Supporting Information for:

Silver-Catalyzed Nucleophilic Substitution of Aminals with Ethyl Diazoacetate: A New Entry to β-Amino-α-Diazoesters

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CONTENTS

1. General experiment details and materials

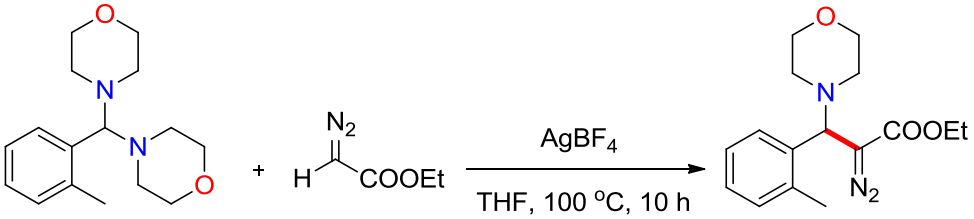
Experimental: All non-aqueous reactions and manipulations were used by standard Schlenk techniques. All solvents before used were dried and degassed by standard methods and stored under argon atmosphere. All reactions were monitored by TLC with silica gel-coated plates. NMR spectra were recorded on BRUKER Avance III (400 MHz) spectrometers. Chemical shifts were reported in parts per million (ppm) down field from TMS with the solvent resonance as the internal standard. Coupling constants (J) were reported in Hz and refered to apparent peak multiplications. High resolution mass spectra (HRMS) were recorded on Bruker MicroTOF-QII mass instrument(ESI). Aminals used here were known compounds and synthesized according to the reported methods.[1]

2. General procedure for the synthesis of *β*-amino-*α*-diazoesters

1a **2a** **3a**

$AgBF_4$ (4.8 mg, 0.025 mmol) was added to a 25 mL flame-dried Young-type tube in the glove box. Then aminal **1a** (0.5 mmol), THF (1.5 mL), diazo acetate **2a** (114.0 mg, 1.0 mmol) were added under argon atmosphere. The mixture was stirred at 100 ^{o}C for 2 hours. After cooling to room temperature, the solvent was removed under reduced pressure. The reaction mixture was purified by flash column chromatography on silica gel and eluted with EtOAc/hexane (1/30 – 1/10) to afford the desired product **3a**. The retention factor (Rf) data of **3a** is 0.35 **(**EtOAc/hexane =1/20**).**

3. Gram-scale reaction

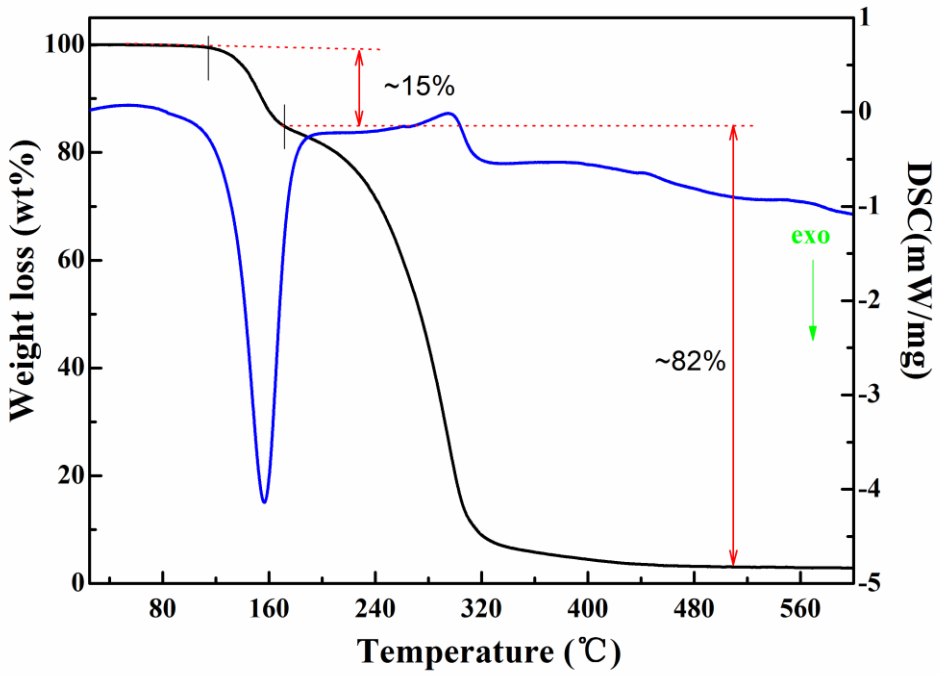


$AgBF_4$ (10. 0 mg, 0.005 mmol) was added to a 100 mL flame-dried Schlenk flask in the glove box. Aminal **1g** (1.38 g, 5.0 mmol), THF (10.0 mL), diazo acetate **2a** (1.14

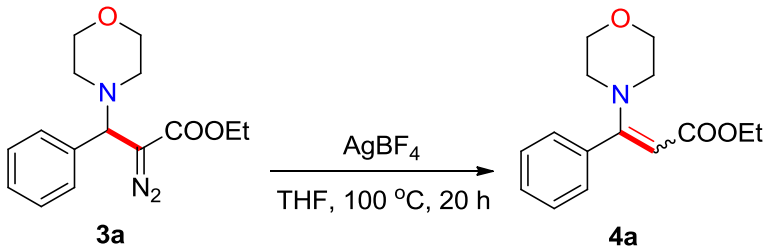
g, 10.0 mmol) were added under argon atmosphere. The mixture was stirred at 100 °C for 10 hours. After cooling to room temperature, the solvent was removed under reduced pressure. The reaction mixture was purified by flash column chromatography on silica gel and eluted with EtOAc/hexane (1/30 – 1/10) to afford the desired product **3g** (751.0 mg, 50% yield).

4. The graph for the thermogravimetric analysis of 3a

To gain the stability parameters of *β*-amino-*α*-diazo compounds we obtained here, a thermogravimetric analysis of **3a** was conducted.



5. The reactivity of 3a in the presence of $AgBF_4$



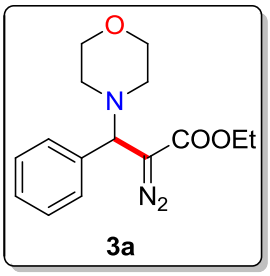
3a **4a**

$AgBF_4$ (4.8 mg, 0.025 mmol) was added to a 25 mL flame-dried Young-type tube in the glove box. Then **3a** (144.5 mg, 0.5 mmol), THF (1.5 mL) were added under argon atmosphere. The mixture was stirred at 100 °C for 20 hours. After cooling to room temperature, the solvent was removed under reduced pressure. The reaction mixture was purified by flash column chromatography on silica gel and eluted with

EtOAc/hexane (1/30 – 1/5) to afford the desired product **4a** 67.6 mg (52.0% isolated yield).

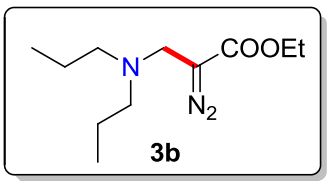
6. Experimental characterization data for products

ethyl 2-diazo-3-morpholino-3-phenylpropanoate (3a): The title compound was prepared according to the general procedure and purified by flash column chromatography on silica gel to give the yellow oil, 107 mg, 74 % yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.28 (t, *J* = 6.8 Hz, 3H), 2.44-2.55 (m, 4H), 3.67-3.76 (m, 4H), 4.25 (q, *J* = 7.2 Hz, 2H), 4.31 (s, 1H), 7.28-7.36 (m, 5H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 14.2, 51.3, 60.7, 63.8, 66.1, 126.8, 127.9, 128.9, 138.4, 165.2; HRMS (ESI) calcd. for $C_{15}H_{20}N_3O_3$ [M+H]: 290.1499, found: 290.1495.



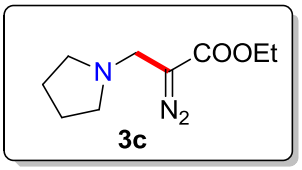
3a

ethyl 2-diazo-3-(dipropylamino)propanoate (3b): The title compound was prepared according to the general procedure and purified by flash column chromatography on silica gel to give the yellow oil, 97 mg, 85 % yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.89 (t, *J* = 7.2 Hz, 6H), 1.30 (t, *J* = 7.2 Hz, 3H), 1.42-1.51 (m, 4H), 2.43 (t, *J* = 7.2 Hz, 4H), 3.53 (s, 2H), 4.25 (q, *J* = 6.8 Hz, 2H); ^{13}C NMR (100 MHz, $CO(CD_3)_2$) δ 12.2, 14.9, 21.2, 48.8, 56.0, 61.2, 167.2; HRMS (ESI) calcd. for $C_{11}H_{22}N_3O_2$ [M+H]: 228.1707, found: 228.1700.



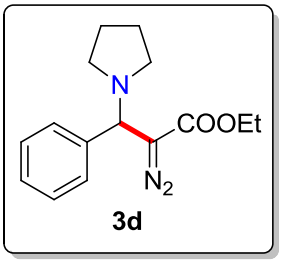
3b

ethyl 2-diazo-3-(pyrrolidin-1-yl)propanoate (3c): The title compound was prepared according to the general procedure and purified by flash column chromatography on silica gel to give the yellow oil, 79 mg, 81 % yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.30 (t, *J* = 7.2 Hz, 3H), 1.8 (s, 4H), 2.57 (s, 4H), 3.52 (s, 2H), 4.26 (q, *J* = 7.2 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.4, 23.5, 49.0, 52.9, 60.7, 166.8; HRMS (ESI) calcd. for $C_9H_{16}N_3O_2$ [M+H]: 198.1237, found: 198.1234.



3c

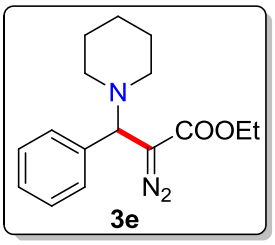
ethyl 2-diazo-3-phenyl-3-(pyrrolidin-1-yl)propanoate (3d): The title compound was prepared according to the general procedure and purified by



3d

flash column chromatography on silica gel to give the yellow oil, 105 mg, 77 % yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.27 (t, *J* = 7.2 Hz, 3H), 1.78-1.81 (m, 4H), 2.50-2.59 (m, 4H), 4.17-4.23 (m, 3H), 7.26-7.38 (m, 5H); ^{13}C NMR (100 MHz, $CO(CD_3)_2$) δ 14.8, 24.3, 53.3, 61.4, 64.2, 127.6, 128.7, 129.6, 142.1, 166.0; HRMS (ESI) calcd. for $C_{15}H_{20}N_3O_2$ [M+H]: 274.1550, found: 274.1547.

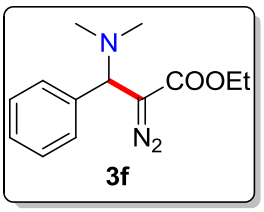
ethyl 2-diazo-3-phenyl-3-(piperidin-1-yl)propanoate (3e): The title compound was



3e

prepared according to the general procedure and purified by flash column chromatography on silica gel to give the yellow oil, 100 mg, 70 % yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.28 (t, *J* = 6.8 Hz, 3H), 1.44-1.48 (m, 2H), 1.53-1.61 (m, 4H), 2.40-2.50 (m, 4H), 4.25 (q, *J* = 6.8 Hz, 2H), 4.40 (s, 1H), 7.25-7.29 (m, 1H), 7.33-7.39 (m, 4H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 14.2, 24.0, 25.6, 51.5, 60.7, 64.0, 126.7, 127.6, 128.8, 138.9, 165.5; HRMS (ESI) calcd. for $C_{16}H_{22}N_3O_2$ [M+H]: 288.1707, found: 288.1701.

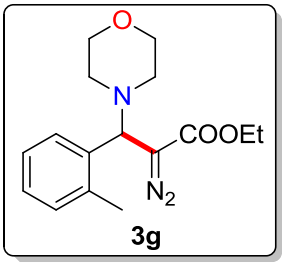
ethyl 2-diazo-3-(dimethylamino)-3-phenylpropanoate (3f): The title compound was



3f

prepared according to the general procedure and purified by flash column chromatography on silica gel to give the yellow oil, 91 mg, 74 % yield. ^{1}H NMR (400 MHz, DMSO-d_6) δ 1.21 (t, *J* = 7.2 Hz, 3H), 2.19 (s, 6H), 4.14-4.19 (m, 3H), 7.31-7.34 (m, 3H), 7.38-7.42 (m, 2H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 14.2, 43.0, 60.7, 64.7, 126.6, 127.8, 128.9, 139.5, 165.3; HRMS (ESI) calcd. for $C_{13}H_{18}N_3O_2$ [M+H]: 248.1394, found: 248.1389.

ethyl 2-diazo-3-morpholino-3-(o-tolyl)propanoate (3g): The title compound was

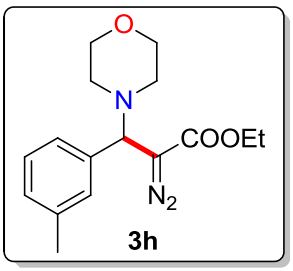


3g

prepared according to the general procedure and purified by flash column chromatography on silica gel to give a yellow solid, 132 mg, 87 % yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.28

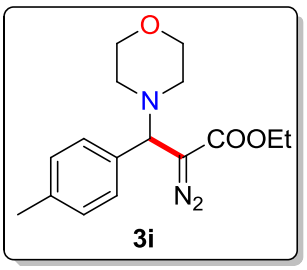
(t, *J* = 6.8 Hz, 3H), 2.35 (s, 3H), 2.46-2.52 (m, 4H), 3.66-3.76 (m, 4H), 4.25 (q, *J* = 6.8 Hz, 2H), 4.46 (s, 1H), 7.16-7.22 (m, 3H), 7.41-7.44 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.5, 19.5, 51.8, 60.3, 60.9, 67.0, 126.2, 126.3, 127.4, 131.0, 136.2, 136.8, 166.4; HRMS (ESI) calcd. for $C_{16}H_{22}N_3O_3$ [M+H]: 304.1656, found: 304.1657.

ethyl 2-diazo-3-morpholino-3-(m-tolyl)propanoate (3h): The title compound was prepared according to the general procedure and purified by flash column chromatography on silica gel to give the yellow oil, 119 mg, 79 % yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.28 (t, *J* = 7.2 Hz, 3H), 2.35 (s, 3H), 2.43-2.53 (m, 4H), 3.67-3.76 (m, 4H), 4.19-4.25 (m, 3H), 7.09-7.11 (m, 1H), 7.15-7.16 (m, 2H), 7.22-7.25 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.5, 21.5, 51.7, 60.9, 64.5, 67.0, 124.1, 127.8, 128.7, 128.8, 138.6, 138.7, 166.3; HRMS (ESI) calcd. for $C_{16}H_{22}N_3O_3$ [M+H]: 304.1656, found: 304.1655.



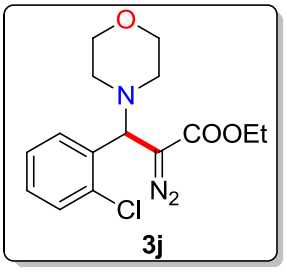
3h

ethyl 2-diazo-3-morpholino-3-(m-tolyl)propanoate (3i): The title compound was prepared according to the general procedure and purified by flash column chromatography on silica gel to give the yellow oil, 123 mg, 82 % yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.28 (t, *J* = 7.2 Hz, 3H), 2.34 (s, 3H), 2.43-2.53 (m, 4H), 3.66-3.75 (m, 4H), 4.24 (q, *J* = 7.2 Hz, 2H), 4.28 (s, 1H), 7.17 (d, *J* = 8.0 Hz, 2H), 7.25 (d, *J* = 8.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.5, 21.1, 51.7, 60.9, 64.3, 67.0, 127.0, 129.6, 135.7, 137.7, 166.4; HRMS (ESI) calcd. for $C_{16}H_{22}N_3O_3$ [M+H]: 304.1656, found: 304.1654.



3i

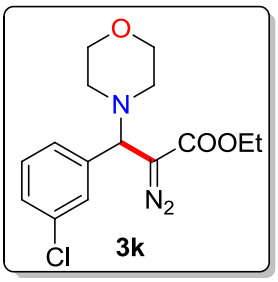
ethyl 3-(2-chlorophenyl)-2-diazo-3-morpholinopropanoate (3j): The title compound was prepared according to the general procedure and purified by flash column chromatography on silica gel to give a yellow solid, 75 mg, 46 % yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.29



3j

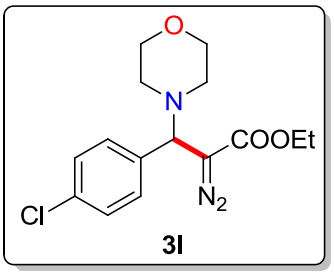
(t, *J* = 7.2 Hz, 3H), 2.47-2.59 (m, 4H), 3.67-3.76 (m, 4H), 4.27 (q, *J* = 7.2 Hz, 2H), 4.78 (s, 1H), 7.21-7.31 (m, 2H), 7.39-7.41 (m, 1H), 7.51-7.54 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.4, 51.6, 60.8, 61.0, 67.0, 127.0, 128.2, 128.8 130.2, 133.9, 136.0, 165.9; HRMS (ESI) calcd. for $C_{15}H_{18}ClN_3NaO_3$ [M+Na]: 346.0929, found: 346.0939.

ethyl 3-(3-chlorophenyl)-2-diazo-3-morpholinopropanoate (3k): The title compound was prepared according to the general procedure and purified by flash column chromatography on silica gel to give the yellow oil, 94 mg, 58 % yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.29 (t, *J* = 7.2 Hz, 3H), 2.44-2.55 (m, 4H), 3.68-3.77 (m, 4H), 4.26 (q, *J* = 6.8 Hz, 2H), 4.31 (s, 1H), 7.24-7.32 (m, 3H), 7.37 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.4, 51.6, 61.1, 64.0, 66.9, 125.3, 127.2, 128.2, 130.3, 134.9, 140.8, 166.1; HRMS (ESI) calcd. for $C_{15}H_{18}ClN_3NaO_3$ [M+Na]: 346.0929, found: 346.0937.



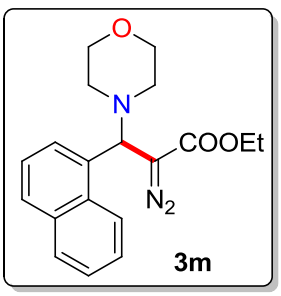
3k

ethyl 3-(4-chlorophenyl)-2-diazo-3-morpholinopropanoate (3l): The title compound was prepared according to the general procedure and purified by flash column chromatography on silica gel to give the yellow oil, 99 mg, 62 % yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.29 (t, *J* = 7.2 Hz, 3H), 2.43-2.54 (m, 4H), 3.67-3.76 (m, 4H), 4.25 (q, *J* = 7.2 Hz, 2H), 4.32 (s, 1H), 7.30-7.35 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.4, 51.6, 61.1, 63.9, 66.9, 128.5, 129.2, 133.7, 137.2, 166.1; HRMS (ESI) calcd. for $C_{15}H_{18}ClN_3NaO_3$ [M+Na]: 346.0929, found: 346.0934.



