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Supporting Information

for

Copper(I)-catalyzed tandem reaction: synthesis of 1,4-disubstituted 1,2,3-triazoles from alkyl diacyl peroxides, azidotrimethylsilane, and alkynes

Muhammad Israr, Changqing Ye, Munira Taj Muhammad, Yajun Li and Hongli Bao

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Detailed experimental procedures and characterization data for all new compounds

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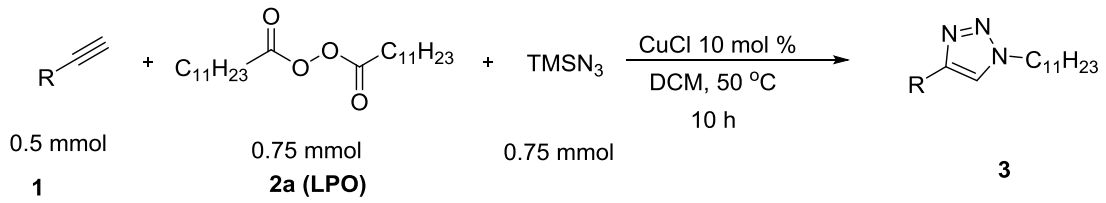
Materials and methods

All reactions were carried out in flame-dried glassware with magnetic stirring unless otherwise indicated. Commercially available reagents were used without further purification. Solvents were dried by distillation from sodium or CaH_2. Solutions and liquids were transferred with the help of syringe. Reactions were monitored by thin-layer chromatography. ^{1}H and ^{13}C NMR spectra were recorded on Bruker-BioSpin AVANCE III HD. Data for ^{1}H NMR spectra are reported relative to chloroform as an internal standard (7.26 ppm) and are reported as follows: chemical shift (ppm), multiplicity, coupling constant (Hz), and integration. Data for ^{13}C NMR spectra are reported relative to chloroform as an internal standard (77.16 ppm) and are reported in terms of chemical shift (ppm). All melting points were determined on a Beijing Science Instrument Dianguang Instrument Factory XT4B melting point apparatus and are uncorrected. IR data were obtained from Bruker VERTEX 70. HRMS data were recorded on Agilent Technologies 6224 TOF LC/MS.

General procedure for the synthesis of triazoles with LPO (for Scheme 2)



0.5 mmol
1

0.75 mmol
2a (LPO)

0.75 mmol

3

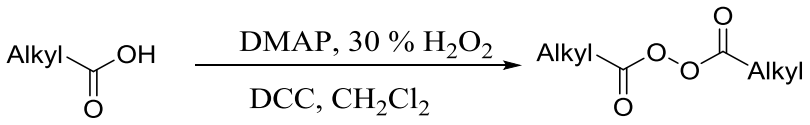
Procedure:

To a flame-dried Schlenk tube containing a magnetic bar, terminal alkyne **1** (0.5 mmol), diacyl peroxide **2a** (0.75 mmol), $TMSN_3$ (90.4 mg, 0.75 mmol), CuCl (4.9 mg, 0.05 mmol) and CH_2Cl_2 (2 mL) were added respectively. The reaction mixture was stirred vigorously for 10 hours at 50 °C. Then, the reaction mixture was cooled to room temperature, poured into saturated sodium bicarbonate solution (25 mL) and extracted with CH_2Cl_2 (3 × 25 mL). After drying over $MgSO_4$, the solvent was removed under reduced pressure by rotary evaporator, the residue was purified by column chromatography on silica gel (PE/EA) to afford **3**.

Synthesis of alkyl diacyl peroxides

Diacyl peroxides were synthesized according to the reported literature.[1,2]

Diacyl peroxides have potentials to explode. Any diacyl peroxide involved in the reaction (as product or substrate) should be carried out with precautions!

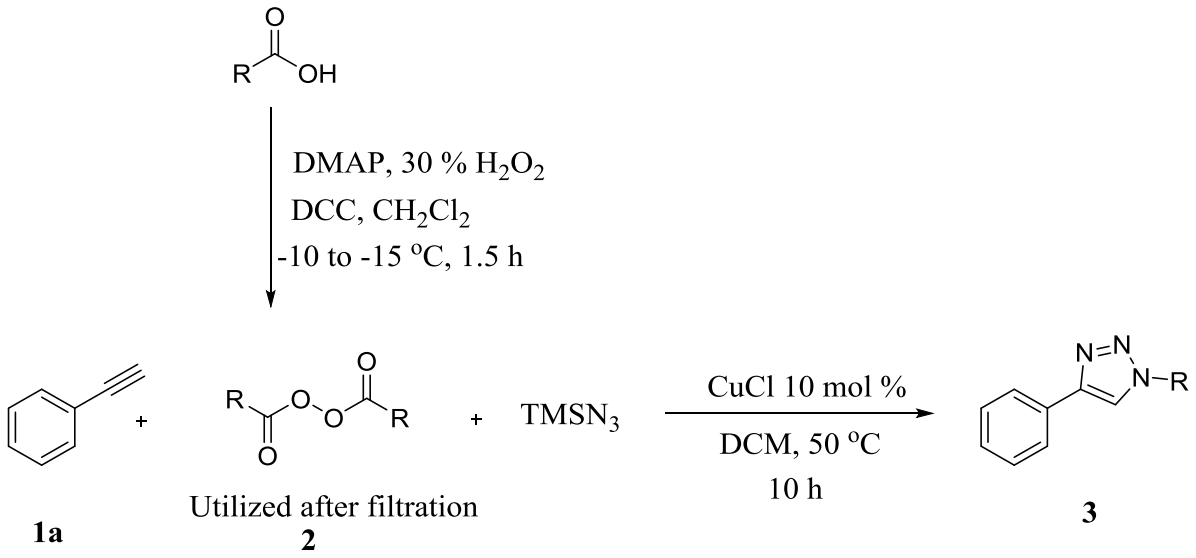


2

4-Dimethylaminopyridine (DMAP, 0.224g, 0.6 mmol), acid (6 mmol), hydrogen peroxide (30 % v/v in H_2O, 7.5 mmol) and CH_2Cl_2 were added to a 50 mL round bottom flask respectively. The reaction mixture was

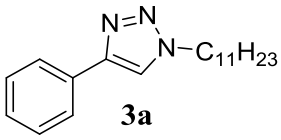
cooled to −15 °C for about 10 min and dicyclohexylcarbodiimide (DCC, 6.72 mmol) was added. Then, the mixture was stirred at −10 to −15°C for 1.5 hours. The solution was filtered with petroleum ether and dried over $MgSO_4$. After filtration the solvent was removed under reduced pressure at 10 to 15 °C. The residue (peroxide **2**) was used without further purification.

General procedure for the synthesis of triazoles with alkyl diacyl peroxides (for Scheme 3)



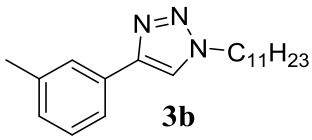
To an oven-dried Schlenk tube equipped with magnetic stirring bar, phenylacetylene (**1a**, 52.6 mg, 0.5 mmol), the above diacyl peroxide **2** (0.75 mmol), $TMSN_3$ (90.4 mg, 0.75 mmol), CuCl (4.9 mg, 0.05 mmol) and CH_2Cl_2 (2 mL) were added, respectively. The reaction mixture was stirred at 50 °C for 10 hours. Then, the reaction mixture was cooled to room temperature, poured into saturated sodium bicarbonate solution (25 mL) and extracted with CH_2Cl_2 (3 × 25 mL). After drying over $MgSO_4$, the solvent was removed under reduced pressure, and the residue was purified by column chromatography on silica gel (PE/EA) to afford **3**.

Characterization data for the products



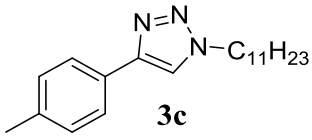
3a

Following the general procedure for Scheme 2, **3a** was obtained as a yellowish solid (145 mg, 97% yield); mp. 85 – 86 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87 – 7.77 (m, 2H), 7.74 (s, 1H), 7.39 (dd, *J* = 8.3, 6.8 Hz, 2H), 7.35 – 7.26 (m, 1H), 4.34 (t, *J* = 7.2 Hz, 2H), 1.90 (t, *J* = 7.1 Hz, 2H), 1.32 – 1.21 (m, 16H), 0.87 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.66, 130.79, 128.82, 128.04, 125.68, 119.53, 50.42, 31.91, 30.36, 29.58, 29.54, 29.41, 29.33, 29.04, 26.51, 22.70, 14.14; IR (KBr): υ 2551, 2917, 2847, 1652, 1558, 1464, 1216, 1078 cm^{-1}; HRMS (ESI) calcd for $[C_{19}H_{29}N_3Na]^+$ ($[M+Na]^+$): 322.2254, found: 322.2254.



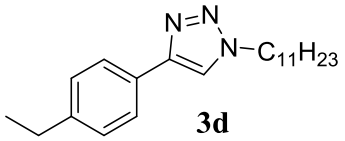
3b

Following the general procedure for Scheme 2, **3b** was obtained as a dull-white solid (130 mg, 83% yield); mp. 66 – 67 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.72 (s, 1H), 7.69 (d, *J* = 2.0 Hz, 1H), 7.60 (d, *J* = 7.7 Hz, 1H), 7.31 (t, *J* = 7.6 Hz, 1H), 7.14 (d, *J* = 7.6 Hz, 1H), 4.38 (t, *J* = 7.2 Hz, 2H), 2.40 (s, 3H), 1.94 (t, *J* = 7.2 Hz, 2H), 1.34 – 1.25 (m, 16H), 0.87 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.96, 138.63, 130.74, 128.96, 128.84, 126.51, 122.91, 119.46, 50.56, 32.01, 30.49, 29.64, 29.51, 29.42, 29.15, 26.63, 22.80, 21.56, 14.24; IR (KBr): υ 2952, 2916, 2847, 1699, 1541, 1466, 1220, 1024 cm^{-1}; HRMS (ESI) calcd for $[C_{20}H_{31}N_3Na]^+$ ($[M+Na]^+$): 336.2410, found: 336.2409.



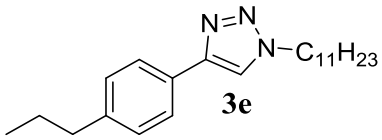
3c

Following the general procedure for Scheme 2, **3c** was obtained as a white solid (140 mg, 86% yield); mp. 86 –87 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.78 – 7.65 (m, 3H), 7.21 (d, *J* = 7.8 Hz, 2H), 4.35 (t, *J* = 7.2 Hz, 2H), 2.37 (s, 3H), 1.91 (t, *J* = 7.1 Hz, 2H), 1.33 – 1.22 (m, 16H), 0.87 (t, *J* = 6.7 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.82, 137.92, 129.55, 128.01, 125.65, 119.18, 50.46, 31.96, 30.43, 29.63, 29.59, 29.46, 29.37, 29.09, 26.58, 22.74, 21.34, 14.18; IR (KBr): υ 2951, 2917, 2847, 1652, 1541, 1461, 1216, 1075 cm^{-1}; HRMS (ESI) calcd for $[C_{20}H_{31}N_3Na]^+$ ($[M+Na]^+$): 336.2410, found: 336.2412.



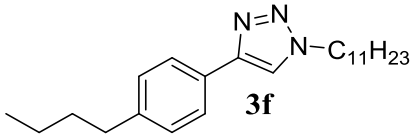
3d

Following the general procedure for Scheme 2, **3d** was obtained as a light brown solid (125 mg, 76% yield); mp. 69 – 70 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.76 (d, *J* = 8.2 Hz, 2H), 7.71 (s, 1H), 7.28 (d, *J* = 2.5 Hz, 2H), 4.39 (t, *J* = 7.2 Hz, 2H), 2.69 (q, *J* = 7.6 Hz, 2H), 1.95 (t, *J* = 7.1 Hz, 2H), 1.35 – 1.25 (m, 19H), 0.91 – 0.87 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 146.95, 143.44, 127.45, 127.28, 124.83, 118.19, 49.55, 31.02, 29.50, 28.69, 28.65, 28.52, 28.43, 28.16, 27.81, 25.64, 21.81, 14.66, 13.24; IR (KBr): υ 2953, 2917, 2847, 1699, 1541, 1457, 1217, 1076 cm^{-1}; HRMS (ESI) calcd for $[C_{21}H_{33}N_3Na]^+$ ($[M+Na]^+$): 350.2567, found: 350.2563.



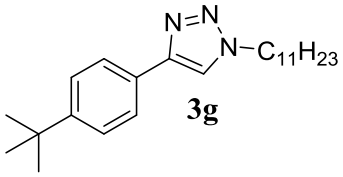
3e

Following the general procedure for Scheme 2, **3e** was obtained as a yellow solid (133 mg, 78% yield); mp. 61 –62 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74 (d, *J* = 8.2 Hz, 2H), 7.70 (s, 1H), 7.23 (d, *J* = 8.1 Hz, 2H), 4.38 (t, *J* = 7.2 Hz, 2H), 2.63 (t, , *J* = 7.4 Hz, 2H), 1.94 (t, *J* = 7.3 Hz, 2H), 1.69 – 1.63 (m, 2H), 1.33 – 1.25 (m, 16H), 0.95 (t, *J* = 7.3 Hz, 3H), 0.87 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.96, 142.88, 129.06, 128.26, 125.73, 119.19, 50.55, 37.95, 32.02, 30.49, 29.68, 29.65, 29.52, 29.43, 29.15, 26.64, 24.62, 22.80, 14.24, 13.92; IR (KBr): υ 2956, 2921, 2851, 1733, 1558, 1465, 1363, 1222 cm^{-1}; HRMS (ESI) calcd for $[C_{22}H_{35}N_3Na]^+$ ($[M+Na]^+$): 364.2723, found: 364.2723.



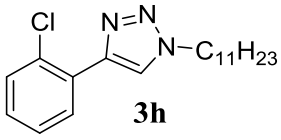
3f

Following the general procedure for Scheme 2, **3f** was obtained as a yellow solid (165 mg, 85% yield); mp. 73 –74 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.79 – 7.65 (m, 3H), 7.23 (d, *J* = 8.1 Hz, 2H), 4.37 (t, *J* = 7.4 Hz, 2H), 2.63 (t, *J* = 7.4 Hz, 2H), 1.93 (t, *J* = 6.9 Hz, 2H), 1.62 (p, *J* = 7.5 Hz, 2H), 1.39 – 1.24 (m, 18H), 0.90 (m, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.91, 143.06, 128.97, 128.22, 125.70, 119.19, 50.50, 35.54, 33.67, 31.99, 30.46, 29.66, 29.62, 29.49, 29.41, 29.12, 26.60, 22.78, 22.44, 14.21, 14.06; IR (KBr): υ 2953, 2817, 2847, 1709, 1463, 1216, 1077 cm^{-1}; HRMS (EI) calcd for $[C_{23}H_{38}N_3][M+H]^+$: 356.3060, found: 356.3061.



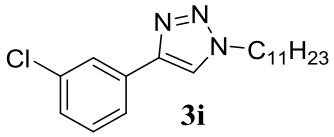
3g

Following the general procedure for Scheme 2, **3g** was obtained as a yellow solid (118 mg, 66% yield); mp. 47 –48 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.76 (d, *J* = 8.4 Hz, 2H), 7.70 (s, 1H), 7.45 (d, *J* = 8.4 Hz, 2H), 4.38 (t, *J* = 7.2 Hz, 2H), 1.95 (t, *J* = 7.1 Hz, 2H), 1.36 (d, *J* = 36.4 Hz, 25H), 0.87 (t, *J* = 6.7 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 151.30, 147.84, 128.05, 125.87, 125.56, 119.23, 50.54, 34.79, 32.02, 31.43, 30.49, 29.69, 29.64, 29.52, 29.43, 29.15, 26.63, 22.81, 14.24; IR (KBr): υ 2953, 2917, 2847, 1684, 1541, 1457, 1217, 1076 cm^{-1}; HRMS (ESI) calcd for $[C_{23}H_{37}N_3Na]^+$ ($[M+Na]^+$): 378.2880, found: 378.2881.



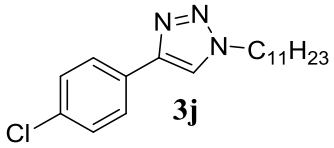
3h

Following the general procedure for Scheme 2, **3h** was obtained as a brown solid (130 mg, 78% yield); mp. 56 –57 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.26 (dd, J = 7.8, 1.8 Hz, 1H), 8.16 (s, 1H), 7.45 (dd, *J* = 8.0, 1.3 Hz, 1H), 7.37 (td, *J* = 7.6, 1.4 Hz, 1H), 7.27 (dd, J = 7.7, 5.9 Hz, 1H), 4.42 (t, *J* = 7.3 Hz, 2H), 1.94 (t, *J* = 7.3 Hz, 2H), 1.35 – 1.26 (m, 16H), 0.88 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.04, 131.27, 130.30, 129.95, 129.53, 129.06, 127.31, 123.22, 50.62, 32.02, 30.48, 29.69, 29.64, 29.52, 29.44, 29.14, 26.63, 22.81, 14.25; IR (KBr): υ 2920, 2853, 1715, 1507, 1489, 1058, 759 cm^{-1}; HRMS (ESI) calcd for $[C_{19}H_{28}ClN_3Na]^+$ ($[M+Na]^+$): 356.1864, found: 356.1866.



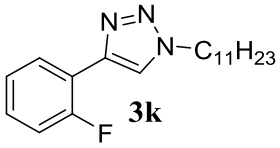
3i

Following the general procedure for Scheme 2, **3i** was obtained as a white solid (151 mg, 91% yield); mp. 79 – 80 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.82 (d, *J* = 2.0 Hz, 1H), 7.75 (s, 1H), 7.72 (d, *J* = 7.6 Hz, 1H), 7.34 (t, *J* = 7.8 Hz, 1H), 7.29 (d, *J* = 8.0 Hz, 1H), 4.39 (t, *J* = 7.3 Hz, 2H), 1.94 (t, *J* = 7.1 Hz, 2H), 1.34 – 1.25 (m, 16H), 0.87 (t, *J* = 6.7 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 146.55, 134.89, 132.64, 130.23, 128.15, 125.86, 123.85, 119.89, 50.65, 32.00, 30.44, 29.66, 29.62, 29.49, 29.41, 29.11, 26.60, 22.79, 14.22; IR (KBr): υ 2917, 2847, 1670, 1575, 1489, 1070, 792 cm^{-1}; HRMS (ESI) calcd for $[C_{19}H_{28}ClN_3Na]^+$ ($[M+Na]^+$): 356.1864, found: 356.1863.



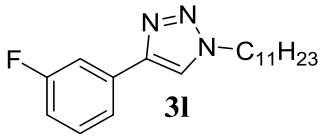
3j

Following the general procedure for Scheme 2, **3j** was obtained as a light yellow solid (153 mg, 92% yield); mp. 97 – 98 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.76 (d, *J* = 8.5 Hz, 2H), 7.73 (s, 1H), 7.38 (d, *J* = 8.5 Hz, 2H), 4.38 (t, *J* = 7.2 Hz, 2H), 1.93 (t, *J* = 7.2 Hz, 2H), 1.34 – 1.24 (m, 16H), 0.87 (t, *J* = 6.7 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 146.79, 133.91, 129.39, 129.14, 127.04, 119.58, 50.63, 32.00, 30.46, 29.67, 29.63, 29.49, 29.41, 29.13, 26.62, 22.79, 14.23; IR (KBr): υ 2916, 2845, 1684, 1558, 1457, 1075, 827 cm^{-1}; HRMS (ESI) calcd for $[C_{19}H_{28}ClN_3Na]^+$ ($[M+Na]^+$): 356.1864, found: 356.1865.



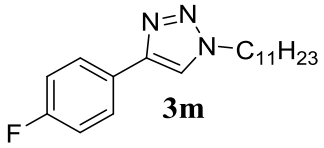
3k

Following the general procedure for Scheme 2, **3k** was obtained as a brown solid (121 mg, 76% yield); mp. 52 –53 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.32 (td, *J* = 7.6, 2.0 Hz, 1H), 7.93 (d, *J* = 3.8 Hz, 1H), 7.33 – 7.22 (m, 2H), 7.14 (ddd, J = 11.2, 8.0, 1.5 Hz, 1H), 4.42 (t, *J* = 7.2 Hz, 2H), 1.92 (t, *J* = 7.2 Hz, 2H), 1.35 – 1.26 (m, 16H), 0.88 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.33 (d, *J* = 247.4 Hz), 141.20, 129.31 (d, *J* = 8.7 Hz), 127.89 (d, *J* = 3.6 Hz), 124.73 (d, *J* = 3.0 Hz), 122.70 (d, *J* = 12.6 Hz), 118.81 (d, *J* = 13.1 Hz), 115.74 (d, *J* = 21.9 Hz) 50.57, 32.00, 30.46, 29.68, 29.63, 29.50, 29.42, 29.12, 26.60, 22.80, 14.24; ^{19}F NMR (376 MHz, $CDCl_3$) δ -114.97 (s, 1F); IR (KBr): υ 2954, 2918, 2851, 1557, 1469, 1227, 1074 cm^{-1}; HRMS (ESI) calcd for $[C_{19}H_{28}FN_3Na]^+$ ($[M+Na]^+$): 340.2159, found: 340.2160.



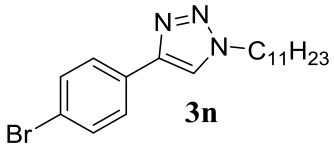
3l

Following the general procedure for Scheme 2, **3l** was obtained as a bright white solid (138 mg, 87% yield); mp. 73 – 74 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.76 (s, 1H), 7.62 – 7.52 (m, 2H), 7.37 (td, *J* = 8.0, 5.9 Hz, 1H), 7.01 (td, *J* = 8.5, 2.6 Hz, 1H), 4.39 (t, *J* = 7.2 Hz, 2H), 1.94 (t, *J* = 7.1 Hz, 2H), 1.34 – 1.25 (m, 16H), 0.87 (t, *J* = 6.7 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.29 (d, *J* = 245.7 Hz), 146.75 (d, *J* = 2.9 Hz), 133.03 (d, *J* = 8.6 Hz), 130.50 (d, *J* = 8.2 Hz), 121.37 (d, *J* = 2.9 Hz), 119.91, 114.95 (d, *J* = 21.2 Hz), 112.70 (d, *J* = 23.0 Hz), 50.63, 31.99, 30.43, 29.66, 29.61, 29.48, 29.40, 29.11, 26.59, 22.77, 14.21; ^{19}F NMR (376 MHz, $CDCl_3$) δ -112.74 (s, 1F); IR (KBr): υ 2951, 2916, 2847, 1588, 1465, 1217, 1156 cm^{-1}; HRMS (ESI) calcd for $[C_{19}H_{28}FN_3Na]^+$ ($[M+Na]^+$): 340.2159, found: 340.2159.



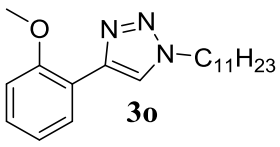
3m

Following the general procedure for Scheme 2, **3m** was obtained as a light yellow solid (141 mg, 89% yield); mp. 89.4 – 90.4 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.86 – 7.73 (m, 2H), 7.70 (s, 1H), 7.11 (t, *J* = 8.5 Hz, 2H), 4.38 (t, *J* = 7.3 Hz, 2H), 1.94 (t, *J* = 7.1 Hz, 2H), 1.33 –1.25 (m, 16H), 0.87 (t, *J* = 6.7 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.76 (d, *J* = 247.2 Hz), 147.01, 127.54 (d, *J* = 8.1 Hz), 127.10 (d, *J* = 3.2 Hz), 119.26, 115.93 (d, *J* = 21.9 Hz), 50.62, 32.01, 30.48, 29.68, 29.64, 29.51, 29.43, 29.14, 26.63, 22.80, 14.24; ^{19}F NMR (376 MHz, $CDCl_3$) δ -113.72 (s, 1F); IR (KBr): υ 2917, 2846, 1716, 1636, 1521, 1454, 1339 cm^{-1}; HRMS (ESI) calcd for $[C_{19}H_{28}FN_3Na]^+$ ($[M+Na]^+$): 340.2159, found: 340.2156.



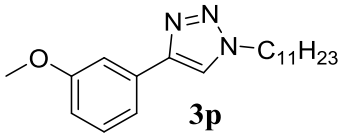
3n

Following the general procedure for Scheme 2, **3n** was obtained as a clear white solid (152 mg, 80% yield), mp. 87 – 88 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74 (s, 1H), 7.70 (d, *J* = 8.4 Hz, 2H), 7.54 (d, *J* = 8.3 Hz, 2H), 4.38 (t, *J* = 7.2 Hz, 2H), 1.90 (t, *J* = 7.2 Hz, 2H), 1.34 – 1.25 (m, 16H), 0.87 (t, *J* = 6.7 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 146.77, 132.06, 129.82, 127.31, 122.02, 119.61, 50.62, 31.98, 30.04, 29.65, 29.61, 29.48, 29.40, 29.10, 26.60, 22.77, 14.22; IR (KBr): υ 2917, 2847, 1684,1541, 1457, 1072, 825 cm^{-1}; HRMS (ESI) calcd for $[C_{19}H_{28}BrN_3Na]^+$ ($[M+Na]^+$): 400.1359, found: 400.1357.



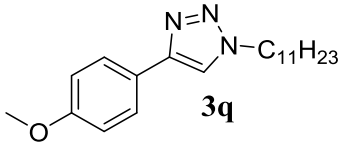
3o

Following the general procedure for Scheme 2, **3o** was obtained as a light yellow semi solid (130 mg, 79% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.34 (dd, *J* = 7.7, 1.8 Hz, 1H), 8.01 (s, 1H), 7.31 (ddd, *J* = 8.3, 7.3, 1.8 Hz, 1H), 7.08 (td, J = 7.5, 1.1 Hz, 1H), 6.98 (dd, *J* = 8.3, 1.0 Hz, 1H), 4.39 (t, *J* = 7.3 Hz, 2H), 3.94 (s, 3H), 1.95 (t, *J* = 7.3 Hz 2H), 1.34 – 1.23 (m, 16H), 0.87 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 155.72, 143.17, 128.92, 127.81, 123.02, 121.21, 119.71, 110.90, 55.51, 50.39, 32.02, 30.55, 29.69, 29.66, 29.55, 29.44, 29.18, 26.67, 22.81, 14.24; IR (KBr): υ 2925, 2853, 1550, 1490, 1247, 1181, 1026 cm^{-1}; HRMS (ESI) calcd for $[C_{20}H_{31}N_3NaO]^+$ ($[M+Na]^+$): 352.2359, found: 352.2359.



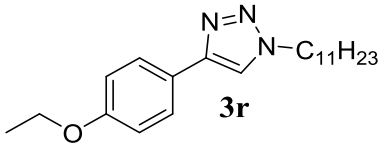
3p

Following the general procedure for Scheme 2, **3p** was obtained as a golden yellow solid (134 mg, 81% yield); mp. 44 – 45 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.73 (s, 1H), 7.44 (dd, *J* = 2.6, 1.5 Hz, 1H), 7.36 – 7.27 (m, 2H), 6.86 (ddd, *J* = 7.9, 2.7, 1.3 Hz, 1H), 4.35 (t, *J* = 7.2 Hz, 2H), 3.84 (s, 3H), 1.91 (t, *J* = 7.2 Hz, 2H), 1.31 – 1.24 (m, 16H), 0.86 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.10, 147.61, 132.13, 129.90, 119.72, 118.13, 114.21, 110.26, 55.39. 50.49, 31.95, 30.40, 29.61, 29.58, 29.45, 29.36, 29.07, 26.55, 22.73, 14.17; IR (KBr): υ 2954, 2919, 2848, 1647, 1456, 1232, 1042 cm^{-1}; HRMS (ESI) calcd for $[C_{20}H_{31}N_3NaO]^+$ ($[M+Na]^+$): 352.2359, found: 352.2360.



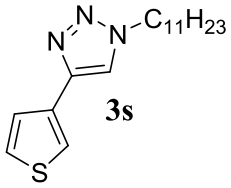
3q

Following the general procedure for Scheme 2, **3q** was obtained as a light yellow solid (126 mg, 77% yield); mp. 66.5 – 67.5 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.82 – 7.71 (m, 2H), 7.65 (s, 1H), 7.01 – 6.90 (m, 2H), 4.37 (t, *J* = 7.2 Hz, 2H), 3.84 (s, 3H), 1.93 (t, *J* = 7.2 Hz, 2H), 1.33 – 1.23 (m, 16H), 0.87 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.67, 147.71, 127.11, 132.61, 118.72, 114.35, 55.45, 50.53, 32.01, 30.50, 29.68, 29.64, 29.51, 29.42, 29.15, 26.64, 22.80, 14.24; IR (KBr): υ 2952, 2917, 2846, 1647, 1541, 1457, 1021 cm^{-1}; HRMS (ESI) calcd for $[C_{20}H_{31}N_3NaO]^+$ ($[M+Na]^+$): 352.2359, found: 352.2363.



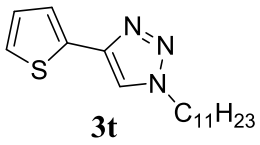
3r

Following the general procedure for Scheme 2, **3r** was obtained as a bright white solid (125 mg, 73% yield); mp. 102 – 103 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.75 – 7.68 (m, 2H), 7.64 (s, 1H), 6.95 – 6.88 (m, 2H), 4.33 (t, *J* = 7.2 Hz, 2H), 4.04 (q, *J*= 7.0 Hz, 2H), 1.90 (t, *J* = 7.2 Hz, 2H), 1.40 (t, *J* = 7.0 Hz, 3H), 1.33 – 1.20 (m, 16H), 0.86 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.96, 147.64, 127.00, 123.40, 118.69, 114.81, 63.53, 50.41, 31.94, 30.41, 29.61, 29.58, 29.45, 29.36, 29.08, 26.56, 22.72, 14.87, 14.16; IR (KBr): υ 2975, 2917, 2846, 1652, 1465, 1250, 1114 cm^{-1}; HRMS (EI) calcd for $[C_{23}H_{34}N_3O][M+H]^+$: 344.2696, found: 344.2693.



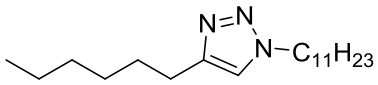
3s

Following the general procedure for Scheme 2, **3s** was obtained as a bright white solid (116 mg, 76% yield); mp. 94 – 95 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.66 (dd, *J* = 2.9, 1.3 Hz, 1H), 7.64 (s, 1H), 7.45 (dd, *J* = 5.0, 1.3 Hz, 1H), 7.36 (dd, *J* = 5.0, 3.0 Hz, 1H), 4.36 (t, *J* = 7.2 Hz, 2H), 1.91 (t, *J* = 7.2 Hz, 2H), 1.33 – 1.22 (m, 16H), 0.87 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 143.96, 132.04, 126.36, 125.89, 120.99, 119.35, 50.47, 31.97, 30.43, 29.64, 29.60, 29.47, 29.39, 29.10, 26.56, 22.76, 14.21; IR (KBr): υ 2951, 2916, 2848, 1558, 1465, 1217, 783 cm^{-1}; HRMS (ESI) calcd for $[C_{17}H_{27}N_3NaS]^+$ ($[M+Na]^+$): 328.1818, found: 328.1818.



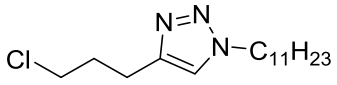
3t

Following the general procedure for Scheme 2, **3t** was obtained as an off-white solid (109 mg, 71% yield); mp. 67 – 68 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.65 (s, 1H), 7.36 (dd, *J* = 3.6, 1.1 Hz, 1H), 7.27 (dd, *J* = 5.0, 1.1 Hz, 1H), 7.06 (dd, *J* = 5.1, 3.6 Hz, 1H), 4.35 (t, *J* = 7.3 Hz, 2H), 1.92 (t, *J* = 7.2 Hz, 2H), 1.32 – 1.24 (m, 16H), 0.87 (t, *J* = 6.7 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 142.86, 133.22, 127.67, 124.98, 124.08, 119.04, 50.57, 31.97, 30.40, 29.64, 29.60, 29.47, 29.39, 29.09, 26.56, 22.76, 14.20; IR (KBr): υ 2952, 2917, 2847, 1558, 1465, 1219, 686 cm^{-1}; HRMS (ESI) calcd for $[C_{17}H_{27}N_3NaS\ ]^+$ ($[M+Na]^+$): 328.1818, found: 328.1818.



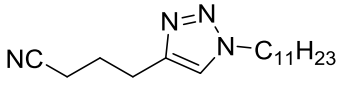
3u

Following the general procedure for Scheme 2, **3u** was obtained as a dull white solid (127.5 mg, 83% yield); mp. = 56–57 °C; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.22 (s, 1H), 4.31 – 4.20 (m, 2H), 2.65 (t, *J* = 7.5 Hz, 2H), 1.82 (t, *J* = 7.8 Hz, 2H), 1.61 (p, *J* = 8.0 Hz, 2H), 1.30 – 1.17 (m, 22H), 0.85 – 0.80 (m, 6H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 148.38, 120.40, 50.17, 31.91, 31.62, 30.39, 29.58, 29.54, 29.50, 29.42, 29.33, 29.04, 28.96, 26.53, 25.74, 22.70, 22.59, 14.12, 14.08; IR (KBr): υ 2953, 2916, 2848, 1636, 1468, 1212, 1154, 1061 cm^{-1}; HRMS (ESI) calcd for $[C_{19}H_{37}N_3Na]^+$ ($[M+Na]^+$): 330.2880, found: 330.2880.



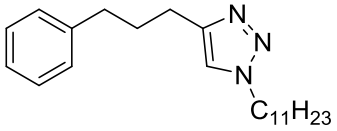
3v

Following the general procedure for Scheme 2, **3v** was obtained as an off-White solid (102 mg, 68% yield); mp. = 48–49 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.30 (s, 1H), 4.54 – 3.92 (m, 2H), 3.55 (t, *J* = 6.4 Hz, 2H), 3.04 – 2.70 (m, 2H), 2.30 – 2.01 (m, 2H), 1.91 – 1.75 (m, 2H), 1.46 – 1.15 (m, 16H), 0.85 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 146.30, 121.02, 50.31, 44.29, 31.96, 31.95, 30.40, 29.62, 29.57, 29.45, 29.37, 29.06, 26.57, 22.74, 22.72, 14.19; IR (KBr): υ 2952, 2916, 2847, 1646, 1489, 1305, 1212 842 cm^{-1};HRMS (ESI) calcd for $[C_{16}H_{30}ClN_3Na]^+$ ($[M+Na]^+$): 322.2020, found: 322.2020.



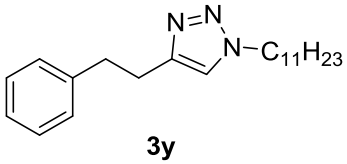
3w

Following the general procedure for Scheme 2, **3w** was obtained as an off-White solid (118 mg, 81% yield), mp. = 60–61 °C; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.31 (s, 1H), 4.27 (t, *J* = 7.3 Hz, 2H), 2.82 (t, *J* = 7.5 Hz, 2H), 2.38 (t, *J* = 7.1 Hz, 2H), 2.03 (t, *J* = 7.2 Hz, 2H), 1.84 (t, *J* = 7.9 Hz, 2H), 1.28 – 1.17 (m, 16H), 0.82 (t, *J* = 8.5 Hz 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 145.44, 121.14, 119.42, 50.29, 31.87, 30.31, 29.54, 29.50, 29.37, 29.29, 28.98, 26.49, 24.92, 24.20, 22.66, 16.47, 14.11; IR (KBr): υ 2953, 2919, 2847, 2246, 1636, 1463, 1372, 1210 cm^{-1}; HRMS (ESI) calcd for $[C_{17}H_{30}N_4Na]^+$ ($[M+Na]^+$): 313.2363, found: 313.2363.



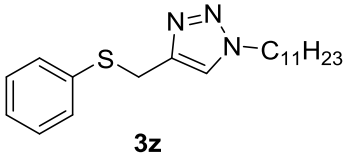
3x

Following the general procedure for Scheme 2, **3x** was obtained as a yellowish solid (133 mg, 78% yield); mp. = 40–41 °C; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.30 – 7.25 (m, 3H), 7.23 – 7.17 (m, 3H), 4.30 (td, *J* = 7.5, 1.4 Hz, 2H), 2.76 (t, *J* = 7.9 Hz, 2H), 2.69 (t, *J* = 8.1 Hz, 2H), 2.05 –2.00 (m, 2H), 1.88 (t, *J* = 7.2 Hz, 2H), 1.37 – 1.21 (m, 16H), 0.89 (t, *J* = 7.6 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 141.93, 128.48, 128.33, 125.82, 120.51, 50.17, 35.40, 31.89, 31.13, 30.35, 29.56, 29.52, 29.40, 29.31, 29.02, 26.51, 25.22, 22.68, 14.13; IR (KBr): υ 2953, 2917, 2847, 1646, 1461, 1211, 1146, 1056 cm^{-1}; HRMS (ESI) calcd for $[C_{22}H_{35}N_3Na]^+$ ($[M+Na]^+$): 364.2723, found: 364.2723.



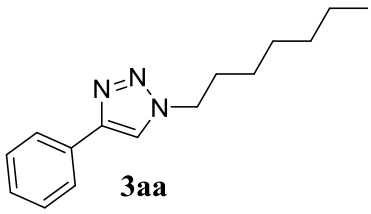
3y

Following the general procedure for Scheme 2, **3y** was obtained as an off-White solid (136 mg, 83% yield); mp. = 55–56 °C; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.24 (t, *J* = 7.4 Hz, 2H), 7.16 (dd, *J* = 13.0, 7.7 Hz, 3H), 7.06 (s, 1H), 4.23 (t, *J* = 7.2 Hz, 2H), 3.09 – 2.88 (m, 2H), 1.81– 1.79 (m, 2H), 1.31 – 1.08 (m, 16H), 0.86 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 147.14, 141.26, 128.53, 128.38, 126.09, 120.82, 50.17, 35.66, 31.92, 30.36, 29.60, 29.56, 29.42, 29.34, 29.04, 27.57, 26.47, 22.71, 14.16; IR (KBr): υ 2952, 2917, 2847, 1636, 1463, 1211, 1152, 1059 cm^{-1}; HRMS (ESI) calcd for $[C_{21}H_{33}N_3Na]^+$ ($[M+Na]^+$): 350.2567, found: 350.2567.



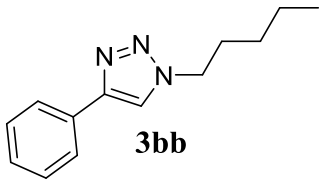
3z

Following the general procedure for Scheme 2, **3z** was obtained as an off-White solid (138 mg, 80% yield); mp. = 76–77 °C; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.31 (d, *J* = 8.3 Hz, 2H), 7.28 (s, 1H), 7.24 (t, *J* = 7.7 Hz, 2H), 7.16 (t, *J* = 8.2 Hz, 1H), 4.27 – 4.19 (m, 4H), 1.80 (t, *J* = 7.4 Hz, 2H), 1.29 – 1.19 (m, 16H), 0.86 (t, *J* = 7.74 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 144.86, 135.62, 129.55, 129.00, 126.47, 121.90, 50.39, 31.94, 30.28, 29.61, 29.56, 29.42, 29.35, 29.02, 28.93, 26.44, 22.73, 14.18; IR (KBr): υ 2952, 2917, 2847, 1635, 1464, 1437, 1240, 1060 cm^{-1}; HRMS (ESI) calcd for $[C_{20}H_{31}N_3SNa]^+$ ($[M+Na]^+$): 368.2131, found: 368.2131.



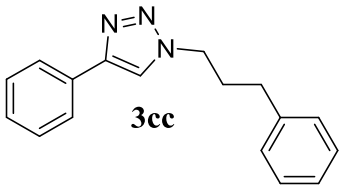
3aa

Following the general procedure for Scheme 3, **3aa** was obtained as a white solid (89 mg, 73% yield); mp. 63 –64 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.83 (dd, *J* = 7.2, 1.7 Hz, 2H), 7.74 (s, 1H), 7.42 (t, *J* = 7.6 Hz, 2H), 7.36 – 7.27 (m, 1H), 4.38 (t, *J* = 7.3 Hz, 2H), 1.94 (dd, *J* = 8.8, 5.7 Hz, 2H), 1.34 – 1.26 (m, 8H), 0.87 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.82, 130.86, 128.93, 128.18, 125.80, 119.51, 50.55, 31.68, 30.47, 28.79, 26.57, 22.63, 14.13; IR (KBr): υ 2952, 2925, 2852, 1616, 1464, 1216, 1078 cm^{-1}; HRMS (ESI) calcd for $[C_{15}H_{21}N_3Na]^+$ ($[M+Na]^+$): 266.1628, found: 266.1629.



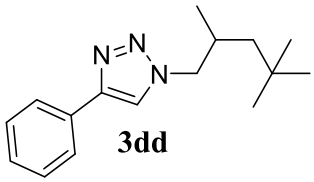
3bb

Following the general procedure for Scheme 3, **3bb** was obtained as a yellowish solid (78 mg, 72% yield); mp. 64 – 65 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87 – 7.80 (m, 2H), 7.74 (s, 1H), 7.46 – 7.39 (m, 2H), 7.35 – 7.29 (m, 1H), 4.39 (t, J = 7.3 Hz, 2H), 1.99 – 1.91 (m, 2H), 1.39 – 1.30 (m, 4H), 0.91 (t, J = 6.9 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.86, 130.86, 128.96, 128.21, 125.82, 119.51, 50.56, 30.18, 28.74, 22.24, 13.98; IR (KBr): υ 2951, 2922, 2855, 1646, 1464, 1216, 1077 cm^{-1}; HRMS (ESI) calcd for $[C_{13}H_{17}N_3Na]^+$ ($[M+Na]^+$): 238.1315, found: 238.1318.



3cc

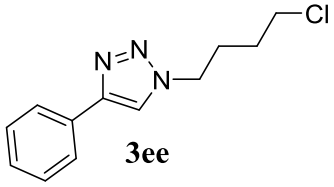
Following the general procedure for Scheme 3, **3cc** was obtained as a light yellow solid (118 mg, 90% yield); mp. 70 – 71 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87 – 7.80 (m, 2H), 7.72 (s, 1H), 7.48 – 7.39 (m, 2H), 7.39 – 7.27 (m, 3H), 7.25 – 7.15 (m, 3H), 4.40 (t, J = 7.1 Hz, 2H), 2.69 (t, J = 7.5 Hz, 2H), 2.36 – 2.24 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.87, 140.25, 130.77, 128.95, 128.74, 128.57, 128.24, 126.49, 125.80, 119.65, 49.68, 32.62, 31.78; IR (KBr): υ 3027, 2922, 2851, 1684, 1456, 1224, 1077 cm^{-1}; HRMS (ESI) calcd for $[C_{17}H_{17}N_3Na]^+$ ($[M+Na]^+$): 286.1315, found: 286.1307.



3dd

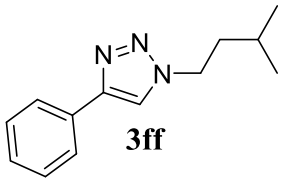
Following the general procedure for Scheme 3, **3dd** was obtained as a bright white solid (89 mg, 69% yield); mp. 99 – 100 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84 (dd, J = 8.3, 1.3 Hz, 2H), 7.73 (s, 1H), 7.45 – 7.39 (m, 2H), 7.35 – 7.30 (m, 1H), 4.27 (dd, J = 13.5, 6.6 Hz, 1H), 4.12 (dd, J = 13.5, 8.0 Hz, 1H), 2.21 – 2.11 (m, 1H), 1.32 (dd, J = 14.1, 3.5 Hz, 1H), 1.16 (dd, J = 14.1, 6.5 Hz, 1H), 0.97 (d, J = 6.7 Hz, 3H), 0.89 (s, 9H); ^{13}C

NMR (100 MHz, $CDCl_3$) δ 147.71, 130.86, 128.95, 128.20, 125.83, 120.02, 57.84, 47.87, 31.28, 31.08, 29.89, 20.45; IR (KBr): υ 3087, 2955, 1647, 1464, 1226, 1082 cm^{-1}; HRMS (ESI) calcd for $[C_{16}H_{23}N_3Na]^+$ ($[M+Na]^+$): 280.1784, found: 280.1784.



