Electronic Supporting Information

A New Paradigm of Copper Oxide Nanoparticles Catalyzed Reactions: Synthesis of 1,2,3-Triazoles Through Oxidative Azide-Olefin Cycloaddition

D. Gangaprasad, J. Paul Raj, T. Kiranmye, S. Sagubar Sadik and J. Elangovan*

[a] *Department of Chemistry, B. S. Abdur Rahman University, Seethakathi Estate, Vandalur, Chennai-600048, India*

e-mail: elangoorganic@gmail.com

Table of contents

General Methods

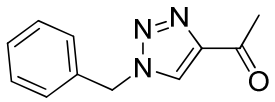
All melting points were taken on Guna melting point apparatus and are uncorrected. Mass spectra were recorded on a JEOL GC Mate using electron impact ionization (EI) techniques. NMR spectra were obtained on a Bruker Ascend TM 400 NMR spectrometer (^{1}H NMR: 400 MHz; ^{13}C NMR: 100 MHz). IR Spectra were recorded on JASCO 6300 spectrometer. Analytical TLC was carried out with Merck plates precoated with silica gel 60 F_{254} (0.25 mm thick). Column chromatography was performed either with Merck silica gel 60 (230–400 mesh) in common glass columns. All solvents were distilled before use. CuO nanoparticles (particle size <50 nm, surface area 29 m^2/g) was purchased from Aldrich. Methyl vinyl ketone (85%) was purchased from Avra synthesis Pvt. Ltd. Chalcones were synthesized as per Vogel's procedure.[1]

Experimental Procedures and Compounds Characterization Data

General Procedure for the Synthesis of 1,4-Disubstituted 1,2,3-Triazoles (3a-3j)

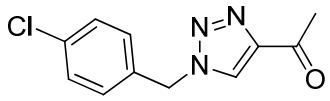
A mixture of azide (1.5 mmol), methyl vinyl ketone (4.5 mmol), CuO nanoparticles (20 mol%) was stirred in water (4 mL) at room temperature for 8h. The reaction mixture was extracted with ethyl acetate (2 x 30 mL) and the combined organic solutions were dried with Na_2SO_4. The solvent was evaporated, and the resulting crude product was purified by column chromatography.

1-(1-Benzyl-1*H*-1,2,3-triazol-4-yl)ethan-1-one (3a)[2, 3, 4]



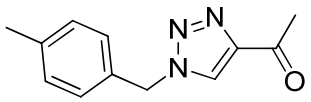
White solid; mp 91-92 °C (lit: 91-92 ºC); FT-IR (neat): υ = 1685 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.95 (s, 1H), 7.40-7.37 (m, 3H), 7.29-7.26 (m, 2H), 5.55 (s, 2H), 2.66 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 192.9, 148.5, 133.7, 129.4, 129.3, 128.4, 125.4, 54.6, 27.2.

1-{1-(4-Chlorobenzyl)-1*H*-1,2,3-triazol-4-yl}ethan-1-one (3b)[2]



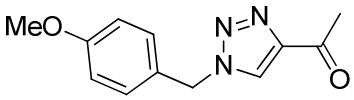
White solid; mp 110-112 °C (lit: 125-126 ºC); FT-IR (neat): υ = 1684 cm^{-1}; ^1H NMR (400MHz, $CDCl_3$): δ (ppm) 7.97 (s, 1H), 7.37-7.34 (m, 2H), 7.26-7.22 (m, 2H), 5.53 (s, 2H), 2.66 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 192.8, 148.6, 135.4, 132.3, 129.7, 129.6, 125.3, 53.8, 27.2.

1-{1-(4-Methylbenzyl)-1*H*-1,2,3-triazol-4-yl}ethan-1-one (3c)[2]



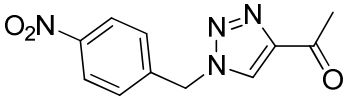
White solid; mp 112-113 °C (lit: 102-103 °C); FT-IR (neat): υ = 1687 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.92 (s, 1H), 7.18 (s, 4H), 5.50 (s, 2H), 2.65 (s, 3H), 2.34 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 192.9, 148.4, 139.3, 130.7, 130.1, 128.5, 125.2, 54.4, 27.2, 21.2.

1-{1-(4-Methoxybenzyl)-1*H*-1,2,3-triazol-4-yl}ethan-1-one (3d)[2]



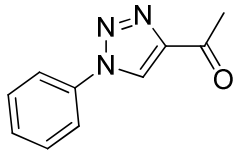
White solid; mp 110-112 °C (lit: 111-112 °C); FT-IR (neat): υ = 1682 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.91 (s, 1H), 7.24-7.22 (m, 2H), 6.90-6.88 (m, 2H), 5.48 (s, 2H), 3.79 (s, 3H), 2.65 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 193.0, 160.3, 148.4, 130.0, 125.7, 125.1, 114.8, 55.5, 54.1, 27.2.

1-{1-(4-Nitrobenzyl)-1*H*-1,2,3-triazol-4-yl}ethan-1-one (3e)[5]



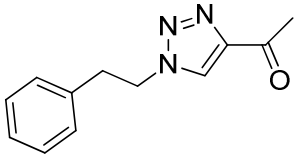
Yellow solid; mp 148-150 °C; FT-IR (neat): υ = 1683, 1517, 1346 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ (ppm) 8.26-8.24 (m, 2H), 8.06 (s, 1H), 7.45 (d, *J* = 12 Hz, 2H), 5.70 (s, 2H), 2.69 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 192.7, 148.9, 148.4, 140.8, 129.0, 125.6, 124.6, 53.5, 27.3.

1-{1-Phenyl-1*H*-1,2,3-triazol-4-yl}ethan-1-one (3f)[2]



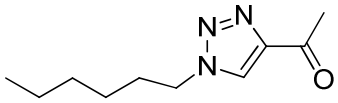
White solid; mp 108-109 °C (lit:108-109 °C); FT-IR (neat): υ = 1691 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 8.49 (s, 1H), 7.76-7.74 (m, 2H), 7.57-7.53 (m, 2H), 7.50-7.47 (m, 1H), 2.74 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 192.9, 148.6, 136.5, 130.1, 129.7, 123.4, 120.9, 27.4.

1-(1-Phenethyl-1*H*-1,2,3-triazol-4-yl)ethan-1-one (3g)[2]



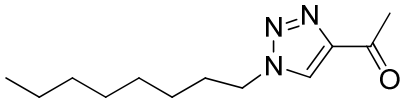
White solid; mp 110–112°C (lit: 114-115 °C); FT-IR (neat): n=1683 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.76 (s, 1H), 7.29-7.22 (m, 3H), 7.08-7.06 (m, 2H), 4.62 (t, *J* = 8.0 Hz, 2H), 3.21 (t, *J* = 8 Hz, 2 H), 2.64 (s, 3H); ^{13}C NMR (100 MHz $CDCl_3$): δ (ppm) 192.9, 147.9, 136.5, 129.0, 128.7, 127.5, 125.7, 52.0, 36.6, 27.2.

1-(1-Hexyl-1*H*-1,2,3-triazol-4-yl)ethan-1-one (3h)[2]



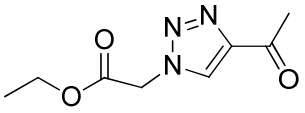
White solid; mp 63-64 °C (lit: 63-64 °C); FT-IR (neat): υ = 1687 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 8.03 (s, 1H), 4.39 (t, *J* = 8.0 Hz, 2H), 2.68 (s, 3H), 1.93-1.90 (m, 2H), 1.33-1.27 (m, 6H), 0.87 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 193.1, 148.2, 125.2, 50.8, 31.2, 30.2, 27.2, 26.1, 22.5, 14.0.

1-(1-Octyl-1*H*-1,2,3-triazol-4-yl)ethan-1-one (3i)



White solid; mp 64-66 °C; FT-IR (neat): υ = 1685 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ (ppm) 8.03 (s, 1H), 4.38 (t, *J* = 7.2 Hz, 2H), 2.67 (s, 3H), 1.91 (t, *J* = 6.8 Hz, 2H), 1.30-1.24 (m, 10H), 0.85 (t, *J* = 6.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 193.1, 148.2, 125.2, 50.8, 31.8, 30.2, 29.1, 29.0, 27.2, 26.5, 22.7, 14.1; HRMS(EI): *m/z* = 223.1682, calcd. for $C_{12}H_{21}N_3O$ (M^+): 223.1685.

Ethyl 2-(4-acetyl-1*H*-1,2,3-triazol-1-yl)acetate (3j)[3]

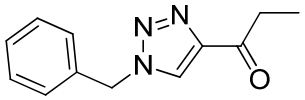


White solid; mp 68-70 °C (lit: 79-80 °C); FT-IR (neat): υ = 1748, 1685 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$):δ (ppm) 8.21 (s, 1H), 5.21 (s, 2H), 4.27 (q, *J* = 7.2 Hz, 2H), 2.68 (s, 3H), 1.29 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 192.7, 165.7, 148.5, 127.0, 62.9, 51.1, 27.3, 14.1; HRMS (EI): *m/z* = 197.0800, calcd. for $C_8H_{11}N_3O_3$ (M^+): 197.0800.

General Procedure for the Synthesis of 1,4-Disubstituted 1,2,3-Triazoles (5a)

A mixture of benzyl azide (1.5 mmol), ethyl vinyl ketone (1.5 mmol), CuO nanoparticles (20 mol%) was stirred in water (4 mL) at room temperature for 8h. The reaction mixture was extracted with ethyl acetate (2 x 30 mL) and the combined organic solutions were dried with Na_2SO_4. The solvent was evaporated and the resulting crude product was purified by column chromatography.

1-(1-Benzyl-1*H*-1,2,3-triazol-4-yl)propan-1-one (5a)[2,6]

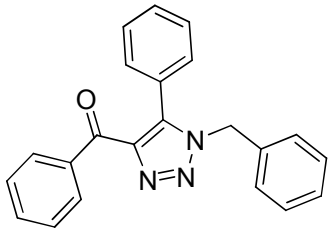


White solid; mp 116-117 °C (lit: 116–117 °C); FT-IR (neat): υ = 1688 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.95 (s, 1H), 7.41-7.37 (m, 3H), 7.29-7.27 (m, 2H), 5.55 (s, 2H), 3.12 (q, *J* = 7.20 Hz, 2H), 1.19 (t, *J* = 7.60 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 195.9, 148.1, 133.8, 129.4, 129.2, 128.4, 125.3, 54.5, 32.9, 7.8.

General Procedure for the Synthesis of 1,4,5-Trisubstituted 1,2,3-Triazoles (5b-5k)

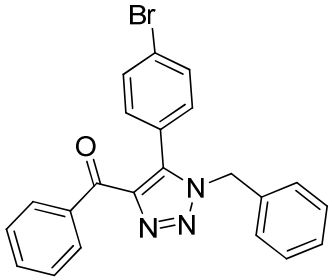
A mixture of azide (2.25 mmol), olefin (1.5 mmol), and CuO nanoparticles (20 mol%) was heated at 90 °C in DMF (6 mL) for 12h. The reaction mixture was extracted with ethyl acetate (2 x 30 mL) and the combined organic solutions were dried with Na_2SO_4. The solvent was evaporated, and the resulting crude product was purified by column chromatography.

(1-Benzyl-5-phenyl-1*H*-1,2,3-triazol-4-yl)(phenyl)methanone (5b)[2,7]



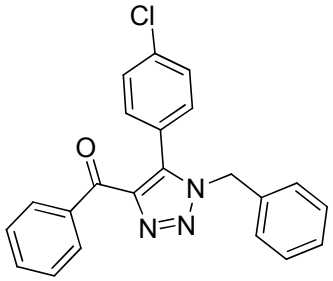
White solid; mp 112-114 °C (lit:114–115 °C); FT-IR (neat): υ = 1647 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ (ppm) 8.28 (d, *J* = 7.6 Hz, 2H), 7.59-7.55 (m, 1H), 7.51-7.42 (m, 6H), 7.30-7.25 (m, 4H), 7.07-7.05 (m, 2H), 5.47 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 186.4, 143.9, 141.9, 137.2, 134.8, 133.1, 130.8, 130.1, 129.9, 129.0, 128.8, 128.6, 128.3, 127.7, 126.4, 52.1.

{1-Benzyl-5-(4-bromophenyl)-1*H*-1,2,3-triazol-4-yl}(phenyl)methanone(5c)



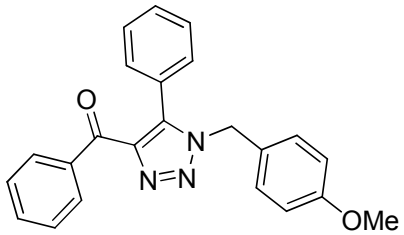
Yellow solid; mp 128 °C; FT-IR (neat): υ = 1644 cm^{-1};^1H NMR (400 MHz, $CDCl_3$): δ (ppm) 8.22 (d, *J* = 7.6 Hz, 2H), 7.53-7.49 (m, 3H), 7.42-7.39 (m, 2H), 7.23-7.22 (m, 3H), 7.05-6.98 (m, 4H), 5.38 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 186.3, 144.1, 140.9, 137.0, 134.6,133.3, 132.1, 131.5, 130.8, 129.1, 128.7, 128.4, 127.7, 125.4, 124.8, 52.3; HRMS(EI): *m/z* = 417.0470, calcd. for $C_{22}H_{18}BrN_3O$ (M^+): 417.0477.

{1-Benzyl-5-(4-chlorophenyl)-1*H*-1,2,3-triazol-4-yl}(phenyl)methanone (5d)[2,8]



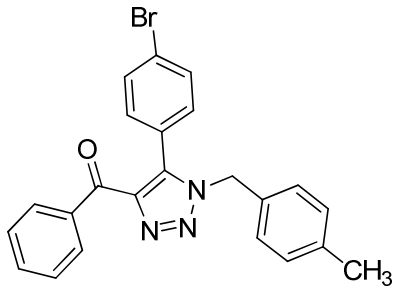
Yellow solid; mp 142-145 °C (lit: 130-131 °C); FT-IR (neat): υ = 1647 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 8.30-8.29 (m, 2H), 7.61- 7.41 (m, 5H), 7.32-7.08 (m, 5H), 7.08-7.06 (m, 2H),5.47 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 186.3, 144.1, 140.9, 137.0, 136.6, 134.6, 133.3, 131.3, 130.8, 129.2, 129.1, 128.8, 128.4, 127.7, 124.9, 52.3.

{1-(4-Methoxybenzyl)-5-phenyl-1*H*-1,2,3-triazol-4-yl}(phenyl)methanone (5e)[2]



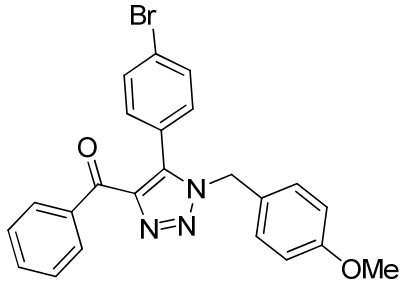
White solid; mp 124-128 °C (lit:153–155 °C); FT-IR (neat): υ = 1645 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 8.28-8.26 (m, 2H), 7.58-7.54 (m, 1H), 7.50-7.44 (m, 5H), 7.28-7.26 (m, 2H), 7.01-6.98 (m, 2H), 6.80-6.78 (m, 2H), 5.40 (s, 2H), 3.77 (s, 3H); ^{13}C NMR (100 MHZ, $CDCl_3$): δ (ppm) 186.5, 159.7, 143.9, 141.7, 137.2, 133.1, 130.8, 130.1, 129.9, 129.3, 128.8, 128.3, 126.8, 126.6, 114.3, 55.4, 51.7.

{5-(4-Bromophenyl)-1-(4-methylbenzyl)-1*H*-1,2,3-triazol-4-yl}(phenyl)methanone (5f)



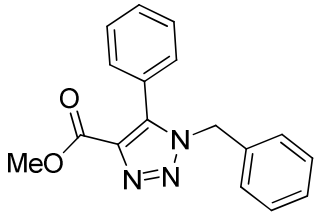
Yellow solid; mp 132 °C; FT-IR (neat): υ = 1640 cm^{-1};^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 8.30-8.28 (m, 2H), 7.59-7.57 (m, 3H), 7.50-7.46 (m, 2H), 7.15-7.09 (m, 4H), 6.97-6.95 (m, 2H), 5.42 (s, 2H), 2.32 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 186.2, 143.9, 140.7, 138.5, 137.0,133.2, 132.0, 131.5, 131.4, 130.7, 129.6, 128.3, 127.6, 125.4, 124.7, 52.0, 21.2; HRMS(EI): *m/z* = 431.0628, calcd. for $C_{22}H_{18}BrN_3O$ (M^+): 431.0633.

{5-(4-Bromophenyl)-1-(4-methoxybenzyl)-1*H*-1,2,3-triazol-4-yl}(phenyl)methanone (5g)



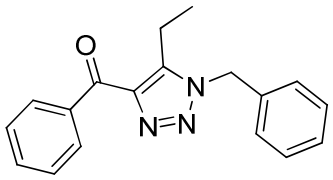
Yellow solid; mp 116 °C; FT-IR (neat): υ = 1647 cm^{-1};^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 8.22-8.21 (m, 2H), 7.53-7.49 (m, 3H), 7.42-7.38 (m, 2H), 7.07-7.05 (m, 2H), 6.94-6.92 (m, 2H), 6.74-6.72 (m, 2H), 5.32 (s, 2H), 3.71 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 186.2, 159.8, 140.0, 140.6, 136.9,133.2, 132.0, 131.5, 130.7, 129.2, 128.3, 126.5, 125.4, 124.7, 114.3, 55.3, 51.8; HRMS(EI): *m/z* = 447.0576, calcd. for $C_{22}H_{18}BrN_3O$ (M^+): 447.0582.

Methyl 1-benzyl-5-phenyl-1*H*-1,2,3-triazol-4-carboxylate (5h)[9]



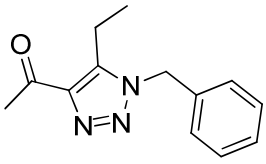
Yellow solid; mp 82-83 °C; FT-IR (neat): υ = 1727 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.50-7.42 (m, 5H), 7.27-6.98 (m, 5H), 5.43 (s, 2H), 3.84 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 161.4, 141.5, 136.9, 134.6, 130.3, 129.8, 128.9, 128.7, 128.5, 127.6, 125.8, 52.3, 52.1.

(1-Benzyl-5-ethyl-1*H*-1,2,3-triazol-4-yl)(phenyl)methanone (5i)



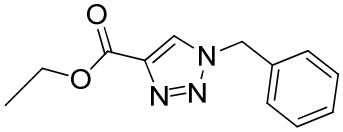
Yellowish semi solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 8.25-8.23 (m, 2H), 7.53-7.48 (m, 1H), 7.43-7.40 (m, 2H), 7.27-7.23 (m, 3H), 7.14 (m, 2H), 5.46 (s, 2H), 2.14 (quint, *J* = 7.6 Hz, 2H), 0.95 (t, *J* = 7.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 185.5, 143.6, 142.4, 136.7, 134.1, 131.8, 129.6, 127.9, 127.4, 127.2, 126.6, 50.8, 20.6, 15.1.

1-(1-benzyl-5-ethyl-1*H*-1,2,3-triazol-4-yl)ethanone (5j)[10]



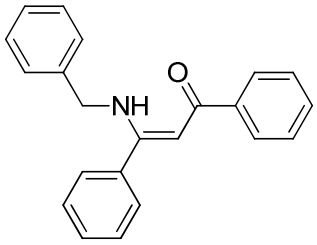
Yellow oil; ^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) 7.32-7.30 (m, 3H), 7.16-7.15 (m, 2H), 5.47 (s, 2H), 2.67 (s, 3H), 2.19 (quint, *J* = 6.0 Hz, 2H), 1.01 (t, *J* = 6.0 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) 193.0, 137.7, 135.1, 128.9, 128.4, 128.0, 127.6, 51.8, 27.8, 15.8, 13.4.

Ethyl-1-benzyl-1H-1,2,3-triazole-4-carboxylate (5k)[11]



White solid; mp 82-86 °C; ^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) 7.97 (s, 1H), 7.40-7.38 (m, 3H), 7.30-7.27 (m, 2H), 5.58 (s, 2H), 4.40 (q, *J* = 6.0 Hz, 2H), 1.39 (t, *J* = 6.0 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) 160.7, 140.7, 133.8, 129.3, 129.2, 128.3, 127.3, 61.3, 54.5, 14.3.

(*Z*)-3-(benzylamino)-1,3-diphenylprop-2-en-1-one (6b)[12]



White solid; mp 102–103 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 11.71 (s, 1H), 7.90 (dd, *J* = 1.2 Hz, 2H), 7.45–7.37 (m, 8H), 7.33-7.30 (m, 2H), 7.27–7.22 (m, 3H), 5.84 (s, 1H), 4.42 (d, *J* = 6.4 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 188.7, 166.8, 140.2, 138.4, 135.5, 130.8, 129.6, 128.8, 128.6, 128.2, 127.8, 127.4, 127.1, 126.9, 93.9, 48.5.

References

1 B. Furniss, J. Hannaford, P. W. G. Smith and A. R. Tatchell**,** *Vogel's Textbook of Practical Organic Chemistry*; 5th Edition, 1997, 1034.

2 D. Janreddy, V. Kavala, C.-W. Kuo, W.-C. Chen, C. Ramesh, K. Trimurtulu, T.-S. Kuo, M.-L. Chen, C.-H. He and C.-F. Yao, *Adv. Synth. Catal*., 2013, **355**, 2229 -2240.

