Organic & Biomolecular Chemistry

Electronic Supplementary Information

1,2-Di(phenylethynyl)ethenes with axially chiral, 2,2′-bridged 1,1′-binaphthyl substituents: potent cholesteric liquid-crystal inducers

Yi-Lin Wu,[a] Fiammetta Ferroni,[b] Silvia Pieraccini,[b] W. Bernd Schweizer,[a] Brian B. Frank,[a] Gian Piero Spada[b] and François Diederich*[a]

[a] Laboratorium für Organische Chemie, ETH Zürich, Hönggerberg, HCI, CH-8093 Zürich, Switzerland.

[b] Alma Mater Studiorum–Università di Bologna, Dipartimento di Chimica Organica "A. Mangini", Via San Giacomo 11, I-40126 Bologna, Italy.

Table of contents

1. UV/Vis spectral changes of (*E*)- and (*Z*)-(*S*,*S*)-5c upon protonation

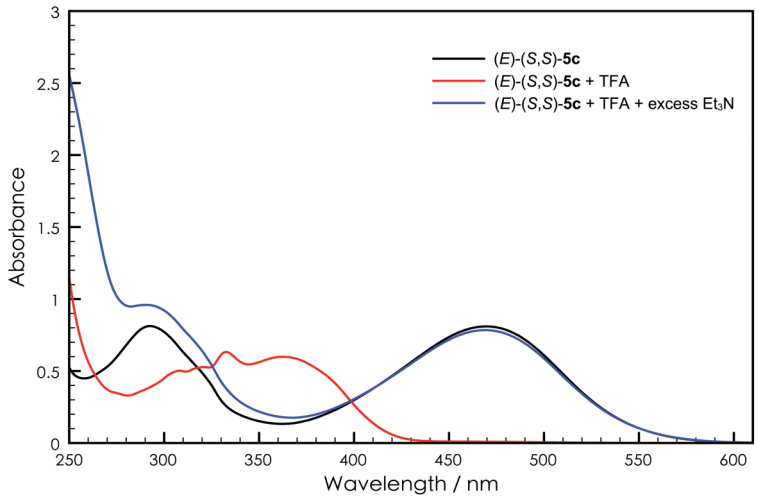


Fig. ESI1 UV/Vis spectrum of (*E*)-(*S*,*S*)-**5c** in CH_2Cl_2 (black line), spectrum taken after the addition of trifluoroacetic acid (red line) and spectrum taken after the addition of excess Et_3N to neutralise the added acid (blue line).

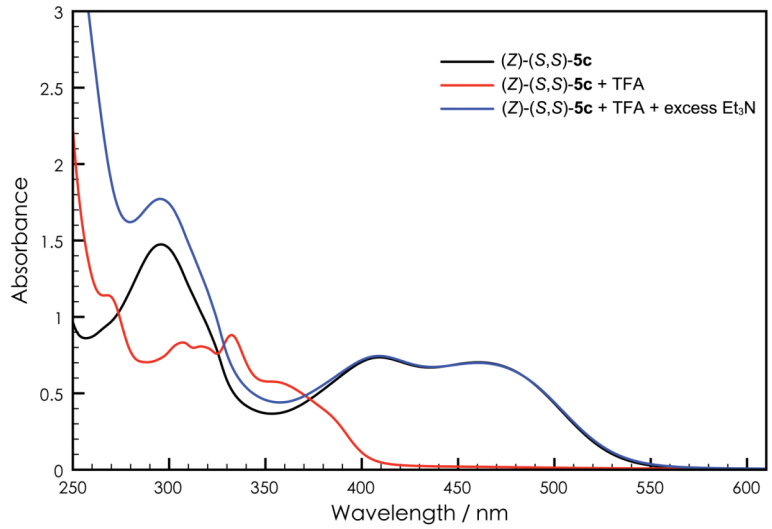


Fig. ESI2 UV/Vis spectrum of (*Z*)-(*S*,*S*)-**5c** in CH_2Cl_2 (black line), spectrum taken after the addition of trifluoroacetic acid (red line) and spectrum taken after the addition of excess Et_3N to neutralise the added acid (blue line).

2. Emission spectrum of (*E*)-(*R*,*R*)-6b

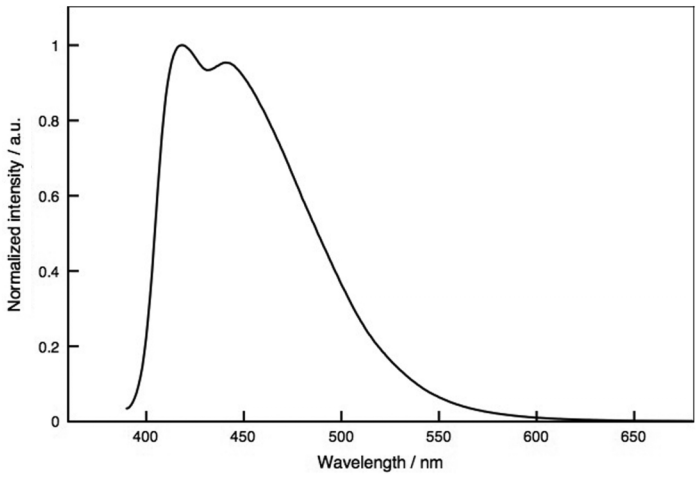


Fig. ESI3 Emission spectrum of (*E*)-(*R*,*R*)-**6b** in CH_2Cl_2, λ_{exc} = 382 nm.

3. ^{1}H NMR of Hg-lamp-irradiated (*S*,*S*)-5c

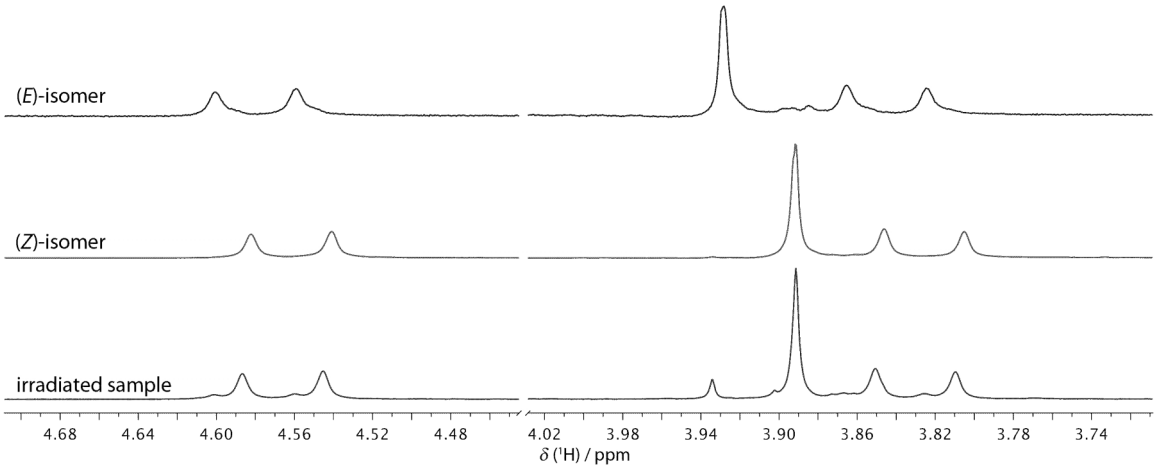


Fig. ESI4 Bottom: partial ^{1}H NMR (300 MHz, $CDCl_3$) spectrum of the sample of (*S*,*S*)-**5c** after irradiation with a medium pressure Hg lamp for 4 h in deoxygenated CH_2Cl_2. Top and middle: partial ^{1}H NMR spectra of configurationally pure (*E*) and (*Z*)-isomers for comparison. The singlet at ~3.9 ppm is assigned to the protons of methyl ester; two doublets at ~3.82 and 4.56 ppm originate from the two CH_2 groups of the dihydroazepine ring.

4. Photoisomerisation kinetics

The photoisomerisation kinetics of (*E*)-(*S*,*S*)-**5c** and (*E*)-(*R*,*R*)-**6b** upon irradiation at 472 and 382 nm, respectively, were subjected to quantitative study.[1,2] The time-dependence of the absorbance ($A_\lambda(t)$) was extracted from a series of UV/Vis spectra (Fig. 6 in the main text) taken at defined time periods during the course of irradiation. When the photostationary state (PSS) was reached, the composition of each sample was analysed by HPLC to determine the ratio between the concentrations of (*Z*)- and (*E*)-isomers (C_Z^{eq} and C_E^{eq}; Fig. ESI5), which is also the photo-equilibrium constant ($K_{\lambda exc}^{eq}$) and equal to the ratio between partial rate constants of isomerisation ($k_{E\to Z}$ and $k_{Z\to E}$) (Eq. 1).

$$K_{\lambda exc}^{eq} = \frac{C_Z^{eq}}{C_E^{eq}} = \frac{k_{E\to Z}}{k_{Z\to E}} \qquad (1)$$

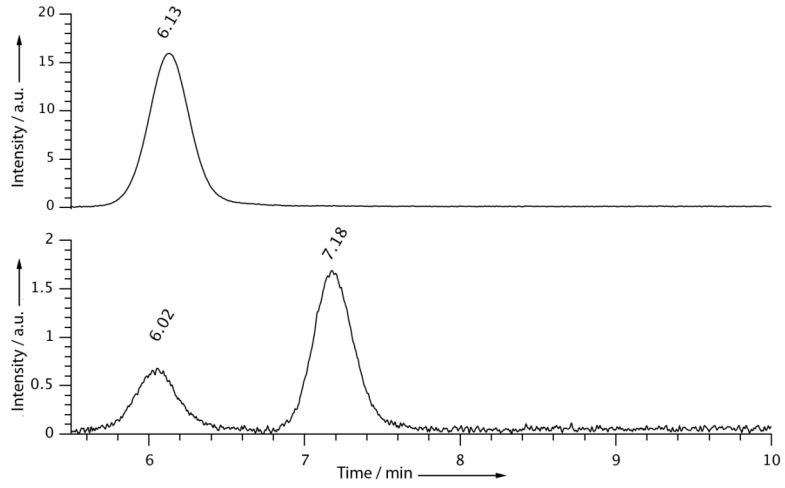


Fig. ESI5 HPLC chromatograms of (*S*,*S*)-**5c** as a representative example of the sample before irradiation (top) and at the photostationary state (bottom).