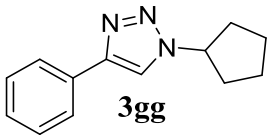
3ee

Following the general procedure for Scheme 3, **3ee** was obtained as a white solid (86 mg, 73% yield); mp. 51 – 52 °C; ^1H NMR (400 MHz, $CDCl_3$) δ 7.83 – 7.79 (m, 2H), 7.77 (s, 1H), 7.40 (td, *J* = 8.5, 7.8, 2.0 Hz, 2H), 7.34 – 7.29 (m, 1H), 4.41 (t, *J* = 7.0 Hz, 2H), 3.54 (t, *J* = 6.3 Hz, 2H), 2.10 (dt, *J* = 14.8, 7.0 Hz, 2H), 1.83 – 1.76 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.90, 130.58, 128.90, 128.22, 125.71, 119.61, 49.55, 44.08, 29.22, 27.59; IR (KBr): υ 2956, 2922, 2850, 1652, 1223, 1077, 746 cm^{-1}; HRMS (ESI) calcd for $[C_{12}H_{14}ClN_3Na]^+$ ($[M+Na]^+$): 258.0768, found: 258.0769.



3ff

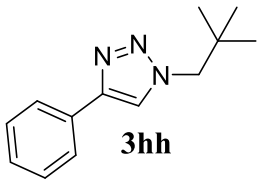
Following the general procedure for Scheme 3, **3ff** was obtained as an off-white solid (84 mg, 78% yield); mp. 77.7 – 78.7 °C; ^1H NMR (400 MHz, $CDCl_3$) δ 7.85 – 7.81 (m, 2H), 7.75 (s, 1H), 7.41 (t, *J* = 7.5 Hz, 2H), 7.34 – 7.30 (m, 1H), 4.43 – 4.38 (m, 2H), 1.84 (dt, *J* = 8.6, 7.1 Hz, 2H), 1.62 (dt, *J* = 13.4, 6.7 Hz, 1H), 0.98 (d, *J* = 6.7 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.83, 130.83, 128.91, 128.15, 125.77, 119.45, 48.84, 39.17, 25.61, 22.30; IR (KBr): υ 3082, 2956, 1733, 1558, 1447, 1222, 1078 cm^{-1}; HRMS (ESI) calcd for $[C_{13}H_{17}N_3Na]^+$ ($[M+Na]^+$): 238.1315, found: 238.1316.



3gg

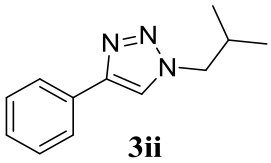
Following the general procedure for Scheme 3, **3gg** was obtained as a yellow solid (93 mg, 87% yield); mp. 68 –69 °C; ^1H NMR (400 MHz, $CDCl_3$) δ 7.83 – 7.80 (m, 2H), 7.75 (s, 1H), 7.42 – 7.38 (m, 2H), 7.33 – 7.28 (m, 1H), 4.95 (tt, *J* = 7.6, 6.2 Hz, 1H), 2.31 – 2.23 (m, 2H), 2.12 – 2.04 (m, 2H), 1.94– 1.88 (m, 2H), 1.82 – 1.73

(m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.53, 130.87, 128.80, 128.00, 125.66, 118.09, 61.91, 33.44, 24.11; IR (KBr): υ 3096, 2955, 2872, 1652, 1455, 1224, 1080 cm^{-1}; HRMS (ESI) calcd for $[C_{13}H_{15}N_3Na]^+$ ($[M+Na]^+$): 236.1158, found: 236.1158.



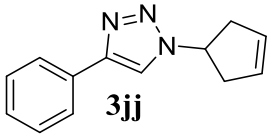
3hh

Following the general procedure for Scheme 3, **3hh** was obtained as a white solid (83 mg, 77% yield); mp. 108 –109 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88 – 7.81 (m, 2H), 7.71 (s, 1H), 7.46 – 7.39 (m, 2H), 7.33 (d, J = 7.4 Hz, 1H), 4.18 (s, 2H), 1.02 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.31, 130.81, 128.93, 128.16, 125.80, 120.87, 61.94, 32.76, 27.62; IR (KBr): υ 2933, 2856, 1655, 1541, 1463, 1226, 1079 cm^{-1}; HRMS (ESI) calcd for $[C_{13}H_{17}N_3Na]^+$ ($[M+Na]^+$): 238.1315, found: 238.1316.



3ii

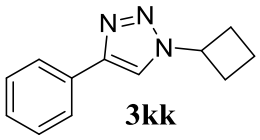
Following the general procedure for Scheme 3, **3ii** was obtained as a light yellow solid (93 mg, 92% yield); mp. 62 – 63 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88 – 7.81 (m, 2H), 7.73 (s, 1H), 7.45 – 7.39 (m, 2H), 7.35 – 7.29 (m, 1H), 4.20 (d, J = 7.2 Hz, 2H), 2.26 (dt, J = 13.7, 6.9 Hz, 1H), 0.97 (d, J = 6.7 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.68, 130.81, 128.92, 128.17, 125.77, 120.00, 57,73, 29.84, 19.95; IR (KBr): υ 2960, 2928, 2872, 1652, 1465, 1224, 1070 cm^{-1}; HRMS (ESI) calcd for $[C_{12}H_{15}N_3Na]^+$ ($[M+Na]^+$): 224.1158, found: 224.1160.



3jj

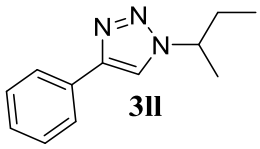
Following the general procedure for Scheme 3, **3jj** was obtained as a brown semi solid (26 mg, 60% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84 – 7.80 (m, 2H), 7.74 (s, 1H), 7.43 – 7.38 (m, 2H), 7.34 – 7.29 (m, 1H), 5.88 (s, 2H), 5.42 (tt, J = 8.3, 3.6 Hz, 1H), 3.12 – 3.04 (m, 2H), 2.74 (dd, J = 15.7, 3.4 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 148.18, 130.86, 128.91, 128.87, 128.18, 125.78, 117.17, 59.32, 40.77; IR (KBr): υ 3061, 2929,

2854, 1653, 1451, 1226, 1077 cm^{-1}; HRMS (ESI) calcd for $[C_{13}H_{13}N_3Na]^+$ ($[M+Na]^+$): 234.1002, found: 234.1001.



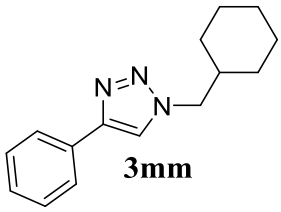
3kk

Following the general procedure for Scheme 3, **3kk** was obtained as a light white solid (95 mg, 95% yield); mp. 74 – 75 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84 – 7.79 (m, 3H), 7.39 (dd, *J* = 8.5, 6.8 Hz, 2H), 7.32 – 7.27 (m, 1H), 5.09 – 4.98 (m, 1H), 2.63 – 2.53 (m, 4H), 1.98 – 1.85 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.53, 130.80, 128.80, 128.02, 125.68, 118.12, 54.11, 30.76, 15.02; IR (KBr): υ 2996, 2871, 1647, 1569, 1456, 1220, 1072 cm^{-1}; HRMS (ESI) calcd for $[C_{12}H_{13}N_3Na]^+$ ($[M+Na]^+$): 222.1002, found: 222.1001.



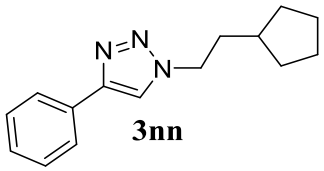
3ll

Following the general procedure for Scheme 3, **3ll** was obtained as a bright white solid (89 mg, 94% yield); mp. 54 – 55 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85 – 7.81 (m, 2H), 7.75 (s, 1H), 7.43 – 7.37 (m, 2H), 7.33 – 7.28 (m, 1H), 4.66 – 4.56 (m, 1H), 2.00 – 1.85 (m, 2H), 1.58 (d, *J* = 6.8 Hz, 3H), 0.87 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.54, 130.93, 128.86, 128.06, 125.70, 117.53, 59.01, 30.38, 21.04, 10.51; IR (KBr): υ 2930, 2854, 1653, 1533, 1452, 1226, 1077 cm^{-1}; HRMS (ESI) calcd for $[C_{12}H_{15}N_3Na]^+$ ($[M+Na]^+$): 224.1158, found: 224.1159.



3mm

Following the general procedure for Scheme 3, **3mm** was obtained as a pale yellow solid (115 mg, 95% yield); mp. 95 – 96 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.86 – 7.79 (m, 2H), 7.71 (s, 1H), 7.44 – 7.36 (m, 2H), 7.34 – 7.28 (m, 1H), 4.20 (d, *J* = 7.2 Hz, 2H), 1.91 – 1.88 (m, 1H), 1.74 – 1.63 (m, 4H), 1.33 – 1.08 (m, 4H), 1.06 – 0.95 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.60, 130.81, 128.89, 128.12, 125.74, 120.14, 56.62, 38.88, 30.57, 26.14, 25.59; IR (KBr): υ 2922, 2849, 1507, 1457, 1221, 1085 cm^{-1}; HRMS (ESI) calcd for $[C_{15}H_{19}N_3Na]^+$ ($[M+Na]^+$): 264.1471, found: 264.1471.

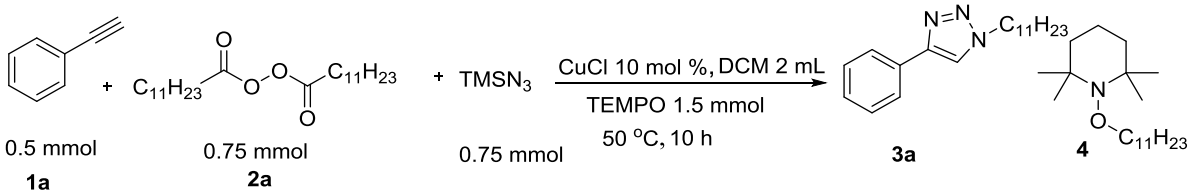


3nn

Following the general procedure for Scheme 3, **3nn** was obtained as a dull white solid (78 mg, 64% yield); mp. 98 – 99 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88 – 7.79 (m, 2H), 7.74 (s, 1H), 7.41 (dd, *J* = 8.3, 6.8 Hz, 2H), 7.35 – 7.29 (m, 1H), 4.39 (t, *J* = 7.3, Hz, 2H), 1.96 (dt, *J* = 8.6, 7.0 Hz, 2H), 1.87 – 1.47 (m, 8H), 1.15 (ddt, *J* = 8.4, 4.9, 2.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.81, 130.84, 128.92, 128.17, 125.79, 119.48, 49.97, 37.30, 36.69, 32.48, 25.16; IR (KBr): υ 2949, 2871, 1647, 1457, 1220, 1086 cm^{-1}; HRMS (ESI) calcd for $[C_{15}H_{19}N_3Na]^+$ ($[M+Na]^+$): 264.1471, found: 264.1472.

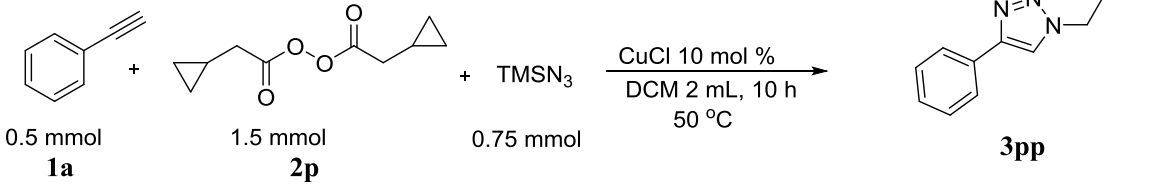
Preliminary mechanistic studies

(a) With TEMPO

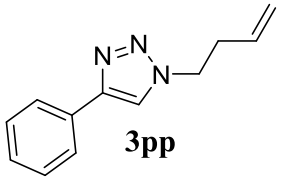


Phenyl acetylene (**1a**, 52.6 mg, 0.5 mmol), LPO (398.96 mg, 0.75 mmol), CuCl (4.9 mg, 0.05 mmol), TEMPO (1.5 mmol), $TMSN_3$ (90.4 mg, 0.75 mmol), and CH_2Cl_2 (2 mL) were added into a flame-dried Schlenk tube with a stirring bar and stirred at 50 °C for 10 hours. Then, the reaction mixture was cooled to ambient temperature, poured into saturated sodium bicarbonate solution (25 mL) and extracted with CH_2Cl_2 (3 × 25 mL) and passed through a short pad of celite. The filtrate was analyzed by GC–MS. No product (**3a**) was observed, only compound **4** was detected by GC–MS.

(b) Ring opening experiment



CuCl (4.9 mg, 10 mol %), $TMSN_3$ (90.4 mg, 0.75 mmol), phenylacetylene (**1a,** 52.6 mg, 0.5 mmol), diacyl peroxide **2p** (1.5 mmol) and CH_2Cl_2 (2 mL) were added to a flame-dried Schlenk tube with a stirring bar. The reaction mixture was heated to 50 °C for 10 hours, then cooled to room temperature. The solvent was removed by rotary evaporation, and the residue was chromatographed on silica gel which afforded the ring-opening product **3pp** in 88% yield.

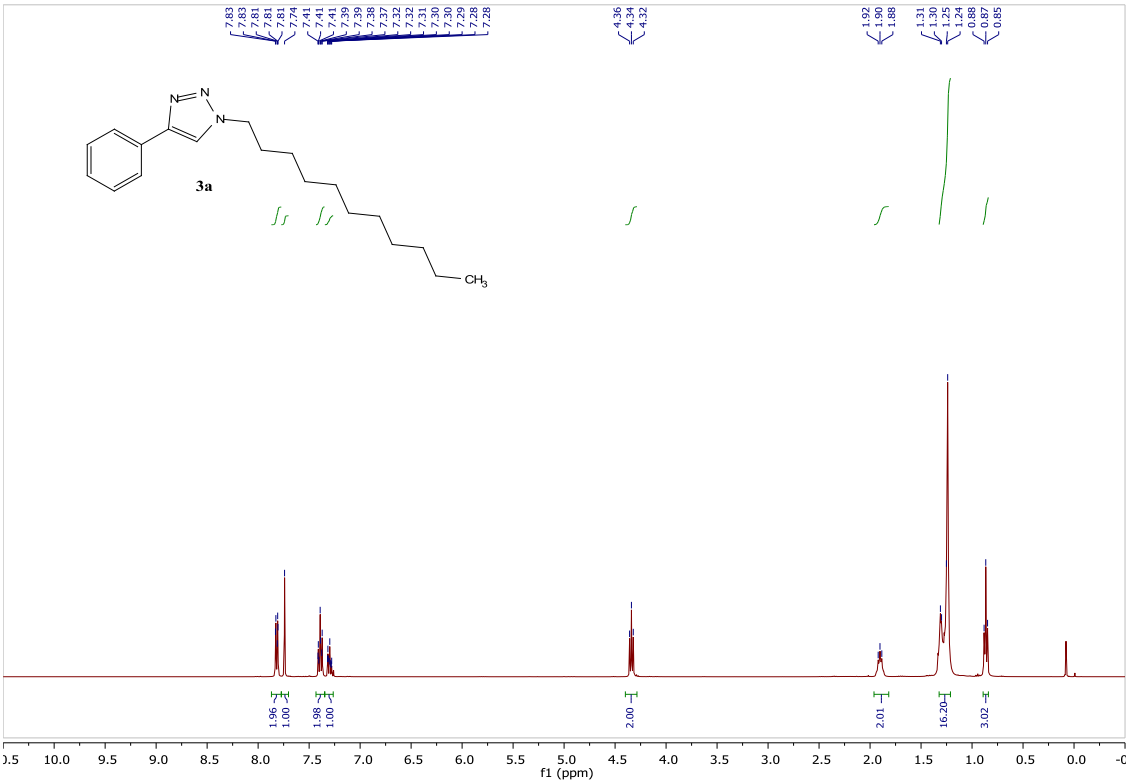


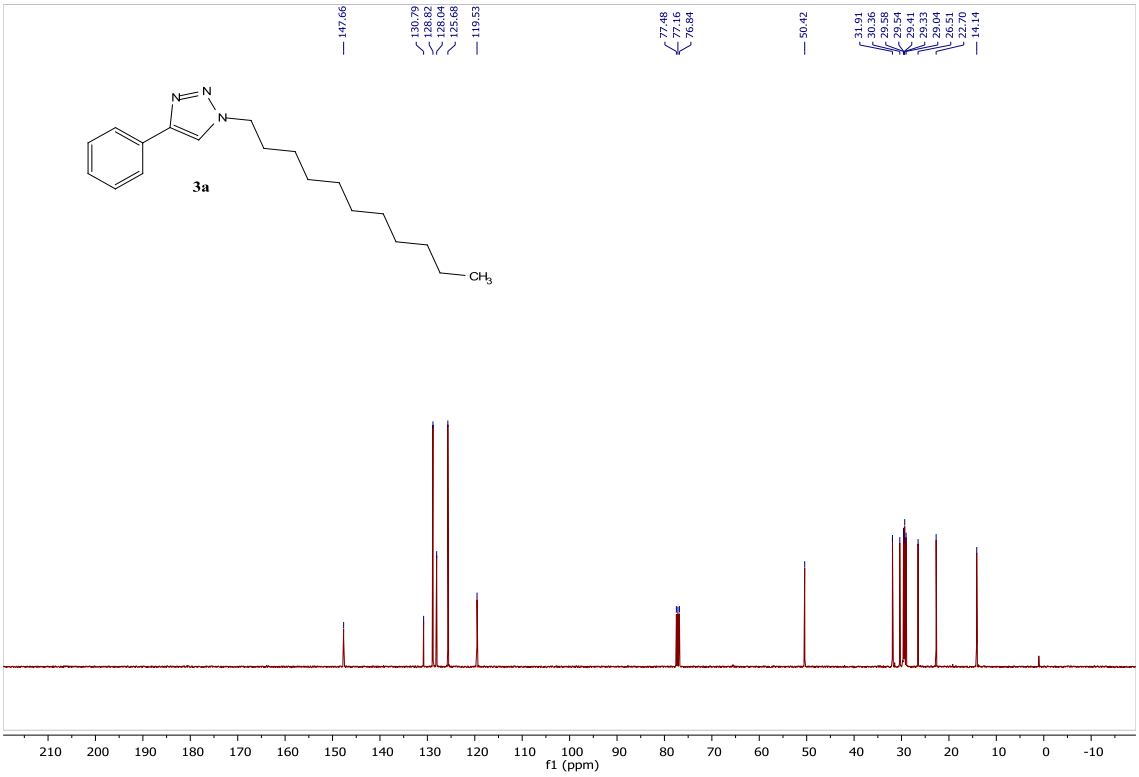
Compound **3pp** was obtained as a light brown solid (94 mg, 88% yield); mp. 53 – 54 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84 – 7.79 (m, 2H), 7.75 (s, 1H), 7.43 – 7.37 (m, 2H), 7.34 – 7.29 (m, 1H), 5.87 – 5.68 (m, 1H), 5.12 (dt, *J* = 2.3, 1.4 Hz, 1H), 5.10 – 5.03 (m, 1H), 4.44 (t, *J* = 7.1 Hz, 2H), 2.71 – 2.65 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.71, 133.23, 130.75, 128.90, 128.16, 125.75, 119.74, 118.58, 49.80, 34.51; IR (KBr): υ 3096, 2927, 2854, 1646, 1450, 1225, 1080 cm^{-1}; HRMS (ESI) calcd for $[C_{12}H_{13}N_3Na]^+$ ($[M+Na]^+$): 222.1002, found: 222.1000.

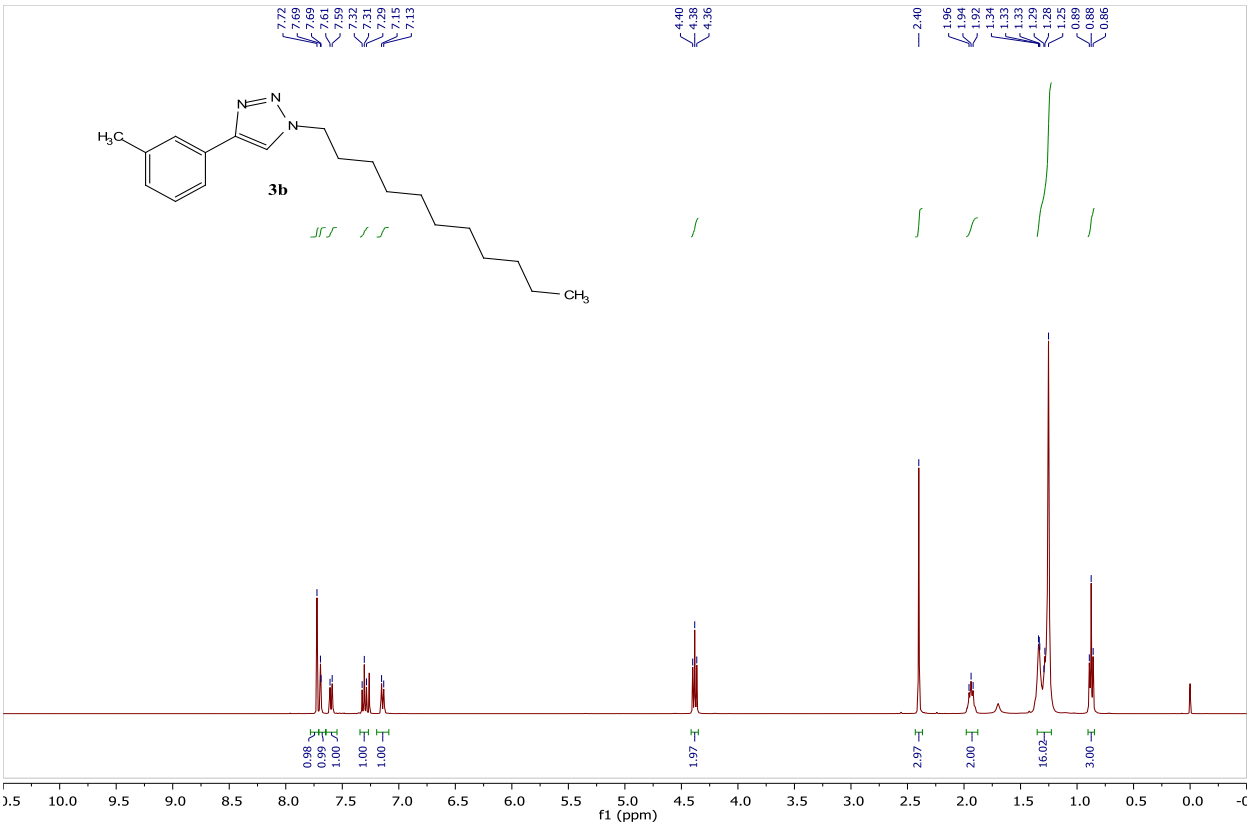
References

1. Li, Y.; Han, Y.; Xiong, H.; Zhu, N.; Qian, B.; Ye, C.; Kantchev, E. A. B.; Bao, H. *Org. Lett*., **2016**, *18*, 392.
2. Sheldon, R. A.; Kochi, J. K. *J. Am. Chem. Soc*., **1970**, *92*, 4395.

