Supporting Information

Palladium-Catalyzed Direct Oxidative Alkenylation of Azoles

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Instrumentation and Chemicals

$^1$H and $^{13}$C NMR spectra were recorded at 400 MHz and 100 MHz, respectively, for CDCl$_3$ solutions. MS data were obtained by EI or CI. GC analysis was carried out using a silicon OV-17 column (i. d. 2.6 mm x 1.5 m) or a CBP-1 capillary column (i. d. 0.5 mm x 25 m). Photoluminescence spectra were measured as described previously.$^1$

2-Phenylthiazole (2g)$^2$ and 2-phenyloxazole (4a)$^3$ were prepared by the methods reported previously. Unless otherwise noted, materials obtained from commercial suppliers were used without further purification.

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Experimental Procedure

Synthesis of 5-(thiazol-2-yl)nonan-5-ol (1c). In a 20 mL two-necked flask were added 2-bromothiazole (3 mmol, 492 mg) and Et₂O (2.5 mL) under nitrogen. "BuMgBr (3.6 mmol, 4.2 mL, 0.85 M, Et₂O solution) were added dropwise, and the resulting mixture was then stirred at 40 °C (bath temperature) for 1 h. The reaction mixture was cooled to room temperature, and 5-nonanone (512 mg, 3.6 mmol) was added. The mixture was stirred overnight, poured into saturated aq. NH₄Cl, and extracted with diethyl ether. The organic layer was dried over Na₂SO₄ and concentrated in vacuo. The product 1c (2.24 mmol, 509 mg, 75%) was isolated by chromatography on silica gel using hexane-ethyl acetate (9:1, v/v).

Synthesis of 2-(2,6-dimethylphenyl)thiazole (1h). In a 20 mL two-necked flask were added 2-bromothiazole (2 mmol, 325 mg), 2,6-dimethylphenylboronic acid (2 mmol, 300 mg), Pd(PPh₃)₄ (0.2 mmol, 231 mg), 2 M aq. Na₂CO₃ (2.4 mL), toluene (2.4 mL) and EtOH (1 mL). The resulting mixture was stirred under nitrogen at 90 °C (bath temperature) overnight. After cooling, the reaction mixture was poured into H₂O, extracted with Et₂O, and dried over Na₂SO₄. The product 1h (0.55 mmol, 104 mg, 28%) was isolated by chromatography on silica gel using hexane-ethyl acetate (95:5, v/v).

Aldol-type reaction of 3bb with benzaldehyde. In a 20 mL two-necked flask were added diisopropylamine (1 mmol, 145 μL) and THF (1 mL) under nitrogen. "BuLi (1 mmol, 625 μL, 1.6 M hexane solution) was added dropwise at 0 °C, and the solution was stirred for 1 h at the same temperature. The reaction mixture was cooled to -78 °C and 3bb (225 mg, 1 mmol) in THF (1 mL) was added. After 1 h, benzaldehyde (127 mg, 1.2 mmol) was added dropwise. The mixture was allowed to warm to room temperature and stirred overnight. The resulting mixture was poured into saturated aq. NH₄Cl and extracted with diethyl ether. The organic layer was dried over Na₂SO₄ and concentrated in vacuo. The product 9a (146 mg, 0.45 mmol, 45%) was isolated by chromatography on silica gel using hexane-ethyl acetate (8:2, v/v).

Dehydration of 9a. In a 20 mL two-necked flask were added 9a (0.4 mmol, 132 mg), MsCl (0.48 mmol, 37 μL) and CH₂Cl₂ (2 mL). Triethylamine (0.96 mmol, 136 μL) was added at 0 °C. The mixture was stirred at room temperature under air overnight and then poured into H₂O. The organic layer was extracted with CH₂Cl₂, dried over Na₂SO₄, and concentrated in vacuo. The product 10a (0.34 mmol, 106 mg, 85%) was isolated by chromatography on silica gel using hexane-ethyl acetate (9:1, v/v).
Characterization Data of Products

5-(Thiazol-2-yl)nonan-5-ol (1c) m.p. 36-38 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 0.85 (t, \(J = 7.3\) Hz), 1.01-1.41 (m, 8H), 1.86-1.95 (m, 4H), 3.06 (s, 1H), 7.27 (d, \(J = 3.2\) Hz, 1H), 7.71 (d, \(J = 3.2\) Hz, 1H); \(^13\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 14.0, 22.9, 25.4, 42.5, 78.0, 118.9, 141.8, 178.0; HRMS m/z (M\(^+\)) calcd for C\(_{12}\)H\(_{21}\)NOS: 227.1344, found: 227.1345.