3 D. R. Roque, J. L. Neill, J. W. Antoon and E. P. Stevens, *Synthesis*, 2005, 2497-2502.

4 S. Ceylan, T. Klande, C. Vogt, C. Friese and A. Kirschning, *Synlett*, 2010, 2009-2013.

5 F. Lazreg, A. M. Z. Slawin, and C. S. J. Cazin, *Organometallics*, 2012, **31**, 7969-7975.

6 S. Hwang, H. Bae, S. Kim and S. Kim, *Tetrahedron*, 2012, **68**, 1460-1465.

7 Y. Chen, Y. Liu, J. L. Petersen and X. Shi, *Chem. Commun.,*2008, 3254-3256.

8 N. Singh, S. K. Pandey and R. P. Tripathi, *Carbohydr. Res.,* 2010, **345**, 1641-1648.

9 Xiaohan Ye and Xiaodong Shi, *Org. Lett.*, 2014, **16**, 4448–4451.

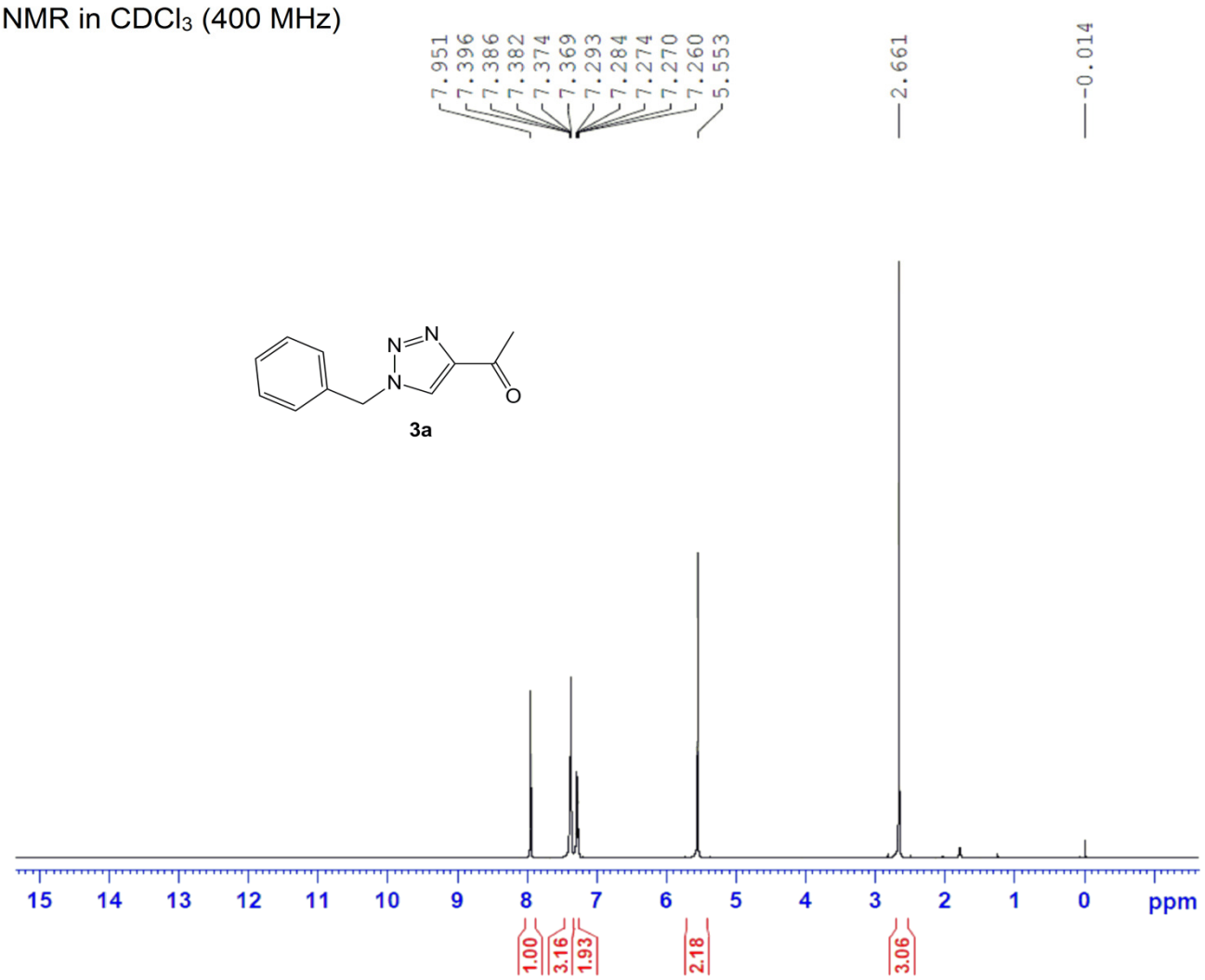
10 M. M. Majireck and S. M. Weinreb, *J. Org. Chem.,* 2006, **71**, 8680-8683.

11 M. Tajbakhsh, M. Farhang, S. M. Baghbanian, R. Hosseinzadeh, and M. Tajbakhsha, *New J. Chem.***,** 2015, **39**, 1827-1839.

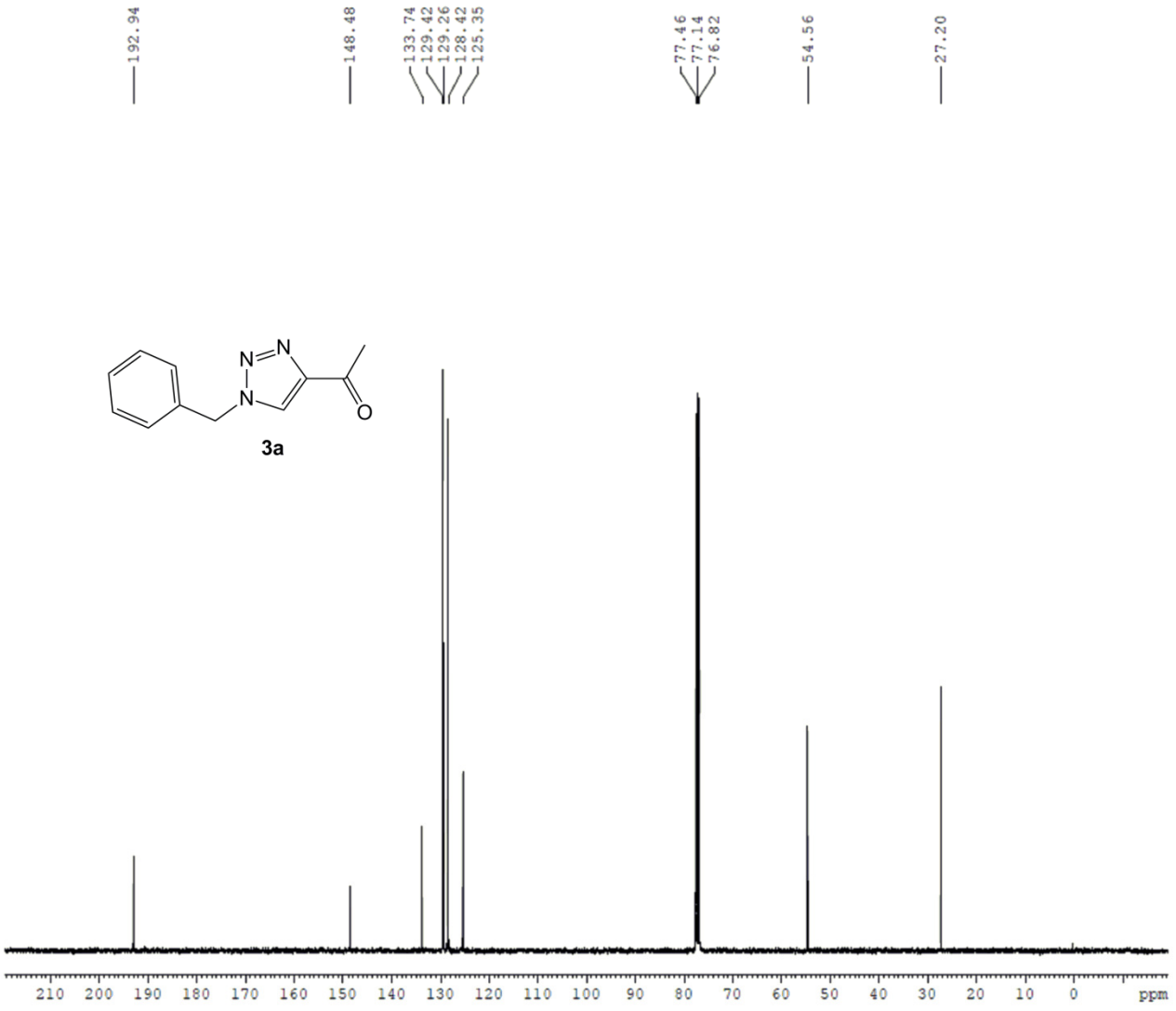
12 Y-Y. Xie, Y-C. Wang, H-E. Qu, X-C. Tan, H-S. Wang and Y-M. Pan, *Adv. Synth. Catal*., 2014, **356**, 3347-3355.

NMR Spectra

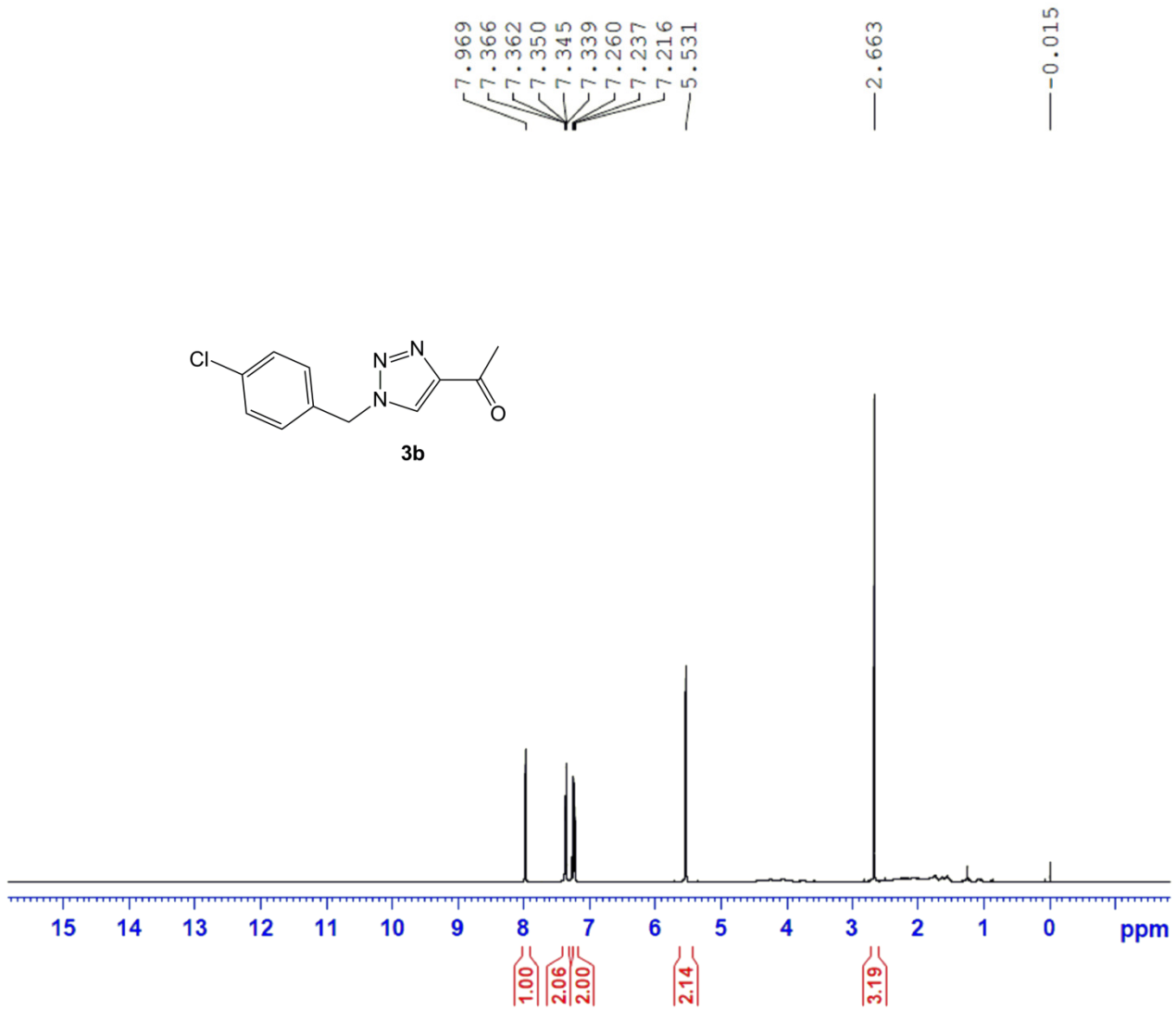
^{1}H NMR in $CDCl_3$ (400 MHz)



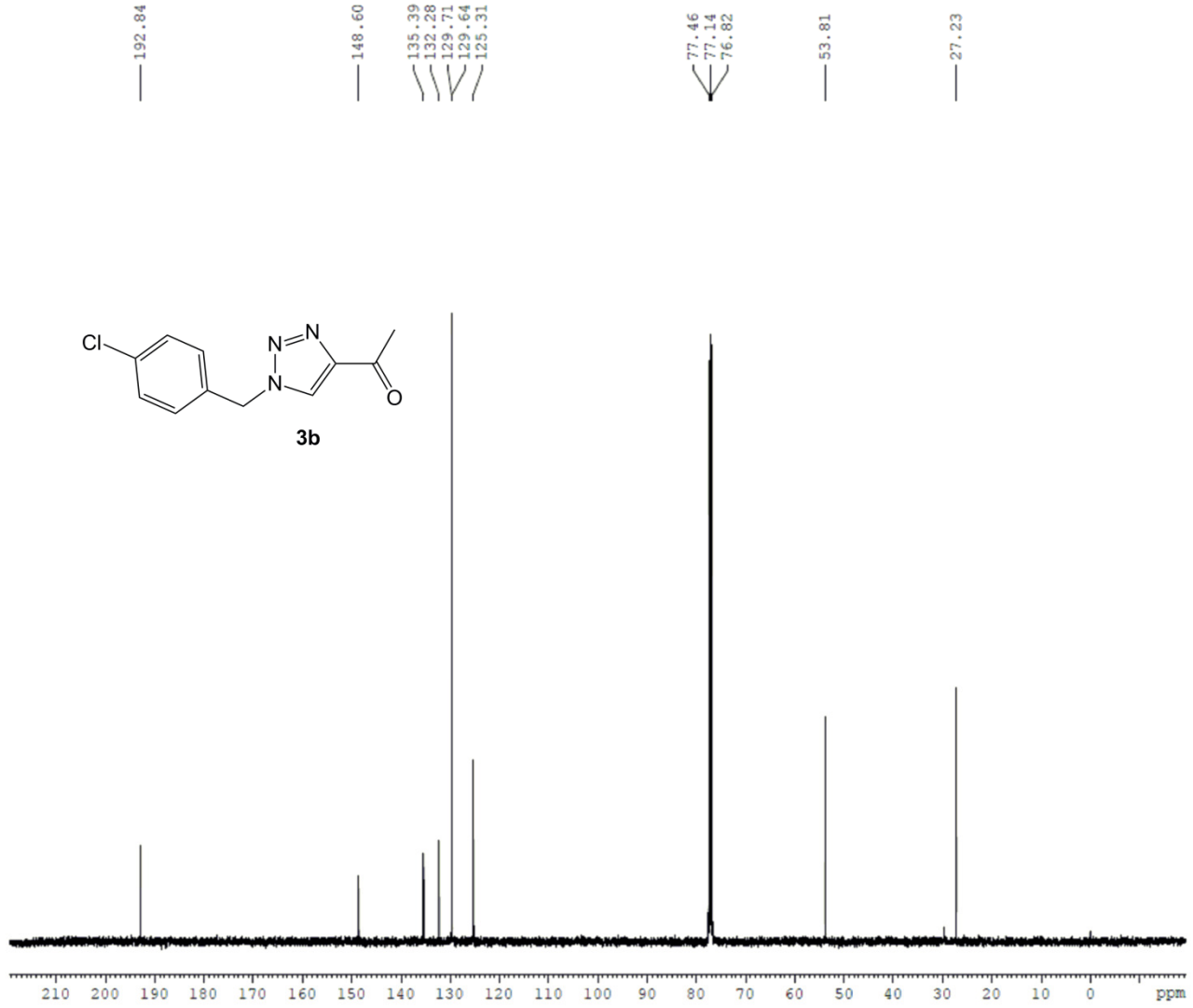
^{13}C NMR in $CDCl_3$ (100 MHz)



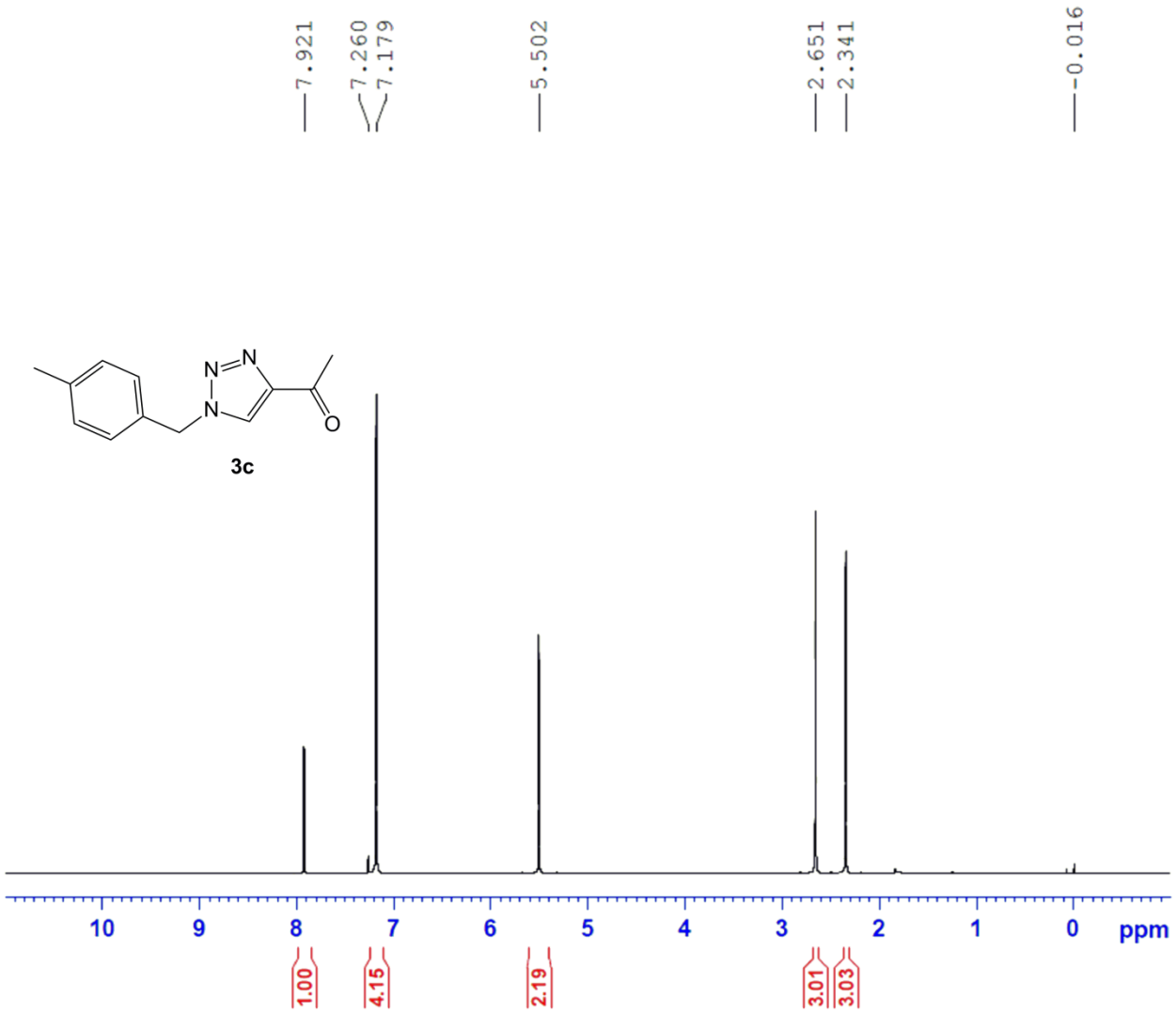
^{1}H NMR in $CDCl_3$ (400 MHz)



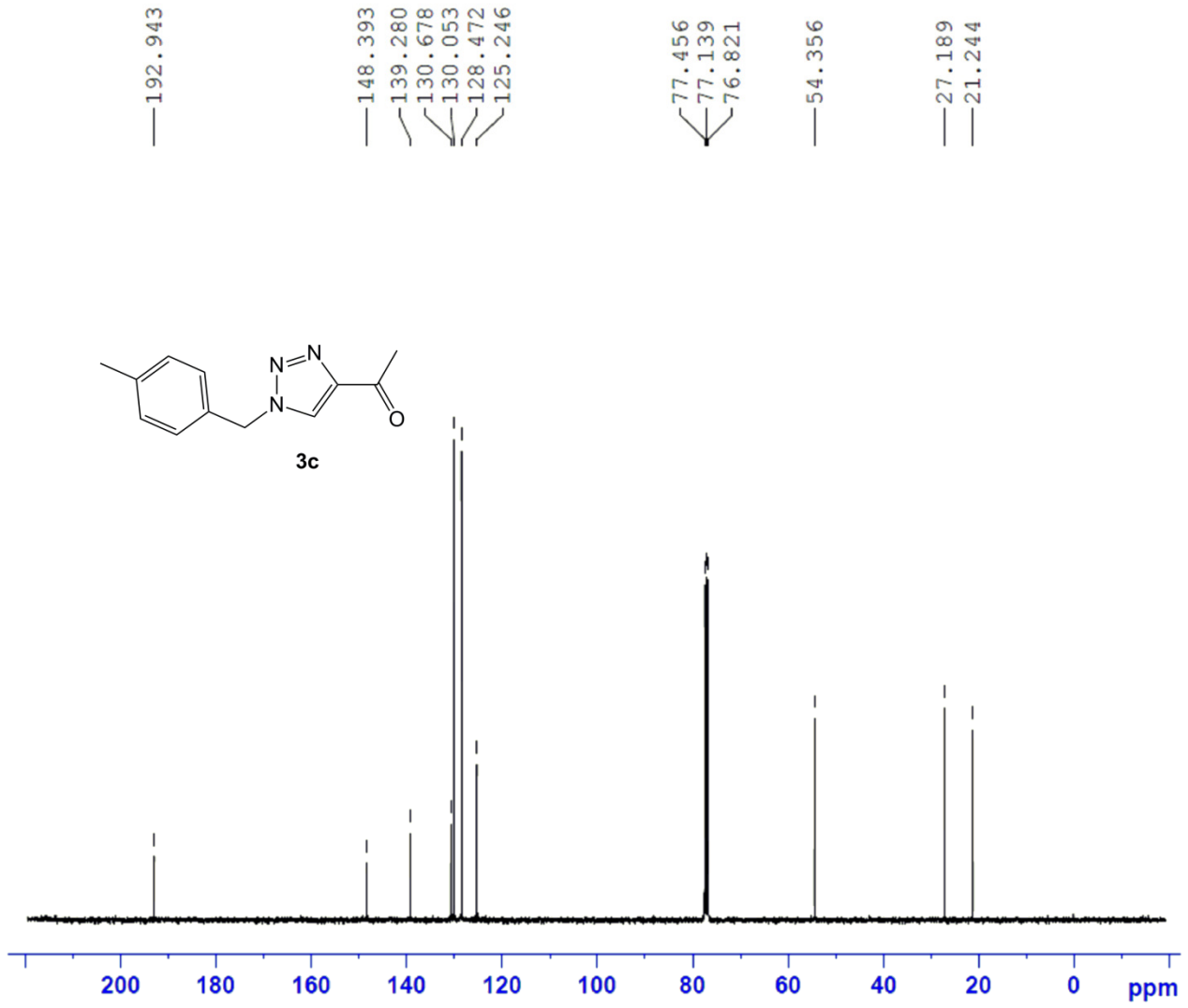
^{13}C NMR in $CDCl_3$ (100 MHz)



^{1}H NMR in $CDCl_3$ (400 MHz)

