Supporting Information

Reactions of Methylenecyclopropanes with Phenylsulfenyl Chloride and Phenylselenenyl Chloride

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¹ H NMR charts of compounds	S2-S22
X-ray data of 5a .	S23-S31
X-ray data of 9a .	S32-S40

Experimental Procedures

General Methods. Melting points are uncorrected. ^1H and ^{13}C NMR spectra were recorded at 300 and 75 MHz respectively. 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 reactions were monitored by TLC with Huanghai GF254 silica gel coated plates. Flash column chromatography was carried out using 300-400 mesh silica gel and 200-300 mesh Al_2O_3 at increased pressure.

Typical reaction procedure for the reaction of MCPs 1 with phenylselenenyl chloride: To a solution of MCP **1a** (62 mg, 0.3 mmol) in dichloromethane (1.0 mL) was added phenylselenenyl chloride (57 mg, 0.3 mmol) in dichloromethane (1.0 mL) at 0 °C, the reaction mixture was stirred for 2 min (monitored by TLC). After the starting materials (MCPs **1**) were consumed, the solvent was removed under reduced pressure and the residue was subjected to a flash column chromatography to give the desired product **5a** (53 mg, 49%) and **6a** (44 mg, 37%) as white solids.

Compound **3a**: a white solid, m.p. 104-106 °C; IR (KBr) ν 3055, 1581, 1475, 1441, 1024, 700 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz, TMS) δ 2.74 (2H, t, $J = 7.2$ Hz, CH_2), 3.66 (2H, t, $J = 7.2$ Hz, CH_2), 7.17-7.28 (10H, m, Ar), 7.30-7.36 (5H, m, Ar); ^{13}C NMR (CDCl_3 , 75 MHz, TMS) δ 35.6, 43.1, 126.6, 127.4, 127.9, 128.4, 129.0 (2C), 129.2, 129.2, 129.5, 130.1, 134.9, 141.4, 142.0, 150.1; MS (EI) m/e 350 (M^+ , 100), 287 ($\text{M}^+ - 63$, 10.30), 205 ($\text{M}^+ - 145$, 40.99), 192 ($\text{M}^+ - 158$, 63.40), 165 ($\text{M}^+ - 185$, 25.07); HRMS (EI) Calcd. for $\text{C}_{22}\text{H}_{19}\text{ClS}$ (M^+) requires 350.0896, Found: 350.0864.

Compound **4a**: a colorless liquid. IR (KBr) ν 3058, 1599, 1492, 1222, 1025, 699 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz, TMS) δ 3.08 (2H, d, $J = 1.5$ Hz, CH_2), 5.84 (1H, t, $J = 1.5$ Hz, $=\text{CH}$), 7.25-7.31 (2H, m, Ar), 7.34-7.43 (11H, m, Ar), 7.57-7.60 (2H, m, Ar); ^{13}C NMR (CDCl_3 , 75 MHz, TMS) δ 45.5, 60.8, 126.4, 126.7, 127.6, 128.2, 128.2, 129.2, 131.1, 133.4, 143.9, 145.5; MS (EI) m/e 314 (M^+ , 15.82), 237 ($\text{M}^+ - 77$, 13.05), 205 ($\text{M}^+ - 109$, 100), 178 ($\text{M}^+ - 136$, 24.39), 165 ($\text{M}^+ - 149$, 29.02), 109 ($\text{M}^+ - 205$, 24.75), 84 ($\text{M}^+ - 230$, 46.31), 77 ($\text{M}^+ - 237$, 21.12); Anal. Calcd. for $\text{C}_{22}\text{H}_{18}\text{S}$ (M^+) required 314.1129, Found: 314.1172.

Compound **3b**: a white solid, m.p. 108-110 °C; IR (KBr) ν 3022, 1582, 1475, 1022, 816, 692 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz, TMS) δ 2.30 (3H, s, CH_3), 2.33 (3H, s, CH_3), 2.74 (2H, t, $J = 7.2$ Hz, CH_2), 3.65 (2H, t, $J = 7.2$ Hz, CH_2), 7.07-7.20 (9H, m, Ar), 7.25-7.34 (4H, m, Ar); ^{13}C NMR (CDCl_3 , 75 MHz, TMS) δ 21.2, 21.3, 35.7, 43.2, 126.5, 128.4, 128.6, 128.9, 129.0, 129.0, 129.1, 129.9, 135.1, 137.1, 137.2, 138.7, 139.3, 150.3; MS (EI) m/e 378 (M^+ , 100), 363 ($\text{M}^+ - 15$, 3.13), 315 ($\text{M}^+ - 63$, 9.42), 233 ($\text{M}^+ - 145$, 32.29), 220 ($\text{M}^+ - 158$, 81.64), 205 ($\text{M}^+ - 173$, 28.11), 189 ($\text{M}^+ - 189$, 12.94), 84 ($\text{M}^+ - 294$, 32.03); HRMS (MALDI) Calcd. for $\text{C}_{24}\text{H}_{24}\text{ClS}^+1$ ($\text{M}^+ + 1$) requires 379.1297, Found: 379.1282.

Compound **4b**: a colorless liquid. IR (KBr) ν 3052, 1575, 1509, 1023, 810, 691 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz, TMS) δ 2.34 (6H, s, CH_3), 3.04 (2H, d, $J = 1.2$ Hz, CH_2), 5.81 (1H, t, $J = 1.2$ Hz, $=\text{CH}$), 7.13 (4H, d, $J = 7.8$ Hz, Ar), 7.25 (4H, d, $J = 7.8$ Hz, Ar), 7.32-7.35 (3H, m, Ar), 7.53-7.54 (2H, m, Ar); ^{13}C NMR (CDCl_3 , 75 MHz, TMS) δ 21.0, 45.6, 60.3, 126.3, 127.4, 128.1, 128.8, 129.2, 131.2, 133.4, 135.8, 141.1, 145.7; MS (EI) m/e 342 (M^+ , 41.42), 327 ($\text{M}^+ - 15$, 2.70), 265 ($\text{M}^+ - 77$, 13.05), 233 ($\text{M}^+ - 109$, 100), 218 ($\text{M}^+ - 124$, 67.34), 202

(M^+ -140, 35.89), 178 (M^+ -164, 25.61), 129 (M^+ -213, 45.79); Calcd. for $C_{24}H_{22}S$ (M^+) requires 342.1442, Found: 342.1404.

Compound **3c**: a white solid, m.p. 102-104 °C; IR (KBr) ν 3032, 1605, 1440, 1218, 1033, 832, 692 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz, TMS) δ 2.76 (2H, t, $J = 7.2$ Hz, CH_2), 3.66 (2H, t, $J = 7.2$ Hz, CH_2), 3.75 (3H, s, OCH_3), 3.79 (3H, s, OCH_3), 6.81 (2H, d, $J = 8.4$ Hz, Ar), 6.87 (2H, d, $J = 8.4$ Hz, Ar), 7.13 (2H, d, $J = 8.4$ Hz, Ar), 7.19 (2H, d, $J = 8.4$ Hz, Ar), 7.20-7.34 (5H, m, Ar); ^{13}C NMR (CDCl_3 , 75 MHz, TMS) δ 35.9, 43.3, 55.1, 55.2, 113.1, 113.7, 126.4, 127.7, 129.0, 129.8, 130.4, 130.7, 134.1, 134.6, 135.3, 149.7, 158.8, 158.9; MS (EI) m/e 410 (M^+ , 70.90), 379 (M^+ -31, 3.10), 347 (M^+ -63, 8.17), 270 (M^+ -140, 13.13), 252 (M^+ -158, 100), 237 (M^+ -173, 24.51), 221 (M^+ -189, 32.60); HRMS (EI) Calcd. for $C_{24}H_{23}\text{ClO}_2\text{S}$ (M^+) requires 410.1107, Found: 410.1074.

Compound **3d**: a white solid, m.p. 96-98 °C; IR (KBr) ν 3060, 1583, 1488, 1015, 909, 826, 692 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz, TMS) δ 2.72 (2H, t, J = 6.9 Hz, CH_2), 3.66 (2H, t, J = 6.9 Hz, CH_2), 7.14-7.17 (4H, m, Ar), 7.24-7.34 (9H, m, Ar); ^{13}C NMR (CDCl_3 , 75 MHz, TMS) δ 36.4, 42.9, 127.1, 128.3, 128.7, 129.2, 130.5, 130.5, 130.7, 131.3, 133.5, 133.6, 134.2, 139.39, 139.9, 147.0; MS (EI) m/e 418 (M^+ , 24.37), 334 (M^+ -84, 4.67), 260 (M^+ -158, 7.86), 238 (M^+ -180, 14.82), 225 (M^+ -193, 21.45), 205 (M^+ -213, 53.09), 189 (M^+ -229, 18.86), 109 (M^+ -309, 20.38), 84 (M^+ -334, 100); HRMS (EI) Calcd. for $\text{C}_{22}\text{H}_{17}\text{Cl}_3\text{S}$ (M^+) requires 418.0117, Found: 418.0096.

