Alkyne-Linked 2,2-Disubstituted-indolin-3-one Oligomers as Extended β-Strand Mimetics

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Supporting Information

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Part I. General Experimental Section

General Methods. All chemicals were obtained from Sigma/Aldrich, Fluka, J.T.Baker, Alfa Aesar and TCI America unless otherwise noted. All air and/or moisture sensitive reactions were carried out under a positive pressure of nitrogen in flame-dried glassware. Solvents tetrahydrofuran (THF), dimethylformamide (DMF) and dichloromethane (DCM) were obtained from commercial sources and dried on an Innovative Technology SPS-400 dry solvent system. Column chromatography was performed using silica gel (230-400 mesh) from Solvent technologies. ^{1}H-NMR and ^{13}C-NMR spectra were recorded either on a Bruker Avance DPX-500 or DPX-400 spectrometer at room temperature. Chemical shifts are expressed in parts per million (ppm) and are referenced to the internal solvent signals. All high-resolution mass spectra (HRMS) were obtained from the Mass Spectrometry Laboratory at the University of Illinois at Urbana-Champaign on a Micromass Q-Tof Ultima quadrupole time of flight mass spectrometer.

Part II. Synthesis

4-((2,2-Dimethyl-3-oxoindolin-7-yl)ethynyl)-7-((7-methoxy-2,2-dimethyl-3-oxoindolin-4-yl)ethynyl)-2,2-dimethylindolin-3-one (1): To a solution of **12** (0.027 g, 0.039 mmol) in MeOH (4 mL) was added a 20% KOH solution in MeOH (0.230 mL) and the reaction was stirred at room temperature overnight. The solvent was evaporated under reduced pressure and the residue was taken up in EtOAc (10 mL), washed with brine and dried with $MgSO_4$. The solvent was removed under reduced pressure and the crude product was purified by flash column chromatography (3:7 EtOAc:Hexane) to yield an orange solid (0.007 g, 32%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.35 (s, 1H), 8.31 (s, 1H), 7.60 (d, *J* = 7.6 Hz, 1H), 7.55 (dd, *J* = 7.4, 1.2 Hz, 1H), 7.48 (d, *J* = 7.7 Hz, 1H), 6.97-6.91 (m, 2H), 6.82 (d, *J* = 7.7 Hz, 1H), 6.69 (t, *J* = 7.5 Hz, 1H), 4.87 (s, 1H), 3.94 (s, 3H), 1.50 (s, 6H), 1.49 (s, 6H), 1.40 (s, 6H); ^{13}C NMR (500 MHz, $CDCl_3$) δ 205.4, 204.0, 203.7, 161.8, 161.7, 151.7, 146.4, 137.8, 136.5, 126.1, 122.6, 120.8, 119.9, 118.3, 118.1, 116.6, 116.4, 115.1, 111.8, 105.8, 105.5, 95.2, 94.3, 92.8, 89.1, 64.9, 64.7, 64.5, 55.7, 24.7, 24.3, 24.1. HRMS (ESI) *m/z* calculated for $C_{35}H_{31}N_3O_4H^+$ 558.2393. Found 558.2374.

4-((2,2-Dimethyl-3-oxoindolin-7-yl)ethynyl)-7-methoxy-2,2-dimethylindolin-3-one (2): To a solution of **9** (0.012 g, 0.026 mmol) in MeOH (2 mL) was added a 20% KOH solution in MeOH (1 mL) and the reaction was stirred at room temperature overnight. The solvent was evaporated under reduced pressure and the residue was taken up in EtOAc (10 mL), washed with brine and dried with $MgSO_4$. The solvent was removed under reduced pressure and the crude product was purified by flash column chromatography (3:7 EtOAc:Hexane) to yield an orange solid (0.007 g, 71%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.08 (s, 1H), 7.57 (d, *J* = 7.6 Hz, 1H), 7.51 (dd, *J* = 7.3, 1

Hz, 1H), 6.96-6.90 (m, 2H), 6.68 (t, *J* = 7.5 Hz, 1H), 4.84 (s, 1H), 3.93 (s, 3H), 1.46 (s, 6H), 1.39 (s, 6H); ^{13}C NMR (500 MHz, $CDCl_3$) δ 205.5, 203.6, 161.5, 151.6, 146.3, 137.2, 125.3, 122.5, 118.2, 118.1, 116.7, 115.0, 112.1, 106.2, 93.5, 89.1, 64.8, 64.4, 55.6, 24.7, 24.1. HRMS (ESI) *m/z* calculated for $C_{23}H_{22}N_2O_3H^+$ 375.1709. Found 375. 1718.

1-Acetyl-4-bromo-7-methoxy-2-methyl-1H-indol-3-yl acetate (3): To a solution of 2-amino-6-bromo-3-methoxybenzoic acid (4.65 g, 0.0189 mol) in water (12 mL) was added NaOH (0.831 g, 0.0208 mol) followed by anhydrous Na_2CO_3 (2.20 g, 0.0208 mmol). A solution of methyl 2-bromopropionate (2.32 mL, 0.0208 mol) in MeOH (12 mL) was added and the mixture stirred at 80 ^{o}C overnight. A solution of NaOH (1.51 g, 0.0378 mol) in water (6 mL) was added and the mixture refluxed for 1.5 h. The solution was cooled to room temperature and the methanol evaporated. Concentrated HCl was added dropwise until the solution reached pH 3 and the aqueous solution was extracted with EtOAc (3 X 30 mL). The combined organic layers were washed with water, brine and dried with $MgSO_4$ and the solvent evaporated under reduced pressure to yield crude 6-bromo-2-(1-carboxyethylamino)-3-methoxybenzoic acid which was used in the following reaction without further purification.

A suspension of 6-bromo-2-(1-carboxyethylamino)-3-methoxybenzoic acid and NaOAc (6.20 g, 0.0756 mol) in acetic anhydride (20 mL) was refluxed for 1 h. The solution was cooled to room temperature and the solvent was evaporated under reduced pressure. The residue was diluted with $CHCl_3$ (100 mL) and washed with water, saturated $NaHCO_3$ solution, and brine and the organic layer was dried with $MgSO_4$. The solvent was removed under reduced pressure and the residue was purified by flash column chromatography (2:8 EtOAc:Hexane) to yield a colorless solid (1.61 g, 25% over two steps). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.25 (d, *J* = 8.5 Hz, 1H), 6.59

(d, J = 8.5 Hz, 1H), 3.90 (s, 3H), 2.52 (s, 3H), 2.39 (s, 3H), 2.29 (s, 3H); ^{13}C NMR (400 MHz, $CDCl_3$) δ 172.9, 169.7, 146.3, 129.0, 127.1, 123.5, 123.4, 106.2, 102.1, 55.8, 28.1, 20.8, 10.6. HRMS (ESI) *m/z* calculated for $C_{14}H_{14}BrNO_4H^+$ 340.0184. Found 340.0183.

1-Acetyl-4-bromo-7-methoxy-2-methylindolin-3-one (4): To a boiling solution of **3** (0.892 g, 2.62 mmol) in EtOH (6 mL) was added a hot solution of Na_2SO_3 (0.364 g, 2.88 mmol) in water (12 mL). The solution was refluxed overnight, cooled to room temperature and the EtOH was removed under reduced pressure. The aqueous solution was extracted with $CHCl_3$ (3 X 10 mL) and the organic extracts were washed with water, brine, dried with $MgSO_4$ and evaporated. The residue was purified by flash column chromatography (4:6 EtOAc:Hexane) to afford a yellow solid (0.393 g, 50%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.35 (d, J = 8.6 Hz, 1H), 7.06 (d, J = 8.6 Hz, 1H), 4.48 (q, J = 7.1 Hz, 1H), 3.94 (s, 3H), 2.28 (s, 3H), 1.55 (d, J = 7.1 Hz, 3H); ^{13}C NMR (500 MHz, $CDCl_3$) δ 197.4, 169.8, 149.2, 143.8, 129.9, 124.2, 119.9, 109.3, 64.7, 56.0, 23.7, 18.2. HRMS (ESI) *m/z* calculated for $C_{12}H_{12}BrNO_3H^+$ 298.0079. Found 298.0068.

1-Acetyl-4-bromo-7-methoxy-2,2-dimethylindolin-3-one (5): To a suspension of NaH (60%, 0.290 g, 7.25 mmol) in dry THF (15 mL) was added a solution of **4** (1.08 g, 3.62 mmol) in dry THF (25 mL) at room temperature. Freshly distilled methyl iodide (4.5 mL, 72.5 mmol) was added after 30 min, and the reaction stirred at room temperature for 30 min. The mixture was carefully quenched with water and extracted with EtOAc (3 X 15 mL). The combined organic extracts were washed with brine, dried with $MgSO_4$, and concentrated. The residue was purified by flash column chromatography (4:6 EtOAc:Hexane) to yield a yellow solid (0.587 g, 52%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30 (d, J = 8.5 Hz, 1H), 7.03 (d, J = 8.5 Hz, 1H), 3.91 (s, 4H), 2.23

(s, 3H), 1.56 (s, 6H); ^{13}C NMR (400 MHz, $CDCl_3$) δ 199.7, 171.0, 147.9, 144.1, 128.7, 122.2, 119.2, 110.1, 71.1, 56.0, 26.1, 23.4. HRMS (ESI) *m/z* calculated for $C_{13}H_{14}BrNO_3H^+$ 312.0235. Found 312.0247.

1-Acetyl-7-methoxy-2,2-dimethyl-4-((trimethylsilyl)ethynyl)indolin-3-one (6): To a solution of **5** (0.157 g, 0.503 mmol) in dry triethylamine (5 mL) was added CuI (0.010 g, 0.0503 mmol) and $PdCl_2(PPh_3)_2$ (0.035 g, 0.0503 mmol). The reaction was purged three times under high vacuum to provide a complete nitrogen atmosphere. Ethynyl-trimethyl-silane (0.209 mL, 1.51 mmol) was added to the reaction mixture and the reaction stirred at 80 ^{o}C overnight. The reaction was diluted with EtOAc (5 mL), filtered over Celite, washed several times with EtOAc, and the solvent was removed under reduced pressure. The crude product was purified by flash column chromatography (2:8 EtOAc:Hexane) to yield a yellow oil (0.135 g, 81%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.28 (d, *J* = 8.3 Hz, 1H), 7.11 (d, *J* = 8.3 Hz, 1H), 3.94 (s, 3H), 2.23 (s, 3H), 1.56 (s, 6H), 0.30 (s, 9H); ^{13}C NMR (500 MHz, $CDCl_3$) δ 200.0, 171.0, 148.6, 142.2, 130.1, 124.7, 117.8, 112.7, 100.6, 100.2, 70.7, 55.8, 26.0, 23.3, -0.1. HRMS (ESI) *m/z* calculated for $C_{18}H_{23}NO_3SiH^+$ 330.1525. Found 330.1522.

1-Acetyl-4-ethynyl-7-methoxy-2,2-dimethylindolin-3-one (7): Anhydrous K_2CO_3 (0.052 g, 0.379 mmol) and **6** (0.125 g, 0.379 mmol) were suspended in MeOH (2 mL) and stirred at room temperature for 1 h. The solvent was removed under reduced pressure and the residue was dissolved in DCM (10 mL), washed with brine and the organic layer was dried over $MgSO_4$. The solvent was removed under reduced pressured and the crude product was purified by flash column chromatography (3:7 EtOAc:Hexane) to yield a yellow solid (0.080 g, 82%). ^{1}H NMR

(400 MHz, $CDCl_3$) δ 7.32 (d, *J* = 8.3 Hz, 1H), 7.12 (d, *J* = 8.3 Hz, 1H), 3.94 (s, 3H), 3.40 (s, 1H), 2.24 (s, 3H), 1.56 (s, 6H); ^{13}C NMR (400 MHz, $CDCl_3$) δ 200.4, 171.0, 148.9, 142.5, 130.4, 124.8, 117.9, 111.5, 82.0, 79.5, 70.8, 55.9, 26.1, 23.3. HRMS (ESI) *m/z* calculated for $C_{15}H_{15}NO_3H^+$ 258.1130. Found 258.1139.