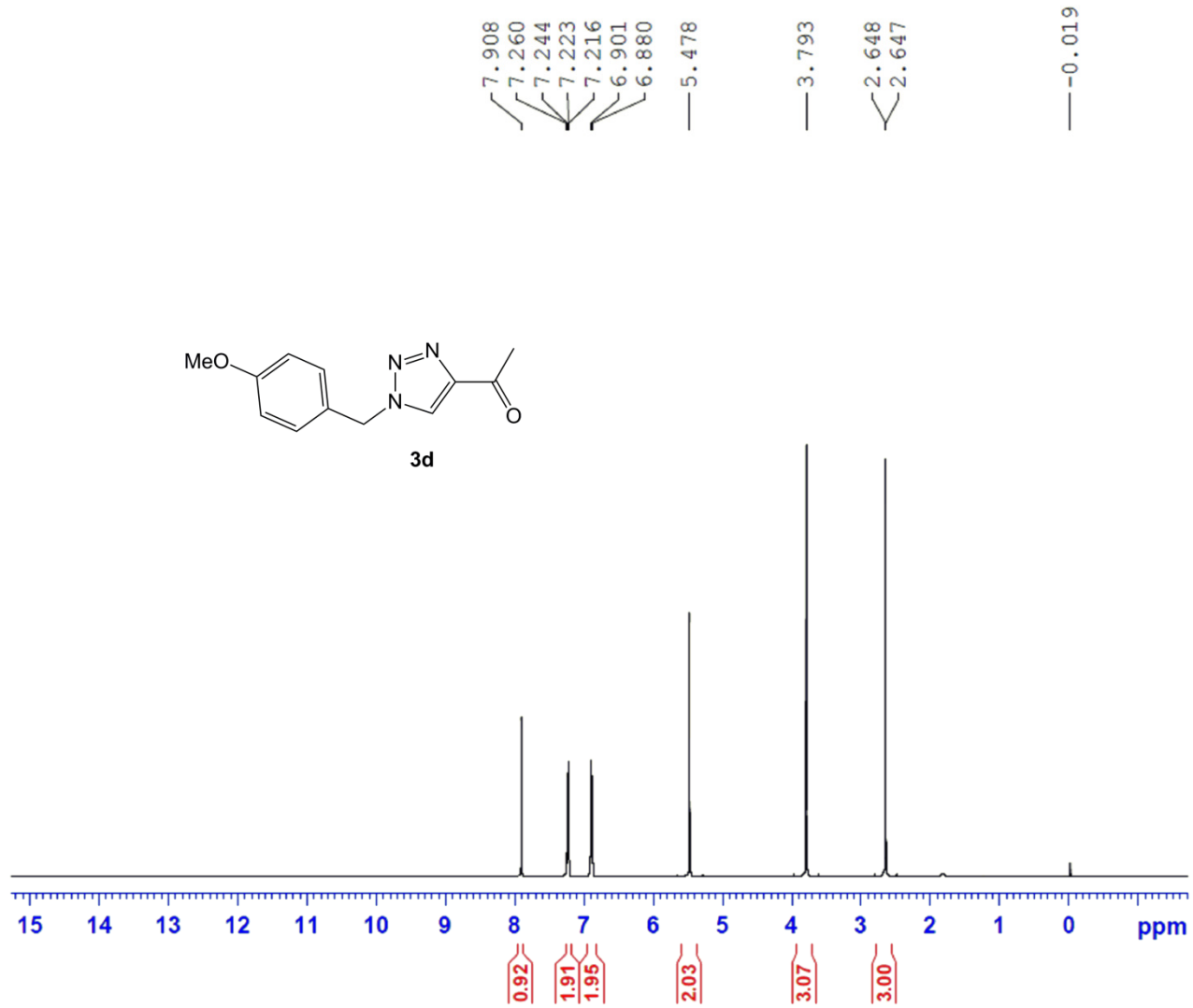


^{13}C NMR in $CDCl_3$ (100 MHz)

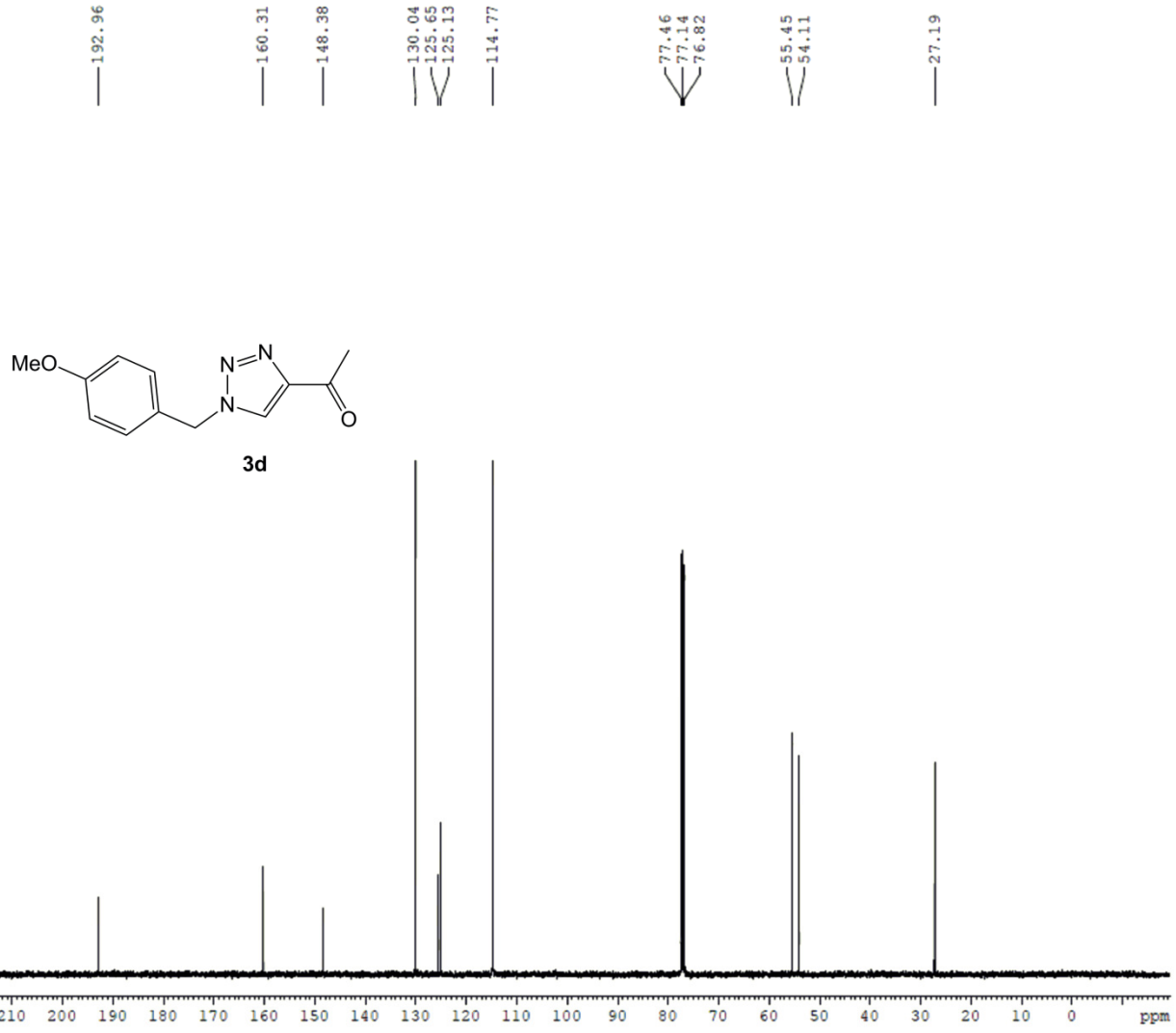


3c

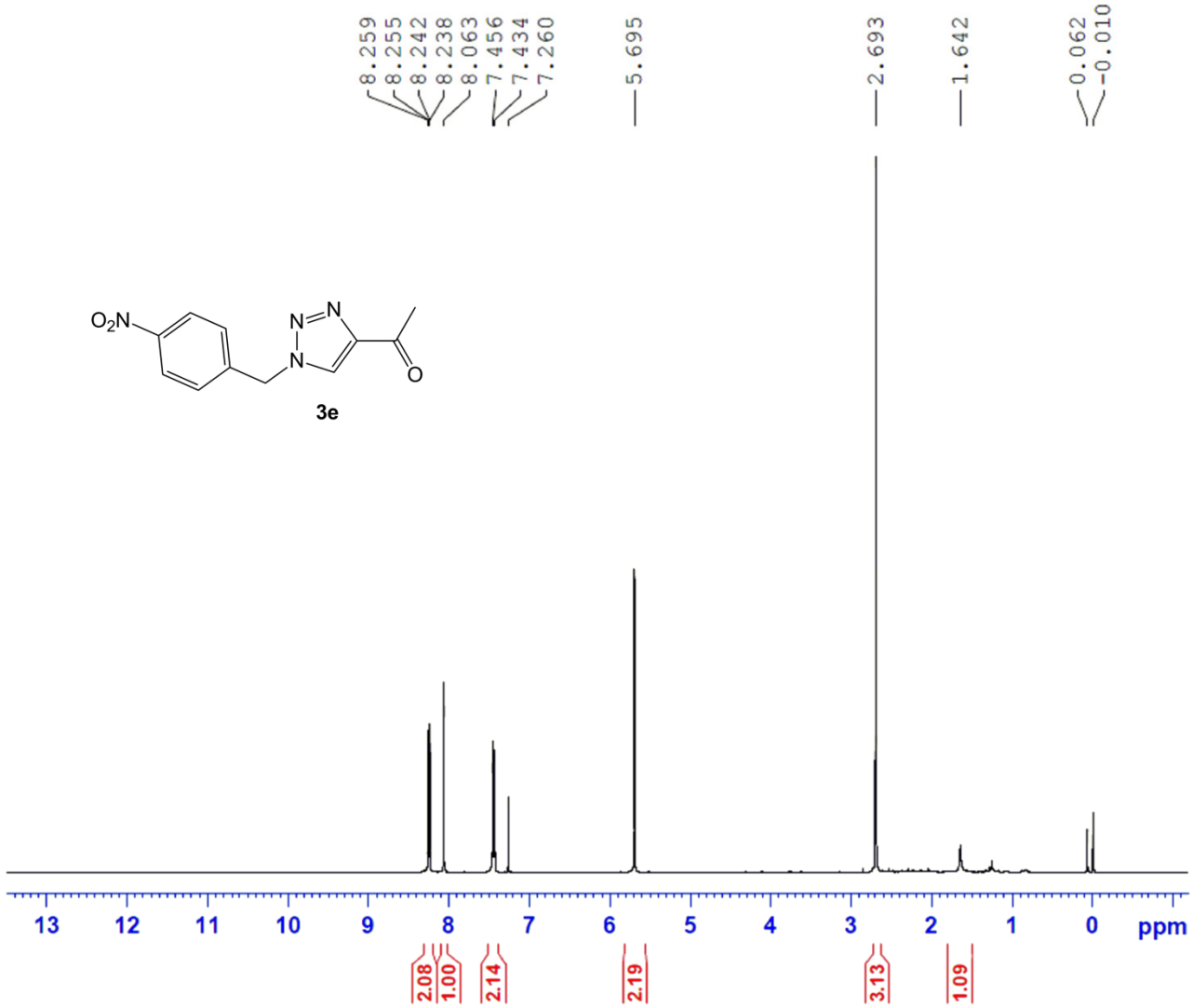
^{1}H NMR in $CDCl_3$ (400 MHz)



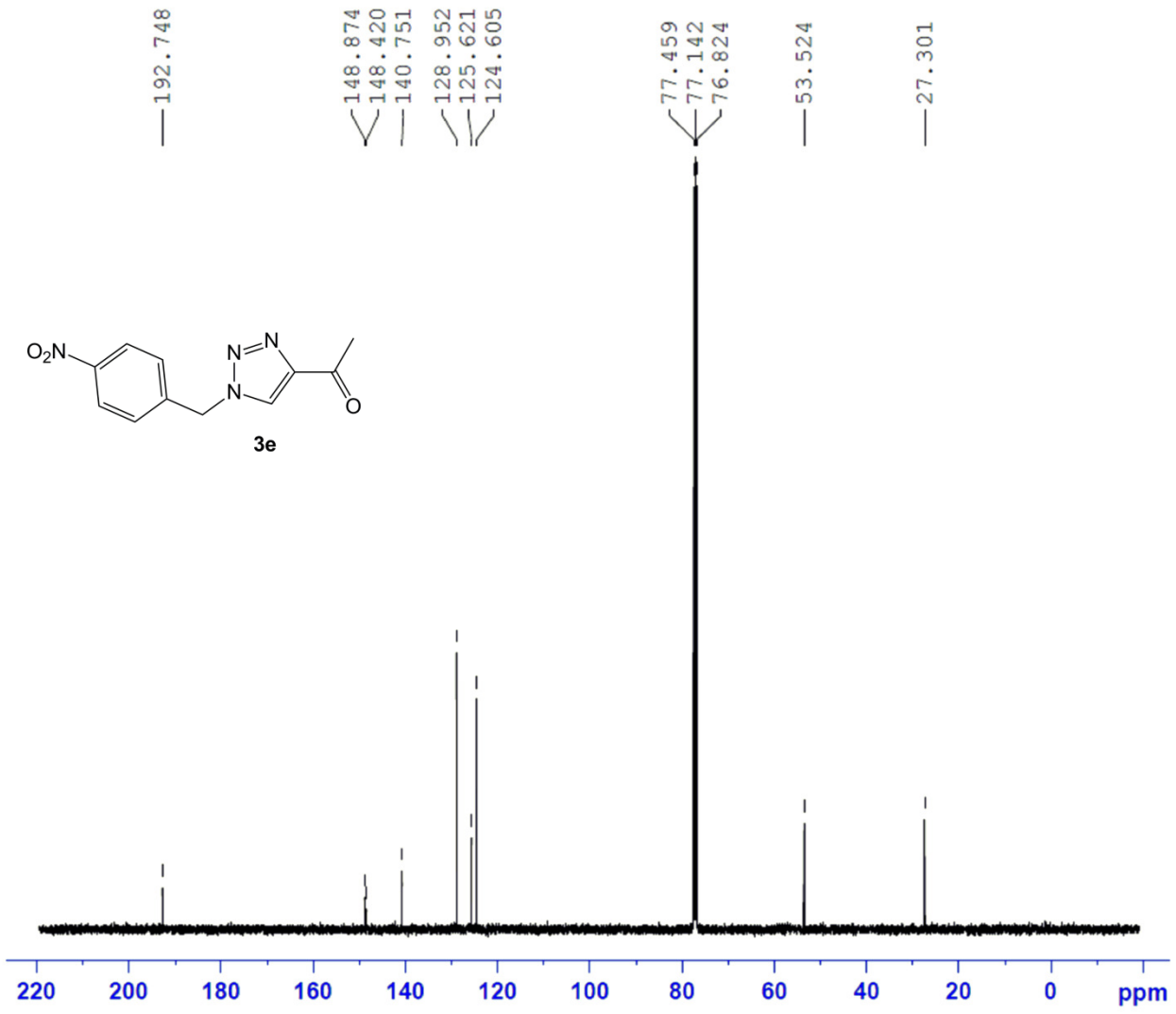
^{13}C NMR in $CDCl_3$ (100 MHz)



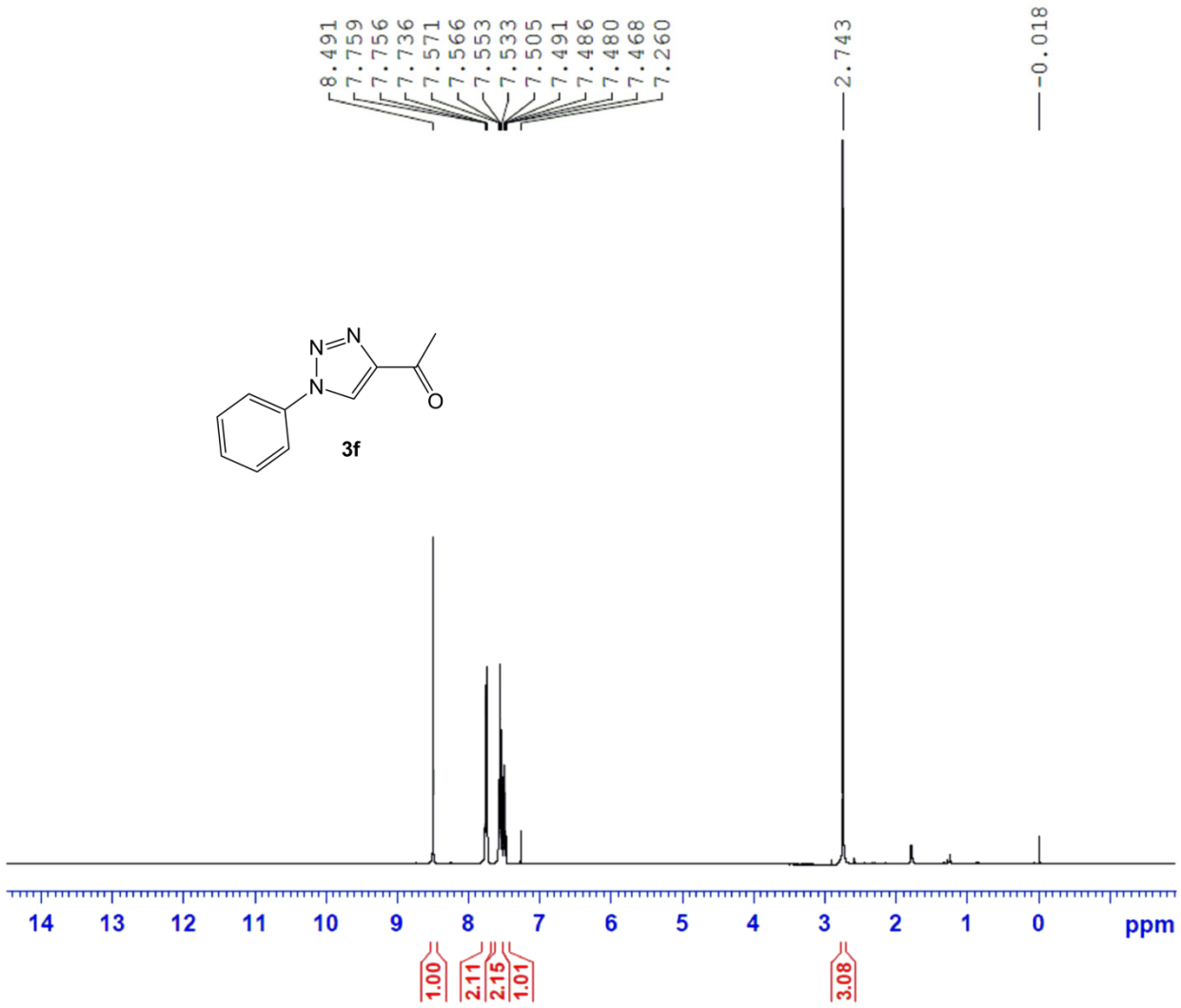
^{1}H NMR in $CDCl_3$ (400 MHz)



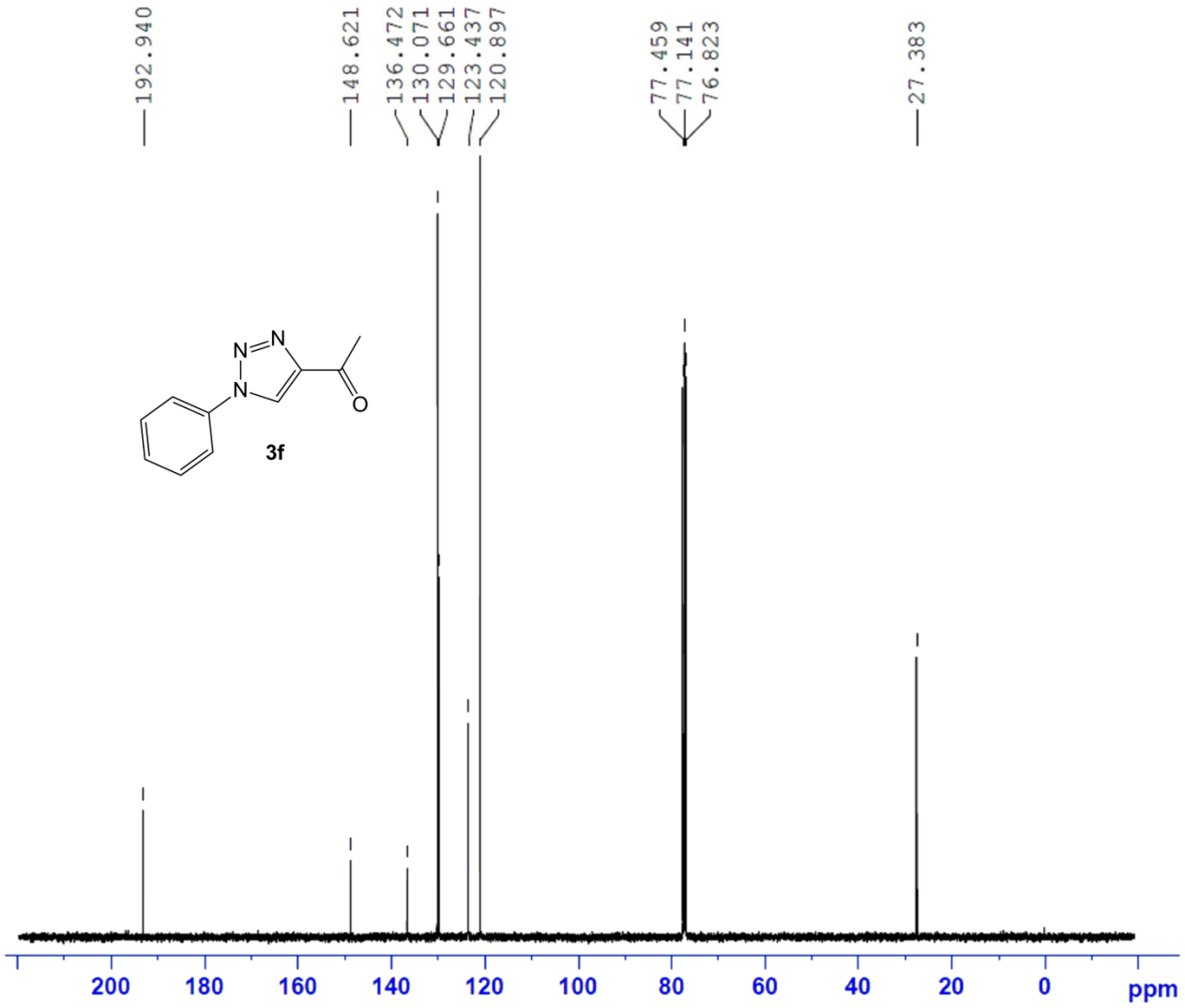
^{13}C NMR in $CDCl_3$ (100 MHz)



