Palladium-Catalyzed Decarboxylative Aza-Michael Addition-Allylation Reactions Between Allyl Carbamates and Activated Olefins. Generation of Quaternary Carbon Adjacent to Secondary Amine Carbon Center

Nitin T. Patil, Zhibao Huo and Yoshinori Yamamoto*

Department of Chemistry, Graduate School of Science, Tohoku University, Sendai 980-8578, Japan

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(3e). ¹H NMR (400MHz, CDCl₃) δ 1.08 (br s, 3H), 2.58 (ddd, *J* = 14.0, 7.0, 1.0 Hz, 1H), 2.65 (ddd,

$J = 14.0, 7.0, 1.0$ Hz, 1H), 4.13 (q, $J = 7.0$ Hz, 2H), 5.26 (dt, $J = 17.0, 1.0$ Hz, 1H), 5.37 (dt, $J = 10.0, 1.0$ Hz, 1H), 5.95-5.83 (m, 2H), 6.89-6.84 (m, 2H), 7.35-7.20 (m, 8H). ^{13}C NMR (100 MHz, CDCl_3) δ 14.4, 41.2, 42.2, 62.9, 63.8, 114.3, 114.4, 123.6, 128.1, 128.7, 128.7, 128.8, 129.6, 129.7, 130.5, 133.1, 137.1, 156.4. IR (neat) 2981, 2248, 1699, 1596, 1456, 1292, 937, 700 cm^{-1} . ESI Calcd for $\text{C}_{22}\text{H}_{21}\text{N}_3\text{O}_2$ (M+Na) 382.1526. Found 382.1525.

(3g). ^1H NMR (400MHz, CDCl_3) δ 2.00-0.50 (m, 13H), 2.65 (ddd, $J = 14.0, 7.0, 1.0$ Hz, 1H), 2.78 (ddd, $J = 14.0, 7.0, 1.0$ Hz, 1H), 3.35 (br s, 1H), 4.22 (q, $J = 7.0$ Hz, 2H), 5.31 (dt, $J = 17.0, 1.0$ Hz, 1H), 5.36 (s, 1H), 5.39 (dt, $J = 10.0, 1.0$ Hz, 1H), 5.92 (ddt, $J = 17.0, 10.0, 1.0$ Hz, 1H), 7.57-7.32 (m, 5H). ^{13}C NMR (100 MHz, CDCl_3) δ 14.5, 25.3, 25.8, 26.2, 38.5, 38.9, 40.9, 62.1, 71.8, 123.5, 126.7, 128.7, 129.1, 129.4, 130.1, 137.2, 142.2, 167.1. IR (neat) 2933, 2250, 1693, 1606, 1440, 1288, 908, 702 cm^{-1} . ESI Calcd for $\text{C}_{22}\text{H}_{27}\text{N}_3\text{O}_2$ (M+Na) 388.1995. Found 388.1996.

(3h). ^1H NMR (400MHz, CDCl_3) δ 1.07 (t, $J = 7.0$ Hz, 3H), 2.56 (ddd, $J = 14.0, 7.0, 1.0$ Hz, 1H), 2.63 (ddd, $J = 14.0, 7.0, 1.0$ Hz, 1H), 3.75 (s, 3H), 4.09 (q, $J = 7.0$ Hz, 2H), 5.25 (dt, $J = 17.0, 1.0$ Hz, 1H), 5.36 (dt, $J = 10.0, 1.0$ Hz, 1H), 5.83 (s, 1H), 5.86 (ddt, $J = 17.0, 10.0, 1.0$ Hz, 1H), 6.77 (d, $J = 8.0$ Hz, 2H), 6.88 (d, $J = 8.0$ Hz, 1H), 6.95 (d, $J = 8.0$ Hz, 1H), 7.32-7.17 (m, 4H), 7.83 (d, $J = 8.0$ Hz, 1H). ^{13}C NMR (100 MHz, CDCl_3) δ 14.3, 41.1, 42.4, 55.2, 62.8, 63.3, 114.1, 114.4, 114.5, 115.1, 123.5, 124.9, 128.3, 128.7, 128.7, 130.7, 131.1, 133.4, 160.4. IR (neat) 2976, 2225, 1705, 1610, 1463, 1294, 1022, 755 cm^{-1} . ESI Calcd for $\text{C}_{23}\text{H}_{23}\text{N}_3\text{O}_3$ (M+Na) 412.1632. Found 412.1631.

(3i). ^1H NMR (400MHz, CDCl_3) δ 1.08 (t, $J = 7.0$ Hz, 3H), 2.62 (ddd, $J = 14.0, 7.0, 1.0$ Hz, 1H), 2.71 (ddd, $J = 14.0, 7.0, 1.0$ Hz, 1H), 4.13 (q, $J = 7.0$ Hz, 2H), 5.28 (dt, $J = 17.0, 1.0$ Hz, 1H), 5.37 (dt, $J = 10.0, 1.0$ Hz, 1H), 5.86 (ddt, $J = 17.0, 10.0, 1.0$ Hz, 1H), 6.14 (br s, 1H), 6.32 (m, 1H), 6.41 (d, $J = 3.0$ Hz, 1H), 6.89-6.81 (m, 2H), 7.38-7.19 (m, 4H). ^{13}C NMR (100 MHz, CDCl_3) δ 14.3, 40.6, 41.4, 57.6, 63.0, 110.9, 111.9, 113.6, 113.7, 123.8, 127.9, 128.8, 128.8, 129.9, 129.9, 143.2, 146.1, 155.4. IR (neat) 2977, 2254, 1708, 1596, 1444, 1298, 914, 732 cm^{-1} . ESI Calcd for $\text{C}_{20}\text{H}_{19}\text{N}_3\text{O}_3$ (M+Na) 372.1319. Found 372.1319.

(3j). ^1H NMR (400MHz, CDCl_3) δ 1.18 (t, $J = 7.0$ Hz, 3H), 1.36 (t, $J = 7.0$ Hz, 3H), 2.74 (ddd, $J = 14.0, 7.0, 1.0$ Hz, 1H), 2.79 (ddd, $J = 14.0, 7.0, 1.0$ Hz, 1H), 3.76 (dq, $J = 10.0, 7.0$ Hz, 1H), 4.01 (dq, $J = 10.0, 7.0$ Hz, 1H), 4.22 (dq, $J = 10.0, 7.0$ Hz, 2H), 5.37 (dt, $J = 17.0, 1.0$ Hz, 1H), 5.41 (dt, $J = 10.0, 1.0$ Hz, 1H), 5.87 (ddt, $J = 17.0, 10.0, 1.0$ Hz, 1H), 6.01 (br s, 1H), 7.44-7.29 (m, 5H). ^{13}C NMR (100 MHz, CDCl_3) δ 14.2, 14.5, 40.0, 42.3, 62.9, 65.3, 84.6, 111.7, 112.9, 123.3, 128.3, 129.0, 129.1, 130.1, 134.7, 150.6. IR (neat) 2981, 2254, 1710, 1596, 1464, 1301, 914, 732 cm^{-1} . ESI Calcd for $\text{C}_{18}\text{H}_{21}\text{N}_3\text{O}_3$ (M+Na) 350.1475. Found 350.1474.

