Supporting Information

Palladium-mediated Ring Enlargement of Aryl Substituted Methylenecyclopropanes to Cyclobutenes

Min Shi,^{a*} Le-Ping Liu^b and Jie Tang^b

^aState Key Laboratory of Organometallic Chemistry, Shanghai Institute of Organic Chemistry,
Chinese Academy of Sciences, 354 Fenglin Lu, Shanghai 200032 China, mshi@pub.sioc.ac.cn.

Fax: 86-21-64166128

^bDepartment of Chemistry, East China Normal University, 3663 Zhongshanbei Lu,
Shanghai 200062 China

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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. Tetrahydrofuran (THF), toluene and Et_2O were dried by sodium/benzophenone. DCE, acetonitrile and chloroform were dried by calcium hydride. Commercially obtained reagents were used without further purification. All 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. Methylenecyclopropanes **1b-1f** are known compounds.

Typical reaction procedure for the ring enlargement of methylenecyclopropanes to cyclobutenes: To a solution of MCP **1a** (71 mg, 0.3 mmol) in 1,2-dichloroethane (DCE) (2.0 mL) was added palladium acetate (2.0 mg, 3 mol%) and copper(II) bromide (7.0 mg, 10 mol%), 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 flash column chromatography to give the desired product **2a** (66 mg, 93%) as a white solid.

Typical reaction procedure for the synthesis of deuterated methylenecyclopropane 1a-d (Scheme SI-1):

Scheme SI-1. The synthesis of methylenecyclopropane **1a-d**

1) To a solution of salicylic acid (1.38 g, 10 mmol) in acetone (20 mL) was added potassium carbonate (3.45 g, 25 mmol) and benzyl bromide (3.42 g, 20 mmol). The mixture was stirred for 10 hours under reflux (monitored by TLC) and then cooled to room temperature. The mixture was filtered and the filtrate was concentrated under reduced pressure. The residue was subjected to flash column chromatography to give the product **3** (2.68 g, 84%) as a white solid.

2) To a solution of compound **3** (1.60 g, 5.0 mmol) in THF (15 mL) was added LiAlD_4 (200 mg, 5.3 mmol) in small portion at 0°C . The mixture was warmed to room temperature and stirred for 1 hour. After the substrate was consumed (monitored by TLC), the reaction was quenched with diluted hydrochloric acid and extracted with diethyl ether. The organic layer was washed with brine and dried over anhydrous Na_2SO_4 , then the solvent was removed under reduced pressure and the residue was subjected to flash column chromatography to give the inseparable mixture of compound **4** and benzyl alcohol in 1:1 molar ratio (1.54 g, 94%) as a colorless oil.

3) To a solution of the mixture of compound **4** and benzyl alcohol (1.0 g, 3.0 mmol for each component) in dichloromethane (50 mL) was added PCC (2.6 g, 12 mmol). The mixture was stirred for 1 hour at room temperature (monitored by TLC) and then filtered, the filtrate was concentrated under reduced pressure and the residue was subjected to flash column chromatography to give the product **5** (0.61 g, 95%) as a colorless oil.

4) To a suspension of sodium hydride (0.2 g, 60%, 5.0 mmol) in THF (15 mL) was added (3-bromo-propyl)-triphenylphosphonium bromide (1.1 g, 2.4 mmol) and the mixture was stirred for 10 hours under reflux, then a solution of aldehyde **5** (0.42 g, 2.0 mmol) in THF (5.0 mL) was added to the mixture and stirred for another 2 hours. The reaction was quenched with water and extracted with petroleum ether. The organic layer was washed with brine and dried over anhydrous Na_2SO_4 , then the solvent was removed under reduced pressure and the residue was subjected to flash column chromatography to give methylenecyclopropane **1a-d** (266mg, 56%) as a white solid.

Another route for the formation of intermediate A and the corresponding mechanism was outlined in Scheme SI-2.

Scheme SI-2. Another route for the formation of intermediate **A** and the corresponding mechanism.

Compound **1a**: a white solid, m.p. 70-72 °C; IR (KBr) ν 3034, 2974, 1596, 1489, 1452, 1242, 1114, 748, 696 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz, TMS) δ 1.13-1.19 (2H, m, CH_2), 1.37-1.43 (2H, m, CH_2), 5.11 (2H, s, OCH_2), 6.93-6.98 (2H, m, Ar), 7.15-7.20 (1H, m, Ar), 7.23-7.24 (1H, m, $=\text{CH}$), 7.33-7.48 (5H, m, Ar), 7.78-7.81 (1H, m, Ar); ^{13}C NMR (CDCl_3 , 75 MHz, TMS) δ 0.5, 3.9, 70.2, 112.1, 112.3, 120.8, 124.1, 126.5, 127.2, 127.3, 127.6, 127.7, 128.4, 137.2, 155.2; MS (EI) m/z 236 (M^+ , 1), 145 (100), 115 (20), 91 (84); Anal. Calcd. for $\text{C}_{17}\text{H}_{16}\text{O}$ requires C, 86.40; H, 6.82%. Found: C, 86.11; H, 6.98%.

Compound **1a-d**: a white solid, m.p. 70-72 °C; IR (KBr) ν 3034, 2971, 1592, 1449, 1233, 1101, 754, 697 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz, TMS) δ 1.16 (2H, dd, $J = 5.1, 6.6$ Hz, CH_2), 1.40 (2H, dd, $J = 5.1, 6.6$ Hz, CH_2), 5.11 (2H, s, OCH_2), 6.93-6.98 (2H, m, Ar), 7.15-7.20 (1H, m, Ar), 7.30-7.48 (5H, m, Ar), 7.80 (1H, d, $J = 8.4$ Hz, Ar); ^{13}C NMR (CDCl_3 , 75 MHz, TMS) δ 0.5, 3.8, 70.1, 111.8 (t, $J_{\text{C-D}}$

= 24.4 Hz), 112.2, 120.8, 123.9, 126.4, 127.19, 127.22, 127.6, 127.7, 128.4, 137.1, 155.1; MS (EI) m/z 237 (M^+ , 1), 146 (64), 91 (100); HRMS (EI) Calcd. for $C_{17}H_{15}DO$ ($M^+ + H$) requires 237.1264, Found: 237.1272.

