Nitrones as Dipoles for Rapid Strain-Promoted 1,3-Dipolar Cycloadditions with Cyclooctynes

Supplementary Material

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Materials and Methods

All chemical reagents were purchased from Sigma-Aldrich and were used without further purification. Deuterated solvents were purchased from Cambridge Isotope laboratories. Thin layer chromatography (TLC) was carried out on Analtech Uniplate® silica gel plates (60 Å F254, layer thickness 250μm) using UV light to visualize the course of the reaction. Flash column chromatography was performed using silica gel (60 Å, particle size 40–63 μm). $^1$H NMR and $^{13}$C NMR spectra were obtained using a 400 MHz Bruker NMR spectrometer. Chemical shifts are reported as δ referenced to solvent and coupling constants (J) are reported in Hz. Acyclic nitrones, N-methyl-α-phenylnitrone, N-benzyl-α-phenylnitrone and N-phenyl-α-phenylnitrone were prepared by micelle catalysis1. Cyclic nitrones, 1-pyrroline N-oxide2, 3,4-dihydroisoquinoline N-oxide2, and (±)-4-(tert-butoxycarbonylamino)-3,4-dihydro-2H-pyrrole 1-oxide3,4 were prepared according to literature procedure.

Synthesis of 5,6-didehydro-11,12-dihydronaphthalene[10][11]cyclooctene (2).

![Synthesis of 5,6-didehydro-11,12-dihydronaphthalene[10][11]cyclooctene (2).](image)

Figure S1. Reagents and conditions for synthesis of 5,6-didehydro-11,12-dihydronaphthalene[10][11]cyclooctene.
2,3:6,7-Dibenzo-9-oxabicyclo[3.3.1]nona-2,6-diene\(^5\). An oven dried 100 mL Erlenmeyer flask was charged with phenylacetaldehyde (12.5 g, 104 mmol) and freshly distilled dichloromethane (52 mL). The flask was stoppered under an atmosphere of nitrogen and cooled in an ice bath. To this solution was added iodotrimethylsilane (17.5 mL, 125 mmol) and the reaction was allowed to stand at 5 \(^\circ\)C for 7 days. Upon completion, sodium thiosulfate solution (1 M, 100 mL) and dichloromethane (100 mL) were added, and the mixture was stirred until the iodine color had been discharged. The organic phase was separated, and dried over anhydrous sodium sulfate, filtered and concentrated in vacuo yielding a dark brown oil that was purified by flash column chromatography (dichloromethane:hexanes 6:4 to 100% dichloromethane); \(R_f = 0.72\). The ether (5.78 g, 50%) was obtained as a brittle brown solid. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta 7.17-6.99\) (m, 8H), 5.32 (d, \(J = 6\) Hz, 2H), 3.58 (dd, \(J = 16.2, 6.2\) Hz), 2.70 (d, \(J = 16.2, 6.2\) Hz); \(^13\)C NMR (100 MHz, CDCl\(_3\)) \(\delta 137.8, 131.6, 129.0, 126.8, 125.9, 125.1, 69.5, 36.1\).

5,6,11,12-Tetrahydro-dibenzo[a,e]cycloocten-5-ol\(^5\)\(^6\)\(^7\). Li metal (300 mg, ~10 mmol, from a 25% wt. dispersion in mineral oil) was placed under Ar in a 15 mL pear shaped flask. The Li metal was washed with THF (3 x 1 mL). The resultant metal was suspended in THF (6 mL) and a catalytic amount of naphthalene (15 mg, 0.1 mmol) was added and the mixture was stirred until it turned a dark green colour. A solution of 2,3,6,7-dibenzo-9-oxabicyclo[3.3.1]nona-2,6-diene (225 mg, 1 mmol) in THF (0.4 mL) was added dropwise over 5 min. After stirring the reaction mixture for 2 h, a solution of water (23 \(\mu\)L, 1.3 mmol) in THF (1 mL) was carefully added. After stirring for 15 min, the mixture was quenched by slow dropwise addition of H\(_2\)O (2 mL) and the resulting mixture extracted with Et\(_2\)O (3 x 10 mL). The combined organic phases were washed with brine (10 mL), dried over anhydrous magnesium sulfate, filtered and concentrated to give a crude yellow oil. Purification by column chromatography, eluting dichloromethane:hexanes/8:2 (\(R_f=0.3\)) to 100% dichloromethane, yielded the title compound as a crystalline white solid (87 mg, 39%). \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta 7.23-6.93\) (m, 8H), 5.26 (t, \(J = 8.0\) Hz, 1H), 3.54 (dd, \(J = 14.1, 7.7\)Hz, 1H), 3.43-3.38 (m, 1H), 3.21-3.01 (m, 4H), 2.31-3.01 (m, 4H), 2.25 (br, 1H).
$^{13}$C NMR (100 MHz, CDCl$_3$) δ 141.5, 139.3, 138.0, 137.5, 130.1, 130.0, 129.8, 127.5, 126.9, 126.5, 126.4, 126.0, 74.8, 43.7, 35.1, 33.6.

