Copper nitrate-mediated synthesis of 3-aryl isoxazolines and isoxazoles from olefinic azlactones

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Supporting Information

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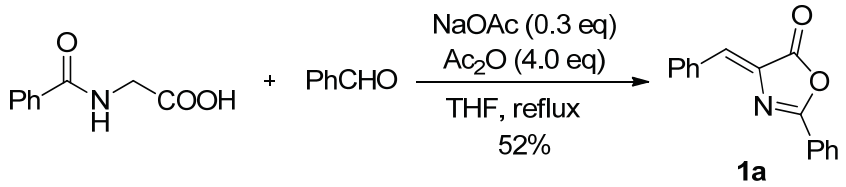
General Information

All reagents were obtained from commercial sources without further purification, and commercially available solvents were purified before use. All new compounds were fully characterized. All melting points were taken on a WRS-1A or a WRS-1B Digital Melting Point Apparatus without correction. Infrared spectra were obtained using an AVATAR 370 FT-IR spectrometer. ^{1}H, ^{13}C and ^{19}F NMR spectra were recorded with a Bruker AV-500 spectrometer operating at 500 MHz, 125 MHz and 470 MHz, respectively, with chemical shift values being reported in ppm relative to chloroform (δ = 7.26 ppm), dimethyl sulfoxide (δ = 2.50 ppm) or TMS (δ = 0.00 ppm) for ^{1}H NMR; with chloroform (δ = 77.16 ppm), dimethyl sulfoxide (δ = 39.52 ppm) for ^{13}C NMR; with C_6F_6 (δ = -164.9 ppm) for ^{19}F NMR. Mass spectra (MS) and high resolution mass spectra (HRMS) were recorded with an Agilent 5975C or Thermo Fisher Scientific LTQ FTICR-MS using an Electron impact (EI) or Electrospray ionization (ESI) techniques. The crystal structure was recorded on SMART APEXII X-ray diffraction spectrometer. Silica gel plate GF254 were used for thin layer chromatography (TLC) and silica gel H or 300–400 mesh were used for flash column chromatography. Yields refer to chromatographically and spectroscopically pure compounds, unless otherwise indicated. *N*-Alkylmaleimides **2a**–**2g** and **2j**–**2n** are all purchased from commercial sources.

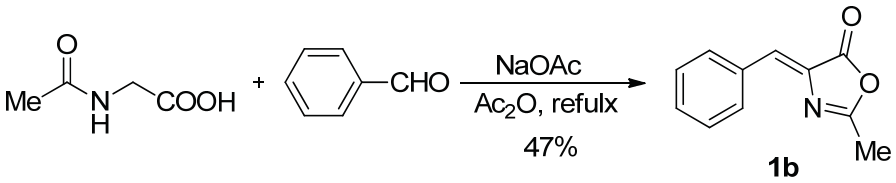
2. Synthesis and Characterization of Substrates

2.1 Synthesis of Olefinic Azlactones



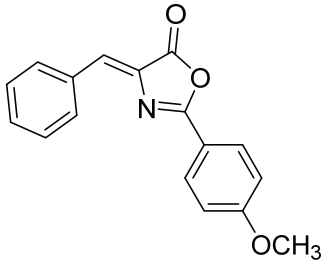
1a

(*Z*)-4-Benzylidene-2-phenyloxazol-5(4*H*)-one (1a):[1] Hippuric acid (1.80 g, 10 mmol), benzaldehyde (1.27 g, 12.0 mmol), NaOAc (0.25 g, 3.0 mmol) and Ac_2O (4.0 mL, 40.0 mmol) in THF (30 mL) was reflux for 3 h. Upon completion, the reaction mixture was cooled down to room temperature. A saturated aqueous solution of Na_2CO_3 was added. The mixture was extracted with CH_2Cl_2, dried with Na_2SO_4, filtered, and the solvent was removed under vacuum. The given residue was purified by recrystallization from EtOH to give **1a** as a yellow solid (1.30 g, 52%). M.p. 169-171 ^oC; IR (KBr, cm^{-1}): 3056, 1794, 1652, 1589, 1549, 1325, 1293, 1160, 982, 861, 764, 689; ^1H NMR ($CDCl_3$, 500 MHz): δ 8.20-8.16 (m, 4H), 7.61 (m, 1H), 7.54-7.43 (m, 5H), 7.23 (s, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 167.6, 163.5, 133.5, 133.4, 133.3, 132.5, 131.8, 131.2, 129.0, 128.9, 128.4, 125.6.