(3k). ^1H NMR (400MHz, CDCl_3) δ 0.90 (t, $J = 7.0$ Hz, 3H), 1.61-1.05 (m, 9H), 2.70 (ddd, $J = 14.0,$

7.0, 1.0 Hz, 1H), 2.77 (ddd, $J = 14.0, 7.0, 1.0$ Hz, 1H), 4.17 (q, $J = 7.0$ Hz, 2H), 4.90 (br s, 1H), 5.37 (dt, $J = 17.0, 1.0$ Hz, 1H), 5.41 (dt, $J = 10.0, 1.0$ Hz, 1H), 5.91 (ddt, $J = 17.0, 10.0, 1.0$ Hz, 1H), 7.42-7.20 (m, 5H). ^{13}C NMR (100 MHz, CDCl_3) δ 13.8, 14.3, 15.2, 22.2, 28.4, 40.6, 43.0, 62.7, 65.8, 113.7, 114.1, 123.3, 128.5, 128.7, 128.9, 129.2, 129.6, 156.6. IR (neat) 2962, 2246, 1705, 1596, 1465, 1299, 937, 702 cm^{-1} . ESI Calcd for $\text{C}_{20}\text{H}_{25}\text{N}_3\text{O}_2$ (M+Na) 362.1839. Found 362.1837.

(3l). ^1H NMR (400MHz, CDCl_3) δ 1.11 (br s, 6H), 1.16 (t, $J = 7.0$ Hz, 3H), 2.84-2.59 (m, 2H), 3.02 (br s, 1H), 3.35 (br s, 1H), 4.20 (br s, 2H), 5.38-5.27 (m, 2H), 5.89 (br s, 1H), 7.36-7.18 (m, 5H). ^{13}C NMR (100 MHz, CDCl_3) δ 14.4, 21.6, 31.1, 42.5, 43.3, 62.6, 72.6, 114.5, 115.1, 123.3, 126.5, 126.8, 128.9, 129.0, 143.8, 154.9. IR (neat) 2977, 2246, 1705, 1596, 1465, 1208, 935, 698 cm^{-1} . ESI Calcd for $\text{C}_{19}\text{H}_{23}\text{N}_3\text{O}_2$ (M+Na) 348.1682. Found 348.1682.

(3m). ^1H NMR (400MHz, CDCl_3) δ 1.25 (s, 9H), 1.29 (t, $J = 7.0$ Hz, 3H), 2.92 (ddd, $J = 14.0, 7.0, 1.0$ Hz, 1H), 3.07 (ddd, $J = 14.0, 7.0, 1.0$ Hz, 1H), 3.75 (s, 1H), 4.33-4.19 (m, 2H), 5.45 (dt, $J = 17.0, 1.0$ Hz, 1H), 5.52 (dt, $J = 10.0, 1.0$ Hz, 1H), 6.10 (ddt, $J = 17.0, 10.0, 1.0$ Hz, 1H), 7.42-7.22 (m, 5H). ^{13}C NMR (100 MHz, CDCl_3) δ 14.4, 29.4, 40.7, 41.3, 43.8, 62.5, 74.5, 115.5, 116.2, 123.8, 126.6, 126.7, 128.8, 129.0, 144.9, 155.3. IR (KBr) 2981, 2246, 1693, 1596, 1471, 1270, 904, 696 cm^{-1} . ESI Calcd for $\text{C}_{20}\text{H}_{25}\text{N}_3\text{O}_2$ (M+Na) 362.1839. Found 362.1840.

(5a). ^1H NMR (400MHz, CDCl_3) δ 1.14 (t, $J = 7.0$ Hz, 3H), 2.22 (s, 3H), 2.70 (ddd, $J = 14.0, 7.0, 1.0$ Hz, 1H), 2.83 (ddd, $J = 14.0, 7.0, 1.0$ Hz, 1H), 4.14 (q, $J = 7.0$ Hz, 2H), 5.28 (dt, $J = 17.0, 1.0$ Hz, 1H), 5.32 (dt, $J = 10.0, 1.0$ Hz, 1H), 5.88 (ddt, $J = 17.0, 10.0, 1.0$ Hz, 1H), 6.56 (br s, 1H), 6.90 (d, $J = 8.0$ Hz, 1H), 7.40-7.15 (m, 7H). ^{13}C NMR (100 MHz, CDCl_3) δ 14.2, 20.2, 38.3, 52.4, 62.9, 85.9, 115.3, 117.3, 117.6, 122.3, 126.8, 127.9, 128.8, 128.8, 129.3, 130.3, 131.9, 137.9, 155.4, 156.3, 182.9. IR (neat) 3390, 2981, 2248, 1699, 1640, 1488, 1199, 935, 732 cm^{-1} . ESI Calcd for $\text{C}_{23}\text{H}_{22}\text{N}_2\text{O}_4$ (M+Na) 413.1472. Found 413.1473.

(5b). ^1H NMR (400MHz, CDCl_3) δ 1.09 (t, $J = 7.0$ Hz, 3H), 2.65 (ddd, $J = 14.0, 7.0, 1.0$ Hz, 1H), 2.75 (ddd, $J = 14.0, 7.0, 1.0$ Hz, 1H), 4.09 (q, $J = 7.0$ Hz, 2H), 5.24 (dt, $J = 17.0, 1.0$ Hz, 1H), 5.30 (dt, $J = 10.0, 1.0$ Hz, 1H), 5.82 (ddt, $J = 17.0, 10.0, 1.0$ Hz, 1H), 6.51 (br s, 1H), 6.87 (d, $J = 9.0$ Hz, 1H), 7.25-7.13 (m, 5H), 7.52 (dd, $J = 9.0, 2.0$ Hz, 1H), 7.66 (br s, 1H). ^{13}C NMR (100 MHz, CDCl_3) δ 14.2, 38.5, 52.5, 63.2, 86.4, 114.9, 119.2, 119.5, 122.8, 128.9, 128.9, 129.0, 129.0, 129.5, 130.1, 136.4, 139.5, 155.3, 157.1, 181.6. IR (KBr) 3360, 2983, 2254, 1701, 1598, 1465, 1286, 1020, 929 cm^{-1} . ESI Calcd for $\text{C}_{22}\text{H}_{19}\text{BrN}_2\text{O}_4$ (M+Na) 477.0420. Found 477.0421.

(5c). ^1H NMR (400MHz, CDCl_3) δ 1.16 (t, $J = 7.0$ Hz, 3H), 2.72 (ddd, $J = 14.0, 7.0, 1.0$ Hz, 1H), 2.80 (ddd, $J = 14.0, 7.0, 1.0$ Hz, 1H), 4.16 (q, $J = 7.0$ Hz, 2H), 5.32 (dt, $J = 17.0, 1.0$ Hz, 1H), 5.38 (dt, $J = 10.0, 1.0$ Hz, 1H), 5.89 (ddt, $J = 17.0, 10.0, 1.0$ Hz, 1H), 6.60 (br s, 1H), 7.06 (t, $J = 7.0$ Hz, 1H),

7.40-7.25 (m, 4H), 7.57-7.52 (m, 2H). ^{13}C NMR (100 MHz, CDCl_3) δ 14.1, 38.5, 52.6, 63.3, 87.2, 114.6, 118.6, 119.6, 123.1, 123.5, 125.1, 127.5, 128.6, 128.9, 129.1, 129.2, 136.2, 152.3, 155.1, 181.1. IR (neat) 3348, 2983, 2252, 1716, 1645, 1456, 1298, 1060 cm^{-1} . ESI Calcd for $\text{C}_{22}\text{H}_{18}\text{Cl}_2\text{N}_2\text{O}_4$ (M+Na) 467.0536. Found 467.0538.

