Organic Letters

SUPPORTING INFORMATION

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"Diels-Alder Reactions of 5-Vinyl-1-acyl-2-aryl-2,3-dihydro-4-pyridones: Regio- and Stereo-selective Synthesis of Octahydroquinolines"

- I.** Experimental procedures and spectral data for compounds **5-17** (9 pages).
- II.** ORTEP plot and X-ray data for **5** (13 pages).

Total pages of supporting information: (23).

Supporting Information

Experimental

Melting points are uncorrected. Reactions were performed in oven-dried glassware under an atmosphere of dry argon and were magnetically stirred. Solutions were evaporated under reduced pressure with a rotary evaporator, and the residue was purified by radial preparative-layer chromatography (radial PLC) using an ethyl acetate/hexane mixture as the eluent unless specified otherwise.

Preparation of (2*R*^{*},6*aS*^{*},9*aS*^{*},9*bR*^{*})-4,7,9-Trioxo-2,8-diphenyl-1-((phenoxy)carbonyl)-2,3,4,6,6*a*,7,8,9,9*a*,9*b*-decahydro-pyrrolo[3,4-*h*]quinoline (5). To a solution of 68 mg (0.213 mmol) of **4** in 7 mL of toluene was added 47 mg (0.271 mmol) of *N*-phenylmaleimide. The mixture was heated to reflux for 12 h, cooled to rt, and concentrated under reduced pressure. The crude residue was purified by PLC (silica gel, EtOAc/hexane) to give 100 mg (96%) of **5** as a white solid: mp 199-200 °C (ethyl acetate/hexane); IR (thin film) 3034, 2929, 1707, 1613, 1493, 1383, 1190, 756 cm⁻¹; ¹H NMR (CDCl₃, 300 MHz) δ 2.41 (m, 1H), 2.84 (dd, 1H, *J* = 15.6 and 2.3 Hz), 3.17 (dd, 1H, *J* = 15.6 and 7.9 Hz), 3.31 (dd, 1H, *J* = 15.6 and 6.3 Hz), 3.44 (t, 1H, *J* = 8.2 Hz), 4.71 (m, 1H), 5.01 (m, 1H), 5.94 (m, 1H), 6.86 (m, 1H), 7.07-7.50 (m, 15H); ¹³C NMR (CDCl₃, 75 MHz) δ 25.6, 39.9, 42.5, 44.3, 55.9, 58.4, 121.9, 125.6, 126.0, 126.6, 128.1, 129.2, 129.5, 129.6, 131.6, 135.8, 141.7, 151.1, 155.0, 176.2, 177.5, 191.7.

Preparation of (2*R*^{*},6*aS*^{*},9*aS*^{*},9*bR*^{*})-4,7,9-Trioxo-1-((phenoxy)carbonyl)-2-phenyl-3,4,6,6*a*,7,9,9*a*,9*b*-octahydro-2*H*-furo[3,4-*h*]quinoline (6). To a solution of 29 mg (0.091 mmol) of **4** in 5 mL of toluene was added 12 mg (0.122 mmol) of maleic anhydride. The mixture was heated at reflux for 12 h, cooled to rt, and concentrated under reduced pressure. The residue was recrystallized from an ethyl acetate/hexane mixture to give 34 mg (90%) of **6** as a

colorless solid: mp 228 °C (decomp); IR (thin film) 1766, 1701, 1360, 1331, 1258, 1211, 1180, 1079, 977 cm⁻¹; ¹H NMR (CDCl₃, 300 MHz) δ 2.39 (m, 1H), 2.89 (d, 1H, *J* = 15.6 Hz), 3.11 (dd, 1H, *J* = 15.6 and 7.7 Hz), 3.28 (dd, 1H, *J* = 15.6 and 6.1 Hz), 3.59 (t, 1H, *J* = 8.6 Hz), 4.87 (m, 1H), 5.97 (m, 1H), 6.84 (d, 1H, *J* = 7.1 Hz), 7.25 (m, 11H); ¹³C NMR (CDCl₃, 75 MHz) δ 25.2, 40.3, 43.6, 44.1, 55.2, 58.4, 121.7, 125.6, 126.2, 128.2, 129.6, 129.7, 135.7, 135.9, 141.3, 150.9, 170.9, 172.6, 190.8. Anal. Calcd. for C₂₄H₁₉NO₆: C, 69.06; H, 4.59; N, 3.36. Found: C, 69.19; H, 4.70; N, 3.33.

Preparation of (2*R*^{*},7*S*^{*},8*R*^{*},8*aR*^{*})-7,8-Bis-benzenesulfonyl-4-oxo-1-((phenoxy)carbonyl)-3,4,6,7,8,8*a*-hexahydro-2*H*-quinoline (7). To a solution of 35 mg (0.110 mmol) of **4** in 7 mL of toluene was added 40 mg (0.130 mmol) of *cis*-1,2-bis(phenylsulfonyl)ethylene. The mixture was heated at reflux for 12h, cooled to rt, and concentrated under reduced pressure. The crude residue was purified by PLC (silica gel, EtOAc/hexane) to give 52 mg (76%) of **7** as a white solid: mp 200 °C (decomp); IR (thin film) 3060, 1713, 1642, 1448, 1385, 1310, 1267, 1197, 1149, 1083 cm⁻¹; ¹H NMR (CDCl₃, 300 MHz) δ 2.13 (m, 1H), 2.96 (m, 1H), 3.15 (dd, 1H, *J* = 17.9 and 4.8 Hz), 3.31 (m, 2H), 5.12 (s, 1H), 5.44 (s, 1H), 5.82 (s, 1H), 6.54 (m, 1H), 6.90 (m, 4H), 7.04 (m, 2H), 7.33 (m, 6H), 7.60 (m, 2H), 7.77 (m, 4H), 8.03 (m, 2H); ¹³C NMR (CDCl₃, 75 MHz) δ 23.7, 42.6, 53.0, 54.2, 59.5, 62.2, 121.9, 126.0, 126.5, 127.6, 128.3, 128.8, 129.0, 129.2, 129.4, 129.6, 132.6, 133.9, 134.1, 138.3, 140.1, 140.3, 150.5, 155.7, 196.9. Anal. Calcd. for C₃₄H₂₉NO₇S₂: C, 65.05; H, 4.66; N, 2.23. Found: C, 64.78; H, 4.67; N, 2.17.

Preparation of (3*aS*^{*},8*R*^{*},9*aR*^{*},9*bS*^{*})-1,6-Dioxo-9-((phenoxy)carbonyl)-8-phenyl-1,2,3,3*a*,4,6,7,8,9*a*,9*b*-decahydro-9-aza-cyclopenta[*a*]naphthalene (8). To a solution of 30 mg (0.0939 mmol) of **4** in 5 mL of toluene was added 10 mg (0.122 mmol) of 2-cyclopentenone. The mixture was heated at 200 °C in a sealed tube for 12 h, cooled to rt, and concentrated under reduced pressure. The crude residue was purified by PLC (silica gel, EtOAc/hexane) to afford 27 mg (74%) of **8** as a

colorless foam: IR (thin film) 3060, 2931, 1731, 1689, 1614, 1493, 1300, 1260, 1197 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz) δ 1.65 (m, 1H), 1.91 (m, 1H), 2.11-2.46 (m, 3H), 2.81 (dd, 1H, J = 15.4 and 3.0 Hz), 2.95 (dd, 1H, J = 16.5 and 10.4 Hz), 3.08 (m, 1H), 3.28 (m, 1H), 3.44 (dd, 1H, J = 15.4 and 6.0 Hz), 3.85 (m, 1H), 4.85 (m, 1H), 5.91 (m, 1H), 6.83 (m, 1H), 7.24 (m, 9H); ^{13}C NMR (CDCl_3 , 75 MHz) δ 29.4, 30.1, 34.5, 39.0, 44.2, 50.9, 56.3, 58.2, 121.9, 125.8, 127.8, 128.5, 128.7, 129.3, 129.4, 136.2, 137.8, 144.9, 151.1, 154.9, 193.2, 224.0; HRMS Calcd. for $\text{C}_{25}\text{H}_{23}\text{NO}_4$: 402.1705 $[\text{M} + \text{H}]^+$; found, 402.1727 $[\text{M} + \text{H}]^+$.

