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Catalytic Alkynylation of Ketones and Aldehydes Using Quaternary Ammonium Hydroxide Base

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Spectral Data for 4, 6, and 7 including analytical data for new compounds

General. ¹H-NMR spectral data (300 MHz for ¹H and 75 MHz for ¹³C) were indicated in the form: δ -value of signal [peak multiplicity, integrated number of protons and coupling constant (if any)]. Splitting patterns are abbreviated as follows: s, singlet; d, doublet; t, triplet; m, multiplet; b, broad. For infrared spectral data only major absorptions were indicated.

Alkynes (1a–e) and carbonyl compounds (2a–p). Almost all of these compounds are commercially available except for **1e**:

3-Benzylloxy-1-propyne (1e). To a solution of propargyl alcohol (2 mL, 33.9 mmol) in THF-DMF (1 : 1 by volume, 100 mL) was added sodium hydride (1.1 equiv) at 0 °C, and the mixture was stirred at 0 °C for 30 min. To the mixture was added benzyl bromide (4.4 mL, 1.1 equiv) at 0 °C during 5 min, and the resulting mixture was stirred at 0 °C to room temperature for 17 h. The reaction was quenched by the addition of aqueous NH₄Cl and extracted several times with ether. The combined ether solutions were dried (MgSO₄) and concentrated by a rotary evaporator to give crude product, which was purified by silica gel column chromatography to afford **1e** (4.01 g, 81%): ¹H NMR δ 2.50 (t, 1H, J = 2.5 Hz), 4.20 (d, 2H, J = 2.5 Hz), 4.64 (s, 2H), 7.26–7.44 (m, 5H); ¹³C NMR δ 57.0, 71.4, 74.6, 79.6, 127.8, 128.0, 128.4, 137.2; IR (film) 3294, 2118 cm⁻¹.

1-(2-Phenylethynyl)cyclohexanol (4a). ^1H NMR δ 1.14–2.08 (m, 10H), 2.06 (s, 1H), 7.28–7.48 (m, 5H); ^{13}C NMR δ 23.4, 25.2, 40.0, 69.1, 84.3, 92.8, 112.8, 128.1, 128.2, 131.6; IR (film) 3350, 2358 cm^{-1} .

2-Methyl-1-(2-phenylethynyl)cyclohexanol (4b); isomer 1: ^1H NMR δ 1.13 (d, 3H, $J = 6.9$ Hz), 1.21–1.85 (m, 9H), 2.11 (bs, 1H), 7.22–7.47 (m, 5H); ^{13}C NMR δ 16.2, 21.1, 25.1, 29.2, 39.3, 40.7, 70.0, 83.1, 93.8, 122.9, 128.1, 128.2, 131.6; IR (film) 3445, 2361 cm^{-1} ; **isomer 2:** ^1H NMR δ 1.12 (d, 3H, $J = 6.3$ Hz), 1.27–1.81 (m, 8H), 2.04–2.17 (m, 1H), 2.36 (bs, 1H), 7.22–7.48 (m, 5H); ^{13}C NMR δ 16.3, 24.4, 25.6, 32.4, 40.8, 42.9, 73.6, 86.3, 90.1, 122.9, 128.18, 128.23, 131.7.

1-(1-Hexynyl)cyclohexanol (4c). ^1H NMR δ 0.90 (t, 3H, $J = 6.9$ Hz), 1.10–2.08 (m, 15H), 2.20 (t, 2H, $J = 6.9$ Hz); ^{13}C NMR δ 13.6, 18.3, 21.9, 23.4, 25.2, 27.0, 30.8, 40.2, 41.9, 68.8, 83.3, 84.7; IR (film) 3337, 2236 cm^{-1} .

Ethyl 3-[(1-hydroxy)cyclohexyl]propionate (4d). ^1H NMR δ 1.31 (t, 3H, $J = 7.2$ Hz), 1.47–1.82 (m, 8H), 1.89–2.05 (m, 2H), 2.38 (s, 1H), 4.23 (q, 2H, $J = 7.2$ Hz); ^{13}C NMR δ 14.0, 22.8, 24.8, 39.0, 62.0, 68.4, 75.9, 90.4, 153.6; IR (film) 3490, 2331, 1713, 1246 cm^{-1} .

