

## **Supporting Information**

### **Triazole-Directed Pd-Catalyzed C(sp<sup>2</sup>)–H Oxygenation of Arenes and Alkenes**

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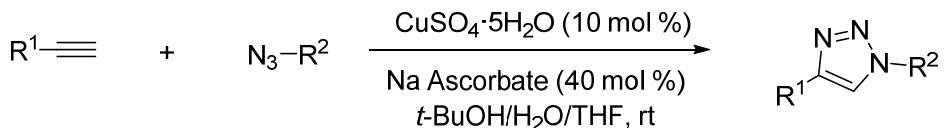
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## 1.-General Considerations

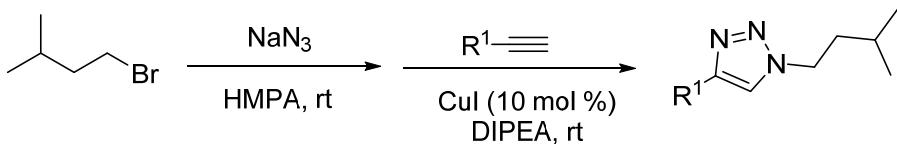
**Reagents.** Commercially available materials were used without further purification. Palladium(II) acetate recrystallized (97% purity), (diacetoxy)iodobenzene (98% purity), bis(*tert*-butylcarbonyloxy)iodobenzene (97% purity), and 1,2-dichloroethane (spectrophotometric grade,  $\geq 99\%$ ) were purchased from Sigma-Aldrich. AcOH (acetic acid glacial, extra pure) was purchased from Scharlau and Ac<sub>2</sub>O (acetic anhydride) was purchased from Panreac.

**Analytical Methods.** <sup>1</sup>H NMR and <sup>13</sup>C NMR spectra as well as IR, HRMS and melting points (where applicable) are included for all new compounds. <sup>1</sup>H and <sup>13</sup>C NMR spectra were recorded on a Bruker 400 MHz and 500 MHz at 20 °C. All <sup>1</sup>H NMR spectra are reported in parts per million (ppm) downfield of TMS and were measured relative to the signals for CHCl<sub>3</sub> (7.26 ppm), unless otherwise indicated. All <sup>13</sup>C NMR spectra were reported in ppm relative to residual CHCl<sub>3</sub> (77 ppm), unless otherwise indicated, and were obtained with <sup>1</sup>H decoupling. Coupling constants, *J*, are reported in hertz. Melting points were measured using open glass capillaries in a Büchi SMP-20 apparatus. Mass spectra were performed by SGiker and were acquired on a time of flight (TOF) mass spectrometer (SYNAPT G2 HDMS from Waters, Milford, MA, USA) equipped with an electrospray source in positive mode (ESI<sup>+</sup>). The chromatographic separation was performed using an ACQUITY UPLC system from Waters (Milford, MA, USA) equipped with an Acquity UPLC BEH C18 1.7 µm, 50 x 2.1 mm column at 30 °C. Mobile phases consisted of 0.1% formic acid in water (A) and 0.1% formic acid in methanol (B). Separation was carried out in 5 min: initial conditions were 5 % B, raised to 100 % B over 2.5 min, held at 100 % B until 4 min, decreased to 5 % B over 0.1 min and held at 5 % B until 5 min for re-equilibration of the system. Flow rate was 0.25 mL/min and injection volume was 5 µL. Infrared spectra were recorded on a Bruker Alpha P. Flash chromatography was performed with EM Science silica gel 60 (230-400 mesh). The yields reported in tables 2-4 correspond to isolated yields and represent an average of at least two independent runs. The procedures described in this section are representative; thus the yields may differ slightly from those given in the tables of the manuscript.

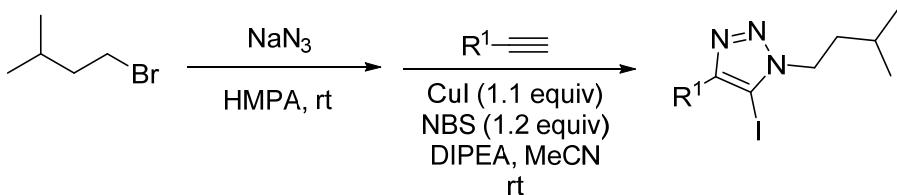
## 2.-Synthesis of Triazoles



**General Procedure A:** Alkyne (1.0 equiv) and the corresponding azide (1.1 equiv) were dissolved in a mixture of deoxygenated *t*-BuOH/H<sub>2</sub>O (4:1, 1 mL/mmol). Then a deoxygenated aq. solution of CuSO<sub>4</sub>·5H<sub>2</sub>O (10 mol %, 1mL/mmol) followed by a deoxygenated aq. solution of sodium ascorbate (40 mol %, 1mL/mmol) was added. The resulting mixture was stirred under inert atmosphere at room temperature overnight. The solvent was partially evaporated under reduced pressure and the resulting solution was washed with aq. NH<sub>4</sub>OH, extracted with AcOEt and washed with brine. The combined organic layers were dried over MgSO<sub>4</sub>, and concentrated under reduced pressure. The crude residue was purified by flash chromatography (hexanes/AcOEt 7/3) unless otherwise noted.

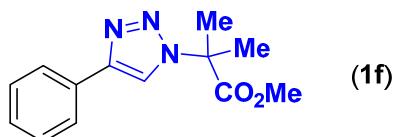


**General Procedure B:** 1-Bromo-3-methylbutane (1.0 equiv) was dropwise added over a solution of NaN<sub>3</sub> (1.1 equiv) in HMPA (0.6 mL/mmol). The resulting solution was stirred under argon at room temperature for 4 hours. Then, CuI (10 mol %), alkyne (1.0 equiv) and DIPEA (5.0 mL/mmol) were subsequently added and the resulting solution was stirred at room temperature overnight. Next, it was washed with HCl 10%, aq. NH<sub>4</sub>OH, extracted with AcOEt and washed with brine. The combined organic layers were dried over MgSO<sub>4</sub>, and concentrated under reduced pressure. The crude residue was purified by flash chromatography (hexanes/AcOEt 7/3) unless otherwise noted.

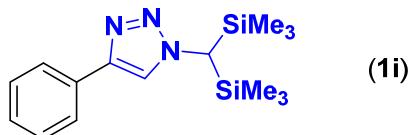


**General Procedure C:** 1-Bromo-3-methylbutane (1.0 equiv) was dropwise added over a solution of NaN<sub>3</sub> (1.1 equiv) in HMPA (0.6 mL/mmol). The resulting solution was stirred under argon at room temperature for 4 hours. In a different flask CuI (1.0 equiv) was dissolved in MeCN (2.4 mL/mmol) under argon at room temperature. A solution of *N*-

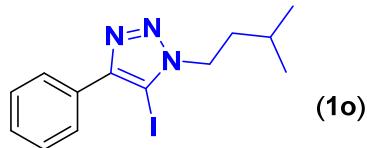
bromosuccinimide (1.2 equiv) in MeCN (2.4 mL/mmol) was added and stirred 5 min at room temperature. Later on, the previously prepared azide-containing solution along with alkyne (1.0 equiv) and DIPEA (1.1 equiv) were subsequently added and the resulting mixture was stirred at room temperature overnight. Next, the mixture was washed with HCl 10%, aq. NH<sub>4</sub>OH, extracted with AcOEt and washed with brine. The combined organic layers were dried over MgSO<sub>4</sub>, and concentrated under reduced pressure. The crude residue was purified by flash chromatography (hexanes/AcOEt 7/3) unless otherwise noted.



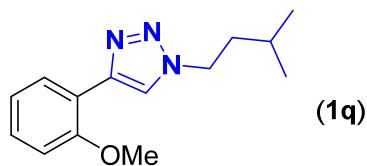
**Methyl 2-methyl-2-(4-phenyl-1*H*-1,2,3-triazol-1-yl)propanoate (1f) (Table 2).** Following the general procedure A, using *in situ* generated methyl 2-azido-2-methylpropanoate (7.4 mmol) and phenylacetylene (8.2 mmol, 0.9 mL) provided 216 mg (13% yield) of **1f** as a white solid. Mp 76-103 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 7.91 (s, 1H), 7.84 (d, *J* = 7.0 Hz, 2H), 7.41 (t, *J* = 7.7 Hz, 2H), 7.33 (d, *J* = 7.4 Hz, 1H), 3.73 (s, 3H), 1.99 (s, 6H) ppm. <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>): δ 171.9, 147.5, 130.7, 128.9, 128.2, 125.8, 118.5, 64.6, 53.4, 25.9 ppm. IR (neat, cm<sup>-1</sup>): 3130, 1739, 1626, 1279, 1163, 1157, 1081, 767, 695. MS (ESI<sup>+</sup>) *m/z* (%) 246 (M+H). HRMS *calcd.* for (C<sub>13</sub>H<sub>16</sub>N<sub>3</sub>O<sub>2</sub>): 246.1243, *found* 246.1240.



**1-[Bis(trimethylsilyl)methyl]-4-phenyl-1*H*-1,2,3-triazole (1i) (Table 2).** Following the general procedure B, using (chloromethylene)bis(trimethylsilane) (5.95 mmol, 1.3 mL) and phenylacetylene (5.95 mmol, 0.65 mL) provided 1.53 g (87% yield) of **1i** as a white solid. Mp 81-86 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 7.86-7.79 (m, 2H), 7.54 (s, 1H), 7.41 (t, *J* = 7.6 Hz, 2H), 7.34-7.27 (m, 1H), 3.65 (s, 1H), 0.13 (s, 18H) ppm. <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>): δ 147.0, 131.2, 128.9, 128.0, 125.6, 120.2, 46.9, -0.9 ppm. IR (neat, cm<sup>-1</sup>): 2882, 1249, 1045, 842, 760, 687. MS (ESI<sup>+</sup>) *m/z* (%) 304 (M+H). HRMS *calcd.* for (C<sub>15</sub>H<sub>26</sub>N<sub>3</sub>Si<sub>2</sub>): 304.1665, *found* 304.1656.



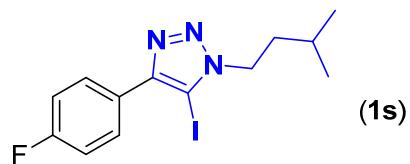
**5-Iodo-1-isopentyl-4-phenyl-1H-1,2,3-triazole (1o) (Table 3).** Following the general procedure C, using phenylacetylene (4.89 mmol, 0.54 mL) provided 1.00 g (66% yield) of **1o** as a white solid. Mp 97-98 °C.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.93 (d,  $J = 7.6$  Hz, 2H), 7.52-7.33 (m, 3H), 4.46 (t,  $J = 7.8$  Hz, 2H), 1.93-1.58 (m, 3H), 1.02 (d,  $J = 6.6$  Hz, 6H) ppm.  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ):  $\delta$  149.6, 130.3, 128.4, 127.4, 76.0, 49.4, 38.6, 25.6, 22.2 ppm. IR (neat,  $\text{cm}^{-1}$ ): 1447, 1224, 1064, 985. MS (ESI $^+$ )  $m/z$  (%) 342 (M+H). HRMS *calcd.* for ( $\text{C}_{13}\text{H}_{17}\text{N}_3\text{I}$ ): 342.0467, *found* 342.0462.



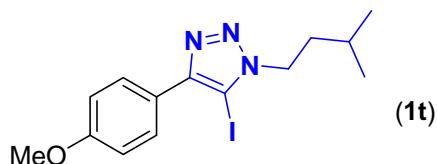
**1-Isopentyl-4-(2-methoxyphenyl)-1H-1,2,3-triazole (1q) (Table 3).** Following the general procedure B, using 1-ethynyl-2-methoxybenzene (3.78 mmol, 0.49 mL) provided 600 mg (69% yield) of **1q** as a yellow oil.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.31 (d,  $J = 7.7$  Hz, 1H), 7.97 (s, 1H), 7.18 (t,  $J = 7.9$  Hz, 1H), 6.97 (t,  $J = 7.5$  Hz, 1H), 6.85 (d,  $J = 8.3$  Hz, 1H), 4.26 (t,  $J = 7.6$  Hz, 2H), 3.79 (s, 3H), 1.71 (q,  $J = 7.3$  Hz, 2H), 1.50 (dt,  $J = 13.4$ , 6.7 Hz, 1H), 0.85 (d,  $J = 6.6$  Hz, 6H) ppm.  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ):  $\delta$  155.1, 142.4, 128.3, 126.9, 122.5, 120.3, 119.0, 110.3, 54.8, 48.0, 38.6, 25.0, 21.7 ppm. IR (neat,  $\text{cm}^{-1}$ ): 1538, 1243, 1069, 752. MS (ESI $^+$ )  $m/z$  (%) 246 (M+H). HRMS *calcd.* for ( $\text{C}_{14}\text{H}_{20}\text{N}_3\text{O}$ ): 246.1606, *found* 246.1607.



