Supporting Information

Diversity oriented expedient route for the synthesis of 3-tetrahydropyrimidinyl-coumarins via MCR

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EXPERIMENT SECTION

GENERAL INFORMATION

Chemical materials, solvents, and reagents were reagent grade and were used without further purification unless otherwise noted. The melting points were recorded using a Fargo melting point apparatus and are shown as uncorrected values. Column chromatography was performed using a silica gel G60 (70-230 mesh, ASTM; Merck and 230-400 mesh). Reaction progress was monitored using analytical thin-layer chromatography (TLC) on 0.25-mm Merck F-254 silica gel glass plates. Visualization was achieved by UV light (254 nm). ^1H NMR spectra and ^{13}C NMR spectra were recorded with a Bruker AVANCE 500 DRX and a 400 MHz spectrometer; chemical shifts were reported in parts per million (δ) using TMS as the internal standard with coupling constants (J) reported in hertz (Hz). The peak shapes were denoted as follows: s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet; dd, double doublet.

General procedure for one-pot synthesis of 3-substituted tetrahydropyrimidinyl coumarins (**1a-t**) via four-component reactions

To a stirring mixture of 3-amino coumarin **2** (1.0 mmol), DMAD **3** (1.0 mmol) in DMF (3 mL) for 10 min, formaldehyde **4** (4.0 mmol), and substituted amines **5a-t** (1.0 mmol) were added successively. The reaction mixture was refluxed for 3–4 h. After completion of the reaction (progress monitored by TLC), the mixture was cooled to room temperature, and water was added. The residue was extracted in ethyl acetate (3×20 mL), washed with water (2×15 mL) and brine (1×15 mL). the solvent was removed at reduced pressure to leave the crude product which was purified by column chromatography eluting with hexane/ethyl acetate (8:2) as the eluent, to afford the desired product **1a-t** (80–93%).

The following compounds given in Table 2 were prepared according to the general procedure.

Dimethyl 1-(4-methoxyphenyl)-3-(2-oxo-2H-chromen-3-yl)-1,2,3,6 tetrahydro-pyrimidine 4,5-dicarboxylate 1a.

Yield: 90%; yellow solid; mp 130–132 °C; ¹H NMR (500 MHz, DMSO-*d*₆) δ 7.65 (dd, *J* = 7.0 and 1.0 Hz, 1H, ArH), 7.57 (t, *J* = 7.0 Hz, 1H, ArH), 7.41 (d, *J* = 8.5 Hz, 1H, ArH), 7.37 (s, 1H, ArH), 7.34 (t, *J* = 7.5 Hz, 1H), 7.01 (d, *J* = 9.0 Hz, 2H, ArH), 6.80 (d, *J* = 9.0 Hz, 2H, ArH), 4.90 (s, 2H, CH₂), 4.13 (s, 2H, CH₂), 3.65 (s, 6H, 1 × OCH₃, 1 × COOCH₃), 3.55 (s, 3H, COOCH₃); ¹³C NMR (125 MHz, DMSO-*d*₆) δ 165.3, 163.6, 158.1, 153.9, 151.6, 144.4, 141.4, 133.9, 131.4, 129.3, 128.2, 125.0, 119.7, 119.2, 118.5, 116.0, 114.4, 114.2, 103.2, 66.7, 55.2, 52.5, 51.6, 47.3; HRMS [ES⁺] calcd for C₂₄H₂₂N₂O₇, 451.1505 [M+H]⁺, found 451.1505.

Dimethyl 3-(2-oxo-2H-chromen-3-yl)-1-(3,4,5-trimethoxyphenyl)-1,2,3,6 tetrahydro-pyrimidine -4,5-dicarboxylate 1b.

Yield: 91%; yellow solid; mp 151–153 °C; ¹H NMR (500 MHz, DMSO-*d*₆) δ 7.72 (dd, *J* = 6.5 and 1.5 Hz, 1H, ArH), 7.61–7.58 (m, 2H, ArH), 7.44 (d, *J* = 8.0 Hz, 1H, ArH), 7.35 (t, *J* = 7.5 Hz, 1H, ArH), 6.33 (s, 2H, ArH), 5.00 (s, 2H, CH₂), 4.23 (s, 2H, CH₂), 3.65 (s, 9H, 2 × OCH₃, 1 × COOCH₃), 3.54 (s, 6H, 1 × OCH₃, 1 × COOCH₃); ¹³C NMR (125 MHz, DMSO-*d*₆) δ 165.0, 163.2, 158.0, 152.9, 151.4, 144.3, 134.3, 131.7, 131.3, 128.9, 128.0, 124.8, 123.5, 118.3, 115.8, 115.7, 102.1, 95.5, 95.4, 64.9, 59.8, 55.5, 52.9, 52.3, 51.3, 47.7; HRMS [ES⁺] calcd for C₂₆H₂₆N₂O₉, 511.1717 [M+H]⁺, found 511.1752.

Dimethyl 1-(3-methoxyphenyl)-3-(2-oxo-2H-chromen-3-yl)-1,2,3,6-tetrahydro-pyrimidine-4,5-dicarboxylate 1c.

Yield: 82%; yellow solid; mp 142–144 °C; ^1H NMR (500 MHz, $\text{DMSO}-d_6$) δ 7.70 (dd, J = 6.5 and 1.5 Hz, 1H, ArH), 7.62–7.58 (m, 1H, ArH), 7.52 (s, 1H, ArH), 7.48–7.46 (m, 1H, ArH), 7.37–7.35 (m, 1H, ArH), 7.13 (t, J = 8.0 Hz, 1H, ArH), 6.65 (dd, J = 6.0 and 2.0 Hz, 1H, ArH), 6.57 (t, J = 2.5 Hz, 1H, ArH), 6.44 (dd, J = 6.0 and 2.0 Hz, 1H, ArH), 5.03 (s, 2H, CH_2), 4.22 (s, 2H, CH_2), 3.68 (s, 3H, OCH_3), 3.66 (s, 3H, COOCH_3), 3.56 (s, 3H, COOCH_3); ^{13}C NMR (125 MHz, $\text{DMSO}-d_6$) δ 165.2, 163.5, 160.2, 158.1, 151.6, 149.0, 144.4, 134.2, 129.9, 129.5, 128.3, 125.0, 123.7, 118.5, 116.0, 109.5, 103.0, 95.6, 65.1, 54.9, 52.6, 51.4, 46.7; HRMS $[\text{ES}^+]$ calcd for $\text{C}_{24}\text{H}_{22}\text{N}_2\text{O}_7$, 451.1505 $[\text{M}+\text{H}]^+$, found 451.1505.

Dimethyl 1-(4-chlorophenyl)-3-(2-oxo-2H-chromen-3-yl)-1,2,3,6-tetrahydropyrimidine-4,5-dicarboxylate 1d.

Yield: 82%; yellow solid; mp 130–132 °C; ^1H NMR (500 MHz, $\text{DMSO}-d_6$) δ 7.69 (dd, J = 6.5 and 1.5 Hz, 1H, ArH), 7.59 (t, J = 7.5 Hz, 1H, ArH), 7.48–7.43 (m, 2H, ArH), 7.34 (t, J = 7.0 Hz, 1H, ArH), 7.24 (dd, J = 4.5 and 2.0 Hz, 2H, ArH), 7.08 (dd, J = 4.5 and 2.0 Hz, 2H, ArH), 5.01 (s, 2H, CH_2), 4.20 (s, 2H, CH_2), 3.66 (s, 3H, COOCH_3), 3.55 (s, 3H, COOCH_3); ^{13}C NMR (125 MHz, $\text{DMSO}-d_6$): δ 165.2, 163.2, 158.4, 152.0, 146.7, 149.1, 144.7, 134.4, 131.6, 129.9, 128.2, 127.5, 125.2, 124.1, 116.3, 102.6, 95.5, 65.3, 51.6, 46.1; HRMS $[\text{ES}^+]$ calcd for $\text{C}_{23}\text{H}_{19}\text{ClN}_2\text{O}_6$, 455.1010 $[\text{M}+\text{H}]^+$, found 455.1048.

Dimethyl 1-(3,5-dimethoxyphenyl)-3-(2-oxo-2H-chromen-3-yl)-1,2,3,6-tetrahydropyrimidine - 4,5-dicarboxylate 1e.

Yield: 84%; yellow solid; mp 195–196 °C; ^1H NMR (500 MHz, CDCl_3) δ 7.48 (t, J = 7.5 Hz, 1H, ArH), 7.35–7.31 (m, 2H, ArH), 7.26–7.21 (m, 2H, ArH), 6.12 (s, 2H, ArH), 6.01 (s, 1H, ArH), 4.96 (s, 2H, CH_2), 4.22 (s, 2H, CH_2), 3.77 (s, 3H, OCH_3), 3.70 (s, 6H, 1 ×

OCH₃, 1 × COOCH₃), 3.68 (s, 3H, COOCH₃); ¹³C NMR (125 MHz, CDCl₃) δ 165.5, 164.0, 161.5, 152.0, 149.7, 144.3, 133.2, 131.1, 129.3, 127.5, 124.9, 116.5, 107.5, 96.2, 92.9, 66.1, 55.3, 52.9, 51.9, 47.5; HRMS [ES⁺] calcd for C₂₅H₂₄N₂O₈, 481.1611 [M+H]⁺, found 481.1629.