The change in the absorbance during irradiation follows first-order kinetics, and is expressed by Eq. 2,

$$A_\lambda(t) = \varepsilon_E C_E^{eq} + \varepsilon_Z C_Z^{eq} + (\varepsilon_E - \varepsilon_Z) C_Z^{eq} \times \exp(-k_{tot}\, t) \qquad (2)$$

where ε_E (known) and ε_Z are the extinction coefficients of (*E*)- and (*Z*)-isomers, respectively, at the wavelength λ of interest (often, but not necessarily, the same as the absorption maximum and the excitation wavelength), k_{tot} is the total rate constant, equal to the sum of partial rate constants of each isomerisation processes ($k_{tot} = k_{E\to Z} + k_{E\to Z}$), and individual C_Z^{eq} and C_E^{eq} were calculated from Eq. 1 with the knowledge of the known, initial concentration. By fitting the plot of $A_\lambda(t)$ vs. time (*t*) to Eq. 2, partial rate constants $k_{E\to Z}$ and $k_{Z\to E}$, as well as ε_Z can be obtained (Fig. ESI6).

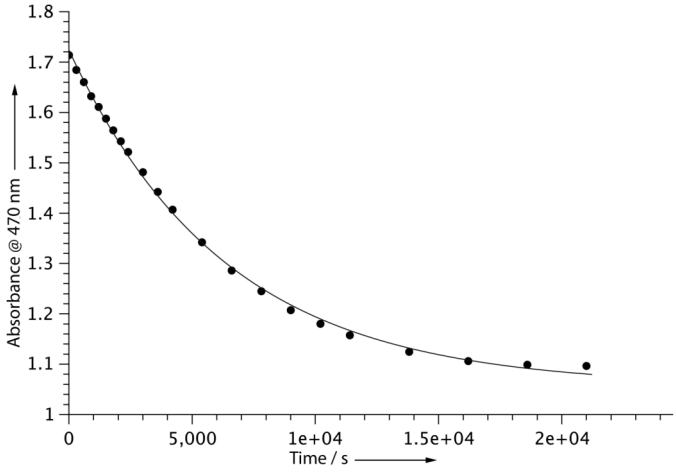


Fig. ESI6 The time dependence of the absorbance of (*S*,*S*)-**5c** at 470 nm in CH_2Cl_2 by irradiation at 472 nm, and the curve fitting according to first-order kinetics (Eq. 2).

To obtain photoisomerisation quantum yields, the light intensity (I_0) at the irradiation wavelength in each experiment was measured by a chemical actinometry method with potassium ferrioxalate;[3] $I_0 = 10^{-(9-10)}$ Einstein s^{-1} is usually observed. With this information, the partial quantum yields (Φ) are then calculated according to Eqs. 3.1 and 3.2,

$$\Phi_{E\rightarrow Z} = \frac{k_{E\rightarrow Z} V A^{\text{eq}}_{\lambda_{\text{exc}}}}{I_0 (1 - 10^{-A^{\text{eq}}_{\lambda_{\text{exc}}}}) \varepsilon_E} \qquad (3.1)$$

$$\Phi_{Z\rightarrow E} = \frac{k_{Z\rightarrow E} V A^{\text{eq}}_{\lambda_{\text{exc}}}}{I_0 (1 - 10^{-A^{\text{eq}}_{\lambda_{\text{exc}}}}) \varepsilon_Z} \qquad (3.2)$$

where V is the volume of the irradiated sample and $A^{\text{eq}}_{\lambda\text{exc}}$ at excitation wavelength the absorbance of the sample at the PSS.

5. UV/Vis spectral changes of (*E*)-(*R*,*R*)-6a during irradiation and the HPLC chromatogram of the irradiated sample

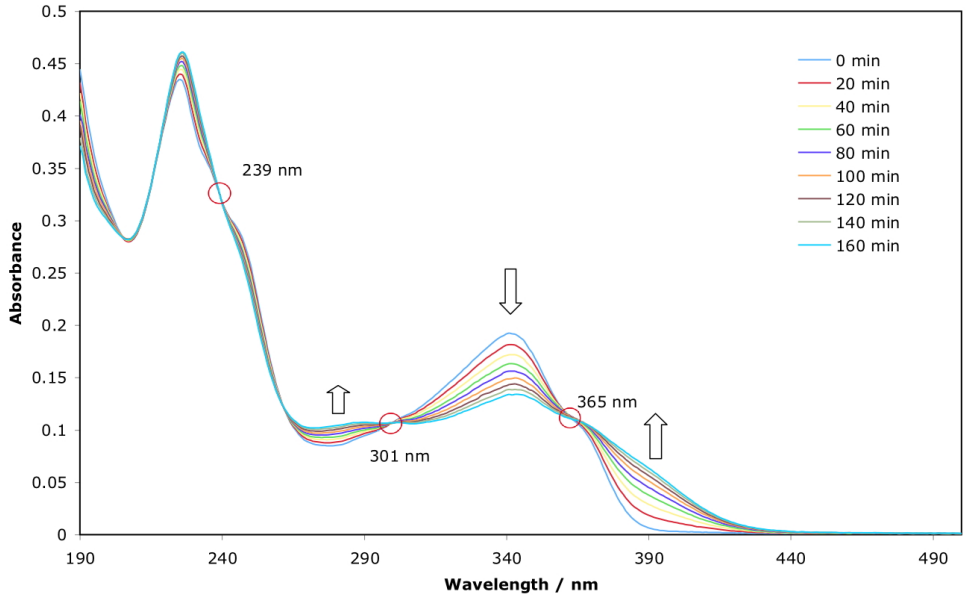


Fig. ESI7 UV/Vis spectral changes (indicated by arrows) of (*E*)-(*R*,*R*)-**6a** in CH_2Cl_2 during the course of excitation at 341 nm.

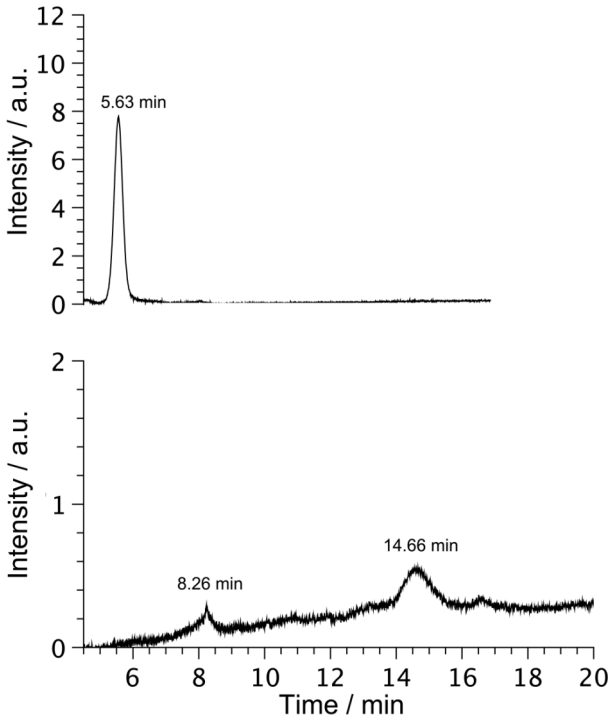


Fig. ESI8 HPLC chromatograms of (*E*)-(*R*,*R*)-**6a** before (top) and after (bottom) irradiation at 341 nm.

6. Synthetic details

(*S*,*S*)-4,4′-{[(3*E*)-3,4-Bis({[*tert*-butyl(dimethyl)silyl]oxy}methyl)hex-3-ene-1,5-diyne-1,6-diyl]di-4,1-phenylene}bis(4,5-dihydro-3*H*-dinaphtho[2,1-*c*:1′,2′-e]azepine) ((*E*)-(*S*,*S*)-5a). To a de-oxygenated solution of (*E*)-**8** (25 mg, 0.069 mmol) and (*S*)-**7** (70 mg, 0.141 mmol) in $^{i}Pr_2NH$ (1 cm^3) and THF (1 cm^3), $[PdCl_2(PPh_3)_2]$ (2 mg, 0.003 mmol) and CuI (4 mg, 0.021 mmol) were added, and the resulting suspension was stirred for 6 h at r.t. in the dark. The mixture was filtered through a SiO_2 pad (CH_2Cl_2), and the solvents were evaporated under reduced pressure. The residue was purified by FC (SiO_2; heptane/ethyl acetate 95:5) (16 mg, 21%). Since the product decomposed gradually in solution and in the solid state, full characterisation was not attempted. δ_H (300 MHz; $CDCl_3$) 0.17 (12 H, s, $SiMe_2$), 0.97 (18 H, s, Si^{t}Bu), 3.84 (4 H, d, *J* 12.3, NCH_2), 4.56 (4 H, d, *J* 12.3, NCH_2), 4.64 (4 H, s, OCH_2), 6.89 (4 H, d, *J* 8.8, NC_6H_4), 7.27–7.38 (8 H, m, Ar-H), 7.46–7.55 (12 H, m, Ar-H), 7.92–7.97 (8 H, m, Ar-H); HR-MALDI-MS *m/z* 1103.5339 (100%, $[M+H]^+$, calcd for $C_{76}H_{75}N_2O_2Si_2^+$: 1103.5362), 1102.5265 (56, M^+).

