

## SUPPORTING INFORMATION

A layered double hydroxide, a synthetically useful heterogeneous catalyst for azide–alkyne cycloadditions in a continuous-flow reactor

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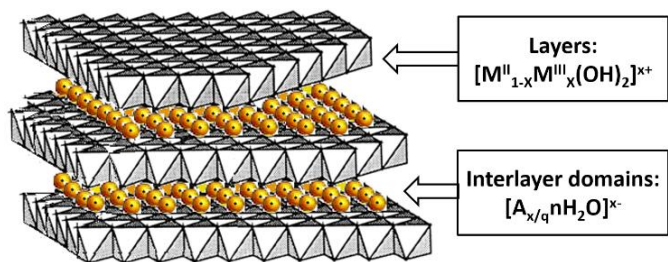


Fig. 1S Schematic structure of LDHs.

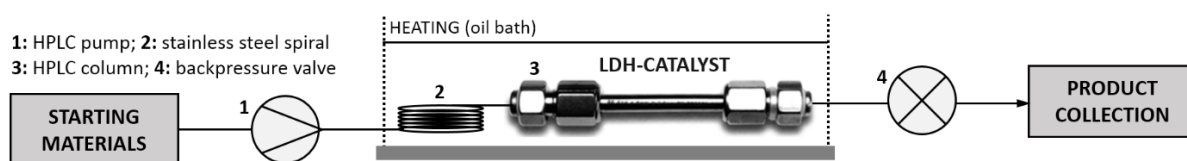


Fig. 2S Experimental setup for the CF reactions.

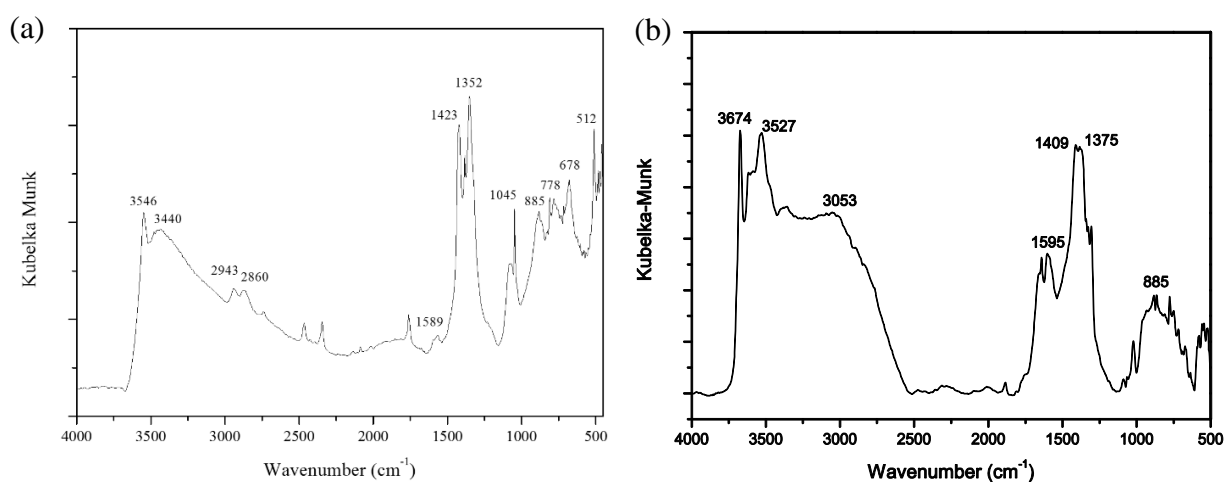


Fig. 3S FT-IR spectra of the Cu(II)Fe(III)-LDH: as-prepared material (a), after the 10-hour long synthesis (b).

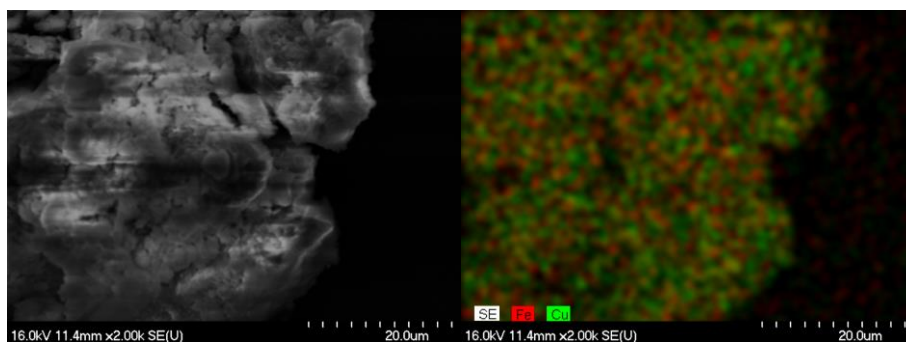
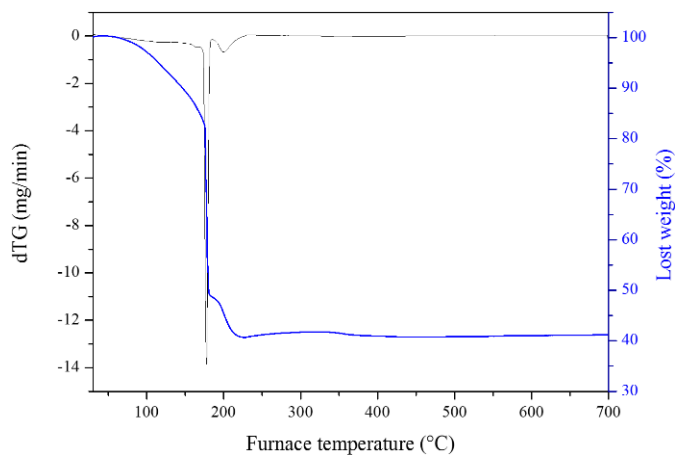
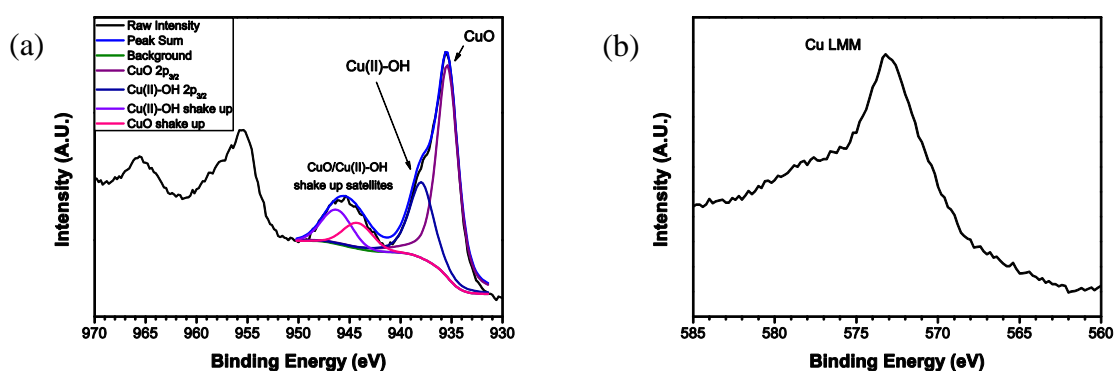


Fig. 4S. Elemental map of the as-prepared Cu(II)Fe(III)-LDH.



**Fig. 5S** TG/dTG curves of the as-prepared Cu(II)Fe(III)-LDH.

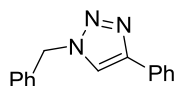


**Fig. 6S** XPS spectra indicating the presence of only Cu(II) ions for the as-prepared material (a) and as well as for the LDH sample after the 10-hour long run.

**Table 1S** Elemental composition of the Cu(II)Fe(III)-LDH determined by ICP–AES.

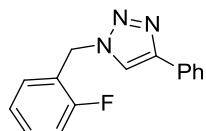
Sample	Cu		Fe		Cu/Fe
	wt%	mmol/g	wt%	mmol/g	
as-prepared	32.4510	5.107	16.2111	2.903	1.76
after the 10-hour long run	32.4422	5.105	16.2084	2.902	1.76

*1-Benzyl-4-phenyl-1H-1,2,3-triazole, 1:*



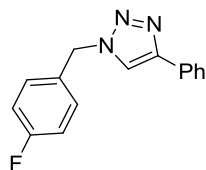
White solid; mp: 121–124 °C (lit.<sup>[S1]</sup> mp: 123–125 °C); NMR data is in agreement with the literature reference.<sup>[S1]</sup> <sup>1</sup>H NMR (400.1 MHz, CDCl<sub>3</sub>) δ<sub>H</sub>: 5.60 (s, 2H, CH<sub>2</sub>), 7.31–7.38 (m, 3H, Ar-H), 7.38–7.47 (m, 5H, Ar-H), 7.70 (s, 1H, Ar-H), 7.80–7.88 (d, 2H, Ar-H, *J*=7.8 Hz).

*1-(2-Fluorobenzyl)-4-phenyl-1H-1,2,3-triazole, 2:*



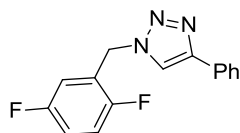
White solid; mp: 87–91 °C (lit.<sup>[S2]</sup> mp: 89–92 °C); NMR data is in agreement with the literature reference.<sup>[S3]</sup> <sup>1</sup>H NMR (400.1 MHz, CDCl<sub>3</sub>) δ<sub>H</sub>: 5.63 (s, 2H, CH<sub>2</sub>), 7.07–7.21 (m, 2H, Ar-H), 7.27–7.47 (m, 5H, Ar-H), 7.77 (s, 1H, Ar-H), 7.78–7.86 (d, 2H, Ar-H, *J*=7.8 Hz).

*1-(4-Fluorobenzyl)-4-phenyl-1H-1,2,3-triazole, 3:*



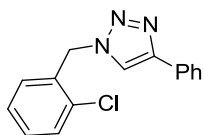
White solid; mp: 130–133 °C (lit.<sup>[S4]</sup> mp: 129–131 °C); NMR data is in agreement with the literature reference.<sup>[S4]</sup> <sup>1</sup>H NMR (400.1 MHz, CDCl<sub>3</sub>) δ<sub>H</sub>: 5.52 (s, 2H, CH<sub>2</sub>), 6.98–7.11 (t, 2H, Ar-H, *J*=8.1 Hz), 7.23–7.35 (m, 3H, Ar-H), 7.36–7.45 (m, 2H, Ar-H), 7.67 (s, 1H, Ar-H), 7.74–7.85 (d, 2H, Ar-H, *J*=7.4 Hz).

*1-(2,5-Difluorobenzyl)-4-phenyl-1H-1,2,3-triazole, 4:*



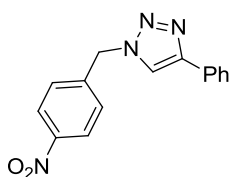
White solid; mp: 100–103 °C (lit.<sup>[S2]</sup> mp: 98–102 °C); NMR data is in agreement with the literature reference.<sup>[S2]</sup> <sup>1</sup>H NMR (400.1 MHz, CDCl<sub>3</sub>) δ<sub>H</sub>: 5.61 (s, 2H, CH<sub>2</sub>), 6.93–7.16 (m, 3H, Ar-H), 7.29–7.36 (m, 1H, Ar-H), 7.37–7.46 (m, 2H, Ar-H), 7.73–7.89 (m, 3H, Ar-H).

*1-(2-Chlorobenzyl)-4-phenyl-1H-1,2,3-triazole, 5:*



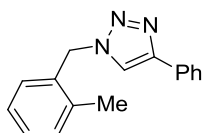
White solid; mp: 80–83 °C (lit.<sup>[S5]</sup> mp: 79–81 °C); NMR data is in agreement with the literature reference.<sup>[S5]</sup> <sup>1</sup>H NMR (400.1 MHz, CDCl<sub>3</sub>)  $\delta_{\text{H}}$ : 5.71 (s, 2H, CH<sub>2</sub>), 7.18–7.35 (m, 4H, Ar-H), 7.36–7.48 (m, 3H, Ar-H), 7.78 (s, 1H, Ar-H), 7.79–7.88 (d, 2H, Ar-H,  $J=7.8$  Hz).

*1-(4-Nitrobenzyl)-4-phenyl-1H-1,2,3-triazole, 6:*



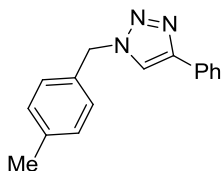
Yellowish solid; mp: 156–159 °C (lit.<sup>[S1]</sup> mp: 158–159 °C); NMR data is in agreement with the literature reference.<sup>[S1]</sup> <sup>1</sup>H NMR (400.1 MHz, CDCl<sub>3</sub>)  $\delta_{\text{H}}$ : 5.71 (s, 2H, CH<sub>2</sub>), 7.30–7.40 (t, 1H, Ar-H,  $J=7.9$  Hz), 7.40–7.50 (m, 4H, Ar-H), 7.77 (s, 1H, Ar-H), 7.80–7.88 (d, 2H, Ar-H,  $J=7.7$  Hz), 8.20–8.31 (d, 2H, Ar-H,  $J=8.3$  Hz).

*1-(2-Methylbenzyl)-4-phenyl-1H-1,2,3-triazole, 7:*



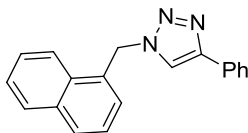
White solid; mp: 91–94 °C (lit.<sup>[S6]</sup> mp: 94–95 °C); NMR data is in agreement with the literature reference.<sup>[S6]</sup> <sup>1</sup>H NMR (400.1 MHz, CDCl<sub>3</sub>)  $\delta_{\text{H}}$ : 2.32 (s, 3H, CH<sub>3</sub>), 5.56 (s, 2H, CH<sub>2</sub>), 7.18–7.27 (m, 3H, Ar-H), 7.27–7.34 (m, 2H, Ar-H), 7.35–7.43 (t, 2H, Ar-H,  $J=7.3$  Hz), 7.55 (s, 1H, Ar-H), 7.75–7.84 (d, 2H, Ar-H,  $J=7.3$  Hz).

*1-(4-Methylbenzyl)-4-phenyl-1H-1,2,3-triazole, 8:*



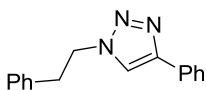
White solid; mp: 92–94 °C (lit.<sup>[S1]</sup> mp: 93–95 °C); NMR data is in agreement with the literature reference.<sup>[S1]</sup> <sup>1</sup>H NMR (400.1 MHz, CDCl<sub>3</sub>)  $\delta_{\text{H}}$ : 2.36 (s, 3H, CH<sub>3</sub>), 5.52 (s, 2H, CH<sub>2</sub>), 7.15–7.25 (m, 4H, Ar-H), 7.29–7.34 (m, 1H, Ar-H), 7.35–7.43 (m, 2H, Ar-H), 7.65 (s, 1H, Ar-H), 7.75–7.83 (d, 2H, Ar-H,  $J=7.4$  Hz).