3l

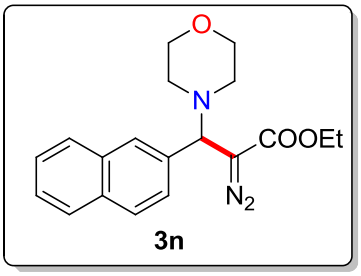
ethyl 2-diazo-3-morpholino-3-(naphthalen-1-yl)propanoate (3m): The title compound was prepared according to the general procedure and purified by flash column chromatography on silica gel to give the yellow oil, 131 mg, 77 % yield. ^{1}H NMR (400 MHz,



3m

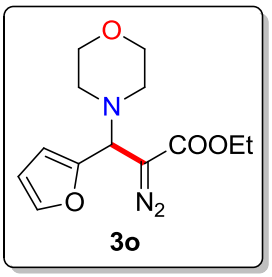
$CDCl_3$) δ 1.25 (m, 3H), 2.62 (m, 4H), 3.70-3.81 (m, 4H), 4.22-4.26 (m, 2H), 5.05 (s, 1H), 7.45-7.56 (m, 3H), 7.69-7.71 (m, 1H), 7.78-7.80 (m, 1H), 7.86-7.89 (m, 1H), 8.21-8.23 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.5, 52.0, 60.7, 61.0, 67.1, 123.1, 124.4, 125.3, 125.9, 126.5, 128.4, 128.9, 131.3, 133.9, 134.2, 166.2; HRMS (ESI) calcd. for $C_{19}H_{22}N_3O_3$ [M+H]: 340.1656, found: 340.1640.

ethyl 2-diazo-3-morpholino-3-(naphthalen-2-yl)propanoate (3n): The title compound was prepared according to the general procedure and purified by flash column chromatography on silica gel to give the yellow oil, 140 mg, 82 % yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.28 (t, *J* = 7.2 Hz, 3H), 2.48-2.60 (m, 4H), 3.69-3.79 (m, 4H), 4.19-4.25 (m, 2H), 4.47 (s, 1H), 7.44-7.51 (m, 3H), 7.81-7.85 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.5, 51.9, 61.0, 64.7, 67.1, 124.7, 126.2, 126.3, 126.4, 127.7, 128.0, 128.9, 133.2, 133.4, 136.3, 166.3; HRMS (ESI) calcd. for $C_{19}H_{22}N_3O_3$ [M+H]: 340.1656, found: 340.1663.



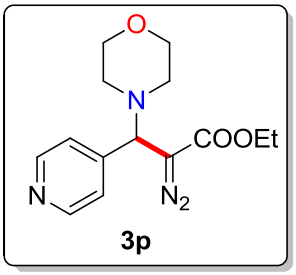
3n

ethyl 2-diazo-3-(furan-2-yl)-3-morpholinopropanoate (3o): The title compound was prepared according to the general procedure and purified by flash column chromatography on silica gel to give the yellow oil, 56 mg, 40 % yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.30 (t, *J* = 6.8 Hz, 3H), 2.38-2.44 (m, 2H), 2.57-2.62 (m, 2H), 3.67-3.76 (m, 4H), 4.26 (q, *J* = 7.2 Hz, 2H), 4.49 (s, 1H), 6.30-6.35 (m, 2H), 7.40 (d, *J* = 0.8 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.5, 51.0, 58.6, 61.1, 66.9, 108.6, 110.2, 142.6, 151.0, 166.3; HRMS (ESI) calcd. for $C_{13}H_{17}N_3NaO_4$ [M+Na]: 302.1111, found: 302.1096.



3o

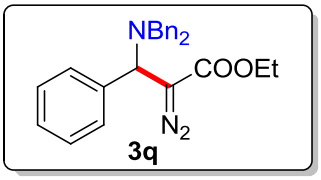
ethyl 2-diazo-3-morpholino-3-(pyridin-4-yl)propanoate (3p): The title compound was prepared according to the general procedure and purified



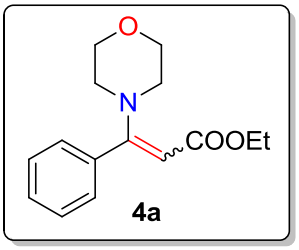
3p

by flash column chromatography on silica gel to give the yellow oil, 20 mg, 13 % yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.30 (t, *J* = 7.2 Hz, 3H), 2.44-2.60 (m, 4H), 3.69-3.78 (m, 4H), 4.27 (q, *J* = 6.8 Hz, 2H), 4.39 (s, 1H), 7.32 (d, *J* = 6.0 Hz, 2H), 8.62 (d, *J* = 5.6 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.4, 51.3, 61.3, 63.6, 66.9, 122.2, 147.5, 150.6, 165.9; HRMS (ESI) calcd. for $C_{14}H_{19}N_4O_3$ [M+H]: 291.1452, found: 291.1463.

ethyl 2-diazo-3-(dibenzylamino)-3-phenylpropanoate (3q): The title compound was prepared according to the general procedure and purified by flash column chromatography on silica gel to give the yellow oil, 156 mg, 78 % yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.26 (t, *J* = 6.8 Hz, 3H), 3.64-3.73 (m, 4H), 4.15-4.29 (m, 2H), 4.92 (s, 1H), 7.19-7.32 (m, 7H), 7.37-7.41 (m, 6H), 7.57 (d, *J* = 7.6 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.5, 55.0, 58.7, 60.9,, 61.0, 127.0, 127.3, 127.7, 128.3, 128.5, 129.0, 139.1, 139.3, 166.9; HRMS (ESI) calcd. for $C_{25}H_{26}N_3O_2$ [M+H]: 400.2020, found: 400.2001.



ethyl 3-morpholino-3-phenylacrylate (4a): ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.08 (t, *J* = 6.8 Hz, 3H), 3.06 (t, *J* = 4.8 Hz, 4H), 3.69 (t, *J* = 4.8 Hz, 4H), 3.94 (q, *J* = 7.2 Hz, 2H), 4.97 (s, 1H), 7.24-7.26 (m, 2H), 7.39-7.40 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.3, 48.1, 58.8, 66.5, 90.8, 128.3, 128.7, 128.9, 136.0, 163.5, 167.8; HRMS (ESI) calcd. for $C_{15}H_{19}NO_3Na$ [M+Na]: 284.1257, found: 284.1265.



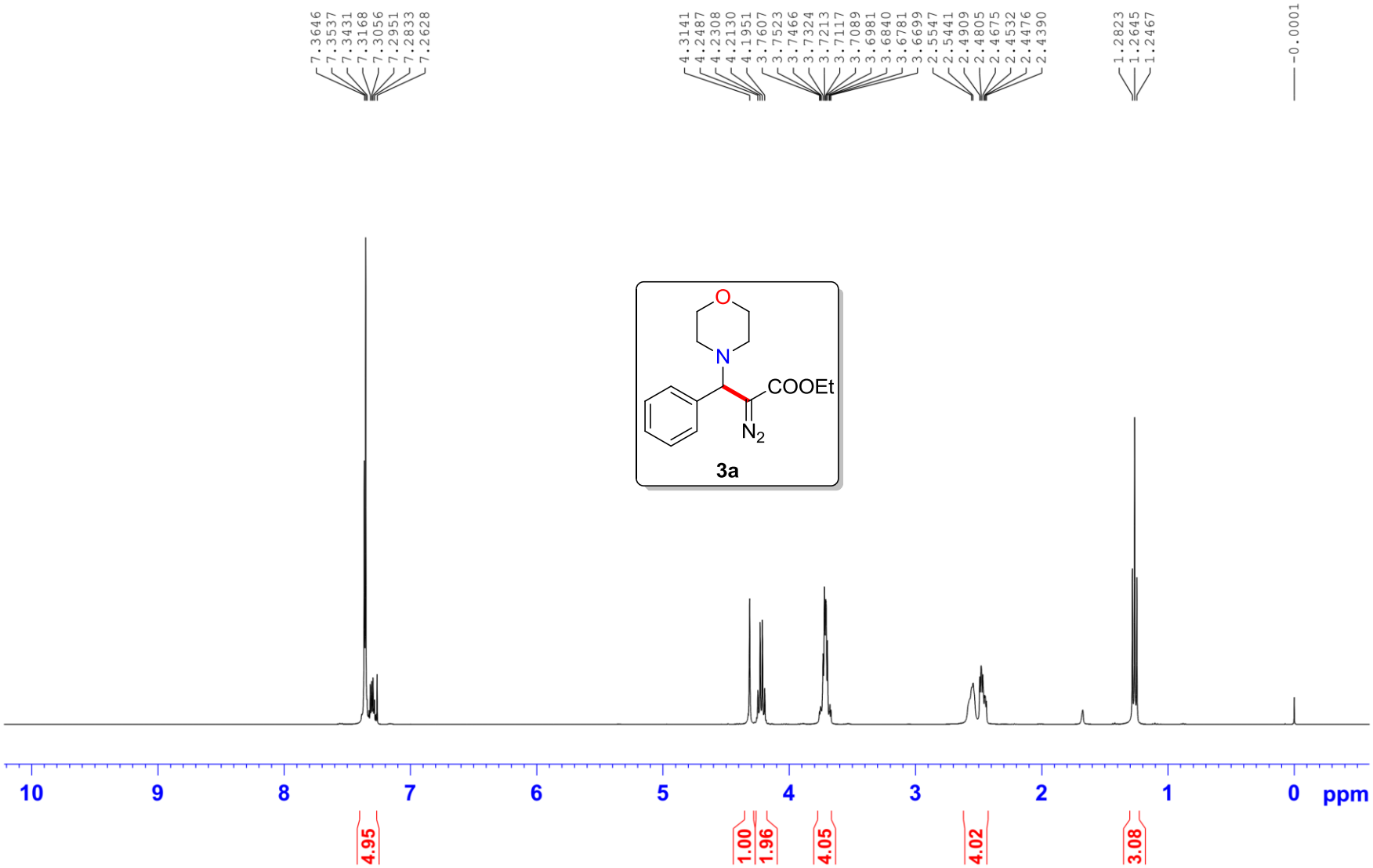
References:

1. (a) H. Heaney, G. Papageorgiou, R. F. Wilkins, *Tetrahedron*, 1997, **53**, 2941; (*b*) T. Rosenau, A. Potthast, P. Kosma, *Tetrahedron*, 2004, **60**, 301; (c) M. Terada.; K. Machioka, K. Sorimachi, *Angew. Chem.*, 2009, **121**, 2591. (d) W. Zhao, L. Huang, Y. Guan and W. D. Wulff, *Angew. Chem., Int. Ed.*, 2014, **53**, 3436. (e) B. Hatano, K. Nagahashi and T. Kijima, *J. Org. Chem.*, 2008, **73**, 9188. (f) D. Mohammad Karimi, S. Mohammad Reza, Phosphorus, Sulfur and Silicon and the Related

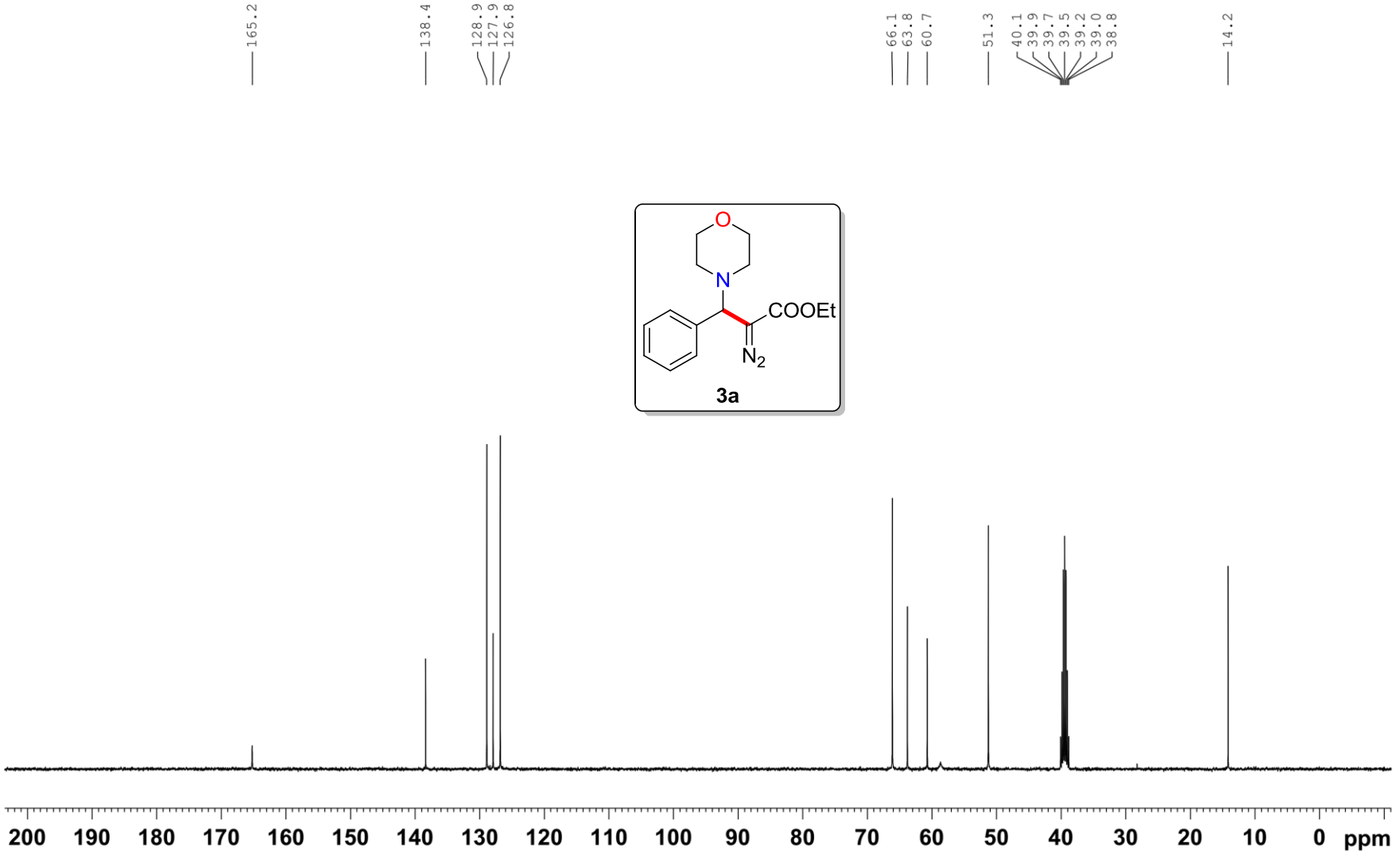
Elements, 2004, *179*, 89. (g) R. A. Henry, W. M. Dehn, J. Am. Chem. Soc., 1949, **71**, 2271. (h) M. F. Sansone, T. Koyanagi, D. E. Przybyla, R. W. Nagorski, Tetrahedron Lett., 2010, **51**, 6031. (i) S. Ryozo, M. Hiroo, S. Kiyotomo, Synthesis, 1993, 705.

