Supporting Information

PhI(OAc)₂ Mediated a Novel 1,3-Dipolar Cycloaddition of Methylenecyclopropanes (MCPs), Vinylenecyclopropanes (VCPs) and Methylenecyclobutane (MCB) with Phthalhydrazide

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Experimental Procedures.

General Methods. Melting points are uncorrected. ^1H and ^{13}C NMR spectra were recorded at 300 and 75 MHz respectively using tetramethylsilane as the internal standard. Mass spectra were recorded by EI, MALDI and ESI methods, and HRMS was measured on a Finnigan MA^+ mass spectrometer. Organic solvents used were dried by standard methods when necessary. Commercially obtained reagents were used without further purification. All of these reactions were monitored by TLC with Huanghai GF254 silica gel coated plates. Flash column chromatography was carried out using 300-400 mesh silica gel or 200-300 mesh Al_2O_3 at increased pressure.

Typical reaction procedure for the iodobenzene diacetate mediated 1,3-dipolar cycloaddition of methylenecyclopropanes (MCPs), vinylenecyclopropanes (VCPs) and a methylenecyclobutane (MCB) with phthalhydrazide. To a solution of MCP **1a** (62 mg, 0.3 mmol) in CH_2Cl_2 (2.0 mL) was added iodobenzene diacetate (116 mg, 0.36 mmol) and phthalhydrazide (58 mg, 0.36 mmol), the mixture was stirred for 3 hours at room temperature (monitored by TLC), then the solvent was removed under reduced pressure and the residue was subjected to a short path flash column chromatography (eluent: acetone) to give product **4a** (97 mg, 88%) as a pale yellow solid.

The procedure for the transformation of compound 4a to 7a. Compound **4a** (110 mg, 0.3 mmol) was heated to $180\text{ }^\circ\text{C}$ in neat for 5 minutes and then the mixture was subjected to a flash column chromatography (eluent: ethyl acetate/petroleum ether = 1/2) to give product **7a** (68 mg, 62%) as a white solid.

Compound **4a**: a pale yellow solid; m.p. 176 °C (decompose); IR (KBr) ν 3058, 1605, 1561, 1513, 1271, 1180, 854, 770, 700 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz, TMS) δ 0.87 (2H, dd, $J = 6.6, 8.1$ Hz, CH_2), 2.08 (2H, dd, $J = 6.6, 8.1$ Hz, CH_2), 7.43 (10H, s, Ar), 7.73-7.78 (1H, m, Ar), 7.88-7.94 (2H, m, Ar), 8.56 (1H, d, $J = 8.1$ Hz, Ar); ^{13}C NMR (CDCl_3 , 75 MHz, TMS) δ 10.3, 54.6, 95.6, 118.7, 123.1, 127.4, 128.4, 128.8, 129.6, 131.7, 134.6, 137.8, 148.3, 167.3; MS (ESI) m/z 367.1 ($\text{M}+\text{H}^+$); HRMS (MALDI) Calcd. for $\text{C}_{24}\text{H}_{19}\text{N}_2\text{O}_2^+$ ($\text{M}+\text{H}^+$) requires 367.1441, Found: 367.1452.

Compound **4b**: a pale yellow solid; m.p. 168 °C (decompose); IR (KBr) ν 3033, 1607, 1563, 1514, 1427, 1177, 812, 735, 685 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz, TMS) δ 0.85 (2H, dd, $J = 6.3, 8.1$ Hz, CH_2), 2.04 (2H, dd, $J = 6.3, 8.1$ Hz, CH_2), 2.38 (6H, s, 2CH_3), 7.21 (4H, d, $J = 8.1$ Hz, Ar), 7.31 (4H, d, $J = 8.1$ Hz, Ar), 7.71-7.77 (1H, m, Ar), 7.86-7.93 (2H, m, Ar), 8.55 (1H, d, $J = 8.4$ Hz, Ar); ^{13}C NMR (CDCl_3 , 75 MHz, TMS) δ 10.2, 21.1, 54.6, 95.8, 118.7, 123.1, 127.3, 128.3, 129.3, 131.0, 131.5, 134.5, 134.9, 139.5, 148.3, 167.3; MS (ESI) m/z 395.2 ($\text{M}+\text{H}^+$); HRMS (MALDI) Calcd. for $\text{C}_{26}\text{H}_{23}\text{N}_2\text{O}_2^+$ ($\text{M}+\text{H}^+$) requires 395.1754, Found: 395.1766.

Compound **4c**: a pale yellow solid; m.p. 164 °C (decompose); IR (KBr) ν 3058, 1607, 1561, 1515, 1427, 1180, 855, 768, 700 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz, TMS) δ 0.85 (2H, dd, $J = 6.0, 8.1$ Hz, CH_2), 2.04 (2H, dd, $J = 6.0, 8.1$ Hz, CH_2), 3.83 (6H, s, 2CH_3), 6.91-6.94 (4H, m, Ar), 7.33-7.39 (4H, m, Ar), 7.72-7.77 (1H, m, Ar), 7.86-7.92 (2H, m, Ar), 8.55 (1H, d, $J = 7.8$ Hz, Ar); ^{13}C NMR (CDCl_3 , 75 MHz, TMS) δ 10.1, 54.7, 55.2, 95.9, 113.8, 118.7, 123.1, 128.1, 128.9, 129.8, 130.9, 131.5, 134.4, 148.2, 160.1, 167.1; MS (ESI) m/z 427.2 ($\text{M}+\text{H}^+$); HRMS (MALDI) Calcd. for $\text{C}_{26}\text{H}_{23}\text{N}_2\text{O}_4^+$ ($\text{M}+\text{H}^+$) requires 427.1652, Found: 427.1666.

Compound **4d**: a pale yellow solid; m.p. 184 °C (decompose); IR (KBr) ν 3048, 1608, 1564, 1514, 1427, 1177, 1094, 822, 735, 685 cm^{-1} ; ^1H NMR (CDCl₃, 300 MHz, TMS) δ 0.83-0.88 (2H, m, CH₂), 2.08-2.13 (2H, m, CH₂), 7.35-7.44 (8H, m, Ar), 7.75-7.80 (1H, m, Ar), 7.89-7.95 (2H, m, Ar), 8.57 (1H, d, J = 8.1 Hz, Ar); ^{13}C NMR (CDCl₃, 75 MHz, TMS) δ 10.1, 54.3, 94.2, 118.2, 122.9, 128.1, 128.5, 129.0, 130.8, 131.7, 134.6, 135.7, 135.8, 147.7, 167.1; MS (ESI) m/z 435.2 ($\text{M}+\text{H}^+$); HRMS (MALDI) Calcd. for C₂₄H₁₆N₂O₂Cl₂Na⁺ ($\text{M}+\text{Na}^+$) requires 457.0481, Found: 457.0478.

