Supporting Information

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

Jean-Hubert Olivier, a Franck Camerel, a Raymond Ziessel, a,* Pascal Retailleau, b Julien Amadou, c Cuong Pham-Huu, c*

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 multi-walled carbon nanotubes

![XRD powder diffraction](Figure S1)

*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 formate (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 x 20 mL of dichloromethane. The combined organic phases were washed with 2 x 20 mL of distilled water and 1 x 20 mL of brine. The resulting organic phase was dried over MgSO₄ 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.

**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 %): ¹H NMR (300MHz, CDCl₃) : δ 7.33-7.18 (m, 5H), 3.66, (s, 3H), 2.96 (t, 2H, ³J = 11.2 Hz), 2.63 (t, 2H, ³J = 11.8 Hz), ¹³C NMR (75 MHz, CDCl₃): δ 173.38, 140.51, 128.89, 128.51, 128.26, 128.07, 1326.27, 51.58, 35.69, 30.95.

**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. Purification by flash chromatography on silica gel (20:80 dichloromethane:petroleum ether) afforded 2 as a yellow oil (90 mg, 99 %): ¹H NMR (300MHz, CDCl₃) : δ 7.31-7.26 (m, 2H), 7.22-7.17 (m, 3H), 4.06 (t, 2H, ³J = 9.6 Hz), 2.96 (t, 2H, ³J = 7.53 Hz), 2.63 (t, 2H, ³J = 7.33 Hz), 1.59 (t, 2H, ³J = 6.9 Hz), 1.27 (m, 14H), 0.89 (t, 3H, ³J = 6.7 Hz); ¹³C NMR (75
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 %): ¹H NMR (300MHz, CDCl₃) : δ 7.31-7.27 (m, 2H), 7.21-7.19 (m, 3H), 4.06 (t, 2H, 3J = 6.9 Hz), 2.96 (t, 2H, 3J = 7.5 Hz), 2.62 (t, 2H, 3J = 8.1 Hz), 1.59 (t, 2H, 3J = 6.6 Hz), 1.27 (m, 16H), 0.89 (t, 3H, 3J = 6.4 Hz); ¹³C NMR (75 MHz, CDCl₃): 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 C₁₉H₃₀O₂: C, 78.57; H, 10.41. Found C, 78.89; H, 10.75.

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 dichloromethane:petroleum ether) afforded 4 as a white solid (111 mg, 98 %): ¹H NMR (200MHz, CDCl₃) : δ 8.24-8.00 (m, 9H), 7.22-7.14 (m, 5H), 5.83 (s, 2H), 2.98 (t, 2H, 3J = 8.0 Hz), 2.75 (t, 2H, 3J = 7.8 Hz); ¹³C NMR (75MHz, CDCl₃): 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\textsubscript{2}O (1:1) and K\textsubscript{2}CO\textsubscript{3} (Table1). To a solution of aryl iodide (0.858 mmol) dissolved in 10 mL (\text{1/1, v/v}) of EtOH/H\textsubscript{2}O were added, 4-ethynyl toluene (2.145 mmol, 250 mg, 0.272 mL), K\textsubscript{2}CO\textsubscript{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\textsubscript{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.

**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): \textsuperscript{1}H NMR (300 MHz, CDCl\textsubscript{3}) : δ 7.94 (AB quartet, 4H, \text{AB}J = 9.2 Hz, νδ\text{AB} = 169.2 Hz), 7.33 (AB quartet, 4H, \text{AB}J = 8.1 Hz, νδ\text{AB} = 77.3 Hz), 2.42 (s, 3H); \textsuperscript{13}C NMR (75 MHz, CDCl\textsubscript{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\textsuperscript{-1}): 2985; 2853, 2289, 2211, 1992, 1590, 1519, 1405, 1368, 1340, 1275, 1260, 1173, 1138, 1106, 1010, 913, 864, 851, 839, 816.
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$_3$) : $\delta$ 7.26 (AB quartet, 4H, $^{4B}J = 9.0$ Hz, $\nu\delta_{AB} = 70.3$ Hz), 7.01 (AB quartet, 4H, $^{4B}J = 9.0$ Hz, $\nu\delta_{AB} = 244.9$ Hz), 3.32 (t, 4H, $^3J = 7.5$ Hz), 2.37 (s, 3H), 1.62-1.55 (m, 4H), 1.38 (dt, 4H, $^3J = 7.3$ Hz), 0.99 (t, 6H, $^3J = 7.2$ Hz); $^{13}$C NMR (75 MHz, CDCl$_3$) : $\delta$ 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.

