Supporting Information

Studies on the Regio- and Stereoselectivity of Halohydroxylation of 1,2-Allenyl

Sulfides or Selenides

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Experimental Section

Synthesis of 3-Benzyl-1,2-butadienyl Phenyl Sulfide (2a). Typical Procedure A.

To a solution of 2.499 g (9.31 mmol) of **1a** in CH₂Cl₂ (50 mL) in a 3-necked flask was added pyridine (3.0 mL, 2.95 g, 37.2 mmol) and P₂S₅ (1.036 g, 4.66 mmol) subsequently with stirring. The reaction was monitored by TLC (eluent: petroleum ether). After stirring for 4 h, the solution was concentrated and immediately purified by column chromatography on silica gel (eluent: petroleum ether) to afford 1.108 g (47%) of **Z-2a**. Oil; ¹H NMR (300 MHz, CDCl₃) 7.20-7.42 (m, 10 H), 5.88-5.97 (m, 1 H), 3.41 (d, *J* = 1.8 Hz, 2 H), 1.82 (d, *J* = 2.1 Hz, 3 H); ¹³C NMR (75.4 MHz, CDCl₃) 203.5, 138.4, 135.9, 129.1, 128.9, 128.7, 128.2, 126.3, 126.1, 104.5, 85.5, 40.8, 18.4; IR (neat) ν(cm⁻¹) = 1945; MS (EI) *m/z* (%): 252 (M⁺, 66.84), 128 (100); HRMS Calcd for C₁₇H₁₆S (M⁺): 252.09727. Found: 252.10052.

The following compounds were prepared according to Typical Procedure A.

(1) **1,2-Propadienyl Phenyl Sulfide (2b)**.¹ The mixture of **1b** (2.070 g, 12.6 mmol), pyridine (4.2 mL, 4.12 g, 52.2 mmol), and P₂S₅ (7.013 g, 31.6 mmol) in CH₂Cl₂ (30 mL) was stirred for 6 h to afford 0.570 g (31%) of **2b**. Oil; ¹H NMR (300 MHz, CDCl₃) 7.45 (d, *J* = 7.2 Hz, 2 H), 7.35 (t, *J* = 7.2 Hz, 2 H), 7.26 (t, *J* = 7.2 Hz, 1 H), 6.02 (t, *J* = 6.4 Hz, 1 H), 5.04 (d, *J* = 6.4 Hz, 2 H); ¹³C NMR (75.4 MHz, CDCl₃) 209.3, 135.7, 128.9, 128.8, 126.4, 85.8, 78.7; IR (neat) ν(cm⁻¹) = 1940; MS (EI) *m/z* (%): 148 (M⁺, 33.81), 147 (100).

(2) **1,2-Butadienyl Phenyl Sulfide (2c)**. The mixture of **1c** (0.876 g, 5.0 mmol),

pyridine (1.61 mL, 1.59 g, 20.0 mmol), and P₂S₅ (0.630 g, 2.8 mmol) in CH₂Cl₂ (25 mL) was stirred for 6 h to afford 0.313 g (39%) of **2c**. Oil; ¹H NMR (300 MHz, CDCl₃) 7.40 (d, *J* = 7.5 Hz, 2 H), 7.32 (t, *J* = 7.5 Hz, 2 H), 7.22 (t, *J* = 7.5 Hz, 1 H), 5.86-5.95 (m, 1 H), 5.32-5.44 (m, 1 H), 1.73 (dd, *J* = 7.5 Hz and 3.0 Hz, 3 H); ¹³C NMR (75.4 MHz, CDCl₃) 205.7, 135.9, 129.0, 128.9, 126.3, 90.4, 85.7, 14.1; IR (neat) ν(cm⁻¹) = 1945, 1582, 1439; MS (EI) *m/z* (%): 162 (M⁺, 23.20), 147 (100); HRMS Calcd for C₉H₇S (M⁺-Me): 147.02685. Found: 147.02626.

(3) 4-Methyl-1,2-pentadienyl Phenyl Sulfide (2d).² The mixture of **1d** (1.688 g, 10 mmol), pyridine (3.2 mL, 3.18 g, 40.0 mmol), and P₂S₅ (1.350 g, 6.1 mmol) in CH₂Cl₂ (25 mL) was stirred for 5.5 h to afford 1.044 g (68%) of **2d**. Oil; ¹H NMR (300 MHz, CDCl₃) 7.45 (d, *J* = 7.5 Hz, 2 H), 7.35 (t, *J* = 7.5 Hz, 2 H), 7.24 (t, *J* = 7.5 Hz, 1 H), 6.00-6.05 (m, 1 H), 5.45-5.49 (m, 1 H), 2.36-2.44 (m, 1 H), 1.06 (d, *J* = 7.2 Hz, 6 H); ¹³C NMR (75.4 MHz, CDCl₃) 203.2, 135.7, 129.2, 128.8, 126.3, 102.9, 87.4, 28.0, 22.05, 22.02; IR (neat) ν(cm⁻¹) = 1946, 1584, 1478, 1439; MS (EI) *m/z* (%): 190 (M⁺, 27.12), 147 (100).

(4) 1,2-Heptadienyl Phenyl Sulfide (2e). The mixture of **1e** (2.053 g, 10.0 mmol), pyridine (3.2 mL, 3.16 g, 40.0 mmol), and P₂S₅ (1.229 g, 5.5 mmol) in CH₂Cl₂ (50 mL) was stirred for 4 h and 20 min to afford 1.140 g (60%) of **2e**. Oil; ¹H NMR (300 MHz, CDCl₃) 7.42 (d, *J* = 6.9 Hz, 2 H), 7.32 (t, *J* = 6.9 Hz, 2 H), 7.24 (t, *J* = 6.9 Hz, 1 H), 5.95 (dt, *J* = 6.9 Hz and 3.0 Hz, 1 H), 5.43 (q, *J* = 6.9 Hz, 1 H), 2.03-2.13 (m, 2 H), 1.25-1.50 (m, 4 H), 0.93 (t, *J* = 7.1 Hz, 3 H); ¹³C NMR (75.4 MHz, CDCl₃)

204.9, 136.0, 129.0, 128.8, 126.2, 95.6, 86.2, 30.8, 28.1, 22.0, 13.8; IR (neat) $\nu(\text{cm}^{-1}) = 1947, 1584, 1478, 1439$; MS (EI) m/z (%): 204 (M^+ , 5.16), 162 (100); HRMS Calcd for $C_{13}H_{16}S$ (M^+): 204.09727. Found: 204.09742.

(5) 1,2-decadienyl Phenyl Sulfide (2f). The mixture of **1f** (2.069 g, 8.0 mmol), pyridine (2.6 mL, 2.55 g, 32.0 mmol), and P_2S_5 (0.974 g, 4.4 mmol) in CH_2Cl_2 (40 mL) was stirred for 4 h to afford 1.233 g (64%) of **2f**. Oil; ^1H NMR (300 MHz, $CDCl_3$)

7.40 (d, $J = 7.5$ Hz, 2 H), 7.31 (t, $J = 7.5$ Hz, 2 H), 7.22 (t, $J = 7.5$ Hz, 1 H), 5.94 (dt, $J = 6.9$ Hz and 3.0 Hz, 1 H), 5.43 (q, $J = 6.9$ Hz, 1 H), 2.01-2.14 (m, 2 H), 1.20-1.47 (m, 10 H), 0.89 (t, $J = 6.6$ Hz, 3 H); ^{13}C NMR (75.4 MHz, $CDCl_3$) 205.0, 136.0, 129.0, 128.8, 126.2, 95.7, 86.1, 31.8, 29.1, 29.0, 28.7, 28.5, 22.6, 14.1; IR (neat) $\nu(\text{cm}^{-1}) = 1945, 1479, 1439$; MS (EI) m/z (%): 246 (M^+ , 5.16), 162 (100); HRMS Calcd for $C_{16}H_{22}S$ (M^+): 246.14422. Found: 246.14412.

(6) 4-Phenylbuta-1,2-dienyl Phenyl Sulfide (2g). The reaction of **1g** (2.001 g, 8.0 mmol), pyridine (2.6 mL, 2.55 g, 32.0 mmol), and P_2S_5 (1.002 g, 4.5 mmol) in CH_2Cl_2 (40 mL) afforded 0.700 g (37%) of **2g**. Oil; ^1H NMR (300 MHz, $CDCl_3$) 7.20-7.41 (m, 10 H), 6.00 (dt, $J = 6.7$ Hz and 2.7 Hz, 1 H), 5.63 (q, $J = 6.7$ Hz, 1 H), 3.46 (dd, $J = 6.7$ Hz and 2.7 Hz, 2 H); ^{13}C NMR (75.4 MHz, $CDCl_3$) 205.2, 139.1, 135.4, 129.3, 128.8, 128.5, 128.4, 126.4, 126.3, 95.2, 87.3, 35.1; IR (neat) $\nu(\text{cm}^{-1}) = 1945, 1583, 1478, 1439$; MS (EI) m/z (%): 238 (M^+ , 81.97), 128(100); HRMS Calcd for $C_{16}H_{14}S$ (M^+): 238.08162. Found: 238.07924.

(7) 3-Methylbuta-1,2-dienyl Phenyl Sulfide (2h).³ The mixture of **1h** (1.538 g, 8.0 mmol), pyridine (2.6 mL, 2.55 g, 32.0 mmol), and P_2S_5 (1.925 g, 8.7 mmol) in

CH₂Cl₂ (40 mL) was stirred for 5.5 h to afford 0.868 g (62%) of **2h**. Oil; ¹H NMR (300 MHz, CDCl₃) 7.41 (d, *J* = 6.9 Hz, 2 H), 7.31 (t, *J* = 6.9 Hz, 2 H), 7.21 (t, *J* = 6.9 Hz, 1 H), 5.80-5.88 (m, 1 H), 1.76 (d, *J* = 2.7 Hz, 6 H); ¹³C NMR (75.4 MHz, CDCl₃) 203.0, 136.2, 129.0, 128.7, 126.1, 100.6, 83.9, 20.3; IR (neat) ν(cm⁻¹) = 1945, 1582, 1478, 1439; MS (EI) *m/z* (%): 176(M⁺, 21.49), 161(100).

(8) 3-Methylpenta-1,2-dienyl Phenyl Sulfide (2i). The reaction of **1i** (1.108 g, 5.0 mmol), pyridine (1.6 mL, 1.59 g, 20.0 mmol), and P₂S₅ (0.702 g, 3.2 mmol) in CH₂Cl₂ (30 mL) afforded 0.825 g (90%) of **2i**. Oil; ¹H NMR (300 MHz, CDCl₃) 7.41 (d, *J* = 7.5 Hz, 2 H), 7.31 (t, *J* = 7.5 Hz, 2 H), 7.21 (t, *J* = 7.5 Hz, 1 H), 5.84-5.98 (m, 1 H), 1.94-2.15 (m, 2 H), 1.76 (d, *J* = 2.1 Hz, 3 H), 1.03 (t, *J* = 7.4 Hz, 3 H); ¹³C NMR (75.4 MHz, CDCl₃) 202.3, 136.2, 129.0, 128.7, 126.1, 106.9, 85.8, 27.0, 18.9, 12.0; IR (neat) ν(cm⁻¹) = 1945, 1583, 1478, 1439; MS (EI) *m/z* (%): 190 (M⁺, 58.95), 161 (100); HRMS Calcd for C₁₂H₁₄S (M⁺): 190.08162. Found: 190.08175.

(9) 3,5-Dimethylhexa-1,2-dienyl Phenyl Sulfide (2j). The mixture of **1j** (1.806 g, 8.0 mmol), pyridine (2.6 mL, 2.55 g, 32.0 mmol), and P₂S₅ (0.912 g, 4.1 mmol) in CH₂Cl₂ (40 mL) was stirred for 7.5 h to afford 0.982 g (58%) of **2j**. Oil; ¹H NMR (300 MHz, CDCl₃) 7.40 (d, *J* = 7.5 Hz, 2 H), 7.30 (t, *J* = 7.5 Hz, 2 H), 7.18 (t, *J* = 7.5 Hz, 1 H), 5.82-5.92 (m, 1 H), 1.78-1.96 (m, 1 H, 1 H, 1H) 1.75-1.78 (m, 3 H), 0.95 (dd, *J* = 6.6 Hz and 1.5 Hz, 6 H); ¹³C NMR (75.4 MHz, CDCl₃) 203.4, 136.5, 128.7, 127.3, 126.0, 103.6, 84.4, 43.4, 26.3, 22.5, 22.4, 19.0; IR (neat) ν(cm⁻¹) = 1943, 1583, 1478, 1439; MS (EI) *m/z* (%): 218 (M⁺, 22.24), 109 (100); HRMS Calcd for C₁₄H₁₈S (M⁺): 218.11292. Found: 218.10971.