*1-(Naphthalen-1-ylmethyl)-4-phenyl-1H-1,2,3-triazole, 9:*



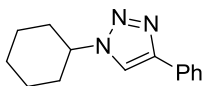
White solid; mp: 138–141 °C (lit.<sup>[S7]</sup> mp: 141–142 °C); NMR data is in agreement with the literature reference.<sup>[S7]</sup> <sup>1</sup>H NMR (400.1 MHz, CDCl<sub>3</sub>) δ<sub>H</sub>: 5.76 (s, 2H, CH<sub>2</sub>), 7.30–7.37 (m, 1H, Ar-H), 7.38–7.45 (m, 3H, Ar-H), 7.50–7.59 (m, 2H, Ar-H), 7.71 (s, 1H, Ar-H), 7.78–7.92 (m, 6H, Ar-H).

*1-Phenethyl-4-phenyl-1H-1,2,3-triazole, 10:*



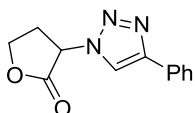
White solid; mp: 139–142 °C (lit.<sup>[S1]</sup> mp: 141–142 °C); NMR data is in agreement with the literature reference.<sup>[S1]</sup> <sup>1</sup>H NMR (400.1 MHz, CDCl<sub>3</sub>) δ<sub>H</sub>: 3.19–3.31 (t, 2H, CH<sub>2</sub>, *J*=7.2 Hz), 4.56–4.68 (t, 2H, CH<sub>2</sub>, *J*=7.2 Hz), 7.09–7.18 (d, 2H, Ar-H, *J*=7.3 Hz), 7.23–7.37 (m, 4H, Ar-H), 7.37–7.46 (m, 2H, Ar-H), 7.49 (s, 1H, Ar-H), 7.73–7.83 (d, 2H, Ar-H, *J*=7.3 Hz).

*1-Cyclohexyl-4-phenyl-1H-1,2,3-triazole, 11:*



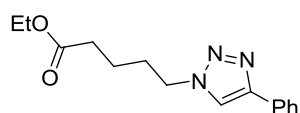
White solid; mp: 106–109 °C (lit.<sup>[S1]</sup> mp: 108–109 °C); NMR data is in agreement with the literature reference.<sup>[S1]</sup> <sup>1</sup>H NMR (400.1 MHz, CDCl<sub>3</sub>) δ<sub>H</sub>: 1.20–1.37 (m, 1H, CH<sub>2</sub>), 1.38–1.56 (m, 2H, CH<sub>2</sub>), 1.69–1.86 (m, 3H, 2 CH<sub>2</sub>), 1.86–2.01 (m, 2H, CH<sub>2</sub>), 2.16–2.32 (m, 2H, CH<sub>2</sub>), 4.40–4.54 (m, 1H, CH), 7.28–7.35 (m, 1H, Ar-H), 7.36–7.46 (m, 2H, Ar-H), 7.70–7.90 (m, 3H, Ar-H).

*3-(4-Phenyl-1H-1,2,3-triazol-1-yl)dihydrofuran-2(3H)-one, 12:*



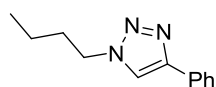
White solid; mp: 138–142 °C (lit.<sup>[S1]</sup> mp: 140–141 °C); NMR data is in agreement with the literature reference.<sup>[S1]</sup> <sup>1</sup>H NMR (400.1 MHz, CDCl<sub>3</sub>) δ<sub>H</sub>: 2.98–3.20 (m, 2H, CH<sub>2</sub>), 4.46–4.62 (m, 1H, CH<sub>2</sub>), 4.62–4.79 (m, 1H, CH<sub>2</sub>), 5.30–5.46 (t, 1H, CH, *J*=9.1 Hz), 7.30–7.39 (m, 1H, Ar-H), 7.39–7.49 (m, 2H, Ar-H), 7.77–7.90 (d, 2H, Ar-H, *J*=7.7 Hz), 8.05 (s, 1H, Ar-H).

*Ethyl 5-(4-phenyl-1H-1,2,3-triazol-1-yl)pentanoate, 13:*



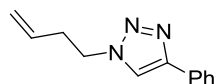
White solid; mp: 50–53 °C (lit.<sup>[S8]</sup> mp: 50–53 °C); NMR data is in agreement with the literature reference.<sup>[S8]</sup> <sup>1</sup>H NMR (400.1 MHz, CDCl<sub>3</sub>)  $\delta_{\text{H}}$ : 1.17–1.29 (m, 3H, CH<sub>3</sub>), 1.61–1.74 (m, 2H, CH<sub>2</sub>), 1.92–2.05 (m, 2H, CH<sub>2</sub>), 2.23–2.40 (m, 2H, CH<sub>2</sub>), 4.03–4.17 (m, 2H, CH<sub>2</sub>), 4.33–4.45 (m, 2H, CH<sub>2</sub>), 7.28–7.35 (m, 1H, Ar-H), 7.36–7.45 (m, 2H, Ar-H), 7.72–7.86 (m, 3H, Ar-H).

*1-Butyl-4-phenyl-1H-1,2,3-triazole, 14:*



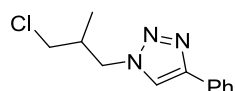
White solid; mp: 46–49 °C (lit.<sup>[S9]</sup> mp: 46–47 °C); NMR data is in agreement with the literature reference.<sup>[S9]</sup> <sup>1</sup>H NMR (400.1 MHz, CDCl<sub>3</sub>)  $\delta_{\text{H}}$ : 0.92–1.04 (t, 3H, CH<sub>3</sub>,  $J=7.4$  Hz), 1.36–1.46 (m, 2H, CH<sub>2</sub>), 1.88–1.99 (m, 2H, CH<sub>2</sub>), 4.35–4.44 (t, 2H, CH<sub>2</sub>,  $J=7.3$  Hz), 7.30–7.37 (m, 1H, Ar-H), 7.38–7.46 (m, 2H, Ar-H), 7.75 (s, 1H, Ar-H), 7.79–7.88 (d, 2H, Ar-H,  $J=7.5$  Hz).

*1-(But-3-en-1-yl)-4-phenyl-1H-1,2,3-triazole, 15:*



Brownish solid, mp: 40–43 °C (lit.<sup>[S10]</sup> mp: 42–44 °C); NMR data is in agreement with the literature reference.<sup>[S11]</sup> <sup>1</sup>H NMR (400.1 MHz, CDCl<sub>3</sub>)  $\delta_{\text{H}}$ : 2.63–2.76 (m, 2H, CH<sub>2</sub>), 4.38–4.53 (t, 2H, CH<sub>2</sub>,  $J=7.2$  Hz), 5.03–5.19 (d, 2H, CH<sub>2</sub>,  $J=12.7$  Hz), 5.71–5.87 (m, 1H, CH), 7.29–7.36 (m, 1H, Ar-H), 7.37–7.46 (m, 2H, Ar-H), 7.75 (s, 1H, Ar-H), 7.78–7.87 (d, 2H, Ar-H,  $J=7.4$  Hz).

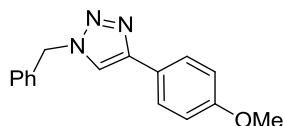
*1-(3-Chloro-2-methylpropyl)-4-phenyl-1H-1,2,3-triazole, 16:*



White solid; mp: 48–50 °C (lit.<sup>[S8]</sup> mp: 50–51 °C); NMR data is in agreement with the literature reference.<sup>[S8]</sup> <sup>1</sup>H NMR (400.1 MHz, CDCl<sub>3</sub>)  $\delta_{\text{H}}$ : 1.06–1.15 (d, 3H, CH<sub>3</sub>,  $J=6.7$  Hz), 2.51–2.64 (m, 1H, CH), ), 3.40–3.52 (m, 2H, CH<sub>2</sub>), 4.28–4.52 (m, 2H, CH<sub>2</sub>), 7.28–7.37 (m, 1H, Ar-H), 7.38–7.47 (m, 2H, Ar-H), 7.75–7.89 (m, 3H, Ar-H).

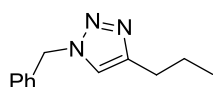


*1-benzyl-4-(4-methoxyphenyl)-1H-1,2,3-triazole, 17:*



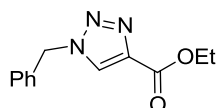
White solid; mp: 140–143 °C (lit.<sup>[S1]</sup> mp: 143–145 °C); NMR data is in agreement with the literature reference.<sup>[S1]</sup> <sup>1</sup>H NMR (400.1 MHz, CDCl<sub>3</sub>) δ<sub>H</sub>: 3.81 (s, 3H, CH<sub>3</sub>), 5.53 (s, 2H, CH<sub>2</sub>), 6.88–6.97 (d, 2H, Ar-H, *J*=8.5 Hz), 7.27–7.41 (m, 5H, Ar-H), 7.59 (s, 1H, Ar-H), 7.69–7.76 (d, 2H, Ar-H, *J*=8.6 Hz).

*1-Benzyl-4-propyl-1H-1,2,3-triazole, 18:*



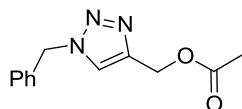
Brownish oil; NMR data is in agreement with the literature reference.<sup>[S11]</sup> <sup>1</sup>H NMR (400.1 MHz, CDCl<sub>3</sub>) δ<sub>H</sub>: 0.93–1.01 (t, 3H, CH<sub>3</sub>, *J*=7.2 Hz), 1.64–1.76 (m, 2H, CH<sub>2</sub>), 2.65–2.76 (t, 2H, CH<sub>2</sub>, *J*=7.6 Hz), 5.52 (s, 2H, CH<sub>2</sub>), 7.25–7.30 (m, 2H, Ar-H), 7.32–7.45 (m, 4H, Ar-H).

*Ethyl 1-benzyl-1H-1,2,3-triazole-4-carboxylate, 19:*



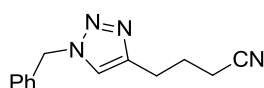
Yellowish solid; mp: 79–82 °C (lit.<sup>[S1]</sup> mp: 82–83 °C); NMR data is in agreement with the literature reference.<sup>[S1]</sup> <sup>1</sup>H NMR (400.1 MHz, CDCl<sub>3</sub>) δ<sub>H</sub>: 1.30–1.46 (t, 3H, CH<sub>3</sub>, *J*=7.2 Hz), 4.32–4.46 (q, 2H, CH<sub>2</sub>, *J*=7.1 Hz), 5.59 (s, 2H, CH<sub>2</sub>), 7.24–7.35 (m, 2H, Ar-H), 7.35–7.46 (m, 3H, Ar-H), 8.02 (s, 1H, Ar-H).

*(1-Benzyl-1H-1,2,3-triazol-4-yl)methyl acetate, 20:*



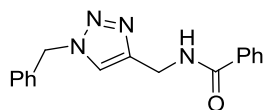
Yellowish solid; mp: 55–58 °C (lit.<sup>[S8]</sup> mp: 55–56 °C); NMR data is in agreement with the literature reference.<sup>[S8]</sup> <sup>1</sup>H NMR (400.1 MHz, CDCl<sub>3</sub>) δ<sub>H</sub>: 2.05 (s, 3H, CH<sub>3</sub>), 5.20 (s, 2H, CH<sub>2</sub>), 5.52 (s, 2H, CH<sub>2</sub>), 7.23–7.31 (m, 2H, Ar-H), 7.34–7.41 (m, 3H, Ar-H), 7.57 (s, 1H, Ar-H).

*4-(1-Benzyl-1H-1,2,3-triazol-4-yl)butanenitrile, 21:*



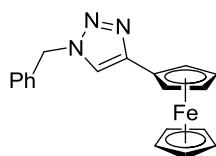
White solid; mp: 61–64 °C (lit.<sup>[S8]</sup> mp: 64–66 °C); NMR data is in agreement with the literature reference.<sup>[S8]</sup> <sup>1</sup>H NMR (400.1 MHz, CDCl<sub>3</sub>) δ<sub>H</sub>: 2.05–2.14 (m, 2H, CH<sub>2</sub>), 2.40–2.47 (t, 2H, CH<sub>2</sub>, *J*=7.0 Hz), 2.83–2.91 (t, 2H, CH<sub>2</sub>, *J*=7.20 Hz), 5.53 (s, 2H, CH<sub>2</sub>), 7.27–7.32 (m, 3H, Ar-H), 7.36–7.45 (m, 3H, Ar-H).

*N*-((1-benzyl-1*H*-1,2,3-triazol-4-yl)methyl)benzamide, **22**:



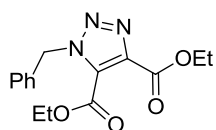
White solid; mp: 125–128 °C (lit.<sup>[S10]</sup> mp: 126–128 °C); NMR data is in agreement with the literature reference.<sup>[S12]</sup> <sup>1</sup>H NMR (400.1 MHz, CDCl<sub>3</sub>) δ<sub>H</sub>: 4.69–4.75 (d, 2H, CH<sub>2</sub>, *J*=5.6 Hz), 5.55 (s, 2H, CH<sub>2</sub>), 6.86 (m, 1H, NH), 7.31–7.34 (m, 1H, Ar-H), 7.37–7.50 (m, 5H, Ar-H), 7.51–7.58 (m, 2H, Ar-H), 7.77–7.84 (m, 2H, Ar-H).

