Kinetic Resolution of Binols and Biphenols via Dehydrogenative Coupling with Hydrosilanes Catalyzed by Chiral FLPs

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Supporting Information

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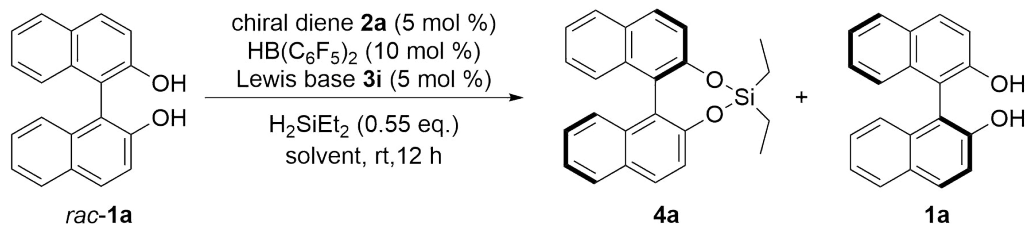
General consideration: All air-sensitive compounds were handled under an atmosphere of argon or in a nitrogen-filled glovebox. ^{1}H NMR, ^{13}C {^{1}H} NMR, ^{19}F NMR and ^{31}P NMR spectra were recorded on Bruker AV 300, 400 or 500 at ambient temperature with $CDCl_3$ as solvent and TMS as internal standard. Chemical shifts (δ) were given in ppm, referenced to the residual proton resonance of TMS (0) or to the carbon resonance of the $CDCl_3$ (77.23). Peak shapes involved s = singlet, d = doublet, t = triplet, q = quartet, dd = double doublet, td = triple doublet, ddd = double double doublet, m = multiplet. Coupling constants (*J*) were given in Hertz (Hz). IR spectrums were recorded on Thermo-Nicolet-6700 spectrometer. High resolution mass spectra (HRMS) were recorded on Thermo Fisher Exactive orbitrap and Solarix 9.4T mass instrument (ESI or APCI). Flash column chromatography was performed on silica gel (200-300 mesh). All solvents were purified by conventional methods, distilled before use. Commercially available reagents were used without further purification. Chiral dienes **2** were prepared according to reported methods.

Z. Cao and H. Du, *Org. Lett*. 2010, **12**, 2602–2605.

Optimization of Reaction Conditions

Table S1. Screening of the solvents[a]



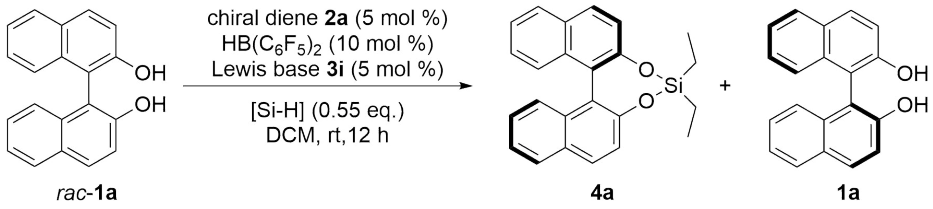
rac-**1a** **4a** **1a**

entry	solvent	Conv. (%)[b]	**4a** ee (%)[c]	**1a** ee (%)[c]	*s*
1	Toluene	36	58	26	5
2	C_6H_5F	49	72	55	11

3	C_6H_5Cl	40	73	45	10
4	*p*-Xylene	49	34	31	3
5	Hexane	<10	-	-	-
6	DCE	36	80	33	8
7	DCM	41	78	52	14

[a]All reactions were carried out with *rac*-**1a** (0.1 mmol), H_2SiEt_2 (0.055 mol), chiral diene **2a** (5 mol %), $HB(C_6F_5)_2$ (10 mol %), Lewis base **3i** (5 mol %), in solvent (1.0 mL) at 25 °C for 12 h, unless otherwise noted. [b]Determined by crude ^{1}H NMR. [c]The ee value was determined by chiral HPLC.

Table S2. Screening of the silanes[a]

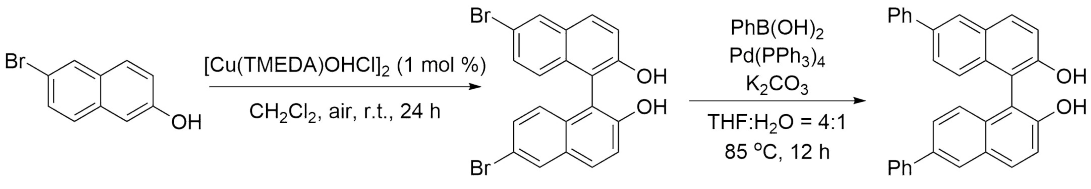


rac-**1a** **4a** **1a**

Entry	silanes	Conv. (%)[b]	**4a** ee (%)[c]	**1a** ee (%)[c]	*s*
1	$H_2SiMePh$	49	77	52	13
2	$HSiMe_2Ph$	48	3	3	1
3	$H_2Si^tBu_2$	<5	-	-	-
4	H_2SiEt_2	41	78	52	14

[a] All reactions were carried out with *rac*-**1a** (0.1 mmol), [Si-H] (0.055 mol), chiral diene **2a** (5 mol %), $HB(C_6F_5)_2$ (10 mol %), Lewis base **3i** (5 mol %), in DCM (1.0 mL) at 25 ºC for 12 h, unless otherwise noted. [b] Determined by crude ^{1}H NMR. [c] The ee value was determined by chiral HPLC.

Representative procedure for the synthesis of diphenols (1a)

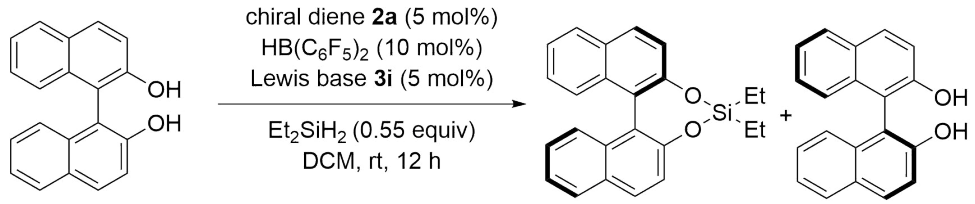


6-Bromonaphthalen-2-ol (7.4 g, 33.5 mmol) was dissolved in CH_2Cl_2 (220 mL) and

$[Cu(TMEDA)OHCl)]_2$ (158 mg, 0.34 mmol) was added. The reaction was stirred for 24 h at room temperature open to air. The reaction mixture was filtered through a short silica plug eluting with CH_2Cl_2 (50 mL). Evaporation of the solvent gave 6,6'-dibromo-[1,1'-binaphthalene]-2,2'-diol (6.8 g, 91%) as an off-white solid.

A flame-dried 50 mL round-bottom flask with magnetic stirring bar and reflux condenser was charged with 6,6'-dibromo-[1,1'-binaphthalene]-2,2'-diol (0.44 g, 1 mmol), boronic acid (0.27 g, 2.2 mmol) and K_2CO_3 (0.41 mg, 3 mmol) and purged with N_2. Then, it was dissolved with THF (15 mL) and a solution of $Pd(PPh_3)_4$ (0.06 g, 0.05 mmol) in THF (5 mL) was added, followed by water (5 mL). The mixture was vigorously stirred at 85 ^oC for 12 h, and then it was cooled to room temperature and extracted with EA (30 mL x 3). The combined organic layers were dried over Na_2SO_4 and concentrated under reduced pressure. The crude product was purified by column chromatography (petroleum ether/ethyl acetate = 3:1 to 1:1) to give compound 1c (0.34 g, 75% yield) as a white solid.

S. Fang, J.-P. Tan, J. Pan, H. Zhang, Y. Chen, X. Ren and T. Wang, *Angew. Chem., Int. Ed.*, 2021, **60**, 14921–14930.

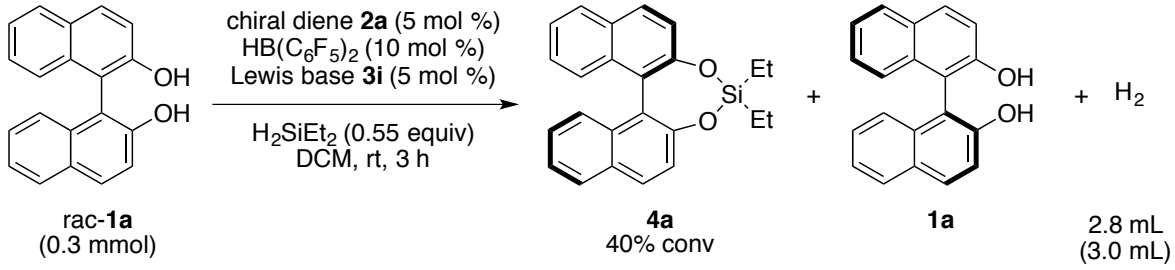


rac-**1a** **4a** **1a**

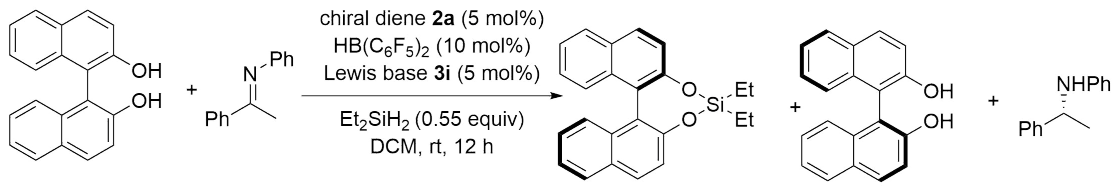
Representative procedure for hydrosilylation of diphenols (*rac*-1a): To a Schlenk tube, were added $HB(C_6F_5)_2$ (10.4 mg, 0.03 mmol), chiral diene **2a** (10.2 mg, 0.015 mmol), and dry dichloromethane (3.0 mL) in a nitrogen atmosphere glovebox. The resulting mixture was stirred

at room temperature for 10 min followed by the addition of Lewis base **3i** (8.7 mg, 0.015 mmol) and *rac*-**1a** (85.9 mg, 0.30 mmol) and stirring. To the resulting solution, Et_2SiH_2 (14.6 mg, 0.165 mmol) was added. After the Schlenk was sealed, the reaction mixture was stirred at room temperature for 12 h. The resulting mixture was directly poured onto silica gel and purified by flash chromatography on silica gel using a mixture of petroleum ether/ethyl acetate (20/1-5/1) to afford **4a** as a colorless oil (36.0 mg, 42% yield, 76% ee) and **1a** as a white solid (48.1 mg, 56% yield, 59% ee).

The H_2 quantification experiment



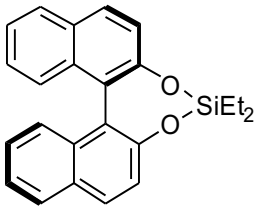
To a Schlenk tube, were added $HB(C_6F_5)_2$ (10.4 mg, 0.03 mmol), chiral diene **2a** (10.2 mg, 0.015 mmol), and dry dichloromethane (3.0 mL) in a nitrogen atmosphere glovebox. The resulting mixture was stirred at room temperature for 10 min followed by the addition of Lewis base **3i** (8.7 mg, 0.015 mmol) and *rac*-**1a** (85.9 mg, 0.30 mmol) and stirring. To the resulting solution, Et_2SiH_2 (14.6 mg, 0.165 mmol) was added. The hydrogen generated was collected by drainage and the reaction mixture was stirred at room temperature for 3 h. After completion of the reaction, 2.8 mL of hydrogen was collected. The conversion of the product was 40% by crude ^{1}H NMR, and the volume of hydrogen at 25 ºC was calculated to be 3.0 mL. The above experimental results demonstrated that hydrogen could be obtained with nearly quantitative yields.



rac-**1a** **5** **4a** **1a** **6**

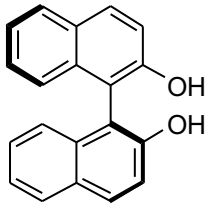
Representative procedure for applications of the dehydrogenative coupling reactions: To a Schlenk tube, were added $HB(C_6F_5)_2$ (10.4 mg, 0.03 mmol), chiral diene **2a** (10.2 mg, 0.015 mmol), and dry dichloromethane (3.0 mL) in a nitrogen atmosphere glovebox. The resulting mixture was stirred at room temperature for 10 min followed by the addition of Lewis base **3i** (8.7 mg, 0.015 mmol) and *rac*-**1a** (85.9 mg, 0.30 mmol,) and imide **5** (58.6 mg, 0.30 mmol) and stirring. To the resulting solution, Et_2SiH_2 (14.6 mg, 0.165 mmol) was added. After the Schlenk was sealed, the reaction mixture was stirred at room temperature for 12 h. The resulting mixture was directly poured onto silica gel and purified by flash chromatography on silica gel using a mixture of petroleum ether/ethyl acetate (20/1-5/1) to afford **4a** as a colorless oil (34.2 mg, 40% yield, 52% ee), **1a** as a white solid (50.2 mg, 52% yield, 40% ee) and **6** as a yellow oil (44.7 mg, 84% yield, 44% ee).

Characterization of products



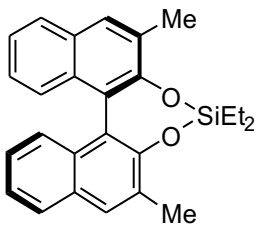
(*R*)-4,4-diethyldinaphtho[2,1-d:1',2'-f][1,3,2]dioxasilepine (4a): The general procedure was followed by using *rac*-**1a** (85.9 mg, 0.30 mmol), Et_2SiH_2 (14.6 mg, 0.165 mmol), $HB(C_6F_5)_2$ (10.4 mg, 0.03 mmol), chiral diene **2a** (10.2 mg, 0.015 mmol), Lewis base **3i** (8.7 mg, 0.015 mmol). Purification by column chromatography (petroleum ether/ethyl acetate = 20/1) yielded

4a as a colorless oil (36.0 mg, 42% yield, 76% ee). $[\alpha]_D^{22}$ = +4.0 (*c* 0.67, THF). IR (film): 3402, 1638 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$, ppm) δ 7.89 (d, *J* = 9.0 Hz, 2H), 7.86 (d, *J* = 8.5 Hz, 2H), 7.38-7.32 (m, 4H), 7.20-7.15 (m, 4H), 0.99 (t, *J* = 8.0 Hz, 6H), 0.82-0.75 (m, 4H); $^{13}C\{^1H\}$ NMR (125 MHz, $CDCl_3$, ppm) δ 150.9, 133.7, 130.5, 130.2, 128.3, 127.2, 126.1, 124.3, 122.0, 121.4, 6.3, 4.3; HRMS (APCI) m/z: $[M+H]^+$ Calcd. for $C_{24}H_{23}O_2Si$ 371.1462; Found 371.1456. HPLC (Chiralpak OJ-H, Daicel Chemical Industries, Ltd., eluent: Hexanes/IPA (80/20); flow rate: 1.0 mL/min; detection: UV 230 nm) t_R = 13.44 min (major), 19.70 min (minor).

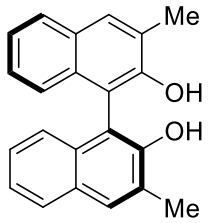


(*S*)-[1,1'-binaphthalene]-2,2'-diol (1a): recovered **1a** as a white solid (48.1 mg, 56% yield, 59% ee). $[\alpha]_D^{22}$ = -3.2 (*c* 0.53, THF) [lit.: $[\alpha]_D^{25}$ = +33.7 (*c* 0.59, THF) (99% ee for (*R*)-isomer)]. ^1H NMR (400 MHz, $CDCl_3$, ppm) δ 7.92 (d, *J* = 8.8 Hz, 2H), 7.86 (d, *J* = 8.0 Hz, 2H), 7.39-7.31 (m, 4H), 7.31-7.25 (m, 2H), 7.13 (d, *J* = 8.4 Hz, 2H), 5.04 (s, 2H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$, ppm) δ 152.9, 133.6, 131.6, 129.6, 128.6, 127.7, 124.4, 124.2, 118.0, 111.1. HPLC (Chiralpak OJ-H, Daicel Chemical Industries, Ltd., eluent: Hexanes/IPA (80/20); flow rate: 1.0 mL/min; detection: UV 230 nm) t_R = 13.99 min (minor), 20.31 min (major).

S. Fang, J.-P. Tan, J. Pan, H. Zhang, Y. Chen, X. Ren and T. Wang, *Angew. Chem., Int. Ed.*, 2021, **60**, 14921–14930.



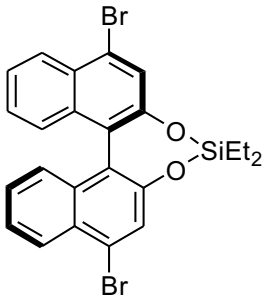
(*R*)-4,4-diethyl-2,6-dimethyldinaphtho[2,1-d:1',2'-f][1,3,2]dioxasilepine (4b): The general procedure was followed by using *rac*-**1b** (94.3 mg, 0.30 mmol), Et_2SiH_2 (14.6 mg, 0.165 mmol), $HB(C_6F_5)_2$ (10.4 mg, 0.03 mmol), chiral diene **2a** (10.2 mg, 0.015 mmol), Lewis base **3i** (8.7 mg, 0.015 mmol). Purification by column chromatography (petroleum ether/ethyl acetate = 20/1) yielded **4b** as a colorless oil (24.9 mg, 21% yield, 31% ee). $[\alpha]_D^{22}$ = +18.7 (*c* 0.52, THF). IR (film): 3402, 1636 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$, ppm) δ 7.77 (d, *J* = 8.5 Hz, 2H), 7.74 (s, 2H), 7.32-7.25 (m, 2H), 7.11-7.02 (m, 4H), 2.49 (s, 6H), 0.96 (t, *J* = 7.5 Hz, 6H), 0.84-0.72 (m, 4H); $^{13}C\{^1H\}$ NMR (125 MHz, $CDCl_3$, ppm) δ 150.1, 132.8, 130.4, 130.1, 129.6, 127.4, 127.1, 125.1, 124.1, 121.3, 17.7, 6.2, 4.6; HRMS (APCI) m/z: $[M+H]^+$ Calcd. for $C_{26}H_{27}O_2Si$ 399.1775; Found 399.1768. HPLC (Chiralpak AS-H, Daicel Chemical Industries, Ltd., eluent: Hexanes/IPA (90/10); flow rate: 1.0 mL/min; detection: UV 230 nm) t_R = 4.83 min (major), 5.69 min (minor).



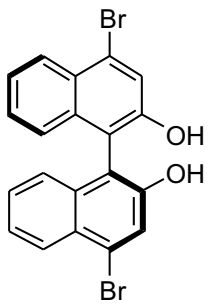
(*S*)-3,3'-dimethyl-[1,1'-binaphthalene]-2,2'-diol (1b): recovered **1b** as a white solid (68.9 mg, 73% yield, 9% ee). $[\alpha]_D^{22}$ = -12.2 (*c* 0.68, THF) [lit.: $[\alpha]_D^{20}$ = -64.0 (*c* 1.0, THF) (99% ee for (*S*)-isomer)]. ^1H NMR (400 MHz, $CDCl_3$, ppm) δ 7.84-7.75 (m, 4H), 7.38-7.27 (m, 2H), 7.26-7.18 (m, 2H), 7.12-7.02 (m, 2H), 5.10 (s, 2H), 2.50 (s, 6H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$, ppm) δ 152.3, 132.4, 130.9, 129.6, 127.8, 127.2, 126.6, 124.3, 124.1, 110.7, 17.2. HPLC (Chiralpak AS-H, Daicel Chemical Industries, Ltd., eluent: Hexanes/IPA (90/10); flow rate: 1.0

mL/min; detection: UV 230 nm) t_R = 4.84 min (minor), 5.69 min (major).

C. Da, J. Wang, X. Yin, X. Fan, Y. Liu, S. Yu, *Org. Lett*. 2009, **11**, 5578-5581.

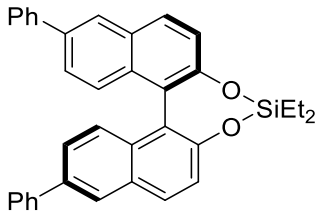


(*R*)-1,7-dibromo-4,4-diethyldinaphtho[2,1-d:1',2'-f][1,3,2]dioxasilepine (4c): The general procedure was followed by using *rac*-**1c** (133.2 mg, 0.30 mmol), Et_2SiH_2 (14.6 mg, 0.165 mmol), $HB(C_6F_5)_2$ (10.4 mg, 0.03 mmol), chiral diene **2a** (10.2 mg, 0.015 mmol), Lewis base **3i** (8.7 mg, 0.015 mmol). Purification by column chromatography (petroleum ether/ethyl acetate = 20/1) yielded **4c** as a colorless oil (68.1 mg, 43% yield, 81% ee). $[\alpha]_D^{21}$ = +50.9 (*c* 0.61, THF), IR (film): 3400, 1638 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$, ppm) δ 8.27 (d, *J* = 8.0 Hz, 2H), 7.70 (s, 2H), 7.49-7.41 (m, 2H), 7.23-7.18 (m, 2H), 7.12 (d, *J* = 8.5 Hz, 2H), 1.00 (t, *J* = 8.0 Hz, 6H), 0.84-0.76 (m, 4H); $^{13}C\{^1H\}$ NMR (125 MHz, $CDCl_3$, ppm) δ 150.5, 134.2, 129.0, 127.5, 127.4, 127.1, 125.9, 125.8, 124.2, 120.9, 6.2, 4.2; HRMS (APCI) m/z: $[M+H]^+$ Calcd. for $C_{24}H_{21}O_2Br_2Si$ 526.9672; Found 526.9664. HPLC (Chiralpak AD-H, Daicel Chemical Industries, Ltd., eluent: Hexanes/IPA (80/20); flow rate: 1.0 mL/min; detection: UV 230 nm) t_R = 13.72 min (major), 20.50 min (minor).



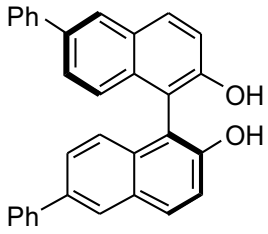
(*S*)-4,4'-dibromo-[1,1'-binaphthalene]-2,2'-diol (1c): recovered **1c** as a yellow solid (73.4 mg, 55% yield, 69% ee). $[\alpha]_D^{22}$ = -84.4 (*c* 0.34, THF) [lit.: $[\alpha]_D^{25}$ = +24.2 (*c* 3.1, $CHCl_3$) (99% ee for (*R*)-isomer)]. ^1H NMR (500 MHz, $CDCl_3$, ppm) δ 8.29 (d, *J* = 8.5 Hz, 2H), 7.74 (s, 2H), 7.53-7.45 (m, 2H), 7.41-7.31 (m, 2H), 7.14 (d, *J* = 8.5 Hz, 2H), 5.00 (s, 2H); $^{13}C\{^1H\}$ NMR (125 MHz, $CDCl_3$, ppm) δ 152.5, 134.1, 128.7, 128.4, 128.0, 126.2, 125.7, 124.7, 122.2, 110.6. HPLC (Chiralpak AD-H, Daicel Chemical Industries, Ltd., eluent: Hexanes/IPA (80/20); flow rate: 1.0 mL/min; detection: UV 230 nm) t_R = 12.60 min (minor), 20.38 min (major).

H. Chow, M. Ng, *Tetrahedron Asymmetry,* 1996, *7*, 2251–2262.



(*R*)-4,4-diethyl-9,14-diphenyldinaphtho[2,1-d:1',2'-f][1,3,2]dioxasilepine (4d): The general procedure was followed by using *rac*-**1d** (131.6 mg, 0.30 mmol), Et_2SiH_2 (14.6 mg, 0.165 mmol), $HB(C_6F_5)_2$ (10.4 mg, 0.03 mmol), chiral diene **2a** (10.2 mg, 0.015 mmol), Lewis base **3i** (8.7 mg, 0.015 mmol). Purification by column chromatography (petroleum ether/ethyl acetate = 20/1) yielded **4d** as a white solid (58.3 mg, 37% yield, 71% ee). m.p. 58-60 ºC. $[\alpha]_D^{21}$ = -77.2 (*c* 0.62, THF). IR (film): 3402, 1639 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$, ppm) δ 8.08 (s, 2H), 7.95 (d, *J* = 8.8 Hz, 2H), 7.72-7.62 (m, 4H), 7.50-7.36 (m, 8H), 7.35-7.28 (m, 4H), 1.01 (t, *J* = 8.0 Hz, 6H), 0.85-0.76 (m, 4H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$, ppm) δ 151.1, 141.0, 137.0, 132.9, 130.8, 130.5, 129.0, 127.8, 127.4, 126.1, 125.9, 122.5, 121.4, 6.3, 4.3; HRMS (APCI) m/z: $[M+H]^+$ Calcd. for $C_{36}H_{31}O_2Si$ 523.2088; Found 523.2081. HPLC (Chiralpak OD-

H, Daicel Chemical Industries, Ltd., eluent: Hexanes/IPA (80/20); flow rate: 1.0 mL/min; detection: UV 254 nm) t_R = 10.37 min (minor), 21.39 min (major).



(*S*)-6,6'-diphenyl-[1,1'-binaphthalene]-2,2'-diol (1d): recovered **1d** as a white solid (83.0 mg, 63% yield, 40% ee). $[\alpha]_D^{21}$ = +54.7 (*c* 0.51, THF) [lit.: $[\alpha]_D^{25}$ = -129.9 (*c* 0.30, $CHCl_3$) (97% ee for (*R*)-isomer)]. ^{1}H NMR (500 MHz, $CDCl_3$, ppm) δ 8.10 (s, 2H), 8.04 (d, *J* = 9.0 Hz, 2H), 7.67 (d, *J* = 7.5 Hz, 2H), 7.59 (dd, *J* = 9.0, 1.5 Hz, 2H), 7.50-7.40 (m, 6H), 7.39-7.33 (m, 2H), 7.27 (d, *J* = 8.5 Hz, 2H), 5.09 (s, 2H); ^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$, ppm) δ 153.1, 141.0, 137.2, 132.8, 132.0, 130.0, 129.1, 127.5, 127.46, 127.43, 126.6, 125.0, 118.5, 110.9. HPLC (Chiralpak OD-H, Daicel Chemical Industries, Ltd., eluent: Hexanes/IPA (80/20); flow rate: 1.0 mL/min; detection: UV 254 nm) t_R = 10.32 min (major), 21.92 min (minor).

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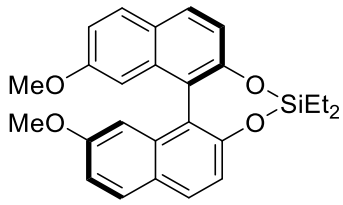
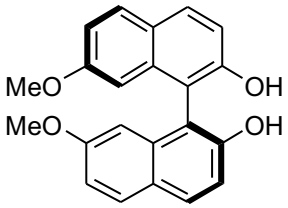


(*R*)-4,4-diethyl-10,13-dimethoxydinaphtho[2,1-d:1',2'-f][1,3,2]dioxasilepine (4e): The general procedure was followed by using *rac*-**1e** (103.9 mg, 0.30 mmol), Et_2SiH_2 (14.6 mg, 0.165 mmol), $HB(C_6F_5)_2$ (10.4 mg, 0.03 mmol), chiral diene **2a** (10.2 mg, 0.015 mmol), Lewis base **3i** (8.7 mg, 0.015 mmol). Purification by column chromatography (petroleum ether/ethyl

acetate = 20/1) yielded **4e** as a colorless oil (54.2 mg, 42% yield, 74% ee). $[\alpha]_D^{22}$ = -28.0 (*c* 0.61, THF). IR (film): 3401, 1633 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$, ppm) δ 7.80 (d, *J* = 8.4 Hz, 2H), 7.74 (d, *J* = 8.8 Hz, 2H), 7.21 (d, *J* = 8.8 Hz, 2H), 7.00 (dd, *J* = 8.8, 2.4 Hz, 2H), 6.50 (d, *J* = 2.4 Hz, 2H), 3.34 (s, 6H), 1.01 (t, *J* = 8.0 Hz, 6H), 0.84-0.78 (m, 4H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$, ppm) δ 157.9, 151.6, 134.5, 129.81, 129.79, 125.9, 120.7, 119.5, 116.8, 106.3, 55.1, 6.3, 4.3; HRMS (APCI) m/z: $[M+H]^+$ Calcd. for $C_{26}H_{27}O_4Si$ 431.1673; Found 431.1666. HPLC (Chiralpak OD-H, Daicel Chemical Industries, Ltd., eluent: Hexanes/IPA (80/20); flow rate: 1.0 mL/min; detection: UV 230 nm) t_R = 14.66 min (minor), 20.09 min (major).



(*S*)-7,7'-dimethoxy-[1,1'-binaphthalene]-2,2'-diol (1e): recovered **1e** as a white solid (58.1 mg, 56% yield, 54% ee). $[\alpha]_D^{22}$ = +17.4 (*c* 0.51, THF) [lit.: $[\alpha]_D^{25}$ = -19.0 (*c* 0.59, $CHCl_3$) (99% ee for (*R*)-isomer)]. ^1H NMR (400 MHz, $CDCl_3$, ppm) δ 7.87 (d, *J* = 8.8 Hz, 2H), 7.78 (d, *J* = 9.2 Hz, 2H), 7.21 (d, *J* = 8.8 Hz, 2H), 7.03 (dd, *J* = 8.8, 2.0 Hz, 2H), 6.48 (s, 2H), 5.07 (s, 2H), 3.57 (s, 6H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$, ppm) δ 159.3, 153.5, 134.9, 131.3, 130.2, 125.0, 116.2, 115.3, 110.3, 103.4, 55.4. HPLC (Chiralpak OD-H, Daicel Chemical Industries, Ltd., eluent: Hexanes/IPA (80/20); flow rate: 1.0 mL/min; detection: UV 230 nm) t_R = 14.46 min (major), 19.69 min (minor).

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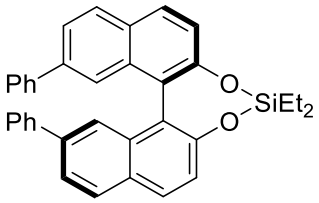
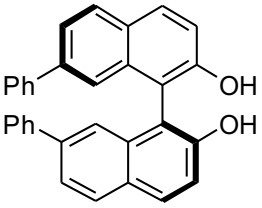


(*R*)-4,4-diethyl-10,13-diphenyldinaphtho[2,1-d:1',2'-f][1,3,2]dioxasilepine (4f): The general procedure was followed by using *rac*-**1f** (131.6 mg, 0.30 mmol), Et_2SiH_2 (14.6 mg, 0.165 mmol), $HB(C_6F_5)_2$ (10.4 mg, 0.03 mmol), chiral diene **2a** (10.2 mg, 0.015 mmol), Lewis base **3i** (8.7 mg, 0.015 mmol). Purification by column chromatography (petroleum ether/ethyl acetate = 20/1) yielded **4f** as a white solid (69.1 mg, 44% yield, 64% ee). m.p. 60-61 ºC. $[\alpha]_D^{21}$ = -70.0 (*c* 0.53, THF). IR (film): 3596, 1633 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$, ppm) δ 7.98-7.87 (m, 4H), 7.62 (d, *J* = 8.4 Hz, 2H), 7.48 (s, 2H), 7.35 (d, *J* = 8.8 Hz, 2H), 7.25-7.10 (m, 10H), 1.02 (t, *J* = 8.0 Hz, 6H), 0.86-0.77 (m, 4H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$, ppm) δ 151.5, 141.6, 139.0, 133.9, 130.0, 129.7, 129.0, 128.8, 127.5, 127.2, 125.7, 124.2, 122.1, 121.6, 6.3, 4.4; HRMS (APCI) m/z: $[M+H]^+$ Calcd. for $C_{32}H_{23}O_2Si$ 439.1693; Found 439.1684. HPLC (Chiralpak OD-H, Daicel Chemical Industries, Ltd., eluent: Hexanes/IPA (90/10); flow rate: 1.0 mL/min; detection: UV 254 nm) t_R = 14.78 min (minor), 25.53 min (major).



(*S*)-7,7'-diphenyl-[1,1'-binaphthalene]-2,2'-diol (1f): recovered **1f** as a yellow solid (71.0 mg, 55% yield, 51% ee) $[\alpha]_D^{21}$ = +60.2 (*c* 0.50, THF) [lit.: $[\alpha]_D^{25}$ = -120.32 (*c* 0.50, $CHCl_3$) (90% ee for (*R*)-isomer)]. ^1H NMR (400 MHz, $CDCl_3$, ppm) δ 8.03-7.94 (m, 4H), 7.63 (dd, *J* = 8.4, 1.2 Hz, 2H), 7.45-7.36 (m, 8H), 7.35-7.28 (m, 4H), 7.26 (d, *J* = 8.0 Hz, 2H), 5.13 (s, 2H);

$^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$, ppm) δ 153.4, 141.2, 140.6, 133.8, 131.5, 129.2, 128.9, 127.7, 127.6, 124.2, 122.3, 118.1, 111.2. HPLC (Chiralpak OD-H, Daicel Chemical Industries, Ltd., eluent: Hexanes/IPA (90/10); flow rate: 1.0 mL/min; detection: UV 254 nm) t_R = 15.14 min (major), 26.27 min (minor).

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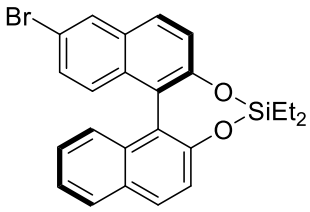
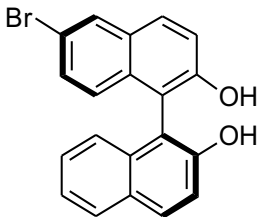


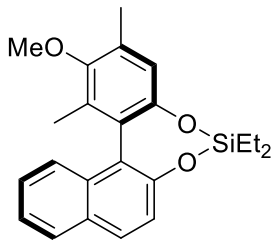
(*R*)-9-bromo-4,4-diethyldinaphtho[2,1-d:1',2'-f][1,3,2]dioxasilepine (4g): The general procedure was followed by using *rac*-**1g** (109.6 mg, 0.30 mmol), Et_2SiH_2 (14.6 mg, 0.165 mmol), $HB(C_6F_5)_2$ (10.4 mg, 0.03 mmol), chiral diene **2a** (10.2 mg, 0.015 mmol), Lewis base **3i** (8.7 mg, 0.015 mmol). Purification by column chromatography (petroleum ether/ethyl acetate = 10/1) yielded **4g** as a colorless oil (52.3 mg, 39% yield, 78% ee). $[\alpha]_D^{22}$ = -7.4 (*c* 0.51, THF). IR (film): 3402, 1637 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$, ppm) δ 7.93 (d, *J* = 1.5 Hz, 1H), 7.80 (d, *J* = 8.5 Hz, 1H), 7.77 (d, *J* = 8.0 Hz, 1H), 7.70 (d, *J* = 8.5 Hz, 1H), 7.30-7.24 (m, 3H), 7.16-7.09 (m, 2H), 7.04 (d, *J* = 8.5 Hz, 1H), 6.96 (d, *J* = 9.0 Hz, 1H), 0.90 (t, *J* = 8.0 Hz, 6H), 0.74-0.65 (m, 4H); $^{13}C\{^{1}H\}$ NMR (125 MHz, $CDCl_3$, ppm) δ 151.2, 151.0, 133.6, 132.3, 131.6, 130.5, 130.4, 130.2, 129.4, 129.2, 129.1, 128.4, 126.9, 126.4, 124.4, 123.2, 121.9, 121.8, 120.8, 118.2, 6.23, 6.21, 4.32, 4.28; HRMS (APCI) m/z: $[M+H]^+$ Calcd. for $C_{24}H_{22}O_2BrSi$ 449.0567; Found 449.0559. HPLC (Chiralpak IC-H, Daicel Chemical Industries, Ltd., eluent: Hexanes/IPA (95/5); flow rate: 1.0 mL/min; detection: UV 230 nm) t_R = 10.48 min (minor),

13.31 min (major).



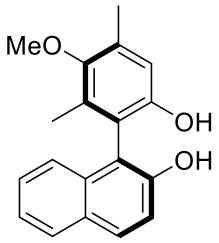
(*S*)-6-bromo-[1,1'-binaphthalene]-2,2'-diol (1g): recovered **1g** as a white solid (60.2 mg, 55% yield, 43% ee). $[\alpha]_D^{22}$ = +1.5 (*c* 0.40, THF) [lit.: $[\alpha]_D^{22}$ = -59.7 (*c* 4.02, $CHCl_3$) (74% ee for (*R*)-isomer)]. ^{1}H NMR (300 MHz, $CDCl_3$, ppm) δ 8.10-8.04 (m, 1H), 8.00 (d, *J* = 9.0 Hz, 1H), 7.95-7.86 (m, 2H), 7.45-7.31 (m, 5H), 7.12 (d, *J* = 8.4 Hz, 1H), 7.04 (d, *J* = 9.0 Hz, 1H), 5.13 (s, 1H), 5.04 (s, 1H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$, ppm) δ 153.2, 152.9, 133.5, 132.2, 131.9, 130.9, 130.8, 130.6, 130.5, 129.7, 128.7, 127.9, 126.3, 124.4, 124.2, 119.1, 118.1, 118.0, 111.5, 110.4. HPLC (Chiralpak IC-H, Daicel Chemical Industries, Ltd., eluent: Hexanes/IPA (95/5); flow rate: 1.0 mL/min; detection: UV 230 nm) t_R = 10.34 min (major), 13.21 min (minor).

S. Narute, R. Parnes, F. Toste, D. Pappo, *J. Am. Chem. Soc*. 2016, **138**, 16553−16560.



(*S*)-6,6-diethyl-2-methoxy-1,3-dimethylbenzo[d]naphtho[1,2-f][1,3,2]dioxasilepine (4h): The general procedure was followed by using *rac*-**1h** (88.3 mg, 0.30 mmol), Et_2SiH_2 (14.6 mg, 0.165 mmol), $HB(C_6F_5)_2$ (10.4 mg, 0.03 mmol), chiral diene **2a** (10.2 mg, 0.015 mmol), Lewis base **3i** (8.7 mg, 0.015 mmol). Purification by column chromatography (petroleum ether/ethyl acetate = 20/1) yielded **4h** as a colorless oil (47.7 mg, 42% yield, 53% ee). $[\alpha]_D^{22}$ = +14.1 (*c*

0.51, THF). IR (film): 3401, 1639 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$, ppm) δ 7.78-7.68 (m, 2H), 7.42-7.33 (m, 1H), 7.32-7.24 (m, 2H), 7.18 (d, *J* = 8.8 Hz, 1H), 6.73 (s, 1H), 3.68 (s, 3H), 2.27 (s, 3H), 1.75 (s, 3H), 0.98-0.82 (m, 6H), 0.76-0.59 (m, 4H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$, ppm) δ 153.0, 150.1, 148.6, 133.3, 132.3, 131.5, 130.4, 129.7, 128.4, 126.5, 126.3, 125.7, 124.2, 122.9, 121.8, 120.5, 60.1, 16.4, 15.5, 6.25, 6.21, 4.1, 3.9; HRMS (APCI) m/z: $[M+H]^+$ Calcd. for $C_{23}H_{27}O_3Si$ 379.1724; Found 379.1717. HPLC (Chiralpak AD-H, Daicel Chemical Industries, Ltd., eluent: Hexanes/IPA (80/20); flow rate: 1.0 mL/min; detection: UV 230 nm) t_R = 16.46 min (minor), 23.23 min (major).