Compound **4e**: a pale yellow solid; m.p. 170 °C (decompose); IR (KBr) ν 3048, 1608, 1564, 1514, 1427, 1177, 1094, 822, 735, 685 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz, TMS) δ 0.88 (2H, dd, $J = 6.3, 8.1$ Hz, CH_2), 2.10 (2H, dd, $J = 6.3, 8.1$ Hz, CH_2), 7.11-7.18 (4H, m, Ar), 7.42-7.48 (4H, m, Ar), 7.76-7.81 (1H, m, Ar), 7.89-7.94 (2H, m, Ar), 8.53-8.56 (1H, m, Ar); ^{13}C NMR (CDCl_3 , 75 MHz, TMS) δ 10.1, 54.6, 94.5, 115.8 (d, $J_{\text{C-F}} = 21.5$ Hz), 118.4, 122.9, 128.2, 129.3 (d, $J_{\text{C-F}} = 8.3$ Hz), 130.9, 131.7, 133.4 (d, $J_{\text{C-F}} = 3.4$ Hz), 134.6, 147.7, 163.0 (d, $J_{\text{C-F}} = 249.3$ Hz), 167.2; MS (ESI) m/z 403.2 ($\text{M}+\text{H}^+$); HRMS (MALDI) Calcd. for $\text{C}_{24}\text{H}_{17}\text{N}_2\text{O}_2\text{F}_2^+$ ($\text{M}+\text{H}^+$) requires 403.1253, Found: 403.1246.

Compound **4f**: a pale yellow solid; m.p. 148 °C (decompose); IR (KBr) ν 3056, 1607, 1564, 1514, 1427, 1176, 1033, 829, 734, 687 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz, TMS) δ 0.69-0.77 (1H, m, CH_2), 0.95-1.04 (1H, m, CH_2), 1.86-1.94 (1H, m, CH_2), 2.18-2.26 (1H, m, CH_2), 3.82 (3H, s, CH_3), 6.91 (2H, d, $J = 9.0$ Hz, Ar), 7.38 (2H, d, $J = 9.0$ Hz, Ar), 7.43 (5H, s, Ar), 7.72-7.77 (1H, m, Ar), 7.86-7.93 (2H, m, Ar), 8.56 (1H, d, $J = 7.8$ Hz, Ar); ^{13}C NMR (CDCl_3 , 75 MHz, TMS) δ 9.6, 10.6, 54.5, 55.1, 95.6, 113.8, 118.6, 123.0, 126.9, 128.1, 128.5, 129.1, 129.2, 129.8, 130.8, 131.5, 134.3, 137.6, 148.0, 160.2, 167.1; MS (ESI) m/z 397.2 ($\text{M}+\text{H}^+$); HRMS (MALDI) Calcd. for $\text{C}_{25}\text{H}_{21}\text{N}_2\text{O}_3^+$ ($\text{M}+\text{H}^+$) requires 397.1547, Found: 397.1560.

Compound **4g**: a pale yellow solid; m.p. 152 °C (decompose); IR (KBr) ν 3057, 1606, 1563, 1514, 1428, 1270, 1093, 824, 735, 685 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz, TMS) δ 0.77-0.85 (1H, m, CH_2), 0.89-0.97 (1H, m, CH_2), 1.96-2.05 (1H, m, CH_2), 2.11-2.19 (1H, m, CH_2), 7.40-7.46 (9H, m, Ar), 7.71-7.76 (1H, m, Ar), 7.87-7.91 (2H, m, Ar), 8.55 (1H, d, $J = 7.8$ Hz, Ar); ^{13}C NMR (CDCl_3 , 75 MHz, TMS) δ 9.9, 10.4, 53.3, 94.9, 118.4, 122.9, 127.0, 128.3, 128.8, 128.86, 128.93, 129.6, 130.8, 131.7, 134.6, 135.7, 136.3, 137.1, 148.0, 167.2; MS (ESI) m/z 401.2 ($\text{M}+\text{H}^+$); HRMS (MALDI) Calcd. for $\text{C}_{24}\text{H}_{18}\text{N}_2\text{O}_2\text{Cl}^+$ ($\text{M}+\text{H}^+$) requires 401.1051, Found: 401.1071.

Compound **4h**: a pale yellow solid; m.p. 180 °C (decompose); IR (KBr) ν 3047, 1605, 1560, 1519, 1432, 1185, 1086, 734, 688 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz, TMS) δ 0.66-0.74 (1H, m, CH_2), 1.05-1.13 (1H, m, CH_2), 1.71-1.80 (1H, m, CH_2), 2.03-2.12 (1H, m, CH_2), 6.25 (1H, s, CH), 7.41-7.47 (5H, m, Ar), 7.75-7.80 (1H, m, Ar), 7.88-7.93 (2H, m, Ar), 8.55 (1H, d, $J = 7.5$ Hz, Ar); ^{13}C NMR (CDCl_3 , 75 MHz, TMS) δ 10.1, 10.3, 52.2, 86.1, 117.8, 122.8, 127.2, 127.8, 128.8, 130.0, 130.5, 131.5, 133.3, 134.3, 149.1, 166.8; MS (ESI) m/z 291.2 ($\text{M} + \text{H}^+$); HRMS (MALDI) Calcd. for $\text{C}_{18}\text{H}_{15}\text{N}_2\text{O}_2^+$ ($\text{M} + \text{H}^+$) requires 291.1128, Found: 291.1128.

Compound **4i**: a pale yellow solid; m.p. 192 °C (decompose); IR (KBr) ν 3047, 1606, 1561,

1518, 1432, 1186, 1086, 773, 734, 687 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz, TMS) δ 0.69-0.77 (1H, m, CH_2), 1.00-1.09 (1H, m, CH_2), 1.81-1.90 (1H, m, CH_2), 2.07-2.16 (1H, m, CH_2), 2.37 (3H, s, CH_3), 6.43 (1H, s, CH), 7.27-7.41 (4H, m, Ar), 7.76-7.81 (1H, m, Ar), 7.88-7.96 (2H, m, Ar), 8.59 (1H, d, $J = 7.8$ Hz, Ar); ^{13}C NMR (CDCl_3 , 75 MHz, TMS) δ 10.8, 10.9, 19.2, 51.8, 84.9, 118.1, 123.0, 126.7, 128.4, 128.8, 130.3, 130.9, 131.3, 131.5, 131.8, 134.7, 136.9, 149.5, 167.3; MS (ESI) m/z 305.2 ($\text{M}+\text{H}^+$); HRMS (MALDI) Calcd. for $\text{C}_{19}\text{H}_{17}\text{N}_2\text{O}_2^+$ ($\text{M}+\text{H}^+$) requires 305.1285, Found: 305.1304.

