Supporting Information

Microwave-promoted Hydrogenation and Alkynylation Reactions with Palladium-loaded Multi-walled Carbon Nanotubes

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1) Microwave Irradiation Experiments. Microwave irradiation experiments were performed using a multi-mode MARS System From CEM Corporation using standard Pyrex vessels (capacity 50 mL). The temperature profiles for microwaves experiments were recorded using a fiber-optic probe protected by a sapphire immersion well inserted directly into the reaction mixture. Pressure profiles were recorded with a pressure sensor directly connected to the reaction vessel.

2) XRD analysis of the mutli-walled carbon nanotubes

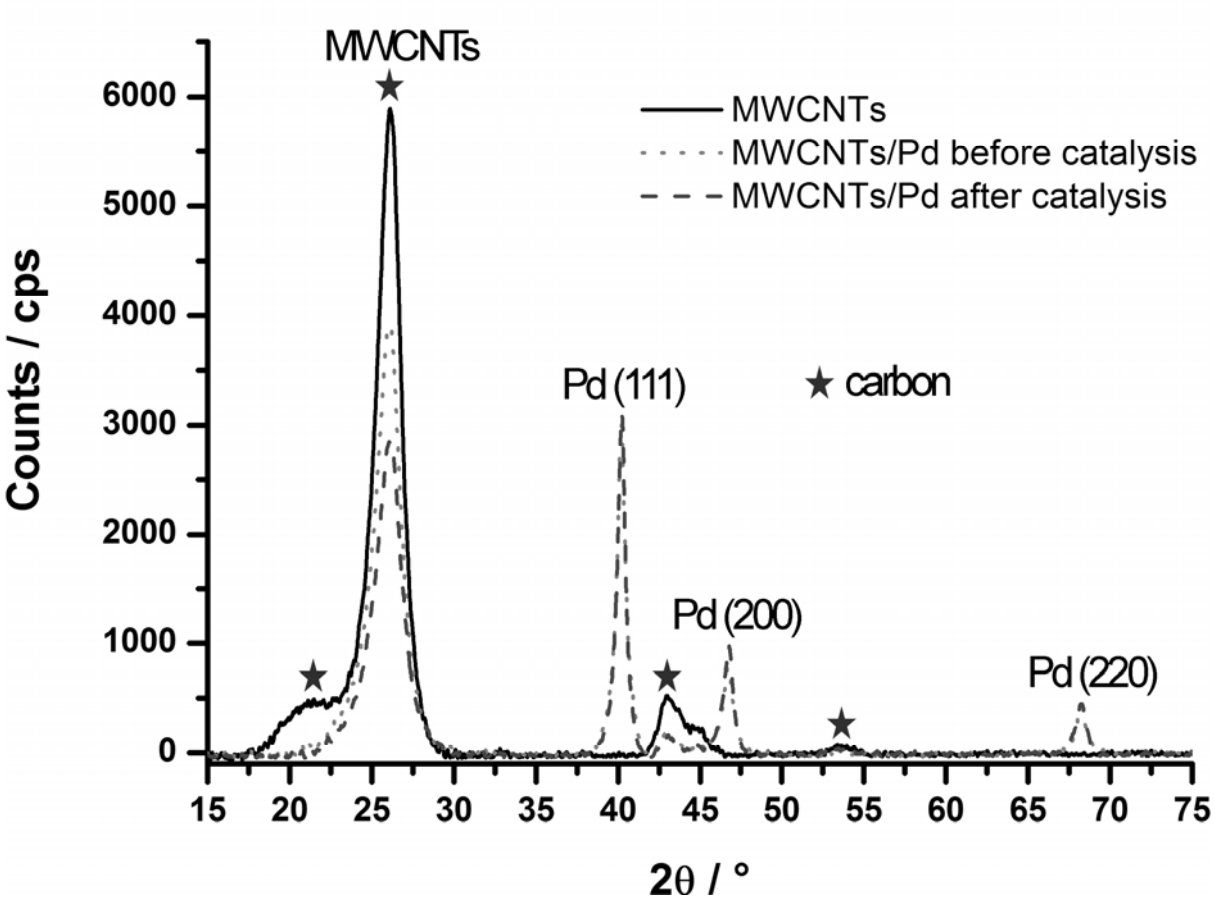
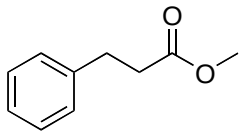


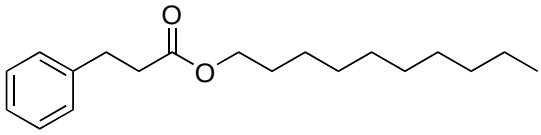
Figure S1. XRD powder diffraction performed on the as prepared MWCNTs, Pd loaded catalysts before nd after catalysts.

3) General procedure for the hydrogenation of various cinnamic esters. To a solution of cinnamic esters (0.31 mmol) dissolved in 10mL of EtOH was added ammonium format (250 mg, 3.10 mmol) and Pd/MWCNTs (10 mg, 10wt.%). The mixture was subsequently heated at different temperature with stirring during 5min in a 50 mL reactor Vessel. The crude reaction mixture was filtered on a glass fritt and the Pd/MWCNTs were washed with 2 x 10 mL of EtOH and was recycled. The solution was diluted with 20 mL of dichloromethane and water was added. The phases were separated and the resulting aqueous phase was extracted with an additional 2 × 20 mL of dichloromethane. The combined organic phases were washed with 2 × 20 mL of distilled water and 1 × 20 mL of brine. The resulting organic phase was dried over $MgSO_4$ and the heterogeneous mixture was filtered. The filtrate was concentrated in vacuum to afford dark red oil. The target product was purified by flash chromatography on silica gel eluting with a dichloromethane:petroleum ether mixture as mobile phase.



1

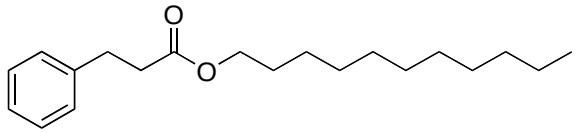
Compound 1. The general procedure was followed using methyl cinnamate esters (50 mg, 0.31 mmol). For the total hydrogenation, the mixture was heated at 140°C (600W, 9 bars) during 5 min. Purification by flash chromatography on silica gel (20:80 dichloromethane:petroleum ether) afforded **1** as a yellow oil (90 mg, 99 %): ^{1}H NMR (300MHz, $CDCl_3$) : δ 7.33-7.18 (m, 5H), 3.66, (s, 3H), 2.96 (t, 2H, ^{3}J = 11.2 Hz), 2.63 (t, 2H, ^{3}J = 11.8 Hz), ^{13}C NMR (75 MHz, $CDCl_3$): δ 173.38, 140.51, 128.89, 128.51, 128.26, 128.07, 1326.27, 51.58, 35.69, 30.95.