(*S*)-1-(6-hydroxy-3-methoxy-2,4-dimethylphenyl)naphthalen-2-ol (1h): recovered **1h** as a white solid (49.3 mg, 56% yield, 36% ee). $[\alpha]_D^{22}$ = -10.4 (*c* 0.45, THF) [lit.: $[\alpha]_D^{25}$ = -64.3 (*c* 0.40, $CHCl_3$) (96% ee for (*S*)-isomer)]. ^1H NMR (300 MHz, $CDCl_3$, ppm) δ 7.94-7.76 (m, 2H), 7.40-7.23 (m, 4H), 6.80 (s, 1H), 5.14 (s, 1H), 4.45 (s, 1H), 3.74 (s, 3H), 2.37 (s, 3H), 1.89 (s, 3H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$, ppm) δ 152.1, 151.5, 150.5, 133.8, 133.2, 132.4, 131.2, 129.5, 128.6, 127.6, 124.1, 124.0, 117.7, 116.8, 115.7, 112.8, 60.4, 16.6, 13.3. HPLC (Chiralpak AD-H, Daicel Chemical Industries, Ltd., eluent: Hexanes/IPA (80/20); flow rate: 1.0 mL/min; detection: UV 254 nm) t_R = 16.23 min (major), 22.93 min (minor).

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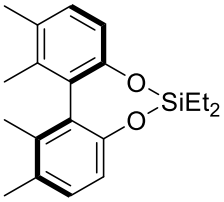
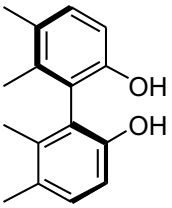


(*R*)-6,6-diethyl-1,2,10,11-tetramethyldibenzo[d,f][1,3,2]dioxasilepine (4i): The general procedure was followed by using *rac*-**1i** (109.6 mg, 0.30 mmol), Et_2SiH_2 (14.6 mg, 0.165 mmol), $HB(C_6F_5)_2$ (10.4 mg, 0.03 mmol), chiral diene **2a** (10.2 mg, 0.015 mmol), Lewis base **3i** (8.7 mg, 0.015 mmol). Purification by column chromatography (petroleum ether/ethyl acetate = 20/1) yielded **4i** as a colorless oil (45.1 mg, 46% yield, 55% ee). $[\alpha]_D^{21}$ = +33.0 (*c* 0.49, THF). IR (film): 3471, 1640 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$, ppm) δ 6.99 (d, *J* = 8.0 Hz, 2H), 6.73 (d, *J* = 8.0 Hz, 2H), 2.18 (s, 6H), 1.88 (s, 6H), 0.90 (t, *J* = 8.0 Hz, 6H), 0.68-0.57 (m, 4H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$, ppm) δ 150.3, 137.5, 131.4, 129.7, 128.9, 117.7, 20.4, 17.6, 6.2, 3.6; HRMS (APCI) m/z: $[M+H]^+$ Calcd. for $C_{20}H_{27}O_2Si$ 327.1775; Found 327.1775. HPLC (Chiralpak IC-H, Daicel Chemical Industries, Ltd., eluent: Hexanes/IPA (95/5); flow rate: 1.0 mL/min; detection: UV 210 nm) t_R = 9.59 min (major), 18.78 min (minor).



(*S*)-5,5',6,6'-tetramethyl-[1,1'-biphenyl]-2,2'-diol (1i): recovered **1i** as a white solid (37.8 mg, 52% yield, 54% ee). $[\alpha]_D^{21}$ = -35.4 (*c* 0.55, THF) [lit.: $[\alpha]_D^{25}$ = -22.2 (*c* 0.60, $CHCl_3$) (64% ee for (*S*)-isomer)]. ^1H NMR (500 MHz, $CDCl_3$, ppm) δ 7.13 (d, *J* = 8.0 Hz, 2H), 6.81 (d, *J* = 8.5 Hz, 2H), 4.52 (s, 2H), 2.25 (s, 6H), 1.89 (s, 6H); $^{13}C\{^1H\}$ NMR (125 MHz, $CDCl_3$, ppm) δ 152.0, 137.1, 131.5, 129.4, 120.4, 112.8, 20.0, 16.5. HPLC (Chiralpak IC-H, Daicel Chemical

Industries, Ltd., eluent: Hexanes/IPA (95/5); flow rate: 1.0 mL/min; detection: UV 230 nm) t_R = 9.55 min (minor), 18.77 min (major).

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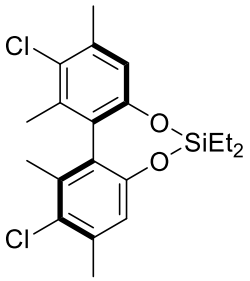
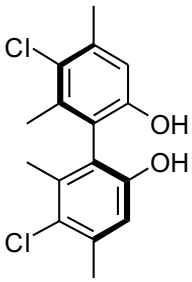
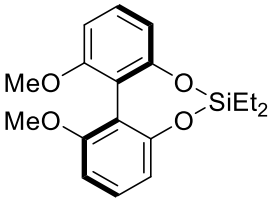


(*R*)-2,10-dichloro-6,6-diethyl-1,3,9,11-tetramethyldibenzo[d,f][1,3,2]dioxasilepine (4j): The general procedure was followed by using *rac*-**1j** (93.4mg, 0.30 mmol), Et_2SiH_2 (14.6 mg, 0.165 mmol), $HB(C_6F_5)_2$ (10.4 mg, 0.03 mmol), chiral diene **2a** (10.2 mg, 0.015 mmol), Lewis base **3i** (8.7 mg, 0.015 mmol). Purification by column chromatography (petroleum ether/ethyl acetate = 20/1) yielded **4j** as a colorless oil (49.8 mg, 42% yield, 48% ee). $[\alpha]_D^{22}$ = +52.6 (*c* 0.53, THF). IR (film): 3401, 1638 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$, ppm) δ 6.81 (s, 2H), 2.39 (s, 6H), 2.10 (s, 6H), 0.98 (t, *J* = 8.1 Hz, 6H), 0.78-0.66 (m, 4H); $^{13}C\{^1H\}$ NMR (125 MHz, $CDCl_3$, ppm) δ 150.3, 137.1, 136.9, 129.7, 127.4, 120.5, 21.1, 19.1, 6.2, 3.6; HRMS (APCI) m/z: $[M+H]^+$ Calcd. for $C_{20}H_{25}O_2Cl_2Si$ 395.0995; Found 395.0993. HPLC (Chiralpak OJ-H, Daicel Chemical Industries, Ltd., eluent: Hexanes/IPA (95/5); flow rate: 1.0 mL/min; detection: UV 230 nm) t_R = 9.53 min (minor), 11.26 min (major).



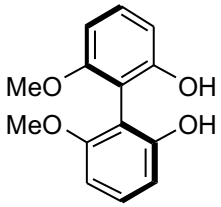
(*S*)-5,5'-dichloro-4,4',6,6'-tetramethyl-[1,1'-biphenyl]-2,2'-diol (1j): recovered **1j** as a yellow solid (50.3 mg, 54% yield, 35% ee). $[\alpha]_D^{22}$ = -47.4 (*c* 0.50, THF) [lit.: $[\alpha]_D^{25}$ = +10.0 (*c* 0.10, $CHCl_3$) (99% ee for (*R*)-isomer)]. ^1H NMR (500 MHz, $CDCl_3$, ppm) δ 6.83 (s, 2H), 4.56 (s, 2H), 2.41 (s, 6H), 2.05 (s, 6H); $^{13}C\{^1H\}$ NMR (125 MHz, $CDCl_3$, ppm) δ 152.0, 139.0, 136.8, 127.4, 118.8, 115.9, 21.3, 17.9. HPLC (Chiralpak OJ-H, Daicel Chemical Industries, Ltd., eluent: Hexanes/IPA (95/5); flow rate: 1.0 mL/min; detection: UV 210 nm) t_R = 9.98 min (major), 11.84 min (minor).

S. Lu, S. B. Poh and Y. Zhao, *Angew. Chem., Int. Ed.*, 2014, **53**, 11041−11045.



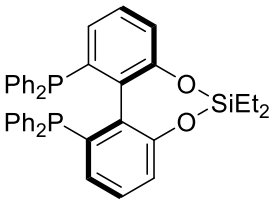
(*R*)-6,6-diethyl-1,11-dimethoxydibenzo[d,f][1,3,2]dioxasilepine (4k): The general procedure was followed by using *rac*-**1k** (109.6 mg, 0.30 mmol), Et_2SiH_2 (14.6 mg, 0.165 mmol), $HB(C_6F_5)_2$ (10.4 mg, 0.03 mmol), chiral diene **2a** (10.2 mg, 0.015 mmol), Lewis base **3i** (8.7 mg, 0.015 mmol). Purification by column chromatography (petroleum ether/ethyl acetate = 20/1) yielded **4k** as a colorless oil (44.4 mg, 46% yield, 62% ee). $[\alpha]_D^{21}$ = +10.0 (*c* 0.51, THF). IR (film): 3402, 1638 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$, ppm) δ 7.25-7.20 (m, 2H), 6.69 (d, *J* = 8.4 Hz, 4H), 3.78 (s, 6H), 0.98 (t, *J* = 8.0 Hz, 6H), 0.79-0.71 (m, 4H); $^{13}C\{^1H\}$ NMR (100

MHz, $CDCl_3$, ppm) δ 158.8, 153.3, 129.1, 115.3, 113.6, 105.8, 56.1, 6.2, 4.1; HRMS (APCI) m/z: $[M+H]^+$ Calcd. for $C_{18}H_{23}O_4Si$ 331.1360; Found 331.1353. HPLC (Chiralpak OD-H, Daicel Chemical Industries, Ltd., eluent: Hexanes/IPA (80/20); flow rate: 1.0 mL/min; detection: UV 230 nm) t_R = 19.57 min (major), 25.35 min (minor).



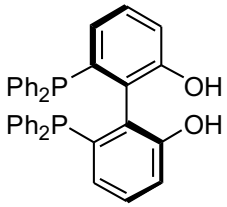
(*S*)-6,6'-dimethoxy-[1,1'-biphenyl]-2,2'-diol (1k): recovered **1k** as a white solid (37.6 mg, 51% yield, 68% ee). $[\alpha]_D^{22}$ = -7.5 (*c* 0.60, THF) [lit.: $[\alpha]_D^{25}$ = +83.7 (*c* 0.10, $CHCl_3$) (99% ee for (*R*)-isomer)]. ^1H NMR (400 MHz, $CDCl_3$, ppm) δ 7.34-7.27 (m, 2H), 6.72 (d, *J* = 8.4 Hz, 2H), 6.62 (d, *J* = 8.0 Hz, 2H), 5.06 (s, 2H), 3.76 (s, 6H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$, ppm) δ 158.2, 155.6, 130.8, 109.4, 107.2, 103.7, 56.3. HPLC (Chiralpak OD-H, Daicel Chemical Industries, Ltd., eluent: Hexanes/IPA (80/20); flow rate: 1.0 mL/min; detection: UV 230 nm) t_R = 19.85 min (minor), 25.14 min (major).

S. Lu, S. B. Poh and Y. Zhao, *Angew. Chem., Int. Ed.*, 2014, **53**, 11041−11045.



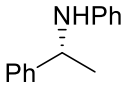
(*R*)-(6,6-diethyldibenzo[d,f][1,3,2]dioxasilepine-1,11-diyl)bis(diphenylphosphane) (4l): The general procedure was followed by using *rac*-**1l** (88.3 mg, 0.30 mmol), Et_2SiH_2 (14.6 mg, 0.165 mmol), $HB(C_6F_5)_2$ (10.4 mg, 0.03 mmol), chiral diene **2a** (10.2 mg, 0.015 mmol), Lewis base **3i** (8.7 mg, 0.015 mmol). Purification by column chromatography (petroleum ether/ethyl

acetate = 20/1) yielded **4l** as a colorless oil (48.6 mg, 38% yield, 26% ee). $[\alpha]_D^{22}$ = +52.8 (*c* 0.57, THF). IR (film): 3441, 1638 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$, ppm) δ 7.60-7.53 (m, 4H), 7.51-7.43 (m, 4H), 7.41-7.37 (m, 2H), 7.25-7.10 (m, 8H), 7.04-6.90 (m, 4H), 6.82-6.67 (m, 4H), 0.74 (t, *J* = 7.5 Hz, 6H), 0.66-0.49 (m, 4H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$, ppm) δ 151.6 (t, *J* = 3.7 Hz), 140.2 (t, *J* = 3.4 Hz), 137.1 (t, *J* = 8.7 Hz), 135.9 (t, *J* = 4.1 Hz), 132.9 (t, *J* = 11.0 Hz), 132.5 (t, *J* = 10.9 Hz), 130.7 (t, *J* = 13.8 Hz), 128.2, 127.4, 127.3, 127.1, 126.7 (t, *J* = 3.4 Hz), 126.5, 119.6, 5.0, 3.4; $^{31}P\{^1H\}$ NMR (162 MHz, $CDCl_3$, ppm) δ -5.3.; HRMS (APCI) m/z: $[M+H]^+$ Calcd. for $C_{40}H_{37}O_2P_2Si$ 639.2033; Found 639.2014. HPLC (Chiralpak OD-H, Daicel Chemical Industries, Ltd., eluent: Hexanes/IPA (85/15); flow rate: 1.0 mL/min; detection: UV 254 nm) t_R = 8.65 min (major), 12.91 min (minor).



(*S*)-6,6'-bis(diphenylphosphaneyl)-[1,1'-biphenyl]-2,2'-diol (1l): recovered **1l** as a white solid (56.6 mg, 51% yield, 8% ee) $[\alpha]_D^{22}$ = -39.8 (*c* 0.53, THF). ^1H NMR (400 MHz, $CDCl_3$, ppm) δ 7.34-7.14 (m, 22H), 6.86-6.76 (m, 4H), 4.22 (s, 2H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$, ppm) δ 154.6 (t, *J* = 5.1 Hz), 141.4 (t, *J* = 5.0 Hz), 137.4 (t, *J* = 6.6 Hz), 136.9 (t, *J* = 5.9 Hz), 134.7 (t, *J* = 11.1 Hz), 133.3 (t, *J* = 10.1 Hz), 130.8, 129.3, 128.7 (t, *J* = 3.7 Hz), 128.4 (t, *J* = 2.8 Hz), 128.3, 127.2, 125.8 (t, *J* = 19.9 Hz), 116.7; $^{31}P\{^1H\}$ NMR (162 MHz, $CDCl_3$, ppm) δ -13.7. HPLC (Chiralpak OD-H, Daicel Chemical Industries, Ltd., eluent: Hexanes/IPA (85/15); flow rate: 1.0 mL/min; detection: UV 254 nm) t_R = 8.55 min (minor), 12.79 min (major).

Z. Zhao, H. Qian, Q. Longmire and X. Zhang, *J. Org. Chem*. 2011, **65**, 6223–6226.

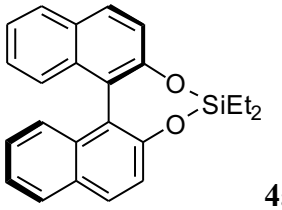


***R*-*N*-(1-phenylethyl)aniline (6):** The general procedure was followed by using *rac*-**1l** (88.3 mg, 0.30 mmol), Et_2SiH_2 (14.6 mg, 0.165 mmol), imide (58.6 mg, 0.30 mmol), $HB(C_6F_5)_2$ (10.4 mg, 0.03 mmol), chiral diene **2a** (10.2 mg, 0.015 mmol), Lewis base **3i** (8.7 mg, 0.015 mmol). Purification by column chromatography (petroleum ether/ethyl acetate = 20/1) yielded **4a** as a colorless oil (34.2 mg, 40% yield, 52% ee), **1a** as a white solid (50.2 mg, 52% yield, 40% ee) and **6** as a yellow oil (44.7 mg, 84% yield, 44% ee). $[\alpha]_D^{22}$ = -16.4 (*c* 0.97, THF) [lit.: $[\alpha]_D^{18}$ = -14.7 (*c* 0.50, MeOH) (91% ee for (*R*)-isomer)]. IR (film): cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$, ppm) δ 7.39-7.27 (m, 4H), 7.22 (d, *J* = 6.4 Hz, 1H), 7.12-7.05 (m, 2H), 6.68-6.60 (m, 1H), 6.51 (d, *J* = 8.4 Hz, 2H), 4.48 (q, *J* = 6.4 Hz, 1H), 4.07 (br, 1H), 1.51 (d, *J* = 6.8 Hz, 3H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$, ppm) δ 147.4, 145.4, 129.3, 128.8, 127.1, 126.1, 117.5, 113.5, 53.7, 25.2; HPLC (Chiralpak OD-H, Daicel Chemical Industries, Ltd., eluent: Hexanes/IPA (98/2); flow rate: 0.5 mL/min; detection: UV 254 nm) t_R = 21.92 min (minor), 29.85 min (major).

Z. Han, Z. Wang, X. Zhao, K. Ding, *Angew. Chem., Int. Ed.*, 2009, **48**, 5345−5349.

The chromatography for the determination of enantiomeric excess

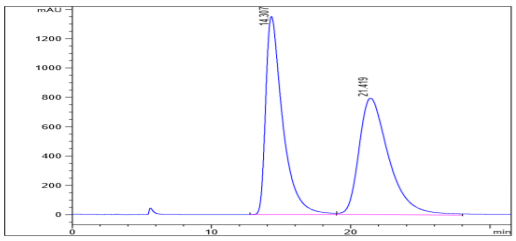
The ee of chiral siloxanes **4** was determined after the removal of the silyl protection



4a

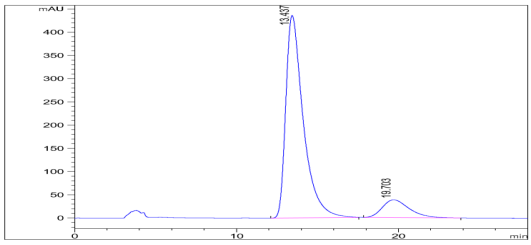
HPLC: Chiralcel OJ-H, ^{i}PrOH/*n*-hexane = 20/80, flow rate = 1.0 mL/min, UV = 230 nm

Racemic

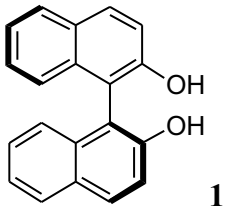


Peak #	RT [min]	Area	Area %
1	14.307	1.144e5	49.237
2	21.419	1.180e5	50.763

Enantioenriched



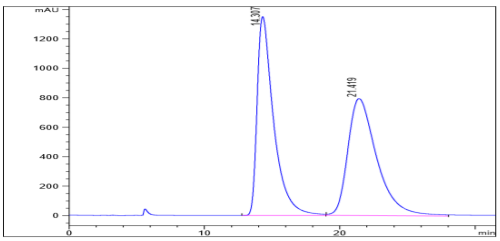
Peak #	RT [min]	Area	Area %
1	13.437	3.272e4	88.145
2	19.703	4.401e3	11.855



1a

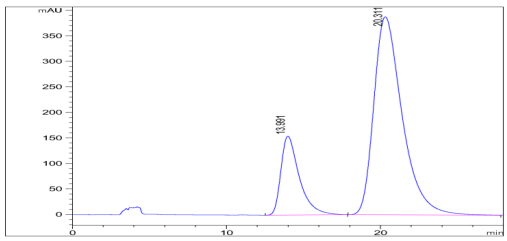
HPLC: Chiralcel OJ-H, ^{i}PrOH/*n*-hexane = 20/80, flow rate = 1.0 mL/min, UV = 230 nm

Racemic

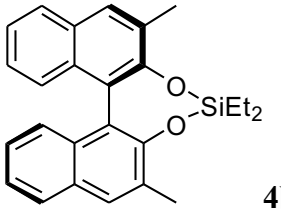


Peak #	RT [min]	Area	Area %
1	14.307	1.144e5	49.237
2	21.419	1.180e5	50.763

Enantioenriched



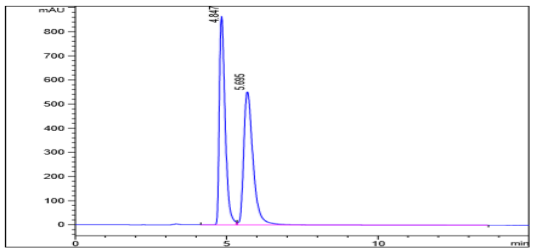
Peak #	RT [min]	Area	Area %
1	13.991	1.283e4	20.488
2	20.311	4.978e4	79.512



4b

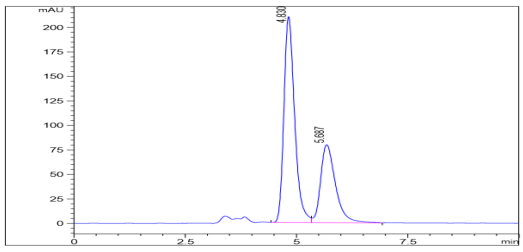
HPLC: Chiralcel AS-H, ^{i}PrOH/*n*-hexane = 10/90, flow rate = 1.0 mL/min, UV = 230 nm

Racemic

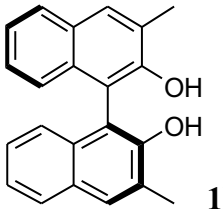


Peak #	RT [min]	Area	Area %
1	4.847	1.155e4	49.186
2	5.695	1.193e4	50.814

Enantioenriched



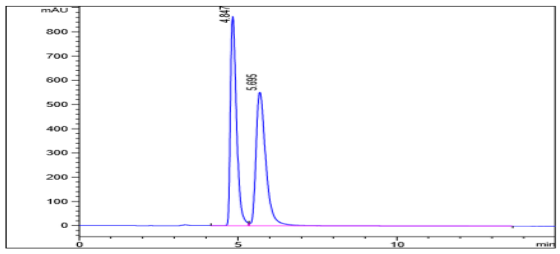
Peak #	RT [min]	Area	Area %
1	4.830	3.516e3	65.584
2	5.687	1.845e3	34.416



1b

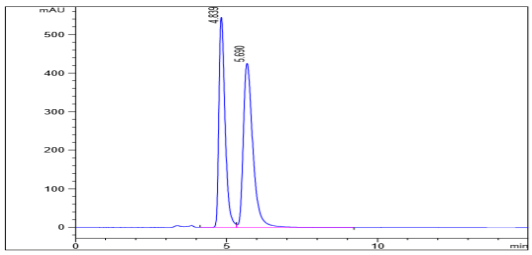
HPLC: Chiralcel AS-H, ^{i}PrOH/*n*-hexane = 10/90, flow rate = 1.0 mL/min, UV = 230 nm

Racemic

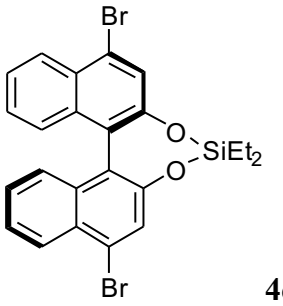


Peak #	RT [min]	Area	Area %
1	4.847	1.155e4	49.186
2	5.695	1.193e4	50.814

Enantioenriched



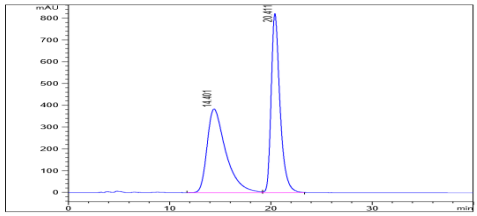
Peak #	RT [min]	Area	Area %
1	4.839	7.761e3	45.302
2	5.690	9.370e3	54.698



4c

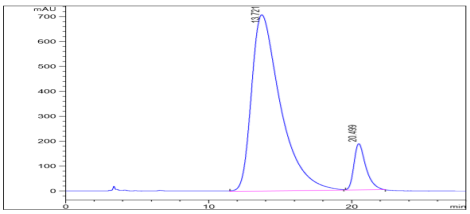
HPLC: Chiralcel AD-H, ^{i}PrOH/*n*-hexane = 10/90, flow rate = 1.0 mL/min, UV = 230 nm

Racemic

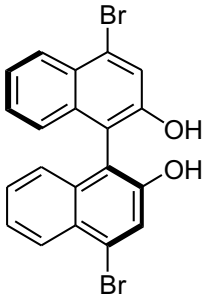


Peak #	RT [min]	Area	Area %
1	14.401	4.917e4	49.702
2	20.411	4.976e4	50.298

Enantioenriched



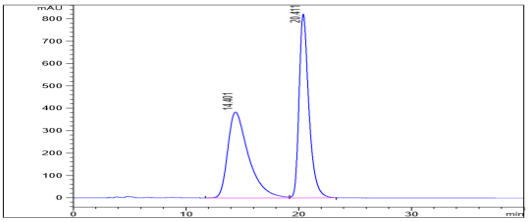
Peak #	RT [min]	Area	Area %
1	13.721	9.960e4	90.324
2	20.499	1.067e4	9.676



1c

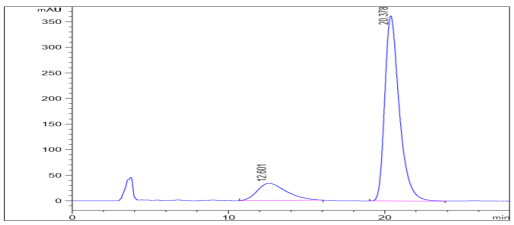
HPLC: Chiralcel AD-H, ^{i}PrOH/*n*-hexane = 10/90, flow rate = 1.0 mL/min, UV = 230 nm

Racemic

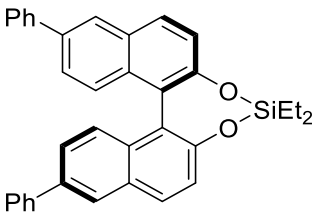


Peak #	RT [min]	Area	Area %
1	14.401	4.917e4	49.702
2	20.411	4.976e4	50.298

Enantioenriched



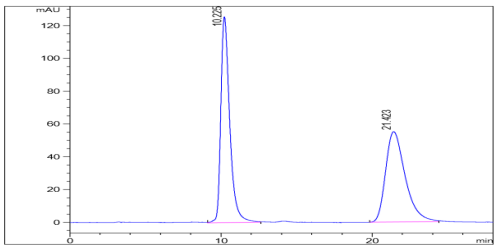
Peak #	RT [min]	Area	Area %
1	12.601	4.345e3	15.668
2	20.378	2.338e4	84.332



4d

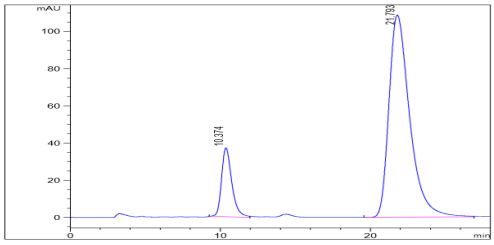
HPLC: Chiralcel OD-H, ^{i}PrOH/*n*-hexane = 20/80, flow rate = 1.0 mL/min, UV = 254 nm

Racemic

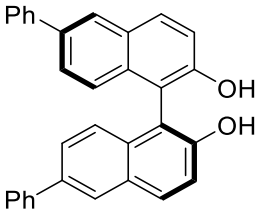


Peak #	RT [min]	Area	Area %
1	10.225	5.153e3	51.021
2	21.423	4.946e3	48.979

Enantioenriched



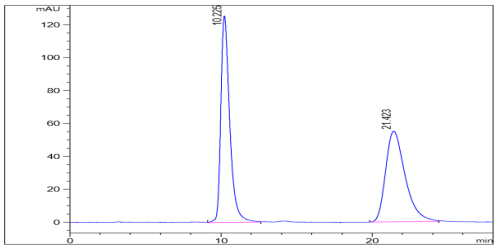
Peak #	RT [min]	Area	Area %
1	10.374	1.764e3	14.429
2	21.793	1.046e4	85.571



1d

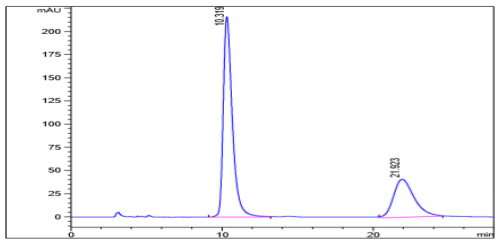
HPLC: Chiralcel OD-H, ^{i}PrOH/*n*-hexane = 20/80, flow rate = 1.0 mL/min, UV = 254 nm

Racemic

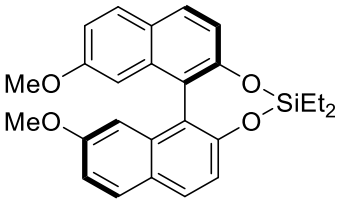


Peak #	RT [min]	Area	Area %
1	10.225	5.153e3	51.021
2	21.423	4.946e3	48.979

Enantioenriched



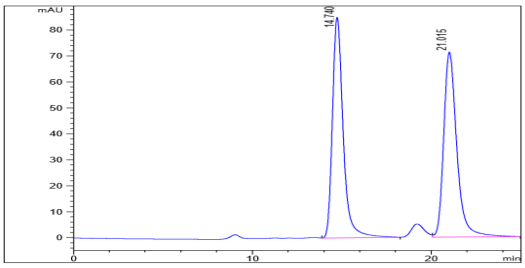
Peak #	RT [min]	Area	Area %
1	10.319	8.993e3	69.842
2	21.923	3.883e3	30.158



4e

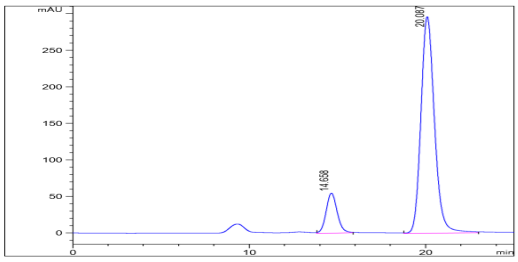
HPLC: Chiralcel OD-H, ^{i}PrOH/*n*-hexane = 20/80, flow rate = 1.0 mL/min, UV = 230 nm

Racemic

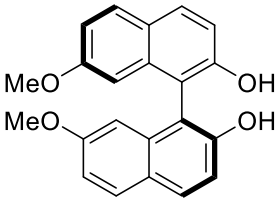


Peak #	RT [min]	Area	Area %
1	14.740	3.564e3	49.550
2	21.015	3.628e3	50.450

Enantioenriched



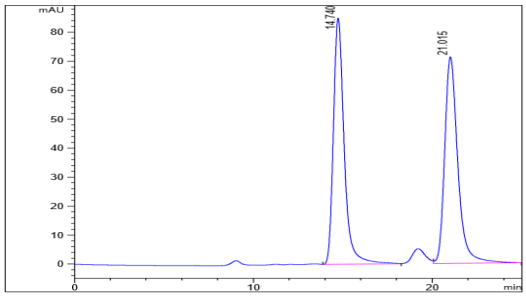
Peak #	RT [min]	Area	Area %
1	14.658	2.400e3	13.141
2	20.087	1.586e4	86.859



1e

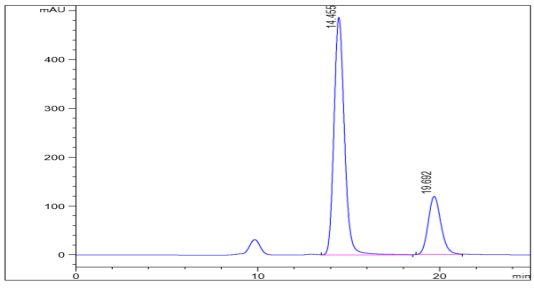
HPLC: Chiralcel OD-H, ^{i}PrOH/*n*-hexane = 20/80, flow rate = 1.0 mL/min, UV = 230 nm

Racemic

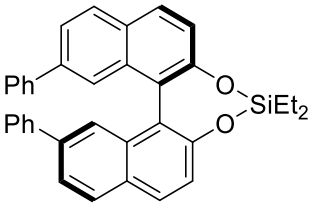


Peak #	RT [min]	Area	Area %
1	14.740	3.564e3	49.550
2	21.015	3.628e3	50.450

Enantioenriched



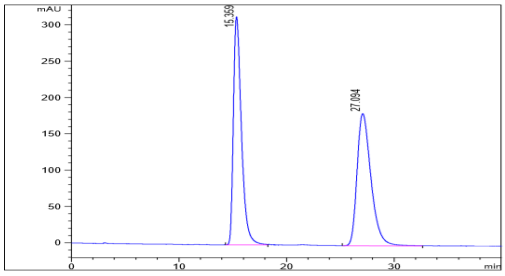
Peak #	RT [min]	Area	Area %
1	14.455	1.946e4	77.021
2	19.692	5.807e3	22.979



4f

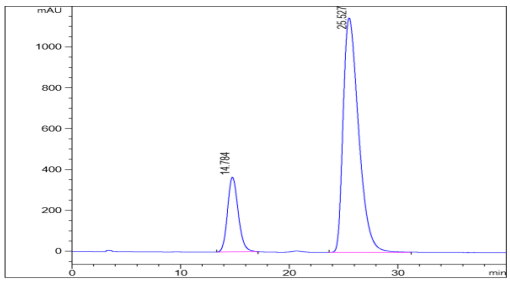
HPLC: Chiralcel OD-H, ^{i}PrOH/*n*-hexane = 10/90, flow rate = 1.0 mL/min, UV = 254 nm

Racemic

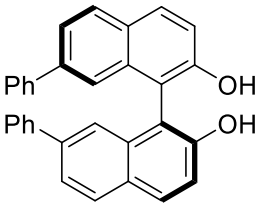


Peak #	RT [min]	Area	Area %
1	15.359	1.630e4	49.947
2	27.094	1.633e4	50.053

Enantioenriched



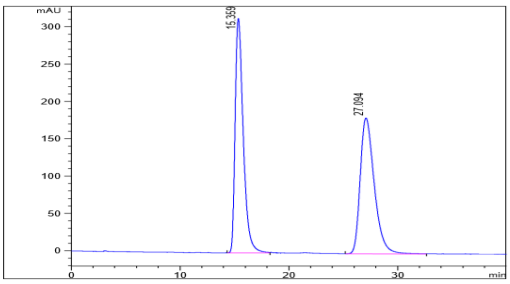
Peak #	RT [min]	Area	Area %
1	14.784	2.489e4	17.998
2	25.527	1.134e5	82.002



1f

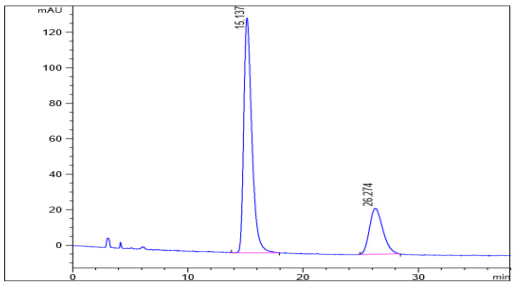
HPLC: Chiralcel OD-H, ^{i}PrOH/*n*-hexane = 10/90, flow rate = 1.0 mL/min, UV = 254 nm

Racemic

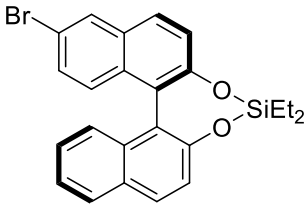


Peak #	RT [min]	Area	Area %
1	15.359	1.630e4	49.947
2	27.094	1.633e4	50.053

Enantioenriched



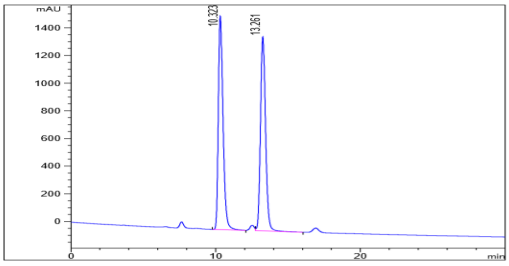
Peak #	RT [min]	Area	Area %
1	15.137	6.458e3	75.629
2	26.274	2.081e3	24.371



4g

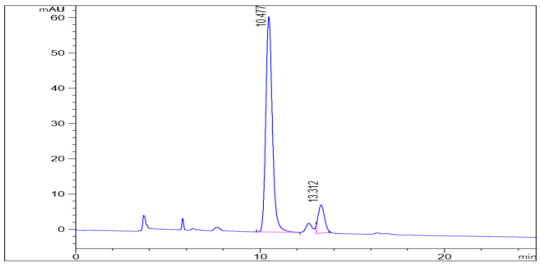
HPLC: Chiralcel IC-H, ^{i}PrOH/*n*-hexane = 5/95, flow rate = 1.0 mL/min, UV = 230 nm

Racemic

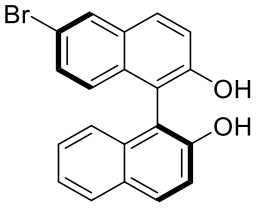


Peak #	RT [min]	Area	Area %
1	10.323	3.498e4	50.462
2	13.261	3.434e4	49.538

Enantioenriched



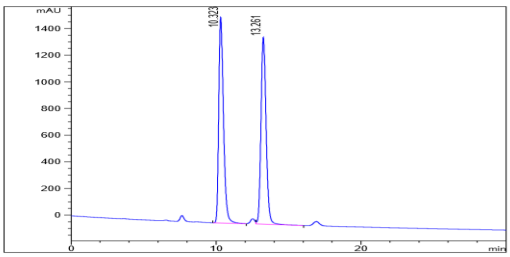
Peak #	RT [min]	Area	Area %
1	10.477	1.509e3	88.876
2	13.312	188.851	11.124



1g

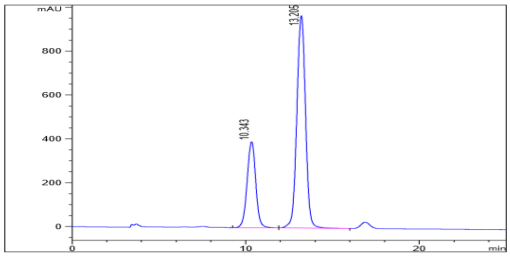
HPLC: Chiralcel IC-H, ^{i}PrOH/*n*-hexane = 5/95, flow rate = 1.0 mL/min, UV = 230 nm

Racemic

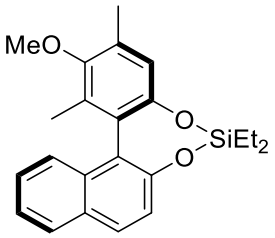


Peak #	RT [min]	Area	Area %
1	10.323	3.498e4	50.462
2	13.261	3.434e4	49.538

Enantioenriched



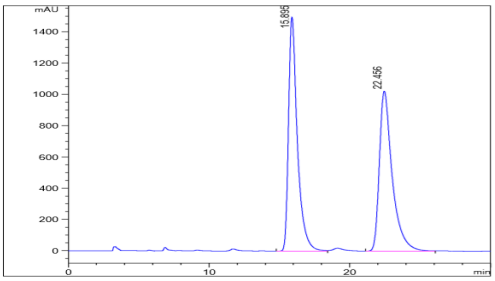
Peak #	RT [min]	Area	Area %
1	10.343	1.356e4	28.649
2	13.205	3.377e4	71.351



4h

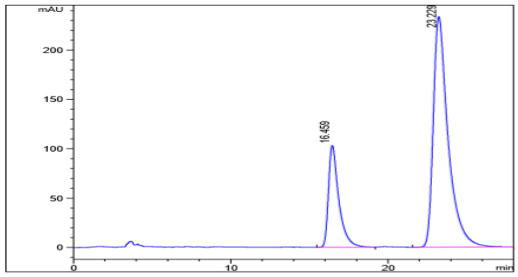
HPLC: Chiralcel AD-H, ^{i}PrOH/*n*-hexane = 20/80, flow rate = 1.0 mL/min, UV = 230 nm

Racemic

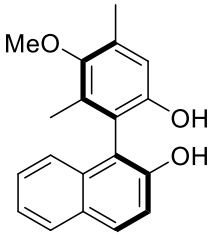


Peak #	RT [min]	Area	Area %
1	15.895	6.540e4	50.188
2	22.456	6.491e4	49.812

Enantioenriched



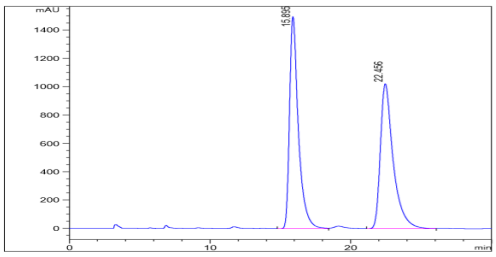
Peak #	RT [min]	Area	Area %
1	16.459	4.711e3	23.571
2	23.229	1.528e4	76.429