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$_3$) : $\delta$ 7.16 (AB quartet, 4H, $^{4B}J = 9.2$ Hz, $\nu\delta_{AB} = 176.4$ Hz), 7.27 (AB quartet, 4H, $^{4B}J = 9.3$ Hz, $\nu\delta_{AB} = 176.4$ Hz), 3.82 (s, 3H), 2.86 (s, 3H); $^{13}$C NMR (75 MHz, CDCl$_3$) : $\delta$ 159.49, 137.98, 132.96, 131.32, 129.06, 120.51, 115.61, 113.96, 85.64, 88.64, 88.18, 55.29, 21.47; IR (KBr, cm$^{-1}$): 3004, 2917, 2947, 2928, 2838, 2665, 2444, 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.

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$_3$): $\delta$ 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$_3$): $\delta$ 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 (Table 1). 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 5 min (300 W, 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 $\times$ 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 $\times$ 20 mL of dichloromethane. The combined organic phases were washed with 2 $\times$ 20 mL of distilled water and 1 $\times$ 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](image)

**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$_3$): $\delta$ 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$_3$): $\delta$ 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 ($\lambda_{\text{max}}$ nm, $\varepsilon = \text{M}^{-1}\text{cm}^{-1}$), CH$_2$Cl$_2$: 251 (29800), 297 (18900), 380 (27400); IR (KBr, cm$^{-1}$): 3118, 3056, 3030, 2918, 2850, 2691, 2445, 2347, 2195, 2114, 1993, 1910, 1796,
$1747, 1671, 1591, 1576, 1563, 1506, 1417, 1376, 1338, 1277, 1261, 1184, 1176, 1107, 1041, 1017, 1004, 969, 882, 864, 842, 813; \text{MALDI-TOF } m/z \text{ (nature of the peak) } 353.1 \text{ ([M+H]$^+$, 100); Anal. Calcd for C}_{24}\text{H}_{19}\text{NO}_2 : C, 81.56; H, 5.42; N, 3.96. Found: C, 81.37; H, 5.28; N, 3.70.$
Figure S2. COSY (Top) and NOESY (Bottom) spectra of compound 8a measured in CDCl₃.
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$_3$) : $\delta$ 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, $^3J = 6.8$ Hz), 2.37 (s, 3H), 1.40 (t, 3H, $^3J = 6.9$ Hz); $^{13}$C NMR (75 MHz, CDCl$_3$): $\delta$ 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$_3$) : $\delta$ 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, $^3J = 6.8$ Hz), 2.41 (s, 6H), 1.40 (t, 3H, $^3J = 6.9$ Hz); $^{13}$C NMR (75 MHz, CDCl$_3$): $\delta$ 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, 126.11, 120.01, 98.31, 87.87, 80.94, 21.58, 21.19, 14.36; UV-vis ($\lambda_{max}$ nm, $\varepsilon$ = M$^{-1}$cm$^{-1}$), CH$_2$Cl$_2$: 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\(_3\)) : \(\delta\) 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\(_3\)) : \(\delta\) 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\(_3\)) : \(\delta\) 7.88 (AB quartet, 4H, \(^{AB}J = 6.9\) Hz, \(\nu\delta_{AB} = 124.3\) Hz), 7.47 (AB quartet, 4H, \(^{AB}J = 6.9\)Hz, \(\nu\delta_{AB} = 136.0\) 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\(_3\)) : \(\delta\) 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 (\(\lambda_{\text{max}}\) nm, \(\varepsilon = \text{M}^{-1}\text{cm}^{-1}\)), CH\(_2\)Cl\(_2\): 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. Caled 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.
6) X-ray experimental section.

The X-ray diffraction data were collected with MoKα radiation (λ=0.71073 Å, 2θmax=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\textsuperscript{[1]} and refined by full-matrix least-squares on F² with SHELXL-97.\textsuperscript{[1]} 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: C_{27}H_{24}O_{2}; Mr=380.46 g.mol\textsuperscript{-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) Å³, T=295 K, ρ_{calc}=1.181 g.cm\textsuperscript{-3}, crystal size 0.55 x 0.50 x 0.18 mm\textsuperscript{3}; absorption coefficient= 0.073 mm\textsuperscript{-1}, Z=2, F(000)=404; limiting indices: -6 ≤ h ≤ 6, -13 ≤ k ≤ 13, -17≤l≤17; reflections collected/unique: 22116/3082; R(int) = 0.0247; data/restraints/parameters: 3081 / 0 / 266, GOF on F²=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\textsuperscript{-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