Dimethyl 1-(2,5-dimethoxyphenyl)-3-(2-oxo-2H-chromen-3-yl)-1,2,3,6-tetrahydro-pyrimidine-4,5-dicarboxylate 1f.

Yield: 81%; yellow solid; mp 174–176 °C; ¹H NMR (500 MHz, CDCl₃) δ 7.46–7.43 (m, 1H, ArH), 7.33–7.29 (m, 2H, ArH), 7.25–7.21 (m, 1H, ArH), 7.10 (s, 1H, ArH), 6.70–6.66 (m, 2H, ArH), 6.46 (dd, *J* = 5.5 and 3.0 Hz, 1H, ArH), 4.89 (s, 2H, CH₂), 4.17 (s, 2H, CH₂), 3.78 (s, 3H, OCH₃), 3.76 (s, 3H, OCH₃), 3.69 (s, 3H, COOCH₃), 3.66 (s, 3H, COOCH₃); ¹³C NMR (125 MHz, CDCl₃) δ 165.7, 164.2, 158.5, 153.9, 151.9, 146.3, 144.3, 137.5, 132.6, 130.8, 129.8, 127.4, 124.8, 118.8, 116.4, 112.1, 108.2, 107.4, 106.9, 66.7, 55.9, 55.7, 52.9, 51.9, 48.1; HRMS [ES⁺] calcd for C₂₅H₂₄N₂O₈, 481.1611 [M+H]⁺, found 481.1642.

Dimethyl 1-(2-methoxy-5-nitrophenyl)-3-(2-oxo-2H-chromen-3-yl)-1,2,3,6-tetrahydro-pyrimidine-4,5-dicarboxylate 1g.

Yield: 82%; yellow solid; mp 165–167 °C; ¹H NMR (500 MHz, DMSO-*d*₆) δ 7.93 (dd, *J* = 7.0 and 2.0 Hz, 1H, ArH), 7.76 (d, *J* = 2.5 Hz, 1H, ArH), 7.70 (d, *J* = 7.5 Hz, 1H, ArH), 7.59 (t, *J* = 7.5 Hz, 1H, ArH), 7.54 (s, 1H, ArH), 7.44 (d, *J* = 8.5 Hz, 1H, ArH), 7.36 (t, *J* = 7.5 Hz, 1H, ArH), 7.13 (d, *J* = 9.0 Hz, 1H), 4.90 (s, 2H, CH₂), 4.18 (s, 2H, CH₂), 3.82 (s, 3H, OCH₃), 3.69 (s, 3H, COOCH₃), 3.59 (s, 3H, COOCH₃); ¹³C NMR (125 MHz, DMSO-*d*₆) δ 165.5, 164.0, 158.4, 157.4, 152.0, 144.1, 141.2, 137.5, 134.3, 131.9, 129.4, 128.8,

125.6, 120.2, 119.0, 116.4, 114.3, 112.1, 106.1, 65.9, 56.9, 53.2, 52.2, 47.9; HRMS [ES⁺]calcd for C₂₄H₂₁N₃O₉, 496.1356 [M+H]⁺, found 496.1356.

Dimethyl 1-(2-methoxy-4-nitrophenyl)-3-(2-oxo-2H-chromen-3-yl)-1,2,3,6-tetrahydro-pyrimidine-4,5-dicarboxylate 1h.

Yield: 86%; yellow solid; mp 170–172 °C; ¹H NMR (500 MHz, DMSO-*d*₆) δ 7.86 (dd, *J* = 6.0 and 2.0 Hz, 1H, ArH), 7.70 (d, *J* = 8.0 Hz, 1H, ArH), 7.65 (d, *J* = 2.5 Hz, 1H, ArH), 7.61–7.57 (m, 1H, ArH), 7.52 (s, 1H, ArH), 7.45 (d, *J* = 8.0 Hz, 1H, ArH), 7.35 (t, *J* = 7.5 Hz, 1H, ArH), 7.11 (d, *J* = 9.0 Hz, 1H, ArH), 5.00 (s, 2H, CH₂), 4.24 (s, 2H, CH₂), 3.74 (s, 3H, OCH₃), 3.70 (s, 3H, COOCH₃), 3.60 (s, 3H, COOCH₃); ¹³C NMR (125 MHz, DMSO-*d*₆) δ 165.5, 164.0, 158.4, 151.9, 150.6, 145.8, 144.2, 143.3, 141.9, 134.1, 131.6, 129.3, 128.4, 125.5, 119.1, 116.5, 108.8, 106.9, 65.3, 56.4, 53.2, 52.3, 48.0; HRMS [ES⁺]: calcd for C₂₄H₂₁N₃O₉, 496.1356 [M+H]⁺, found 496.1359.

Dimethyl 1-(4-methyl-3-nitrophenyl)-3-(2-oxo-2H-chromen-3-yl)-1,2,3,6-tetrahydro-pyrimidine-4,5-dicarboxylate 1i.

Yield: 85%; yellow solid; mp 109–111 °C; ¹H NMR (500 MHz, DMSO-*d*₆) δ 7.63–7.58 (m, 2H, ArH), 7.56 (s, 1H, ArH), 7.45 (d, *J* = 8.5 Hz, 1H, ArH), 7.39–7.36 (m, 2H, ArH), 7.33 (t, *J* = 8.5 Hz, 1H, ArH), 7.70 (dd, *J* = 6.5 and 1.5 Hz, 1H, ArH), 5.07 (s, 2H, CH₂), 4.26 (s, 2H, CH₂), 3.67 (s, 3H, COOCH₃), 3.55 (s, 3H, COOCH₃), 2.37 (s, 3H, CH₃); ¹³C NMR (125 MHz, DMSO-*d*₆) δ 165.5, 163.8, 158.6, 152.2, 150.0, 147.0, 145.0, 135.2, 133.7, 132.0, 129.5, 128.9, 125.4, 123.8, 122.1, 119.0, 116.5, 112.0, 102.8, 64.9, 53.1, 52.1, 47.1, 18.8; HRMS [ES⁺] calcd for C₂₄H₂₁N₃O₈, 480.1407 [M+H]⁺, found 480.1454.

Dimethyl 1-(2-methyl-5-nitrophenyl)-3-(2-oxo-2H-chromen-3-yl)-1,2,3,6-tetrahydropyrimidine-4,5-dicarboxylate 1j.

Yield: 88%; yellow solid; mp 123–124 °C; ^1H NMR (500 MHz, $\text{DMSO}-d_6$) δ 7.93 (d, J = 2.0 Hz, 1H, ArH), 7.87 (dd, J = 6.0 and 2.5 Hz, 1H, ArH), 7.67 (dd, J = 6.5 and 1.5 Hz, 1H, ArH), 7.59–7.57 (m, 2H, ArH), 7.45–7.41 (m, 2H, ArH), 7.35 (t, J = 7.0 Hz, 1H, ArH), 4.81 (s, 2H, CH_2), 4.09 (s, 2H, CH_2), 3.67 (s, 3H, COOCH_3), 3.60 (s, 3H, COOCH_3), 2.36 (s, 3H, CH_3); ^{13}C NMR (125 MHz, $\text{DMSO}-d_6$) δ 165.5, 164.0, 158.5, 152.0, 148.8, 146.7, 144.8, 141.2, 134.9, 132.4, 132.0, 129.4, 128.8, 125.5, 119.1, 118.9, 116.5, 115.8, 103.7, 66.9, 53.2, 52.1, 48.9, 18.7; HRMS $[\text{ES}^+]$ calcd for $\text{C}_{24}\text{H}_{21}\text{N}_3\text{O}_8$, 480.1407 $[\text{M}+\text{H}]^+$, found 480.1427.

Dimethyl 1-(2-methyl-3-nitrophenyl)-3-(2-oxo-2H-chromen-3-yl)-1,2,3,6-tetrahydropyrimidine-4,5-dicarboxylate 1k.

Yield: 86%; yellow solid; mp 122–124 °C; ^1H NMR (500 MHz, $\text{DMSO}-d_6$) δ 7.69 (dd, J = 6.5 and 1.0 Hz, 1H, ArH), 7.62–7.56 (m, 3H, ArH), 7.51 (d, J = 8.0 Hz, 1H, ArH), 7.45–7.39 (m, 2H, ArH), 7.34 (t, J = 7.5 Hz, 1H, ArH), 4.75 (s, 2H, CH_2), 4.05 (s, 2H, CH_2), 3.66 (s, 3H, COOCH_3), 3.60 (s, 3H, COOCH_3), 2.34 (s, 3H, CH_3); ^{13}C NMR (125 MHz, $\text{DMSO}-d_6$) δ 165.5, 164.3, 158.6, 152.0, 149.9, 144.9, 135.1, 132.1, 128.8, 127.7, 126.1, 125.0, 119.8, 118.8, 116.4, 103.7, 67.4, 53.1, 52.1, 49.6, 14.2; HRMS $[\text{ES}^+]$ calcd for $\text{C}_{24}\text{H}_{21}\text{N}_3\text{O}_8$, 480.1407 $[\text{M}+\text{H}]^+$, found 480.1419.

Dimethyl 1-(5-methoxy-2-methylphenyl)-3-(2-oxo-2H-chromen-3-yl)-1,2,3,6-tetrahydropyrimidine-4,5-dicarboxylate 1l.