2-(2,6-Dimethylphenyl)thiazole (1h) oil; \(^1\)H NMR (400 MHz, CDCl\(_3\)) 2.14 (s, 6H), 7.11 (d, \(J = 7.6\) Hz, 2H), 7.23 (t, \(J = 7.6\) Hz, 1H), 7.46-7.47 (m, 1H), 7.50-7.51 (m, 1H), 7.92-7.94 (m, 1H); \(^13\)C NMR (100 MHz, CDCl\(_3\)) 20.1, 120.2, 127.5, 129.7, 133.5, 137.8, 142.9, 166.6; HRMS m/z (M\(^+\)) calcd for C\(_{11}\)H\(_{11}\)NS: 189.0612, found: 189.0602.

tert-Butyl (E)-3-(2-isobutylthiazol-5-yl)acrylate (3ab) oil; \(^1\)H NMR (400 MHz, CDCl\(_3\)) 1.00 (d, \(J = 6.6\) Hz, 6H), 1.52 (s, 9H), 2.08-2.15 (m, 1H), 2.86 (d, \(J = 7.3\) Hz, 2H), 6.06 (d, \(J = 15.7\) Hz, 1H), 7.63 (d, \(J = 15.7\) Hz, 1H), 7.73 (s, 1H); \(^13\)C NMR (100 MHz, CDCl\(_3\)) 22.2, 28.1, 29.7, 42.7, 80.8, 121.7, 133.1, 134.5, 145.1, 165.5, 172.5; HRMS m/z (M\(^+\)) calcd for C\(_{14}\)H\(_{21}\)NO\(_2\)S: 267.1293, found: 267.1291.

Phenyl (E)-3-(2-isobutylthiazol-5-yl)acrylate (3ac) m.p. 81-82 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) 1.02 (d, \(J = 6.6\) Hz, 6H), 2.11-2.18 (m, 1H), 2.90 (d, \(J = 7.3\) Hz, 2H), 6.32 (d, \(J = 15.8\) Hz, 1H), 7.15 (d, \(J = 7.7\) Hz, 2H), 7.22-7.26 (m, 1H), 7.40 (t, \(J = 7.7\) Hz, 2H), 7.83 (s, 1H); \(^13\)C NMR (100 MHz, CDCl\(_3\)) 22.2, 29.8, 42.8, 118.6, 121.5, 125.8, 129.4, 134.1, 136.0, 146.2, 150.7, 164.7, 173.6; HRMS m/z (M\(^+\)) calcd for C\(_{16}\)H\(_{17}\)NO\(_2\)S: 287.0980, found: 287.0983.

(E)-3-(2-isobutylthiazol-5-yl)-N,N-dimethylacrylamide (3ad) m.p. 73-75 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) 1.01 (d, \(J = 6.6\) Hz, 6H), 2.09-2.16 (m, 1H), 2.86 (d, \(J = 7.3\) Hz, 2H), 3.06 (s, 3H), 3.14 (s, 3H), 6.18 (d, \(J = 15.0\) Hz, 1H), 7.72 (s, 1H) 7.74 (d, \(J = 15.0\) Hz, 1H); \(^13\)C NMR (100 MHz, CDCl\(_3\)) 22.2, 29.7, 35.9, 37.2, 42.7, 118.8, 132.1, 135.1, 144.6, 165.8, 171.6; HRMS m/z (M\(^+\)) calcd for C\(_{12}\)H\(_{18}\)N\(_2\)OS: 238.1140, found: 238.1141.

2-Isobutyl-5-[((E))-styryl]thiazole (3ae) m.p. 64-66 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 1.01 (d, \(J = 6.6\) Hz, 6H), 2.08-2.15 (m, 1H), 2.86 (d, \(J = 7.3\) Hz, 2H), 6.32 (d, \(J = 15.0\) Hz, 1H), 7.15 (d, \(J = 7.7\) Hz, 2H), 7.22-7.26 (m, 1H), 7.40 (t, \(J = 7.7\) Hz, 2H), 7.83 (s, 1H), 7.93 (d, \(J = 15.6\) Hz, 1H); \(^13\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 22.2, 28.1, 29.7, 42.7, 60.8, 121.7, 133.1, 134.5, 145.1, 165.5, 172.5; HRMS m/z (M\(^+\)) calcd for C\(_{15}\)H\(_{17}\)NS: 243.1082, found: 243.1078.