(5d). ^1H NMR (400MHz, CDCl_3) δ 1.16 (t, $J = 7.0$ Hz, 3H), 2.73 (ddd, $J = 14.0, 7.0, 1.0$ Hz, 1H), 2.86 (ddd, $J = 14.0, 7.0, 1.0$ Hz, 1H), 4.17 (q, $J = 7.0$ Hz, 2H), 5.30 (dt, $J = 17.0, 1.0$ Hz, 1H), 5.36 (dt, $J = 10.0, 1.0$ Hz, 1H), 5.91 (ddt, $J = 17.0, 10.0, 1.0$ Hz, 1H), 6.65 (br s, 1H), 6.95 (t, $J = 8.0$ Hz, 1H), 7.02 (d, $J = 8.0$ Hz, 1H), 7.30-7.15 (m, 5H), 7.50 (t, $J = 8.0$ Hz, 1H), 7.60 (d, $J = 8.0$ Hz, 1H). ^{13}C NMR (100 MHz, CDCl_3) δ 14.2, 38.4, 52.6, 63.0, 85.9, 115.2, 117.4, 117.9, 122.3, 122.4, 127.3, 128.8, 128.9, 129.2, 130.3, 136.9, 136.9, 155.3, 158.2, 182.7. IR (KBr) 3332, 2981, 2248, 1699, 1606, 1461, 1292, 985, 702 cm^{-1} . ESI Calcd for $\text{C}_{22}\text{H}_{20}\text{N}_2\text{O}_4$ (M+Na) 399.1315. Found 399.1316.

(5e). ^1H NMR (400MHz, CDCl_3) δ 1.07 (t, $J = 7.0$ Hz, 3H), 2.57 (ddd, $J = 14.0, 7.0, 1.0$ Hz, 1H), 2.96 (ddd, $J = 14.0, 7.0, 1.0$ Hz, 1H), 4.04 (q, $J = 7.0$ Hz, 2H), 5.16 (dt, $J = 10.0, 1.0$ Hz, 1H), 5.17 (dt, $J = 17.0, 1.0$ Hz, 1H), 5.71 (ddt, $J = 17.0, 10.0, 1.0$ Hz, 1H), 6.24 (br s, 1H), 7.10 (t, $J = 8.0$ Hz, 2H), 7.37-7.28 (m, 5H), 7.56 (t, $J = 8.0$ Hz, 1H), 7.91 (d, $J = 8.0$ Hz, 1H), 9.65 (br s, 1H). ^{13}C NMR (100 MHz, CDCl_3) δ 14.2, 35.6, 62.7, 62.7, 89.8, 118.3, 120.7, 120.9, 122.5, 127.5, 128.7, 129.1, 129.4, 130.5, 136.3, 139.3, 155.4, 158.5, 187.6, 194.9. IR (neat) 3363, 2981, 1716, 1606, 1463, 1218, 810 cm^{-1} . ESI Calcd for $\text{C}_{22}\text{H}_{21}\text{NO}_5$ (M+Na) 402.1312. Found 402.1313.

(5f). ^1H NMR (400MHz, CDCl_3) δ 1.03 (t, $J = 7.0$ Hz, 3H), 2.66 (ddd, $J = 14.0, 7.0, 1.0$ Hz, 1H), 2.84 (ddd, $J = 14.0, 7.0, 1.0$ Hz, 1H), 3.99 (q, $J = 7.0$ Hz, 2H), 5.17 (dt, $J = 17.0, 1.0$ Hz, 1H), 5.18 (dt, $J = 10.0, 1.0$ Hz, 1H), 5.67 (ddt, $J = 17.0, 10.0, 1.0$ Hz, 1H), 6.09 (br s, 1H), 7.43-7.34 (m, 5H), 7.90 (dd, $J = 7.0, 3.0$ Hz, 1H), 7.95 (dd, $J = 7.0, 3.0$ Hz, 1H), 9.72 (br s, 1H). ^{13}C NMR (100 MHz, CDCl_3) δ 14.5, 21.0, 29.3, 63.1, 96.7, 117.1, 118.2, 118.6, 121.9, 123.7, 128.6, 128.9, 129.1, 129.2, 129.5, 133.3, 152.8, 156.0, 185.1, 185.8. IR (KBr) 3404, 3082, 2868, 1710, 1625, 1465, 1244, 867, 696 cm^{-1} . ESI Calcd for $\text{C}_{22}\text{H}_{19}\text{FN}_2\text{O}_7$ (M+Na) 465.1069. Found 465.1067.

(5g). ^1H NMR (400MHz, CDCl_3) δ 1.12 (t, $J = 7.0$ Hz, 3H), 2.42 (ddd, $J = 14.0, 7.0, 1.0$ Hz, 1H), 2.68 (ddd, $J = 14.0, 7.0, 1.0$ Hz, 1H), 4.20-4.07 (m, 2H), 5.13 (dt, $J = 17.0, 1.0$ Hz, 1H), 5.37 (dt, $J = 10.0, 1.0$ Hz, 1H), 5.94 (ddt, $J = 17.0, 10.0, 1.0$ Hz, 1H), 6.97 (br s, 1H), 7.45-7.24 (m, 9H). ^{13}C NMR (100 MHz, CDCl_3) δ 14.2, 38.9, 50.4, 50.4, 62.7, 115.9, 116.6, 116.9, 122.9, 125.7, 125.9, 128.5, 129.1, 129.1, 129.5, 131.2, 150.6, 156.3, 161.3, 190.1. IR (KBr) 3527, 2991, 2252, 1705, 1639, 1452, 1112, 904, 765 cm^{-1} . ESI Calcd for $\text{C}_{22}\text{H}_{20}\text{N}_2\text{O}_4$ (M+Na) 399.1315. Found 399.1317.

(5h). ^1H NMR (400MHz, CDCl_3) δ 1.20 (t, $J = 7.0$ Hz, 3H), 2.70-2.44 (m, 6H), 4.11 (q, $J = 7.0$ Hz, 2H), 5.11 (dt, $J = 17.0, 1.0$ Hz, 1H), 5.14 (dt, $J = 10.0, 1.0$ Hz, 1H), 5.74-5.59 (m, 2H), 6.02-5.96 (m,

1H), 6.45 (br s, 1H), 6.94 (t, $J = 7.0$ Hz, 1H), 7.59-7.16 (m, 4H). ^{13}C NMR (100 MHz, CDCl_3) δ 14.5, 26.1, 35.8, 41.4, 49.3, 61.1, 61.2, 63.1, 118.7, 123.2, 128.5, 128.9, 130.7, 131.9, 137.9, 153.6, 182.1. IR (neat) 3328, 2981, 2236, 1722, 1598, 1444, 1224, 698 cm^{-1} . ESI Calcd for $\text{C}_{19}\text{H}_{22}\text{N}_2\text{O}_3$ (M+Na) 349.1523. Found 349.1525.