1h

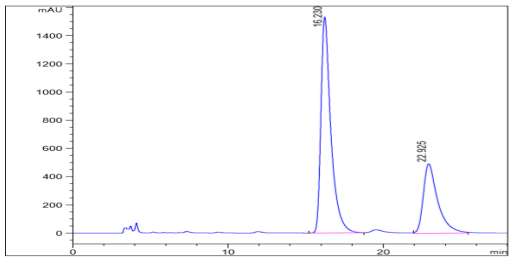
HPLC: Chiralcel AD-H, ^{i}PrOH/*n*-hexane = 20/80, flow rate = 1.0 mL/min, UV = 254 nm

Racemic

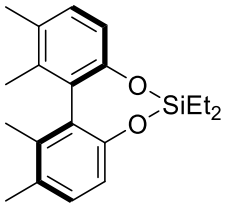


Peak #	RT [min]	Area	Area %
1	15.895	6.540e4	50.188
2	22.456	6.491e4	49.812

Enantioenriched



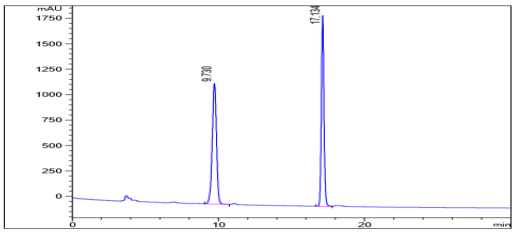
Peak #	RT [min]	Area	Area %
1	16.230	6.801e4	68.185
2	22.925	3.173e4	31.815



4i

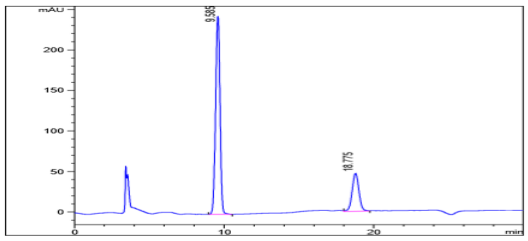
HPLC: Chiralcel IC-H, ^{i}PrOH/*n*-hexane = 5/95, flow rate = 1.0 mL/min, UV = 210 nm

Racemic

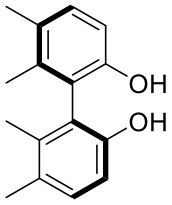


Peak #	RT [min]	Area	Area %
1	9.730	2.410e4	50.932
2	17.134	2.322e4	49.068

Enantioenriched



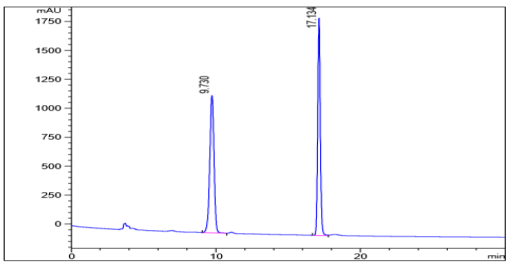
Peak #	RT [min]	Area	Area %
1	9.585	5.046e3	77.302
2	18.775	1.482e3	22.698



1i

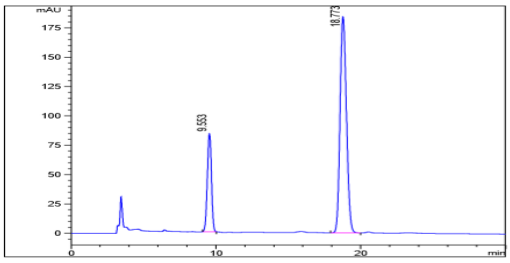
HPLC: Chiralcel IC-H, ^{i}PrOH/*n*-hexane = 5/95, flow rate = 1.0 mL/min, UV = 230 nm

Racemic

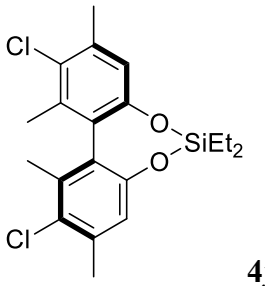


Peak #	RT [min]	Area	Area %
1	9.730	2.410e4	50.932
2	17.134	2.322e4	49.068

Enantioenriched



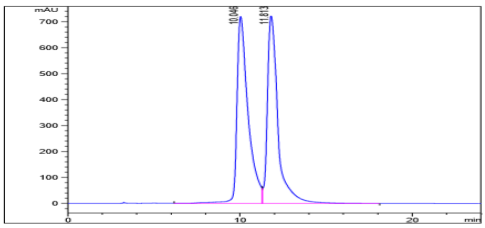
Peak #	RT [min]	Area	Area %
1	9.553	1.663e3	22.731
2	18.773	5.652e3	77.269



4j

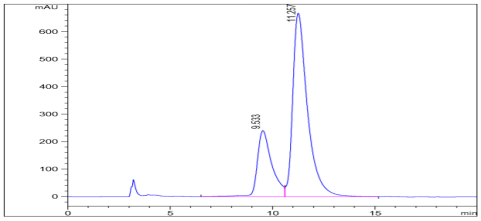
HPLC: Chiralcel OJ-H, ^{i}PrOH/*n*-hexane = 5/95, flow rate = 1.0 mL/min, UV = 230 nm

Racemic

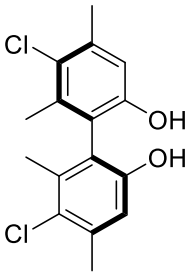


Peak #	RT [min]	Area	Area %
1	10.046	3.267e4	51.576
2	11.813	3.067e4	48.424

Enantioenriched



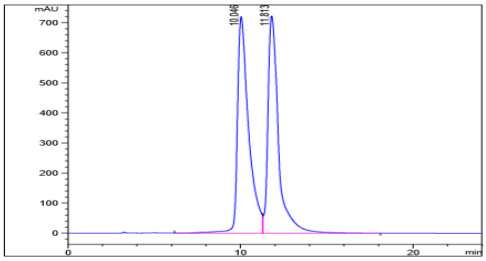
Peak #	RT [min]	Area	Area %
1	9.533	1.187e4	26.247
2	11.257	3.336e4	73.753



1j

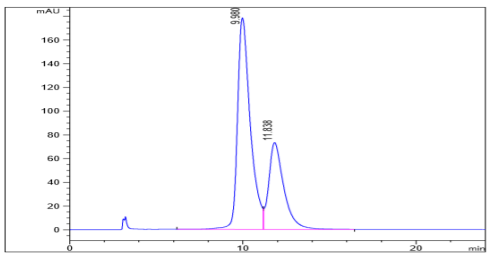
HPLC: Chiralcel OJ-H, ^{i}PrOH/*n*-hexane = 5/95, flow rate = 1.0 mL/min, UV = 210 nm

Racemic

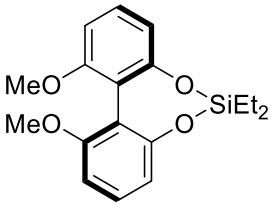


Peak #	RT [min]	Area	Area %
1	10.046	3.267e4	51.576
2	11.813	3.067e4	48.424

Enantioenriched



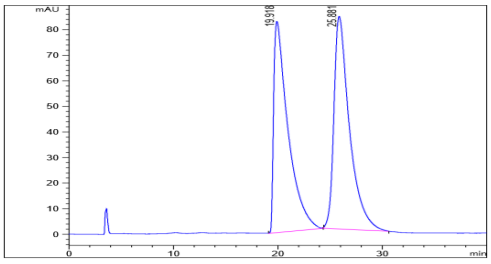
Peak #	RT [min]	Area	Area %
1	9.980	9.173e3	67.411
2	11.838	4.435e3	32.589



4k

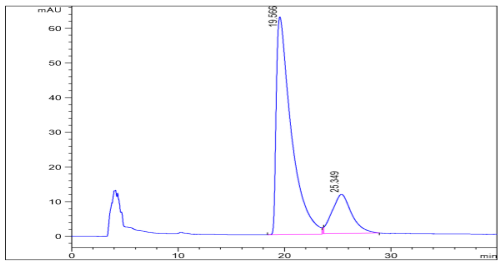
HPLC: Chiralcel OJ-H, ^{i}PrOH/*n*-hexane = 5/95, flow rate = 1.0 mL/min, UV = 230 nm

Racemic

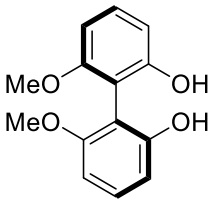


Peak #	RT [min]	Area	Area %
1	19.918	7.918e3	47.919
2	25.881	8.606e3	52.081

Enantioenriched



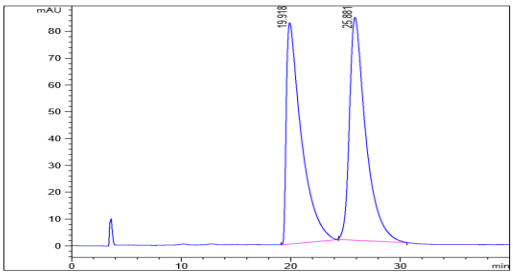
Peak #	RT [min]	Area	Area %
1	19.566	6.176e3	81.156
2	25.349	1.434e3	18.844



1k

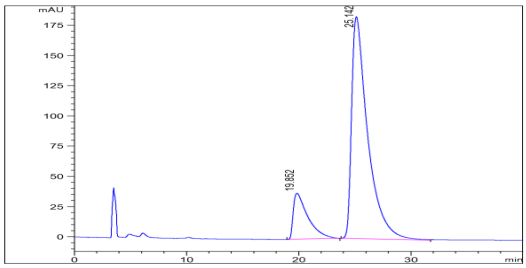
HPLC: Chiralcel OJ-H, ^{i}PrOH/*n*-hexane = 5/95, flow rate = 1.0 mL/min, UV = 230 nm

Racemic

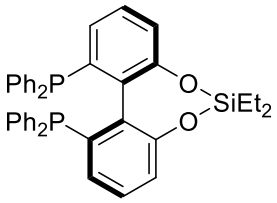


Peak #	RT [min]	Area	Area %
1	19.918	7.918e3	47.919
2	25.881	8.606e3	52.081

Enantioenriched



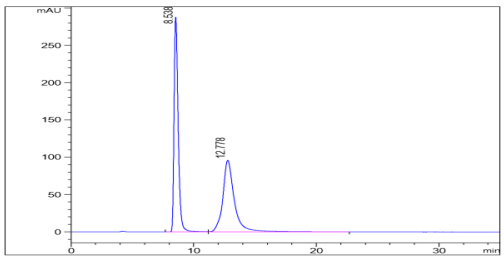
Peak #	RT [min]	Area	Area %
1	19.852	3.436e3	15.798
2	25.142	1.831e4	84.202



4l

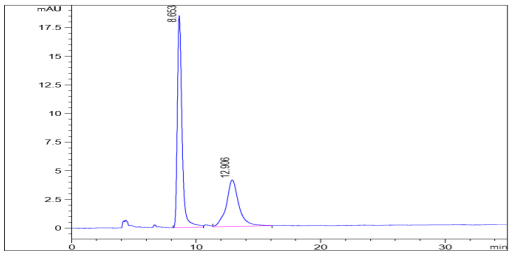
HPLC: Chiralcel OD-H, ^{i}PrOH/*n*-hexane = 15/85, flow rate = 1.0 mL/min, UV = 254 nm

Racemic

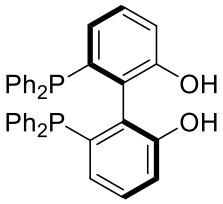


Peak #	RT [min]	Area	Area %
1	8.538	6.708e3	51.587
2	12.778	6.295e3	48.413

Enantioenriched



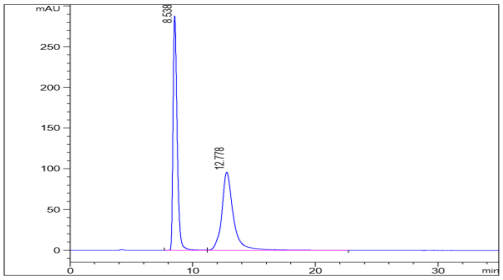
Peak #	RT [min]	Area	Area %
1	8.653	480.467	62.942
2	12.906	282.878	37.058



1l

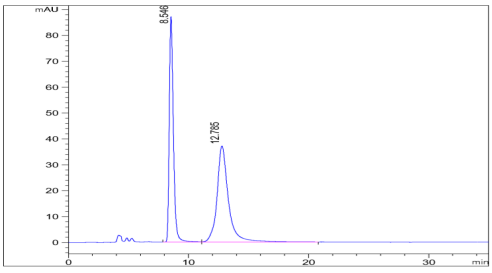
HPLC: Chiralcel OD-H, ^{i}PrOH/*n*-hexane = 15/95, flow rate = 1.0 mL/min, UV = 254 nm

Racemic

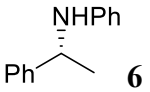


Peak #	RT [min]	Area	Area %
1	8.538	6.708e3	51.587
2	12.778	6.295e3	48.413

Enantioenriched



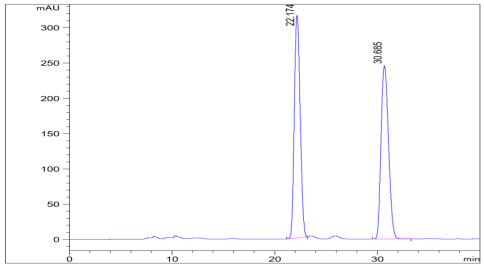
Peak #	RT [min]	Area	Area %
1	8.546	2.066e3	45.869
2	12.785	2.438e3	54.131



6

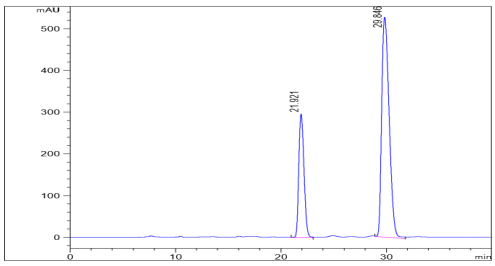
HPLC: Chiralcel OD-H, ^{i}PrOH/*n*-hexane = 2/98, flow rate = 0.5 mL/min, UV = 254 nm

Racemic

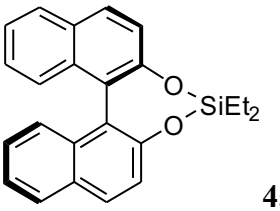


Peak #	RT [min]	Area	Area %
1	22.174	1.252e4	50.152
2	30.685	1.245e4	49.848

Enantioenriched



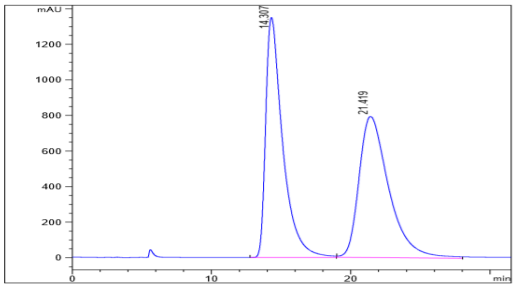
Peak #	RT [min]	Area	Area %
1	21.921	1.015e4	28.185
2	29.846	2.586e4	71.815



4a

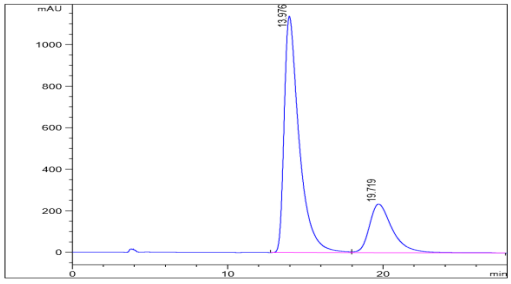
HPLC: Chiralcel OJ-H, ^{i}PrOH/*n*-hexane = 20/80, flow rate = 1.0 mL/min, UV = 230 nm

Racemic

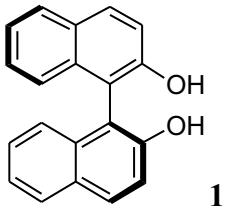


Peak #	RT [min]	Area	Area %
1	14.307	1.144e5	49.237
2	21.419	1.180e5	50.763

Chiral



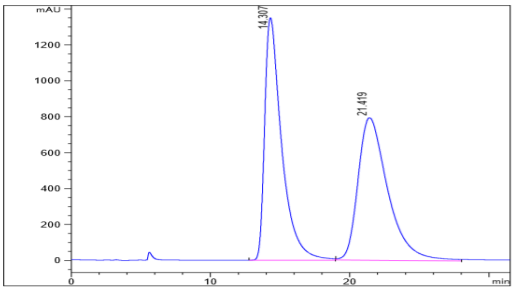
Peak #	RT [min]	Area	Area %
1	13.976	7.637e4	76.087
2	19.719	2.400e4	23.913



1a

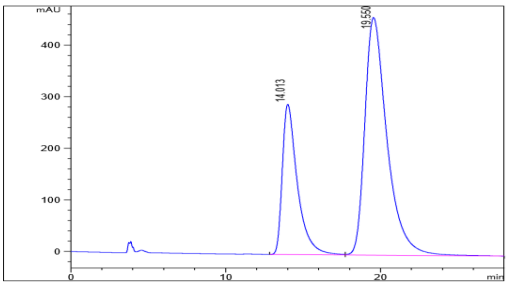
HPLC: Chiralcel OJ-H, ^{i}PrOH/*n*-hexane = 20/80, flow rate = 1.0 mL/min, UV = 230 nm

Racemic



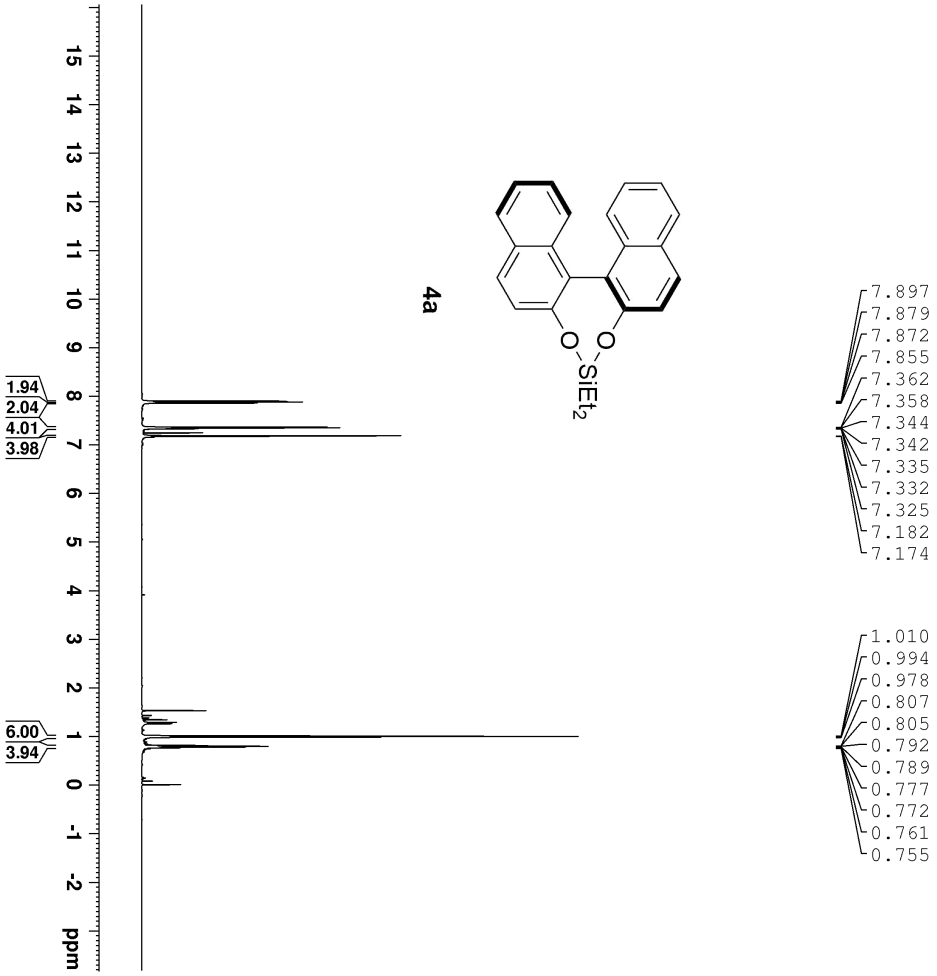
Peak #	RT [min]	Area	Area %
1	14.307	1.144e5	49.237
2	21.419	1.180e5	50.763

Chiral

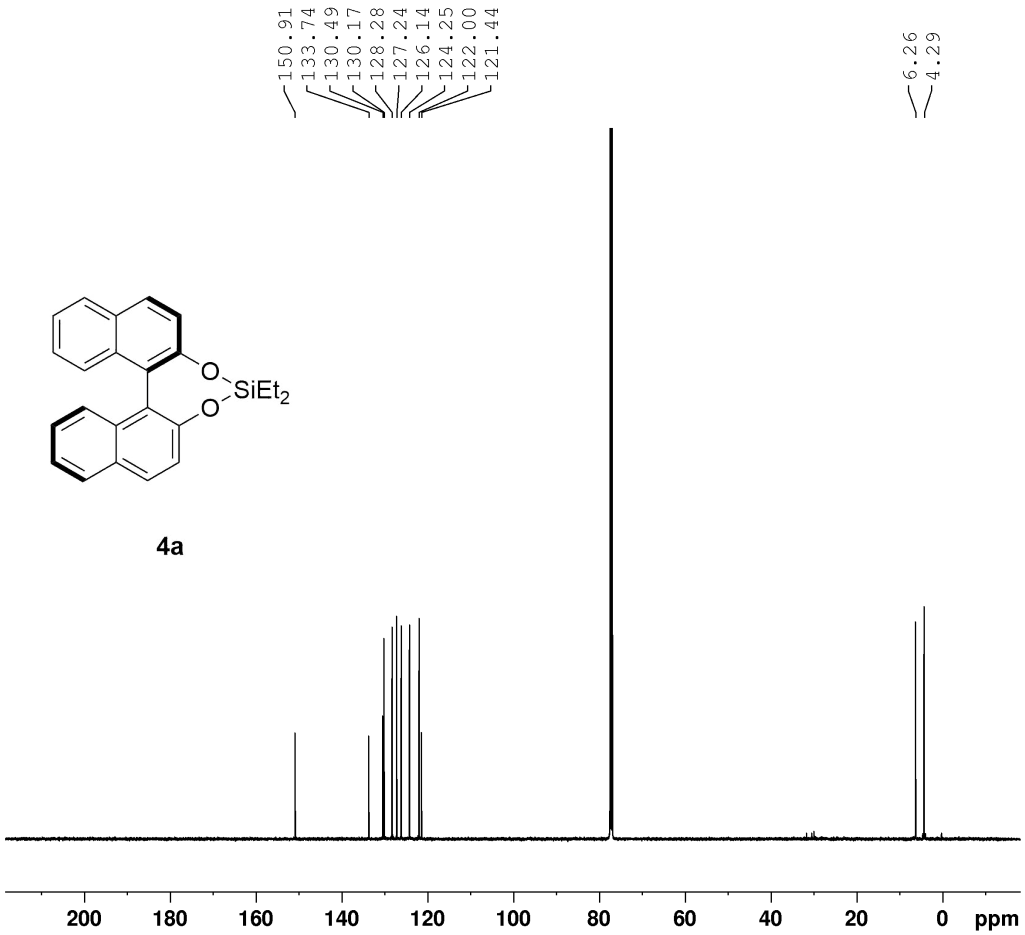


Peak #	RT [min]	Area	Area %
1	14.013	1.973e4	30.066
2	19.550	4.588e4	69.934

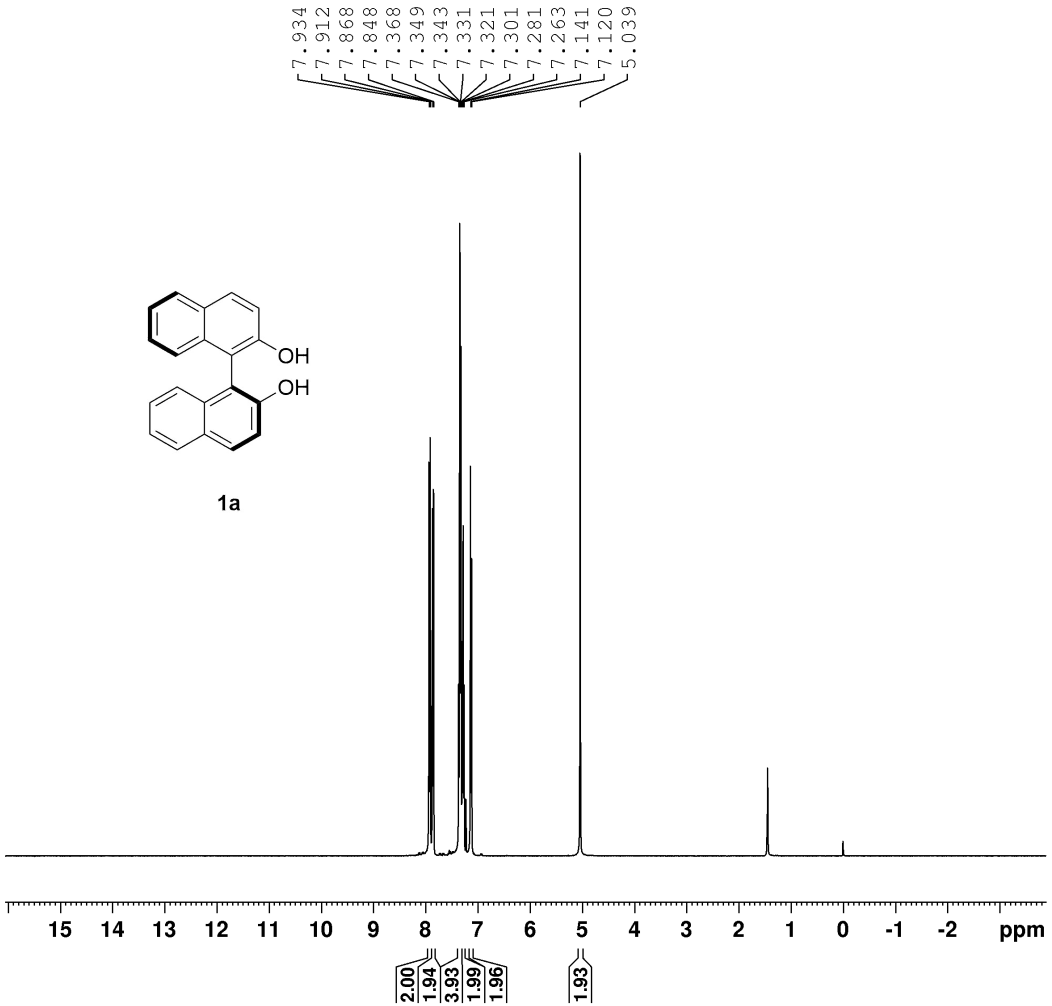
Copies of the NMR spectra



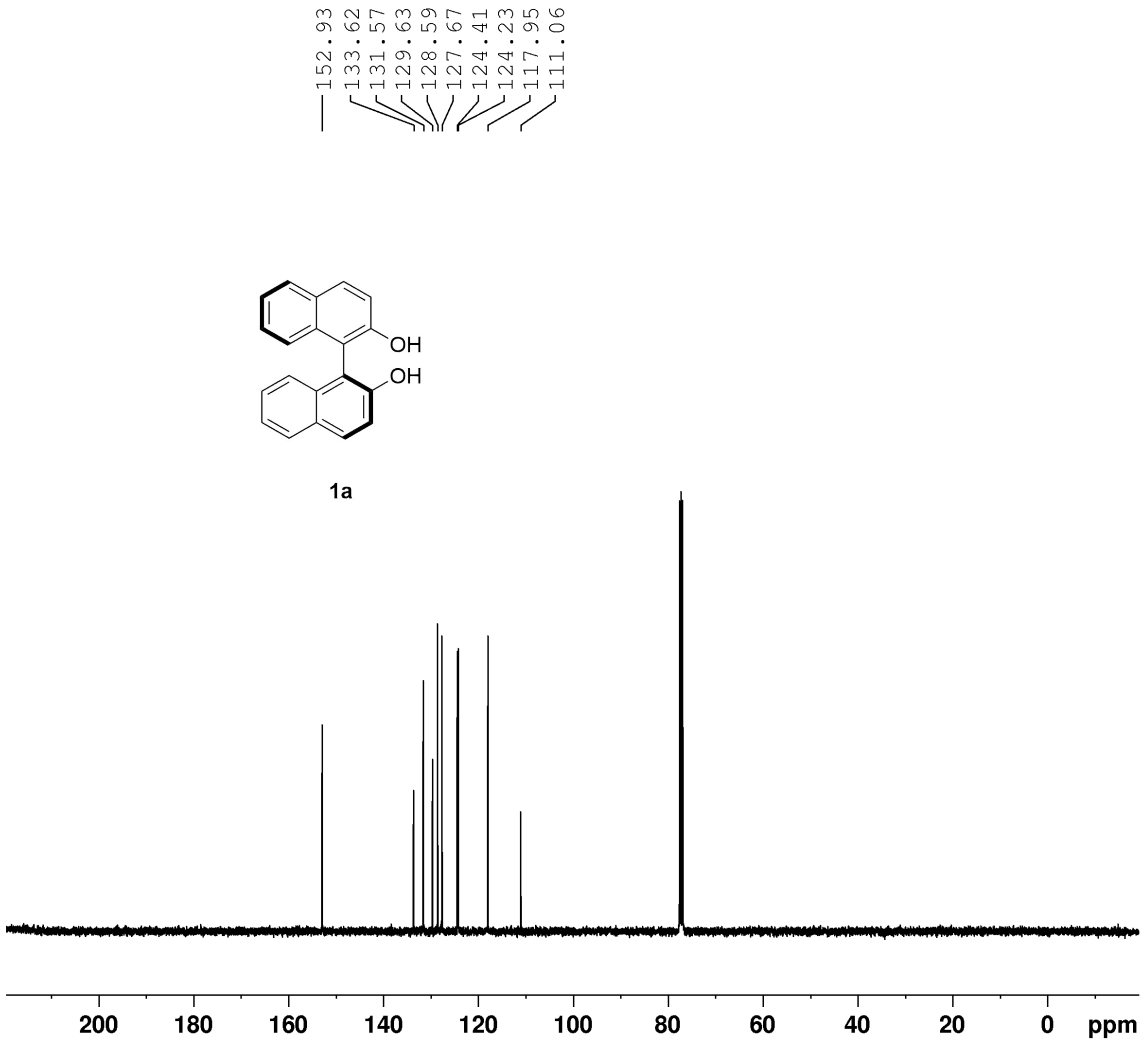

```
Current Data Parameters
NAME                  2024-500
EXPNO                       40
PROCNO                       1

F2 - Acquisition Parameters
Date_                20240423
Time                     0.35 h
INSTRUM                  spect
PROBHD     5 mm CPPBBO BB
PULPROG                  zg30
TD                      65536
SOLVENT                 CDCl3
NS                         16
DS                          2
SWH                 10000.000 Hz
FIDRES                  0.305176 Hz
AQ                  3.2767999 sec
RG                       55.37
DW                     50.000 usec
DE                      6.50 usec
TE                     298.0 K
D1                  1.00000000 sec
TD0                         1
SFO1          500.1330885 MHz
NUC1                       1H
P1                     11.85 usec
PLW1           20.00000000 W

F2 - Processing parameters
SI                      65536
SF          500.1300215 MHz
WDW                        EM
SSB                         0
LB                       0.30 Hz
GB                          0
PC                       1.00
```



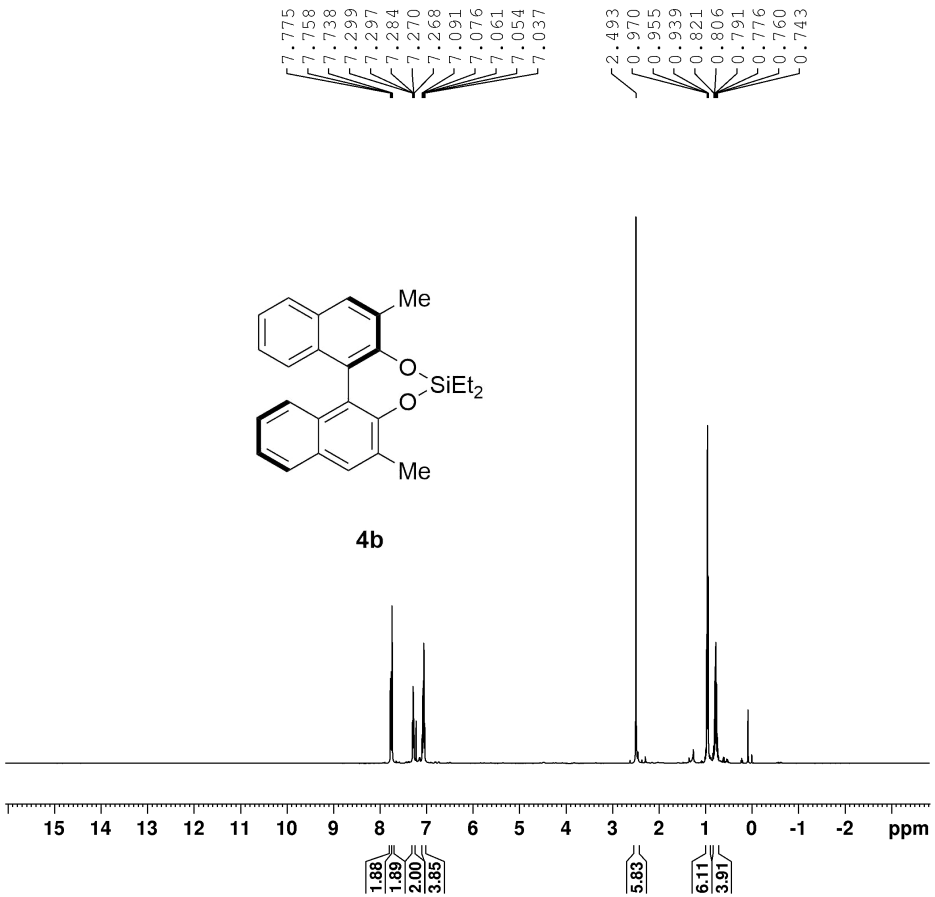

```
Current Data Parameters
NAME          ÐÂ½¨ÎÄ¼þ¼Ð
EXPNO                 41
PROCNO                 1

F2 - Acquisition Parameters
Date_           20240423
Time                1.02 h
INSTRUM             spect
PROBHD   5 mm CPPBBO BB
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   500
DS                     4
SWH            29761.904 Hz
FIDRES          0.908261 Hz
AQ             1.1010048 sec
RG                192.89
DW                16.800 usec
DE                 18.00 usec
TE                 298.0 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                    1
SFO1        125.7703637 MHz
NUC1                13C
P1                 10.75 usec
PLW1         57.00000000 W
SFO2        500.1320005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2              80.00 usec
PLW2         20.00000000 W
PLW12         0.43882000 W
PLW13         0.28084999 W

F2 - Processing parameters
SI                 32768
SF          125.7577649 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```



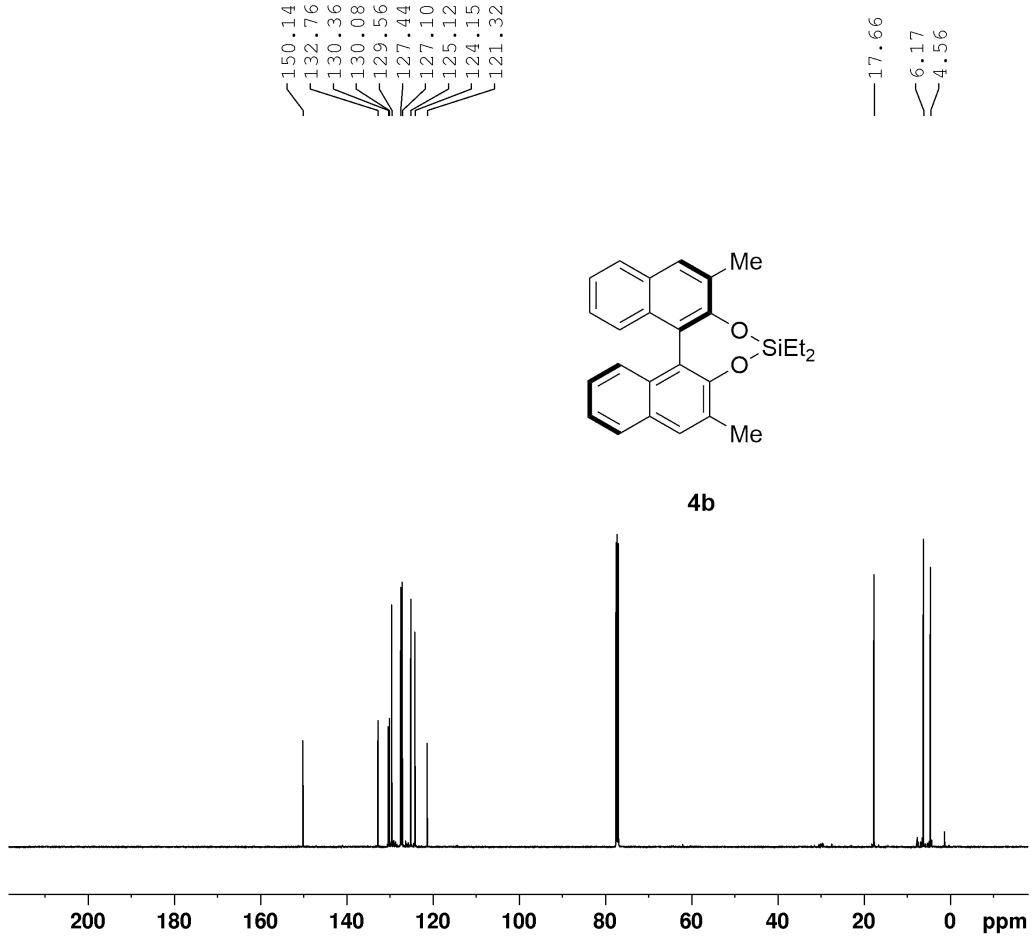

```
Current Data Parameters
NAME            2024-400
EXPNO                131
PROCNO                 1

F2 - Acquisition Parameters
Date_           20240718
Time               14.50 h
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                     4
DS                     0
SWH             8012.820 Hz
FIDRES          0.489064 Hz
AQ             2.0447233 sec
RG                 92.09
DW                62.400 usec
DE                 6.50 usec
TE                299.1 K
D1            2.00000000 sec
TD0                    1
SFO1         400.2424716 MHz
NUC1                  1H
P1                14.30 usec
PLW1         12.00000000 W