Ethyl 3-[(1-hydroxy-2-methyl)cyclohexyl]propionate (4e). **isomer 1:** ^1H NMR δ 1.06 (d, 3H, $J = 6.6$ Hz), 1.10–1.25 (m, 2H), 1.30 (t, 3H, $J = 7.1$ Hz), 1.42–1.80 (m, 6H), 2.00–2.16 (m, 1H), 2.21 (bs, 1H), 4.23 (q, 2H, $J = 7.1$ Hz); ^{13}C NMR δ 14.0, 16.0, 24.0, 25.2, 32.0, 40.0, 42.5, 62.0, 73.2, 88.1, 111.8, 153.5; IR (film) 3490, 2340, 1713, 1246 cm^{-1} ; exact mass, m/z 210.12511 (calcd for $\text{C}_{12}\text{H}_{18}\text{O}_3$ m/z 210.12560). Anal. Calcd for $\text{C}_{12}\text{H}_{18}\text{O}_3$: C, 68.54; H, 8.63. Found: C, 68.44; H, 8.55.

4,4-Dimethyl-1-phenyl-1-pentyn-3-ol (4f). ^1H NMR δ 1.07 (s, 9H), 1.88 (d, 1H, $J = 5.6$ Hz), 4.24 (d, 1H, $J = 5.6$ Hz), 7.28–7.47 (m, 5H); ^{13}C NMR δ 25.4, 36.1, 71.9, 85.7, 88.9, 122.8, 128.32, 128.3, 131.7; IR (film) 3405, 2363 cm^{-1} .

4-Methyl-1-phenyl-1-pentyn-3-ol (4g). ^1H NMR δ 1.06 (d, 3H, $J = 7.1$ Hz), 1.09 (d, 3H, $J = 6.9$ Hz), 1.91–2.06 (m, 1H), 2.03 (d, 1H, $J = 5.5$ Hz), 4.37–4.43 (m, 1H), 7.25–7.47 (m, 5H); ^{13}C NMR δ 17.5, 18.2, 34.7, 68.3, 85.5, 88.8, 122.7, 128.2, 128.3, 131.6; IR (film) 3368, 2360 cm^{-1} .

1-Cyclohexyl-3-phenyl-2-butyn-1-ol (4h). ^1H NMR δ 1.15–1.40 (m, 5H), 1.56–1.97 (m, 7H), 4.38 (m, 1H), 7.28–7.49 (m, 5H); ^{13}C NMR δ 25.8, 26.3, 28.1, 28.6, 44.2, 67.5, 85.5, 89.2, 122.6, 128.1, 128.2, 131.5; IR (film) 3425, 3305, 2233 cm^{-1} .

2,2-Dimethyl-4-nonyn-3-ol (4i). ^1H NMR δ 0.89 (t, 3H, $J = 7.4$ Hz), 0.95 (s, 9H), 1.32–1.52 (m, 4H), 1.85 (bs, 1H), 2.17–2.23 (m, 2H), 3.96 (d, 1H, $J = 1.9$ Hz); ^{13}C NMR δ 13.5, 18.3, 21.9, 25.2, 30.8, 35.8, 71.5, 79.7, 86.1; IR (film) 3438, 2224 cm^{-1} .

2-Methyl-4-phenyl-3-butyn-2-ol (4j). ^1H NMR δ 1.63 (s, 6H), 2.40 (s, 1H), 7.35–7.45 (m, 5H); ^{13}C NMR δ 31.4, 65.5, 82.0, 93.8, 122.7, 128.2, 131.6; IR (film) 3356, 2231 cm^{-1} .