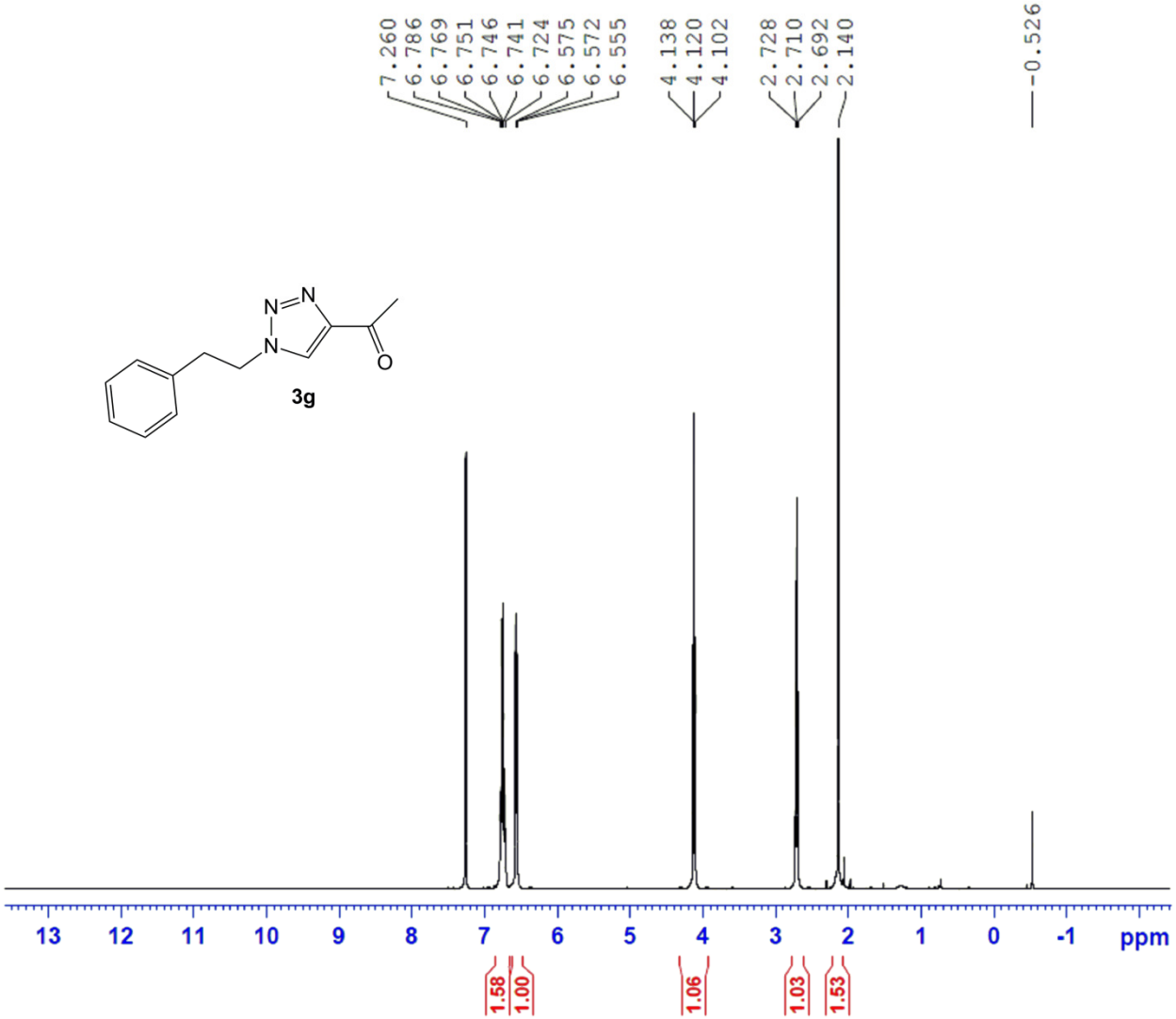
^{1}H NMR in $CDCl_3$ (400 MHz)



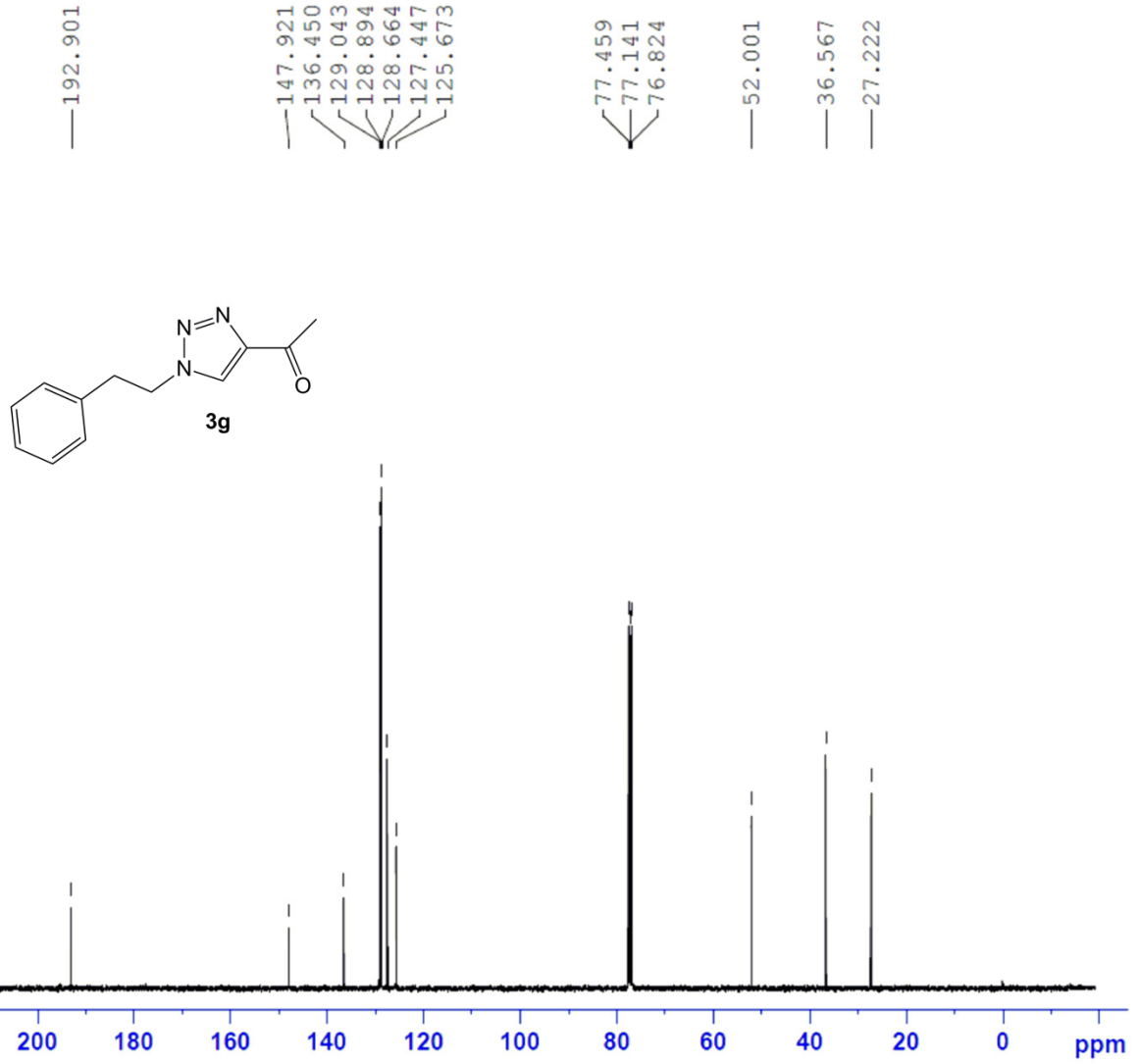
^{13}C NMR in $CDCl_3$ (100 MHz)



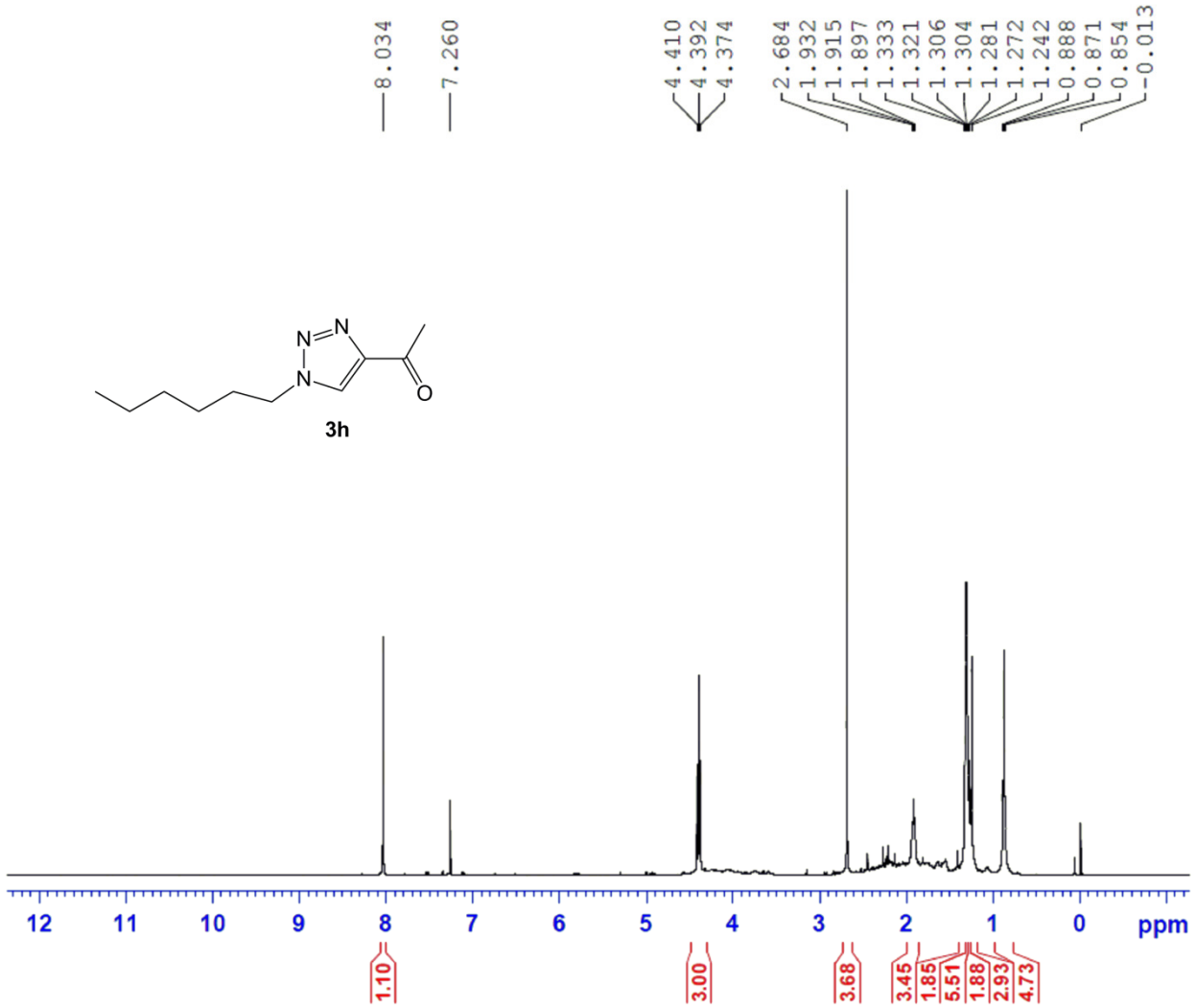
^{1}H NMR in $CDCl_3$ (400 MHz)



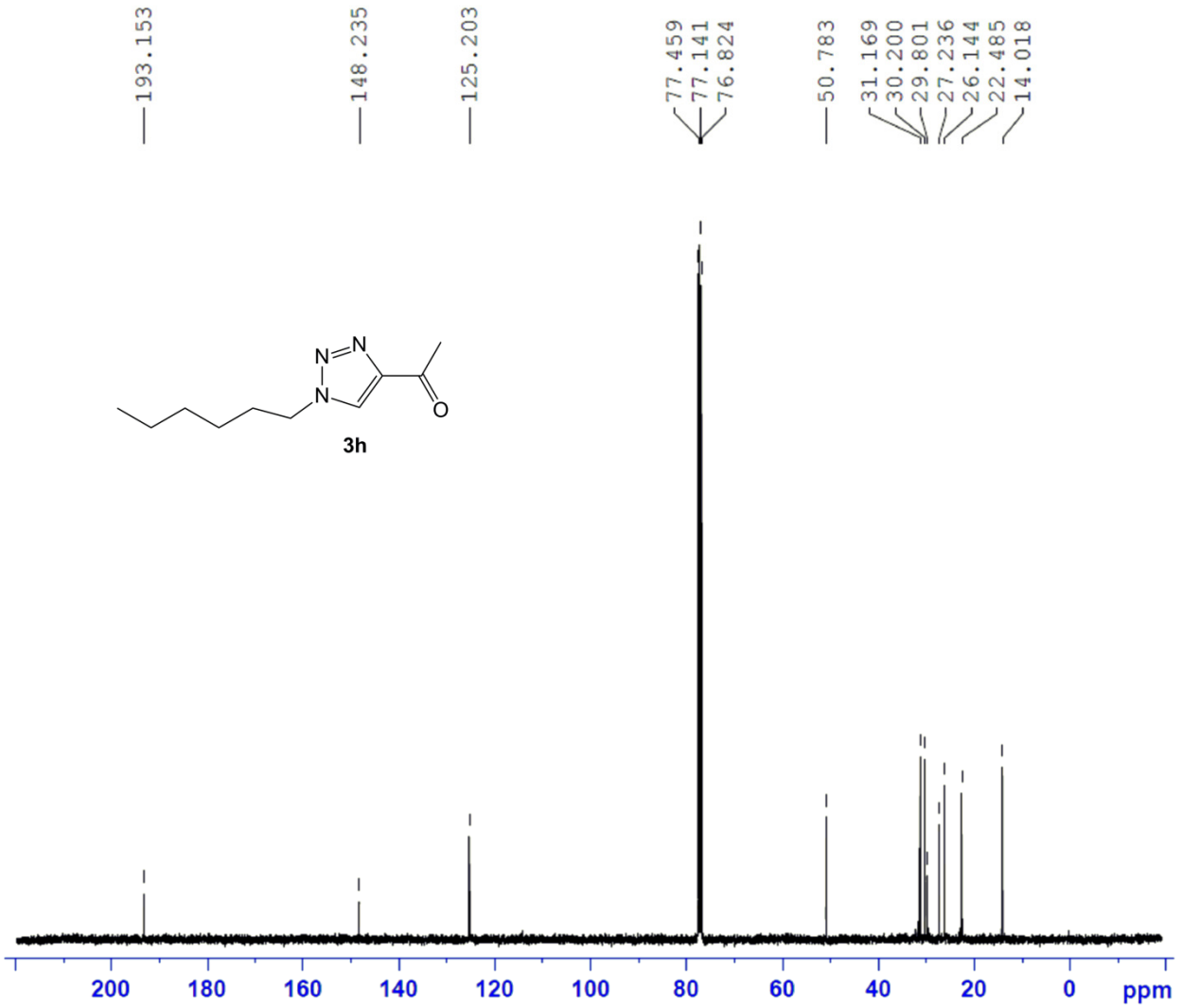
^{13}C NMR in $CDCl_3$ (100 MHz)



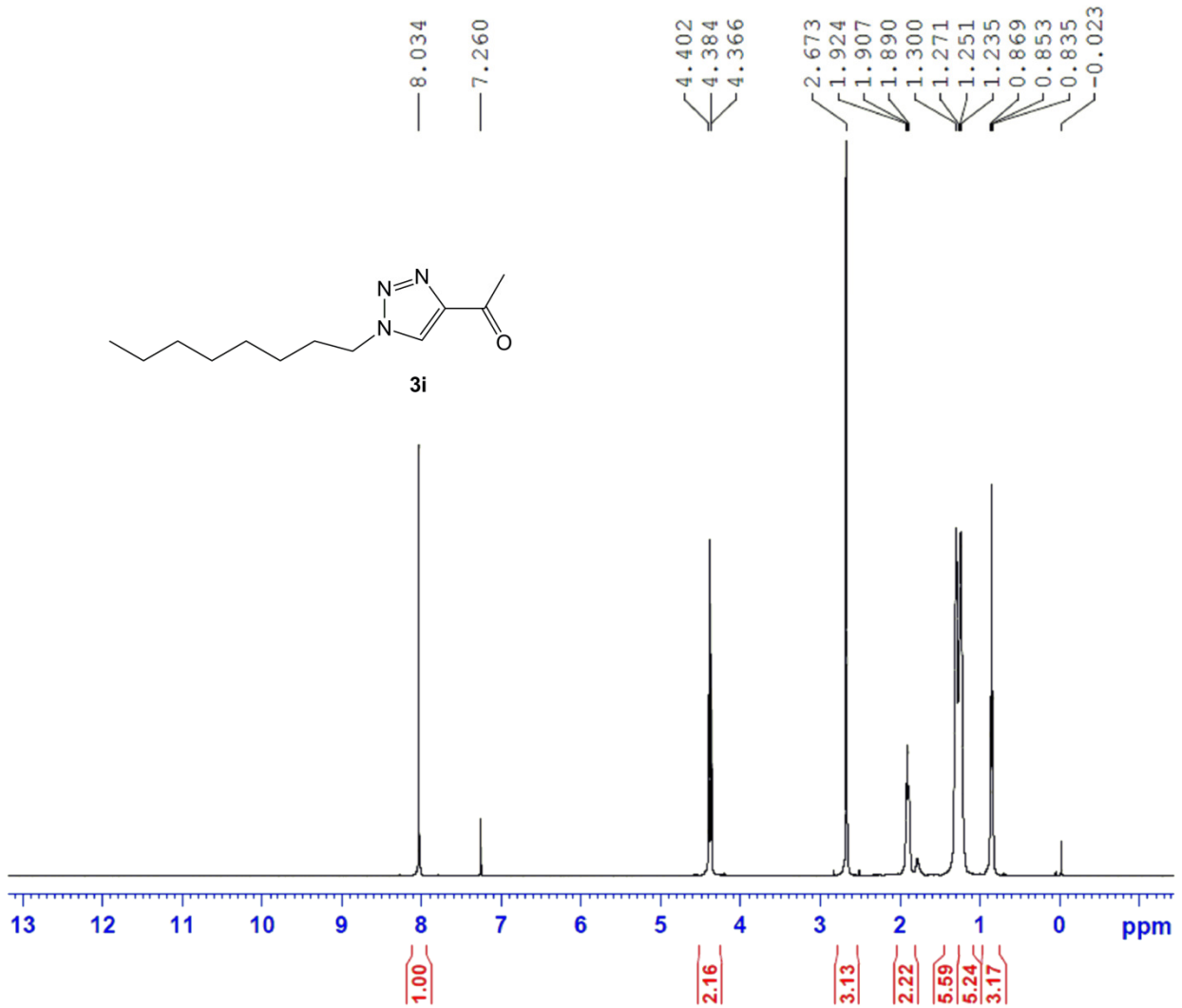
^{1}H NMR in $CDCl_3$ (400 MHz)



^{13}C NMR in $CDCl_3$ (100 MHz)

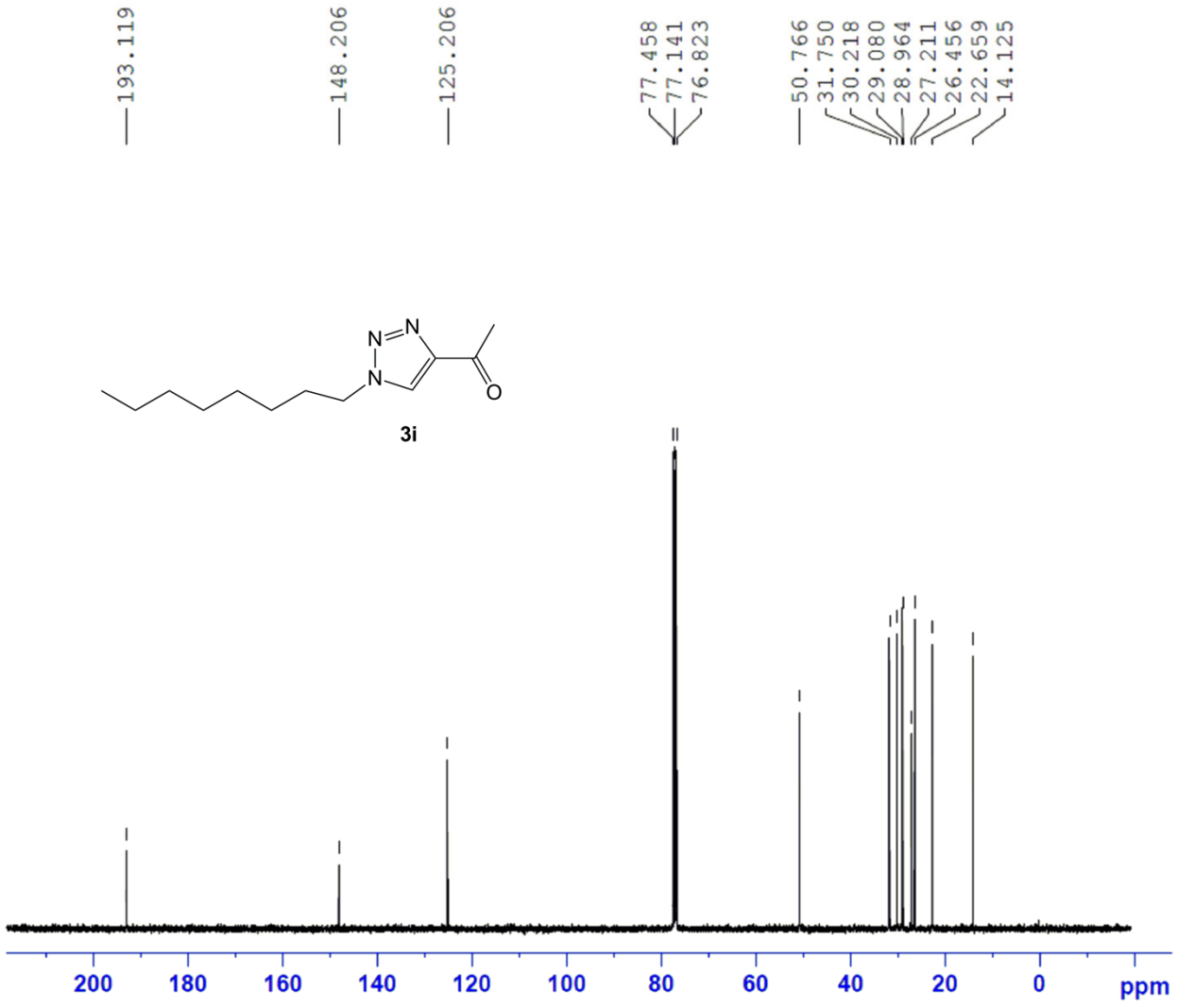


^{1}H NMR in $CDCl_3$ (400 MHz)

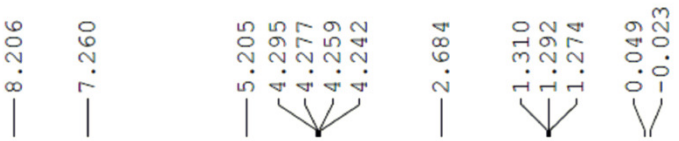


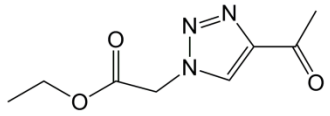
3i

^{13}C NMR in $CDCl_3$ (100 MHz)