(10) 3,4,4-Trimethylpenta-1,2-dienyl Phenyl Sulfide (2k). The reaction of **1k** (1.188 g, 5.0 mmol), pyridine (1.6 mL, 1.58 g, 20.0 mmol), and P₂S₅ (0.637 g, 2.9 mmol) in CH₂Cl₂ (30 mL) afforded 0.566 g (51%) of **2k**. Oil; ¹H NMR (300 MHz, CDCl₃) 7.41 (d, *J* = 7.5 Hz, 2 H), 7.30 (t, *J* = 7.5 Hz, 2 H), 7.20 (t, *J* = 7.5 Hz, 1 H), 5.86 (q, *J* = 2.4 Hz, 1 H), 1.73 (d, *J* = 2.4 Hz, 3 H), 1.03 (s, 9 H); ¹³C NMR (75.4 MHz, CDCl₃) 201.4, 136.0, 129.5, 128.8, 126.3, 114.0, 85.5, 33.9, 28.8, 14.8; IR (neat) ν(cm⁻¹) = 1941, 1583, 1477, 1439; MS (EI) *m/z* (%): 218 (M⁺, 24.45), 161 (100); HRMS Calcd for C₁₄H₁₈S (M⁺): 218.11292. Found: 218.11717.

(11) 3-Ethylpenta-1,2-dienyl Phenyl Sulfide (2l). The mixture of **1l** (1.118 g, 5.0 mmol), pyridine (1.6 mL, 1.58 g, 20.0 mmol), and P₂S₅ (0.602 g, 2.7 mmol) in CH₂Cl₂ (40 mL) was stirred for 5 h to afford 0.524 g (51%) of **2l**. Oil; ¹H NMR (300 MHz, CDCl₃) 7.39 (d, *J* = 7.5 Hz, 2 H), 7.28 (t, *J* = 7.5 Hz, 2 H), 7.19 (t, *J* = 7.5 Hz, 1 H), 6.00 (pentet, *J* = 3.0 Hz, 1 H), 2.02 (dq, *J* = 7.2 Hz, and 3.0 Hz, 4 H), 1.03 (t, *J* = 7.5 Hz, 6 H); ¹³C NMR (75.4 MHz, CDCl₃) 201.8, 136.3, 129.0, 128.8, 126.1, 113.7, 87.7, 25.7, 12.1; IR (neat) ν(cm⁻¹) = 1943, 1583, 1478, 1456, 1439; MS (EI) *m/z* (%): 204 (M⁺, 41.65), 175 (100); HRMS Calcd for C₁₃H₁₆S (M⁺): 204.09727. Found: 204.09757.

(12) 3-Butylhepta-1,2-dienyl Phenyl Sulfide (2m). The reaction of **1m** (1.487 g, 5.4 mmol), pyridine (1.74 mL, 1.707 g, 21.6 mmol), and P₂S₅ (0.628 g, 2.8 mmol) in CH₂Cl₂ (40 mL) afforded 1.094 g (71%) of **2m**. Oil; ¹H NMR (300 MHz, CDCl₃) 7.44 (d, *J* = 7.5 Hz, 2 H), 7.32 (t, *J* = 7.5 Hz, 2 H), 7.22 (t, *J* = 7.5 Hz, 1 H), 5.78-6.02 (m, 1 H), 1.98-2.18 (m, 4 H), 1.30-1.56 (m, 8 H), 0.98(t, *J* = 7.2 Hz, 6 H); ¹³C NMR

(75.4 MHz, CDCl₃) 202.5, 136.6, 128.8, 128.7, 125.9, 109.7, 86.5, 32.3, 29.5, 22.3, 13.9; IR (neat) $\nu(\text{cm}^{-1})$ = 1941, 1478, 1465, 1439; MS (EI) m/z (%): 260 (M^+ , 8.65), 109 (100); HRMS Calcd for C₁₇H₂₄S (M^+): 260.15987. Found: 260.15615.

(13) 3-Pentyllocta-1,2-dienyl Phenyl Sulfide (2n). The reaction of **1n** (1.342 g, 4.4 mmol), pyridine (1.45 mL, 1.423 g, 16.6 mmol), and P₂S₅ (0.498 g, 2.2 mmol) in CH₂Cl₂ (40 mL) afforded 1.009 g (79%) of **2n**. Oil; ¹H NMR (300 MHz, CDCl₃) 7.41 (d, J = 7.8 Hz, 2 H), 7.30 (t, J = 7.8 Hz, 2 H), 7.20 (t, J = 7.8 Hz, 1 H), 5.90-5.97 (m, 1 H), 1.80-2.10 (m, 4 H), 1.23-1.54 (m, 12 H), 0.91 (t, J = 6.9 Hz, 6 H); ¹³C NMR (75.4 MHz, CDCl₃) 202.6, 136.7, 128.8, 126.0, 109.9, 86.3, 32.5, 31.4, 27.1, 22.5, 14.0; IR (neat) $\nu(\text{cm}^{-1})$ = 1941, 1478, 1466, 1439; MS (EI) m/z (%): 288 (M^+ , 12.40), 123 (100); HRMS Calcd for C₁₉H₂₈S (M^+): 288.19117. Found: 288.19066.

(14) 2-(Cyclopentylidenylethenyl) Phenyl Sulfide (2o). The reaction of **1o** (2.181 g, 10.0 mmol), pyridine (3.2 mL, 3.16 g, 40.0 mmol), and P₂S₅ (6.669 g, 5.0 mmol) in CH₂Cl₂ (30 mL) afforded 1.515 g (75%) of **2o**. Oil; ¹H NMR (300 MHz, CDCl₃) 7.42 (d, J = 7.2 Hz, 2 H), 7.32 (t, J = 7.2 Hz, 2 H), 7.22 (t, J = 7.2 Hz, 1 H), 5.86-5.97 (m, 1 H), 2.32-2.60 (m, 4 H), 1.60-1.80 (m, 4 H); ¹³C NMR (75.4 MHz, CDCl₃) 198.2, 136.0, 129.2, 128.7, 126.2, 109.4, 86.5, 31.2, 26.8; IR (neat) $\nu(\text{cm}^{-1})$ = 1945, 1582, 1478, 1449, 1438; MS (70 eV, EI) m/z (%): 202 (M^+ , 77.40), 77 (100); HRMS Calcd for C₁₃H₁₄S (M^+): 202.08162. Found: 202.08096.

(15) 2-(Cyclohexylidenylethenyl) Phenyl Sulfide (2p). The reaction of **1p** (1.865 g, 8.0 mmol), pyridine (2.6 mL, 2.58 g, 32.0 mmol), and P₂S₅ (0.893 g, 4.0 mmol) in CH₂Cl₂ (30 mL) afforded 0.911 g (52%) of **2p**. Oil; ¹H NMR (300 MHz, CDCl₃)

7.34 (d, $J = 7.2$ Hz, 2 H), 7.24 (t, $J = 7.2$ Hz, 2 H), 7.14 (t, $J = 7.2$ Hz, 1 H), 5.71-5.79 (m, 1 H), 1.98-2.20 (m, 4 H), 1.36-1.58 (m, 6 H); ^{13}C NMR (75.4 MHz, CDCl_3) 199.5, 136.1, 129.4, 128.8, 126.2, 107.7, 83.7, 31.2, 27.0, 25.8; IR (neat) $\nu(\text{cm}^{-1}) = 1950$, 1583, 1478, 1439; MS (EI) m/z (%): 216 (M^+ , 83.98), 173 (100); HRMS Calcd for $\text{C}_{14}\text{H}_{16}\text{S}$ (M^+): 216.09727. Found: 216.09443.

(16) 1,2-Heptadienyl 4'-Bromophenyl Sulfide (2q). The mixture of **1q** (1.451 g, 5.0 mmol), pyridine (1.62 mL, 1.59 g, 20.0 mmol), and P_2S_5 (0.680 g, 3.1 mmol) in CH_2Cl_2 (30 mL) was stirred for 2 h to afford 0.855 g (62%) of **2q**. Oil; ^1H NMR (300 MHz, CDCl_3) 7.40 (d, $J = 7.2$ Hz, 2 H), 7.23 (d, $J = 7.2$ Hz, 2 H), 5.86 (dt, $J = 6.5$ Hz and 2.7 Hz, 1 H), 5.42 (q, $J = 6.5$ Hz, 1 H), 1.96-2.13 (m, 2 H), 1.20-1.44 (m, 4 H), 0.89 (t, $J = 6.9$ Hz, 3 H); ^{13}C NMR (75.4 MHz, CDCl_3) 205.3, 135.2, 131.8, 130.4, 120.1, 95.9, 85.5, 30.8, 28.1, 22.0, 13.9; IR (neat) $\nu(\text{cm}^{-1}) = 1947$, 1472; MS (EI) m/z (%): 284 (M^+ (^{81}Br), 8.51), 282 (M^+ (^{79}Br), 9.71), 242 (97.65), 240 (100); HRMS Calcd for $\text{C}_{13}\text{H}_{15}^{79}\text{BrS}$ (M^+): 282.00778. Found: 282.00813.

Synthesis of 1,2-Heptadien-3-yl Phenyl Sulfide (2r).⁴ To a solution of **1r** (1.235 g, 5.6 mmol), NEt_3 (3.2 mL, 22.8 mmol), and anhydrous NaI (1.908 g, 12.7 mmol) in anhydrous acetone (28 mL) in a 3-necked flask was added $(\text{CF}_3\text{CO})_2\text{O}$ (1.96 mL) at -50°C during 1 min. After being stirred for 0.5 h, the bath temperature is gradually allowed to rise to -30°C in a course of 15 min. Then the cold mixture was poured into a two-phase mixture consisting of hexane (15 mL), aq 5% NaHCO_3 solution (10 mL), and aq 5% Na_2SO_3 solution (10 mL). The mixture was extracted with hexane (2 x 20 mL). The combined hexane layer was washed subsequently with water (3 x 15 mL),

brine (20 mL), and dried over MgSO_4 . The solution was concentrated and immediately purified by column chromatography on silica gel (petroleum ether) to afford 224 mg (20%) **2r**. Oil; ^1H NMR (300 MHz, CDCl_3) 7.10-7.36 (m, 5 H), 4.76 (t, $J = 3.2$ Hz, 2 H), 2.08-2.15 (m, 2 H), 1.40-1.50 (m, 2 H), 1.21-1.33 (m, 2 H), 0.83 (t, $J = 7.2$ Hz, 3 H); ^{13}C NMR (75.4 MHz, CDCl_3) 208.1, 134.7, 130.3, 128.8, 126.6, 99.6, 76.6, 32.7, 29.9, 22.0, 13.9; IR (neat) $\nu(\text{cm}^{-1}) = 1943$; MS (EI) m/z (%): 204 (M^+ , 14.00), 147 (100); HRMS Calcd for $\text{C}_{13}\text{H}_{16}\text{S}$ (M^+): 204.09727. Found: 204.09700.

Synthesis of 1,2-Butadien-3-yl Phenyl Sulfide (2s).⁵ To a suspension of *t*-BuOK 0.568 g (5.07 mmol) in *t*-BuOH (50 mL) was added 3-phenylthio-1-butyne (0.858 g, 5.3 mmol). After stirring for 1 h at 85 °C, the solution was poured into ice water. The reaction was then extracted with petroleum ether and the organic layer was dried over MgSO_4 . The solution was concentrated and immediately purified by column chromatography on silica gel (petroleum ether) to afford compound **2s** (0.715 g, 83%): Oil. ^1H NMR (300 MHz, CDCl_3) 7.21-7.45 (m, 5 H), 4.80 (q, $J = 3.2$ Hz, 2 H), 1.97 (t, $J = 3.2$ Hz, 3 H); ^{13}C NMR (75.4 MHz, CDCl_3) 208.3, 134.1, 130.4, 128.8, 126.7, 94.8, 75.7, 19.9; IR (neat) $\nu(\text{cm}^{-1}) = 1945$; MS (EI) m/z (%): 162 (M^+ , 20.96), 147 (100).

Reaction of 1a with I_2 in aqueous MeCN: A solution of allenyl phenyl sulfide (**2a**) (64 mg, 0.25 mmol) in 1 mL of CH_3CN was added slowly to a solution of iodine (127 mg, 0.5 mmol) in CH_3CN (7 mL) and water (2 mL) under a N_2 atmosphere. After being stirred at rt for 16 h, a saturated aqueous solution of $\text{Na}_2\text{S}_2\text{O}_3$ was added. The

mixture was extracted with ether and the organic layer was dried over MgSO_4 . Evaporation and column chromatography on silica gel (petroleum ether/ethyl acetate = 20:1) afforded **5a** (12 mg, 17%, $E/Z = 1.1/1$) as a solid.

