# Direct C-H Arylation and Alkenylation of 1-Substituted Tetrazoles: Phosphine As Stabilizing Factor

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#### Experimental

#### **General Experimental Procedures**

All substances were used as received. All anhydrous reactions were performed in flamedried Schlenk tubes under Ar atmosphere. <sup>1</sup>H and <sup>13</sup>C NMR spectra were recorded for CDCl<sub>3</sub> solutions at ambient temperature on a spectrometer operating at 300 MHz for <sup>1</sup>H and 75 MHz for <sup>13</sup>C. Chemical shifts were recorded as  $\delta$  values in parts per million (ppm), and were indirectly referenced to tetramethylsilane (TMS) *via* the solvent signal. Coupling constants (*J*) are given in Hz.

## Characterization of compounds 1a-3f

5-(4-Methylphenyl)-1-phenyl-1*H*-tetrazole (**1a**):<sup>1</sup> Yield: 69 %. Colourless crystals, mp 136-137 °C; <sup>1</sup>H NMR: (300 MHz, CDCl<sub>3</sub>)  $\delta$  7.58-7.48 (3H, m), 7.48-7.36 (4H, m), 7.22-7.16 (2H, m), 2.36 (3H, s); <sup>13</sup>C NMR: (75 MHz, CDCl<sub>3</sub>)  $\delta$  154.1, 142.2, 135.1, 130.7, 130.2, 130.1, 129.2, 125.7, 121.1, 21.9; IR: (ATR) v<sub>max</sub> 1073, 1104, 1182, 1278, 1294, 1450, 1471, 1497, 1592, 1614, 2859, 2922, 2957, 3060 cm<sup>-1</sup>; LRMS: *m/z* (relative intensity) 237.1 [M+H]<sup>+</sup> (100), 209.3 (11), 194.2 (5), 182.2 (1), 175.1 (1), 153.2 (1); Anal. Calcd for C<sub>14</sub>H<sub>12</sub>N<sub>4</sub>: C, 71.17; H, 5.12; N, 23.71. Found: C, 71.18; H, 5.01; N, 23.64.

1-Phenyl-5-(thiophen-2-yl)-1*H*-tetrazole (**1b**): Yield: 49 %. Yellowish crystals, mp 87-88 °C; <sup>1</sup>H NMR: (300 MHz, CDCl<sub>3</sub>)  $\delta$  7.67-7.56 (3H, m), 7.52-7.44 (3H, m), 7.26-7.22 (1H, m), 7.06-7.01 (1H, m); <sup>13</sup>C NMR: (75 MHz, CDCl<sub>3</sub>)  $\delta$  149.8, 134.0, 131.2, 130.6, 130.5, 130.0, 128.0, 126.4, 124.2; IR: (ATR)  $\nu_{max}$  1047, 1100, 1126, 1217, 1271, 1407, 1424, 1456, 1481, 1499, 1556, 2851, 2921, 2953, 3105 cm<sup>-1</sup>; LRMS: *m/z* (relative intensity) 229.1 [M+H]<sup>+</sup>

(100), 201.2 (3), 186.2 (3), 174.3 (2), 149.1 (2); Anal. Calcd for C<sub>11</sub>H<sub>8</sub>N<sub>4</sub>S: C, 57.88; H, 3.53; N, 24.54; S, 14.05. Found: C, 57.89; H, 3.51; N, 24.62; S, 14.12.