2-Isobutyl-5-[((E))-4-methoxystyryl]thiazole (3af) m.p. 59-61 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 1.01 (d, \(J = 6.6\) Hz, 6H), 2.08-2.15 (m, 1H), 2.86 (d, \(J = 7.3\) Hz, 2H), 6.32 (d, \(J = 15.0\) Hz, 1H); \(^13\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 22.2, 28.1, 29.7, 42.7, 60.8, 121.7, 133.1, 134.5, 145.1, 165.5, 172.5; HRMS m/z (M\(^+\)) calcd for C\(_{16}\)H\(_{19}\)NOS: 273.1187, found: 273.1182.

5-[((E))-4-Fluorostyryl]-2-isobutylthiazole (3ag) m.p. 51-52 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 1.01 (d, \(J = 6.6\) Hz, 6H), 2.08-2.15 (m, 1H), 2.85 (d, \(J = 7.3\) Hz, 2H), 3.83 (s, 3H), 6.76 (d, \(J = 16.1\) Hz, 1H), 6.87-6.90 (m, 2H), 7.03 (d, \(J = 16.1\) Hz, 1H), 7.37-7.40 (m, 2H), 7.55 (s, 1H); \(^13\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 22.3, 29.7, 42.7, 55.3, 114.2, 116.6, 127.6, 129.4, 130.5, 137.7, 140.1, 159.5, 168.6; HRMS m/z (M\(^+\)) calcd for C\(_{16}\)H\(_{19}\)NOS: 273.1187, found: 273.1182.

Butyl 2-[(2-isobutylthiazol-5-yl)methyl]acrylate (3ah) oil; \(^1\)H NMR (400 MHz,
CDCl$_3$) $\delta$ 0.93 (t, $J = 7.3$ Hz, 3H), 0.97 (d, $J = 6.5$ Hz, 6H), 1.33-1.43 (m, 2H), 1.61-1.68 (m, 2H), 2.04-2.11 (m, 1H), 2.80 (d, $J = 7.3$ Hz, 2H), 3.78 (s, 2H), 4.17 (t, $J = 7.0$ Hz, 2H), 5.59-5.60 (m, 1H), 6.22 (s, 1H), 7.37 (s, 1H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 13.7, 19.1, 22.3, 29.4, 29.7, 30.6, 42.5, 64.9, 126.4, 134.7, 139.1, 140.3, 166.4, 169.8; HRMS m/z (M$^+$) calcd for C$_{13}$H$_{22}$NO$_2$: 281.1449, found: 281.1443.

Butyl (E)-3-(2-isobutylthiazol-5-yl)-2-methylacrylate (3ah$^+$) oil; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 0.97 (t, $J = 7.3$ Hz, 3H), 1.00 (d, $J = 6.6$ Hz, 6H), 1.39-1.47 (m, 2H), 1.66-1.73 (m, 2H), 2.11-2.18 (m, 4H), 2.90 (d, $J = 166.3$, 168.7; HRMS m/z (M$^+$) calcd for C$_{14}$H$_{24}$NO: 225.1449, found: 225.1444.

Butyl (E)-3-(2-methylthiazol-5-yl)acrylate (3ba) oil; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 0.96 (t, $J = 7.7$ Hz, 3H), 1.38-1.47 (m, 2H), 1.64-1.69 (m, 2H), 2.73 (s, 3H), 4.20 (t, $J = 7.0$ Hz, 2H), 6.12 (d, $J = 15.7$ Hz, 1H), 7.72, (d, $J = 15.7$ Hz, 1H), 7.73 (s, 1H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 13.7, 19.2, 19.7, 30.7, 64.6, 119.8, 134.0, 134.8, 145.5, 166.3, 168.4; HRMS m/z (M$^+$) calcd for C$_{15}$H$_{23}$NO$_2$: 225.0823, found: 225.0822.