F2 - Processing parameters
SI                 65536
SF           400.2400233 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                 1.00
```



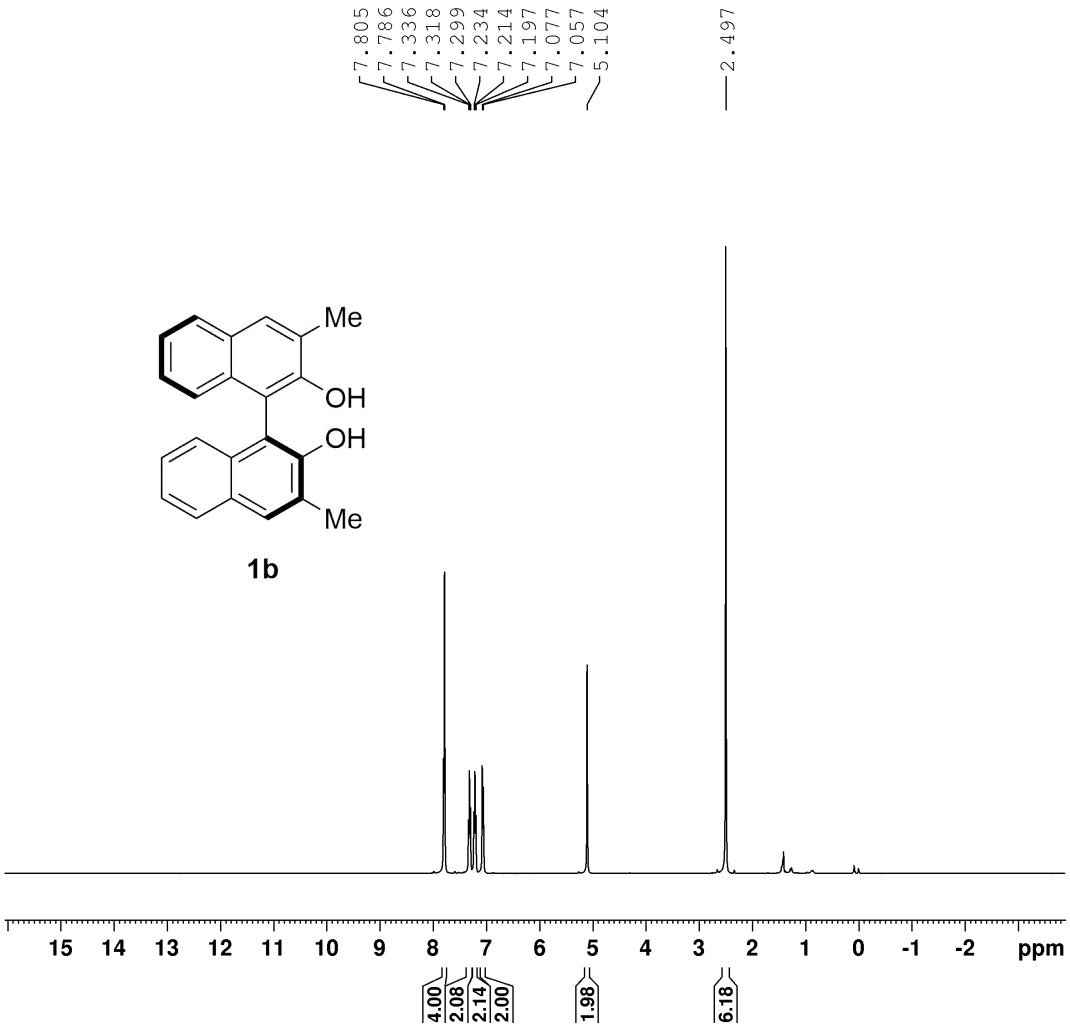

```
Current Data Parameters
NAME            2024-400
EXPNO                132
PROCNO                 1

F2 - Acquisition Parameters
Date_           20240718
Time               15.08 h
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG           zgpg30
TD                 65536
SOLVENT             CDCl3
NS                    300
DS                      4
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ              1.3631488 sec
RG                206.33
DW                20.800 usec
DE                 6.50 usec
TE                299.8 K
D1             2.00000000 sec
d11            0.03000000 sec
DELTA          1.89999998 sec
TD0                    1
SFO1         100.6504916 MHz
NUC1                 13C
P1                10.00 usec
PLW1          54.00000000 W
SFO2         400.2416010 MHz
NUC2                 1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2          12.00000000 W
PLW12          0.30294999 W
PLW13          0.24539000 W

F2 - Processing parameters
SI                 32768
SF           100.6404119 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```



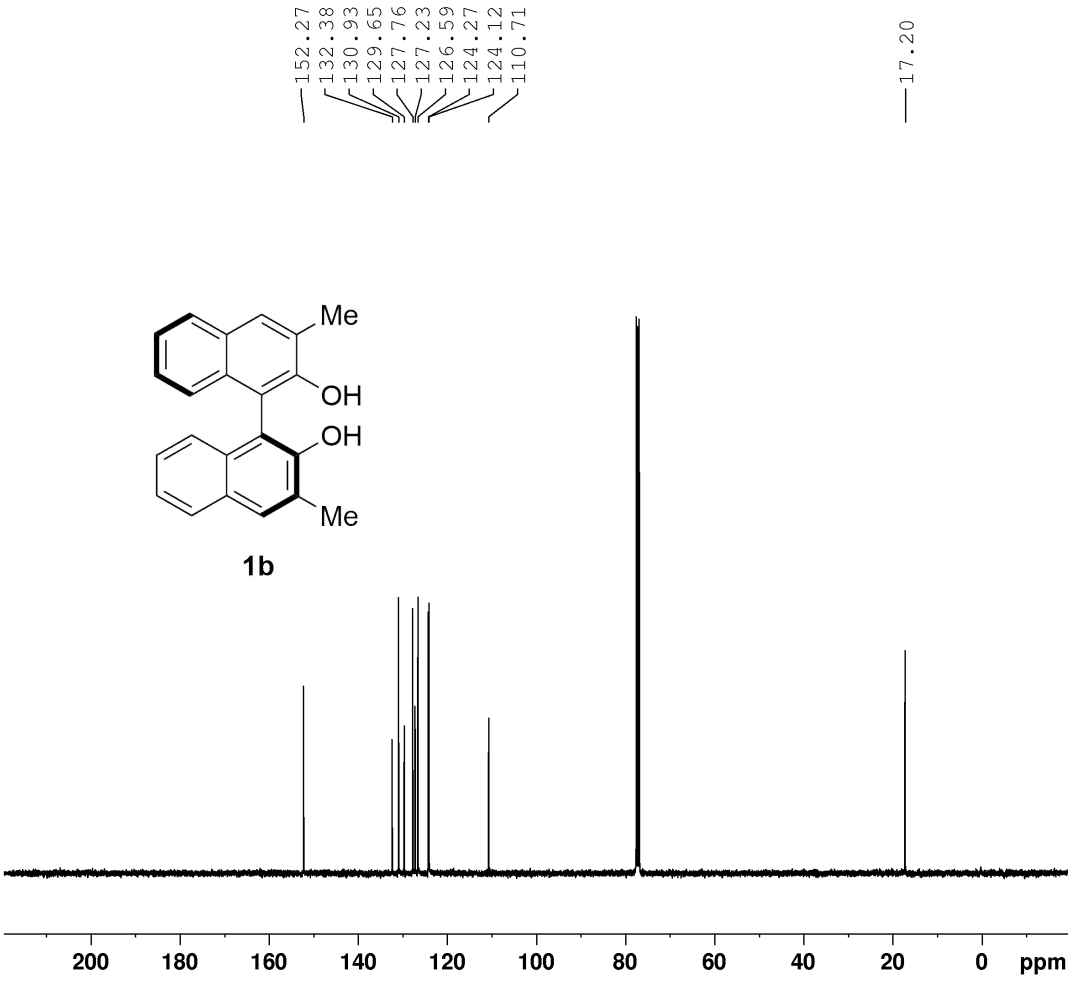

```
Current Data Parameters
NAME            2024-500
EXPNO                 59
PROCNO                 1

F2 - Acquisition Parameters
Date_           20240604
Time                7.24 h
INSTRUM            spect
PROBHD   5 mm CPPBBO BB
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     2
SWH            10000.000 Hz
FIDRES          0.305176 Hz
AQ              3.2767999 sec
RG                 31.72
DW                 50.000 usec
DE                  6.50 usec
TE                 298.0 K
D1             1.00000000 sec
TD0                    1
SFO1         500.1330885 MHz
NUC1                  1H
P1                 11.85 usec
PLW1         20.00000000 W

F2 - Processing parameters
SI                 65536
SF           500.1300310 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



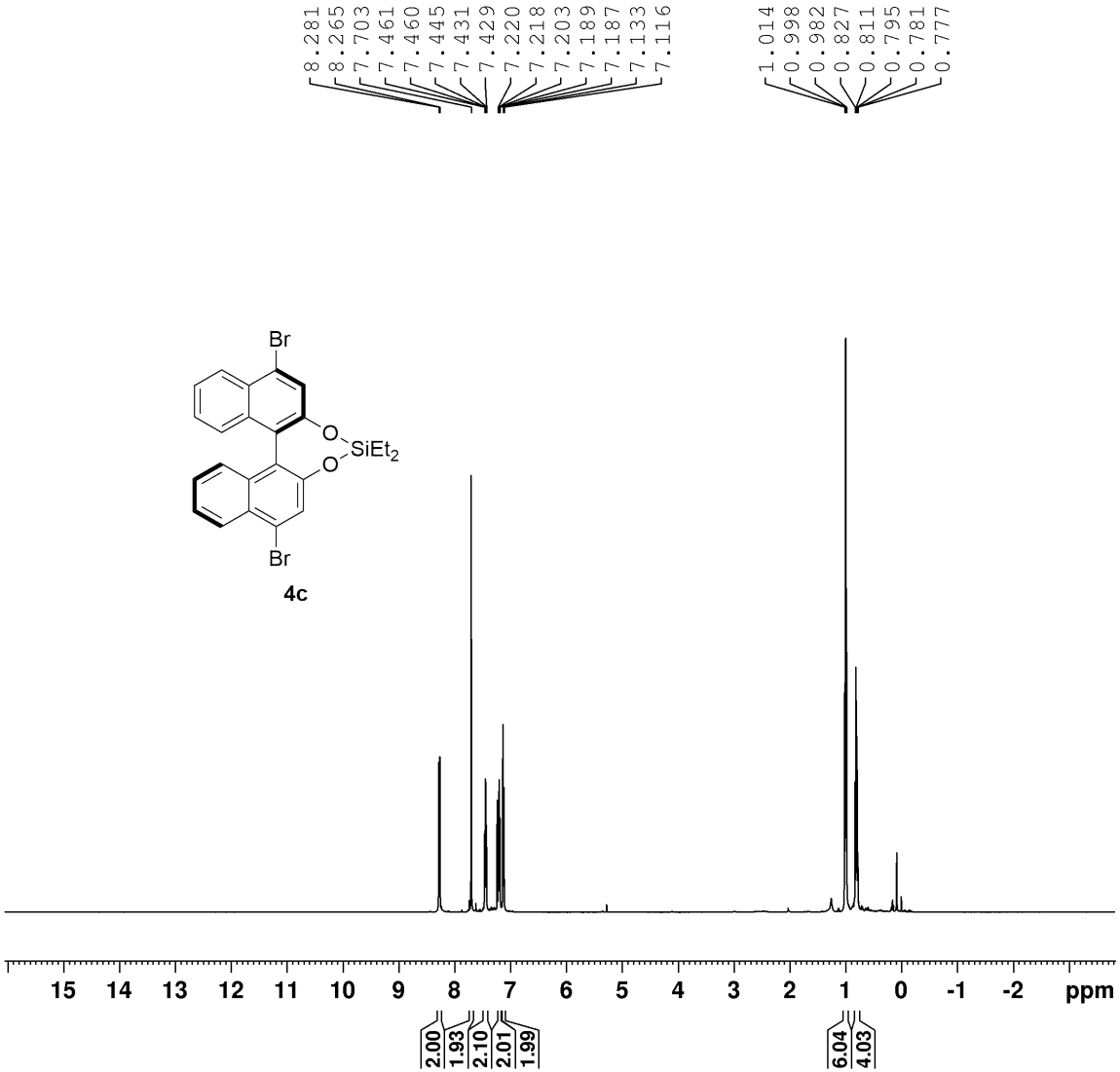

```
Current Data Parameters
NAME            2024-500
EXPNO                 60
PROCNO                 1

F2 - Acquisition Parameters
Date_           20240604
Time                7.51 h
INSTRUM             spect
PROBHD   5 mm CPPBBO BB
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                    500
DS                      4
SWH             29761.904 Hz
FIDRES          0.908261 Hz
AQ              1.1010048 sec
RG                 192.89
DW                 16.800 usec
DE                 18.00 usec
TE                 298.0 K
D1              2.00000000 sec
d11             0.03000000 sec
DELTA           1.89999998 sec
TD0                     1
SFO1         125.7703637 MHz
NUC1                13C
P1                 10.75 usec
PLW1         57.00000000 W
SFO2         500.1320005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2              80.00 usec
PLW2         20.00000000 W
PLW12         0.43882000 W
PLW13         0.28084999 W

F2 - Processing parameters
SI                  32768
SF           125.7577677 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```



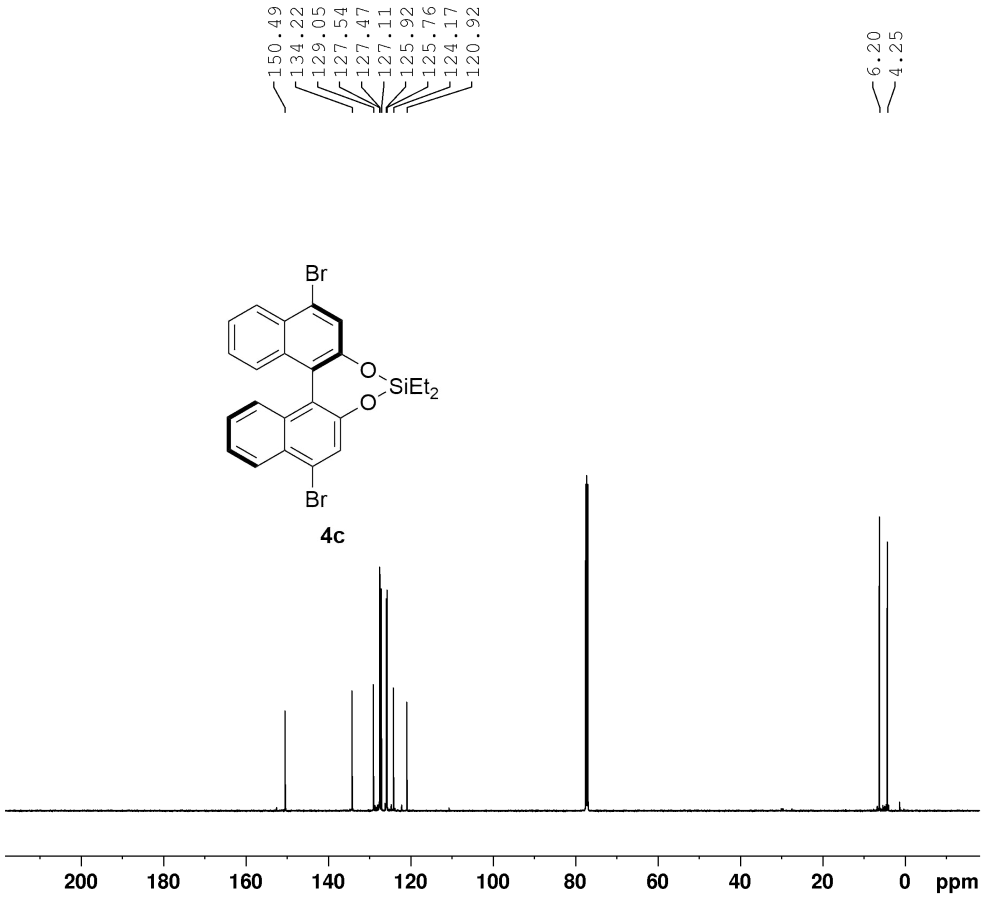

```
Current Data Parameters
NAME            2024-400
EXPNO                135
PROCNO                 1

F2 - Acquisition Parameters
Date_           20240718
Time               17.29 h
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     0
SWH              8012.820 Hz
FIDRES           0.489064 Hz
AQ               2.0447233 sec
RG                 92.09
DW                62.400 usec
DE                 6.50 usec
TE                299.1 K
D1            2.00000000 sec
TD0                    1
SFO1         400.2424716 MHz
NUC1                  1H
P1                14.30 usec
PLW1         12.00000000 W

F2 - Processing parameters
SI                 65536
SF           400.2400244 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                 1.00
```



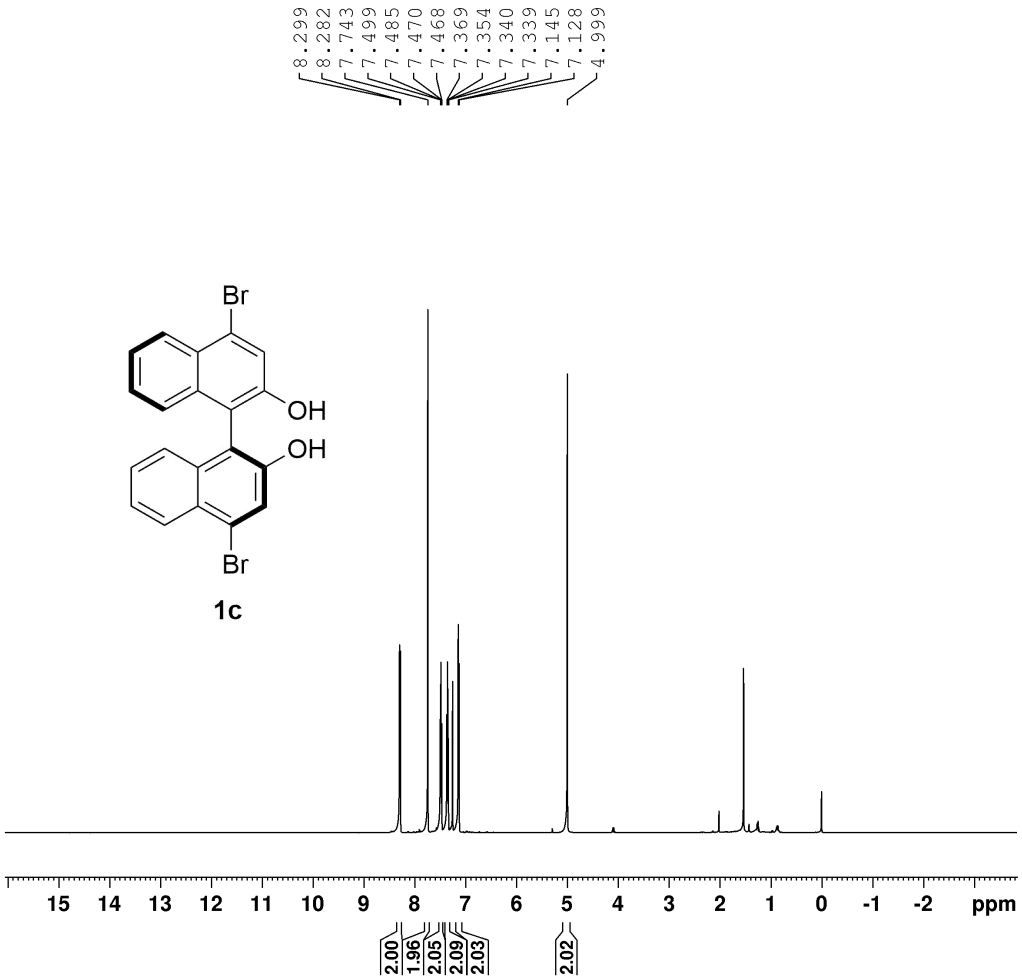

```
Current Data Parameters
NAME            2024-400
EXPNO                136
PROCNO                 1

F2 - Acquisition Parameters
Date_           20240718
Time               18.28 h
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1024
DS                     4
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ             1.3631488 sec
RG                206.33
DW                20.800 usec
DE                 6.50 usec
TE                299.6 K
D1             2.00000000 sec
d11            0.03000000 sec
DELTA          1.89999998 sec
TD0                    1
SFO1         100.6504916 MHz
NUC1                13C
P1                10.00 usec
PLW1          54.00000000 W
SFO2         400.2416010 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2          12.00000000 W
PLW12          0.30294999 W
PLW13          0.24539000 W

F2 - Processing parameters
SI                 32768
SF           100.6404110 MHz
WDW                   EM
SSB                    0
LB                 1.00 Hz
GB                     0
PC                 1.40
```



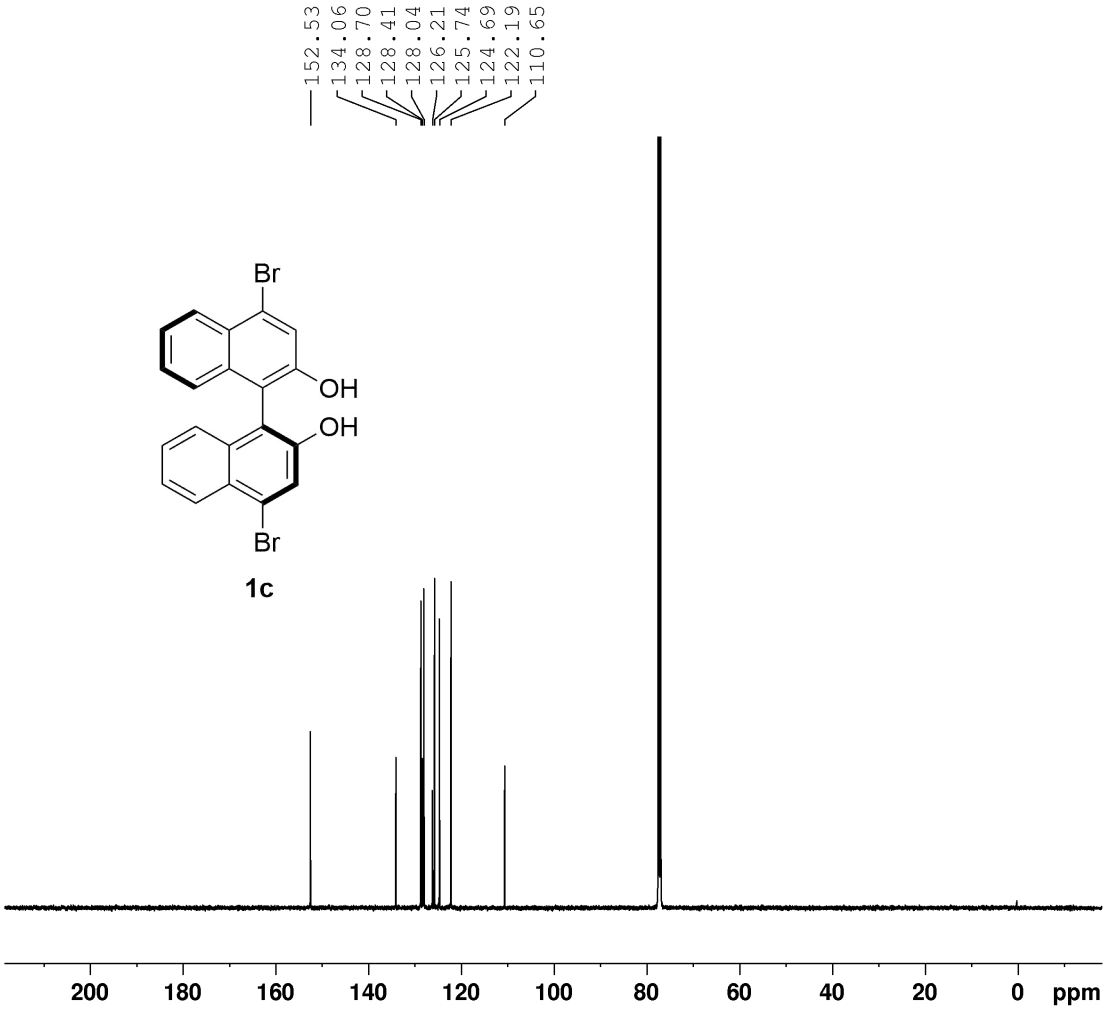

```
Current Data Parameters
NAME            2024-500
EXPNO                 55
PROCNO                 1

F2 - Acquisition Parameters
Date_           20240604
Time                6.23 h
INSTRUM            spect
PROBHD   5 mm CPPBBO BB
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     2
SWH            10000.000 Hz
FIDRES          0.305176 Hz
AQ              3.2767999 sec
RG                 49.27
DW                50.000 usec
DE                 6.50 usec
TE                298.0 K
D1             1.00000000 sec
TD0                    1
SFO1         500.1330885 MHz
NUC1                  1H
P1                 11.85 usec
PLW1         20.00000000 W

F2 - Processing parameters
SI                 65536
SF           500.1300240 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



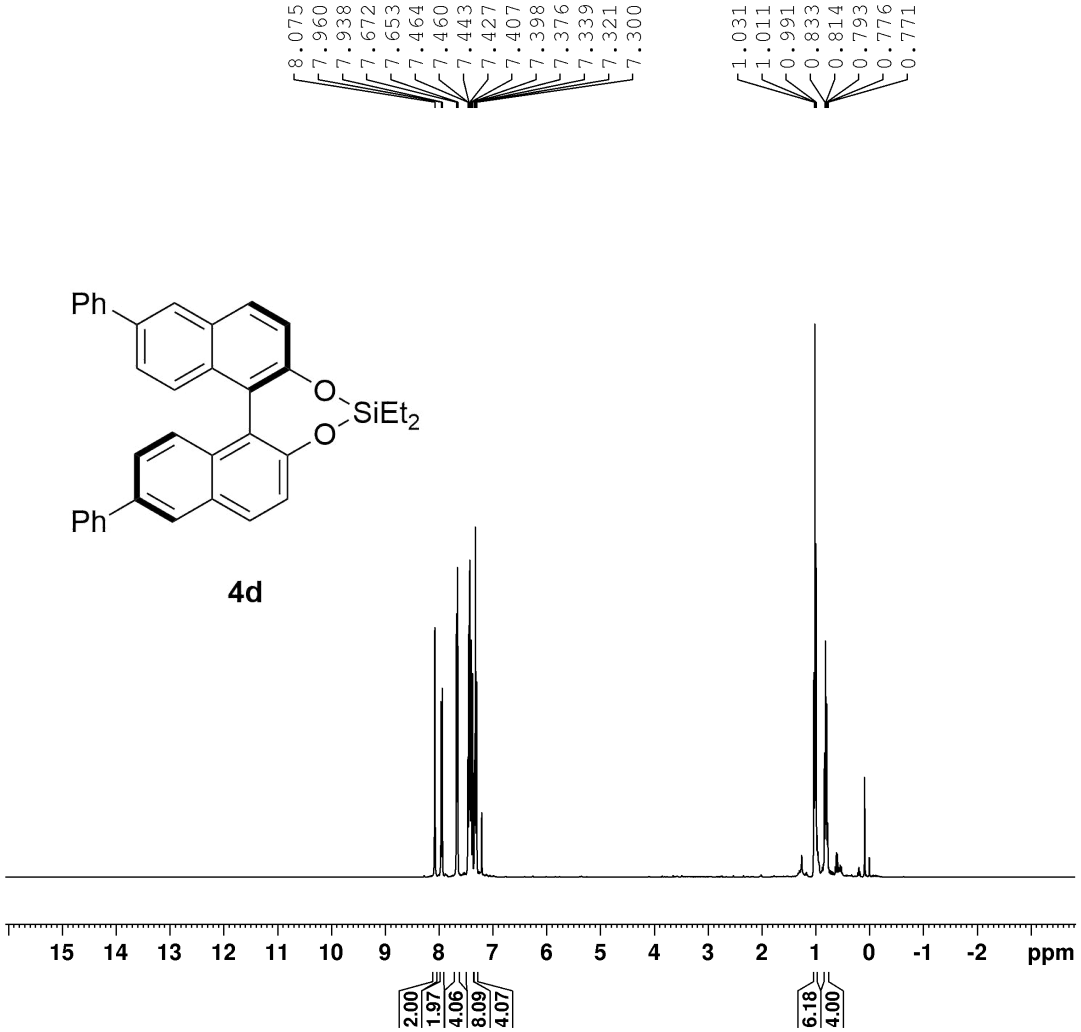

```
Current Data Parameters
NAME            2024-500
EXPNO                 56
PROCNO                 1

F2 - Acquisition Parameters
Date_           20240604
Time                6.50 h
INSTRUM            spect
PROBHD   5 mm CPPBBO BB
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   500
DS                     4
SWH            29761.904 Hz
FIDRES          0.908261 Hz
AQ             1.1010048 sec
RG                192.89
DW                16.800 usec
DE                18.00 usec
TE                298.0 K
D1             2.00000000 sec
d11            0.03000000 sec
DELTA          1.89999998 sec
TD0                    1
SFO1         125.7703637 MHz
NUC1                13C
P1                 10.75 usec
PLW1          57.00000000 W
SFO2          500.1320005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2              80.00 usec
PLW2          20.00000000 W
PLW12          0.43882000 W
PLW13          0.28084999 W

F2 - Processing parameters
SI                 32768
SF           125.7577667 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```



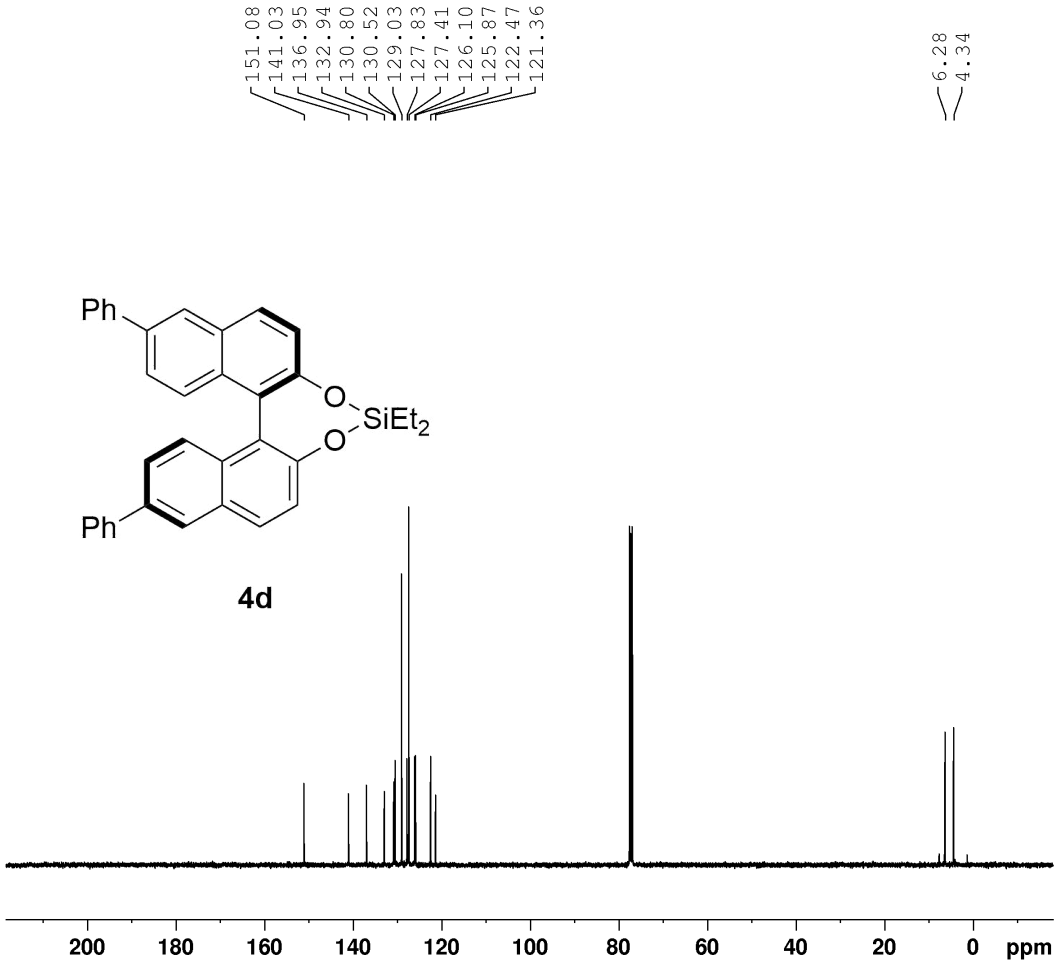

```
Current Data Parameters
NAME            2024-500
EXPNO                 23
PROCNO                 1

F2 - Acquisition Parameters
Date_           20240329
Time                2.25 h
INSTRUM            spect
PROBHD   5 mm CPPBBO BB
PULPROG             zg30
TD                 65536
SOLVENT            CDC13
NS                    16
DS                     2
SWH            10000.000 Hz
FIDRES          0.305176 Hz
AQ             3.2767999 sec
RG                 77.18
DW                50.000 usec
DE                 6.50 usec
TE                298.0 K
D1            1.00000000 sec
TD0                    1
SFO1         500.1330885 MHz
NUC1                  1H
P1                11.85 usec
PLW1         20.00000000 W

F2 - Processing parameters
SI                 65536
SF           500.1300154 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



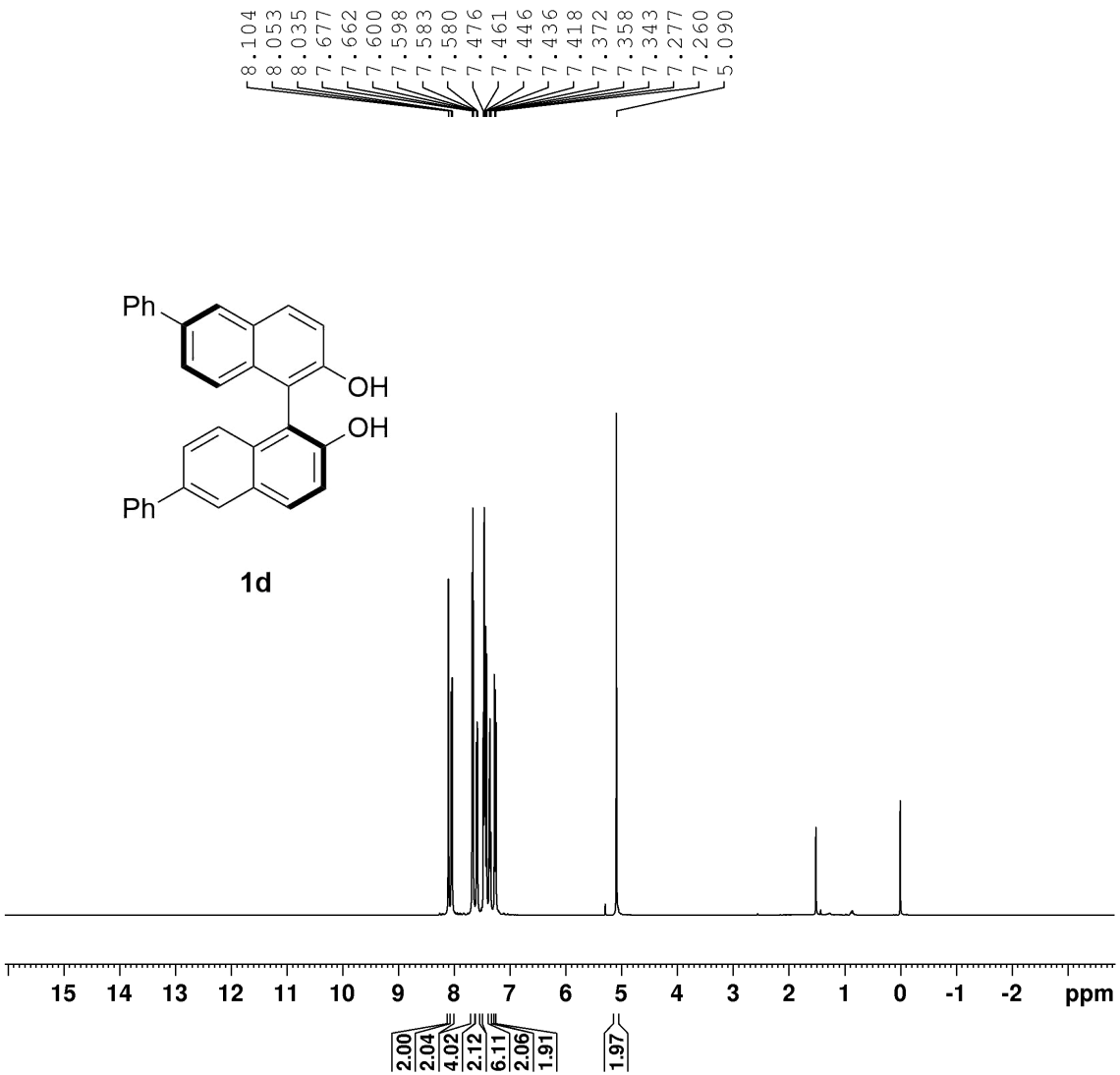

```
Current Data Parameters
NAME            2024-500
EXPNO                 24
PROCNO                 1

F2 - Acquisition Parameters
Date_           20240329
Time                3.20 h
INSTRUM            spect
PROBHD   5 mm CPPBBO BB
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1024
DS                     4
SWH            29761.904 Hz
FIDRES          0.908261 Hz
AQ             1.1010048 sec
RG                192.89
DW                16.800 usec
DE                18.00 usec
TE                298.0 K
D1             2.00000000 sec
d11            0.03000000 sec
DELTA          1.89999998 sec
TD0                    1
SFO1         125.7703637 MHz
NUC1                13C
P1                 10.75 usec
PLW1          57.00000000 W
SFO2         500.1320005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2              80.00 usec
PLW2          20.00000000 W
PLW12          0.43882000 W
PLW13          0.28084999 W

F2 - Processing parameters
SI                 32768
SF           125.7577640 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```