Yield: 90%; yellow solid; mp 152–154 °C; ^1H NMR (500 MHz, $\text{DMSO}-d_6$) δ 7.68 (d, J = 7.0 Hz, 1H, ArH), 7.61 (s, 1H, ArH), 7.55–7.58 (m, 2H, ArH), 7.40 (d, J = 8.5 Hz, 1H,

ArH), 7.34 (t, $J = 7.5$ Hz, 1H, ArH), 6.84 (s, 1H, ArH), 6.62 (s, 1H, ArH), 4.68 (s, 2H, CH₂), 3.95 (s, 2H, CH₂), 3.69 (s, 3H, OCH₃), 3.63 (s, 3H, COOCH₃), 3.59 (s, 3H, COOCH₃), 2.02 (s, 3H, CH₃); ¹³C NMR (125 MHz, DMSO-*d*₆) δ 165.8, 164.2, 158.5, 155.8, 152.0, 146.8, 144.5, 134.5, 132.3, 131.8, 129.4, 128.6, 125.5, 124.4, 123.3, 119.0, 116.4, 104.8, 104.3, 67.8, 55.7, 53.1, 52.0, 49.1, 17.4; HRMS [ES⁺] calcd for C₂₅H₂₄N₂O₇, 465.1662 [M+H]⁺, found 465.1679.

Dimethyl 1-(2,4-dimethoxyphenyl)-3-(2-oxo-2H-chromen-3-yl)-1,2,3,6-tetrahydropyrimidine-4,5-dicarboxylate 1m.

Yield: 87%; yellow solid; mp 174–176 °C; ¹H NMR (500 MHz, DMSO-*d*₆) δ 7.64 (dd, $J = 7.0$ and 1.0 Hz, 1H, ArH), 7.56 (t, $J = 8.5$ Hz, 1H, ArH), 7.40 (d, $J = 8.5$ Hz, 1H, ArH), 7.34–7.32 (m, 2H, ArH), 6.94 (d, $J = 9.5$ Hz, 1H, ArH), 6.47–7.45 (m, 2H, ArH), 4.72 (s, 2H, CH₂), 4.01 (s, 2H, CH₂), 3.67 (s, 9H, 2 $\times$ OCH₃, 1 $\times$ COOCH₃), 3.66 (s, 3H, COOCH₃); ¹³C NMR (125 MHz, DMSO-*d*₆) δ 165.8, 164.2, 158.4, 156.7, 153.3, 151.8, 144.1, 133.4, 131.7, 130.7, 129.7, 128.5, 125.5, 120.5, 119.0, 116.4, 107.0, 104.5, 100.4, 67.2, 55.9, 55.6, 53.1, 52.1, 47.9; HRMS [ES⁺] calcd for C₂₅H₂₄N₂O₈, 481.1611 [M+H]⁺, found 481.1629.

Dimethyl 1-(4-cyanophenyl)-3-(2-oxo-2H-chromen-3-yl)-1,2,3,6-tetrahydropyrimidine-4,5-dicarboxylate 1n.

Yield: 84%; yellow solid; mp 165–167 °C; ¹H NMR (500 MHz, DMSO-*d*₆) δ 7.70 (d, $J = 7.0$ Hz, 1H, ArH), 7.63–7.58 (m, 4H, ArH), 7.45 (d, $J = 8.0$ Hz, 1H, ArH), 7.36 (t, $J = 6.5$, 1H, ArH), 7.18 (d, $J = 8.0$ Hz, 2H, ArH), 5.13 (s, 2H, CH₂), 4.31 (s, 2H, CH₂), 3.68 (s, 3H, COOCH₃), 3.52 (s, 3H, COOCH₃); ¹³C NMR (125 MHz, DMSO-*d*₆) δ 165.3, 163.8, 158.6, 152.2, 151.2, 144.9, 135.0, 134.0, 132.0, 129.3, 128.9, 125.4, 120.0, 119.0, 116.5,

116.2, 103.7, 101.1, 63.6, 53.1, 52.2, 46.2; HRMS [ES⁺] calcd for C₂₄H₁₉N₃O₆, 446.1352 [M+H]⁺, found 446.1333.

Dimethyl 1-(4-bromophenyl)-3-(2-oxo-2H-chromen-3-yl)-1,2,3,6-tetrahydropyrimidine-4,5-dicarboxylate 1o.

Yield: 85%; yellow solid; mp 130–132 °C; ¹H NMR (500 MHz, DMSO-*d*₆) δ 7.65 (dd, *J* = 6.5 and 1.5 Hz, 1H, ArH), 7.57 (t, *J* = 7.0 Hz, 1H, ArH), 7.42 (d, *J* = 8.5 Hz, 1H, ArH), 7.37 (s, 1H, ArH), 7.34 (t, *J* = 7.5 Hz, 1H, ArH), 7.01 (d, *J* = 9.0 Hz, 2H, ArH), 6.80 (d, *J* = 9.0 Hz, 2H, ArH), 4.90 (s, 2H, CH₂), 4.13 (s, 2H, CH₂), 3.65 (s, 6H, 2 × COOCH₃); ¹³C NMR (125 MHz, DMSO-*d*₆) δ 165.3, 163.6, 158.1, 153.9, 151.6, 144.4, 141.4, 133.9, 131.4, 129.3, 128.2, 125.0, 119.7, 119.2, 118.5, 116.0, 114.4, 114.2, 103.2, 66.7, 55.2, 52.5; HRMS [ES⁺] calcd for C₂₃H₁₉BrN₂O₆, 499.0505 [M+H]⁺, found 499.0519.

Dimethyl 1-(2-chloro-5-nitrophenyl)-3-(2-oxo-2H-chromen-3-yl)-1,2,3,6-tetrahydropyrimidine-4,5-dicarboxylate 1p.

Yield: 82%; yellow solid; mp 165–166 °C; ¹H NMR (500 MHz, DMSO-*d*₆) δ 7.77–7.76 (m, 1H, ArH), 7.94 (dd, *J* = 6.5 and 1.5 Hz, 1H, ArH), 7.70 (d, *J* = 7.5 Hz, 1H, ArH), 7.59 (t, *J* = 7.5 Hz, 1H, ArH), 7.55 (s, 1H, ArH), 7.44 (d, *J* = 8.5, 1H, ArH), 7.36 (t, *J* = 7.0 Hz, 1H, ArH), 7.13 (d, *J* = 9.0 Hz, 1H, ArH), 4.90 (s, 2H, CH₂), 4.18 (s, 2H, CH₂), 3.82 (s, 3H, COOCH₃), 3.69 (s, 3H, COOCH₃); ¹³C NMR (125 MHz, DMSO-*d*₆) δ 165.5, 164.0, 158.4, 157.4, 152.0, 144.1, 141.2, 137.5, 134.3, 131.9, 129.4, 128.8, 125.6, 120.2, 119.0, 116.4, 114.3, 112.1, 106.1, 65.9, 56.9, 53.2; HRMS [ES⁺] calcd for C₂₃H₁₈ClN₃O₈, 500.0861 [M+H]⁺, found 500.0832.

Dimethyl 1-(4-methylpyridin-2-yl)-3-(2-oxo-2H-chromen-3-yl)-1,2,3,6-tetrahydropyrimidine-4,5-dicarboxylate 1q.

Yield: 80%; brown solid; mp 120–122 °C; ^1H NMR (500 MHz, DMSO- d_6) δ 7.96 (d, J = 8.0 Hz, 1H, ArH), 7.74 (d, J = 7.0 Hz, 1H, ArH), 7.67–7.63 (m, 4H, ArH), 7.45 (d, J = 8.0, 1H, ArH), 7.12 (d, J = 8.0, 1H, ArH), 5.13 (s, 2H, CH₂), 4.31 (s, 2H, CH₂), 3.68 (s, 3H, COOCH₃), 3.52 (s, 3H, COOCH₃), 2.39 (s, 3H, CH₃); ^{13}C NMR (125 MHz, DMSO- d_6) δ 165.5, 164.0, 158.4, 151.9, 150.6, 145.8, 144.2, 143.3, 141.9, 137.5, 134.1, 131.6, 129.3, 128.4, 125.5, 119.1, 116.5, 108.8, 106.9, 65.3, 56.4, 53.2, 52.3, 48.0, 16.0; HRMS [ES⁺] calcd for C₂₄H₂₁N₃O₆, 436.1509 [M+H]⁺, found 436.1493.

Dimethyl 1-cyclohexyl-3-(2-oxo-2H-chromen-3-yl)-1,2,3,6-tetrahydropyrimidine-4,5-dicarboxylate 1r.

Yield: 93%; brown gum; ^1H NMR (500 MHz, DMSO- d_6) δ 7.74 (dd, J = 6.5 and 1.5 Hz, 1H, ArH), 7.65 (s, 1H, ArH), 7.60–7.56 (m, 1H, ArH), 7.42 (d, J = 8.0 Hz, 1H, ArH), 7.37–7.34 (m, 1H, ArH), 4.39 (s, 2H, CH₂), 3.62 (s, 3H, COOCH₃), 3.61 (s, 2H, CH₂), 3.56 (s, 3H, COOCH₃), 2.61 (m, 1H, CH), 1.80–1.68 (m, 6H, 3 $\times$ CH₂), 1.40–1.20 (m, 4H, 2 $\times$ CH₂); ^{13}C NMR (125 MHz, DMSO- d_6) δ 166.0, 164.3, 162.7, 158.6, 152.0, 144.6, 134.1, 131.7, 129.7, 128.7, 125.4, 119.2, 116.4, 103.8, 66.6, 58.1, 52.9, 51.9, 45.5, 36.2, 30.6, 25.9, 25.1; HRMS [ES⁺] calcd for C₂₃H₂₆N₂O₆, 427.1869 [M+H]⁺, found 427.1892.