1-Acetyl-2,2-dimethyl-3-oxoindolin-7-yl trifluoromethanesulfonate (8): To a solution of **17** (0.570 g, 2.60 mmol) in DCM (25 mL) was added anhydrous diisopropylethylamine (0.679 mL, 3.90 mmol) and the resulting mixture was cooled to 0 ^oC. Trifilic anhydride (0.524 mL, 3.12 mmol) was added dropwise and the mixture was stirred for 10 min at 0 ^oC after which the reaction was quenched with water, warmed to room temperature and extracted with DCM (3 X 10 mL). The organic layers were washed with brine, dried with $MgSO_4$ and concentrated under reduced pressure. The crude product was purified by flash column chromatography (3:7 EtOAc:Hexane) to yield a yellow oil (0.870 g, 95%). ^1H NMR (400 MHz, $CDCl_3$) δ 7.76 (dd, *J* = 8, 1.1 Hz, 1H), 7.52 (dd, *J* = 8, 1.1 Hz, 1H), 7.25 (t, *J* = 8 Hz, 1H), 2.34 (s, 3H), 1.54 (s, 6H); ^{13}C NMR (400 MHz, $CDCl_3$) δ 200.0, 169.1, 144.0, 138.0, 130.9, 126.7, 125.1, 124.8, 123.2, 120.0, 116.8, 113.6, 69.3, 24.9, 23.3. HRMS (ESI) *m/z* calculated for $C_{13}H_{12}F_3NO_5SH^+$ 352.0467. Found 352.0482.

1-Acetyl-4-((1-acetyl-2,2-dimethyl-3-oxoindolin-7-yl)ethynyl)-7-methoxy-2,2-dimethylindolin-3-one (9): A mixture of **7** (0.050 g, 0.194 mmol), **8** (0.082 g, 0.233 mmol), CuI (0.004 g, 0.019 mmol), $PdCl_2(PPh_3)_2$ (0.008 g, 0.012 mmol) and $^iPr_2NH$ (0.041 mL, 0.292 mmol) in deoxygenated DMF (2 mL) was stirred at 60 ^oC for 6 h. The reaction was cooled to room temperature, water was added to the mixture and the solution was extracted with DCM (3 X 10

mL). The organic fractions were washed with brine, dried over $MgSO_4$, and evaporated under reduced pressure. The crude residue was purified by flash column chromatography (1:1 EtOAc:Hexane) to give a yellow solid (0.049 g, 55%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.04 (dd, *J* = 7.6, 1.3 Hz, 1H), 7.78 (dd, *J* = 7.6, 1.3 Hz, 1H), 7.34 (d, *J* = 8.3 Hz, 1H), 7.25 (t, *J* = 7.6 Hz, 1H), 7.16 (d, *J* = 8.3 Hz, 1H), 3.97 (s, 3H), 2.50 (s, 3H), 2.26 (s, 3H), 1.61 (s, 6H), 1.58 (s, 6H); ^{13}C NMR (500 MHz, $CDCl_3$) δ 201.5, 200.4, 171.0, 170.5, 152.5, 148.9, 142.6, 142.1, 129.1, 125.1, 124.2, 124.0, 118.1, 113.0, 112.1, 93.7, 90.8, 70.8, 70.5, 56.0, 26.4, 26.2, 23.3. HRMS (ESI) *m/z* calculated for $C_{27}H_{26}N_2O_5H^+$ 459.1920. Found 459.1915.

1-Acetyl-4-((1-acetyl-2,2-dimethyl-3-oxoindolin-7-yl)ethynyl)-7-hydroxy-2,2-dimethylindolin-3-one (10): To a solution of **9** (0.079 g, 0.172 mmol) in DCM (2 mL) at -78 ^{o}C was added a solution of 1M BBr_3 in DCM (0.861 mL). The solution was allowed to warm up to room temperature over 3 hrs after which it was quenched with saturated $NaHCO_3$ solution. The mixture was extracted with DCM (3 X 10 mL), washed with brine and dried with $MgSO_4$, and the solvent was removed under reduced pressure. The crude product was purified by flash column chromatography (3:7 EtOAc:Hexane) to give a yellow solid (0.058 g, 76%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 11.39 (s, 1H), 8.03 (dd, *J* = 7.6, 1.3 Hz, 1H), 7.78 (dd, *J* = 7.6, 1.3 Hz, 1H), 7.43 (d, *J* = 8.3 Hz, 1H), 7.29-7.23 (m, 2H), 2.56 (s, 3H), 2.51 (s, 3H), 1.70 (s, 6H), 1.62 (s, 6H); ^{13}C NMR (500 MHz, $CDCl_3$) δ 201.5, 198.3, 171.2, 170.3, 152.5, 148.2, 141.9, 138.8, 132.2, 126.8, 125.0, 124.2, 123.9, 123.9, 113.1, 110.9, 94.1, 90.7, 70.4, 68.0, 26.4, 24.7, 24.2, 23.3. HRMS (ESI) *m/z* calculated for $C_{26}H_{24}N_2O_5H^+$ 445.1763. Found 445.1747.

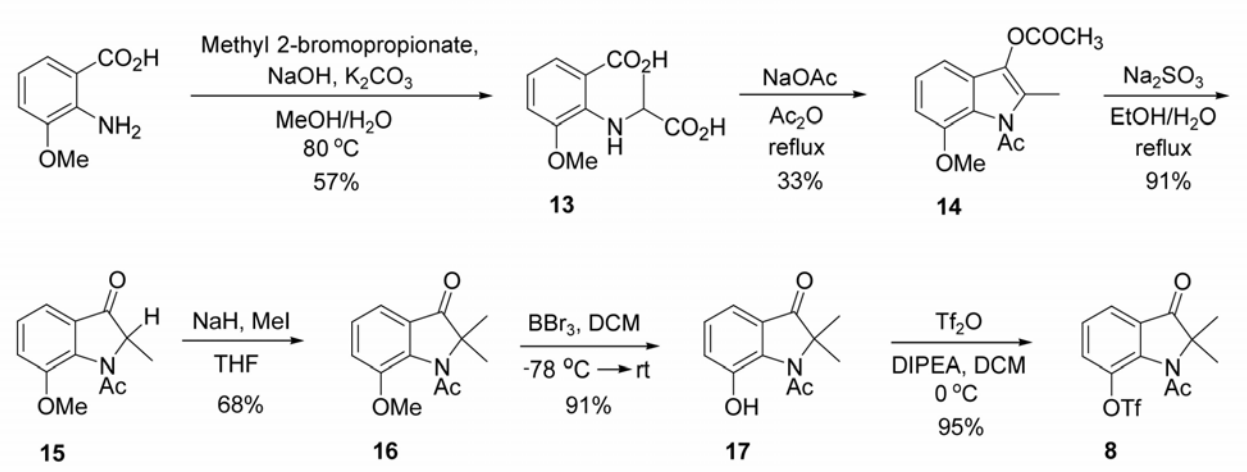
1-Acetyl-4-((1-acetyl-2,2-dimethyl-3-oxoindolin-7-yl)ethynyl)-2,2-dimethyl-3-oxoindolin-7-yl trifluoromethanesulfonate (11): A solution of **10** (0.109 g, 0.245 mmol) and

diisopropylethylamine (0.031 mL, 0.179 mmol) in DCM (2 mL) was cooled to 0 ^{o}C. Triflic anhydride (0.024 mL, 0.143 mmol) was added dropwise and the solution was stirred at 0 ^{o}C for 1 hr after which it was quenched with water. The mixture was extracted with DCM (3 X 10 mL), washed with brine and dried with $MgSO_4$, and the solvent was evaporated under reduced pressure. The crude product was purified by flash column chromatography (3:7 EtOAc:Hexane) to give a yellow solid (0.105 g, 74%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.04 (dd, *J* = 7.6, 1.3 Hz, 1H), 7.82 (dd, *J* = 7.6, 1.3 Hz, 1H), 7.53 (d, *J* = 8.4 Hz, 1H), 7.40 (d, *J* = 8.4 Hz, 1H), 7.27 (t, *J* = 7.6 Hz, 1H), 2.48 (s, 3H), 2.40 (s, 3H), 1.61 (s, 12H); ^{13}C NMR (400 MHz, $CDCl_3$) δ 201.1, 198.3, 170.0, 169.1, 152.9, 144.7, 142.3, 137.3, 130.3, 128.6, 126.0, 125.5, 124.2, 124.0, 121.1, 112.3, 94.6, 92.6, 70.0, 69.3, 26.2, 25.0, 23.6, 23.4. HRMS (ESI) *m/z* calculated for $C_{27}H_{23}F_3N_2O_7SH^+$ 577.1256. Found 577.1266.

1-Acetyl-4-((1-acetyl-2,2-dimethyl-3-oxoindolin-7-yl)ethynyl)-7-((1-acetyl-7-methoxy-2,2-dimethyl-3-oxoindolin-4-yl)ethynyl)-2,2-dimethylindolin-3-one (12): A mixture of **11** (0.044 g, 0.076 mmol), **7** (0.024 g, 0.092 mmol), CuI (0.0015 g, 0.0076 mmol), $PdCl_2(PPh_3)_2$ (0.003 g, 0.0046 mmol) and $^{i}Pr_2NH$ (0.016 mL, 0.114 mmol) in deoxygenated DMF (1 mL) was stirred at 60 ^{o}C overnight. The reaction was cooled to room temperature, diluted with water and extracted with EtOAc (3 X 5 mL). The organic solution was washed with brine, dried with $MgSO_4$ and the solvent was evaporated under reduced pressure. The crude product was purified by flash column chromatography (1:1 EtOAc:Hexane) to yield a yellow solid (0.031 g, 59%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.08 (dd, *J* = 7.6, 1.3 Hz, 1H), 7.97 (d, *J* = 8.0 Hz, 1H), 7.82 (dd, *J* = 7.6, 1.3 Hz, 1H), 7.35 (d, *J* = 8.0 Hz, 2H), 7.6 (t, *J* = 7.6 Hz, 1H), 7.17 (d, *J* = 8.4 Hz, 1H), 3.97 (s, 3H), 2.51 (s, 3H), 2.50 (s, 3H), 2.27 (s, 3H), 1.64 (s, 6H), 1.63 (s, 6H), 1.59 (s, 6H); ^{13}C NMR (400

MHz, $CDCl_3$) δ 201.3, 200.4, 199.6, 171.0, 170.6, 170.2, 153.2, 152.8, 149.0, 142.7, 142.4, 140.8, 129.1, 127.5, 125.7, 124.2, 124.1, 124.0, 123.2, 120.6, 118.1, 112.9, 112.6, 111.9, 95.5, 94.6, 94.1, 90.7, 70.8, 70.3, 70.2, 56.0, 26.5, 26.3, 26.1, 23.5, 23.4, 23.3. HRMS (ESI) *m/z* calculated for $C_{41}H_{37}N_3O_7H^+$ 684.2710. Found 684.2693.

Scheme 1. Synthesis of **8**



2-(1-Carboxyethylamino)-3-methoxybenzoic acid (13): To a solution of 2-amino-3-methoxybenzoic acid (11.50 g, 0.0688 mol) in water (30 mL) was added NaOH (3.03 g, 0.0757 mol) followed by anhydrous K_2CO_3 (10.45 g, 0.0757 mol). A solution of methyl 2-bromopropionate (7.67 mL, 0.0688 mol) in MeOH (30 mL) was added and the mixture stirred at 80 ^oC overnight. A solution of NaOH (5.5 g, 0.1376 mol) in water (15 mL) was added and the mixture refluxed for 1.5 h. The solution was cooled to room temperature and the methanol evaporated. Concentrated HCl was added dropwise until the solution reached pH 3 and the aqueous solution was extracted with EtOAc (3 X 50 mL). The combined organic layers were washed with water, brine and dried with $MgSO_4$ and the solvent evaporated under reduced

pressure. The crude product was recrystallized from ethyl acetate-petroleum ether to yield a pale yellow solid (9.4 g, 57 %). ^{1}H NMR (500 MHz, $(CD_3)_2CO$) δ 7.58 (dd, *J* = 8.0 Hz, 1.4 Hz, 1H), 7.07 (dd, *J* = 8.0 Hz, 1.4 Hz, 1H), 6.72 (t, *J* = 8.0 Hz, 1H), 4.68 (q, *J* = 7.1 Hz, 1H), 3.80 (s, 3H), 1.40 (d, *J* = 7.1 Hz, 3H); ^{13}C NMR (500 MHz, $(CD_3)_2CO$) δ 176.6, 171.4, 152.0, 143.6, 125.7, 119.0, 118.2, 116.7, 57.1, 55.6, 21.0. HRMS (ESI) *m/z* calculated for $C_{11}H_{13}NO_5H^+$ 240.0872. Found 240.0872.