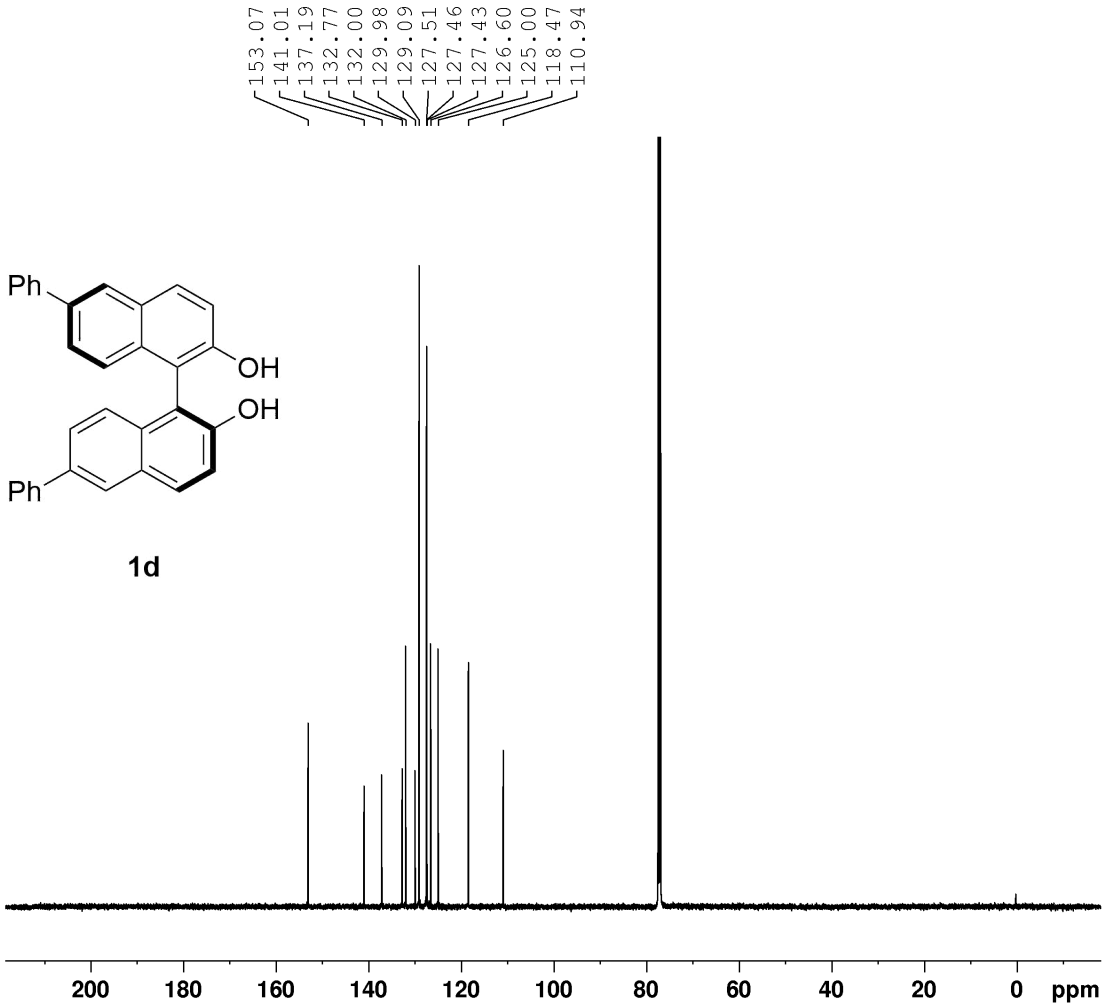

```
Current Data Parameters
NAME            2024-400
EXPNO                102
PROCNO                 1

F2 - Acquisition Parameters
Date_           20240531
Time                0.31 h
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 32768
SOLVENT            CDC13
NS                     8
DS                     0
SWH              8012.820 Hz
FIDRES           0.489064 Hz
AQ             2.0447233 sec
RG                  73.9
DW                62.400 usec
DE                 6.50 usec
TE                298.7 K
D1            2.00000000 sec
TD0                    1
SFO1         400.2424716 MHz
NUC1                  1H
P1                 14.30 usec
PLW1         12.00000000 W

F2 - Processing parameters
SI                 65536
SF           400.2400319 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



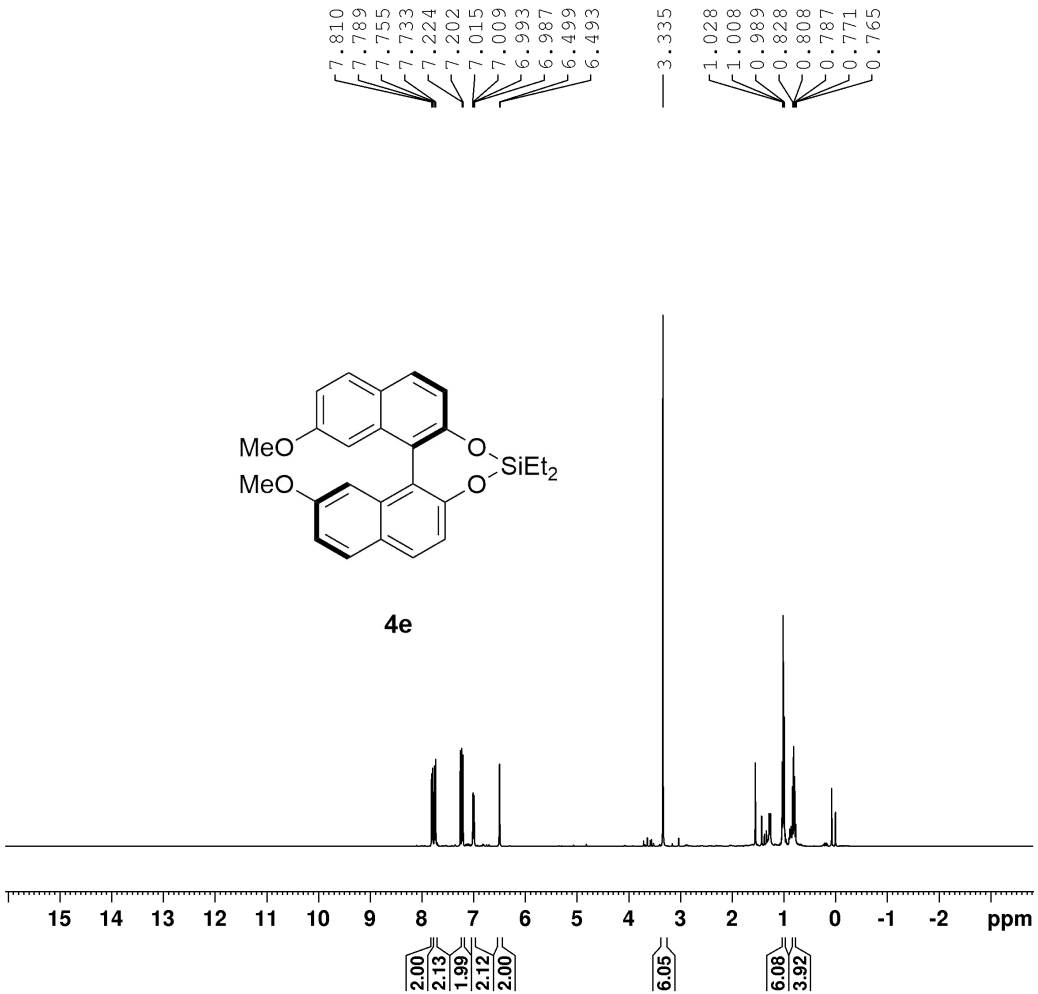

```
Current Data Parameters
NAME            2024-400
EXPNO                103
PROCNO                 1

F2 - Acquisition Parameters
Date_           20240531
Time                1.00 h
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  500
DS                    4
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ             1.3631488 sec
RG                206.33
DW                20.800 usec
DE                 6.50 usec
TE                299.3 K
D1             2.00000000 sec
d11            0.03000000 sec
DELTA          1.89999998 sec
TD0                    1
SFO1         100.6504916 MHz
NUC1                 13C
P1                10.00 usec
PLW1          54.00000000 W
SFO2         400.2416010 MHz
NUC2                 1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2          12.00000000 W
PLW12          0.30294999 W
PLW13          0.24539000 W

F2 - Processing parameters
SI                 32768
SF           100.6404129 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```



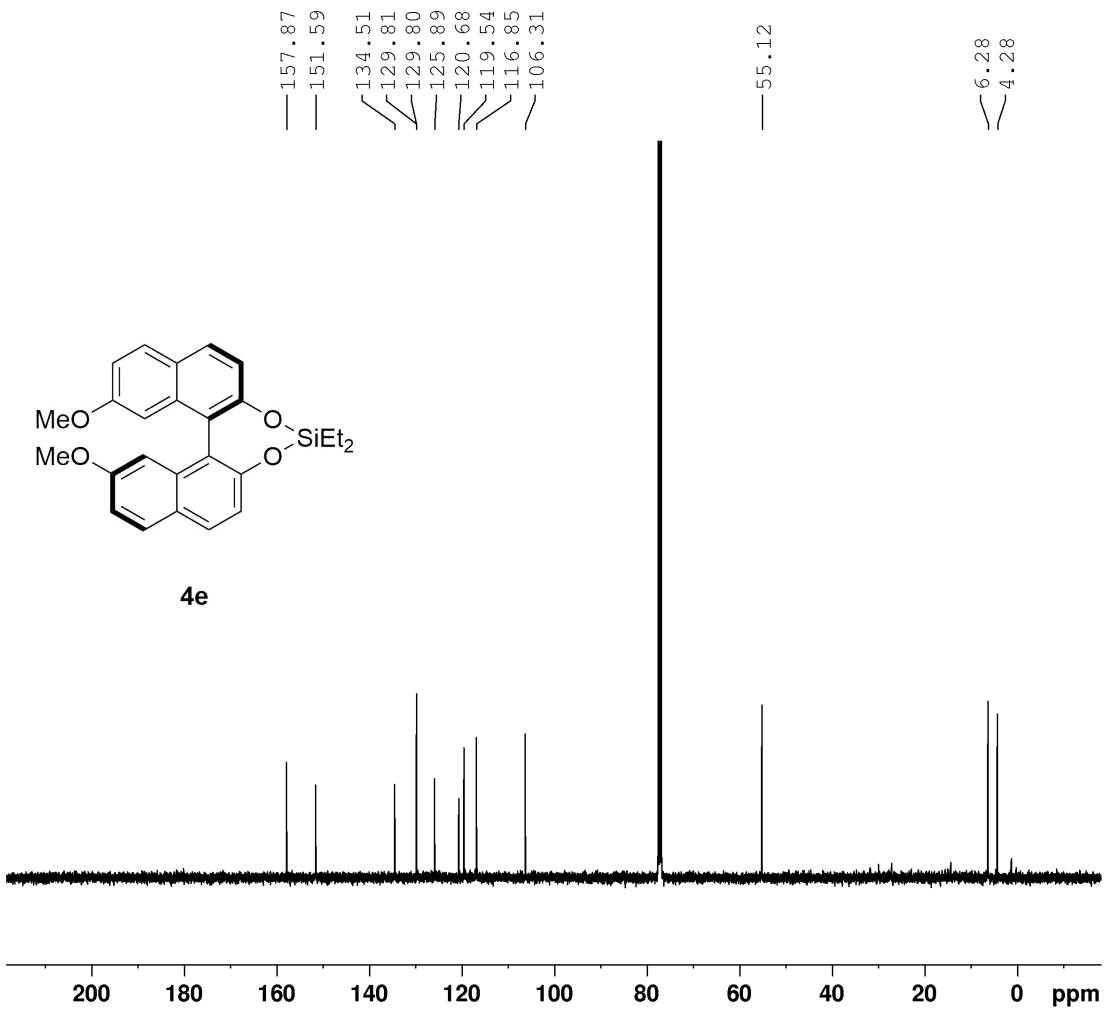

```
Current Data Parameters
NAME            2024-500
EXPNO                 15
PROCNO                 1

F2 - Acquisition Parameters
Date_           20240229
Time               18.19 h
INSTRUM             spect
PROBHD   5 mm CPPBBO BB
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH             10000.000 Hz
FIDRES          0.305176 Hz
AQ              3.2767999 sec
RG                  87.79
DW                 50.000 usec
DE                  6.50 usec
TE                 298.0 K
D1            1.00000000 sec
TD0                    1
SFO1          500.1330885 MHz
NUC1                  1H
P1                 11.85 usec
PLW1         20.00000000 W

F2 - Processing parameters
SI                  65536
SF            500.1300176 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```




```
Current Data Parameters
NAME            2024-500
EXPNO                 16
PROCNO                 1

F2 - Acquisition Parameters
Date_           20240229
Time              18.46 h
INSTRUM             spect
PROBHD   5 mm CPPBBO BB
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   500
DS                     4
SWH            29761.904 Hz
FIDRES          0.908261 Hz
AQ             1.1010048 sec
RG                192.89
DW                16.800 usec
DE                18.00 usec
TE                298.0 K
D1             2.00000000 sec
d11            0.03000000 sec
DELTA          1.89999998 sec
TD0                    1
SFO1         125.7703637 MHz
NUC1                13C
P1                10.75 usec
PLW1          57.00000000 W
SFO2         500.1320005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             80.00 usec
PLW2          20.00000000 W
PLW12          0.43882000 W
PLW13          0.28084999 W

F2 - Processing parameters
SI                 32768
SF           125.7577644 MHz
WDW                   EM
SSB                    0
LB                 1.00 Hz
GB                     0
PC                 1.40
```

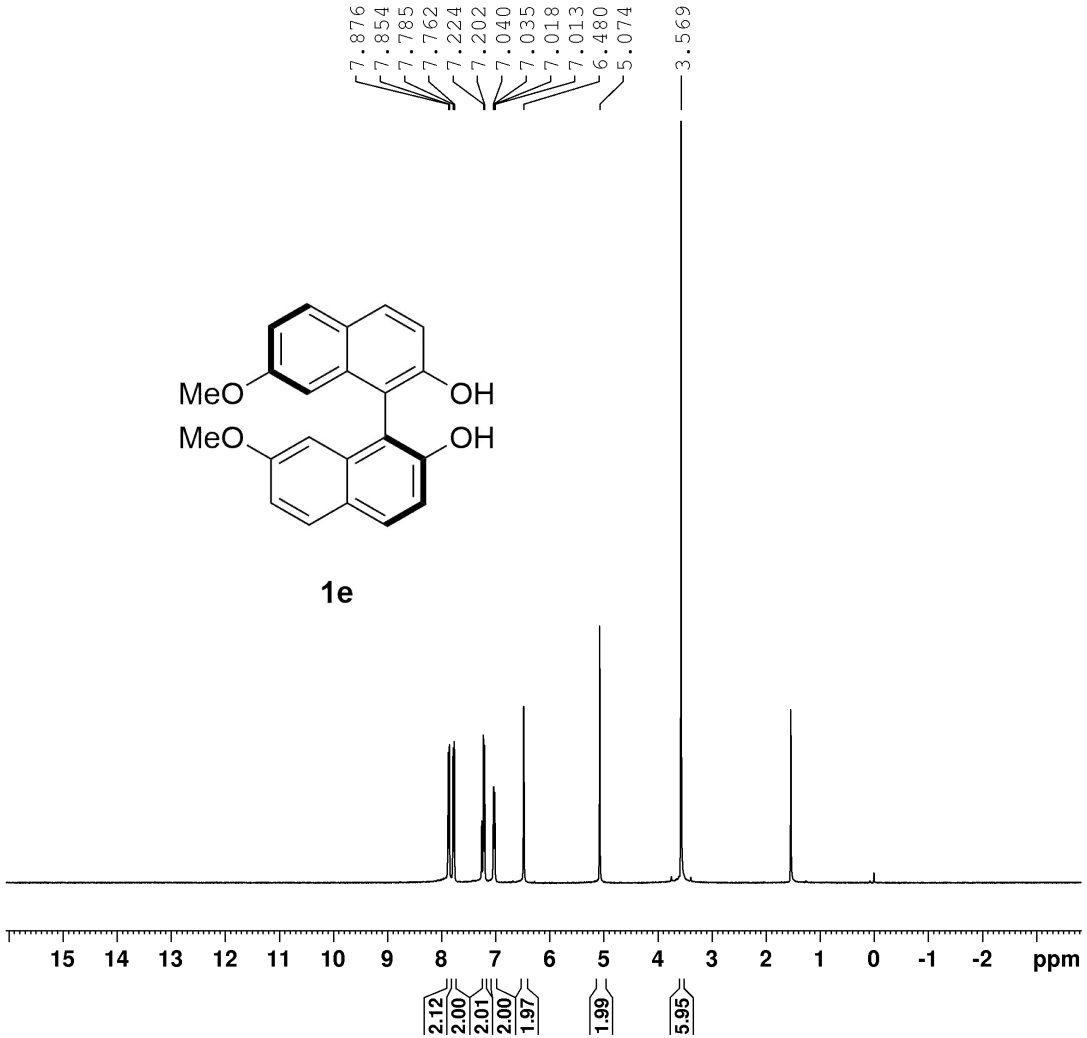



```
Current Data Parameters
NAME            2024-400
EXPNO                 74
PROCNO                 1

F2 - Acquisition Parameters
Date_           20240426
Time               18.58 h
INSTRUM             spect
PROBHD    5 mm PABBO BB/
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     0
SWH             8012.820 Hz
FIDRES          0.489064 Hz
AQ             2.0447233 sec
RG                206.33
DW                62.400 usec
DE                 6.50 usec
TE                298.0 K
D1             2.00000000 sec
TD0                    1
SFO1         400.2424716 MHz
NUC1                  1H
P1                14.30 usec
PLW1         12.00000000 W

F2 - Processing parameters
SI                 65536
SF           400.2400122 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

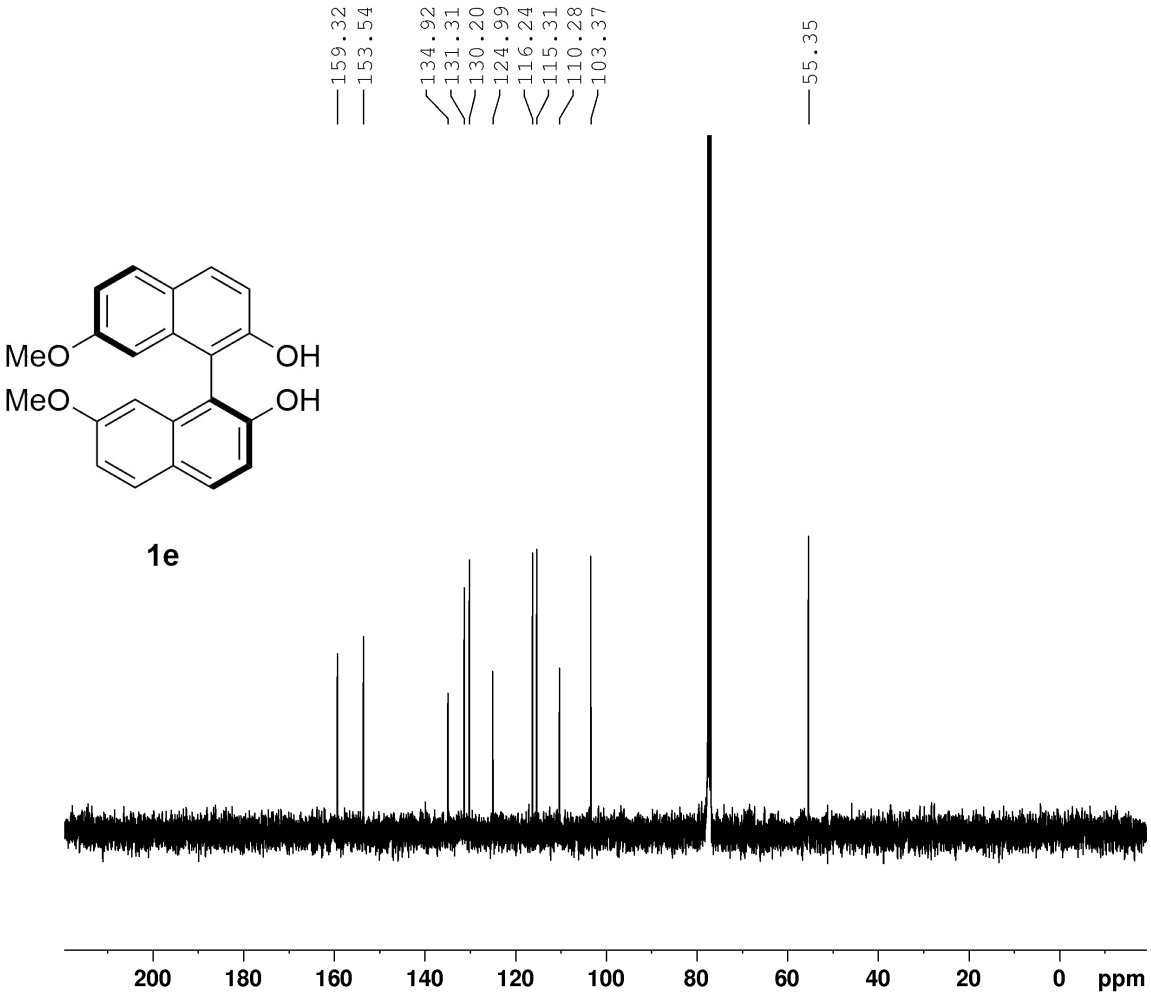


4e

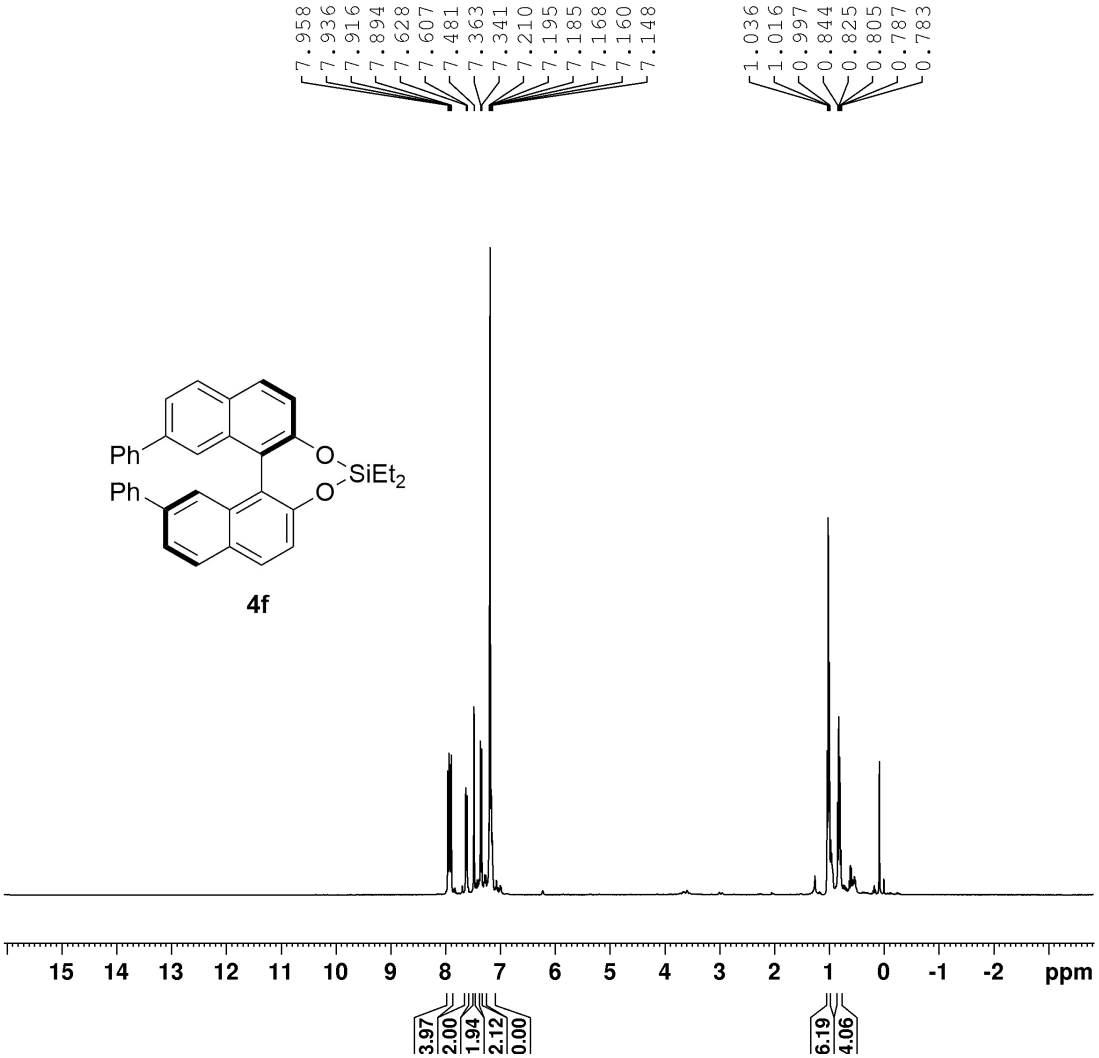
```
Current Data Parameters
NAME            2024-400
EXPNO                 75
PROCNO                 1

F2 - Acquisition Parameters
Date_           20240426
Time              19.57 h
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1024
DS                     4
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ             1.3631488 sec
RG                206.33
DW                20.800 usec
DE                 6.50 usec
TE                298.0 K
D1             2.00000000 sec
d11            0.03000000 sec
DELTA          1.89999998 sec
TD0                    1
SFO1         100.6504916 MHz
NUC1                13C
P1                10.00 usec
PLW1          54.00000000 W
SFO2         400.2416010 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2          12.00000000 W
PLW12          0.30294999 W
PLW13          0.24539000 W

F2 - Processing parameters
SI                 32768
SF           100.6404078 MHz
WDW                   EM
SSB                    0
LB                 1.00 Hz
GB                     0
PC                 1.40
```



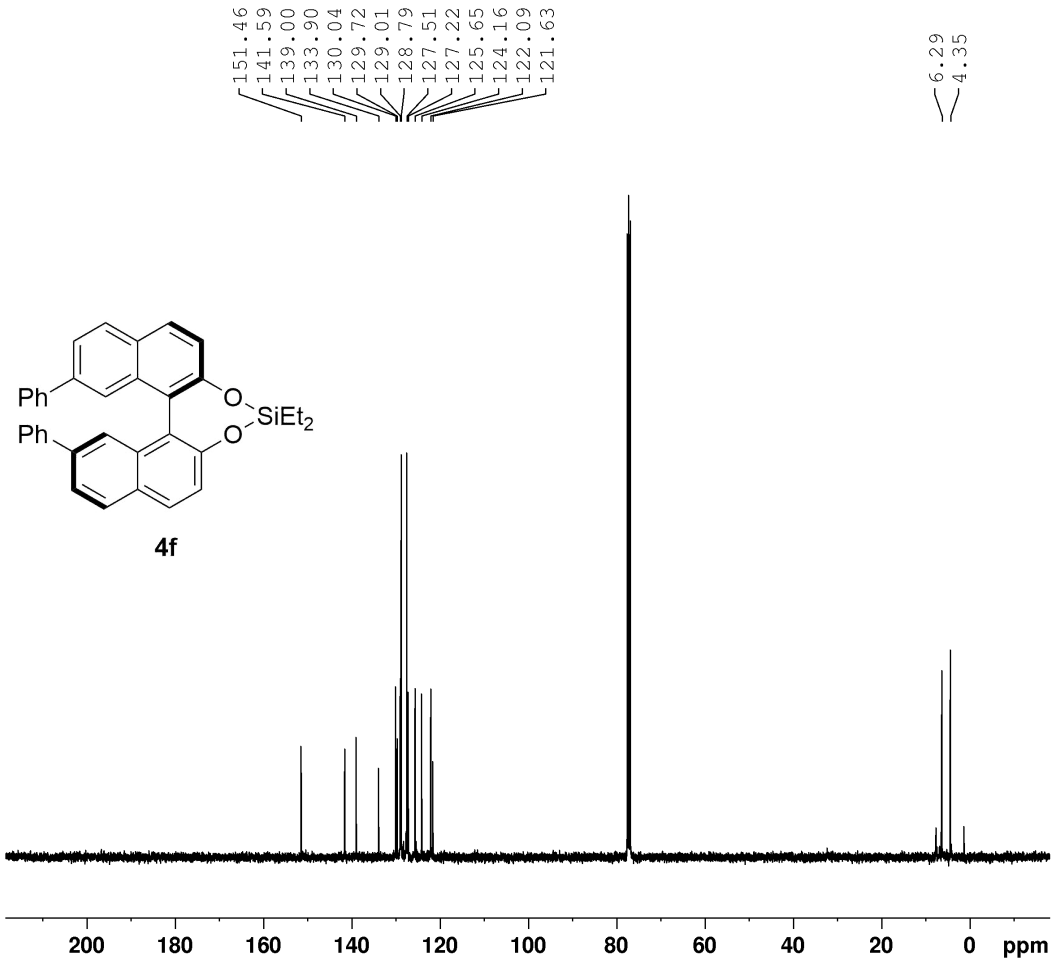

```
Current Data Parameters
NAME            2024-400
EXPNO                127
PROCNO                 1

F2 - Acquisition Parameters
Date_           20240718
Time               14.09 h
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                     4
DS                     0
SWH             8012.820 Hz
FIDRES          0.489064 Hz
AQ             2.0447233 sec
RG                206.33
DW                62.400 usec
DE                 6.50 usec
TE                299.1 K
D1           2.00000000 sec
TD0                    1
SFO1         400.2424716 MHz
NUC1                  1H
P1                14.30 usec
PLW1        12.00000000 W

F2 - Processing parameters
SI                 65536
SF           400.2400123 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                1.00
```



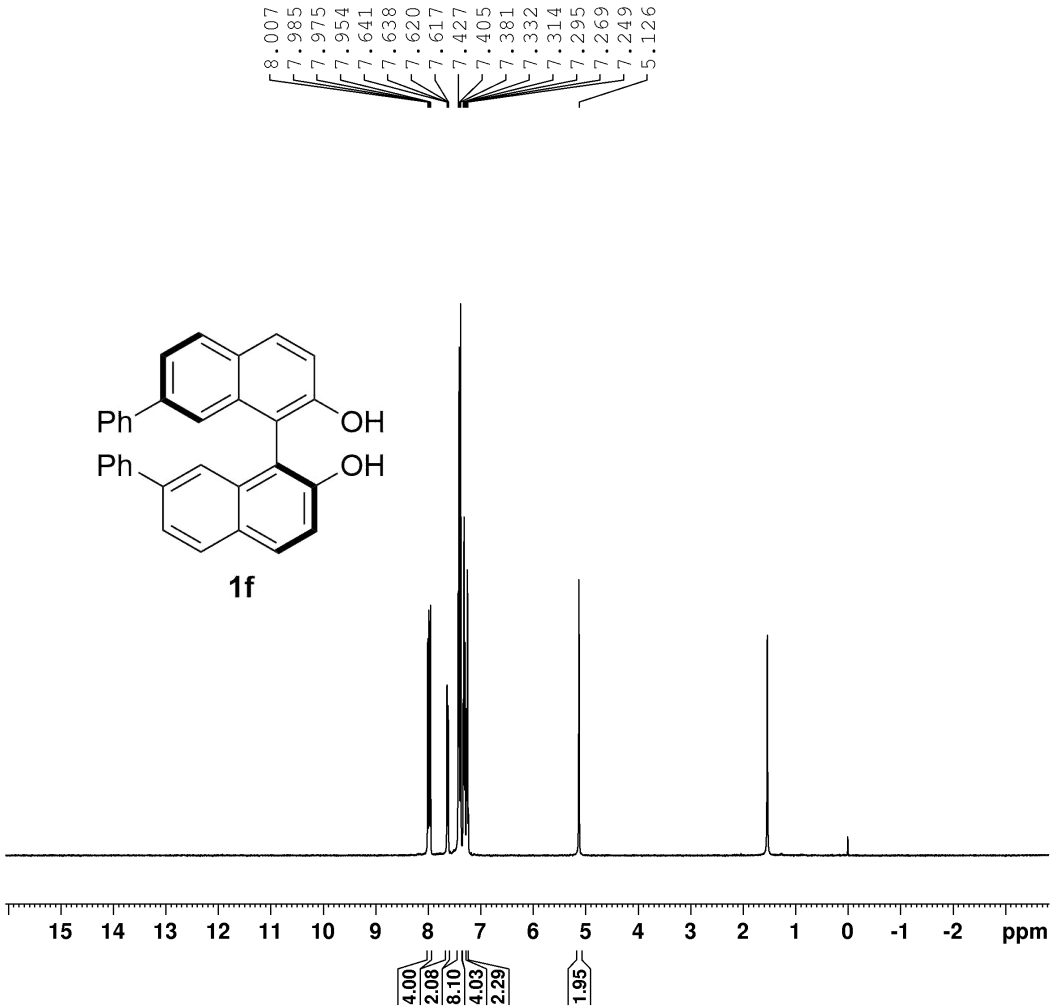

```
Current Data Parameters
NAME            2024-400
EXPNO                128
PROCNO                 1

F2 - Acquisition Parameters
Date_           20240718
Time               14.27 h
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG           zgpg30
TD                 65536
SOLVENT             CDCl3
NS                    300
DS                      4
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ             1.3631488 sec
RG                206.33
DW                20.800 usec
DE                 6.50 usec
TE                299.8 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                    1
SFO1       100.6504916 MHz
NUC1                 13C
P1                10.00 usec
PLW1       54.00000000 W
SFO2       400.2416010 MHz
NUC2                 1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2       12.00000000 W
PLW12       0.30294999 W
PLW13       0.24539000 W

F2 - Processing parameters
SI                 32768
SF         100.6404084 MHz
WDW                   EM
SSB                    0
LB                 1.00 Hz
GB                     0
PC                 1.40
```



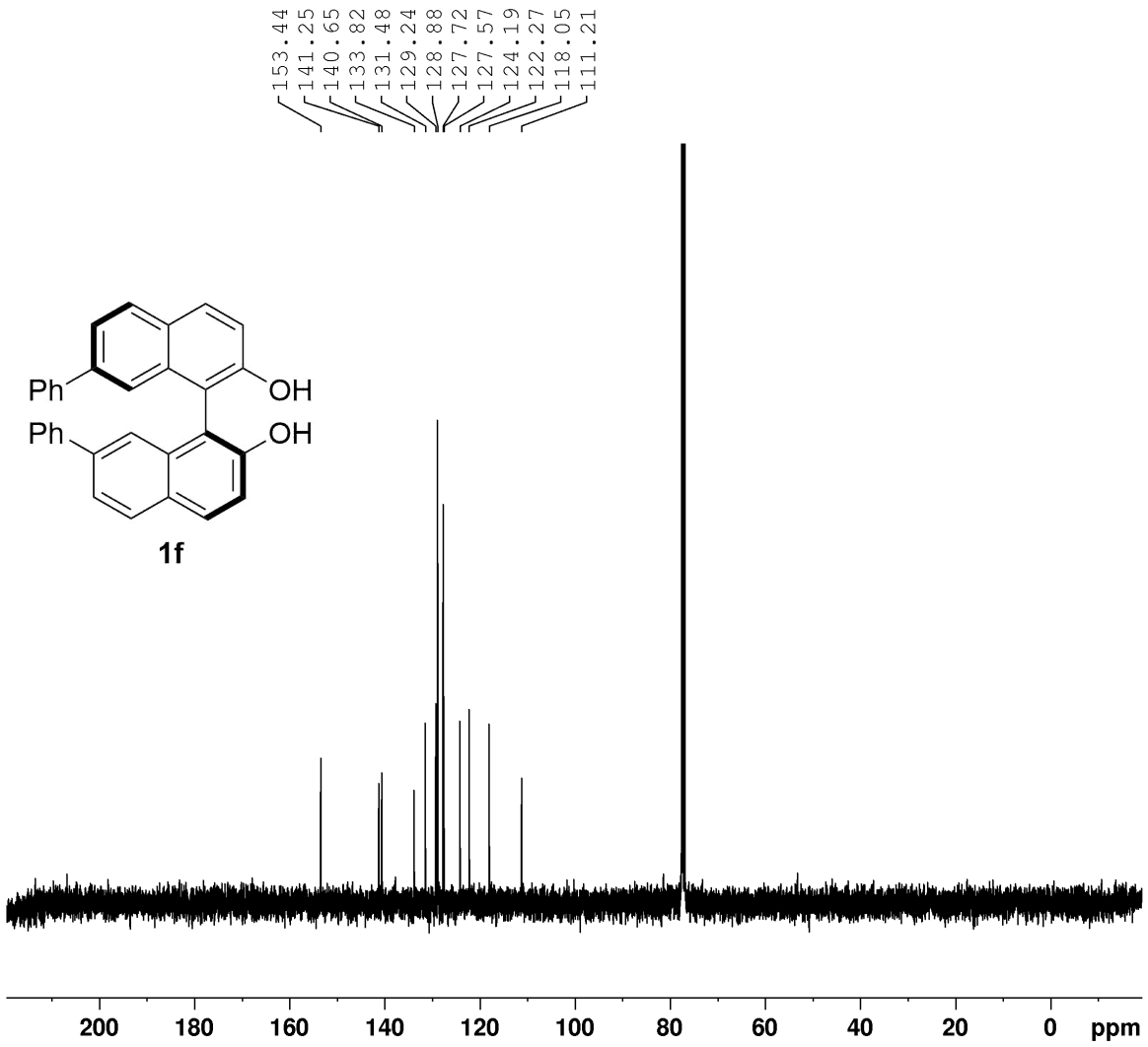

```
Current Data Parameters
NAME            2024-400
EXPNO                100
PROCNO                 1

F2 - Acquisition Parameters
Date_           20240530
Time               23.58 h
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                     8
DS                     0
SWH             8012.820 Hz
FIDRES          0.489064 Hz
AQ             2.0447233 sec
RG                  73.9
DW                62.400 usec
DE                 6.50 usec
TE                298.7 K
D1           2.00000000 sec
TD0                    1
SFO1        400.2424716 MHz
NUC1                1H
P1                14.30 usec
PLW1        12.00000000 W

F2 - Processing parameters
SI                 65536
SF          400.2400295 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



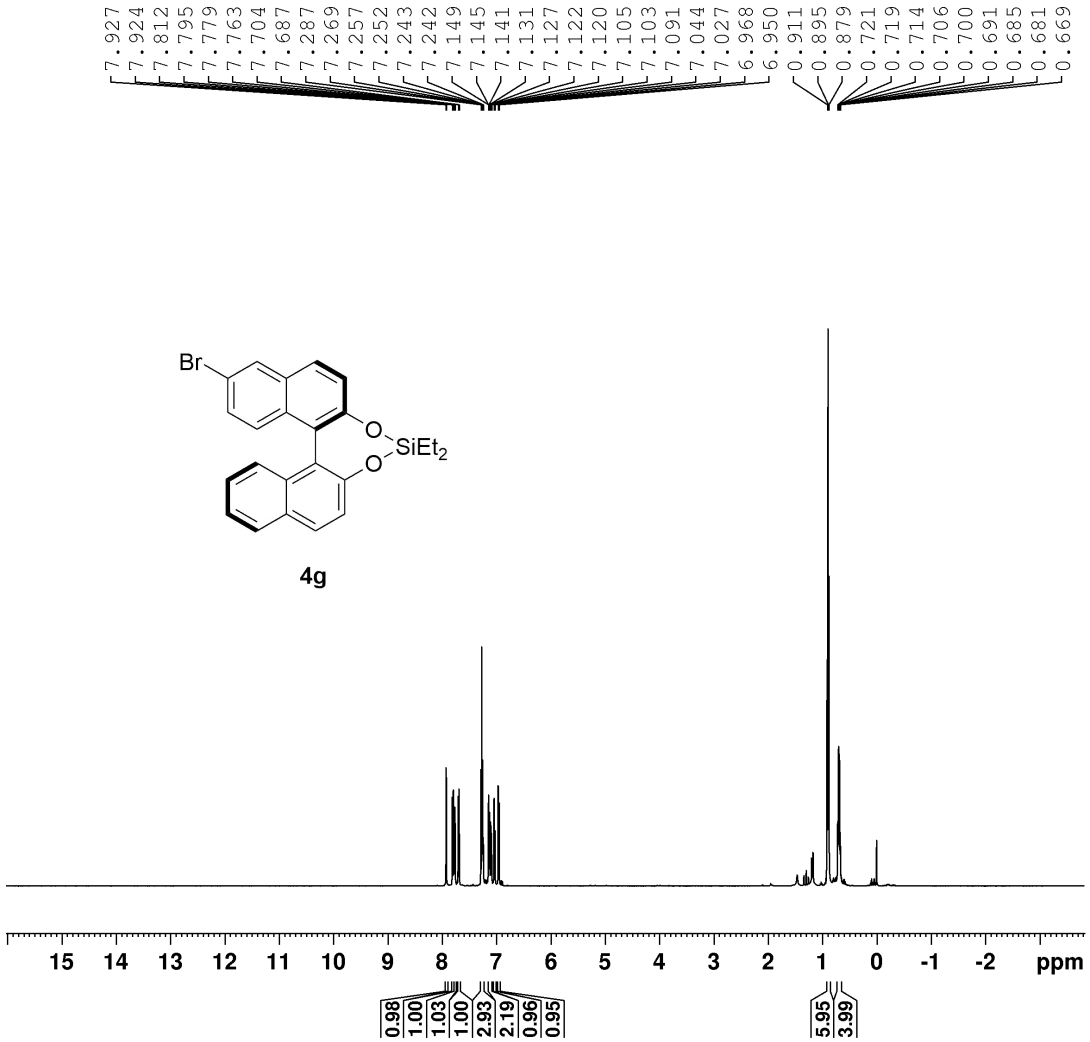

```
Current Data Parameters
NAME            2024-400
EXPNO                101
PROCNO                 1

F2 - Acquisition Parameters
Date_           20240531
Time                0.27 h
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   500
DS                     4
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ              1.3631488 sec
RG                206.33
DW                20.800 usec
DE                 6.50 usec
TE                299.3 K
D1             2.00000000 sec
d11            0.03000000 sec
DELTA          1.89999998 sec
TD0                    1
SFO1         100.6504916 MHz
NUC1                 13C
P1                10.00 usec
PLW1          54.00000000 W
SFO2         400.2416010 MHz
NUC2                 1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2          12.00000000 W
PLW12          0.30294999 W
PLW13          0.24539000 W