3-Ethyl-1-phenyl-1-pentyn-3-ol (4k). ^1H NMR δ 1.11 (t, 6H, $J = 7.5$ Hz), 1.11 (t, 3H, $J = 7.5$ Hz), 1.67–1.88 (m, 4H), 2.05 (bs, 1H), 7.27–7.47 (m, 5H); ^{13}C NMR δ 8.7, 34.4, 72.5, 84.4, 91.6, 122.8, 128.1, 128.2, 131.6; IR (film) 3400, 2226 cm^{-1} .

3,4-Dimethyl-1-phenyl-1-pentyn-4-ol (4l). ^1H NMR δ 1.07 (d, 3H, $J = 6.8$ Hz), 1.11 (d, 3H, $J = 6.8$ Hz), 1.54 (s, 3H), 1.91 (m, 1H, $J = 6.8$ Hz), 2.03 (s, 1H), 7.27–7.46 (m, 5H); ^{13}C NMR δ 17.5, 18.0, 39.1, 72.1, 84.0, 92.0, 122.9, 128.16, 128.21, 131.6; IR (film) 4408, 2230 cm^{-1} .

Ethyl 4-hydroxy-4,5-dimethyl-2-hexynoate (4m). ^1H NMR δ 1.02 (d, 3H, $J = 7.1$ Hz), 1.04 (d, 3H, $J = 6.9$ Hz), 1.30 (t, 3H, $J = 7.1$ Hz), 1.48 (s, 3H), 1.80–1.94 (m, 1H), 2.21 (bs, 1H), 4.22 (q, 2H, $J = 7.1$ Hz); ^{13}C NMR δ 14.0, 17.2, 17.6, 26.3, 38.6, 62.1, 71.5, 75.9, 89.8, 153.5; IR (film) 3504, 2240, 1714 cm^{-1} .

Ethyl 4-cyclohexyl-4-hydroxybutynoate (4n). ^1H NMR δ 1.15–1.40 (m, 5H), 1.30 (t, 1H, $J = 7.1$ Hz), 1.44–2.03 (m, 7H), 4.23 (q, 2H, $J = 7.1$ Hz), 4.26–4.40 (m, 1H); ^{13}C NMR δ 14.0, 22.8, 24.8, 39.0, 62.0, 68.4, 75.9, 90.4, 153.6; IR (film) 3412, 2360, 1713 cm^{-1} .

1-(3-Benzyloxy-1-propynyl)cyclohexanol (4o). ^1H NMR δ 1.45–1.98 (m, 10H), 2.11 (s, 1H), 4.23 (s, 2H), 4.61 (s, 2H), 7.26–7.39 (m, 5H); ^{13}C NMR δ 23.5, 25.1, 39.8, 57.4, 68.6, 71.5, 79.9, 90.3, 127.8, 128.1, 128.4, 137.4; IR (film) 3421, 2391 cm^{-1} ; exact mass, m/z 244.32811 (calcd for $\text{C}_{16}\text{H}_{20}\text{O}_2$ m/z 244.32880). Anal. Calcd for $\text{C}_{16}\text{H}_{20}\text{O}_2$: C, 78.65; H, 8.25. Found: C, 78.44; H, 8.33.

1-(3-Benzyloxy-1-propynyl)-2-methylcyclohexanol (4p). ^1H NMR δ 1.06 (d, 3H, $J = 6.6$ Hz), 1.11–1.28 (m, 2H), 1.40–1.85 (m, 6H), 2.00–2.16 (m, 1H), 2.11 (s, 1H), 4.23 (s, 2H), 4.61 (s, 2H), 7.26–7.39 (m, 5H); ^{13}C NMR δ 16.5, 23.5, 25.1, 39.8, 57.4, 68.6, 71.5, 79.9, 90.3, 127.8,

128.1, 128.4, 137.4; IR (film) 3421, 2380 cm^{-1} ; exact mass, m/z 258.16144 (calcd for $\text{C}_{17}\text{H}_{22}\text{O}_2$ m/z 258.16198). Anal. Calcd for $\text{C}_{17}\text{H}_{22}\text{O}_2$: C, 79.03; H, 8.58. Found: C, 78.96; H, 8.51.