(*S*,*S*)-4,4′-({(3*E*)-3,4-Bis[(triisopropylsilyl)ethynyl]hex-3-ene-1,5-diyne-1,6-diyl}di-4,1-phenylene)bis(4,5-dihydro-3*H*-dinaphtho[2,1-*c*:1′,2′-*e*]azepine) ((*E*)-(*S*,*S*)-5b). To a de-oxygenated solution of (*E*)-**9** (20 mg, 0.046 mmol) and (*S*)-**7** (53 mg, 0.107 mmol) in Et_3N (6 cm^3) and THF (2 cm^3), $[PdCl_2(PPh_3)_2]$ (2 mg, 0.003 mmol) and CuI (4 mg, 0.021 mmol) were added, and the resulting suspension was stirred for 14 h at r.t. in the dark. The mixture was filtered through a plug (SiO_2; CH_2Cl_2), and the solvents were evaporated under reduced pressure. The residue was purified by FC (SiO_2;

hexane/CH_2Cl_2 80:20 → 80:35) (30 mg, 55%). Since the product decomposed gradually in solution and in the solid state, full characterisation was not attempted. δ_H (300 MHz; $CDCl_3$) 1.14 (42 H, s, Si^{i}Pr$_3$), 3.85 (4 H, d, *J* 12.3, NCH_2), 4.56 (4 H, d, *J* 12.3, NCH_2), 6.88 (4 H, d, *J* 8.7, NC_6H_4), 7.27–7.34 (4 H, m, naphthalene), 7.38 (4 H, d, *J* 8.7, NC_6H_4), 7.46–7.57 (12 H, m, naphthalene), 7.93–7.97 (8 H, m, Ar-H); HR-MALDI-MS *m/z* 1175.6028 (100%, $[M+H]^+$, calcd for $C_{84}H_{83}N_2Si_2^+$: 1175.6089), 1174.5982 (60, M^+).

(*S*)-4-(4-Ethynylphenyl)-4,5-dihydro-3*H*-dinaphtho[2,1-*c*:1′,2′-*e*]azepine ((*S*)-10). A solution of (*S*)-**7** (250 mg, 0.5 mmol), $[PdCl_2(PPh_3)_2]$ (20 mg, 0.03 mmol) and CuI (10 mg, 0.05 mmol) in Et_3N/THF (1:1, 30 cm^3) was purged with Ar for 20 min, then treated with trimethylsilylacetylene (0.1 cm^3, 0.72 mmol), stirred at r.t. for 10 h and filtered through a pad of SiO_2 (CH_2Cl_2). Evaporation of the filtrate and FC (SiO_2; hexane/CH_2Cl_2 5:1) gave the $SiMe_3$-protected acetylene intermediate (230 mg, 98%). A suspension of K_2CO_3 (440 mg, 3.19 mmol) in THF/methanol (1:1, 20 cm^3) was purged with Ar for 20 min, charged with TMS-protected acetylene (148 mg, 0.32 mmol) and stirred at r.t. for 3 h. After dilution with water, the aqueous solution was extracted with CH_2Cl_2. The combined organic layers were dried over Na_2SO_4 and evaporated. FC (SiO_2; CH_2Cl_2/hexane 1:3 → 1:2) gave (*S*)-**10** (654 mg, 90%) as a white solid (which sometimes turned dark). Mp 105–107 °C; $[\alpha]_D^{20}$ –562 (*c* 0.10 in $CHCl_3$); UV/Vis λ_{max}(MeCN)/nm 217 (ε/dm^3 mol^{-1} cm^{-1} 102200), 227sh (74300), 290 (38900); CD λ_{max}(MeCN)/nm 216 ($\Delta\varepsilon$/dm^3 mol^{-1} cm^{-1} –365), 228 (+455), 257 (+34), 291 (–33), 312 (–32); $\tilde{\nu}_{max}$(ATR)/cm^{-1} 3284, 3046, 2923, 2849, 2100, 1601, 1507, 1249, 1176, 816, 751;

δ_H (300 MHz; $CDCl_3$) 3.00 (1 H, s, CCH), 3.82 (2 H, d, *J* 12.3, NCH_2), 4.54 (2 H, d, *J* 12.3, NCH_2), 6.86 (2 H, d, *J* 9.0, NC_6H_4), 7.26–7.33 (2 H, m, naphthalene), 7.39 (2 H, d, *J* 9.0, NC_6H_4), 7.45–7.53 (6 H, m, naphthalene), 7.91–7.96 (4 H, m, naphthalene); δ_C (75 MHz; $CDCl_3$) 52.07, 75.24, 84.47, 110.70, 114.48, 125.73, 126.01, 127.36, 127.47, 128.33, 129.03, 131.30, 133.21, 133.26, 134.75, 149.55 (16 out of 17 expected peaks were found); HR-ESI-MS *m/z* 397.1767 (34%), 396.1737 (100, $[M+H]^+$, calcd for $C_{30}H_{22}N^+$: 396.1747), 219.1869 (33).

(*S*,*S*)-(4*E*)-4,5-Bis{[4-(3,5-dihydro-4*H*-dinaphtho[2,1-*c*:1′,2′-*e*]azepin-4-yl)phenyl]ethynyl}oct-4-ene-2,6-diynedial ((*E*)-(*S*,*S*)-5d). A 1:2 mixture of anhydrous Et_3N/THF (10 cm^3) was freshly subjected to 5 freeze-pump-thaw cycles before use. The inner atmosphere of a flask containing (*E*)-**12** (40 mg, 0.165 mmol), (*S*)-**10** (131 mg, 0.331 mmol), CuI (3 mg, 0.015 mmol) and $[Pd(PPh_3)_4]$ (10 mg, 0.009 mmol) was changed to Ar by three evacuation-refilling cycles. The oxygen-free Et_3N/THF mixture was then transferred to the above flask (the solution was coloured immediately) and the mixture stirred at r.t. for 10 h under Ar. The crude mixture was passed through a pad of SiO_2 (CH_2Cl_2). The filtrate was concentrated under reduced pressure and the residue purified by FC (SiO_2; CH_2Cl_2/hexane 1:1 → 6:1) to afford (*E*)-(*S*,*S*)-**5d** as a purple solid (63 mg, 44%). All the above operations were performed with a minimum exposure to laboratory light. However, the product was found to decompose gradually both in solution and in the solid state and full characterisation was not attempted. δ_H (300 MHz; $CDCl_3$) 3.82 (4 H, d, *J* 12.4, NCH_2), 4.58 (4 H, d, *J* 12.4, NCH_2), 6.85 (4 H, d, *J* 8.7,

NC_6H_4), 7.26–7.32 (4 H, m, Ar-H), 7.44–7.52 (16 H, m, Ar-H), 7.92–7.96 (8 H, m, Ar-H), 10.45 (2 H, s, CHO).

(*R*)-4-(4-Iodophenyl)dinaphtho[2,1-*d*:1′,2′-*f*][1,3,2]dithiazepine 3,3,5,5-tetraoxide ((*R*)-13). Anhydrous pyridine (5 cm^3, de-oxygenated in 3 freeze-pump-thaw cycles) was added to a Schlenk tube containing (*R*)-1,1′-binaphthyl-2,2′-disulfonyl dichloride (100 mg, 0.22 mmol) and 4-iodoaniline (49 mg, 0.22 mmol, purified by sublimation) under Ar. The mixture was stirred at r.t. for 8 h. The solution turned brick-red in colour after 10–15 min, and a large amount of precipitates formed during the reaction. The precipitates were removed by filtration. Evaporation of the filtrate and FC (SiO_2; CH_2Cl_2/hexane 2:1) gave (*R*)-**13** (48 mg, 36%) as a white solid. Mp 280–281 °C; $[\alpha]_D^{20}$ +209 (*c* 0.05 in $CHCl_3$); UV/Vis λ_{max}(MeCN)/nm 226 (ε/dm^3 mol^{-1} cm^{-1} 81000), 237 (71900), 344 (7600); CD λ_{max}(MeCN)/nm 223 ($\Delta\varepsilon$/dm^3 mol^{-1} cm^{-1} +146), 240 (–246), 260 (+14), 304 (+23), 338 (+17); $\tilde{\nu}_{max}$(ATR)/cm^{-1} 3080, 2923, 2852, 1480, 1365, 1332, 1179, 822, 749, 715; δ_H (300 MHz; $CDCl_3$) 7.08 (2 H, d, *J* 8.7, NC_6H_4I), 7.35–7.52 (4 H, m, naphthalene), 7.71 (2 H, ddd, *J* 8.3, 6.5, 1.5, naphthalene), 7.79 (2 H, d, *J* 8.7, NC_6H_4I), 8.10 (2 H, d, *J* 8.3, naphthalene), 8.23–8.30 (4 H, m, naphthalene); δ_C (75 MHz; $CDCl_3$) 97.20, 123.00, 128.21, 128.44, 128.78, 129.68, 131.05, 131.72, 131.83, 133.02, 134.57, 135.73, 135.75, 138.91; HR-ESI-MS *m/z* 597.9638 (100%, $[M+H]^+$, calcd for $C_{26}H_{17}INO_4S_2^+$: 597.9638).

