

Supporting information

Magnetically recoverable graphene-based nanocomposite material as an efficient catalyst for the synthesis of propargylamines via A³ coupling reaction

Najrul Hussain^{ab*} and Manash R. Das^{ab}

^a Materials Sciences & Technology Division, CSIR-North East Institute of Science and Technology, Jorhat-785006, Assam, India.

^b Academy of Scientific and Innovative Research, CSIR-NEIST Campus, India

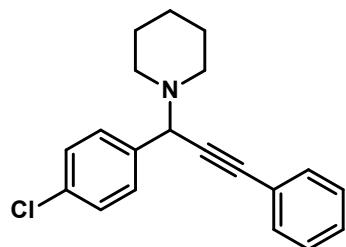
*To whom correspondence should be addressed

Najrul Hussain

Materials Sciences & Technology Division,
CSIR–North East Institute of Science and Technology, Jorhat–785006, Assam, India
Tel: +91-9864582365
Fax: +91–376–2370011,
E Mail: najrul0588@gmail.com

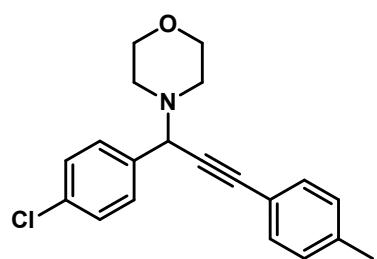
Analytical data:

1-(1-(4-chlorophenyl)-3-phenylprop-2-yn-1-yl)piperidine: 4a



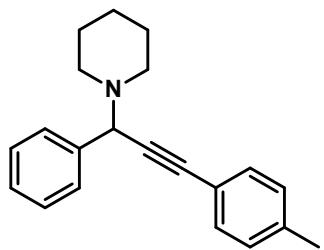
[Yield = 97%]; ^1H NMR (500 MHz, CDCl_3): δ = 7.55-7.59 (m, 2H), 7.49-7.53 (m, 2H), 7.29-7.35 (m, 5H), 4.75 (s, 1H), 2.50-2.55 (m, 4H), 1.51-1.64 (m, 4H), 1.41-1.45 (m, 2H); ^{13}C NMR (125 MHz, CDCl_3): δ = 137.17, 133.07, 131.70, 129.71, 128.21, 128.10, 128.08, 122.97, 88.11, 85.25, 61.61, 50.51, 26.04, 24.26; IR (CHCl_3): 3064, 2926, 2854, 1597, 1485, 1454, 1091, 848, 765, 688 cm^{-1} .

4-(1-(4-chlorophenyl)-3-(p-tolyl)prop-2-yn-1-yl)morpholine: 4b



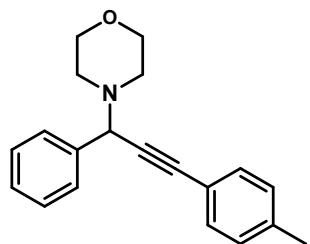
[Yield = 97%]; ^1H NMR (500 MHz, CDCl_3): δ = 7.57 (d, J = 8.5 Hz, 2H), 7.40 (d, J = 8 Hz, 2H), 7.31-7.34 (m, 2H), 7.14 (d, J = 7.5 Hz, 2H), 4.74 (s, 1H), 3.69-3.76 (m, 4H), 2.58-2.62 (m, 4H), 2.36 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3): δ = 138.44, 136.45, 133.40, 131.58, 129.81, 128.99, 128.24, 119.5, 88.88, 83.45, 67.00, 61.26, 49.63, 21.39; IR (CHCl_3): 2969, 2924, 2852, 1724, 1450, 1216, 1111, 1008, 817, 761 cm^{-1} .