Preparation of (7*R,8*aR**)-1,2-((Diethoxy)carbonyl)-5-oxo-8-((phenoxy)carbonyl)-7-phenyl-3,6,7,8a-tetrahydro-5*H*-pyrido[2,3-*c*]pyridazine (9).** To a solution of 38 mg (0.119 mmol) of **4** in 10 mL of benzene was added 27 mg (0.155 mmol) of diethyl azodicarboxylate. The mixture was heated at reflux for 10 min, cooled to rt, and concentrated under reduced pressure. The crude residue was purified by PLC (silica gel, EtOAc/hexane) to afford 55 mg (93%) of **9** as a colorless oil: IR (neat) 2977, 1711, 1649, 1405, 1311, 1233, 1205 cm^{-1} ; ^1H NMR ($\text{C}_2\text{D}_6\text{SO}$, 300 MHz, 130 $^\circ\text{C}$) δ 1.17 (m, 6H), 3.22 (dt, 1H, J = 17.4 and 2.8 Hz), 3.62 (m, 1H), 3.69 (m, 1H), 4.00 (m, 1H), 4.17 (m, 4H), 5.48 and 5.71 (m, due to rotamers, 1H), 6.39 and 6.44 (m, due to rotamers, 1H), 6.77 and 6.95 (m, due to rotamers, 1H), 7.16-7.50 (m, 10H); ^{13}C NMR ($\text{C}_2\text{D}_6\text{SO}$, 75 MHz, 130 $^\circ\text{C}$) δ 13.4 and 13.5 (due to rotamers), 13.7, 41.4 and 42.5 (due to rotamers), 52.3 and 53.6 (due to rotamers), 61.2, 62.0, 61.9 and 62.5 (due to rotamers), 63.3, 120.4 and 120.8 (due to rotamers), 124.4, 125.0 and 125.1 (due to rotamers), 127.4 and 127.7 (due to rotamers), 128.4, 128.8, 131.9, 133.2, 137.2 and 139.1 (due to rotamers), 139.6 and 140.1 (due to rotamers), 150.9, 153.6 and 154.2 (due to rotamers), 195.0; HMRS Calcd. for $\text{C}_{26}\text{H}_{27}\text{N}_3\text{O}_7$: 493.1849; found, 493.1844.

Preparation of (2*R,7*S**,8*S**,8*aR**)-4-Oxo-1-((phenoxy)carbonyl)-2-phenyl-3,4,6,7,8,8a-hexahydro-2*H*-quinoline-7,8-dicarboxylic acid-7,8-**

dimethyl ester (10). To a solution of 31 mg (0.971 mmol) of **4** in 6 mL of toluene was added 18 mg (0.126 mmol) of dimethyl maleate. The mixture was heated at 200 °C in a sealed tube for 12 h, cooled to rt, and concentrated under reduced pressure. The crude residue was purified by PLC (silica gel, EtOAc/hexane) to afford 36 mg (82%) of **10** as a colorless solid: mp 206-207 °C (ethyl acetate/hexane); IR (thin film) 3055, 2954, 1725, 1691, 1626, 1450, 1430, 1351, 1315, 1271, 1239, 1204, 1161 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz) δ 2.60 (m, 1H), 2.82-3.10 (m, 3H), 3.18 (dd, 1H, $J = 16.3$ and 4.9 Hz), 3.71 (s, 6H), 4.47 (m, 1H), 4.99 (m, 1H), 5.58 (m, 1H), 6.77 (d, 1H, $J = 7.8$ Hz), 6.87 (m, 1H), 7.29 (m, 9H); ^{13}C NMR (CDCl_3 , 75 MHz) δ 26.1, 41.0, 43.3, 52.2, 52.5, 57.1, 57.2, 121.7, 125.8, 126.4, 128.0, 129.2, 133.5, 137.6, 140.2, 150.8, 155.0, 171.3, 172.5, 195.7. Anal. Calcd. for $\text{C}_{26}\text{H}_{25}\text{NO}_7$: C, 67.38; H, 5.44; N, 3.02. Found: C, 67.41; H, 5.52; N, 3.00.

Diels Alder Reaction of 4 with Dimethyl fumarate. To a solution of 63 mg (0.197 mmol) of **4** in 8 mL of toluene was added 34 mg (0.257 mmol) of dimethyl fumarate. The mixture was heated at reflux for 12 h, cooled to rt, and concentrated under reduced pressure. The crude residue was purified by PLC (silica gel, EtOAc/hexane) to give 70 mg (78%) of an inseparable 1:1 mixture of diastereomers as a colorless foam: IR (thin film) 3027, 2953, 1734, 1627, 1483, 1434, 1397, 1317, 1269, 1205, 1023, 811, 735 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz) (diastereomer **11a**) δ 1.96 (m, 1H), 2.45-3.32 (m, 4H), 3.69 (s, 3H), 3.74 (s, 3H), 4.86 (m, 1H), 5.15 (m, 1H), 6.12 (d, 1H, $J = 5.1$ Hz), 6.83 (m, 1H), 7.36 (m, 10H); (diastereomer **11b**) δ 2.16 (t, 1H, $J = 11.0$ Hz), 2.45-3.32 (m, 4H), 3.59 (s, 6H), 3.70 (m, 1H), 4.34 (t, 1H, $J = 5.2$ Hz), 5.74 (t, 1H, $J = 5.1$ Hz), 6.70 (m, 1H), 7.00 (m, 1H), 7.36 (m, 9H); ^{13}C NMR (CDCl_3 , 75 MHz) δ 25.9, 28.6, 40.9, 42.0, 43.6, 45.0, 47.1, 52.3, 52.4, 52.5, 52.7, 54.1, 54.3, 55.1, 56.0, 57.7, 121.4, 121.7, 126.0, 126.8, 127.9, 128.2, 128.4, 128.6, 128.8, 129.2, 129.4, 129.5, 134.0, 134.3, 135.7, 136.5, 137.4, 141.0, 150.8, 151.3, 154.5, 154.8, 172.2, 172.4,

173.5, 173.9, 193.7; HRMS Calcd. for $C_{26}H_{25}NO_7$: 464.1709 $[M + H]^+$; found, 464.1733 $[M + H]^+$.

Diels Alder Reaction of 4 with Methyl Acrylate. To a solution of 50 mg (0.157 mmol) of **4** in 7 mL of toluene was added 67 mg (0.783 mmol) of methyl acrylate. The mixture was heated at reflux for 12 h, cooled to rt, and concentrated under reduced pressure. The crude residue was purified by PLC (silica gel, EtOAc/hexane) to give 54 mg (86%) of an inseparable 2.5:1 mixture of diastereomers as a colorless foam: IR (thin film) 2953, 1729, 1699, 1617, 1495, 1529, 1399, 1318, 1266, 1202, 1164 cm^{-1} ; (major diastereomer **12a**) 1H NMR ($CDCl_3$, 300 MHz) δ 1.89-2.46 (m, 4H), 2.94 (dd, 1H, $J = 16.1$ and 5.9 Hz), 3.26 (d, 1H, $J = 16.1$ Hz), 3.71 (s, 3H), 3.89 (m, 1H), 4.84 (m, 1H), 5.73 (t, 1H, $J = 5.9$ Hz), 6.76 (m, 2H), 7.35 (m, 9H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 23.5, 23.9, 42.2, 43.6, 51.9, 55.8, 57.3, 121.7, 125.6, 126.2, 127.7, 128.5, 129.1, 129.4, 134.4, 138.1, 140.1, 150.9, 173.1, 195.5. (Minor diastereomer **12b**) 1H NMR ($CDCl_3$, 300 MHz) δ 1.89-2.46 (m, 4H), 2.98 (dd, 1H, $J = 16.1$ and 5.9 Hz), 3.27 (d, 1H, $J = 16.1$ Hz), 3.58 (s, 3H), 3.68 (m, 1H), 5.07 (m, 1H), 6.08 (m, 1H), 6.91 (m, 2H), 7.35 (m, 9H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 23.7, 24.7, 42.0, 45.7, 52.2, 54.5, 57.3, 121.6, 125.7, 126.2, 127.8, 128.1, 128.2, 128.5, 134.0, 138.1, 141.1, 154.6, 173.5, 195.5; HRMS Calcd. for $C_{24}H_{23}NO_5$: 406.1654 $[M + H]^+$; found, 406.1665 $[M + H]^+$.