<table>
<thead>
<tr>
<th>Formula sum</th>
<th>C_{27}H_{24}O_{2}</th>
</tr>
</thead>
<tbody>
<tr>
<td>Formula weight</td>
<td>380.46</td>
</tr>
<tr>
<td>Crystal system</td>
<td>triclinic</td>
</tr>
<tr>
<td>Space group</td>
<td>P -1 (no. 2)</td>
</tr>
<tr>
<td>Unit cell dimensions</td>
<td>a = 5.644(1) Å</td>
</tr>
<tr>
<td></td>
<td>b = 12.047(1) Å</td>
</tr>
<tr>
<td></td>
<td>c = 16.159(1) Å</td>
</tr>
<tr>
<td></td>
<td>α = 82.02(5) °</td>
</tr>
<tr>
<td></td>
<td>β = 82.48(5) °</td>
</tr>
<tr>
<td></td>
<td>γ = 81.90(4) °</td>
</tr>
<tr>
<td>Cell volume</td>
<td>1070.24(20) Å³</td>
</tr>
<tr>
<td>Z</td>
<td>2</td>
</tr>
<tr>
<td>Density, calculated</td>
<td>1.181 g/cm³</td>
</tr>
<tr>
<td>R_{All}</td>
<td>0.072</td>
</tr>
<tr>
<td>Pearson code</td>
<td>aP106</td>
</tr>
<tr>
<td>Formula type</td>
<td>N2O24P27</td>
</tr>
<tr>
<td>Wyckoff sequence</td>
<td>i\textsuperscript{33}</td>
</tr>
</tbody>
</table>

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

<table>
<thead>
<tr>
<th>Atom</th>
<th>Wyck.</th>
<th>x</th>
<th>y</th>
<th>z</th>
<th>U</th>
</tr>
</thead>
<tbody>
<tr>
<td>O1</td>
<td>2i</td>
<td>0.3314(3)</td>
<td>0.27326(11)</td>
<td>0.02914(9)</td>
<td></td>
</tr>
<tr>
<td>O2</td>
<td>2i</td>
<td>0.4340(3)</td>
<td>0.37680(14)</td>
<td>0.11883(12)</td>
<td></td>
</tr>
<tr>
<td>C1</td>
<td>2i</td>
<td>1.3142(3)</td>
<td>-0.10998(16)</td>
<td>0.23877(12)</td>
<td></td>
</tr>
<tr>
<td>C2</td>
<td>2i</td>
<td>1.1489(3)</td>
<td>-0.08999(16)</td>
<td>0.18312(12)</td>
<td></td>
</tr>
<tr>
<td>H2</td>
<td>2i</td>
<td>1.14400</td>
<td>-0.15010</td>
<td>0.15330</td>
<td>0.0810</td>
</tr>
<tr>
<td>C3</td>
<td>2i</td>
<td>0.9774(3)</td>
<td>0.00888(16)</td>
<td>0.16158(12)</td>
<td></td>
</tr>
<tr>
<td>C4</td>
<td>2i</td>
<td>0.9582(4)</td>
<td>0.11224(18)</td>
<td>0.19392(14)</td>
<td></td>
</tr>
<tr>
<td>H4</td>
<td>2i</td>
<td>1.06210</td>
<td>0.12130</td>
<td>0.23200</td>
<td>0.0930</td>
</tr>
</tbody>
</table>
### Anisotropic displacement parameters (in Å²)