(**5j**). ^1H NMR (400MHz, CDCl_3) δ 1.04-1.45 (m, 13H), 2.67 (br s, 1H), 2.84-2.75 (m, 1H), 3.47 (br s, 1H), 4.10 (q, $J = 7.0$ Hz, 2H), 4.50 (br s, 1H), 5.44-5.28 (m, 2H), 6.05-5.91 (m, 1H), 6.94 (br s, 1H), 7.13-7.04 (m, 1H), 7.52 (br s, 1H), 7.98-7.90 (m, 1H). ^{13}C NMR (100 MHz, CDCl_3) δ 14.2, 14.6, 24.8, 25.4, 25.9, 33.4, 60.3, 62.2, 85.8, 115.6, 118.1, 118.9, 121.9, 122.1, 123.1, 127.7, 130.3, 132.1, 156.9, 175.6. IR (neat) 3332, 2979, 2256, 1699, 1608, 1468, 1228, 1058 cm^{-1} . ESI Calcd for $\text{C}_{22}\text{H}_{26}\text{N}_2\text{O}_4$ (M+Na) 405.1785. Found 405.1787.

(**5k**). ^1H NMR (400MHz, CDCl_3) δ 1.30-1.17 (m, 3H), 2.71-2.54 (m, 1H), 2.91-2.77 (m, 1H), 3.11 (s, 3H), 4.15 (q, $J = 7.0$ Hz, 2H), 5.18 (dt, $J = 17.0, 1.0$ Hz, 1H), 5.19 (dt, $J = 10.0, 1.0$ Hz, 1H), 5.82 (ddt, $J = 17.0, 10.0, 1.0$ Hz, 1H), 6.15 (br s, 1H), 7.01 (d, $J = 8.0$ Hz, 1H), 7.07 (t, $J = 8.0$ Hz, 1H), 7.53 (t, $J = 8.0$ Hz, 1H), 7.86 (d, $J = 8.0$ Hz, 1H). ^{13}C NMR (100 MHz, CDCl_3) δ 14.5, 30.0, 30.1, 50.8, 62.9, 85.3, 115.5, 117.9, 118.1, 122.9, 128.4, 128.4, 129.5, 137.5, 159.4, 165.6, 183.8. IR (neat) 3390, 2981, 2235, 1705, 1608, 1463, 1222, 931, 765 cm^{-1} . ESI Calcd for $\text{C}_{17}\text{H}_{18}\text{N}_2\text{O}_4$ (M+Na) 337.1159. Found 337.1159.

(**5l**). ^1H NMR (400MHz, CDCl_3) δ 1.09 (t, $J = 7.0$ Hz, 3H), 2.19 (s, 3H), 2.65 (ddd, $J = 14.0, 7.0, 1.0$ Hz, 1H), 2.78 (ddd, $J = 14.0, 7.0, 1.0$ Hz, 1H), 4.09 (q, $J = 7.0$ Hz, 2H), 5.23 (dt, $J = 17.0, 1.0$ Hz, 1H), 5.28 (dt, $J = 10.0, 1.0$ Hz, 1H), 5.83 (ddt, $J = 17.0, 10.0, 1.0$ Hz, 1H), 6.56 (br s, 1H), 6.88 (t, $J = 8.0$ Hz, 2H), 6.94 (d, $J = 8.0$ Hz, 2H), 7.05 (br s, 2H), 7.43 (t, $J = 8.0$ Hz, 1H), 7.53 (d, $J = 8.0$ Hz, 1H). ^{13}C NMR (100 MHz, CDCl_3) δ 14.3, 21.0, 38.3, 52.5, 62.9, 85.9, 115.2, 117.5, 117.9, 122.1, 122.4, 127.3, 129.3, 129.5, 130.0, 131.1, 136.9, 138.9, 158.3, 161.6, 182.8. IR (KBr) 3386, 2981, 2248, 1710, 1606, 1463, 1290, 1016, 777, 657 cm^{-1} . ESI Calcd for $\text{C}_{23}\text{H}_{22}\text{N}_2\text{O}_4$ (M+Na) 413.1472. Found 413.1471.

(**5m**). ^1H NMR (400MHz, CDCl_3) δ 1.17 (t, $J = 7.0$ Hz, 3H), 2.72 (ddd, $J = 14.0, 7.0, 1.0$ Hz, 1H), 2.86 (ddd, $J = 14.0, 7.0, 1.0$ Hz, 1H), 3.73 (br s, 3H), 4.16 (q, $J = 7.0$ Hz, 2H), 5.30 (dt, $J = 17.0, 1.0$ Hz, 1H), 5.36 (dt, $J = 10.0, 1.0$ Hz, 1H), 5.91 (ddt, $J = 17.0, 10.0, 1.0$ Hz, 1H), 6.67 (br s, 3H), 6.95 (t, $J = 8.0$ Hz, 1H), 7.00 (d, $J = 8.0$ Hz, 1H), 7.14 (br s, 2H), 7.50 (t, $J = 8.0$ Hz, 1H), 7.59 (d, $J = 8.0$ Hz, 1H). ^{13}C NMR (100 MHz, CDCl_3) δ 14.2, 38.3, 52.6, 55.3, 62.9, 85.9, 113.9, 115.3, 115.3, 117.4, 117.9, 122.2, 122.4, 127.3, 128.4, 129.2, 131.6, 136.9, 158.2, 159.6, 182.8. IR (neat) 3388, 2981, 2250, 1716, 1643, 1456, 1288, 753 cm^{-1} . ESI Calcd for $\text{C}_{23}\text{H}_{22}\text{N}_2\text{O}_5$ (M+Na) 429.1421. Found 429.1420.

(**5n**). ^1H NMR (400MHz, CDCl_3) δ 2.48 (ddd, $J = 14.0, 7.0, 1.0$ Hz, 1H), 2.59 (ddd, $J = 14.0, 7.0, 1.0$ Hz, 1H), 5.03 (dt, $J = 17.0, 1.0$ Hz, 1H), 5.10 (dt, $J = 10.0, 1.0$ Hz, 1H), 5.35 (d, $J = 10.0$ Hz, 1H), 5.61 (dd, $J = 17.0, 10.0$ Hz, 1H), 5.66 (ddt, $J = 17.0, 10.0, 1.0$ Hz, 1H), 6.20 (d, $J = 17.0$ Hz, 1H), 6.66 (t, $J = 8.0$ Hz, 1H), 6.71 (d, $J = 8.0$ Hz, 1H), 7.02-6.97 (m, 6H), 7.21 (t, $J = 8.0$ Hz, 1H), 7.31 (d, $J = 8.0$ Hz, 1H). ^{13}C NMR (100 MHz, CDCl_3) δ 38.7, 52.5, 90.9, 115.3, 117.4, 117.9, 122.3, 122.6, 122.6, 126.9, 127.3, 129.2, 129.6, 129.7, 130.8, 130.8, 136.9, 158.3, 166.4, 182.9. IR (KBr) 3350, 2983, 2245, 1676, 1604, 1463, 1197, 929, 707 cm^{-1} . ESI Calcd for $\text{C}_{22}\text{H}_{18}\text{N}_2\text{O}_3$ (M+Na) 381.1210. Found 381.1211.