Diels Alder Reaction of 4 with Acrylonitrile. To a solution of 60 mg (0.188 mmol) of **4** in 8 mL of toluene was added 50 mg (0.939 mmol) of acrylonitrile. The mixture was heated at reflux for 12 h, cooled to rt, and concentrated under reduced pressure. The crude residue was purified by PLC (silica gel, EtOAc/hexane) to give 63 mg (91%) of an inseparable 2:1 mixture of diastereomers as a colorless foam: IR (thin film) 3050, 2943, 2247, 1728, 1700, 1627, 1493, 1278 cm^{-1} ; (major diastereomer **12a**) 1H NMR ($CDCl_3$, 300 MHz) δ 1.76-2.48 (m, 3H), 2.69 (m, 1H), 3.14 (m, 2H), 4.12 (m, 1H), 4.89 (m, 1H), 5.65 (m, 1H), 6.73 (d, 1H, $J = 7.7$ Hz), 6.94 (m, 1H), 7.33

(m, 9H); ^{13}C NMR (CDCl_3 , 75 MHz) δ 22.6, 23.9, 30.5, 43.6, 56.4, 57.3, 121.5, 126.0, 126.8, 127.7, 128.1, 129.1, 129.5, 133.8, 139.2, 150.5, 155.2, 195.6. (Minor diastereomer **12b**) ^1H NMR (CDCl_3 , 300 MHz) δ 1.76-2.48 (m, 4H), 2.69 (m, 1H), 3.14 (m, 2H), 5.24 (m, 1H), 6.13 (d, 1H, $J = 5.8$ Hz), 6.79 (m, 1H), 7.33 (m, 10H); ^{13}C NMR (CDCl_3 , 75 MHz) δ 24.7, 25.6, 33.4, 41.4, 51.7, 53.9, 119.8, 122.0, 126.1, 126.8, 127.7, 128.1, 129.5, 129.5, 133.6, 138.4, 139.5, 151.4, 195.6; HRMS Calcd. for $\text{C}_{23}\text{H}_{20}\text{N}_2\text{O}_3$: 373.1552 $[\text{M} + \text{H}]^+$; found, 373.1571 $[\text{M} + \text{H}]^+$.

Diels Alder Reaction of 1 with Phenyl Vinylsulfone. To a solution of 50 mg (0.157 mmol) of **1** in 8 mL of toluene was added 34 mg (0.201 mmol) of phenyl vinylsulfone. The mixture was heated at 200 °C in a sealed tube for 12h, cooled to rt, and concentrated under reduced pressure. The crude residue was purified by PLC (silica gel, EtOAc/hexane) to give 49 mg (65%) of a mixture of diastereomers. Fractional crystallization from CHCl_3 /hexane afforded the minor diastereomer (**12b**) as a white solid: mp 240 °C (decomp); IR (thin film) 1714, 1697, 1629, 1401, 1316, 1278, 1202, 1142 cm^{-1} ; ^1H NMR (CDCl_3 , 500 MHz) δ 1.42 (m, 1H), 1.85 (m, 2H), 2.26 (m, 1H), 2.38 (br s, 1H), 3.10 (dd, 1H, $J = 19.0$ and 6.5 Hz), 3.23 (d, 1H, $J = 19.0$ Hz), 5.75 (br s, 1H), 6.23 (d, 1H, $J = 6.5$ Hz), 6.74 (m, 1H), 7.20 (m, 1H), 7.29-7.56 (m, 10H), 7.58 (m, 3H), 7.72 (m, 1H); ^{13}C NMR (CDCl_3 , 100 MHz) δ 23.3, 24.7, 41.6, 51.2, 52.9, 61.0, 122.2, 125.7, 128.3, 128.4, 128.6, 128.8, 129.0, 129.1, 129.2, 129.5, 133.8, 134.9, 138.7, 139.1, 152.0, 197.7. Anal. Calcd. for $\text{C}_{28}\text{H}_{25}\text{NO}_5\text{S}$: C, 68.98; H, 5.17; N, 2.87. Found: C, 68.68; H, 4.86; N, 2.56.

The major diastereomer (**12a**) was obtained from the mother liquor as a colorless oil: IR (thin film) 1714, 1699, 1625, 1400, 1317, 1278, 1200, 1145 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz) δ 2.16 (m, 1H), 2.27 (m, 2H), 2.76 (m, 1H), 2.82 (d, 1H, $J = 15.1$ Hz), 3.85 (dd, 1H, $J = 15.1$ and 6.3 Hz), 5.02 (m, 1H), 5.23 (s, 1H), 5.69 (m, 1H), 6.45 (d, 1H, $J = 7.4$ Hz), 7.26 (m, 12H), 7.64 (m, 2H), 7.96 (d, 1H, $J = 7.9$ Hz); ^{13}C NMR (CDCl_3 , 75 MHz) δ 22.8, 44.1, 57.5, 59.1, 59.4, 121.6, 125.8, 125.8, 128.0, 128.1,

129.4, 129.6, 132.8, 133.8, 138.4, 141.4, 142.5, 150.8, 155.4, 193.8; HRMS Calcd. for $C_{28}H_{24}NO_5S$: 488.1532 $[M + H]^+$; found: 488.1532 $[M + H]^+$.

Lucas Reduction of 4. To a stirred solution of 120 mg (0.376 mmol) of **4** in 20 mL of MeOH was added 154 mg (0.413 mmol) of cerium trichloride heptahydrate. After stirring for 5 min, 18 mg (0.476 mmol) of sodium borohydride was added and stirring was continued for 10 min. The reaction mixture was diluted with water and extracted with CH_2Cl_2 , and the combined extracts were dried over $MgSO_4$. The solvent was removed under reduced pressure and the residue was purified by PLC (silica gel, EtOAc/hexane). The first product to elute (18 mg, 15%) was identified as (2*R**,4*R**)-4-hydroxy-1-((phenoxy)carbonyl)-2-phenyl-5-vinyl-3,4-dihydro-2*H*-pyridine **14** as a colorless oil: IR (neat) 3565, 3060, 2922, 1727, 1643, 1493, 1423, 1376, 1327, 1195 cm^{-1} ; 1H NMR ($CDCl_3$, 300 MHz) δ 0.80 (d, 1H, $J = 9.3$ Hz), 2.38 (m, 1H), 2.70 (d, 1H, $J = 14.7$ Hz), 4.46 (m, 1H), 5.05 (d, 1H, $J = 10.6$ Hz), 5.33 (d, 1H, $J = 17.4$ Hz), 5.67 (m, 1H), 6.36 (dd, 1H, $J = 17.4$ and 11.4 Hz), 6.90 (m, 1H), 7.29 (m, 10H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 36.4, 32.7, 53.4, 60.7, 111.5, 111.9, 121.6, 125.2, 126.1, 126.6, 127.6, 129.3, 129.6, 135.0, 140.2, 152.5; HRMS Calcd. for $C_{20}H_{19}NO_3$: 321.1365; found, 321.1362.

The second product to elute (90 mg, 74%) was identified as (2*R**,4*S**)-4-hydroxy-1-((phenoxy)carbonyl)-2-phenyl-5-vinyl-3,4-dihydro-2*H*-pyridine **13** as a colorless foam: IR (thin film) 3400, 3049, 2924, 1723, 1636, 1493, 1339, 1198 cm^{-1} ; 1H NMR ($CDCl_3$, 300 MHz) δ 1.86 (d, 1H, $J = 5.4$ Hz), 2.26 (m, 1H), 2.47 (m, 1H), 4.36 (m, 1H), 5.05 (d, 1H, $J = 11.2$ Hz), 5.32 (d, 1H, $J = 17.7$ Hz), 5.42 (t, 1H, $J = 4.8$ Hz), 6.29 (dd, 1H, $J = 17.7$ and 11.2 Hz), 6.91 (m, 1H), 7.30 (m, 10H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 38.5, 55.2, 60.9, 112.5, 121.6, 125.3, 126.0, 126.8, 127.5, 128.9, 129.5, 134.1, 141.0, 150.8, 152.1; HRMS Calcd. for $C_{20}H_{19}NO_3$: 321.1365; found, 321.1364.

