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Electronic Supplementary Information

Synthesis of [7]Phenacene Incorporating Tetradecyl Chains in the Axis Positions and Its Application toward Field-effect Transistor

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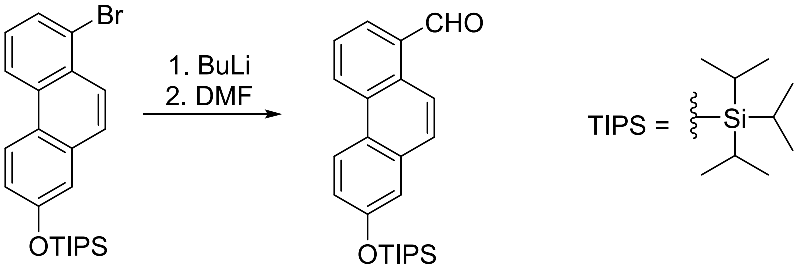
Experimental

Compound characterization

^{1}H and ^{13}C NMR spectra were recorded on VARIAN NMR System 600 (600 MHz) spectrometer. IR spectra were performed on a SHIMADZU IR Prestige-21 spectrometer. Absorption spectra in $CHCl_3$ were obtained on a JASCO V-530 UV-VIS spectrophotometer. Fluorescence spectra were collected in $CHCl_3$ on a JASCO FP-6300 spectrofluorometer at room temperature. Elemental analyses were measured on a PERKIN-ELMER 2400II CHN-S analyzer at the Micro Elemental Analysis Laboratory of Okayama University. High-resolution mass spectra (FAB) were recorded on a JEOL JMS-700 MStation spectrometer at Institute for Materials Chemistry and Engineering, Kyushu University.

Materials

Compound 2



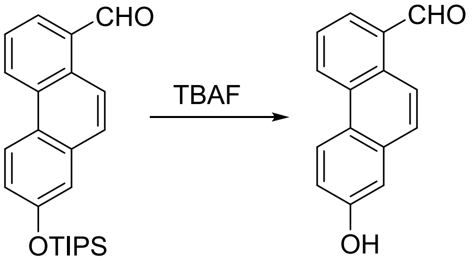
1 **2**

To a solution of compound **1** (859 mg, 2.0 mmol) in THF (15 ml) was dropwise added BuLi (2.6 M in hexanes, 1.5 ml, 4.0 mmo) at −60°C. The mixture was stirred at −70°C ~ −60°C for 3 h, then DMF (774 µl, 10 mmol) was added to the mixture. The resulting mixture was stirred at ca. −60°C for 30 min, and allowed to warm to −10°C. The reaction mixture was stirred at −10°C for 3 h and then quenched with saturated aqueous NH_4Cl (3 ml). THF was removed under reduced pressure. The residue was dissolved in CH_2Cl_2 (20 ml), washed with water, and dried (Na_2SO_4). The solvent was removed and the residue was filtered through a silica-gel pad (CH_2Cl_2). The crude product obtained was chromatographed on silica-gel (hexane/CH_2Cl_2 =

1/0 ~ 1/1 v/v) to afford aldehyde **2** (665 mg, 87%). Analytical sample was prepared by recrystallization from MeCN.

Colorless plates, mp 72–73°C. ^{1}H NMR (600 MHz, $CDCl_3$) δ_H = 10.50 (s, 1H), 9.11 (d, 1H, J = 9.2 Hz), 8.89 (d, 1H, J = 8.2 Hz), 8.57 (d, 1H, J = 9.1 Hz), 8.03 (dd, 1H, J = 7.4, 1.1 Hz), 7.84 (d, 1H, J = 9.2 Hz), 7.78 (dd, 1H, J = 8.2, 7.4 Hz), 7.35 (d, 1H, J = 2.5 Hz), 7.31 (dd, 1H, J = 9.1, 2.5 Hz). ^{13}C NMR (151 MHz, $CDCl_3$) δ_C = 193.9, 155.5, 134.6, 133.5, 131.8, 131.2, 129.9, 129.5, 128.8, 125.8, 124.5, 124.5, 122.7, 121.9, 116.6, 18.1, 12.9. IR ν_{max} 2941, 2864, 2708, 1691, 1458, 1269, 1201, 974, 877, 792, 687, 656 cm^{-1}. Anal. Calcd. for C, 76.14; H, 7.99. Found C, 76.22; H, 8.20.

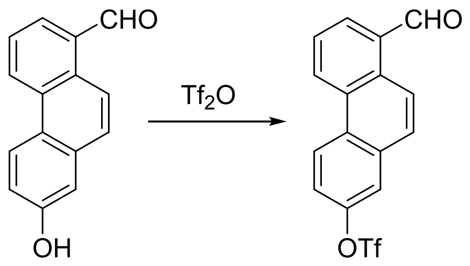
Compound 3



2

To a solution of compound **2** (665 mg, 1.76 mmol) in THF (5 ml) was dropwise added a solution of TFAF (1 M in THF, 2.1 ml, 2.1 mmol) at room temp. The mixture was stirred for 3 h. The reaction mixture obtained was poured into a mixture of water (5 ml) and AcOEt (20 ml). The AcOEt layer was collected, washed with water, dried (Na_2SO_4) and concentrated. The residual yellow solid was collected and washed with toluene to afford the hydroxylaldehyde (350 mg, 89.5%).

Yellow cubic crystals, mp 218–220°C. ^{1}H NMR (600 MHz, DMSO-d_6) δ_H = 10.49 (s, 1H), 10.06 (bs, 1H), 9.07 (d, 1H, J = 8.3 Hz), 9.00 (d, 1H, J = 9.2 Hz), 8.75 (d, 1H, J = 9.0 Hz), 8.16 (d, 1H, J = 7.2 Hz), 7.91 (d, 1H, J = 9.2 Hz), 7.86 (t, 1H, J = 7.9 Hz), 7.29 (d, 1H, J = 2.6 Hz), 7.26 (dd, 1H, J = 9.0, 2.6 Hz). ^{13}C NMR (151 MHz, DMSO-d_6) δ_C = 194.6, 156.9, 133.9, 133.1, 131.3, 130.8, 129.4, 128.9, 128.1, 126.3, 125.2, 122.7, 122.0, 118.3, 111.0. IR ν_{max} 3271, 2747, 1667, 1286 cm^{-1}. Anal. Calcd. for C, 81.07; H, 4.54. Found C, 80.95; H, 4.26.

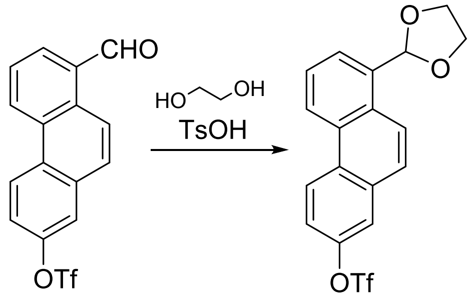


3

To an ice-cooled solution of hydroxyaldehyde (300 mg, 1.35 mmol) in pyridine (5 ml) was dropwise added a solution of Tf_2O (341 μl, 2.03 mmol). The mixture was stirred overnight. During the stirring, the mixture was allowed to warm to room. temp. The resulting dark brown solution was poured into water (5 ml) and extracted with CH_2Cl_2 (20 ml). The extract was washed with water, dried ($MgSO_4$) and concentrated. The residue was chromatographed on silica gel (CH_2Cl_2) to afford compound **3** (409 mg, 85%).

Pale yellow needles, mp 103.5–104°C. ^{1}H NMR (600 MHz, $CDCl_3$) δ_H = 10.50 (s, 1H), 9.30 (d, 1H, J = 9.3 Hz), 8.94 (brd, 1H, J = 8.4 Hz), 8.78 (d, 1H, J = 9.1 Hz), 8.78 (d, 1H, J = 9.1 Hz), 8.15 (dd, 1H, J = 7.1, 1.0 Hz), 7.95 (d, 1H, J = 9.3 Hz), 7.89 (dd, 1H, J = 8.4, 7.1 Hz), 7.85 (d, 1H, J = 2.6 Hz), 7.60 (dd, 1H, J = 9.1, 2.6 Hz). ^{13}C NMR (151 MHz, $CDCl_3$) δ_C =193.5, 148.2, 136.2, 132.8, 131.9, 130.5, 130.3, 129.4, 129.3, 129.1, 126.7, 125.7, 124.6, 122.1* 120.4, 120.1, 120.0*, 111.9*, 115.8*. Asterisked signals are assigned to CF_3 (J_{CF} = 321 Hz). IR ν_{max} 1690, 1420, 1204, 1188, 1134, 1120, 945, 905, 868, 799, 607, 596 cm^{-1}. Anal. Calcd. for C, 54.24; H, 2.56. Found C, 54.14; H, 2.22.