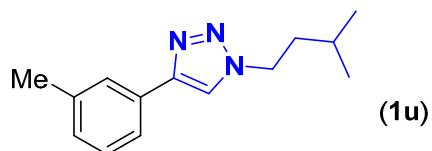
**1-Isopentyl-4-[2-(trifluoromethyl)phenyl]-1H-1,2,3-triazole (1r) (Table 3).** Following the general procedure B, using 1-ethynyl-2-(trifluoromethyl)benzene (2.94 mmol, 0.41 mL) provided 413 mg (50% yield) of **1r** as a white solid. Mp 40-41 °C.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.95 (d,  $J = 7.8$  Hz, 1H), 7.79-7.66 (m, 2H), 7.59 (t,  $J = 7.7$  Hz, 1H), 7.44 (t,  $J = 7.8$  Hz, 1H), 4.42 (t,  $J = 7.5$  Hz, 2H), 1.83 (q,  $J = 7.3$  Hz, 2H), 1.59 (dt,  $J = 13.5$ , 6.7 Hz, 1H), 0.95 (d,  $J = 6.6$  Hz, 6H) ppm.  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ):  $\delta$  144.0, 131.9, 131.5, 129.6, 128.1, 127.0 (d,  $J = 30$  Hz), 125.9 (q,  $J = 10$  Hz), 125.4, 122.6 (q,  $J = 10$  Hz), 48.7, 38.9, 25.4, 22.1 ppm. IR (neat,  $\text{cm}^{-1}$ ): 1581, 1312, 1100, 767. MS (ESI $^+$ )  $m/z$  (%) 284 (M+H). HRMS *calcd.* for ( $\text{C}_{14}\text{H}_{27}\text{N}_3\text{F}_3$ ): 284.1375, *found* 284.1381.



**4-(4-Fluorophenyl)-5-iodo-1-isopentyl-1*H*-1,2,3-triazole (**1s**) (Table 3).** Following the general procedure C, using 4-fluorophenylacetylene (4.16 mmol, 0.48 mL) provided 790 mg (53% yield) of **1s** as a white solid. Mp 118-120 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 8.00-7.75 (m, 2H), 7.13 (t, *J* = 8.7 Hz, 2H), 4.48-4.30 (m, 2H), 1.82 (ddd, *J* = 9.5, 7.8, 6.5 Hz, 2H), 1.68 (dt, *J* = 13.3, 6.7 Hz, 1H), 1.00 (d, *J* = 6.6 Hz, 6H) ppm. <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>): δ 162.3 (d, *J* = 247 Hz), 148.9, 129.3 (d, *J* = 9 Hz), 126.4 (d, *J* = 10 Hz), 115.4 (d, *J* = 21 Hz), 75.9, 49.4, 38.5, 25.6, 22.2 ppm. IR (neat, cm<sup>-1</sup>): 1607, 1476, 1223, 838. MS (ESI<sup>+</sup>) *m/z* (%) 360 (M+H). HRMS *calcd.* for (C<sub>13</sub>H<sub>16</sub>N<sub>3</sub>FI): 360.0373, *found* 360.0373.

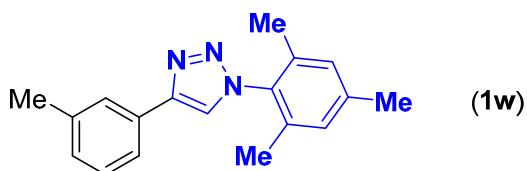


**5-Iodo-1-isopentyl-4-(4-methoxyphenyl)-1*H*-1,2,3-triazole (**1t**) (Table 3).** Following the general procedure C, using 4-methoxyphenylacetylene (3.78 mmol, 500 mg) provided 600 mg (43% yield) of **1t** as a white solid. Mp 95-96 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 7.85 (d, *J* = 8.3 Hz, 2H), 6.98 (d, *J* = 8.3 Hz, 2H), 4.43 (t, *J* = 7.8 Hz, 2H), 3.84 (s, 3H), 1.95-1.60 (m, 3H), 1.00 (d, *J* = 6.6 Hz, 6H) ppm. <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>): δ 159.7, 149.5, 128.7, 122.8, 113.9, 75.3, 55.2, 49.4, 38.6, 25.6, 22.2 ppm. IR (neat, cm<sup>-1</sup>): 1614, 1339, 1117, 1066. MS (ESI<sup>+</sup>) *m/z* (%) 372 (M+H). HRMS *calcd.* for (C<sub>14</sub>H<sub>19</sub>N<sub>3</sub>OI): 372.0573, *found* 372.0565.

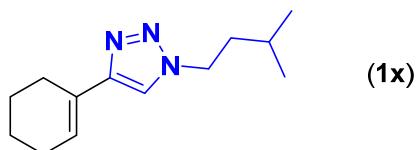


**1-Isopentyl-4-(*m*-tolyl)-1*H*-1,2,3-triazole (**1u**) (Table 3).** Following the general procedure B, using 1-ethynyl-3-methylbenzene (8.61 mmol, 1.09 mL) provided 1.70 g (86% yield) of **1u** as a white solid. Mp 51-52 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 7.76 (s, 1H), 7.70 (s, 1H), 7.61 (d, *J* = 7.7 Hz, 1H), 7.30 (t, *J* = 7.7 Hz, 1H), 7.14 (d, *J* = 7.5 Hz, 1H), 4.39 (t, *J* = 7.5 Hz, 2H), 2.40 (s, 3H), 1.83 (q, *J* = 7.3 Hz, 2H), 1.61 (dt, *J* = 13.4, 6.7 Hz, 1H), 0.97 (d, *J* = 6.6 Hz, 6H) ppm. <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>): δ 147.6, 138.3, 130.5, 128.6, 128.5, 126.2, 122.6, 119.2, 48.5, 38.9, 25.3, 22.0, 21.3 ppm. IR (neat, cm<sup>-1</sup>):

1433, 1079, 839, 716. MS (ESI<sup>+</sup>) *m/z* (%) 230 (M+H). HRMS *calcd.* for (C<sub>14</sub>H<sub>20</sub>N<sub>3</sub>): 230.1657, *found* 230.1661.



**1-Mesityl-4-(*m*-tolyl)-1*H*-1,2,3-triazole (**1w**) (Table 3).** Following the general procedure A, using 2-azido-1,3,5-trimethylbenzene<sup>1</sup> (3.90 mmol, 630 mg) and 3-ethynyltoluene (4.30 mmol, 0.54 mL) provided 1.05 g (97% yield) of **1w** as a brown solid. Mp 87–92 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 7.82 (d, *J* = 12.4 Hz, 2H), 7.71 (d, *J* = 7.7 Hz, 1H), 7.34 (t, *J* = 7.7 Hz, 1H), 7.17 (d, *J* = 7.7 Hz, 1H), 7.00 (s, 2H), 2.42 (s, 3H), 2.39 (s, 3H), 2.01 (s, 6H) ppm. <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>): δ 147.7, 140.0, 138.6, 135.1, 133.5, 130.4, 129.1, 129.0, 128.8, 126.4, 122.9, 121.5, 21.5, 21.2, 17.3 ppm. IR (neat, cm<sup>−1</sup>): 2918, 1491, 1378, 1226, 1036, 784, 695. MS (ESI<sup>+</sup>) *m/z* (%) 278 (M+H). HRMS *calcd.* for (C<sub>18</sub>H<sub>20</sub>N<sub>3</sub>): 278.1657, *found* 278.1654.



**4-(Cyclohex-1-en-1-yl)-1-isopentyl-1*H*-1,2,3-triazole (**1x**) (Table 3).** Following the general procedure B, using 1-bromo-3-methylbutane (10 mmol, 1.20 mL) and 1-ethynylcyclohexene (10 mmol, 1.30 mL) provided 1.80 g (82% yield) of **1x** as a white solid. Mp 38–42 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 7.39 (s, 1H), 6.46 (s, 1H), 4.30 (t, *J* = 7.5 Hz, 2H), 2.36 (s, 2H), 2.15 (s, 2H), 1.73 (p, *J* = 6.7, 6.0 Hz, 4H), 1.63 (p, *J* = 6.1 Hz, 2H), 1.54 (dt, *J* = 13.4, 6.6 Hz, 1H), 0.92 (d, *J* = 6.6 Hz, 6H) ppm. <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>): δ 149.4, 127.4, 124.6, 118.1, 48.5, 39.1, 26.3, 25.4, 25.2, 22.5, 22.2, 22.1 ppm. IR (neat, cm<sup>−1</sup>): 2951, 2929, 2868, 2837, 1459, 1434, 1216, 1052, 919, 832. MS (ESI<sup>+</sup>) *m/z* (%) 220 (M+H). HRMS *calcd.* for (C<sub>13</sub>H<sub>22</sub>N<sub>3</sub>): 220.1814, *found* 220.1804.

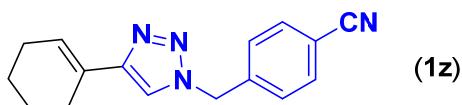


**Benzyl 2-(4-(cyclohex-1-en-1-yl)-1*H*-1,2,3-triazol-1-yl)acetate (**1y**) (Table 3).** Following the general procedure A, using benzyl 2-azidoacetate<sup>2</sup> (9 mmol, 1.73 g) and 1-ethynylcyclohexene (10 mmol, 1.20 mL) provided 2.30 g (85% yield) of **1y** as a white

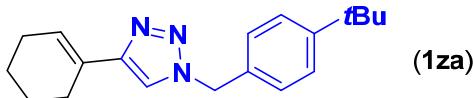
<sup>1</sup> Abram, S.-L.; Monte-Pérez, I.; Pfaff, F. F.; Farquhar, E. R.; Ray, K. *Chem. Commun.* **2014**, 50, 9852.

<sup>2</sup> Pokorski, J. K.; Jenkins, L. M. M.; Feng, H.; Durell, S. R.; Bai, Y.; Appella, D. H. *Org. Lett.* **2007**, 9, 2381.

solid. Mp 95-98 °C.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.51 (s, 1H), 7.36 (d,  $J = 7.3$  Hz, 5H), 6.54 (s, 1H), 5.19 (d,  $J = 21.5$  Hz, 4H), 2.38 (s, 2H), 2.20 (s, 2H), 1.72 (dd,  $J = 34.5, 5.9$  Hz, 4H) ppm.  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ):  $\delta$  166.4, 150.1, 134.7, 128.9, 128.8, 128.6, 127.1, 125.5, 119.7, 68.0, 50.9, 26.5, 25.4, 22.5, 22.3 ppm. IR (neat,  $\text{cm}^{-1}$ ): 2927, 2857, 2831, 1750, 1675, 1454, 1385, 1195, 942, 748, 698. MS (ESI $^+$ )  $m/z$  (%) 298 (M+H). HRMS *calcd.* for ( $\text{C}_{17}\text{H}_{20}\text{N}_3\text{O}_2$ ): 298.1556, *found* 298.1552.



**4-[(4-Cyclohex-1-en-1-yl)-1H-1,2,3-triazol-1-yl]methylbenzonitrile (1z) (Table 3).** Following the general procedure A, using 4-(azidomethyl)benzonitrile<sup>3</sup> (8.60 mmol, 1.36 g) and 1-ethynylcyclohexene (9.50 mmol, 1.00 mL) provided 2.20 g (97% yield) of **1z** as a white solid without requiring chromatographic purification. Mp 131-134 °C.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.62 (d,  $J = 7.9$  Hz, 2H), 7.36 (s, 1H), 7.30 (d,  $J = 7.9$  Hz, 2H), 6.50 (s, 1H), 5.56 (s, 2H), 2.32 (s, 2H), 2.16 (s, 2H), 1.68 (dd,  $J = 32.6, 5.9$  Hz, 4H) ppm.  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ):  $\delta$  150.4, 140.3, 132.9, 128.3, 127.0, 125.7, 118.5, 118.3, 112.6, 53.3, 26.4, 25.3, 22.4, 22.2 ppm. IR (neat,  $\text{cm}^{-1}$ ): 2934, 2860, 2831, 2232, 1446, 1314, 1197, 1036, 798, 768, 546. MS (ESI $^+$ )  $m/z$  (%) 265 (M+H). HRMS *calcd.* for ( $\text{C}_{16}\text{H}_{17}\text{N}_4$ ): 265.1453, *found* 265.1452.

