SUPPORTING INFORMATION

Exploiting The Electrophilic and Nucleophilic Dual Role Of Nitrile imines: One-pot Three-component Synthesis Of Furo[2,3-*d*]pyridazin-4(5*H*)-ones

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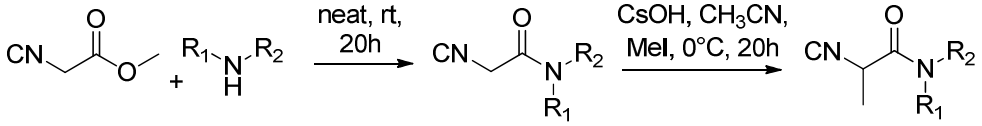
Contents:

General Methods. Commercially available reagents and solvents were used without further purification. Dichloromethane was dried by distillation from P_2O_5 and stored over activated molecular sieves (4 Å). When necessary the reactions were performed in oven-dried glassware under a positive pressure of dry nitrogen. Melting points were determined in open glass capillaries and are uncorrected. All the compounds were characterized by IR. ^{1}H and ^{13}C APT NMR were recorded on a 400 MHz. High-resolution ESI-MS spectra were performed on a Thermo LTQ Orbitrap XL mass spectrometer. The spectra were recorded by infusion into the ESI source using MeOH as the solvent. Chemical shifts (δ) are reported in part per million (ppm) relative to the residual solvent peak. Column chromatography was performed on silica gel (70‑230 mesh ASTM) using the reported eluents. Thin layer chromatography (TLC) was carried out on 5 x 20 cm plates with a layer thickness of 0.25 mm (Silica gel 60 F254). When necessary they were developed with $KMnO_4$.

General preparation of isocyanoacetamides (5, 20-23).[1] The α-methyl-α-isocyanoacetamides where readily synthesized in two steps starting from the commercially available methylisocyanoacetate:



1, 16-18

2-isocyano-1-morpholinopropan-1-one (5).

Compound **1** characterization data were compared to literature reported ones.[1]

2-isocyano-1-(pyrrolidin-1-yl)propan-1-one (20).

Compound **16** characterization data were compared to literature reported ones.[1]

2-isocyano-*N*-methyl-*N*-(prop-2-yn-1-yl)propanamide (21).

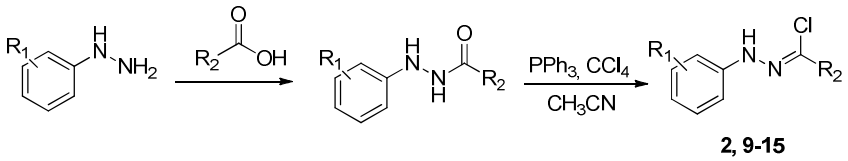
Compound **17** characterization data were compared to literature reported ones.[2]

***N*,*N*-diethyl-2-isocyanopropanamide (23).**

Compound **23** characterization data were compared to literature reported ones.[3]

2-isocyano-1-(4-methylpiperazin-1-yl)propan-1-one (22). The crude material was purified by column chromatography (dichloromethane/methanol 95:5) to give the product as a colorless oil (46% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.45 (br q, 1H), 3.62-3.34 (m, 4H), 2.35-2.29 (m, 4H), 2.20 (s, 3H), 1.46 (d, J = 6.68 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.9, 158.6, 54.5, 54.3, 49.5, 45.8, 45.6, 42.4, 18.7. IR (KBr) 2939, 2148, 1665, 1451, 1289, 1144, 998 ν_{max}/cm^{-1}. MS (ESI) *m/z* Calcd for $C_9H_{15}N_3O$: 181,1215; Found: 182.1290 $[M+H]^+$.

General preparation of hydrazonoyl chlorides (6, 13-18).[4] The hydrazonoyl chlorides where readily synthesized in two steps:



2, 9-15

Preparation of acylhydrazines. To a stirred solution of acid on a 5 mmol scale in THF (0.2 M) were added EDC HCl (5.5 mmol, 1.1 eq.), DMAP (1 mmol, 0.2 eq.), triethylamine (10 mmol, 2 eq.) and hydrazine (5 mmol, 1 eq.) at 0°C. The resulting mixture was allowed to warm to room temperature over 24 h. The crude reaction mixture was washed with HCl 1M sol. (x2), $NaHCO_3$ sat. sol. (x2) and brine (x1), evaporated to dryness and used in the next step without further purification.

Preparation of hydrazonoyl chlorides (6, 13-19). The corresponding acylhydrazine (2.5 mmol) was dissolved in CH_3CN (0.5 M) and triphenylphosphine (3 mmol, 1.2 eq.) and carbon tetrachloride (3 mmol, 1.2 eq.) were added. The reaction was stirred at room temperature until all the acylhydrazine was consumed as judged by TLC (typically 8-12 hours). The reaction was concentrated under reduced pressure and purified by column chromatography (*n*-hexane/EtOAc 30:1) and stored below 0°C. Due their delicate nature, neither HMRS nor elemental analyses of nitrile imines gave satisfactory results.

(*Z*)-4-methyl-*N*'-phenylbenzohydrazonoyl chloride (6).

Compound **2** characterization data were compared to literature reported ones.[4]

(*Z*)-*N*'-phenylbenzohydrazonoyl chloride (13).

Compound **9** characterization data were compared to literature reported ones.[5]

(*Z*)-4-iodo-*N*'-phenylbenzohydrazonoyl chloride (14).

Compound **10** characterization data were compared to literature reported ones. [4]

(*Z*)-4-phenoxy-*N*'-phenylbenzohydrazonoyl chloride (15). The crude material was purified by column chromatography (*n*-hexane/ EtOAc 30:1) to give the product as an amorphous yellowish solid (59% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.07 (br s, *NH*), 7.98 (d, *J* = 8.72 Hz, 2H), 7.48-7.39 (m, 4H), 7.27-7.23 (m, 3H), 7.18 (br d, AA'XX', 2H), 7.12 (br d, AA'XX', 2H), 7.04 (br t, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.6, 156.6, 143.5, 130.1 (2C), 129.5 (2C), 129.4 (2C), 128.1, 124.3, 124.0, 121.2, 119.5 (2C), 118.4 (2C), 113.5 (2C).

(*Z*)-2-(naphthalen-2-yl)-*N*'-phenylacetohydrazonoyl chloride (16). The crude material was purified by column chromatography (*n*-hexane/ EtOAc 30:1) to give the product as a pink oil (59% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.26 (d, *J* = 8.28, 1H), 7.91 (d, *J* = 7.88, 1H), 7.86-7.84 (m, 1H), 7.70 (br s, *NH*), 7.59-7.47 (m, 4H), 7.33-3.29 (m, 2H), 7.09-7.07 (m, 2H), 6.96-6.93 (m, 1H), 4.42 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 143.8, 134.0, 132.3, 131.7, 129.5 (2C), 128.9, 128.4, 128.2, 126.5, 126.0, 125.9, 125.6, 124.0, 120.9, 113.3 (2C), 42.6.

(Z)-*N*'-(4-chlorophenyl)benzohydrazonoyl chloride (17).

Compound **13** characterization data were compared to literature reported ones. [4]

(Z)-*N*-(3-methoxyphenyl)benzohydrazonoyl chloride (18).

The crude material was purified by column chromatography (PE/EtOAc 95:5) to give the product as a brown solid (55% yield). ^{1}H-NMR (300 MHz, $CDCl_3$) δ 8.07 (br s, *NH*), 7.75 (br d, 2H), 7.45-7.39 (m, 3H), 7.24 (br t, 1H), 6.85 (br s, 1H), 6.75 (dd, *J* = 7.95, 1.86 Hz, 1H), 6.54 (dd, *J* = 8.11, 2.46 Hz, 1H), 3.84 (s, 3H); ^{13}C-NMR (75 MHz, $CDCl_3$) δ 160.9, 144.8, 134.5, 130.3, 129.3, 128.5 (2C), 126.5 (2C), 124.8, 106.6, 106.2, 99.6, 55.3.

(Z)-4-chloro-*N*-(3-methoxyphenyl)benzohydrazonoyl chloride (19).

The crude material was purified by column chromatography (PE/EtOAc 95:5) to give the product as a brown solid (47% yield). ^{1}H-NMR (300 MHz, $CDCl_3$+CD_3OD) δ 7.79 (d, AA'XX, *J* = 8.25 Hz, 2H), 7.33 (d, AA'XX, *J* = 8.25 Hz, 2H), 7.19-7.15 (m, 1H), 6.75 (br s, 1H), 6.69 (dd, *J* = 7.95, 1.86 Hz, 1H), 6.47 (dd, *J* = 8.11, 2.46 Hz, 1H), 3.82 (s, 3H); ^{13}C-NMR (75 MHz, $CDCl_3$+CD_3OD) δ 160.8, 144.4, 135.1, 132.9, 130.1, 128.6, 127.5, 123.5, 106.6, 106.2, 99.6, 55.3.

Synthesis of 4-(4-methyl-2-((2-phenylhydrazono)(*p*-tolyl)methyl)oxazol-5-yl)morpholine (7). The hydrazonoyl chloride (0.8 mmol, 1 eq.), the isocyanoacetamide (0.8 mmol, 1 eq.) and TEA (0.8 mmol, 1 eq.) were one-pot mixed in DCM (0.8 M, 1 mL) and stirred at room temperature under a nitrogen atmosphere overnight. After evaporation of the solvent, the crude material was purified by column chromatograph (*n*-hexane/ EtOAc 8:2) to give the product as yellow solid (226 mg, 75% yield). Data are referred to the main isomer. ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.77 (br s, *NH*), 7.68 (br d, AA'XX', 2H), 7.31-7.22 (m, 7H), 3.81-3.79 (m, 4H), 3.12-3.10 (m, 4H), 2.40 (s, 3H), 2.26 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.3, 163.4, 143.8, 138.4, 133.4, 130.8, 129.6 (2C), 129.2 (2C), 128.4 (2C), 126.0, 121.4, 113.6 (2C), 66.7, 66.5, 45.9, 42.5, 21.3, 18.8. IR (KBr) 3219, 2945, 2846, 1947, 1909, 1665, 1596, 1495, 1251 ν_{max}/cm^{-1}; Mp 137-138°C; MS (ESI) *m/z* Calcd for $C_{22}H_{24}N_4O_2$: 376.1899; Found: 377.1963 $[M+H]^+$.

General one-pot preparation of furo[2,3-*d*]pyridazin-4(5*H*)-ones (8, 24-34). The hydrazonoyl chloride (0.8 mmol, 1 eq.), the isocyanoacetamide (0.8 mmol, 1 eq.) and TEA (0.8 mmol, 1 eq.) were one-pot mixed in DCM (0.8 M, 1 mL) and stirred at room temperature under a nitrogen atmosphere overnight. The formation of the intermediate oxazole was monitored by TLC, and after evaporation of the solvent, toluene (0.2M, 4 ml) and DMAD (dimethyl acetylenedicarboxylate) (1.6 mmol, 2 equiv.) were added to the crude mixture and the reaction was stirred at reflux temperature overnight. After evaporation of the solvent, the crude material was purified by column chromatography.

Methyl 2-morpholino-4-oxo-5-phenyl-7-(*p*-tolyl)-4,5-dihydrofuro[2,3-*d*]pyridazine-3-carboxylate (8). The crude material was purified by column chromatography (*n*-hexane/ EtOAc 7:3) to give the product as yellow solid (160 mg, 45% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85 (br d, AA'XX',

2H), 7.61 (br d, AA'XX', 2H), 7.50-7.46 (m, 2H), 7.40-7.36 (m, 1H), 7.29 (br d, 2H), 3.91 (s, 3H), 3.89-3.87 (m, 4H), 3.67-3.64 (m, 4H), 2.41 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.8, 161.5, 156.8, 143.6, 142.3, 139.9, 134.8, 129.5 (2C), 129.4, 128.7 (2C), 127.9, 127.4 (2C), 126.4 (2C), 123.5, 90.5, 66.3 (2C), 52.2, 48.2 (2C), 21.4. IR (KBr) 2956, 2846, 1695, 1585, 1448, 1294, 1072 ν_{max}/cm^{-1}; Mp 211-212°C; MS (ESI) *m/z* Calcd for $C_{25}H_{23}N_3O_5$: 445.1638; Found: 446.1693 $[M+H]^+$.

methyl 5-(4-chlorophenyl)-2-(methyl(prop-2-yn-1-yl)amino)-4-oxo-7-phenyl-4,5-dihydrofuro [2,3-*d*]pyridazine-3-carboxylate (24). The crude material was purified by column chromatography (*n*-hexane/ EtOAc 7:3) to give the product as yellow solid (118 mg, 33% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.01 (br d, AA'XX', 2H), 7.60 (br d, AA'XX', 2H), 7.50-7.43 (m, 5H), 4.36 (s, 2H), 3.93 (s, 3H), 3.27 (s, 3H), 2.37 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.6, 160.9, 156.6, 143.5, 140.8, 134.9, 133.6, 132.1, 129.7, 128.7 (4C), 127.6 (2C), 127.5 (2C), 123.8, 90.6, 77.5, 73.6, 52.2, 42.3, 38.1. IR (KBr) 3214, 2945, 1673, 1591, 1489, 1286, 1067 ν_{max}/cm^{-1}; Mp 86-87°C; MS (ESI) *m/z* Calcd for $C_{24}H_{18}ClN_3O_4$: 447.0986; Found: 448.1036 $[M+H]^+$.

methyl 7-(4-iodophenyl)-2-(4-methylpiperazin-1-yl)-4-oxo-5-phenyl-4,5-dihydrofuro[2,3-*d*]pyridazine-3-carboxylate (25). The crude material was purified by column chromatography (dichloromethane/methanol 95:5) to give the product as yellow solid (141 mg, 31% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.83 (br d, AA'XX', 2H), 7.71 (br d, AA'XX', 2H), 7.59 (br d, 2H), 7.50-7.46 (m, 2H), 7.41-7.37 (m, 1H), 3.90 (s, 3H), 3.72-3.69 (m, 4H), 2.66-2.62 (m, 4H), 2.40 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.8, 161.5, 156.7, 143.1, 142.2, 138.0, 133.7, 131.8, 129.1, 128.8, 128.1, 126.4, 123.8, 96.0, 90.2, 54.3 (2C), 52.3, 47.9 (2C), 46.0. IR (KBr) 2939, 2802, 1681, 1575, 1451, 1275, 1072 ν_{max}/cm^{-1}; Mp 236-237°C; MS (ESI) *m/z* Calcd for $C_{25}H_{23}IN_4O_4$: 570.0764; Found: 571.0777 $[M+H]^+$.

methyl 2-(4-methylpiperazin-1-yl)-4-oxo-7-(4-phenoxyphenyl)-5-phenyl-4,5-dihydrofuro[2,3-*d*] pyridazine-3-carboxylate (26). The crude material was purified by column chromatography (dichloromethane/methanol 95:5) to give the product as yellow solid (167 mg, 39% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.94 (br d, AA'XX', 2H), 7.60 (br d, AA'XX', 2H), 7.49-7.45 (m, 2H), 7.39-7.35 (m, 3H), 7.16 (br t, 1H), 7.09-7.05 (m, 4H), 3.90 (s, 3H), 3.75-3.71 (m, 4H), 2.71-2.66 (m, 4H), 2.43 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.8, 161.3, 158.9, 156.7, 156.4, 143.4, 142.4, 134.1, 129.9 (2C), 129.0 (2C), 128.6 (2C), 127.8, 127.0, 126.3 (2C), 124.0, 123.6, 119.6 (2C), 118.5 (2C), 94.1, 54.2 (2C), 52.1, 47.8 (2C), 45.7. IR (KBr) 2945, 2796, 1681, 1585, 1486, 1237, 1155, 1067

ν_{max}/cm^{-1}; Mp 193-194°C; MS (ESI) *m/z* Calcd for $C_{31}H_{28}N_4O_5$: 536.2060; Found: 537.2079 $[M+H]^+$.

methyl 2-(4-methylpiperazin-1-yl)-7-(naphthalen-2-ylmethyl)-4-oxo-5-phenyl-4,5-dihydrofuro [2,3-*d*]pyridazine-3-carboxylate (27). The crude material was purified by column chromatography (dichloromethane/methanol 98:2) to give the product as light yellow solid (89.5 mg, 22% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.31 (d, *J* = 8.04 Hz, 1H), 7.87 (d, *J* = 8.12 Hz, 1H), 7.78 (d, *J* = 8.12 Hz, 1H), 7.58-7.47 (m, 7H), 7.43-7.37 (m, 2H), 4.61 (s, 2H), 3.83 (s, 3H), 3.40-3.38 (m, 4H), 2.41-2.39 (m, 4H), 2.30 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.8, 161.6, 156.9, 144.3, 142.3, 136.0, 134.0, 132.6, 132.1, 128.8 (3C), 128.0, 127.9, 127.6, 126.5 (2C), 126.3, 125.8, 125.2, 124.0, 123.3, 89.5, 54.3 (2C), 52.0, 48.0 (2C), 46.0, 35.1. IR (KBr) 2939, 2796, 1670, 1583, 1448, 1253, 1144, 1061 ν_{max}/cm^{-1}; Mp 202-203°C; MS (ESI) *m/z* Calcd for $C_{30}H_{28}N_4O_4$: 508.2111; Found: 509.2176 $[M+H]^+$.

methyl 2-(methyl(prop-2-yn-1-yl)amino)-4-oxo-5,7-diphenyl-4,5-dihydrofuro[2,3-*d*]pyridazine-3-carboxylate (28). The crude material was purified by column chromatography (*n*-hexane/ EtOAc 7:3) to give the product as yellowish solid (106 mg, 32% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.03 (br d, AA' XX', 2H), 7.62 (br d, AA' XX', 2H), 7.52-7.46 (m, 5H), 7.41-7.37 (m, 1H), 4.37 (br d, 2H), 3.93 (s, 3H), 3.27 (s, 3H), 2.36 (br t, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.7, 161.0, 156.8, 143.6, 142.3, 134.7, 132.2, 129.7, 128.8 (2C), 128.7 (2C), 127.9, 127.5 (2C), 126.4 (2C), 123.7, 90.5, 73.6, 52.4, 42.3 (2C), 38.2. IR (KBr) 3252, 1703, 1668, 1610, 1215, 1070 ν_{max}/cm^{-1}; Mp 146-147°C; MS (ESI) *m/z* Calcd for $C_{24}H_{19}N_3O_4$: 413.1376; Found: 414.1458 $[M+H]^+$.

Methyl 7-(4-chlorophenyl)-5-(3-methoxyphenyl)-4-oxo-2-(pyrrolidin-1-yl)-4,5-dihydrofuro[2,3-*d*]pyridazine-3-carboxylate (29). The crude material was purified by column chromatography (PE/EtOAc 7:3) to give the product as yellowish solid (169 mg, 44% yield). ^{1}H-NMR (300 MHz, $CDCl_3$) δ 7.95 (d, AA'XX', *J* = 8.25 Hz, 2H), 7.43 (d, AA'XX', *J* = 8.25 Hz, 2H), 7.40-7.45 (m, 1H), 7.18-7.13 (m, 2H), 6.93 (dd, *J* = 8.25, 2.13 Hz, 1H), 3.89 (s, 3H), 3.83 (s, 3H), 3.67 (br t, 4H), 2.05 (br t , 4H); ^{13}C-NMR (75 MHz, $CDCl_3$) δ 163.7, 160.0, 159.9, 156.9, 143.5, 142.6, 135.6, 133.2, 131.0, 129.5, 129.0, 128.8, 124.4, 119.0, 114.1, 112.4, 86.8, 55.6, 52.1, 49.8, 25.6; IR (KBr) 2952, 2873, 1738, 1683, 1589, 1489, 1271, 1089, 835 ν_{max}/cm^{-1}; Mp 162.2-163.1 °C; MS (ESI) *m/z* Calcd for $C_{25}H_{22}ClN_3O_5$: 479.1248; Found: 480.1327 $[M+H]^+$.

Methyl 5-(3-methoxyphenyl)-2-morpholino-4-oxo-7-phenyl-4,5-dihydrofuro[2,3-*d*]pyridazine-3-carboxylate (30). The crude material was purified by column chromatography (PE/EtOAc 6:4) to give the product as white solid (185 mg, 50% yield). ^{1}H-NMR (300 MHz, $CDCl_3$) δ 7.99 (br d, 2H), 7.47-7.33 (m, 4H), 7.21-7.15 (m, 2H), 6.92 (dd, *J* = 8.1, 2.1 Hz, 1H), 3.91 (s, 3H), 3.82 (s, 3H), 3.66 (br t, 4H), 2.02 (br t, 4H); ^{13}C-NMR (75 MHz, $CDCl_3$) δ 163.8, 160.0, 159.9, 156.9, 143.6, 142.9, 134.4, 132.5, 129.5, 129.4, 128.7, 127.5, 124.2, 119.0, 114.0, 112.4, 86.7, 55.5, 52.0, 49.7, 25.5; IR (KBr) 2955, 2877, 1687, 1601, 1580, 1491, 1270, 1087, 767 ν_{max}/cm^{-1}; Mp 194.7-195.8 °C; MS (ESI) *m/z* Calcd for $C_{25}H_{23}N_3O_6$: 461.1587; Found: 462.1664 $[M+H]^+$.

Methyl 5-(3-methoxyphenyl)-4-oxo-7-phenyl-2-(pyrrolidin-1-yl)-4,5-dihydrofuro[2,3-*d*]pyridazine-3-carboxylate (31). The crude material was purified by column chromatography (PE/EtOAc 6:4) to give the product as white solid (89 mg, 25% yield). ^{1}H-NMR (300 MHz, $CDCl_3$) δ 7.96 (d, *J* = 7.6 Hz, 2H), 7.48-7.35 (m, 4H), 7.20-7.14 (m, 2H), 6.94 (br d, 1H), 3.91 (s, 3H), 3.89-3.86 (m, 4H), 3.83 (s, 3H), 3.67-3.34 (m, 4H); ^{13}C-NMR (75 MHz, $CDCl_3$) δ 163.9, 161.6, 160.0, 156.8, 143.6, 143.5, 134.7, 132.3, 129.8, 129.6, 128.9, 127.6, 123.7, 119.0, 114.2, 112.5, 90.7, 66.4, 55.6, 52.4, 48.4; IR (KBr) 2952, 2857, 1687, 1673, 1577, 1492, 1288, 1070, 1031, 773 ν_{max}/cm^{-1}; Mp 184.2-184.8 °C; MS (ESI) *m/z* Calcd for $C_{25}H_{23}N_3O_5$: 445.1638; Found: 446.1676 $[M+H]^+$.