2

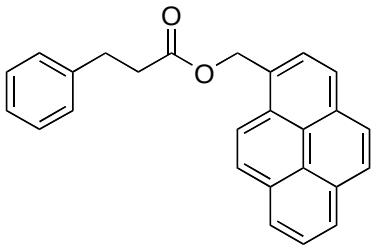
Compound 2. The general procedure was followed using decyl cinnamate ester (90 mg, 0.31 mmol). For the total hydrogenation, the mixture was heated at 200°C (1200W, 15 bars) during 5min. Purifiaction by flash chromatography on silica gel (20:80 dichloromethane:petroleum ether) afforded **2** as a yellow oil (90 mg, 99 %): ^{1}H NMR (300MHz, $CDCl_3$) : δ 7.31-7.26 (m, 2H), 7.22-7.17 (m, 3H), 4.06 (t, 2H, ^{3}J = 9.6 Hz), 2.96 (t, 2H, ^{3}J = 7.53 Hz), 2.63 (t, 2H, ^{3}J = 7.33 Hz), 1.59 (t, 2H, ^{3}J = 6.9 Hz), 1.27 (m, 14H), 0.89 (t, 3H, ^{3}J = 6.7 Hz); ^{13}C NMR (75

MHz, $CDCl_3$): δ 172.98, 140.58, 128.44, 128.26, 126.20, 64.64, 35.92, 31.89, 31.00, 29.58, 29.55, 29.49, 29.31, 29.22, 28.60, 26.10, 25.88, 22.66, 14.08; EI-MS m/z (nature of the peak, intensity) 290.2 ($[M+H]^+$, 100); Anal. Calcd for $C_{19}H_{30}O_2$: C, 78.57; H, 10.41. Found C, 78.89; H, 10.75.



3

Compound 3. The general procedure was followed using 10-undecenyl cinnamate ester (94 mg, 0.31 mmol). For the total hydrogenation, the mixture was heated at 160°C (600W, 12 bars) during 5 min. Purification by flash chromatography on silica gel (20:80 dichloromethane:petroleum ether) afforded **3** as a yellow oil (94 mg, 99 %): ^{1}H NMR (300MHz, $CDCl_3$) : δ 7.31-7.27 (m, 2H), 7.21-7.19 (m, 3H), 4.06 (t, 2H, ^{3}J = 6.9 Hz), 2.96 (t, 2H, ^{3}J = 7.5 Hz), 2.62 (t, 2H, ^{3}J = 8.1 Hz), 1.59 (t, 2H, ^{3}J = 6.6 Hz), 1.27 (m, 16H), 0.89 (t, 3H, ^{3}J = 6.4 Hz); ^{13}C NMR (75 MHz, $CDCl^3$): δ 172.98, 140.58, 128.44, 128.26, 126.20, 64.64, 35.92, 31.89, 31.00, 29.58, 29.55, 29.49, 29.42, 29.31, 29.28, 29.22, 28.60, 25.88, 22.66, 14.08; EI-MS m/z (nature of the peak, intensity) 304.1 ($[M+H]^+$, 100); Anal. Calcd for $C_{20}H_{32}O_2$: C, 78.90; H, 10.59. Found C, 79.34; H, 10.87.

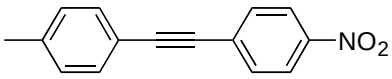


4

Compound 4. The general procedure was followed using methylpyrene cinnamate ester (112 mg, 0.31 mmol). For the total hydrogenation the mixture was heated at 160°C (600W, 12 bars) during 5 min. Purification by flash chromatography on silica gel (30:70 dichloromehtane:petroleum ether) afforded **4** as a white solid (111 mg, 98 %): ^{1}H NMR (200MHz, $CDCl_3$) : δ 8.24-8.00 (m, 9H), 7.22-7.14 (m, 5H), 5.83 (s, 2H), 2.98 (t, 2H, ^{3}J = 8.0 Hz), 2.75 (t, 2H, ^{3}J = 7.8 Hz); ^{13}C NMR (75MHz, $CDCl_3$) : δ 172.94, 136.29, 131.43, 130.88, 129.86, 128.60, 128.43, 128.23, 127.47, 127.26, 127.19, 126.62, 126.19, 125.80, 125.10,

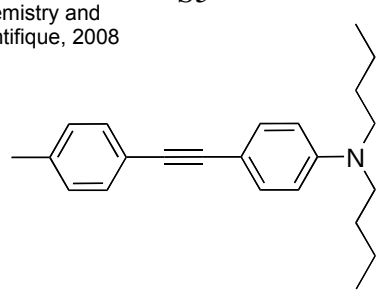
125.02, 124.88, 124.78, 124.70, 123.25, 64.28, 28.60, 28.02; EI-MS m/z (nature of the peak, intensity) 364.1 ($[M+H]^+$, 100); Anal. Calcd for $C_{26}H_{20}O_2$: C, 85.69; H, 5.53. Found C, 85.52; H, 5.22.

4) General procedure for the cross-coupling between Aryl iodide and 4-ethynyl toluene in EtOH/H_2O (1:1) and K_2CO_3 (Table1). To a solution of aryl iodide (0.858 mmol) dissolved in 10 mL (1/1, v/v) of EtOH/H_2O were added, 4-ethynyl toluene (2.145 mmol, 250 mg, 0.272 mL), K_2CO_3 (2.57 mmol, 300 mg) and Pd/MWCNTs or Pd/charcoal (10 mg, 10 wt%). The mixture was subsequently heated at 160°C with stirring during 5 min (300 W, 12-13 bars) in a 50 mL reactor Vessel. The crude reaction mixture was filtered and the Pd/MWCNTs or Pd/charcoal were washed with 2 x 10 mL of EtOH and recycled. The solution was diluted with 20 mL of dichloromethane and 15 mL of water were added. The phases were separated and the resulting aqueous phase was extracted with an additional 2 × 20 mL of dichloromethane. The combined organic phases were washed with 2 × 20 mL of distilled water and 1 × 20 mL of brine. The resulting organic phase was dried over $MgSO_4$ and the heterogeneous mixture was filtered. The filtrate was concentrated in vacuum to afford dark red oil. The residue was purified by flash chromatography on silica gel eluting with a mixture of dichloromethane and petroleum ether to give the desired product.



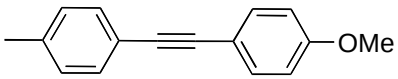
7a

Compound 7a. The general procedure was followed using 4-iodo-nitrobenzene (0.858 mmol, 200 mg). Purification by flash chromatography on silica gel (10:90 dichloromethane:petroleum ether) afforded **7a** as a yellow solid (167 mg, 82 %) or (107 mg, 57% with Pd/charcoal): ^{1}H NMR (300 MHz, $CDCl_3$) : δ 7.94 (AB quartet, 4H, ^{AB}J = 9.2 Hz, $\nu\delta_{AB}$ = 169.2 Hz), 7.33 (AB quartet, 4H, ^{AB}J = 8.1 Hz, $\nu\delta_{AB}$ = 77.3 Hz), 2.42 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 146.87, 139.65, 132.15, 131.76, 130.53, 129.31, 124.37, 123.62, 119.04, 95.10, 87.07, 21.604; IR (KBr, cm^{-1}): 2985; 2853, 2289, 2211, 1992, 1590, 1519, 1405, 1368, 1340, 1275, 1260, 1173, 1138, 1106, 1010, 913, 864, 851, 839, 816.