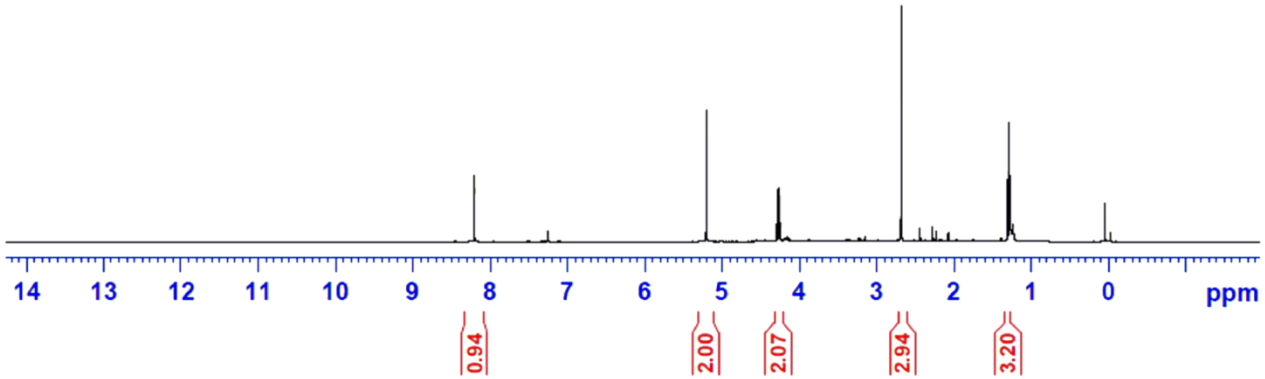


^{1}H NMR in $CDCl_3$ (400 MHz)

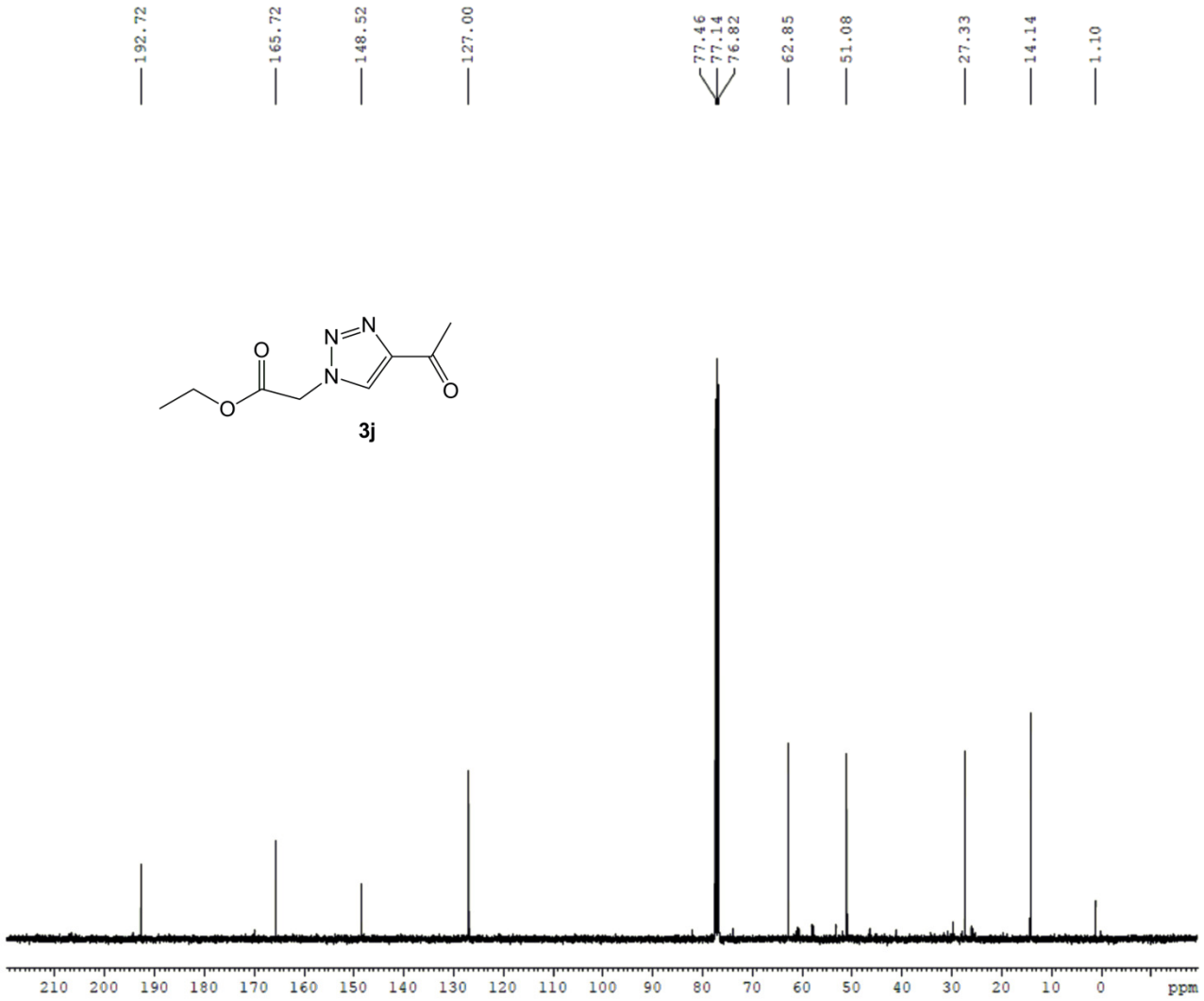




3j

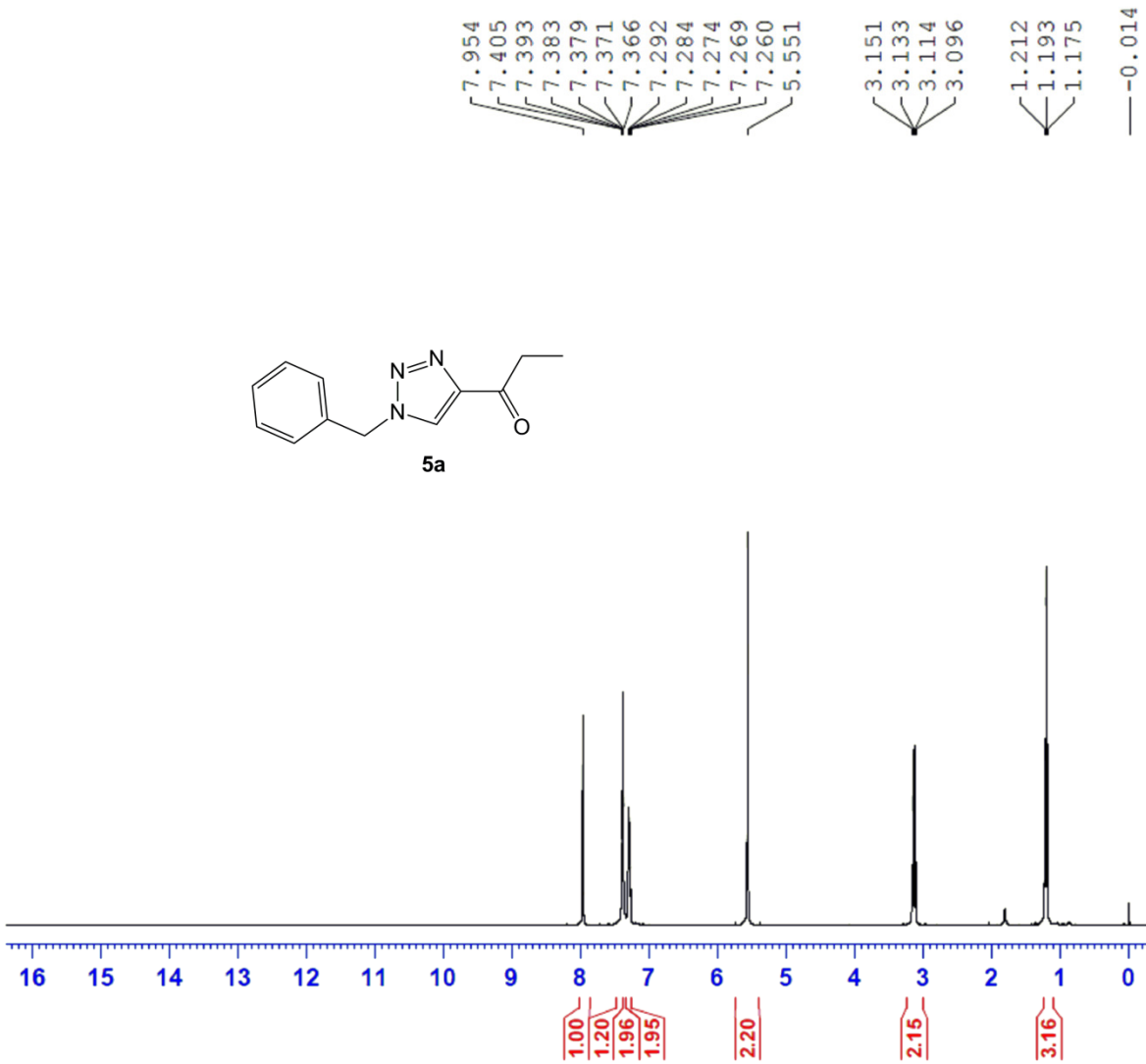


^{13}C NMR in $CDCl_3$ (100 MHz)



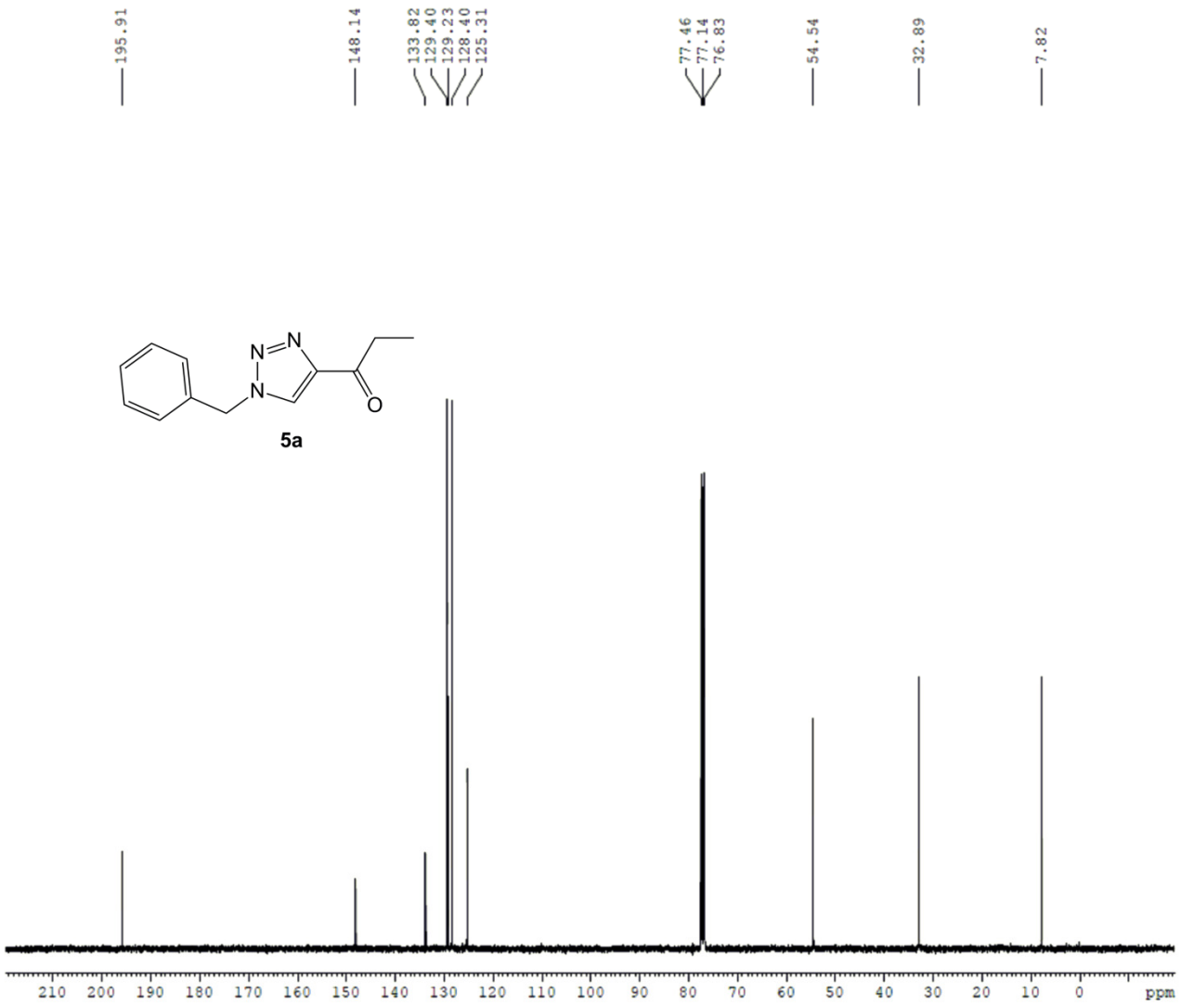
3j

^{1}H NMR in $CDCl_3$ (400 MHz)

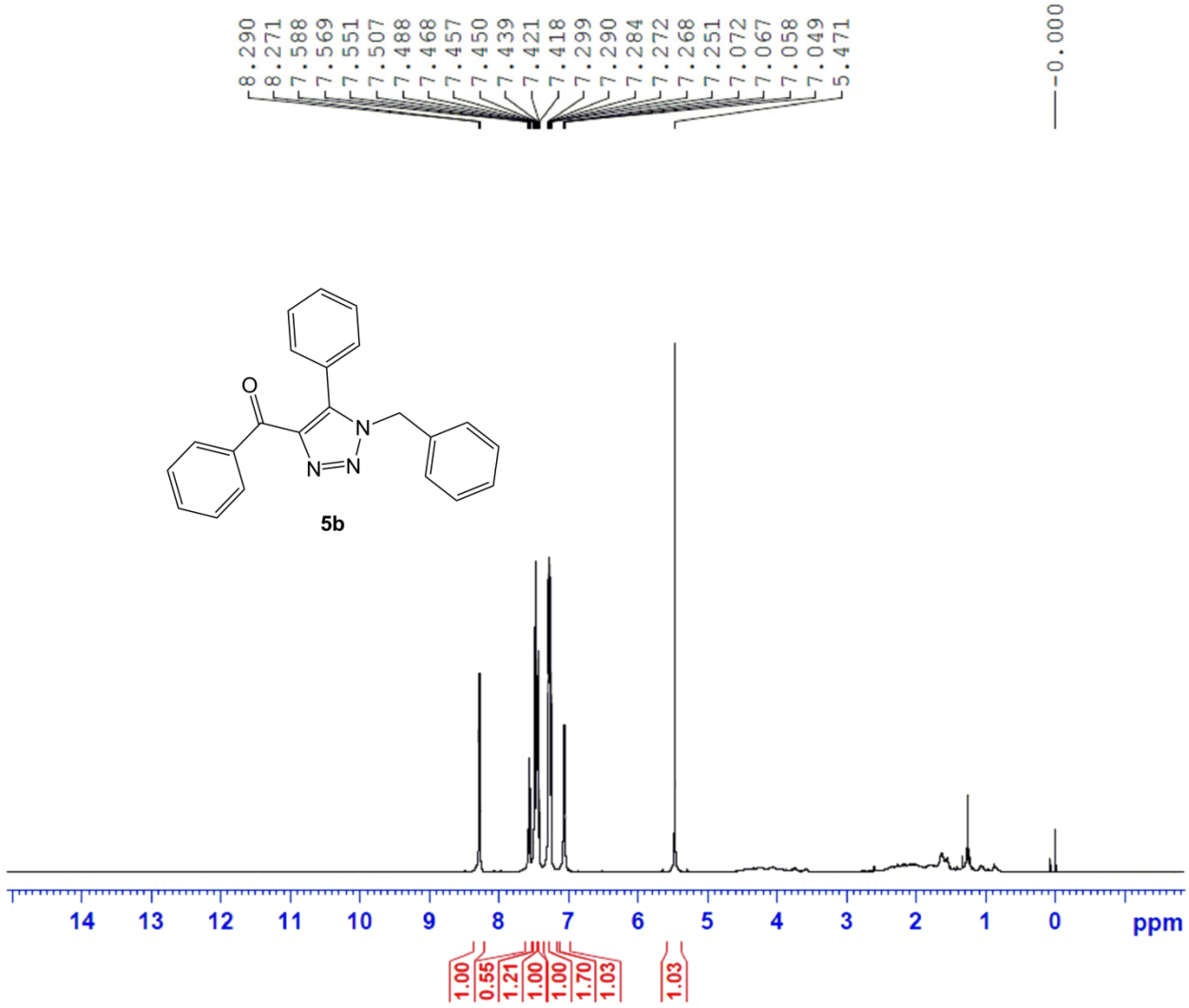


5a

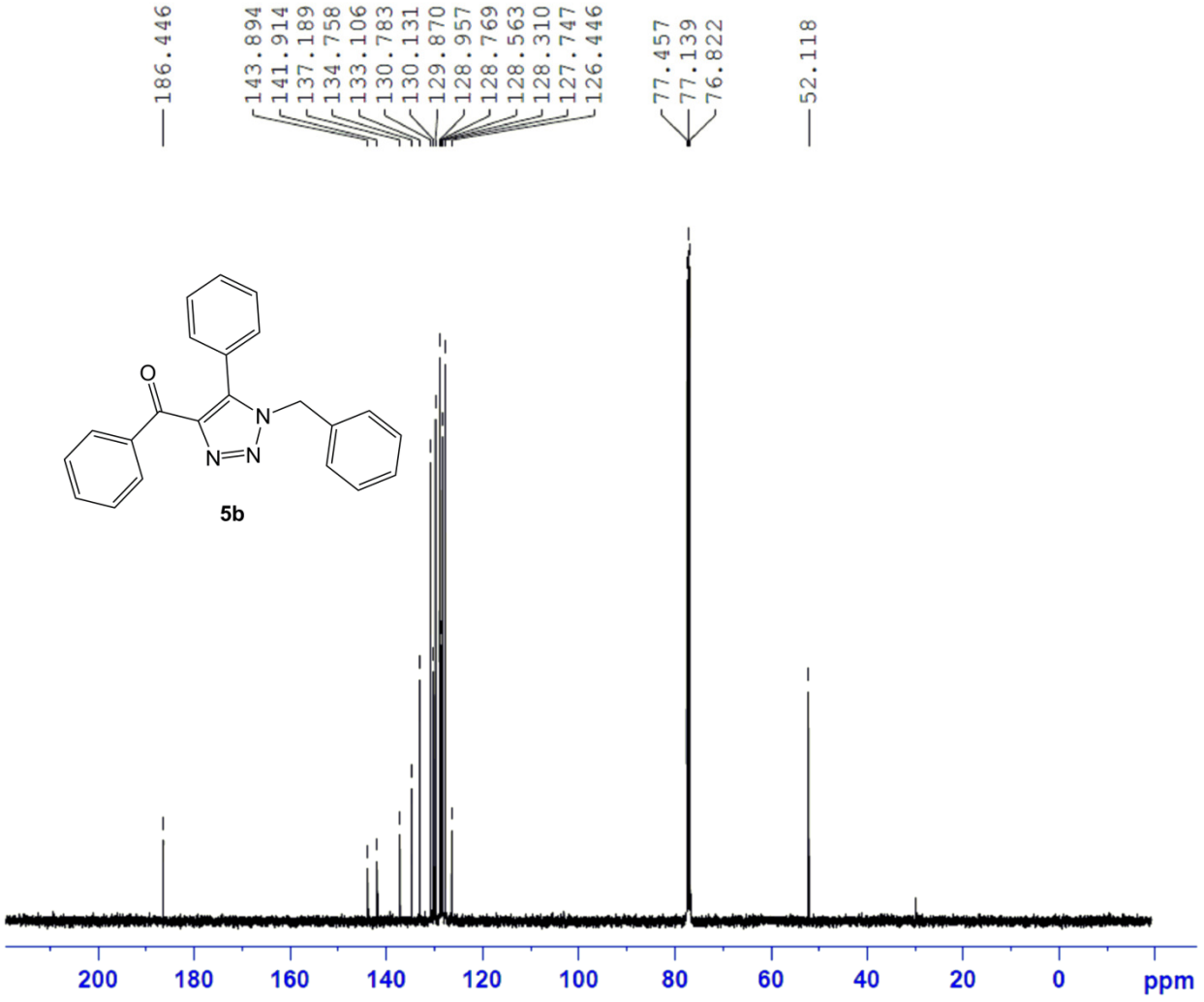
^{13}C NMR in $CDCl_3$ (100 MHz)



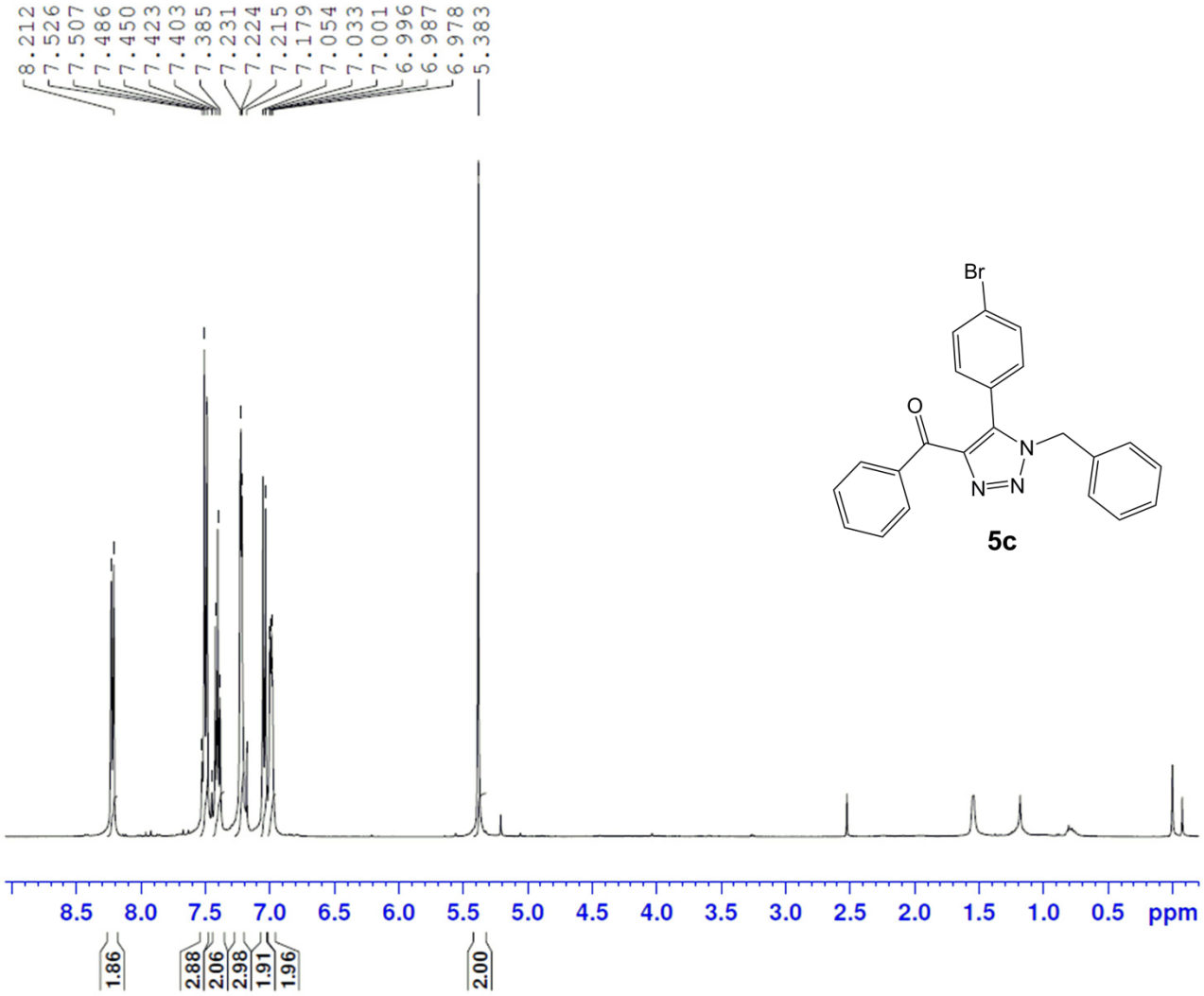
^{1}H NMR in $CDCl_3$ (400 MHz)