*1*-Benzyl-4-ferrocenyl-1*H*-1,2,3-triazole, **23**:



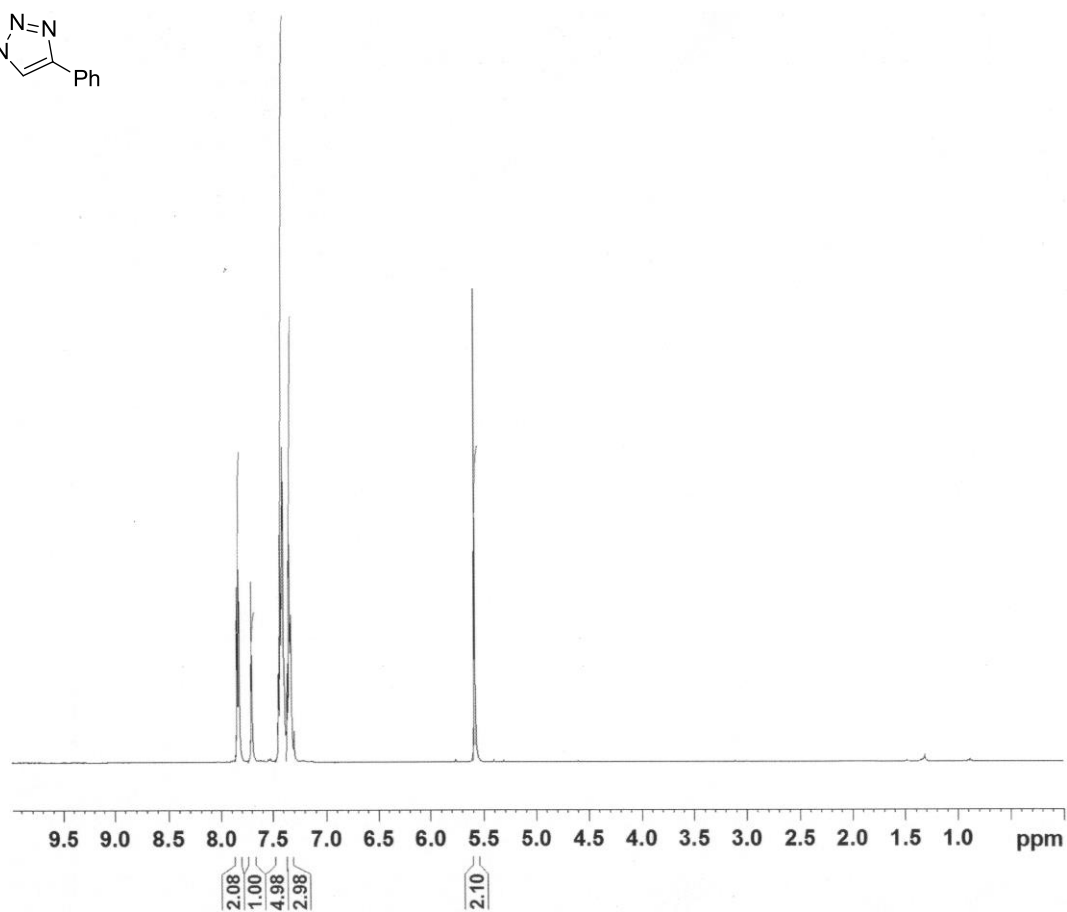
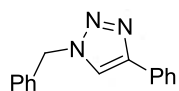
Golden yellow solid; mp: 147–150 °C (lit.<sup>[S13]</sup> mp: 145–147 °C); NMR data is in agreement with the literature reference.<sup>[S3]</sup> <sup>1</sup>H NMR (400.1 MHz, CDCl<sub>3</sub>) δ<sub>H</sub>: 4.08 (s, 5H, Ar-H), 4.27–4.32 (m, 2H, Ar-H), 4.68–4.75 (m, 2H, Ar-H), 5.58 (s, 2H, CH<sub>2</sub>), 7.31–7.34 (m, 2H, Ar-H), 7.35–7.45 (m, 4H, Ar-H).

Diethyl 1-benzyl-1*H*-1,2,3-triazole-4,5-dicarboxylate, **24**:

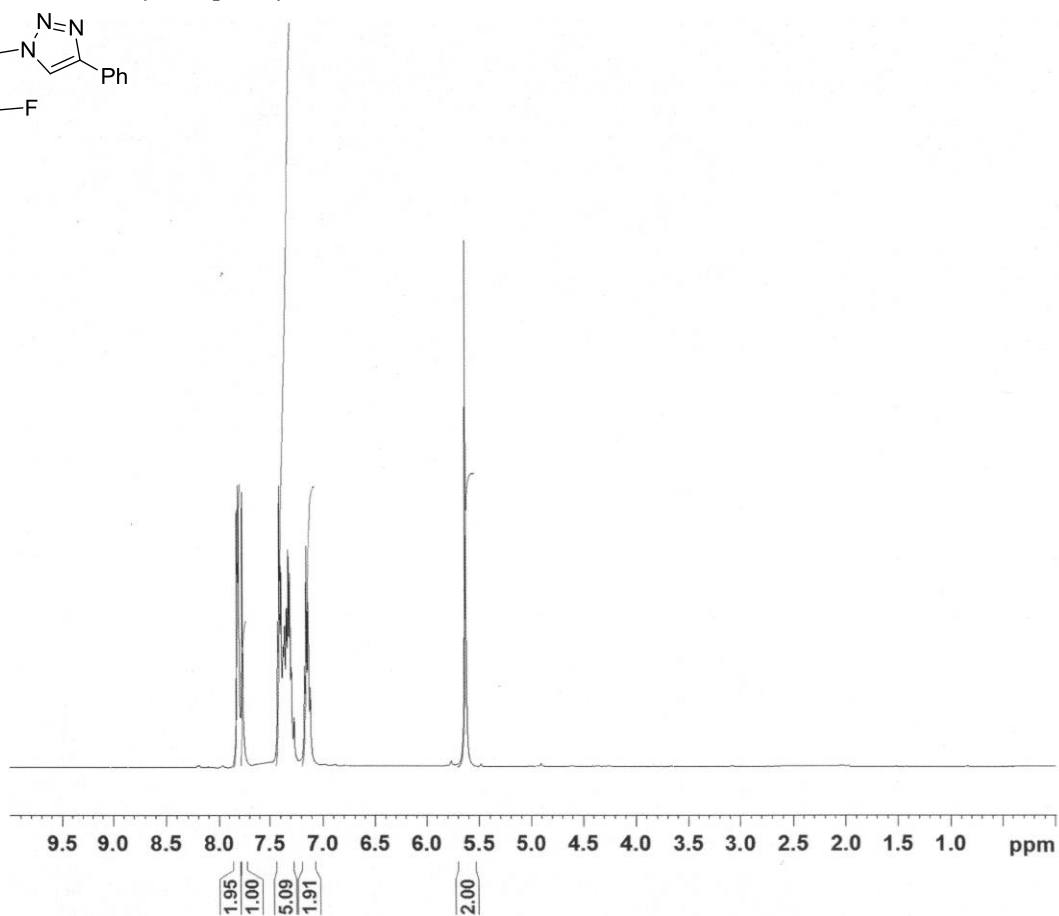
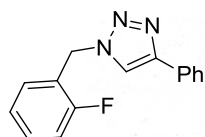


Colorless oil; NMR data is in agreement with the literature reference.<sup>[S14]</sup> <sup>1</sup>H NMR (400.1 MHz, CDCl<sub>3</sub>) δ<sub>H</sub>: 1.26–1.33 (t, 3H, CH<sub>3</sub>, *J*=7.3 Hz), 1.39–1.44 (t, 3H, CH<sub>3</sub>, *J*=7.3 Hz), 4.31–4.40 (q, 2H, CH<sub>2</sub>, *J*=7.3 Hz), 4.41–4.50 (q, 2H, CH<sub>2</sub>, *J*=7.3 Hz), 5.85 (s, 2H, CH<sub>2</sub>), 7.26–7.31 (m, 3H, Ar-H), 7.33–7.38 (m, 2H, Ar-H).

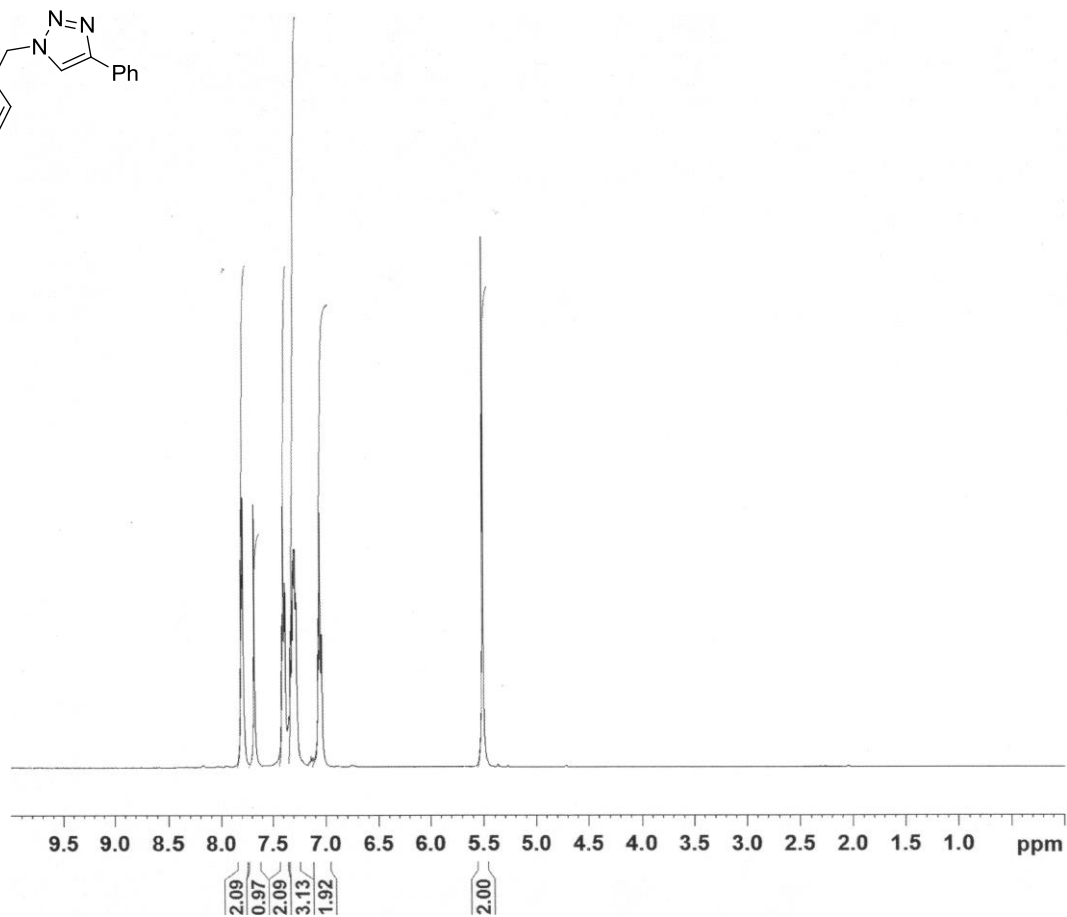
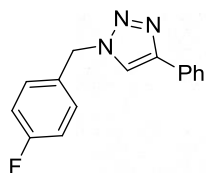
*1-Benzyl-4-phenyl-1H-1,2,3-triazole, 1, <sup>1</sup>H NMR in CDCl<sub>3</sub>:*



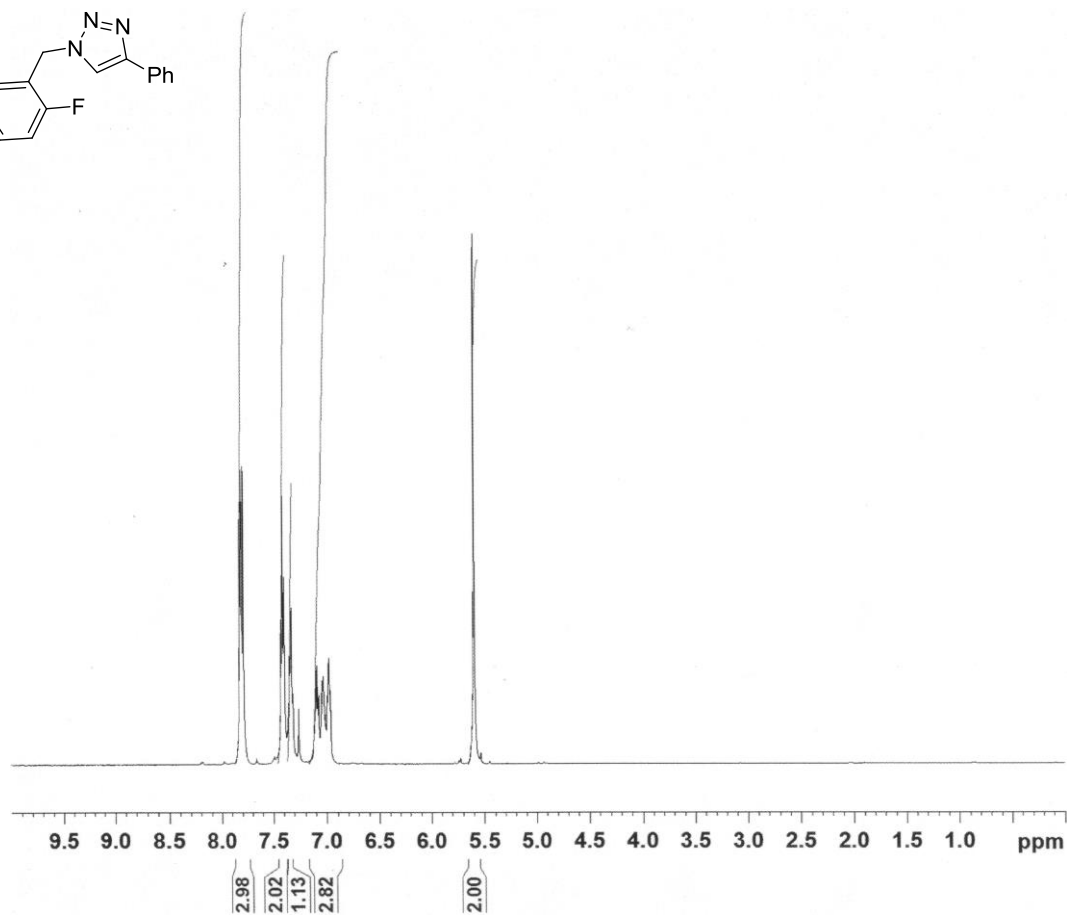
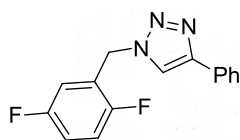
*1-(2-Fluorobenzyl)-4-phenyl-1H-1,2,3-triazole, 2, <sup>1</sup>H NMR in CDCl<sub>3</sub>:*