Compound **1g**: a colorless oil; IR (KBr) ν 2924, 2853, 1595, 1464, 1378, 683; ¹H NMR (CDCl₃, 300 MHz, TMS) δ 1.17-1.22 (2H, m, CH₂), 1.41-1.47 (2H, m, CH₂), 6.68-7.71 (1H, m, Ar), 7.15-7.27 (2H, m, 1H for Ar and 1H for =CH), 7.37-7.39 (1H, m, Ar), 7.51-7.53 (1H, m, Ar); ¹³C NMR (CDCl₃, 75 MHz, TMS) δ 0.6, 4.2, 117.1, 124.8, 126.2, 126.3, 126.5, 126.6, 129.6, 140.0; MS (EI) m/z 164

(M⁺, 12), 129 (100), 105 (18); HRMS (EI) Calcd. for C₁₀H₉Cl requires 164.0393, Found: 164.0385.

Compound **1h**: a white solid, m.p. 68-70 °C; IR (KBr) ν 3030, 2972, 1596, 1489, 1454, 1239, 1008, 747 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz, TMS) δ 1.10-1.15 (2H, m, CH_2), 1.34-1.40 (2H, m, CH_2), 2.37 (3H, s, CH_3), 5.05 (2H, s, OCH_2), 6.93-6.98 (2H, m, Ar), 7.15-7.25 (5H, m, 4H for Ar and 1H for $=\text{CH}$), 7.43-7.46 (1H, m, Ar), 7.78-7.81 (1H, m, Ar); ^{13}C NMR (CDCl_3 , 75 MHz, TMS) δ 0.5, 3.9, 18.9, 68.8, 112.0, 112.2, 120.8, 124.2, 125.9, 126.5, 127.4, 127.6, 128.0, 128.4, 130.2, 135.1, 136.5, 155.3; MS (EI) m/z 250 (M^+ , 1), 145 (57), 105 (100); Anal. Calcd. for $\text{C}_{18}\text{H}_{18}\text{O}$ requires C, 86.36; H, 7.25%. Found: C, 86.35; H, 7.23%.

Compound **1i**: a white solid, m.p. 44-46 °C; IR (KBr) ν 3066, 2925, 1597, 1409, 1241, 1031, 815, 751, 692 cm⁻¹; ¹H NMR (CDCl₃, 300 MHz, TMS) δ 1.14-1.20 (2H, m, CH₂), 1.36-1.42 (2H, m, CH₂), 5.01 (2H, s, OCH₂), 6.90 (1H, d, J = 8.1 Hz, Ar), 7.01 (1H, t, J = 7.5 Hz, Ar), 7.17-7.36 (3H, m, 2H for Ar and 1H for =CH), 7.47 (1H, d, J = 8.1 Hz, Ar), 7.56-7.57 (1H, m, Ar), 7.82-7.85 (1H, m, Ar); ¹³C NMR (CDCl₃, 75 MHz, TMS) δ 0.6, 3.9, 68.9, 111.8, 112.3, 121.3, 124.8, 126.4, 126.7, 127.5, 127.7, 129.1, 130.5, 131.7, 132.6, 137.5, 154.7; MS (EI) m/z 304 (M⁺, 1), 159 (35), 145 (100); Anal. Calcd. for C₁₇H₁₄Cl₂O requires C, 66.90; H, 4.62%. Found: C, 66.91; H, 4.70%.

Compound **1j**: a white solid, m.p. 50-52 °C; IR (KBr) ν 3030, 2936, 1579, 1474, 1271, 1090, 745, 697 cm⁻¹; ¹H NMR (CDCl₃, 300 MHz, TMS) δ 1.16-1.22 (2H, m, CH₂), 1.40-1.45 (2H, m, CH₂), 3.91 (3H, s, OCH₃), 5.05 (2H, s, OCH₂), 6.84-6.87 (1H, m, Ar), 7.09 (1H, t, J = 7.5 Hz, Ar), 7.20 (1H, m, =CH), 7.34-7.49 (4H, m, Ar), 7.55-7.58 (2H, m, Ar); ¹³C NMR (CDCl₃, 75 MHz, TMS) δ 0.5, 3.9, 55.7, 75.1, 110.4, 112.2, 118.4, 123.9, 125.3, 127.8, 128.1, 128.2, 132.4, 137.7, 144.9, 153.0; MS (EI) m/z 266 (M⁺, 1), 175 (84), 91 (100); Anal. Calcd. for C₁₈H₁₈O₂ requires C, 81.17; H, 6.81%. Found: C, 81.03; H, 6.89%.

Compound **1k**: a white solid, m.p. 64-66 °C; IR (KBr) ν 3022, 2978, 1557, 1455, 1374, 1215, 1168, 977, 749, 695 cm⁻¹; ¹H NMR (CDCl₃, 300 MHz, TMS) δ 1.15-1.20 (2H, m, CH₂), 1.38-1.43 (2H, m, CH₂), 4.93 (2H, s, OCH₂), 6.98-6.99 (1H, m, Ar), 7.25-7.26 (1H, m, =CH), 7.34-7.52 (5H, m, Ar), 7.64-7.65 (1H, m, Ar); ¹³C NMR (CDCl₃, 75 MHz, TMS) δ 0.8, 4.0, 75.6, 111.1, 124.9, 127.9, 128.2, 128.3, 128.49, 128.54, 129.2, 129.6, 134.7, 136.4, 150.3; MS (EI) m/z 304 (M⁺, 1), 213 (8), 91 (100); Anal. Calcd. for C₁₇H₁₄Cl₂O requires C, 66.90; H, 4.62%. Found: C, 66.83; H, 4.70%.

Compound **11**: a white solid, m.p. 100-102 °C; IR (KBr) ν 3057, 2973, 1578, 1474, 1270, 1091, 812, 750, 715 cm⁻¹; ¹H NMR (CDCl₃, 300 MHz, TMS) δ 1.14-1.19 (2H, m, CH₂), 1.38-1.44 (2H, m, CH₂), 2.47 (3H, s, CH₃), 3.90 (3H, s, OCH₃), 5.03 (2H, s, OCH₂), 6.83-6.87 (1H, m, Ar), 7.09 (1H, t, J = 8.1 Hz, Ar), 7.14-1-7.15 (1H, m, =CH), 7.24-7.29 (3H, m, Ar), 7.44-7.48 (1H, m, Ar), 7.57-7.60 (1H, m, Ar); ¹³C NMR (CDCl₃, 75 MHz, TMS) δ 0.6, 4.0, 18.8, 55.7, 73.0, 110.4, 112.2, 118.4, 123.9, 125.4, 125.8, 127.9, 128.8, 130.0, 132.6, 135.9, 136.5, 145.1, 153.2; MS (EI) m/z 280 (M⁺, 1), 175 (53), 105 (100); Anal. Calcd. for C₁₉H₂₀O₂ requires C, 81.40; H, 7.19%. Found: C, 81.38; H, 7.16%.