Methyl 4-oxo-5,7-diphenyl-2-(pyrrolidin-1-yl)-4,5-dihydrofuro[2,3-*d*]pyridazine-3-carboxylate (32). The crude material was purified by column chromatography (PE/EtOAc 7:3) to give the product as white solid (103 mg, 31% yield). ^{1}H-NMR (300 MHz, $CDCl_3$) δ 8.01 (d, *J* = 7.6 Hz, 2H), 7.62 (d, *J* = 7.6 Hz, 2H), 7.50-7.42 (m, 5H), 7.39-7.35 (m, 1H), 3.90 (s, 3H), 3.70-3.66 (m, 4H), 2.06-2.02 (m, 4H); ^{13}C-NMR (75 MHz, $CDCl_3$) δ 163.9, 160.1, 157.2, 143.0, 142.6, 134.6, 132.6, 129.6, 128.8, 127.9, 127.6, 127.3, 126.6, 124.2, 86.8, 52.1, 49.8, 25.6; IR (KBr) 2972, 2875, 1709, 1670, 1613, 1585, 1496, 1263, 1080, 912, 773 ν_{max}/cm^{-1}; Mp 231.1-231.6 °C; MS (ESI) *m/z* Calcd for $C_{24}H_{21}N_3O_4$: 415.1532; Found: 416.1614 $[M+H]^+$.

Methyl 4-oxo-5-phenyl-2-(pyrrolidin-1-yl)-7-(*p*-tolyl)-4,5-dihydrofuro[2,3-*d*]pyridazine-3-carboxylate (33). The crude material was purified by column chromatography (PE/EtOAc 7:3) to give the product as white solid (120 mg, 35% yield). ^{1}H-NMR (300 MHz, $CDCl_3$) δ 7.88 (d, AA'XX', *J* = 8.25 Hz, 2H), 7.63 (d, AA'XX', *J* = 8.25 Hz, 2H), 7.50-7.46 (m, 2H), 7.43-7.33 (m, 1H), 7.27-7.25 (m, 2H), 3.92 (s, 3H), 3.68-3.67 (m, 4H), 2.40 (br s, 3H), 2.05-2.03 (m,4H); ^{13}C-NMR (75 MHz, $CDCl_3$) δ 163.8, 160.0, 157.0, 142.9, 142.6, 139.6, 134.5, 129.7, 129.4, 128.6, 127.8, 126.5, 124.1, 86.7, 52.0, 49.6, 25.5, 21.4; IR (KBr) 2968, 2862, 1680, 1565, 1442, 1078, 988

ν_{max}/cm^{-1}; Mp 220.2-220.9 °C; MS (ESI) *m/z* $(M+H)^+$ Calcd for $C_{25}H_{23}N_3O_4$: 429.1689; Found: 430.1735 $[M+H]^+$.

Methyl 2-(diethylamino)-4-oxo-5,7-diphenyl-4,5-dihydrofuro[2,3-*d*]pyridazine-3-carboxylate (34). The crude material was purified by column chromatography (PE/EtOAc 7:3) to give the product as white solid (104 mg, 31% yield). ^{1}H-NMR (300 MHz, $CDCl_3$) δ 8.03 (d, *J* = 7.8 Hz, 2H), 7.67 (d, *J* = 7.8 Hz, 2H), 7.53-7.39 (m, 6H), 3.96 (s, 3H), 3.65 (q, *J* = 7.0 Hz, 4H), 1.31 (t, *J* = 7.0 Hz, 6H); ^{13}C-NMR (75 MHz, $CDCl_3$) δ 164.4, 160.3, 157.1, 142.8, 142.6, 134.6, 132.7, 129.6, 128.8, 128.0, 127.6, 126.6, 124.7, 87.9, 52.5, 45.3, 13.5; IR (KBr) 2865, 1698, 1667, 1611, 1572, 1487, 1315, 891, 801 ν_{max}/cm^{-1}; Mp 225.2-226.1 °C; MS (ESI) *m/z* $(M+H)^+$ Calcd for $C_{24}H_{23}N_3O_4$: 417.1689; Found: 418.1769 $[M+H]^+$.

Preparation of Lithium 7-(4-iodophenyl)-2-(4-methylpiperazin-1-yl)-4-oxo-5-phenyl-4,5-dihydrofuro[2,3-*d*]pyridazine-3-carboxylate (35). The furo[2,3-*d*]pyridazin-4(5*H*)-one **25** (0.05 mmol, 1 eq.) was dissolved in THF/H_2O 3:1 (0.2M), lithium hydroxide (0.05 mmol, 1 eq.) was added and the rection mixture was stirred at 100°C overnight. The hydrolysis of the ester function was monitored by TLC (95:5 DCM/MeOH). The conversion as revealed by NMR of the crude reaction mixture was quantitative. ^{1}H-NMR (400 MHz, D_2O-DMSO-d_6) δ 7.86 (br d, AA'XX', 2H), 7.71 (br d, AA'XX', 2H), 7.52-7.37 (m, 5H), 3.51-3.48 (m, 4H), 2.43-2.39 (m, 4H), 2.16 (s, 3H); ^{13}C-NMR (100 MHz, D_2O-DMSO-d_6) δ 167.7, 166.4, 158.6, 158.4, 142.6, 141.9, 138.2 (2C), 134.1, 132.0, 129.8 (2C), 129.1 (2C), 128.4, 126.8 (2C), 124.9, 96.7, 54.2 (2C), 47.2 (2C), 45.9; IR (KBr) 2934, 2780, 17111, 1569, 1489, 1358, 1144, 998 ν_{max}/cm^{-1}; Mp 204-205°C; MS (ESI) *m/z* Calcd for $C_{24}H_{21}IN_4O_4$: 556.0607; Found: 557.0647 $[M+H]^+$.

References:

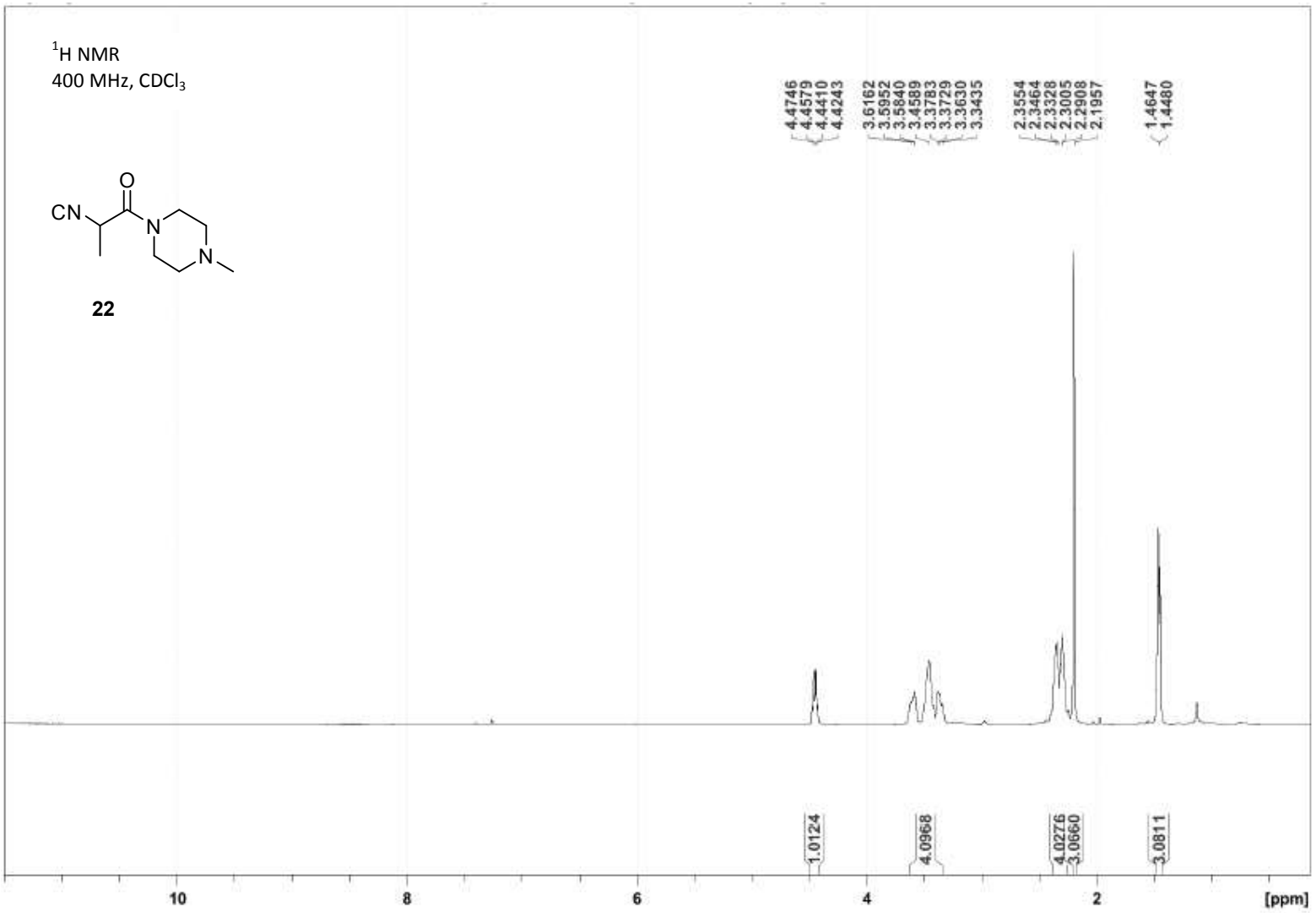
[1] Mossetti R., Caprioglio D., Colombano G., Tron G. C., Pirali T. *Org. Biomol. Chem.* **2011**, (9), 1627-1631.

[2] Pirali T., Tron G. C., Zhu J. *Org. Lett.* **2006**, *8* (18), 4145-4148.

[3] Schöllkopf U., Hausberg H. H., Segal M., Reiter U., Hoppe I., Saenger W., Lindner K. *Liebigs Ann. Chem.* **1981**, (3), 439-58

[4] Giustiniano M., Meneghetti F., Mercalli V., Varese M., Giustiniano F., Novellino E., Tron G.C. *Org. Lett.* **2014**, *16* (20), 5332-5335.

[5] Patel H. V., Vyas K. A., Pandey S.P., Fernandes P. S. *Tetrahedron* **1996**, *52* (2), 661-668.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CN
O
N
N
22
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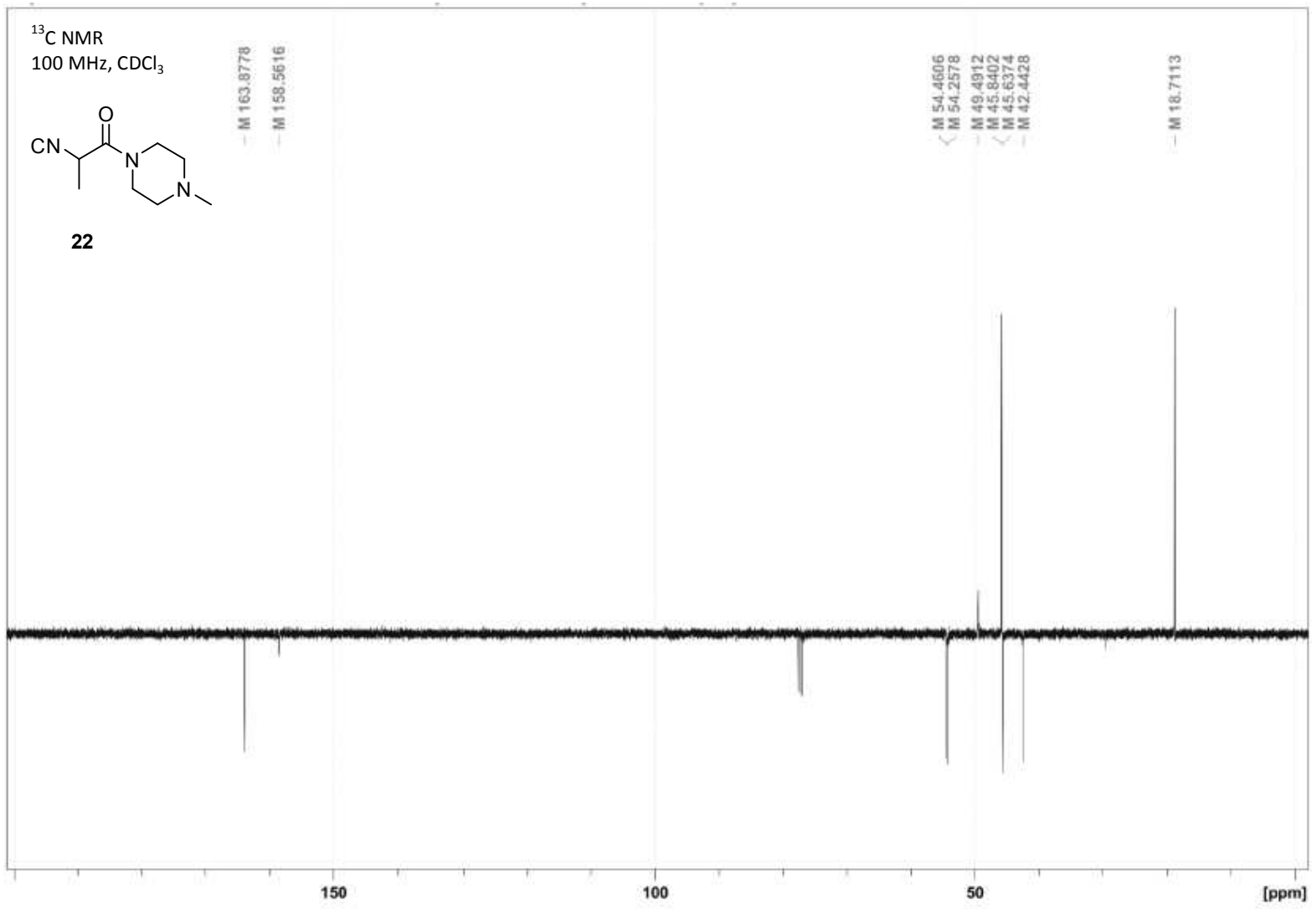
4.4579
4.4410
4.4243
3.6162
3.5952
3.5840
3.4589
3.3783
3.3729
3.3630
3.3435
2.3554
2.3464
2.3328
2.3005
2.2908
2.1957
1.4647
1.4480
1.0124
4.0968
4.0276
3.0660
3.0811
10
8
6
4
2
[ppm]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100 MHz, $CDCl_3$
CN
O
N
N
22
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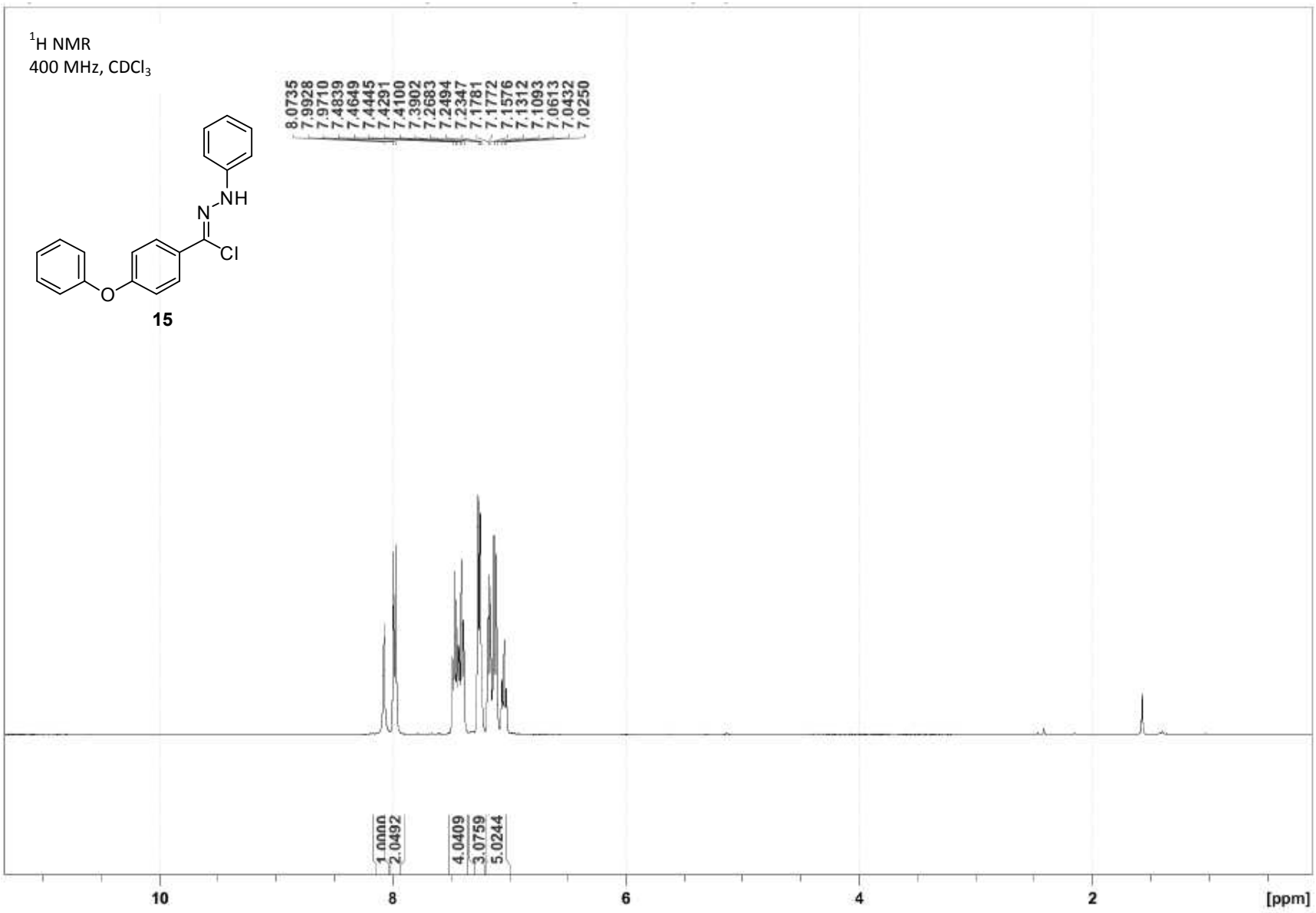
M 158.5616
M 54.4606
M 54.2578
M 49.4912
M 45.8402
M 45.6374
M 42.4428
M 18.7113
150
100
50
[ppm]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400 MHz, $CDCl_3$
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7.9710
7.4839
7.4649
7.4445
7.4291
7.4100
7.3902
7.2683
7.2494
7.2347
7.1781
7.1772
7.1576
7.1312
7.1093
7.0613
7.0432
7.0250
NH
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Cl
O
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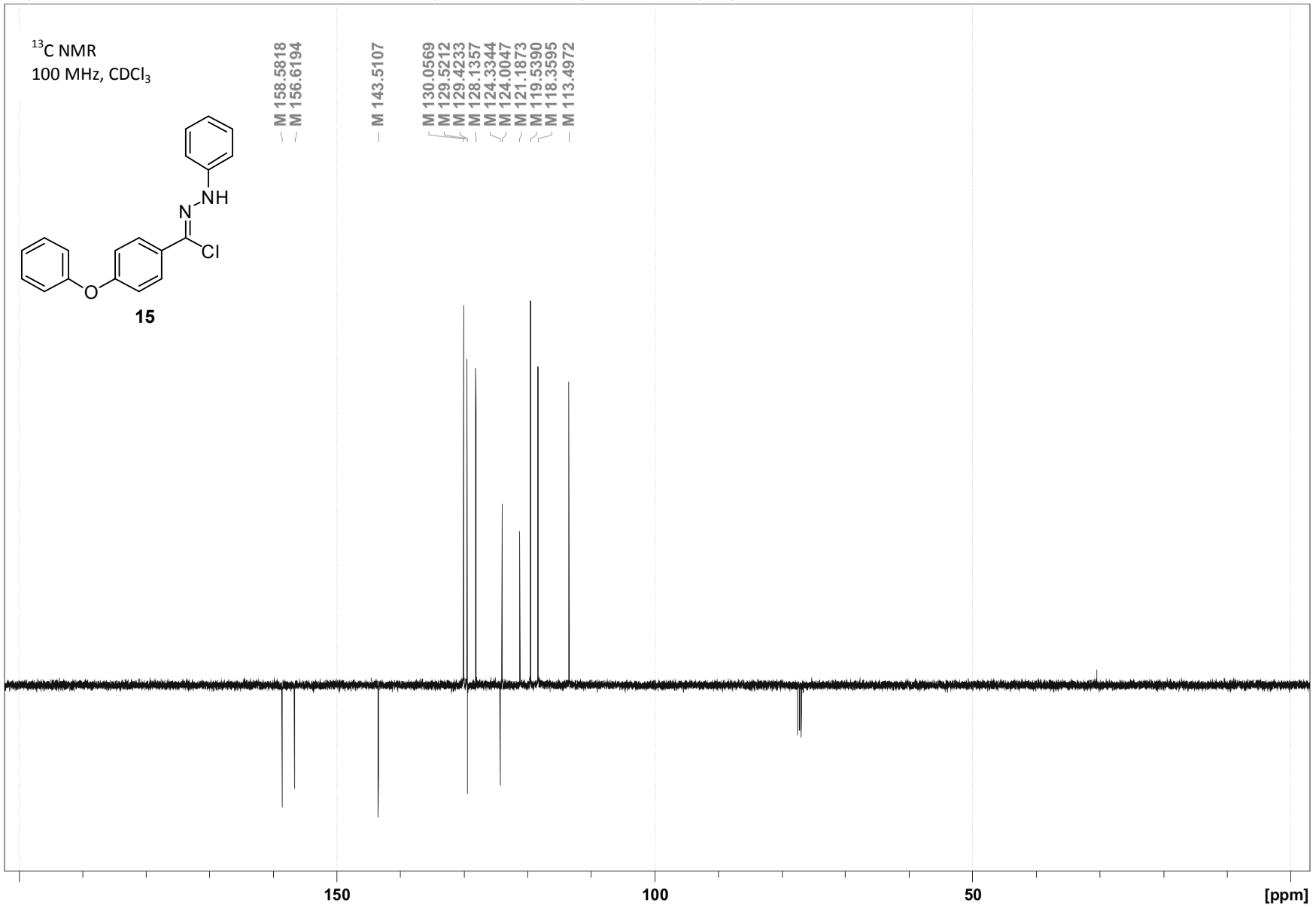
6
4
2
[ppm]