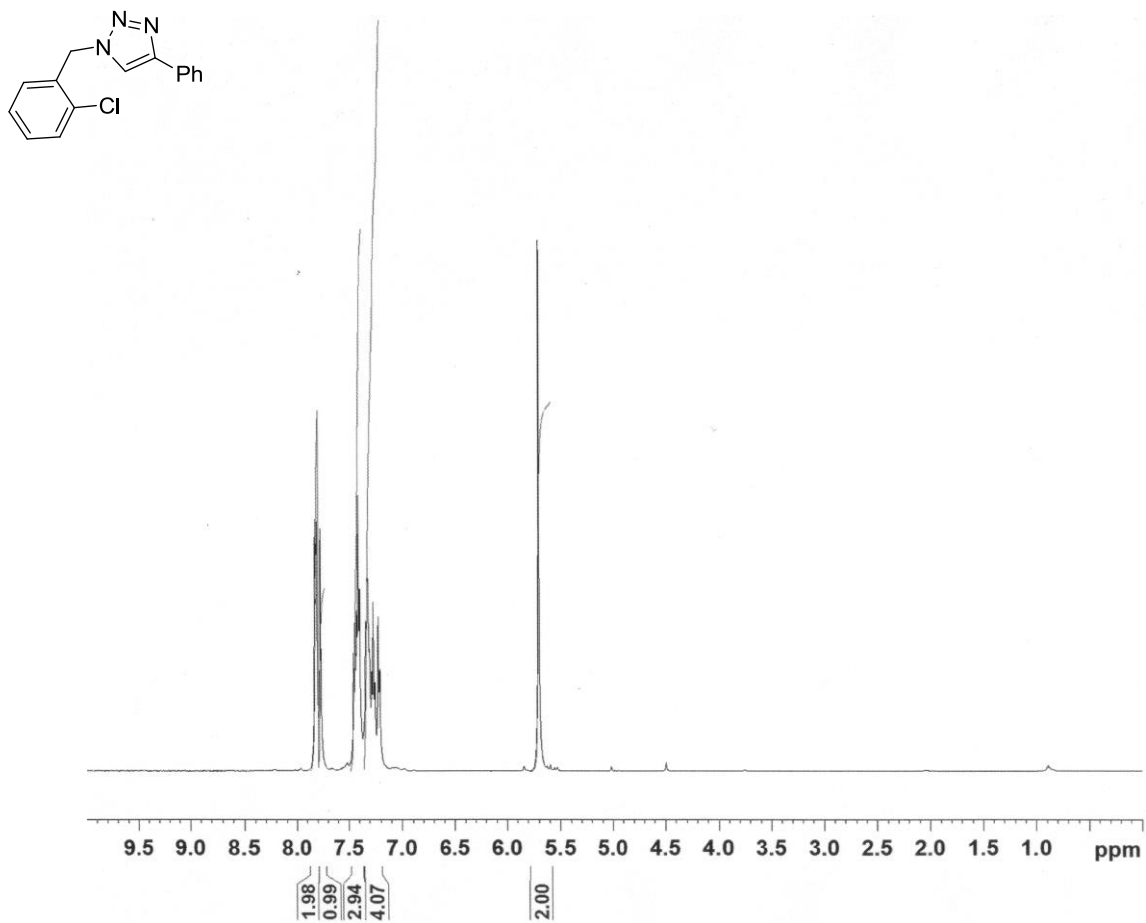
1-(4-Fluorobenzyl)-4-phenyl-1H-1,2,3-triazole, **3**,  $^1\text{H}$  NMR in  $\text{CDCl}_3$ :



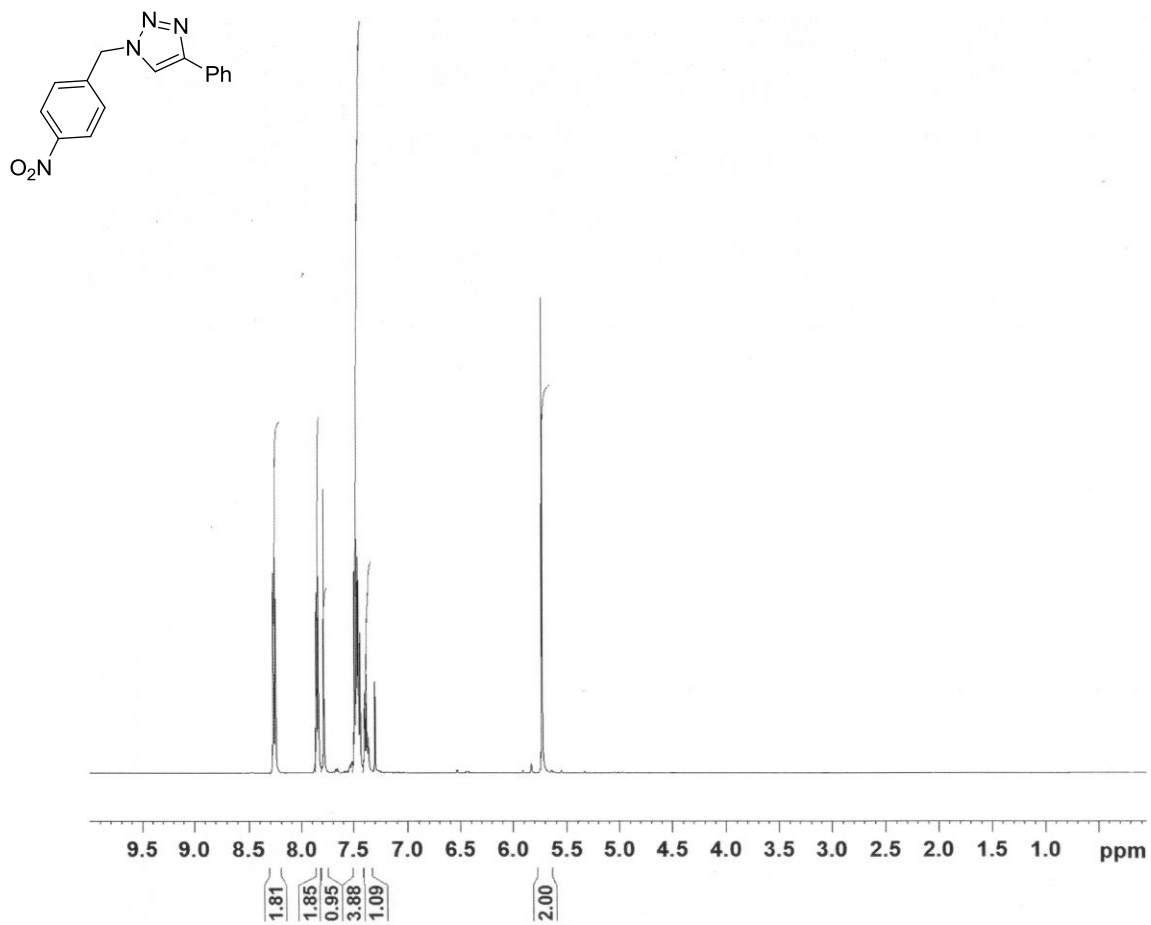
1-(2,5-Difluorobenzyl)-4-phenyl-1H-1,2,3-triazole, **4**,  $^1\text{H}$  NMR in  $\text{CDCl}_3$ :



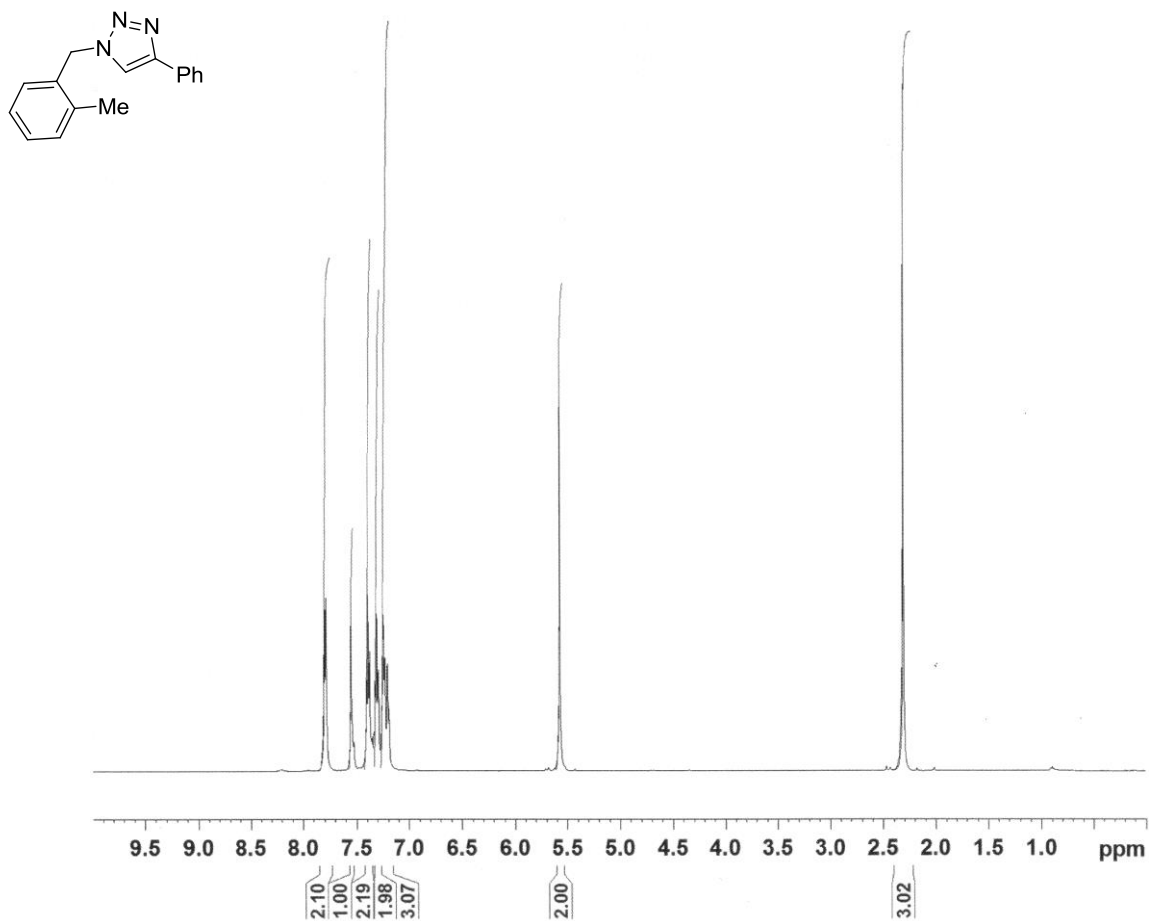
1-(2-Chlorobenzyl)-4-phenyl-1H-1,2,3-triazole, **5**,  $^1\text{H}$  NMR in  $\text{CDCl}_3$ :



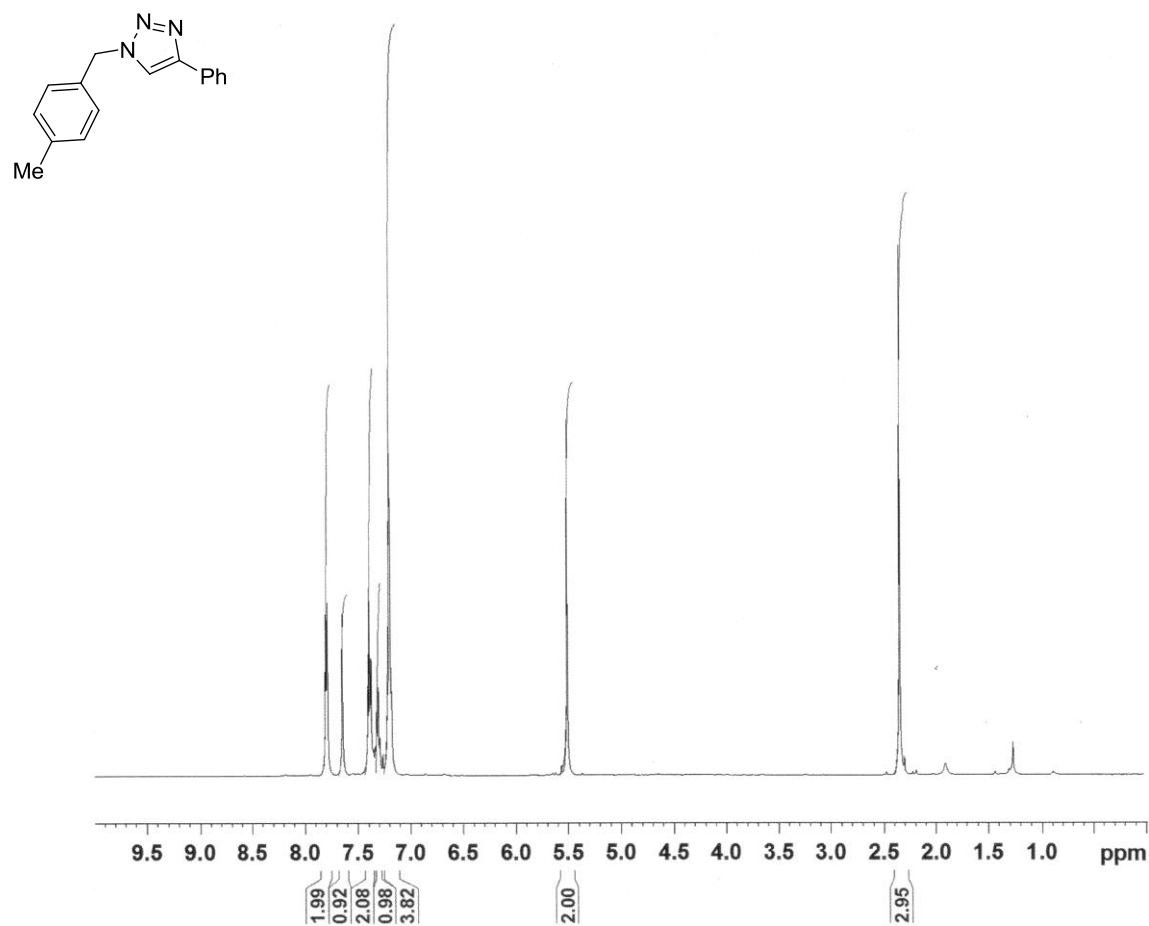
1-(4-Nitrobenzyl)-4-phenyl-1H-1,2,3-triazole, **6**,  $^1\text{H}$  NMR in  $\text{CDCl}_3$ :



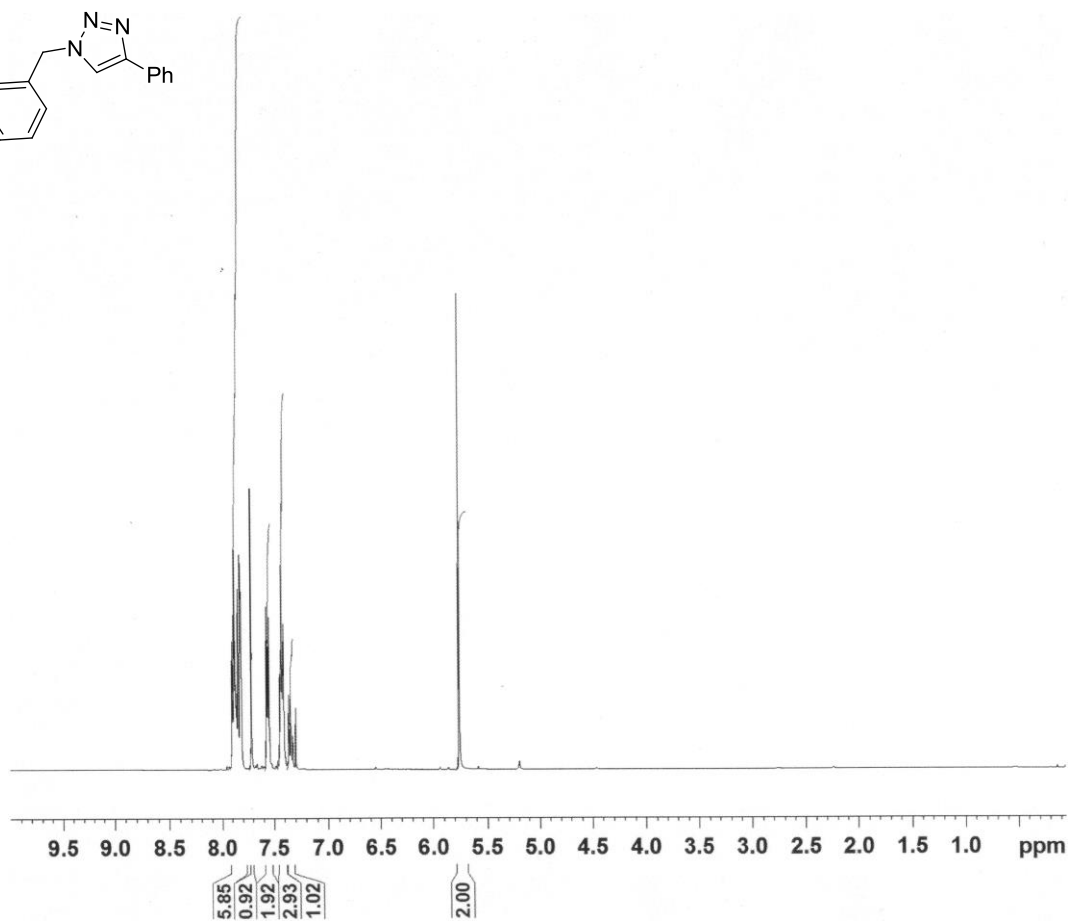
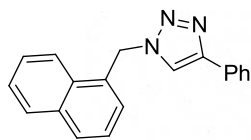
1-(2-Methylbenzyl)-4-phenyl-1H-1,2,3-triazole, **7**,  $^1\text{H}$  NMR in  $\text{CDCl}_3$ :