F2 - Processing parameters
SI                 32768
SF           100.6404120 MHz
WDW                   EM
SSB                    0
LB                 1.00 Hz
GB                     0
PC                 1.40
```



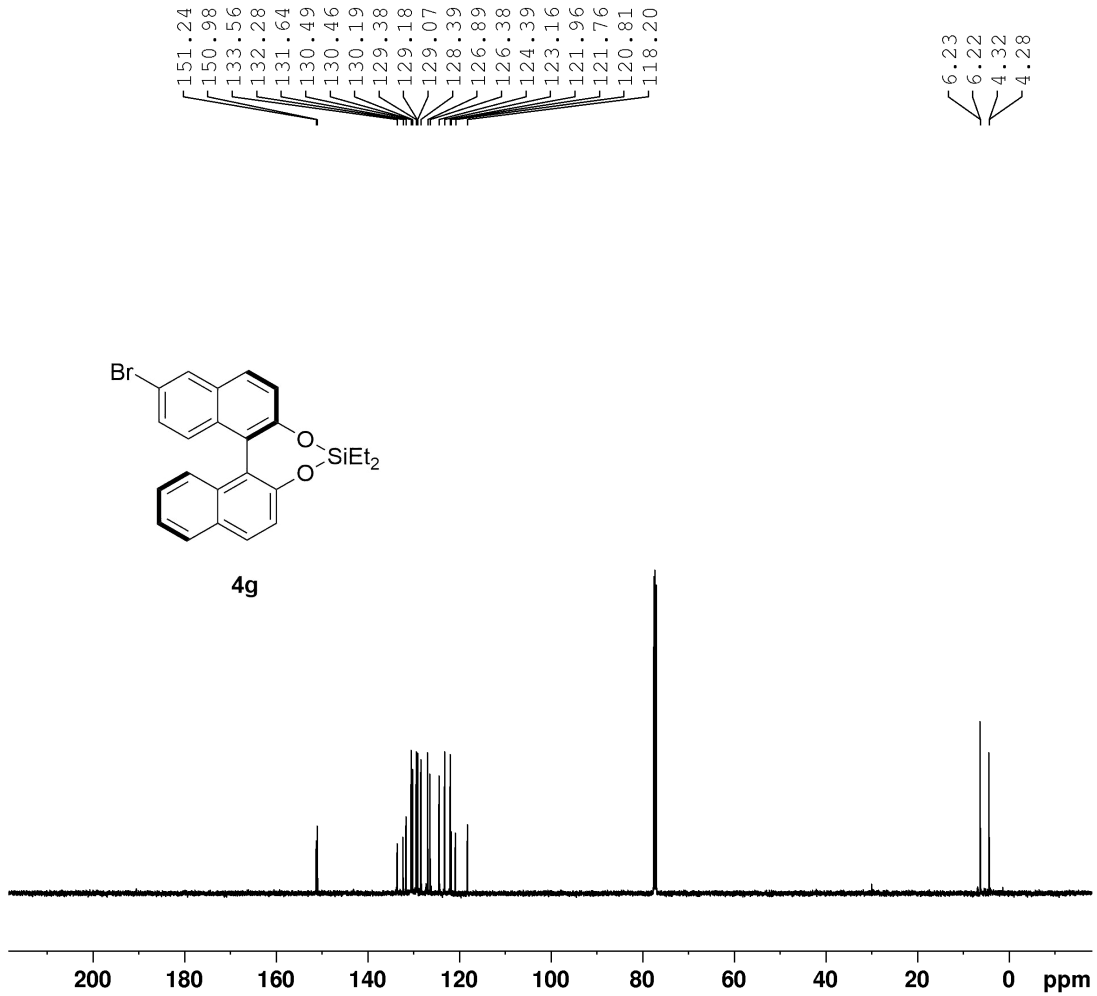

```
Current Data Parameters
NAME            2024-400
EXPNO                125
PROCNO                 1

F2 - Acquisition Parameters
Date_           20240718
Time               13.49 h
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                     4
DS                     0
SWH             8012.820 Hz
FIDRES          0.489064 Hz
AQ             2.0447233 sec
RG                206.33
DW                62.400 usec
DE                 6.50 usec
TE                299.0 K
D1            2.00000000 sec
TD0                    1
SFO1        400.2424716 MHz
NUC1                1H
P1                14.30 usec
PLW1         12.00000000 W

F2 - Processing parameters
SI                 65536
SF          400.2400143 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                 1.00
```



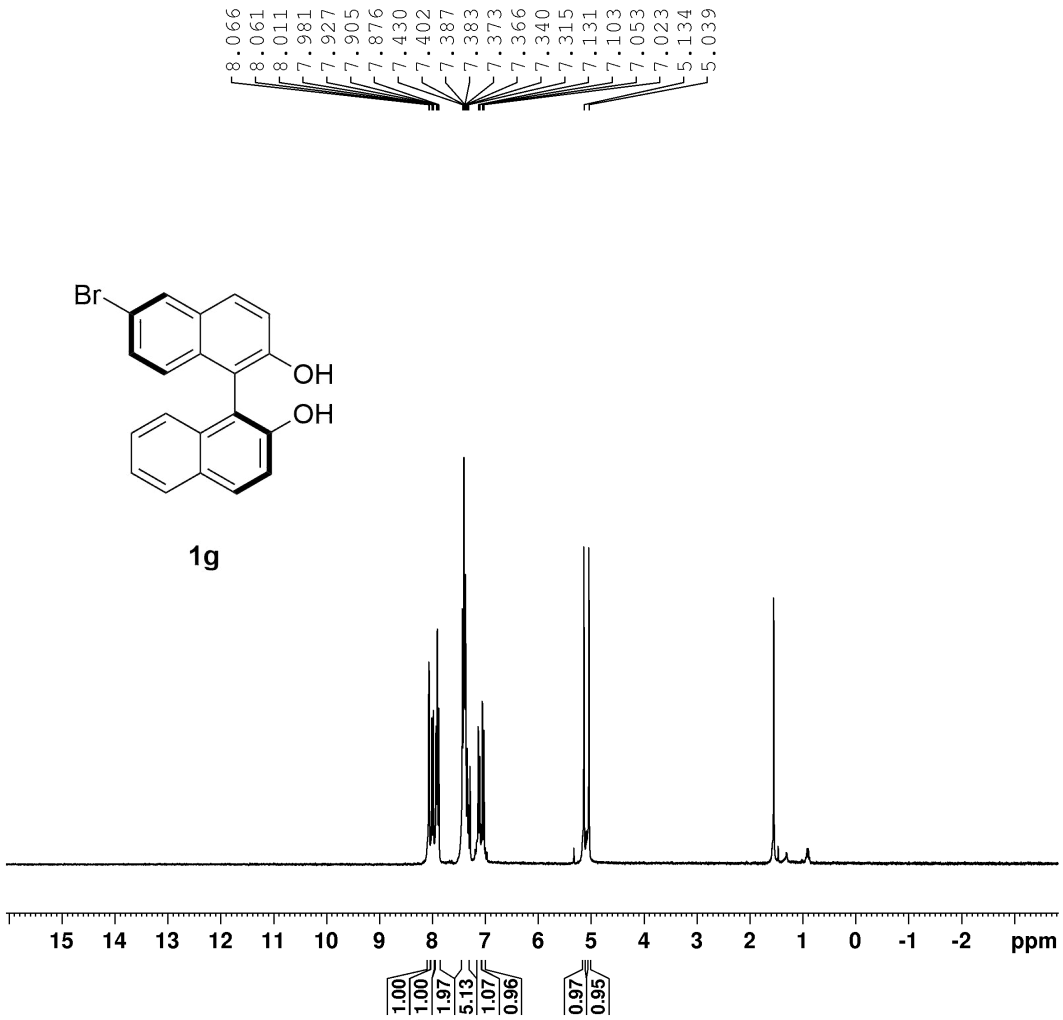

```
Current Data Parameters
NAME            2024-400
EXPNO                126
PROCNO                 1

F2 - Acquisition Parameters
Date_           20240718
Time               14.06 h
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                    300
DS                      4
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ             1.3631488 sec
RG                 206.33
DW                 20.800 usec
DE                   6.50 usec
TE                  299.7 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                    1
SFO1        100.6504916 MHz
NUC1                13C
P1                 10.00 usec
PLW1        54.00000000 W
SFO2        400.2416010 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2              90.00 usec
PLW2        12.00000000 W
PLW12        0.30294999 W
PLW13        0.24539000 W

F2 - Processing parameters
SI                 32768
SF          100.6404083 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```



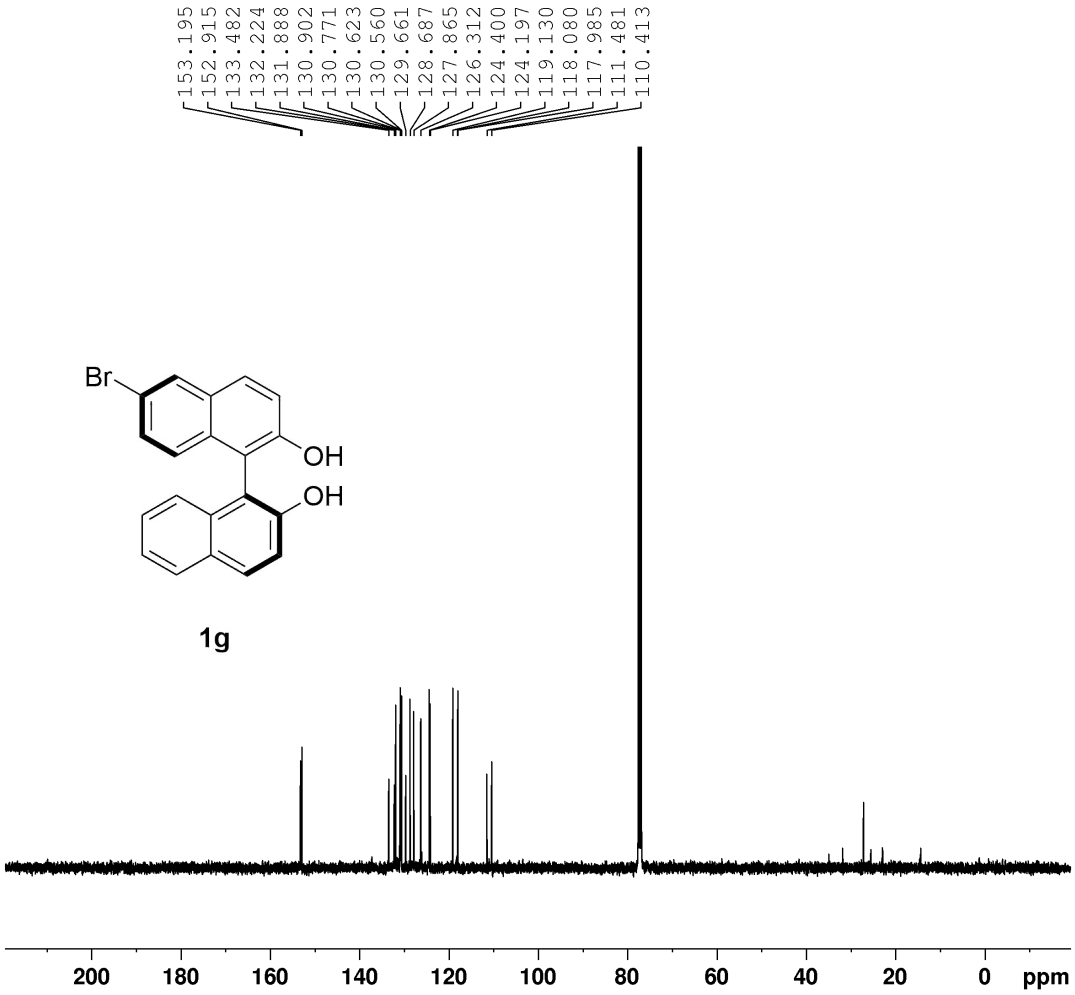

```
Current Data Parameters
NAME            2024-500
EXPNO                 51
PROCNO                 1

F2 - Acquisition Parameters
Date_           20240525
Time               22.51 h
INSTRUM            spect
PROBHD   5 mm CPPBBO BB
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     3
DS                     2
SWH            10000.000 Hz
FIDRES          0.305176 Hz
AQ             3.2767999 sec
RG                 31.72
DW                50.000 usec
DE                 6.50 usec
TE                298.0 K
D1             1.00000000 sec
TD0                    1
SFO1          500.1330885 MHz
NUC1                  1H
P1                11.85 usec
PLW1          20.00000000 W

F2 - Processing parameters
SI                 65536
SF            500.1300707 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



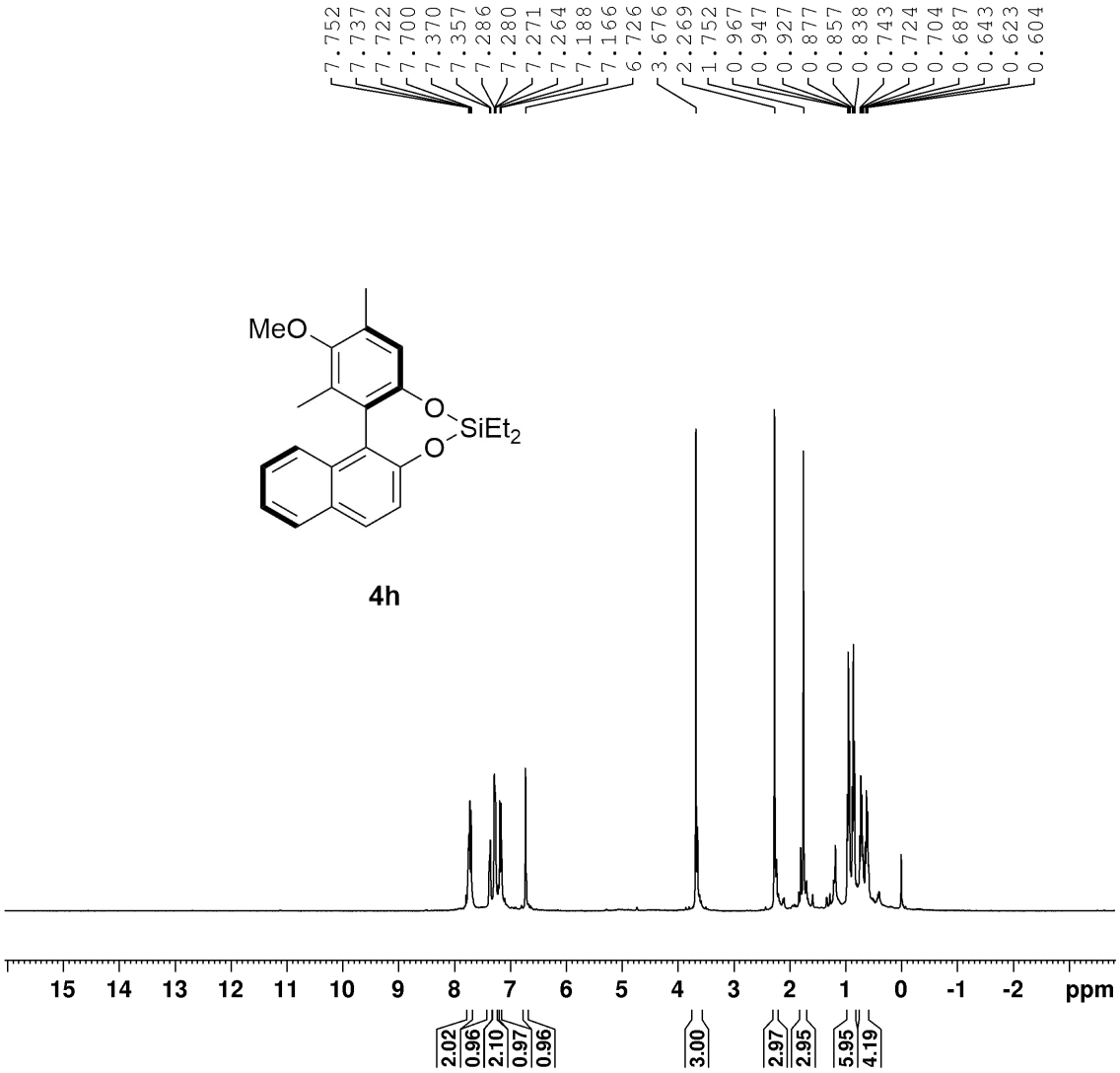

```
Current Data Parameters
NAME            2024-500
EXPNO                 52
PROCNO                 1

F2 - Acquisition Parameters
Date_           20240525
Time               22.52 h
INSTRUM             spect
PROBHD   5 mm CPPBBO BB
PULPROG           zgpg30
TD                 65536
SOLVENT             CDCl3
NS                     19
DS                      4
SWH             29761.904 Hz
FIDRES          0.908261 Hz
AQ              1.1010048 sec
RG                 192.89
DW                 16.800 usec
DE                 18.00 usec
TE                 298.0 K
D1              2.00000000 sec
d11             0.03000000 sec
DELTA           1.89999998 sec
TD0                    1
SFO1          125.7703637 MHz
NUC1                  13C
P1                 10.75 usec
PLW1           57.00000000 W
SFO2          500.1320005 MHz
NUC2                   1H
CPDPRG[2          waltz16
PCPD2              80.00 usec
PLW2           20.00000000 W
PLW12           0.43882000 W
PLW13           0.28084999 W

F2 - Processing parameters
SI                 32768
SF            125.7577686 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```



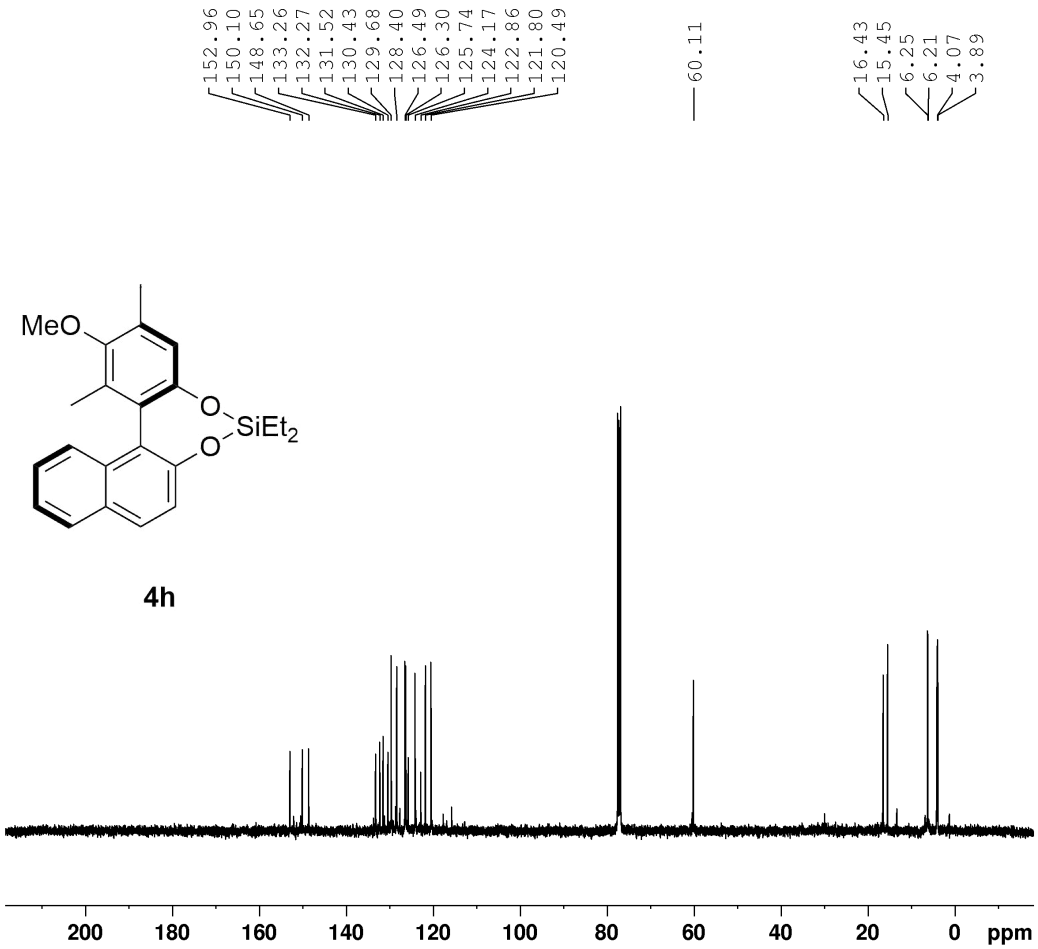

```
Current Data Parameters
NAME          2024-400-p
EXPNO                189
PROCNO                 1

F2 - Acquisition Parameters
Date_           20240523
Time               21.12 h
INSTRUM             spect
PROBHD   5 mm DUL 13C-1
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     0
SWH             6009.615 Hz
FIDRES          0.183399 Hz
AQ             5.4525952 sec
RG                209.09
DW                83.200 usec
DE                 6.50 usec
TE                300.0 K
D1            1.00000000 sec
TD0                    1
SFO1          300.1318534 MHz
NUC1                  1H
P1                 8.00 usec
PLW1         18.00000000 W

F2 - Processing parameters
SI                 65536
SF          300.1300000 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



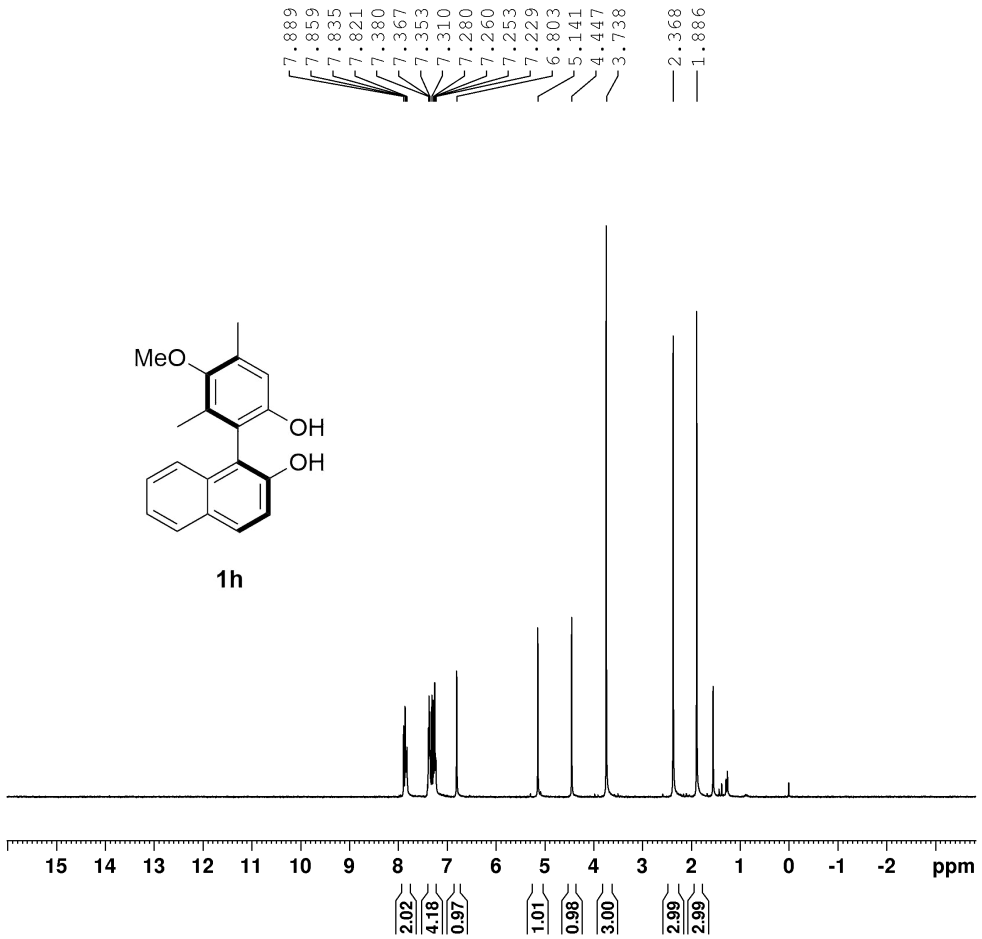

```
Current Data Parameters
NAME            2024-400
EXPNO                 87
PROCNO                 1

F2 - Acquisition Parameters
Date_           20240514
Time                3.24 h
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT            CDCl3
NS                 1024
DS                    4
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ            1.3631488 sec
RG               206.33
DW                20.800 usec
DE                 6.50 usec
TE                298.9 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                   1
SFO1        100.6504916 MHz
NUC1               13C
P1                10.00 usec
PLW1        54.00000000 W
SFO2        400.2416010 MHz
NUC2               1H
CPDPRG[2        waltz16
PCPD2             90.00 usec
PLW2        12.00000000 W
PLW12        0.30294999 W
PLW13        0.24539000 W

F2 - Processing parameters
SI                32768
SF          100.6404093 MHz
WDW                EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```



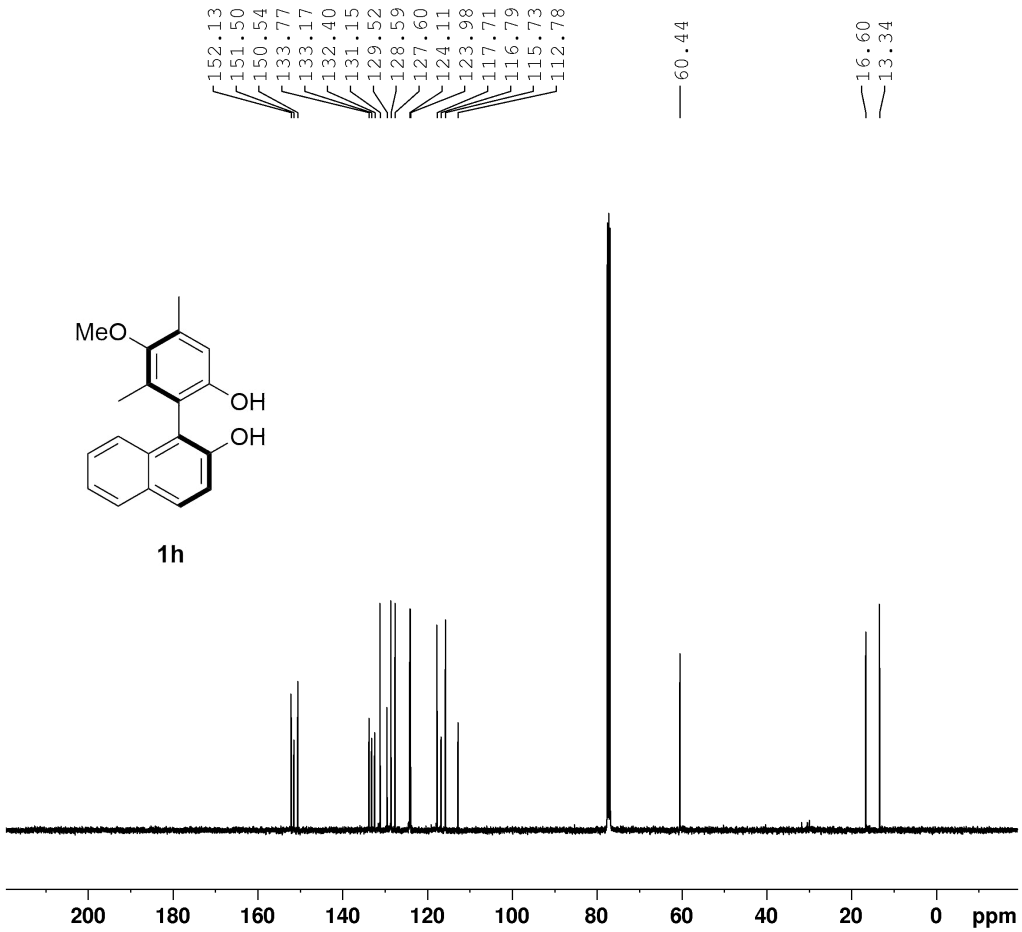

```
Current Data Parameters
NAME            2024-400
EXPNO                115
PROCNO                 1

F2 - Acquisition Parameters
Date_           20240615
Time               23.29 h
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                     4
DS                     0
SWH             8012.820 Hz
FIDRES          0.489064 Hz
AQ             2.0447233 sec
RG                 65.71
DW                62.400 usec
DE                 6.50 usec
TE                299.1 K
D1             2.00000000 sec
TD0                    1
SFO1         400.2424716 MHz
NUC1                  1H
P1                 14.30 usec
PLW1         12.00000000 W

F2 - Processing parameters
SI                 65536
SF           400.2400524 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



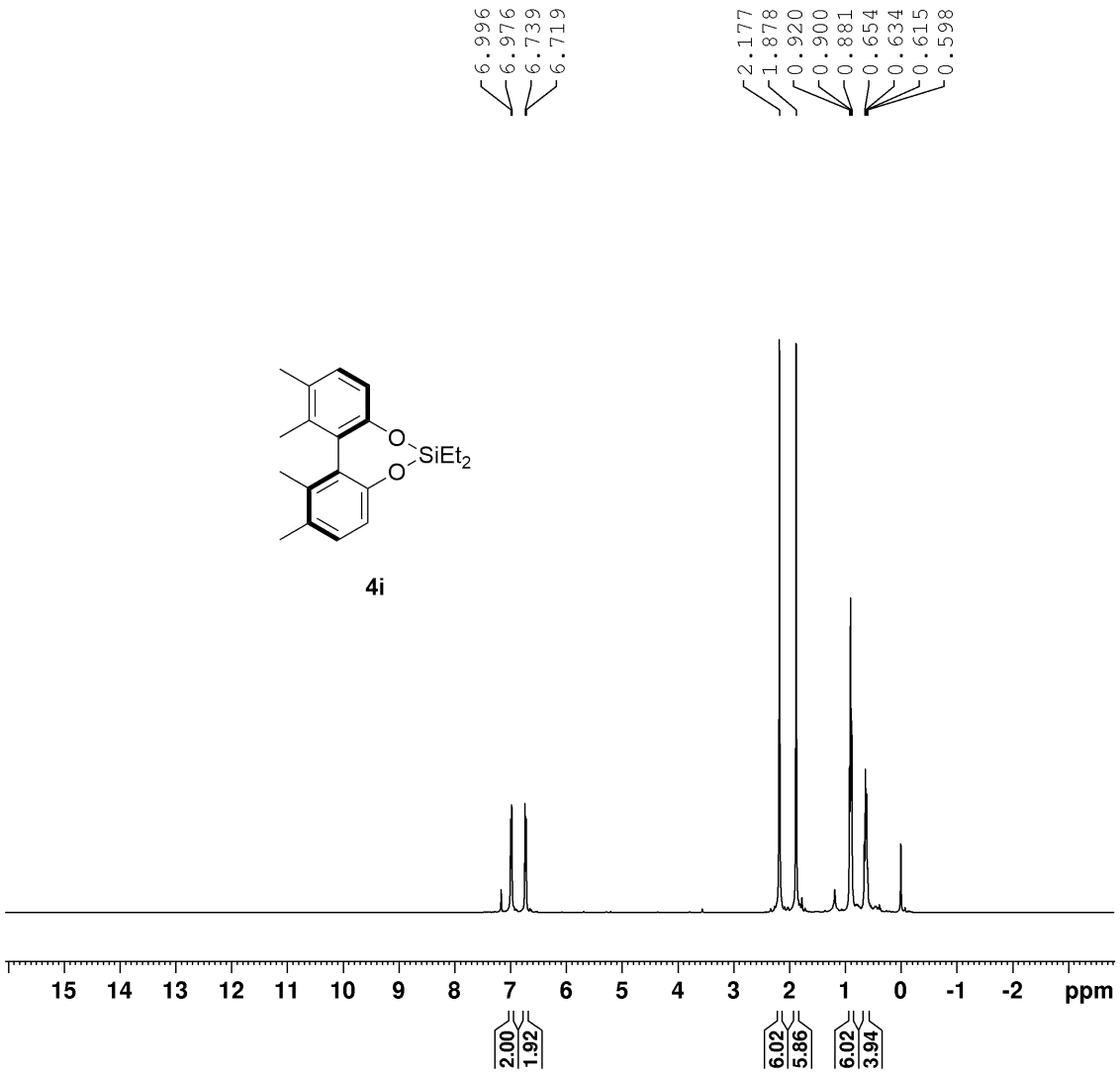

```
Current Data Parameters
NAME            2024-400
EXPNO                116
PROCNO                 1

F2 - Acquisition Parameters
Date_           20240615
Time               23.47 h
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   300
DS                     4
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ              1.3631488 sec
RG                206.33
DW                20.800 usec
DE                 6.50 usec
TE                299.7 K
D1             2.00000000 sec
d11            0.03000000 sec
DELTA          1.89999998 sec
TD0                    1
SFO1         100.6504916 MHz
NUC1                 13C
P1                 10.00 usec
PLW1          54.00000000 W
SFO2         400.2416010 MHz
NUC2                 1H
CPDPRG[2          waltz16
PCPD2              90.00 usec
PLW2          12.00000000 W
PLW12          0.30294999 W
PLW13          0.24539000 W

F2 - Processing parameters
SI                 32768
SF           100.6404102 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```



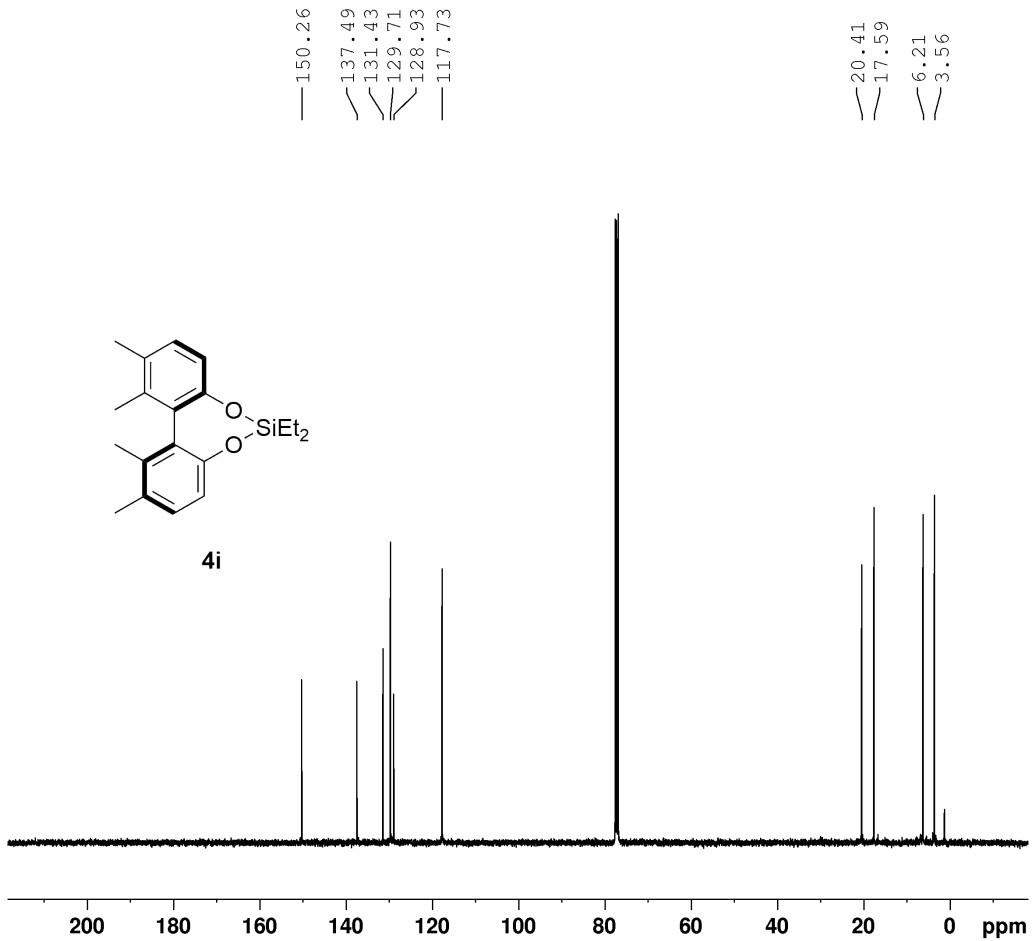

```
Current Data Parameters
NAME          2024-400-p
EXPNO                230
PROCNO                 1

F2 - Acquisition Parameters
Date_           20240614
Time               15.14 h
INSTRUM             spect
PROBHD   5 mm DUL 13C-1
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     0
SWH             6009.615 Hz
FIDRES          0.183399 Hz
AQ              5.4525952 sec
RG                209.09
DW                83.200 usec
DE                 6.50 usec
TE                301.2 K
D1             1.00000000 sec
TD0                    1
SFO1         300.1318534 MHz
NUC1                  1H
P1                 8.00 usec
PLW1         18.00000000 W

F2 - Processing parameters
SI                 65536
SF           300.1300092 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



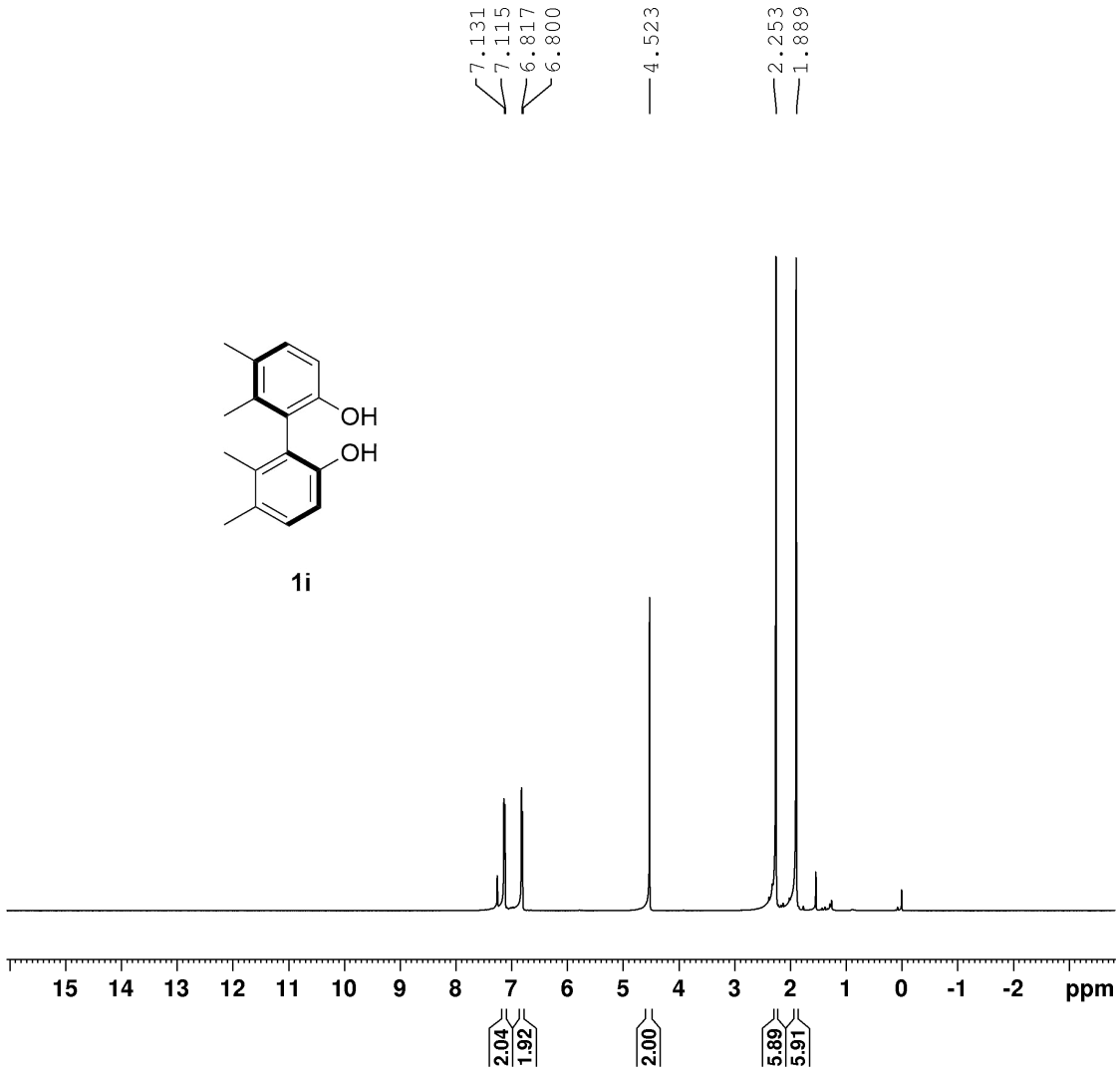

```
Current Data Parameters
NAME                  2024-400
EXPNO                      114
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20240615
Time                     23.26 h
INSTRUM                   spect
PROBHD    5 mm PABBO BB/
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                        1024
DS                           4
SWH                  24038.461 Hz
FIDRES                0.733596 Hz
AQ                    1.3631488 sec
RG                       206.33
DW                       20.800 usec
DE                        6.50 usec
TE                       299.7 K
D1                    2.00000000 sec
d11                   0.03000000 sec
DELTA                 1.89999998 sec
TD0                          1
SFO1                100.6504916 MHz
NUC1                       13C
P1                       10.00 usec
PLW1                54.00000000 W
SFO2                400.2416010 MHz
NUC2                       1H
CPDPRG[2                waltz16
PCPD2                    90.00 usec
PLW2                12.00000000 W
PLW12                0.30294999 W
PLW13                0.24539000 W