1-[3-(2-Tetrahydropyranyloxy)-1-propynyl]-2-methylcyclohexanol (4q). ^1H NMR δ 1.04 (d, 3H, $J = 6.6$ Hz), 1.17–1.89 (m, 15H), 1.94–2.06 (m, 1H), 3.47–3.56 (m, 1H), 3.79–3.87 (m, 1H), 4.26 and 4.32 (ABq, 2H, $J = 15.7$ Hz), 4.81 (t, 1H, $J = 3.2$ Hz); ^{13}C NMR δ 16.1, 19.0, 21.0, 25.0, 25.4, 29.1, 30.3, 39.3, 40.4, 40.5, 54.2, 62.0, 69.5, 78.9, 90.6, 96.5, 96.6; IR (film) 3448, 2362 cm^{-1} ; exact mass, m/z 252.34897 (calcd for $\text{C}_{15}\text{H}_{24}\text{O}_3$ m/z 252.34926). Anal. Calcd for $\text{C}_{15}\text{H}_{24}\text{O}_3$: C, 71.39; H, 9.59. Found: C, 71.23; H, 9.48.

1-O-Benzyl-5,5-dimethyl-2-hexyne-1,4-diol (4r). ^1H NMR δ 1.02 (s, 9H), 1.71 (b, 1H), 4.08 (t, 1H, $J = 1.8$ Hz), 4.23 (d, 2H, $J = 1.8$ Hz), 4.61 (s, 2H), 7.27–7.37 (m, 5H); ^{13}C NMR δ 25.3, 35.8, 57.4, 71.4, 71.6, 81.7, 86.3, 127.9, 128.1, 128.4, 137.4; IR (film) 3440, 2280 cm^{-1} .

1-O-Benzyl-5-methyl-2-hexyne-1,4-diol (4s). ^1H NMR δ 1.01 (d, 3H, $J = 6.6$ Hz), 1.03 (d, 3H, $J = 6.6$ Hz), 1.82–1.94 (m, 1H), 2.18 (bs, 1H), 4.22 (s, 2H), 4.22 (m, 1H), 4.60 (s, 2H), 7.26–7.38 (m, 5H); ^{13}C NMR δ 17.5, 18.1, 34.4, 57.3, 67.8, 71.5, 81.3, 86.4, 127.8, 128.0, 128.4, 137.3; IR (film) 3411, 2280 cm^{-1} .

4-Methyl-1-(O-2-tetrahydropyranyl)-2-pentyne-1,4-diol (4t). ^1H NMR δ 1.47 (s, 6H), 1.42–1.86 (m, 6H), 2.74 (bs, 1H), 3.46–3.54 (m, 1H), 3.80 (m, 1H), 4.20 and 4.28 (ABq, 2H, $J = 15.7$ Hz), 4.78 (t, 1H, $J = 3.0$ Hz); ^{13}C NMR δ 18.8, 25.2, 30.1, 31.2, 54.2, 61.7, 64.8, 77.7, 90.7, 96.5; IR (film) 3421, 2360 cm^{-1} .

4-Ethyl-1-(O-2-tetrahydropyranyl)-2-hexyne-1,4-diol (4u). ^1H NMR δ 1.01 (t, 6H, $J = 7.4$ Hz), 1.44–1.91 (m, 10H), 2.13 (s, 1H), 3.45–3.55 (m, 1H), 3.76–3.87 (m, 1H), 4.82 (t, 1H, $J = 3.0$ Hz); ^{13}C NMR δ 8.5, 19.0, 25.3, 30.2, 34.2, 34.2, 54.1, 61.9, 72.0, 80.1, 88.4, 96.4; IR (film) 3450, 2362 cm^{-1} ; exact mass, m/z 226.15672 (calcd for $\text{C}_{13}\text{H}_{22}\text{O}_3$ m/z 226.15689). Anal. Calcd for $\text{C}_{13}\text{H}_{22}\text{O}_3$: C, 68.99; H, 9.80. Found: C, 69.15; H, 9.92.