Preparation of (2*R,4*S**,6*aS**,9*aS**,9*bR**)-4-Hydroxy-7,9-dioxo-2,8-diphenyl-1-((phenoxy)carbonyl)-2,3,4,6,6*a*,7,8,9,9*a*,9*b*-decahydro-pyrrolo[3,4-*h*]quinoline (15).** To a solution of 46 mg (0.143 mmol) of **13** in 10 mL of toluene was added 32 mg (0.186 mmol) of *N*-phenylmaleimide. The mixture was heated at reflux for 30 min, cooled to rt, and concentrated under reduced pressure. The crude residue was purified by PLC (silica gel, EtOAc/hexane) to give 62 mg (87%) of **15** as a colorless solid: mp 202-203 °C (ethyl acetate/hexane); IR (thin film) 3473, 3063, 2958, 1709, 1596, 1495, 1381, 1359, 1208, 1180 cm⁻¹; ¹H NMR (CDCl₃, 300 MHz) δ 1.72 (d, 1H, *J* = 5.4 Hz), 2.03 (m, 2H), 2.26 (m, 1H), 3.00 (dd, 1H, *J* = 15.0 and 7.0 Hz), 3.31 (t, 1H, *J* = 7.7 Hz), 4.36 (m, 2H), 5.11 (m, 1H), 5.53 (m, 1H), 6.10 (m, 1H), 6.64 (d, 1H, *J* = 8.2 Hz), 7.10-7.50 (m, 14H); ¹³C NMR (CDCl₃, 75 MHz) δ 23.9, 38.7, 39.6, 41.8, 55.4, 58.6, 62.7, 118.9, 121.7, 125.4, 125.6, 126.7, 127.1, 128.8, 129.0, 129.2, 129.4, 132.0, 140.2, 144.8, 151.0, 154.6, 177.1, 178.7. Anal. Calcd. for C₃₀H₂₆N₂O₅: C, 72.86; H, 5.30; N, 5.66. Found: C, 72.65; H, 5.29; N, 5.68.

Preparation of (2*R,4*R**,6*aS**,9*aS**,9*bR**)-4-Hydroxy-7,9-dioxo-2,8-diphenyl-1-((phenoxy)carbonyl)-2,3,4,6,6*a*,7,8,9,9*a*,9*b*-decahydro-pyrrolo[3,4-*h*]quinoline (16).** To a solution of 35 mg (0.109 mmol) of **14** in 7 mL of toluene was added 25 mg (0.144 mmol) of *N*-phenylmaleimide. The mixture was heated at reflux for 30 min, cooled to rt, and concentrated under reduced pressure. The crude residue was purified by PLC (silica gel, EtOAc/hexane) to give 50 mg (93%) of **26** as a colorless solid: mp 115-116 °C (ethyl acetate/hexane); IR (thin film) 3479, 3060, 2919, 1711, 1596, 1494, 1383, 1204 cm⁻¹; ¹H NMR (CDCl₃, 300 MHz) δ 2.34 (m, 3H), 2.92 (dd, 1H, *J* = 15.2 and 7.5 Hz), 3.34 (t, 1H, *J* = 8.0 Hz), 4.39 (s, 1H), 4.51 (m, 1H), 5.09 (s, 1H), 5.50 (m, 1H), 6.07 (m, 1H), 6.64 (d, 1H, *J* = 7.5 Hz), 7.05-7.49 (m, 15H); ¹³C NMR (CDCl₃, 75 MHz) δ 24.4, 36.4, 39.7, 41.7, 52.5, 55.5, 66.6, 121.8, 125.5, 125.6, 126.0, 126.6, 127.0, 128.9, 129.0, 129.3, 129.4, 131.9, 138.7,

146.8, 151.8, 155.0, 176.9, 178.6. Anal. Calcd. for $C_{30}H_{26}N_2O_5$: C, 72.86; H, 5.30; N, 5.66. Found: C, 72.70; H, 5.31; N, 5.70.

Preparation of (3a*R,6*S**,8*R**,9a*S**,9b*R**)-6-Hydroxy-1-oxo-9-((phenoxy)carbonyl)-8-phenyl-1,2,3,3a,4,6,7,8,9a,9b-decahydro-9-azacyclopenta[*a*]naphthalene (17).** To a solution of 25 mg (0.0778 mmol) of **13** was added 10 mg (0.12 mmol) of 2-cyclopentenone. The mixture was heated at 200 °C in a sealed tube for 36 h, cooled to rt, and concentrated under reduced pressure. The crude residue was purified by PLC (silica gel, EtOAc/hexane) to give 21 mg (68%) of **17** as a colorless solid: mp 172-173 °C (ethyl acetate/hexane); IR (thin film) 3448, 2931, 1731, 1707, 1495, 1348, 1202 cm^{-1} ; 1H NMR ($CDCl_3$, 300 MHz) δ 1.41 (m, 1H), 1.71 (m, 1H), 2.02-2.59 (m, 6H), 2.94 (m, 1H), 3.58 (m, 1H), 4.31 (m, 1H), 4.97 (s, 1H), 5.56 (m, 1H), 5.88 (m, 1H), 6.53 (d, 1H, $J = 7.7$ Hz), 7.24 (m, 10H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 28.4, 29.2, 33.4, 38.5, 38.8, 50.5, 55.8, 59.0, 63.7, 119.1, 121.8, 125.7, 125.8, 127.0, 128.7, 129.2, 129.5, 138.7, 145.6, 154.7, 219.7; HRMS Calcd. for $C_{25}H_{25}NO_4$: 404.1862 $[M + H]^+$, found: 404.1862 $[M + H]^+$.

Crytallographic data for 5

X98040: -125 DEC C

Table: Crystallographic data for 5

Space Group and Cell Dimensions Triclinic, P -1
 a 10.6023(12) b 11.0790(16) c 12.6073(15)
 alpha 73.726(13) beta 87.496(15) gamma 69.650(12)
 Volume 1330.4(3) Å³

Empirical formula : C₃₁ H₂₆ N₂ O₅ Cl

Cell dimensions were obtained from 25 reflections with 2Theta angle
 in the range 35.00 - 40.00 degrees.

Crystal dimensions : .40 X .30 X .28 mm

FW = 542.00 Z = 2 F(000) = 566.63

Dcalc 1.353Mg.m⁻³, mu .19mm⁻¹, lambda .71073Å, 2Theta(max) 59.8

The intensity data were collected on a Nonius diffractometer,
 using the omega scan mode.

The h,k,l ranges used during structure solution and refinement are :--

Hmin,max -13 14; Kmin,max 0 15; Lmin,max -16 17

No. of reflections measured 7684

No. of unique reflections 7684

No. of reflections with I_{net} > 1.0sigma(I_{net}) 6169

Merging R-value on intensities .000

No correction was made for absorption

The last least squares cycle was calculated with
 65 atoms, 456 parameters and 6163 out of 7684 reflections.
 Weights based on counting-statistics were used.
 The weight modifier K in KFo² is .000450

The residuals are as follows :--

For significant reflections, RF .054, Rw .073 GoF 2.42

For all reflections, RF .054, Rw .073.

where RF = Sum(Fo-Fc)/Sum(Fo),

Rw = Sqrt[Sum(w(Fo-Fc)²)/Sum(wFo²)] andGoF = Sqrt[Sum(w(Fo-Fc)²)/(No. of reflns - No. of params.)]

The maximum shift/sigma ratio was .002.

In the last D-map, the deepest hole was -.770e/Å³,
 and the highest peak .780e/Å³.

The following references are relevant to the NRCVAX System.

1. Full System Reference :

Gabe, E.J., Le Page, Y., Charland, J.-P., Lee, F.L. and White, P.S.
 (1989) J. Appl. Cryst., 22, 384-387.

2. Scattering Factors from Int. Tab. Vol. 4 :

International Tables for X-ray Crystallography, Vol. IV, (1974)
 Kynoch Press, Birmingham, England.