1,5-Diphenyl-1*H*-tetrazole (**1c**):<sup>2</sup> Yield: 85 %. Colourless crystals, mp 144-145 °C; <sup>1</sup>H NMR: (300 MHz, CDCl<sub>3</sub>)  $\delta$  7.51-7.39 (6H, m), 7.36-7.29 (4H, m); <sup>13</sup>C NMR: (75 MHz, CDCl<sub>3</sub>)  $\delta$  153.4, 134.2, 131.1, 130.2, 129.7, 128.8, 128.6, 125.0, 123.3; IR: (ATR) v<sub>max</sub> 1000, 1071, 1143, 1268, 1278, 1411, 1449, 1468, 1497, 1594, 2852, 2922, 3067 cm<sup>-1</sup>; LRMS: *m/z* (relative intensity) 223.2 [M+H]<sup>+</sup> (100), 217.1 (5), 195.2 (22), 180.1 (4), 173.2 (2), 119.1 (2); Anal. Calcd for C<sub>13</sub>H<sub>10</sub>N<sub>4</sub>: C, 70.26; H, 4.54; N, 25.21. Found: C, 70.05; H, 4.43; N, 24.99.

5-(4-Methoxyphenyl)-1-phenyl-1*H*-tetrazole (**1d**):<sup>3</sup> Yield: 76 %. White crystals, mp 106-108 °C; <sup>1</sup>H NMR: (300 MHz, CDCl<sub>3</sub>)  $\delta$  7.53-7.40 (5H, m), 7.39-7.36 (2H, m), 6.88-6.84 (2H, m), 3.78 (3H, s); <sup>13</sup>C NMR: (75 MHz, CDCl<sub>3</sub>)  $\delta$  161.6, 153.2, 134.5, 130.3, 130.2, 129.7, 125.2, 115.3, 114.3, 55.2; IR: (ATR) v<sub>max</sub> 1018, 1075, 1102, 1184, 1257, 1307, 1400, 1440, 1453, 1468, 1474, 1578, 1610, 2841, 2923, 2968, 3027 cm<sup>-1</sup>; LRMS: *m/z* (relative intensity) 253.1 [M+H]<sup>+</sup> (100), 233.0 (4), 225.1 (18), 210.2 (9), 183.3 (3), 157.3 (2), 122.2 (2); Anal. Calcd for C<sub>14</sub>H<sub>12</sub>N<sub>4</sub>O: C, 66.65; H, 4.79; N, 22.21. Found: C, 66.89; H, 4.94; N, 22.02.

5-(4-Chlorophenyl)-1-phenyl-1*H*-tetrazole (**1e**):<sup>4</sup> Yield: 87 %. Colourless crystals, mp 157-158 °C; <sup>1</sup>H NMR: (300 MHz, CDCl<sub>3</sub>)  $\delta$  7.50-7.39 (5H, m), 7.34-7.25 (4H, m); <sup>13</sup>C NMR: (75 MHz, CDCl<sub>3</sub>)  $\delta$  152.5, 137.4, 134.0, 130.4, 130.0, 129.8, 129.1, 125.1, 121.8; IR: (ATR)  $\nu_{max}$  997, 1093, 1104, 1134, 1263, 1297, 1431, 1449, 1463, 1495, 1591, 1602, 2852, 2922, 2955, 3068 cm<sup>-1</sup>; LRMS: *m/z* (relative intensity) 257.1 [M+H]<sup>+</sup> (100), 249.0 (6), 229.4 (8), 216.1 (5), 195.3 (5), 161.2 (2), 119.1 (5); Anal. Calcd for C<sub>13</sub>H<sub>9</sub>ClN<sub>4</sub>: C, 60.83; H, 3.53; N, 21.83. Found: C, 60.98; H, 3.62; N, 22.00.

(Z)-5-[1,4-Bis(tetrahydropyran-2-yloxy)but-2-en-2-yl]-1-phenyl-1*H*-tetrazole (**1f**): Yield: 56 %. Yellow oil, mixture of diastereomers; <sup>1</sup>H NMR: (300 MHz, CDCl<sub>3</sub>) δ 7.49-7.43 (5H, m), 6.14-6.05 (1H, m), 4.54-4.18 (6H, m), 3.72-3.62 (1H, m), 3.58-3.45 (1H, m), 3.40-3.26