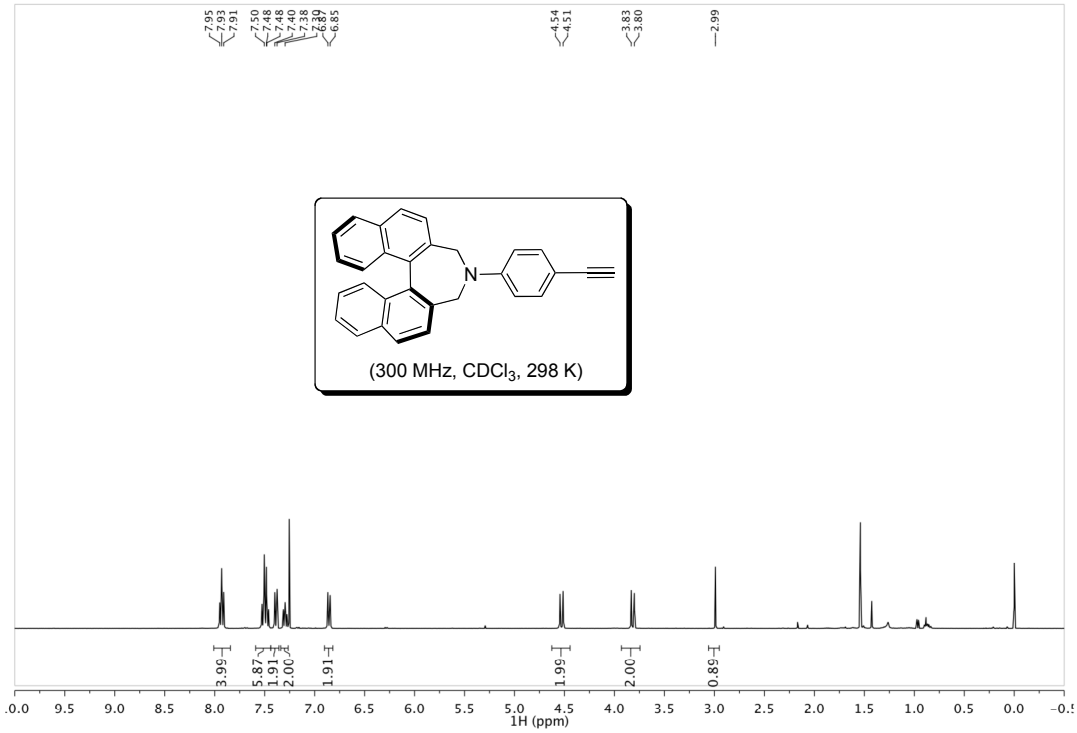
(*R*)-4-{4-[(Trimethylsilyl)ethynyl]phenyl}dinaphtho[2,1-*d*:1′,2′-*f*][1,3,2]dithiazepine 3,3,5,5-tetraoxide ((*R*)-15). A mixture of (*R*)-**13** (274 mg, 0.46 mmol), $[PdCl_2(PPh_3)_2]$

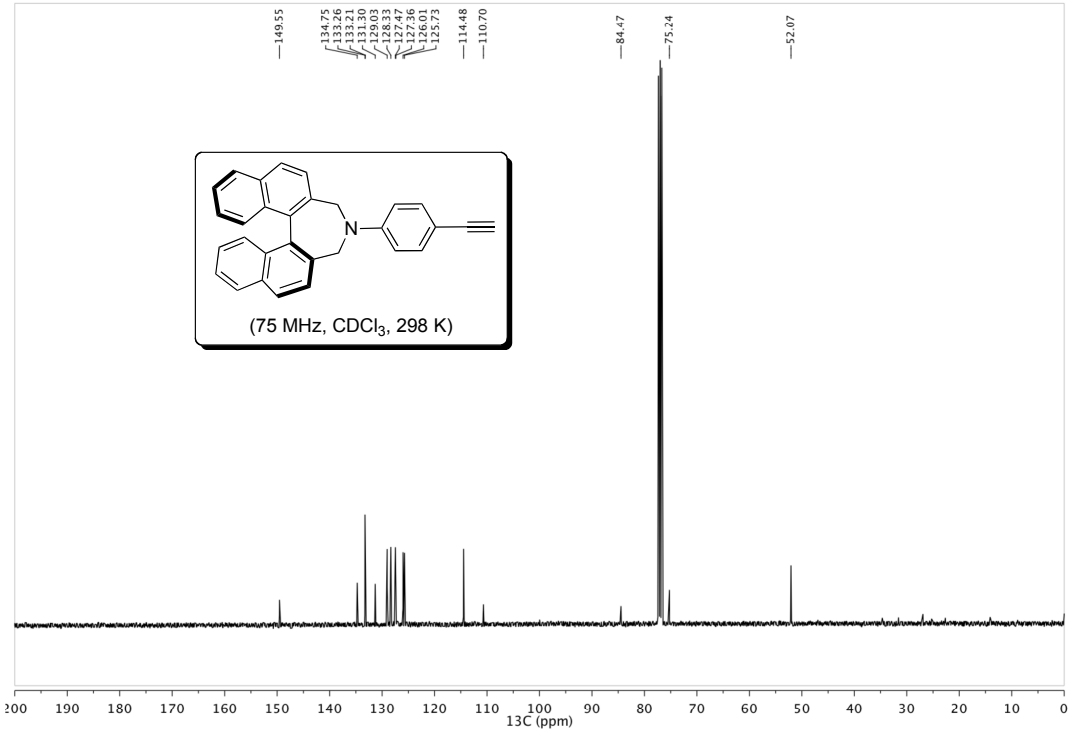
(16 mg, 0.023 mmol) and CuI (7 mg, 0.037 mmol) in THF (20 cm^3) and Et_3N (10 cm^3) was purged with Ar at r.t. for 20 min. Trimethylsilylacetylene (0.3 cm^3, 2.16 mmol) was injected to the above solution, and the mixture was let stand at r.t. for 10 h. After removal of the salts (Et_3NHI) and solvents, the residue was purified by FC (SiO_2; hexane/CH_2Cl_2 1:1) to afford (*R*)-**15** (250 mg, 96%). Mp 125–127 °C; $[\alpha]_D^{20}$ = +269 (*c* 0.02 in $CHCl_3$); UV/Vis λ_{max}(MeCN)/nm 224 (ε/dm^3 mol^{-1} cm^{-1} 69800), 245 (56900), 272 (35300), 344 (6700); CD λ_{max}(MeCN)/nm 222 ($\Delta\varepsilon$/dm^3 mol^{-1} cm^{-1} +125), 240 (–220), 262 (+21), 303 (+23), 339 (+14); $\tilde{\nu}_{max}$(ATR)/cm^{-1} 3058, 2956, 2853, 2159, 1493, 1346, 1180, 858, 841, 816, 746, 703; δ_H (300 MHz; $CDCl_3$) 0.29 (9 H, s, $SiMe_3$), 7.31 (2 H, d, *J* 8.4, NC_6H_4), 7.38–7.48 (4 H, m, naphthalene), 7.55 (2 H, d, *J* 8.4, NC_6H_4), 7.70 (2 H, ddd, *J* 2.0, 6.1, 8.4 Hz, naphthalene), 8.09 (2 H, d, *J* 8.4, naphthalene), 8.22–8.32 (4 H, m, naphthalene); δ_C (75 MHz; $CDCl_3$) –0.01, 96.90, 103.70, 122.97, 125.61, 128.14, 128.29, 128.68, 129.52, 130.90, 131.13, 131.62, 131.70, 132.92, 134.63, 135.60, 135.63; HR-ESI-MS *m/z* 568.1067 (56%, $[M+H]^+$, calcd for $C_{31}H_{26}NO_4S_2Si^+$: 568.1067), 585.1331 (100, $[M+NH_4]^+$, calcd for $C_{31}H_{29}N_2O_4S_2Si^+$: 585.1333).

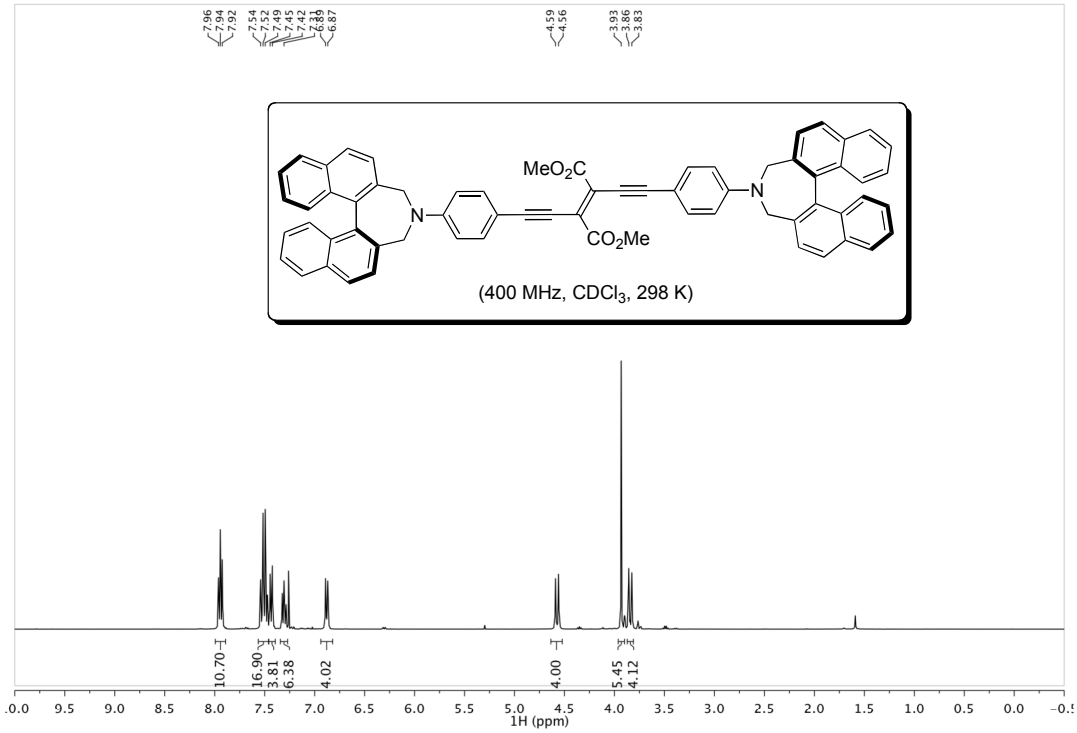
(*R*)-4-(4-Ethynylphenyl)dinaphtho[2,1-*d*:1′,2′-*f*][1,3,2]dithiazepine 3,3,5,5-tetraoxide ((*R*)-14). To the solution of (*R*)-**15** (88 mg, 0.101 mmol) in anhydrous THF (20 cm^3) at 0 °C was added $^nBu_4NF$ on Al_2O_3 (15 wt%, 264 mg, 0.099 mmol) in one portion. The mixture was stirred at 0 °C for 20 min and filtered through a plug (SiO_2; CH_2Cl_2). After removal of solvents, the residue was purified by FC (SiO_2; CH_2Cl_2/hexane 2:1) to give (*R*)-**14** (69 mg, 90%). This compound gradually turned black at *ca*. 80 °C, and the final