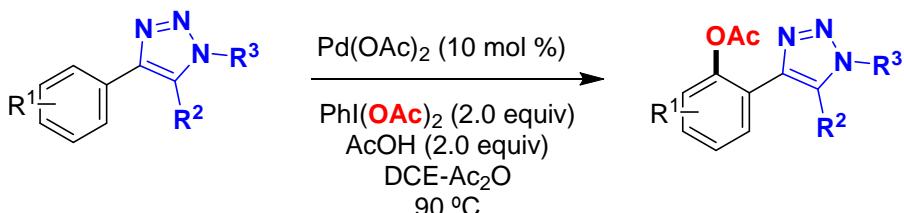


**1-[4-(Tert-butyl)benzyl]-4-(cyclohex-1-en-1-yl)-1H-1,2,3-triazole (1za) (Table 3).** Following the general procedure A, using 1-ethynylcyclohex-1-ene (9.42 mmol, 1.10 mL) and 4-(*tert*-butyl)benzyl azide<sup>4</sup> (10.4 mmol, 1.96 g) provided 2.76 g (99% yield) of **1za** as a white solid without requiring chromatographic purification. Mp 132-133 °C.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.40 (d,  $J = 8.0$  Hz, 2H), 7.33 (s, 1H), 7.22 (d,  $J = 7.9$  Hz, 2H), 6.50 (t,  $J = 4.0$  Hz, 1H), 5.48 (s, 2H), 2.37 (dq,  $J = 6.1, 3.3$  Hz, 2H), 2.25-2.12 (m, 2H), 1.82-1.56 (m, 4H), 1.33 (s, 9H) ppm.  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ):  $\delta$  151.6, 149.8, 131.9, 127.7, 127.3, 125.9, 124.9, 118.1, 53.6, 34.5, 31.2, 26.3, 25.2, 22.4, 22.1 ppm. IR (neat,  $\text{cm}^{-1}$ ): 1433, 1050, 710, 679. MS (ESI $^+$ )  $m/z$  (%) 296 (M+H). HRMS *calcd.* for ( $\text{C}_{19}\text{H}_{26}\text{N}_3$ ): 296.2127, *found* 296.2129.

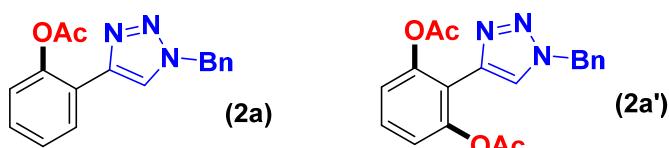
<sup>3</sup> Liu, M.; Reiser, O. *Org. Lett.* **2011**, *13*, 1102.

<sup>4</sup> Robinson, S. W.; Mustoe, C. L.; White, N. G.; Brown, A.; Thompson, A. L.; Kennepohl, P.; Beer, P. D. *J. Am. Chem. Soc.* **2015**, *137*, 499.

### 3.-Pd-Catalyzed C(sp<sup>2</sup>)-H Acetoxylation (Table 2-3)

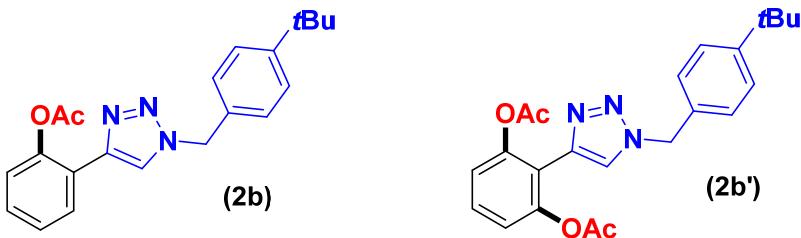


**General Procedure:** A reaction tube containing a stirring bar was charged with the corresponding triazole (if solid) (0.25 mmol, 1.0 equiv), PhI(OAc)<sub>2</sub> (0.50 mmol, 2.00 equiv) and Pd(OAc)<sub>2</sub> (10 mol %). The reaction tube was then evacuated and back-filled with dry argon (this sequence was repeated up to three times). The triazole (if liquid) (0.25 mmol, 1.00 equiv), AcOH (0.50 mmol, 2.00 equiv), 1,2-dichloroethane (0.50 mL) and Ac<sub>2</sub>O (0.50 mL) were then added under argon atmosphere. The reaction tube was next warmed up to 90 °C and stirred for 24 hours. The mixture was then allowed to warm to room temperature, filtered off through a pad of celite and washed with CH<sub>2</sub>Cl<sub>2</sub>. The resulting mixture was concentrated under reduced pressure and the corresponding product was purified by flash chromatography (hexanes/AcOEt 7/3). The yields reported in the manuscript refer to isolated yields and represent an average of at least two independent runs.



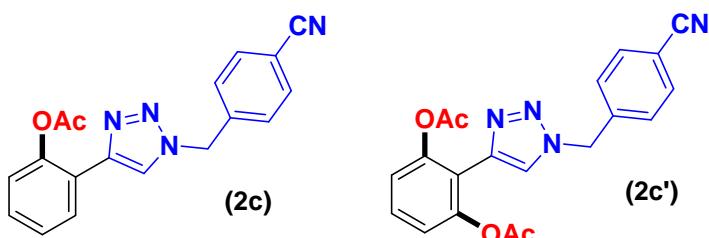
**2-(1-Benzyl-1*H*-1,2,3-triazol-4-yl)phenyl acetate (**2a**) (Table 2).** Following the general procedure, using 1-benzyl-4-phenyl-1*H*-1,2,3-triazole<sup>5</sup> (**1a**) (0.25 mmol, 59 mg) provided 33 mg (45% yield) of **2a** as a white solid along with 23 mg (25% yield) of **2a'** as a white solid. **2a**: Mp 162-163 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 8.07 (d, *J* = 7.4 Hz, 1H), 7.64 (s, 1H), 7.54-7.25 (m, 7H), 7.16 (d, *J* = 7.8 Hz, 1H), 5.59 (s, 2H), 2.16 (s, 3H) ppm. <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>): δ 168.8, 147.0, 143.3, 134.4, 129.1, 129.0, 128.8, 128.6, 128.2, 126.3, 123.2, 123.0, 121.7, 54.2, 21.0 ppm. IR (neat, cm<sup>-1</sup>): 1738, 1191, 852. MS (ESI<sup>+</sup>) *m/z* (%) 294 (M+H). HRMS *calcd.* for (C<sub>17</sub>H<sub>16</sub>N<sub>3</sub>O<sub>2</sub>): 294.1243, *found* 294.1243. **2a'**: Mp 172-173 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 7.53 (s, 1H), 7.44-7.34 (m, 3H), 7.29 (dd, *J* = 7.3, 2.1 Hz, 2H), 7.07 (d, *J* = 8.2 Hz, 2H), 5.58 (s, 2H), 2.04 (s, 6H) ppm. <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>): δ 168.9, 149.0, 139.3, 134.7, 129.1, 129.1, 128.8, 128.1, 123.2, 120.7, 54.1, 20.7 ppm. IR (neat, cm<sup>-1</sup>): 1735, 1197, 883. MS (ESI<sup>+</sup>) *m/z* (%) 352 (M+H). HRMS *calcd.* for (C<sub>19</sub>H<sub>18</sub>N<sub>3</sub>O<sub>4</sub>): 352.1297, *found* 352.1292.

<sup>5</sup> Sharghi, H.; Khalifeh, R.; Doroodmand, M. M. *Adv. Synth. Catal.* **2009**, 351, 207.



**2-[1-(4-(*tert*-butyl)benzyl)-1*H*-1,2,3-triazol-4-yl]phenyl acetate (**2b**) (Table 2).**

Following the general procedure, using 1-[4-*tert*-butyl]benzyl]-4-phenyl-1*H*-1,2,3-triazole<sup>6</sup> (**1b**) (0.25 mmol, 73 mg) provided 27.9 mg (32% yield) of **2b** as a white solid along with 30.5 mg (30% yield) of **2b'** as a white solid. **2b**: Mp 163-165 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 8.09 (d, *J* = 7.0 Hz, 1H), 7.62 (s, 1H), 7.45 (d, *J* = 7.9 Hz, 2H), 7.40-7.32 (m, 2H), 7.28 (d, *J* = 8.0 Hz, 2H), 7.16 (d, *J* = 8.1 Hz, 1H), 5.56 (s, 2H), 2.13 (s, 3H), 1.34 (s, 9H) ppm. <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>): δ 168.9, 152.2, 147.2, 143.4, 131.6, 129.0, 128.7, 128.2, 126.5, 126.2, 123.5, 123.1, 121.7, 54.0, 34.8, 31.3, 21.2 ppm. IR (neat, cm<sup>-1</sup>): 1750, 1202, 1178, 762. MS (ESI<sup>+</sup>) *m/z* (%) 350 (M+H). HRMS *calcd.* for (C<sub>21</sub>H<sub>24</sub>N<sub>3</sub>O<sub>2</sub>): 350.1869, *found* 350.1869. **2b'**: Mp 189-196 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.51 (s, 1H), 7.45-7.33 (m, 3H), 7.24 (d, *J* = 8.0 Hz, 2H), 7.06 (d, *J* = 8.2 Hz, 2H), 5.53 (s, 2H), 2.02 (s, 6H), 1.31 (s, 9H) ppm. <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>): δ 169.1, 152.3, 149.2, 139.5, 131.9, 129.3, 128.2, 126.3, 123.3, 121.0, 118.2, 54.0, 34.8, 31.4, 20.9 ppm. IR (neat, cm<sup>-1</sup>): 1752, 1750, 1216, 1187, 1028. MS (ESI<sup>+</sup>) *m/z* (%) 408 (M+H). HRMS *calcd.* for (C<sub>23</sub>H<sub>26</sub>N<sub>3</sub>O<sub>4</sub>): 408.1923, *found* 408.1927.

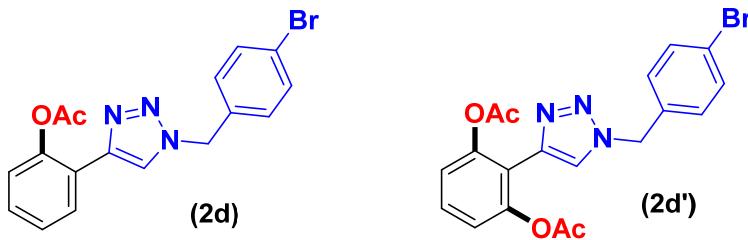


**2-[1-(4-Cyanobenzyl)-1*H*-1,2,3-triazol-4-yl]phenyl acetate (**2c**) (Table 2).** Following the general procedure, using 4-[(4-phenyl-1*H*-1,2,3-triazol-1-yl)methyl]benzonitrile<sup>7</sup> (**1c**) (0.25 mmol, 65 mg) provided 35 mg (44% yield) of **2c** as a yellow oil along with 25.1 mg (27% yield) of **2c'** as a white solid. **2c**: <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 8.05 (dd, *J* = 7.5, 2.1 Hz, 1H), 7.79 (s, 1H), 7.69 (d, *J* = 8.2 Hz, 2H), 7.46-7.32 (m, 4H), 7.21 (dd, *J* = 7.7, 1.7 Hz, 1H), 5.65 (s, 2H), 2.28 (s, 3H) ppm. <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>): δ 169.1, 147.2, 144.0, 139.9, 132.9, 129.3, 128.7, 128.4, 126.5, 123.2, 123.1, 122.0, 118.2, 112.7, 53.3, 21.3 ppm. IR (neat, cm<sup>-1</sup>): 1750, 1216, 1038, 765. MS (ESI<sup>+</sup>) *m/z* (%) 319 (M+H).

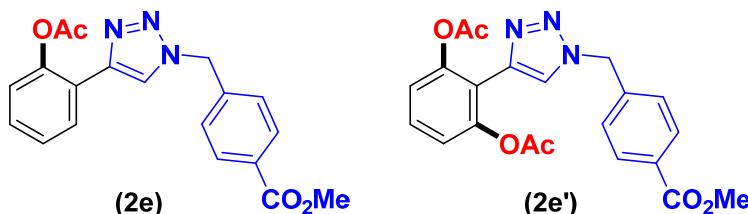
<sup>6</sup> Özçubukçu, S.; Ozkal, E.; Jimeno, C.; Pericàs, M. A. *Org. Lett.* **2009**, *11*, 4680.

<sup>7</sup> Bidal, Y. D.; Lesieur, M.; Melaimi, M.; Cordes, D. B.; Slawin, A. M. Z.; Bertrand, G.; Cazin, C. S. J. *Chem. Commun.* **2015**, *51*, 4778.

HRMS *calcd.* for (C<sub>18</sub>H<sub>15</sub>N<sub>4</sub>O<sub>2</sub>): 319.1195, *found* 319.1193. **2d'**: Mp 201-206 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.69 (d, *J* = 8.2 Hz, 2H), 7.62 (s, 1H), 7.41 (t, *J* = 8.2 Hz, 1H), 7.34 (d, *J* = 8.1 Hz, 2H), 7.09 (d, *J* = 8.2 Hz, 2H), 5.65 (s, 2H), 2.09 (s, 6H) ppm. <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>): δ 169.0, 149.3, 140.1, 133.1, 129.7, 128.4, 123.6, 121.0, 118.1, 117.9, 113.1, 53.5, 31.1, 21.0 ppm. IR (neat, cm<sup>-1</sup>): 1747, 1197, 1026, 884. MS (ESI<sup>+</sup>) *m/z* (%) 377 (M+H). HRMS *calcd.* for (C<sub>20</sub>H<sub>17</sub>N<sub>4</sub>O<sub>4</sub>): 377.1250, *found* 377.1240.