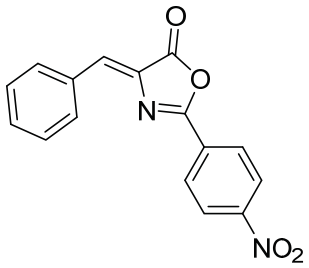
1b

(*Z*)-4-Benzylidene-2-methyloxazol-5(4*H*)-one (1b):[1] *N*-Acetylglycine (3.42 g, 29.2 mmol), benzaldehyde (2.4 mL, 24.0 mmol), NaOAc (9.99 g, 122.0 mmol) in Ac_2O (70 mL) was reflux for 7 h. Upon completion, the reaction mixture was cooled down to room temperature. After being maintained 4 ^oC overnight. The solid was filtered and washed with water and a little EtOH. The desired product **1b** was obtained as a light yellow solid (2.10 g, 47%). M.p. 152-153 ^oC; IR (KBr, cm^{-1}): 3058, 1774, 1653, 1595, 1261, 1165, 898, 766, 688; ^1H NMR ($CDCl_3$, 500 MHz): δ 8.08-8.06 (m, 2H), 7.45-7.43 (m, 3H), 7.14 (s, 1H), 2.40 (s, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 167.8, 166.1, 133.1, 132.6, 132.2, 131.5, 131.1, 128.9, 15.7.

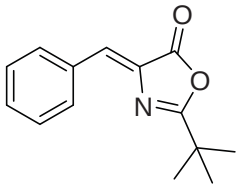


(*Z*)-4-Benzylidene-2-(4-methoxyphenyl)oxazol-5(4*H*)-one (1c):[2] Following the general procedure as for **1b**, (4-methoxybenzoyl)glycine (836.0 mg, 4.0 mmol), benzaldehyde (808.0 μL, 8.0 mmol), NaOAc (328 mg, 4 mmol) in Ac_2O (2.5 mL) was

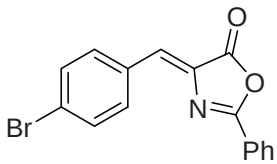
reflux for 1 h. The solid was filtered and washed with water and a little EtOH. The desired product **1c** was obtained as a light yellow solid (248.0 mg, 28%). M.p. 212-214 ^{o}C; IR (KBr, cm^{-1}): 3000, 2980, 1777, 1648, 1601, 1550, 1503, 1304, 1260, 1165, 981, 876, 840, 764, 685; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 8.19 (d, J = 7.2 Hz, 2H), 8.14 (d, J = 8.9 Hz, 2H), 7.49-7.42 (m, 3H), 7.19 (s, 1H), 7.03 (d, J = 8.9 Hz, 2H), 3.91 (s, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 168.0, 163.9, 163.4, 133.8, 133.6, 132.3, 130.9, 130.5, 130.2, 128.9, 117.9, 114.5, 55.6. EI-MS m/z: 279 [M^{+}].



(*Z*)-4-Benzylidene-2-(4-nitrophenyl)oxazol-5(4*H*)-one (1d):[3] Following the general procedure as for **1b**, (4-nitrobenzoyl)glycine (336.0 mg, 1.5 mmol), benzaldehyde (303.0 μL, 3.0 mmol), NaOAc (615.0 mg, 7.5 mmol) in Ac_2O (4 mL) was reflux for 1 h. The solid was filtered to give the desired product **1d** as a light yellow solid (441 mg, 49%). M.p. 230-232 ^{o}C; IR (KBr, cm^{-1}): 3063, 1788, 1651, 1519, 1344, 1318, 1161, 892, 856, 767, 696; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 8.40-8.35 (m, 4H), 8.23-8.21 (m, 2H), 7.52-7.51 (m, 3H), 7.39 (s, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 166.8, 161.5, 150.4, 134.6, 133.1, 132.9, 132.6, 132.0, 131.2, 129.2, 129.1, 124.2. EI-MS m/z: 294 [M^{+}].