7. Copies for ^{1}H NMR and ^{13}C NMR of products

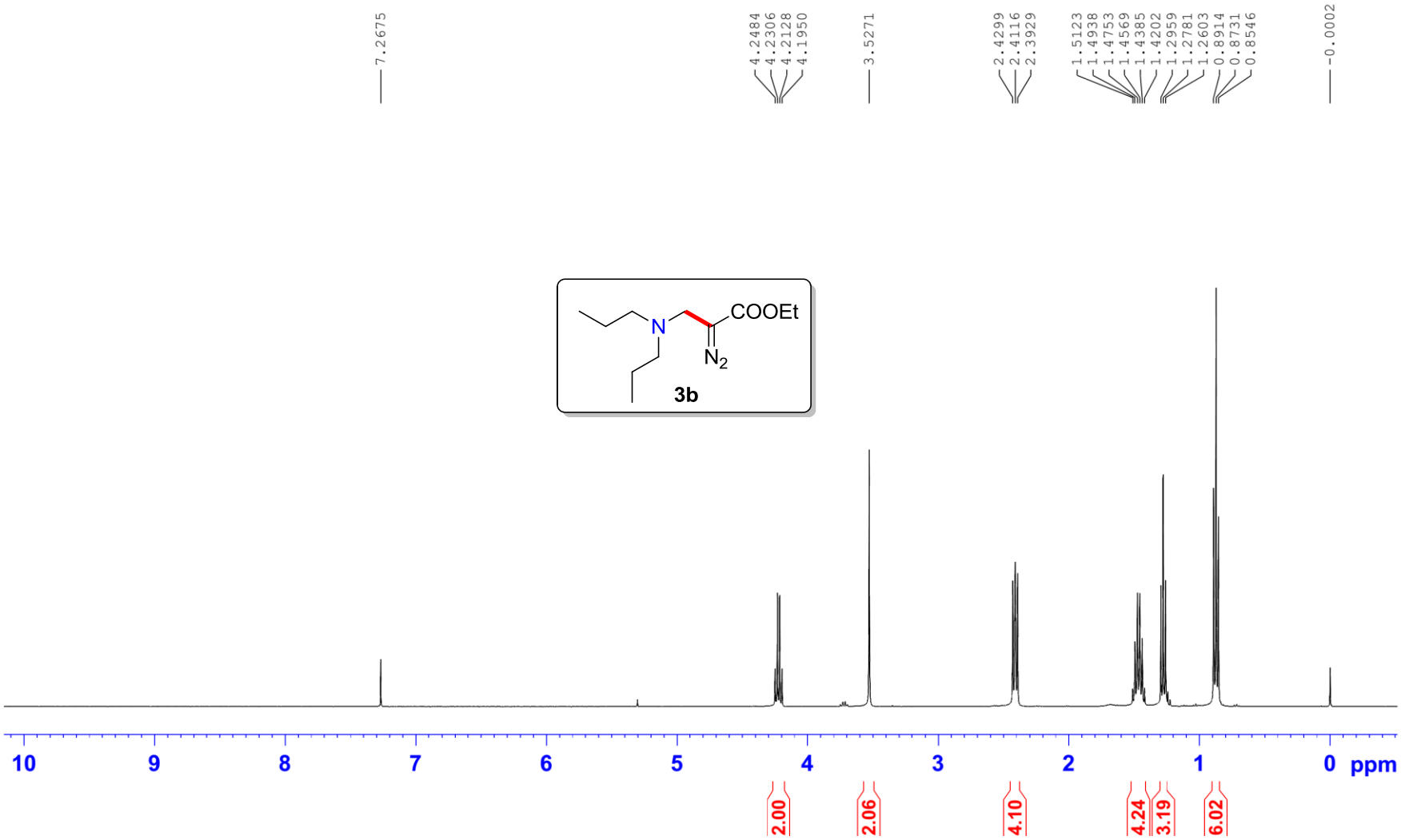
LJW-X15Y09-1-HNMR



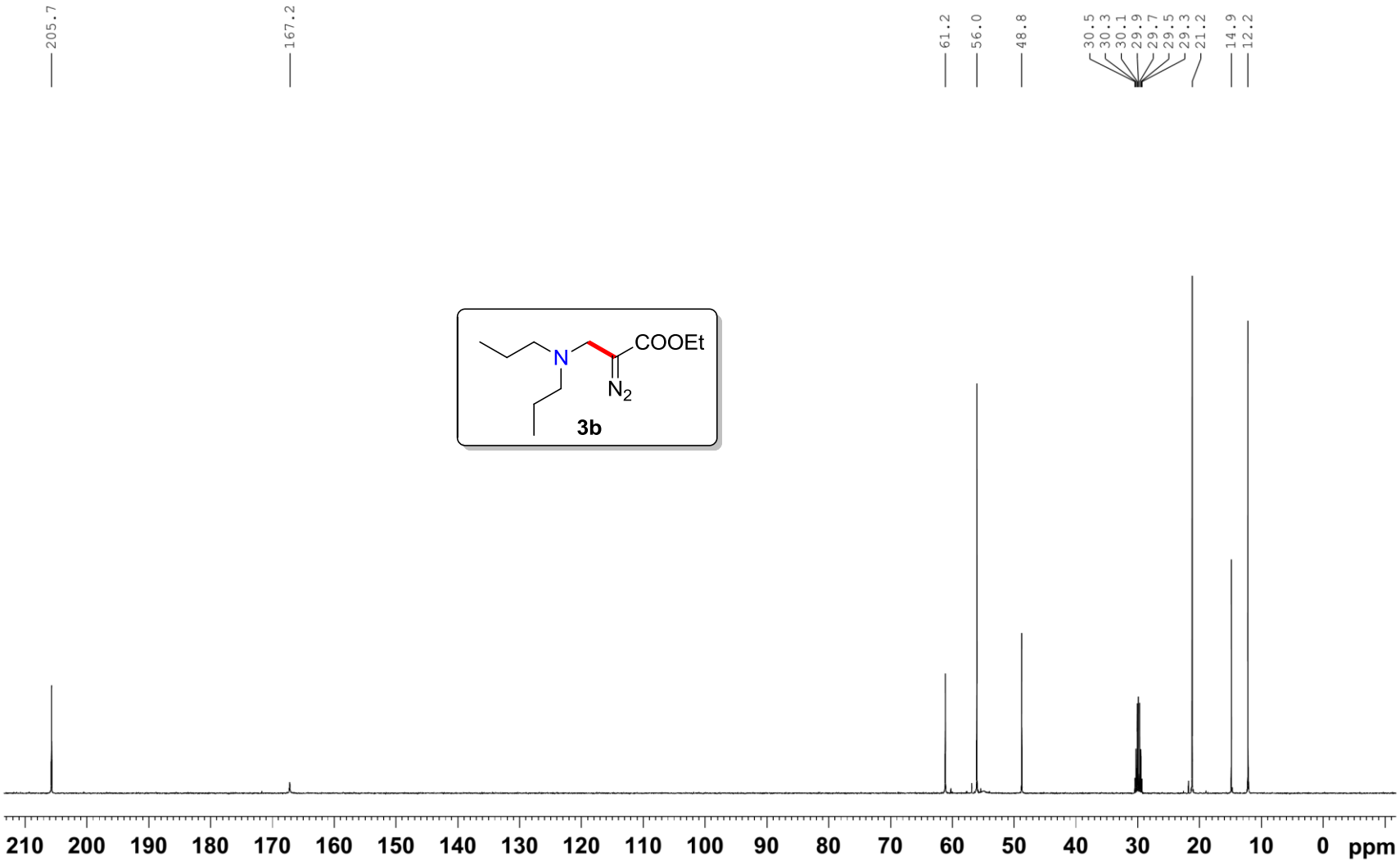
LJW-X15Z16-6-CNMR DMSO-d6



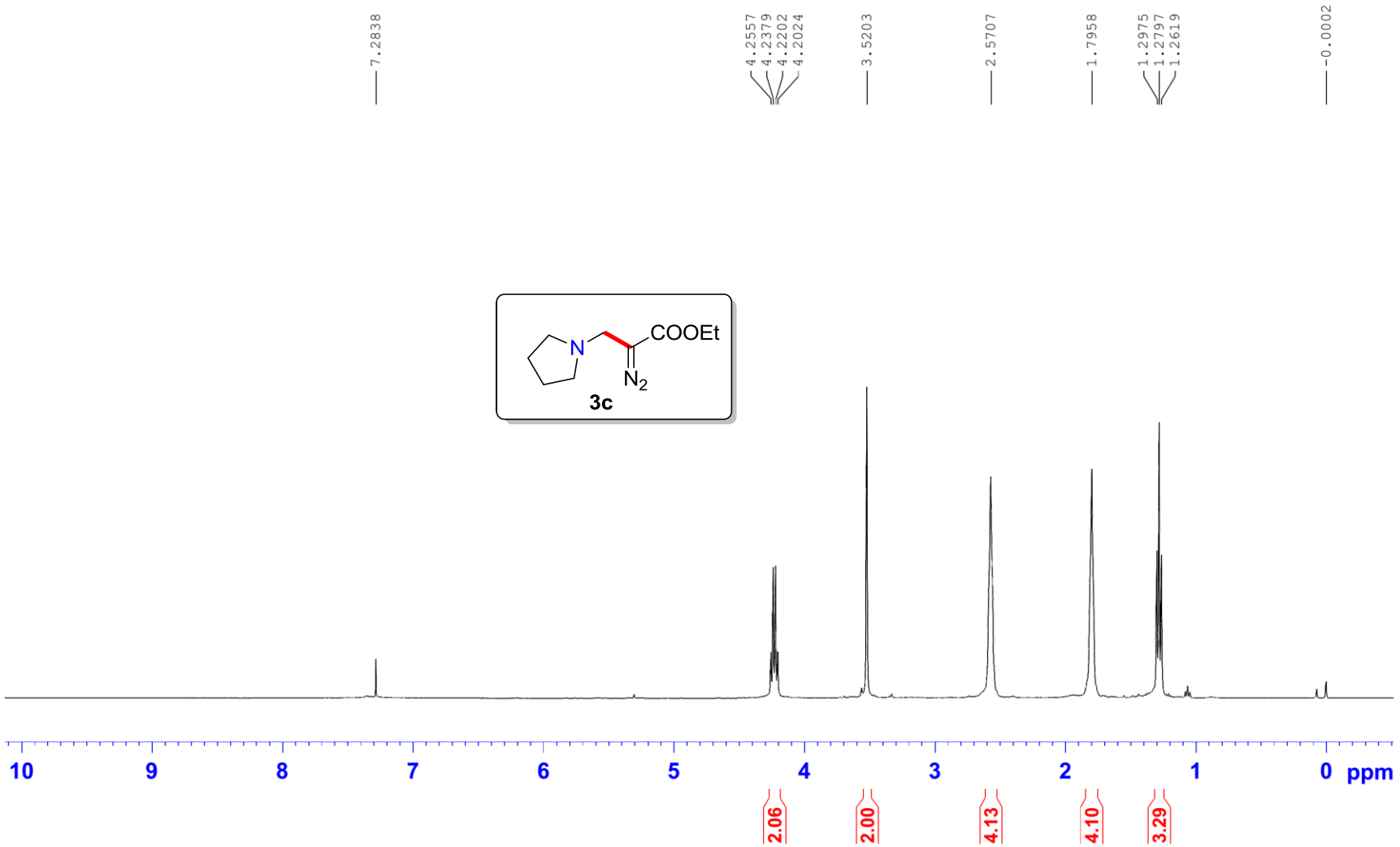
LJW-X15Y26-2-HNMR CDCl3



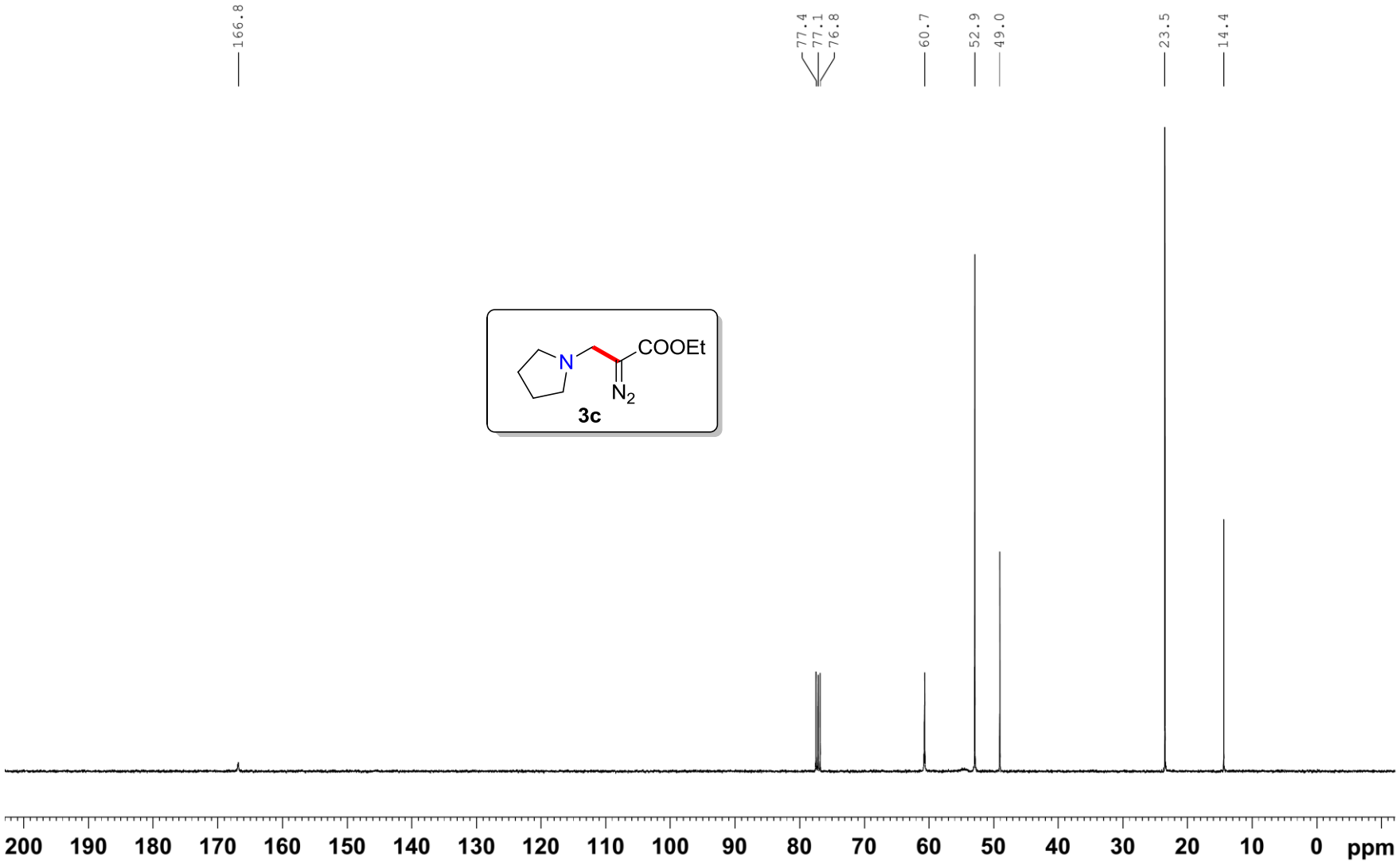
LJW-X15Z23-1-CNMR acetone-d6



LJW-X15Y30-3-HNMR



LJW-X15Z24-4-CNMR CDCl3



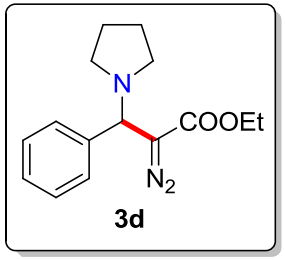
LJW-X15Y09-2-HNMR CDCl3

7.3818
7.3775
7.3606
7.3578
7.3510
7.3491
7.3438
7.3317
7.3274
7.3126
7.2938
7.2896
7.2850
7.2793
7.2722
7.2649
7.2591
7.2553

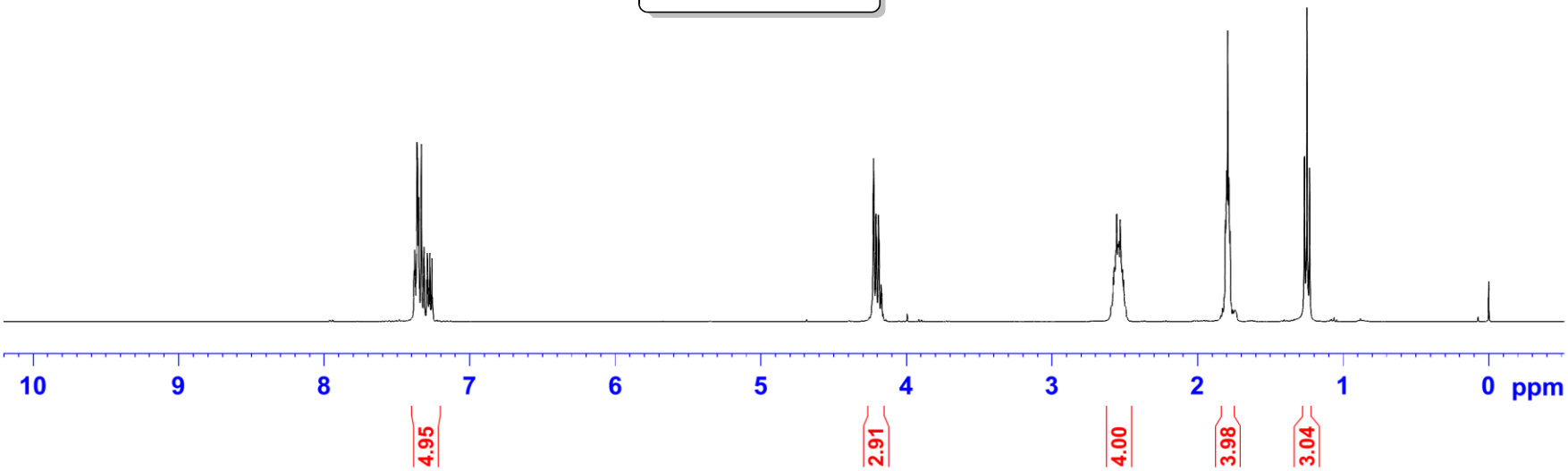
4.2258
4.2102
4.2058
4.1924
4.1881
4.1746
4.1704

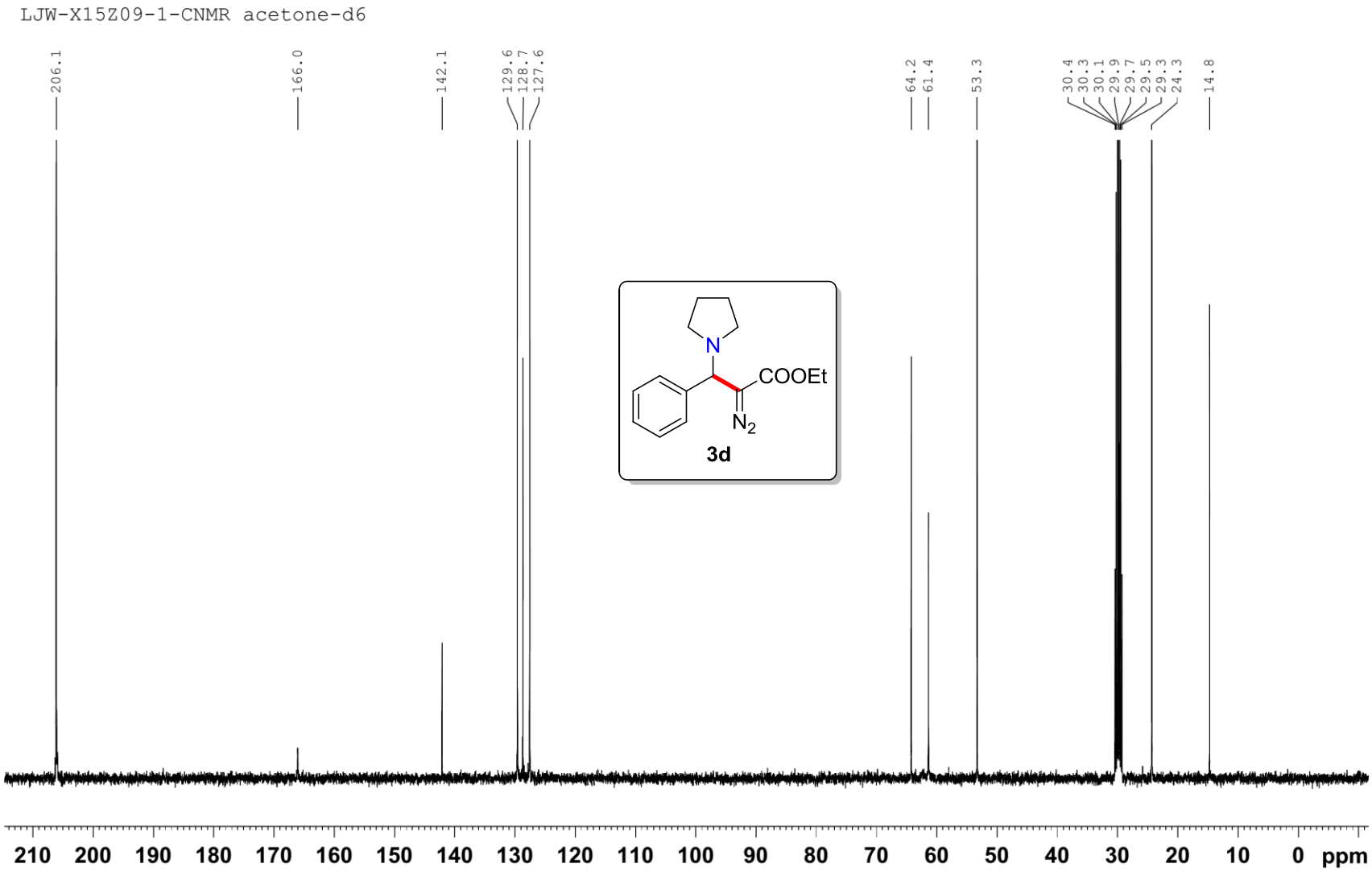
2.5909
2.5788
2.5725
2.5557
2.5478
2.5394
2.5314
2.5151
2.5084
2.4967
1.8096
1.8006
1.7930
1.7854
1.7764
1.2661
1.2484
1.2306

