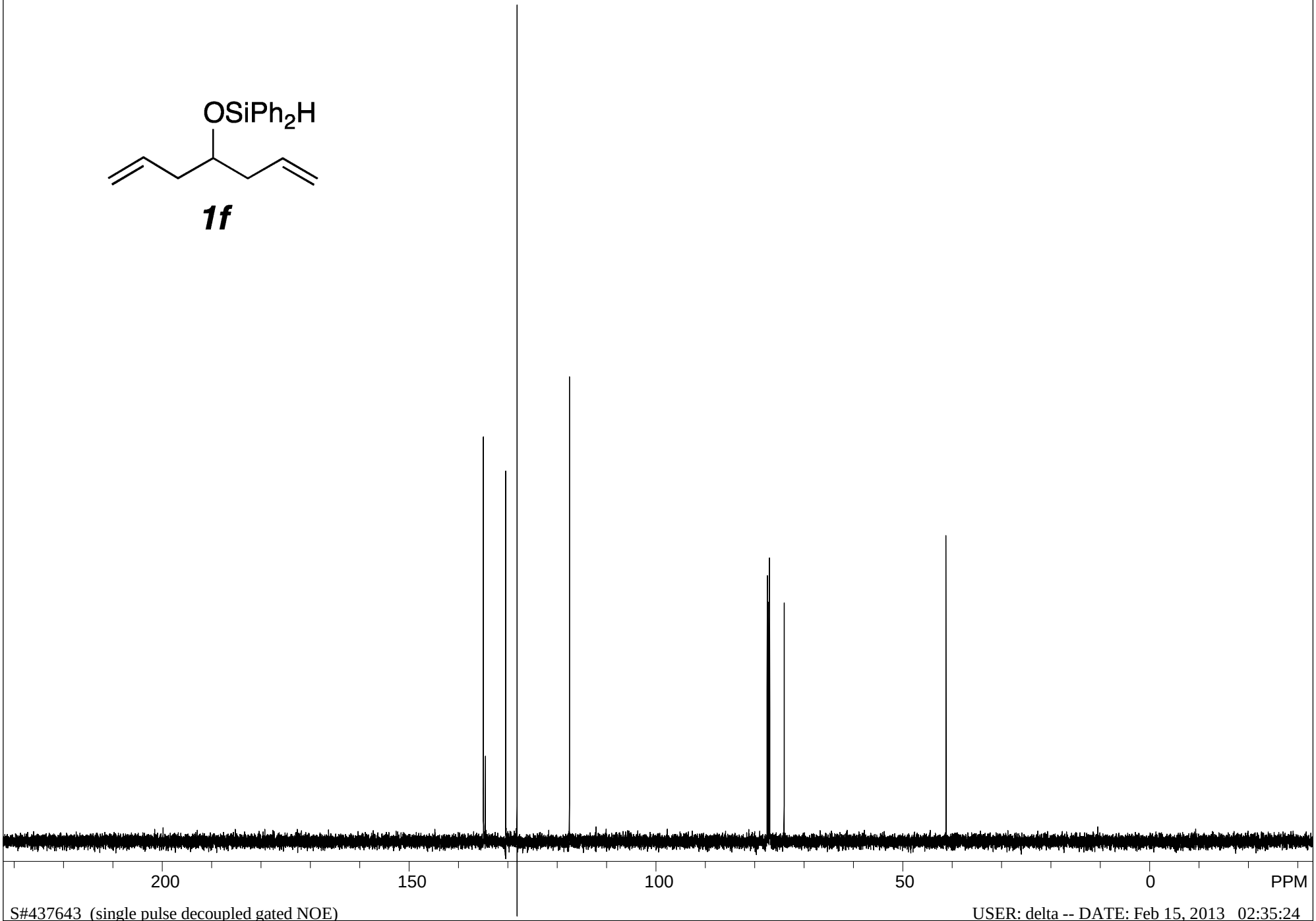


S#432727 (single pulse decoupled gated NOE) USER: delta -- DATE: Feb 15, 2013 02:29:06:

F1: 125.778	F2: 500.162	SW1: 39308		OF1: 12585.4		PTS1d: 32768 , 32768
EX: single_pulse_dec		PW: 3.6 us, 30deg	PD: 2.0 sec	NA: 77	LB: 0.0	Nuts - YH_silyether_Napht_M1_13C-1_1e.jdf

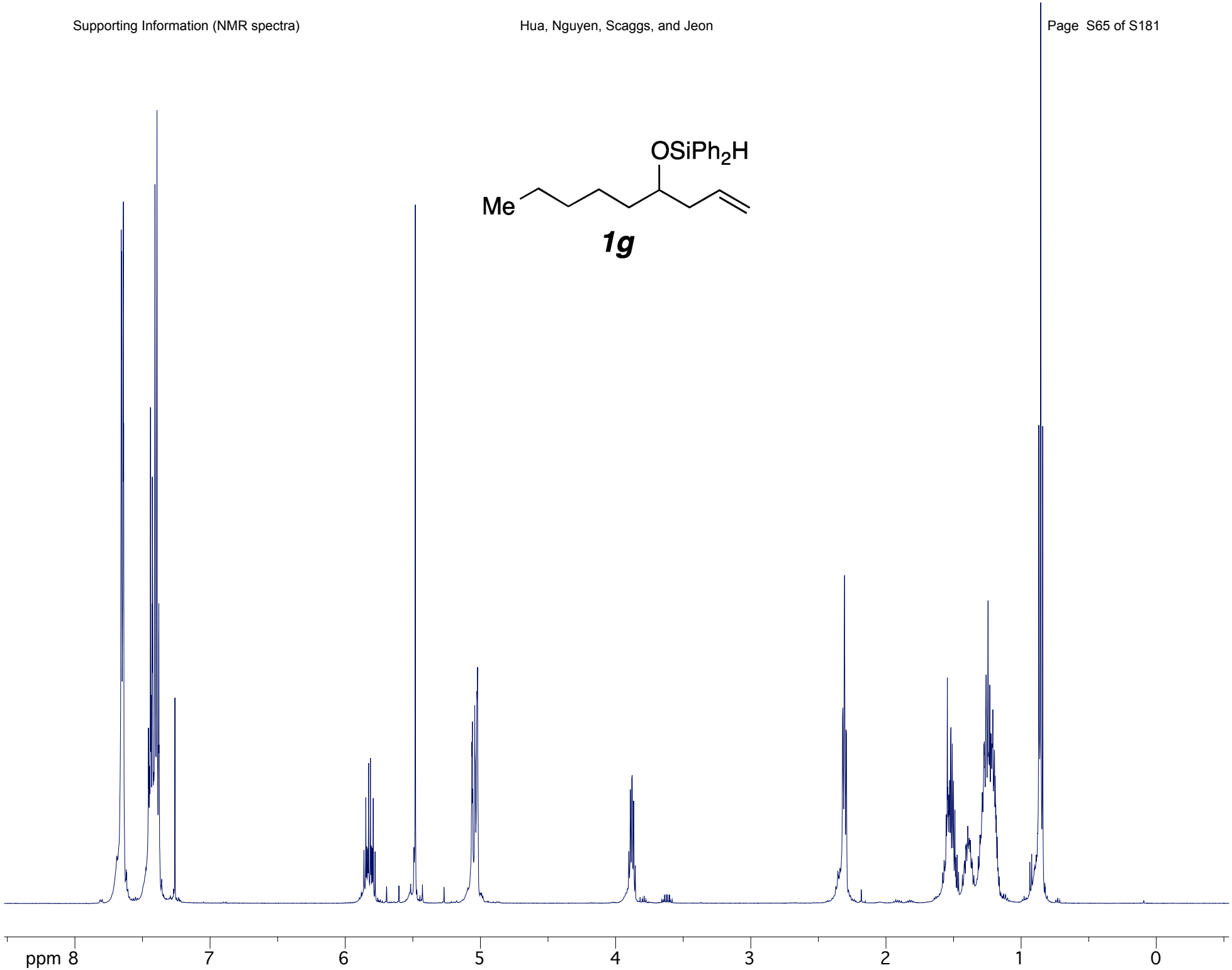

OSiPh2H
1f
ppm 8
7
6
5
4
3
2
1
0

1f

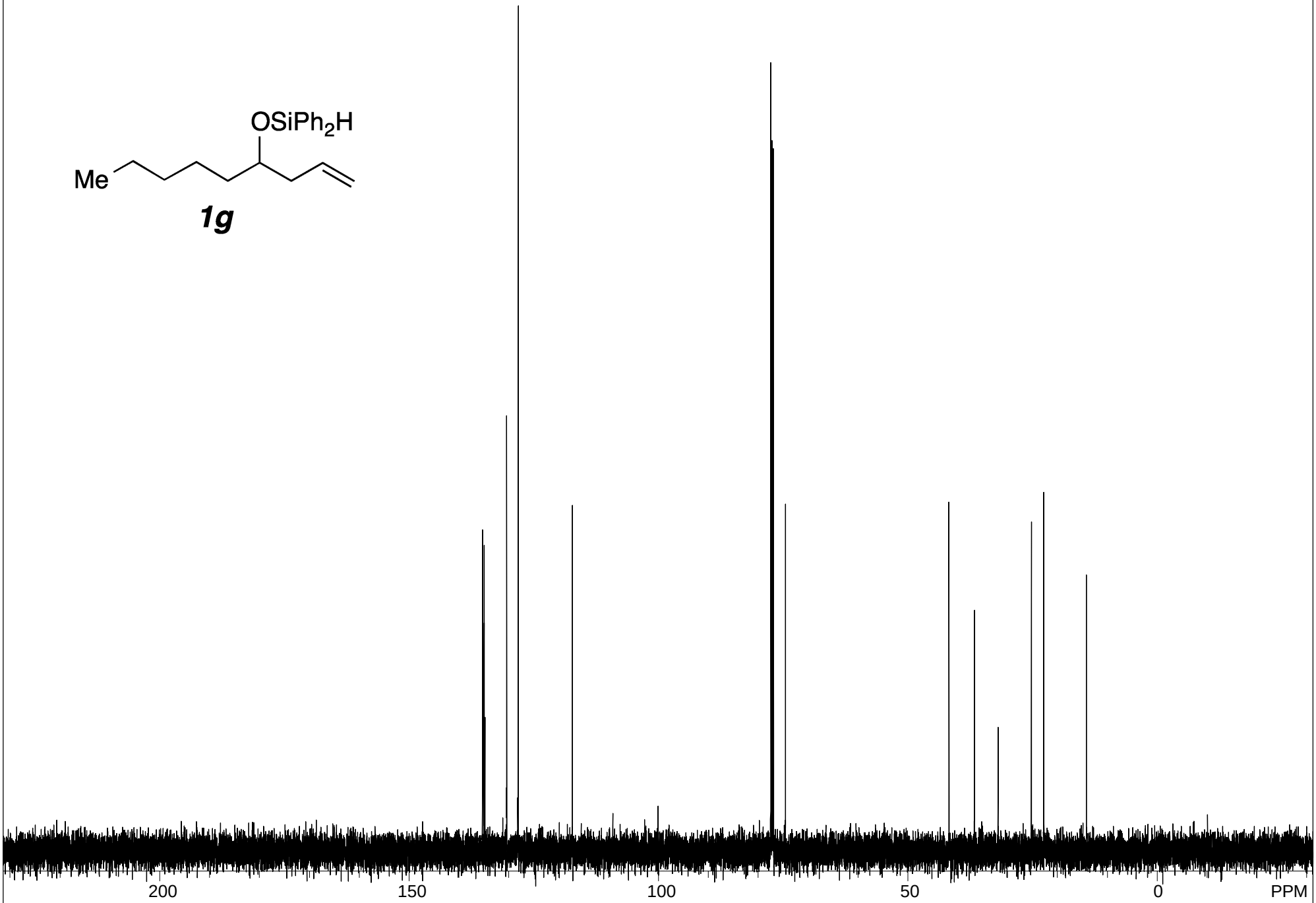


S#437643 (single pulse decoupled gated NOE) USER: delta -- DATE: Feb 15, 2013 02:35:24

F1: 125.778	F2: 500.162	SW1: 39308		OF1: 12587.8		PTS1d: 32768 , 32768
EX: single_pulse_dec		PW: 3.6 us, 30deg	PD: 2.0 sec	NA: 36	LB: 0.0	Nuts - YH_silyether_Allyl_M1_13C-1_1f.jdf


OSiPh2H
Me
1g
ppm 8
7
6
5
4
3
2
1
0

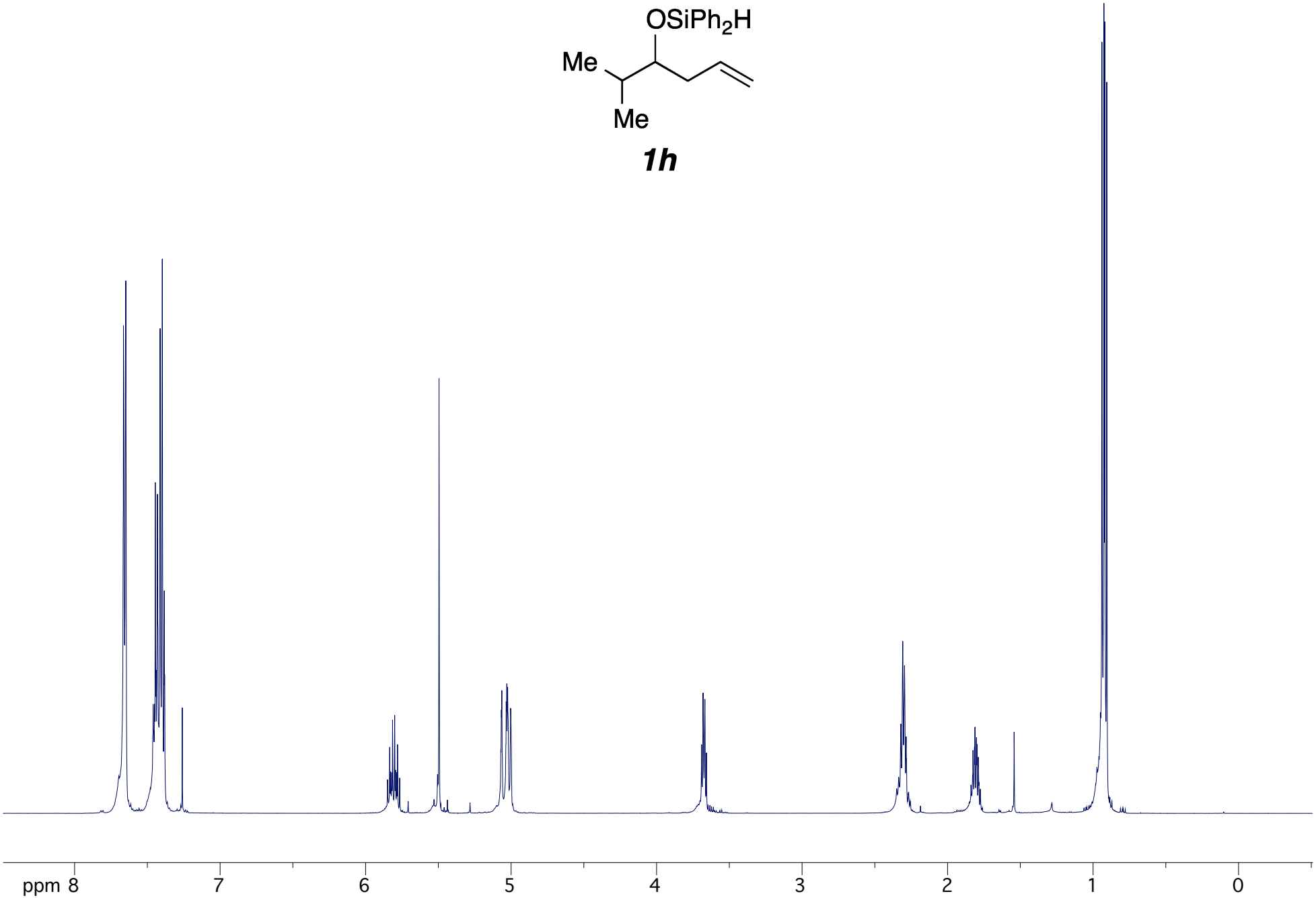
1g



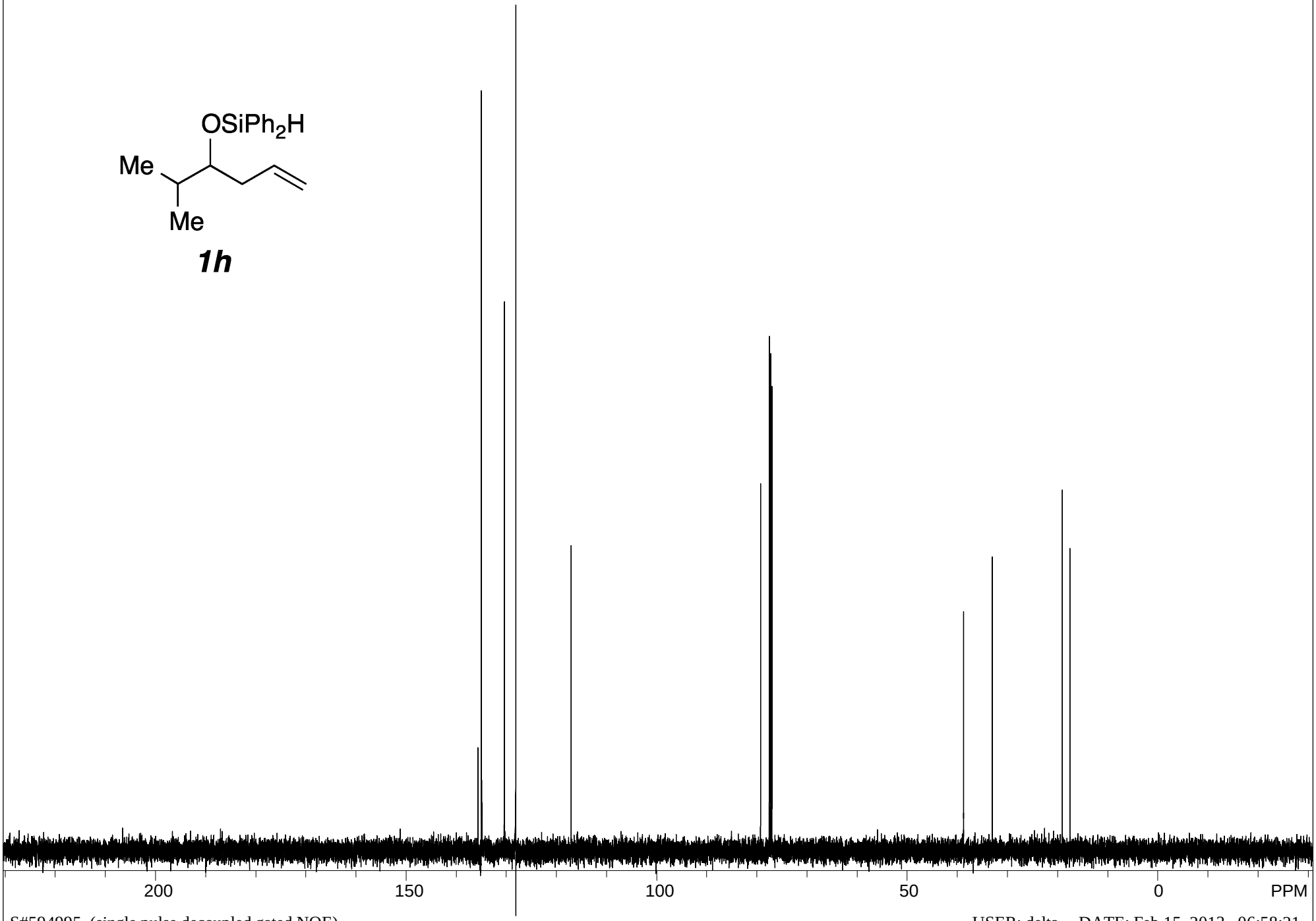
S#590911 (single pulse decoupled gated NOE) USER: delta -- DATE: Feb 15, 2013 06:51:22

F1: 125.778	F2: 500.162	SW1: 39308		OF1: 12591.4		PTS1d: 32768 , 32768
EX: single_pulse_dec		PW: 3.6 us, 30deg	PD: 2.0 sec	NA: 47	LB: 0.0	Nuts - YH_silyether_Pent_M1_13C-1_1g.jdf

1h



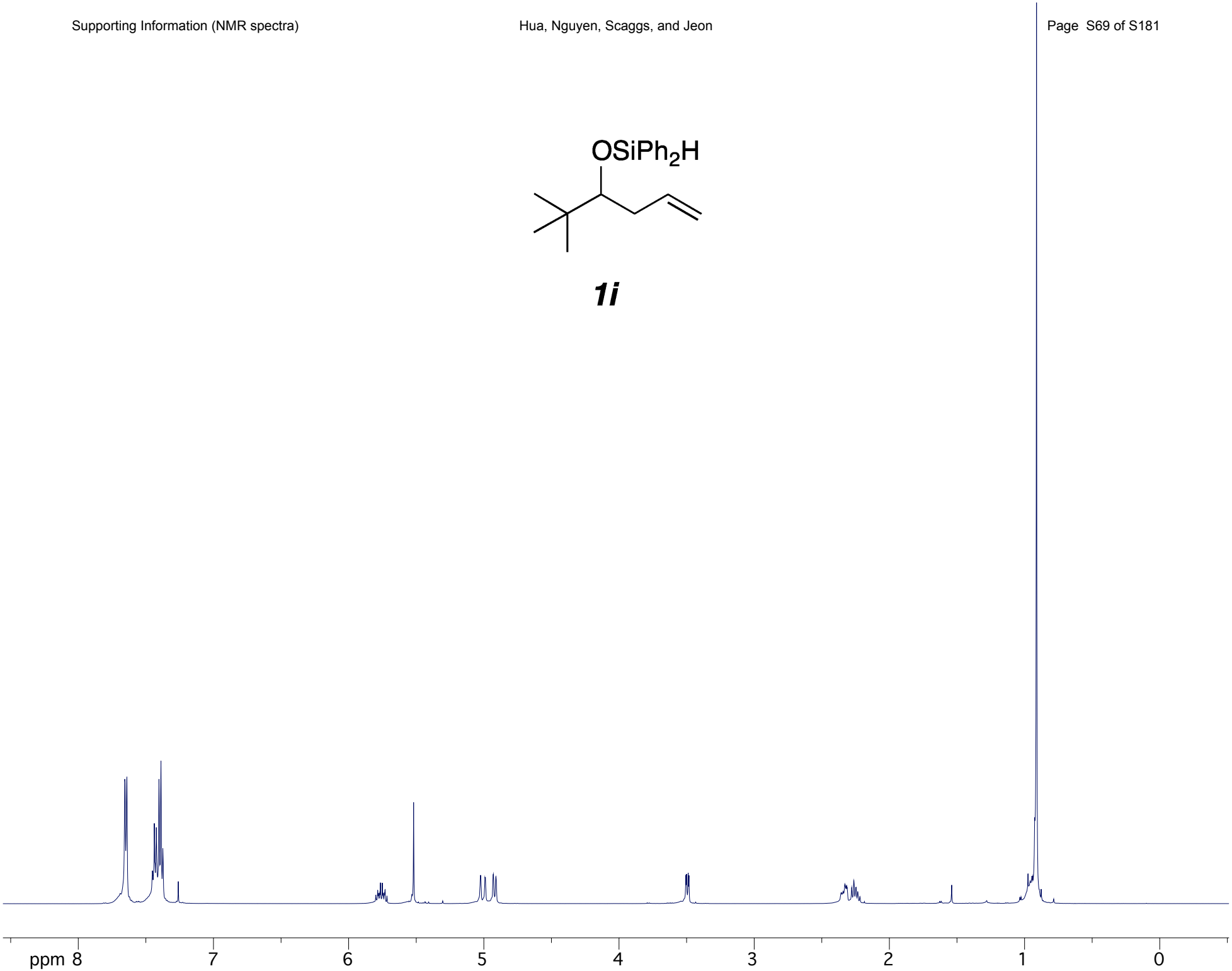
1h



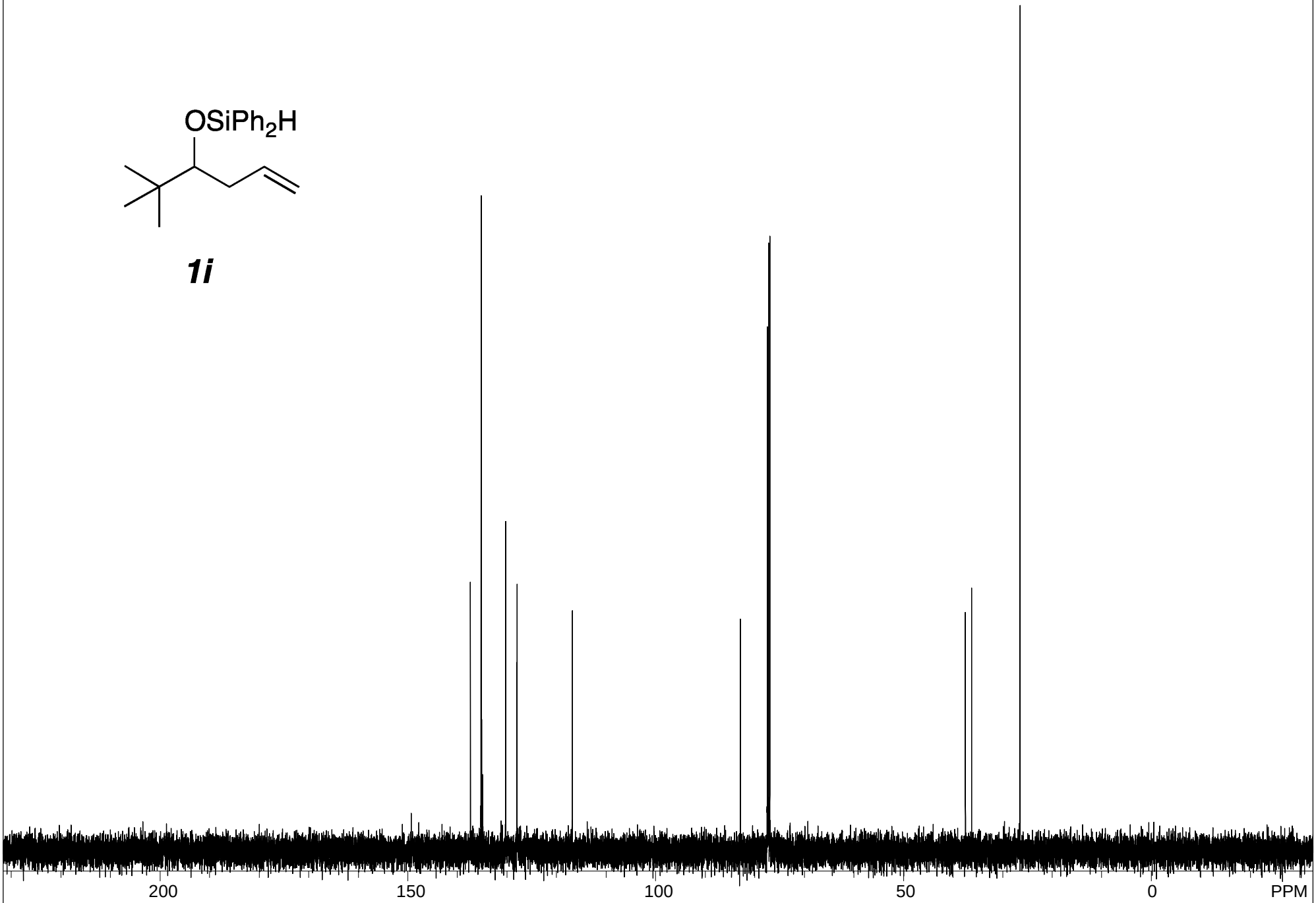
S#594995 (single pulse decoupled gated NOE) USER: delta -- DATE: Feb 15, 2013 06:58:21

F1: 125.778	F2: 500.162	SW1: 39308		OF1: 12590.2		PTS1d: 32768 , 32768
EX: single_pulse_dec		PW: 3.6 us, 30deg	PD: 2.0 sec	NA: 51	LB: 0.0	Nuts - YH_silyether_iPr_M1_13C-1_1g.jdf

1i

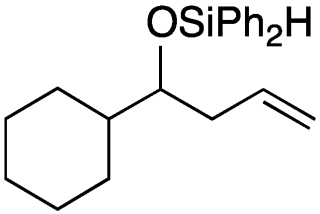


1i

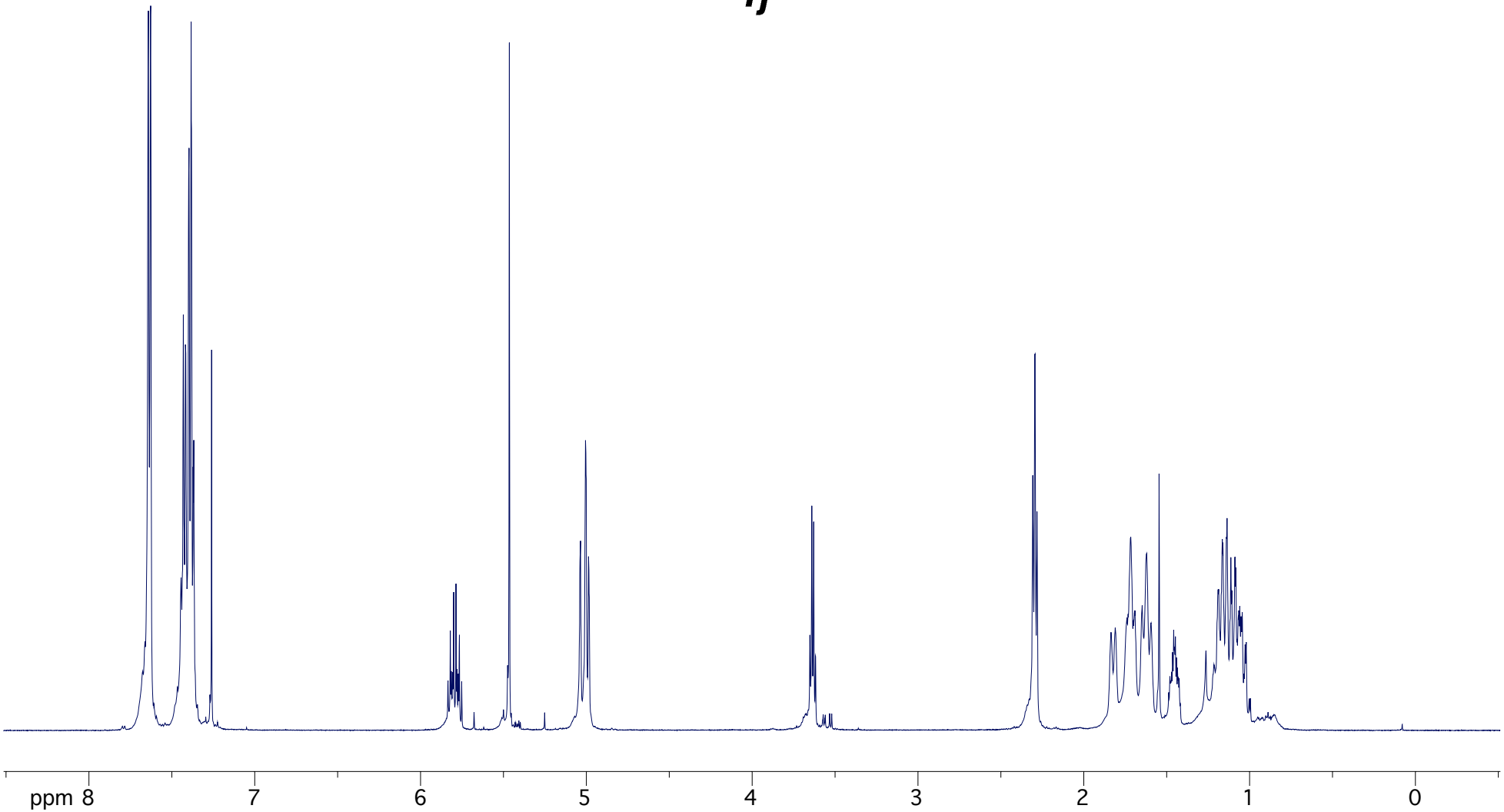


S#599236 (single pulse decoupled gated NOE) USER: delta -- DATE: Feb 15, 2013 07:05:06:

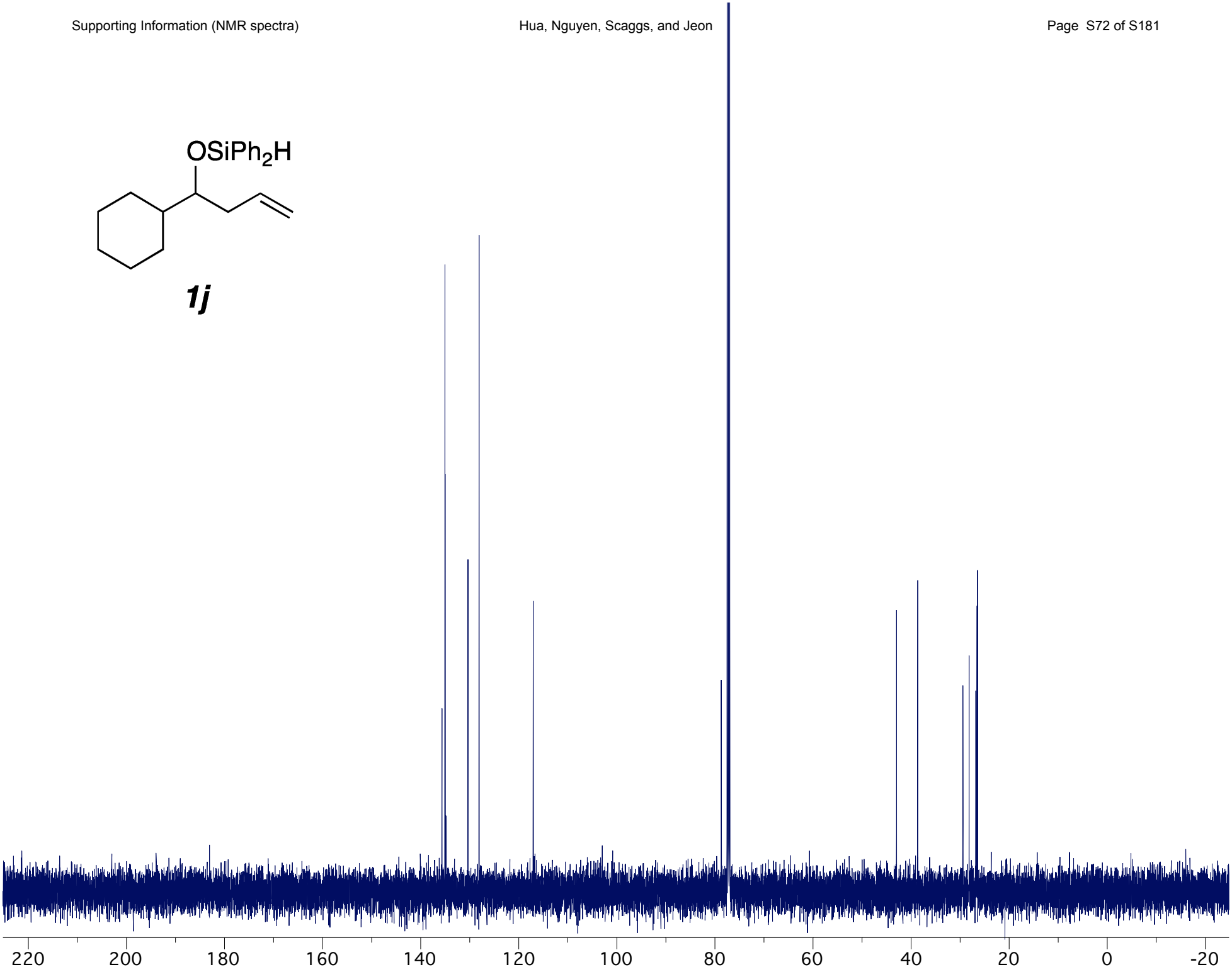
F1: 125.778	F2: 500.162	SW1: 39308		OF1: 12590.2		PTS1d: 32768 , 32768
EX: single_pulse_dec		PW: 3.6 us, 30deg	PD: 2.0 sec	NA: 44	LB: 0.0	Nuts - YH_silyether_tBu_M1_13C-1_1i_.jdf

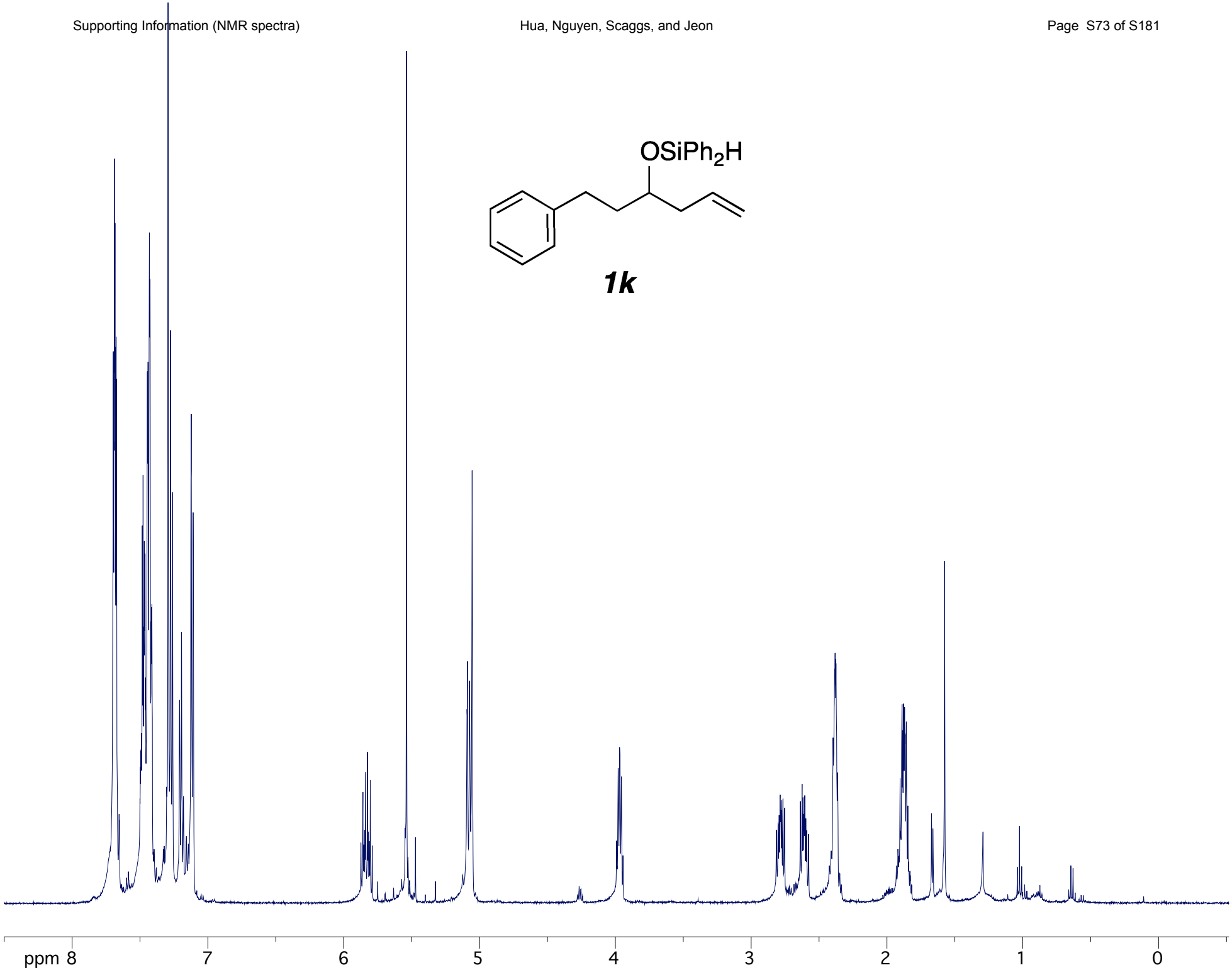


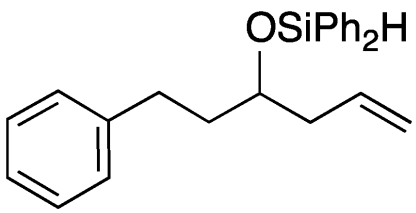
1j



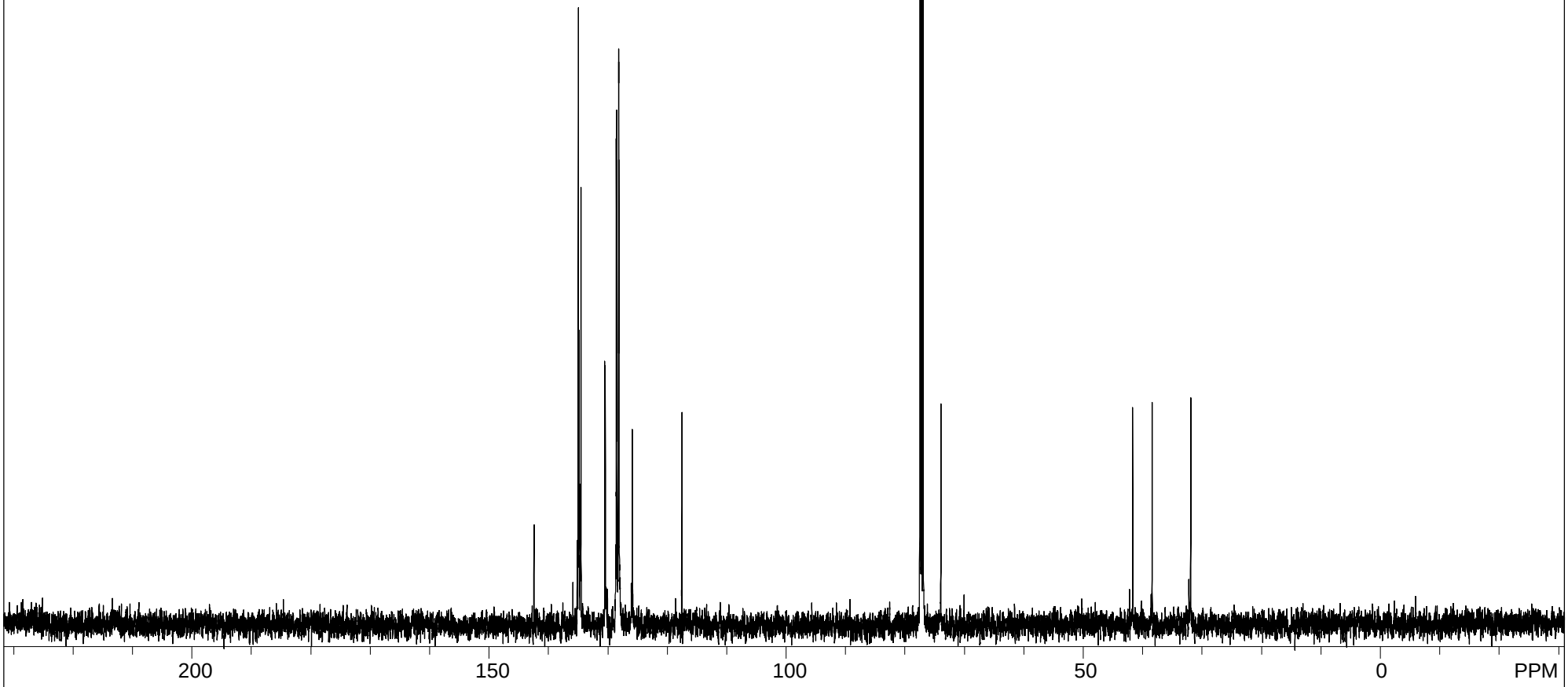
1j




OSiPh2H
1k
ppm 8
7
6
5
4
3
2
1
0



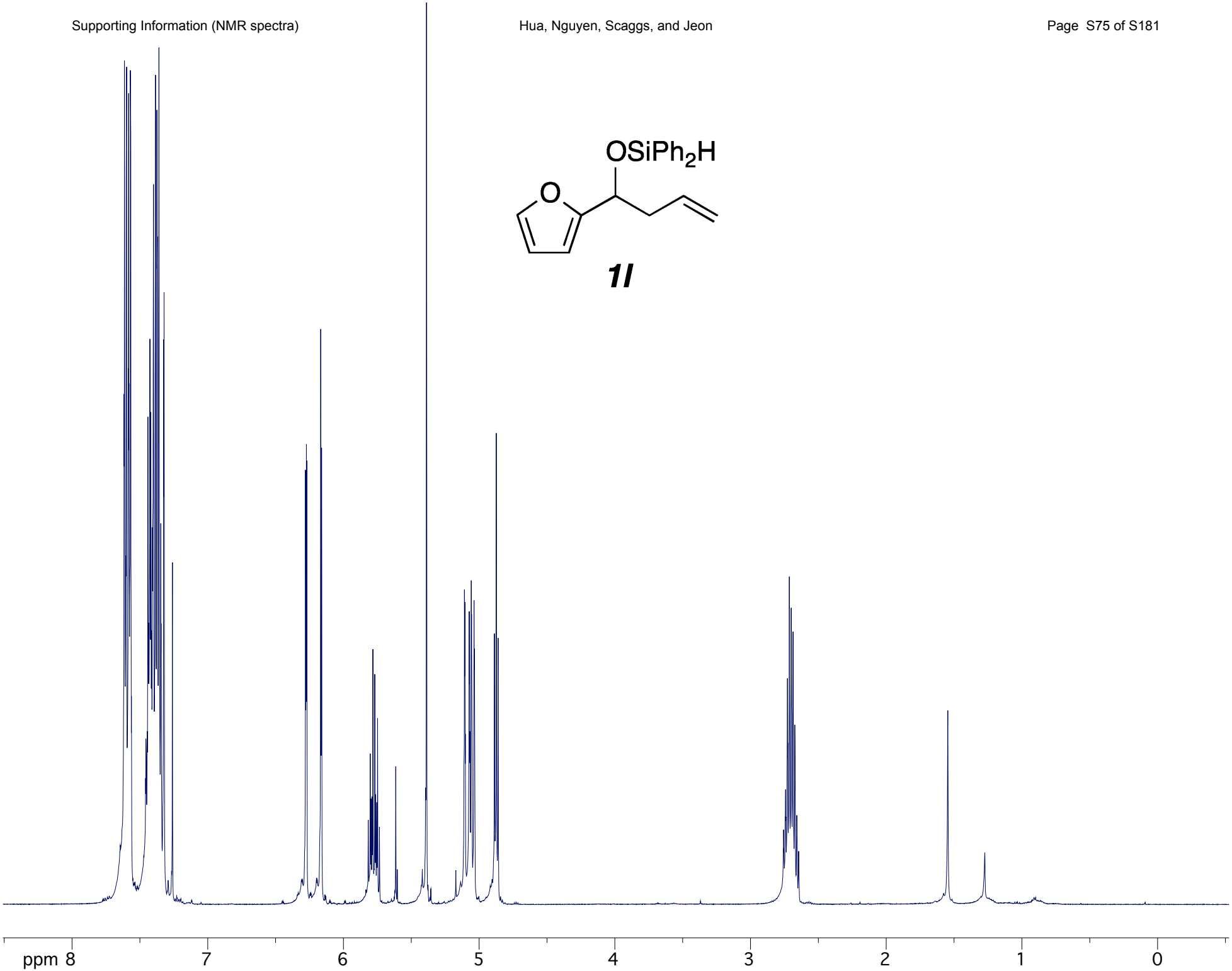
1k



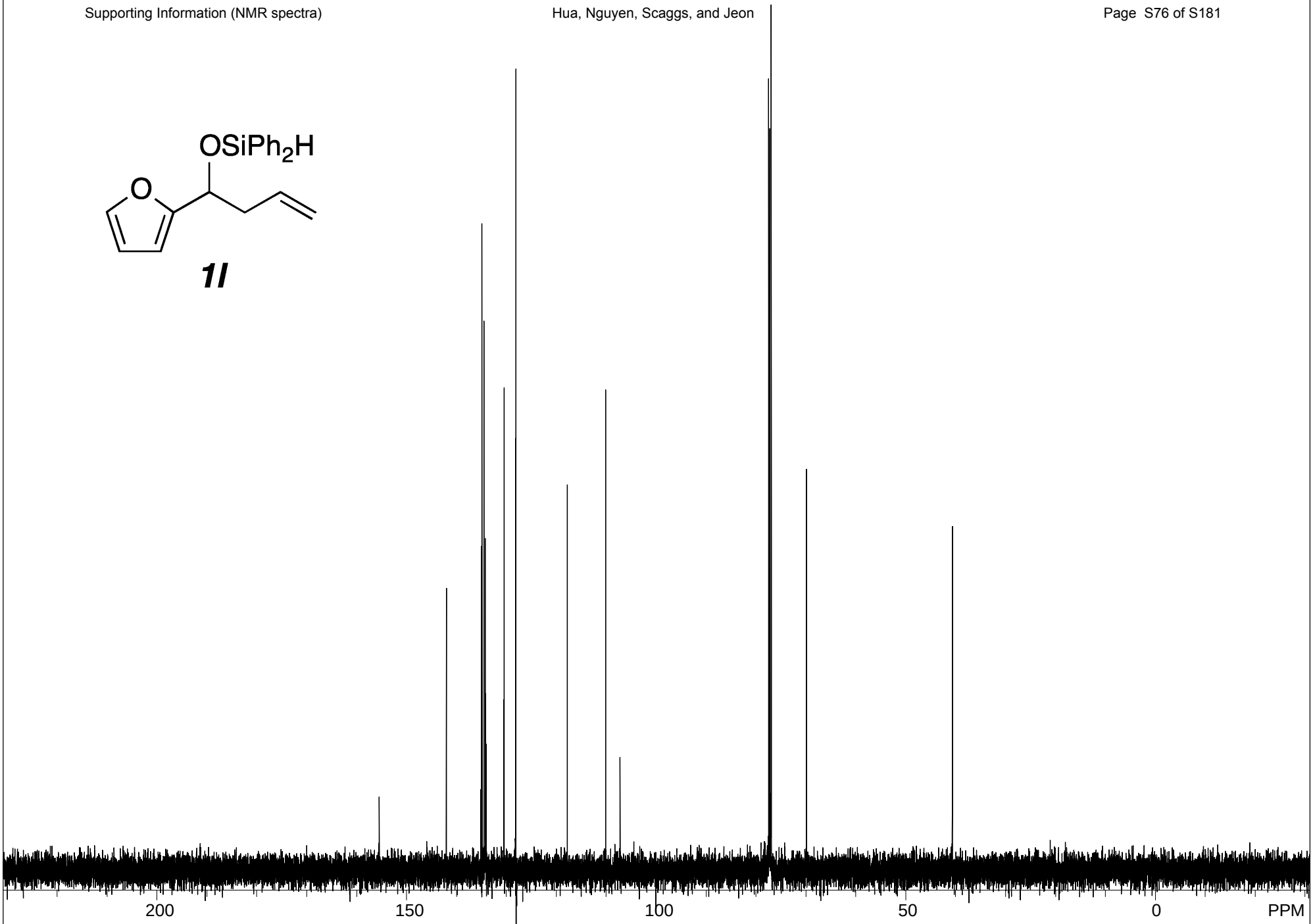
S#812073 (single pulse decoupled gated NOE) USER: delta -- DATE: Feb 8, 2013 13:02:43

F1: 125.778	F2: 500.162	SW1: 39308		OF1: 12587.8		PTS1d: 32768	
EX: single_pulse_dec		PW: 3.6 us, 30deg	PD: 2.0 sec	NA: 106	LB: 0.0	Nuts - $YH_silyether_HomoBenz_1k_13C-2.jdf	

1l



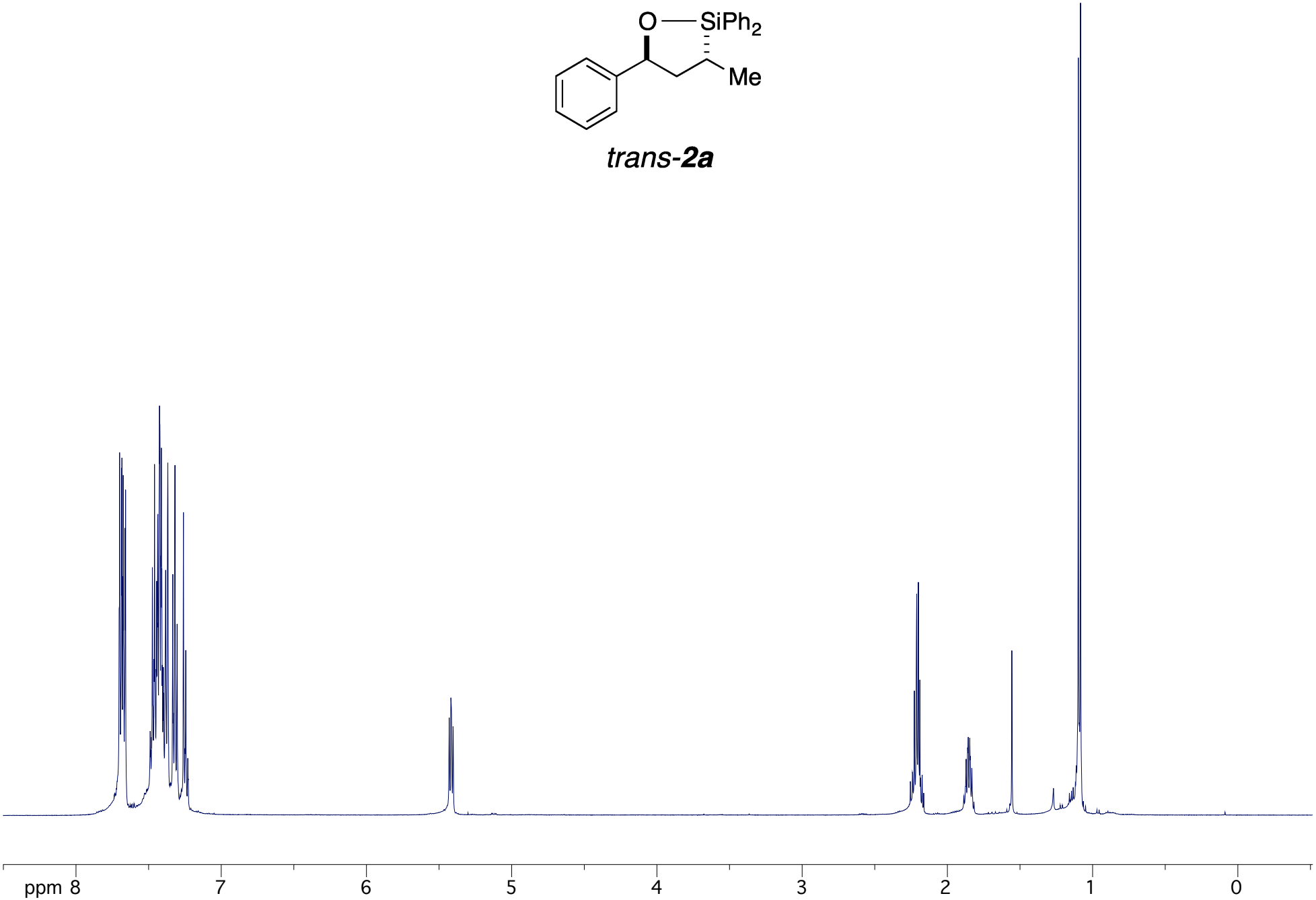
1l



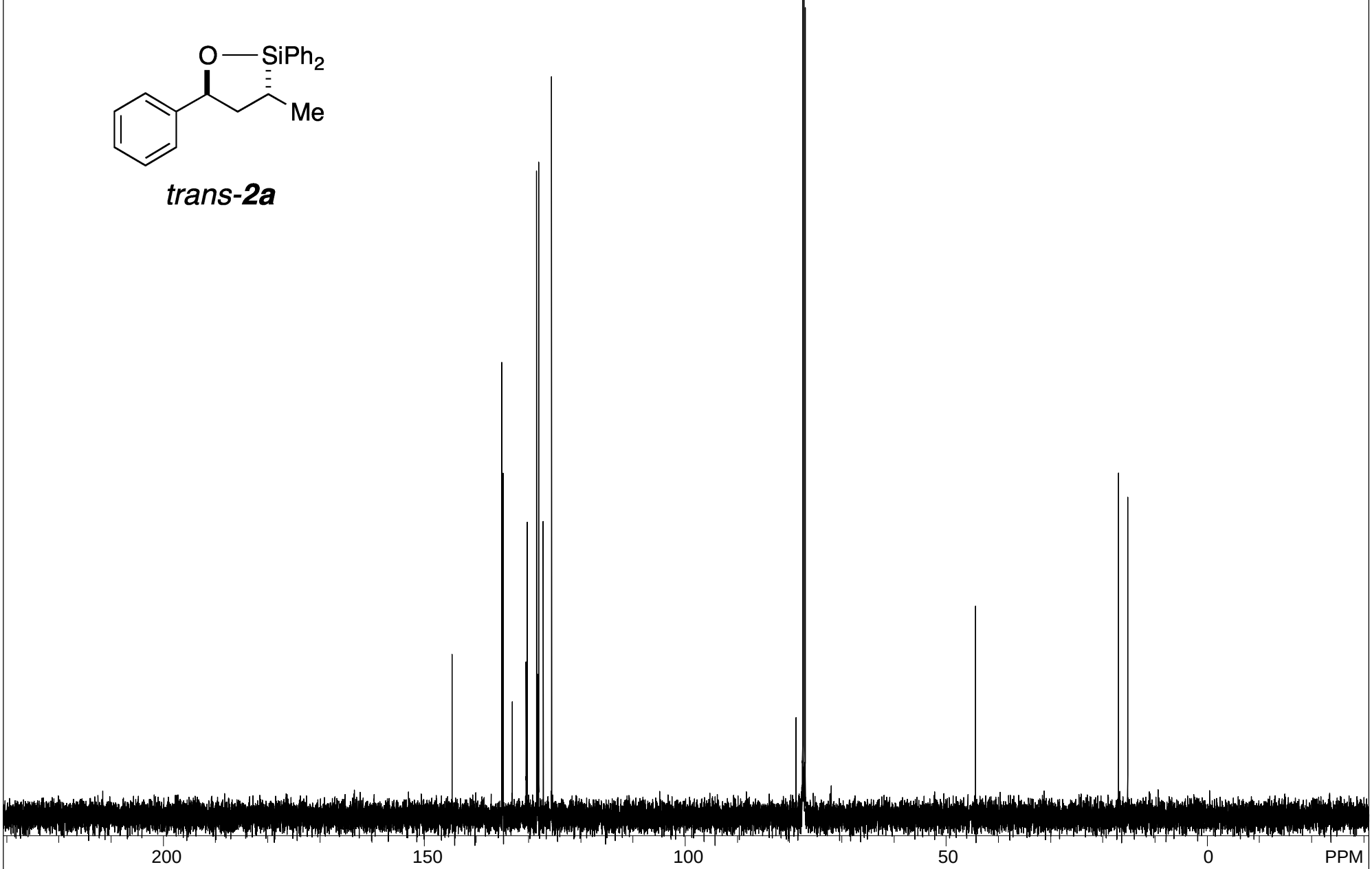
S#615539 (single pulse decoupled gated NOE) USER: delta -- DATE: Feb 15, 2013 07:34:02:

F1: 125.778	F2: 500.162	SW1: 39308		OF1: 12589.0		PTS1d: 32768 , 32768
EX: single_pulse_dec		PW: 3.6 us, 30deg	PD: 2.0 sec	NA: 82	LB: 0.0	Nuts - YH_silyether_Furanyl_M1_13C-1_1l.jdf

*trans-**2a***



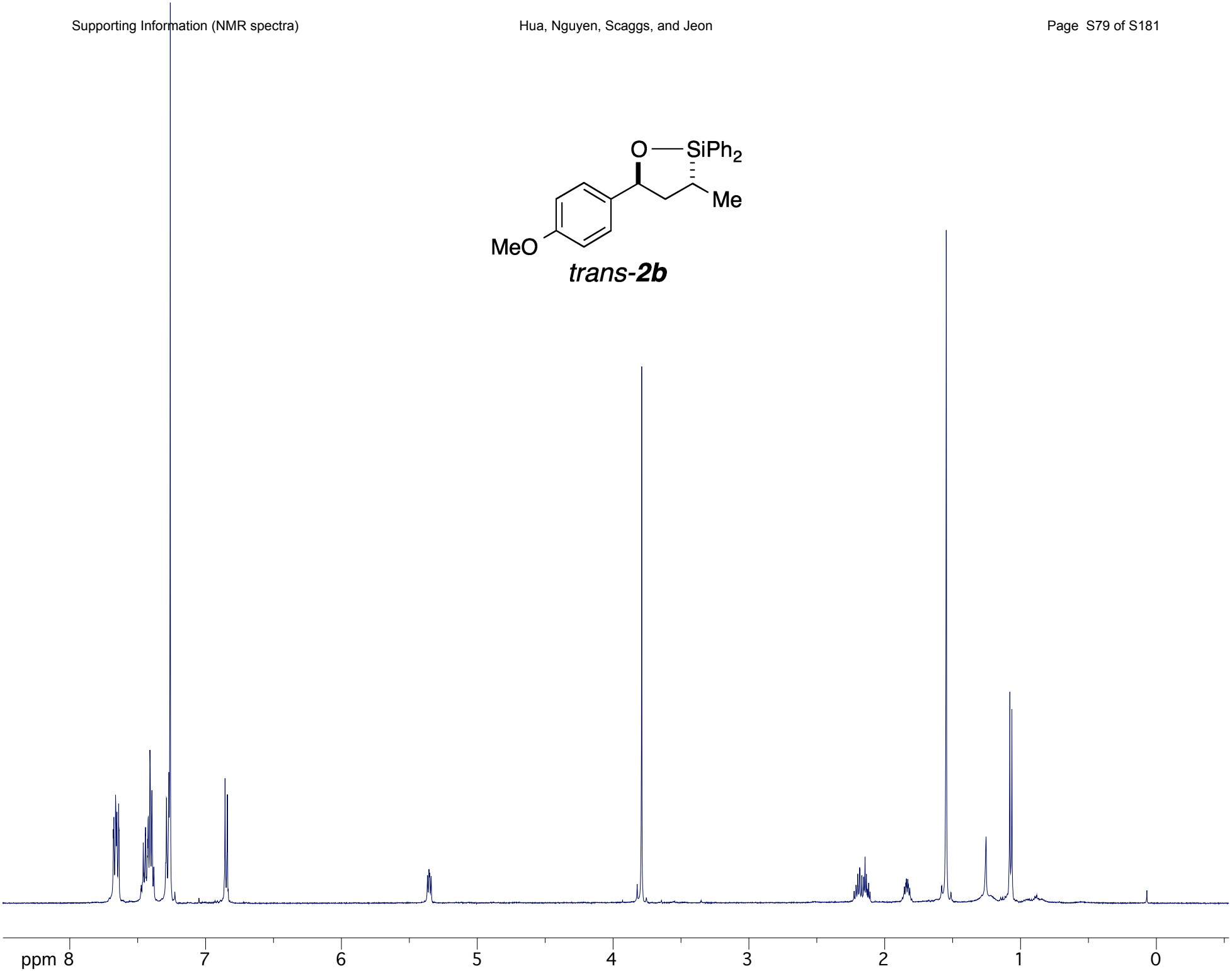
*trans-**2a***

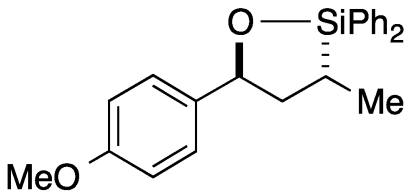


S#426037 (single pulse decoupled gated NOE) USER: delta -- DATE: Feb 22, 2013 02:22:17

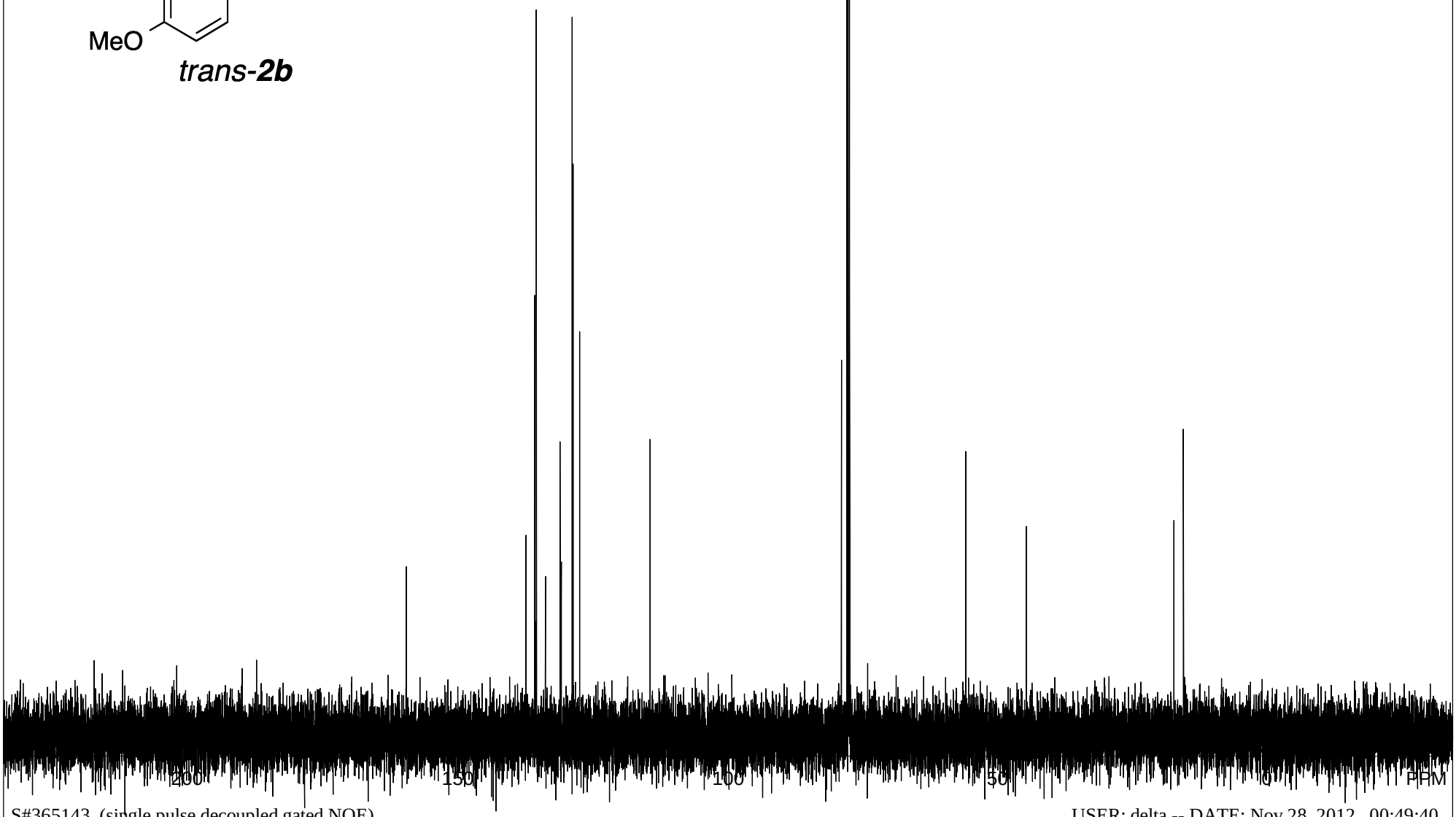
F1: 125.778	F2: 500.162	SW1: 39308		OF1: 12591.4		PTS1d: 32768 , 32768
EX: single_pulse_dec		PW: 3.6 us, 30deg	PD: 2.0 sec	NA: 166	LB: 0.0	Nuts - YH2078_racBinap_M1_3_13C-1_2a.jdf

*trans-**2b***





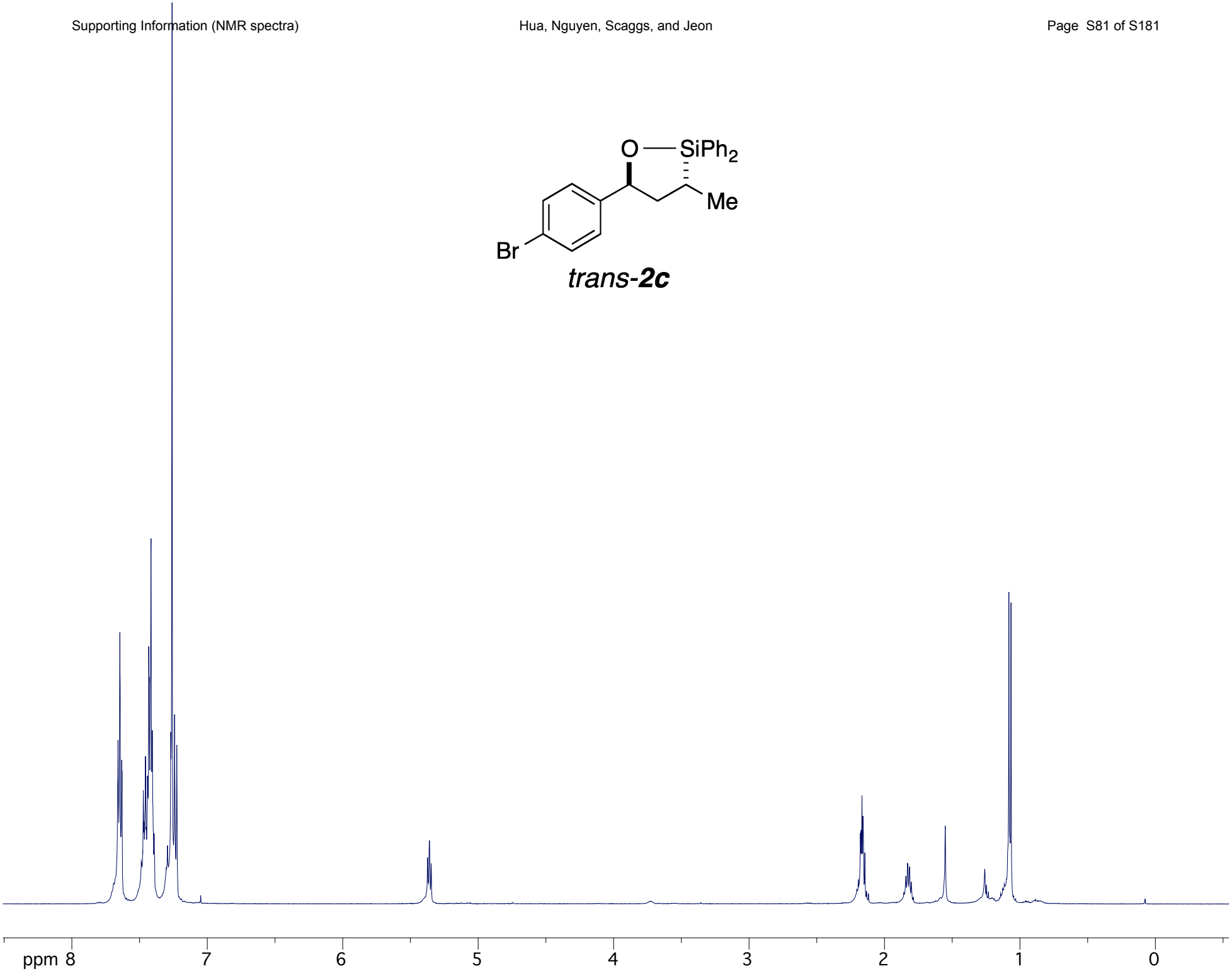
*trans-**2b***



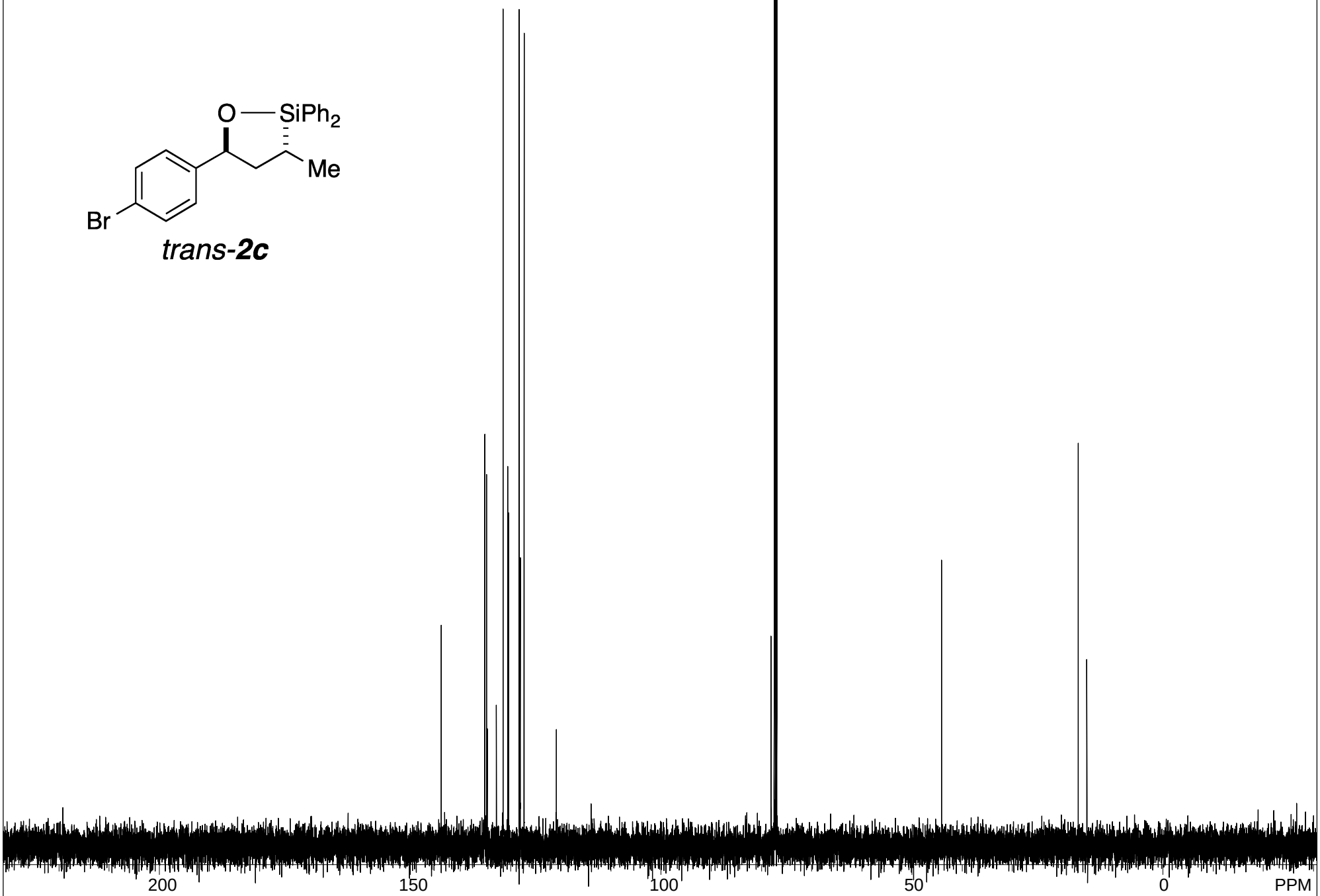
S#365143 (single pulse decoupled gated NOE) USER: delta -- DATE: Nov 28, 2012 00:49:40

F1: 125.778	F2: 500.162	SW1: 39308		OF1: 12592.6		PTS1d: 32768 , 32768
EX: single_pulse_dec		PW: 3.6 us, 30deg	PD: 2.0 sec	NA: 227	LB: 0.0	Nuts - YH2021_A_MPLC_r3_C-1-2b.jdf

*trans-**2c***



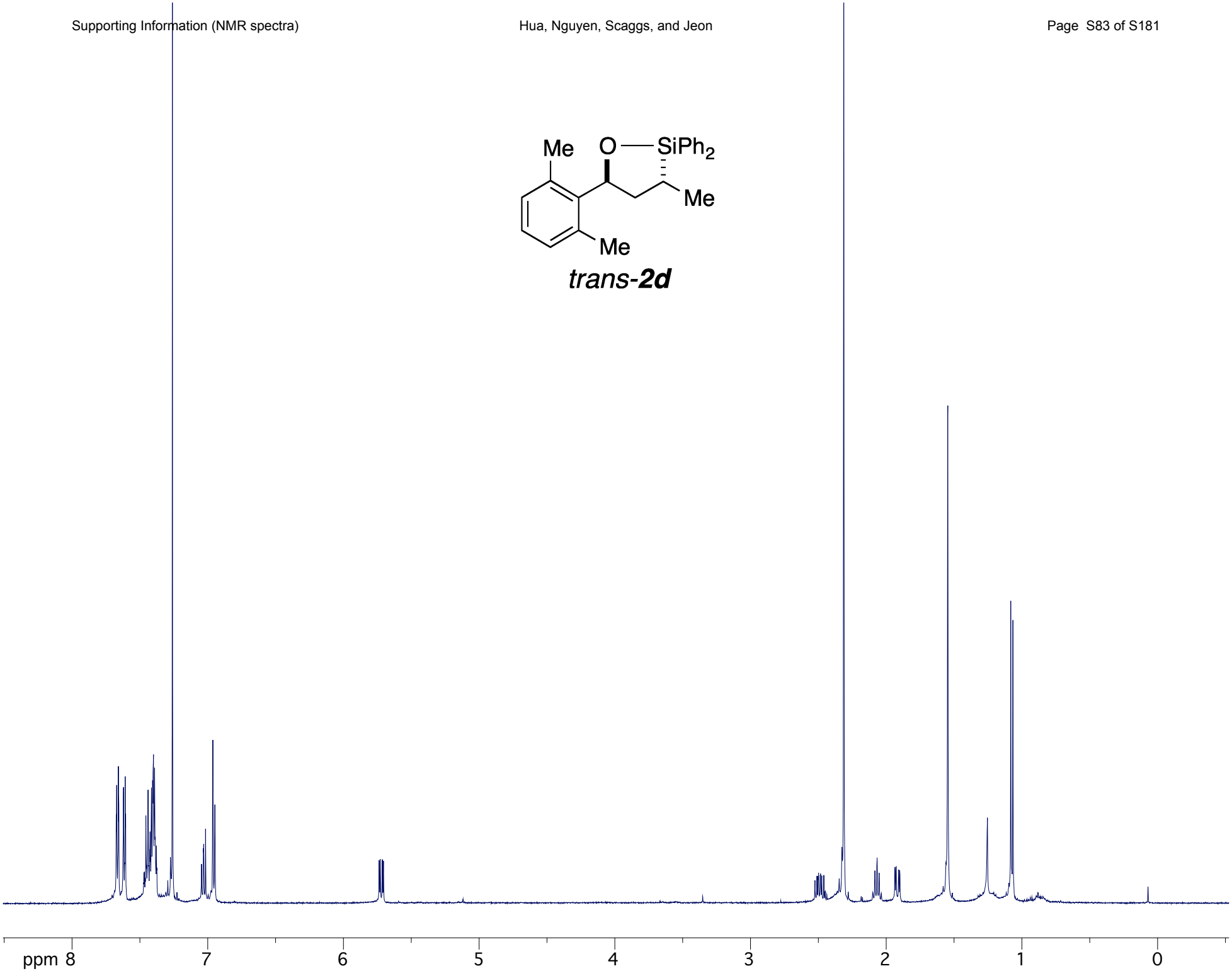
*trans-**2c***



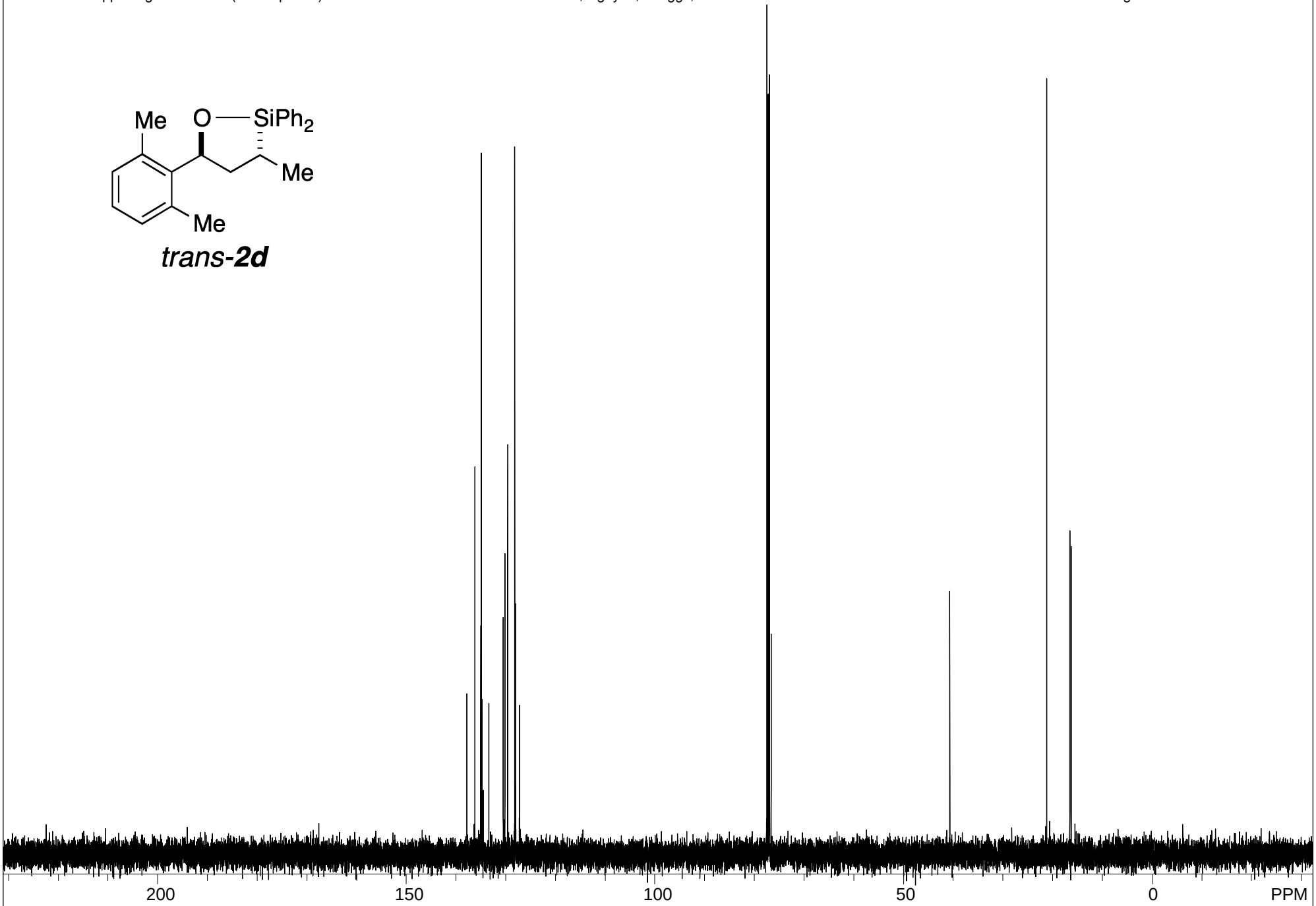
S#629252 (single pulse decoupled gated NOE) USER: delta -- DATE: Nov 27, 2012 08:02:03:

F1: 125.778	F2: 500.162	SW1: 39308		OF1: 12590.2		PTS1d: 32768 , 32768
EX: single_pulse_dec		PW: 3.6 us, 30deg	PD: 2.0 sec	NA: 62	LB: 0.0	Nuts - YH2021_B_MPLC_r1_4_C-1_2c.jdf

*trans-**2d***



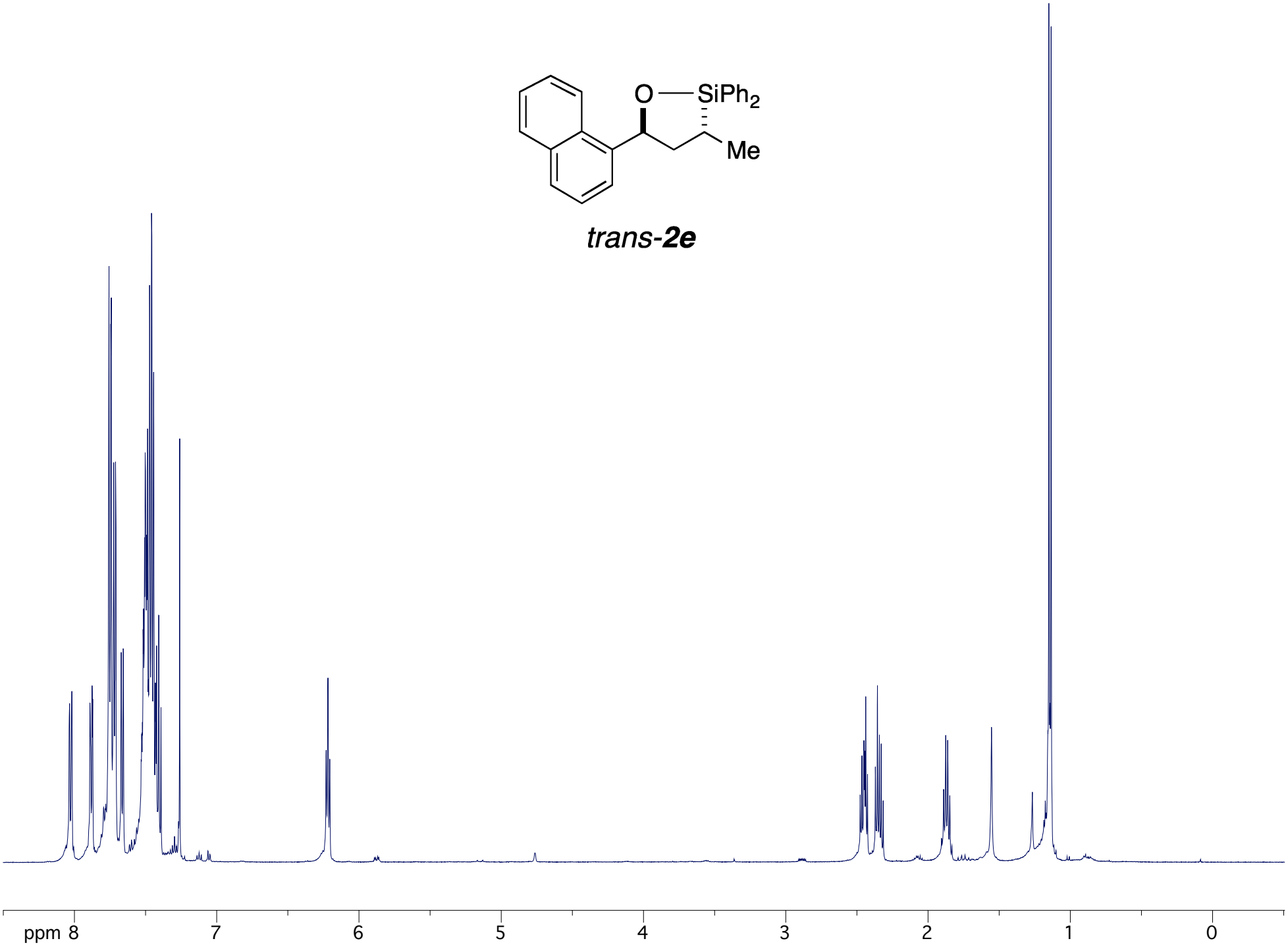
*trans-**2d***



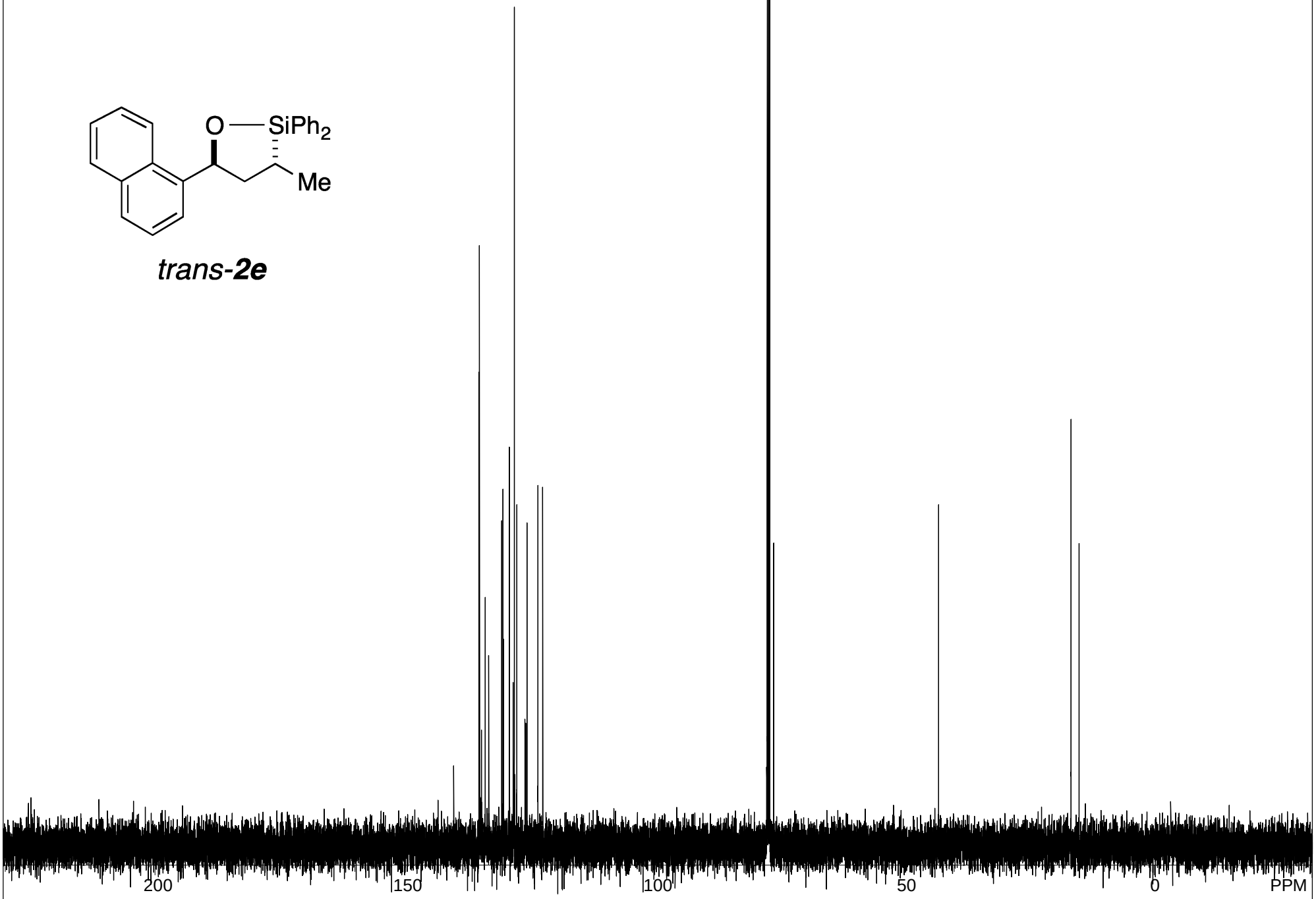
S#375636 (single pulse decoupled gated NOE) USER: delta -- DATE: Nov 28, 2012 00:59:06:

F1: 125.778	F2: 500.162	SW1: 39308		OF1: 12589.0		PTS1d: 32768 , 32768
EX: single_pulse_dec		PW: 3.6 us, 30deg	PD: 2.0 sec	NA: 57	LB: 0.0	Nuts - YH2021_C_MPLC_r1_3_C-1 2_2d.jdf

*trans-**2e***

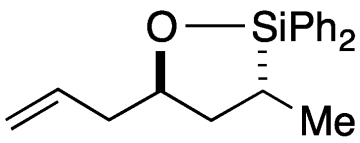


*trans-**2e***

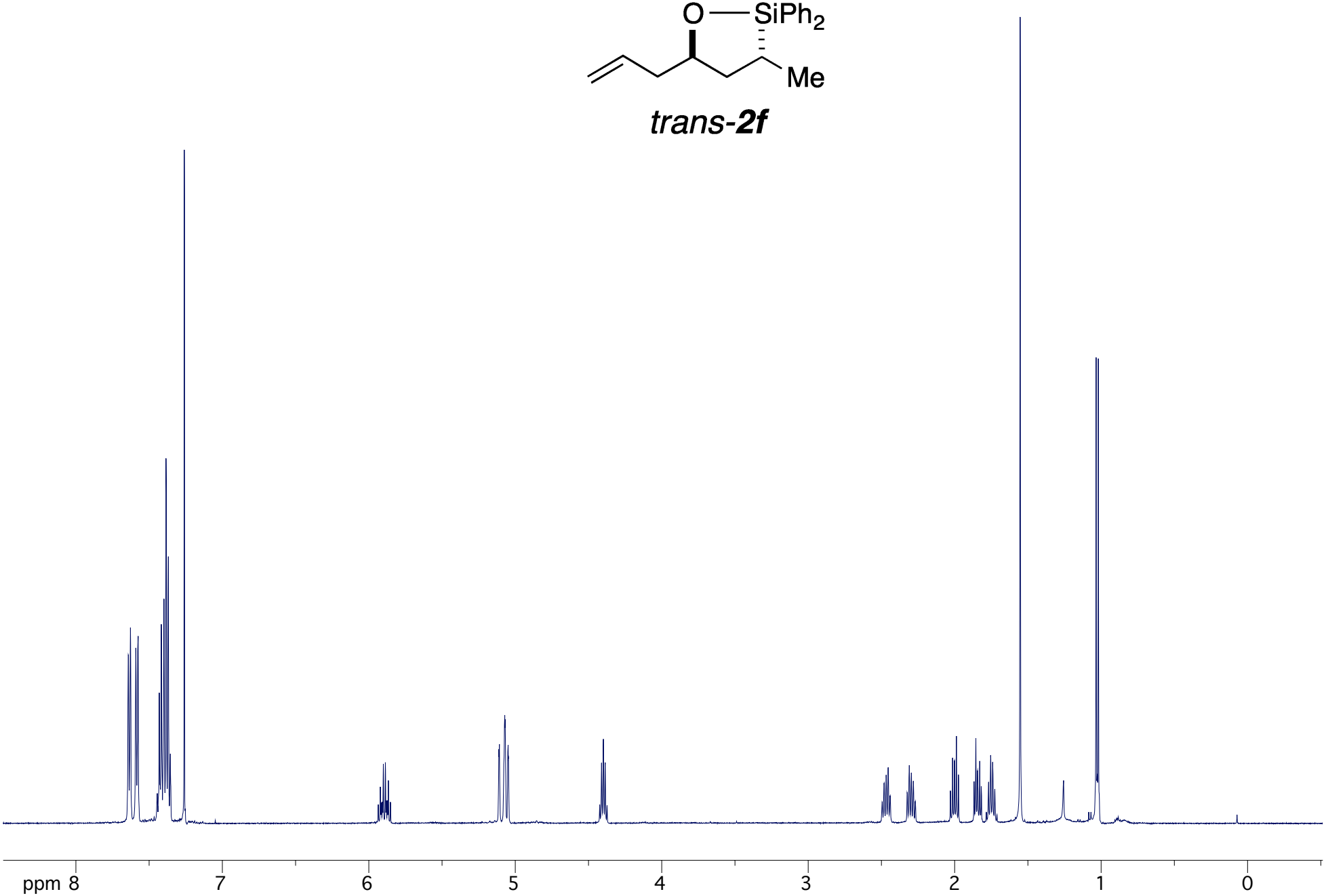


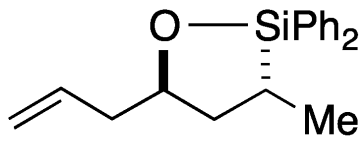
S#761368 (single pulse decoupled gated NOE) USER: delta -- DATE: Feb 15, 2013 11:39:50

F1: 125.778	F2: 500.162	SW1: 39308		OF1: 12590.2		PTS1d: 32768 , 32768
EX: single_pulse_dec		PW: 3.6 us, 30deg	PD: 2.0 sec	NA: 138	LB: 0.0	Nuts - YH1097_C_5ring_Napht_13C-1_2e.jdf

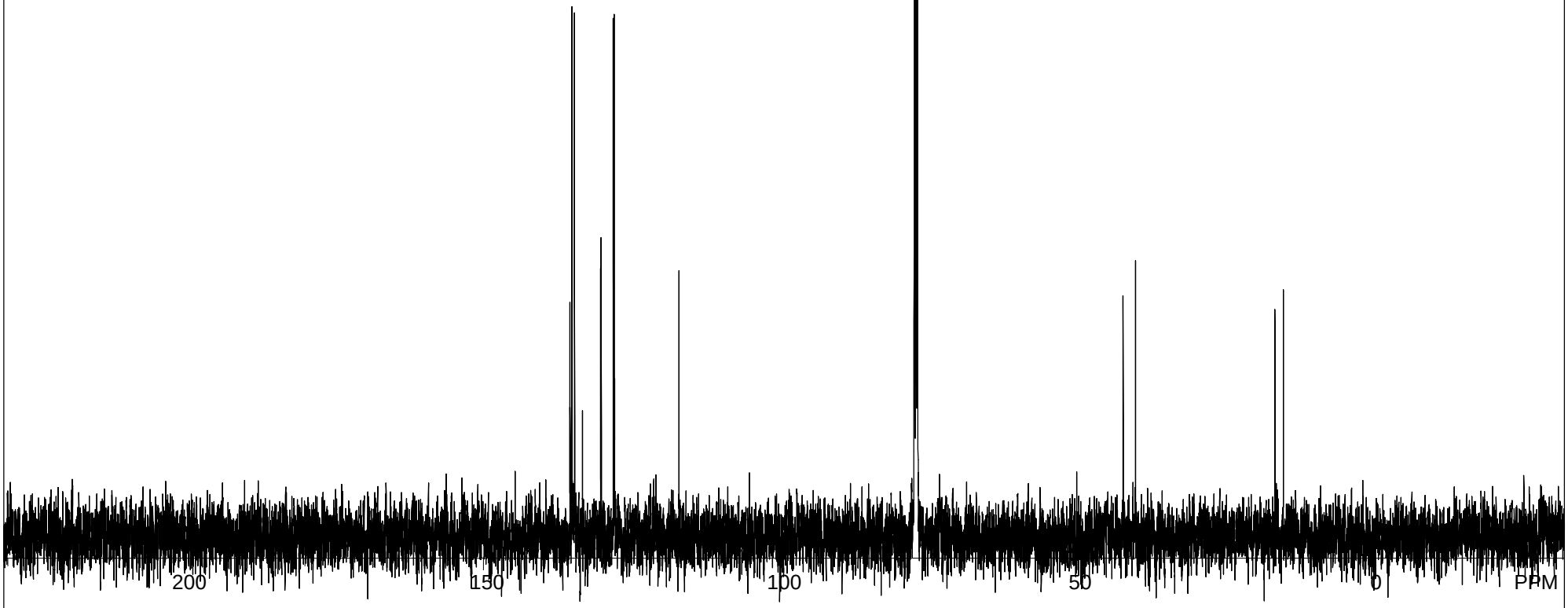


*trans-**2f***



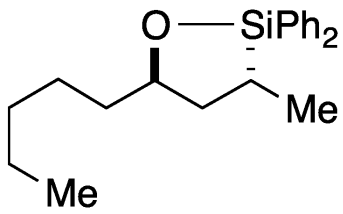


*trans-**2f***

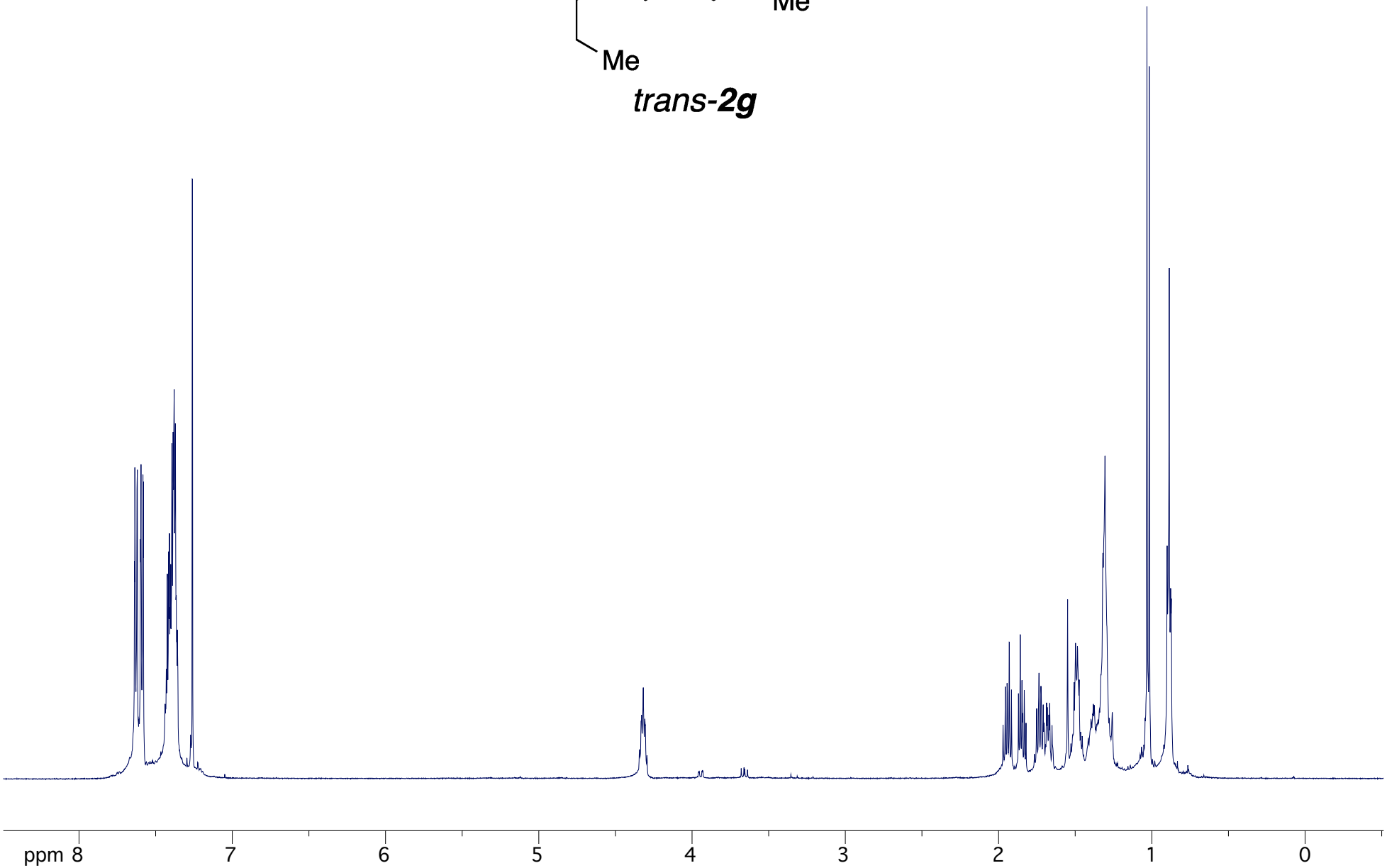


S#529681 (single pulse decoupled gated NOE) USER: delta -- DATE: May 29, 2012 04:37:23

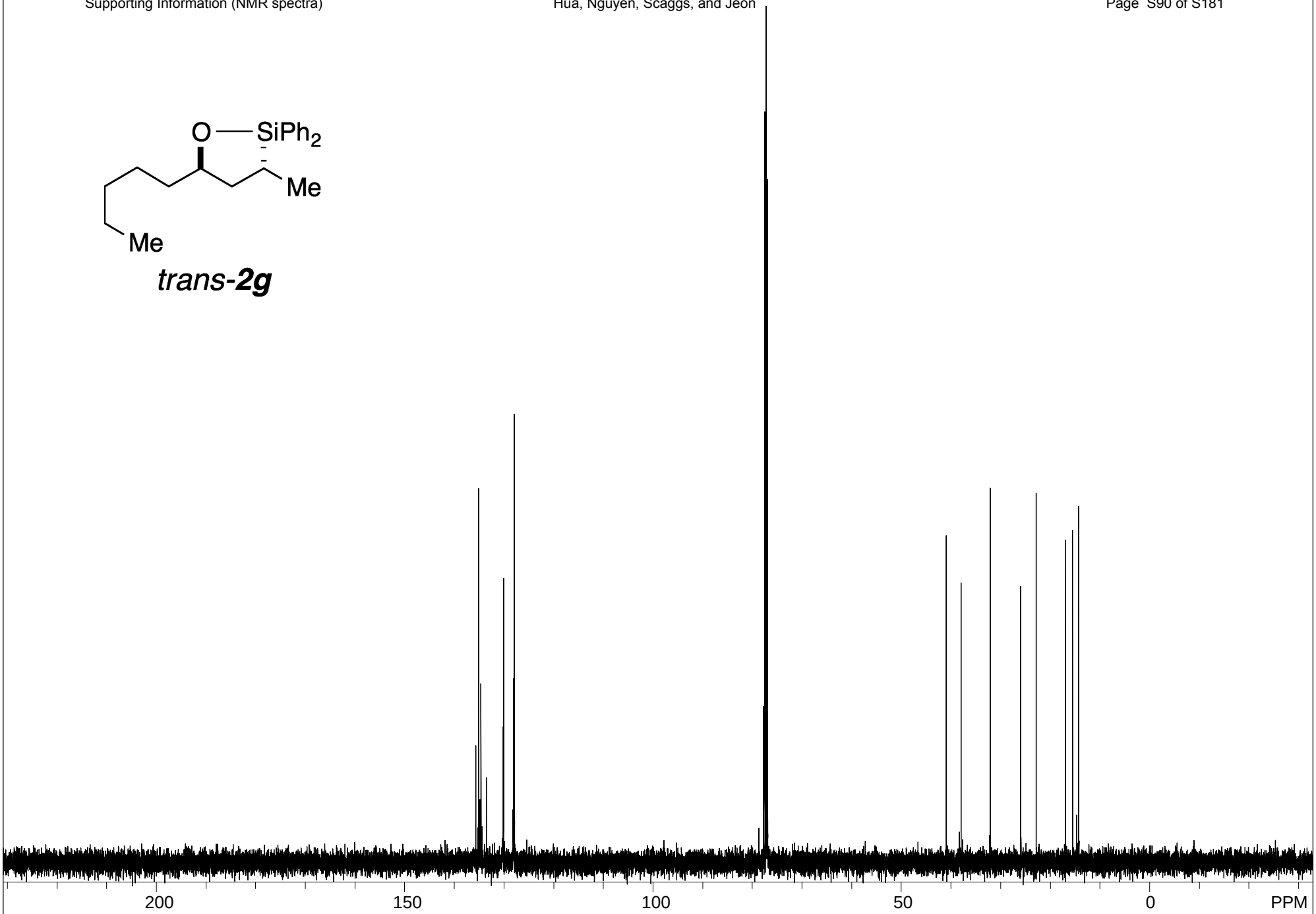
F1: 125.778	F2: 500.162	SW1: 39308		OF1: 12593.8		PTS1d: 32768	
EX: single_pulse_dec		PW: 3.6 us, 30deg	PD: 2.0 sec	NA: 110	LB: 0.0	Nuts - $HHN1035_13C_mplc10-2 2.jdf	



*trans-**2g***



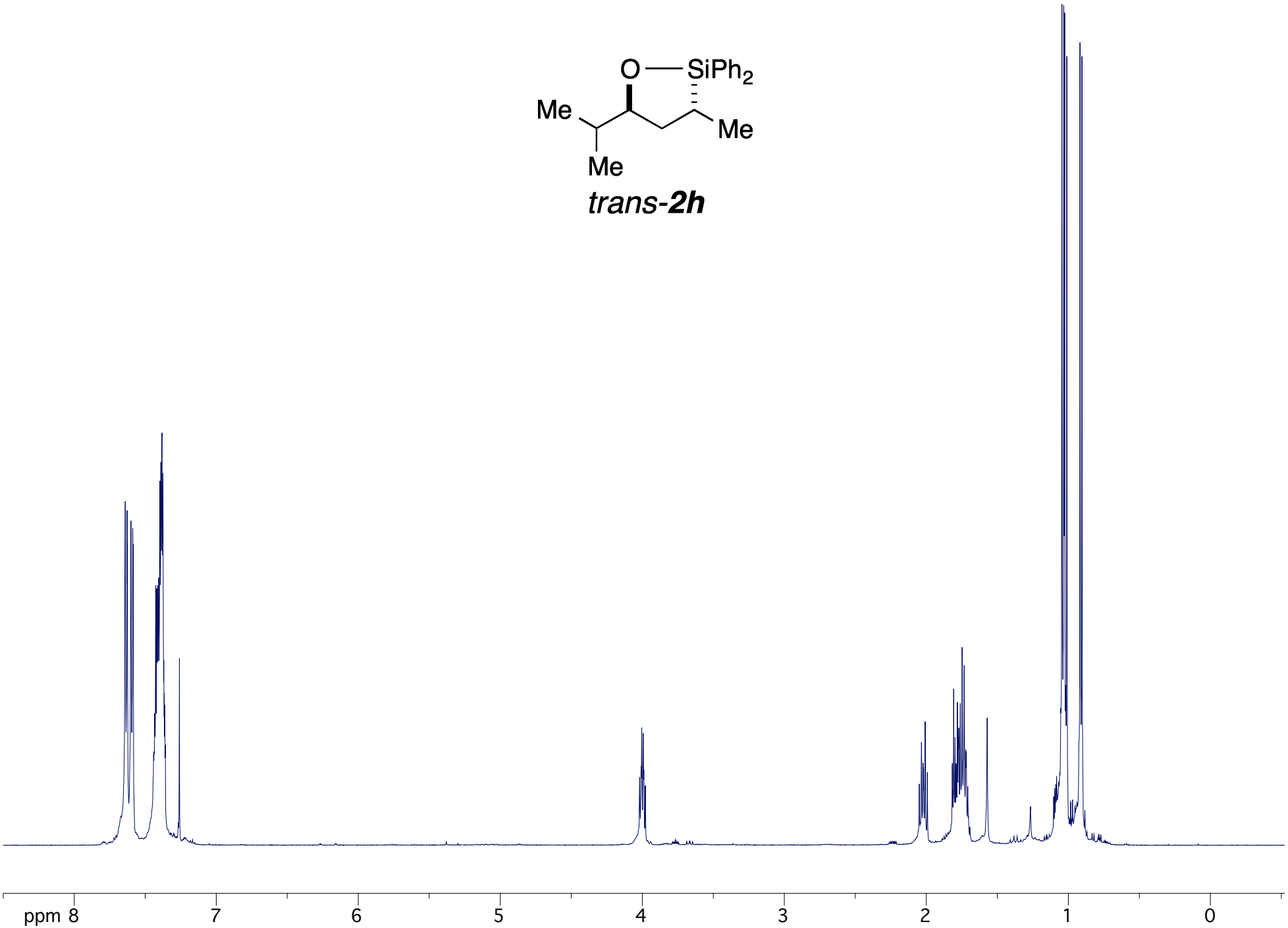
*trans-**2g***

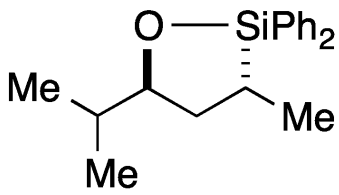


S#686785 (single pulse decoupled gated NOE) USER: delta -- DATE: Sep 6, 2012 09:02:32

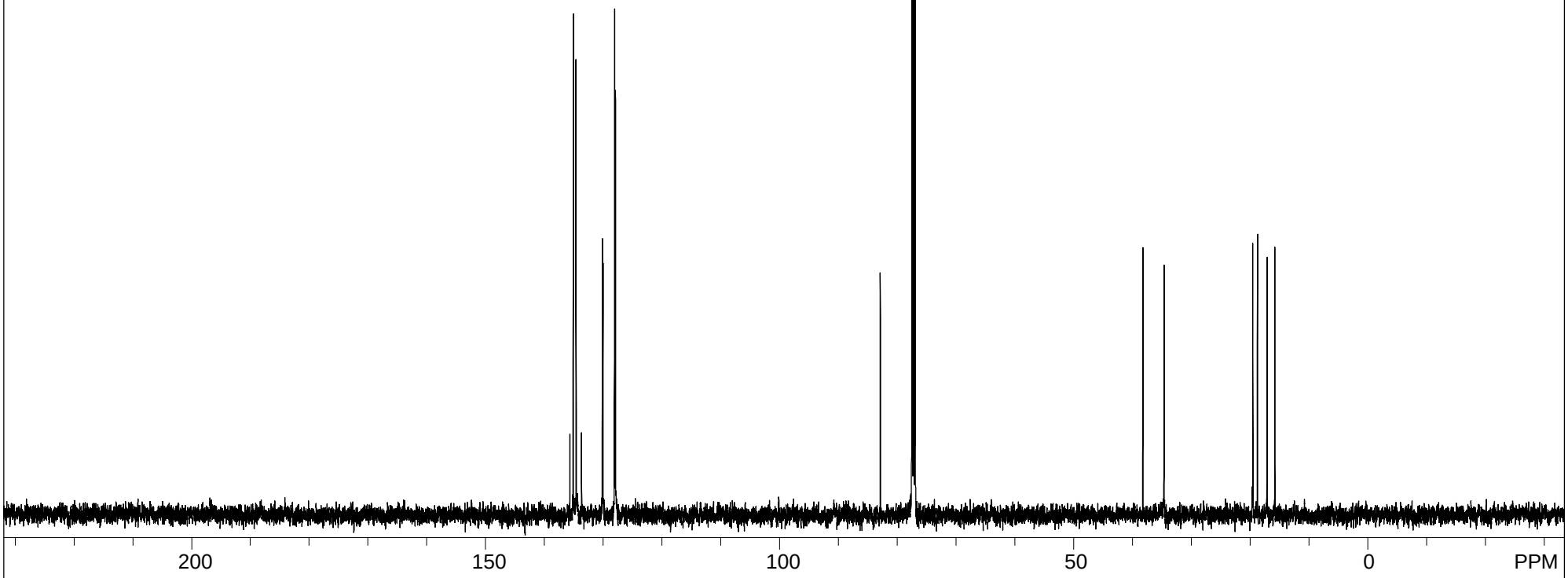
F1: 125.778	F2: 500.162	SW1: 39308		OF1: 12591.4		PTS1d: 32768 , 32768
EX: single_pulse_dec		PW: 3.6 us, 30deg	PD: 2.0 sec	NA: 120	LB: 0.0	Nuts - YH1021_D_4_C-1_2g.jdf