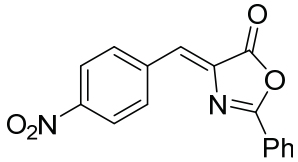


(*Z*)-4-Benzylidene-2-(*tert*-butyl)oxazol-5(4*H*)-one (1e):[4] Following the general procedure as for **1b**, pivaloylglycine (286.0 mg, 1.8 mmol), benzaldehyde (152.0 μL, 1.5 mmol), NaOAc (615.0 mg, 7.5 mmol) in Ac_2O (3 mL) was reflux for 1 h. The solid was filtered and washed with water and a little EtOH. The desired product **1e** was obtained as a light yellow solid (75.4 mg, 22%). M.p. 88-90 ^{o}C; IR (KBr, cm^{-1}): 3055, 2970, 2871, 1796, 1654, 1590, 1294, 1146, 1017, 859, 765, 692; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 8.12 (dd, J = 7.8, 2.5 Hz, 2H), 7.47-7.41 (m, 3H), 7.15 (s, 1H), 1.39 (s, 9H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 174.8, 168.3, 133.3, 133.0, 132.3, 131.5, 131.0, 128.8, 34.4, 27.0; EI-MS m/z: 229 [M^{+}].

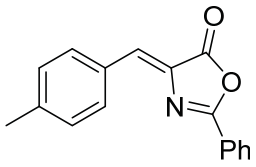


(*Z*)-4-(4-Bromobenzylidene)-2-phenyloxazol-5(4*H*)-one (1f):[1] Following the general procedure as for **1a**, hippuric acid (358.5 mg, 2.0 mmol), 4-bromo-

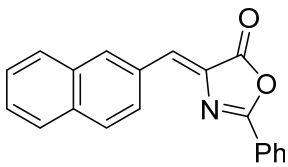
benzaldehyde (444.0 mg, 2.4 mmol), NaOAc (49.2 mg, 0.6 mmol), Ac_2O (0.8 mL, 8.0 mmol) in THF (8 mL) was reflux for 3 h. The given residue was purified by recrystallization from ethyl acetate/DCM to give **1f** as a light yellow solid (435.0 mg, 66%). M.p. 207-208 ^oC; IR (KBr, cm^{-1}): 3054, 1794, 1650, 1554, 1482, 1158, 1067, 983, 893, 824, 694; ^1H NMR ($CDCl_3$, 500 MHz): δ 8.18 (d, J = 7.4 Hz, 2H), 8.07 (d, J = 8.5 Hz, 2H), 7.65-7.60 (m, 3H), 7.54 (t, J = 7.8 Hz, 2H), 7.16 (s, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 167.4, 164.0, 133.8, 133.7, 133.6, 132.4, 132.2, 130.1, 129.0, 128.5, 125.9, 125.4.



(*Z*)-4-(4-Nitrobenzylidene)-2-phenyloxazol-5(4*H*)-one (1g):[1] Following the general procedure as for **1a**, hippuric acid (358.5 mg, 2.0 mmol), 4-nitrobenzaldehyde (362.6 mg, 2.4 mmol), NaOAc (49.2mg, 0.6 mmol), Ac_2O (0.8 mL, 8 mmol) in THF (8 mL) was reflux for 3 h. The solid was filtered and washed with water and a little ethyl acetate. The desired produc **1g** was obtained as a light yellow solid (335.7 mg, 57%). M.p. 238-240 ^oC; IR (KBr, cm^{-1}): 3100, 2900, 1796, 1651, 1552, 1517, 1336, 1295, 1160, 976, 858, 688; ^1H NMR (DMSO-d_6, 500 MHz): δ 8.55 (d, J = 8.8 Hz, 2H), 8.35 (d, J = 8.9 Hz, 2H), 8.19-8.18 (m, 2H), 7.77 (t, J = 7.5 Hz, 1H), 7.67 (t, J = 8.0 Hz, 2H), 7.48 (s, 1H); ^{13}C NMR (DMSO-d_6, 125 MHz): δ 166.9, 165.3, 148.3, 140.1, 136.7, 134.8, 133.4, 129.9, 128.9, 127.4, 125.3, 124.4.