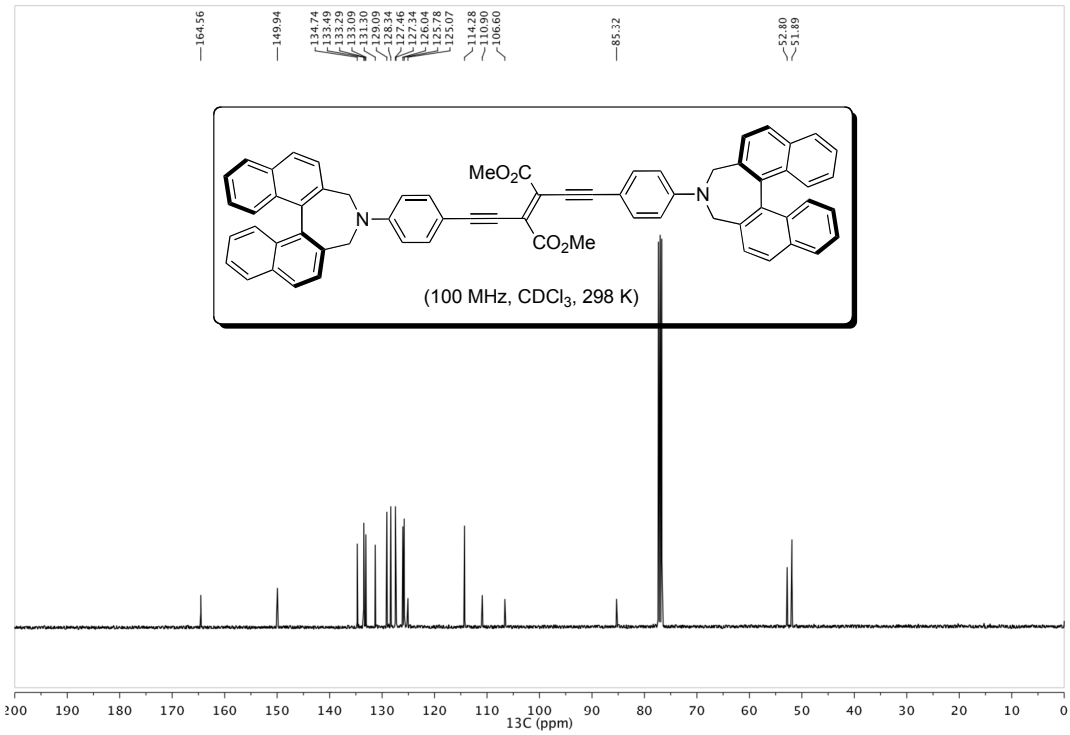
black material did not melt up to 400 °C; $[\alpha]_D^{20}$ = +247 (*c* 0.05 in $CHCl_3$); UV/Vis λ_{max}(MeCN)/nm 224 (ε/dm^3 mol^{-1} cm^{-1} 69700), 245 (63781), 343 (7000); CD λ_{max}(MeCN)/nm 220 ($\Delta\varepsilon$/dm^3 mol^{-1} cm^{-1} +119), 239 (–226), 260 (+22), 303 (+20), 341 (+14); $\tilde{\nu}_{max}$(ATR)/cm^{-1} 3282, 3075, 2924, 2854, 1708, 1583, 1493, 1343, 1180, 919, 896, 651; δ_H (300 MHz; $CDCl_3$) 3.20 (1 H, s, CCH), 7.34 (2 H, d, *J* 8.6, NC_6H_4), 7.39–7.49 (4 H, m, naphthalene), 7.58 (2 H, d, *J* 8.6, NC_6H_4), 7.71 (2 H, ddd, *J* 1.9, 6.1, 8.2, naphthalene), 8.10 (2 H, d, *J* 8.2, naphthalene), 8.18–8.34 (4 H, m, naphthalene); δ_C (75 MHz; $CDCl_3$) 79.50, 82.39, 122.89, 124.54, 128.06, 128.30, 128.66, 129.54, 130.93, 131.21, 131.57, 132.12, 133.13, 134.50, 135.57, 135.61; HR-ESI-MS *m/z* 514.0972 (35%), 513.0938 (100, $[M+NH_4]^+$, calcd for $C_{28}H_{21}N_2O_4S_2^+$: 513.0937).