*trans-**2h***





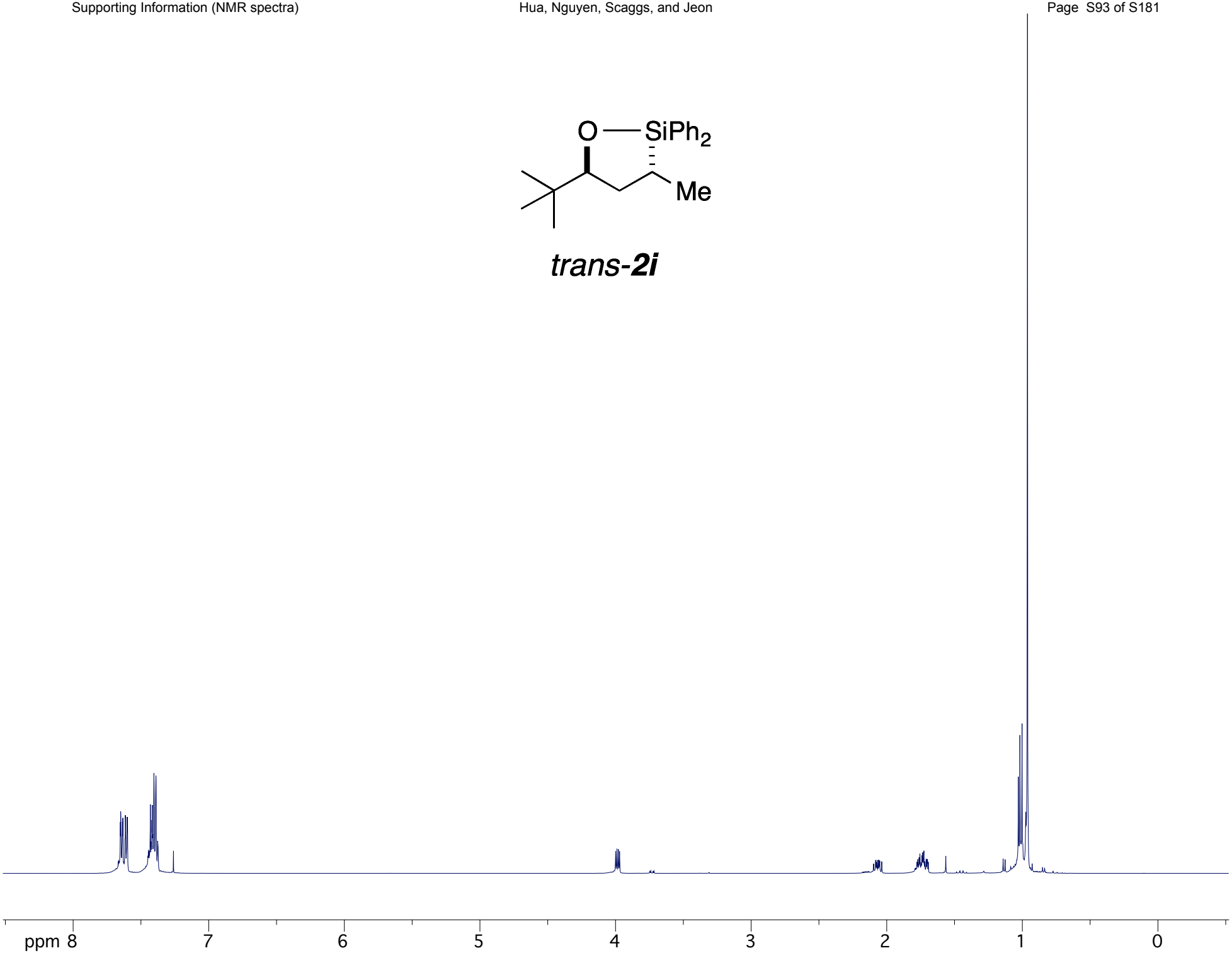
*trans-**2h***

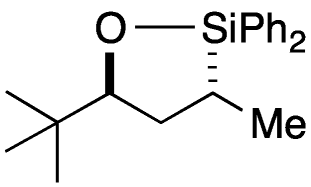


S#679404 (single pulse decoupled gated NOE) USER: delta -- DATE: Mar 15, 2013 08:24:55

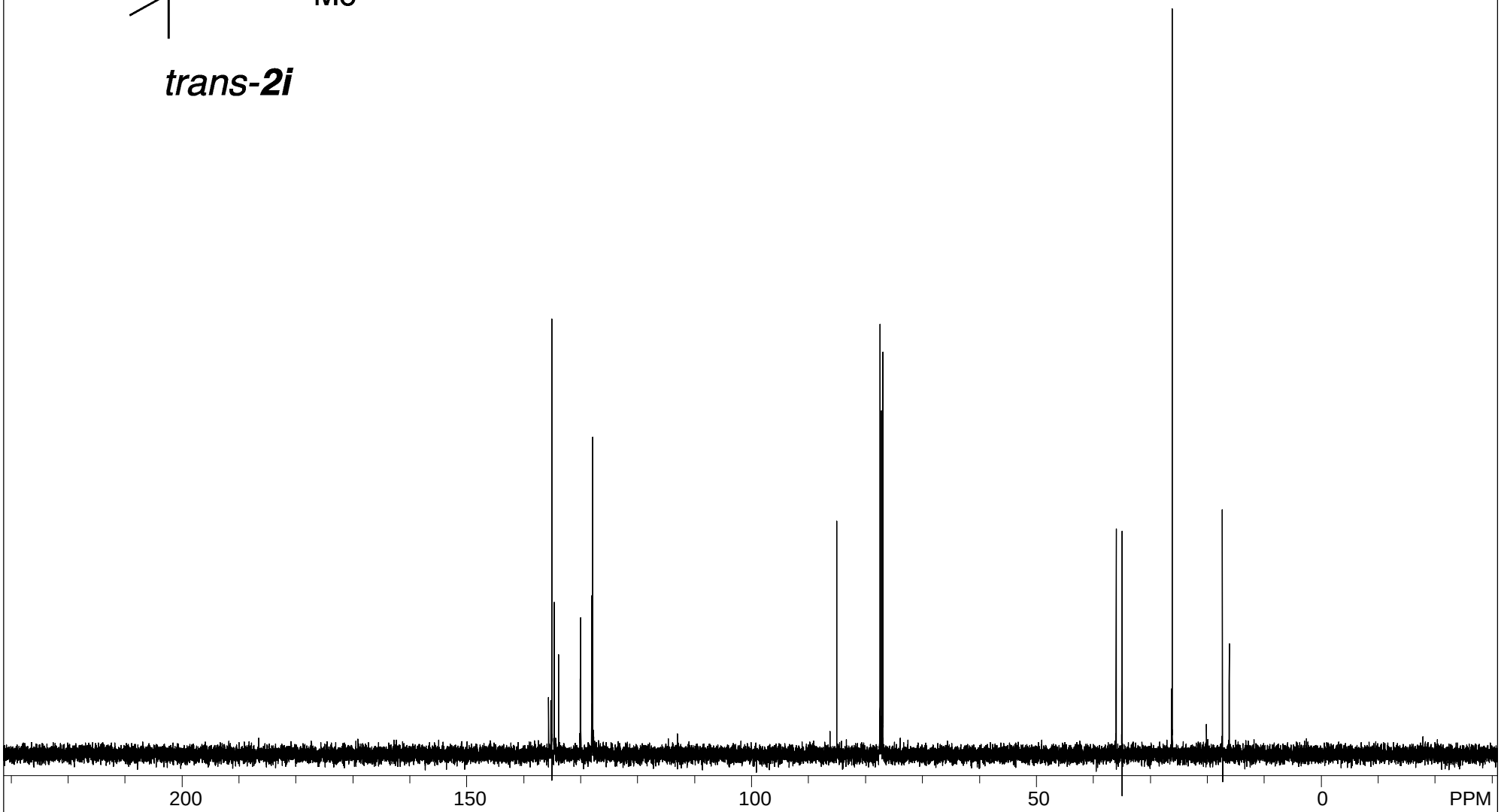
F1: 125.778	F2: 500.162	SW1: 39308		OF1: 12591.4		PTS1d: 32768	
EX: single_pulse_dec		PW: 3.6 us, 30deg	PD: 2.0 sec	NA: 170	LB: 0.0	Nuts - $YH1035B_M4_1_iPr_13C-2.jdf	

*trans-**2i***





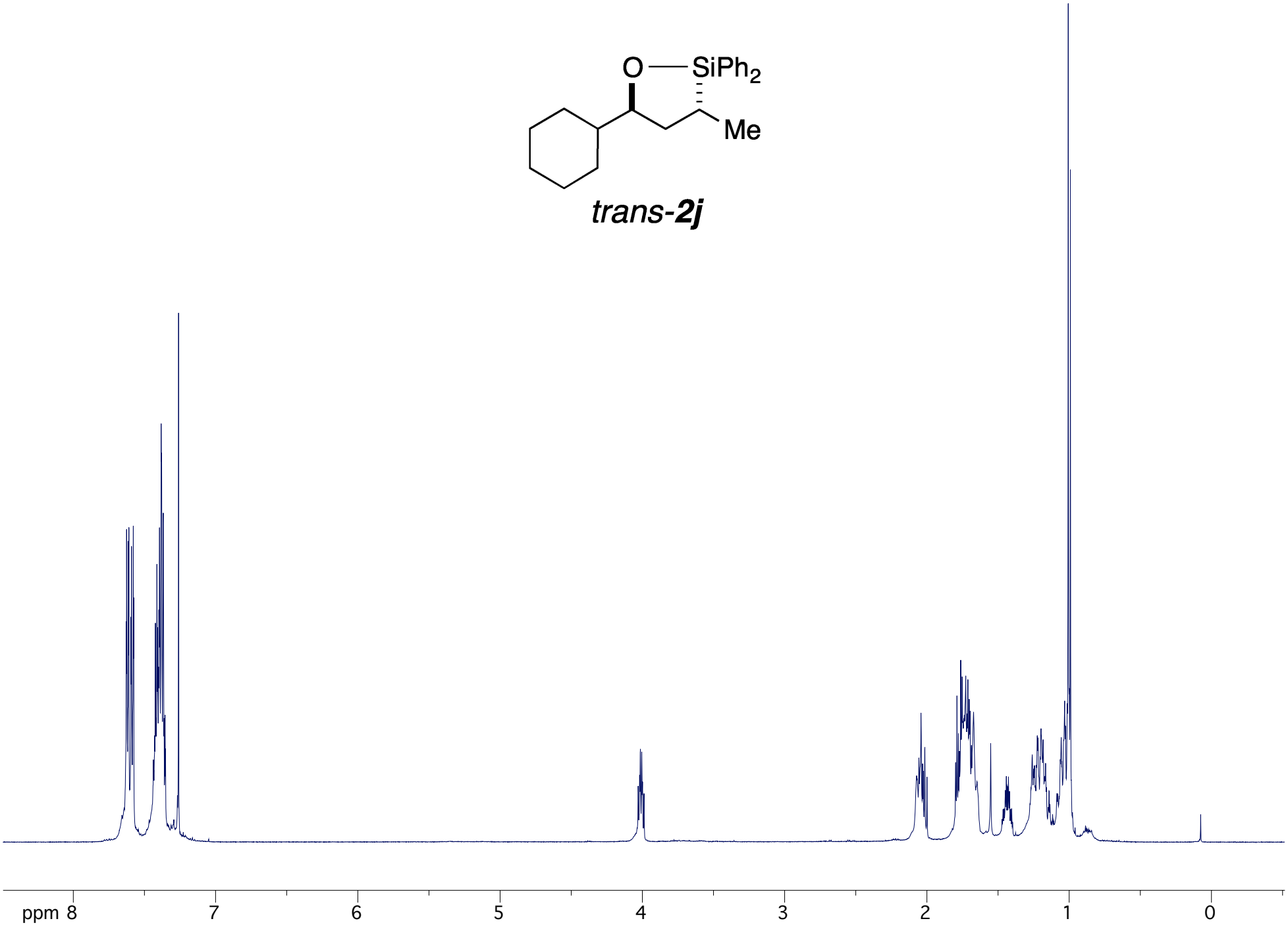
*trans-**2i***

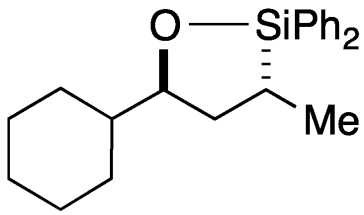


S#756520 (single pulse decoupled gated NOE) USER: delta -- DATE: Feb 15, 2013 11:28:03:

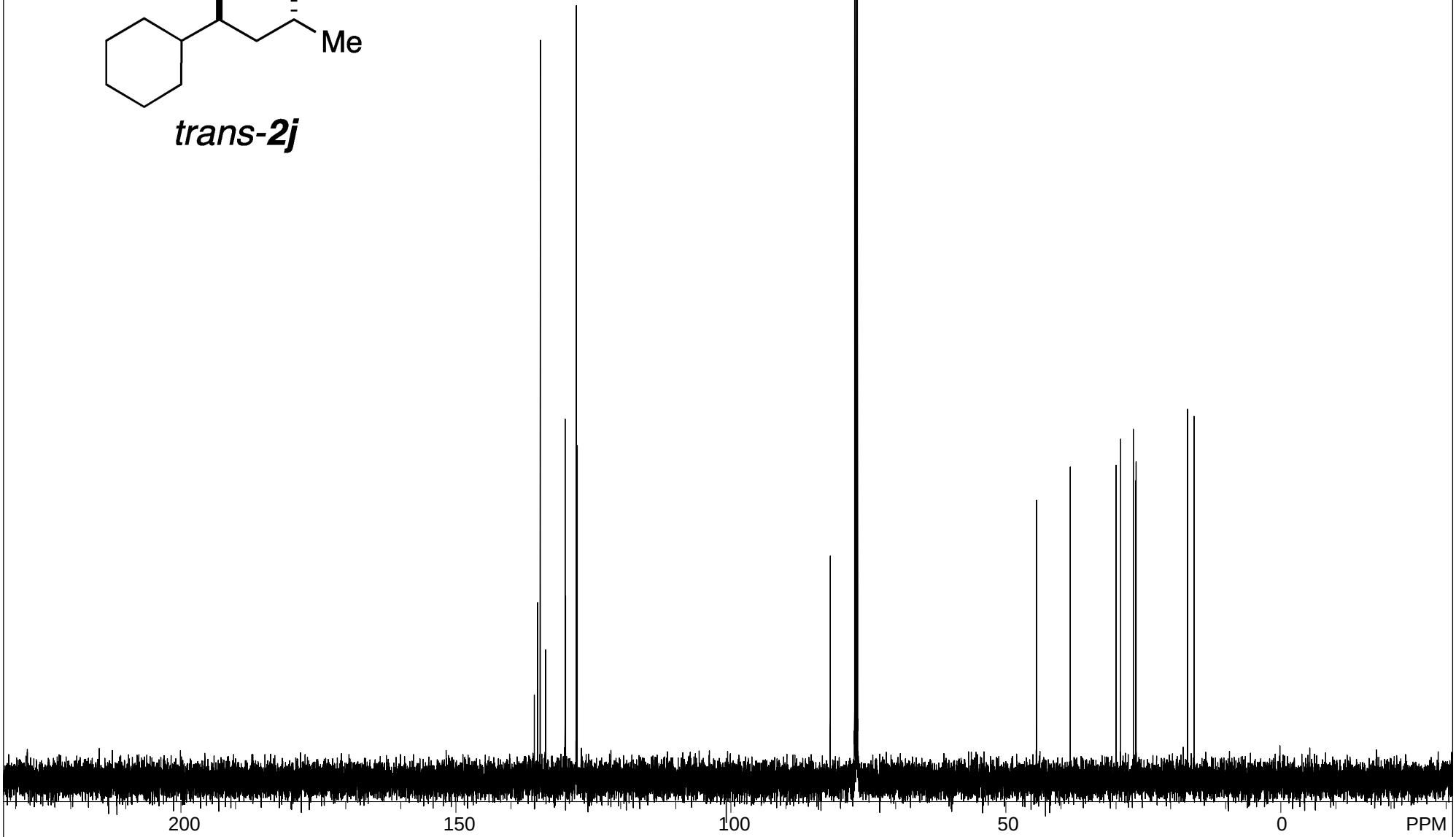
F1: 125.778	F2: 500.162	SW1: 39308		OF1: 12590.2		PTS1d: 32768 , 32768
EX: single_pulse_dec		PW: 3.6 us, 30deg	PD: 2.0 sec	NA: 62	LB: 0.0	Nuts - HHN2017_4_4_5ring_tBu_13C-1_2i.jdf

*trans-**2j***





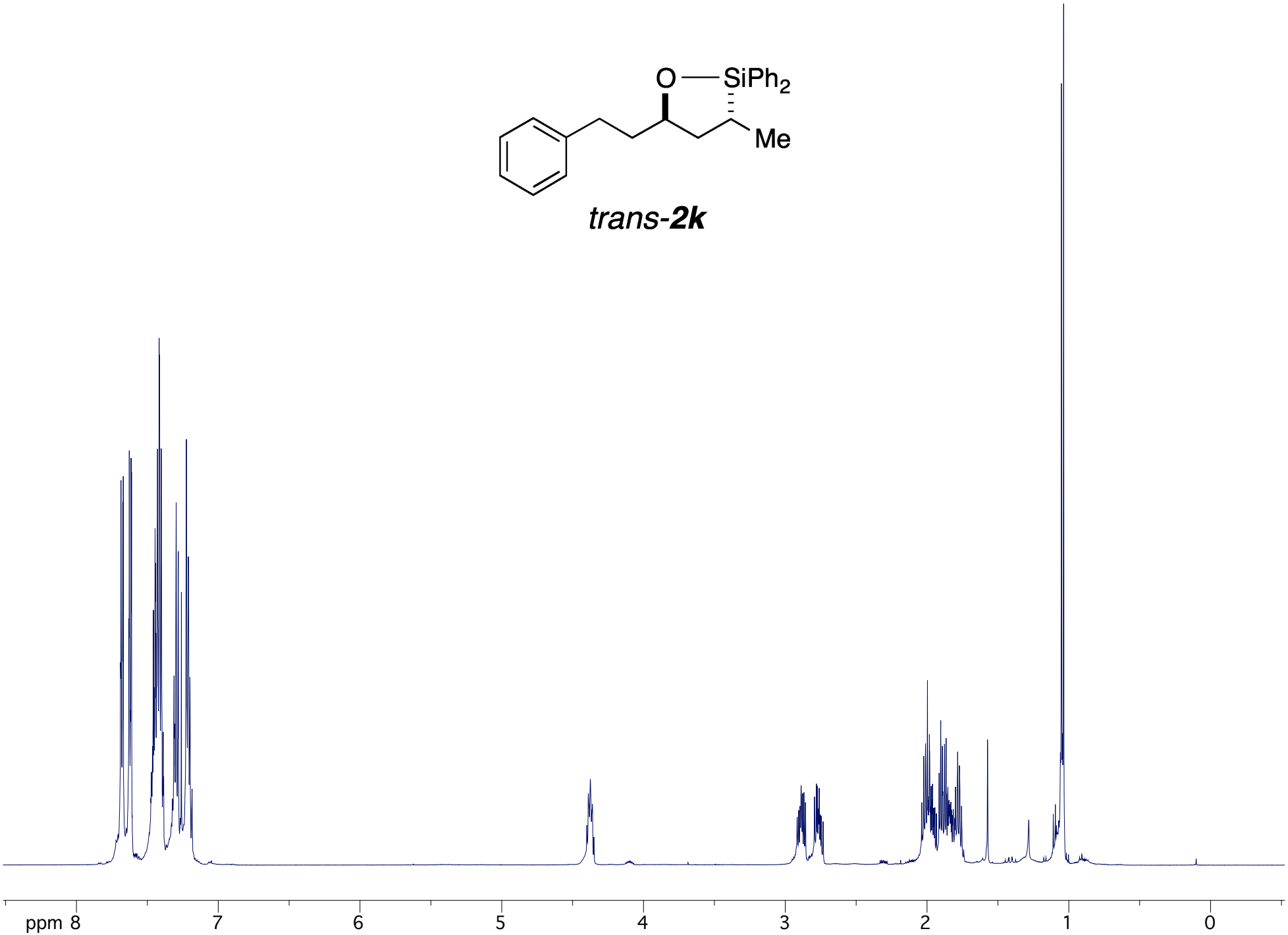
*trans-**2j***



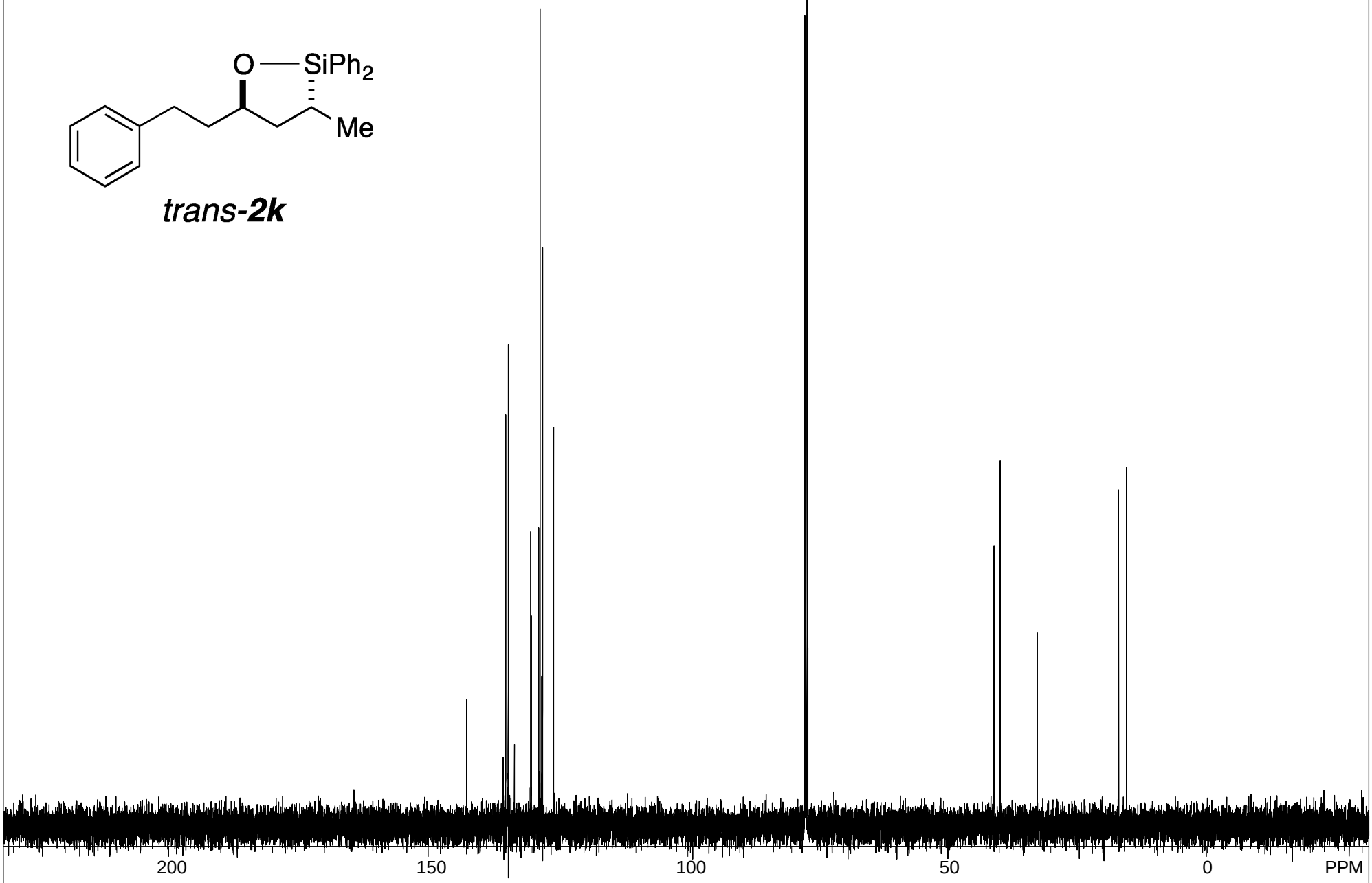
S#619609 (single pulse decoupled gated NOE) USER: delta -- DATE: Mar 1, 2013 07:58:02:

F1: 125.778	F2: 500.162	SW1: 39308		OF1: 12592.6		PTS1d: 32768 , 32768
EX: single_pulse_dec		PW: 3.6 us, 30deg	PD: 2.0 sec	NA: 431	LB: 0.0	Nuts - YH1020_C_5ring_Cy_13C-1_2j.jdf

*trans-**2k***

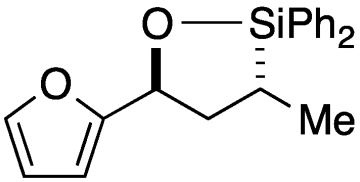


*trans-**2k***

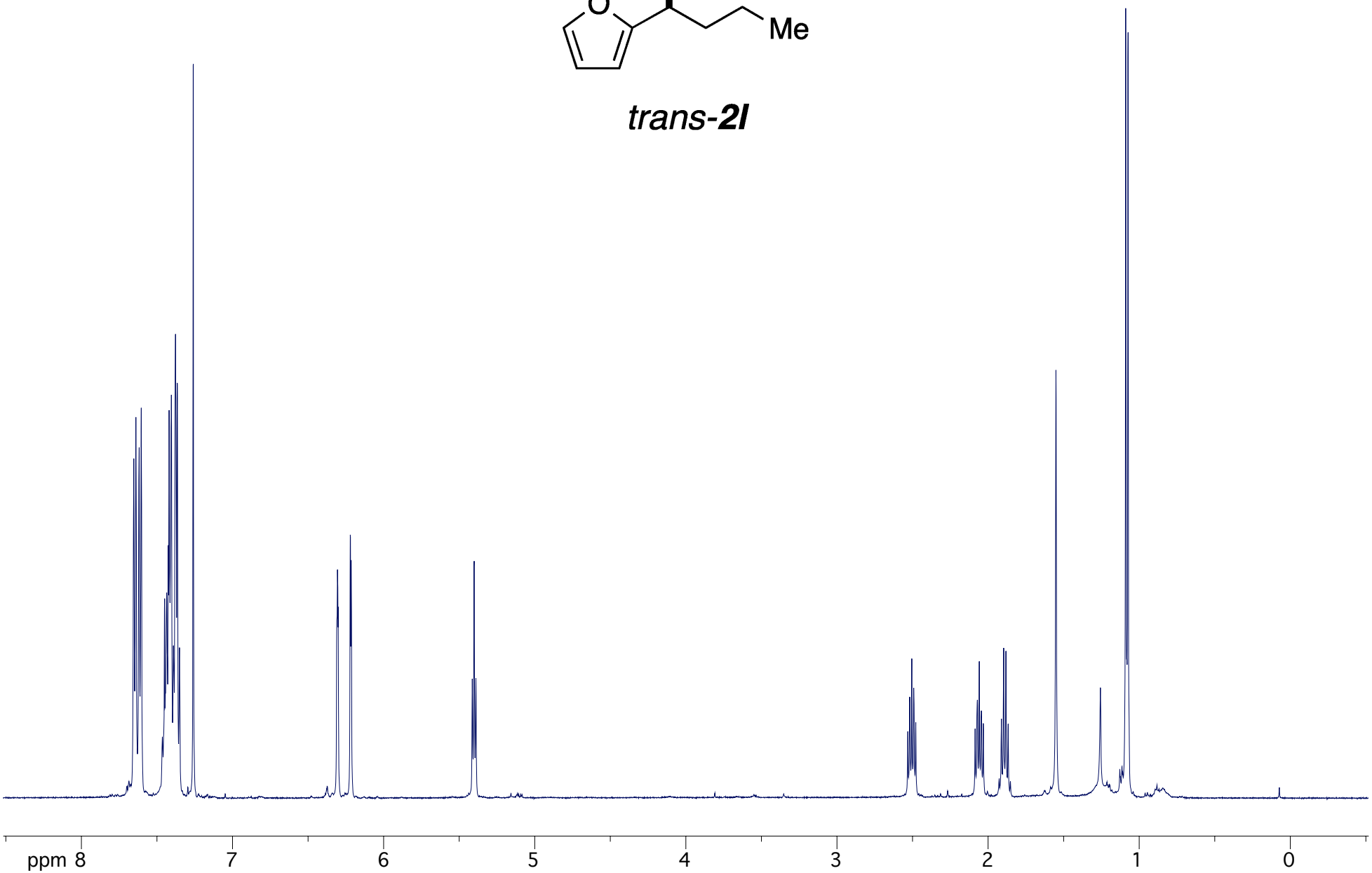


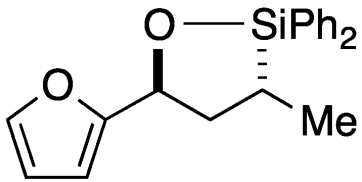
S#768334 (single pulse decoupled gated NOE) USER: delta -- DATE: Feb 15, 2013 11:48:03:

F1: 125.778	F2: 500.162	SW1: 39308		OF1: 12589.0		PTS1d: 32768 , 32768
EX: single_pulse_dec		PW: 3.6 us, 30deg	PD: 2.0 sec	NA: 68	LB: 0.0 Nuts	- YH1020_B_2_5ring_HomoBenzyl_13C-1_2k.jdf

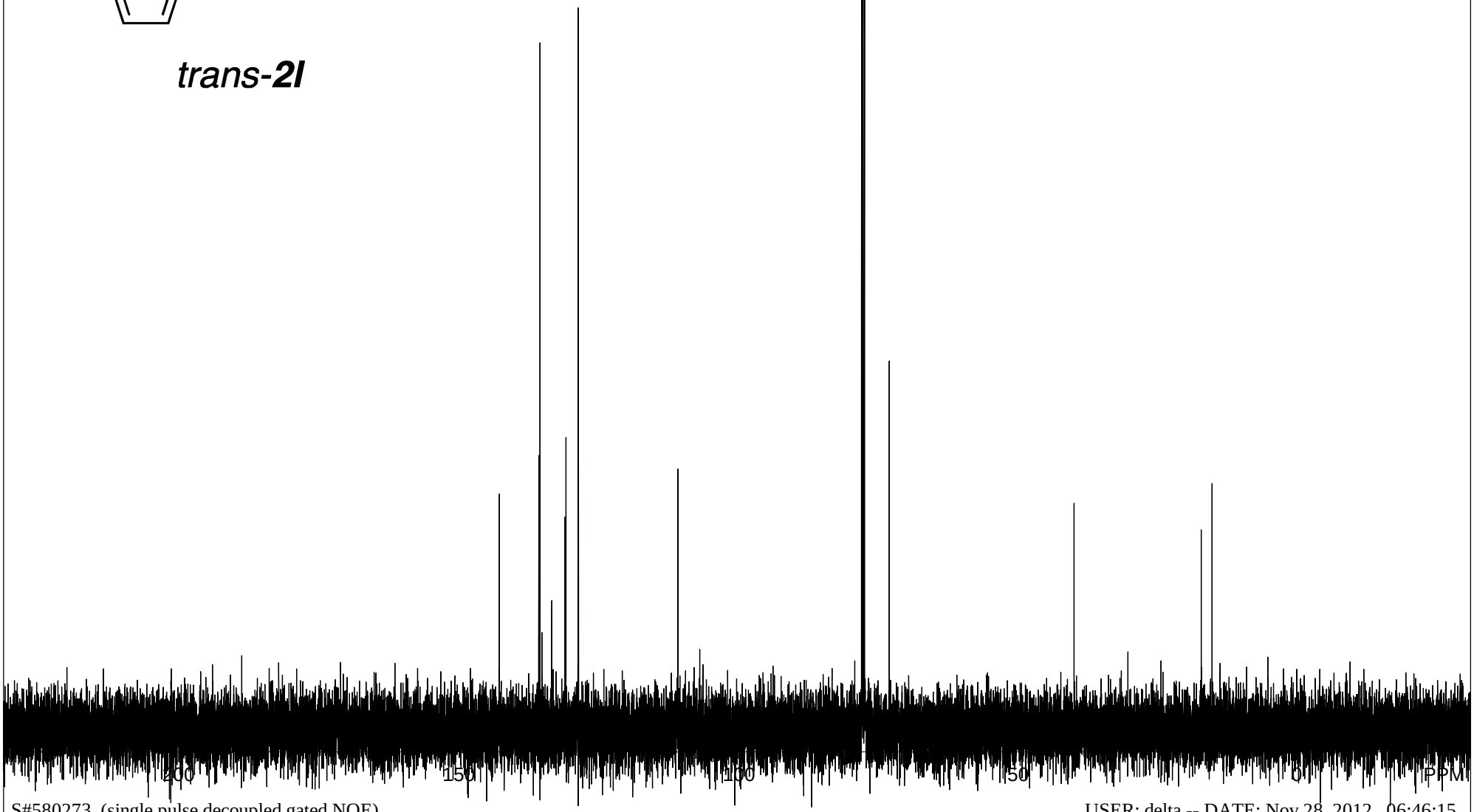


trans-2l





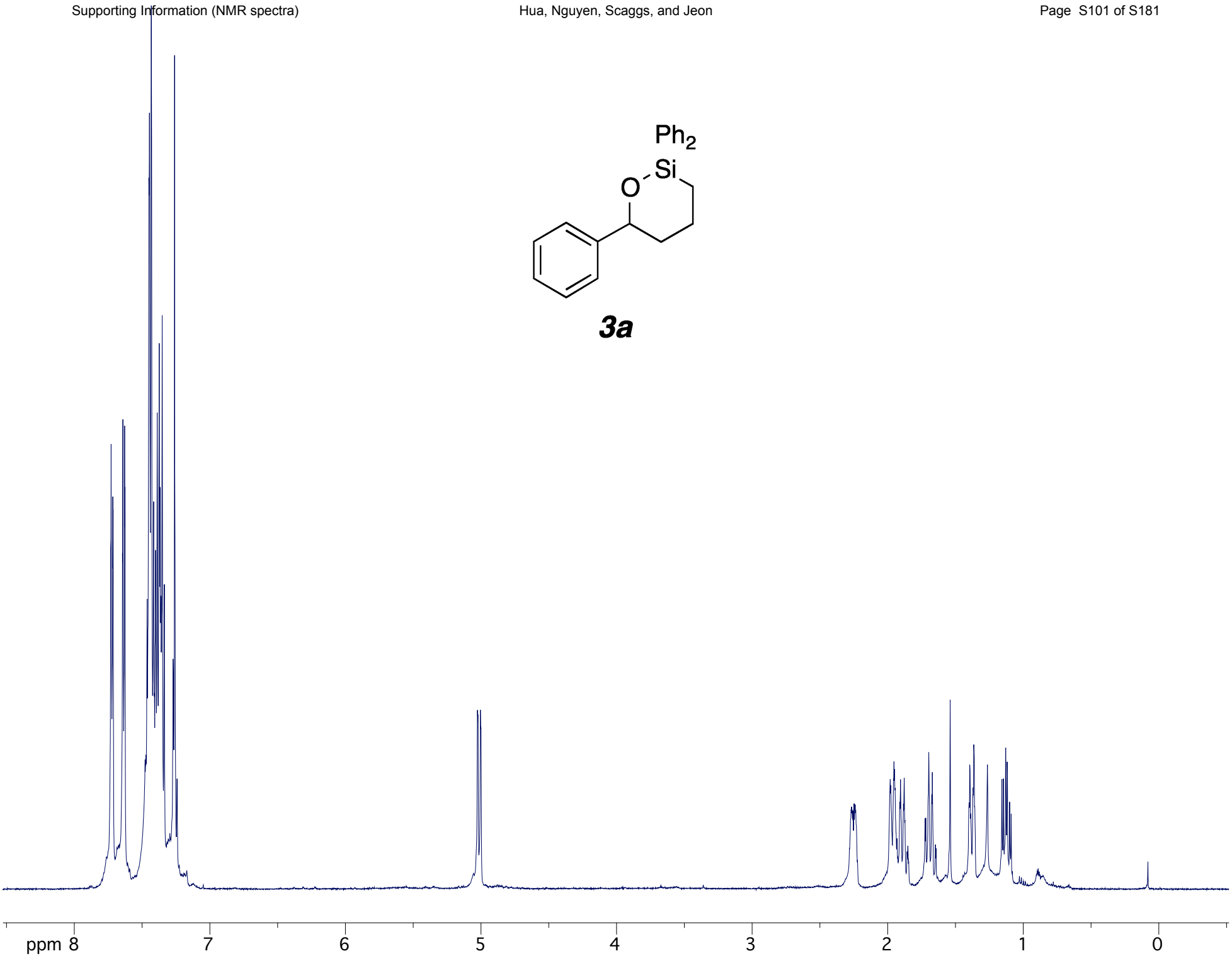
trans-2l



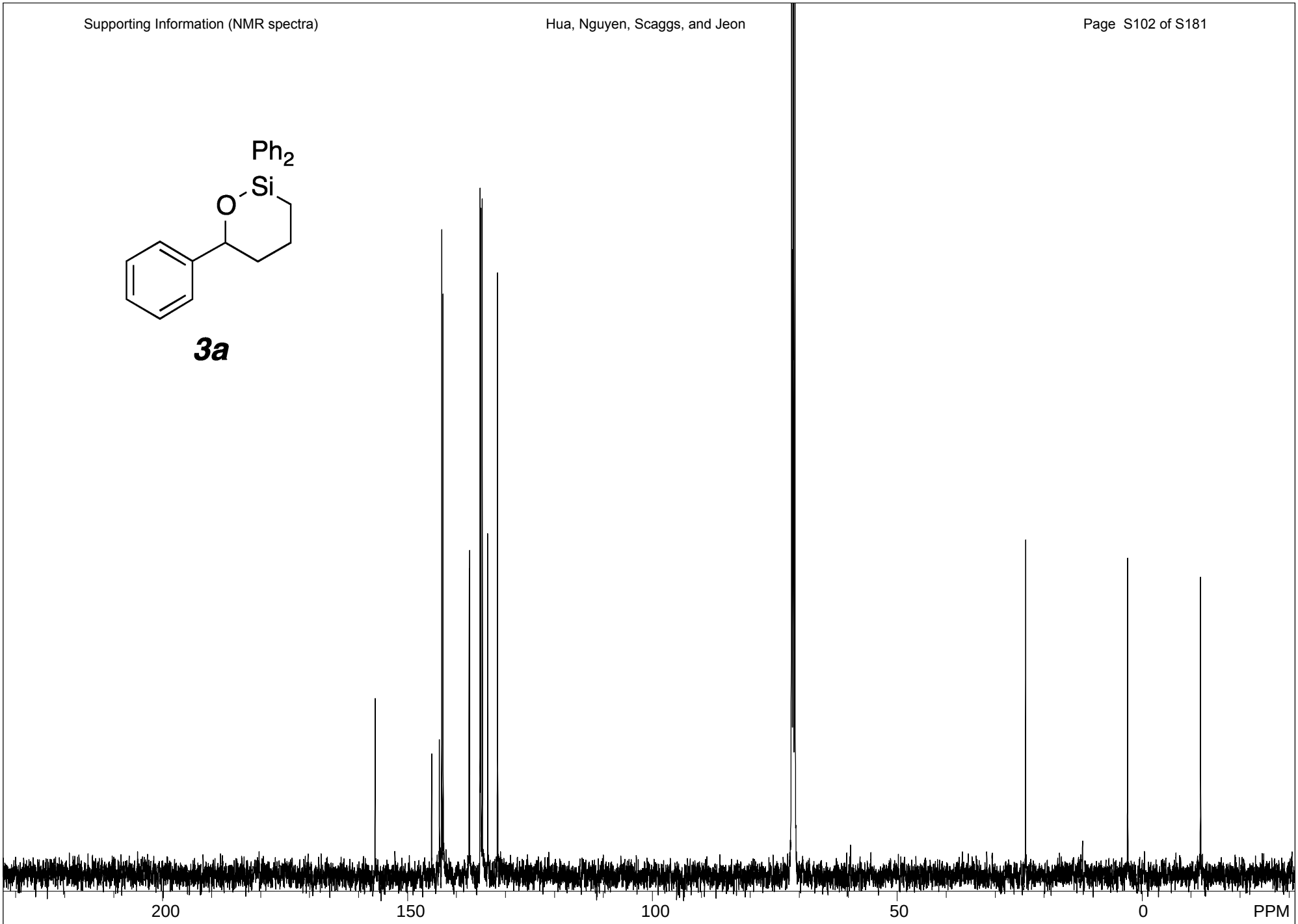
S#580273 (single pulse decoupled gated NOE) USER: delta -- DATE: Nov 28, 2012 06:46:15

F1: 125.778	F2: 500.162	SW1: 39308		OF1: 12592.6		PTS1d: 32768 , 32768
EX: single_pulse_dec		PW: 3.6 us, 30deg	PD: 2.0 sec	NA: 185	LB: 0.0	Nuts - YH2021_D_r2_1_C-1_2l_13C 2.jdf

3a

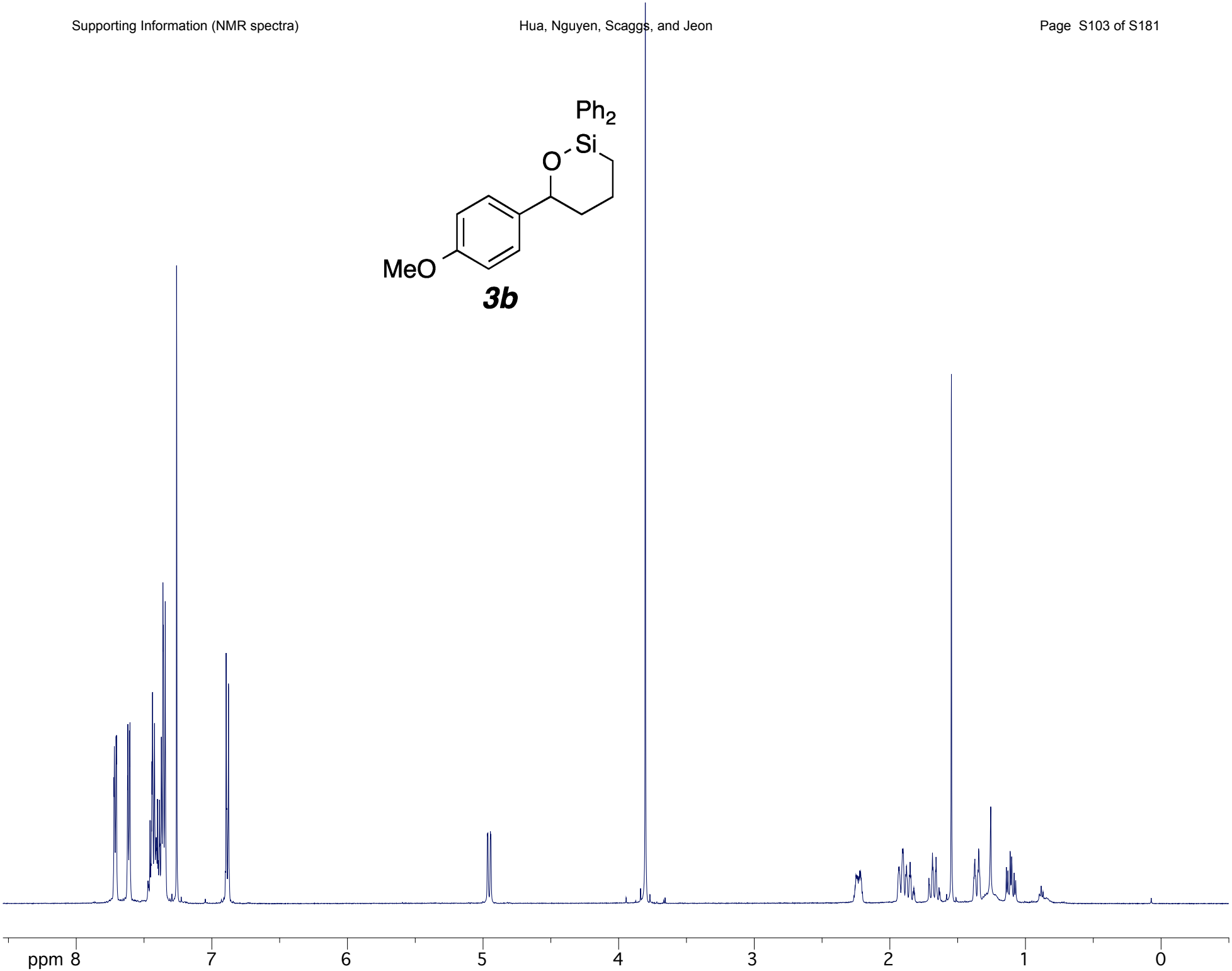


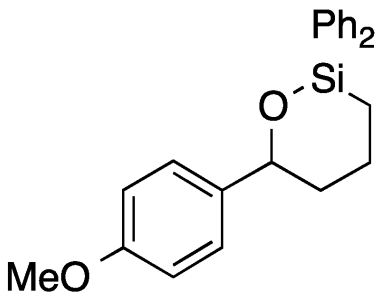
3a



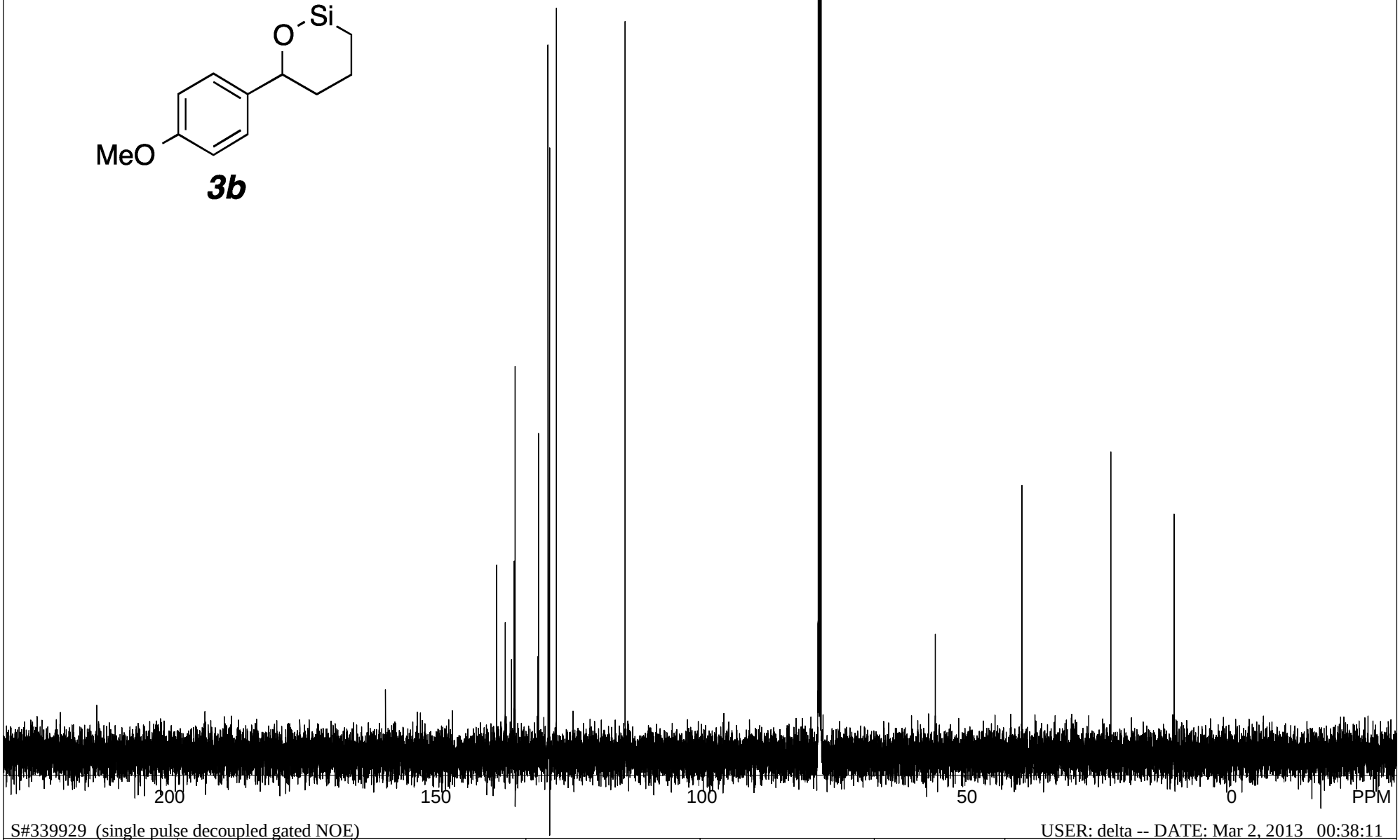
S#635169 (single pulse decoupled gated NOE) USER: delta -- DATE: Mar 1, 2013 08:42:38

F1: 125.778	F2: 500.162	SW1: 39308		OF1: 12556.6		PTS1d: 26224	
EX: single_pulse_dec		PW: 3.6 us, 30deg	PD: 2.0 sec	NA: 839	LB: 0.0	Nuts - $YH2076_6ring_Ph_13C-3.jdf	


Ph2
Si
O
MeO
3b
ppm 8
7
6
5
4
3
2
1
0



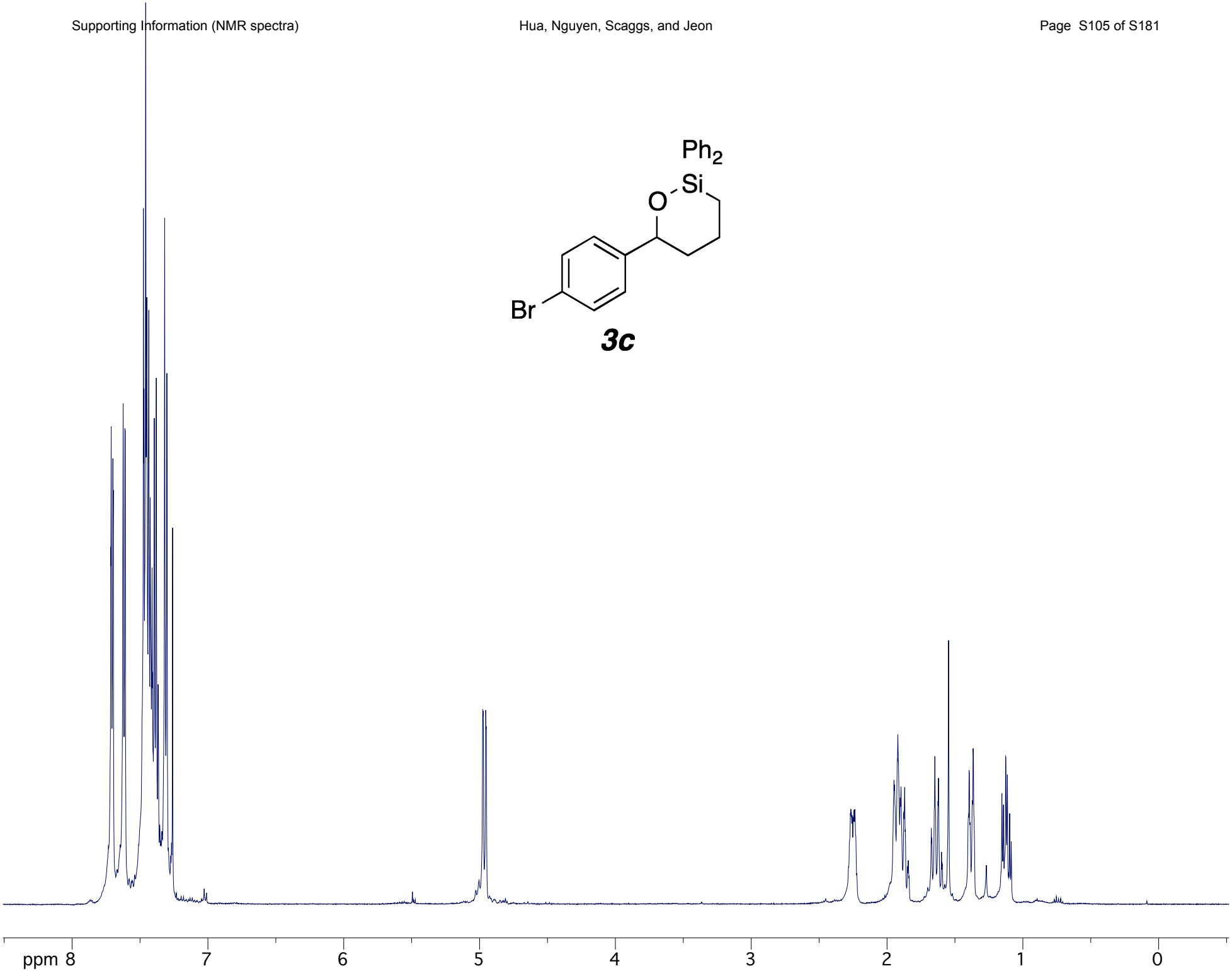
3b



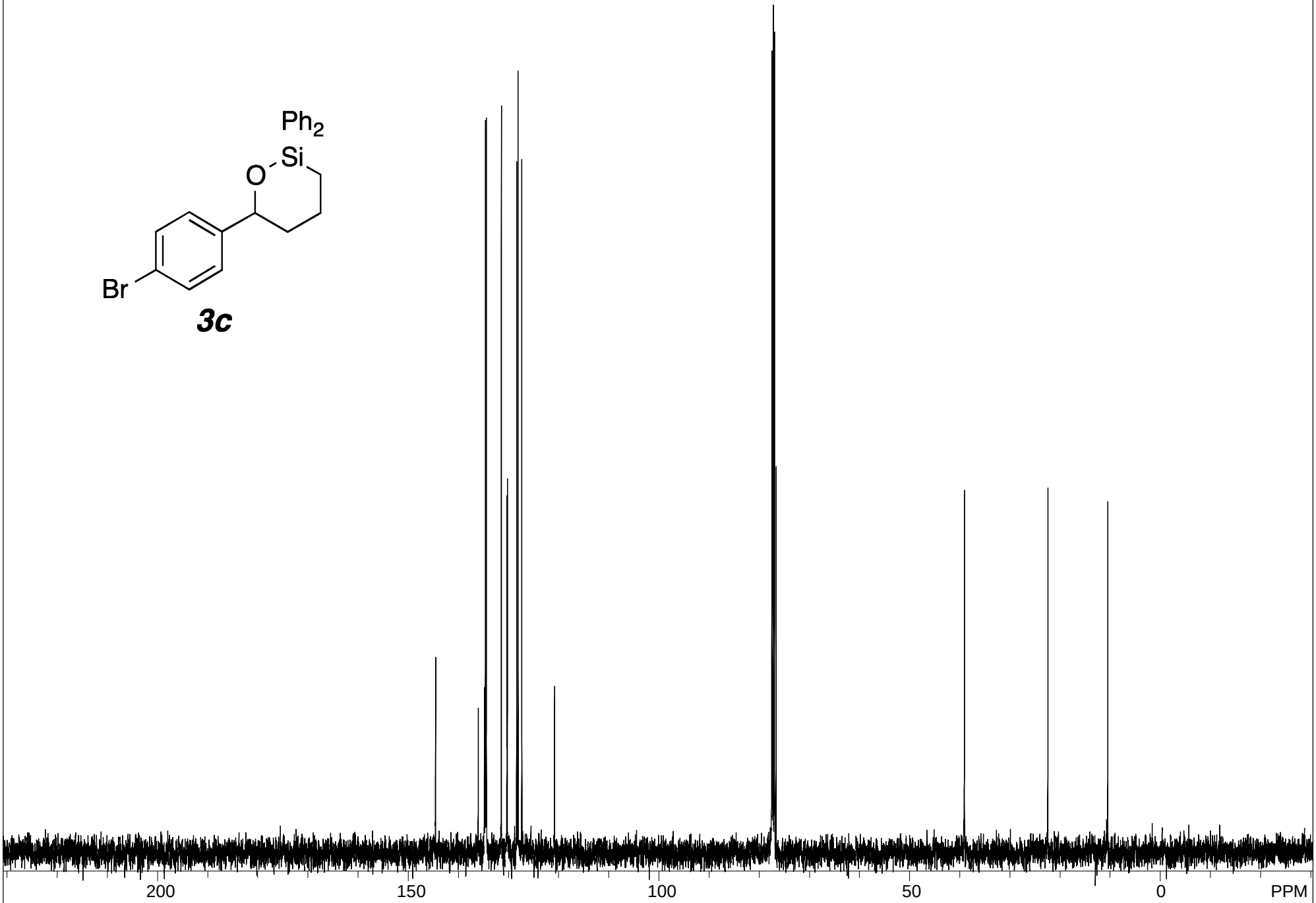
S#339929 (single pulse decoupled gated NOE) USER: delta -- DATE: Mar 2, 2013 00:38:11

F1: 125.778	F2: 500.162	SW1: 39308		OF1: 12592.6		PTS1d: 32768 , 32768
EX: single_pulse_dec		PW: 3.6 us, 30deg	PD: 2.0 sec	NA: 1000	LB: 0.0	Nuts - YH2021_A_6ring_pMeOPh_13C-1_3b.jdf

3c



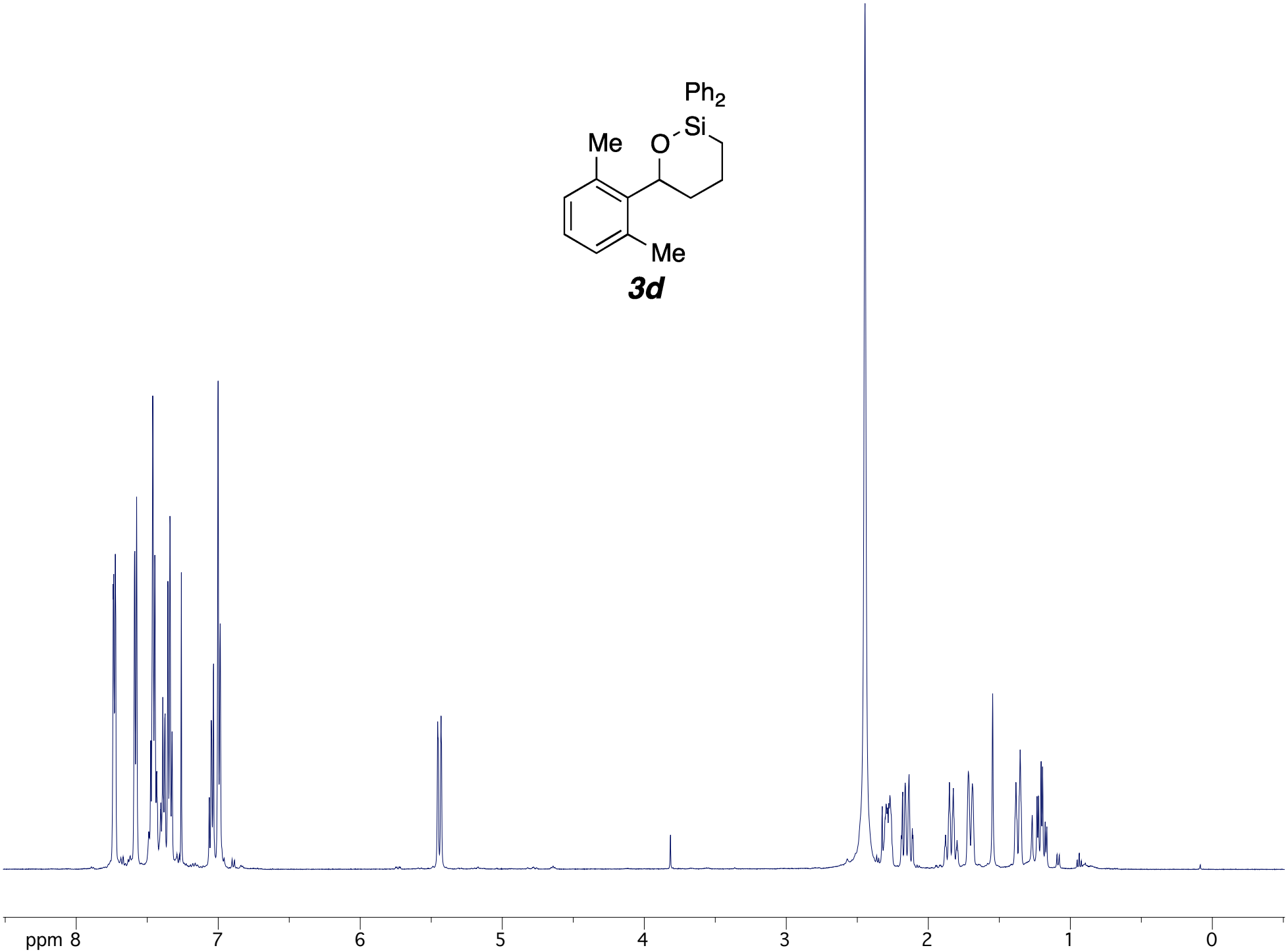
3c

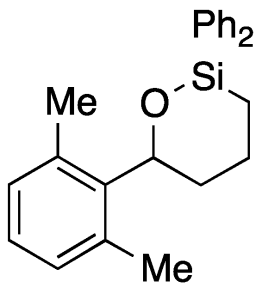


S#615652 (single pulse decoupled gated NOE) USER: delta -- DATE: Feb 26, 2013 07:32:03:

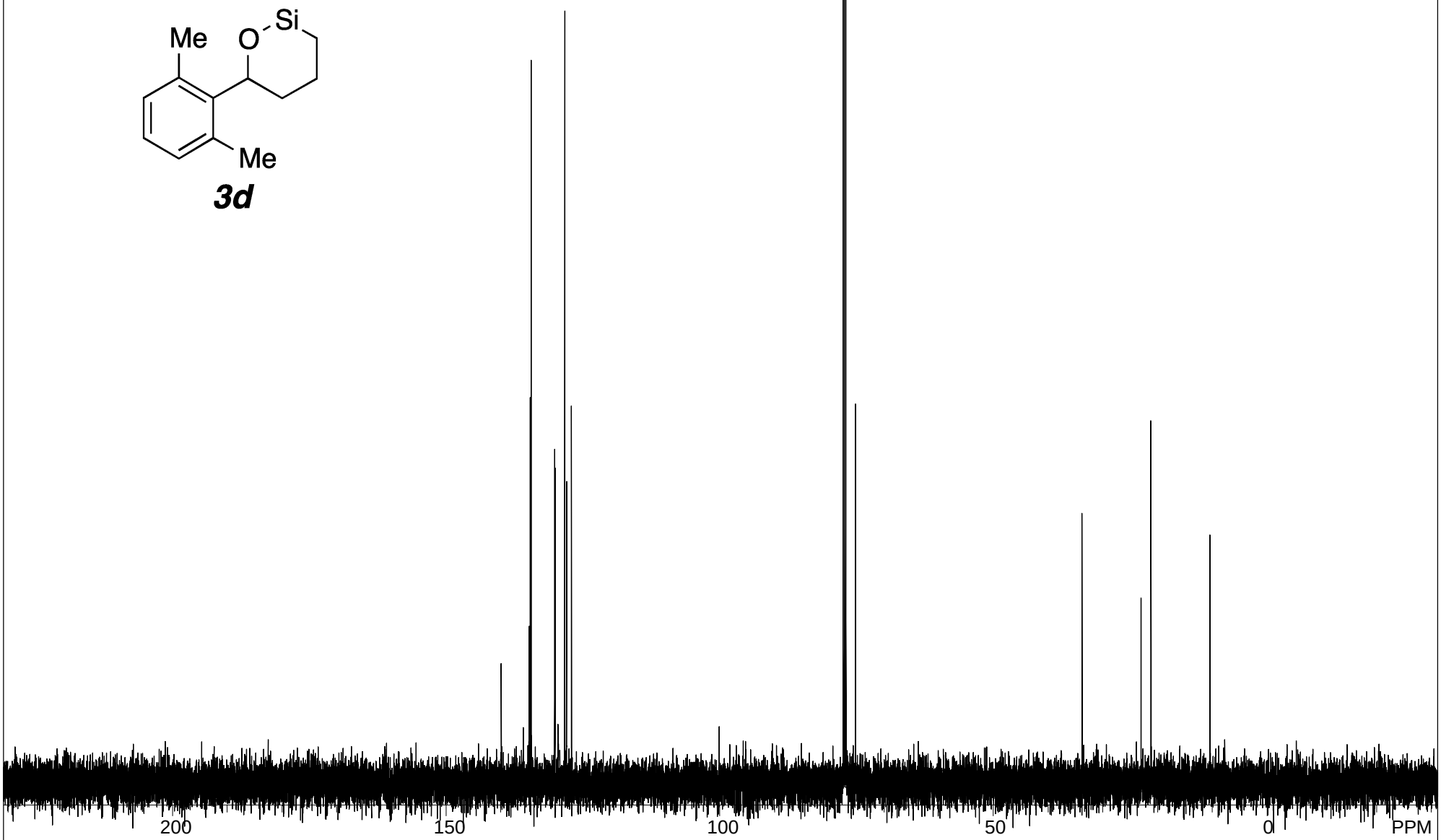
F1: 125.778	F2: 500.162	SW1: 39308		OF1: 12585.4		PTS1d: 32768	
EX: single_pulse_dec		PW: 3.6 us, 30deg	PD: 2.0 sec	NA: 33	LB: 0.0	Nuts - $YH2080_A_M1_1_pBr_6ring_13C-2.jdf	

3d





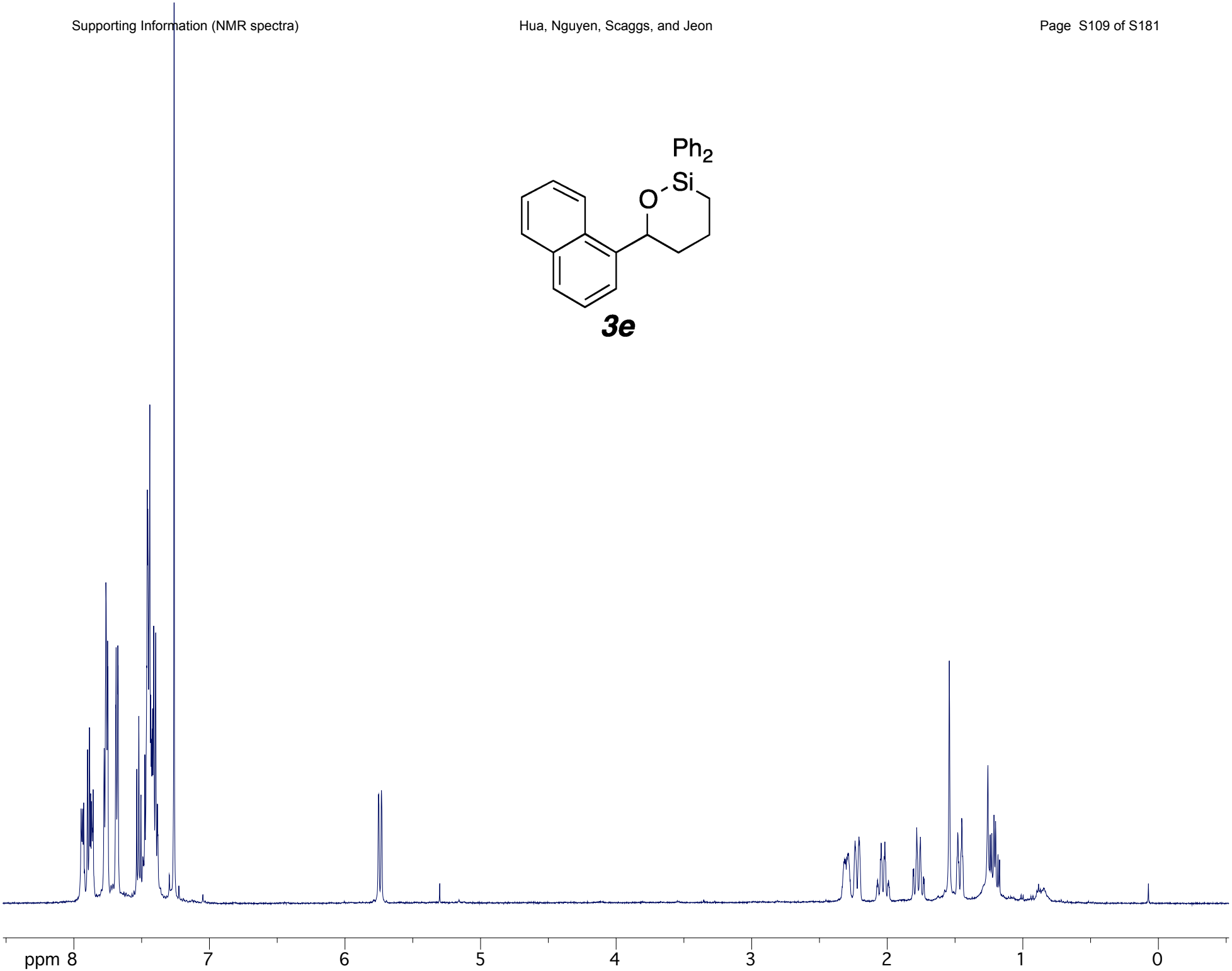
3d



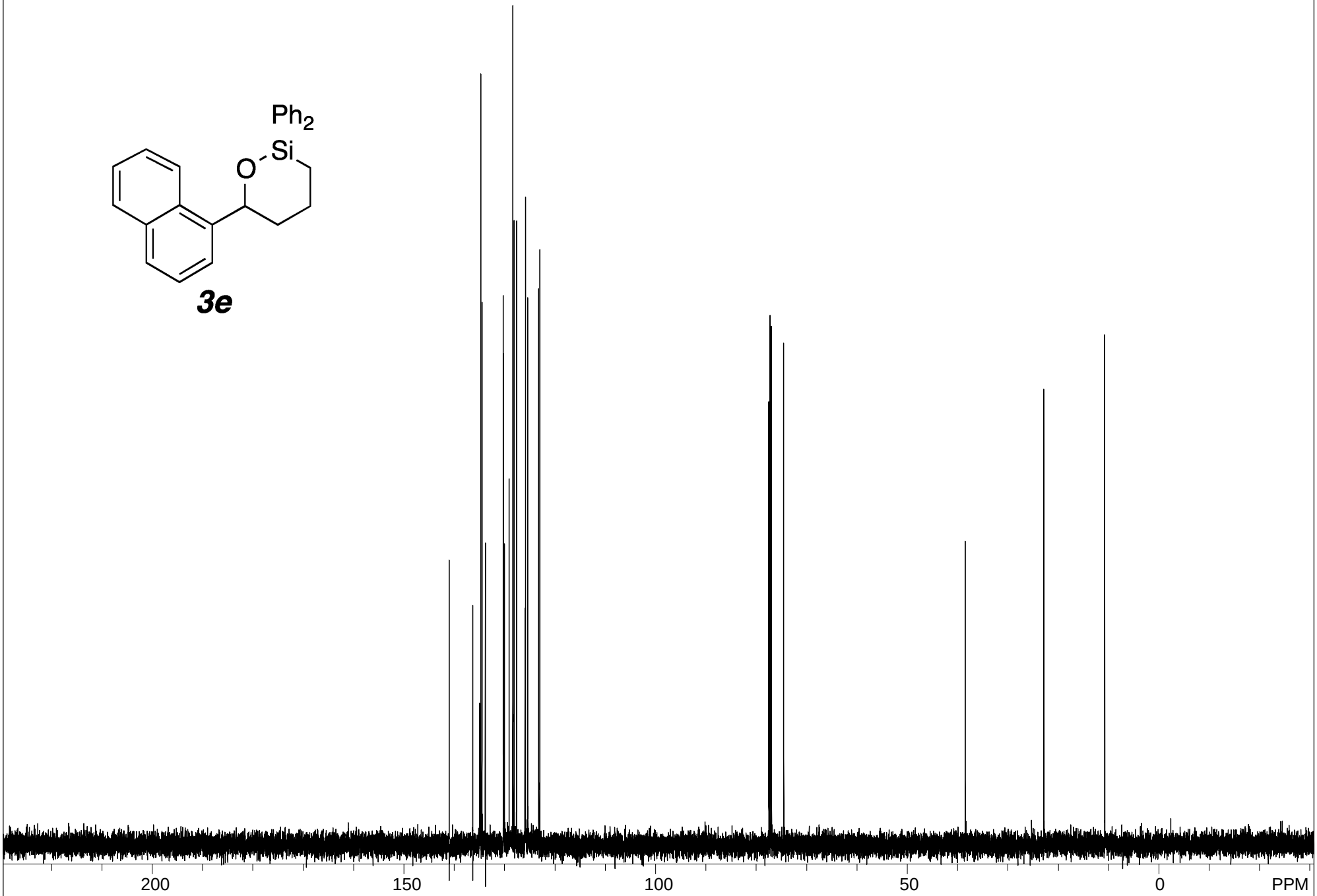
S#384608 (single pulse decoupled gated NOE) USER: delta -- DATE: Nov 28, 2012 01:17:39

F1: 125.778	F2: 500.162	SW1: 39308		OF1: 12591.4		PTS1d: 32768 , 32768
EX: single_pulse_dec		PW: 3.6 us, 30deg	PD: 2.0 sec	NA: 133	LB: 0.0	Nuts - $YH2021_C_MPLC_r1_2_C-1.jdf

3e



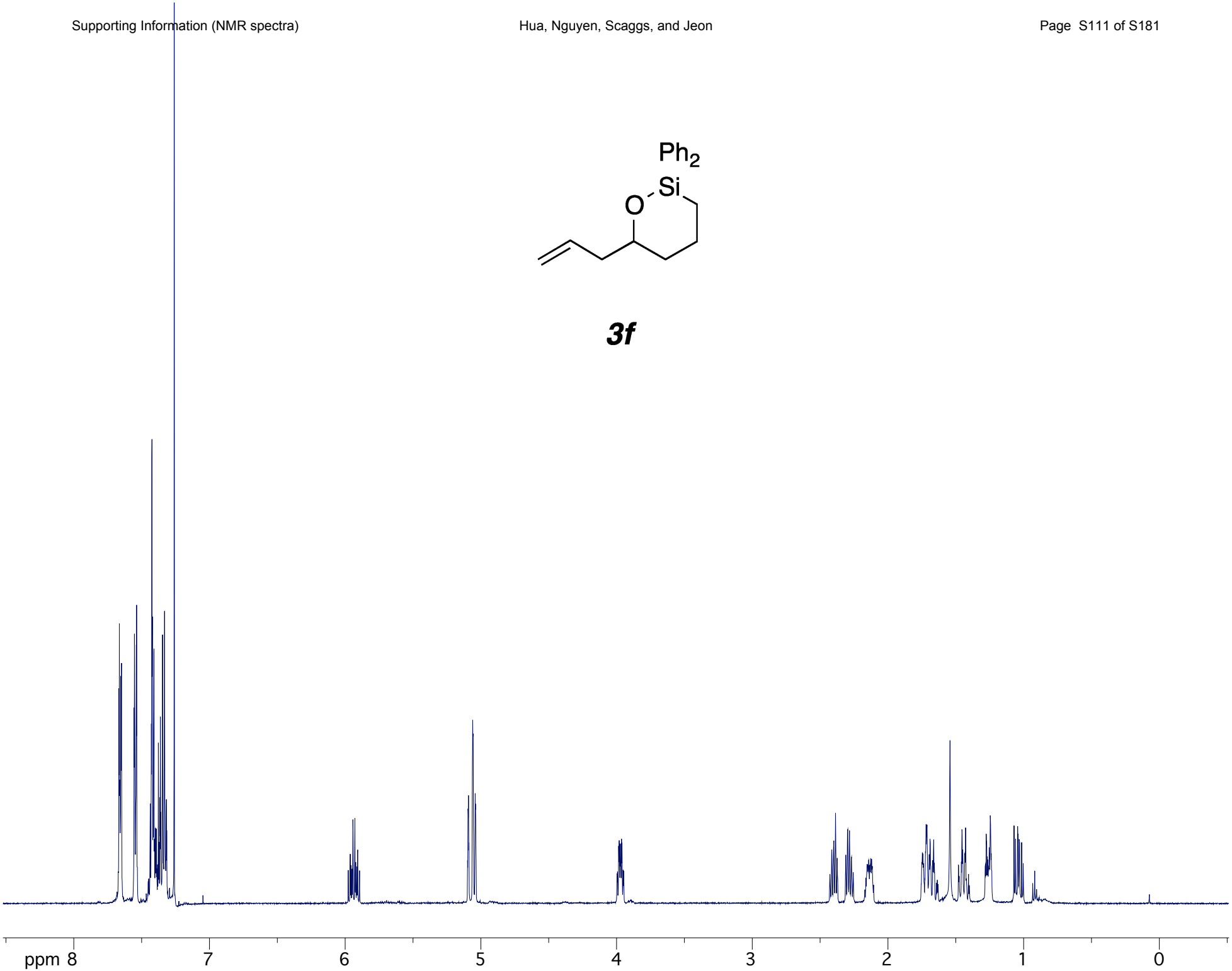
3e

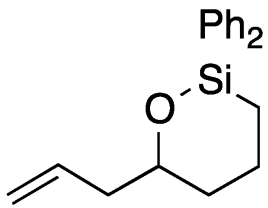


S#738341 (single pulse decoupled gated NOE) USER: delta -- DATE: Mar 2, 2013 10:57:16

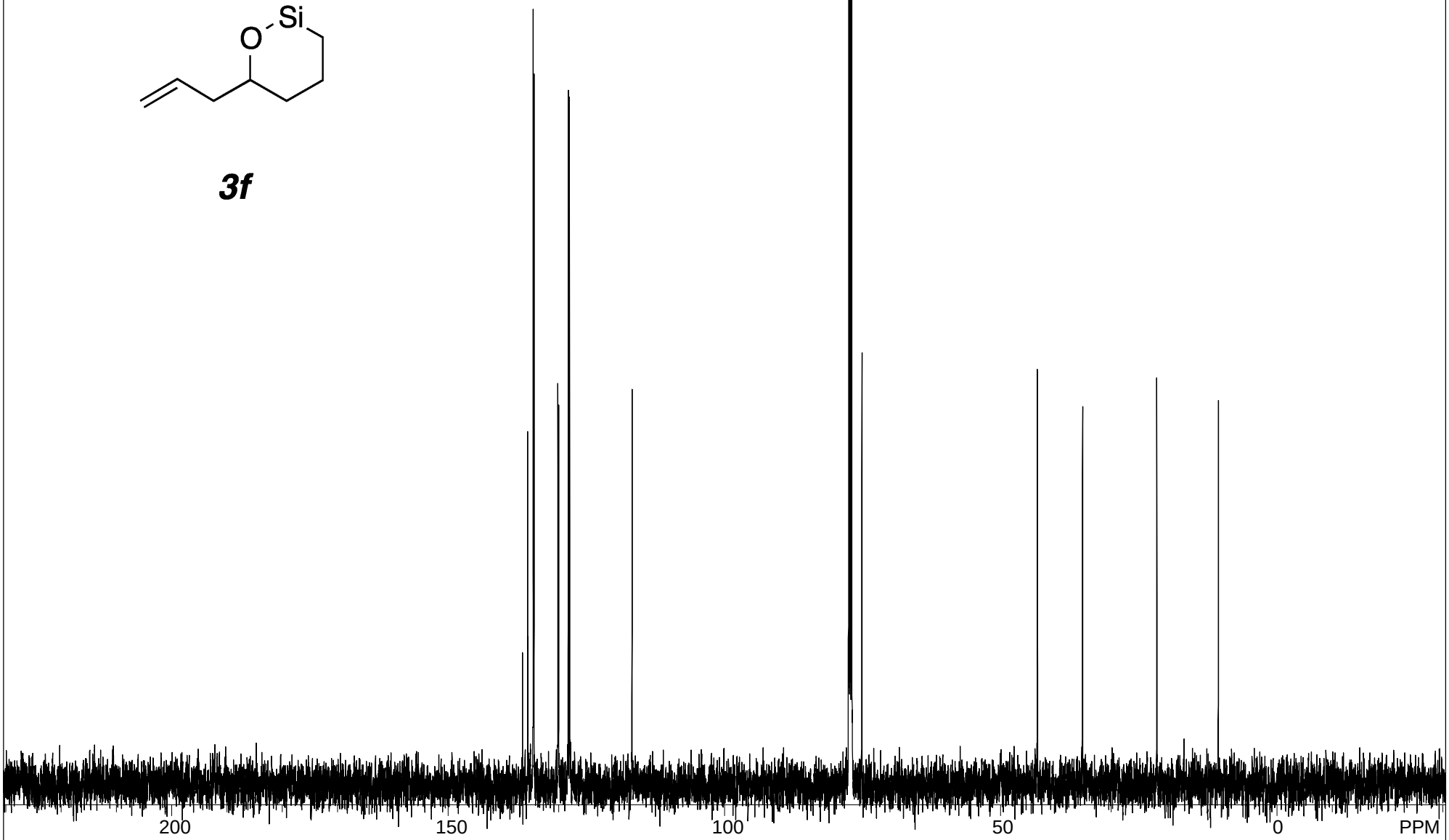
F1: 125.778	F2: 500.162	SW1: 39308		OF1: 12577.0		PTS1d: 32768 , 32768
EX: single_pulse_dec		PW: 3.6 us, 30deg	PD: 2.0 sec	NA: 48	LB: 0.0	Nuts - YH2082_M1_6ring_naph_13C-1_3e.jdf

3f



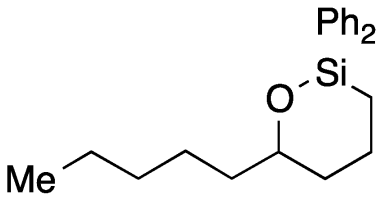


3f

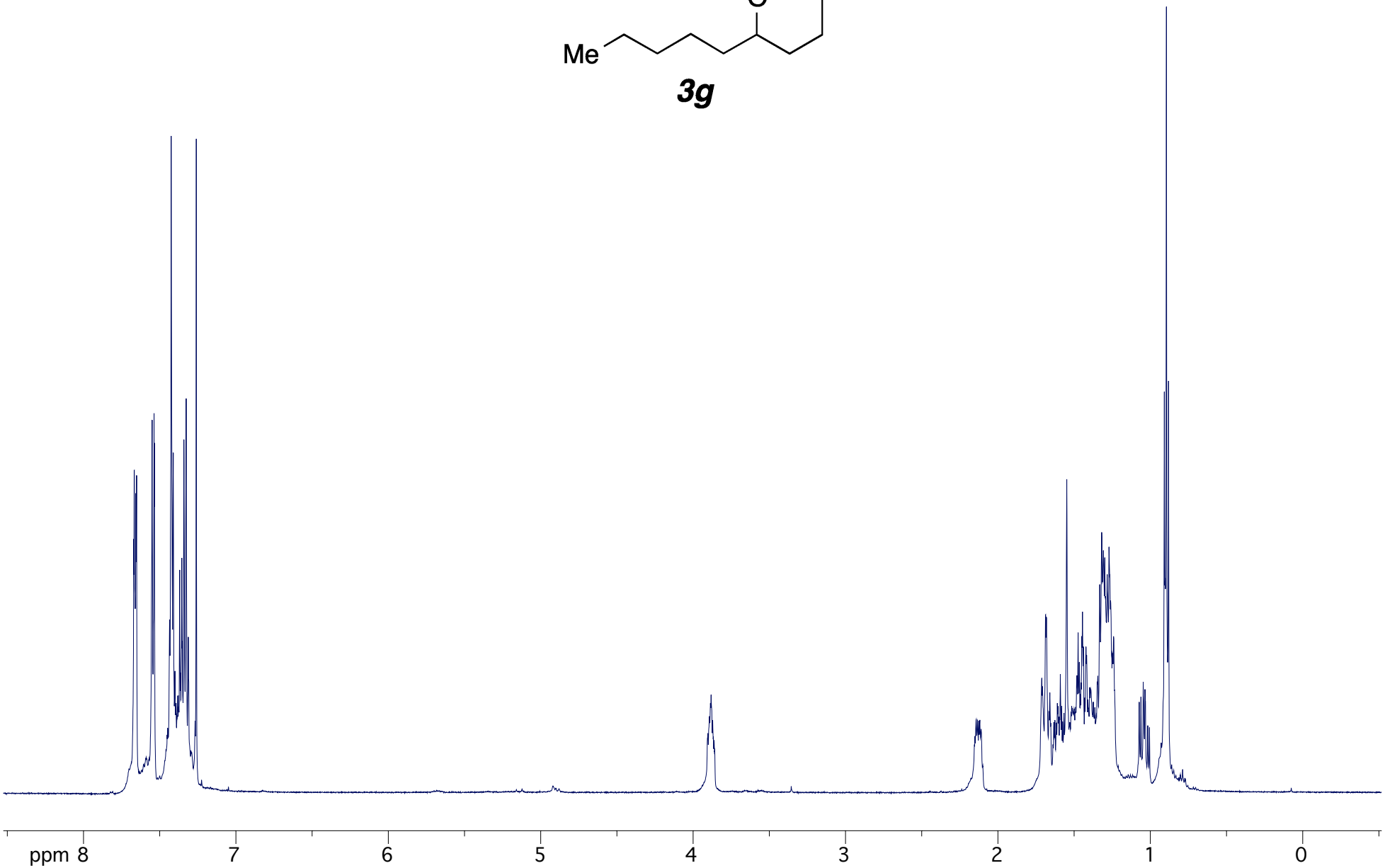


S#726498 (single pulse decoupled gated NOE) USER: delta -- DATE: Feb 21, 2013 10:39:26

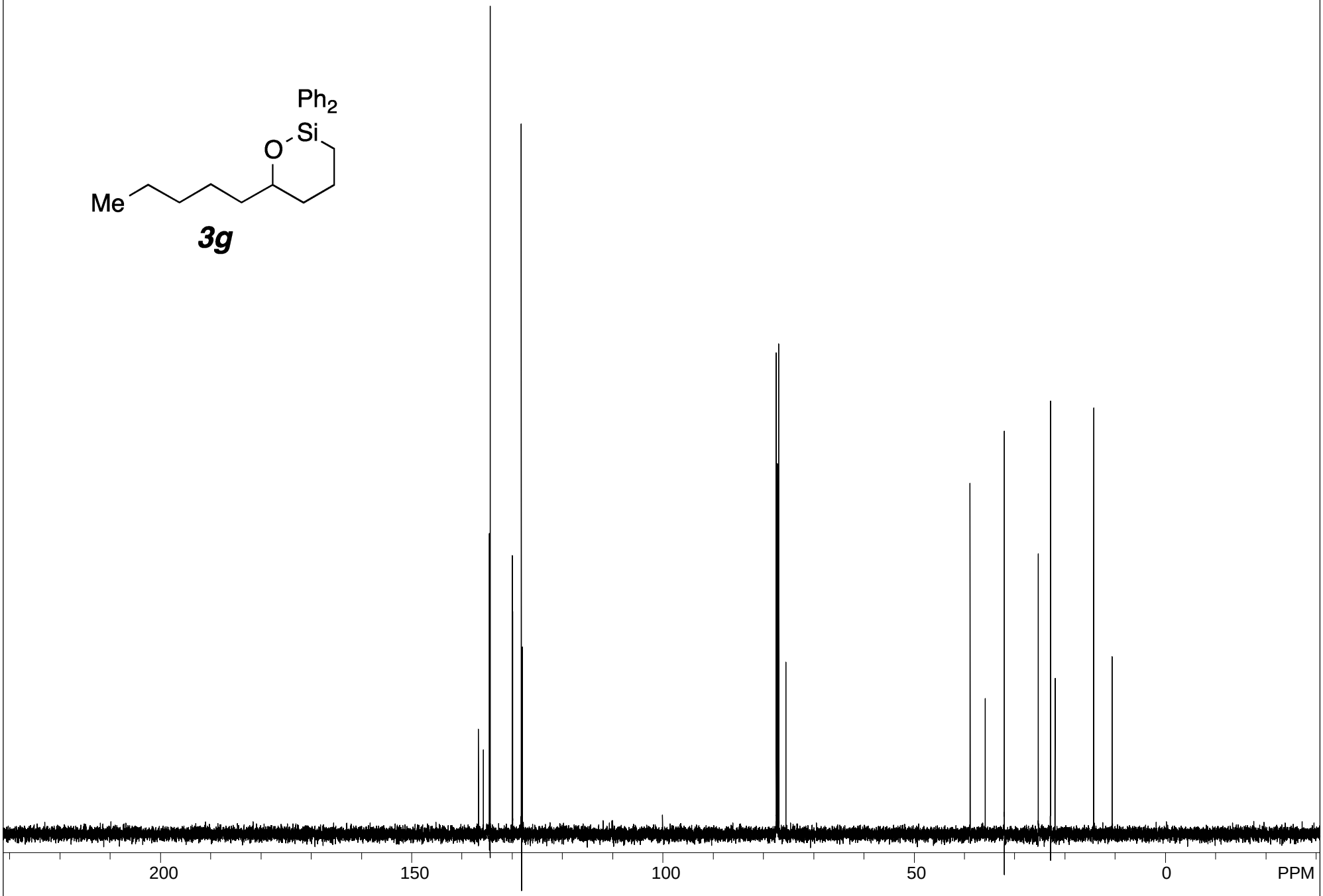
F1: 125.778	F2: 500.162	SW1: 39308		OF1: 12591.4		PTS1d: 32768	
EX: single_pulse_dec		PW: 3.6 us, 30deg	PD: 2.0 sec	NA: 90	LB: 0.0	Nuts - $YH_6ring_Allyl_13C-2.jdf	



3g

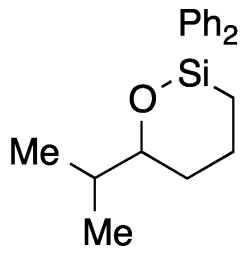


3g

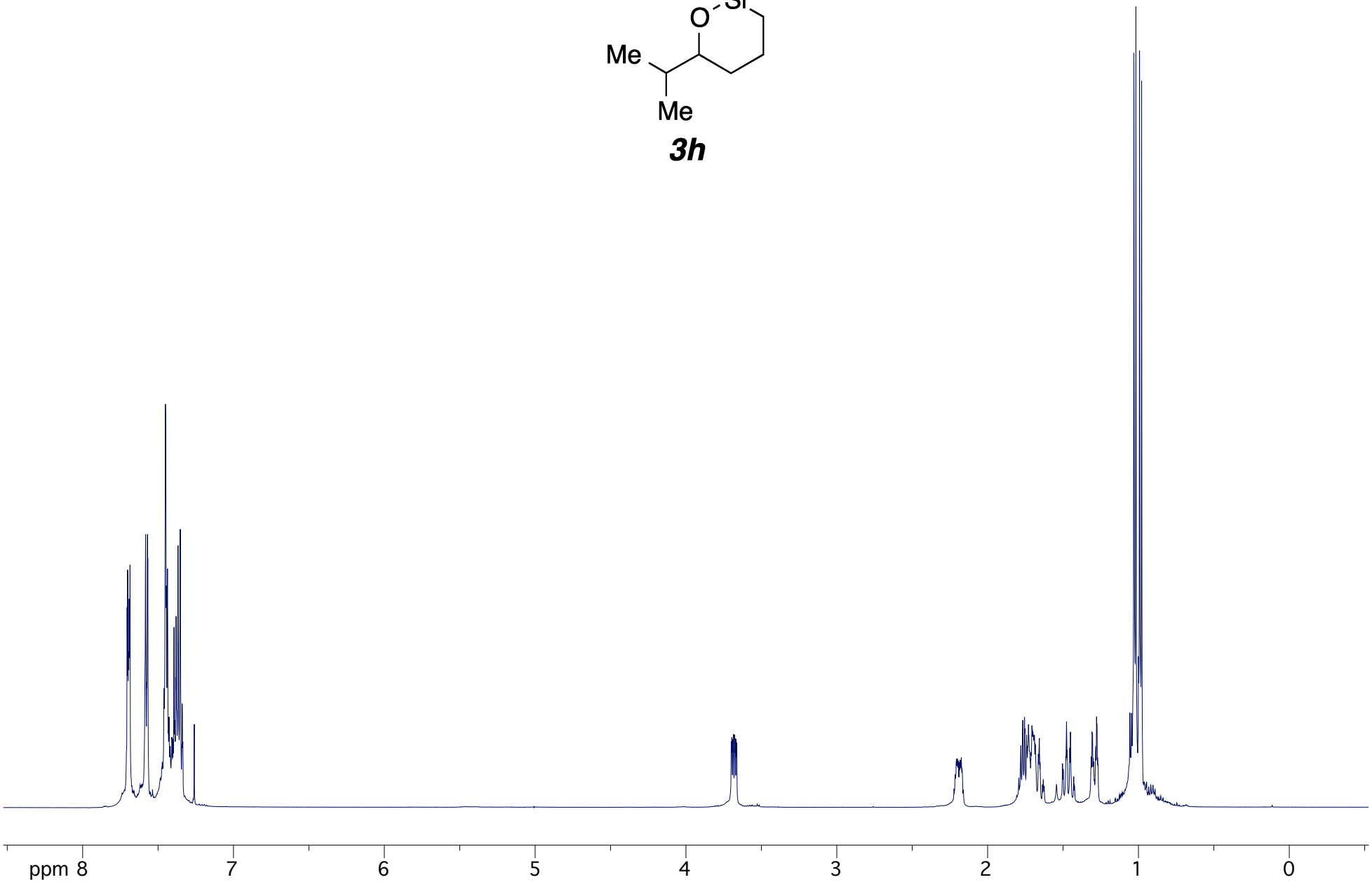


S#695839 (single pulse decoupled gated NOE) USER: delta -- DATE: Jul 31, 2012 09:16:08:

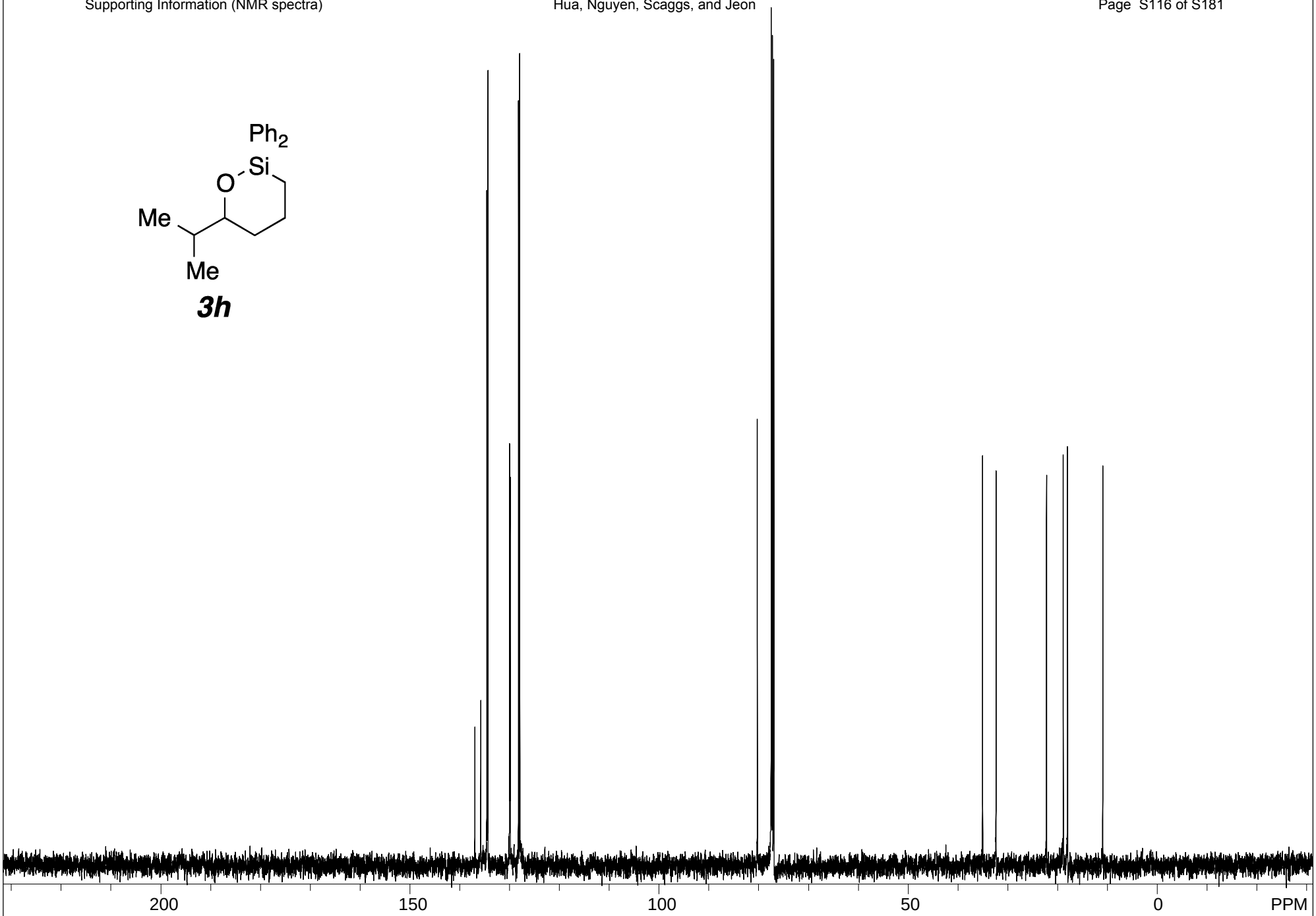
F1: 125.778	F2: 500.162	SW1: 39308		OF1: 12587.8		PTS1d: 32768 , 32768
EX: single_pulse_dec		PW: 3.6 us, 30deg	PD: 2.0 sec	NA: 82	LB: 0.0	Nuts - WRS2085_1_pent_C13-1_3g.jdf



3h



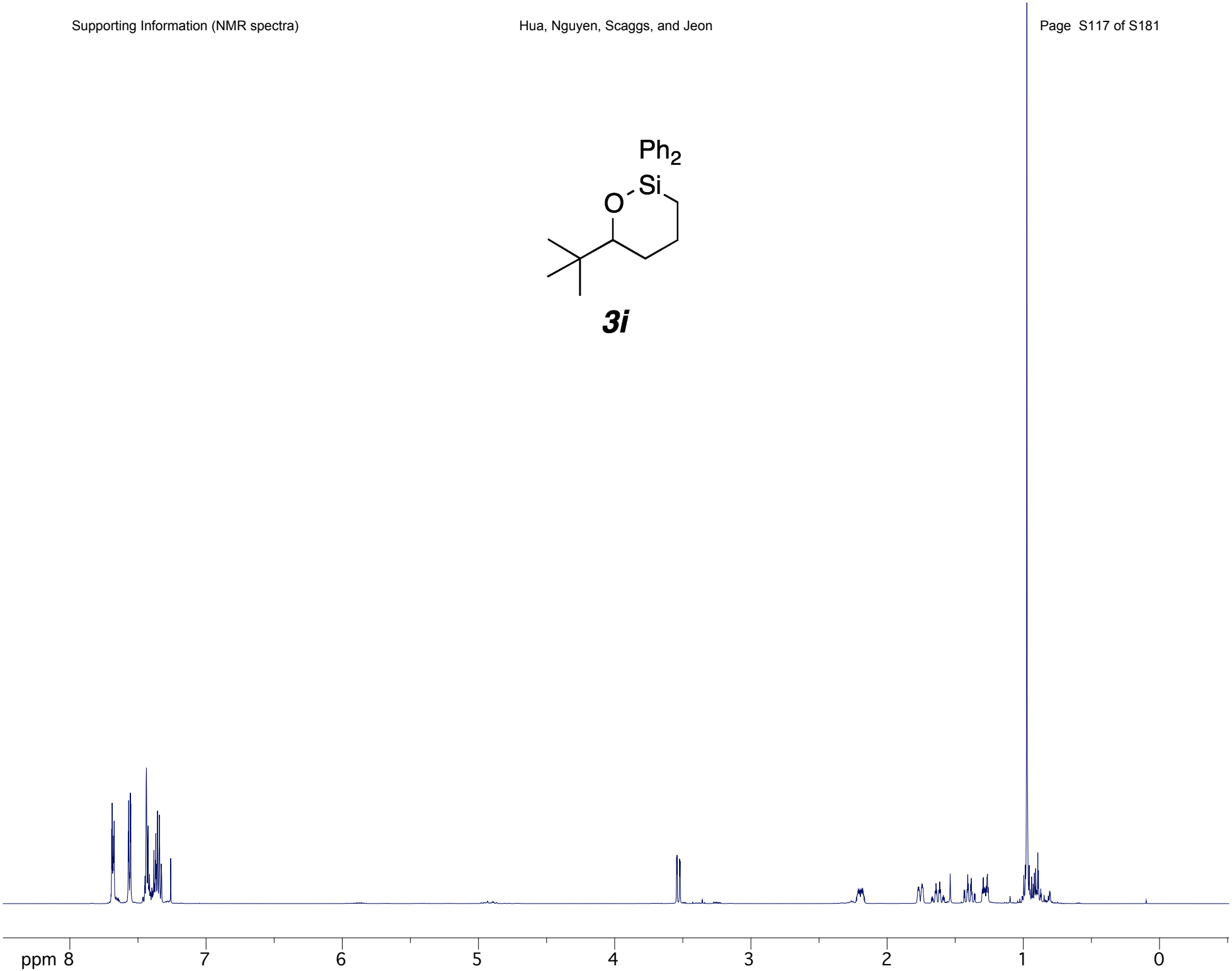
3h

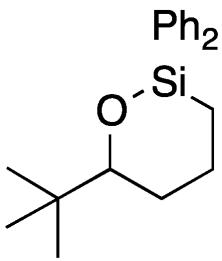


S#603188 (single pulse decoupled gated NOE) USER: delta -- DATE: Mar 14, 2013 06:13:15

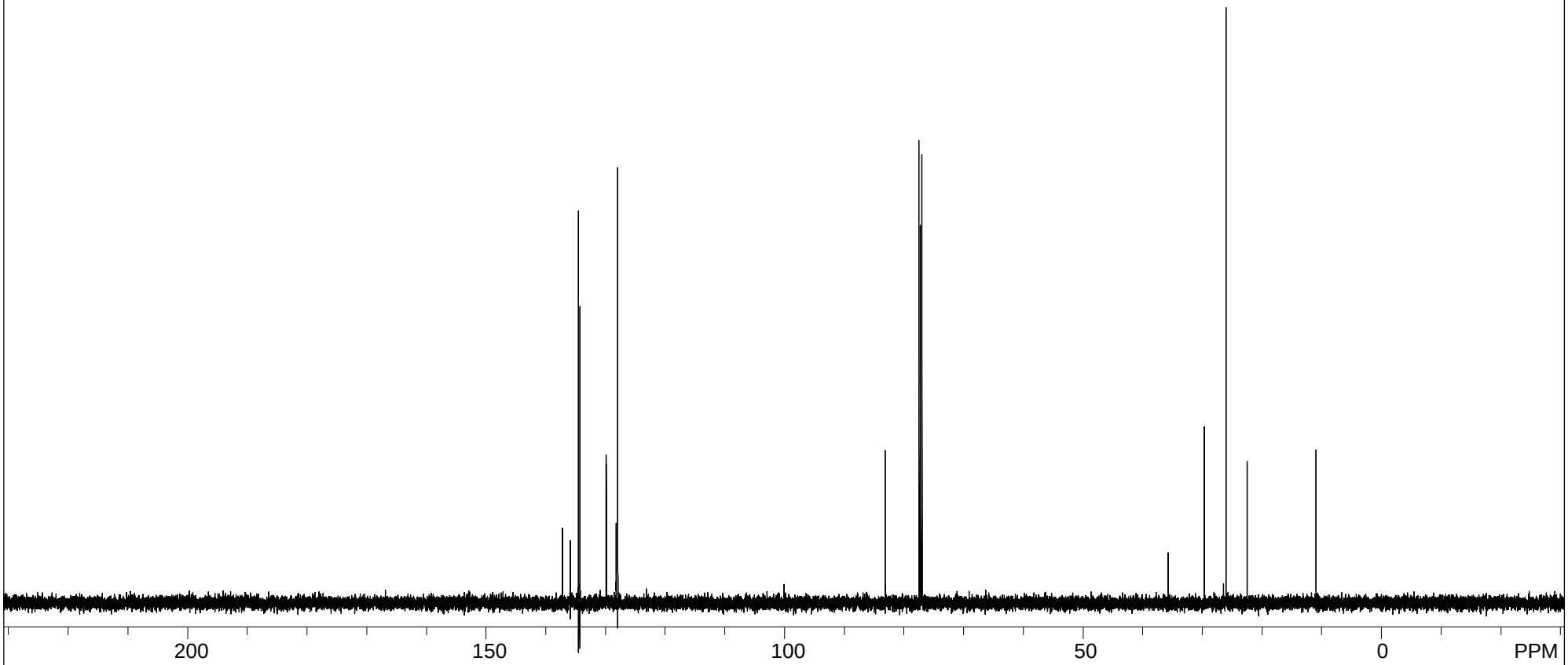
F1: 125.778	F2: 500.162	SW1: 39308		OF1: 12589.0		PTS1d: 32768	
EX: single_pulse_dec		PW: 3.6 us, 30deg	PD: 2.0 sec	NA: 72	LB: 0.0	Nuts - YH1035B_M1_1_iPr_13C-2_3h.jdf	

3i



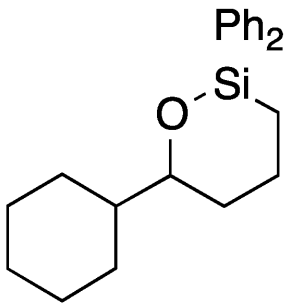


3i

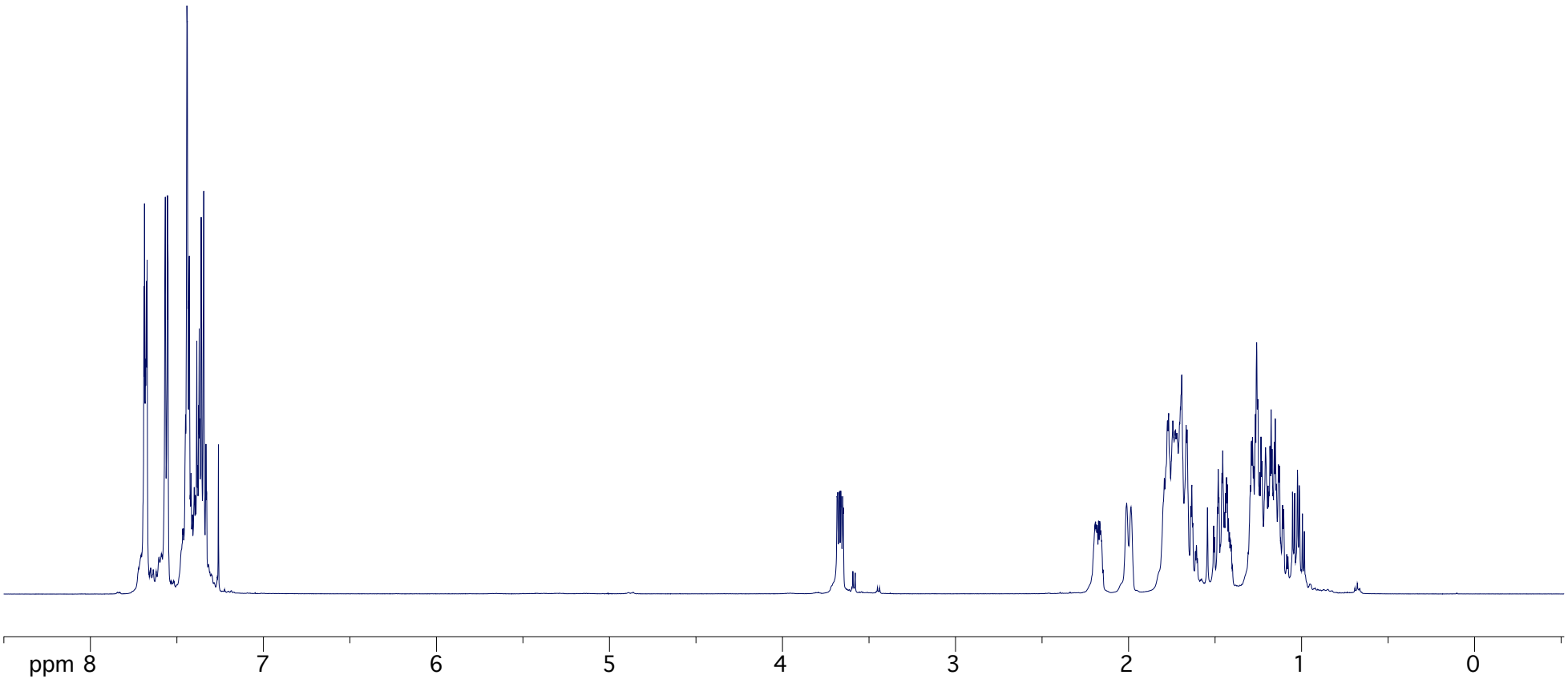


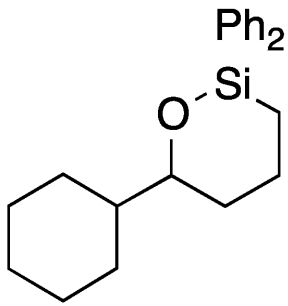
S#702139 (single pulse decoupled gated NOE) USER: delta -- DATE: Jul 31, 2012 09:25:13

F1: 125.778	F2: 500.162	SW1: 39308		OF1: 12591.4		PTS1d: 32768 , 32768
EX: single_pulse_dec		PW: 3.6 us, 30deg	PD: 2.0 sec	NA: 65	LB: 0.0	Nuts - WRS2085_2_tBut_C13-1_3i.jdf

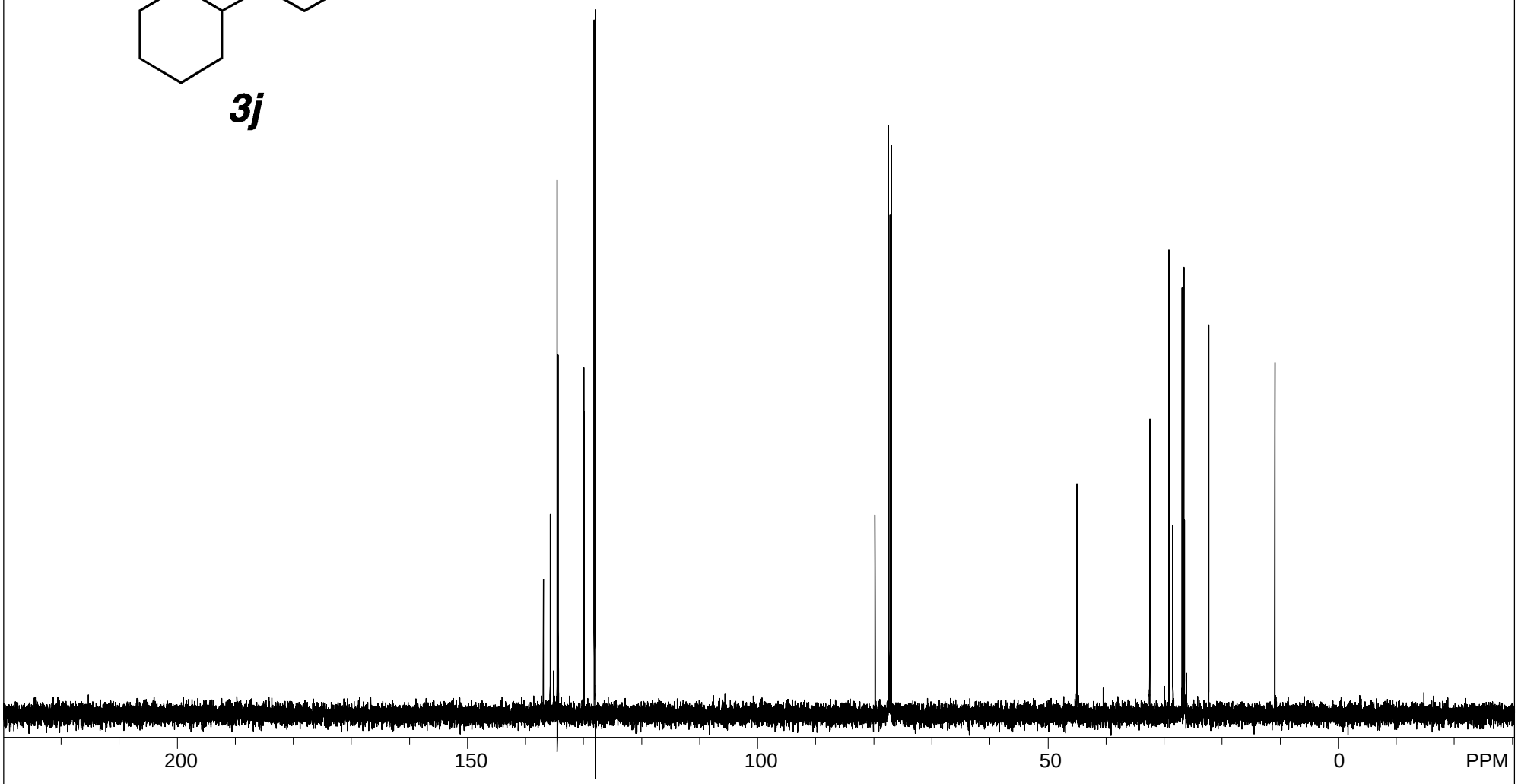


3j





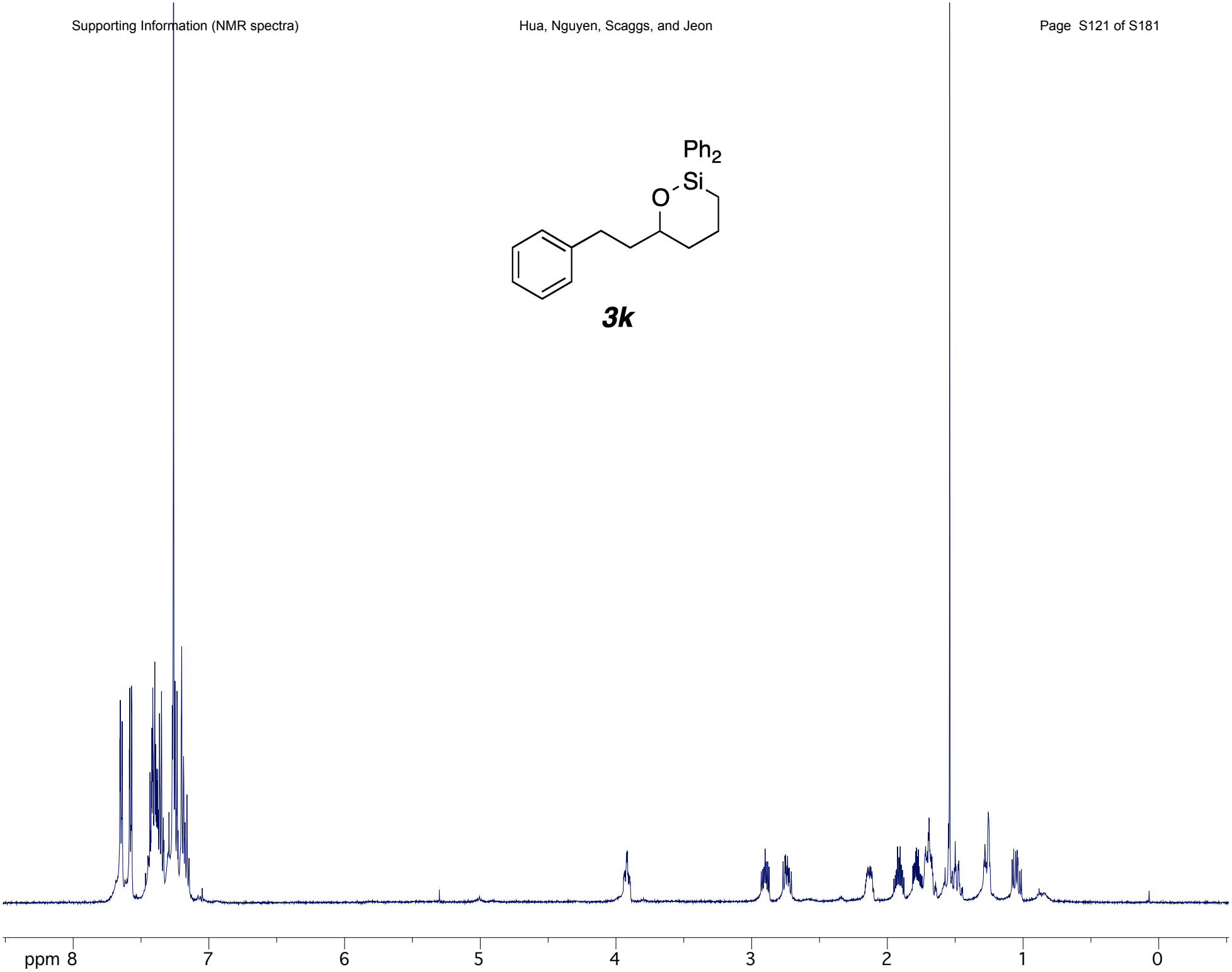
3j



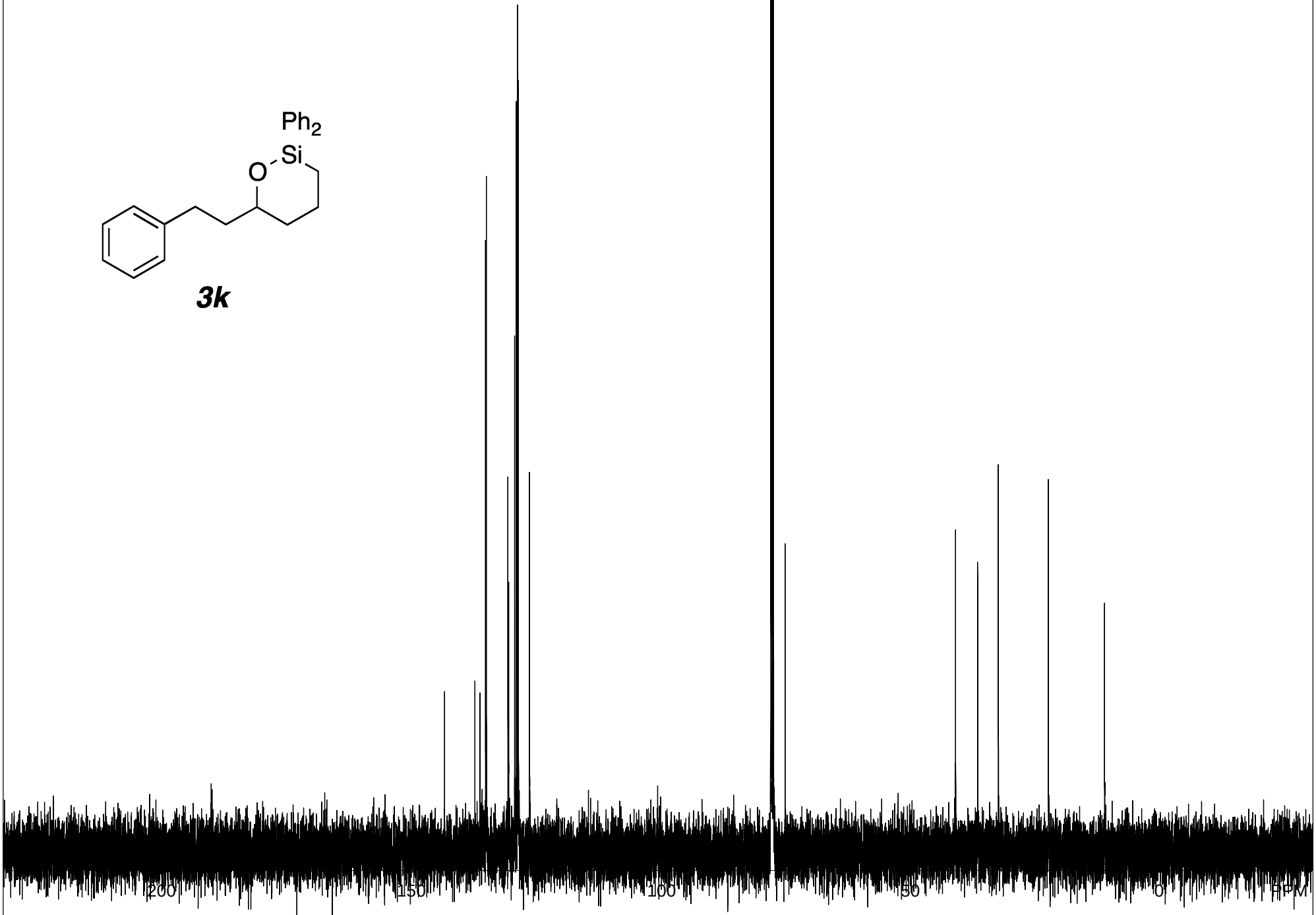
S#742655 (single pulse decoupled gated NOE) USER: delta -- DATE: Mar 2, 2013 11:06:07:

F1: 125.778	F2: 500.162	SW1: 39308		OF1: 12589.0		PTS1d: 32768 , 32768
EX: single_pulse_dec		PW: 3.6 us, 30deg	PD: 2.0 sec	NA: 83	LB: 0.0	Nuts - YH_6ring_Cy_13C-1_3j.jdf

3k

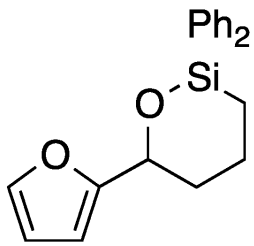


3k

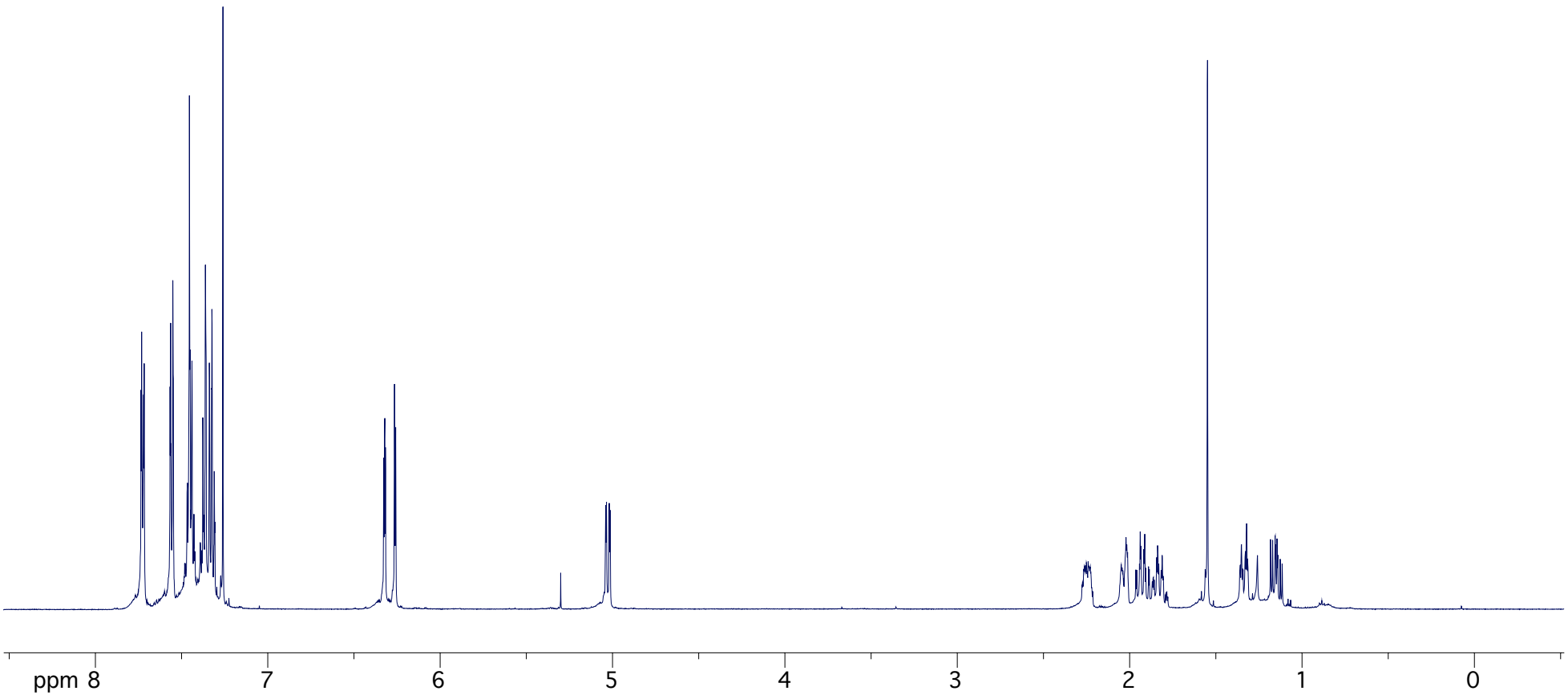


S#707332 (single pulse decoupled gated NOE) USER: delta -- DATE: Jul 31, 2012 09:36:07:

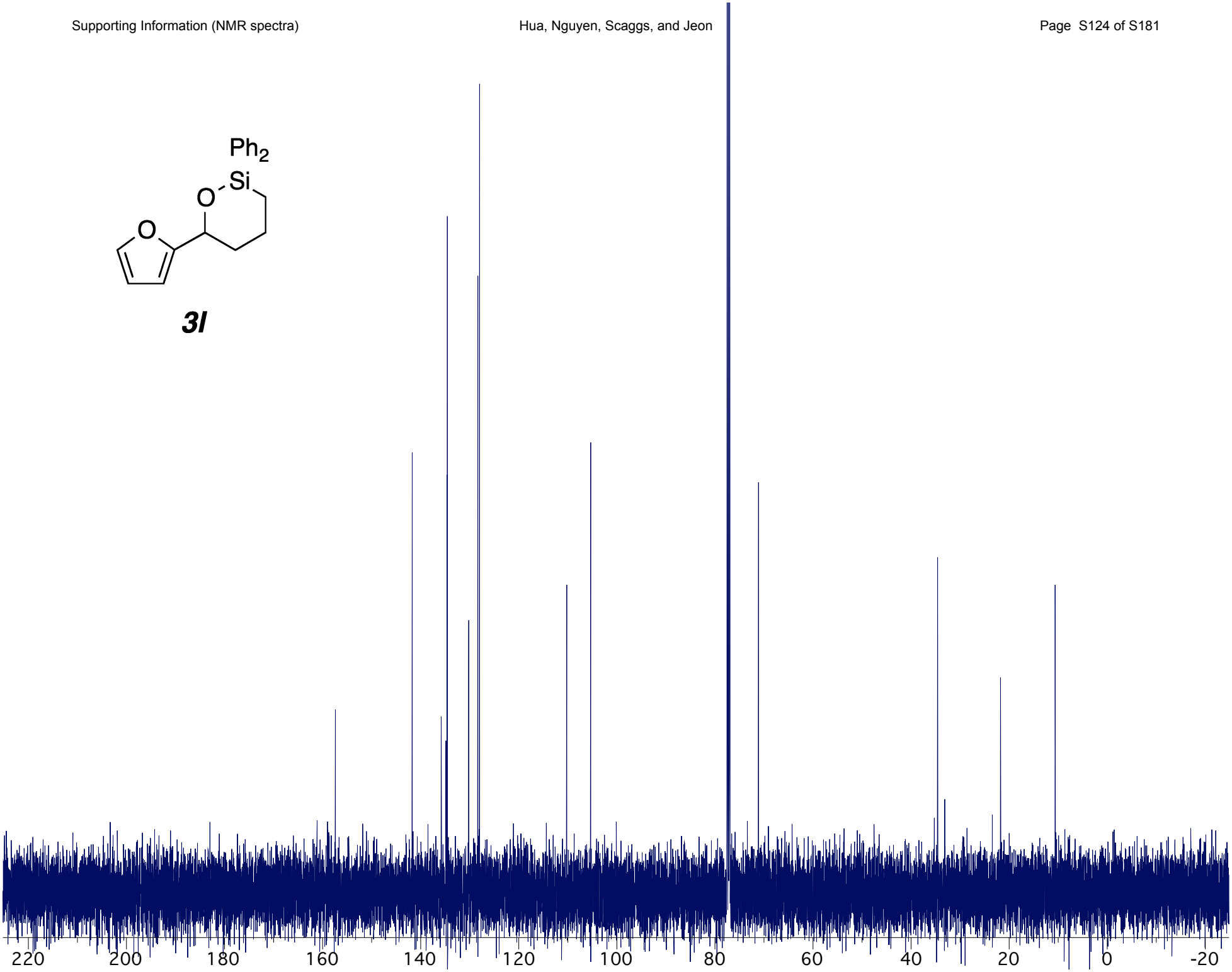
F1: 125.778	F2: 500.162	SW1: 39308		OF1: 12587.8		PTS1d: 32768 , 32768
EX: single_pulse_dec		PW: 3.6 us, 30deg	PD: 2.0 sec	NA: 113	LB: 0.0	Nuts - WRS2085_3_Benzyl_C13-1 2_3k.jdf



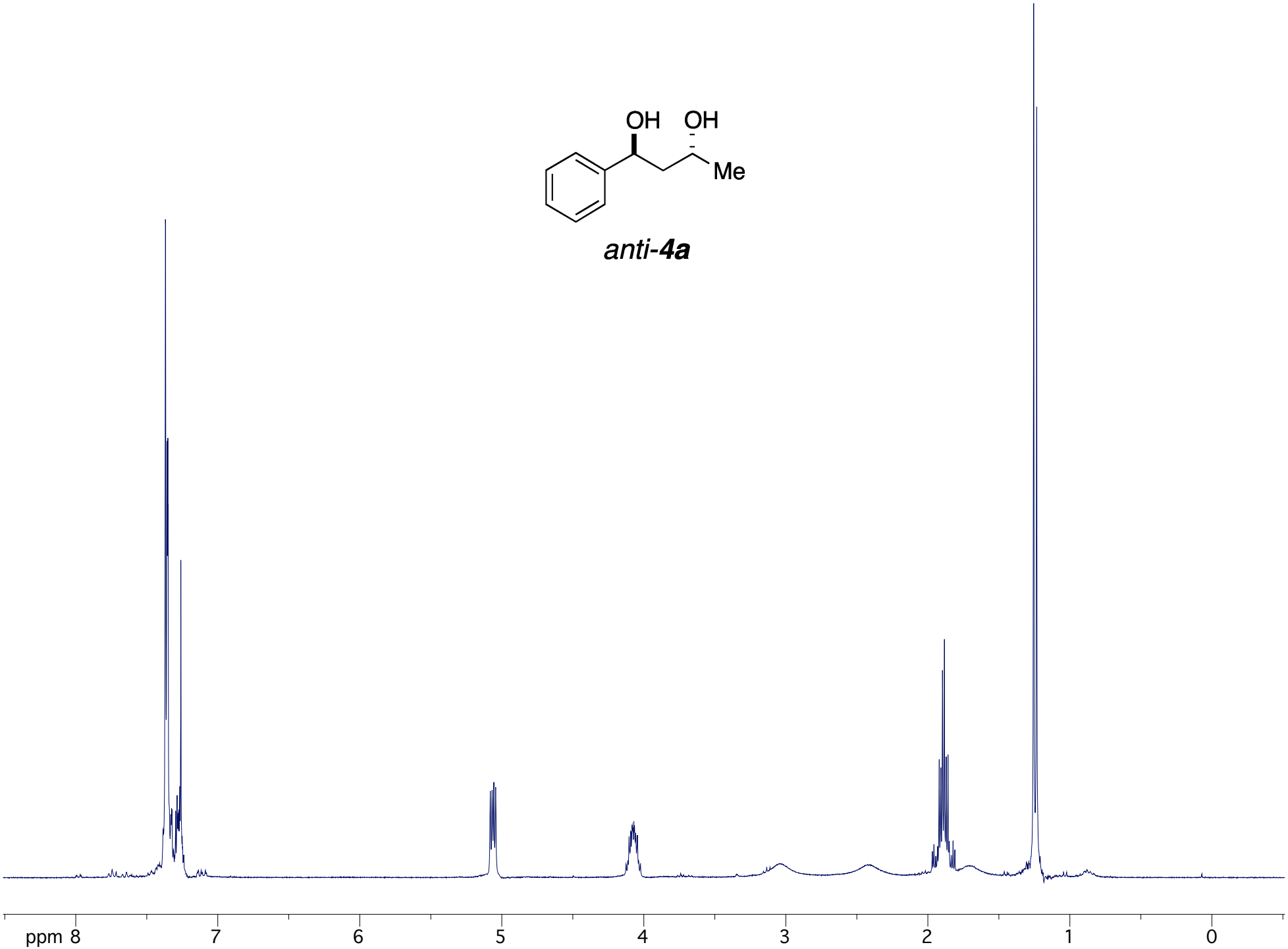
3l



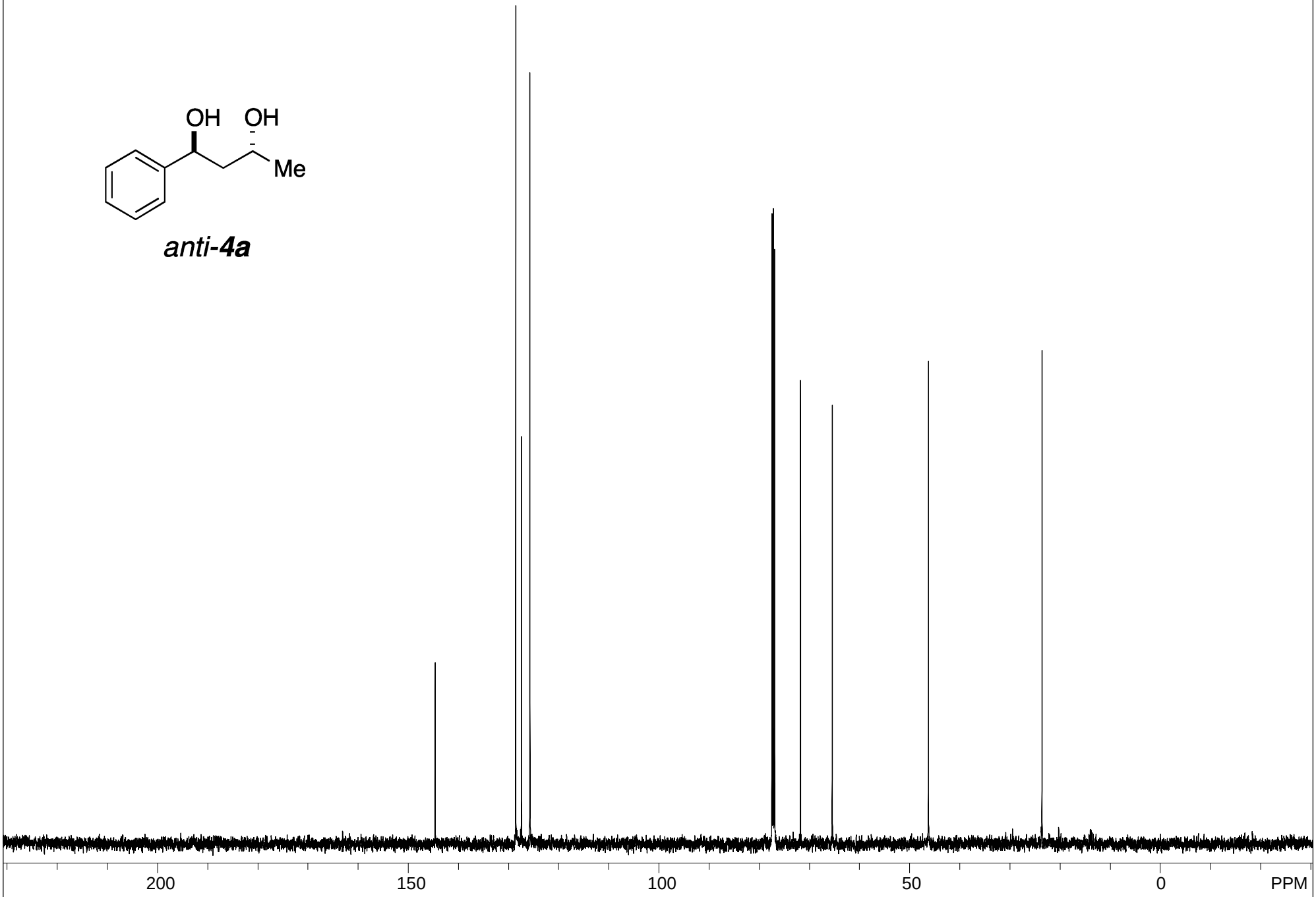
3l



*anti-**4a***



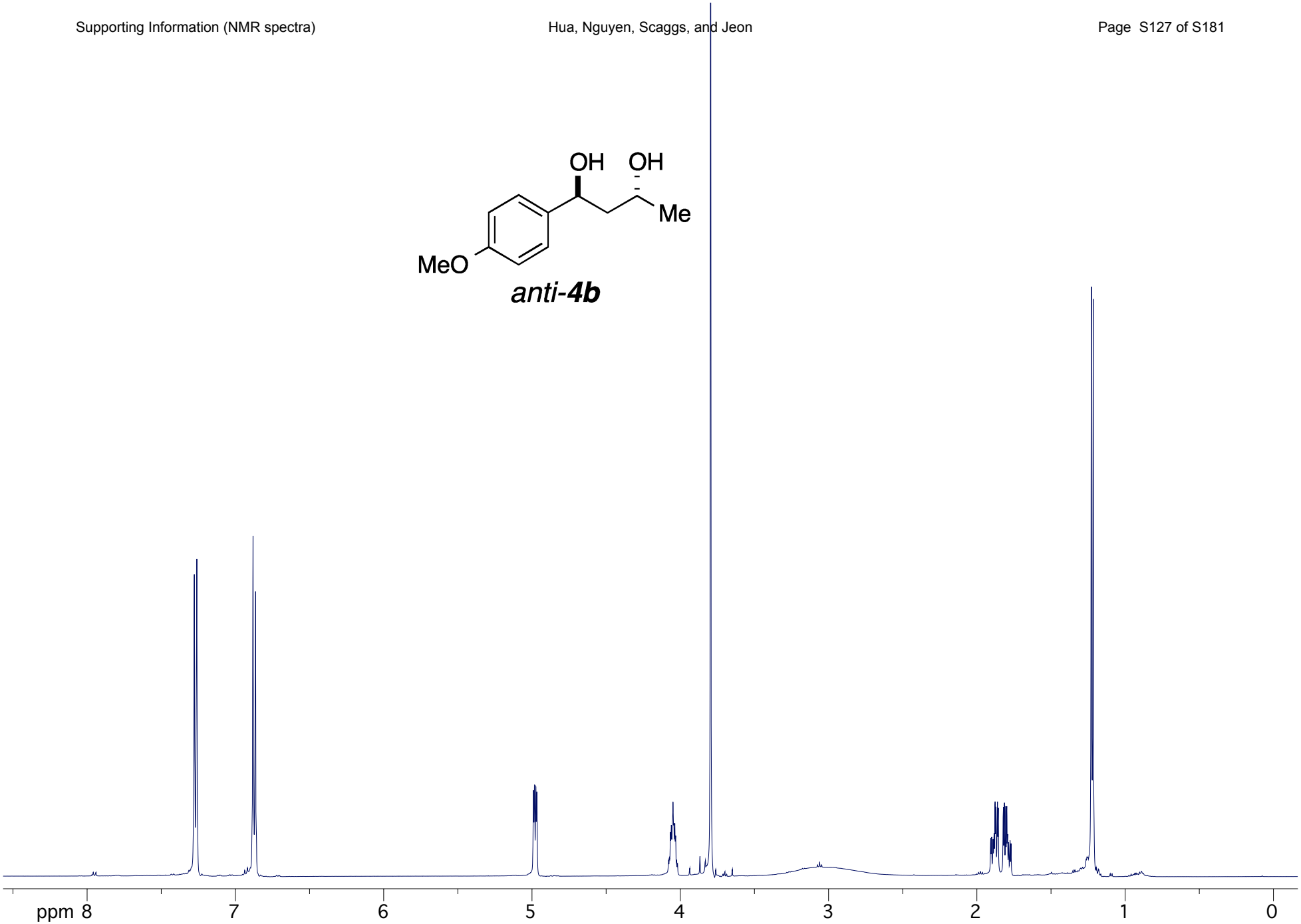
*anti-**4a***



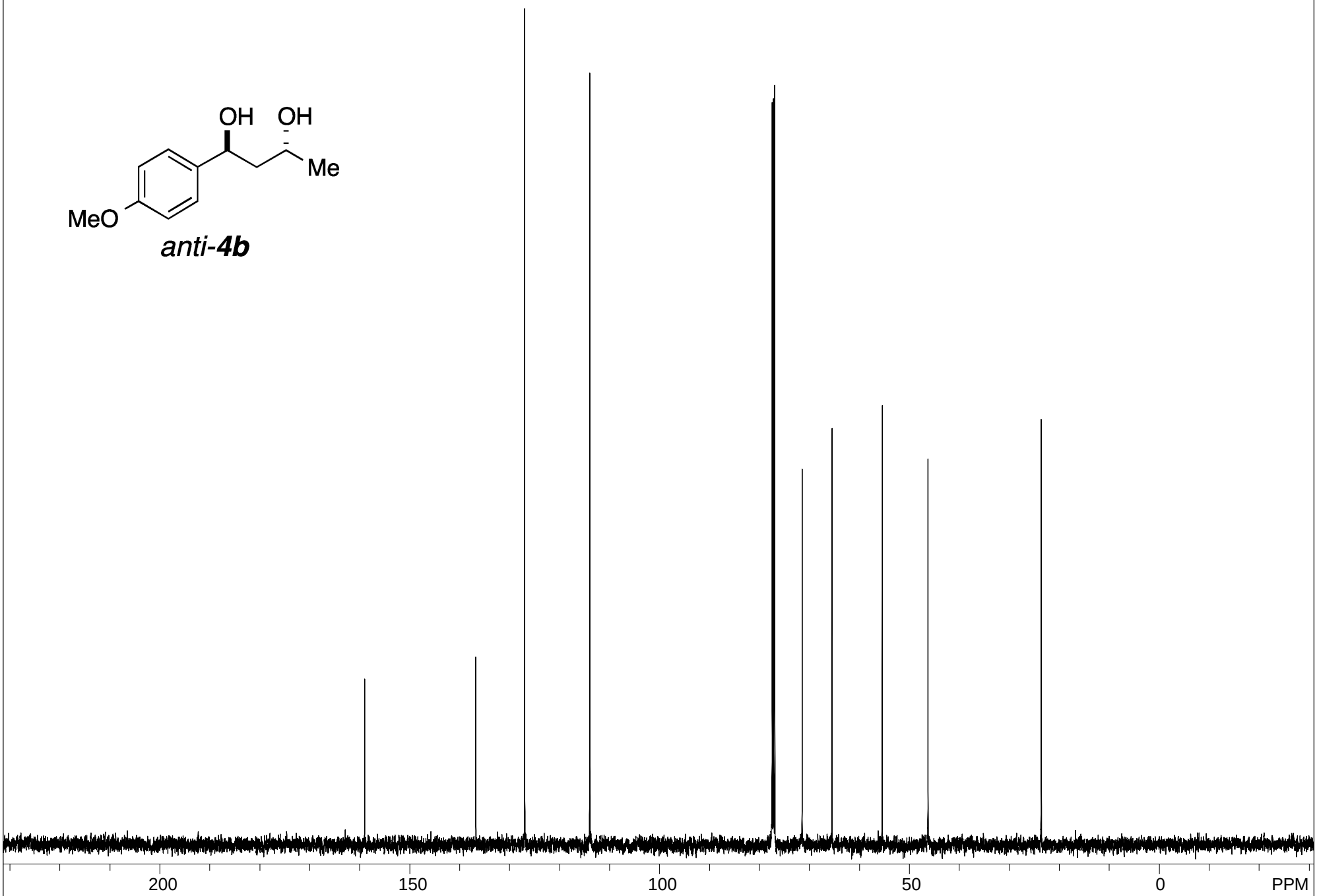
S#366442 (single pulse decoupled gated NOE) USER: delta -- DATE: Dec 11, 2012 00:44:40

F1: 125.778	F2: 500.162	SW1: 39308		OF1: 12586.6		PTS1d: 32768	
EX: single_pulse_dec		PW: 3.6 us, 30deg	PD: 2.0 sec	NA: 72	LB: 0.0	Nuts - JHJ1210_rxn6_13C_MMP_Ph-3_13a.jdf	

*anti-**4b***



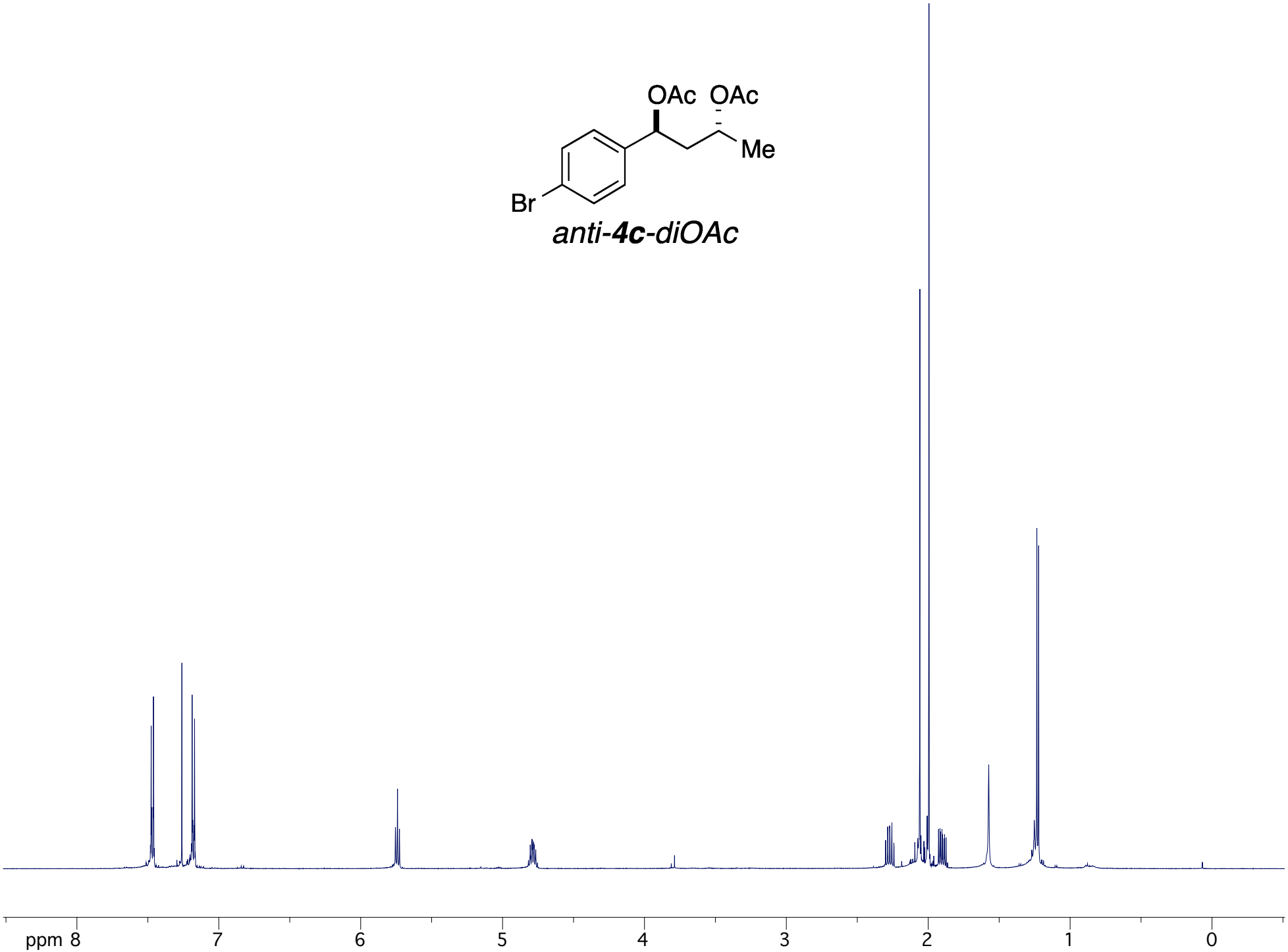
*anti-**4b***



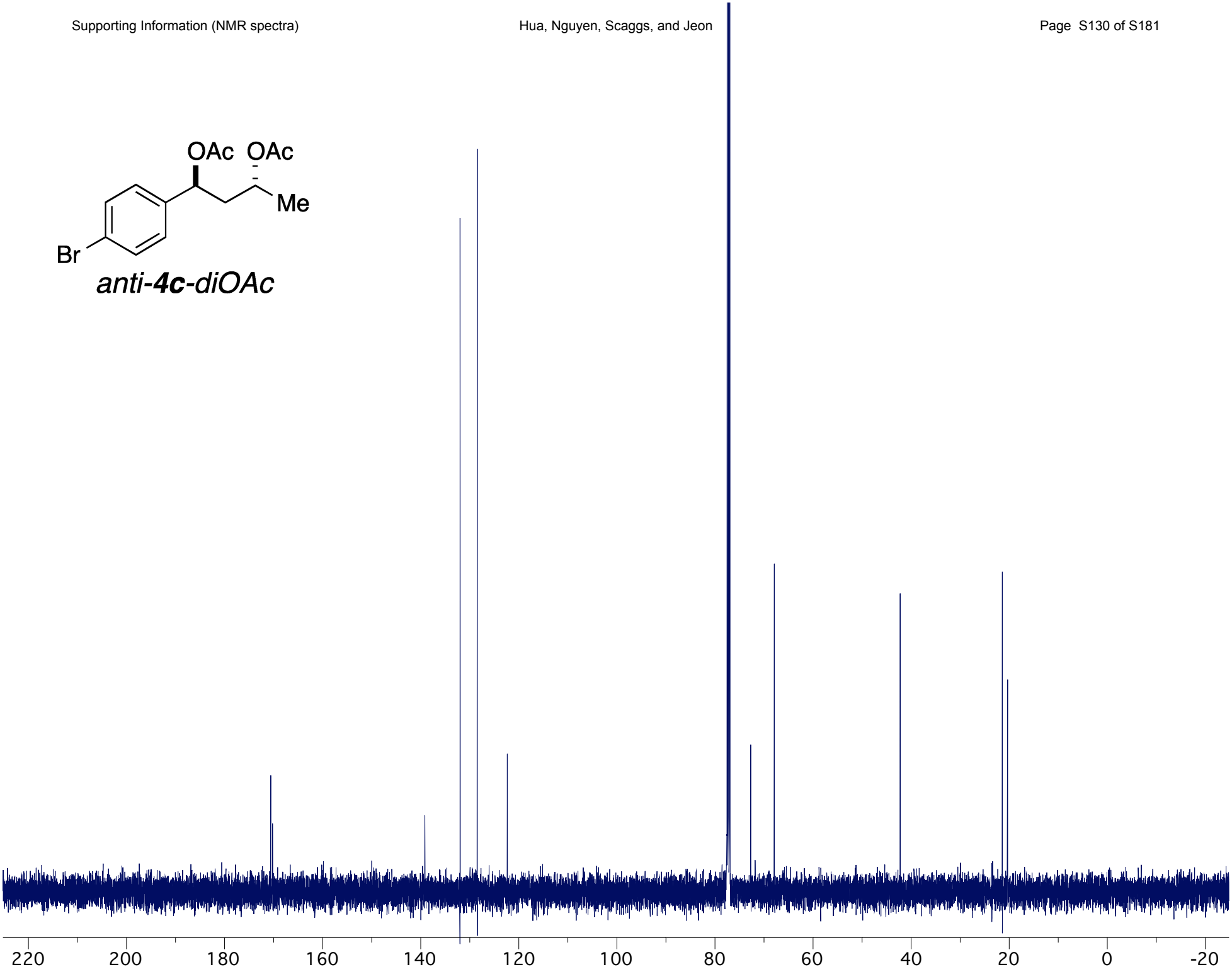
S#377924 (single pulse decoupled gated NOE) USER: delta -- DATE: Dec 11, 2012 01:03:53

F1: 125.778	F2: 500.162	SW1: 39308		OF1: 12587.8		PTS1d: 32768	
EX: single_pulse_dec		PW: 3.6 us, 30deg	PD: 2.0 sec	NA: 74	LB: 0.0	Nuts - JHJ1210_rxn9_13C_MMP_pMeO_13b.jdf	

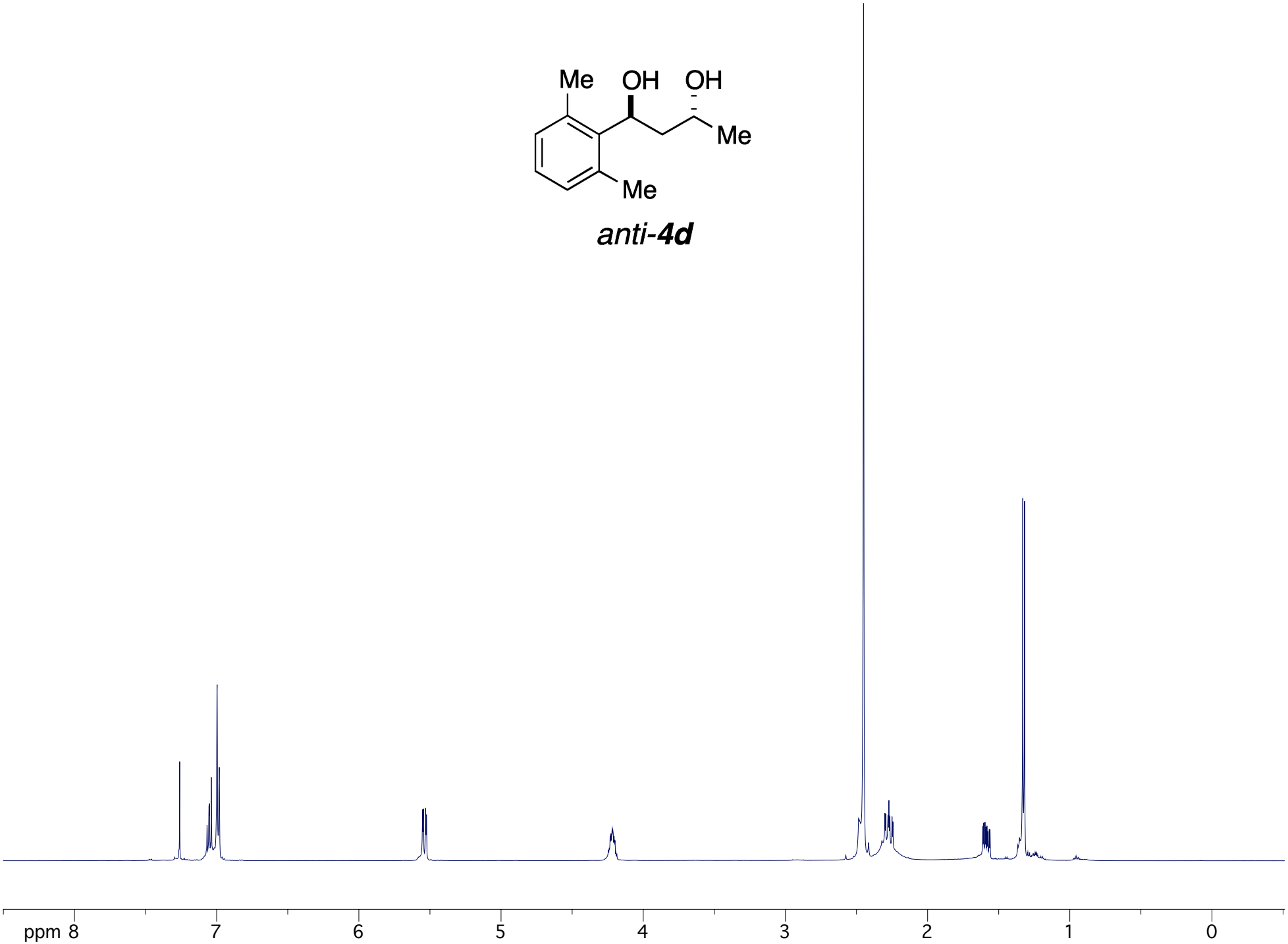
*anti-**4c**-diOAc*



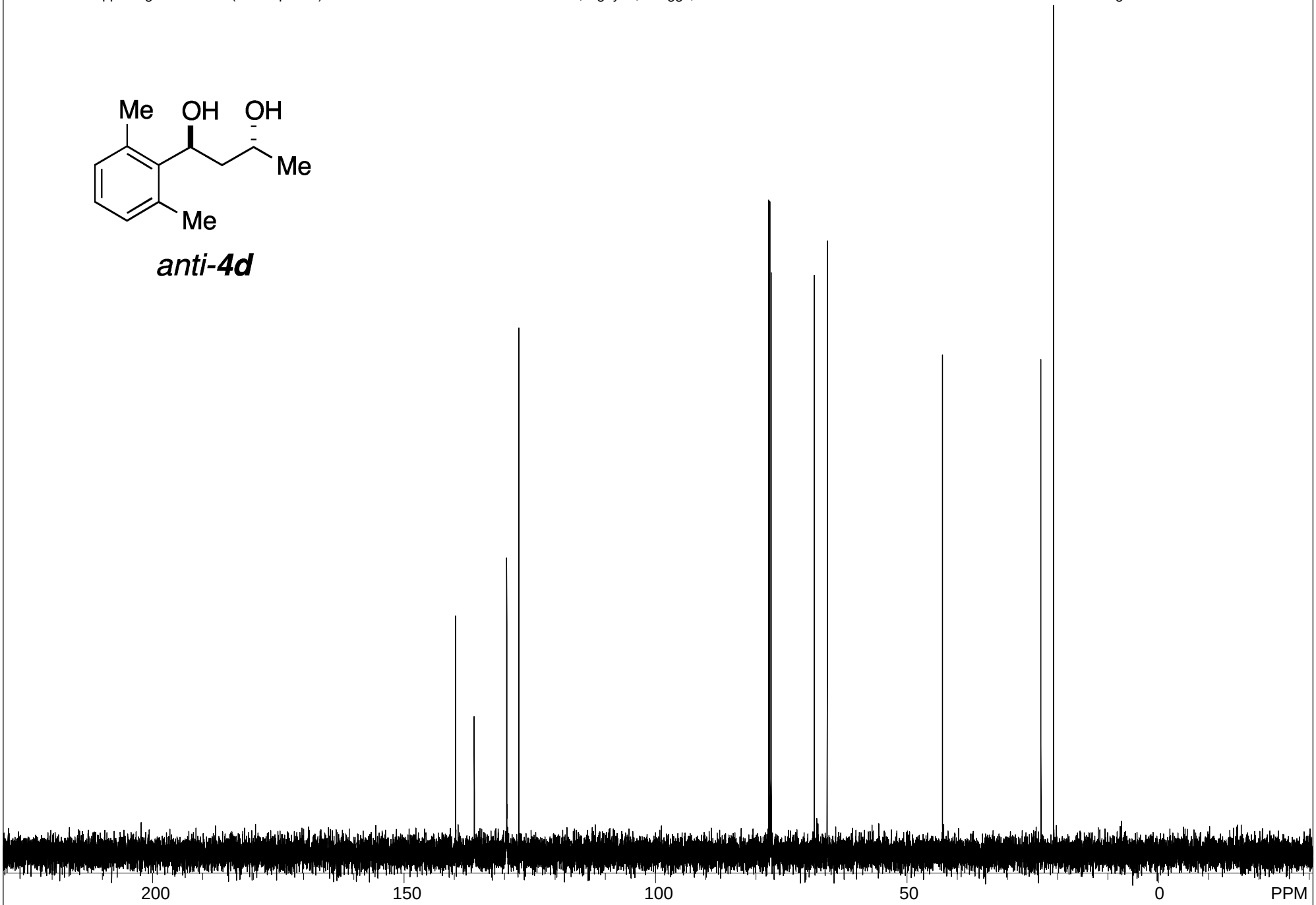
*anti-**4c**-diOAc*



*anti-**4d***



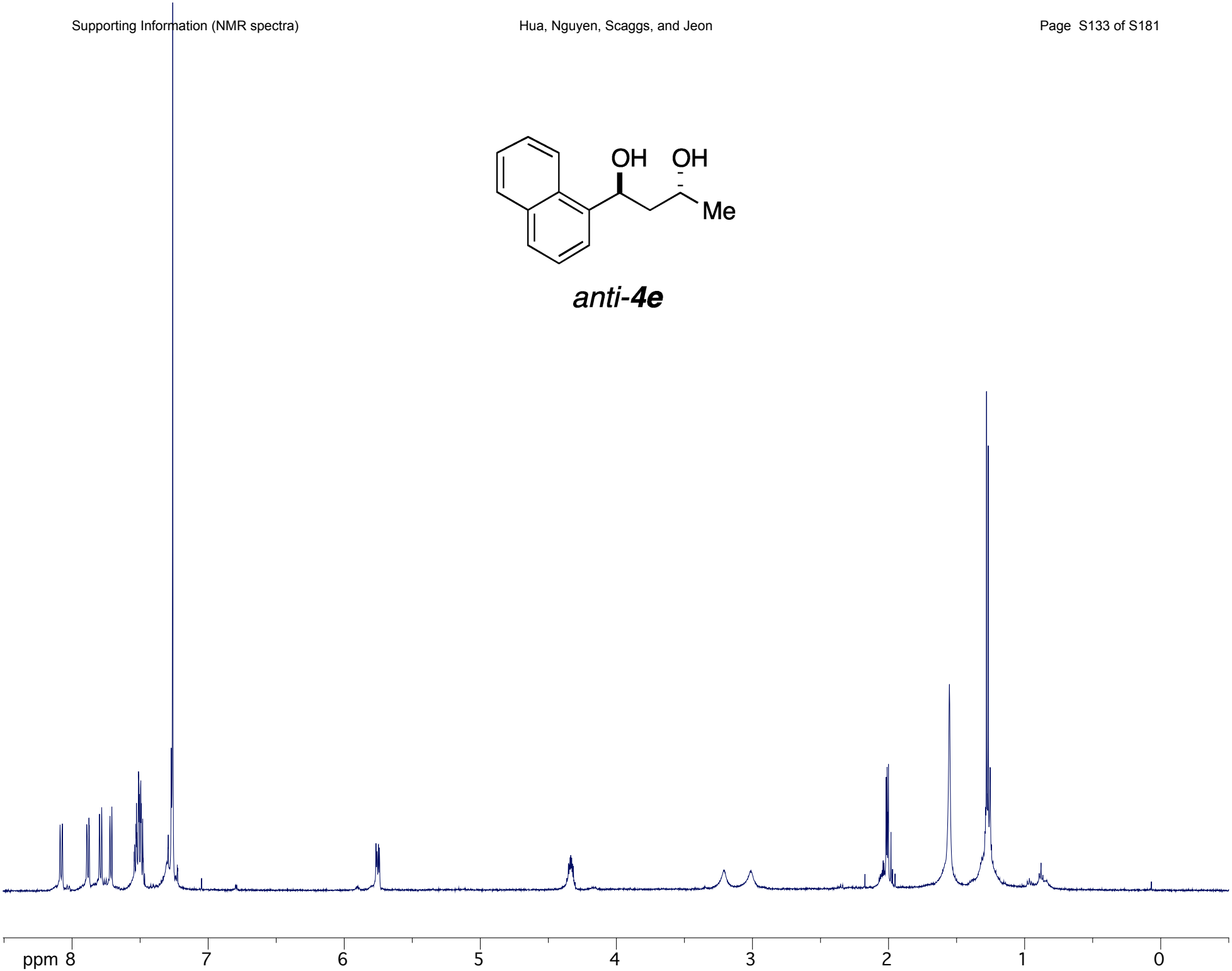
anti-4d



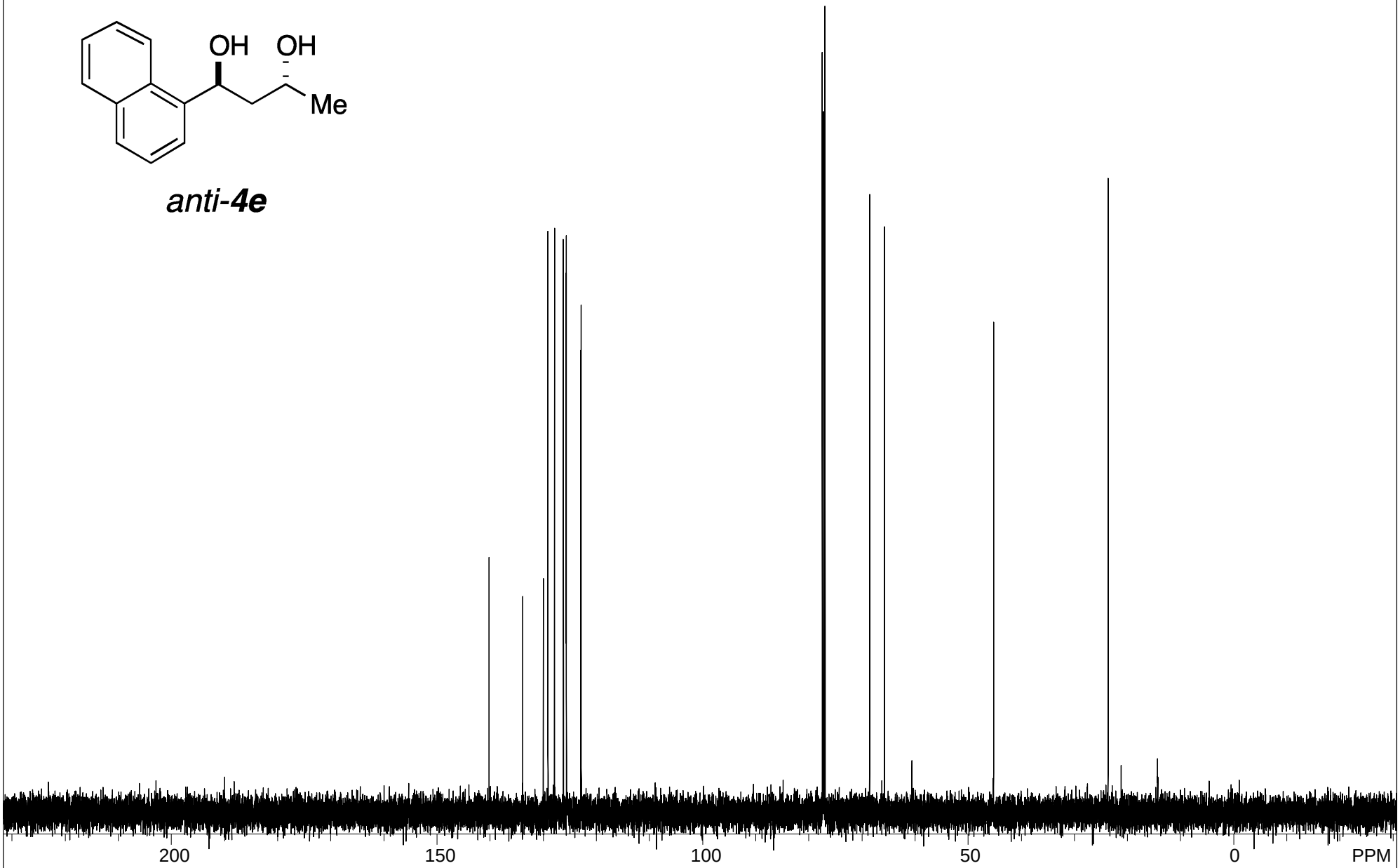
S#440386 (single pulse decoupled gated NOE) USER: delta -- DATE: Dec 9, 2012 02:46:59

F1: 125.778	F2: 500.162	SW1: 39308		OF1: 12585.4		PTS1d: 32768 , 32768
EX: single_pulse_dec		PW: 3.6 us, 30deg	PD: 2.0 sec	NA: 54	LB: 0.0	Nuts - JHJ1210_rxn11_13C_MP_diMePh-1_13d.jdf

*anti-**4e***



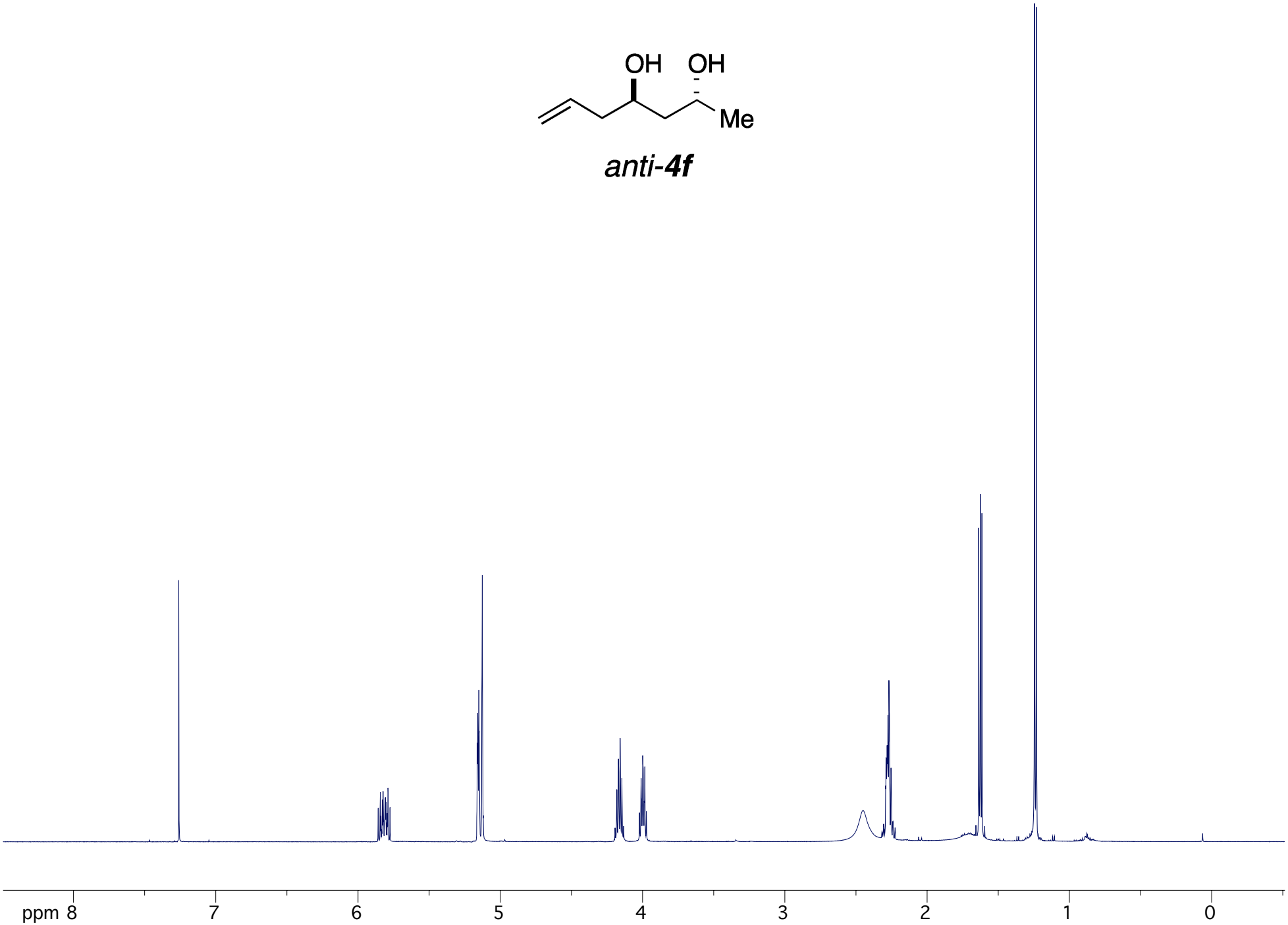
*anti-**4e***

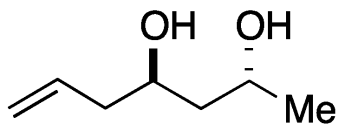


S#455903 (single pulse decoupled gated NOE) USER: delta -- DATE: Dec 9, 2012 03:12:56

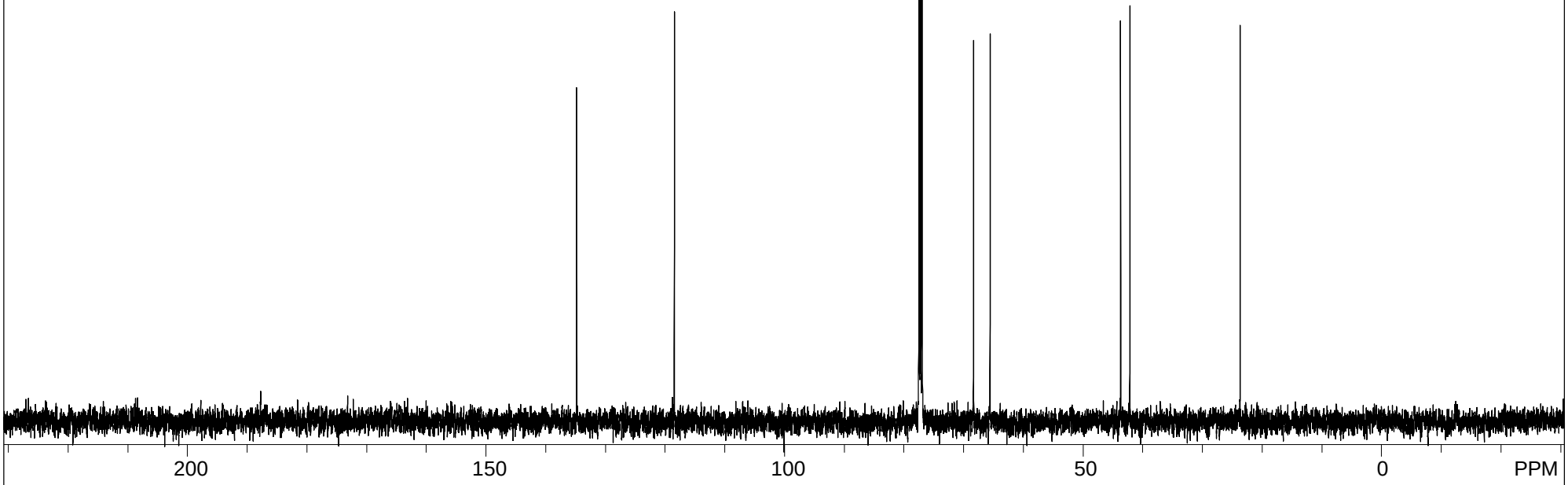
F1: 125.778	F2: 500.162	SW1: 39308		OF1: 12585.4		PTS1d: 32768 , 32768
EX: single_pulse_dec		PW: 3.6 us, 30deg	PD: 2.0 sec	NA: 55	LB: 0.0	Nuts - HJ1210_rxn5_13C_MMP_naph_13e.jdf

