Supporting Information

Further Studies Towards the Stereocontrolled Synthesis of Silicon-Containing Peptide Mimics

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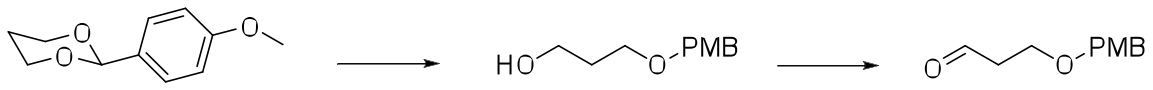
General Methods.

Unless otherwise noted all reactions were carried out under inert atmosphere. Solvents were dried according to standard procedures, reactions were monitored by thin-layer chromatography (TLC) analysis. All other chemicals were used as recieved from the appropriate suppliers. Flash chromatography was carried out on silica gel 60 (230-400 mesh). The ^{1}H NMR spectra were recorded at 400 MHz and ^{13}C NMR spectra were recorded at 100 MHz. The chemical shifts are reported in ppm downfield to TMS ($\delta = 0$) and referenced using the residual $CHCl_3$ resonance ($\delta = 7.26$) for ^{1}H NMR and the central $CDCl_3$ resonance ($\delta = 77.16$) for ^{13}C NMR. ^{1}H NMR spectra are reported as follows (s = singlet, d = doublet, t = triplet, q = quartet, quin = quintet, hex = hextet, br = broad; coupling constant(s) in Hz; integration). Optical rotations were measured at the sodium line at ambient temperature (22 ºC) in $CHCl_3$ solutions.

Additional experimental procedures.



74 **75** **76**

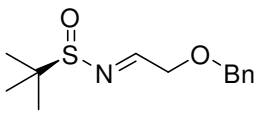
2-(4-Methoxyphenyl)-1,3-dioxane (74).[1] 4-Methoxybenzaldehyde dimethyl acetal (565 mg, 3.10 mmol) was dissolved in dry toluene (10 mL) in a dry round-bottomed flask. TsOH (31 mg, 0.18 mmol) and anhydrous $MgSO_4$ (1.0 g, 8.3 mmol) were added to the stirred solution, followed by addition of 1,3-propanediol (474 mg, 6.23 mmol). The reaction mixture was kept under argon flow and heated to 90 °C to remove methanol. After 2.5 h the reaction was judged complete by TLC and all volatiles were removed *in vacuo*. The resulting mixture was dissolved in EtOAc (30 mL) and washed with sat $NaHCO_3$ (4 × 30 mL). The organic phase was dried ($MgSO_4$), filtered and evaporated *in vacuo*, giving

the crude compound **74** (602 mg, 95% purity, 2.944 mmol, 95%) which could not be separated from residual anisaldehyde (approx. 5% according to ^{1}H NMR) by column chromatography. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.43–7.39 (m, 2H), 6.90–6.87 (m, 2H), 5.46 (s, 1H), 4.28–4.23 (m, 2H) 4.01–3.94 (m, 2H), 3.80 (s, 3H), 2.28–2.16 (m, 1H), 1.46–1.40 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 160.1, 131.5, 127.4 (2C) 113.7 (2C), 101.7, 67.5 (2C), 55.4, 25.9. HRMS $C_{11}H_{14}O_3$ [M+Na^+]; calculated: 217.0841, found: 217.0844.

3-(4-Methoxybenzyloxy)propan-1-ol (75). [1] Acetal **74** (597 mg, 3.07 mmol) was dissolved in Et_2O (15 mL) and then cooled to 0 °C under argon atmosphere. A 1.7 M solution of DIBAL in toluene (4.0 mL, 6.8 mmol) was added via syringe and stirring continued at 0 °C for 3.5 h. Then the reaction was quenched with an aqueous solution of Rochelles salt (0.5 M, 15 mL), resulting in a white jelly which was left to stir for additional 20 min. The aqueous solution was extracted with Et_2O (4 × 40 mL) and the combined organic phases were dried ($MgSO_4$), filtered and concentrated *in vacuo*. The pure product was obtained by column chromatography (increasing polarity from 25% to 65% EtOAc in pentane as eluant), which gave **75** (584 mg, 2.98 mmol, 97%) as a colourless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.25 (d, *J* = 8.4 Hz, 2H), 6.88 (d, *J* = 8.0 Hz, 2H), 4.45 (s, 2H) 3.80 (s, 3H), 3.78 (t, *J* = 5.6 Hz, 2H), 3.63 (t, *J* = 5.6 Hz, 2H), 2.32 (br s, 1H), 1.85 (quin, *J* = 5.6 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 159.4, 130.3, 129.4 (2C), 114.0 (2C), 73.1, 69.3, 62.1, 55.4, 32.2. HRMS $C_{11}H_{16}O_3$ [M+Na^+]; calculated: 219.0997, found: 219.1004.

3-(4-Methoxybenzyloxy)propanal (76). To a dry round-bottomed flask was added DMSO (1.7 mL, 24 mmol) and dry CH_2Cl_2 (25 mL), and the mixture was cooled to −60 °C under argon, followed by the dropwise addition of oxalylchloride (1.0 mL, 11.0 mmol). After 30 min alcohol **75** (974 mg, 4.96 mmol) in CH_2Cl_2 (5 mL) was added and the reaction was stirred for additional 1.5 h. NEt_3 (5 mL, 36.0 mmol) was then added and the reaction mixture was allowed to warm to rt. Then it was quenched with water

(50 mL), the phases were separated and the aqueous layer was extracted with CH_2Cl_2 (3 × 50 mL). The combined organic phases were washed with sat $NaHCO_3$ (3 × 50 mL), and brine (50 mL), dried ($MgSO_4$), filtered, and evaporated *in vacuo* giving **76** (1.02 g, 4.91 mmol, 99%), as a pungent yellow oil, which was used without further purification. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 9.78 (t, *J* = 2.0 Hz, 1H), 7.25 (d, *J* = 8.8 Hz, 2H), 6.88 (d, *J* = 8.4 Hz, 2H), 4.46 (s, 2H), 3.80 (s, 3H), 3.78 (t, *J* = 6.0 Hz, 2H), 2.67 (dt, *J* = 6.0, 2.0 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 201.3, 159.5, 130.1, 129.5 (2C), 114.0 (2C), 73.1, 63.7, 55.4, 44.0. HRMS $C_{11}H_{14}O_3$ [M+Na$^+$]; calculated: 217.0841, found: 217.0842.



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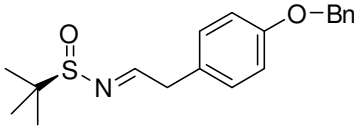

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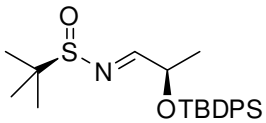

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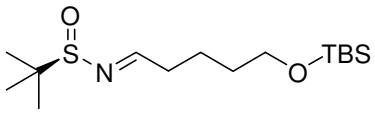
(*R*,*E*)-*N*-(2-(Benzyloxy)ethylidene)-2-methylpropane-2-sulfinamide (12).[2] General Procedure for Preparation of Sulfinimines. Method A. 2-Benzyloxyethanal[3] (400 mg, 2.66 mmol) was dissolved in dry CH_2Cl_2 (25 mL) and (*R*)-*tert*-butylsulfinamide (330 mg, 2.71 mmol), PPTS (5 mg, 0.02 mmol) and $MgSO_4$ were added. The reaction was heated at reflux for 18 h. Then the reaction mixture was filtered and the solids washed with CH_2Cl_2 (2 × 10 mL). The combined filtrates were evaporated *in vacuo* and the pure product was obtained by column chromatography (20% EtOAc in pentane as eluant) which gave **12** (667 mg, 79%) as a colourless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 8.13 (t, *J* = 3.2 Hz, 1H), 7.32–7.38 (m, 5H), 4.64 (s, 2H), 4.43 (dd, *J* = 16.4, 3.2 Hz, 1H), 4.38 (dd, *J* = 16.4, 3.2 Hz, 1H), 1.21 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 166.9, 137.4, 128.7 (2C), 128.2, 128.0 (2C), 73.5, 71.4, 57.1, 22.6 (3C). HRMS $C_{13}H_{19}NO_2S$ [M+Na$^+$]; calculated: 276.1029, found: 276.1038.

(*R*,*E*)-*N*-(2-(4-(Benzyloxy)phenyl)ethylidene)-2-methylpropane-2-sulfinamide (14). It was prepared from 2-(4-(Benzyloxy)phenyl)ethanal[4] (520 mg, 2.20 mmol 1.22 equiv) according to Method A. Increasing polarity from 15% to 20% EtOAc in pentane was used as eluant for column chromatography giving **14** (58%) as an oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 8.12 (t, *J* = 4.8 Hz, 1H), 7.45–7.30 (m, 5H), 7.15 (d, *J* = 8.4 Hz, 2H), 6.95 (d, *J* = 8.4 Hz, 2H), 5.06 (s, 2H), 3.77 (ABX system, *J* = 5.0 Hz, 1H), 3.74 (ABX system, *J* = 5.0 Hz, 1H), 1.21 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 167.7, 158.0, 137.0, 130.3 (2C), 128.6 (2C), 128.0, 127.5 (2C), 127.1, 115.3 (2C), 70.1, 56.9, 41.8, 22.4 (3C). HRMS $C_{19}H_{23}NO_2S$ [M+Na$^+$]; calculated: 352.1347, found: 352.1351.

(*R*,*E*)-*N*-((*R*)-2-(*tert*-Butyldiphenylsilyloxy)propylidene)-2-methylpropane-2-sulfinamide (16).[5] It was prepared from (*R*)-2-(*tert*-butyldiphenylsilyloxy)propanal[6] according to Method A. Increasing polarity from 5% to 10% EtOAc in pentane was used as eluant for column chromatography giving **16** (89%) as an oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 8.02 (d, *J* = 4.0 Hz, 1H), 7.69–7.63 (m, 4H), 7.46–7.33 (m, 6H), 4.59 (qd, *J* = 6.8, 4.0 Hz, 1H), 1.28 (d, *J* = 6.8 Hz, 3H), 1.14 (s, 9H), 1.08 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 171.4, 136.0 (4C), 134.0 (2C) 130.0 (2C), 127.8 (4C), 71.5, 56.9, 27.0 (3C), 22.5 (3C), 21.7, 19.4. HRMS $C_{23}H_{33}NO_2SSi$ [M+Na$^+$]; calculated: 438.1899, found: 438.1901.



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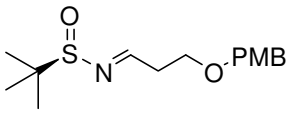

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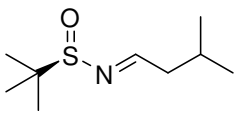

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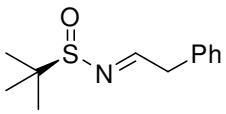
(*R*,*E*)-*N*-(5-(*tert*-Butyldimethylsilyloxy)pentylidene)-2-methylpropane-2-sulfinamide (77). It was prepared from 5-(*tert*-butyldimethylsilyloxy)pentanal[7] according to Method A. Increasing polarity from 5% to 20% EtOAc in pentane was used as eluant for column chromatography giving **77** (85%) as an oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 8.07 (t, *J* = 4.8 Hz, 1H), 3.62 (t, *J* = 6.4 Hz, 2H), 2.53 (td, *J* = 6.8,

4.8 Hz, 2H), 1.73–1.64 (m, 2H), 1.62–1.53 (m, 2H), 1.18 (s, 9H), 0.88 (s, 9H), 0.03 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 169.7, 62.8, 36.0, 32.4, 28.3, 26.1 (3C), 24.9, 22.5 (3C), 22.1, 18.5, –5.2. HRMS $C_{15}H_{33}NO_2SSi$ [M+Na^+]; calculated: 342.1899, found: 342.1896.

(*R*,*E*)-*N*-(3-(4-Methoxybenzyloxy)propylidene)-2-methylpropane-2-sulfinamide (78). It was prepared from aldehyde **76** according to Method A. Increasing polarity from 5% to 20% EtOAc in pentane was used as eluant for column chromatography giving **78** (77%) as a pale yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 8.09 (t, *J* = 4.4 Hz, 1H), 7.23 (m, 2H), 6.87 (m, 2H), 4.44 (s, 2H), 3.79 (s, 3H), 3.74 (t, *J* = 6.0 Hz, 2H), 2.79 (td, *J* = 6.0, 4.4 Hz, 2H), 1.20 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 167.5, 159.4, 130.1, 129.4 (2C), 113.9 (2C), 73.0, 66.0, 56.8, 55.4, 36.7, 22.5 (3C). HRMS $C_{15}H_{23}NO_3S$ [M+Na^+]; calculated: 320.1296, found: 320.1295.

(*R*,*E*)-2-Methyl-*N*-(3-methylbutylidene)propane-2-sulfinamide (46).[8] It was prepared from isovaleraldehyde according to Method A. Increasing polarity from 5% to 10% EtOAc in pentane was used as eluant for column chromatography, giving **46** (95%) as an oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 8.05 (t, *J* = 5.2 Hz, 1H), 2.40 (ddd, *J* = 6.8, 5.6, 1.6 Hz, 2H), 2.05 (m, 1H), 1.19 (s, 9H), 0.98 (d, *J* = 6.8 Hz, 3H), 0.98 (d, *J* = 6.8 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 169.4, 56.5, 45.0, 26.2, 22.6, 22.5, 22.4 (3C). HRMS $C_9H_{19}NOS$ [M+Na^+]; calculated: 212.1085, found: 212.1087.



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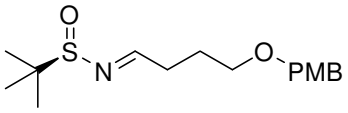

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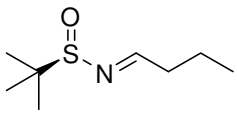

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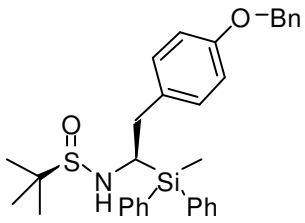
(*R*,*E*)-2-Methyl-*N*-(2-phenylethylidene)propane-2-sulfinamide (56).[9] It was prepared from phenyl acetaldehyde according to Method A. CH_2Cl_2 was used as eluant for column chromatography giving **56** (87%) as an oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 8.13 (t, *J* = 5.2 Hz, 1H), 7.35–7.22 (m, 5H), 3.85

(dd, *J* = 15.2, 5.2 Hz, 1H), 3.81 (dd, *J* = 15.2, 5.2 Hz, 1H), 1.19 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 167.6, 135.0, 129.4 (2C), 129.0 (2C), 127.3, 57.0, 42.8, 22.5 (3C). HRMS $C_{12}H_{17}NOS$ [M+Na^+]; calculated: 246.0929, found: 246.0933.

(*R*,*E*)-*N*-(4-(4-Methoxybenzyloxy)butylidene)-2-methylpropane-2-sulfinamide (79).[10] General Procedure for Preparation of Sulfinimines. Method B. (*R*)-*tert*-Butylsulfinamide (672 mg, 5.54 mmol) and 4-(4-methoxybenzyloxy)butanal[11] (5.09 mmol 0.92 equiv) were dissolved in CH_2Cl_2 (18 mL) and Cs_2CO_3 (2.17 g, 6.65 mmol, 1.2 equiv) was added. The mixture was heated to reflux for 18 h, then cooled and filtered through a pad of celite. The solids were washed with CH_2Cl_2, and then the combined filtrates were evaporated *in vacuo*. The pure product was obtained by column chromatography using 5% to 40% EtOAc in pentane as eluant, giving **79** (1.0 g, 3.21 mmol, 63%) as an oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 8.08 (t, *J* = 4.4 Hz, 1H), 7.23 (dt, *J* = 8.8, 2.0 Hz, 2H), 6.86 (dt, *J* = 8.8, 2.0 Hz, 2H), 4.41 (s, 2H), 3.78 (s, 3H), 3.49 (t, *J* = 6.0 Hz, 2H), 2.60 (td, *J* = 7.2, 4.4 Hz, 2H), 1.92 (pent, *J* = 6.8 Hz, 2H), 1.16 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 169.0, 159.1, 130.3, 129.2 (2C), 113.7 (2C), 72.6, 68.8, 56.4, 55.2, 32.9, 25.5, 22.2 (3C). HRMS $C_{16}H_{25}NO_3S$ [M+Na^+]; calculated: 334.1453, found: 334.1407.

(*R*,*E*)-*N*-Butylidene-2-methylpropane-2-sulfinamide (80).[12] It was prepared from butyraldehyde according to Method B. 10% EtOAc in pentane was used as eluant for column chromatography, giving **80** (63%) as an oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 8.06 (t, *J* = 4.8 Hz, 1H), 2.49 (td, *J* = 7.2, 4.8 Hz, 2H), 1.65 (sext, *J* = 7.2 Hz, 2H), 1.19 (s, 9H), 0.99 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 169.8, 56.6, 38.2, 22.5 (3C), 19.1, 13.9. HRMS $C_8H_{17}NOS$ [M+Na^+]; calculated: 198.0929, found: 198.0925.



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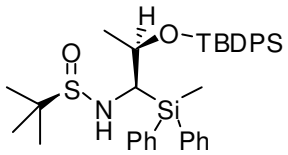

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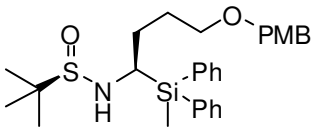

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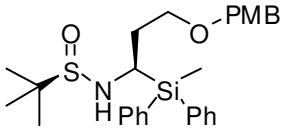

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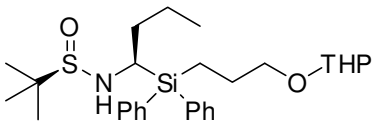
(*R*)-*N*-((*R*)-2-(4-(Benzyloxy)phenyl)-1-(methyldiphenylsilyl)ethyl)-2-methylpropane-2-sulfinamide (15). General Procedure for the Addition of (Diphenylmethylsilyl)lithium to Sulfinimines. Lithium (36 mg, 6.0 mmol, 12.0 equiv) was suspended in THF (5 mL) under argon atmosphere, and then diphenylmethylchlorosilane (0.31 ml, 1.50 mmol, 3.0 equiv) was added, before the mixture was stirred at rt for 4 h. In a separate flask sulfinimine **14** (164.5 mg, 0.50 mmol, 1 equiv) was dissolved in THF (5 mL) and the solution cooled to −78 ^{o}C under argon atmosphere. To this cooled solution, the solution of lithium diphenylmethylsilane was added dropwise over 5 min via syringe. The solution was stirred at −78 ^{o}C for 18h, then water (2 mL) was added and the mixture allowed to warm to rt. The mixture was poured into water (50 mL) and extracted with EtOAc (3 × 20 mL), then the combined organic portions were dried ($MgSO_4$), filtered and evaporated *in vacuo*. The pure product was obtained by column chromatography using 15% to 30% EtOAc in pentane as eluant, giving **15** (120 mg, 0.23 mmol, 45%) as an oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.57−7.31 (m, 15H), 7.10 (d, *J* = 8.8 Hz, 2H), 6.87 (d, *J* = 8.8 Hz, 2H), 5.03 (s, 2H), 3.75 (dt, *J* = 9.2, 6.4 Hz, 1H), 3.17 (d, *J* = 9.2 Hz, 1H), 3.14 (ABX system, *J* = 6.4 Hz, 1H), 3.06 (ABX system, *J* = 6.4 Hz, 1H), 1.02 (s, 9H), 0.47 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 157.5, 137.2, 135.0 (4C), 134.8, 134.7, 131.0 (2C), 130.8 (2C), 129.6 (2C), 128.6 (2C), 128.0 (4C), 127.5 (2C), 114.8 (2C), 70.0, 56.4, 47.5, 38.5, 22.5 (3C), −5.1. HRMS $C_{32}H_{37}NO_2SSi$ [M+Na^+]; calculated: 550.2212, found: 550.2222.

(*R*)-*N*-((1*R*,2*R*)-2-(*tert*-Butyldiphenylsilyloxy)-1-(methyldiphenylsilyl)propyl)-2-methylpropane-2-sulfinamide (17). It was prepared from sulfinimine **16** according to the previous general procedure. An 8:8:1 mixture of pentane:CH_2Cl_2:diethylether was used as eluant for column chromatography, giving **17** (48%) as an oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.71–7.56 (m, 6H), 7.47–7.30 (m, 14H), 4.48 (qd, *J* = 6.4, 2.8 Hz, 1H), 3.45 (m, 1H), 3.36 (m, 1H), 1.16 (d, *J* = 6.4 Hz, 3H), 1.07 (s, 9H), 0.99 (s, 9H), 0.83 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 136.1 (2C), 136.0 (2C), 135.8, 135.5 (2C), 135.3, 134.9 (2C), 134.2, 133.7, 129.8, 129.7 (2C), 129.5, 128.0 (4C), 127.8 (2C), 127.6 (2C), 71.5, 56.4, 52.8, 27.3 (3C), 22.7 (3C), 21.9, 19.3, −3.4. HRMS $C_{36}H_{47}NO_2SSi_2$ [M+Na^+]; calculated: 636.2746, found: 636.2767.

(*R*)-*N*-((*R*)-4-(4-Methoxybenzyloxy)-1-(methyldiphenylsilyl)butyl)-2-methylpropane-2-sulfinamide (23).[10] It was prepared from sulfinimine **79** according to the previous general procedure. Increasing polarity from 10% to 60% EtOAc in pentane was used as eluant for column chromatography giving **23** (97%) as an oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.53–7.60 (m, 4H), 7.32–7.43 (m, 6H), 7.20 (dt, *J* = 8.4, 2.8 Hz, 2H), 6.86 (dt, *J* = 8.4, 2.8 Hz, 2H), 4.38 (s, 2H), 3.79 (s, 3H), 3.36–3.50 (m, 2H), 2.87 (d, *J* = 10 Hz, 1H), 1.87–2.04 (m, 2H), 1.59–1.80 (m, 2H), 1.01 (s, 9H), 0.63 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 158.9, 134.9 (2C), 134.8 (2C), 134.5, 134.4, 130.7, 129.6, 129.5, 129.0 (2C), 127.93 (2C), 127.91 (2C), 113.6 (2C), 72.2, 69.4, 56.1, 55.1, 46.5, 29.9, 27.4, 22.5 (3C), −5.2. HRMS $C_{29}H_{39}NO_3SSi$ [M+Na^+]; calculated: 532.2318, found: 532.2310.

(*R*)-*N*-((*R*)-3-(4-Methoxybenzyloxy)-1-(methyldiphenylsilyl)propyl)-2-methylpropane-2-sulfinamide (31). It was prepared from sulfinimine **78** according to the previous general procedure. Increasing polarity from 25% to 60% EtOAc in pentane was used as eluant for column chromatography giving **31** (87%) as a colourless wax. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.57–7.55 (m, 4H), 7.40–7.33 (m, 6H),

7.23–7.21 (m, 2H), 6.86–6.84 (m, 2H), 4.33 (s, 2H), 3.80 (s, 3H), 3.69 (td, *J* = 8.8, 4.8 Hz, 1H), 3.65–3.60 (m, 2H), 3.39 (d, *J* = 8.8 Hz, 1H), 2.21–2.13 (m, 1H), 1.81–1.73 (m, 1H), 0.97 (s, 9H), 0.62 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 159.2, 135.2 (2C), 135.1 (2C), 134.81, 134.79, 130.8, 129.8 (2C), 129.6 (2C), 128.1 (4C), 113.8 (2C), 72.7, 68.9, 56.3, 55.4, 44.8, 33.1, 22.8 (3C), –4.9. HRMS $C_{28}H_{37}NO_3SSi$ [M+Na$^+$]; calculated: 518.2161, found: 518.2167.



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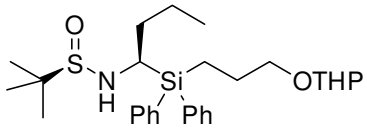

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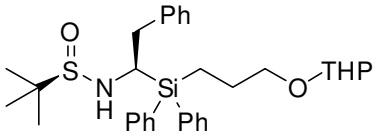

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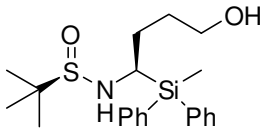
(*R*)-*N*-((1*R*)-1-{Diphenyl[3-(tetrahydro-*2H*-pyran-2-yloxy)propyl]silyl}butyl)-2-methylpropane-2-sulfinamide (39).[10] General Procedure for the Addition of (Diphenylalkylsilyl)lithium to Sulfinimines. Diphenyl[3-(tetrahydro-2*H*-pyran-2-yloxy)propyl]silane[10] (70 mg, 0.21 mmo) was dissolved in dry THF (2 mL) and then freshly cut lithium pieces (15 mg, 2.1 mmol) were added. The mixture was stirred under argon atmosphere for 4 h, by which time the mixture had turned a rich dark brown colour. In a separate flask, the imine **80** (19 mg, 0.11 mmol) was dissolved in dry THF (2 mL) and the solution was cooled to −78 ^{o}C. To this cooled solution was added the silyl lithium reagent (2 mL) dropwise via syringe over 3-5 min. The mixture was stirred at –78 ^{o}C for 18 h, and then quenched via the addition of water. It was poured into water (30 mL) and extracted with EtOAc (3 × 20 mL). The combined organic portions were dried ($MgSO_4$), filtered and evaporated *in vacuo*. The pure product was obtained by column chromatography (increasing polarity from 20% to 30% EtOAc in pentane as eluant) which gave **39** (42 mg, 0.084 mmol, 76%) as a colorless oil ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.57–7.53 (m, 4H), 7.44–7.34 (m, 6H), 4.51 (t, *J* = 2.8 Hz, 1H), 3.84–3.79 (m, 1H), 3.70–3.64 (m, 1H), 3.48–3.38 (m, 2H), 3.56–3.31 (m, 1H), 2.68 (br d, *J* = 10.0 Hz, 1H), 1.82–1.37 (m, 12H), 1.19–1.14 (m,

2H), 1.04 (s, 9H), 0.88 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 135.7 (2C), 135.6 (2C), 133.3, 133.1, 129.94, 129.88, 128.2 (2C), 128.1 (2C), 98.9, 70.1, 62.5, 56.6, 46.0, 36.0, 30.9, 25.6, 23.9, 22.8 (3C), 21.0, 19.8, 14.2, 8.4. HRMS $C_{28}H_{43}NO_3SSi$ [M+Na^+]; calculated: 524.2631, found: 524.2624.

(*R*)-*N*-((1*R*)-1-(Diphenyl(3-(tetrahydro-2*H*-pyran-2-yloxy)propyl)silyl)-3-methylbutyl)-2-methyl-propane-2-sulfinamide (48). It was prepared from sulfinimine **46** according to the previous general procedure. The pure product was obtained by column chromatography using 5% to 30% EtOAc in pentane as eluant which gave **48** (99%) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.60–7.50 (m, 4H), 7.44–7.32 (m, 6H), 4.55–4.46 (m 1H), 3.81 (ddd, *J* = 10.8, 7.6, 3.2 Hz, 1H), 3.66 (dtd, *J* = 8.8, 7.2, 2.0 Hz, 1H), 5.53–3.40 (m, 2H), 3.32 (dtd, *J* = 9.6, 6.8, 2.8 Hz, 1H), 2.56 (d, *J* = 9.6 Hz, 1H), 2.13–2.02 (m, 1H), 1.85–1.36 (m, 10H), 1.20–1.10 (m, 2H), 1.02 (s, 9H), 0.94 (d, *J* = 6.4 Hz, 3H), 0.87 (d, *J* = 6.8 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 135.7 (2C), 135.6 (2C), 133.1, 132.8, 129.91, 128.89, 128.2 (2C), 128.1 (2C), 109.9, 98.9, 70.0, 62.5, 56.6, 43.6, 42.9, 30.8, 25.6, 25.0, 23.9, 22.8 (3C), 21.3, 19.8, 8.3. HRMS $C_{29}H_{45}NO_3SSi$ [M+Na^+]; calculated: 538.2782, found: 538.2801.

***S*(*R*)-*N*-((1*R*)-1-{Diphenyl[3-(tetrahydro-2*H*-pyran-2-yloxy)propyl]silyl}-2-phenylethyl)-2-methyl-ropane-2-sulfinamide (58).** It was prepared from sulfinimine **56** according to the previous general procedure. The pure product was obtained by column chromatography using 10% to 50% EtOAc in pentane as eluant which gave **58** (47%) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.48–7.53 (m, 4H), 7.33–7.42 (m, 5H), 7.15–7.23 (m, 5H), 4.46 (dt, *J* = 5.2, 3.2 Hz, 1H), 3.76–3.81 (m, 2H), 3.54 (dtd, *J* = 8.0, 6.8, 1.2 Hz, 1H), 3.43–3.45 (m, 1H), 3.90 (d, *J* = 9.6, 6.8, 4.8 Hz, 1H), 3.08–3.11 (m, 2H), 3.02 (d, *J* = 8.4 Hz, 1H), 1.75–1.83 (m, 1H), 1.62–1.69 (m, 1H),

1.40−1.55 (m, 6H), 0.96−1.06 (m, 2H), 1.00 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 138.9, 135.6 (4C), 132.9 (2C), 129.8, 129.7, 129.7 (2C), 128.4 (2C), 128.1 (2C), 128.0 (2C), 126.6, 98.7, 69.9, 62.3, 56.6, 46.8, 39.8, 30.8, 25.5, 23.9, 22.6 (3C), 19.7, 8.7. HRMS $C_{32}H_{43}NO_3SSi$ [M+Na$^+$]; calculated: 572.2631, found: 572.2629.



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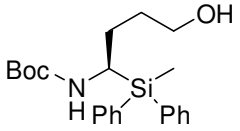

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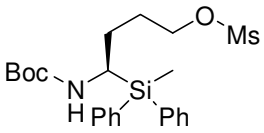

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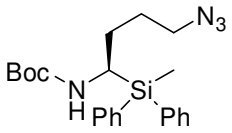

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(*R*)-*N*-((*R*)-4-Hydroxy-1-(methyldiphenylsilyl)butyl)-2-methylpropane-2-sulfinamide (24). *p*-Methoxybencyl ether **23** (635 mg, 1.23 mmol) was dissolved in CH_2Cl_2 (45 mL), and then water (6 mL) and DDQ (362 mg, 1.55 mmol) were added. The mixture was stirred vigorously at rt for 3 h, then poured into water (50 mL) and extracted with CH_2Cl_2 (3 × 40 mL). The combined organic portions were dried ($MgSO_4$), filtered and evaporated *in vacuo*. The pure product was obtained by column chromatography (increasing polarity from 1% to 5% MeOH in CH_2Cl_2 as eluant) which gave **24** (461 mg, 1.18 mmol, 96%) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.59−7.52 (m, 4H), 7.43−7.33 (m, 6H), 3.63 (dt, *J* = 5.4, 5.4 Hz, 1H), 3.52 (ddd, *J* = 11.6, 7.2, 4.4 Hz, 1H), 3.42 (ddd, *J* = 10.0, 8.0, 4.4 Hz, 1H), 3.07 (d, *J* = 10.0 Hz, 1H), 2.84 (br s, 1H), 2.05−1.94 (m, 1H), 1.85−1.73 (m, 1H), 1.73−1.61 (m, 2H), 1.02 (s, 9H), 0.62 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 135.1 (2C), 134.9 (2C), 134.6, 134.4, 129.8, 129.7, 128.1 (2C), 128.0 (2C), 62.2, 56.4, 47.2, 30.4, 29.7, 22.7 (3C), −5.2. HRMS $C_{21}H_{31}NO_2SSi$ [M+Na$^+$]; calculated: 412.1737, found: 412.1744.

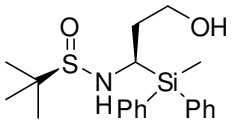
(*R*)-*tert*-Butyl 4-hydroxy-1-(methyldiphenylsilyl)butylcarbamate (25). Sulfinamide **24** (461 mg, 1.18 mmol) was dissolved in anhydrous HCl in MeOH (20 mL, 0.5 M) and the mixture was stirred at rt for 3 h. All volatiles were removed *in vacuo* giving the crude amino alcohol. The residue was dissolved in dry

CH_2Cl_2 (15 mL) and then Boc_2O (250 mg, 1.19 mmol) and NEt_3 (250 mg, 2.50 mmol) were added. The mixture was stirred at rt for 18 h, then poured into water (50 mL) and extracted with CH_2Cl_2 (3 × 20 mL). The combined organic portions were dried ($MgSO_4$), filtered and evaporated in vacuo. The pure product was obtained by column chromatography (increasing polarity from 10% to 50% EtOAc in pentane as eluant) which gave **25** (434 mg, 1.13 mmol, 95%) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.57 (dd, *J* = 7.6, 1.6 Hz, 2H), 7.53 (dd, *J* = 7.6, 1.6 Hz, 2H), 7.43–7.33 (m, 6H), 4.32 (d, *J* = 10.0 Hz, 1H), 3.85 (td, *J* = 10.8, 2.8 Hz, 1H), 3.70–3.56 (m, 2H), 1.93 (br s, 1H), 1.76–1.49 (m, 3H), 1.48–1.36 (m, 1H), 1.39 (s, 9H), 0.60 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 156.7, 135.1 (2C), 135.0 (2C), 134.5, 134.3, 129.82, 129.76, 128.1 (4C), 79.4, 62.7, 38.6, 29.9, 28.8, 28.5 (3C), –5.5. HRMS $C_{22}H_{31}NO_3Si$ [M+Na^+]; calculated: 408.1965, found: 408.1979.

(*R*)-4-(*tert*-Butoxycarbonylamino)-4-(methyldiphenylsilyl)butyl methanesulfonate (26). The alcohol **25** (57 mg, 0.15 mmol) was dissolved in CH_2Cl_2 (5 mL) and then NEt_3 (30 mg, 0.30 mmol) and MsCl (19 mg, 0.23 mmol) were added. The mixture was stirred at rt for 30 min, then poured into water (50 mL) and extracted with CH_2Cl_2 (3 × 20 mL). The combined organic portions were dried ($MgSO_4$), filtered and evaporated *in vacuo*, giving the crude mesylate **26** that was used without further purification. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.59–7.50 (m, 4H), 7.44–7.32 (m, 6H), 4.30–4.20 (m, 2H), 3.84–3.74 (m, 1H), 3.65 (br s, 1H), 2.92 (s, 3H), 1.94–1.80 (m, 1H), 1.80–1.67 (m, 2H), 1.47–1.34 (m, 1H), 1.38 (s, 9H), 0.60 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 156.5, 135.0 (2C), 134.9 (2C), 134.1, 134.0, 129.95, 129.89, 128.2 (4C), 79.4, 69.9, 37.9, 37.2, 28.4 (3C), 27.8, 26.6, –5.7. HRMS $C_{23}H_{33}NO_5SSi$ [M+Na^+]; calculated: 486.1741, found: 486.1754.

(*R*)-*tert*-Butyl 4-azido-1-(methyldiphenylsilyl)butylcarbamate (28). Mesylate **26** (68 mg, 0.15 mmol) was dissolved in DMF (3 mL) and then NaN_3 (165 mg, 2.54 mmol) was added. The mixture was stirred at rt for 18 h, then poured into water (50 mL) and extracted with EtOAc (3 × 50 mL). The organic

portion was dried ($MgSO_4$), filtered and evaporated *in vacuo*. The pure product was obtained by column chromatography (increasing polarity from 5% to 10% EtOAc in pentane as eluant) which gave **28** (47 mg, 0.114 mmol, 77%) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.60–7.52 (m, 4H), 7.44–7.34 (m, 6H), 4.24 (d, *J* = 10.4 Hz, 1H), 3.81 (td, *J* = 11.2, 2.0 Hz, 1H), 3.35–3.20 (m, 2H), 1.76–1.56 (m, 3H), 1.50–1.35 (m, 1H), 1.40 (s, 9H), 0.61 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 156.4, 135.1 (2C), 135.0 (2C), 134.4, 134.1, 129.92, 129.86, 128.2 (4C), 79.3, 51.1, 38.3, 29.2, 28.5 (3C), 26.5, −5.6. HRMS $C_{22}H_{30}N_4O_2Si$ [M+Na^+]; calculated: 433.2030, found: 433.2034.



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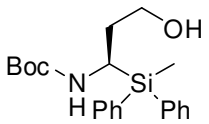
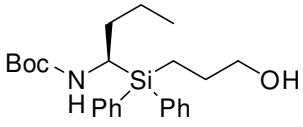


33

(*R*)-*N*-((*R*)-3-Hydroxy-1-(methyldiphenylsilyl)propyl)-2-methylpropane-2-sulfinamide (32). A solution of DDQ (996 mg, 4.29 mmol) in CH_2Cl_2 (10 mL) and water (1 mL, 55.0 mmol) was cooled to 0 °C. Compound **31** (532 mg, 1.07 mmol) was dissolved in CH_2Cl_2 (1 mL) and added to the stirred solution. After 1 h the reaction mixture was allowed to warm up to rt and the stirring continued for 3 h. Them the reaction was quenched with water (10 mL) and extracted with CH_2Cl_2 (3 × 30 mL). The combined organic portions were washed with sat $NaHCO_3$, dried ($MgSO_4$), filtered and evaporated *in vacuo*. The pure product was obtained by column chromatography (increasing polarity from 0:2:1 to 4:96:0 MeOH:EtOAc:pentane as eluant), which gave **32** (385 mg, 1.03 mmol, 96%) as a redish wax. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.57–7.54 (m, 4H), 7.43–7.34 (m, 6H), 4.03 (t, *J* = 6.6 Hz, 1H), 3.79 (q, *J* = 6.0 Hz, 2H), 3.63 (ddd, *J* = 11.6, 8.8, 3.2 Hz, 1H), 3.07 (d, *J* = 9.2 Hz, 1H), 2.02–1.94 (m, 1H), 1.67–1.59 (m, 1H), 1.03 (s, 9H), 0.61 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 135.2 (2C), 135.1

(2C), 134.4, 134.2, 129.91, 129.87, 128.2 (4C), 60.8, 56.4, 45.3, 34.7, 22.8 (3C), –5.5. HRMS $C_{20}H_{29}NO_2SSi$ [M+Na^+]; calculated: 398.1586, found: 398.1578.

