Electronic Supplementary information

Organo-NHC catalyzed domino addition approach for the selective synthesis of 5-butynylisoxazoles and subsequent Sonogashira coupling

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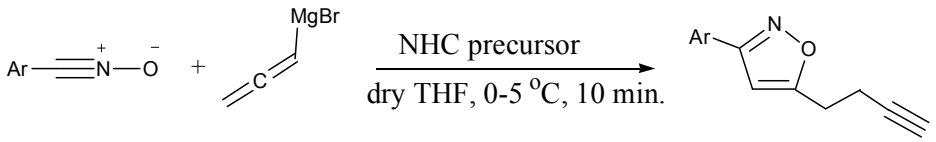
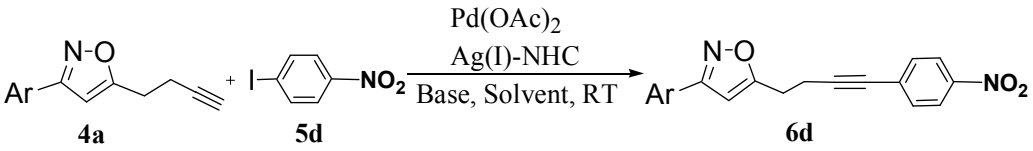
1. **Table S1**. Optimization of reaction conditions for organo-NHC catalyzed synthesis of 5-butynylisoxazole (**4f**).



1f **2** **4f**

Entry	NHC Precursor	GC yield (%)
1	i	94
2	ii	90
3	iii	90
4	iv	92
5	v	90

2. **Table S2**. Optimization of reaction conditions for Pd/Ag catalyzed Sonogashira cross-coupling for the synthesis of Internal alkyne of 5-butynylisoxazole (**6d**).



4a **5d** **6d**

Ar = 4-OMe-C_6H_4

Entry	Base	Solvent	GC yield (%)
1	Et_3N	DCE	74
2	Et_3N	DCM	85
3	Et_3N	Toluene	79
4	pyrrolidine	Toluene	71
5	pyrrolidine	DCM	93
6	K_2CO_3	DCE	82
7	K_2CO_3	DCM	83

3. Experimental

General

All commercially available reagents were used without further purification. Reaction solvents were dried by standard methods before use. Purity of the compounds was checked by TLC using Merck 60F254 silica gel plates. Elemental analyses were obtained with an Elemental Analyser Perkin-Elmer 240C apparatus. IR spectra were recorded with a Perkin-Elmer 881 spectrometer. ^{1}H and ^{13}C NMR spectra were recorded with a Mercuryplus 400 spectrometer (operating at 400 MHz for ^{1}H and 100.58 MHz for ^{13}C); chemical shifts were referenced to TMS. EI (electron impact) mass spectra (at an ionising voltage of 70 eV) were obtained using a Shimadzu QP5050A quadrupole-based mass spectrometer.

General procedure for the synthesis of 5-butynylisoxazoles (4a-l):

To a suspension of magnesium turnings (8 mmol, 8 equiv) in anhydrous tetrahydrofuran (20 mL) added mercury(II) chloride (5 mg, 1% w/w of propargyl bromide) and propargyl bromide (80 wt % solution in toluene, 6 mmol, 6 equiv) in small portions slowly to generate a cloudy pale yellow solution of allenylmagnesium bromide. This was cooled to 0-5 ºC and added with NHC precursor (1,3-Di-*tert*-butylimidazolium chloride (i)) (2 mmol). To this, a solution of *in situ* generated benzonitrile oxide (1 mmol), was added by maintaining the temperature between 0-5 ºC under stirring. The reaction mass was allowed to stir for 5 minutes at room temperature. After this conversion, the mixture is quenched by addition of saturated solution of NH_4Cl (2 mL) and diluted with dichloromethane (20 mL). The organic layer was separated and the aqueous layer extracted with dichloromethane (2 x 20 mL). The combined organic layers were dried (anhydrous Na_2SO_4) and evaporated under reduced pressure to afford a crude product which was subjected to column chromatography (silica gel, 60-120 mesh, eluent; n-hexane/EtOAc gradient) to afford pure products (**4a-l**).