**2-[1-(4-Bromobenzyl)-1*H*-1,2,3-triazol-4-yl]phenyl acetate (2d) (Table 2).** Following the general procedure, using 1-(4-bromobenzyl)-4-phenyl-1*H*-1,2,3-triazole<sup>8</sup> (**1d**) (0.25 mmol, 78 mg) provided 34.4 mg (37% yield) of **2d** as a white solid along with 33.3 mg (31% yield) of **2d'** as a yellowish solid. **2d**: Mp 135-139 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 8.04 (d, *J* = 7.6 Hz, 1H), 7.66 (s, 1H), 7.54 (d, *J* = 8.1 Hz, 2H), 7.36 (t, *J* = 10.0 Hz, 2H), 7.19 (d, *J* = 9.0 Hz, 3H), 5.54 (s, 2H), 2.22 (s, 3H) ppm. <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>): δ 169.0, 147.3, 143.9, 133.7, 132.5, 129.8, 129.2, 128.8, 126.5, 123.3, 123.2, 123.1, 121.7, 53.6, 21.3 ppm. IR (neat, cm<sup>-1</sup>): 1754, 1199, 1189, 1010, 760. MS (ESI<sup>+</sup>) *m/z* (%) 372 (M+H). HRMS *calcd.* for (C<sub>17</sub>H<sub>15</sub>N<sub>3</sub>O<sub>2</sub>Br): 372.0348, *found* 372.0349. **2d'**: Mp 179-199 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.57-7.49 (m, 3H), 7.39 (t, *J* = 8.2 Hz, 1H), 7.15 (d, *J* = 8.1 Hz, 2H), 7.07 (d, *J* = 8.2 Hz, 2H), 5.52 (s, 2H), 2.07 (s, 6H) ppm. <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>): δ 169.1, 149.2, 139.8, 133.9, 132.5, 129.8, 129.5, 123.4, 123.2, 121.0, 118.0, 53.6, 21.0 ppm. IR (neat, cm<sup>-1</sup>): 1744, 1196, 1029, 883. MS (ESI<sup>+</sup>) *m/z* (%) 430 (M+H). HRMS *calcd.* for (C<sub>19</sub>H<sub>17</sub>N<sub>3</sub>O<sub>4</sub>Br): 430.0402, *found* 430.0405.

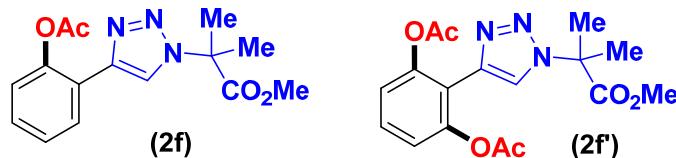


**Methyl 4-[(4-(2-acetoxyphenyl)-1*H*-1,2,3-triazol-1-yl)methyl]benzoate (2e) (Table 2).** Following the general procedure, using methyl 4-[(4-phenyl-1*H*-1,2,3-triazol-1-yl)methyl]benzoate<sup>9</sup> (**1e**) (0.25 mmol, 73 mg) provided 29 mg (33% yield) of **2e** as a white solid along with 28 mg (28% yield) of **2e'** as a white solid. **2e**: Mp 162-163 °C. <sup>1</sup>H

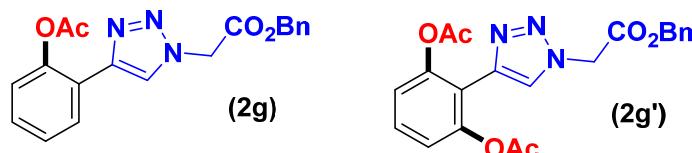
<sup>8</sup> Bent, S. J.; Mahon, M. F.; Webster, R. L. *Dalton Trans* **2015**, 44, 10253.

<sup>9</sup> Shin, J.-A.; Lim, Y.-G.; Lee, K.-H. *J. Org. Chem.* **2012**, 77, 4117.

NMR (400 MHz, CDCl<sub>3</sub>):  $\delta$  8.05 (t,  $J$  = 7.9 Hz, 3H), 7.70 (s, 1H), 7.35 (q,  $J$  = 6.0 Hz, 4H), 7.16 (d,  $J$  = 7.9 Hz, 1H), 5.63 (s, 2H), 3.93 (s, 3H), 2.20 (s, 3H) ppm. <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>):  $\delta$  168.9, 166.2, 147.1, 143.7, 139.4, 130.5, 130.3, 129.1, 128.6, 127.7, 126.3, 123.1, 121.7, 53.6, 52.2, 21.1 ppm. IR (neat, cm<sup>-1</sup>): 1743, 1720, 1196, 726. MS (ESI<sup>+</sup>) *m/z* (%) 352 (M+H). HRMS *calcd.* for (C<sub>19</sub>H<sub>18</sub>N<sub>3</sub>O<sub>4</sub>): 352.1297, *found* 352.1289. **2e'**: Mp 172-173 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):  $\delta$  8.05 (d,  $J$  = 8.0 Hz, 2H), 7.59 (s, 1H), 7.39 (t,  $J$  = 8.0 Hz, 1H), 7.31 (d,  $J$  = 7.0 Hz, 2H), 7.07 (d,  $J$  = 8.2 Hz, 2H), 5.63 (s, 2H), 3.92 (s, 3H), 2.06 (s, 6H) ppm. <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>):  $\delta$  168.9, 166.2, 149.1, 139.7, 139.6, 130.7, 130.4, 129.4, 127.7, 123.4, 120.8, 117.9, 53.6, 52.3, 20.8 ppm. IR (neat, cm<sup>-1</sup>): 1741, 1716, 1194, 729. MS (ESI<sup>+</sup>) *m/z* (%) 410 (M+H). HRMS *calcd.* for (C<sub>21</sub>H<sub>20</sub>N<sub>3</sub>O<sub>6</sub>): 410.1352, *found* 410.1342.



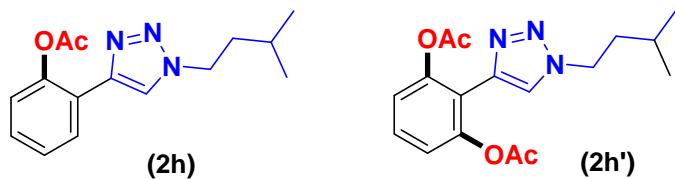
**Methyl 2-[4-(2-acetoxyphenyl)-1*H*-1,2,3-triazol-1-yl]-2-methylpropanoate (2f) (Table 2).** Following the general procedure, using triazole **1f** (0.25 mmol, 61 mg) at 110 °C provided 22.7 mg (30% yield) of **2f** as a yellowish oil along with 46 mg (51% yield) of **2f'** as a pale brown solid. **2f**: <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):  $\delta$  8.06 (dd,  $J$  = 7.6, 2.0 Hz, 1H), 7.93 (s, 1H), 7.41-7.30 (m, 2H), 7.18 (dd,  $J$  = 7.8, 1.6 Hz, 1H), 3.75 (s, 3H), 2.35 (s, 3H), 1.99 (s, 6H) ppm. <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>):  $\delta$  171.9, 169.2, 147.4, 143.1, 129.2, 128.9, 126.6, 123.6, 123.2, 120.9, 64.6, 53.5, 26.1, 21.4 ppm. IR (neat, cm<sup>-1</sup>): 1751, 1749, 1172, 1009, 831. MS (ESI<sup>+</sup>) *m/z* (%) 304 (M+H). HRMS *calcd.* for (C<sub>15</sub>H<sub>18</sub>N<sub>3</sub>O<sub>4</sub>): 304.1297, *found* 304.1287. **2f'**: Mp 90-94 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.84 (s, 1H), 7.39 (t,  $J$  = 8.2 Hz, 1H), 7.09 (d,  $J$  = 8.2 Hz, 2H), 3.73 (s, 3H), 2.20 (s, 6H), 1.96 (s, 6H) ppm. <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>):  $\delta$  171.8, 169.2, 149.2, 138.6, 129.3, 122.9, 120.9, 118.2, 64.6, 53.4, 25.8, 21.0 ppm. IR (neat, cm<sup>-1</sup>): 1743, 1741, 1186, 1184, 1024. MS (ESI<sup>+</sup>) *m/z* (%) 362 (M+H). HRMS *calcd.* for (C<sub>17</sub>H<sub>20</sub>N<sub>3</sub>O<sub>6</sub>): 362.1352, *found* 362.1347.



**Benzyl 2-[4-(2-acetoxyphenyl)-1*H*-1,2,3-triazol-1-yl] acetate (2g) (Table 2).** Following the general procedure, using benzyl 2-(4-phenyl-1*H*-1,2,3-triazol-1-yl)acetate<sup>10</sup> (**1g**) (0.25

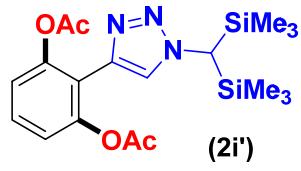
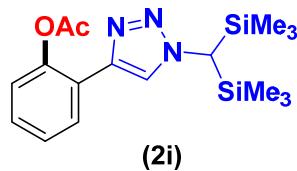
<sup>10</sup> Song, J. H.; Choi, P.; Lee, S. E.; Jeong, K. H.; Kim, T.; Kang, K. S.; Choi, Y. S.; Ham, J. *Eur. J. Org. Chem.* **2013**, 6249.

mmol, 73 mg) at 110 °C provided 41.3 mg (47% yield) of **2g** as a yellowish solid along with 24.6 mg (24% yield) of **2g'** as a white solid. **2g**: Mp 81-87 °C.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.06 (d,  $J = 7.6$  Hz, 1H), 7.94 (s, 1H), 7.44-7.28 (m, 7H), 7.18 (d,  $J = 7.8$  Hz, 1H), 5.24 (s, 4H), 2.30 (s, 3H) ppm.  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ):  $\delta$  169.2, 166.2, 147.4, 143.6, 134.6, 129.3, 129.0, 128.9, 128.8, 128.7, 126.5, 123.4, 123.2, 123.2, 68.2, 51.0, 21.3 ppm. IR (neat,  $\text{cm}^{-1}$ ): 1749, 1261, 1175, 696. MS (ESI $^+$ )  $m/z$  (%) 352 (M+H). HRMS *calcd.* for ( $\text{C}_{19}\text{H}_{18}\text{N}_3\text{O}_4$ ): 352.1297, *found* 352.1292. **2g'**: Mp 181-187 °C.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.84 (s, 1H), 7.47-7.31 (m, 6H), 7.10 (d,  $J = 8.2$  Hz, 2H), 5.23 (s, 4H), 2.19 (s, 6H) ppm.  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ):  $\delta$  169.3, 166.2, 149.4, 139.4, 134.6, 129.6, 129.0, 128.9, 128.7, 125.2, 120.9, 118.1, 68.2, 51.0, 21.0 ppm. IR (neat,  $\text{cm}^{-1}$ ): 1760, 1744, 1222, 1185, 1030. MS (ESI $^+$ )  $m/z$  (%) 410 (M+H). HRMS *calcd.* for ( $\text{C}_{21}\text{H}_{20}\text{N}_3\text{O}_6$ ): 410.1352, *found* 410.1353.



**2-(1-Isopentyl-1*H*-1,2,3-triazol-4-yl)phenyl acetate (**2h**) (Table 2).** Following the general procedure, using 1-(isopentyl)-4-phenyl-1*H*-1,2,3-triazole<sup>11</sup> (**1h**) (0.25 mmol, 54 mg) at 110 °C provided 28.7 mg (42% yield) of **2h** as a white solid along with 36.4 mg (44% yield) of **2h'** as a yellowish solid. **2h**: Mp 83-90 °C.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.02 (d,  $J = 6.4$  Hz, 1H), 7.72 (s, 1H), 7.32 (p,  $J = 7.2, 6.7$  Hz, 2H), 7.15 (d,  $J = 7.7$  Hz, 1H), 4.38 (t,  $J = 7.5$  Hz, 2H), 2.32 (s, 3H), 1.80 (q,  $J = 7.3$  Hz, 2H), 1.59 (dt,  $J = 13.5, 6.7$  Hz, 1H), 0.95 (d,  $J = 6.6$  Hz, 6H) ppm.  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ):  $\delta$  169.1, 147.2, 143.2, 129.0, 128.8, 126.4, 123.6, 123.1, 121.5, 48.7, 39.1, 25.5, 22.2, 21.4 ppm. IR (neat,  $\text{cm}^{-1}$ ): 1758, 1371, 1189, 758. MS (ESI $^+$ )  $m/z$  (%) 274 (M+H). HRMS *calcd.* for ( $\text{C}_{15}\text{H}_{20}\text{N}_3\text{O}_2$ ): 274.1556, *found* 274.1553. **2h'**: Mp 136-144 °C.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.64 (s, 1H), 7.39 (t,  $J = 8.2$  Hz, 1H), 7.09 (d,  $J = 8.1$  Hz, 2H), 4.41 (t,  $J = 7.4$  Hz, 2H), 2.19 (s, 6H), 1.81 (q,  $J = 7.1$  Hz, 2H), 1.57 (dt,  $J = 13.3, 6.8$  Hz, 1H), 0.96 (d,  $J = 6.6$  Hz, 6H) ppm.  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ):  $\delta$  169.1, 149.3, 138.9, 129.3, 123.3, 120.9, 118.3, 48.8, 39.2, 25.6, 22.3, 21.1 ppm. IR (neat,  $\text{cm}^{-1}$ ): 1747, 1197, 1027, 883. MS (ESI $^+$ )  $m/z$  (%) 332 (M+H). HRMS *calcd.* for ( $\text{C}_{17}\text{H}_{22}\text{N}_3\text{O}_4$ ): 332.1610, *found* 332.1622.