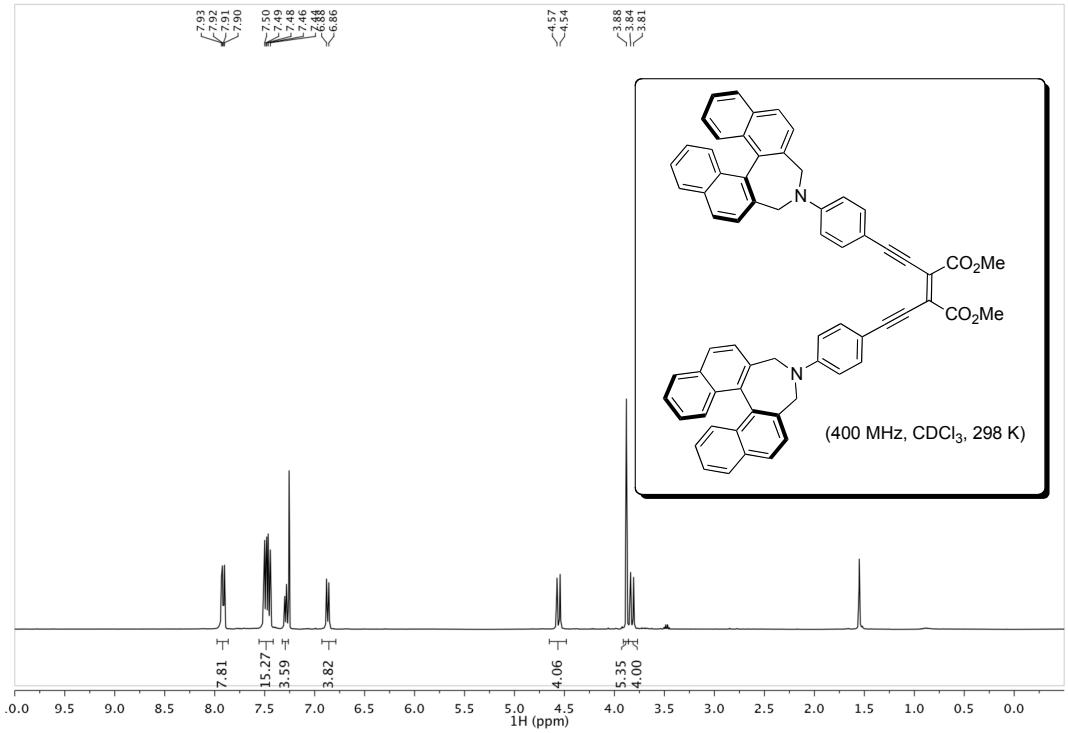
7. ^{1}H and ^{13}C NMR spectra

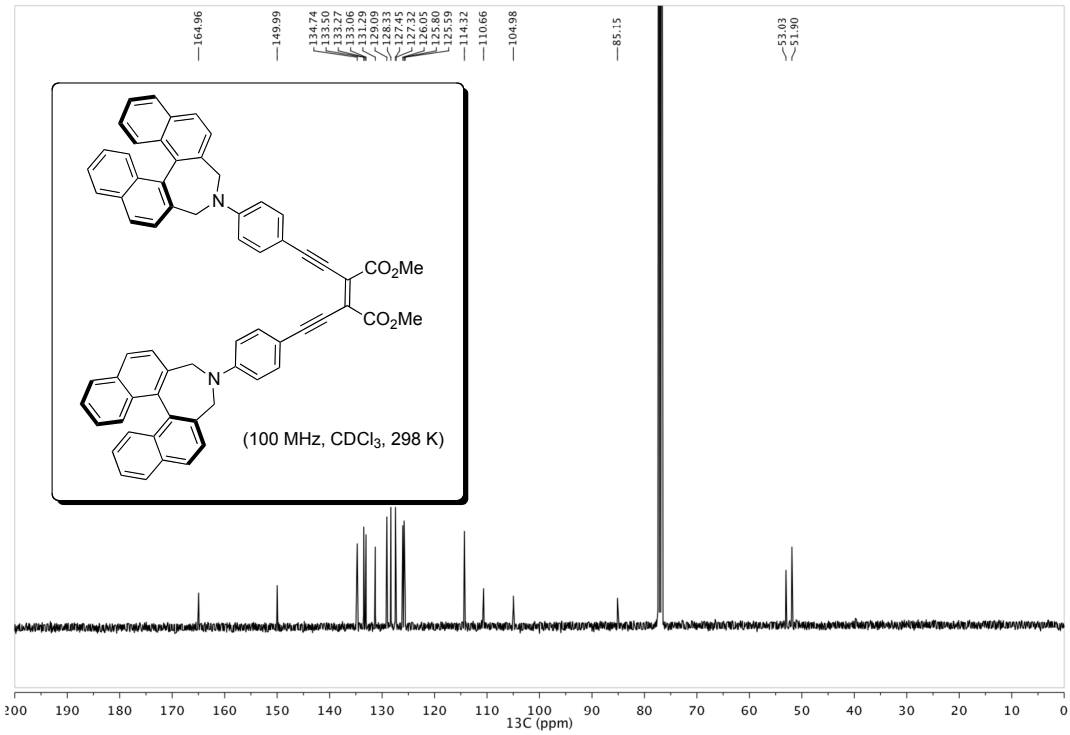


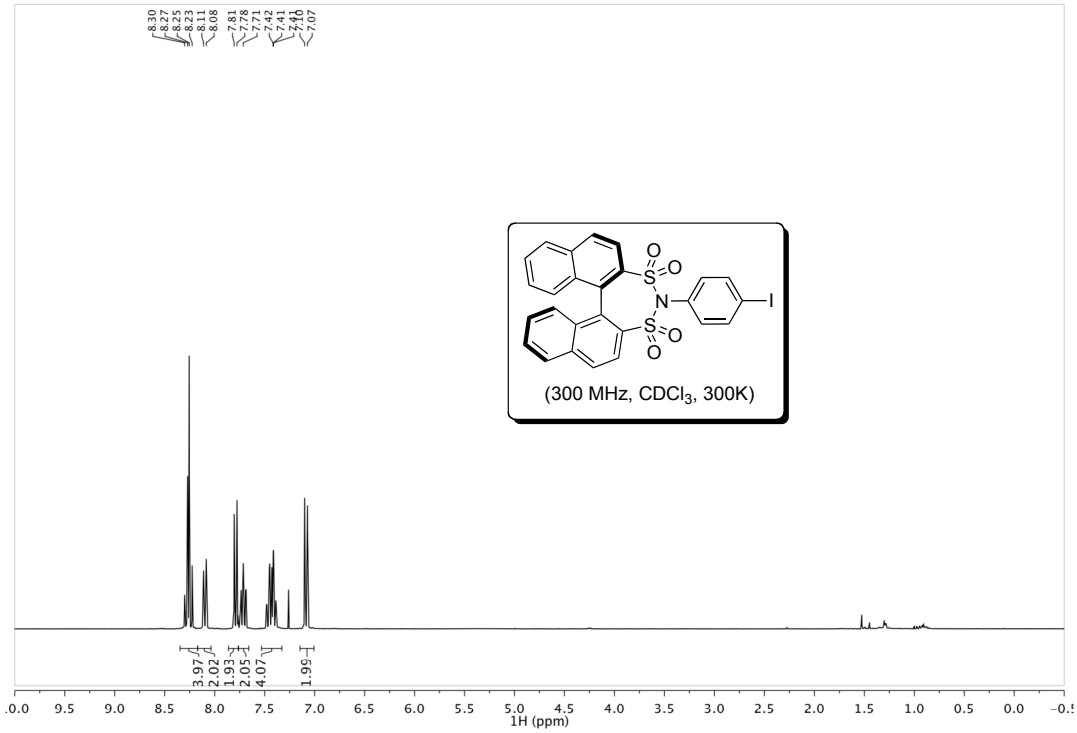



7.96
7.94
7.92
7.54
7.52
7.49
7.45
7.42
7.41
7.39
6.88
6.87
4.59
4.56
3.93
3.86
3.83
MeO2C
CO2Me
N
N
(400 MHz, CDCl3, 298 K)
10.70
16.90
3.81
6.38
4.02
4.00
5.45
4.12
1H (ppm)

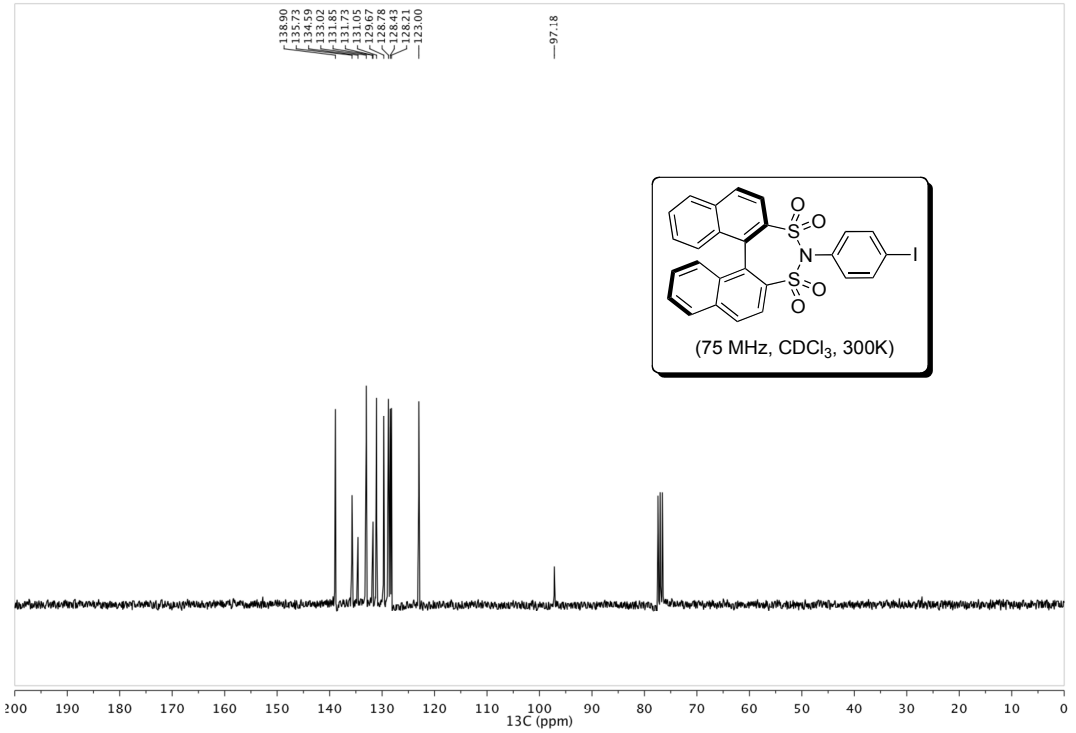

164.56
149.94
134.74
133.49
133.29
133.09
131.30
129.09
128.34
127.46
127.34
126.04
125.78
125.07
114.28
111.00
106.60
85.32
52.80
51.89
MeO2C
CO2Me
N
N
(100 MHz, CDCl3, 298 K)
13C (ppm)


7.93
7.92
7.91
7.90
7.50
7.49
7.48
7.46
7.46
6.88
6.86
4.57
4.54
3.88
3.84
3.81
CO2Me
CO2Me
N
N
(400 MHz, CDCl3, 298 K)
7.81
15.27
3.59
3.82
4.06
5.35
4.00
1H (ppm)

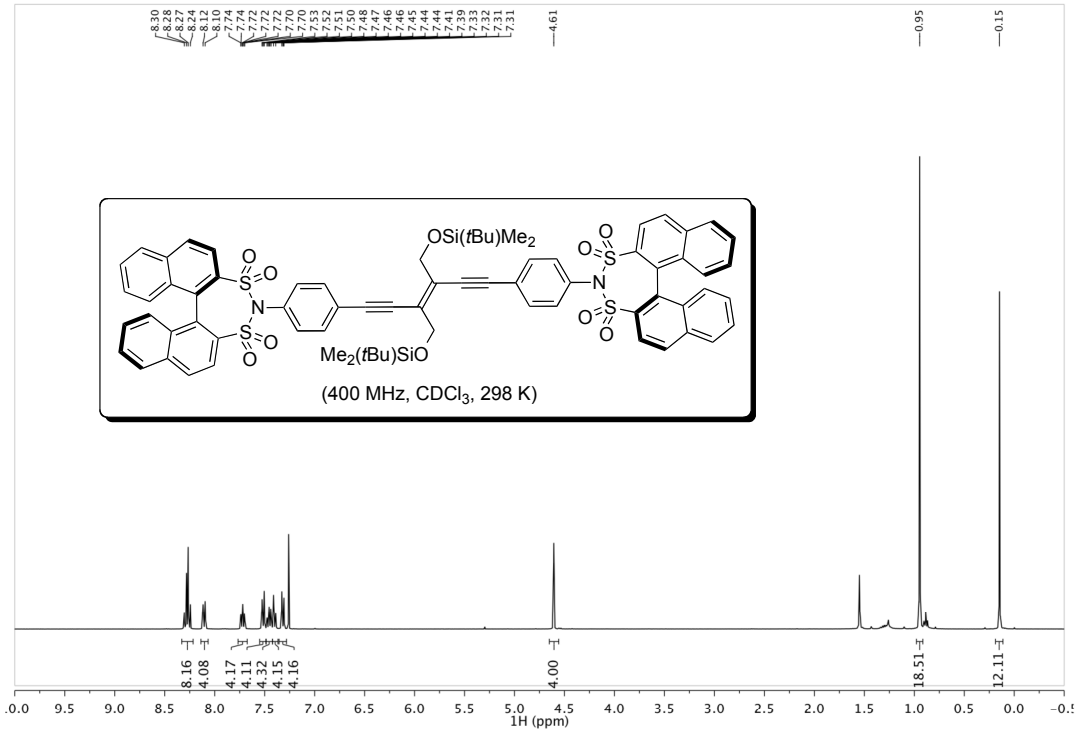

164.96
149.99
134.74
133.50
133.27
133.06
131.29
129.09
128.33
127.45
127.32
126.05
125.80
125.59
114.32
110.66
104.98
85.15
53.03
51.90
CO2Me
CO2Me
N
N
(100 MHz, CDCl3, 298 K)
13C (ppm)