(2H, m), 1.73-1.22 (12H, m); <sup>13</sup>C NMR: (75 MHz, CDCl<sub>3</sub>)  $\delta$  153.4, 138.7, 138.7, 134.3, 129.9, 129.4, 124.7, 124.0, 124.0, 98.1, 97.9, 97.8, 97.7, 62.8, 62.7, 62.4, 62.4, 61.9, 61.7, 30.1, 29.9, 25.0, 24.9, 18.9, 18.7, 18.7; IR: (ATR)  $v_{max}$  1021, 1059, 1075, 1117, 1200, 1261, 1352, 1388, 1419, 1440, 1453, 1492, 1596, 2851, 2866, 2939 cm<sup>-1</sup>; LRMS: *m/z* (relative intensity) 423.3 [M+Na]<sup>+</sup> (100), 395.2 (5), 317.3 (16), 292.0 (2), 269.9 (1), 233.2 (13), 190.2 (3), 159.1 (2); Anal. Calcd for C<sub>21</sub>H<sub>28</sub>N<sub>4</sub>O<sub>4</sub>: C, 62.98; H, 7.05; N, 13.99. Found: C, 62.74; H, 7.00; N, 13.91.

1-(3,4-Dichlorophenyl)-5-(4-methylphenyl)-1*H*-tetrazole (**2a**): Yield: 73 %. White crystals, mp 146-148 °C; <sup>1</sup>H NMR: (300 MHz, CDCl<sub>3</sub>)  $\delta$  7.60 (1H, d, *J* = 2.5 Hz), 7.58 (1H, d, *J* = 8.5 Hz), 7.46-7.40 (2H, m), 7.26-7.23 (2H, m), 7.22 (1H, dd, *J*<sub>1</sub> = 8.5 Hz, *J*<sub>2</sub> = 2.5 Hz, overlapped), 2.40 (3H, s); <sup>13</sup>C NMR: (75 MHz, CDCl<sub>3</sub>)  $\delta$  153.7, 142.3, 134.9, 134.1, 133.6, 131.5, 129.9, 128.8, 127.1, 124.3, 120.0, 21.5; IR: (ATR) v<sub>max</sub> 1006, 1035, 1099, 1122, 1182, 1242, 1282, 1380, 1465, 1485, 1571, 1590, 1610, 3094 cm<sup>-1</sup>; LRMS: *m*/*z* (relative intensity) 305.1 [M+H]<sup>+</sup> (100), 277.2 (22), 262.1 (15), 242.1 (14), 233.3 (16), 177.8 (4), 157.0 (4); Anal. Calcd for C<sub>14</sub>H<sub>10</sub>Cl<sub>2</sub>N<sub>4</sub>: C, 55.10; H, 3.30; N, 18.36. Found: C, 55.16; H, 3.21; N, 18.24.

1-(3,4-Dichlorophenyl)-5-(thiophen-2-yl)-1*H*-tetrazole (**2b**): Yield: 57 %. Brownish crystals, mp 113-114 °C; <sup>1</sup>H NMR: (300 MHz, CDCl<sub>3</sub>)  $\delta$  7.69 (1H, d, *J* = 8.8 Hz), 7.65 (1H, d, *J* = 2.5 Hz), 7.57 (1H, dd, *J*<sub>1</sub> = 5.2 Hz, *J*<sub>2</sub> = 1.1 Hz), 7.36 (1H, dd, *J*<sub>1</sub> = 8.8 Hz, *J*<sub>2</sub> = 2.5 Hz), 7.34 (1H, dd, *J*<sub>1</sub> = 3.4 Hz, *J*<sub>2</sub> = 1.1 Hz), 7.10 (1H, dd, *J*<sub>1</sub> = 5.2 Hz, *J*<sub>2</sub> = 3.4 Hz); <sup>13</sup>C NMR: (75 MHz, CDCl<sub>3</sub>)  $\delta$  149.7, 136.0, 134.3, 133.0, 131.7, 131.1, 130.9, 128.3, 128.2, 125.4, 123.5; IR: (ATR) v<sub>max</sub> 962, 1033, 1103, 1122, 1226, 1267, 1382, 1406, 1419, 1464, 1481, 1558, 1569, 1575, 2851, 2921, 3069, 3089, 3107 cm<sup>-1</sup>; LRMS: *m/z* (relative intensity) 297.0 [M+H]<sup>+</sup> (100), 269.5 (33), 254.3 (66), 234.3 (86), 205.2 (12), 169.3 (7), 150.1 (11); Anal. Calcd for C<sub>11</sub>H<sub>6</sub>Cl<sub>2</sub>N<sub>4</sub>S: C, 44.46; H, 2.04; N, 18.85; S, 10.79. Found: C, 44.26; H, 2.00; N, 18.81; S, 10.62.