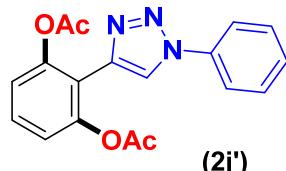
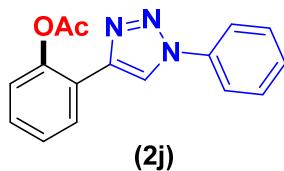
<sup>11</sup> Chen, Z.; Yan, Q.; Yi, H.; Liu, Z.; Lei, A.; Zhang, Y. *Chem. Eur. J.* **2014**, *20*, 13692.



**2-[1-(Bis(trimethylsilyl)methyl)-1*H*-1,2,3-triazol-4-yl]phenyl acetate (2i) (Table 2).**

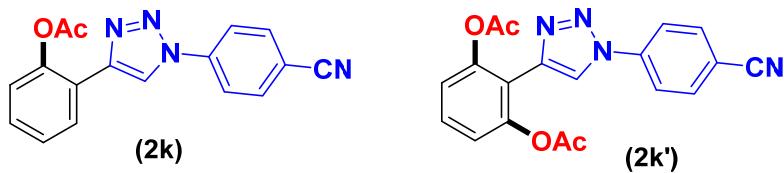
Following the general procedure, using triazole **1i** (0.25 mmol, 76 mg) provided 18.9 mg (21% yield) of **2i** as a white solid along with 47.2 mg (45% yield) of **2i'** as a white solid.

**1i:** Mp 139-143 °C.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.15-8.09 (m, 1H), 7.55 (s, 1H), 7.33 (dt,  $J = 6.0, 2.5$  Hz, 2H), 7.19-7.14 (m, 1H), 3.68 (s, 1H), 2.32 (s, 3H), 0.12 (s, 18H) ppm.  $^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ ):  $\delta$  168.9, 147.2, 142.3, 128.8, 126.5, 123.9, 123.0, 122.4, 46.7, 21.5, -1.0 ppm. IR (neat,  $\text{cm}^{-1}$ ): 1749, 1249, 1197, 843, 765. MS ( $\text{ESI}^+$ )  $m/z$  (%) 362 (M+H). HRMS *calcd.* for ( $\text{C}_{17}\text{H}_{28}\text{N}_3\text{O}_2\text{Si}$ ): 362.1720, *found* 362.1716. **1i':** Mp 137-139 °C.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.42-7.34 (m, 2H), 7.07 (d,  $J = 8.2$  Hz, 2H), 3.66 (s, 1H), 2.13 (s, 6H), 0.10 (s, 18H) ppm.  $^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ ):  $\delta$  168.9, 149.4, 138.0, 129.2, 124.0, 120.6, 119.0, 46.6, 21.0, -1.1 ppm. IR (neat,  $\text{cm}^{-1}$ ): 1764, 1760, 1217, 1189, 1027, 843. MS ( $\text{ESI}^+$ )  $m/z$  (%) 420 (M+H). HRMS *calcd.* for ( $\text{C}_{19}\text{H}_{30}\text{N}_3\text{O}_4\text{Si}$ ): 420.1775, *found* 420.1769.

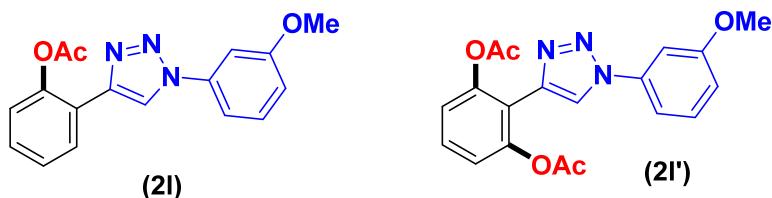


**2-(1-Phenyl-1*H*-1,2,3-triazol-4-yl)phenyl acetate (2j) (Table 2).** Following the general procedure, using 1,4-diphenyl-1*H*-1,2,3-triazole<sup>12</sup> (**1j**) (0.25 mmol, 55 mg) provided 21 mg (30% yield) of **2j** as a white solid along with 21.1 mg (25% yield) of **2j'** as a yellowish solid. **2j:** Mp 117-119 °C.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.25 (s, 1H), 8.16 (dd,  $J = 7.2, 1.9$  Hz, 1H), 7.80 (d,  $J = 7.2$  Hz, 2H), 7.58 (t,  $J = 7.7$  Hz, 2H), 7.54-7.36 (m, 3H), 7.26 (dd,  $J = 7.6, 1.7$  Hz, 1H), 2.43 (s, 3H) ppm.  $^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ ):  $\delta$  169.2, 147.4, 144.0, 137.0, 129.9, 129.3, 128.9, 128.8, 126.5, 123.3, 123.1, 120.6, 119.8, 21.5 ppm. IR (neat,  $\text{cm}^{-1}$ ): 1742, 1506, 1214, 1189, 1036, 753. MS ( $\text{ESI}^+$ )  $m/z$  (%) 280 (M+H). HRMS *calcd.* for ( $\text{C}_{16}\text{H}_{14}\text{N}_3\text{O}_2$ ): 280.1086, *found* 280.1083. **2j':** Mp 130-140 °C.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.12 (s, 1H), 7.79-7.73 (m, 2H), 7.55 (t,  $J = 7.8$  Hz, 2H), 7.50-7.39 (m, 2H), 7.13 (d,  $J = 8.2$  Hz, 2H), 2.24 (s, 6H) ppm.  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ):  $\delta$  169.2, 149.3, 139.8, 136.9, 130.0, 129.5, 129.1, 121.5, 121.1, 120.6, 117.8, 21.2 ppm. IR (neat,  $\text{cm}^{-1}$ ): 1744, 1368, 1187, 1024, 762. MS ( $\text{ESI}^+$ )  $m/z$  (%) 338 (M+H). HRMS *calcd.* for ( $\text{C}_{18}\text{H}_{16}\text{N}_3\text{O}_4$ ): 338.1141, *found* 338.1139.

<sup>12</sup> Deraedt, C.; Pinaud, N.; Astruc, D. *J. Am. Chem. Soc.* **2014**, *136*, 12092.



**2-[1-(4-Cyanophenyl)-1*H*-1,2,3-triazol-4-yl]phenyl acetate (**2k**) (Table 2).** Following the general procedure, using 4-(4-phenyl-1*H*-1,2,3-triazol-1-yl)benzonitrile<sup>13</sup> (**1k**) (0.25 mmol, 62 mg) provided 29 mg (38% yield) of **2k** as a white solid along with 24.4 mg (27% yield) of **2k'** as a yellowish solid. **2k:** Mp 201–207 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 8.24 (s, 1H), 8.07 (dd, *J* = 7.6, 1.8 Hz, 1H), 7.90 (dd, *J* = 33.1, 8.4 Hz, 4H), 7.47–7.33 (m, 2H), 7.23 (d, *J* = 8.0 Hz, 1H), 2.38 (s, 3H) ppm. <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>): δ 169.1, 147.6, 144.9, 139.8, 134.2, 129.9, 129.0, 126.7, 123.5, 122.6, 120.7, 119.3, 117.8, 112.7, 21.6 ppm. IR (neat, cm<sup>−1</sup>): 2223, 1748, 1519, 1215, 1185, 1025, 838. MS (ESI<sup>+</sup>) *m/z* (%) 305 (M+H). HRMS *calcd.* for (C<sub>17</sub>H<sub>13</sub>N<sub>4</sub>O<sub>2</sub>): 305.1039, *found* 305.1037. **2k':** Mp 142–158 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.19 (s, 1H), 7.95–7.80 (m, 4H), 7.43 (t, *J* = 8.2 Hz, 1H), 7.12 (d, *J* = 8.2 Hz, 2H), 2.22 (s, 6H) ppm. <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>): δ 169.1, 149.2, 140.4, 139.6, 134.1, 129.9, 121.2, 121.1, 120.6, 117.8, 117.2, 112.5, 21.1 ppm. IR (neat, cm<sup>−1</sup>): 2231, 1754, 1190, 1031, 836. MS (ESI<sup>+</sup>) *m/z* (%) 363 (M+H). HRMS *calcd.* for (C<sub>19</sub>H<sub>15</sub>N<sub>4</sub>O<sub>4</sub>): 363.1093, *found* 363.1082.

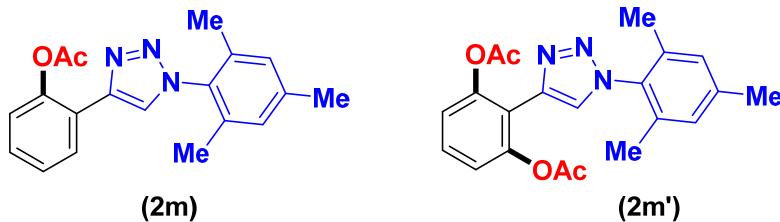


**2-[1-(3-Methoxyphenyl)-1*H*-1,2,3-triazol-4-yl]phenyl acetate (**2l**) (Table 2).** Following the general procedure, using 1-(3-methoxyphenyl)-4-phenyl-1*H*-1,2,3-triazole<sup>14</sup> (**1l**) (0.25 mmol, 63 mg) provided 30.2 mg (39% yield) of **2l** as a white solid along with 21.1 mg (23% yield) of **2l'** as a yellowish solid. **2l:** Mp 117–121 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 8.21 (s, 1H), 8.14 (dd, *J* = 7.6, 2.0 Hz, 1H), 7.49–7.35 (m, 4H), 7.27 (ddd, *J* = 24.7, 7.8, 1.2 Hz, 2H), 7.02 (dd, *J* = 8.3, 1.9 Hz, 1H), 3.92 (s, 3H), 2.41 (s, 3H) ppm. <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>): δ 169.2, 160.7, 147.4, 143.9, 138.0, 130.7, 129.4, 129.0, 126.5, 123.3, 123.1, 119.9, 114.7, 112.4, 106.6, 55.7, 21.5 ppm. IR (neat, cm<sup>−1</sup>): 1738, 1610, 1499, 1212, 1158, 1040, 754. MS (ESI<sup>+</sup>) *m/z* (%) 310 (M+H). HRMS *calcd.* for (C<sub>17</sub>H<sub>16</sub>N<sub>3</sub>O<sub>3</sub>): 310.1192, *found* 310.1194. **2l':** Mp 126–132 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.16 (s, 1H), 7.48 (t, *J* = 8.2 Hz, 2H), 7.43 (t, *J* = 2.3 Hz, 1H), 7.32 (dd, *J* = 7.9, 2.0 Hz, 1H), 7.18 (d, *J* = 8.2 Hz, 2H), 7.05 (dd, *J* = 8.3, 2.4 Hz, 1H), 3.94 (s, 3H), 2.29 (s, 6H) ppm. <sup>13</sup>C

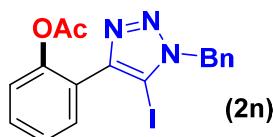
<sup>13</sup> Ramachary, D. B.; Shashank, A. B.; Karthik, S. *Angew. Chem. Int. Ed.* **2014**, *53*, 10420.

<sup>14</sup> Chen, Z.; Yan, Q.; Liu, Z.; Xu, Y.; Zhang, Y. *Angew. Chem. Int. Ed.* **2013**, *52*, 13324.

NMR (126 MHz, CDCl<sub>3</sub>):  $\delta$  169.2, 160.8, 149.3, 139.7, 137.9, 130.8, 129.5, 121.5, 121.1, 117.8, 114.8, 112.4, 106.5, 55.8, 21.2 ppm. IR (neat, cm<sup>-1</sup>): 1749, 1747, 1610, 1484, 1195, 1028. MS (ESI<sup>+</sup>)  $m/z$  (%) 368 (M+H). HRMS *calcd.* for (C<sub>19</sub>H<sub>18</sub>N<sub>3</sub>O<sub>5</sub>): 368.1246, *found* 368.1245.