C:\Documents and Settings\All Users\Documents\DATA\521\huo\huo636.als
huo636

DFILE C:\Documents and Settings\All Users\Documents\DATA\521\huo\huo636.als
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DATIM Wed Apr 13 17:55:55 2005
ORNUC 1H
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OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 7993.60 Hz
SCANS 5
ACQTM 4.0993 sec
PD 2.9010 sec
PWI 5.50 usec
IRNUC 1H
CTEMP 21.5 °C
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.30 Hz
RGAIN 22

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huo722

DFILE C:\Documents and Settings\All Users\Documents\DATA\521\huo\huo722a.als
COMNT huo722
DATIM Mon Mar 20 19:58:37 2006
ORNUC 1H
EXMOD NON
OBFRO 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 7993.60 Hz
SCANS 8
ACQTM 4.0993 sec
PD 2.9010 sec
PWI 5.50 usec
IRNUC 1H
CTEMP 23.6 °C
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 18

C:\Documents and Settings\All Users\Documents\DATA\521\huo\huo679.als
huo679

DFILE C:\Documents and Settings\All Users\Documents\DATA\521\huo\huo679.als
COMPT huo679
DATIM Mon Mar 20 15:40:00 2006
ORNUC 1H
EXMOD NON
ORFRO 399.65 MHz
ORSET 124.00 KHz
ORFIN 10500.00 Hz
POINT 32768
FREOU 7993.60 Hz
SCANS 8
ACQTM 4.0993 sec
PD 2.9010 sec
PW1 5.50 usec
IRNUC 1H
CTEMP 23.1 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.10 Hz
RGAIN 16

C:\Documents and Settings\All Users\Documents\DATA\521\huo\huo685.als
huo685

DFILE C:\Documents and Settings\All Users\Documents\DATA\521\huo\huo685.als
COMPT huo685
DATIM Mon Mar 20 19:09:40 2006
ORNUC 1H
EXMOD NON
ORFRO 399.65 MHz
ORSET 124.00 KHz
ORFIN 10500.00 Hz
POINT 32768
FREOU 7993.60 Hz
SCANS 8
ACQTM 4.0993 sec
PD 2.9010 sec
PW1 5.50 usec
IRNUC 1H
CTEMP 23.3 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 19

C:\Documents and Settings\All Users\Documents\DATA\521\huo\huo684.als
huo684

DFILE C:\Documents and Settings\All Users\
COMNT huo684
DATIM Wed May 25 12:10:13 2005
DENUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 7993.60 Hz
SCANS 8
AQTM 4.0993 sec
PD 2.9010 sec
PW1 5.50 usec
IRNUC 1H
CTEMP 23.2 c
SLVNT CDCL3
EXREF 7.24 ppm
BF 0.10 Hz
RGAIN 19

C:\Documents and Settings\All Users\Documents\DATA\521\huo\huo687-2.als
huo687-2

DFILE C:\Documents and Settings\All Users\
COMNT huo687-2
DATIM Fri May 27 14:19:34 2005
DENUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 7993.60 Hz
SCANS 5
AQTM 4.0993 sec
PD 2.9010 sec
PW1 5.50 usec
IRNUC 1H
CTEMP 23.3 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 18

C:\Documents and Settings\All Users\Documents\DATA\521\huo\huo686.als
huo686

DFILE C:\Documents and Settings\All Users\Documents\DATA\521\huo\huo686.als
COMPT huo686
DATIM Fri May 27 12:42:31 2005
OBNUC 1H
EXMOD NON
OBFRO 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQ 7993.60 Hz
SCANS 8
ACQTM 4.0993 sec
PD 2.9010 sec
PWI 5.50 usec
IRNUC 1H
CTEMP 23.5 c
SLVNT CDCL3
EXREF 7.24 ppm
BF 0.12 Hz
RGAIN 16

C:\Documents and Settings\All Users\Documents\DATA\521\huo\huo680-2.als
huo680-2

DFILE C:\Documents and Settings\All Users\Documents\DATA\521\huo\huo680-2.als
COMPT huo680-2
DATIM Fri May 20 14:59:30 2005
OBNUC 1H
EXMOD NON
OBFRO 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQ 7993.60 Hz
SCANS 5
ACQTM 4.0993 sec
PD 2.9010 sec
PWI 5.50 usec
IRNUC 1H
CTEMP 23.3 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.80 Hz
RGAIN 21

C:\Documents and Settings\All Users\Documents\DATA\521\huo\huo699.als
huo699

DFILE C:\Documents and Settings\All Users\Documents\DATA\521\huo\huo699.als
COMNT huo699
DATIM Tue Jul 05 11:16:32 2006
ORNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 7993.60 Hz
SCANS 6
ACQTM 4.0993 sec
PD 2.9010 sec
PWI 5.50 usec
IRNUC 1H
CTEMP 26.8 c
SLVNT CDCL3
EXREF 7.24 ppm
BF 0.12 Hz
RGAIN 17

C:\Documents and Settings\All Users\Documents\DATA\521\huo\huo717-ck.als
huo717

DFILE C:\Documents and Settings\All Users\Documents\DATA\521\huo\huo717-ck.als
COMNT huo717
DATIM Thu Jan 19 22:32:27 2006
ORNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 7993.60 Hz
SCANS 8
ACQTM 4.0993 sec
PD 2.9010 sec
PWI 5.50 usec
IRNUC 1H
CTEMP 25.3 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 1.20 Hz
RGAIN 18

C:\Documents and Settings\All Users\Documents\DATA\521\Who\Who719.xls
huo719

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C:\Documents and Settings\All Users\ND
FILE      huo719
COUNT    Tue Jul 05 21:24:48 2005
DATE
OBNUC     1H
EXMOD     NON
OBFREQ     399.65 MHz
OBSET      124.00 KHz
OBFIN      10500.00 Hz
POINT      32768
FREQU      7993.60 Hz
SCANS      5
ACQTH      4.0893 sec
PD          2.9010 sec
PW1        5.50 usec
IRNUC      1H
CTEMP      26.5 c
SLVNT      CDCL3
EXREF      7.26 ppm
BF          0.10 Hz
RGAIN      20

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C:\Documents and Settings\All Users\Documents\DATA\521\huo\huo710.xls
huo710