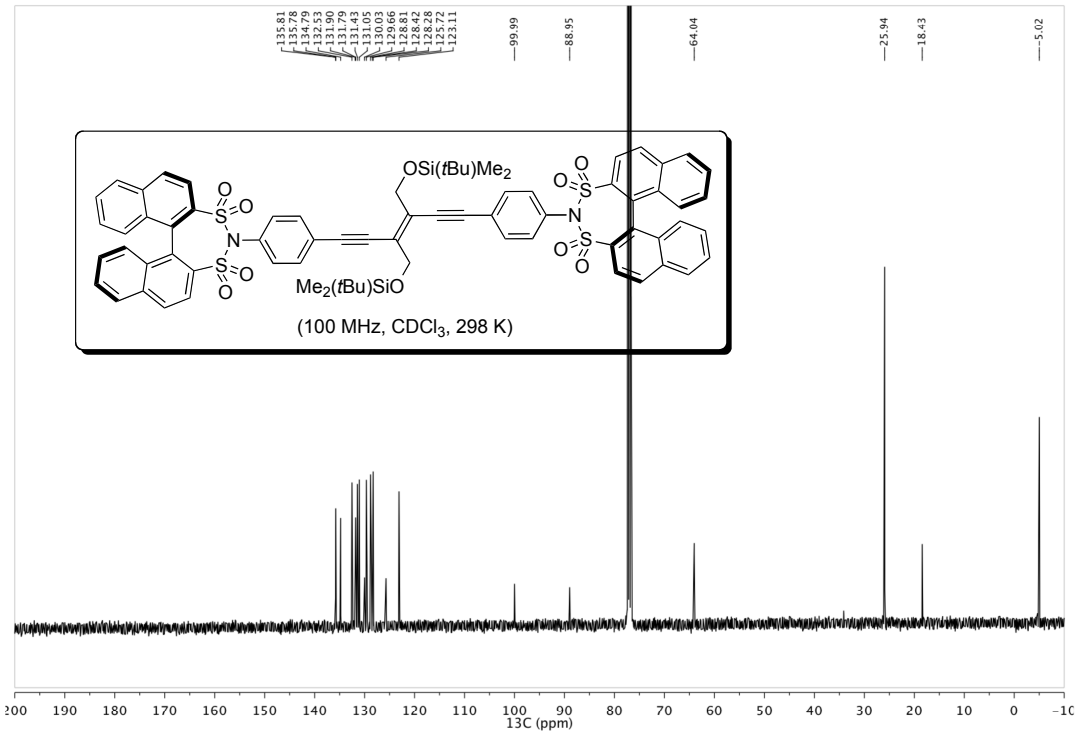


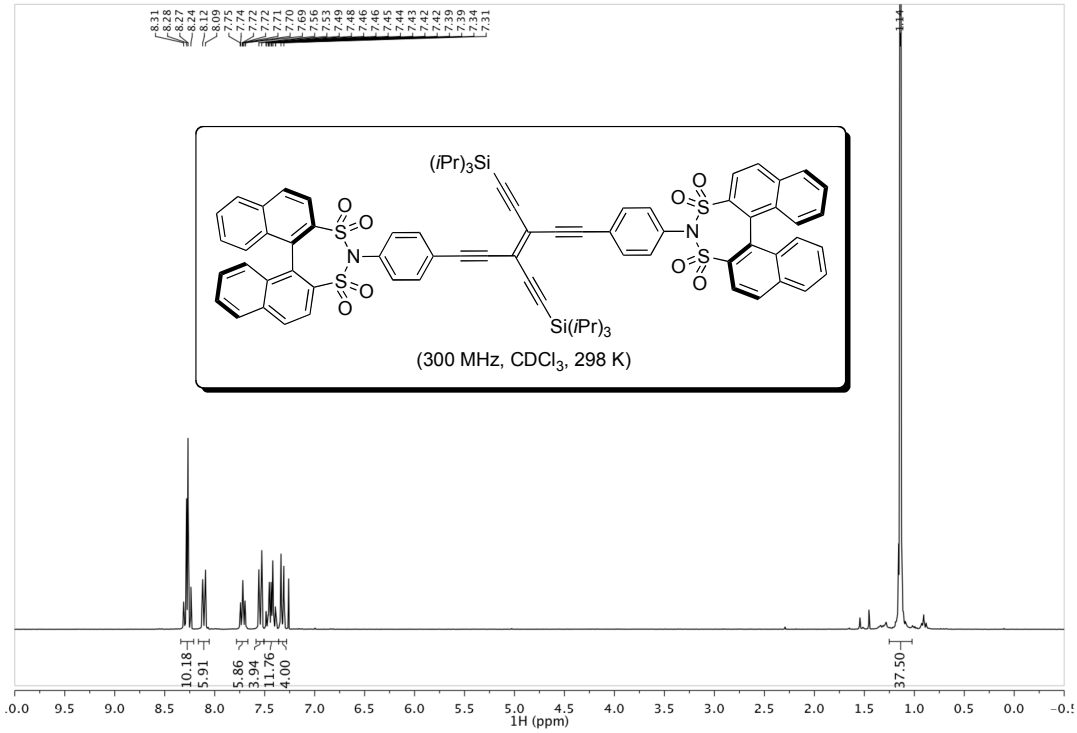
(300 MHz, $CDCl_3$, 300K)

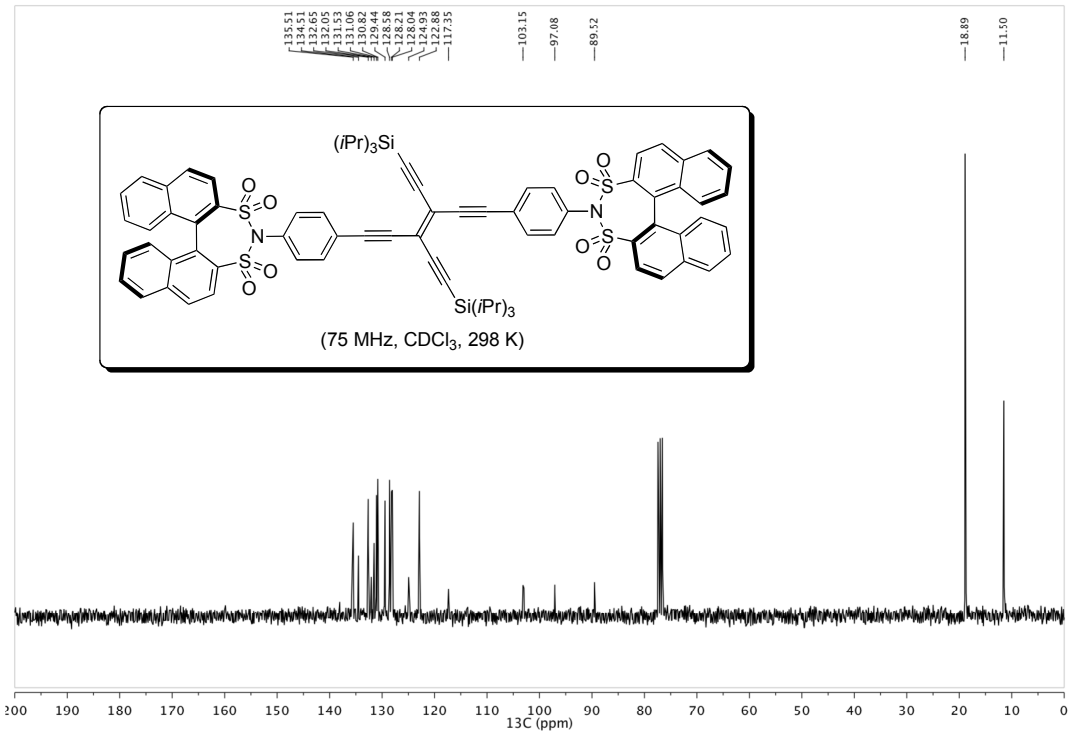


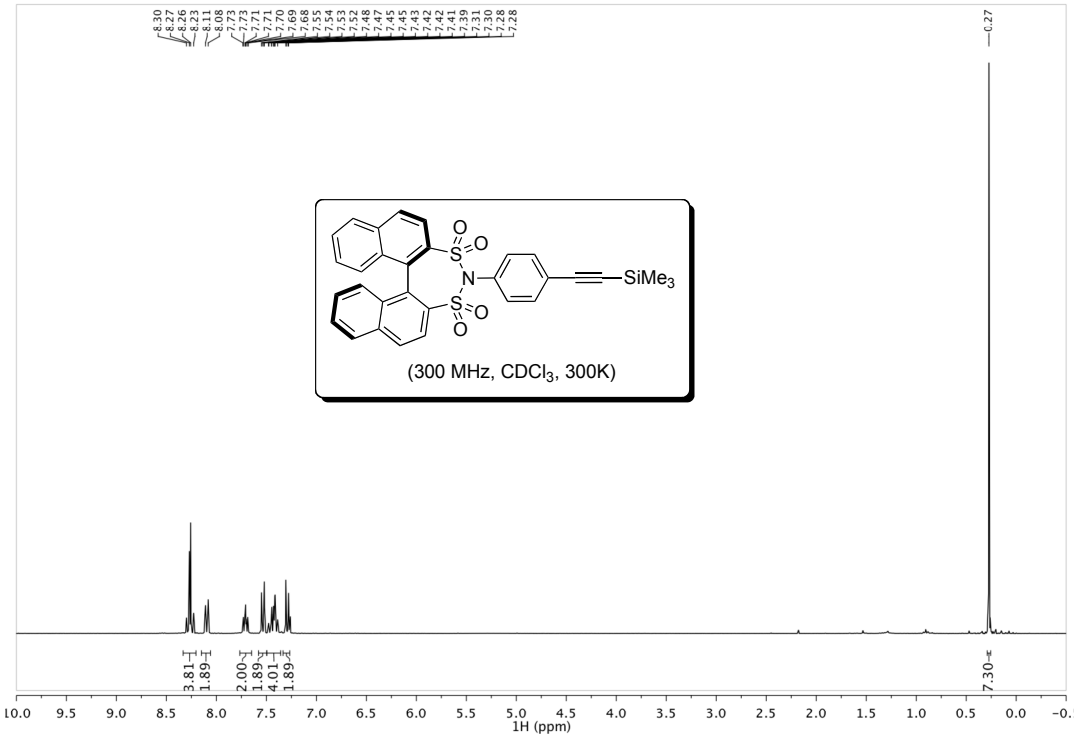
(75 MHz, $CDCl_3$, 300K)