Copies of ^{1}H, ^{13}C, and ^{19}F NMR spectra



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7.29
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7.13
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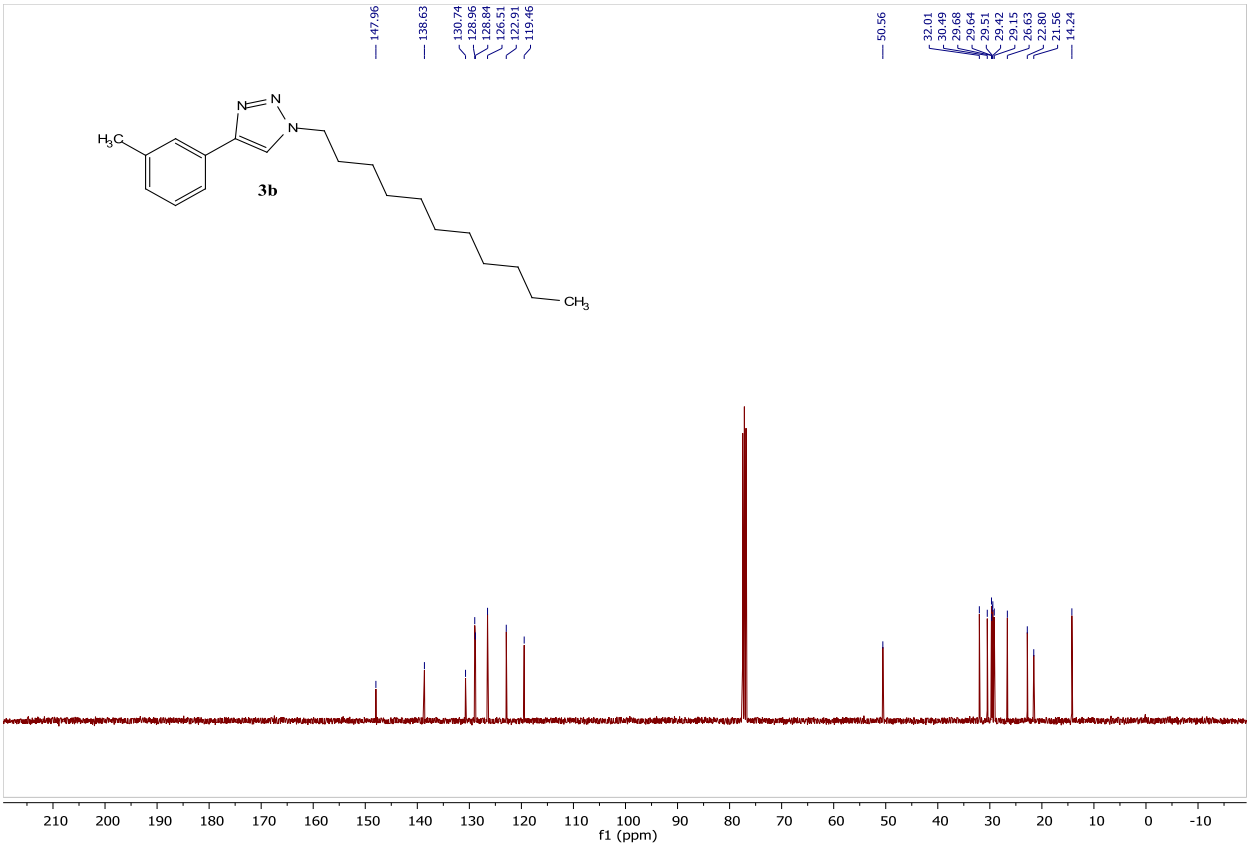
4.36
2.40
1.96
1.94
1.92
1.34
1.33
1.29
1.28
1.25
0.89
0.88
0.86
3b
0.98
0.99
1.00
1.00
1.00
1.97
2.97
2.00
16.02
3.00
f1 (ppm)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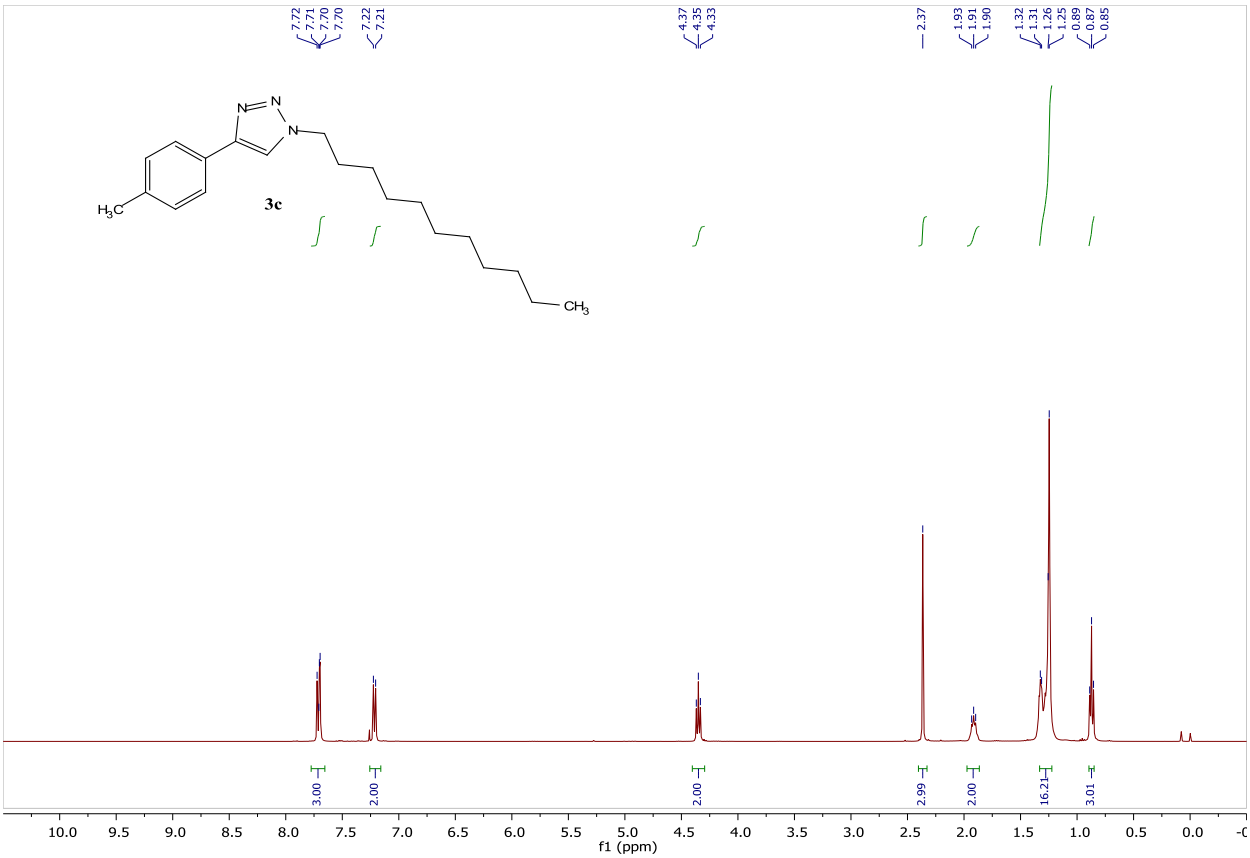
14.24
3b
f1 (ppm)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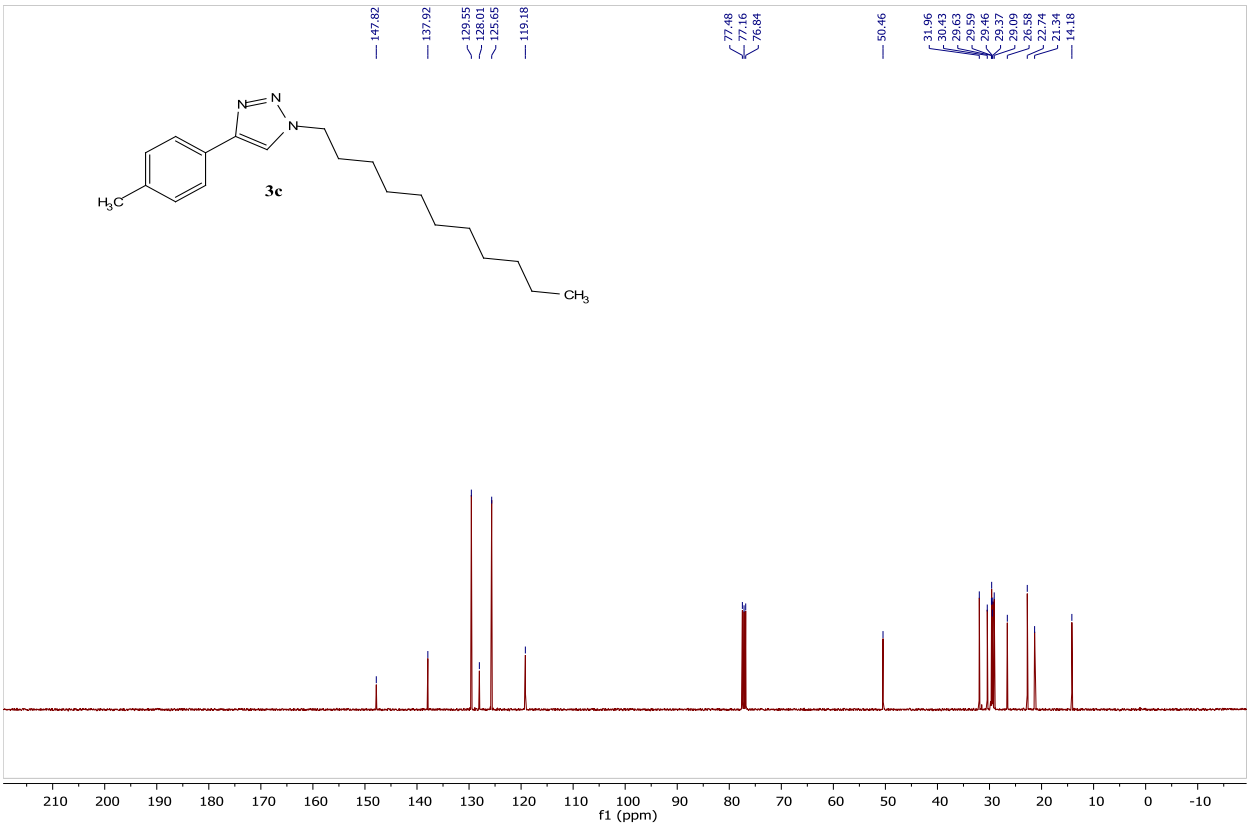
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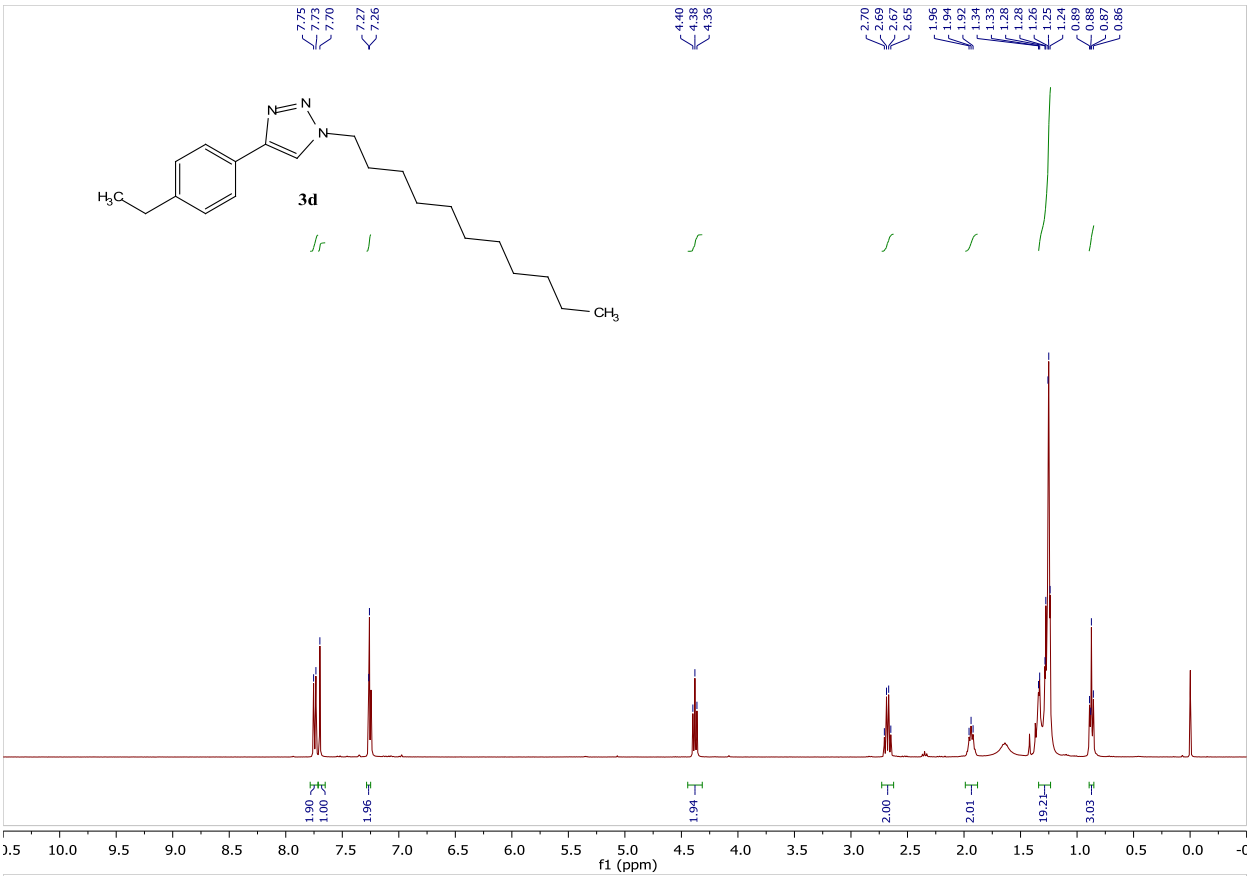
f1 (ppm)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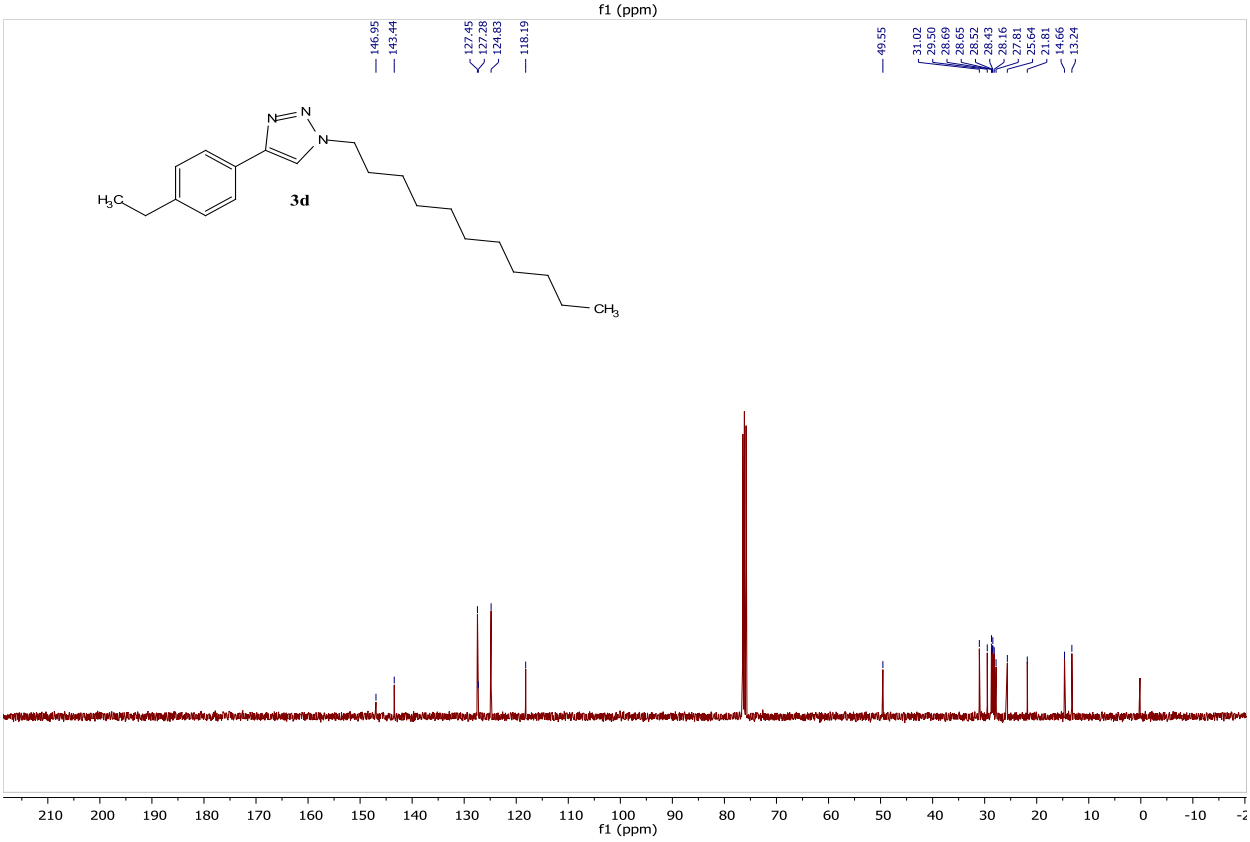
3.03
f1 (ppm)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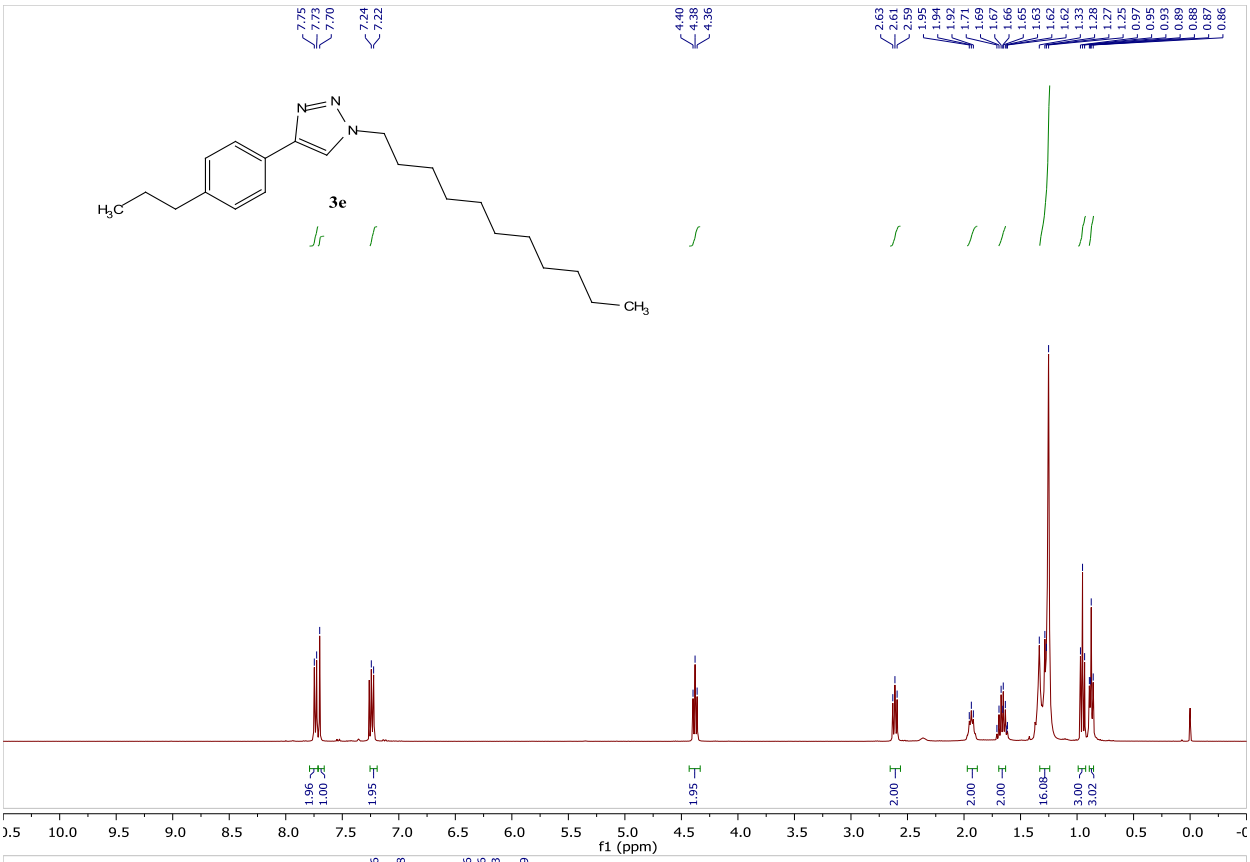
3d
f1 (ppm)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1.69
1.67
1.66
1.65
1.63
1.62
1.62
1.33
1.28
1.27
1.25
0.97
0.95
0.93
0.89
0.88
0.87
0.86
3e
CH3
1.96
1.00
1.95
1.95
2.00
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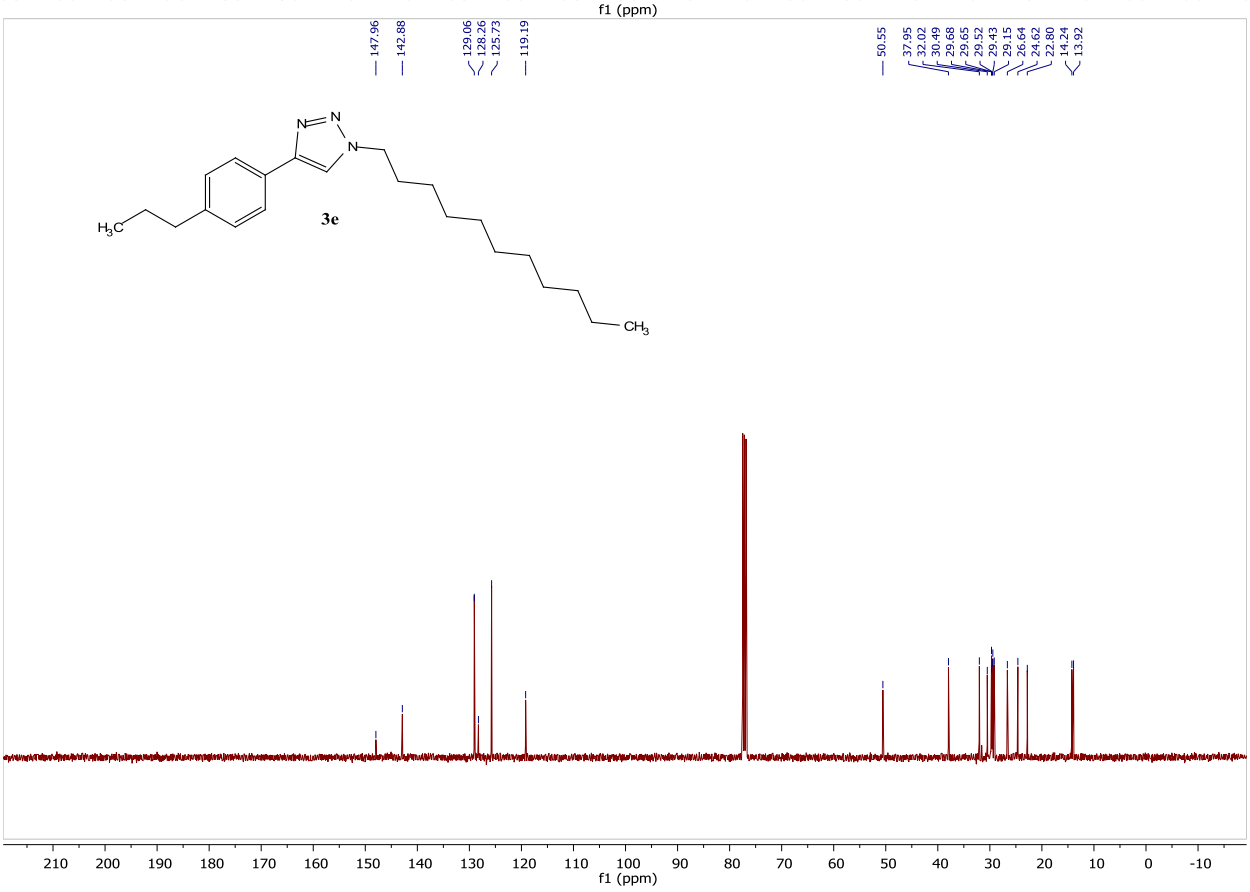
2.00
16.08
3.00
3.02
f1 (ppm)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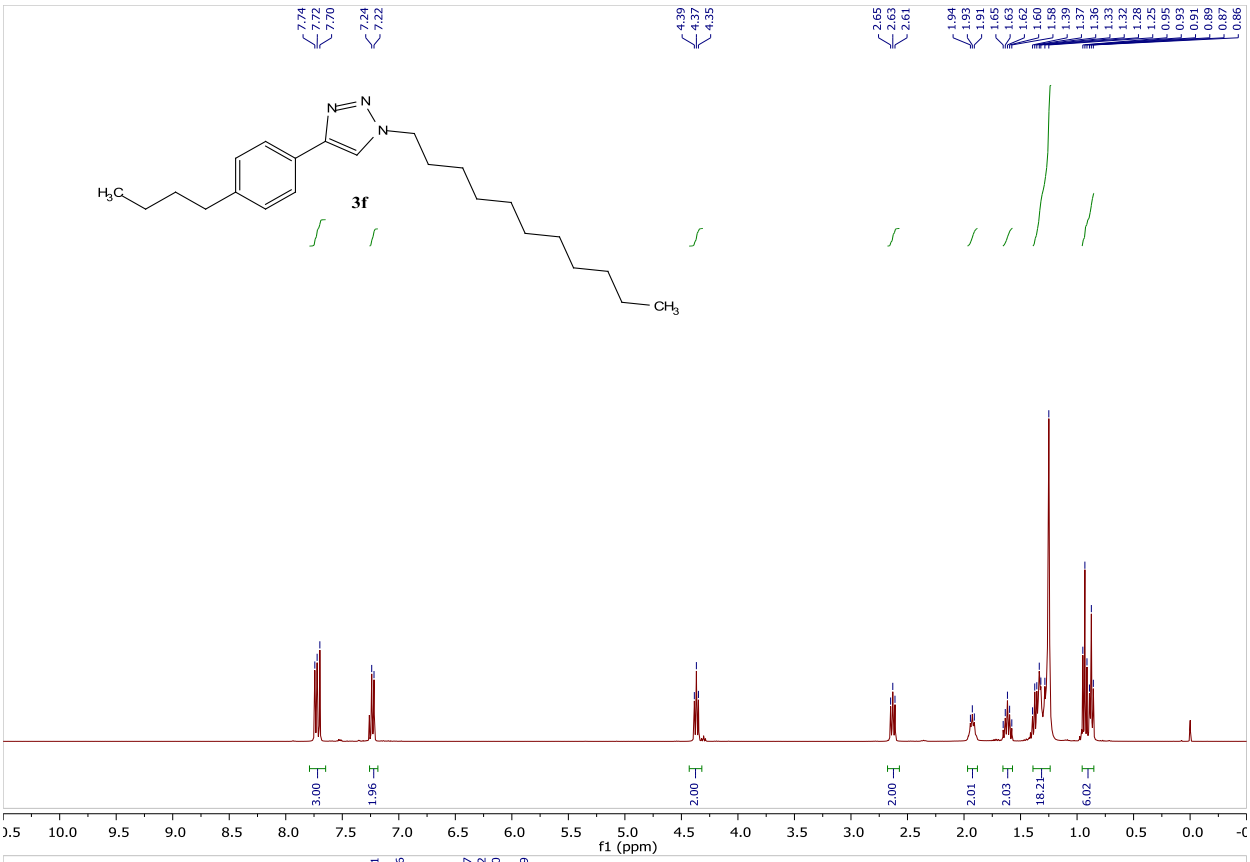
14.24
13.92
3e
CH3
f1 (ppm)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1.91
1.65
1.63
1.62
1.60
1.58
1.39
1.37
1.36
1.33
1.32
1.28
1.25
0.95
0.93
0.91
0.89
0.87
0.86
N
N
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CH3
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2.00
2.00
2.01
2.03
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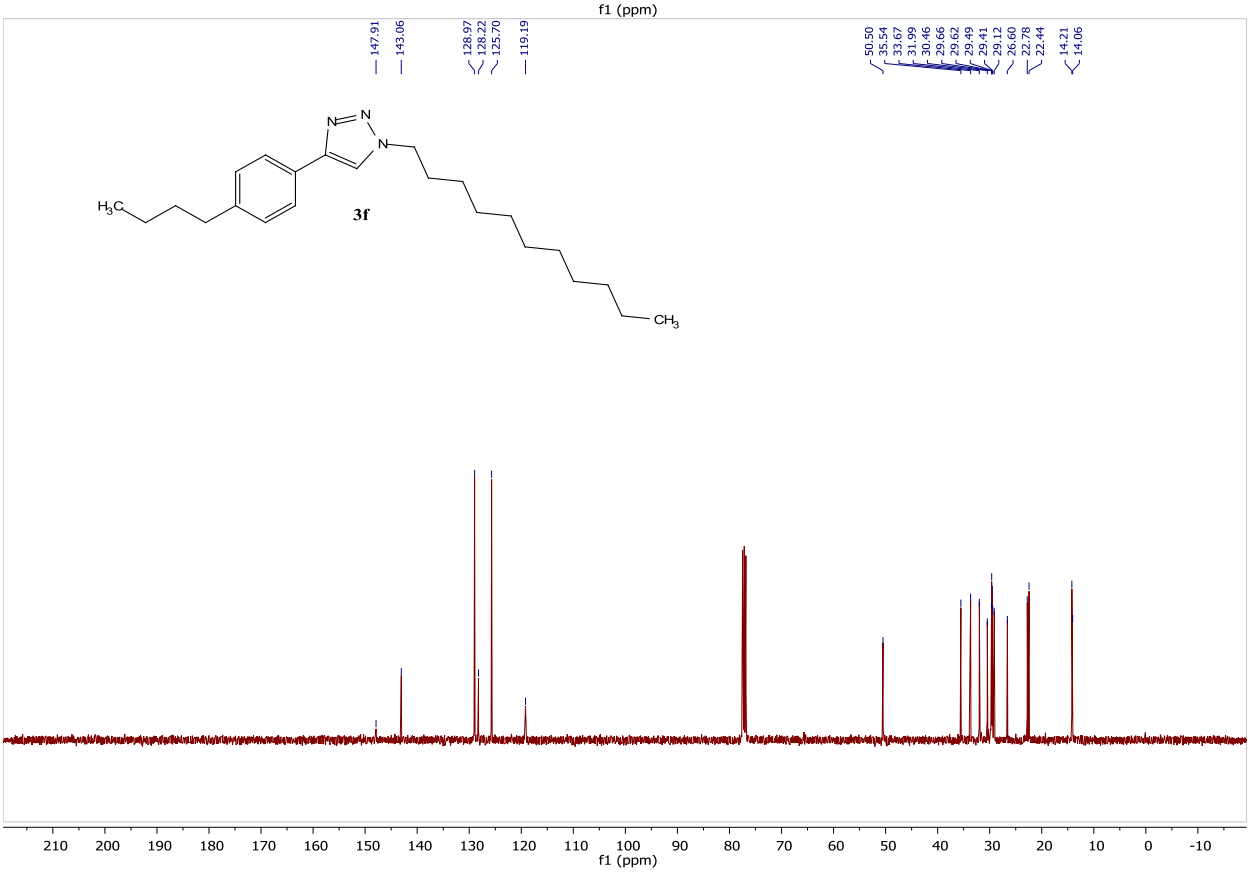
6.02
f1 (ppm)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29.41
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N
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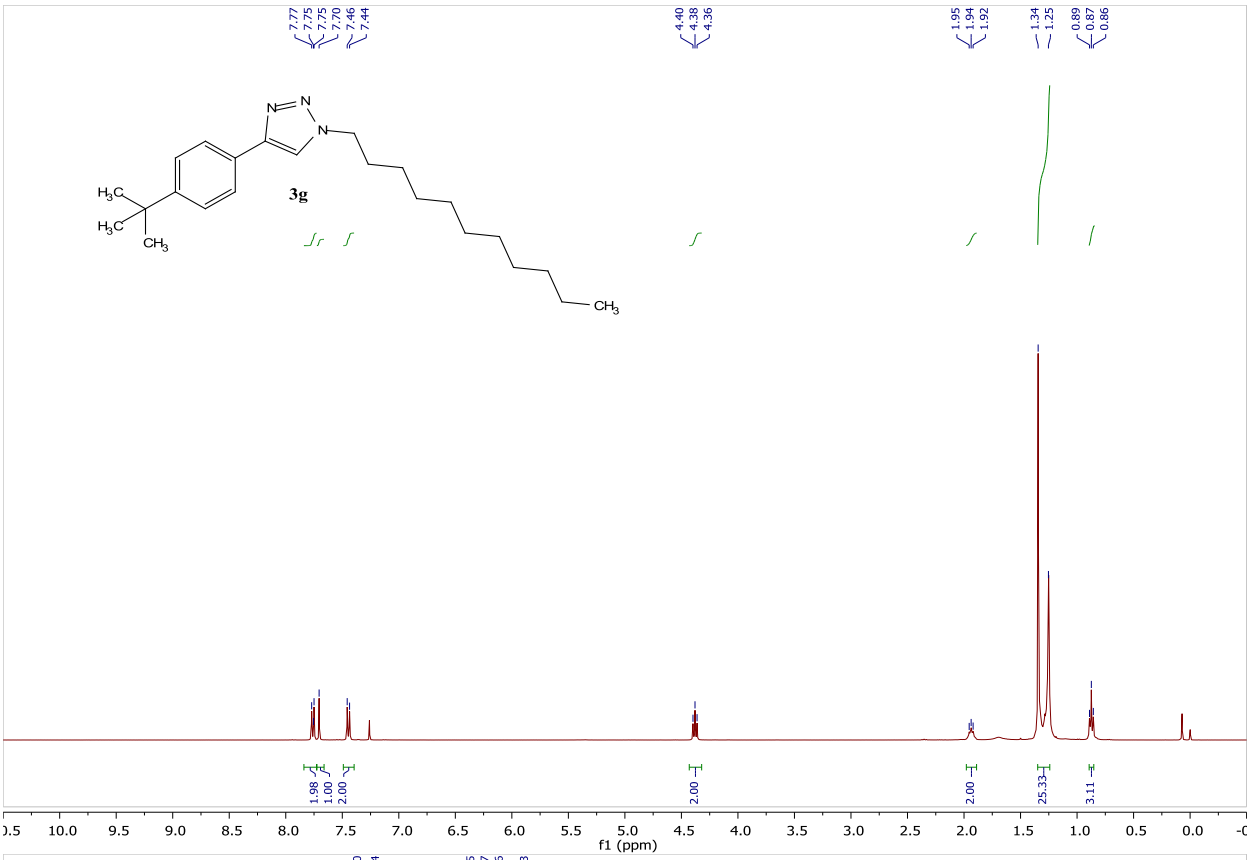
3f
CH3
f1 (ppm)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1.94
1.92
1.34
1.25
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2.00
2.00
2.00
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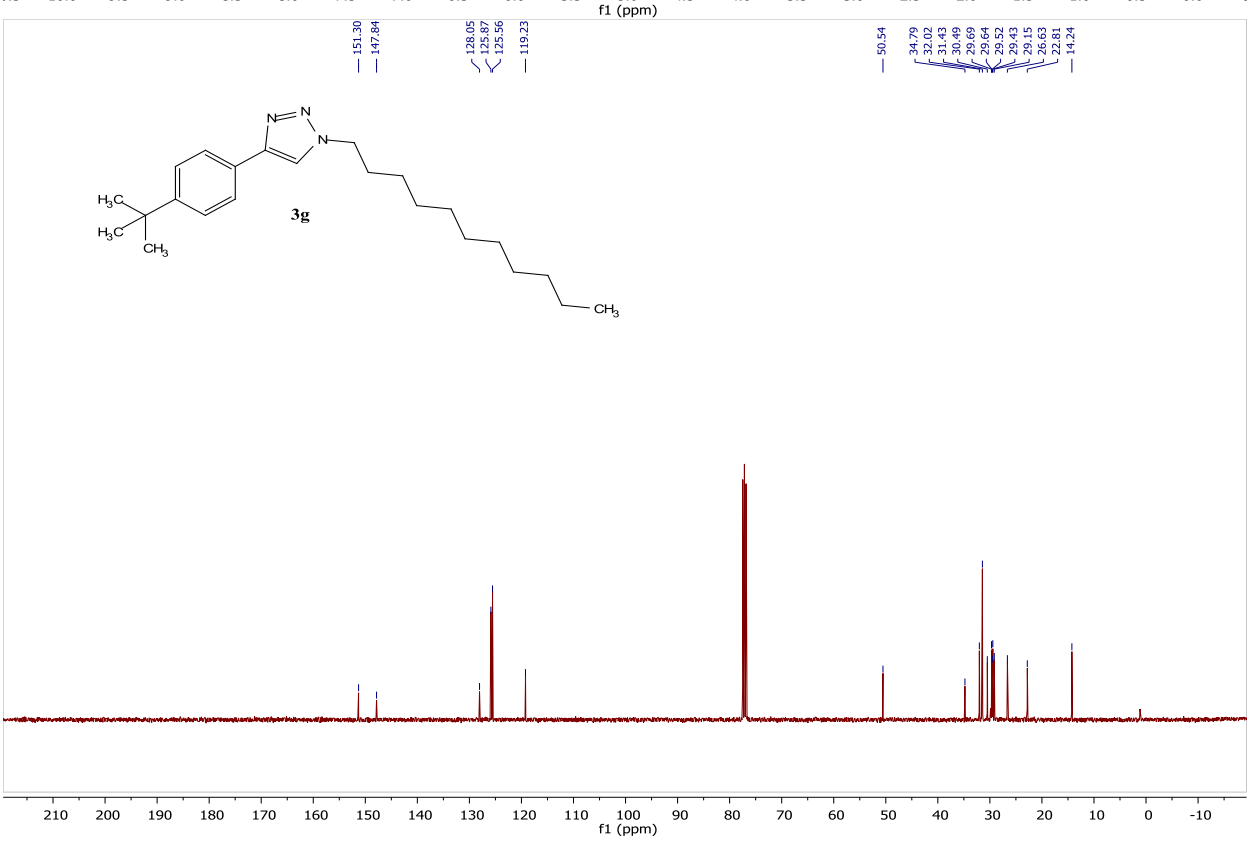
f1 (ppm)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26.63
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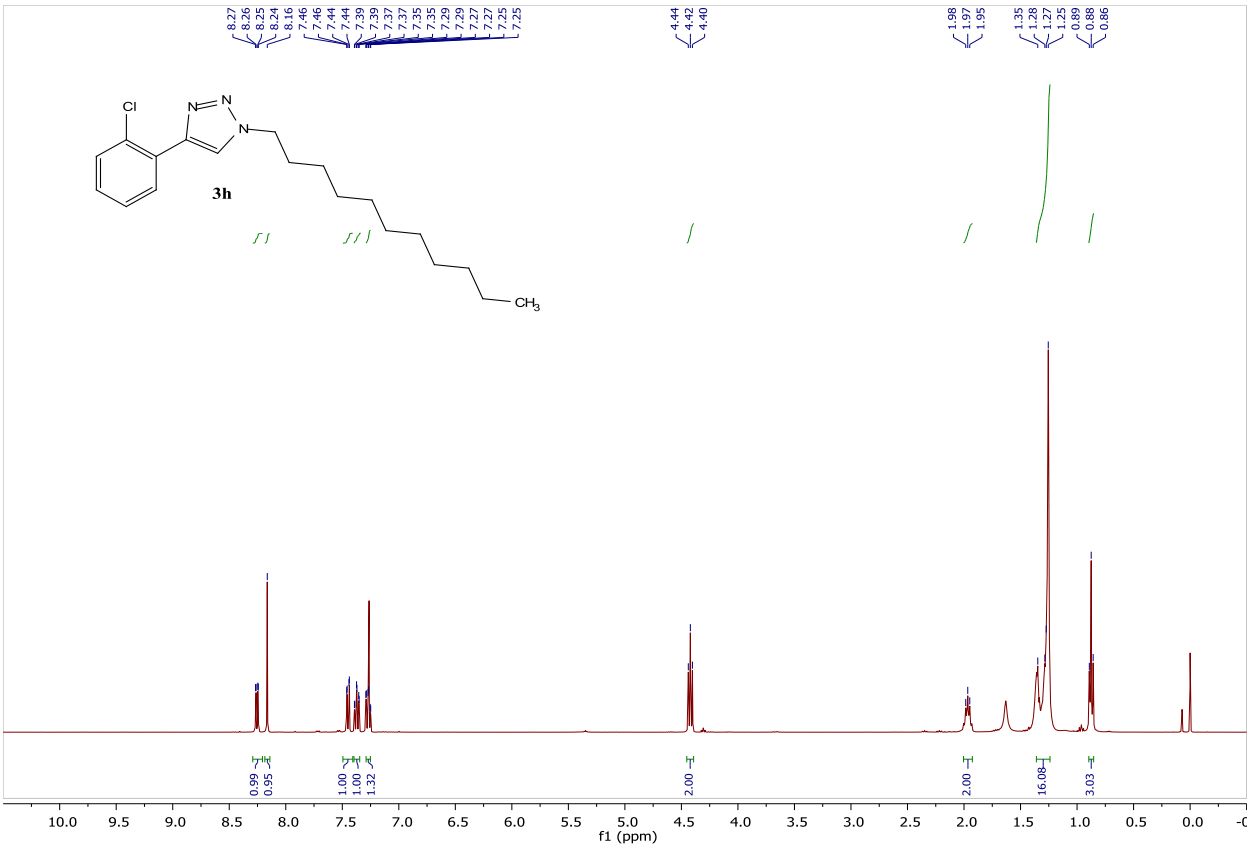
3g
f1 (ppm)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7.29