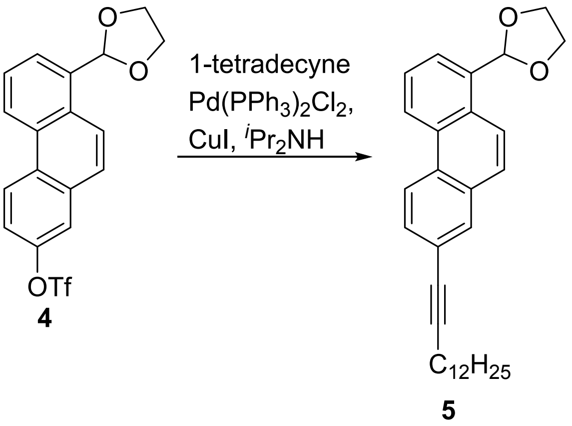
Compound 4



3 **4**

A mixture of **3** (1.06 g, 3.0 mmol), ethylene glycol (2 ml), and TsOH-H_2O (61 mg, 3.2 mmol) in toluene (100 ml) was refluxed for 20 h by using a Dean-Stark apparatus. The resulting mixture was washed with water, $NaHCO_3$ aq, and dried (Na_2SO_4). The solvent was removed and the residue was washed with MeCN to give off-white solid (A). The washings were concentrated and the residue was washed with MeOH to give second crop (B). Washings of B were concentrated and the residue was chromatographed on silica gel (CH_2Cl_2) to obtain third crop (C). The total yield of acetal **4** (A + B+ C) was 1.18 g (98%). Colorless plates, mp 115–115.5°C. ^{1}H NMR (600 MHz, $CDCl_3$) δ_H 8.76 (d, 1H, J = 9.2 Hz), 8.79 (d, 1H, J = 8.4 Hz), 8.27 (d, 1H, J = 9.2 Hz), 7.96 (d, 1H, J = 7.3 Hz), 7.78–7.81 (2H, two doublet signals overlap), 7.71 (dd, 1H, J = 8.3, 7.2 Hz), 7.55 (dd, 1H, J = 8.3, 7.3 Hz), 6.52 (s, 1H), 4.17–4.27 (AA'BB', 4H). ^{13}C NMR (151 MHz, $CDCl_3$) δ_C 147.9, 134.3, 132.5, 130.1, 130.03, 129.97, 126.8, 126.5, 125.7, 125.2, 124.8, 124.1, 122.1*, 120.02*, 119.96, 119.8, 117.9*, 115.8*, 102.1, 65.6. Asterisked signals are assigned to CF_3 (J_{CF} = 320 Hz). IR (neat) ν_{max} 1418, 1203, 795, 598 cm^{-1}. HRMS (FAB) m/z Calcd. for $C_{18}H_{13}F_3O_5S$: 398.0436. Found. 398.0471 (M^+). Anal. Calcd for $C_{18}H_{13}F_3O_5S$: C, 54.27; H, 3.29. Found: C, 54.19; H, 3.14.

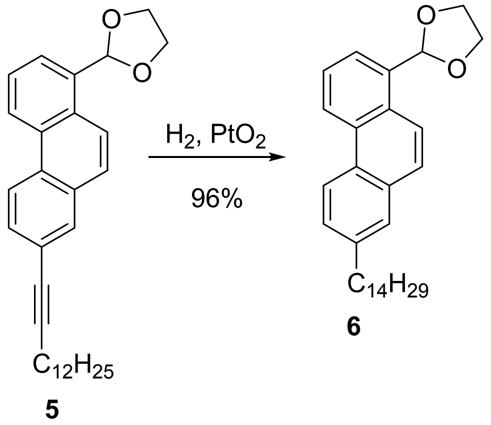
Compound 5



To a solution of compound **4** (796 mg, 2.0 mmol),1-teteradecyne (571 mg, 3.0 mmol), and ^{i}Pr$_2$NH (5 ml) in DMF (7 ml) was added $Pd(PPh_3)_2Cl_2$ (140 mg, 0.2 mmol) and CuI (76 mg, 0.4 mmol). The mixture was stirred at 40ºC under Ar atmosphere for 14 h. The reaction was quenched with water (2 mL) and 1 M HCl (2 ml). The resulting mixture was diluted with toluene (50 ml), washed twice with water, and dried (Na_2SO_4). The solvent was removed and the residue was repeatedly chromatographed on silica gel (hexane~toluene). The crude product was purified by alumina column chromatography (hexane : toluene = 1 : 1) to afford acetylene **5**

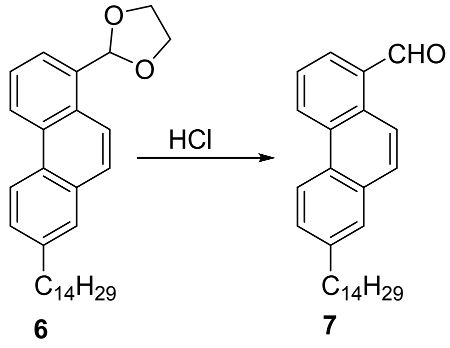
(834 mg, 94%). Off-white crystals, mp 57–57.5℃. ^{1}H NMR (600 MHz, $CDCl_3$) δ_H 8.69 (d, 1H, J = 8.4 Hz), 8.60 (d, 1H, J = 8.6 Hz), 8.15 (d, 1H, J = 9.2 Hz), 7.96 (d, 1H, J = 1.4 Hz), 7.89 (d, 1H,, J = 7.9 Hz), 7.71 (d, 1H, J = 9.2 Hz), 7.63–7.68 (2H, two signals overlap), 6.52 (s, 1H), 4.20 (AA'BB', 4H), 2.48 (t, 2H, J = 7.2 Hz), 1.66 (quint, 2H, J = 7.2 Hz), 1.49 (quint, 2H, J = 7.2 Hz), 1.22–1.40 (m, 16H), 0.89 (t, 3H, J = 7.2 Hz). ^{13}C NMR (151 MHz, $CDCl_3$) δ_C 133.9, 131.6, 131.4, 130.6, 129.9, 129.8, 129.6, 127.1, 126.1, 124.4, 124.1, 123.04, 123.0, 122.4, 102.3, 91.6, 80.7, 65.5, 32.1, 29.84, 29.81 (two signals overlap), 29.7, 29.5, 29.3, 29.1, 28.9, 22.8, 19.7, 14.3. IR (neat) ν_{max} 2919, 2851, 2223 cm^{-1}. Anal. Calcd for $C_{31}H_{38}O_2$: C, 84.12; H, 8.65. Found: C, 84.07; H, 8.58.

Compound 6



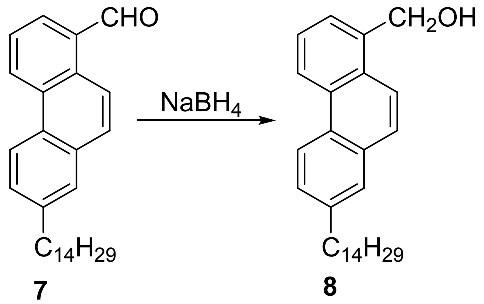
A mixture of compound **5** (813 mg, 1.84 mmol) and PtO_2 (21 mg, 0.09 mmol) in AcOEt (50 ml) was stirred under H_2 at r.t. for 18 h. The solvent was removed and the residue was chromatographed on silica gel (hexane : toluene 1 : 1) to afford compound **6** (790 mg, 96%). Colorless plates, mp 74.5–75℃. ^{1}H NMR (600 MHz, $CDCl_3$) δ_H 8.71 (d, 1H, J = 8.3 Hz), 8.60 (d, 1H, J = 8.6 Hz), 8.10 (d, 1H, J = 9.1 Hz), 7.87 (d, 1H, J = 7.2 Hz), 7.77 (d, 1H, J = 9.1 Hz), 7.68 (d, 1H, J = 1.6 Hz), 7.64 (dd, 1H, J = 8.2, 7.4 Hz), 7.50 (dd, 1H, J = 8.6, 1.6 Hz), 6.54 (s, 1H), 4.21 (AA'BB', 4H), 2.82 (t, 2H, J = 7.7 Hz), 1.74 (quin, 2H, J = 7.7 Hz), 1.201.45 (m, 22H), 0.88 (t, 3H, J = 7.1 Hz). ^{13}C NMR (151 MHz, $CDCl_3$) δ_C 141.6, 133.7, 131.8, 130.9, 129.5, 128.6, 128.0, 127.6, 127.5, 125.9, 123.9, 123.8, 122.9, 122.4, 102.3, 65.5, 36.0, 32.1, 31.6, 29.85, 29.83, 29.81, 29.75, 29.7, 29.54, 29.52, 22.8, 14.3. IR (neat) ν_{max} 2953, 2918, 2849, 1470, 1103, 800, 766 cm^{-1}. Anal. Calcd for $C_{31}H_{42}O_2$: C, 83.36; H, 9.48. Found: C, 83.37; H, 9.64.

Compound 7



To a solution of acetal **6** (764 mg, 1.71 mmol) in THF (20 ml) was added 1 M HCl (4 ml) and the mixture was stirred at r.t. for 19 h. The reaction was quenched with sat. $NaHCO_3$ (2 ml) and the solvent was removed under reduced pressure. The residue was dissolved in toluene (50 ml) and washed successively with brine, $NaHCO_3$ aq., water, and dried (Na_2SO_4). Toluene was removed under reduced pressure and the residue was chromatographed on silica gel (toluene) to afford aldehyde **7** (685 mg, 99%). Colorless crystals, mp 82.5–83°C. ^{1}H NMR (600 MHz, $CDCl_3$) δ_H 10.52 (s, 1H), 9.13 (d, 1H, J = 9.2 Hz), 8.96 (d, 2H, J = 8.9 Hz), 8.61 (d, 1H, J = 8.6 Hz), 8.07 (dd, 1H, J = 8.1, 1.1 Hz) 7.92 (d, 1H, J = 9.2 Hz), 7.79 (dd, 1H, J = 8.3, 7.2 Hz), 7.73 (d, 1H, J = 1.1 Hz), 7.55 (dd, 1H, J = 8.4, 1.7 Hz), 2.84 (t, 2H, J = 7.2Hz), 1.74 (quin, 2H, J = 7.2Hz), 1.20–1.45 (m, 22H), 0.88 (t, 3H, J = 7.0 Hz). ^{13}C NMR (151 MHz, $CDCl_3$) δ_C 193.9, 142.4, 135.0, 132.1, 131.7, 131.1, 130.3, 130.2, 129.1, 128.5, 128.0, 127.7, 125.7, 122.9, 122.3, 36.0, 32.1, 31.6, 29.84, 29.83, 29.82, 29.81, 29.74, 29.68, 29.5, 22.8, 14.3. IR (neat) ν_{max} 2913, 2846, 2731, 1683, 1468, 1185, 882, 799, 759, 715, 657 cm^{-1}. HRMS (FAB) Calcd for $C_{29}H_{38}O$: 402.2923. Found: m/z 402.2925.