-0.0002

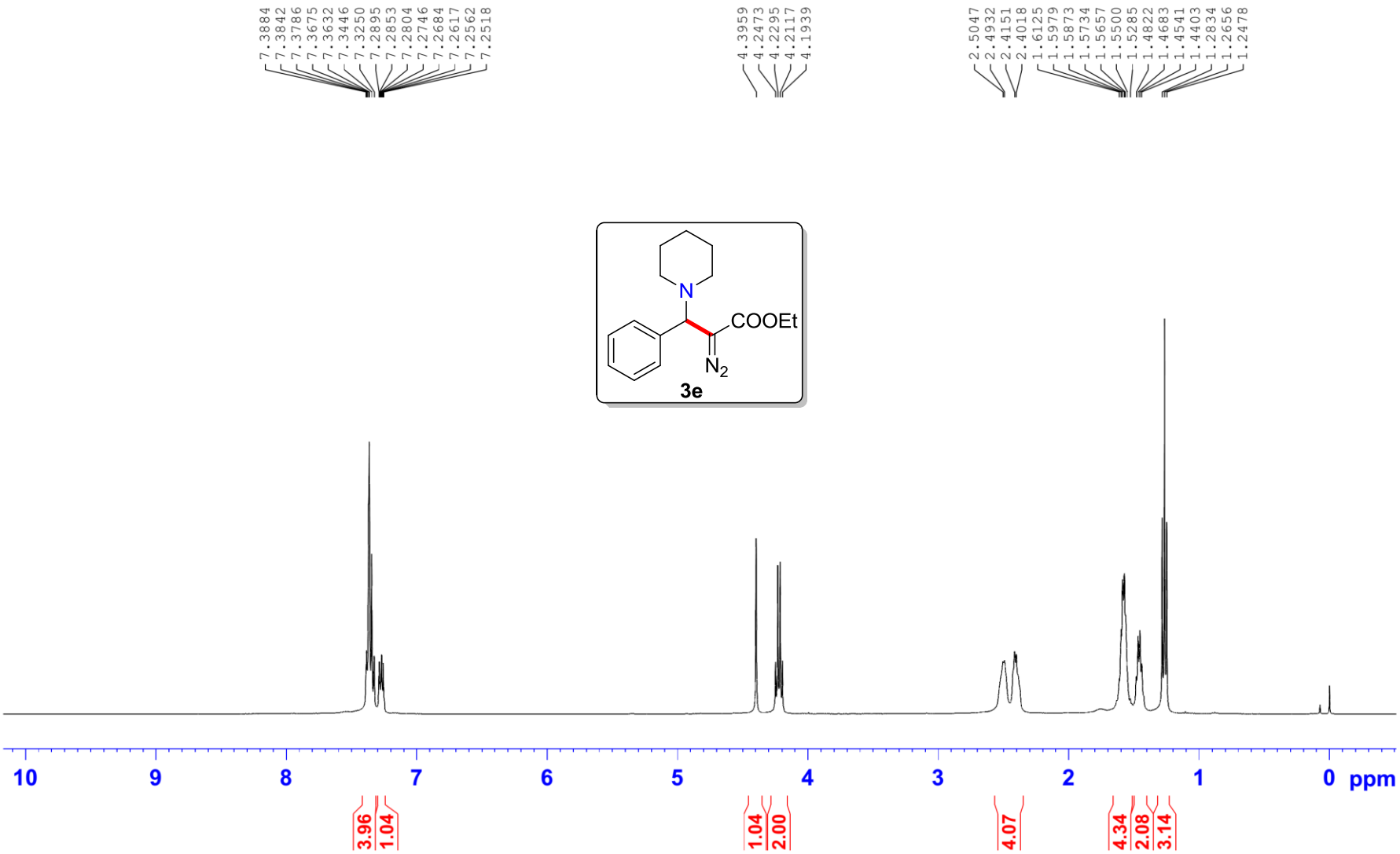


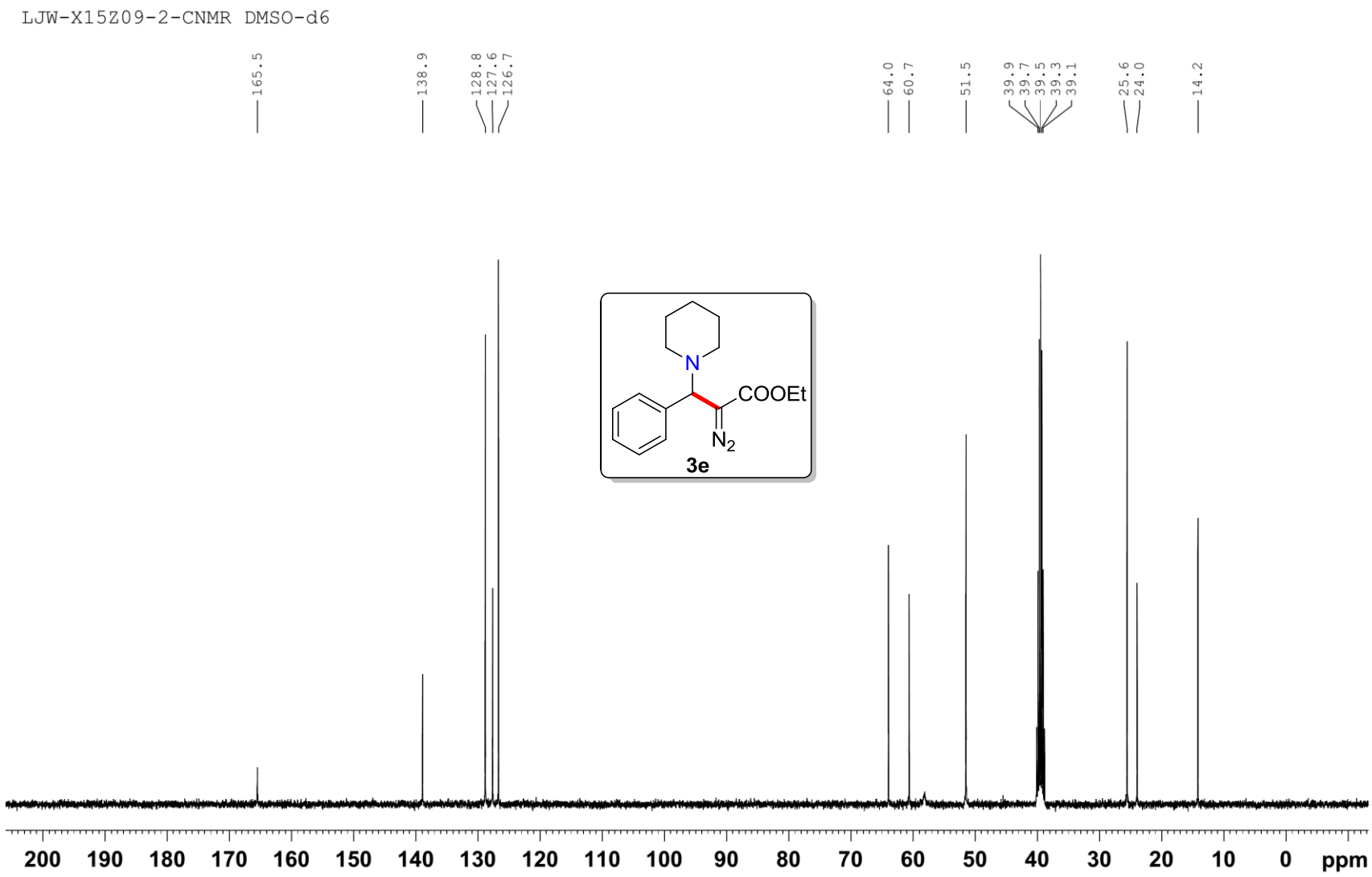
3d



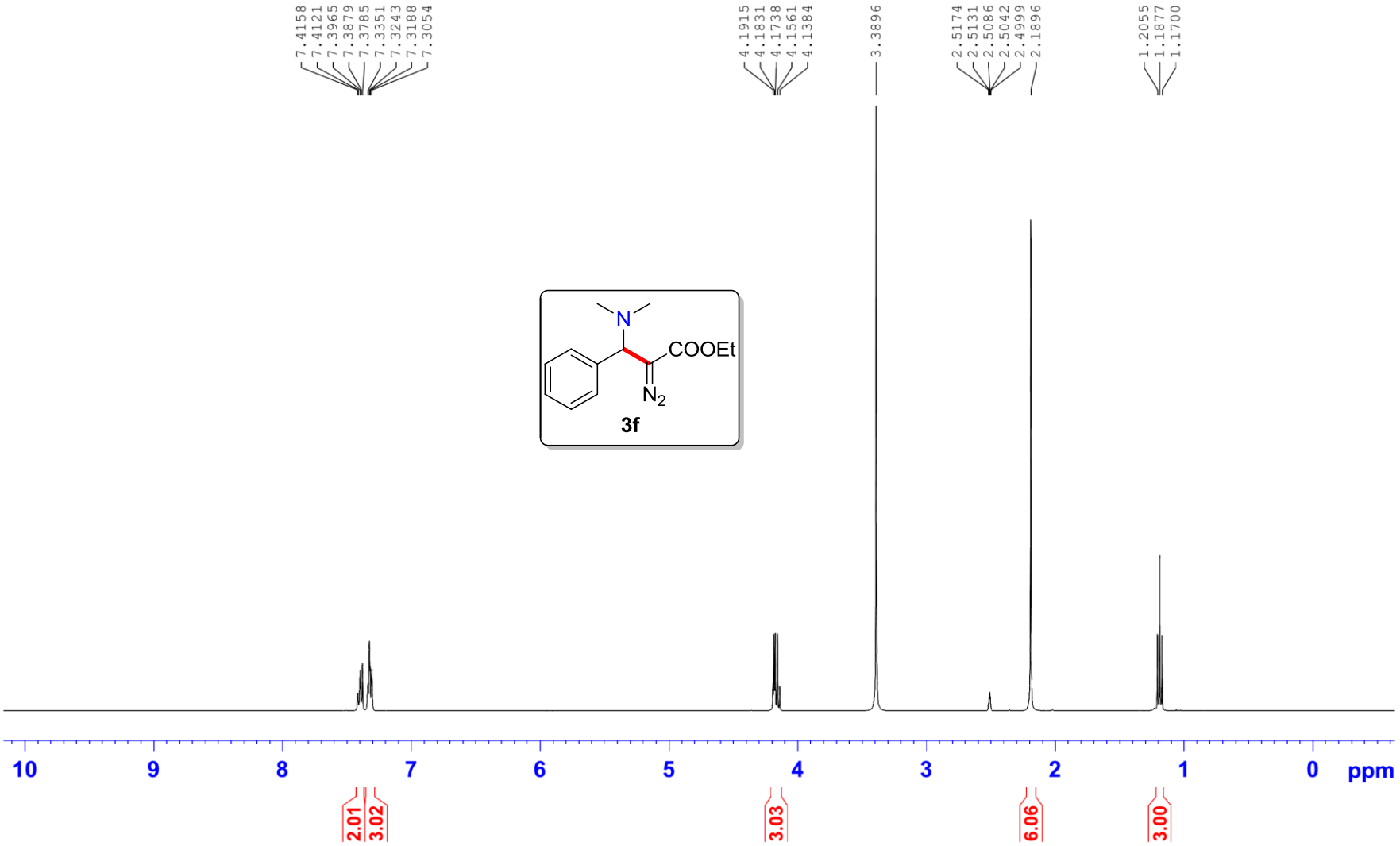

LJW-X15Z09-1-CNMR acetone-d6
206.1
166.0
142.1
129.6
128.7
127.6
64.2
61.4
53.3
30.4
30.3
30.1
29.9
29.7
29.5
29.3
24.3
14.8
N
COOEt
N2
3d
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

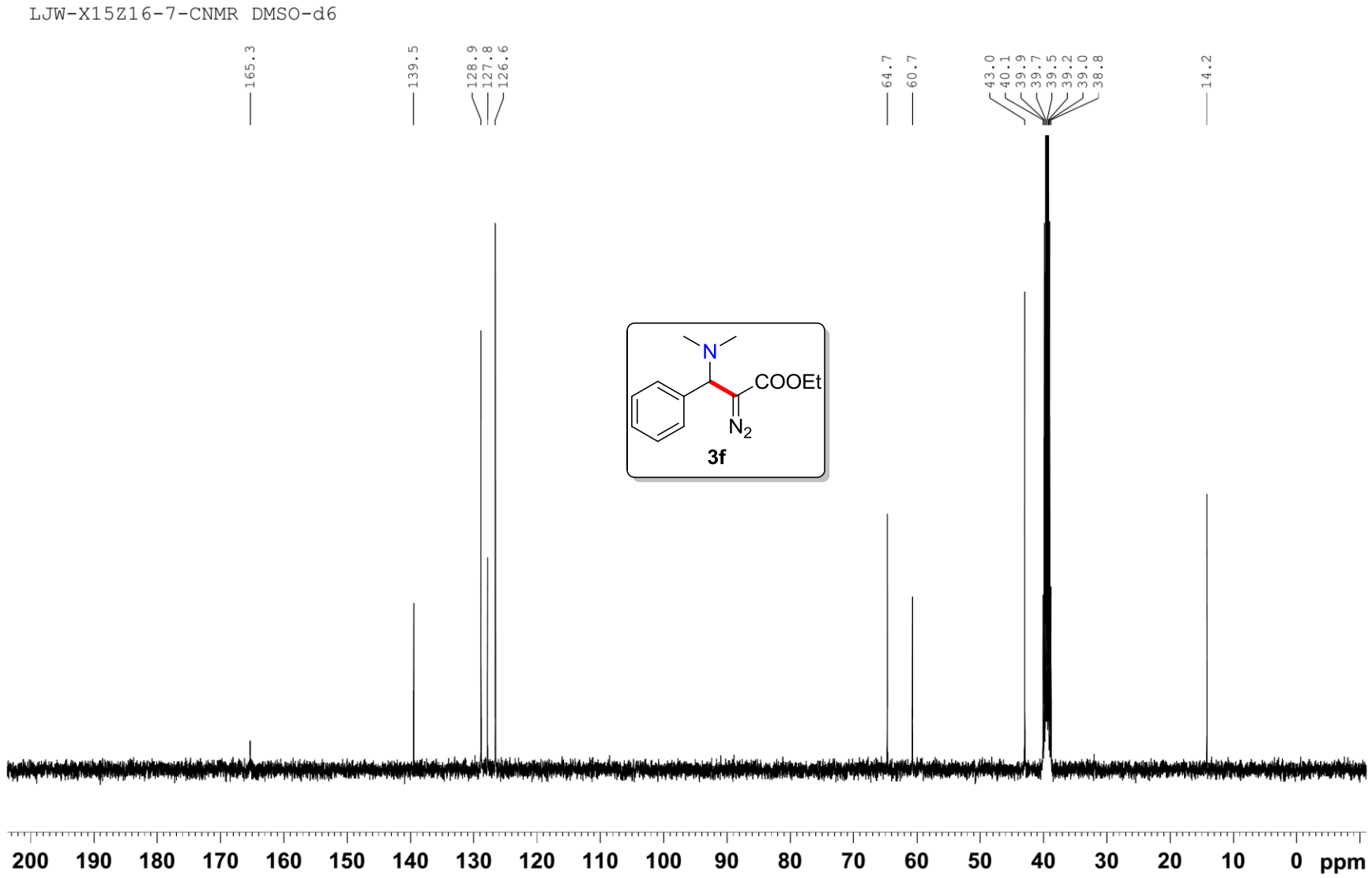
LJW-X15Y23-2-HNMR CDCl3



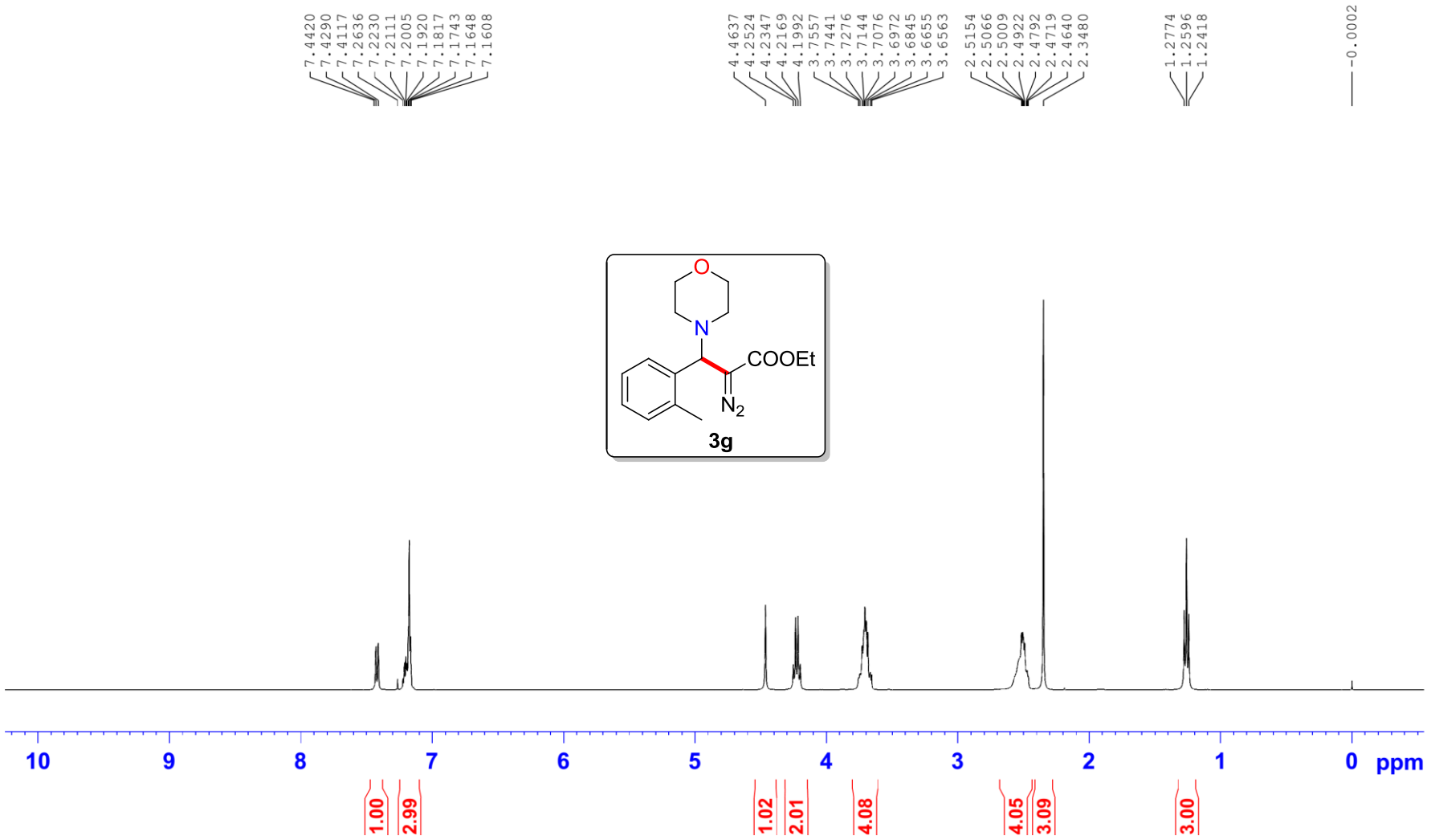

LJW-X15Z09-2-CNMR DMSO-d6
165.5
138.9
128.8
127.6
126.7
64.0
60.7
51.5
39.9
39.7
39.5
39.3
39.1
25.6
24.0
14.2
N
COOEt
N2
3e
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

LJW-X15Z17-3-HNMR DMSO-d6

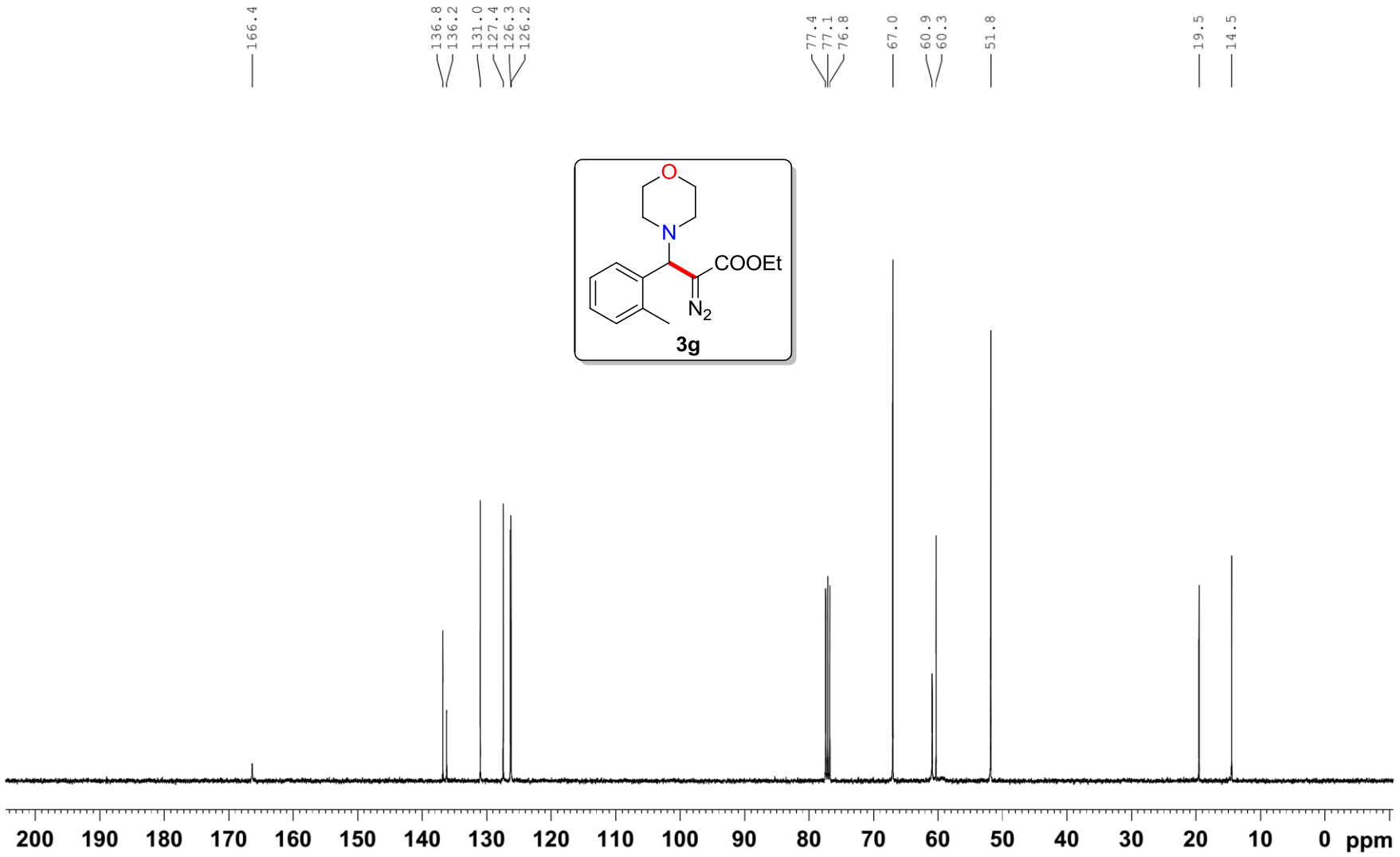



LJW-X15Z16-7-CNMR DMSO-d6
165.3
139.5
128.9
127.8
126.6
64.7
60.7
43.0
40.1
39.9
39.7
39.5
39.2
39.0
38.8
14.2
N
COOEt
N2
3f
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