Butyl (E)-3-(2-(5-hydroxynan-5-yl)thiazol-5-yl)acrylate (3ca) oil; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 0.86 (t, $J = 7.4$ Hz, 6H), 0.96 (t, $J = 7.3$ Hz, 3H), 1.03-1.10 (m, 2H), 1.23-1.47 (m, 8H), 1.64-1.71 (m, 2H), 1.82-1.97 (m, 4H), 2.85 (s, 1H), 4.20 (t, $J = 6.9$ Hz, 2H), 6.18 (d, $J = 15.6$ Hz, 1H), 7.74 (d, $J = 15.6$ Hz, 1H), 7.79 (s, 1H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 13.7, 13.9, 19.1, 22.8, 25.4, 30.7, 42.2, 64.6, 78.3, 120.0, 134.1, 134.9, 145.2, 166.4, 180.5; HRMS m/z (M$^+$) calcd for C$_{19}$H$_{29}$NO$_2$: 353.2025, found: 353.2019.

Butyl (E)-3-(2-methoxythiazol-5-yl)acrylate (3da) oil; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 0.96 (t, $J = 7.3$ Hz, 3H), 1.42 (sex, $J = 7.3$ Hz, 2H), 1.67 (quin, $J = 7.3$ Hz, 2H), 4.11 (s, 3H), 4.18 (t, $J = 7.3$ Hz, 2H), 5.94 (d, $J = 15.6$ Hz, 1H), 7.29 (s, 1H), 7.64 (d, $J = 15.6$ Hz, 1H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 13.7, 19.1, 30.7, 58.6, 64.4, 117.3, 127.9, 134.9, 141.5, 166.6, 176.0; HRMS m/z (M$^+$) calcd for C$_{17}$H$_{25}$NO$_2$: 241.0773, found: 241.0777.

Butyl (E)-3-[2-(methylthio)thiazol-5-yl]acrylate (3ea) oil; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 0.96 (t, $J = 7.7$ Hz, 3H), 1.42 (sex, $J = 7.7$ Hz, 2H), 1.63-1.70 (m, 2H), 2.72 (s, 3H), 4.19 (t, $J = 6.9$ Hz, 2H), 6.03 (d, $J = 15.8$ Hz, 1H), 7.70, (d, $J = 15.8$ Hz, 1H) 7.71 (s, 1H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 13.7, 16.3, 19.1, 30.7, 64.5, 119.2, 133.4, 134.1, 145.7, 166.3, 169.7; HRMS m/z (M$^+$) calcd for C$_{17}$H$_{23}$NO$_2$: 257.0544, found: 257.0543.

Butyl (E)-3-[2-(N-butyacetamido)thiazol-5-yl]acrylate (3fa) oil; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 0.94-1.01 (m, 6H), 1.40-1.46 (m, 4H), 1.64-1.78 (m, 4H), 2.43 (s, 3H), 4.12-4.20 (m, 4H), 6.15 (d, $J = 15.8$ Hz, 1H), 7.60 (s, 1H), 7.74 (d, $J = 15.8$ Hz, 1H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 13.71, 13.73, 19.2, 20.0, 22.6, 30.6, 30.7, 48.3, 64.4, 117.5, 129.7, 135.0, 141.3, 160.2, 166.8, 169.9; HRMS m/z (M$^+$) calcd for C$_{18}$H$_{24}$N$_2$O$_3$: 324.1508, found: 324.1511.

Butyl (E)-3-[2-(2,6-dimethylphenyl)thiazol-5-yl]acrylate (3ha) oil; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 0.97 (t, $J = 7.3$ Hz, 3H), 1.44 (sex, $J = 7.3$ Hz, 2H), 1.69 (quin, $J = 7.3$ Hz, 2H), 2.18 (s, 6H), 4.21 (t, $J = 7.3$ Hz, 2H), 6.23 (d, $J = 15.1$ Hz, 1H), 7.12 (d, $J = 7.8$ Hz, 2H), 7.26 (t, $J = 7.8$ Hz, 1H), 7.85 (d, $J = 15.1$ Hz, 1H), 8.01 (s, 1H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 13.7, 19.2, 20.2, 30.8, 64.6, 120.5, 127.7, 129.7, 132.9, 133.8, 136.3, 137.6, 145.9, 166.3, 168.7; HRMS m/z (M$^+$) calcd for C$_{18}$H$_{24}$N$_2$O$_3$: 315.1293, found: 315.1298.
Butyl (E)-3-(4-methyl-2-phenylthiazol-5-yl)acrylate (3ia) m.p. 53-55 °C; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 0.96 (t, $J = 7.7$ Hz, 3H), 1.44 (sex, $J = 7.7$ Hz, 2H), 1.66-1.72 (m, 2H), 2.57 (s, 3H), 4.21 (t, $J = 7.0$ Hz, 2H), 6.13 (d, $J = 15.4$ Hz, 1H), 7.42-7.46 (m, 3H), 7.80 (d, $J = 15.4$ Hz, 1H), 7.92-7.95 (m, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 13.7, 15.7, 19.2, 30.7, 64.5, 118.9, 126.7, 128.5, 129.0, 130.7, 133.1, 133.8, 156.6, 166.6, 167.5; HRMS m/z ($M^+$) calcd for C$_{17}$H$_{19}$NO$_2$: 281.1316, found: 281.1318.