***E*-2-Iodo-3-methyl-4-phenylbut-2-enal (*E*-5a).** solid; m.p. 65-67°C(*n*-hexane); ^1H NMR (300 MHz, CDCl_3) 9.19 (s, 1 H), 7.22-7.27 (m, 3 H), 7.07-7.09 (m, 2 H), 4.10 (s, 2 H) 2.15 (s, 3 H); ^{13}C NMR (75.4 MHz, CDCl_3) 184.65, 165.45, 136.34, 128.99, 128.46, 127.27, 109.54, 41.10, 32.43; IR (KBr) $\nu(\text{cm}^{-1}) = 3080, 3058, 3021, 2926, 2855, 1660, 1585, 1575, 1492, 1448, 1396, 1369, 1224, 1185, 1089, 1073, 1030$; MS (EI) $m/z(\%)$: 286 (M^+ , 3.50), 91 (100); HRMS Calcd for $\text{C}_{11}\text{H}_{11}\text{IO}$ (M^+): 285.98546. Found: 285.98692.

***Z*-2-Iodo-3-methyl-4-phenylbut-2-enal (*Z*-5a).** solid; m.p. 79-81°C(*n*-hexane); ^1H NMR (300 MHz, CDCl_3) 9.17 (s, 1 H), 7.24-7.33 (m, 5 H), 3.99 (s, 2 H) 2.33 (s, 3 H); ^{13}C NMR (75.4 MHz, CDCl_3) 185.32, 165.22, 136.35, 128.85, 128.73, 127.23, 108.30, 52.41, 20.25; IR (KBr) $\nu(\text{cm}^{-1}) = 3022, 1681, 1664, 1585, 1493, 1450, 1430, 1246, 1091, 1071, 1029, 732$; MS (EI) $m/z(\%)$: 286 (M^+ , 15.65), 91 (100); HRMS Calcd for $\text{C}_{11}\text{H}_{11}\text{IO}$ (M^+): 285.98546. Found: 285.98558.

Reaction of Allenyl Sulfide 2 with I_2 in Aqueous Acetone. Synthesis of

***Z*-3-Iodo-2-methyl-1-phenyl-4-(phenylsulfanyl)but-3-en-2-ol (*Z*-6a). Typical**

Procedure B. A solution of allenyl phenyl sulfide **2a** (64 mg, 0.25 mmol) in 1 mL of acetone was added slowly to a solution of iodine (127 mg, 0.5 mmol) in acetone (7 mL) and water (2 mL). After being stirred at rt for about 13 h, a saturated aqueous solution of $\text{Na}_2\text{S}_2\text{O}_3$ was added. The mixture was extracted with ether and the organic

layer was dried over MgSO_4 . Evaporation and column chromatography on silica gel (petroleum ether/ethyl acetate = 5:1) afforded **Z-6a** (68 mg, 68%) : Oil; ^1H NMR (300 MHz, CDCl_3) 7.27-7.36 (m, 10 H), 6.80 (s, 1 H), 3.21 (d, $J = 13.2$ Hz, 1H), 2.96 (d, $J = 13.8$ Hz, 1 H), 2.11 (s, 1 H) 1.56 (s, 3 H); ^{13}C NMR (75.4 MHz, CDCl_3) 136.04, 134.12, 133.70, 130.61, 130.43, 129.11, 128.21, 127.46, 126.93, 111.26, 77.43, 47.17, 28.00; IR (neat) $\nu(\text{cm}^{-1}) = 3538, 3449, 3059, 3027, 2927, 2925, 1581, 1568, 1478, 1452, 1440, 1372, 1341, 1239, 1113, 1090, 860$; MS (EI) m/z (%): 396 (M^+ , 0.95), 43 (100); HRMS Calcd for $\text{C}_{17}\text{H}_{17}\text{IOS}$ (M^+): 396.00448. Found: 396.00417.

Synthesis of 1-(phenylsulfinyl)-2-iodohept-1-en-3-ol (Z-8e). A solution of **Z-6e** (90 mg, 0.25 mmol) in CH_2Cl_2 (2 mL) was added *m*-CPBA (59 mg, 0.275 mmol) with stirring at 0°C . Then temperature was raised to rt. After stirring for 4 h, the reaction mixture was diluted with ether, and a saturated aq Na_2CO_3 solution was added. The mixture was extracted with ether. The combined organic layer was dried over MgSO_4 . Filtration, evaporation, and chromatography on silica gel (petroleum ether/ethyl acetate = 1:1) afforded 65 mg (69%) of **Z-8e**. Oil: ^1H NMR (300 MHz, CDCl_3) *Z*-isomer: 7.69-7.75 (m, 2 H), 7.44-7.55 (m, 3 H), 7.11 (d, $J = 0.9$ Hz, 1 H), 4.10-4.17 (m, 1 H), 3.30-3.76 (bs, 1 H), 1.72-1.82 (m, 1 H), 1.42-1.55 (m, 1 H), 1.22-1.30 (m, 4 H), 0.84 (t, $J = 9.6$ Hz, 3 H); ^{13}C NMR (75.4 MHz, CDCl_3) 143.2, 140.8, 131.3, 129.5, 124.3(1), 124.2(8), 78.1, 35.0, 26.9, 22.3, 13.9; *E*-isomer: 7.51-7.62 (m, 5 H), 6.98 (s, 1H), 5.07 (d, $J = 3.3$ Hz, 1H), 4.36-4.42 (m, 1H), 1.70 (t, $J = 4.9$ Hz, 1 H), 1.21-1.46 (m, 5 H), 0.89 (t, $J = 7.1$ Hz, 3 H); ^{13}C NMR (75.4 MHz, CDCl_3) 142.5,

142.1, 131.5, 129.6, 128.9, 124.3, 77.3, 36.5, 27.3, 22.5, 13.9; IR (neat) $\nu(\text{cm}^{-1})$ = 3356, 2955, 2930, 2859, 1582, 1443, 1081, 1031, 997; MS (EI) m/z (%): 364 (M^+ , 5.84), 77 (100); HRMS Calcd for $\text{C}_{13}\text{H}_{17}\text{IO}_2\text{S}$ (M^+): 363.99940. Found: 364.00432.

Synthesis of Z-1-(1'-cyclopentenyl)-1-iodo-2-(phenylsulfanyl) ethene (Z-9o).

According to Procedure B, the reaction of **2o** (204 mg, 1 mmol) and I_2 (508 mg, 2 mmol) in acetone/ H_2O (4:1, 15 mL) afforded 119 mg (37%) of **Z-9o** (eluent: petroleum ether). Oil; ^1H NMR (300 MHz, CDCl_3) 7.16-7.42 (m, 5 H), 6.72 (s, 1 H), 5.86 (s, 1 H), 2.42-2.56 (m, 2 H), 2.32-2.42 (m, 2H), 1.90-2.41(m, 2H); ^{13}C NMR (75.4 MHz, CDCl_3) 143.0, 135.2, 134.3, 134.1, 130.6, 129.3, 127.6, 96.8, 32.4, 32.0, 24.5; IR (neat) $\nu(\text{cm}^{-1})$ = 3056, 2951, 1582, 1478, 1439; MS (EI) m/z (%): 328 (4.50), 109 (100); HRMS Calcd for $\text{C}_{13}\text{H}_{11}\text{IS}$ (M^+): 327.97827. Found: 327.97744.

Bromohydroxylation of allenyl sulfides. Synthesis of Z-2-Bromo-1-(phenylsulfanyl)hept-1-en-3-ol (Z-10e). Typical Procedure C.

Bromine (1.2 mL, 0.5 mol/L in CH_3CN , 0.6 mmol) was added to a solution of Na_2CO_3 (32 mg, 0.3 mmol) in CH_3CN (3.8 mL) and water (1.5 mL). Then a solution of allenyl phenyl sulfide (**2e**) (62 mg, 0.3 mmol) in 1 mL of CH_3CN was added slowly to this solution at 0°C . After being stirred at 0°C for 2 h as monitored by TLC (eluent: petroleum ether/ethyl acetate = 5:1), a saturated aqueous solution of $\text{Na}_2\text{S}_2\text{O}_3$ was added. The mixture was extracted with ether (25 mL x 3) and dried over MgSO_4 . Evaporation and column chromatography on silica gel (petroleum ether/ethyl acetate = 8:1) afforded **10e** (53 mg, 58%): Oil; ^1H NMR (300 MHz, CDCl_3) 7.19-7.40 (m, 2 H), 7.01 (d, J = 0.6 Hz, 1 H), 4.10 (t, J = 6.6 Hz, 1 H), 1.92-2.21 (bs, 1 H),

1.56-1.71 (m, 2 H), 1.16-1.28 (m, 4 H), 0.84 (t, $J = 6.9$ Hz, 3 H); ^{13}C NMR (75.4 MHz, CDCl_3) 133.7, 130.5, 129.1, 127.7, 127.5, 126.6, 76.0, 35.3, 27.3, 22.3, 13.9; IR (neat) $\nu(\text{cm}^{-1}) = 1583$; MS (EI) m/z (%): 302 ($\text{M}^+(\text{}^{81}\text{Br})$, 28.06), 300 ($\text{M}^+(\text{}^{79}\text{Br})$, 28.15), 243 (100); HRMS Calcd for $\text{C}_{13}\text{H}_{17}\text{}^{79}\text{BrOS}$ (M^+): 300.01835. Found: 300.01545.

Synthesis of 1,2-Propadienyl Phenyl Selenide (4a). Typical Procedure D.⁶ A solution of EtONa in EtOH was prepared by adding a piece of sodium (0.210 g, 9.17 mmol) to EtOH (9.5 mL) with stirring at rt. Then compound **3a** (2.486 g, 12.8 mmol) was added at 48 °C. After stirring for 70 min at 48 °C, the solution was poured into a mixture of ice water and extracted with petroleum ether. The combined petroleum ether mixture was dried over MgSO_4 . The solution was concentrated and immediately purified by column chromatography (petroleum ether) to afford compound **4a** (1.502 g, 60%): Oil; ^1H NMR (300 MHz, CDCl_3) 7.41-7.52 (m, 2 H), 7.18-7.28 (m, 3 H), 6.00 (t, $J = 6.5$ Hz, 1 H), 4.73 (d, $J = 6.6$ Hz, 2 H); ^{13}C NMR (75.4 MHz, CDCl_3) 207.5, 131.6, 130.4, 129.0, 126.9, 77.1, 76.9; IR (neat) $\nu(\text{cm}^{-1}) = 1942, 1577, 1476, 1438$; MS (EI) m/z (%): 196(M^+ , 47.23), 115(100); Elemental Analysis: Calcd for $\text{C}_9\text{H}_8\text{Se}$: C 55.42, H 4.13. Found: C 55.45, H 4.21.

Iodohydroxylation of 1,2-allenyl organoselenides with I_2 and H_2O . Typical Procedure E. Synthesis of Z-2-iodo-3-phenylseleno-2-propen-1-ol (Z-11a).

A solution of phenyl 1,2-propadienyl arylselenide **4a** (100 mg, 0.5 mmol) in 1 mL of MeCN was added to a solution of iodine (508 mg, 2 mmol) in acetonitrile (7 mL) with water (2 mL) under a N_2 atmosphere. After being stirred at 0 °C for about 16 hours,

saturated aqueous solution of Na₂S₂O₃ was added. The mixture was extracted with ether and dried over MgSO₄. Evaporation and column chromatography on silica gel (petroleum ether/ethyl acetate =5:1) afforded **Z-11a** (119 mg, 69%). Oil: ¹H NMR (300 MHz, CDCl₃) 7.57-7.60 (m, 2 H), 7.43-7.44 (t, *J* = 1.4 Hz, 1 H), 7.31-7.34 (m, 3 H), 4.21 (d, *J* = 4.8 Hz, 2 H), 2.66 (t, *J* = 4.8 Hz, 1 H); ¹³C NMR (75 MHz, CDCl₃) 133.6, 133.1, 129.4, 129.2, 128.1, 104.8, 71.7; IR (neat) ν(cm⁻¹) = 3349, 1577, 1477, 1438, 1070, 1021, 999, 789, 739, 690; MS (EI) *m/z* (%): 340 (M⁺, 86.43), 157 (100); HRMS Calcd for C₉H₉IOSe (M⁺): 339.88633 Found: 339.88904.

The following compounds were prepared according to Typical Procedure B.