Compound **1m**: a colorless oil; IR (KBr) ν 3075, 2975, 1597, 1488, 1452, 1243, 1114, 935, 749 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz, TMS) δ 1.14-1.19 (2H, m, CH_2), 1.37-1.42 (2H, m, CH_2), 4.56-4.59 (2H, m, OCH_2), 5.27-5.31 (1H, m, $=\text{CH}_2$), 5.41-5.48 (1H, m, $=\text{CH}_2$), 6.05-6.14 (1H, m, $=\text{CH}$), 6.86-6.97 (2H, m, Ar), 7.14-7.20 (2H, m, 1H for Ar and 1H for $=\text{CH}$), 7.75-7.79 (1H, m, Ar); ^{13}C NMR (CDCl_3 , 75 MHz, TMS) δ 0.5, 3.8, 69.1, 112.1, 112.3, 117.1, 120.7, 124.1, 126.5, 127.3, 127.6, 133.4, 155.0; MS (EI) m/z 186 (M^+ , 1), 145 (100), 115 (53), 91 (32), 41 (17); HRMS (EI) Calcd. for $\text{C}_{10}\text{H}_9\text{O}^+$ ($\text{M}^+ - \text{C}_3\text{H}_5$) requires 145.0653, Found: 145.0647.

Compound **1n**: a white solid, m.p. 78-80 °C; IR (KBr) ν 3038, 2975, 1589, 1483, 1236, 1106, 749, 691 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz, TMS) δ 1.11-1.16 (2H, m, CH_2), 1.39-1.45 (2H, m, CH_2), 6.93-6.95 (3H, m, Ar), 7.02-7.07 (2H, m, Ar), 7.14-7.33 (4H, m, 3H for Ar and 1H for $=\text{CH}$), 7.88-7.90 (1H, m, Ar); ^{13}C NMR (CDCl_3 , 75 MHz, TMS) δ 0.6, 4.0, 111.8, 117.4, 120.3, 122.4, 124.1, 125.7, 126.9, 127.8, 129.6, 130.4, 152.8, 158.3; MS (EI) m/z 222 (M^+ , 16), 181 (34), 129 (100); Anal. Calcd. for $\text{C}_{16}\text{H}_{14}\text{O}$ requires C, 86.45; H, 6.35%. Found: C, 86.29; H, 6.40%.

Compound **10**: a colorless oil; IR (KBr) ν 3036, 2931, 1598, 1492, 1454, 1245, 1113, 939, 750 cm^{-1} ; ¹H NMR (CDCl₃, 300 MHz, TMS) δ 0.90 (3H, t, J = 7.2 Hz, CH₃), 1.13-1.18 (2H, m, CH₂), 1.33-1.39 (6H, m, 3CH₂), 1.44-1.54 (2H, m, CH₂), 1.77-1.87 (2H, m, CH₂), 3.98 (2H, t, J = 7.2 Hz, OCH₂), 6.84-6.94 (2H, m, Ar), 7.13-7.19 (2H, m, 1H for Ar and 1H for =CH), 7.74-7.77 (1H, m, Ar); ¹³C NMR (CDCl₃, 75 MHz, TMS) δ 0.5, 3.9, 14.0, 22.6, 25.8, 29.3, 31.6, 68.3, 111.9, 112.1, 120.3, 123.8, 126.4, 127.1, 127.6, 155.6; MS (EI) m/z 230 (M^+ , 5), 145 (100), 131 (29); HRMS (EI) Calcd. for C₁₆H₂₂O requires 230.1671, Found: 230.1661.

Compound **1p**: a white solid, m.p. 38-40 °C; IR (KBr) ν 3050, 2975, 1590, 1509, 1453, 1263, 1095, 808, 745, 695 cm⁻¹; ¹H NMR (CDCl₃, 300 MHz, TMS) δ 1.13-1.19 (2H, m, CH₂), 1.34-1.40 (2H, m, CH₂), 5.23 (2H, s, OCH₂), 7.26-7.48 (9H, m, 8H for Ar and 1H for =CH), 7.73-7.81 (2H, m, Ar), 8.00-8.03 (1H, m, Ar); ¹³C NMR (CDCl₃, 75 MHz, TMS) δ 3.3, 5.4, 71.4, 112.5, 115.2, 122.3, 123.5, 125.3, 125.9, 127.2, 127.7, 128.1, 128.3, 128.4, 129.2, 129.4, 132.3, 137.4, 153.4; MS (EI) m/z 286 (M⁺, 5), 195 (97), 91 (100); Anal. Calcd. for C₂₁H₁₈O requires C, 88.08; H, 6.34%. Found: C, 87.78; H, 6.20%.

Compound **2a**: a white solid, m.p. 40-42 °C; IR (KBr) ν 3066, 2914, 1594, 1577, 1491, 1244, 748, 697 cm⁻¹; ¹H NMR (CDCl₃, 300 MHz, TMS) δ 2.53 (2H, t, J = 3.0 Hz, CH₂), 2.83 (2H, t, J = 3.0 Hz, CH₂), 5.08 (2H, s, OCH₂), 6.30 (1H, t, J = 0.9 Hz, =CH), 6.91 (2H, t, J = 8.4 Hz, Ar), 7.13-7.21 (2H, m, Ar), 7.30-7.44 (5H, m, Ar); ¹³C NMR (CDCl₃, 75 MHz, TMS) δ 27.3, 29.8, 70.0, 111.4, 120.4, 123.9, 126.8, 127.6, 127.9, 128.2, 128.5, 132.4, 136.9, 142.7, 157.1; MS (EI) m/z 236 (M⁺, 1), 145 (9), 91 (100); HRMS (EI) Calcd. for C₁₇H₁₆O requires 236.1201, Found: 236.1210.

2a-d

Compound **2a-d**: a white solid, m.p. 40-42 °C; IR (KBr) ν 3064, 2916, 1602, 1570, 1489, 1242, 748, 697 cm⁻¹; ¹H NMR (CDCl₃, 300 MHz, TMS) δ 2.53 (2H, t, J = 3.3 Hz, CH₂), 2.84 (2H, t, J = 3.3 Hz, CH₂), 5.13 (2H, s, OCH₂), 6.91-6.96 (2H, m, Ar), 7.16-7.23 (2H, m, Ar), 7.33-7.48 (5H, m, Ar); ¹³C

NMR (CDCl₃, 75 MHz, TMS) δ 27.2, 29.7, 70.0, 111.4, 120.5, 123.9, 126.8, 127.6, 127.9, 128.2, 128.5, 132.2 (t, J_{C-D} = 27.2 Hz), 136.9, 142.5, 157.1; MS (EI) m/z 237 (M^+ , 6), 146 (17), 91 (100); HRMS (EI) Calcd. for C₁₇H₁₅DO requires 237.1264, Found: 237.1259.