Compound **4d**: a colorless liquid. IR (KBr) ν 3061, 1575, 1439, 1096, 1015, 817, 690 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz, TMS) δ 3.02 (2H, d, J = 0.9 Hz, CH_2), 5.83 (1H, t, J = 0.9 Hz, $=\text{CH}$), 7.21-7.32 (7H, m, Ar), 7.33-7.37 (4H, m, Ar), 7.48-7.53 (2H, m, Ar); ^{13}C NMR (CDCl_3 , 75 MHz, TMS) δ 45.5, 59.8, 127.1, 128.4, 128.5, 128.9, 129.3, 130.4, 132.5, 133.5, 142.0, 145.2; MS (EI) m/e 382 (M^+ , 25.97), 347 (M^+ -35, 5.23), 273 (M^+ -109, 55.57), 238 (M^+ -144, 89.07), 202 (M^+ -180, 100), 178 (M^+ -204, 32.32), 134 (M^+ -248, 35.52); HRMS (EI) Calcd. for $\text{C}_{22}\text{H}_{16}\text{Cl}_2\text{S}$ (M^+) requires 382.0350, Found: 382.0395.

3e

Compound **4e**: a colorless liquid. IR (KBr) ν 3061, 1601, 1440, 1160, 1015, 829, 691 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz, TMS) δ 3.03 (2H, s, CH_2), 5.83 (1H, s, $=\text{CH}$), 6.98-7.04 (4H, m, Ar), 7.23-7.36 (7H, m, Ar), 7.50-7.53 (2H, m, Ar); ^{13}C NMR (CDCl_3 , 75 MHz, TMS) δ 45.8, 59.6, 115.0 (d, $J_{\text{C-F}} = 21.3$ Hz), 126.8, 128.4, 129.1 (d, $J_{\text{C-F}} = 7.7$ Hz), 129.3, 130.5, 133.4, 139.5 (d, $J_{\text{C-F}} = 3.3$ Hz), 145.5, 161.5 (d, $J_{\text{C-F}} = 244.6$ Hz); MS (EI) m/e 350 (M^+ , 53.87), 272 ($\text{M}^+ - 78$, 21.54), 259 ($\text{M}^+ - 91$, 19.26), 241 ($\text{M}^+ - 109$, 100), 220 ($\text{M}^+ - 130$, 71.82), 201 ($\text{M}^+ - 149$, 55.66), 134 ($\text{M}^+ - 216$, 35.14), 101 ($\text{M}^+ - 249$, 18.49); HRMS (EI) Calcd. for $\text{C}_{22}\text{H}_{16}\text{F}_2\text{S}$ (M^+) requires 350.0941, Found: 350.0961.

Compound **5a**: a white solid, m.p. 74-75 $^{\circ}\text{C}$; IR (KBr) ν 3056, 1578, 1492, 1218, 1022, 756, 698 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz, TMS) δ 3.18 (2H, d, $J = 0.9$ Hz, CH_2), 6.13 (1H, t, $J = 0.9$ Hz, $=\text{CH}$), 7.23-7.35 (13H, m, Ar), 7.59-7.62 (2H, m, Ar); ^{13}C NMR (CDCl_3 , 75 MHz, TMS) δ 46.9, 61.8, 126.3, 127.5, 127.5, 128.0, 128.1, 129.3, 133.8, 134.8, 141.8, 144.1; MS (EI) m/e 362 (M^+ , 10.92), 281 ($\text{M}^+ - 81$, 12.60), 205 ($\text{M}^+ - 157$, 100), 190 ($\text{M}^+ - 172$, 20.45), 178 ($\text{M}^+ - 184$, 22.73), 165 ($\text{M}^+ - 197$, 18.97), 127 ($\text{M}^+ - 235$, 10.41); HRMS (EI) Calcd. for $\text{C}_{22}\text{H}_{18}\text{Se}$ (M^+) requires 362.0574, Found: 362.0534.

Compound **6a**: a white solid, m.p. 106-108 °C; IR (KBr) ν 3056, 1577, 1441, 1022, 909, 738, 700 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz, TMS) δ 2.73 (2H, t, $J = 7.2$ Hz, CH_2), 3.61 (2H, t, $J = 7.2$ Hz, CH_2), 7.19-7.34 (13H, m, Ar), 7.47-7.51 (2H, m, Ar); ^{13}C NMR (CDCl_3 , 75 MHz, TMS) δ 36.9, 43.7, 127.3, 127.5, 127.6, 128.0, 128.4, 129.0, 129.1, 129.2, 129.5, 129.9, 133.6, 141.4, 143.2, 148.9; MS (EI) m/e 398 (M^+ , 100), 335 ($\text{M}^+ - 63$, 7.38), 269 ($\text{M}^+ - 129$, 4.64), 205 ($\text{M}^+ - 193$, 75.22), 191 ($\text{M}^+ - 207$, 38.56), 178 ($\text{M}^+ - 220$, 32.25), 165 ($\text{M}^+ - 233$, 28.18); HRMS (EI) Calcd. for $\text{C}_{22}\text{H}_{19}\text{ClSe}$ (M^+) requires 398.0340, Found: 398.0302.

Compound **6b**: a white solid, m.p. 112-114 °C; IR (KBr) ν 3047, 1578, 1508, 1438, 1265, 1021, 816, 692 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz, TMS) δ 2.31 (6H, s, CH_3), 2.73 (2H, t, $J = 7.2$ Hz, CH_2), 3.59 (2H, t, $J = 7.2$ Hz, CH_2), 7.06-7.13 (8H, m, Ar), 7.24-7.26 (3H, m, Ar), 7.47-7.50 (2H, m, Ar); ^{13}C NMR (CDCl_3 , 75 MHz, TMS) δ 21.2, 21.3, 36.9, 43.8, 127.2, 127.4, 128.7, 128.9, 128.9, 129.0, 129.2, 130.1, 133.5, 136.9, 137.3, 138.6, 140.5, 149.0; MS (EI) m/e 426 (M^+ , 48.23), 363 ($\text{M}^+ - 63$, 3.13), 233 ($\text{M}^+ - 193$, 35.15), 218 ($\text{M}^+ - 208$, 57.20), 205 ($\text{M}^+ - 221$, 36.14), 189 ($\text{M}^+ - 237$, 19.00), 84 ($\text{M}^+ - 342$, 100); HRMS (MALDI) Calcd. for $\text{C}_{24}\text{H}_{24}\text{ClSe}^{+1}$ ($\text{M}^+ + 1$) requires 427.0722, Found: 427.0726.

Compound **6c**: a white solid, m.p. 104-106 °C; IR (KBr) ν 3031, 1605, 1508, 1439, 1246, 1033, 833, 693 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz, TMS) δ 2.75 (2H, t, $J = 7.2$ Hz, CH_2), 3.61 (2H, t, $J = 7.2$ Hz, CH_2), 3.78 (3H, s, OCH_3), 3.79 (3H, s, OCH_3), 6.81-6.87 (4H, m, Ar), 7.09-7.14 (4H, m, Ar), 7.25-7.28 (3H, m, Ar), 7.47-7.50 (2H, m, Ar); ^{13}C NMR (CDCl_3 , 75 MHz, TMS) δ 37.1, 43.9, 55.1, 55.2, 113.2, 113.6, 127.4, 128.0, 129.2, 130.2, 130.4, 130.5, 133.4, 134.0, 135.9, 148.3, 158.7, 158.9; MS (EI) m/e 458 (M^+ , 66.50), 427 ($\text{M}^+ - 31$, 6.11), 301 ($\text{M}^+ - 157$, 9.63), 252 ($\text{M}^+ - 206$, 100), 237 ($\text{M}^+ - 221$, 40.60), 226

(M^+ -232, 42.46), 157 (M^+ -301, 34.66), 91 (M^+ -367, 56.65); HRMS (MALDI) Calcd. for $C_{24}H_{24}ClO_2Se^{+1}$ (M^++1) requires 459.0622, Found: 459.0625.

Compound **5d**: a white solid, m.p. 70-72 °C; IR (KBr) ν 3058, 1577, 1489, 1218, 1096, 1014, 817, 690 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz, TMS) δ 3.12 (2H, s, CH_2), 6.14 (1H, s, $=\text{CH}$), 7.21-7.32 (11H, m, Ar), 7.54-7.57 (2H, m, Ar); ^{13}C NMR (CDCl_3 , 75 MHz, TMS) δ 46.9, 60.8, 126.7, 128.2, 128.4, 128.8, 129.3, 132.4, 134.3, 134.8, 141.3, 142.1; MS (EI) m/e 430 (M^+ , 7.13), 346 (M^+ -84, 29.27), 314 (M^+ -116, 30.20), 295 (M^+ -135, 34.50), 261 (M^+ -169, 50.91), 238 (M^+ -192, 78.91), 202 (M^+ -228, 100), 84 (M^+ -346, 91.93); HRMS (MALDI) Calcd. for $\text{C}_{22}\text{H}_{17}\text{Cl}_2\text{Se}^{+1}$ (M^++1) requires 430.9851, Found: 430.9867.