(1) Z-3-Iodo-1-(phenylsulfanyl)prop-1-en-3-ol (Z-6b). The reaction of **2b** (75 mg, 0.5 mmol) and I₂ (253 mg, 1.0 mmol) in acetone/H₂O (totally 4:1, 10 mL) afforded 61mg (41%) of **Z-6b** (eluent: petroleum ether/ethyl acetate = 5:1). Oil; ¹H NMR (300 MHz, CDCl₃) 7.17-7.36 (m, 5 H), 6.98 (t, *J* = 1.2 Hz, 1 H), 4.19 (s, 2 H), 2.68 (bs, 1 H); ¹³C NMR (75.4 MHz, CDCl₃) 134.3, 133.4, 130.7, 129.2, 127.7, 101.1, 70.9; IR (neat) ν(cm⁻¹) = 3348, 3.57, 1581, 1477, 1439; MS (EI) *m/z* (%): 292 (M⁺, 21.11), 43 (100); HRMS Calcd for C₁₀H₁₁IOS (M⁺): 291.94188. Found: 291.94205.

(2) Z-3-Iodo-4-(phenylsulfanyl)but-3-en-2-ol (Z-6c). The reaction of **2c** (163 mg, 1.0 mmol) and I₂ (507 mg, 2.0 mmol) in acetone/H₂O (totally 4:1, 10 mL) afforded 186 mg (61%) of **Z-6c** (eluent: petroleum ether/ethyl acetate = 5:1). Oil; ¹H NMR (300 MHz, CDCl₃) 7.41-7.44 (m, 2 H), 7.25-7.35 (m, 3 H), 7.07 (s, 1 H), 4.03 (q, *J* = 6.0 Hz, 1 H), 2.71 (bs, 1 H), 1.32 (d, *J* = 6.0 Hz, 3 H); ¹³C NMR (75.4 MHz, CDCl₃)

133.5, 133.3, 130.8, 129.2, 127.7, 109.8, 73.9, 23.8; IR (neat) $\nu(\text{cm}^{-1})$ = 3373, 2976, 2926, 1580, 1479, 1439, 1369, 1126, 1069, 817; MS (EI) m/z (%): 306 (M^+ , 49.30), 69 (100); HRMS Calcd for $C_{10}H_{11}IOS$ (M^+): 305.95753. Found: 305.95662.

(3) Z-2-Iodo-4-methyl-1-(phenylsulfanyl)pent-1-en-3-ol (Z-6d). The reaction of **2d** (95 mg, 0.5 mmol) and I_2 (253 mg, 1.0 mmol) in acetone/ H_2O (totally 4:1, 10 mL) afforded 93 mg (56%) of **Z-6d** (eluent: petroleum ether/ethyl acetate = 20:1). Oil; ^1H NMR (300 MHz, $CDCl_3$) 7.42-7.46 (m, 2 H), 7.28-7.37 (m, 3 H), 7.04 (t, J = 0.9 Hz, 1 H), 3.28 (d, J = 8.1 Hz, 1 H), 1.82-1.93 (m, 2 H), 1.05 (d, J = 6.3 Hz, 3 H), 0.84 (d, J = 6.9 Hz, 3 H); ^{13}C NMR (75.4 MHz, $CDCl_3$) 134.6, 133.8, 130.8, 129.3, 127.7, 108.8, 83.5, 33.9, 19.2, 17.8; IR (neat) $\nu(\text{cm}^{-1})$ = 3402, 2961, 2929, 2871, 1581, 1478, 1468, 1440, 1385, 1070, 1025, 864, 809; MS (EI) m/z (%): 334 (M^+ , 19.82), 291 (100); HRMS Calcd for $C_{12}H_{15}IOS$ (M^+): 333.98883. Found: 333.99041.

(4) Z-2-Iodo-1-(phenylsulfanyl)hept-1-en-3-ol (Z-6e). The reaction of **2e** (50 mg, 0.25 mmol) and I_2 (127 mg, 0.5 mmol) in acetone/ H_2O (totally 4:1, 10 mL) afforded 63 mg (74%) of **Z-6e** (eluent: petroleum ether/ethyl acetate = 5:1). Oil; ^1H NMR (300 MHz, $CDCl_3$) 7.18-7.37 (m, 5 H), 6.98 (s, 1 H), 3.64 (t, J = 6.5 Hz, 1 H), 2.01 (bs, 1 H), 1.54 (q, J = 6.9 Hz, 2 H), 1.18-1.28 (m, 4 H), 0.83 (t, J = 6.9 Hz, 3 H); ^{13}C NMR (75.4 MHz, $CDCl_3$) 134.2, 133.7, 130.8, 129.3, 127.7, 109.5, 77.9, 36.9, 29.3, 22.4, 14.0; IR (neat) $\nu(\text{cm}^{-1})$ = 3375, 2955, 2928, 2857, 1581, 1479, 1465, 1440, 1025, 825; MS (EI) m/z (%): 348 (M^+ , 1.28), 41 (100); HRMS Calcd for $C_{13}H_{17}IOS$ (M^+): 348.00448. Found: 348.00211.

(5) Z-2-Iodo-1-(phenylsulfanyl)dec-1-en-3-ol (Z-6f). The reaction of **2f** (62 mg, 0.25

mmol) and I₂ (127 mg, 0.5 mmol) in acetone/H₂O (totally 4:1, 10 mL) afforded 66 mg (64%) of **Z-6f** (eluent: petroleum ether/ethyl acetate = 5:1). Oil; ¹H NMR (300 MHz, CDCl₃) 7.42-7.45 (m, 2 H), 7.26-7.37 (m, 3 H), 7.06 (d, *J* = 0.6 Hz, 1 H), 3.71 (t, *J* = 6.5 Hz, 1 H), 1.95 (bs, 1 H), 1.62 (t, *J* = 6.9 Hz, 2 H), 1.22-1.29 (m, 10 H), 0.88 (t, *J* = 6.8 Hz, 3 H); ¹³C NMR (75.4 MHz, CDCl₃) 134.5, 134.0, 131.2, 129.6, 128.0, 110.0, 78.3, 37.5, 32.0, 29.5, 29.4, 25.5, 22.9, 14.4; IR (neat) ν(cm⁻¹) = 3373, 2925, 2855, 1581, 1479, 1440, 1024, 823; MS (EI) *m/z* (%): 390 (M⁺, 24.33), 43 (100); HRMS Calcd for C₁₆H₂₃IOS (M⁺): 390.05143. Found: 390.05517.

(6) Z-3-Iodo-1-phenyl-4-(phenylsulfanyl)but-3-en-2-ol (Z-6g). The reaction of **2g** (59 mg, 0.25 mmol) and I₂ (128 mg, 0.5 mmol) in acetone/H₂O (totally 4:1, 10 mL) afforded 61 mg (65%) of **Z-6g** (eluent: petroleum ether/ethyl acetate = 5:1). Oil; ¹H NMR (300 MHz, CDCl₃) 7.22-7.36 (m, 10 H), 6.85 (d, *J* = 0.6 Hz, 1 H), 4.03 (t, *J* = 6.8 Hz, 1 H), 2.88-3.03 (m, 2 H), 2.15 (bs, 1 H); ¹³C NMR (75.4 MHz, CDCl₃) 136.8, 134.7, 133.7, 130.7, 129.6, 129.2, 128.5, 127.6, 126.7, 107.7, 79.0, 43.3; IR (neat) ν(cm⁻¹) = 3409, 3059, 3026, 2923, 2855, 1723, 1581, 1478, 1440, 1374, 1247, 1085, 1042, 826; MS (EI) *m/z* (%): 382 (M⁺, 5.59), 291 (100); HRMS Calcd for C₁₆H₁₅IOS (M⁺): 381.98883. Found: 381.98958.

(7) Z-3-Iodo-2-methyl-4-(phenylsulfanyl)but-3-en-2-ol (Z-6h). The reaction of **2h** (52 mg, 0.3 mmol) and I₂ (153 mg, 0.6 mmol) in acetone/H₂O (totally 4:1, 10 mL) afforded 59 mg (62%) of **Z-6h** (eluent: petroleum ether/ethyl acetate = 20:1). Oil; ¹H NMR (300 MHz, CDCl₃) 7.44-7.47 (m, 2 H), 7.31-7.36 (m, 3 H), 7.04 (s, 1 H), 2.04 (bs, 1 H), 1.51 (s, 6 H); ¹³C NMR (75.4 MHz, CDCl₃) 134.0, 132.4, 130.9,

129.3, 127.7, 113.6, 75.0, 29.7; IR (neat) $\nu(\text{cm}^{-1})$ = 3403, 2979, 2928, 1582, 1479, 1440, 1364, 1207, 1167, 925, 860; MS (EI) m/z (%): 320 (M^+ , 51.98), 305 (100); HRMS Calcd for $C_{11}H_{13}IOS$ (M^+): 319.97318. Found: 319.97130.

(8) Z-2-Iodo-3-methyl-1-(phenylsulfanyl)pent-1-en-3-ol (Z-6i). The reaction of **2i** (96 mg, 0.5 mmol) and I_2 (257 mg, 1.0 mmol) in acetone/ H_2O (totally 4:1, 10 mL) afforded 158 mg (94%) of **Z-6i** (eluent: petroleum ether/ethyl acetate = 20:1). Oil; ^1H NMR (300 MHz, $CDCl_3$) 7.43-7.46 (m, 2 H), 7.28-7.36 (m, 3 H), 7.05 (s, 1 H), 2.10 (s, 1 H), 1.67-1.82 (m, 2 H), 1.48 (s, 3 H), 0.85 (t, J = 7.4 Hz, 3 H); ^{13}C NMR (75.4 MHz, $CDCl_3$) 134.1, 132.9, 130.6, 129.2, 127.5, 111.9, 77.4, 34.5, 27.0, 8.1; IR (neat) $\nu(\text{cm}^{-1})$ = 3425, 2967, 2929, 2876, 1581, 1478, 1457, 1440, 1374, 1159, 1121, 1024, 947, 911, 860; MS (EI) m/z (%): 334 (M^+ , 19.13), 305 (100); HRMS Calcd for $C_{12}H_{15}IOS$ (M^+): 333.98883. Found: 333.98984.

(9) Z-2-Iodo-3, 5-dimethyl-1-(phenylsulfanyl)hex-1-en-3-ol (Z-6j). The reaction of **2j** (109 mg, 0.5 mmol) and I_2 (254 mg, 1.0 mmol) in acetone/ H_2O (totally 4:1, 10 mL) afforded 96 mg (53%) of **Z-6j** (eluent: petroleum ether/ethyl acetate = 20:1). Oil; ^1H NMR (300 MHz, $CDCl_3$) 7.36-7.39 (m, 2 H), 7.20-7.29 (m, 3 H), 7.01 (s, 1 H), 1.76 (s, 1 H), 1.61-1.70 (m, 2H), 1.49-1.54 (m, 1H), 1.43 (s, 3 H), 0.90 (d, J = 3.3 Hz, 3 H), 0.87 (d, J = 3.3 Hz, 3 H); ^{13}C NMR (75.4 MHz, $CDCl_3$) 134.2, 132.5, 130.7, 129.2, 127.5, 112.5, 77.9, 49.9, 28.9, 24.4, 24.3, 24.2; IR (neat) $\nu(\text{cm}^{-1})$ = 3447, 2953, 2927, 2868, 1582, 1568, 1478, 1466, 1440, 1366, 1158, 1088, 877, 855; MS (EI) m/z (%): 362 (M^+ , 14.43), 305 (100); HRMS Calcd for $(C_{13}H_{16}IOS) (M^+ - CH_3)$: 347.99666. Found: 347.00060.

(10) Z-2-Iodo-3, 4, 4-trimethyl-1-(phenylsulfanyl)pent-1-en-3-ol (Z-6k). The reaction of **2k** (108 mg, 0.5 mmol) and I₂ (257 mg, 1.0 mmol) in acetone/H₂O (totally 4:1, 10 mL) afforded 153 mg (85%) of **Z-6k** (eluent: petroleum ether/ethyl acetate = 20:1). Oil; ¹H NMR (300 MHz, CDCl₃) 7.34-7.37 (m, 2 H), 7.19-7.27 (m, 3 H), 7.01 (s, 1 H), 1.77 (s, 1 H), 1.47 (s, 3 H), 0.98 (s, 9 H); ¹³C NMR (75.4 MHz, CDCl₃) 135.9, 134.4, 130.5, 129.2, 127.4, 108.1, 79.5, 39.4, 26.6, 26.4; IR (neat) ν(cm⁻¹) = 3478, 2957, 2872, 1582, 1558, 1479, 1440, 1371, 1073, 901, 861; MS (EI) m/z (%): 362 (M⁺, 1.02), 305 (100); HRMS Calcd for C₁₄H₁₉IOS (M⁺): 362.02013. Found: 362.01818.