Compound 8

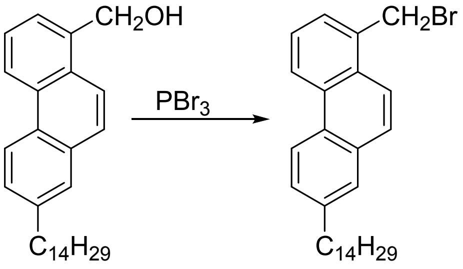


To an ice cooled solution of aldehyde **7** (433 mg, 1.08 mmol) in EtOH (20 ml) and THF (20 ml) was added $NaBH_4$ (38 mg, 2.2 mmol) in one portion. White precipitate appeared soon after the addition. The mixture was stirred at 0°C ~ r.t. for 20 h. The reaction was quenched with saturated aqueous NH_4Cl and concentrated under reduced pressure. The residue was extracted with $CHCl_3$ (50 ml). The extract was washed twice with brine, dried and $CHCl_3$ was removed under reduced pressure to afford alcohol **8** (435 mg, 99%).

Colorless plates, mp 142–143.5°C.

^{1}H NMR (600 MHz, $CDCl_3$) δ_H 8.67 (m, 1H), 8.61 (d, 1H, *J* = 8.4 Hz), 8.05 (d, 1H, *J* = 9.1 Hz), 7.78 (d, 1H, *J* = 7.8 Hz), 7.69 (d, 1H, *J* = 1.2 Hz), 7.60–7.63 (m, 2H), 7.51 (dd, 1H, *J* = 8.4, 1.8 Hz), 5.20 (d, 2H, *J* = 5.1 Hz), 2.82 (t, 2H, *J* = 7.8 Hz), 1.78 (t, 2H, *J* = 5.1 Hz), 1.74 (quin, 2H, *J* = 7.6 Hz), 1.20–1.43 (m, 22H), 0.88 (t, 3H, *J* = 7.1 Hz). ^{13}C NMR (151 MHz, $CDCl_3$) δ_C 141.78, 136.9, 131.9, 130.9, 129.7, 128.7, 128.0, 127.6, 127.5, 126.2, 126.0, 123.0, 122.9, 122.2, 64.2, 36.0, 32.1, 31.6, 29.85, 29.83, 29.81, 29.75, 29.69, 29.53, 29.52, 22.8, 14.3. HRMS (FAB) Calcd. for $C_{29}H_{40}O$: 404.3079. Found *m*/*z* 404.3078.

Compound 9

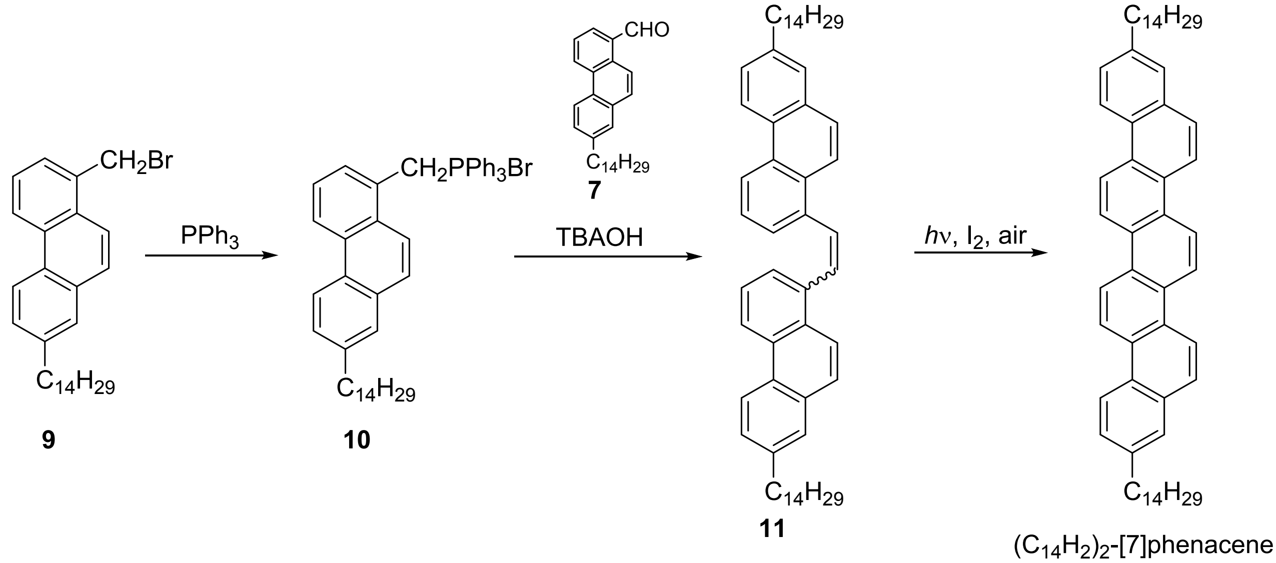


8 **9**

To an ice-cooled solution of alcohol **8** (313 mg, 0.77 mmol) in dioxane (20 ml) was dropwise added a solution of PBr_3 (314 mg, 1.16 mmol) in dioxane (5 ml). After the addition, the reaction mixture was stirred at r.t. for 16 h. The reaction was quenched with sat. aqueous $NaHCO_3$ (5 ml) and the water (20 ml) was added. The precipitate formed was collected, washed with water, and dried under reduced pressure. The crude material was chromatographed on silica gel ($CHCl_3$) to afford bromide **9** (175 mg, 52%).

Colorless crystals, mp 94–94.5°C. ^{1}H NMR (600 MHz, $CDCl_3$) δ_H 8.69 (d, 1H, *J* = 8.4 Hz), 8.60 (d, 1H, *J* = 8.6 Hz), 8.06 (d, 1H, *J* = 9.1 Hz), 7.85 (d, 1H, *J* = 9.1 Hz), 7.70 (d, 1H, *J* = 1.4 Hz), 7.61 (dd, 1H, *J* = 7.1, 1.1 Hz), 7.57 (dd, 1H, *J* = 8.1, 7.2 Hz), 7.52 (dd, 1H, *J* = 8.5 Hz, 1.7 Hz), 5.02 (s, 2H), 2.82 (t, 2H, *J* = 7.6 Hz), 1.74 (quin, 2H, *J* = 7.4 Hz), 1.20–1.42 (m, 22H), 0.88 (t, 3H, *J* = 7.1 Hz). ^{13}C NMR (151 MHz, $CDCl_3$) δ_C 141.9, 133.9, 132.0, 131.3, 129.6, 128.6, 128.2, 128.1, 127.8, 127.7, 126.1, 124.1, 122.9, 122.1, 36.0, 32.4, 32.1, 31.6, 29.82, 29.75, 29.69, 29.52, 29.50, 22.8. HRMS (FAB) Calcd. for $C_{29}H_{39}{}^{79}Br$: 466.2235. Found *m*/*z* 466.2236.

($C_{14}H_{29}$)$_2$-[7]phenacene



A solution of bromide **9** (125 mg, 0.27 mmol) and PPh_3 (173 mg, 0.66 mg) in DMF (10 ml) was refluxed for 5 h. The solvent was removed. The residue and aldehyde **7** (109 mg, 0.27 mmol) were dissolved in CH_2Cl_2 (5 ml). To the resulting solution was dropwise added a 1. M solution of TBAOH in MeOH (0.4 ml, 0.4 mmol) and the mixture was stirred at r.t. for 1 h. The precipitated diarylethene **11** was collected by suction filtration (155 mg, 75%). ^{1}H NMR spectrum of the product suggested that the product was 1:1 mixture of *E* and *Z* isomers (Fig. S11). The obtained diarylethene **11** (115 mg) and I_2 (5.3 mg, 0.02 mmol) was dissolved in toluene (200 ml). the solution was irradiated with 365-nm fluorescent lamps (6 × 15 W) for 1.5 h. Because the starting material precipitated during the photolysis, the starting material was recovered and dissolved in refluxing chlorobenzene (300 ml). I_2 (mg, mmol) was added to the solution, and the resulting solution was irradiated with 365-nm fluorescent lamps (6 × 15 W) for 1.5 h. The precipitate formed was collected to afford ($C_{14}H_{29}$)$_2$-[7]phenacene (145 mg, 68% from **9**).

Off-white plates, mp >300°C. ^{1}HNMR (600 MHz, $CDCl_2CDCl_2$, 80°C) δ_H 9.09 (d, 2H, $J = 9.0$ Hz), 9.04 (s, 2H), 9.00 (d, 2H, $J = 9.0$ Hz), 8.86 (d, 2H, $J = 9.0$ Hz), 8.81 (d, 2H, $J = 8.4$ Hz), 8.06 (d, 2H, $J = 9.0$ Hz), 7.85 (s, 2H), 7.65 (d, 2H, $J = 7.8$ Hz), 2.92 (t, 4H, $J = 7.5$ Hz), 1.86-

1.36 (m, numbers of protons were not determined due to overlapping signal of water.), 0.92 (t, 6H, *J*= 6.9 Hz). ^{13}C NMR spectrum of $(C_{14}H_{29})_2$-[7]phenacene could not be observed due to the low solubility. IR ν_{max} 2915, 2847, 1459, 1277, 876, 803, 405 cm^{-1}. HRMS (MALDI-TOF-MS, DCTB) Calcd. for $C_{58}H_{74}$: 770.5791. Found *m*/*z* 770.5788.