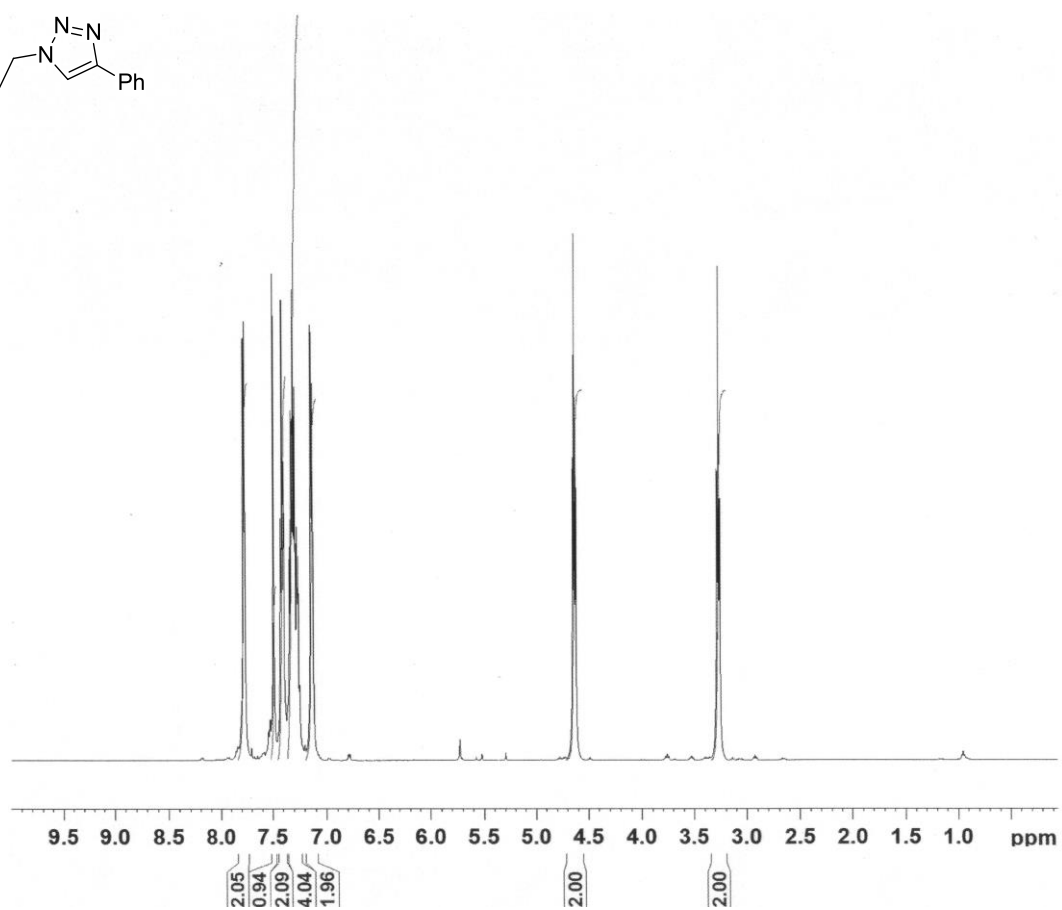
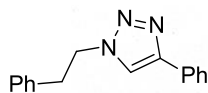
1-(4-Methylbenzyl)-4-phenyl-1H-1,2,3-triazole, **8**,  $^1\text{H}$  NMR in  $\text{CDCl}_3$ :



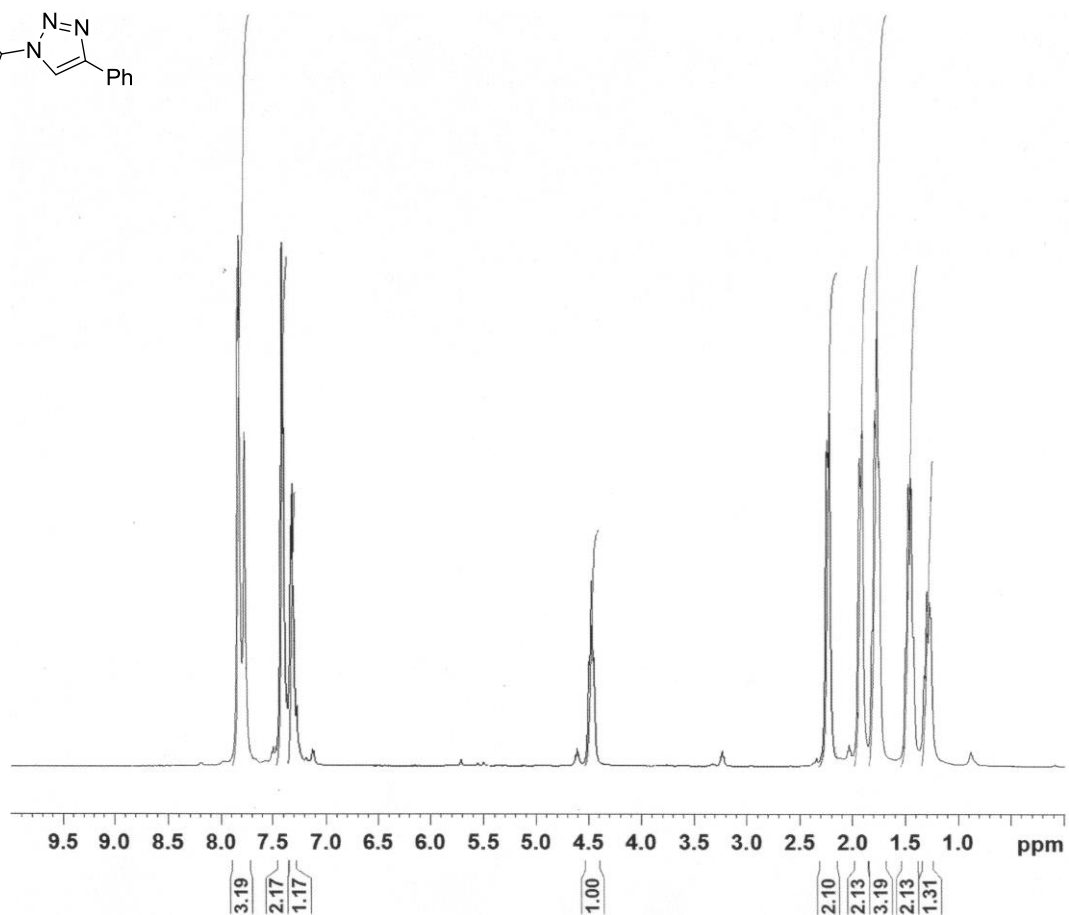
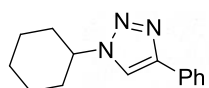
1-(Naphthalen-1-ylmethyl)-4-phenyl-1H-1,2,3-triazole, **9**,  $^1\text{H}$  NMR in  $\text{CDCl}_3$ :



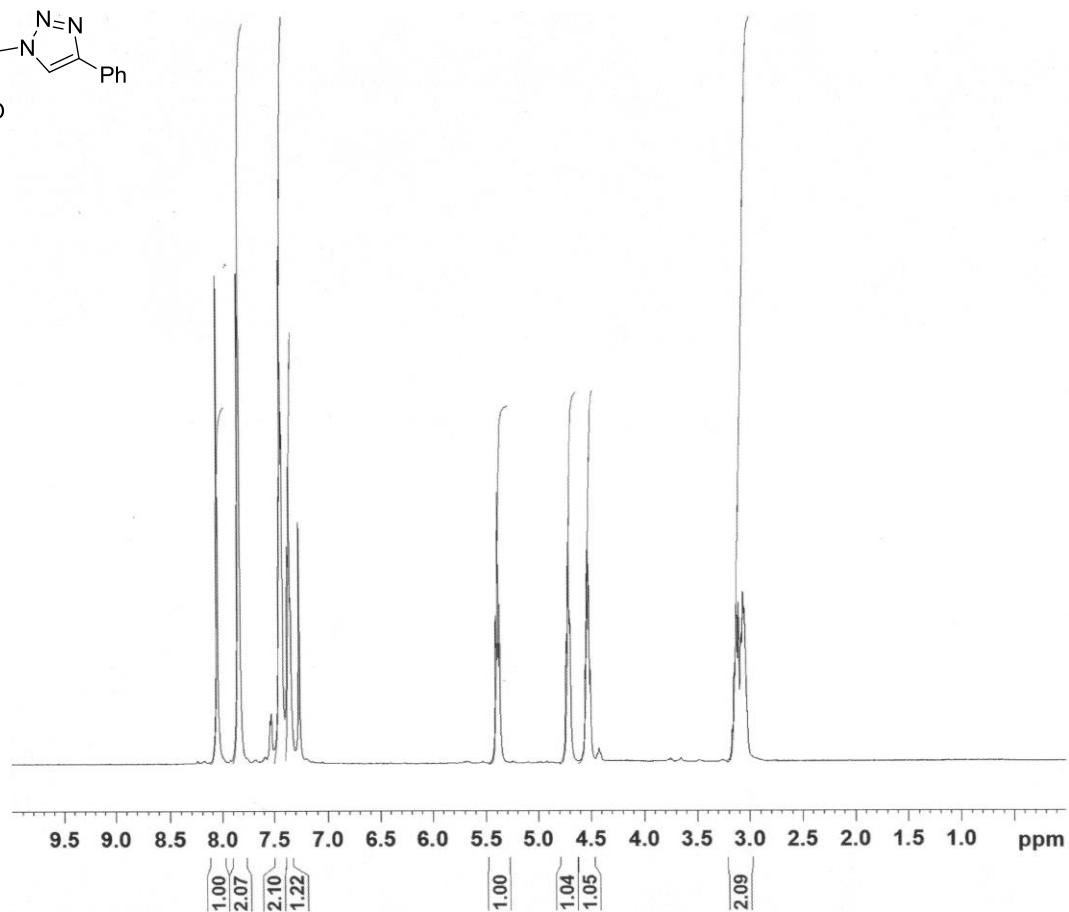
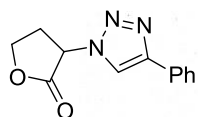
1-Phenethyl-4-phenyl-1H-1,2,3-triazole, **10**,  $^1\text{H}$  NMR in  $\text{CDCl}_3$ :



1-Cyclohexyl-4-phenyl-1H-1,2,3-triazole, **11**,  $^1\text{H}$  NMR in  $\text{CDCl}_3$ :

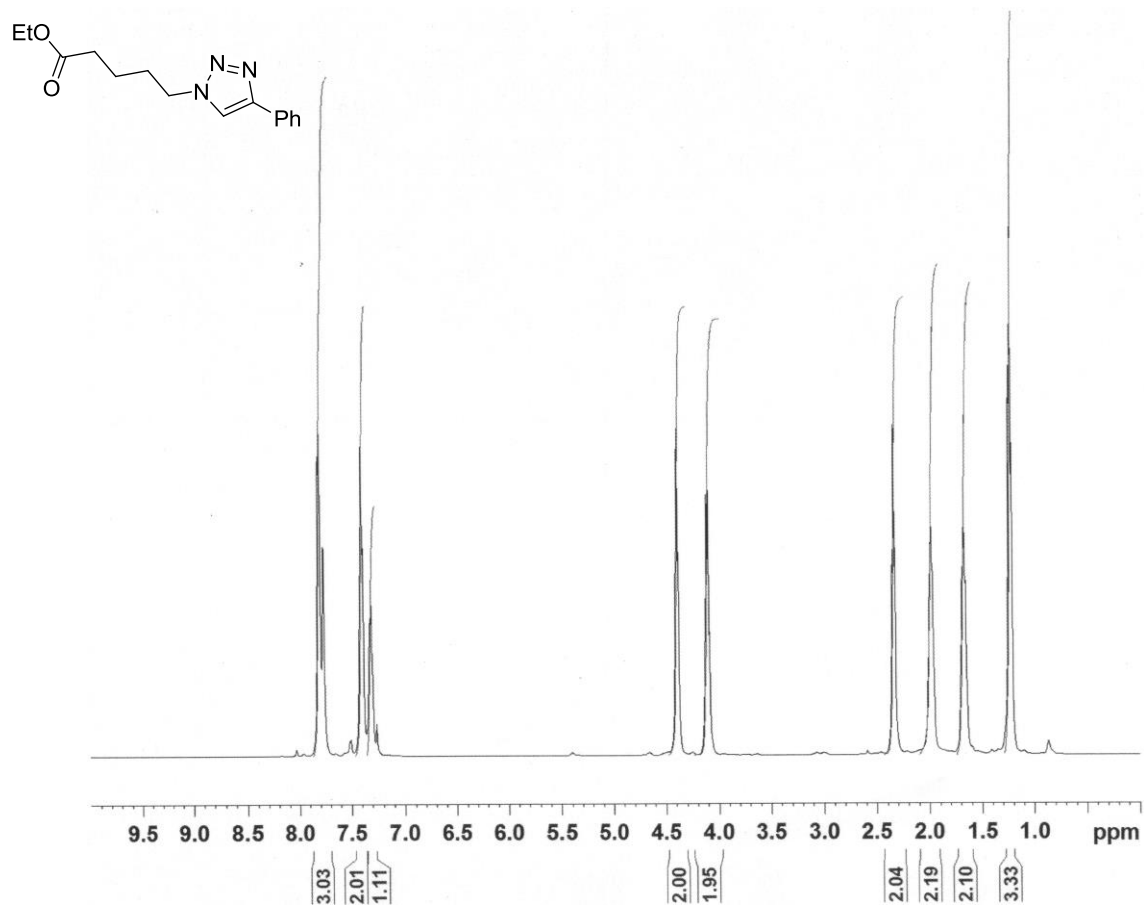


3-(4-Phenyl-1H-1,2,3-triazol-1-yl)dihydrofuran-2(3H)-one, **12**,  $^1\text{H}$  NMR in  $\text{CDCl}_3$ :