Dimethyl 1-(2-morpholinoethyl)-3-(2-oxo-2H-chromen-3-yl)-1,2,3,6-tetrahydropyrimidine-4,5-dicarboxylate 1s.

Yield: 90%; brown gum; ^1H NMR (500 MHz, DMSO- d_6) δ 7.73–7.71 (m, 2H, ArH), 7.58 (t, J = 7.5 Hz, 1H, ArH), 7.42 (d, J 8.0 Hz, 1H, ArH), 7.36 (t, J = 7.5 Hz, 1H, ArH), 4.40 (s, 2H, CH₂), 3.61 (s, 3H, COOCH₃), 3.60 (s, 2H, CH₂), 3.57 (s, 3H, COOCH₃), 3.45 (t,

4H, $J = 4.5$, $2 \times \text{CH}_2$), 2.74–2.72 (t, 4H, $J = 1.0$, $2 \times \text{CH}_2$), 2.50–2.49 (m, 2H, CH_2), 2.43 (t, $J = 6.5$, 2H, CH_2); ^{13}C NMR (125 MHz, $\text{DMSO}-d_6$) δ 166.3, 164.2, 162.7, 158.6, 152.0, 143.9, 133.9, 131.7, 130.3, 128.7, 125.4, 119.2, 116.4, 102.3, 69.3, 66.6, 57.6, 53.9, 52.9, 51.8, 49.0, 48.6, 36.2, 31.2; HRMS $[\text{ES}^+]$ calcd for $\text{C}_{23}\text{H}_{27}\text{N}_3\text{O}_7$, 458.1927 $[\text{M}+\text{H}]^+$, found 458.1962.

Dimethyl 1-(2-(diethylamino)ethyl)-3-(2-oxo-2H-chromen-3-yl)-1,2,3,6-tetrahydropyrimidine-4,5-dicarboxylate 1t.

Yield: 89%; brown gum; ^1H NMR (500 MHz, $\text{DMSO}-d_6$) δ 7.73–7.71 (m, 2H, ArH), 7.60–7.56 (m, 1H, ArH), 7.42 (d, $J = 8.0$ Hz, 1H, ArH), 7.38–7.35 (m, 1H, ArH), 4.40 (s, 2H, CH_2), 3.64 (s, 3H, COOCH_3), 3.59 (s, 2H, CH_2), 3.56 (s, 3H, COOCH_3), 3.32–3.29 (m, 4H, $2 \times \text{CH}_2$), 2.74–2.72 (m, 4H, $2 \times \text{CH}_2$), 1.40–1.20 (m, 6H, $2 \times \text{CH}_3$); ^{13}C NMR (125 MHz, $\text{DMSO}-d_6$) δ 166.3, 164.1, 162.2, 152.0, 158.6, 143.9, 133.9, 131.7, 130.3, 128.7, 125.4, 119.2, 116.4, 102.3, 66.3, 61.6, 53.9, 52.9, 49.0, 48.6, 36.2, 12.2; HRMS $[\text{ES}^+]$ calcd for $\text{C}_{23}\text{H}_{29}\text{N}_3\text{O}_6$, 444.2135 $[\text{M}+\text{H}]^+$, found 444.2110.

¹H and ¹³C NMR spectra of compound 1a (500 MHz, DMSO-*d*₆)

DEPT and HRMS of Compound 1a

¹H and ¹³C spectra of compound 1b (500 MHz, DMSO-d₆)

¹H

DEPT and HRMS of compound 1b

DEPT

20141119_BO_2726_ESI_POS

¹H and ¹³C NMR spectra of compound 1g (500 MHz, DMSO-d₆).

DEPT and HRMS of Compound 1g

20140717_BO2635_ESI_POS_0

¹H and ¹³C NMR spectra of compound 1i (500 MHz, DMSO-d₆)

¹H and ¹³C NMR spectra of compound 1n (500 MHz, DMSO-d₆)

¹H NMR

¹³C NMR

HRMS of Compound 1n.

X-ray Structure Report

for

Compound **1i**

Experimental

Data Collection

A yellow block crystal of $C_{24}H_{21}N_3O_8$ having approximate dimensions of 0.570 x 0.560 x 0.300 mm was mounted on a glass fiber. All measurements were made on a Rigaku SCX mini diffractometer using graphite monochromated Mo-K α radiation.

The crystal-to-detector distance was 52.00 mm.

Cell constants and an orientation matrix for data collection corresponded to a primitive triclinic cell with dimensions:

$$a = 9.3279(6) \text{ \AA} \quad \alpha = 70.589(3)^\circ$$

$$b = 10.2240(7) \text{ \AA} \quad \beta = 86.780(3)^\circ$$

$$c = 14.0365(9) \text{ \AA} \quad \gamma = 81.824(3)^\circ$$

$$V = 1249.7(2) \text{ \AA}^3$$

For $Z = 2$ and F.W. = 479.45, the calculated density is 1.274 g/cm³. Based on a statistical analysis of intensity distribution, and the successful solution and refinement of the structure, the space group was determined to be:

P-1 (#2)

The data were collected at a temperature of $20 \pm 1^{\circ}\text{C}$ to a maximum 2θ value of 55.0° . A total of 540 oscillation images were collected. A sweep of data was done using ω oscillations from -120.0 to 60.0° in 1.0° steps. The exposure rate was 10.0 [sec./ $^{\circ}$]. The detector swing angle was -30.80° . A second sweep was performed using ω oscillations from -120.0 to 60.0° in 1.0° steps. The exposure rate was 10.0 [sec./ $^{\circ}$]. The detector swing angle was -30.80° . Another sweep was performed using ω oscillations from -120.0 to 60.0° in 1.0° steps. The exposure rate was 10.0 [sec./ $^{\circ}$]. The detector swing angle was -30.80° . The crystal-to-detector distance was 52.00 mm. Readout was performed in the 0.146 mm pixel mode.

Data Reduction

Of the 12848 reflections that were collected, 5699 were unique ($R_{\text{int}} = 0.0229$); equivalent reflections were merged. Data were collected and processed using CrystalClear (Rigaku).

The linear absorption coefficient, μ , for Mo-K α radiation is 0.972 cm^{-1} . An empirical absorption correction was applied which resulted in transmission factors ranging from 0.740 to 0.971 . The data were corrected for Lorentz and polarization effects. A correction for secondary extinction² was applied (coefficient = 0.003870).

Structure Solution and Refinement

The structure was solved by direct methods³ and expanded using Fourier techniques. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were refined using the riding model. The final cycle of full-matrix least-squares refinement⁴ on F^2 was based on 5699 observed reflections and 317 variable parameters and converged (largest parameter shift was 0.01 times its esd) with unweighted and weighted agreement factors of:

$$R1 = \Sigma ||F_o| - |F_c|| / \Sigma |F_o| = 0.1608$$

$$wR2 = [\Sigma (w (F_o^2 - F_c^2)^2) / \Sigma w(F_o^2)^2]^{1/2} = 0.4840$$

The standard deviation of an observation of unit weight⁵ was 2.00. Unit weights were used. The maximum and minimum peaks on the final difference Fourier map corresponded to 3.77 and -0.82 e-/Å³, respectively.

Neutral atom scattering factors were taken from Cromer and Waber⁶. Anomalous dispersion effects were included in F_{calc} ⁷; the values for $\Delta f'$ and $\Delta f''$ were those of Creagh and McAuley⁸. The values for the mass attenuation coefficients are those of Creagh and Hubbell⁹. All calculations were performed

using the CrystalStructure¹⁰ crystallographic software package except for refinement, which was performed using SHELXL-97¹¹.

References

- (1) CrystalClear: Rigaku Corporation, 1999. CrystalClear Software User's Guide, Molecular Structure Corporation, (c) 2000. J.W. Pflugrath (1999) Acta Cryst. D55, 1718-1725.

- (2) Larson, A.C. (1970), Crystallographic Computing, 291-294. F.R. Ahmed, ed. Munksgaard, Copenhagen (equation 22, with V replaced by the cell volume).

- (3) SIR2004: M.C. Burla, R. Caliendo, M. Camalli, B. Carrozzini, G.L. Cascarano, L. De Caro, C. Giacovazzo, G. Polidori, R. Spagna (2005)

- (4) Least Squares function minimized: (SHELXL97)

$$\sum w(F_o^2 - F_c^2)^2 \quad \text{where } w = \text{Least Squares weights.}$$

- (5) Standard deviation of an observation of unit weight:

$$[\sum w(F_o^2 - F_c^2)^2 / (N_o - N_v)]^{1/2}$$

where: N_o = number of observations

N_v = number of variables

(6) Cromer, D. T. & Waber, J. T.; "International Tables for X-ray Crystallography", Vol. IV, The Kynoch Press, Birmingham, England, Table 2.2 A (1974).

(7) Ibers, J. A. & Hamilton, W. C.; Acta Crystallogr., 17, 781 (1964).

(8) Creagh, D. C. & McAuley, W.J. ; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.6.8, pages 219-222 (1992).

(9) Creagh, D. C. & Hubbell, J.H.; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.4.3, pages 200-206 (1992).

(10) CrystalStructure 4.0: Crystal Structure Analysis Package, Rigaku Corporation (2000-2010). Tokyo 196-8666, Japan.

