Electronic Supplemental Information (ESI)

Scratching the catalytic surface of mechanochemistry: A multi-component CuAAC reaction using a copper reaction vial

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Electronic Supplementary Material (ESI) for Green Chemistry

General Information

^{1}H NMR spectra were obtained using a Bruker Avance 400MHz spectrometer. Deuterated chloroform was obtained from Cambridge Isotope Laboratories, Inc., Andover MA., and used without further purification. All reagents and were purchased from Acros Organics and used without further purification. Copper ball-bearings were purchased from McMaster-Carr Supply. Copper rods used to make copper vials were purchased from McMaster-Carr Supply. Teflon rods used to make Teflon vials were purchased from McMaster-Carr Supply. Stainless steel rods used to make stainless steel vials were purchased from McMaster-Carr Supply. Ball-milling was carried out in an 8000M SpexCertiprep Mixer/Mill purchased from Spex certiprep. All product yields reported are isolated yields.

Typical reaction procedure for the CuAAC between alkyl azide and an acetylene derivative:

Phenyl acetylene (0.172g, 1.6mmol), and benzyl azide (0.250g, 1.5mmol), were added to a custom-made 2.0 x 0.5 inch screw capped copper vial inserted with a perfluoroelastomer O-ring, along with a 0.250 inch copper ball bearing ball bearing. After placement of the vial in a Spex Certiprep 8000M mixer/mill, the reagents were ball milled for 15 minutes. The resulting mixture was removed from the vial with ethyl acetate. The organic layer was removed under reduced pressure to give 1-phenyl-4-benzyl-triazole in 99% yield (0.353g**,** 0.0015 moles).

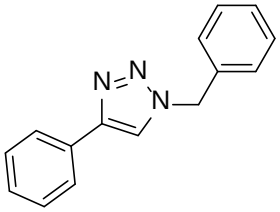
Typical reaction procedure for the multi-component CuAAC reaction:

Phenyl acetylene (0.172g, 1.6mmol), and benzyl bromide (0.260g, 1.5mmol) and sodium azide (0.200g, 3.0mmol) were added to a custom-made 2.0 x 0.5 inch screw capped copper vial inserted with a perfluoroelastomer O-ring, along with a 0.250 inch copper ball bearing ball bearing. After placement of the vial in a Spex Certiprep 8000M mixer/mill, the reagents were ball milled for 16 hours. The resulting mixture was removed from the vial with ethyl acetate. The organic layer was removed under reduced pressure to give 1-phenyl-4-benzyl-triazole in 99% yield (0.353g, 0.0015 moles).

Using our methodology we synthesized the following compounds which have been synthesized previously; These molecules are consistent with the spectra in the following journals

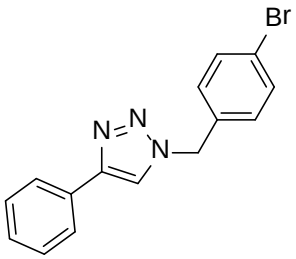
Advanced Synthesis & Catalysis
Adv. Synth. Catal., 352: 1587–1592. doi: 10.1002/adsc.200900768

1-Benzyl-4-phenyl-1*H*-1,2,3-triazole (3a):[1]



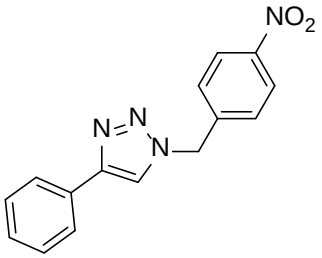
3a
White solid, mp: 130-131 ^{o}C (PE-EtOAc) (lit.[1] 132-133 ^{o}C). ^{1}H NMR: δ 7.78-7.80 (m, 2H), 7.66 (s, 1H), 7.30-7.38 (m, 8H), 5.55 (s, 2H). ^{13}C NMR: δ 148.1, 134.6, 130.5, 129.1 (2C), 128.7 (2C), 128.1 (2C), 128.0 (2C), 125.6 (2C), 119.5, 54.1.

1-(4-Bromobenzyl)-4-phenyl-1*H*-1,2,3-triazole (3b):[2]



3b
White solid, mp: 150-151 ^{o}C (PE-EtOAc) (lit.[2] 150-152 ^{o}C). ^{1}H NMR: δ 7.78-7.81 (m, 2H), 7.71 (s, 1H), 7.30-7.51 (m, 5H), 7.19-7.22 (m, 2H), 5.50 (s, 2H). ^{13}C NMR: δ 148.2, 136.8, 131.8, 130.8, 130.6, 130.3, 128.7 (2C), 128.2, 126.4, 125.6 (2C), 122.9, 119.6, 53.3.

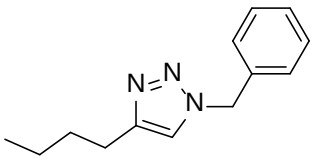
1-(4-Nitrobenzyl)-4-phenyl-1*H*-1,2,3-triazole (3c):[2]



3c

Yellow solid, mp: 156-158 °C (PE-EtOAc) (lit.[2] 157-159 °C). ^{1}H NMR: δ 8.18-8.21 (m, 2H), 7.79-7.82 (m, 3H), 7.33-7.43 (m, 5H), 5.68 (s, 2H); ^{13}C NMR: δ 148.5, 147.9, 141.7, 130.4, 128.8 (2C), 128.5 (2C), 128.4, 125.6 (2C), 124.2 (2C), 119.8, 53.1. This compound was isolated as described in C. Shao, D. Chemistry, G. Cheng, D. Su, J. Xu, X. Wang, and Y. Hu, Adv.Syn.Cat., 2010, 352, 1587-1592.

1-Benzyl-4-butyl-1H-1,2,3-triazole (3g): [1]

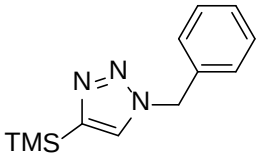


3g

White solid, mp: 61-62 °C (PE-EtOAc) (lit.[1] 62-63 °C). ^{1}H NMR: δ 7.22-7.35 (m, 6H), 5.44 (s, 2H), 2.67 (t, *J* = 7.8 Hz, 2H), 1.56-1.64 (m, 2H), 1.30-1.38 (m, 2H), 0.86-0.91 (m, 3H). ^{13}C NMR: δ 148.1, 134.7, 128.4 (2C), 127.9, 127.3 (2C), 120.3, 53.2, 31.0, 24.8, 21.7, 13.3.

Chemical Communications
Chem. Commun., 2010,**46**, 439-441. doi**:** 10.1039/B917781G

1-benzyl-4-trimethylsilanyl-1H-1,2,3-triazole(3d):



$_{1}$H-NMR ($CDCl_3$, 300 MHz): δ = 7.54 (s, 1H), 7.35-7.22 (m, 5H), 5.53 (s, 2H), 0.29 (s, 9H). $_{13}$C-NMR ($CDCl_3$, 75 MHz): δ = 148.2, 136.17, 130.2, 129.8, 129.4, 129.2, 54.5.