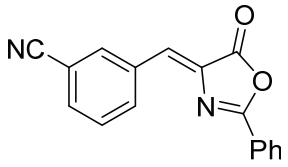


(*Z*)-4-(4-Methylbenzylidene)-2-phenyloxazol-5(4*H*)-one (1h):[1] Following the general procedure as for **1a**, hippuric acid (537.6 mg, 3.0 mmol), 4-methyl-benzaldehyde (425.0 μL, 3.6 mmol), NaOAc (73.8 mg, 0.9 mmol), Ac_2O (1.2 mL, 12 mmol) in THF (10 mL) was reflux for 3 h. The given residue was purified by recrystallization from EtOH/DCM to give **1h** as a yellow solid (412 mg, 52%). M.p. 144-145 ^oC; IR (KBr, cm^{-1}): 1795, 1649, 1551, 1324, 1157, 859, 816, 692; ^1H NMR ($CDCl_3$, 500 MHz): δ 8.17 (d, J = 7.4 Hz, 2H), 8.10 (d, J = 8.1 Hz, 2H), 7.60 (t, J = 7.4 Hz, 1H), 7.52 (t, J = 7.8 Hz, 2H), 7.29 (d, J = 8.0 Hz, 2H), 7.23 (s, 1H), 2.43 (s, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 167.8, 163.0, 142.1, 133.2, 132.6, 132.4, 132.1, 130.9, 129.7, 128.9, 128.3, 125.7, 21.8.

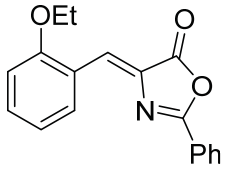


(*Z*)-4-(Naphthalen-2-ylmethylene)-2-phenyloxazol-5(4*H*)-one (1i):[5] Following the

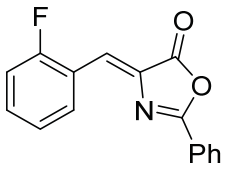
general procedure as for **1a**, hippuric acid (537.6 mg, 3.0 mmol), 2-naphthaldehyde (562.2 mg, 3.2 mmol), NaOAc (73.8 mg, 0.9 mmol), Ac_2O (1.2 mL, 12.0 mmol) in THF (10 mL) was reflux for 3 h. The given residue was purified by recrystallization from EtOH/ethyl acetate to give **1i** as a yellow solid (408.5 mg, 45%). M.p. 150-152 ^oC; IR (KBr, cm^{-1}): 3054, 1793, 1650, 1557, 1330, 1161, 912, 879, 692; ^1H NMR ($CDCl_3$, 500 MHz): δ 8.52 (d, J = 8.5 Hz, 1H), 8.46 (s, 1H), 8.21 (d, J = 7.3 Hz, 2H), 7.94-7.90 (m, 2H), 7.85 (d, J = 7.8 Hz, 1H), 7.62 (t, J = 7.3 Hz, 1H), 7.58-7.52 (m, 4H), 7.39 (s, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 167.7, 163.4, 134.5, 134.1, 133.3, 133.2, 131.9, 131.3, 129.2, 129.0, 128.7, 128.4, 128.1, 127.9, 127.8, 126.7, 125.7.