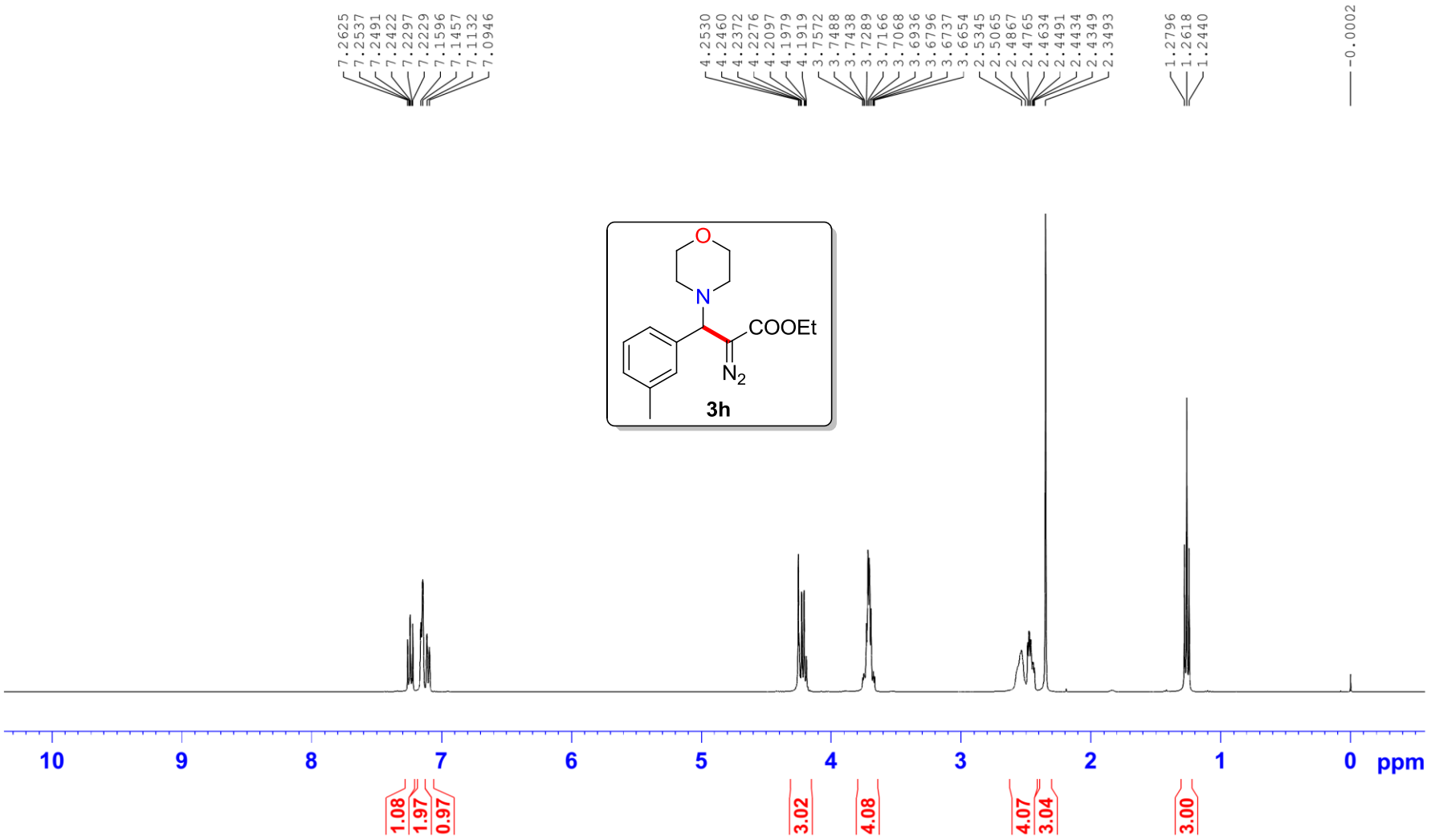
LJW-X15Z30-7-HNMR CDCl3



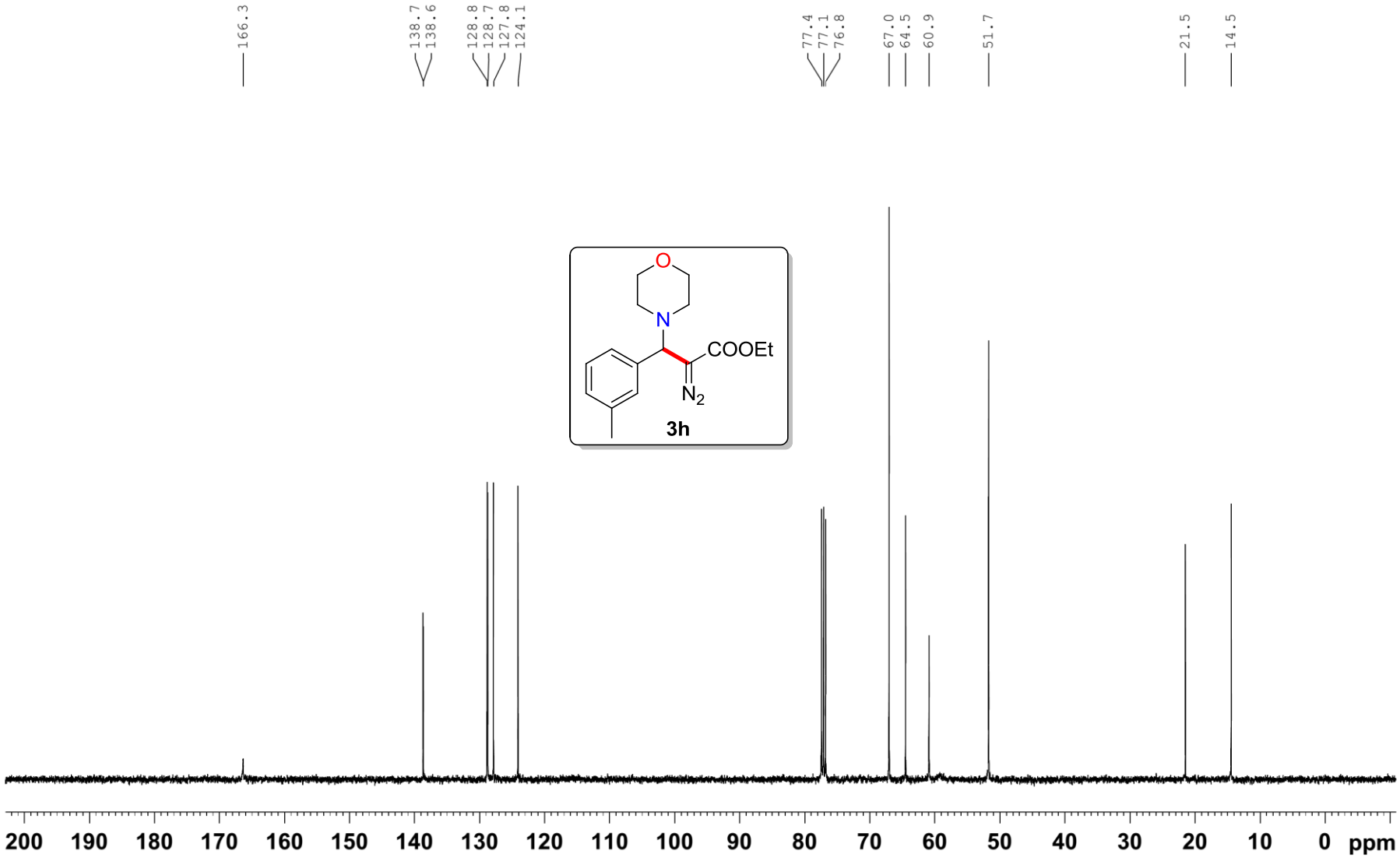
LJW-X160104-2-CNMR CDCl3



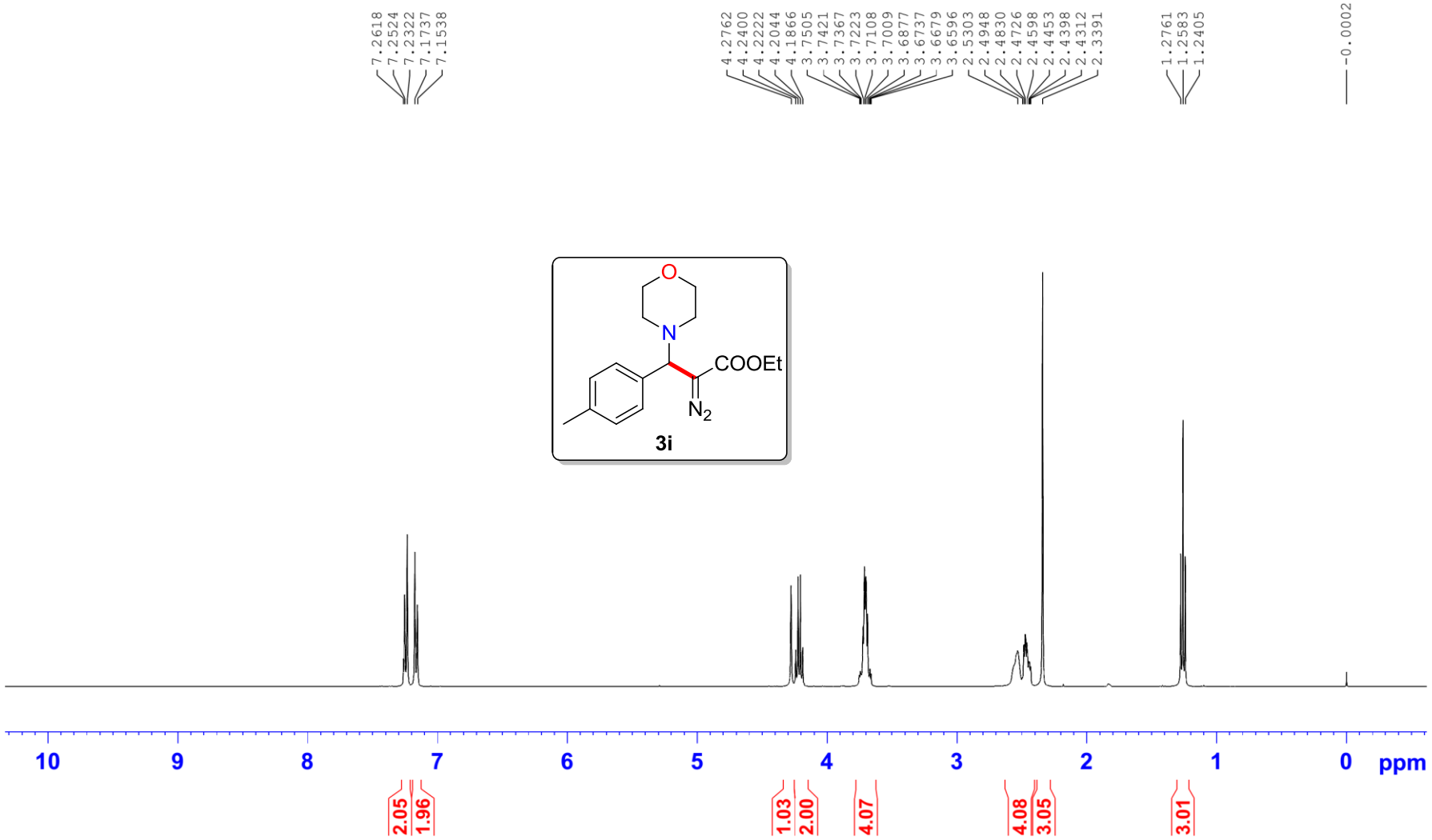
LJW-X15Z30-8-HNMR CDCl3



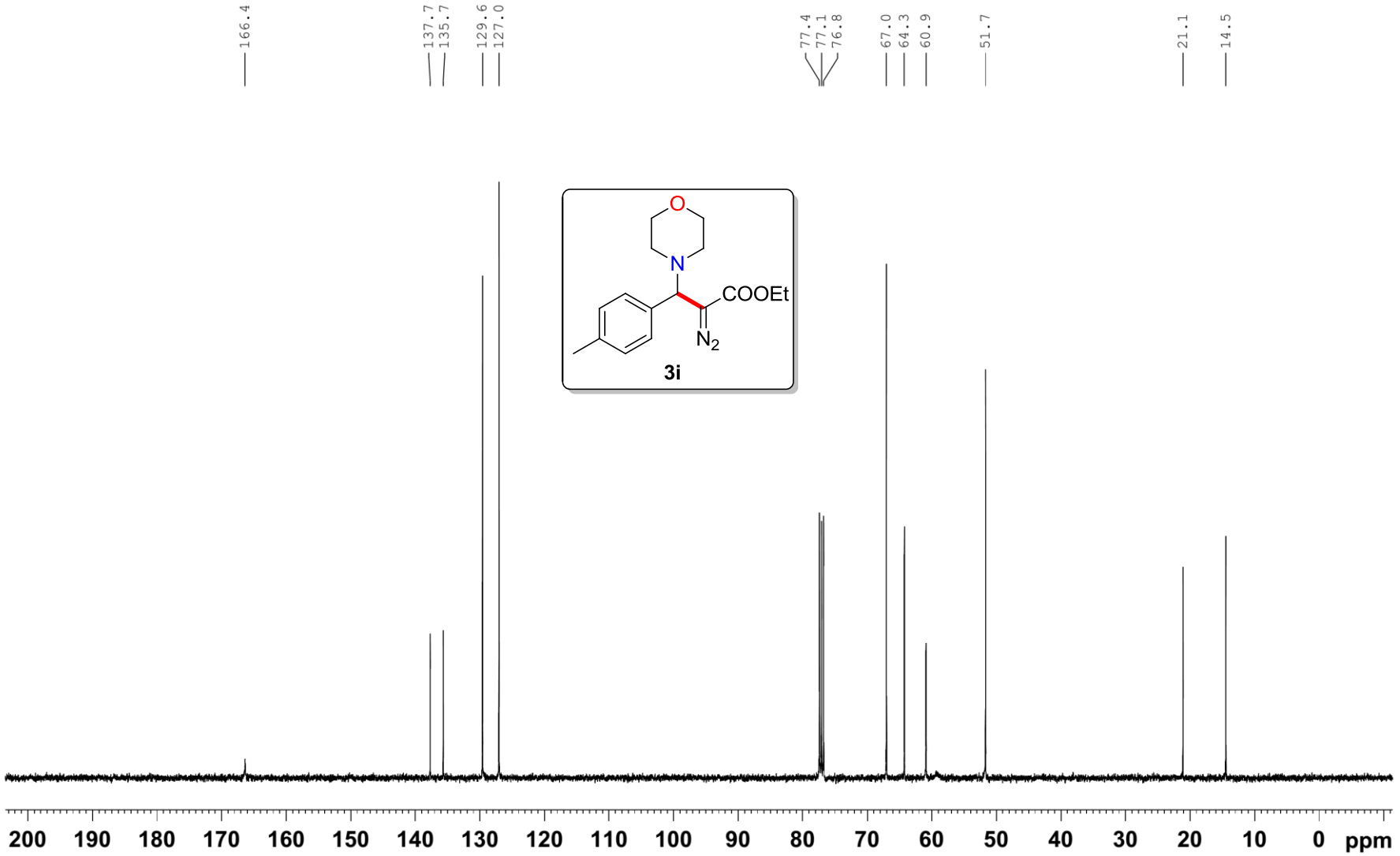
LJW-X160104-3-CNMR CDCl3



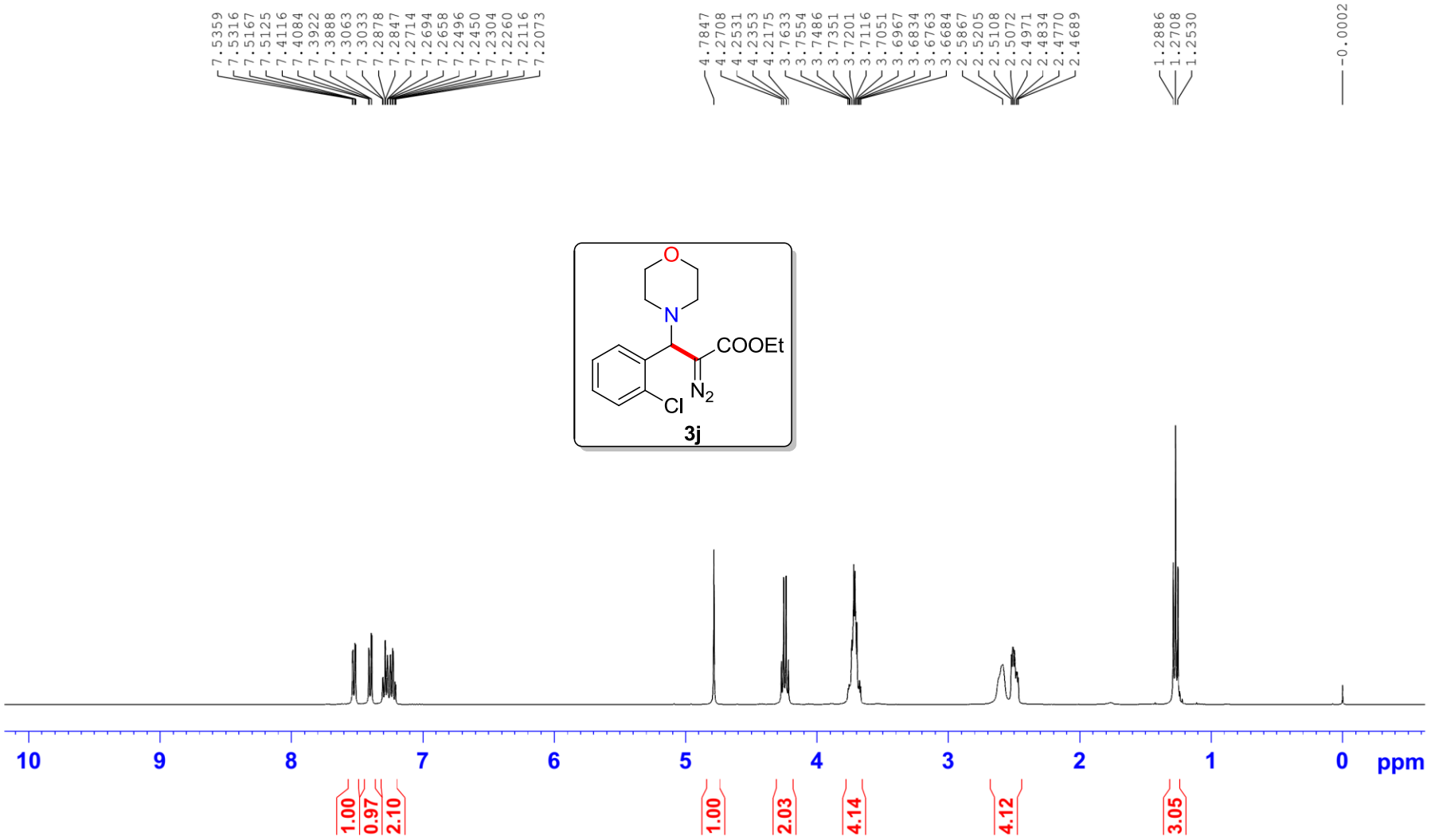
LJW-X15Z30-9-HNMR CDCl3



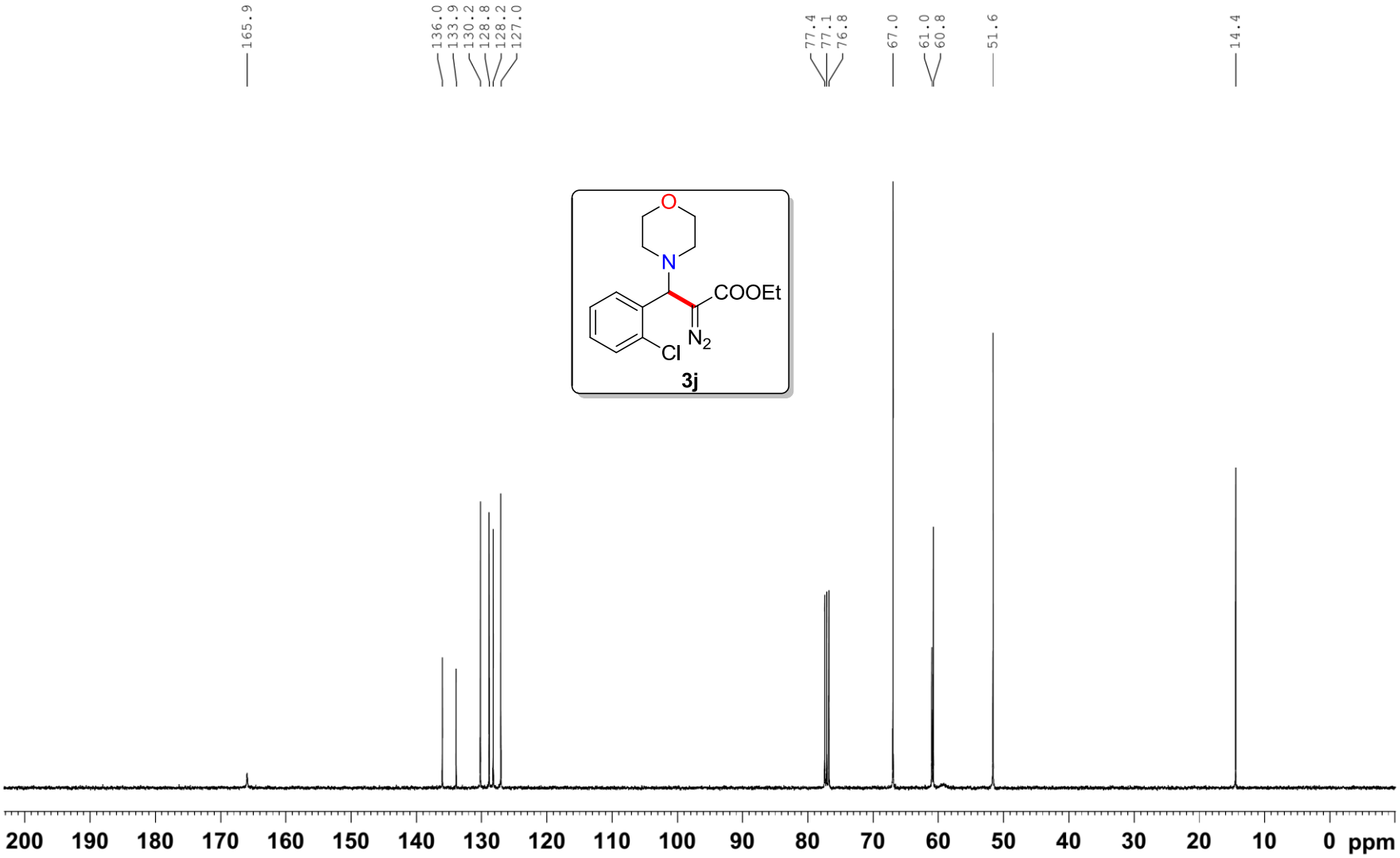
LJW-X160104-4-CNMR CDCl3