(*R*)-*tert*-Butyl 3-hydroxy-1-(methyldiphenylsilyl)propylcarbamate (33). Sulfinamide **32** (51 mg, 0.14 mmol) was dissolved in anhydrous 0.5 M HCl in MeOH (4 mL) and the mixture was stirred at rt for 5 h. All volatiles were removed *in vacuo* to give the crude amine as its hydrochloride. This was dissolved in THF (2 mL) and then NEt_3 (0.1 mL, 73 mg, 0.721 mmol) and Boc_2O (104 mg, 0.48 mmol) were added. The mixture was stirred at rt for 22 h, and then 2 M NaOH (10 mL) was added. The mixture was stirred vigorously for a further 1 h. The two phases were separated and the aqueous portion was extracted with CH_2Cl_2 (3 × 20 mL). The combined organic portions were dried ($MgSO_4$), filtered and evaporated *in vacuo*. The pure product was obtained by column chromatography (increasing polarity from 5% to 35% EtOAc in pentane as eluant) which gave **33** (21 mg, 0.06 mmol, 40%) as a pale yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.57–7.51 (m, 4H), 7.44–7.37 (m, 6H), 4.31 (d, *J* = 10.4 Hz, 1H), 3.98 (ddd, *J* = 13.2, 10.0, 2.8 Hz, 1H), 3.85 (dd, *J* = 10.0, 4.4 Hz, 1H), 3.62–3.51 (m, 2H), 1.85 (dddd, *J* = 13.6, 11.2, 5.6, 2.8 Hz, 1H), 1.44–1.35 (m, 1H), 1.41 (s, 9H), 0.61 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 158.5, 135.1 (4C), 134.0, 133.8, 130.1, 130.0, 128.34 (2C), 128.31 (2C), 80.3, 58.2, 34.4, 34.0, 28.4 (3C), –5.3. HRMS $C_{21}H_{29}NO_3Si$ [M+Na^+]; calculated: 394.1814, found: 394.1819.



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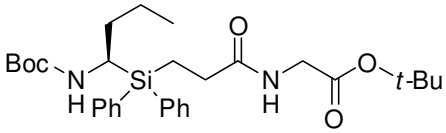

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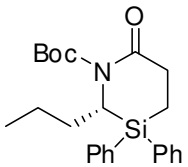


43

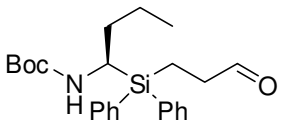
(*R*)-*tert*-Butyl 1-[(3-hydroxypropyl)diphenylsilyl]butylcarbamate (40). Sulfinamide **39** (424 mg, 0.85 mmol) was dissolved in methanol containing anhyd HCl (50 mL, 0.4 M). The mixture was stirred at rt for 18 h, and all volatiles were removed *in vacuo* which gave the crude amine as its corresponding

HCl salt. Toluene (2 mL) was added and evaporated repeatedly until dryness. The residue was dissolved in CH_2Cl_2 (60 mL), and triethyl amine (0.18 mL, 1.3 mmol, 1.5 equiv) and di-*tert*-butyldicarbonate (0.55 g, 2.5 mmol, 3 equiv) was added and the reaction stirred at rt for 18 h. Aqueous NaOH (50 mL, 1 M) was added and the mixture stirred for 10 min and then extracted with CH_2Cl_2 (3 × 50 mL). The combined organic extracts were washed with brine (50 mL), dried ($MgSO_4$), and concentrated *in vacuo*. The pure product was obtained by column chromatography (25% ethyl acetate in pentane as eluant) which gave **40** (229 mg, 0.55 mmol, 65%) as a colourless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.58–7.55 (m, 2H), 7.52–7.50 (m, 2H), 7.45–7.35 (m, 6H), 4.14 (br d, J = 10.8 Hz, 1H), 3.90 (td, *J* = 10.8, 2.8 Hz, 1H), 3.63–3.51 (m, 2H), 1.64–1.52 (m, 5H), 1.49–1.44 (m, 2H), 1.41 (s, 9H), 1.36–1.06 (m, 2H), 0.87 (t, *J* = 6.8 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 156.5, 135.7 (2C), 135.5 (2C), 133.5, 133.1, 129.9, 129.8, 128.2 (2C), 128.1 (2C), 79.2, 65.4, 37.8, 34.4, 28.5 (3C), 26.9, 20.4, 13.9, 7.5. HRMS (ES) $C_{24}H_{35}NO_3Si$ [M+Na^+]; calcd: 436.2284, found: 436.2277.

(*R*)-*tert*-Butyl 2,2-dimethyl-4,10-dioxo-7,7-diphenyl-6-propyl-3-oxa-5,11-diaza-7-silatridecan-13-oate (42). Alcohol **40** (45 mg, 0.11 mmol) was dissolved in a mixture of MeCN, ethyl acetate and H_2O (1 mL, 2:2:3), and NaIO4 (95 mg, 0.44 mmol, 4.1 equiv) and $RuCl_3$ (1 mg) were added. The reaction was stirred at rt for 2 h, and then H_2O (5 mL) was added. The mixture was extracted with ethyl acetate (2 × 10 mL) and the combined organic extracts were dried ($MgSO_4$), filtered and concentrated *in vacuo* to give the crude carboxylic acid. The residue was dissolved in CH_2Cl_2 (3 mL) and NMM (60 µL, 0.54 mmol, 5 equiv), *tert*-butyl glycinate hydrochloride (18 mg, 0.11 mmol, 1 equiv), HOBt (33 mg, 0.22 mmol, 2 equiv), and EDC•HCl (41 mg, 0.22 mmol, 2 equiv) were added and the reaction stirred at rt for 20 h. H_2O (15 mL) was added and the mixture extracted with CH_2Cl_2 (2 × 10 mL). The combined organic extracts were dried ($MgSO_4$), filtered and concentrated *in vacuo*. The pure product was obtained by column chromatography (increasing polarity from 10% to 25% ethyl acetate in pentane as eluant)

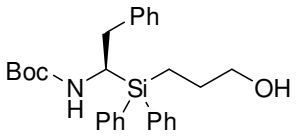
which gave **42** (37 mg, 0.069 mmol, 63%) as a colourless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.57–7.54 (m, 2H), 7.51–7.49 (m, 2H), 7.46–7.35 (m 6H), 5.98 (br s, 1H), 4.22 (br d, *J* = 10.4 Hz, 1H), 3.93–3.90 (m, 1H), 3.87–3.84 (m, 2H), 2.34–2.26 (m, 1H), 2.19–2.11 (m, 1H), 1.61–1.47 (m, 2H), 1.46 (s, 9H), 1.40 (s, 9H), 1.36–1.19 (m, 4H), 0.86 (t, *J* = 6.8 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 174.0, 169.2, 156.5, 135.6 (2C), 135.5 (2C), 132.8, 132.5, 130.0 (2C), 128.3 (2C), 128.2 (2C), 82.3, 79.2, 42.2, 37.6, 34.2, 30.4, 28.5 (3C), 28.2 (3C), 20.4, 13.9, 7.4. HRMS (ES) $C_{30}H_{44}N_2O_5Si$ [M+Na^+]; calcd: 563.2917, found: 563.2916.

(*R*)-*tert*-Butyl 6-oxo-3,3-diphenyl-2-propyl-1,3-azasilinane-1-carboxylate (43). Alcohol **40** (45 mg, 0.11 mmol) was dissolved in acetone and cooled to 0 ° C. Aqueous $H_2Cr_2O_7$ (0.32 mL, 0.5 M, 0.16 mmol) was added and the reaction stirred at 0 °C for 2 h. Excess oxidant was quenched by addition of 2-propanol and the mixture filtered through celite. Solvents were removed *in vacuo* and the residue dissolved in CH_2Cl_2 (20 mL) and washed successively with H_2O (10 mL) and brine (10 mL), dried ($MgSO_4$), filtered and concentrated *in vacuo*. The crude product was reacted with NMM (60 µL, 0.54 mmol), *tert*-butyl glycinate hydrochloride (18 mg, 0.11 mmol), HOBt (33 mg, 0.22 mmol), and EDC•HCl (41 mg, 0.22 mmol) and the reaction stirred at rt for 20 h. The pure product was obtained by column chromatography (increasing polarity from 10% to 25% ethyl acetate in pentane) which gave amide **42** (34 mg, 0.062 mmol, 58%) and **43** (7.3 mg, 0.017 mmol, 17%). ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.56–7.52 (m, 4H), 7.48–7.34 (m, 6H), 4.49 (dd, *J* = 10.0, 5.6 Hz, 1H), 2.98 (ddd, *J* = 15.6, 6.0, 3.6 Hz, 1H), 2.84 (ddd, *J* = 15.6, 14.4, 5.6 Hz, 1H), 1.69 (ddd, *J* = 15.6, 5.6, 3.6 Hz, 1H), 1.57–1.51 (m, 1H), 1.48 (s, 9H), 1.45–1.32 (m, 3H), 1.26–1.15 (m, 1H), 0.76 (t, *J* = 6.0 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 174.9, 154.1, 135.3 (2C), 134.7 (2C), 134.0, 132.5, 130.5, 130.2, 128.4 (2C), 128.3 (2C), 82.8, 44.1, 35.6, 34.5, 28.1 (3C), 20.9, 14.0, 5.6. HRMS (ES) $C_{24}H_{31}NO_3Si$ [M+Na^+]; calcd: 432.1971, found: 432.1966.



44

(*R*)-*tert*-Butyl 1-[(3-oxo-propyl)-di-phenyl-silyl]-butyl-carbamate (44). Alcohol **40** (43 mg, 0.10 mmol) was dissolved in CH_2Cl_2 (0.5 mL) and TEMPO (approx. 1 mg, 3 μmol), H_2O (0.3 mL), sat aqueous $NaHCO_3$ (0.9 mL), potassium bromide (approx. 1 mg, 0.01 mmol), and TBAB (approx. 2 mg, 0.5 μmol) were added. The mixture was cooled to 0 °C and aqueous NaOCl (0.16 mL, 15 vol%, 0.31 mmol) was added dropwise. The reaction was stirred at rt for 1.5 h and after addition of a few drops of methanol it was stirred for 5 min. The solution was acidified by dropwise addition of concd HCl and extracted with CH_2Cl_2 (3 × 20 mL), and the combined organic extracts were dried over anhyd $MgSO_4$ and concentrated *in vacuo*.[13] The title compound was obtained with a minor impurity of the starting material. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 9.70 (s, 1H), 7.58–7.38 (m, 10H), 4.15 (br d, J = 11.2 Hz, 1H), 3.93 (td, J = 11.2, 2.8 Hz, 1H), 2.51 (d_{AB}dd, J_{AB} = 17.2 Hz, J = 11.6, 4.8 Hz, 1H), 2.39 (d_{AB}dd, J_{AB} = 17.2 Hz, J = 11.2, 5.2 Hz, 1H), 1.50–1.45 (m, 3H), 1.41 (s, 9H), 1.39–1.12 (m, 3H), 0.88 (t, J = 6.8 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 202.2, 135.5 (2C), 135.3 (2C), 135.1, 134.5, 130.0, 129.7, 128.2, 128.0, 79.1, 38.0, 37.4, 34.2, 28.4 (3C), 20.2, 13.7, 3.2.



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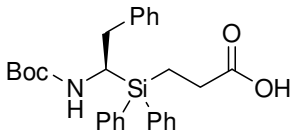

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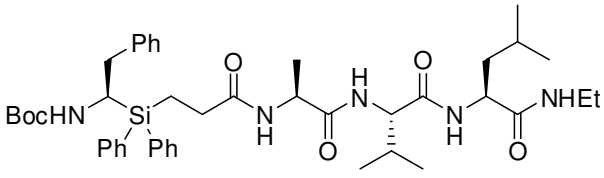

62

(*R*)-*tert*-Butyl 1-((3-hydroxypropyl)diphenylsilyl)-2-phenylethylcarbamate (59). Sulfinamide **58** (110 mg, 0.20 mmol) was dissolved in anhydrous 0.5 M HCl in MeOH (5 mL) and the mixture was stirred at rt for 18 h. All volatiles were removed *in vacuo* to give the crude amine as its hydrochloride

salt. This was dissolved in CH_2Cl_2 (4 mL) and then NEt_3 (137 μL, 0.98 mmol) and Boc_2O (86 mg, 0.40 mmol) were added. The mixture was stirred at rt for 18 h, and the 2 M NaOH (5 mL) was added. The mixture was stirred vigorously for a further 2 h. The two phases were separated and the aqueous portion was extracted with CH_2Cl_2 (3 × 20 mL). The combined organic portions were dried ($MgSO_4$), filtered and evaporated *in vacuo*. The pure product was obtained by column chromatography (increasing polarity from 10% to 40% EtOAc in pentane as eluant), which gave **59** (68 mg, 0.15 mmol, 74%) as a colorless gum. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) major rotamer 7.62–7.57 (m, 4H), 7.39 (m, 7H), 7.25–7.22 (m, 2H), 7.17–7.15 (m, 3H), 4.25–4.20 (m, 1H), 3.59–3.48 (m, 2H), 3.01–2.97 (m, 1H), 2.53 (dd, *J* = 14.0, 10.0 Hz, 1H), 1.85 (br s, 1H), 1.64–1.51 (m, 2H), 1.28 (s, 9H), 1.20–1.05 (m, 2H). Minor rotamer *inter alia* 5.73 (d, *J* = 10.4 Hz, 1H), 4.20 (d, *J* = 7.2 Hz, 1H), 1.10 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 156.0, 139.5, 135.7 (2C), 135.5 (2C), 133.3, 132.9, 130.0 (2C), 129.1 (2C), 128.3 (4C), 128.2 (2C), 126.2, 79.3, 65.3, 39.5, 38.3, 28.4 (3C), 26.9, 7.6. HRMS $C_{28}H_{35}NO_3Si$ [M+Na^+]; calculated: 484.2278, found: 484.2278.

(*R*)-3-((1-(*tert*-Butoxycarbonylamino)-2-phenylethyl)diphenylsilyl)propanoic acid (60). Alcohol **59** (61 mg, 0.13 mmol) was dissolved in a mixture of H_2O, MeCN and CCl_4 (3.5 mL, 3:2:2). $NaIO_4$ (113 mg, 0.53 mmol) and $RuCl_3$ (2 mg, 0.01 mmol) were added and the reaction was stirred under air at rt for 2 h. The reaction mixture was diluted with H_2O (5 mL) and extracted with EtOAc (2 × 10 mL), and then the combined organic portions were dried ($MgSO_4$), filtered and concentrated *in vacuo*. The obtained crude mixture of title compound **60** was used in the following step without further purification or characterization.

***tert*-Butyl (5*S*,8*S*,11*S*,17*R*)-5-isobutyl-8-isopropyl-11-methyl-4,7,10,13-tetraoxo-16,16,18-triphenyl-3,6,9,12-tetraaza-16-silaoctadecan-17-ylcarbamate (62).** Trifluoroacetic acid (4 mL) was added to a solution of *N*-Boc-L-alanyl-L-isoleucyl-L-leucinamide **61** (88 mg, 0.20 mmol) in CH_2Cl_2 (4 mL).

The mixture was stirred at rt for 1 h and then the solvents were evaporated *in vacuo*. The residue was redissolved in CH_2Cl_2 (4 mL) and the solvent was evaporated *in vacuo*, giving the crude trifluoroacetate ammonium salt as a colorless gum. The crude material was redissolved in CH_2Cl_2 (3 mL) and *N*-methyl morpholine (73 µL, 0.66 mmol), carboxylic acid **60** (0.13 mmol), HOBt (41 mg, 0.26 mmol) and EDC (51 mg, 0.26 mmol) were added. The mixture was stirred at rt for 3 days and then poured into water (10 mL). The aqueous phase was extracted with CH_2Cl_2 (3 × 10 mL) and the combined organic portions were dried ($MgSO_4$), filtered and evaporated *in vacuo*. The pure product was obtained by column chromatography (increasing polarity from 40% to 80% EtOAc in CH_2Cl_2 as eluant), which gave **62** (63 mg, 0.078 mmol, 59%) as a colorless solid. ^{1}H NMR (400 MHz, CD_3OD) δ (ppm) major rotamer 7.58–7.65 (m, 4H), 7.37–7.47 (m, 6H), 7.10–7.22 (m, 5H), 6.23 (d, *J* = 10.4 Hz, 1H), 4.35 (dd, *J* = 9.6, 5.2 Hz, 1H), 4.26 (q, *J* = 6.8 Hz, 1H), 4.15 (d, *J* = 7.2 Hz, 1H), 4.01–4.08 (m, 1H), 3.19 (q, *J* = 7.2 Hz, 2H), 2.87 (dd, *J* = 14.4, 3.6 Hz, 1H), 2.59 (dd, *J* = 14.0, 12.2 Hz, 1H), 2.27–2.31 (m, 2H), 1.81–1.88 (m, 1H), 1.46–1.66 (m, 6H), 1.29 (d, *J* = 7.2 Hz, 3H), 1.25 (s, 9H), 1.14–1.23 (m, 1H), 1.11 (t, *J* = 6.8 Hz, 3H), 0.88–0.93 (m, 12H). Minor rotamer *inter alia* 5.73 (d, *J* = 10.4 Hz, 1H), 4.20 (d, *J* = 7.2 Hz, 1H), 1.10 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 173.8, 173.1, 171.9, 171.5, 155.8, 139.4, 135.54 (2C), 135.48 (2C), 132.8, 132.7, 130.0 (2C), 129.1 (2C), 128.3 (4C), 128.2 (2C), 126.2, 79.0, 57.9, 51.7, 48.7, 41.4, 39.6, 38.2, 34.3, 29.9, 28.4 (3C), 28.0, 25.4, 25.1, 22.9, 22.5, 20.1, 15.4, 14.7, 11.7, 7.1. HRMS $C_{45}H_{65}N_5O_6Si$ [M+Na^+]; calculated: 822.4596, found: 822.4604.

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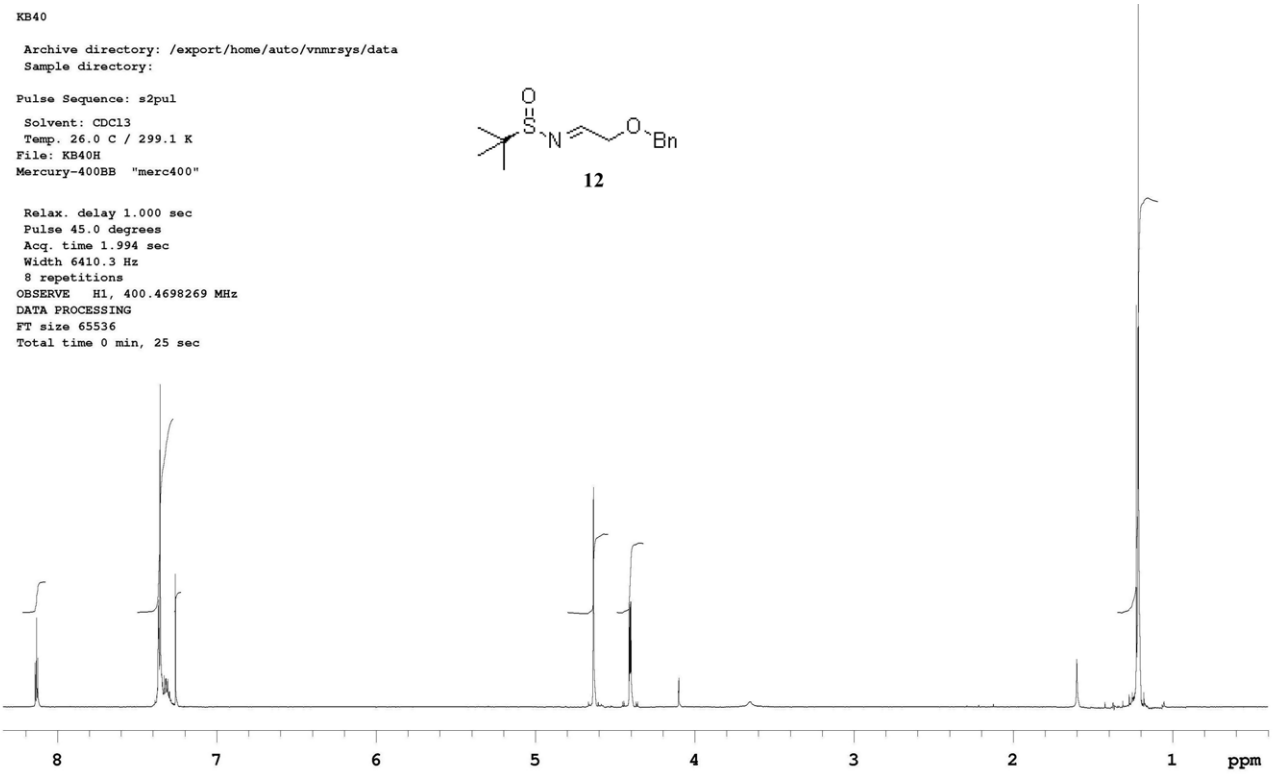
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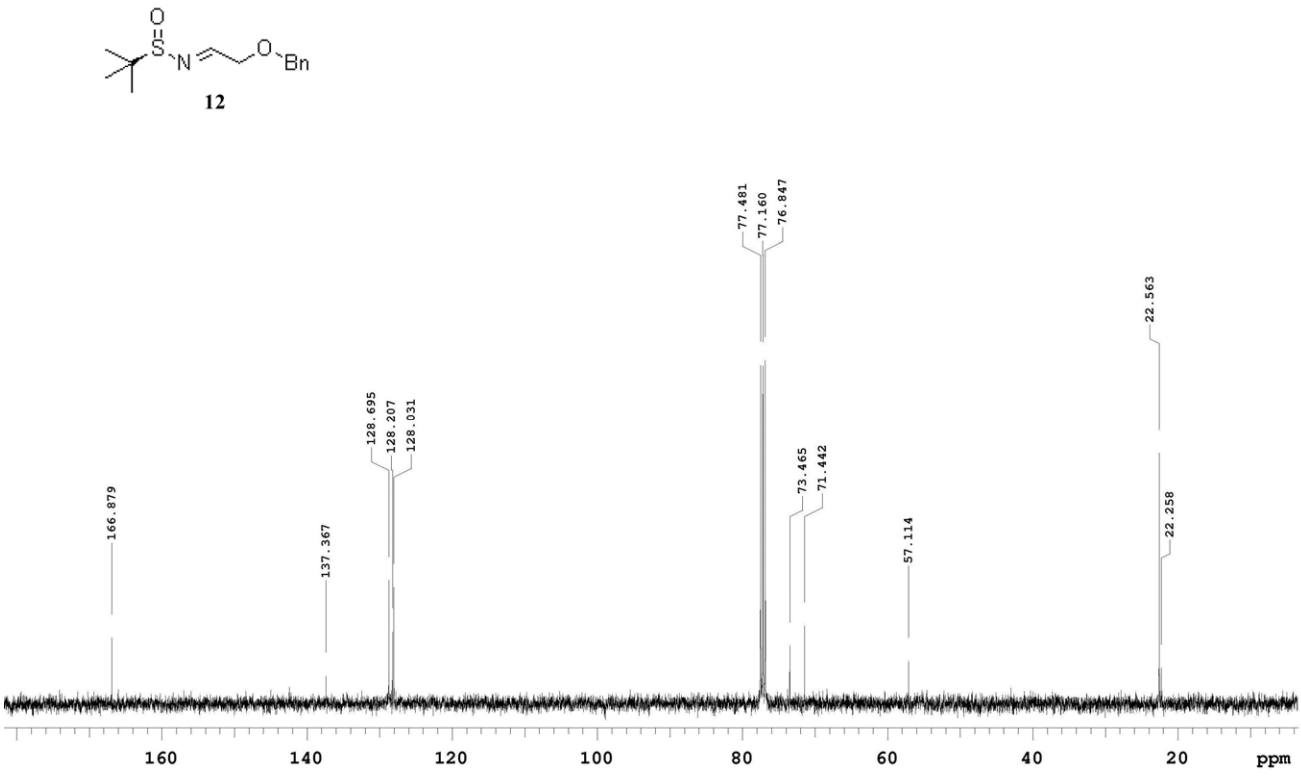
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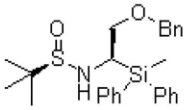

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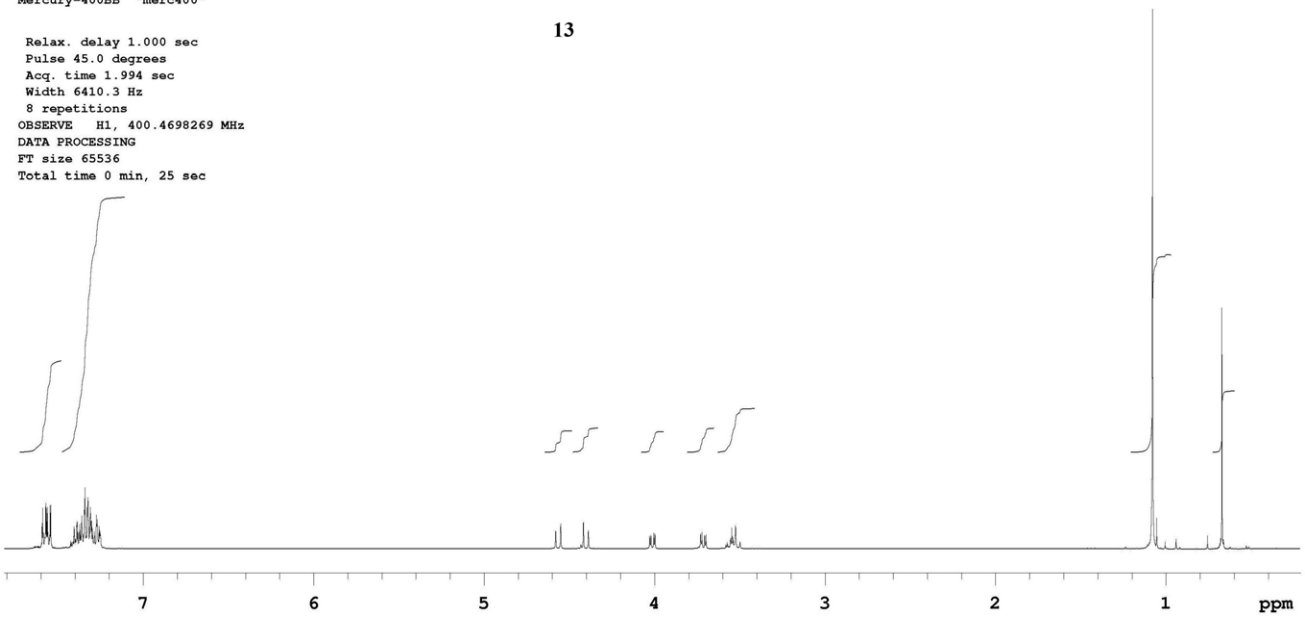

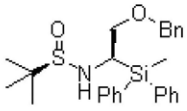

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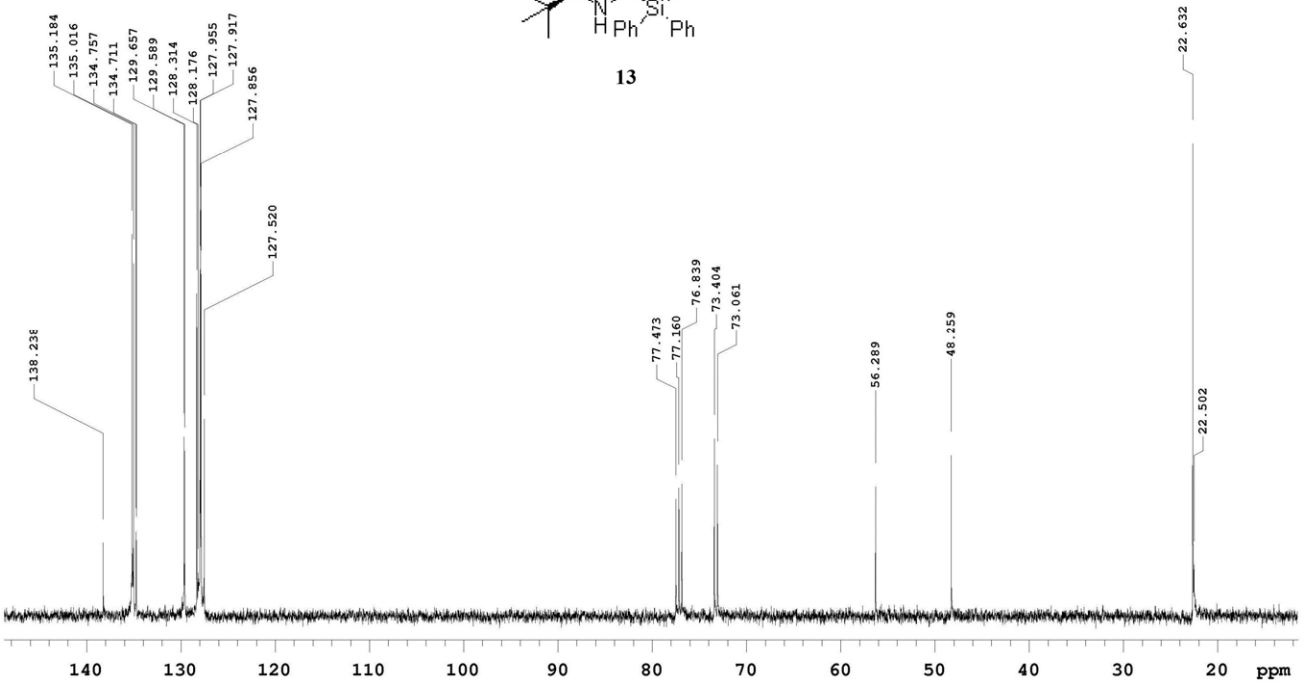

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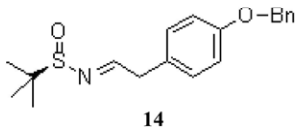
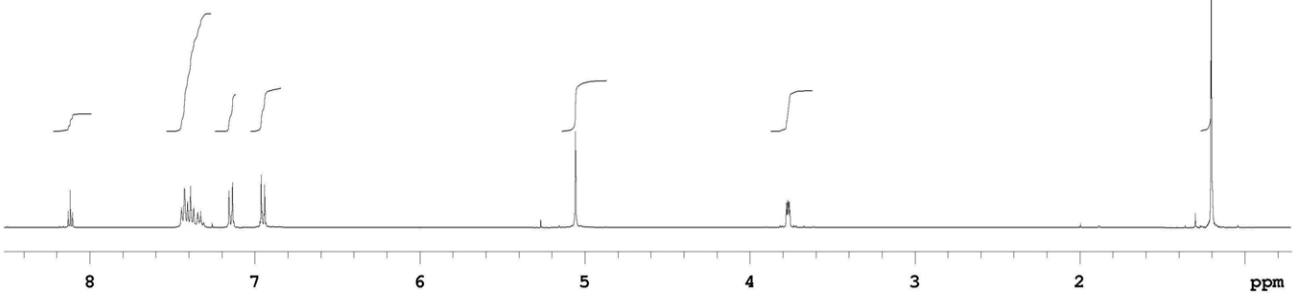
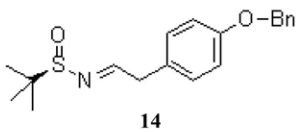
FT size 32768
Total time 0 min, 25 sec

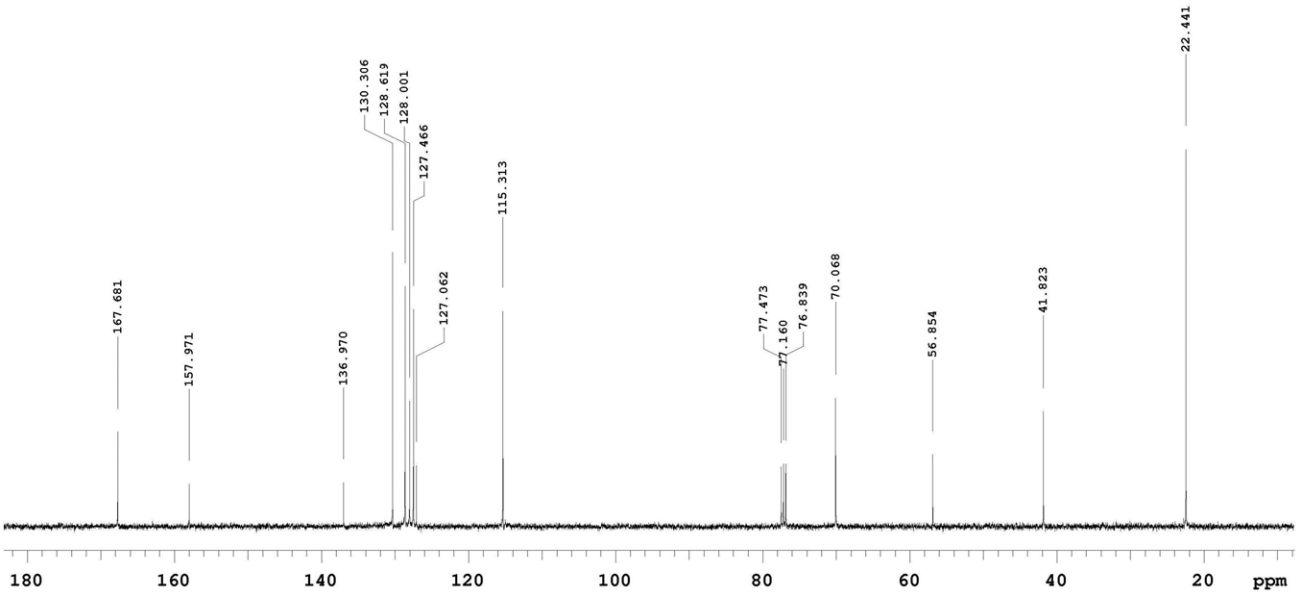


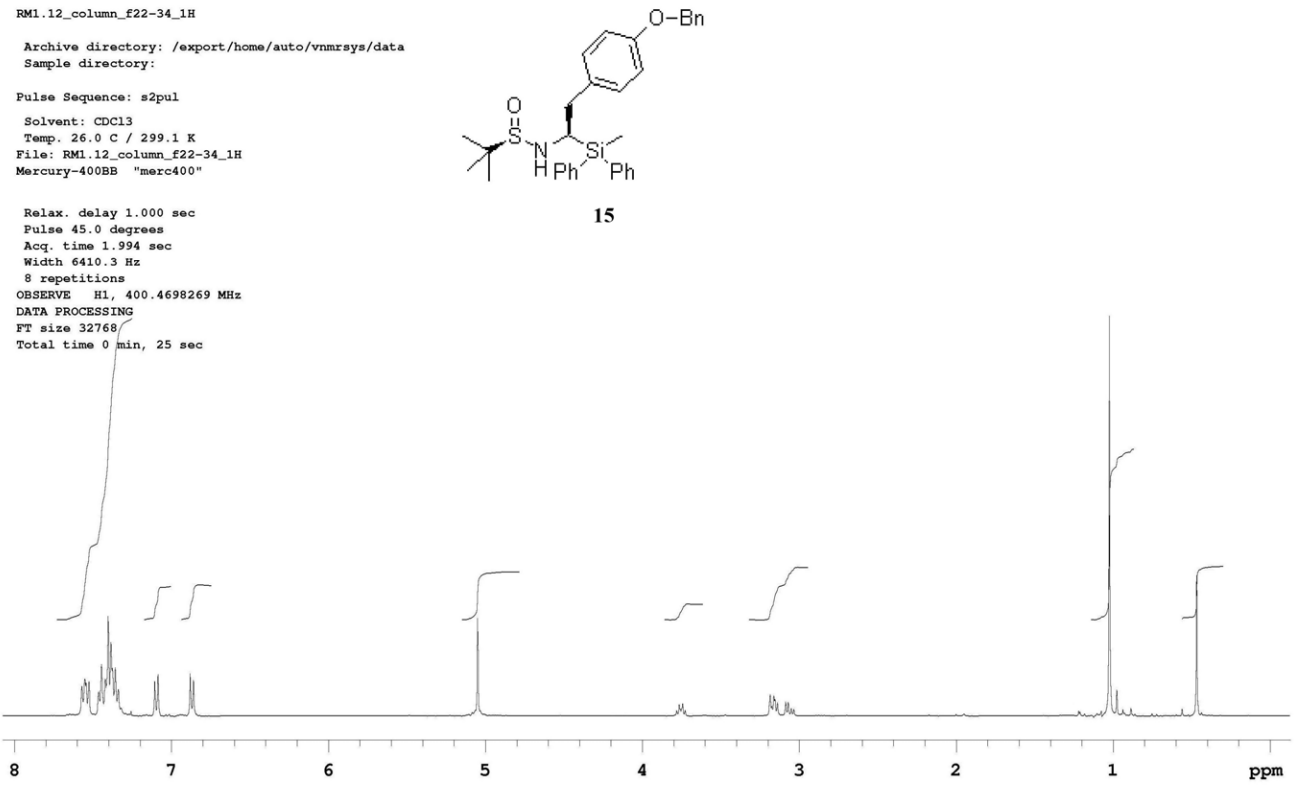
14



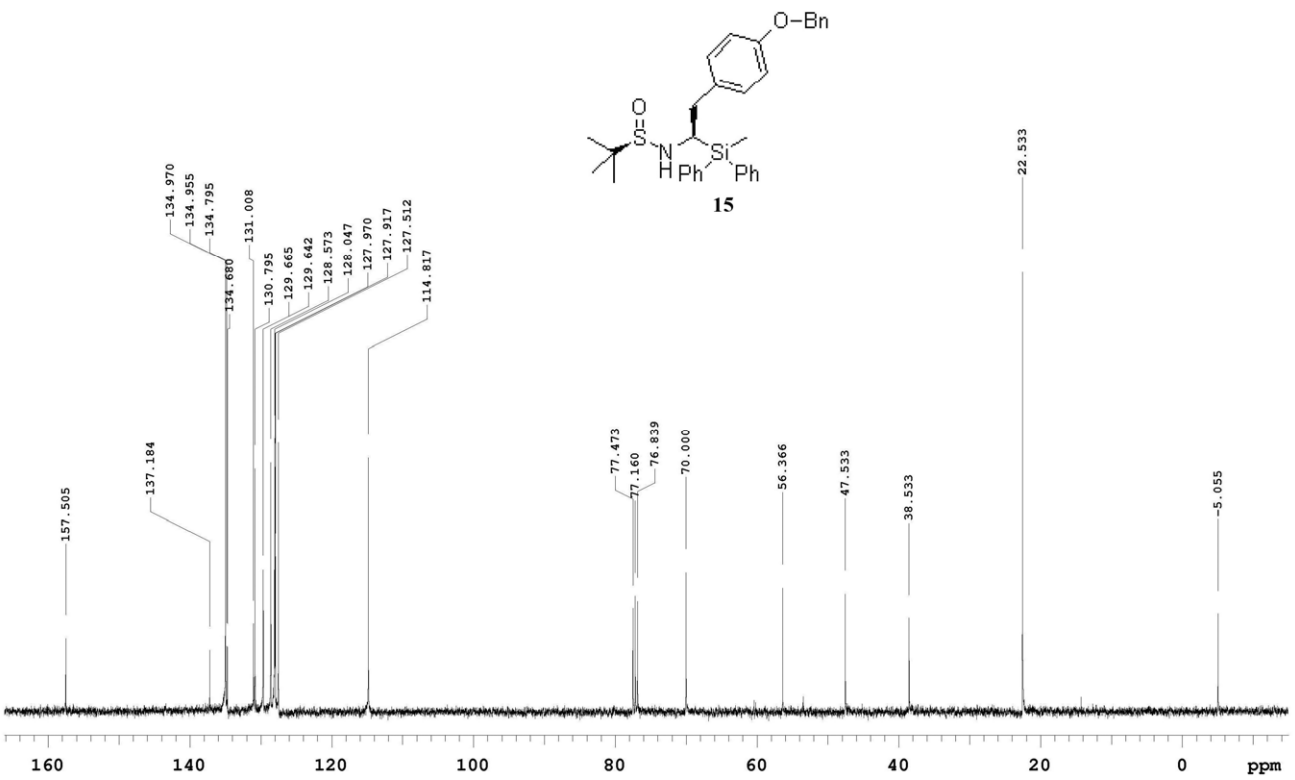


14




RM1.12_column_f22-34_1H
Archive directory: /export/home/auto/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 26.0 C / 299.1 K
File: RM1.12_column_f22-34_1H
Mercury-400BB "merc400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.994 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 400.4698269 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 25 sec
O-Bn
O
S
N
H
Ph
Si
Ph
8
7
6
5
4
3
2
1
ppm