1-O-Benzyl-4,5-dimethyl-2-hexyne-1,4-diol (4v). ^1H NMR δ 1.02 (d, 3H, $J = 7.1$ Hz), 1.04 (d, 3H, $J = 6.9$ Hz), 1.48 (s, 3H), 1.80–1.94 (m, 1H), 2.21 (s, 1H), 4.24 (s, 2H), 4.61 (s, 2H), 7.27–7.37 (m, 5H); ^{13}C NMR δ 17.5, 18.1, 34.4, 57.3, 67.8, 71.5, 81.3, 86.4, 127.8, 128.0, 128.4, 137.3; IR (film) 3410, 2360 cm^{-1} ; exact mass, m/z 232.14610 (calcd for $\text{C}_{15}\text{H}_{20}\text{O}_2$ m/z 232.14633). Anal. Calcd for $\text{C}_{15}\text{H}_{20}\text{O}_2$: C, 77.55; H, 8.68. Found: C, 77.27; H, 8.70.

2,4-Diphenyl-3-butyn-2-ol (4w). ^1H NMR δ 1.89 (s, 3H), 2.59 (bs, 1H), 7.22–7.77 (m, 10H); ^{13}C NMR δ 33.2, 70.4, 84.9, 92.4, 122.5, 124.9, 127.7, 128.26, 128.30, 128.5, 131.7, 145.6; IR (film) 3380, 2235 cm^{-1} .

1-O-Benzyl-4-phenyl-2-pentyne-1,4-diol (4x). ^1H NMR δ 1.80 (s, 3H), 2.55 (bs, 1H), 4.28 (s, 2H), 4.63 (s, 2H), 7.24–7.70 (m, 10H); ^{13}C NMR δ 33.2, 57.4, 69.9, 71.7, 80.8, 90.0, 124.8, 127.7, 127.9, 128.1, 128.3, 128.4, 137.3, 145.3; IR (film) 3420, 2362 cm^{-1} ; exact mass, m/z 266.13022 (calcd for $\text{C}_{18}\text{H}_{18}\text{O}_2$ m/z 266.13068). Anal. Calcd for $\text{C}_{18}\text{H}_{18}\text{O}_2$: C, 81.17; H, 6.81. Found: C, 81.02; H, 6.90.

4-Phenyl-1-(O-2-tetrahydropyranyl)-2-pentyne-1,4-diol (4y). *isomer 1:* ^1H NMR δ 1.24–1.91 (m, 6H), 1.77 (s, 3H), 2.78 (s, 1H), 3.47–3.55 (m, 1H), 3.78–3.89 (m, 1H), 4.32 and 4.39 (ABq, 2H, $J = 15.7$ Hz), 4.84 (t, 1H, $J = 2.9$ Hz), 7.25–7.70 (m, 5H); ^{13}C NMR δ 18.9, 25.3, 30.2, 33.2, 54.3, 61.9, 69.8, 80.8, 89.3, 96.7, 96.7, 124.9, 127.6, 128.2, 145.4; IR (film) 3407, 2336 cm^{-1} ; exact mass, m/z 260.12098 (calcd for $\text{C}_{16}\text{H}_{20}\text{O}_3$ m/z 260.12124). Anal. Calcd for $\text{C}_{16}\text{H}_{20}\text{O}_3$: C, 73.82; H, 7.74. Found: C, 73.62; H, 7.68.

1-Phenyl-4-(O-2-tetrahydropyranyl)-2-butyne-1,4-diol (4z). *isomer 1:* ^1H NMR δ 1.39–1.90 (m, 6H), 2.22 (d, 1H, $J = 5.8$ Hz), 2.48–2.56 (m, 1H), 3.76–3.89 (m, 1H), 4.28–4.42 (m, 2H), 4.82 (t, 1H, $J = 3.2$ Hz), 5.52 (d, 1H, $J = 5.2$ Hz), 7.25–7.58 (m, 5H); ^{13}C NMR δ 19.0, 25.3, 30.2, 54.3, 62.0, 64.6, 82.7, 85.6, 126.6, 128.4, 128.6, 140.4; IR (film) 3422, 2358 cm^{-1} .