<table>
<thead>
<tr>
<th>Atom</th>
<th>$U_{11}$</th>
<th>$U_{22}$</th>
<th>$U_{33}$</th>
<th>$U_{12}$</th>
<th>$U_{13}$</th>
<th>$U_{23}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>O1</td>
<td>0.0949(10)</td>
<td>0.0590(8)</td>
<td>0.0839(10)</td>
<td>0.0050(7)</td>
<td>-0.0334(8)</td>
<td>-0.0155(7)</td>
</tr>
<tr>
<td>O2</td>
<td>0.1460(15)</td>
<td>0.0778(11)</td>
<td>0.1318(14)</td>
<td>0.0262(10)</td>
<td>-0.0685(12)</td>
<td>-0.0474(10)</td>
</tr>
<tr>
<td>C1</td>
<td>0.0583(11)</td>
<td>0.0629(12)</td>
<td>0.0722(12)</td>
<td>-0.0136(9)</td>
<td>-0.0108(9)</td>
<td>-0.0086(9)</td>
</tr>
<tr>
<td>C2</td>
<td>0.0678(12)</td>
<td>0.0607(12)</td>
<td>0.0790(13)</td>
<td>-0.0097(10)</td>
<td>-0.0181(10)</td>
<td>-0.0145(10)</td>
</tr>
<tr>
<td>C3</td>
<td>0.0648(12)</td>
<td>0.0581(12)</td>
<td>0.0682(12)</td>
<td>-0.0093(9)</td>
<td>-0.0116(9)</td>
<td>-0.0080(9)</td>
</tr>
<tr>
<td>C4</td>
<td>0.0845(14)</td>
<td>0.0722(14)</td>
<td>0.0851(14)</td>
<td>-0.0063(11)</td>
<td>-0.0326(12)</td>
<td>-0.0196(11)</td>
</tr>
<tr>
<td>C5</td>
<td>0.0927(15)</td>
<td>0.0594(12)</td>
<td>0.0826(14)</td>
<td>-0.0001(11)</td>
<td>-0.0287(12)</td>
<td>-0.0198(10)</td>
</tr>
<tr>
<td>C6</td>
<td>0.0711(12)</td>
<td>0.0602(12)</td>
<td>0.0617(11)</td>
<td>-0.0057(9)</td>
<td>-0.0123(9)</td>
<td>-0.0100(9)</td>
</tr>
<tr>
<td>C7</td>
<td>0.0980(14)</td>
<td>0.0604(12)</td>
<td>0.0757(13)</td>
<td>-0.0051(11)</td>
<td>-0.0252(11)</td>
<td>-0.0134(10)</td>
</tr>
<tr>
<td>C8</td>
<td>0.0610(13)</td>
<td>0.0682(12)</td>
<td>0.0832(14)</td>
<td>-0.0025(9)</td>
<td>-0.0163(10)</td>
<td>-0.0126(10)</td>
</tr>
<tr>
<td>C9</td>
<td>0.0849(14)</td>
<td>0.0625(13)</td>
<td>0.0722(13)</td>
<td>-0.0036(11)</td>
<td>-0.0194(11)</td>
<td>-0.0169(10)</td>
</tr>
<tr>
<td>C10</td>
<td>0.1006(16)</td>
<td>0.0596(12)</td>
<td>0.0932(15)</td>
<td>-0.0112(11)</td>
<td>-0.0388(13)</td>
<td>-0.0114(11)</td>
</tr>
<tr>
<td>C11</td>
<td>0.0564(11)</td>
<td>0.0651(12)</td>
<td>0.0668(12)</td>
<td>-0.0128(9)</td>
<td>-0.0087(9)</td>
<td>-0.0091(9)</td>
</tr>
<tr>
<td>C12</td>
<td>0.0646(12)</td>
<td>0.0705(13)</td>
<td>0.0887(14)</td>
<td>-0.0081(10)</td>
<td>-0.0246(11)</td>
<td>-0.0178(11)</td>
</tr>
<tr>
<td>C13</td>
<td>0.0705(13)</td>
<td>0.0631(13)</td>
<td>0.0912(15)</td>
<td>-0.0111(11)</td>
<td>-0.0180(11)</td>
<td>-0.0167(10)</td>
</tr>
<tr>
<td>C14</td>
<td>0.0620(12)</td>
<td>0.0661(13)</td>
<td>0.0693(12)</td>
<td>-0.0053(10)</td>
<td>-0.0080(9)</td>
<td>-0.0069(10)</td>
</tr>
<tr>
<td>C15</td>
<td>0.0665(12)</td>
<td>0.0828(15)</td>
<td>0.0819(14)</td>
<td>-0.0033(11)</td>
<td>-0.0258(11)</td>
<td>-0.0158(11)</td>
</tr>
<tr>
<td>C16</td>
<td>0.0686(13)</td>
<td>0.0702(14)</td>
<td>0.0872(14)</td>
<td>-0.0093(10)</td>
<td>-0.0243(11)</td>
<td>-0.0199(11)</td>
</tr>
<tr>
<td>C17</td>
<td>0.0863(15)</td>
<td>0.0755(15)</td>
<td>0.1044(17)</td>
<td>0.0014(12)</td>
<td>-0.0191(13)</td>
<td>-0.0098(13)</td>
</tr>
<tr>
<td>C18</td>
<td>0.0648(12)</td>
<td>0.0643(12)</td>
<td>0.0748(13)</td>
<td>-0.0085(10)</td>
<td>-0.0156(10)</td>
<td>-0.0098(10)</td>
</tr>
<tr>
<td>C19</td>
<td>0.0676(12)</td>
<td>0.0646(12)</td>
<td>0.0738(13)</td>
<td>-0.0065(10)</td>
<td>-0.0196(10)</td>
<td>-0.0163(10)</td>
</tr>
<tr>
<td>C20</td>
<td>0.0658(12)</td>
<td>0.0702(13)</td>
<td>0.0632(12)</td>
<td>-0.0089(10)</td>
<td>-0.0169(9)</td>
<td>-0.0120(9)</td>
</tr>
<tr>
<td>C21</td>
<td>0.0866(15)</td>
<td>0.0845(16)</td>
<td>0.129(2)</td>
<td>0.0084(13)</td>
<td>-0.0470(14)</td>
<td>-0.0428(14)</td>
</tr>
<tr>
<td>C22</td>
<td>0.1231(14)</td>
<td>0.0845(16)</td>
<td>0.129(2)</td>
<td>0.0084(13)</td>
<td>-0.0470(14)</td>
<td>-0.0428(14)</td>
</tr>
</tbody>
</table>