15


O-Bn
O
S
N
H
Ph
Si
Ph
134.970
134.955
134.795
134.680
131.008
130.795
129.665
129.642
128.573
128.047
127.970
127.917
127.512
114.817
157.505
137.184
77.473
77.160
76.839
70.000
56.366
47.533
38.533
22.533
-5.055
160
140
120
100
80
60
40
20
0
ppm

15

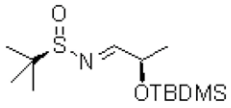
RM1.23_column_1H

Archive directory: /export/home/auto/vnmrsys/data
Sample directory:

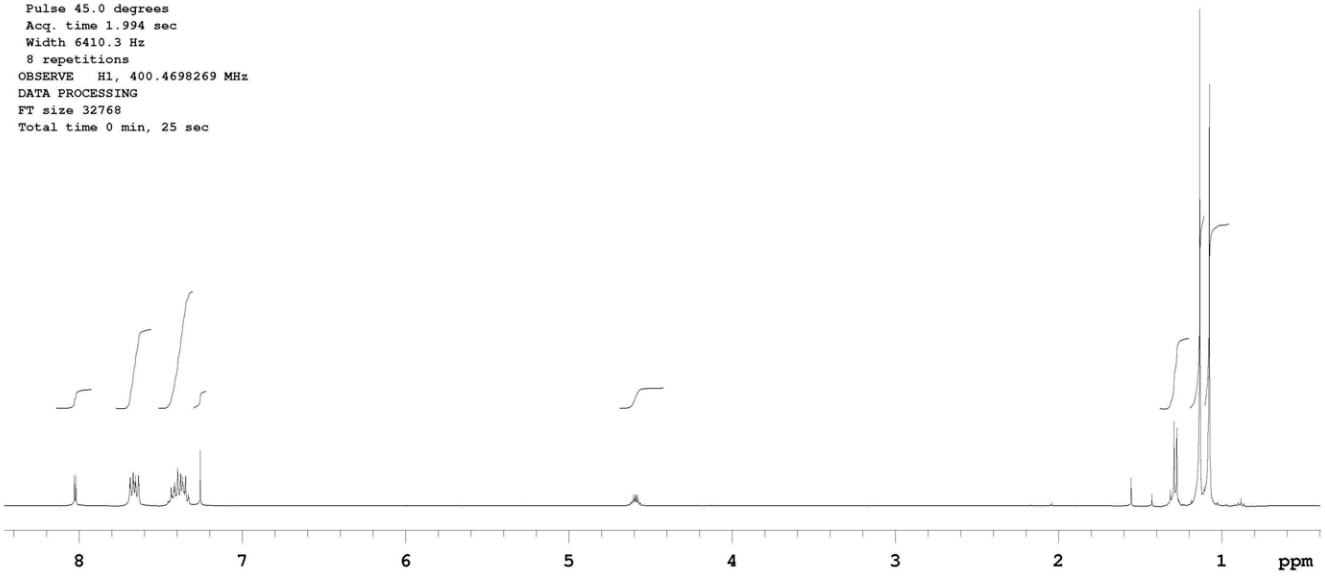
Pulse Sequence: s2pul

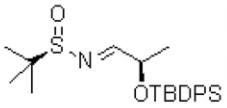
Solvent: CDCl3
Temp. 26.0 C / 299.1 K
File: RM1.23_column_1H
Mercury-400BB "merc400"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.994 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 400.4698269 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 25 sec

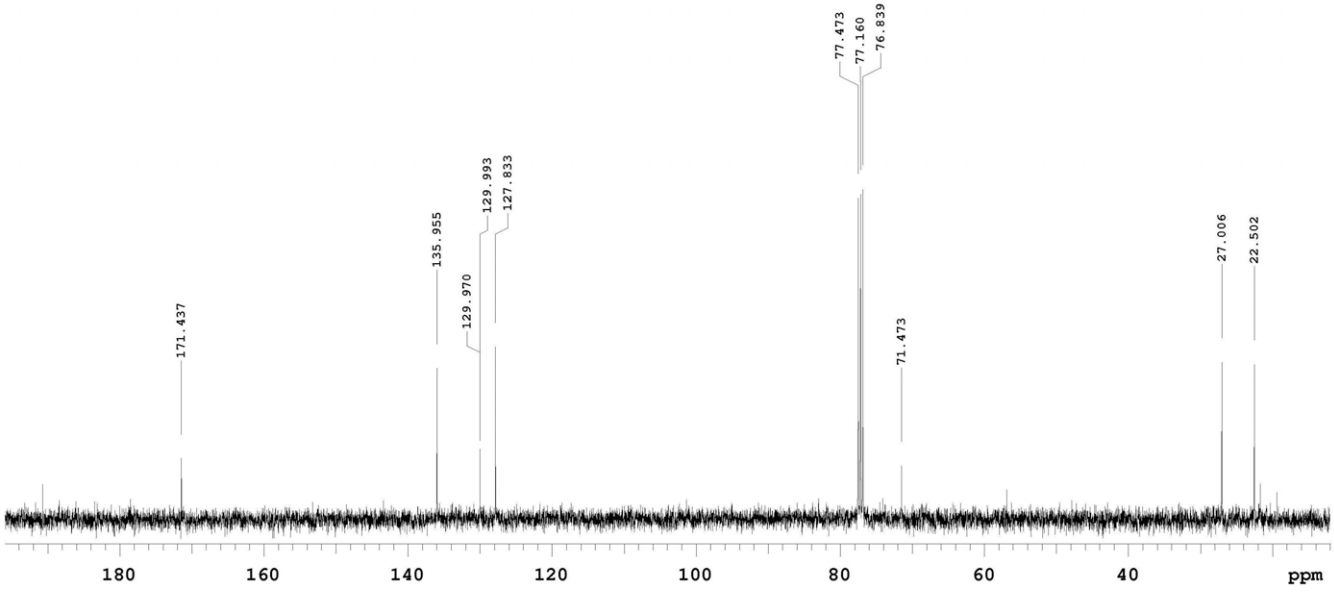


16





16



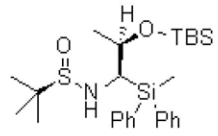
RM1.24_column(2)_f20-22_1H

Archive directory: /export/home/auto/vnmrsys/data
Sample directory:

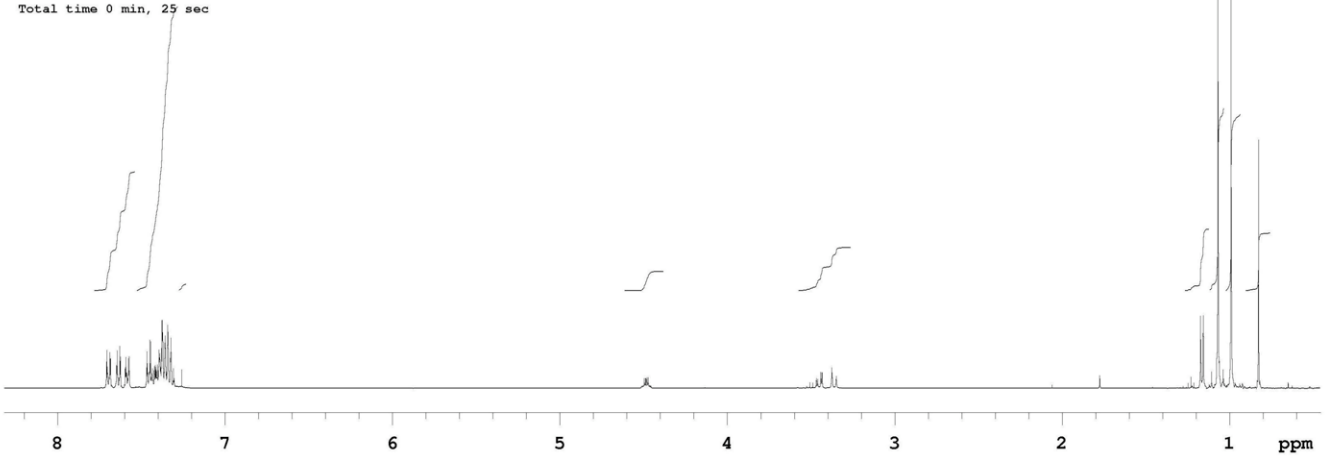
Pulse Sequence: s2pul

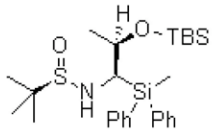
Solvent: CDCl3
Temp. 26.0 C / 299.1 K
File: RM1.24_column2_f20-22_1H
Mercury-400BB "merc400"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.994 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 400.4698269 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 25 sec

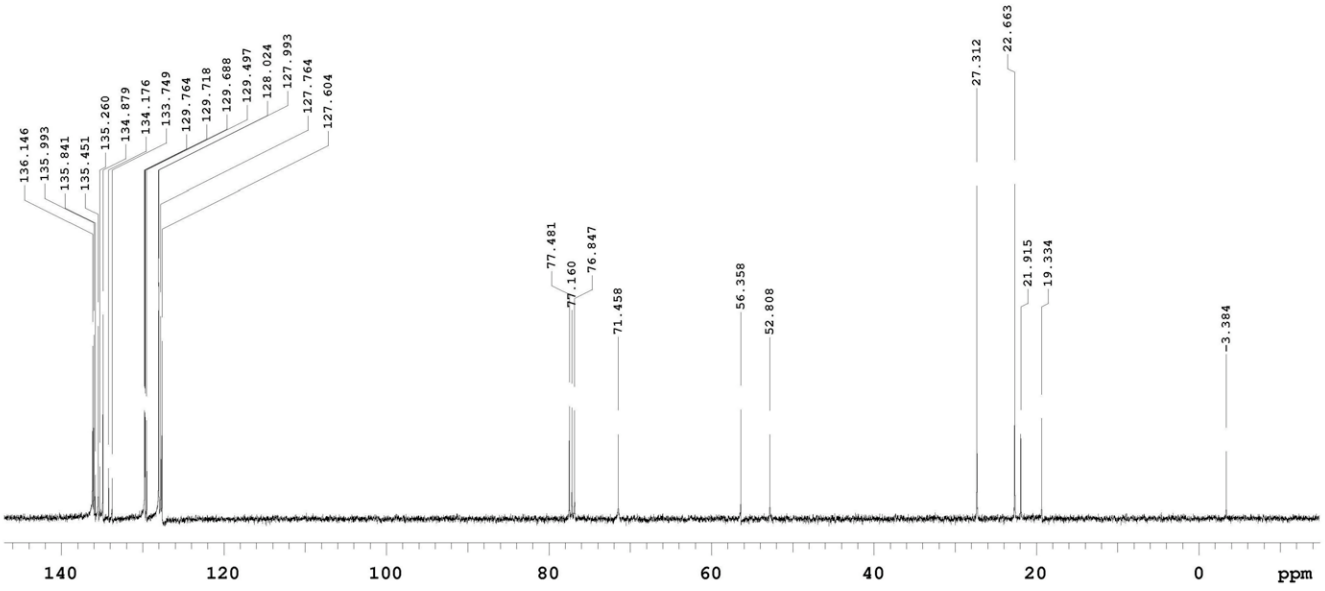


17





17

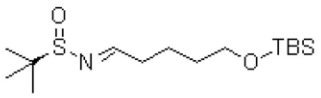


kb1572a6_9

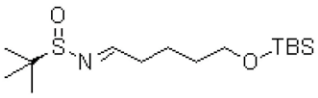
Archive directory: /export/home/auto/vnmrsys/data
Sample directory:

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 26.0 C / 299.1 K
File: kb1572a6_9
Mercury-400BB "merc400"

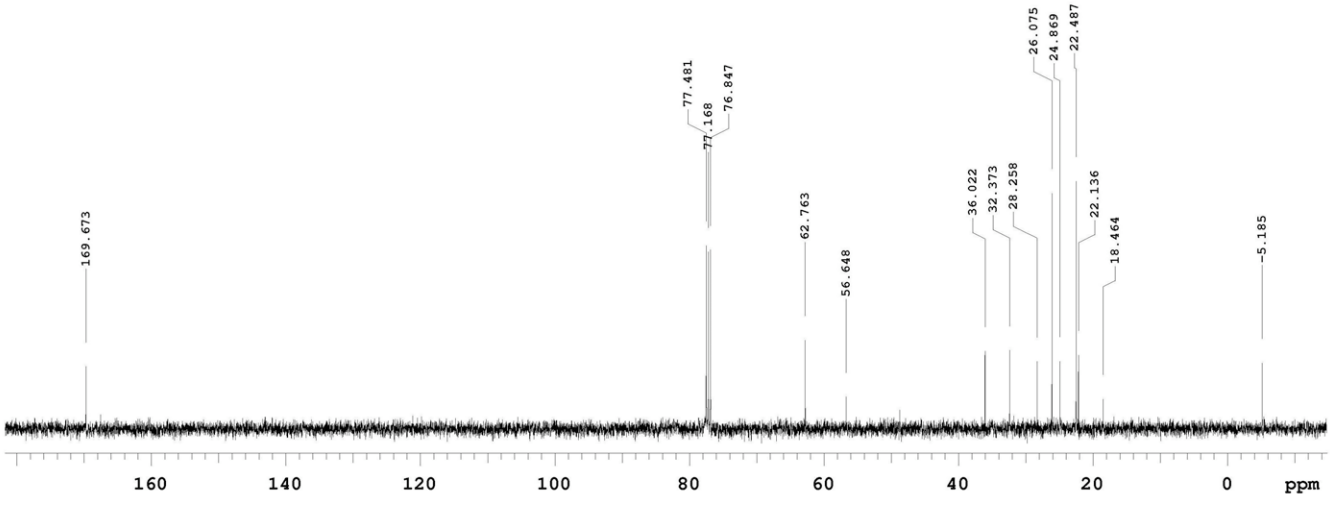
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.994 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 400.4698269 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 25 sec



77



77



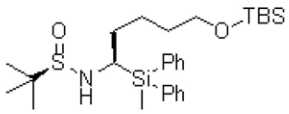
kbl573a13_22

Archive directory: /export/home/auto/vnmrsys/data
Sample directory:

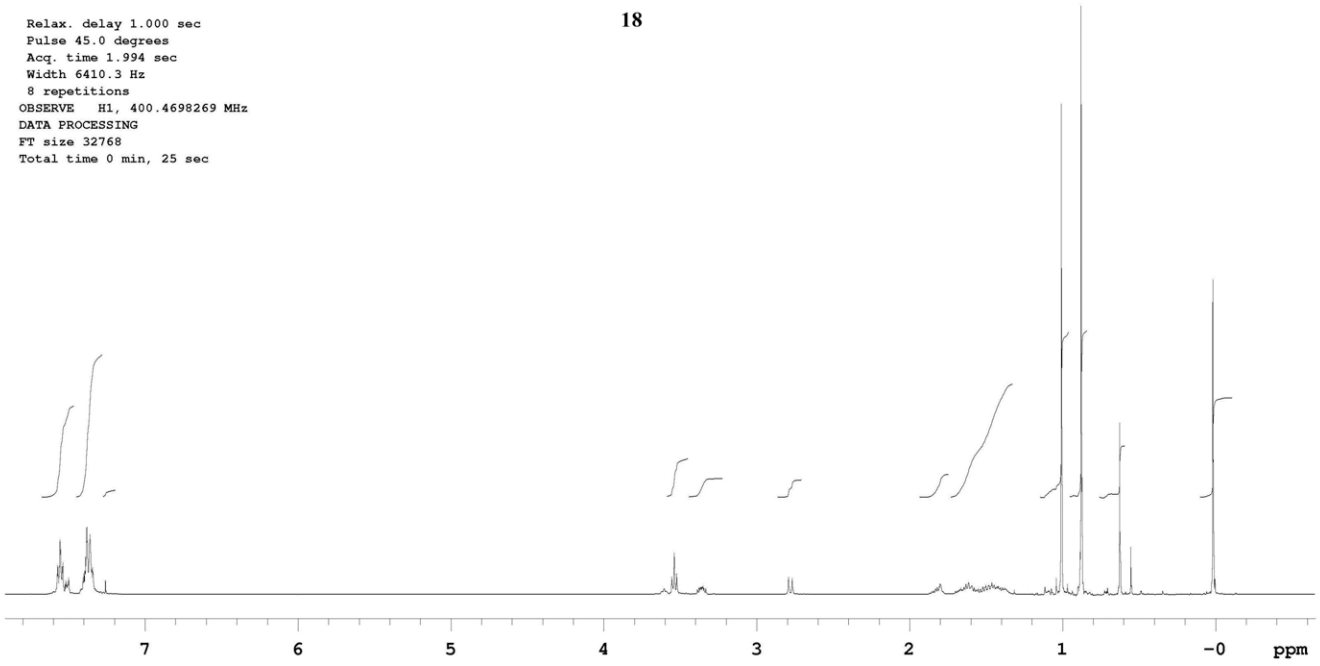
Pulse Sequence: s2pul

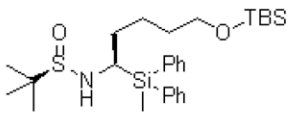
Solvent: CDCl3
Temp. 26.0 C / 299.1 K
File: kbl573a13_22
Mercury-400BB "merc400"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.994 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 400.4698269 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 25 sec

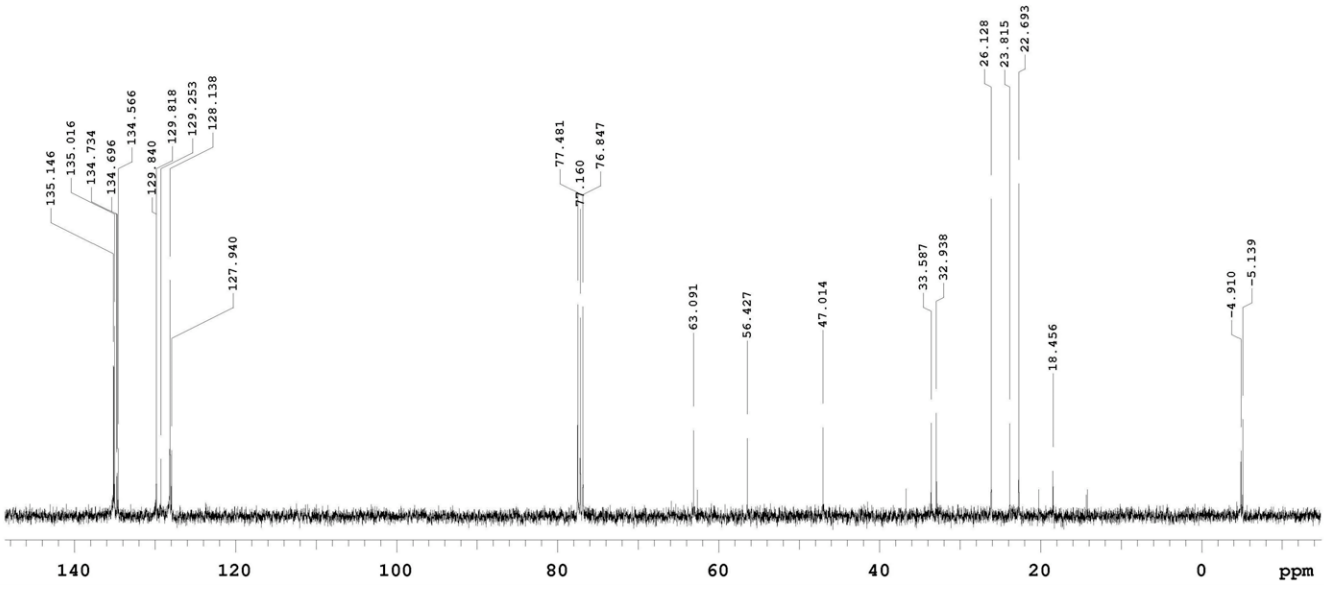


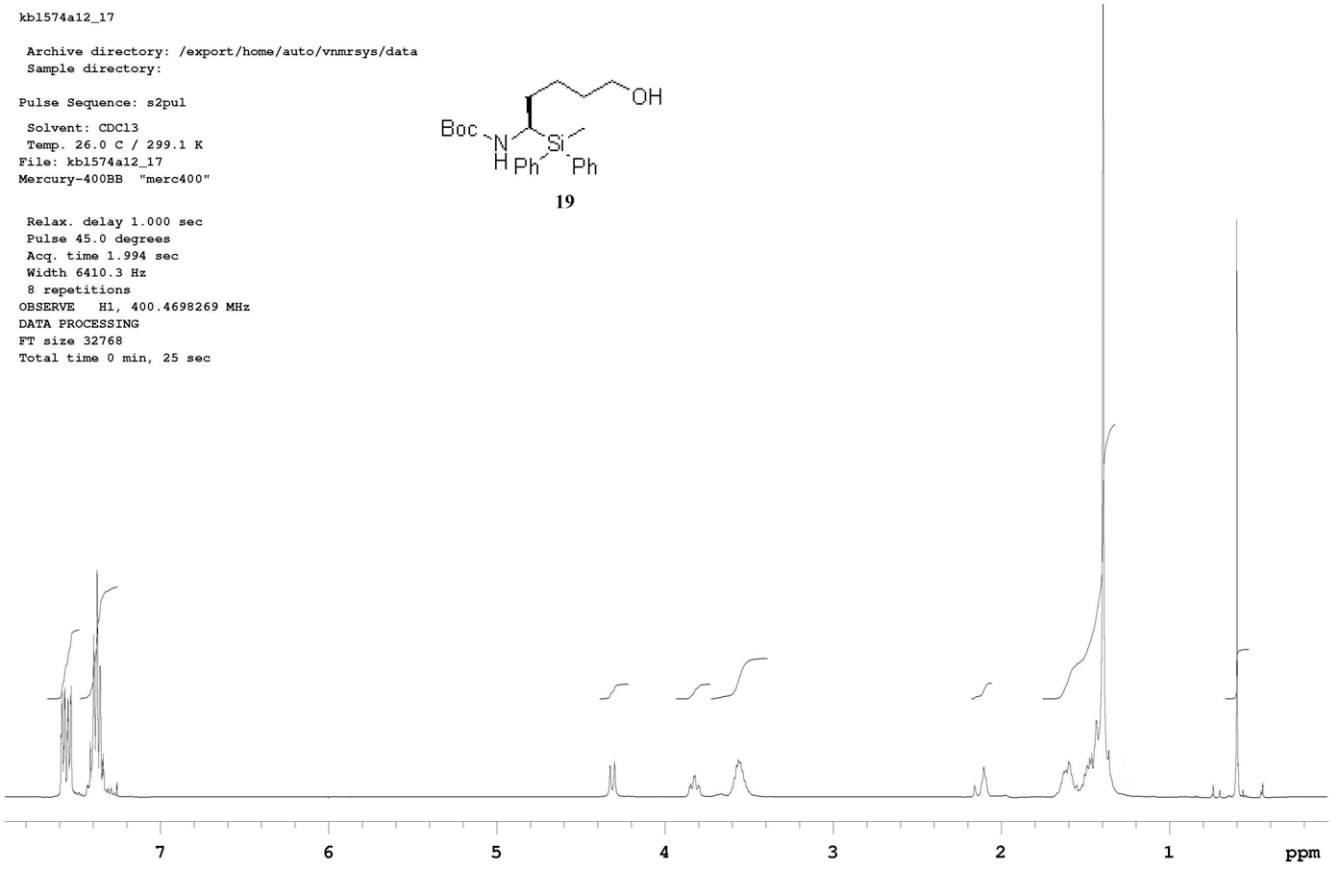
18

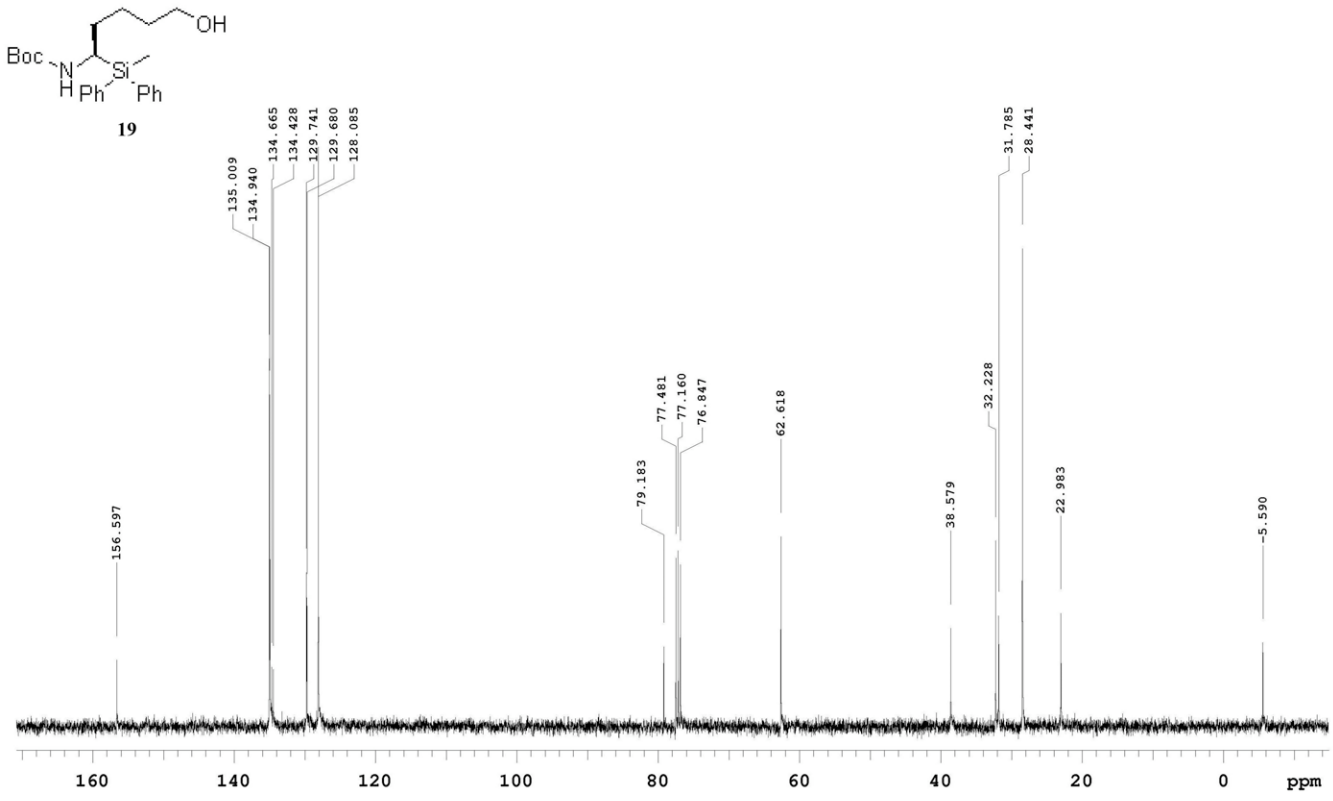


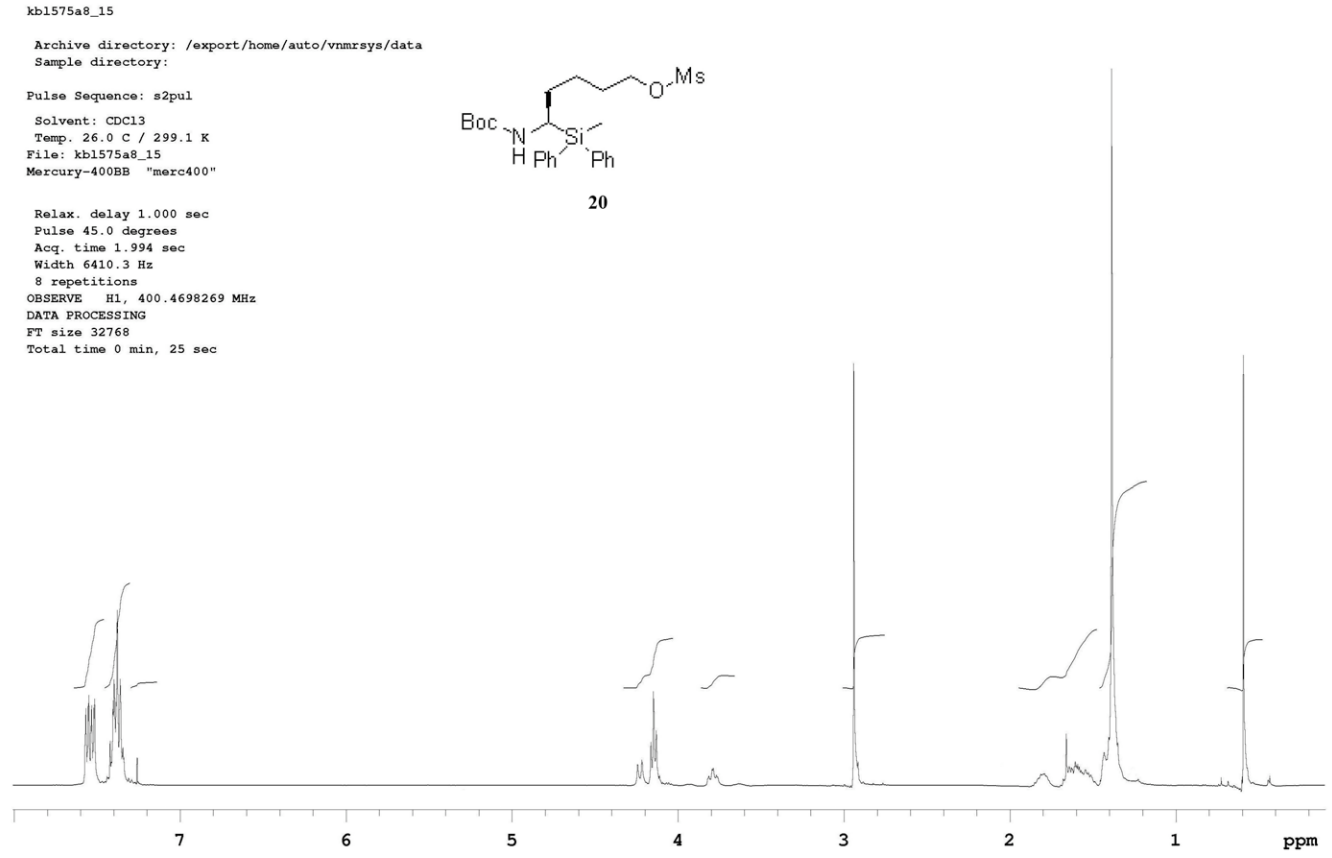


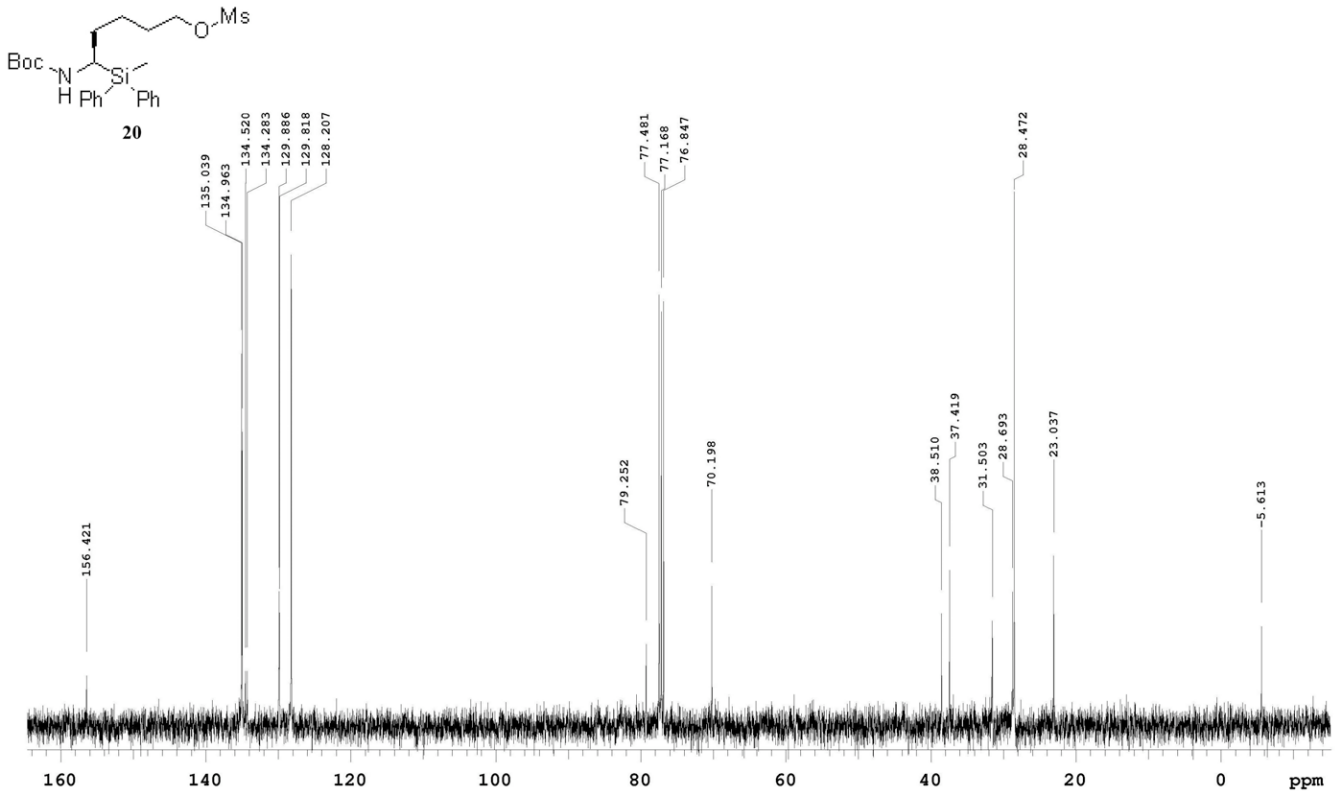
18




kbl574a12_17
Archive directory: /export/home/auto/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 26.0 C / 299.1 K
File: kbl574a12_17
Mercury-400BB "merc400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.994 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 400.4698269 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 25 sec
Boc
N
H
Si
Ph
Ph
OH
19
7
6
5
4
3
2
1
ppm