1-Acetyl-7-methoxy-2-methyl-1H-indol-3-yl acetate (14): A suspension of **13** (5.03 g, 0.0210 mmol) and NaOAc (6.90 g, 0.0841 mol) in acetic anhydride (30 mL) was refluxed for 1 h. The solution was cooled to room temperature and the solvent was evaporated under reduced pressure. The residue was diluted with $CHCl_3$ (150 mL) and washed with water, saturated $NaHCO_3$ solution and brine, and the organic layer was dried with $MgSO_4$. The solvent was removed under reduced pressure and the residue was purified by flash column chromatography (2:8 EtOAc:Hexane) to yield a colorless solid (1.79 g, 33%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.16 (t, *J* = 7.9 Hz, 1H), 6.93 (dd, *J* = 7.9 Hz, 0.8 Hz, 1H), 6.75 (d, *J* = 7.9 Hz, 1H), 3.93 (s, 3H), 2.56 (s, 3H), 2.38 (s, 3H), 2.34 (s, 3H); ^{13}C NMR (400 MHz, $CDCl_3$) δ 172.9, 168.5, 146.7, 130.2, 127.0, 125.5, 123.4, 122.1, 109.5, 105.2, 55.1, 27.7, 20.1, 10.8. HRMS (ESI) *m/z* calculated for $C_{14}H_{15}NO_4Na^+$ 284.0899. Found 284.0898.

1-Acetyl-7-methoxy-2-methylindolin-3-one (15): To a boiling solution of **14** (1.79 g, 6.85 mmol) in EtOH (15 mL) was added a hot solution of Na_2SO_3 (0.950 g, 7.54 mmol) in water (30 mL). The solution was refluxed for 15 min, cooled to room temperature and the EtOH was removed under reduced pressure. The aqueous solution was extracted with $CHCl_3$ (3 X 15 mL)

and the organic extracts were washed with water, brine, dried with $MgSO_4$ and evaporated. The residue was purified by flash column chromatography (4:6 EtOAc:Hexane) to afford a pale yellow solid (1.37 g, 91%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.37 (dd, *J* = 6.2, 2.5 Hz, 1H), 7.25-7.22 (m, 2H), 4.51 (q, *J* = 7.1 Hz, 1H), 3.96 (s, 3H), 2.31 (s, 3H), 1.54 (d, *J* = 7.1 Hz, 3H); ^{13}C NMR (500 MHz, $CDCl_3$) δ 200.1, 170.3, 149.8, 141.8, 127.5, 126.0, 119.4, 116.1, 64.4, 55.8, 23.9, 18.1. HRMS (ESI) *m/z* calculated for $C_{12}H_{13}NO_3H^+$ 220.0974. Found 220.0983.

1-Acetyl-7-methoxy-2,2-dimethylindolin-3-one (16): To a suspension of NaH (60%, 0.111 g, 2.78 mmol) in dry THF (8 mL) was added a solution of **15** (0.305 g, 1.39 mmol) in dry THF (5 mL) at room temperature. Freshly distilled methyl iodide (1.7 mL, 27.82 mmol) was added after 30 min, and the reaction stirred at room temperature for 4 h. The mixture was carefully quenched with water and extracted with EtOAc (3 X 10 mL). The combined organic extracts were washed with brine, dried with $MgSO_4$ and concentrated. The residue was purified by flash column chromatography (3:7 EtOAc:Hexane) to yield a yellow solid (0.221 g, 68%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.41 (dd, *J* = 5.9, 2.7 Hz, 1H), 7.20-7.18 (m, 2H), 3.93 (s, 3H), 2.27 (s, 3H), 1.56 (s, 6H); ^{13}C NMR (400 MHz, $CDCl_3$) δ 202.3, 171.1, 148.7, 141.9, 125.3, 124.9, 118.8, 116.5, 70.9, 55.7, 26.2, 23.1. HRMS (ESI) *m/z* calculated for $C_{13}H_{15}NO_3H^+$ 234.1130. Found 234.1139.

1-Acetyl-7-hydroxy-2,2-dimethylindolin-3-one (17): To a solution of **16** (0.500 g, 2.14 mmol) in DCM (20 mL) cooled to -78 ^{o}C was added a solution of 1M BBr_3 in DCM (10.7 mL) and the reaction was slowly allowed to warm up to room temperature overnight. The reaction was carefully quenched with saturated $NaHCO_3$ solution and the mixture extracted with DCM (3 X

20 mL). The combined organic extracts were washed with brine, dried with $MgSO_4$ and evaporated under reduced pressure. The residue was purified by flash column chromatography (3:7 EtOAc:Hexane) to give a pale yellow solid (0.426 g, 91%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 11.15 (s, 1H), 7.36 (dd, *J* = 7.1, 1.7 Hz, 1H), 7.32-7.26 (m, 2H), 2.53 (s, 3H), 1.66 (s, 6H); ^{13}C NMR (500 MHz, $CDCl_3$) δ 200.1, 170.7, 147.3, 138.2, 127.6, 126.9, 124.5, 115.1, 67.8, 24.3, 23.8. HRMS (ESI) *m/z* calculated for $C_{12}H_{13}NO_3H^+$ 220.0974. Found 220.0977.

Part III. ^{1}H NMR Procedures for VT Experiments

A 0.005 M solution of **2** in d_6-DMSO was prepared. ^{1}H-NMR measurements were made in the range 298-368K. The first measurement was made at the lowest temperature and then the temperature was increased in 10K increments. All the spectra obtained were referenced to the solvent peak and the ppm change of the two indolinone NH peaks was monitored. The change in chemical shift was plotted versus the change in temperature and the data was fitted to a linear equation using EXCEL (2000, Microsoft Corporation).

Part IV. Crystallographic Data

Compound 1

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

atom	x	y	z	B_{eq}
O(1)	1.0248(2)	0.2203(2)	0.2079(3)	5.49(8)
O(2)	0.6942(2)	0.30698(19)	0.3647(3)	4.85(7)
O(3)	0.3675(2)	0.32091(17)	0.5920(2)	4.28(6)
O(4)	0.1709(2)	0.43026(19)	0.9347(3)	4.71(7)
N(1)	0.8693(2)	0.3082(2)	0.3321(3)	4.27(8)
N(2)	0.5418(2)	0.3445(2)	0.5324(4)	4.46(8)
N(3)	0.2060(3)	0.3282(2)	0.7531(3)	4.29(8)
C(1)	1.0424(3)	0.4016(2)	0.2458(4)	4.48(9)
C(2)	1.0331(3)	0.4766(2)	0.2773(4)	4.53(9)
C(3)	0.9668(3)	0.4989(2)	0.3287(4)	4.10(9)

C(4)	0.9084(3)	0.4465(2)	0.3493(4)	3.65(8)
C(5)	0.9175(2)	0.3684(2)	0.3164(3)	3.54(8)
C(6)	0.9847(2)	0.3473(2)	0.2668(4)	3.76(8)
C(7)	0.9788(3)	0.2636(2)	0.2475(4)	4.17(9)
C(8)	0.9004(3)	0.2371(2)	0.2883(4)	3.99(8)
C(9)	0.8340(4)	0.2024(3)	0.1739(5)	6.43(13)
C(10)	0.9282(4)	0.1801(2)	0.3998(5)	5.88(12)
C(11)	0.8417(3)	0.4631(2)	0.4062(4)	3.85(8)
C(12)	0.7842(2)	0.4648(2)	0.4567(4)	3.59(8)
C(13)	0.7175(2)	0.4657(2)	0.5182(4)	3.48(8)
C(14)	0.7106(3)	0.5251(2)	0.6008(4)	4.03(9)
C(15)	0.6459(3)	0.5264(2)	0.6624(4)	4.16(9)
C(16)	0.5857(2)	0.4677(2)	0.6454(4)	3.63(8)
C(17)	0.5922(2)	0.4073(2)	0.5625(4)	3.46(8)
C(18)	0.6580(2)	0.4053(2)	0.5008(3)	3.40(8)
C(19)	0.6480(3)	0.3343(2)	0.4265(4)	4.00(8)
C(20)	0.5674(3)	0.2941(2)	0.4414(4)	4.37(9)
C(21)	0.5020(3)	0.2952(3)	0.3096(5)	6.51(13)
C(22)	0.5865(4)	0.2135(3)	0.4944(6)	6.70(14)
C(23)	0.5194(3)	0.4672(2)	0.7069(4)	3.69(8)
C(24)	0.4604(3)	0.4606(2)	0.7523(4)	3.96(8)
C(25)	0.3895(3)	0.4530(2)	0.8041(4)	3.78(8)
C(26)	0.3762(3)	0.5029(2)	0.8973(5)	5.10(11)
C(27)	0.3044(3)	0.4965(3)	0.9435(5)	5.28(11)
C(28)	0.2448(3)	0.4406(2)	0.8993(4)	4.25(9)
C(29)	0.2571(2)	0.3884(2)	0.8065(4)	3.41(8)
C(30)	0.3283(2)	0.3942(2)	0.7591(3)	3.34(7)

Table 1. Atomic coordinates and B_{iso}/B_{eq} (continued)

atom	x	y	z	B_{eq}
C(31)	0.3200(2)	0.3362(2)	0.6599(3)	3.34(7)
C(32)	0.2377(2)	0.2918(2)	0.6518(4)	3.69(8)
C(33)	0.1750(3)	0.3034(3)	0.5189(4)	5.23(11)
C(34)	0.2561(3)	0.2072(2)	0.6810(5)	5.08(10)
C(35)	0.1475(3)	0.4898(3)	1.0082(4)	5.49(12)

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

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

atom	x	y	z	B_{eq}
H(1)	1.0872	0.3867	0.2104	5.38
H(2)	1.0721	0.5141	0.2642	5.44
H(3)	0.9618	0.5515	0.3499	4.92
H(4)	0.7981	0.1667	0.2059	7.72
H(5)	0.8625	0.1746	0.1182	7.72
H(6)	0.7988	0.2435	0.1239	7.72
H(7)	0.9882	0.1886	0.4452	7.05
H(8)	0.9205	0.1276	0.3656	7.05
H(9)	0.8937	0.1876	0.4604	7.05
H(10)	0.7508	0.5658	0.6158	4.83
H(11)	0.6427	0.5684	0.7174	5.00
H(12)	0.4601	0.2544	0.3061	7.81
H(13)	0.5304	0.2867	0.2415	7.81
H(14)	0.4732	0.3452	0.2963	7.81
H(15)	0.6466	0.2097	0.5428	8.04
H(16)	0.5740	0.1766	0.4225	8.04
H(17)	0.5511	0.2018	0.5523	8.04
H(18)	0.4166	0.5423	0.9305	6.12
H(19)	0.2970	0.5318	1.0070	6.33
H(20)	0.1351	0.2601	0.4997	6.27
H(21)	0.2060	0.3060	0.4528	6.27
H(22)	0.1436	0.3514	0.5185	6.27
H(23)	0.3151	0.2009	0.7332	6.09
H(24)	0.2472	0.1787	0.5995	6.09
H(25)	0.2178	0.1872	0.7292	6.09
H(26)	0.1731	0.4801	1.1008	6.59
H(27)	0.0854	0.4911	0.9903	6.59
H(28)	0.1677	0.5392	0.9844	6.59
H(1A)	0.809(3)	0.316(2)	0.347(4)	6.8(13)
H(2A)	0.497(2)	0.340(2)	0.558(3)	2.5(8)
H(3A)	0.154(4)	0.322(3)	0.754(5)	7.5(17)