13C NMR
100 MHz, CDCl3
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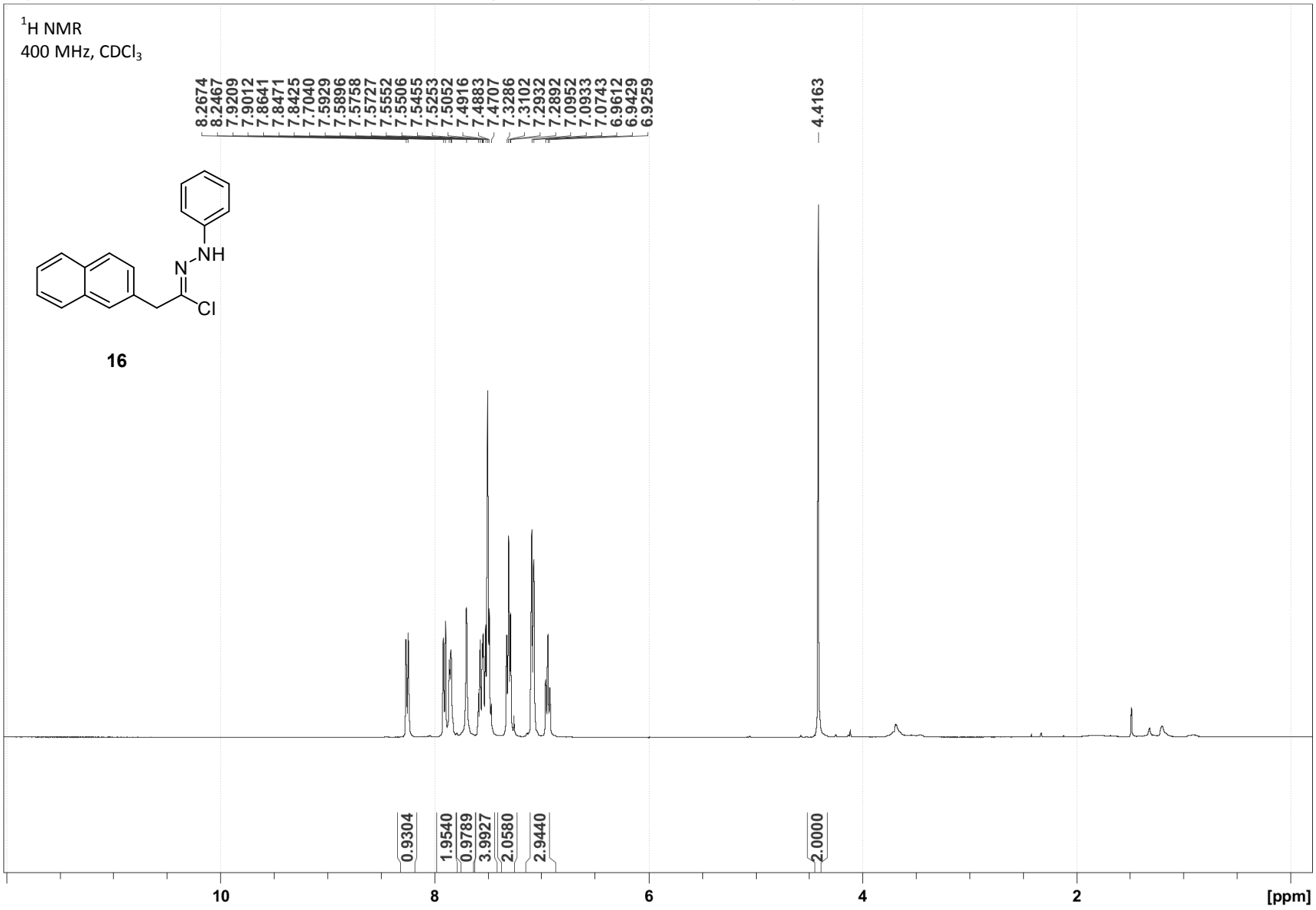
M 156.6194
M 143.5107
M 130.0569
M 129.5212
M 129.4233
M 128.1357
M 124.3344
M 124.0047
M 121.1873
M 119.5390
M 118.3595
M 113.4972
NH
N
Cl
O
15
150
100
50
[ppm]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400 MHz, CDCl3
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7.9209
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7.8641
7.8471
7.8425
7.7040
7.5929
7.5896
7.5758
7.5727
7.5552
7.5506
7.5455
7.5253
7.5052
7.4916
7.4883
7.4707
7.3286
7.3102
7.2932
7.2892
7.0952
7.0933
7.0743
6.9612
6.9429
6.9259
4.4163
NH
N
Cl
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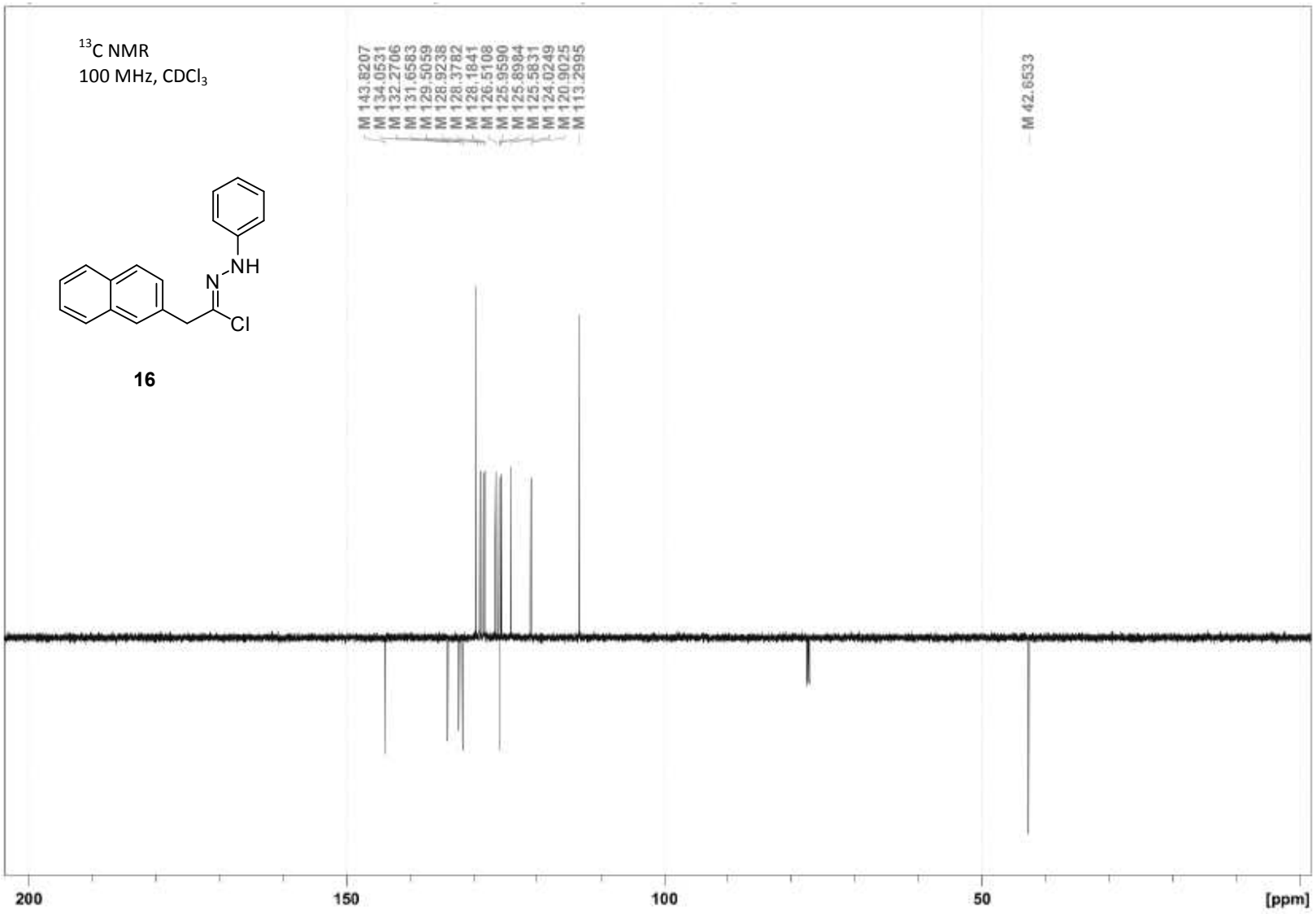
2.0580
2.9440
2.0000
10
8
6
4
2
[ppm]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100 MHz, CDCl3
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M 132.2706
M 131.6583
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M 128.9238
M 128.3782
M 128.1841
M 126.5108
M 125.9590
M 125.8984
M 125.5831
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M 120.9025
M 113.2995
M 42.6533
NH
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Cl
16
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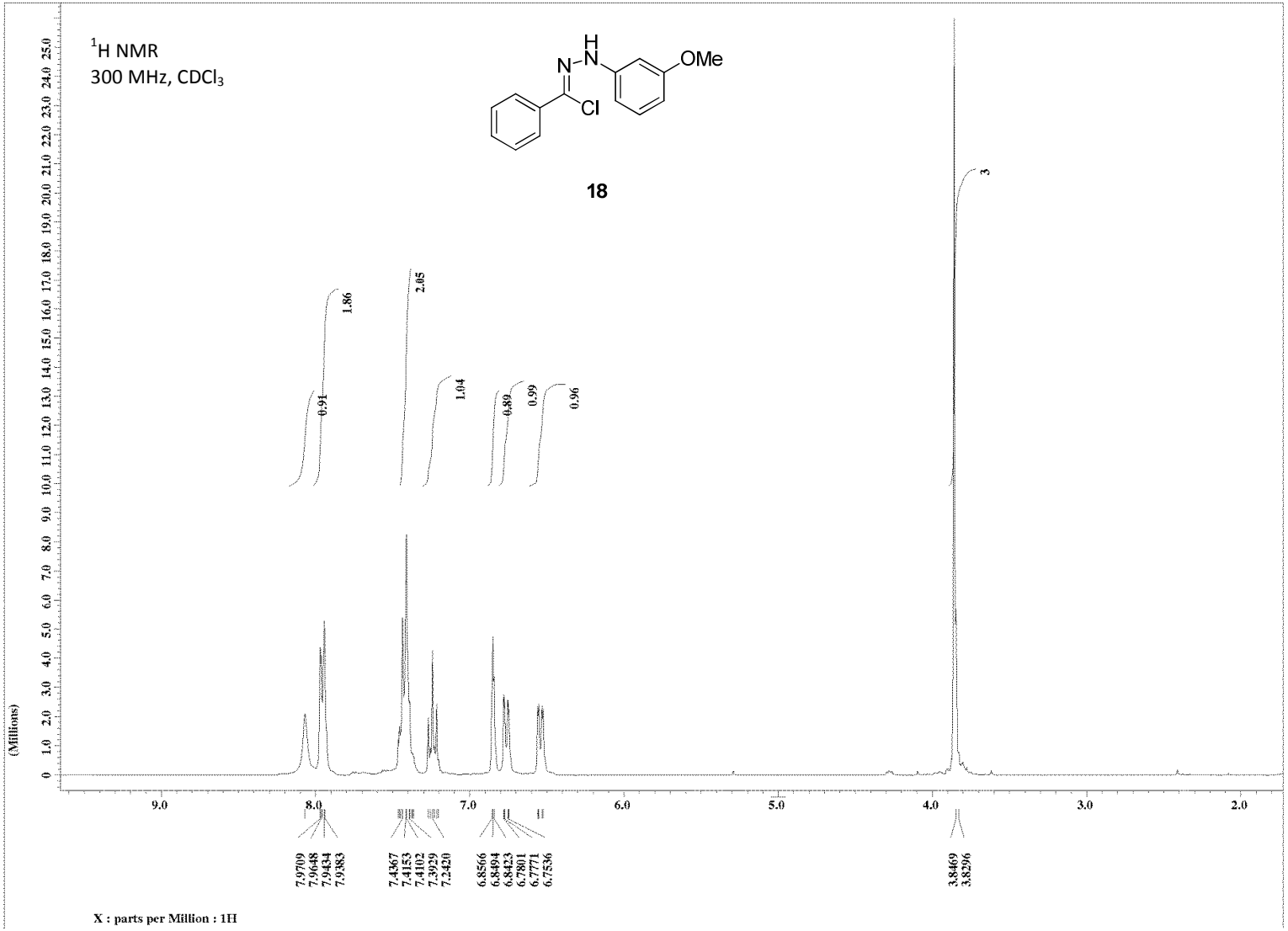
100
50
[ppm]


1H NMR
300 MHz, CDCl3
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(Millions)
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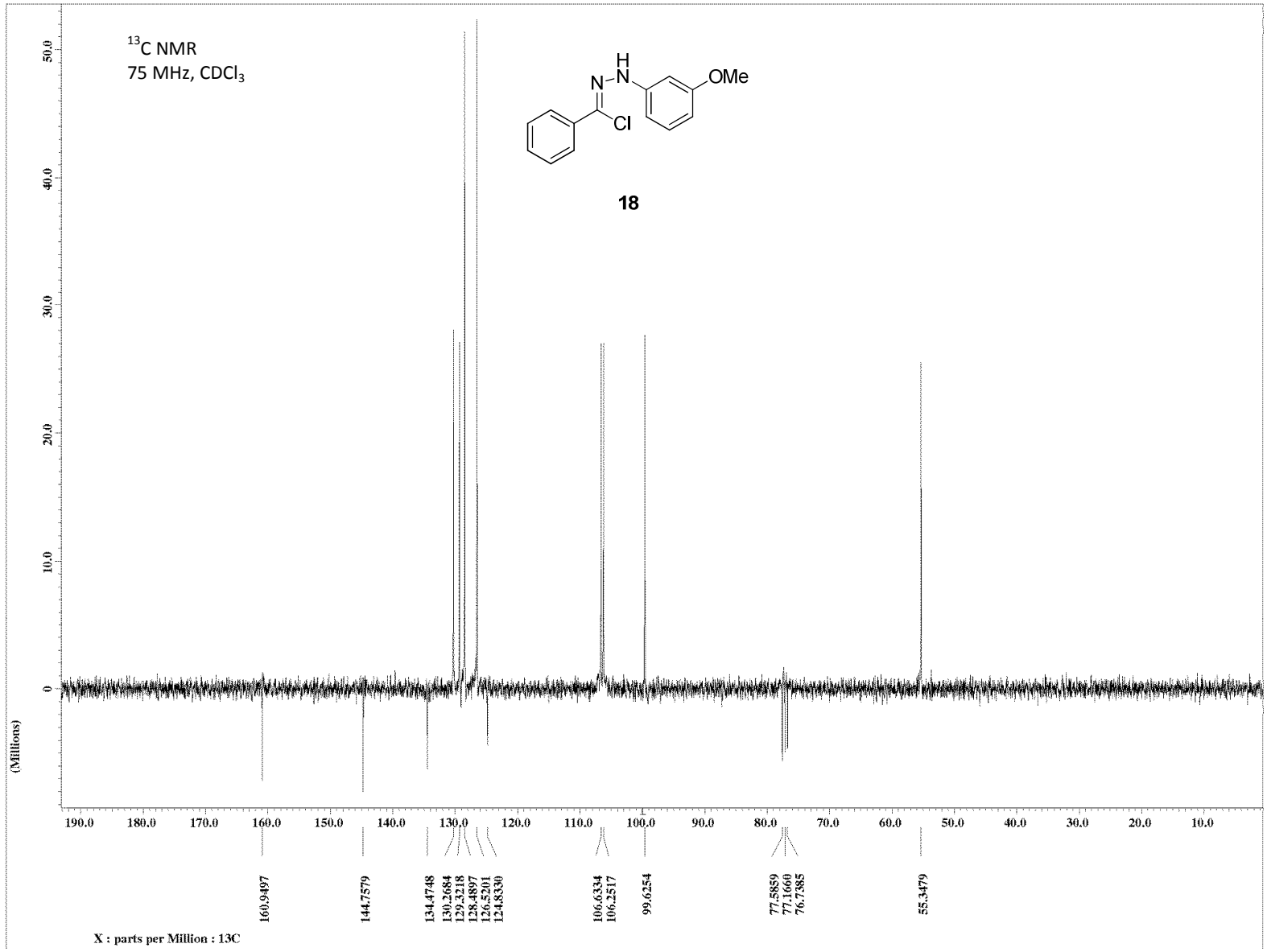
2.05
1.04
0.91
0.89
0.99
0.96
3
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
7.9709
7.9648
7.9434
7.9383
7.4367
7.4153
7.4102
7.3929
7.2420
6.8566
6.8494
6.8423
6.7801
6.7771
6.7536
3.8469
3.8296
X : parts per Million : 1H


^{13}C NMR
75 MHz, $CDCl_3$
H
N
N
OMe
Cl
18
(Millions)
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40.0
30.0
20.0
10.0
0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
160.9497
144.7579
134.4748
130.2684
129.3218
128.4897
126.5201
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106.2517
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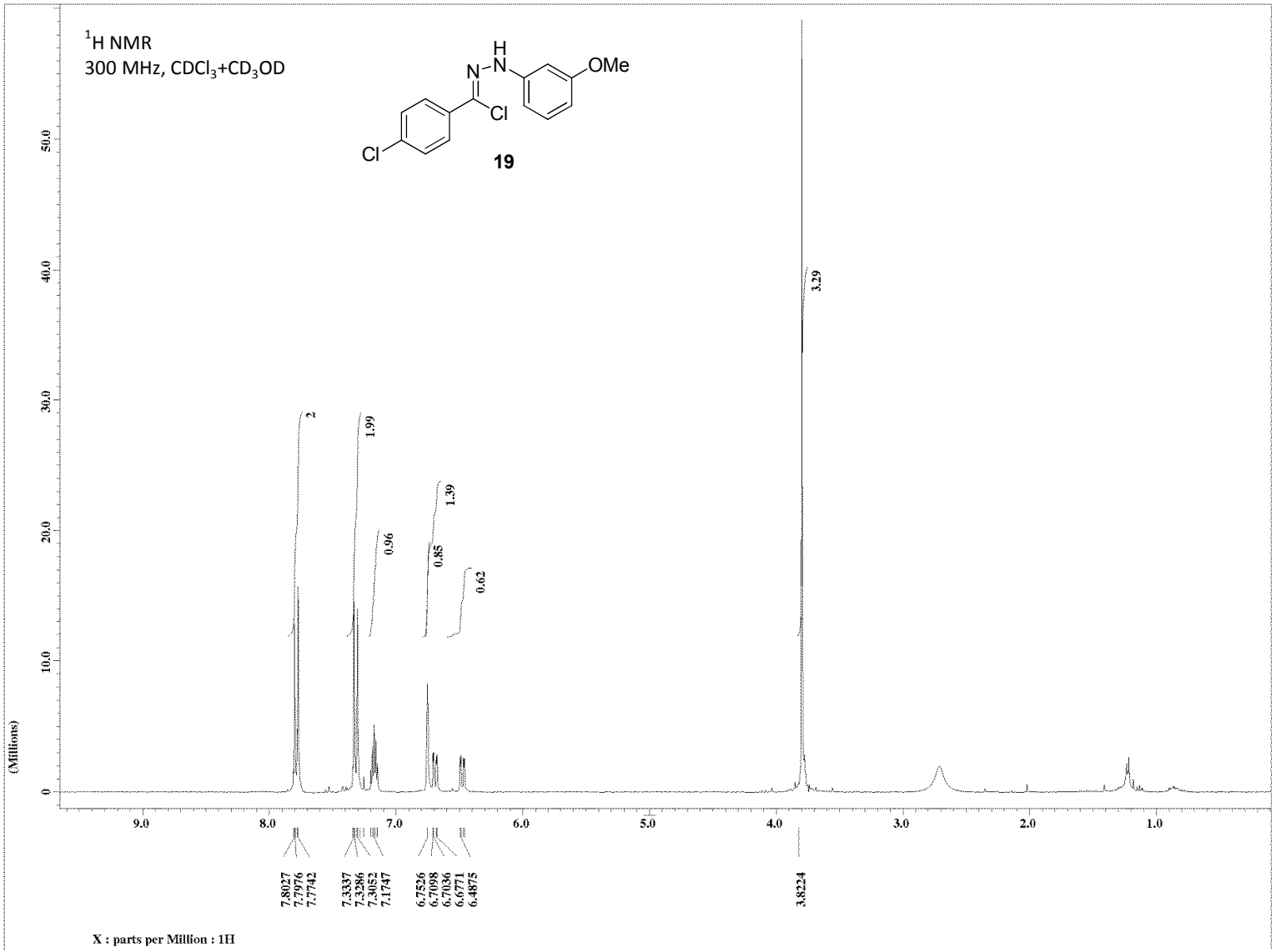
76.7385
55.3479
X : parts per Million : 13C