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C:\Documents and Settings\All Users\W
DFILE      huo710
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DBNUC      1H
EXMOD      NON
OBFREQ     399.65 MHz
ORSET      124.00 kHz
OBF-IN     10500.00 Hz
POINT      32768
FREQU      7993.60 Hz
SCANS      6
ACQTM      4.0993 sec
PD         2.8010 sec
PR1        5.50 usec
IRNUC      1H
CTEMP      26.5 c
SLVNT      CDCL3
XREF      7.26 ppm
BF         0.10 Hz
RGAIN      17

```

C:\Documents and Settings\All Users\Documents\DATA\521\huo\huo718.als
huo718

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CONV huo718
DATIM Mon Jul 04 19:55:34 2005
OBNUC 1H
EXMOD NON
OBFRO 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQ 7993.60 Hz
SCANS 6
ACQTM 4.0993 sec
PD 2.9010 sec
PW1 5.50 usec
IRNUC 1H
CTEMP 26.6 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 22

C:\Documents and Settings\All Users\Documents\DATA\521\huo\huo724-gp1-ok.als
huo724-gp1-ok

DFILE C:\Documents and Settings\All Users\Documents\DATA\521\huo\huo724-gp1-ok.als
CONV huo724-gp1-ok
DATIM Fri Feb 03 18:53:04 2006
OBNUC 1H
EXMOD NON
OBFRO 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQ 7993.60 Hz
SCANS 8
ACQTM 4.0993 sec
PD 2.9010 sec
PW1 5.50 usec
IRNUC 1H
CTEMP 25.3 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 22

C:\Documents and Settings\All Users\Documents\DATA\521\huo\huo727.als
huo727

DFILE C:\Documents and Settings\All Users\Documents\DATA\521\huo\huo727.als
COMNT huo727
DATIM Fri Jan 20 19:23:58 2006
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 kHz
OBFIN 10500.00 Hz
POINT 32768
FREQ 7993.60 Hz
SCANS 8
ACQTM 4.0993 sec
PD 2.9010 sec
PWI 5.50 usec
IRNUC 1H
CTEMP 24.5 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 18

C:\Documents and Settings\All Users\Documents\DATA\521\huo\huo707-crude.als
huo707-crude

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DATIM Fri Jun 17 12:33:43 2005
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 kHz
OBFIN 10500.00 Hz
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SCANS 8
ACQTM 4.0993 sec
PD 2.9010 sec
PWI 5.50 usec
IRNUC 1H
CTEMP 24.7 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.10 Hz
RGAIN 19

C:\Documents and Settings\All Users\Documents\DATAN521\huo728-again.als
huo728

DFILE C:\Documents and Settings\All Users\Documents\DATAN521\huo728-again.als
COMNT huo728
DATIM Mon Mar 20 14:04:07 2006
OBRUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 32768
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SCANS 8
ACQTM 4.0993 sec
PD 2.9010 sec
PWT 5.50 usec
IRNUC 1H
CTEMP 23.3 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.10 Hz
RGAIN 16

C:\Documents and Settings\All Users\Documents\DATAN521\huo742.als
huo742

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DATIM Sat Jan 21 13:58:58 2006
OBRUC 1H
EXMOD NON
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OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 32768
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SCANS 8
ACQTM 4.0993 sec
PD 2.9010 sec
PWT 5.50 usec
IRNUC 1H
CTEMP 24.5 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 20

C:\Documents and Settings\All Users\Documents\DATA\521\huo\huo815.als
huo815

DFILE C:\Documents and Settings\All Users\Documents\DATA\521\huo\huo815.als
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DATIM Sat Jan 21 12:55:31 2006
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQ 7993.60 Hz
SCANS 8
ACQTM 4.0993 sec
PD 2.9010 sec
PW1 5.50 usec
IRNUC 1H
CTEMP 24.5 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 18

C:\Documents and Settings\All Users\Documents\DATA\521\huo\huo814.als
huo814

DFILE C:\Documents and Settings\All Users\Documents\DATA\521\huo\huo814.als
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DATIM Fri Jan 20 21:50:59 2006
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EXMOD NON
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OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQ 7993.60 Hz
SCANS 8
ACQTM 4.0993 sec
PD 2.9010 sec
PW1 5.50 usec
IRNUC 1H
CTEMP 24.4 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 1.20 Hz
RGAIN 18

C:\Documents and Settings\VA11 Users\Documents\DATA\521\huo\huo778.als
huo788

DFILE C:\Documents and Settings\VA11 Users\Documents\DATA\521\huo\huo778.als
CONNT hu0788
DATIM Mon Jan 23 18:08:30 2006
ORNUC 1H
EXMOD NON
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ORSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 7993.60 Hz
SCANS 8
AQTM 4.0993 sec
PD 2.9010 sec
PW1 5.50 usec
IRNUC 1H
CTEMP 24.7 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 25

X-Ray Structure of **5n**

Experimental Details

A. Crystal Data

Empirical Formula	C ₂₂ H ₁₈ N ₂ O ₃
Formula Weight	358.40
Crystal Color, Habit	colorless, prism