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

Table 3. Anisotropic displacement parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(1)	0.063(2)	0.069(2)	0.081(2)	0.0180(19)	0.0273(19)	-0.0064(17)
O(2)	0.047(2)	0.069(2)	0.076(2)	-0.0044(16)	0.0305(17)	-0.0230(17)
O(3)	0.045(2)	0.0575(19)	0.0668(19)	-0.0043(15)	0.0259(16)	-0.0079(15)
O(4)	0.049(2)	0.068(2)	0.073(2)	0.0066(16)	0.0361(17)	0.0000(16)
N(1)	0.053(2)	0.042(2)	0.077(2)	-0.0035(18)	0.036(2)	-0.0058(18)
N(2)	0.046(2)	0.052(2)	0.083(2)	-0.0120(19)	0.036(2)	-0.0174(19)
N(3)	0.046(2)	0.057(2)	0.068(2)	-0.008(2)	0.028(2)	-0.0010(18)
C(1)	0.041(2)	0.073(3)	0.061(2)	0.001(2)	0.022(2)	0.005(2)
C(2)	0.051(3)	0.059(3)	0.069(2)	-0.010(2)	0.028(2)	0.008(2)
C(3)	0.050(3)	0.046(2)	0.062(2)	-0.003(2)	0.019(2)	0.001(2)
C(4)	0.044(2)	0.049(2)	0.052(2)	-0.001(2)	0.024(2)	0.0030(19)
C(5)	0.038(2)	0.052(2)	0.049(2)	0.002(2)	0.0186(19)	0.0039(19)
C(6)	0.039(2)	0.056(2)	0.049(2)	0.003(2)	0.014(2)	0.0017(19)
C(7)	0.047(3)	0.061(2)	0.050(2)	0.006(2)	0.014(2)	-0.006(2)
C(8)	0.049(3)	0.049(2)	0.056(2)	-0.001(2)	0.018(2)	-0.006(2)
C(9)	0.068(4)	0.089(4)	0.087(3)	-0.012(3)	0.019(3)	-0.027(3)
C(10)	0.092(4)	0.060(3)	0.075(3)	0.001(3)	0.030(3)	0.003(2)
C(11)	0.049(2)	0.045(2)	0.054(2)	-0.003(2)	0.015(2)	0.0024(19)
C(12)	0.040(2)	0.038(2)	0.061(2)	0.0004(19)	0.017(2)	0.0019(19)
C(13)	0.034(2)	0.045(2)	0.059(2)	0.0011(19)	0.020(2)	0.0086(19)
C(14)	0.052(3)	0.037(2)	0.070(2)	-0.011(2)	0.026(2)	-0.007(2)
C(15)	0.052(3)	0.043(2)	0.070(2)	-0.007(2)	0.028(2)	-0.012(2)
C(16)	0.041(2)	0.044(2)	0.057(2)	-0.000(2)	0.020(2)	0.0001(19)
C(17)	0.037(2)	0.039(2)	0.058(2)	-0.0065(19)	0.017(2)	-0.0008(18)
C(18)	0.042(2)	0.042(2)	0.049(2)	0.0010(19)	0.019(2)	-0.0023(18)
C(19)	0.042(2)	0.055(2)	0.057(2)	0.001(2)	0.016(2)	-0.006(2)
C(20)	0.049(3)	0.052(2)	0.070(2)	-0.009(2)	0.024(2)	-0.014(2)
C(21)	0.048(3)	0.106(4)	0.089(3)	-0.010(3)	0.011(2)	-0.034(3)
C(22)	0.086(4)	0.054(3)	0.131(5)	-0.002(3)	0.057(4)	-0.006(3)
C(23)	0.043(2)	0.041(2)	0.060(2)	-0.005(2)	0.021(2)	-0.0089(19)
C(24)	0.049(3)	0.044(2)	0.062(2)	-0.007(2)	0.025(2)	-0.011(2)
C(25)	0.041(2)	0.050(2)	0.060(2)	-0.001(2)	0.025(2)	-0.003(2)
C(26)	0.058(3)	0.058(2)	0.085(3)	-0.020(2)	0.031(2)	-0.025(2)
C(27)	0.068(3)	0.068(3)	0.073(3)	-0.007(2)	0.035(2)	-0.024(2)
C(28)	0.049(3)	0.063(3)	0.059(2)	0.004(2)	0.029(2)	0.008(2)
C(29)	0.037(2)	0.041(2)	0.052(2)	0.0006(19)	0.0144(19)	0.0056(18)
C(30)	0.039(2)	0.043(2)	0.050(2)	0.0023(19)	0.0202(19)	0.0020(18)

Table 3. Anisotropic displacement parameters (continued)

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(31)	0.040(2)	0.040(2)	0.049(2)	0.0036(18)	0.0154(19)	0.0065(17)
C(32)	0.041(2)	0.048(2)	0.054(2)	-0.001(2)	0.018(2)	0.0030(19)
C(33)	0.042(3)	0.090(3)	0.068(3)	-0.012(2)	0.016(2)	-0.004(2)
C(34)	0.051(3)	0.049(2)	0.098(3)	-0.004(2)	0.030(2)	0.005(2)
C(35)	0.066(3)	0.088(3)	0.064(3)	0.013(3)	0.034(2)	-0.007(2)

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

Table 4. Bond lengths (Å)

atom	atom	distance	atom	atom	distance
O(1)	C(7)	1.221(6)	O(2)	C(19)	1.231(6)
O(3)	C(31)	1.232(6)	O(4)	C(28)	1.377(6)
O(4)	C(35)	1.416(6)	N(1)	C(5)	1.348(6)
N(1)	C(8)	1.462(6)	N(2)	C(17)	1.353(5)
N(2)	C(20)	1.456(6)	N(3)	C(29)	1.362(5)
N(3)	C(32)	1.470(6)	C(1)	C(2)	1.366(7)
C(1)	C(6)	1.398(7)	C(2)	C(3)	1.401(7)
C(3)	C(4)	1.383(6)	C(4)	C(5)	1.421(6)
C(4)	C(11)	1.420(7)	C(5)	C(6)	1.396(7)
C(6)	C(7)	1.469(6)	C(7)	C(8)	1.536(7)
C(8)	C(9)	1.521(6)	C(8)	C(10)	1.523(6)
C(11)	C(12)	1.208(7)	C(12)	C(13)	1.424(7)
C(13)	C(14)	1.386(6)	C(13)	C(18)	1.409(6)
C(14)	C(15)	1.394(7)	C(15)	C(16)	1.396(6)
C(16)	C(17)	1.398(6)	C(16)	C(23)	1.419(7)
C(17)	C(18)	1.412(7)	C(18)	C(19)	1.454(6)
C(19)	C(20)	1.539(7)	C(20)	C(21)	1.520(6)
C(20)	C(22)	1.514(7)	C(23)	C(24)	1.201(7)
C(24)	C(25)	1.424(7)	C(25)	C(26)	1.385(7)
C(25)	C(30)	1.420(5)	C(26)	C(27)	1.401(8)
C(27)	C(28)	1.366(7)	C(28)	C(29)	1.402(6)
C(29)	C(30)	1.399(7)	C(30)	C(31)	1.444(5)
C(31)	C(32)	1.533(6)	C(32)	C(33)	1.522(5)
C(32)	C(34)	1.518(6)			

Table 5. Bond lengths involving hydrogens (Å)

atom	atom	distance	atom	atom	distance
N(1)	H(1A)	1.06(6)	N(2)	H(2A)	0.86(4)
N(3)	H(3A)	0.86(7)	C(1)	H(1)	0.950
C(2)	H(2)	0.950	C(3)	H(3)	0.950
C(9)	H(4)	0.980	C(9)	H(5)	0.980
C(9)	H(6)	0.980	C(10)	H(7)	0.980
C(10)	H(8)	0.980	C(10)	H(9)	0.980
C(14)	H(10)	0.950	C(15)	H(11)	0.950
C(21)	H(12)	0.980	C(21)	H(13)	0.980
C(21)	H(14)	0.980	C(22)	H(15)	0.980
C(22)	H(16)	0.980	C(22)	H(17)	0.980
C(26)	H(18)	0.950	C(27)	H(19)	0.950
C(33)	H(20)	0.980	C(33)	H(21)	0.980
C(33)	H(22)	0.980	C(34)	H(23)	0.980
C(34)	H(24)	0.980	C(34)	H(25)	0.980
C(35)	H(26)	0.980	C(35)	H(27)	0.980
C(35)	H(28)	0.980			

Table 6. Bond angles (°)

atom	atom	atom	angle	atom	atom	atom	angle
C(28)	O(4)	C(35)	116.9(3)	C(5)	N(1)	C(8)	110.3(4)
C(17)	N(2)	C(20)	112.1(4)	C(29)	N(3)	C(32)	109.9(4)
C(2)	C(1)	C(6)	118.6(5)	C(1)	C(2)	C(3)	120.9(4)
C(2)	C(3)	C(4)	121.7(4)	C(3)	C(4)	C(5)	117.7(4)
C(3)	C(4)	C(11)	125.6(4)	C(5)	C(4)	C(11)	116.6(4)
N(1)	C(5)	C(4)	127.2(4)	N(1)	C(5)	C(6)	113.2(4)
C(4)	C(5)	C(6)	119.6(4)	C(1)	C(6)	C(5)	121.5(4)
C(1)	C(6)	C(7)	131.9(4)	C(5)	C(6)	C(7)	106.6(4)
O(1)	C(7)	C(6)	129.5(4)	O(1)	C(7)	C(8)	124.0(4)
C(6)	C(7)	C(8)	106.5(4)	N(1)	C(8)	C(7)	103.3(3)
N(1)	C(8)	C(9)	111.1(4)	N(1)	C(8)	C(10)	110.6(4)
C(7)	C(8)	C(9)	110.9(4)	C(7)	C(8)	C(10)	109.1(4)
C(9)	C(8)	C(10)	111.5(4)	C(4)	C(11)	C(12)	169.6(4)
C(11)	C(12)	C(13)	178.8(4)	C(12)	C(13)	C(14)	120.8(4)
C(12)	C(13)	C(18)	121.5(4)	C(14)	C(13)	C(18)	117.7(4)
C(13)	C(14)	C(15)	121.4(4)	C(14)	C(15)	C(16)	121.9(4)
C(15)	C(16)	C(17)	117.2(4)	C(15)	C(16)	C(23)	123.2(4)
C(17)	C(16)	C(23)	119.6(4)	N(2)	C(17)	C(16)	127.6(4)
N(2)	C(17)	C(18)	111.2(4)	C(16)	C(17)	C(18)	121.2(3)
C(13)	C(18)	C(17)	120.5(3)	C(13)	C(18)	C(19)	132.4(4)
C(17)	C(18)	C(19)	107.1(3)	O(2)	C(19)	C(18)	129.0(4)
O(2)	C(19)	C(20)	123.6(4)	C(18)	C(19)	C(20)	107.3(4)
N(2)	C(20)	C(19)	102.2(3)	N(2)	C(20)	C(21)	110.9(4)
N(2)	C(20)	C(22)	111.9(4)	C(19)	C(20)	C(21)	107.6(4)
C(19)	C(20)	C(22)	111.5(4)	C(21)	C(20)	C(22)	112.3(4)
C(16)	C(23)	C(24)	173.7(4)	C(23)	C(24)	C(25)	179.0(4)
C(24)	C(25)	C(26)	122.0(4)	C(24)	C(25)	C(30)	120.5(4)
C(26)	C(25)	C(30)	117.4(4)	C(25)	C(26)	C(27)	121.3(4)
C(26)	C(27)	C(28)	121.5(5)	O(4)	C(28)	C(27)	126.5(4)
O(4)	C(28)	C(29)	114.9(4)	C(27)	C(28)	C(29)	118.6(5)
N(3)	C(29)	C(28)	127.1(4)	N(3)	C(29)	C(30)	112.3(4)
C(28)	C(29)	C(30)	120.6(3)	C(25)	C(30)	C(29)	120.6(4)
C(25)	C(30)	C(31)	131.9(4)	C(29)	C(30)	C(31)	107.4(3)
O(3)	C(31)	C(30)	129.7(4)	O(3)	C(31)	C(32)	123.1(3)
C(30)	C(31)	C(32)	107.2(4)	N(3)	C(32)	C(31)	103.0(3)
N(3)	C(32)	C(33)	110.5(3)	N(3)	C(32)	C(34)	110.8(4)
C(31)	C(32)	C(33)	110.0(3)	C(31)	C(32)	C(34)	110.8(3)

Table 6. Bond angles (°) (continued)

atom	atom	atom	angle	atom	atom	atom	angle
C(33)	C(32)	C(34)	111.4(3)				

Table 7. Bond angles involving hydrogens (°)

atom	atom	atom	angle	atom	atom	atom	angle
C(5)	N(1)	H(1A)	122(2)	C(8)	N(1)	H(1A)	125(2)
C(17)	N(2)	H(2A)	121(2)	C(20)	N(2)	H(2A)	126(2)
C(29)	N(3)	H(3A)	125(4)	C(32)	N(3)	H(3A)	121(3)
C(2)	C(1)	H(1)	120.7	C(6)	C(1)	H(1)	120.7
C(1)	C(2)	H(2)	119.6	C(3)	C(2)	H(2)	119.6
C(2)	C(3)	H(3)	119.2	C(4)	C(3)	H(3)	119.2
C(8)	C(9)	H(4)	109.5	C(8)	C(9)	H(5)	109.5
C(8)	C(9)	H(6)	109.5	H(4)	C(9)	H(5)	109.5
H(4)	C(9)	H(6)	109.5	H(5)	C(9)	H(6)	109.5
C(8)	C(10)	H(7)	109.5	C(8)	C(10)	H(8)	109.5
C(8)	C(10)	H(9)	109.5	H(7)	C(10)	H(8)	109.5
H(7)	C(10)	H(9)	109.5	H(8)	C(10)	H(9)	109.5
C(13)	C(14)	H(10)	119.3	C(15)	C(14)	H(10)	119.3
C(14)	C(15)	H(11)	119.1	C(16)	C(15)	H(11)	119.1
C(20)	C(21)	H(12)	109.5	C(20)	C(21)	H(13)	109.5
C(20)	C(21)	H(14)	109.5	H(12)	C(21)	H(13)	109.5
H(12)	C(21)	H(14)	109.5	H(13)	C(21)	H(14)	109.5
C(20)	C(22)	H(15)	109.5	C(20)	C(22)	H(16)	109.5
C(20)	C(22)	H(17)	109.5	H(15)	C(22)	H(16)	109.5
H(15)	C(22)	H(17)	109.5	H(16)	C(22)	H(17)	109.5
C(25)	C(26)	H(18)	119.4	C(27)	C(26)	H(18)	119.3
C(26)	C(27)	H(19)	119.2	C(28)	C(27)	H(19)	119.2
C(32)	C(33)	H(20)	109.5	C(32)	C(33)	H(21)	109.5
C(32)	C(33)	H(22)	109.5	H(20)	C(33)	H(21)	109.5
H(20)	C(33)	H(22)	109.5	H(21)	C(33)	H(22)	109.5
C(32)	C(34)	H(23)	109.5	C(32)	C(34)	H(24)	109.5
C(32)	C(34)	H(25)	109.5	H(23)	C(34)	H(24)	109.5
H(23)	C(34)	H(25)	109.5	H(24)	C(34)	H(25)	109.5
O(4)	C(35)	H(26)	109.5	O(4)	C(35)	H(27)	109.5
O(4)	C(35)	H(28)	109.5	H(26)	C(35)	H(27)	109.5
H(26)	C(35)	H(28)	109.5	H(27)	C(35)	H(28)	109.5