Boc
N
H
Si
Ph
Ph
OH
19
135.009
134.940
134.665
134.428
129.741
129.680
128.085
156.597
79.183
77.481
77.160
76.847
62.618
38.579
32.228
31.785
28.441
22.983
-5.590
160
140
120
100
80
60
40
20
0
ppm


kbl575a8_15
Archive directory: /export/home/auto/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 26.0 C / 299.1 K
File: kbl575a8_15
Mercury-400BB "merc400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.994 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 400.4698269 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 25 sec
Boc
N
H
Si
Ph
Ph
O
Ms
20
7
6
5
4
3
2
1
ppm


Boc
N
H
Si
Ph
Ph
O
Ms
20
135.039
134.963
134.520
134.283
129.886
129.818
128.207
77.481
77.168
76.847
28.472
79.252
70.198
38.510
37.419
31.503
28.693
23.037
156.421
-5.613
160
140
120
100
80
60
40
20
0
ppm

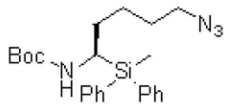
kbl576a4_9

Archive directory: /export/home/auto/vnmrsys/data
Sample directory:

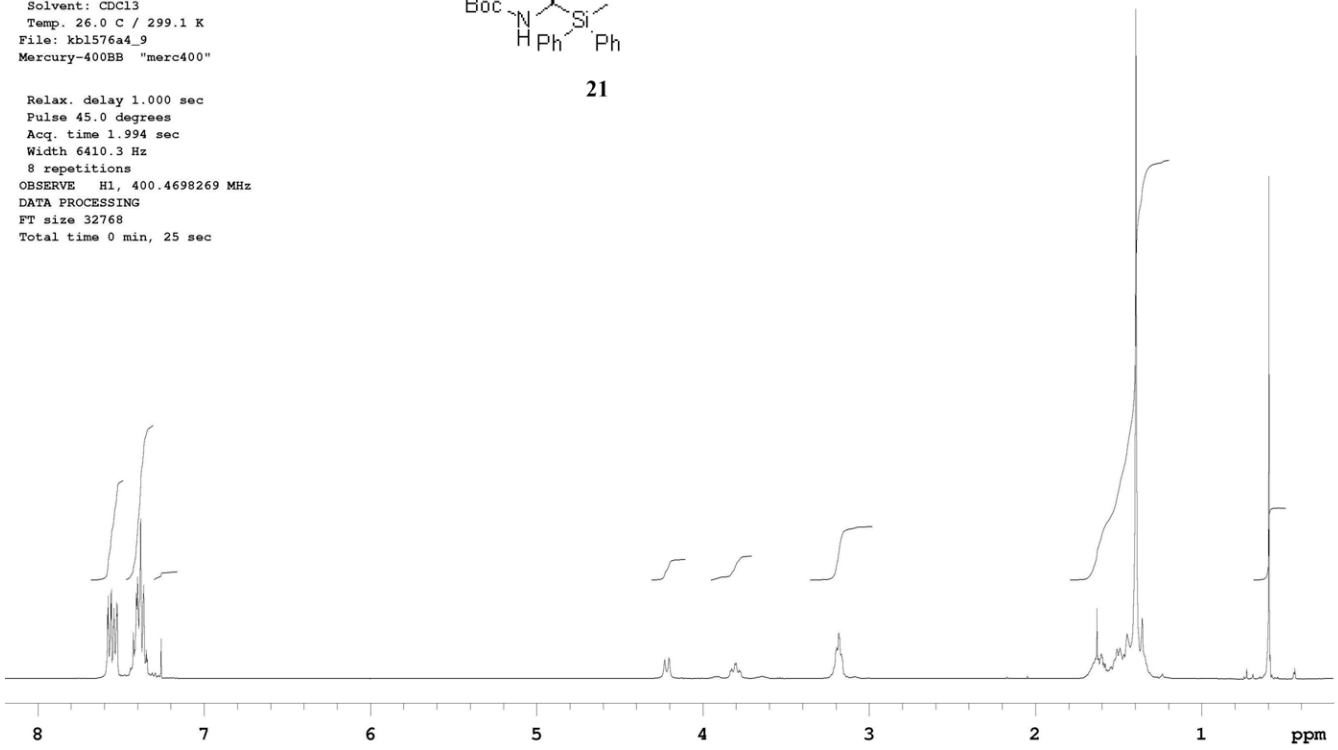
Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 26.0 C / 299.1 K
File: kbl576a4_9
Mercury-400BB "merc400"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.994 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 400.4698269 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 25 sec



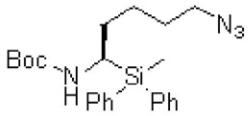
21



ref

exp3 s2pul

SAMPLE		SPECIAL	
date	Aug 24 2009	temp	26.0
solvent	CDCl3	gain	not used
file	exp	spin	20
ACQUISITION		hst	0.008
sw	25188.9	pw90	10.500
at	1.199	alfa	9.550
np	60422	FLAGS	
fb	13800	il	n
bs	16	in	n
d1	1.000	dp	y
nt	256	hs	nn
ct	96	PROCESSING	
TRANSMITTER		lb	1.00
tn	C13	fn	not used
sfrq	100.709	DISPLAY	
tof	1561.6	sp	-4537.6
tpwr	62	wp	18232.1
pw	5.250	rfl	9298.3
DECOUPLER		rfp	7769.9
dn	H1	rp	+19.9
dof	0	lp	-252.5
dm	yyy	PLOT	
dmm	w	wc	270
dpwr	38	sc	0
dmf	11800	vs	92
		th	6
		nm	135.070 134.386 ph



21

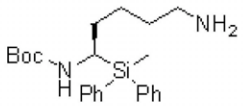
kb1577a_3_4

Archive directory: /export/home/auto/vnmrsys/data
Sample directory:

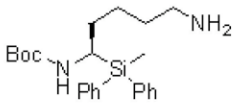
Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 26.0 C / 299.1 K
File: kb1577a_3_4
Mercury-400BB "merc400"

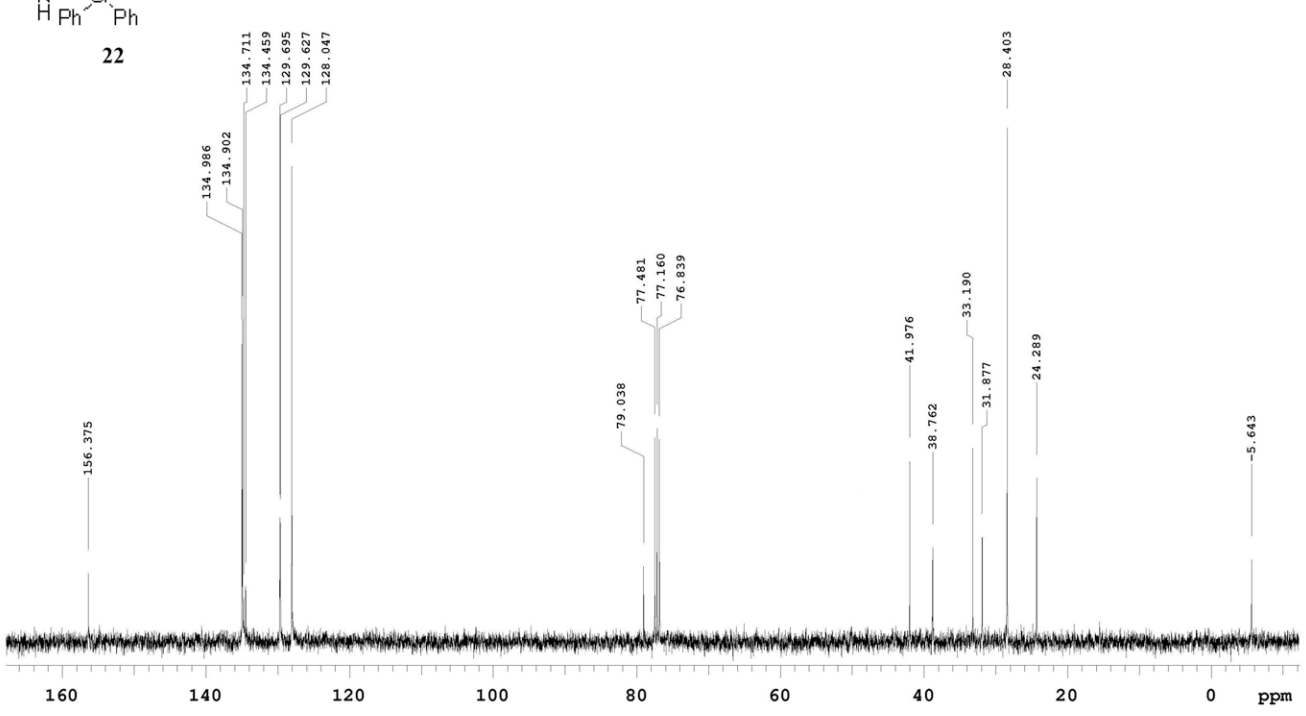
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.994 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 400.4698269 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 25 sec



22



22



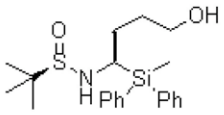
kbl361b11_16

Archive directory: /export/home/auto/vnmrsys/data
Sample directory:

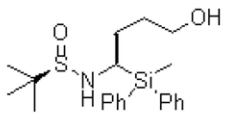
Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 26.0 C / 299.1 K
File: kbl361b11_16
Mercury-400BB "merc400"

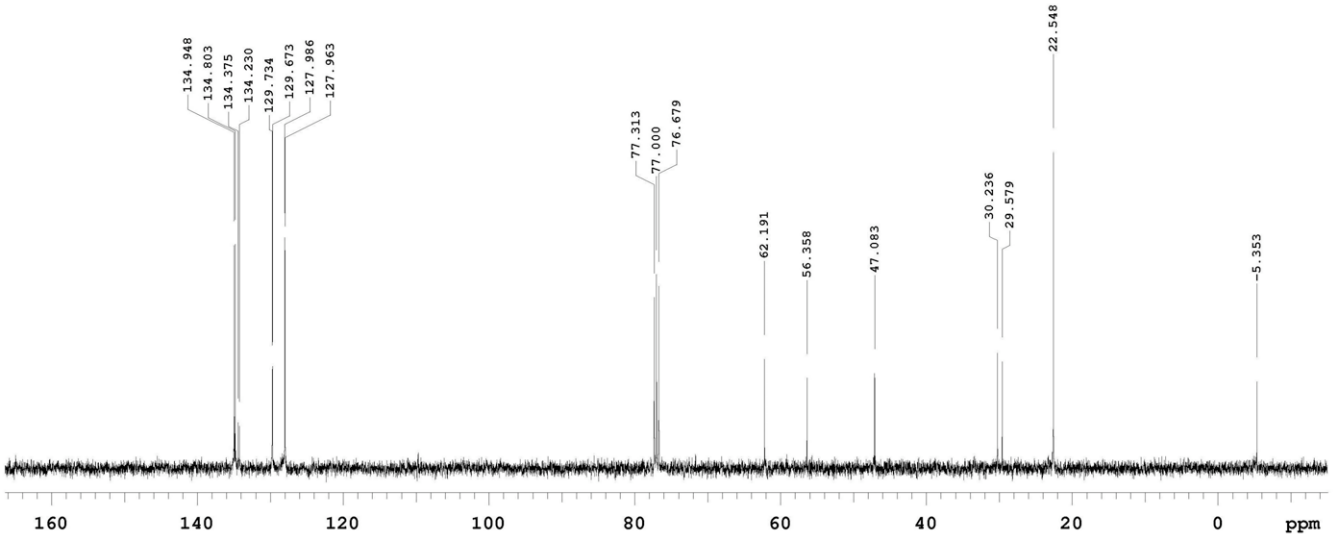
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.994 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 400.4698269 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 25 sec

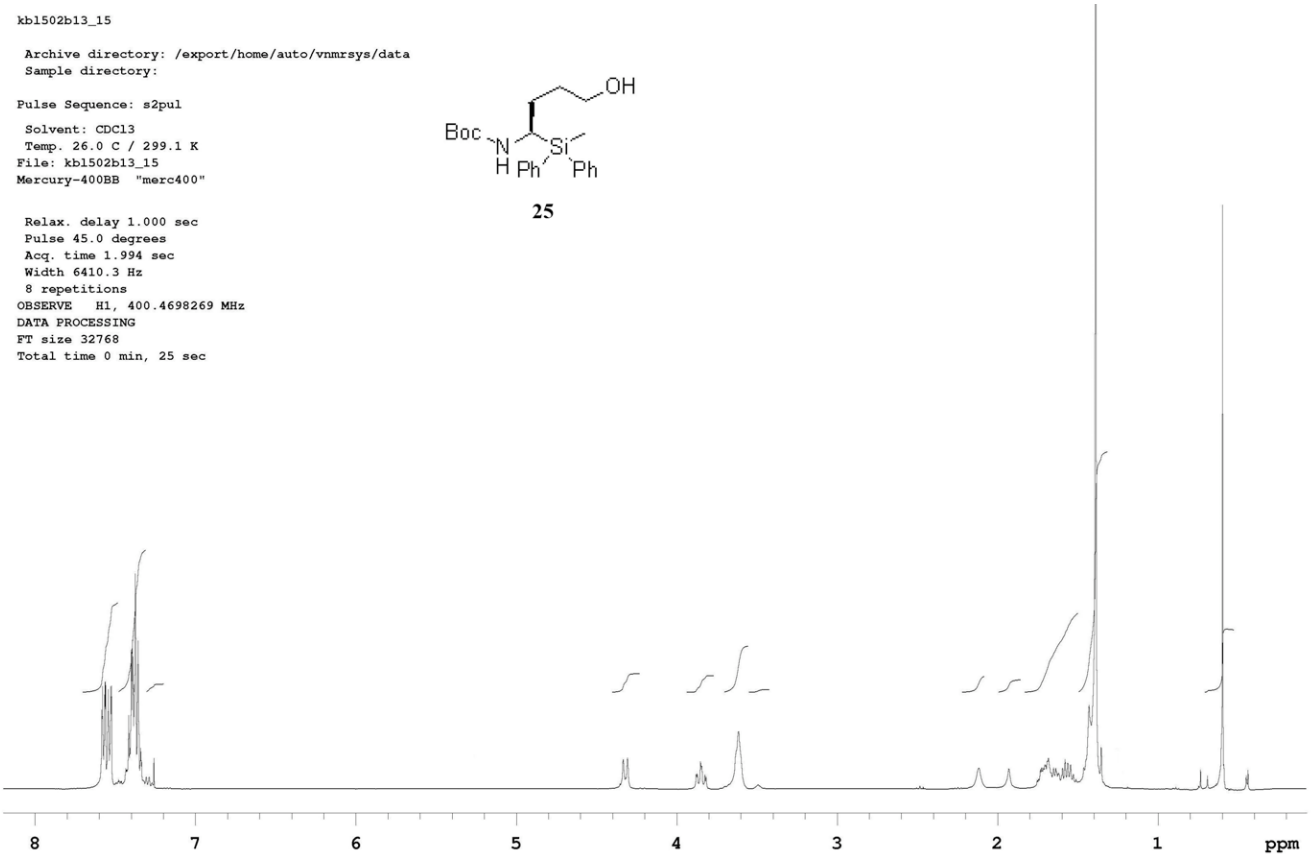


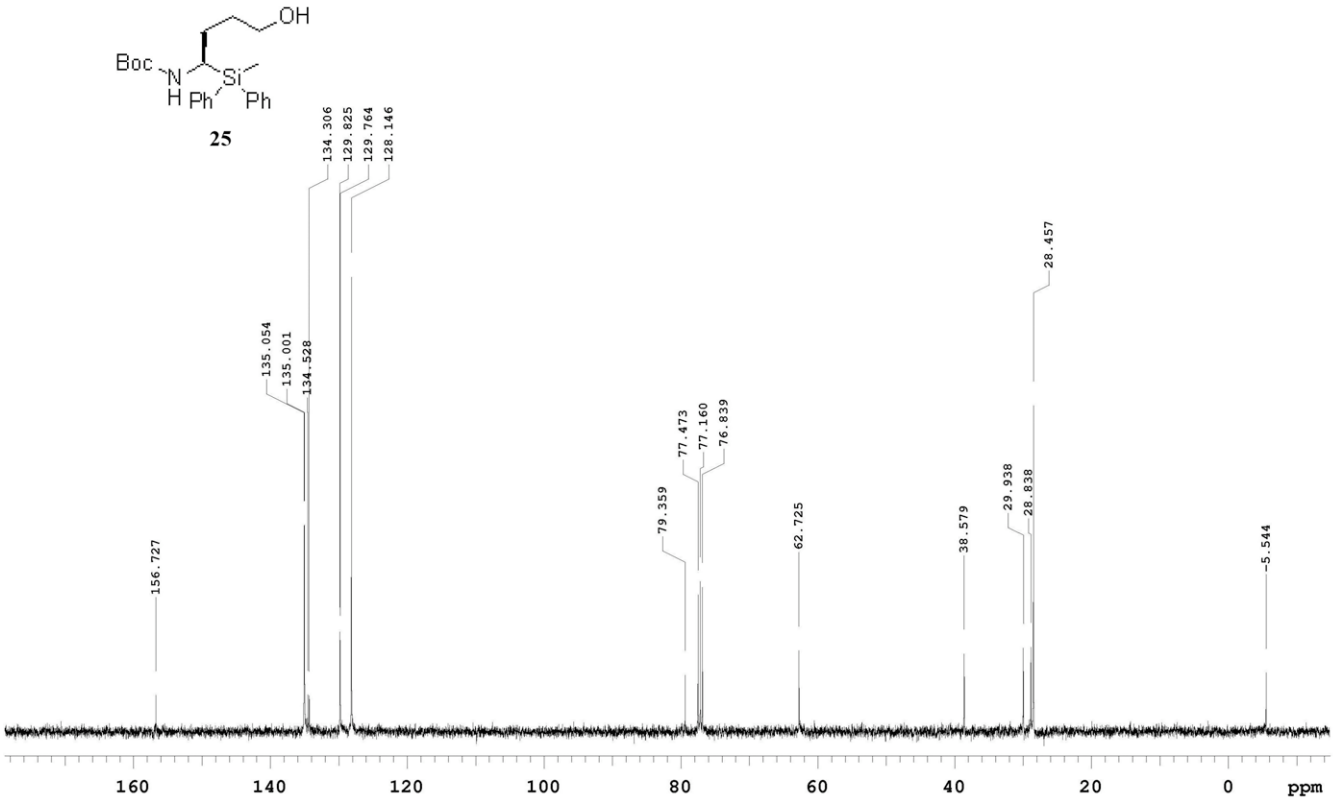
24

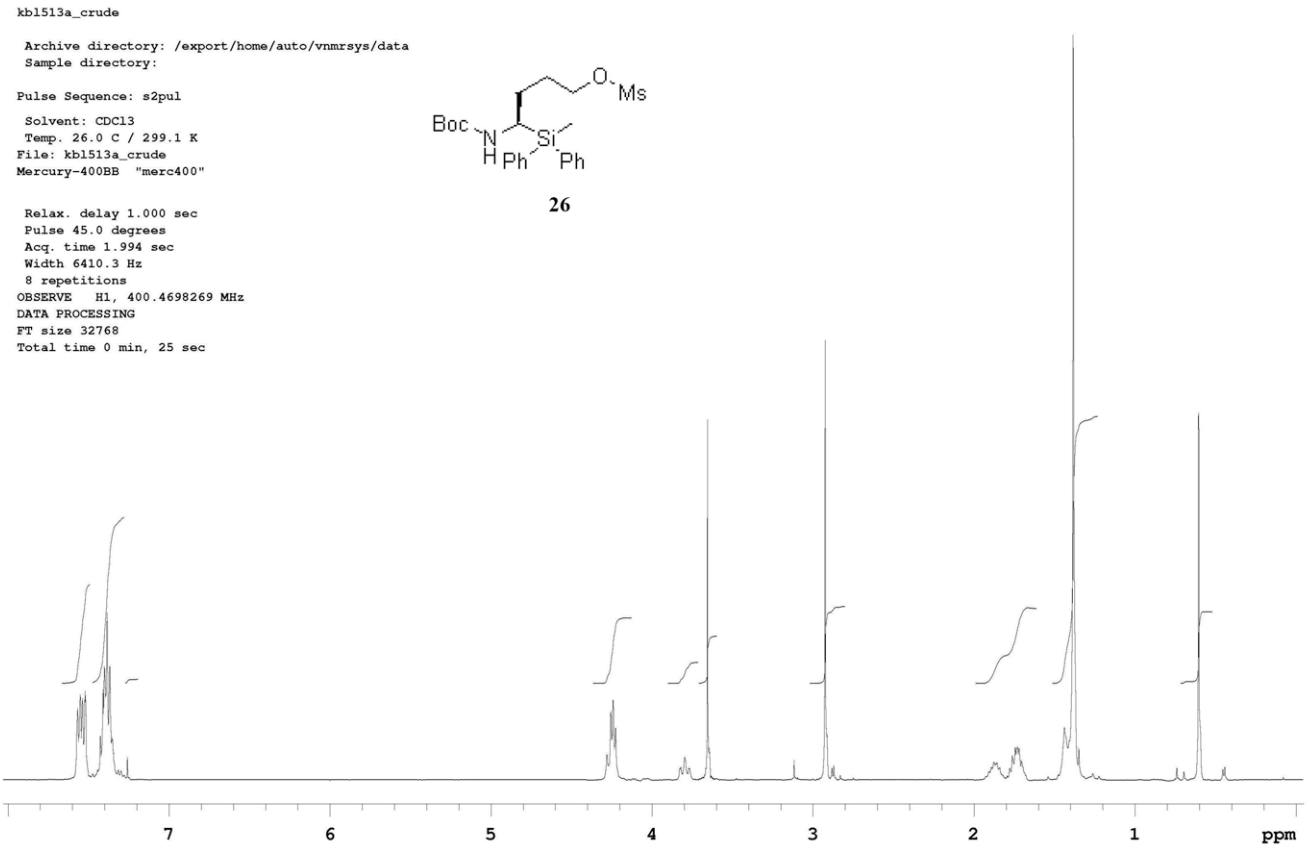


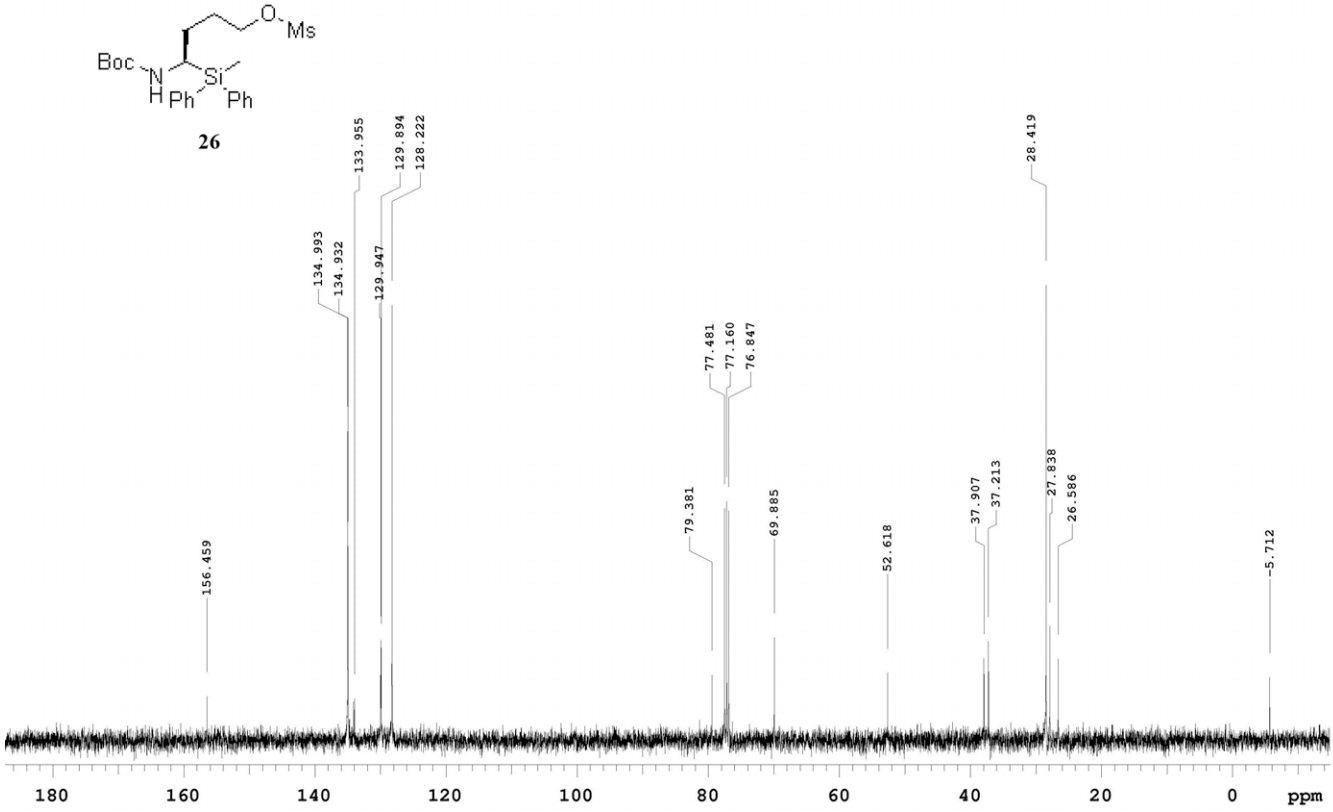
24




kb1502b13_15
Archive directory: /export/home/auto/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 26.0 C / 299.1 K
File: kb1502b13_15
Mercury-400BB "merc400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.994 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 400.4698269 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 25 sec
OH
Boc
N
H
Si
Ph
Ph
25
8
7
6
5
4
3
2
1
ppm


OH
Boc
N
H
Si
Ph
Ph
25
134.306
129.825
129.764
128.146
135.054
135.001
134.528
28.457
77.473
77.160
76.839
79.359
62.725
38.579
29.938
28.838
156.727
-5.544
160
140
120
100
80
60
40
20
0
ppm


kbl513a_crude
Archive directory: /export/home/auto/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 26.0 C / 299.1 K
File: kbl513a_crude
Mercury-400BB "merc400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.994 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 400.4698269 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 25 sec
Boc
N
H
Si
Ph
Ph
O
Ms
26
7
6
5
4
3
2
1
ppm

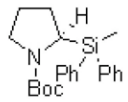

Boc
N
H
Si
Ph
Ph
O
Ms
26
156.459
134.993
134.932
133.955
129.947
129.894
128.222
79.381
77.481
77.160
76.847
69.885
52.618
37.907
37.213
28.419
27.838
26.586
-5.712
180
160
140
120
100
80
60
40
20
0
ppm

kbl513a3_5

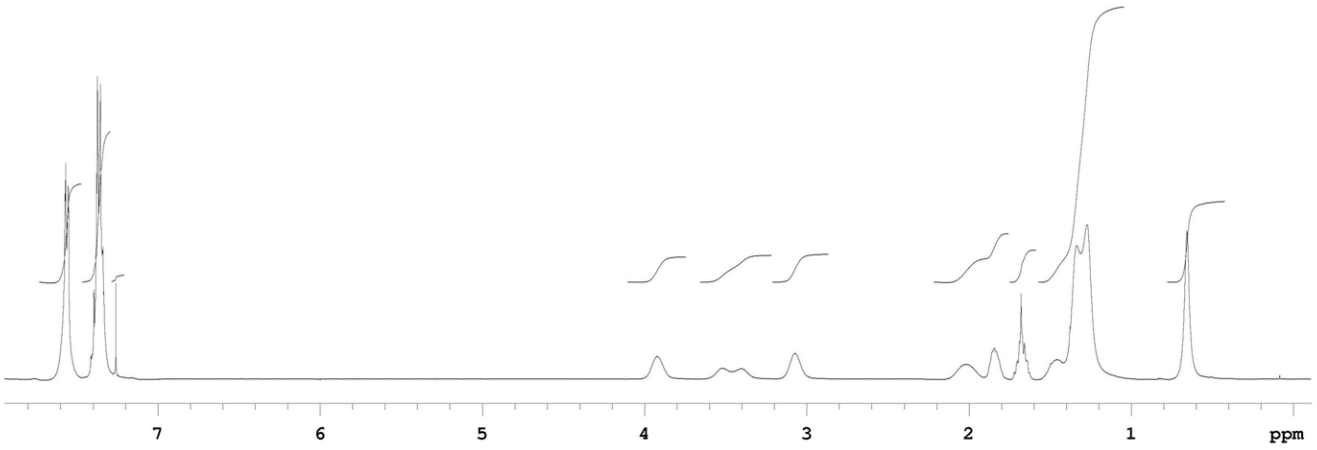
Archive directory: /export/home/auto/vnmrsys/data
Sample directory:

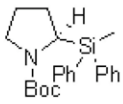
Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 26.0 C / 299.1 K
File: kbl513a3_5
Mercury-400BB "merc400"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.994 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 400.4698269 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 25 sec

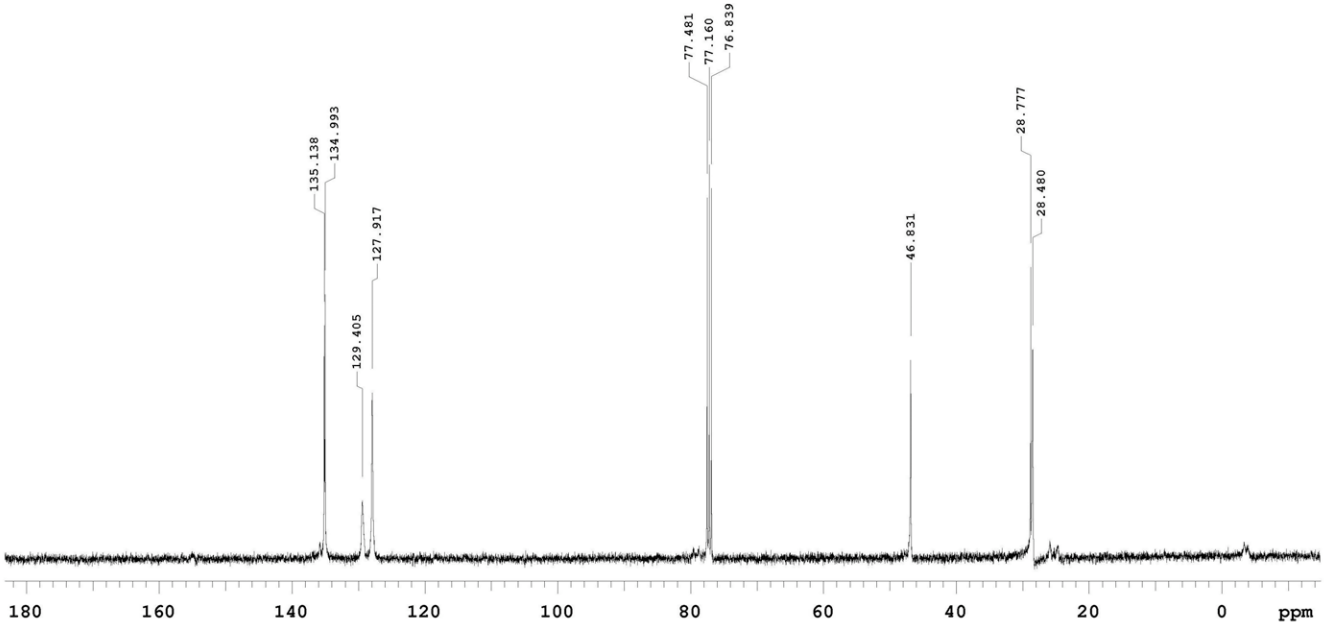


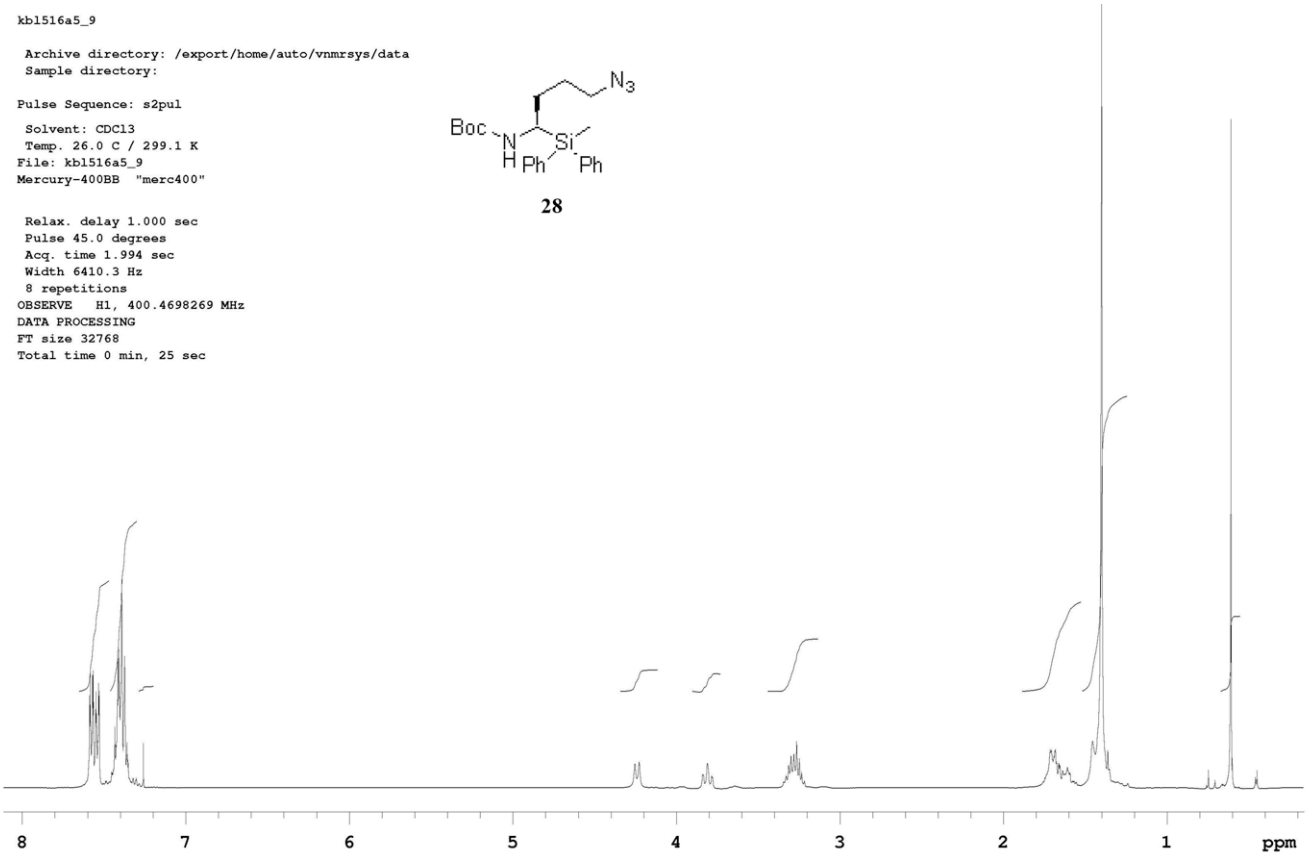
27

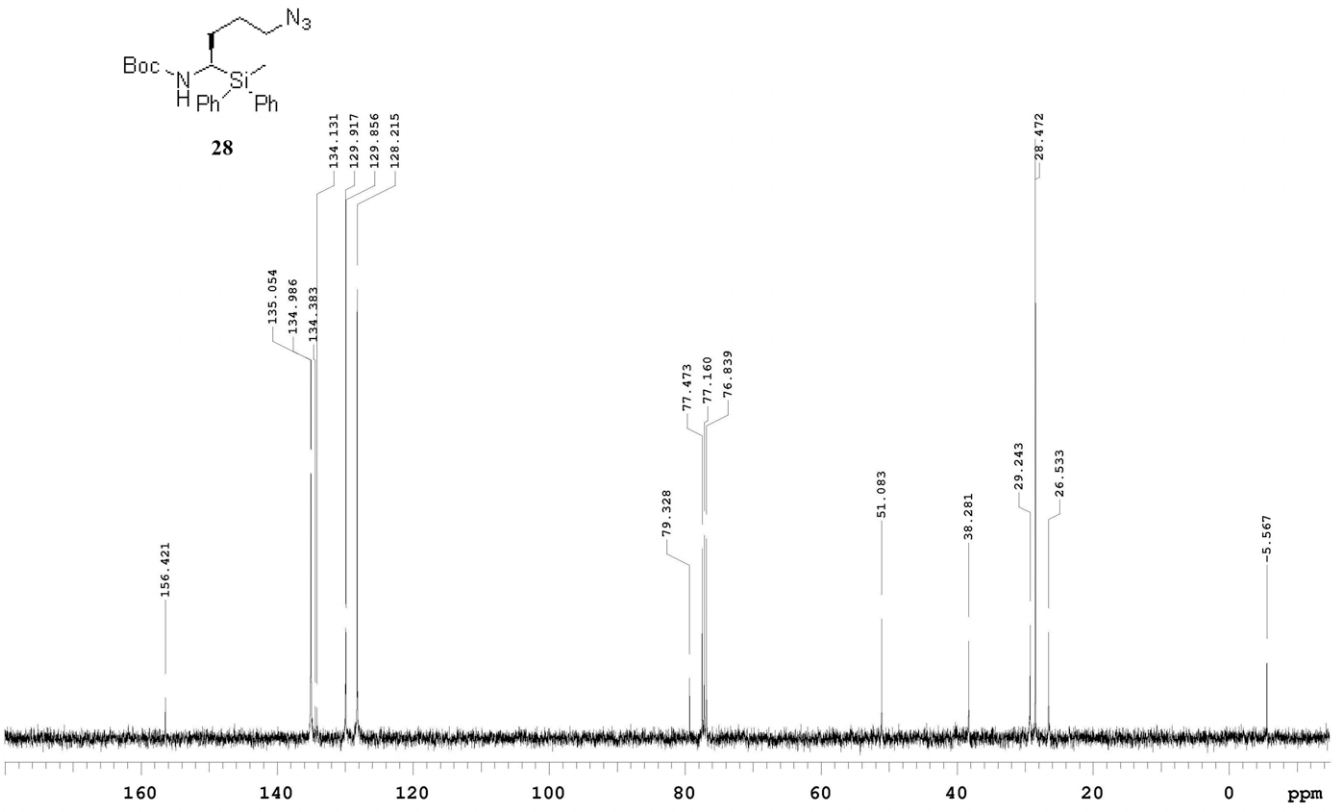




27




kbl516a5_9
Archive directory: /export/home/auto/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 26.0 C / 299.1 K
File: kbl516a5_9
Mercury-400BB "merc400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.994 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 400.4698269 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 25 sec
Boc
N
H
Ph
Si
Ph
Ph
N3
28
8
7
6
5
4
3
2
1
ppm