1-O-Benzyl-4-(4-methoxyphenyl)-2-butyne-1,4-diol (4aa). ^1H NMR δ 2.17 (d, 1H, $J = 5.8$ Hz), 3.82 (s, 3H), 4.27 (s, 2H), 4.60 (s, 2H), 5.48 (d, 1H, $J = 5.8$ Hz), 6.91 (d, 2H, $J = 8.8$ Hz), 7.36–7.46 (m, 5H), 7.47 (d, 2H, $J = 8.8$ Hz); ^{13}C NMR δ 55.3, 57.5, 64.3, 71.7, 82.5, 86.4, 114.0, 127.9, 128.0, 128.1, 132.7, 137.3, 159.7; IR (film) 3421, 2360 cm^{-1} ; exact mass, m/z 282.12588 (calcd for $\text{C}_{18}\text{H}_{18}\text{O}_3$ m/z 282.12559). Anal. Calcd for $\text{C}_{18}\text{H}_{18}\text{O}_3$: C, 76.57; H, 6.43. Found: C, 76.66; H, 6.50.

1-O-Benzyl-4-(4-chlorophenyl)-2-butyne-1,4-diol (4bb). ^1H NMR δ 2.10 (d, 1H, $J = 5.8$ Hz), 4.06 (s, 2H), 4.63 (s, 2H), 5.55 (d, 1H, $J = 5.8$ Hz), 7.11–7.14 (m, 2H), 7.18–7.21 (m, 2H), 7.28–7.49 (m, 5H); ^{13}C NMR δ 57.7, 66.4, 74.5, 84.9, 85.0, 127.6, 127.7, 128.4, 128.7, 129.1, 132.7, 137.2, 139.0; IR (film) 3418, 2365 cm^{-1} ;

1-O-Benzyl-4-(3-chlorophenyl)-2-butyne-1,4-diol (4cc). ^1H NMR δ 2.11 (d, 1H, $J = 5.5$ Hz), 4.06 (s, 2H), 4.63 (s, 2H), 5.55 (d, 1H, $J = 5.5$ Hz), 7.07–7.20 (m, 4H), 7.28–7.49 (m, 5H);

^{13}C NMR δ 57.6, 65.8, 84.8, 84.9, 125.4, 127.5, 127.6, 127.7, 128.4, 128.7, 130.1, 134.0, 137.2, 142.3; IR (film) 3420, 2361 cm^{-1} ;

1-(3-methoxyphenyl)-3-phenyl-2-propyn-1-ol (4dd). ^1H NMR δ 2.12 (bs, 1H), 3.81 (s, 3H), 5.65 (s, 1H), 7.30–7.50 (m, 5H), 7.54–7.65 (m, 4H); ^{13}C NMR δ 51.5, 85.6, 87.2, 122.4, 128.2, 128.4, 131.6; IR (film) 2220, 3400 cm^{-1} .

4'-Methoxychalcone (6b). ^1H NMR δ 3.88 (s, 3H), 6.96–6.99 (m, 2H), 7.39–7.43 (m, 3H), 7.53 (d, 1H, $J = 15.6$ Hz), 7.63–7.66 (m, 2H), 7.80 (d, 1H, $J = 15.6$ Hz), 8.03–8.06 (m, 2H); ^{13}C NMR δ 55.5, 113.8, 121.9, 128.3, 128.9, 130.3, 130.8, 131.1, 135.1, 143.9, 163.4, 188.6; IR (film) 1655, 974 cm^{-1} .