*anti-**4f***





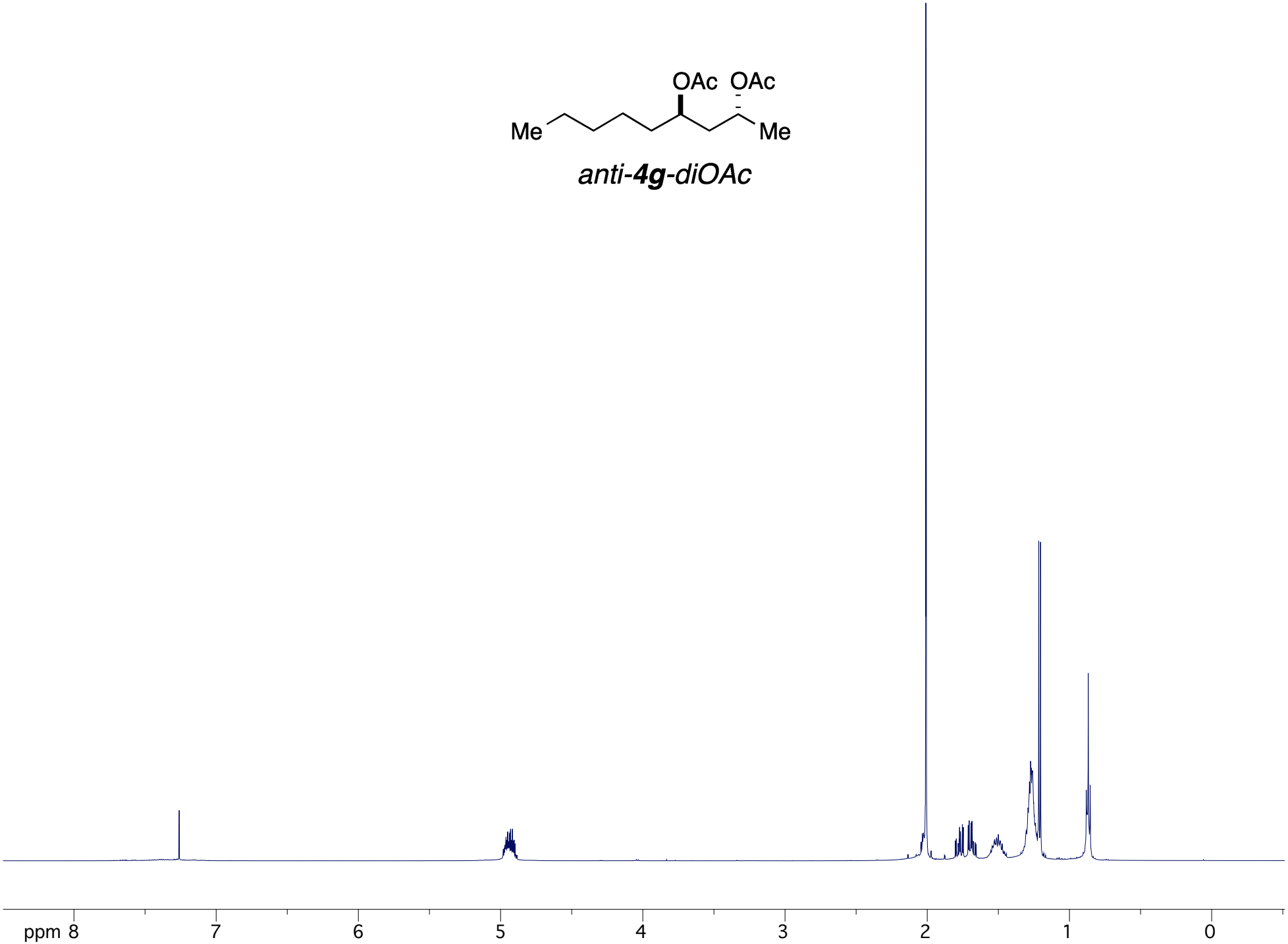
anti-4f

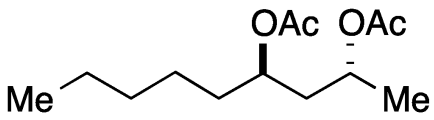


S#639319 (single pulse decoupled gated NOE) USER: delta -- DATE: Jul 5, 2012 07:41:31

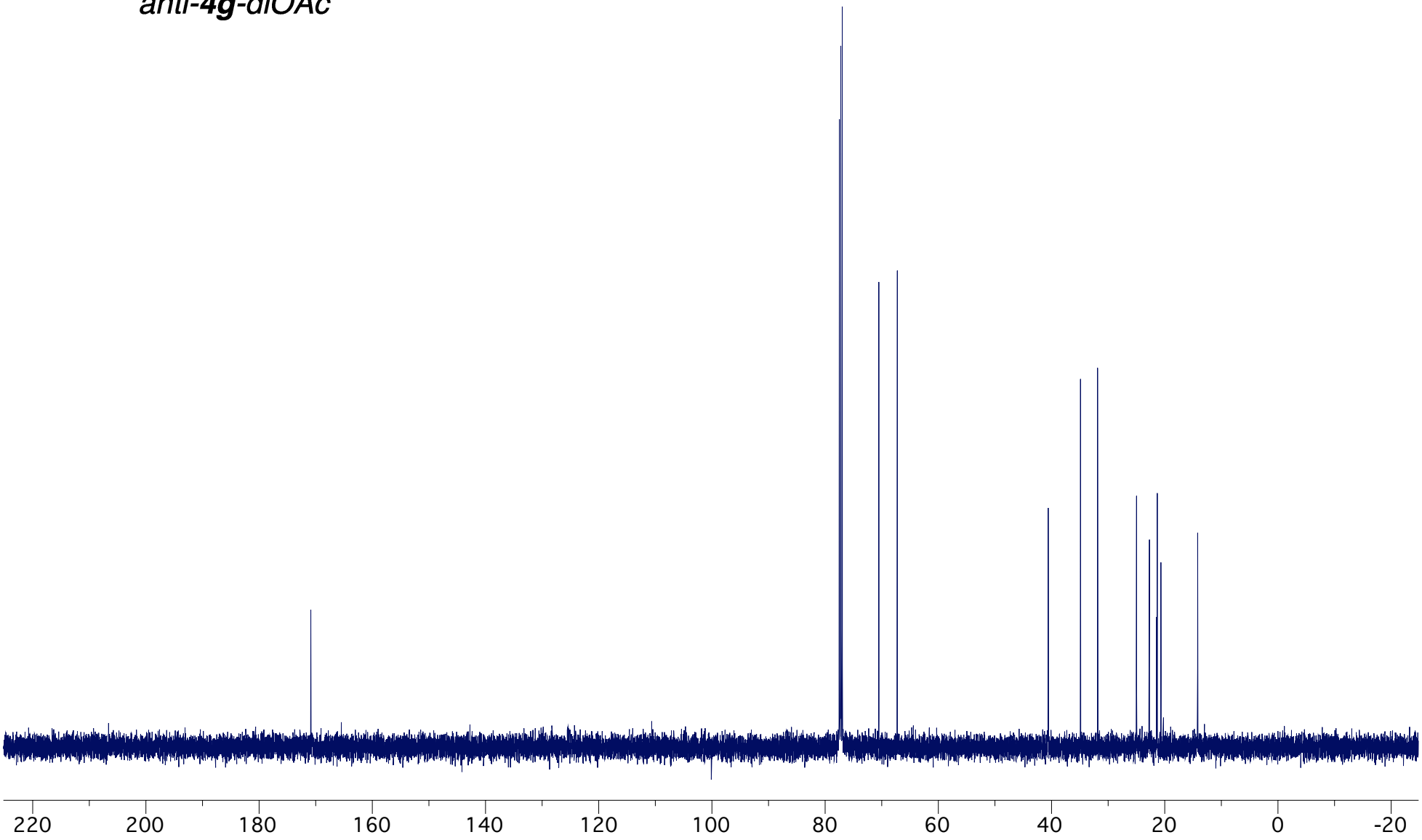
F1: 125.778	F2: 500.162	SW1: 39308		OF1: 12591.4		PTS1d: 32768	
EX: single_pulse_dec		PW: 3.6 us, 30deg	PD: 2.0 sec	NA: 91	LB: 0.0	Nuts - JHJ1152_13C_M_oxidation-2 2_13f.jdf	

*anti-**4g**-diOAc*

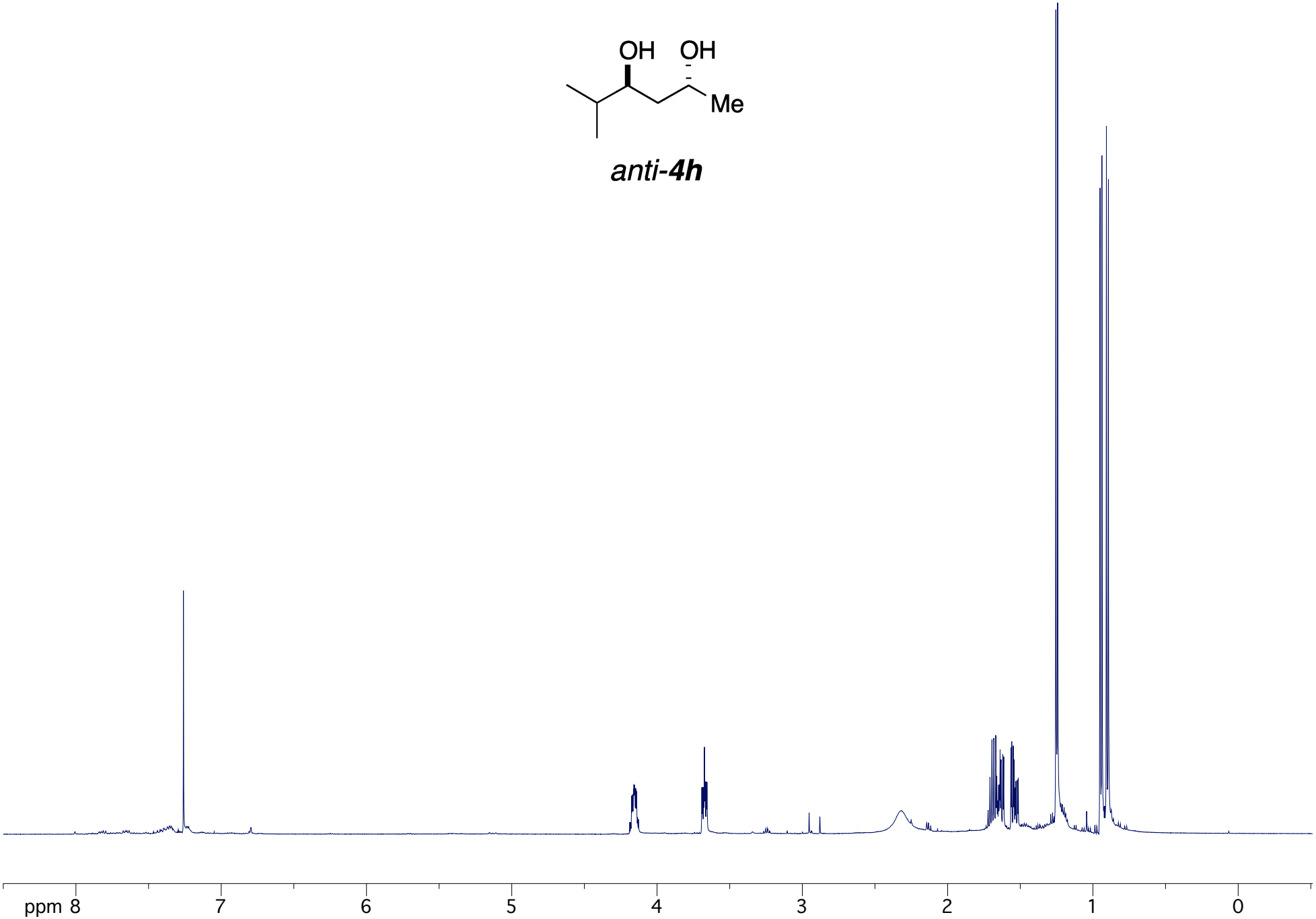


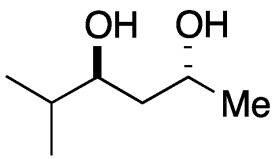


*anti-**4g**-diOAc*

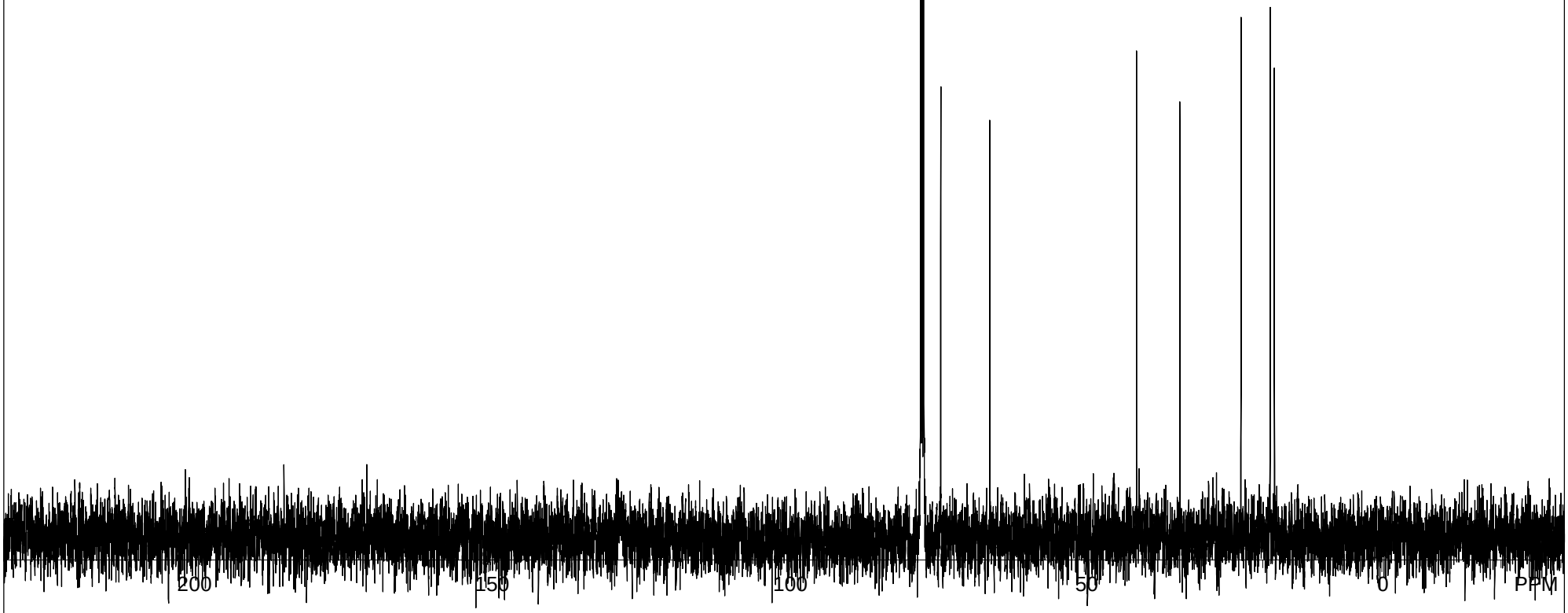


*anti-**4h***





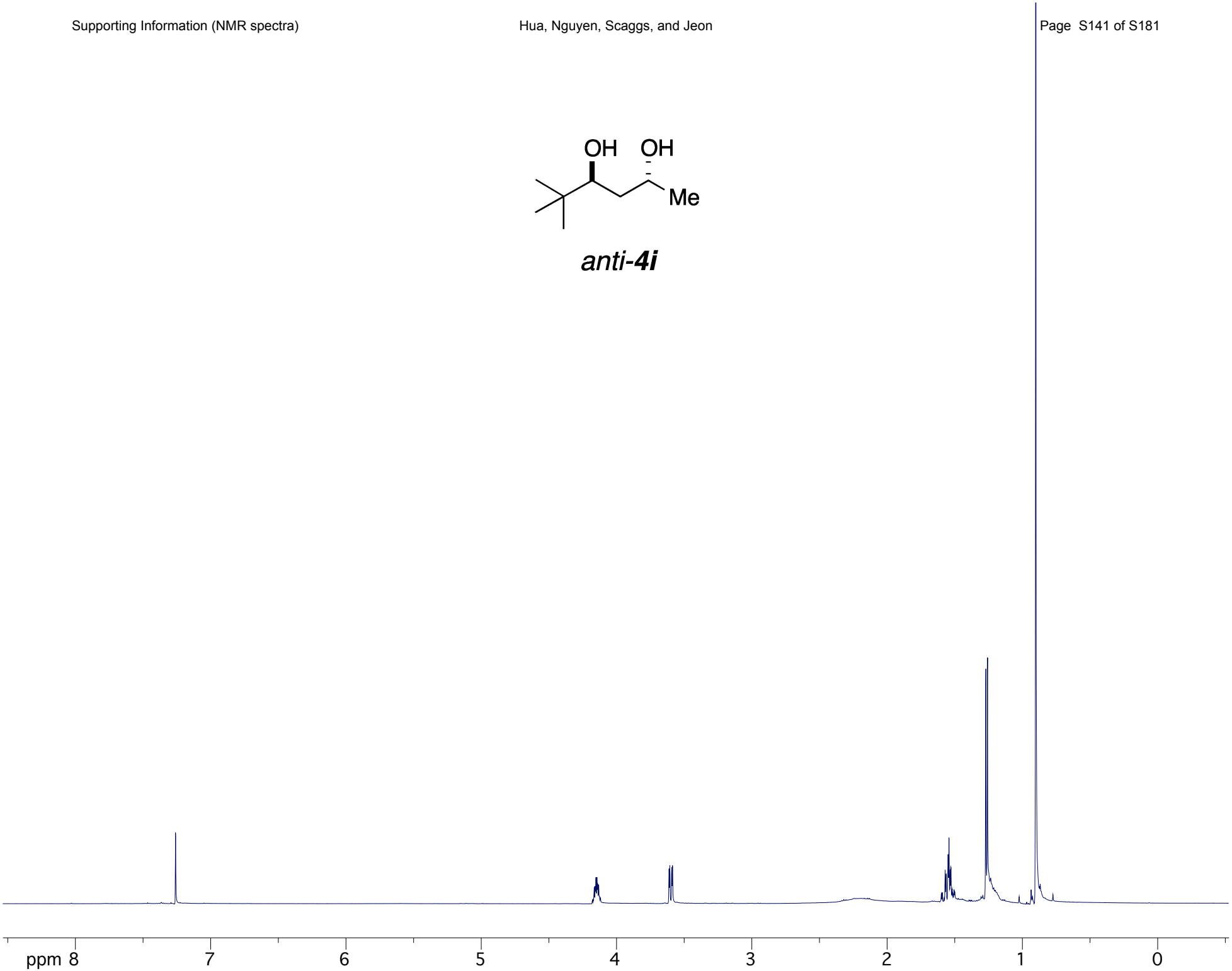
anti-4h



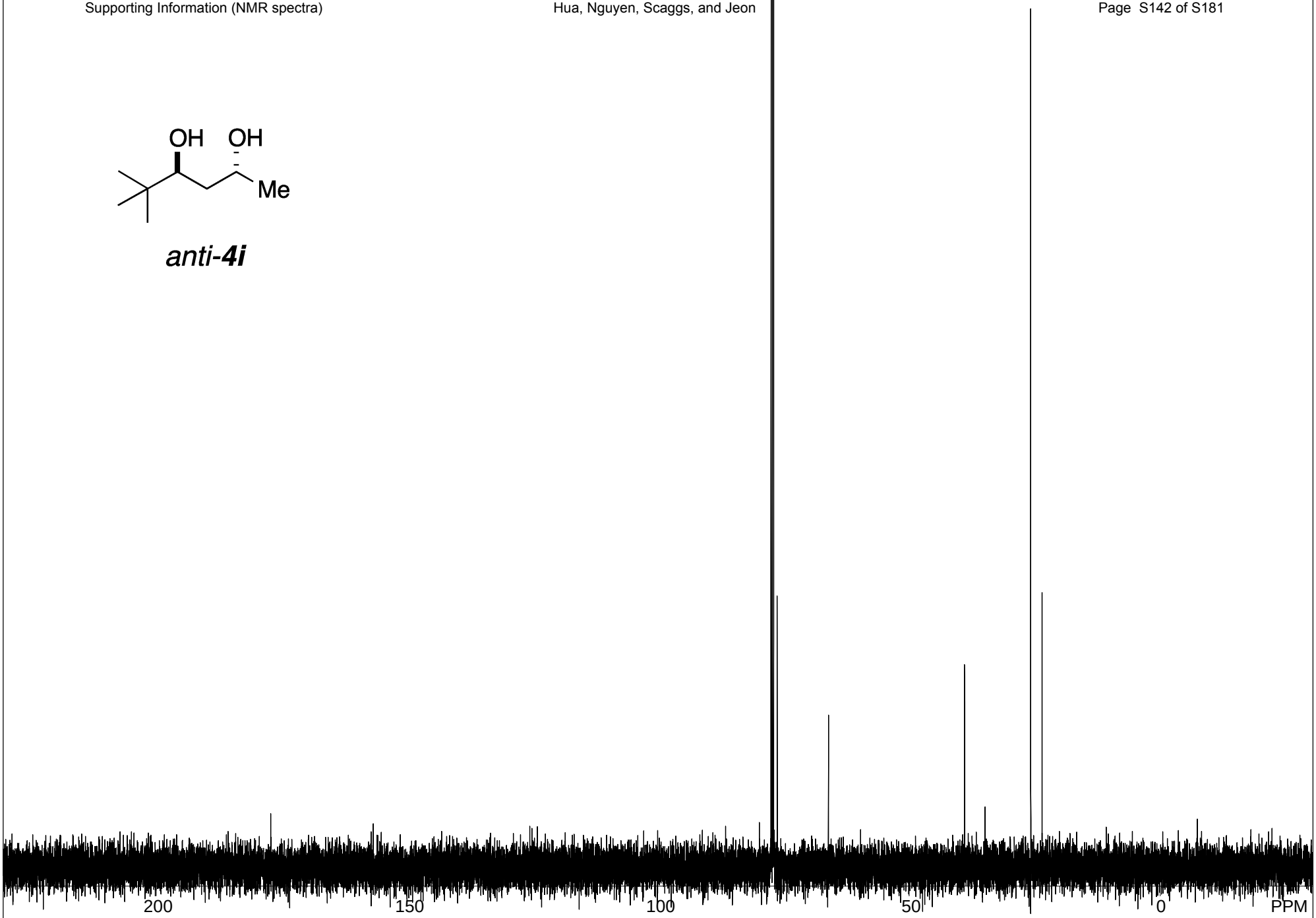
S#467562 (single pulse decoupled gated NOE) USER: delta -- DATE: Nov 20, 2012 03:31:16

F1: 125.778	F2: 500.162	SW1: 39308		OF1: 12592.6		PTS1d: 32768	
EX: single_pulse_dec		PW: 3.6 us, 30deg	PD: 2.0 sec	NA: 60	LB: 0.0	Nuts - JHJ1207_exp1_M_iPr_13h.jdf	

*anti-**4i***



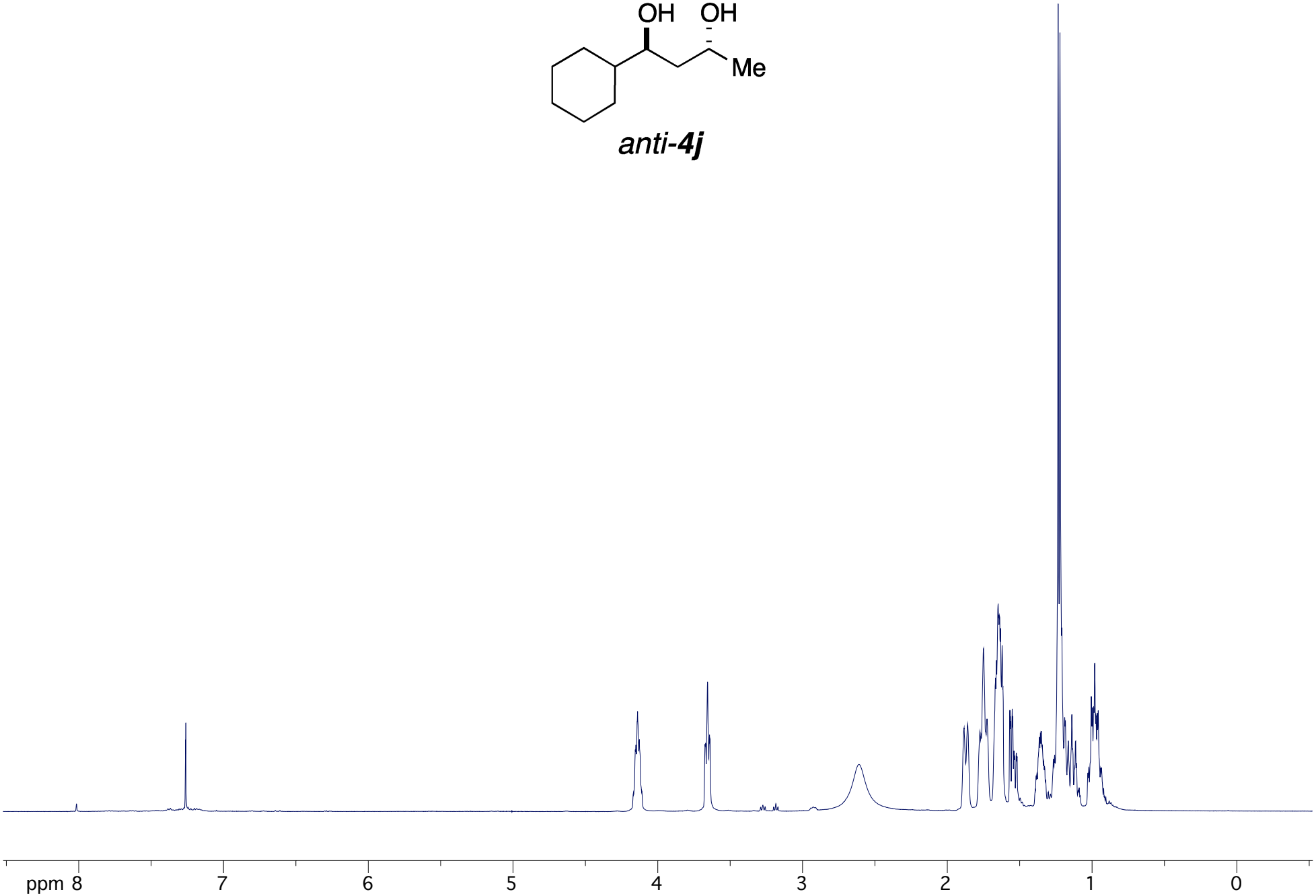
*anti-**4i***



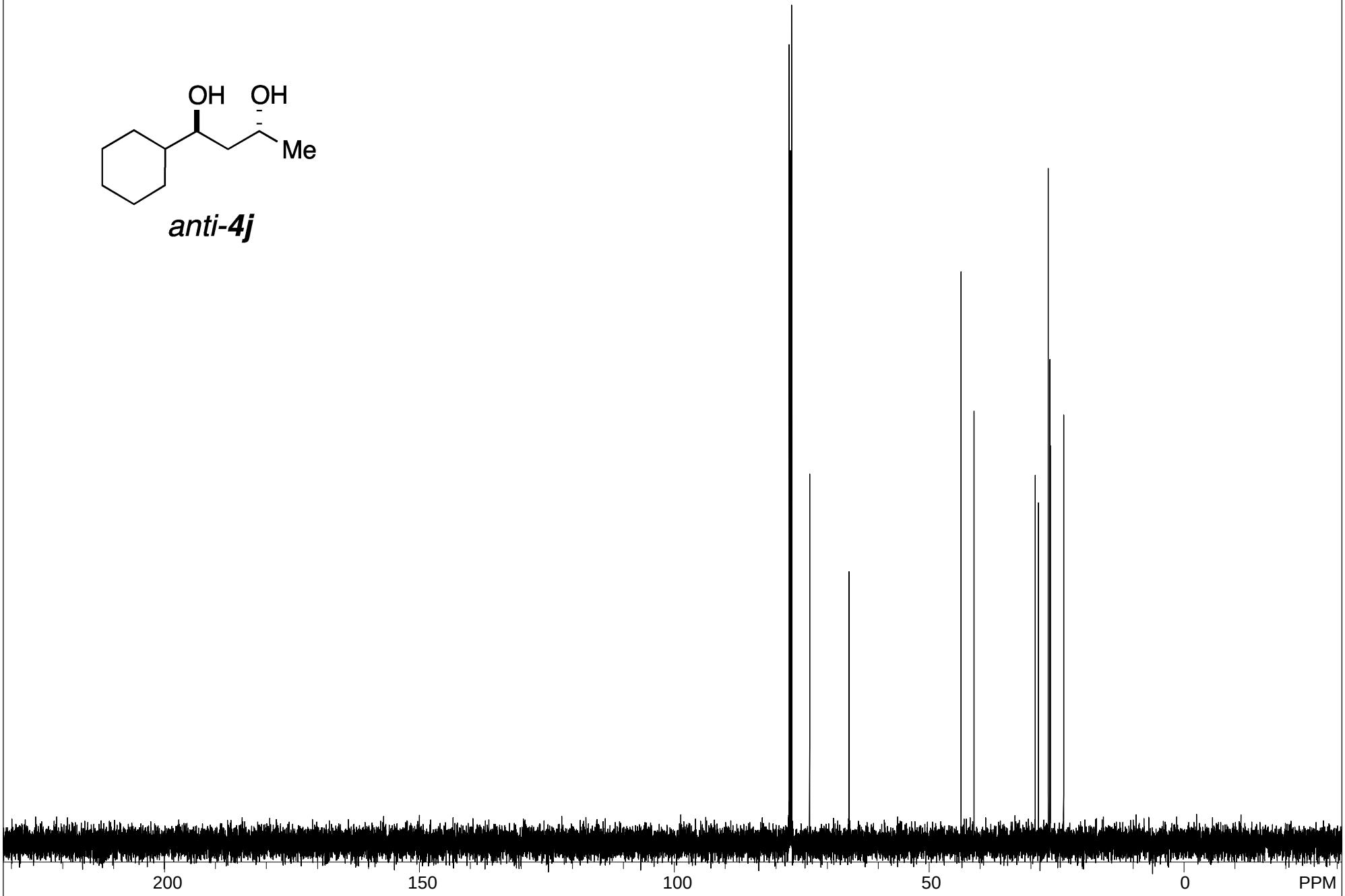
S#435554 (single pulse decoupled gated NOE) USER: delta -- DATE: Dec 6, 2012 02:42:58

F1: 125.778	F2: 500.162	SW1: 39308		OF1: 12592.6		PTS1d: 32768 , 32768
EX: single_pulse_dec		PW: 3.6 us, 30deg	PD: 2.0 sec	NA: 139	LB: 0.0	Nuts - JHJ1210_exp3_13C_MP_tBu_13i.jdf

*anti-**4j***



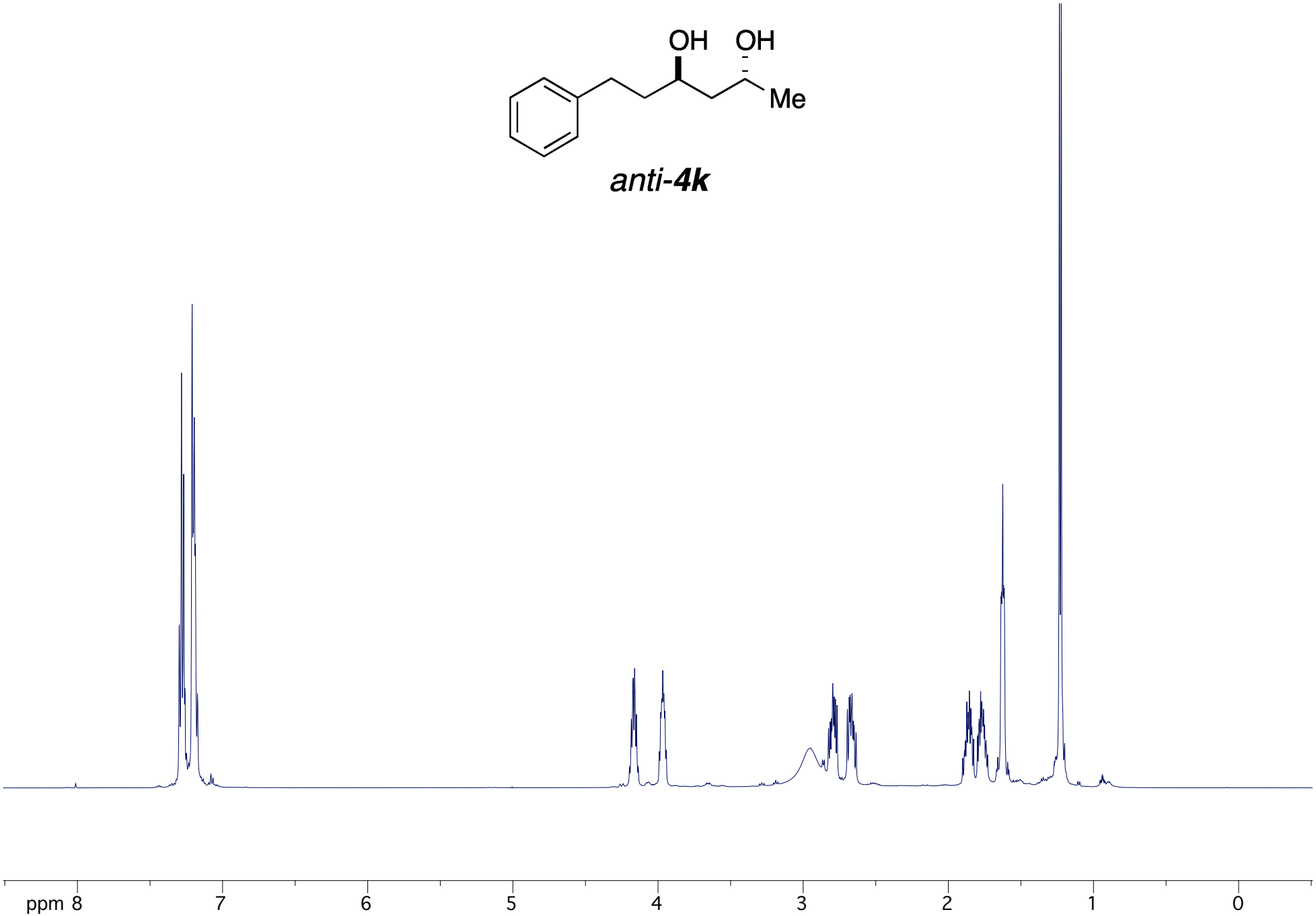
*anti-**4j***

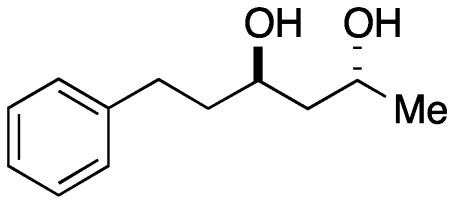


S#372282 (single pulse decoupled gated NOE) USER: delta -- DATE: Dec 11, 2012 00:53:38

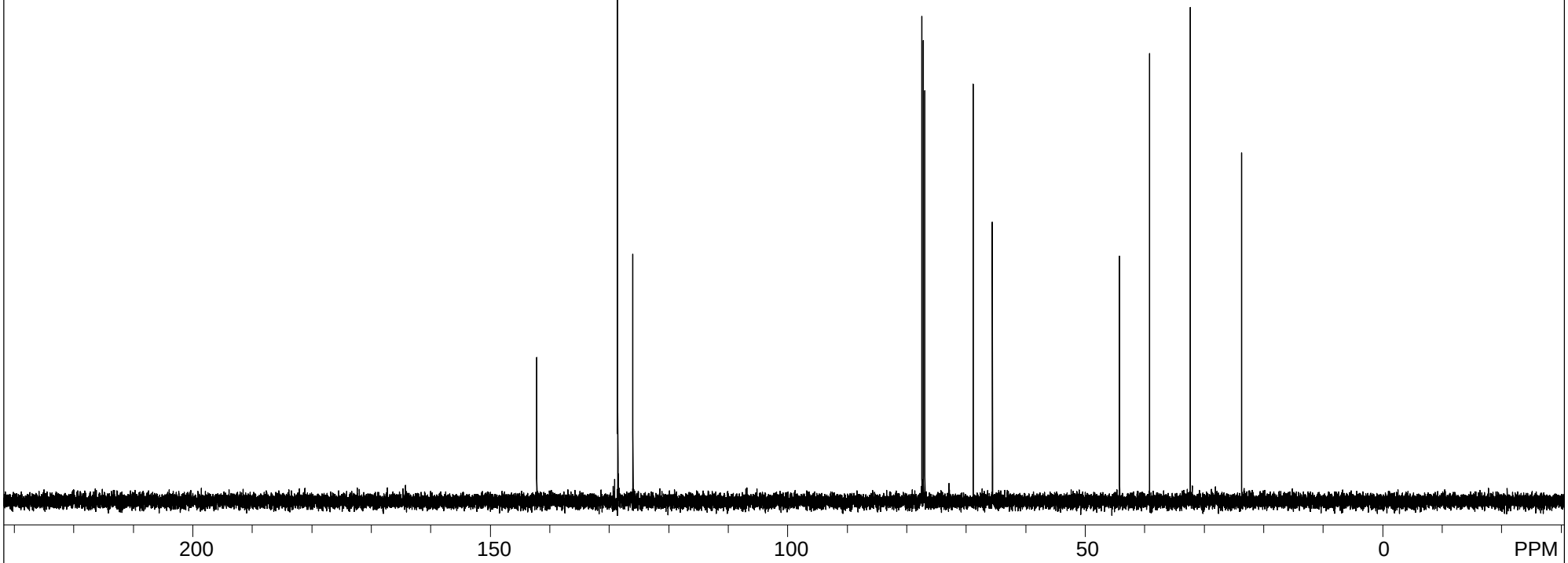
F1: 125.778	F2: 500.162	SW1: 39308		OF1: 12590.2		PTS1d: 32768 , 32768
EX: single_pulse_dec		PW: 3.6 us, 30deg	PD: 2.0 sec	NA: 56	LB: 0.0	Nuts - JHJ1210_rxn7_13C_MMP_Cy_13j.jdf

anti-4k





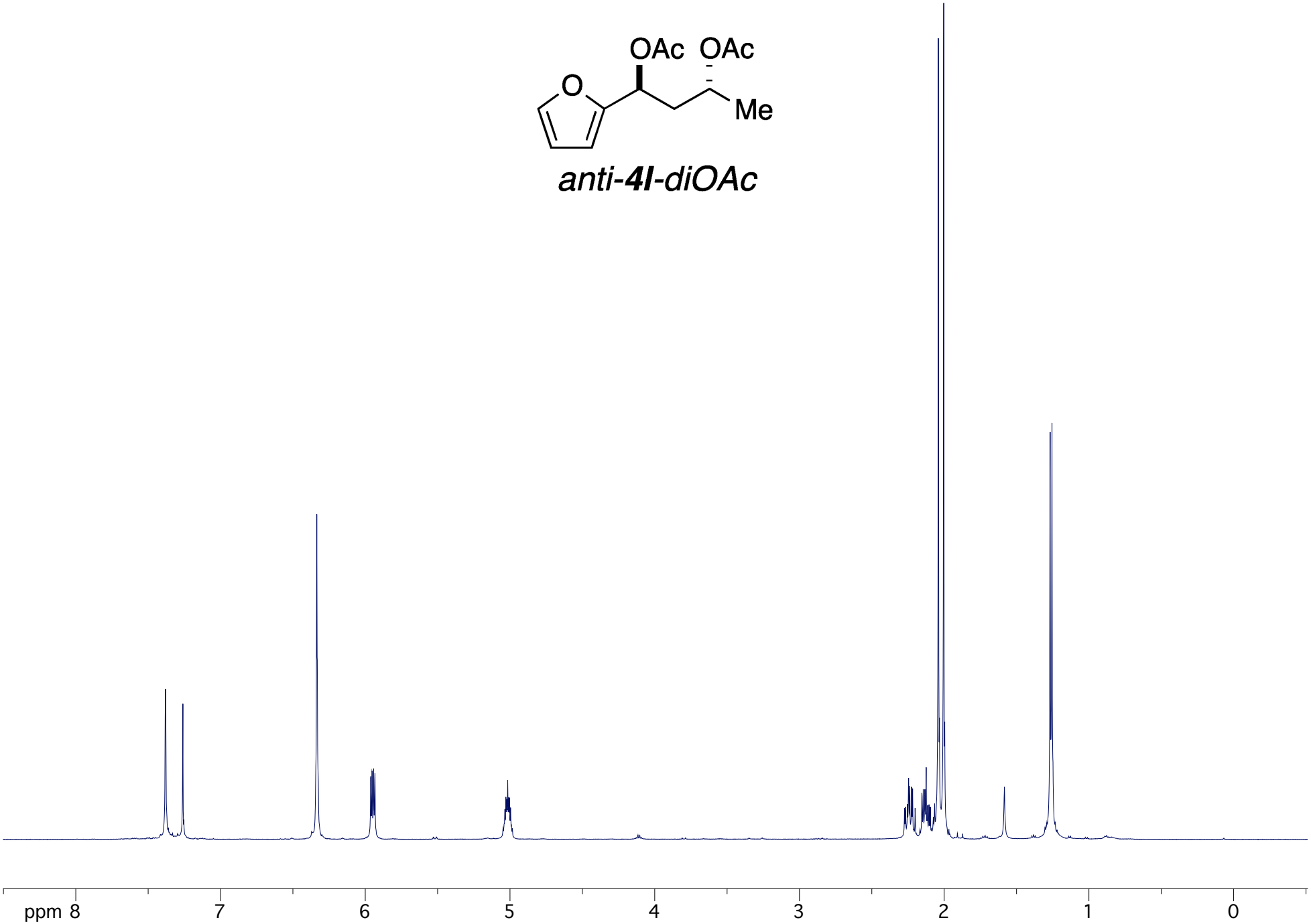
*anti-**4k***

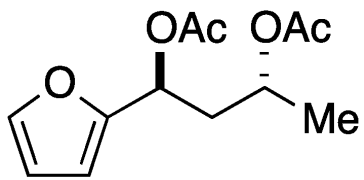


S#418717 (single pulse decoupled gated NOE) USER: delta -- DATE: Dec 6, 2012 02:12:12

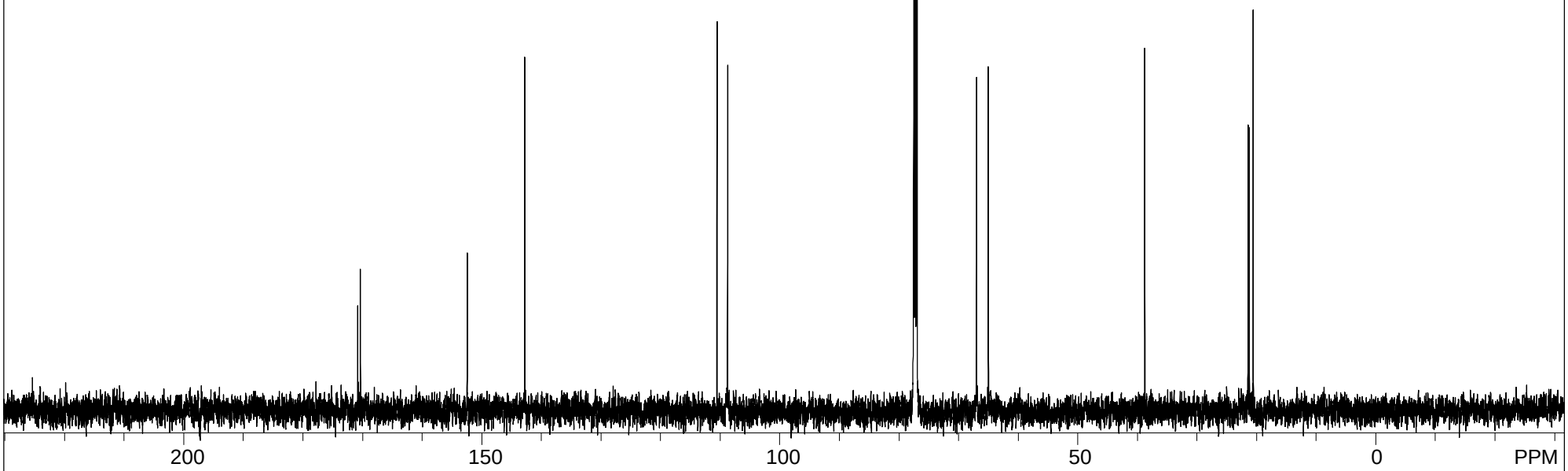
F1: 125.778	F2: 500.162	SW1: 39308		OF1: 12586.6		PTS1d: 32768 , 32768
EX: single_pulse_dec		PW: 3.6 us, 30deg	PD: 2.0 sec	NA: 82	LB: 0.0	Nuts - JHJ1210_exp4_13C_MP_homoBn_13k.jdf

*anti-**4l**-diOAc*





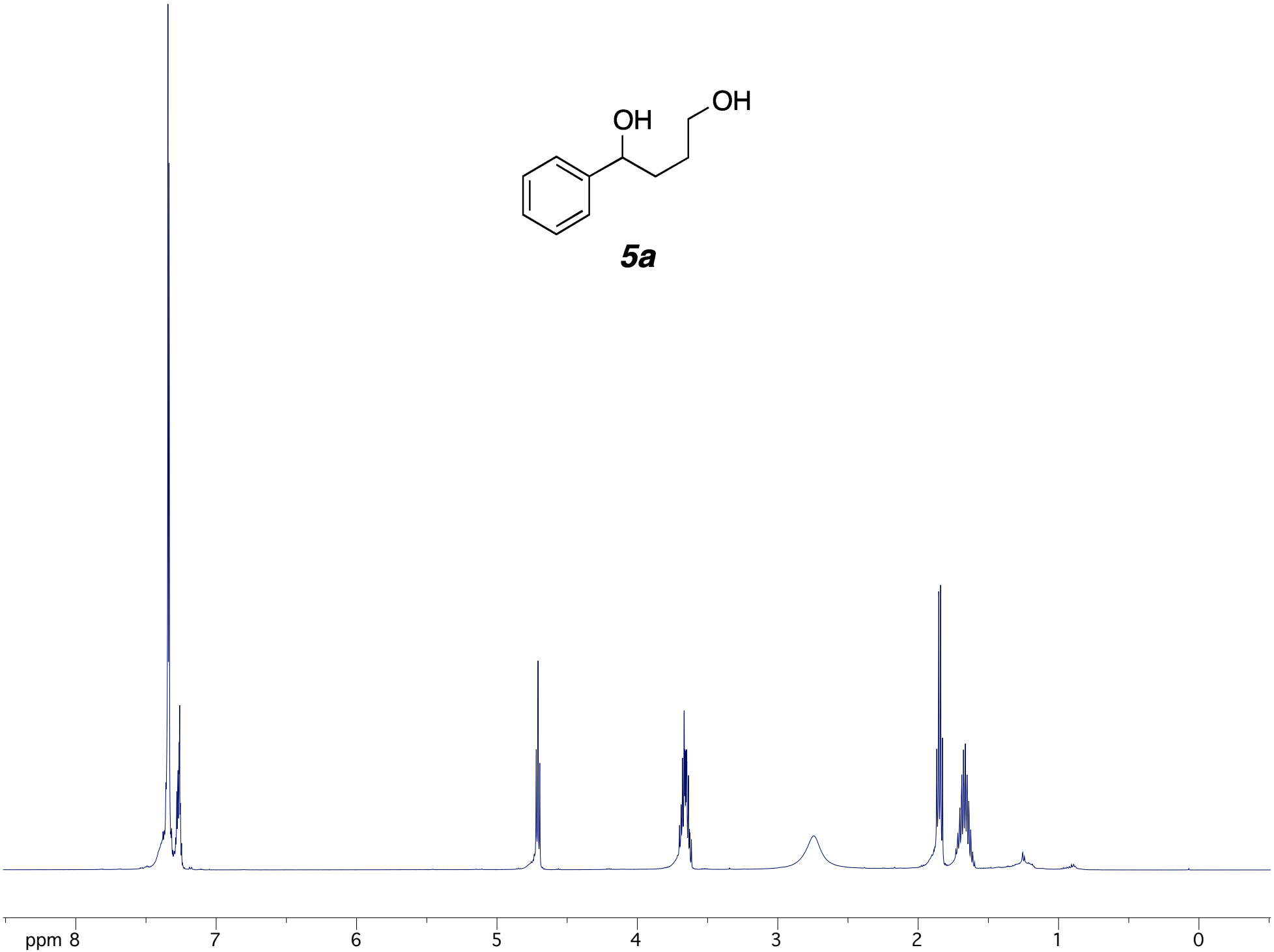
*anti-**4l**-diOAc*

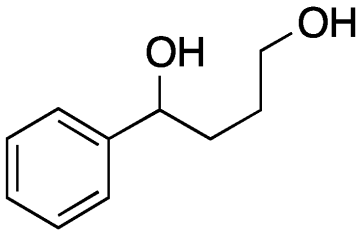


S#646523 (single pulse decoupled gated NOE) USER: delta -- DATE: Mar 25, 2013 07:40:41

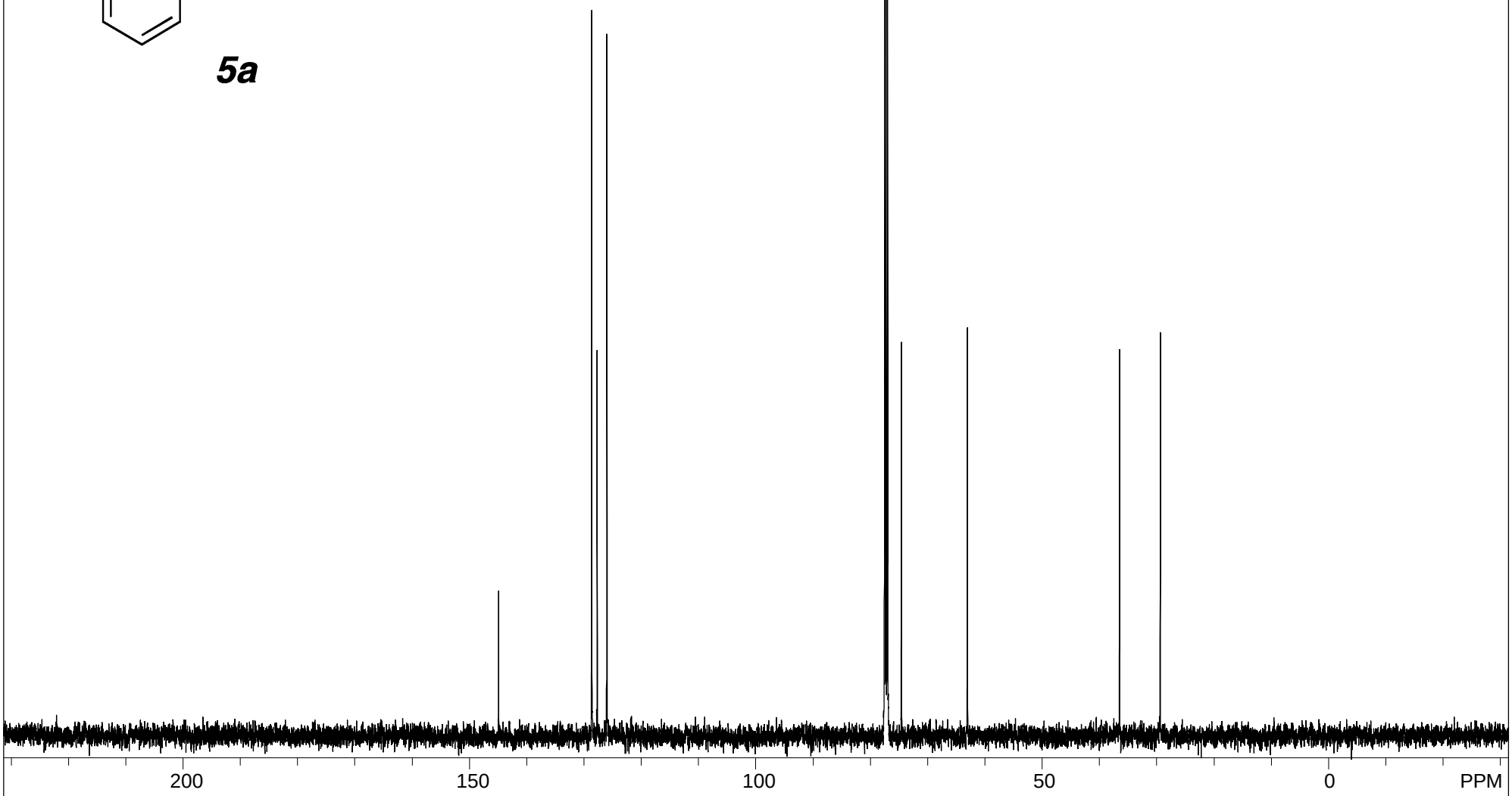
F1: 125.778	F2: 500.162	SW1: 39308		OF1: 12592.6		PTS1d: 32768	
EX: single_pulse_dec		PW: 3.6 us, 30deg	PD: 2.0 sec	NA: 348	LB: 0.0	Nuts - $YH2093C_furan_13DiAc_M1_13C-2.jdf	

5a



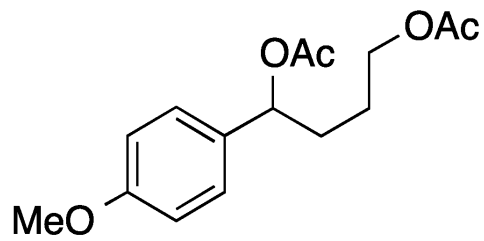


5a

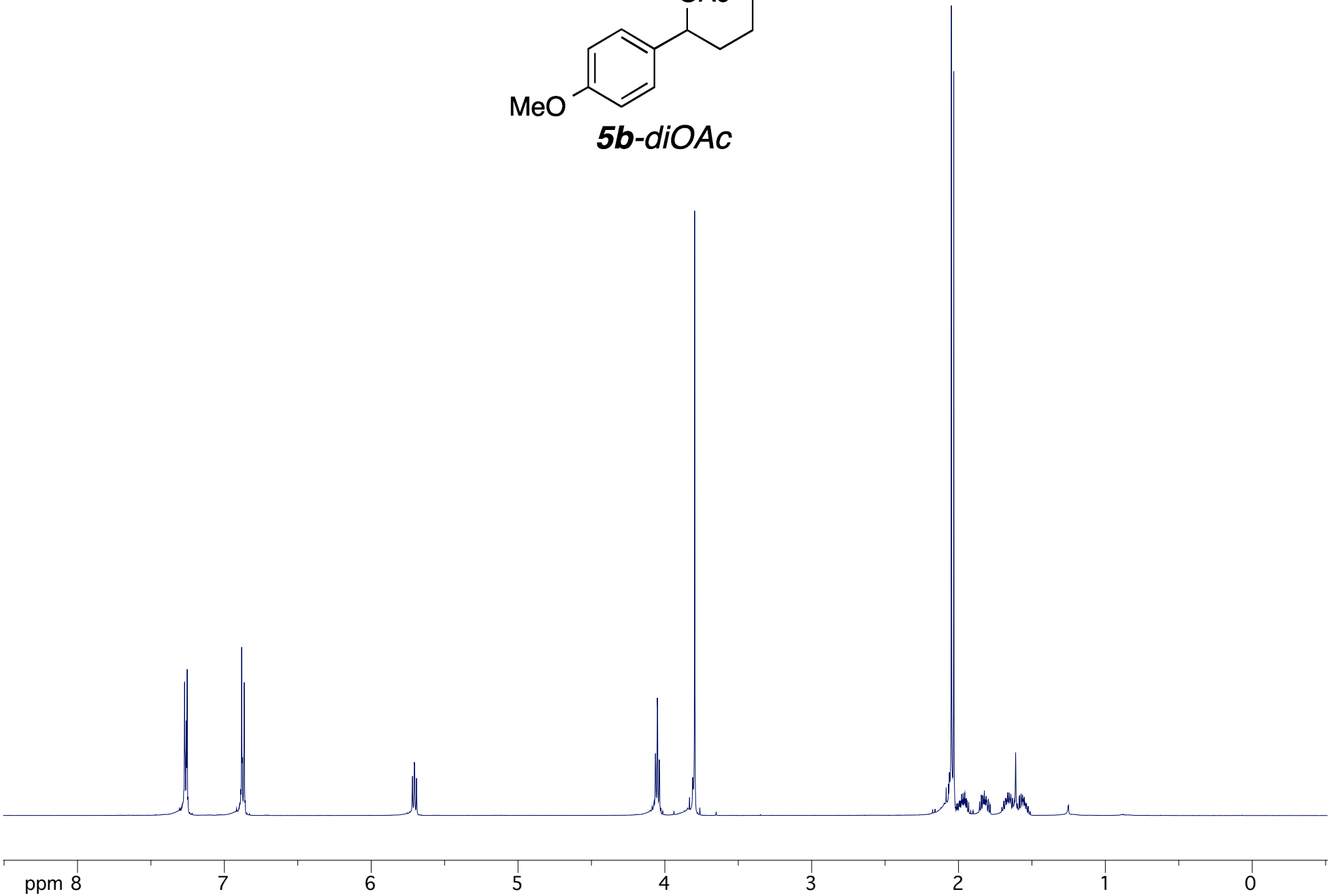


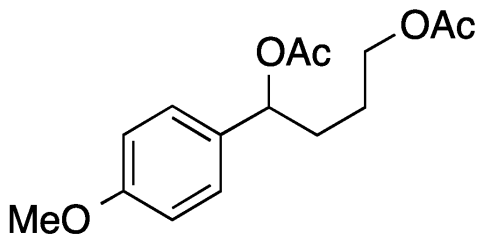
S#354588 (single pulse decoupled gated NOE) USER: delta -- DATE: Jan 15, 2013 00:21:47

F1: 125.778	F2: 500.162	SW1: 39308		OF1: 12590.2		PTS1d: 32768	
EX: single_pulse_dec		PW: 3.6 us, 30deg	PD: 2.0 sec	NA: 145	LB: 0.0	Nuts - YH_Rush_Ph_14Diol_13C-2 2_14a.jdf	