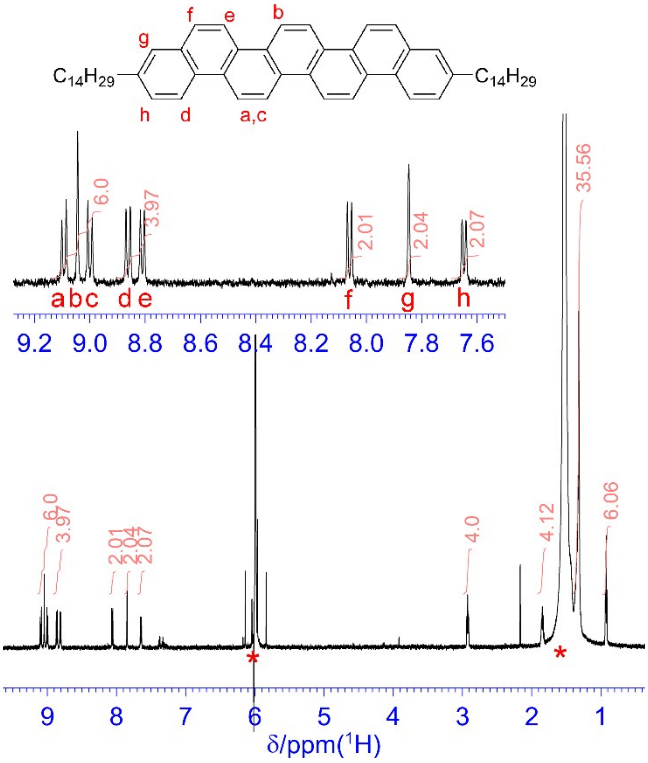


Fig. S1 ^{1}H NMR (600 MHz, $CDCl_2CDCl_2$, 80°C) spectrum of $(C_{14}H_{29})_2$-[7]phenacene. The inset shows the aromatic region. The asterisked signals are due to the solvent and water.

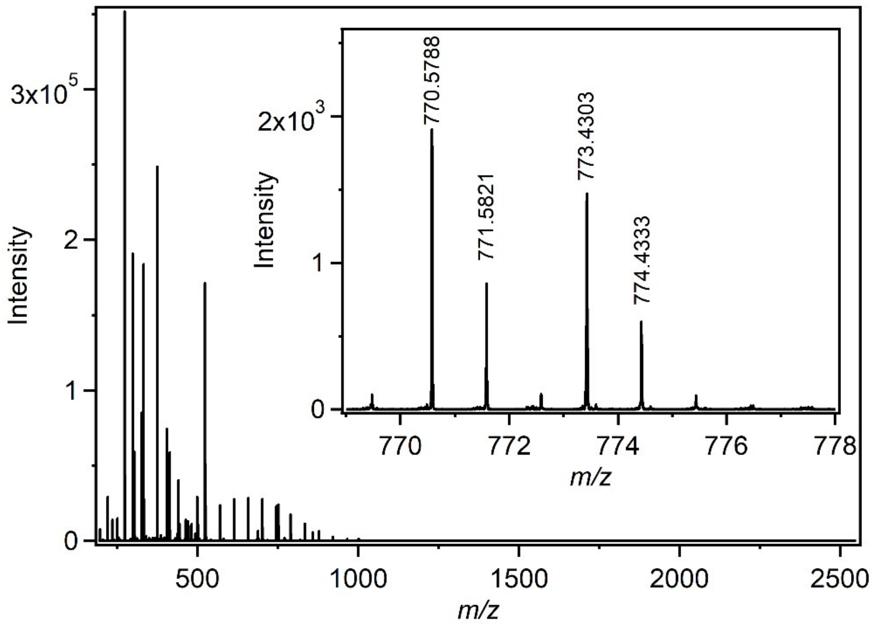


Fig. S2 MALDI-TOF MS spectrum (DCTB) of $(C_{14}H_{29})_2$-[7]phenacene.

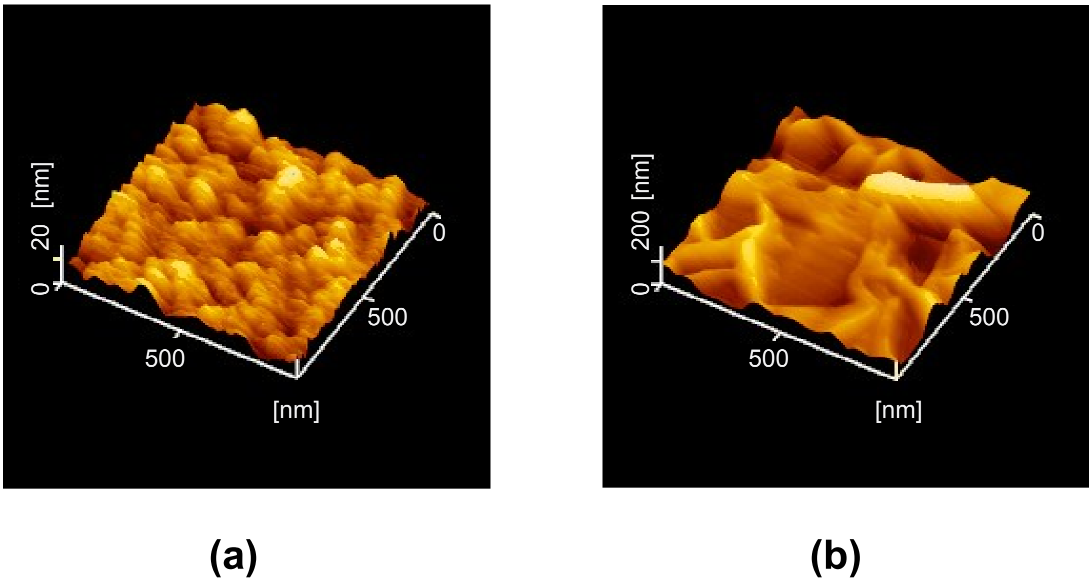


Fig. S3 AFM images of $(C_{14}H_{29})_2$-[7]phenacene thin films on (a) SiO_2 and (b) ZrO_2 surfaces.

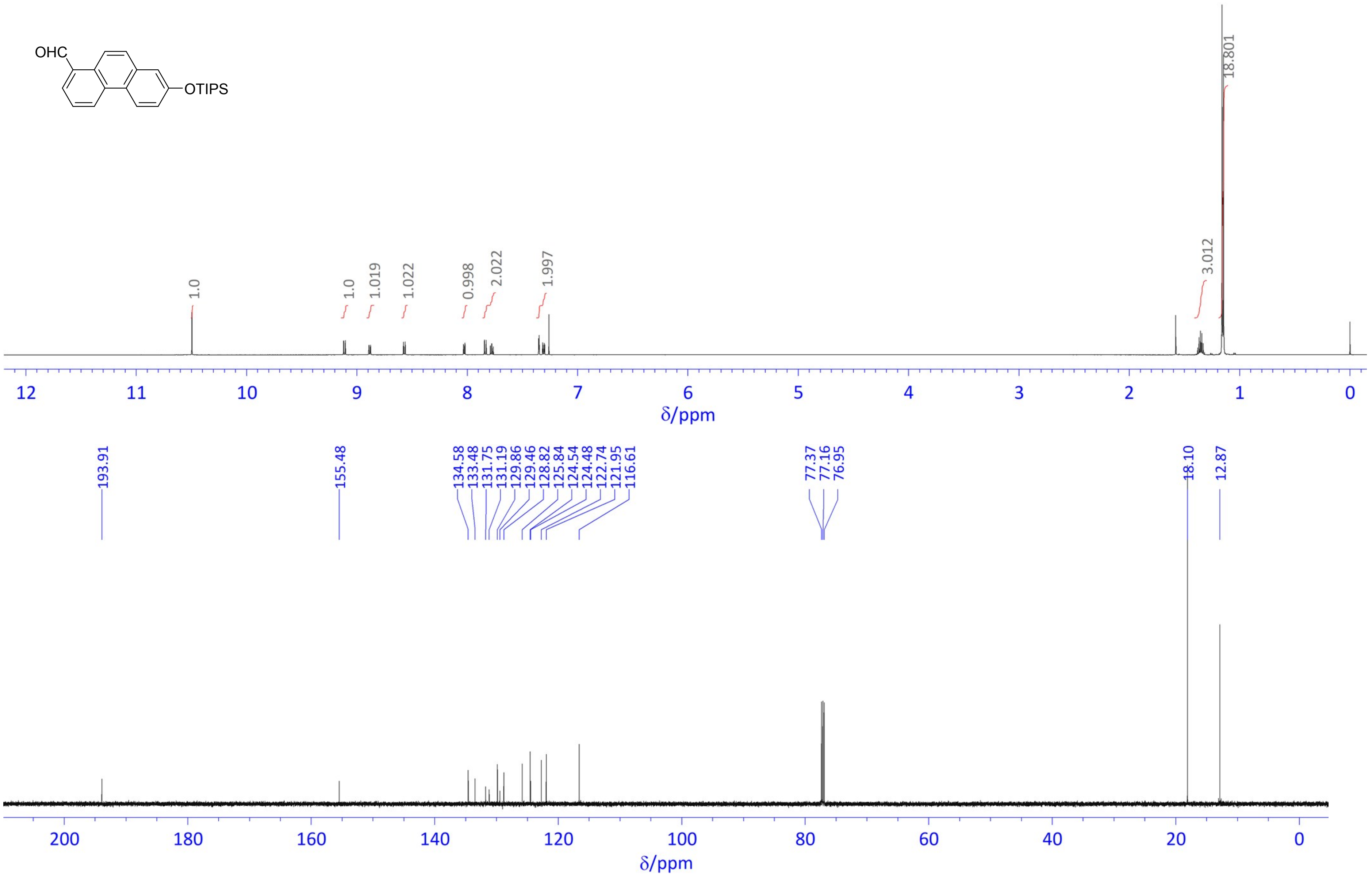


Fig. S4 ^{1}H (600 MHz, upper) and ^{13}C (151 MHz, lower) NMR spectrum of compound **2** ($CDCl_3$).