The following references may also be relevant.

3. ORTEP Plotting :

Johnson, C.K., (1976) ORTEP - A Fortran Thermal Ellipsoid Plot

Program, Technical Report ORNL-5138, Oak Ridge

4. Pluto Plotting :
S. Motherwell, University Chemical Laboratory, Cambridge, 1978
5. Missing Symmetry Treatment by MISSYM :
Le Page, Y., (1988) J. Appl. Cryst., 21, 983-984.

X98040: -125 DEC C

2-Sep-1998

Table . Atomic Parameters x,y,z and Biso.
E.S.Ds. refer to the last digit printed.

	x	y	z	Biso
O1	.75624 (14)	.20953 (13)	.68250 (10)	2.00 (5)
O2	.92467 (13)	.37926 (12)	1.07468 (10)	1.63 (5)
O3	.88429 (14)	.18647 (13)	1.15491 (9)	1.87 (5)
O4	1.14835 (13)	.13956 (12)	.92367 (11)	2.00 (5)
O5	1.17818 (14)	.43625 (13)	.59751 (10)	2.08 (5)
N1	.85914 (13)	.27047 (13)	.96958 (10)	1.19 (5)
N2	1.18909 (14)	.26629 (14)	.75595 (11)	1.44 (5)
C1	.79072 (15)	.17461 (15)	.97047 (13)	1.18 (6)
C2	.83393 (17)	.11428 (16)	.87340 (13)	1.33 (6)
C3	.80189 (16)	.22157 (16)	.76495 (13)	1.36 (6)
C4	.83366 (15)	.34256 (16)	.76259 (13)	1.30 (6)
C5	.87135 (16)	.36885 (15)	.86684 (12)	1.19 (6)
C6	1.01428 (16)	.37913 (16)	.85430 (13)	1.25 (6)
C7	1.12164 (16)	.24692 (16)	.85312 (13)	1.39 (6)
C8	1.13672 (17)	.39815 (16)	.68723 (13)	1.45 (6)
C9	1.02283 (16)	.47924 (16)	.74335 (13)	1.36 (6)
C10	.89216 (18)	.53920 (17)	.66656 (14)	1.62 (6)
C11	.84247 (16)	.42868 (17)	.66607 (14)	1.50 (6)
C12	.89140 (16)	.28826 (16)	1.06619 (13)	1.32 (6)
C13	.92087 (18)	.19111 (16)	1.25970 (13)	1.61 (7)
C14	1.0537 (2)	.13180 (19)	1.29911 (16)	2.16 (8)
C15	1.0857 (2)	.1277 (2)	1.40687 (17)	2.46 (8)
C16	.9850 (2)	.18232 (20)	1.47123 (15)	2.30 (8)
C17	.8525 (2)	.2418 (2)	1.42956 (15)	2.17 (8)
C18	.81940 (19)	.24692 (19)	1.32266 (15)	1.90 (7)
C19	.63760 (16)	.23461 (17)	.97107 (13)	1.40 (6)
C20	.5687 (2)	.1503 (2)	1.02379 (20)	2.90 (9)
C21	.4290 (2)	.1983 (3)	1.0263 (2)	3.45 (10)
C22	.35589 (19)	.3327 (2)	.97613 (18)	2.53 (9)
C23	.4224 (2)	.4158 (2)	.9206 (3)	3.55 (11)
C24	.5625 (2)	.3681 (2)	.9181 (2)	3.32 (10)
C25	1.29681 (17)	.16232 (17)	.72562 (14)	1.58 (6)
C26	1.26857 (19)	.06371 (19)	.69412 (17)	2.23 (8)
C27	1.3717 (2)	-.0316 (2)	.65867 (20)	2.70 (9)
C28	1.5014 (2)	-.0288 (2)	.65587 (20)	2.67 (9)
C29	1.52958 (19)	.0686 (2)	.68984 (19)	2.53 (9)
C30	1.42695 (18)	.16485 (19)	.72500 (17)	2.03 (8)
H1	.8245 (17)	.1024 (17)	1.0381 (14)	.3 (3)
H2a	.792 (2)	.053 (2)	.8738 (17)	1.7 (4)
H2b	.926 (2)	.0721 (19)	.8801 (16)	1.0 (3)
H5	.8132 (19)	.4534 (19)	.8680 (15)	1.0 (3)
H6	1.040 (2)	.402 (2)	.9118 (18)	1.7 (4)
H9	1.051 (2)	.549 (2)	.7541 (17)	1.4 (4)
H10a	.908 (2)	.589 (2)	.5933 (17)	1.5 (4)
H10b	.826 (2)	.608 (2)	.6955 (18)	2.0 (4)
H11	.821 (2)	.414 (2)	.605 (2)	2.6 (5)
H14	1.120 (2)	.094 (2)	1.2508 (19)	2.7 (5)
H15	1.180 (3)	.089 (2)	1.436 (2)	2.8 (5)
H16	1.008 (3)	.175 (3)	1.548 (2)	3.7 (6)
H17	.781 (3)	.278 (3)	1.472 (2)	3.3 (5)
H18	.733 (2)	.289 (2)	1.2898 (20)	2.7 (5)
H20	.616 (3)	.053 (3)	1.059 (2)	4.4 (6)
H21	.377 (3)	.137 (3)	1.061 (3)	5.8 (8)
H22	.256 (3)	.367 (3)	.981 (2)	3.8 (6)

H23	.375	(5)	.514	(5)	.863	(4)	9.1 (12)
H24	.608	(3)	.427	(3)	.876	(3)	5.7 (8)
H26	1.176	(2)	.059	(2)	.6988	(19)	2.3 (5)
H27	1.352	(3)	-.098	(3)	.636	(2)	4.3 (6)
H28	1.569	(3)	-.092	(3)	.633	(2)	3.9 (6)
H29	1.622	(3)	.067	(2)	.689	(2)	3.0 (5)
H30	1.449	(2)	.230	(2)	.751	(2)	2.9 (5)