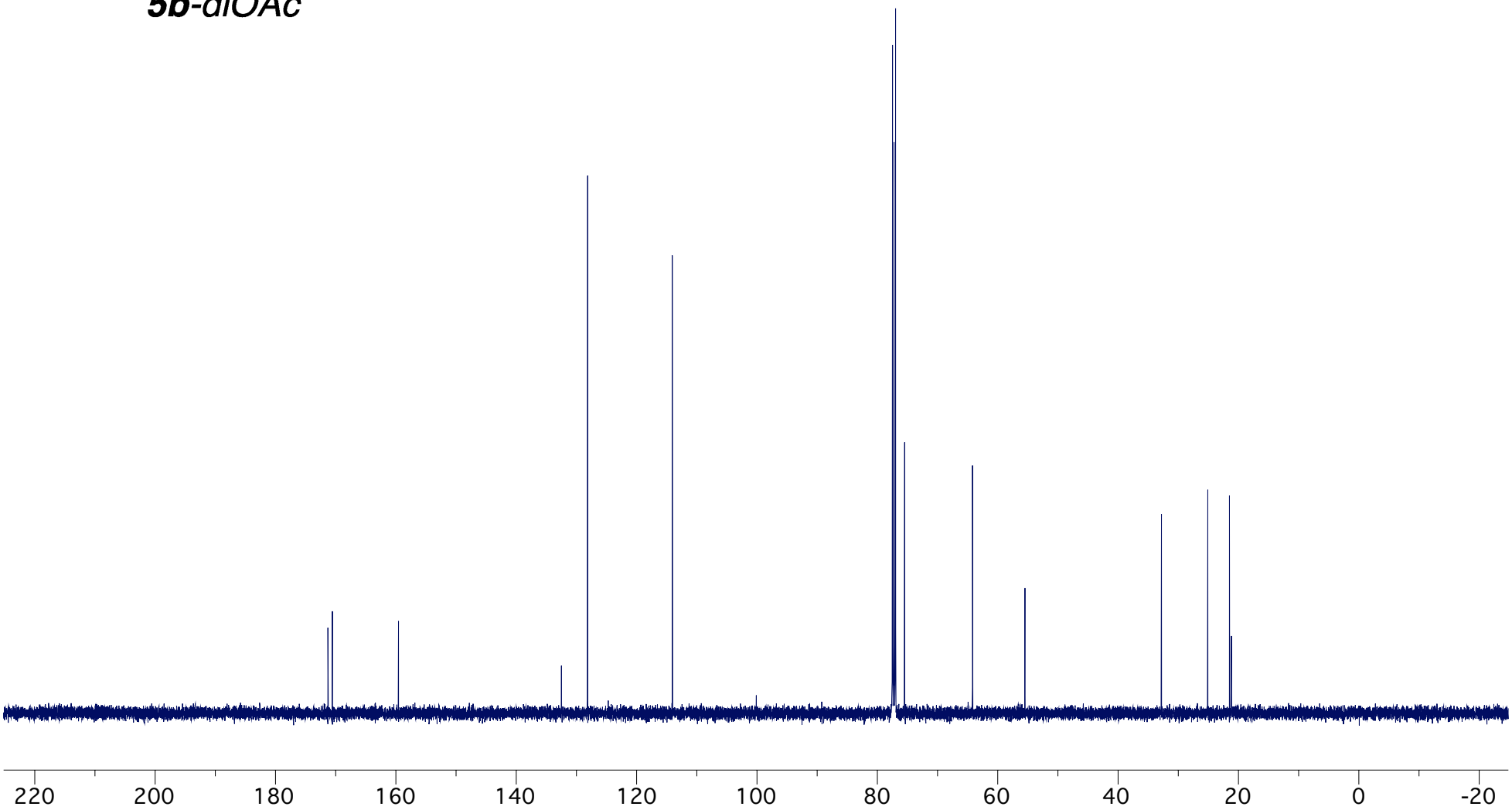


5b*-*diOAc

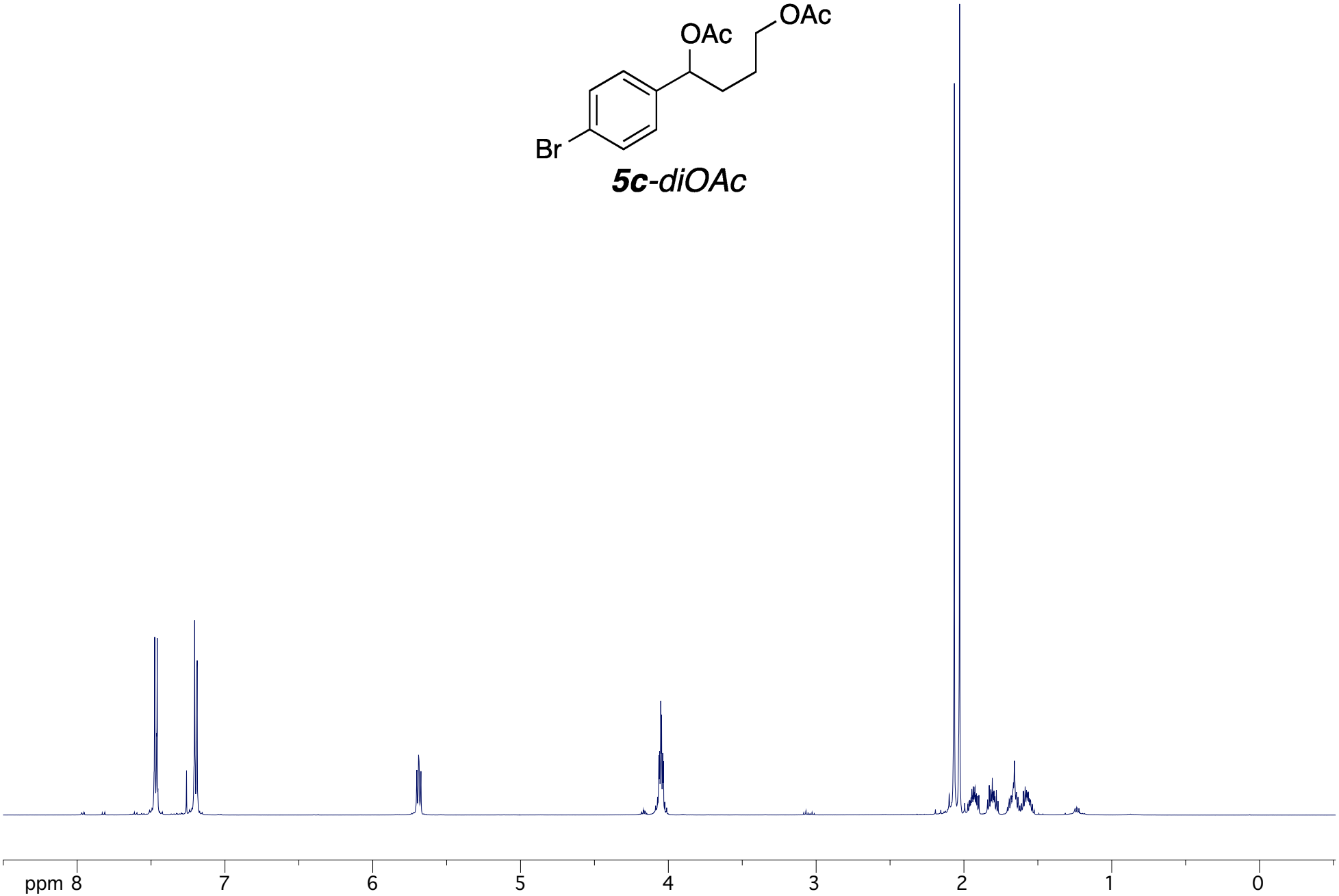


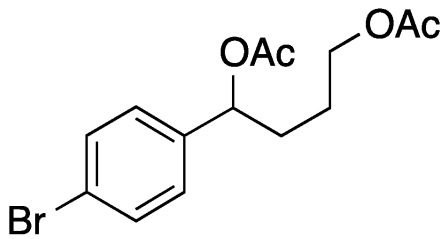


***5b**-diOAc*

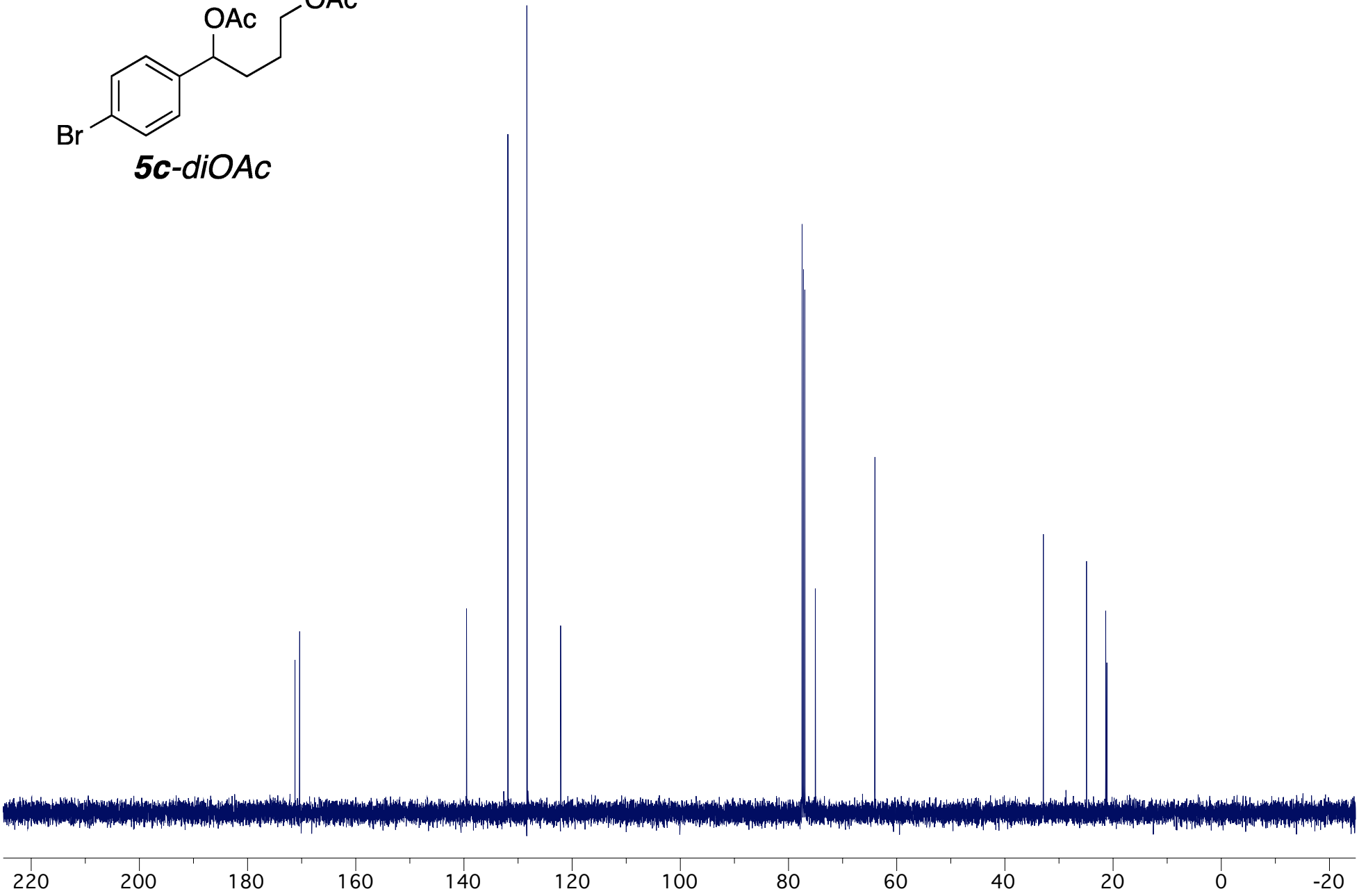


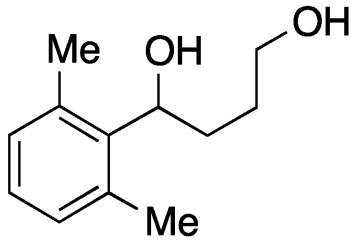
***5c**-diOAc*



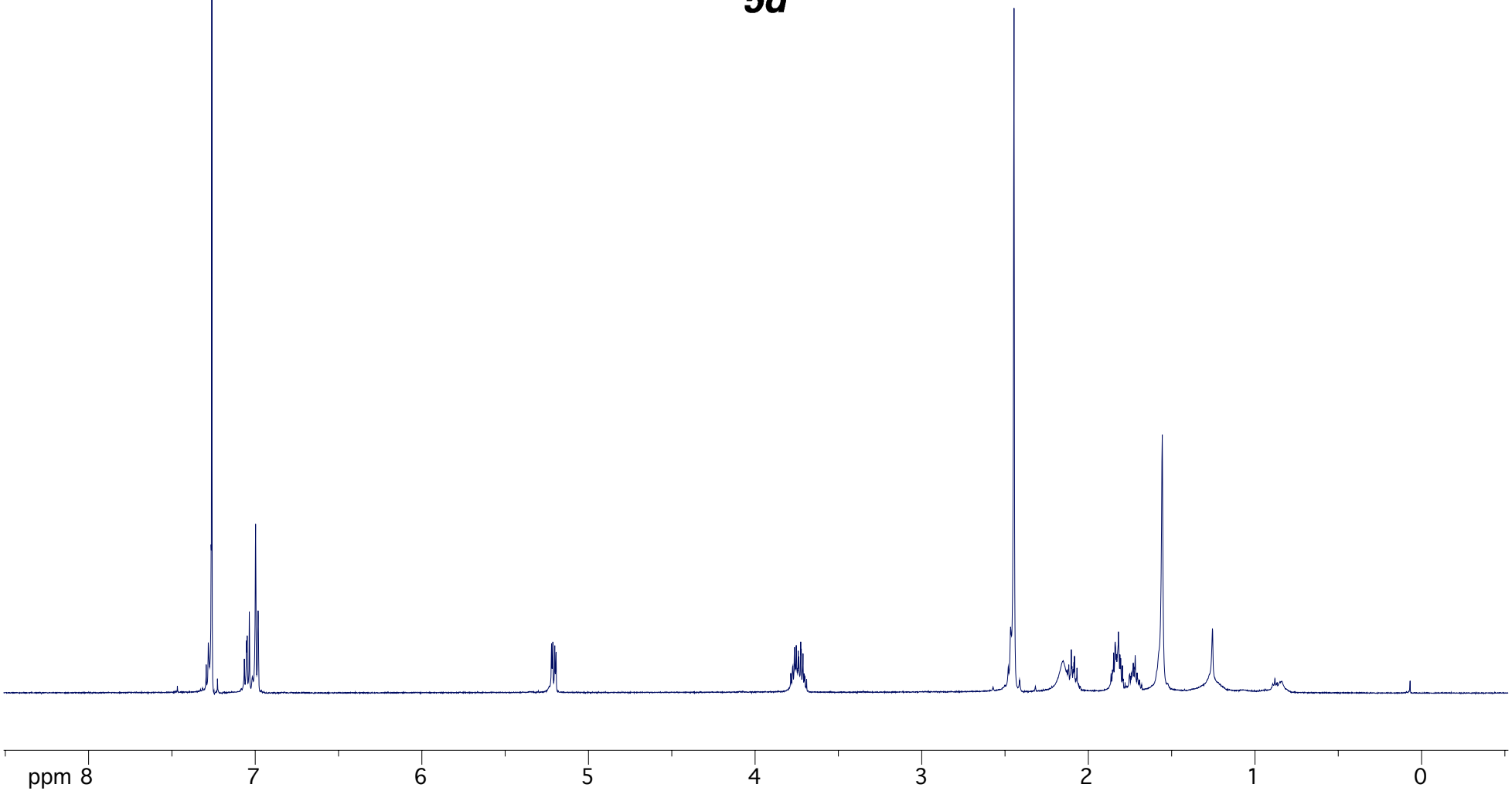


***5c**-diOAc*

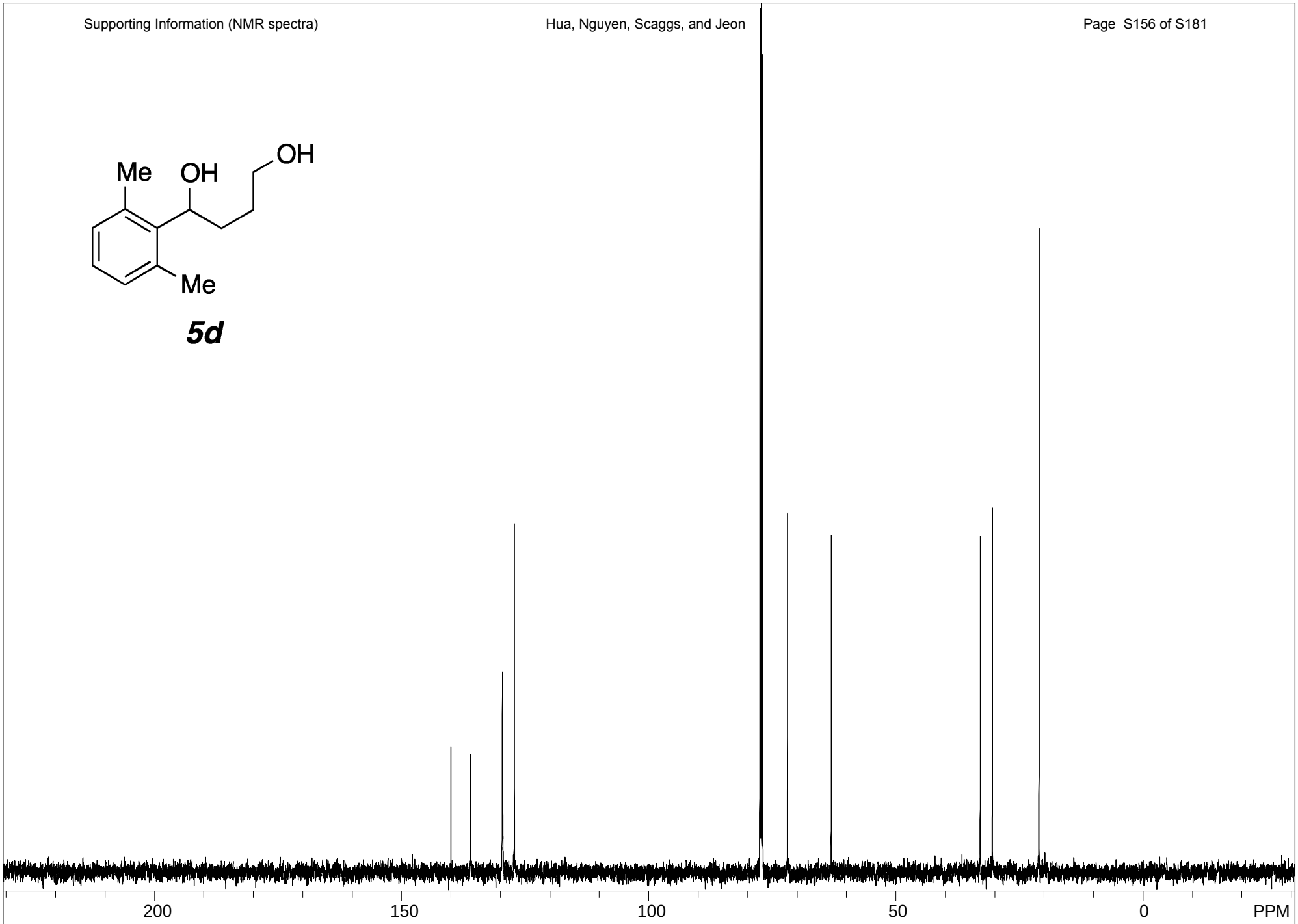




5d



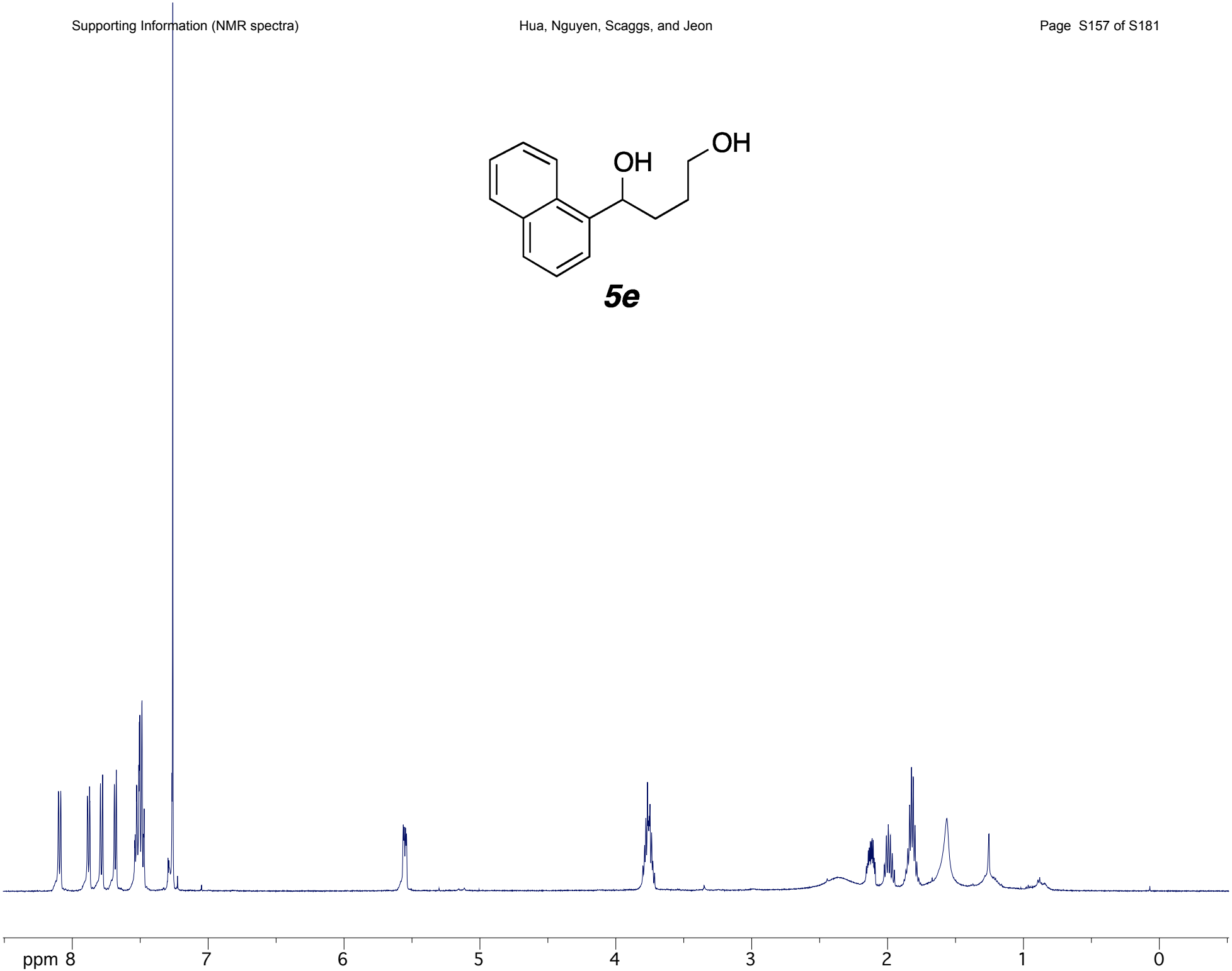
5d

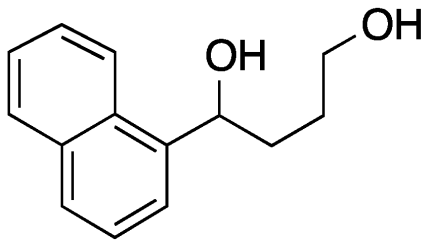


S#727150 (single pulse decoupled gated NOE) USER: delta -- DATE: Jan 15, 2013 10:41:24

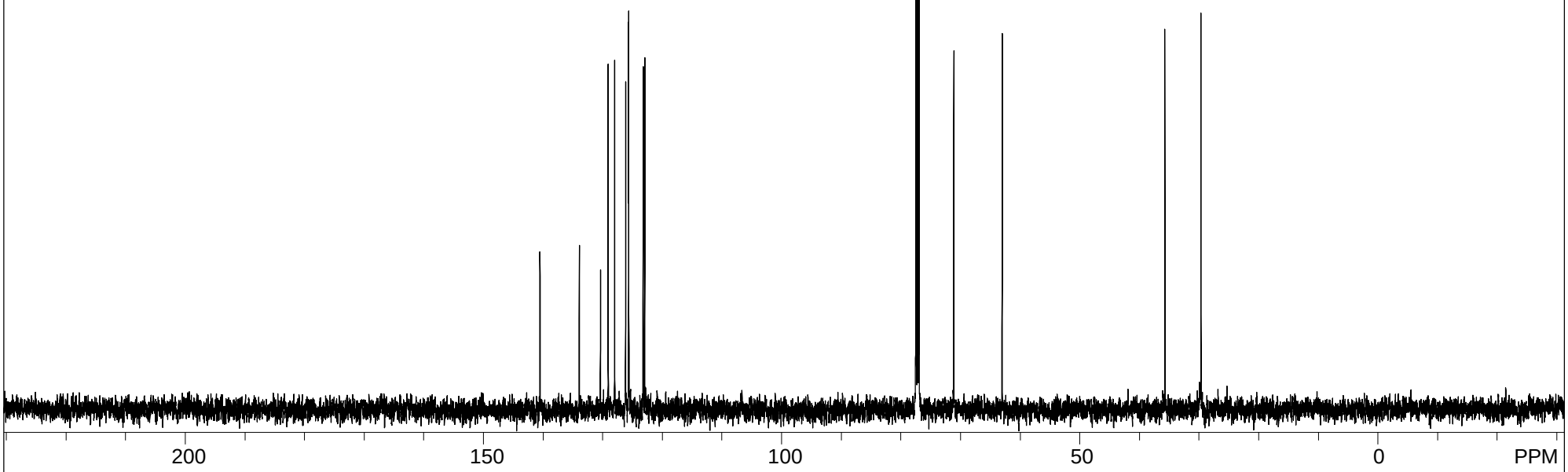
F1: 125.778	F2: 500.162	SW1: 39308		OF1: 12589.0		PTS1d: 32768	
EX: single_pulse_dec		PW: 3.6 us, 30deg	PD: 2.0 sec	NA: 117	LB: 0.0 Nuts -	$YH_Rush_14Diol_DiMe_MPLC_r1_2_13C-2.jdf	

5e





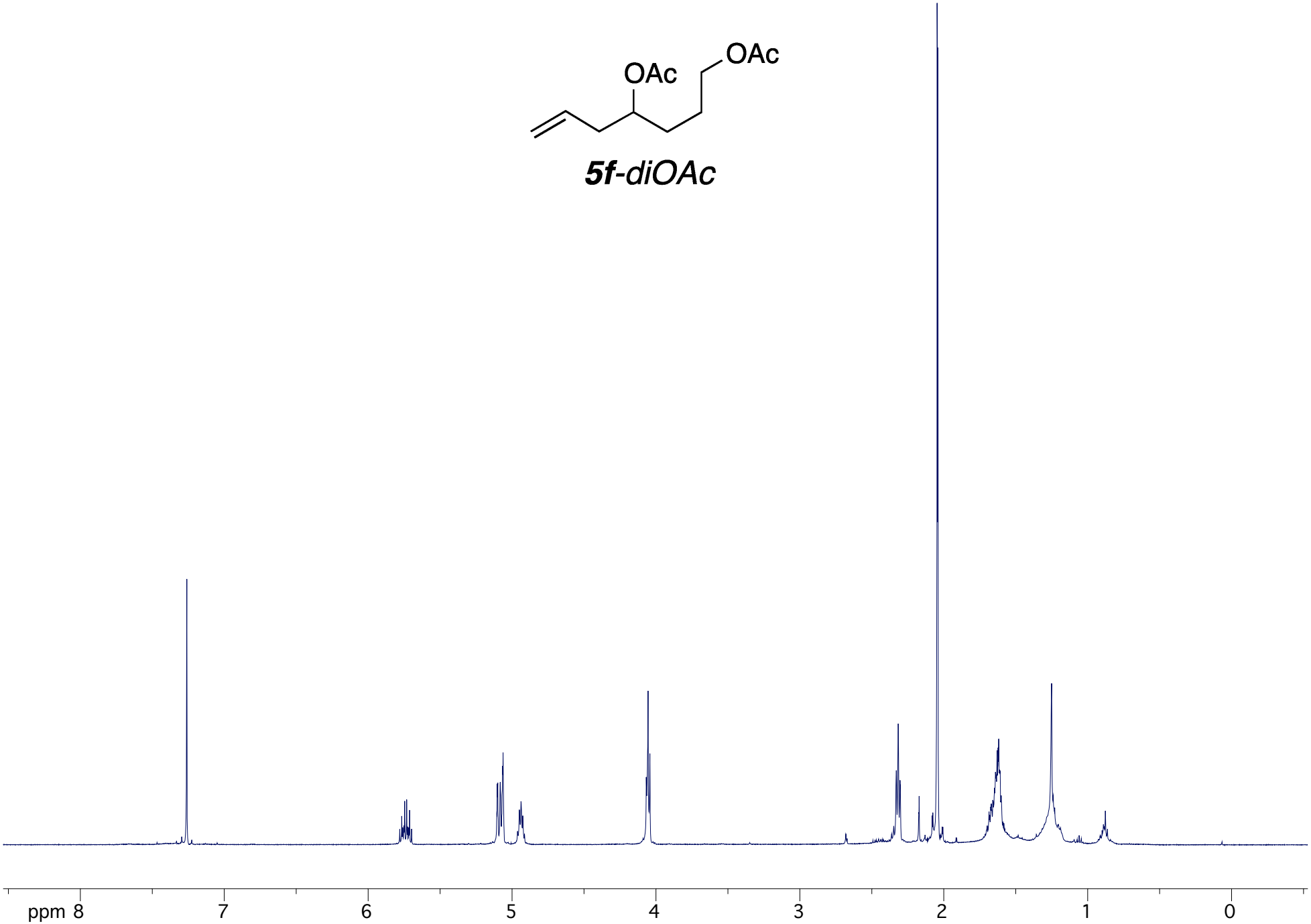
5e



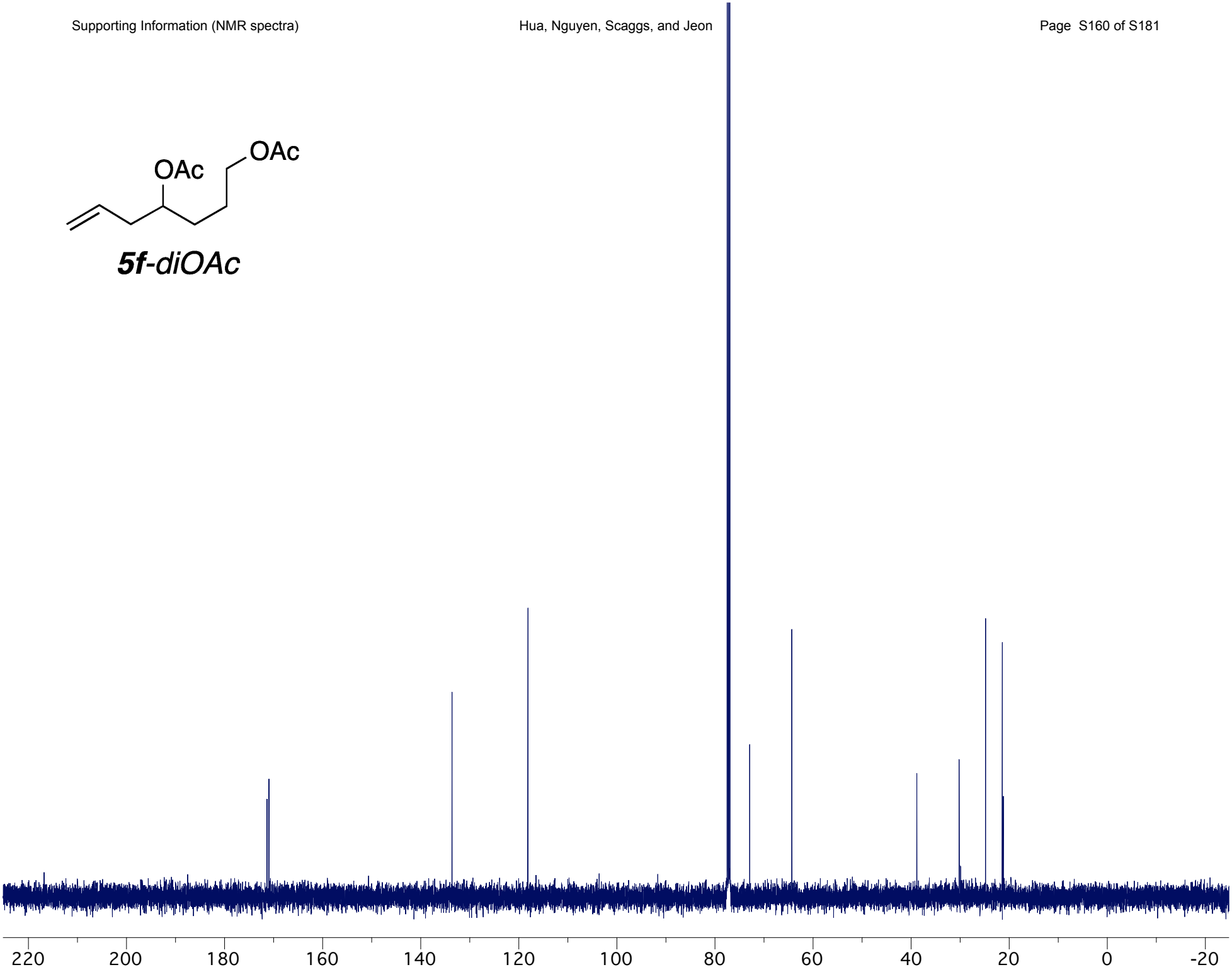
S#613964 (single pulse decoupled gated NOE) USER: delta -- DATE: Jan 16, 2013 07:31:49

F1: 125.778	F2: 500.162	SW1: 39308		OF1: 12587.8		PTS1d: 32768	
EX: single_pulse_dec		PW: 3.6 us, 30deg	PD: 2.0 sec	NA: 97	LB: 0.0	Nuts - YH_Rush_14Diol_Napht_13C-2 3_14e.jdf	

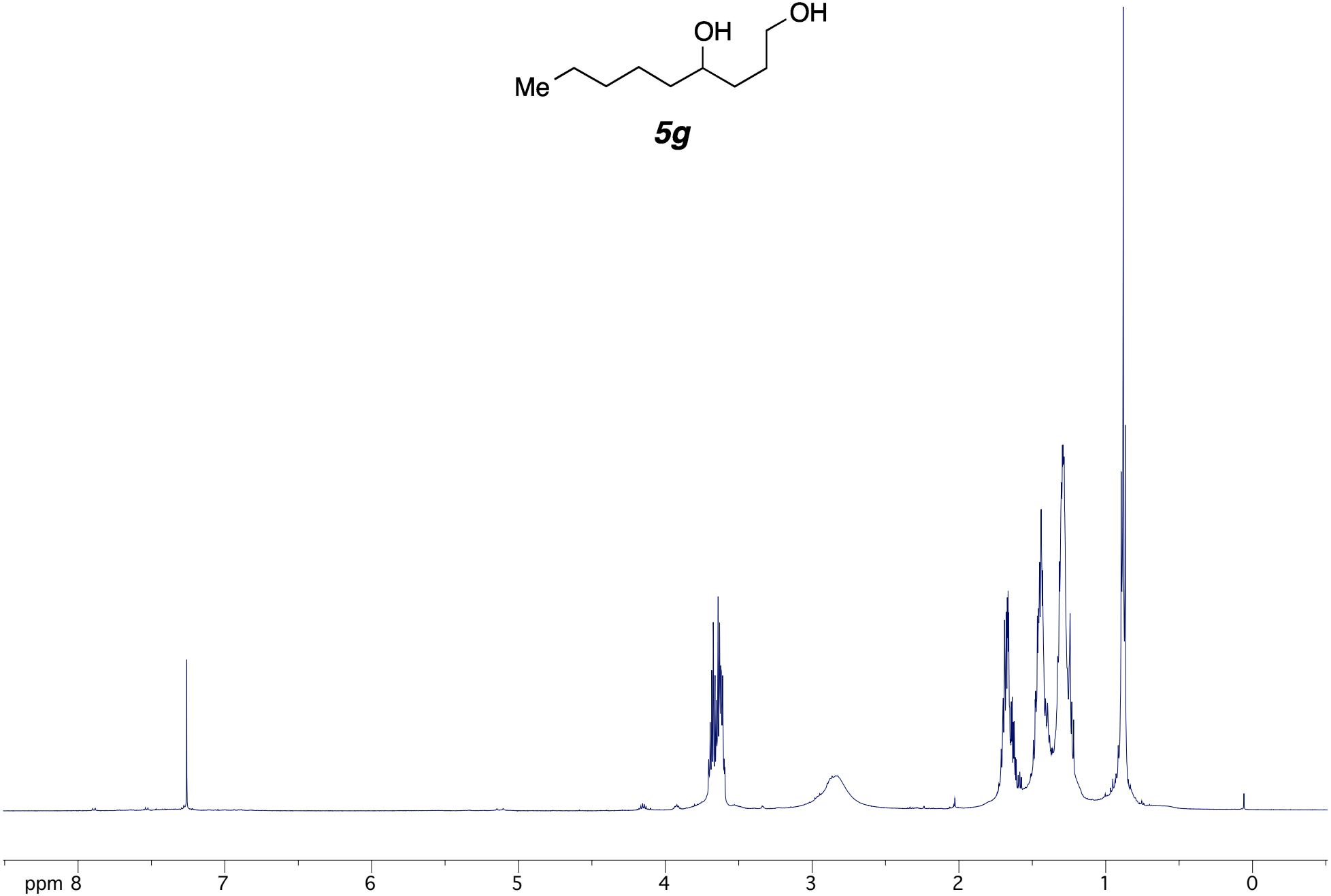
***5f*-diOAc**

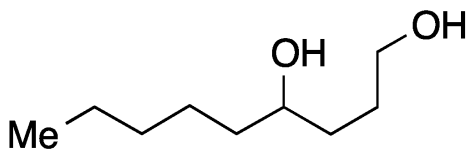


***5f*-diOAc**

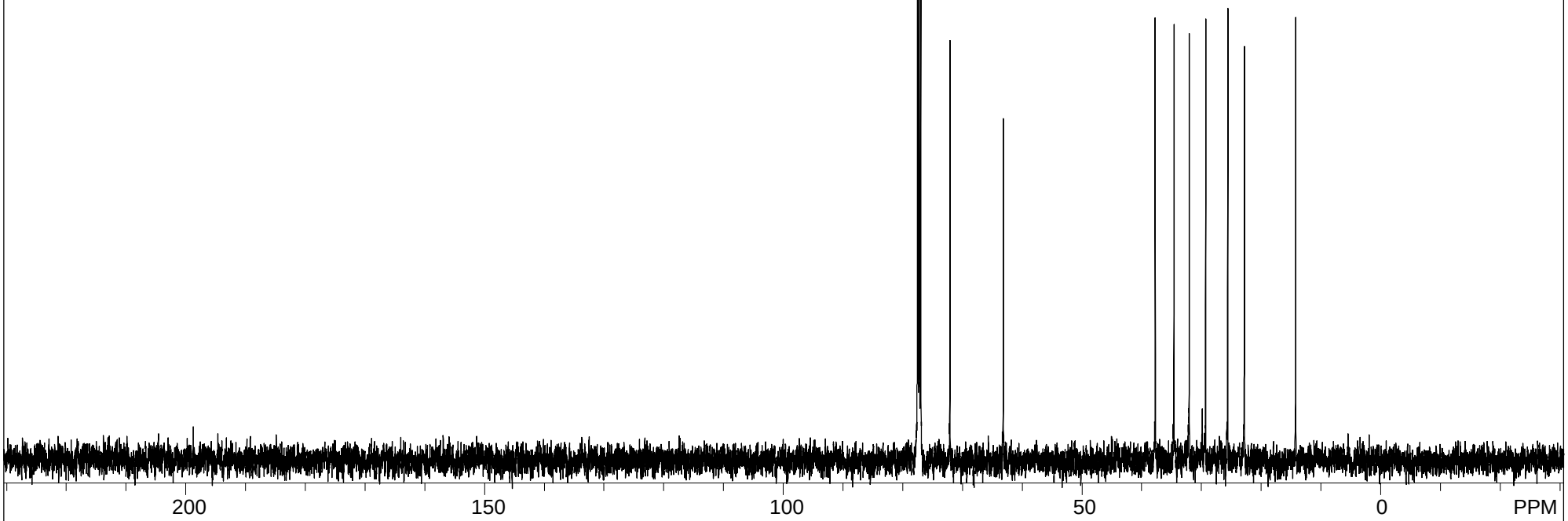


5g





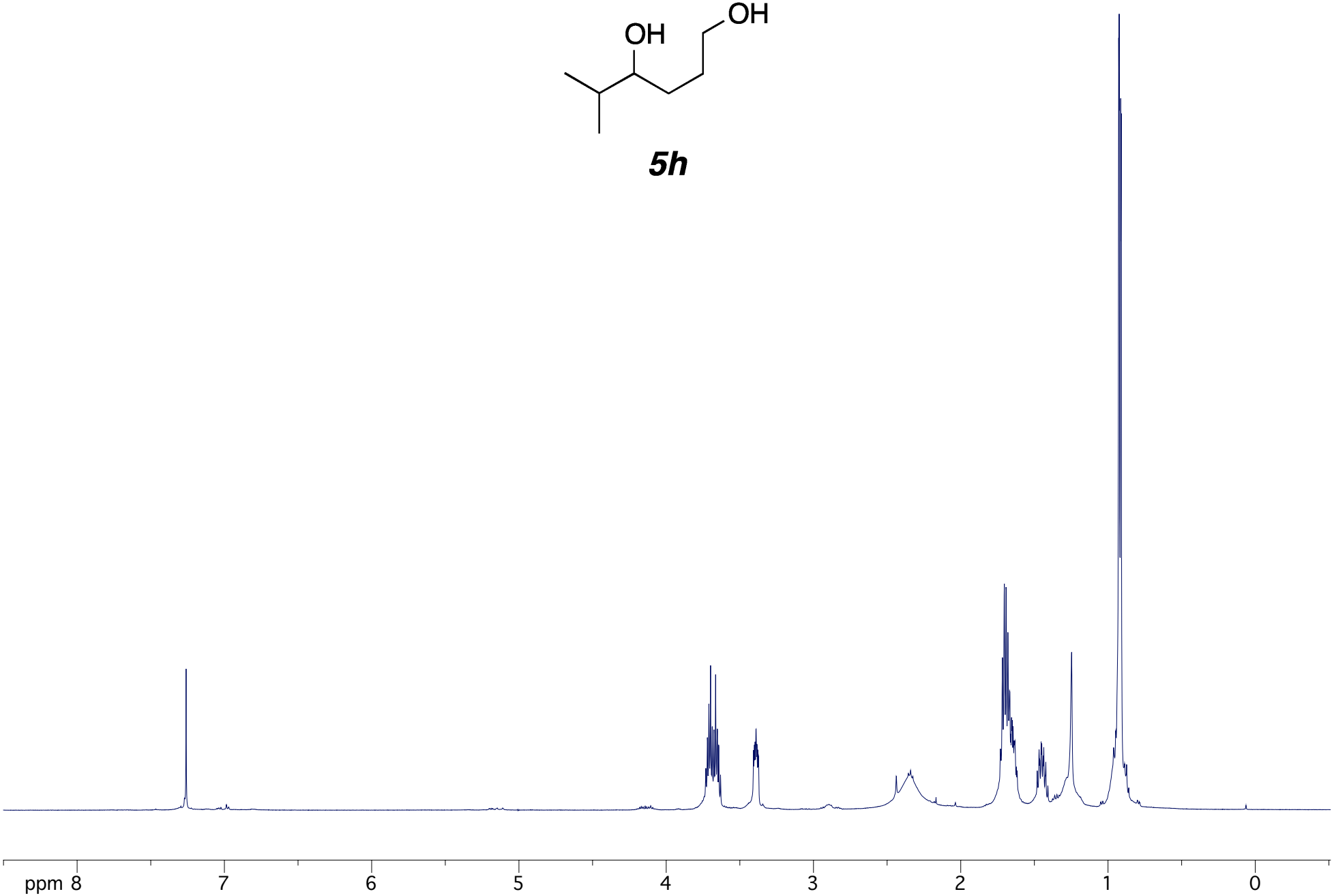
5g

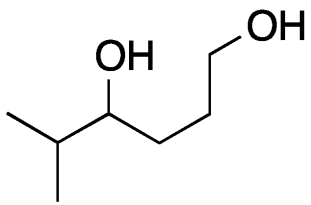


S#615993 (single pulse decoupled gated NOE) USER: delta -- DATE: Jan 17, 2013 07:33:35

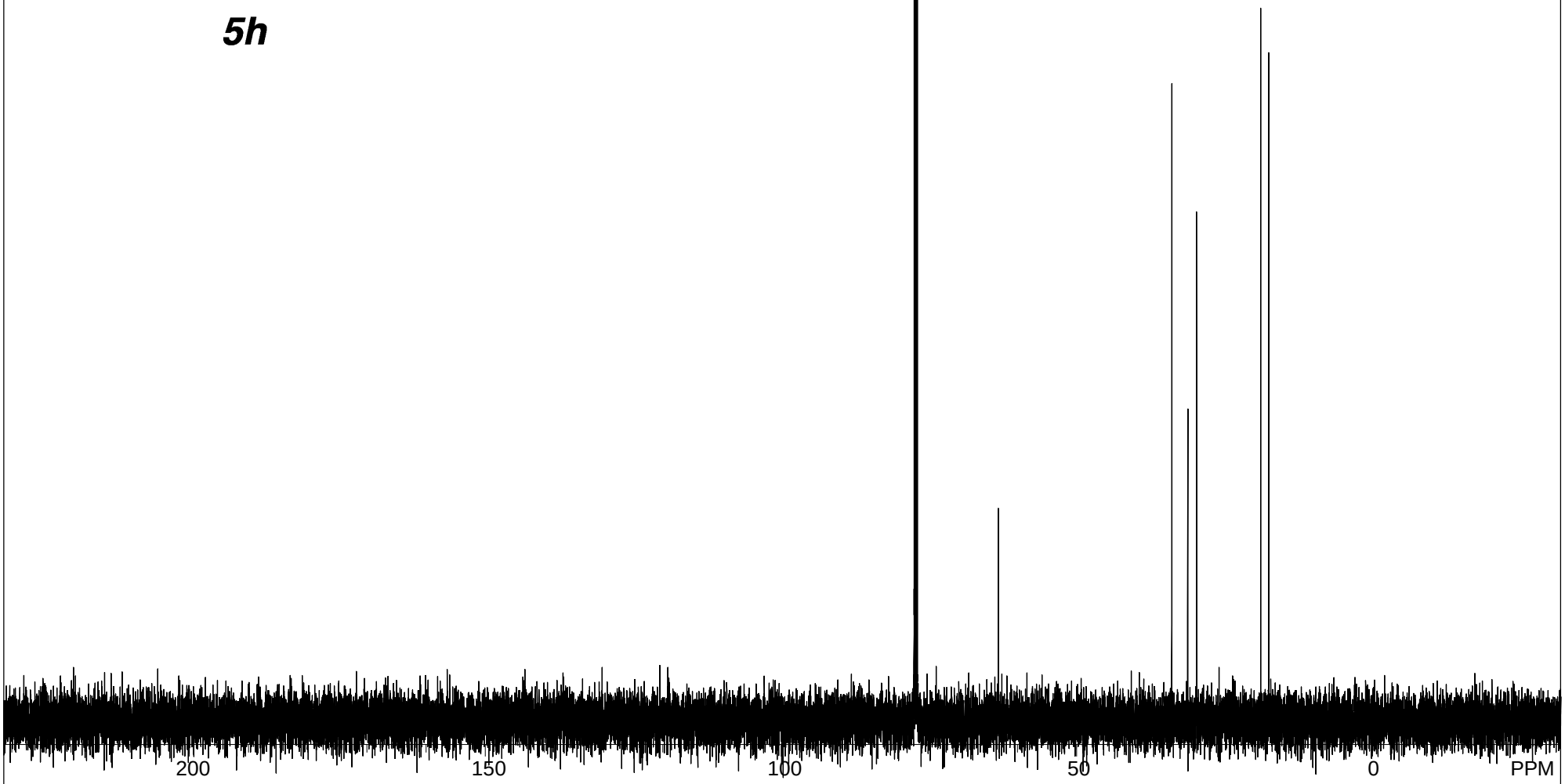
F1: 125.778	F2: 500.162	SW1: 39308		OF1: 12591.4		PTS1d: 32768	
EX: single_pulse_dec		PW: 3.6 us, 30deg	PD: 2.0 sec	NA: 62	LB: 0.0	Nuts - JHJ1220_13C_M1_pent-3 2_14g.jdf	

5h





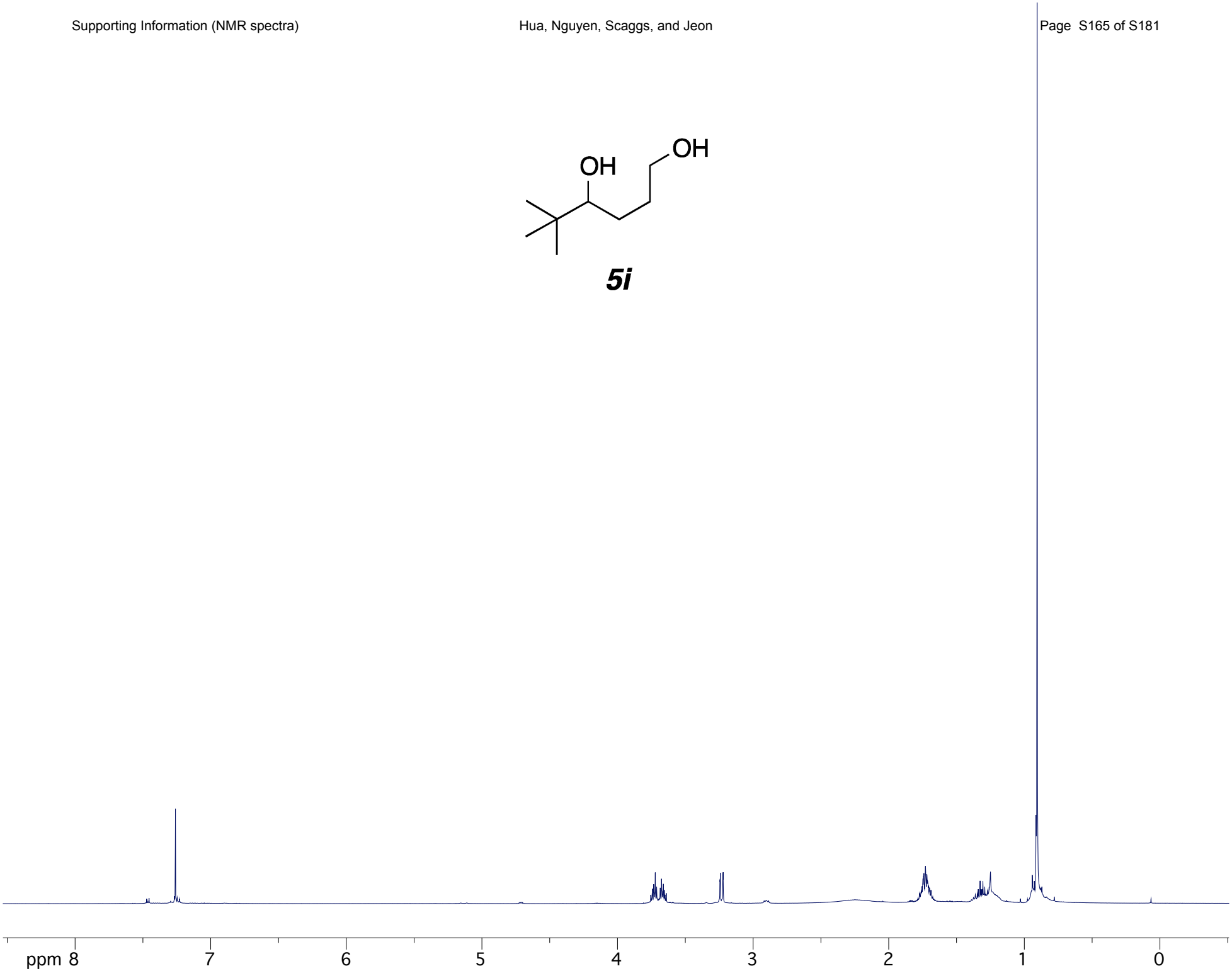
5h



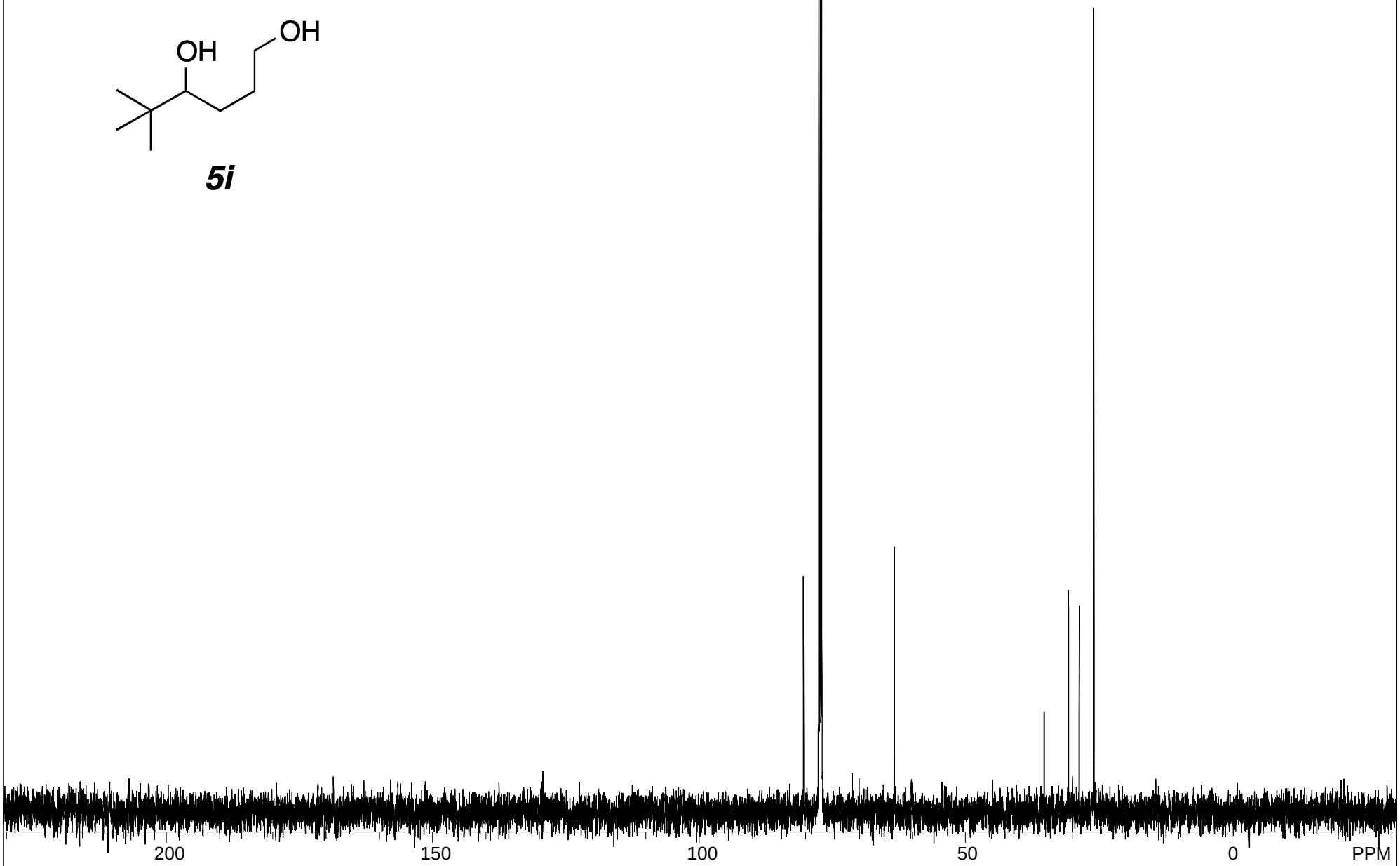
S#593200 (single pulse decoupled gated NOE) USER: delta -- DATE: Jan 16, 2013 06:58:23

F1: 125.778	F2: 500.162	SW1: 39308		OF1: 12592.6		PTS1d: 32768 , 32768
EX: single_pulse_dec		PW: 3.6 us, 30deg	PD: 2.0 sec	NA: 121	LB: 0.0	Nuts - YH_Rush_14Diol_iPr_13C-1_14h.jdf