7.27
7.27
7.25
7.25
4.44
4.42
4.40
1.98
1.97
1.95
1.35
1.28
1.27
1.25
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0.88
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N
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1.00
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2.00
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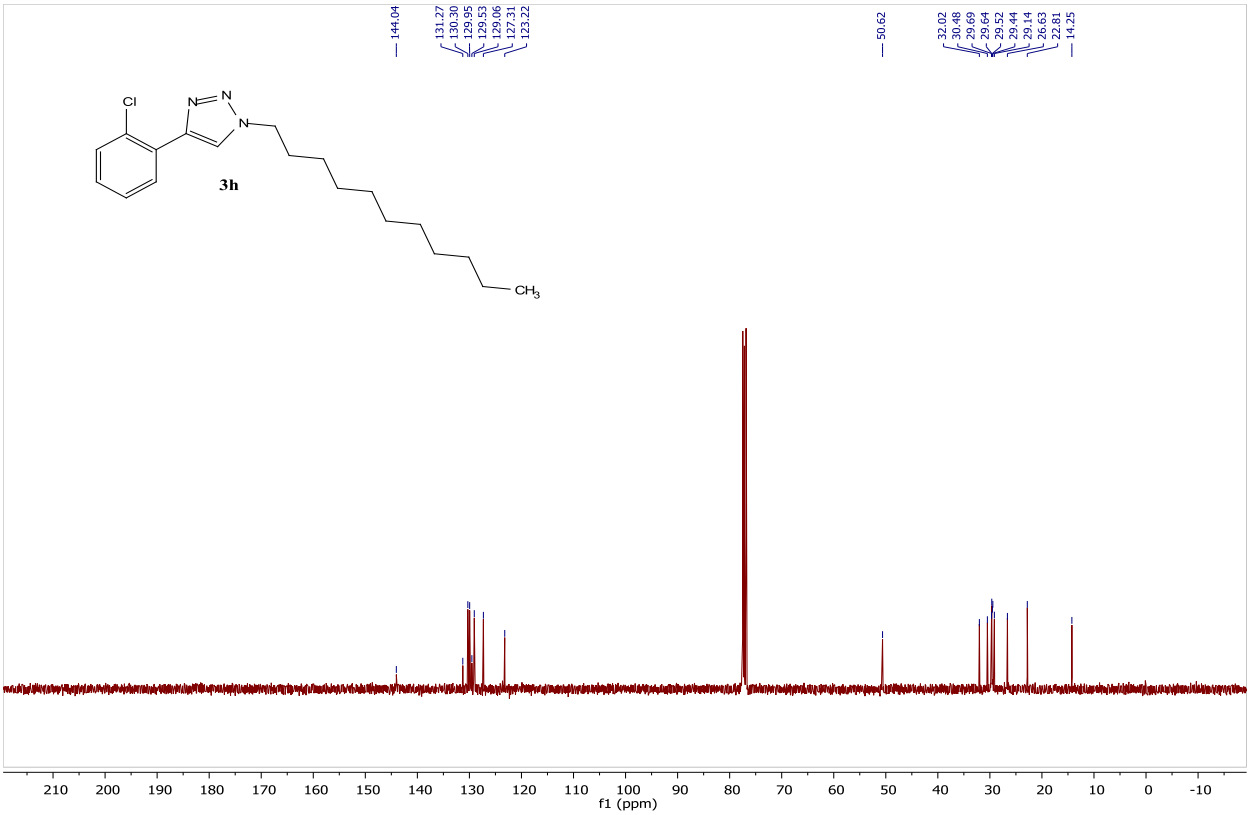
3.03
f1 (ppm)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29.14
26.63
22.81
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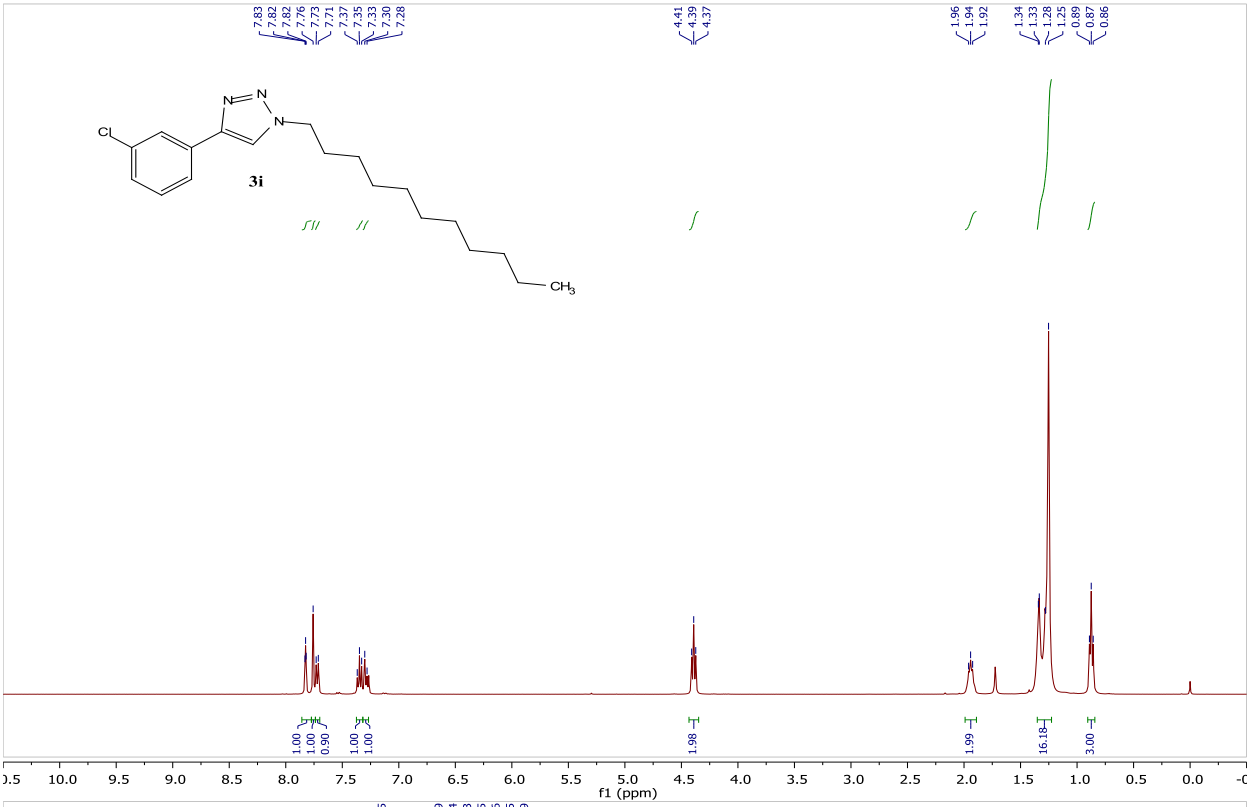
N
N
3h
CH3
f1 (ppm)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7.33
7.30
7.28
4.41
4.39
4.37
1.96
1.94
1.92
1.34
1.33
1.28
1.25
0.89
0.87
0.86
Cl
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N
N
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CH3
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1.00
1.00
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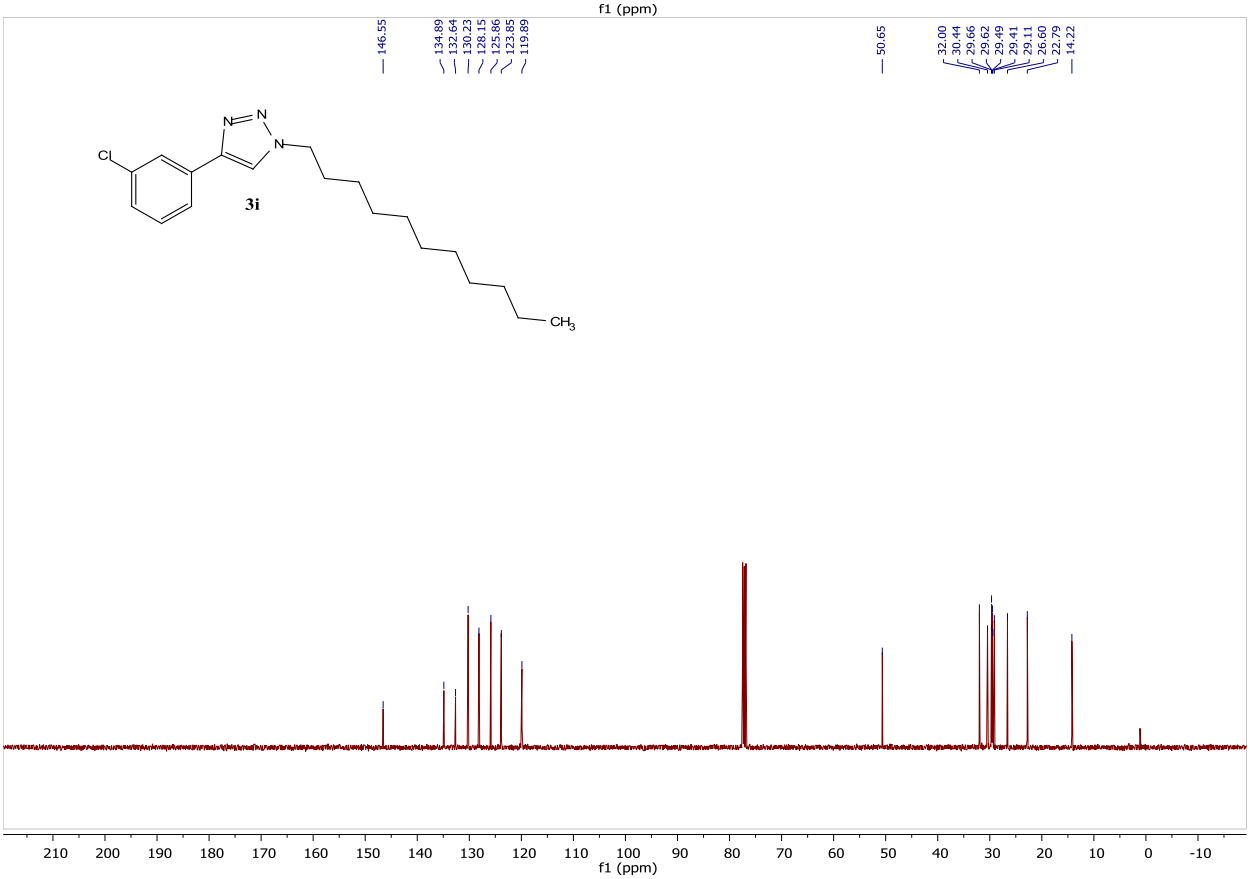
1.99
16.18
3.00
f1 (ppm)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29.66
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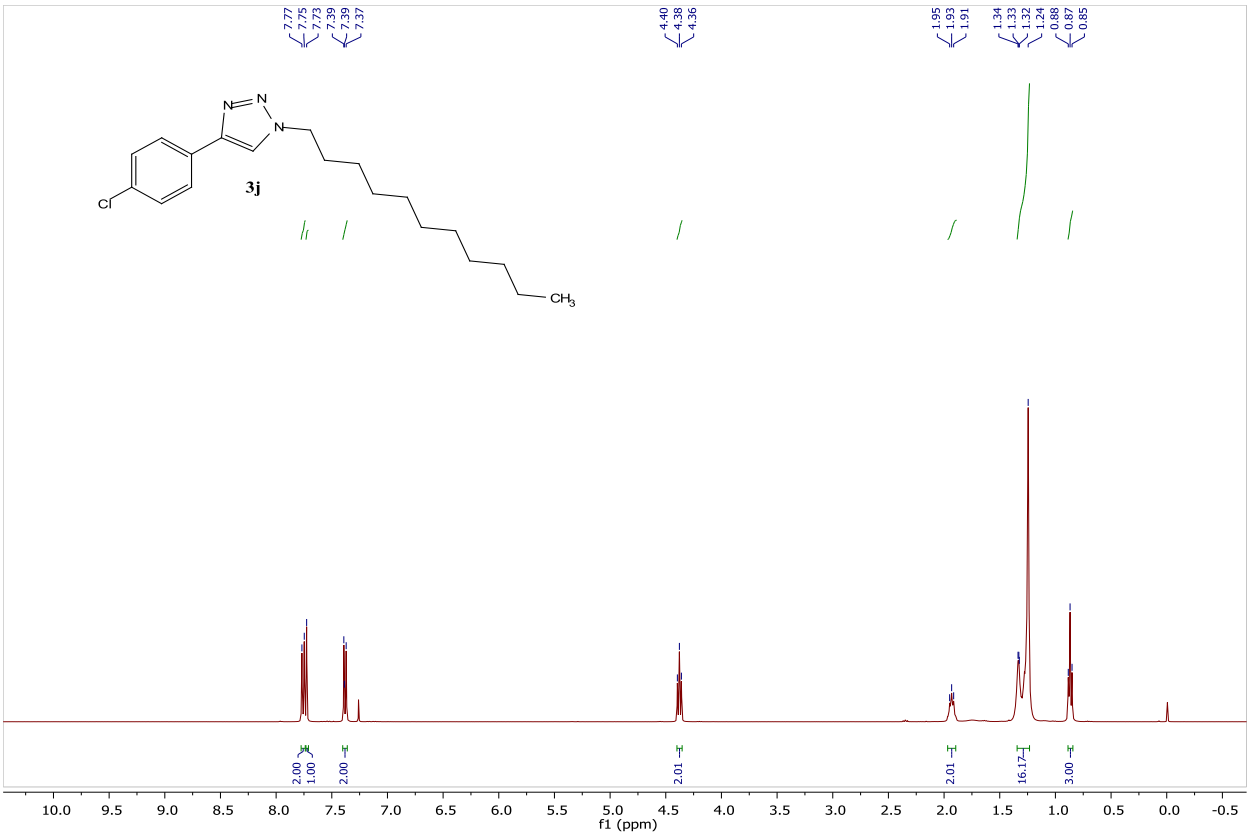
Cl
N
N
N
3i
CH3
f1 (ppm)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4.40
4.38
4.36
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1.93
1.91
1.34
1.33
1.32
1.24
0.88
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Cl
CH3
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1.00
2.00
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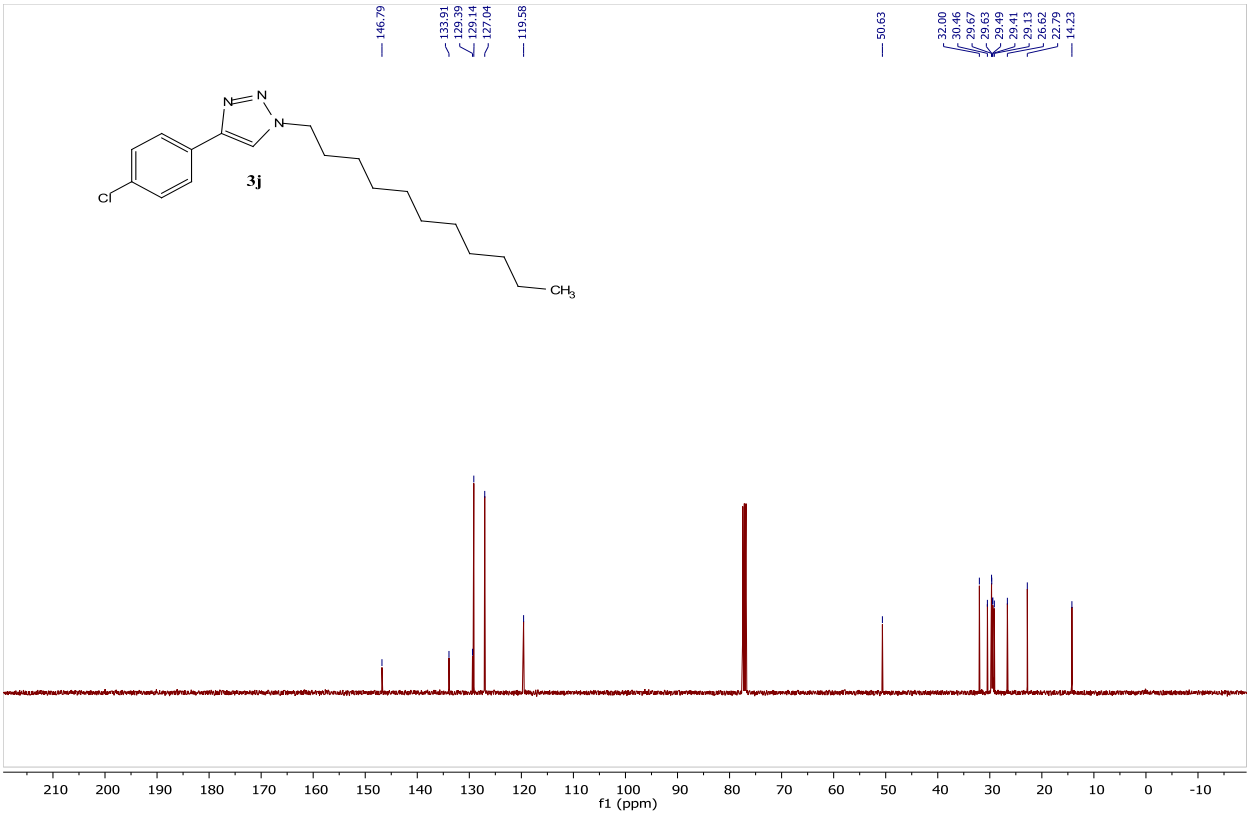
2.01
16.17
3.00
f1 (ppm)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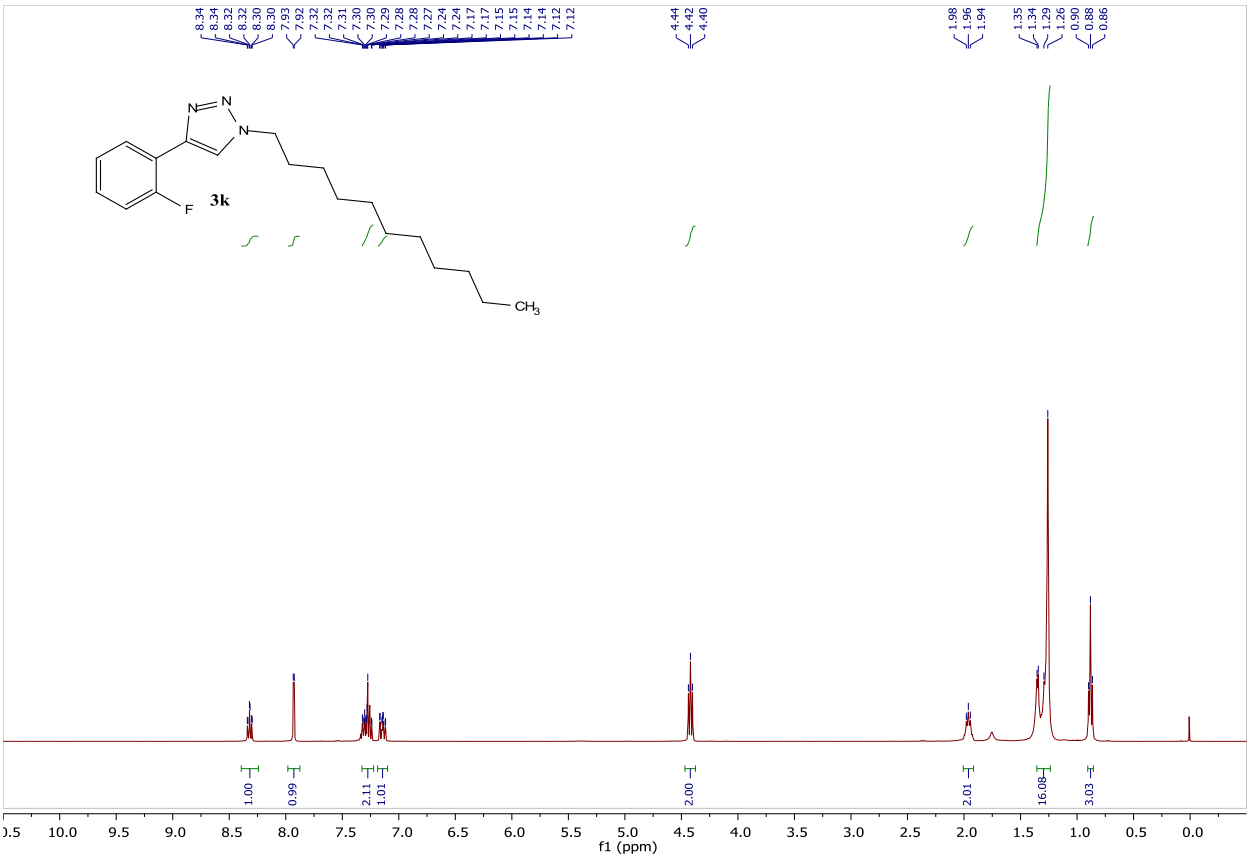
29.41
29.13
26.62
22.79
14.23
3j
Cl
CH3
f1 (ppm)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7.24
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7.15
7.14
7.14
7.12
7.12
4.44
4.42
4.40
1.98
1.96
1.94
1.35
1.34
1.29
1.26
0.90
0.88
0.86
3k
F
CH3
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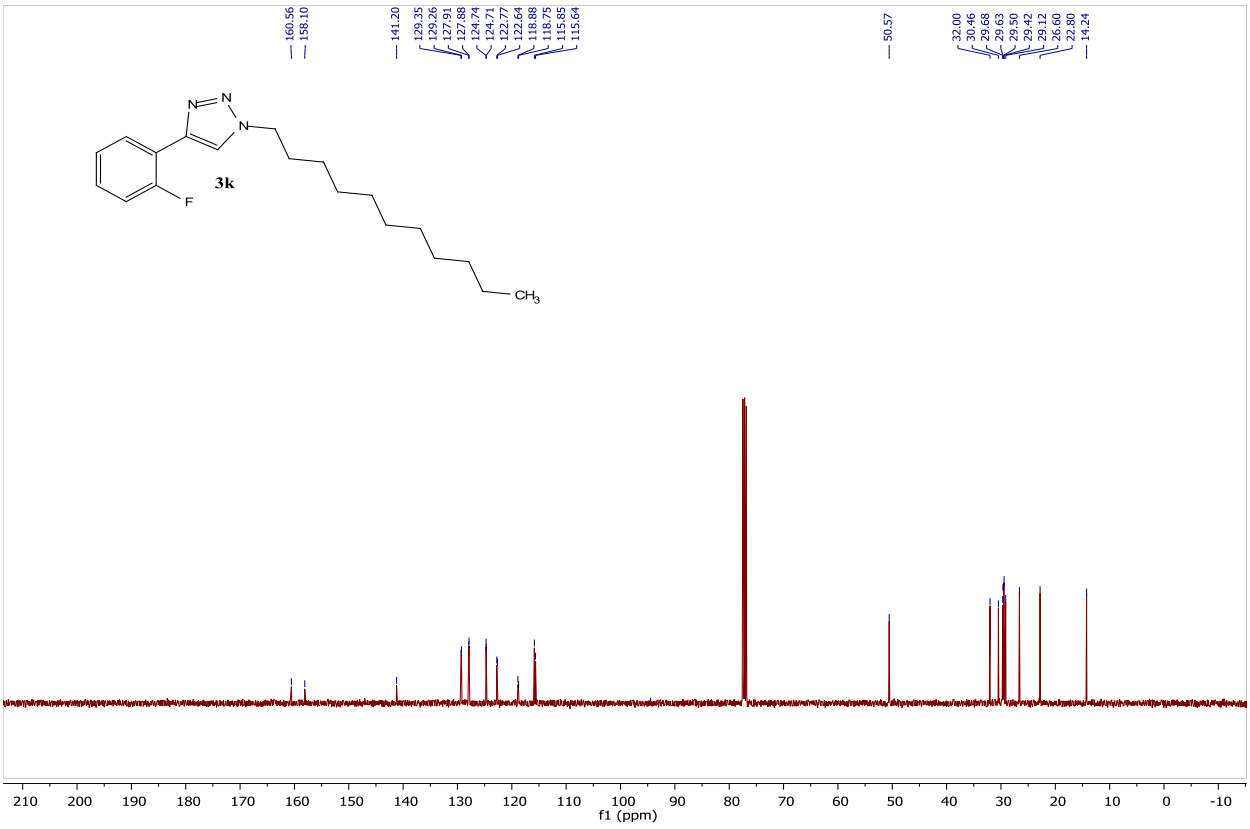
0.99
2.11
1.01
2.00
2.01
16.08
3.03
f1 (ppm)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115.64
50.57
32.00
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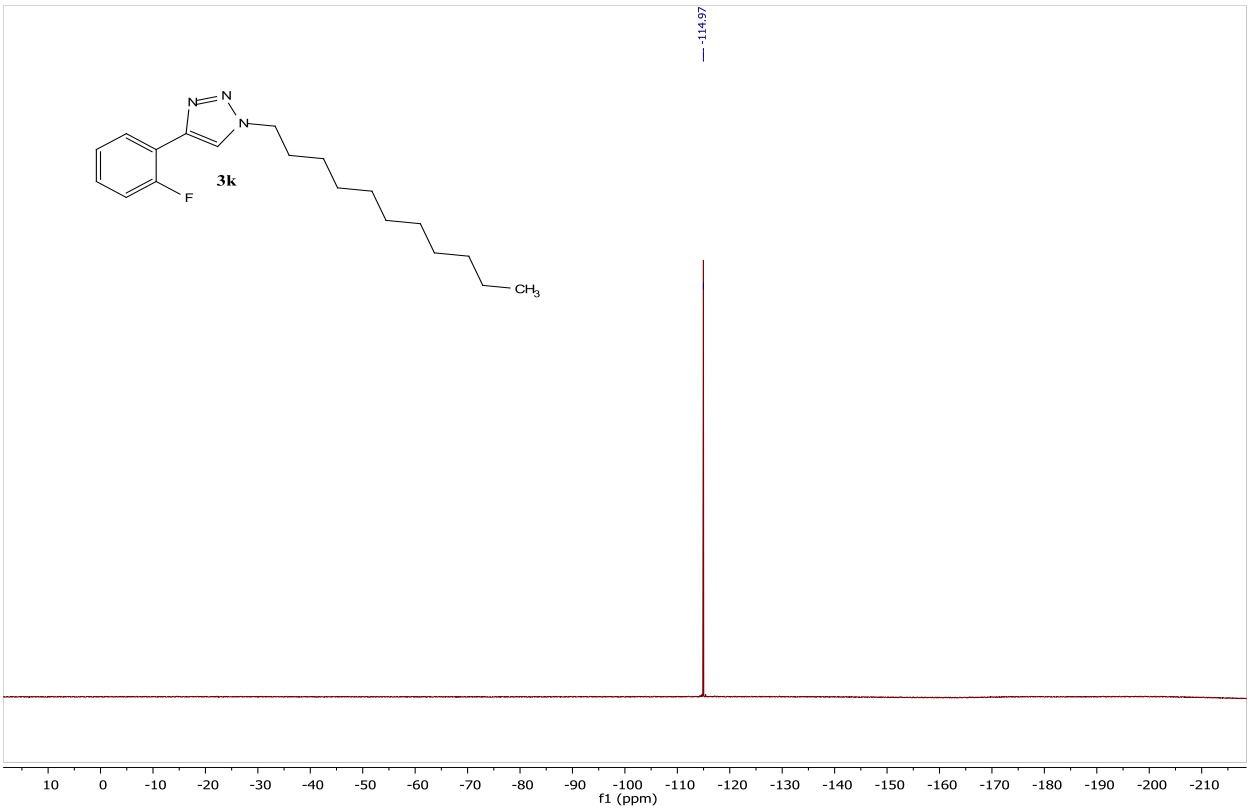
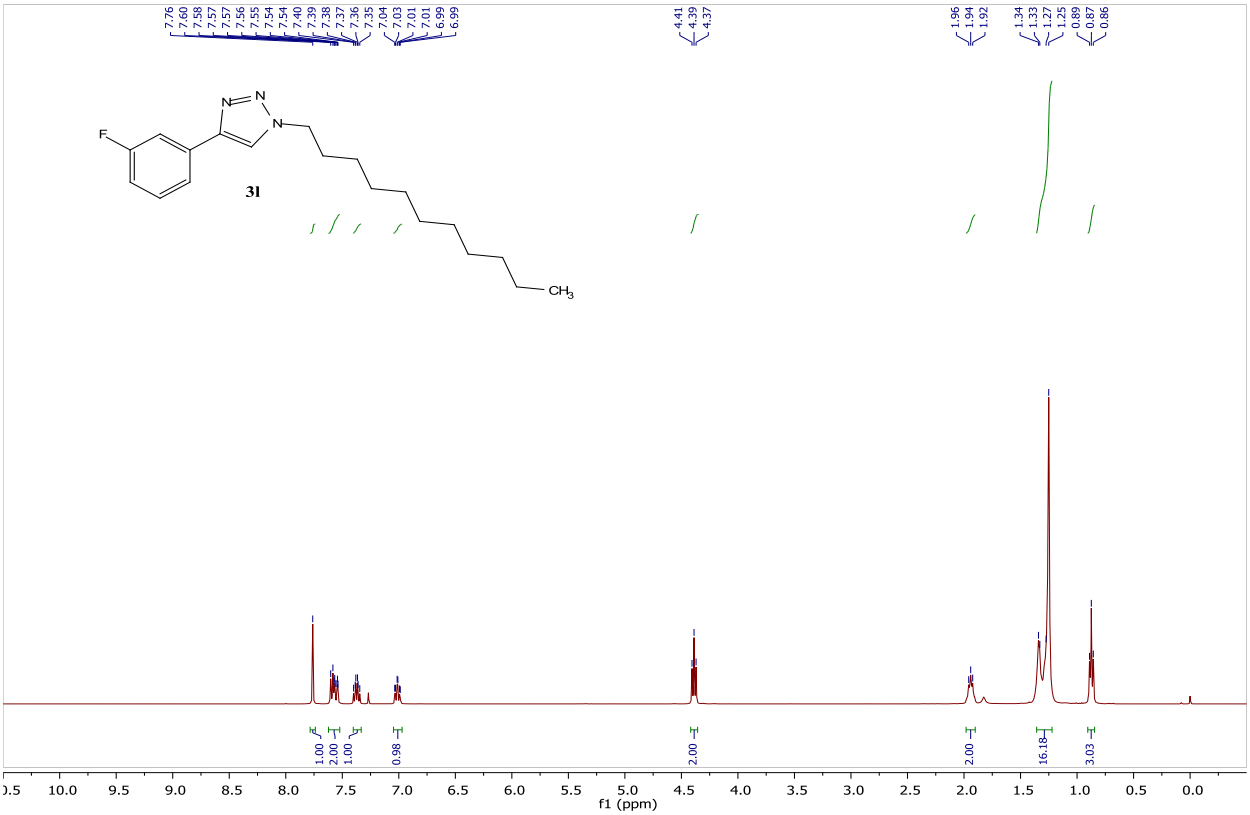
29.50
29.42
29.12
26.60
22.90
14.24
3k
F
CH3
f1 (ppm)


