Electronic Supplementary Material (ESI) for ChemComm.

Decatungstate-Catalysed C(sp^3)–H Azidation

Yen-Chu Lu, Shi-Chieh Kao and Julian G. West*

Department of Chemistry, Rice University, 6500 Main St, Houston, TX, USA. Email: jgwest@rice.edu. Web: westchem.org

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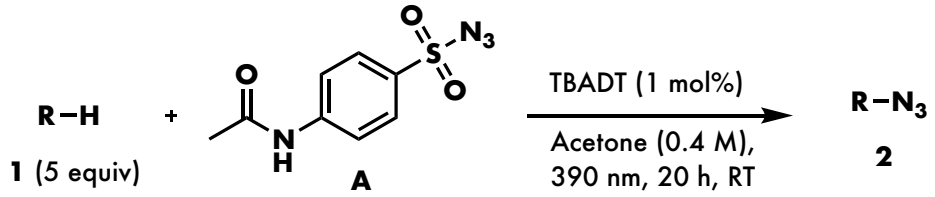
General Information

All reagents were purchased from the commercially available sources and used without further purification. All reactions were carried out in a vial with magnetic stirring. Irradiation of reactions was achieved using Kessil PR160L-390 or 370 lamps. All reactions were monitored by either ^{1}H NMR or thin layer chromatography (TLC) carried out on 0.25 mm pre-coated silia plates (F-254) purchased from Silicycle, Quebec, Canada, using shortwave UV light as visualizing agent and $KMnO_4$ or phosphomolybdic acid (PMA) as developing agents. Flash column chromatography was performed using SiliaFlash-P60 silica gel (40 – 63 µm) purchased from Silicycle, Quebec, Canada. ^{1}H and ^{13}C NMR spectra were recorded on a Bruker DRX-600 spectrometers operating at 600 MHz for proton nuclei and 151 MHz for carbon were calibrated using residual undeuterated solvent as an internal reference ($CDCl_3$: 7.26 ppm ^{1}H NMR and 77.20 ppm ^{13}C NMR). For reporting NMR peak multiplicities, the following abbreviations were used: s = singlet, d = doublet, t = triplet, q = quartet, quin = quintet, hept = heptet, m = multiplet. High-resolution mass spectra (HRMS) were recorded on an Agilent UHPLC TOF mass spectrometer using electrospray ionization time-of-flight (ESI-TOF) or chemical ionization time-of-flight (CI-TOF) reflectron experiments.

CAUTION: Azides are potentially explosive compounds; Extreme caution is necessary and proper safety measures should be taken. (although we have not encountered any problem during the project) Organic azides with $(N_C + N_O)/N_N$ ratio more than 3 are normally stable and can be isolated and stored in pure form. Azides with $(N_C + N_O)/N_N$ less than 3 but more than 1 can be isolated but should be stored in fridge at no more than 1 M concentration and at a maximum of 5 grams of material. Azides with $(N_C + N_O)/N_N < 1$ should never be isolated, as they are very unstable and potentially explosive.

Experimental Section

General procedure for the TBADT-catalysed C–H azidation



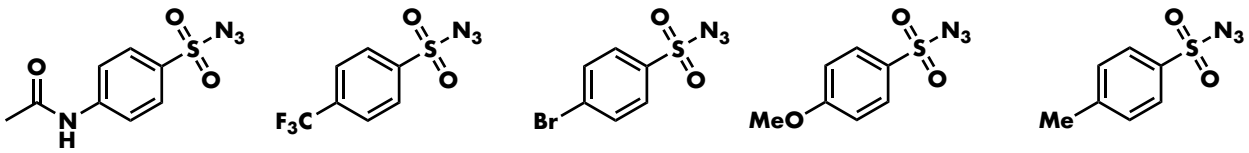
Azidation substrate (1.0 mmol, 5.0 equiv), 4-acetamidobenzenesulfonyl azide (48 mg, 0.2 mmol, 1.0 equiv) and TBADT (6.64 mg, 0.002 mmol, 0.01 equiv) were loaded to a vial which was capped and then evacuated and backfilled with N_2 twice. Acetone (0.5 mL) was subsequently added to the mixture, sparged with N_2 balloon for 5 min and sealed with parafilm. The reaction was stirred under the irradiation of 390 nm LED light (Kessil PR160L-370, 5-10 cm from the reaction vial) for 20 h. Upon completion, the reaction was extracted with CH_2Cl_2, dried over Na_2SO_4, filtered and concentrated in vacuo for column purification (100% hexane elution) to afford pure azidocyclooctane (**2a**).

Preparation of tetrabutylammonium decatungstate (TBADT)

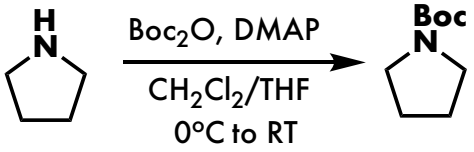
TBADT was prepared according to the literature.[1]

Preparation of sulfonyl azides

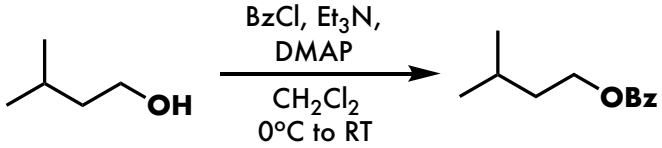
4-Acetamidobenzenesulfonyl azide (**A**) was purchased directly from Sigma-Aldrich and used without further purification. **4-(Trifluoromethyl)benzenesulfonyl azide** (**B**), **4-bromobenzenesulfonyl azide** (**C**), **4-methoxybenzenesulfonyl azide** (**D**), **4-methylbenzenesulfonyl azide** (**E**) were prepared according to the literature.[2]



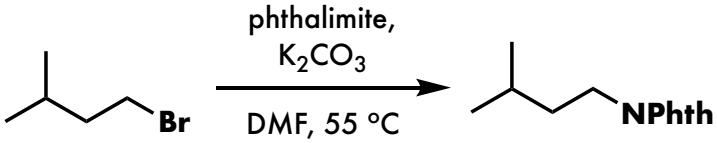
Preparation of substrates



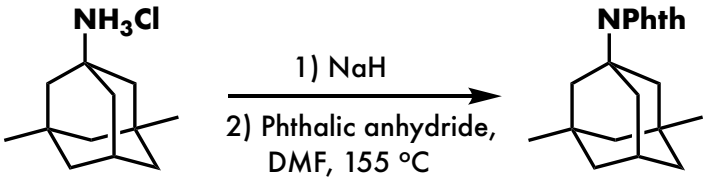
***t*-Butyl pyrrolidine-1-carboxylate**[3]: To a CH_2Cl_2 solution (50 mL) of pyrrolidine (1.0 g, 14.06 mmol, 1.0 equiv) was added Boc_2O (16.8 mL, 16.87 mmol, 1.2 equiv (1 M in THF)) and DMAP (342 mg, 2.8 mmol, 0.2 equiv) at 0 °C. The reaction was then allowed to warm to RT and stirred until the consumption of starting material as monitored by TLC. The mixture was extracted with CH_2Cl_2, dried over Na_2SO_4, filtered and concentrated in vacuo for column purification to afford pure t-Butyl pyrrolidine-1-carboxylate.



Isopentyl benzoate[4]**:** To a CH_2Cl_2 solution (30 mL) of 3-methylbutan-1-ol (1.0 g, 11.3 mmol, 1.0 equiv) was added triethylamine (1.7 g, 16.95 mmol, 1.5 equiv) and DMAP (cat.). The reaction mixture was stirred at 0 °C followed by the slow addition of benzoyl chloride (1.58 mL, 13.6 mmol, 1.2 equiv) and stirred at RT. Upon completion, the reaction mixture was washed with ammonium chloride, dried over Na_2SO_4, filtered and concentrated in vacuo for column purification to afford pure isopentyl benzoate. *** The same procedure was followed for the synthesis of 5-methylhexan-2-yl benzoate, substituting 5-methylhexan-2-ol for an equal amount of 3-methylbutan-1-ol.**



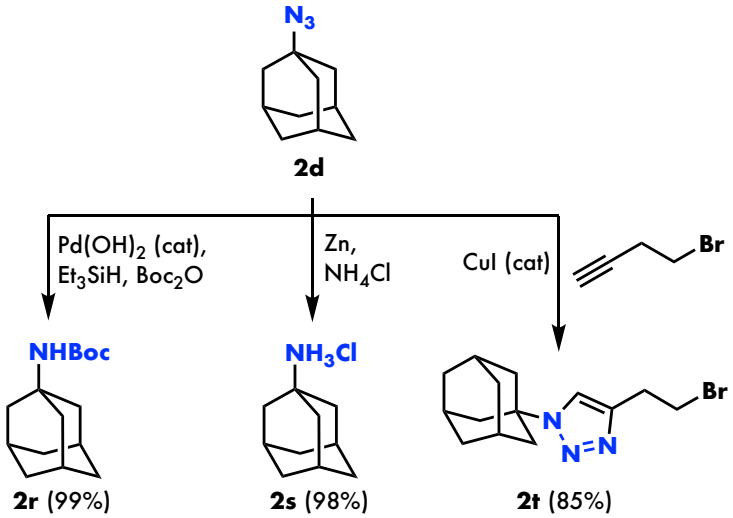
2-Isopentylisoindoline-1,3-dione: To a DMF solution (20 mL) of phthalimite (1.17 g, 7.94 mmol, 1.2 equiv) and K_2CO_3 (1.1 g, 7.94 mmol, 1.2 equiv) was added 1-bromo-3-methylbutane (1 g, 6.62 mmol, 1.0 equiv). The reaction was stirred vigorously at 55 °C. Upon completion, the reaction was diluted with diethyl ether, washed with water, dried over Na_2SO_4, filtered and concentrated in vacuo for column purification to afford pure 2-Isopentylisoindoline-1,3-dione.

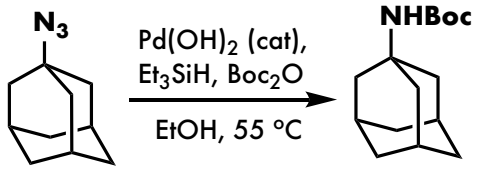


***N*-Phth-Memantine[5]:** To a DMF (20 mL) solution of 3,5-Dimethyl-1-adamantanamine hydrochloride (1.4 g, 6.5 mmol, 1.0 equiv) was added sodium hydride (260 mg, 10.8 mmol, 1.66 equiv) and stirred at RT for 20 min. Phthalic anhydride (1.44 g, 9.73 mmol, 1.5 equiv) was then added to the reaction and stirred at 155 °C. Upon completion, the reaction was cooled down to RT, diluted with diethyl ether, washed by water, dried over Na_2SO_4, filtered and concentrated in vacuo for column purification to afford pure *N*-Phth-Memantine.

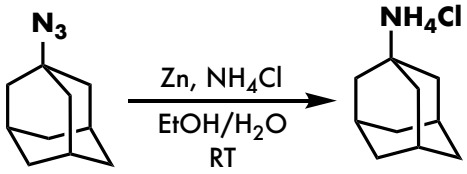
Derivatization of Azide Products

To demonstrate the applications of azido compounds, the post-functional group modifications of **2d** were conducted. First, the reductive *tert*-butyloxycarbonylation of azide afforded *N*-Boc-protected adamantane **2r** in a 99% yield. Similarly, the treatment of **2d** with Zn/NH_4Cl generated 98% of the corresponding ammonium chloride salt **2s** as a stable amine building block. Finally, a copper-catalysed azide-alkyne 1,3-dipolar cycloaddition (CuAAC) with 4-bromobut-1-yne furnished 85% of triazole product **2t**, showing a convenient ligation strategy of simple hydrocarbons with various molecules.

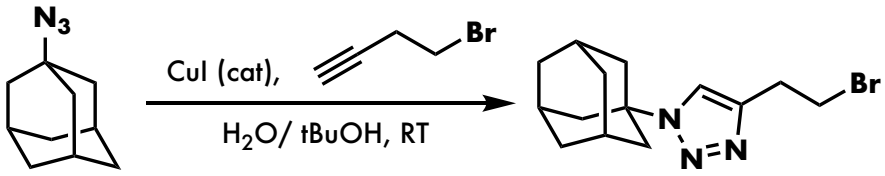




To an ethanol (1 mL) solution of 1-azido-adamantane **2d** (35.4 mg, 0.2 mmol, 1.0 equiv), $Pd(OH)_2/C$ (2.8 mg, 0.02 mmol, 10 mol%) and Boc_2O (87.3 mg, 0.4 mmol, 2.0 equiv) was added Et_3SiH (46.5 mg, 0.4 mmol, 2.0 equiv) and the reaction was stirred at 55 °C for 20 h. Until completion, the reaction solution was filtered through the celite and concentrated for column purification to afford the pure *N*-Boc-protected **2r** (49.8 mg, 99%).



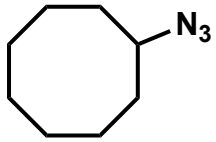
To a mixture of 1-azido-adamantane **2d** (35.4 mg, 0.2 mmol, 1.0 equiv), Zinc (15.6 mg, 0.24 mmol, 1.2 equiv) and NH_4Cl (26.8 mg, 0.5 mmol, 2.5 equiv) was added ethanol (0.5 mL) and water (0.2 mL) then stirred at RT for 20 h. Until completion, the solution was directly concentrated for column purification to afford the pure ammonium salt **2s** (36.8 mg, 98%). * The addition of NH_3 solution follow by an extraction with H_2O would afford the free amine compound.



To a mixture of 1-azido-adamantane **2d** (35.4 mg, 0.2 mmol, 1.0 equiv), 4-bromobut-1-yne (26.6 mg, 0.2 mmol, 1.0 equiv) and copper iodide (1.9 mg, 0.01 mmol, 5 mol%) was added H_2O (0.5 mL) and *t*BuOH (0.2 mL) and stirred under air for 20 h. Until completion, the solution was extracted with H_2O, dried over Na_2SO_4, filtered and concentrated in vacuo for column purification to afford the triazole product **2t** (52.7 mg, 85%).

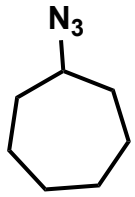
Characterization of Azide Compounds

azidocyclooctane (2a)



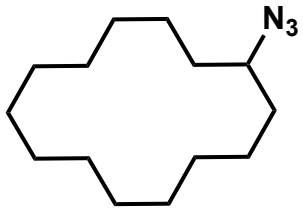
15 mg; **^{1}H NMR (600 MHz, $CDCl_3$):** δ 3.56 (hept, *J* = 4.2 Hz, 1H), 1.92 – 1.81 (m, 2H), 1.78 – 1.65 (m, 4H), 1.63 – 1.44 (m, 8H). **^{13}C NMR (150 MHz, $CDCl_3$):** δ 62.4, 30.9, 27.4, 25.3, 23.3. **HRMS (APCI):** calc'd for $C_8H_{16}N$ $[M\text{-}N_2+H]^+$ 126.1277; Found 126.1279.

azidocycloheptane (2b)



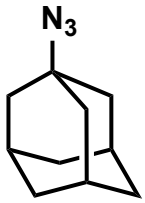
8.6 mg; **^{1}H NMR (600 MHz, $CDCl_3$):** δ 3.52 (hept, *J* = 4.3 Hz, 1H), 1.96 – 1.89 (m, 2H), 1.72 – 1.51 (m, 8H), 1.48 – 1.38 (m, 2H). **^{13}C NMR (150 MHz, $CDCl_3$):** δ 62.8, 33.9, 28.0, 23.6. **HRMS (APCI):** calc'd for $C_7H_{14}N_3$ $[M+H]^+$ 140.1182; Found 140.1182.

azidocyclotetradecane (2c)



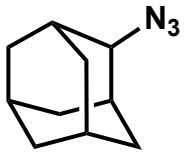
17.1 mg; **^{1}H NMR (600 MHz, $CDCl_3$):** δ 3.51 – 3.45 (m, 1H), 1.73 – 1.63 (m, 2H), 1.55 – 1.27 (m, 20H). **^{13}C NMR (150 MHz, $CDCl_3$):** δ 59.3, 29.2, 24.1, 23.8, 23.5, 23.4, 21.4. **HRMS (APCI):** calc'd for $C_{14}H_{28}N_3$ $[M+H]^+$ 238.2278; Found 238.2360.

(3s,5s,7s)-1-azidoadamantane (2d)



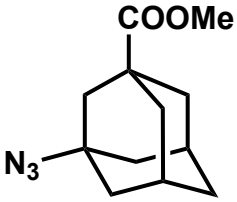
15.5 mg; **^{1}H NMR (600 MHz, $CDCl_3$):** δ 2.14 (br, 3H), 1.79 (d, *J* = 2.7 Hz, 6H), 1.66 (dd, *J* = 12.4, 30.4 Hz, 6H). **^{13}C NMR (150 MHz, $CDCl_3$):** δ 59.2, 41.7, 36.0, 30.0. **HRMS (APCI):** calc'd for $C_{10}H_{16}N$ [M -N_2+H]$^+$ 150.1277; Found 150.1277.

(1R,3S,5r,7r)-2-azidoadamantane (2d´)



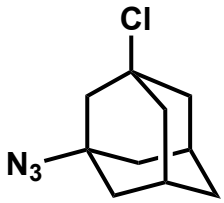
4.7 mg; **^{1}H NMR (600 MHz, $CDCl_3$):** δ 3.80 (br, 1H), 1.99 (d, *J* = 13.8 Hz, 4H), 1.91 – 1.80 (m, 4H), 1.76 – 1.69 (m, 4H), 1.57 – 1.52 (m, 2H). **^{13}C NMR (150 MHz, $CDCl_3$):** δ 66.6, 37.5, 36.8, 31.9, 31.7, 27.4, 27.1. **HRMS (APCI):** calc'd for $C_{10}H_{16}N$ [M -N_2+H]$^+$ 150.1277; Found 150.1278.

methyl (1r,3s,5R,7S)-3-azidoadamantane-1-carboxylate (2e)



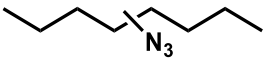
30.1 mg; **^{1}H NMR (600 MHz, $CDCl_3$):** δ 3.68 (s, 3H), 2.28 (br, 2H), 1.93 (s, 2H), 1.87 – 1.75 (m, 8H), 1.66 – 1.61 (br, 2H). **^{13}C NMR (150 MHz, $CDCl_3$):** δ 176.7, 59.0, 52.1, 43.2, 42.8, 40.8, 37.7, 35.0, 29.5. **HRMS (APCI):** calc'd for $C_{12}H_{18}NO_2$ [M -N_2+H]$^+$ 208.1332; Found 208.1333.