Compound **6d**: a white solid, m.p. 108-110 °C; IR (KBr) ν 3058, 1578, 1488, 1090, 1015, 836, 738, 692 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz, TMS) δ 2.70 (2H, t, $J = 7.2$ Hz, CH_2), 3.60 (2H, t, $J = 7.2$ Hz, CH_2), 7.10-7.14 (4H, m, Ar), 7.27-7.32 (7H, m, Ar), 7.45-7.48 (2H, m, Ar); ^{13}C NMR (CDCl_3 , 75 MHz, TMS) δ 36.7, 43.6, 127.9, 128.4, 128.7, 129.3, 129.4, 130.5, 130.52, 131.3, 133.4, 133.6, 133.8, 139.4, 141.1, 146.0; MS (EI) m/e 466 (M^+ , 22.96), 273 ($\text{M}^+ - 193$, 8.60), 238 ($\text{M}^+ - 228$, 45.54), 225 ($\text{M}^+ - 241$, 19.10), 202 ($\text{M}^+ - 264$, 43.55), 189 ($\text{M}^+ - 277$, 18.70), 137 ($\text{M}^+ - 329$, 11.71), 84 ($\text{M}^+ - 382$, 100); HRMS (MALDI) Calcd. for $\text{C}_{22}\text{H}_{18}\text{Cl}_3\text{Se}^{+1}$ ($\text{M}^+ + 1$) requires 466.9614, Found: 466.9634.

Compound **5e**: a white solid, m.p. 68-70 °C; IR (KBr) ν 3059, 1601, 1505, 1232, 828, 739, 691 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz, TMS) δ 3.13 (2H, s, CH_2), 6.14 (1H, s, $=\text{CH}$), 6.96-7.02 (4H, m, Ar), 7.24-7.31 (7H, m, Ar), 7.55-7.58 (2H, m, Ar); ^{13}C NMR (CDCl_3 , 75 MHz, TMS) δ 47.2, 60.7, 115.0 (d, $J_{\text{C-F}} = 21.2$ Hz), 126.9, 128.2, 129.0 (d, $J_{\text{C-F}} = 7.7$ Hz), 129.3, 134.1, 134.8, 139.6 (d, $J_{\text{C-F}} = 3.3$ Hz), 142.3, 161.4 (d, $J_{\text{C-F}} = 244.5$ Hz); MS (EI) m/e 398 (M^+ , 10.86), 278 ($\text{M}^+ - 120$, 35.00), 241 ($\text{M}^+ - 157$, 66.19), 229 ($\text{M}^+ - 169$, 100),

220 ($M^+ - 178$, 86.83), 201 ($M^+ - 197$, 45.60), 133 ($M^+ - 265$, 70.95), 101 ($M^+ - 297$, 27.96); HRMS (EI) Calcd. for $C_{22}H_{16}F_2Se$ (M^+) requires 398.0385, Found: 398.0389.

Compound **6e**: a white solid, m.p. 92-94 °C; IR (KBr) ν 3056, 1600, 1505, 1265, 1156, 836, 739, 692 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz, TMS) δ 2.71 (2H, t, $J = 6.9$ Hz, CH_2), 3.61 (2H, t, $J = 6.9$ Hz, CH_2), 6.96-7.05 (4H, m, Ar), 7.13-7.19 (4H, m, Ar), 7.27-7.29 (3H, m, Ar), 7.46-7.49 (2H, m, Ar); ^{13}C NMR (CDCl_3 , 75 MHz, TMS) δ 36.7, 43.6, 115.0 (d, $J_{\text{C-F}} = 21.4$ Hz), 115.3 (d, $J_{\text{C-F}} = 21.7$ Hz), 127.7, 129.2, 129.5, 130.5, 130.8 (2C, d, $J_{\text{C-F}} = 8.0$ Hz), 133.6, 137.1 (d, $J_{\text{C-F}} = 3.5$ Hz), 138.9 (d, $J_{\text{C-F}} = 4.0$ Hz), 146.4, 161.9 (d, $J_{\text{C-F}} = 246.4$ Hz), 162.0 (d, $J_{\text{C-F}} = 246.4$ Hz); MS (EI) m/e 434 (M^+ , 85.54), 371 ($M^+ - 63$, 7.54), 241 ($M^+ - 193$, 99.72), 228 ($M^+ - 206$, 84.57), 220 ($M^+ - 214$, 100), 201 ($M^+ - 233$, 43.73), 109 ($M^+ - 325$, 49.05); HRMS (MALDI) Calcd. for $C_{22}H_{18}\text{ClF}_2\text{Se}^{+1}$ ($M^+ + 1$) required 435.0225, Found: 435.0225.

Compound **7a**: a colorless liquid. IR (KBr) ν 3056, 1583, 1479, 1439, 1027, 906, 775, 691 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz, TMS) δ 1.30 (2H, d, $J = 1.8$ Hz, CH_2), 1.32 (2H, d, $J = 1.8$ Hz, CH_2), 5.03 (1H, d, $J = 0.9$ Hz, $=\text{CH}_2$), 5.33 (1H, d, $J = 0.9$ Hz, $=\text{CH}_2$), 7.20-7.36 (8H, m, Ar), 7.54-7.57 (2H, m, Ar); ^{13}C NMR (CDCl_3 , 75 MHz, TMS) δ 16.3, 30.9, 114.5, 126.5, 127.1, 127.7, 128.0, 128.5, 130.7, 136.3, 139.5, 148.3; MS (EI) m/e 252 (M^+ , 100), 219 ($\text{M}^+ - 33$, 19.45), 161 ($\text{M}^+ - 91$, 23.57), 141 ($\text{M}^+ - 111$, 52.96), 128 ($\text{M}^+ - 124$, 94.11), 115 ($\text{M}^+ - 137$, 62.97), 91 ($\text{M}^+ - 161$, 52.48), 77 ($\text{M}^+ - 175$, 52.12); HRMS (EI) Calcd. for $\text{C}_{17}\text{H}_{16}\text{S}$ (M^+) requires 252.0973, Found: 252.1022.

Compound **7b**: a colorless liquid. IR (KBr) ν 3057, 1583, 1479, 1157, 1026, 823, 738, 691 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz, TMS) δ 1.30 (2H, s, CH_2), 1.32 (2H, s, CH_2), 4.99 (1H, d, $J = 0.9$ Hz, $=\text{CH}_2$), 5.31 (1H, d, $J = 0.9$ Hz, $=\text{CH}_2$), 7.15 (2H, d, $J = 7.8$ Hz, Ar), 7.20-7.27 (3H, m, Ar), 7.35-7.36 (2H, m, Ar), 7.46 (2H, d, $J = 7.8$ Hz, Ar); ^{13}C NMR (CDCl_3 , 75 MHz, TMS) δ 16.3, 21.2, 30.9, 113.8, 126.5, 126.9, 128.5, 128.8, 130.7, 136.4, 136.6, 137.5, 148.1; MS (EI) m/e 266 (M^+ , 98.23), 251 ($\text{M}^+ - 15$, 11.01), 218 ($\text{M}^+ - 48$, 28.76), 194 ($\text{M}^+ - 72$, 22.00), 157 ($\text{M}^+ - 109$, 41.45), 142 ($\text{M}^+ - 124$, 100), 128 ($\text{M}^+ - 138$, 53.93), 115 ($\text{M}^+ - 151$, 70.92); HRMS (MALDI) Calcd. for $\text{C}_{18}\text{H}_{19}\text{S}^{+1}$ ($\text{M}^+ + 1$) requires 267.1221, Found: 267.1202.

Compound **8a**: a colorless liquid. IR (KBr) ν 3056, 1611, 1513, 1477, 1149, 1022, 824, 737, 691 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz, TMS) δ 1.26 (2H, s, CH_2), 1.34 (2H, s, CH_2), 2.36 (3H, s, CH_3), 4.89 (1H, s, $=\text{CH}_2$), 5.30 (1H, s, $=\text{CH}_2$), 7.16 (2H, d, $J = 7.8$ Hz, Ar), 7.23-7.25 (3H, m, Ar), 7.45-7.47 (2H, m, Ar), 7.50 (2H, d, $J = 7.8$ Hz, Ar); ^{13}C NMR (CDCl_3 , 75 MHz, TMS) δ 15.9, 21.2, 26.7, 113.0, 126.7, 127.4, 128.6, 128.8, 130.9, 134.0, 136.8, 137.5, 149.1; MS (EI) m/e 314 (M^+ , 30.98), 233 ($\text{M}^+ - 81$, 33.43), 218 ($\text{M}^+ - 96$, 13.54), 157 ($\text{M}^+ - 157$, 64.20), 142 ($\text{M}^+ - 172$, 100), 129 ($\text{M}^+ - 185$, 32.02), 115 ($\text{M}^+ - 199$, 44.64); HRMS (MALDI) Calcd. for $\text{C}_{18}\text{H}_{19}\text{Se}^{+1}$ ($\text{M}^+ + 1$) requires 315.0653, Found: 315.0647.