F2 - Processing parameters
SI                       32768
SF                  100.6404090 MHz
WDW                         EM
SSB                          0
LB                        1.00 Hz
GB                           0
PC                        1.40
```



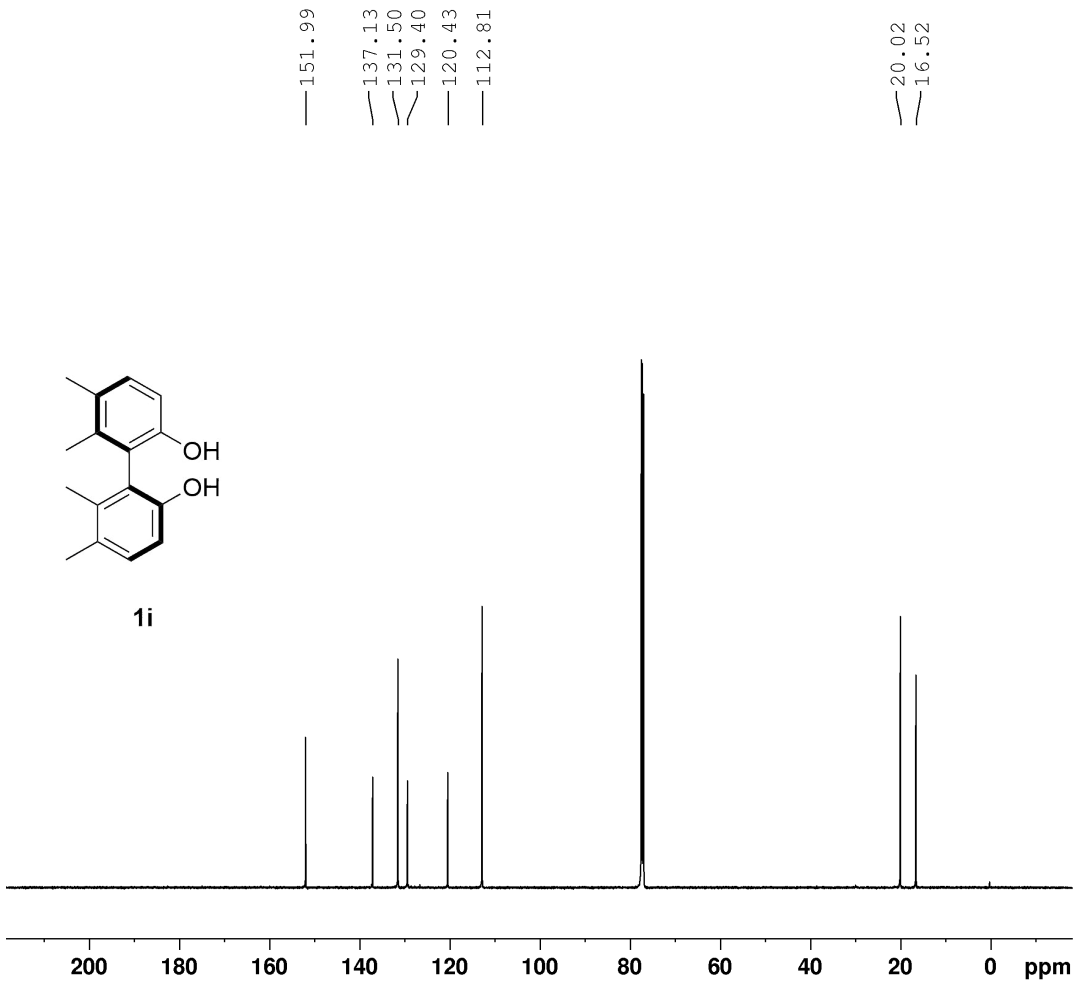

```
Current Data Parameters
NAME                  2024-400
EXPNO                      104
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20240602
Time                     18.02 h
INSTRUM                  spect
PROBHD   5 mm PABBO BB/
PULPROG                  zg30
TD                      32768
SOLVENT                 CDCl3
NS                          8
DS                          0
SWH                  8012.820 Hz
FIDRES               0.489064 Hz
AQ                  2.0447233 sec
RG                      65.71
DW                     62.400 usec
DE                      6.50 usec
TE                     298.7 K
D1                  2.00000000 sec
TD0                         1
SFO1             400.2424716 MHz
NUC1                       1H
P1                     14.30 usec
PLW1            12.00000000 W

F2 - Processing parameters
SI                      65536
SF               400.2400476 MHz
WDW                        EM
SSB                         0
LB                       0.30 Hz
GB                          0
PC                       1.00
```



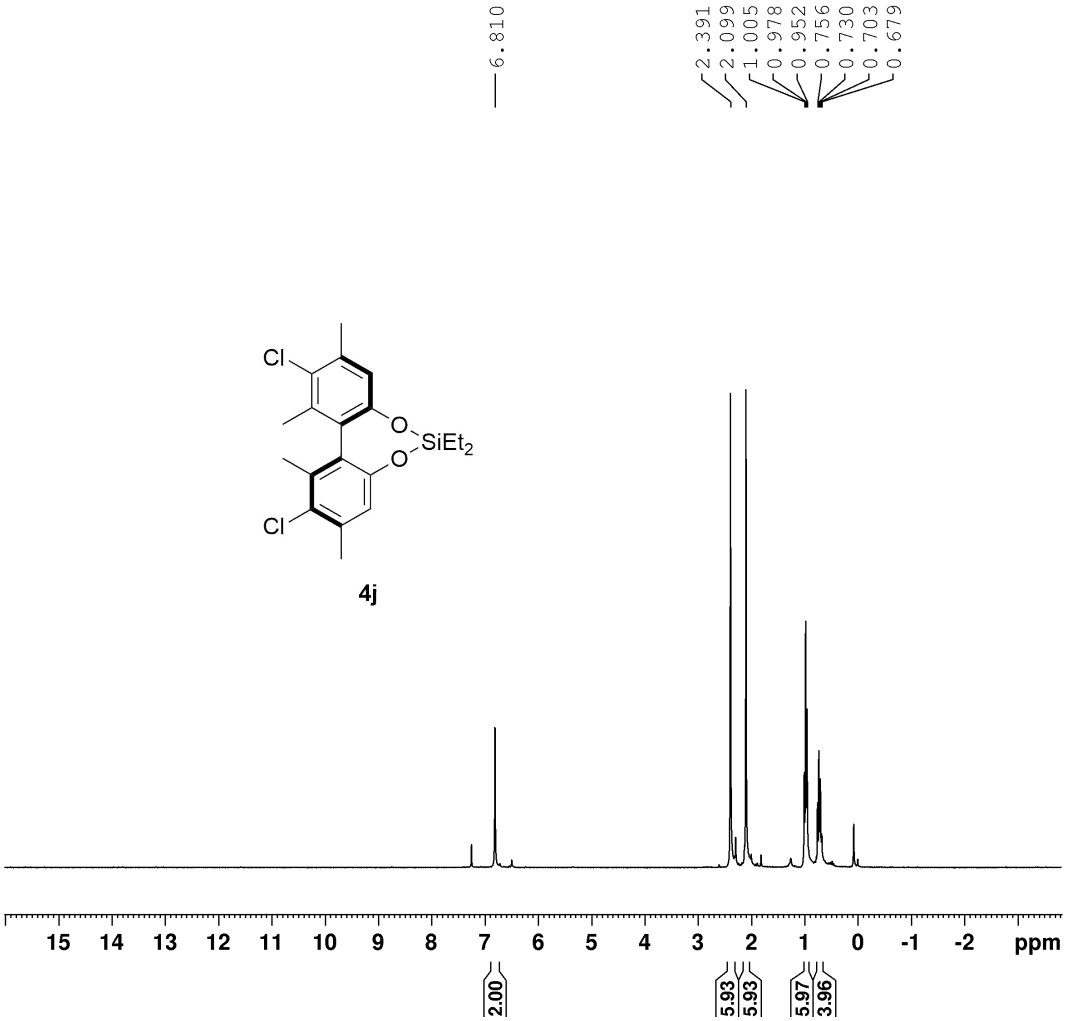

```
Current Data Parameters
NAME            2024-400
EXPNO                105
PROCNO                 1

F2 - Acquisition Parameters
Date_           20240602
Time               19.01 h
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1024
DS                     4
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ              1.3631488 sec
RG                206.33
DW                20.800 usec
DE                 6.50 usec
TE                299.4 K
D1             2.00000000 sec
d11            0.03000000 sec
DELTA          1.89999998 sec
TD0                    1
SFO1         100.6504916 MHz
NUC1                13C
P1                10.00 usec
PLW1         54.00000000 W
SFO2         400.2416010 MHz
NUC2                  1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         12.00000000 W
PLW12         0.30294999 W
PLW13         0.24539000 W

F2 - Processing parameters
SI                 32768
SF           100.6404086 MHz
WDW                   EM
SSB                    0
LB                 1.00 Hz
GB                     0
PC                 1.40
```



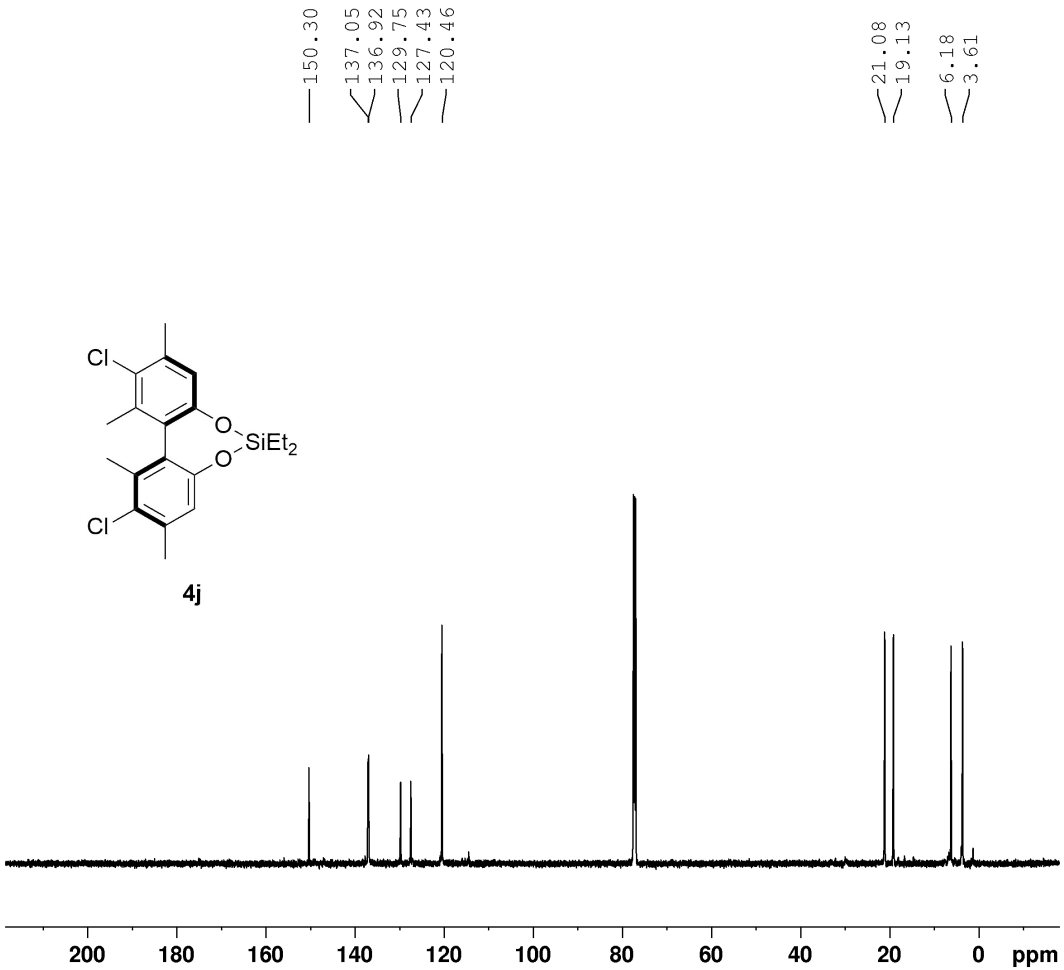

```
Current Data Parameters
NAME                  2024-500
EXPNO                       33
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20240412
Time                      1.08 h
INSTRUM                  spect
PROBHD    5 mm CPPBBO BB
PULPROG                   zg30
TD                       65536
SOLVENT                  CDCl3
NS                          16
DS                           2
SWH                  10000.000 Hz
FIDRES                0.305176 Hz
AQ                    3.2767999 sec
RG                        62.06
DW                       50.000 usec
DE                        6.50 usec
TE                       298.0 K
D1                   1.00000000 sec
TD0                          1
SFO1               500.1330885 MHz
NUC1                        1H
P1                       11.85 usec
PLW1               20.00000000 W

F2 - Processing parameters
SI                       65536
SF                 500.1300186 MHz
WDW                         EM
SSB                          0
LB                        0.30 Hz
GB                           0
PC                        1.00
```



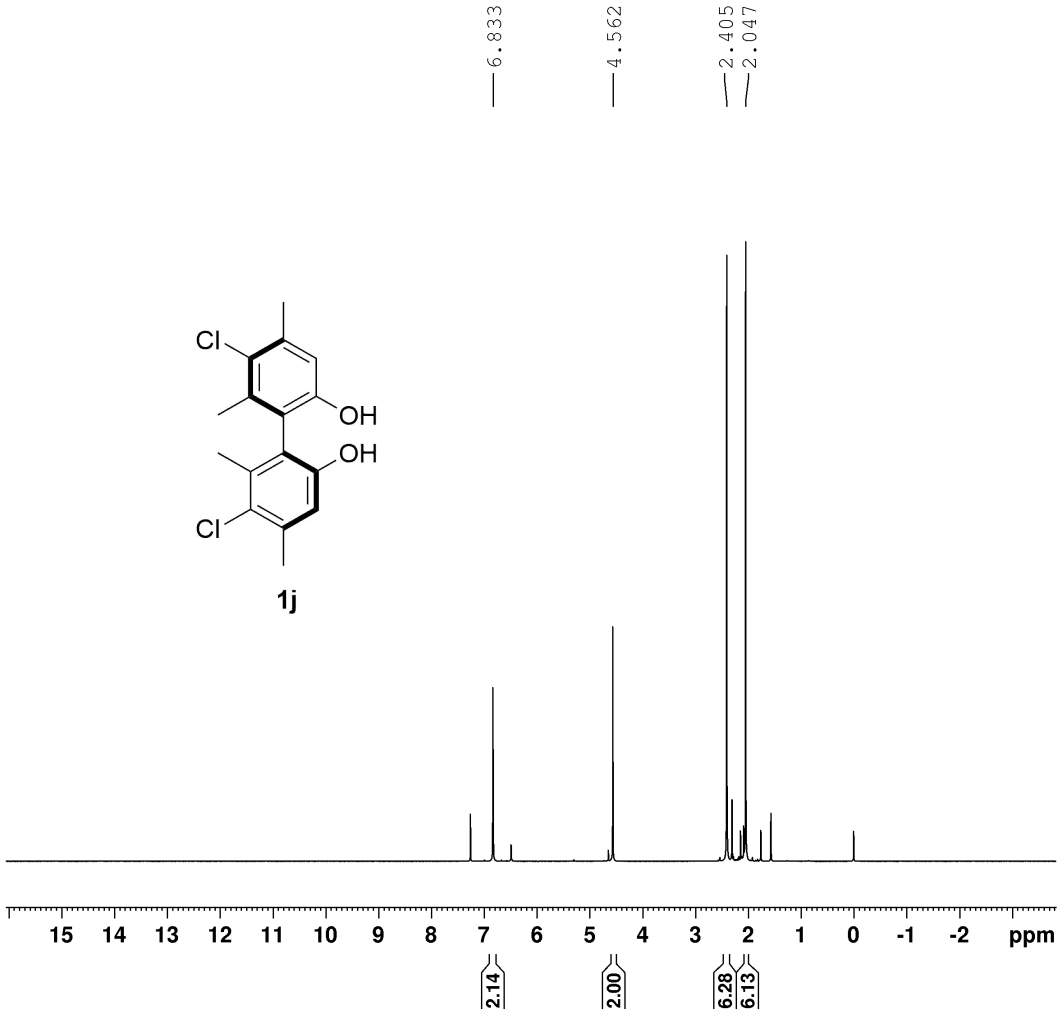

```
Current Data Parameters
NAME            2024-500
EXPNO                 34
PROCNO                 1

F2 - Acquisition Parameters
Date_           20240412
Time                2.03 h
INSTRUM             spect
PROBHD   5 mm CPPBBO BB
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1024
DS                     4
SWH            29761.904 Hz
FIDRES          0.908261 Hz
AQ             1.1010048 sec
RG                192.89
DW                16.800 usec
DE                18.00 usec
TE                298.0 K
D1             2.00000000 sec
d11            0.03000000 sec
DELTA          1.89999998 sec
TD0                    1
SFO1         125.7703637 MHz
NUC1                13C
P1                10.75 usec
PLW1          57.00000000 W
SFO2         500.1320005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             80.00 usec
PLW2          20.00000000 W
PLW12          0.43882000 W
PLW13          0.28084999 W

F2 - Processing parameters
SI                 32768
SF           125.7577659 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```



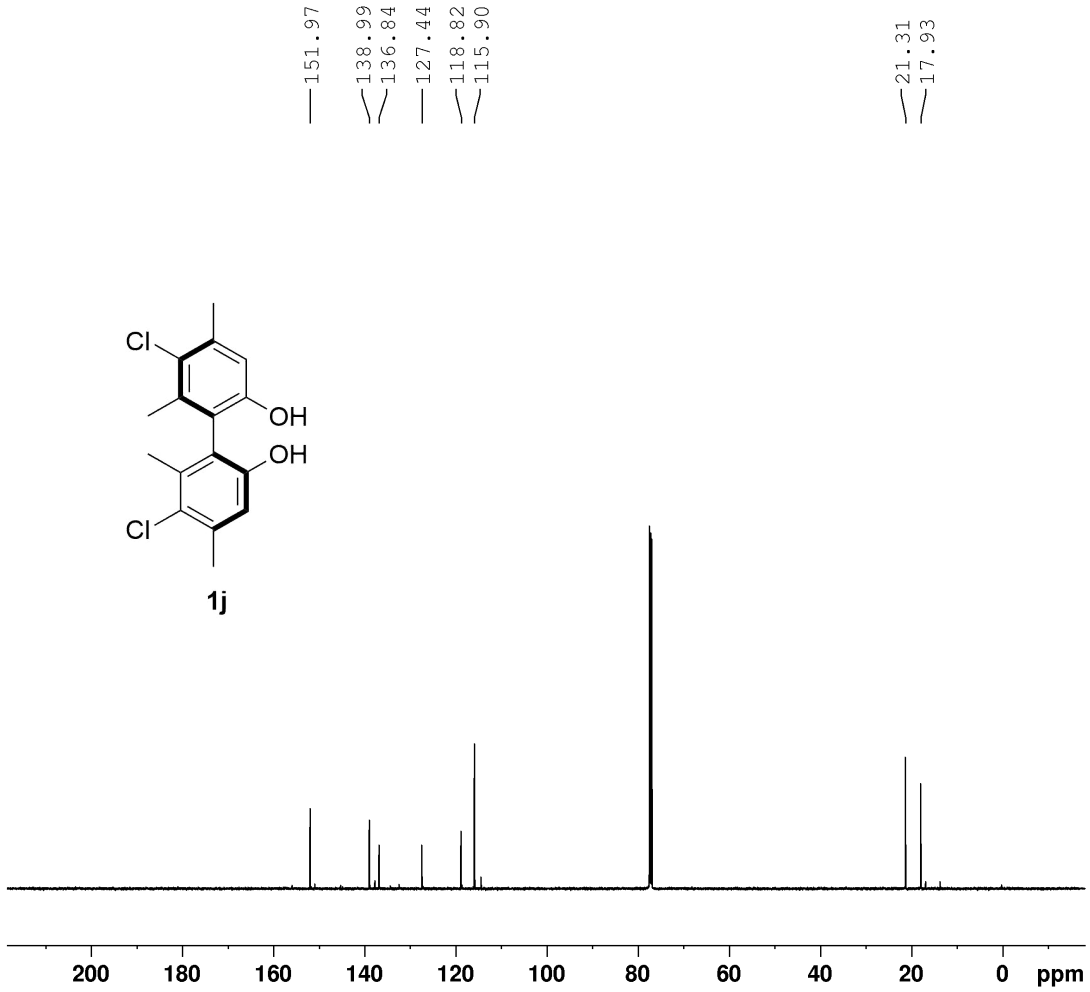

```
Current Data Parameters
NAME          2024-400-p
EXPNO                216
PROCNO                 1

F2 - Acquisition Parameters
Date_           20240603
Time               11.58 h
INSTRUM             spect
PROBHD   5 mm DUL 13C-1
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      0
SWH              6009.615 Hz
FIDRES           0.183399 Hz
AQ              5.4525952 sec
RG                 209.09
DW                 83.200 usec
DE                   6.50 usec
TE                  300.7 K
D1             1.00000000 sec
TD0                    1
SFO1         300.1318534 MHz
NUC1                  1H
P1                  8.00 usec
PLW1        18.00000000 W

F2 - Processing parameters
SI                  65536
SF           300.1300097 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```



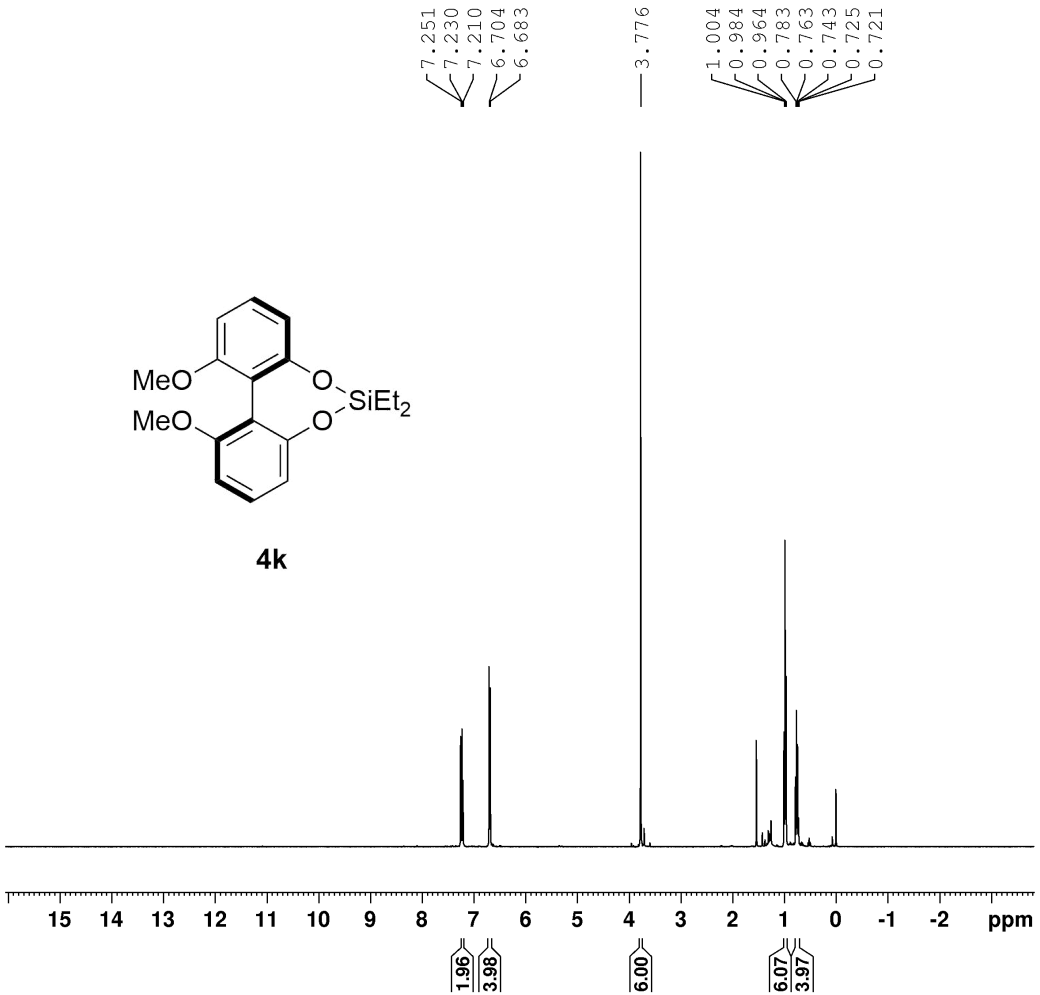

```
Current Data Parameters
NAME                  2024-500
EXPNO                       54
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20240604
Time                      6.19 h
INSTRUM                   spect
PROBHD    5 mm CPPBBO BB
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                         500
DS                           4
SWH                  29761.904 Hz
FIDRES                0.908261 Hz
AQ                    1.1010048 sec
RG                       192.89
DW                       16.800 usec
DE                       18.00 usec
TE                       298.0 K
D1                    2.00000000 sec
d11                   0.03000000 sec
DELTA                 1.89999998 sec
TD0                          1
SFO1                125.7703637 MHz
NUC1                        13C
P1                       10.75 usec
PLW1                57.00000000 W
SFO2                500.1320005 MHz
NUC2                        1H
CPDPRG[2                waltz16
PCPD2                    80.00 usec
PLW2                20.00000000 W
PLW12                0.43882000 W
PLW13                0.28084999 W

F2 - Processing parameters
SI                       32768
SF                  125.7577636 MHz
WDW                         EM
SSB                          0
LB                        1.00 Hz
GB                           0
PC                        1.40
```



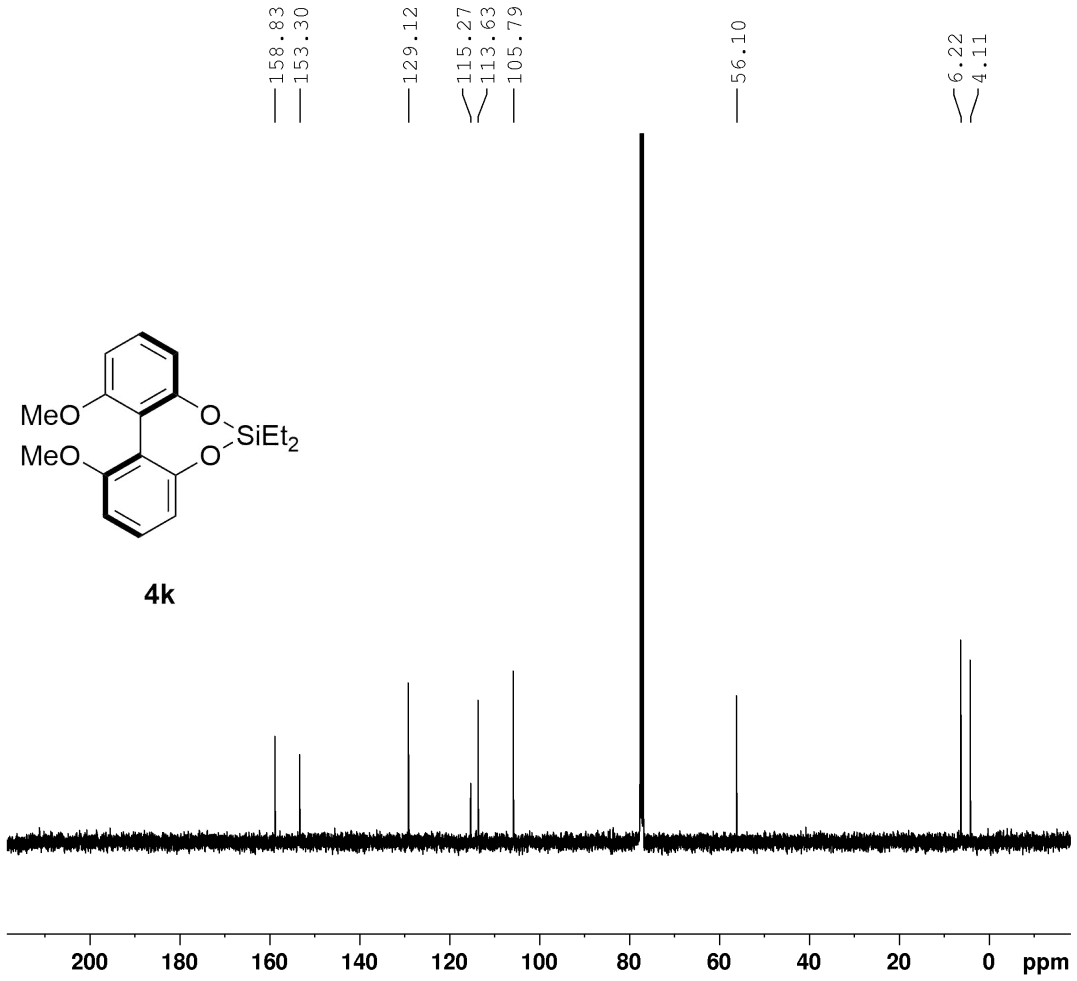

```
Current Data Parameters
NAME            2024-500
EXPNO                 35
PROCNO                 1

F2 - Acquisition Parameters
Date_           20240412
Time                2.07 h
INSTRUM            spect
PROBHD   5 mm CPPBBO BB
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH            10000.000 Hz
FIDRES             0.305176 Hz
AQ              3.2767999 sec
RG                 62.06
DW                 50.000 usec
DE                  6.50 usec
TE                 298.0 K
D1              1.00000000 sec
TD0                    1
SFO1           500.1330885 MHz
NUC1                  1H
P1                 11.85 usec
PLW1           20.00000000 W

F2 - Processing parameters
SI                 65536
SF            500.1300131 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```




```
Current Data Parameters
NAME                  2024-500
EXPNO                       39
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20240412
Time                     12.38 h
INSTRUM                  spect
PROBHD   5 mm CPPBBO BB
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                         200
DS                           4
SWH                  29761.904 Hz
FIDRES                0.908261 Hz
AQ                   1.1010048 sec
RG                      192.89
DW                      16.800 usec
DE                      18.00 usec
TE                      298.0 K
D1                   2.00000000 sec
d11                  0.03000000 sec
DELTA                1.89999998 sec
TD0                          1
SFO1               125.7703637 MHz
NUC1                        13C
P1                      10.75 usec
PLW1                57.00000000 W
SFO2               500.1320005 MHz
NUC2                        1H
CPDPRG[2                waltz16
PCPD2                   80.00 usec
PLW2                20.00000000 W
PLW12                0.43882000 W
PLW13                0.28084999 W

F2 - Processing parameters
SI                       32768
SF                 125.7577638 MHz
WDW                         EM
SSB                          0
LB                        1.00 Hz
GB                           0
PC                        1.40
```

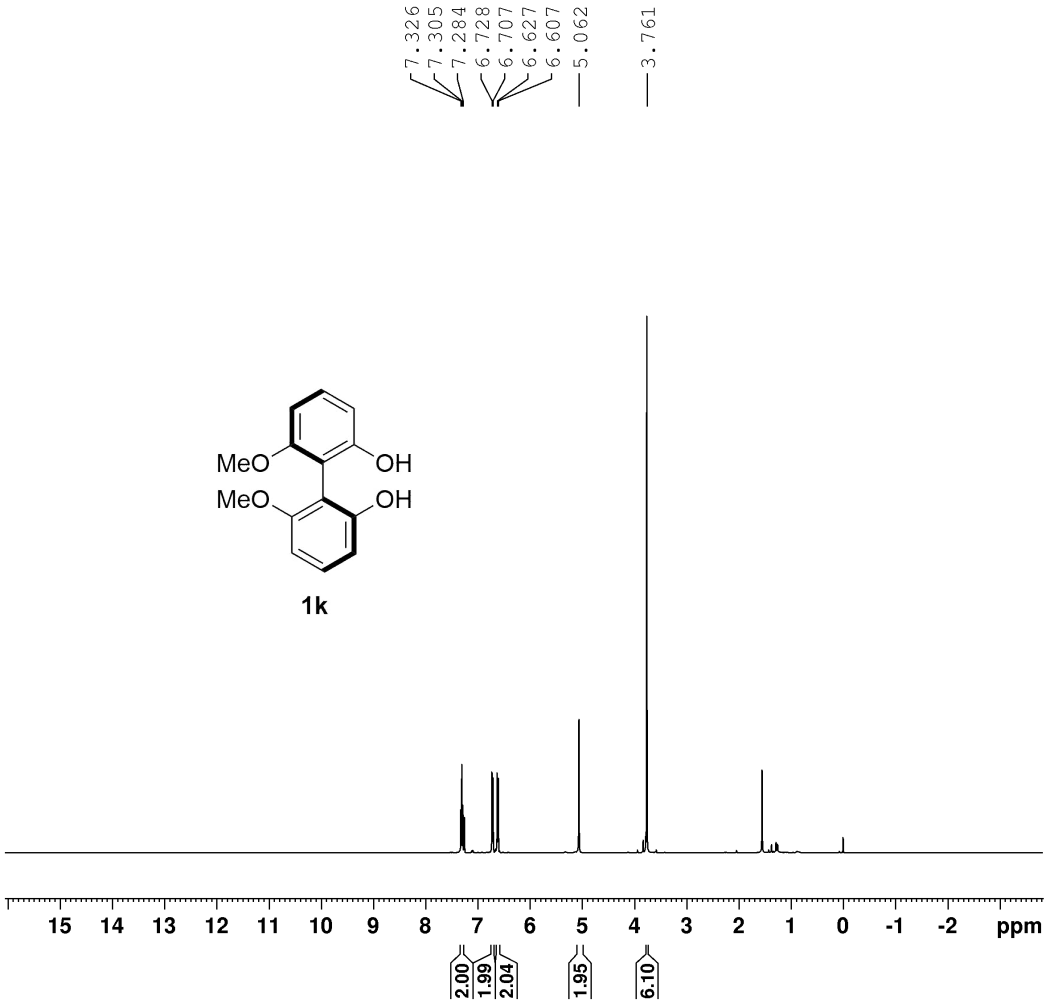



```
Current Data Parameters
NAME            2024-400
EXPNO                 72
PROCNO                 1

F2 - Acquisition Parameters
Date_           20240425
Time               16.29 h
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                     5
DS                     0
SWH             8012.820 Hz
FIDRES          0.489064 Hz
AQ             2.0447233 sec
RG                206.33
DW                62.400 usec
DE                 6.50 usec
TE                298.0 K
D1            2.00000000 sec
TD0                    1
SFO1         400.2424716 MHz
NUC1                  1H
P1                14.30 usec
PLW1         12.00000000 W

F2 - Processing parameters
SI                 65536
SF           400.2400112 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                1.00
```

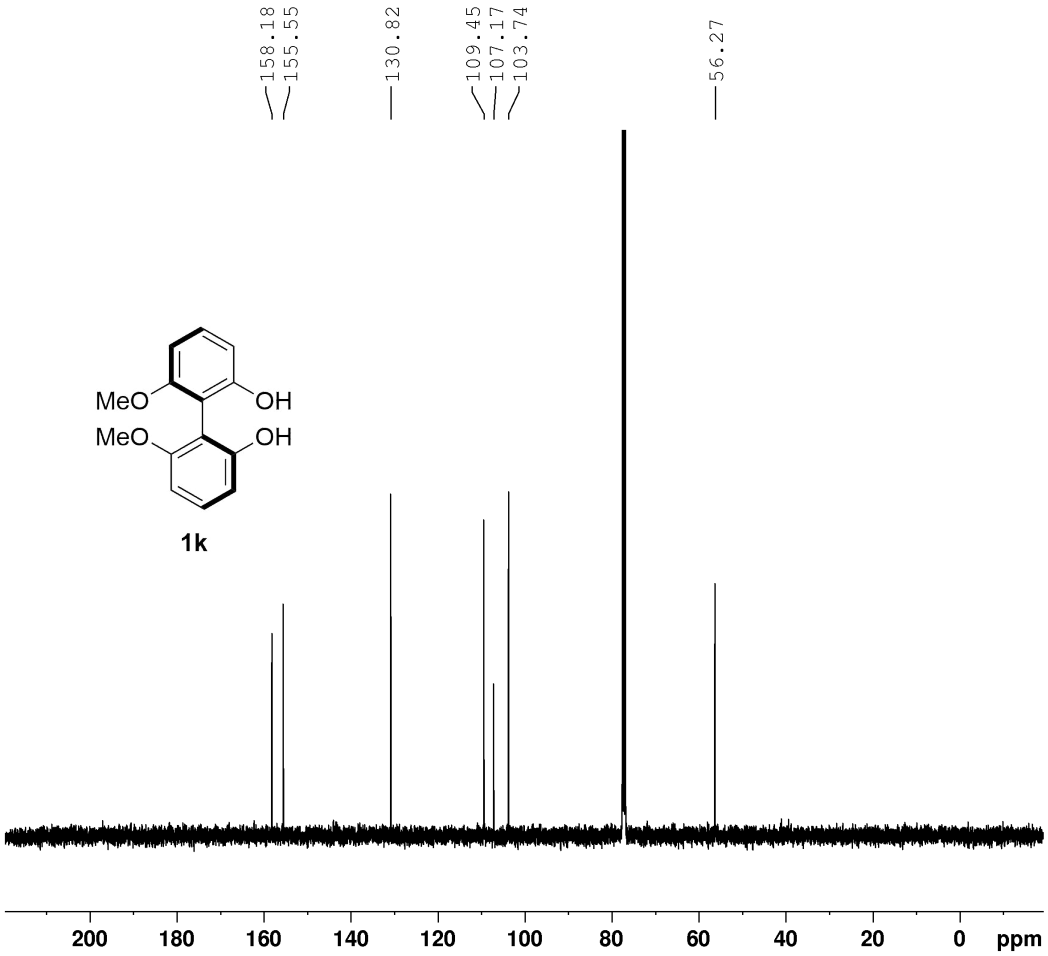


4k

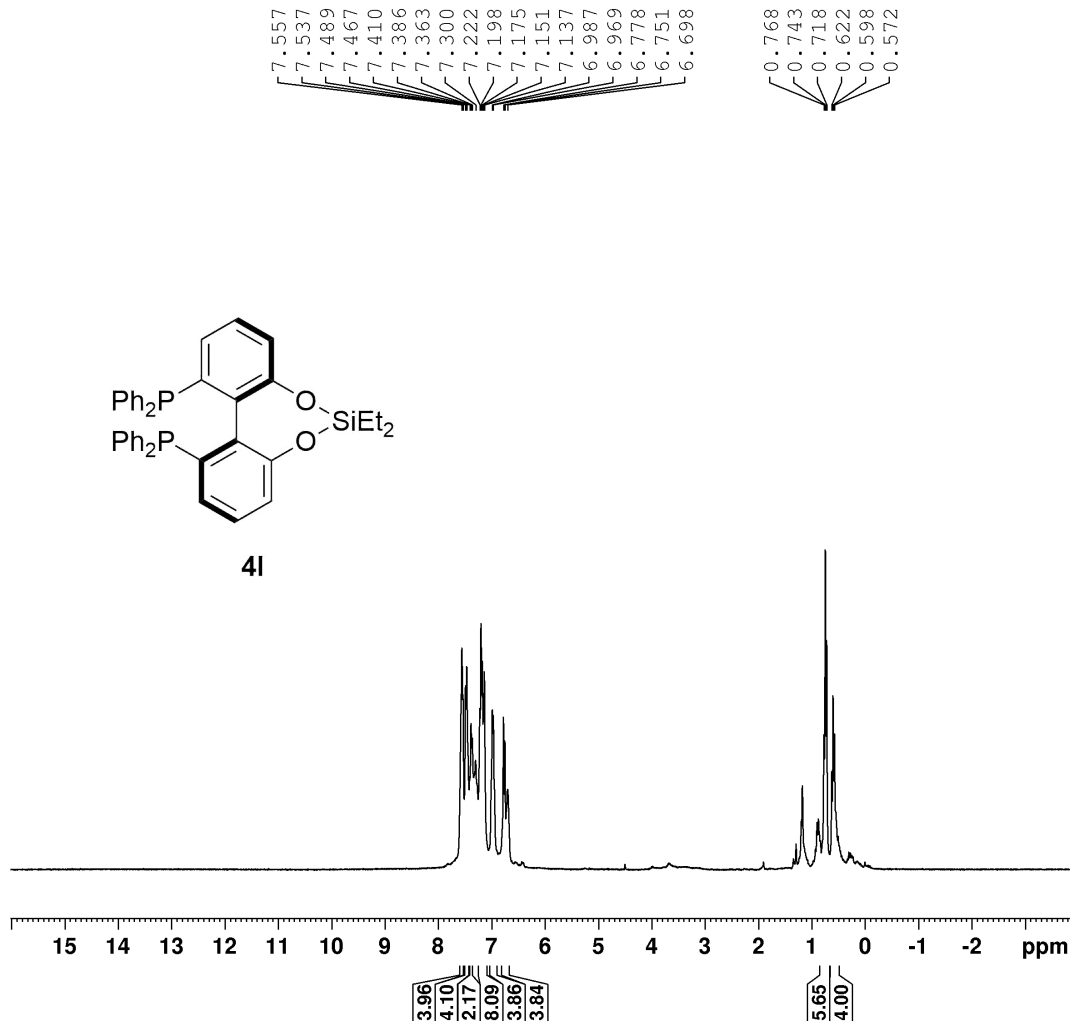
```
Current Data Parameters
NAME                  2024-400
EXPNO                       73
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20240425
Time                     16.30 h
INSTRUM                  spect
PROBHD    5 mm PABBO BB/
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                        1024
DS                           4
SWH                  24038.461 Hz
FIDRES                0.733596 Hz
AQ                    1.3631488 sec
RG                       206.33
DW                       20.800 usec
DE                        6.50 usec
TE                       298.2 K
D1                    2.00000000 sec
d11                   0.03000000 sec
DELTA                 1.89999998 sec
TD0                          1
SFO1                100.6504916 MHz
NUC1                        13C
P1                       10.00 usec
PLW1                54.00000000 W
SFO2                400.2416010 MHz
NUC2                        1H
CPDPRG[2                waltz16
PCPD2                    90.00 usec
PLW2                12.00000000 W
PLW12                 0.30294999 W
PLW13                 0.24539000 W

F2 - Processing parameters
SI                       32768
SF                  100.6404075 MHz
WDW                         EM
SSB                          0
LB                       1.00 Hz
GB                           0
PC                       1.40
```