(11) SHELX97: Sheldrick, G.M. (2008). Acta Cryst. A64, 112-122.

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$\text{C}_{24}\text{H}_{21}\text{N}_3\text{O}_8$
Formula Weight	479.45
Crystal Color, Habit	yellow, block
Crystal Dimensions	0.570 X 0.560 X 0.300 mm
Crystal System	triclinic
Lattice Type	Primitive
Lattice Parameters	$a = 9.3279(6) \text{ \AA}$ $b = 10.2240(7) \text{ \AA}$ $c = 14.0365(9) \text{ \AA}$ $\alpha = 70.589(3)^\circ$ $\beta = 86.780(3)^\circ$ $\gamma = 81.824(3)^\circ$ $V = 1249.7(2) \text{ \AA}^3$

Space Group	P-1 (#2)
Z value	2
D _{calc}	1.274 g/cm ³
F ₀₀₀	500.00
$\mu(\text{MoK}\alpha)$	0.972 cm ⁻¹

B. Intensity Measurements

Diffractometer	SCX mini
Radiation	MoK α ($\lambda = 0.71075 \text{ \AA}$) graphite monochromated
Voltage, Current	50kV, 30mA
Temperature	20.0°C
Detector Aperture	75 mm (diameter)
Data Images	540 exposures
ω oscillation Range	-120.0 - 60.0°
Exposure Rate	10.0 sec./°
Detector Swing Angle	-30.80°
ω oscillation Range	-120.0 - 60.0°

Exposure Rate	10.0 sec./ $^{\circ}$
Detector Swing Angle	-30.80 $^{\circ}$
ω oscillation Range	-120.0 - 60.0 $^{\circ}$
Exposure Rate	10.0 sec./ $^{\circ}$
Detector Swing Angle	-30.80 $^{\circ}$
Detector Position	52.00 mm
Pixel Size	0.146 mm
$2\theta_{\max}$	55.0 $^{\circ}$
No. of Reflections Measured	Total: 12848 Unique: 5699 ($R_{\text{int}} = 0.0229$)
Corrections	Lorentz-polarization Absorption (trans. factors: 0.740 - 0.971) Secondary Extinction (coefficient: 3.87000e-003)

C. Structure Solution and Refinement

Structure Solution	Direct Methods
Refinement	Full-matrix least-squares on F^2
Function Minimized	$\sum w (F_o^2 - F_c^2)^2$
Least Squares Weights	$w = 1 / [\sigma^2(F_o^2) + (0.2000 \cdot P)^2 + 0.0000 \cdot P]$ where $P = (\text{Max}(F_o^2, 0) + 2F_c^2)/3$
$2\theta_{\text{max}}$ cutoff	55.0°
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (All reflections)	5699
No. Variables	317
Reflection/Parameter Ratio	17.98
Residuals: R_1 ($I > 2.00\sigma(I)$)	0.1608

Residuals: R (All reflections)	0.1831
Residuals: wR2 (All reflections)	0.4840
Goodness of Fit Indicator	2.001
Max Shift/Error in Final Cycle	0.005
Maximum peak in Final Diff. Map	3.77 e-/Å ³
Minimum peak in Final Diff. Map	-0.82 e-/Å ³

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

atom	x	y	z	B _{eq}
O1	0.2436(4)	0.4866(4)	0.4927(3)	3.75(7)
O2	0.0609(5)	0.6516(5)	0.4354(3)	4.91(9)
O3	0.3015(4)	0.9177(4)	0.5553(3)	4.74(8)
O4	0.3126(4)	0.8182(4)	0.7234(3)	3.84(7)
O5	-0.0798(5)	1.0657(4)	0.7798(3)	5.13(9)
O6	0.1391(4)	1.0677(4)	0.7032(3)	4.39(8)
O7	-0.212(2)	0.634(2)	1.0551(7)	16.5(5)
O8	-0.339(3)	0.448(2)	1.0965(7)	24.6(9)
N1	0.0566(4)	0.7464(4)	0.6004(3)	2.94(7)
N2	-0.1816(4)	0.7500(4)	0.6790(3)	3.27(7)
N3	-0.235(3)	0.540(2)	1.0393(6)	17.8(7)
C1	0.3515(5)	0.4185(5)	0.5614(4)	3.35(8)
C2	0.4449(7)	0.3140(6)	0.5413(5)	4.6(1)
C3	0.5554(7)	0.2445(6)	0.6075(5)	5.0(2)
C4	0.5737(6)	0.2811(6)	0.6918(6)	5.3(2)
C5	0.4773(6)	0.3846(5)	0.7135(4)	4.3(1)
C6	0.3633(5)	0.4565(5)	0.6473(4)	3.13(8)
C7	0.2588(5)	0.5628(4)	0.6629(3)	2.86(8)
C8	0.1571(5)	0.6335(4)	0.5936(3)	2.75(7)
C9	-0.1012(5)	0.7452(5)	0.5885(4)	3.21(8)
C10	-0.1712(5)	0.8861(5)	0.6904(5)	4.0(1)
C11	-0.0145(5)	0.9137(5)	0.6857(4)	3.07(8)

C12	0.0886(5)	0.8396(4)	0.6454(3)	2.72(7)
C13	0.2465(5)	0.8651(5)	0.6344(4)	3.11(8)
C14	0.4626(6)	0.8467(9)	0.7169(7)	7.1(2)
C15	0.0091(5)	1.0220(5)	0.7271(4)	3.49(9)
C16	0.1730(7)	1.1630(6)	0.7517(6)	5.4(2)
C17	-0.1707(5)	0.6292(5)	0.7645(4)	3.40(9)
C18	-0.1422(6)	0.4949(5)	0.7577(4)	4.1(1)
C19	-0.1378(9)	0.3763(7)	0.8415(5)	6.0(2)
C20	-0.161(2)	0.3830(8)	0.9408(6)	8.0(3)
C21	-0.194(2)	0.5141(9)	0.9420(5)	8.4(3)
C22	-0.1958(9)	0.6360(7)	0.8599(5)	6.1(2)
C23	-0.148(2)	0.250(2)	1.0257(8)	12.2(5)
C24	0.1474(5)	0.5960(5)	0.5024(4)	3.30(8)

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

Table 2. Atomic coordinates and B_{iso} involving hydrogen atoms

atom	x	y	z	B _{iso}
H2	0.4335	0.2907	0.4838	5.57
H3	0.6182	0.1724	0.5954	5.98
H4	0.6512	0.2360	0.7346	6.33
H5	0.4883	0.4061	0.7718	5.12
H7	0.2606	0.5841	0.7223	3.43
H9A	-0.1377	0.8252	0.5316	3.85
H9B	-0.1152	0.6611	0.5746	3.85
H10A	-0.2139	0.8879	0.7547	4.77
H10B	-0.2255	0.9596	0.6372	4.77
H14A	0.4650	0.9459	0.6909	8.54
H14B	0.5166	0.8034	0.6726	8.54
H14C	0.5048	0.8096	0.7829	8.54
H16A	0.1714	1.1186	0.8237	6.49
H16B	0.1027	1.2452	0.7334	6.49
H16C	0.2676	1.1888	0.7306	6.49
H18	-0.1257	0.4850	0.6943	4.97
H19	-0.1193	0.2892	0.8328	7.18
H22	-0.2140	0.7225	0.8696	7.35
H23A	-0.2365	0.2436	1.0640	14.63
H23B	-0.0697	0.2476	1.0685	14.63
H23C	-0.1285	0.1734	1.0001	14.63

Table 3. Anisotropic displacement parameters

atom	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
O1	0.054(2)	0.051(2)	0.046(2)	0.000(2)	0.002(2)	-
	0.031(2)					
O2	0.064(3)	0.081(3)	0.045(2)	0.011(2)	-0.014(2)	-
	0.031(2)					
O3	0.054(2)	0.072(3)	0.057(3)	-0.019(2)	0.017(2)	-
	0.024(2)					
O4	0.035(2)	0.052(2)	0.060(2)	-0.000(2)	-0.010(2)	-
	0.021(2)					
O5	0.071(3)	0.055(2)	0.083(3)	-0.019(2)	0.031(2)	-
	0.042(2)					
O6	0.048(2)	0.044(2)	0.083(3)	-0.008(2)	0.007(2)	-
	0.033(2)					
O7	0.34(2)	0.21(1)	0.116(7)	0.00(1)	0.017(7)	-
	0.126(8)					
O8	0.66(4)	0.139(8)	0.087(6)	0.05(2)	0.12(2)	-
	0.030(6)					
N1	0.029(2)	0.040(2)	0.045(2)	0.002(2)	-0.002(2)	-
	0.020(2)					
N2	0.032(2)	0.045(2)	0.049(2)	0.001(2)	0.002(2)	-
	0.021(2)					
N3	0.47(3)	0.126(8)	0.044(4)	0.06(1)	0.026(8)	-
	0.029(5)					