8b

MCP **1i**: a white solid, m.p. 78-80 °C; IR (KBr) ν 2967, 1581, 1458, 1237, 1132, 1006, 842, 680, 630 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz, TMS) δ 1.19 (2H, t, $J = 7.5$ Hz, CH_2), 1.42 (2H, td, $J = 7.5, 1.8$ Hz, CH_2), 3.85 (3H, s, OCH_3), 3.87 (6H, s, OCH_3), 6.68 (1H, t, $J = 1.8$ Hz, $=\text{CH}$), 6.78 (2H, s, Ar); ^{13}C NMR (CDCl_3 , 75 MHz, TMS) δ 0.5, 3.9, 55.8, 60.8, 103.4, 118.0, 123.6, 134.0, 136.9, 153.1; MS (EI) m/e 220 (M^+ , 14.11), 205 ($\text{M}^+ - 15$, 6.65), 189 ($\text{M}^+ - 31$, 100), 162 ($\text{M}^+ - 58$, 12.83), 145 ($\text{M}^+ - 75$, 16.32), 119 ($\text{M}^+ - 101$, 12.89); Anal. Calcd. for $\text{C}_{13}\text{H}_{16}\text{O}_3$ requires C, 70.91; H, 7.27%. Found: C, 70.89; H, 7.42%.

Compound **9a**: a white solid, m.p. 94-96 °C; IR (KBr) ν 3056, 1579, 1340, 1266, 1130, 1002, 827, 738, 704 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz, TMS) δ 2.58 (2H, t, $J = 3.6$ Hz, CH_2), 2.87 (2H, t, $J = 3.6$ Hz, CH_2), 3.85 (3H, s, OCH_3), 3.86 (6H, s, OCH_3), 6.70 (2H, s, Ar), 7.29-7.31 (3H, m, Ar), 7.54-7.57 (2H, m, Ar); ^{13}C NMR (CDCl_3 , 75 MHz, TMS) δ 28.5, 31.2, 56.0, 60.9, 102.4, 123.9, 127.5, 128.3, 129.1, 130.4, 133.4, 137.6, 145.8, 153.1; MS (EI) m/e 376 (M^+ , 48.69), 295 ($\text{M}^+ - 81$, 6.47), 280 ($\text{M}^+ - 96$, 8.81), 264 ($\text{M}^+ - 112$, 61.86), 204 ($\text{M}^+ - 172$, 54.40), 193 ($\text{M}^+ - 183$, 73.36), 188 ($\text{M}^+ - 188$, 100), 115 ($\text{M}^+ - 261$, 35.00); Anal. Calcd. for $\text{C}_{19}\text{H}_{20}\text{O}_3\text{Se}$ requires C, 60.64; H, 5.32%. Found: C, 60.71; H, 5.40%.

Compound **10a**: a colorless liquid. IR (KBr) ν 3478, 3057, 1592, 1463, 1329, 1233, 1128, 1006, 913, 737, 693 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz, TMS) δ 1.05-1.18 (4H, m, CH_2), 2.70 (1H, s, OH), 3.80 (6H, s, OCH_3), 3.81 (3H, s, OCH_3), 4.45 (1H, s, OCH), 6.57 (2H, s, Ar), 7.19-7.21 (3H, m, Ar), 7.36-7.39 (2H, m, Ar); ^{13}C NMR (CDCl_3 , 75 MHz, TMS) δ 12.7, 14.6, 30.0, 55.9, 60.7, 78.2, 103.7, 127.0, 128.7, 130.0, 132.5, 136.8, 137.2, 152.7; MS (EI) m/e 394 (M^+ , 58.87), 237 ($\text{M}^+ - 157$, 15.38), 205 ($\text{M}^+ - 189$, 20.02), 197 ($\text{M}^+ - 197$, 100), 181 ($\text{M}^+ - 213$, 32.77), 169 ($\text{M}^+ - 225$, 97.43), 154 ($\text{M}^+ - 240$, 43.10); HRMS (MALDI) Calcd. for $\text{C}_{19}\text{H}_{22}\text{O}_4\text{SeNa}^{+1}$ ($\text{M}^+ + 23$) requires 417.0561, Found: 417.0576.

Compound **9b**: a white solid, m.p. 60-62 °C; IR (KBr) ν 3057, 1601, 1506, 1248, 1175, 1028, 832, 738, 691 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz, TMS) δ 2.58 (2H, t, $J = 3.6$ Hz, CH_2), 2.87 (2H, t, $J = 3.6$ Hz, CH_2), 3.82 (3H, s, OCH_3), 6.90 (2H, d, $J = 8.7$ Hz, Ar), 7.27-7.30 (3H, m, Ar), 7.44 (2H, d, $J = 8.7$ Hz, Ar), 7.54-7.56 (2H, m, Ar); ^{13}C NMR (CDCl_3 , 75 MHz, TMS) δ 28.5, 31.3, 55.3, 113.7, 113.9, 121.2, 126.8, 127.3, 127.7, 129.1, 133.4, 146.3, 159.0; MS (EI) m/e 316 (M^+ , 92.97), 235 ($\text{M}^+ - 81$, 41.88), 220 ($\text{M}^+ - 96$, 29.75), 204 ($\text{M}^+ - 112$, 17.82), 144 ($\text{M}^+ - 172$, 67.57), 133 ($\text{M}^+ - 183$, 100), 128 ($\text{M}^+ - 188$, 52.73), 115 ($\text{M}^+ - 201$, 82.43); Anal. Calcd. for $\text{C}_{19}\text{H}_{20}\text{O}_3\text{Se}$ requires C, 64.76; H, 5.08%. Found: C, 60.53; H, 5.22%.

Compound **10b**: a colorless liquid. IR (KBr) ν 3447, 3059, 1611, 1512, 1248, 1174, 1034, 832, 737, 692 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz, TMS) δ 0.94-1.08 (4H, m, CH_2), 2.61 (1H, s, OH), 3.77 (3H, s, OCH_3), 4.57 (1H, s, OCH), 6.81 (2H, d, $J = 8.4$ Hz, Ar), 7.21-7.23 (3H, m, Ar), 7.25 (2H, d, $J = 8.4$ Hz, Ar), 7.43-7.46 (2H, m, Ar); ^{13}C NMR (CDCl_3 , 75 MHz, TMS) δ 11.7, 14.3, 30.2, 55.2, 77.3, 113.3, 127.0, 127.9, 128.9, 130.1, 132.6, 132.9, 159.0; MS (EI) m/e 334 (M^+ , 25.85), 198 ($\text{M}^+ - 136$, 45.36), 183 ($\text{M}^+ - 151$, 14.42), 157 ($\text{M}^+ - 177$, 13.14), 137 ($\text{M}^+ - 197$, 100), 117 ($\text{M}^+ - 217$, 48.35), 109 ($\text{M}^+ - 225$, 27.63); HRMS (MALDI) Calcd. for $\text{C}_{17}\text{H}_{19}\text{O}_2\text{Se}^{+1}$ ($\text{M}^+ + 1$) requires 335.0536, Found: 335.0545.

Compound **9c** a colorless liquid. IR (KBr) ν 3057, 1600, 1578, 1476, 1269, 1022, 905, 739, 691 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz, TMS) δ 2.56 (2H, t, $J = 3.0$ Hz, CH_2), 2.86 (2H, t, $J = 3.0$ Hz, CH_2), 7.24-7.40 (6H, m, Ar), 7.44-7.50 (2H, m, Ar), 7.56-7.59 (2H, m, Ar); MS (EI) m/e 286 (M^+ , 44.07), 205 ($\text{M}^+ - 81$, 40.92), 180 ($\text{M}^+ - 106$, 69.64), 128 ($\text{M}^+ - 158$, 100), 115 ($\text{M}^+ - 171$, 19.09), 105 ($\text{M}^+ - 181$, 21.72), 77 ($\text{M}^+ - 209$, 43.49); HRMS (MALDI) Calcd. for $\text{C}_{16}\text{H}_{15}\text{Se}^{+1}$ ($\text{M}^+ + 1$) requires 287.0341, Found: 287.0334.