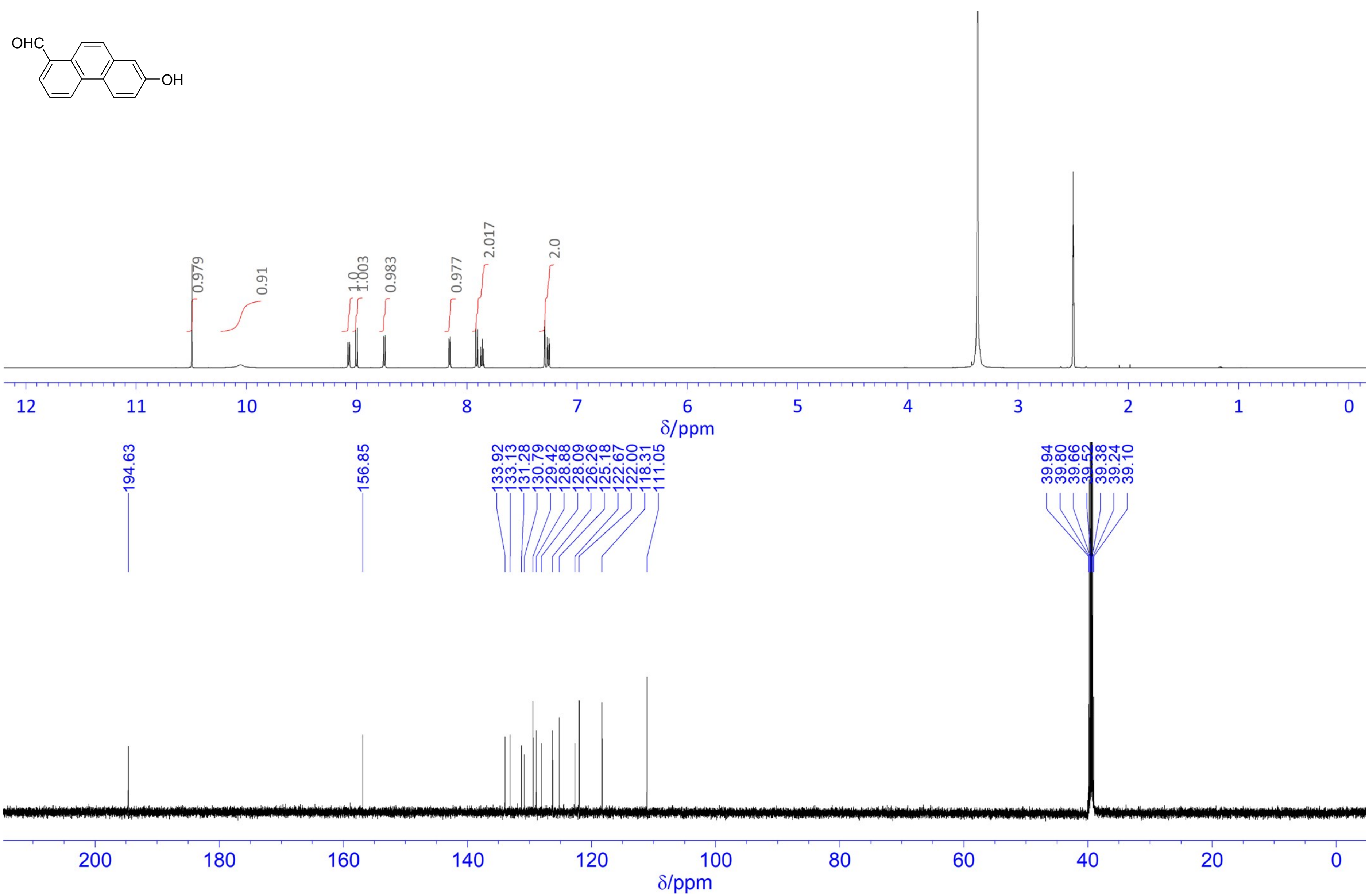


Fig. S5 ^{1}H (600 MHz, upper) and ^{13}C (151 MHz, lower) NMR spectrum of compound 2-hydroxylphenanthrene carbaldehyde (DMSO-d_6).

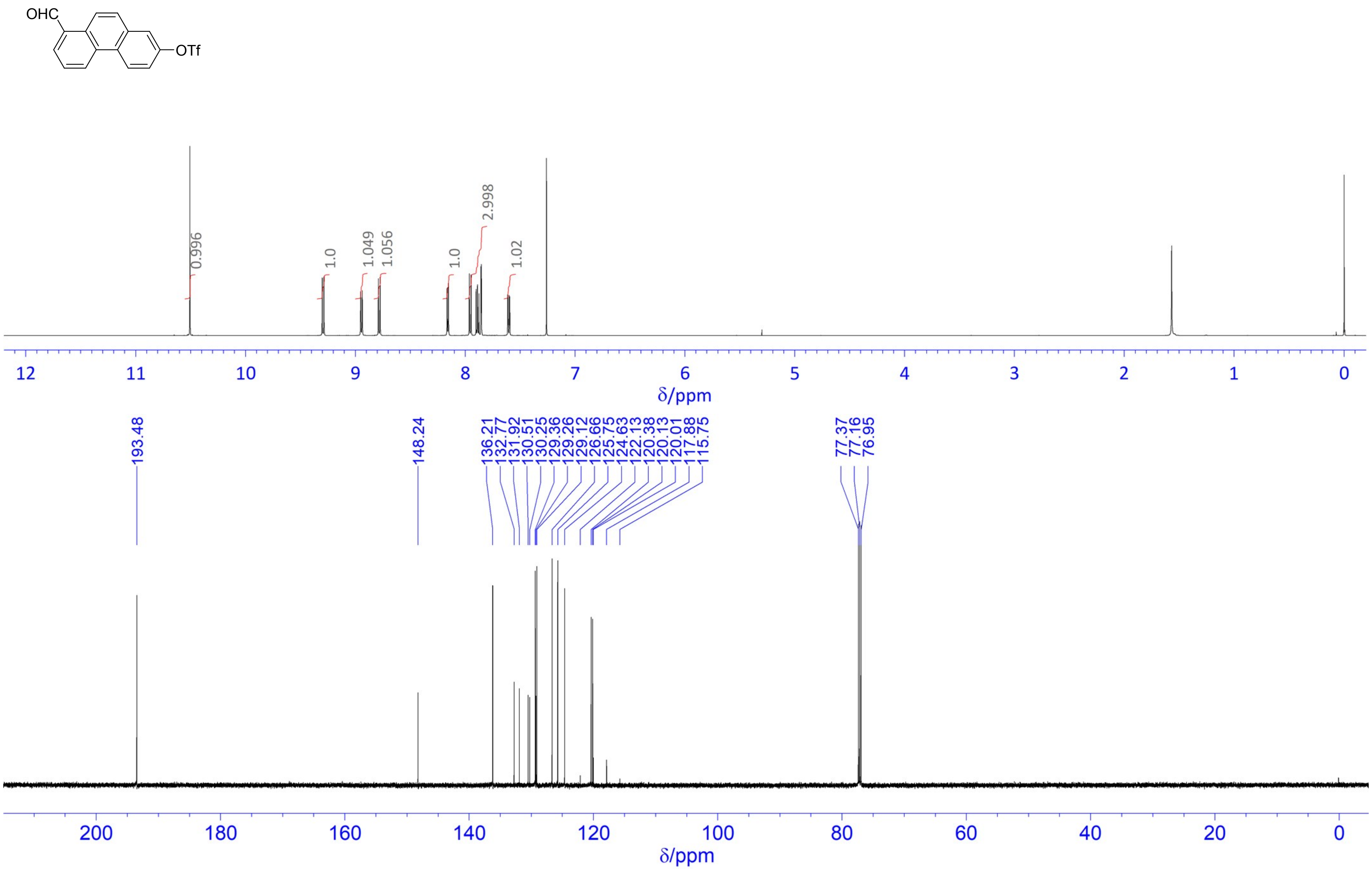


Fig. S6 ^{1}H (600 MHz, upper) and ^{13}C (151 MHz, lower) NMR spectrum of compound **3** ($CDCl_3$).