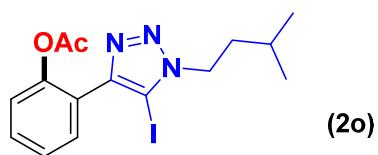
**2-(1-Mesityl-1*H*-1,2,3-triazol-4-yl)phenyl acetate (**2m**) (Table 2).** Following the general procedure, using 1-mesityl-4-phenyl-1*H*-1,2,3-triazole<sup>15</sup> (**1m**) (0.25 mmol, 66 mg) at 110 °C provided 19 mg (24% yield) of **1m** as a white solid along with 61.2 mg (64% yield) of **1m'** as white solid. **1m**: Mp 107-117 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):  $\delta$  8.20 (dd,  $J$  = 6.8, 2.6 Hz, 1H), 7.84 (s, 1H), 7.44-7.33 (m, 2H), 7.19 (s, 1H), 7.02 (s, 2H), 2.34 (s, 3H), 2.31 (s, 3H), 2.00 (s, 6H) ppm. <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>):  $\delta$  168.9, 147.3, 143.0, 140.2, 135.2, 133.4, 129.2, 129.1, 128.8, 126.5, 123.7, 123.4, 123.1, 21.4, 21.2, 17.4 ppm. IR (neat, cm<sup>-1</sup>): 1763, 1481, 1207, 1184, 1047, 758. MS (ESI<sup>+</sup>)  $m/z$  (%) 322 (M+H). HRMS *calcd.* for (C<sub>19</sub>H<sub>20</sub>N<sub>3</sub>O<sub>2</sub>): 322.1556, *found* 322.1553. **1m'**: Mp 155-164 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):  $\delta$  7.70 (s, 1H), 7.35 (t,  $J$  = 8.2 Hz, 1H), 7.07 (d,  $J$  = 8.2 Hz, 2H), 6.95 (s, 2H), 2.28 (s, 3H), 2.11 (s, 6H), 1.92 (s, 6H) ppm. <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):  $\delta$  168.5, 149.1, 139.9, 138.5, 134.7, 133.0, 129.2, 128.9, 125.3, 120.5, 118.0, 20.9, 20.6, 16.9 ppm. IR (neat, cm<sup>-1</sup>): 1745, 1496, 1457, 1185, 1024. MS (ESI<sup>+</sup>)  $m/z$  (%) 380 (M+H). HRMS *calcd.* for (C<sub>21</sub>H<sub>22</sub>N<sub>3</sub>O<sub>4</sub>): 380.1610, *found* 380.1602.



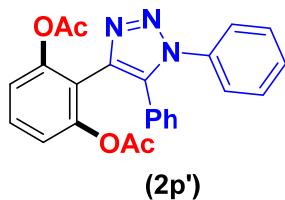
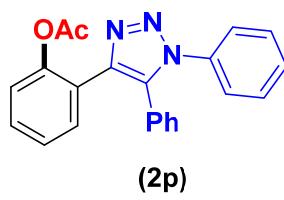
**2-(1-Benzyl-5-iodo-1*H*-1,2,3-triazol-4-yl)phenyl acetate (**2n**) (Table 2).** Following the general procedure, using 1-benzyl-5-iodo-4-phenyl-1*H*-1,2,3-triazole<sup>16</sup> (**1n**) (0.25 mmol, 90 mg) provided 71 mg (68% yield) of **2n** as a white solid. Mp 130-131 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):  $\delta$  7.61 (d,  $J$  = 7.7 Hz, 1H), 7.53-7.15 (m, 8H), 5.68 (s, 2H), 2.13 (s, 3H) ppm. <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>):  $\delta$  169.0, 148.5, 134.3, 131.0, 130.1, 128.9, 128.5, 127.7, 125.9, 123.3, 123.1, 79.7, 54.5, 21.0 ppm. IR (neat, cm<sup>-1</sup>): 1737, 1195, 912, 761. MS (ESI<sup>+</sup>)  $m/z$  (%) 420 (M+H). HRMS *calcd.* for (C<sub>17</sub>H<sub>15</sub>N<sub>3</sub>O<sub>2</sub>I): 420.0209, *found*

<sup>15</sup> Sau, S. C.; Roy, S. R.; Sen, T. K.; Mullangi, D.; Mandal, S. K. *Adv. Synth. Catal.* **2013**, 355, 2982.

<sup>16</sup> Zhou, Y.; Lecourt, T.; Micouin, L. *Angew. Chem. Int. Ed.* **2010**, 49, 2607.

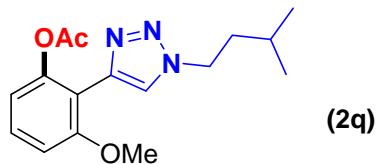


**2-(5-Iodo-1-isopentyl-1H-1,2,3-triazol-4-yl)phenyl acetate (2o) (Table 2).** Following the general procedure, using triazole **1o** (0.25 mmol, 85 mg) provided 98 mg (98% yield) of **2o** as a white solid. Mp 94-95 °C.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.62 (d,  $J$  = 7.7 Hz, 1H), 7.48 (t,  $J$  = 7.4 Hz, 1H), 7.36 (t,  $J$  = 7.5 Hz, 1H), 7.27 (t,  $J$  = 7.7 Hz, 1H), 4.60-4.36 (m, 2H), 2.20 (s, 3H), 1.88 (q,  $J$  = 7.3 Hz, 2H), 1.70 (dq,  $J$  = 13.7, 6.3 Hz, 1H), 1.04 (d,  $J$  = 6.6 Hz, 6H) ppm.  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ):  $\delta$  169.1, 148.5, 147.7, 131.1, 130.0, 125.8, 123.4, 123.2, 79.1, 49.6, 38.6, 25.7, 22.3, 21.1 ppm. IR (neat,  $\text{cm}^{-1}$ ): 1753, 1197, 907, 768. MS ( $\text{ESI}^+$ )  $m/z$  (%) 400 (M+H). HRMS *calcd.* for ( $\text{C}_{15}\text{H}_{19}\text{N}_3\text{O}_2\text{I}$ ): 400.0522, *found* 400.0522.



**2-(1,5-Diphenyl-1H-1,2,3-triazol-4-yl)phenyl acetate (2p) (Table 2).** Following the general procedure, using 1,4,5-triphenyl-1H-1,2,3-triazole<sup>17</sup> (**1p**) (0.25 mmol, 74 mg) provided 40 mg (45% yield) of **2p** as a white solid along with 36.2 mg (35% yield) of **2p'** as white solid. **2p**: Mp 137-153 °C.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.47-7.10 (m, 14H), 2.11 (s, 3H) ppm.  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ):  $\delta$  169.2, 148.6, 141.7, 136.4, 134.8, 131.1, 129.6, 129.3, 129.1, 129.0, 128.8, 126.9, 125.8, 125.1, 123.7, 123.1, 20.9 ppm. IR (neat,  $\text{cm}^{-1}$ ): 1761, 1495, 1365, 1198, 1173, 995, 915, 772, 761, 691. MS ( $\text{ESI}^+$ )  $m/z$  (%) 356 (M+H). HRMS *calcd.* for ( $\text{C}_{22}\text{H}_{18}\text{N}_3\text{O}_2$ ): 356.1399, *found* 356.1400. **2p'**: Mp 61-78 °C.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.49-7.09 (m, 13H), 1.97 (s, 6H) ppm.  $^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ ):  $\delta$  168.5, 150.0, 137.3, 136.8, 136.1, 129.8, 129.5, 129.5, 129.3, 129.1, 129.0, 127.0, 125.4, 120.4, 117.9, 20.9 ppm. IR (neat,  $\text{cm}^{-1}$ ): 1759, 1365, 1179, 1026, 996, 755, 696. MS ( $\text{ESI}^+$ )  $m/z$  (%) 414 (M+H). HRMS *calcd.* for ( $\text{C}_{24}\text{H}_{20}\text{N}_3\text{O}_4$ ): 414.1454, *found* 414.1449.

<sup>17</sup> Shashank, A. B.; Karthik, S.; Madhavachary, R.; Ramachary, D. B. *Chem. Eur. J.* **2014**, 20, 16877.



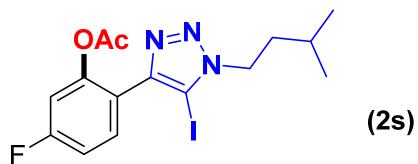
**2-(1-Isopentyl-1*H*-1,2,3-triazol-4-yl)-3-methoxyphenyl acetate (2q) (Table 3).**

Following the general procedure, using triazole **1q** (0.25 mmol, 61 mg) provided 71 mg (93% yield) of **2q** as a white solid. Mp 99-100 °C.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.90 (s, 1H), 7.32 (q,  $J = 7.4, 6.6$  Hz, 1H), 6.86 (dd,  $J = 29.7, 8.3$  Hz, 2H), 4.41 (t,  $J = 7.7$  Hz, 2H), 3.88 (s, 3H), 2.31 (s, 3H), 1.85 (q,  $J = 7.3$  Hz, 2H), 1.64 (dt,  $J = 13.4, 6.7$  Hz, 1H), 0.99 (d,  $J = 6.6$  Hz, 6H) ppm.  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ):  $\delta$  170.1, 157.3, 149.2, 139.3, 128.9, 124.2, 116.0, 113.4, 108.5, 55.9, 48.5, 38.9, 25.5, 22.1, 21.1 ppm. IR (neat,  $\text{cm}^{-1}$ ): 1756, 1261, 1074, 737. MS (ESI $^+$ )  $m/z$  (%) 304 (M+H). HRMS *calcd.* for ( $\text{C}_{16}\text{H}_{22}\text{N}_3\text{O}_3$ ): 304.1661, *found* 304.1662.



**2-(1-Isopentyl-1*H*-1,2,3-triazol-4-yl)-3-(trifluoromethyl)phenyl acetate (2r) (Table 3).**

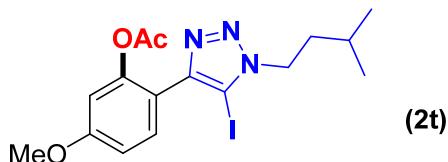
Following the general procedure, using triazole **1r** (0.25 mmol, 71 mg) provided 66 mg (77% yield) of **2r** as a white solid. Mp 60-62 °C.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.66 (d,  $J = 7.9$  Hz, 1H), 7.55 (d,  $J = 7.9$  Hz, 2H), 7.35 (d,  $J = 8.2$  Hz, 1H), 4.44 (t,  $J = 7.3$  Hz, 2H), 2.03 (s, 3H), 1.82 (q,  $J = 7.2$  Hz, 2H), 1.55 (dt,  $J = 13.4, 6.7$  Hz, 1H), 0.95 (d,  $J = 6.6$  Hz, 6H) ppm.  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ):  $\delta$  169.0, 150.4, 138.9, 131.0 (q,  $J = 30$  Hz), 129.8, 126.7, 124.6, 123.7 (q,  $J = 6$  Hz), 121.9, 48.7, 38.9, 25.3, 22.1, 20.4 ppm. IR (neat,  $\text{cm}^{-1}$ ): 1769, 1319, 1190, 1007, 807. MS (ESI $^+$ )  $m/z$  (%) 342 (M+H). HRMS *calcd.* for ( $\text{C}_{16}\text{H}_{19}\text{N}_3\text{O}_2\text{F}_3$ ): 342.1429, *found* 342.1433.



**5-Fluoro-2-(5-iodo-1-isopentyl-1*H*-1,2,3-triazol-4-yl)phenyl acetate (2s) (Table 3).**

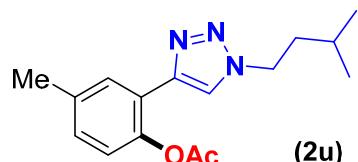
Following the general procedure, using triazole **1s** (0.25 mmol, 90 mg) provided 73 mg (71% yield) of **2s** as a brown solid. Mp 62-63 °C.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.55 (dd,  $J = 8.6, 6.1$  Hz, 1H), 7.14-6.89 (m, 2H), 4.45 (t,  $J = 7.7$  Hz, 2H), 2.17 (s, 3H), 1.84 (q,  $J = 7.4$  Hz, 2H), 1.67 (dt,  $J = 13.5, 7.2$  Hz, 1H), 1.01 (d,  $J = 6.6$  Hz, 6H) ppm.  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ):  $\delta$  168.6, 162.8 (d,  $J = 249$  Hz), 149.4 (d,  $J = 11$  Hz), 147.1,

132.1 (d,  $J = 10$  Hz), 119.6 (d,  $J = 3$  Hz), 113.1 (d,  $J = 21$  Hz), 111.1 (d,  $J = 25$  Hz), 79.2, 49.6, 38.5, 25.6, 22.2, 21.0 ppm. IR (neat,  $\text{cm}^{-1}$ ): 1763, 1459, 1200, 833. MS ( $\text{ESI}^+$ )  $m/z$  (%) 418 (M+H). HRMS *calcd.* for ( $\text{C}_{15}\text{H}_{18}\text{N}_3\text{O}_2\text{FI}$ ): 418.0428, *found* 418.0441.