Table 8. Torsion Angles(°)

atom1	atom2	atom3	atom4	angle
C(35)	O(4)	C(28)	C(27)	10.6(6)
C(5)	N(1)	C(8)	C(7)	2.2(4)
C(5)	N(1)	C(8)	C(10)	118.8(4)
C(8)	N(1)	C(5)	C(6)	-1.7(4)
C(17)	N(2)	C(20)	C(21)	-110.6(4)
C(20)	N(2)	C(17)	C(16)	179.5(3)
C(29)	N(3)	C(32)	C(31)	4.0(4)
C(29)	N(3)	C(32)	C(34)	122.6(3)
C(32)	N(3)	C(29)	C(30)	-5.3(4)
C(2)	C(1)	C(6)	C(7)	-177.9(4)
C(1)	C(2)	C(3)	C(4)	-0.1(5)
C(2)	C(3)	C(4)	C(11)	176.9(4)
C(3)	C(4)	C(5)	C(6)	0.8(5)
C(5)	C(4)	C(11)	C(12)	12(2)
C(11)	C(4)	C(5)	C(6)	-176.5(3)
N(1)	C(5)	C(6)	C(7)	0.4(4)
C(4)	C(5)	C(6)	C(7)	177.9(3)
C(1)	C(6)	C(7)	C(8)	-179.7(4)
C(5)	C(6)	C(7)	C(8)	1.0(4)
O(1)	C(7)	C(8)	C(9)	-63.0(5)
C(6)	C(7)	C(8)	N(1)	-1.9(4)
C(6)	C(7)	C(8)	C(10)	-119.6(3)
C(11)	C(12)	C(13)	C(14)	101(20)
C(12)	C(13)	C(14)	C(15)	-179.6(3)
C(12)	C(13)	C(18)	C(19)	0.2(5)
C(14)	C(13)	C(18)	C(19)	-177.8(4)
C(13)	C(14)	C(15)	C(16)	0.9(6)
C(14)	C(15)	C(16)	C(23)	179.8(3)
C(15)	C(16)	C(17)	C(18)	1.3(5)
C(17)	C(16)	C(23)	C(24)	-4(4)
C(23)	C(16)	C(17)	C(18)	-179.2(3)
N(2)	C(17)	C(18)	C(19)	-0.5(4)
C(16)	C(17)	C(18)	C(19)	177.9(3)
C(13)	C(18)	C(19)	C(20)	-177.2(4)
C(17)	C(18)	C(19)	C(20)	2.9(4)
O(2)	C(19)	C(20)	C(21)	-69.1(5)
C(18)	C(19)	C(20)	N(2)	-3.9(4)
C(35)	O(4)	C(28)	C(29)	-168.2(3)
C(5)	N(1)	C(8)	C(9)	-116.8(4)
C(8)	N(1)	C(5)	C(4)	-179.0(3)
C(17)	N(2)	C(20)	C(19)	3.8(4)
C(17)	N(2)	C(20)	C(22)	123.1(4)
C(20)	N(2)	C(17)	C(18)	-2.2(4)
C(29)	N(3)	C(32)	C(33)	-113.4(4)
C(32)	N(3)	C(29)	C(28)	175.1(3)
C(2)	C(1)	C(6)	C(5)	1.3(6)
C(6)	C(1)	C(2)	C(3)	-0.5(6)
C(2)	C(3)	C(4)	C(5)	-0.1(4)
C(3)	C(4)	C(5)	N(1)	178.0(3)
C(3)	C(4)	C(11)	C(12)	-165(2)
C(11)	C(4)	C(5)	N(1)	0.7(5)
N(1)	C(5)	C(6)	C(1)	-179.0(3)
C(4)	C(5)	C(6)	C(1)	-1.5(5)
C(1)	C(6)	C(7)	O(1)	0.5(7)
C(5)	C(6)	C(7)	O(1)	-178.8(4)
O(1)	C(7)	C(8)	N(1)	177.9(3)
O(1)	C(7)	C(8)	C(10)	60.2(5)
C(6)	C(7)	C(8)	C(9)	117.2(4)
C(4)	C(11)	C(12)	C(13)	45(21)
C(11)	C(12)	C(13)	C(18)	-77(20)
C(12)	C(13)	C(18)	C(17)	-179.9(3)
C(14)	C(13)	C(18)	C(17)	2.1(5)
C(18)	C(13)	C(14)	C(15)	-1.5(6)
C(14)	C(15)	C(16)	C(17)	-0.7(6)
C(15)	C(16)	C(17)	N(2)	179.4(4)
C(15)	C(16)	C(23)	C(24)	175(3)
C(23)	C(16)	C(17)	N(2)	-1.1(6)
N(2)	C(17)	C(18)	C(13)	179.5(3)
C(16)	C(17)	C(18)	C(13)	-2.1(5)
C(13)	C(18)	C(19)	O(2)	5.0(7)
C(17)	C(18)	C(19)	O(2)	-175.0(4)
O(2)	C(19)	C(20)	N(2)	174.1(3)
O(2)	C(19)	C(20)	C(22)	54.4(5)
C(18)	C(19)	C(20)	C(21)	112.9(4)

Table 8. Torsion angles (°) (continued)

atom1	atom2	atom3	atom4	angle
C(18)	C(19)	C(20)	C(22)	-123.6(4)
C(23)	C(24)	C(25)	C(26)	-114(27)
C(24)	C(25)	C(26)	C(27)	177.5(4)
C(24)	C(25)	C(30)	C(31)	-2.4(6)
C(26)	C(25)	C(30)	C(31)	176.3(4)
C(25)	C(26)	C(27)	C(28)	0.4(7)
C(26)	C(27)	C(28)	C(29)	0.7(7)
O(4)	C(28)	C(29)	C(30)	177.9(3)
C(27)	C(28)	C(29)	C(30)	-1.0(6)
N(3)	C(29)	C(30)	C(31)	4.1(4)
C(28)	C(29)	C(30)	C(31)	-176.2(3)
C(25)	C(30)	C(31)	C(32)	-177.2(4)
C(29)	C(30)	C(31)	C(32)	-1.4(4)
O(3)	C(31)	C(32)	C(33)	-64.5(5)
C(30)	C(31)	C(32)	N(3)	-1.5(4)
C(30)	C(31)	C(32)	C(34)	-120.1(4)
C(16)	C(23)	C(24)	C(25)	-68(29)
C(23)	C(24)	C(25)	C(30)	64(28)
C(24)	C(25)	C(30)	C(29)	-177.8(3)
C(26)	C(25)	C(30)	C(29)	0.9(5)
C(30)	C(25)	C(26)	C(27)	-1.2(6)
C(26)	C(27)	C(28)	O(4)	-178.0(4)
O(4)	C(28)	C(29)	N(3)	-2.5(6)
C(27)	C(28)	C(29)	N(3)	178.6(4)
N(3)	C(29)	C(30)	C(25)	-179.5(3)
C(28)	C(29)	C(30)	C(25)	0.1(4)
C(25)	C(30)	C(31)	O(3)	3.8(7)
C(29)	C(30)	C(31)	O(3)	179.6(4)
O(3)	C(31)	C(32)	N(3)	177.6(3)
O(3)	C(31)	C(32)	C(34)	59.1(5)
C(30)	C(31)	C(32)	C(33)	116.4(4)

The sign is positive if when looking from atom 2 to atom 3 a clock-wise motion of atom 1 would superimpose it on atom 4.

Table 9. Distances beyond the asymmetric unit out to 3.60 Å

atom	atom	distance	atom	atom	distance
O(1)	N(3)[1)]	2.991(6)	O(2)	C(9)[2)]	3.474(6)
O(3)	C(14)[3)]	3.408(5)	O(3)	C(21)[2)]	3.395(6)
O(4)	C(3)[3)]	3.325(5)	O(4)	C(34)[2)]	3.549(5)
N(3)	O(1)[4)]	2.991(6)	C(1)	C(33)[5)]	3.562(6)
C(3)	O(4)[3)]	3.325(5)	C(3)	C(3)[6)]	3.533(6)
C(3)	C(35)[3)]	3.585(6)	C(6)	C(33)[5)]	3.597(5)
C(9)	O(2)[7)]	3.474(6)	C(9)	C(18)[7)]	3.507(7)
C(9)	C(19)[7)]	3.501(6)	C(11)	C(29)[3)]	3.528(5)
C(12)	C(30)[3)]	3.521(5)	C(14)	O(3)[3)]	3.408(5)
C(18)	C(9)[2)]	3.507(7)	C(19)	C(9)[2)]	3.501(6)
C(21)	O(3)[7)]	3.395(6)	C(29)	C(11)[3)]	3.528(5)
C(30)	C(12)[3)]	3.521(5)	C(33)	C(1)[8)]	3.562(6)
C(33)	C(6)[8)]	3.597(5)	C(34)	O(4)[7)]	3.549(5)
C(35)	C(3)[3)]	3.585(6)			

Symmetry Operators:

(1) X+1,-Y+1/2,Z+1/2-1
(2) X,-Y+1/2,Z+1/2
(3) -X+1,-Y+1,-Z+1
(4) X-1,-Y+1/2,Z+1/2
(5) X+1,Y,Z
(6) -X+2,-Y+1,-Z+1
(7) X,-Y+1/2,Z+1/2-1
(8) X-1,Y,Z

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
O(1)	H(3)[1)]	3.020	O(1)	H(7)[2)]	3.143
O(1)	H(9)[2)]	3.327	O(1)	H(20)[3)]	3.226
O(1)	H(20)[4)]	3.254	O(1)	H(22)[4)]	3.412
O(1)	H(25)[4)]	3.495	O(1)	H(3A)[4)]	2.17(6)
O(2)	H(5)[5)]	3.308	O(2)	H(6)[5)]	2.966
O(2)	H(15)[2)]	3.334	O(2)	H(17)[2)]	3.521
O(3)	H(10)[6)]	3.195	O(3)	H(12)[5)]	2.712
O(3)	H(13)[5)]	3.289	O(4)	H(3)[6)]	3.236
O(4)	H(20)[5)]	3.466	O(4)	H(24)[5)]	2.656
N(1)	H(5)[5)]	3.116	N(2)	H(13)[5)]	3.240
N(3)	H(3)[6)]	3.386	N(3)	H(20)[5)]	3.521
N(3)	H(21)[5)]	3.168	N(3)	H(24)[5)]	3.589
C(1)	H(7)[2)]	3.473	C(1)	H(10)[7)]	3.347
C(1)	H(21)[3)]	3.402	C(1)	H(22)[3)]	3.063
C(1)	H(25)[4)]	3.308	C(1)	H(26)[8)]	3.271
C(1)	H(27)[8)]	3.391	C(1)	H(27)[6)]	3.367
C(2)	H(8)[9)]	3.234	C(2)	H(10)[7)]	3.483
C(2)	H(22)[3)]	3.481	C(2)	H(26)[8)]	3.352
C(2)	H(27)[8)]	3.425	C(2)	H(27)[6)]	3.039
C(3)	H(3)[7)]	3.434	C(3)	H(27)[6)]	3.293
C(3)	H(28)[6)]	3.532	C(4)	H(3)[7)]	3.337
C(4)	H(28)[6)]	3.457	C(5)	H(28)[6)]	3.528
C(6)	H(7)[2)]	3.522	C(6)	H(9)[2)]	3.268
C(6)	H(20)[3)]	3.346	C(6)	H(22)[3)]	3.190
C(7)	H(7)[2)]	3.392	C(7)	H(9)[2)]	3.126
C(7)	H(20)[3)]	3.168	C(7)	H(3A)[4)]	3.22(6)
C(9)	H(2)[1)]	3.599	C(9)	H(9)[2)]	3.330
C(9)	H(15)[2)]	3.368	C(9)	H(1A)[2)]	3.43(5)
C(10)	H(2)[1)]	3.379	C(10)	H(20)[3)]	3.542
C(10)	H(27)[10)]	3.519	C(10)	H(28)[10)]	3.329
C(11)	H(2)[7)]	3.448	C(11)	H(3)[7)]	3.550
C(11)	H(5)[5)]	3.255	C(11)	H(22)[6)]	3.317
C(12)	H(2)[7)]	3.277	C(12)	H(4)[5)]	3.477
C(12)	H(5)[5)]	3.048	C(12)	H(22)[6)]	3.392
C(13)	H(4)[5)]	3.102	C(13)	H(5)[5)]	3.365
C(14)	H(2)[7)]	3.517	C(14)	H(21)[6)]	3.353
C(14)	H(25)[11)]	3.385	C(14)	H(26)[12)]	3.241

