Phosphoramidite Accelerated Copper (I)-Catalyzed [3+2] Cycloadditions of Azides and Alkynes

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Supplementary Information

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General Information

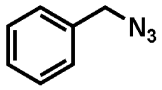
All reactions were carried out in oven dried glassware. $CuSO_4 \cdot 5H_2O$, sodium ascorbate and dimethyl sulfoxide were purchased from Aldrich and used as received. ^{1}H-, ^{13}C-, and ^{19}F-NMR were recorded on a Varian AMX400 (400, 100.59 MHz, and 200 MHz respectively) using $CDCl_3$ as solvent unless otherwise indicated. Chemical shift values are reported in ppm with the solvent resonance as the internal standard ($CHCl_3$: δ 7.26 for ^{1}H and δ 77.0 for ^{13}C). Data are reported as follows: chemical shifts, multiplicity (s=singlet, d=doublet, t=triplet, q=quartet, dd=doublet of doublets, dt=doublet of triplets, td=triplet of doublets, m=multiplet, br=broad), coupling constants (Hz), and integration. Flash chromatography was performed on silica gel. All reactions were monitored by thin layer chromotography on Merck F-254 silica gel plates or by ^{1}H NMR . Visualization of the TLC plates was performed with $KMnO_4$ staining reagent and UV (254 nm). Conversion of reactions was determined by GC-MS (GC HP6890, MS HP5973) with an HP5 column (Agilent Technologies, Palo Alto, CA) or by ^{1}H NMR. Mass spectra were recorded on an AEI-MS-902 mass spectrometer by EI (70 eV) measurements. Melting points are uncorrected. Phosphoramidites were synthesized as described in the literature (1). ^{1}H and ^{13}C NMR data is provided for all synthesized compounds. Spectra were in accordance with published experimental data and references are provided for known compounds. HRMS mass data is provided for all new compounds.

Safety

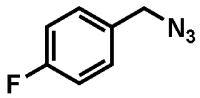
Working with azides should always be done carefully. Organic azides, particularly those of low molecular weight, or with high nitrogen content, are potentially explosive. Heat, light and pressure can cause decomposition of the azides. Furthermore, the azide ion is toxic, and sodium azide should always be handled with gloves. Heavy metal azides are particularly unstable, and may explode if heated or shaken. Any experiment in which azides are to be heated in the presence of copper should involve the use of a blast shield.

Experimental Procedure for the Preparation of Azides

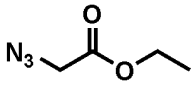
General Procedure A: To a stirred solution of the corresponding bromide (1.0 eq) in a 50 mL water/acetone mixture (1:4) was added NaN_3 (1.5 eq).The resulting suspension was stirred at room temperature for 24 hours. DCM was added to the mixture and the organic layer was separated. The aqueous layer was extracted with 3 x 10 mL aliquots of DCM and the combined organic layers were dried over $MgSO_4$. Solvent was removed under reduced pressure, and the azide was sufficiently pure to use without further work up. In the case of the synthesis of 1-azidooctane, DMSO was used as solvent and the reaction was quenched with water.



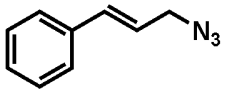
Benzyl azide[2]. Yellow oil. From 2.5 mmol benzyl bromide, 100 % yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.25-7.43 (m, 5H), 4.35 (s, 2H); ^{13}C NMR (100.59 MHz, $CDCl_3$): δ 135.5, 129.3, 128.3, 128.2, 54.8.



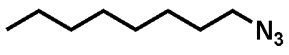
1-(Azidomethyl)-4-fluorobenzene[2]. Pale yellow oil. From 2.5 mmol 1-(bromomethyl)-4-fluorobenzene, 99 % yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.27-7.39 (m, 2H), 7.00-7.11 (m, 2H), 4.30 (s, 2H); ^{13}C NMR (100.59 MHz, $CDCl_3$): δ 162.5 (d, *J*= 328.7 Hz), 131.4, 129.9 (d, J= 11.4 Hz), 115.7 (d, *J*= 27.6 Hz), 54.0; ^{19}F NMR (200 MHz, $CDCl_3$): δ -112.3.