1H NMR
300 MHz, CDCl3+CD3OD
19
(Millions)
50.0
40.0
30.0
20.0
10.0
0
2
1.99
0.96
1.39
0.85
0.62
3.29
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
7.8027
7.7976
7.7742
7.3337
7.3286
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6.7036
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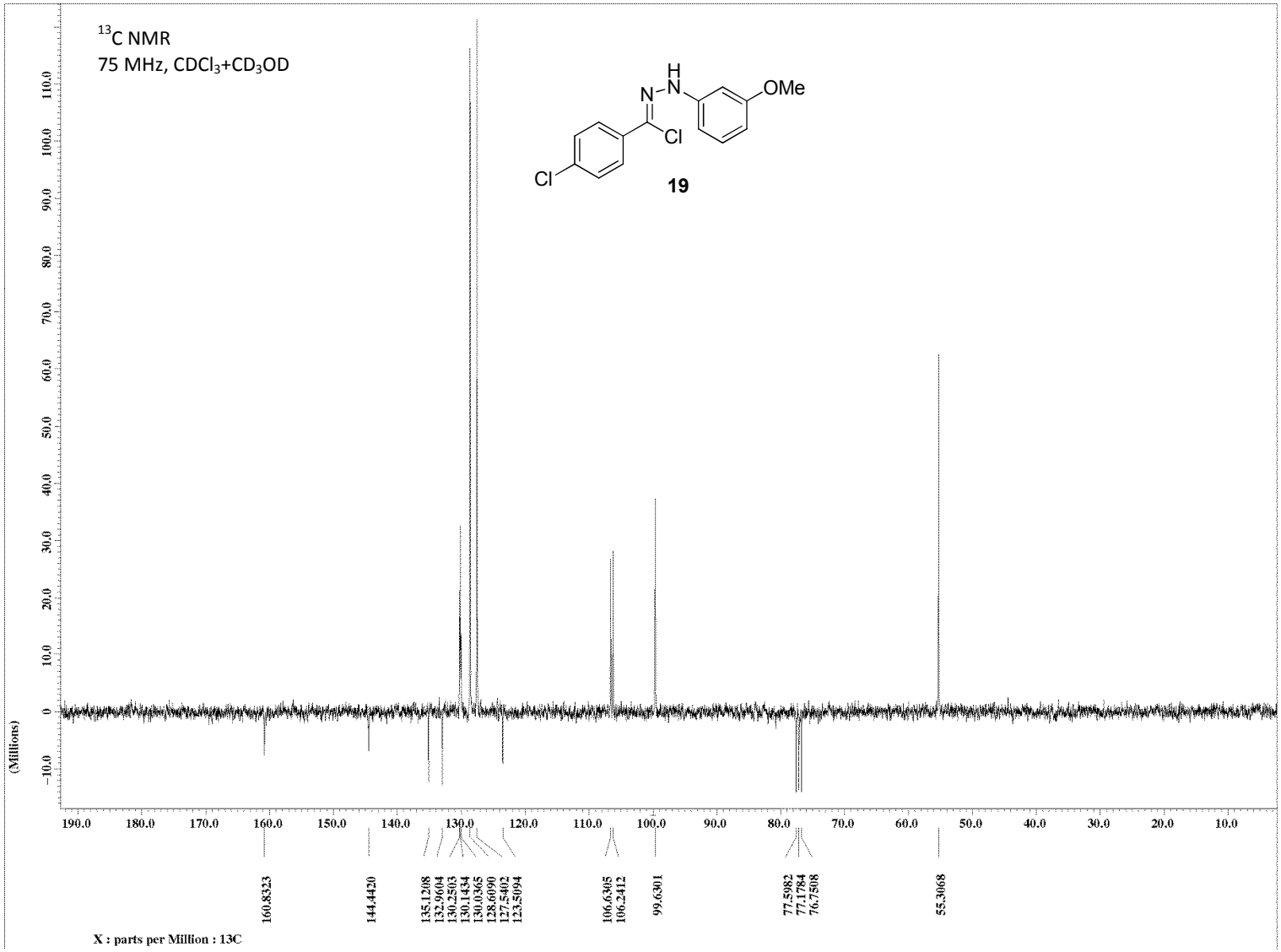
6.4875
3.8224
X : parts per Million : 1H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75 MHz, $CDCl_3$+CD_3OD
H
N
N
OMe
Cl
Cl
19
(Millions)
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100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
-10.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
160.8323
144.4420
135.1208
132.9604
130.2503
130.1434
130.0365
128.6090
127.5402
123.5094
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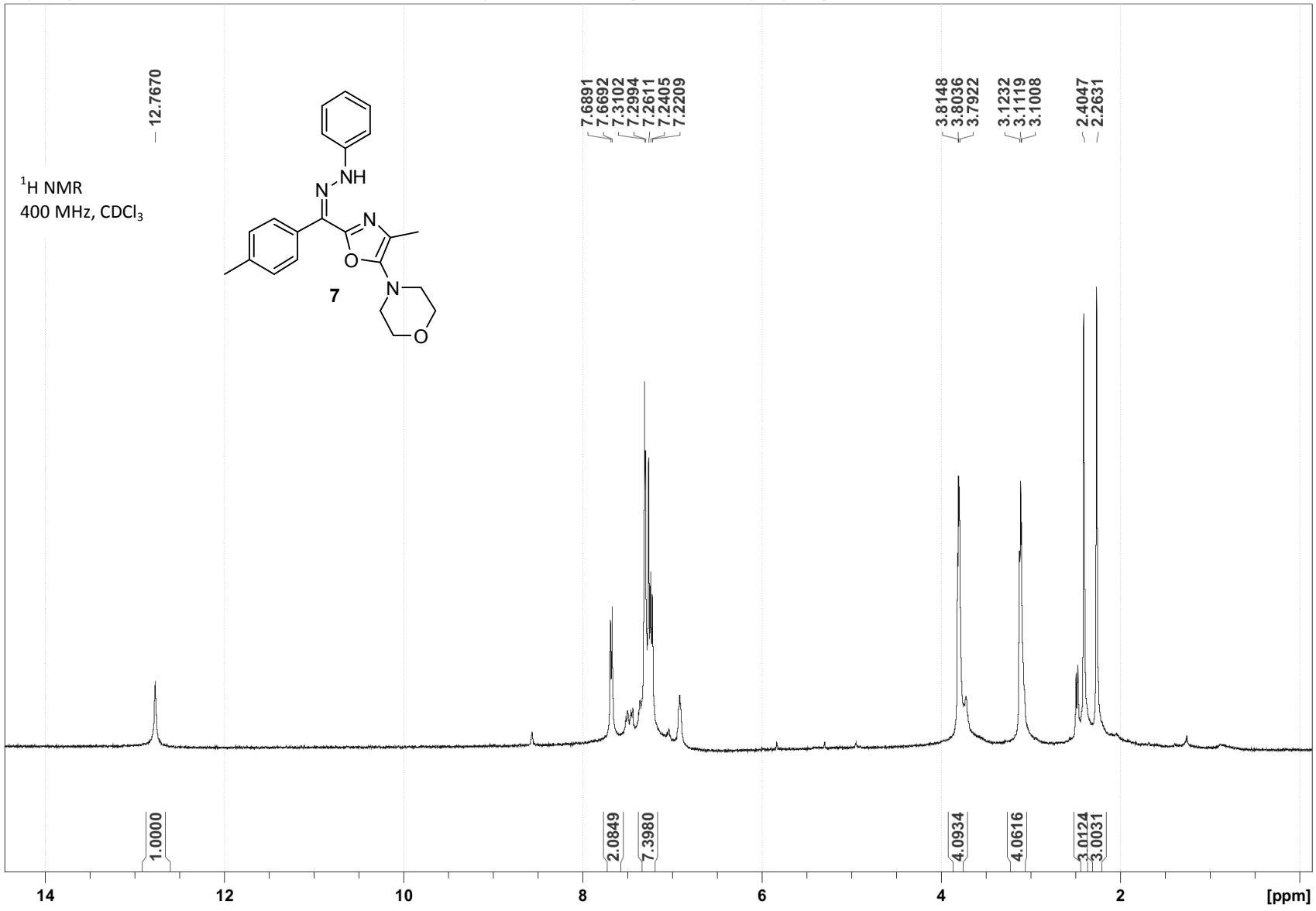
106.2412
99.6301
77.5982
77.1784
76.7508
55.3068
X : parts per Million : 13C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7.2209
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3.8036
3.7922
3.1232
3.1119
3.1008
2.4047
2.2631
1H NMR
400 MHz, CDCl3
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N
N
O
N
O
7
1.0000
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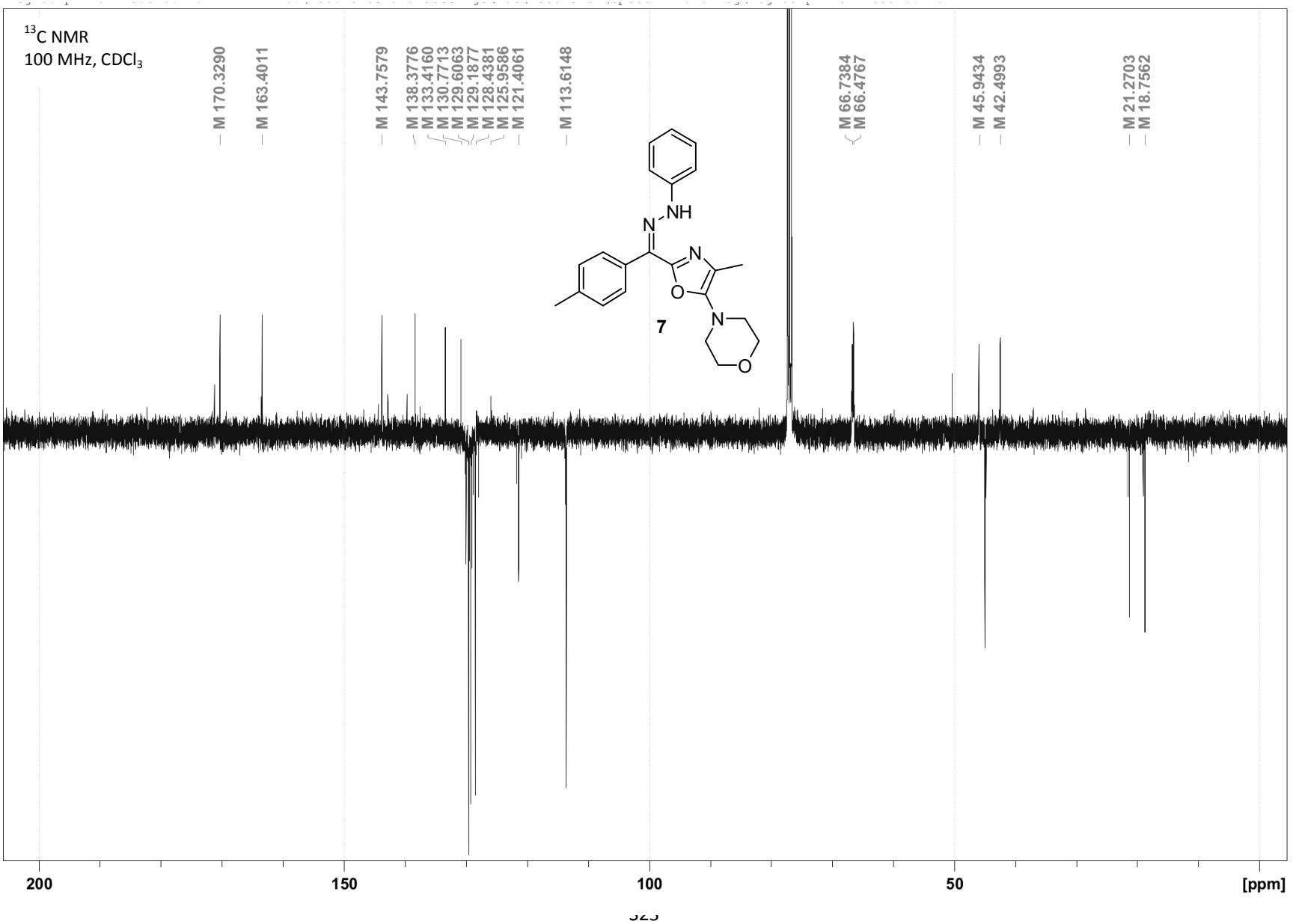
4.0934
4.0616
3.0124
3.0031
14
12
10
8
6
4
2
[ppm]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M 130.7713
M 129.6063
M 129.1877
M 128.4381
M 125.9586
M 121.4061
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M 66.7384
M 66.4767
M 45.9434
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N
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N
O
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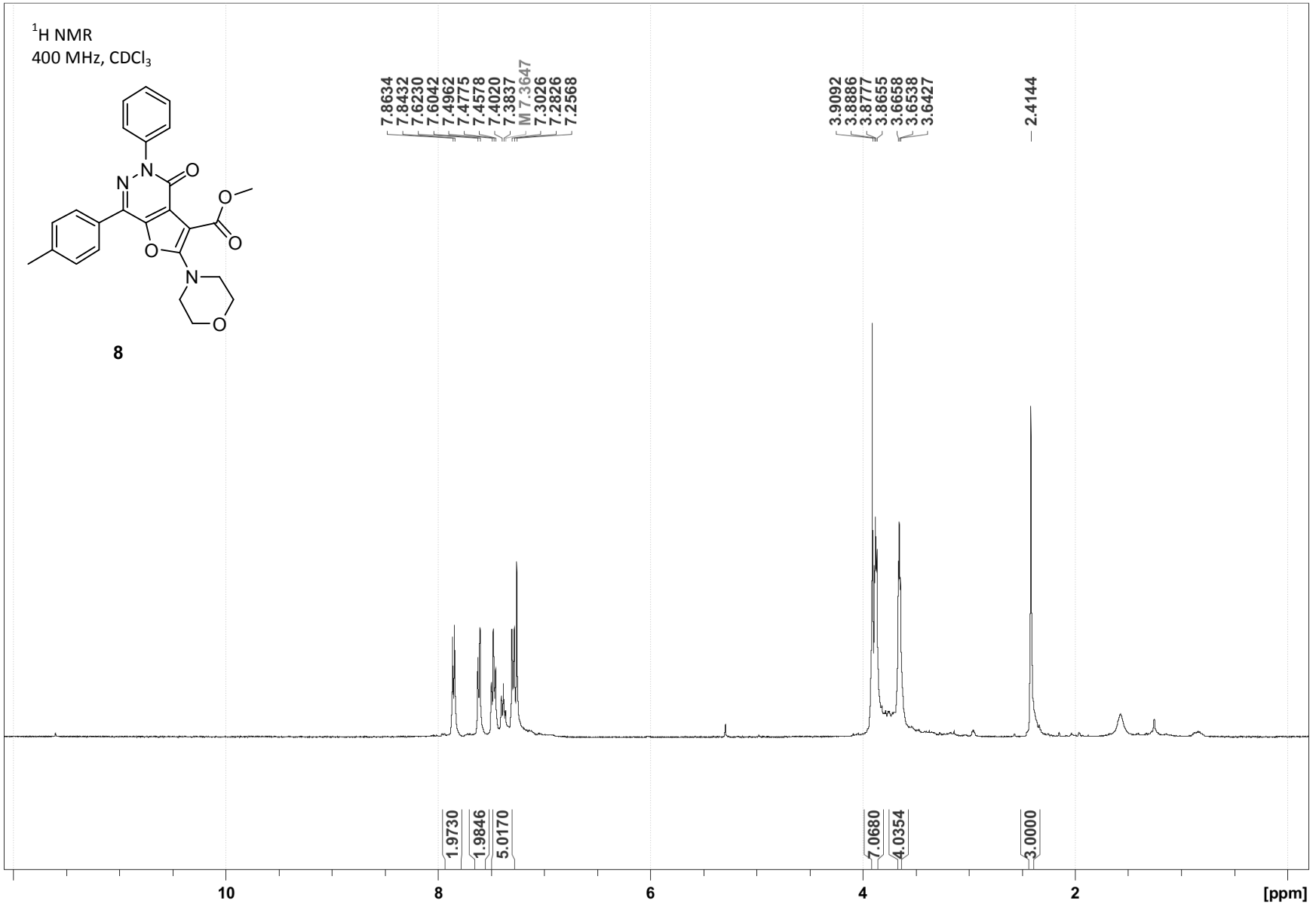
50
[ppm]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400 MHz, CDCl3
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7.8432
7.6230
7.6042
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7.4775
7.4578
7.4020
7.3837
M 7.3647
7.3026
7.2826
7.2568
3.9092
3.8886
3.8777
3.8655
3.6658
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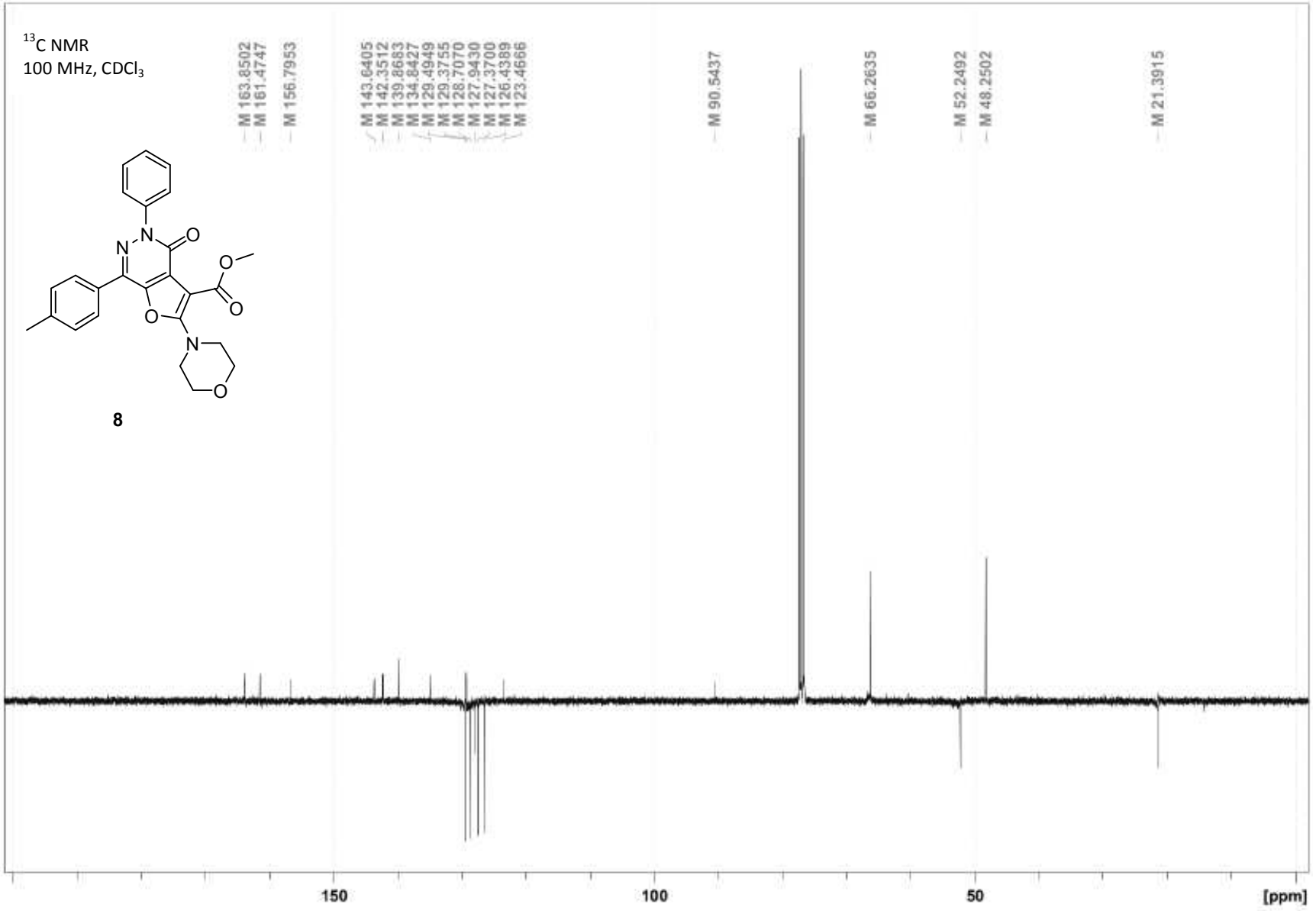
4
2
[ppm]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100 MHz, $CDCl_3$
M 163.8502
M 161.4747
M 156.7953
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M 139.8683
M 134.8427
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M 129.3755
M 128.7070
M 127.9430
M 127.3700
M 126.4389
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M 21.3915
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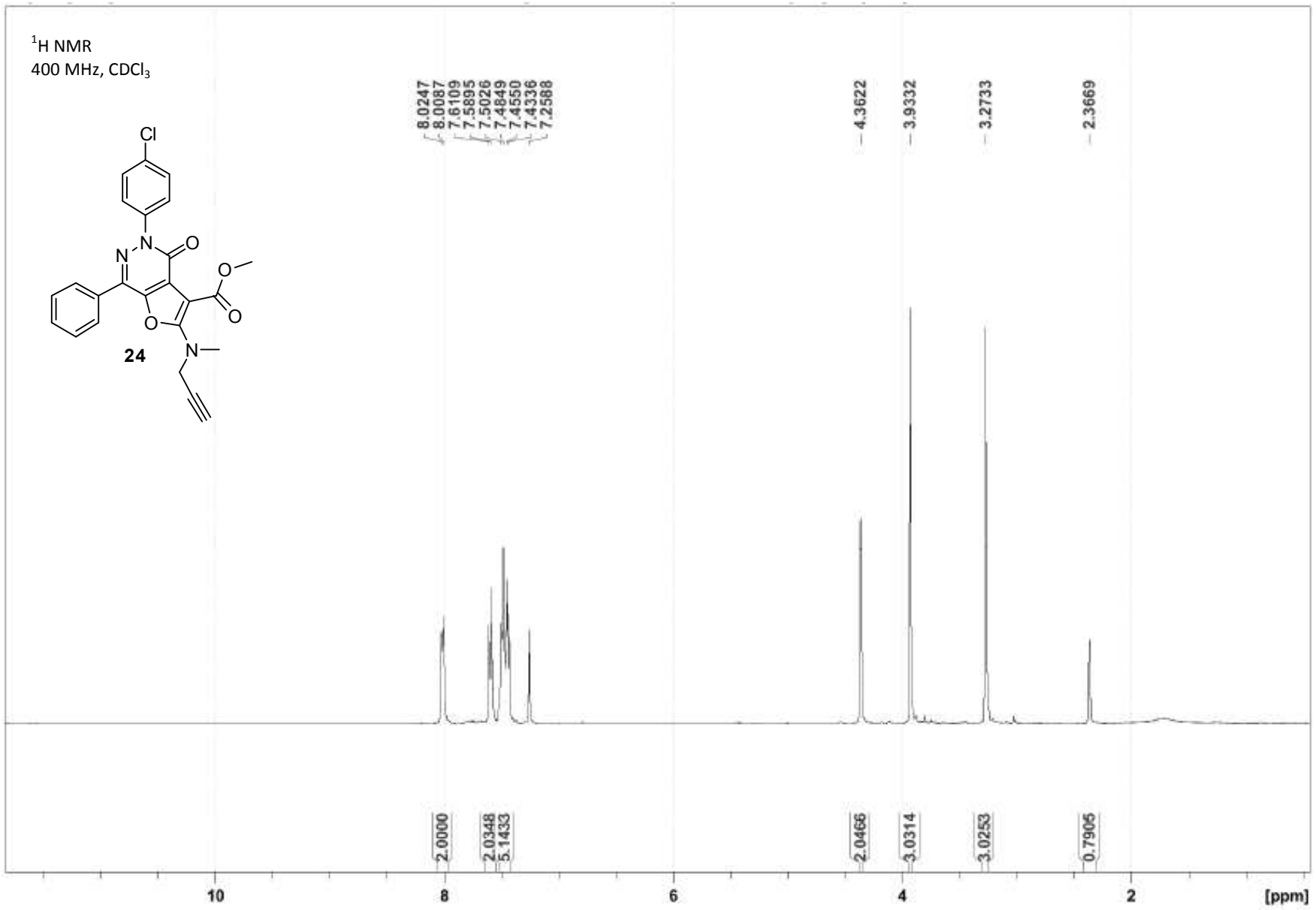
150
100
50
[ppm]