C1	0.041(3)	0.038(2)	0.052(3)	-0.006(2)	0.010(2)	-
	0.021(2)					
C2	0.067(4)	0.046(3)	0.069(4)	-0.006(3)	0.025(3)	-
	0.031(3)					
C3	0.063(4)	0.046(3)	0.072(4)	0.009(3)	0.023(3)	-
	0.018(3)					
C4	0.048(3)	0.045(3)	0.094(5)	0.011(3)	0.000(3)	-
	0.012(3)					
C5	0.046(3)	0.047(3)	0.060(3)	0.003(2)	0.003(3)	-
	0.011(3)					
C6	0.044(3)	0.035(2)	0.039(3)	-0.005(2)	0.006(2)	-
	0.012(2)					
C7	0.041(2)	0.036(2)	0.034(2)	-0.003(2)	0.002(2)	-
	0.016(2)					
C8	0.036(2)	0.035(2)	0.034(2)	-0.004(2)	0.006(2)	-
	0.015(2)					
C9	0.030(2)	0.050(3)	0.042(3)	-0.005(2)	-0.005(2)	-
	0.014(2)					
C10	0.029(2)	0.046(3)	0.078(4)	0.003(2)	0.004(2)	-
	0.028(3)					
C11	0.032(2)	0.033(2)	0.048(3)	-0.000(2)	0.003(2)	-
	0.011(2)					
C12	0.034(2)	0.032(2)	0.035(2)	0.002(2)	-0.002(2)	-
	0.011(2)					

C13	0.035(2)	0.035(2)	0.050(3)	-0.001(2)	0.005(2)	-
	0.018(2)					
C14	0.040(3)	0.112(6)	0.127(7)	-0.003(4)	-0.022(4)	-
	0.050(5)					
C15	0.045(3)	0.032(2)	0.053(3)	0.002(2)	0.010(2)	-
	0.014(2)					
C16	0.067(4)	0.054(3)	0.098(5)	-0.018(3)	0.004(4)	-
	0.038(3)					
C17	0.036(2)	0.049(3)	0.049(3)	-0.002(2)	0.001(2)	-
	0.024(2)					
C18	0.063(3)	0.046(3)	0.051(3)	-0.004(3)	0.008(3)	-
	0.023(3)					
C19	0.104(5)	0.057(4)	0.069(4)	-0.005(4)	0.007(4)	-
	0.028(3)					
C20	0.152(8)	0.068(5)	0.063(5)	0.003(5)	0.025(5)	-
	0.003(4)					
C21	0.18(1)	0.094(6)	0.048(4)	-0.014(6)	0.017(5)	-
	0.034(4)					
C22	0.126(6)	0.055(4)	0.051(3)	0.018(4)	-0.001(4)	-
	0.027(3)					
C23	0.24(2)	0.098(7)	0.087(7)	-0.003(8)	0.014(8)	
	0.010(6)					
C24	0.046(3)	0.047(3)	0.035(3)	-0.004(2)	0.000(2)	-
	0.018(2)					

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

Table 4. Bond lengths (Å)

atom	atom	distance	atom	atom
		distance		
O1	C1	1.376(6)	O1	C24
		1.370(6)		
O2	C24	1.207(6)	O3	C13
		1.184(6)		
O4	C13	1.333(6)	O4	C14
		1.461(7)		
O5	C15	1.216(7)	O6	C15
		1.346(6)		
O6	C16	1.437(9)	O7	N3
		1.11(2)		
O8	N3	1.47(3)	N1	C8
		1.405(6)		
N1	C9	1.493(6)	N1	C12
		1.378(7)		
N2	C9	1.450(6)	N2	C10
		1.470(8)		
N2	C17	1.403(5)	N3	C21
		1.495(14)		
C1	C2	1.373(8)	C1	C6
		1.399(8)		

C2	C3	1.376(8)	C3	C4
		1.380(11)		
C4	C5	1.390(8)	C5	C6
		1.403(7)		
C6	C7	1.419(6)	C7	C8
		1.345(6)		
C8	C24	1.464(7)	C10	C11
		1.521(7)		
C11	C12	1.355(6)	C11	C15
		1.457(8)		
C12	C13	1.522(6)	C17	C18
		1.395(8)		
C17	C22	1.369(9)	C18	C19
		1.378(8)		
C19	C20	1.422(11)	C20	C21
		1.337(13)		
C20	C23	1.473(12)	C21	C22
		1.387(9)		

Table 5. Bond lengths involving hydrogens (Å)

atom	atom	distance	atom	atom	
		distance			
C2	H2	0.930	C3	H3	0.930
C4	H4	0.930	C5	H5	0.930
C7	H7	0.930	C9	H9A	0.970
C9	H9B	0.970	C10	H10A	0.970
C10	H10B	0.970	C14	H14A	0.960
C14	H14B	0.960	C14	H14C	0.960
C16	H16A	0.960	C16	H16B	0.960
C16	H16C	0.960	C18	H18	0.930
C19	H19	0.930	C22	H22	0.930
C23	H23A	0.960	C23	H23B	0.960
C23	H23C	0.960			

Table 6. Bond angles ($^{\circ}$)

atom	atom	atom	angle	atom	atom	atom	angle
C1	O1	C24	122.6(4)	C13	O4	C14	
			113.3(5)				
C15	O6	C16	115.7(5)	C8	N1	C9	
			119.9(4)				
C8	N1	C12	122.6(4)	C9	N1	C12	
			114.9(4)				
C9	N2	C10	107.5(4)	C9	N2	C17	
			118.6(4)				
C10	N2	C17	119.5(5)	O7	N3	O8	
			124.1(13)				
O7	N3	C21	123.2(13)	O8	N3	C21	
			110.8(12)				
O1	C1	C2	117.2(5)	O1	C1	C6	
			120.2(4)				
C2	C1	C6	122.7(5)	C1	C2	C3	
			119.0(6)				
C2	C3	C4	120.4(6)	C3	C4	C5	
			120.6(6)				
C4	C5	C6	119.9(6)	C1	C6	C5	
			117.3(5)				
C1	C6	C7	118.3(4)	C5	C6	C7	
			124.3(5)				

C6	C7	C8	121.4(5)	N1	C8	C7
	124.6(5)					
N1	C8	C24	115.3(4)	C7	C8	C24
	120.0(4)					
N1	C9	N2	111.1(4)	N2	C10	C11
	111.4(4)					
C10	C11	C12	119.8(5)	C10	C11	C15
	114.3(4)					
C12	C11	C15	125.9(4)	N1	C12	C11
	122.6(4)					
N1	C12	C13	114.1(4)	C11	C12	C13
	122.8(5)					
O3	C13	O4	125.6(5)	O3	C13	C12
	122.9(5)					
O4	C13	C12	111.5(4)	O5	C15	O6
	123.0(5)					
O5	C15	C11	123.0(5)	O6	C15	C11
	114.0(4)					
N2	C17	C18	122.5(5)	N2	C17	C22
	121.7(5)					
C18	C17	C22	115.8(5)	C17	C18	C19
	122.3(6)					
C18	C19	C20	122.1(7)	C19	C20	C21
	112.7(6)					

C19	C20	C23	117.9(8)	C21	C20	C23
	129.4(9)					
N3	C21	C20	120.0(7)	N3	C21	C22
	113.2(9)					
C20	C21	C22	126.8(8)	C17	C22	C21
	120.1(7)					
O1	C24	O2	116.9(5)	O1	C24	C8
	117.2(4)					
O2	C24	C8	125.9(5)			

Table 7. Bond angles involving hydrogens (°)

atom	atom	atom	angle	atom	atom	atom	angle
C1	C2	H2	120.5	C3	C2	H2	120.5
C2	C3	H3	119.8	C4	C3	H3	119.8
C3	C4	H4	119.7	C5	C4	H4	119.7
C4	C5	H5	120.0	C6	C5	H5	120.0
C6	C7	H7	119.3	C8	C7	H7	119.3
N1	C9	H9A	109.4	N1	C9	H9B	109.4
N2	C9	H9A	109.4	N2	C9	H9B	109.4
H9A	C9	H9B	108.0	N2	C10	H10A	109.4
N2	C10	H10B	109.4	C11	C10	H10A	109.4
C11	C10	H10B	109.4	H10A	C10	H10B	108.0
O4	C14	H14A	109.5	O4	C14	H14B	109.5
O4	C14	H14C	109.5	H14A	C14	H14B	109.5
H14A	C14	H14C	109.5	H14B	C14	H14C	109.5
O6	C16	H16A	109.5	O6	C16	H16B	109.5
O6	C16	H16C	109.5	H16A	C16	H16B	109.5
H16A	C16	H16C	109.5	H16B	C16	H16C	109.5
C17	C18	H18	118.8	C19	C18	H18	118.8
C18	C19	H19	119.0	C20	C19	H19	119.0
C17	C22	H22	119.9	C21	C22	H22	120.0
C20	C23	H23A	109.5	C20	C23	H23B	109.5
C20	C23	H23C	109.5	H23A	C23	H23B	109.5
H23A	C23	H23C	109.5	H23B	C23	H23C	109.5

Table 8. Torsion Angles(°)

(Those having bond angles > 160 or < 20 degrees are excluded.)