2b

Compound **2b**. This is a known compound.¹ a colorless oil; ¹H NMR (CDCl₃, 300 MHz, TMS) δ 2.55 (2H, t, *J* = 3.0 Hz, CH₂), 2.83 (2H, t, *J* = 3.0 Hz, CH₂), 6.31 (1H, s, =CH), 7.22-7.40 (5H, m, Ar); ¹³C NMR (CDCl₃, 75 MHz, TMS) δ 26.2, 28.7, 124.2, 127.1, 127.4, 128.3, 135.0, 146.4.

Compound **2c**. This is a known compound.¹ a colorless oil; ¹H NMR (CDCl₃, 300 MHz, TMS) δ 2.36 (3H, s, CH₃), 2.54 (2H, t, J = 3.0 Hz, CH₂), 2.81 (2H, t, J = 3.0 Hz, CH₂), 6.24 (1H, s, =CH), 7.14 (2H, d, J = 8.1 Hz, Ar), 7.27 (2H, d, J = 8.1 Hz, Ar); ¹³C NMR (CDCl₃, 75 MHz, TMS) δ 21.3, 26.1, 28.7, 124.1, 125.9, 128.9, 132.4, 137.1, 146.3.

Compound **2d**. This is a known compound.¹ a white solid, m.p. 54-56 °C; IR (KBr) ν 2921, 1602,

1. The ¹H and ¹³C NMR spectroscopic data of compounds **2b**, **2c** and **2d** are similar to those reported in previous publications. See: (a) Leigh, W. J.; Postigo, J. A. *Can. J. Chem.* **1995**, 73, 191. (b) Barbero, A.; Cuadrado, P.; Carcia, C.; Rincon, J. A.; Pulido, F. J. *J. Org. Chem.* **1998**, 63, 7531. (c) Wilt, J. W.; Kosturik, J. M.; Orłowski, R. C. *J. Org. Chem.* **1965**, 30, 1052. (d) Hill, E. A.; Engel, M.

1506, 1252, 1173, 836, 758, 709 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz, TMS) δ 2.51 (2H, t, $J = 3.0$ Hz, CH_2), 2.78 (2H, t, $J = 3.0$ Hz, CH_2), 3.81 (3H, s, OCH_3), 6.14 (1H, s, $=\text{CH}$), 6.85 (2H, d, $J = 8.7$ Hz, Ar), 7.29 (2H, d, $J = 8.7$ Hz, Ar); ^{13}C NMR (CDCl_3 , 75 MHz, TMS) δ 26.0, 28.8, 55.2, 113.6, 124.5, 125.5, 128.2, 145.9, 159.0; MS (EI) m/z 160 (M^+ , 100), 145 (40), 129 (42), 115 (67); HRMS (EI) Calcd. for $\text{C}_{11}\text{H}_{12}\text{O}$ requires 160.0888, Found: 160.0896.

Compound **2e**: a white solid, m.p. 58-60 $^{\circ}\text{C}$; IR (KBr) ν 3056, 2921, 1602, 1503, 1466, 1209, 1158, 1031, 835, 761 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz, TMS) δ 2.57 (2H, t, $J = 3.3$ Hz, CH_2), 2.83 (2H, t, $J = 3.3$ Hz, CH_2), 3.82 (3H, s, OCH_3), 3.86 (3H, s, OCH_3), 6.22 (1H, s, $=\text{CH}$), 6.45-6.48 (2H, m, Ar), 7.09-7.12 (1H, m, Ar); ^{13}C NMR (CDCl_3 , 75 MHz, TMS) δ 27.3, 29.7, 55.1, 55.3, 98.3, 103.8, 117.4, 127.3, 129.0, 142.5, 159.2, 160.1; MS (EI) m/z 190 (M^+ , 100), 175 (54), 159 (9), 147 (18); HRMS

(EI) Calcd. for $C_{12}H_{14}O_2$ requires 190.0994, Found: 190.0997.

Compound **2f**: a colorless oil; IR (KBr) ν 3044, 2922, 1488, 1462, 1239, 1092, 1013, 832, 754 cm^{-1} ; 1H NMR (CDCl₃, 300 MHz, TMS) δ 2.54 (2H, t, J = 3.3 Hz, CH₂), 2.80 (2H, t, J = 3.3 Hz, CH₂), 6.30 (1H, s, =CH), 7.25-7.29 (4H, m, Ar); ^{13}C NMR (CDCl₃, 75 MHz, TMS) δ 26.2, 28.7, 125.5, 127.8, 128.4, 129.6, 133.4, 145.2; MS (EI) m/z 164 (M^+ , 17), 129 (100), 101 (14); HRMS (EI) Calcd. for $C_{10}H_9Cl$ requires 164.0393, Found: 164.0400.

Compound **2g**: a colorless oil; IR (KBr) ν 2924, 2853, 1588, 1463, 1377, 753 cm^{-1} ; ^1H NMR (CDCl₃, 300 MHz, TMS) δ 2.53 (2H, t, $J = 3.3$ Hz, CH₂), 2.79 (2H, t, $J = 3.3$ Hz, CH₂), 6.33 (1H, s, =CH), 7.12-7.31 (4H, m, Ar); ^{13}C NMR (CDCl₃, 75 MHz, TMS) δ 26.3, 28.7, 122.3, 124.3, 126.0, 126.5, 127.3, 128.4, 128.9, 129.6; MS (EI) m/z 164 (M^+ , 27), 129 (63), 101 (100); HRMS (EI) Calcd. for C₁₀H₉Cl requires 164.0393, Found: 164.0402.

Compound **2h**: a white solid, m.p. 42–44 °C; IR (KBr) ν 3069, 2919, 1596, 1491, 1242, 1005, 854, 749, 719 cm^{-1} ; ¹H NMR (CDCl₃, 300 MHz, TMS) δ 2.37 (3H, s, CH₃), 2.49 (2H, t, J = 3.3 Hz, CH₂), 2.81 (2H, t, J = 3.3 Hz, CH₂), 5.06 (2H, s, OCH₂), 6.20 (1H, s, =CH), 6.90–6.95 (2H, m, Ar), 7.16–7.24 (5H, m, Ar), 7.38–7.41 (1H, m, Ar); ¹³C NMR (CDCl₃, 75 MHz, TMS) δ 18.8, 27.3, 29.7, 68.5, 111.3, 120.4, 123.9, 125.9, 126.8, 128.17, 128.23, 128.8, 130.3, 132.3, 134.8, 136.7, 142.7, 157.2; MS (EI) m/z 250 (M⁺, 5), 235 (3), 145 (20), 105 (100); HRMS (EI) Calcd. for C₁₈H₁₈O requires 250.1358, Found: 250.1366.