1H NMR
400 MHz, CDCl3
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O
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N
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4.3622
3.9332
3.2733
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4
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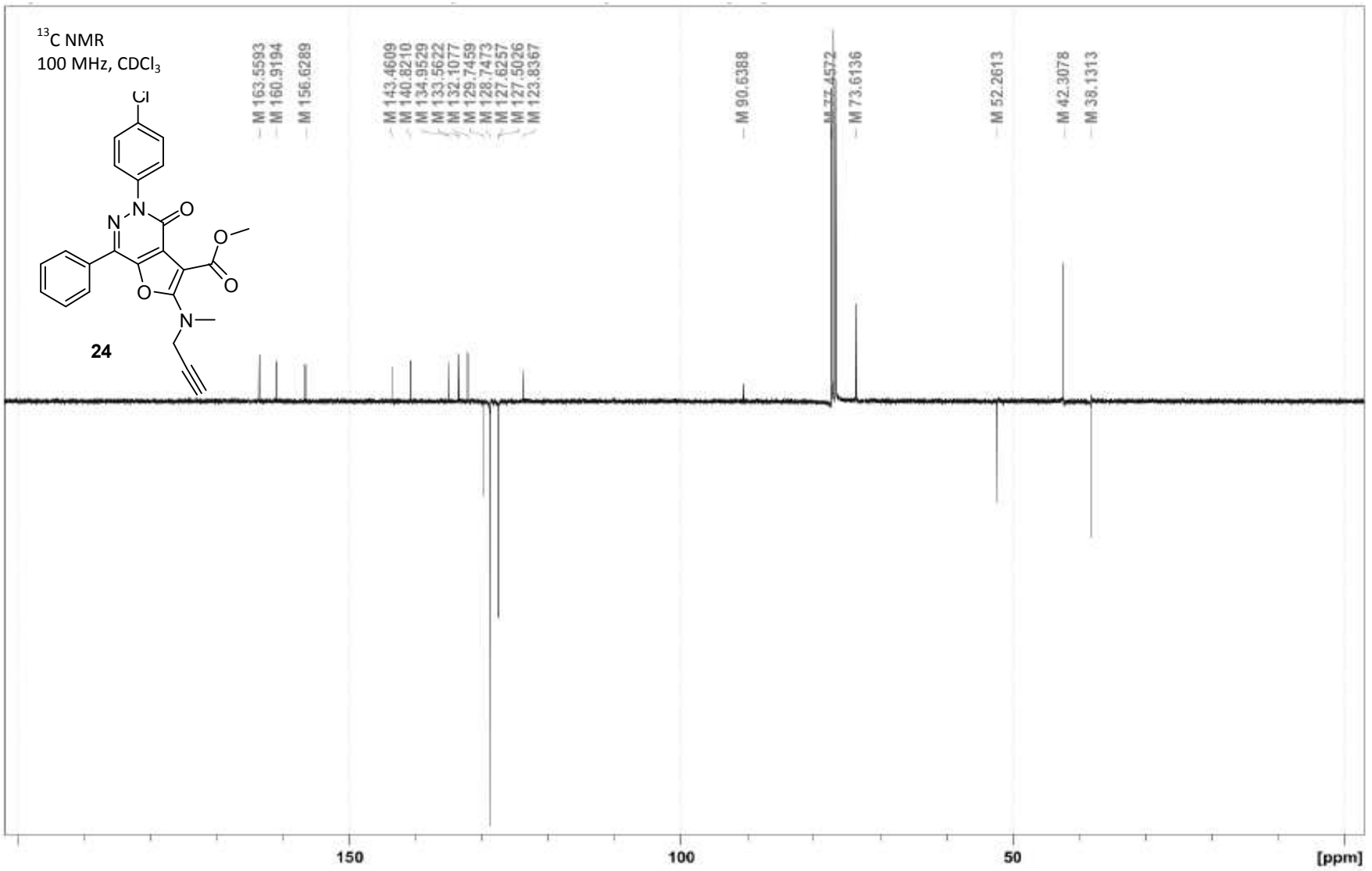
[ppm]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100 MHz, $CDCl_3$
24
M 163.5593
M 160.9194
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M 140.8210
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M 38.1313
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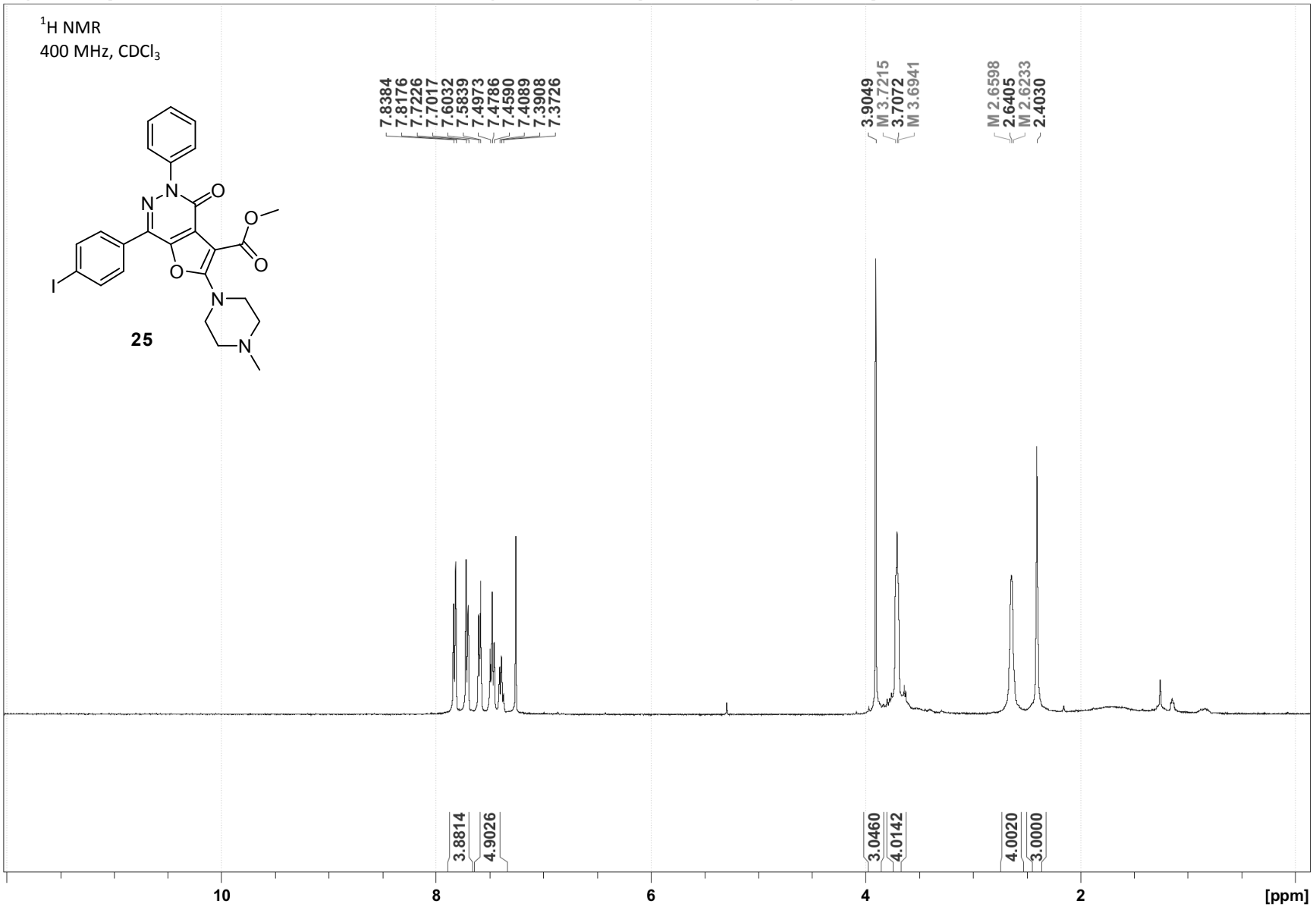
50
[ppm]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400 MHz, CDCl3
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7.4786
7.4590
7.4089
7.3908
7.3726
3.9049
M 3.7215
3.7072
M 3.6941
M 2.6598
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M 2.6233
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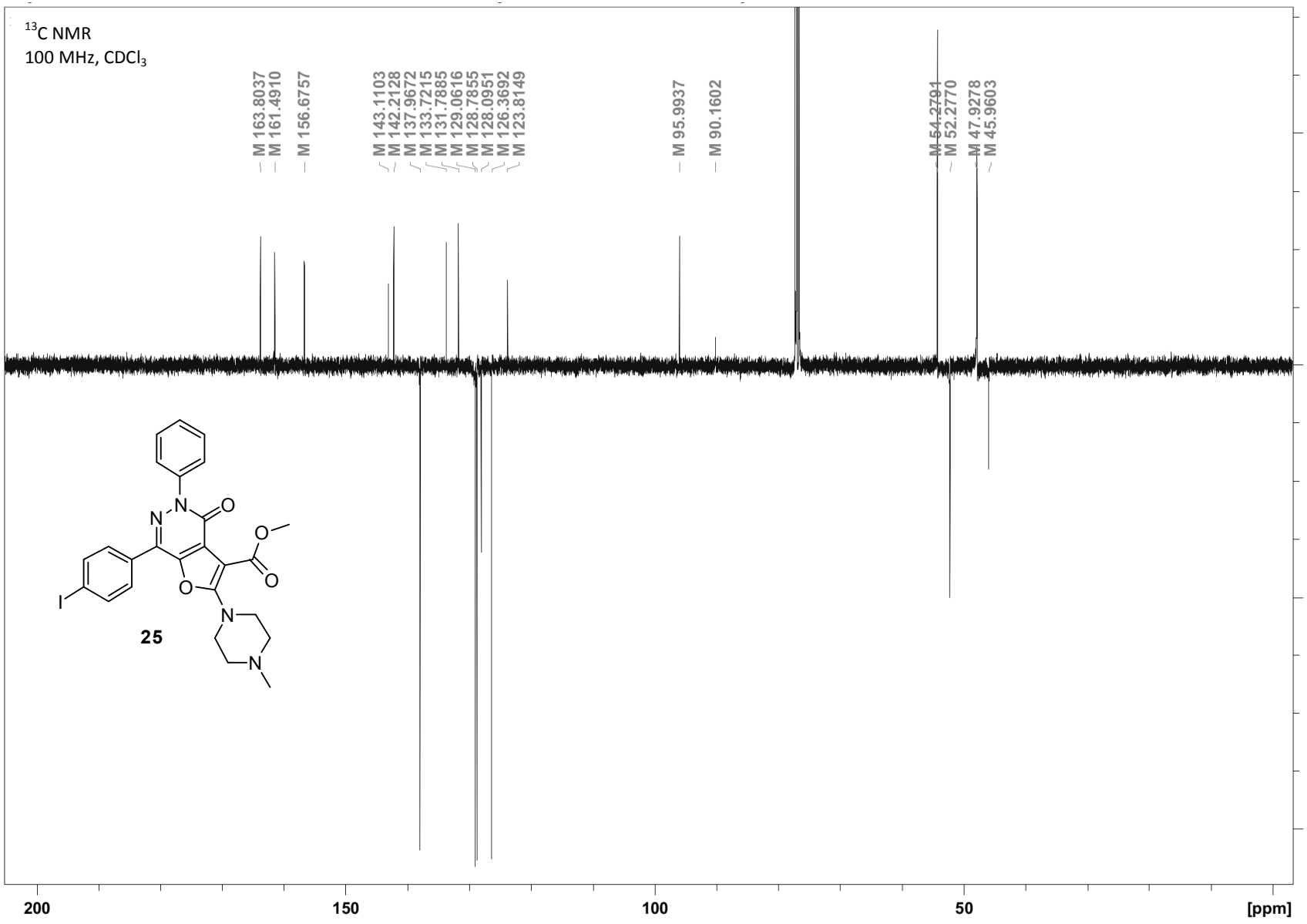
2
[ppm]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M 131.7885
M 129.0616
M 128.7855
M 128.0951
M 126.3692
M 123.8149
M 95.9937
M 90.1602
M 54.2791
M 52.2770
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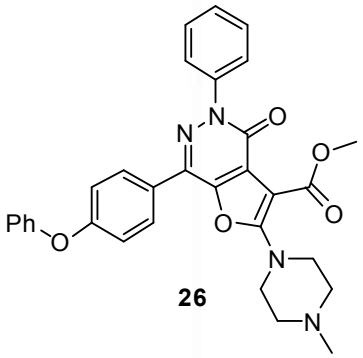
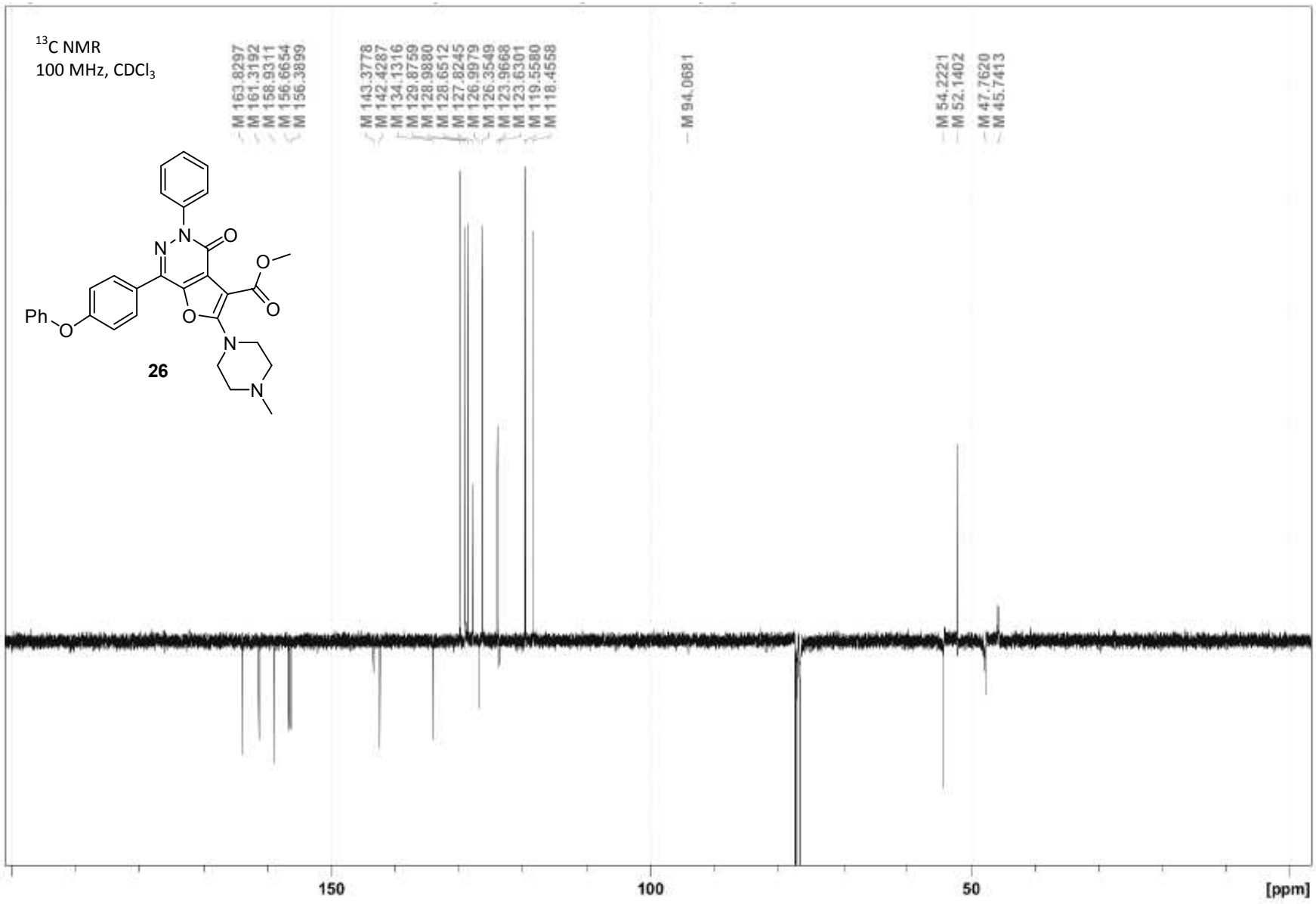
100
50
[ppm]

^{1}H NMR
400 MHz, $CDCl_3$



26


^{13}C NMR
100 MHz, $CDCl_3$
M 163.8297
M 161.3192
M 158.9311
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M 156.3899
M 143.3778
M 142.4287
M 134.1316
M 129.8759
M 128.9880
M 128.6512
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M 123.9668
M 123.6301
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M 52.1402
M 47.7620
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O
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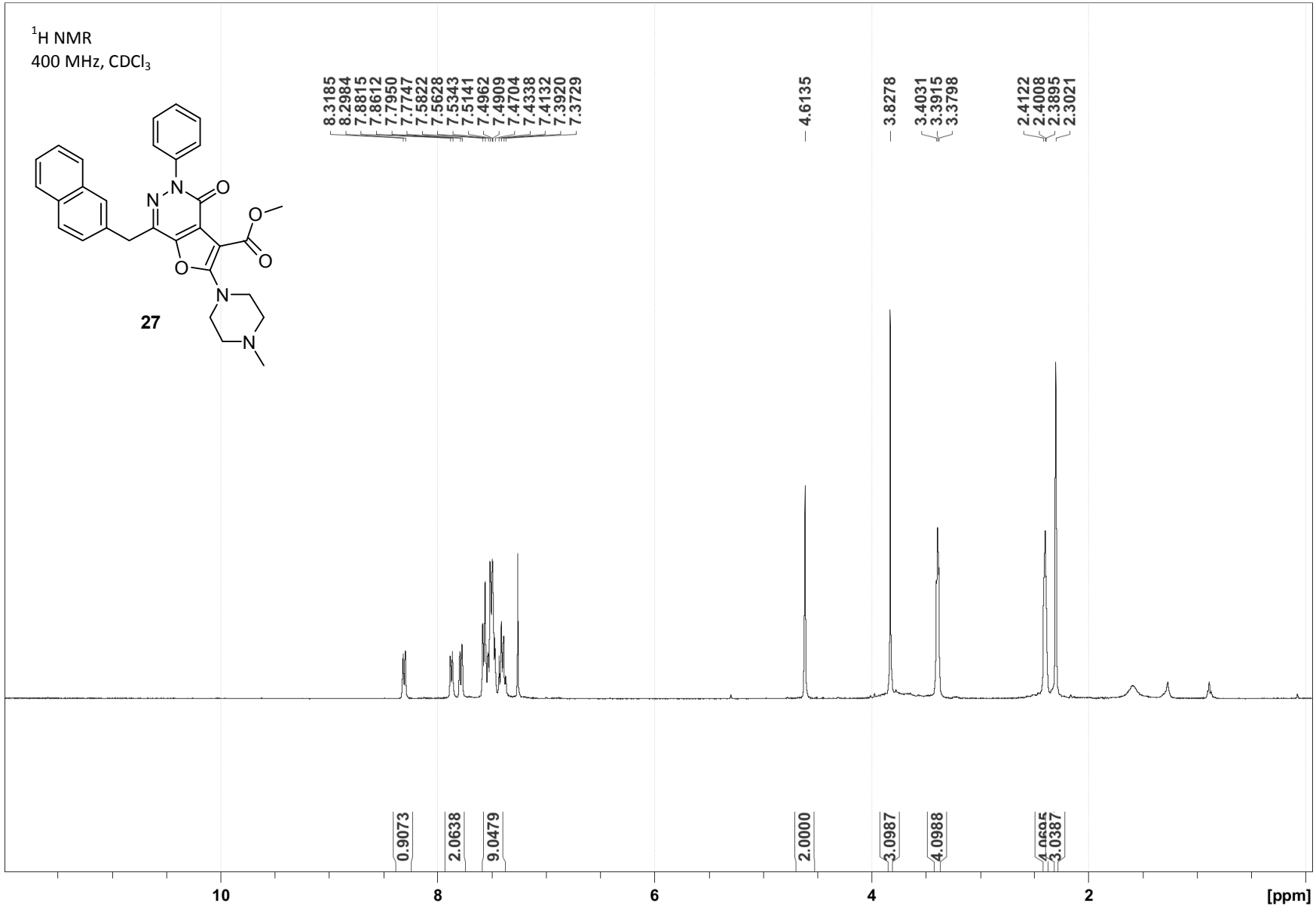
[ppm]