**11,12-Dihydro-dibenzo[a,e]cycloocten-5(6H)-one**$^{7,8}$: To a stirring solution of 5,6,11,12-tetrahydro-dibenzo[a,e]cycloocten-5-ol (87 mg, 0.39 mmol) in freshly distilled dichloromethane (4 mL), was added crushed 3Å molecular sieves (150 mg) and the resultant mixture was stirred at room temperature under a stream of nitrogen gas for 5 min. Then pyridinium dichromate (293 mg, 0.78 mmol) was added and the reaction was stirred at room temperature for 8 h until the reaction was complete as evident by thin layer chromatography. The reaction was diluted with dichloromethane (25 mL) and was filtered through a plug of celite. The solvent was removed by rotary evaporation providing a brown crude orange solid. Purification by flash column chromatography eluting dichloromethane: hexanes (6:4) (R$_f$ = 0.38) yielded the title compound (81.2 mg, 94%) as a white solid. $^1$H NMR (400 MHz, CDCl$_3$) δ 7.42-6.99 (m, 8H), 4.16 (s, 2H), 3.40-3.25 (m, 4H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 204.4, 138.7, 138.4, 137.8, 133.5, 131.5, 130.8, 130.5, 129.6, 128.0, 127.4, 126.7, 126.5, 51.5, 34.7, 33.7.

$^{5,6}$-Didehydro-11,12-dihydridibenzo[a,e]cyclooctene (2)$^{9,10}$: A stirred solution of 11,12-dihydro-dibenzo[a,e]-cycloocten-5(6H)-one (991 mg, 4.46 mmol) in freshly distilled THF (44 mL) was cooled to −78°C and N-phenyl-trifluoromethanesulphonamide (1.911 g, 5.35 mmol) was added, followed by the slow addition of a potassium bis(trimethylsilyl)amide solution (10.7 mL, 5.35 mmol of a 0.5 M solution in toluene). The resulting mixture was stirred at −78 °C and was allowed to warm to 0 °C. Potassium bis(trimethylsilyl)amide solution (10.7 mL, 5.35 mmol) was then added dropwise over 45 minutes at 0 °C. The reaction was allowed to warm to room temperature over 30 minutes and was concentrated in vacuo. Purification by flash column chromatography, eluting Hexanes: Dichloromethane (97:3 to 95:5), yielded 5,6-dihydro-11,12-didehydridibenzo[a,e]cyclooctyne (544 mg, 60%) as an off-white solid. $^1$H NMR (400 MHz, CDCl$_3$) δ 7.39-7.27 (m, 8H), 3.38-3.29 (m, 2H),
2.51-2.41 (m, 2H). $^{13}$C NMR (100 MHz, CDCl$_3$) δ 153.5, 129.35, 127.6, 126.5, 126.1, 123.9, 111.5, 36.4.

**General procedure for strain-promoted 1,3-dipolar cycloadditions of nitrones with alkynes.**

To a stirring solution of dibenzo[cyclooctyne], 2 (10.2 mg, 0.05 mmol) in toluene (500 μL) was added nitrone, 1a-n (0.05 mmol). Reactions were stirred open to air and were monitored by thin layer chromatography for disappearance of the nitrone. Upon completion, the solvent was removed under reduced pressure and the crudes were purified by flash column chromatography to afford pure isoxazoline products.