1-(3,4-Dichlorophenyl)-5-phenyl-1*H*-tetrazole (**2c**):<sup>5</sup> Yield: 66 %. Colourless crystals, mp 122-124 °C; <sup>1</sup>H NMR: (300 MHz, CDCl<sub>3</sub>)  $\delta$  7.59-7.39 (7H, m), 7.21 (1H, dd,  $J_I = 8.5$  Hz,  $J_2 = 2.5$  Hz); <sup>13</sup>C NMR: (75 MHz, CDCl<sub>3</sub>)  $\delta$  153.5, 134.9, 134.0, 133.4, 131.6, 131.4, 129.1, 128.8, 126.9, 124.2, 122.8; IR: (ATR)  $\nu_{max}$  1010, 1036, 1125, 1244, 1284, 1386, 1408, 1468, 1479, 2852, 2923, 3076, 3091 cm<sup>-1</sup>; LRMS: *m/z* (relative intensity) 291.1 [M+H]<sup>+</sup> (100), 263.1 (47), 250.2 (25), 228.3 (16), 214.2 (6), 160.1 (7); Anal. Calcd for C<sub>13</sub>H<sub>8</sub>Cl<sub>2</sub>N<sub>4</sub>: C, 53.63; H, 2.77; N, 19.24. Found: C, 53.65; H, 2.92; N, 19.29.

1-(3,4-Dichlorophenyl)-5-(4-methoxyphenyl)-1*H*-tetrazole (**2d**): Yield: 79 %. Yellowish crystals, mp 121-122 °C; <sup>1</sup>H NMR: (300 MHz, CDCl<sub>3</sub>)  $\delta$  7.60-7.55 (2H, m), 7.49-7.44 (2H, m), 7.23 (1H, dd,  $J_I = 8.5$  Hz,  $J_2 = 2.5$  Hz), 6.95-6.90 (2H, m), 3.74 (3H, s); <sup>13</sup>C NMR: (75 MHz, CDCl<sub>3</sub>)  $\delta$  162.4, 153.7, 135.2, 134.4, 134.0, 131.8, 130.8, 127.4, 124.6, 115.1, 115.0, 55.7; IR: (ATR) v<sub>max</sub> 1017, 1032, 1099, 1127, 1179, 1253, 1308, 1442, 1461, 1469, 1480, 1578, 1608, 2851, 2923, 2956, 3076, 3096 cm<sup>-1</sup>; LRMS: *m/z* (relative intensity) 321.0 [M+H]<sup>+</sup> (100), 293.2 (51), 278.3 (53), 258.3 (71), 232.7 (9), 190.3 (3), 164.9 (3), 147.3 (2), 132.1 (8); Anal. Calcd for C<sub>14</sub>H<sub>10</sub>Cl<sub>2</sub>N<sub>4</sub>O: C, 52.36; H, 3.14; N, 17.45. Found: C, 52.09; H, 3.01; N, 17.24.