(11) Z-3-Ethyl-2-iodo-1-(phenylsulfanyl)pent-1-en-3-ol (Z-6l). The reaction of **2l** (102 mg, 0.5 mmol) and I₂ (256 mg, 1.0 mmol) in acetone/H₂O (totally 4:1, 10 mL) afforded 126 mg (72%) of **Z-6l** (eluent: petroleum ether/ethyl acetate = 5:1). Oil; ¹H NMR (300 MHz, CDCl₃) 7.43-7.46 (m, 2 H), 7.27-7.37 (m, 3 H), 7.08 (s, 1 H), 1.83-1.93 (m, 2 H), 1.79 (s, 1H), 1.61-1.69 (m, 2 H), 0.87 (t, *J* = 7.4 Hz, 6 H); ¹³C NMR (75.4 MHz, CDCl₃) 134.4, 133.5, 130.5, 129.2, 127.4, 109.3, 79.9, 32.6, 7.4; IR (neat) ν(cm⁻¹) = 3470, 2967, 2933, 2877, 2852, 1581, 1478, 1458, 1440, 1376, 1158, 1088, 1024, 961, 865; MS (EI) m/z (%): 348 (M⁺, 20.01), 319 (100); HRMS Calcd for C₁₃H₁₇IOS (M⁺): 348.00448. Found: 348.00385.

(12) Z-2-Iodo-1-(phenylsulfanyl)-3-butylhept-1-en-3-ol (Z-6m). The reaction of **2m** (130 mg, 0.5 mmol) and I₂ (256 mg, 1.0 mmol) in acetone/H₂O (totally 4:1, 10 mL) afforded 188 mg (93%) of **Z-6m** (eluent: petroleum ether/ethyl acetate = 20:1). Oil; ¹H NMR (300 MHz, CDCl₃) 7.35-7.38 (m, 2 H), 7.20-7.29 (m, 3 H), 6.99 (s, 1 H),

1.73-1.79 (m, 2 H), 1.68 (s, 1H), 1.52-1.58 (m, 2 H), 1.14-1.26 (m, 8 H), 0.84 (t, J = 6.9 Hz, 6 H); ^{13}C NMR (75.4 MHz, CDCl_3) 134.5, 132.9, 130.5, 129.2, 127.4, 110.0, 79.6, 40.1, 3.1, 22.8, 14.0; IR (Neat) $\nu(\text{cm}^{-1})$ = 3475, 2955, 2870, 1723, 1582, 1570, 1478, 1465, 1440, 1380, 1252, 1089, 865; MS (EI) m/z (%): 404 (M^+ , 4.96), 347 (100); HRMS Calcd for $\text{C}_{17}\text{H}_{25}\text{IOS}$ (M^+): 404.06708. Found: 404.06309.

(13) Z-2-Iodo-1-(phenylsulfanyl)-5-pentylocta-1-en-3-ol (Z-6n). The reaction of **2n** (114 mg, 0.4 mmol) and I_2 (205 mg, 0.8 mmol) in acetone/ H_2O (totally 4:1, 10 mL) afforded 136 mg (80%) of **Z-6n** (eluent: petroleum ether/ethyl acetate = 20:1). Oil; ^1H NMR (300 MHz, CDCl_3) 7.36-7.39 (m, 2 H), 7.19-7.30 (m, 3 H), 6.99 (s, 1 H), 1.73-1.81 (m, 2 H), 1.65 (s, 1 H), 1.48-1.58 (m, 2 H), 1.19-1.29 (m, 12 H), 0.82 (t, J = 6.9 Hz, 6 H); ^{13}C NMR (75.4 MHz, CDCl_3) 134.5, 132.8, 130.4, 129.2, 127.3, 110.2, 79.6, 40.3, 31.9, 22.6, 22.5, 14.0; IR (neat) $\nu(\text{cm}^{-1})$ = 3473, 2928, 2859, 1673, 1581, 1478, 1466, 1440, 1378, 1025, 872; MS (EI) m/z (%): 432 (M^+ , 5.36), 361 (100); HRMS Calcd for $\text{C}_{19}\text{H}_{29}\text{IOS}$ (M^+): 432.09838 Found: 432.09890.

(14) Z-1-(1'-cyclohexenyl)-1-iodo-2-(phenylsulfanyl) ethene (Z-9p). The reaction of **2p** (108 mg, 0.5 mmol) and I_2 (256 mg, 1.0 mmol) in acetone/ H_2O (totally 4:1, 10 mL) afforded 146 mg (85%) of **Z-9p** (eluent: petroleum ether). Oil; ^1H NMR (300 MHz, CDCl_3) 7.43-7.46 (m, 2 H), 7.24-7.35 (m, 3 H), 6.87 (s, 1 H), 6.08 (t, J = 4.1 Hz, 1 H), 2.28-2.31 (m, 2 H), 2.13-2.17 (m, 2H), 1.53-1.68 (m, 4 H); ^{13}C NMR (75.4 MHz, CDCl_3) 135.8, 134.6, 132.3, 131.5, 130.5, 129.2, 127.5, 105.9, 27.1, 26.2, 22.8, 22.1; IR (neat) $\nu(\text{cm}^{-1})$ = 3056, 2928, 2856, 1581, 1477, 1439, 1024; MS (EI) m/z (%): 342 (M^+ , 59.10), 218 (100); HRMS Calcd for $\text{C}_{14}\text{H}_{15}\text{IS}$ (M^+): 341.99392. Found:

341.99254.

(15) Z-1-(1'-cyclohexenyl)-1-bromo-2-(phenylsulfanyl) ethene (Z-11p). The reaction of **2p** (220 mg, 1.0 mmol), Br₂ (324 mg, 2.0 mmol) in the solution of acetone/H₂O (totally 4:1, 15 mL) afforded 186 mg (63%) of **Z-11p** (eluent: petroleum ether). Oil; ¹H NMR (300 MHz, CDCl₃) 7.45 (d, *J* = 7.2 Hz, 2 H), 7.22-7.40 (m, 3 H), 6.76 (s, 1 H), 6.24 (t, *J* = 4.1, 1 H), 2.16-2.30 (m, 4 H), 1.64-1.72 (m, 2H), 1.51-1.64 (m, 2H); ¹³C NMR (75.4 MHz, CDCl₃) 134.6, 133.1, 130.4, 129.5, 129.2, 127.4, 125.0, 124.2, 26.4, 25.8, 22.6, 21.9; IR (neat) ν(cm⁻¹) = 3057, 1582, 1478, 1439; MS (EI) *m/z* (%): 296 (M⁺ (⁸¹Br), 12.23), 294 (M⁺ (⁷⁹Br), 10.07), 109(100); Elemental Analysis Calcd for C₁₄H₁₅BrOS: C: 56.96, H: 5.12. Found: C: 57.23, H: 5.23.

The following compounds were prepared according to Typical Procedure C.

(1) Synthesis of Z-2-Bromo-3-(phenylsulfanyl)prop-2-en-1-ol (Z-10b). The reaction of **2c** (75 mg, 0.5 mmol) and Br₂ (2.0 mL, 0.5 mol/L in CH₃CN, 1.0 mmol) in MeCN/H₂O (totally 4:1, 10 mL) at 25°C afforded 110 mg (60%) of **Z-10c** (eluent: petroleum ether/ethyl acetate = 8:1). Oil; ¹H NMR (300 MHz, CDCl₃) 7.15-7.38 (m, 5 H), 6.84 (s, 1 H), 4.21 (d, *J* = 4.5 Hz, 2 H), 2.55 (t, *J* = 4.5 Hz, 1 H); ¹³C NMR (75.4 MHz, CDCl₃) 133.5, 130.7, 129.2, 128.3, 127.7, 121.5, 67.8; IR (neat) ν(cm⁻¹) = 3338, 1583; MS (EI) *m/z* (%): 246 (M⁺ (⁸¹Br), 100), 244 (M⁺ (⁷⁹Br), 99.09); HRMS Calcd for C₉H₉⁷⁹BrOS (M⁺): 243.95575. Found: 243.95581.

The NOE of compound **Z-10b**

(2) **Z-3-Bromo-4-(phenylsulfanyl)but-3-en-2-ol (Z-10c)**. The reaction of **2c** (114 mg, 0.7 mmol), Br₂ (2.8 mL, 0.5 mol/L in CH₃CN, 1.4 mmol), and Na₂CO₃ (75 mg, 0.7 mmol) in MeCN/H₂O (totally 4:1, 12.5 mL) afforded 110 mg (60%) of **Z-10c** (eluent: petroleum ether/ethyl acetate = 8:1). Oil; ¹H NMR (300 MHz, CDCl₃) 7.17-7.42 (m, 5 H), 6.85 (s, 1 H), 4.27-4.40 (m, 1 H), 2.03 (d, *J* = 5.1 Hz, 1 H), 1.33 (d, *J* = 6.1 Hz, 3 H); ¹³C NMR (75.4 MHz, CDCl₃) 133.7, 130.8, 129.3, 127.7, 127.2, 127.1, 72.1, 22.4; IR (neat) ν(cm⁻¹) = 3357, 3058, 2979, 1582, 1479, 1440; MS (EI) *m/z* (%): 260 (M⁺ (⁸¹Br), 81.16), 258 (M⁺ (⁷⁹Br), 80.94), 179 (100); HRMS Calcd for C₁₀H₁₁⁷⁹BrOS (M⁺): 257.97140. Found: 257.97045.

(3) Z-2-bromo-4-methyl-1-(phenylsulfanyl)pent-1-en-3-ol (Z-10d). The reaction of **2d** (133 mg, 0.7 mmol), Br₂ (2.8 mL, 0.5 mol/L in CH₃CN, 1.4 mmol), and Na₂CO₃ (75 mg, 0.7 mmol) in MeCN/H₂O (totally 4:1, 12.5 mL) afforded 122 mg (61%) of **Z-10d** (eluent: petroleum ether/ethyl acetate = 8:1). Oil; ¹H NMR (300 MHz, CDCl₃) 7.16-7.39 (m, 5 H), 6.80 (s, 1 H), 3.67 (dd, *J* = 6.4 Hz and 1.2 Hz, 1 H), 2.02 (d, *J* = 6.4 Hz, 1 H), 1.80-1.96 (m, 1 H), 0.95 (d, *J* = 6.6 Hz, 3 H), 0.80 (d, *J* = 6.3 Hz, 3 H); ¹³C NMR (75.4 MHz, CDCl₃) 133.8, 130.6, 129.2, 128.4, 127.6, 125.9, 81.7, 32.6, 19.1, 17.8; IR (neat) ν(cm⁻¹) = 3398, 3059, 2960, 1582, 1479, 1440; MS (EI) *m/z* (%): 288(M⁺ (⁸¹Br), 18.13), 286(M⁺ (⁷⁹Br), 17.81), 243(100); HRMS Calcd for C₁₂H₁₅⁷⁹BrOS (M⁺): 286.00270. Found: 285.99995.

(3) Z-2-Bromo-1-(phenylsulfanyl)dec-1-en-3-ol (Z-10f). The reaction of **2f** (123 mg, 0.5 mmol), Br₂ (2.0 mL, 0.5 mol/L in CH₃CN, 1.0 mmol), and Na₂CO₃ (53 mg, 0.5 mmol) in MeCN/H₂O (totally 4:1, 10 mL) afforded 122 mg (72%) of **Z-10f** (eluent: petroleum ether/ethyl acetate = 8:1). Oil; ¹H NMR (300 MHz, CDCl₃) 7.16-7.39 (m, 5 H), 6.81 (s, 1 H), 4.02-4.12 (m, 1 H), 2.18 (d, *J* = 5.1 Hz, 1 H), 1.51-1.71 (m, 2 H), 1.11-1.33 (m, 10 H), 0.80 (t, *J* = 6.8 Hz, 3 H); ¹³C NMR (75.4 MHz, CDCl₃) 133.7, 130.7, 129.2, 127.9, 127.6, 126.6, 76.2, 35.7, 31.7, 29.2, 29.1, 25.2, 22.6, 14.1; IR (neat) ν(cm⁻¹) = 3364, 2926, 1583, 1479, 1440; MS (EI) *m/z* (%): 344 (M⁺ (⁸¹Br), 1.75), 342 (M⁺ (⁷⁹Br), 1.66), 43 (100); HRMS Calcd for C₁₆H₂₃⁷⁹BrOS (M⁺): 342.06530. Found: 342.06523.