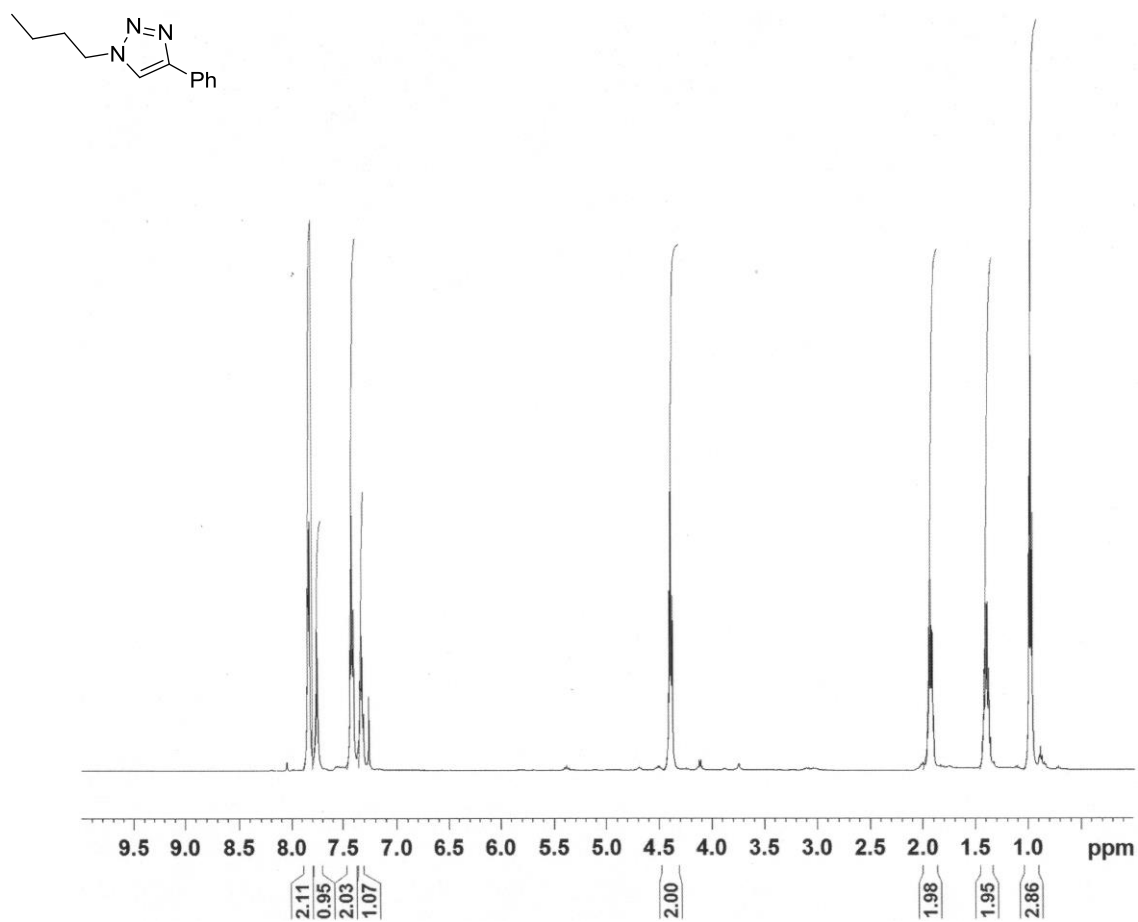




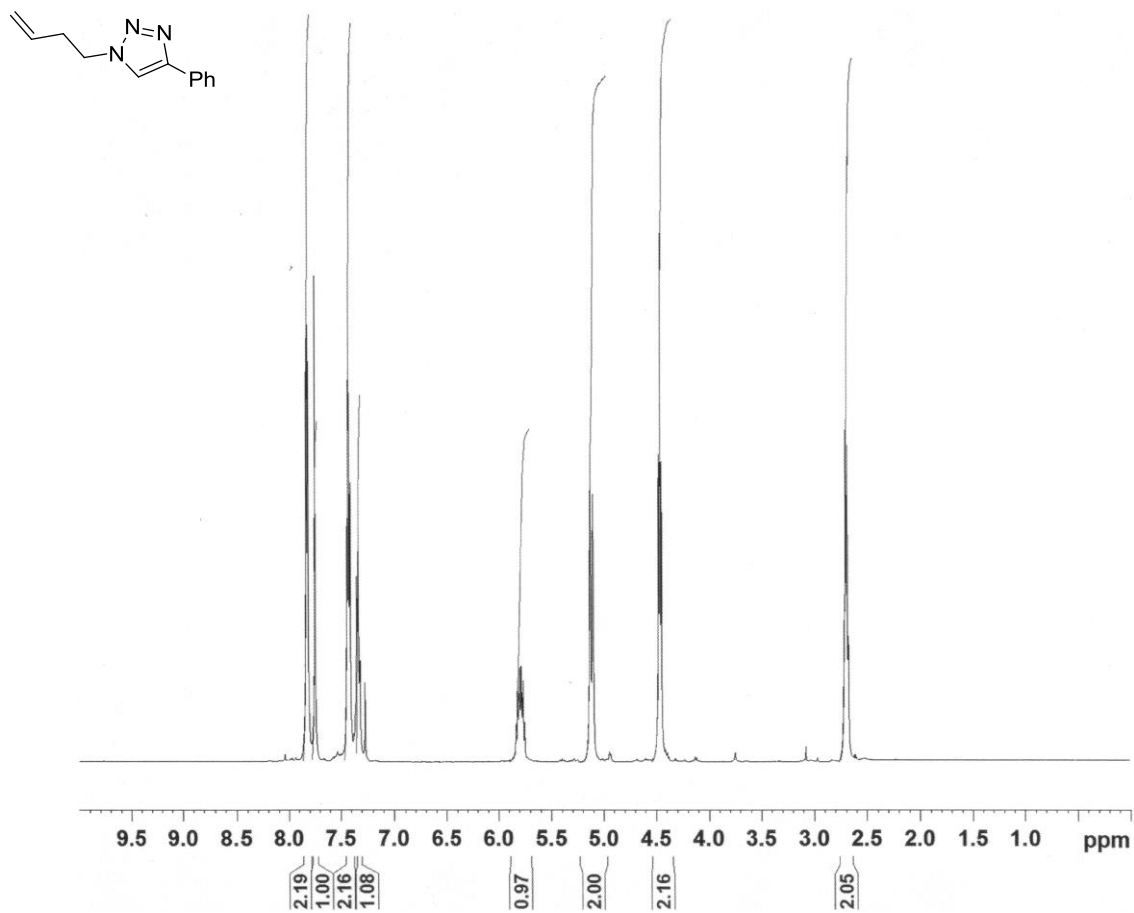
Ethyl 5-(4-phenyl-1H-1,2,3-triazol-1-yl)pentanoate, **13**,  $^1\text{H}$  NMR in  $\text{CDCl}_3$ :



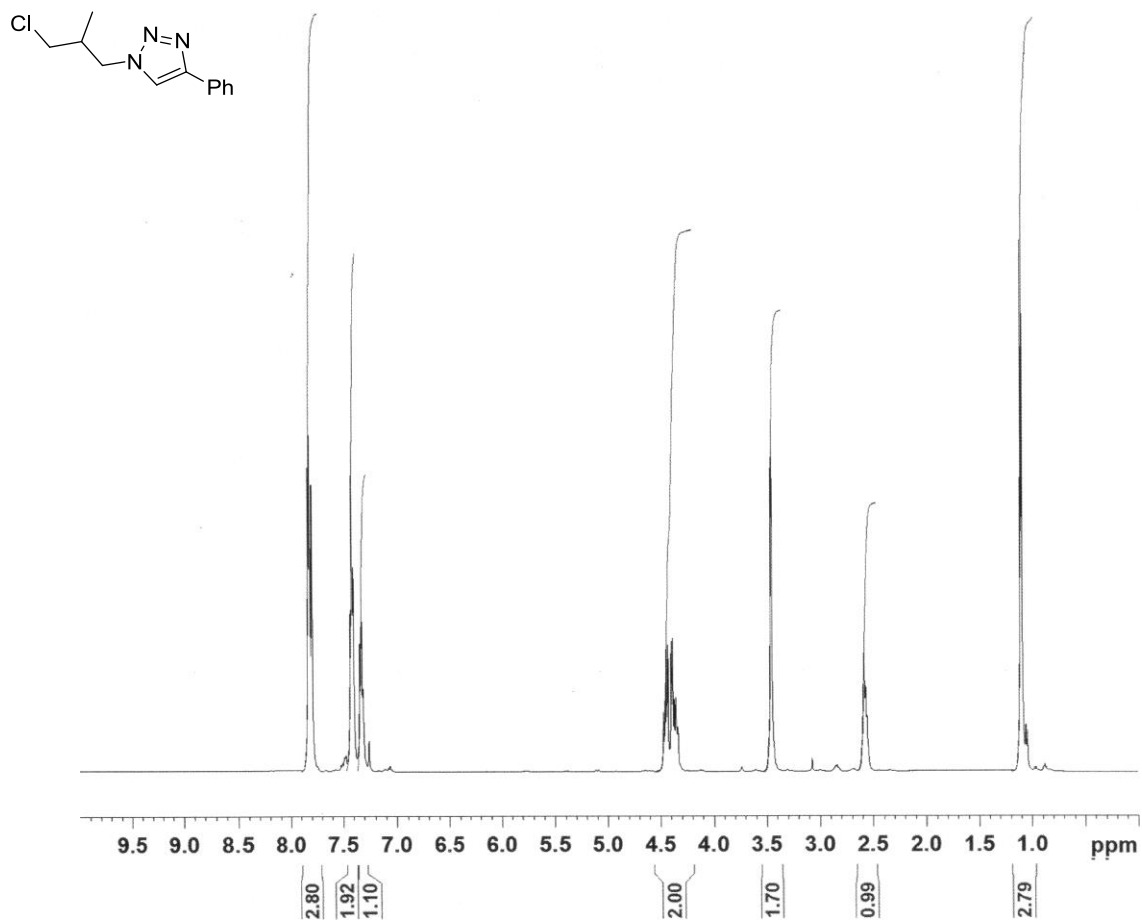
1-Butyl-4-phenyl-1H-1,2,3-triazole, **14**,  $^1\text{H}$  NMR in  $\text{CDCl}_3$ :



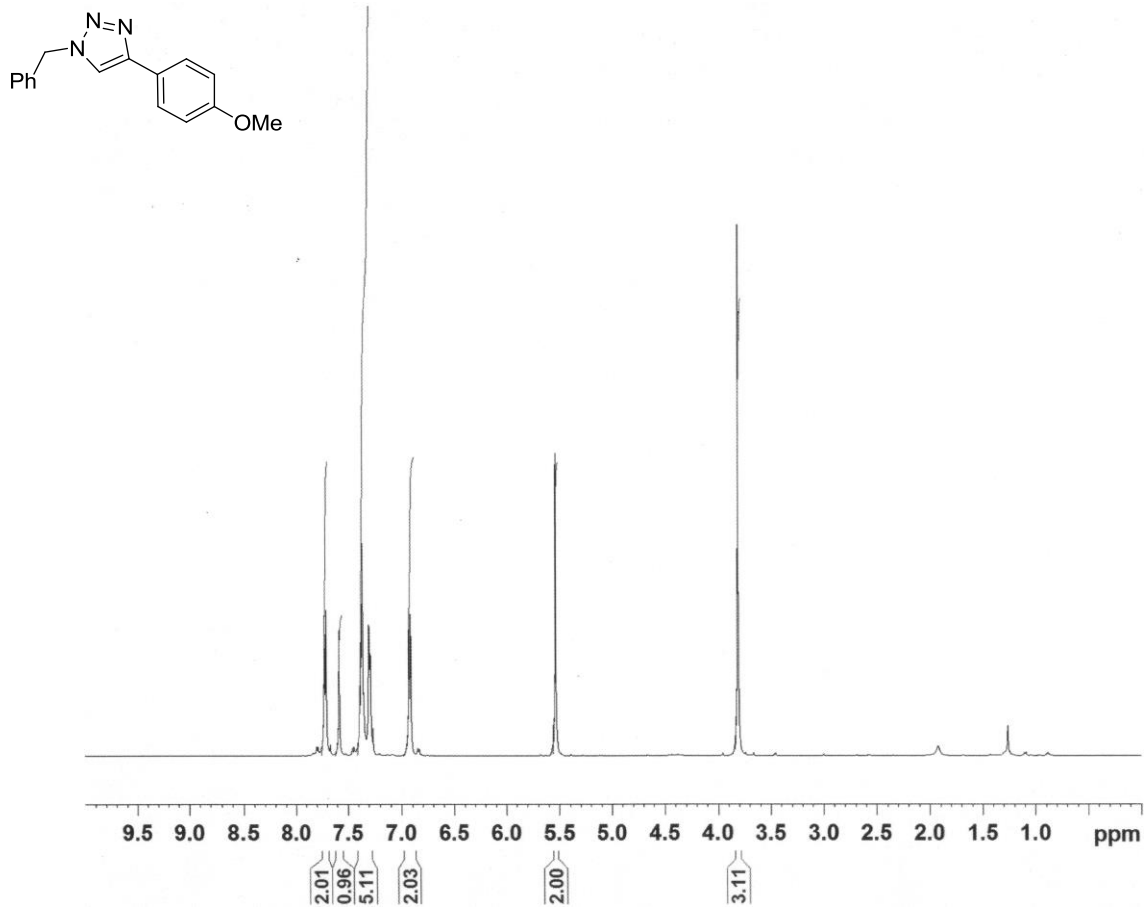
1-(But-3-en-1-yl)-4-phenyl-1H-1,2,3-triazole, **15**,  $^1\text{H}$  NMR in  $\text{CDCl}_3$ :