Ethyl 2-azidoacetate[3]. Colorless oil. From 2.5 mmol ethyl bromoacetate, 100% yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 4.15 (q, *J*= 6.8 Hz, 2H), 3.77 (d, *J*= 1.2 Hz, 2H), 1.20 (td, *J*= 7.2, 1.2 Hz, 3H). ^{13}C NMR (100.59 MHz, $CDCl_3$): δ 168.1, 61.5, 50.0, 13.8.

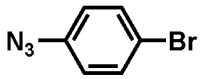


Cinnamyl azide. Colorless oil. From 2.5 mmol cinnamyl bromide, 97 % yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.31-7.45 (m, 5H), 6.67 (d, *J*= 16 Hz, 1H), 6.26 (dt, *J*= 16.0, 6.4 Hz, 1H), 3.95 (d, *J*= 6.4 Hz, 2H); ^{13}C NMR (100.59 MHz, $CDCl_3$): δ 135.7, 134.5, 128.7, 128.2, 126.7, 122.4, 53.0; HRMS (EI) calcd for $C_9H_9N_3$ 159.0796, found 159.0790.

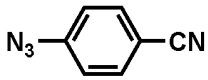


1-Azidooctane[2]. Clear yellow oil. From 3.0 mmol of 1-bromooctane, 83 % yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.21 (t, *J*= 6.8 Hz, 2H), 1.56 (q, *J*= 6.8 Hz, 2H), 1.24-1.34 (m, 10 H), 0.85 (t, *J*= 6.4 Hz, 3H); ^{13}C NMR (100.59 MHz, $CDCl_3$): δ 51.5, 31.8, 29.2, 29.2, 28.9, 26.8, 22.7, 14.1.

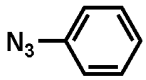
General Procedure B: To a stirred solution of the corresponding aromatic amine (1.0 eq) in acetonitrile (0.5 M) at 0 ºC was added tert-butyl nitrite (1.5 eq), followed by azidotrimethylsilane (1.2 eq) in a dropwise fashion. The roundbottom flask was then removed from the ice bath and allowed to warm to room temerature and stir for a further 2 h. Solvent was removed under reduced pressure, and the azides were purified by column chromatography (pentane/ether).



1-azido-4-bromobenzene[4]. Yellow-orange solid. From 2.0 mmol 4-bromoaniline, 91% yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.44 (d, *J*= 6.8 Hz, 2H), 6.89 (d, *J*= 8.8 Hz, 2H); ^{13}C NMR (100.59 MHz, $CDCl_3$): δ 139.1, 132.7, 120.5, 117.7.

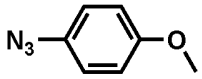


4-Azidobenzonitrile[5]. Orange solid. From 3.0 mmol 4-aminobenzonitrile, 83% yield. ^{1}H NMR (400 MHz, $CDCl_3$): 7.58 (td, *J*= 9.6, 2.0 Hz, 2H), 7.05 (td, *J*= 9.6 Hz, 0.8 Hz, 2H); ^{13}C NMR (100.59 MHz, $CDCl_3$): δ 144.5, 133.5, 119.5, 118.2, 108.0.



Azidobenzene[5]. Pale yellow oil. From 3.0 mmol aniline, 65 % yield. ^{1}H NMR (400 MHz, $CDCl_3$): 7.36-7.41 (m, 2H), 7.15-7.20 (m, 1H), 7.05-7.08 (m, 2H); ^{13}C NMR (100.59 MHz, $CDCl_3$): δ 140.0, 129.7, 124.8, 118.9.

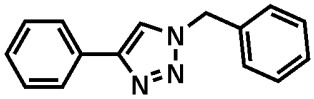
General Procedure C: 1-azido-4-methoxybenzene was prepared according to literature procedures[4].



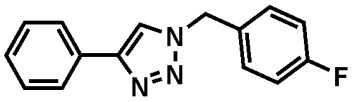
1-azido-4-methoxybenzene[4]. Dark orange solid. From 2.0 mmol iodoanisole, 57 % yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 6.96 (d, *J*= 8.8 Hz, 2H), 6.89 (d, *J*= 8.4 Hz, 2H), 3.79 (s, 3H); ^{13}C NMR (100.59 MHz, $CDCl_3$): δ 156.9, 132.2, 119.9, 115.0, 55.4.