-114.97
3k
F
CH3
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)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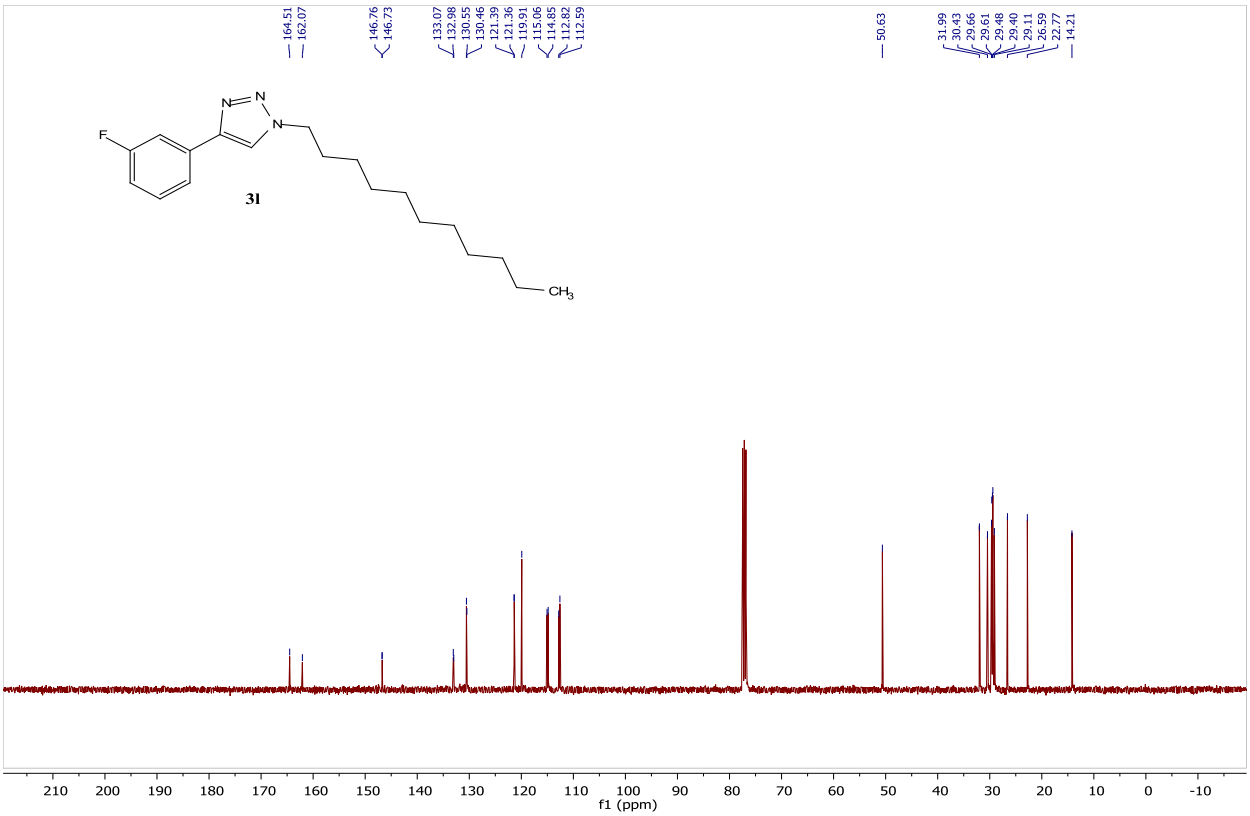
4.41 4.39 4.37
1.96 1.94 1.92
1.34 1.33 1.27 1.25
0.89 0.87 0.86
3l
F
CH3
1.00 2.00 1.00 0.98 2.00 2.00 16.18 3.03
0.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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115.06
114.85
112.82
112.59
50.63
31.99
30.43
29.66
29.61
29.48
29.40
29.11
26.59
22.77
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F
N
N
31
CH3
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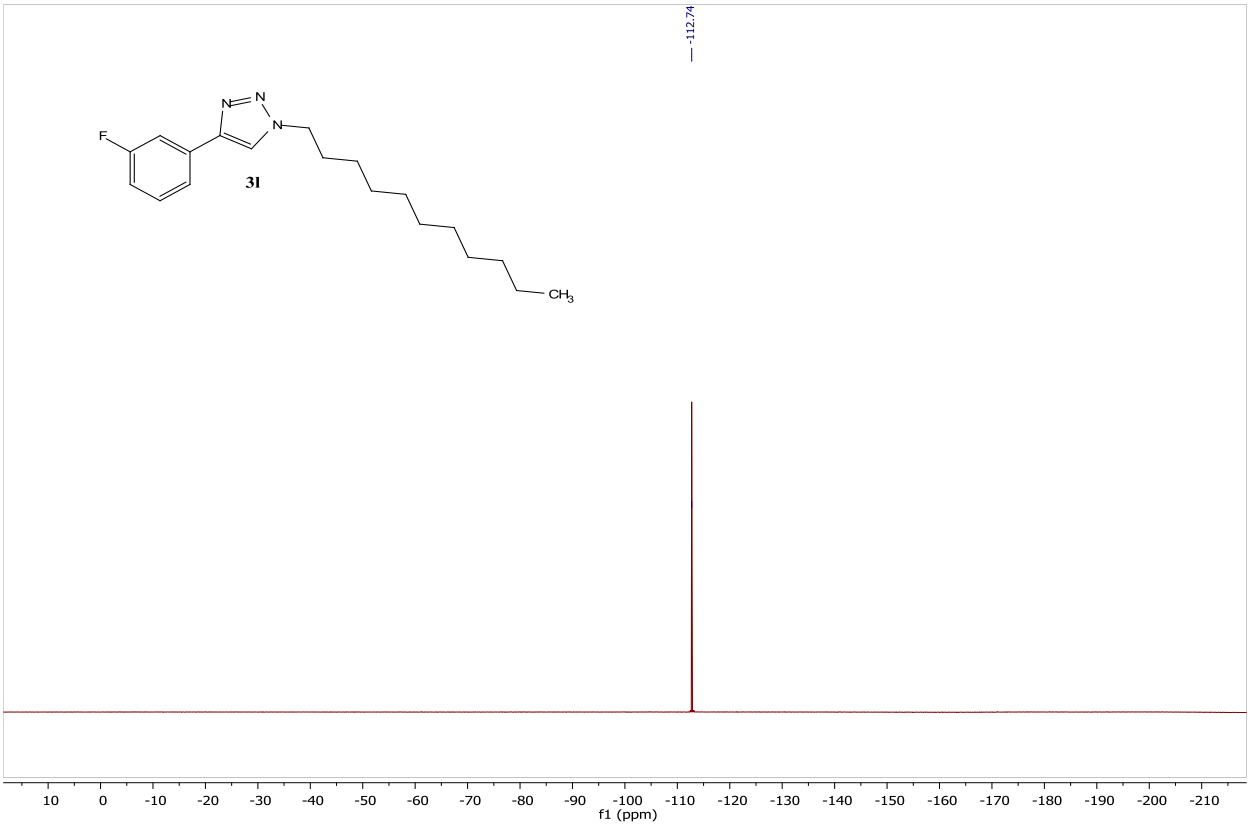
f1 (ppm)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CH3
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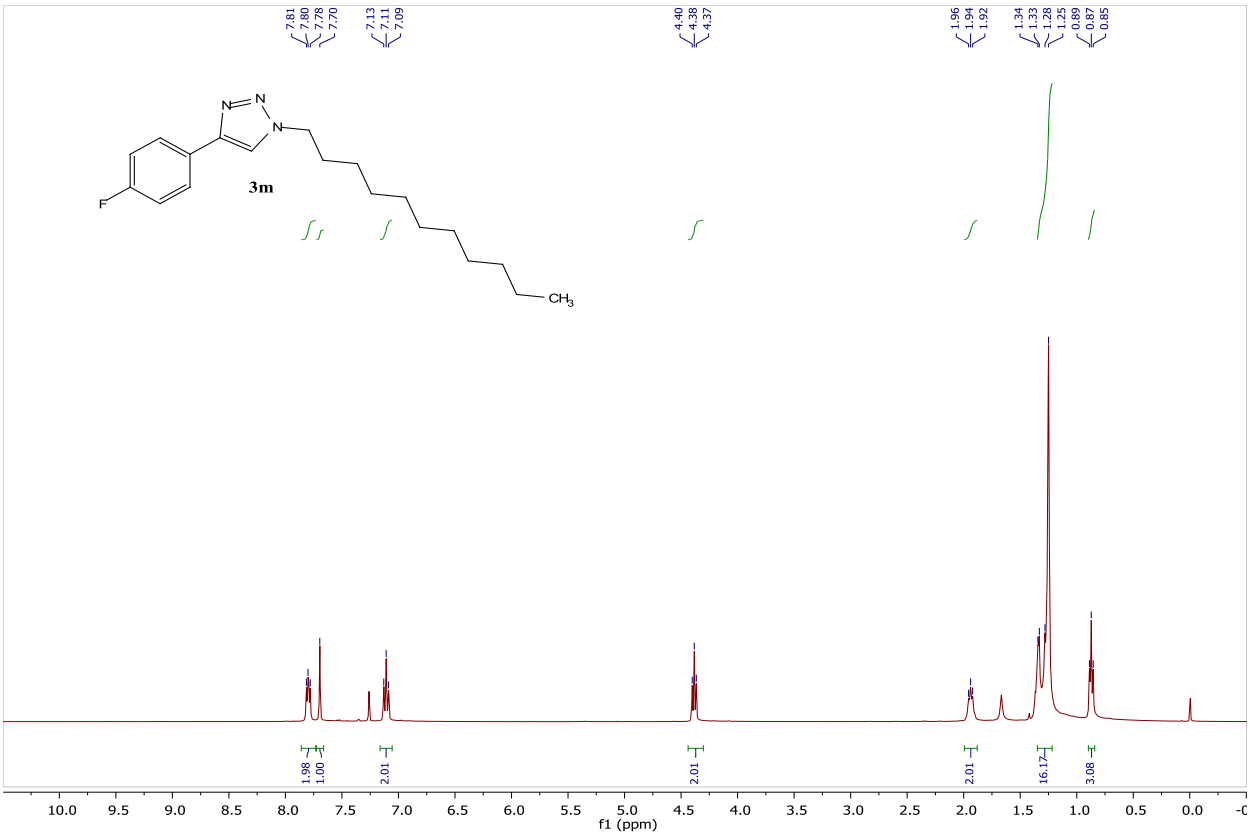
f1 (ppm)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4.37
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1.94
1.92
1.34
1.33
1.28
1.25
0.89
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F
CH3
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1.00
2.01
2.01
2.01
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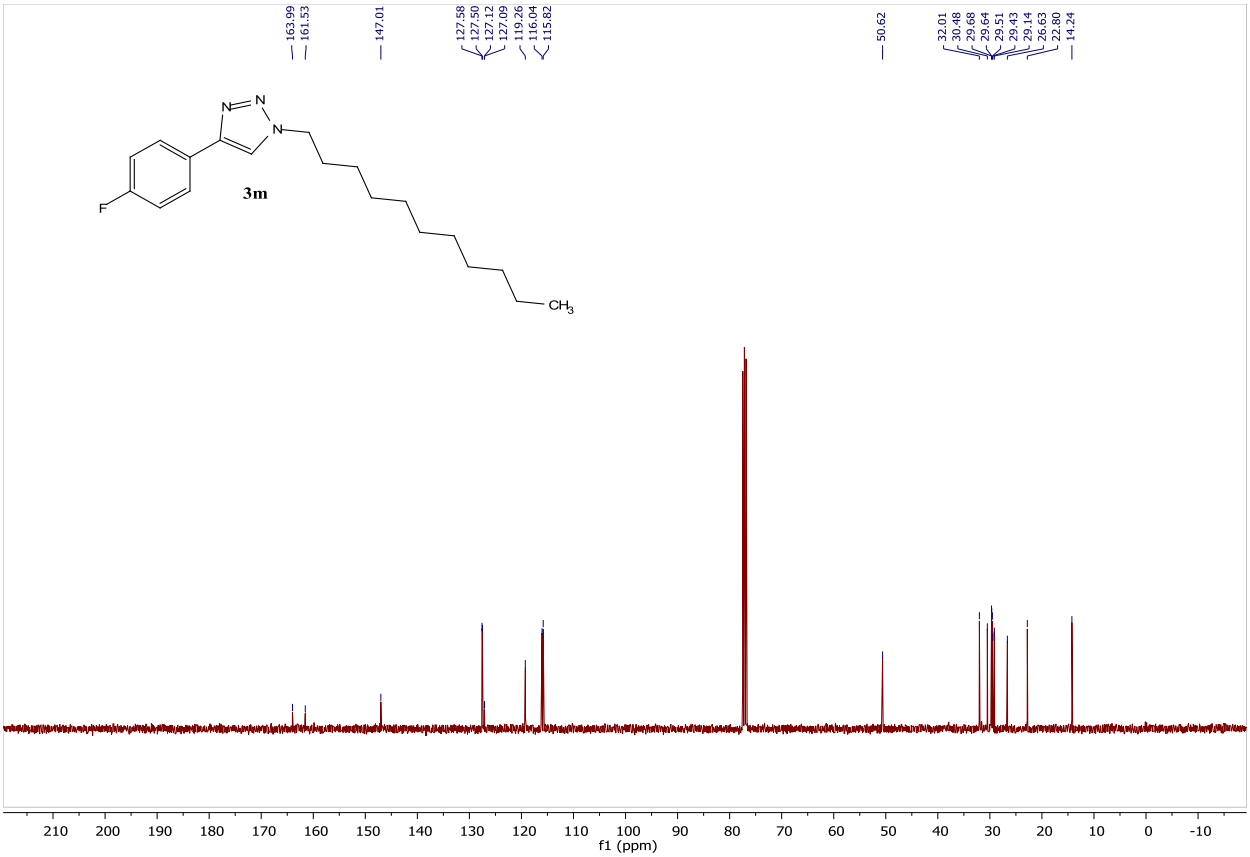
3.08
f1 (ppm)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50.62
32.01
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29.51
29.43
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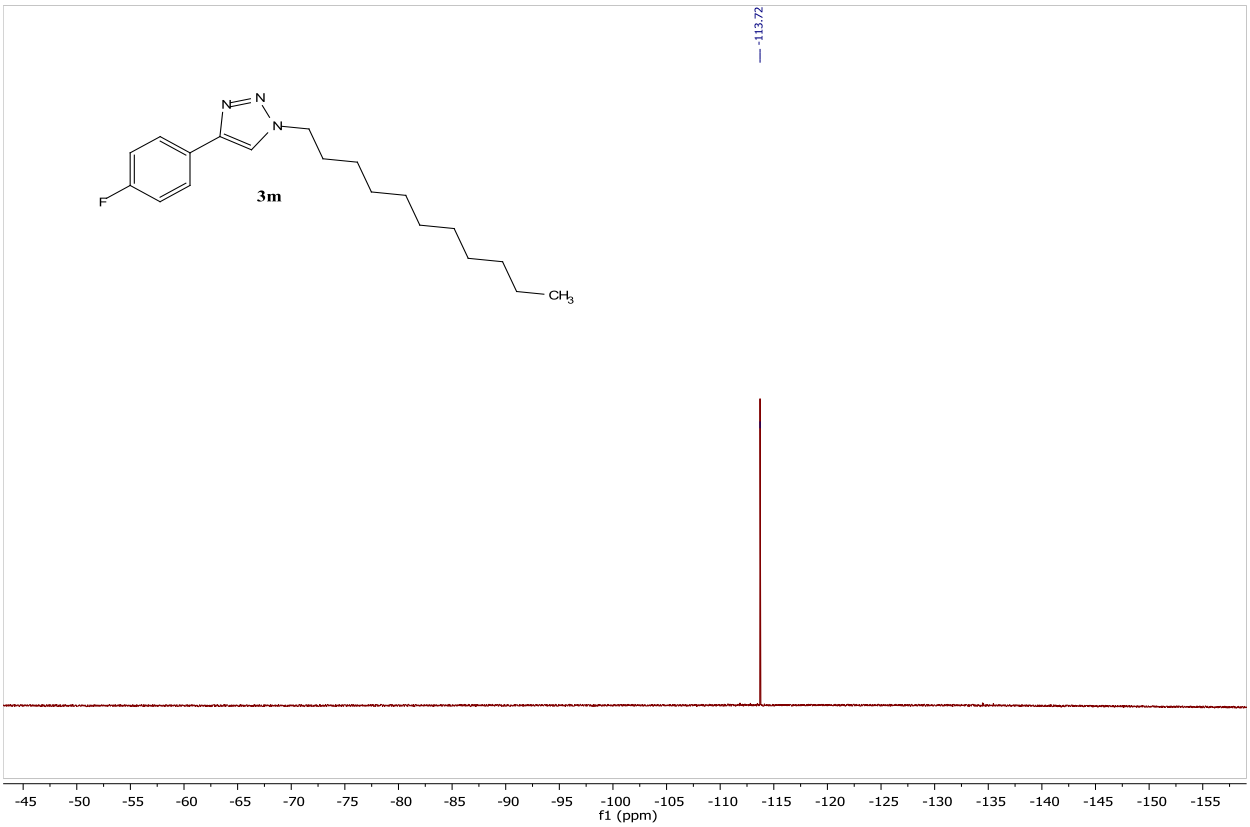
26.63
22.80
14.24
3m
F
CH3
f1 (ppm)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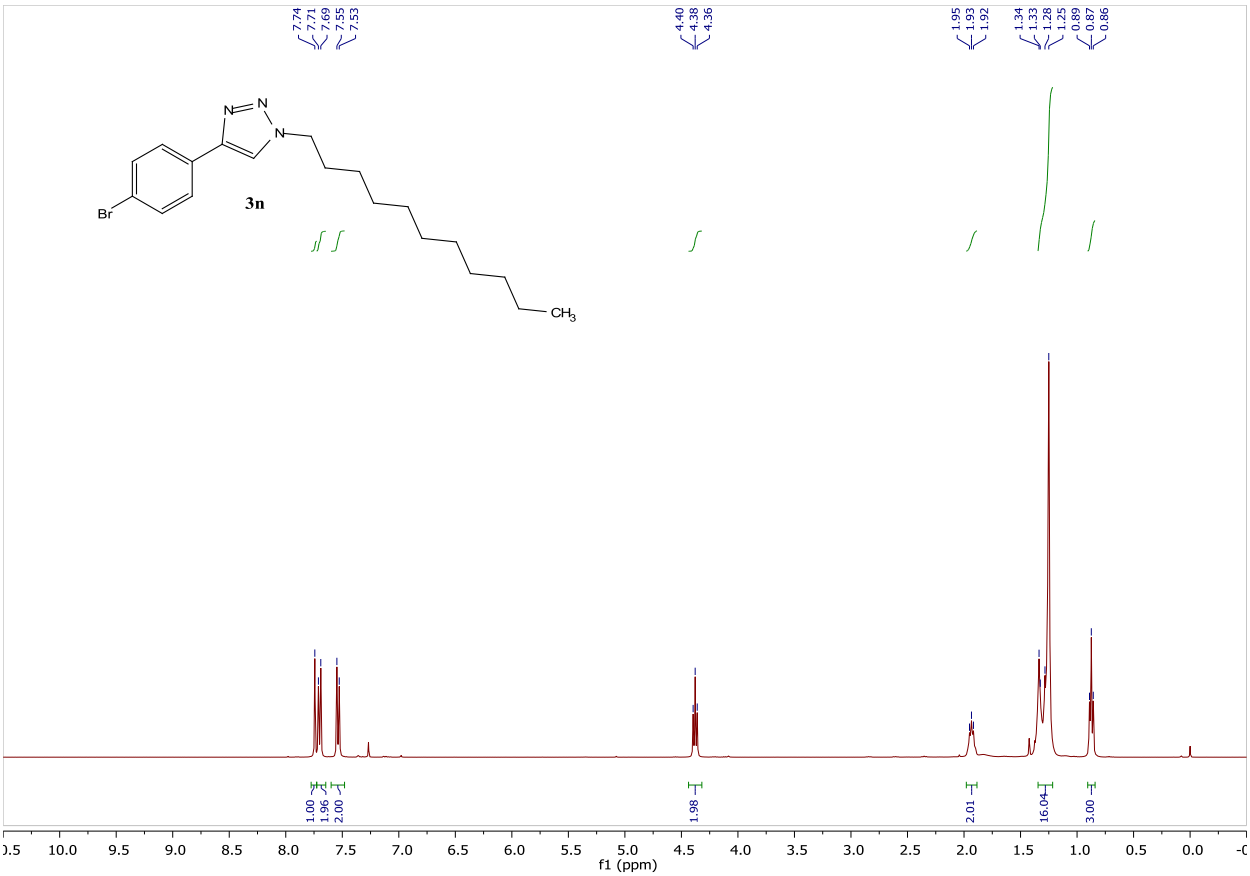
-120
-125
-130
-135
-140
-145
-150
-155
f1 (ppm)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1.34
1.33
1.28
1.25
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0.87
0.86
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CH3
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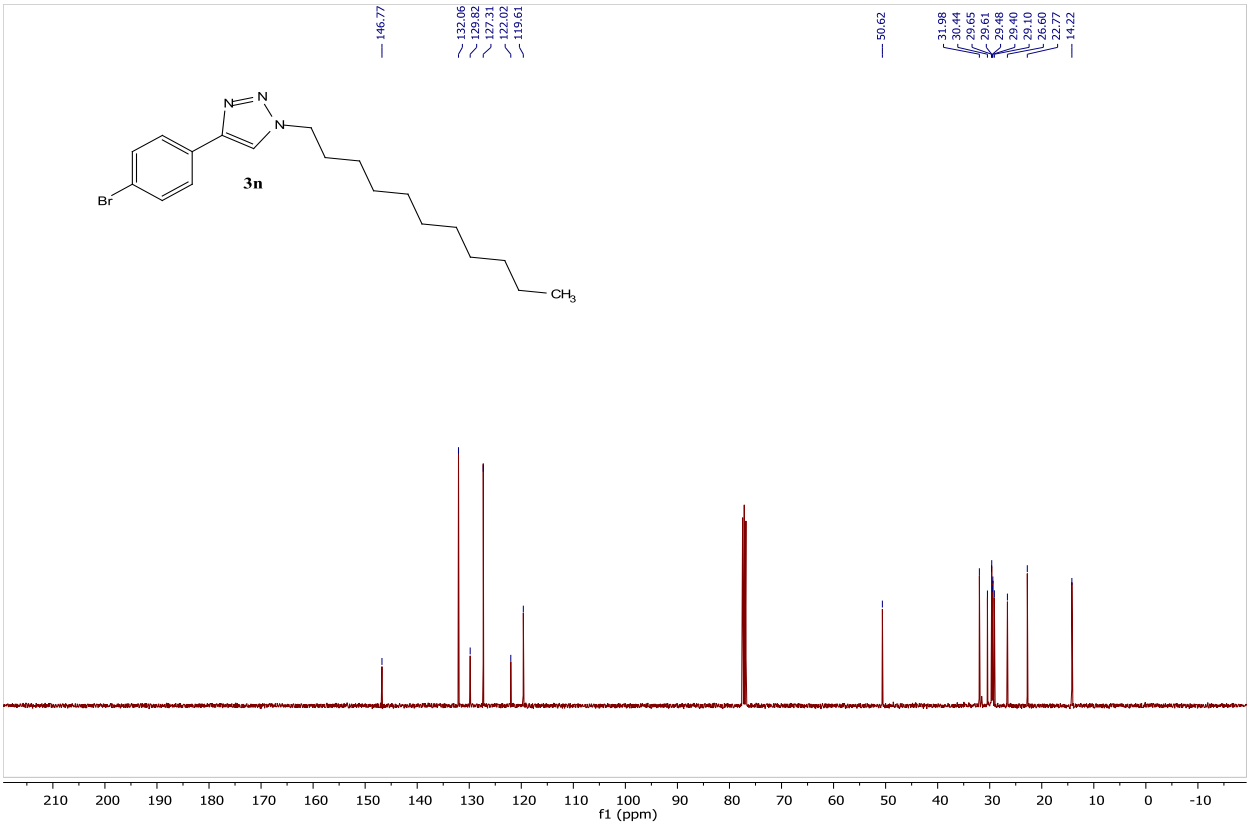
1.96
2.00
1.98
2.01
16.04
3.00
f1 (ppm)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N
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Br
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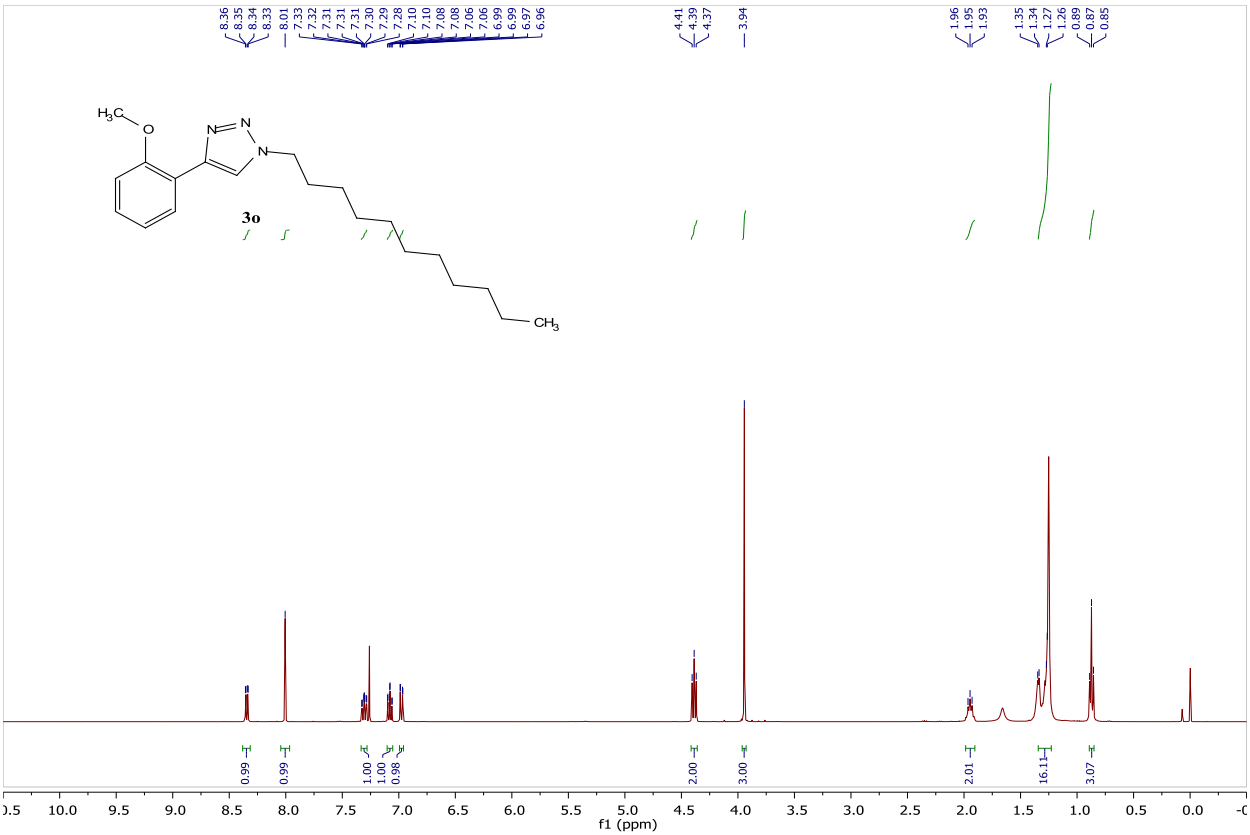
CH3
f1 (ppm)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7.10
7.10
7.08
7.08
7.06
7.06
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6.99
6.97
6.96
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4.37
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1.27
1.26
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0.85
H3C
O
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N
N
3o
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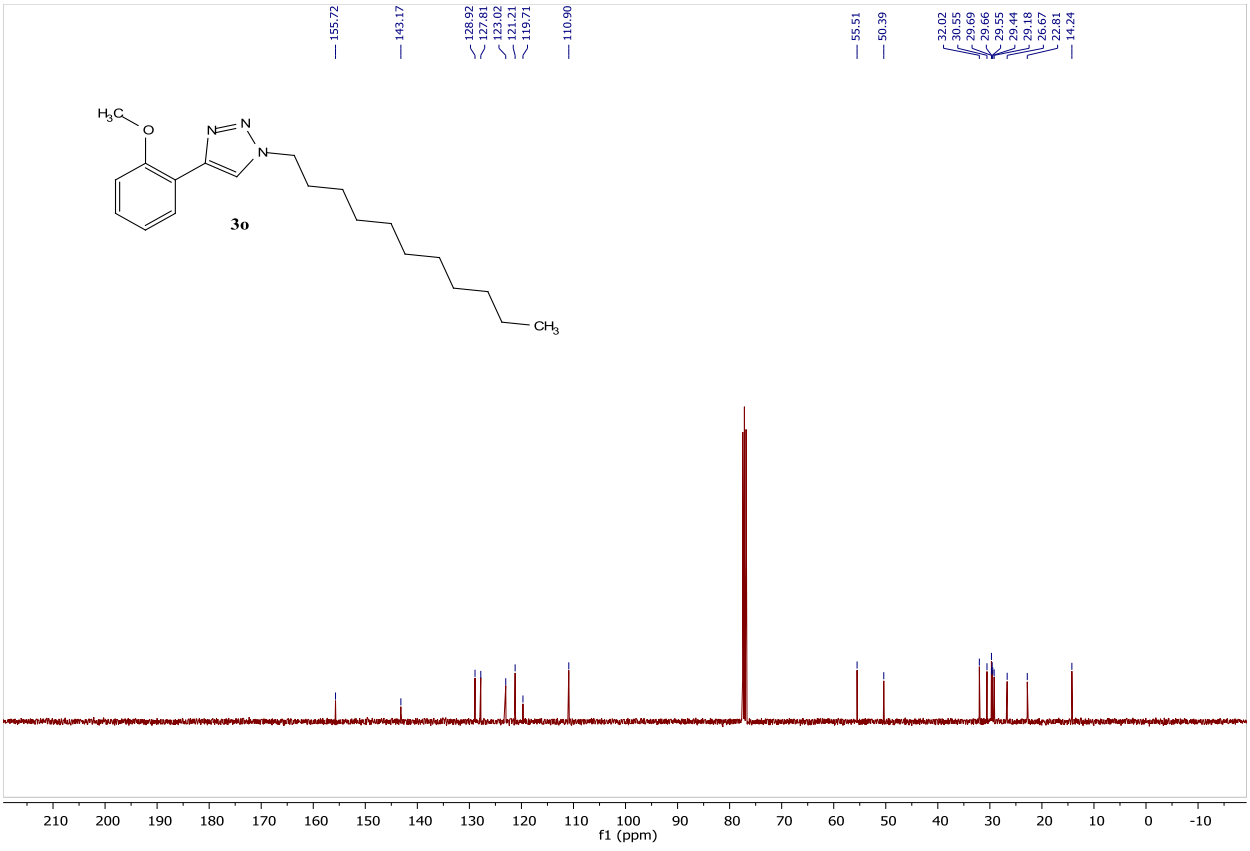
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29.69
29.66
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N
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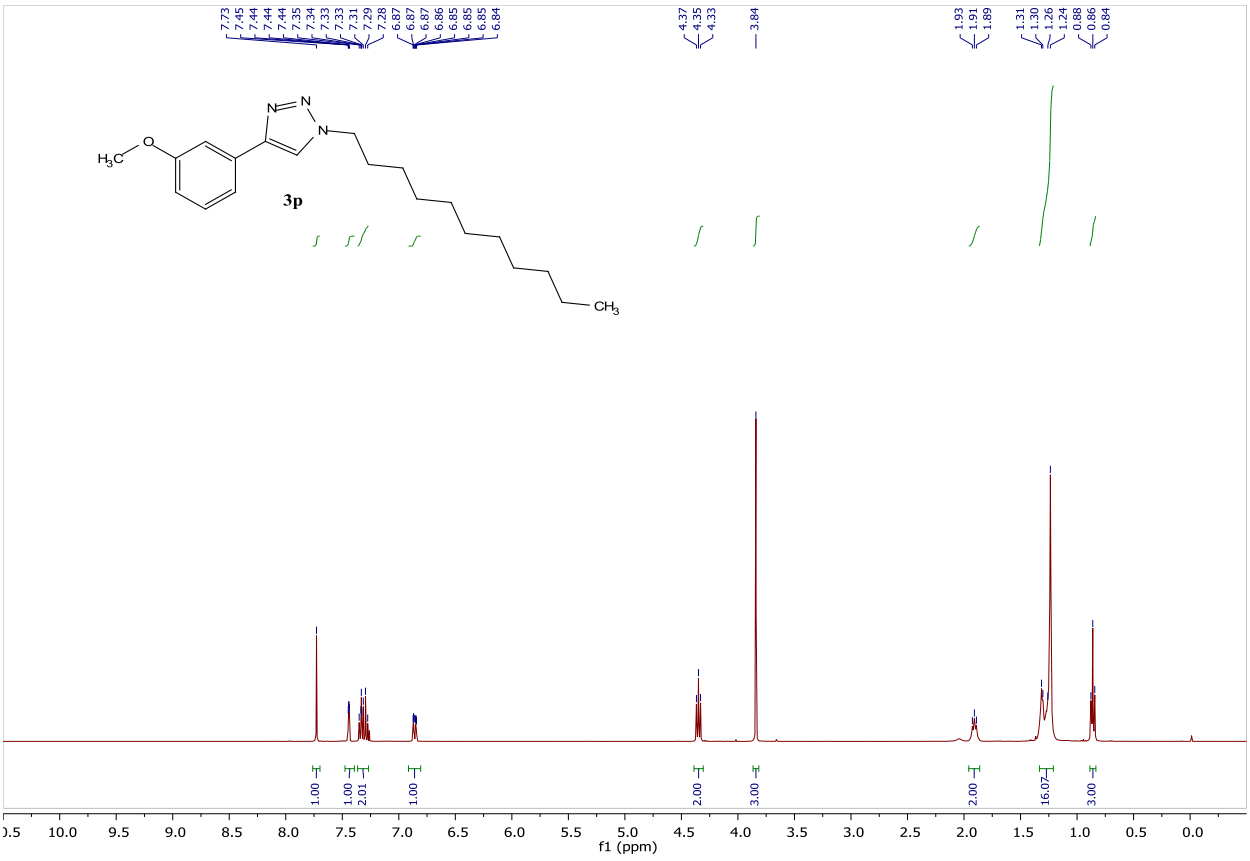
N
3o
CH3
f1 (ppm)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1.91
1.89
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1.30
1.26
1.24
0.88
0.86
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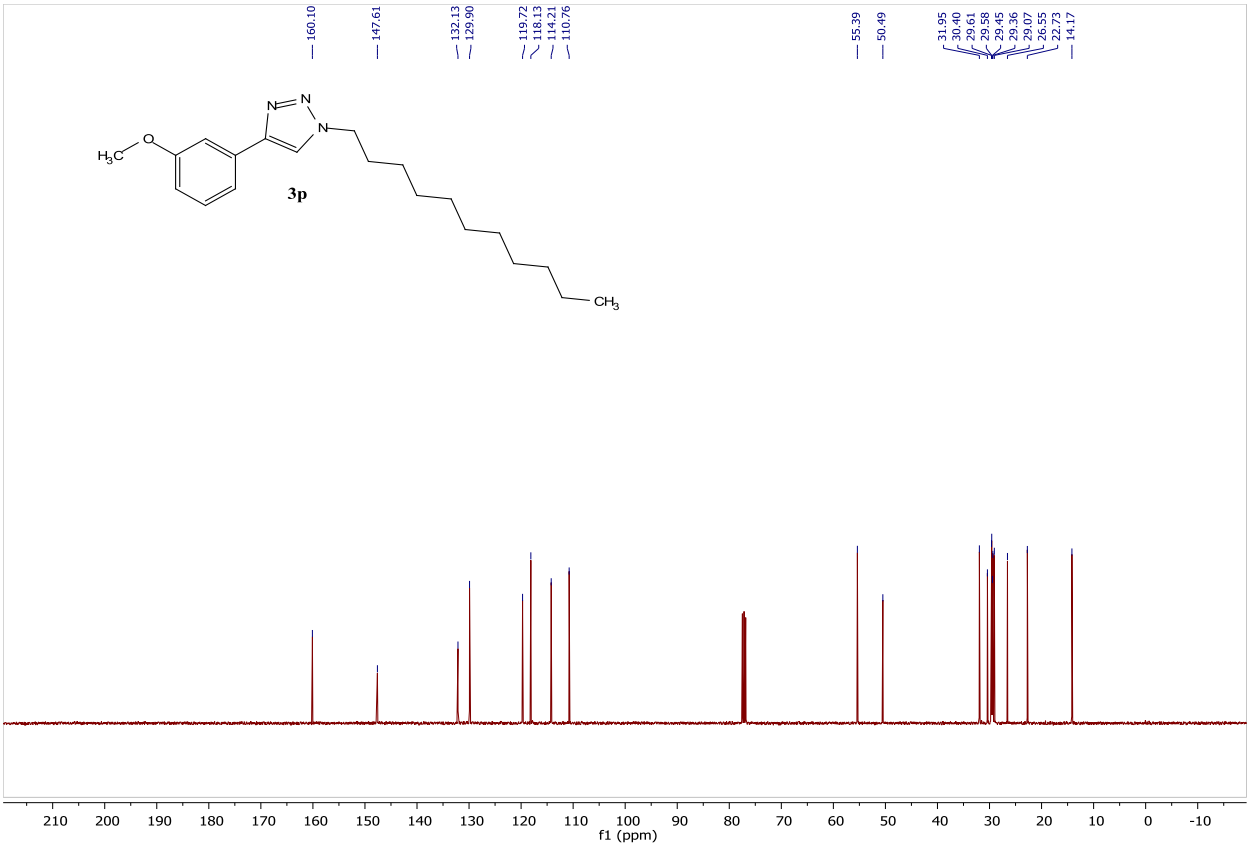
2.01
1.00
2.00
3.00
2.00
16.07
3.00
f1 (ppm)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26.55
22.73
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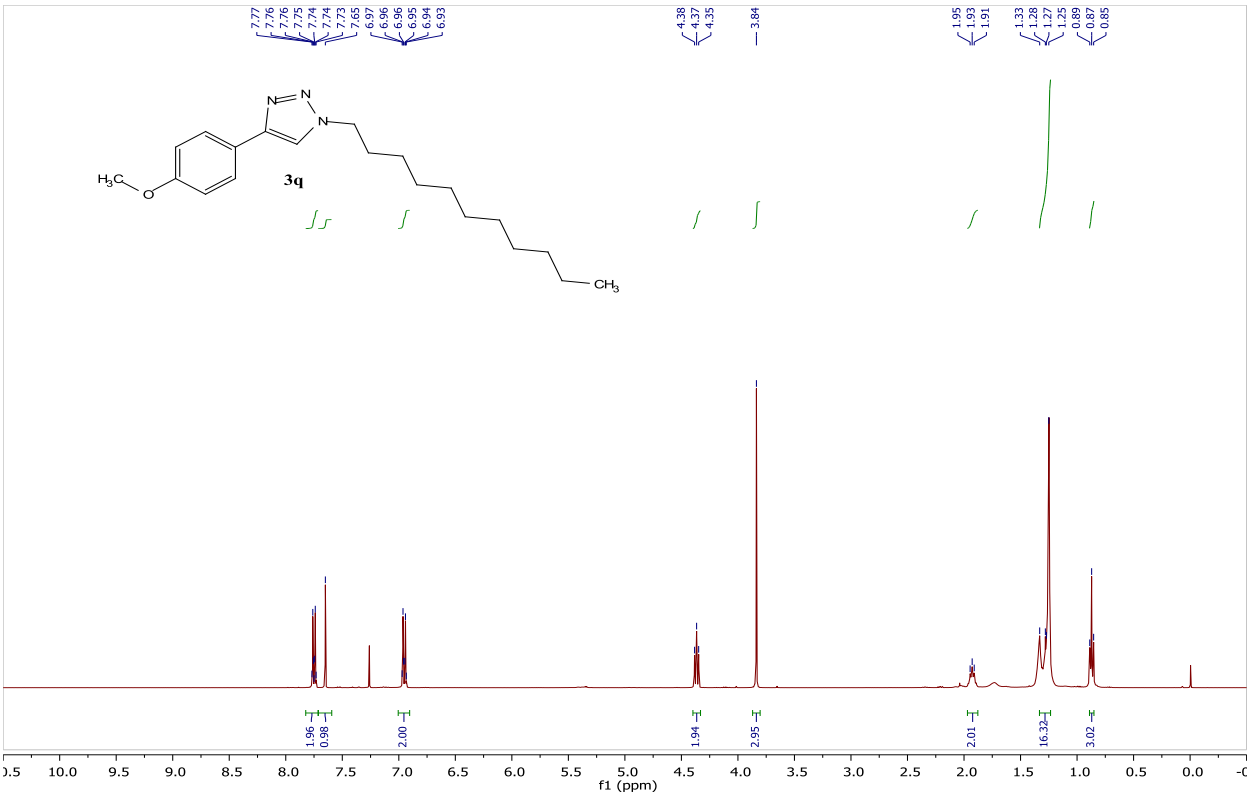
f1 (ppm)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1.28
1.27
1.25
0.89
0.87
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0.98
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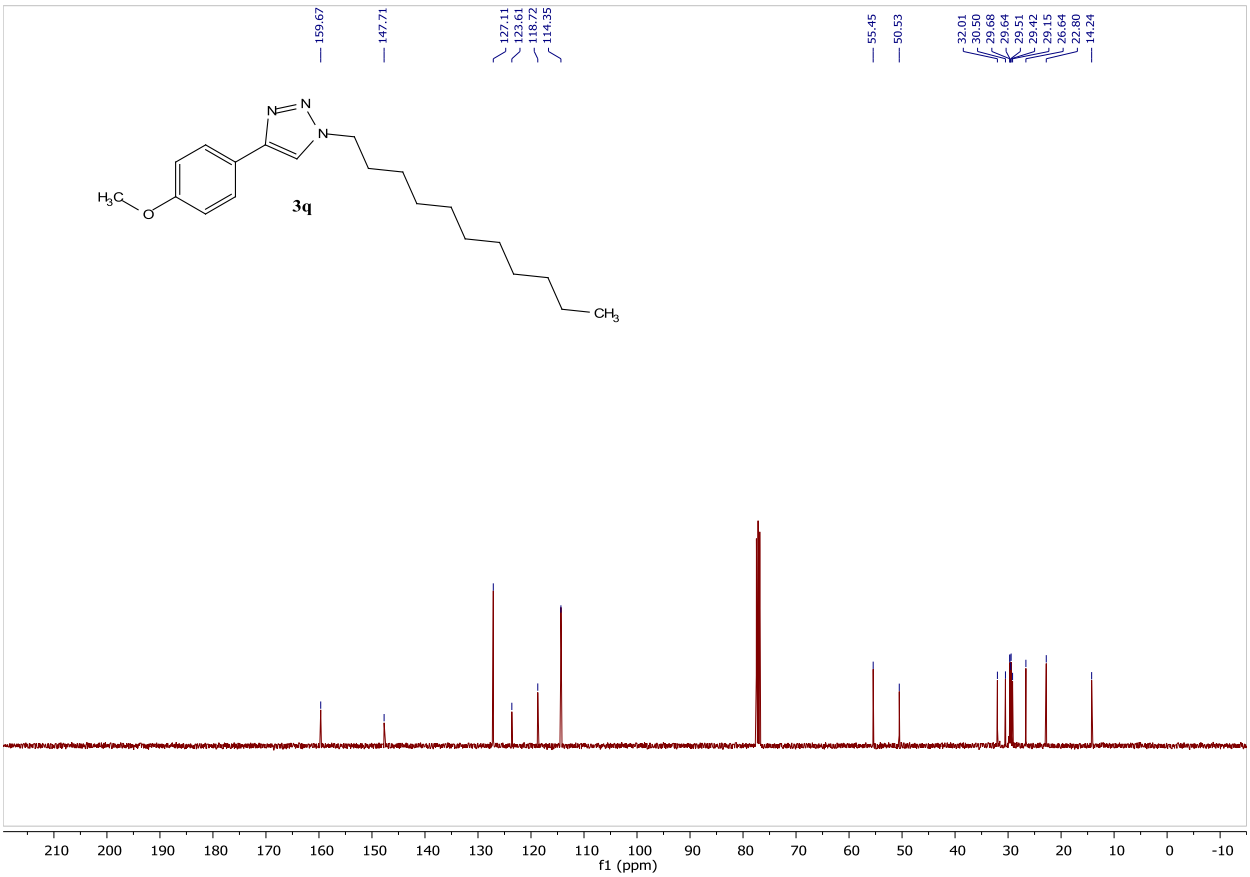
2.01
16.32
3.02
f1 (ppm)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22.80
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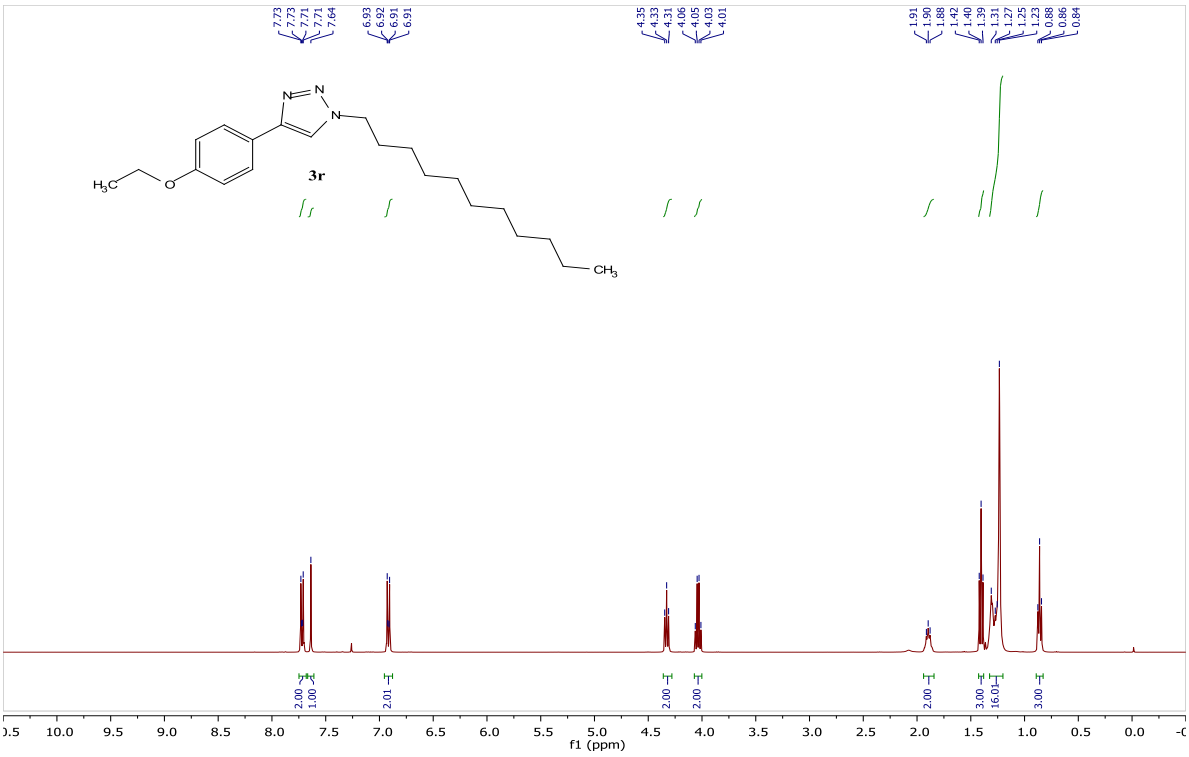
3q
CH3
f1 (ppm)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7.71
7.64
6.93
6.92
6.91
6.91
4.35
4.33
4.31
4.06
4.05
4.03
4.01
1.91
1.90
1.88
1.42
1.40
1.39
1.31
1.27
1.25
1.23
0.88
0.86
0.84
3r
CH3
H3C
O
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N
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2.00
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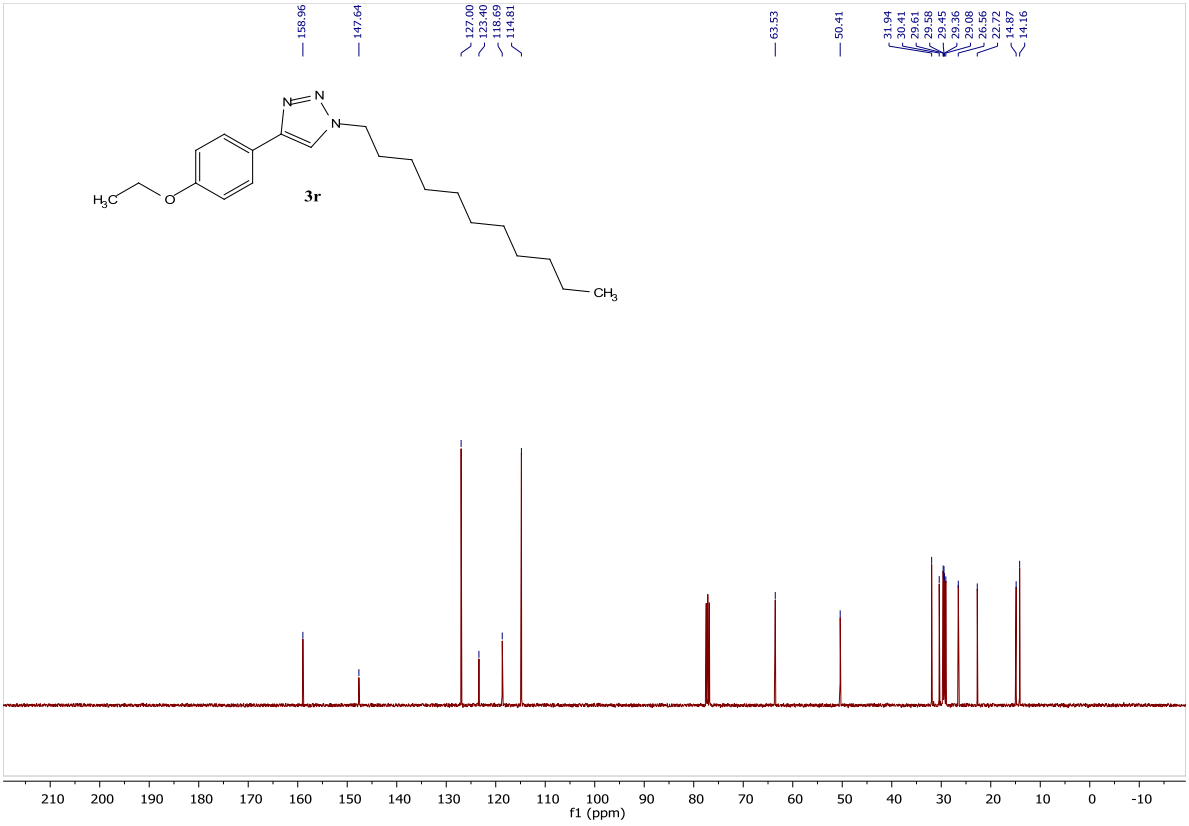
f1 (ppm)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29.45