1-(1-phenyl-3-(p-tolyl)prop-2-yn-1-yl)piperidine: 4c



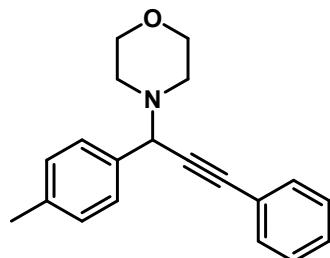
[Yields = 96 %]; ^1H NMR (500 MHz, CDCl_3): δ = 7.62 (d, $J=7.5$ Hz, 2H), 7.22-7.43 (m, 5H), 7.12 (d, $J=8$ Hz, 2H), 4.78 (s, 1H), 2.53-2.56 (m, 4H), 2.35 (s, 1H), 1.51-1.64 (m, 4H), 1.39-1.46 (m, 2H); ^{13}C NMR (125 MHz, CDCl_3): δ = 138.54, 138.0, 131.57, 128.91, 128.48, 127.92, 127.31, 120.12, 87.77, 85.17, 62.29, 50.55, 26.05, 24.32, 21.37; IR (CHCl_3): 2953, 2929, 2852, 1504, 1446, 1273, 1103, 981, 815, 768, 704 cm^{-1} .

4-(1-phenyl-3-(p-tolyl)prop-2-yn-1-yl)morpholine: 4d



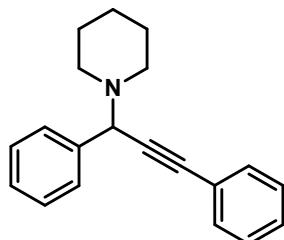
[Yield = 95 %]; ^1H NMR (500 MHz, CDCl_3): δ = 7.63 (d, $J=7$ Hz, 2H), 7.28-7.43 (m, 5H), 7.14 (d, $J=8$ Hz, 2H), 4.78 (s, 1H), 3.70-3.78 (m, 4H), 2.63 (t, $J=5$ Hz, 4H), 2.36 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3): δ = 138.31, 137.68, 131.59, 128.99, 128.56, 128.15, 127.71, 119.69, 88.44, 84.08, 84.08, 67.06, 61.91, 49.84, 21.48; IR (CHCl_3): 3013, 2922, 2852, 1718, 1502, 1450, 1278, 1114, 1002, 864, 763 cm^{-1} .

4-(3-phenyl-1-(p-tolyl)prop-2-yn-1-yl)morpholine: 4e



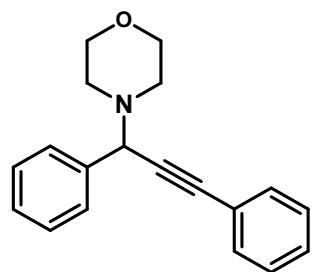
[Yield = 91%]; ^1H NMR (500 MHz, CDCl_3): δ = 7.48-7.52 (m, 4H), 7.31-7.34 (m, 3H), 7.17 (d, J = 8 Hz, 2H), 4.75 (s, 1H), 3.70-3.77 (m, 4H), 2.59-2.64 (m, 4H), 2.35 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3): δ = 137.41, 134.62, 131.69, 128.82, 128.43, 128.19, 128.11, 122.9, 88.12, 85.18, 67.05, 61.67, 49.77, 21.03; IR (CHCl_3): 3010, 2932, 2842, 1711, 1493, 1457, 1268, 1018, 1022, 868, 760 cm^{-1}

1-(1,3-diphenylprop-2-yn-1-yl)piperidine: 4f



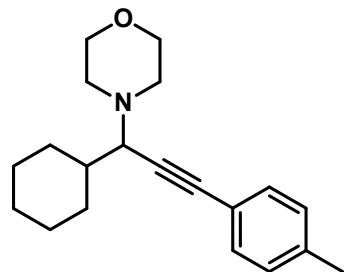
[Yield = 94%]; ^1H NMR (500 MHz, CDCl_3): δ = 7.62 (d, J = 7.5 Hz, 2H), 7.49-7.53 (m, 2H), 7.26-7.38 (m, 6H), 4.79 (s, 1H), 2.53-2.58 (m, 4H), 1.53-1.64 (m, 4H), 1.40-1.47 (m, 2H); ^{13}C NMR (125 MHz, CDCl_3): δ = 138.45, 131.69, 128.43, 128.15, 127.94, 127.34, 123.22, 87.71, 85.95, 62.27, 50.59, 26.05, 24.31; IR (CHCl_3): 3094, 2884, 1597, 1487, 1101, 852, 768, 700 cm^{-1} .