General Procedure for the Synthesis of 1,4-Disubstituted Triazoles: In a sample vial, 5.6 x 10^{-3} mmol of $CuSO_4 \cdot 5H_2O$ and 2.8 x 10^{-2} mmol sodium ascorbate were dissolved in 1.2 mL distilled water. To the sample vial was added 6.2 x 10^{-3} mmol of MonoPhos in 0.4 mL DMSO. The resulting solution was vigorously stirred for 15 min. The solution was then added to a 25 mL oven-dried roundbottom flask containing 0.56 mmol of azide and 1.12 mmol of alkyne in 3 mL of a DMSO:H_2O mixture (1:3). The roundbottom flask was sealed and the reaction mixture was vigorously stirred. Reaction progress was monitored by ^{1}H NMR. Upon reaction completion, 10 mL of H_2O was added to the reaction mixture. For solid products, the reaction mixture was placed in an ice bath and

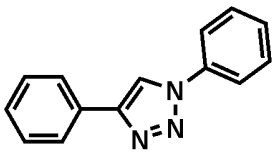
the precipitated solid product was filtered and washed with 3 x 5 mL of cold water. For oil products, the resulting reaction mixture was extracted with 3 x 15 mL of dichloromethane. The organic layers were combined and dried over $MgSO_4$ and the solvent was removed by vacuum evaporation. Crude oils were purified using silica gel chromatography (pentane:ether). The phosphoramidite ligands were also recovered from the mixture by column chromatography.



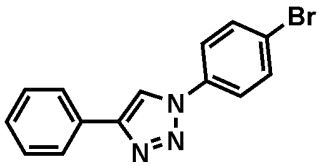
1-benzyl-4-phenyl-1*H*-1,2,3-triazole. White solid: mp 128-130 ºC. 91 % yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.80 (d, *J*= 7.2 Hz, 2H), 7.66 (s, 1H), 7.32-7.42 (m, 8H), 5.58 (s, 2H); ^{13}C NMR (100.59 MHz, $CDCl_3$): δ 148.0, 135.0, 130.7, 129.1, 128.9, 128.7, 128.2, 128.1, 125.7, 120.1, 41.0. HRMS (EI) calcd for $C_{15}H_{13}N_3$ 235.1109, found 235.1098.



1-(4-fluorobenzyl)-4-phenyl-1*H*-1,2,3-triazole[6]. White solid: mp 129-131 ºC. 84 % yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.78 (d, *J*= 7.2 Hz, 2H), 7.67 (s, 1H), 7.37 (t, *J*= 7.2 Hz, 2H), 7.25-7.28 (m, 3H), 7.03 (t, *J*= 8.8 Hz, 2H), 5.49 (s, 2H); ^{13}C NMR (100.59 MHz, $CDCl_3$): δ 163.0 (d, *J*= 248.0 Hz) , 148.5, 130.8 (d, *J*= 3.0 Hz), 130.6, 130.2 (d, *J*= 9.0 Hz), 129.0, 128.4, 125.9, 119.7, 116.3 (d, *J*= 21.0 Hz), 53.6.

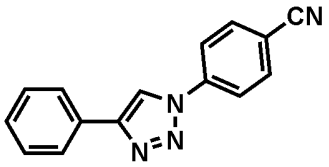


1-(4-diphenyl)-1*H*-1,2,3-triazole[7]. Pale yellow solid: mp183-184 ºC. 88 % yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.20 (s, 1H), 7.92 (dt, *J*= 8.0, 1.6 Hz, 2H), 7.79 (dt, *J*= 8.4 Hz, 1.2 Hz, 2H), 7.55 (tt, *J*= 8.4, 1.6 Hz, 2H), 7.43-7.48 (m, 3H), 7.38 (tt, *J*= 7.6, 0.8 Hz, 1H); ^{13}C NMR (100.59 MHz, $CDCl_3$): δ 148.8, 137.4, 130.6, 130.2, 129.3, 129.2, 128.8, 126.2, 120.9, 118.0.