Biso is the Mean of the Principal Axes of the Thermal Ellipsoid

Table of u(i,j) or U values *100.
E.S.Ds. refer to the last digit printed

	u11(U)	u22	u33	u12	u13	u23
O1	3.37(7)	2.99(7)	1.95(6)	-1.88(6)	-.09(5)	-.78(5)
O2	3.04(6)	1.89(6)	1.84(5)	-1.35(5)	.26(5)	-.79(4)
O3	4.23(7)	2.44(6)	1.31(5)	-2.20(6)	.22(5)	-.56(5)
O4	2.33(6)	2.03(6)	2.56(6)	-.77(5)	.17(5)	.41(5)
O5	3.10(7)	2.46(6)	1.94(6)	-.93(5)	.62(5)	-.11(5)
N1	1.93(6)	1.53(6)	1.39(6)	-.96(5)	.18(5)	-.46(5)
N2	1.95(6)	1.66(6)	1.88(6)	-.76(5)	.19(5)	-.36(5)
C1	1.69(7)	1.37(7)	1.64(7)	-.73(5)	.13(5)	-.50(5)
C2	2.00(7)	1.51(7)	1.78(7)	-.76(6)	.19(6)	-.65(6)
C3	1.74(7)	1.91(7)	1.76(7)	-.84(6)	.19(5)	-.68(6)
C4	1.68(7)	1.59(7)	1.76(7)	-.70(6)	-.14(5)	-.44(6)
C5	1.83(7)	1.35(7)	1.49(7)	-.75(6)	.08(5)	-.39(5)
C6	1.98(7)	1.61(7)	1.40(7)	-.97(6)	.01(5)	-.35(5)
C7	1.72(7)	1.99(7)	1.76(7)	-1.02(6)	-.08(5)	-.29(6)
C8	2.19(7)	1.70(7)	1.79(7)	-1.01(6)	-.01(6)	-.34(6)
C9	2.17(7)	1.51(7)	1.66(7)	-.98(6)	.04(6)	-.30(6)
C10	2.34(8)	1.67(7)	1.96(8)	-.83(6)	-.20(6)	-.02(6)
C11	2.00(7)	1.97(8)	1.75(7)	-.78(6)	-.25(6)	-.38(6)
C12	1.88(7)	1.70(7)	1.60(7)	-.73(6)	.27(5)	-.58(6)
C13	3.43(9)	1.85(7)	1.39(7)	-1.57(7)	.25(6)	-.50(6)
C14	3.15(9)	2.74(9)	2.48(9)	-.88(8)	.39(7)	-1.24(7)
C15	3.24(10)	3.00(10)	2.84(9)	-.82(8)	-.50(8)	-.65(8)
C16	4.39(11)	2.89(9)	1.70(8)	-1.61(8)	-.10(7)	-.53(7)
C17	3.59(10)	3.11(10)	1.92(8)	-1.49(8)	.77(7)	-.99(7)
C18	2.74(9)	2.61(9)	2.16(8)	-1.32(7)	.36(7)	-.69(7)
C19	1.74(7)	2.08(8)	1.87(7)	-.89(6)	.19(6)	-.88(6)
C20	2.17(9)	3.17(11)	4.58(12)	-1.25(8)	-.39(8)	1.07(9)
C21	2.35(10)	4.91(13)	5.11(14)	-1.97(10)	.01(9)	.63(11)
C22	1.84(8)	4.18(11)	4.06(11)	-1.01(8)	.25(7)	-1.93(9)
C23	2.31(10)	2.63(10)	7.63(17)	-.13(8)	.62(10)	-1.03(11)
C24	2.35(10)	2.07(9)	7.26(17)	-.50(8)	1.20(10)	-.39(10)
C25	1.98(7)	1.75(7)	2.19(8)	-.68(6)	.20(6)	-.44(6)
C26	2.22(8)	2.64(9)	4.04(11)	-1.11(7)	.12(7)	-1.30(8)
C27	3.18(10)	2.72(10)	5.13(13)	-1.18(8)	.30(9)	-2.08(9)
C28	2.81(10)	2.76(10)	4.92(12)	-.92(8)	1.00(9)	-1.81(9)
C29	2.13(9)	2.86(10)	4.87(12)	-1.13(7)	.67(8)	-1.25(9)
C30	2.24(8)	2.26(8)	3.64(10)	-1.17(7)	.28(7)	-1.04(7)
H1	.4(4)					
H2a	2.1(5)					
H2b	1.3(4)					
H5	1.2(4)					
H6	2.1(5)					
H9	1.8(5)					
H10a	1.8(5)					
H10b	2.5(5)					
H11	3.3(6)					
H14	3.4(6)					
H15	3.6(6)					
H16	4.7(7)					
H17	4.2(7)					
H18	3.4(6)					
H20	5.6(8)					
H21	7.3(10)					
H22	4.9(7)					
H23	11.5(15)					

H24	7.2(10)
H26	2.9(6)
H27	5.5(8)
H28	4.9(8)
H29	3.7(6)
H30	3.6(6)

Anisotropic Temperature Factors are of the form

Temp=-2*Pi*Pi*(h*h*ul1*astar*astar+---+2*h*k*ul2*astar*bstar+---)

DISANG -- The NRCVAX Distance and Angle Program

The Space Group is P -1
 The Equivalent Positions are:

Centrosymmetric

1) x y z

The Lattice is Primitive. There are no Centring Vectors

X98040: -125 DEC C

2-Sep-1998

The following Atoms are from the CD File

* Indicates that there are Symmetry Equivalents of an atom.

Name	x	y	z
O1	.75624 (13)	.20953 (13)	.68250 (10)
O2	.92467 (12)	.37926 (11)	1.07468 (9)
O3	.88429 (13)	.18647 (12)	1.15491 (9)
O4	1.14835 (12)	.13956 (12)	.92367 (10)
O5	1.17818 (13)	.43625 (13)	.59751 (10)
N1	.85914 (13)	.27047 (13)	.96958 (10)
N2	1.18909 (13)	.26629 (13)	.75595 (11)
C1	.79072 (15)	.17461 (15)	.97047 (12)
C2	.83393 (16)	.11428 (15)	.87340 (13)
C3	.80189 (15)	.22157 (16)	.76495 (13)
C4	.83366 (15)	.34256 (15)	.76259 (12)
C5	.87135 (15)	.36885 (15)	.86684 (12)
C6	1.01428 (15)	.37913 (15)	.85430 (12)
C7	1.12164 (15)	.24692 (16)	.85312 (13)
C8	1.13672 (16)	.39815 (16)	.68723 (13)
C9	1.02283 (16)	.47924 (15)	.74335 (12)
C10	.89216 (17)	.53920 (16)	.66656 (14)
C11	.84247 (16)	.42868 (16)	.66607 (13)
C12	.89140 (15)	.28826 (15)	1.06619 (12)
C13	.92087 (18)	.19111 (16)	1.25970 (12)
C14	1.05373 (20)	.13180 (19)	1.29911 (15)
C15	1.08571 (21)	.12766 (20)	1.40687 (16)
C16	.98502 (21)	.18232 (19)	1.47123 (15)
C17	.85253 (20)	.24180 (20)	1.42956 (14)
C18	.81940 (19)	.24692 (18)	1.32266 (14)
C19	.63760 (16)	.23461 (16)	.97107 (13)
C20	.56870 (20)	.15026 (22)	1.02379 (19)
C21	.42898 (21)	.19830 (26)	1.02626 (21)
C22	.35589 (19)	.33268 (22)	.97613 (18)
C23	.42239 (21)	.41582 (23)	.92061 (25)
C24	.56250 (21)	.36807 (21)	.91808 (23)
C25	1.29681 (16)	.16232 (16)	.72562 (14)
C26	1.26857 (19)	.06371 (19)	.69412 (17)
C27	1.37174 (21)	-.03163 (21)	.65867 (19)
C28	1.50143 (21)	-.02882 (21)	.65587 (19)
C29	1.52958 (19)	.06859 (20)	.68984 (18)
C30	1.42695 (18)	.16485 (18)	.72500 (16)
H1	.82447 (168)	.10240 (168)	1.03814 (141)
H2a	.79224 (209)	.05270 (212)	.87383 (171)
H2b	.92591 (205)	.07212 (192)	.88009 (157)
H5	.81324 (191)	.45339 (194)	.86798 (151)
H6	1.03974 (213)	.40225 (210)	.91180 (175)
H9	1.05087 (201)	.54900 (205)	.75414 (165)
H10a	.90833 (201)	.58916 (203)	.59326 (169)

H10b	.82584 (222)	.60780 (221)	.69546 (175)
H11	.82073 (240)	.41371 (240)	.60491 (200)
H14	1.12027 (245)	.09380 (240)	1.25080 (194)
H15	1.17991 (253)	.08910 (247)	1.43641 (201)
H16	1.00756 (266)	.17523 (268)	1.54806 (226)
H17	.78107 (264)	.27836 (259)	1.47160 (215)
H18	.73264 (248)	.28889 (238)	1.28978 (196)
H20	.61629 (296)	.05326 (307)	1.05919 (245)
H21	.37669 (336)	.13696 (335)	1.06092 (269)
H22	.25634 (281)	.36668 (268)	.98145 (220)
H23	.37546 (451)	.51449 (456)	.86322 (355)
H24	.60813 (336)	.42660 (332)	.87552 (273)
H26	1.17561 (235)	.05876 (224)	.69879 (186)
H27	1.35213 (293)	-.09759 (300)	.63572 (238)
H28	1.56895 (291)	-.09235 (285)	.63271 (231)
H29	1.62214 (258)	.06656 (244)	.68887 (205)
H30	1.44929 (246)	.22985 (246)	.75062 (200)