Butyl (E)-3-(2,4-dimethylthiazol-5-yl)acrylate (3ja) oil; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 0.97 (t, $J = 7.3$ Hz, 3H), 1.43 (sex, $J = 7.3$ Hz, 2H), 1.64-1.71 (m, 2H), 2.48 (s, 3H), 2.67 (s, 3H), 4.19 (t, $J = 6.6$ Hz, 2H), 6.01 (d, $J = 15.4$ Hz, 1H), 7.74 (d, $J = 15.4$ Hz, 1H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 13.7, 15.5, 19.1, 19.6, 30.7, 64.4, 118.5, 128.2, 133.9, 155.1, 166.4, 166.7; HRMS m/z ($M^+$) calcd for C$_{17}$H$_{19}$NO$_2$: 239.0980, found: 239.0977.

Butyl (E)-3-(2-phenyloxazol-5-yl)acrylate (5aa) m.p. 83-85 °C; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 0.97 (t, $J = 7.3$ Hz, 3H), 1.44 (sex, $J = 7.3$ Hz, 2H), 1.66-1.73 (m, 2H), 4.20 (t, $J = 6.6$ Hz, 2H), 6.47 (d, $J = 15.7$ Hz, 1H), 7.37 (s, 1H), 7.47-7.51 (m, 4H), 8.07-8.10 (m, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 13.7, 19.1, 30.7, 64.4, 118.2, 126.7, 126.8, 128.9, 131.2, 131.9, 148.0, 163.0, 166.5; HRMS m/z ($M^+$) calcd for C$_{16}$H$_{17}$NO$_3$: 271.1208, found: 271.1199.

Butyl (E)-3-(2,4-dimethylthiazol-5-yl)acrylate (5ba) oil; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 0.96 (t, $J = 7.4$ Hz, 3H), 1.43 (sex, $J = 7.4$ Hz, 2H), 1.64-1.71 (m, 2H), 2.24 (s, 3H), 2.46 (s, 3H), 4.20 (t, $J = 6.9$ Hz, 2H), 6.20 (d, $J = 15.6$ Hz, 1H), 7.42 (d, $J = 15.6$ Hz, 1H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 11.8, 13.7, 14.1, 19.2, 30.7, 64.4, 115.4, 127.1, 140.6, 143.4, 162.4, 167.0; HRMS m/z ($M^+$) calcd for C$_{12}$H$_{17}$NO$_3$: 233.1206, found: 233.1206.

2,4-Dimethyl-5-[(E)-3-styryl]oxazole (5be) m.p. 65-67 ºC; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 2.21 (s, 3H), 2.45 (s, 3H), 6.81 (d, $J = 16.5$ Hz, 1H), 6.90 (d, $J = 16.5$ Hz, 1H), 7.22-7.26 (m, 1H), 7.32-7.36 (m, 2H), 7.45 (d, $J = 7.3$ Hz, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 11.5, 13.9, 112.6, 126.2, 126.7, 127.6, 128.7, 133.8, 136.8, 145.0, 159.3; HRMS m/z ($M^+$) calcd for C$_{13}$H$_{13}$N$_2$O: 210.0827, found: 210.0828.

(E)-3-(4,5-Dimethylthiazol-2-yl)-N,N-dimethylacrylamide (7) m.p. 93-94 ºC; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 2.36 (s, 3H), 2.38 (s, 3H), 3.06 (s, 3H), 3.17 (s, 3H), 7.14 (d, $J = 15.0$ Hz, 1H), 7.60 (d, $J = 15.0$ Hz, 1H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 11.6, 14.8, 35.9, 37.4, 119.9, 129.2, 133.6, 150.5, 159.2, 165.8; HRMS m/z ($M^+$) calcd for C$_{10}$H$_{14}$N$_2$OS: 210.0827, found: 210.0828.