Compound **4j**: a pale yellow solid; m.p. 186 °C (decompose); IR (KBr) ν 3043, 1608, 1562, 1513, 1428, 1252, 1177, 1032, 829, 734, 687 cm^{-1} ; ¹H NMR (CDCl₃, 300 MHz, TMS) δ 0.68-0.77 (1H, m, CH₂), 0.98-1.07 (1H, m, CH₂), 1.76-1.85 (1H, m, CH₂), 2.07-2.16 (1H, m, CH₂), 2.40 (3H, s, CH₃), 6.10 (1H, s, CH), 7.17-7.19 (2H, m, Ar), 7.28-7.39 (2H, m, Ar), 7.77-7.82 (1H, m, Ar), 7.91-7.97 (2H, m, Ar), 8.58-8.61 (1H, m, Ar); ¹³C NMR (CDCl₃, 75 MHz, TMS) δ 10.6, 10.7, 21.3, 52.4, 86.7, 118.1, 123.0, 124.6, 128.0, 128.3, 129.0, 130.9, 131.1, 131.7, 133.4, 134.6, 139.2, 149.5, 167.2; MS (ESI) m/z 305.2 (M+H⁺); HRMS (MALDI) Calcd. for C₁₉H₁₇N₂O₂⁺ (M+H⁺) requires 305.1285, Found: 305.1284.

Compound **4k**: a pale yellow solid; m.p. 188 °C (decompose); IR (KBr) ν 3042, 1606, 1564, 1519, 1433, 1182, 1085, 771, 734, 687 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz, TMS) δ 0.70-0.78 (1H, m, CH_2), 0.98-1.06 (1H, m, CH_2), 1.76-1.84 (1H, m, CH_2), 2.06-2.14 (1H, m, CH_2), 2.41 (3H, s, CH_3), 6.13 (1H, s, CH), 7.29 (4H, s, Ar), 7.76-7.81 (1H, m, Ar), 7.90-7.95 (2H, m, Ar), 8.58 (1H, d, $J = 8.1$ Hz, Ar); ^{13}C NMR (CDCl_3 , 75 MHz, TMS) δ 10.5, 10.7, 21.3, 52.4, 86.7, 118.2, 123.1, 127.7, 128.3, 129.8, 130.5, 130.9, 131.7, 134.6, 140.7, 149.5, 167.2; MS (ESI) m/z 305.2 ($\text{M}+\text{H}^+$); HRMS (MALDI) Calcd. for $\text{C}_{19}\text{H}_{17}\text{N}_2\text{O}_2^+$ ($\text{M}+\text{H}^+$) requires 305.1285, Found: 305.1282.

Compound **4l**: a pale yellow solid; m.p. 196 °C (decompose); IR (KBr) ν 3043, 1609, 1561, 1517, 1434, 1253, 1177, 832, 733, 686 cm^{-1} ; ¹H NMR (CDCl₃, 300 MHz, TMS) δ 0.73-0.81 (1H, m, CH₂), 0.97-1.06 (1H, m, CH₂), 1.77-1.85 (1H, m, CH₂), 2.03-2.12 (1H, m, CH₂), 3.85 (3H, s, CH₃), 6.15 (1H, s, CH), 6.98 (2H, d, J = 8.4 Hz, Ar), 7.36 (2H, d, J = 8.4 Hz, Ar), 7.75-7.80 (1H, m, Ar), 7.88-7.94 (2H, m, Ar), 8.56 (1H, d, J = 7.8 Hz, Ar); ¹³C NMR (CDCl₃, 75 MHz, TMS) δ 10.4, 10.6, 52.1, 55.3, 86.5, 114.4, 118.1, 123.0, 125.2, 128.1, 129.4, 130.8, 131.6, 134.4, 149.3, 161.0, 167.0; MS (ESI) m/z 321.2 (M+H⁺); HRMS (MALDI) Calcd. for C₁₉H₁₇N₂O₃⁺ (M+H⁺) requires 321.1234, Found: 321.1231.

Compound **4m**: a pale yellow solid; m.p. 196 °C (decompose); IR (KBr) ν 3036, 1604, 1560, 1520, 1433, 1247, 1186, 735, 686 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz, TMS) δ 0.67-0.75 (1H, m, CH_2), 0.92-1.00 (1H, m, CH_2), 1.73-1.82 (1H, m, CH_2), 1.93-2.02 (1H, m, CH_2), 4.86 (1H, d, $J = 10.5$ Hz, CH_2), 6.15 (1H, s, CH), 4.91 (1H, d, $J = 10.5$ Hz, CH_2), 6.20 (1H, s, CH), 7.02-7.14 (7H, m, Ar), 7.28-7.30 (1H, m, Ar), 7.44-7.50 (1H, m, Ar), 7.65-7.84 (3H, m, Ar), 8.43 (1H, d, $J = 7.8$ Hz, Ar); ^{13}C NMR (CDCl_3 , 75 MHz, TMS) δ 10.1, 11.8, 51.3, 70.5, 85.0, 111.9, 118.0, 120.8, 122.0, 122.6, 127.5, 128.2, 128.3, 128.4, 129.8, 130.7, 131.1, 131.8, 133.7, 134.4, 149.3, 157.1, 166.8; MS (ESI) m/z 397.2 ($\text{M}+\text{H}^+$); HRMS (MALDI) Calcd. for $\text{C}_{25}\text{H}_{20}\text{N}_2\text{O}_3\text{Na}^+$ ($\text{M}+\text{Na}^+$) requires 419.1366, Found: 419.1383.

Compound **4n**: a pale yellow solid; m.p. 198 °C (decompose); IR (KBr) ν 3045, 1606, 1560, 1518, 1190, 1087, 774, 687 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz, TMS) δ 0.66-0.74 (1H, m, CH_2), 1.08-1.14 (1H, m, CH_2), 1.71-1.80 (1H, m, CH_2), 2.02-2.11 (1H, m, CH_2), 6.27 (1H, s, CH), 7.38-7.47 (4H, m, Ar), 7.76-7.81 (1H, m, Ar), 7.88-7.94 (2H, m, Ar), 8.52 (1H, d, $J = 8.1$ Hz, Ar); ^{13}C NMR (CDCl_3 , 75 MHz, TMS) δ 10.3, 10.5, 52.3, 85.6, 117.9, 122.9, 128.0, 128.9, 129.3, 130.6, 131.7, 131.9, 134.7, 136.3, 149.3, 167.0; MS (ESI) m/z 325.2 ($\text{M}+\text{H}^+$); HRMS (MALDI) Calcd. for $\text{C}_{18}\text{H}_{14}\text{N}_2\text{O}_2\text{Cl}^+$ ($\text{M}+\text{H}^+$) requires 325.0738, Found: 325.0722.