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26.56
22.72
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14.16
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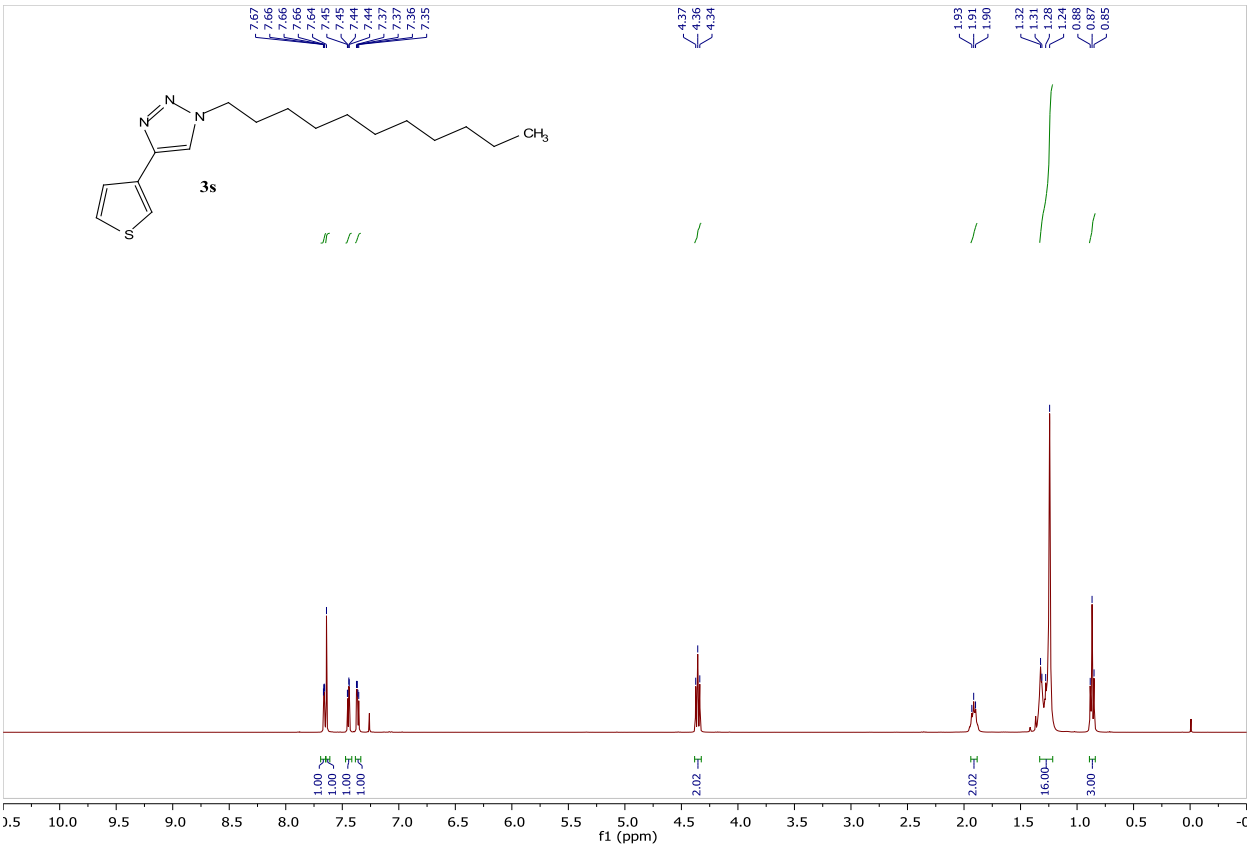
CH3
H3C
O
N
N
f1 (ppm)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7.37
7.36
7.35
4.37
4.36
4.34
1.93
1.91
1.90
1.32
1.31
1.28
1.24
0.88
0.87
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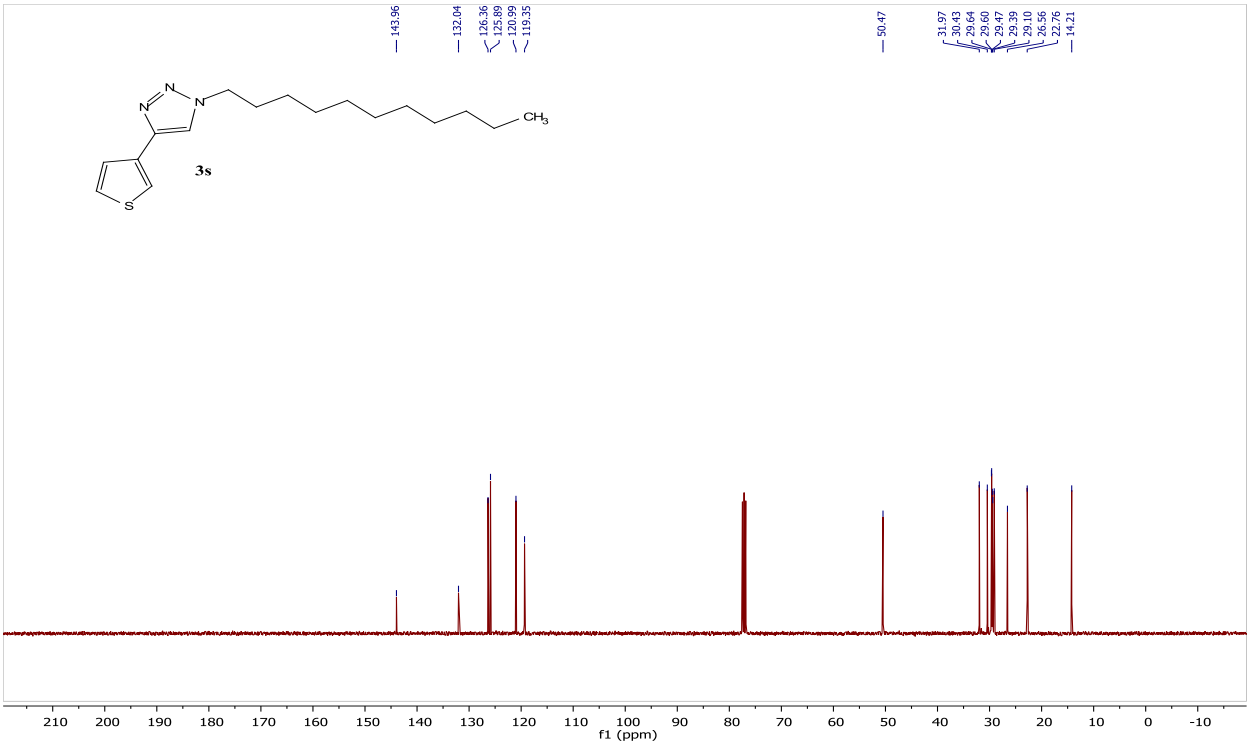
1.00
1.00
1.00
2.02
2.02
16.00
3.00
f1 (ppm)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50.47
31.97
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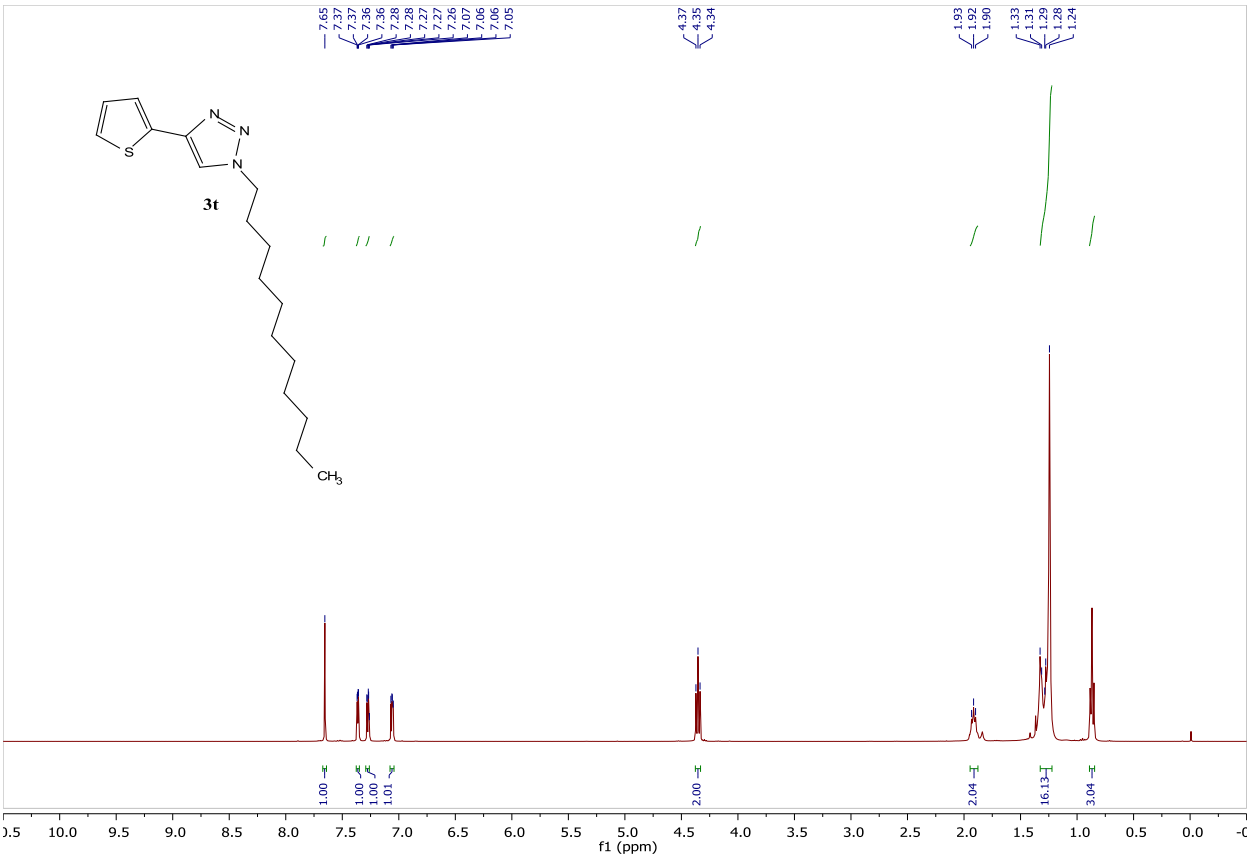
29.47
29.39
29.10
26.56
22.76
14.21
CH3
3s
f1 (ppm)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7.27
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7.07
7.06
7.06
7.05
4.37
4.35
4.34
1.93
1.92
1.90
1.33
1.31
1.29
1.28
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CH3
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1.00
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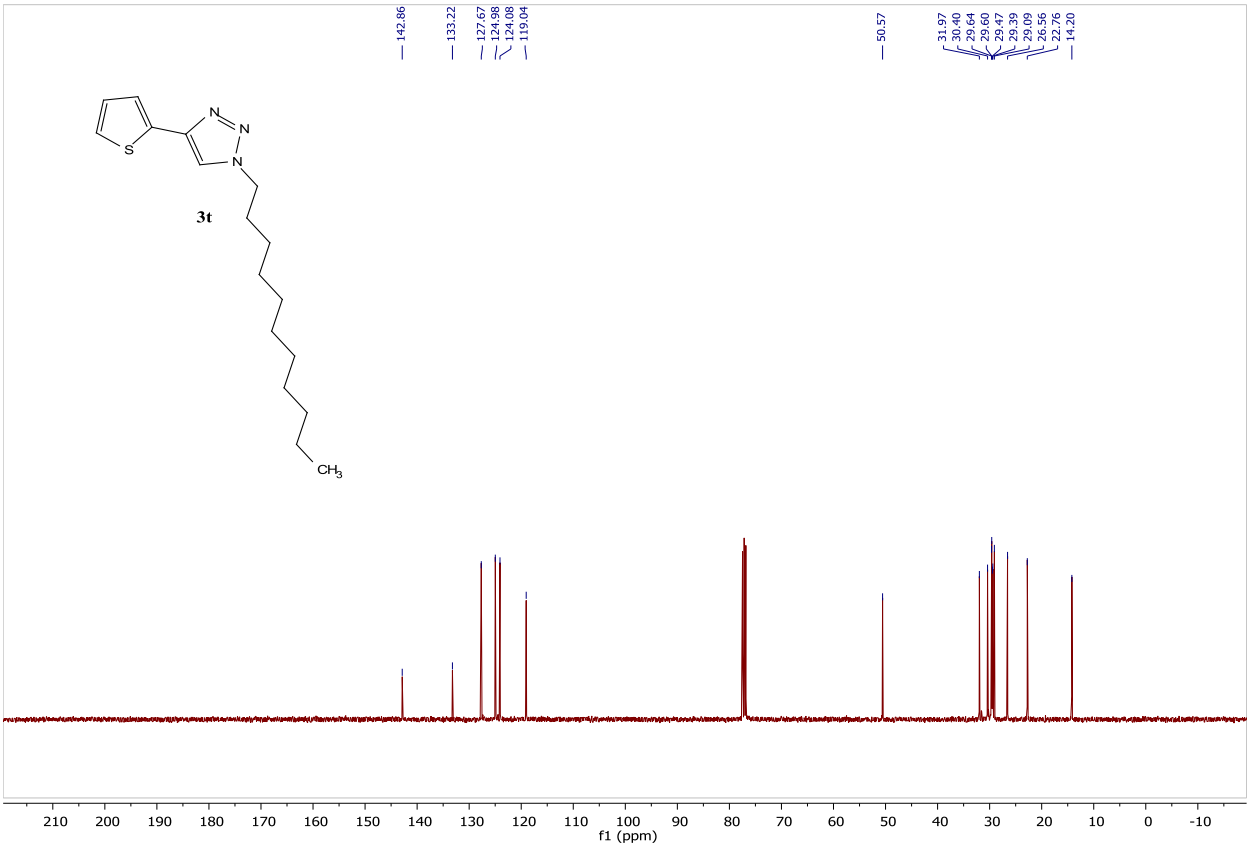
2.00
2.04
16.13
3.04
f1 (ppm)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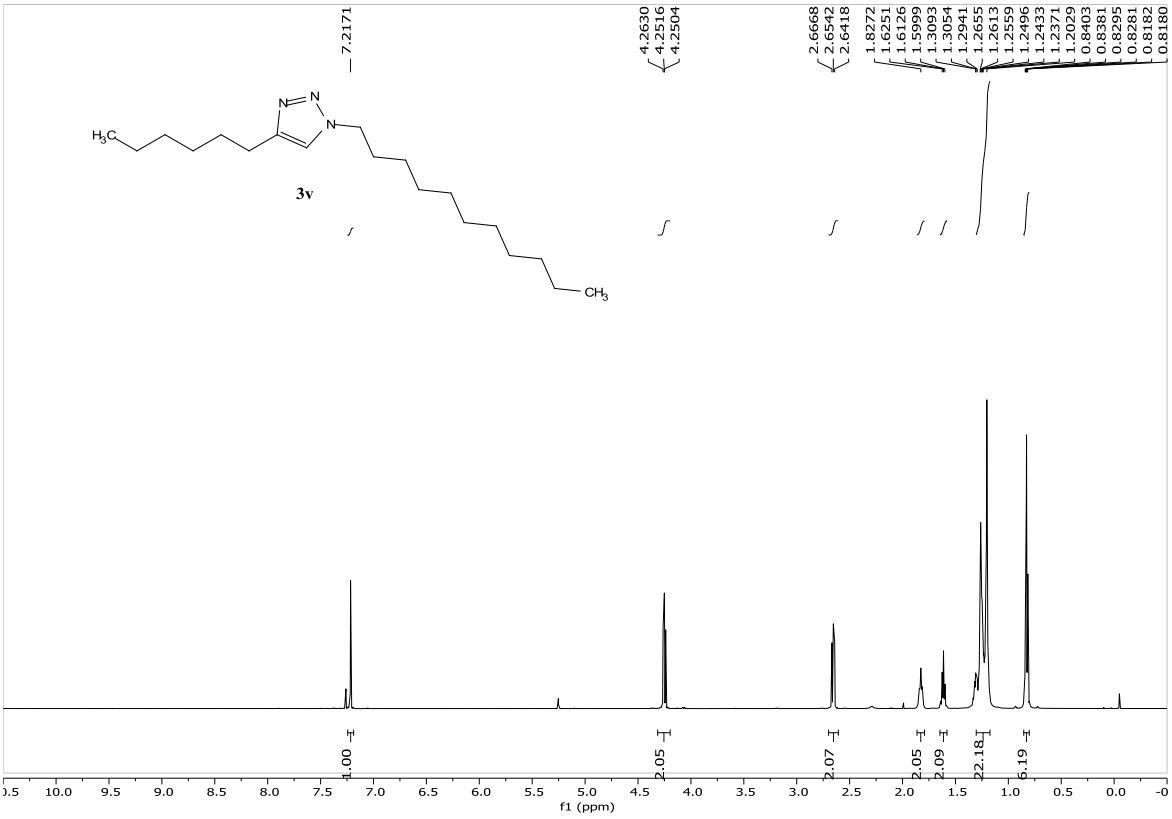
26.56
22.76
14.20
3t
CH3
f1 (ppm)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1.3093
1.3054
1.2941
1.2655
1.2613
1.2559
1.2496
1.2433
1.2371
1.2029
0.8403
0.8381
0.8295
0.8281
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CH3
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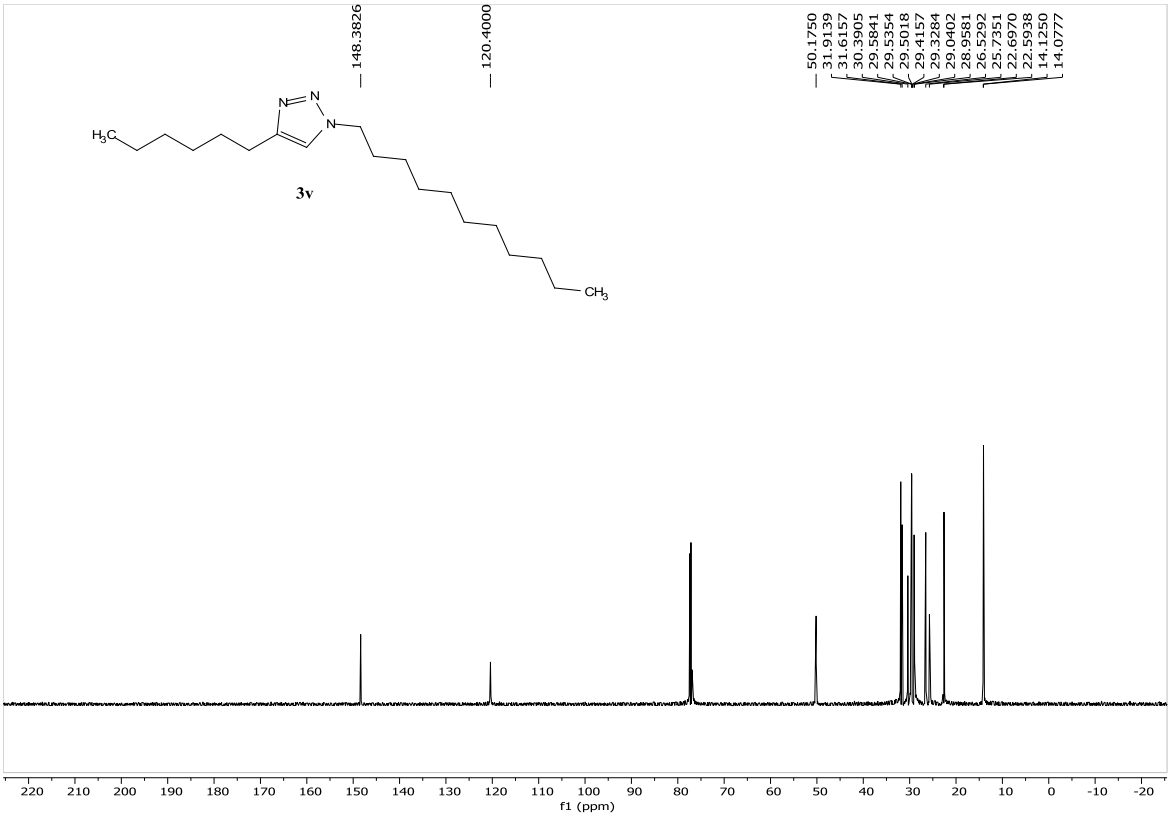
2.07
2.05
2.09
22.18
6.19
f1 (ppm)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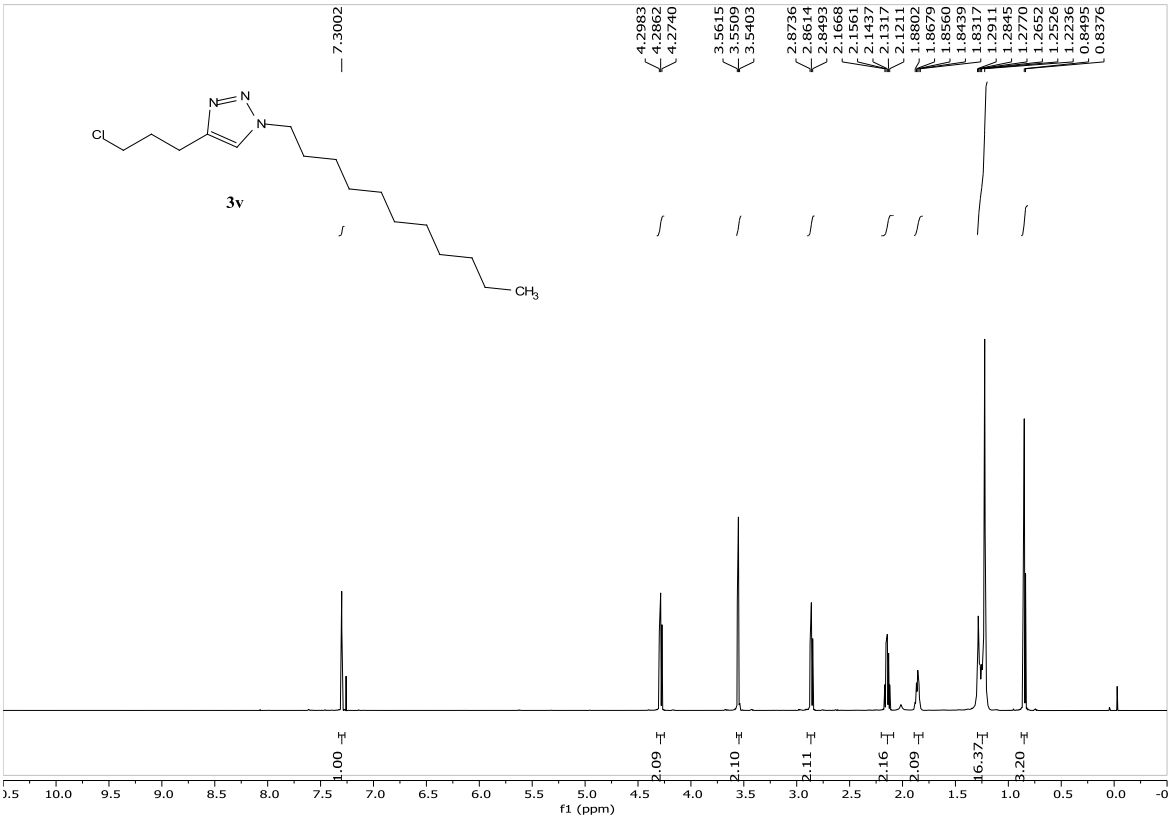
14.0777
3v
CH3
f1 (ppm)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4.2740
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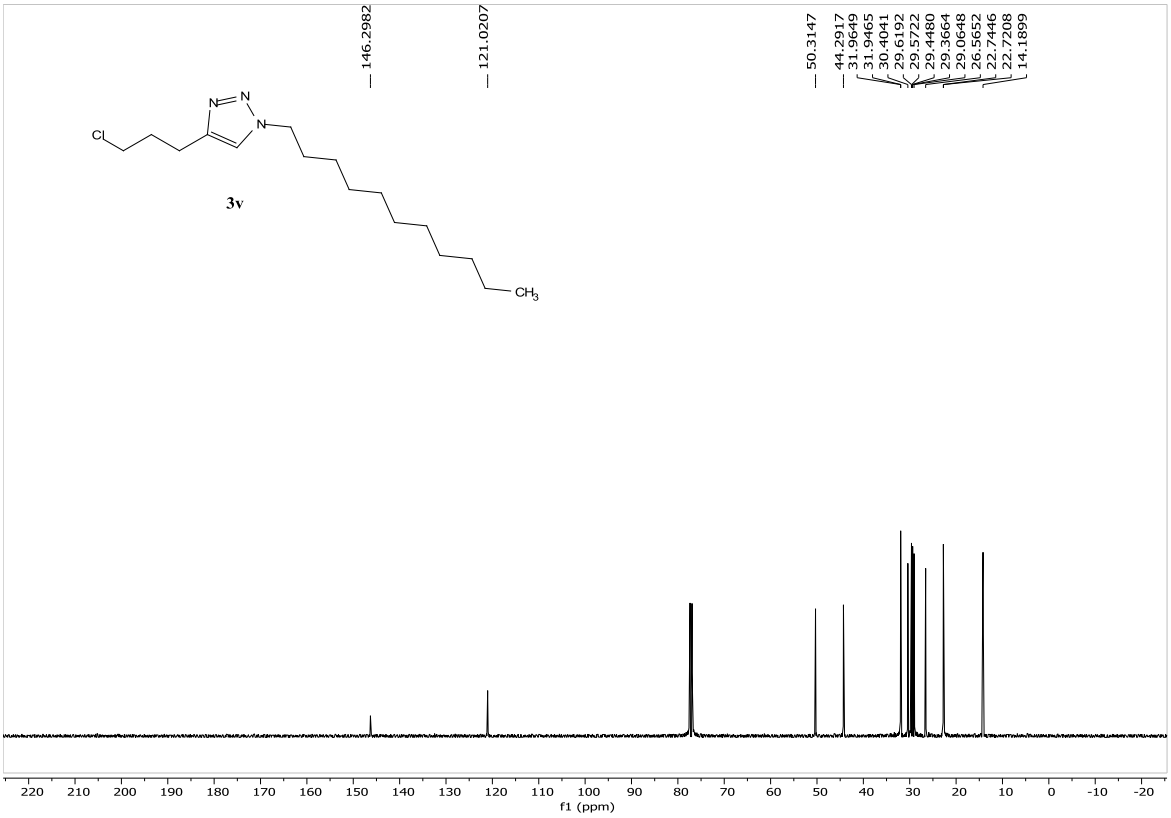
2.1668
2.1561
2.1437
2.1317
2.1211
1.8802
1.8679
1.8560
1.8439
1.8317
1.2911
1.2845
1.2770
1.2652
1.2526
1.2236
0.8495
0.8376
Cl
N
N
N
3v
CH3
1.00
2.09
2.10
2.11
2.16
2.09
16.37
3.20
f1 (ppm)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31.9465
30.4041
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29.5722
29.4480
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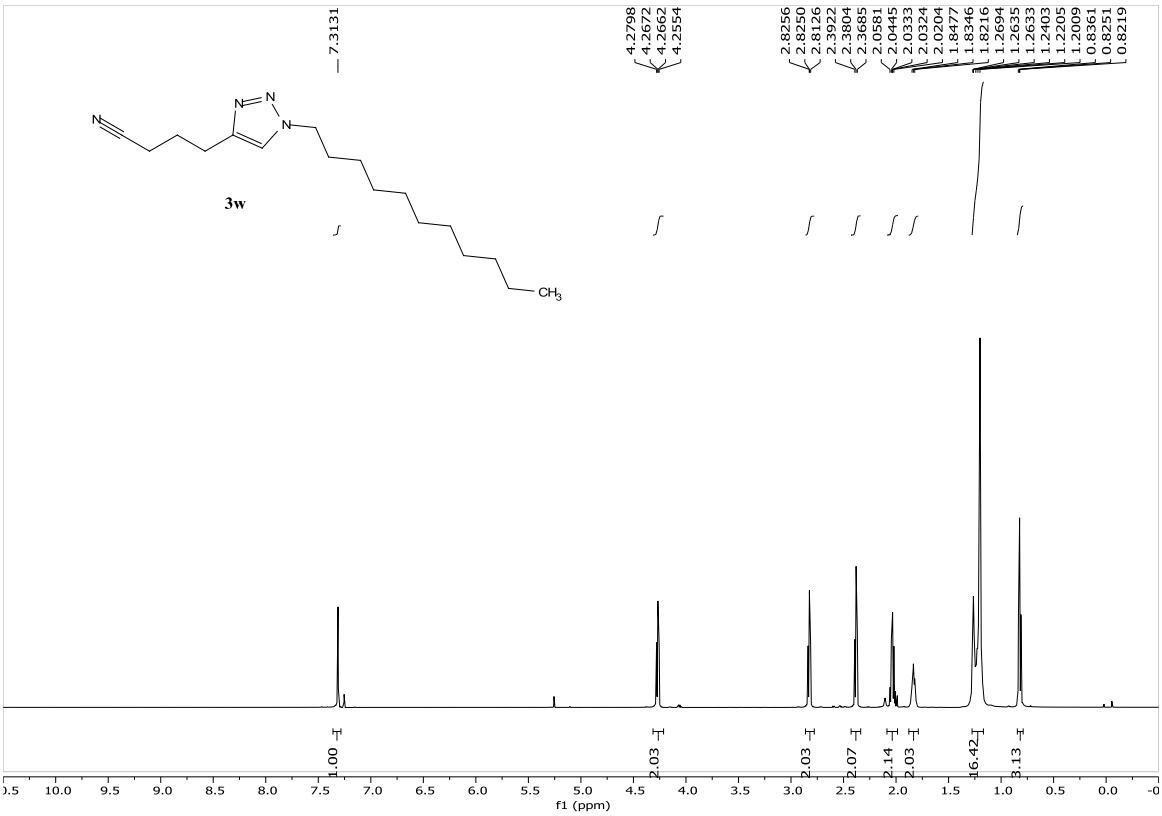
22.7446
22.7208
14.1899
Cl
N
N
N
3v
CH3
f1 (ppm)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2.8250
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2.3922
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2.3685
2.0581
2.0445
2.0333
2.0324
2.0204
1.8477
1.8346
1.8216
1.2694
1.2635
1.2633
1.2403
1.2205
1.2009
0.8361
0.8251
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3w
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2.03
2.07
2.14
2.03
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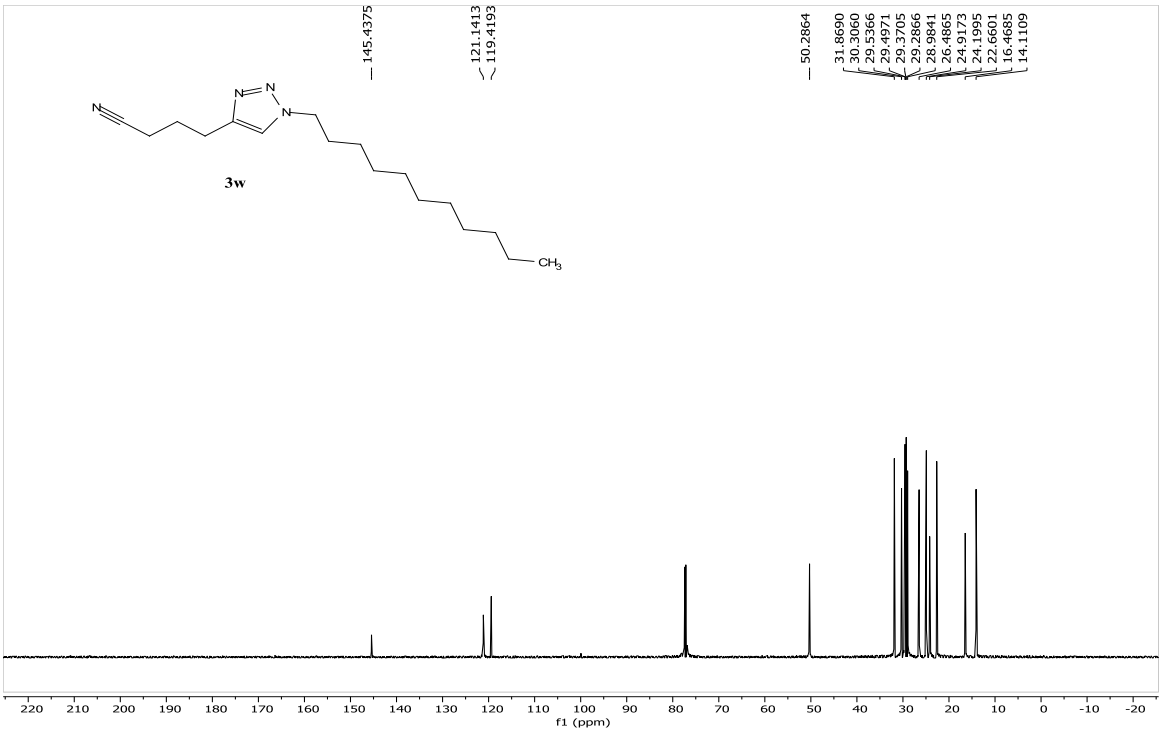
3.13
f1 (ppm)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26.4865
24.9173
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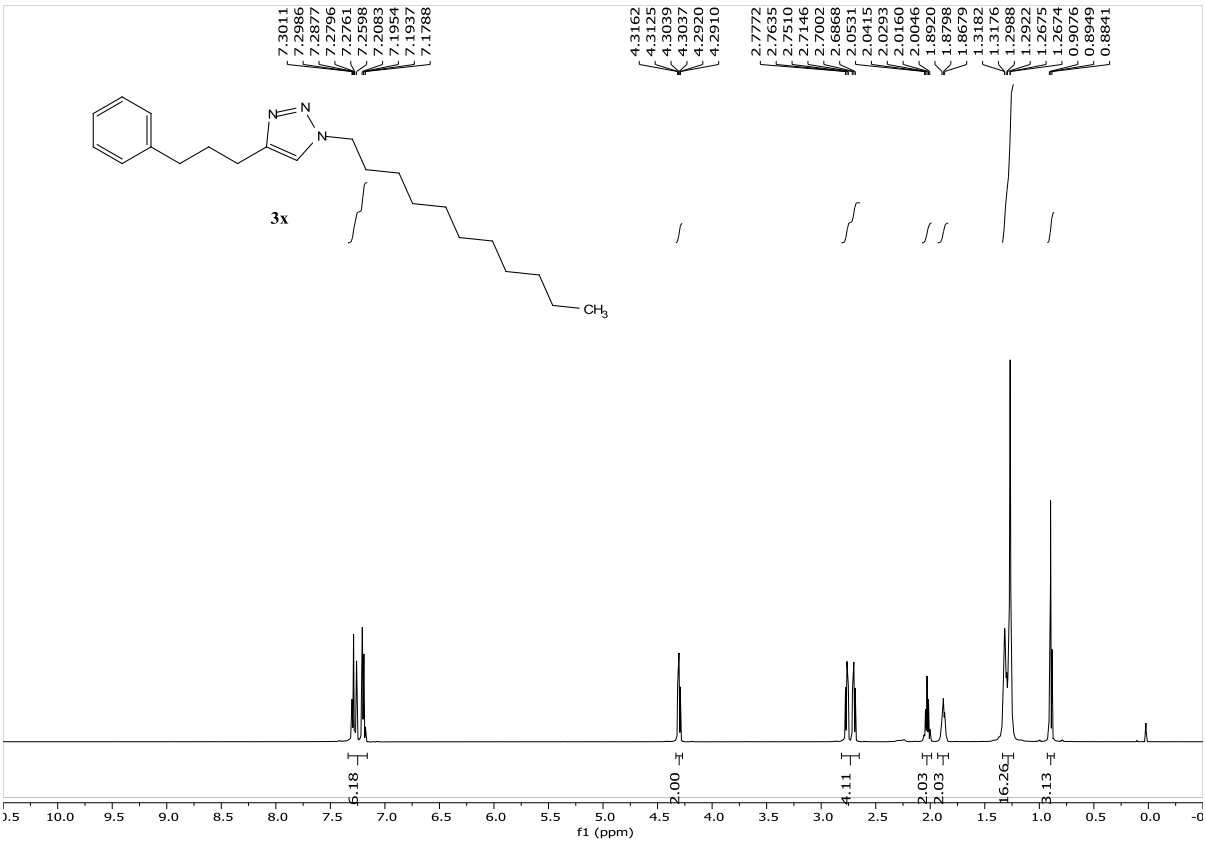
14.1109
3w
CH3
f1 (ppm)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4.3039
4.3037
4.2920
4.2910
2.7772
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2.7510
2.7146
2.7002
2.6868
2.0531
2.0415
2.0293
2.0160
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1.8920
1.8798
1.8679
1.3182
1.3176
1.2988
1.2922
1.2675
1.2674
0.9076
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CH3
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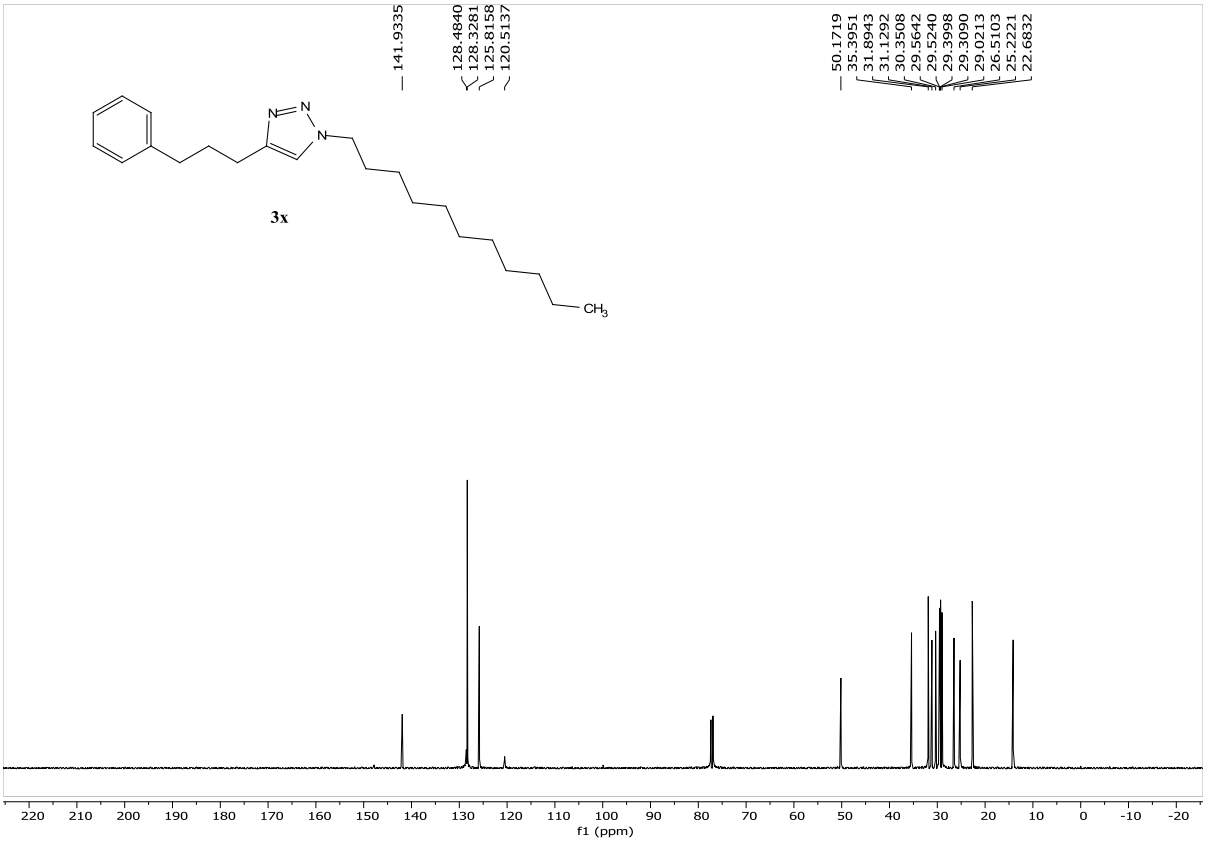
2.03
16.26
3.13
f1 (ppm)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29.3090
29.0213
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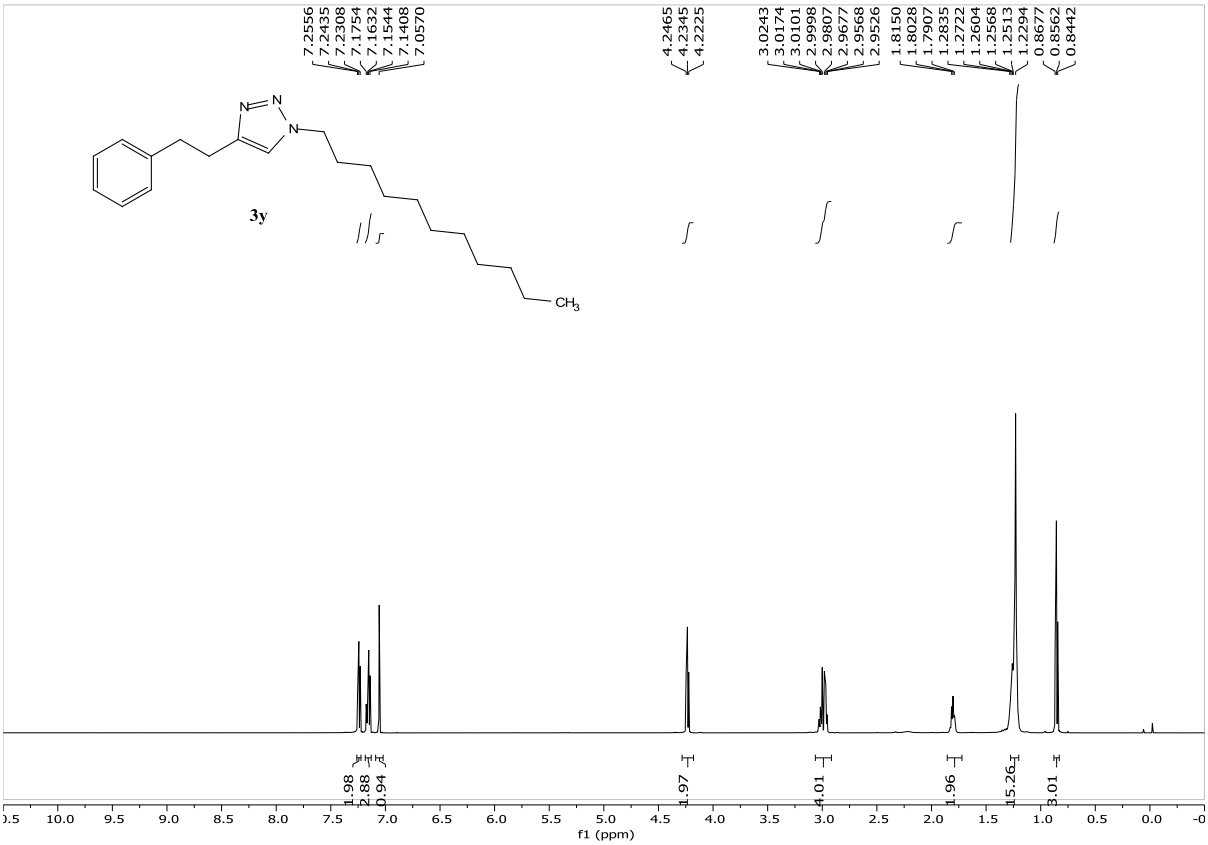
22.6832
3x
CH3
f1 (ppm)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3.0174
3.0101
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1.2835
1.2722
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1.2568
1.2513
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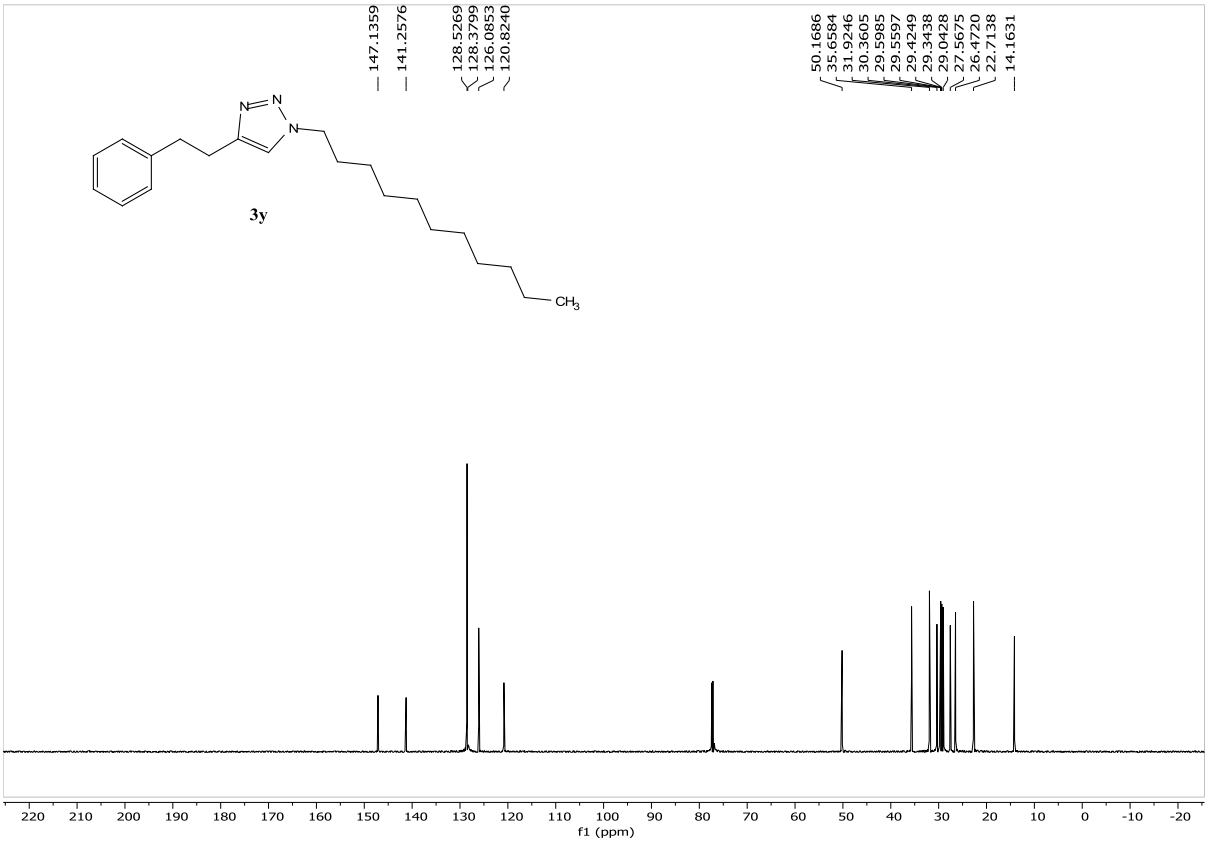
f1 (ppm)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29.0428
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26.4720