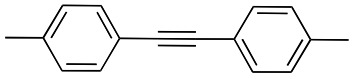
7d

Compound 7d. The general procedure was followed using 4-iodo-N,N'dibuty-aniline (0.858 mmol, 284 mg). Purification by flash chromatography on silica gel (Petroleum ether) afforded **7d** as a yellow oil (123 mg, 45 %) or (46 mg, 17% with Pd/charcoal): ^{1}H NMR (300 MHz, $CDCl_3$) : δ 7.26 (AB quartet, 4H, ^{AB}J = 9.0 Hz, $\nu\delta_{AB}$ = 70.3 Hz), 7.01 (AB quartet, 4H, ^{AB}J = 9.0 Hz, $\nu\delta_{AB}$ = 244.9 Hz), 3.32 (t, 4H, ^{3}J = 7.5 Hz), 2.37 (s, 3H), 1.62-1.55 (m, 4H), 1.38 (dt, 4H, ^{3}J = 7.3 Hz), 0.99 (t, 6H, ^{3}J = 7.2 Hz); ^{13}C NMR (75 MHz, $CDCl_3$): δ 147.79, 137.22, 132.72, 131.08, 128.92, 121.21, 111.18, 108.94, 90.04, 87.05, 50.63, 29.34, 21.38, 20.27, 13.95; IR (KBr, cm^{-1}): 3027, 2955, 2928, 2871, 2733, 2367, 2208, 2123, 1996, 1871, 1603, 1519, 1463, 1423, 1400, 1366, 1287, 1252, 1220, 1195, 1180, 1134, 1108, 1048, 1019, 1004, 926, 900, 852, 809.



7e

Compound 7e. The general procedure was followed using 4-iodoanisole (0.858 mmol, 201 mg). Purification by flash chromatography on silica gel (10:90 dichloromethane:petroleum ether) afforded **7e** as a white solid (86 mg, 45 %) or (34 mg, 18% with Pd/charcoal): ^{1}H NMR (300 MHz, $CDCl_3$) : δ 7.16 (AB quartet, 4H, ^{AB}J = 9.2 Hz, $\nu\delta_{AB}$ = 176.4 Hz), 7.27 (AB quartet, 4H, ^{AB}J = 9,3 Hz, $\nu\delta_{AB}$ = 176,4 Hz), 3.82 (s, 3H), 2.86 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 159.49, 137.98, 132.96, 131.32, 129.06, 120.51, 115.61, 113.96, 88.64, 88.18, 55.29, 21.47; IR (KBr, cm^{-1}): 3004, 2917, 2947, 2928, 2838, 2665, 2644, 2323, 2107, 1997, 1945, 1912, 1834, 1791, 1746, 1666, 1599, 1567, 1510, 1450, 1405, 1287, 1270, 1245, 1173, 1135, 1106, 1027, 942, 885, 829, 818.

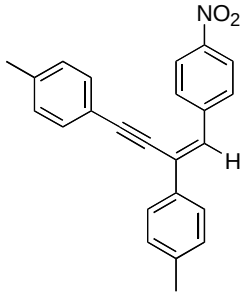


7f

Compound 7f. The general procedure was followed using 4-iodo-toluene (0.858 mmol, 201 mg). Purification by flash chromatography on silica gel (Petroleum ether) afforded **7f** as a

white solid (70 mg, 40 %) or (30 mg, 17% with Pd/charcoal):: ^{1}H NMR (300 MHz, $CDCl_3$) : δ 7.28 (AB quartet, 8H, ^{AB}J = 8,02 Hz, $\nu\delta_{AB}$ = 80,04 Hz), 2.36 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 138.15, 131.42, 129.06, 120.40, 88.86, 21.47; IR (KBr, cm^{-1}): 30189, 2921, 2853, 2715, 2658, 2611, 2343, 2111, 1995, 1912, 1855, 1738, 1652, 1540, 1515, 1446, 1410, 1306, 1263, 1211, 1184, 1123, 1037, 1019, 839, 814.

5) General procedure for the cross-coupling between Aryl iodide and 4-ethynyl toluene in EtOH/piperidine (Table1). To a solution of aryl iodide (0.858 mmol) dissolved in 10 mL of EtOH were added 4-ethynyl toluene (2.145 mmol, 250 mg, 0.272 mL), piperidine (2.574 mmol, 219 mg) and Pd/MWCNTs or Pd/charcoal (40 mg, 10 wt%). The mixture was subsequently heated at 120°C with stirring during 5min (300W, 6-7 bars) in a 50 mL reactor Vessel. The crude reaction mixture was filtered and the Pd/MWCNTs or Pd/charcoal were washed with 2 x 10 mL of EtOH and recycled. The solution was diluted with 20 mL of dichloromethane and 15 mL of water was added. The phases were separated and the resulting aqueous phase was extracted with an additional 2 × 20 mL of dichloromethane. The combined organic phases were washed with 2 × 20 mL of distilled water and 1 × 20 mL of brine. The resulting organic phase was dried over $MgSO_4$ and the heterogeneous mixture was filtered. The filtrate was concentrated in vacuum to afford dark red oil. The residue was purified by flash chromatography on silica gel eluting with a mixture of dichloromethane and petroleum ether to give the product.



Compound 8a. The general procedure was followed using 4-iodo-nitrobenzene (0. 858 mmol, 200 mg). Purification by flash chromatography on silica gel (20:80 dichloromethane:petroleum ether) afforded **8a** as a fluorescent yellow solid (36 mg, 12 %). ^{1}H NMR (300 MHz, $CDCl_3$) : δ 8.21 (AB quartet, 4H, ^{AB}J = 6.8 Hz, $\nu\delta_{AB}$ = 33.7 Hz), 7.49 (AB quartet, 4H, ^{AB}J = 6.3 Hz, $\nu\delta_{AB}$ = 136.1 Hz), 7.34 (AB quartet, 4H, ^{AB}J = 6.3 Hz, $\nu\delta_{AB}$ = 68.1 Hz), 7.20 (s, 1H), 2.42 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 146.60, 143.36, 139.53, 138.95, 135.98, 131.57, 130.43, 129.36, 126.59, 126.24, 123.61, 119.58, 99.36, 87.45, 21.61, 21.24; UV-vis (λ_{max} nm, ε = $M^{-1}cm^{-1}$), CH_2Cl_2: 251 (29800), 297 (18900), 380 (27400); IR (KBr, cm^{-1}): 3118, 3056, 3030, 2918, 2850, 2691, 2445, 2347, 2195, 2114, 1993, 1910, 1796,

1747, 1671, 1591, 1576, 1565, 1506, 1417, 1376, 1338, 1277, 1261, 1184, 1176, 1107, 1041, 1017, 1004, 969, 882, 864, 842, 813; MALDI-TOF m/z (nature of the peak) 353.1 ($[M+H]^+$, 100); Anal. Calcd for $C_{24}H_{19}NO_2$: C, 81.56; H, 5.42; N, 3.96. Found: C, 81.37; H, 5.28; N, 3.70.