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
C(15)	H(14)$^{6)}$	3.076	C(15)	H(19)$^{12)}$	3.555
C(15)	H(23)$^{11)}$	3.236	C(15)	H(25)$^{11)}$	3.566
C(15)	H(26)$^{12)}$	3.329	C(16)	H(14)$^{6)}$	3.499
C(17)	H(4)$^{5)}$	3.539	C(18)	H(4)$^{5)}$	2.979
C(18)	H(5)$^{5)}$	3.520	C(18)	H(6)$^{5)}$	3.474
C(19)	H(4)$^{5)}$	3.309	C(19)	H(5)$^{5)}$	3.549
C(19)	H(6)$^{5)}$	3.085	C(21)	H(11)$^{6)}$	3.307
C(21)	H(17)$^{2)}$	3.081	C(21)	H(23)$^{2)}$	2.941
C(21)	H(2A)$^{2)}$	3.56(3)	C(22)	H(6)$^{5)}$	3.450
C(22)	H(13)$^{5)}$	3.034	C(22)	H(18)$^{10)}$	3.087
C(23)	H(14)$^{6)}$	3.265	C(23)	H(16)$^{5)}$	3.355
C(24)	H(16)$^{5)}$	3.257	C(24)	H(18)$^{12)}$	3.441
C(26)	H(16)$^{11)}$	3.557	C(26)	H(18)$^{12)}$	3.474
C(28)	H(24)$^{5)}$	2.978	C(29)	H(24)$^{5)}$	3.397
C(30)	H(12)$^{5)}$	3.313	C(31)	H(10)$^{6)}$	3.330
C(31)	H(12)$^{5)}$	2.865	C(33)	H(1)$^{13)}$	3.533
C(33)	H(7)$^{13)}$	3.553	C(33)	H(10)$^{6)}$	3.114
C(33)	H(25)$^{2)}$	3.372	C(33)	H(3A)$^{2)}$	3.53(5)
C(34)	H(1)$^{14)}$	3.300	C(34)	H(10)$^{10)}$	3.307
C(34)	H(11)$^{10)}$	2.958	C(34)	H(12)$^{5)}$	3.306
C(34)	H(14)$^{5)}$	3.544	C(34)	H(21)$^{5)}$	3.247
C(34)	H(26)$^{2)}$	3.542	C(35)	H(1)$^{15)}$	3.173
C(35)	H(2)$^{15)}$	3.333	C(35)	H(8)$^{11)}$	3.105
C(35)	H(9)$^{11)}$	3.538	C(35)	H(24)$^{5)}$	3.365
H(1)	C(33)$^{3)}$	3.533	H(1)	C(34)$^{4)}$	3.300
H(1)	C(35)$^{8)}$	3.173	H(1)	H(7)$^{2)}$	3.143
H(1)	H(10)$^{7)}$	2.908	H(1)	H(21)$^{3)}$	3.117
H(1)	H(22)$^{3)}$	3.234	H(1)	H(24)$^{4)}$	3.361
H(1)	H(25)$^{4)}$	2.454	H(1)	H(26)$^{8)}$	2.629
H(1)	H(27)$^{8)}$	2.972	H(2)	C(9)$^{9)}$	3.599
H(2)	C(10)$^{9)}$	3.379	H(2)	C(11)$^{7)}$	3.448
H(2)	C(12)$^{7)}$	3.277	H(2)	C(14)$^{7)}$	3.517
H(2)	C(35)$^{8)}$	3.333	H(2)	H(4)$^{9)}$	3.359
H(2)	H(5)$^{9)}$	3.126	H(2)	H(8)$^{9)}$	2.436
H(2)	H(10)$^{7)}$	3.152	H(2)	H(26)$^{8)}$	2.785
H(2)	H(27)$^{8)}$	3.032	H(2)	H(27)$^{6)}$	3.196
H(3)	O(1)$^{9)}$	3.020	H(3)	O(4)$^{6)}$	3.236

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(3)	N(3)$^{6)}$	3.386	H(3)	C(3)$^{7)}$	3.434
H(3)	C(4)$^{7)}$	3.337	H(3)	C(11)$^{7)}$	3.550
H(3)	H(3)$^{7)}$	3.593	H(3)	H(5)$^{9)}$	3.525
H(3)	H(22)$^{6)}$	3.028	H(3)	H(27)$^{6)}$	3.591
H(3)	H(3A)$^{6)}$	2.913	H(4)	C(12)$^{2)}$	3.477
H(4)	C(13)$^{2)}$	3.102	H(4)	C(17)$^{2)}$	3.539
H(4)	C(18)$^{2)}$	2.979	H(4)	C(19)$^{2)}$	3.309
H(4)	H(2)$^{1)}$	3.359	H(4)	H(15)$^{2)}$	3.380
H(5)	O(2)$^{2)}$	3.308	H(5)	N(1)$^{2)}$	3.116
H(5)	C(11)$^{2)}$	3.255	H(5)	C(12)$^{2)}$	3.048
H(5)	C(13)$^{2)}$	3.365	H(5)	C(18)$^{2)}$	3.520
H(5)	C(19)$^{2)}$	3.549	H(5)	H(2)$^{1)}$	3.126
H(5)	H(3)$^{1)}$	3.525	H(5)	H(9)$^{2)}$	3.057
H(5)	H(1A)$^{2)}$	2.801	H(6)	O(2)$^{2)}$	2.966
H(6)	C(18)$^{2)}$	3.474	H(6)	C(19)$^{2)}$	3.085
H(6)	C(22)$^{2)}$	3.450	H(6)	H(9)$^{2)}$	2.903
H(6)	H(15)$^{2)}$	2.535	H(6)	H(1A)$^{2)}$	3.189
H(7)	O(1)$^{5)}$	3.143	H(7)	C(1)$^{5)}$	3.473
H(7)	C(6)$^{5)}$	3.522	H(7)	C(7)$^{5)}$	3.392
H(7)	C(33)$^{3)}$	3.553	H(7)	H(1)$^{5)}$	3.143
H(7)	H(20)$^{3)}$	2.625	H(7)	H(27)$^{4)}$	3.479
H(8)	C(2)$^{1)}$	3.234	H(8)	C(35)$^{10)}$	3.105
H(8)	H(2)$^{1)}$	2.436	H(8)	H(26)$^{10)}$	3.058
H(8)	H(27)$^{10)}$	2.847	H(8)	H(27)$^{4)}$	3.368
H(8)	H(28)$^{10)}$	2.883	H(9)	O(1)$^{5)}$	3.327
H(9)	C(6)$^{5)}$	3.268	H(9)	C(7)$^{5)}$	3.126
H(9)	C(9)$^{5)}$	3.330	H(9)	C(35)$^{10)}$	3.538
H(9)	H(5)$^{5)}$	3.057	H(9)	H(6)$^{5)}$	2.903
H(9)	H(27)$^{10)}$	3.458	H(9)	H(28)$^{10)}$	2.888
H(10)	O(3)$^{6)}$	3.195	H(10)	C(1)$^{7)}$	3.347
H(10)	C(2)$^{7)}$	3.483	H(10)	C(31)$^{6)}$	3.330
H(10)	C(33)$^{6)}$	3.114	H(10)	C(34)$^{11)}$	3.307
H(10)	H(1)$^{7)}$	2.908	H(10)	H(2)$^{7)}$	3.152
H(10)	H(21)$^{6)}$	2.508	H(10)	H(22)$^{6)}$	2.917
H(10)	H(23)$^{11)}$	3.205	H(10)	H(25)$^{11)}$	2.647
H(10)	H(26)$^{12)}$	3.056	H(11)	C(21)$^{6)}$	3.307
H(11)	C(34)$^{11)}$	2.958	H(11)	H(12)$^{6)}$	3.487

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(11)	H(14)$^{6)}$	2.392	H(11)	H(19)$^{12)}$	3.337
H(11)	H(23)$^{11)}$	2.421	H(11)	H(24)$^{11)}$	2.971
H(11)	H(25)$^{11)}$	3.014	H(11)	H(26)$^{12)}$	3.218
H(11)	H(2A)$^{6)}$	3.572	H(12)	O(3)$^{2)}$	2.712
H(12)	C(30)$^{2)}$	3.313	H(12)	C(31)$^{2)}$	2.865
H(12)	C(34)$^{2)}$	3.306	H(12)	H(11)$^{6)}$	3.487
H(12)	H(17)$^{2)}$	3.528	H(12)	H(23)$^{2)}$	2.413
H(12)	H(2A)$^{2)}$	3.317	H(13)	O(3)$^{2)}$	3.289
H(13)	N(2)$^{2)}$	3.240	H(13)	C(22)$^{2)}$	3.034
H(13)	H(15)$^{2)}$	3.225	H(13)	H(17)$^{2)}$	2.157
H(13)	H(23)$^{2)}$	3.507	H(13)	H(2A)$^{2)}$	2.901
H(14)	C(15)$^{6)}$	3.076	H(14)	C(16)$^{6)}$	3.499
H(14)	C(23)$^{6)}$	3.265	H(14)	C(34)$^{2)}$	3.544
H(14)	H(11)$^{6)}$	2.392	H(14)	H(17)$^{2)}$	3.315
H(14)	H(23)$^{2)}$	2.613	H(15)	O(2)$^{5)}$	3.334
H(15)	C(9)$^{5)}$	3.368	H(15)	H(4)$^{5)}$	3.380
H(15)	H(6)$^{5)}$	2.535	H(15)	H(13)$^{5)}$	3.225
H(15)	H(18)$^{10)}$	3.129	H(15)	H(19)$^{10)}$	3.313
H(16)	C(23)$^{2)}$	3.355	H(16)	C(24)$^{2)}$	3.257
H(16)	C(26)$^{10)}$	3.557	H(16)	H(18)$^{10)}$	2.798
H(16)	H(19)$^{10)}$	3.238	H(17)	O(2)$^{5)}$	3.521
H(17)	C(21)$^{5)}$	3.081	H(17)	H(12)$^{5)}$	3.528
H(17)	H(13)$^{5)}$	2.157	H(17)	H(14)$^{5)}$	3.315
H(17)	H(18)$^{10)}$	2.819	H(18)	C(22)$^{11)}$	3.087
H(18)	C(24)$^{12)}$	3.441	H(18)	C(26)$^{12)}$	3.474
H(18)	H(15)$^{11)}$	3.129	H(18)	H(16)$^{11)}$	2.798
H(18)	H(17)$^{11)}$	2.819	H(18)	H(18)$^{12)}$	3.096
H(19)	C(15)$^{12)}$	3.555	H(19)	H(11)$^{12)}$	3.337
H(19)	H(15)$^{11)}$	3.313	H(19)	H(16)$^{11)}$	3.238
H(20)	O(1)$^{13)}$	3.226	H(20)	O(1)$^{14)}$	3.254
H(20)	O(4)$^{2)}$	3.466	H(20)	N(3)$^{2)}$	3.521
H(20)	C(6)$^{13)}$	3.346	H(20)	C(7)$^{13)}$	3.168
H(20)	C(10)$^{13)}$	3.542	H(20)	H(7)$^{13)}$	2.625
H(20)	H(3A)$^{2)}$	3.090	H(21)	N(3)$^{2)}$	3.168
H(21)	C(1)$^{13)}$	3.402	H(21)	C(14)$^{6)}$	3.353
H(21)	C(34)$^{2)}$	3.247	H(21)	H(1)$^{13)}$	3.117
H(21)	H(10)$^{6)}$	2.508	H(21)	H(23)$^{2)}$	3.328