O1-C3	1.2244 (20)	C13-C18	1.381 (3)
O2-C12	1.2098 (19)	C14-C15	1.399 (3)
O3-C12	1.3687 (20)	C14-H14	.97 (2)
O3-C13	1.4125 (19)	C15-C16	1.384 (3)
O4-C7	1.217 (2)	C15-H15	.98 (3)
O5-C8	1.212 (2)	C16-C17	1.382 (3)
N1-C1	1.4774 (20)	C16-H16	.98 (3)
N1-C5	1.4747 (20)	C17-C18	1.388 (3)
N1-C12	1.3627 (20)	C17-H17	.94 (3)
N2-C7	1.393 (2)	C18-H18	.93 (2)
N2-C8	1.400 (2)	C19-C20	1.390 (3)
N2-C25	1.439 (2)	C19-C24	1.393 (3)
C1-C2	1.533 (2)	C20-C21	1.391 (3)
C1-C19	1.526 (2)	C20-H20	.99 (3)
C1-H1	.970 (17)	C21-C22	1.389 (3)
C2-C3	1.499 (2)	C21-H21	1.02 (4)
C2-H2a	.93 (2)	C22-C23	1.372 (3)
C2-H2b	.92 (2)	C22-H22	1.00 (3)
C3-C4	1.484 (2)	C23-C24	1.395 (3)
C4-C5	1.521 (2)	C23-H23	1.08 (4)
C4-C11	1.342 (2)	C24-H24	.97 (3)
C5-C6	1.557 (2)	C25-C26	1.386 (3)
C5-H5	.929 (20)	C25-C30	1.389 (2)
C6-C7	1.514 (2)	C26-C27	1.389 (3)
C6-C9	1.544 (2)	C26-H26	1.00 (2)
C6-H6	.91 (2)	C27-C28	1.385 (3)
C8-C9	1.514 (2)	C27-H27	.94 (3)
C9-C10	1.554 (2)	C28-C29	1.390 (3)
C9-H9	.96 (2)	C28-H28	.92 (3)
C10-C11	1.493 (2)	C29-C30	1.391 (3)
C10-H10a	.98 (2)	C29-H29	.97 (3)
C10-H10b	.98 (2)	C30-H30	.97 (3)
C11-H11	.89 (3)	H2a-H2b	1.51 (3)
C13-C14	1.377 (3)	H10a-H10b	1.55 (3)

C12-O3-C13	116.10 (12)	O3-C12-N1	110.88 (13)
C1-N1-C5	122.13 (12)	O3-C13-C14	119.54 (16)
C1-N1-C12	120.44 (13)	O3-C13-C18	118.11 (16)
C5-N1-C12	116.35 (12)	C14-C13-C18	122.18 (16)
C7-N2-C8	112.56 (14)	C13-C14-C15	118.42 (18)
C7-N2-C25	124.51 (14)	C13-C14-H14	118.1 (14)
C8-N2-C25	122.87 (14)	C15-C14-H14	123.4 (14)
N1-C1-C2	109.72 (12)	C14-C15-C16	120.07 (19)
N1-C1-C19	113.60 (13)	C14-C15-H15	120.4 (14)
N1-C1-H1	105.6 (10)	C16-C15-H15	119.5 (14)
C2-C1-C19	111.47 (13)	C15-C16-C17	120.38 (17)
C2-C1-H1	107.4 (10)	C15-C16-H16	119.6 (16)
C19-C1-H1	108.7 (10)	C17-C16-H16	119.9 (16)
C1-C2-C3	111.19 (13)	C16-C17-C18	120.17 (18)
C1-C2-H2a	110.4 (13)	C16-C17-H17	122.7 (16)
C1-C2-H2b	108.6 (12)	C18-C17-H17	117.1 (16)
C3-C2-H2a	110.1 (13)	C13-C18-C17	118.78 (18)
C3-C2-H2b	106.7 (12)	C13-C18-H18	117.7 (15)
H2a-C2-H2b	109.8 (17)	C17-C18-H18	123.5 (15)
O1-C3-C2	122.72 (15)	C1-C19-C20	118.21 (15)
O1-C3-C4	121.75 (15)	C1-C19-C24	123.64 (15)

C2-C3-C4	115.49 (13)	C20-C19-C24	118.11 (17)
C3-C4-C5	122.61 (13)	C19-C20-C21	121.08 (19)
C3-C4-C11	120.77 (14)	C19-C20-H20	121.7 (17)
C5-C4-C11	116.35 (14)	C21-C20-H20	117.2 (17)
N1-C5-C4	113.33 (12)	C20-C21-C22	120.09 (20)
N1-C5-C6	113.85 (12)	C20-C21-H21	122.0 (19)
N1-C5-H5	108.3 (11)	C22-C21-H21	117.8 (18)
C4-C5-C6	107.54 (12)	C21-C22-C23	119.32 (19)
C4-C5-H5	107.5 (11)	C21-C22-H22	119.3 (16)
C6-C5-H5	105.9 (12)	C23-C22-H22	121.4 (16)
C5-C6-C7	112.16 (13)	C22-C23-C24	120.7 (2)
C5-C6-C9	111.62 (13)	C22-C23-H23	125 (2)
C5-C6-H6	111.8 (13)	C24-C23-H23	112 (2)
C7-C6-C9	105.07 (13)	C19-C24-C23	120.6 (2)
C7-C6-H6	105.9 (13)	C19-C24-H24	119.3 (19)
C9-C6-H6	110.0 (13)	C23-C24-H24	120.0 (19)
O4-C7-N2	123.78 (15)	N2-C25-C26	119.58 (15)
O4-C7-C6	127.44 (15)	N2-C25-C30	119.38 (15)
N2-C7-C6	108.78 (13)	C26-C25-C30	121.01 (16)
O5-C8-N2	123.61 (16)	C25-C26-C27	119.20 (17)
O5-C8-C9	127.59 (15)	C25-C26-H26	121.0 (13)
N2-C8-C9	108.79 (13)	C27-C26-H26	119.8 (13)
C6-C9-C8	104.76 (13)	C26-C27-C28	120.33 (18)
C6-C9-C10	113.89 (13)	C26-C27-H27	119.2 (18)
C6-C9-H9	111.9 (12)	C28-C27-H27	120.5 (18)
C8-C9-C10	109.08 (13)	C27-C28-C29	120.15 (19)
C8-C9-H9	105.5 (12)	C27-C28-H28	119.8 (18)
C10-C9-H9	111.1 (12)	C29-C28-H28	120.1 (18)
C9-C10-C11	108.71 (13)	C28-C29-C30	119.98 (18)
C9-C10-H10a	110.2 (12)	C28-C29-H29	118.3 (15)
C9-C10-H10b	107.8 (12)	C30-C29-H29	121.7 (15)
C11-C10-H10a	114.2 (12)	C25-C30-C29	119.30 (17)
C11-C10-H10b	111.5 (13)	C25-C30-H30	122.0 (15)
H10a-C10-H10b	104.2 (17)	C29-C30-H30	118.6 (15)
C4-C11-C10	119.29 (15)	C2-H2a-H2b	34.8 (11)
C4-C11-H11	116.7 (15)	C2-H2b-H2a	35.4 (11)
C10-C11-H11	123.9 (15)	C10-H10a-H10b	38.0 (11)
O2-C12-O3	123.31 (14)	C10-H10b-H10a	37.8 (11)
O2-C12-N1	125.79 (15)		