Compound **4o**: a pale yellow solid; m.p. 194 °C (decompose); IR (KBr) ν 3046, 1606, 1560, 1518, 1185, 1085, 771, 735, 685 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz, TMS) δ 0.66-0.74 (1H, m, CH_2), 1.01-1.10 (1H, m, CH_2), 1.74-1.83 (1H, m, CH_2), 2.06-2.14 (1H, m, CH_2), 6.17 (1H, s, CH), 7.30 (2H, d, $J = 8.7$ Hz, Ar), 7.62 (2H, d, $J = 8.7$ Hz, Ar), 7.76-7.82 (1H, m, Ar), 7.88-7.95 (2H, m, Ar), 8.56 (1H, d, $J = 7.8$ Hz, Ar); ^{13}C NMR (CDCl_3 , 75 MHz, TMS) δ 10.5, 10.7, 52.4, 85.7, 118.0, 123.0, 124.7, 128.3, 129.2, 130.9, 131.8, 132.4, 132.5, 134.7, 149.3, 167.2; MS (ESI) m/z 369.0 ($\text{M}+\text{H}^+$); HRMS (MALDI) Calcd. for $\text{C}_{18}\text{H}_{14}\text{N}_2\text{O}_2\text{Br}^+$ ($\text{M}+\text{H}^+$) requires 369.0233, Found: 369.0224.

Compound **4p**: a pale yellow solid; m.p. 182 °C (decompose); IR (KBr) ν 3060, 1607, 1561, 1518, 1275, 1200, 1049, 772, 736, 687 cm^{-1} ; ¹H NMR (CDCl₃, 300 MHz, TMS) δ 0.50-0.58 (1H, m, CH₂), 1.14-1.22 (1H, m, CH₂), 1.63-1.71 (1H, m, CH₂), 1.99-2.10 (1H, m, CH₂), 2.04 (3H, s, CH₃), 7.37-7.49 (5H, m, Ar), 7.75-7.81 (1H, m, Ar), 7.91-7.96 (2H, m, Ar), 8.59 (1H, d, J = 8.7 Hz, Ar); ¹³C NMR (CDCl₃, 75 MHz, TMS) δ 8.2, 11.5, 23.9, 57.2, 91.0, 118.3, 123.0, 125.2, 128.4, 128.9, 129.3, 131.1, 131.7, 134.6, 138.2, 148.6, 167.3; MS (ESI) m/z 305.2 (M+H⁺); HRMS (MALDI) Calcd. for C₁₉H₁₇N₂O₂⁺ (M+H⁺) requires 305.1285, Found: 305.1295.

4q

Compound **4q**: a pale yellow solid; m.p. 178 °C (decompose); IR (KBr) ν 3067, 1607, 1559, 1518, 1277, 1196, 1042, 771, 687 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz, TMS) δ 0.47-0.56 (1H, m, CH_2), 1.01-1.09 (1H, m, CH_2), 1.69-1.77 (1H, m, CH_2), 2.09-2.18 (1H, m, CH_2), 2.09 (3H, s, CH_3), 2.26 (3H, s, CH_3), 7.26-7.31 (2H, m, Ar), 7.35-7.40 (2H, m, Ar), 7.78-7.84 (1H, m, Ar), 7.91-7.99 (2H, m, Ar), 8.62 (1H, d, $J = 8.1$ Hz, Ar); ^{13}C NMR (CDCl_3 , 75 MHz, TMS) δ 7.5, 12.5, 21.1, 25.5, 56.0, 92.0, 118.5, 122.9, 126.2, 127.0, 128.5, 130.1, 131.2, 131.8, 133.0, 134.1, 134.7, 138.2, 148.4, 167.2; MS (ESI) m/z 319.2 ($\text{M}+\text{H}^+$); HRMS (MALDI) Calcd. for $\text{C}_{20}\text{H}_{19}\text{N}_2\text{O}_2^+$ ($\text{M}+\text{H}^+$) requires 319.1441, Found: 319.1452.

Compound **5a**: a yellow solid; m.p. 142 °C (decompose); IR (KBr) ν 3056, 2920, 1602, 1560, 1508, 1202, 1080, 762, 698 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz, TMS) δ 0.76 (6H, s, CH_3), 1.46 (6H, s, CH_3), 7.42-7.45 (10H, m, Ar), 7.73-7.78 (1H, m, Ar), 7.88-7.93 (1H, m, Ar), 7.98-8.01 (1H, m, Ar), 8.58 (1H, d, $J = 8.1$ Hz, Ar); ^{13}C NMR (CDCl_3 , 75 MHz, TMS) δ 19.9, 20.9, 24.3, 26.2, 94.5, 118.3, 123.3, 127.9, 128.50, 128.53, 129.5, 131.4, 131.5, 134.9, 138.2, 138.4, 149.3, 167.0; MS (ESI) m/z 435.2 ($\text{M}+\text{H}^+$); HRMS (MALDI) Calcd. for $\text{C}_{29}\text{H}_{27}\text{N}_2\text{O}_2^+$ ($\text{M}+\text{H}^+$) requires 435.2067, Found: 435.2059.

5b

Compound **5b**: a yellow solid; m.p. 140 °C (decompose); IR (KBr) ν 3046, 2920, 1600, 1560, 1509, 1202, 1179, 808, 732, 695 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz, TMS) δ 0.78 (6H, s, CH_3), 1.47 (6H, s, CH_3), 2.38 (6H, s, CH_3), 7.22 (4H, d, $J = 8.4$ Hz, Ar), 7.29 (4H, d, $J = 8.4$ Hz, Ar), 7.71-7.76 (1H, m, Ar), 7.86-7.91 (1H, m, Ar), 7.96-7.99 (1H, m, Ar), 8.57 (1H, d, $J = 7.8$ Hz, Ar); ^{13}C NMR (CDCl_3 , 75 MHz, TMS) δ 19.9, 20.8, 21.0, 24.1, 25.9, 94.8, 118.3, 123.3, 127.8, 128.3, 129.0, 129.6, 131.2, 131.3, 134.7, 135.5, 137.6, 139.4, 149.4, 166.7; MS (ESI) m/z 463.3 ($\text{M}+\text{H}^+$); HRMS (MALDI) Calcd. for $\text{C}_{31}\text{H}_{30}\text{N}_2\text{O}_2\text{Na}^+$ ($\text{M}+\text{Na}^+$) requires 485.2200, Found: 485.2211.