^{1}H NMR
400 MHz, $CDCl_3$
27
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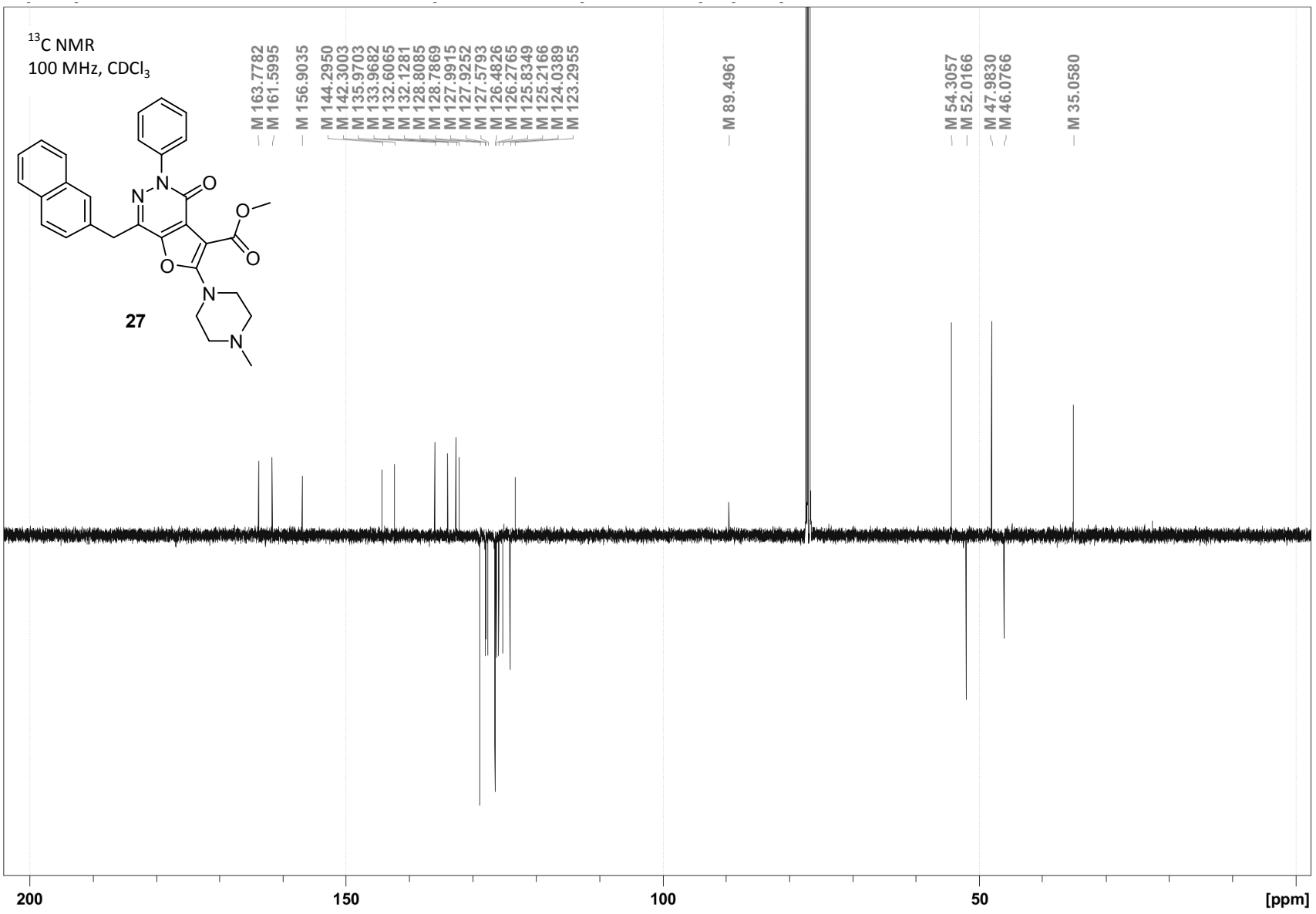
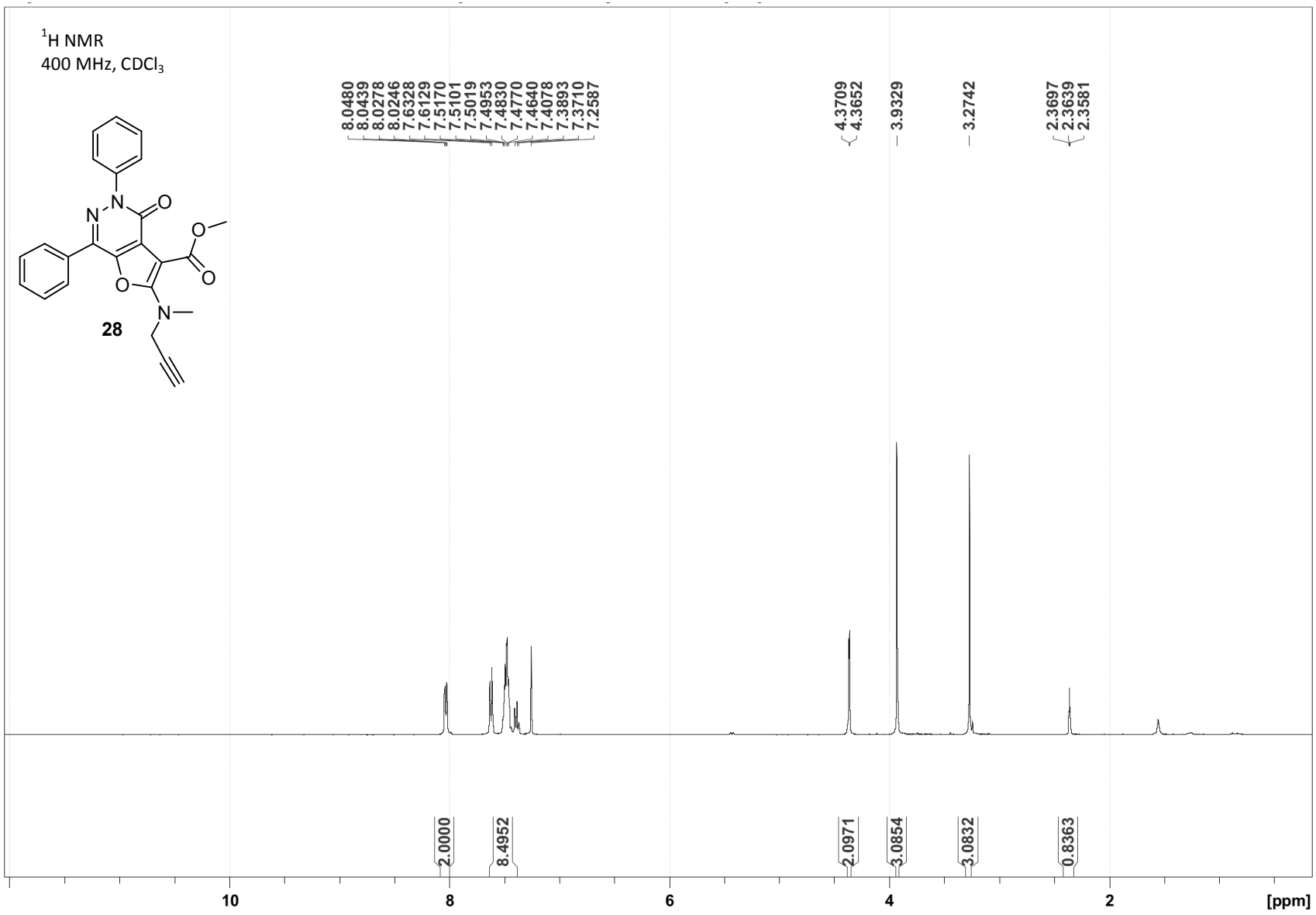
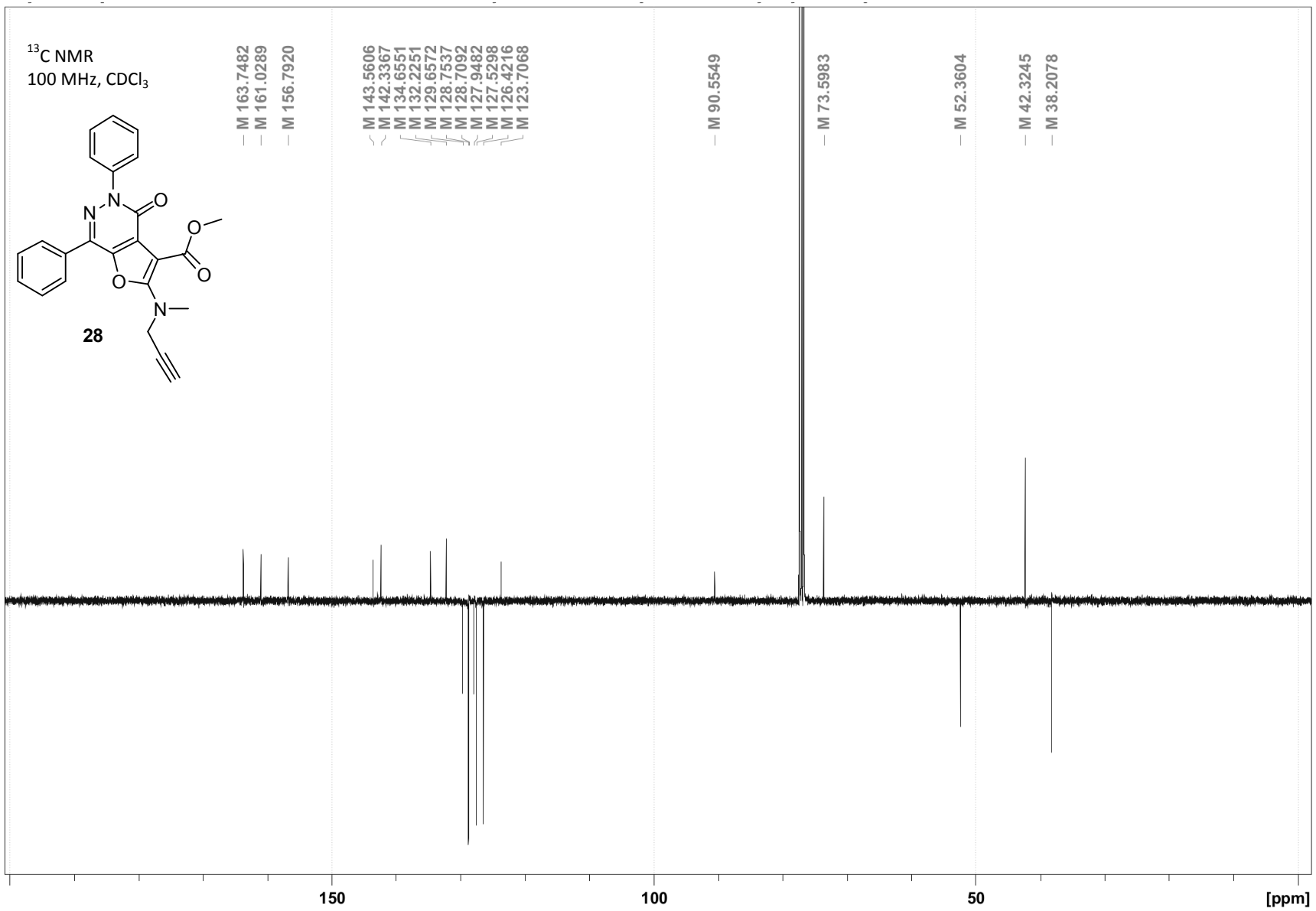
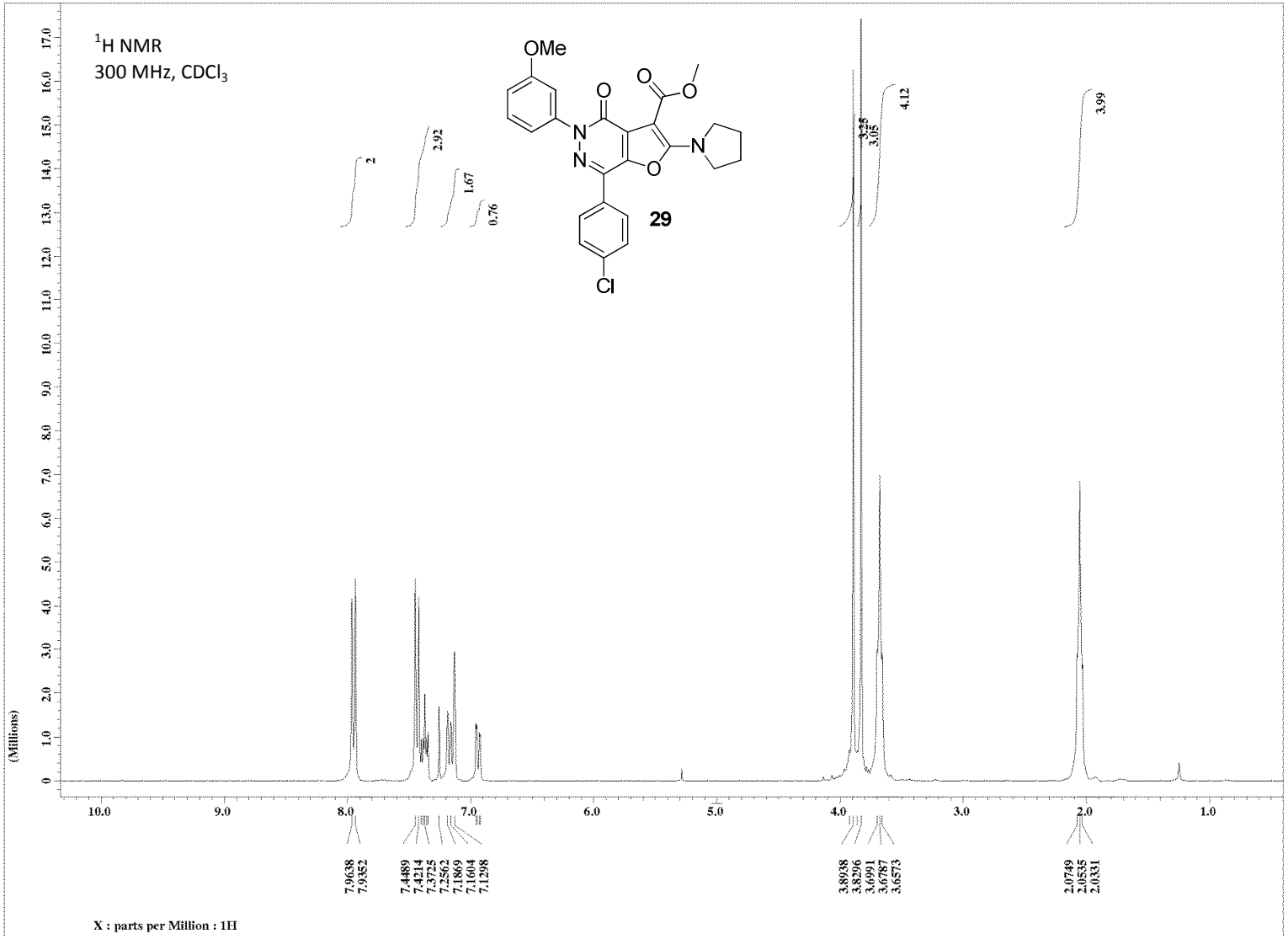
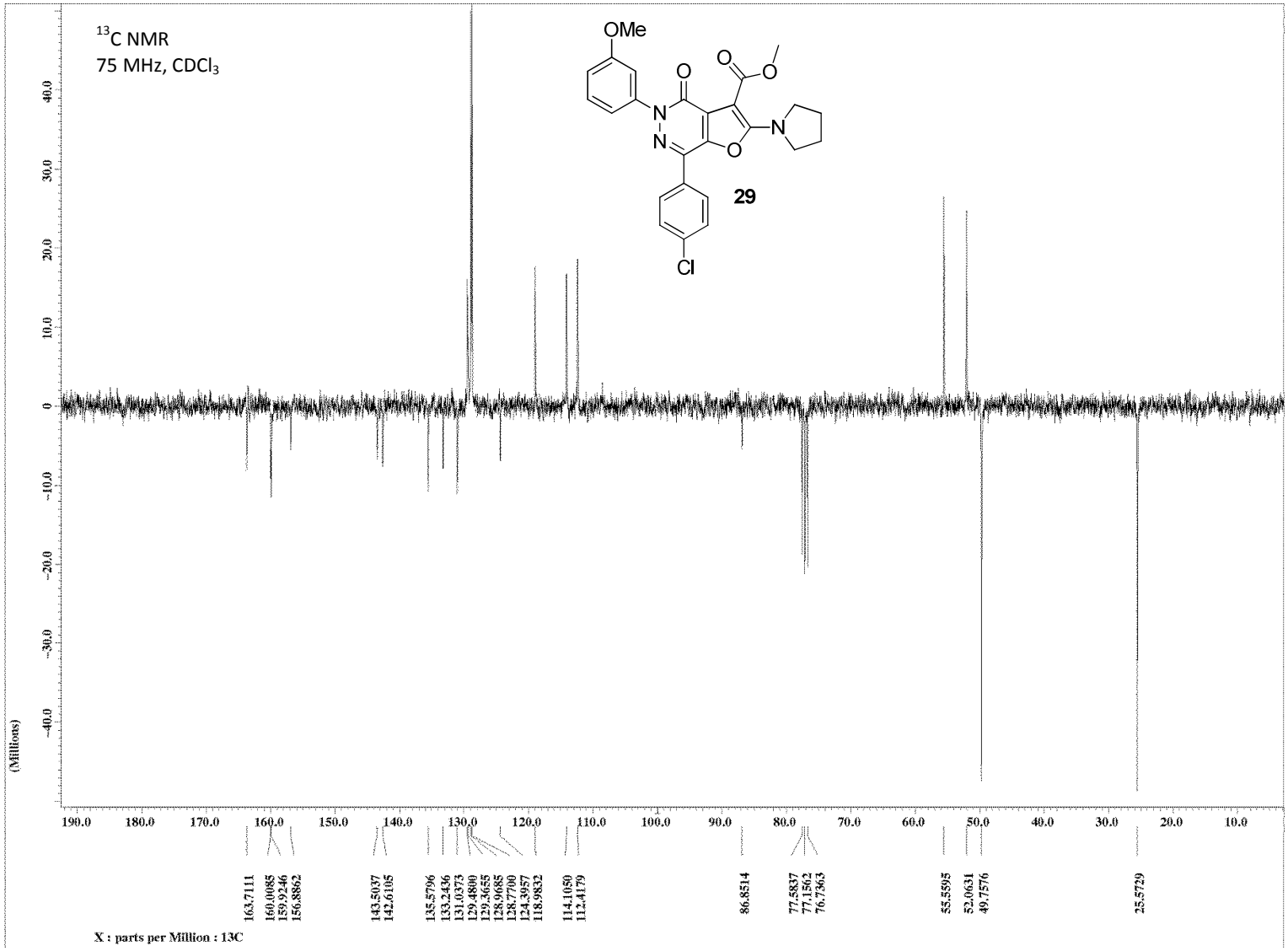
7.8815
7.8612
7.7950
7.7747
7.5822
7.5628
7.5343
7.5141
7.4962
7.4909
7.4704
7.4338
7.4132
7.3920
7.3729
4.6135
3.8278
3.4031
3.3915
3.3798
2.4122
2.4008
2.3895
2.3021
0.9073
2.0638
9.0479
2.0000
3.0987
4.0988
4.0695
3.0387
10
8
6
4
2
[ppm]

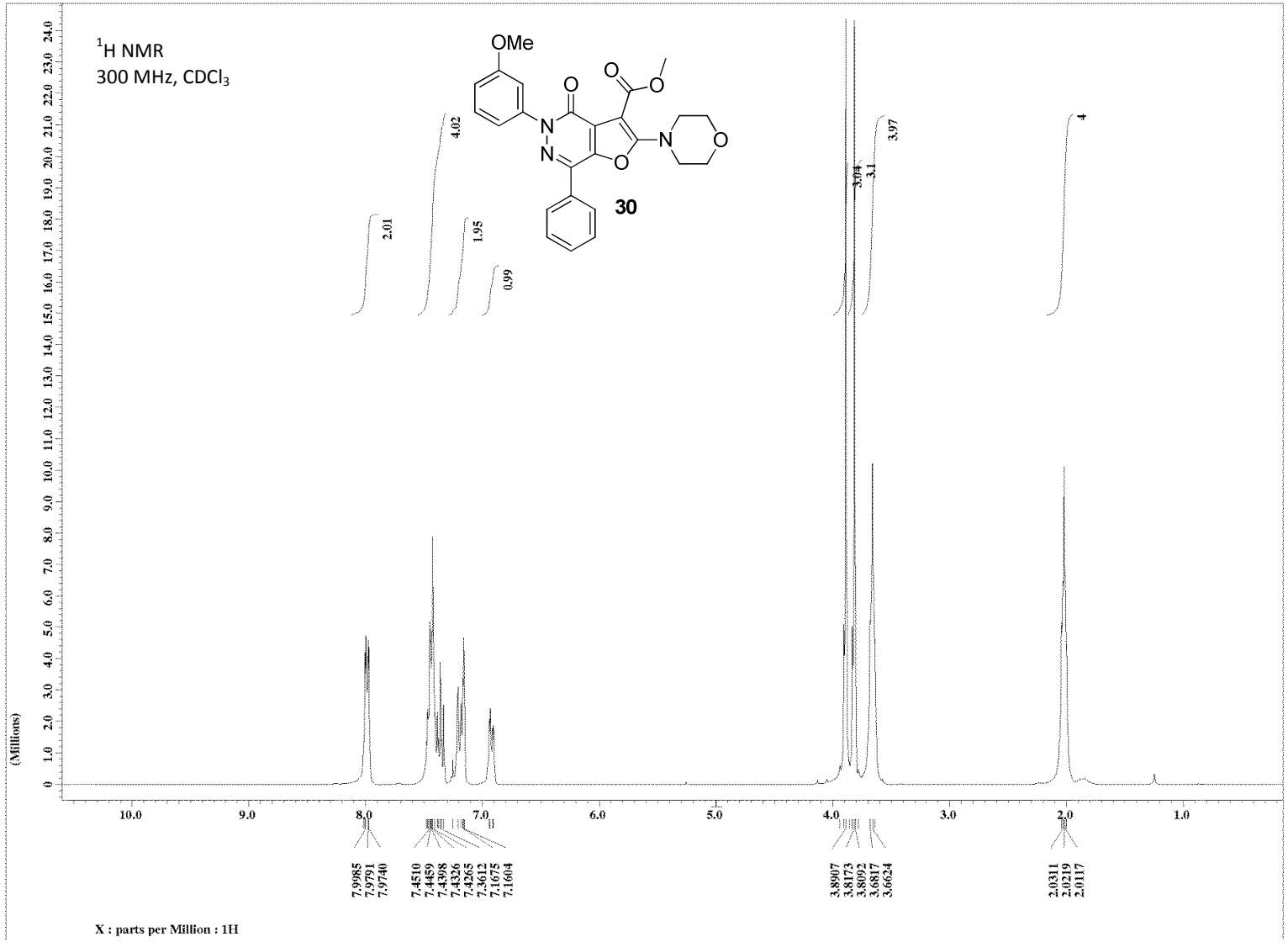

^{13}C NMR
100 MHz, $CDCl_3$
M 163.7782
M 161.5995
M 156.9035
M 144.2950
M 142.3003
M 135.9703
M 133.9682
M 132.6065
M 132.1281
M 128.8085
M 128.7869
M 127.9915
M 127.9252
M 127.5793
M 126.4826
M 126.2765
M 125.8349
M 125.2166
M 124.0389
M 123.2955
M 89.4961
M 54.3057
M 52.0166
M 47.9830
M 46.0766
M 35.0580
27
200
150
100
50
[ppm]

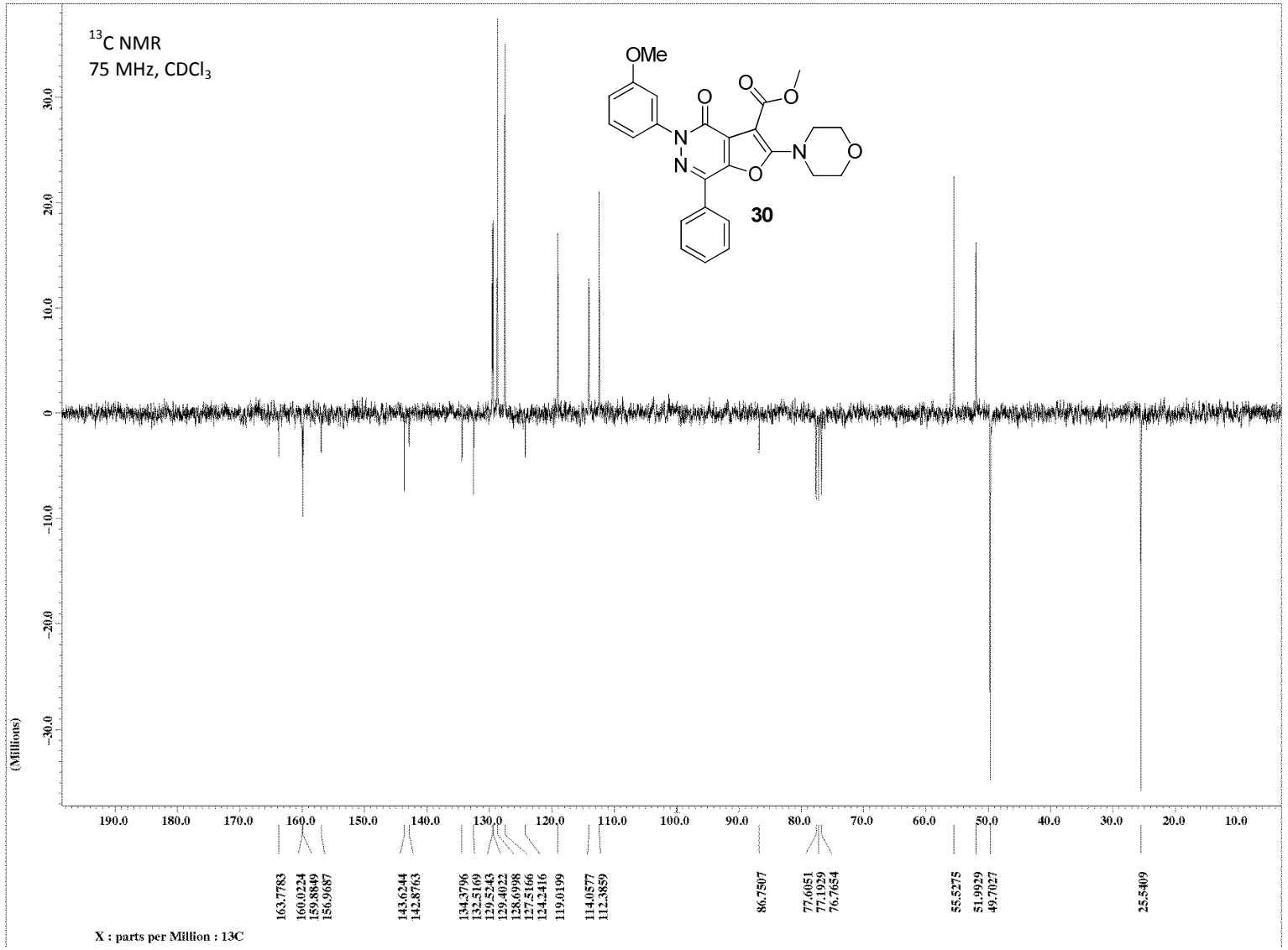

1H NMR
400 MHz, CDCl3
28
8.0480
8.0439
8.0278
8.0246
7.6328
7.6129
7.5170
7.5101
7.5019
7.4953
7.4830
7.4770
7.4640
7.4078
7.3893
7.3710
7.2587
4.3709
4.3652
3.9329
3.2742
2.3697
2.3639
2.3581
2.0000
8.4952
2.0971
3.0854
3.0832
0.8363
10
8
6
4
2
[ppm]