$^{4,5,5’,5’}$-Tetramethyl-2,2’-bithiazole (8)$^4$ $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 2.36 (s, 6H), 2.38 (s, 6H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 11.5, 14.7, 128.1, 149.5, 157.0.

tert-Butyl (E)-3-(2-methylthiazol-5-yl)acrylate (3bb) m.p. 58-59 ºC; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 1.58 (s, 9H), 2.72 (s, 3H), 6.04 (d, $J = 15.7$ Hz, 1H), 7.63 (d, $J = 15.7$ Hz, 1H), 7.70 (s, 1H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 19.7, 28.1, 80.8, 121.7, 133.0, 134.9, 145.1, 165.5, 168.1; HRMS m/z ($M^+$) calcd for C$_{11}$H$_{16}$NO$_2$: 225.0821, found: 225.0821.

tert-Butyl (E)-3-[2-(2-hydroxy-2-phenylethyl)thiazol-5-yl]acrylate (9a) oil; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 1.58 (s, 9H), 3.33-3.35 (m, 2H), 3.92 (bs, 1H), 5.16-5.19 (m, 1H), 6.08 (d, $J = 15.6$ Hz, 1H), 7.28-7.42 (m, 5H), 7.63 (d, $J = 15.6$ Hz, 1H), 7.77 (s, 1H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 28.1, 42.5, 72.8, 81.0, 122.3, 125.7, 127.9, 128.6, 132.6.

tert-Butyl $(E)$-3-[2-(2-hydroxy-2-(4-methoxyphenyl)ethyl)thiazol-5-yl]acrylate (9b) m.p. 125-126 °C; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 1.51 (s, 9H), 3.31-3.33 (m, 2H), 3.81 (s, 3H), 3.81-3.83 (m, 1H), 6.07 (d, $J = 15.8$ Hz, 1H), 6.89 (d, $J = 8.8$ Hz, 2H), 7.32 (d, $J = 8.8$ Hz, 2H), 7.63 (d, $J = 15.8$ Hz, 1H), 7.75 (s, 1H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 28.1, 42.6, 55.3, 72.4, 80.9, 113.9, 122.2, 127.0, 132.7, 134.7, 144.7, 159.2, 165.4, 169.6; HRMS m/z (M$^+$) calcd for C$_{18}$H$_{22}$NO$_3$S: 332.1320, found: 332.1317.  

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 1.51 (s, 9H), 3.31-3.33 (m, 2H), 3.81 (s, 3H), 3.81-3.83 (m, 1H), 6.07 (d, $J = 15.8$ Hz, 1H), 6.89 (d, $J = 8.8$ Hz, 2H), 7.32 (d, $J = 8.8$ Hz, 2H), 7.63 (d, $J = 15.8$ Hz, 1H), 7.75 (s, 1H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 28.1, 42.6, 55.3, 72.4, 80.9, 113.9, 122.2, 127.0, 132.7, 134.7, 144.7, 159.2, 165.4, 169.6; HRMS m/z (M$^+$) calcd for C$_{18}$H$_{22}$NO$_3$S: 332.1320, found: 332.1317.

tert-Butyl $(E)$-3-[2-(4-dimethylaminophenyl)-2-hydroxyethyl]thiazol-5-yl]acrylate (9c) m.p. 99-100 °C; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 1.52 (s, 9H), 2.95 (s, 6H), 3.28-3.40 (m, 2H), 3.49 (bs, 1H), 5.06-5.07 (m, 1H), 6.07 (d, $J = 15.8$ Hz, 1H), 6.71 (d, $J = 8.8$ Hz, 2H), 7.26 (d, $J = 8.8$ Hz, 2H), 7.63 (d, $J = 15.8$ Hz, 1H), 7.75 (s, 1H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 28.1, 40.6, 42.6, 72.7, 80.9, 112.5, 122.2, 126.7, 130.4, 132.8, 134.8, 150.4, 165.5, 170.0; HRMS m/z (M$^+$) calcd for C$_{20}$H$_{26}$N$_2$O$_3$S: 374.1664, found: 374.1668.

tert-Butyl $(E)$-3-[2-(4-trifluoromethylphenyl)-2-hydroxyethyl]thiazol-5-yl]acrylate (9d) m.p. 164-165 °C; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 1.52 (s, 9H), 3.29-3.34 (m, 2H), 4.42 (bs, 1H), 5.24-5.26 (m, 1H), 6.08 (d, $J = 15.6$ Hz, 1H), 7.53 (d, $J = 7.8$ Hz, 2H), 7.62 (d, $J = 15.6$ Hz, 1H), 7.63 (d, $J = 15.6$ Hz, 1H), 7.77 (s, 1H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 28.1, 42.1, 72.0, 81.0, 122.6, 124.1 (q, $J = 270.3$ Hz), 125.5 (q, $J = 3.8$ Hz), 126.0, 130.0 (q, $J = 32.4$ Hz), 132.4, 134.9, 144.6, 146.5, 165.3, 168.9; HRMS m/z (M$^+$) calcd for C$_{19}$H$_{20}$F$_3$NO$_3$S: 399.1114, found: 399.1111.