^{13}C NMR in $CDCl_3$ (100 MHz)

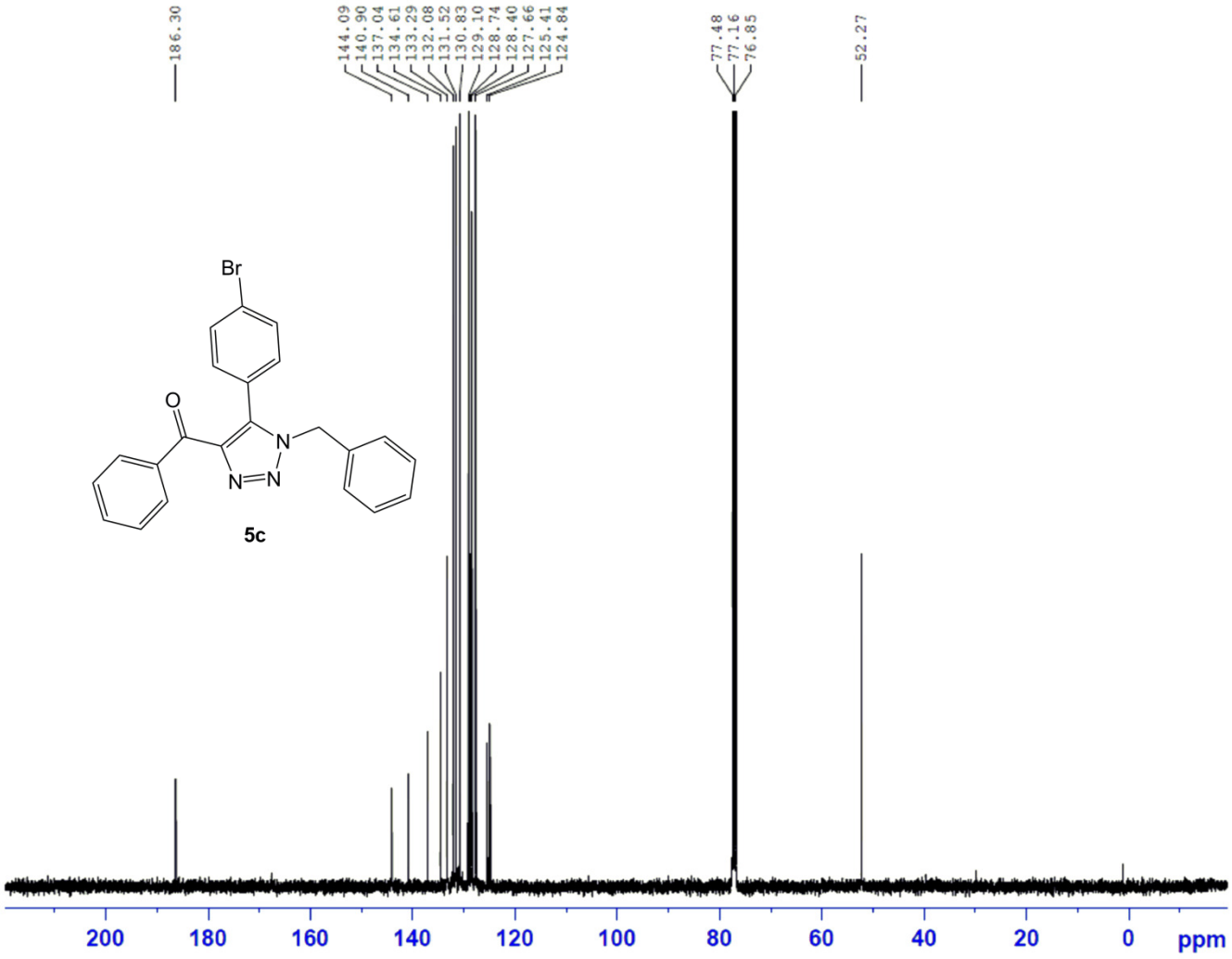


^{1}H NMR in $CDCl_3$ (400 MHz)

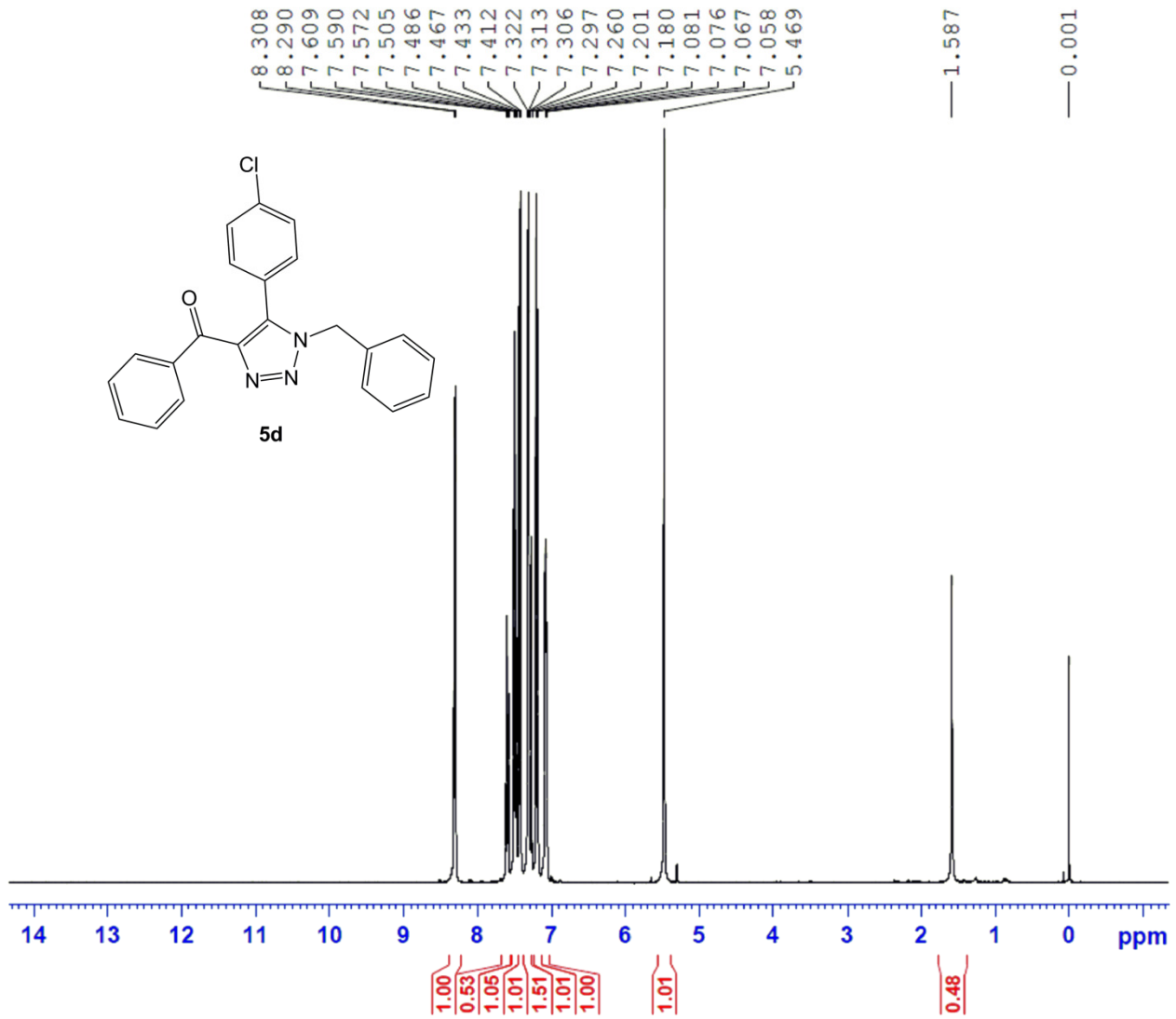


5c

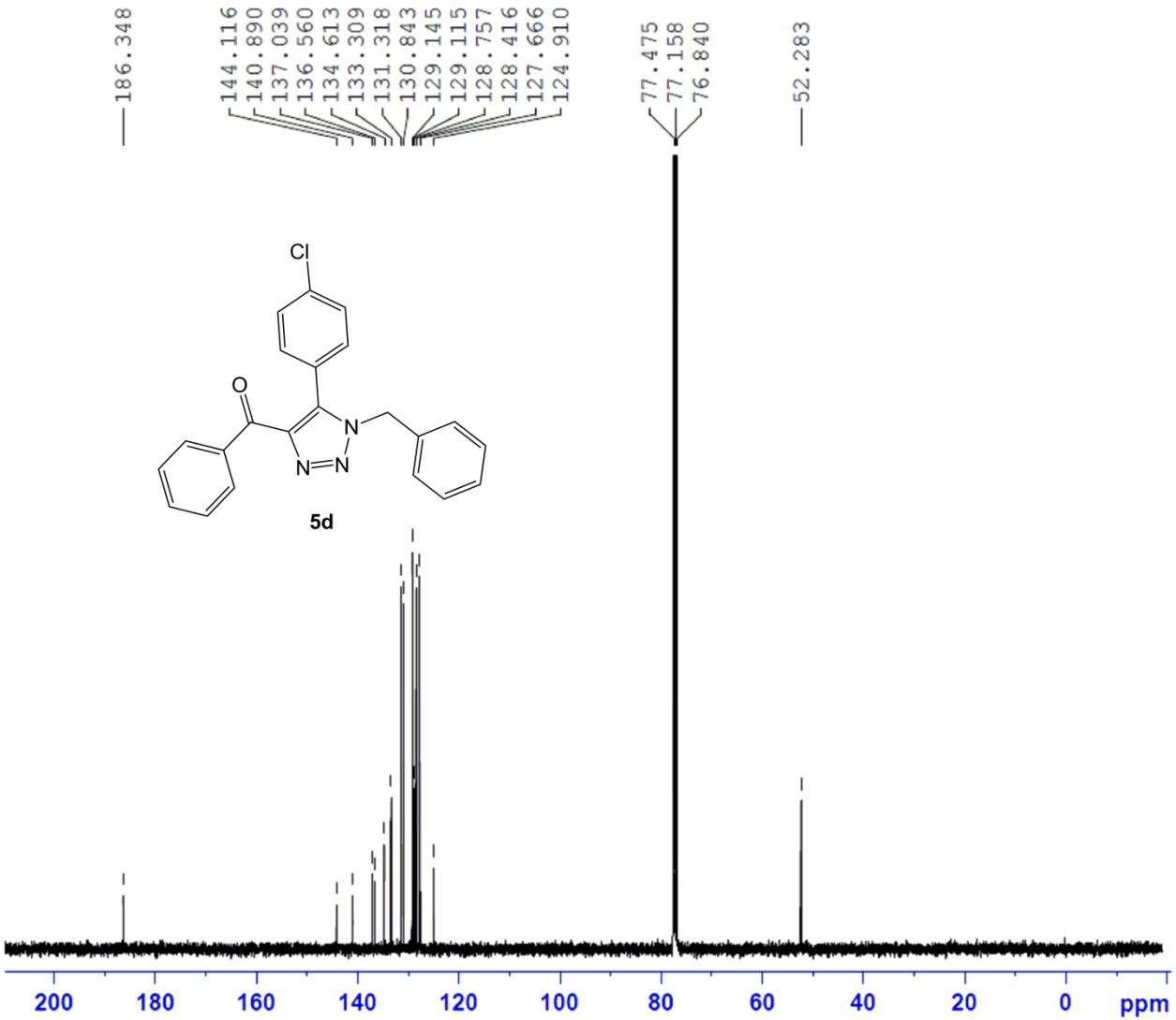
^{13}C NMR in $CDCl_3$ (100 MHz)



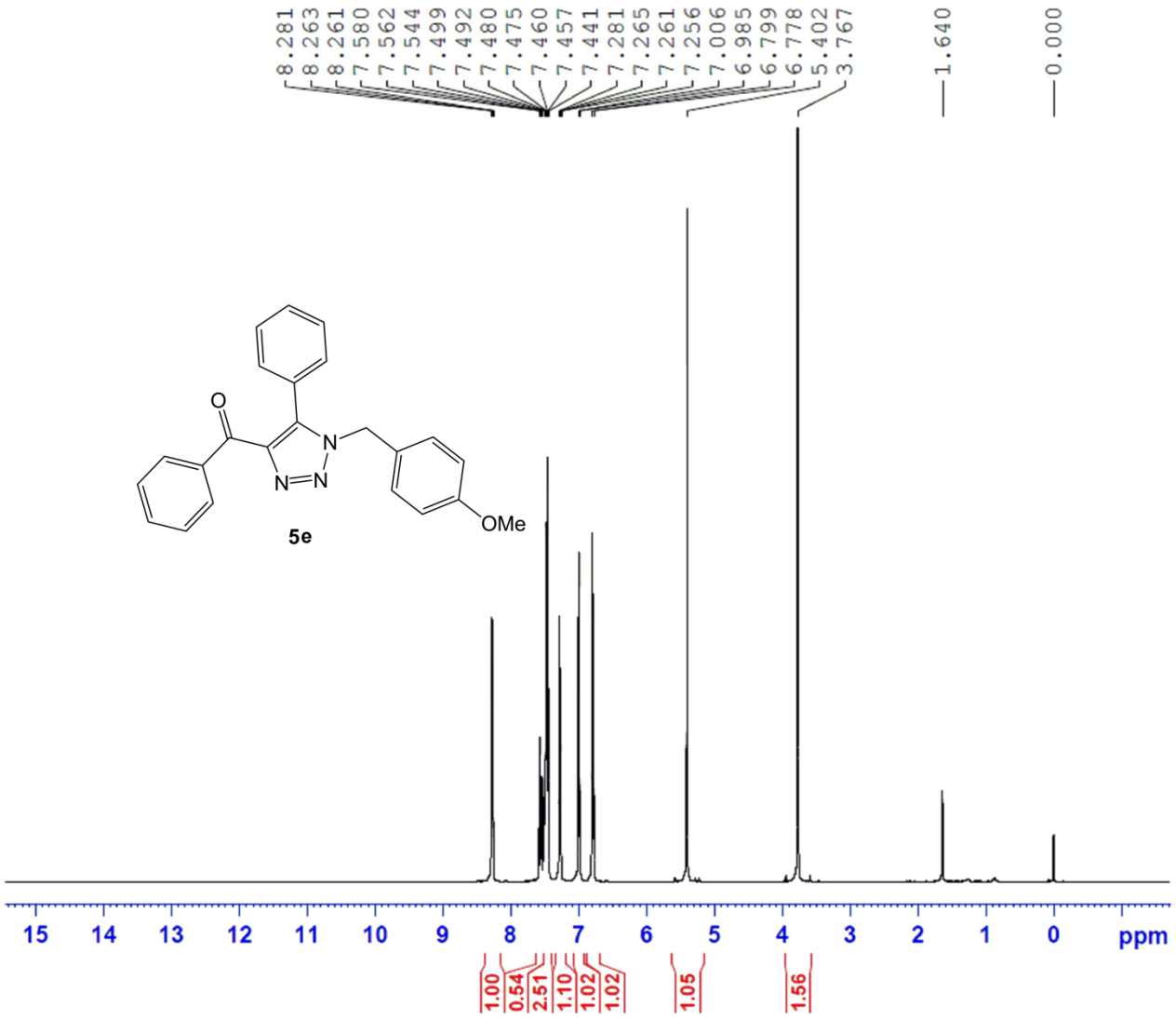
^{1}H NMR in $CDCl_3$ (400 MHz)



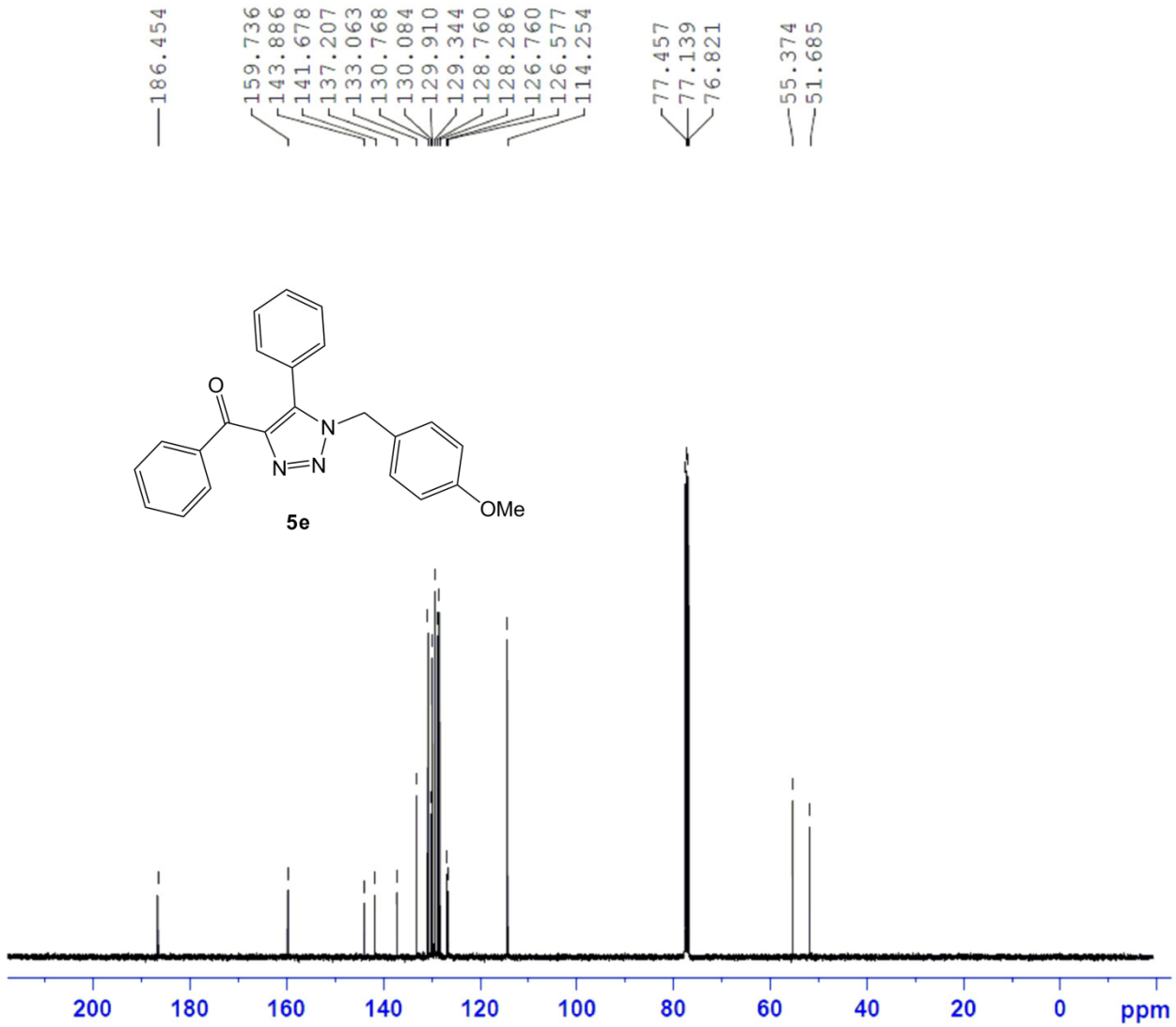
^{13}C NMR in $CDCl_3$ (100 MHz)



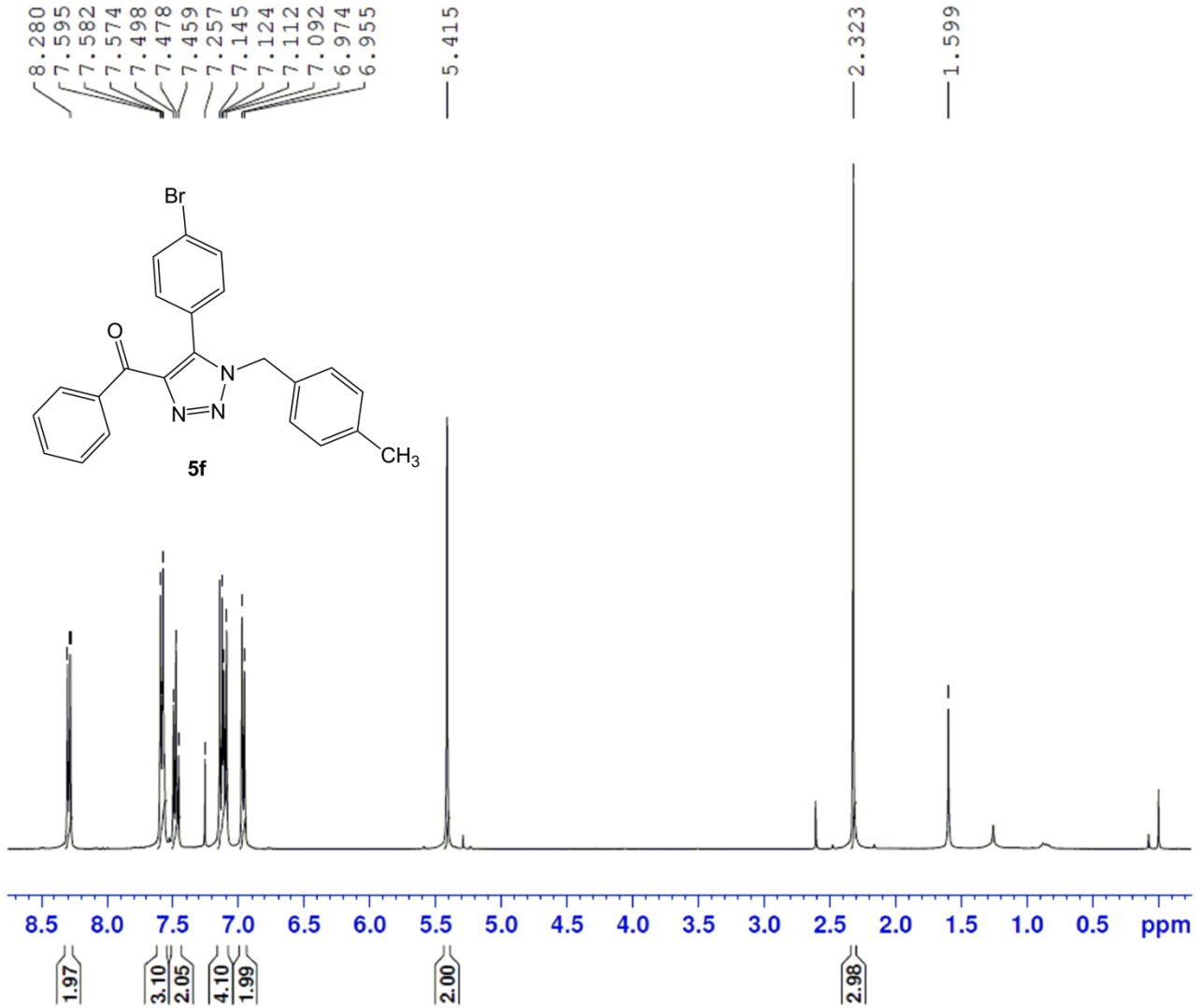
^{1}H NMR in $CDCl_3$ (400 MHz)