5-(4-Chlorophenyl)-1-(3,4-dichlorophenyl)-1*H*-tetrazole (**2e**): Yield: 53 %. Yellowish crystals, mp 162-164 °C; <sup>1</sup>H NMR: (300 MHz, CDCl<sub>3</sub>) δ 7.63-7.58 (2H, m), 7.53-7.48 (2H, m), 7.46-7.41 (2H, m), 7.21 (1H, dd,  $J_I = 8.8$  Hz,  $J_2 = 2.5$  Hz); <sup>13</sup>C NMR: (75 MHz, CDCl<sub>3</sub>) δ 152.7, 138.2, 135.3, 134.4, 133.3, 131.7, 130.1, 129.7, 127.1, 124.2, 121.4; IR: (ATR) ν<sub>max</sub> 1007, 1035, 1095, 1125, 1145, 1244, 1264, 1393, 1433, 1458, 1474, 1481, 1572, 1601, 2852, 2922, 2954, 3097 cm<sup>-1</sup>; LRMS: *m/z* (relative intensity) 325.0 [M+H]<sup>+</sup> (100), 297.1 (57), 282.3 (67), 262.2 (66), 237.4 (31), 227.1 (25), 206.3 (13), 156.5 (12); Anal. Calcd for C<sub>13</sub>H<sub>7</sub>Cl<sub>3</sub>N<sub>4</sub>: C, 47.96; H, 2.17; N, 17.21. Found: C, 47.75; H, 2.11; N, 17.02.

(*Z*)-5-[1,4-Bis(tetrahydropyran-2-yloxy)but-2-en-2-yl]-1-(3,4-dichlorophenyl)-1*H*-tetrazole (**2f**): Yield: 59 %. Yellow oil, mixture of diastereomers; <sup>1</sup>H NMR: (300 MHz, CDCl<sub>3</sub>)  $\delta$  7.78-7.71 (1H, m), 7.60 (1H, d, *J* = 8.5 Hz), 7.50-7.42 (1H, m), 6.12-6.02 (1H, m), 4.60-4.21 (6H, m), 3.75-3.65 (1H, m), 3.62-3.53 (1H, m), 3.48-3.37 (2H, m), 1.79-1.28 (12H, m); <sup>13</sup>C NMR: (75 MHz, CDCl<sub>3</sub>)  $\delta$  153.6, 139.2, 139.2, 134.6, 133.7, 133.4, 131.2, 126.8, 124.0, 123.9, 123.9, 98.7, 98.5, 98.2, 98.2, 63.3, 63.3, 62.6, 62.6, 62.1, 62.1, 30.3, 30.1, 25.2, 25.0, 19.0; IR: (ATR) v<sub>max</sub> 960, 1022, 1059, 1076, 1118, 1201, 1262, 1352, 1387, 1440, 1474, 1575, 1595, 2851, 2940, 3067 cm<sup>-1</sup>; LRMS: *m/z* (relative intensity) 491.2 [M+Na]<sup>+</sup> (100), 423.1 (2), 403.7 (5), 384.8 (6), 301.0 (20), 283.1 (8), 254.9 (5), 229.1 (1); Anal. Calcd for C<sub>21</sub>H<sub>26</sub>Cl<sub>2</sub>N<sub>4</sub>O<sub>4</sub>: C, 53.74; H, 5.58; N, 11.94. Found: C, 53.84; H, 5.63; N, 11.89.

1-Cyclohexyl-5-(4-methylphenyl)-1*H*-tetrazole (**3a**): Yield: 69 %. Brownish crystals, mp 129-131 °C; <sup>1</sup>H NMR: (300 MHz, CDCl<sub>3</sub>)  $\delta$  7.51-7.42 (2H, m), 7.29-7.21 (2H, m), 4.38-4.25 (1H, m), 2.44 (3H, s), 2.18-1.85 (6H, m), 1.78-1.70 (1H, m), 1.40-1.26 (3H, m); <sup>13</sup>C NMR: (75 MHz, CDCl<sub>3</sub>)  $\delta$  153.7, 141.4, 129.9, 128.7, 121.4, 58.1, 33.2, 25.3, 24.8, 21.5; IR: (ATR) v<sub>max</sub> 1004, 1028, 1095, 1159, 1180, 1260, 1332, 1391, 1409, 1442, 1455, 1477, 1616, 2858, 2923, 2945 cm<sup>-1</sup>; LRMS: *m/z* (relative intensity) 243.1 [M+H]<sup>+</sup> (100), 218.2 (2), 161.1 (18), 133.1 (1), 118.1 (2); Anal. Calcd for C<sub>14</sub>H<sub>18</sub>N<sub>4</sub>: C, 69.39; H, 7.49; N, 23.12. Found: C, 69.31; H, 7.41; N, 23.23.