Compound **10c**: a colorless liquid. IR (KBr) ν 3430, 3058, 1578, 1477, 1239, 1040, 736, 699 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz, TMS) δ 0.96-1.15 (4H, m, CH_2), 2.60 (1H, s, OH), 4.61 (1H, s, OCH), 7.21-7.34 (8H, m, Ar), 7.43-7.46 (2H, m, Ar); ^{13}C NMR (CDCl_3 , 75 MHz, TMS) δ 11.8, 14.7, 30.2, 77.9, 126.8, 127.1, 127.7, 128.0, 128.9, 130.0, 132.7, 140.7; MS (EI) m/e 304 (M^+ , 50.56), 198 ($\text{M}^+ - 106$, 25.70), 183 ($\text{M}^+ - 121$, 19.68), 157 ($\text{M}^+ - 147$, 31.43), 129 ($\text{M}^+ - 175$, 36.20), 117 ($\text{M}^+ - 187$, 79.50), 105 ($\text{M}^+ - 199$, 91.69), 77 ($\text{M}^+ - 227$, 100); HRMS (MALDI) Calcd. for $\text{C}_{16}\text{H}_{17}\text{OSe}^{+1}$ ($\text{M}^+ + 1$) requires 305.0421, Found: 305.0439.

10c

9d

Compound **9d**: a colorless liquid. IR (KBr) ν 3038, 1576, 1475, 1396, 1306, 1217, 1069, 902, 735, 688 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz, TMS) δ 2.53 (2H, t, $J = 3.0$ Hz, CH_2), 2.83 (2H, t, $J = 3.0$ Hz, CH_2), 7.24-7.31 (5H, m, Ar), 7.47 (2H, d, $J = 8.4$ Hz, Ar), 7.55-7.59 (2H, m, Ar); MS (EI) m/e 364 (M^+ , 30.57), 204 ($\text{M}^+ - 160$, 59.08), 183 ($\text{M}^+ - 181$, 11.37), 158 ($\text{M}^+ - 206$, 10.39), 128 ($\text{M}^+ - 236$, 100), 102 ($\text{M}^+ - 262$, 21.73), 77 ($\text{M}^+ - 287$, 16.78); HRMS (MALDI) Calcd. for $\text{C}_{16}\text{H}_{14}\text{BrSe}^{+1}$ ($\text{M}^+ + 1$) requires 364.9452, Found: 364.9439.

10d

Compound **10d**: a colorless liquid. IR (KBr) ν 3444, 3057, 1578, 1478, 1401, 1239, 1071, 909, 829, 736, 691 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz, TMS) δ 0.96-1.12 (4H, m, CH_2), 2.57 (1H, d, $J = 4.8$ Hz, OH), 4.52 (1H, d, $J = 4.8$ Hz, OCH), 7.20-7.27 (5H, m, Ar), 7.38-7.44 (4H, m, Ar); ^{13}C NMR (CDCl_3 , 75 MHz, TMS) δ 12.2, 14.4, 30.2, 77.5, 121.6, 127.3, 128.5, 129.0, 129.6, 131.0, 133.0, 139.8; MS (EI) m/e 382 (M^+ , 35.99), 278 ($\text{M}^+ - 104$, 11.95), 197 ($\text{M}^+ - 185$, 24.60), 185 ($\text{M}^+ - 197$, 64.63), 157 ($\text{M}^+ - 225$, 49.61), 145 ($\text{M}^+ - 237$, 42.10), 117 ($\text{M}^+ - 265$, 100); HRMS (FT) Calcd. for $\text{C}_{16}\text{H}_{16}\text{BrO}^{74}\text{Se}$ (M^+) requires 376.9597, Found: 376.9604.

Figure 1. The X-ray Crystal Structure of **5a**.

Table 1. Crystal data and structure refinement for cd23463.

Identification code	cd23463
Empirical formula	C22 H18 Se
Formula weight	361.32
Temperature	293(2) K
Wavelength	0.71073 Å
Crystal system, space group	Monoclinic, P2(1)/n
Unit cell dimensions	a = 11.9201(12) Å alpha = 90 deg. b = 9.1090(10) Å beta = 105.248(2) deg. c = 16.1219(16) Å gamma = 90 deg.
Volume	1688.9(3) Å ³
Z, Calculated density	4, 1.421 Mg/m ³
Absorption coefficient	2.220 mm ⁻¹
F(000)	736
Crystal size	0.514 x 0.323 x 0.065 mm
Theta range for data collection	2.46 to 28.32 deg.
Limiting indices	-15<=h<=10, -11<=k<=11, -21<=l<=19
Reflections collected / unique	10045 / 3892 [R(int) = 0.0953]
Completeness to theta = 28.32	92.5 %
Absorption correction	Sadabs
Max. and min. transmission	1.00000 and 0.55590
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	3892 / 5 / 281
Goodness-of-fit on F ²	0.914
Final R indices [I>2sigma(I)]	R1 = 0.0459, wR2 = 0.1012
R indices (all data)	R1 = 0.0621, wR2 = 0.1053
Extinction coefficient	0.0022(9)
Largest diff. peak and hole	0.744 and -0.744 e.Å ⁻³

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for cd23463. U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)
Se	1776(1)	7392(1)	2075(1)	48(1)
C(1)	739(2)	8921(3)	1568(2)	37(1)
C(2)	848(2)	10265(3)	1274(2)	44(1)
C(3)	-414(3)	10594(3)	1069(2)	44(1)
C(4)	-578(2)	9004(3)	1409(2)	36(1)
C(5)	-959(2)	8949(3)	2246(2)	36(1)
C(6)	-561(3)	9992(3)	2886(2)	50(1)
C(7)	-880(3)	9941(4)	3647(2)	61(1)
C(8)	-1582(3)	8833(4)	3797(2)	60(1)
C(9)	-1969(3)	7789(4)	3176(2)	61(1)
C(10)	-1669(3)	7841(3)	2406(2)	48(1)
C(11)	-1300(2)	8012(3)	725(2)	39(1)
C(12)	-2415(3)	8429(4)	287(2)	50(1)
C(13)	-3105(3)	7553(4)	-349(2)	61(1)
C(14)	-2695(3)	6240(5)	-551(2)	64(1)
C(15)	-1605(3)	5810(4)	-127(2)	66(1)
C(16)	-906(3)	6679(4)	508(2)	53(1)
C(17)	3170(2)	8173(3)	1861(2)	42(1)
C(18)	3335(3)	8122(4)	1047(2)	59(1)
C(19)	4356(4)	8614(5)	914(3)	72(1)
C(20)	5213(3)	9150(4)	1576(3)	77(1)
C(21)	5055(3)	9217(4)	2394(3)	72(1)
C(22)	4032(3)	8722(3)	2531(2)	53(1)

Table 3. Bond lengths [Å] and angles [deg] for cd23463.

Se-C(1)	1.899(3)
Se-C(17)	1.920(3)
C(1)-C(2)	1.332(4)
C(1)-C(4)	1.525(3)
C(2)-C(3)	1.483(4)
C(2)-H(1)	0.910(17)
C(3)-C(4)	1.579(4)
C(3)-H(2)	0.95(3)
C(3)-H(3)	0.97(3)
C(4)-C(11)	1.508(4)
C(4)-C(5)	1.533(3)
C(5)-C(10)	1.385(4)
C(5)-C(6)	1.391(4)
C(6)-C(7)	1.377(4)
C(6)-H(4)	0.91(3)
C(7)-C(8)	1.373(5)
C(7)-H(5)	0.98(4)
C(8)-C(9)	1.370(5)
C(8)-H(6)	0.94(3)
C(9)-C(10)	1.380(5)
C(9)-H(7)	0.90(4)
C(10)-H(8)	0.92(3)
C(11)-C(16)	1.380(4)
C(11)-C(12)	1.383(4)
C(12)-C(13)	1.386(5)
C(12)-H(9)	0.87(3)
C(13)-C(14)	1.364(5)
C(13)-H(10)	0.99(4)
C(14)-C(15)	1.356(5)
C(14)-H(11)	0.94(3)
C(15)-C(16)	1.386(4)
C(15)-H(12)	0.910(18)
C(16)-H(13)	0.902(17)
C(17)-C(18)	1.378(4)
C(17)-C(22)	1.375(4)
C(18)-C(19)	1.365(5)
C(18)-H(14)	0.910(18)
C(19)-C(20)	1.359(6)
C(19)-H(15)	0.85(3)
C(20)-C(21)	1.382(6)
C(20)-H(16)	0.901(18)
C(21)-C(22)	1.371(5)
C(21)-H(17)	0.97(4)
C(22)-H(18)	0.86(3)
C(1)-Se-C(17)	98.45(11)
C(2)-C(1)-C(4)	95.0(2)
C(2)-C(1)-Se	135.6(2)
C(4)-C(1)-Se	129.37(19)
C(1)-C(2)-C(3)	94.4(2)
C(1)-C(2)-H(1)	132.4(18)
C(3)-C(2)-H(1)	133.2(18)
C(2)-C(3)-C(4)	87.0(2)
C(2)-C(3)-H(2)	117.6(17)
C(4)-C(3)-H(2)	116.4(17)
C(2)-C(3)-H(3)	113.9(16)
C(4)-C(3)-H(3)	113.0(17)
H(2)-C(3)-H(3)	108(2)
C(11)-C(4)-C(1)	117.2(2)
C(11)-C(4)-C(5)	112.3(2)
C(1)-C(4)-C(5)	112.3(2)
C(11)-C(4)-C(3)	113.3(2)
C(1)-C(4)-C(3)	83.6(2)
C(5)-C(4)-C(3)	115.4(2)