Spectral Data of Compounds (4a-l):

3-(4-Methoxyphenyl)-5-butynylisoxazole (4a). ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C): δ = 2.04 (s, 1H), 2.64 (t, 2H, J = 7.2 Hz), 3.02 (t, 2H, J = 7.2 Hz), 3.85 (s, 3H), 6.37 (s, 1H), 6.99 (d, 2H, J = 8.7 Hz, Ar-H), 7.51 (d, 2H, J = 8.7 Hz, Ar-H) ppm. ^{13}C NMR (100 MHz, CDCl3, 25 °C): δ = 17.14, 26.11, 55.32, 69.72, 82.15, 99.42, 114.36, 121.74, 128.21, 160.94, 162.05, 171.42 ppm. IR (KBr): = 3281, 2964, 2937, 2837, 1608, 1524, 1462, 1430, 1253, 1176, 1052, 840, 792, 659, 533cm^{-1}. MS (EI, 70 eV): m/z (%) = 227 $[M]^+$. EA calcd (%) for $C_{14}H_{13}NO_2$ (227.09): calcd. C 73.99, H 5.77, N 6.16; found C 73.98, H 5.75, N 6.14.

3-(4-Methylphenyl)-5-butynylisoxazole (4b). ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C): δ = 2.04 (s, 1H), 2.28 (s, 3H), 2.64 (t, 2H, J = 7.2 Hz), 3.02 (t, 2H, J = 7.2 Hz), 6.37 (s, 1H), 6.99 (d, 2H, J = 8.7 Hz, Ar-H), 7.51 (d, 2H, J = 8.7 Hz, Ar-H) ppm. ^{13}C NMR (100 MHz, CDCl3, 25 °C): δ = 17.14, 19.24, 26.11, 69.72, 82.15, 99.44, 114.36, 121.72, 128.21, 160.94, 162.05, 171.46 ppm. IR (KBr): = 3281, 2964, 2937, 2835, 1608, 1520, 1462, 1430, 1253, 1176, 1052, 840, 792, 532cm^{-1}. MS (EI, 70 eV): m/z (%) = 211 $[M]^+$. EA calcd (%) for $C_{14}H_{13}NO$ (211.10): calcd. C 79.59, H 6.20, N 6.63; found C 79.58, H 6.18, N 6.60.

3-(2-Nitrophenyl)-5-butynylisoxazole (4c). ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C): δ = 2.04 (s, 1H), 2.64 (t, 2H, J = 7.2 Hz), 3.02 (t, 2H, J = 7.2 Hz), 6.37 (s, 1H), 7.05 (m, 3H, Ar-H), 8.12 (m, 1H, Ar-H) ppm. ^{13}C NMR (100 MHz, CDCl3, 25 °C): δ = 17.14, 26.11, 69.72, 82.15, 99.42, 114.36, 125.55, 129.26, 160.94, 162.05, 171.42 ppm. IR (KBr): = 3281, 2964, 2932, 2837, 1610, 1524, 1450, 1430, 1253, 1176, 1052, 840, 792, 659, 533cm^{-1}. MS (EI, 70 eV): m/z (%) = 242 $[M]^+$. EA calcd (%) for $C_{13}H_{10}N_2O_3$ (242.07): calcd. C 64.46, H 4.16, N 11.56; found C 64.45, H 4.13, N 11.55.

3-(4-Chlorophenyl)-5-butynylisoxazole (4d). ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C): δ = 2.04 (s, 1H), 2.64 (t, 2H, J = 7.2 Hz), 3.02 (t, 2H, J = 7.2 Hz), 6.37 (s, 1H), 6.99 (d, 2H, J = 6.7 Hz, Ar-H), 7.54 (d, 2H, J = 6.7 Hz, Ar-H) ppm. IR (KBr): = 3281, 2964, 2937,

2837, 1606, 1524, 1460, 1430, 1253, 1176, 1052, 840, 792, 659, 533cm^{-1}. MS (EI, 70 eV): *m*/*z* (%) = 231 [M]$^{+}$. EA calcd (%) for $C_{13}H_{10}ClNO$ (231.05): calcd. C 67.40, H 4.35, N 6.05; found C 67.38, H 4.33, N 6.05.