(4) Z-3-Bromo-2-methyl-4-(phenylsulfanyl)but-3-en-2-ol (Z-10h). The reaction of **2h** (50 mg, 0.3 mmol), Br₂ (1.2 mL, 0.5 mol/L in CH₃CN, 0.6 mmol), and Na₂CO₃

(32 mg, 0.3 mmol) in MeCN/H₂O (totally 4:1, 7.5 mL) afforded 67 mg (86%) of **Z-10h** (eluent: petroleum ether/ethyl acetate = 8:1). Oil; ¹H NMR (300 MHz, CDCl₃) 7.18-7.41 (m, 5 H), 6.88 (s, 1 H), 1.68 (bs, 1 H), 1.43 (s, 6 H); ¹³C NMR (75.4 MHz, CDCl₃) 134.0, 130.8, 130.4, 129.2, 127.7, 125.8, 74.4, 29.0; IR (neat) ν(cm⁻¹) = 3388, 3057, 2980, 1582, 1479, 1440; MS (EI) m/z (%): 274 (M⁺ (⁸¹Br), 23.04), 272 (M⁺ (⁷⁹Br), 23.09), 43 (100); HRMS Calcd for C₁₁H₁₃⁷⁹BrOS (M⁺): 271.98705. Found: 271.99086.

(5) Z-2-Bromo-3-methyl-1-(phenylsulfanyl)pent-1-en-3-ol (Z-10i). The reaction of **2i** (95 mg, 0.5 mmol), Br₂ (2.0 mL, 0.5 mol/L in CH₃CN, 1.0 mmol), and Na₂CO₃ (53 mg, 0.5 mmol) in MeCN/H₂O (totally 4:1, 10 mL) afforded 120 mg (86%) of **Z-10i** (eluent: petroleum ether/ethyl acetate = 8:1). Oil; ¹H NMR (300 MHz, CDCl₃) 7.17-7.40 (m, 5 H), 6.88 (s, 1 H), 1.85 (s, 1 H), 1.56-1.83 (m, 2 H), 1.40 (s, 3 H), 0.80 (t, *J* = 7.7 Hz, 3 H); ¹³C NMR (75.4 MHz, CDCl₃) 134.2, 130.7, 129.2, 129.0, 127.6, 126.5, 77.2, 33.7, 26.8, 8.1; IR (neat) ν(cm⁻¹) = 3428, 3058, 2971, 1583, 1479, 1440; MS (EI) m/z (%): 288 (M⁺ (⁸¹Br), 15.17), 286 (M⁺ (⁷⁹Br), 14.81), 126 (100); HRMS Calcd for C₁₂H₁₅⁷⁹BrOS (M⁺): 286.00270. Found: 286.00071.

(6) Z-2-Bromo-3, 5-dimethyl-1-(phenylsulfanyl)hex-1-en-3-ol (Z-10j). The reaction of **2j** (109 mg, 0.5 mmol), Br₂ (2.0 mL, 0.5 mol/L in CH₃CN, 1.0 mmol), and Na₂CO₃ (53 mg, 0.5 mmol) in MeCN/H₂O (totally 4:1, 10 mL) afforded 108 mg (68%) of **Z-10j** (eluent: petroleum ether/ethyl acetate = 25:1). Oil; ¹H NMR (300 MHz, CDCl₃) 7.44 (d, *J* = 6.6 Hz, 2 H), 7.22-7.39 (m, 3 H), 7.00 (s, 1 H), 1.89 (s, 1 H), 1.64-1.82 (m, 2 H), 1.51-1.62 (m, 1 H), 1.48 (s, 3 H), 0.96 (d, *J* = 6.9 Hz, 3 H), 0.95 (d, *J* = 7.2

Hz, 3H); ^{13}C NMR (75.4 MHz, CDCl_3) 134.2, 130.6, 129.6, 129.2, 127.5, 126.0, 77.5, 49.1, 28.4, 24.4, 24.23, 24.17; IR (neat) $\nu(\text{cm}^{-1})$ = 3445, 3058, 2954, 1583, 1479, 1440; MS (EI) m/z (%): 316 (M^+ (^{81}Br), 11.88), 314 (M^+ (^{79}Br), 11.64), 257 (100); HRMS Calcd for $\text{C}_{13}\text{H}_{16}^{79}\text{BrOS}$ ($\text{M}^+ - \text{CH}_3$): 299.01052. Found: 299.00854.

(7) Z-2-Bromo-3,4,4-trimethyl-1-(phenylsulfanyl)pent-1-en-3-ol (Z-10k). The reaction of **2k** (108 mg, 0.5 mmol), Br_2 (2.0 mL, 0.5 mol/L in CH_3CN , 1.0 mmol), and Na_2CO_3 (53 mg, 0.5 mmol) in $\text{MeCN}/\text{H}_2\text{O}$ (totally 4:1, 10 mL) afforded 133 mg (85%) of **Z-10k** (eluent: petroleum ether/ethyl acetate = 25:1). Oil; ^1H NMR (300 MHz, CDCl_3) 7.16-7.39 (m, 5 H), 6.93 (s, 1 H), 1.78 (bs, 1 H), 1.45 (s, 3 H), 0.97 (s, 9 H); ^{13}C NMR (75.4 MHz, CDCl_3) 134.4, 130.5, 129.2, 129.1, 127.4, 127.0, 80.4, 39.0, 26.2, 24.9; IR (neat) $\nu(\text{cm}^{-1})$ = 3473, 3058, 2959, 1581, 1479, 1440; MS (EI) m/z (%): 316 (M^+ (^{81}Br), 4.38), 314 (M^+ (^{79}Br), 4.37), 43 (100); HRMS Calcd for $\text{C}_{13}\text{H}_{16}\text{BrOS}$ ($\text{M}^+ - \text{CH}_3$): 299.01052. Found: 299.01070.

(8) Z-3-Ethyl-2-bromo-1-(phenylsulfanyl)pent-1-en-3-ol (Z-10l). The reaction of **2l** (123 mg, 0.6 mmol), Br_2 (2.4 mL, 0.5 mol/L in CH_3CN , 1.2 mmol), and Na_2CO_3 (64 mg, 0.6 mmol) in $\text{MeCN}/\text{H}_2\text{O}$ (totally 4:1, 12.5 mL) afforded 127 mg (70%) of **Z-10l** (eluent: petroleum ether/ethyl acetate = 8:1). Oil; ^1H NMR (300 MHz, CDCl_3) 7.14-7.49 (m, 5 H), 6.98 (s, 1 H), 1.72-1.88 (m, 2 H), 1.68 (bs, 1 H), 1.48-1.62 (m, 2 H), 0.90 (t, J = 6.8 Hz, 6 H); ^{13}C NMR (75.4 MHz, CDCl_3) 134.4, 130.4, 129.2, 127.4, 127.01, 126.96, 80.0, 32.0, 7.5; IR (neat) $\nu(\text{cm}^{-1})$ = 3470, 3058, 2969, 1582, 1479, 1440; MS (EI) m/z (%): 302 (M^+ (^{81}Br), 14.19), 300 (M^+ (^{79}Br), 13.98), 271 (100); HRMS Calcd for $\text{C}_{13}\text{H}_{17}^{79}\text{BrOS}$ (M^+): 300.01835. Found: 300.02108.

(9) Z-2-Bromo-1-(phenylsulfanyl)-3-butylhept-1-en-3-ol (Z-10m). The reaction of **2m** (130 mg, 0.5 mmol), Br₂ (2.0 mL, 0.5 mol/L in CH₃CN, 1.0 mmol), and Na₂CO₃ (53 mg, 0.5 mmol) in MeCN/H₂O (totally 4:1, 10 mL) afforded 97 mg (54%) of **Z-10m** (eluent: petroleum ether/ethyl acetate = 20:1). Oil; ¹H NMR (300 MHz, CDCl₃) 7.16-7.39 (m, 5 H), 6.89 (s, 1 H), 1.71-1.84 (m, 2 H), 1.70 (s, 1 H), 1.45-1.58 (m, 2 H), 1.10-1.34 (m, 8 H), 0.84 (t, *J* = 6.8 Hz, 6 H); ¹³C NMR (75.4 MHz, CDCl₃) 134.5, 130.4, 129.2, 127.8, 127.4, 126.4, 79.6, 39.4, 25.2, 22.8, 14.0; IR (Neat) ν(cm⁻¹) = 3474, 3059, 2956, 1583, 1466, 1440; MS (EI) *m/z* (%): 358 (M⁺ (⁸¹Br), 2.38), 356 (M⁺ (⁷⁹Br), 2.47), 126 (100); HRMS Calcd for C₁₇H₂₅⁷⁹BrOS (M⁺): 356.08095. Found: 356.08416.

(10) Z-2-Bromo-1-(phenylsulfanyl)-5-pentyloct-1-en-3-ol (Z-10n). The reaction of **2n** (144 mg, 0.5 mmol), Br₂ (2.0 mL, 0.5 mol/L in CH₃CN, 1.0 mmol), and Na₂CO₃ (53 mg, 0.5 mmol) in MeCN/H₂O (totally 4:1, 10 mL) afforded 130 mg (68%) of **Z-10n** (eluent: petroleum ether/ethyl acetate = 20:1). Oil; ¹H NMR (300 MHz, CDCl₃) 7.16-7.39 (m, 5 H), 6.89 (s, 1 H), 1.70-1.84 (m, 2 H), 1.69 (s, 1 H), 1.45-1.58 (m, 2 H), 1.16-1.33 (m, 12 H), 0.82 (t, *J* = 6.8 Hz, 6 H); ¹³C NMR (75.4 MHz, CDCl₃) 134.5, 130.4, 129.2, 127.9, 127.4, 126.4, 79.7, 39.7, 32.0, 22.7, 22.5, 14.1; IR (neat) ν(cm⁻¹) = 3481, 3059, 2959, 1583, 1479, 1440; MS (EI) *m/z* (%): 386 (M⁺ (⁸¹Br), 2.33), 384 (M⁺ (⁷⁹Br), 2.27), 43 (100); HRMS Calcd for C₁₇H₂₉⁷⁹BrOS (M⁺): 384.11225. Found: 384.11032.

(11) Z-2-Bromo-1-(4'-bromophenylsulfanyl)hept-1-en-3-ol (Z-10q). The reaction of **2q** (142 mg, 0.5 mmol), Br₂ (2.0 mL, 0.5 mol/L in CH₃CN, 1.0 mmol), and Na₂CO₃

(53 mg, 0.5 mmol) in MeCN/H₂O (totally 4:1, 10 mL) afforded 122 mg (64%) of **Z-10q** (eluent: petroleum ether/ethyl acetate = 8:1). Oil; ¹H NMR (300 MHz, CDCl₃) 7.38 (d, *J* = 6.6 Hz, 2 H), 7.21 (d, *J* = 6.6 Hz, 2 H), 6.76 (s, 1 H), 4.08 (q, *J* = 6.5 Hz, 1 H), 2.02 (bs, 1 H), 1.50-1.70 (m, 2 H), 1.12-1.32 (m, 4 H), 0.84 (t, *J* = 6.9 Hz, 3 H); ¹³C NMR (75.4 MHz, CDCl₃) 133.0, 132.3, 132.1, 127.5, 126.9, 121.8, 76.2, 35.5, 27.4, 22.4, 14.0; IR (neat) $\nu(\text{cm}^{-1})$ = 3362, 2955, 1591, 1473, 1387; MS (EI) *m/z* (%): 382 (*M*⁺ (2 ⁸¹Br), 8.48), 380 (*M*⁺ (⁸¹Br, ⁷⁹Br), 16.22), 378 (*M*⁺ (2 ⁷⁹Br), 8.07), 117 (100); HRMS Calcd for C₁₃H₁₆⁷⁹Br₂OS (*M*⁺): 377.92886. Found: 377.92910.

(12) Z-2-Bromo-3-(phenylsulfanyl)hept-2-en-1-ol (Z-10r). The reaction of **2r** (48 mg, 0.24 mmol), Br₂ (0.96 mL, 0.5 mol/L in CH₃CN, 0.48 mmol), and Na₂CO₃ (30 mg, 0.24 mmol) in MeCN/H₂O (totally 4:1, 15 mL) afforded 25 mg (35%) of **Z-10r** (eluent: petroleum ether/ethyl acetate = 8:1). Oil; ¹H NMR (300 MHz, CDCl₃) 7.30-7.51 (m, 5 H), 4.42 (s, 2 H), 2.20 (t, *J* = 8.0 Hz, 2 H), 2.01 (bs, 1 H), 1.28-1.44 (m, 2 H), 1.01-1.21 (m, 2 H), 0.73 (t, *J* = 7.1 Hz, 3 H); ¹³C NMR (75.4 MHz, CDCl₃) 140.8, 133.9, 132.1, 129.1, 128.4, 122.1, 64.8, 31.8, 31.5, 22.0, 13.6; IR (neat) $\nu(\text{cm}^{-1})$ = 3369, 3058, 1594, 1477, 1440; MS (EI) *m/z* (%): 302 (*M*⁺ (⁸¹Br), 83.33), 300 (*M*⁺ (⁷⁹Br), 85.89), 111 (100); Elemental Analysis Calcd for C₁₃H₁₇BrOS: C: 51.83, H: 5.69. Found: C: 52.09, H: 5.67.