C(10)-C(5)-C(6)	117.7(3)
C(10)-C(5)-C(4)	121.7(2)
C(6)-C(5)-C(4)	120.5(2)
C(7)-C(6)-C(5)	121.1(3)
C(7)-C(6)-H(4)	120(2)
C(5)-C(6)-H(4)	119.3(19)
C(8)-C(7)-C(6)	120.4(3)
C(8)-C(7)-H(5)	118(2)
C(6)-C(7)-H(5)	121(2)
C(9)-C(8)-C(7)	119.0(3)
C(9)-C(8)-H(6)	119(2)
C(7)-C(8)-H(6)	122(2)
C(8)-C(9)-C(10)	121.1(3)
C(8)-C(9)-H(7)	120(2)
C(10)-C(9)-H(7)	119(2)
C(9)-C(10)-C(5)	120.6(3)
C(9)-C(10)-H(8)	120.8(19)
C(5)-C(10)-H(8)	118.6(19)
C(16)-C(11)-C(12)	117.3(3)
C(16)-C(11)-C(4)	123.1(2)
C(12)-C(11)-C(4)	119.7(3)
C(11)-C(12)-C(13)	121.5(3)
C(11)-C(12)-H(9)	117(2)
C(13)-C(12)-H(9)	121(2)
C(14)-C(13)-C(12)	120.1(3)
C(14)-C(13)-H(10)	118(2)
C(12)-C(13)-H(10)	121(2)
C(15)-C(14)-C(13)	119.3(3)
C(15)-C(14)-H(11)	119.6(18)
C(13)-C(14)-H(11)	121.2(18)
C(14)-C(15)-C(16)	121.2(3)
C(14)-C(15)-H(12)	120(2)
C(16)-C(15)-H(12)	119(2)
C(11)-C(16)-C(15)	120.7(3)
C(11)-C(16)-H(13)	125(2)
C(15)-C(16)-H(13)	115(2)
C(18)-C(17)-C(22)	119.7(3)
C(18)-C(17)-Se	120.4(2)
C(22)-C(17)-Se	119.8(2)
C(19)-C(18)-C(17)	119.7(4)
C(19)-C(18)-H(14)	124(2)
C(17)-C(18)-H(14)	117(2)
C(20)-C(19)-C(18)	120.8(4)
C(20)-C(19)-H(15)	122(3)
C(18)-C(19)-H(15)	117(3)
C(19)-C(20)-C(21)	120.1(4)
C(19)-C(20)-H(16)	124(2)
C(21)-C(20)-H(16)	116(2)
C(22)-C(21)-C(20)	119.4(4)
C(22)-C(21)-H(17)	118(2)
C(20)-C(21)-H(17)	123(2)
C(21)-C(22)-C(17)	120.3(4)
C(21)-C(22)-H(18)	118.6(19)
C(17)-C(22)-H(18)	121.0(19)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for cd23463.
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
Se	42(1)	51(1)	49(1)	8(1)	10(1)	0(1)
C(1)	37(1)	47(2)	28(1)	-3(1)	10(1)	-2(1)
C(2)	47(2)	49(2)	40(2)	-2(1)	19(1)	-8(1)
C(3)	54(2)	45(2)	36(2)	4(1)	16(1)	1(1)
C(4)	38(1)	41(1)	30(1)	1(1)	11(1)	0(1)
C(5)	36(1)	44(1)	29(1)	1(1)	10(1)	6(1)
C(6)	62(2)	52(2)	38(2)	-2(1)	18(2)	-9(2)
C(7)	80(2)	66(2)	37(2)	-9(2)	19(2)	3(2)
C(8)	68(2)	80(2)	42(2)	5(2)	28(2)	10(2)
C(9)	62(2)	74(2)	55(2)	9(2)	29(2)	-6(2)
C(10)	50(2)	54(2)	44(2)	-3(1)	20(1)	-7(1)
C(11)	40(1)	52(2)	29(1)	1(1)	13(1)	-5(1)
C(12)	45(2)	61(2)	45(2)	5(2)	12(1)	2(2)
C(13)	46(2)	85(3)	46(2)	8(2)	2(2)	-10(2)
C(14)	63(2)	85(3)	40(2)	-13(2)	10(2)	-27(2)
C(15)	66(2)	73(2)	60(2)	-30(2)	18(2)	-6(2)
C(16)	48(2)	59(2)	49(2)	-16(2)	6(2)	3(2)
C(17)	34(1)	41(1)	52(2)	2(1)	13(1)	6(1)
C(18)	57(2)	68(2)	52(2)	4(2)	16(2)	8(2)
C(19)	70(3)	79(3)	76(3)	13(2)	38(2)	13(2)
C(20)	52(2)	69(2)	118(4)	12(2)	39(3)	0(2)
C(21)	47(2)	64(2)	99(3)	-16(2)	9(2)	-7(2)
C(22)	47(2)	52(2)	58(2)	-9(2)	13(2)	4(1)

Table 5. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for cd23463.

	x	y	z	U(eq)
H(1)	1475(18)	10780(30)	1216(18)	52(8)
H(2)	-650(20)	11390(30)	1363(17)	47(8)
H(3)	-780(20)	10710(30)	457(19)	50(8)
H(4)	-60(20)	10700(40)	2807(19)	61(10)
H(5)	-580(30)	10660(40)	4100(20)	81(11)
H(6)	-1840(30)	8790(40)	4300(20)	77(10)
H(7)	-2420(30)	7040(40)	3270(20)	81(12)
H(8)	-1940(30)	7150(30)	1990(20)	50(9)
H(9)	-2660(30)	9280(40)	423(19)	59(10)
H(10)	-3880(30)	7880(40)	-690(20)	77(11)
H(11)	-3150(20)	5640(30)	-980(20)	56(9)
H(12)	-1340(30)	4910(20)	-230(20)	69(10)
H(13)	-186(17)	6320(30)	737(18)	55(9)
H(14)	2730(20)	7800(30)	618(18)	62(10)
H(15)	4440(30)	8550(40)	410(20)	73(12)
H(16)	5920(20)	9410(40)	1530(20)	82(12)
H(17)	5660(30)	9560(40)	2890(20)	87(12)
H(18)	3950(20)	8730(30)	3045(18)	44(9)

Table 6. Torsion angles [deg] for cd23463.