Compound **2i**: a white solid, m.p. 52-54 °C; IR (KBr) ν 3072, 2916, 1596, 1492, 1448, 1242, 1031, 751, 717, 691 cm^{-1} ; ¹H NMR (CDCl₃, 300 MHz, TMS) δ 2.55 (2H, t, J = 3.3 Hz, CH₂), 2.84 (2H, t, J = 3.3 Hz, CH₂), 5.04 (2H, s, OCH₂), 6.28 (1H, s, =CH), 6.83-6.97 (2H, m, Ar), 7.15-7.29 (3H, m, Ar), 7.43-7.46 (1H, m, Ar), 7.53-7.54 (1H, m, Ar); ¹³C NMR (CDCl₃, 75 MHz, TMS) δ 27.3, 29.8, 68.5, 111.3, 120.8, 123.9, 126.7, 126.9, 128.2, 129.3, 130.5, 131.8, 132.4, 132.5, 137.1, 142.5, 156.5; MS (EI) m/z 304 (M⁺, 8), 269 (2), 159 (100), 145 (61); HRMS (EI) Calcd. for C₁₇H₁₄Cl₂O requires 304.0422, Found: 304.0426.

Compound **2j**: a white solid, m.p. 64-66 °C; IR (KBr) ν 3064, 2919, 1572, 1466, 1438, 1262, 1130, 750, 697 cm⁻¹; ¹H NMR (CDCl₃, 300 MHz, TMS) δ 2.58 (2H, t, J = 3.3 Hz, CH₂), 2.90 (2H, t, J = 3.3 Hz, CH₂), 3.90 (3H, s, OCH₃), 5.09 (2H, s, CH₂), 6.44 (1H, s, =CH), 6.88-6.94 (2H, m, Ar), 7.06-7.12 (1H, m, Ar), 7.38-7.47 (3H, m, Ar), 7.56-7.59 (2H, m, Ar); ¹³C NMR (CDCl₃, 75 MHz, TMS) δ 27.3, 30.3, 55.8, 73.6, 111.7, 118.7, 123.8, 127.6, 127.9, 128.2, 128.9, 132.4, 137.9, 142.7, 145.9, 152.7; MS (EI) m/z 266 (M⁺, 4), 251 (1), 238 (6), 175 (22), 91 (100); HRMS (EI) Calcd. for C₁₈H₁₈O₂ requires 266.1307, Found: 266.1309.

Compound **2k**: a colorless oil; IR (KBr) ν 3067, 2918, 1576, 1498, 1446, 1251, 1005, 855, 765, 695 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz, TMS) δ 2.54 (2H, t, $J = 3.3$ Hz, CH_2), 2.81 (2H, t, $J = 3.3$ Hz, CH_2), 4.96 (2H, s, OCH_2), 6.45 (1H, s, $=\text{CH}$), 7.09-7.10 (1H, m, Ar), 7.28-7.29 (1H, m, Ar), 7.38-7.41 (3H, m, Ar), 7.50-7.53 (2H, m, Ar); ^{13}C NMR (CDCl_3 , 75 MHz, TMS) δ 27.4, 30.3, 73.8, 125.4, 127.9, 128.22, 128.24, 128.5, 128.6, 129.3, 129.4, 131.1, 135.4, 136.6, 140.8; MS (EI) m/z 304 (M^+ , 1), 213 (9), 91 (100); HRMS (EI) Calcd. for $\text{C}_{17}\text{H}_{14}\text{Cl}_2\text{O}$ requires 304.0422, Found: 304.0413.

Compound **2l**: a white solid, m.p. 68-69 °C; IR (KBr) ν 3063, 2915, 1572, 1466, 1397, 1261, 1040, 749 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz, TMS) δ 2.40 (3H, s, CH_3), 2.49 (2H, t, $J = 3.3$ Hz, CH_2), 2.82 (2H, t, $J = 3.3$ Hz, CH_2), 3.84 (3H, s, OCH_3), 5.01 (2H, s, OCH_2), 6.32 (1H, s, $=\text{CH}$), 6.83-6.88 (2H, m, Ar), 7.01-7.07 (1H, m, Ar), 7.19-7.26 (3H, m, Ar), 7.54-7.57 (1H, m, Ar); ^{13}C NMR (CDCl_3 , 75 MHz, TMS) δ 18.7, 27.3, 30.2, 55.8, 71.7, 111.7, 118.8, 123.9, 125.8, 127.6, 128.3, 129.0, 130.0, 132.5, 136.1, 136.3, 142.6, 146.0, 153.1; MS (EI) m/z 280 (M^+ , 2), 175 (20), 105 (100); Anal. Calcd. for $\text{C}_{19}\text{H}_{20}\text{O}_2$ requires C, 81.40; H, 7.19%. Found: C, 81.10; H, 7.45%.

Compound **2m**: a colorless oil; IR (KBr) ν 3075, 2917, 1597, 1490, 1449, 1286, 1242, 753, 719 cm^{-1} ; ¹H NMR (CDCl₃, 300 MHz, TMS) δ 2.57 (2H, t, J = 3.0 Hz, CH₂), 2.86 (2H, t, J = 3.0 Hz, CH₂), 4.59-4.62 (2H, m, OCH₂), 5.27-5.32 (1H, m, =CH₂), 5.39-5.46 (1H, m, =CH₂), 6.04-6.17 (1H, m, =CH), 6.39 (1H, s, =CH), 6.85-6.95 (2H, m, Ar), 7.15-7.21 (2H, m, Ar); ¹³C NMR (CDCl₃, 75 MHz, TMS) δ 27.3, 29.8, 68.8, 111.4, 117.6, 120.4, 123.9, 126.7, 128.2, 132.2, 133.3, 142.8, 157.0; MS (EI) m/z 186 (M⁺, 12), 145 (54), 131 (39), 115 (100), 41 (43); HRMS (EI) Calcd. for C₁₃H₁₄O requires 186.1045, Found: 186.1053.