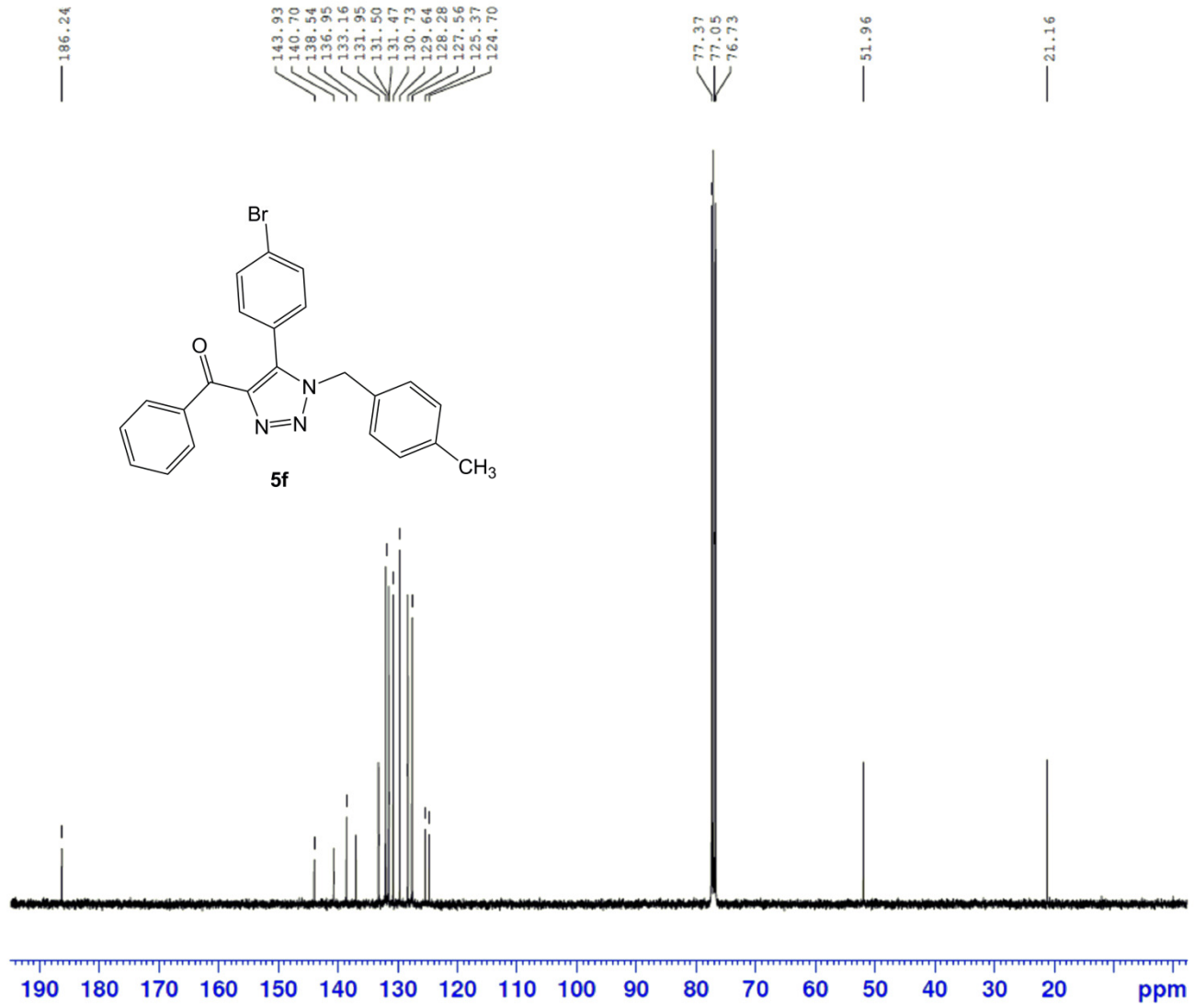
^{13}C NMR in $CDCl_3$ (100 MHz)



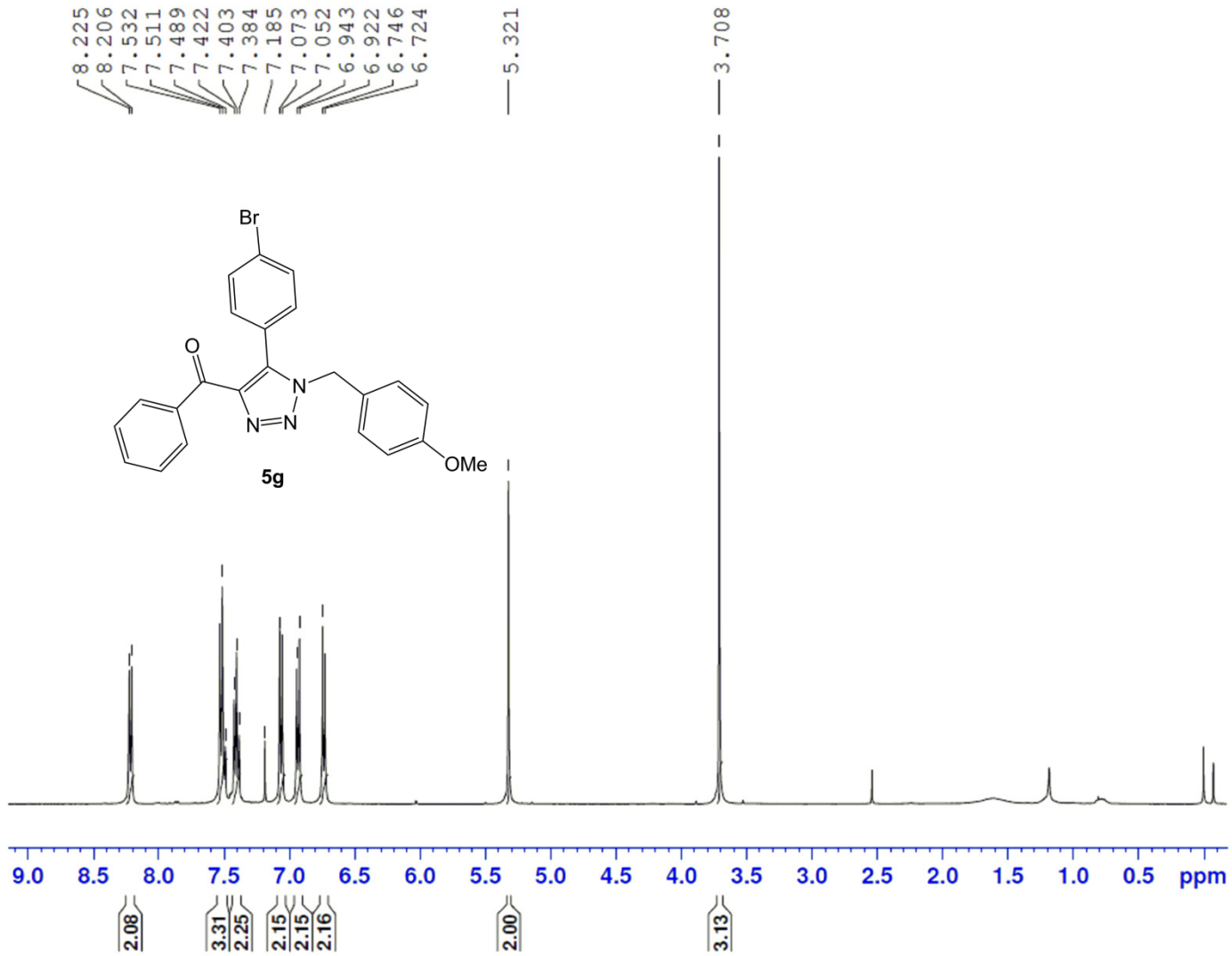
^{1}H NMR in $CDCl_3$ (400 MHz)



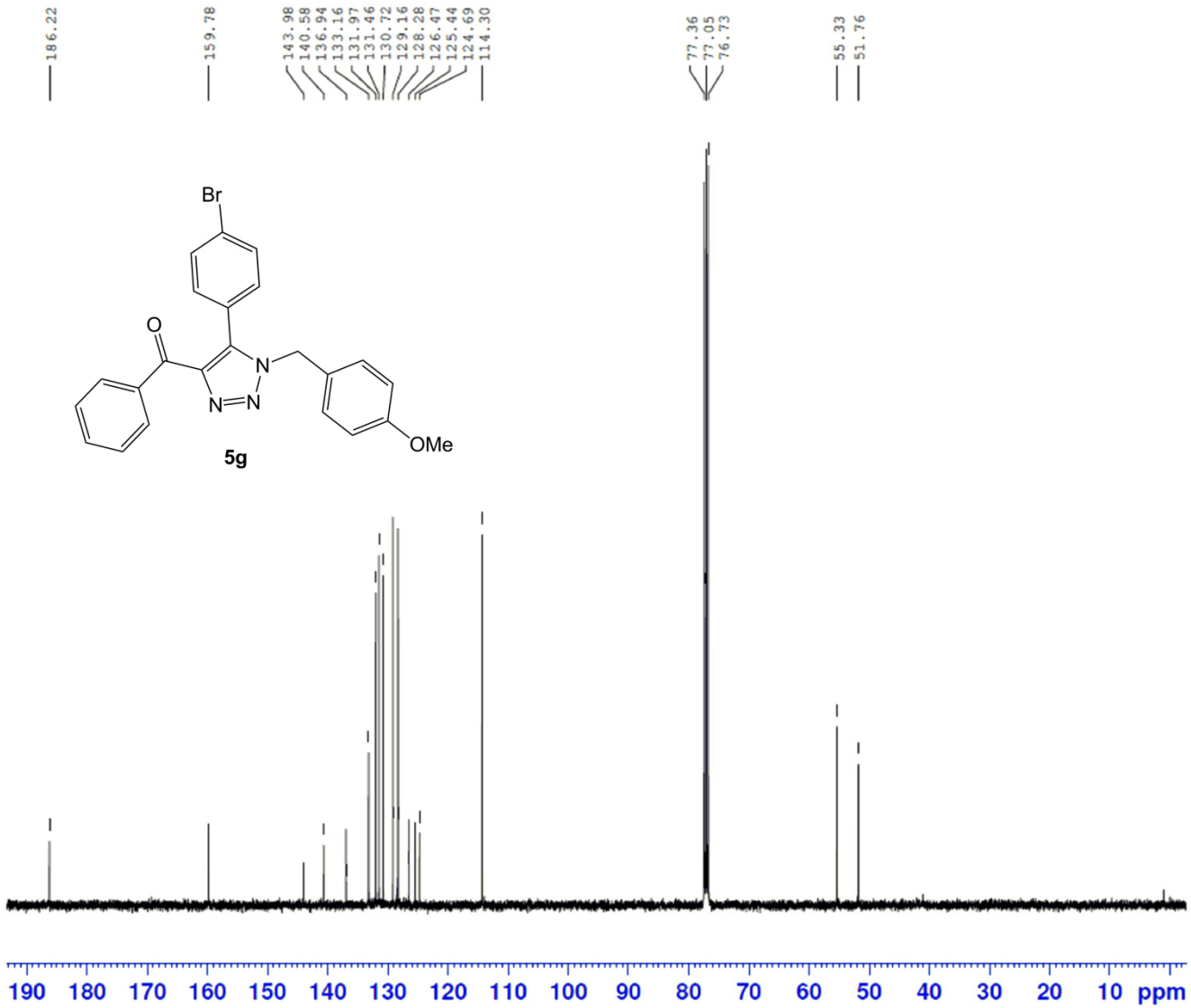
^{13}C NMR in $CDCl_3$ (100 MHz)



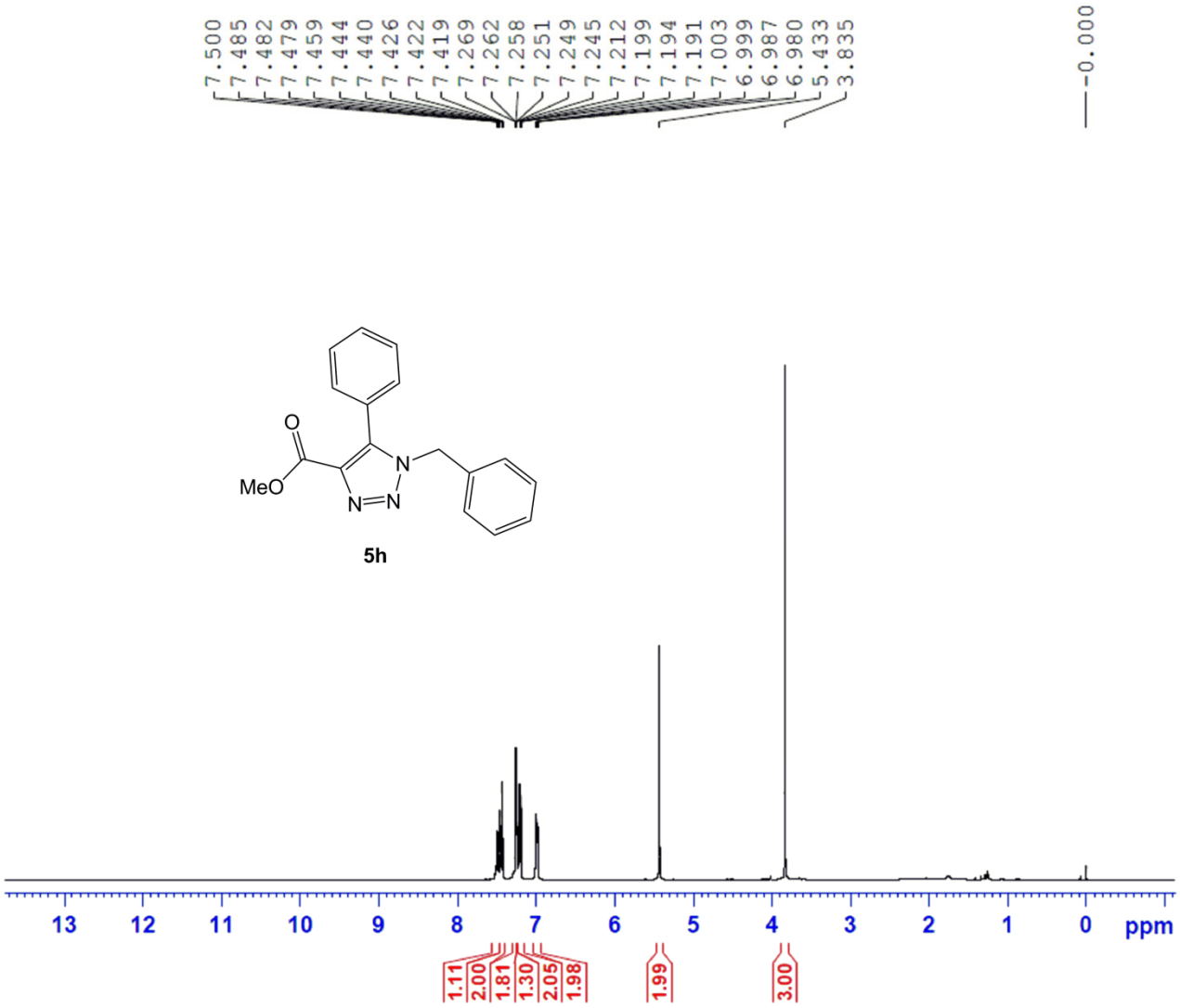
^{1}H NMR in $CDCl_3$ (400 MHz)



^{13}C NMR in $CDCl_3$ (100 MHz)

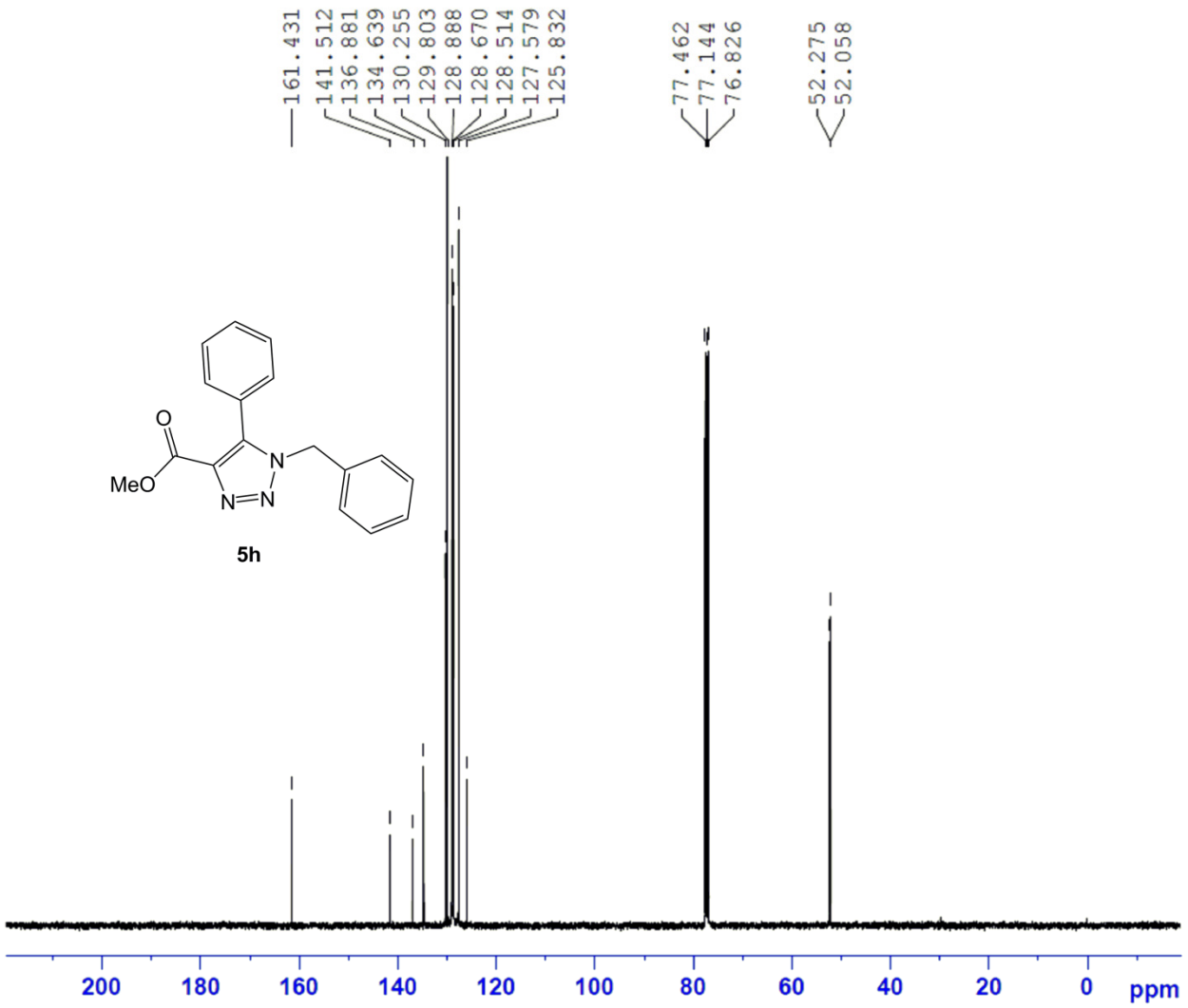


^{1}H NMR in $CDCl_3$ (400 MHz)

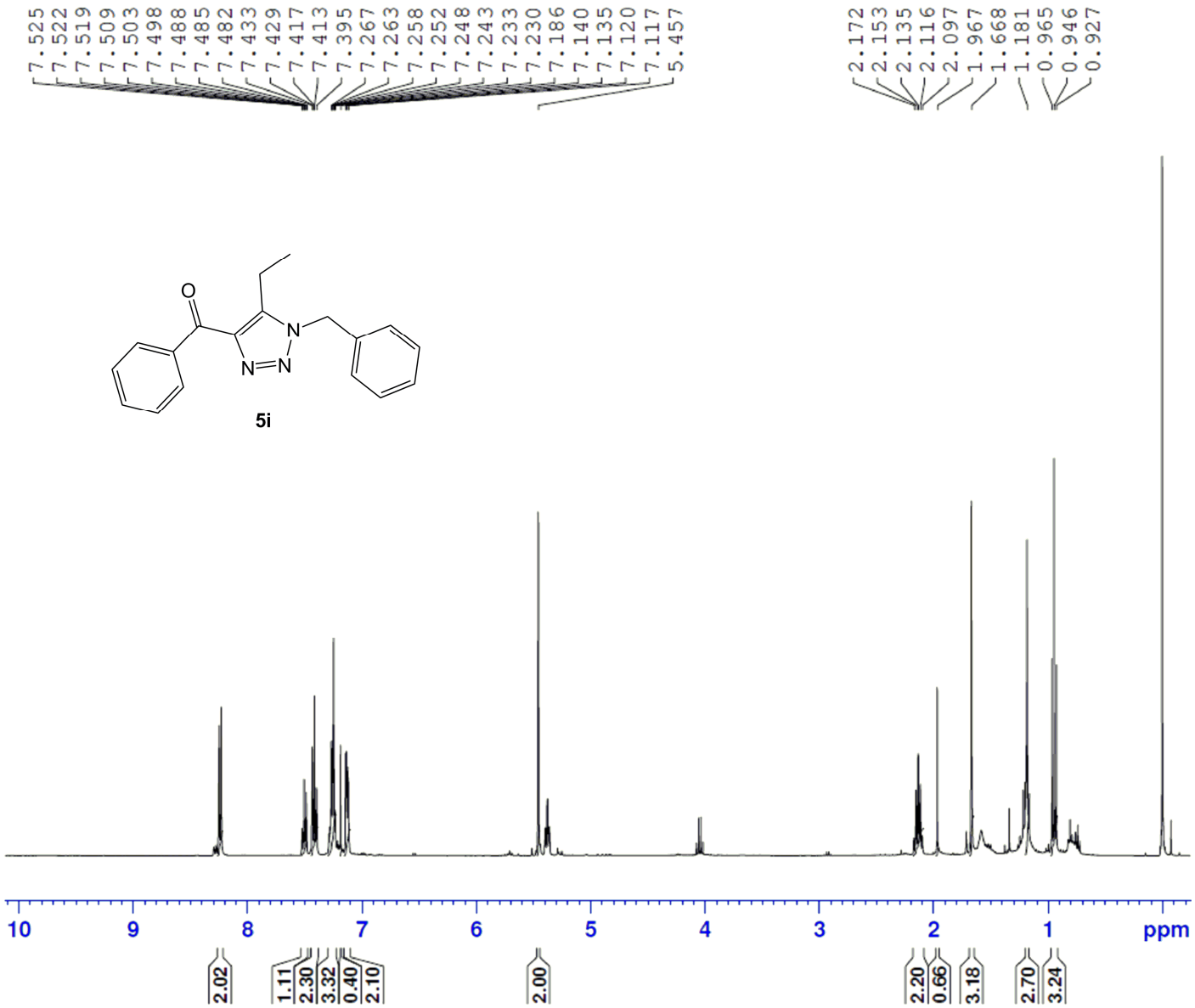


5h

^{13}C NMR in $CDCl_3$ (100 MHz)