5i



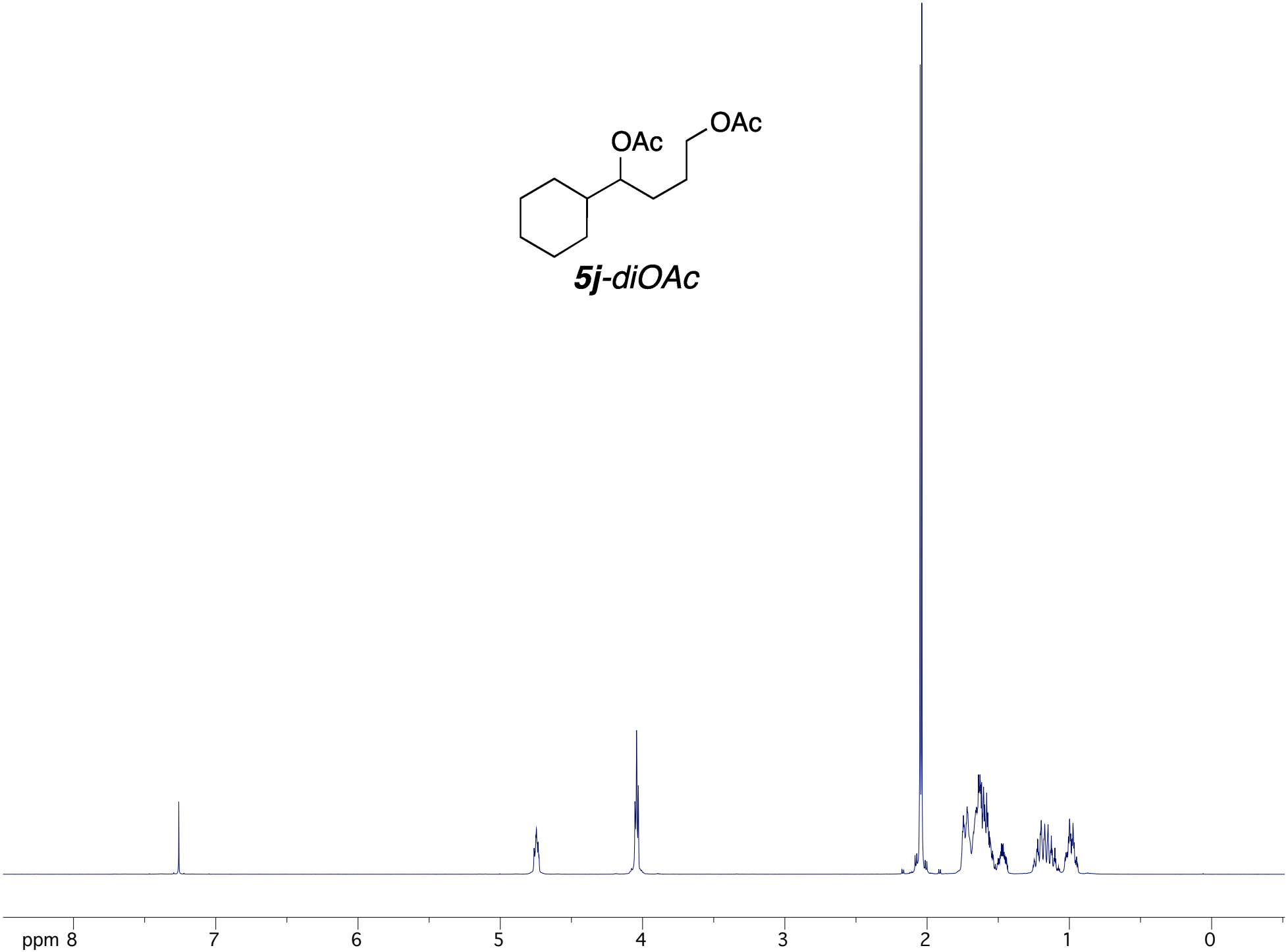
5i



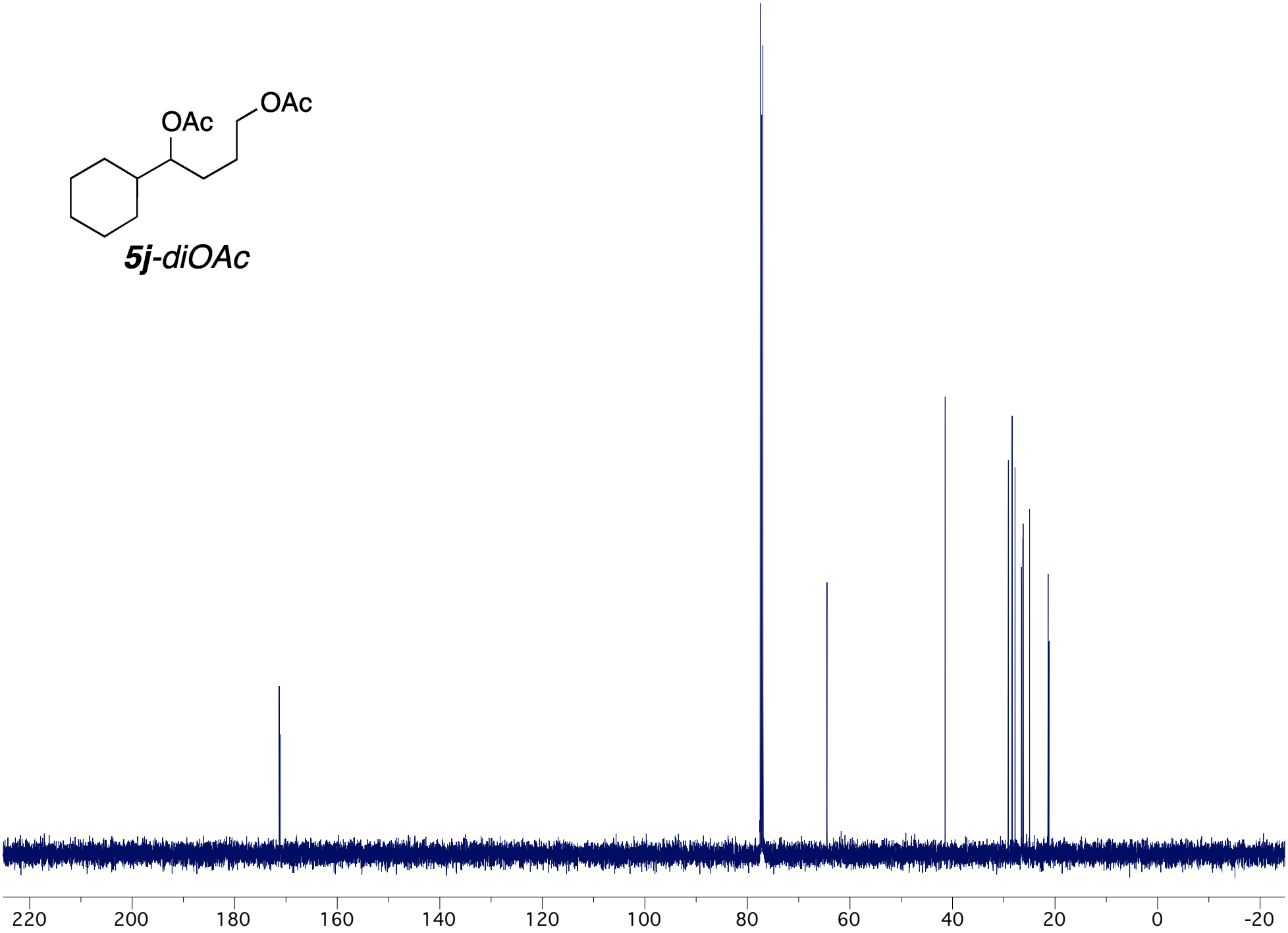
S#606581 (single pulse decoupled gated NOE) USER: delta -- DATE: Jan 17, 2013 07:24:29

F1: 125.778	F2: 500.162	SW1: 39308		OF1: 12592.6		PTS1d: 32768	
EX: single_pulse_dec		PW: 3.6 us, 30deg	PD: 2.0 sec	NA: 201	LB: 0.0	Nuts - JHJ1220_13C_M1_tBu-3 2_14i.jdf	

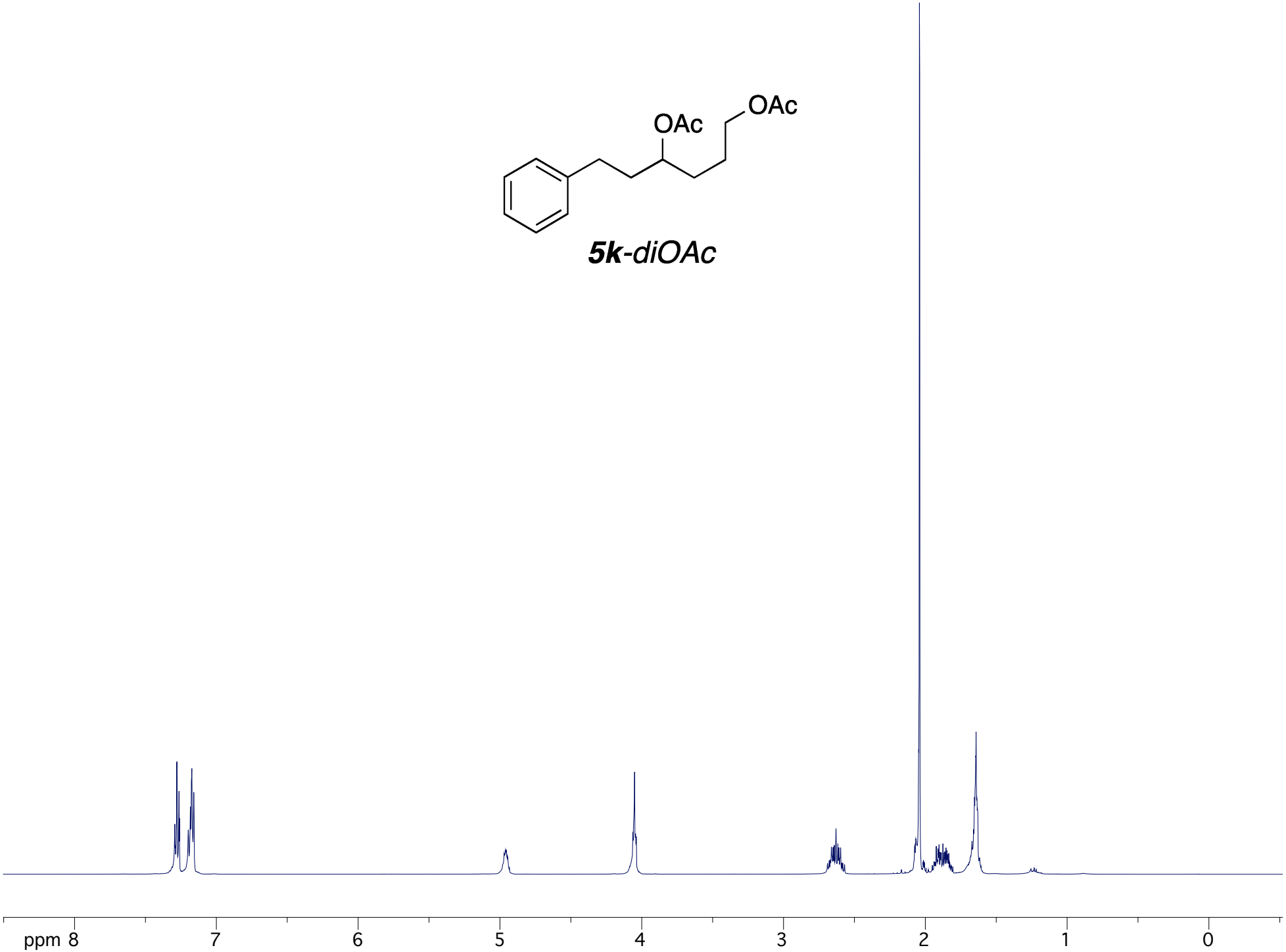
***5j*-diOAc**

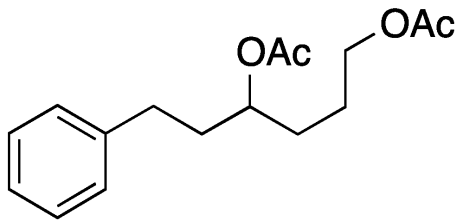


***5j**-diOAc*

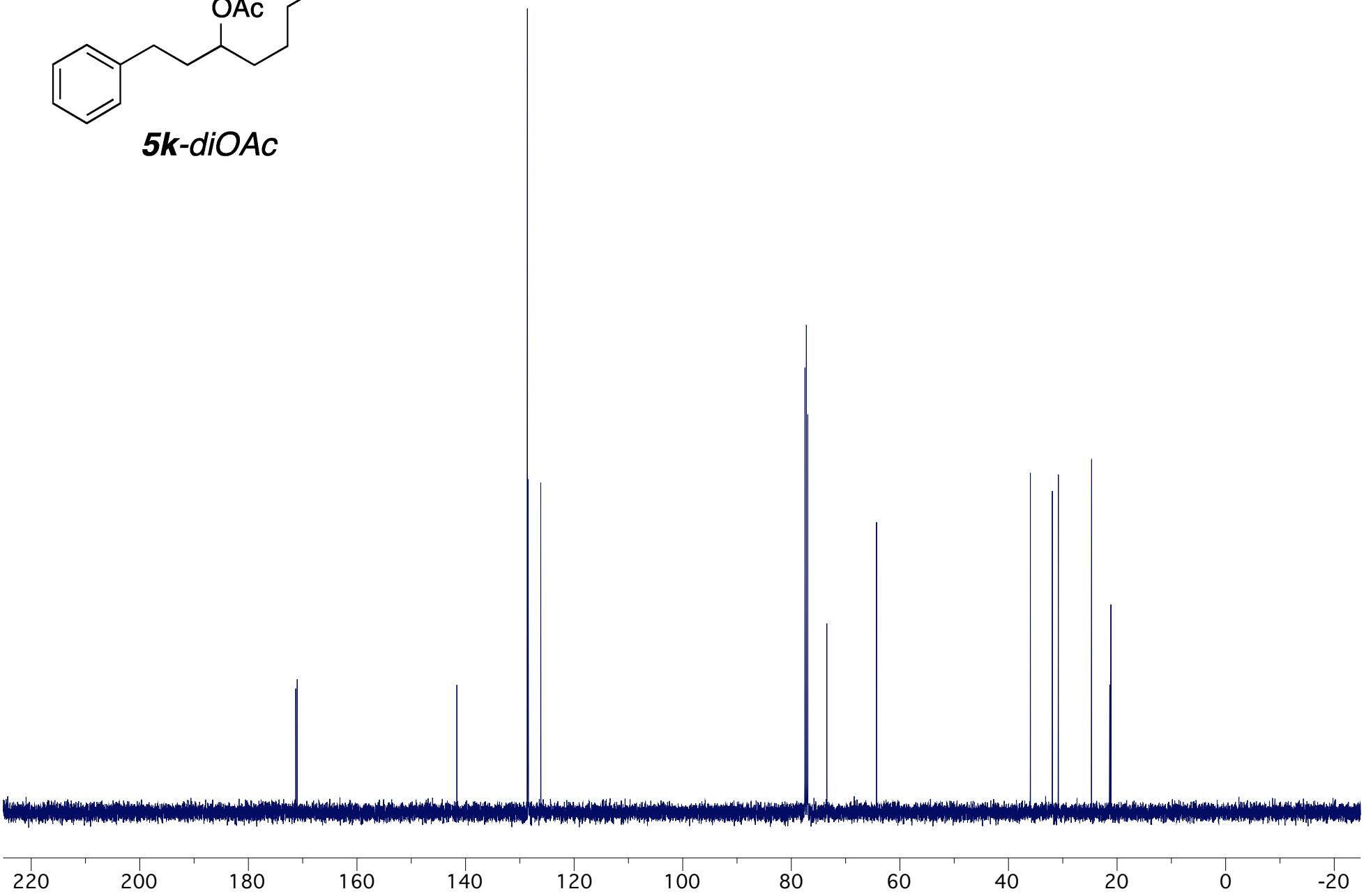


***5k**-diOAc*

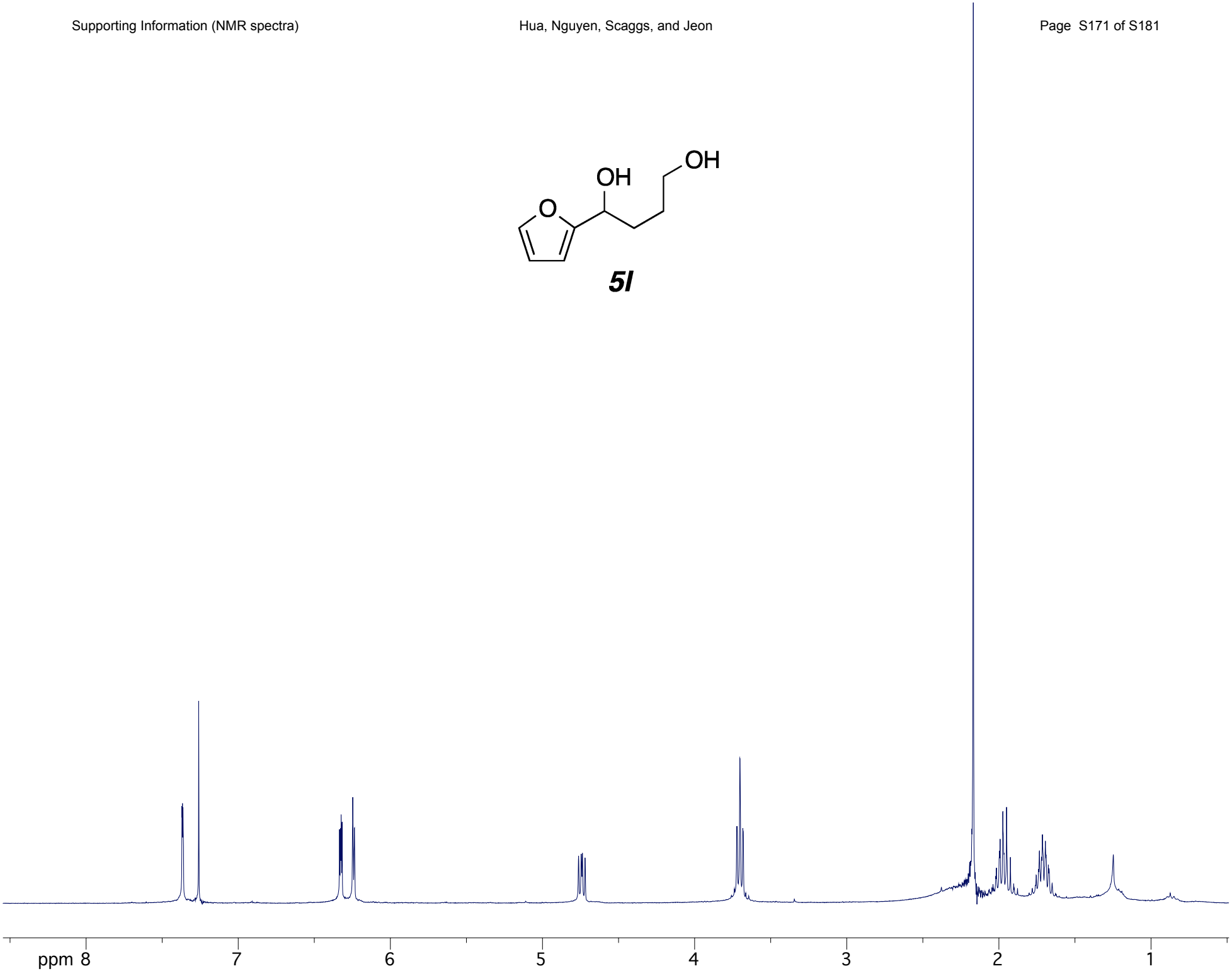




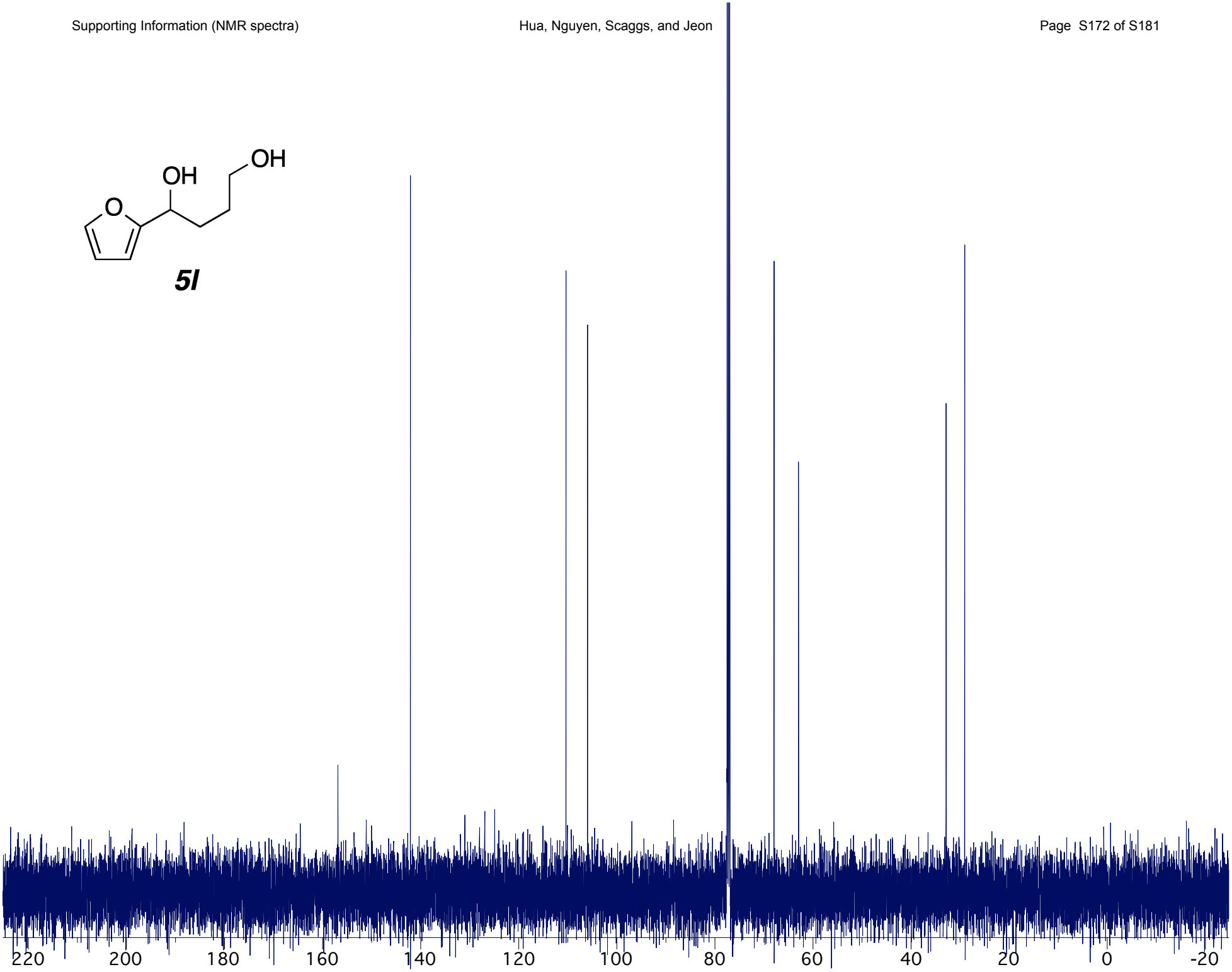
***5k**-diOAc*

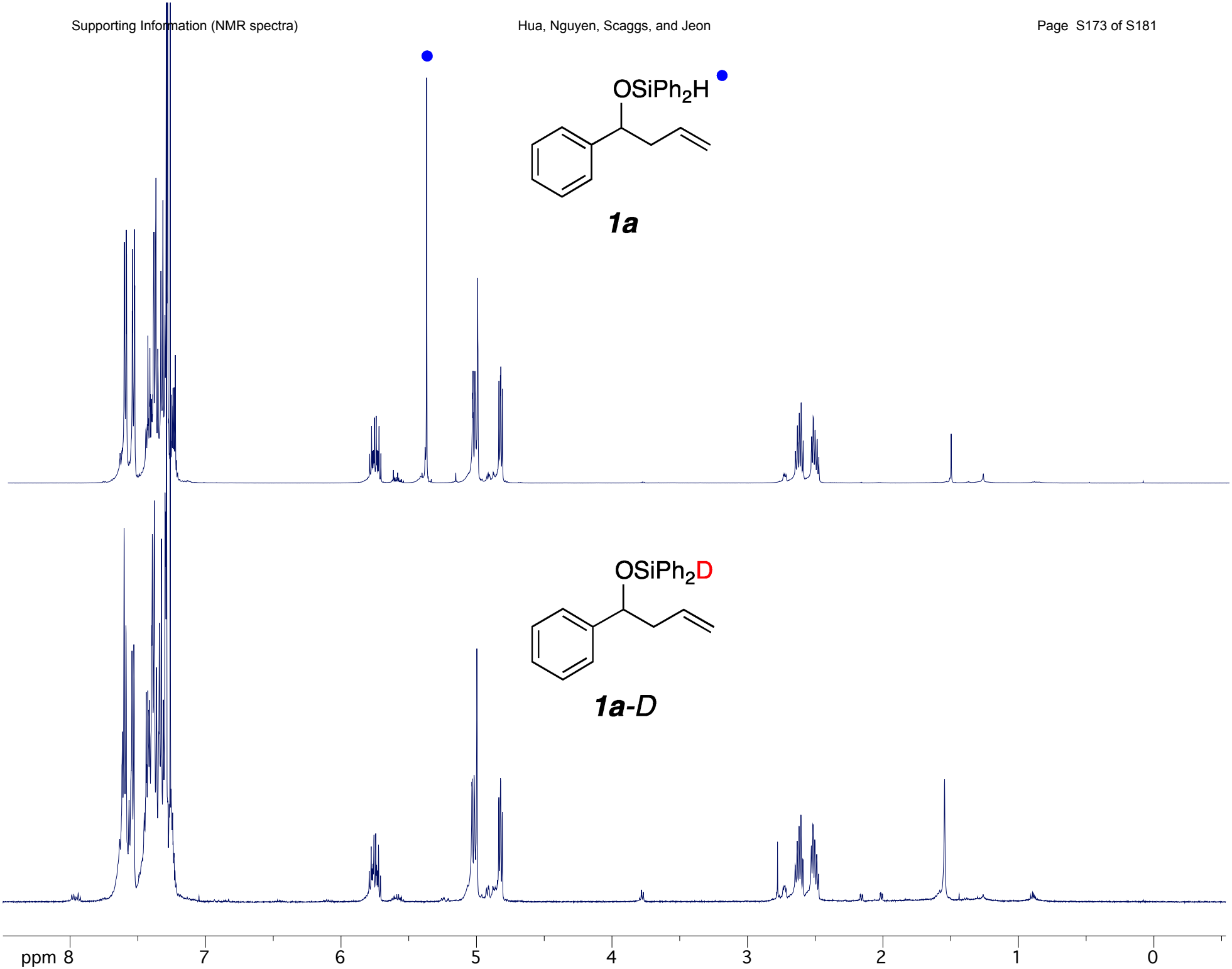


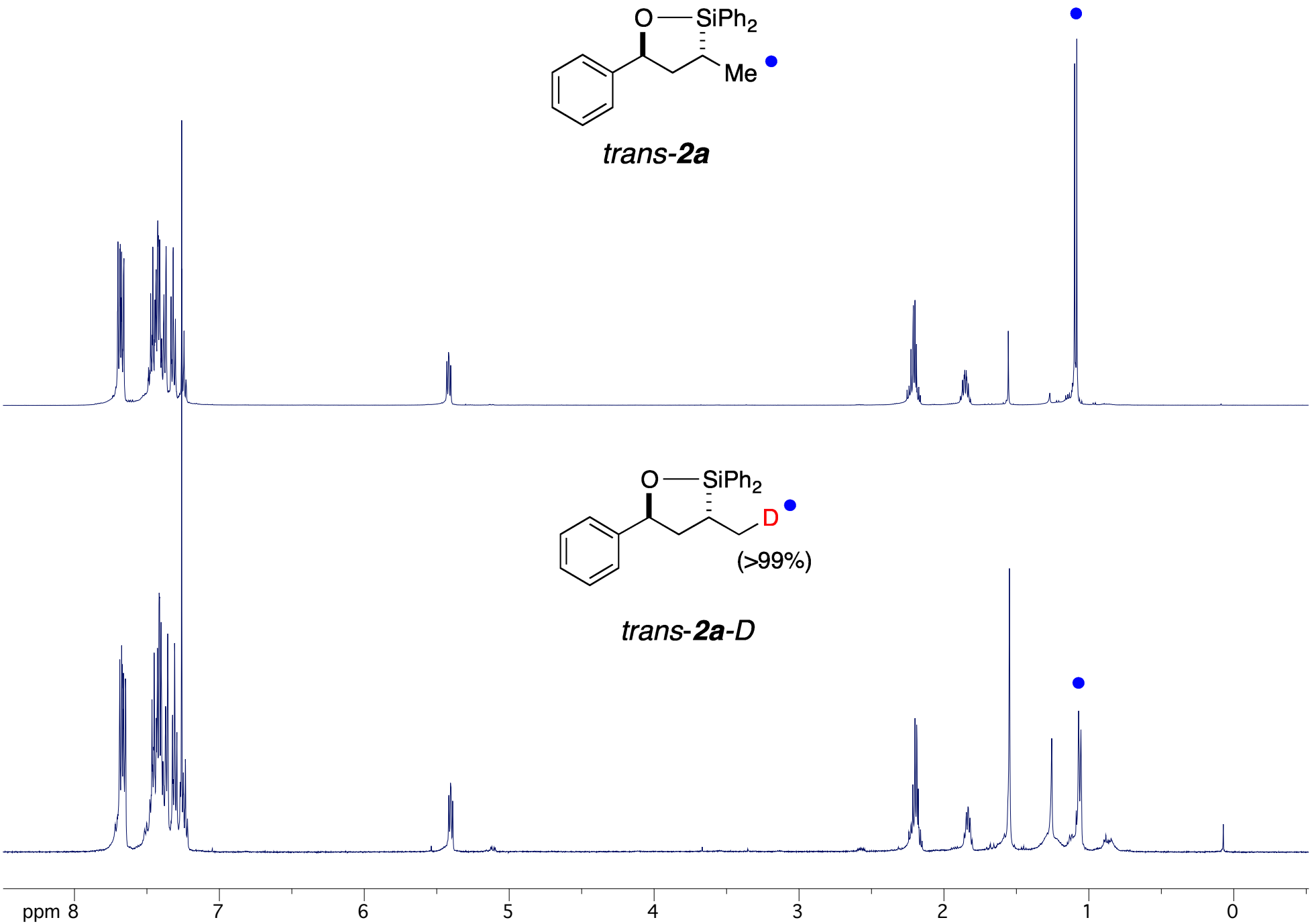
5l

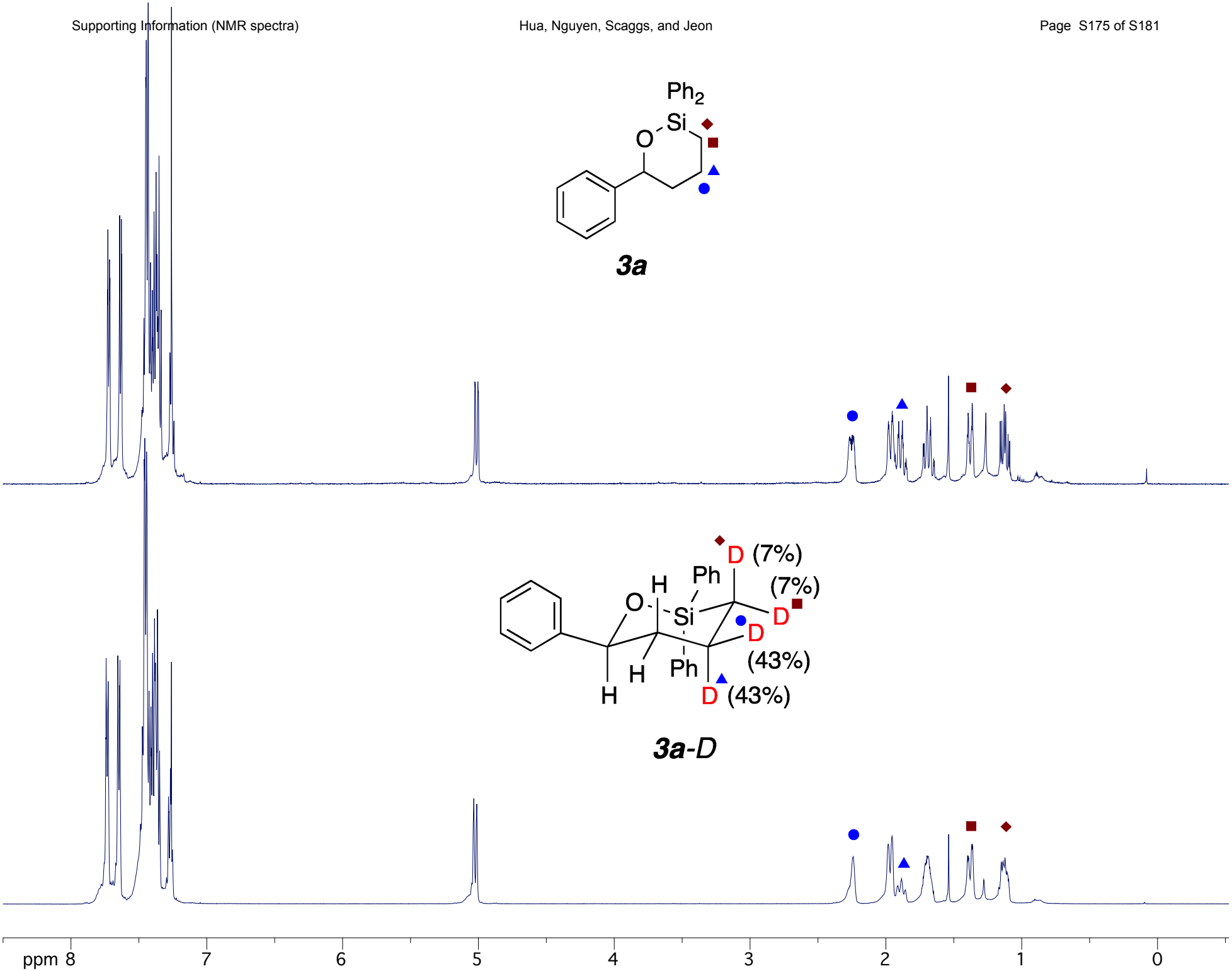


5l

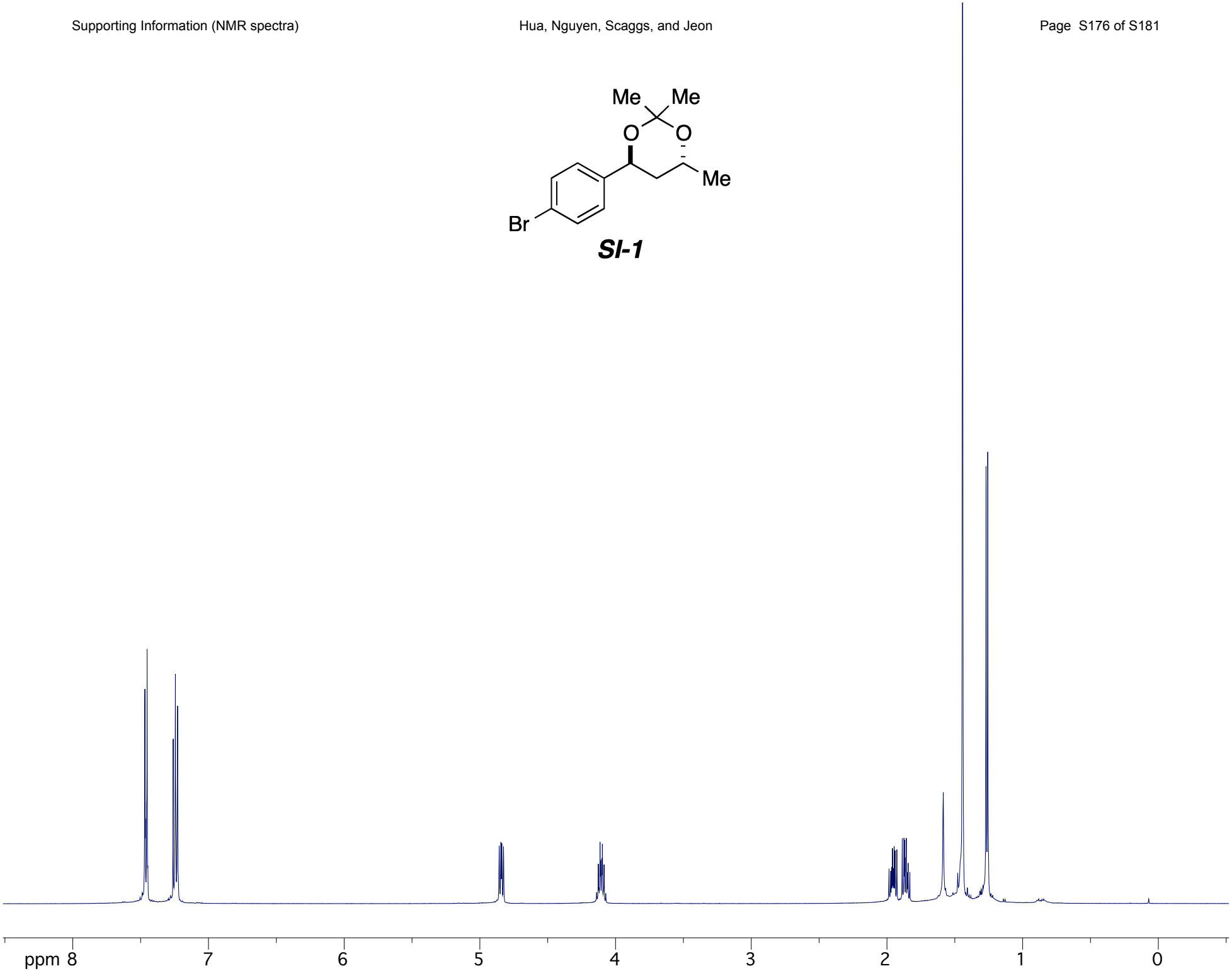



OSiPh2H
1a
OSiPh2D
1a-D
ppm 8
7
6
5
4
3
2
1
0

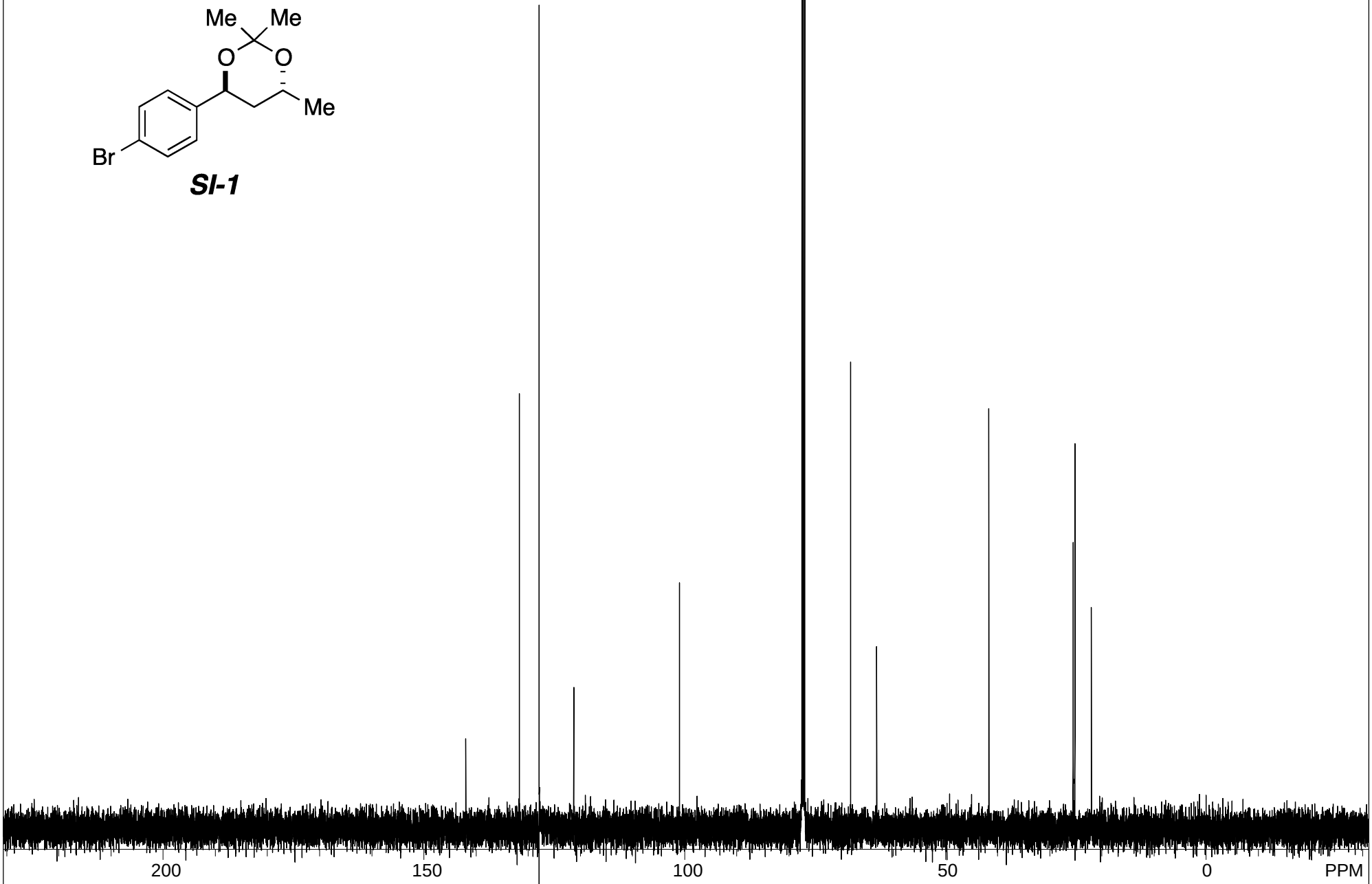

O
SiPh2
Me
trans-2a
O
SiPh2
D
(>99%)
trans-2a-D
ppm 8
7
6
5
4
3
2
1
0


Ph2
Si
O
3a
D (7%)
(7%)
D
D
(43%)
D (43%)
Ph
Ph
H
H
H
O
Si
3a-D
ppm 8
7
6
5
4
3
2
1
0

SI-1

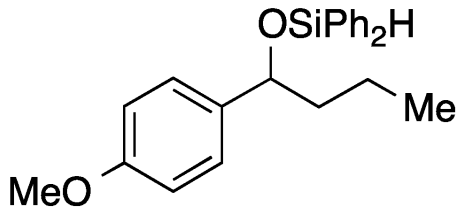


SI-1

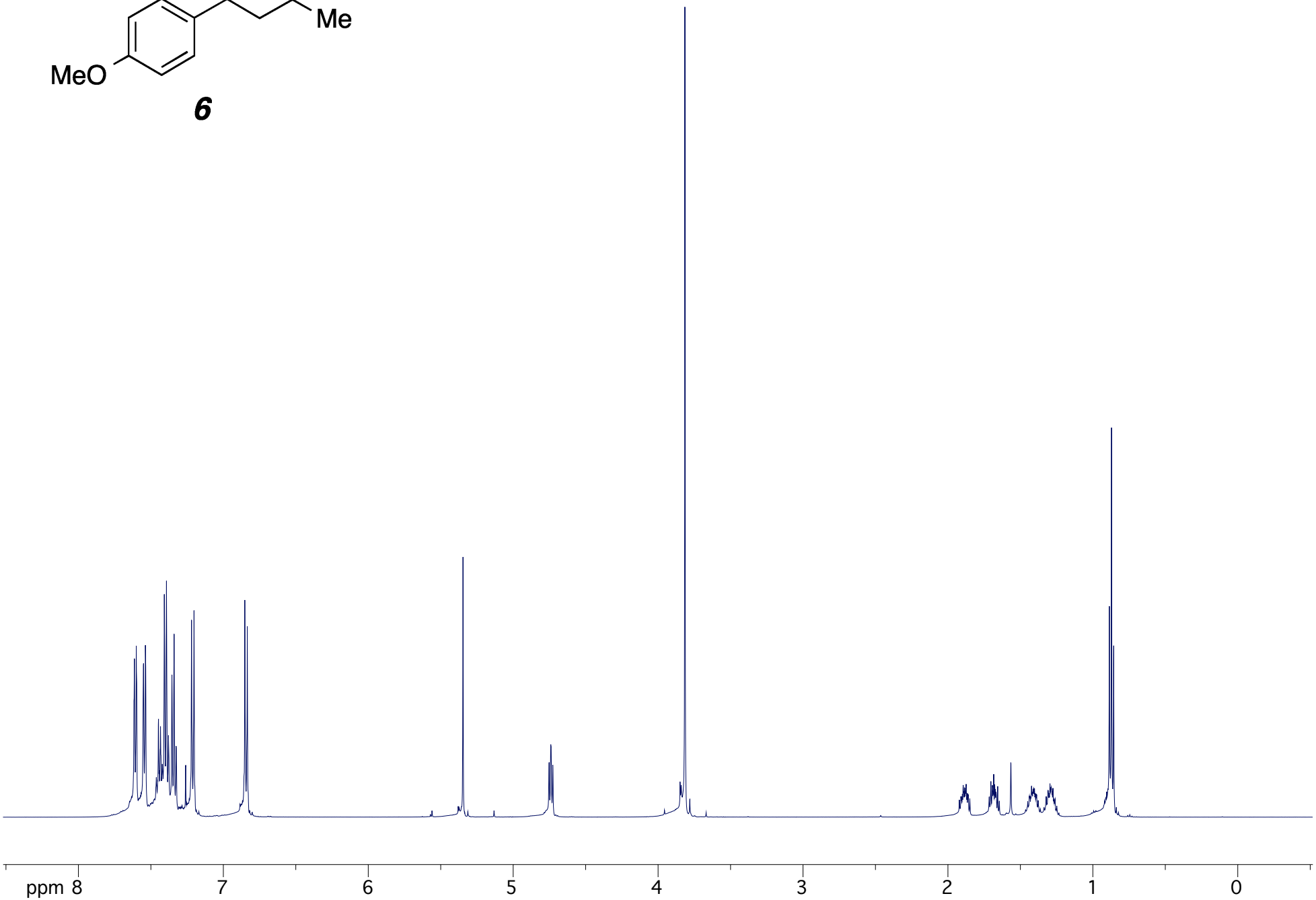


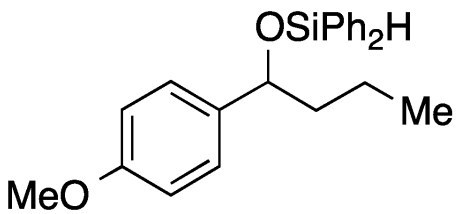
S#668626 (single pulse decoupled gated NOE) USER: delta -- DATE: Feb 8, 2013 09:08:10

F1: 125.778	F2: 500.162	SW1: 39308		OF1: 12592.6		PTS1d: 32768 , 32768
EX: single_pulse_dec		PW: 3.6 us, 30deg	PD: 2.0 sec	NA: 202	LB: 0.0	Nuts - JHJ1243_13C_MP_acetonide-1_13c_pBr.jdf

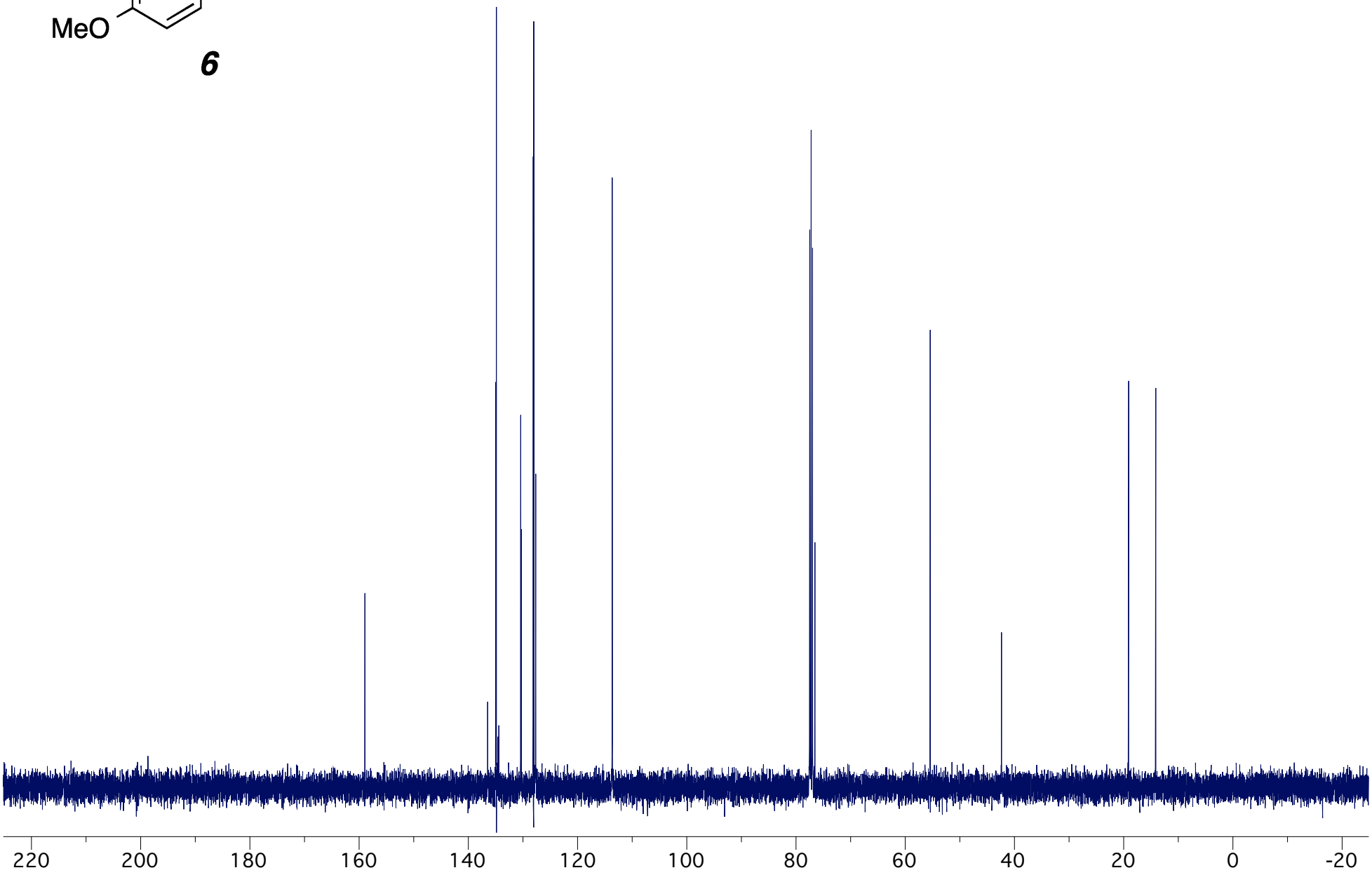


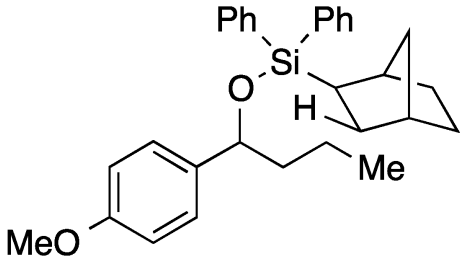
6



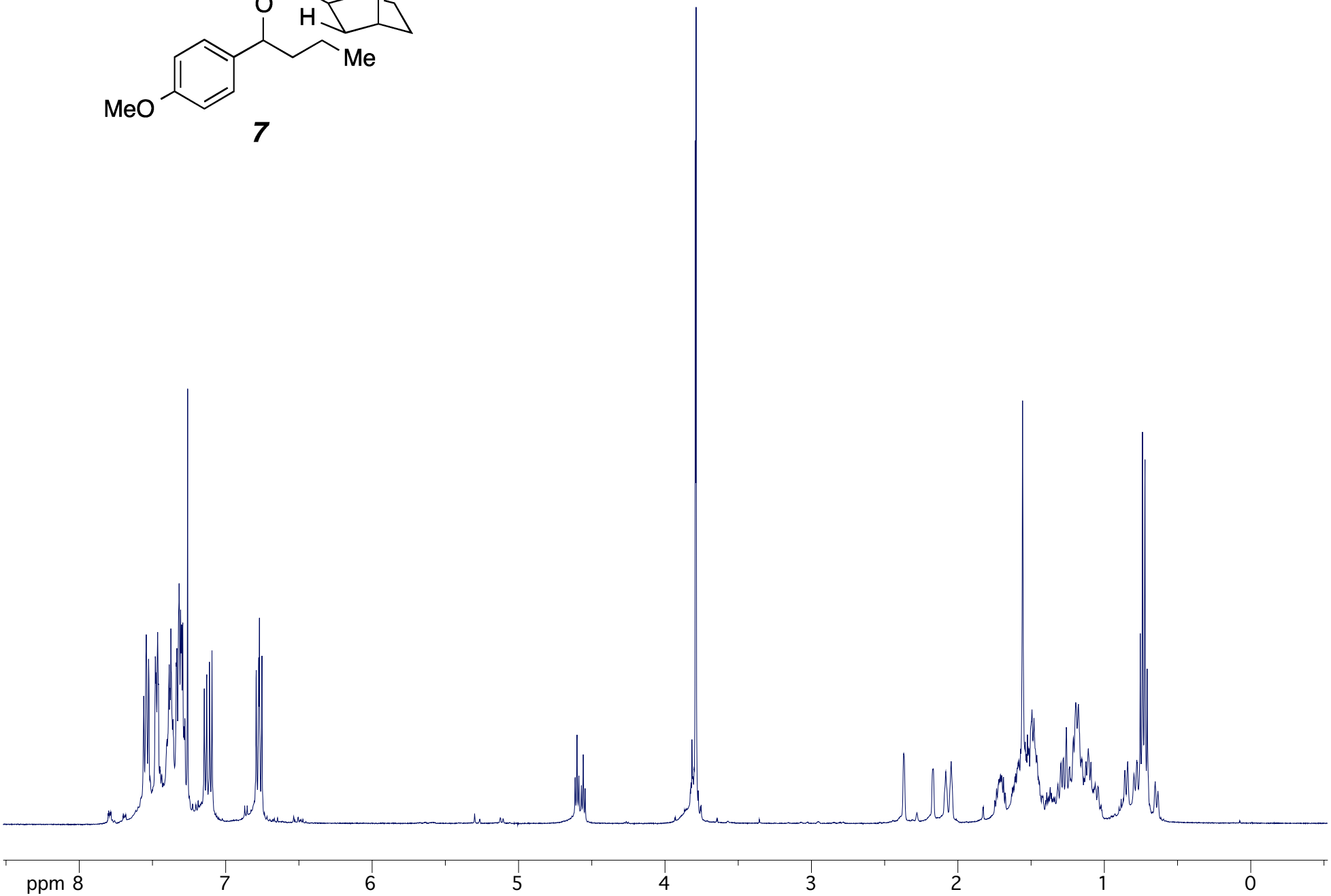
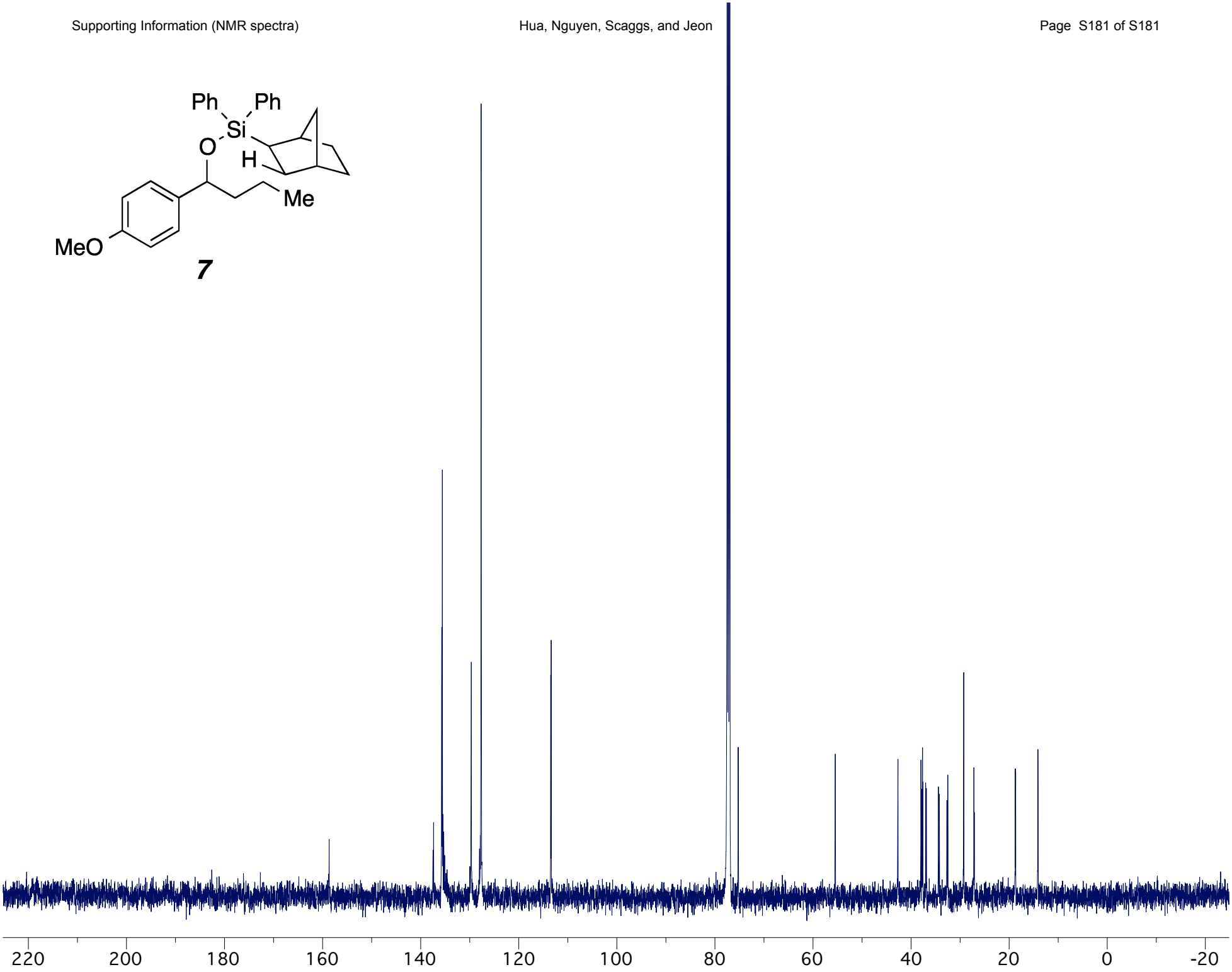


6





7



7