Boc
N
H
Ph
Si
Ph
Ph
N3
28
134.131
129.917
129.856
128.215
135.054
134.986
134.383
77.473
77.160
76.839
79.328
156.421
51.083
38.281
29.243
28.472
26.533
-5.567
160
140
120
100
80
60
40
20
0
ppm

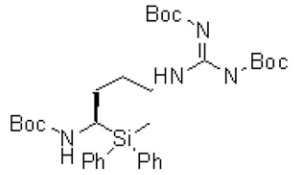
kbl519c12_15

Archive directory: /export/home/auto/vnmrsys/data
Sample directory:

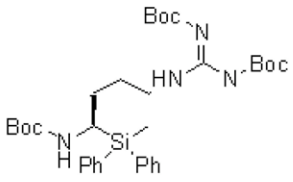
Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 26.0 C / 299.1 K
File: kbl519c12_15
Mercury-400BB "merc400"

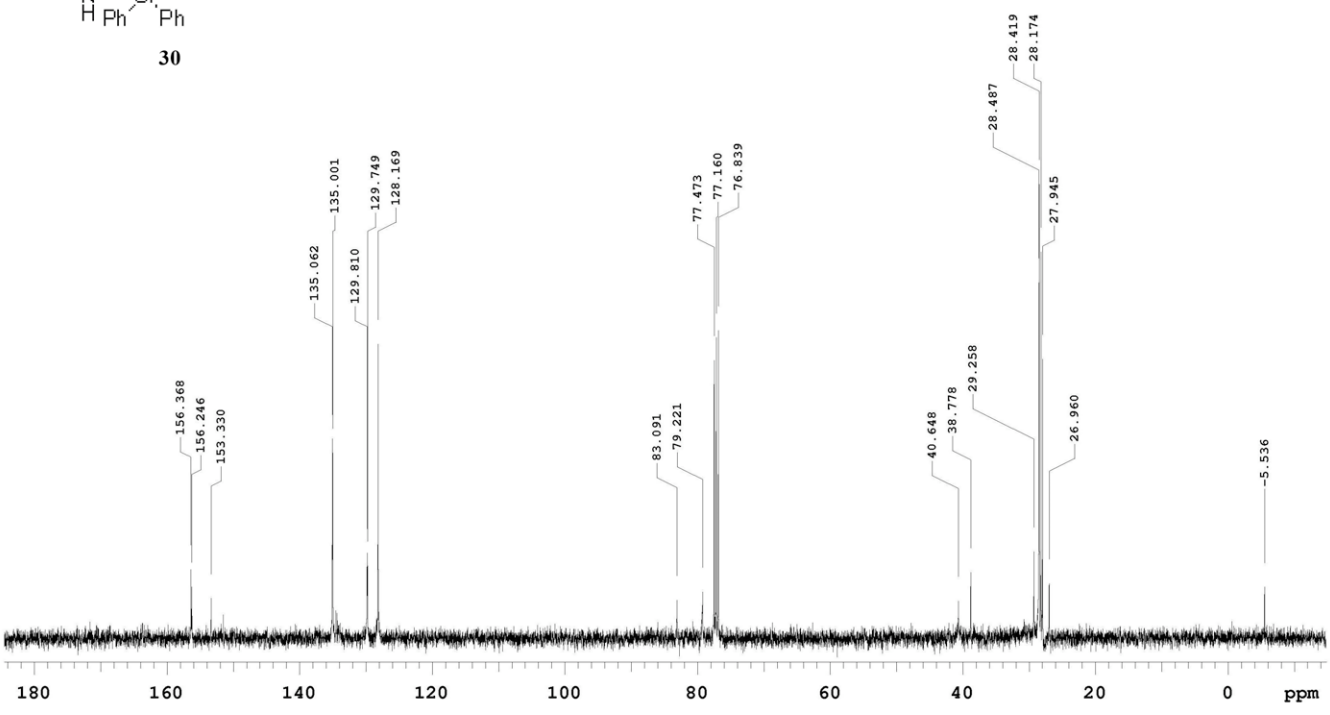
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.994 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 400.4698269 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 25 sec



30



30



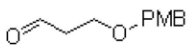
Friis_1-12_crude

Archive directory: /export/home/auto/vnmrsys/data
Sample directory:

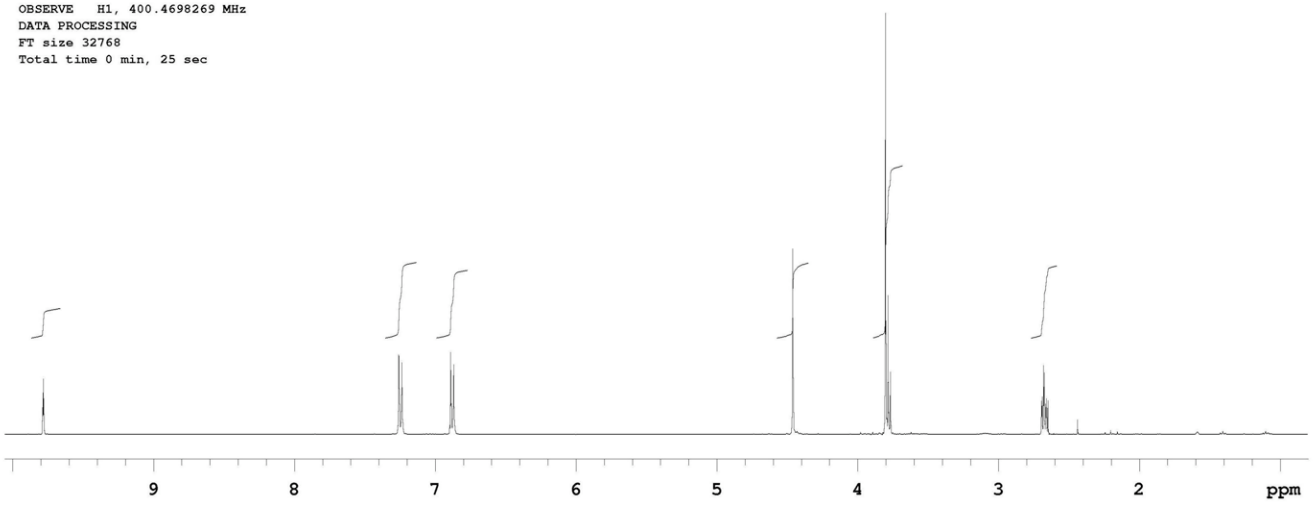
Pulse Sequence: s2pul

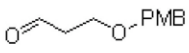
Solvent: CDCl3
Temp. 26.0 C / 299.1 K
File: Friis_1-12_crude
Mercury-400BB "merc400"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.994 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 400.4698269 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 25 sec

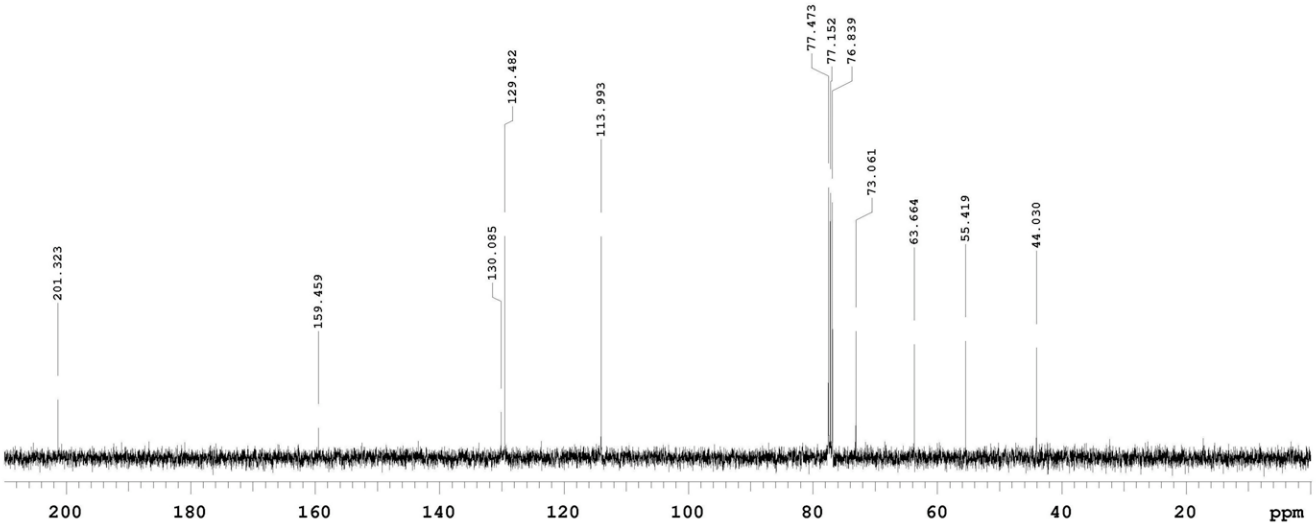


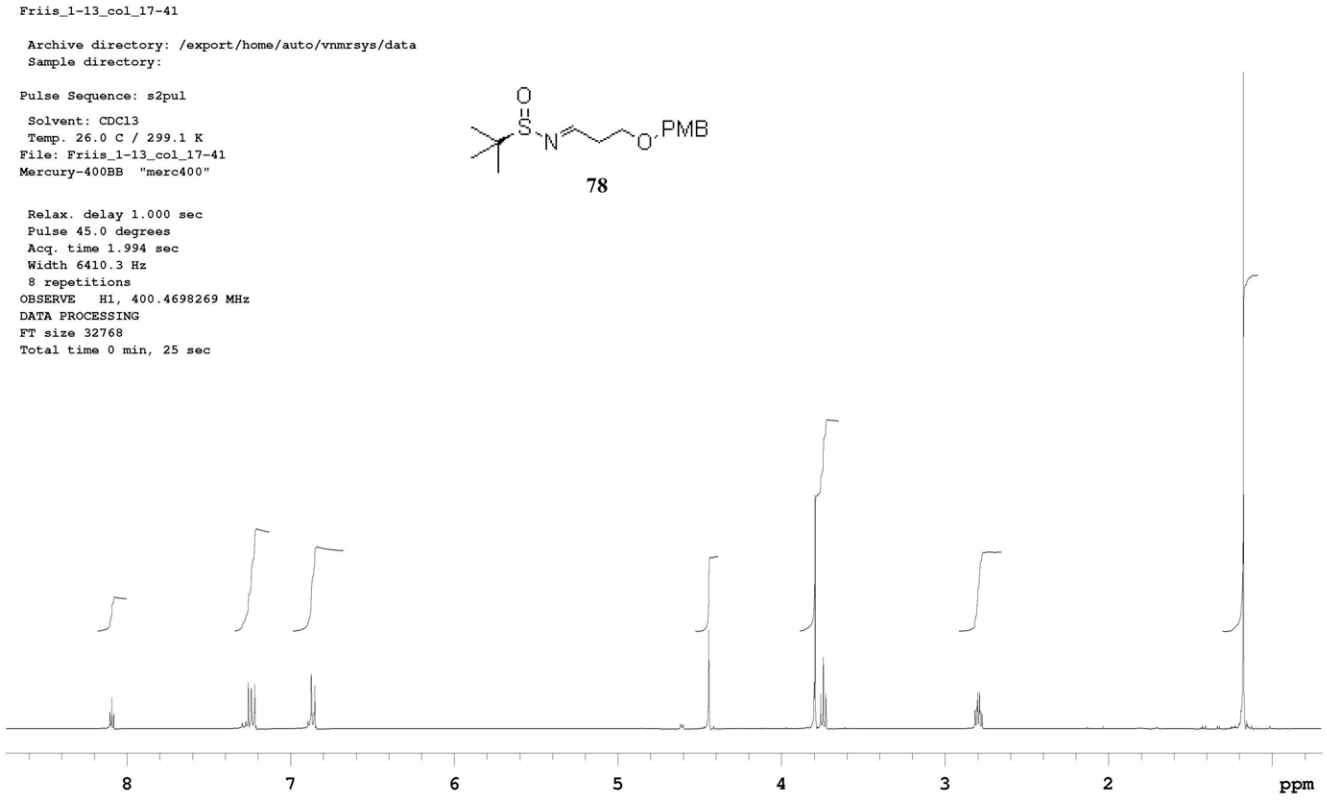
76

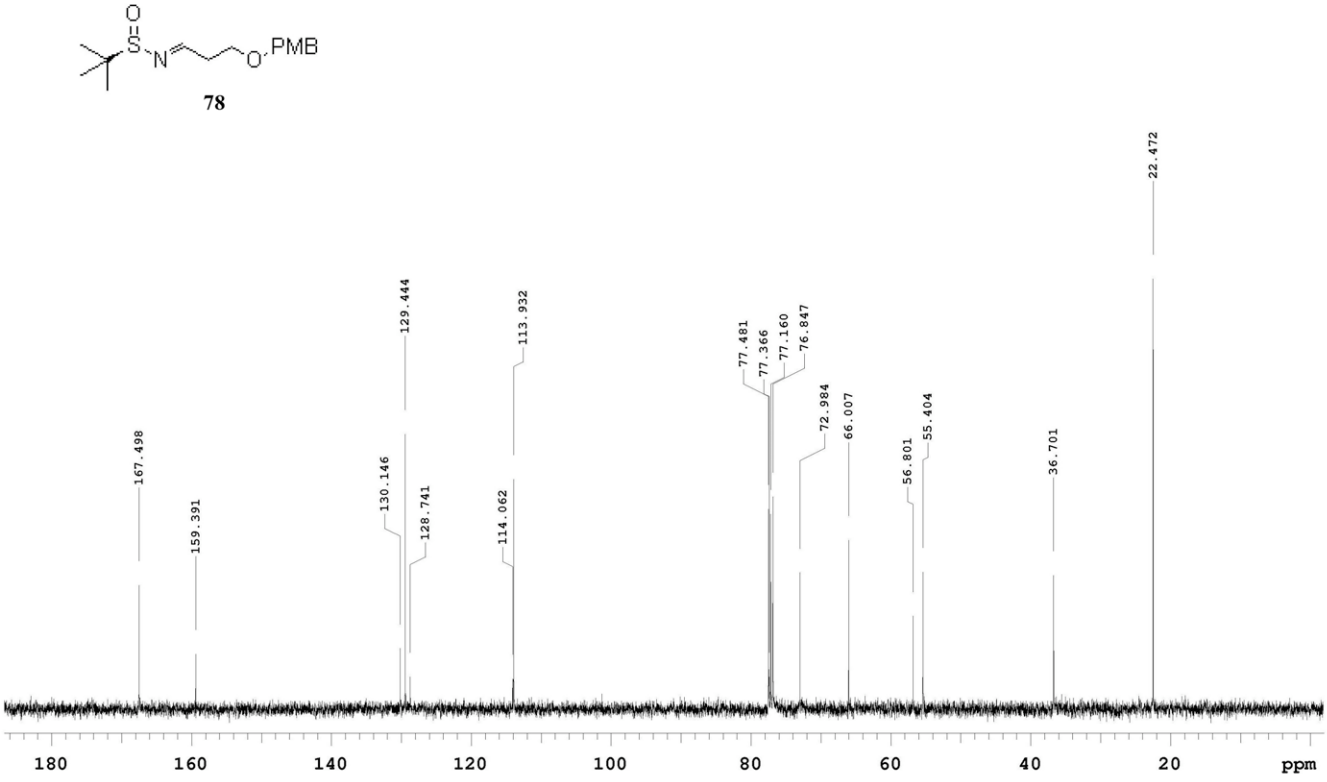


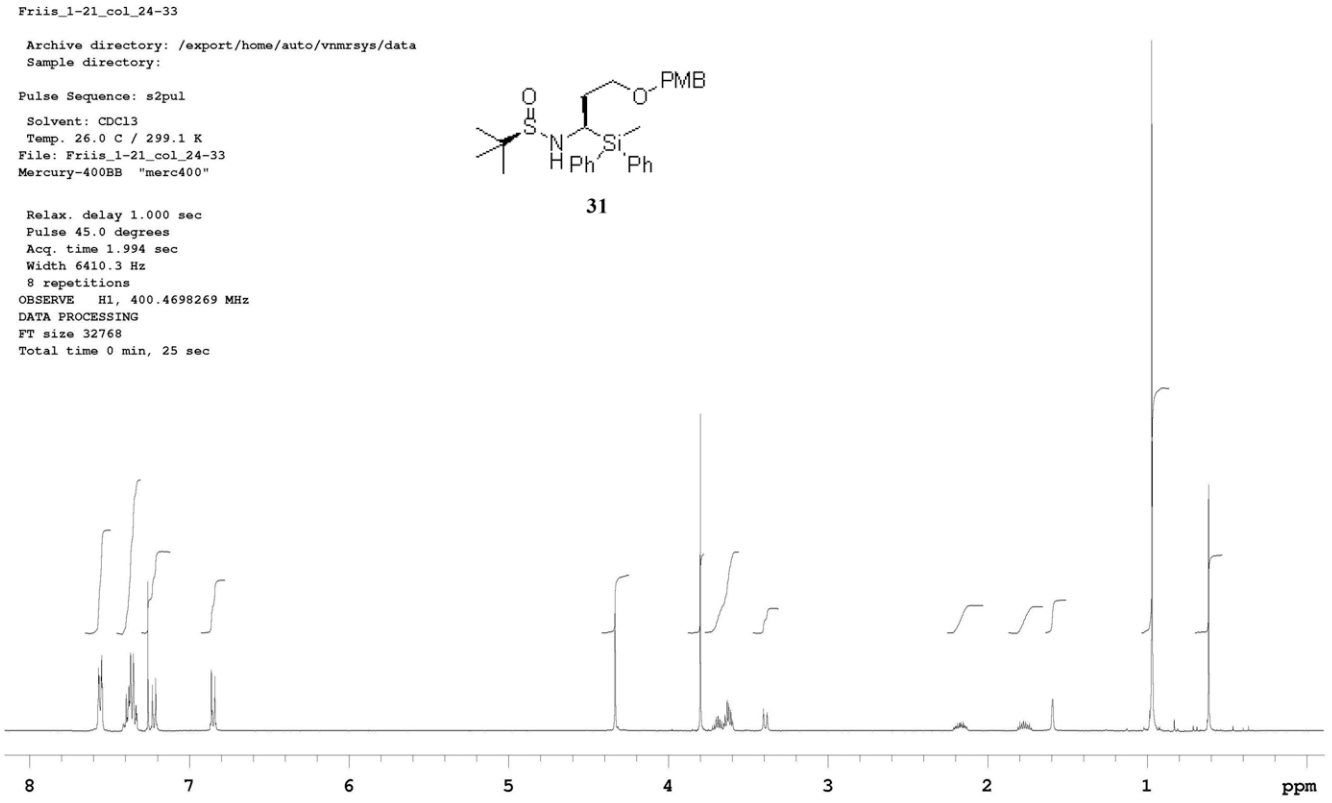


76

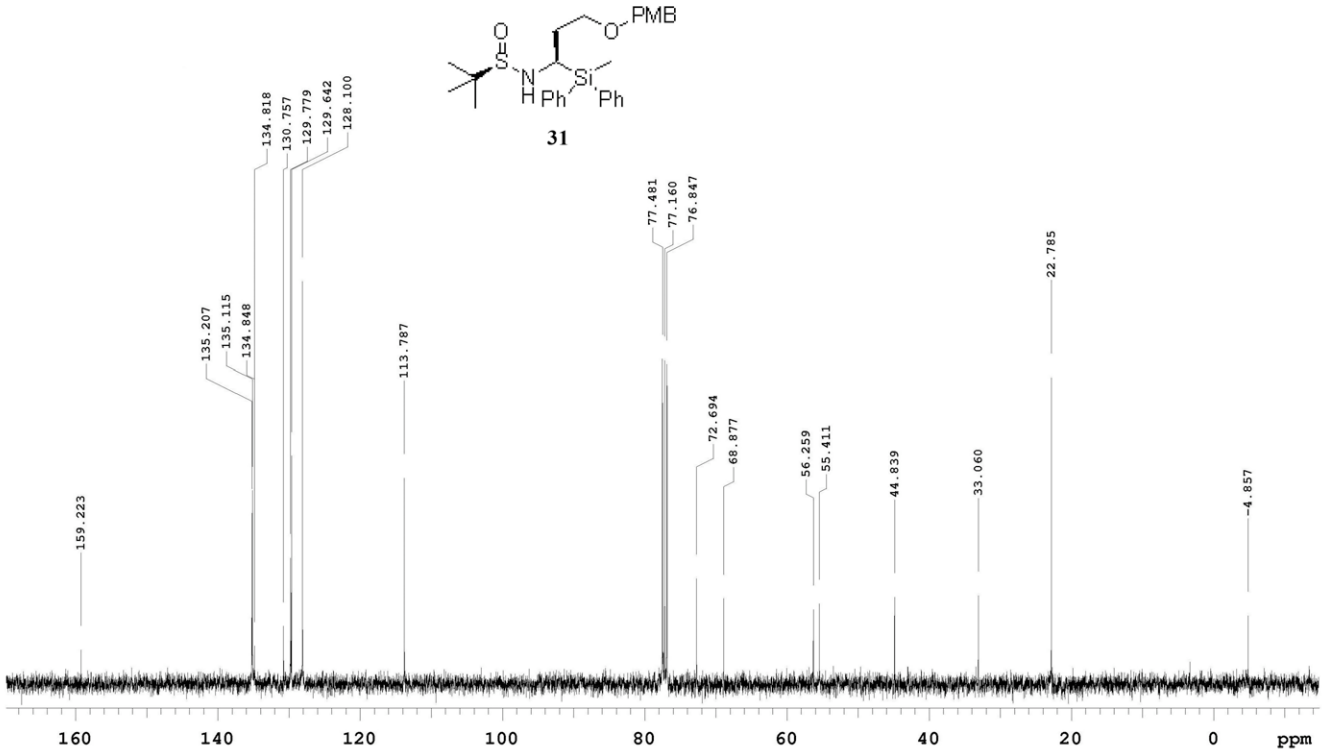



Friis_1-13_col_17-41
Archive directory: /export/home/auto/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 26.0 C / 299.1 K
File: Friis_1-13_col_17-41
Mercury-400BB "merc400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.994 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 400.4698269 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 25 sec
O
S
N
O
PMB
78
8
7
6
5
4
3
2
ppm

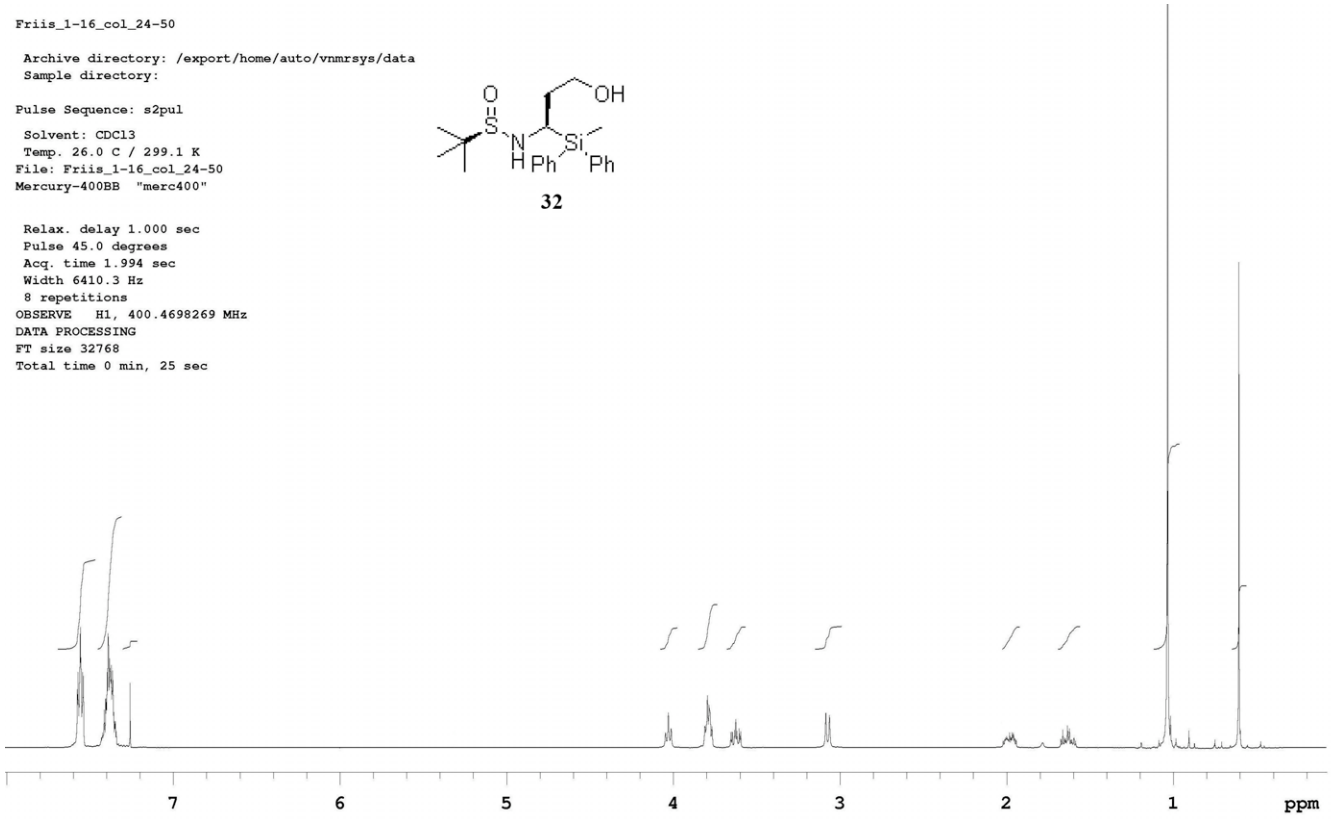

O
S
N
O
PMB
78
167.498
159.391
130.146
129.444
128.741
114.062
113.932
77.481
77.366
77.160
76.847
72.984
66.007
56.801
55.404
36.701
22.472
180
160
140
120
100
80
60
40
20
ppm

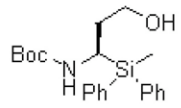

Friis_1-21_col_24-33
Archive directory: /export/home/auto/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 26.0 C / 299.1 K
File: Friis_1-21_col_24-33
Mercury-400BB "merc400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.994 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 400.4698269 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 25 sec
PMB
Ph
Ph
8
7
6
5
4
3
2
1
ppm

31

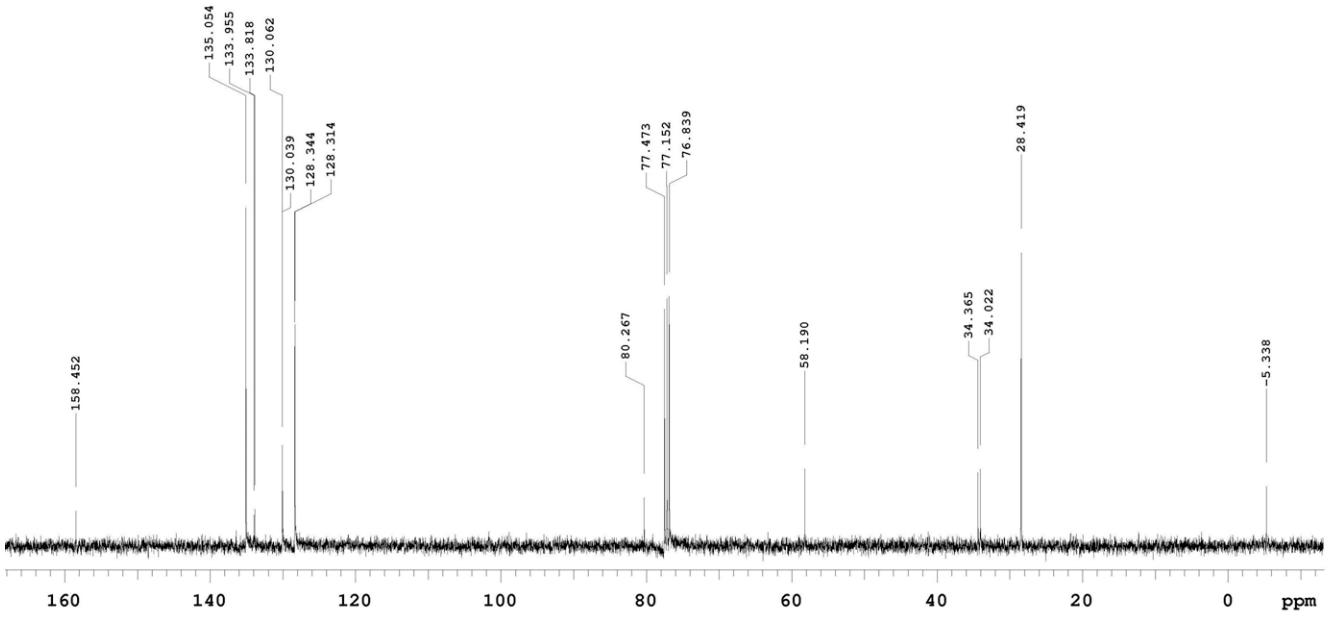

PMB
Ph
Ph
134.818
130.757
129.779
129.642
128.100
135.207
135.115
134.848
113.787
77.481
77.160
76.847
72.694
68.877
56.259
55.411
44.839
33.060
22.785
159.223
-4.857
160
140
120
100
80
60
40
20
0
ppm

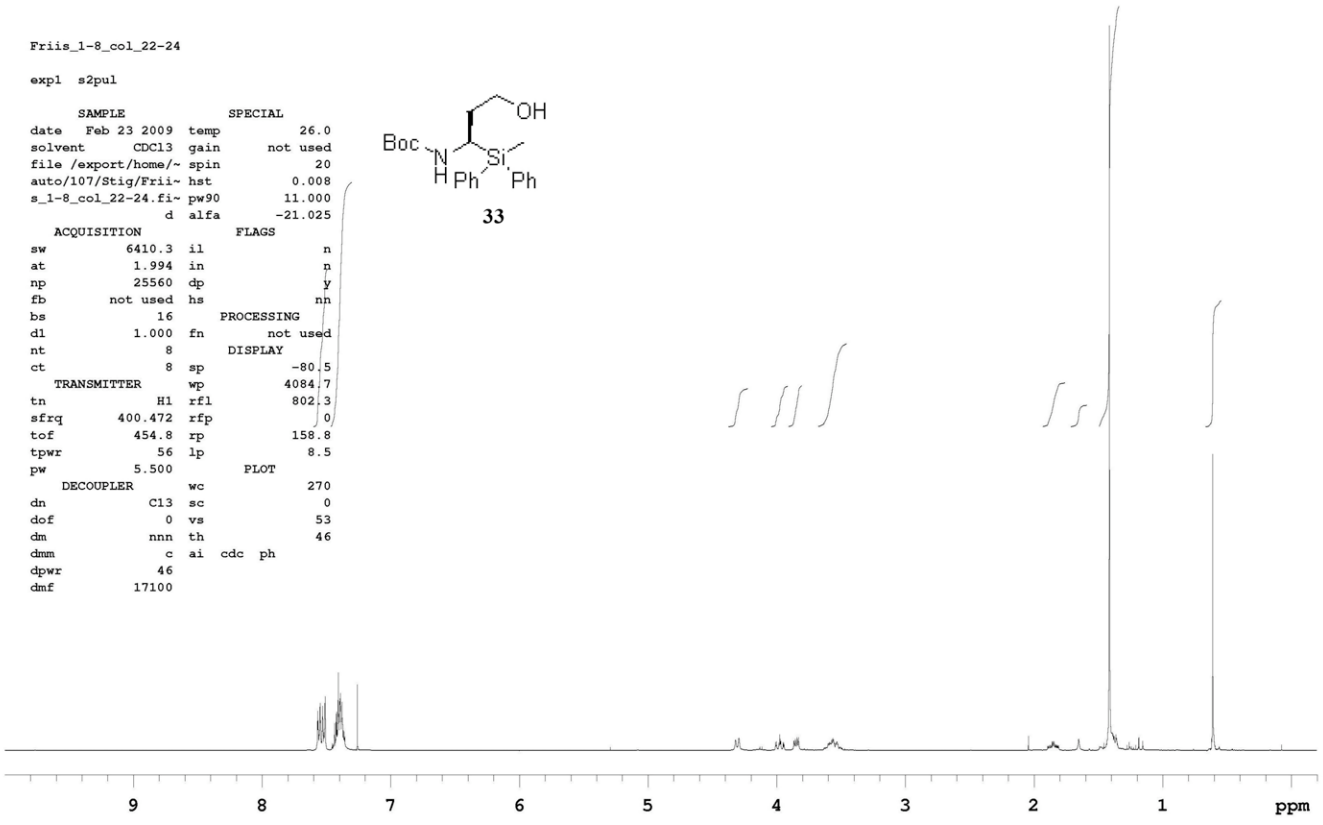
31

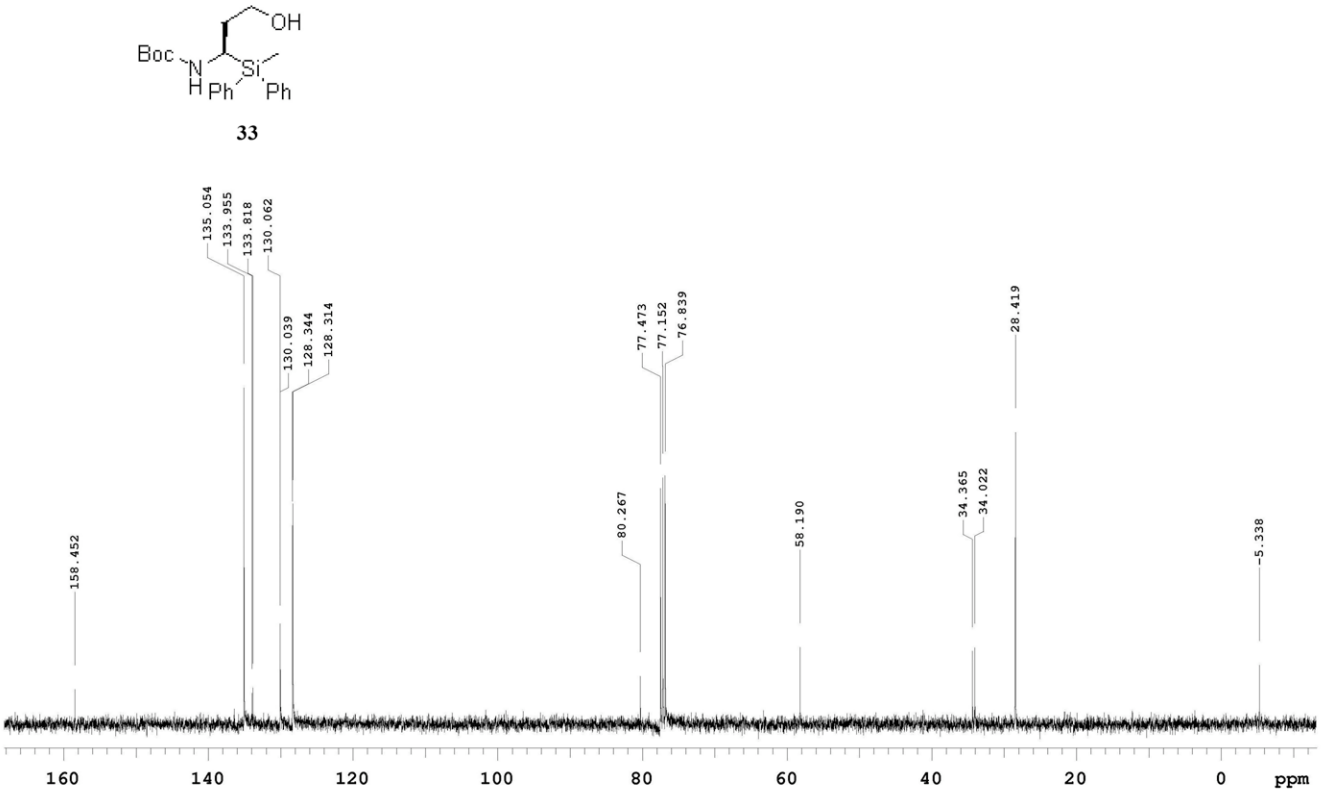

Friis_1-16_col_24-50
Archive directory: /export/home/auto/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 26.0 C / 299.1 K
File: Friis_1-16_col_24-50
Mercury-400BB "merc400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.994 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 400.4698269 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 25 sec
O
S
N
H
Ph
Si
Ph
OH
32
7
6
5
4
3
2
1
ppm