Compound **2n**: a white solid, m.p. 84-86 °C; IR (KBr) ν 3070, 2920, 1597, 1484, 1443, 1236, 1166, 752, 689 cm⁻¹; ¹H NMR (CDCl₃, 300 MHz, TMS) δ 2.51 (2H, t, J = 3.0 Hz, CH₂), 2.84 (2H, t, J = 3.0 Hz, CH₂), 6.31 (1H, s, =CH), 6.85-6.97 (3H, m, Ar), 7.03-7.19 (3H, m, Ar), 7.28-7.33 (3H, m, Ar); ¹³C NMR (CDCl₃, 75 MHz, TMS) δ 27.4, 29.9, 117.9, 119.5, 122.7, 123.6, 126.7, 127.1, 128.3, 129.7, 132.9, 142.1, 154.5, 157.2; MS (EI) m/z 222 (M⁺, 52), 194 (100), 165 (55), 131 (72), 107 (82); HRMS (EI) Calcd. for C₁₆H₁₄O requires 222.1045, Found: 222.1050.

Compound **2o**: a colorless oil; IR (KBr) ν 3074, 2929, 1577, 1493, 1248, 1129, 851, 748, 718 cm^{-1} ; ¹H NMR (CDCl_3 , 300 MHz, TMS) δ 0.91 (3H, t, J = 6.6 Hz, CH_3), 1.34-1.36 (4H, m, 2CH_2), 1.47-1.54 (2H, m, CH_2), 1.81-1.86 (2H, m, CH_2), 2.57 (2H, t, J = 3.0 Hz, CH_2), 2.85 (2H, t, J = 3.0 Hz, CH_2), 4.00 (2H, t, J = 6.6 Hz, OCH_2), 6.38 (1H, s, =CH), 6.82-6.91 (2H, m, Ar), 7.14-7.19 (2H, m, Ar); ¹³C NMR (CDCl_3 , 75 MHz, TMS) δ 14.0, 22.6, 25.9, 27.3, 29.4, 29.8, 31.6, 67.9, 110.9, 119.9, 123.6, 126.7, 128.3, 131.9, 143.6, 157.6; MS (EI) m/z 230 (M^+ , 49), 202 (7), 159 (16), 145 (100), 131 (73); HRMS (EI) Calcd. for $\text{C}_{16}\text{H}_{22}\text{O}$ requires 230.1671, Found: 230.1677.

Compound **2p**: a yellow oil; IR (KBr) ν 3062, 2922, 1593, 1510, 1454, 1244, 1064, 808, 750, 697 cm^{-1} ; ¹H NMR (CDCl₃, 300 MHz, TMS) δ 2.69 (2H, t, J = 3.3 Hz, CH₂), 3.21 (2H, t, J = 3.3 Hz, CH₂), 5.23 (2H, s, OCH₂), 6.46 (1H, s, =CH), 7.27-7.49 (8H, m, Ar), 7.69-7.78 (2H, m, Ar), 8.37-8.40 (1H, m, Ar); ¹³C NMR (CDCl₃, 75 MHz, TMS) δ 28.9, 35.2, 71.5, 115.1, 119.6, 123.6, 124.8, 126.4, 127.5, 127.9, 128.2, 128.5, 128.8, 129.4, 132.6, 135.3, 137.2, 142.8, 154.5; MS (EI) m/z 286 (M^+ , 2), 195 (6), 105 (53), 91 (35), 84 (100); HRMS (MALDI) Calcd. for C₂₁H₁₉O⁺ (M^+ +H) requires 287.1430, Found: 287.1427.

Compound **5**: a colorless oil; IR (KBr) ν 3066, 2926, 1735, 1665, 1598, 1454, 1287, 1005, 757, 697 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz, TMS) δ 5.20 (2H, s, OCH_2), 7.03-7.07 (2H, m, Ar), 7.36-7.46 (5H, m, Ar), 7.51-7.57 (1H, m, Ar), 7.85-7.89 (1H, m, Ar); ^{13}C NMR (CDCl_3 , 75 MHz, TMS) δ 70.0, 112.7, 120.7, 124.7, 127.0, 127.9, 128.0, 128.4, 135.7, 135.8, 160.7, 189.0 (t, $J_{\text{C-D}} = 27.2$ Hz); MS (EI) m/z 213 (M^+ , 2), 183 (4), 122 (6), 91 (100); HRMS (EI) Calcd. for $\text{C}_{14}\text{H}_{11}\text{DO}_2$ requires 213.0900, Found: 213.0896.

The crystal data of **2I** has been deposited in CCDC with number 289394. Empirical Formula: $C_{19}H_{20}O_2$; Formula Weight: 280.35; Crystal Color, Habit: colorless, prismatic; Crystal Dimensions: 0.357 x 0.350 x 0.159 mm; Crystal System: Triclinic; Lattice Type: Primitive; Lattice Parameters: $a = 7.3656(8)\text{\AA}$, $b = 8.1841(10)\text{\AA}$, $c = 13.2944(15)\text{\AA}$, $\alpha = 98.814(2)^\circ$, $\beta = 93.413(2)^\circ$, $\gamma = 103.371(2)^\circ$, $V = 766.70(15)\text{\AA}^3$; Space group: P-1; $Z = 2$; $D_{\text{calc}} = 1.214\text{ g/cm}^3$; $F_{000} = 300$; Diffractometer: Rigaku AFC7R; Residuals: R ; R_w : 0.0446, 0.0698.

Table 1. Crystal data and structure refinement for cd25518.

Identification code	cd25518
Empirical formula	C19 H20 O2
Formula weight	280.35
Temperature	293(2) K
Wavelength	0.71073 Å
Crystal system, space group	Triclinic, P-1
Unit cell dimensions	a = 7.3656(8) Å alpha = 98.814(2) deg. b = 8.1841(10) Å beta = 93.413(2) deg. c = 13.2944(15) Å gamma = 103.371(2) deg.
Volume	766.70(15) Å ³
Z, Calculated density	2, 1.214 Mg/m ³
Absorption coefficient	0.077 mm ⁻¹
F(000)	300
Crystal size	0.357 x 0.350 x 0.159 mm
Theta range for data collection	1.56 to 26.99 deg.
Limiting indices	-8<=h<=9, -10<=k<=10, -16<=l<=12
Reflections collected / unique	4567 / 3259 [R(int) = 0.0300]
Completeness to theta = 26.99	97.1 %
Absorption correction	Empirical
Max. and min. transmission	1.00000 and 0.75954
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	3259 / 0 / 228
Goodness-of-fit on F ²	0.791
Final R indices [I>2sigma(I)]	R1 = 0.0446, wR2 = 0.0698
R indices (all data)	R1 = 0.1030, wR2 = 0.0845
Largest diff. peak and hole	0.119 and -0.134 e.Å ⁻³