3-phenyl-5-butynylisoxazole (4e). ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C): δ = 2.04 (s, 1H), 2.64 (t, 2H, *J* = 7.2 Hz), 3.02 (t, 2H, *J* = 7.2 Hz), 6.37 (s, 1H), 6.84-7.10 (m, 5H, Ar-H) ppm. IR (KBr): = 3281, 2964, 2937, 2837, 1608, 1524, 1462, 1430, 1253, 1176, 1052, 840, 792, 659, 533cm^{-1}. MS (EI, 70 eV): *m*/*z* (%) = 197 [M]$^{+}$. EA calcd (%) for $C_{13}H_{11}NO$ (197.08): calcd. C 79.16, H 5.62, N 7.10; found C 79.12, H 5.60, N 7.08.

3-(4-Nitrophenyl)-5-butynylisoxazole (4f). ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C): δ = 2.04 (s, 1H), 2.64 (t, 2H, *J* = 7.2 Hz), 3.02 (t, 2H, *J* = 7.2 Hz), 6.37 (s, 1H), 6.94 (d, 2H, *J* = 8.7 Hz, Ar-H), 8.101 (d, 2H, *J* = 8.7 Hz, Ar-H) ppm. IR (KBr): = 3285, 2964, 2937, 2837, 1608, 1520, 1462, 1434, 1250, 1176, 1052, 840, 792, 659, 533cm^{-1}. MS (EI, 70 eV): *m*/*z* (%) = 242 [M]$^{+}$. EA calcd (%) for $C_{13}H_{10}N_2O_3$ (242.07): calcd. C 64.46, H 4.16, N 11.56; found C 64.45, H 4.13, N 11.54.

3-(4-Flurophenyl)-5-butynylisoxazole (4g). ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C): δ = 2.04 (s, 1H), 2.64 (t, 2H, *J* = 7.2 Hz), 3.02 (t, 2H, *J* = 7.2 Hz), 6.37 (s, 1H), 6.94 (d, 2H, *J* = 8.1 Hz, Ar-H), 7.56 (d, 2H, *J* = 8.1 Hz, Ar-H) ppm. IR (KBr): = 3277, 2964, 2937, 2832, 1606, 1460, 1430, 1253, 1176, 1052, 842, 796, 659, 530cm^{-1}. MS (EI, 70 eV): *m*/*z* (%) = 215 [M]$^{+}$. EA calcd (%) for $C_{13}H_{10}FNO$ (215.07): calcd. C 72.55, H 4.68, N 6.51; found C 72.52, H 4.66, N 6.50.

3-(2,3-DiMethoxyphenyl)-5-butynylisoxazole (4h). ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C): δ = 2.04 (s, 1H), 2.64 (t, 2H, *J* = 7.2 Hz), 3.02 (t, 2H, *J* = 7.2 Hz), 3.62 (s, 3H), 3.85 (s, 3H), 6.38 (s, 1H), 6.99-7.10 (m, 3H, Ar-H) ppm. IR (KBr): = 3281, 2960, 2937, 2837, 1612, 1528, 1462, 1432, 1253, 1176, 1052, 845, 784, 655 cm^{-1}. MS (EI, 70 eV): *m*/*z* (%) = 257 [M]$^{+}$. EA calcd (%) for $C_{15}H_{15}NO_3$ (257.11): calcd. C 70.02, H 5.88, N 5.44; found C 69.96, H 5.86, N 5.42.

3-(2-Naphthyl)-5-butynylisoxazole (4i). ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C): δ = 2.04 (s, 1H), 2.64 (t, 2H, J = 7.2 Hz), 3.02 (t, 2H, J = 7.2 Hz), 6.35 (s, 1H), 7.21-7.56 (m, 7H, Ar-H) ppm. IR (KBr): = 3280, 2964, 2937, 2834, 1608, 1524, 1462, 1430, 1253, 1176, 1052, 792, 644, 530 cm^{-1}. MS (EI, 70 eV): m/z (%) = 247 $[M]^+$. EA calcd (%) for $C_{17}H_{13}NO$ (247.10): calcd. C 82.57, H 5.30, N 5.66; found C 82.54, H 5.28, N 5.64.

3-(4-Cyanophenyl)-5-butynylisoxazole (4j). ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C): δ = 2.06 (s, 1H), 2.65 (t, 2H, J = 7.2 Hz), 3.04 (t, 2H, J = 7.2 Hz), 6.37 (s, 1H), 7.12 (d, 2H, J = 6.7 Hz, Ar-H), 7.36 (d, 2H, J = 6.7 Hz, Ar-H) ppm. IR (KBr): = 3275, 2964, 2937, 2832, 1606, 1454, 1432, 1253, 1176, 1056, 842, 796, 664, 530cm^{-1}. MS (EI, 70 eV): m/z (%) = 222 $[M]^+$. EA calcd (%) for $C_{14}H_{10}N_2O$ (222.08): calcd. C 75.66, H 4.54, N 12.60; found C 75.64, H 4.52, N 12.58.