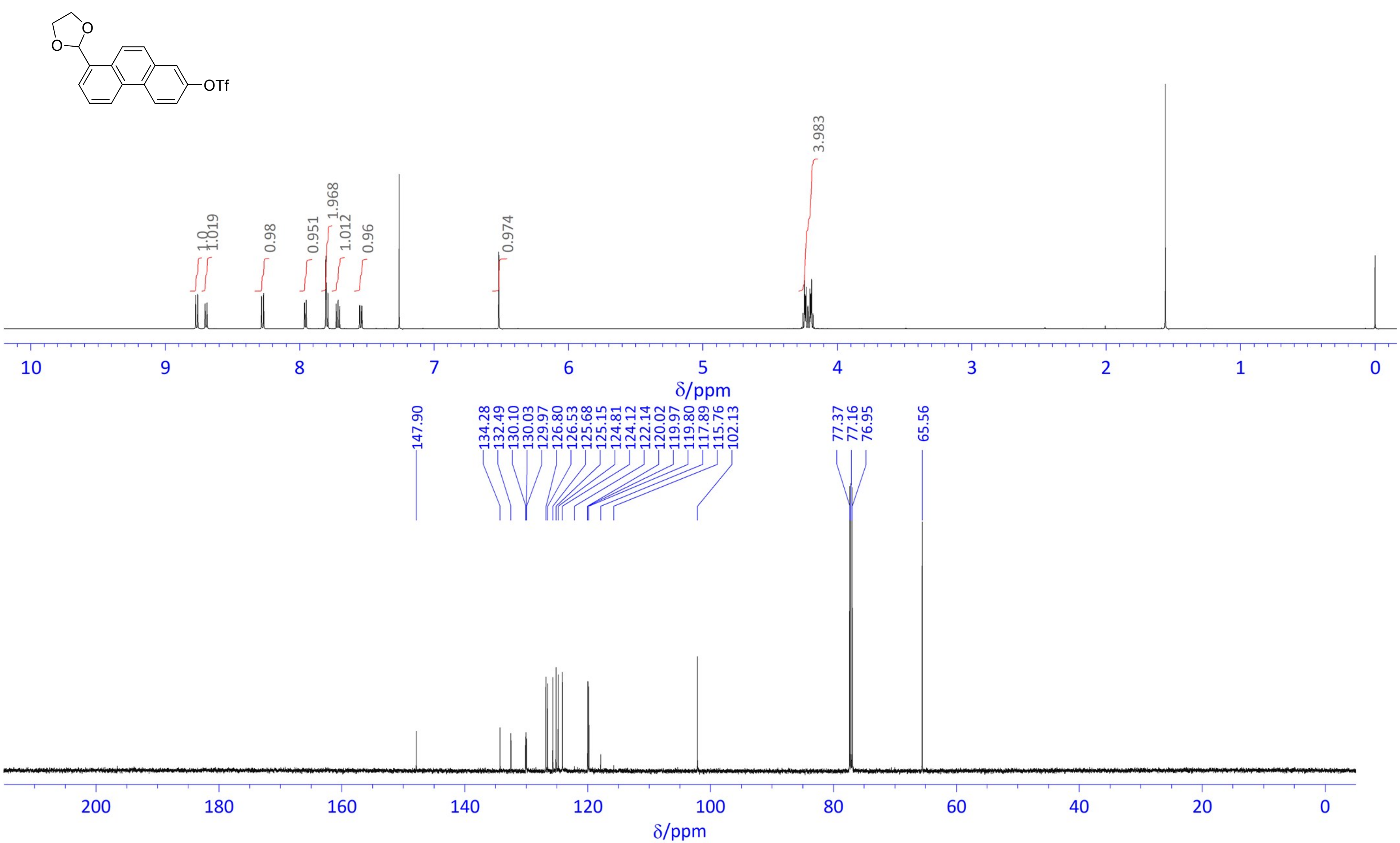


Fig. S7 ^{1}H (600 MHz, upper) and ^{13}C (151 MHz, lower) NMR spectrum of compound **4** ($CDCl_3$)

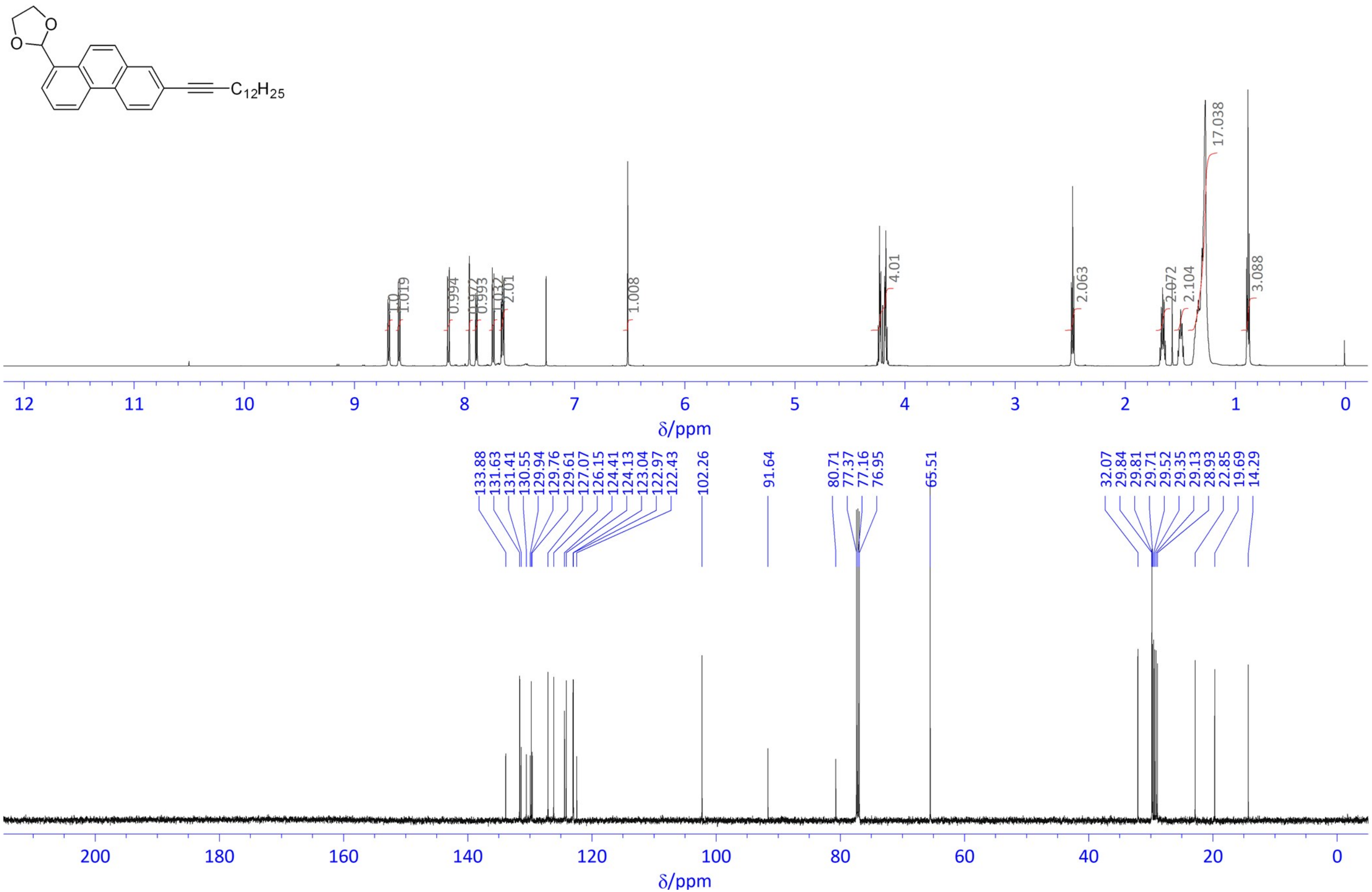


Fig. S8 ^{1}H (600 MHz, upper) and ^{13}C (151 MHz, lower) NMR spectrum of compound **5** ($CDCl_3$)