Crystal Dimensions	0.20 X 0.20 X 0.20 mm
Crystal System	orthorhombic
Lattice Type	Primitive

Detector Position	50.96 mm
Pixel Size	0.137 mm
Lattice Parameters	a = 15.706(5) Å b = 8.930(3) Å c = 12.969(5) Å V = 1818.8(11) Å ³
Space Group	Pna2 ₁ (#33)
Z value	4
D _{calc}	1.309 g/cm ³
F ₀₀₀	752.00
μ(MoKα)	0.881 cm ⁻¹

B. Intensity Measurements

Detector	Rigaku Saturn
Goniometer	Rigaku AFC10
Radiation	MoKα (λ = 0.71070 Å) graphite monochromated
Detector Aperture	70 mm x 70 mm
Data Images	1800 exposures
ω oscillation Range (χ=45.0, φ=0.0)	-110.0 - 70.0°
Exposure Rate	40.0 sec./°
Detector Swing Angle	-19.65°

ω oscillation Range ($\chi=45.0$, $\phi=90.0$)	-110.0 - 70.0°
Exposure Rate	40.0 sec./°
Detector Swing Angle	-19.65°
ω oscillation Range ($\chi=45.0$, $\phi=180.0$)	-110.0 - 70.0°
Exposure Rate	40.0 sec./°
Detector Swing Angle	-19.65°
ω oscillation Range ($\chi=45.0$, $\phi=270.0$)	-110.0 - 70.0°
Exposure Rate	40.0 sec./°
Detector Swing Angle	-19.65°
Detector Position	50.96 mm
Pixel Size	0.137 mm
$2\theta_{\max}$	54.9°
No. of Reflections Measured	Total: 23884 Unique: 2026 ($R_{\text{int}} = 0.045$)
Corrections	Lorentz polarization Absorption (trans. factors: 0.836 - 0.983)

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SIR97)
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Refinement	Full-matrix least-squares on F^2
Function Minimized	$\sum w (F_o^2 - F_c^2)^2$
Least Squares Weights	$1/[1.0000\sigma(F_o^2)]/(4F_o^2)$
$2\theta_{\max}$ cutoff	54.9°
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (All reflections)	2026
No. Variables	262
Reflection/Parameter Ratio	7.73
Residuals: R1 ($I > 2.00\sigma(I)$)	0.0264
Residuals: R (All reflections)	0.0297
Residuals: wR2 (All reflections)	0.0595
Goodness of Fit Indicator	0.994
Max Shift/Error in Final Cycle	0.000
Maximum peak in Final Diff. Map	$0.17 \text{ e}^-/\text{\AA}^3$
Minimum peak in Final Diff. Map	$-0.16 \text{ e}^-/\text{\AA}^3$

Table 1. Atomic coordinates and $B_{\text{iso}}/B_{\text{eq}}$

atom	x	y	z	B_{eq}
O(1)	0.97423(8)	-0.02305(12)	0.31643(9)	2.06(2)
O(2)	0.97875(6)	0.28712(12)	0.08122(9)	1.80(2)
O(3)	0.83751(8)	0.19045(14)	-0.10286(9)	2.44(2)

N(1)	0.84498(8)	0.15837(13)	0.07136(10)	1.53(2)
N(2)	0.89265(10)	-0.22605(16)	0.10636(12)	2.33(3)
C(1)	0.97710(10)	0.02367(17)	0.13307(12)	1.39(2)
C(2)	0.97950(10)	0.06919(19)	0.24791(12)	1.49(3)
C(3)	0.99492(12)	0.22972(18)	0.26454(12)	1.59(3)
C(4)	1.01022(12)	0.2852(2)	0.36419(12)	2.02(3)
C(5)	1.02715(12)	0.4355(2)	0.37964(13)	2.45(4)
C(6)	1.02902(12)	0.5317(2)	0.29542(14)	2.43(3)
C(7)	1.01350(12)	0.48045(19)	0.19671(13)	2.03(3)
C(8)	0.99517(11)	0.32975(18)	0.18160(12)	1.62(3)
C(9)	0.93702(10)	0.14647(16)	0.06403(12)	1.46(2)
C(10)	0.80526(10)	0.19647(19)	0.16891(12)	1.67(3)
C(11)	0.78083(11)	0.0841(2)	0.23685(13)	2.29(3)
C(12)	0.74298(13)	0.1232(2)	0.32975(14)	3.25(4)
C(13)	0.72899(12)	0.2712(2)	0.35375(16)	3.56(5)
C(14)	0.75243(13)	0.3827(2)	0.28555(14)	3.10(4)
C(15)	0.79091(12)	0.3457(2)	0.19231(13)	2.25(3)
C(16)	1.07154(10)	0.00107(18)	0.09708(12)	1.64(3)
C(17)	1.11514(11)	-0.13015(19)	0.14591(13)	1.98(3)
C(18)	1.14987(12)	-0.2399(2)	0.09273(17)	3.01(4)
C(19)	0.92929(10)	-0.11722(17)	0.11937(12)	1.56(3)
C(20)	0.80055(11)	0.18061(18)	-0.02029(12)	1.66(3)
C(21)	0.70599(12)	0.1853(2)	-0.01235(13)	2.07(3)
C(22)	0.65884(13)	0.2226(2)	-0.09241(16)	2.79(4)

$$B_{eq} = 8/3 \pi^2 (U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos \gamma + 2U_{13}(aa^*cc^*)\cos \beta + 2U_{23}(bb^*cc^*)\cos \omega)$$

Table 2. Atomic coordinates and B_{iso} involving hydrogens/ B_{eq}

atom	x	y	z	B_{eq}
H(1)	1.0084	0.2190	0.4212	2.43
H(2)	1.0379	0.4734	0.4469	2.94
H(3)	1.0414	0.6344	0.3063	2.91
H(4)	1.0148	0.5475	0.1400	2.41
H(5)	0.9493	0.1186	-0.0046	1.72
H(6)	0.7900	-0.0181	0.2194	2.74

H(7)	0.7269	0.0477	0.3772	3.88
H(8)	0.7034	0.2976	0.4175	4.28
H(9)	0.7420	0.4845	0.3023	3.70
H(10)	0.8074	0.4207	0.1450	2.70
H(11)	1.1023	0.0889	0.1141	1.96
H(12)	1.0713	-0.0128	0.0247	1.98
H(13)	1.1175	-0.1340	0.2186	2.38
H(14)	1.1770	-0.3199	0.1277	3.61
H(15)	1.1486	-0.2382	0.0193	3.64
H(16)	0.6794	0.1604	0.0511	2.48
H(17)	0.5989	0.2256	-0.0869	3.34
H(18)	0.6860	0.2474	-0.1558	3.36

$$B_{eq} = 8/3 \pi^2 (U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos\gamma + 2U_{13}(aa^*cc^*)\cos\beta + 2U_{23}(bb^*cc^*)\cos\omega)$$

Table 3. Anisotropic displacement parameters

atom	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
O(1)	0.0317(6)	0.0266(6)	0.0199(5)	-0.0021(5)	0.0006(5)	0.0068(5)
O(2)	0.0303(6)	0.0190(5)	0.0190(5)	-0.0041(4)	-0.0027(5)	0.0035(4)
O(3)	0.0296(7)	0.0455(7)	0.0177(6)	0.0003(5)	-0.0030(5)	0.0013(5)
N(1)	0.0205(6)	0.0225(6)	0.0152(6)	0.0032(5)	-0.0007(5)	-0.0010(5)
N(2)	0.0276(7)	0.0259(7)	0.0350(9)	-0.0029(6)	-0.0011(6)	-0.0029(6)
C(1)	0.0178(7)	0.0176(7)	0.0175(7)	-0.0012(5)	-0.0010(6)	0.0014(5)