(1r,3s,5R,7S)-1-azido-3-chloroadamantane (2f)



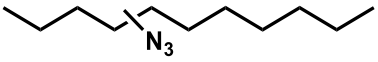
29.2 mg; **^{1}H NMR (600 MHz, $CDCl_3$):** δ 2.34 – 2.30 (br, 2H), 2.13 (s, 2H), 2.04 (q, *J* = 12.5 Hz, 4H), 1.74 (br, 4H), 1.62 – 1.53 (m, 2H). **^{13}C NMR (150 MHz, $CDCl_3$):** δ 66.5, 60.5, 51.2, 46.1, 39.9, 34.1, 31.9. **HRMS (APCI):** calc'd for $C_{10}H_{15}ClN$ $[M-N_2+H]^+$ 184.0888; Found 184.0889.

azidooctane isomeric mixture (2g)



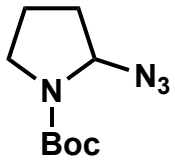
5.3 mg; **HRMS (APCI):** calc'd for $C_8H_{18}N$ $[M+H]^+$ 128.1434; Found 128.1431.

azidododecane isomeric mixture (2h)



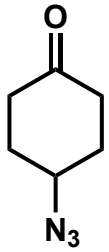
9.3 mg; **HRMS (APCI):** calc'd for $C_{12}H_{26}N$ $[M-N_2+H]^+$ 184.2060; Found 184.2054.

tert-butyl 2-azidopyrrolidine-1-carboxylate (2i)



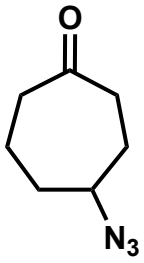
Crude ^{1}H NMR spectrum matches the reported literature and the yield was determined using trimethoxybenzene as the internal standard.[6] Note: This compound was unable to be isolated in a pure form and is known to decompose via column chromatography.

4-azidocyclohexan-1-one (2j)



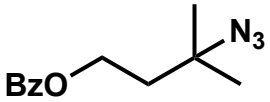
10.9 mg; **^{1}H NMR (600 MHz, $CDCl_3$):** δ 3.98 – 3.90 (m, 1H), 2.60 – 2.52 (m, 2H), 2.39 – 2.31 (m, 2H), 2.13 – 2.00 (m, 4H). **^{13}C NMR (150 MHz, $CDCl_3$):** δ 209.3, 56.9, 37.7, 30.8. **HRMS (APCI):** calc'd for $C_6H_9N_3O$ $[M+H]^+$ 140.0818; Found 140.0818.

4-azidocycloheptan-1-one (2k)



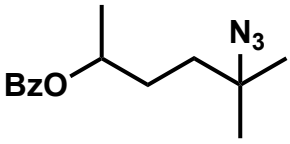
15 mg; **^{1}H NMR (600 MHz, $CDCl_3$):** δ 3.73 – 3.65 (m, 1H), 2.72 – 2.62 (m, 1H), 2.54 – 2.36 (m, 3H), 2.04 – 1.90 (m, 3H), 1.89 – 1.72 (m, 2H), 1.71 – 1.60 (m, 1H). **^{13}C NMR (150 MHz, $CDCl_3$):** δ 213.0, 61.7, 43.3, 38.4, 34.8, 29.0, 19.4. **HRMS (APCI):** calc'd for $C_7H_{12}NO$ $[M-N_2+H]^+$ 126.0913; Found 126.0914.

3-azido-3-methylbutyl benzoate (2l)



15.9 mg; **^{1}H NMR (600 MHz, $CDCl_3$):** δ 8.06 – 8.01 (m, 2H), 7.58 – 7.54 (m, 1H), 7.44 (t, *J* = 8.1 Hz, 2H), 4.44 (t, *J* = 6.8 Hz, 2H), 1.98 (t, *J* = 6.8 Hz, 2H), 1.38 (s, 6H). **^{13}C NMR (150 MHz, $CDCl_3$):** δ 166.7, 133.2, 130.3, 129.7, 128.6, 61.4, 60.4, 39.9, 26.5. **HRMS (APCI):** calc'd for $C_{12}H_{16}NO_2$ $[M-N_2+H]^+$ 206.1176; Found 206.1174.

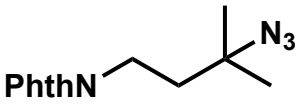
5-azido-5-methylhexan-2-yl benzoate (2m)



9.9 mg; **^{1}H NMR (600 MHz, $CDCl_3$):** δ 8.08 – 8.00 (m, 2H), 7.59 – 7.52 (m, 1H), 7.48 – 7.39 (m, 2H), 5.18 – 5.10 (m, 1H), 1.85 – 1.67 (m, 2H), 1.66 – 1.49 (m, 2H), 1.40 – 1.33 (m, 3H), 1.28 (s, 6H). **^{13}C**

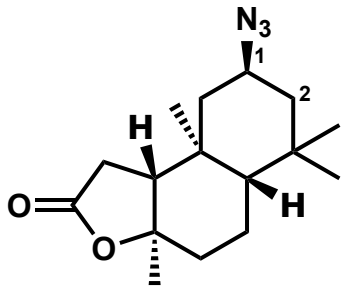
NMR (150 MHz, $CDCl_3$): δ 166.3, 133.0, 130.8, 129.7, 128.5, 71.6, 61.4, 37.2, 30.9, 26.2, 26.1, 20.3. **HRMS (APCI):** calc'd for $C_{14}H_{20}NO_2$ $[M\text{-}N_2+H]^+$ 234.1489; Found 234.1488.

2-(3-azido-3-methylbutyl)isoindoline-1,3-dione (2n)



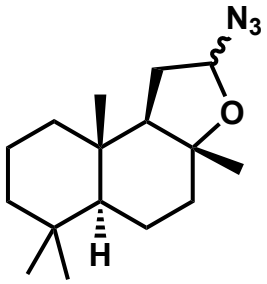
11.4 mg; **^{1}H NMR (600 MHz, $CDCl_3$):** δ 7.84 – 7.80 (m, 2H), 7.71 – 7.67 (m, 2H), 3.79 – 3.74 (m, 2H), 1.86 – 1.81 (m, 2H), 1.35 (s, 6H). **^{13}C NMR (150 MHz, $CDCl_3$):** δ 168.3, 134.1, 132.3, 123.4, 60.3, 39.3, 33.9, 26.1. **HRMS (APCI):** calc'd for $C_{13}H_{15}N_2O_2$ $[M\text{-}N_2+H]^+$ 231.1128; Found 231.1127.

(3aR,5aS,9aS,9bR)-8-azido-3a,5a,6,6,9a,9b-hexamethyldecahydronaphtho[2,1-b]furan-2(1H)-one (2o)



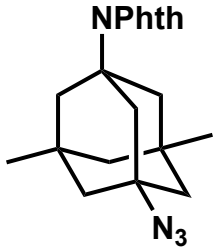
25.6 mg; **^{1}H NMR (major isomer, 600 MHz, $CDCl_3$):** δ 3.60 (t, 13.2 Hz, 1H), 2.47 – 2.35 (m, 1H), 2.30 – 2.20 (m, 1H), 2.12 – 1.85 (m, 3H), 1.85 – 1.56 (m, 5H), 1.46 – 0.83 (m, 18H). **^{13}C NMR (major isomer, 150 MHz, $CDCl_3$):** δ 176.2, 86.0, 58.8, 56.1, 54.0, 47.0, 44.6, 38.5, 37.1, 34.6, 33.1, 28.7, 21.7, 21.6, 20.3, 16.0. **HRMS (APCI):** calc'd for $C_{18}H_{30}NO_2$ $[M\text{-}N_2+CH_3OH+H]^+$ 296.2220; Found 296.2220.

(3aR,5aS,9aS,9bR)-2-azido-3a,6,6,9a-tetramethyldodecahydronaphtho[2,1-b]furan (2p)



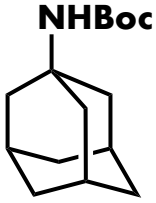
19.4 mg; ^{1}H NMR and ^{13}C NMR spectra match the reported literature as an isomeric mixture.[6] **HRMS (APCI):** calc'd for $C_{16}H_{28}NO$ $[M\text{-}N_2+H]^+$ 250.2165; Found 250.2164.

2-((1r,3s,5R,7S)-3-azido-5,7-dimethyladamantan-1-yl)isoindoline-1,3-dione (2q)



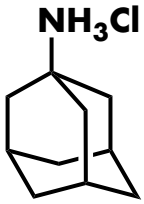
12.6 mg; **^{1}H NMR (600 MHz, $CDCl_3$):** δ 7.78 – 7.73 (m, 2H), 7.70 – 7.65 (m, 2H), 2.46 (s, 2H), 2.14 (s, 4H), 1.58 – 1.55 (m, 2H), 1.44 (d, *J* = 11.8 Hz, 2H), 1.29 – 1.23 (m, 2H), 1.17 (d, *J* = 12.7 Hz, 1H), 0.99 (s, 6H). **^{13}C NMR (150 MHz, $CDCl_3$):** δ 169.6, 134.1, 131.9, 122.9, 61.9, 60.8, 49.2, 46.6, 45.0, 42.8, 34.0, 29.6. **HRMS (APCI):** calc'd for $C_{20}H_{23}N_2O_2$ $[M\text{-}N_2+H]^+$ 323.1754; Found 323.1752.

tert-butyl (3s,5s,7s)-adamantan-1-ylcarbamate (2r)



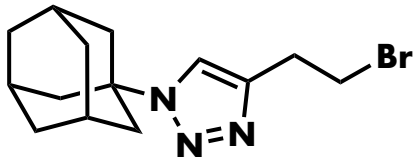
49.8 mg; **^{1}H NMR (600 MHz, $CDCl_3$):** δ 4.36 (br, 1H), 2.03 (s, 3H), 1.90 (s, 6H), 1.63 (s, 6H), 1.40 (s, 9H). **^{13}C NMR (150 MHz, $CDCl_3$):** δ 154.2, 78.7, 50.5, 42.0, 36.5, 29.6, 28.6. **HRMS (ESI):** calc'd for $C_{15}H_{26}NO_2$ $[M+H]^+$ 252.1951; Found 252.1958.

(3s,5s,7s)-adamantan-1-amine hydrochloride salt (2s)



36.8 mg; **^{1}H NMR (600 MHz, $CDCl_3$):** δ 8.05 (br, 3H), 2.13 (s, 3H), 2.02 (s, 6H), 1.67 (s, 6H). **^{13}C NMR (150 MHz, $CDCl_3$):** δ 53.1, 40.7, 35.5, 29.1. **HRMS (APCI):** calc'd for $C_{10}H_{18}N$ $[M\text{-}Cl]^+$ 152.1434; Found 152.1426.

1-((3s,5s,7s)-adamantan-1-yl)-4-(2-bromoethyl)-1H-1,2,3-triazole (2t)



52.7 mg; **^{1}H NMR (600 MHz, $CDCl_3$):** δ 7.49 (s, 1H), 3.63 (t, *J* = 7.0 Hz, 2H), 3.28 (t, *J* = 7.0 Hz, 2H), 2.27 – 2.19 (m, 9H), 1.82 – 1.73 (m, 6H). **HRMS (APCI):** calc'd for $C_{14}H_{21}BrN_3$ $[M+H]^+$ 312.0893; Found 312.0880.

References

(1) S. Protti, D. Ravelli, M. Fagnoni and A. Albini, *Chem. Commun.*, 2009, 7351 – 7353.

(2) (a) D. Meyer and P. Renaud*, Angew. Chem. Int. Ed.*, 2017, **56**, 10858 – 10861. (b) D. S. Breslow, M. F. Sloan, N. R. Newburg and W. B. Renfrow, *J. Am. Chem. Soc.*, 1969, **91**, 2273 – 2279.

(3) N. J. Taylor, E. Emer, S. Preshlock, M. Schedler, M. Tredwell, S. Verhoog, J. Mercier, C. Genicot and V. Gouverneur, *J. Am. Chem. Soc.*, 2017, **139**, 8267 – 8276.

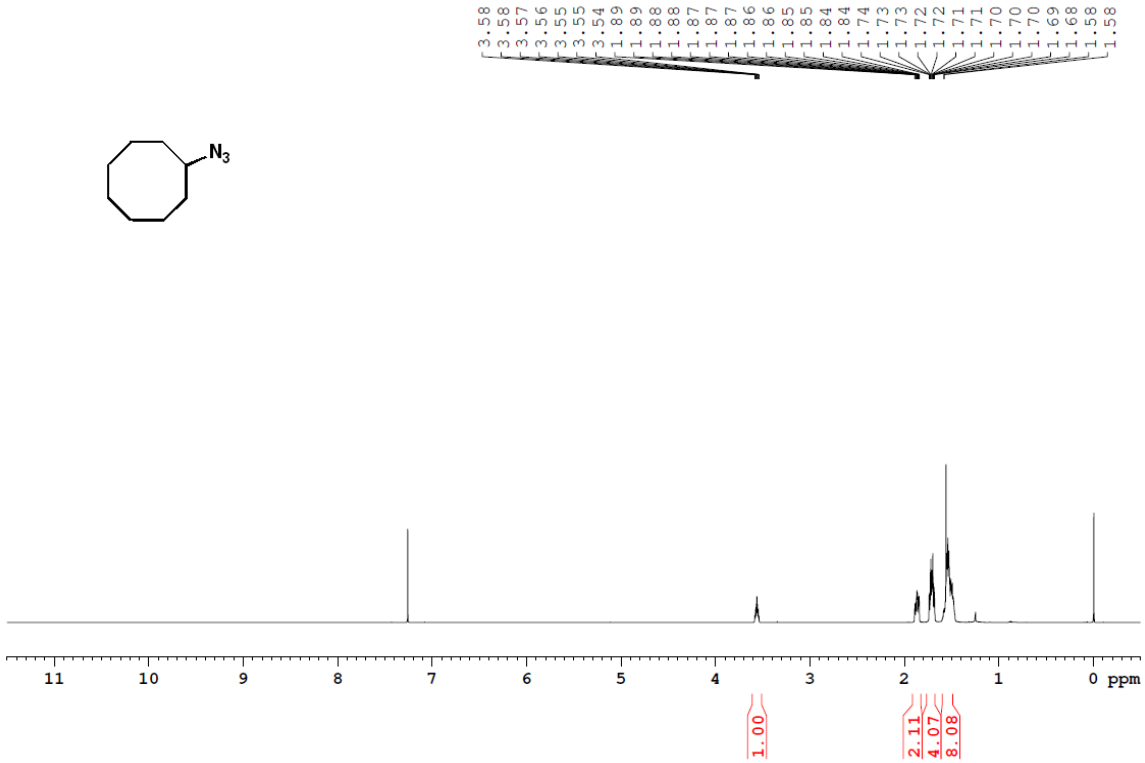
(4) S. Guo, X. Zhang and P. Tang, *Angew. Chem. Int. Ed.*, 2015, **54**, 4065 – 4069.

(5) V. A. Schmidt, R. K. Quinn, A. T. Brusoe and E. J. Alexanian, *J. Am. Chem. Soc.*, 2014, **136**, 14389 – 14392.

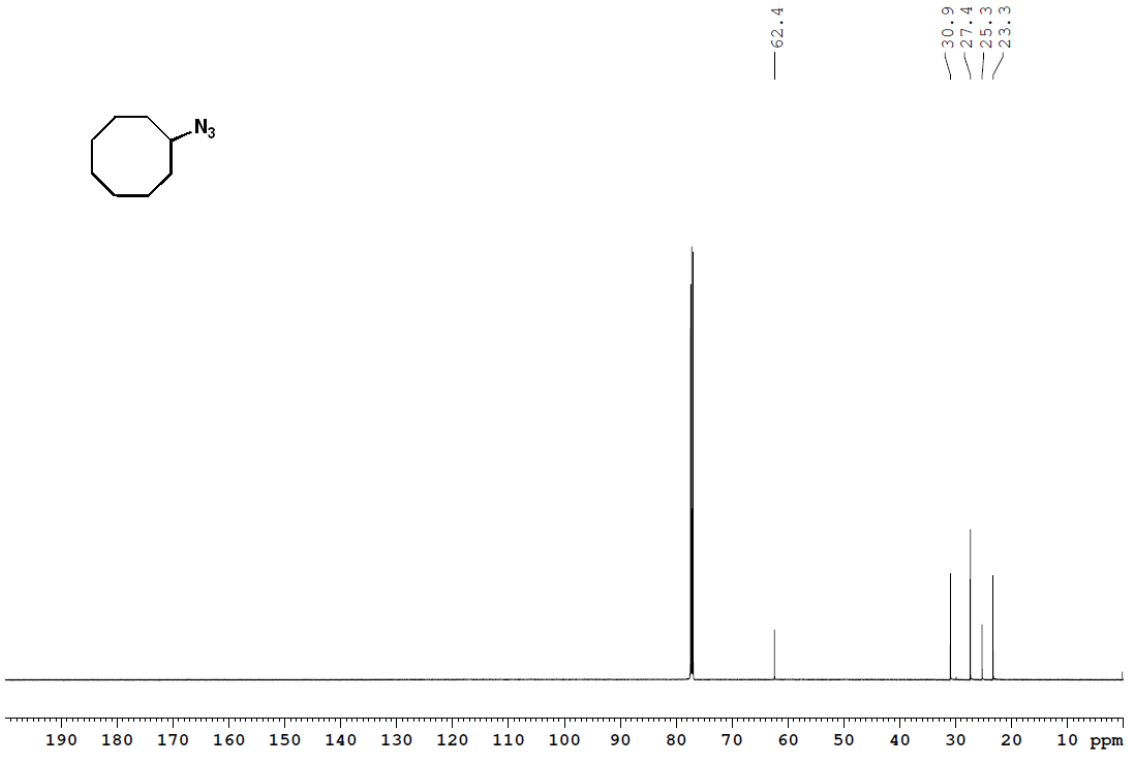
(6) S. Kamijo, M. Watanabe, K. Kamijo, K. Tao and T. Murafuji, *Synthesis*, 2016, **48**, 115 – 121.