4-(1,3-diphenylprop-2-yn-1-yl)morpholine: 4g



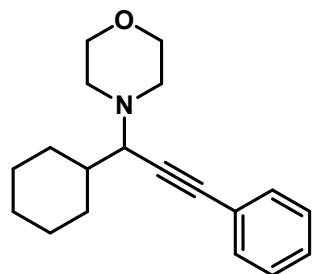
[Yield = 94%]; ^1H NMR (500 MHz, CDCl_3): δ = 7.62 (d, $J=7$ Hz, 2H), 7.48-7.54 (m, 2H), 7.29-7.39 (m, 6H), 4.79 (s, 1H), 3.68-3.78 (m, 4H), 2.60-2.68 (m, 4H); ^{13}C NMR (125 MHz, CDCl_3): δ = 137.64, 131.71, 128.50, 128.22, 128.17, 128.14, 127.69, 122.84, 88.38, 84.90, 67.04, 61.92, 49.71; IR (CHCl_3): 2965, 2941, 2839, 1712, 1465, 1294, 1114, 858, 765, 700 cm^{-1} .

4-(1-cyclohexyl-3-(p-tolyl)prop-2-yn-1-yl)morpholine: 4h



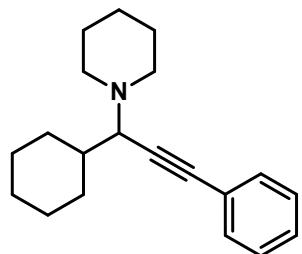
[Yield = 95 %]; ^1H NMR (500 MHz, CDCl_3): δ = 7.31-7.34 (m, 2H), 7.08-7.12 (m, 2H), 3.68-3.79 (m, 4H), 3.11 (d, $J=9.5$ Hz, 1H), 2.66-2.74 (m, 2H), 2.47-2.53 (m, 2H), 2.34 (s, 3H), 1.99-2.13 (m, 2H), 1.55-1.78 (m, 6H), 0.86-1.08 (m, 3H); ^{13}C NMR (125 MHz, CDCl_3): δ = 137.5, 131.4, 128.8, 120.2, 86.7, 85.6, 67.1, 63.8, 49.8, 39.0, 26.6, 26.0, 25.9, 21.2); IR (CHCl_3): 2923, 285, 1593, 1321, 1263, 683 cm^{-1} .

4-(1-cyclohexyl-3-phenylprop-2-yn-1-yl)morpholine: 4i



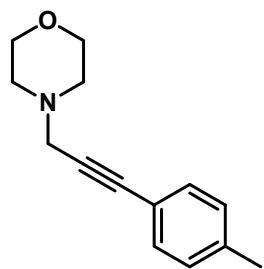
[Yield = 93%]; ^1H NMR (500 MHz, CDCl_3): δ = 7.42-7.46 (m, 2H), 7.28-7.33 (m, 3H), 3.69-3.84 (m, 4H), 3.13 (d, J = 10 Hz, 1H), 2.66-2.75 (m, 2H), 2.47-2.55 (m, 2H), 1.98-2.15 (m, 2H), 1.54-1.83 (m, 3H), 0.88-1.33 (m, 6H); ^{13}C NMR (125 MHz, CDCl_3): δ = 131.63, 128.19, 127.84, 123.15, 86.59, 86.48, 67.10, 63.80, 49.68, 38.85, 30.88, 30.22, 26.59, 26.09, 25.94; IR (CHCl_3): 2924, 2850, 1597, 1489, 1219, 1116, 1004, 864, 782, 690, 565 cm^{-1} .