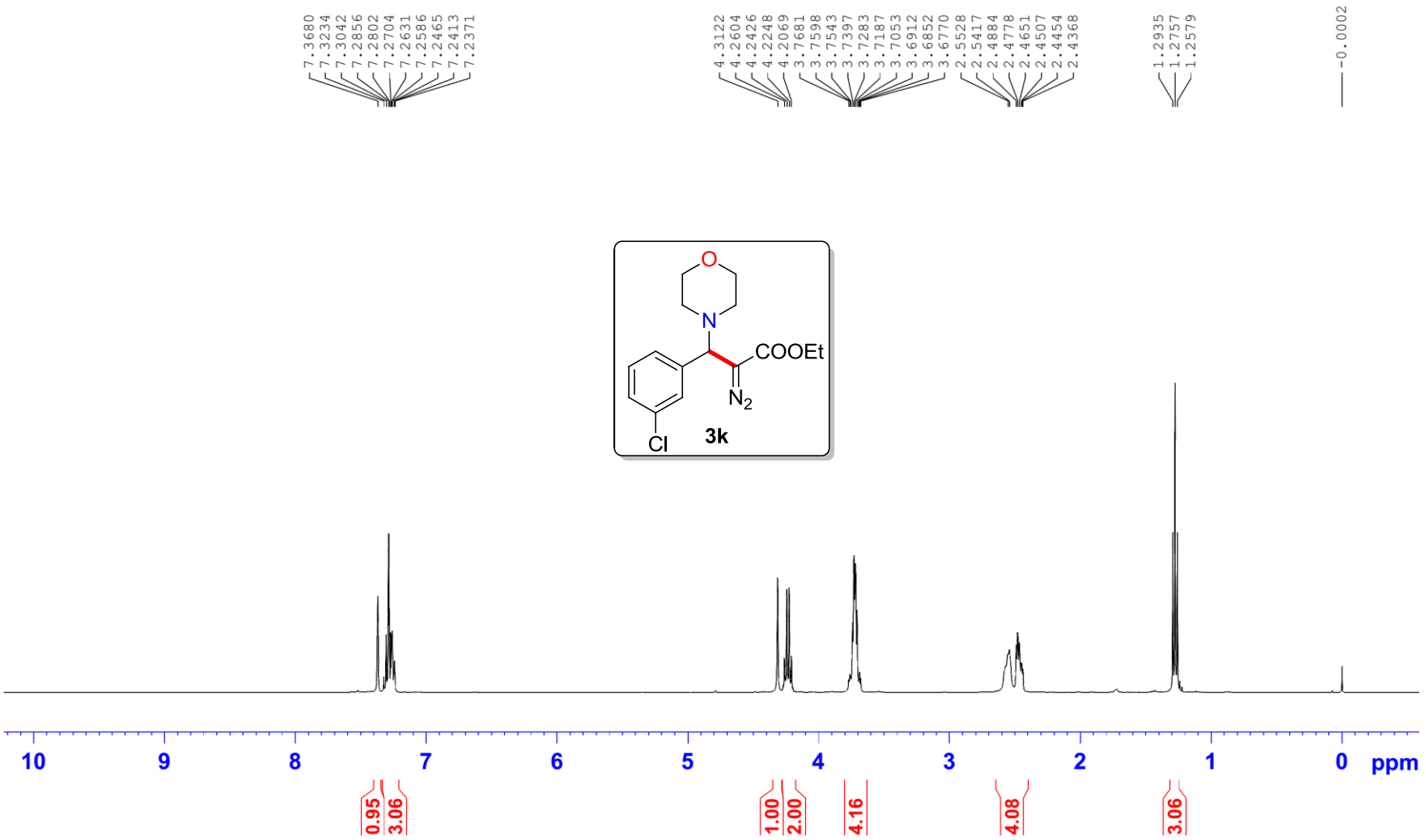
LJW-X15Z29-7-HNMR CDCl3



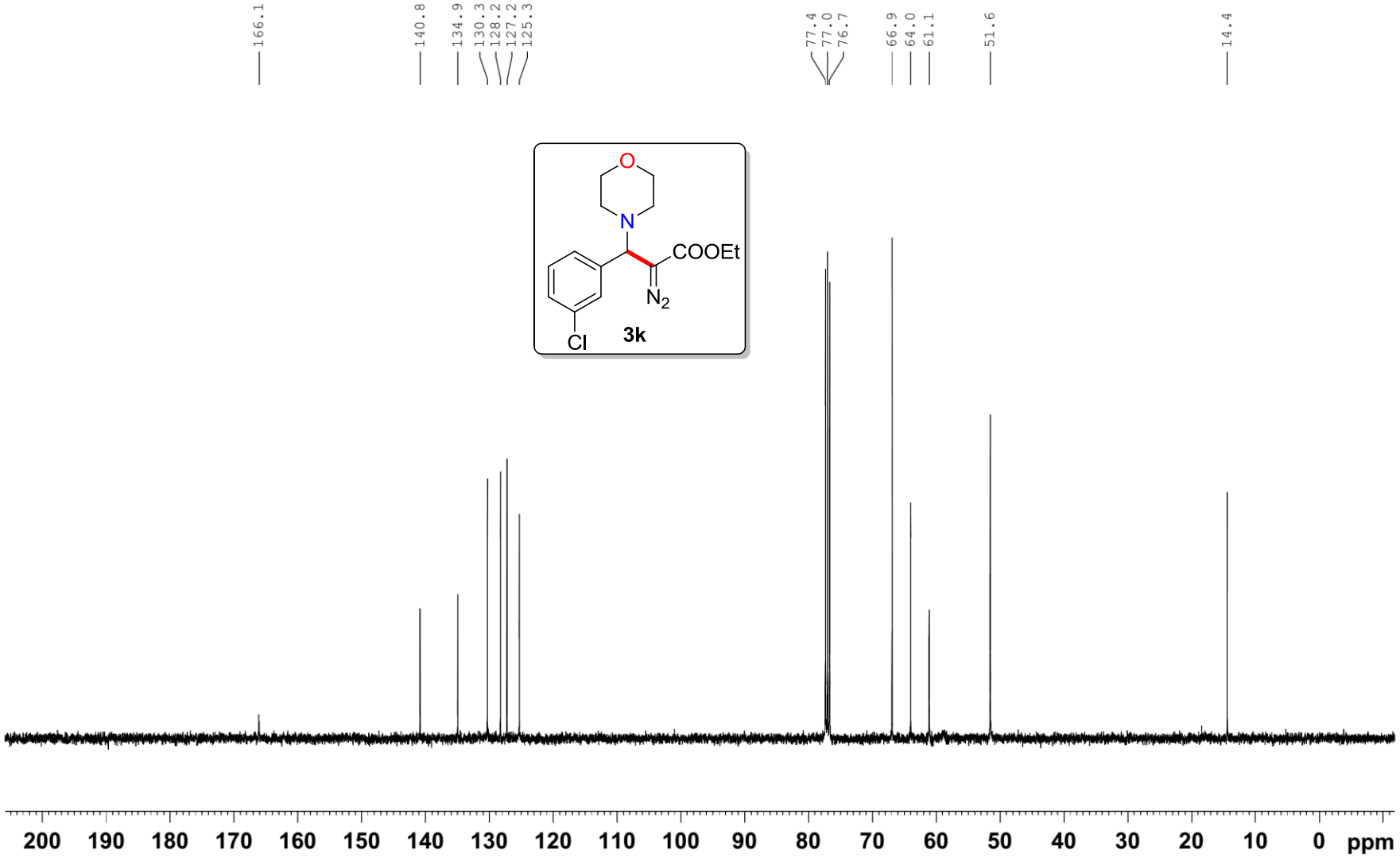
LJW-X160104-6-CNMR CDCl3



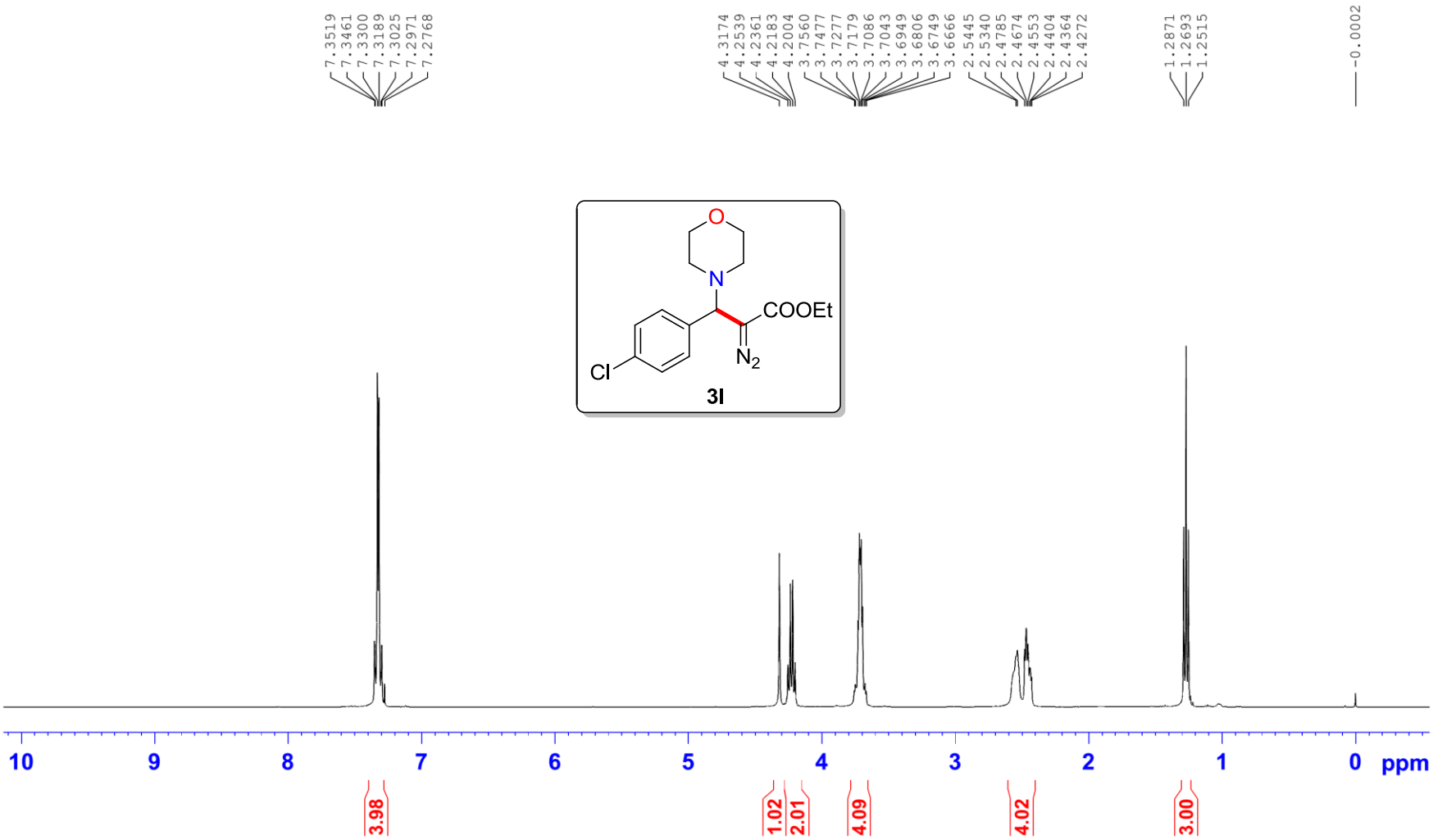
LJW-X15Z29-8-HNMR CDCl3

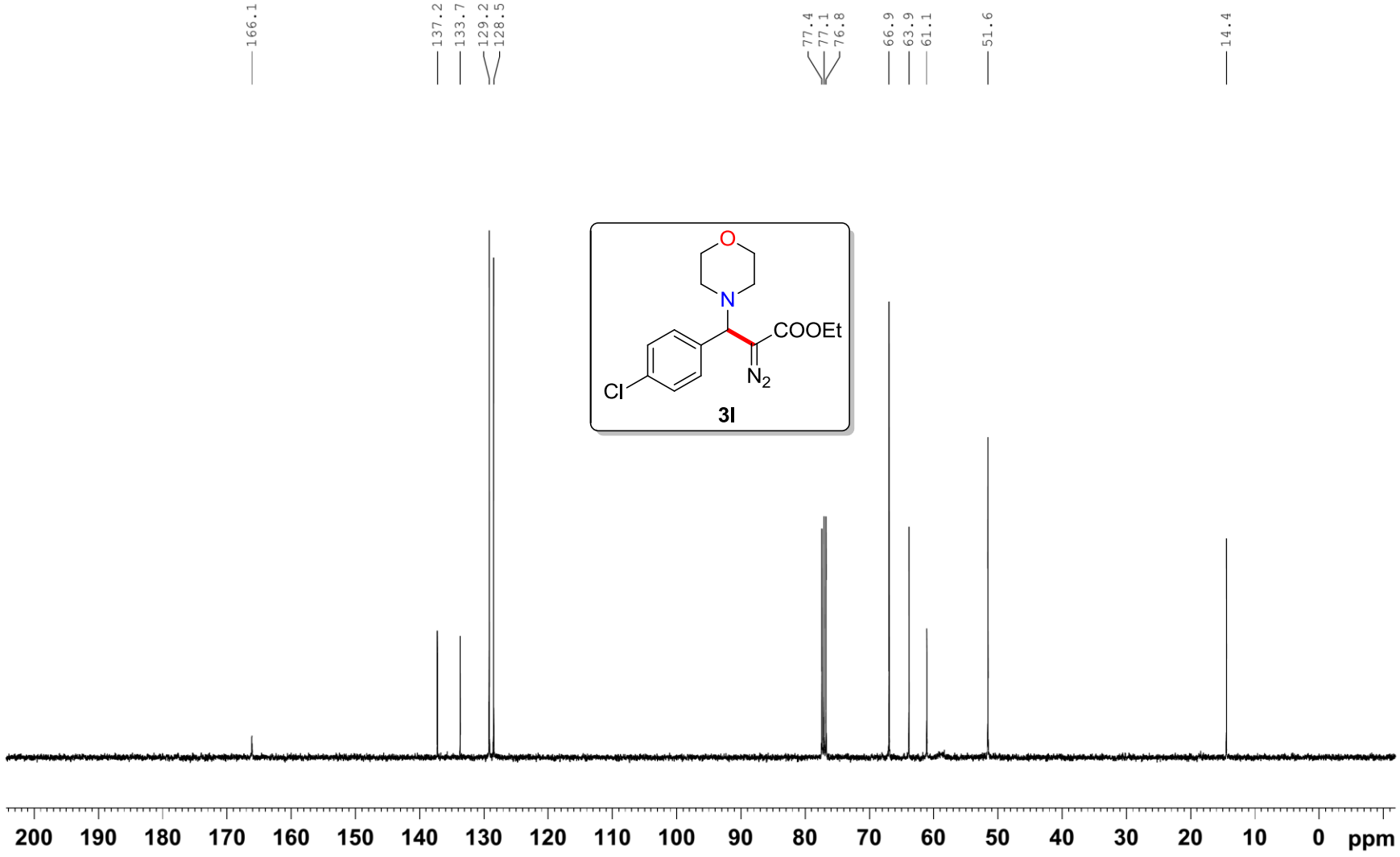


LJW-X15Z30-5-CNMR CDCl3

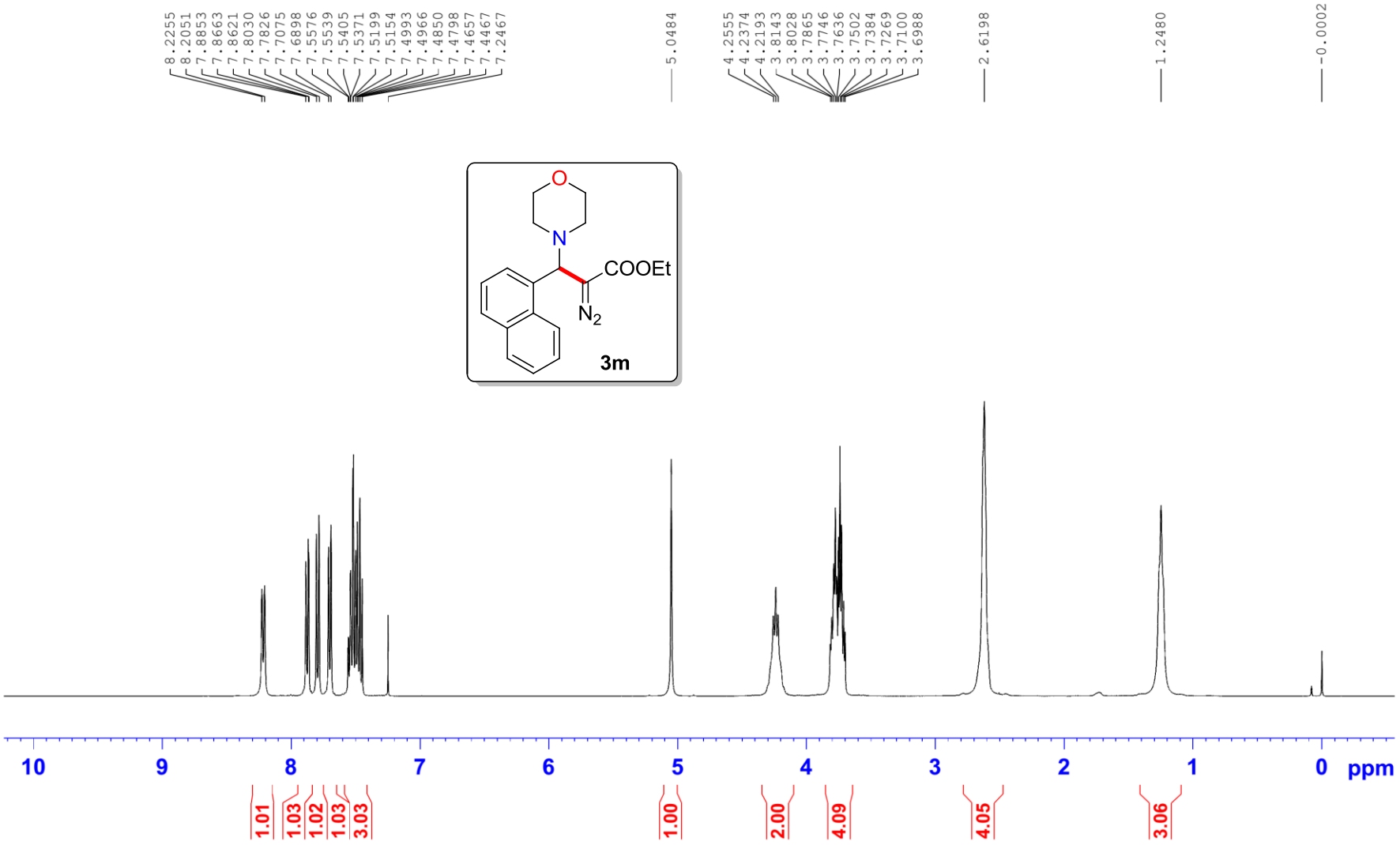


LJW-X15Z29-9-HNMR CDCl3

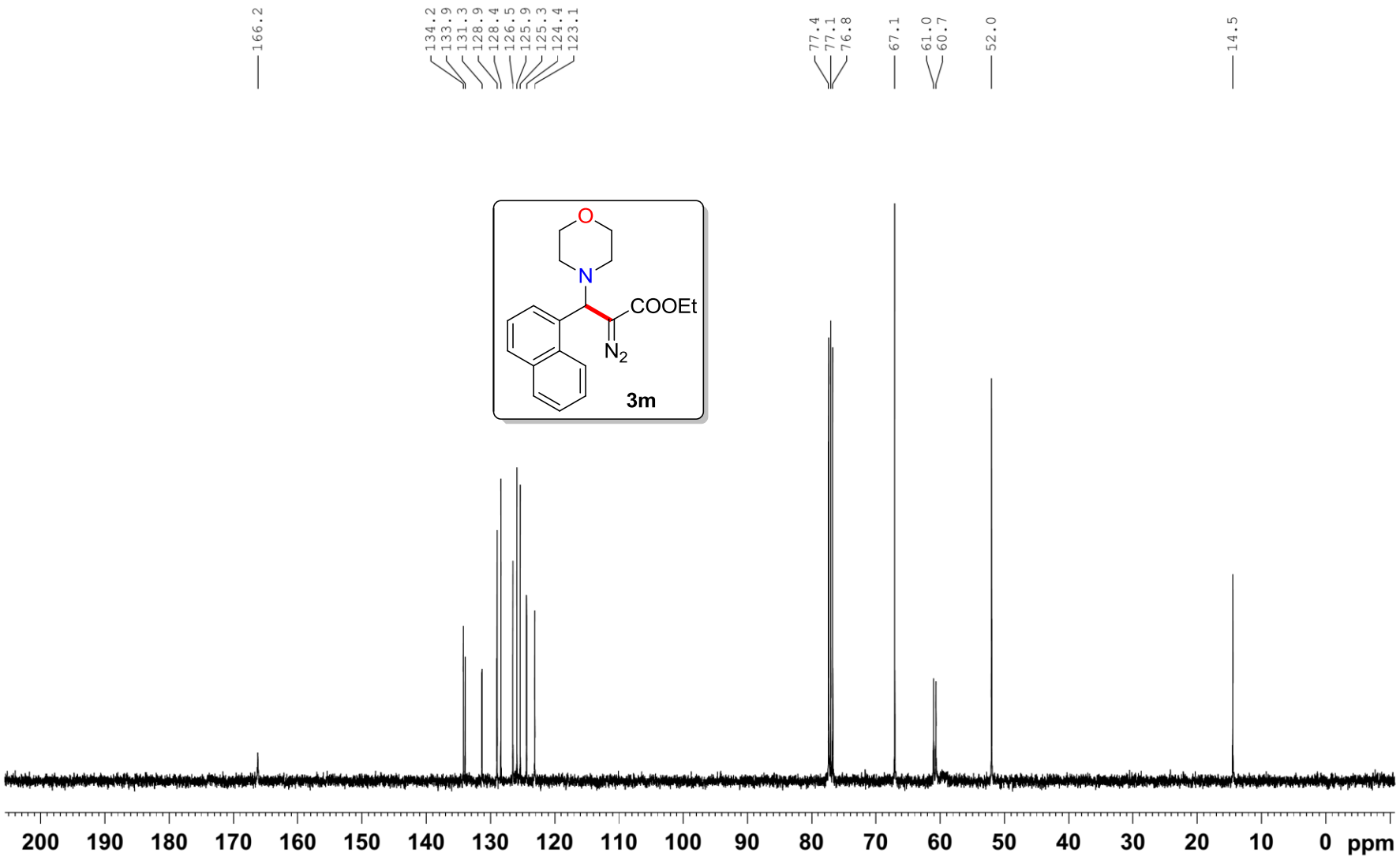



LJW-X15Z30-6-CNMR CDCl3
166.1