This journal is (c) The Royal Society of Chemistry and The Centre National de la Recherche Scientifique, 2008.
**Selected geometric parameters (Å, °)**

<table>
<thead>
<tr>
<th>Bond/Angle</th>
<th>Distance/Angle</th>
</tr>
</thead>
<tbody>
<tr>
<td>O1—C9</td>
<td>1.332(28)</td>
</tr>
<tr>
<td>O1—C10</td>
<td>1.449(44)</td>
</tr>
<tr>
<td>C1—C2</td>
<td>1.354(33)</td>
</tr>
<tr>
<td>C1—C9</td>
<td>1.477(44)</td>
</tr>
<tr>
<td>C2—C3</td>
<td>1.387(30)</td>
</tr>
<tr>
<td>C3—C4</td>
<td>1.387(30)</td>
</tr>
<tr>
<td>C4—C5</td>
<td>1.387(30)</td>
</tr>
<tr>
<td>C5—C6</td>
<td>1.387(30)</td>
</tr>
<tr>
<td>C6—C7</td>
<td>1.387(30)</td>
</tr>
<tr>
<td>C7—C8</td>
<td>1.387(30)</td>
</tr>
<tr>
<td>C8—C9</td>
<td>1.387(30)</td>
</tr>
<tr>
<td>C9—O1</td>
<td>1.387(30)</td>
</tr>
<tr>
<td>C9—O2</td>
<td>1.387(30)</td>
</tr>
<tr>
<td>C2—C1</td>
<td>1.387(30)</td>
</tr>
<tr>
<td>C2—C3</td>
<td>1.387(30)</td>
</tr>
<tr>
<td>C3—C4</td>
<td>1.387(30)</td>
</tr>
<tr>
<td>C4—C5</td>
<td>1.387(30)</td>
</tr>
<tr>
<td>C5—C6</td>
<td>1.387(30)</td>
</tr>
<tr>
<td>C6—C7</td>
<td>1.387(30)</td>
</tr>
<tr>
<td>C7—C8</td>
<td>1.387(30)</td>
</tr>
<tr>
<td>C8—C9</td>
<td>1.387(30)</td>
</tr>
<tr>
<td>C9—O1</td>
<td>1.387(30)</td>
</tr>
<tr>
<td>C9—O2</td>
<td>1.387(30)</td>
</tr>
<tr>
<td>C10—C11</td>
<td>1.483(32)</td>
</tr>
<tr>
<td>C10—H10B</td>
<td>1.483(32)</td>
</tr>
<tr>
<td>C10—H10C</td>
<td>1.483(32)</td>
</tr>
<tr>
<td>C10—H10D</td>
<td>1.483(32)</td>
</tr>
</tbody>
</table>

**Notes:**
- All bond lengths are given in Ångstroms (Å).
- All angles are measured in degrees (°).
- The bond lengths and angles are rounded to two decimal places.
- The table includes all significant bonds, angles, and distances involved in the structure.
<table>
<thead>
<tr>
<th>C13—C12—C1</th>
<th>122.58(18)</th>
</tr>
</thead>
<tbody>
<tr>
<td>C14—C13—C12</td>
<td>121.68(20)</td>
</tr>
<tr>
<td>C14—C13—H13</td>
<td>119.14(20)</td>
</tr>
<tr>
<td>C12—C13—H13</td>
<td>119.19(20)</td>
</tr>
<tr>
<td>C15—C14—C13</td>
<td>121.69(20)</td>
</tr>
<tr>
<td>C15—C14—H14</td>
<td>119.14(22)</td>
</tr>
<tr>
<td>C13—C14—H14</td>
<td>119.17(21)</td>
</tr>
<tr>
<td>C14—C15—C16</td>
<td>117.01(19)</td>
</tr>
<tr>
<td>C14—C15—C18</td>
<td>122.07(19)</td>
</tr>
</tbody>
</table>