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 1.53 (s, 9H), 6.13 (d, $J = 15.8$ Hz, 1H), 7.25 (d, $J = 16.5$ Hz, 1H), 7.34-7.40 (m, 3H), 7.42 (d, $J = 16.5$ Hz, 1H), 7.54-7.56 (m, 2H), 7.67 (d, $J = 15.8$ Hz, 1H), 7.85 (s, 1H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 28.1, 80.9, 121.2, 122.1, 127.3, 129.0, 129.4, 132.8, 134.3, 135.4, 136.1, 146.5, 165.3, 168.9; HRMS m/z (M$^+$) calcd for C$_{18}$H$_{19}$NO$_2$S: 313.1136, found: 313.1122.

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 1.53 (s, 9H), 3.85 (s, 3H), 6.10 (d, $J = 15.7$ Hz, 1H), 6.92 (d, $J = 8.8$ Hz, 2H), 7.12 (d, $J = 16.5$ Hz, 1H), 7.41 (d, $J = 16.5$ Hz, 1H), 7.49 (d, $J = 8.8$ Hz, 2H), 7.66 (d, $J = 15.7$ Hz, 1H), 7.81 (s, 1H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 28.1, 80.8, 114.4, 119.1, 121.7, 128.1, 128.8, 132.9, 133.8, 135.9, 146.5, 160.7, 165.5, 168.9; HRMS m/z (M$^+$) calcd for C$_{19}$H$_{20}$F$_3$NO$_3$S: 399.1114, found: 399.1111.

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 1.53 (s, 9H), 6.10 (d, $J = 15.7$ Hz, 1H), 6.92 (d, $J = 8.8$ Hz, 2H), 7.12 (d, $J = 16.5$ Hz, 1H), 7.41 (d, $J = 16.5$ Hz, 1H), 7.49 (d, $J = 8.8$ Hz, 2H), 7.66 (d, $J = 15.7$ Hz, 1H), 7.81 (s, 1H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 28.1, 80.8, 114.4, 119.1, 121.7, 128.1, 128.8, 132.9, 133.8, 135.9, 146.5, 160.7, 165.5, 168.9; HRMS m/z (M$^+$) calcd for C$_{19}$H$_{20}$F$_3$NO$_3$S: 399.1114, found: 399.1111.
$^1$H, $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 28.1, 81.0, 122.6, 123.4, 124.0 (q, $J = 273.3$ Hz), 125.9 (q, $J = 3.2$ Hz), 127.4, 130.8 (q, $J = 32.8$ Hz), 132.5, 134.0, 135.0, 138.7, 146.6, 165.3, 167.2; HRMS $m/z$ (M$^+$) calcd for C$_{19}$H$_{18}$F$_3$NO$_2$S: 381.1010, found: 381.1009.
[1H and 13C NMR Spectra of 1c]
[ $^1\text{H}$ and $^{13}\text{C}$ NMR Spectra of $\text{1h}$ ]

$$
\text{No.} & \delta / \text{ppm} & \text{Hz} & \text{Height} \\
1 & 166.54 & 167.49 & 7 \text{ppm} \\
2 & 142.90 & 143.09 & 33 \text{ppm} \\
3 & 137.77 & 138.48 & 40 \text{ppm} \\
4 & 133.46 & 134.15 & 12 \text{ppm} \\
5 & 129.22 & 129.88 & 38 \text{ppm} \\
6 & 127.52 & 128.18 & 100 \text{ppm} \\
7 & 120.16 & 120.80 & 33 \text{ppm} \\
8 & 20.21 & 203.15 & 84 \text{ppm}
$$
[ $^{1}$H and $^{13}$C NMR Spectra of 3aa ]