Compound **5c**: a yellow solid; m.p. 150 °C (decompose); IR (KBr) ν 3031, 2917, 1592, 1554, 1489, 1201, 1091, 814, 721, 689 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz, TMS) δ 0.81 (6H, s, CH_3), 1.46 (6H, s, CH_3), 7.33 (4H, d, $J = 8.4$ Hz, Ar), 7.42 (4H, d, $J = 8.4$ Hz, Ar), 7.74-7.79 (1H, m, Ar), 7.90-7.98 (2H, m, Ar), 8.58 (1H, d, $J = 7.5$ Hz, Ar); ^{13}C NMR (CDCl_3 , 75 MHz, TMS) δ 20.2, 20.9, 24.5, 26.5, 93.2, 118.2, 123.2, 128.7, 128.9, 129.1, 129.2, 131.6, 135.2, 136.1, 136.7, 139.0, 149.0, 167.0; MS (ESI) m/z 503.2 ($\text{M}+\text{H}^+$); HRMS (MALDI) Calcd. for $\text{C}_{29}\text{H}_{25}\text{N}_2\text{O}_2\text{Cl}_2^+$ ($\text{M}+\text{H}^+$) requires 503.1288, Found: 503.1283.

Compound **5d**: a yellow solid; m.p. 138 °C (decompose); IR (KBr) ν 3047, 2919, 1601, 1560, 1508, 1235, 1160, 1104, 837, 734, 694 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz, TMS) δ 0.80 (6H, s, CH_3), 1.46 (6H, s, CH_3), 7.10-7.16 (4H, m, Ar), 7.37-7.41 (4H, m, Ar), 7.74-7.79 (1H, m, Ar), 7.90-7.99 (2H, m, Ar), 8.59 (1H, d, $J = 7.8$ Hz, Ar); ^{13}C NMR (CDCl_3 , 75 MHz, TMS) δ 20.0, 20.8, 24.3, 26.2, 93.3, 115.6 (d, $J_{\text{C-F}} = 21.9$ Hz), 118.1, 123.2, 128.4, 129.3, 129.8 (d, $J_{\text{C-F}} = 8.1$ Hz), 131.3, 131.5, 134.1 (d, $J_{\text{C-F}} = 3.2$ Hz), 135.0, 138.5, 149.0, 163.1 (d, $J_{\text{C-F}} = 248.8$ Hz), 166.8; MS (ESI) m/z 471.2 ($\text{M}+\text{H}^+$); HRMS (MALDI) Calcd. for $\text{C}_{29}\text{H}_{25}\text{N}_2\text{O}_2\text{F}_2^+$ ($\text{M}+\text{H}^+$) requires 471.1879, Found: 471.1887.

Compound **5e**: a yellow solid; m.p. 132 °C (decompose); IR (KBr) ν 3045, 2917, 1588, 1530, 1490, 1234, 1095, 812, 733, 699 cm⁻¹; ¹H NMR (CDCl₃, 300 MHz, TMS) δ 0.75 (3H, s, CH₃), 0.83 (3H, s, CH₃), 1.46 (3H, s, CH₃), 1.48 (3H, s, CH₃), 7.35-7.46 (9H, m, Ar), 7.74-7.80 (1H, m, Ar), 7.89-8.00 (2H, m, Ar), 8.58 (1H, d, J = 8.1 Hz, Ar); ¹³C NMR (CDCl₃, 75 MHz, TMS) δ 19.9, 20.1, 20.8, 20.9, 24.4, 26.3, 93.8, 118.2, 123.2, 127.7, 128.5, 128.6, 128.7, 129.2, 129.3, 129.7, 131.46, 131.50, 135.0, 135.7, 137.0, 138.0, 138.6, 149.1, 166.9; MS (ESI) m/z 469.2 (M+H⁺); HRMS (MALDI) Calcd. for C₂₉H₂₆N₂O₂Cl⁺ (M+H⁺) requires 469.1677, Found: 469.1673.

Compound **6a**: a pale yellow solid; m.p. 142 °C (decompose); IR (KBr) ν 3058, 2952, 1607, 1564, 1428, 1202, 1084, 886, 769, 704 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz, TMS) δ 1.50-1.62 (1H, m, CH_2), 2.12-2.22 (1H, m, CH_2), 2.30-2.40 (2H, m, CH_2), 3.16-3.27 (2H, m, CH_2), 7.37-7.45 (10H, m, Ar), 7.71-7.77 (1H, m, Ar), 7.87-7.94 (2H, m, Ar), 8.62 (1H, d, $J = 8.4$ Hz, Ar); ^{13}C NMR (CDCl_3 , 75 MHz, TMS) δ 13.5, 29.8, 75.7, 98.7, 118.5, 123.5, 127.1, 128.0, 128.5, 129.3, 130.9, 131.5, 134.6, 136.8, 146.5, 167.5; MS (ESI) m/z 381.2 ($\text{M}+\text{H}^+$); HRMS (MALDI) Calcd. for $\text{C}_{25}\text{H}_{21}\text{N}_2\text{O}_2^+$ ($\text{M}+\text{H}^+$) requires 381.1598, Found: 381.1594.

Compound **7a**: a white solid; m.p. 234-236 °C; IR (KBr) ν 1658, 1596, 1326, 1209, 898, 733, 694 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz, TMS) δ 5.00 (1H, d, J = 17.1 Hz, =CH₂), 5.16 (1H, d, J = 10.8 Hz, =CH₂), 6.66 (1H, dd, J = 10.8, 17.1 Hz, =CH), 6.99-7.08 (3H, m, Ar), 7.16-7.19 (2H, m, Ar), 7.38-7.48 (5H, m, Ar), 7.81-7.88 (2H, m, Ar), 8.04-8.07 (1H, m, Ar), 8.44-8.48 (1H, m, Ar); ^{13}C NMR (CDCl_3 , 75 MHz, TMS) δ 115.5, 125.1, 125.3, 127.78, 127.82, 127.9, 128.0, 128.3, 128.6, 129.5, 130.7, 130.8, 133.0, 133.3, 134.6, 139.4, 139.6, 144.9, 153.7, 157.8; MS (ESI) m/z 367.3 ($\text{M}+\text{H}^+$); HRMS (MALDI) Calcd. for $\text{C}_{24}\text{H}_{19}\text{N}_2\text{O}_2^+$ ($\text{M}+\text{H}^+$) requires 367.1441, Found: 367.1450.

The crystal data of **4a** has been deposited in CCDC with number 622720. Empirical Formula: $C_{24}H_{20}O_3N_2$; Formula Weight: 384.42; Crystal Color, Habit: colorless, prismatic; Crystal Dimensions: 0.480 x 0.337 x 0.180 mm; Crystal System: Monoclinic; Lattice Type: Primitive; Lattice Parameters: $a = 9.0803(9)\text{\AA}$, $b = 21.286(2)\text{\AA}$, $c = 9.9234(9)\text{\AA}$, $\alpha = 90^\circ$, $\beta = 101.687(2)^\circ$, $\gamma = 90^\circ$, $V = 1878.3(3)\text{\AA}^3$; Space group: $P2(1)/n$; $Z = 4$; $D_{calc} = 1.359\text{ g/cm}^3$; $F_{000} = 808$; Diffractometer: Rigaku AFC7R; Residuals: R ; R_w : 0.0470, 0.1026.