NMR Spectra of Azide Compounds

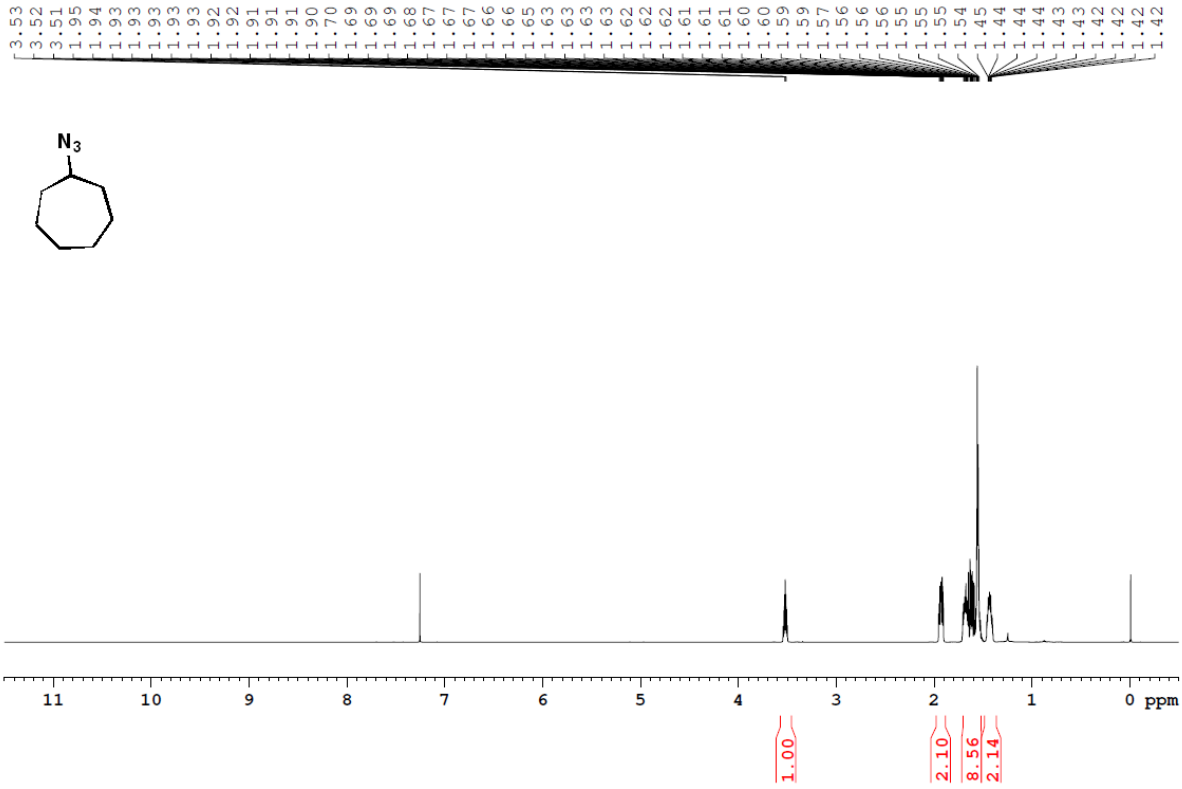
^{1}H NMR spectrum of **2a** (600 MHz, $CDCl_3$)



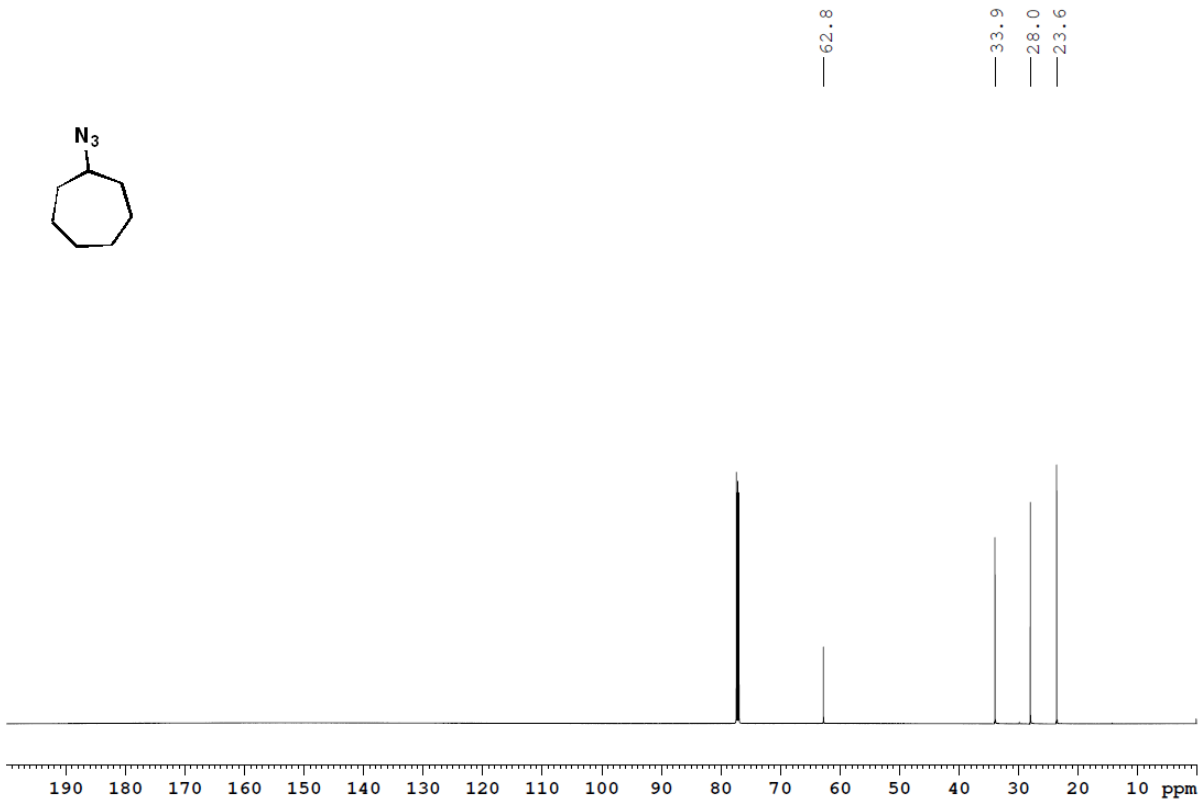
^{13}C NMR spectrum of **2a** (600 MHz, $CDCl_3$)



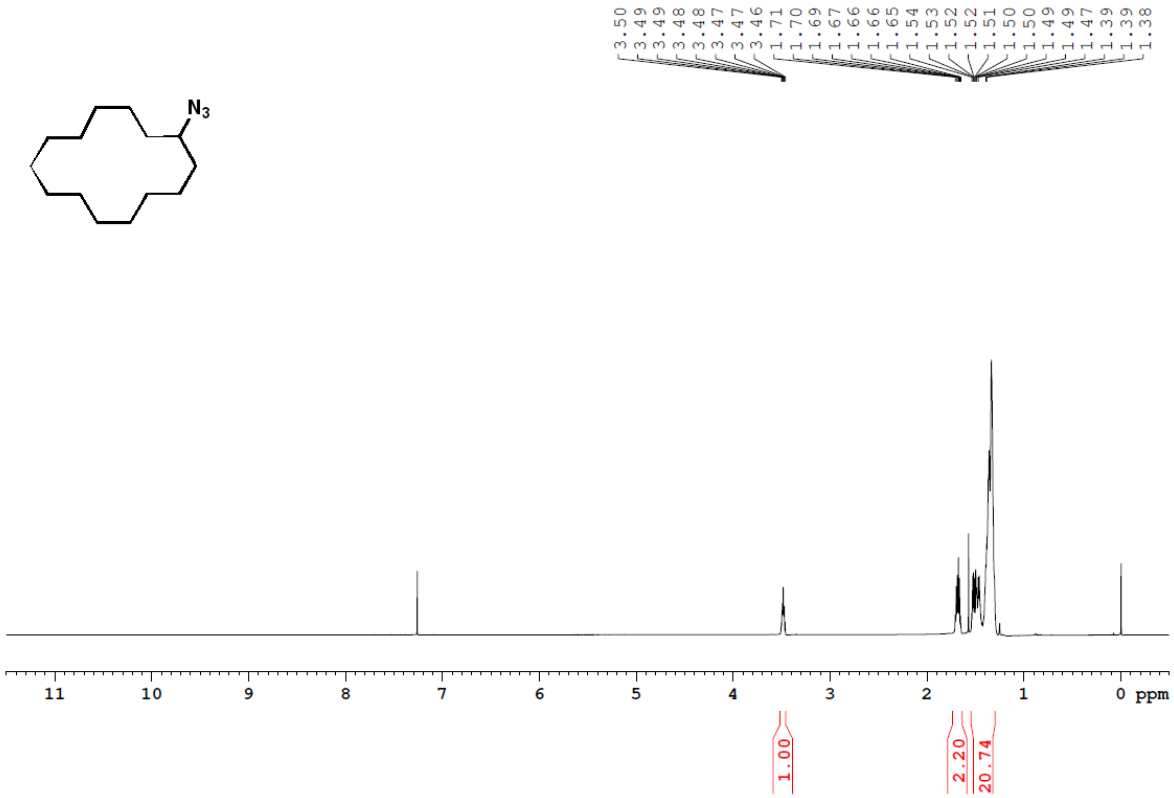
^{1}H NMR spectrum of **2b** (600 MHz, $CDCl_3$)



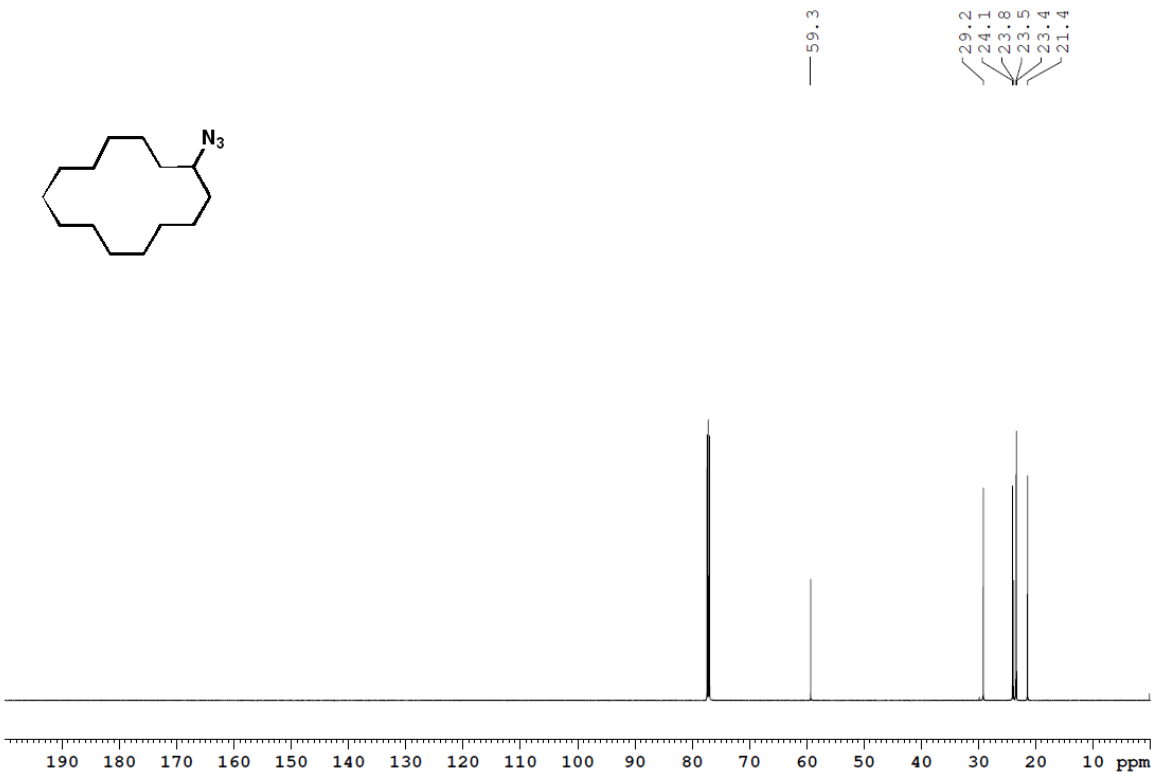
^{13}C NMR spectrum of **2b** (600 MHz, $CDCl_3$)



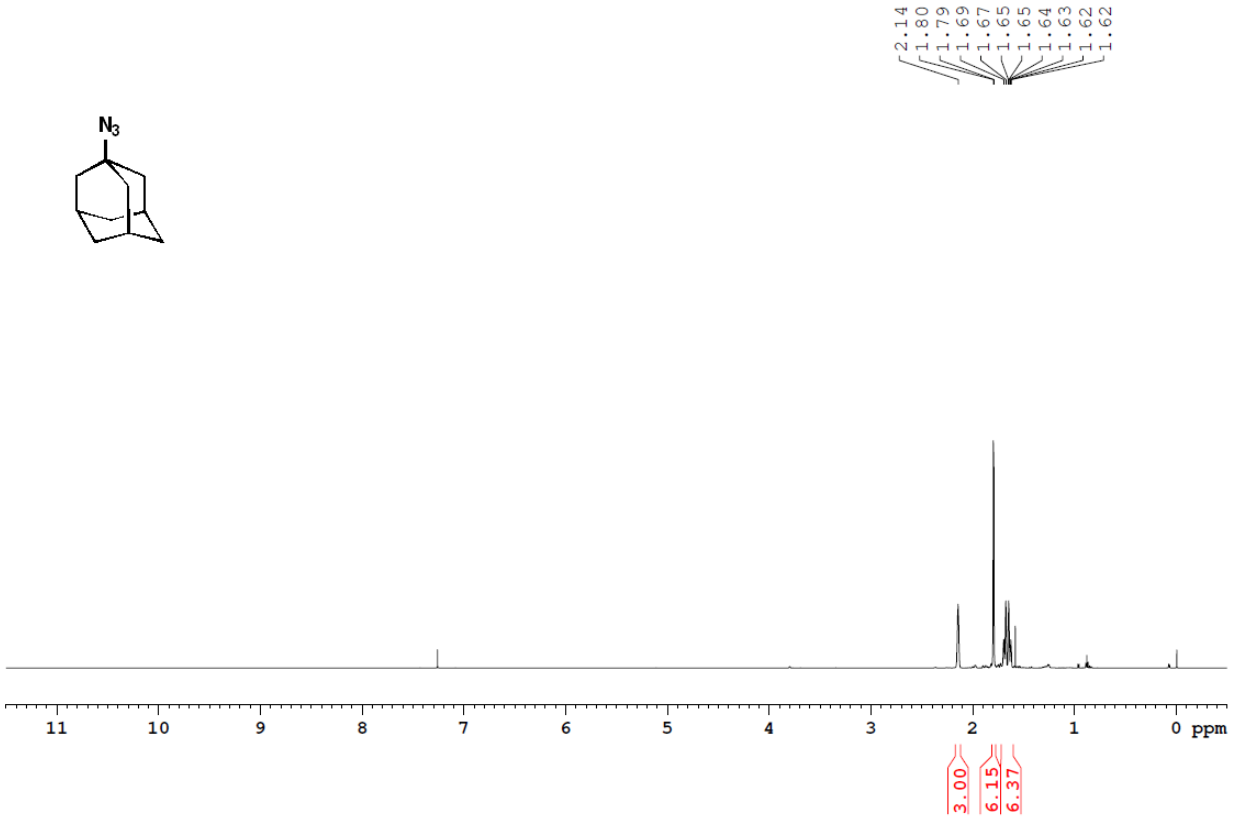
^{1}H NMR spectrum of **2c** (600 MHz, $CDCl_3$)



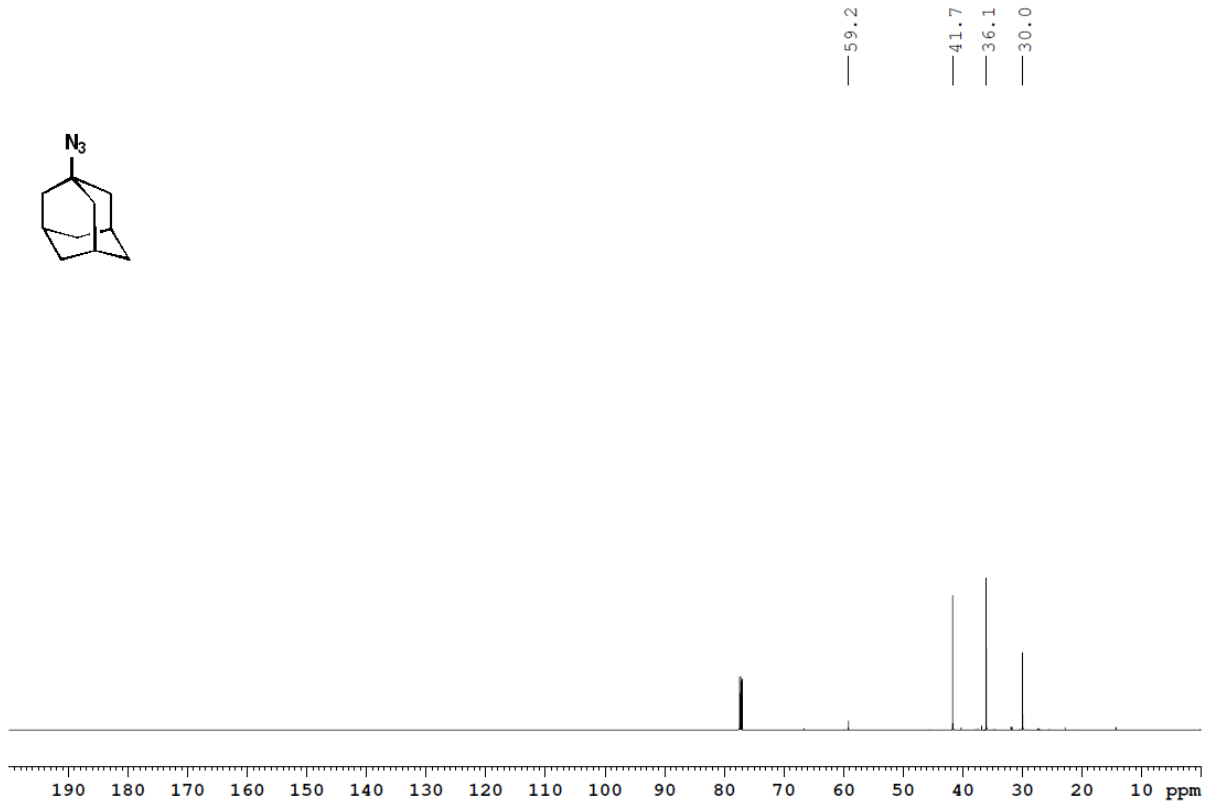
^{13}C NMR spectrum of **2c** (600 MHz, $CDCl_3$)