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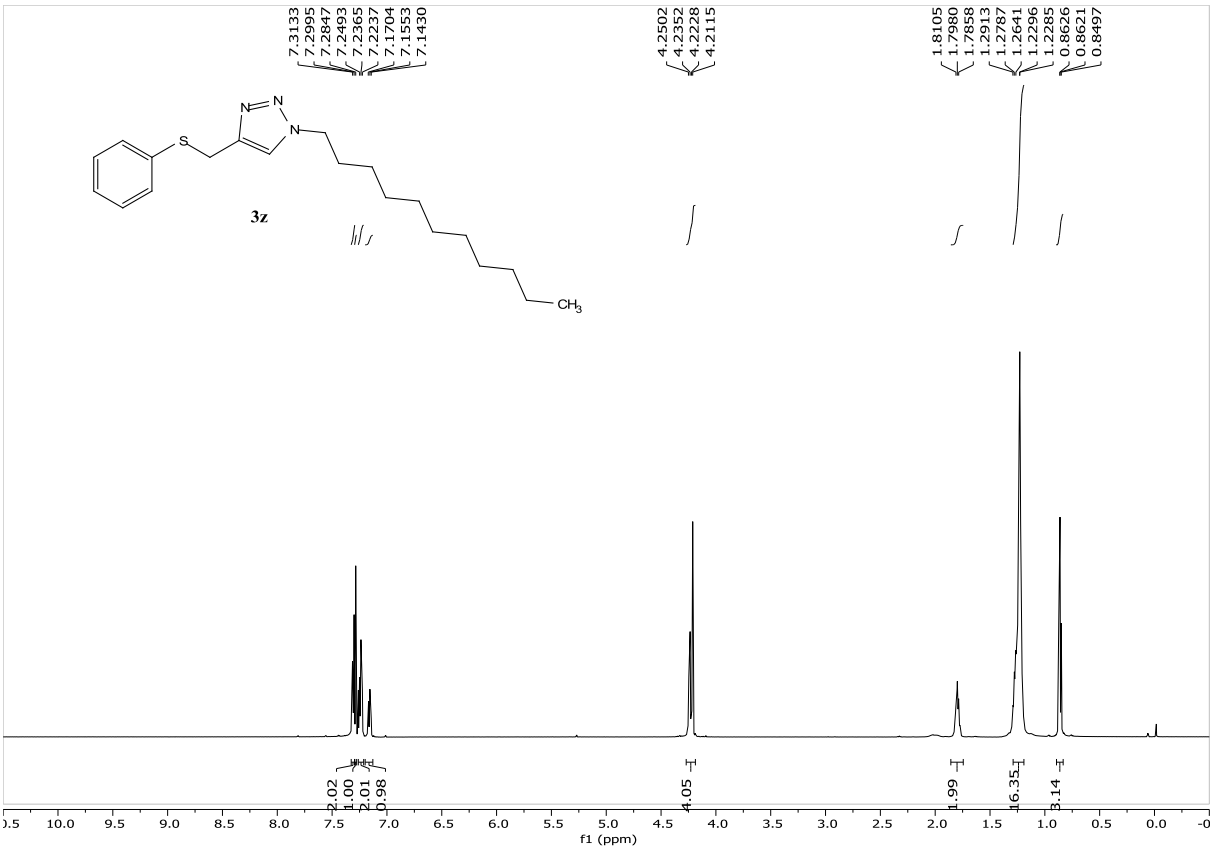
3y
CH3
f1 (ppm)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7.1430
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4.2228
4.2115
1.8105
1.7980
1.7858
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1.2787
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1.2285
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0.8621
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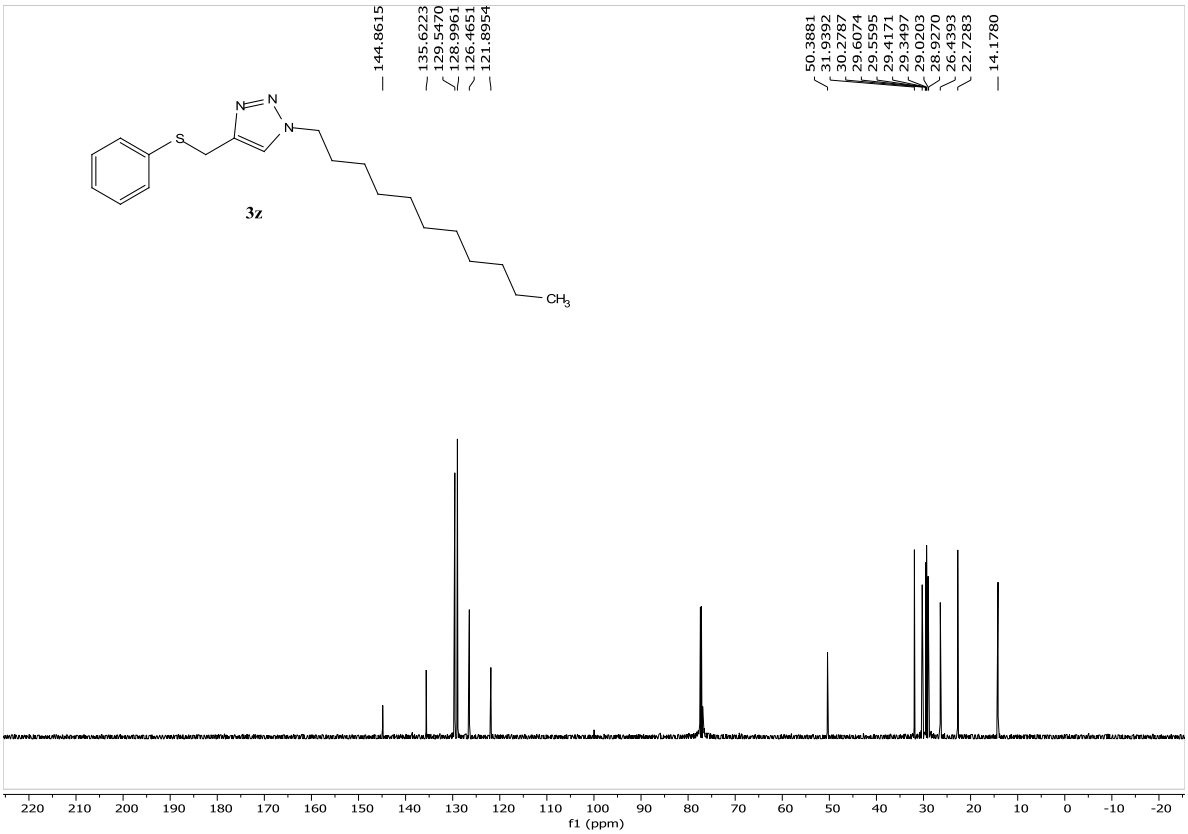
3.14
f1 (ppm)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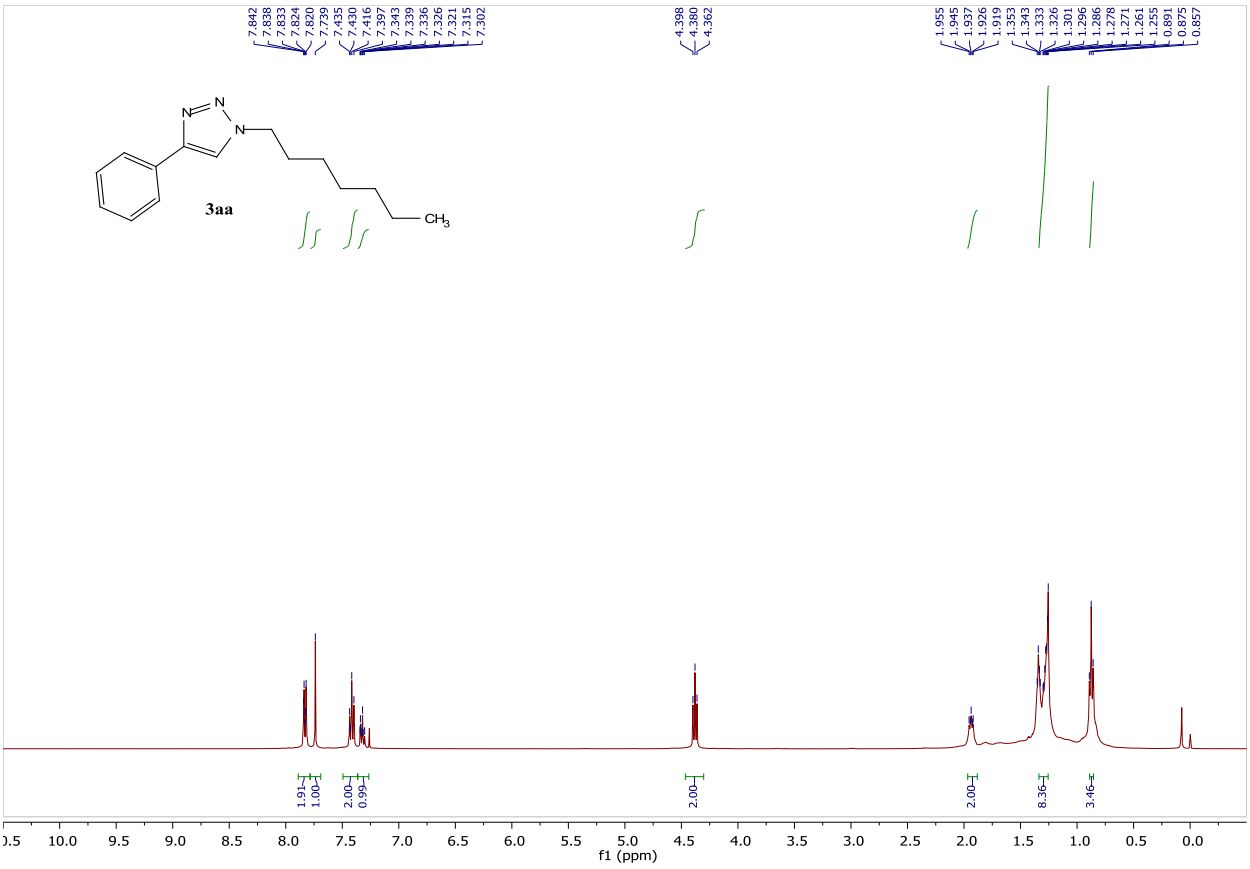
f1 (ppm)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7.321
7.315
7.302
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4.362
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1.945
1.937
1.926
1.919
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1.343
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1.271
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0.875
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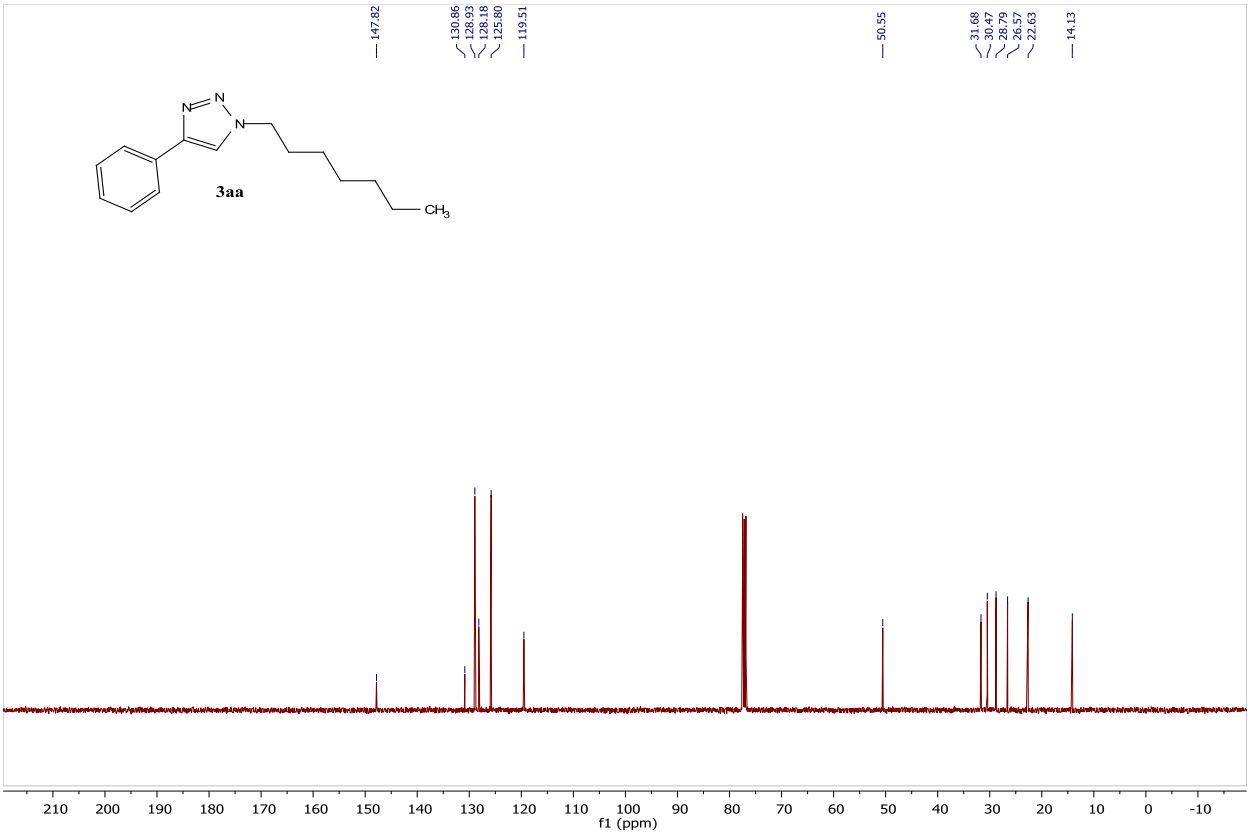
f1 (ppm)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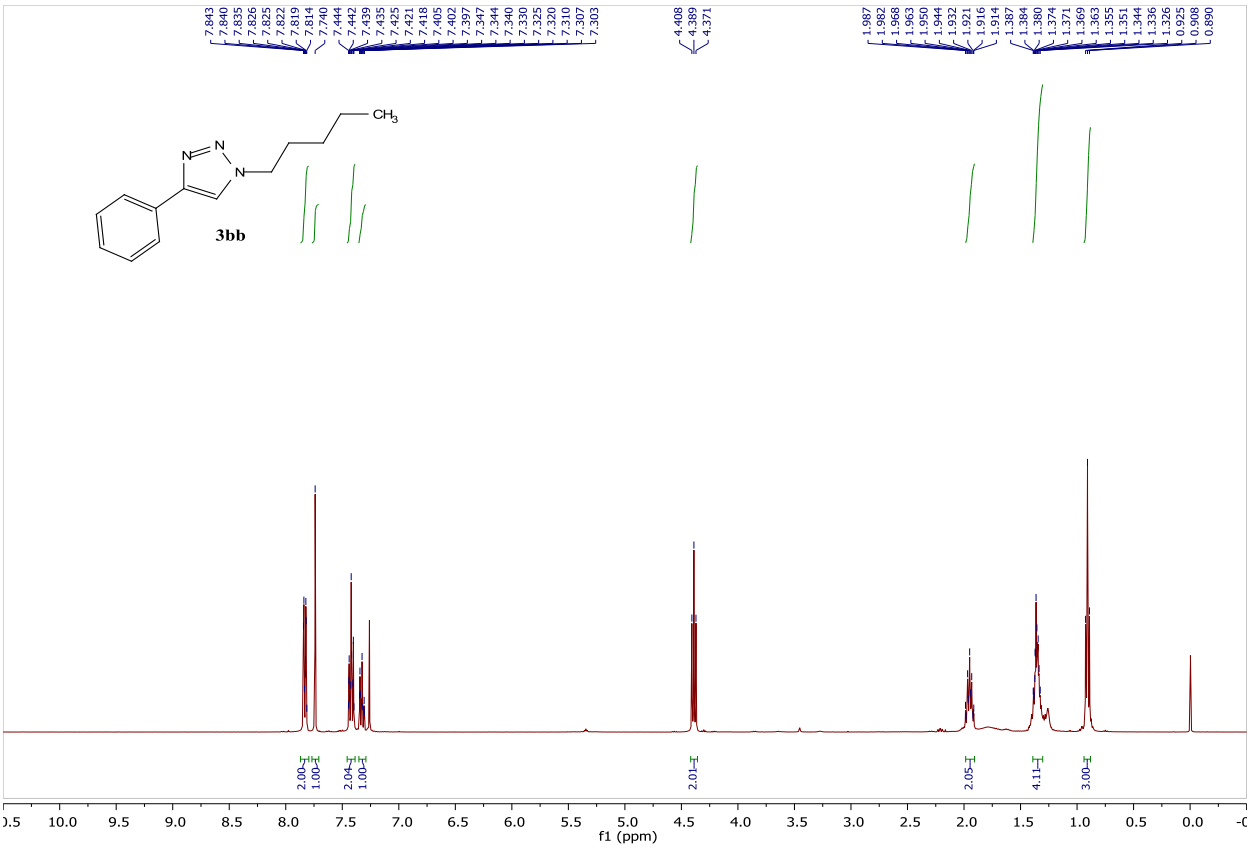
CH3
f1 (ppm)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1.914
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1.355
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1.336
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CH3
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3.00
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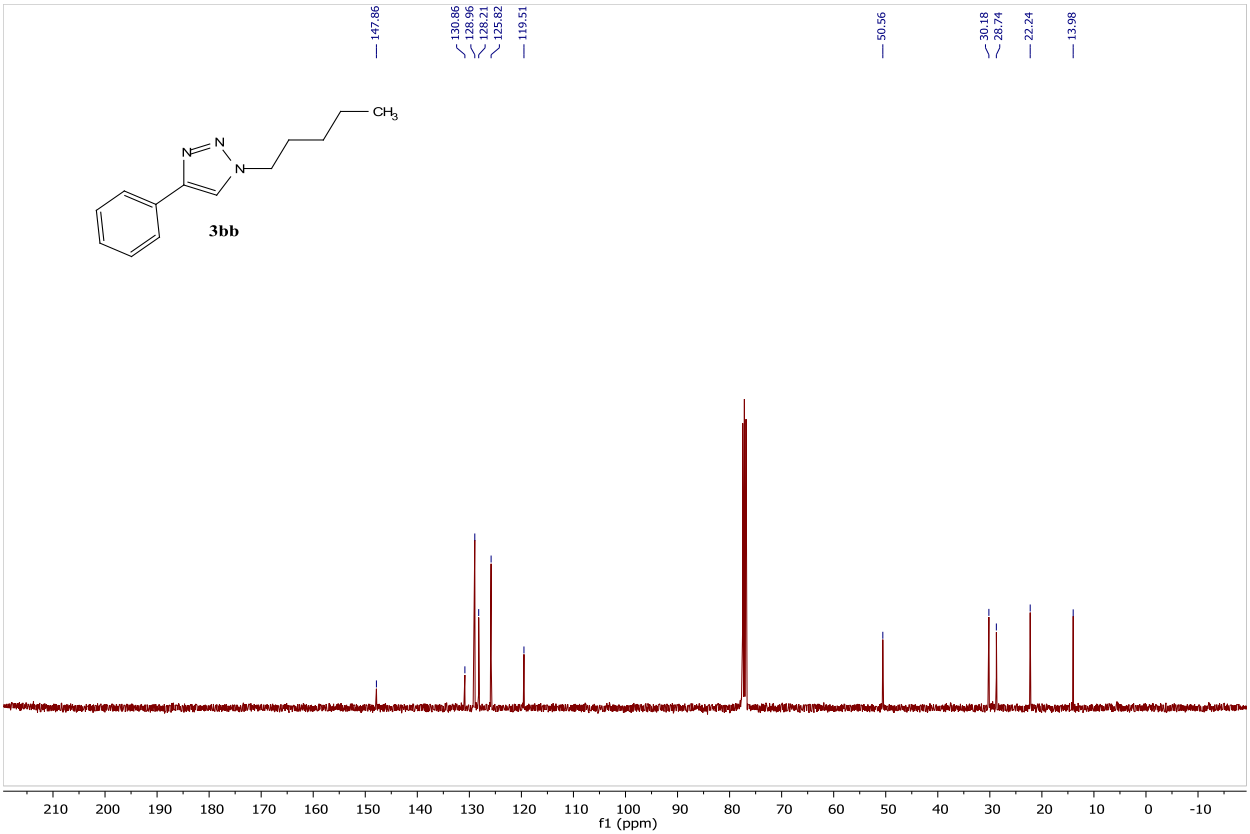
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CH3
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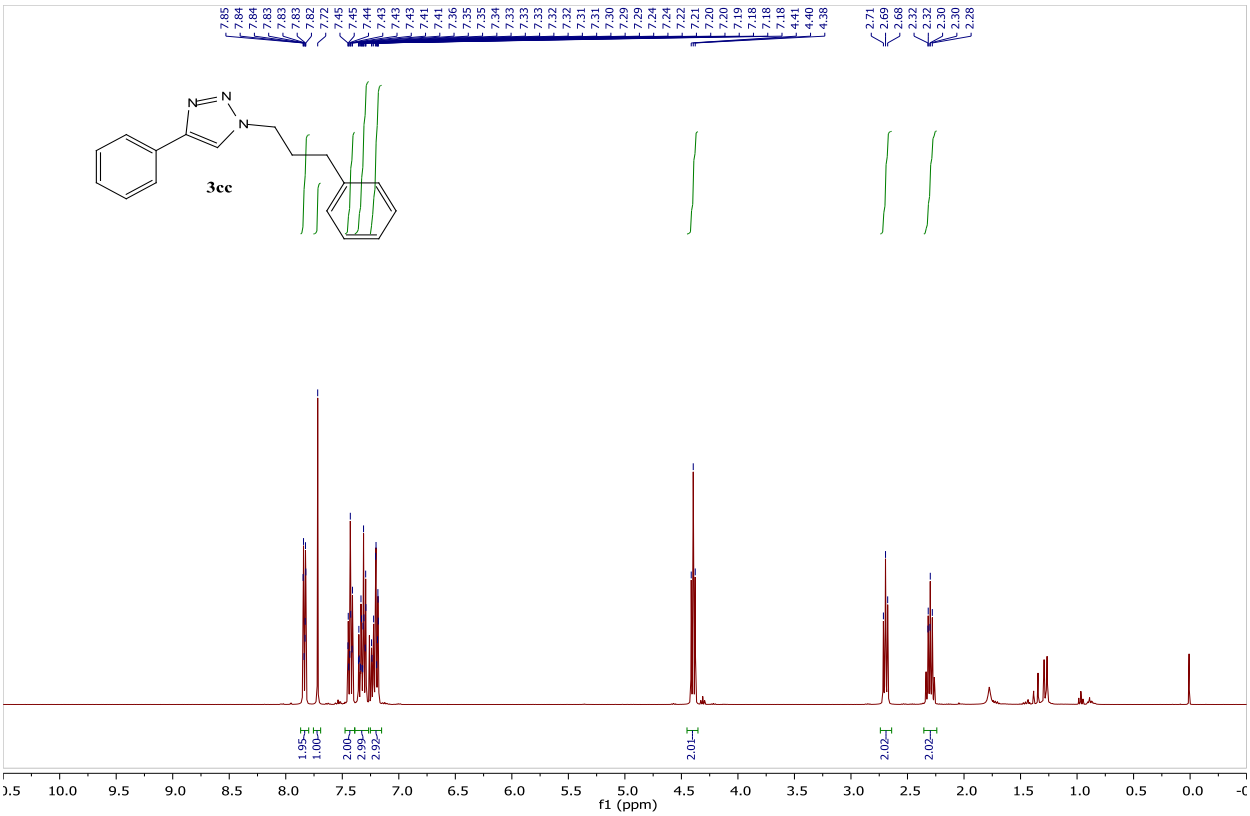
N
3bb
f1 (ppm)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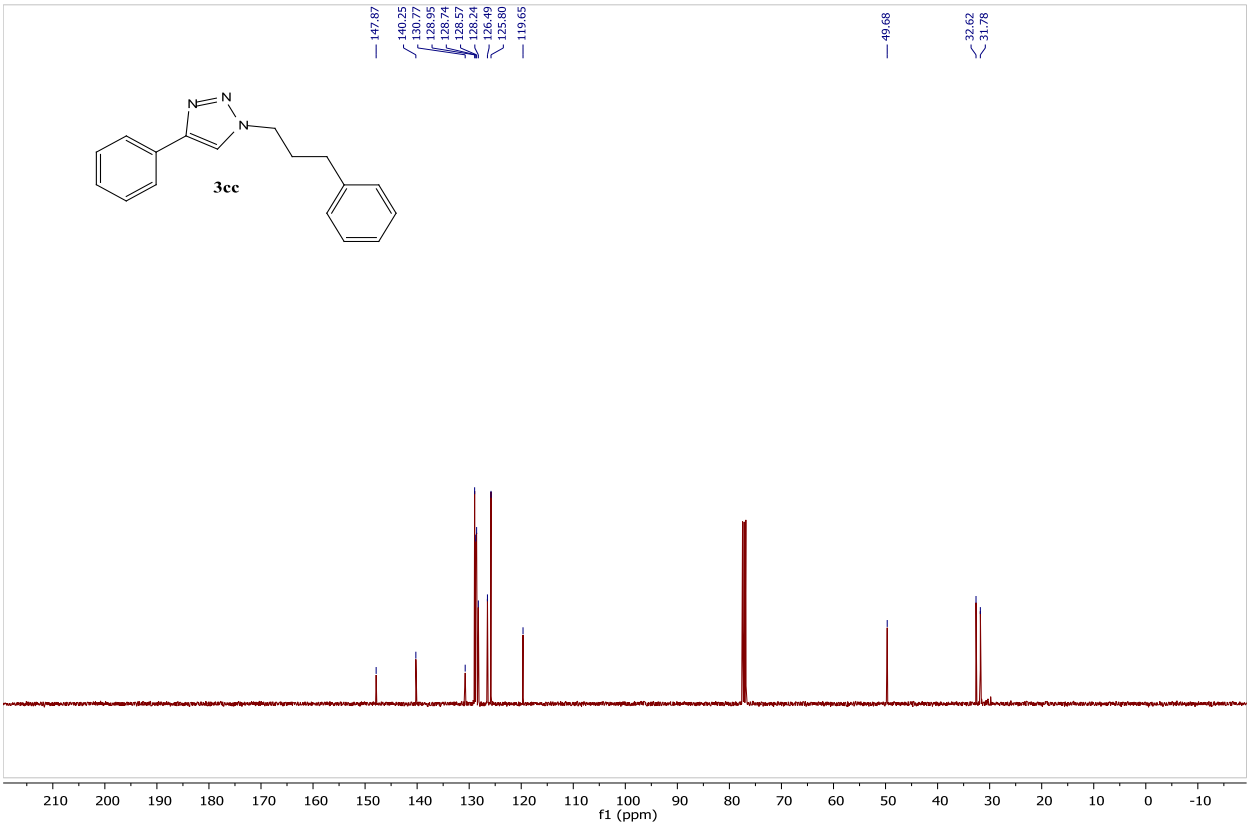
2.02
f1 (ppm)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49.68
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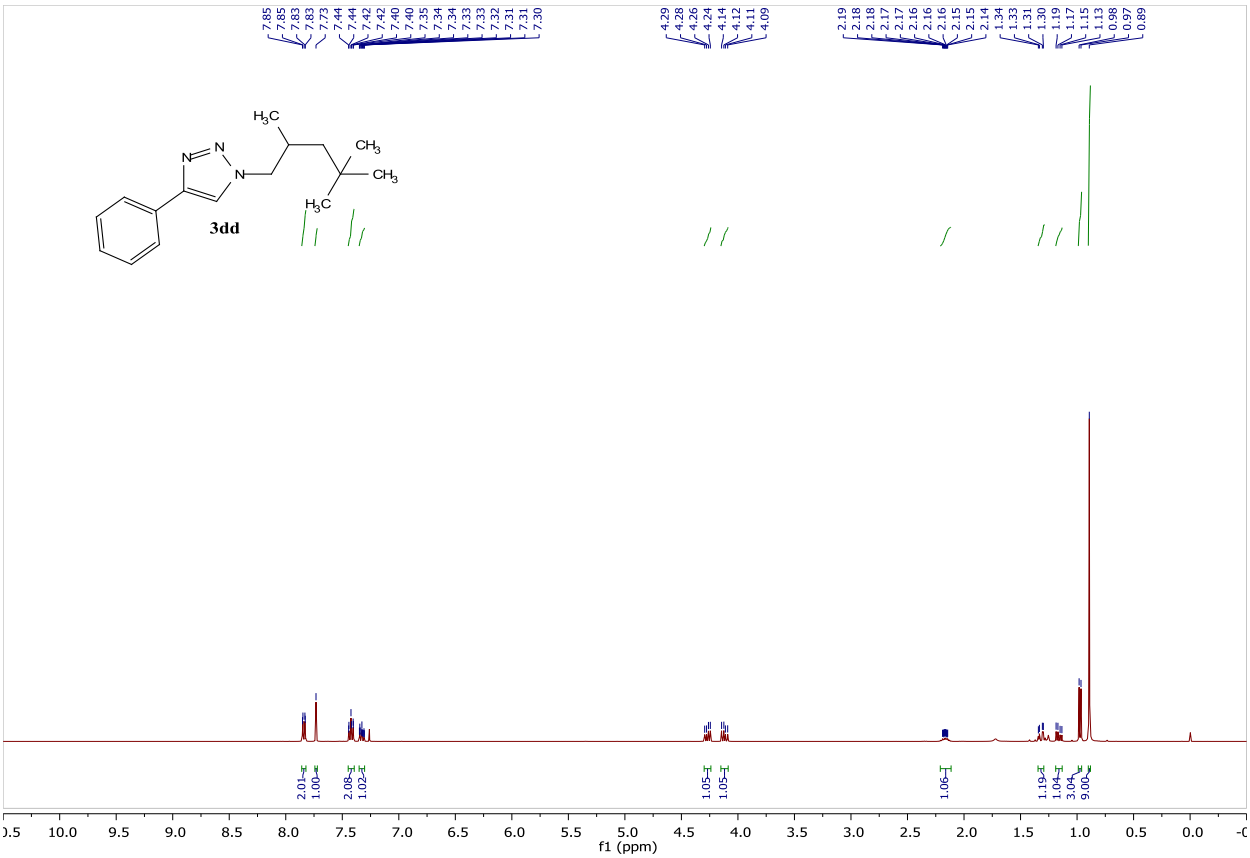
3cc
f1 (ppm)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7.31
7.31
7.30
4.29
4.28
4.26
4.24
4.14
4.12
4.11
4.09
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2.18
2.18
2.17
2.17
2.16
2.16
2.16
2.15
2.15
2.14
1.34
1.33
1.31
1.30
1.19
1.17
1.15
1.13
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0.97
0.89
H3C
CH3
CH3
H3C
N
N
N
3dd
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1.05
1.05
1.06
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1.04
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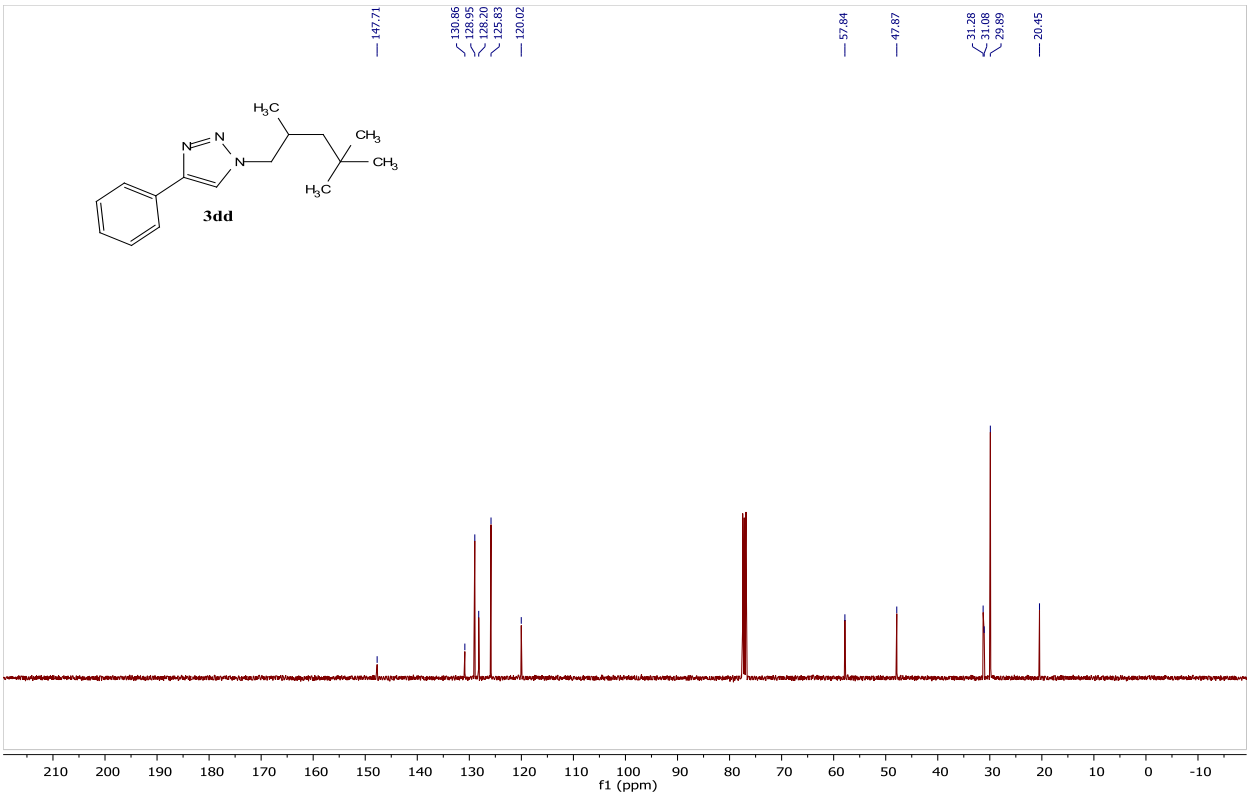
f1 (ppm)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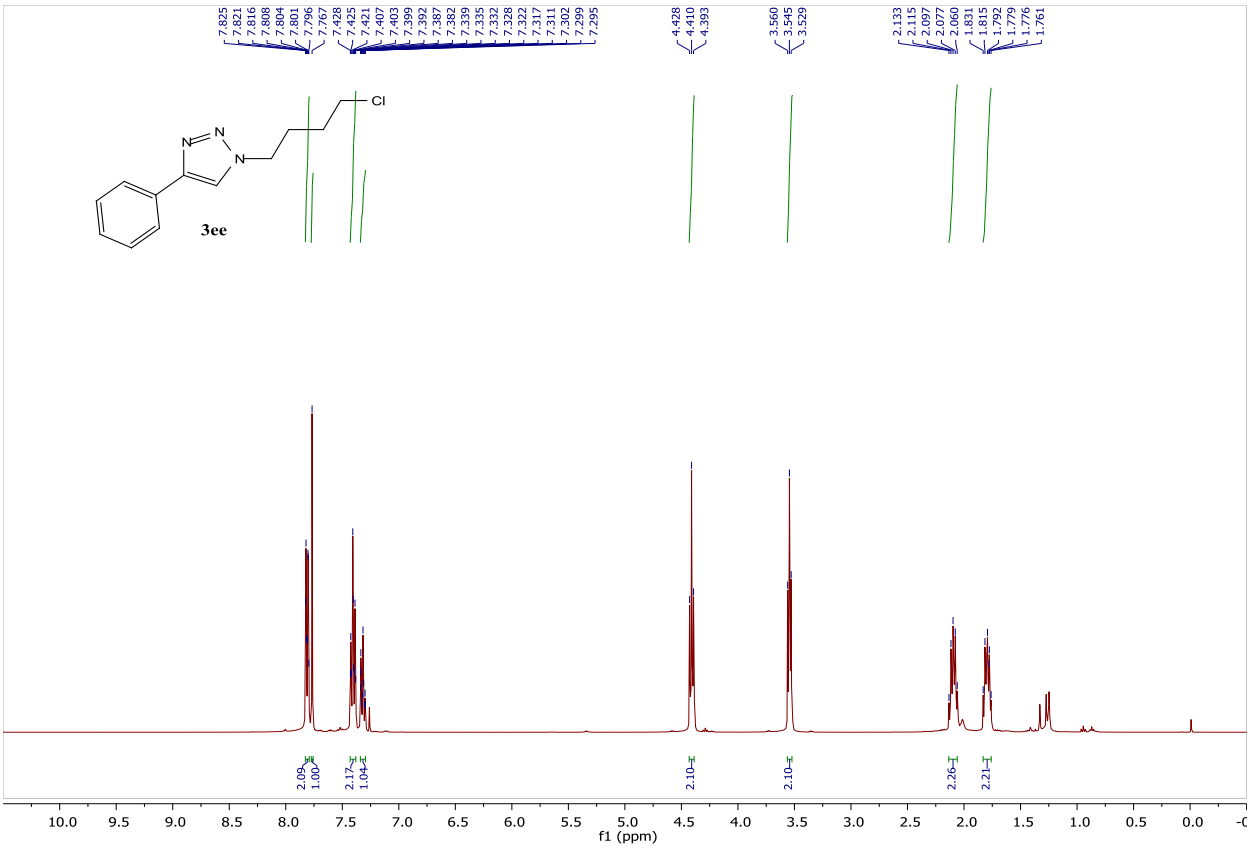
29.89
20.45
H3C
CH3
CH3
H3C
N
N
N
3dd
f1 (ppm)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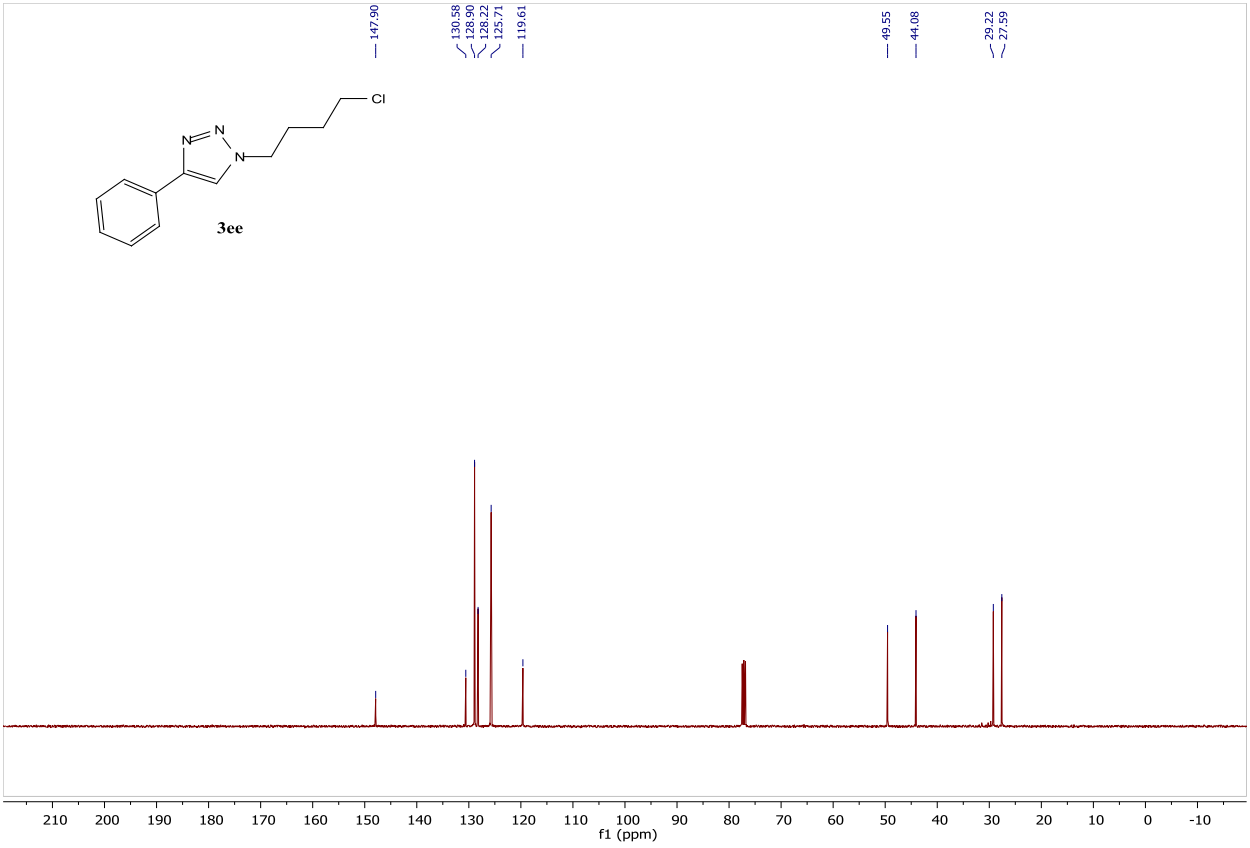
7.332
7.328
7.322
7.317
7.311
7.302
7.299
7.295
4.428
4.410
4.393
3.560
3.545
3.529
2.133
2.115
2.097
2.077
2.060
1.831
1.815
1.792
1.779
1.776
1.761
Cl
N
N
N
3ee
2.09
1.00
2.17
1.04
2.10
2.10
2.26
2.21
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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130.58
128.90
128.22
125.71
119.61
49.55
44.08
29.22
27.59
Cl
N
N
N
3ee
210
200
190
180
170
160
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140
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120
110
100
90
80
70
60
50
40
30
20
10
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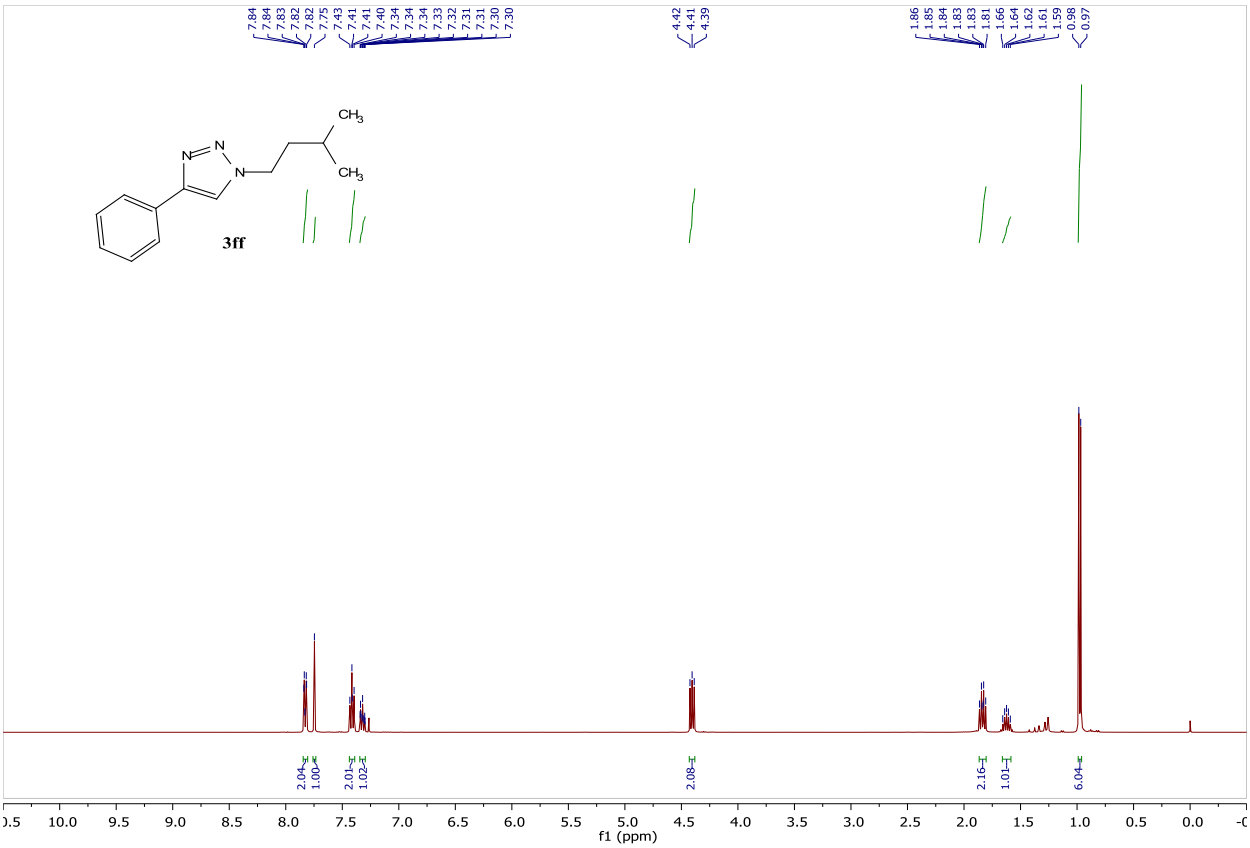
-10
f1 (ppm)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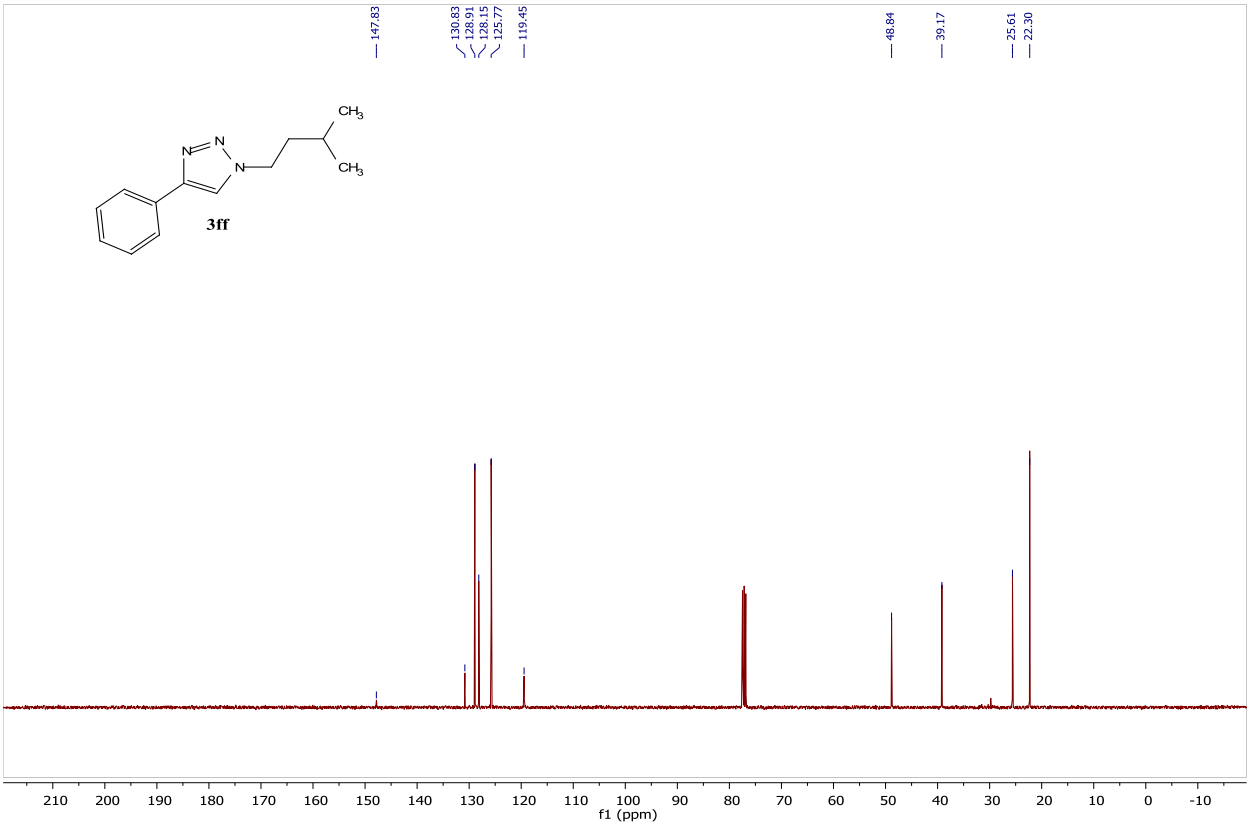
7.84
7.83
7.82
7.82
7.75
7.43
7.41
7.41
7.40
7.34
7.34
7.34
7.33
7.32
7.31
7.31
7.30
7.30
4.42
4.41
4.39
1.86
1.85
1.84
1.83
1.83
1.81
1.66
1.64
1.62
1.61
1.59
0.98
0.97
CH3
CH3
N
N
N
3ff
2.04
1.00
2.01
1.02
2.08
2.16
1.01
6.04
f1 (ppm)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128.91
128.15
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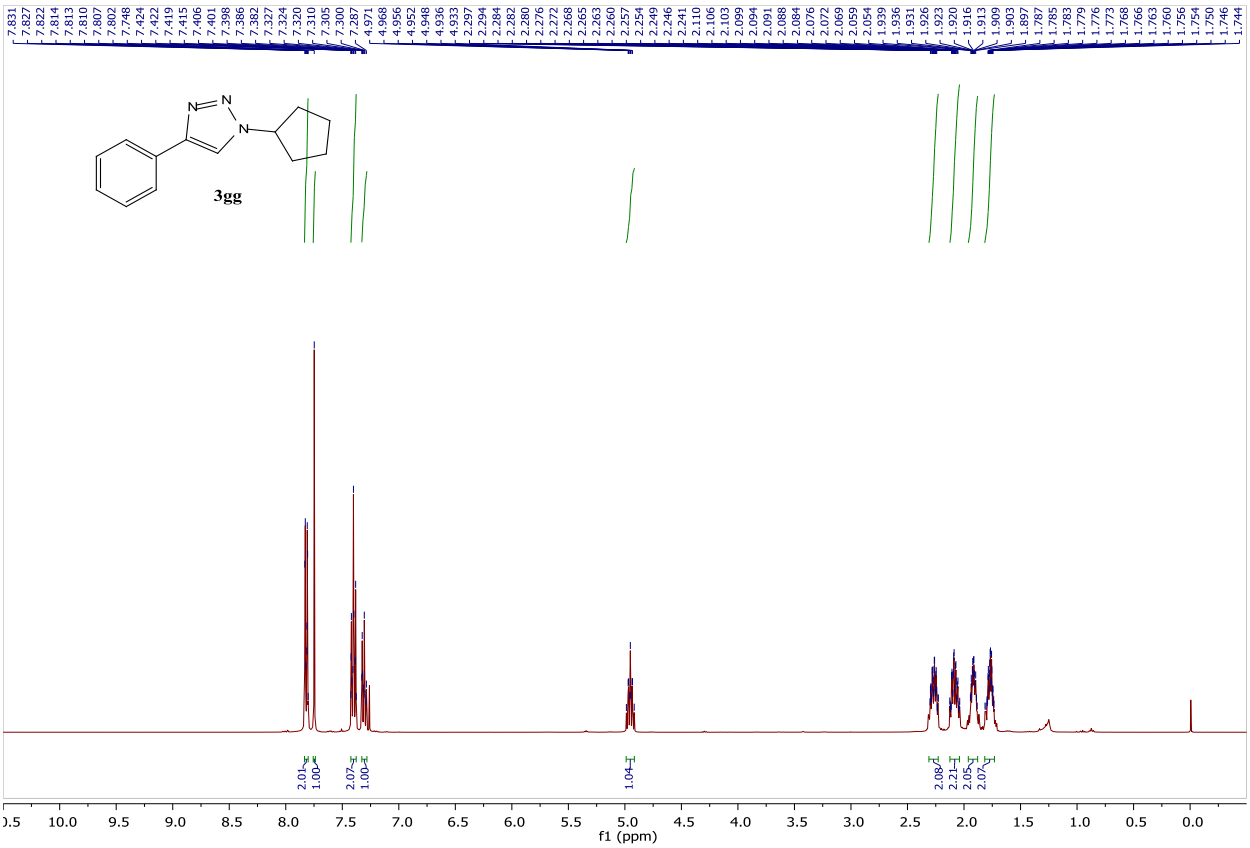
119.45
48.84
39.17
25.61
22.30
CH3
CH3
N
N
N
3ff
f1 (ppm)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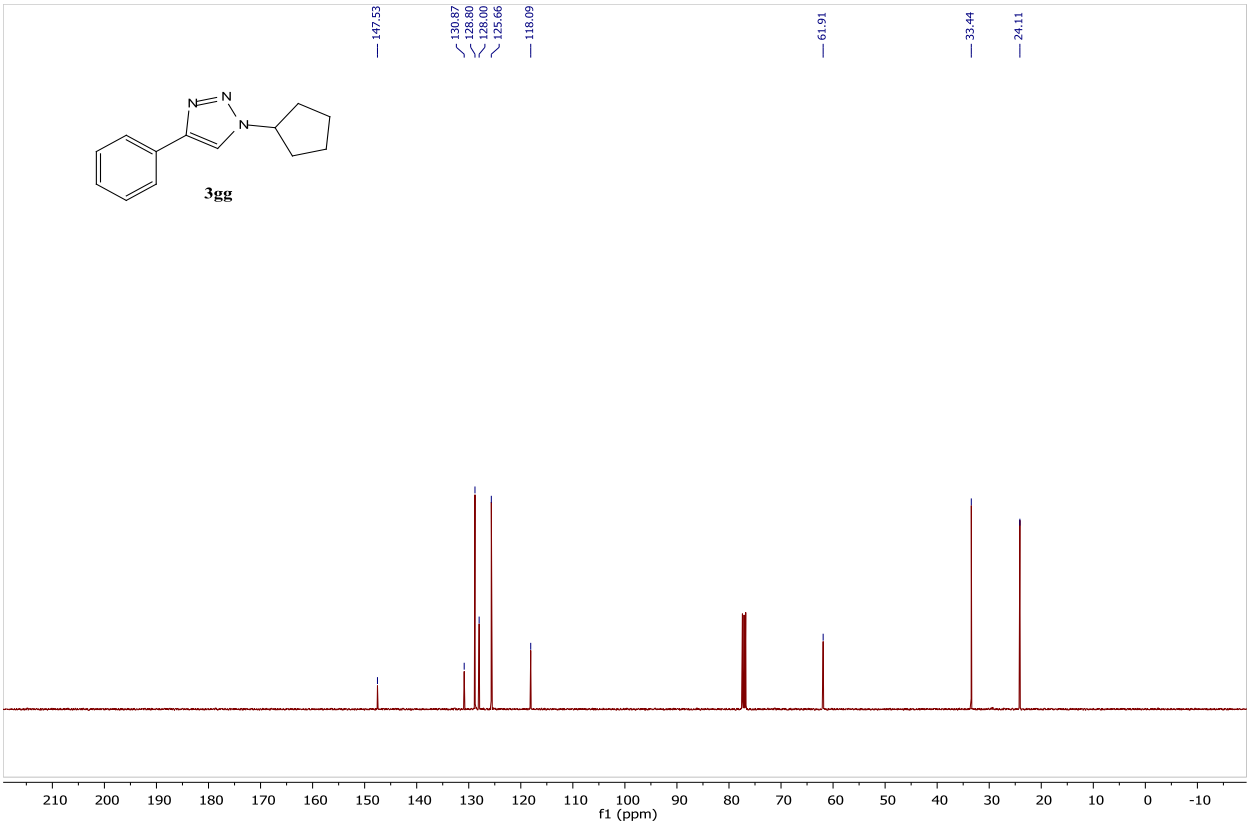
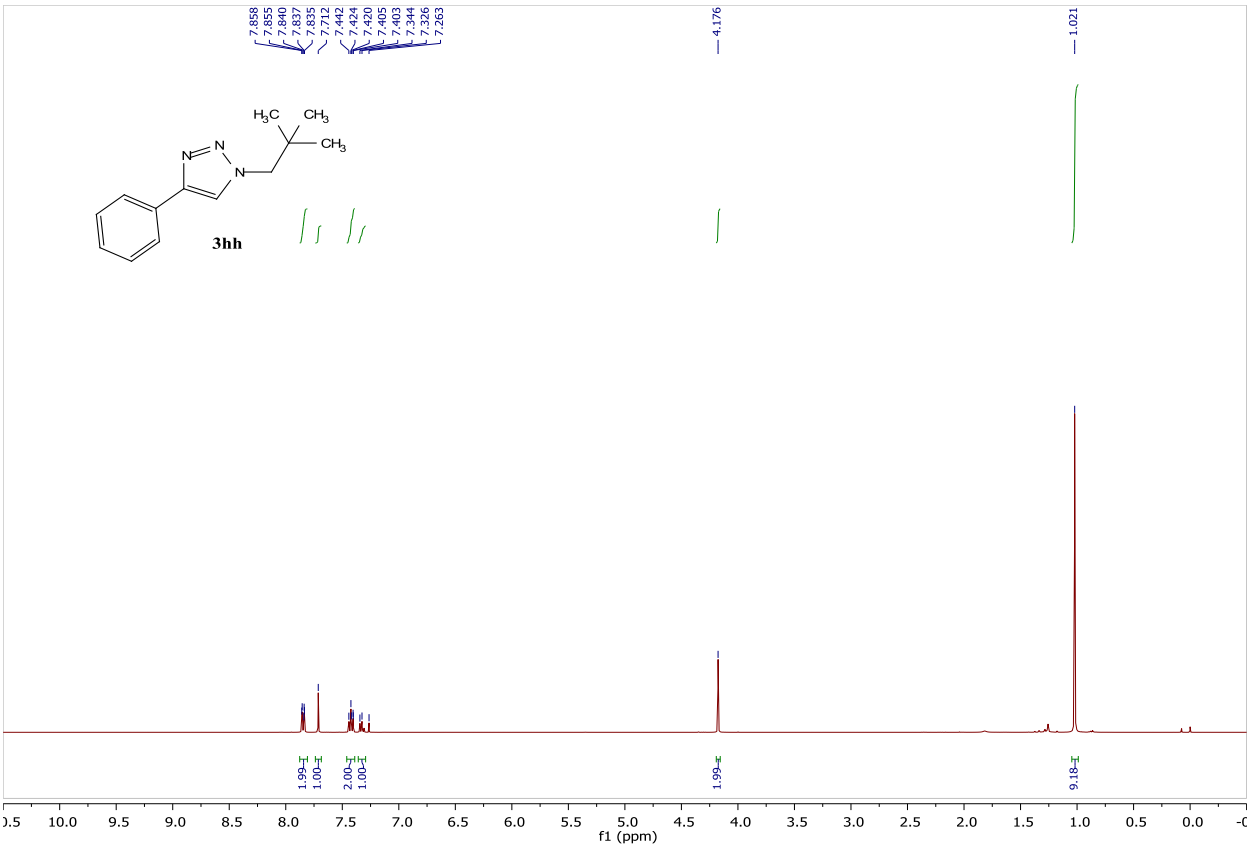
f1 (ppm)