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for cd25518. $U(\text{eq})$ is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)
O(1)	2338(2)	4167(2)	7655(1)	49(1)
O(2)	-845(2)	1717(2)	7468(1)	62(1)
C(1)	2300(3)	2772(2)	8132(1)	45(1)
C(2)	632(3)	1501(2)	8057(1)	48(1)
C(3)	561(3)	158(3)	8572(2)	59(1)
C(4)	2140(3)	75(3)	9160(2)	66(1)
C(5)	3772(3)	1330(3)	9242(2)	60(1)
C(6)	3886(3)	2700(2)	8719(1)	46(1)
C(7)	5657(3)	3988(2)	8816(1)	48(1)
C(8)	6343(3)	5411(3)	8445(2)	62(1)
C(9)	8277(3)	5738(2)	8987(2)	63(1)
C(10)	7479(3)	4107(3)	9434(2)	60(1)
C(11)	2435(3)	3830(2)	6573(1)	52(1)
C(12)	2548(2)	5436(2)	6136(2)	44(1)
C(13)	2701(3)	6981(3)	6768(2)	53(1)
C(14)	2826(3)	8460(3)	6360(2)	62(1)
C(15)	2780(3)	8383(4)	5321(2)	71(1)
C(16)	2606(3)	6837(3)	4692(2)	65(1)
C(17)	2500(2)	5351(3)	5084(2)	51(1)
C(18)	2333(3)	3695(3)	4373(1)	67(1)
C(19)	-2570(2)	447(3)	7371(2)	75(1)

Table 3. Bond lengths [Å] and angles [deg] for cd25518.

O(1)-C(1)	1.384(2)
O(1)-C(11)	1.4316(19)
O(2)-C(2)	1.364(2)
O(2)-C(19)	1.4284(18)
C(1)-C(6)	1.383(2)
C(1)-C(2)	1.401(2)
C(2)-C(3)	1.374(3)
C(3)-C(4)	1.384(3)
C(3)-H(3)	0.977(16)
C(4)-C(5)	1.375(2)
C(4)-H(4)	0.987(19)
C(5)-C(6)	1.397(2)
C(5)-H(5)	0.9300
C(6)-C(7)	1.461(2)
C(7)-C(8)	1.337(3)
C(7)-C(10)	1.506(3)
C(8)-C(9)	1.504(3)
C(8)-H(8)	0.902(18)
C(9)-C(10)	1.550(3)
C(9)-H(9A)	0.9700
C(9)-H(9B)	0.9700
C(10)-H(10A)	0.983(18)
C(10)-H(10B)	0.994(18)
C(11)-C(12)	1.504(2)
C(11)-H(11A)	0.9700
C(11)-H(11B)	0.9700
C(12)-C(13)	1.384(2)
C(12)-C(17)	1.387(2)
C(13)-C(14)	1.387(3)
C(13)-H(13)	0.972(16)
C(14)-C(15)	1.371(3)
C(14)-H(14)	0.986(16)
C(15)-C(16)	1.380(3)
C(15)-H(15)	0.94(2)
C(16)-C(17)	1.383(3)
C(16)-H(16)	0.965(17)
C(17)-C(18)	1.504(2)
C(18)-H(18A)	0.9600
C(18)-H(18B)	0.9600
C(18)-H(18C)	0.9600
C(19)-H(19A)	0.9600
C(19)-H(19B)	0.9600
C(19)-H(19C)	0.9600
C(1)-O(1)-C(11)	114.15(13)
C(2)-O(2)-C(19)	117.56(15)
C(6)-C(1)-O(1)	119.31(16)
C(6)-C(1)-C(2)	121.03(17)
O(1)-C(1)-C(2)	119.57(17)
O(2)-C(2)-C(3)	124.31(18)
O(2)-C(2)-C(1)	115.85(17)
C(3)-C(2)-C(1)	119.83(19)
C(2)-C(3)-C(4)	119.5(2)
C(2)-C(3)-H(3)	117.8(10)
C(4)-C(3)-H(3)	122.7(11)
C(5)-C(4)-C(3)	120.7(2)
C(5)-C(4)-H(4)	119.3(11)
C(3)-C(4)-H(4)	120.0(11)
C(4)-C(5)-C(6)	120.8(2)
C(4)-C(5)-H(5)	119.6
C(6)-C(5)-H(5)	119.6
C(1)-C(6)-C(5)	118.06(17)
C(1)-C(6)-C(7)	123.02(18)
C(5)-C(6)-C(7)	118.92(18)
C(8)-C(7)-C(6)	137.5(2)
C(8)-C(7)-C(10)	93.18(18)
C(6)-C(7)-C(10)	129.31(19)
C(7)-C(8)-C(9)	94.9(2)

C(7)-C(8)-H(8)	135.2(12)
C(9)-C(8)-H(8)	129.7(12)
C(8)-C(9)-C(10)	85.27(15)
C(8)-C(9)-H(9A)	114.4
C(10)-C(9)-H(9A)	114.4
C(8)-C(9)-H(9B)	114.4
C(10)-C(9)-H(9B)	114.4
H(9A)-C(9)-H(9B)	111.6
C(7)-C(10)-C(9)	86.61(17)
C(7)-C(10)-H(10A)	114.5(10)
C(9)-C(10)-H(10A)	114.6(11)
C(7)-C(10)-H(10B)	113.9(11)
C(9)-C(10)-H(10B)	118.0(11)
H(10A)-C(10)-H(10B)	108.1(16)
O(1)-C(11)-C(12)	110.00(15)
O(1)-C(11)-H(11A)	109.7
C(12)-C(11)-H(11A)	109.7
O(1)-C(11)-H(11B)	109.7
C(12)-C(11)-H(11B)	109.7
H(11A)-C(11)-H(11B)	108.2
C(13)-C(12)-C(17)	120.1(2)
C(13)-C(12)-C(11)	120.88(18)
C(17)-C(12)-C(11)	118.99(19)
C(12)-C(13)-C(14)	120.5(2)
C(12)-C(13)-H(13)	120.3(10)
C(14)-C(13)-H(13)	119.2(10)
C(15)-C(14)-C(13)	119.6(3)
C(15)-C(14)-H(14)	121.1(11)
C(13)-C(14)-H(14)	119.2(11)
C(14)-C(15)-C(16)	119.8(2)
C(14)-C(15)-H(15)	121.7(12)
C(16)-C(15)-H(15)	118.4(12)
C(15)-C(16)-C(17)	121.6(2)
C(15)-C(16)-H(16)	122.1(11)
C(17)-C(16)-H(16)	116.3(11)
C(16)-C(17)-C(12)	118.4(2)
C(16)-C(17)-C(18)	119.9(2)
C(12)-C(17)-C(18)	121.63(19)
C(17)-C(18)-H(18A)	109.5
C(17)-C(18)-H(18B)	109.5
H(18A)-C(18)-H(18B)	109.5
C(17)-C(18)-H(18C)	109.5
H(18A)-C(18)-H(18C)	109.5
H(18B)-C(18)-H(18C)	109.5
O(2)-C(19)-H(19A)	109.5
O(2)-C(19)-H(19B)	109.5
H(19A)-C(19)-H(19B)	109.5
O(2)-C(19)-H(19C)	109.5
H(19A)-C(19)-H(19C)	109.5
H(19B)-C(19)-H(19C)	109.5