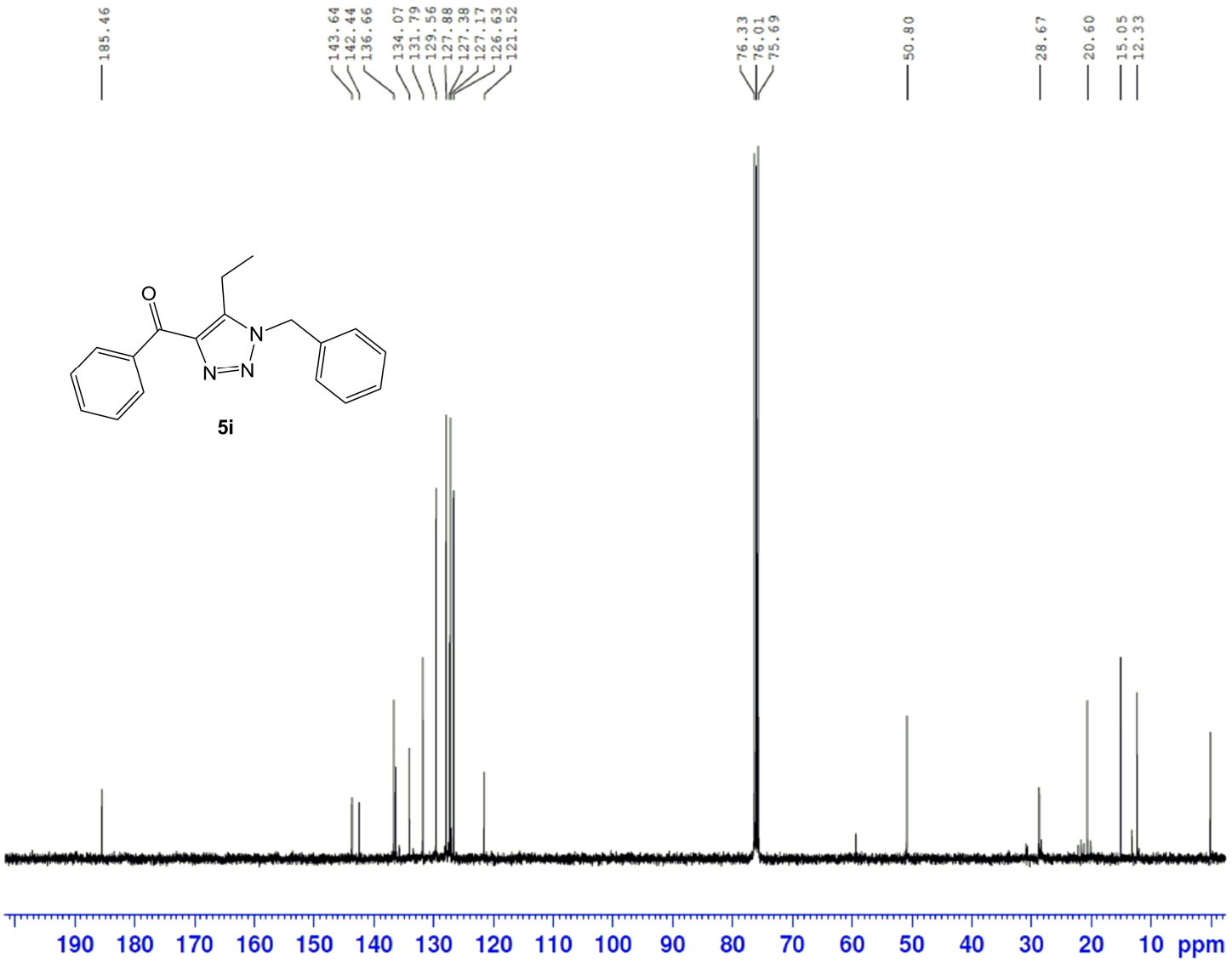


^{1}H NMR in $CDCl_3$ (400 MHz)

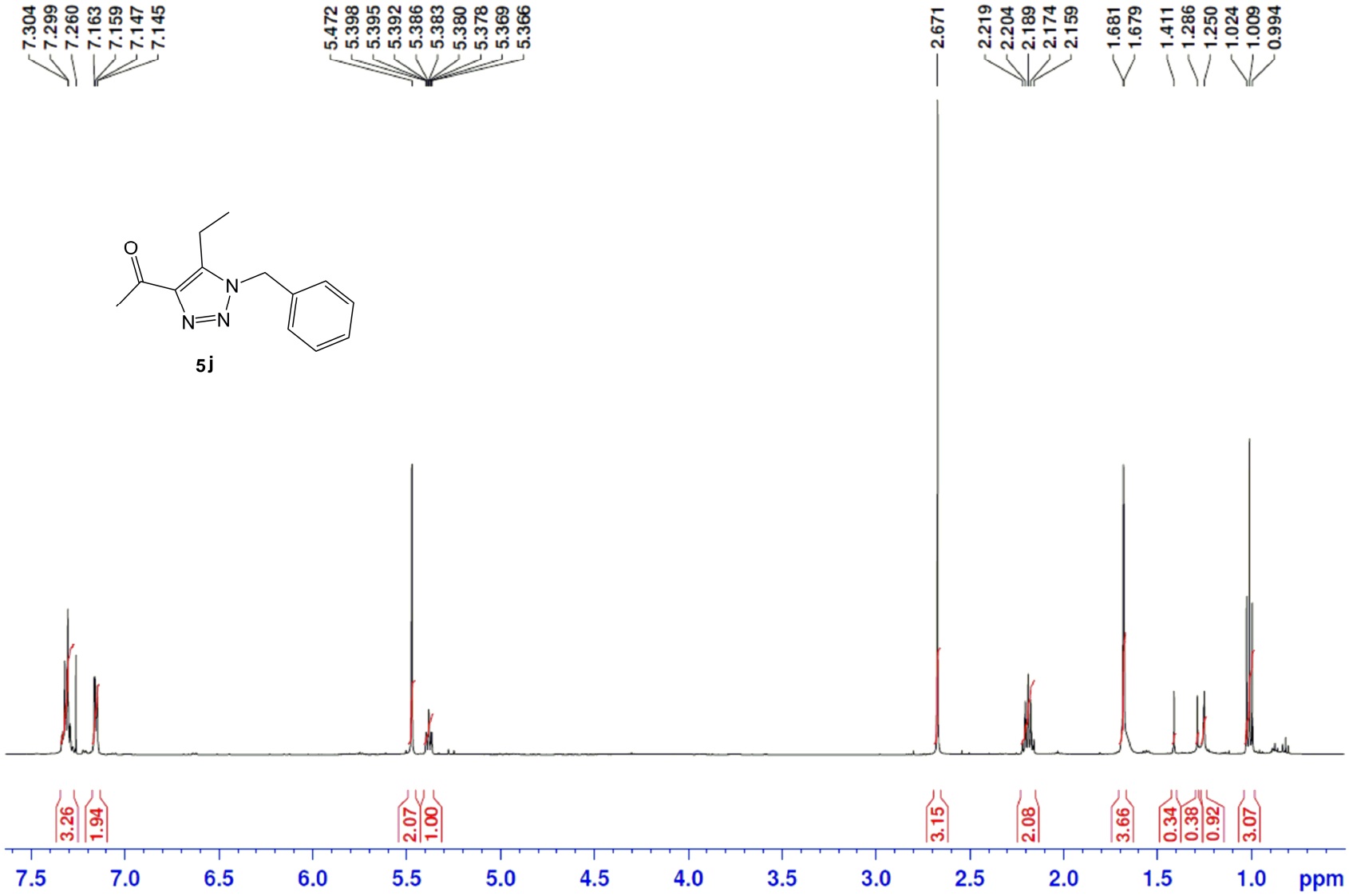


5i

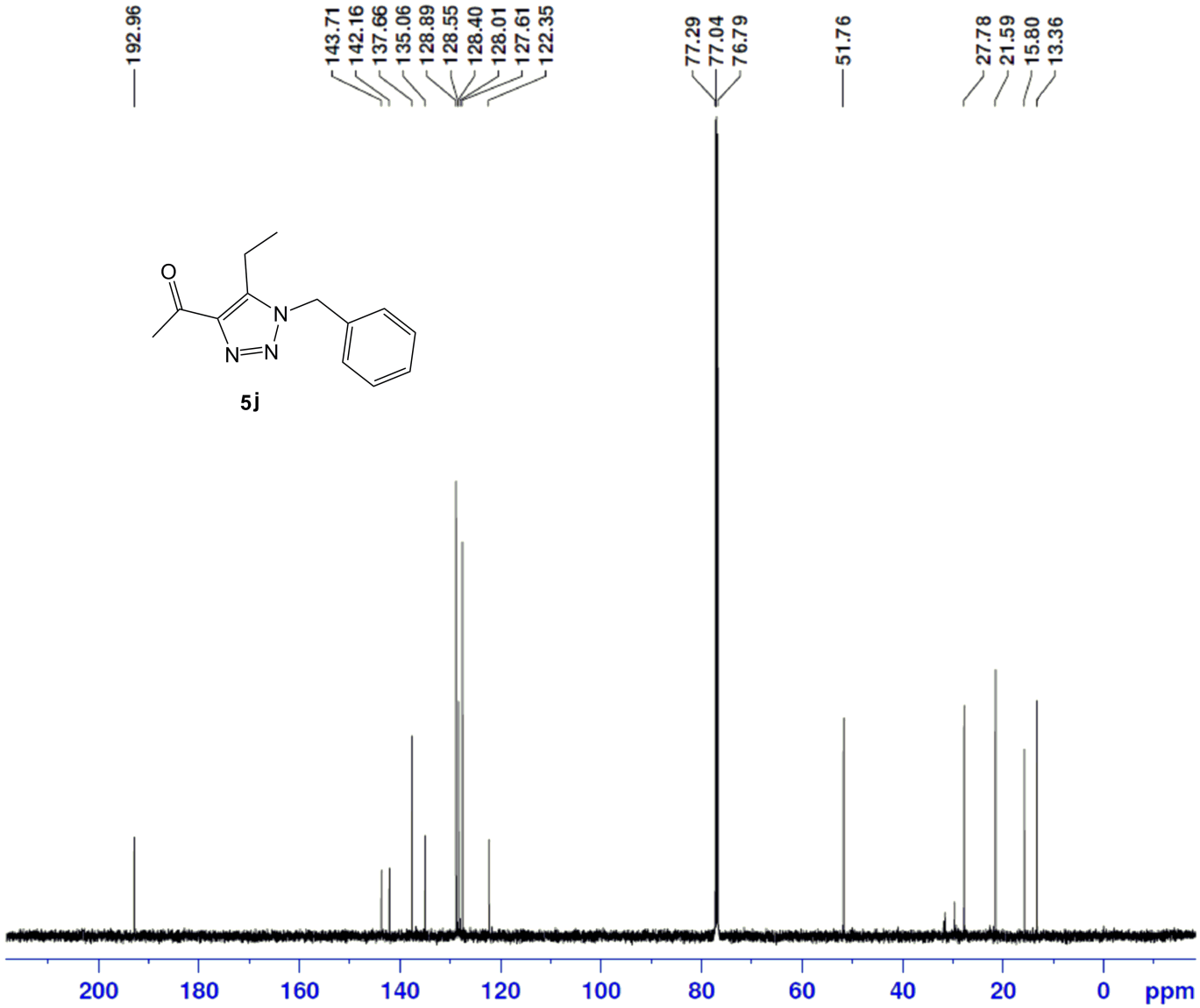
^{13}C NMR in $CDCl_3$ (100 MHz)



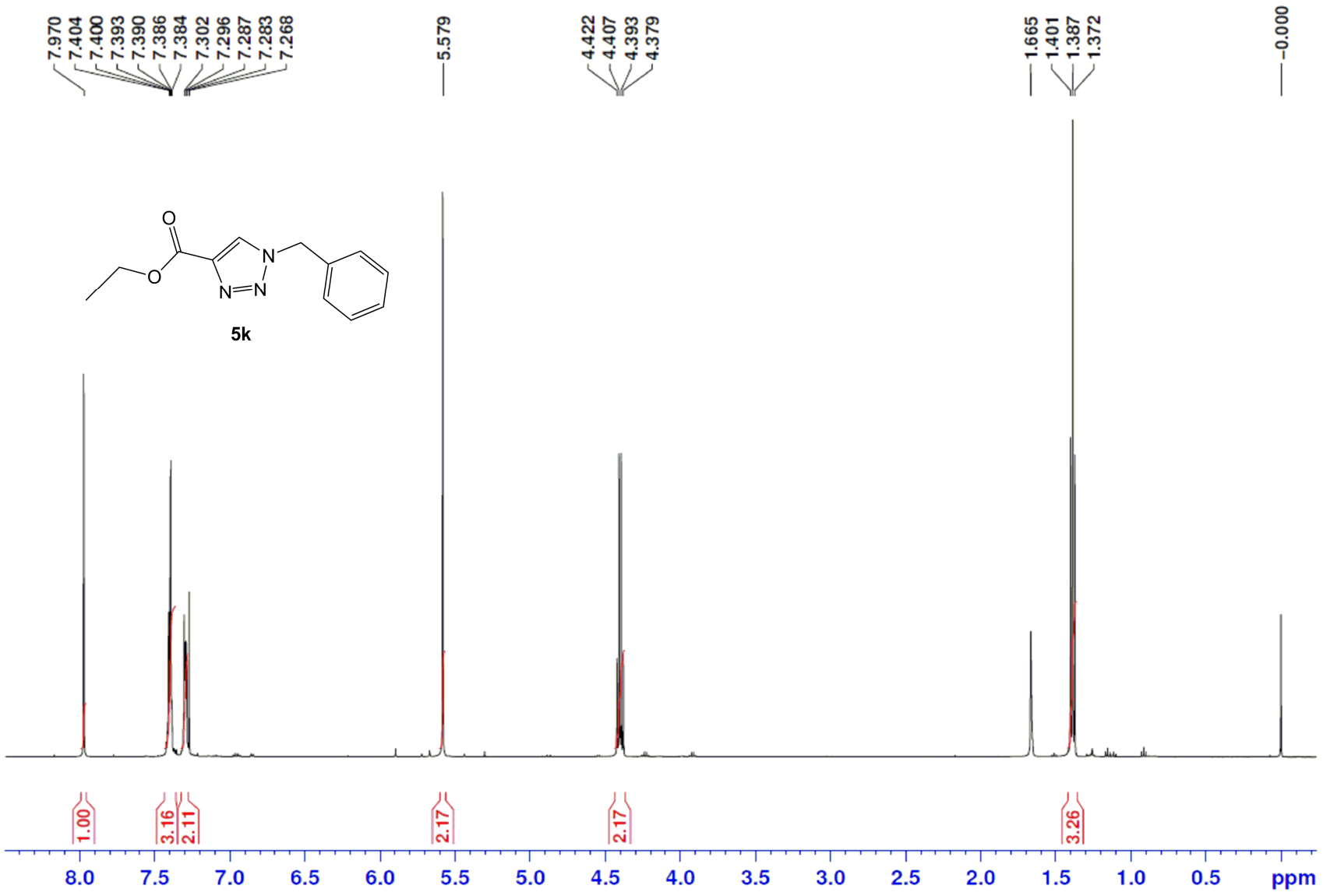
^{1}H NMR in $CDCl_3$ (500 MHz)



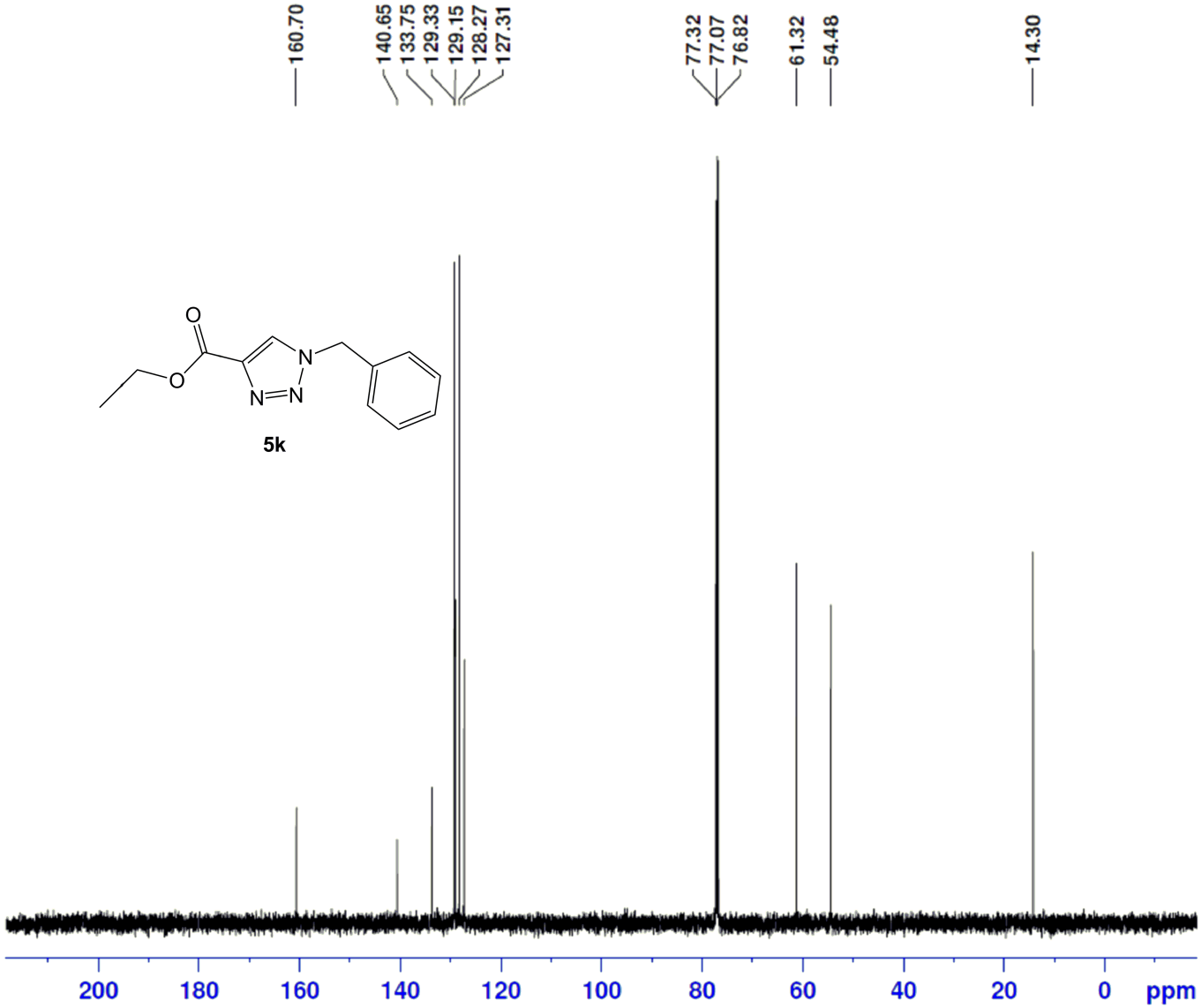
^{13}C NMR in $CDCl_3$ (125 MHz)

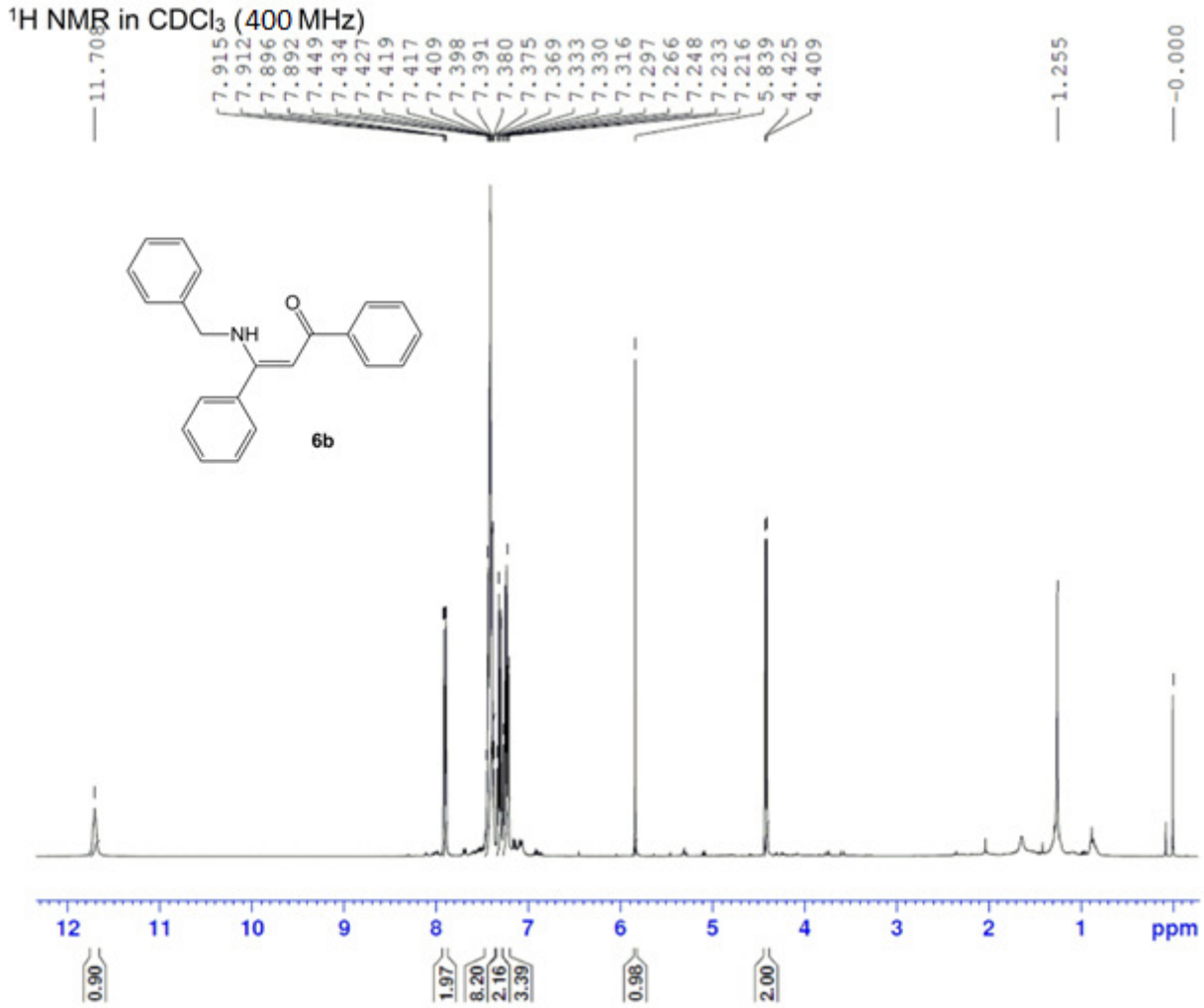


^{1}H NMR in $CDCl_3$ (500 MHz)



^{13}C NMR in $CDCl_3$ (125 MHz)




¹H NMR in CDCl₃ (400 MHz)
11.703
7.915
7.912
7.896
7.892
7.449
7.434
7.427
7.419
7.417
7.409
7.398
7.391
7.380
7.375
7.369
7.333
7.330
7.316
7.297
7.266
7.248
7.233
7.216
5.839
4.425
4.409
1.255
0.000
NH
O
6b
12
11
10
9
8
7
6
5
4
3
2
1
ppm
0.90
1.97
8.20
2.16
3.39
0.98
2.00

^{13}C NMR in $CDCl_3$ (100 MHz)