3gg
f1 (ppm)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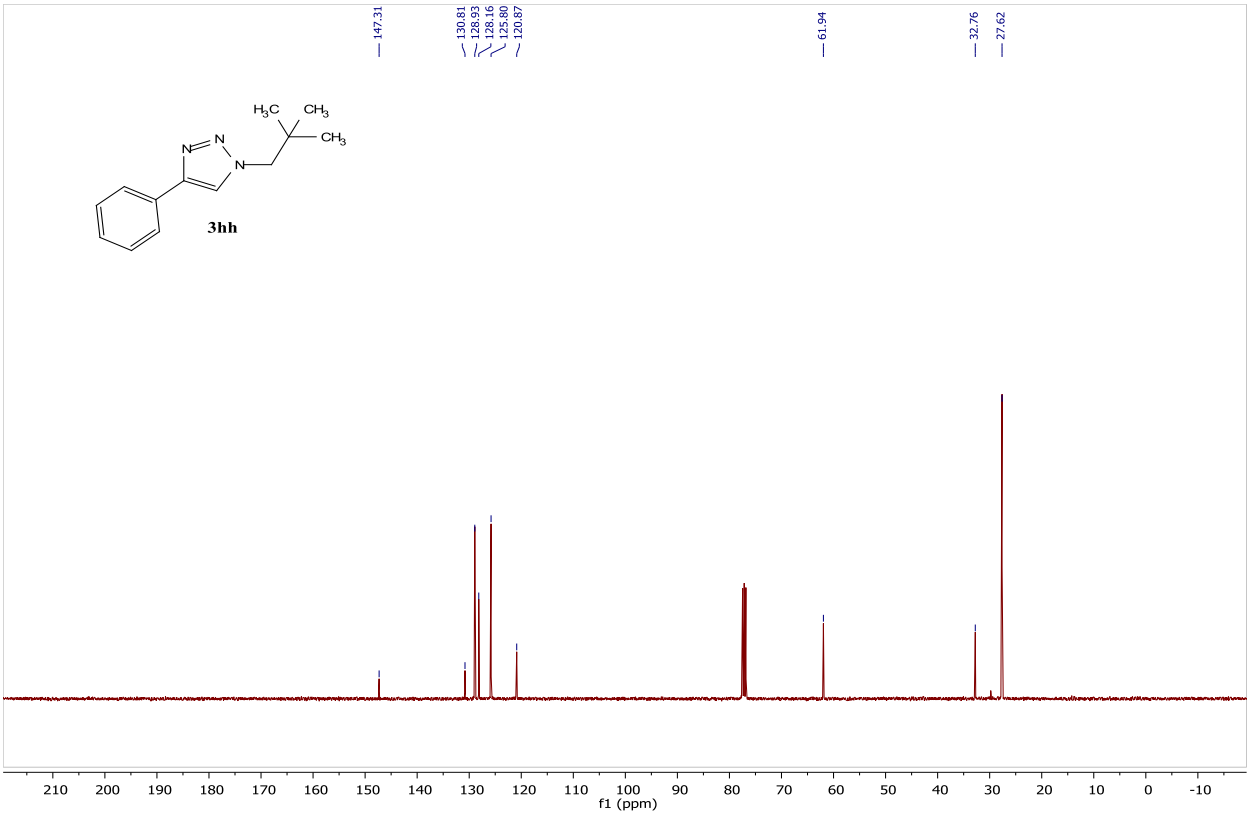
7.835
7.712
7.442
7.424
7.420
7.405
7.403
7.344
7.326
7.263
4.176
1.021
H3C
CH3
CH3
N
N
N
3hh
1.99
1.00
2.00
1.00
1.99
9.18
f1 (ppm)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130.81
128.93
128.16
125.80
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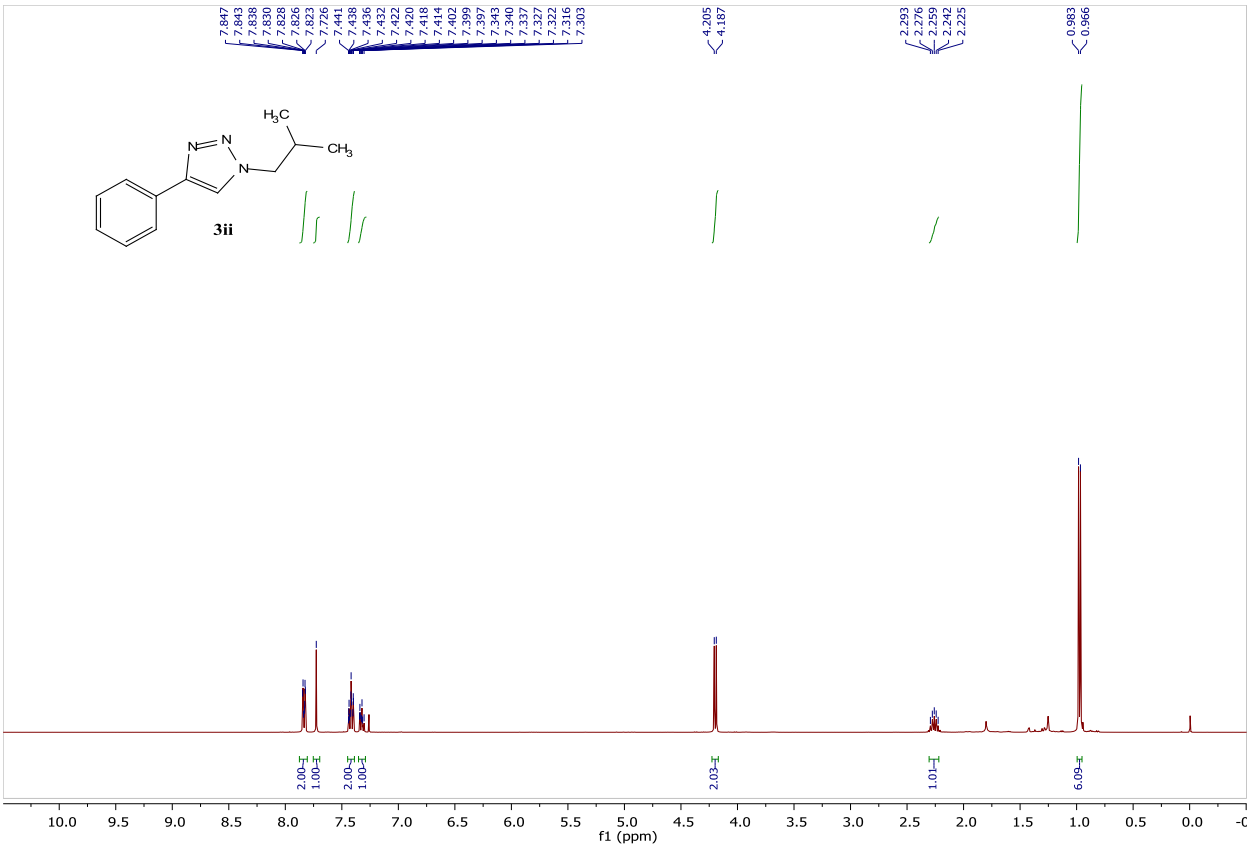
61.94
32.76
27.62
H3C
CH3
CH3
N
N
N
3hh
f1 (ppm)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7.826
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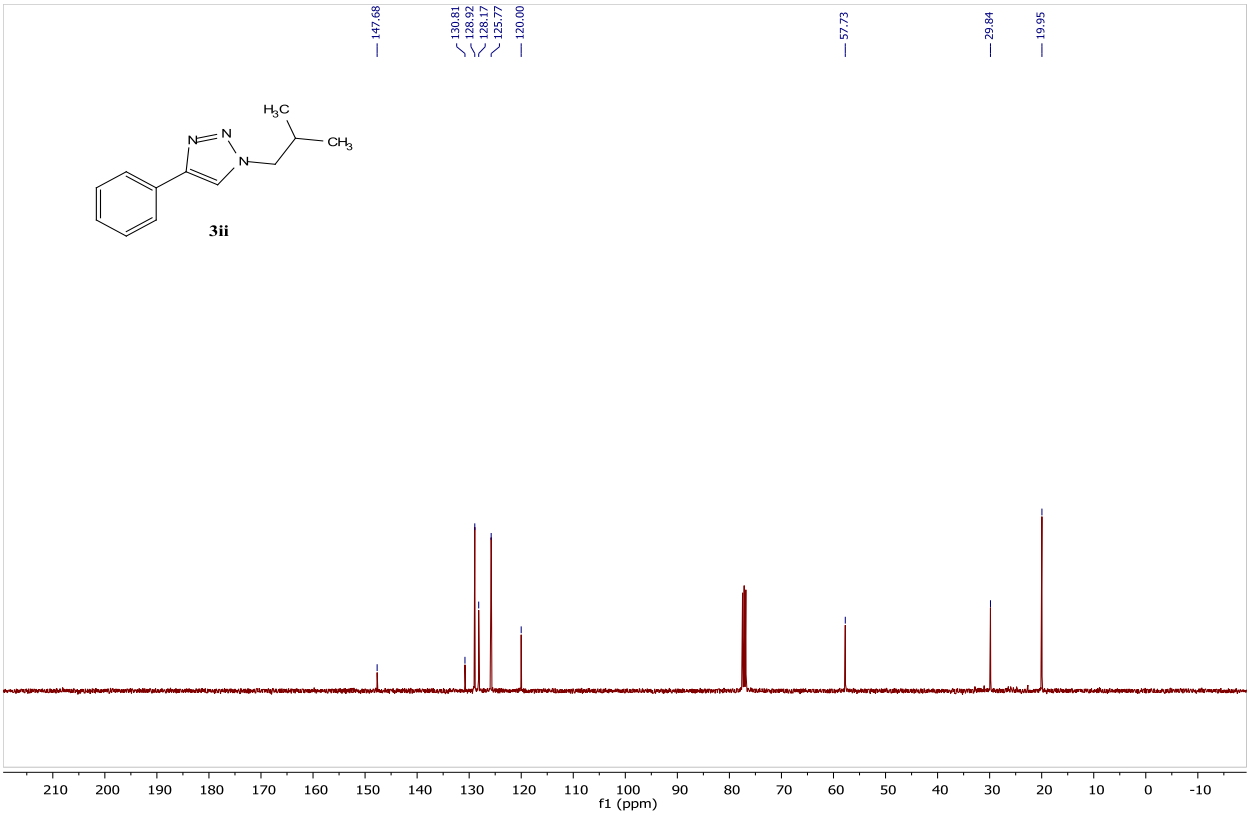
7.726
7.441
7.438
7.436
7.432
7.422
7.420
7.418
7.414
7.402
7.399
7.397
7.343
7.340
7.337
7.327
7.322
7.316
7.303
4.205
4.187
2.293
2.276
2.259
2.242
2.225
0.983
0.966
H3C
CH3
N
N
N
3ii
2.00
1.00
2.00
1.00
2.03
1.01
6.09
f1 (ppm)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128.92
128.17
125.77
120.00
57.73
29.84
19.95
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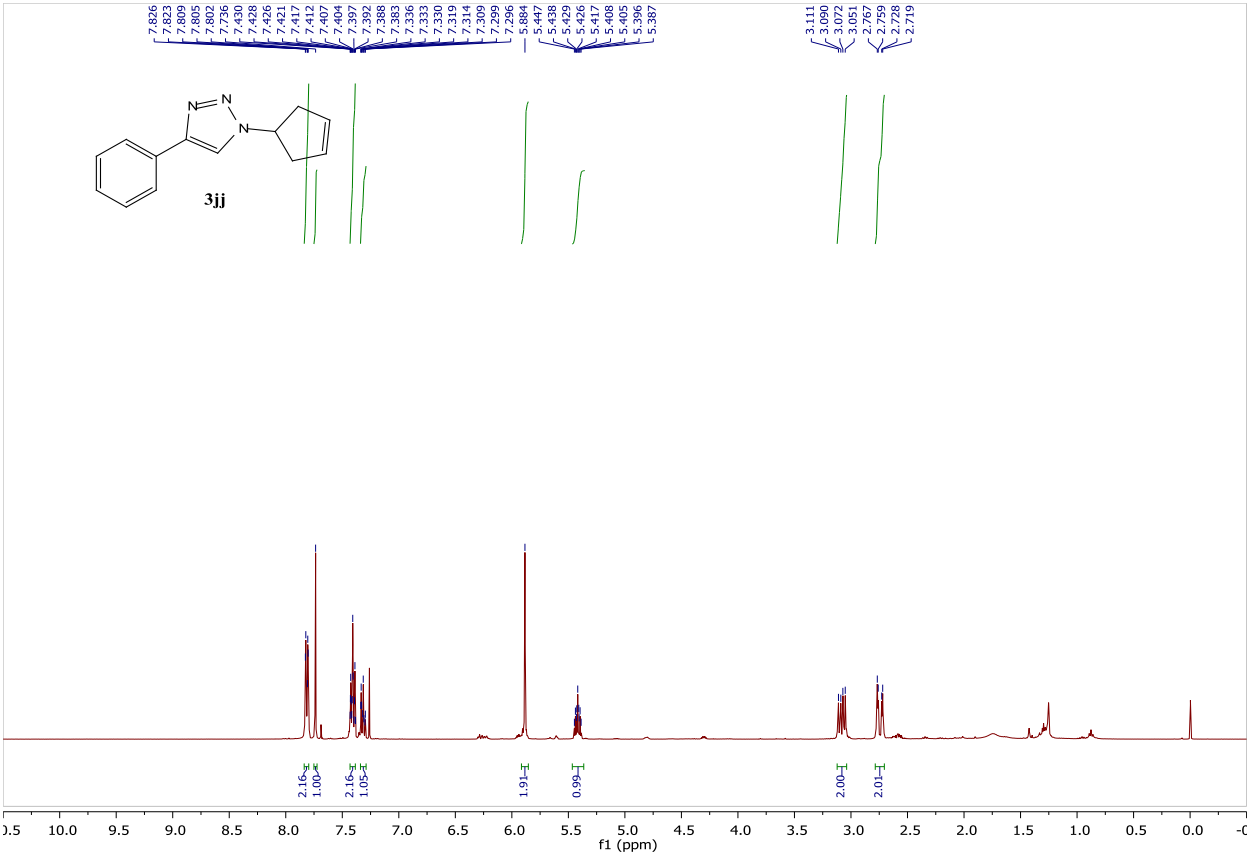
CH3
N
N
N
3ii
f1 (ppm)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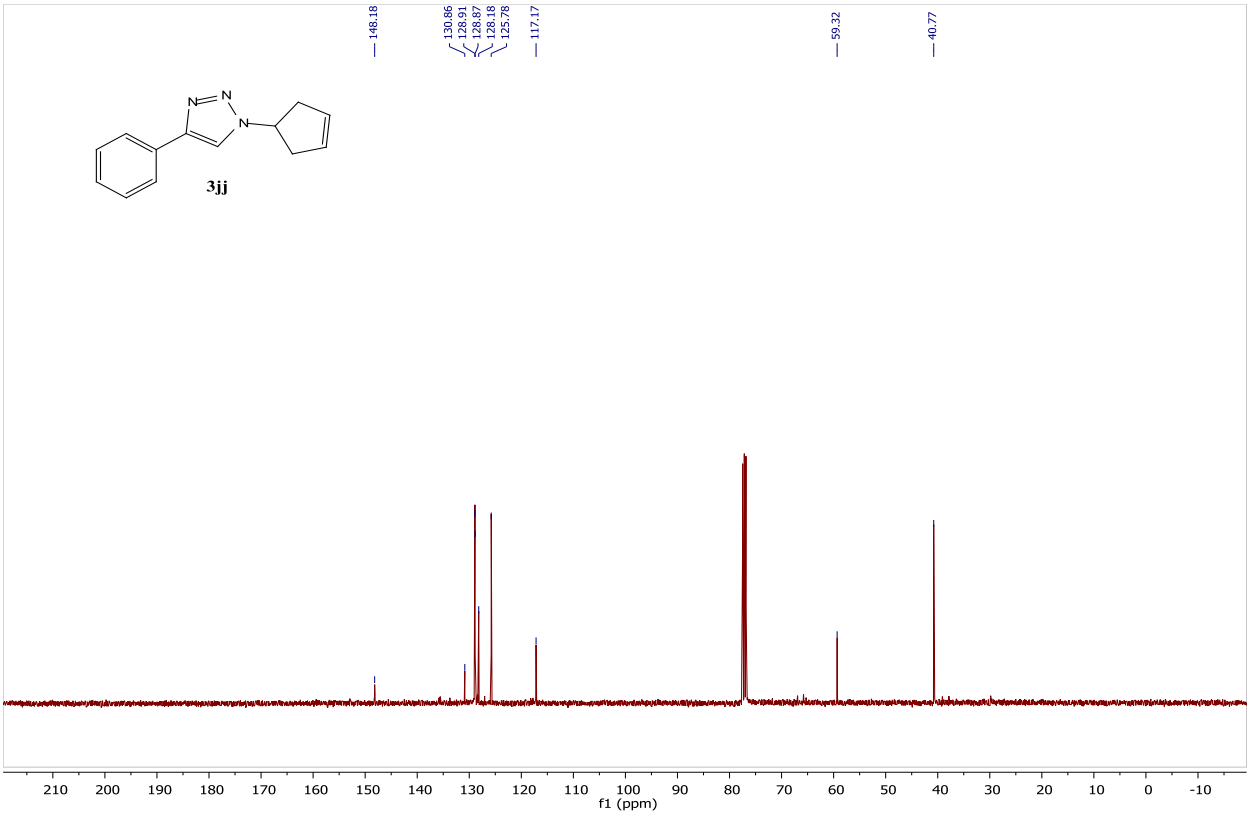
7.823
7.809
7.805
7.802
7.736
7.430
7.428
7.426
7.421
7.417
7.412
7.407
7.404
7.397
7.392
7.388
7.383
7.336
7.333
7.330
7.319
7.314
7.309
7.299
7.296
5.884
5.447
5.438
5.429
5.426
5.417
5.408
5.405
5.396
5.387
3.111
3.090
3.072
3.051
2.767
2.759
2.728
2.719
3jj
2.16
1.00
2.16
1.05
1.91
0.99
2.00
2.01
f1 (ppm)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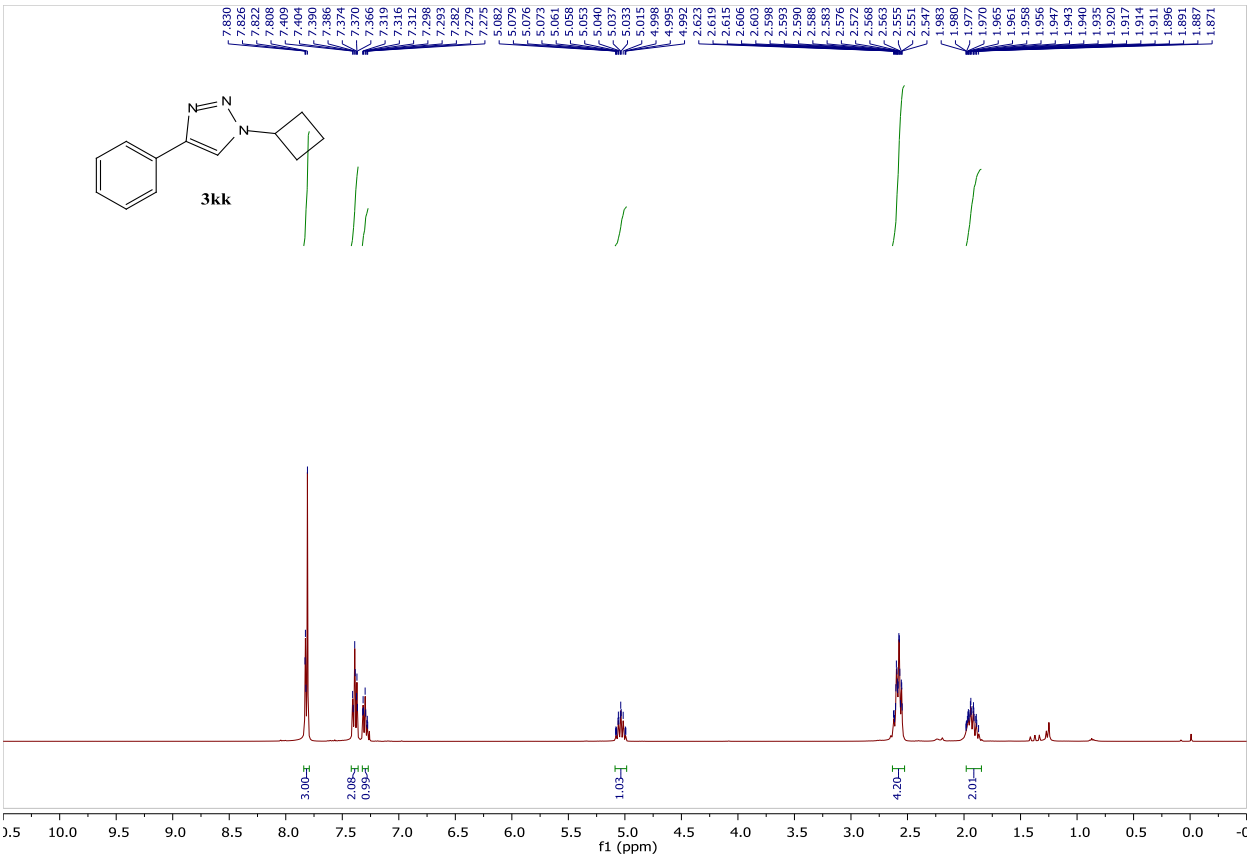
128.87
128.18
125.78
117.17
59.32
40.77
3jj
f1 (ppm)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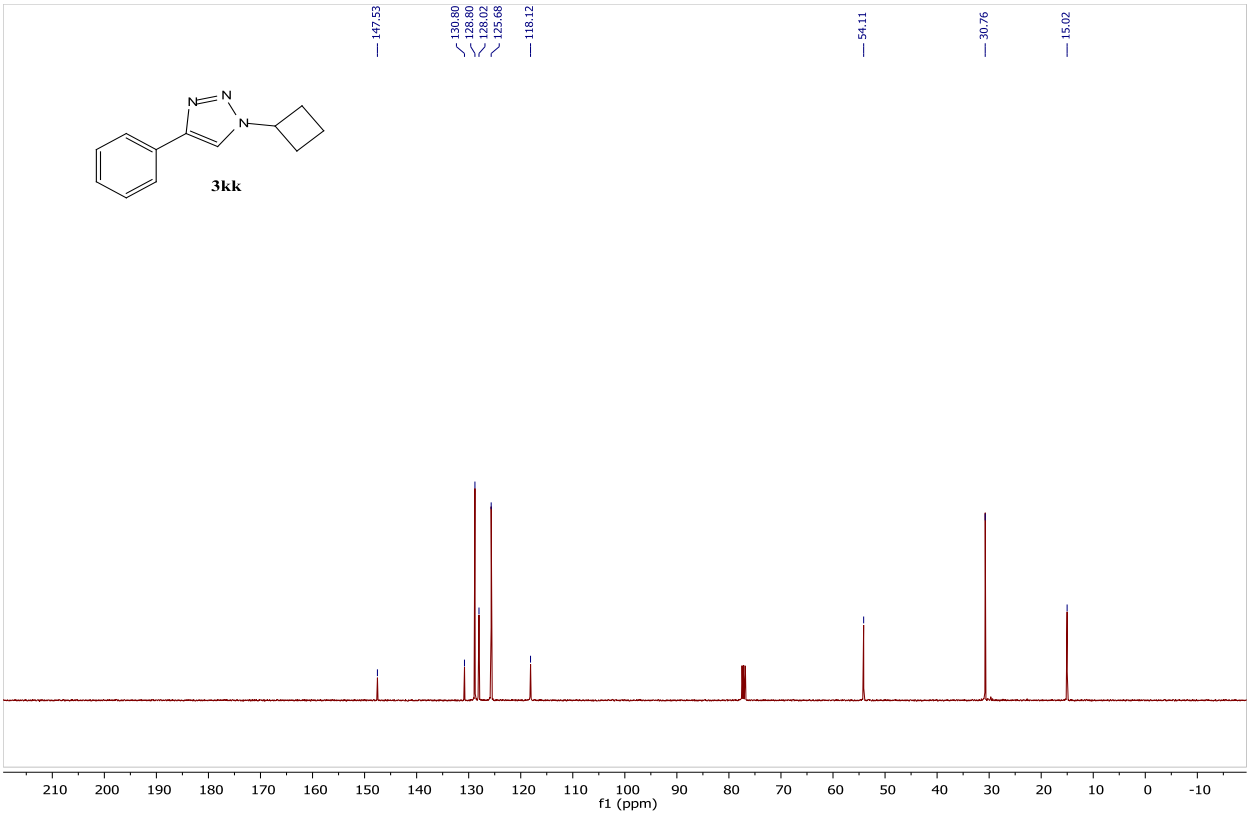
7.386
7.374
7.370
7.366
7.319
7.316
7.312
7.298
7.293
7.282
7.279
7.275
5.082
5.079
5.076
5.073
5.061
5.058
5.053
5.040
5.037
5.033
5.015
4.998
4.995
4.992
2.623
2.619
2.615
2.606
2.603
2.598
2.593
2.590
2.588
2.583
2.576
2.572
2.568
2.563
2.555
2.551
2.547
1.983
1.980
1.977
1.970
1.965
1.961
1.958
1.956
1.947
1.943
1.940
1.935
1.920
1.917
1.914
1.911
1.896
1.891
1.887
1.871
3kk
3.00
2.08
0.99
1.03
4.20
2.01
f1 (ppm)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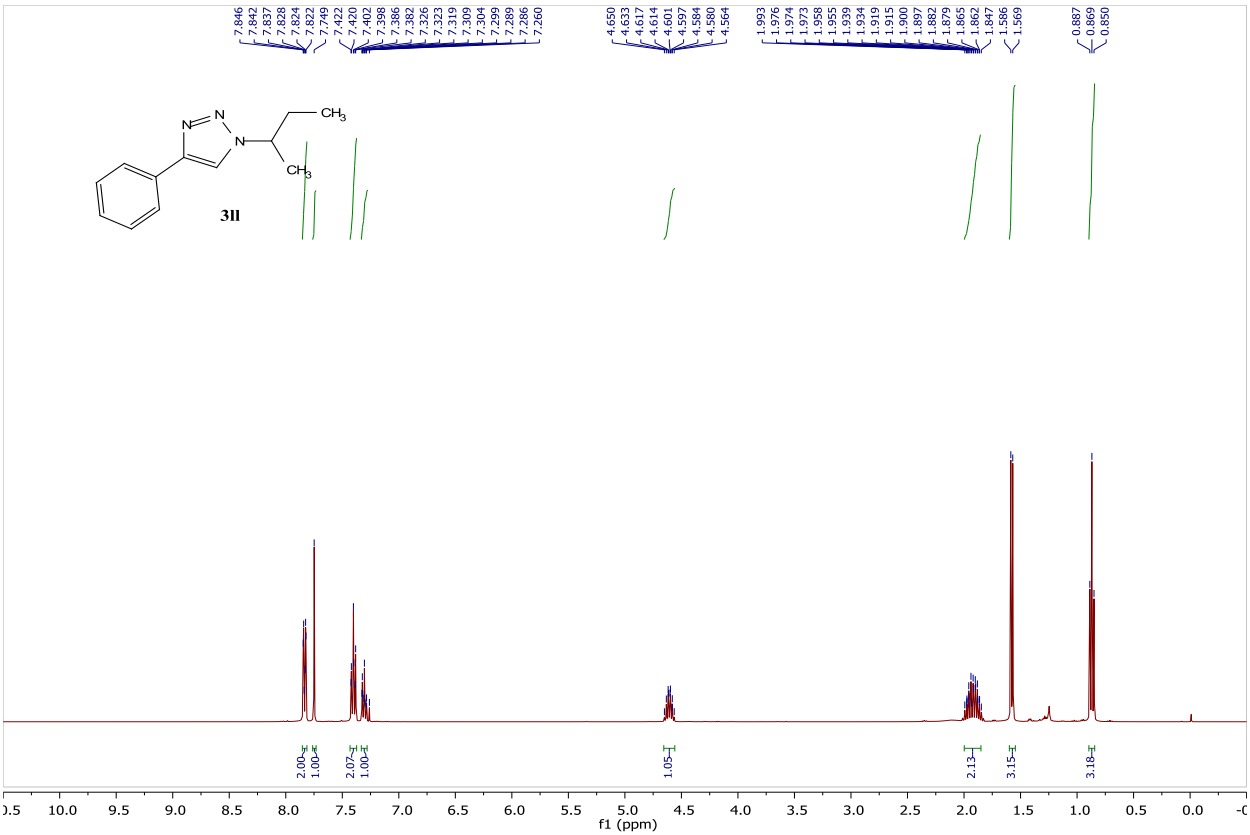
128.80
128.02
125.68
118.12
54.11
30.76
15.02
3kk
f1 (ppm)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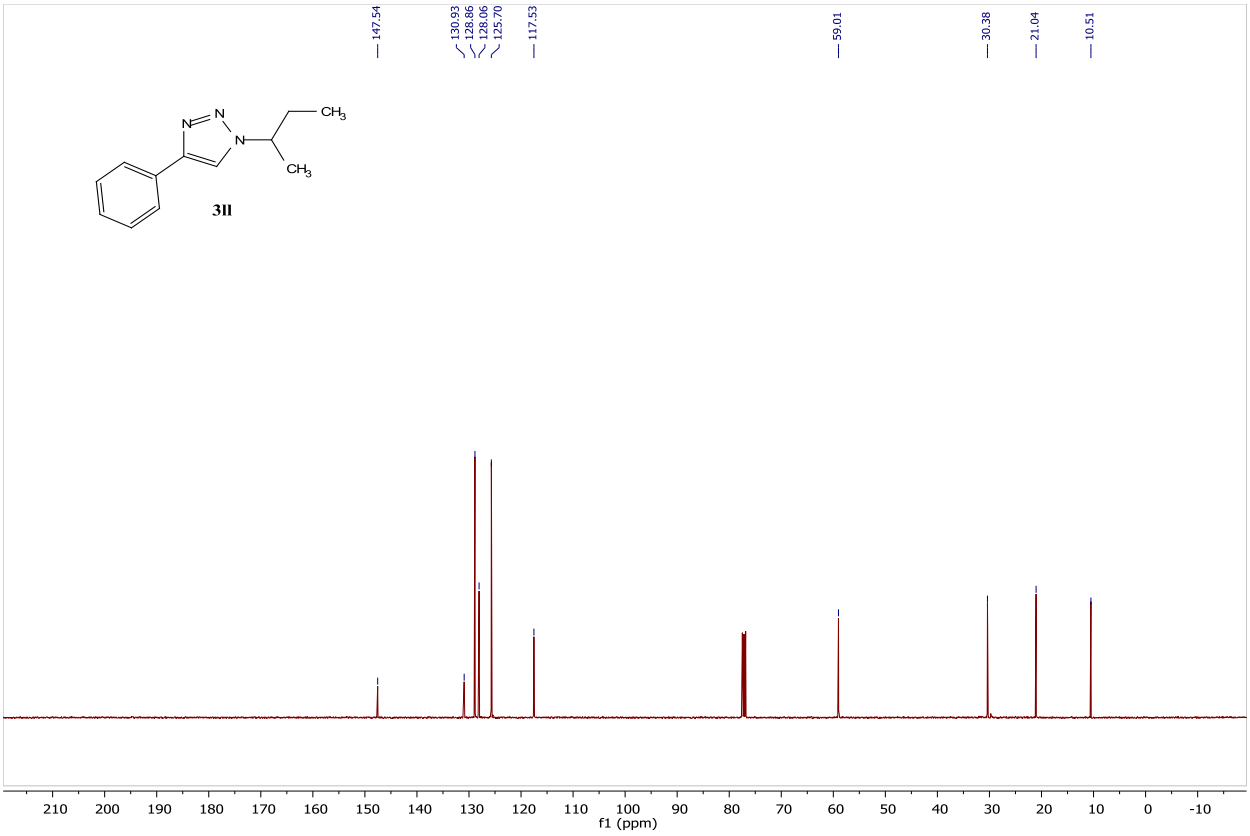
7.420
7.402
7.398
7.386
7.382
7.326
7.323
7.319
7.309
7.304
7.299
7.289
7.286
7.260
4.650
4.633
4.617
4.614
4.601
4.597
4.584
4.580
4.564
1.993
1.976
1.974
1.973
1.958
1.955
1.939
1.934
1.919
1.915
1.900
1.897
1.882
1.879
1.865
1.862
1.847
1.586
1.569
0.887
0.869
0.850
CH3
CH3
3ll
2.00
1.00
2.07
1.00
1.05
2.13
3.15
3.18
f1 (ppm)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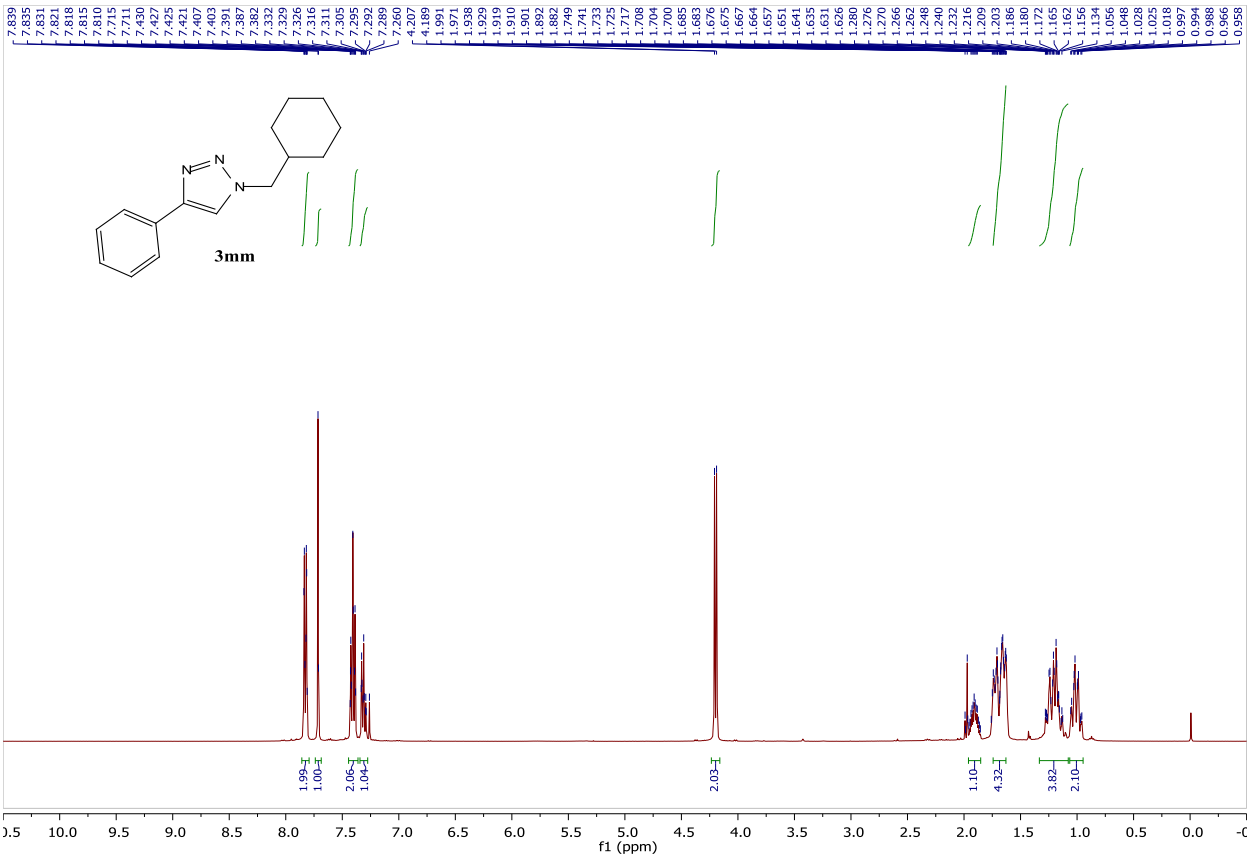
130.93
128.86
128.06
125.70
117.53
59.01
30.38
21.04
10.51
CH3
CH3
3ll
f1 (ppm)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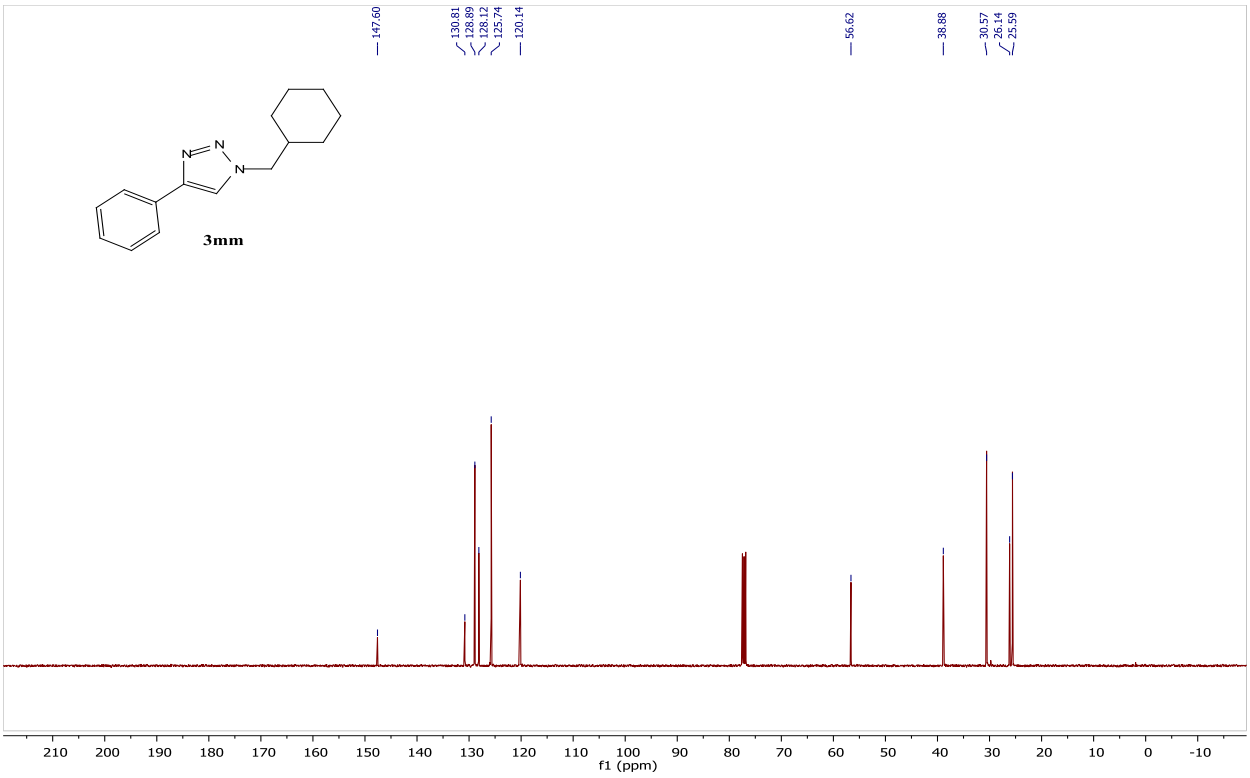
1.00
2.06
1.04
2.03
1.10
4.32
3.82
2.10
f1 (ppm)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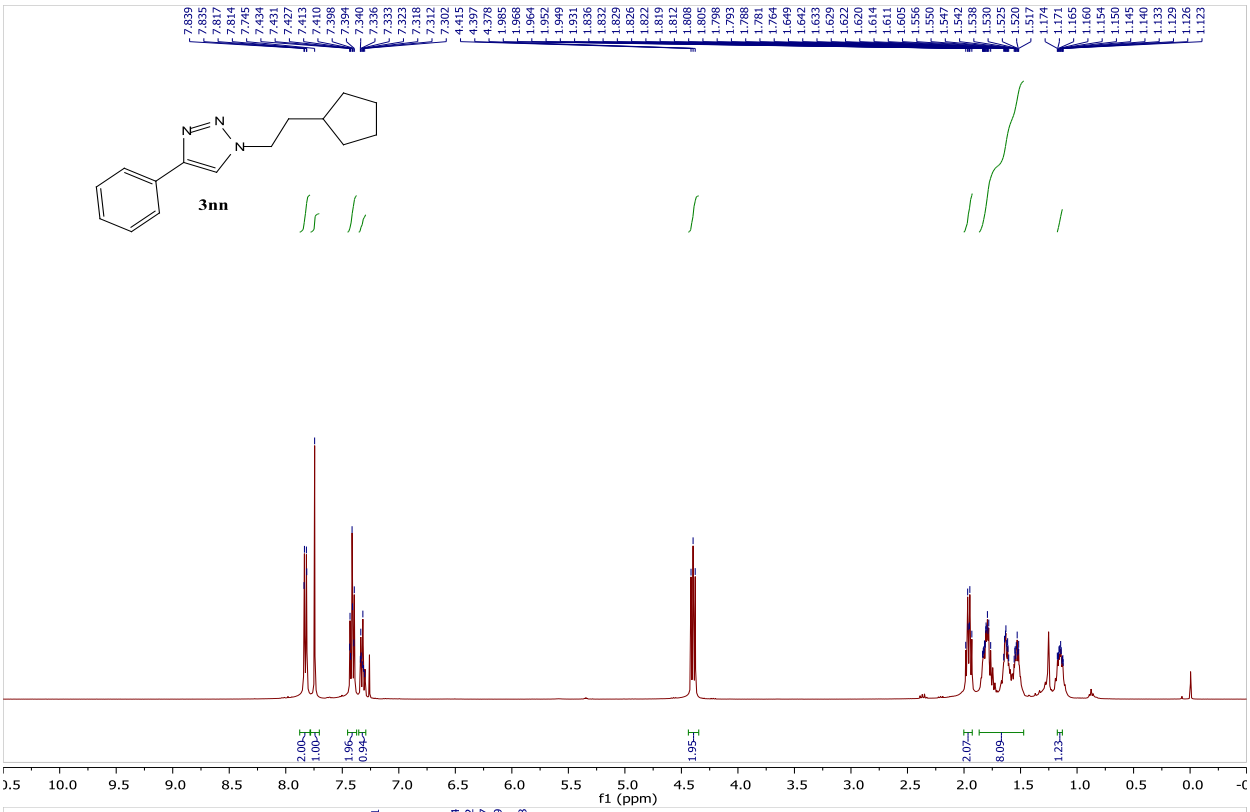
120.14
56.62
38.88
30.57
26.14
25.59
3mm
f1 (ppm)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7.427
7.413
7.410
7.398
7.394
7.340
7.336
7.333
7.323
7.318
7.312
7.302
4.415
4.397
4.378
1.985
1.968
1.964
1.952
1.949
1.931
1.836
1.832
1.829
1.826
1.822
1.819
1.812
1.808
1.805
1.798
1.793
1.788
1.781
1.764
1.649
1.642
1.633
1.629
1.622
1.620
1.614
1.611
1.605
1.556
1.550
1.547
1.542
1.538
1.530
1.525
1.520
1.517
1.174
1.171
1.165
1.160
1.154
1.150
1.145
1.140
1.133
1.129
1.126
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1.00
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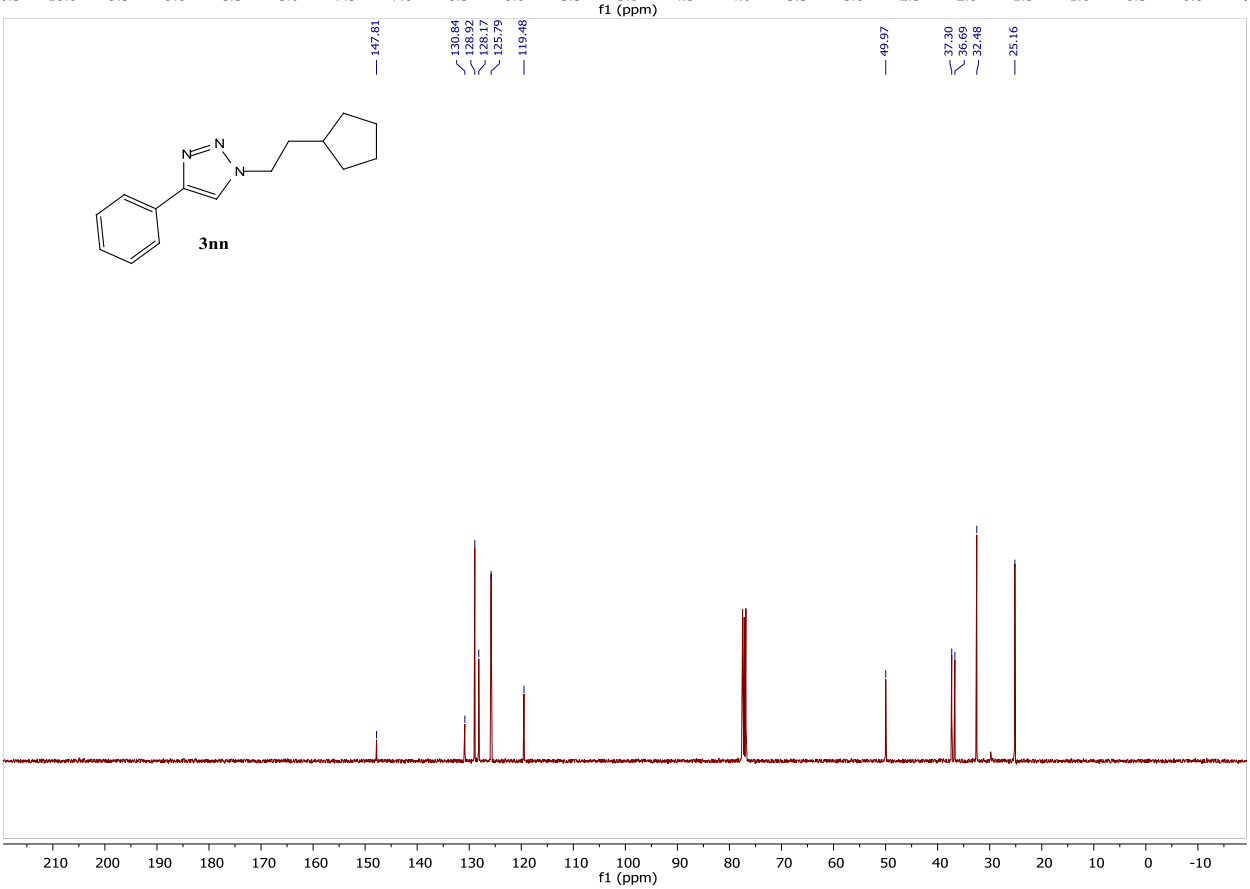
0.94
1.95
2.07
8.09
1.23
f1 (ppm)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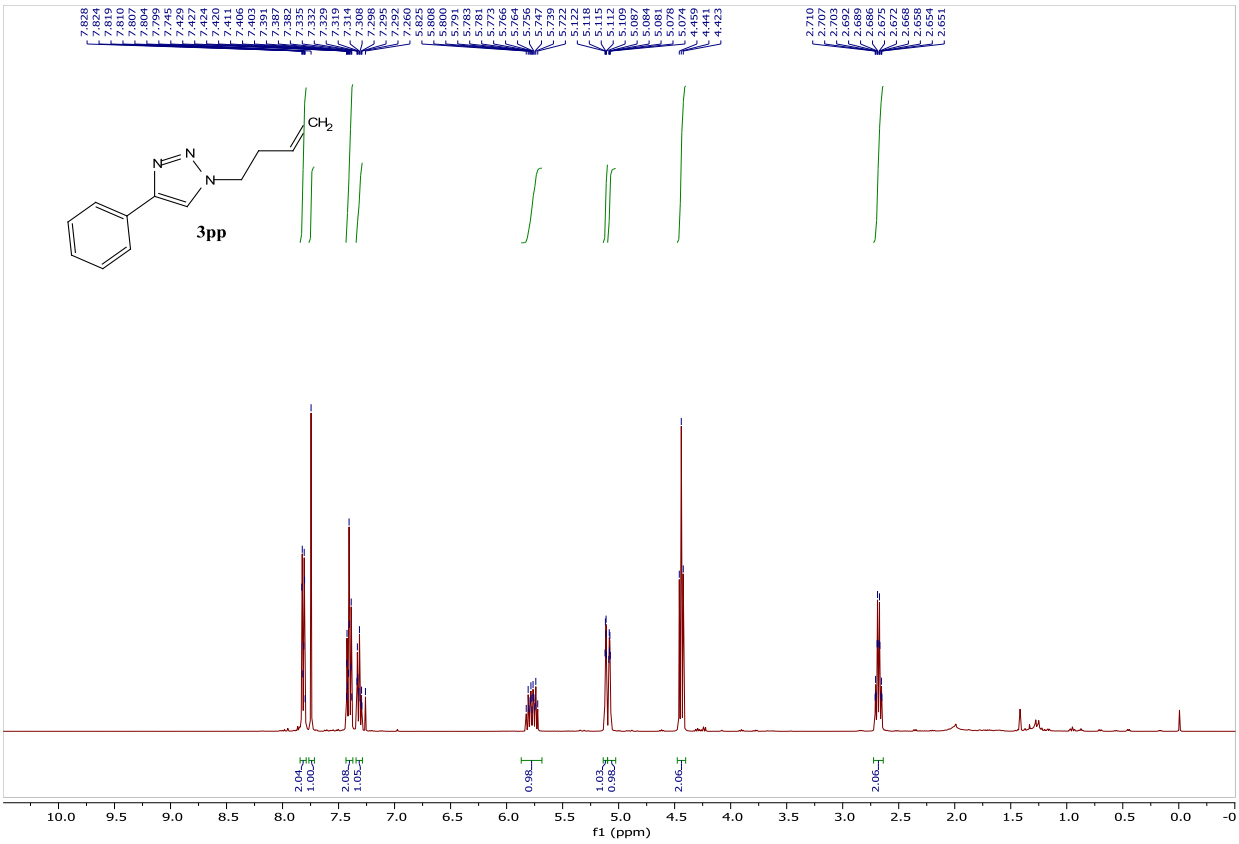
37.30
36.69
32.48
25.16
3nn
f1 (ppm)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7.819
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7.807
7.804
7.799
7.745
7.429
7.427
7.424
7.410
7.411
7.406
7.403
7.391
7.387
7.382
7.335
7.332
7.329
7.319
7.314
7.308
7.298
7.295
7.292
7.260
5.825
5.808
5.800
5.791
5.783
5.781
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5.766
5.764
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5.747
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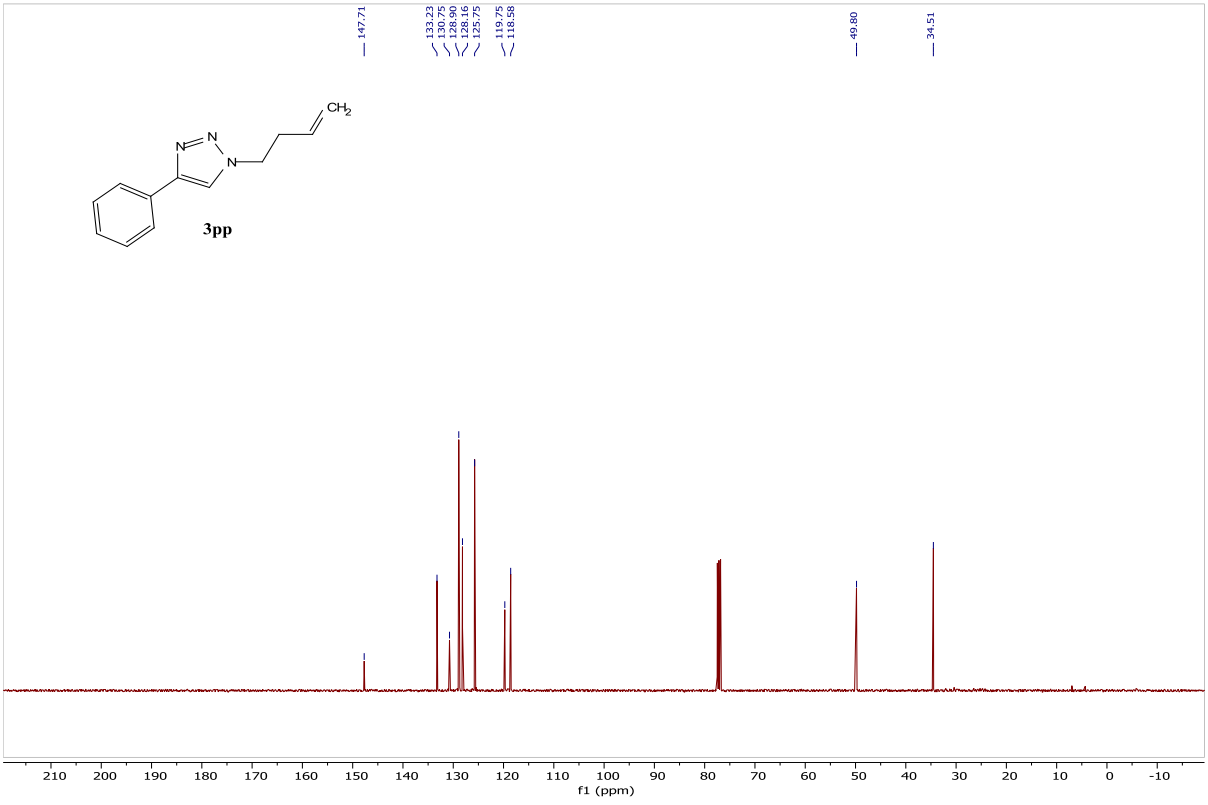
5.722
5.122
5.118
5.115
5.112
5.109
5.087
5.084
5.081
5.078
5.074
4.459
4.441
4.423
2.710
2.707
2.703
2.693
2.689
2.686
2.675
2.672
2.668
2.658
2.654
2.651
CH2
N
N
N
3pp
2.04
1.00
2.08
1.05
0.98
1.03
0.98
2.06
2.06
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0
f1 (ppm)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116.58
49.80
34.51
CH2
N
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N
3pp
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)