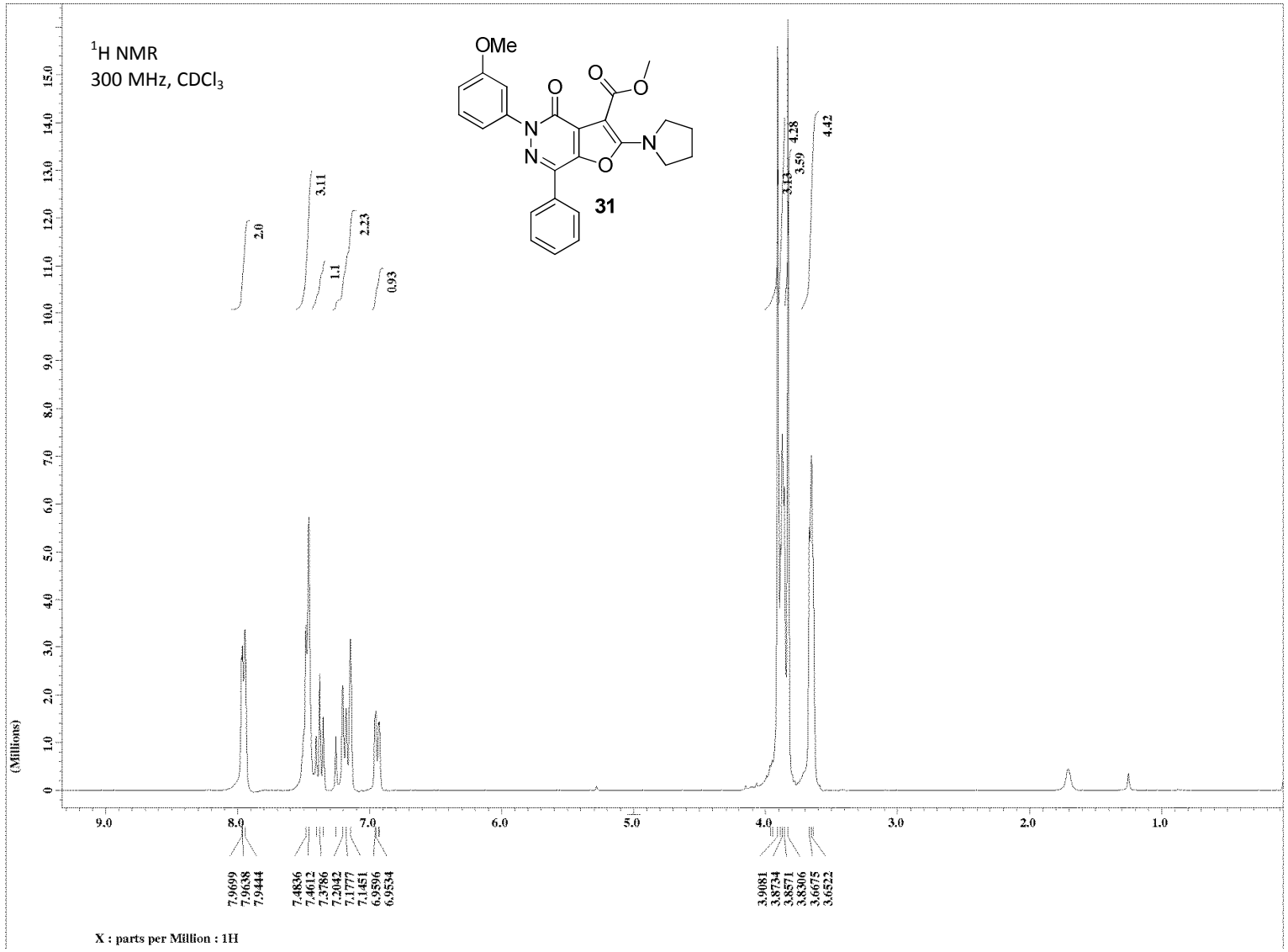

^{13}C NMR
100 MHz, $CDCl_3$
M 163.7482
M 161.0289
M 156.7920
M 143.5606
M 142.3367
M 134.6551
M 132.2251
M 129.6572
M 128.7537
M 128.7092
M 127.9482
M 127.5298
M 126.4216
M 123.7068
M 90.5549
M 73.5983
M 52.3604
M 42.3245
M 38.2078
28
150
100
50
[ppm]

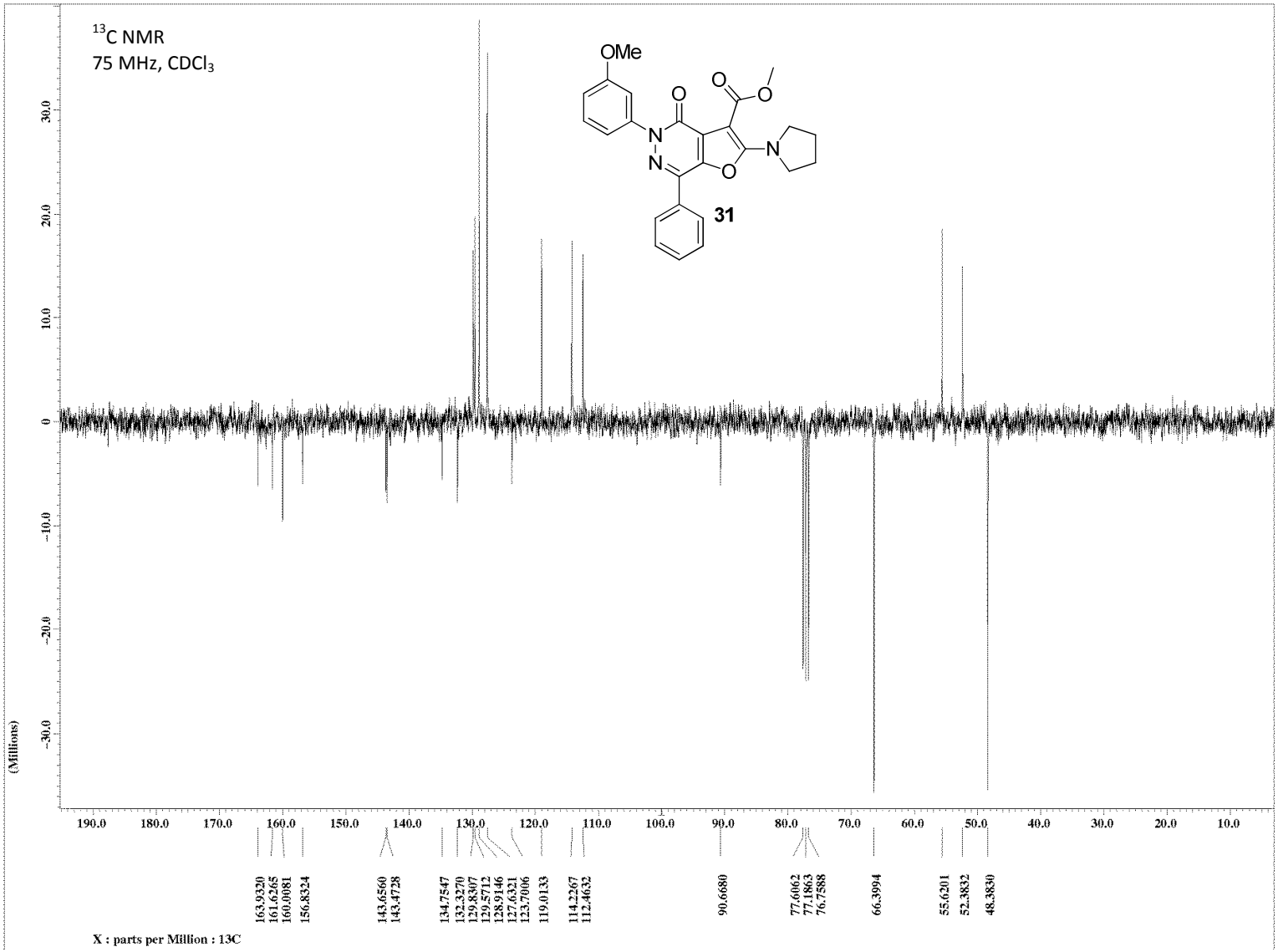

1H NMR
300 MHz, CDCl3
OMe
O
O
O
N
N
N
O
Cl
29
2
2.92
1.67
0.76
4.12
3.99
(Millions)
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
7.9638
7.9352
7.4489
7.4214
7.3725
7.2562
7.1869
7.1604
7.1298
3.8938
3.8296
3.6991
3.6787
3.6573
2.0749
2.0535
2.0331
X : parts per Million : 1H

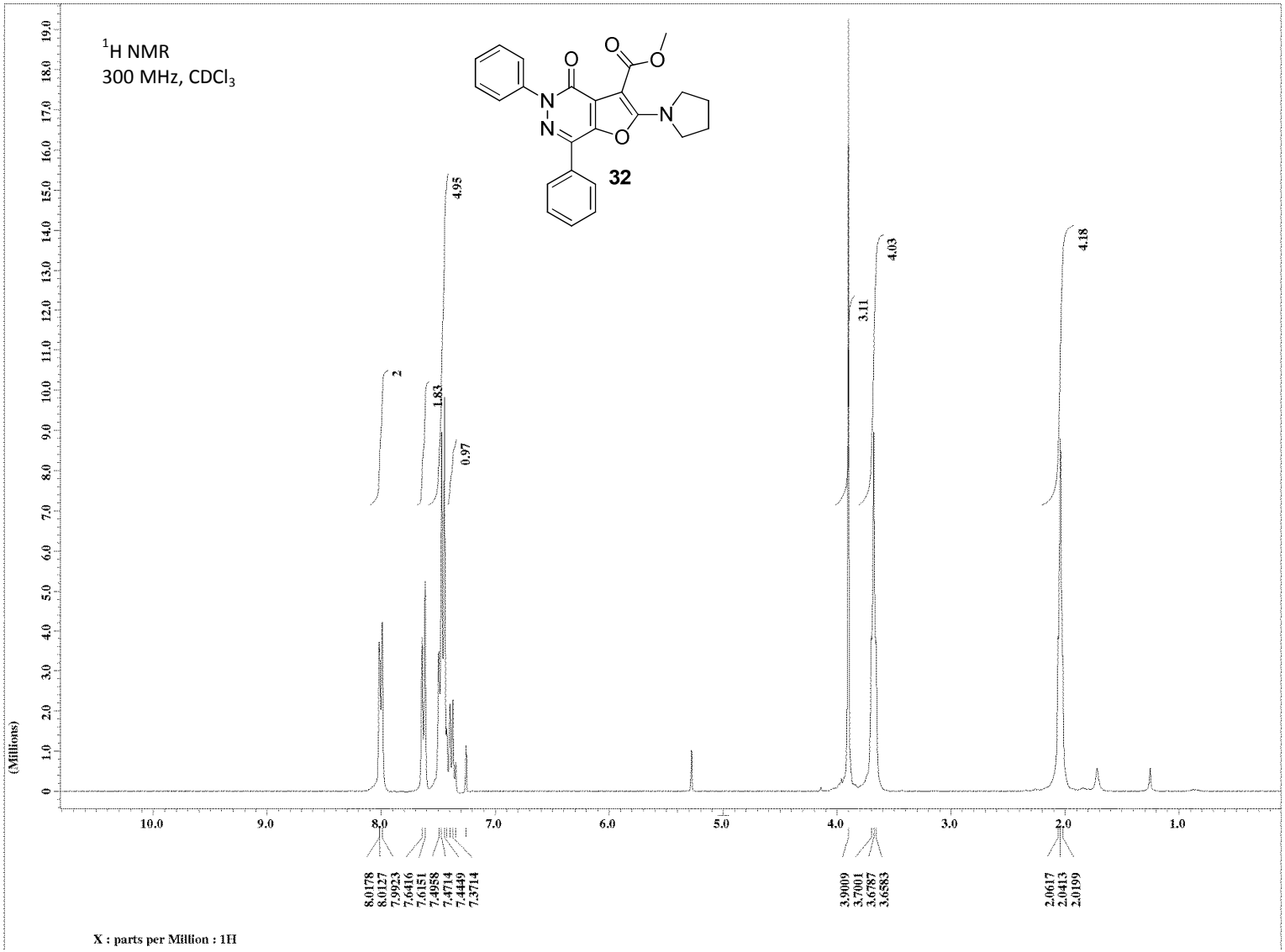

13C NMR
75 MHz, CDCl3
OMe
O
O
O
N
N
N
O
Cl
29
(Millions)
40.0
30.0
20.0
10.0
0
-10.0
-20.0
-30.0
-40.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
163.7111
160.0085
159.9246
156.8862
143.5037
142.6105
135.5796
133.2436
131.0373
129.4800
129.3655
128.9685
128.7700
124.3957
118.9832
114.1050
112.4179
86.8514
77.5837
77.1502
76.7363
55.5595
52.0631
49.7576
25.5729
X : parts per Million : 13C

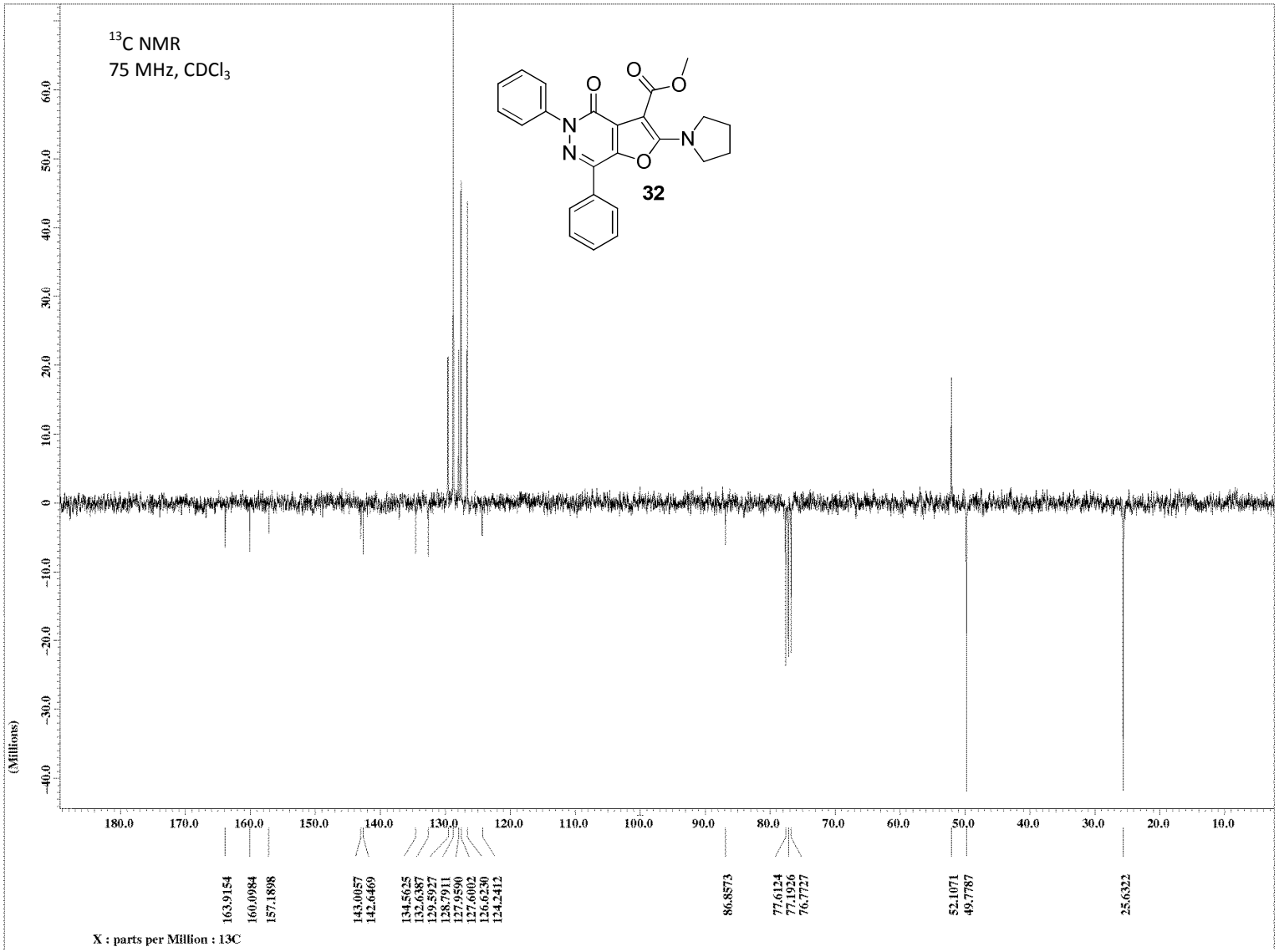

1H NMR
300 MHz, CDCl3
OMe
O
O
O
N
N
O
N
O
30
2.01
4.02
1.95
0.99
3.04
3.1
3.97
4
(Millions)
0 1.0 2.0 3.0 4.0 5.0 6.0 7.0 8.0 9.0 10.0 11.0 12.0 13.0 14.0 15.0 16.0 17.0 18.0 19.0 20.0 21.0 22.0 23.0 24.0
10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0
7.9985
7.9791
7.9740
7.4510
7.4459
7.4398
7.4326
7.4265
7.3612
7.1675
7.1604
3.8907
3.8173
3.8092
3.6817
3.6624
2.0311
2.0219
2.0117
X : parts per Million : 1H

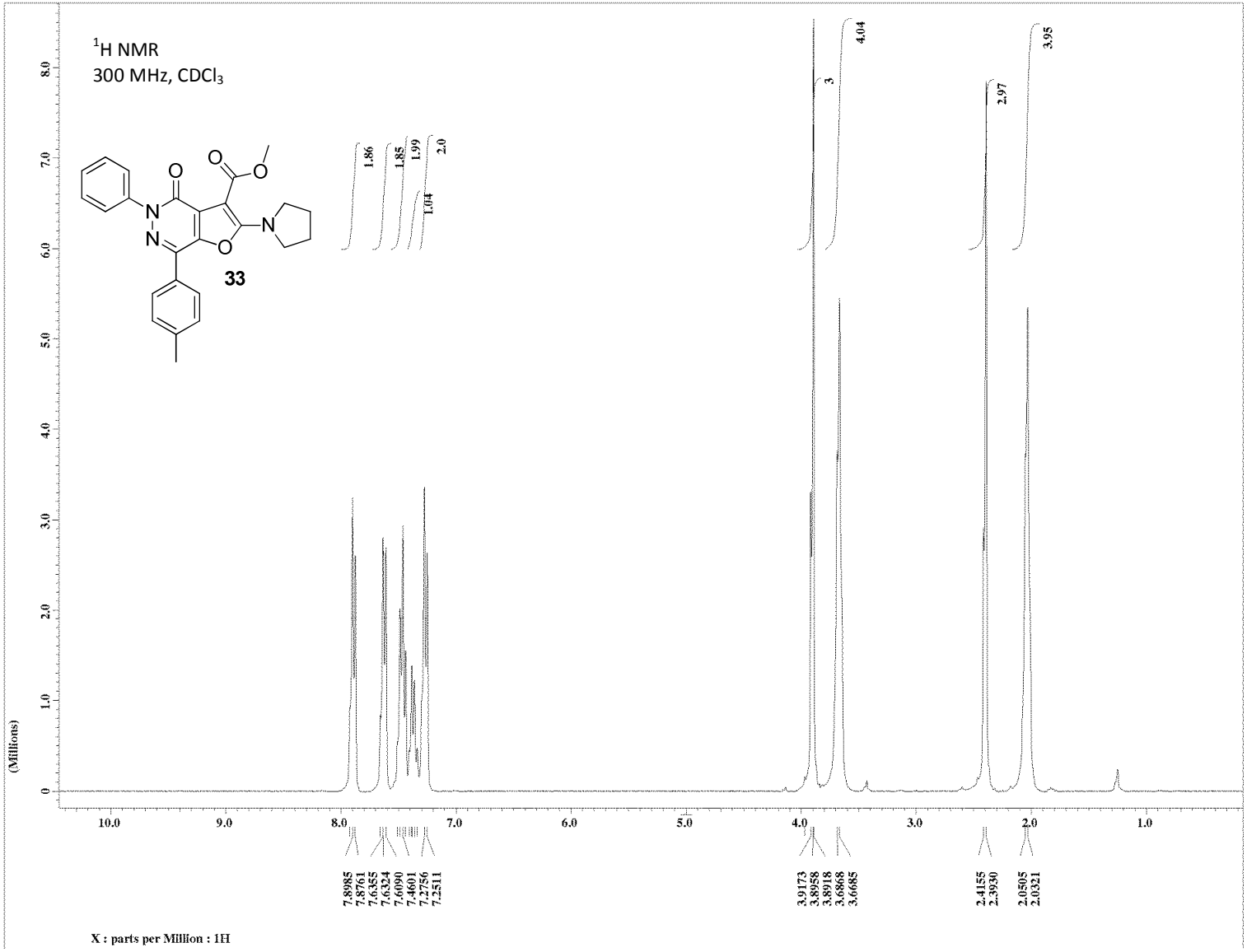

^{13}C NMR
75 MHz, $CDCl_3$
OMe
O
O
O
N
N
O
N
O
30
(Millions)
30.0
20.0
10.0
0
-10.0
-20.0
-30.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
163.7783
160.0224
159.8849
156.9687
143.6244
142.8763
134.3796
132.5169
129.5243
129.4022
128.6998
127.5166
124.2416
119.0199
114.0577
112.3859
86.7507
77.6051
77.1929
76.7654
55.5275
51.9929
49.7027
25.5409
X : parts per Million : 13C