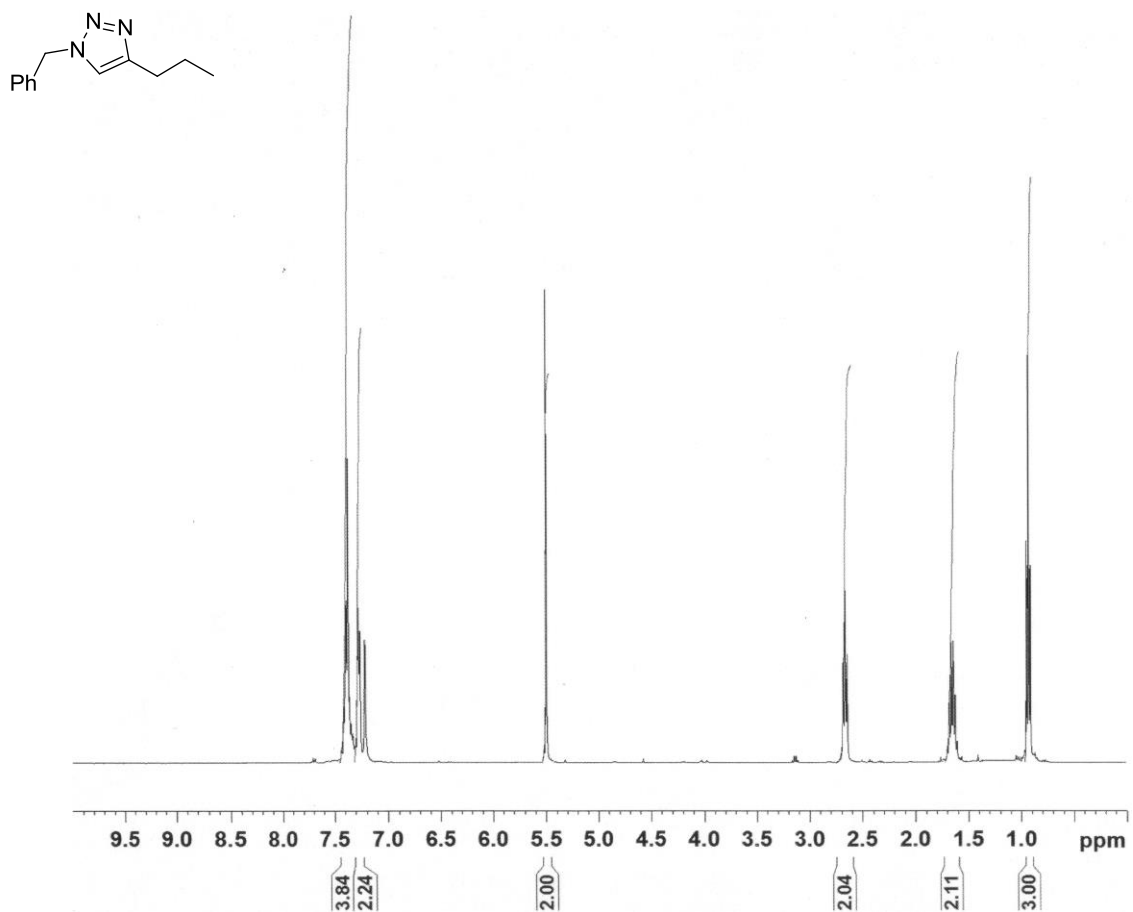
1-(3-Chloro-2-methylpropyl)-4-phenyl-1H-1,2,3-triazole, **16**,  $^1\text{H}$  NMR in  $\text{CDCl}_3$ :



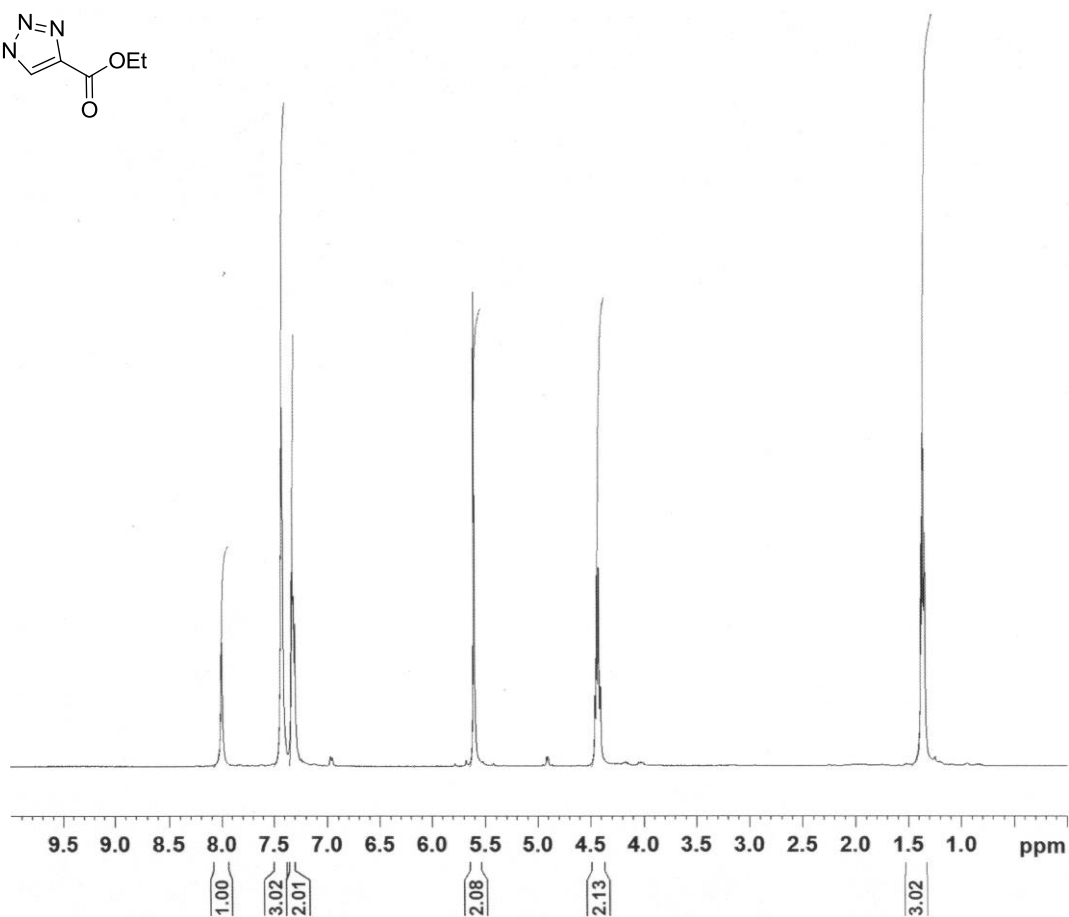
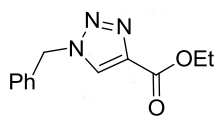
1-benzyl-4-(4-methoxyphenyl)-1H-1,2,3-triazole, **17**,  $^1\text{H}$  NMR in  $\text{CDCl}_3$ :



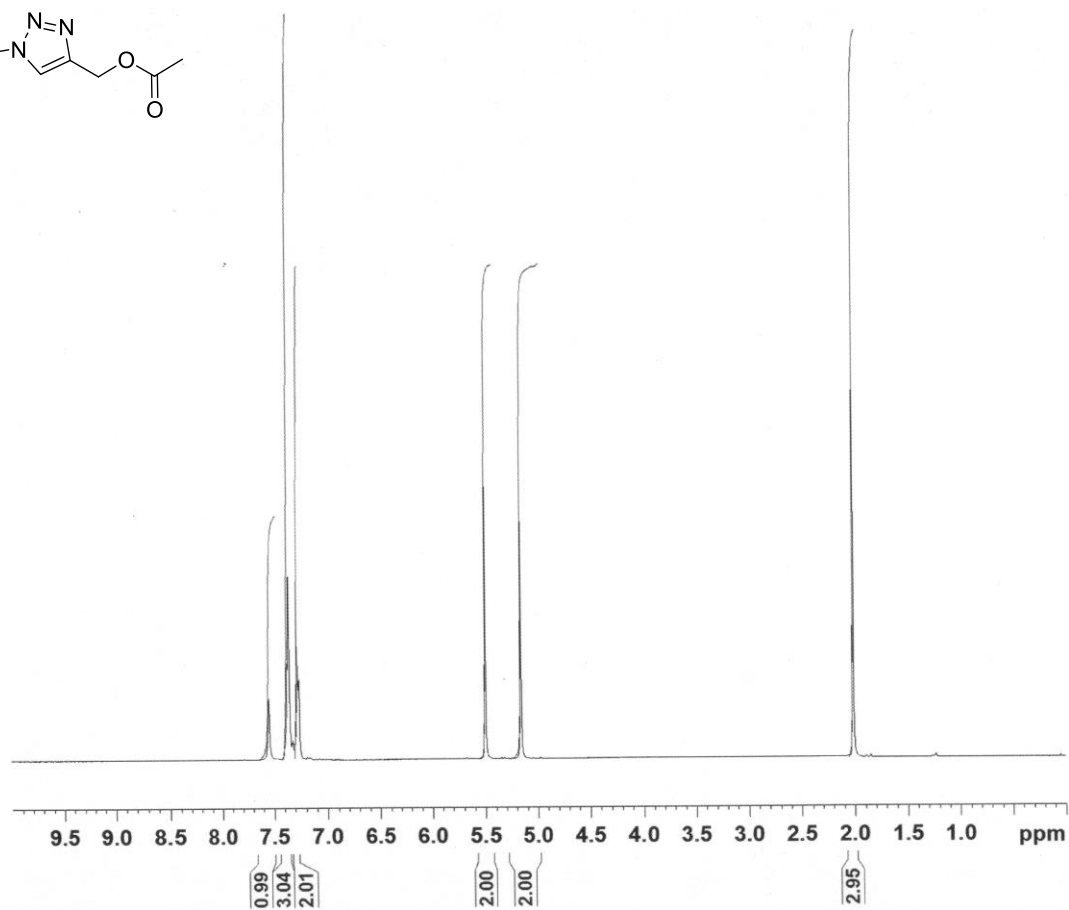
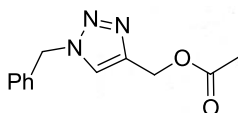
1-Benzyl-4-propyl-1H-1,2,3-triazole, **18**,  $^1\text{H}$  NMR in  $\text{CDCl}_3$ :



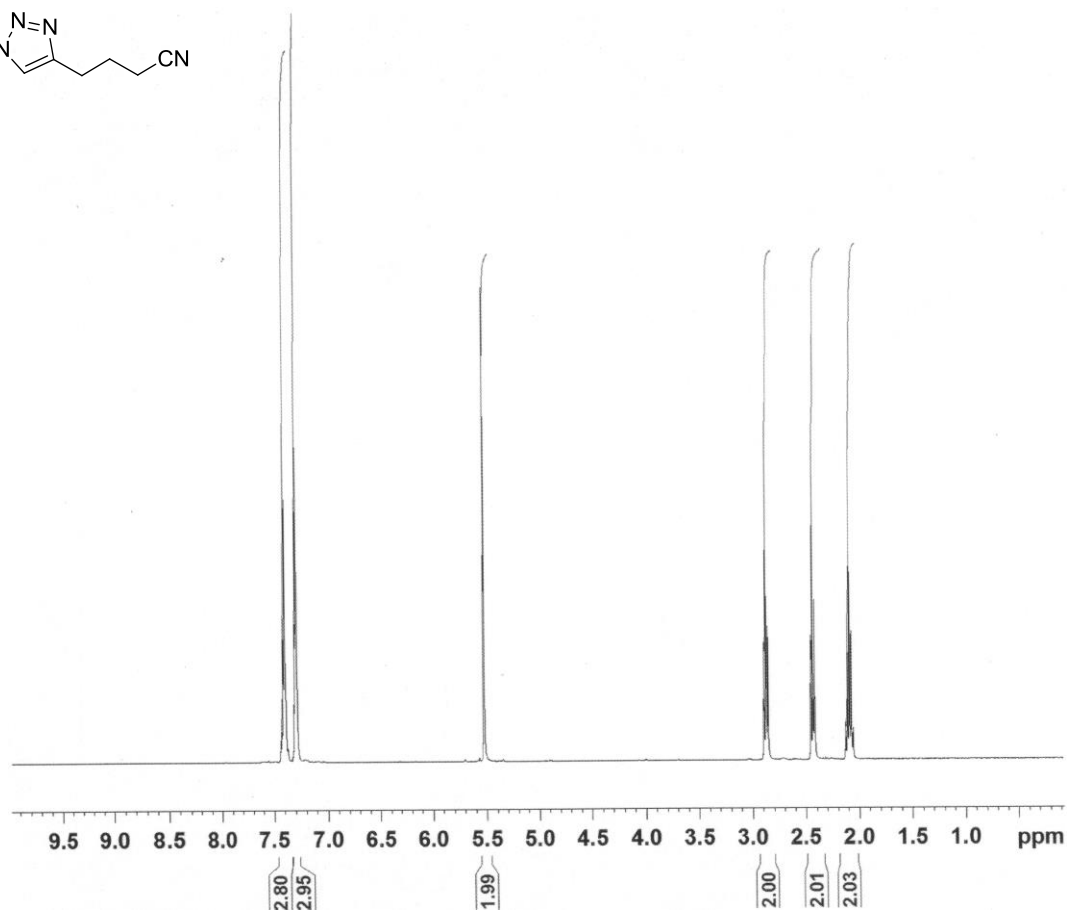
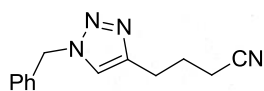
Ethyl 1-benzyl-1H-1,2,3-triazole-4-carboxylate, **19**,  $^1\text{H}$  NMR in  $\text{CDCl}_3$ :



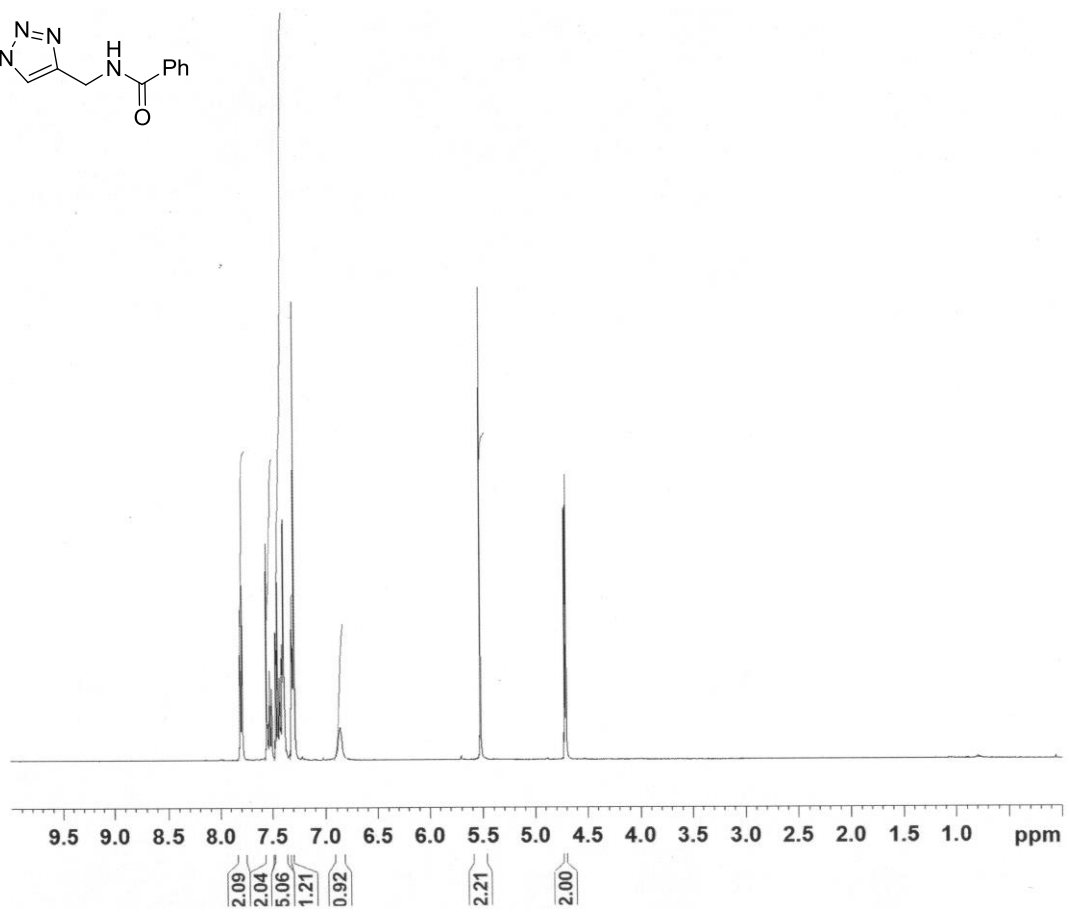
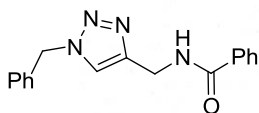
(1-Benzyl-1H-1,2,3-triazol-4-yl)methyl acetate, **20**,  $^1\text{H}$  NMR in  $\text{CDCl}_3$ :