3-(3-Bromo-4-methoxyphenyl)-5-butynylisoxazole (4k). ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C): δ = 2.04 (s, 1H), 2.64 (t, 2H, J = 7.2 Hz), 3.02 (t, 2H, J = 7.2 Hz), 6.38 (s, 1H), 6.82 (d, 1H, Ar-H), 7.56 (d, 1H, Ar-H), 7.68 (s, 1H, Ar-H) ppm. IR (KBr): = 3275, 2965, 2932, 2832, 1604, 1460, 1430, 1255, 1176, 1055, 842, 796, 650 cm^{-1}. MS (EI, 70 eV): m/z (%) = 305 $[M]^+$. EA calcd (%) for $C_{14}H_{12}BrNO_2$ (305.01): calcd. C 54.92, H 3.95, N 4.58; found C 54.90, H 3.92, N 4.56.

3-(4-Hydroxyphenyl)-5-butynylisoxazole (4l). ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C): δ = 2.04 (s, 1H), 2.64 (t, 2H, J = 7.2 Hz), 3.02 (t, 2H, J = 7.2 Hz), 5.12 (s, 1H), 6.37 (s, 1H), 6.83 (d, 2H, J = 7.2 Hz, Ar-H), 7.44 (d, 2H, J = 7.2 Hz, Ar-H) ppm. IR (KBr): = 3524, 3270, 2962, 2930, 2832, 1610, 1462, 1430, 1253, 1176, 1052, 845, 795, 659 cm^{-1}. MS (EI, 70 eV): m/z (%) = 213 $[M]^+$. EA calcd (%) for $C_{13}H_{11}NO_2$ (213.08): calcd. C 73.22, H 5.20, N 6.57; found C 73.20, H 5.18, N 6.53.

General procedure for Sonogashira cross-coupling. In a typical procedure, to a suspension of $Pd(OAc)_2$ (0.03 mmol) and Ag(I)-NHC (NHC = 1,3-Di-*tert*-butylimidazolin-2-ylidene (i)) (0.03 mmol) was dissolved in DCM (10 mL). Then, the indicated amount of above dichloromethane solution was added to a mixture of aryl

iodides (1 mmol), 5-butynylisoxazoles (1.2 mmol), pyrrolidine (3 mmol, 3 equiv), and DCM (15 mL). Then, the mixture was stirred at room temperature for 2 h to form the desired coupled products. Complete consumption of starting material was judged by TLC and GC analysis. After, the mixture was filtered and evaporated, the residue was purified by column chromatography (silica gel, 60-120 mesh, eluent; n-hexane/EtOAc (8:2) gradient) to afford the desired coupled product (**6a-o**).

Spectral Data of Compounds (6a-o):

3-(4-methoxyphenyl)-5-(4-p-tolylbut-3-ynyl)isoxazole (6a). ^{1}H NMR (400 MHz, $CDCl_3$, 25 °C): δ = 2.45 (s, 3H), 2.66 (t, 2 H, *J*=6.9 Hz), 3.08 (t, 2 H, *J*=6.9 Hz), 3.82 (s, 3H), 6.43 (s, 1 H), 6.82 (d, 2H, Ar-H), 7.20 (d, 2H, Ar-H), 7.32-7.39 (m, 4H, Ar-H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C): δ = 17.40, 21.82, 31.24, 36.92, 84.94, 86.82, 93.15, 114.52, 119.34, 119.82, 126.30, 129.81, 131.42, 138.92, 157.60, 159.45, 172.50 ppm. IR (KBr): = 3080, 2964, 2937, 2837, 1980, 1866, 1612, 1524, 1462, 1273, 1256, 1166, 1052, 948, 840, 792, 788, 659, 533cm^{-1}. MS (EI, 70 eV): *m*/*z* (%) = 318 $[M + H]^+$. EA calcd (%) for $C_{21}H_{19}NO_2$ (317.03): calcd. C 79.47, H 6.03, N 4.41; found C 79.45, H 6.01, N 4.40.