**Characterization Data for New Compounds**

**Compound 3a**

Purified by eluting hexanes:EtOAc (9:1, R$_f$ = 0.34) and was obtained as a white solid (19.0 mg, 95%). $^1$H NMR (400 MHz, CDCl$_3$) δ 7.96 (d, $J$ = 8.3Hz, 2H), 7.52-7.50 (m, 1H), 7.45 (d, $J$ = 8.3Hz, 2H), 7.18-7.04 (m, 7H), 5.09 (s, 1H), 3.89 (s, 3H), 3.41-3.33 (m, 1H), 3.15 (s, 3H), 3.19-2.98 (m, 2H), 2.88-2.81 (m, 1H). $^{13}$C NMR (100 MHz, CDCl$_3$) δ 167.0, 147.3, 146.2, 141.1, 139.1, 131.9, 131.0, 129.9, 129.8, 129.7, 129.3, 128.8, 127.9, 127.5, 127.2, 126.5, 125.8, 125.5, 109.8, 80.3, 52.0, 47.0, 36.7, 35.7, 32.8. HRMS calcd. for C$_{26}$H$_{23}$NO$_3$ [M+1]$^+$ 398.1756, found 398.1744.

**Compound 3b**

Purified by eluting hexanes:EtOAc (96:4, R$_f$ = 0.41) and was obtained as a white solid (15.2 mg, 90%). $^1$H NMR (400 MHz, CDCl$_3$) δ 7.54-6.98 (m, 13H), 5.03 (m, 1H), 3.45-3.38 (m, 1H), 3.23-3.08 (m, 1H), 3.13 (s, 3H), 3.00-2.93 (m, 1H). $^{13}$C NMR (100 MHz, CDCl$_3$) δ 147.3, 141.1, 138.9, 132.4, 131.0, 129.8, 129.6,
128.6, 128.6, 127.9, 127.8, 127.6, 127.0, 126.8, 125.7, 125.5, 110.2, 80.9, 47.1, 36.9, 32.9. HRMS calcd. for C_{24}H_{21}NO [M+1]^+ 340.1701, found 340.1660.

**Compound 3c**

Purified by eluting hexanes:EtOAc (95:5, R_f = 0.43) and was obtained as a white solid (19.0 mg, 94%). \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.53-7.51 (m, 1H), 7.32 (d, \(J = 8.7\)Hz, 2H), 7.18-6.94 (m, 7H), 6.84 (d, \(J = 8.7\)Hz, 2H), 4.99 (s, 1H), 3.78 (s, 3H), 3.46-3.39 (m, 1H), 3.26-3.08 (m, 2H), 3.10 (s, 3H), 3.05-2.98 (m, 1H). \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 159.1, 147.3, 141.0, 138.8, 132.5, 131.0, 129.8, 129.6, 128.5, 128.2, 128.0, 127.7, 126.9, 125.7, 125.5, 114.0, 110.3, 80.5, 55.2, 37.0, 32.9. HRMS calcd. for C\(_{25}\)H\(_{23}\)NO\(_2\) [M+1]^+ 370.1807, found 370.1721.

**Compound 3d**

Purified eluting hexanes:EtOAc (9:1, R_f = 0.18) and was obtained as a yellow solid (22.3 mg, 97%). \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 8.11 (d, \(J = 8.8\) Hz, 2H), 7.55-7.09 (m, 15H), 5.36 (s, 1H), 4.72 (d, \(J = 12.7\)Hz, 1H), 4.33 (d, \(J = 12.7\)Hz, 1H), 3.39-3.32 (m, 1H), 3.14-2.85 (m, 2H), 2.84-2.78 (m, 1H). \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 148.5, 148.3, 147.2, 141.1, 139.2, 135.7, 131.6, 131.0, 130.0, 129.3, 129.3, 129.1, 128.7, 127.9, 127.4, 127.3, 127.2, 126.0, 125.6, 123.7, 109.5, 76.3, 62.9, 36.5, 33.0. HRMS calcd. for C\(_{30}\)H\(_{24}\)N\(_2\)O\(_3\) [M+1]^+ 461.1865, found 461.1866.

**Compound 3e**

Purified eluting hexanes:EtOAc (9:1, R_f = 0.31) and was obtained as a white solid (19.2 mg, 96%). \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.93 (d, \(J = 8.3\)Hz, 2H), 7.53-7.02 (m, 15H), 5.30 (s, 1H), 4.67 (d, \(J = 12.8\)Hz, 1H), 4.32 (d, \(J = 12.8\)Hz, 1H), 3.89 (s, 3H), 3.39-3.32 (m, 1H), 3.15-2.98 (m, 2H), 2.87-2.80 (m, 1H). \(^{13}\)C NMR (100 MHz, CDCl\(_3\))

S-7
δ 167.0, 148.0, 146.4, 141.1, 139.2, 136.0, 132.0, 131.0, 130.0, 129.8, 129.3, 129.2, 128.8, 128.6, 128.0, 127.8, 127.6, 127.2, 126.6, 125.8, 125.6, 110.0, 76.9, 63.0, 52.0, 36.6, 33.0. HRMS calcd. for C$_{32}$H$_{27}$NO$_3$ [M+1]$^+$ 474.2069, found 474.2080.