(13) Z-2-Bromo-3-(phenylsulfanyl)but-2-en-1-ol (Z-10s). The reaction of **2s** (82 mg, 0.5 mmol), Br₂ (2.0 mL, 0.5 mol/L in CH₃CN, 1.0 mmol), and Na₂CO₃ (53 mg, 0.5 mmol) in MeCN/H₂O (totally 4:1, 10 mL) afforded 57 mg (44%) of **Z-10s** (eluent: petroleum ether/ethyl acetate = 20:1). Oil; ¹H NMR (300 MHz, CDCl₃) 7.47 (d, *J*

= 5.4 Hz, 2 H), 7.32-7.39 (m, 3 H), 4.43 (s, 2 H), 2.20 (bs, 1 H), 1.81 (s, 3 H); ^{13}C NMR (75.4 MHz, CDCl_3) 136.0, 134.7, 131.6, 129.1, 128.8, 119.1, 65.0, 19.3; IR (neat) $\nu(\text{cm}^{-1})$ = 3346, 3057, 1601, 1475, 1439; MS (EI) m/z (%): 260 ($\text{M}^+(\text{}^{81}\text{Br})$, 3.65), 258 ($\text{M}^+(\text{}^{79}\text{Br})$, 3.66), 43 (100); HRMS Calcd for $\text{C}_{10}\text{H}_{11}\text{}^{79}\text{BrOS}$ (M^+): 257.99140. Found: 257.97030.

The following compounds were prepared according to Typical Procedure D.

(1) 1,2-Propadienyl *p*-Tolyl Selenide (4b). The reaction of **3b** (2.076 g, 12.75 mmol), Na (0.210 g, 13.5 mmol), and EtOH (9.3 mL) afforded 1.223 g (59%) of **4b**. Oil; ^1H NMR (300 MHz, CDCl_3) 7.42 (d, J = 8.1 Hz, 2 H), 7.11 (d, J = 8.1 Hz, 2 H), 6.04 (t, J = 6.1 Hz, 1 H), 4.77 (d, J = 6.1 Hz, 2 H), 2.34 (s, 3 H); ^{13}C NMR (75.4 MHz, CDCl_3) 207.2, 137.3, 132.5, 129.9, 126.5, 77.8, 77.0, 21.1; IR (Neat) $\nu(\text{cm}^{-1})$ = 1942, 1489; MS (EI) m/z (%): 209 ($\text{M}^+(\text{}^{80}\text{Se})$, 44.60), 207 ($\text{M}^+(\text{}^{78}\text{Se})$, 26.91), 91(100); Elemental Analysis Calcd for $\text{C}_{10}\text{H}_{10}\text{}^{80}\text{Se}$: C 57.44, H 4.82. Found: C 57.57, H 5.08.

(2) 1,2-Propadienyl *o*-Tolyl Selenide (4c). The reaction of **3c** (2.288 g, 12.8 mmol), Na (0.212 g, 13.5 mmol), and EtOH (9.3 mL) afforded 1.468 g (64%) of **4c**. Oil; ^1H NMR (300 MHz, CDCl_3) 7.54 (d, J = 6.6 Hz, 1 H), 7.12-7.24 (m, 3 H), 6.02 (t, J = 6.3 Hz, 1 H), 4.81 (d, J = 6.3 Hz, 2 H), 2.42 (s, 3 H); ^{13}C NMR (75.4 MHz, CDCl_3) 208.1, 138.7, 131.6, 131.4, 130.0, 127.2, 126.6, 76.6, 76.3, 22.0; IR (neat) $\nu(\text{cm}^{-1})$ = 1942, 1465; MS (EI) m/z (%): 210 ($\text{M}^+(\text{}^{80}\text{Se})$, 50.65), 207 ($\text{M}^+(\text{}^{78}\text{Se})$, 31.85), 91 (100); Elemental Analysis Calcd for $\text{C}_{10}\text{H}_{10}\text{}^{80}\text{Se}$: C 57.44, H 4.82. Found: C 57.60, H 4.96.

(3) 1,2-Propadienyl *m*-Tolyl Selenide (4d). The reaction of **3d** (2.445 g, 12.75 mmol), Na (0.210 g, 13.5 mmol), and EtOH (9.5 mL) afforded 1.575 g (64%) of **4d**.

Oil; ^1H NMR (300 MHz, CDCl_3) 7.37 (s, 1 H), 7.33 (d, $J = 7.5$ Hz, 1 H), 7.20 (t, $J = 7.5$ Hz, 1 H), 7.10 (d, $J = 7.5$ Hz, 1 H), 6.09 (t, $J = 6.3$ Hz, 1 H), 4.82 (d, $J = 6.3$ Hz, 2 H), 2.36 (s, 3 H); ^{13}C NMR (75.4 MHz, CDCl_3) 207.6, 138.8, 132.2, 130.2, 128.9, 128.9, 127.9, 77.2, 76.8, 21.2; IR (neat) $\nu(\text{cm}^{-1}) = 1941, 1590, 1471$; MS (EI) m/z (%): 210 (M^+ (^{80}Se), 56.67), 208 (M^+ (^{78}Se), 28.72), 129 (100); Elemental Analysis Calcd for $\text{C}_{10}\text{H}_{10}\text{Se}$: C 57.44, H 4.82. Found: C 57.51, H 4.96.

(4) 1,2-Propadienyl 4'-Bromophenyl Selenide (4b). The reaction of **3e** (3.647 g, 12.8 mmol), Na (0.216 g, 13.5 mmol), and EtOH (9.3 mL) afforded 1.623 g (45%) of **4e**. Oil; ^1H NMR (300 MHz, CDCl_3) 7.40 (d, $J = 6.3$ Hz, 2 H), 7.35 (d, $J = 6.3$ Hz, 2 H), 6.03 (t, $J = 6.5$ Hz, 1 H), 4.81 (d, $J = 6.5$ Hz, 2 H); ^{13}C NMR (75.4 MHz, CDCl_3) 207.9, 133.3, 132.1, 129.4, 121.3, 77.2, 76.8; IR (neat) $\nu(\text{cm}^{-1}) = 1941, 1466, 1381$; MS (EI) m/z (%): 276 (M^+ (^{81}Br , ^{80}Se), 28.86), 274 (M^+ ($^{79}\text{Br}/^{80}\text{Se}$ or $^{81}\text{Br}/^{78}\text{Se}$), 40.37), 272 (M^+ (^{79}Br , ^{78}Se), 24.93), 195 (100); Elemental Analysis Calcd for $\text{C}_9\text{H}_7^{79}\text{Br}^{80}\text{Se}$: C 39.46, H 2.58. Found: C 39.61, H 2.70.

(5) 1,2-Propadienyl 4'-Chlorophenyl Selenide (4f). The reaction of **3f** (3.688 g, 12.8 mmol), Na (0.212 g, 13.5 mmol), and EtOH (9.5 mL) afforded 1.746 g (47%) of **4f**. Oil; ^1H NMR (300 MHz, CDCl_3) 7.44 (d, $J = 5.4$ Hz, 2 H), 7.25 (d, $J = 5.4$ Hz, 2 H), 6.03 (t, $J = 6.3$ Hz, 1 H), 4.81 (d, $J = 6.3$ Hz, 2 H); ^{13}C NMR (75.4 MHz, CDCl_3) 207.8, 133.3, 133.2, 129.2, 128.7, 77.3, 77.0; IR (neat) $\nu(\text{cm}^{-1}) = 1942, 1473, 1387$; MS (70 eV, EI) m/z (%): 232 (M^+ (^{37}Cl , ^{80}Se), 17.18), 230 (M^+ ($^{35}\text{Cl}/^{80}\text{Se}$ or $^{37}\text{Cl}/^{78}\text{Se}$), 40.12), 272 (M^+ (^{35}Cl , ^{78}Se), 20.87), 195 (100); Elemental Analysis Calcd for $\text{C}_9\text{H}_7^{35}\text{Cl}^{80}\text{Se}$: C 47.10, H 3.07. Found: C 47.16, H 3.26.

(6) 1,2-Propadienyl 4'-Methoxyphenyl Selenide (4g). The reaction of **3g** (2.583 g, 12.8 mmol), Na (0.227 g, 13.5 mmol), and EtOH (9.5 mL) afforded 0.499 g (19%) of **4g**. Oil; ^1H NMR (300 MHz, CDCl_3) 7.48 (d, $J = 6.9$ Hz, 2 H), 6.84 (d, $J = 6.9$ Hz, 2 H), 6.00 (t, $J = 6.3$ Hz, 1 H), 4.73 (d, $J = 6.3$ Hz, 2 H), 3.80 (s, 3 H); ^{13}C NMR (75.4 MHz, CDCl_3) 206.2, 159.3, 135.1, 119.8, 114.7, 78.8, 77.4, 55.1; IR (neat) $\nu(\text{cm}^{-1}) = 1941, 1590, 1490$; MS (EI) m/z (%): 226 (M^+ (^{80}Se), 47.08), 224 (M^+ (^{78}Se), 25.17), 187 (100); Elemental Analysis Calcd for $\text{C}_{10}\text{H}_{10}\text{O}^{80}\text{Se}$: C 53.36, H 4.48. Found: C: 53.00, H: 4.59.

(7) 1,2-Propadienyl Benzyl Selenide (4h). The reaction of **3h** (2.508 g, 12.8 mmol), Na (0.106 g, 4.61 mmol), and EtOH (4.6 mL) afforded 1.493 g (60%) of **4h**. Oil; ^1H NMR (300 MHz, CDCl_3) 7.20-7.39 (m, 5 H), 5.88 (t, $J = 6.4$ Hz, 1 H), 4.79 (d, $J = 6.4$ Hz, 2 H), 3.95 (s, 2 H); ^{13}C NMR (75.4 MHz, CDCl_3) 205.9, 138.3, 128.9, 128.4, 126.9, 77.6, 76.3, 29.9; IR (neat) $\nu(\text{cm}^{-1}) = 1941, 1494$; MS (EI) m/z (%): 210 (M^+ (^{80}Se), 7.03), 208 (M^+ (^{78}Se), 3.93), 91 (100); Elemental Analysis Calcd for $\text{C}_{10}\text{H}_{10}^{80}\text{Se}$: C: 57.44, H: 4.82. Found: C 57.59, H 4.87.

(8) 1,2-Propadienyl Heptyl Selenide (4i). The reaction of **3i** (1.612 g, 12.8 mmol), Na (0.216 g, 13.5 mmol), and EtOH (9.3 mL) afforded 0.709 g (44%) of **4i**. Oil; ^1H NMR (300 MHz, CDCl_3) 5.84 (t, $J = 6.3$ Hz, 1 H), 4.74 (d, $J = 6.3$ Hz, 2 H), 2.67 (t, $J = 7.5$ Hz, 2 H), 1.60-1.76 (m, 2 H), 1.16-1.44 (m, 8 H), 0.86 (t, $J = 6.6$ Hz, 3 H); ^{13}C NMR (75.4 MHz, CDCl_3) 204.9, 77.9, 76.3, 31.6, 30.1, 29.7, 28.8, 26.2, 22.5, 14.0; IR (neat) $\nu(\text{cm}^{-1}) = 1942, 1465$; MS (EI) m/z (%): 218 (M^+ (^{80}Se), 18.77), 216 (M^+ (^{80}Se), 9.91), 57 (100); HRMS Calcd for $\text{C}_{10}\text{H}_{18}^{80}\text{Se}$ (M^+): 218.05737. Found:

218.05537.