3-(4-methoxyphenyl)-5-(4-(4-methoxyphenyl)but-3-ynyl)isoxazole (6b). ^{1}H NMR (400 MHz, $CDCl_3$, 25 °C): δ = 2.66 (t, 2 H, *J*=6.9 Hz), 3.08 (t, 2 H, *J*=6.9 Hz), 3.82 (s, 6H), 6.43 (s, 1H), 6.75-6.82 (m, 4H, Ar-H), 7.32-7.39 (m, 4H, Ar-H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C): δ = 17.40, 31.22, 36.19, 54.23, 85.21, 86.81, 92.32, 113.52, 114.54, 114.24, 120.21, 127.30, 134.64, 158.34, 159.52, 160.15, 172.34 ppm. IR (KBr): = 3080, 2964, 2937, 2837, 1612, 1524, 1462, 1273, 1256, 1166, 1052, 948, 840, 792, 788, 659, 533cm^{-1}. MS (EI, 70 eV): *m*/*z* (%) = 334 $[M + H]^+$. EA calcd (%) for $C_{21}H_{19}NO_3$ (333.11): calcd. C 75.66, H 5.74, N 4.20; found C 75.64, H 5.73, N 4.17.

3-(4-methoxyphenyl)-5-(4-phenylbut-3-ynyl)isoxazole (6c). ^{1}H NMR (400 MHz, $CDCl_3$, 25 °C): δ = 2.66 (t, 2 H, *J*=6.9 Hz), 3.08 (t, 2 H, *J*=6.9 Hz), 3.82 (s, 3H), 6.43 (s, 1H), 6.85 (d, 2 H, Ar-H), 7.05-7.12 (m, 5H, Ar-H), 7.61 (d, 2H, Ar-H) ppm. ^{13}C NMR

(100 MHz, $CDCl_3$, 25 °C): δ = 17.45, 28.33, 39.20, 72.61, 89.15, 99.42, 114.38, 121.70, 122.42, 126.44, 129.86, 131.82, 159.22, 160.92, 162.10, 171.44 ppm. IR (KBr): = 3080, 2964, 2937, 2837, 1612, 1524, 1462, 1298, 1256, 1176, 1052, 948, 840, 792, 788, 659, 533cm^{-1}. MS (EI, 70 eV): *m*/*z* (%) = 304 $[M + H]^+$. EA calcd (%) for $C_{20}H_{17}NO_2$ (303.00): calcd. C 79.19, H 5.65, N 4.62; found C 79.17, H 5.64, N 4.61.

3-(4-methoxyphenyl)-5-(4-(4-nitrophenyl)but-3-ynyl)isoxazole (6d). ^{1}H NMR (400 MHz, $CDCl_3$, 25 °C): δ = 2.66 (t, 2 H, *J*=6.9 Hz), 3.08 (t, 2 H, *J*=6.9 Hz), 3.82 (s, 3H), 6.43 (s, 1H), 6.82 (d, 2H, Ar-H), 7.32-7.39 (m, 4H, Ar-H), 8.26 (d, 2H, Ar-H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C): δ = 17.44, 31.42, 37.82, 85.15, 87.23, 93.60, 114.32, 120.14, 123.60, 126.84, 132.42, 132.80, 148.24, 159.35, 159.60, 173.42 ppm. IR (KBr): = 3102, 3080, 2964, 2917, 2832, 1612, 1520, 1462, 1273, 1256, 1162, 1054, 948, 842, 792, 788, 659, 533 cm^{-1}. MS (EI, 70 eV): *m*/*z* (%) = 349 $[M + H]^+$. EA calcd (%) for $C_{20}H_{16}N_2O_4$ (348.12): calcd. C 68.96, H 4.63, N 8.04; found C 68.94, H 4.62, N 8.01.