Table 1. Crystal data and structure refinement for cd26374.

Identification code	cd26374
Empirical formula	C ₂₄ H ₂₀ N ₂ O ₃
Formula weight	384.42
Temperature	293(2) K
Wavelength	0.71073 Å
Crystal system, space group	Monoclinic, P2(1)/n
Unit cell dimensions	a = 9.0803(9) Å alpha = 90 deg. b = 21.286(2) Å beta = 101.687(2) deg. c = 9.9234(9) Å gamma = 90 deg.
Volume	1878.3(3) Å ³
Z, Calculated density	4, 1.359 Mg/m ³
Absorption coefficient	0.091 mm ⁻¹
F(000)	808
Crystal size	0.480 x 0.337 x 0.180 mm
Theta range for data collection	1.91 to 27.00 deg.
Limiting indices	-11<=h<=10, -27<=k<=26, -12<=l<=10
Reflections collected / unique	10997 / 4082 [R(int) = 0.0766]
Completeness to theta = 27.00	99.3 %
Absorption correction	Empirical
Max. and min. transmission	1.00000 and 0.56006
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	4082 / 2 / 271
Goodness-of-fit on F ²	0.921
Final R indices [I>2sigma(I)]	R1 = 0.0470, wR2 = 0.1026
R indices (all data)	R1 = 0.0693, wR2 = 0.1120
Extinction coefficient	0.0041(12)
Largest diff. peak and hole	0.196 and -0.216 e.Å ⁻³

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for cd26374. $U(\text{eq})$ is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	$U(\text{eq})$
O(1)	1457(1)	44(1)	2076(1)	53(1)
O(2)	4228(1)	1474(1)	6224(1)	34(1)
O(3)	-1508(2)	-239(1)	768(2)	78(1)
N(1)	1387(2)	700(1)	3831(1)	37(1)
N(2)	2268(1)	1046(1)	4843(1)	31(1)
C(1)	2165(2)	377(1)	3027(2)	37(1)
C(2)	3815(2)	421(1)	3270(2)	33(1)
C(3)	4620(2)	97(1)	2419(2)	43(1)
C(4)	6153(2)	139(1)	2642(2)	48(1)
C(5)	6948(2)	502(1)	3720(2)	47(1)
C(6)	6198(2)	824(1)	4571(2)	40(1)
C(7)	4633(2)	784(1)	4345(2)	32(1)
C(8)	3724(2)	1101(1)	5147(2)	31(1)
C(9)	2887(2)	1827(1)	6508(2)	32(1)
C(10)	1591(2)	1405(1)	5810(2)	32(1)
C(11)	-18(2)	1589(1)	5431(2)	42(1)
C(12)	464(2)	1080(1)	6467(2)	43(1)
C(13)	2996(2)	1867(1)	8050(2)	35(1)
C(14)	1968(2)	2240(1)	8550(2)	46(1)
C(15)	1957(3)	2249(1)	9939(2)	60(1)
C(16)	2984(3)	1898(1)	10840(2)	64(1)
C(17)	4015(3)	1542(1)	10363(2)	60(1)
C(18)	4017(2)	1519(1)	8958(2)	44(1)
C(19)	2951(2)	2462(1)	5805(2)	34(1)
C(20)	3681(2)	2969(1)	6526(2)	45(1)
C(21)	3849(2)	3531(1)	5880(2)	56(1)
C(22)	3304(2)	3592(1)	4486(2)	54(1)
C(23)	2600(2)	3094(1)	3750(2)	51(1)
C(24)	2404(2)	2532(1)	4403(2)	42(1)

Table 3. Bond lengths [Å] and angles [deg] for cd26374.

O(1)-C(1)	1.2495(18)
O(2)-C(8)	1.3369(17)
O(2)-C(9)	1.5052(18)
O(3)-H(3A)	0.969(16)
O(3)-H(3B)	0.943(18)
N(1)-C(1)	1.355(2)
N(1)-N(2)	1.3650(16)
N(2)-C(8)	1.3004(19)
N(2)-C(10)	1.4571(18)
C(1)-C(2)	1.472(2)
C(2)-C(7)	1.402(2)
C(2)-C(3)	1.404(2)
C(3)-C(4)	1.368(2)
C(3)-H(3)	0.9300
C(4)-C(5)	1.396(2)
C(4)-H(4)	0.9300
C(5)-C(6)	1.372(2)
C(5)-H(5)	0.9300
C(6)-C(7)	1.396(2)
C(6)-H(6)	0.9300
C(7)-C(8)	1.426(2)
C(9)-C(13)	1.516(2)
C(9)-C(19)	1.528(2)
C(9)-C(10)	1.530(2)
C(10)-C(11)	1.486(2)
C(10)-C(12)	1.490(2)
C(11)-C(12)	1.496(2)
C(11)-H(11A)	0.9700
C(11)-H(11B)	0.9700
C(12)-H(12A)	0.9700
C(12)-H(12B)	0.9700
C(13)-C(18)	1.373(2)
C(13)-C(14)	1.391(2)
C(14)-C(15)	1.381(2)
C(14)-H(14)	0.9300
C(15)-C(16)	1.375(3)
C(15)-H(15)	0.9300
C(16)-C(17)	1.361(3)
C(16)-H(16)	0.9300
C(17)-C(18)	1.396(2)
C(17)-H(17)	0.9300
C(18)-H(18)	0.9300
C(19)-C(20)	1.386(2)
C(19)-C(24)	1.387(2)
C(20)-C(21)	1.381(2)
C(20)-H(20)	0.9300
C(21)-C(22)	1.377(3)
C(21)-H(21)	0.9300
C(22)-C(23)	1.370(3)
C(22)-H(22)	0.9300
C(23)-C(24)	1.389(2)
C(23)-H(23)	0.9300
C(24)-H(24)	0.9300
C(8)-O(2)-C(9)	106.68(11)
H(3A)-O(3)-H(3B)	110(3)
C(1)-N(1)-N(2)	114.16(13)
C(8)-N(2)-N(1)	129.45(13)
C(8)-N(2)-C(10)	110.17(12)
N(1)-N(2)-C(10)	120.33(12)
O(1)-C(1)-N(1)	118.92(15)
O(1)-C(1)-C(2)	120.70(15)
N(1)-C(1)-C(2)	120.38(14)
C(7)-C(2)-C(3)	117.93(15)
C(7)-C(2)-C(1)	121.62(14)
C(3)-C(2)-C(1)	120.45(15)
C(4)-C(3)-C(2)	120.49(16)
C(4)-C(3)-H(3)	119.8