C13	O3	C12	O2	-1.1(2)	C13	O3	C12	N1	177.6(4)
C12	O3	C13	C14	-88.1(4)	C12	O3	C13	C18	96.7(4)
C5	N1	C1	C2	43.8(2)	C5	N1	C1	C19	-81.7(3)
C5	N1	C1	H1	159. (2)	C12	N1	C1	C2	-148.5(4)
C12	N1	C1	C19	86.0(3)	C12	N1	C1	H1	-33. (2)
C1	N1	C5	C4	-11.81(19)	C1	N1	C5	C6	-135.1(4)
C1	N1	C5	H5	107. (3)	C12	N1	C5	C4	-180.0(4)
C12	N1	C5	C6	56.7(3)	C12	N1	C5	H5	-61. (3)
C1	N1	C12	O2	-165.9(4)	C1	N1	C12	O3	15.41(19)
C5	N1	C12	O2	2.46(19)	C5	N1	C12	O3	-176.2(4)
C8	N2	C7	O4	178.4(5)	C8	N2	C7	C6	-1.91(20)
C25	N2	C7	O4	1.1(2)	C25	N2	C7	C6	-179.2(4)
C7	N2	C8	O5	-178.2(5)	C7	N2	C8	C9	1.1(2)
C25	N2	C8	O5	-.8(2)	C25	N2	C8	C9	178.5(4)
C7	N2	C25	C26	71.4(3)	C7	N2	C25	C30	-110.6(4)
C8	N2	C25	C26	-105.7(4)	C8	N2	C25	C30	72.3(3)
N1	C1	C2	C3	-58.3(3)	N1	C1	C2	H2a	179. (3)
N1	C1	C2	H2b	59. (3)	C19	C1	C2	C3	68.4(3)
C19	C1	C2	H2a	-54. (3)	C19	C1	C2	H2b	-174. (3)
H1	C1	C2	C3	-173. (2)	H1	C1	C2	H2a	65. (4)
H1	C1	C2	H2b	-55. (4)	N1	C1	C19	C20	-149.2(5)
N1	C1	C19	C24	33.4(3)	C2	C1	C19	C20	86.2(4)
C2	C1	C19	C24	-91.2(4)	H1	C1	C19	C20	-32. (2)
H1	C1	C19	C24	151. (2)	C1	C2	C3	O1	-138.9(4)
C1	C2	C3	C4	43.2(2)	H2a	C2	C3	O1	-16. (3)
H2a	C2	C3	C4	166. (3)	H2b	C2	C3	O1	103. (3)
H2b	C2	C3	C4	-75. (3)	C1	C2	H2a	H2b	-120. (3)
C3	C2	H2a	H2b	117. (3)	H2b	C2	H2a	H2b	0. (3)
C1	C2	H2b	H2a	121. (3)	C3	C2	H2b	H2a	-119. (3)
H2a	C2	H2b	H2a	0. (4)	O1	C3	C4	C5	171.1(5)
O1	C3	C4	C11	-15.1(2)	C2	C3	C4	C5	-10.94(19)
C2	C3	C4	C11	162.8(5)	C3	C4	C5	N1	-6.02(18)
C3	C4	C5	C6	120.7(4)	C3	C4	C5	H5	-126. (3)
C11	C4	C5	N1	179.9(4)	C11	C4	C5	C6	-53.3(3)
C11	C4	C5	H5	60. (3)	C3	C4	C11	C10	-172.0(4)
C3	C4	C11	H11	6. (3)	C5	C4	C11	C10	2.15(20)
C5	C4	C11	H11	180. (4)	N1	C5	C6	C7	60.1(3)
N1	C5	C6	C9	177.7(4)	N1	C5	C6	H6	-59. (3)
C4	C5	C6	C7	-66.3(3)	C4	C5	C6	C9	51.3(2)
C4	C5	C6	H6	175. (3)	H5	C5	C6	C7	179. (3)
H5	C5	C6	C9	-63. (3)	H5	C5	C6	H6	60. (4)
C5	C6	C7	O4	-57.0(3)	C5	C6	C7	N2	123.3(4)
C9	C6	C7	O4	-178.4(5)	C9	C6	C7	N2	1.89(18)
H6	C6	C7	O4	65. (3)	H6	C6	C7	N2	-115. (3)
C5	C6	C9	C8	-123.0(4)	C5	C6	C9	C10	-3.91(19)
C5	C6	C9	H9	123. (3)	C7	C6	C9	C8	-1.21(19)
C7	C6	C9	C10	117.9(4)	C7	C6	C9	H9	-115. (3)
H6	C6	C9	C8	112. (3)	H6	C6	C9	C10	-129. (3)
H6	C6	C9	H9	-2. (4)	O5	C8	C9	C6	179.4(5)
O5	C8	C9	C10	57.1(3)	O5	C8	C9	H9	-62. (3)
N2	C8	C9	C6	.17(18)	N2	C8	C9	C10	-122.1(4)
N2	C8	C9	H9	118. (3)	C6	C9	C10	C11	-44.3(2)
C6	C9	C10	H10a	-170. (3)	C6	C9	C10	H10b	77. (3)
C8	C9	C10	C11	72.3(3)	C8	C9	C10	H10a	-54. (3)
C8	C9	C10	H10b	-167. (3)	H9	C9	C10	C11	-172. (3)

H9	C9	C10	H10a	62. (4)
C9	C10	C11	C4	47.8 (3)
H10a	C10	C11	C4	171. (3)
H10b	C10	C11	C4	-71. (3)
C9	C10	H10a	H10b	-115. (3)
H10b	C10	H10a	H10b	0. (4)
C11	C10	H10b	H10a	-124. (4)
O3	C13	C14	C15	-174.6 (5)
C18	C13	C14	C15	.5 (3)
O3	C13	C18	C17	174.5 (5)
C14	C13	C18	C17	-.6 (3)
C13	C14	C15	C16	.0 (2)
H14	C14	C15	C16	-179. (3)
C14	C15	C16	C17	-.3 (3)
H15	C15	C16	C17	177. (3)
C15	C16	C17	C18	.1 (3)
H16	C16	C17	C18	-177. (4)
C16	C17	C18	C13	.3 (2)
H17	C17	C18	C13	-178. (4)
C1	C19	C20	C21	-179.3 (5)
C24	C19	C20	C21	-1.7 (3)
C1	C19	C24	C23	179.0 (6)
C20	C19	C24	C23	1.6 (3)
C19	C20	C21	C22	-.4 (2)
H20	C20	C21	C22	-179. (4)
C20	C21	C22	C23	2.6 (3)
H21	C21	C22	C23	-175. (4)
C21	C22	C23	C24	-2.7 (3)
H22	C22	C23	C24	177. (4)
C22	C23	C24	C19	.6 (2)
H23	C23	C24	C19	-168. (5)
N2	C25	C26	C27	176.2 (5)
C30	C25	C26	C27	-1.7 (3)
N2	C25	C30	C29	-176.5 (5)
C26	C25	C30	C29	1.4 (3)
C25	C26	C27	C28	.6 (3)
H26	C26	C27	C28	-178. (3)
C26	C27	C28	C29	.8 (3)
H27	C27	C28	C29	-180. (4)
C27	C28	C29	C30	-1.1 (3)
H28	C28	C29	C30	-180. (4)
C28	C29	C30	C25	.0 (3)
H29	C29	C30	C25	-179. (3)
C2	H2a	H2b	C2	.0 (3)

H9	C9	C10	H10b	-51. (4)
C9	C10	C11	H11	-130. (4)
H10a	C10	C11	H11	-6. (4)
H10b	C10	C11	H11	112. (5)
C11	C10	H10a	H10b	122. (4)
C9	C10	H10b	H10a	117. (4)
H10a	C10	H10b	H10a	0. (3)
O3	C13	C14	H14	5. (3)
C18	C13	C14	H14	180. (3)
O3	C13	C18	H18	-8. (3)
C14	C13	C18	H18	177. (3)
C13	C14	C15	H15	-178. (3)
H14	C14	C15	H15	3. (5)
C14	C15	C16	H16	177. (4)
H15	C15	C16	H16	-5. (5)
C15	C16	C17	H17	179. (4)
H16	C16	C17	H17	1. (5)
C16	C17	C18	H18	-177. (3)
H17	C17	C18	H18	4. (5)
C1	C19	C20	H20	-1. (4)
C24	C19	C20	H20	176. (4)
C1	C19	C24	H24	3. (4)
C20	C19	C24	H24	-175. (4)
C19	C20	C21	H21	177. (4)
H20	C20	C21	H21	-2. (6)
C20	C21	C22	H22	-177. (4)
H21	C21	C22	H22	6. (5)
C21	C22	C23	H23	165. (5)
H22	C22	C23	H23	-15. (6)
C22	C23	C24	H24	177. (4)
H23	C23	C24	H24	8. (7)
N2	C25	C26	H26	-6. (3)
C30	C25	C26	H26	176. (3)
N2	C25	C30	H30	6. (3)
C26	C25	C30	H30	-176. (3)
C25	C26	C27	H27	-179. (4)
H26	C26	C27	H27	3. (5)
C26	C27	C28	H28	180. (4)
H27	C27	C28	H28	-1. (6)
C27	C28	C29	H29	178. (3)
H28	C28	C29	H29	-1. (5)
C28	C29	C30	H30	178. (3)
H29	C29	C30	H30	-1. (5)
C10	H10a	H10b	C10	.0 (3)