1-(4-(4-(3-(4-methoxyphenyl)isoxazol-5-yl)but-1-ynyl)phenyl)ethanone (6e). ^{1}H NMR (400 MHz, $CDCl_3$, 25 °C): δ = 2.56 (s, 3H), 2.66 (t, 2 H, *J*=6.9 Hz), 3.08 (t, 2 H, *J*=6.9 Hz), 3.82 (s, 3H), 6.43 (s, 1H), 6.82 (d, 2H, Ar-H), 7.32-7.39 (m, 4H, Ar-H), 7.89 (d, 2H, Ar-H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C): δ = 17.45, 24.82, 31.20, 37.31, 85.24, 88.65, 93.60, 114.32, 120.41, 124.20, 126.32, 129.84, 132.42, 137.20, 159.54, 160.32, 172.40, 195.82 ppm. IR (KBr): = 3080, 2964, 2937, 2837, 1696, 1524, 1462, 1273, 1256, 1166, 1052, 948, 840, 792, 788, 659, 533cm^{-1}. MS (EI, 70 eV): *m*/*z* (%) = 346 $[M + H]^+$. EA calcd (%) for $C_{22}H_{19}NO_3$ (345.02): calcd. C 76.50, H 5.54, N 4.06; found C 76.48, H 5.52, N 4.05.

3-phenyl-5-(4-*p*-tolylbut-3-ynyl)isoxazole (6f). ^{1}H NMR (400 MHz, $CDCl_3$, 25 °C): δ = 2.45 (s, 3H), 2.66 (t, 2 H, *J*=6.9 Hz), 3.08 (t, 2 H, *J*=6.9 Hz), 6.43 (s, 1H), 7.13 (d, 2H, Ar-H), 7.24-7.32 (m, 5H, Ar-H), 7.61 (d, 2H, Ar-H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C): δ = 17.32, 22.41, 30.80, 85.26, 88.22, 93.21, 119.24, 123.81, 125.16, 125.28, 126.22, 130.27, 131.48, 140.52, 159.14, 172.53 ppm. IR (KBr): = 3080, 2975, 2965, 2938, 2918, 1605, 1527, 1495, 1466, 1298, 1256, 1176, 1052, 948, 840, 792, 788, 659,

533cm^{-1}. MS (EI, 70 eV): *m*/*z* (%) = 288 [M + H]$^{+}$. EA calcd (%) for $C_{20}H_{17}NO$ (287.09): calcd. C 83.59, H 5.96, N 4.87; found C 83.57, H 5.92, N 4.85.

5-(4-(4-methoxyphenyl)but-3-ynyl)-3-phenylisoxazole (6g). ^{1}H NMR (400 MHz, $CDCl_3$, 25 °C): δ = 2.66 (t, 2 H, *J*=6.9 Hz), 3.08 (t, 2 H, *J*=6.9 Hz), 3.82 (s, 3H), 6.43 (s, 1H), 6.84 (d, 2H, Ar-H), 7.24-7.32 (m, 5H, Ar-H), 7.61 (d, 2H, Ar-H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C): δ = 17.34, 29.62, 55.60, 85.22, 87.62, 93.20, 113.64, 115.82, 123.24, 125.42, 125.81, 126.35, 135.84, 159.20, 162.21, 173.10 ppm. IR (KBr): = 3082, 2964, 2937, 2837, 1610, 1527, 1459, 1422, 1298, 1256, 1176, 1052, 948, 840, 792, 788, 659, 533cm^{-1}. MS (EI, 70 eV): *m*/*z* (%) = 304 [M + H]$^{+}$. EA calcd (%) for $C_{20}H_{17}NO_2$ (303.00): calcd. C 79.19, H 5.65, N 4.62; found C 79.17, H 5.64, N 4.61.

3-phenyl-5-(4-phenylbut-3-ynyl)isoxazole (6h). ^{1}H NMR (400 MHz, $CDCl_3$, 25 °C): δ = 2.66 (t, 2 H, *J*=6.9 Hz), 3.08 (t, 2 H, *J*=6.9 Hz), 6.43 (s, 1 H), 6.99-7.12 (m, 4H, Ar-H), 7.31-7.39 (m, 2H, Ar-H) 7.42-7.51 (m, 4H, Ar-H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C): δ = 17.42, 29.60, 85.24, 87.65, 93.72, 123.40, 123.82, 124.12, 125.27, 126.32, 128.50, 128.16, 131.28, 159.52, 171.82 ppm. IR (KBr): = 3082, 2964, 2937, 2837, 1610, 1527, 1459, 1422, 948, 840, 792, 788, 659, 533cm^{-1}. MS (EI, 70 eV): *m*/*z* (%) = 274 [M + H]$^{+}$. EA calcd (%) for $C_{19}H_{15}NO$ (273.02): calcd. C 83.49, H 5.53, N 5.12; found C 83.48, H 5.51, N 5.11.