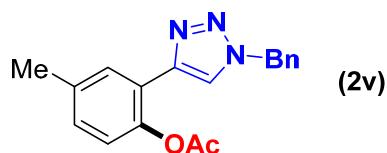
**2-(5-Iodo-1-isopentyl-1*H*-1,2,3-triazol-4-yl)-5-methoxyphenyl acetate (2t) (Table 3).**

Following the general procedure, using triazole **1t** (0.25 mmol, 93 mg) provided 77 mg (72% yield) of **2t** as a white solid. Mp 86–87 °C. <sup>1</sup>H NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.48 (d,  $J = 8.6$  Hz, 1H), 6.87 (dd,  $J = 8.7, 2.6$  Hz, 1H), 6.77 (d,  $J = 2.6$  Hz, 1H), 4.43 (t,  $J = 7.8$  Hz, 2H), 3.83 (s, 3H), 2.15 (s, 3H), 1.83 (q,  $J = 7.3$  Hz, 2H), 1.66 (dt,  $J = 13.4, 6.7$  Hz, 1H), 1.00 (d,  $J = 6.6$  Hz, 6H) ppm. <sup>13</sup>C NMR (101 MHz,  $\text{CDCl}_3$ ):  $\delta$  169.0, 160.8, 149.5, 147.6, 131.6, 115.7, 111.8, 108.8, 78.9, 55.5, 49.5, 38.5, 25.6, 22.2, 21.0 ppm. IR (neat,  $\text{cm}^{-1}$ ): 1764, 1622, 1187, 1090, 840. MS ( $\text{ESI}^+$ )  $m/z$  (%) 430 (M+H). HRMS *calcd.* for ( $\text{C}_{16}\text{H}_{21}\text{N}_3\text{O}_3\text{I}$ ): 430.0628, *found* 430.0632.



**2-(1-Isopentyl-1*H*-1,2,3-triazol-4-yl)-4-methylphenyl acetate (2u) (Table 3).**

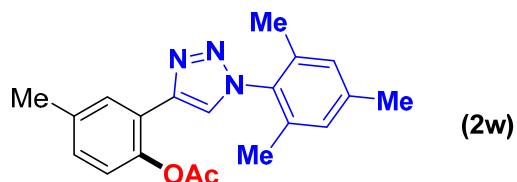
Following the general procedure, using triazole **1u** (0.25 mmol, 57 mg) provided 59 mg (82% yield) of **2u** as a white solid. Mp 67–69 °C. <sup>1</sup>H NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.86 (s, 1H), 7.71 (s, 1H), 7.09 (dd,  $J = 45.9, 8.3$  Hz, 2H), 4.40 (t,  $J = 7.5$  Hz, 2H), 2.38 (s, 3H), 2.31 (s, 3H), 1.82 (q,  $J = 7.3$  Hz, 2H), 1.60 (dt,  $J = 13.3, 6.5$  Hz, 1H), 0.97 (d,  $J = 6.6$  Hz, 6H) ppm. <sup>13</sup>C NMR (101 MHz,  $\text{CDCl}_3$ ):  $\delta$  169.2, 144.9, 143.1, 136.1, 129.5, 129.0, 122.9, 122.7, 121.3, 48.6, 39.0, 25.4, 22.1, 21.2, 20.8 ppm. IR (neat,  $\text{cm}^{-1}$ ): 1769, 1430, 1214, 948. MS ( $\text{ESI}^+$ )  $m/z$  (%) 288 (M+H). HRMS *calcd.* for ( $\text{C}_{16}\text{H}_{22}\text{N}_3\text{O}_2$ ): 288.1712, *found* 288.1711.



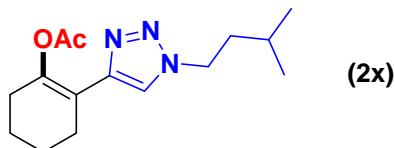
**2-(1-Benzyl-1*H*-1,2,3-triazol-4-yl)-3-methylphenyl acetate (2v) (Table 3).** Following the general procedure, using 1-benzyl-4-(*m*-tolyl)-1*H*-1,2,3-triazole<sup>18</sup> (**1v**) (0.25 mmol, 62

<sup>18</sup> Yamaguchi, K.; Oishi, T.; Katayama, T.; Mizuno, N. *Chem. Eur. J.* **2009**, 15, 10464.

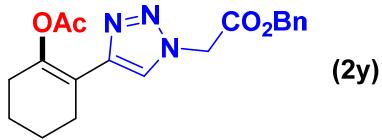
mg) provided 38.4 mg (50% yield) of **2v** as a yellowish solid. Mp 119-122 °C.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.90-7.84 (m, 1H), 7.59 (s, 1H), 7.38 (dt,  $J$  = 4.7, 1.7 Hz, 3H), 7.30 (dd,  $J$  = 7.4, 2.2 Hz, 2H), 7.17-7.10 (m, 1H), 7.00 (d,  $J$  = 8.2 Hz, 1H), 5.56 (s, 2H), 2.37 (s, 3H), 2.11 (s, 3H) ppm.  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ):  $\delta$  169.2, 145.0, 143.6, 136.2, 134.7, 129.8, 129.3, 129.1, 129.0, 128.3, 123.0, 122.8, 121.7, 54.3, 21.2, 21.0 ppm. IR (neat,  $\text{cm}^{-1}$ ): 1755, 1496, 1371, 1182, 1069, 822, 715. MS (ESI $^+$ )  $m/z$  (%) 308 (M+H). HRMS *calcd.* for ( $\text{C}_{18}\text{H}_{18}\text{N}_3\text{O}_2$ ): 308.1399, *found* 308.1397.



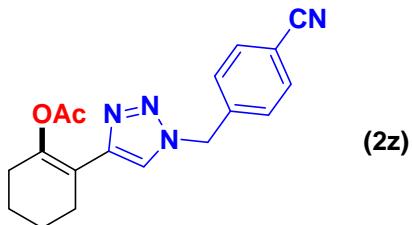
**2-(1-Mesityl-1*H*-1,2,3-triazol-4-yl)-3-methylphenyl acetate (2w) (Table 2).** Following the general procedure, using triazole **1w** (0.25 mmol, 69 mg) provided 60.4 mg (72% yield) of **2w** as a yellow solid. Mp 107-109 °C.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.03 (d,  $J$  = 1.5 Hz, 1H), 7.82 (s, 1H), 7.19 (dd,  $J$  = 8.4, 2.1 Hz, 1H), 7.08 (d,  $J$  = 8.2 Hz, 1H), 7.01 (s, 2H), 2.42 (s, 3H), 2.36 (s, 3H), 2.29 (s, 3H), 2.00 (s, 6H) ppm.  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ):  $\delta$  169.2, 145.0, 143.6, 136.2, 134.7, 129.8, 129.3, 129.1, 129.0, 128.3, 123.0, 122.8, 121.7, 54.3, 21.2, 21.0 ppm. IR (neat,  $\text{cm}^{-1}$ ): 1760, 1494, 1367, 1183, 1039, 908, 730. MS (ESI $^+$ )  $m/z$  (%) 336 (M+H). HRMS *calcd.* for ( $\text{C}_{20}\text{H}_{22}\text{N}_3\text{O}_2$ ): 336.1712, *found* 336.1701.



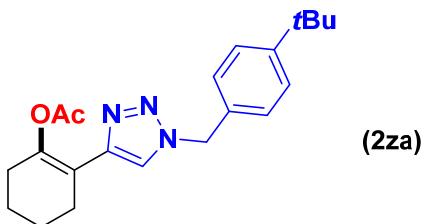
**2-(1-Isopentyl-1*H*-1,2,3-triazol-4-yl)cyclohex-1-en-1-yl acetate (2x) (Table 3).** Following the general procedure, using triazole **1x** (0.25 mmol, 55 mg) at 110 °C provided 51.9 mg (75% yield) of **2x** as a yellow solid. Mp 73-81 °C.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.45 (s, 1H), 4.28 (t,  $J$  = 7.4 Hz, 2H), 2.64-2.53 (m, 2H), 2.26 (t,  $J$  = 6.0 Hz, 2H), 2.15 (s, 3H), 1.70 (q,  $J$  = 7.1 Hz, 6H), 1.49 (dt,  $J$  = 13.4, 6.7 Hz, 1H), 0.88 (d,  $J$  = 6.6 Hz, 6H) ppm.  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ):  $\delta$  168.6, 144.9, 144.2, 120.9, 115.1, 48.3, 38.9, 27.8, 26.2, 25.3, 22.4, 22.0, 21.9, 21.2 ppm. IR (neat,  $\text{cm}^{-1}$ ): 2938, 2846, 1748, 1371, 1215, 1198, 1150, 1103, 1058. MS (ESI $^+$ )  $m/z$  (%) 278 (M+H). HRMS *calcd.* for ( $\text{C}_{15}\text{H}_{24}\text{N}_3\text{O}_2$ ): 278.1869, *found* 278.1860.



**Benzyl 2-[4-(2-acetoxycyclohex-1-en-1-yl)-1*H*-1,2,3-triazol-1-yl]acetate (2y) (Table 3).** Following the general procedure, using triazole **1y** (0.25 mmol, 74 mg) provided 65.7 mg (74% yield) of **2y** as a yellow solid. Mp 59-66 °C.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.67 (s, 1H), 7.37-7.23 (m, 5H), 5.13 (d,  $J = 8.0$  Hz, 4H), 2.64-2.55 (m, 2H), 2.32-2.24 (m, 2H), 2.11 (s, 3H), 1.79-1.65 (m, 4H) ppm.  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ):  $\delta$  168.7, 166.2, 145.5, 144.7, 134.5, 128.7, 128.6, 128.4, 122.8, 115.0, 67.7, 50.6, 27.8, 26.2, 22.4, 21.9, 21.1 ppm. IR (neat,  $\text{cm}^{-1}$ ): 2937, 1751, 1457, 1188, 1104, 1056, 749, 698. MS (ESI $^+$ )  $m/z$  (%) 356 (M+H). HRMS *calcd.* for ( $\text{C}_{19}\text{H}_{22}\text{N}_3\text{O}_4$ ): 356.1610, *found* 356.1601.



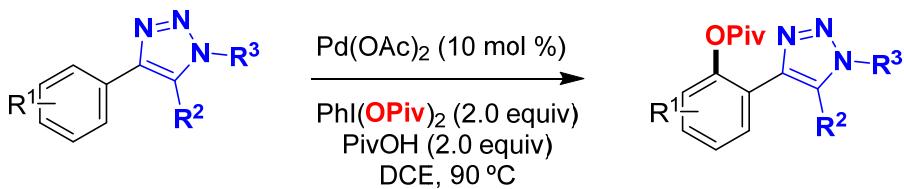
**2-[1-(4-Cyanobenzyl)-1*H*-1,2,3-triazol-4-yl]cyclohex-1-en-1-yl acetate (2z) (Table 3).** Following the general procedure, using triazole **1z** (0.25 mmol, 66 mg) provided 58 mg (72% yield) of **2z** as a white solid. Mp 111-117 °C.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.64 (d,  $J = 8.2$  Hz, 2H), 7.44 (s, 1H), 7.30 (d,  $J = 8.3$  Hz, 2H), 5.57 (s, 2H), 2.64-2.56 (m, 2H), 2.32-2.24 (m, 2H), 2.12 (s, 3H), 1.81-1.69 (m, 4H) ppm.  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ):  $\delta$  168.7, 145.9, 145.5, 140.2, 132.9, 128.3, 121.3, 118.2, 115.0, 53.3, 48.6, 28.0, 26.5, 22.6, 22.0, 21.4 ppm. IR (neat,  $\text{cm}^{-1}$ ): 2941, 2226, 1738, 1374, 1220, 1097, 765. MS (ESI $^+$ )  $m/z$  (%) 323 (M+H). HRMS *calcd.* for ( $\text{C}_{18}\text{H}_{19}\text{N}_4\text{O}_2$ ): 323.1508, *found* 323.1502.



**2-{1-[4-(*Tert*-butyl)benzyl]-1*H*-1,2,3-triazol-4-yl}cyclohex-1-en-1-yl acetate (2za) (Table 3).** Following the general procedure, using triazole **1za** (0.25 mmol, 74 mg) provided 66 mg (75% yield) of **2za** as a white solid. Mp 134-135 °C.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.39 (d,  $J = 7.9$  Hz, 2H), 7.32 (s, 1H), 7.20 (d,  $J = 7.9$  Hz, 2H), 5.47 (s, 2H), 2.64 (dd,  $J = 5.9, 3.3$  Hz, 2H), 2.28 (d,  $J = 6.0$  Hz, 2H), 2.01 (s, 3H), 1.88-1.50 (m, 4H),

1.30 (s, 9H) ppm.  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ):  $\delta$  168.6, 151.9, 145.2, 144.7, 131.6, 128.1, 128.0, 126.1, 126.0, 121.0, 115.3, 53.7, 34.6, 31.2, 27.8, 26.2, 22.5, 22.0, 21.1 ppm. IR (neat,  $\text{cm}^{-1}$ ): 1750, 1213, 1191. MS (ESI $^+$ )  $m/z$  (%) 354 (M+H). HRMS *calcd.* for ( $\text{C}_{21}\text{H}_{28}\text{N}_3\text{O}_2$ ): 354.2182, *found* 354.2180.