C(2)	0.0177(8)	0.0245(8)	0.0146(7)	0.0001(6)	0.0003(5)	0.0015(6)
C(3)	0.0207(8)	0.0217(8)	0.0179(7)	-0.0021(6)	-0.0007(6)	0.0011(6)
C(4)	0.0276(9)	0.0319(9)	0.0174(7)	-0.0022(7)	0.0015(7)	-0.0014(7)
C(5)	0.0333(10)	0.0347(10)	0.0251(9)	-0.0054(8)	0.0004(7)	-0.0116(7)
C(6)	0.0308(9)	0.0229(9)	0.0386(11)	-0.0048(7)	0.0018(8)	-0.0081(7)
C(7)	0.0276(9)	0.0218(8)	0.0279(9)	-0.0027(7)	-0.0001(7)	0.0018(7)
C(8)	0.0192(7)	0.0221(8)	0.0205(8)	0.0001(6)	-0.0005(7)	-0.0012(6)
C(9)	0.0212(7)	0.0186(7)	0.0156(7)	0.0001(5)	0.0003(6)	-0.0006(5)
C(10)	0.0159(7)	0.0321(9)	0.0156(7)	0.0032(6)	-0.0020(6)	-0.0048(6)
C(11)	0.0217(8)	0.0391(10)	0.0261(9)	0.0010(7)	0.0015(7)	0.0025(7)
C(12)	0.0264(9)	0.0690(14)	0.0279(10)	0.0035(10)	0.0065(8)	0.0070(9)
C(13)	0.0277(11)	0.0855(17)	0.0222(9)	0.0143(10)	0.0027(8)	-0.0153(10)
C(14)	0.0290(9)	0.0550(12)	0.0339(9)	0.0134(10)	-0.0073(9)	-0.0232(9)
C(15)	0.0250(8)	0.0355(10)	0.0249(9)	0.0062(7)	-0.0048(7)	-0.0087(7)
C(16)	0.0187(7)	0.0234(8)	0.0201(7)	-0.0015(6)	0.0024(6)	0.0010(6)
C(17)	0.0182(8)	0.0297(8)	0.0274(9)	0.0007(6)	-0.0008(6)	0.0047(7)
C(18)	0.0320(10)	0.0356(10)	0.0469(12)	0.0105(8)	0.0045(9)	0.0069(9)
C(19)	0.0195(8)	0.0222(8)	0.0175(7)	0.0027(6)	0.0014(6)	-0.0007(6)
C(20)	0.0244(9)	0.0177(7)	0.0211(8)	0.0015(6)	-0.0026(7)	-0.0033(6)
C(21)	0.0276(9)	0.0278(9)	0.0233(8)	0.0029(7)	-0.0014(7)	-0.0060(7)
C(22)	0.0319(10)	0.0376(10)	0.0364(10)	0.0079(8)	-0.0097(8)	-0.0060(8)

The general temperature factor expression: $\exp(-2\pi^2(a^2U_{11}h^2 + b^2U_{22}k^2 + c^2U_{33}l^2 + 2a*b*U_{12}hk + 2a*c*U_{13}hl + 2b*c*U_{23}kl))$

Table 4. Bond lengths (Å)

atom	atom	distance	atom	atom	distance
O(1)	C(2)	1.2145(19)	O(2)	C(8)	1.3806(19)
O(2)	C(9)	1.4341(18)	O(3)	C(20)	1.221(2)
N(1)	C(9)	1.453(2)	N(1)	C(10)	1.451(2)
N(1)	C(20)	1.393(2)	N(2)	C(19)	1.142(2)
C(1)	C(2)	1.544(2)	C(1)	C(9)	1.549(2)
C(1)	C(16)	1.568(2)	C(1)	C(19)	1.476(2)
C(2)	C(3)	1.470(2)	C(3)	C(4)	1.405(2)
C(3)	C(8)	1.398(2)	C(4)	C(5)	1.382(2)
C(5)	C(6)	1.390(2)	C(6)	C(7)	1.381(2)
C(7)	C(8)	1.390(2)	C(10)	C(11)	1.390(2)
C(10)	C(15)	1.385(2)	C(11)	C(12)	1.388(2)
C(12)	C(13)	1.376(3)	C(13)	C(14)	1.382(3)
C(14)	C(15)	1.392(2)	C(16)	C(17)	1.498(2)
C(17)	C(18)	1.316(2)	C(20)	C(21)	1.489(2)
C(21)	C(22)	1.318(2)			

Table 5. Bond lengths involving hydrogens (Å)

atom	atom	distance	atom	atom	distance
C(4)	H(1)	0.947	C(5)	H(2)	0.951
C(6)	H(3)	0.948	C(7)	H(4)	0.949
C(9)	H(5)	0.945	C(11)	H(6)	0.951
C(12)	H(7)	0.947	C(13)	H(8)	0.949
C(14)	H(9)	0.948	C(15)	H(10)	0.944
C(16)	H(11)	0.947	C(16)	H(12)	0.947
C(17)	H(13)	0.944	C(18)	H(14)	0.948
C(18)	H(15)	0.953	C(21)	H(16)	0.949
C(22)	H(17)	0.944	C(22)	H(18)	0.952

Table 6. Bond angles (°)

atom	atom	atom	angle	atom	atom	atom	angle
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C(8)	O(2)	C(9)	118.26(12)	C(9)	N(1)	C(10)	120.14(12)
C(9)	N(1)	C(20)	116.95(12)	C(10)	N(1)	C(20)	119.69(12)
C(2)	C(1)	C(9)	112.39(12)	C(2)	C(1)	C(16)	107.33(12)
C(2)	C(1)	C(19)	110.66(12)	C(9)	C(1)	C(16)	107.66(12)
C(9)	C(1)	C(19)	109.09(12)	C(16)	C(1)	C(19)	109.62(12)
O(1)	C(2)	C(1)	121.70(14)	O(1)	C(2)	C(3)	124.43(14)
C(1)	C(2)	C(3)	113.72(13)	C(2)	C(3)	C(4)	120.48(14)
C(2)	C(3)	C(8)	120.71(14)	C(4)	C(3)	C(8)	118.81(14)
C(3)	C(4)	C(5)	120.57(15)	C(4)	C(5)	C(6)	119.35(16)
C(5)	C(6)	C(7)	121.32(17)	C(6)	C(7)	C(8)	119.21(16)
O(2)	C(8)	C(3)	123.28(14)	O(2)	C(8)	C(7)	116.01(14)
C(3)	C(8)	C(7)	120.71(14)	O(2)	C(9)	N(1)	112.37(11)
O(2)	C(9)	C(1)	110.14(12)	N(1)	C(9)	C(1)	114.73(12)
N(1)	C(10)	C(11)	120.15(15)	N(1)	C(10)	C(15)	119.11(14)
C(11)	C(10)	C(15)	120.73(15)	C(10)	C(11)	C(12)	119.15(18)
C(11)	C(12)	C(13)	120.4(2)	C(12)	C(13)	C(14)	120.35(19)
C(13)	C(14)	C(15)	120.0(2)	C(10)	C(15)	C(14)	119.31(17)
C(1)	C(16)	C(17)	114.04(13)	C(16)	C(17)	C(18)	123.38(17)
N(2)	C(19)	C(1)	178.41(16)	O(3)	C(20)	N(1)	121.37(15)
O(3)	C(20)	C(21)	122.18(15)	N(1)	C(20)	C(21)	116.40(14)
C(20)	C(21)	C(22)	120.85(17)				

Table 7. Bond angles involving hydrogens ($^{\circ}$)

atom	atom	atom	angle	atom	atom	atom	angle
C(3)	C(4)	H(1)	119.5	C(5)	C(4)	H(1)	120.0
C(4)	C(5)	H(2)	120.9	C(6)	C(5)	H(2)	119.8
C(5)	C(6)	H(3)	119.0	C(7)	C(6)	H(3)	119.6
C(6)	C(7)	H(4)	120.4	C(8)	C(7)	H(4)	120.4
O(2)	C(9)	H(5)	106.5	N(1)	C(9)	H(5)	106.5
C(1)	C(9)	H(5)	106.0	C(10)	C(11)	H(6)	120.0
C(12)	C(11)	H(6)	120.8	C(11)	C(12)	H(7)	120.0
C(13)	C(12)	H(7)	119.6	C(12)	C(13)	H(8)	120.2
C(14)	C(13)	H(8)	119.4	C(13)	C(14)	H(9)	119.9
C(15)	C(14)	H(9)	120.1	C(10)	C(15)	H(10)	119.7
C(14)	C(15)	H(10)	121.0	C(1)	C(16)	H(11)	107.9

C(1)	C(16)	H(12)	107.9	C(17)	C(16)	H(11)	108.4
C(17)	C(16)	H(12)	108.6	H(11)	C(16)	H(12)	109.9
C(16)	C(17)	H(13)	118.0	C(18)	C(17)	H(13)	118.7
C(17)	C(18)	H(14)	119.8	C(17)	C(18)	H(15)	120.2
H(14)	C(18)	H(15)	120.0	C(20)	C(21)	H(16)	119.5
C(22)	C(21)	H(16)	119.6	C(21)	C(22)	H(17)	120.5
C(21)	C(22)	H(18)	119.1	H(17)	C(22)	H(18)	120.4