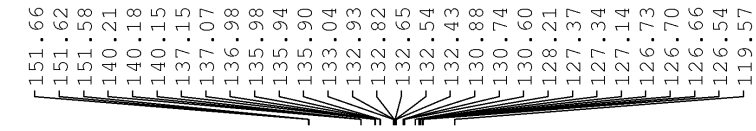

```
Current Data Parameters
NAME            2024-400
EXPNO                137
PROCNO                 1

F2 - Acquisition Parameters
Date_           20240718
Time               18.32 h
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     0
SWH             8012.820 Hz
FIDRES          0.489064 Hz
AQ             2.0447233 sec
RG                206.33
DW                62.400 usec
DE                 6.50 usec
TE                298.9 K
D1             2.00000000 sec
TD0                    1
SFO1         400.2424716 MHz
NUC1                  1H
P1                14.30 usec
PLW1         12.00000000 W

F2 - Processing parameters
SI                 65536
SF           400.2400111 MHz
WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                  1.00
```

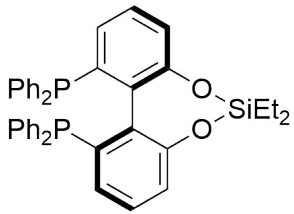



```
Current Data Parameters
NAME            2024-400
EXPNO                138
PROCNO                 1

F2 - Acquisition Parameters
Date_           20240718
Time               19.31 h
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1024
DS                     4
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ              1.3631488 sec
RG                206.33
DW                20.800 usec
DE                 6.50 usec
TE                299.7 K
D1             2.00000000 sec
d11            0.03000000 sec
DELTA          1.89999998 sec
TD0                    1
SFO1         100.6504916 MHz
NUC1                13C
P1                 10.00 usec
PLW1          54.00000000 W
SFO2         400.2416010 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2              90.00 usec
PLW2          12.00000000 W
PLW12          0.30294999 W
PLW13          0.24539000 W

F2 - Processing parameters
SI                 32768
SF           100.6404078 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

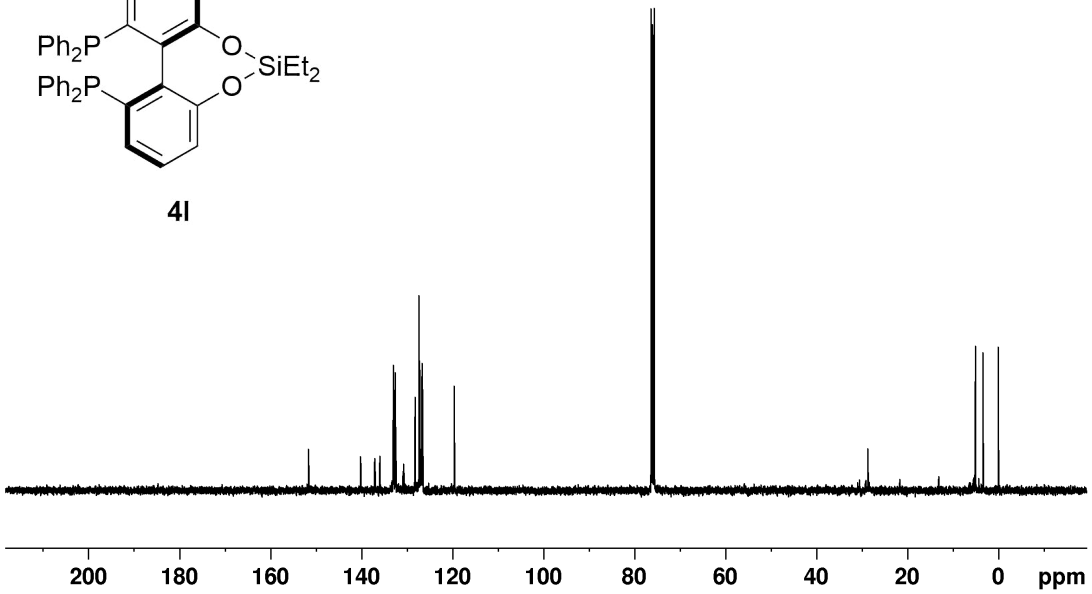



```
Current Data Parameters
NAME          2024-400-p
EXPNO                239
PROCNO                 1

F2 - Acquisition Parameters
Date_           20240618
Time               17.32 h
INSTRUM             spect
PROBHD   5 mm DUL 13C-1
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      0
SWH              6009.615 Hz
FIDRES           0.183399 Hz
AQ             5.4525952 sec
RG                209.09
DW                 83.200 usec
DE                  6.50 usec
TE                 302.9 K
D1            1.00000000 sec
TD0                    1
SFO1         300.1318534 MHz
NUC1                  1H
P1                 8.00 usec
PLW1         18.00000000 W

F2 - Processing parameters
SI                  65536
SF           300.1300443 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```

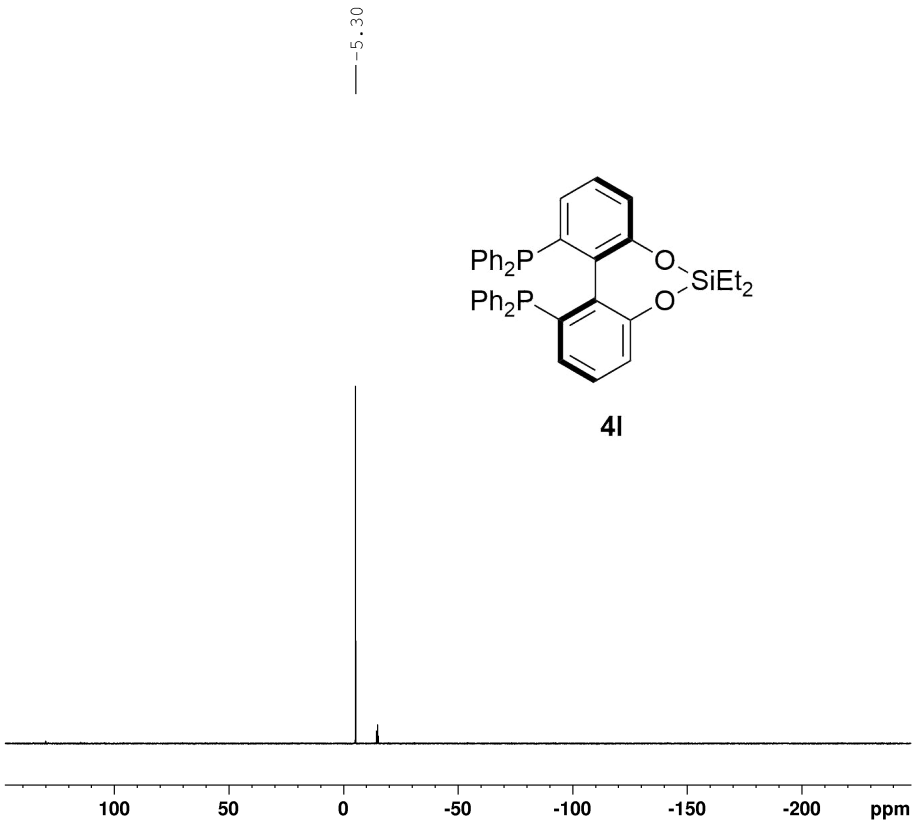




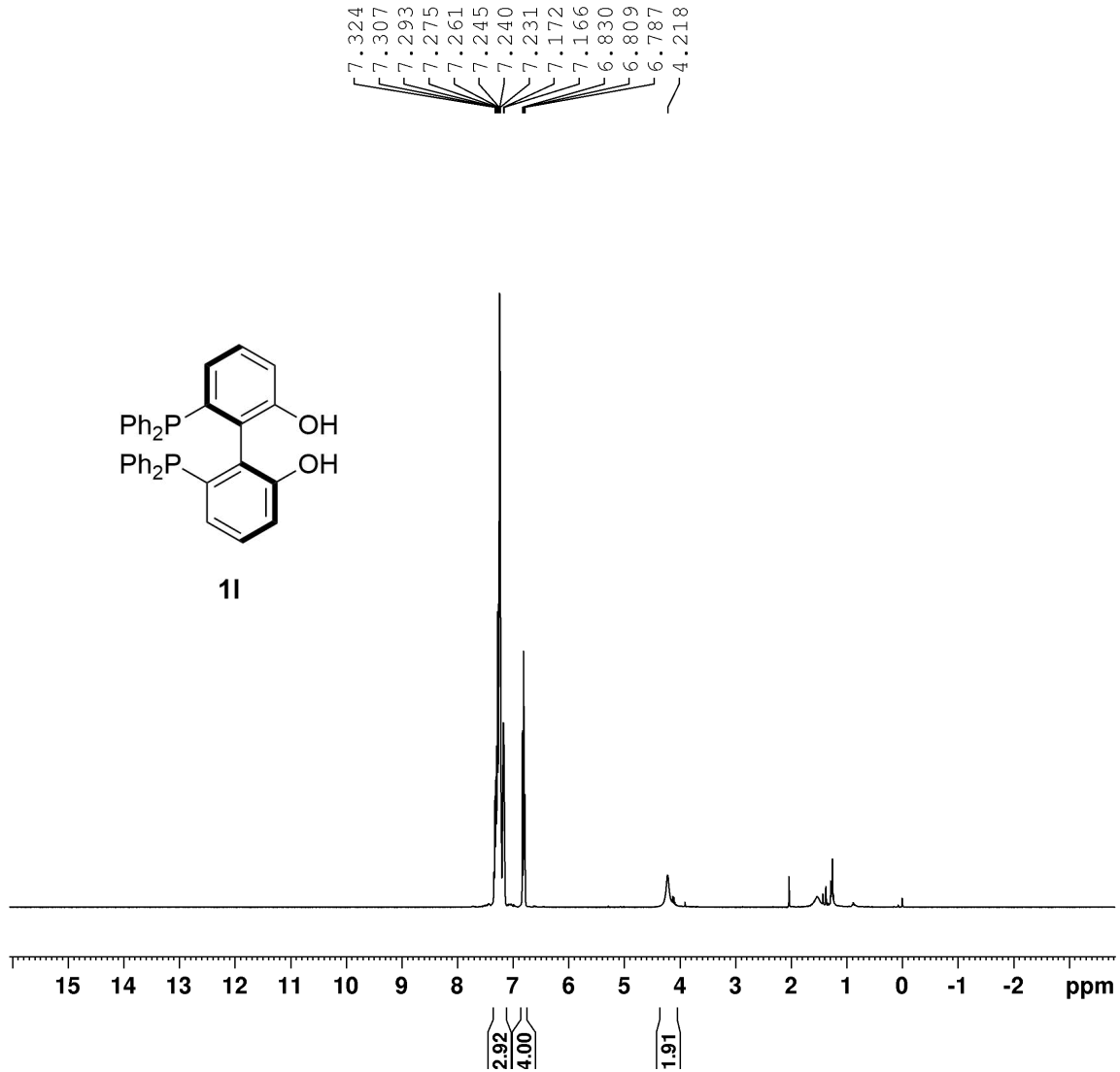
4l



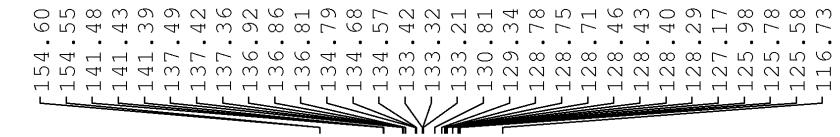

```
Current Data Parameters
NAME            2024-400
EXPNO                143
PROCNO                 1

F2 - Acquisition Parameters
Date_           20240815
Time                10.09 h
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   400
DS                     4
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ             1.3631488 sec
RG                206.33
DW                20.800 usec
DE                 6.50 usec
TE                299.1 K
D1             2.00000000 sec
d11            0.03000000 sec
DELTA          1.89999998 sec
TD0                    1
SFO1         100.6504916 MHz
NUC1                 13C
P1                10.00 usec
PLW1         54.00000000 W
SFO2         400.2416010 MHz
NUC2                 1H
CPDPRG[2          waltz16
PCPD2             90.00 usec
PLW2         12.00000000 W
PLW12         0.30294999 W
PLW13         0.24539000 W

F2 - Processing parameters
SI                 32768
SF           100.6405366 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```



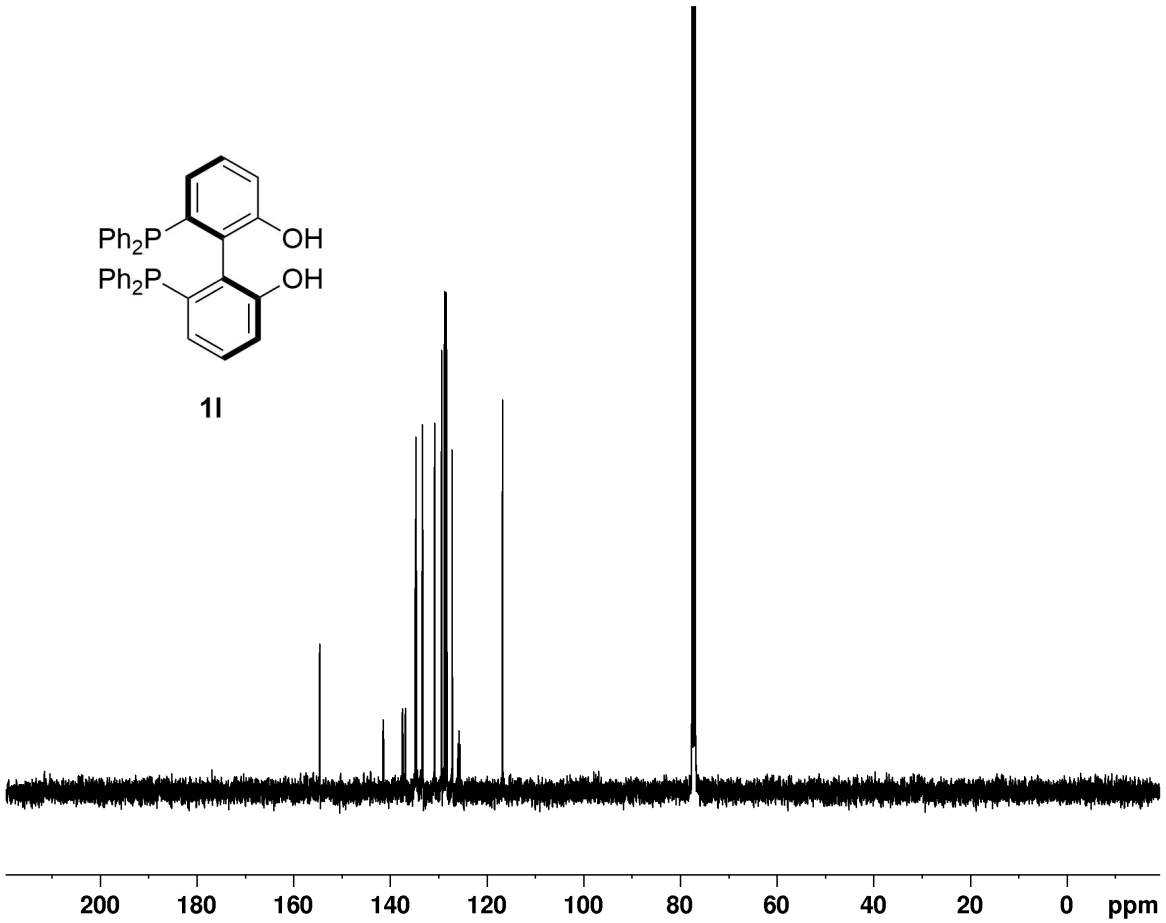

```
Current Data Parameters
NAME          2024-400-p
EXPNO                  5
PROCNO                 1

F2 - Acquisition Parameters
Date_           20240815
Time               10.35 h
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                    4
DS                    4
SWH           64102.563 Hz
FIDRES         1.956255 Hz
AQ            0.5111808 sec
RG               206.33
DW                7.800 usec
DE                6.50 usec
TE                298.6 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                   1
SFO1        162.0120208 MHz
NUC1                31P
P1                14.60 usec
PLW1        13.00000000 W
SFO2        400.2416010 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2        12.00000000 W
PLW12        0.30294999 W
PLW13        0.24539000 W

F2 - Processing parameters
SI                32768
SF          162.0201218 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```



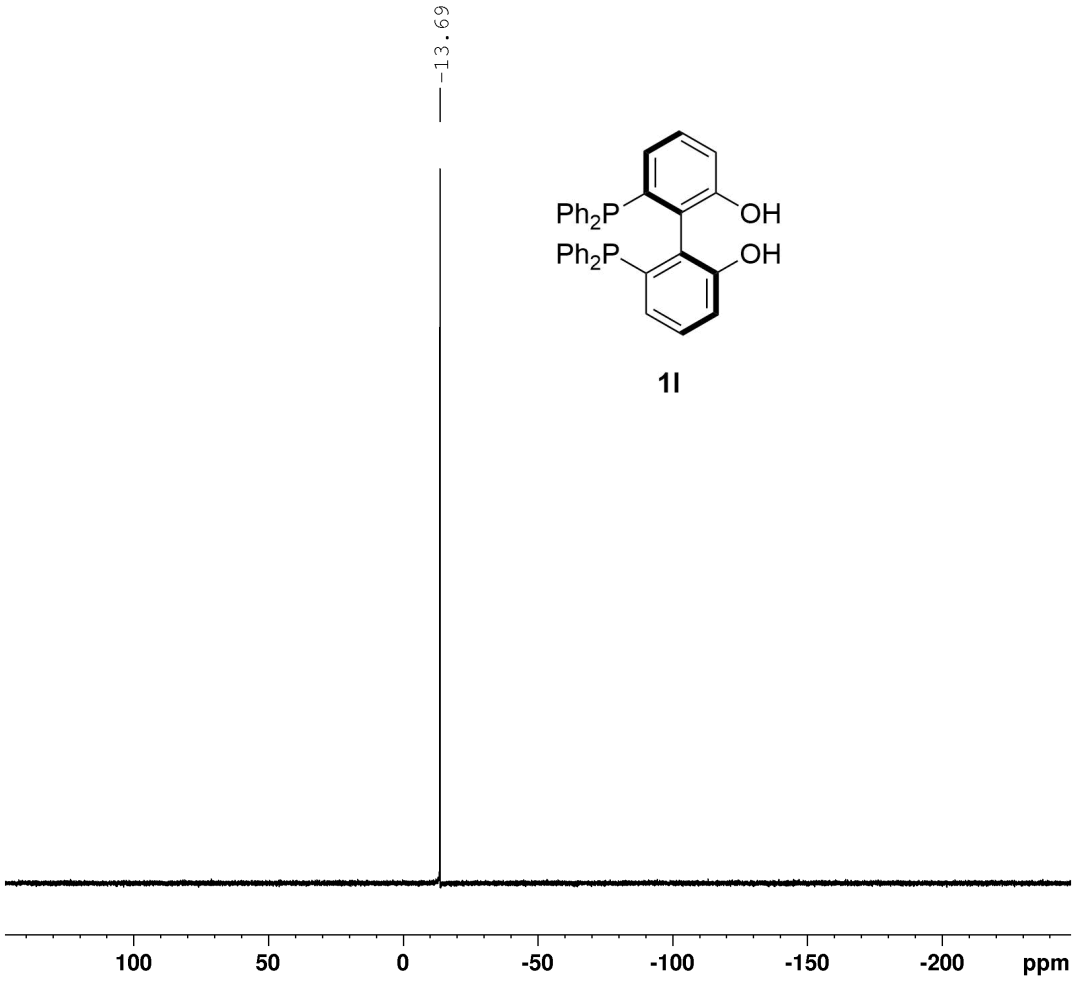

```
Current Data Parameters
NAME            2024-400
EXPNO                119
PROCNO                 1

F2 - Acquisition Parameters
Date_           20240618
Time                3.40 h
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     0
SWH             8012.820 Hz
FIDRES          0.489064 Hz
AQ             2.0447233 sec
RG                206.33
DW                62.400 usec
DE                 6.50 usec
TE                298.9 K
D1            2.00000000 sec
TD0                    1
SFO1         400.2424716 MHz
NUC1                  1H
P1                14.30 usec
PLW1         12.00000000 W

F2 - Processing parameters
SI                 65536
SF           400.2400152 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



1l

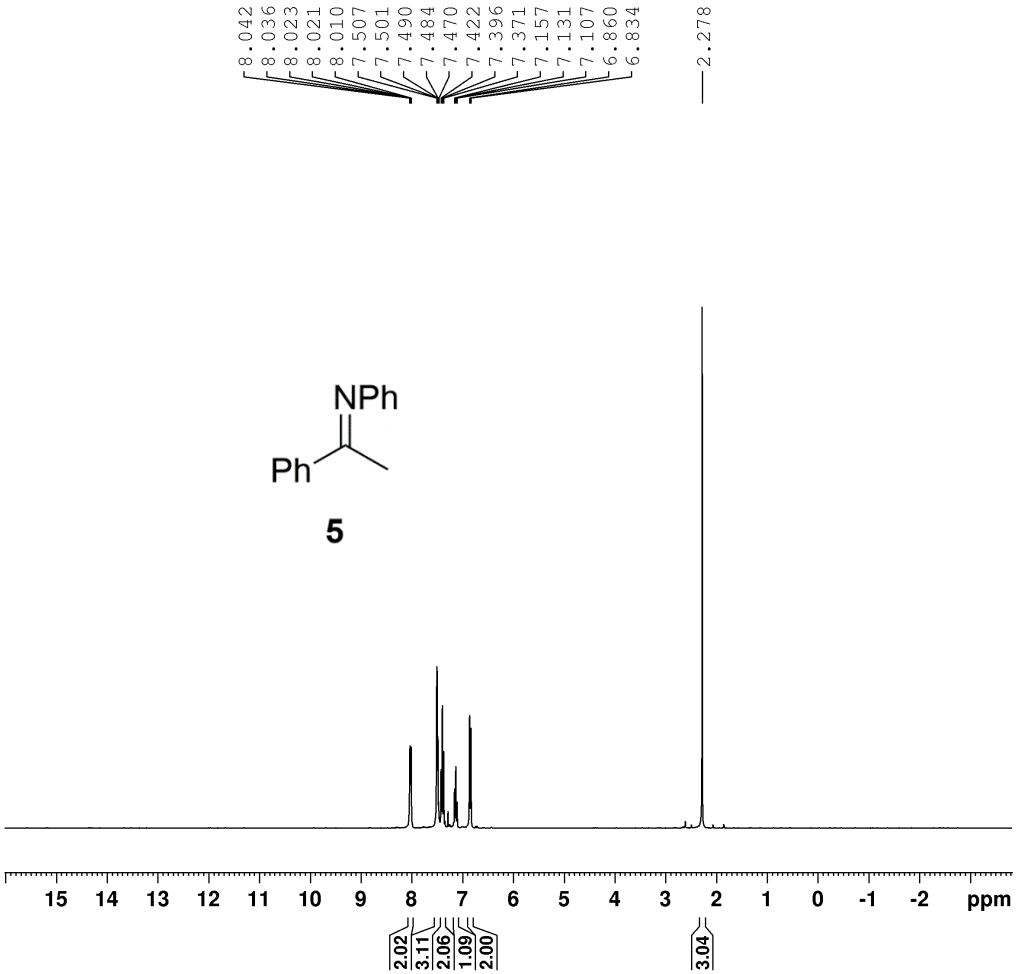



```
Current Data Parameters
NAME            2024-400
EXPNO                120
PROCNO                 1

F2 - Acquisition Parameters
Date_           20240618
Time                5.06 h
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1500
DS                     4
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ              1.3631488 sec
RG                 206.33
DW                 20.800 usec
DE                  6.50 usec
TE                 299.5 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                    1
SFO1         100.6504916 MHz
NUC1                13C
P1                 10.00 usec
PLW1         54.00000000 W
SFO2         400.2416010 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2              90.00 usec
PLW2         12.00000000 W
PLW12         0.30294999 W
PLW13         0.24539000 W

F2 - Processing parameters
SI                 32768
SF           100.6404090 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

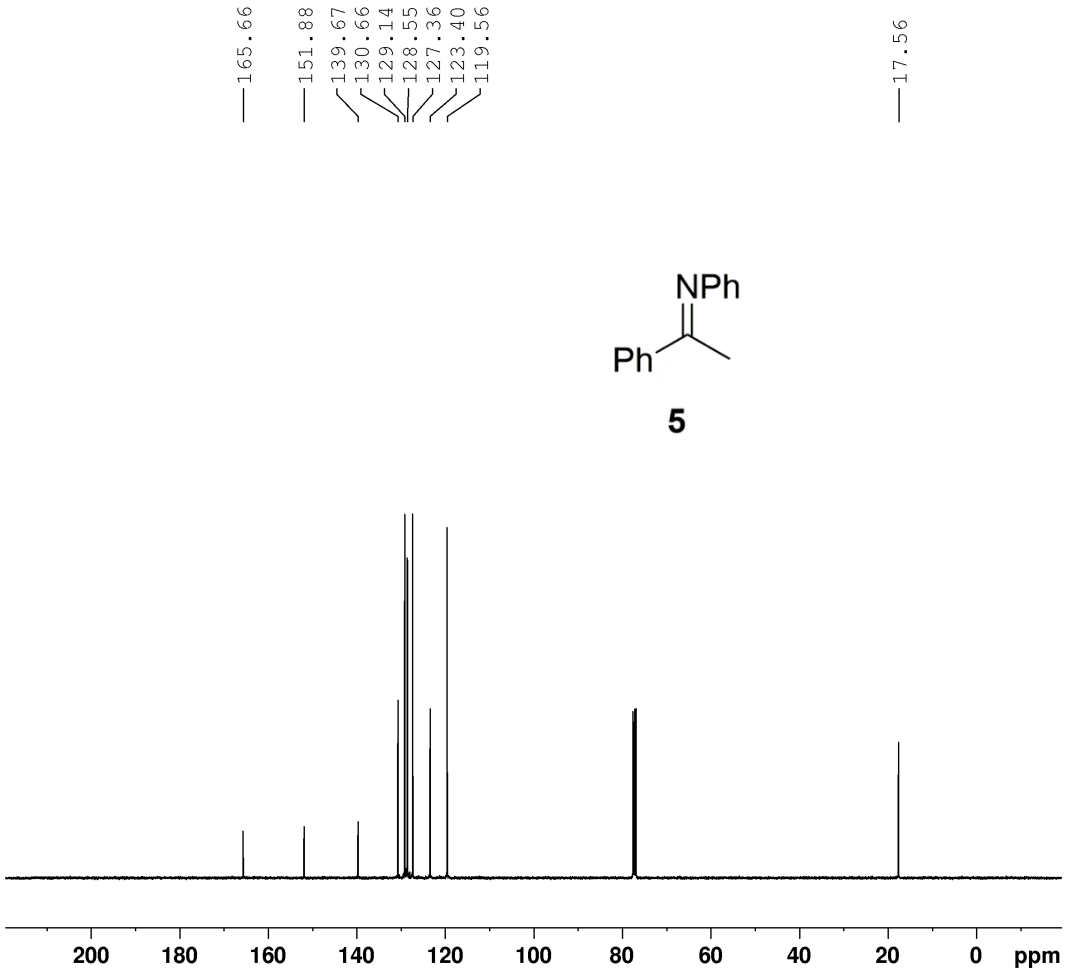


1l

```
Current Data Parameters
NAME          2024-400-p
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20240619
Time               22.47 h
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                     7
DS                     4
SWH            64102.563 Hz
FIDRES          1.956255 Hz
AQ             0.5111808 sec
RG                206.33
DW                 7.800 usec
DE                 6.50 usec
TE                298.8 K
D1             2.00000000 sec
d11            0.03000000 sec
DELTA          1.89999998 sec
TD0                    1
SFO1         162.0120208 MHz
NUC1                31P
P1                14.60 usec
PLW1         13.00000000 W
SFO2         400.2416010 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         12.00000000 W
PLW12         0.30294999 W
PLW13         0.24539000 W

F2 - Processing parameters
SI                 32768
SF           162.0201218 MHz
WDW                  EM
SSB                  0
LB                1.00 Hz
GB                   0
PC                1.40
```

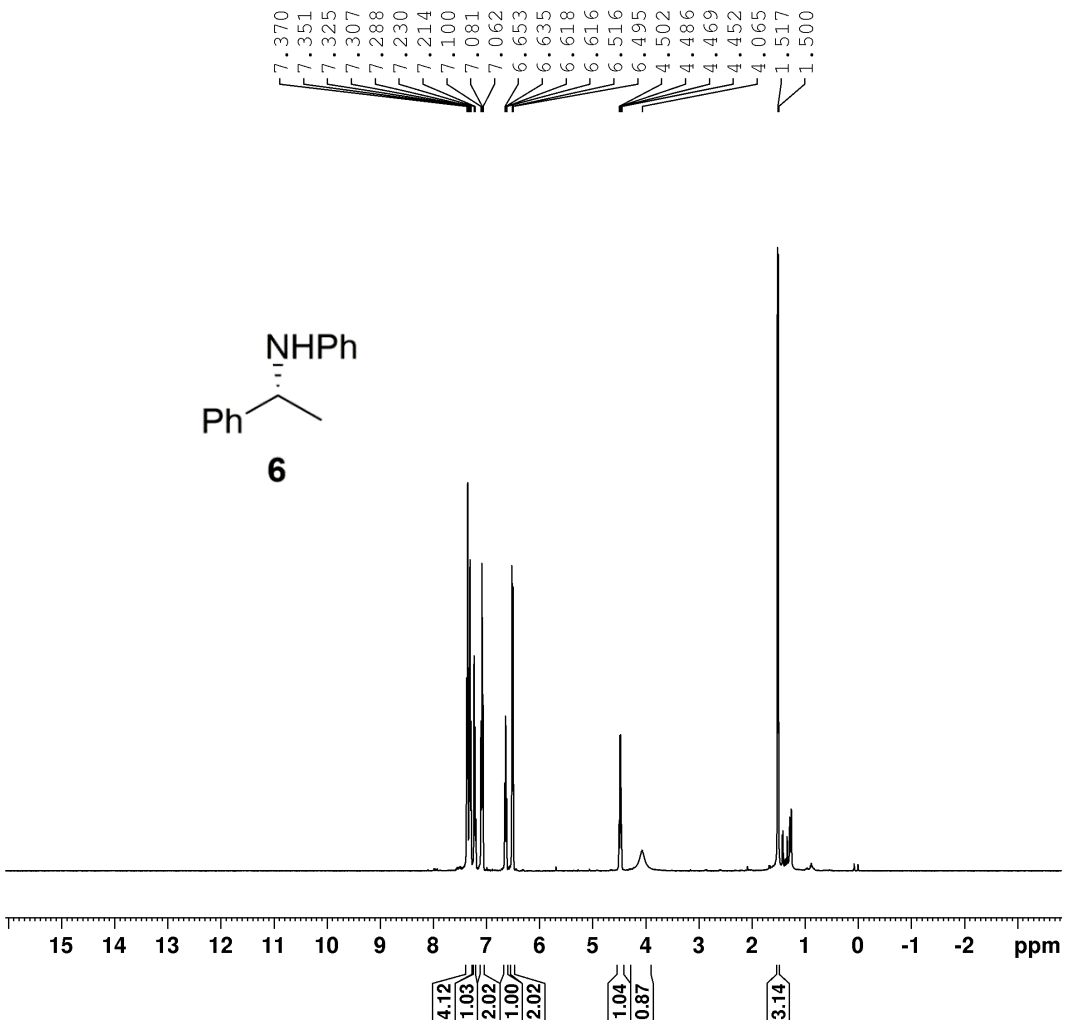



```
Current Data Parameters
NAME         ZZJ-445-NOE
EXPNO                  6
PROCNO                 1

F2 - Acquisition Parameters
Date_           20241230
Time               16.15 h
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     0
SWH             6009.615 Hz
FIDRES          0.183399 Hz
AQ              5.4525952 sec
RG                209.09
DW                83.200 usec
DE                 6.50 usec
TE                298.3 K
D1             1.00000000 sec
TD0                    1
SFO1         300.1318534 MHz
NUC1                  1H
P1                 8.00 usec
PLW1         18.00000000 W

F2 - Processing parameters
SI                 65536
SF           300.1300000 MHz
WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                 1.00
```

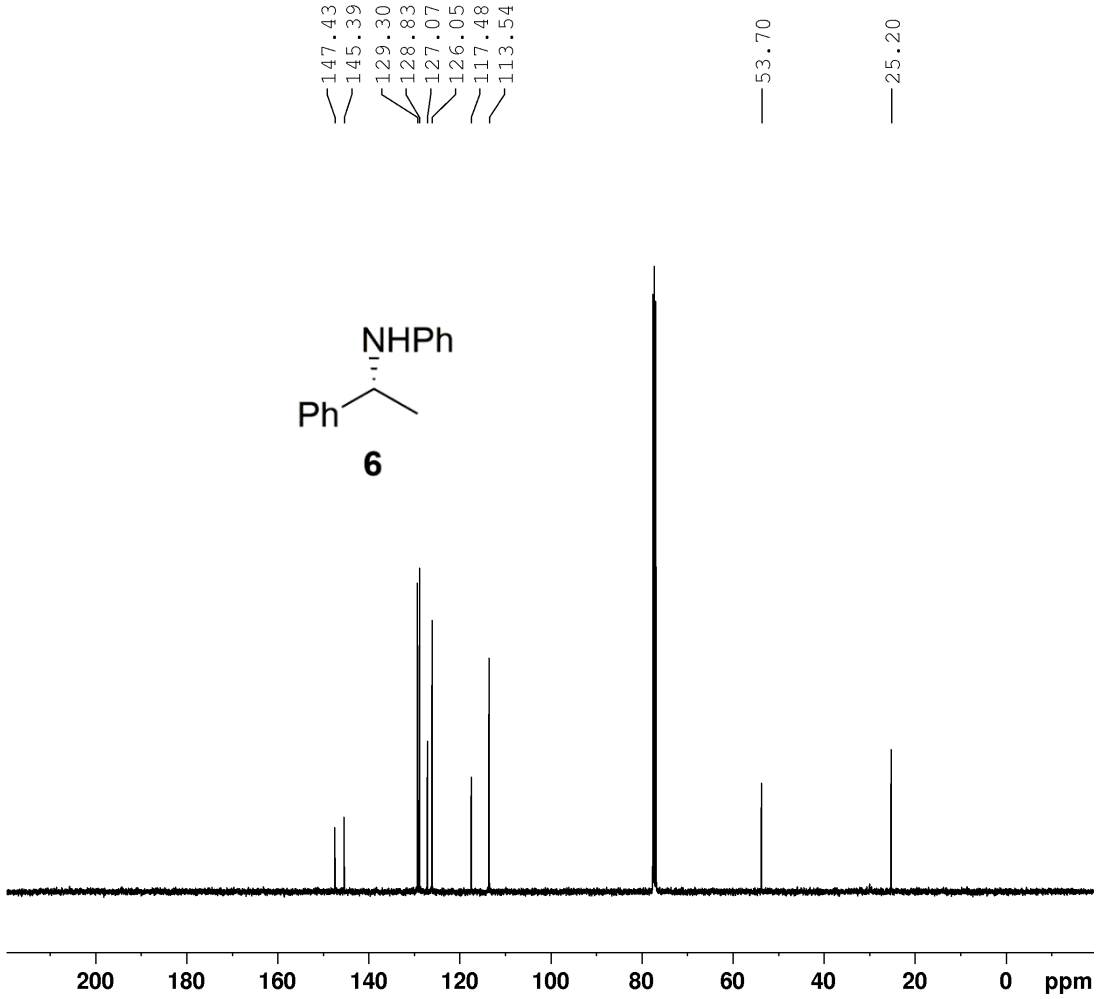


5

```
Current Data Parameters
NAME         ZZJ-445-NOE
EXPNO                  8
PROCNO                 1

F2 - Acquisition Parameters
Date_           20241230
Time              19.11 h
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG           zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ            1.3631488 sec
RG               206.33
DW               20.800 usec
DE                6.50 usec
TE               298.0 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                   1
SFO1        100.6504916 MHz
NUC1                13C
P1               10.00 usec
PLW1        54.00000000 W
SFO2        400.2416010 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2        12.00000000 W
PLW12        0.30294999 W
PLW13        0.24539000 W

F2 - Processing parameters
SI                32768
SF          100.6404126 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```




```
Current Data Parameters
NAME            2024-400
EXPNO                117
PROCNO                 1

F2 - Acquisition Parameters
Date_           20240618
Time                2.37 h
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                     8
DS                     0
SWH              8012.820 Hz
FIDRES           0.489064 Hz
AQ             2.0447233 sec
RG                116.67
DW                62.400 usec
DE                 6.50 usec
TE                298.8 K
D1            2.00000000 sec
TD0                    1
SFO1         400.2424716 MHz
NUC1                  1H
P1                14.30 usec
PLW1         12.00000000 W

F2 - Processing parameters
SI                 65536
SF           400.2400216 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```




```
Current Data Parameters
NAME            2024-400
EXPNO                118
PROCNO                 1

F2 - Acquisition Parameters
Date_           20240618
Time                3.36 h
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1024
DS                     4
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ             1.3631488 sec
RG                206.33
DW                20.800 usec
DE                 6.50 usec
TE                299.5 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                    1
SFO1         100.6504916 MHz
NUC1                13C
P1                10.00 usec
PLW1         54.00000000 W
SFO2         400.2416010 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         12.00000000 W
PLW12         0.30294999 W
PLW13         0.24539000 W

F2 - Processing parameters
SI                 32768
SF           100.6404105 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```