C(17)-Se-C(1)-C(2)	-10.8(3)
C(17)-Se-C(1)-C(4)	173.2(2)
C(4)-C(1)-C(2)-C(3)	0.2(2)
Se-C(1)-C(2)-C(3)	-176.7(2)
C(1)-C(2)-C(3)-C(4)	-0.2(2)
C(2)-C(1)-C(4)-C(11)	112.6(2)
Se-C(1)-C(4)-C(11)	-70.2(3)
C(2)-C(1)-C(4)-C(5)	-115.1(2)
Se-C(1)-C(4)-C(5)	62.1(3)
C(2)-C(1)-C(4)-C(3)	-0.2(2)
Se-C(1)-C(4)-C(3)	177.0(2)
C(2)-C(3)-C(4)-C(11)	-116.7(2)
C(2)-C(3)-C(4)-C(1)	0.21(19)
C(2)-C(3)-C(4)-C(5)	111.9(2)
C(11)-C(4)-C(5)-C(10)	15.3(3)
C(1)-C(4)-C(5)-C(10)	-119.3(3)
C(3)-C(4)-C(5)-C(10)	147.2(3)
C(11)-C(4)-C(5)-C(6)	-167.1(2)
C(1)-C(4)-C(5)-C(6)	58.3(3)
C(3)-C(4)-C(5)-C(6)	-35.2(4)
C(10)-C(5)-C(6)-C(7)	-1.2(4)
C(4)-C(5)-C(6)-C(7)	-178.9(3)
C(5)-C(6)-C(7)-C(8)	1.4(5)
C(6)-C(7)-C(8)-C(9)	-0.6(5)
C(7)-C(8)-C(9)-C(10)	-0.3(5)
C(8)-C(9)-C(10)-C(5)	0.5(5)
C(6)-C(5)-C(10)-C(9)	0.3(4)
C(4)-C(5)-C(10)-C(9)	177.9(3)
C(1)-C(4)-C(11)-C(16)	28.4(4)
C(5)-C(4)-C(11)-C(16)	-103.9(3)
C(3)-C(4)-C(11)-C(16)	123.2(3)
C(1)-C(4)-C(11)-C(12)	-151.8(2)
C(5)-C(4)-C(11)-C(12)	75.9(3)
C(3)-C(4)-C(11)-C(12)	-57.0(3)
C(16)-C(11)-C(12)-C(13)	-0.5(4)
C(4)-C(11)-C(12)-C(13)	179.7(3)
C(11)-C(12)-C(13)-C(14)	0.5(5)
C(12)-C(13)-C(14)-C(15)	-0.3(5)
C(13)-C(14)-C(15)-C(16)	0.1(5)
C(12)-C(11)-C(16)-C(15)	0.3(5)
C(4)-C(11)-C(16)-C(15)	-179.9(3)
C(14)-C(15)-C(16)-C(11)	-0.1(5)
C(1)-Se-C(17)-C(18)	-75.1(3)
C(1)-Se-C(17)-C(22)	108.0(2)
C(22)-C(17)-C(18)-C(19)	0.2(5)
Se-C(17)-C(18)-C(19)	-176.7(3)
C(17)-C(18)-C(19)-C(20)	0.2(6)
C(18)-C(19)-C(20)-C(21)	-0.7(6)
C(19)-C(20)-C(21)-C(22)	0.7(6)
C(20)-C(21)-C(22)-C(17)	-0.3(5)
C(18)-C(17)-C(22)-C(21)	-0.1(5)
Se-C(17)-C(22)-C(21)	176.8(3)

Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for cd23463 [Å and deg.].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
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Figure 2. The ORTEP Draw of **9a**.

Table 1. Crystal data and structure refinement for cd23468.

Identification code	cd23468
Empirical formula	C19 H20 O3 Se
Formula weight	375.31
Temperature	293(2) K
Wavelength	0.71073 Å
Crystal system, space group	Triclinic, P-1
Unit cell dimensions	a = 8.1918(11) Å alpha = 112.611(2) deg. b = 9.9371(13) Å beta = 91.577(2) deg. c = 11.6367(16) Å gamma = 100.579(3) deg.
Volume	854.5(2) Å ³
Z, Calculated density	2, 1.459 Mg/m ³
Absorption coefficient	2.209 mm ⁻¹
F(000)	384
Crystal size	0.485 x 0.313 x 0.116 mm
Theta range for data collection	2.27 to 28.28 deg.
Limiting indices	-10<=h<=10, -13<=k<=10, -15<=l<=14
Reflections collected / unique	5211 / 3793 [R(int) = 0.0748]
Completeness to theta = 28.28	89.4 %
Absorption correction	Sadabs
Max. and min. transmission	1.00000 and 0.36647
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	3793 / 1 / 288
Goodness-of-fit on F ²	0.875
Final R indices [I>2sigma(I)]	R1 = 0.0495, wR2 = 0.1104
R indices (all data)	R1 = 0.0695, wR2 = 0.1167
Largest diff. peak and hole	0.893 and -0.653 e.Å ⁻³

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for cd23468. $U(\text{eq})$ is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	$U(\text{eq})$
Se	1001(1)	3259(1)	9433(1)	53(1)
O(1)	4039(3)	3703(3)	13721(2)	50(1)
O(2)	5019(3)	1450(3)	14059(2)	51(1)
O(3)	4515(3)	-1262(3)	12226(2)	51(1)
C(1)	1288(4)	1267(4)	8824(3)	40(1)
C(2)	831(6)	-1(4)	7547(3)	51(1)
C(3)	1548(6)	-947(4)	8159(3)	46(1)
C(4)	1919(4)	457(4)	9355(3)	39(1)
C(5)	2706(4)	732(4)	10583(3)	38(1)
C(6)	3216(5)	-430(4)	10786(3)	41(1)
C(7)	3975(4)	-196(4)	11938(3)	40(1)
C(8)	4227(4)	1208(4)	12911(3)	40(1)
C(9)	3732(4)	2371(4)	12710(3)	39(1)
C(10)	2974(4)	2132(4)	11553(3)	39(1)
C(11)	4343(7)	-2693(5)	11248(5)	58(1)
C(12)	3886(8)	1321(6)	14933(4)	63(1)
C(13)	3489(7)	4910(5)	13569(4)	57(1)
C(17)	763(5)	3437(4)	7854(3)	45(1)
C(18)	1902(6)	4474(5)	7617(4)	59(1)
C(19)	1745(7)	4595(6)	6470(5)	69(1)
C(20)	462(7)	3669(5)	5579(4)	67(1)
C(21)	-702(6)	2662(5)	5821(4)	57(1)
C(22)	-564(5)	2555(5)	6965(4)	52(1)

Table 3. Bond lengths [Å] and angles [deg] for cd23468.

Se-C(1)	1.891(3)
Se-C(17)	1.920(3)
O(1)-C(9)	1.366(4)
O(1)-C(13)	1.424(5)
O(2)-C(8)	1.382(4)
O(2)-C(12)	1.422(6)
O(3)-C(7)	1.366(4)
O(3)-C(11)	1.417(5)
C(1)-C(4)	1.347(5)
C(1)-C(2)	1.514(5)
C(2)-C(3)	1.559(5)
C(2)-H(1)	0.95(3)
C(2)-H(2)	0.88(4)
C(3)-C(4)	1.518(5)
C(3)-H(3)	0.98(5)
C(3)-H(4)	0.93(4)
C(4)-C(5)	1.455(4)
C(5)-C(10)	1.387(5)
C(5)-C(6)	1.395(5)
C(6)-C(7)	1.379(5)
C(6)-H(5)	0.87(4)
C(7)-C(8)	1.392(5)
C(8)-C(9)	1.389(5)
C(9)-C(10)	1.381(4)
C(10)-H(6)	0.903(18)
C(11)-H(7)	1.01(4)
C(11)-H(8)	0.88(4)
C(11)-H(9)	0.89(5)
C(12)-H(10)	0.88(7)
C(12)-H(11)	0.90(5)
C(12)-H(12)	1.03(6)
C(13)-H(13)	0.92(4)
C(13)-H(14)	0.90(4)
C(13)-H(15)	1.06(4)
C(17)-C(18)	1.374(6)
C(17)-C(22)	1.382(6)
C(18)-C(19)	1.391(6)
C(18)-H(16)	0.95(4)
C(19)-C(20)	1.371(7)
C(19)-H(17)	0.93(4)
C(20)-C(21)	1.368(6)
C(20)-H(18)	1.06(4)
C(21)-C(22)	1.380(5)
C(21)-H(19)	0.88(4)
C(22)-H(20)	0.94(4)
C(1)-Se-C(17)	98.15(14)
C(9)-O(1)-C(13)	117.0(3)
C(8)-O(2)-C(12)	113.1(3)
C(7)-O(3)-C(11)	117.2(3)
C(4)-C(1)-C(2)	94.7(3)
C(4)-C(1)-Se	133.6(3)
C(2)-C(1)-Se	131.7(3)
C(1)-C(2)-C(3)	85.6(3)
C(1)-C(2)-H(1)	115(2)
C(3)-C(2)-H(1)	117(2)
C(1)-C(2)-H(2)	115(2)
C(3)-C(2)-H(2)	113(2)
H(1)-C(2)-H(2)	110(3)
C(4)-C(3)-C(2)	86.4(3)
C(4)-C(3)-H(3)	112(3)
C(2)-C(3)-H(3)	110(3)
C(4)-C(3)-H(4)	114(2)
C(2)-C(3)-H(4)	113(2)