1-Cyclohexyl-5-(thiophen-2-yl)-1*H*-tetrazole (**3b**): Yield: 26 %. Yellow solid; <sup>1</sup>H NMR: (300 MHz, CDCl<sub>3</sub>)  $\delta$  7.64-7.58 (2H, m), 7.27-7.21 (1H, m), 4.57-4.46 (1H, m), 2.18-1.88 (6H, m), 1.80-1.71 (1H, m), 1.48-1.23 (3H, m); <sup>13</sup>C NMR: (75 MHz, CDCl<sub>3</sub>)  $\delta$  148.8, 130.0, 129.9, 128.3, 124.4, 58.6, 32.9, 25.3, 24.8; IR: (ATR)  $\nu_{max}$  1047, 1086, 1153, 1186, 1226, 1243, 1346, 1410, 1448, 1558, 2214, 2851, 2926, 3101 cm<sup>-1</sup>; LRMS: *m/z* (relative intensity) 235.2 [M+H]<sup>+</sup> (100), 225.3 (85), 196.1 (18), 180.4 (16), 167.5 (17), 153.1 (49), 143.1 (9), 127.1

(20); Anal. Calcd for C<sub>11</sub>H<sub>14</sub>N<sub>4</sub>S: C, 56.38; H, 6.02; N, 23.91; S, 13.68. Found: C, 56.61; H, 6.23; N, 24.07; S, 13.80.

1-Cyclohexyl-5-phenyl-1*H*-tetrazole (**3c**):<sup>6</sup> Yield: 66 %. White crystals, mp 131-133 °C; <sup>1</sup>H NMR: (300 MHz, CDCl<sub>3</sub>)  $\delta$  7.62-7.54 (5H, m), 4.39-4.25 (1H, m), 2.20-1.90 (6H, m), 1.77-1.72 (1H, m), 1.41-1.29 (3H, m); <sup>13</sup>C NMR: (75 MHz, CDCl<sub>3</sub>)  $\delta$  153.6, 131.1, 129.3, 128.9, 124.5, 58.2, 33.2, 25.3, 24.8; IR: (ATR) v<sub>max</sub> 1007, 1078, 1100, 1162, 1244, 1278, 1335, 1378, 1394, 1454, 1468, 1532, 2861, 2946 cm<sup>-1</sup>; LRMS: *m/z* (relative intensity) 229.1 [M+H]<sup>+</sup> (100), 218.9 (2), 195.1 (3), 165.3 (2), 147.1 (19); Anal. Calcd for C<sub>13</sub>H<sub>16</sub>N<sub>4</sub>: C, 68.39; H, 7.06; N, 24.54. Found: C, 68.58; H, 7.27; N, 24.42.