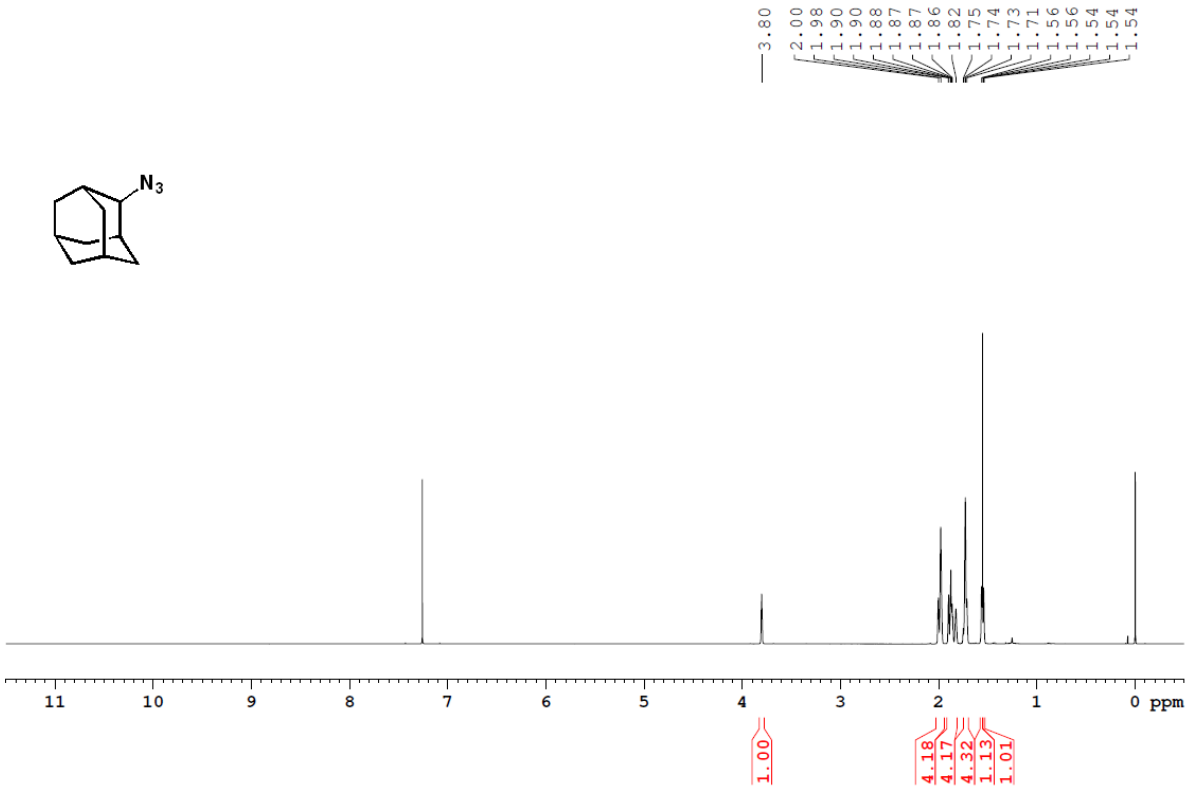
^{1}H NMR spectrum of **2d** (600 MHz, $CDCl_3$)



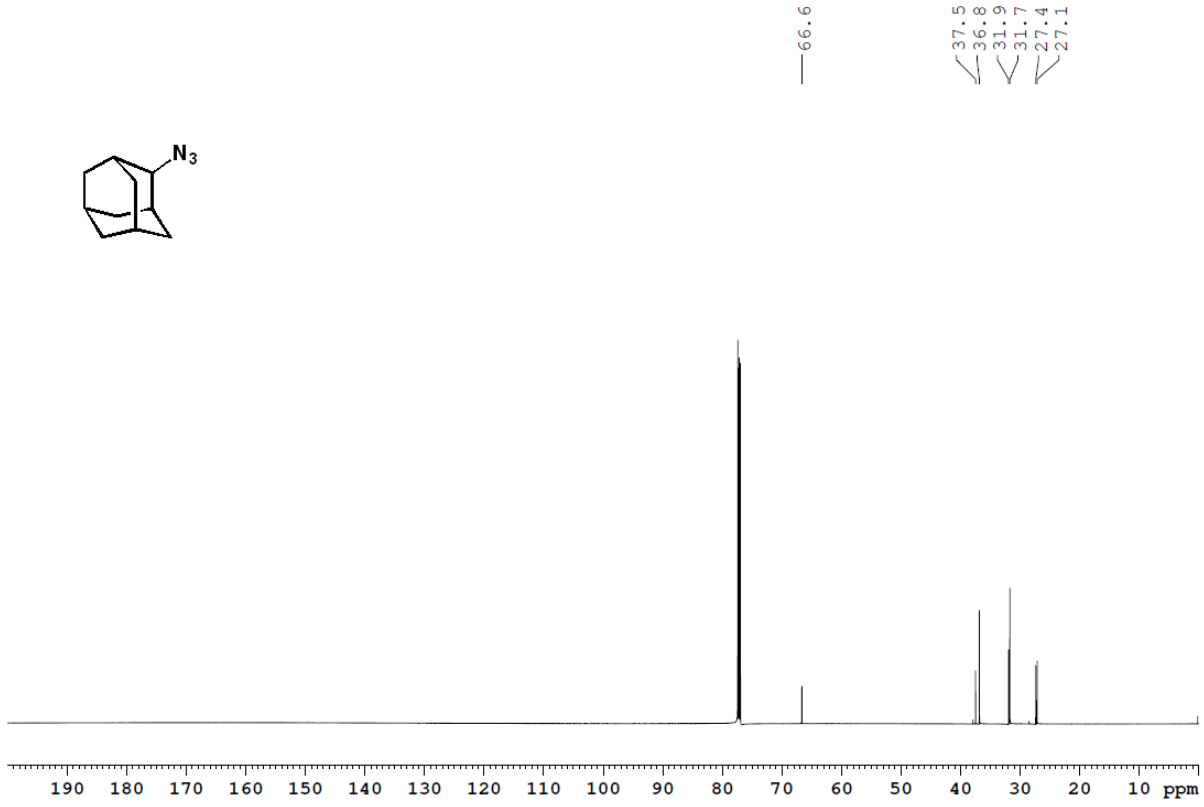
^{13}C NMR spectrum of **2d** (600 MHz, $CDCl_3$)



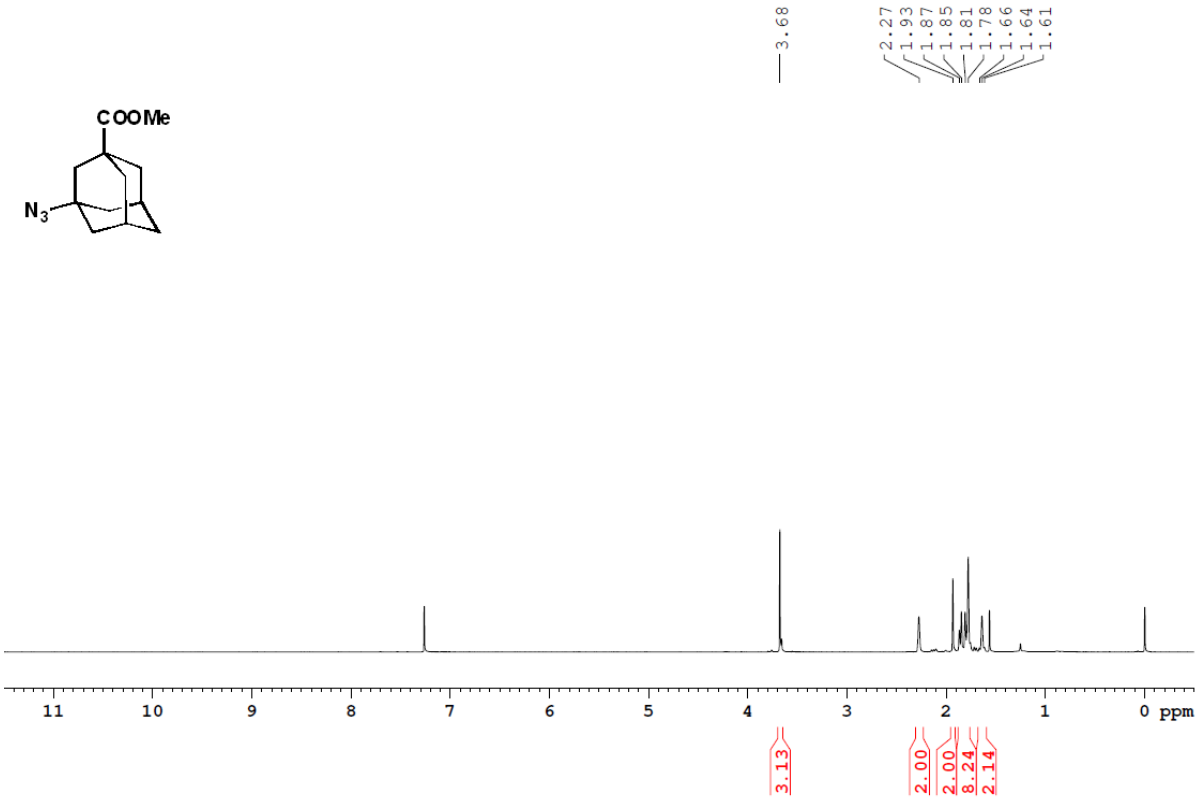
^{1}H NMR spectrum of **2d'** (600 MHz, $CDCl_3$)



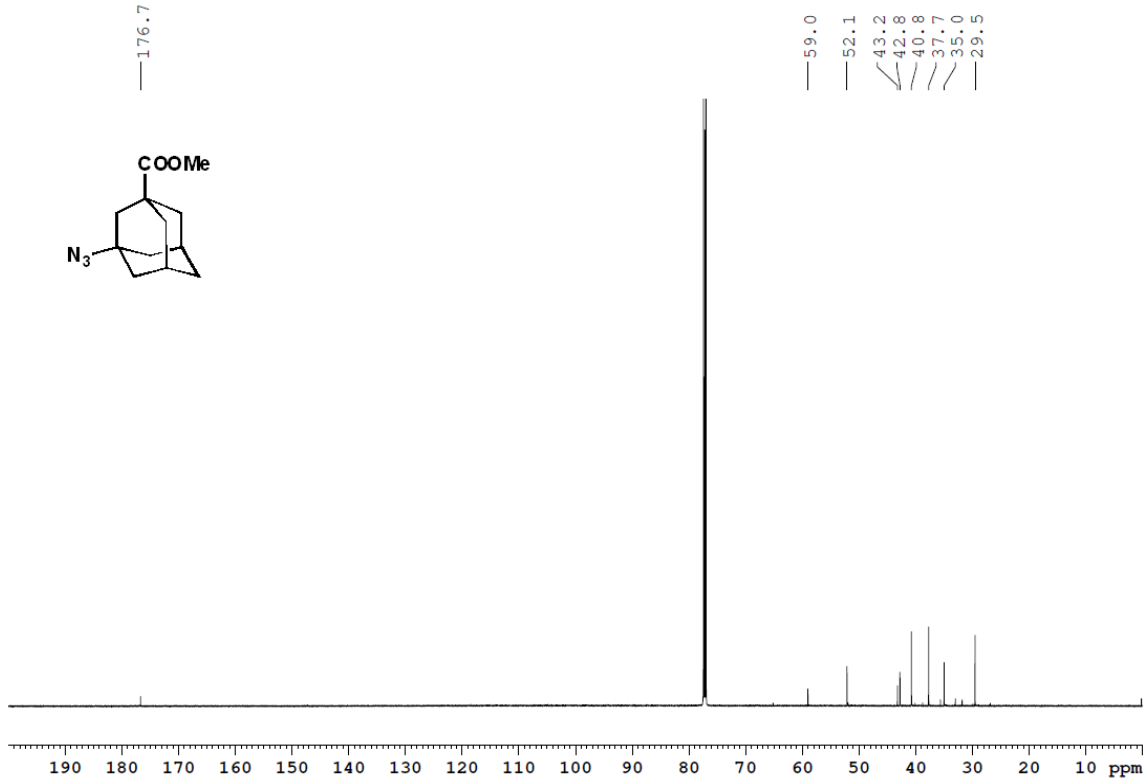
^{13}C NMR spectrum of **2d'** (600 MHz, $CDCl_3$)



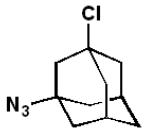
^{1}H NMR spectrum of **2e** (600 MHz, $CDCl_3$)



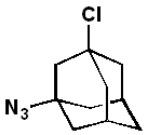
^{13}C NMR spectrum of **2e** (600 MHz, $CDCl_3$)



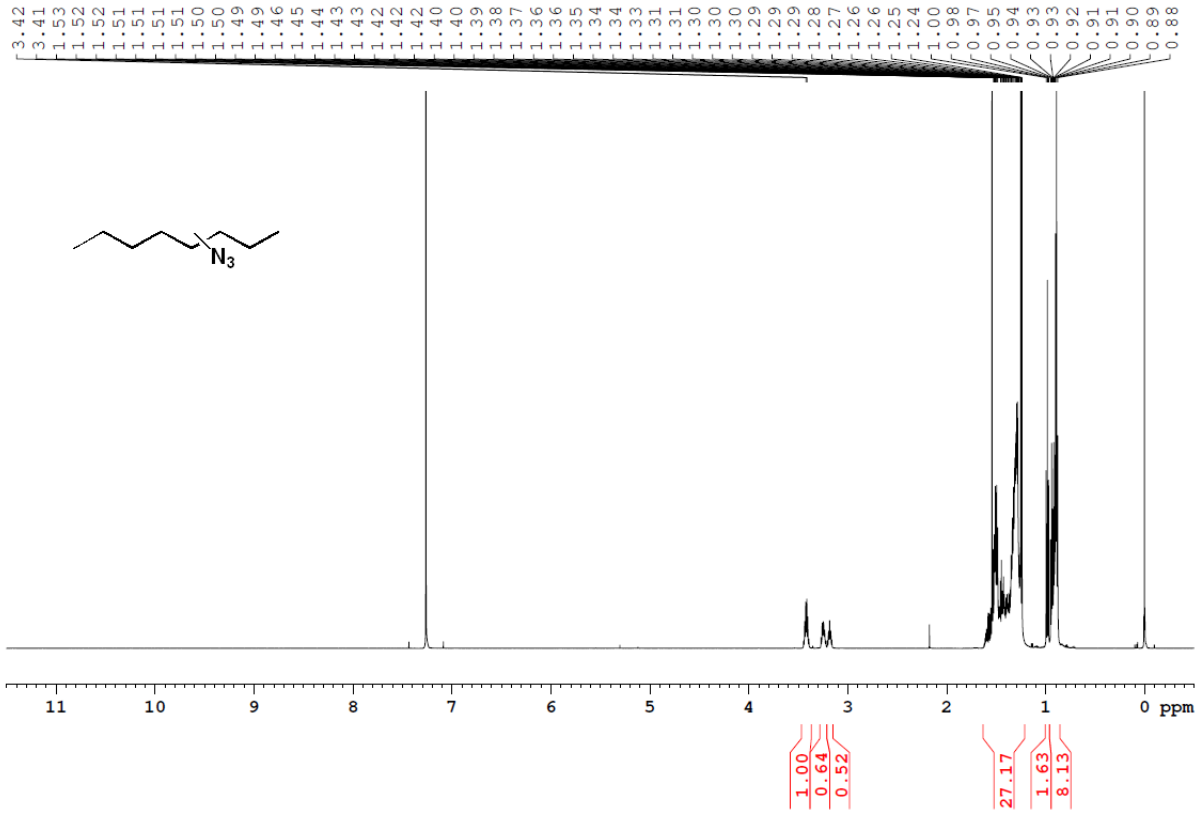
^{1}H NMR spectrum of **2f** (600 MHz, $CDCl_3$)



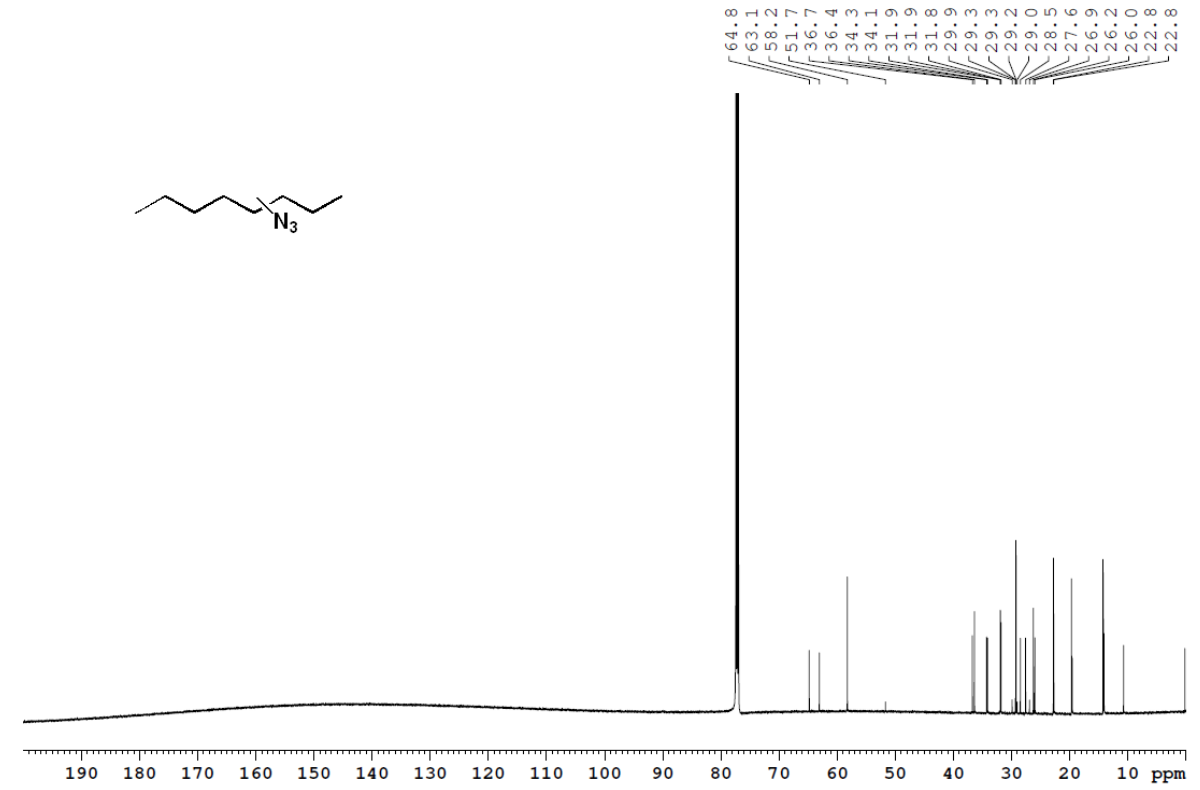
^{13}C NMR spectrum of **2f** (600 MHz, $CDCl_3$)