H(3)-C(3)-H(4)	117(3)
C(1)-C(4)-C(5)	136.0(3)
C(1)-C(4)-C(3)	93.4(3)
C(5)-C(4)-C(3)	130.6(3)
C(10)-C(5)-C(6)	119.3(3)
C(10)-C(5)-C(4)	121.3(3)
C(6)-C(5)-C(4)	119.4(3)
C(7)-C(6)-C(5)	120.5(3)
C(7)-C(6)-H(5)	121(2)
C(5)-C(6)-H(5)	118(2)
O(3)-C(7)-C(6)	124.7(3)
O(3)-C(7)-C(8)	115.4(3)
C(6)-C(7)-C(8)	119.9(3)
O(2)-C(8)-C(9)	120.3(3)
O(2)-C(8)-C(7)	119.9(3)
C(9)-C(8)-C(7)	119.8(3)
O(1)-C(9)-C(10)	124.6(3)
O(1)-C(9)-C(8)	115.2(3)
C(10)-C(9)-C(8)	120.2(3)
C(9)-C(10)-C(5)	120.3(3)
C(9)-C(10)-H(6)	119(2)
C(5)-C(10)-H(6)	120(2)
O(3)-C(11)-H(7)	108(2)
O(3)-C(11)-H(8)	112(3)
H(7)-C(11)-H(8)	118(3)
O(3)-C(11)-H(9)	105(4)
H(7)-C(11)-H(9)	101(4)
H(8)-C(11)-H(9)	111(4)
O(2)-C(12)-H(10)	117(4)
O(2)-C(12)-H(11)	109(3)
H(10)-C(12)-H(11)	116(5)
O(2)-C(12)-H(12)	110(3)
H(10)-C(12)-H(12)	101(5)
H(11)-C(12)-H(12)	103(4)
O(1)-C(13)-H(13)	107(2)
O(1)-C(13)-H(14)	115(2)
H(13)-C(13)-H(14)	109(4)
O(1)-C(13)-H(15)	111(2)
H(13)-C(13)-H(15)	111(3)
H(14)-C(13)-H(15)	103(3)
C(18)-C(17)-C(22)	119.7(4)
C(18)-C(17)-Se	119.7(3)
C(22)-C(17)-Se	120.6(3)
C(17)-C(18)-C(19)	119.8(5)
C(17)-C(18)-H(16)	121(2)
C(19)-C(18)-H(16)	119(2)
C(20)-C(19)-C(18)	119.7(5)
C(20)-C(19)-H(17)	122(3)
C(18)-C(19)-H(17)	118(3)
C(21)-C(20)-C(19)	120.8(4)
C(21)-C(20)-H(18)	121(2)
C(19)-C(20)-H(18)	119(2)
C(20)-C(21)-C(22)	119.7(5)
C(20)-C(21)-H(19)	120(3)
C(22)-C(21)-H(19)	120(3)
C(21)-C(22)-C(17)	120.3(4)
C(21)-C(22)-H(20)	117(2)
C(17)-C(22)-H(20)	123(2)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for cd23468.
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
Se	77(1)	43(1)	36(1)	11(1)	-1(1)	18(1)
O(1)	68(2)	36(1)	38(1)	8(1)	-10(1)	11(1)
O(2)	55(2)	54(2)	42(1)	18(1)	-8(1)	12(1)
O(3)	63(2)	40(1)	52(2)	17(1)	-1(1)	18(1)
C(1)	42(2)	37(2)	36(2)	11(2)	6(2)	8(2)
C(2)	60(3)	50(2)	35(2)	9(2)	-6(2)	8(2)
C(3)	48(2)	41(2)	42(2)	8(2)	2(2)	10(2)
C(4)	43(2)	36(2)	33(2)	12(1)	0(1)	2(2)
C(5)	37(2)	39(2)	36(2)	15(2)	4(1)	3(2)
C(6)	45(2)	36(2)	38(2)	10(2)	3(2)	7(2)
C(7)	44(2)	39(2)	40(2)	18(2)	6(2)	11(2)
C(8)	41(2)	40(2)	37(2)	17(2)	-2(2)	4(2)
C(9)	40(2)	35(2)	38(2)	11(2)	1(1)	5(2)
C(10)	47(2)	36(2)	37(2)	15(2)	2(2)	10(2)
C(11)	69(3)	42(2)	67(3)	21(2)	7(3)	21(2)
C(12)	87(4)	62(3)	44(2)	25(2)	1(2)	16(3)
C(13)	85(4)	35(2)	44(2)	9(2)	-4(2)	16(2)
C(17)	56(2)	44(2)	40(2)	16(2)	10(2)	19(2)
C(18)	54(3)	57(3)	69(3)	26(2)	3(2)	13(2)
C(19)	80(3)	67(3)	78(3)	47(3)	23(3)	15(3)
C(20)	92(4)	73(3)	61(3)	40(3)	19(3)	41(3)
C(21)	69(3)	59(3)	48(2)	23(2)	1(2)	26(2)
C(22)	52(2)	51(2)	53(2)	22(2)	3(2)	9(2)

Table 5. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for cd23468.

	x	y	z	U(eq)
H(1)	1390(40)	150(40)	6890(30)	47(10)
H(2)	-240(50)	-320(40)	7310(30)	38(10)
H(3)	650(60)	-1710(50)	8210(40)	75(14)
H(4)	2490(50)	-1260(40)	7820(30)	46(10)
H(5)	3090(50)	-1290(40)	10160(30)	48(10)
H(6)	2730(40)	2910(30)	11420(30)	44(10)
H(7)	5040(50)	-2570(40)	10570(40)	59(12)
H(8)	3290(60)	-3170(40)	11030(40)	52(12)
H(9)	4930(70)	-3180(60)	11550(50)	104(19)
H(10)	3060(80)	540(70)	14670(60)	120(20)
H(11)	4480(60)	1510(50)	15660(50)	88(16)
H(12)	3250(70)	2180(60)	15170(50)	105(18)
H(13)	3710(50)	5710(50)	14340(40)	64(12)
H(14)	3960(50)	5190(40)	12990(40)	54(12)
H(15)	2200(50)	4620(40)	13250(40)	55(11)
H(16)	2780(50)	5130(40)	8230(30)	43(10)
H(17)	2550(60)	5290(50)	6320(40)	67(13)
H(18)	360(50)	3780(40)	4710(40)	71(12)
H(19)	-1530(50)	2080(50)	5250(40)	61(13)
H(20)	-1390(50)	1850(40)	7100(30)	51(11)

Table 6. Torsion angles [deg] for cd23468.

C(17)-Se-C(1)-C(4)	160.8(4)
C(17)-Se-C(1)-C(2)	-21.0(4)
C(4)-C(1)-C(2)-C(3)	0.4(3)
Se-C(1)-C(2)-C(3)	-178.3(3)
C(1)-C(2)-C(3)-C(4)	-0.4(3)
C(2)-C(1)-C(4)-C(5)	178.6(4)
Se-C(1)-C(4)-C(5)	-2.8(7)
C(2)-C(1)-C(4)-C(3)	-0.4(4)
Se-C(1)-C(4)-C(3)	178.2(3)
C(2)-C(3)-C(4)-C(1)	0.4(3)
C(2)-C(3)-C(4)-C(5)	-178.7(4)
C(1)-C(4)-C(5)-C(10)	-0.7(6)
C(3)-C(4)-C(5)-C(10)	178.0(4)
C(1)-C(4)-C(5)-C(6)	-180.0(4)
C(3)-C(4)-C(5)-C(6)	-1.3(6)
C(10)-C(5)-C(6)-C(7)	0.2(5)
C(4)-C(5)-C(6)-C(7)	179.5(3)
C(11)-O(3)-C(7)-C(6)	2.9(6)
C(11)-O(3)-C(7)-C(8)	-177.5(4)
C(5)-C(6)-C(7)-O(3)	179.9(3)
C(5)-C(6)-C(7)-C(8)	0.4(5)
C(12)-O(2)-C(8)-C(9)	86.0(4)
C(12)-O(2)-C(8)-C(7)	-96.2(4)
O(3)-C(7)-C(8)-O(2)	1.7(5)
C(6)-C(7)-C(8)-O(2)	-178.7(3)
O(3)-C(7)-C(8)-C(9)	179.5(3)
C(6)-C(7)-C(8)-C(9)	-0.9(5)
C(13)-O(1)-C(9)-C(10)	1.8(6)
C(13)-O(1)-C(9)-C(8)	-177.5(3)
O(2)-C(8)-C(9)-O(1)	-2.1(5)
C(7)-C(8)-C(9)-O(1)	-179.8(3)
O(2)-C(8)-C(9)-C(10)	178.6(3)
C(7)-C(8)-C(9)-C(10)	0.8(5)
O(1)-C(9)-C(10)-C(5)	-179.5(3)
C(8)-C(9)-C(10)-C(5)	-0.2(5)
C(6)-C(5)-C(10)-C(9)	-0.3(5)
C(4)-C(5)-C(10)-C(9)	-179.6(3)
C(1)-Se-C(17)-C(18)	-118.3(3)
C(1)-Se-C(17)-C(22)	63.3(3)
C(22)-C(17)-C(18)-C(19)	-2.2(6)
Se-C(17)-C(18)-C(19)	179.3(3)
C(17)-C(18)-C(19)-C(20)	-0.6(7)
C(18)-C(19)-C(20)-C(21)	2.4(7)
C(19)-C(20)-C(21)-C(22)	-1.4(7)
C(20)-C(21)-C(22)-C(17)	-1.5(6)
C(18)-C(17)-C(22)-C(21)	3.2(6)
Se-C(17)-C(22)-C(21)	-178.3(3)

Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for cd23468 [Å and deg.].

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