1-Cyclohexyl-5-(4-methoxyphenyl)-1*H*-tetrazole (**3d**): Yield: 54 %. Brownish crystals, mp 109-111 °C; <sup>1</sup>H NMR: (300 MHz, CDCl<sub>3</sub>)  $\delta$  7.57-7.52 (2H, m), 7.08-7.03 (2H, m), 4.38-4.25 (1H, m), 3.88 (3H, s), 2.19-1.89 (6H, m), 1.79-1.70 (1H, m), 1.43-1.25 (3H, m); <sup>13</sup>C NMR: (75 MHz, CDCl<sub>3</sub>)  $\delta$  161.7, 153.5, 130.3, 116.4, 114.7, 58.1, 55.5, 33.2, 25.3, 24.8; IR: (ATR) v<sub>max</sub> 1001, 1026, 1041, 1099, 1176, 1260, 1299, 1381, 1438, 1458, 1478, 1578, 1612, 2360, 2856, 2937, 3009 cm<sup>-1</sup>; LRMS: *m/z* (relative intensity) 259.1 [M+H]<sup>+</sup> (100), 248.3 (23), 225.3 (21), 209.4 (2), 177.1 (45), 167.1 (3), 147.2 (4), 134.2 (6); Anal. Calcd for C<sub>14</sub>H<sub>18</sub>N<sub>4</sub>O: C, 65.09; H, 7.02; N, 21.69. Found: C, 65.00; H, 6.81; N, 21.45.

5-(4-Chlorophenyl)-1-cyclohexyl-1*H*-tetrazole (**3e**): Yield: 35 %. Yellowish crystals, mp 145-147 °C; <sup>1</sup>H NMR: (300 MHz, CDCl<sub>3</sub>)  $\delta$  7.58-7.46 (4H, m), 4.33-4.20 (1H, m), 2.14-1.84 (6H, m), 1.77-1.68 (1H, m), 1.41-1.22 (3H, m); <sup>13</sup>C NMR: (75 MHz, CDCl<sub>3</sub>)  $\delta$  152.7, 137.4, 130.1, 129.6, 122.7, 58.2, 33.1, 25.1, 24.6; IR: (ATR) v<sub>max</sub> 1004, 1089, 1095, 1118, 1160, 1243, 1275, 1328, 1348, 1374, 1421, 1446, 1462, 1603, 2859, 2933, 3082 cm<sup>-1</sup>; LRMS: *m/z* (relative intensity) 263.1 [M+H]<sup>+</sup> (100), 252.3 (84), 243.3 (7), 231.9 (5), 224.3 (8), 180.9 (34), 169.7 (9), 161.3 (7), 153.1 (4); Anal. Calcd for C<sub>13</sub>H<sub>15</sub>ClN<sub>4</sub>: C, 59.43; H, 5.75; N, 21.32. Found: C, 59.40; H, 5.71; N, 21.08.

(*Z*)-5-[1,4-Bis(tetrahydropyran-2-yloxy)but-2-en-2-yl]-1-cyclohexyl-1*H*-tetrazole (**3f**): Yield: 63 %. Yellow oil, mixture of diastereomers; <sup>1</sup>H NMR: (300 MHz, CDCl<sub>3</sub>)  $\delta$  6.06 (1H, t, *J* = 6.0 Hz), 4.65-4.26 (7H, m), 3.85-3.77 (1H, m), 3.72-3.63 (1H, m), 3.53-3.39 (2H, m), 2.02-1.25 (22H, m); <sup>13</sup>C NMR: (75 MHz, CDCl<sub>3</sub>)  $\delta$  153.3, 137.6, 124.7, 98.5, 63.9, 63.0, 62.3, 62.2, 57.8, 33.0, 32.9, 30.4, 30.1, 25.2, 25.0, 24.7, 19.2, 19.1; IR: (ATR) v<sub>max</sub> 1022, 1061, 1076, 1117, 1201, 1262, 1351, 1405, 1452, 1502, 2857, 2935 cm<sup>-1</sup>; LRMS: *m/z* (relative intensity) 429.3 [M+Na]<sup>+</sup> (100), 407.1 [M+H]<sup>+</sup> (6), 384.3 (3), 323.2 (26), 269.2 (2), 239.2 (15), 139.0 (6); Anal. Calcd for C<sub>21</sub>H<sub>34</sub>N<sub>4</sub>O<sub>4</sub>: C, 62.04; H, 8.43; N, 13.78. Found: C, 62.21; H, 8.31; N, 13.88.

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