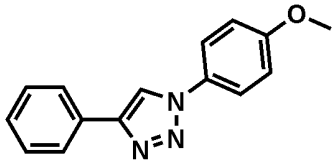


1-(4-bromophenyl)-4-phenyl-1*H*-1,2,3-triazole. White solid: mp 231 ºC. 71 % yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.17 (d, *J*= 3.2 Hz, 1H), 7.91 (dq, *J*= 6.4, 3.2 Hz, 2H), 7.68-7.70 (m, 4H), 7.47 (tt, *J*= 8.0 Hz, 1.6 Hz, 2H), 7.40 (dt, *J*= 8.0, 1.6 Hz, 1H); ^{13}C NMR

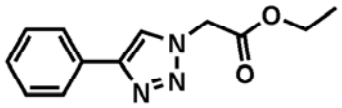
(100.59 MHz, $CDCl_3$): δ 149.1, 133.3, 136.4, 130.4, 129.4, 129.0, 126.3, 122.8, 122.3, 117.7. HRMS (EI) calcd for $C_{14}H_{10}N_3Br$ 300.0131, found 300.0130.



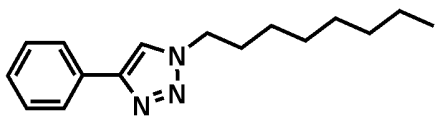
4-(4-phenyl-1*H*-1,2,3-triazol-1-yl)benzonitrile. Pale yellow solid: mp 234 ºC. 81 % yield. ^{1}H NMR (400 MHz, d6-DMSO): δ 9.45 (s, 1H), 8.15 (q, *J*= 8.1 Hz, 4H), 7.93 (d, *J*= 7.8 Hz, 2H), 7.50 (t, *J*= 7.8 Hz, 2H), 7.36-7.41 (m, 1H); ^{13}C NMR (100.59 MHz, d6-DMSO): δ 148.4, 140.2, 135.0, 130.5, 129.8, 129.2, 126.1, 121.0, 120.5, 118.8, 111.7. HRMS (EI) calcd for $C_{15}H_{10}N_4$ 247.0978, found 247.0977.



1-(4-methoxyphenyl)-4-phenyl-1*H*-1,2,3-triazole. Pale yellow solid: mp 160-161 ºC. 80 % yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.11 (s, 1H), 7.90 (dd, *J*= 7.6, 0.8 Hz, 2H), 7.69 (dt, *J*= 9.2, 2.4 Hz, 2H), 7.46 (tt, *J*= 7.6 Hz, 1.2 Hz, 2H), 7.36 (tt, *J*= 7.6, 1.2 Hz, 1H), 7.04 (dt, *J*= 8.4, 2.4 Hz , 2H), 3.88 (s, 3H); ^{13}C NMR (100.59 MHz, $CDCl_3$): δ 160.2, 148.6, 130.9, 130.8, 129.3, 128.7, 126.2, 122.6, 118.2, 115.2, 56.0. HRMS (EI) calcd for $C_{16}H_{15}N_3O$ 265.1215, found 265.1215.

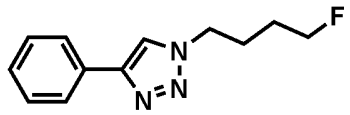


Ethyl 2-(4-phenyl-1*H*-1,2,3-triazol-1-yl)acetate[8]. White solid: mp 99 ºC. 83 % yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.91 (s, 1H), 7.84 (d, *J*= 7.2 Hz, 2H), 7.43 (t, *J*= 7.2 Hz, 2H), 7.34 (t, *J*= 7.2 Hz, 1H), 5.20 (s, 2H), 4.28 (q, *J*= 6.8 Hz, 2H), 1.31 (t, *J*= 6.8 Hz, 3H); ^{13}C NMR (100.59 MHz, $CDCl_3$): δ 166.5, 148.4, 130.6, 129.1, 128.5, 126.0, 121.3, 62.7, 51.2, 14.3.