No. | ppm  | Hz  | Height |
---|------|-----|--------|
1  | 172.78 | 17198.4 | 8.33  |
2  | 166.33 | 16655.4 | 12.68 |
3  | 145.43 | 14475.5 | 24.10 |
4  | 134.28 | 13395.9 | 13.79 |
5  | 134.01 | 13338.5 | 26.70 |
6  | 119.85 | 11908.2 | 27.79 |
7  | 64.49  | 6419.1  | 15.40 |
8  | 42.72  | 4251.7  | 30.99 |
9  | 30.67  | 3052.3  | 36.46 |
10 | 29.89  | 2955.6  | 33.24 |
11 | 22.18  | 2207.5  | 74.81 |
12 | 19.10  | 1900.8  | 40.49 |
13 | 13.65  | 1356.2  | 33.36 |
[ $^{1}\text{H}$ and $^{13}\text{C}$ NMR Spectra of 3ab ]

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[ $^1$H and $^{13}$C NMR Spectra of 3ac ]

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2   | 164.70 | 16393.3 | 8.84
3   | 150.96 | 14995.4 | 9.49
4   | 146.23 | 14500.4 | 20.43
5   | 135.98 | 13534.4 | 22.03
6   | 134.12 | 13349.5 | 12.63
7   | 129.44 | 12884.1 | 47.36
8   | 125.85 | 12526.4 | 20.29
9   | 121.53 | 12096.1 | 46.37
10  | 118.58 | 11832.7 | 24.18
11  | 42.83  | 4262.7  | 22.65
12  | 29.77  | 2963.6  | 22.37
13  | 22.24  | 2214.0  | 46.62
[\(^1\text{H} \text{ and } ^{13}\text{C}\) NMR Spectra of 3ad]
[\textsuperscript{1}H and \textsuperscript{13}C NMR Spectra of 3ae]
[ $^1$H and $^{13}$C NMR Spectra of 3af]

![NMR Spectra Image]

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[ $^1$H and $^{13}$C NMR Spectra of 3ah ]

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2   | 166.36 | 16658.8 | 7.60 | 14 | 13.66 | 1360.1 | 21.50
3   | 140.30 | 13864.8 | 20.98 | 5  | 135.66 | 13403.5 | 9.90
4   | 139.14 | 13849.1 | 11.10 | 6  | 128.45 | 12586.6 | 19.33
5   | 134.66 | 13403.5 | 9.90 | 7  | 64.88  | 6457.6  | 22.40
6   | 126.45 | 12586.6 | 19.33 | 8  | 42.47  | 4227.0  | 23.94
7   | 30.59  | 3044.3  | 23.48 | 9  | 29.70  | 2956.0  | 22.02
8   | 29.39  | 2925.2  | 21.89 | 10 | 22.26  | 2215.5  | 47.56

S-17
[\textsuperscript{1}H and \textsuperscript{13}C NMR Spectra of \textbf{3ah}']

\begin{figure}
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9 & 30.74 & 3059.5 & 19.16 & & & & & \\
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[ $^1$H and $^{13}$C NMR Spectra of 3ba ]

![NMR Spectra of 3ba](image)

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[ $^1$H and $^{13}$C NMR Spectra of 3ca ]
[ $^1$H and $^{13}$C NMR Spectra of 3da ]

![NMR Spectra of 3da](image)

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[ $^1$H and $^{13}$C NMR Spectra of 3fa ]

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S-24
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[\textsuperscript{1}H and \textsuperscript{13}C NMR Spectra of 5aa]

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[\textsuperscript{1}H and \textsuperscript{13}C NMR Spectra of 5ba]
[ $^{1}$H and $^{13}$C NMR Spectra of 7 ]

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6  | 119.92 | 11935.9 | 52.91  |
7  | 37.41  | 3723.5  | 15.96  |
8  | 35.90  | 3573.6  | 18.48  |
9  | 14.72  | 1464.8  | 53.56  |
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[ $^1$H and $^{13}$C NMR Spectra of 8 ]

![NMR Spectra Diagram]

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[ $^1$H and $^{13}$C NMR Spectra of 3bb ]

![NMR Spectra Image]

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S-32
[\textsuperscript{1}H and \textsuperscript{13}C NMR Spectra of 9a]
$^{1}H$ and $^{13}C$ NMR Spectra of 9b

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[ $^1$H and $^{13}$C NMR Spectra of 9d ]

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$[{^1}H\ and\ {^{13}}C\ NMR\ Spectra\ of\ 10b\ ]$

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