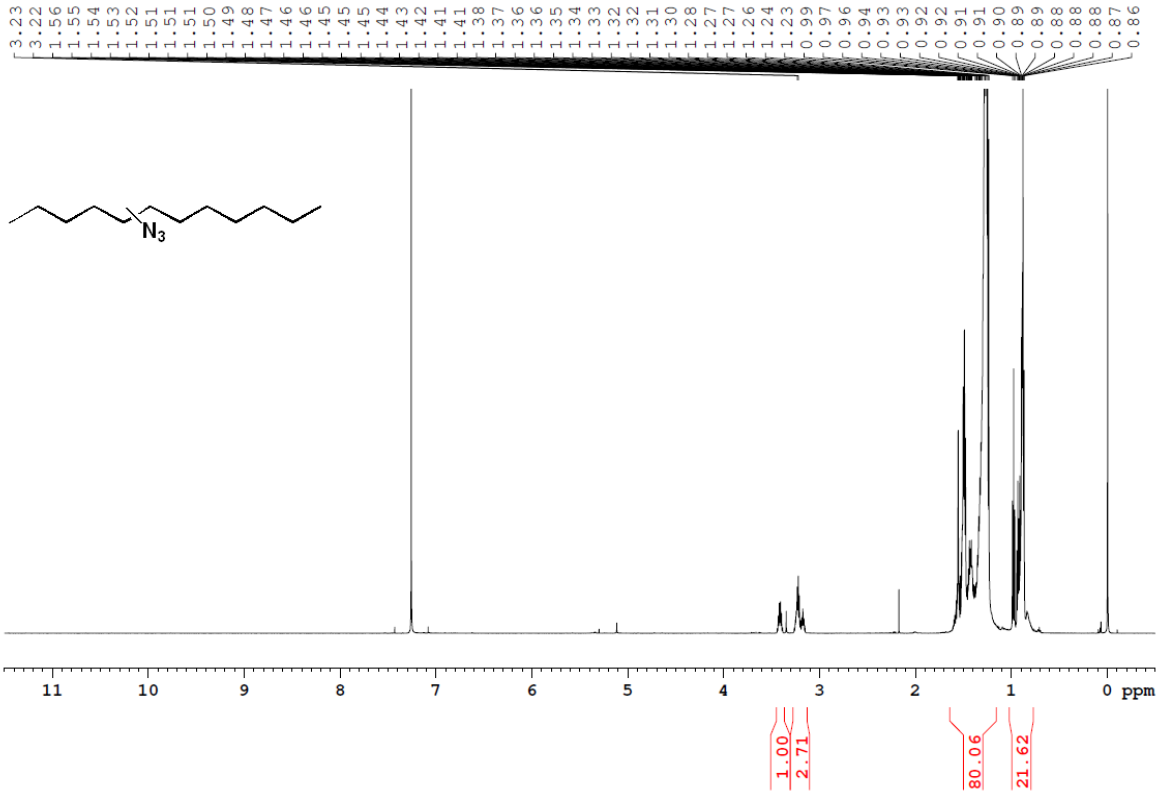
^{1}H NMR spectrum of **2g** isomeric mixture (600 MHz, $CDCl_3$)



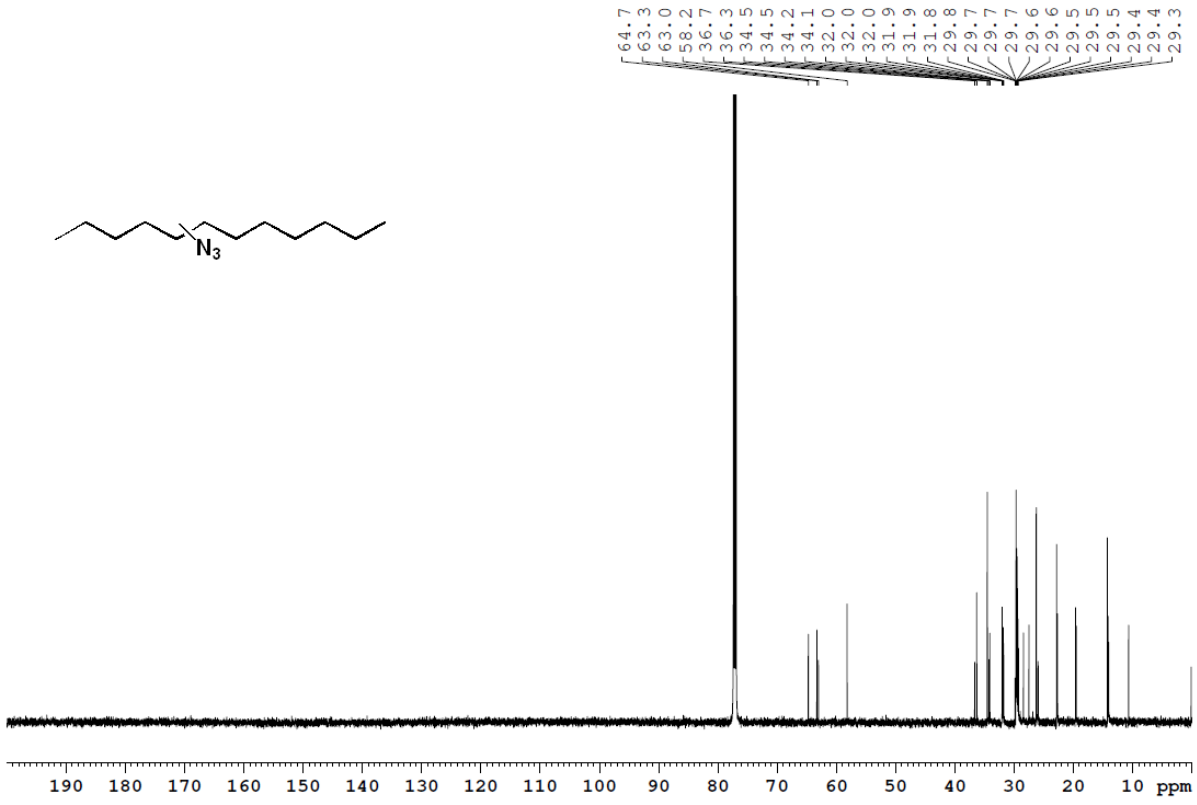
^{13}C NMR spectrum of **2g** isomeric mixture (600 MHz, $CDCl_3$)



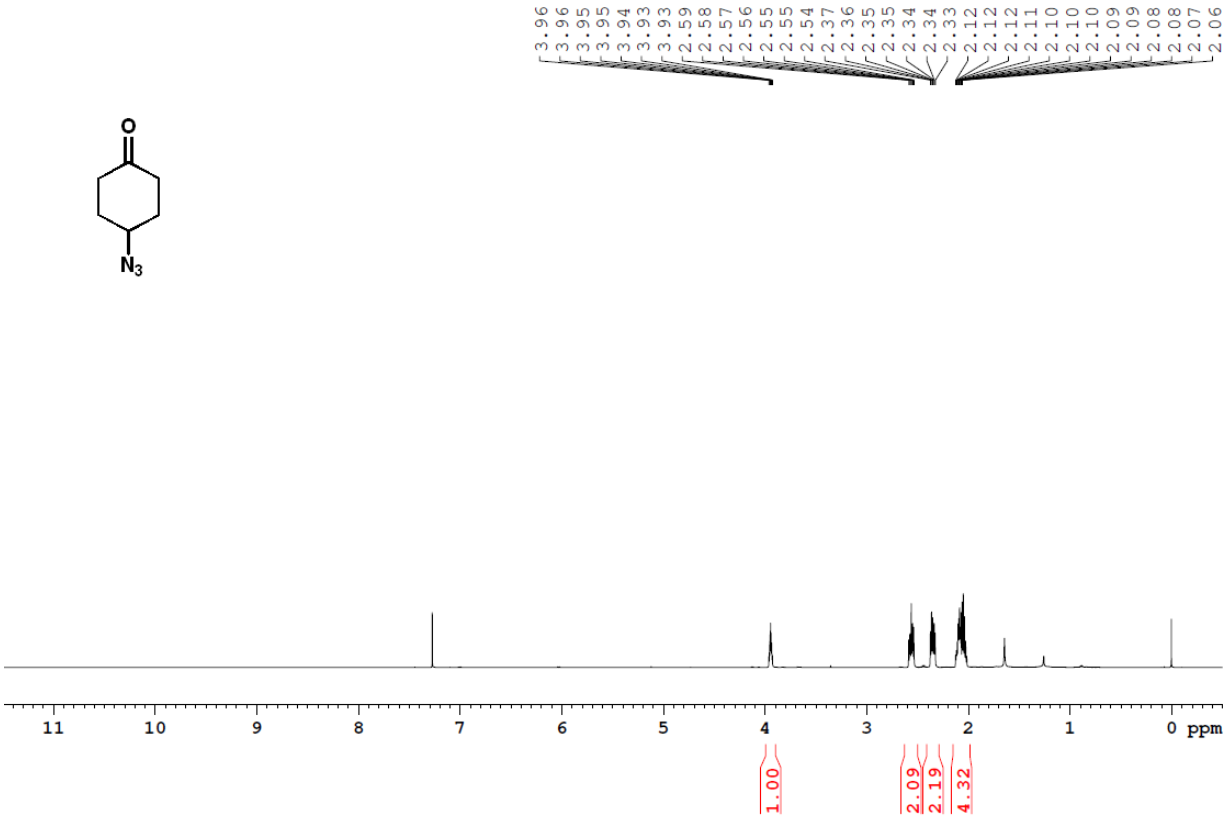
^{1}H NMR spectrum of **2h** isomeric mixture (600 MHz, $CDCl_3$)



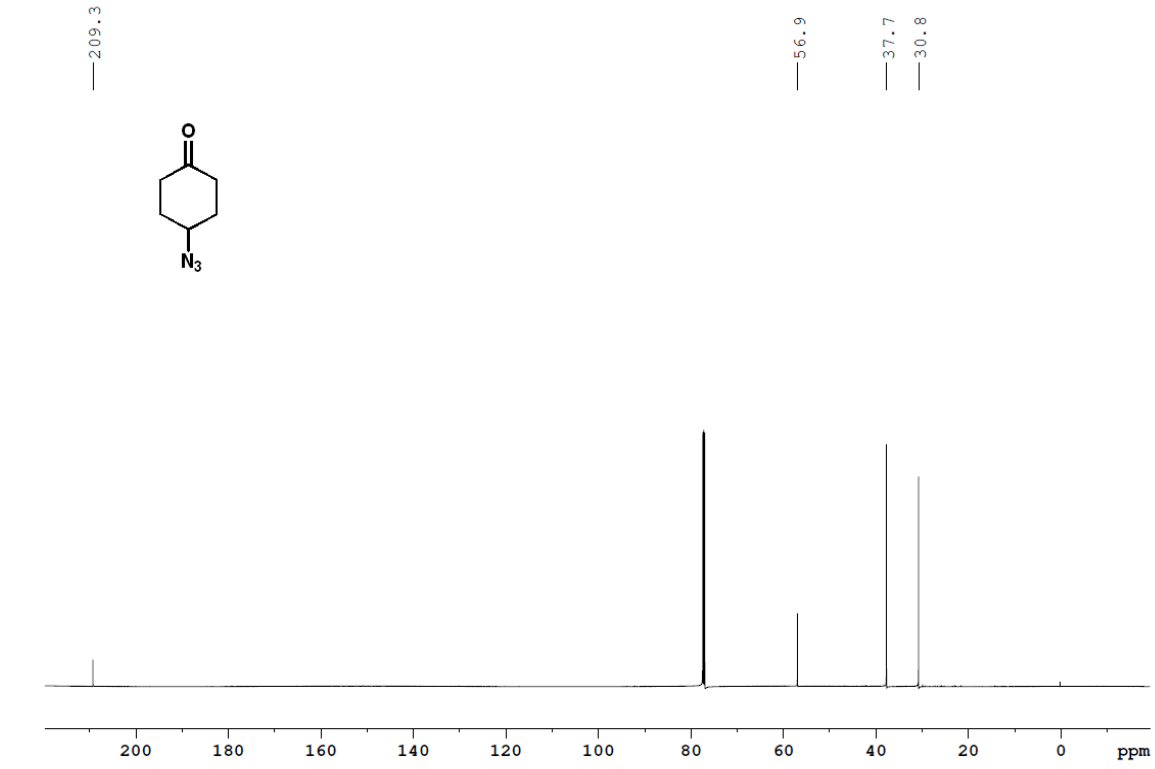
^{13}C NMR spectrum of **2h** isomeric mixture (600 MHz, $CDCl_3$)



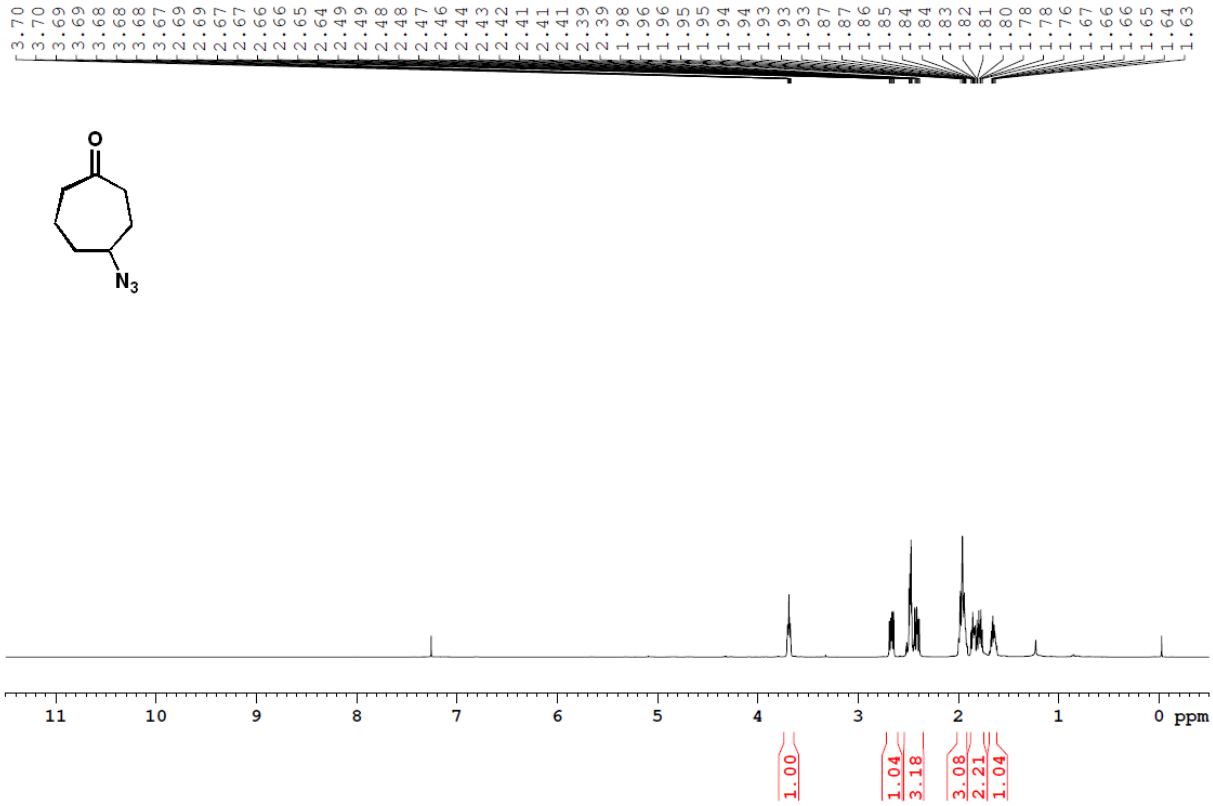
^{1}H NMR spectrum of **2j** (600 MHz, $CDCl_3$)