137.2
133.7
129.2
128.5
77.4
77.1
76.8
66.9
63.9
61.1
51.6
14.4
O
N
COOEt
N2
Cl
3l
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

LJW-X160106-4-HNMR CDCl3



LJW-X160106-4-CNMR CDCl3



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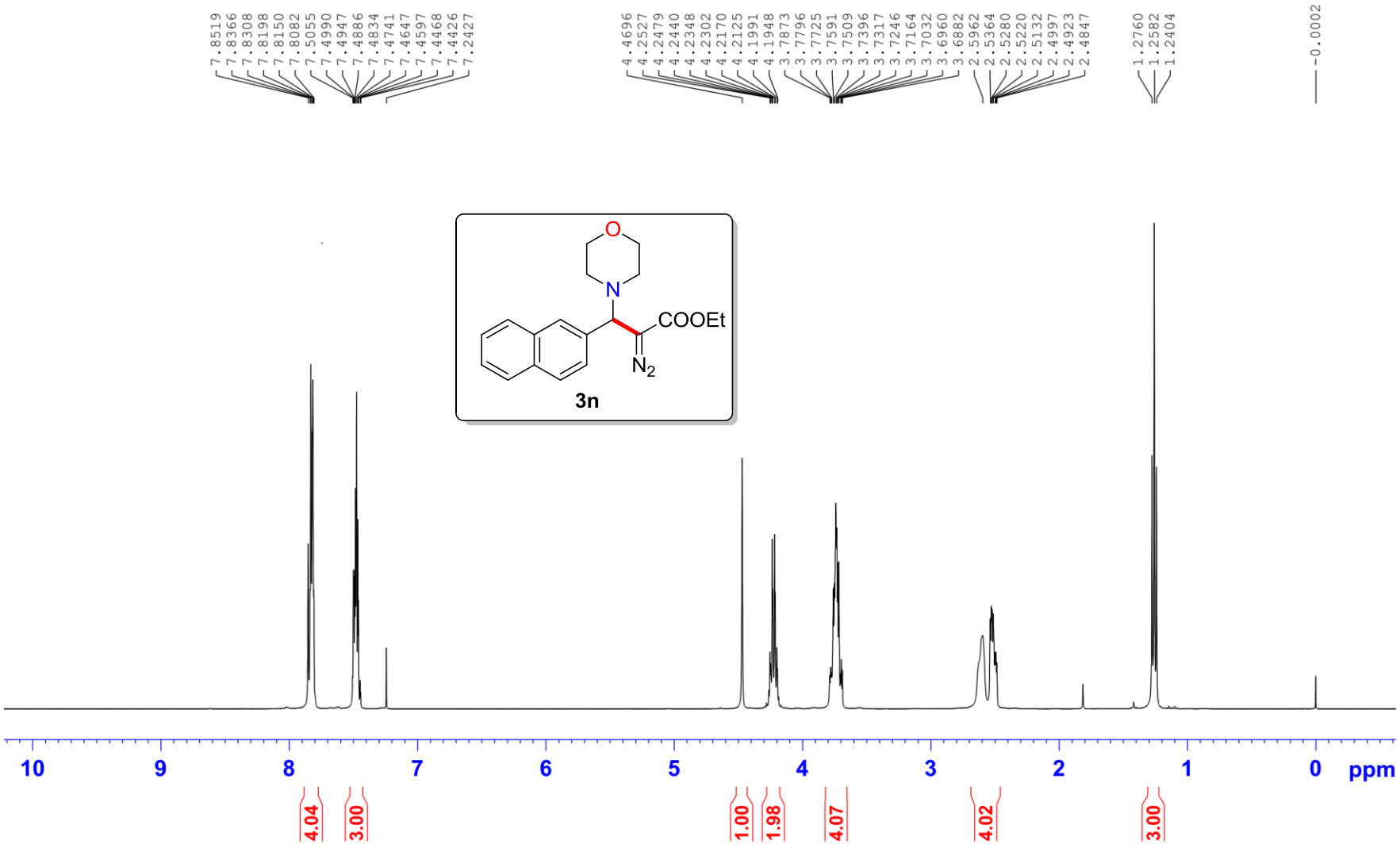
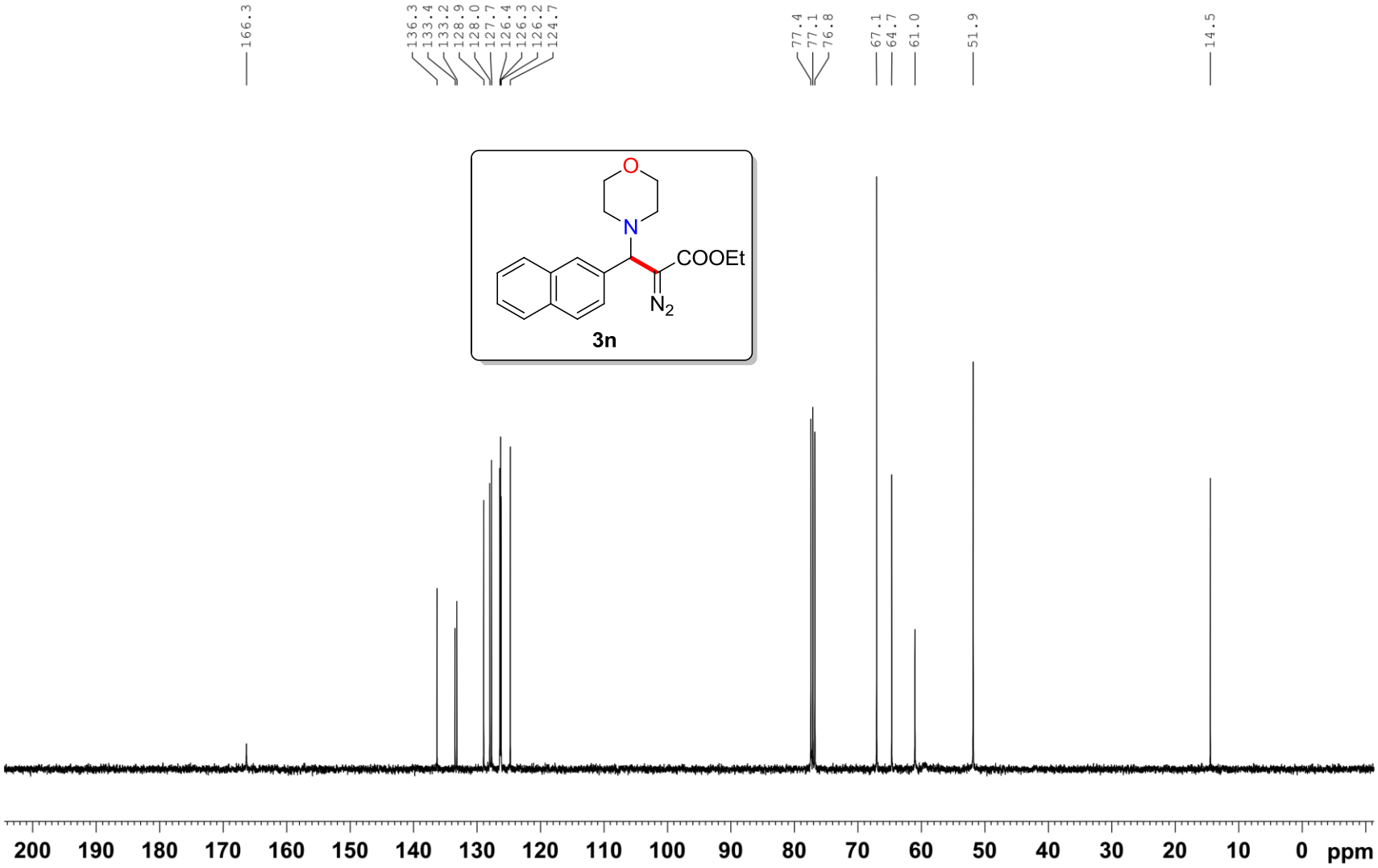
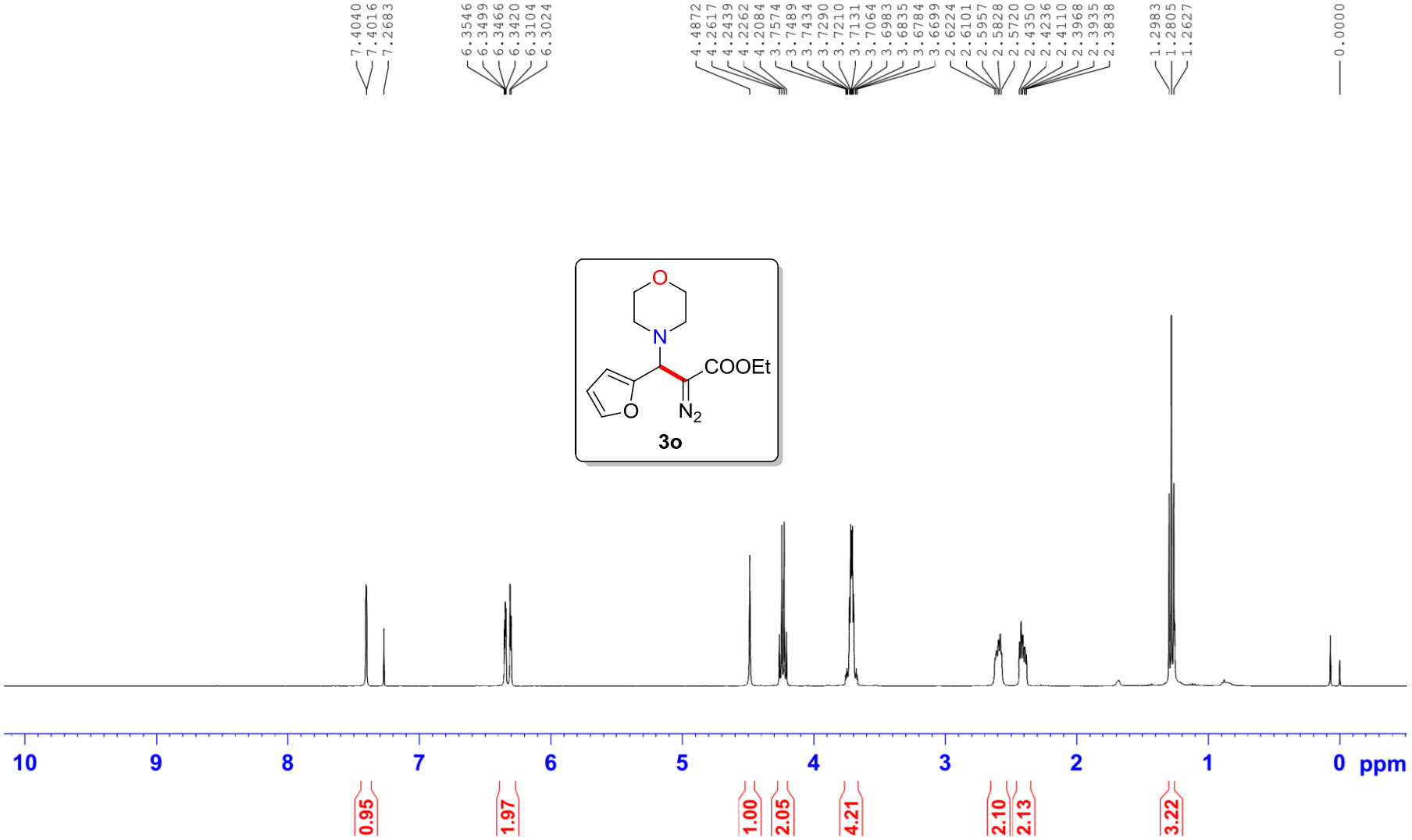
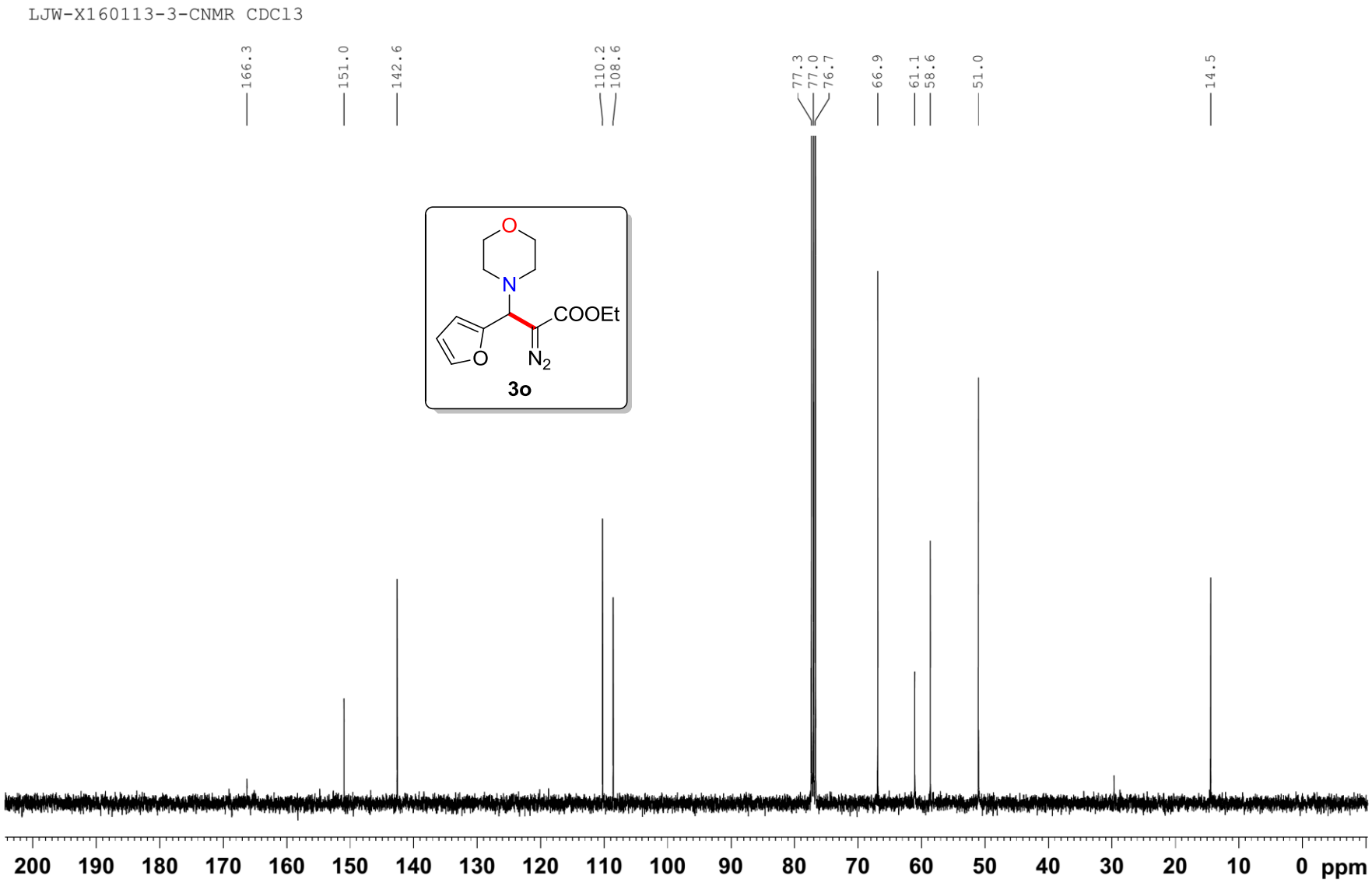