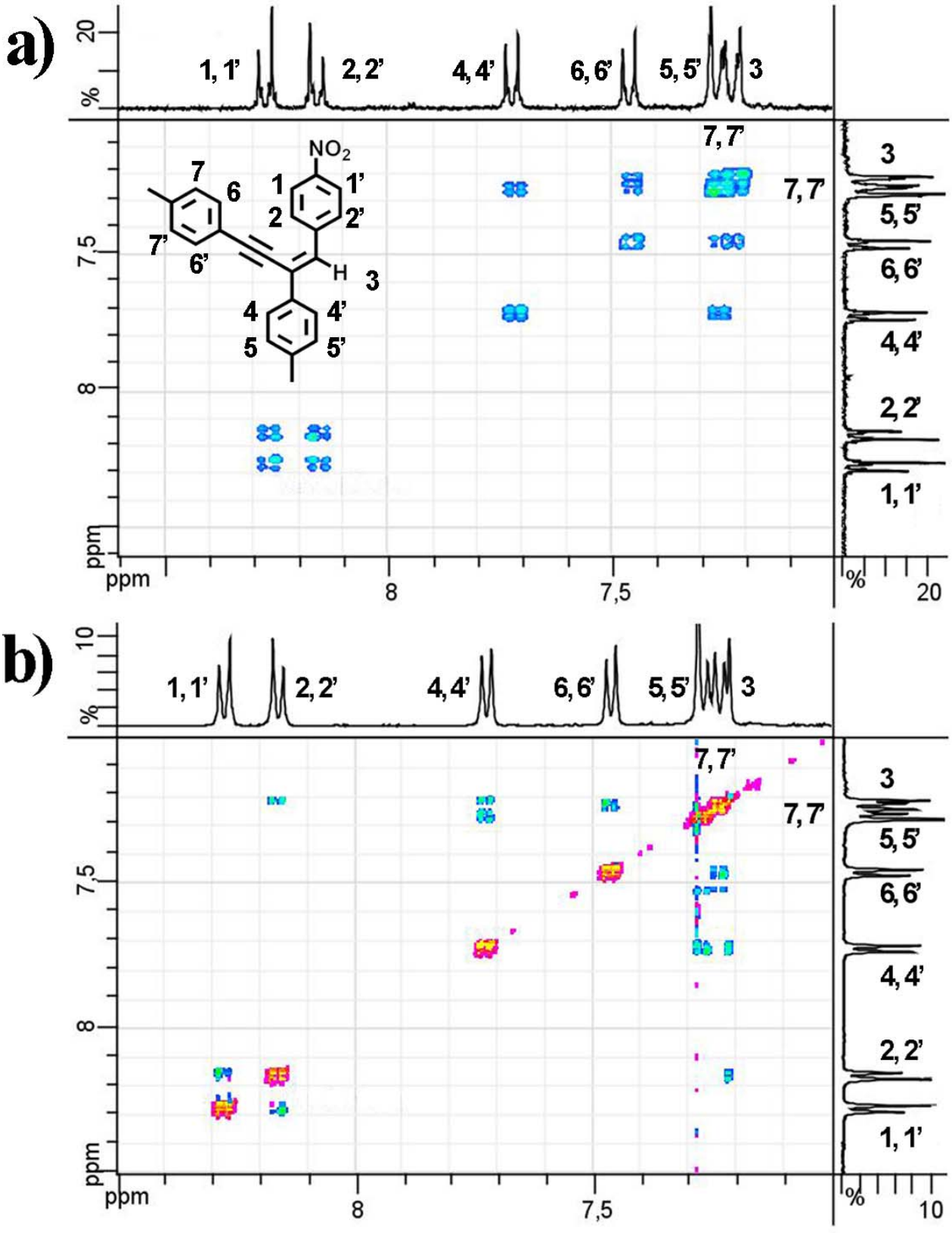
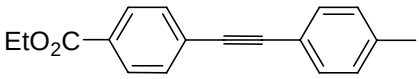
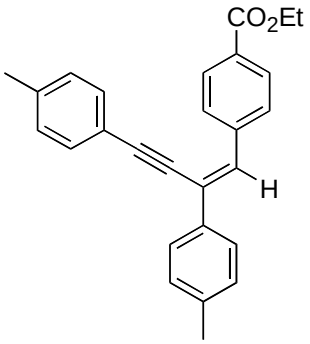


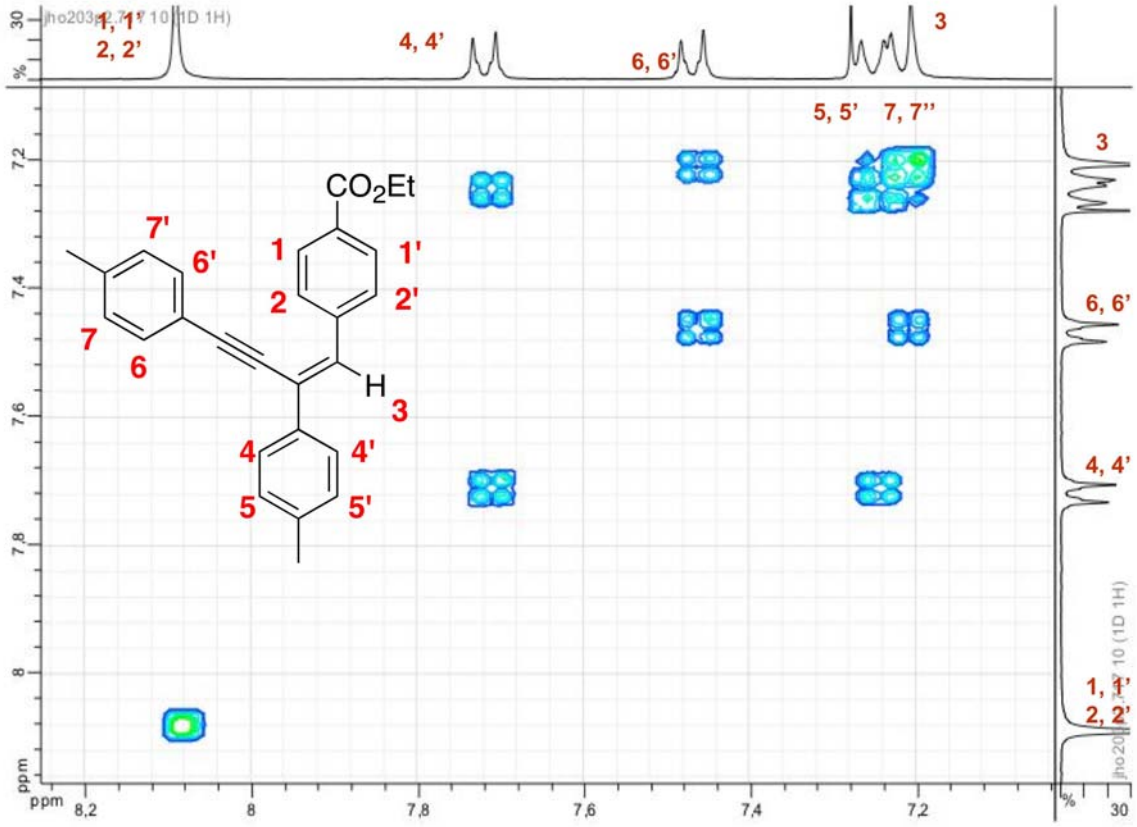
Figure S2. COSY (Top) and NOESY (Bottom) spectra of compound **8a** measured in $CDCl_3$.