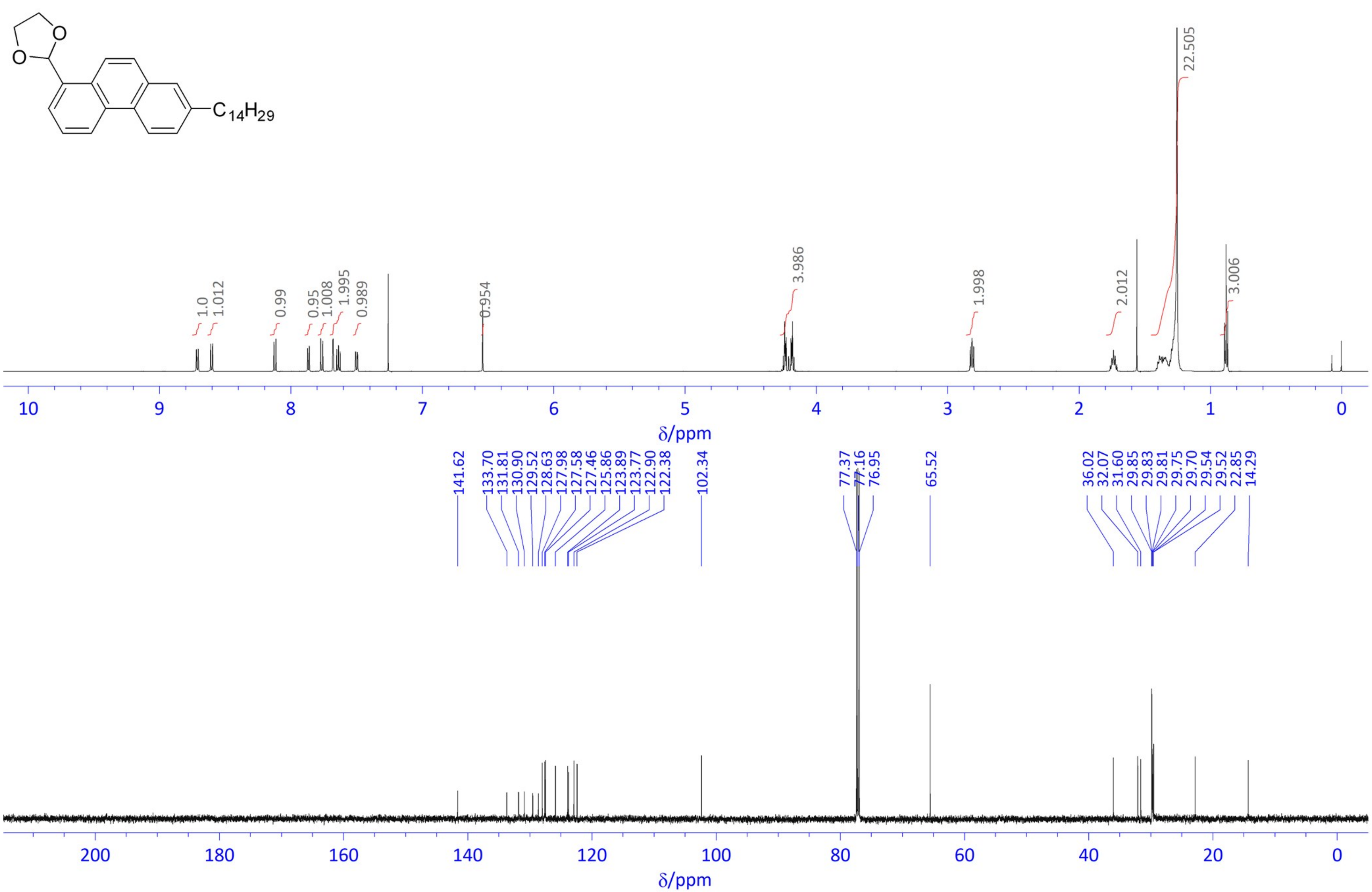


Fig. S9 ^{1}H (600 MHz, upper) and ^{13}C (151 MHz, lower) NMR spectrum of compound **6** ($CDCl_3$)

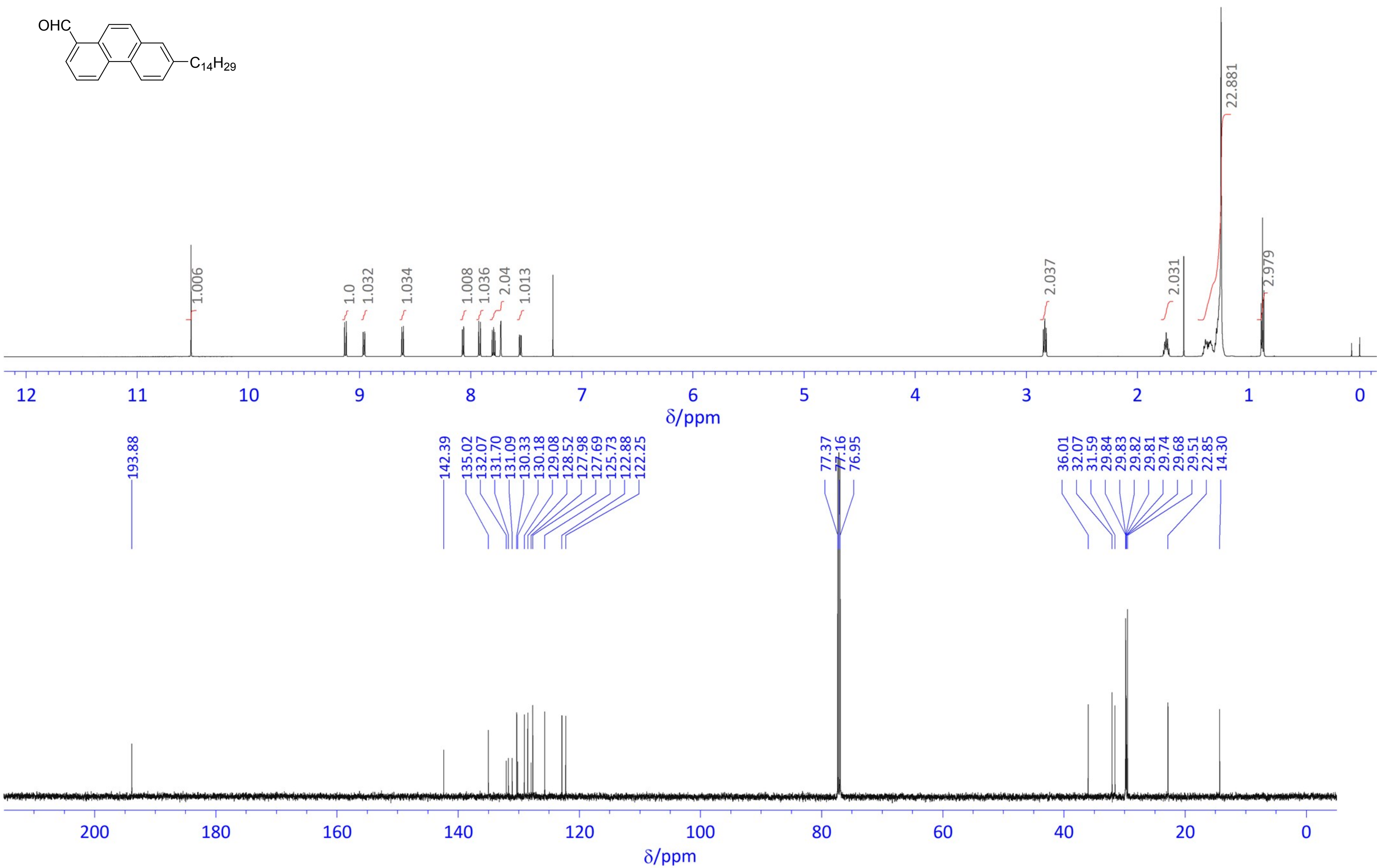


Fig. S10 ^{1}H (600 MHz, upper) and ^{13}C (151 MHz, lower) NMR spectrum of compound **7** ($CDCl_3$).

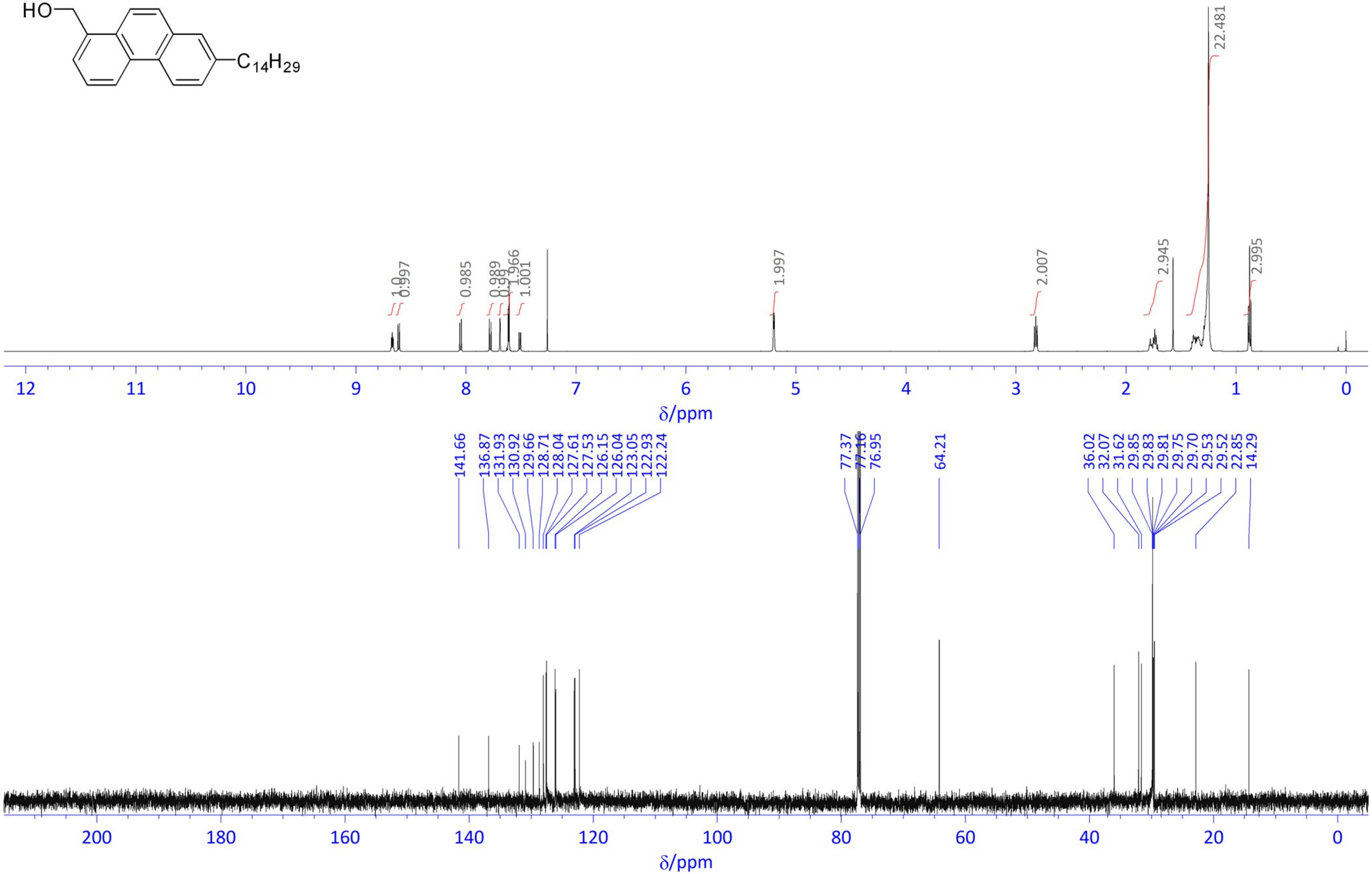


Fig. S11 ^{1}H (600 MHz, upper) and ^{13}C (151 MHz, lower) NMR spectrum of compound **8** ($CDCl_3$).