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(21)	H(25)$^{2)}$	2.461	H(21)	H(3A)$^{2)}$	3.042
H(22)	O(1)$^{14)}$	3.412	H(22)	C(1)$^{13)}$	3.063
H(22)	C(2)$^{13)}$	3.481	H(22)	C(6)$^{13)}$	3.190
H(22)	C(11)$^{6)}$	3.317	H(22)	C(12)$^{6)}$	3.392
H(22)	H(1)$^{13)}$	3.234	H(22)	H(3)$^{6)}$	3.028
H(22)	H(10)$^{6)}$	2.917	H(23)	C(15)$^{10)}$	3.236
H(23)	C(21)$^{5)}$	2.941	H(23)	H(10)$^{10)}$	3.205
H(23)	H(11)$^{10)}$	2.421	H(23)	H(12)$^{5)}$	2.413
H(23)	H(13)$^{5)}$	3.507	H(23)	H(14)$^{5)}$	2.613
H(23)	H(21)$^{5)}$	3.328	H(24)	O(4)$^{2)}$	2.656
H(24)	N(3)$^{2)}$	3.589	H(24)	C(28)$^{2)}$	2.978
H(24)	C(29)$^{2)}$	3.397	H(24)	C(35)$^{2)}$	3.365
H(24)	H(1)$^{14)}$	3.361	H(24)	H(11)$^{10)}$	2.971
H(24)	H(26)$^{2)}$	3.018	H(24)	H(3A)$^{2)}$	3.595
H(25)	O(1)$^{14)}$	3.495	H(25)	C(1)$^{14)}$	3.308
H(25)	C(14)$^{10)}$	3.385	H(25)	C(15)$^{10)}$	3.566
H(25)	C(33)$^{5)}$	3.372	H(25)	H(1)$^{14)}$	2.454
H(25)	H(10)$^{10)}$	2.647	H(25)	H(11)$^{10)}$	3.014
H(25)	H(21)$^{5)}$	2.461	H(25)	H(26)$^{2)}$	3.215
H(26)	C(1)$^{15)}$	3.271	H(26)	C(2)$^{15)}$	3.352
H(26)	C(14)$^{12)}$	3.241	H(26)	C(15)$^{12)}$	3.329
H(26)	C(34)$^{5)}$	3.542	H(26)	H(1)$^{15)}$	2.629
H(26)	H(2)$^{15)}$	2.785	H(26)	H(8)$^{11)}$	3.058
H(26)	H(10)$^{12)}$	3.056	H(26)	H(11)$^{12)}$	3.218
H(26)	H(24)$^{5)}$	3.018	H(26)	H(25)$^{5)}$	3.215
H(27)	C(1)$^{15)}$	3.391	H(27)	C(1)$^{6)}$	3.367
H(27)	C(2)$^{15)}$	3.425	H(27)	C(2)$^{6)}$	3.039
H(27)	C(3)$^{6)}$	3.293	H(27)	C(10)$^{11)}$	3.519
H(27)	H(1)$^{15)}$	2.972	H(27)	H(2)$^{15)}$	3.032
H(27)	H(2)$^{6)}$	3.196	H(27)	H(3)$^{6)}$	3.591
H(27)	H(7)$^{14)}$	3.479	H(27)	H(8)$^{11)}$	2.847
H(27)	H(8)$^{14)}$	3.368	H(27)	H(9)$^{11)}$	3.458
H(27)	H(27)$^{16)}$	2.879	H(28)	C(3)$^{6)}$	3.532
H(28)	C(4)$^{6)}$	3.457	H(28)	C(5)$^{6)}$	3.528
H(28)	C(10)$^{11)}$	3.329	H(28)	H(8)$^{11)}$	2.883
H(28)	H(9)$^{11)}$	2.888	H(1A)	C(9)$^{5)}$	3.43(5)
H(1A)	H(5)$^{5)}$	2.801	H(1A)	H(6)$^{5)}$	3.189

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(2A)	C(21)[5)]	3.56(3)	H(2A)	H(11)[6)]	3.572
H(2A)	H(12)[5)]	3.317	H(2A)	H(13)[5)]	2.901
H(3A)	O(1)[14)]	2.17(6)	H(3A)	C(7)[14)]	3.22(6)
H(3A)	C(33)[5)]	3.53(5)	H(3A)	H(3)[6)]	2.913
H(3A)	H(20)[5)]	3.090	H(3A)	H(21)[5)]	3.042
H(3A)	H(24)[5)]	3.595			

Symmetry Operators:

(1) -X+2,Y+1/2-1,-Z+1/2
(2) X,-Y+1/2,Z+1/2-1
(3) X+1,Y,Z
(4) X+1,-Y+1/2,Z+1/2-1
(5) X,-Y+1/2,Z+1/2
(6) -X+1,-Y+1,-Z+1
(7) -X+2,-Y+1,-Z+1
(8) X+1,Y,Z-1
(9) -X+2,Y+1/2,-Z+1/2
(10) -X+1,Y+1/2-1,-Z+1/2+1
(11) -X+1,Y+1/2,-Z+1/2+1
(12) -X+1,-Y+1,-Z+2
(13) X-1,Y,Z
(14) X-1,-Y+1/2,Z+1/2
(15) X-1,Y,Z+1
(16) -X,-Y+1,-Z+2

Compound **2**

Table 1. Crystal data and structure refinement for **2**.

Identification code	Hamilton_pw02	
Empirical formula	$C_{23}\ H_{22}\ N_2\ O_3$	
Formula weight	374.43	
Temperature	173(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)/n	
Unit cell dimensions	a = 11.677(2) Å	α= 90°.
	b = 16.749(3) Å	β= 94.81(3)°.
	c = 20.009(4) Å	γ = 90°.
Volume	3899.6(13) $Å^3$	
Z	8	
Density (calculated)	1.276 g/cm^3	
Absorption coefficient	0.85 cm^{-1}	
F(000)	1584	
Crystal size	0.15 x 0.15 x 0.10 mm^3	
Theta range for data collection	1.59 to 28.95°.	
Index ranges	-15<=h<=15, -22<=k<=16, -27<=l<=27	
Reflections collected	16118	
Independent reflections	10195 [R(int) = 0.0662]	
Completeness to theta = 28.95°	98.8 %	
Absorption correction	None	
Max. and min. transmission	0.9915 and 0.9873	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	10195 / 0 / 521	
Goodness-of-fit on F^2	1.011	
Final R indices [I>2sigma(I)]	R1 = 0.0628, wR2 = 0.0990	
R indices (all data)	R1 = 0.1773, wR2 = 0.1213	
Largest diff. peak and hole	0.223 and -0.208 $e.Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for hamilton_pw02. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(1)	6590(1)	7173(1)	4144(1)	43(1)
O(2)	8698(1)	7838(1)	1530(1)	38(1)
O(3)	9364(1)	9321(1)	-1034(1)	36(1)
N(1)	7305(1)	7985(1)	2608(1)	33(1)
N(2)	9597(1)	8096(1)	-95(1)	30(1)
C(1)	6029(2)	9009(1)	3935(1)	34(1)
C(2)	5894(2)	9772(1)	3685(1)	37(1)
C(3)	6257(2)	9964(1)	3059(1)	34(1)
C(4)	6757(2)	9406(1)	2660(1)	28(1)
C(5)	6881(2)	8624(1)	2914(1)	27(1)
C(6)	7318(2)	7280(1)	3037(1)	29(1)
C(7)	6773(2)	7597(1)	3658(1)	32(1)
C(8)	6525(2)	8431(1)	3549(1)	28(1)
C(9)	6570(2)	6621(1)	2706(1)	40(1)
C(10)	8542(2)	6999(1)	3226(1)	40(1)
C(11)	7140(2)	9547(1)	2011(1)	30(1)
C(12)	7516(2)	9531(1)	1472(1)	29(1)
C(13)	7973(2)	9504(1)	829(1)	27(1)
C(14)	7865(2)	10133(1)	383(1)	33(1)
C(15)	8320(2)	10097(1)	-247(1)	33(1)
C(16)	8892(2)	9436(1)	-442(1)	29(1)
C(17)	9045(2)	8791(1)	10(1)	26(1)
C(18)	9589(2)	7584(1)	496(1)	28(1)
C(19)	8908(2)	8084(1)	970(1)	28(1)
C(20)	8583(2)	8819(1)	632(1)	24(1)
C(21)	8904(2)	6827(1)	325(1)	40(1)
C(22)	10788(2)	7412(1)	816(1)	39(1)
C(23)	9227(2)	9943(1)	-1524(1)	42(1)
O(1')	3374(1)	2331(1)	891(1)	50(1)
O(2')	1347(1)	2103(1)	3628(1)	37(1)
O(3')	284(1)	818(1)	6205(1)	39(1)

N(1')	2650(1)	1795(1)	2504(1)	34(1)
N(2')	216(2)	1973(1)	5198(1)	35(1)
C(1')	4029(2)	566(1)	1338(1)	40(1)
C(2')	4193(2)	-139(1)	1683(1)	42(1)
C(3')	3824(2)	-224(1)	2326(1)	37(1)
C(4')	3290(2)	393(1)	2647(1)	29(1)
C(5')	3134(2)	1118(1)	2293(1)	28(1)
C(6')	2624(2)	2419(1)	1994(1)	33(1)
C(7')	3204(2)	2002(1)	1424(1)	35(1)
C(8')	3493(2)	1201(1)	1644(1)	33(1)
C(9')	1398(2)	2649(1)	1760(1)	41(1)
C(10')	3343(2)	3137(1)	2240(1)	44(1)
C(11')	2878(2)	340(1)	3300(1)	29(1)
C(12')	2464(2)	399(1)	3824(1)	29(1)
C(13')	1927(2)	481(1)	4437(1)	28(1)
C(14')	1954(2)	-116(1)	4911(1)	33(1)
C(15')	1414(2)	-29(1)	5509(1)	34(1)
C(16')	836(2)	655(1)	5646(1)	30(1)
C(17')	768(2)	1266(1)	5160(1)	27(1)
C(18')	315(2)	2443(1)	4588(1)	29(1)
C(19')	1062(2)	1904(1)	4183(1)	27(1)
C(20')	1313(2)	1186(1)	4569(1)	25(1)
C(21')	-843(2)	2584(1)	4207(1)	40(1)
C(22')	958(2)	3216(1)	4753(1)	39(1)
C(23')	333(2)	222(1)	6720(1)	43(1)

Table 3. Bond lengths [Å] and angles [°] for hamilton_pw02.

O(1)-C(7)	1.239(2)	O(2')-C(19')	1.232(2)
O(2)-C(19)	1.2377(19)	O(3')-C(16')	1.364(2)
O(3)-C(16)	1.362(2)	O(3')-C(23')	1.432(2)
O(3)-C(23)	1.430(2)	N(1')-H(1')	0.989(18)
N(1)-H(1)	0.945(18)	N(1')-C(5')	1.350(2)
N(1)-C(5)	1.346(2)	N(1')-C(6')	1.460(2)
N(1)-C(6)	1.458(2)	N(2')-H(2')	0.938(17)
N(2)-H(2)	0.947(17)	N(2')-C(17')	1.354(2)
N(2)-C(17)	1.356(2)	N(2')-C(18')	1.464(2)
N(2)-C(18)	1.463(2)	C(1')-C(2')	1.373(3)
C(1)-C(2)	1.377(3)	C(1')-C(8')	1.401(3)
C(1)-C(8)	1.394(2)	C(2')-C(3')	1.398(3)
C(2)-C(3)	1.393(2)	C(3')-C(4')	1.392(2)
C(3)-C(4)	1.389(2)	C(4')-C(5')	1.410(3)
C(4)-C(5)	1.408(3)	C(4')-C(11')	1.432(2)
C(4)-C(11)	1.429(2)	C(5')-C(8')	1.405(2)
C(5)-C(8)	1.408(2)	C(6')-C(9')	1.518(3)
C(6)-C(10)	1.523(3)	C(6')-C(10')	1.525(3)
C(6)-C(9)	1.523(3)	C(6')-C(7')	1.542(3)
C(6)-C(7)	1.538(2)	C(7')-C(8')	1.443(3)
C(7)-C(8)	1.439(3)	C(11')-C(12')	1.195(2)
C(11)-C(12)	1.199(2)	C(12')-C(13')	1.431(2)
C(12)-C(13)	1.435(2)	C(13')-C(14')	1.376(2)
C(13)-C(14)	1.380(2)	C(13')-C(20')	1.418(2)
C(13)-C(20)	1.423(2)	C(14')-C(15')	1.406(2)
C(14)-C(15)	1.409(2)	C(15')-C(16')	1.368(3)
C(15)-C(16)	1.367(3)	C(16')-C(17')	1.411(2)
C(16)-C(17)	1.411(3)	C(17')-C(20')	1.394(2)
C(17)-C(20)	1.397(2)	C(18')-C(21')	1.515(3)
C(18)-C(22)	1.517(3)	C(18')-C(22')	1.520(3)
C(18)-C(21)	1.523(3)	C(18')-C(19')	1.533(2)
C(18)-C(19)	1.535(2)	C(19')-C(20')	1.447(3)
C(19)-C(20)	1.440(3)		
O(1')-C(7')	1.231(2)	C(16)-O(3)-C(23)	117.55(16)