C(2)-C(3)-H(3)	119.8
C(3)-C(4)-C(5)	120.76(17)
C(3)-C(4)-H(4)	119.6
C(5)-C(4)-H(4)	119.6
C(6)-C(5)-C(4)	120.28(17)
C(6)-C(5)-H(5)	119.9
C(4)-C(5)-H(5)	119.9
C(5)-C(6)-C(7)	119.13(16)
C(5)-C(6)-H(6)	120.4
C(7)-C(6)-H(6)	120.4
C(6)-C(7)-C(2)	121.42(15)
C(6)-C(7)-C(8)	124.51(14)
C(2)-C(7)-C(8)	114.07(14)
N(2)-C(8)-O(2)	113.98(13)
N(2)-C(8)-C(7)	120.29(14)
O(2)-C(8)-C(7)	125.73(14)
O(2)-C(9)-C(13)	109.12(12)
O(2)-C(9)-C(19)	104.41(11)
C(13)-C(9)-C(19)	114.17(12)
O(2)-C(9)-C(10)	101.55(11)
C(13)-C(9)-C(10)	112.16(12)
C(19)-C(9)-C(10)	114.21(13)
N(2)-C(10)-C(11)	119.74(13)
N(2)-C(10)-C(12)	117.60(13)
C(11)-C(10)-C(12)	60.38(11)
N(2)-C(10)-C(9)	102.58(12)
C(11)-C(10)-C(9)	125.91(13)
C(12)-C(10)-C(9)	127.44(13)
C(10)-C(11)-C(12)	59.95(11)
C(10)-C(11)-H(11A)	117.8
C(12)-C(11)-H(11A)	117.8
C(10)-C(11)-H(11B)	117.8
C(12)-C(11)-H(11B)	117.8
H(11A)-C(11)-H(11B)	114.9
C(10)-C(12)-C(11)	59.67(10)
C(10)-C(12)-H(12A)	117.8
C(11)-C(12)-H(12A)	117.8
C(10)-C(12)-H(12B)	117.8
C(11)-C(12)-H(12B)	117.8
H(12A)-C(12)-H(12B)	114.9
C(18)-C(13)-C(14)	119.16(16)
C(18)-C(13)-C(9)	121.96(15)
C(14)-C(13)-C(9)	118.77(15)
C(15)-C(14)-C(13)	120.32(19)
C(15)-C(14)-H(14)	119.8
C(13)-C(14)-H(14)	119.8
C(16)-C(15)-C(14)	120.05(19)
C(16)-C(15)-H(15)	120.0
C(14)-C(15)-H(15)	120.0
C(17)-C(16)-C(15)	120.00(18)
C(17)-C(16)-H(16)	120.0
C(15)-C(16)-H(16)	120.0
C(16)-C(17)-C(18)	120.5(2)
C(16)-C(17)-H(17)	119.8
C(18)-C(17)-H(17)	119.8
C(13)-C(18)-C(17)	119.96(18)
C(13)-C(18)-H(18)	120.0
C(17)-C(18)-H(18)	120.0
C(20)-C(19)-C(24)	118.07(15)
C(20)-C(19)-C(9)	120.79(14)
C(24)-C(19)-C(9)	120.88(14)
C(21)-C(20)-C(19)	121.42(17)
C(21)-C(20)-H(20)	119.3
C(19)-C(20)-H(20)	119.3
C(22)-C(21)-C(20)	119.72(17)
C(22)-C(21)-H(21)	120.1
C(20)-C(21)-H(21)	120.1
C(23)-C(22)-C(21)	119.87(17)
C(23)-C(22)-H(22)	120.1
C(21)-C(22)-H(22)	120.1
C(22)-C(23)-C(24)	120.43(17)
C(22)-C(23)-H(23)	119.8

C(24)-C(23)-H(23)	119.8
C(19)-C(24)-C(23)	120.46(16)
C(19)-C(24)-H(24)	119.8
C(23)-C(24)-H(24)	119.8

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for cd26374.
 The anisotropic displacement factor exponent takes the form:
 $-2 \pi^2 [h^2 a^{*2} U_{11} + \dots + 2 h k a^* b^* U_{12}]$

	U11	U22	U33	U23	U13	U12
O(1)	58(1)	57(1)	41(1)	-20(1)	4(1)	-12(1)
O(2)	34(1)	35(1)	33(1)	-9(1)	5(1)	0(1)
O(3)	62(1)	109(1)	63(1)	5(1)	18(1)	-12(1)
N(1)	38(1)	37(1)	34(1)	-6(1)	1(1)	-6(1)
N(2)	34(1)	30(1)	29(1)	-3(1)	5(1)	-2(1)
C(1)	47(1)	32(1)	31(1)	-2(1)	4(1)	-5(1)
C(2)	45(1)	26(1)	30(1)	2(1)	10(1)	-1(1)
C(3)	60(1)	34(1)	37(1)	-5(1)	13(1)	0(1)
C(4)	59(1)	40(1)	50(1)	-4(1)	26(1)	5(1)
C(5)	45(1)	41(1)	57(1)	-2(1)	19(1)	1(1)
C(6)	39(1)	36(1)	45(1)	-5(1)	9(1)	-2(1)
C(7)	39(1)	26(1)	32(1)	1(1)	7(1)	1(1)
C(8)	35(1)	28(1)	28(1)	-1(1)	4(1)	-2(1)
C(9)	33(1)	32(1)	33(1)	-4(1)	9(1)	2(1)
C(10)	34(1)	31(1)	32(1)	-4(1)	8(1)	-1(1)
C(11)	36(1)	41(1)	49(1)	-3(1)	7(1)	2(1)
C(12)	45(1)	43(1)	43(1)	-2(1)	14(1)	-8(1)
C(13)	41(1)	34(1)	32(1)	-4(1)	9(1)	-9(1)
C(14)	48(1)	51(1)	41(1)	-8(1)	14(1)	-2(1)
C(15)	67(1)	66(1)	55(1)	-21(1)	34(1)	-18(1)
C(16)	97(2)	64(1)	37(1)	-6(1)	28(1)	-25(1)
C(17)	89(2)	49(1)	37(1)	9(1)	5(1)	-11(1)
C(18)	59(1)	36(1)	37(1)	-1(1)	9(1)	-6(1)
C(19)	36(1)	32(1)	35(1)	-1(1)	12(1)	0(1)
C(20)	54(1)	39(1)	41(1)	-7(1)	9(1)	-6(1)
C(21)	70(1)	32(1)	66(1)	-7(1)	15(1)	-11(1)
C(22)	62(1)	36(1)	68(1)	13(1)	23(1)	1(1)
C(23)	63(1)	48(1)	44(1)	11(1)	11(1)	3(1)
C(24)	52(1)	38(1)	37(1)	-2(1)	9(1)	-4(1)