1-(1-cyclohexyl-3-phenylprop-2-yn-1-yl)piperidine: 4j



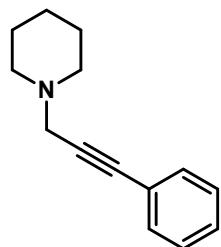
[Yield = 93 %]; ^1H NMR (500 MHz, CDCl_3): δ = 7.42-7.45 (m, 2H), 7.25-7.31 (m, 3H), 3.10 (d, J = 9.5 Hz, 1H), 2.59-2.66 (m, 2H), 2.36-2.43 (m, 2H), 2.03-2.14 (m, 2H), 1.70-1.79 (m, 2H), 1.50-1.67 (m, 6H), 1.38-1.47 (m, 2H), 1.12-1.33 (m, 3H), 0.85-1.06 (m, 2H); ^{13}C NMR (125 MHz, CDCl_3): δ = 131.60, 128.07, 127.51, 123.67, 87.64, 86.03, 64.27, 50.66, 39.47, 31.22, 30.33, 26.69, 26.16, 26.01, 24.6; IR (CHCl_3): 2900, 2842, 1567, 1234, 1214, 1034, 869, 772, 678, 561 cm^{-1} .

4-(3-(p-tolyl)prop-2-yn-1-yl)morpholine: 4k



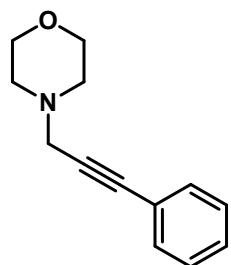
[Yield = 92%]; ^1H NMR (500 MHz, CDCl_3): δ = 7.23-7.27 (m, 2H), 7.01-7.04 (m, 2H), 3.67-3.72 (m, 4H), 3.42 (s, 2H), 2.54-2.60 (m, 4H), 2.26 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3): δ = 138.1, 131.4, 128.8, 119.7, 85.5, 83.0, 66.7, 52.2, 47.9, 21.3; IR (CHCl_3): 2931, 2843, 1718, 998, 851 cm^{-1} .

1-(3-phenylprop-2-yn-1-yl)piperidine: 4l



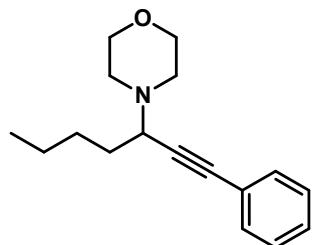
[Yield = 91%]; ^1H NMR (500 MHz, CDCl_3): δ = 7.40-7.46 (m, 2H), 7.27-7.32(m, 3H), 3.5 (s, 2H), 2.56(s, 4H), 1.62-1.69(m, 4H), 1.41-1.49 (m, 2H); ^{13}C NMR (125 MHz, CDCl_3): δ = 131.6, 128.1, 128.0, 122.9, 85.2, 84.2, 53.0, 48.05, 25.5, 23.6; IR (CHCl_3): 2931, 1537, 1219, 1109, 772, 690, 441 cm^{-1} .

4-(3-phenylprop-2-yn-1-yl)morpholine: 4m



[Yield = 91%]; ^1H NMR (500 MHz, CDCl_3): δ = 7.42-7.46(m, 2H), 7.28-7.32(m, 3H), 3.76-3.80(m, 4H), 3.5 (s, 2H), 2.62-2.69(m, 4H); ^{13}C NMR (125 MHz, CDCl_3): δ = 131.6, 128.2, 128.1, 122.7, 85.5, 83.8, 66.7, 52.2, 47.92; IR (CHCl_3): 2921, 1723, 1528, 673 cm^{-1} .

4-(1-phenylhept-1-yn-3-yl)morpholine: 4n



[Yield = 89 %]; ^1H NMR (500 MHz, CDCl_3): δ = 7.42-7.45 (m, 2H), 7.28-7.32 (m, 3H), 3.72-3.81 (m, 4H), 3.47-3.52 (m, 1H), 2.73-2.78 (m, 2H), 2.54-2.61 (m, 2H), 1.70-1.74 (m, 3H), 1.34-1.43 (m, 4H), 0.83-0.91 (m, 3H); ^{13}C NMR (125 MHz, CDCl_3): δ = 131.6, 128.1, 127.8, 123.0, 87.0, 86.1, 67.0, 58.1, 49.6, 32.5, 29.6, 28.7, 22.3, 13.9; IR (CHCl_3): 2964, 2813, 1705, 891 cm^{-1} .