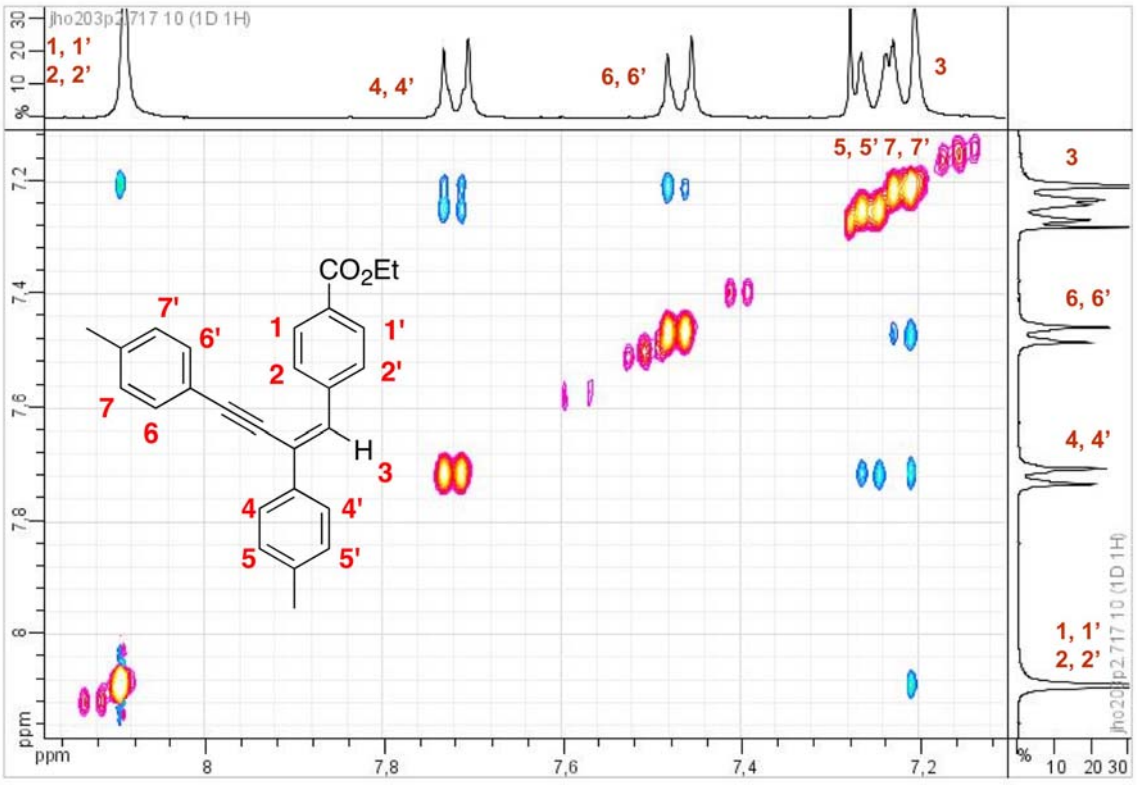
Compound 7b. The general procedure was followed using 4-iodo-ethylbenzoate (0. 858 mmol, 238 mg). Purification by flash chromatography on silica gel (20:80 dichloromethane:petroleum ether) afforded **7b** as a white solid (192 mg, 85 %) or (108 mg, 48% with Pd/charcoal):: ^{1}H NMR (300 MHz, $CDCl_3$) : δ 7.78 (AB quartet, 4H, ^{AB}J = 9.4 Hz, $\nu\delta_{AB}$ = 133.1 Hz), 7.30 (AB quartet, 4H, ^{AB}J = 7.9 Hz, $\nu\delta_{AB}$ = 80.5 Hz), 4.38 (q, 2H, ^{3}J = 6.8 Hz), 2.37 (s, 3H), 1.40 (t, 3H, ^{3}J = 6.9 Hz); ^{13}C NMR (75 MHz, $CDCl_3$): δ 166.10, 138.96, 131.96, 131.62, 131.36, 129.64, 129.44, 129.19, 128.11, 119.65, 92.53, 88.10, 61.08, 21.53, 14.30; IR (KBr, cm^{-1}): 3120, 2982, 2691, 2308, 2213, 1914, 1716, 1602, 1519, 1462, 1387, 1368, 1306, 1270, 1163, 1138, 1108, 1016, 1001, 859, 837, 813.



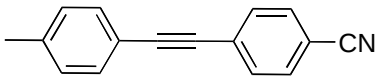
Compound 8b. The general procedure was followed using 4-iodo-ethylbenzoate (0.858 mmol, 238 mg). Purification by flash chromatography on silica gel (20:80 dichloromethane:petroleum ether) afforded **8b** as a yellow fluorescent solid (19 mg, 6 %). ^{1}H NMR (300 MHz, $CDCl_3$) : δ 8.08 (AB system, 4H), 7.47 (AB quartet, 4H, ^{AB}J = 7.2 Hz, $\nu\delta_{AB}$ = 140.5 Hz), 7.33 (AB quartet, 4H, ^{AB}J = 7.5 Hz, $\nu\delta_{AB}$ = 74.9 Hz), 7.19 (s, 1H), 4.38 (q, 2H, ^{3}J = 6.8 Hz), 2.41 (s, 6H), 1.40 (t, 3H, ^{3}J = 6.9 Hz); ^{13}C NMR (75 MHz, $CDCl_3$): δ 166.40, 141.24, 139.06, 138.33, 136.46, 132.16, 131.51, 130.73, 129.49, 129.42, 129.30, 129.25, 129.20, 129.17, 129.16, 128.76, 127.74, 126.48, 126.09, 124.11, 120.01, 98.31, 87.87, 80.94, 21.58, 21.19, 14.36; UV-vis (λ_{max} nm, ε = $M^{-1}cm^{-1}$), CH_2Cl_2: 242 (24900), 274 (27000), 356 (29000); IR (KBr, cm^{-1}): 3421, 3028, 2980, 2922, 2867, 2323, 2195, 2112, 1991, 1908, 1803, 1713, 1605, 1509, 1445, 1409, 1391, 1366, 1310, 1271, 1209, 1177, 1102, 1019, 965, 883, 847, 814; MALDI-TOF m/z (nature of the peak, intensity) 381.2 ($[M+H]^+$, 100); Anal. Calcd for $C_{27}H_{24}O_2$: C, 85.23; H, 6.36 Found: C, 85.17; H, 6.08.