H(1)-N(1)-C(5)	124.7(12)	C(15)-C(16)-C(17)	118.36(16)
H(1)-N(1)-C(6)	121.8(12)	N(2)-C(17)-C(20)	113.02(16)
C(5)-N(1)-C(6)	111.28(14)	N(2)-C(17)-C(16)	126.42(16)
H(2)-N(2)-C(17)	122.5(11)	C(20)-C(17)-C(16)	120.54(17)
H(2)-N(2)-C(18)	121.9(11)	N(2)-C(18)-C(22)	112.59(15)
C(17)-N(2)-C(18)	109.99(14)	N(2)-C(18)-C(21)	110.18(16)
C(2)-C(1)-C(8)	118.87(17)	C(22)-C(18)-C(21)	112.71(17)
C(1)-C(2)-C(3)	120.48(18)	N(2)-C(18)-C(19)	102.63(15)
C(4)-C(3)-C(2)	122.35(19)	C(22)-C(18)-C(19)	110.54(16)
C(3)-C(4)-C(5)	117.10(16)	C(21)-C(18)-C(19)	107.61(15)
C(3)-C(4)-C(11)	126.20(18)	O(2)-C(19)-C(20)	130.17(17)
C(5)-C(4)-C(11)	116.69(16)	O(2)-C(19)-C(18)	122.01(17)
N(1)-C(5)-C(8)	112.05(17)	C(20)-C(19)-C(18)	107.81(15)
N(1)-C(5)-C(4)	127.40(16)	C(17)-C(20)-C(13)	120.83(17)
C(8)-C(5)-C(4)	120.54(17)	C(17)-C(20)-C(19)	106.48(16)
N(1)-C(6)-C(10)	111.12(16)	C(13)-C(20)-C(19)	132.62(16)
N(1)-C(6)-C(9)	110.75(16)	C(16')-O(3')-C(23')	117.18(15)
C(10)-C(6)-C(9)	112.06(17)	H(1')-N(1')-C(5')	128.9(11)
N(1)-C(6)-C(7)	102.18(15)	H(1')-N(1')-C(6')	119.1(11)
C(10)-C(6)-C(7)	110.45(16)	C(5')-N(1')-C(6')	111.53(15)
C(9)-C(6)-C(7)	109.85(15)	H(2')-N(2')-C(17')	123.7(11)
O(1)-C(7)-C(8)	129.06(17)	H(2')-N(2')-C(18')	123.3(11)
O(1)-C(7)-C(6)	123.22(18)	C(17')-N(2')-C(18')	110.56(15)
C(8)-C(7)-C(6)	107.72(15)	C(2')-C(1')-C(8')	118.76(18)
C(1)-C(8)-C(5)	120.65(18)	C(1')-C(2')-C(3')	120.67(19)
C(1)-C(8)-C(7)	132.62(17)	C(2')-C(3')-C(4')	122.3(2)
C(5)-C(8)-C(7)	106.73(16)	C(3')-C(4')-C(5')	116.83(17)
C(12)-C(11)-C(4)	168.9(2)	C(3')-C(4')-C(11')	125.03(19)
C(11)-C(12)-C(13)	179.2(2)	C(5')-C(4')-C(11')	118.14(17)
C(14)-C(13)-C(20)	117.29(16)	N(1')-C(5')-C(8')	111.79(18)
C(14)-C(13)-C(12)	122.23(17)	N(1')-C(5')-C(4')	127.32(16)
C(20)-C(13)-C(12)	120.48(16)	C(8')-C(5')-C(4')	120.89(17)
C(13)-C(14)-C(15)	121.54(18)	N(1')-C(6')-C(9')	111.18(16)
C(16)-C(15)-C(14)	121.41(18)	N(1')-C(6')-C(10')	111.12(16)
O(3)-C(16)-C(15)	127.39(18)	C(9')-C(6')-C(10')	112.26(17)
O(3)-C(16)-C(17)	114.24(17)	N(1')-C(6')-C(7')	102.02(16)

C(9')-C(6')-C(7')	110.40(16)
C(10')-C(6')-C(7')	109.38(16)
O(1')-C(7')-C(8')	129.08(18)
O(1')-C(7')-C(6')	123.37(19)
C(8')-C(7')-C(6')	107.54(16)
C(1')-C(8')-C(5')	120.5(2)
C(1')-C(8')-C(7')	132.34(18)
C(5')-C(8')-C(7')	107.11(17)
C(12')-C(11')-C(4')	170.7(2)
C(11')-C(12')-C(13')	177.7(2)
C(14')-C(13')-C(20')	117.33(16)
C(14')-C(13')-C(12')	122.36(17)
C(20')-C(13')-C(12')	120.29(17)
C(13')-C(14')-C(15')	121.69(18)
C(16')-C(15')-C(14')	121.43(18)
O(3')-C(16')-C(15')	127.38(17)
O(3')-C(16')-C(17')	114.69(17)
C(15')-C(16')-C(17')	117.92(16)
N(2')-C(17')-C(20')	112.69(17)
N(2')-C(17')-C(16')	126.49(16)
C(20')-C(17')-C(16')	120.82(18)
N(2')-C(18')-C(21')	111.98(16)
N(2')-C(18')-C(22')	110.45(15)
C(21')-C(18')-C(22')	112.33(17)
N(2')-C(18')-C(19')	102.31(15)
C(21')-C(18')-C(19')	110.40(16)
C(22')-C(18')-C(19')	108.88(15)
O(2')-C(19')-C(20')	130.41(17)
O(2')-C(19')-C(18')	121.82(17)
C(20')-C(19')-C(18')	107.77(15)
C(17')-C(20')-C(13')	120.77(17)
C(17')-C(20')-C(19')	106.64(16)
C(13')-C(20')-C(19')	132.56(16)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for hamilton_pw02. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(1)	43(1)	52(1)	37(1)	10(1)	14(1)	3(1)
O(2)	49(1)	36(1)	31(1)	7(1)	15(1)	11(1)
O(3)	42(1)	41(1)	27(1)	10(1)	6(1)	-2(1)
N(1)	45(1)	27(1)	29(1)	2(1)	15(1)	4(1)
N(2)	35(1)	31(1)	25(1)	5(1)	10(1)	4(1)
C(1)	30(1)	42(1)	30(1)	-6(1)	6(1)	-4(1)
C(2)	32(1)	40(1)	38(1)	-17(1)	8(1)	-3(1)
C(3)	30(1)	30(1)	42(1)	-7(1)	3(1)	0(1)
C(4)	25(1)	29(1)	30(1)	-3(1)	2(1)	-2(1)
C(5)	23(1)	30(1)	28(1)	-6(1)	4(1)	-3(1)
C(6)	33(1)	27(1)	28(1)	2(1)	6(1)	0(1)
C(7)	27(1)	42(1)	27(1)	1(1)	4(1)	-4(1)
C(8)	25(1)	33(1)	27(1)	-5(1)	4(1)	-2(1)
C(9)	41(1)	35(1)	44(1)	-1(1)	7(1)	0(1)
C(10)	38(1)	47(1)	35(1)	2(1)	7(1)	7(1)
C(11)	26(1)	23(1)	39(1)	-1(1)	0(1)	1(1)
C(12)	27(1)	21(1)	37(1)	-1(1)	-1(1)	2(1)
C(13)	26(1)	25(1)	29(1)	1(1)	1(1)	-4(1)
C(14)	35(1)	27(1)	38(1)	0(1)	1(1)	4(1)
C(15)	37(1)	28(1)	34(1)	8(1)	-3(1)	-4(1)
C(16)	29(1)	34(1)	24(1)	1(1)	1(1)	-7(1)
C(17)	25(1)	25(1)	28(1)	1(1)	0(1)	-4(1)
C(18)	32(1)	28(1)	26(1)	2(1)	8(1)	4(1)
C(19)	29(1)	29(1)	26(1)	-2(1)	6(1)	-3(1)
C(20)	24(1)	23(1)	25(1)	2(1)	1(1)	-2(1)
C(21)	46(1)	32(1)	43(1)	-5(1)	16(1)	0(1)
C(22)	36(1)	48(1)	34(1)	4(1)	6(1)	11(1)
C(23)	48(1)	48(1)	30(1)	16(1)	-1(1)	-12(1)
O(1')	48(1)	70(1)	33(1)	6(1)	12(1)	-10(1)
O(2')	44(1)	38(1)	31(1)	7(1)	13(1)	8(1)
O(3')	48(1)	43(1)	25(1)	10(1)	7(1)	-1(1)

N(1')	44(1)	31(1)	27(1)	0(1)	13(1)	4(1)
N(2')	44(1)	33(1)	28(1)	5(1)	14(1)	7(1)
C(1')	27(1)	59(2)	34(1)	-16(1)	6(1)	-5(1)
C(2')	32(1)	48(2)	45(1)	-20(1)	4(1)	3(1)
C(3')	28(1)	37(1)	45(1)	-11(1)	-1(1)	1(1)
C(4')	23(1)	32(1)	33(1)	-6(1)	2(1)	-1(1)
C(5')	22(1)	33(1)	27(1)	-8(1)	2(1)	-2(1)
C(6')	36(1)	37(1)	26(1)	3(1)	7(1)	-2(1)
C(7')	26(1)	52(2)	28(1)	-1(1)	4(1)	-10(1)
C(8')	23(1)	45(1)	31(1)	-6(1)	4(1)	-4(1)
C(9')	37(1)	48(1)	37(1)	1(1)	4(1)	-1(1)
C(10')	46(1)	42(1)	43(1)	0(1)	5(1)	-4(1)
C(11')	25(1)	24(1)	38(1)	-1(1)	-1(1)	1(1)
C(12')	25(1)	25(1)	37(1)	-1(1)	1(1)	2(1)
C(13')	23(1)	29(1)	30(1)	0(1)	0(1)	-1(1)
C(14')	30(1)	26(1)	43(1)	2(1)	-2(1)	2(1)
C(15')	33(1)	33(1)	35(1)	11(1)	-5(1)	-1(1)
C(16')	28(1)	35(1)	26(1)	4(1)	2(1)	-4(1)
C(17')	26(1)	27(1)	26(1)	-1(1)	0(1)	-3(1)
C(18')	33(1)	30(1)	23(1)	4(1)	7(1)	5(1)
C(19')	25(1)	32(1)	25(1)	0(1)	2(1)	-3(1)
C(20')	24(1)	26(1)	24(1)	0(1)	1(1)	-2(1)
C(21')	32(1)	48(1)	39(1)	-2(1)	6(1)	8(1)
C(22')	50(1)	31(1)	35(1)	0(1)	6(1)	0(1)
C(23')	46(1)	52(2)	31(1)	18(1)	1(1)	-4(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for hamilton_pw02.

	x	y	z	U(eq)
H(1)	7736(16)	8010(11)	2230(9)	49(6)
H(2)	10176(15)	8055(11)	-398(9)	40(6)
H(1A)	5788	8878	4363	40
H(2A)	5551	10170	3941	44
H(3A)	6160	10496	2900	41
H(9A)	6925	6419	2313	60
H(9B)	5807	6836	2566	60
H(9C)	6497	6186	3027	60
H(10A)	8874	6792	2827	59
H(10B)	8535	6575	3563	59
H(10C)	9006	7447	3410	59
H(14A)	7474	10603	504	40
H(15A)	8227	10541	-541	40
H(21A)	9319	6494	23	60
H(21B)	8150	6969	105	60
H(21C)	8800	6530	738	60
H(22A)	11192	7917	915	59
H(22B)	11212	7096	506	59
H(22C)	10735	7112	1233	59
H(23A)	9602	9786	-1924	64
H(23B)	9578	10435	-1338	64
H(23C)	8407	10033	-1645	64
H(1')	2252(16)	1886(11)	2915(9)	46(6)
H(2')	-333(15)	2077(11)	5503(8)	36(6)
H(1'A)	4274	622	899	48
H(2'A)	4562	-573	1483	50
H(3'A)	3942	-721	2551	44
H(9'A)	1042	2915	2126	61
H(9'B)	959	2168	1626	61
H(9'C)	1401	3013	1376	61

H(10D)	2975	3404	2601	65
H(10E)	3404	3512	1868	65
H(10F)	4113	2958	2407	65
H(14B)	2348	-598	4833	40
H(15B)	1451	-454	5825	41
H(21D)	-1309	2930	4471	59
H(21E)	-1236	2072	4125	59
H(21F)	-732	2842	3777	59
H(22D)	486	3566	5011	58
H(22E)	1122	3484	4336	58
H(22F)	1682	3098	5019	58
H(23D)	-88	410	7094	65
H(23E)	1137	122	6880	65
H(23F)	-16	-273	6539	65