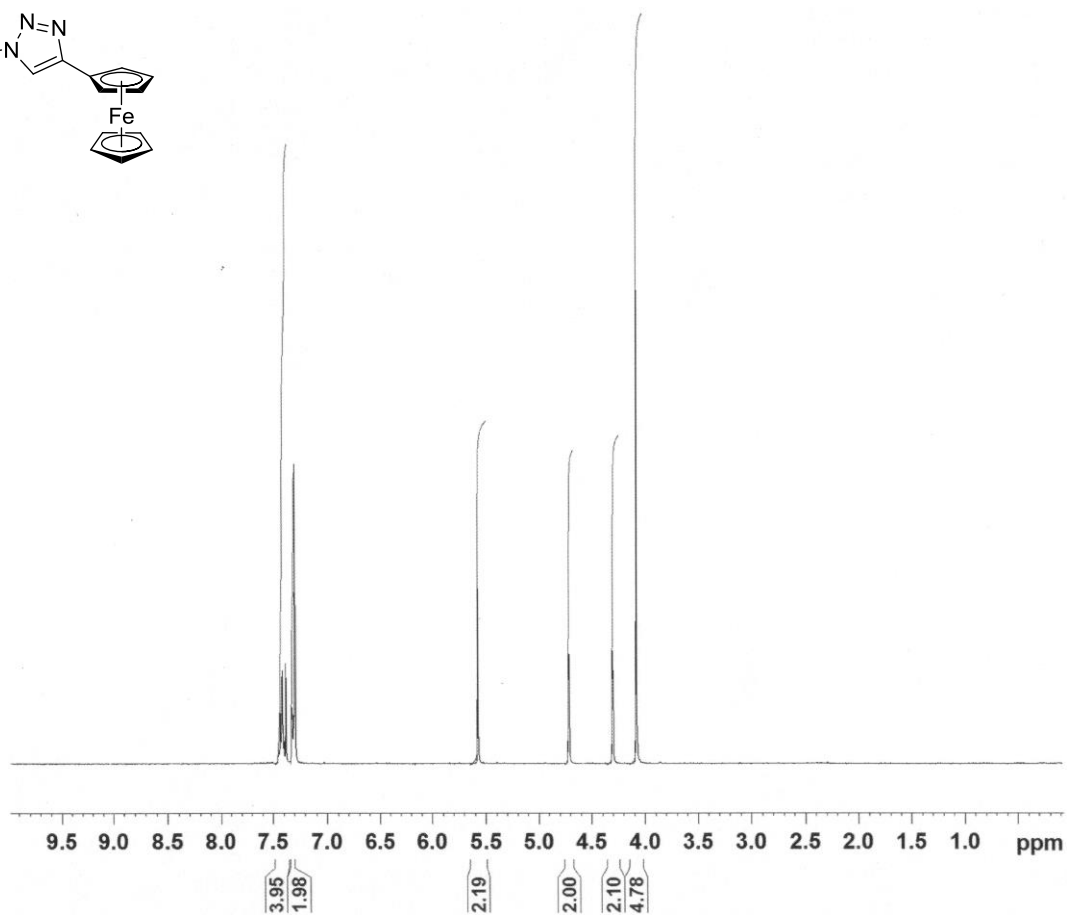
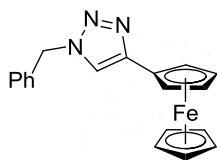
4-(1-Benzyl-1H-1,2,3-triazol-4-yl)butanenitrile, **21**,  $^1\text{H}$  NMR in  $\text{CDCl}_3$ :



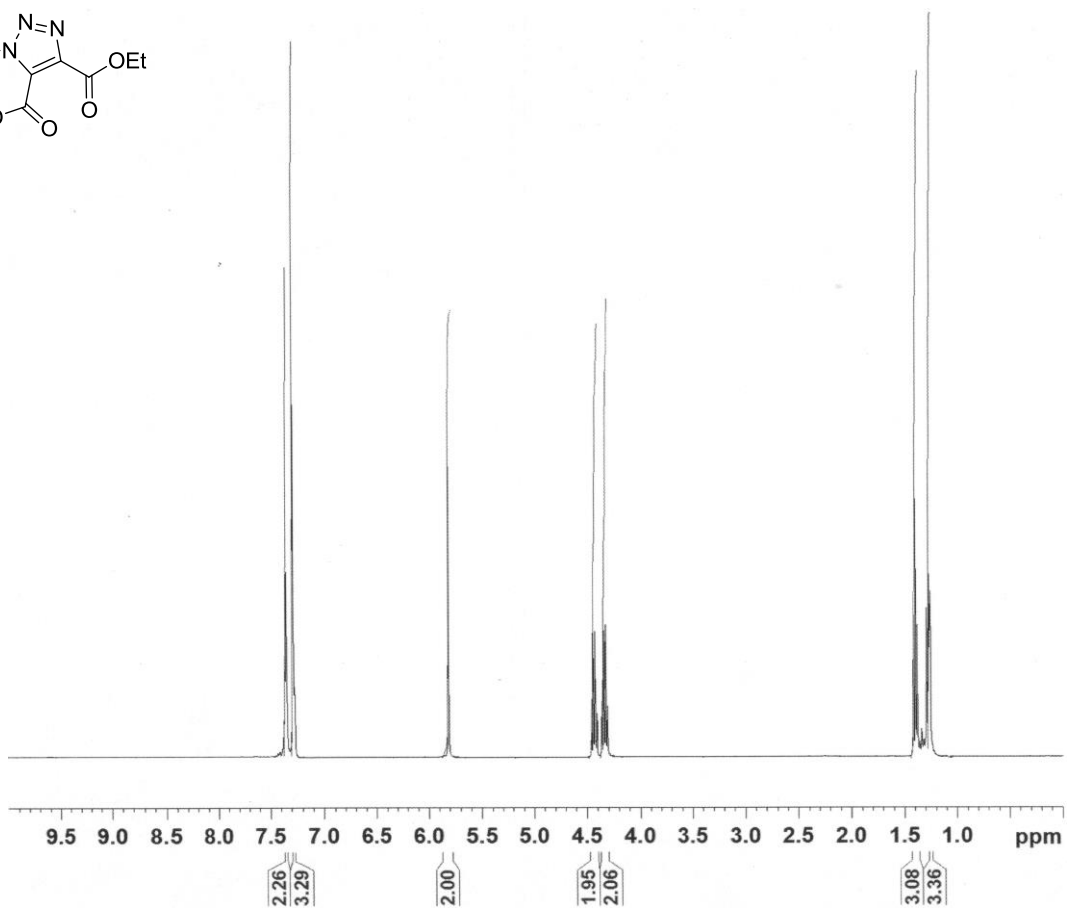
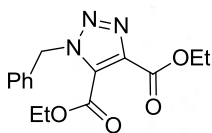
*N*-((1-benzyl-1H-1,2,3-triazol-4-yl)methyl)benzamide, **22**,  $^1\text{H}$  NMR in  $\text{CDCl}_3$ :



*1-Benzyl-4-ferrocenyl-1H-1,2,3-triazole, 23, <sup>1</sup>H NMR in CDCl<sub>3</sub>:*



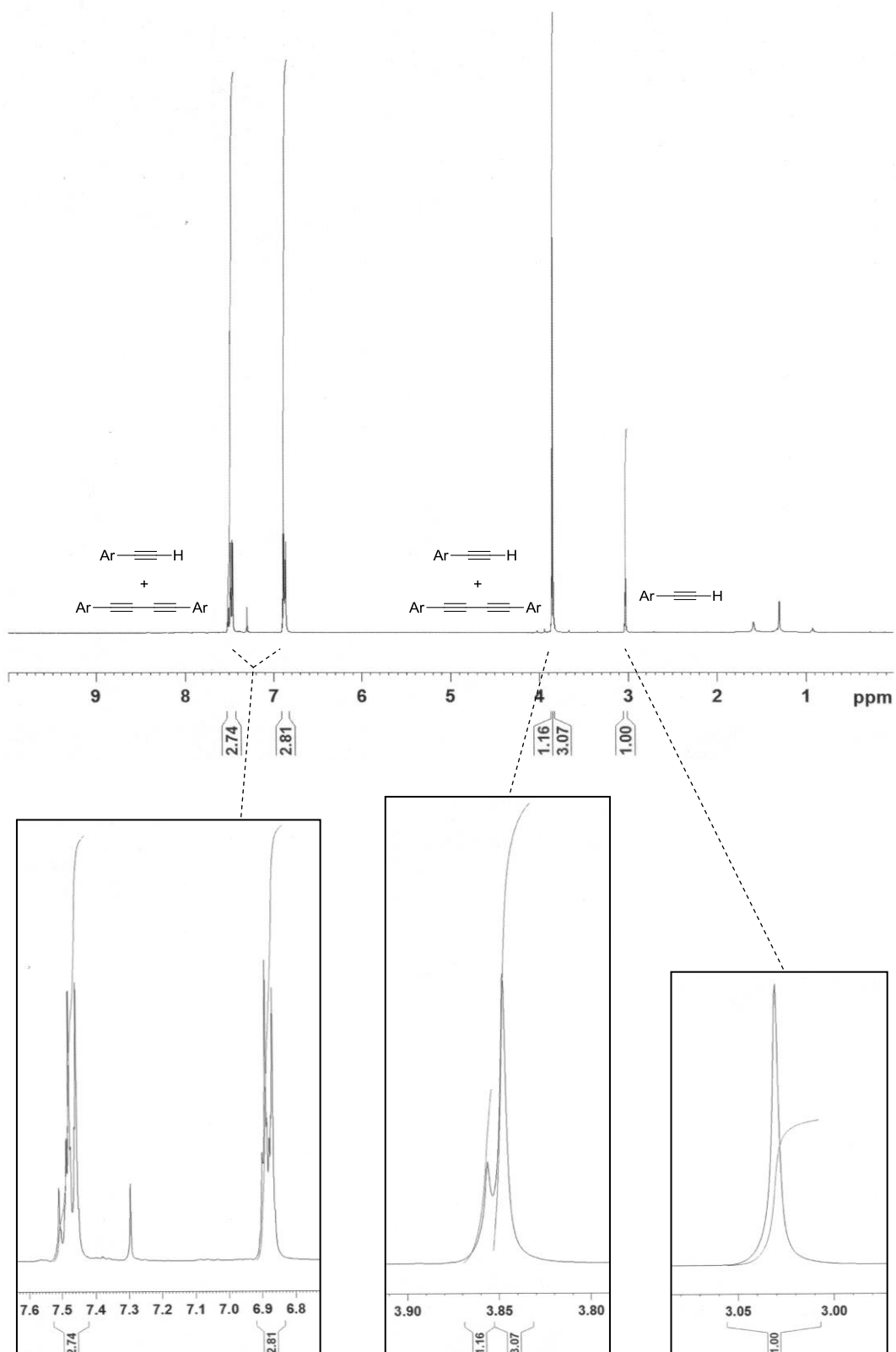
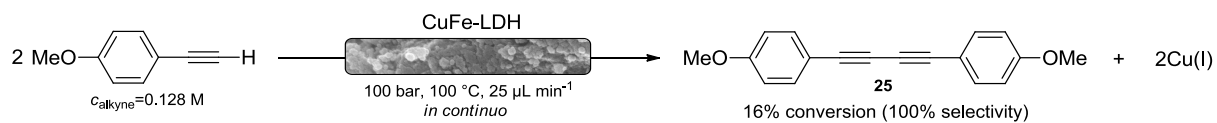
*Diethyl 1-benzyl-1H-1,2,3-triazole-4,5-dicarboxylate, 24, <sup>1</sup>H NMR in CDCl<sub>3</sub>:*



The test reactions to check the efficacy of the individual LDH components ( $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$  and  $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ ) were carried out as follows.

The benzyl azide–phenylacetylene model reaction (1 equiv. azide, 1.5 equiv. alkyne,  $c_{\text{azide}}=0.085 \text{ M}$ ) was repeated in a flask with 10 mol% of  $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$  and then with 10 mol% of  $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$  in 5 mL acetonitrile as solvent (the metal salts were not soluble in  $\text{CH}_2\text{Cl}_2$ ). After 6 hours of stirring at reflux temperature, the mixture was concentrated under vacuum and was worked up with aqueous NaCl solution and  $\text{CH}_2\text{Cl}_2$ . The combined organic layers were dried over  $\text{Na}_2\text{SO}_4$  and concentrated under reduced pressure. No traces of triazole products was found according to  $^1\text{H}$  NMR measurements with either of the metal salts.

Continuous-flow oxidative homocoupling of 4-ethynylanisole (in  $\text{CH}_2\text{Cl}_2$  as solvent) mediated by the Cu(II)Fe(III)-LDH (1 g loaded into the catalyst bed, unused portion), and the  $^1\text{H}$  NMR spectra of the crude product in  $\text{CDCl}_3$  with signal assignments. [S15]





The test reactions to check the efficacy of the individual LDH components ( $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$  and  $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ ) were carried out as follows.

The benzyl azide–phenylacetylene model reaction (1 equiv. azide, 1.5 equiv. alkyne,  $c_{\text{azide}}=0.085 \text{ M}$ ) was repeated in a flask with 10 mol% of  $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$  and then with 10 mol% of  $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$  in 5 mL acetonitrile as solvent (the metal salts were not soluble in  $\text{CH}_2\text{Cl}_2$ ). After 6 hours of stirring at reflux temperature, the mixture was concentrated under vacuum and was worked up with aqueous NaCl solution and  $\text{CH}_2\text{Cl}_2$ . The combined organic layers were dried over  $\text{Na}_2\text{SO}_4$  and concentrated under reduced pressure. No traces of triazole products was found according to  $^1\text{H}$  NMR measurements with either of the metal salts.

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