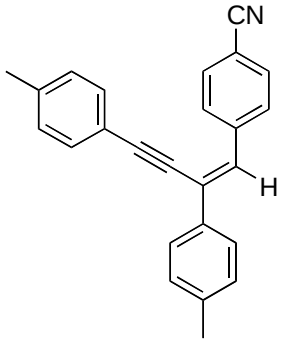
COSY



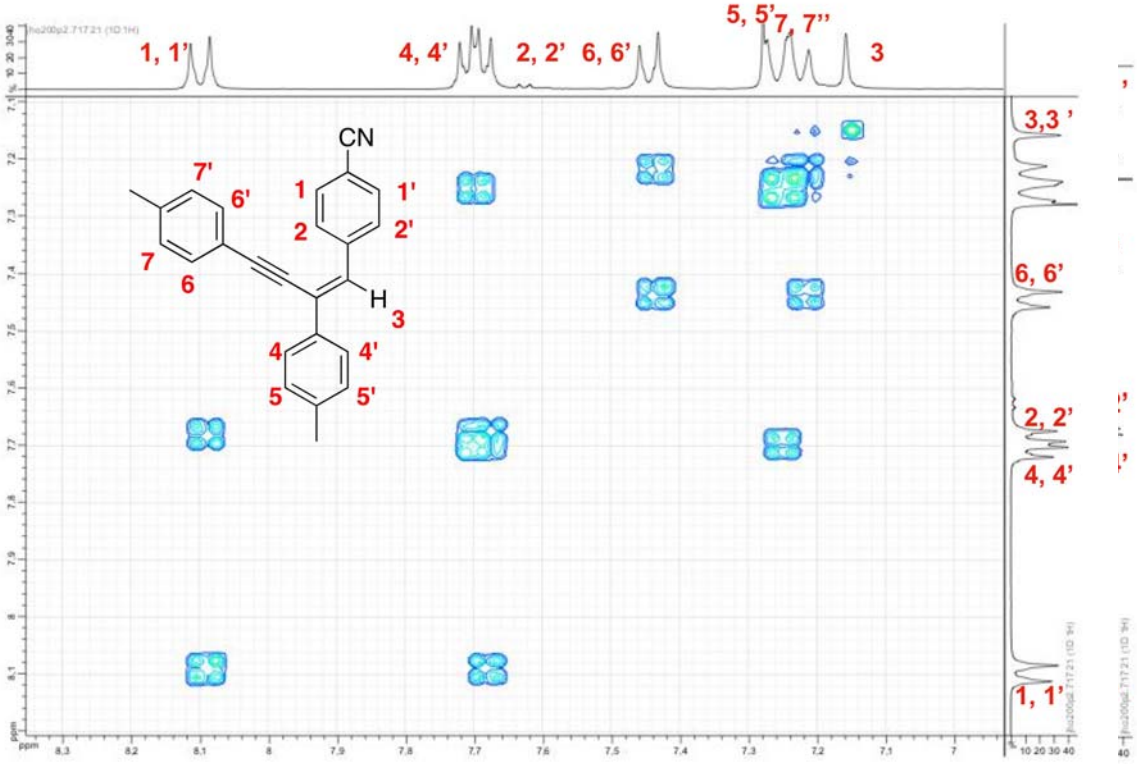
NOESY



Compound 7c. The general procedure was followed using 4-iodo-cyanobenzene (0. 858 mmol, 197 mg). Purification by flash chromatography on silica gel (20:80 dichloromethane:petroleum ether) afforded **7c** as a white solid (158 mg, 85%) or (38 mg, 20% with Pd/charcoal): ^{1}H NMR (300 MHz, $CDCl_3$) : δ 7.60 (AB quartet, 4H, ^{AB}J = 8.3 Hz, $\nu\delta_{AB}$ = 10.2 Hz), 7.31 (AB quartet, 4H, ^{AB}J = 8.1 Hz, $\nu\delta_{AB}$ = 75.6 Hz), 2.38 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 139.44, 132.00, 131.97, 131.69, 129.27, 128.48, 119.14, 118.56, 111.25, 94.11, 87.19, 21,56; IR (KBr, cm^{-1}): 3130, 3056, 2903, 2980, 2663, 2324, 2230, 2210, 2090, 1992, 1902, 1857, 1798, 1715, 1678, 1640, 1598, 1511, 1446, 1411, 1308, 1214, 1274, 1178, 1132, 1041, 1013, 918, 840, 819.



Compound 8c. The general procedure was followed using 4-iodo-cyanobenzene (0.858 mmol, 197 mg). Purification by flash chromatography on silica gel (20:80 dichloromethane:petroleum ether) afforded **8c** as a yellow fluorescent solid (23 mg, 8 %). ^{1}H NMR (300 MHz, $CDCl_3$) : δ 7.88 (AB quartet, 4H, ^{AB}J = 6.9 Hz, $\nu\delta_{AB}$ = 124.3 Hz), 7.47 (AB quartet, 4H, ^{AB}J = 6.9Hz, $\nu\delta_{AB}$ = 136.0 Hz), 7.32 (AB quartet, 4H, ^{AB}J = 7.9 Hz, $\nu\delta_{AB}$ = 65.6 Hz), 7.14 (s, 1H), 2.41 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 141.34, 139.40, 138.77, 136.05, 131.99, 131.52, 130.97, 129.38, 129.33, 129.27, 126.53, 125.47, 119.67, 119.06, 110.75, 99.00, 87.44, 21.60, 21.60; UV-vis (λ_{max} nm, ε = $M^{-1}cm^{-1}$), CH_2Cl_2: 250 (29,600) 269 (28,100), 353 (34,100). IR (KBr, cm^{-1}): 3125, 3028, 2985, 2777, 2663, 2601, 2324, 2223, 2195, 2091, 1995, 1907, 1881, 1867, 1834, 1805, 1789, 1748, 1685, 1704, 1666, 1652, 1601, 1584, 1545, 1457, 1417, 1372, 1339, 1315, 1299, 1270, 1178, 1114, 1041, 1022, 1001, 937, 913, 880, 842, 814; MALDI-TOF m/z (nature of the peak) 334.2 ($[M+H]^+$, 100); Anal. Calcd for $C_{24}H_{19}NO_2$: C, 90.06; H, 5.74; N, 4.20 Found: C, 89.79; H, 5.56; N, 3.89.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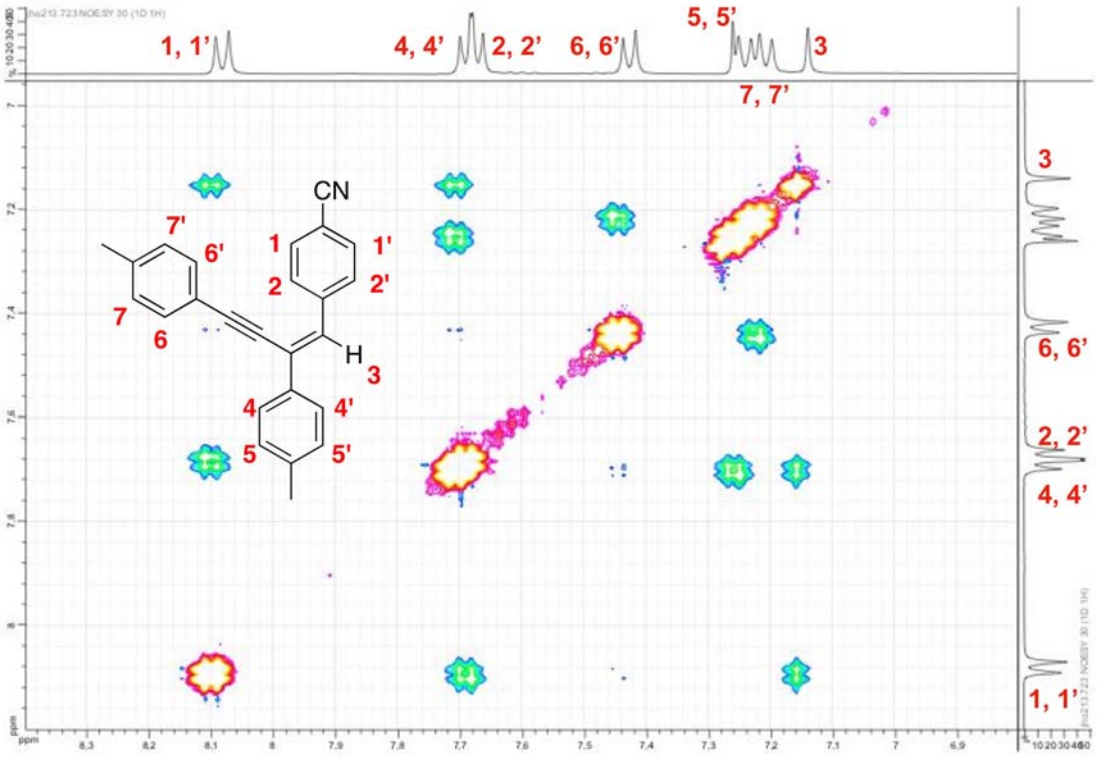
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NOESY

6) X-ray experimental section.

The X-ray diffraction data were collected with MoKα radiation (λ=0.71073 =, $2\theta_{max}$=46.7°, φ + ω scan mode) on a Bruker KAPPA CCD diffractometer at room temperature. The structure was solved by direct methods using SHELXS-97[*] and refined by full-matrix least-squares on F^2 with SHELXL-97.[*] All non-hydrogen atoms were refined anisotropically and all hydrogen atoms were located on difference–Fourier syntheses but refined with a riding model and with $U_{iso}(H)=1.2\ U_{eq}(C)$ (or 1.5 for methyl group).