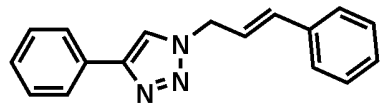


1-octyl-4-phenyl-1*H*-1,2,3-triazole[9]. White solid: mp 74-75 ºC. 99 % yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.78 (d, *J*= 7.2 Hz, 2H), 7.73 (s, 1H), 7.36 (t, *J*= 7.2 Hz, 2H), 7.27 (t, *J*= 7.2 Hz, 1H), 4.32 (t, *J*= 7.2 Hz, 2H), 1.87 (br s, 2H), 1.20-1.27 (m, 10H), 0.82 (t, *J*=

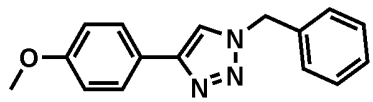
3.2 Hz, 3H) ; ^{13}C NMR (100.59 MHz, $CDCl_3$): δ 147.7, 130.9, 128.9, 128.1, 125.7, 119.7, 50.5, 31.8, 30.4, 29.1, 29.0, 26.6, 22.7, 14.2.



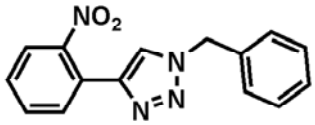
1-(4-fluorobutyl)-4-phenyl-1*H*-1,2,3-triazole. White solid: mp 54-55 ºC. 90 % yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.82 (d, *J*= 10.0 Hz, 2H), 7.76 (s, 1H), 7.42 (t, *J*= 10.0 Hz, 2H), 7.30-7.35 (m, 1H), 4.56 (t, *J*= 7.2 Hz, 1H), 4.38-4.47 (m, 3H), 2.05-2.15 (m, 2H), 1.65-1.82 (m, 2H); ^{13}C NMR (100.59 MHz, $CDCl_3$): δ 148.1, 130.8, 129.1, 128.4, 125.9, 119.7, 83.5 (d, *J*= 330.1 Hz), 50.1, 27.3 (d, *J*= 91.3 Hz), 27.2. HRMS (EI) calcd for $C_{12}H_{14}N_3F$ 219.1172, found 219.1169.



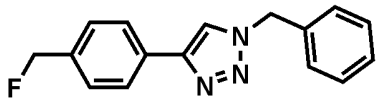
1-cinnamyl-4-phenyl-1*H*-1,2,3-triazole[7]. Pale yellow solid: mp 134 ºC. 96 % yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.85 (s, 1H), 7.82 (d, *J*= 6.8 Hz, 2H), 7.40-7.44 (m, 4H), 7.27-7.37 (m, 4H), 6.70 (d, *J*= 15.6 Hz, 1H), 6.37 (dt, *J*= 16.0 Hz, 6.4 Hz, 1H), 5.17 (d, 6.8 Hz, 2H); ^{13}C NMR (100.59 MHz, $CDCl_3$): δ 148.4, 135.7, 135.6, 130.8, 129.1, 129.0, 128.8, 128.4, 127.0, 126.0, 122.2, 119.6, 52.7.



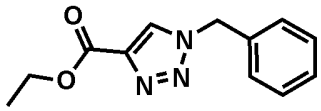
1-benzyl-4-(4-methoxyphenyl)-1*H*-1,2,3-triazole[10]**.** Pale yellow solid: mp 143-144 ºC. 86 % yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.72 (d, *J*= 8.8 Hz, 2H), 7.56 (s, 1H), 7.35-7.75 (m, 3H), 7.29-7.34 (m, 2H), 6.93 (d, *J*= 8.8 Hz, 2H), 5.55 (s, 2H), 3.82 (s, 3H); ^{13}C NMR (100.59 MHz, $CDCl_3$): δ 159.9, 148.4, 135.1, 129.4, 129.0, 128.3, 127.3, 123.6, 119.0, 114.5, 55.5, 54.5.



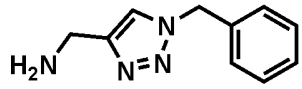
1-benzyl-4-(2-nitrophenyl)-1*H*-1,2,3-triazole. Pale yellow solid: mp 110-112 ºC. 62 % yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.01 (d, *J*= 7.6 Hz, 1H), 7.79 (d, *J*= 8.0 Hz, 1H), 7.73 (s, 1H), 7.63 (t, *J*= 7.6 Hz, 1H), 7.47 (t, *J*= 8.0 Hz, 1H), 7.34-7.40 (m, 3H), 7.28-7.31 (m, 2H), 5.58 (s, 2H); ^{13}C NMR (100.59 MHz, $CDCl_3$): δ 148.4, 142.7, 134.6, 132.8, 131.3, 129.4, 129.2, 129.1, 128.3, 124.9, 124.3, 123.2, 54.6. HRMS (EI) calcd for $C_{15}H_{12}N_4O_2$ 281.1033, found 281.1030.