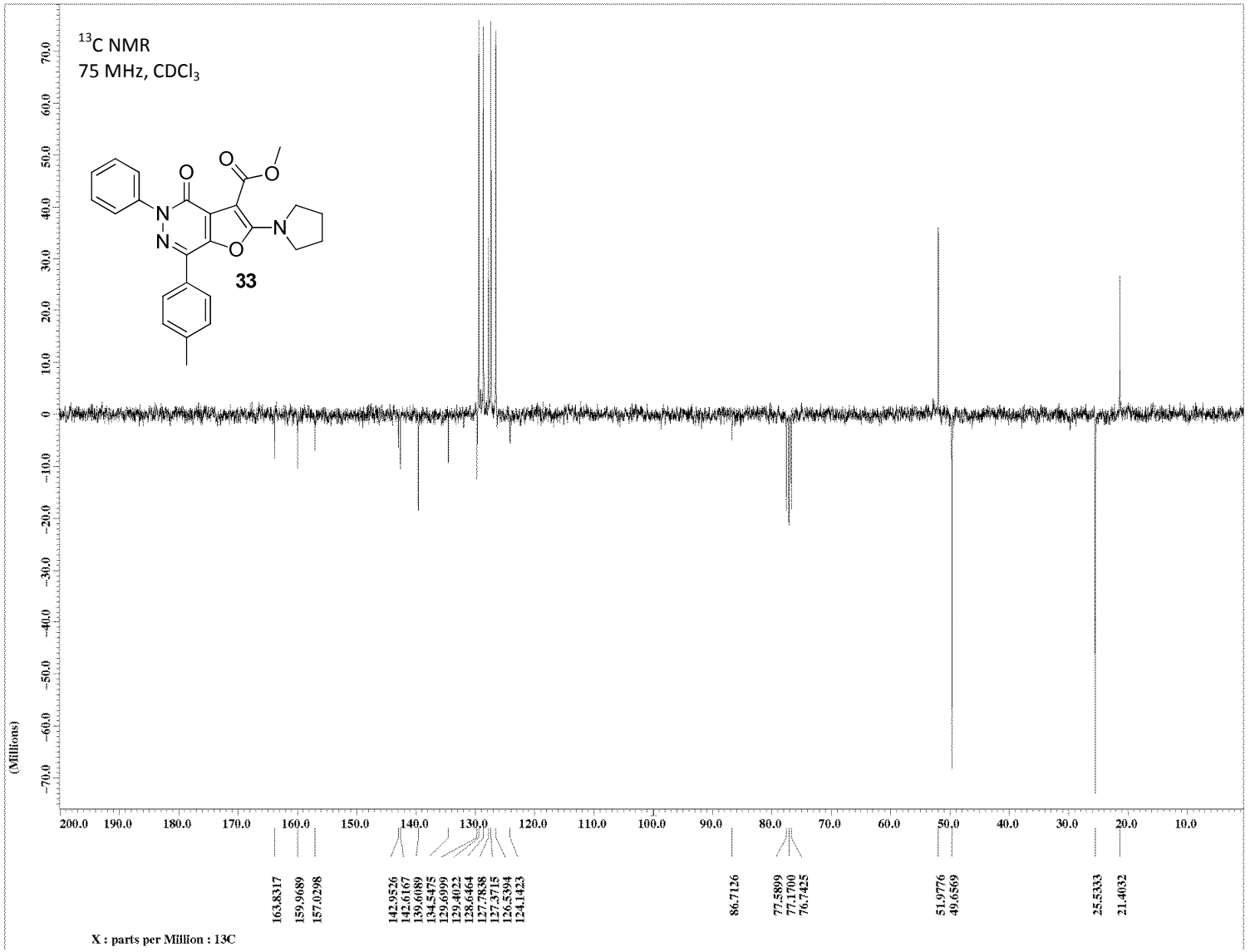

^{1}H NMR
300 MHz, $CDCl_3$
OMe
O
O
O
N
N
O
N
31
2.0
3.11
1.1
2.23
0.93
3.13
3.59
4.28
4.42
(Millions)
15.0
14.0
13.0
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
7.9699
7.9638
7.9444
7.4836
7.4612
7.3786
7.2042
7.1777
7.1451
6.9596
6.9534
3.9081
3.8734
3.8571
3.8306
3.6675
3.6522
X : parts per Million : 1H

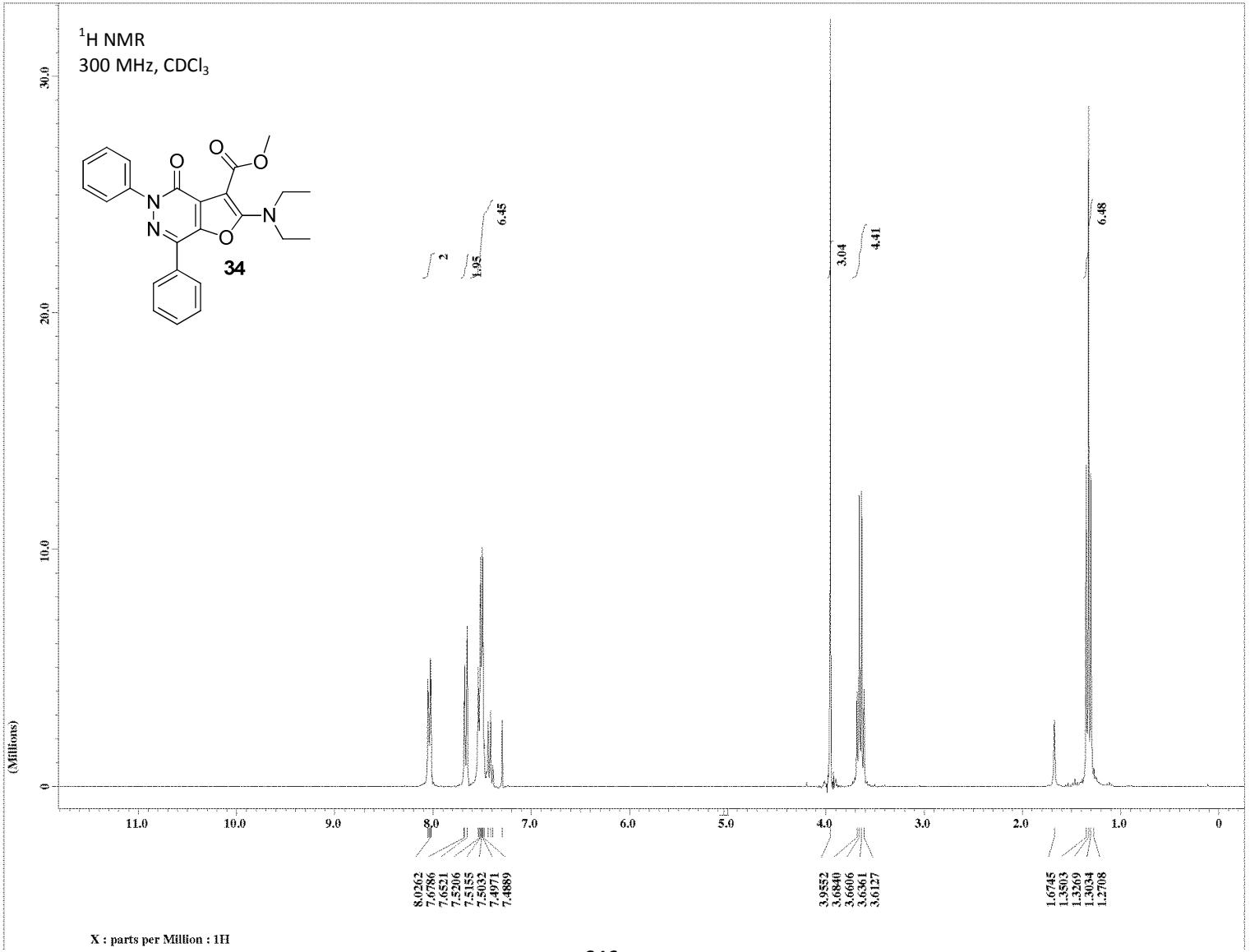

13C NMR
75 MHz, CDCl3
OMe
O
O
O
N
N
N
O
31
(Millions)
30.0
20.0
10.0
0
-10.0
-20.0
-30.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
163.9320
161.6265
160.0081
156.8324
143.6560
143.4728
134.7547
132.3270
129.8307
129.5712
128.9146
127.6321
123.7006
119.0133
114.2267
112.4632
90.6680
77.6062
77.1863
76.7588
66.3994
55.6201
52.3832
48.3830
X : parts per Million : 13C

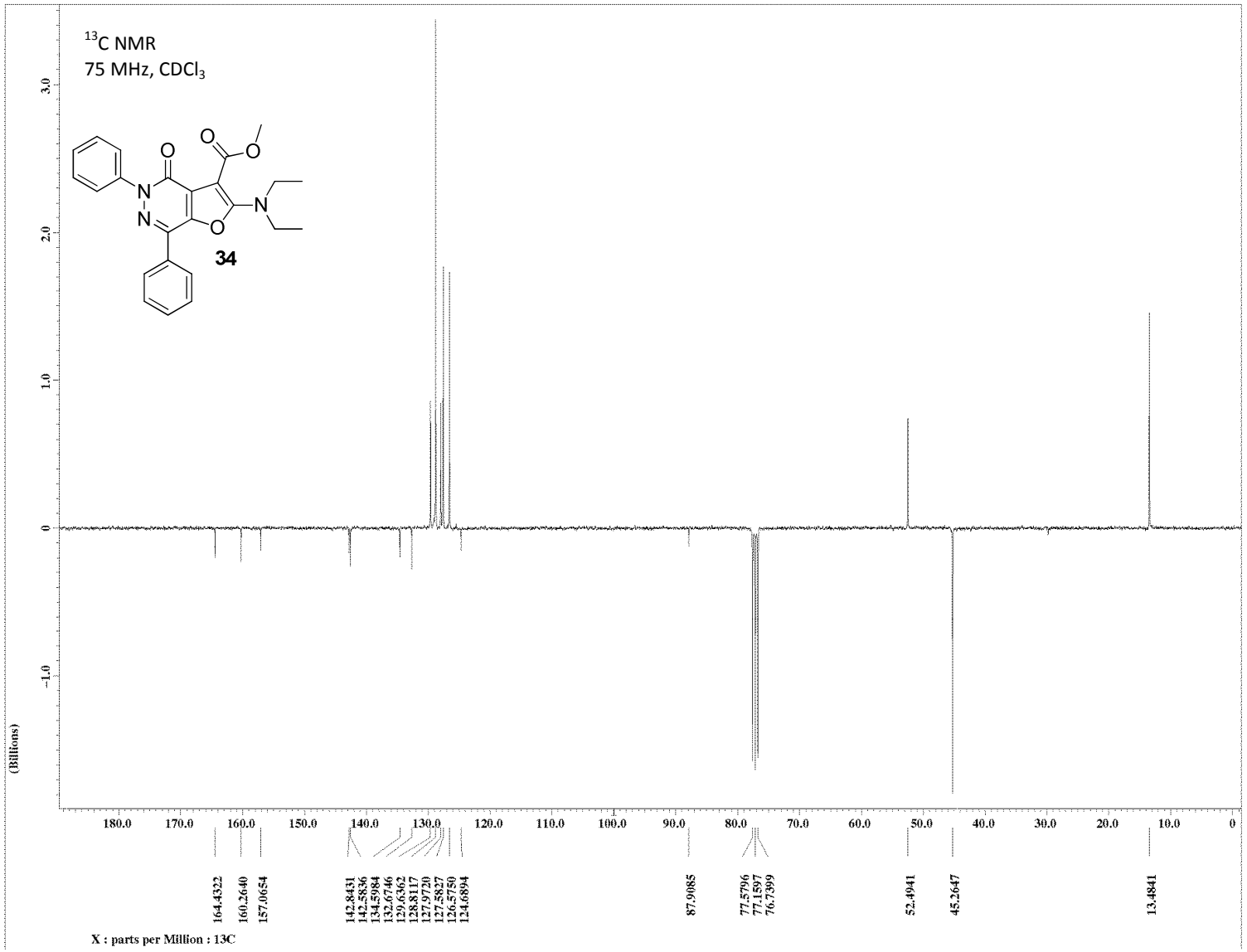

^{1}H NMR
300 MHz, $CDCl_3$
32
4.95
4.18
4.03
3.11
2
1.83
0.97
(Millions)
8.0178
8.0127
7.9923
7.6416
7.6151
7.4958
7.4714
7.4449
7.3714
3.9009
3.7001
3.6787
3.6583
2.0617
2.0413
2.0199
X : parts per Million : 1H

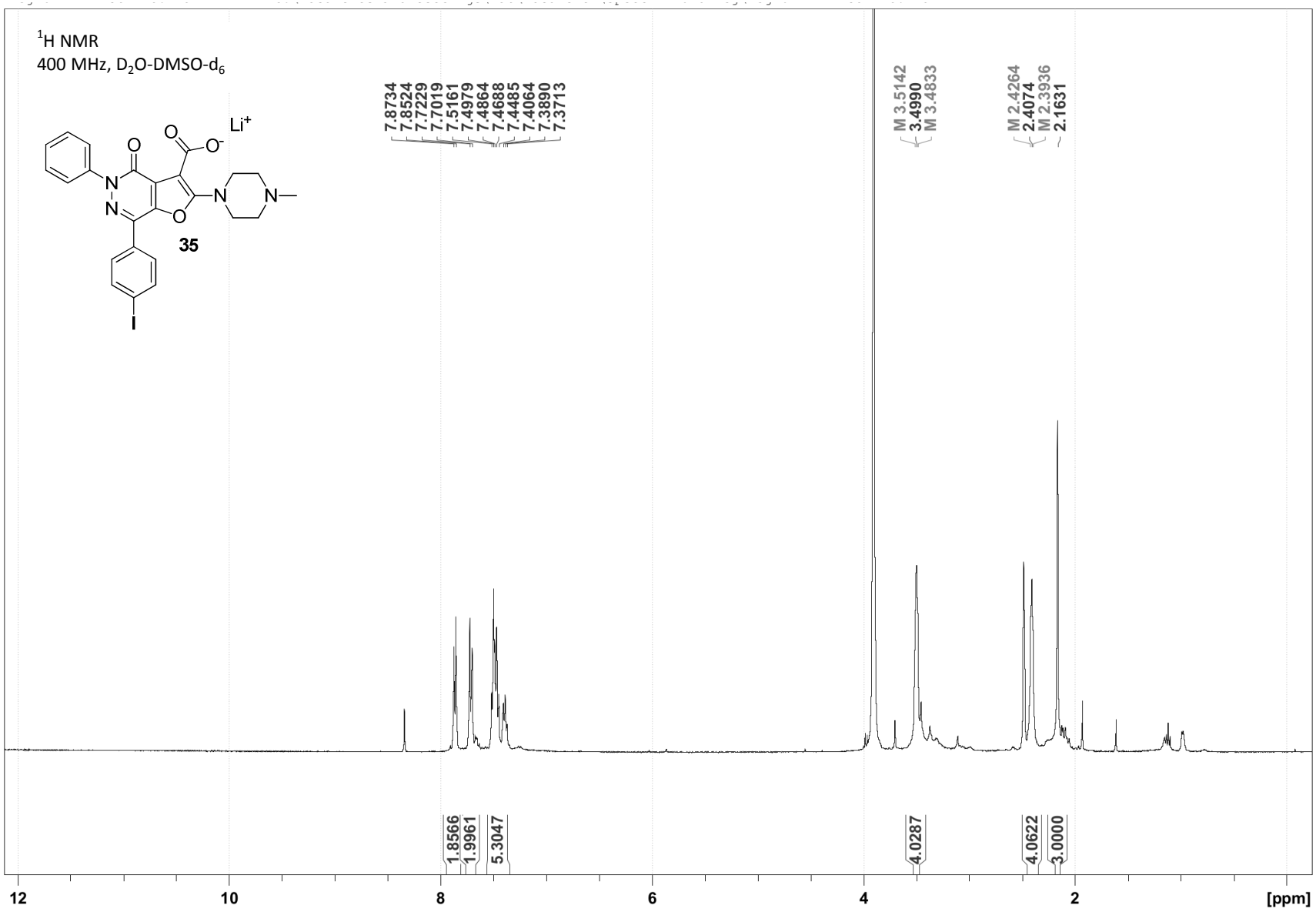

^{13}C NMR
75 MHz, $CDCl_3$
32
(Millions)
60.0
50.0
40.0
30.0
20.0
10.0
0
-10.0
-20.0
-30.0
-40.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
163.9154
160.0984
157.1898
143.0057
142.6469
134.5625
132.6387
129.8927
128.7911
127.9590
127.6002
126.6230
124.2412
86.8573
77.6124
77.1926
76.7727
52.1071
49.7787
25.6322
X : parts per Million : 13C

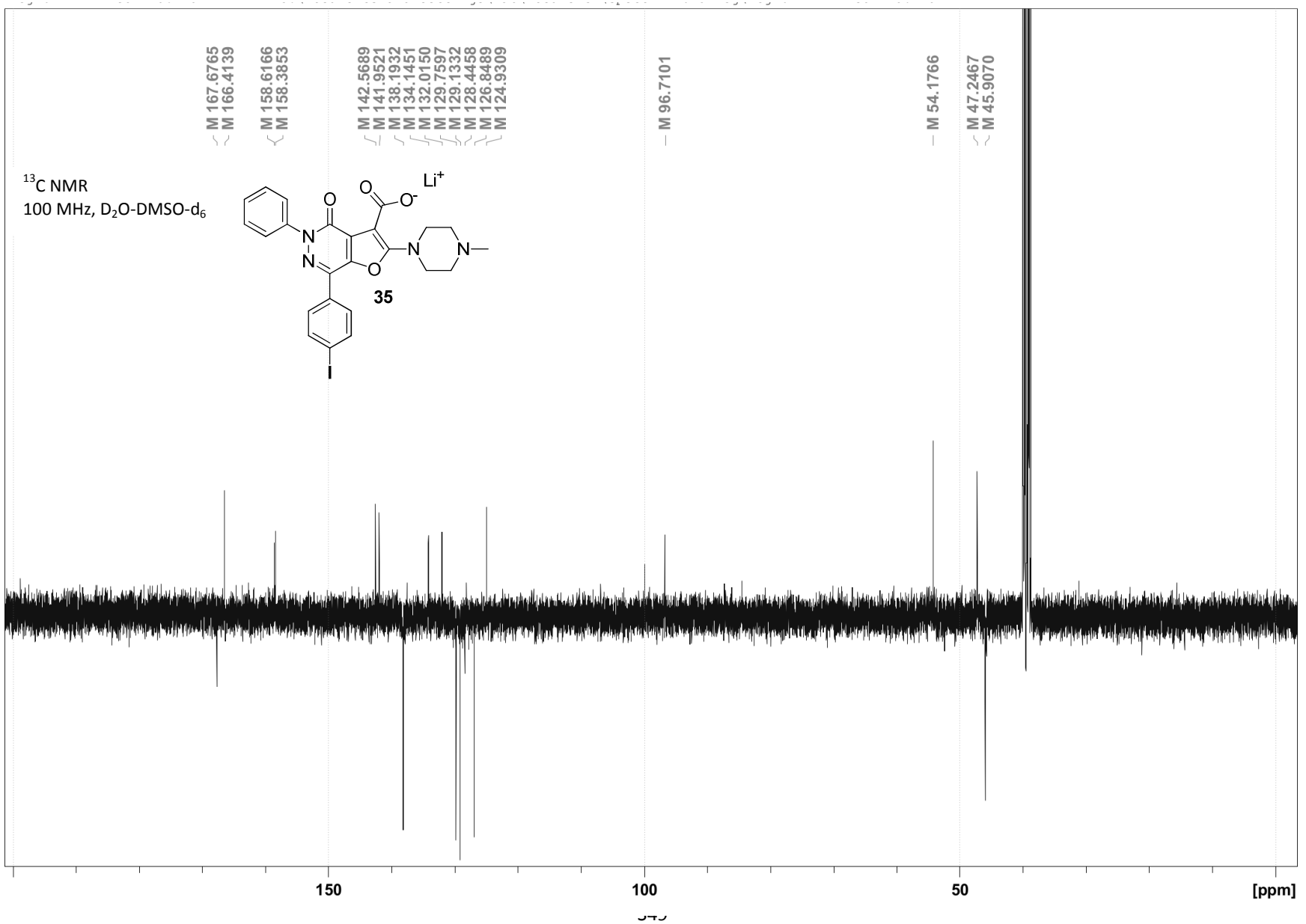

^{1}H NMR
300 MHz, $CDCl_3$
33
1.86
1.85
1.99
2.0
1.04
3
4.04
2.97
3.95
(Millions)
7.8985
7.8761
7.6355
7.6324
7.6090
7.4601
7.2756
7.2511
3.9173
3.8958
3.8918
3.6868
3.6685
2.4155
2.3930
2.0505
2.0321
X : parts per Million : 1H


^{13}C NMR
75 MHz, $CDCl_3$
33
(Millions)
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
-10.0
-20.0
-30.0
-40.0
-50.0
-60.0
-70.0
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
163.8317
159.9689
157.0298
142.9526
142.6167
139.6089
134.5475
129.6999
129.4022
128.6464
127.7838
127.3715
126.5394
124.1423
86.7126
77.5899
77.1700
76.7425
51.9776
49.6569
25.5333
21.4032
X : parts per Million : 13C


1H NMR
300 MHz, CDCl3
34
(Millions)
30.0
20.0
10.0
0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
2
1.95
6.45
3.04
4.41
6.48
8.0262
7.6786
7.6521
7.5206
7.5155
7.5032
7.4971
7.4889
3.9552
3.6840
3.6606
3.6361
3.6127
1.6745
1.3503
1.3269
1.3034
1.2708
X : parts per Million : 1H


13C NMR
75 MHz, CDCl3
34
(Billions)
3.0
2.0
1.0
0
-1.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
164.4322
160.2640
157.0654
142.8431
142.5836
134.5984
132.6746
129.6362
128.8117
127.9720
127.5827
126.5750
124.6894
87.9085
77.5796
77.1597
76.7399
52.4941
45.2647
13.4841
X : parts per Million : 13C


1H NMR
400 MHz, D2O-DMSO-d6
35
7.8734
7.8524
7.7229
7.7019
7.5161
7.4979
7.4864
7.4688
7.4485
7.4064
7.3890
7.3713
M 3.5142
3.4990
M 3.4833
M 2.4264
2.4074
M 2.3936
2.1631
1.8566
1.9961
5.3047
4.0287
4.0622
3.0000
12
10
8
6
4
2
[ppm]


M 167.6765
M 166.4139
M 158.6166
M 158.3853
M 142.5689
M 141.9521
M 138.1932
M 134.1451
M 132.0150
M 129.7597
M 129.1332
M 128.4458
M 126.8489
M 124.9309
M 96.7101
M 54.1766
M 47.2467
M 45.9070
13C NMR
100 MHz, D2O-DMSO-d6
Li+
O
O-
N
N
O
N
N
35
I
150
100
50
[ppm]