Table 5. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for cd26374.

	x	y	z	U(eq)
H(3)	4106	-147	1698	52
H(4)	6672	-78	2070	57
H(5)	7991	526	3861	56
H(6)	6725	1067	5289	48
H(11A)	-576	1468	4529	51
H(11B)	-307	1990	5763	51
H(12A)	468	1174	7424	51
H(12B)	199	651	6190	51
H(14)	1284	2485	7945	55
H(15)	1256	2493	10265	72
H(16)	2975	1903	11775	77
H(17)	4722	1312	10978	72
H(18)	4710	1267	8637	53
H(20)	4066	2929	7464	53
H(21)	4327	3868	6384	67
H(22)	3415	3969	4046	64
H(23)	2251	3132	2805	62
H(24)	1902	2201	3898	51
H(3A)	-500(20)	-109(11)	1210(20)	105(9)
H(3B)	-1660(40)	-179(17)	-190(20)	189(16)

Table 6. Torsion angles [deg] for cd26374.

C(1)-N(1)-N(2)-C(8)	-1.0(2)
C(1)-N(1)-N(2)-C(10)	-178.42(13)
N(2)-N(1)-C(1)-O(1)	179.59(13)
N(2)-N(1)-C(1)-C(2)	-0.6(2)
O(1)-C(1)-C(2)-C(7)	-178.93(14)
N(1)-C(1)-C(2)-C(7)	1.3(2)
O(1)-C(1)-C(2)-C(3)	1.2(2)
N(1)-C(1)-C(2)-C(3)	-178.62(14)
C(7)-C(2)-C(3)-C(4)	0.0(2)
C(1)-C(2)-C(3)-C(4)	179.89(15)
C(2)-C(3)-C(4)-C(5)	0.1(3)
C(3)-C(4)-C(5)-C(6)	-0.1(3)
C(4)-C(5)-C(6)-C(7)	0.0(3)
C(5)-C(6)-C(7)-C(2)	0.1(2)
C(5)-C(6)-C(7)-C(8)	-179.38(15)
C(3)-C(2)-C(7)-C(6)	-0.1(2)
C(1)-C(2)-C(7)-C(6)	-179.99(14)
C(3)-C(2)-C(7)-C(8)	179.44(13)
C(1)-C(2)-C(7)-C(8)	-0.4(2)
N(1)-N(2)-C(8)-O(2)	-179.05(12)
C(10)-N(2)-C(8)-O(2)	-1.38(17)
N(1)-N(2)-C(8)-C(7)	1.8(2)
C(10)-N(2)-C(8)-C(7)	179.49(13)
C(9)-O(2)-C(8)-N(2)	-13.25(16)
C(9)-O(2)-C(8)-C(7)	165.83(14)
C(6)-C(7)-C(8)-N(2)	178.57(15)
C(2)-C(7)-C(8)-N(2)	-1.0(2)
C(6)-C(7)-C(8)-O(2)	-0.4(2)
C(2)-C(7)-C(8)-O(2)	-179.97(13)
C(8)-O(2)-C(9)-C(13)	139.56(12)
C(8)-O(2)-C(9)-C(19)	-98.00(13)
C(8)-O(2)-C(9)-C(10)	20.99(14)
C(8)-N(2)-C(10)-C(11)	159.92(14)
N(1)-N(2)-C(10)-C(11)	-22.2(2)
C(8)-N(2)-C(10)-C(12)	-130.17(15)
N(1)-N(2)-C(10)-C(12)	47.74(19)
C(8)-N(2)-C(10)-C(9)	14.87(16)
N(1)-N(2)-C(10)-C(9)	-167.22(12)
O(2)-C(9)-C(10)-N(2)	-20.80(13)
C(13)-C(9)-C(10)-N(2)	-137.16(13)
C(19)-C(9)-C(10)-N(2)	90.94(14)
O(2)-C(9)-C(10)-C(11)	-162.92(14)
C(13)-C(9)-C(10)-C(11)	80.71(19)
C(19)-C(9)-C(10)-C(11)	-51.2(2)
O(2)-C(9)-C(10)-C(12)	119.45(16)
C(13)-C(9)-C(10)-C(12)	3.1(2)
C(19)-C(9)-C(10)-C(12)	-128.82(16)
N(2)-C(10)-C(11)-C(12)	106.79(16)
C(9)-C(10)-C(11)-C(12)	-116.85(17)
N(2)-C(10)-C(12)-C(11)	-110.29(16)
C(9)-C(10)-C(12)-C(11)	114.49(18)
O(2)-C(9)-C(13)-C(18)	-11.8(2)
C(19)-C(9)-C(13)-C(18)	-128.15(16)
C(10)-C(9)-C(13)-C(18)	99.93(18)
O(2)-C(9)-C(13)-C(14)	172.22(13)
C(19)-C(9)-C(13)-C(14)	55.9(2)
C(10)-C(9)-C(13)-C(14)	-76.07(18)
C(18)-C(13)-C(14)-C(15)	-1.3(3)
C(9)-C(13)-C(14)-C(15)	174.86(15)
C(13)-C(14)-C(15)-C(16)	1.2(3)
C(14)-C(15)-C(16)-C(17)	0.2(3)
C(15)-C(16)-C(17)-C(18)	-1.5(3)
C(14)-C(13)-C(18)-C(17)	0.0(2)
C(9)-C(13)-C(18)-C(17)	-176.03(16)
C(16)-C(17)-C(18)-C(13)	1.4(3)
O(2)-C(9)-C(19)-C(20)	-92.53(16)
C(13)-C(9)-C(19)-C(20)	26.5(2)
C(10)-C(9)-C(19)-C(20)	157.46(14)

O(2)-C(9)-C(19)-C(24)	81.51(17)
C(13)-C(9)-C(19)-C(24)	-159.41(15)
C(10)-C(9)-C(19)-C(24)	-28.5(2)
C(24)-C(19)-C(20)-C(21)	0.7(3)
C(9)-C(19)-C(20)-C(21)	174.96(17)
C(19)-C(20)-C(21)-C(22)	-1.1(3)
C(20)-C(21)-C(22)-C(23)	0.0(3)
C(21)-C(22)-C(23)-C(24)	1.3(3)
C(20)-C(19)-C(24)-C(23)	0.6(2)
C(9)-C(19)-C(24)-C(23)	-173.62(16)
C(22)-C(23)-C(24)-C(19)	-1.6(3)

Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for cd26374 [Å and deg.].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O(3)-H(3A)...O(1)	0.969(16)	1.842(17)	2.807(2)	173(2)
O(3)-H(3B)...O(1)#1	0.943(18)	1.94(2)	2.862(2)	167(4)

Symmetry transformations used to generate equivalent atoms:
#1 -x, -y, -z