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	
				angle					
C1	O1	C24	O2	177.0(4)	C1	O1	C24	C8	-
				3.9(6)					
C24	O1	C1	C2	-177.5(4)	C24	O1	C1	C6	2.4(6)
C14	O4	C13	O3	4.4(8)	C14	O4	C13	C12	-
				177.2(5)					
C16	O6	C15	O5	5.6(6)	C16	O6	C15	C11	-
				173.3(4)					
C8	N1	C9	N2	119.2(4)	C9	N1	C8	C7	-
				128.1(4)					
C9	N1	C8	C24	53.0(5)	C8	N1	C12	C11	-
				155.0(3)					
C8	N1	C12	C13	32.2(5)	C12	N1	C8	C7	
				32.8(6)					
C12	N1	C8	C24	-146.1(4)	C9	N1	C12	C11	6.8(5)
C9	N1	C12	C13	-166.0(3)	C12	N1	C9	N2	-
				43.1(5)					
C9	N2	C10	C11	-52.1(5)	C10	N2	C9	N1	
				65.6(4)					
C9	N2	C17	C18	-28.4(6)	C9	N2	C17	C22	
				155.5(4)					

C17	N2	C9	N1	-73.9(5)	C10	N2	C17	C18	-
163.1(4)									
C10	N2	C17	C22	20.9(6)	C17	N2	C10	C11	
87.0(5)									
O7	N3	C21	C20	149.0(16)	O7	N3	C21	C22	-31(3)
O8	N3	C21	C20	-46.4(18)	O8	N3	C21	C22	
134.0(11)									
O1	C1	C2	C3	179.2(4)	O1	C1	C6	C5	-
178.9(4)									
O1	C1	C6	C7	2.1(6)	C2	C1	C6	C5	0.9(7)
C2	C1	C6	C7	-178.1(4)	C6	C1	C2	C3	-
0.6(8)									
C1	C2	C3	C4	-1.2(8)	C2	C3	C4	C5	2.7(8)
C3	C4	C5	C6	-2.4(8)	C4	C5	C6	C1	0.6(7)
C4	C5	C6	C7	179.5(4)	C1	C6	C7	C8	-
5.0(6)									
C5	C6	C7	C8	176.1(4)	C6	C7	C8	N1	-
175.3(4)									
C6	C7	C8	C24	3.5(6)	N1	C8	C24	O1	
179.9(4)									
N1	C8	C24	O2	-1.1(7)	C7	C8	C24	O1	0.9(6)
C7	C8	C24	O2	179.9(4)	N2	C10	C11	C12	
18.2(6)									
N2	C10	C11	C15	-161.7(4)	C10	C11	C12	N1	5.3(6)

C10	C11	C12	C13	177.6(3)	C10	C11	C15	O5	
				15.0(6)					
C10	C11	C15	O6	-166.2(4)	C12	C11	C15	O5	-
				165.0(4)					
C12	C11	C15	O6	13.9(6)	C15	C11	C12	N1	-
				174.7(4)					
C15	C11	C12	C13	-2.5(6)	N1	C12	C13	O3	
				64.1(5)					
N1	C12	C13	O4	-114.3(4)	C11	C12	C13	O3	-
				108.7(6)					
C11	C12	C13	O4	72.8(5)	N2	C17	C18	C19	-
				177.1(4)					
N2	C17	C22	C21	175.3(5)	C18	C17	C22	C21	-
				1.0(9)					
C22	C17	C18	C19	-0.8(8)	C17	C18	C19	C20	-
				0.6(10)					
C18	C19	C20	C21	3.7(13)	C18	C19	C20	C23	-
				177.5(7)					
C19	C20	C21	N3	174.7(8)	C19	C20	C21	C22	-
				5.9(17)					
C23	C20	C21	N3	-3.9(19)	C23	C20	C21	C22	
				175.5(12)					

Table 8. Torsion angles ($^{\circ}$) (continued)

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
N3	C21	C22	C17	-175.7(11)	C20	C21	C22	C17	
				4.8(17)					

Table 9. Intramolecular contacts less than 3.60 Å

atom	atom	distance	atom	atom
	distance			
O1	C7	2.764(6)	O2	N1
	2.789(7)			
O2	C1	3.473(6)	O2	C7
	3.562(6)			
O2	C9	2.900(7)	O3	O6
	3.172(6)			
O3	N1	2.987(6)	O3	C7
	3.507(6)			
O3	C8	3.246(7)	O3	C11
	3.379(6)			
O3	C14	2.628(9)	O4	O6
	2.758(5)			
O4	N1	3.320(6)	O4	C7
	3.112(7)			
O4	C8	3.514(7)	O4	C11
	3.088(5)			
O4	C15	3.276(6)	O5	C10
	2.787(8)			
O5	C12	3.592(7)	O5	C16
	2.647(8)			

O6	C12	2.817(7)	O6	C13
		2.619(7)		
O6	C14	3.477(8)	O7	C20
		3.422(17)		
O7	C22	2.731(12)	O8	C20
		2.869(17)		
O8	C22	3.534(13)	O8	C23
		2.89(2)		
N1	C10	2.862(7)	N1	C17
		3.094(6)		
N1	C18	3.471(6)	N2	C8
		3.505(6)		
N2	C12	2.767(6)	N3	C23
		3.025(18)		
C1	C4	2.740(8)	C1	C8
		2.787(7)		
C2	C5	2.786(10)	C3	C6
		2.794(8)		
C6	C24	2.819(6)	C7	C12
		2.982(6)		
C7	C13	2.968(7)	C8	C13
		2.854(7)		
C9	C11	2.750(8)	C9	C18
		2.910(6)		

C9	C24	3.011(7)	C10	C22
		2.883(7)		
C11	C17	3.270(7)	C12	C17
		3.451(6)		
C13	C15	3.037(7)	C17	C20
		2.879(8)		
C18	C21	2.671(10)	C19	C22
		2.727(10)		

Table 10. Intramolecular contacts less than 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
O1	H2	2.510	O2	H9A	2.963
O2	H9B	2.499	O3	H7	3.497
O3	H14A	2.617	O3	H14B	2.555
O3	H14C	3.572	O4	H7	2.514
O5	H10A	2.472	O5	H10B	3.033
O5	H16A	2.628	O5	H16B	2.581
O5	H16C	3.591	O6	H14A	3.135
O7	H22	2.456	O8	H23A	2.349
O8	H23B	3.105	N1	H7	2.623
N1	H10B	3.303	N1	H18	3.236

N2	H18	2.623	N2	H22	2.601
N3	H22	2.507	N3	H23A	2.938
N3	H23B	3.072	C1	H3	3.209
C1	H5	3.240	C1	H7	3.256
C2	H4	3.227	C3	H5	3.243
C4	H2	3.233	C5	H3	3.243
C5	H7	2.689	C6	H2	3.268
C6	H4	3.256	C7	H5	2.693
C8	H9A	3.124	C8	H9B	2.534
C8	H18	3.249	C9	H10A	3.198
C9	H10B	2.608	C9	H18	2.616
C10	H9A	2.500	C10	H9B	3.203

C10	H22	2.562	C11	H9A	2.949
C11	H22	3.330	C12	H7	2.773
C12	H9A	2.767	C12	H9B	3.215
C12	H10A	3.191	C12	H10B	3.005
C13	H7	2.711	C13	H14A	2.561
C13	H14B	2.552	C13	H14C	3.140
C14	H7	3.472	C15	H10A	2.593
C15	H10B	2.834	C15	H16A	2.579
C15	H16B	2.586	C15	H16C	3.156
C16	H14A	3.514	C17	H9A	3.242
C17	H9B	2.606	C17	H10A	2.577
C17	H10B	3.235	C17	H19	3.255

C18	H9B	2.588	C18	H22	3.194
C19	H23A	3.108	C19	H23B	3.080
C19	H23C	2.483	C20	H18	3.278

Table 10. Intramolecular contacts less than 3.60 Å involving hydrogens
(continued)

atom	atom	distance	atom	atom	distance
C20	H22	3.248	C21	H19	3.145
C21	H23A	2.797	C21	H23B	2.836
C21	H23C	3.276	C22	H10A	2.494
C22	H18	3.184	C23	H19	2.607
C24	H7	3.276	C24	H9A	3.387
C24	H9B	2.678	H2	H3	2.310
H3	H4	2.303	H4	H5	2.317
H5	H7	2.556	H7	H14B	3.393
H9A	H10A	3.418	H9A	H10B	2.378
H9A	H18	3.459	H9B	H10B	3.454

H9B	H18	2.023	H10A	H22	1.911
H10B	H22	3.353	H14A	H16C	3.061
H18	H19	2.277	H19	H23A	3.269
H19	H23B	3.243	H19	H23C	2.253

Table 11. Intermolecular contacts less than 3.60 Å

atom	atom	distance	atom	atom
	distance			
O1	O2 ¹	3.310(6)	O1	C3 ²
	3.413(7)			
O1	C4 ²	3.439(7)	O1	C9 ¹
	3.398(7)			
O2	O1 ¹	3.310(6)	O2	O2 ¹
	3.351(6)			
O2	O5 ³	3.434(5)	O2	O6 ³
	3.267(5)			
O2	C15 ³	3.354(6)	O2	C16 ³
	3.378(7)			
O2	C18 ¹	3.508(8)	O2	C24 ¹
	3.269(7)			
O3	C3 ²	3.361(8)	O3	C10 ³
	3.547(7)			
O4	O8 ⁴	3.029(10)	O5	O2 ³
	3.434(5)			
O5	C19 ⁵	3.524(9)	O6	O2 ³
	3.267(5)			
O7	C20 ⁴	3.458(18)	O8	O4 ⁴
	3.029(10)			