LJW-X160510-1-CNMR(3)

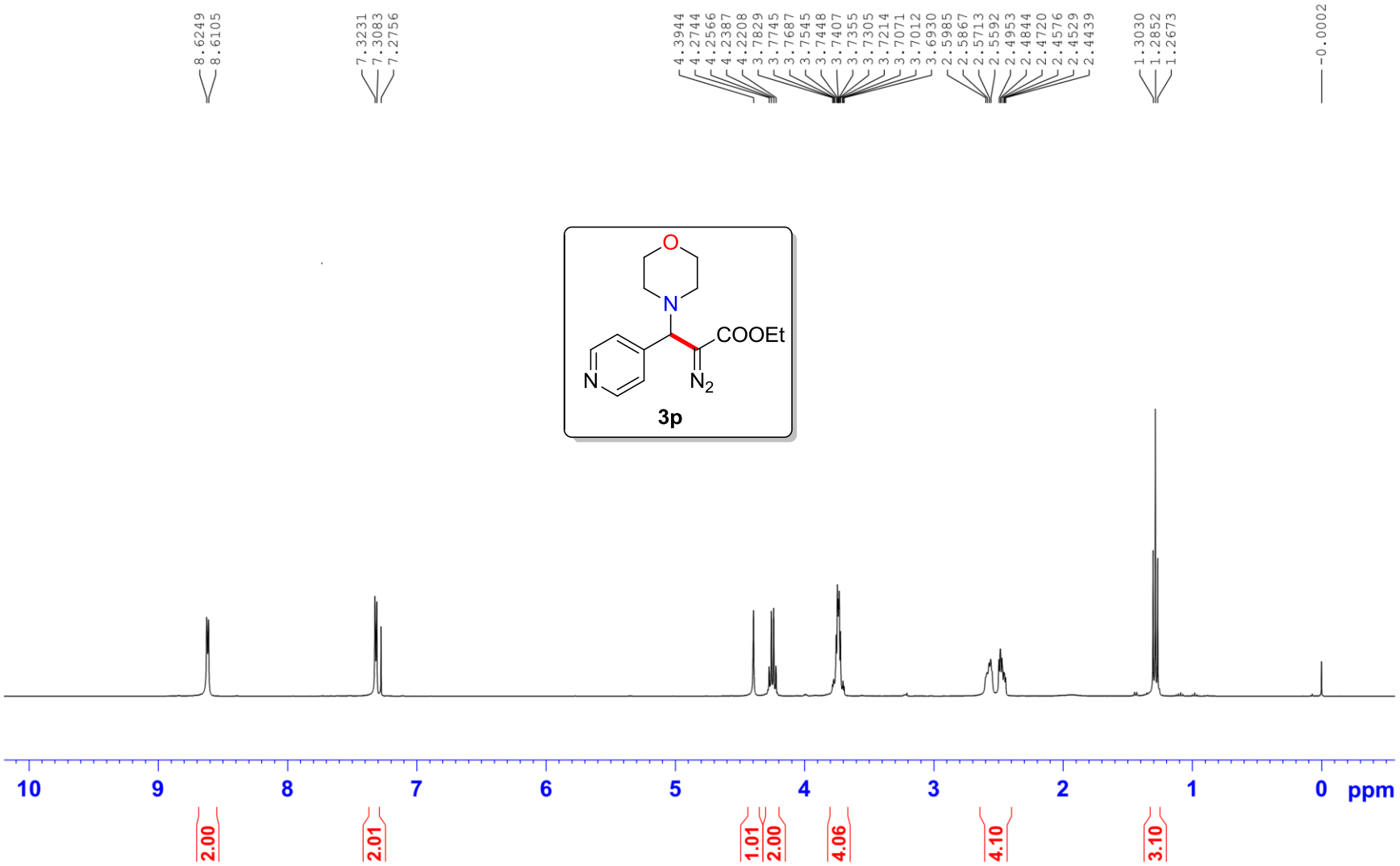


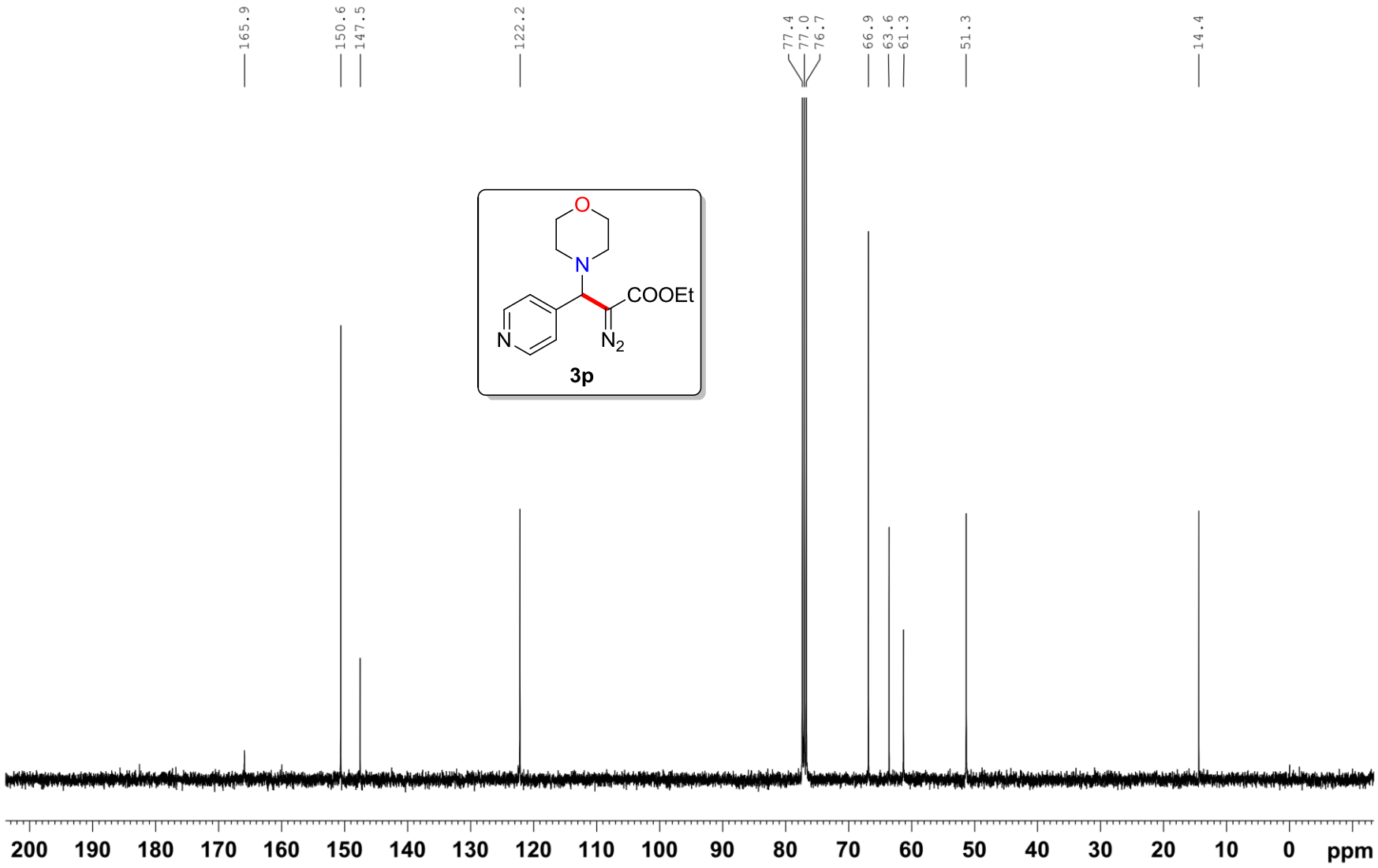
LJW-X160113-3-HNMR CDCl3



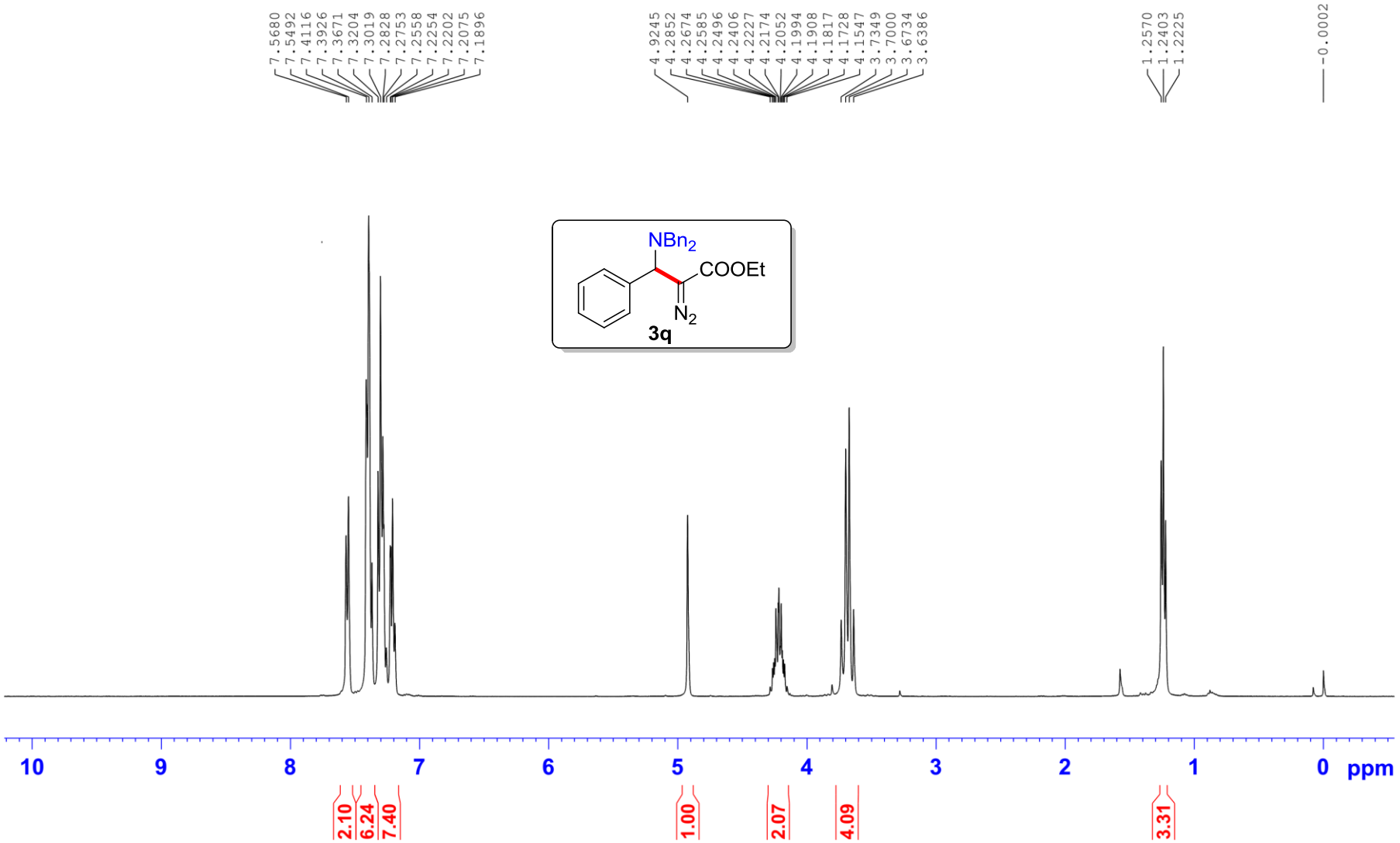

LJW-X160113-3-CNMR CDCl3
166.3
151.0
142.6
110.2
108.6
77.3
77.0
76.7
66.9
61.1
58.6
51.0
14.5
O
N
COOEt
O
N2
3o
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

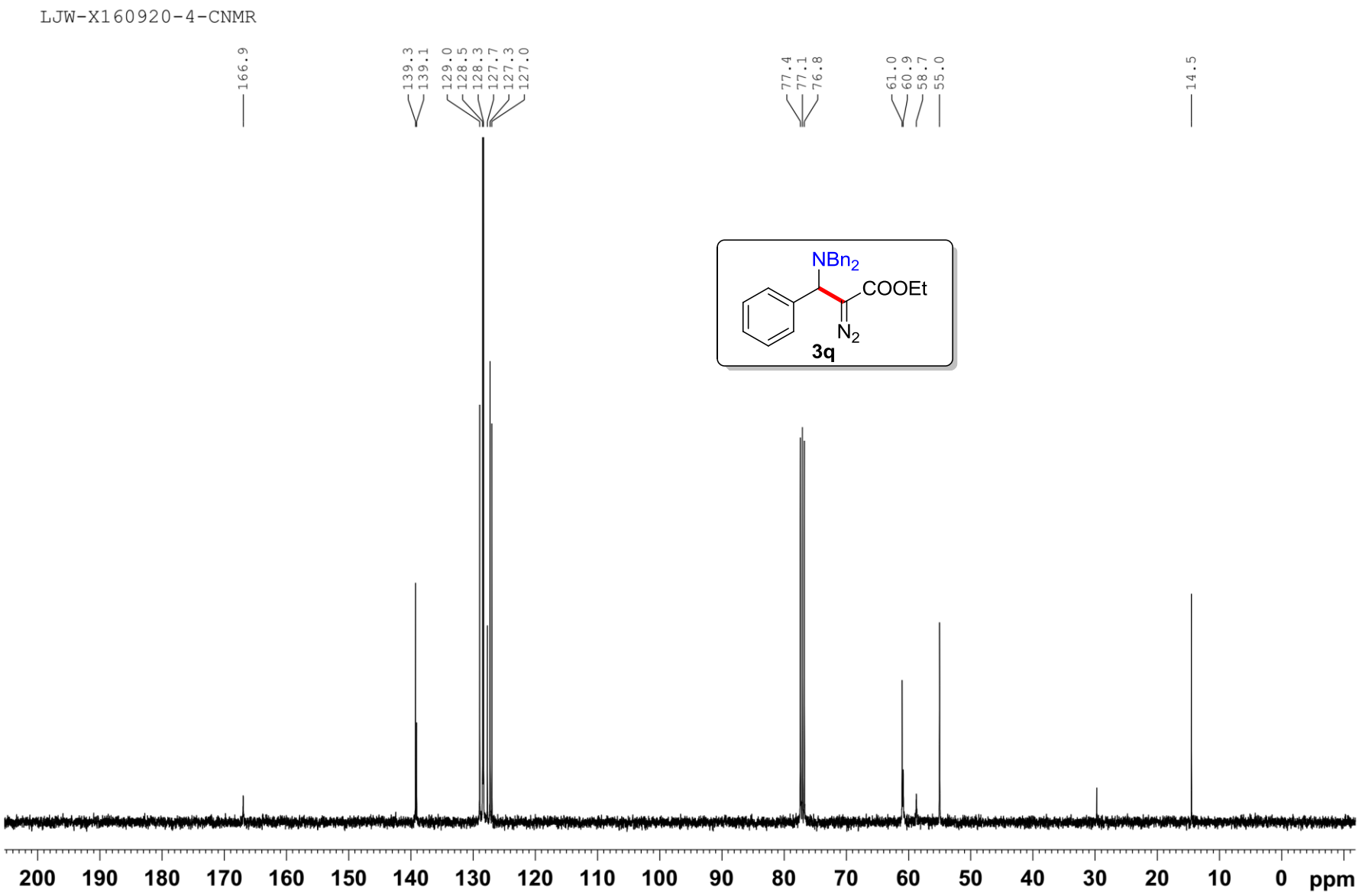
LJW-X160510-2-HNMR(1)



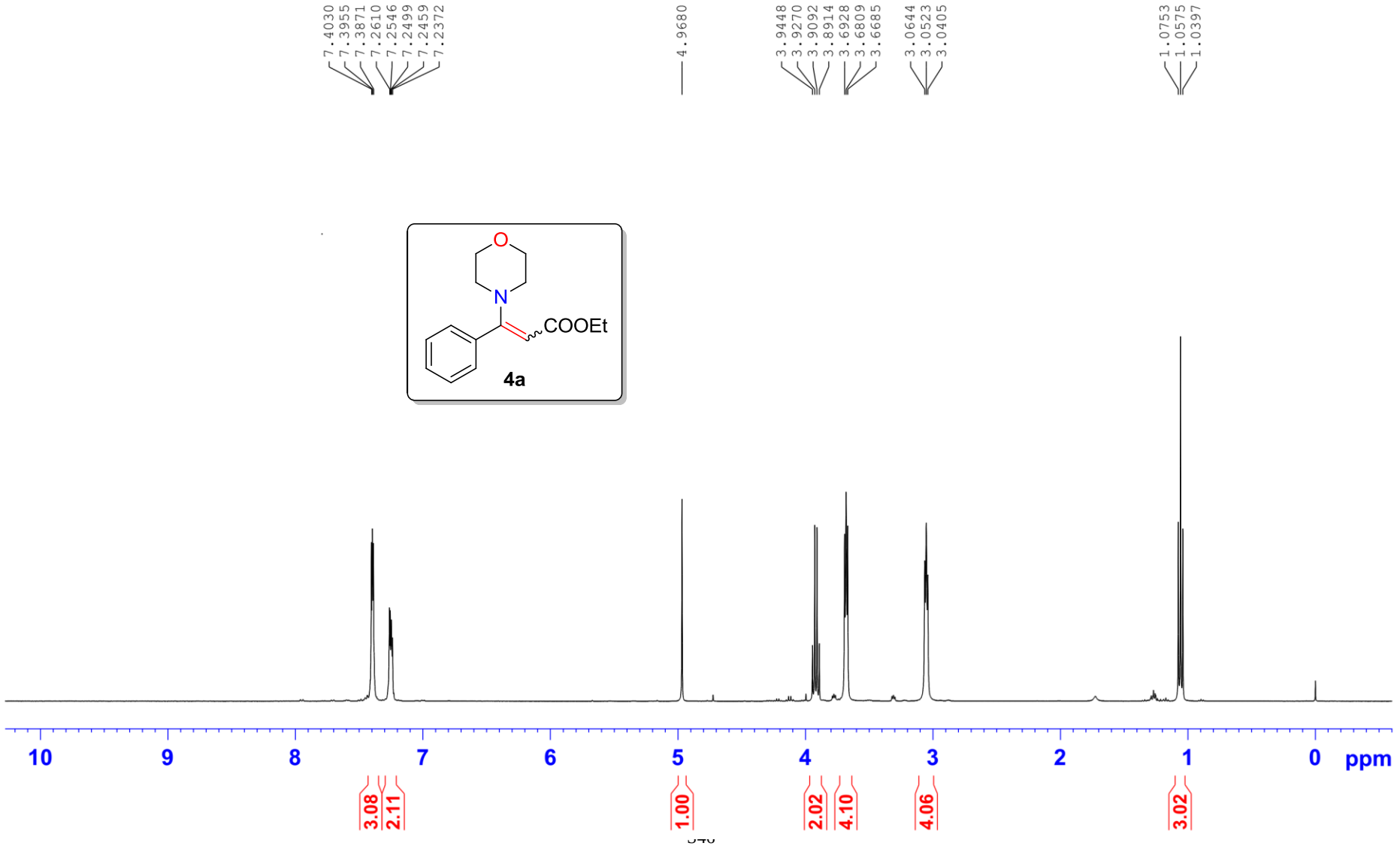

LJW-X160510-2-CNMR(13)
165.9
150.6
147.5
122.2
77.4
77.0
76.7
66.9
63.6
61.3
51.3
14.4
O
N
COOEt
N
N2
3p
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

LJW-X160920-4-HNMR




LJW-X160920-4-CNMR
166.9
139.3
139.1
129.0
128.5
128.3
127.7
127.3
127.0
77.4
77.1
76.8
61.0
60.9
58.7
55.0
14.5
NBn2
COOEt
N2
3q
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

LJW-X16X15-3-HNMR



LJW-X16X15-3-CNMR