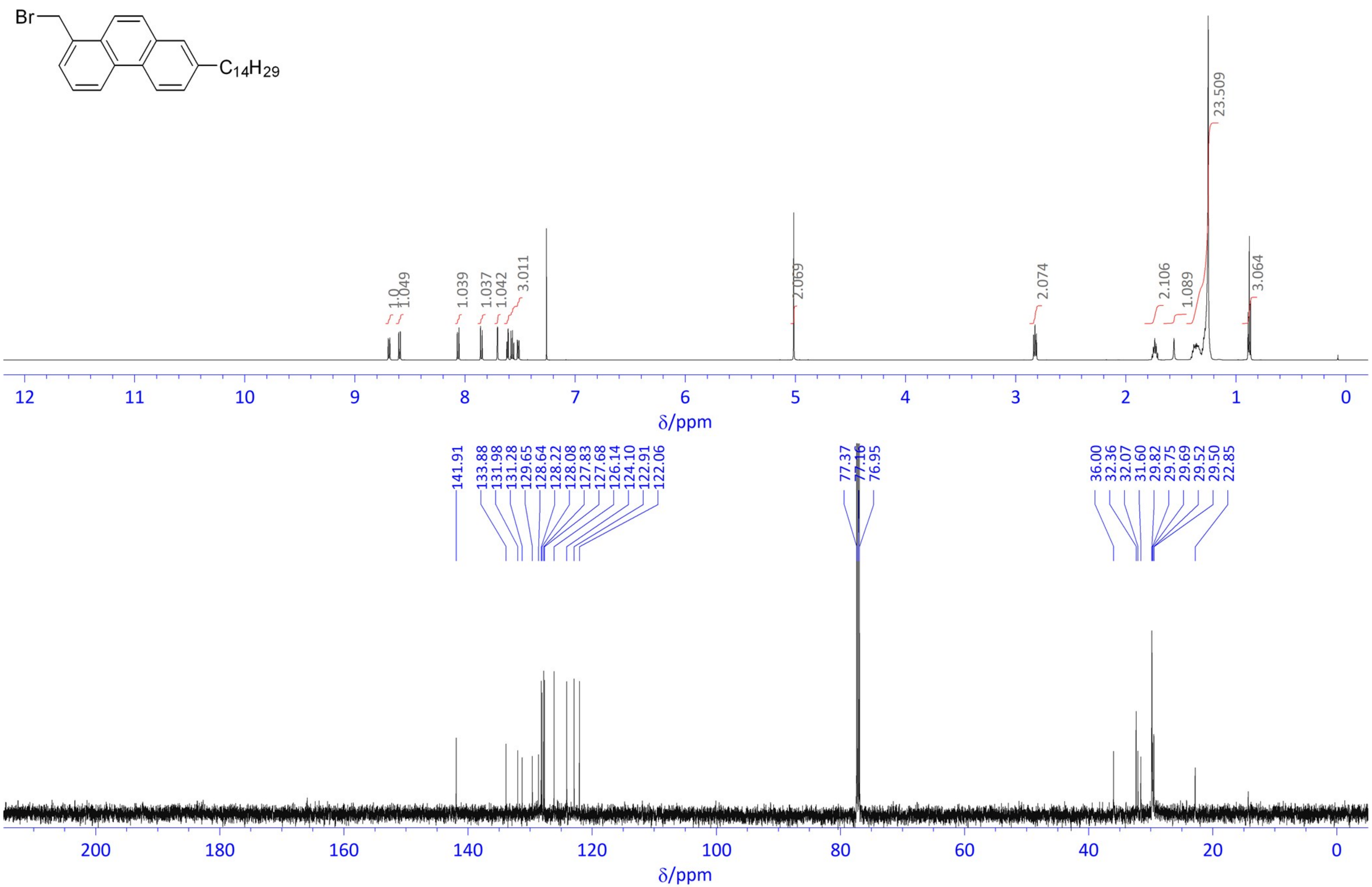


Fig. S12 ^{1}H (600 MHz, upper) and ^{13}C (151 MHz, lower) NMR spectrum of compound **9** ($CDCl_3$).

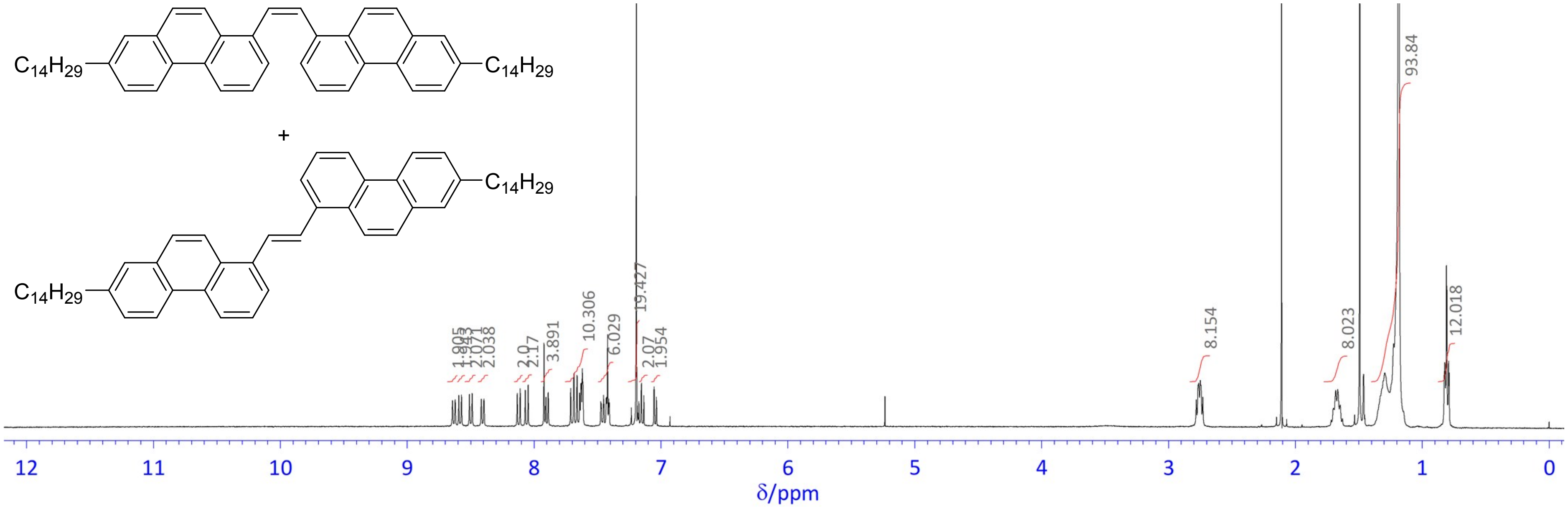


Fig. S13 ^{1}H NMR spectrum of compound **11** (400 MHz, $CDCl_3$).

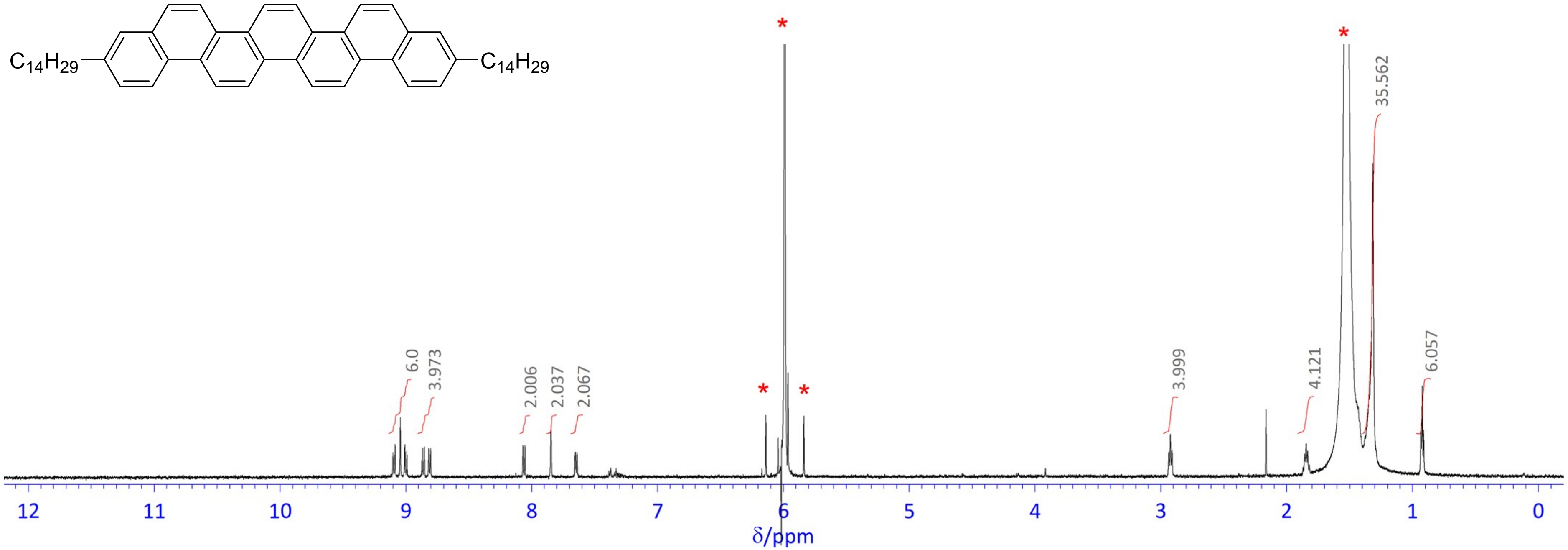


Fig. S14 ^{1}H NMR spectrum of compound $(C_{14}H_{29})_2$-[7]phenacene (600 MHz, $CDCl_2CDCl_2$, 80℃). The asterisked signals were due to residual solvent (*δ* 5.99) and the satellite bands, and water (*δ* 1.53).