(9) 1,2-Propadienyl Dodecanyl Selenide (4j). The reaction of **3j** (1.303 g, 4.5 mmol), Na (0.214 g, 13.5 mmol), and EtOH (9.3 mL) afforded 0.303 g (23%) of **4j**. Oil; ^1H NMR (300 MHz, CDCl_3) 5.85 (t, $J = 7.2$ Hz, 1 H), 4.75 (d, $J = 7.2$ Hz, 2 H), 2.68 (t, $J = 7.5$ Hz, 2 H), 1.60-1.74 (m, 2 H), 1.27-1.44 (m, 18 H), 0.87 (t, $J = 6.8$ Hz, 3 H); ^{13}C NMR (75.4 MHz, CDCl_3) 205.0, 77.9, 76.3, 31.9, 30.1, 29.8, 29.63, 29.61, 29.58, 29.49, 29.3, 29.1, 26.3, 22.7, 14.1; IR (neat) $\nu(\text{cm}^{-1}) = 1943, 1465$; HRMS (EI) m/z (%): 288 (M^+ (^{80}Se), 25.37), 286 (M^+ (^{80}Se), 12.94), 43 (100); HRMS Calcd for $\text{C}_{15}\text{H}_{28}^{80}\text{Se}$ (M^+): 288.13562. Found: 288.13642.

The following compounds were prepared according to Typical Procedure E.

(1) Z-2-iodo-3-(p-tolylseleno)-2-propen-1-ol (Z-11b). The reaction of **4b** (104 mg, 0.5 mmol) and I_2 (506 mg, 2.0 mmol) in MeCN/ H_2O (totally 4:1, 10 mL) afforded 126 mg (72%) of **Z-11b**. Oil, ^1H NMR (300 MHz, CDCl_3) 7.46-7.49 (d, $J = 8.4$ Hz, 2 H), 7.40 (t, $J = 0.9$ Hz, 1 H), 7.13 (d, $J = 8.4$ Hz, 2 H), 4.20 (d, $J = 2.4$ Hz, 2 H), 2.47 (bs, 1 H), 2.34 (s, 3H); ^{13}C NMR (75.4 MHz, CDCl_3) 138.3, 133.8, 133.6, 130.2, 125.4, 104.3, 71.7, 21.1; IR (neat) $\nu(\text{cm}^{-1}) = 3344, 3019, 2919, 1585, 1490, 1446$; MS (EI) m/z (%): 354 (M^+ (^{80}Se), 57.17), 352 (M^+ (^{78}Se), 29.93), 91 (100); HRMS Calcd for $\text{C}_{10}\text{H}_{11}\text{IO}^{80}\text{Se}$ (M^+): 353.90198. Found: 353.90438.

(2) Z-2-iodo-3-(o-tolylseleno)-2-propen-1-ol (Z-11c). The reaction of **4c** (104 mg, 0.5 mmol) and I_2 (507 mg, 2.0 mmol) in MeCN/ H_2O (totally 4:1, 10 mL) afforded 126 mg (72%) of **Z-11c**. Oil; ^1H NMR (300 MHz, CDCl_3) 7.50-7.53 (d, $J = 7.2$ Hz, 1 H), 7.28 (t, $J = 1.2$ Hz, 1 H), 7.17-7.19 (m, 2 H), 7.05-7.09 (m, 1 H), 4.14 (d, $J = 5.4$

Hz, 2 H), 2.67 (bs, 1 H), 2.38 (s, 3H); ^{13}C NMR (75.4 MHz, CDCl_3) 140.2, 134.3, 132.3, 130.3, 130.0, 128.5, 126.8, 105.3, 71.6, 22.8; IR (neat) $\nu(\text{cm}^{-1})$ = 3348, 3057, 2917, 1586, 1467, 1454, 1379; MS (EI) m/z (%): 354 (M^+ (^{80}Se), 437.44), 352 (M^+ (^{79}Se), 19.21), 91 (100); HRMS Calcd for $\text{C}_{10}\text{H}_{11}\text{IO}^{80}\text{Se}$ (M^+): 353.90198 Found: 353.90593

(3) Z-2-iodo-3-(*m*-tolylseleno)-2-propen-1-ol (Z-11d). The reaction of **4d** (105 mg, 0.5 mmol) and I_2 (508 mg, 2.0 mmol) in MeCN/ H_2O (totally 4:1, 10 mL) afforded 131 mg (74%) of **Z-11d**. Oil; ^1H NMR (300 MHz, CDCl_3) 7.30-7.38 (m, 3 H), 7.04-7.19 (m, 2 H), 4.14 (d, J = 3.9 Hz, 2 H), 2.48 (bs, 1 H), 2.27 (s, 3H); ^{13}C NMR (75 MHz, CDCl_3) 139.3, 134.1, 133.2, 130.5, 129.2, 128.9(4), 128.9(0), 104.7, 71.7, 21.2; IR (neat) $\nu(\text{cm}^{-1})$ = 3346, 2917, 2853, 1588, 1570, 1472, 1447, 1071, 997, 774, 688, 579; MS (EI) m/z (%): 354 (M^+ (^{80}Se), 47.48), 352 (M^+ (^{79}Se), 14.82), 91 (100); HRMS Calcd for $\text{C}_{10}\text{H}_{11}\text{IO}^{80}\text{Se}$ (M^+): 353.90198 Found: 353.89803.

(4) Z-3-(4-bromophenylseleno)-2-iodo-2-propen-1-ol (Z-11e). The reaction of **4e** (138 mg, 0.5 mmol) and I_2 (508 mg, 2.0 mmol) in MeCN/ H_2O (totally 4:1, 10 mL) afforded 139 mg (66%) of **Z-11e**. Solid, m.p: 86-88°C ($\text{CH}_2\text{Cl}_2/n$ -Hexane); ^1H NMR (300 MHz, CDCl_3) 7.42-7.45 (m, 4 H), 7.38 (t, J = 1.5 Hz, 1 H), 4.23 (d, J = 5.4 Hz, 2 H), 2.20 (bs, 1 H); ^{13}C NMR (75 MHz, CDCl_3) 134.9, 132.5, 132.0, 128.0, 122.5, 105.4, 71.5; IR (KBr) $\nu(\text{cm}^{-1})$ = 3241, 1607, 1466, 1380, 1361; MS (EI) m/z (%): 420 (M^+ (^{81}Br , ^{80}Se), 48.32), 418 (M^+ ($^{79}\text{Br}/^{80}\text{Se}$ or $^{81}\text{Br}/^{78}\text{Se}$), 63.04), 416 (M^+ (^{79}Br , ^{78}Se), 28.79), 156 (100); HRMS Calcd for $\text{C}_9\text{H}_8\text{BrIO}^{80}\text{Se}$ (M^+): 417.81684 Found: 417.79839.

Fig. 1. ORTEP representation of **Z-11e**

(5) Z-3-(4-chlorophenylseleno)-2-iodo-2-propen-1-ol (Z-11f). The reaction of **4f** (115 mg, 0.5 mmol) and I_2 (508 mg, 2.0 mmol) in MeCN/ H_2O (totally 4:1, 10 mL) afforded 134 mg (72%) of **Z-11f**. Solid, m.p: 82-84°C (CH_2Cl_2/n -Hexane); ^1H NMR (300 MHz, $CDCl_3$) 7.41-7.44 (m, 2 H), 7.31 (t, $J = 1.2$ Hz, 1 H), 7.19-7.22 (m, 2 H), 4.14 (d, $J = 2.1$ Hz, 2 H), 2.57 (bs, 1 H); ^{13}C NMR (75 MHz, $CDCl_3$) 134.9, 134.5, 132.3, 129.6, 127.3, 105.3, 71.7; IR (KBr) $\nu(cm^{-1}) = 3240, 1608, 1472, 1387, 1359$; MS (EI) m/z (%): 376 (M^+ ($^{37}Cl, ^{80}Se$), 20.35), 374 (M^+ ($^{35}Cl/^{80}Se$ or $^{37}Cl/^{78}Se$), 49.16), 372 (M^+ ($^{35}Cl, ^{78}Se$), 22.50), 191 (100); HRMS Calcd for $C_9H_8ClIO^{80}Se$ (M^+): 373.84736 Found: 373.84444.

(6) Z-2-iodo-3-(*p*-methoxy-phenylseleno)-2-propen-1-ol (Z-11g). The reaction of **4g** (115 mg, 0.5 mmol) and I_2 (509 mg, 2.0 mmol) in MeCN/ H_2O (totally 4:1, 10 mL) afforded 140 mg (74%) of **Z-11g**. Oil, ^1H NMR (300 MHz, $CDCl_3$) 7.50-7.52 (m, 2 H), 7.34 (t, $J = 0.9$ Hz, 1 H), 6.83 (d, $J = 8.7$ Hz, 2 H), 4.17 (s, 2 H), 3.79 (s, 3 H), 2.60 (bs, 1 H); ^{13}C NMR (75 MHz, $CDCl_3$) 159.8, 135.9, 134.3, 119.1, 115.0, 103.8, 71.6, 55.3; IR (neat) $\nu(cm^{-1}) = 3352, 2934, 2835, 1589, 1572, 1491, 1460$; MS (EI) m/z (%): 370 (M^+ (^{80}Se), 75.66), 368 (M^+ (^{78}Se), 38.15), 187 (100); HRMS Calcd for $C_{10}H_{11}IO_2Se$ (M^+): 369.89690 Found: 369.89946.

(7) Z-3-(benzylseleno)-2-iodo-2-propen-1-ol (Z-11h). The reaction of **4h** (103 mg, 0.5 mmol) and I₂ (511 mg, 2.0 mmol) in MeCN/H₂O (totally 4:1, 10 mL) afforded 101 mg (58%) of **Z-11h**. Oil, ¹H NMR (300 MHz, CDCl₃) 7.15-7.26 (m, 6 H), 4.05 (s, 2 H), 3.96 (s, 2 H), 2.55 (bs, 1 H); ¹³C NMR (75.4 MHz, CDCl₃) 138.5, 130.9, 129.2, 129.1, 127.6, 105.6, 72.1, 30.2; IR (neat) ν(cm⁻¹) = 3354, 3025, 2923, 2853, 1584, 1493, 1452; MS (EI) m/z (%): 354 (M⁺ (⁸⁰Se), 4.85), 352 (M⁺ (⁷⁸Se), 2.70), 91 (100); HRMS Calcd for C₁₀H₁₁IO⁸⁰Se (M⁺): 353.90463 Found: 353.90463.

(8) Z-3-(heptylseleno)-2-iodo-2-propen-1-ol (Z-11i). The reaction of **4i** (104 mg, 0.5 mmol) and I₂ (507 mg, 2.0 mmol) in MeCN/H₂O (totally 4:1, 10 mL) afforded 110 mg (64%) of **Z-11i**. Oil: ¹H NMR (300 MHz, CDCl₃) 7.27 (d, *J* = 3.9 Hz, 1H), 4.20 (d, *J* = 3.3 Hz, 2 H), 2.80 (t, *J* = 7.5 Hz, 2 H), 2.46 (bs, 1 H), 1.69-1.78 (m, 2 H), 1.21-1.42 (m, 8 H), 0.86 (t, *J* = 6.8 Hz, 3 H); ¹³C NMR (75.4 MHz, CDCl₃) 131.7, 104.9, 71.9, 31.6, 31.0, 29.6, 28.7, 26.9, 22.5, 14.0; IR (neat) ν(cm⁻¹) = 3337, 2955, 1585, 1457, 1377; MS (EI) m/z (%): 362 (M⁺ (⁸⁰Se), 32.54), 360 (M⁺ (⁷⁸Se), 18.35), 57 (100); HRMS Calcd for C₁₀H₁₉IO⁸⁰Se (M⁺): 361.96458. Found: 361.96409.

(9) Z-3-(Dodecylseleno)-2-iodo-2-propen-1-ol (Z-11j). The reaction of **4j** (144 mg, 0.5 mmol) and I₂ (511 mg, 2.0 mmol) in MeCN/H₂O (totally 4:1, 10 mL) afforded 71 mg (33%) of **Z-11j**. Solid, m.p: 26-27°C (CH₂Cl₂/*n*-Hexane); ¹H NMR (300 MHz, CDCl₃) 7.30 (t, *J* = 0.9 Hz, 1H), 4.22 (dd, *J* = 6.5 Hz and 0.9 Hz, 2 H), 2.81 (t, *J* = 7.4 Hz, 2 H), 2.10 (bs, 1 H), 1.69-1.78 (m, 2 H), 1.26-1.30 (m, 18 H), 0.88 (t, *J* = 6.8 Hz, 3 H); ¹³C NMR (75.4 MHz, CDCl₃) 131.6, 104.9, 72.0, 31.9, 31.1, 29.7, 29.6, 29.5(8), 29.5(5), 29.4, 29.3, 29.0, 26.9, 22.7, 14.1; IR (KBr) ν(cm⁻¹) = 3317, 2922,

1468; MS (EI) m/z (%): 432 (M^+ (^{80}Se), 83.86), 362 (M^+ (^{80}Se), 48.56), 57 (100);

HRMS Calcd for $\text{C}_{15}\text{H}_{29}\text{IO}^{80}\text{Se}$ (M^+): 432.04283. Found: 432.03847.

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