Crystal data for compound **8b**: Empirical formula: C27 H24 O2; Mr=380.46 g.mol^{-1}; triclinic, space group P -1, a=5.644(1), b=12.047(2), c=16.159(2) Å, α=82.02(5), β=82.48(5), γ=81.90(4)°, V=1070.2(3) Å^3, T=295 K, ρ_{calcd}=1.181 g.cm^{-3}, crystal size 0.55 x 0.50 x 0.18 mm^3; absorption coefficient= 0.073 mm^{-1}, Z=2, F(000)=404;
limiting indices: $-6 \le h \le 6$, $-13 \le k \le 13$, $-17 \le l \le 17$; reflections collected/unique: 22116/3082; R(int) = 0.0247; data/restraints/parameters: 3081 / 0 / 266, GOF on F^2=1.038. Final R indexes (I>2σ(I)): R1=0.0475, wR2=0.1244; for all data: R1=0.0718, wR2=0.1436. Largest difference peak and hole: + 0.16 and -0.12 e.Å^{-3}.
CCDC-661999 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

Crystal data

Formula sum	$C_{27}\ H_{24}\ O_2$
Formula weight	380.46
Crystal system	triclinic
Space group	*P* -1 (no. 2)
Unit cell dimensions	*a* = 5.644(1) Å
	b = 12.047(1) Å
	c = 16.159(1) Å
	α = 82.02(5) °
	β = 82.48(5) °
	γ = 81.90(5) °
Cell volume	1070.24(20) Å^3
Z	2
Density, calculated	1.181 g/cm^3
R_{All}	0.072
Pearson code	aP106
Formula type	N2O24P27
Wyckoff sequence	i^{53}

Atomic coordinates and isotropic displacement parameters (in Å ^{2})

Atom	Wyck.	x	y	z	U
O1	2*i*	0.3314(3)	0.27326(11)	0.02914(9)	
O2	2*i*	0.4340(3)	0.37680(14)	0.11883(12)	
C1	2*i*	1.3142(3)	-0.10998(16)	0.23877(12)	
C2	2*i*	1.1489(3)	-0.08999(16)	0.18312(12)	
H2	2*i*	1.14400	-0.15010	0.15330	0.0810
C3	2*i*	0.9774(3)	0.00888(16)	0.16158(12)	
C4	2*i*	0.9582(4)	0.11224(18)	0.19392(14)	
H4	2*i*	1.06210	0.12130	0.23200	0.0930

C5	2*i*	0.7886(4)	0.20027(17)	0.17030(13)	
H5	2*i*	0.77840	0.26780	0.19310	0.0920
C6	2*i*	0.6329(3)	0.19071(16)	0.11335(11)	
C7	2*i*	0.6496(4)	0.08930(17)	0.08038(13)	
H7	2*i*	0.54610	0.08090	0.04200	0.0850
C8	2*i*	0.8193(4)	0.00091(17)	0.10444(13)	
H8	2*i*	0.82820	-0.06650	0.08160	0.0860
C9	2*i*	0.4591(4)	0.28981(17)	0.08910(13)	
C10	2*i*	0.1593(4)	0.36755(18)	0.00123(15)	
H10A	2*i*	0.24290	0.43000	-0.02650	0.1010
H10B	2*i*	0.05310	0.39290	0.04900	0.1010
C12	2*i*	1.4684(3)	-0.22104(16)	0.25178(11)	
C13	2*i*	1.4185(3)	-0.31829(17)	0.22266(13)	
H13	2*i*	1.28390	-0.31440	0.19420	0.0870
C14	2*i*	1.5632(4)	-0.41953(17)	0.23495(13)	
H14	2*i*	1.52440	-0.48230	0.21450	0.0880
C15	2*i*	1.7638(3)	-0.43035(17)	0.27667(12)	
C16	2*i*	1.8127(4)	-0.33463(19)	0.30685(13)	
H16	2*i*	1.94630	-0.33940	0.33590	0.0910
C17	2*i*	1.6690(3)	-0.23297(18)	0.29504(13)	
H17	2*i*	1.70720	-0.17080	0.31650	0.0870
C18	2*i*	1.9294(4)	-0.53931(19)	0.28697(15)	
H18A	2*i*	1.83590	-0.60080	0.30500	0.1340
H18B	2*i*	2.03490	-0.53570	0.32830	0.1340
H18C	2*i*	2.02320	-0.55100	0.23420	0.1340
C19	2*i*	1.3578(3)	-0.02665(16)	0.28825(12)	
C20	2*i*	1.4154(3)	0.03721(17)	0.33053(13)	
C21	2*i*	1.4914(3)	0.11424(17)	0.37893(12)	
C22	2*i*	1.3810(4)	0.2225(2)	0.38003(17)	
H22	2*i*	1.24970	0.24670	0.34990	0.1150
C23	2*i*	1.4601(5)	0.2965(2)	0.42488(19)	
H23	2*i*	1.38280	0.37010	0.42330	0.1290
C11	2*i*	0.0179(4)	0.32905(18)	-0.05814(13)	
H11A	2*i*	0.12440	0.30460	-0.10530	0.1240
H11B	2*i*	-0.09780	0.39030	-0.07740	0.1240
H11C	2*i*	-0.06430	0.26740	-0.03010	0.1240
C24	2*i*	1.6473(5)	0.2652(2)	0.47120(14)	
C25	2*i*	1.7583(5)	0.1572(3)	0.47001(16)	
H25	2*i*	1.88890	0.13340	0.50050	0.1350
C26	2*i*	1.6819(5)	0.0818(2)	0.42464(17)	
H26	2*i*	1.76110	0.00870	0.42540	0.1240
C27	2*i*	1.7360(6)	0.3463(3)	0.52026(17)	
H27A	2*i*	1.87860	0.31060	0.54430	0.2050
H27B	2*i*	1.61330	0.36720	0.56430	0.2050
H27C	2*i*	1.77250	0.41270	0.48320	0.2050

Anisotropic displacement parameters (in Å ^{2})

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O1	0.0949(10)	0.0590(8)	0.0839(10)	0.0050(7)	-0.0334(8)	-0.0155(7)
O2	0.1460(15)	0.0778(11)	0.1318(14)	0.0262(10)	-0.0685(12)	-0.0474(10)
C1	0.0583(11)	0.0629(12)	0.0722(12)	-0.0136(9)	-0.0108(9)	-0.0086(9)
C2	0.0678(12)	0.0607(12)	0.0790(13)	-0.0097(10)	-0.0181(10)	-0.0145(10)
C3	0.0648(12)	0.0581(12)	0.0682(12)	-0.0093(9)	-0.0116(9)	-0.0080(9)
C4	0.0845(14)	0.0722(14)	0.0851(14)	-0.0063(11)	-0.0326(12)	-0.0196(11)
C5	0.0927(15)	0.0594(12)	0.0826(14)	-0.0001(11)	-0.0287(12)	-0.0198(10)
C6	0.0711(12)	0.0602(12)	0.0617(11)	-0.0057(9)	-0.0123(9)	-0.0100(9)
C7	0.0808(14)	0.0604(12)	0.0757(13)	-0.005(1)	-0.0252(11)	-0.0134(10)
C8	0.0801(13)	0.0566(12)	0.0832(14)	-0.0025(10)	-0.0266(11)	-0.0161(10)
C9	0.0848(14)	0.0625(13)	0.0722(13)	-0.0036(11)	-0.0194(11)	-0.0169(10)
C10	0.1006(16)	0.0596(12)	0.0932(15)	0.0112(11)	-0.0388(13)	-0.0114(11)
C12	0.0564(11)	0.0651(12)	0.0668(12)	-0.0128(9)	-0.0087(9)	-0.0091(9)
C13	0.0646(12)	0.0705(13)	0.0887(14)	-0.0081(10)	-0.0246(11)	-0.0178(11)
C14	0.0705(13)	0.0631(13)	0.0912(15)	-0.011(1)	-0.0180(11)	-0.0167(10)
C15	0.0620(12)	0.0661(13)	0.0693(12)	-0.0053(10)	-0.0080(9)	-0.0069(10)
C16	0.0665(12)	0.0828(15)	0.0819(14)	-0.0033(11)	-0.0258(11)	-0.0158(11)
C17	0.0686(13)	0.0702(14)	0.0872(14)	-0.0093(10)	-0.0243(11)	-0.0199(11)
C18	0.0863(15)	0.0755(15)	0.1044(17)	0.0014(12)	-0.0191(13)	-0.0098(13)
C19	0.0648(12)	0.0643(12)	0.0748(13)	-0.0085(10)	-0.0156(10)	-0.0098(10)
C20	0.0676(12)	0.0687(13)	0.0738(13)	-0.0065(10)	-0.0196(10)	-0.0103(10)
C21	0.0685(12)	0.0702(13)	0.0632(12)	-0.0089(10)	-0.0169(9)	-0.0120(9)
C22	0.0866(15)	0.0845(16)	0.129(2)	0.0084(13)	-0.0470(14)	-0.0428(14)