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for cd25518.
 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)	57(1)	44(1)	45(1)	12(1)	2(1)	9(1)
O(2)	45(1)	64(1)	69(1)	19(1)	-5(1)	-2(1)
C(1)	53(1)	38(1)	45(1)	12(1)	9(1)	9(1)
C(2)	46(1)	48(1)	48(1)	11(1)	5(1)	7(1)
C(3)	55(1)	51(2)	68(2)	19(1)	8(1)	1(1)
C(4)	68(2)	61(2)	74(2)	35(1)	12(1)	11(1)
C(5)	58(1)	60(2)	70(2)	29(1)	4(1)	16(1)
C(6)	47(1)	45(1)	45(1)	11(1)	5(1)	9(1)
C(7)	44(1)	49(1)	51(1)	9(1)	7(1)	10(1)
C(8)	49(1)	66(2)	68(2)	25(1)	1(1)	3(1)
C(9)	47(1)	63(2)	74(2)	15(1)	6(1)	0(1)
C(10)	51(1)	61(2)	66(2)	10(1)	-1(1)	13(1)
C(11)	53(1)	55(1)	48(1)	11(1)	5(1)	11(1)
C(12)	34(1)	47(1)	51(1)	15(1)	2(1)	3(1)
C(13)	49(1)	52(2)	56(2)	16(1)	1(1)	8(1)
C(14)	56(1)	49(2)	80(2)	18(1)	3(1)	6(1)
C(15)	65(2)	67(2)	89(2)	43(2)	7(1)	11(1)
C(16)	57(1)	81(2)	62(2)	30(2)	3(1)	11(1)
C(17)	36(1)	62(2)	54(1)	17(1)	3(1)	5(1)
C(18)	60(1)	82(2)	53(1)	10(1)	5(1)	9(1)
C(19)	50(1)	76(2)	81(2)	8(1)	-6(1)	-11(1)

Table 5. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for cd25518.

	x	y	z	U(eq)
H(5)	4814	1266	9652	72
H(9A)	8639	6776	9490	76
H(9B)	9250	5640	8537	76
H(11A)	1333	2967	6253	63
H(11B)*	3532	3399	6432	63
H(18A)	2346	3895	3680	100
H(18B)	1177	2910	4445	100
H(18C)	3367	3223	4541	100
H(19A)	-2400	-619	7027	112
H(19B)	-3517	787	6982	112
H(19C)	-2952	321	8038	112
H(3)	-620(20)	-710(20)	8499(12)	64(6)
H(4)	2100(20)	-890(20)	9530(13)	75(7)
H(8)	5860(20)	6050(20)	8054(14)	74(7)
H(10A)	8070(20)	3160(20)	9229(13)	68(7)
H(10B)	7390(20)	4230(20)	10184(14)	75(7)
H(13)	2730(20)	7040(20)	7504(13)	52(6)
H(14)	2880(20)	9540(20)	6821(13)	64(6)
H(15)	2810(30)	9360(20)	5016(15)	84(8)
H(16)	2510(20)	6720(20)	3956(13)	59(6)

Table 6. Torsion angles [deg] for cd25518.

C(11)-O(1)-C(1)-C(6)	105.97(18)
C(11)-O(1)-C(1)-C(2)	-77.44(19)
C(19)-O(2)-C(2)-C(3)	-0.8(3)
C(19)-O(2)-C(2)-C(1)	179.86(16)
C(6)-C(1)-C(2)-O(2)	179.17(16)
O(1)-C(1)-C(2)-O(2)	2.7(2)
C(6)-C(1)-C(2)-C(3)	-0.2(3)
O(1)-C(1)-C(2)-C(3)	-176.69(18)
O(2)-C(2)-C(3)-C(4)	-179.20(19)
C(1)-C(2)-C(3)-C(4)	0.1(3)
C(2)-C(3)-C(4)-C(5)	0.6(3)
C(3)-C(4)-C(5)-C(6)	-1.3(3)
O(1)-C(1)-C(6)-C(5)	176.09(16)
C(2)-C(1)-C(6)-C(5)	-0.4(3)
O(1)-C(1)-C(6)-C(7)	-3.8(3)
C(2)-C(1)-C(6)-C(7)	179.69(16)
C(4)-C(5)-C(6)-C(1)	1.2(3)
C(4)-C(5)-C(6)-C(7)	-178.97(18)
C(1)-C(6)-C(7)-C(8)	-1.9(4)
C(5)-C(6)-C(7)-C(8)	178.3(2)
C(1)-C(6)-C(7)-C(10)	178.8(2)
C(5)-C(6)-C(7)-C(10)	-1.1(3)
C(6)-C(7)-C(8)-C(9)	-178.5(2)
C(10)-C(7)-C(8)-C(9)	0.97(19)
C(7)-C(8)-C(9)-C(10)	-0.95(19)
C(8)-C(7)-C(10)-C(9)	-0.94(19)
C(6)-C(7)-C(10)-C(9)	178.63(19)
C(8)-C(9)-C(10)-C(7)	0.84(17)
C(1)-O(1)-C(11)-C(12)	-177.72(14)
O(1)-C(11)-C(12)-C(13)	5.3(2)
O(1)-C(11)-C(12)-C(17)	-174.84(15)
C(17)-C(12)-C(13)-C(14)	-0.6(3)
C(11)-C(12)-C(13)-C(14)	179.25(18)
C(12)-C(13)-C(14)-C(15)	0.6(3)
C(13)-C(14)-C(15)-C(16)	0.1(3)
C(14)-C(15)-C(16)-C(17)	-0.9(3)
C(15)-C(16)-C(17)-C(12)	0.8(3)
C(15)-C(16)-C(17)-C(18)	-179.35(19)
C(13)-C(12)-C(17)-C(16)	-0.1(3)
C(11)-C(12)-C(17)-C(16)	-179.96(17)
C(13)-C(12)-C(17)-C(18)	-179.93(16)
C(11)-C(12)-C(17)-C(18)	0.2(3)

Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for cd25518 [Å and deg.].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
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