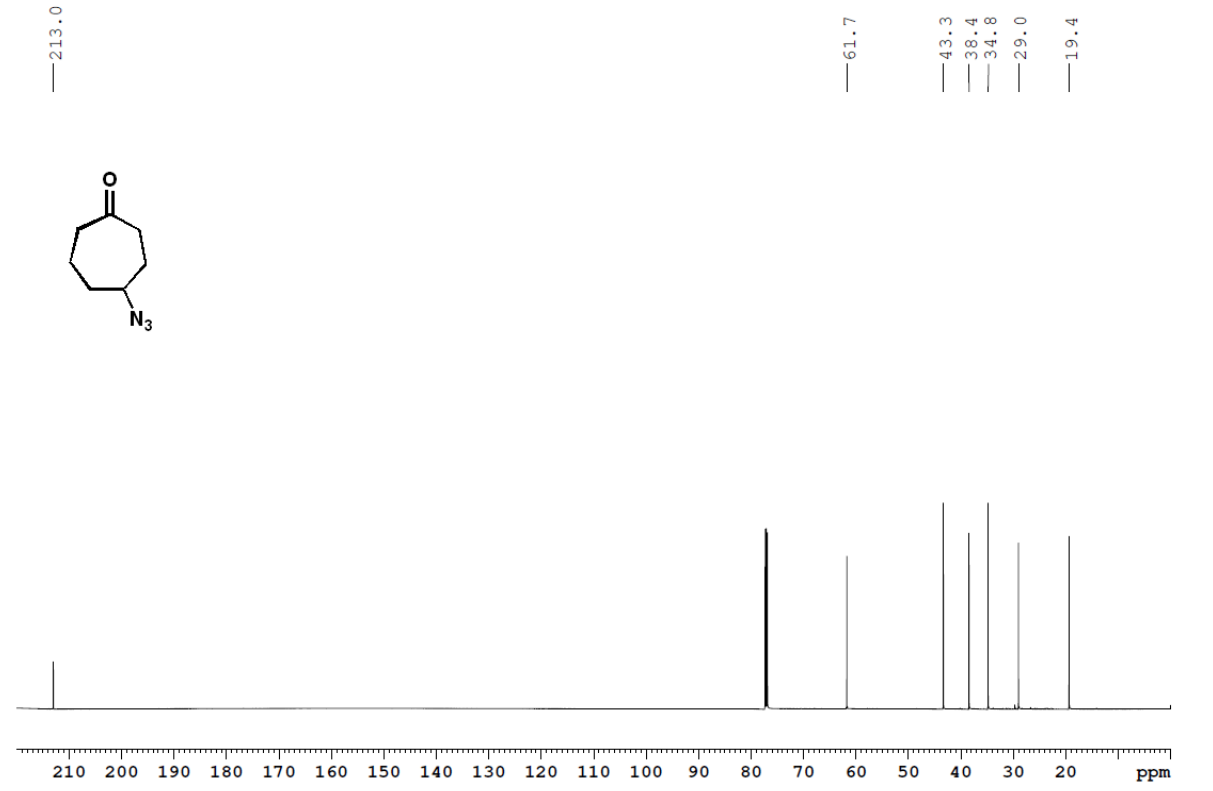
^{13}C NMR spectrum of **2j** (600 MHz, $CDCl_3$)



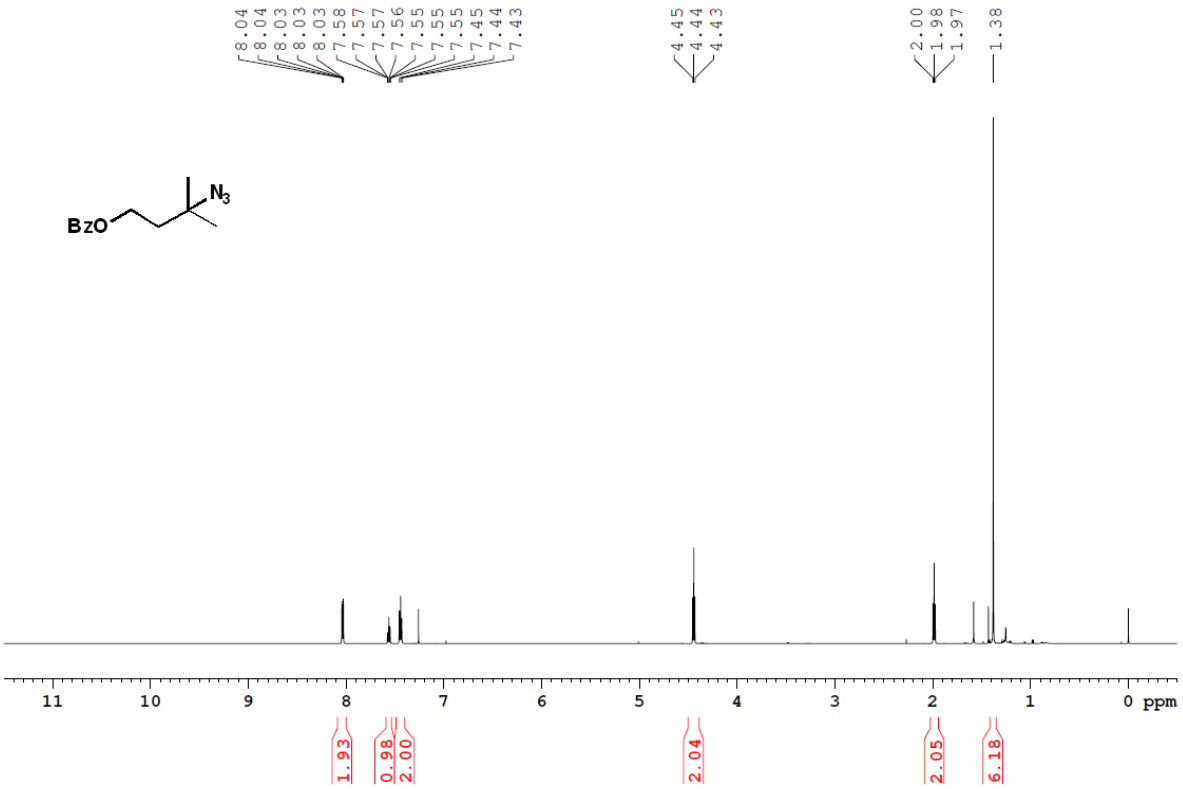
^{1}H NMR spectrum of **2k** (600 MHz, $CDCl_3$)



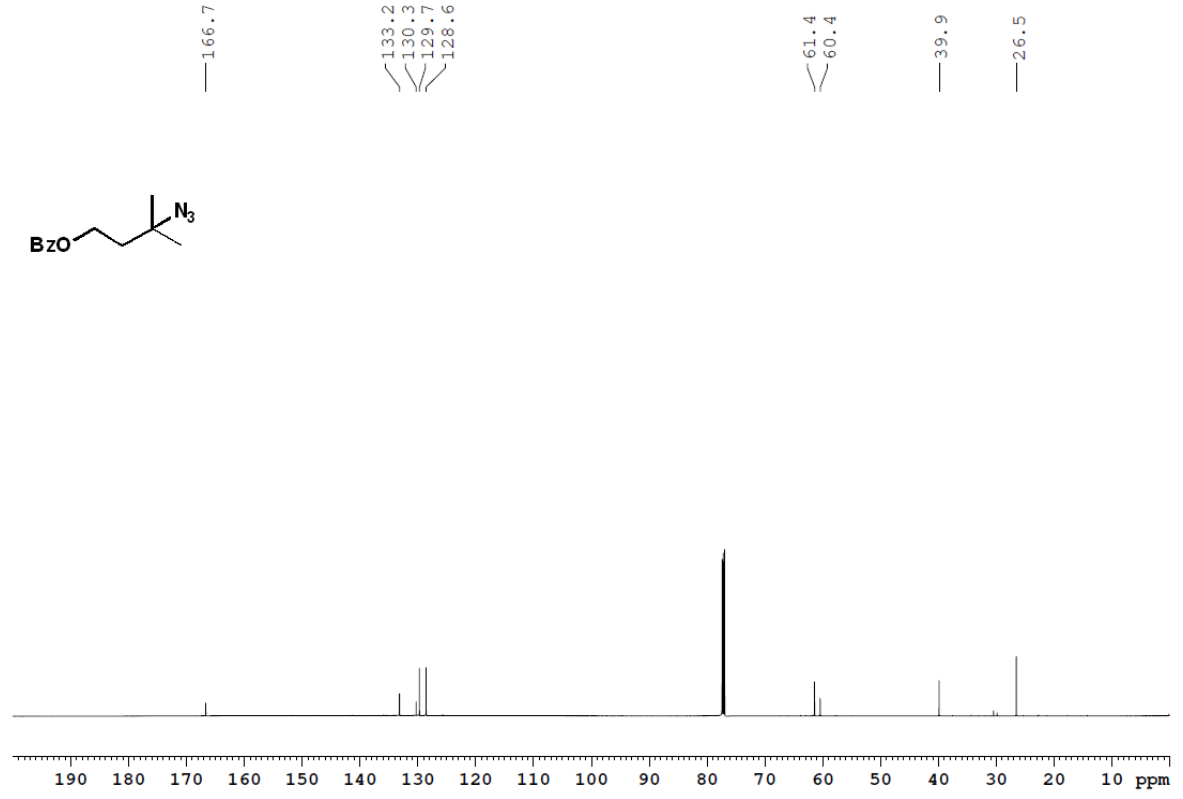
^{13}C NMR spectrum of **2k** (600 MHz, $CDCl_3$)



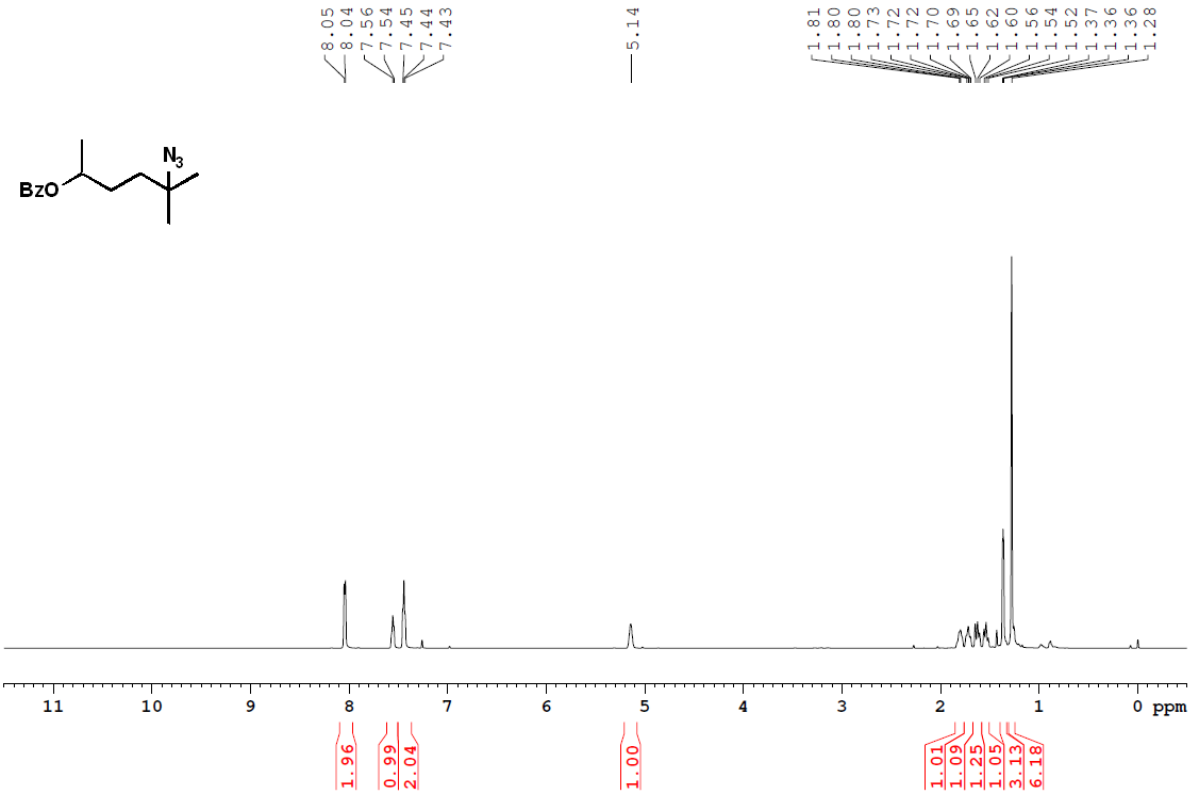
^{1}H NMR spectrum of **2l** (600 MHz, $CDCl_3$)



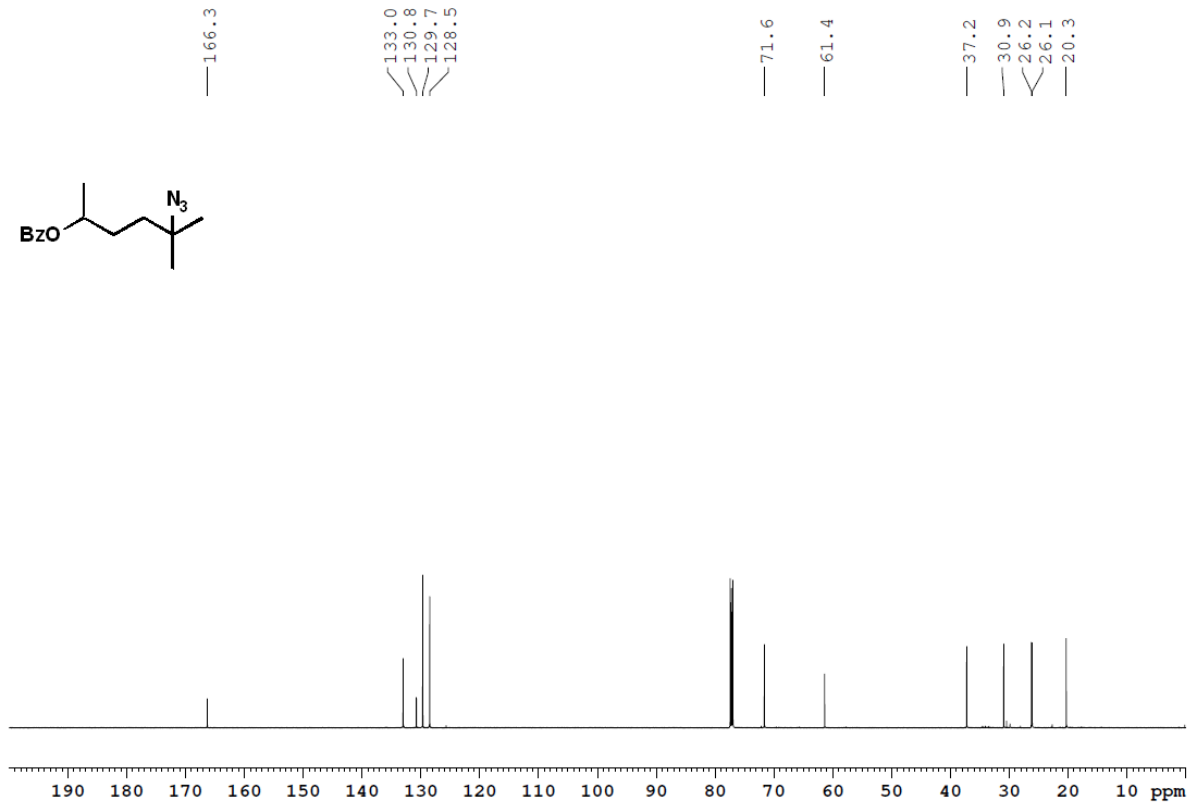
^{13}C NMR spectrum of **2l** (600 MHz, $CDCl_3$)



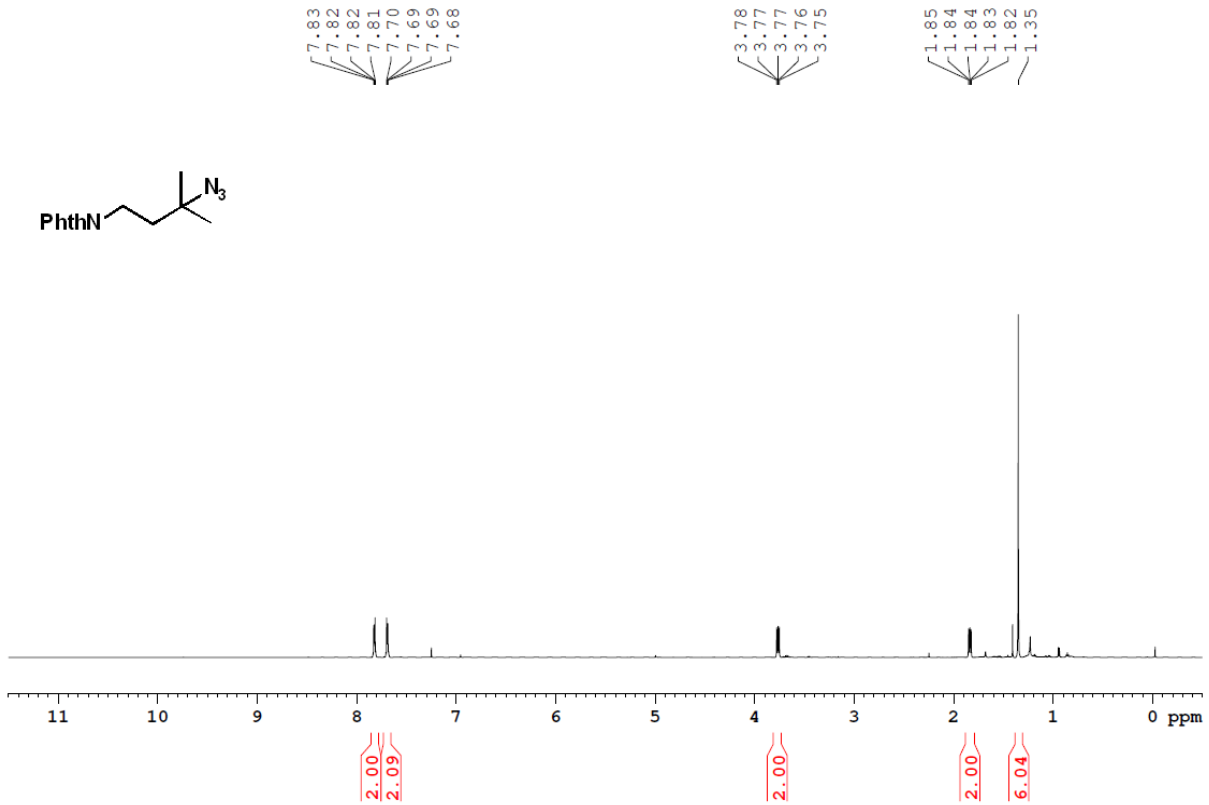
^{1}H NMR spectrum of **2m** (600 MHz, $CDCl_3$)