**Compound 3f.**

Purified eluting hexanes:EtOAc (9:1, R$_f$ = 0.55) and was obtained as an off white solid (24.2 mg, 98%). $^1$H NMR (400 MHz, CDCl$_3$) δ 7.52-7.00 (m, 17H), 5.21 (s, 1H), 4.65 (d, $J$ = 12.8 Hz, 1H), 4.30 (d, $J$ = 12.8 Hz, 1H), 3.42-3.35 (m, 1H), 3.20-3.04 (m, 2H), 2.95-2.88 (m, 1H). $^{13}$C NMR (100 MHz, CDCl$_3$) δ 148.0, 141.1, 140.5, 139.1, 136.0, 132.1, 131.6, 131.0, 130.0, 129.8, 129.3, 128.8, 128.6, 128.5, 127.9, 127.8, 127.7, 127.1, 125.8, 125.6, 121.3, 110.0, 63.0, 36.7, 33.0. HRMS calcd. for C$_{30}$H$_{24}$NOBr [M+1]$^+$ 494.1120, found 494.1098.

**Compound 3g**

Purified eluting hexanes:EtOAc (9:1, R$_f$ = 0.26) and was obtained as a off white solid (21.7 mg, 99%). $^1$H NMR (400 MHz, CDCl$_3$) δ 7.55-7.05 (m, 17H), 5.30 (s, 1H), 4.70 (d, $J$ = 12.7 Hz, 1H), 4.30 (d, $J$ = 12.7 Hz, 1H), 3.39-3.32 (m, 1H), 3.15-2.96 (m, 2H), 2.87-2.80 (m, 1H). $^{13}$C NMR (100 MHz, CDCl$_3$) δ 148.2, 146.5, 141.0, 139.2, 135.7, 132.3, 131.6, 131.0, 129.9, 129.3, 129.0, 128.6, 127.9, 127.9, 127.3, 127.1, 125.9, 125.6, 118.9, 111.1, 109.5, 76.5, 62.8, 36.5, 32.9. LRMS calcd. for C$_{31}$H$_{24}$N$_2$O [M+1]$^+$ 455.4, found 455.4.
Compound 3h

Purified eluting hexanes:EtOAc (9:1, R\textsubscript{f} = 0.43) and was obtained as a slow crystallizing white solid (23.2 mg, 94%).

\(^1\)H NMR (400 MHz, CDCl\textsubscript{3}) \(\delta 7.53-6.96\) (m, 17H), 5.22 (s, 1H), 4.62 (d, \(J = 12.9\) Hz, 1H), 4.31 (d, \(J = 12.9\) Hz, 1H), 3.45-3.38 (m, 1H), 3.25-3.09 (m, 2H), 3.01-2.94 (m, 1H), 2.31 (s, 3H). \(^13\)C NMR (100 MHz, CDCl\textsubscript{3}) \(\delta 147.9, 141.0, 138.9, 138.4, 137.1, 136.3, 132.6, 131.0, 129.9, 129.6, 129.4, 129.2, 128.5, 128.5, 128.1, 127.9, 127.6, 126.9, 126.9, 125.7, 125.5, 110.5, 63.1, 36.9, 33.0, 21.1. HRMS calcd. for C\textsubscript{31}H\textsubscript{27}NO [M+1]\(^+\) 430.2171, found 430.2108.

Compound 3i

Purified eluting hexanes:EtOAc (9:1, R\textsubscript{f} = 0.33) and was obtained as a white solid (19.2 mg, 92%). \(^1\)H NMR (400 MHz, CDCl\textsubscript{3}) \(\delta 7.53-6.96\) (m, 18H), 5.24 (s, 1H), 4.63 (d, \(J = 12.9\) Hz, 1H), 4.31 (d, \(J = 12.9\) Hz, 1H), 3.43-3.35 (m, 1H), 3.22-3.05 (m, 2H), 2.97-2.90 (m, 1H). \(^13\)C NMR (100 MHz, CDCl\textsubscript{3}) \(\delta 147.9, 141.4, 141.0, 139.0, 136.3, 132.5, 131.0, 130.0, 129.7, 129.4, 128.6, 128.5, 128.5, 127.9, 127.6, 127.4, 127.0, 125.7, 125.5, 110.4, 63.1, 36.8, 33.0. HRMS calcd. for C\textsubscript{30}H\textsubscript{25}NO [M+1]\(^+\) 416.2014, found 416.1977.