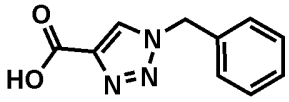
1-benzyl-4-(4-fluoromethyl)phenyl)-1*H*-1,2,3-triazole. White solid: mp 127-130 ºC. 93 % yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.82 (d, *J*= 7.6 Hz, 2H), 7.67 (s, 1H), 7.31-7.42 (m, 7H), 5.58 (s, 2H), 5.38 (d, *J*= 48.0 Hz, 2H); ^{13}C NMR (100.59 MHz, $CDCl_3$): δ 148.0, 136.3 (d, *J*= 16.8 Hz), 134.9, 131.3 (d, *J*= 3.1 Hz), 129.5, 129.1, 128.3, 128.2 (d, *J*= 5.3 Hz), 126.1, 120.0, 84.6 (d, *J*= 166.4 Hz), 54.5. HRMS (EI) calcd for $C_{16}H_{14}N_3F$ 268.1245, found 268.1243.



ethyl 1-benzyl-1*H*-1,2,3-triazole-4-carboxylate[11]. White solid: mp 83-85 ºC. 87 % yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.97 (s, 1H), 7.36 (br s, 3H), 7.25-7.28 (m, 2H), 5.56 (s, 2H), 4.36 (q, *J*= 6.8 Hz, 2H), 1.35 (t, *J*= 6.8 Hz, 3H); ^{13}C NMR (100.59 MHz, $CDCl_3$): δ 160.9, 140.8, 134.0, 129.5, 129.3, 128.5, 127.6, 61.5, 54.7, 14.5.

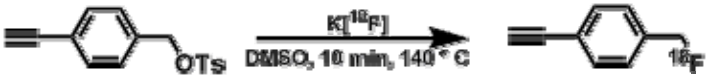


(1-benzyl-1*H*-1,2,3-triazol-4-yl)methanamine. White solid: mp 109-112 ºC. 65 % yield. ^{1}H NMR (400 MHz, DMSO): δ 7.91 (s, 1H), 7.28-7.34 (m, 5H), 5.54 (s, 2H), 4.15 (s, 2H); ^{13}C NMR (100.59 MHz, DMSO): δ 147.0, 136.9, 129.4, 128.7, 123.3, 122.7, 53.4, 40.1. HRMS (EI) calcd for $C_{10}H_{12}N_4$ 189.1135, found 189.1135.



1-benzyl-1*H*-1,2,3-triazole-4-carboxylic acid. White solid: mp 128 ºC. 59 % yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.48 (s, 1H), 7.33-7.38 (m, 5H), 5.64 (s, 2H); ^{13}C NMR (100.59 MHz, $CDCl_3$): δ 162.1, 140.4, 135.2, 128.9, 128.6, 128.3, 128.1, 54.0. HRMS (EI) calcd for $C_{10}H_9N_3O_2$ 204.0768, found 204.0768.

Radiolabelling Procedure



[^{18}F]- fluoride in [^{18}O]-enriched H_2O was captured on a QMA light Sep-Pak cartridge. The [^{18}F]-fluoride was eluted with 4.5 mg of potassium bicarbonate. 20 mg of Kriptofix was subsequently added. Under argon atmosphere, the fluoride was dried three times with 0.5 mL of pure acetonitrile at 130 °C. The dried [^{18}F]-fluoride was then added to 3.0 mg of 4-ethynylbenzyl 4-methylbenzenesulfonate in 0.5 mL dry DMSO. The solution was allowed to react for 10 min at 140 °C. The labeled product was adsorbed on a light C18 Sep-Pak cartridge and eluted with 5 mL pure methanol. The Sep-Pak eluate was then treated with semi-preparative HPLC on a C18-reversed phase column (mobile phase 60/40 methanol/water, retention time=12 min). For conditions of the click reaction, see general procedure. Conversion of the reaction was monitored by radio-TLC.

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