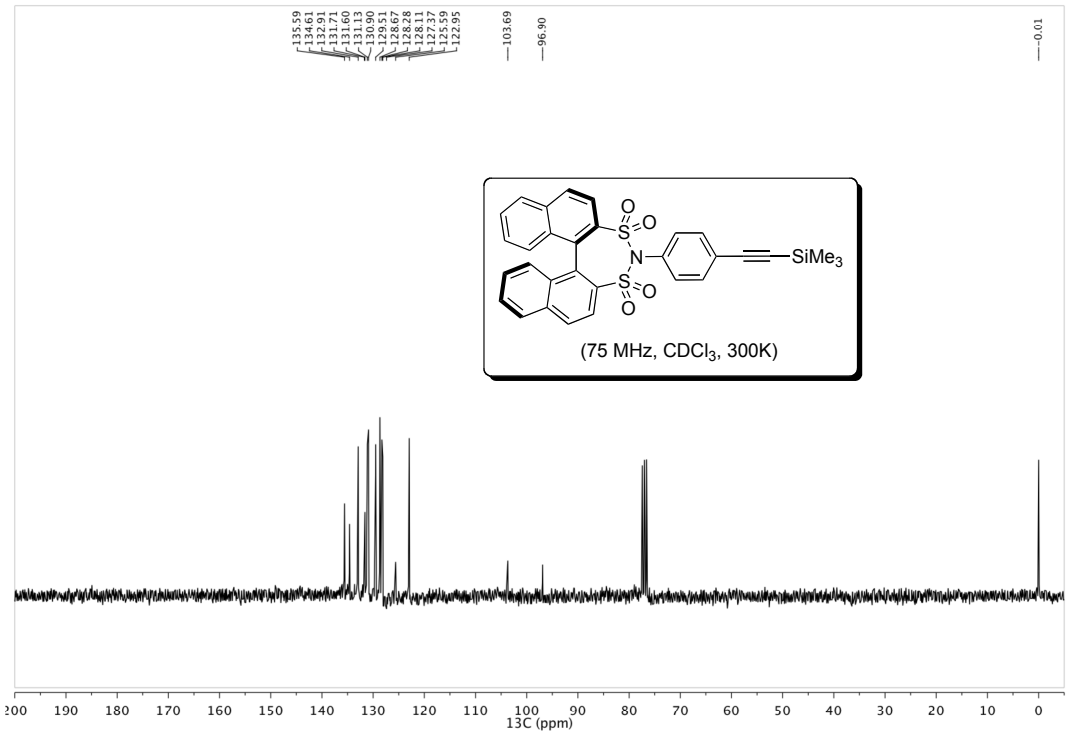

OSi(tBu)Me2
Me2(tBu)SiO
(400 MHz, CDCl3, 298 K)
1H (ppm)

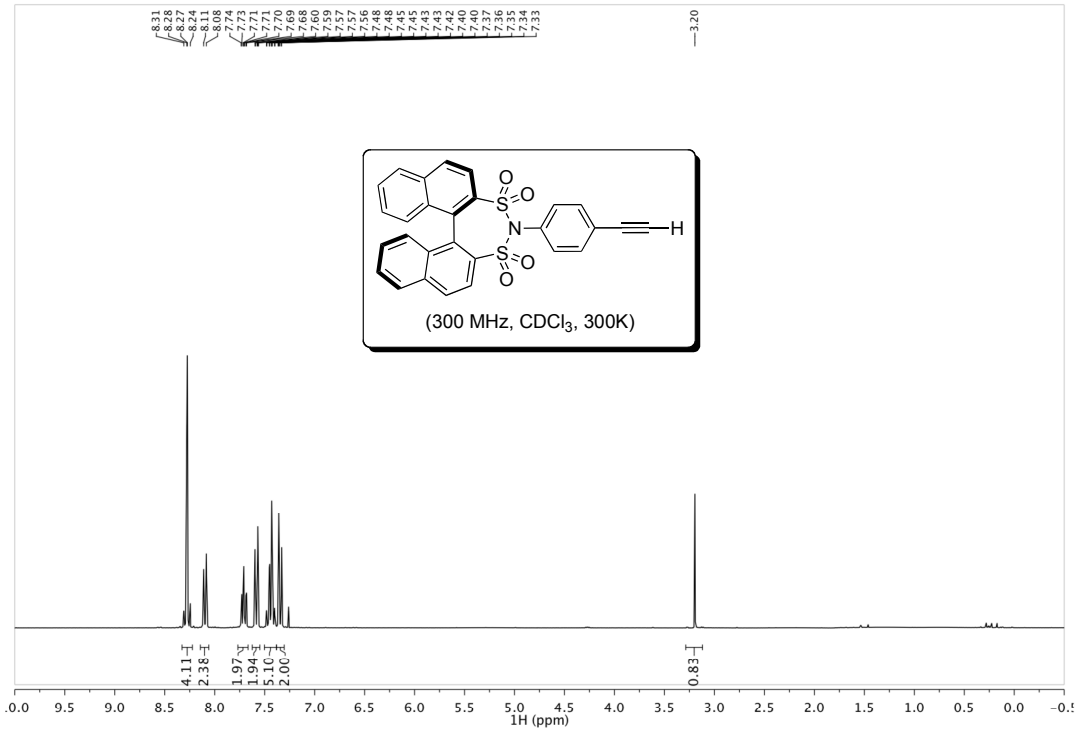

OSi(tBu)Me2
Me2(tBu)SiO
(100 MHz, CDCl3, 298 K)
13C (ppm)

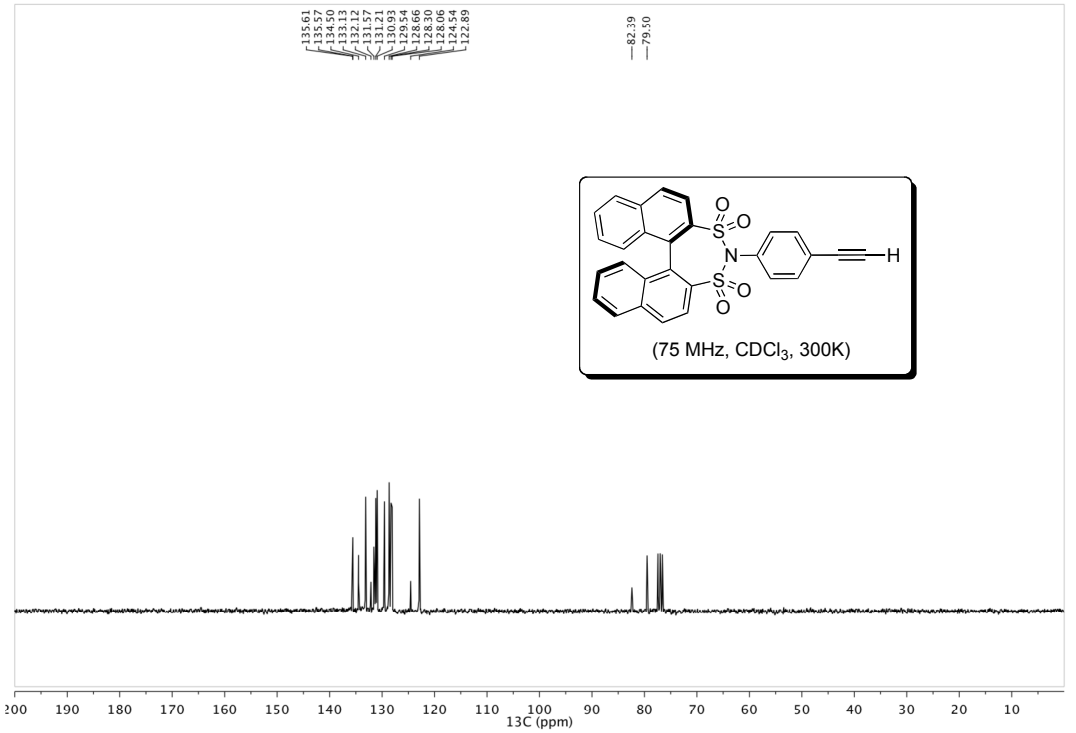

(iPr)3Si
Si(iPr)3
(300 MHz, CDCl3, 298 K)
10.18
5.91
5.86
3.94
11.76
4.00
37.50
1H (ppm)


(iPr)3Si
Si(iPr)3
(75 MHz, CDCl3, 298 K)
13C (ppm)


8.30
8.27
8.26
8.23
8.11
8.08
7.73
7.71
7.70
7.69
7.68
7.55
7.54
7.53
7.52
7.48
7.47
7.45
7.43
7.42
7.41
7.39
7.31
7.30
7.28
7.28
0.27
3.81
1.89
2.00
1.89
4.01
1.89
7.30
SiMe3
(300 MHz, CDCl3, 300K)
1H (ppm)


135.59
134.61
132.91
131.71
131.60
131.13
130.90
129.51
128.67
128.28
128.11
127.37
125.59
122.95
103.69
96.90
0.01
SiMe3
(75 MHz, CDCl3, 300K)
13C (ppm)





8. References

1. R. E. Martin, J. Bartek, F. Diederich, R. R. Tykwinski, E. C. Meister, A. Hilger and H. P. Lüthi, *J. Chem. Soc., Perkin Trans. 2*, 1998, 233–241.

2. R. E. Martin, Doctoral Dissertation No. 12821, ETH Zürich, 1998.

3. M. Montalti, A. Credi, L. Prodi and M. T. Gandolfi, in *Handbook of Photochemistry,* CRC Press, 2006, 3rd. edn., pp. 601–616.