Compound 3j

Purified eluting hexanes:EtOAc (9:1, R\textsubscript{f} = 0.44) and was obtained as a white solid (21.0 mg, 95%). \(^1\)H NMR (400 MHz, CDCl\textsubscript{3}) \(\delta 7.52-6.81\) (m, 17H), 5.21 (s, 1H), 4.61 (d, \(J = 12.9\) Hz, 1H), 4.30 (d, \(J = 12.9\) Hz, 1H), 3.78 (s, 3H), 3.45-3.37 (m, 1H), 3.26-3.09 (m, 2H), 3.03-2.96 (m, 1H). \(^13\)C NMR (100 MHz, CDCl\textsubscript{3}) \(\delta 159.0, 147.9, 141.0, 138.9, 136.3, 133.6, 132.6, 131.0, 129.9, 129.6, 129.4, 128.5, 128.5, 128.2, 128.0, 127.8, 127.6, 126.9, 125.7, 125.5, 113.9, 110.4, 63.0, 55.2, 36.9, 33.0. HRMS calcd. for C\textsubscript{31}H\textsubscript{27}NO\textsubscript{2} [M+1]\(^+\) 446.1946, found 446.1952.
Compound 3k

Purified eluting hexanes:EtOAc / 95:5 (Rf = 0.52) and was obtained as an off white solid (19.3 mg, 96%). ¹H NMR (400 MHz, CDCl₃) δ 7.86-6.80 (m, 18H), 6.73 (s, 1H), 3.94-3.83 (m, 2H), 3.25-3.13 (m, 2H). ¹³C NMR (100 MHz, CDCl₃) δ 147.3, 141.5, 139.6, 139.5, 138.5, 131.0, 130.8, 130.1,129.7, 129.1, 128.9, 128.8, 128.3, 127.0, 126.3, 126.1, 125.7, 121.0, 119.6, 117.8, 97.9, 35.6, 35.3. LRMS calcd. for C₂₉H₂₃NO [M+H]⁺ 402.4, found 402.4.

Compound 3l

Purified eluting hexanes:EtOAc (7:3, Rf = 0.33) and was obtained as a white solid (14.4 mg, 100%). ¹H NMR (400 MHz, CDCl₃) δ 7.47-7.08 (m, 8H), 5.21 (dd, J = 7.3 Hz, 2.2 Hz, 1H), 3.62-3.49 (m, 2H), 3.47-3.40 (m, 1H), 3.32-3.25 (m, 1H), 3.17-3.09 (m, 1H), 2.99-2.92 (m, 1H). 2.07-1.82 (m, 4H). ¹³C NMR (100 MHz, CDCl₃) δ 148.0, 140.8, 138.2, 132.6, 130.9, 129.7, 129.5, 128.4, 128.0, 127.0, 126.0, 125.6, 109.1, 74.9, 59.9, 36.9, 33.3, 31.3, 22.9. LRMS calcd. for C₂₀H₁₉NO [M+1]⁺ 290.3, found 290.3.

(±)-Compound 3m

Purified eluting hexanes:EtOAc (6:4, Rf = 0.56) and was obtained as a white crystalline solid (20.2 mg, 100%). ¹H NMR (400 MHz, CDCl₃) δ 7.43-7.08 (m, 8H), 4.94 (s, 1H), 4.76 (d, J = 1.9Hz, 1H), 4.07 (m, 1H), 3.65-3.56 (m, 1H), 3.55-3.47 (m, 2H), 3.43-3.36 (m, 1H), 3.17-3.09 (m, 1H), 3.00-2.94 (m, 1H), 2.37-2.32 (m, 1H), 1.91-1.88 (m, 1H), 1.37 (s, 9H). ¹³C NMR (100 MHz, CDCl₃) δ 154.8, 149.0, 140.5, 138.4, 131.6, 130.9, 129.9, 129.4, 128.7, 128.1, 127.5, 127.2, 126.1, 125.6, 57.8, 36.7, 33.3, 30.6, 28.3. LRMS calcd. for C₂₅H₂₈N₂O₃ [M+1]⁺ 405.4, found 405.4.
Compound 3n