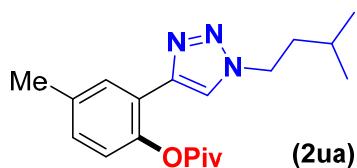
#### 4.-Pd-Catalyzed C(sp<sup>2</sup>)-H Pivaloxylation (Table 3)



**General Procedure:** A reaction tube containing a stirring bar was charged with the corresponding triazole (0.25 mmol, 1.00 equiv), PhI(OPiv)<sub>2</sub> (0.50 mmol, 2.00 equiv), PivOH (0.50 mmol, 2.00 equiv) and Pd(OAc)<sub>2</sub> (10 mol %). The reaction tube was then evacuated and back-filled with dry argon (this sequence was repeated up to three times). Then 1,2-dichloroethane (1.00 mL) was added under argon atmosphere. The reaction tube was next warmed up to 90 °C and stirred for 24 hours. The mixture was then allowed to warm to room temperature, filtered off through a pad of celite and washed with CH<sub>2</sub>Cl<sub>2</sub>. The resulting mixture was concentrated under reduced pressure and the corresponding product was purified by flash chromatography (hexanes/AcOEt 7/3). The yields reported in the manuscript refer to isolated yields and represent an average of at least two independent runs.

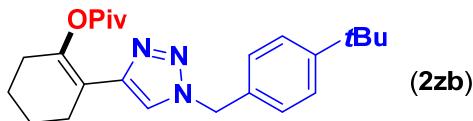


**2-(5-Iodo-1-isopentyl-1H-1,2,3-triazol-4-yl)phenyl pivalate (2na) (Table 3).** Following the general procedure, using triazole **1n** (0.25 mmol, 90 mg) provided 78 mg (68% yield) of **2na** as a white solid. Mp 105-107 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):  $\delta$  7.55-7.43 (m, 3H), 7.42-7.26 (m, 5H), 7.19 (d,  $J$  = 8.1 Hz, 1H), 5.67 (s, 2H), 1.12 (s, 9H) ppm. <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>):  $\delta$  176.3, 149.1, 148.5, 134.2, 131.3, 130.1, 128.8, 128.4, 127.9, 125.6, 123.6, 122.9, 80.1, 54.4, 38.8, 26.9 ppm. IR (neat, cm<sup>-1</sup>): 1747, 1730, 1093, 731. MS (ESI<sup>+</sup>)  $m/z$  (%) 462 (M+H). HRMS *calcd.* for (C<sub>20</sub>H<sub>21</sub>N<sub>3</sub>O<sub>2</sub>I): 462.0678, *found* 462.0688.



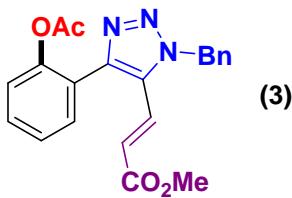
**2-(5-Iodo-1-isopentyl-1H-1,2,3-triazol-4-yl)phenyl pivalate (2ua) (Table 3).** Following the general procedure, using triazole **1u** (0.25 mmol, 57 mg) provided 58 mg (70% yield)

of **2ua** as a white solid. Mp 78-80 °C.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.81 (s, 1H), 7.68 (s, 1H), 7.15 (d,  $J$  = 7.9 Hz, 1H), 6.93 (d,  $J$  = 8.3 Hz, 1H), 4.40 (t,  $J$  = 7.5 Hz, 2H), 2.39 (s, 3H), 1.81 (q,  $J$  = 7.3 Hz, 2H), 1.60 (d,  $J$  = 5.3 Hz, 1H), 1.36 (s, 9H), 0.97 (d,  $J$  = 6.6 Hz, 6H) ppm.  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ):  $\delta$  176.9, 145.7, 143.2, 135.8, 129.7, 129.6, 123.3, 122.4, 121.6, 48.6, 39.1, 27.3, 25.4, 22.2, 20.8 ppm. IR (neat,  $\text{cm}^{-1}$ ): 1742, 1498, 1110, 790. MS (ESI $^+$ )  $m/z$  (%) 330 (M+H). HRMS *calcd.* for ( $\text{C}_{19}\text{H}_{28}\text{N}_3\text{O}_2$ ): 330.2182, *found* 330.2193.

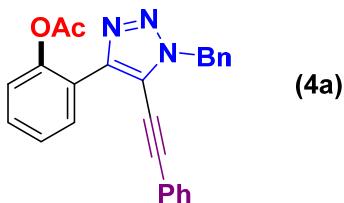


**2-(5-Iodo-1-isopentyl-1*H*-1,2,3-triazol-4-yl)phenyl pivalate (2zb) (Table 3).** Following the general procedure, using triazole **1z** (0.25 mmol, 74 mg) provided 76 mg (77% yield) of **2zb** as a white solid. Mp 88-89 °C.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.37 (d,  $J$  = 8.0 Hz, 2H), 7.33 (s, 1H), 7.19 (d,  $J$  = 8.0 Hz, 2H), 5.43 (s, 2H), 2.67 (d,  $J$  = 5.2 Hz, 2H), 2.19 (t,  $J$  = 5.6 Hz, 2H), 1.74 (dt,  $J$  = 8.1, 5.3 Hz, 4H), 1.29 (s, 9H), 1.05 (s, 9H) ppm.  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ):  $\delta$  175.9, 151.8, 145.2, 144.3, 131.2, 128.2, 126.0, 121.2, 115.4, 53.8, 38.6, 34.5, 31.2, 27.3, 26.9, 26.7, 26.4, 22.5, 22.0 ppm. IR (neat,  $\text{cm}^{-1}$ ): 1739, 1115, 715. MS (ESI $^+$ )  $m/z$  (%) 396 (M+H). HRMS *calcd.* for ( $\text{C}_{24}\text{H}_{34}\text{N}_3\text{O}_2$ ): 396.2651, *found* 396.2666.

## 5.-Further Transformations (Scheme 2)

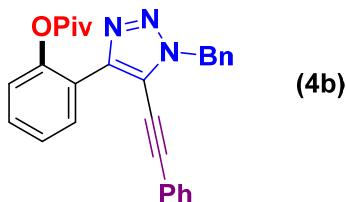


**Methyl (*E*)-3-(4-(2-acetoxyphenyl)-1-benzyl-1*H*-1,2,3-triazol-5-yl)acrylate (3).** A reaction tube containing a stirring bar was charged with triazole **2n** (0.17 mmol, 70 mg), Pd(OAc)<sub>2</sub> (0.017 mmol, 1.9 mg), TBAB (0.013 mmol, 4.2 mg) and NaHCO<sub>3</sub> (0.42 mmol, 35 mg). The reaction tube was then evacuated and back-filled with dry argon (this sequence was repeated up to three times). Methyl acrylate (0.42 mmol, 38 µL), and DMF (2.0 mL) were then added under argon atmosphere. The reaction tube was next warmed up to 80 °C and stirred 12 hours. The resulting mixture was concentrated under reduced pressure and the corresponding product was purified by flash chromatography (hexanes/AcOEt 7/3) to provide 44 mg (69% yield) of **3** as an orange oil. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 7.63-7.51 (m, 3H), 7.47-7.39 (m, 4H), 7.37-7.22 (m, 3H), 6.14 (d, *J* = 16.1 Hz, 1H), 5.77 (s, 2H), 3.78 (s, 3H), 2.08 (s, 3H) ppm. <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>): δ 168.7, 166.1, 148.4, 143.9, 134.4, 131.1, 130.4, 129.5, 129.2, 128.6, 126.9, 126.8, 126.3, 123.6, 123.4, 123.2, 52.8, 52.0, 20.7 ppm. IR (neat, cm<sup>-1</sup>): 1715, 1644, 1176, 908, 726. MS (ESI<sup>+</sup>) *m/z* (%) 378 (M+H). HRMS *calcd.* for (C<sub>21</sub>H<sub>20</sub>N<sub>3</sub>O<sub>4</sub>): 378.1454, *found* 378.1467.



**2-(1-Benzyl-5-(phenylethynyl)-1*H*-1,2,3-triazol-4-yl)phenyl acetate (4a).** A reaction tube containing a stirring bar was charged with triazole **2n** (0.12 mmol, 50 mg), PdCl<sub>2</sub>(PPh<sub>3</sub>)<sub>2</sub> (0.012 mmol, 8.20 mg), and K<sub>2</sub>CO<sub>3</sub> (0.18 mmol, 25 mg). The reaction tube was then evacuated and back-filled with dry argon (this sequence was repeated up to three times). Phenylacetylene (0.18 mmol, 20 µL), and THF (1.0 mL) were then added under argon atmosphere. The reaction tube was next warmed up to 80 °C and stirred 12 hours. The resulting mixture was concentrated under reduced pressure and the corresponding product was purified by flash chromatography (hexanes/AcOEt 8/2) to provide 39 mg (83% yield) of **4a** as an orange oil. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 8.00 (d, *J* = 7.7 Hz, 1H), 7.57-7.34 (m, 11H), 7.34-7.23 (m, 2H), 5.75 (s, 2H), 2.28 (s, 3H) ppm. <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>): δ 169.7, 148.0, 145.34, 134.6, 131.5, 130.0, 130.1, 129.7, 128.9,

128.6, 128.5, 128.0, 125.9, 123.4, 123.0, 121.2, 119.1, 102.2, 75.0, 53.1, 21.2 ppm. IR (neat,  $\text{cm}^{-1}$ ): 1763, 1190, 729, 689. MS (ESI $^+$ )  $m/z$  (%) 394 (M+H). HRMS *calcd.* for (C<sub>25</sub>H<sub>20</sub>N<sub>3</sub>O<sub>2</sub>): 394.1556, *found* 394.1566.



**2-(1-Benzyl-5-(phenylethynyl)-1H-1,2,3-triazol-4-yl)phenyl pivalate (4b).** Following the procedure for the synthesis of **6a**, using triazole **2na** (0.13 mmol, 60 mg) provided 40 mg (71% yield) of **4b** as an orange oil. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):  $\delta$  7.80 (d,  $J$  = 7.4 Hz, 2H), 7.44-7.31 (m, 11H), 7.16 (d,  $J$  = 8.0 Hz, 2H), 5.68 (s, 2H), 1.19 (s, 9H) ppm. <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>):  $\delta$  176.8, 148.8, 145.7, 134.6, 131.6, 130.4, 129.8, 129.6, 128.8, 128.5, 128.4, 128.1, 125.6, 123.2, 123.1, 121.3, 119.4, 101.9, 74.9, 53.1, 39.1, 27.1 ppm. IR (neat,  $\text{cm}^{-1}$ ): 1746, 1199, 1106, 756, 729. MS (ESI $^+$ )  $m/z$  (%) 436 (M+H). HRMS *calcd.* for (C<sub>28</sub>H<sub>26</sub>N<sub>3</sub>O<sub>2</sub>): 436.2025, *found* 436.2022.



**2-(1-Benzyl-1H-1,2,3-triazol-4-yl)phenol (5).** A reaction tube containing a stirring bar was charged with triazole **2a** (0.17 mmol, 50 mg) and dissolved in MeOH (4 mL) at 0 °C. Then Cs<sub>2</sub>CO<sub>3</sub> (0.17 mmol, 55 mg) was added and the resulting solution was stirred at room temperature for 5 hours under argon atmosphere. The solvent was partially evaporated under reduced pressure and the resulting solution was washed with aq. NH<sub>4</sub>Cl, extracted with CH<sub>2</sub>Cl<sub>2</sub> and washed with brine. The combined organic layers were dried over MgSO<sub>4</sub>, and concentrated under reduced pressure. The crude residue was purified by flash chromatography (hexanes/AcOEt 9/1) to provide 40 mg (94% yield) of **5** as a white solid. Mp 142-144 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):  $\delta$  7.73 (s, 1H), 7.46-7.27 (m, 6H), 7.21 (t,  $J$  = 8 Hz, 1H), 7.04 (d,  $J$  = 8.2 Hz, 1H), 6.85 (t,  $J$  = 8.0 Hz, 1H), 5.58 (s, 2H) ppm. <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>):  $\delta$  155.8, 148.0, 134.0, 129.7, 129.2, 129.0, 128.10, 125.8, 119.4, 118.8, 117.6, 113.8, 54.5 ppm. IR (neat,  $\text{cm}^{-1}$ ): 3200, 1210, 690. MS (ESI $^+$ )  $m/z$  (%) 252 (M+H). HRMS *calcd.* for (C<sub>15</sub>H<sub>14</sub>N<sub>3</sub>O): 252.1137, *found* 252.1149.

## 6.-<sup>1</sup>H NMR and <sup>13</sup>C NMR Spectra