4'-Chlorochalcone (6c). ^1H NMR δ 7.18–7.43 (m, 5H), 7.56 (d, 1H, $J = 15.6$ Hz), 7.63–7.66 (m, 2H), 7.90 (d, 1H, $J = 15.6$ Hz), 7.90–8.00 (m, 2H); ^{13}C NMR δ 123.3, 126.2, 128.4, 127.7, 129.4, 131.1, 134.8, 134.9, 139.6, 142.8, 187.0; IR (film) 1652, 977 cm^{-1} .

3'-Chlorochalcone (6d). ^1H NMR δ 7.12–7.44 (m, 4H), 7.56 (d, 1H, $J = 15.7$ Hz), 7.54–7.65 (m, 5H), 7.90 (d, 1H, $J = 15.7$ Hz); ^{13}C NMR δ 123.4, 126.2, 127.7, 127.8, 128.4, 130.1, 130.4, 134.3, 134.7, 134.9, 138.1, 142.9, 187.2; IR (film) 1650, 981 cm^{-1} .

4'-Methylchalcone (6e). ^1H NMR δ 2.09 (s, 3H), 7.15–7.26 (m, 4H), 7.35–7.60 (m, 5H), 7.60 (d, 1H, $J = 15.6$ Hz), 7.80 (d, 1H, $J = 15.6$ Hz); ^{13}C NMR δ 25.8, 111.0, 122.5, 126.0, 128.2, 125.9, 128.2, 128.8, 129.9, 130.8, 135.5, 142.5, 153.4, 190.1; IR (film) 1649, 971 cm^{-1} .

4'-(Dimethylamino)chalcone (6f). ^1H NMR δ 3.08 (s, 6H), 6.68–6.75 (m, 2H), 7.35–7.45 (m, 5H), 7.60 (d, 1H, $J = 15.6$ Hz), 7.63–7.68 (m, 2H), 7.80 (d, 1H, $J = 15.6$ Hz), 7.96–8.04 (m, 2H); ^{13}C NMR δ 40.0, 110.8, 122.1, 125.9, 128.2, 125.9, 128.2, 128.8, 129.9, 130.8, 135.5, 142.5, 153.4, 188.7; IR (film) 1658, 970 cm^{-1} .

3'-Methoxychalcone (6g). ^1H NMR δ 3.88 (s, 3H), 7.12–7.16 (m, 1H), 7.38–7.44 (m, 3H), 7.50 (d, 1H, $J = 15.6$ Hz), 7.54–7.65 (m, 5H), 7.80 (d, 1H, $J = 15.6$ Hz); ^{13}C NMR δ 55.4, 112.8, 119.2, 121.0, 122.0, 128.4, 128.9, 129.5, 130.5, 134.8, 139.5, 144.8, 159.8, 190.1; IR (film) 1650, 980 cm^{-1} .

3-Phenyl-2-propyn-1-ol (7a). ^1H NMR δ 2.33 (bs, 1H), 4.50 (d, 2H, $J = 5.5$ Hz), 7.28–7.47 (m, 5H); ^{13}C NMR δ 51.5, 85.6, 87.2, 122.4, 128.2, 128.4, 131.6; IR (film) 2228, 3405 cm^{-1} .

Ethyl 4-hydroxy-2-butynoate (7c). ^1H NMR δ 1.30 (t, 3H, $J = 7.1$ Hz), 2.00 (s, 1H), 4.20 (s, 2H), 4.27 (q, 2H, $J = 7.1$ Hz); ^{13}C NMR δ 12.7, 49.9, 58.3, 76.7, 86.7, 143.0; IR (film) 1713, 2226, 3490 cm^{-1} .

1-*O*-Benzyl-2-butyne-1,4-diol (7d). ^1H NMR δ 1.71 (b, 1H), 4.08 (t, 2H, $J = 1.8$ Hz), 4.23 (d, 2H, $J = 1.8$ Hz), 4.61 (s, 2H), 7.27–7.37 (m, 5H); ^{13}C NMR δ 57.4, 71.4, 71.6, 81.7, 86.3, 127.9, 128.1, 128.4, 137.4; IR (film) 2218, 3440 cm^{-1} .