O8	C7 ⁴	3.462(13)	O8	C14 ⁴
	3.562(15)			
N2	C14 ⁶	3.390(7)	C1	C1 ²
	3.505(7)			
C1	C2 ²	3.422(8)	C1	C16 ⁷
	3.582(8)			
C2	C1 ²	3.422(8)	C2	C6 ²
	3.500(7)			
C3	O1 ²	3.413(7)	C3	O3 ²
	3.361(8)			
C3	C24 ²	3.444(8)	C4	O1 ²
	3.439(7)			
C6	C2 ²	3.500(7)	C6	C16 ⁷
	3.550(8)			
C7	O8 ⁴	3.462(13)	C9	O1 ¹
	3.398(7)			
C10	O3 ³	3.547(7)	C10	C14 ⁶
	3.481(8)			
C14	O8 ⁴	3.562(15)	C14	N2 ⁸
	3.390(7)			
C14	C10 ⁸	3.481(8)	C15	O2 ³
	3.354(6)			
C16	O2 ³	3.378(7)	C16	C1 ⁵
	3.582(8)			

C16	C6 ⁵	3.550(8)	C18	O2 ¹
	3.508(8)			
C19	O5 ⁷	3.524(9)	C20	O7 ⁴
	3.458(18)			
C24	O2 ¹	3.269(7)	C24	C3 ²
	3.444(8)			

Symmetry Operators:

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

Table 12. Intermolecular contacts less than 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	
	distance				
O1	H9A ¹	3.593	O1	H9B ¹	2.490
O1	H18 ¹	2.813	O2	H4 ²	3.544
O2	H9B ¹	3.214	O2	H16B ³	2.710
O2	H18 ¹	2.638	O3	H2 ²	3.174
O3	H3 ²	2.608	O3	H9A ³	2.769
O3	H10B ³	2.662	O4	H23A ⁴	2.905
O4	H23B ⁴	3.536	O5	H4 ⁵	2.817
O5	H19 ⁶	2.602	O5	H23B ⁴	3.356
O5	H23C ⁶	3.598	O6	H9A ³	3.108
O7	H5 ⁴	3.413	O7	H7 ⁴	3.233

O7	H16A ⁷	3.551	O7	H23B ⁴	3.220
O8	H5 ⁴	2.919	O8	H7 ⁴	2.588
O8	H14C ⁴	3.179	N2	H2 ¹	3.525
N2	H14B ⁸	2.790	N2	H14C ⁸	3.265
N3	H5 ⁴	3.581	N3	H7 ⁴	3.169
C1	H9B ¹	3.345	C1	H16B ⁹	3.471
C1	H16C ⁹	2.894	C2	H9B ¹	3.497
C2	H14B ²	3.568	C2	H16C ⁹	3.046
C3	H10B ¹⁰	3.229	C3	H14A ⁹	3.106
C3	H16C ⁹	3.126	C4	H16C ⁹	3.096
C4	H19 ¹¹	3.597	C5	H16C ⁹	2.938
C6	H2 ²	3.391	C6	H16B ⁹	3.386

C6	H16C ⁹	2.847	C7	H16B ⁹	3.562
C8	H3 ²	3.525	C9	H2 ¹	3.420
C10	H3 ⁵	3.205	C10	H14A ⁸	3.362
C10	H14B ⁸	3.191	C10	H14C ⁸	3.315
C11	H23B ⁴	3.376	C12	H9A ³	3.576
C13	H3 ²	3.517	C13	H9A ³	3.306
C14	H2 ²	3.575	C14	H10A ¹¹	3.203
C14	H10B ¹¹	3.283	C14	H23A ⁴	3.563
C15	H19 ⁶	3.558	C15	H23B ⁴	3.261
C16	H19 ⁶	3.165	C17	H14B ⁸	3.281
C17	H14C ⁸	3.358	C18	H4 ⁸	3.594
C18	H16B ⁹	3.269	C19	H4 ⁸	3.279

C19	H16B ⁹	3.041	C22	H14C ⁸	3.126
C22	H23B ⁴	3.237	C24	H3 ²	3.353
C24	H9B ¹	3.213	C24	H18 ¹	3.160

Table 12. Intermolecular contacts less than 3.60 Å involving hydrogens
(continued)

atom	atom	distance	atom	atom	
	distance				
H2	O3 ²	3.174	H2	N2 ¹	3.525
H2	C6 ²	3.391	H2	C9 ¹	3.420
H2	C14 ²	3.575	H2	H9A ¹	3.198
H2	H9B ¹	3.053	H2	H14B ²	2.668
H2	H16C ⁹	3.597	H3	O3 ²	2.608
H3	C8 ²	3.525	H3	C10 ¹⁰	3.205
H3	C13 ²	3.517	H3	C24 ²	3.353
H3	H10A ¹⁰	3.269	H3	H10B ¹⁰	2.355
H3	H14A ⁹	2.805	H4	O2 ²	3.544
H4	O5 ¹⁰	2.817	H4	C18 ¹¹	3.594

H4	C19 ¹¹	3.279	H4	H10A ¹⁰	3.525
H4	H10B ¹⁰	3.556	H4	H18 ¹¹	3.398
H4	H19 ¹¹	2.818	H5	O7 ⁴	3.413
H5	O8 ⁴	2.919	H5	N3 ⁴	3.581
H5	H16C ⁹	3.435	H7	O7 ⁴	3.233
H7	O8 ⁴	2.588	H7	N3 ⁴	3.169
H9A	O1 ¹	3.593	H9A	O3 ³	2.769
H9A	O6 ³	3.108	H9A	C12 ³	3.576
H9A	C13 ³	3.306	H9A	H2 ¹	3.198
H9B	O1 ¹	2.490	H9B	O2 ¹	3.214
H9B	C1 ¹	3.345	H9B	C2 ¹	3.497
H9B	C24 ¹	3.213	H9B	H2 ¹	3.053

H10A	C14 ⁸	3.203	H10A	H3 ⁵	3.269
H10A	H4 ⁵	3.525	H10A	H14A ⁸	3.094
H10A	H14B ⁸	3.162	H10A	H14C ⁸	2.821
H10A	H23B ⁴	3.534	H10B	O3 ³	2.662
H10B	C3 ⁵	3.229	H10B	C14 ⁸	3.283
H10B	H3 ⁵	2.355	H10B	H4 ⁵	3.556
H10B	H14A ⁸	2.954	H10B	H14B ⁸	3.003
H10B	H14C ⁸	3.362	H14A	C3 ⁶	3.106
H14A	C10 ¹¹	3.362	H14A	H3 ⁶	2.805
H14A	H10A ¹¹	3.094	H14A	H10B ¹¹	2.954
H14B	N2 ¹¹	2.790	H14B	C2 ²	3.568
H14B	C10 ¹¹	3.191	H14B	C17 ¹¹	3.281

H14B	H2 ²	2.668	H14B	H10A ¹¹	3.162
H14B	H10B ¹¹	3.003	H14C	O8 ⁴	3.179
H14C	N2 ¹¹	3.265	H14C	C10 ¹¹	3.315

Table 12. Intermolecular contacts less than 3.60 Å involving hydrogens
(continued)

atom	atom	distance	atom	atom	
	distance				
H14C	C17 ¹¹	3.358	H14C	C22 ¹¹	3.126
H14C	H10A ¹¹	2.821	H14C	H10B ¹¹	3.362
H14C	H22 ¹¹	2.852	H14C	H23A ⁴	3.190
H16A	O7 ⁷	3.551	H16A	H19 ⁶	3.029
H16A	H23A ⁴	3.486	H16A	H23C ⁴	3.243
H16B	O2 ³	2.710	H16B	C1 ⁶	3.471
H16B	C6 ⁶	3.386	H16B	C7 ⁶	3.562
H16B	C18 ⁶	3.269	H16B	C19 ⁶	3.041
H16B	H18 ⁶	2.933	H16B	H19 ⁶	2.503
H16C	C1 ⁶	2.894	H16C	C2 ⁶	3.046

H16C	C3 ⁶	3.126	H16C	C4 ⁶	3.096
H16C	C5 ⁶	2.938	H16C	C6 ⁶	2.847
H16C	H2 ⁶	3.597	H16C	H5 ⁶	3.435
H18	O1 ¹	2.813	H18	O2 ¹	2.638
H18	C24 ¹	3.160	H18	H4 ⁸	3.398
H18	H16B ⁹	2.933	H19	O5 ⁹	2.602
H19	C4 ⁸	3.597	H19	C15 ⁹	3.558
H19	C16 ⁹	3.165	H19	H4 ⁸	2.818
H19	H16A ⁹	3.029	H19	H16B ⁹	2.503
H22	H14C ⁸	2.852	H22	H23B ⁴	2.918
H23A	O4 ⁴	2.905	H23A	C14 ⁴	3.563
H23A	H14C ⁴	3.190	H23A	H16A ⁴	3.486

H23B	O4 ⁴	3.536	H23B	O5 ⁴	3.356
H23B	O7 ⁴	3.220	H23B	C11 ⁴	3.376
H23B	C15 ⁴	3.261	H23B	C22 ⁴	3.237
H23B	H10A ⁴	3.534	H23B	H22 ⁴	2.918
H23C	O5 ⁹	3.598	H23C	H16A ⁴	3.243

Symmetry Operators:

(1) -X,-Y+1,-Z+1	(2) -X+1,-Y+1,-Z+1
(3) -X,-Y+2,-Z+1	(4) -X,-Y+1,-Z+2
(5) X-1,Y+1,Z	(6) X,Y+1,Z
(7) -X,-Y+2,-Z+2	(8) X-1,Y,Z
(9) X,Y-1,Z	(10) X+1,Y-1,Z
(11) X+1,Y,Z	