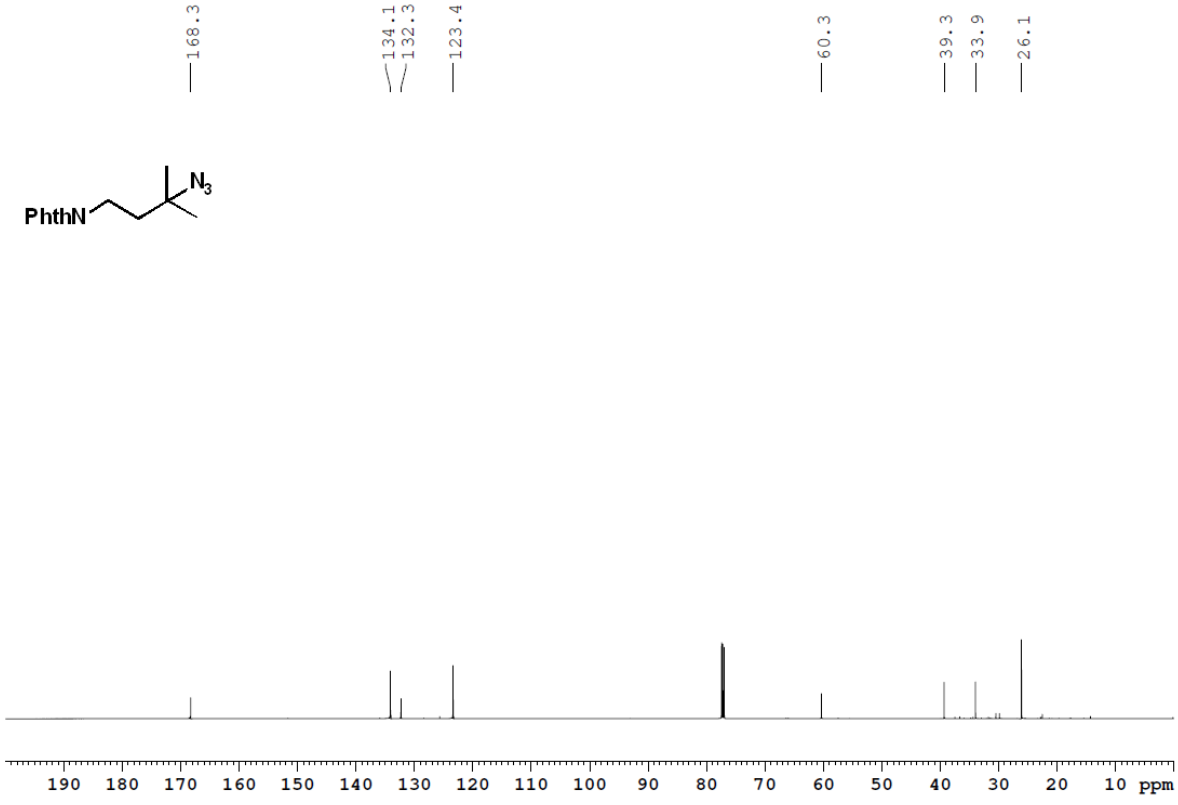
^{13}C NMR spectrum of **2m** (600 MHz, $CDCl_3$)



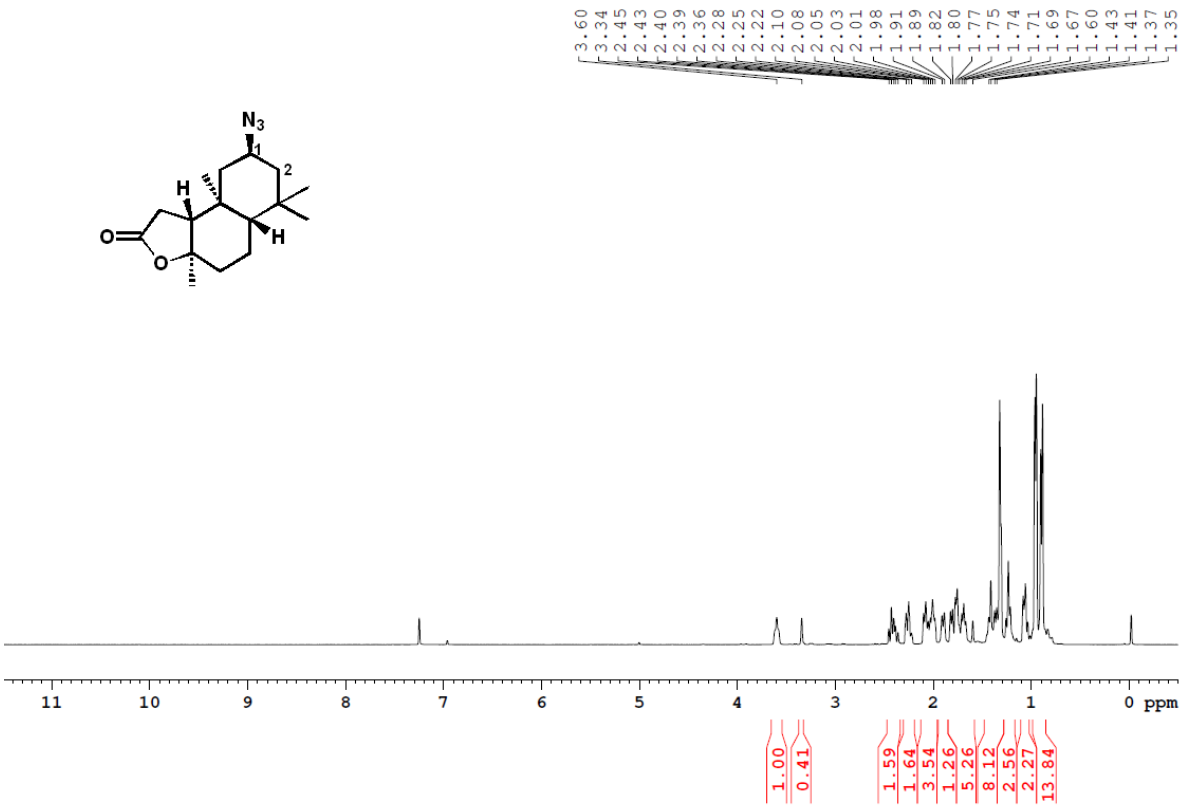
^{1}H NMR spectrum of **2n** (600 MHz, $CDCl_3$)



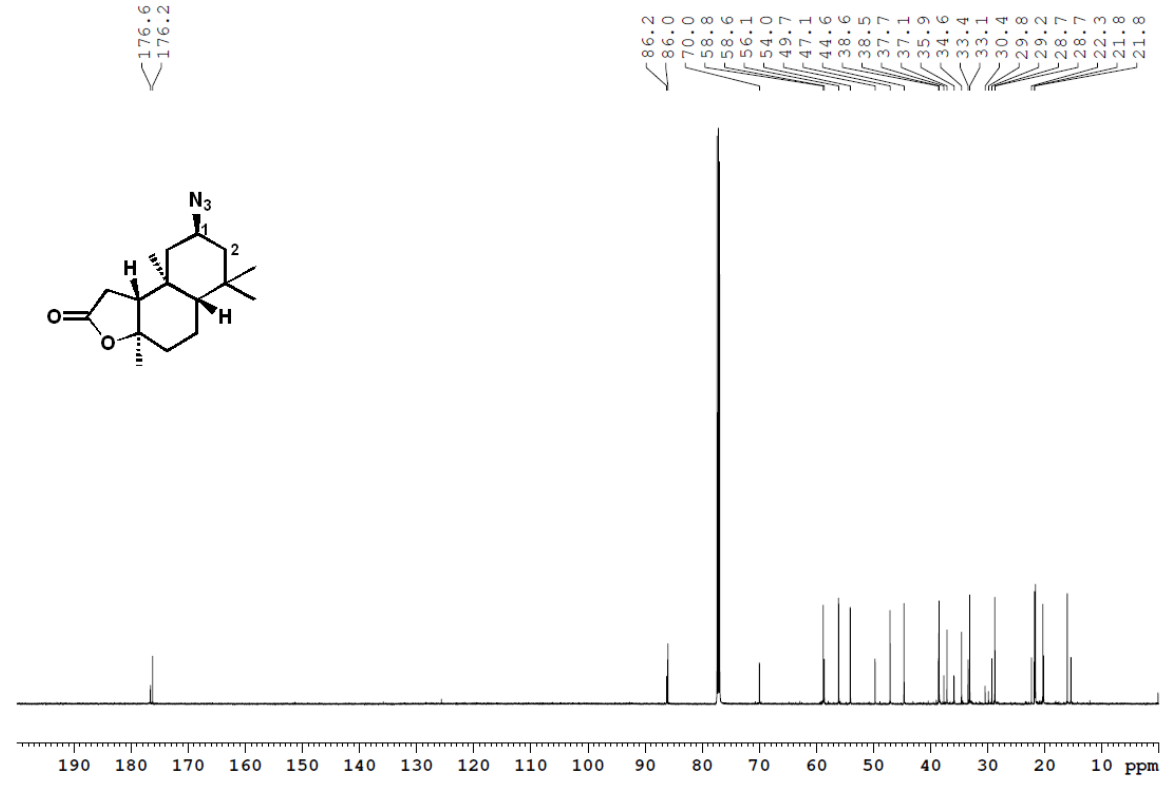
^{13}C NMR spectrum of **2n** (600 MHz, $CDCl_3$)



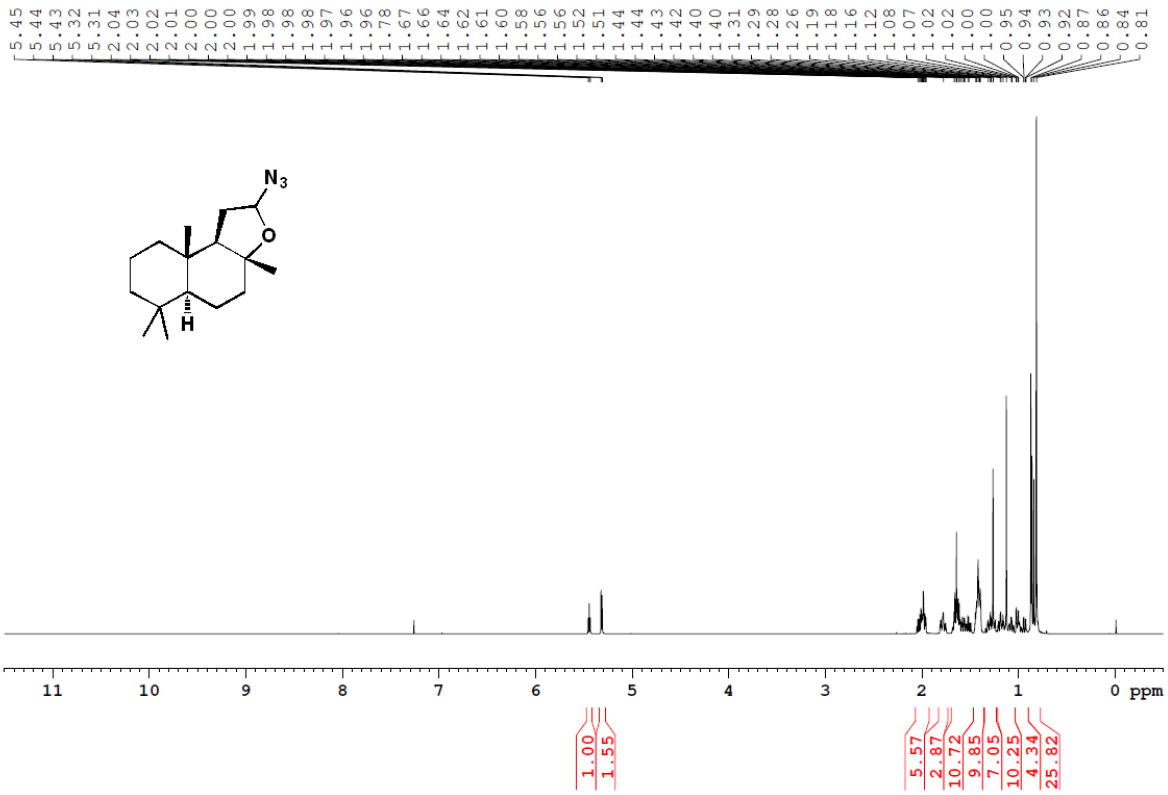
^{1}H NMR spectrum of **2o** (600 MHz, $CDCl_3$)



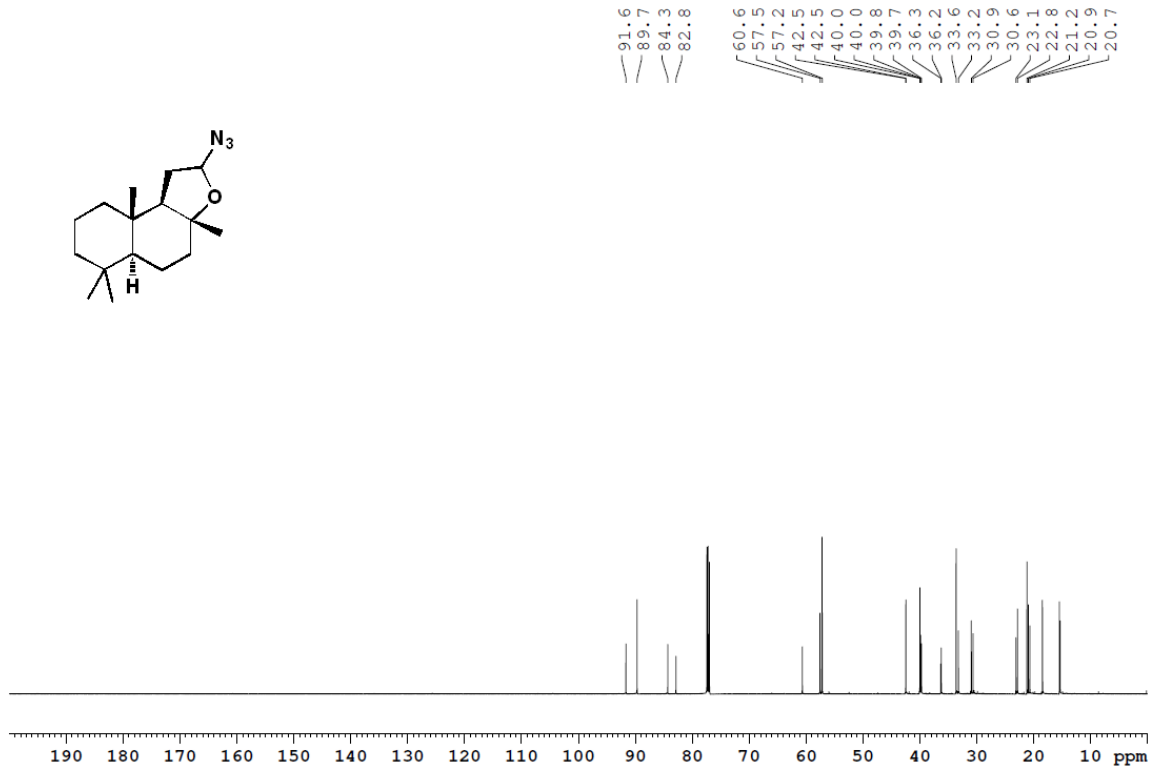
^{13}C NMR spectrum of **2o** (600 MHz, $CDCl_3$)



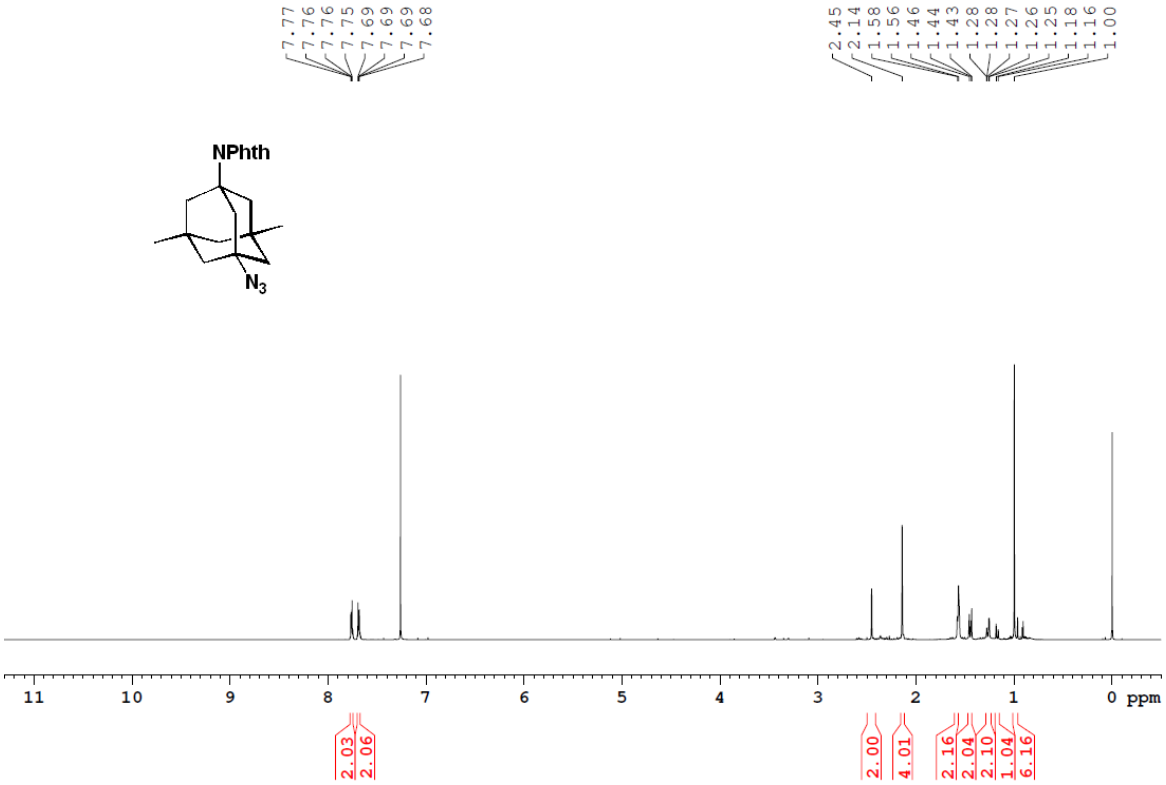
^{1}H NMR spectrum of **2p** (600 MHz, $CDCl_3$)