5-(4-(4-nitrophenyl)but-3-ynyl)-3-phenylisoxazole (6i). ^{1}H NMR (400 MHz, $CDCl_3$, 25 °C): δ = 2.66 (t, 2 H, *J*=6.9 Hz), 3.08 (t, 2 H, *J*=6.9 Hz), 6.43 (s, 1 H), 7.24-7.32 (m, 5H, Ar-H), 7.61 (d, 2H, Ar-H) 8.22 (d, 2H, Ar-H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C): δ = 17.34, 31.40, 85.12, 86.72, 93.51, 122.80, 123.64, 124.34, 125.70, 126.44, 131.80, 132.32, 148.54, 159.22, 173.24 ppm. IR (KBr): = 3100, 3080, 2975, 2965, 1605, 1523, 1495, 1347, 1298, 1256, 1176, 1052, 948, 854, 792, 788, 659, 533cm^{-1}. MS (EI, 70 eV): *m*/*z* (%) = 319 [M + H]$^{+}$. EA calcd (%) for $C_{19}H_{14}N_2O_3$ (318.10): calcd. C 71.69, H 4.43, N 8.80; found C 71.65, H 4.41, N 8.78.

1-(4-(4-(3-phenylisoxazol-5-yl)but-1-ynyl)phenyl)ethanone (6j). ^{1}H NMR (400 MHz, $CDCl_3$, 25 °C): δ = 2.52 (s, 3H), 2.66 (t, 2 H, *J*=6.9 Hz), 3.08 (t, 2 H, *J*=6.9 Hz), 6.43 (s, 1 H), 7.24-7.32 (m, 5H, Ar-H), 7.61 (d, 2H, Ar-H) 7.92 (d, 2H, Ar-H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C): δ = 17.42, 26.41, 31.20, 86.16, 87.92, 92.32, 123.80, 124.64, 125.42, 125.81, 126.20, 129.32, 131.19, 137.15, 159.15, 172.61, 193.30 ppm. IR (KBr): = 3080, 2975, 2963, 2918, 1697, 1605, 1527, 1495, 1466, 1298, 1256, 1176, 1050, 946, 840, 792, 788, 659, 533cm^{-1}. MS (EI, 70 eV): *m*/*z* (%) = 316 $[M + H]^+$. EA calcd (%) for $C_{21}H_{17}NO_2$ (315.03): calcd. C 79.98, H 5.43, N 4.44; found C 79.97, H 5.42, N 4.41.

3-(4-nitrophenyl)-5-(4-p-tolylbut-3-ynyl)isoxazole (6k). ^{1}H NMR (400 MHz, $CDCl_3$, 25 °C): δ = 2.43 (s, 3H), 2.66 (t, 2 H, *J*=6.9 Hz), 3.08 (t, 2 H, *J*=6.9 Hz), 6.43 (s, 1H), 7.12 (d, 2H, Ar-H), 7.32-7.39 (m, 4H, Ar-H), 8.25 (d, 2H, Ar-H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C): δ = 17.42, 21.80, 31.22, 85.24, 87.10, 93.12, 113.84, 115.20, 126.31, 127.20, 132.47, 134.02, 148.45, 158.13, 160.21, 172.18 ppm. IR (KBr): = 3100, 3080, 2964, 2917, 2832, 1615, 1521, 1462, 1273, 1254, 1162, 1054, 936, 842, 792, 788, 659, 538cm^{-1}. MS (EI, 70 eV): *m*/*z* (%) = 333 $[M + H]^+$. EA calcd (%) for $C_{20}H_{16}N_2O_3$ (332.10): calcd. C 72.28, H 4.85, N 8.43; found C 72.25, H 4.84, N 8.41.