Boc
N
H
Ph
Si
Ph
OH

32


135.054
133.955
133.818
130.062
130.039
128.344
128.314
77.473
77.152
76.839
80.267
58.190
34.365
34.022
28.419
158.452
-5.338
160
140
120
100
80
60
40
20
0
ppm


Friis_1-8_col_22-24
exp1 s2pul
SAMPLE
date Feb 23 2009
solvent CDCl3
file /export/home/~
auto/107/Stig/Friis~
s_1-8_col_22-24.fi~
d
ACQUISITION
sw 6410.3
at 1.994
np 25560
fb not used
bs 16
dl 1.000
nt 8
ct 8
TRANSMITTER
tn H1
sfrq 400.472
tof 454.8
tpwr 56
pw 5.500
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 46
dmf 17100
SPECIAL
temp 26.0
gain not used
spin 20
hst 0.008
pw90 11.000
alfa -21.025
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn not used
DISPLAY
sp -80.5
wp 4084.7
rfl 802.3
rfp 0
rp 158.8
lp 8.5
PLOT
wc 270
sc 0
vs 53
th 46
ai cdc ph
OH
Boc
N
H
Si
Ph
Ph
33
9
8
7
6
5
4
3
2
1
ppm


OH
Boc
N
H
Si
Ph
Ph
33
135.054
133.955
133.818
130.062
130.039
128.344
128.314
77.473
77.152
76.839
28.419
158.452
80.267
58.190
34.365
34.022
-5.338
160
140
120
100
80
60
40
20
0
ppm

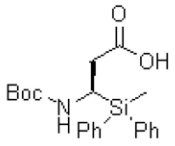
Friis_1-19_col_75-79_vacline

Archive directory: /export/home/auto/vnmrsys/data
Sample directory:

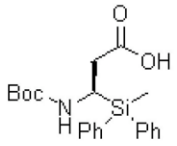
Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 26.0 C / 299.1 K
File: Friis_1-19_col_75-79_vacline
Mercury-400BB "merc400"

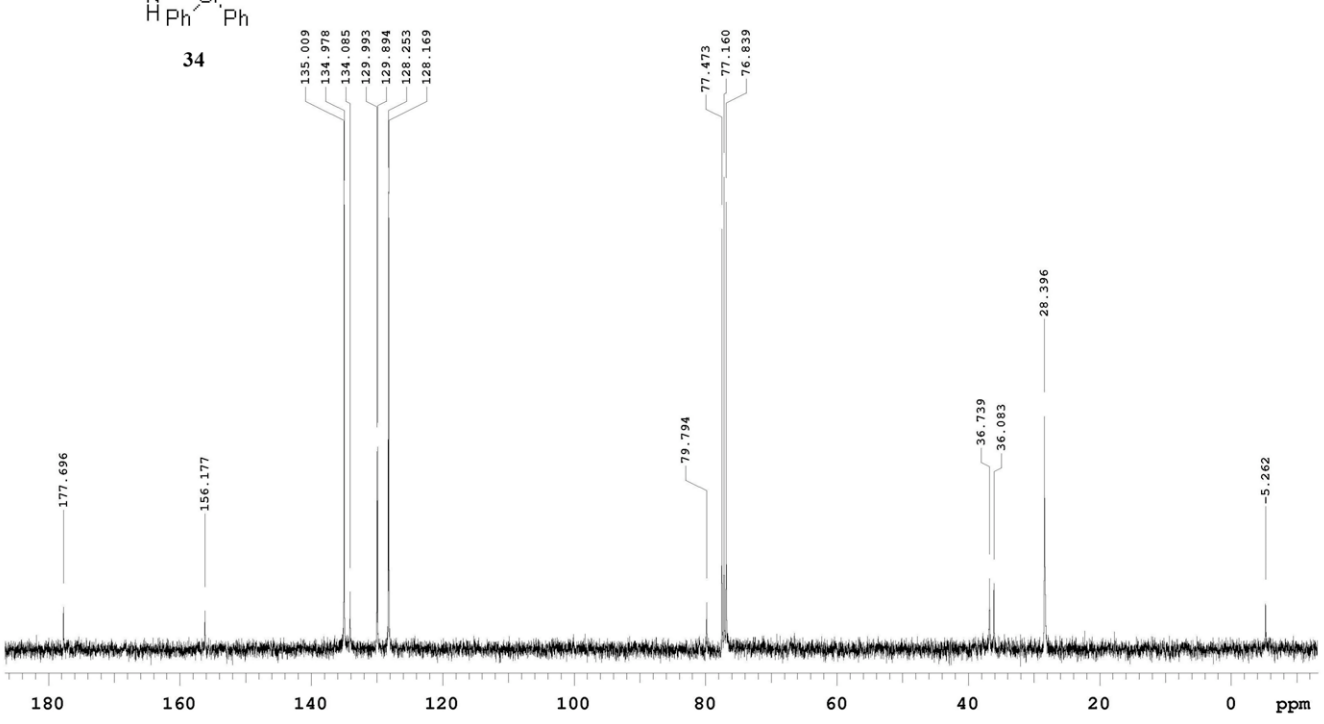
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.994 sec
Width 6410.3 Hz
16 repetitions
OBSERVE H1, 400.4698269 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 50 sec

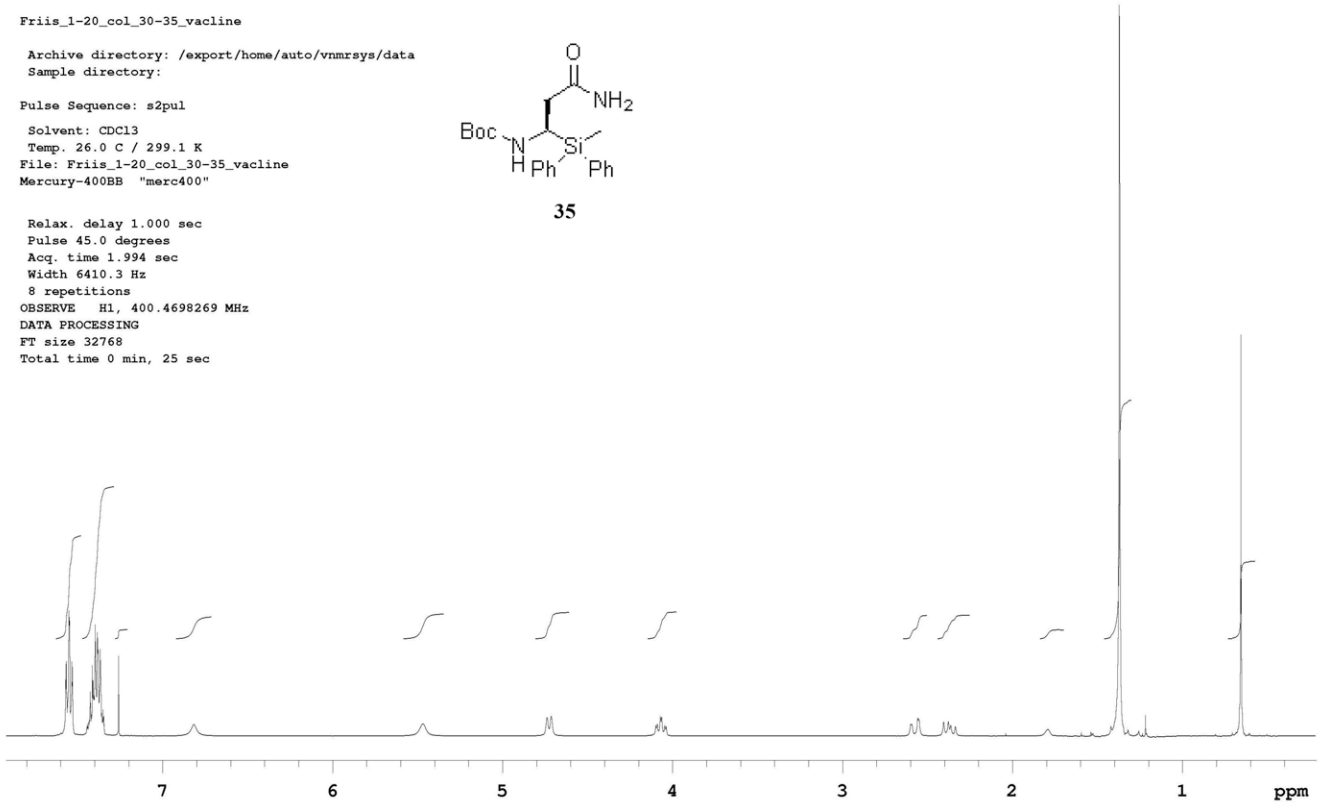


34

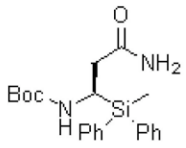


34

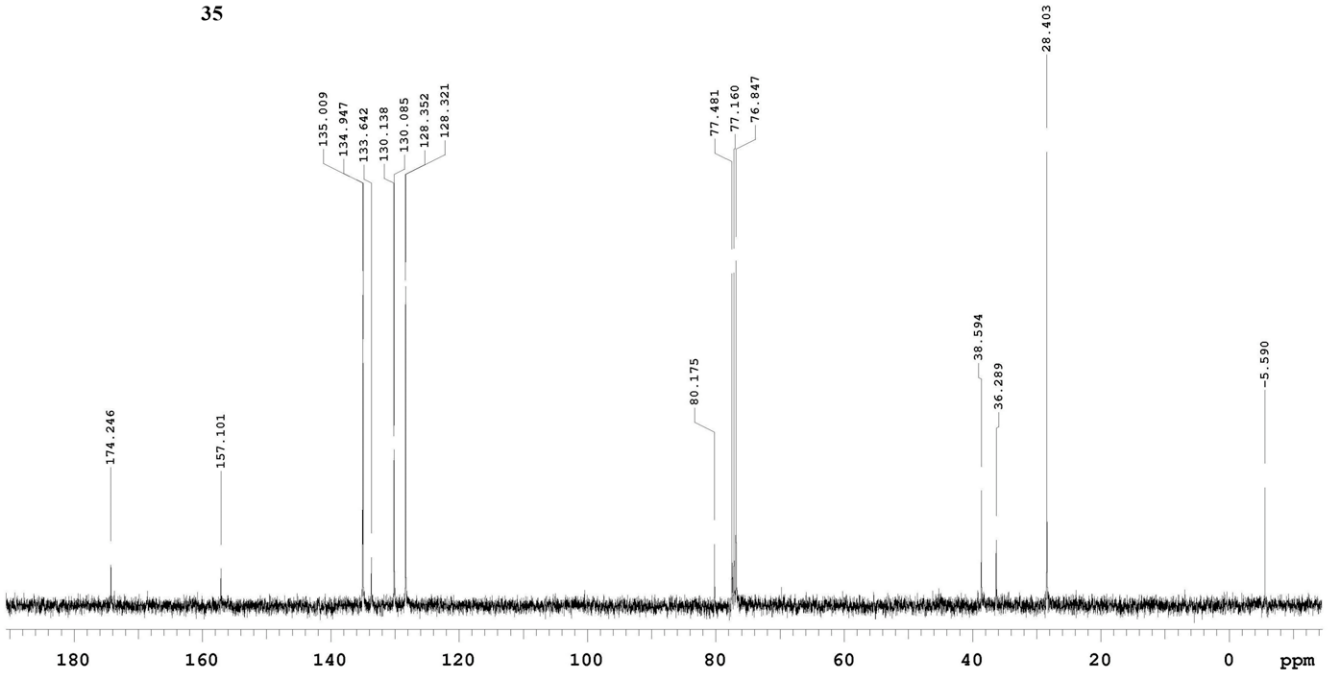


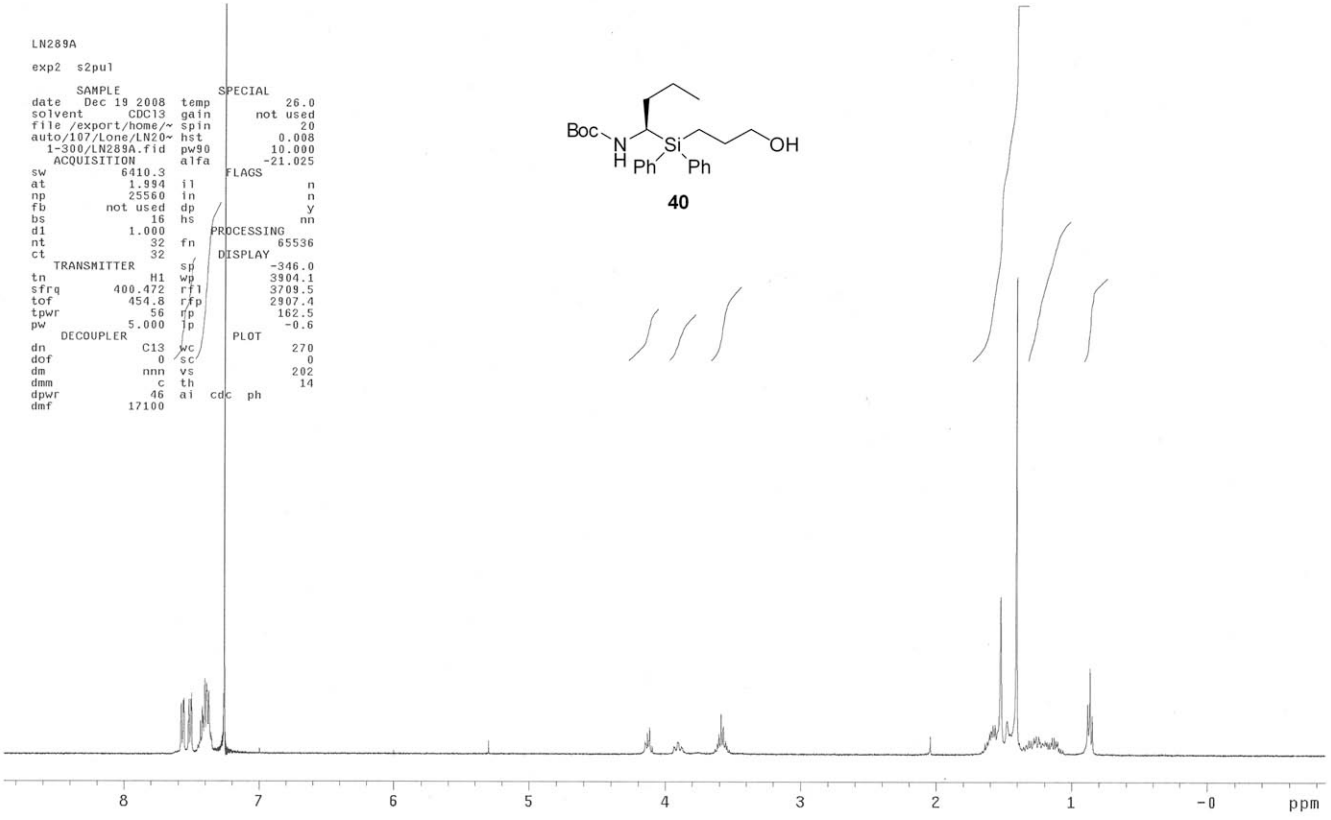

Friis_1-20_col_30-35_vacline
Archive directory: /export/home/auto/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 26.0 C / 299.1 K
File: Friis_1-20_col_30-35_vacline
Mercury-400BB "merc400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.994 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 400.4698269 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 25 sec
O
NH2
Boc
N
H
Si
Ph
Ph
7
6
5
4
3
2
1
ppm

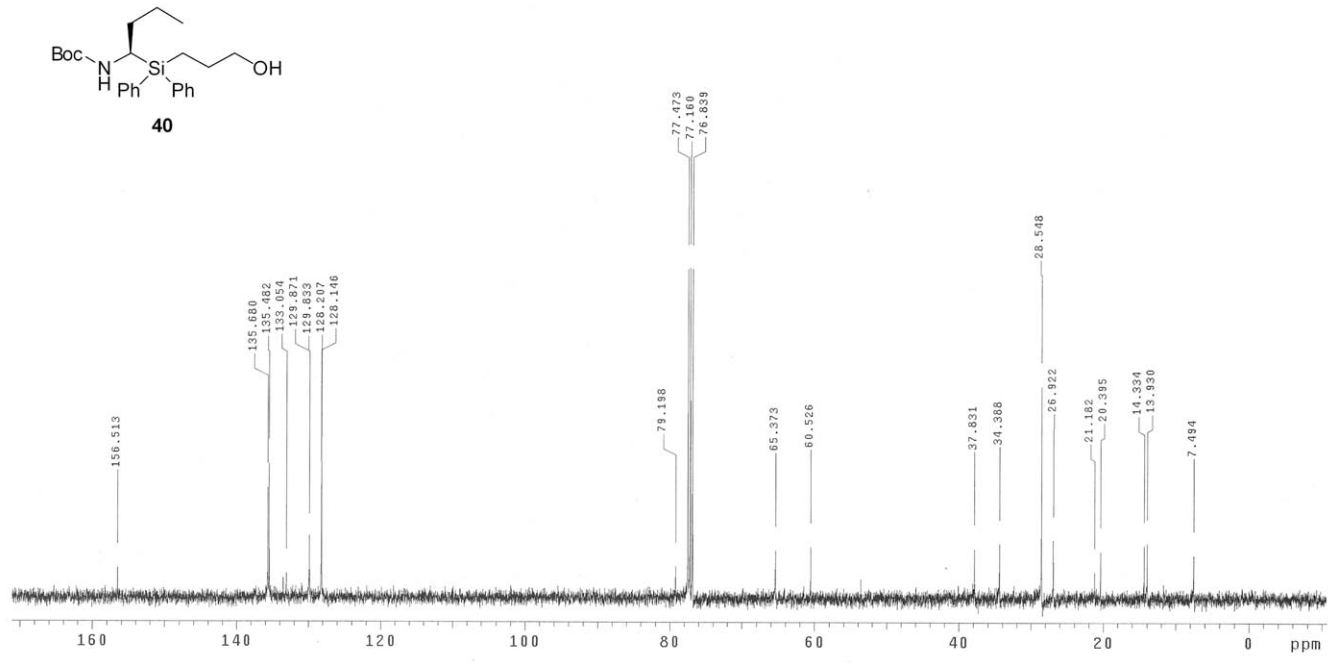
35

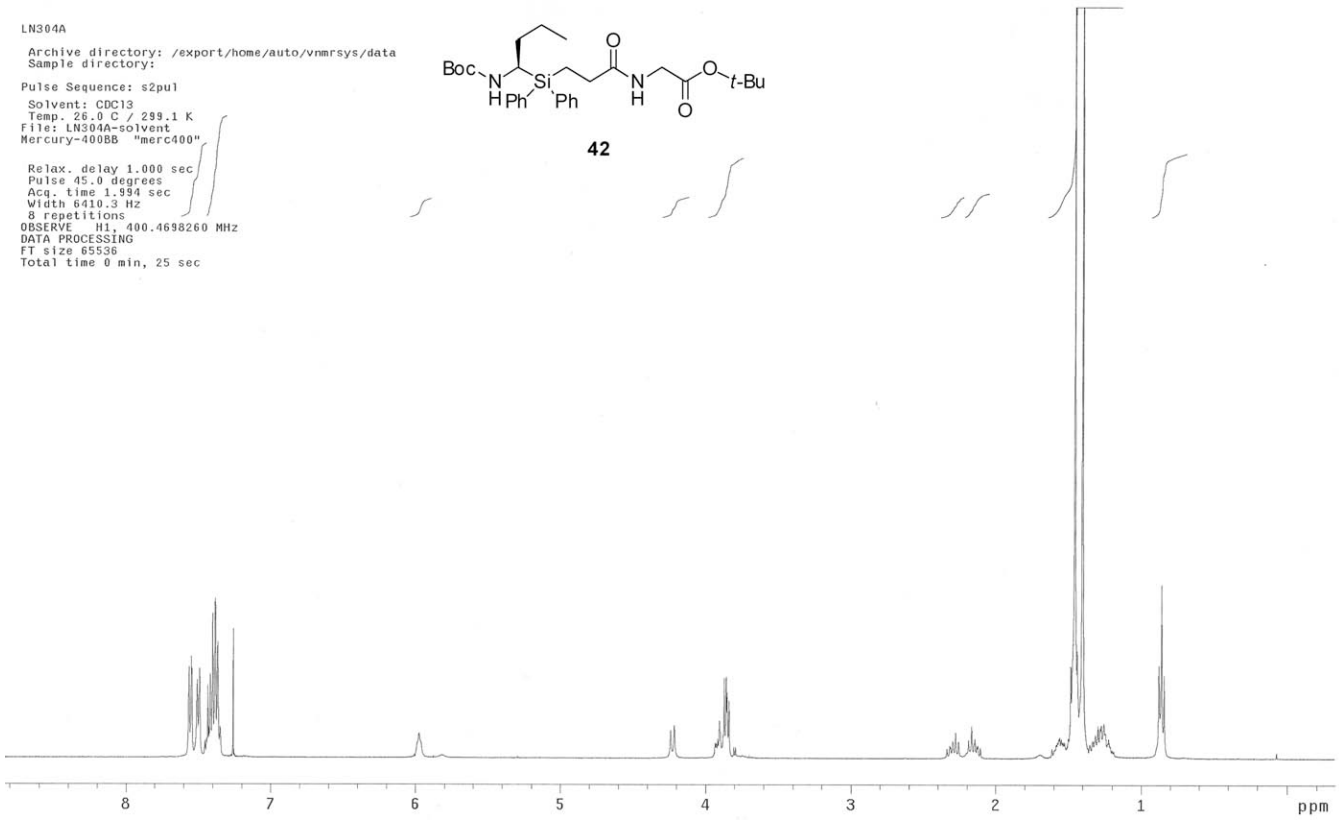


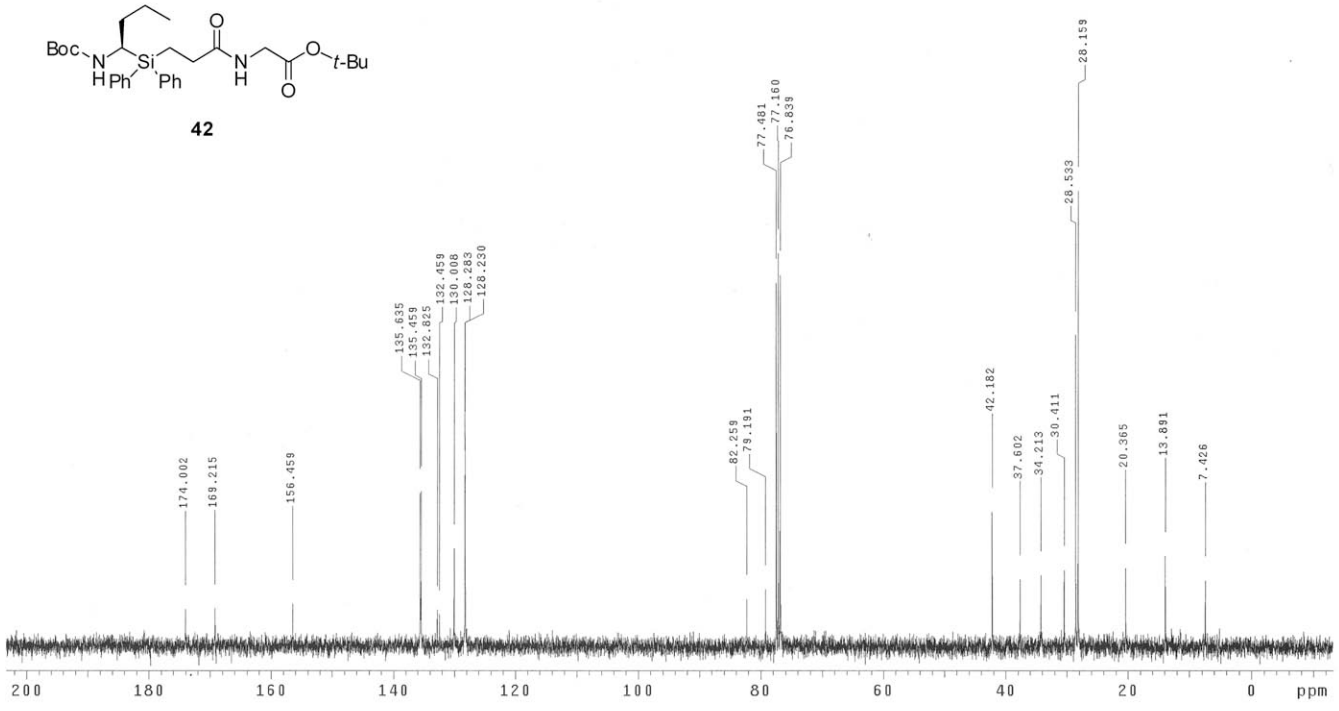
35

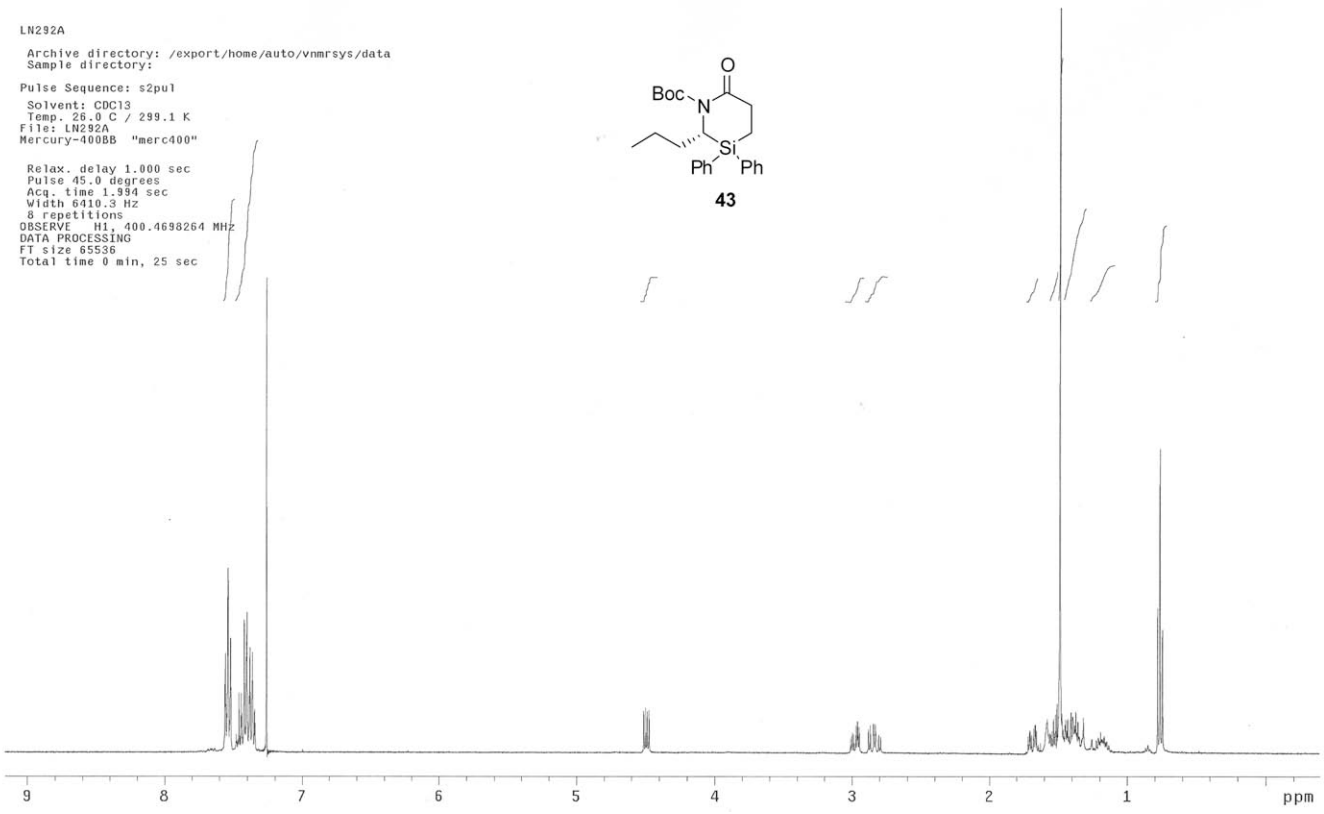

135.009
134.947
133.642
130.138
130.085
128.352
128.321
77.481
77.160
76.847
28.403
38.594
36.289
80.175
174.246
157.101
5.590
180
160
140
120
100
80
60
40
20
0
ppm

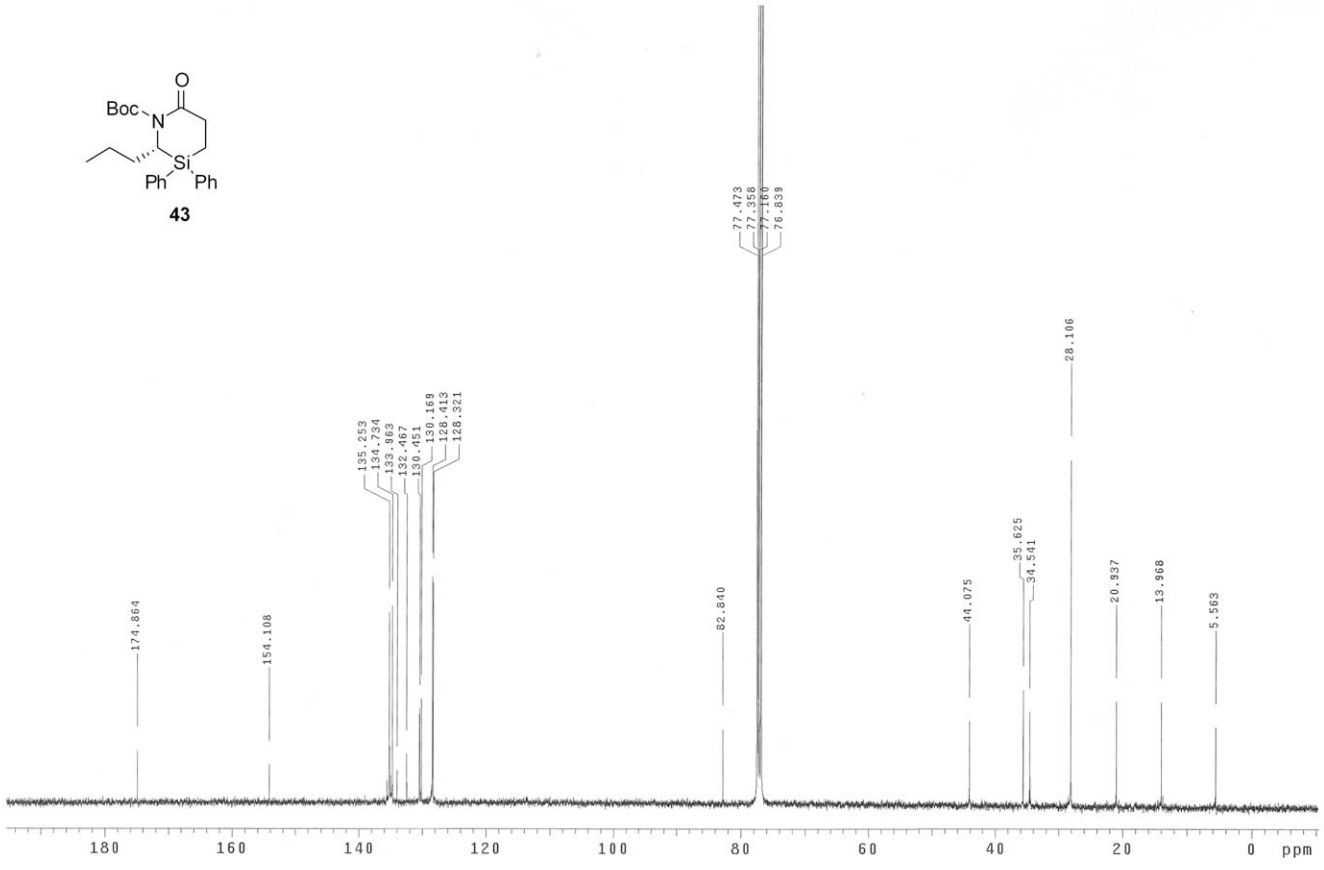

LN289A
exp2 s2pul
SAMPLE
date Dec 19 2008
solvent CDCl3
file /export/home/~
auto/107/Lone/LN20~
1-300/LN289A.fid
ACQUISITION
sw 6410.3
at 1.994
np 25560
fb not used
bs 16
d1 1.000
nt 32
ct 32
TRANSMITTER
tn H1
sfrq 400.472
tof 454.8
tpwr 56
pw 5.000
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 46
dmf 17100
SPECIAL
temp 26.0
gain not used
spin 20
hst 0.008
pw90 10.000
alfa -21.025
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn 65536
DISPLAY
sp -346.0
wp 3904.1
rfl 3709.5
rfp 2907.4
rp 162.5
lp -0.6
PLOT
wc 270
sc 0
vs 202
th 14
ai cdc ph
Boc
N
H
Si
Ph
Ph
OH
40
8
7
6
5
4
3
2
1
-0
ppm

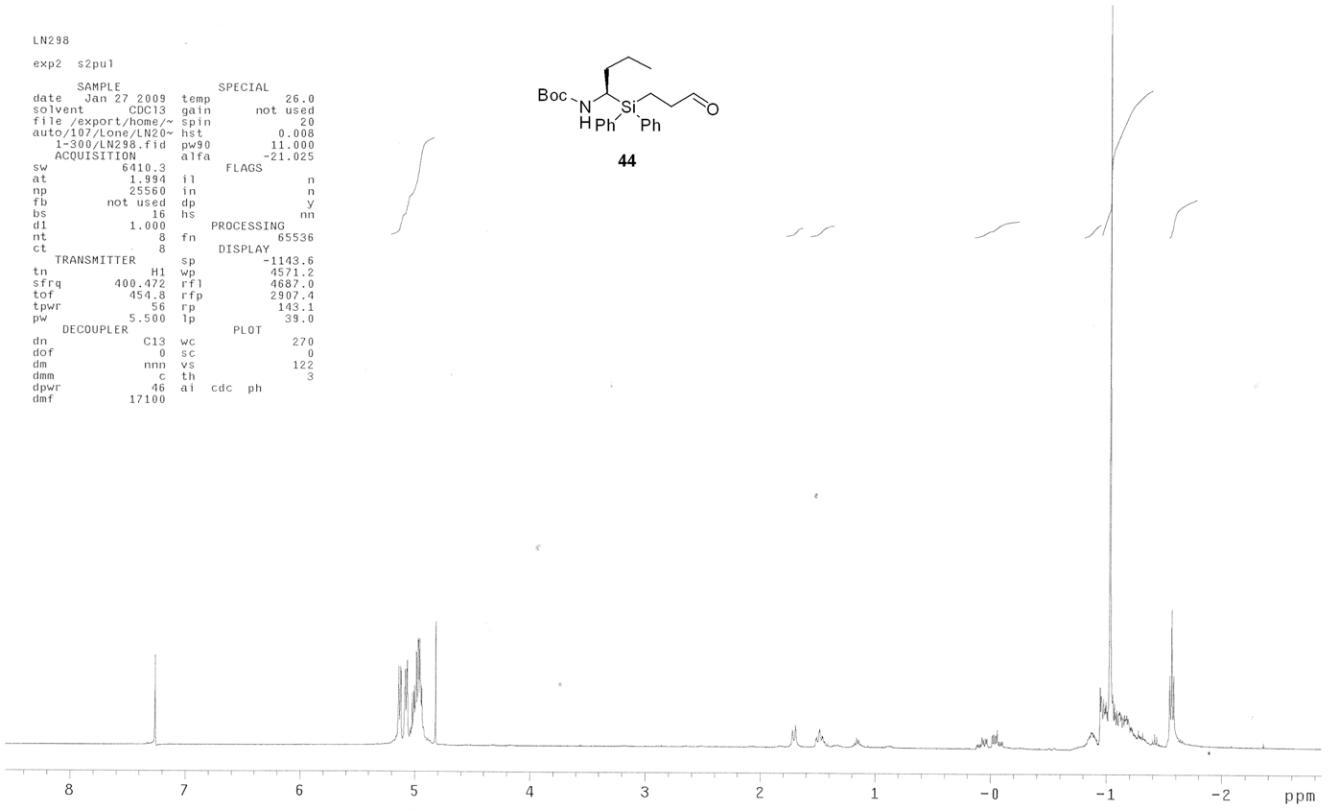

Boc
N
H
Si
Ph
Ph
OH
40
77.473
77.160
76.839
135.680
135.482
133.054
129.871
129.833
128.207
128.146
156.513
79.188
65.373
60.526
37.831
34.388
28.548
26.922
21.182
20.395
14.334
13.930
7.494
160
140
120
100
80
60
40
20
0
ppm