Purified eluting hexanes:EtOAc (9:1, Rf = 0.23) and was obtained as a white crystalline solid (17.3 mg, 98%). 1H NMR (400 MHz, CDCl₃) δ 7.50-7.48 (m, 1H), 7.17-7.06 (m, 9H), 6.96-6.92 (m, 1H), 6.58 (d, J = 7.7 Hz, 1H), 5.97 (s, 1H), 3.93-3.89 (m, 1H), 3.36-3.22 (m, 3H), 3.10-3.03 (m, 1H), 2.85-2.75 (m, 3H). 13C NMR (100 MHz, CDCl₃) δ 148.3, 141.3, 138.9, 135.1, 133.6, 132.1, 131.0, 130.0, 129.4, 129.3, 128.8, 128.3, 127.8, 127.2, 126.6, 126.4, 126.0, 125.6, 125.5, 111.3, 71.1, 50.8, 36.7, 32.7, 24.5. HRMS calcd. for C₂₅H₂₁NO [M+1]⁺ 352.1701, found 352.1683.
Kinetics measurements of 1b, 1i and 1n with 2

Figure S2 (A, B, C and D). The appropriate nitrone or benzyl azide and 2 were predissolved in C_6D_6 and mixed at equimolar concentrations of ~50mM. Percent conversion was monitored both by disappearance of starting materials and by appearance of product as determined by integration at multiple chemical shifts in the ^1H NMR spectrum. No other products were detected by ^1H NMR and all reactions were performed in triplicate. Second order rate constants in units of M^{-1}s^{-1} were determined by plotting 1/[nitrone] or 1/[azide] versus time and subsequently using analysis by linear regression. The second order rate constant $k_2$ (M^{-1}s^{-1}) corresponds to the determined slope. A representative ^1H NMR spectrum at ~50 to 75% conversion is included.

A) 1b with 2

![Kinetics: 1b with 2](image)

$$\begin{align*}
y &= 0.1299x + 14.335 \\
R^2 &= 0.9945 \\
k_2 &= 0.13 \pm 0.01 \text{ M}^{-1}\text{s}^{-1}
\end{align*}$$
B) 1i with 2

Kinetics: 1i with 2

\[
y = 0.0878x + 18.091 \\
R^2 = 0.9993 \\
k_2 = 0.088 \pm 0.004 \text{ M}^{-1}\text{s}^{-1}
\]

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C) **1n with 2**

**Kinetics: 1n with 2**

\[
y = 1.4768x + 20 \\
R^2 = 0.9959 \\
k_2 = 1.48 \pm 0.11 \text{ M}^{-1}\text{s}^{-1}
\]

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D) Benzyl azide with dibenzocyclooctyne

![Graph showing the kinetics of benzyl azide with dibenzocyclooctyne]

- \( y = 0.0615x + 19.389 \)
- \( R^2 = 0.9998 \)
- \( k = 0.062 \pm 0.006 \text{ M}^{-1}\text{s}^{-1} \)
$^1$H and $^{13}$C Spectra
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Date:
10 Oct 2009

Document Title:

Spectrum Title:

1H-CPMG 200 ms

Frequency (MHz):

100.61

Original Points Count:

32759

Actual Points Count:

32759

Acquisition Time (sec):

20

Spectral Width (ppm):

2000

Pulse Program:

Unknown

ppm (01)
Data:
12-Oct-2009
Document Title:
Hi-Co FSPE
Spectrum Title:

Frequency (MHz):
(1) 400.132
Organic Frame Count:
(1) 112834
Actual Points Count:
(1) 32768
Accumulation Time (sec):
(1) 1.9239
Spectral Width (ppm):
(1) 122.86
Pulse Program:
2H520
Temperature:
303 K
Number of Scans:
52
Avg. Scan:
Mon Oct 05 06:56:58 PM

S-32
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Date:
12-Oct-2010

Document Title:
13C NMR (500 MHz)

Spectrum Title:
emt588 p-CO2Me

Frequency (MHz):
500

Number of Scans:
1000

Acq. Date:
Tue Oct 26 01:07:16 AM

S-33
References