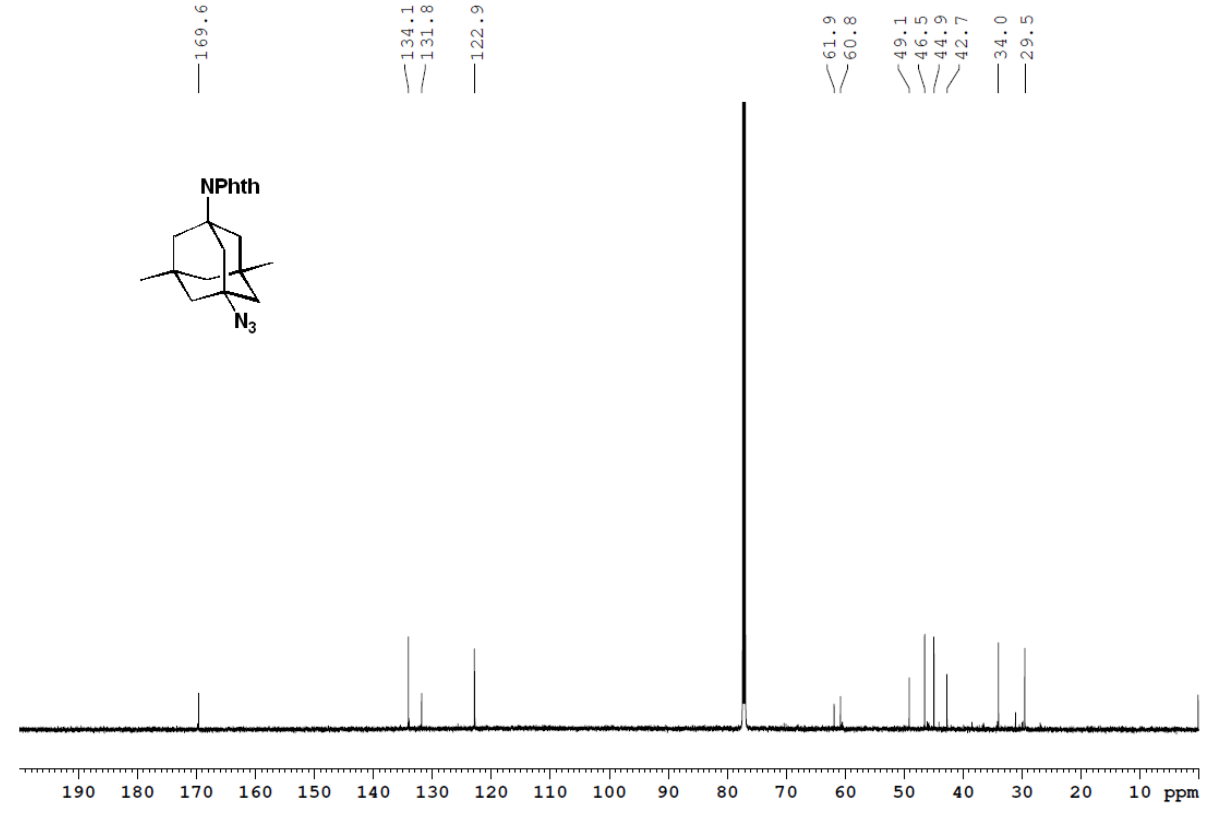
^{13}C NMR spectrum of **2p** (600 MHz, $CDCl_3$)



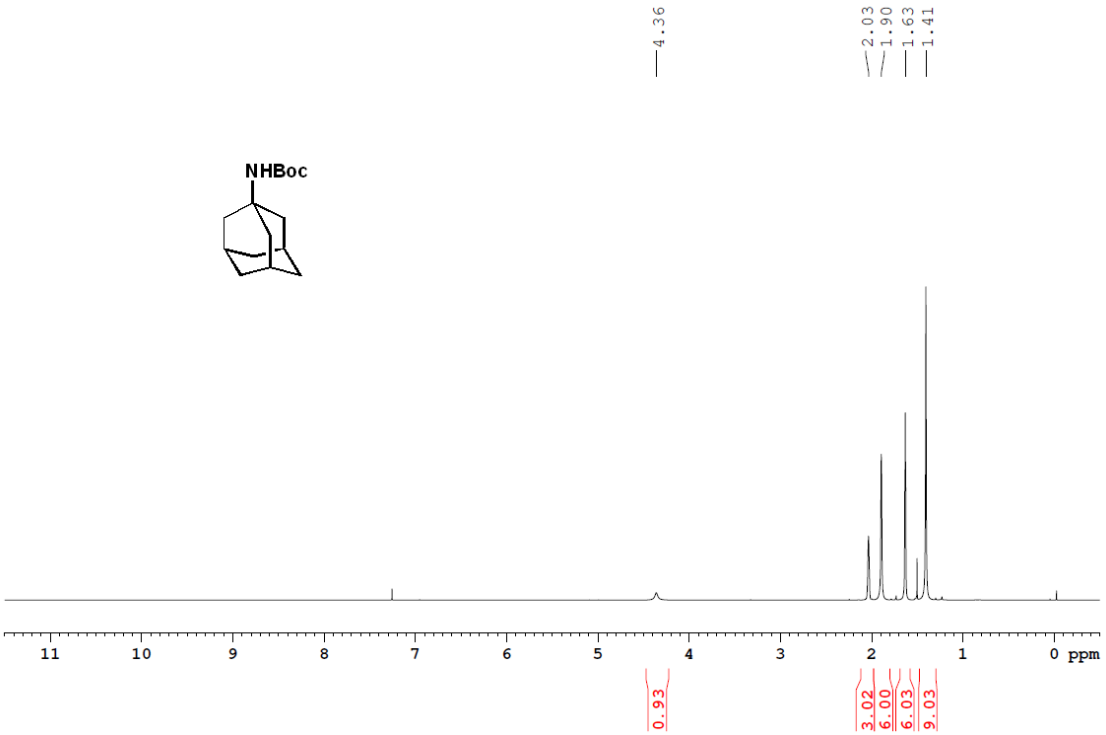
^{1}H NMR spectrum of **2q** (600 MHz, $CDCl_3$)



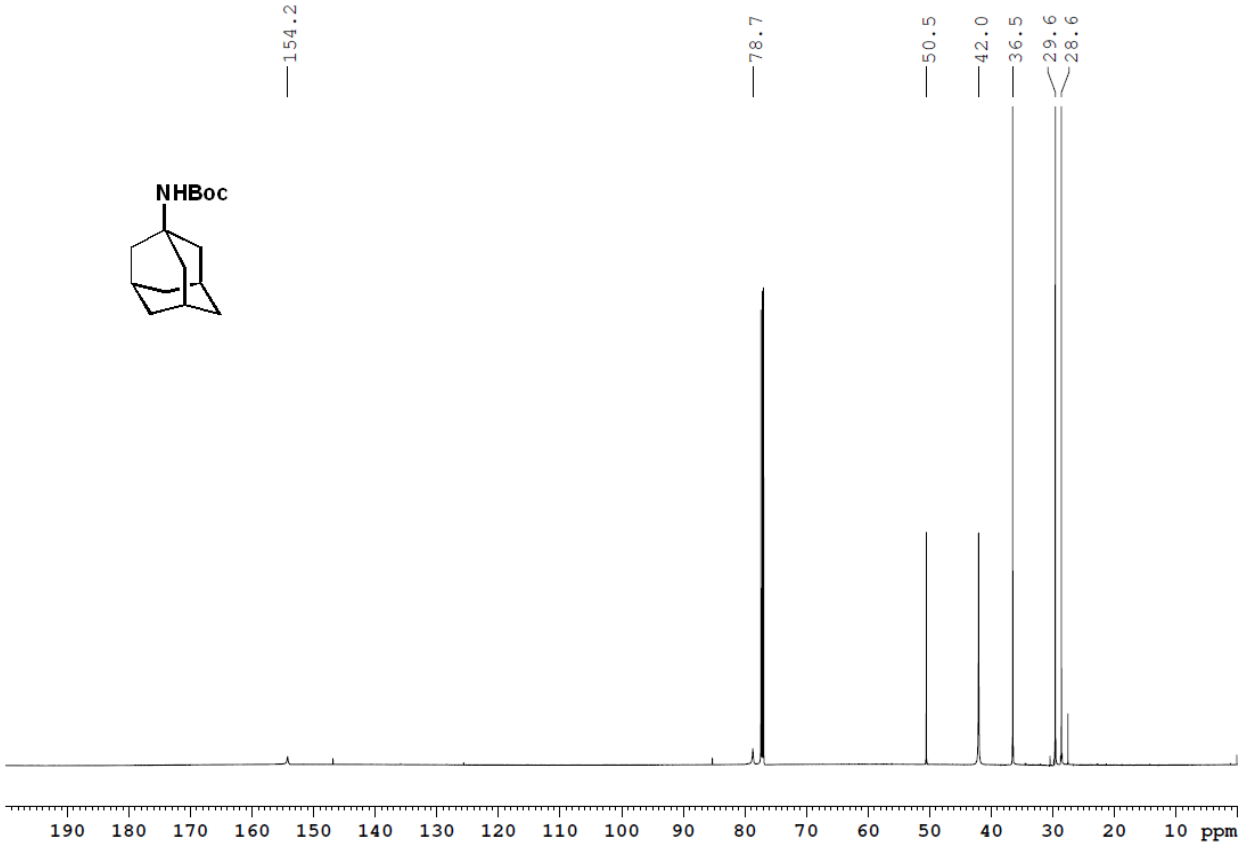
^{13}C NMR spectrum of **2q** (600 MHz, $CDCl_3$)



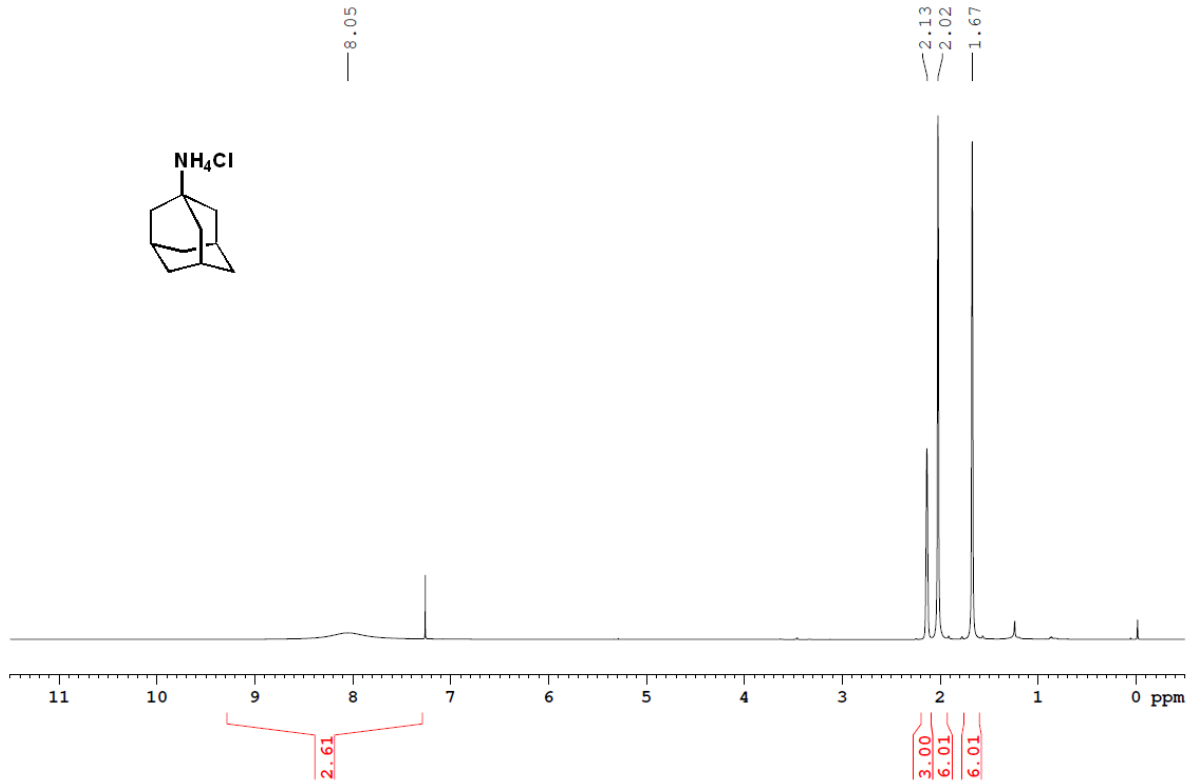
^{1}H NMR spectrum of **2r** (600 MHz, $CDCl_3$)



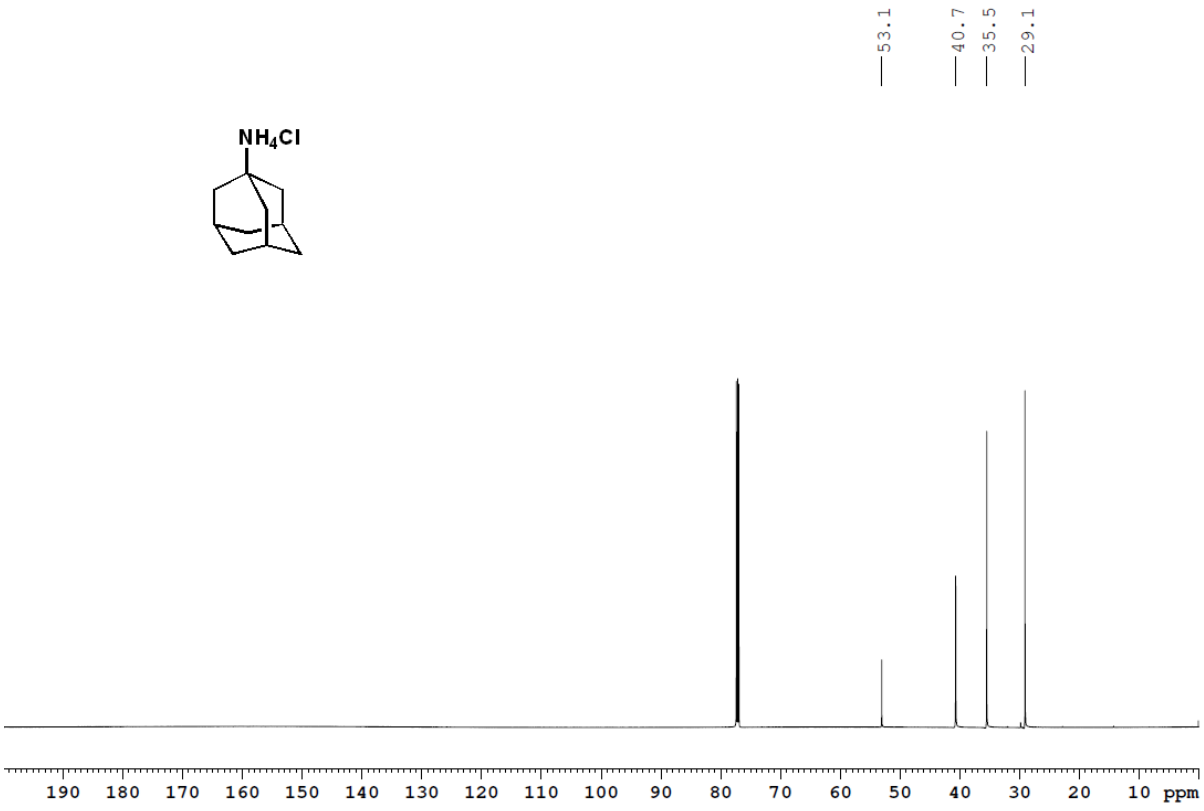
^{13}C NMR spectrum of **2r** (600 MHz, $CDCl_3$)



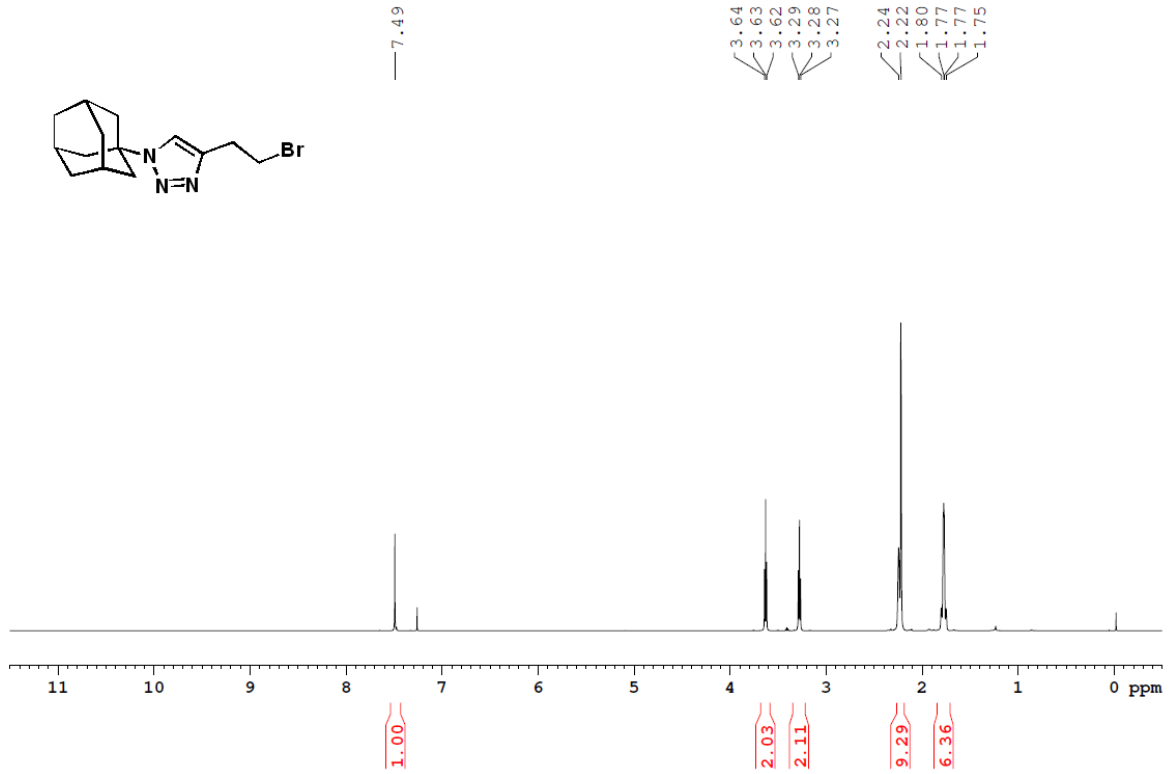
^{1}H NMR spectrum of **2s** (600 MHz, $CDCl_3$)



^{13}C NMR spectrum of **2s** (600 MHz, $CDCl_3$)



^{1}H NMR spectrum of **2t** (600 MHz, $CDCl_3$)



^{13}C NMR spectrum of **2t** (600 MHz, $CDCl_3$)