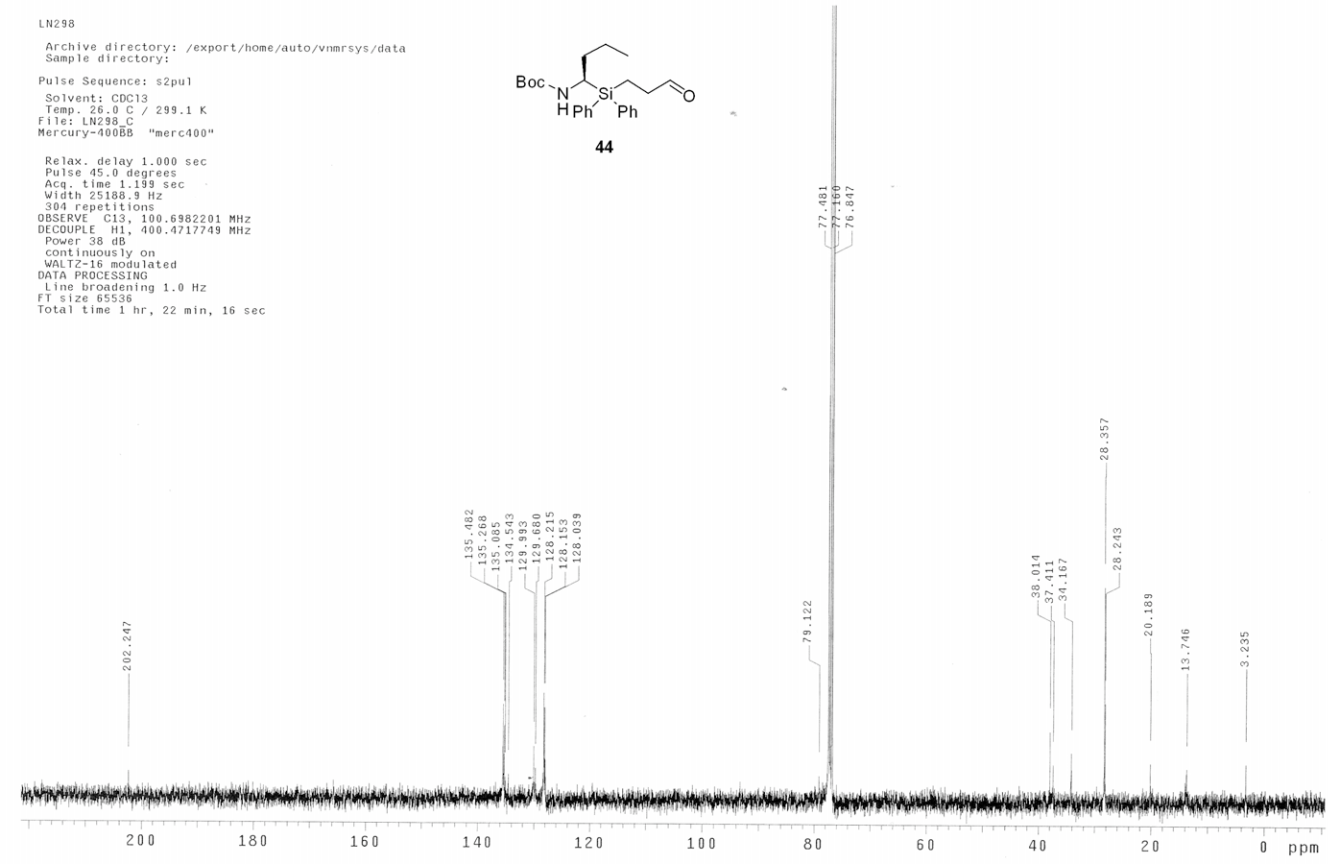

LN304A
Archive directory: /export/home/auto/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 26.0 C / 299.1 K
File: LN304A-solvent
Mercury-400BB "merc400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.994 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 400.4698260 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 25 sec
Boc
N
H
Ph
Si
Ph
O
N
H
O
O
t-Bu
42
8
7
6
5
4
3
2
1
ppm

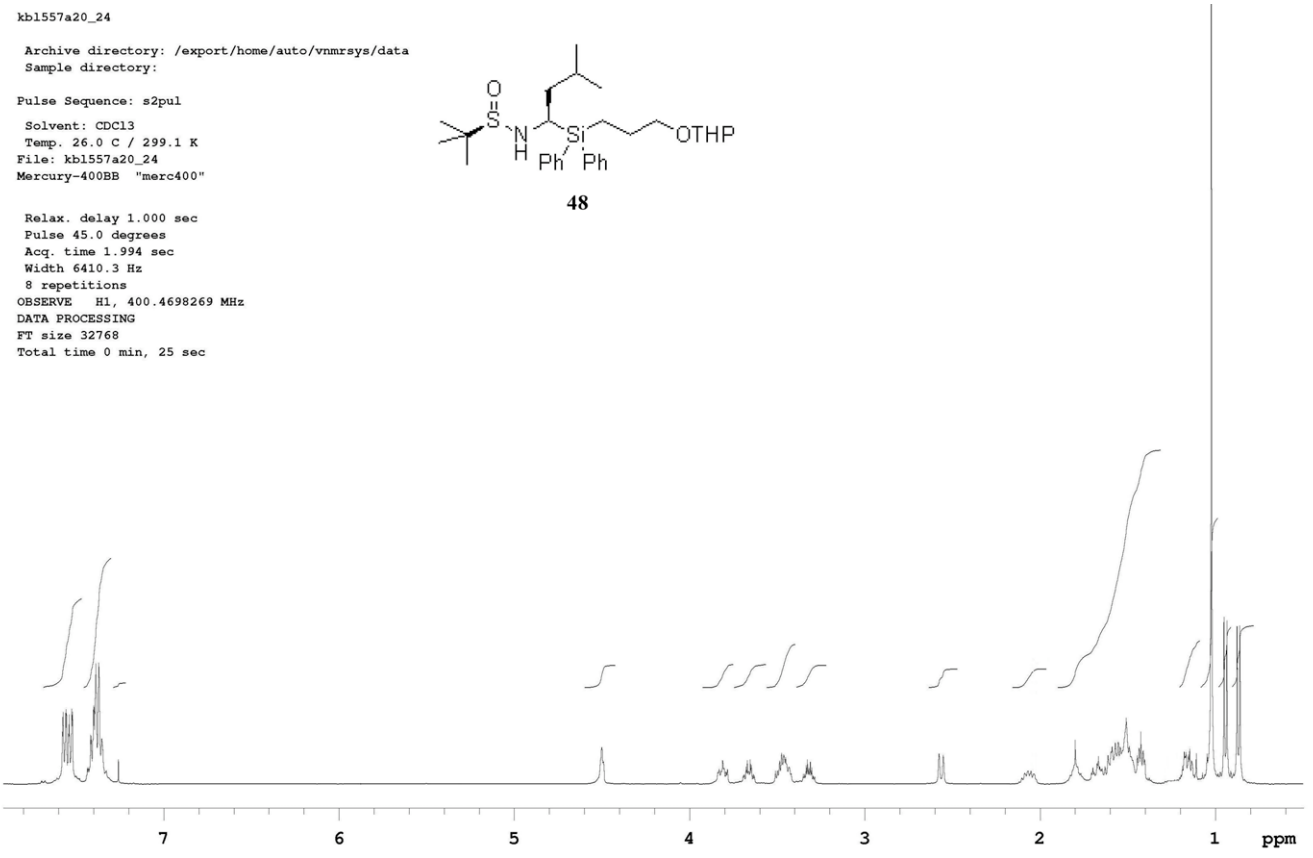

Boc
N
H
Ph
Si
Ph
O
N
H
O
O
t-Bu
42
174.002
169.215
156.459
135.635
135.459
132.825
132.459
130.008
128.283
128.230
82.259
79.191
77.481
77.160
76.839
42.182
37.602
34.213
30.411
28.533
28.159
20.365
13.891
7.426
200
180
160
140
120
100
80
60
40
20
0
ppm


LN292A
Archive directory: /export/home/auto/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 26.0 C / 299.1 K
File: LN292A
Mercury-400BB "merc400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.994 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 400.4698264 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 25 sec
Boc
N
O
Si
Ph
Ph
43
9
8
7
6
5
4
3
2
1
ppm

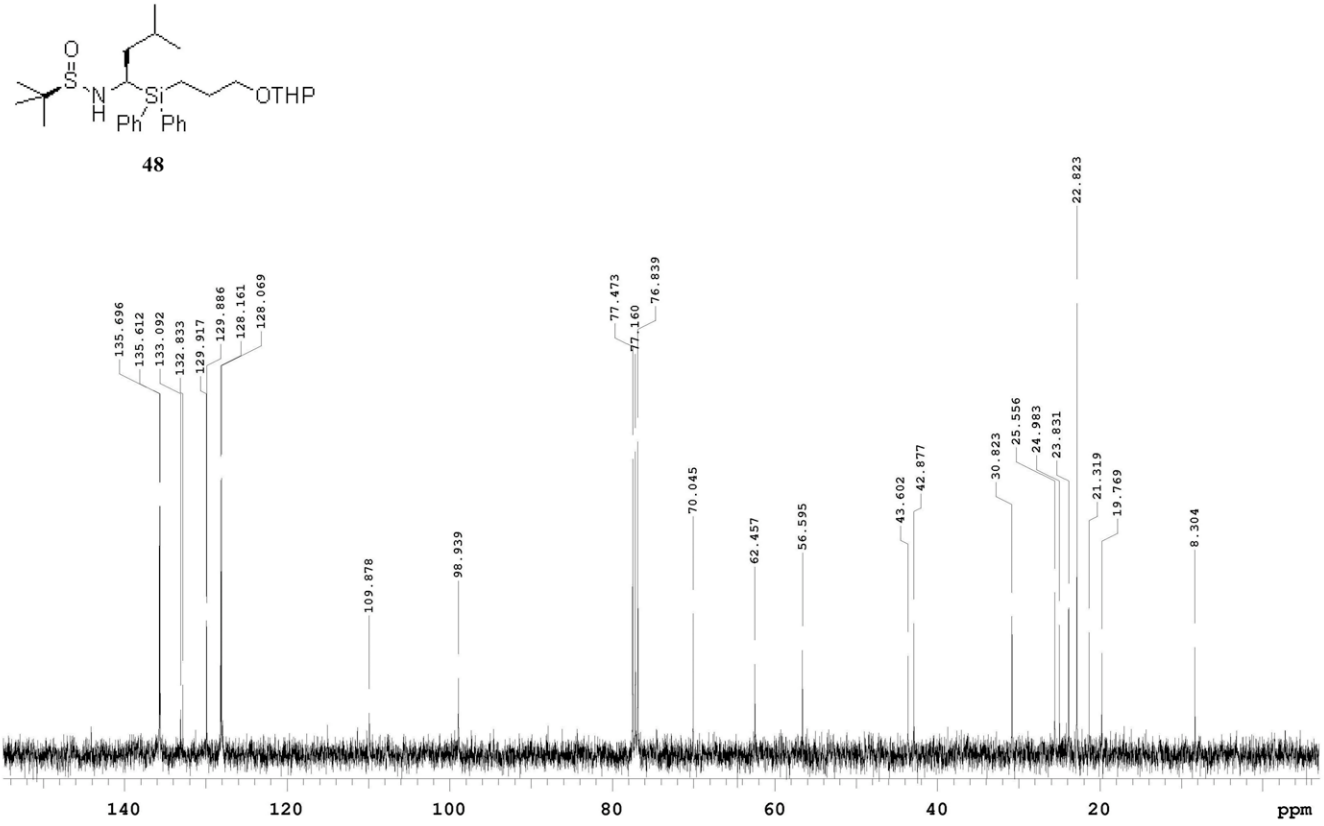

Boc
N
O
Si
Ph
Ph
43
174.864
154.108
135.253
134.734
133.963
132.467
130.451
130.169
128.413
128.321
82.840
77.473
77.358
77.150
76.839
44.075
35.625
34.541
28.106
20.837
13.968
5.563
180
160
140
120
100
80
60
40
20
0
ppm


LN298
exp2 s2pul
SAMPLE
date Jan 27 2009
solvent CDCl3
file /export/home/~
auto/107/Lone/LN20~
1-300/LN298.fid
ACQUISITION
sw 6410.3
at 1.994
np 25560
fb not used
bs 16
d1 1.000
nt 8
ct 8
TRANSMITTER
tn H1
sfrq 400.472
tof 454.8
tpwr 56
pw 5.500
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 46
dmf 17100
SPECIAL
temp 26.0
gain not used
spin 20
hst 0.008
pw90 11.000
alfa -21.025
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn 65536
DISPLAY
sp -1143.6
wp 4571.2
rfl 4687.0
rfp 2907.4
rp 143.1
lp 39.0
PLOT
wc 270
sc 0
vs 122
th 3
al cdc ph
Boc
N
H
Ph
Si
Ph
O
44
8
7
6
5
4
3
2
1
-0
-1
-2
ppm

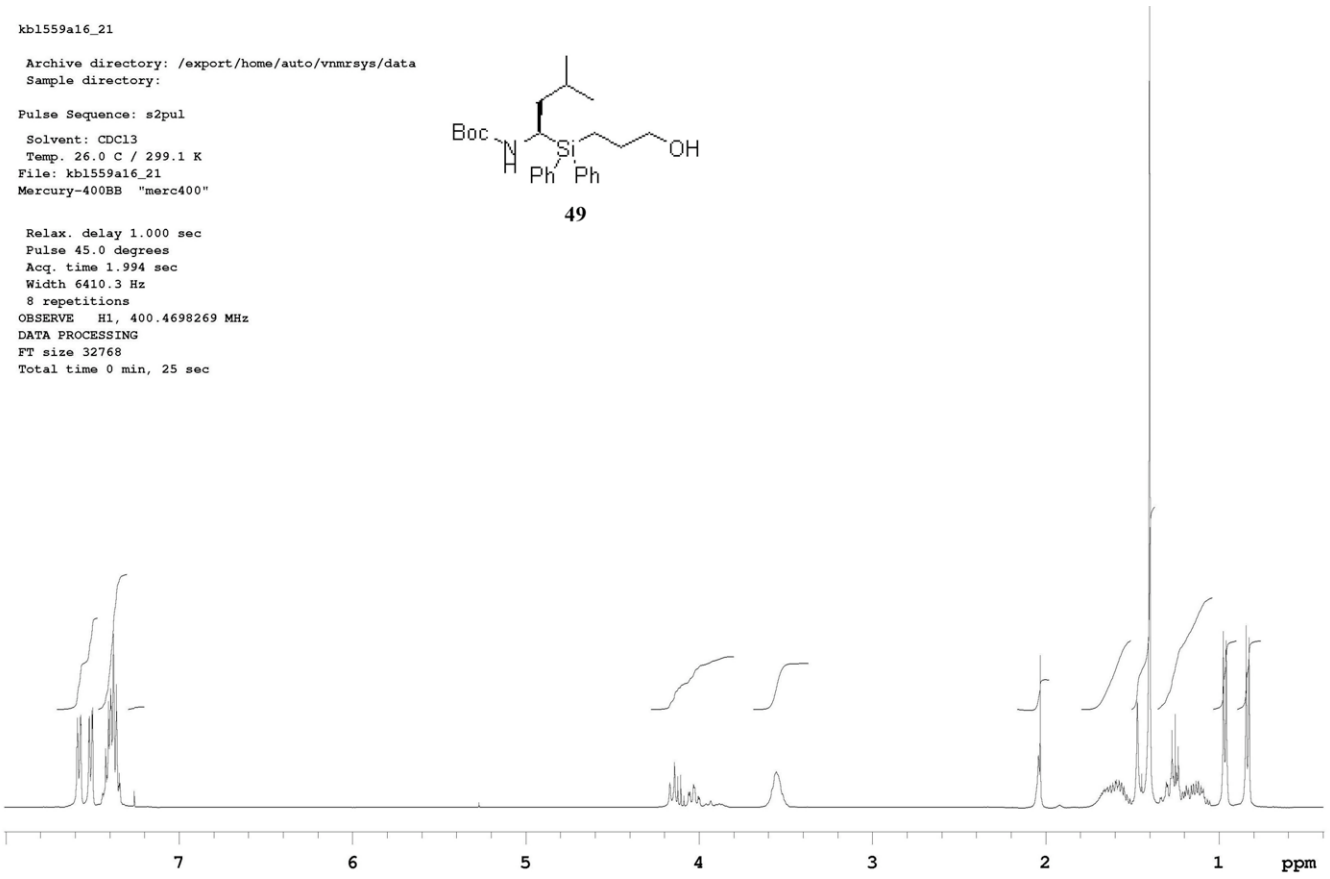

LN298
Archive directory: /export/home/auto/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 26.0 C / 299.1 K
File: LN298_C
Mercury-400BB "merc400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.199 sec
Width 25188.9 Hz
304 repetitions
OBSERVE C13, 100.6982201 MHz
DECOUPLE H1, 400.4717749 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 1 hr, 22 min, 16 sec
Boc
N
H
Ph
Si
Ph
O
44
77.481
77.160
76.847
135.482
135.268
135.085
134.543
129.993
129.680
128.215
128.153
128.039
79.122
38.014
37.411
34.167
28.357
28.243
20.189
13.746
3.235
202.247
200
180
160
140
120
100
80
60
40
20
0
ppm


kbl557a20_24
Archive directory: /export/home/auto/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 26.0 C / 299.1 K
File: kbl557a20_24
Mercury-400BB "merc400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.994 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 400.4698269 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 25 sec
OTHP
Ph
Ph
7
6
5
4
3
2
1
ppm

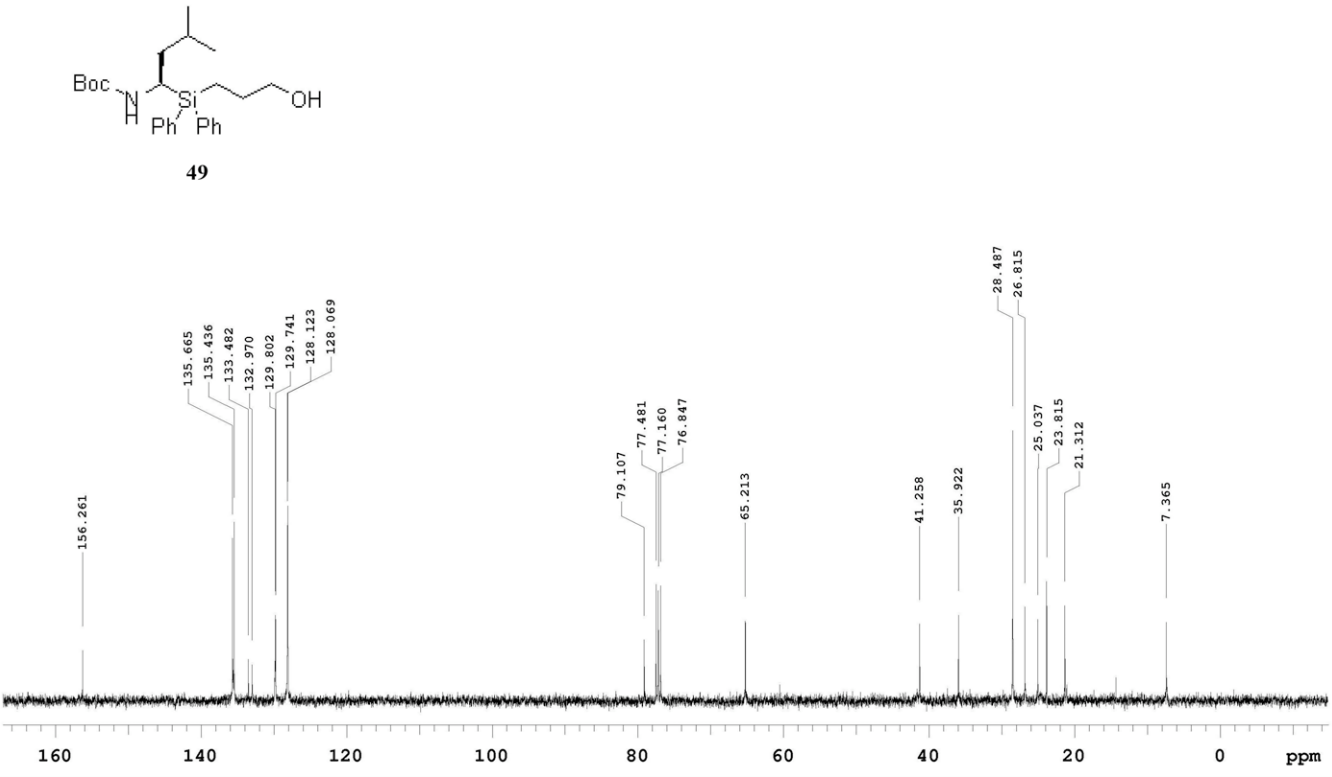
48


OTHP
Ph
Ph
135.696
135.612
133.092
132.833
129.917
129.886
128.161
128.069
77.473
77.160
76.839
70.045
62.457
56.595
43.602
42.877
30.823
25.556
24.983
23.831
22.823
21.319
19.769
109.878
98.939
8.304
140
120
100
80
60
40
20
ppm

48


kbl559a16_21
Archive directory: /export/home/auto/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 26.0 C / 299.1 K
File: kbl559a16_21
Mercury-400BB "merc400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.994 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 400.4698269 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 25 sec
Boc
N
H
Si
Ph
Ph
OH
7
6
5
4
3
2
1
ppm

49


Boc
N
H
Si
Ph
Ph
OH
156.261
135.665
135.436
133.482
132.970
129.802
129.741
128.123
128.069
79.107
77.481
77.160
76.847
65.213
41.258
35.922
28.487
26.815
25.037
23.815
21.312
7.365
160
140
120
100
80
60
40
20
0
ppm

49

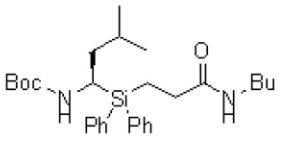
kbl561a17_22_2nd

Archive directory: /export/home/auto/vnmrsys/data
Sample directory:

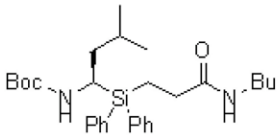
Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 26.0 C / 299.1 K
File: kbl561a17_22_2nd
Mercury-400BB "merc400"

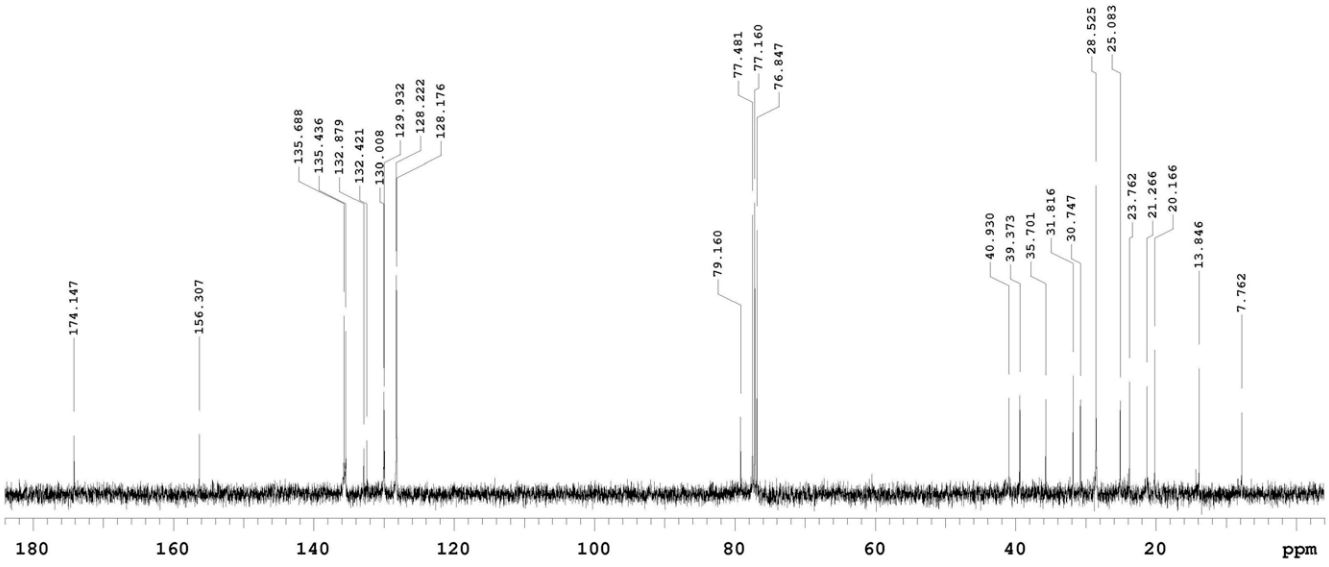
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.994 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 400.4698269 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 25 sec

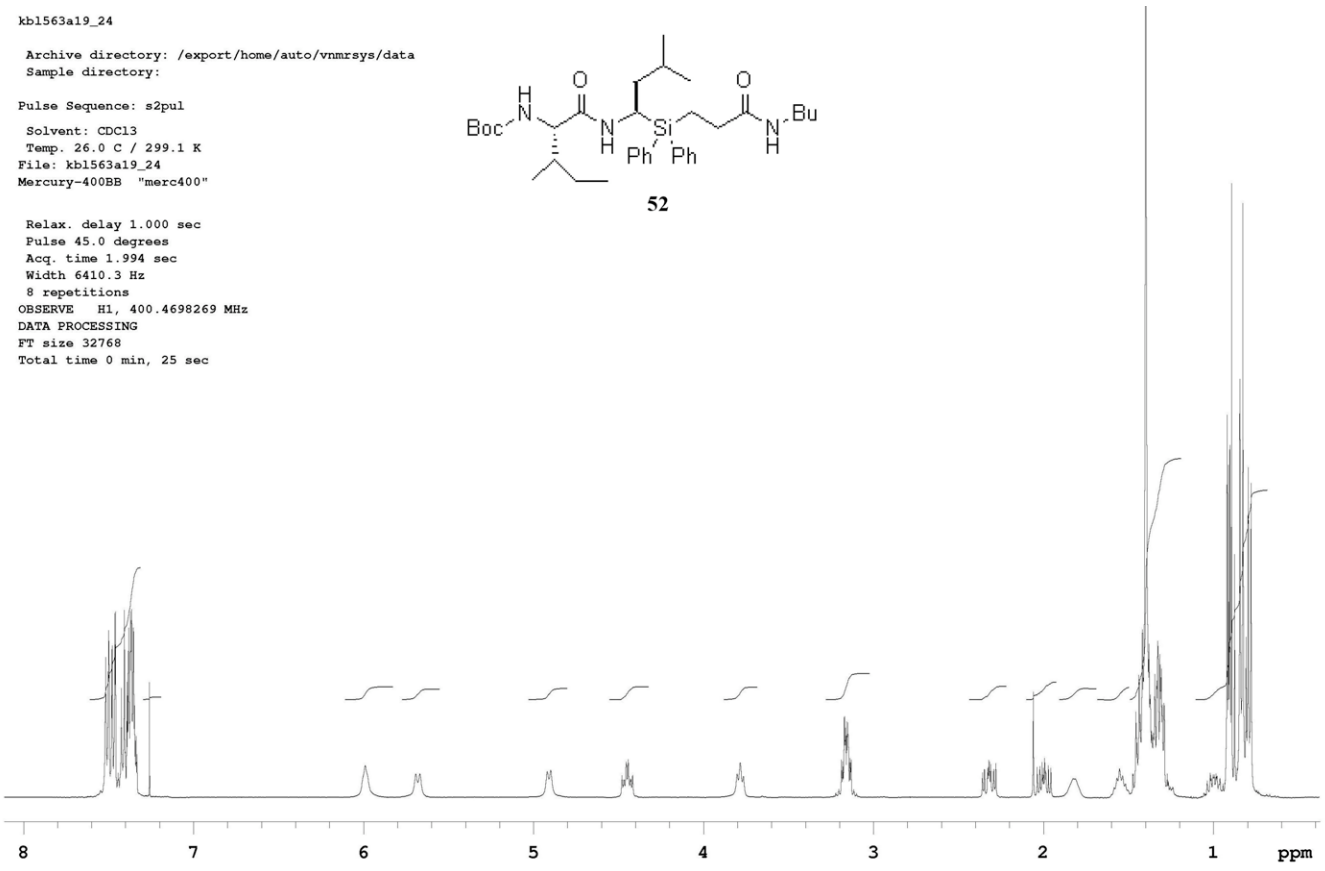


51

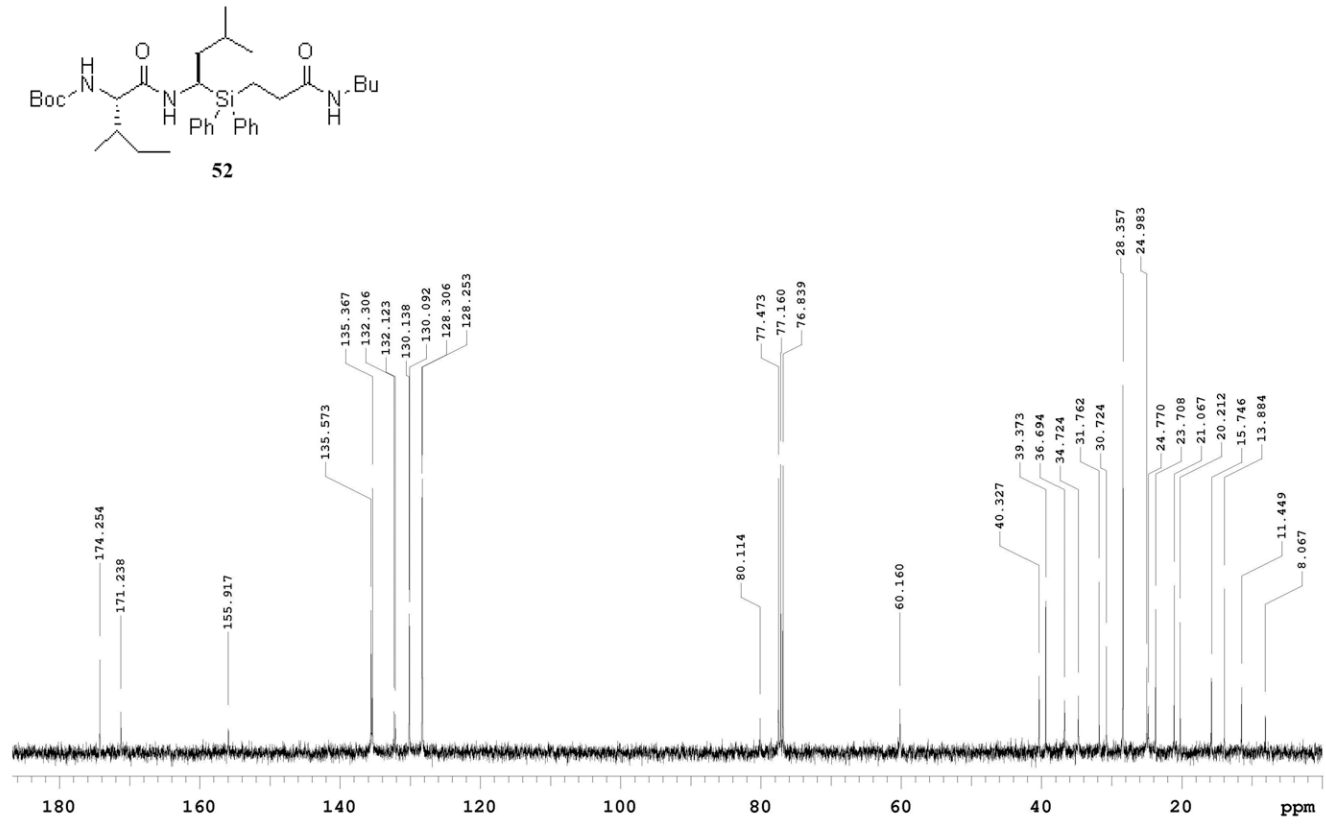


51




kbl563a19_24
Archive directory: /export/home/auto/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 26.0 C / 299.1 K
File: kbl563a19_24
Mercury-400BB "merc400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.994 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 400.4698269 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 25 sec
Boc
Bu
Ph
Ph
8
7
6
5
4
3
2
1
ppm

52


Boc
Bu
Ph
Ph
135.367
132.306
132.123
130.138
130.092
128.306
128.253
135.573
174.254
171.238
155.917
77.473
77.160
76.839
80.114
60.160
28.357
24.983
40.327
39.373
36.694
34.724
31.762
30.724
24.770
23.708
21.067
20.212
15.746
13.884
11.449
8.067
180
160
140
120
100
80
60
40
20
ppm

52

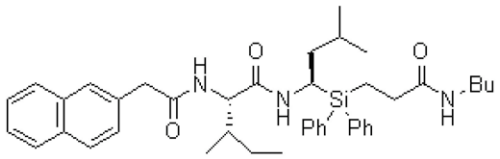
kb1565a7_8

Archive directory: /export/home/auto/vnmrsys/data
Sample directory:

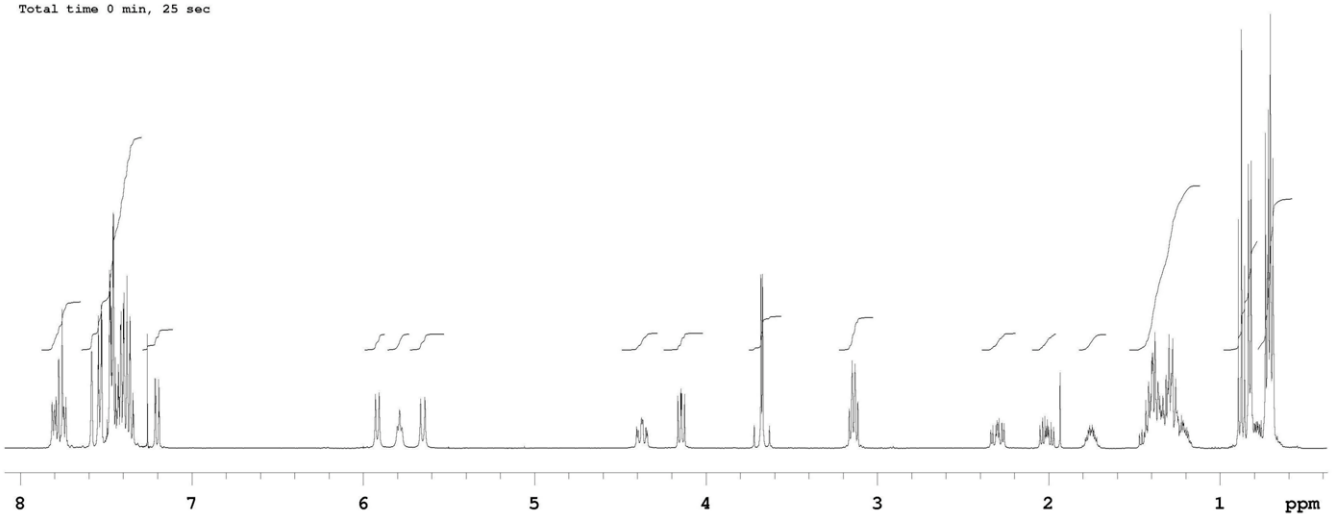
Pulse Sequence: s2pul

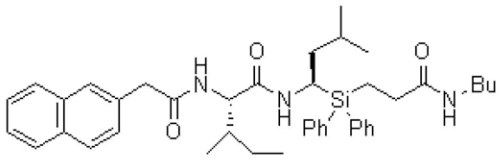
Solvent: CDCl3
Temp. 26.0 C / 299.1 K
File: kb1565a7_8
Mercury-400BB "merc400"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.994 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 400.4698269 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 25 sec