C23	0.0952(18)	0.0982(18)	0.154(2)	0.0006(15)	-0.0320(17)	-0.0597(17)
C11	0.0882(15)	0.0776(15)	0.0830(15)	-0.0054(12)	-0.0228(12)	-0.0092(11)
C24	0.0961(18)	0.114(2)	0.0674(14)	-0.0368(16)	-0.0070(12)	-0.0269(13)
C25	0.122(2)	0.125(2)	0.107(2)	-0.0292(19)	-0.0692(17)	-0.0010(17)
C26	0.1134(19)	0.0829(17)	0.123(2)	-0.0037(14)	-0.0628(17)	-0.0079(14)
C27	0.153(3)	0.188(3)	0.100(2)	-0.083(3)	-0.0061(18)	-0.067(2)

Selected geometric parameters (Å, °)

O1—C9	1.332(28)	C15—C16	1.387(24)
O1—C10	1.449(44)	C15—C18	1.503(41)
O2—C9	1.196(22)	C16—C17	1.375(37)
C1—C2	1.354(33)	C16—H16	0.930(19)
C1—C19	1.432(30)	C17—H17	0.930(17)
C1—C12	1.494(40)	C18—H18A	0.960(24)
C2—C3	1.458(43)	C18—H18B	0.960(21)
C2—H2	0.929(19)	C18—H18C	0.960(25)
C3—C8	1.387(30)	C19—C20	1.200(26)
C3—C4	1.401(24)	C20—C21	1.435(31)
C4—C5	1.373(42)	C21—C26	1.365(34)
C4—H4	0.93(2)	C21—C22	1.365(30)
C5—C6	1.379(30)	C22—C23	1.377(30)
C5—H5	0.929(16)	C22—H22	0.930(24)
C6—C7	1.386(24)	C23—C24	1.355(34)
C6—C9	1.477(44)	C23—H23	0.930(21)
C7—C8	1.377(42)	C11—H11A	0.960(28)
C7—H7	0.93(2)	C11—H11B	0.96(3)
C8—H8	0.930(16)	C11—H11C	0.960(27)
C10—C11	1.483(32)	C24—C25	1.364(30)
C10—H10A	0.970(27)	C24—C27	1.513(33)
C10—H10B	0.970(28)	C25—C26	1.388(30)
C12—C17	1.387(29)	C25—H25	0.930(24)
C12—C13	1.398(23)	C26—H26	0.929(21)
C13—C14	1.374(37)	C27—H27A	0.960(26)
C13—H13	0.931(19)	C27—H27B	0.960(29)
C14—C15	1.374(28)	C27—H27C	0.960(27)
C14—H14	0.929(17)		
C9—O1—C10	116.06(18)	C16—C15—C18	120.89(20)
C2—C1—C19	122.98(18)	C17—C16—C15	121.80(21)
C2—C1—C12	121.99(18)	C17—C16—H16	119.11(23)
C19—C1—C12	115.03(17)	C15—C16—H16	119.09(22)
C1—C2—C3	131.57(18)	C16—C17—C12	121.47(19)
C1—C2—H2	114.18(19)	C16—C17—H17	119.25(23)
C3—C2—H2	114.25(19)	C12—C17—H17	119.27(20)
C8—C3—C4	116.77(19)	C15—C18—H18A	109.49(21)
C8—C3—C2	117.83(18)	C15—C18—H18B	109.44(21)
C4—C3—C2	125.40(19)	H18A—C18—H18B	109.49(24)
C5—C4—C3	121.07(20)	C15—C18—H18C	109.46(21)
C5—C4—H4	119.48(23)	H18A—C18—H18C	109.47(24)
C3—C4—H4	119.45(22)	H18B—C18—H18C	109.48(23)
C4—C5—C6	121.25(21)	C20—C19—C1	173.73(21)
C4—C5—H5	119.42(22)	C19—C20—C21	177.99(22)
C6—C5—H5	119.33(21)	C26—C21—C22	117.46(21)
C5—C6—C7	118.56(19)	C26—C21—C20	120.61(20)
C5—C6—C9	118.40(18)	C22—C21—C20	121.91(20)
C7—C6—C9	123.03(19)	C21—C22—C23	121.30(23)
C8—C7—C6	120.05(20)	C21—C22—H22	119.33(24)
C8—C7—H7	119.98(23)	C23—C22—H22	119.37(26)
C6—C7—H7	119.98(21)	C24—C23—C22	121.90(26)
C7—C8—C3	122.29(21)	C24—C23—H23	119.07(27)
C7—C8—H8	118.90(22)	C22—C23—H23	119.03(25)
C3—C8—H8	118.81(21)	C10—C11—H11A	109.46(19)
O2—C9—O1	122.53(20)	C10—C11—H11B	109.42(21)
O2—C9—C6	124.55(20)	H11A—C11—H11B	109.52(23)
O1—C9—C6	112.93(18)	C10—C11—H11C	109.47(21)
O1—C10—C11	107.99(19)	H11A—C11—H11C	109.48(21)
O1—C10—H10A	110.08(20)	H11B—C11—H11C	109.49(22)
C11—C10—H10A	110.12(21)	C23—C24—C25	116.89(25)
O1—C10—H10B	110.11(20)	C23—C24—C27	122.17(25)
C11—C10—H10B	110.11(21)	C25—C24—C27	120.92(26)
H10A—C10—H10B	108.45(23)	C24—C25—C26	121.91(29)
C17—C12—C13	116.34(18)	C24—C25—H25	119.03(31)

C17—C12—C1	121.02(17)	C26—C25—H25	119.07(28)
C13—C12—C1	122.58(18)	C21—C26—C25	120.52(25)
C14—C13—C12	121.68(20)	C21—C26—H26	119.78(26)
C14—C13—H13	119.14(20)	C25—C26—H26	119.71(27)
C12—C13—H13	119.19(20)	C24—C27—H27A	109.52(28)
C15—C14—C13	121.69(20)	C24—C27—H27B	109.50(26)
C15—C14—H14	119.14(22)	H27A—C27—H27B	109.52(34)
C13—C14—H14	119.17(21)	C24—C27—H27C	109.44(31)
C14—C15—C16	117.01(19)	H27A—C27—H27C	109.41(31)
C14—C15—C18	122.07(19)	H27B—C27—H27C	109.43(28)