5-(4-(4-methoxyphenyl)but-3-ynyl)-3-(4-nitrophenyl)isoxazole (6l). ^{1}H NMR (400 MHz, $CDCl_3$, 25 °C): δ = 2.66 (t, 2 H, *J*=6.9 Hz), 3.08 (t, 2 H, *J*=6.9 Hz), 3.82 (s, 3H), 6.43 (s, 1H), 6.82 (d, 2H, Ar-H), 7.32-7.39 (m, 4H, Ar-H), 8.25 (d, 2H, Ar-H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C): δ = 17.42, 31.20, 54.64, 85.26, 86.81, 93.20, 113.18, 115.42, 126.53, 127.10, 132.70, 134.12, 148.24, 158.30, 160.21, 172.84 ppm. IR (KBr): = 3100, 3080, 2964, 2917, 2832, 1613, 1525, 1462, 1273, 1254, 1162, 1054, 936, 842, 790, 788, 659, 533cm^{-1}. MS (EI, 70 eV): *m*/*z* (%) = 349 $[M + H]^+$. EA calcd (%) for $C_{20}H_{16}N_2O_4$ (348.12): calcd. C 68.96, H 4.63, N 8.04; found C 68.94, H 4.62, N 8.01.

3-(4-nitrophenyl)-5-(4-phenylbut-3-ynyl)isoxazole (6m). ^{1}H NMR (400 MHz, $CDCl_3$, 25 °C): δ = 2.66 (t, 2 H, *J*=6.9 Hz), 3.08 (t, 2 H, *J*=6.9 Hz), 6.43 (s, 1H), 7.24-7.32 (m, 5H, Ar-H), 7.61 (d, 2H, Ar-H), 8.24 (d, 2H, Ar-H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C): δ = 17.42, 31.22, 85.24, 88.61, 93.10, 124.24, 126.35, 127.83, 128.42, 128.62,

129.80, 132.40, 149.52, 160.32, 172.40 ppm. IR (KBr): = 3108, 3082, 2964, 2937, 1608, 1527, 1459, 1298, 1256, 1176, 1052, 948, 834, 790, 782, 659, 533cm^{-1}. MS (EI, 70 eV): *m*/*z* (%) = 319 [M + H]$^{+}$. EA calcd (%) for $C_{19}H_{14}N_2O_3$ (318.10): calcd. C 71.69, H 4.43, N 8.80; found C 71.65, H 4.41, N 8.78.

3-(4-nitrophenyl)-5-(4-(4-nitrophenyl)but-3-ynyl)isoxazole (6n). ^{1}H NMR (400 MHz, $CDCl_3$, 25 °C): δ = 2.66 (t, 2 H, *J*=6.9 Hz), 3.08 (t, 2 H, *J*=6.9 Hz), 6.43 (s, 1H), 7.78-7.84 (m, 4H, Ar-H), 8.30-8.39 (m, 4H, Ar-H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C): δ = 17.44, 31.20, 85.25, 87.10, 93.21, 123.18, 126.30, 127.10, 129.82, 130.22, 132.72, 148.35, 148.92, 158.34, 172.81 ppm. IR (KBr): = 3100, 3080, 2964, 2917, 2832, 1521, 1462, 1273, 1236, 1054, 936, 842, 790, 788, 652, 525cm^{-1}. MS (EI, 70 eV): *m*/*z* (%) = 364 [M + H]$^{+}$. EA calcd (%) for $C_{19}H_{13}N_3O_5$ (363.05): calcd. C 62.81, H 3.61, N 11.57; found C 62.79, H 3.58, N 11.56.

1-(4-(4-(3-(4-nitrophenyl)isoxazol-5-yl)but-1-ynyl)phenyl)ethanone (6o). ^{1}H NMR (400 MHz, $CDCl_3$, 25 °C): δ = 2.51 (s, 3H), 2.66 (t, 2 H, *J*=6.9 Hz), 3.08 (t, 2 H, *J*=6.9 Hz), 6.43 (s, 1H), 7.35-7.44 (m, 4H, Ar-H), 7.64 (d, 2H, Ar-H), 8.27 (d, 2H, Ar-H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C): δ = 17.42, 24.81, 31.20, 85.24, 88.62, 93.65, 124.34, 126.42, 127.20, 129.84, 130.62, 132.40, 137.21, 149.14, 158.17, 172.42, 194.82 ppm. IR (KBr): = 3100, 3080, 2960, 2917, 2832, 1694, 1521, 1460, 1273, 1236, 1050, 930, 842, 790, 768, 652, 542 cm^{-1}. MS (EI, 70 eV): *m*/*z* (%) = 361 [M + H]$^{+}$. EA calcd (%) for $C_{21}H_{16}N_2O_4$ (360.02): calcd. C 69.99, H 4.48, N 7.77; found C 69.98, H 4.46, N 7.75.