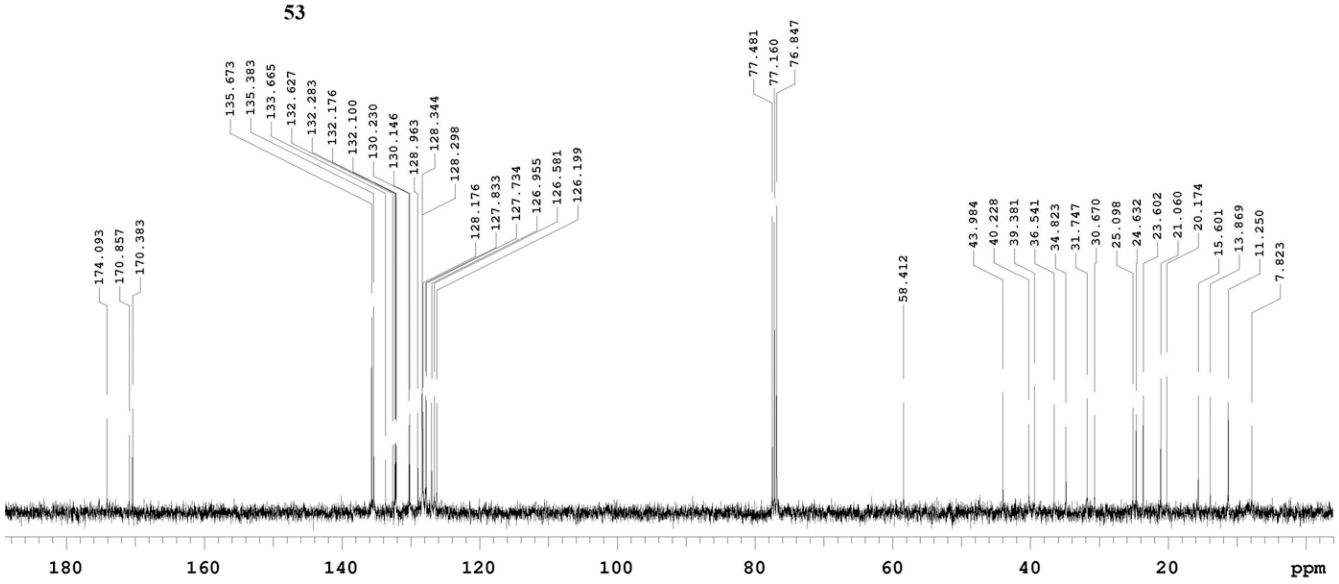


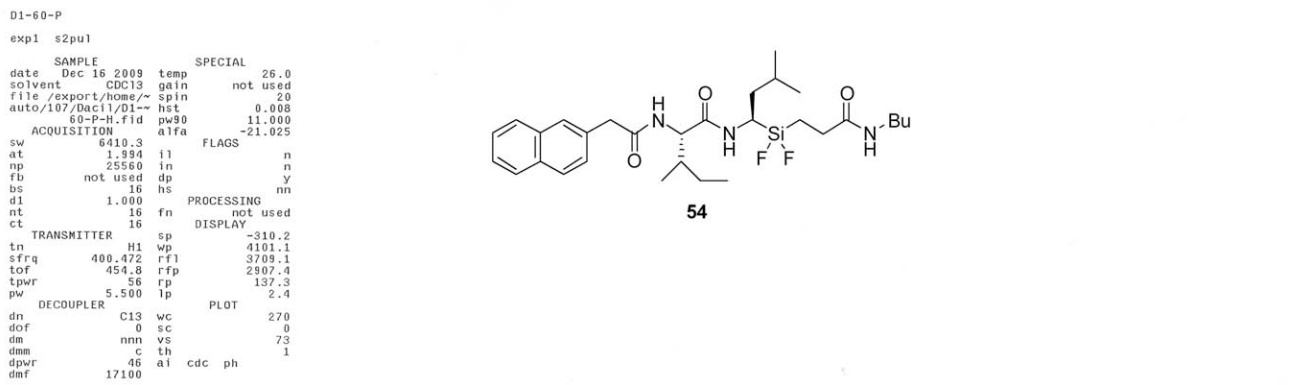
53

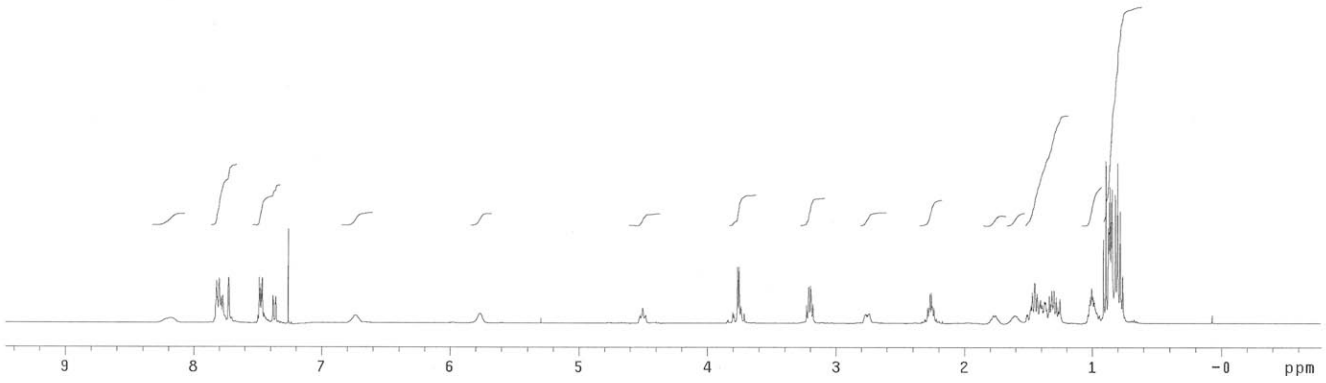


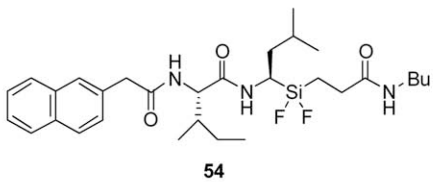


53

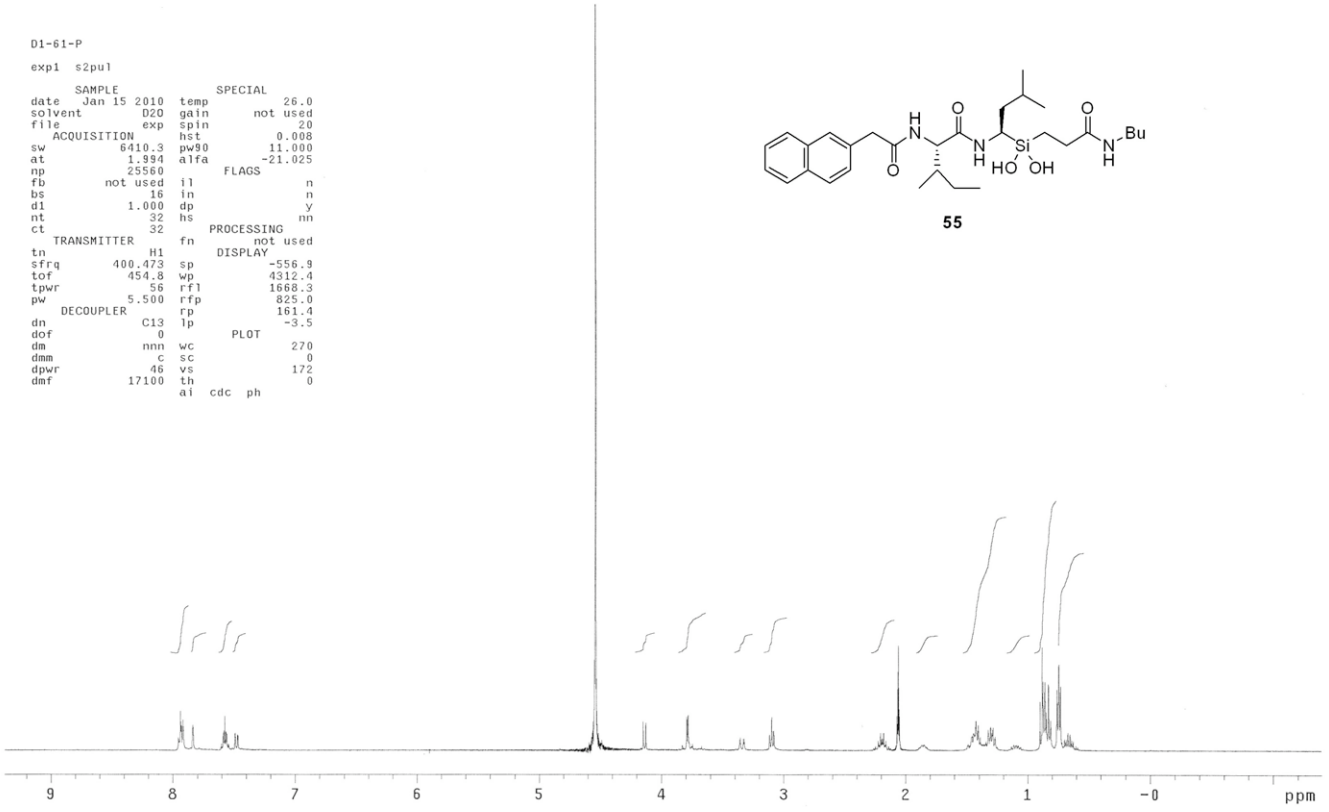


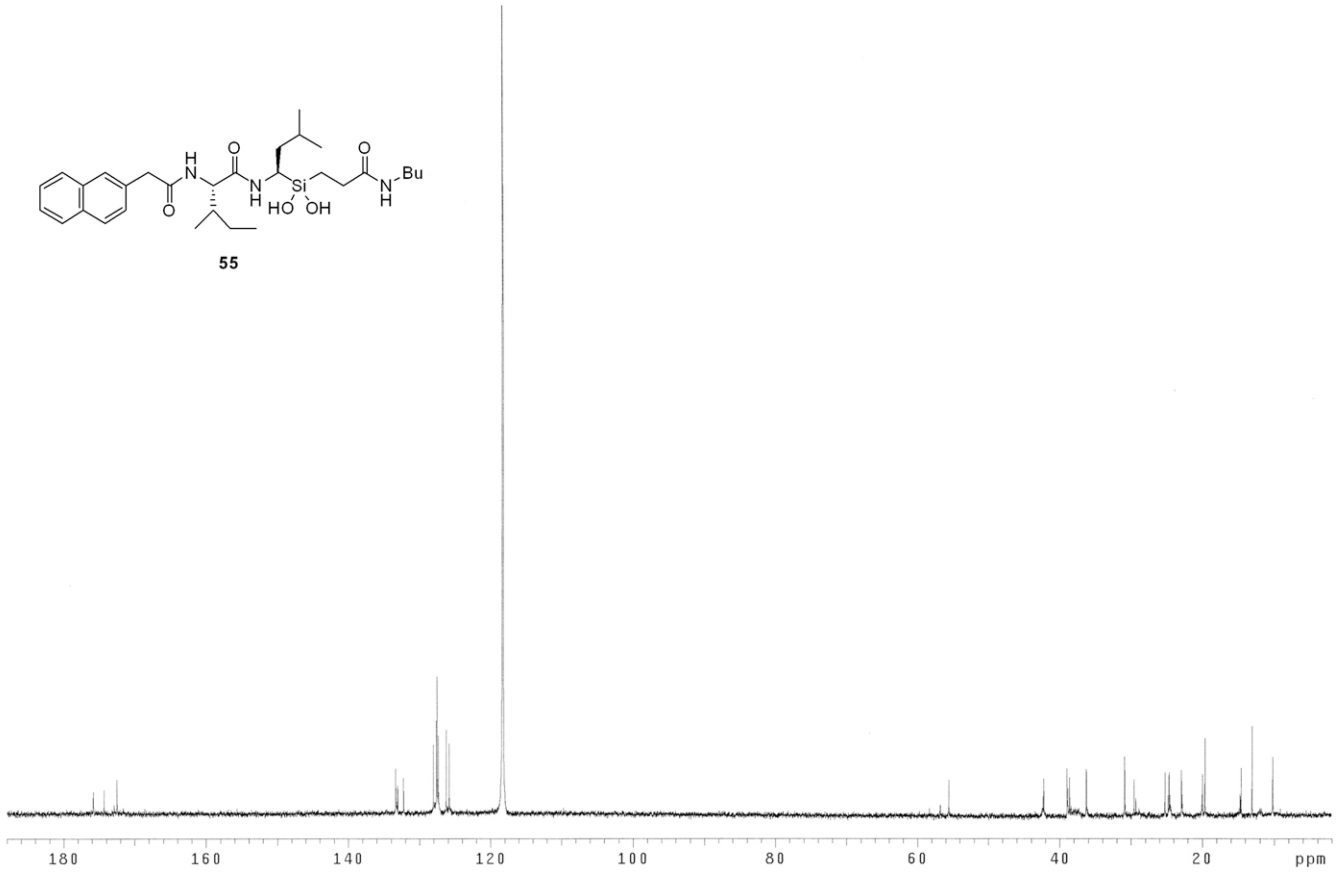

D1-60-P
exp1 s2pul
SAMPLE
date Dec 16 2009
solvent CDCl3
file /export/home/~
auto/107/Dac1/D1~
60-P-H.fid
ACQUISITION
sw 6410.3
at 1.994
np 25560
fb not used
bs 16
d1 1.000
nt 16
ct 16
TRANSMITTER
tn H1
sfrq 400.472
tof 454.8
tpwr 56
pw 5.500
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 46
dmf 17100
SPECIAL
temp 26.0
gain not used
spin 20
hst 0.008
pw90 11.000
alfa -21.025
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn not used
DISPLAY
sp -310.2
wp 4101.1
rfl 3709.1
rfp 2907.4
rp 137.3
lp 2.4
PLOT
wc 270
sc 0
vs 73
th 1
ai cdc ph
54


9
8
7
6
5
4
3
2
1
-0
ppm


54

77.481
77.366
77.160
76.847
174.971
173.857
171.864
133.657
132.457
132.021
128.970
128.879
128.367
128.288
128.207
127.840
127.795
127.604
127.344
127.077
126.993
126.558
126.260
126.199
55.755
43.678
43.584
39.370
39.686
38.968
38.678
38.465
38.411
38.197
36.991
31.686
30.701
29.831
25.907
25.044
23.716
21.006
20.174
20.037
15.266
13.884
12.120
11.914
11.716
11.098
10.953
180
160
140
120
100
80
60
40
20
0 ppm

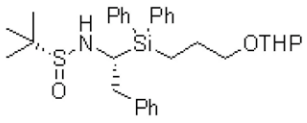

D1-61-P
exp1 s2pul
SAMPLE
date Jan 15 2010
solvent D2O
file exp
ACQUISITION
sw 6410.3
at 1.994
np 25560
fb not used
bs 16
d1 1.000
nt 32
ct 32
TRANSMITTER
tn H1
sfrq 400.473
tof 454.8
tpwr 56
pw 5.500
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 46
dmf 17100
SPECIAL
temp 26.0
gain not used
spin 20
hst 0.008
pw90 11.000
alfa -21.025
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn not used
DISPLAY
sp -556.9
wp 4312.4
rfl 1668.3
rfp 825.0
rp 161.4
lp -3.5
PLOT
wc 270
sc 0
vs 172
th 0
ai cdc ph
55
9
8
7
6
5
4
3
2
1
-0
ppm


55
180
160
140
120
100
80
60
40
20
ppm

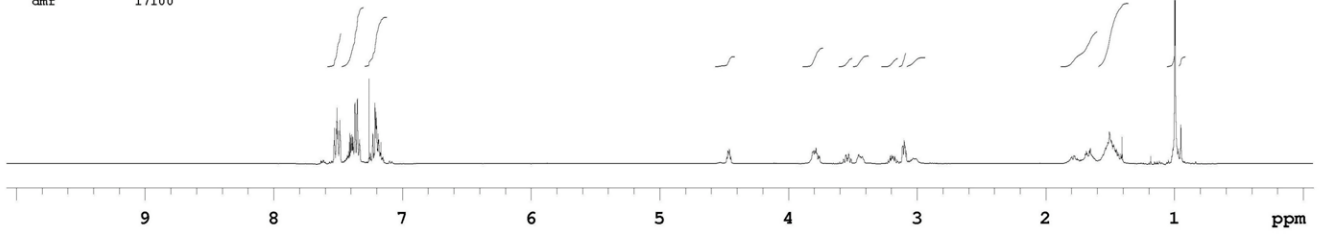
tm-289_19-20

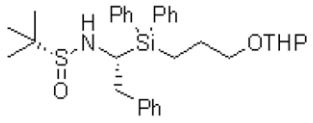
expl s2pul

SAMPLE		SPECIAL	
date	Jul 13 2009	temp	26.0
solvent	CDCl3	gain	not used
file	/export/home/~	spin	20
auto/107/Tina/200-~		hst	0.008
299/tm-289_19-20.f~		pw90	11.000
id		alfa	-21.025
ACQUISITION		**FLAGS**	
sw	6410.3	il	n
at	1.994	in	n
np	25560	dp	y
fb	not used	hs	nn
bs	16	**PROCESSING**	
d1	1.000	fn	not used
nt	8	**DISPLAY**	
ct	8	sp	-31.9
TRANSMITTER		wp	4067.1
tn	H1	rfl	802.3
sfrq	400.472	rfp	0
tof	454.8	rp	161.1
tpwr	56	lp	7.8
pw	5.500	**PLOT**	
DECOUPLER		wc	270
dn	C13	sc	0
dof	0	vs	120
dm	nnn	th	20
dmm	c	nm cdc ph	
dpwr	46		
dmf	17100		

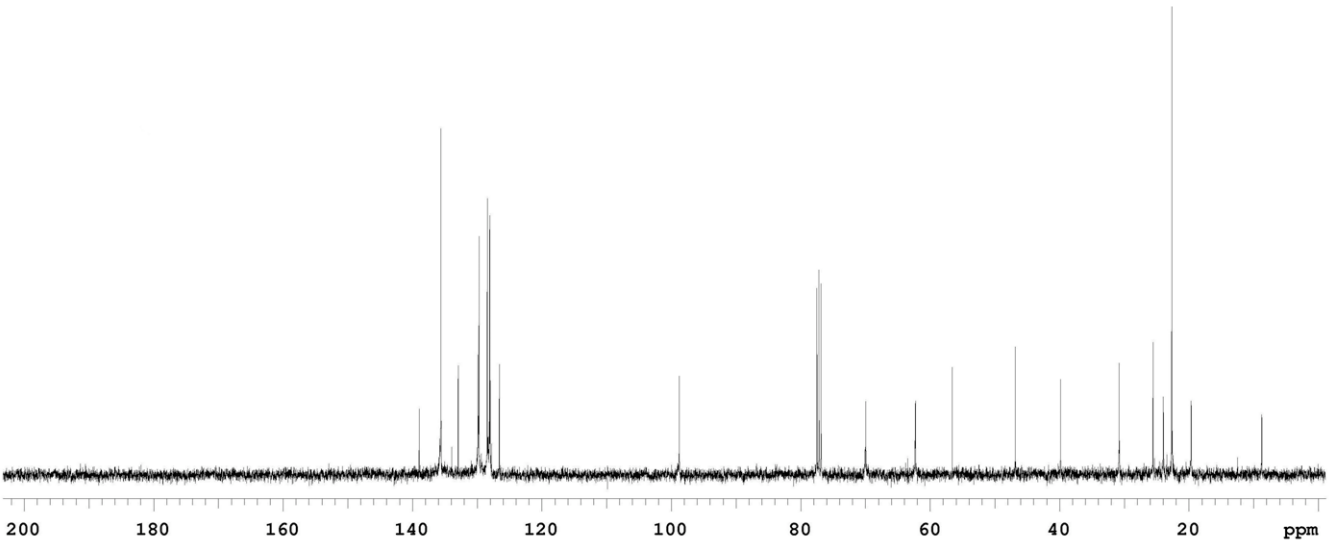


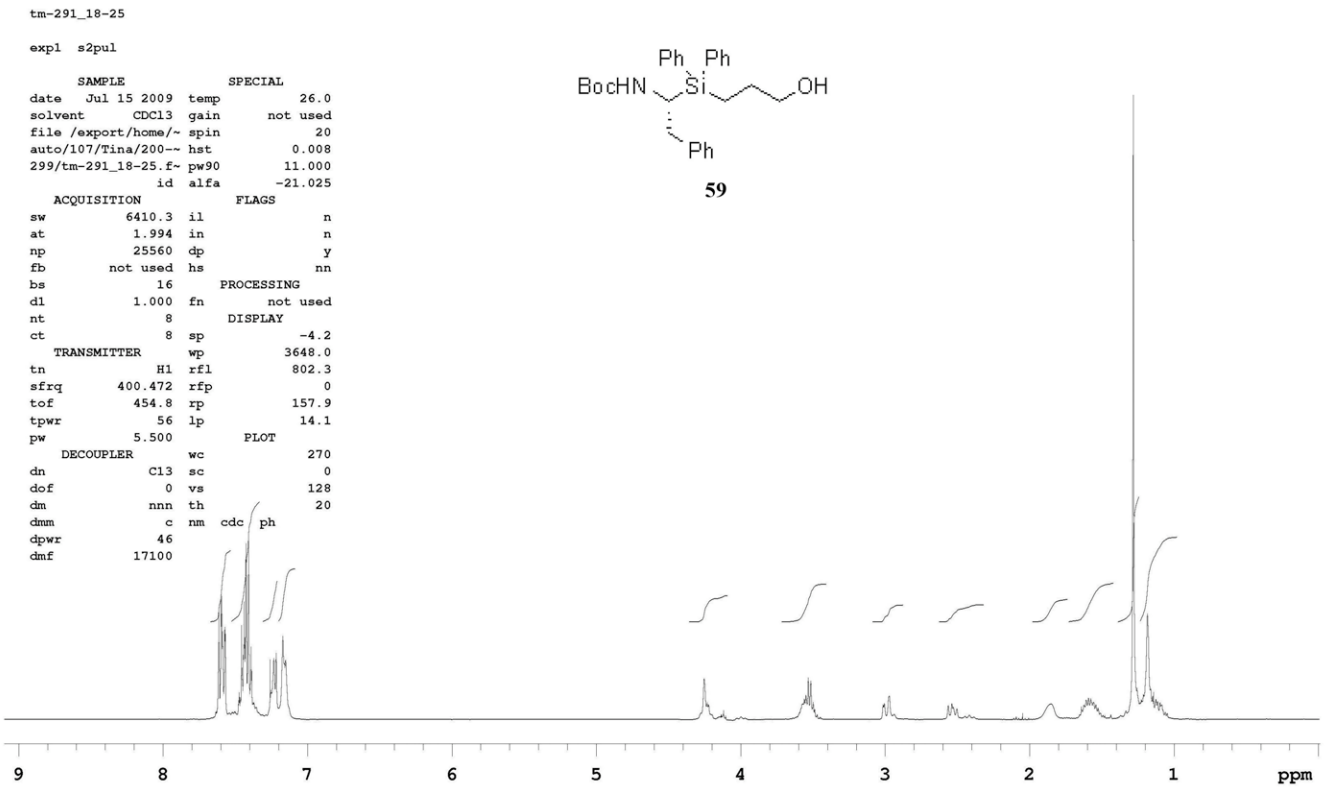
58

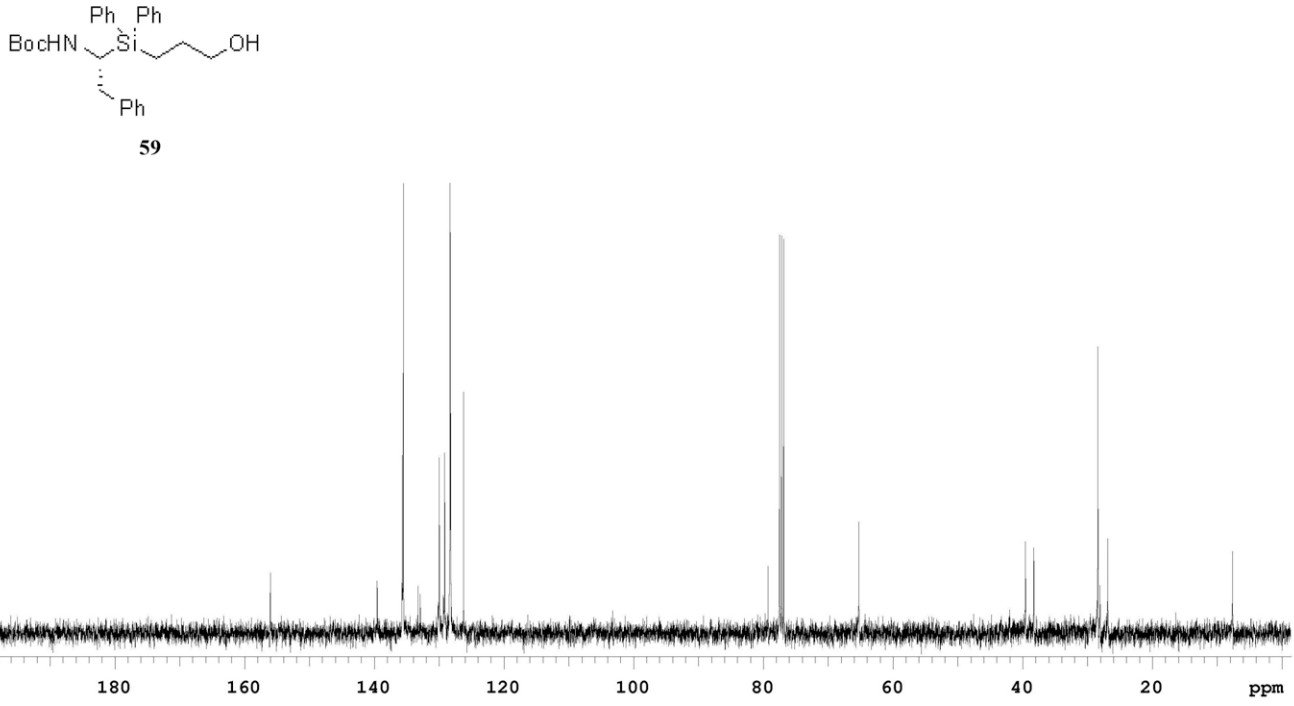


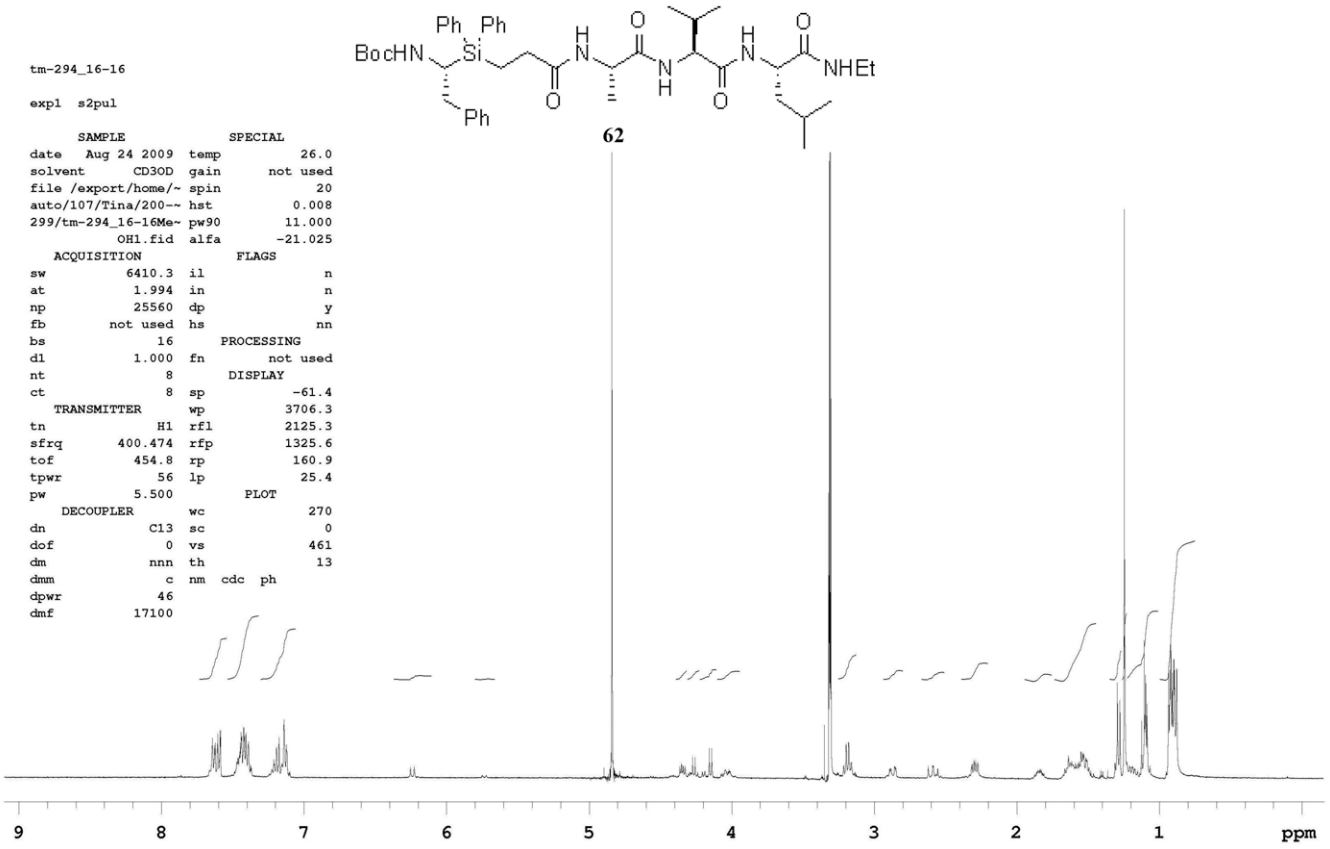


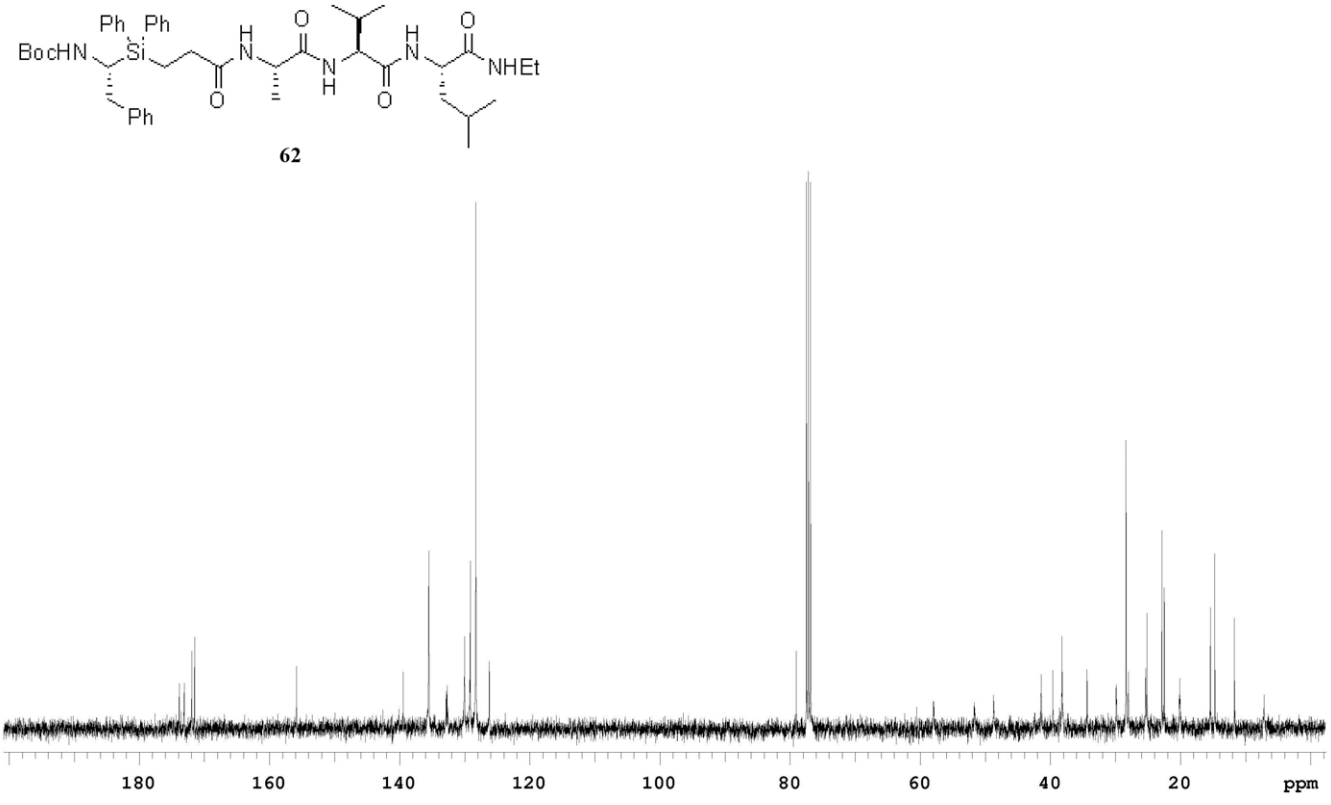
58

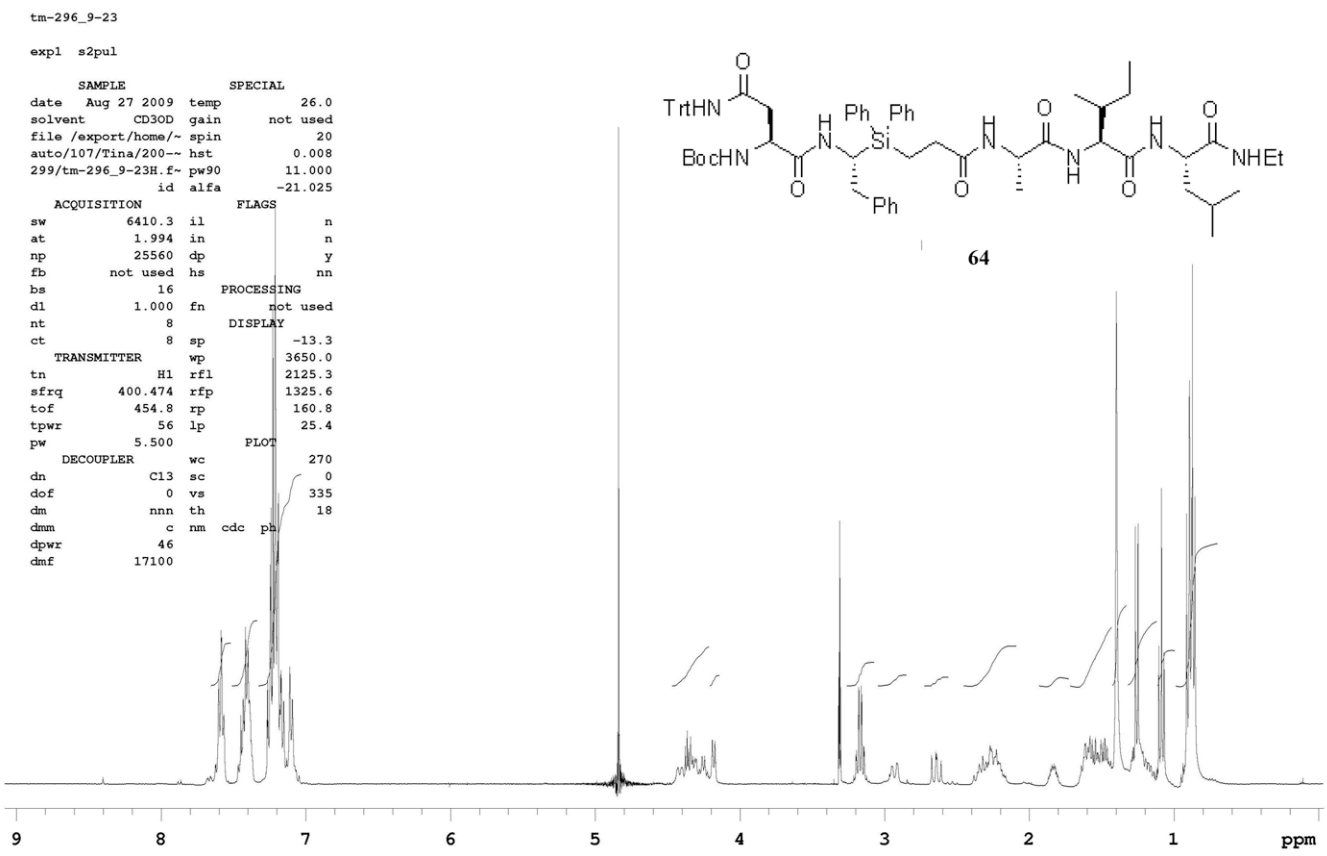


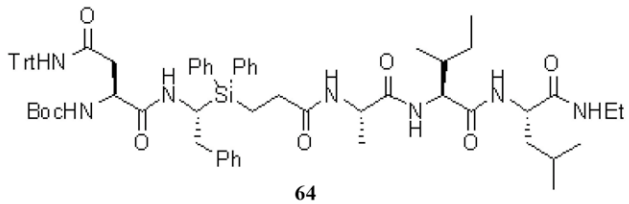

tm-291_18-25
expl s2pul
SAMPLE
date Jul 15 2009
solvent CDCl3
file /export/home/~
auto/107/Tina/200-~
299/tm-291_18-25.f~
id
ACQUISITION
sw 6410.3
at 1.994
np 25560
fb not used
bs 16
dl 1.000
nt 8
ct 8
TRANSMITTER
tn H1
sfrq 400.472
tof 454.8
tpwr 56
pw 5.500
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 46
dmf 17100
SPECIAL
temp 26.0
gain not used
spin 20
hst 0.008
pw90 11.000
alfa -21.025
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn not used
DISPLAY
sp -4.2
wp 3648.0
rfl 802.3
rfp 0
rp 157.9
lp 14.1
PLOT
wc 270
sc 0
vs 128
th 20
nm cdc ph
Ph Ph
BocHN Si OH
Ph
59
9 8 7 6 5 4 3 2 1 ppm


Ph Ph
BocHN Si OH
Ph
59
180 160 140 120 100 80 60 40 20 ppm


tm-294_16-16
expl s2pul
SAMPLE
date Aug 24 2009
solvent CD3OD
file /export/home/~
auto/107/Tina/200~~
299/tm-294_16-16Me~
OH1.fid
ACQUISITION
sw 6410.3
at 1.994
np 25560
fb not used
bs 16
d1 1.000
nt 8
ct 8
TRANSMITTER
tn H1
sfrq 400.474
tof 454.8
tpwr 56
pw 5.500
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 46
dmf 17100
SPECIAL
temp 26.0
gain not used
spin 20
hst 0.008
pw90 11.000
alfa -21.025
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn not used
DISPLAY
sp -61.4
wp 3706.3
rfl 2125.3
rfp 1325.6
rp 160.9
lp 25.4
PLOT
wc 270
sc 0
vs 461
th 13
nm cdc ph
BocHN
Ph
Ph
Si
Ph
NHEt
62
9
8
7
6
5
4
3
2
1
ppm


BocHN
Ph
Ph
Si
Ph
NHEt
62
180
160
140
120
100
80
60
40
20
ppm


tm-296_9-23
expl s2pul
SAMPLE
date Aug 27 2009
solvent CD3OD
file /export/home/~
auto/107/Tina/200-~
299/tm-296_9-23H.f~
id
ACQUISITION
sw 6410.3
at 1.994
np 25560
fb not used
bs 16
d1 1.000
nt 8
ct 8
TRANSMITTER
tn H1
sfrq 400.474
tof 454.8
tpwr 56
pw 5.500
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 46
dmf 17100
SPECIAL
temp 26.0
gain not used
spin 20
hst 0.008
pw90 11.000
alfa -21.025
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn not used
DISPLAY
sp -13.3
wp 3650.0
rfl 2125.3
rfp 1325.6
rp 160.8
lp 25.4
PLOT
wc 270
sc 0
vs 335
th 18
nm cdc ph
TrtHN
BocHN
Ph
Ph
Si
Ph
NHEt
64
9
8
7
6
5
4
3
2
1
ppm


TrtHN
BocHN
Ph
Ph
Si
Ph
NHEt

64

200
180
160
140
120
100
80
60
40
20
ppm