(*Z*)-4-(3-Cyanobenzylidene)-2-phenyloxazol-5(4*H*)-one (1j):[6] Following the general procedure as for **1a**, hippuric acid (268.5 mg, 1.5 mmol), 3-formylbenzonitrile (236.0 mg, 1.8 mmol), NaOAc (36.9 mg, 0.45 mmol), Ac_2O (0.6 mL, 6.0 mmol) in THF (8 mL) was reflux for 3 h. The given residue was purified by recrystallization from EtOH to give **1j** as a light yellow solid (268.1 mg, 65%). M.p. 209-211 ^oC; IR (KBr, cm^{-1}): 3080, 2223, 1800, 1653, 1553, 1289, 1166, 989, 922, 879, 688; ^1H NMR ($CDCl_3$, 500 MHz): δ 8.67 (s, 1H), 8.27 (d, J = 7.9 Hz, 1H), 8.21-8.20 (m, 2H), 7.71 (dt, J = 7.8, 1.3 Hz, 1H), 7.68-7.65 (m, 1H), 7.61-7.56 (m, 3H), 7.17 (s, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 166.8, 165.1, 136.1, 135.3 (two overlapped peaks), 134.6, 134.1, 133.6, 129.7, 129.1, 128.8, 127.9, 125.0, 118.4, 113.4; EI-MS m/z: 274 [M^+].



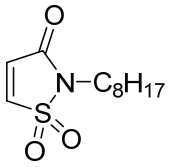
(*Z*)-4-(2-Ethoxybenzylidene)-2-phenyloxazol-5(4*H*)-one (1k):[1] Following the general procedure as for **1a**, hippuric acid (537.6 mg, 3.0 mmol), 2-ethoxy-benzaldehyde (424.0 μL, 3.6 mmol), NaOAc (73.8 mg, 0.9 mmol), Ac_2O (1.2 mL, 12.0 mmol) in THF (10 mL) was reflux for 3 h. The given residue was purified by recrystallization to give **1k** as a light yellow solid (451.7 mg, 51%). M.p. 180-182 ^oC; IR (KBr, cm^{-1}): 3064, 2984, 2884, 1785, 1644, 1586, 1558, 1446, 1246, 1161, 1037, 980, 859, 754, 689; ^1H NMR ($CDCl_3$, 500 MHz): δ 8.88 (dd, J = 7.9, 1.7 Hz, 1H), 8.19-8.17 (m, 2H), 7.89 (s, 1H), 7.60 (t, J = 7.3 Hz, 1H), 7.54 (t, J = 7.8, 2H), 7.42-7.39 (m, 1H), 7.08 (t, J = 7.6 Hz, 1H), 6.91 (d, J = 8.3 Hz, 1H), 4.13 (q, J = 7.0 Hz, 2H), 1.50 (t, J = 7.0 Hz, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 167.9, 162.9, 158.8, 133.1, 133.0 (two overlapped peaks), 132.4, 128.9, 128.3, 126.3, 125.8, 122.7, 120.8, 111.7, 64.2, 14.8; EI-MS m/z: 293 [M^+].



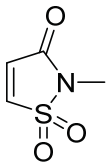
(*Z*)-4-(2-Fluorobenzylidene)-2-phenyloxazol-5(4*H*)-one (1l):[7] Following the general procedure as for **1a**, hippuric acid (537.6 mg, 3.0 mmol), 2-fluoro-benzaldehyde (446.8 mg, 3.6 mmol), NaOAc (73.8 mg, 0.9 mmol), Ac_2O (1.2 mL, 12.0 mmol) in THF (10 mL) was reflux for 3 h. The given residue was purified by recrystallization to give **1l** as a yellow solid (548.8 mg, 68%). M.p. 172-174 ^oC; IR (KBr, cm^{-1}): 3061, 3001, 1796, 1651, 1601, 1558, 1444, 1327, 1292, 1203, 985, 871, 761, 690; ^1H NMR ($CDCl_3$, 500 MHz): δ 8.89 (t, J = 7.4 Hz, 1H), 8.19 (d, J = 7.5 Hz, 2H), 7.63 (t, J = 7.4 Hz, 1H), 7.58 (s, 1H), 7.54 (t, J = 7.8 Hz, 2H), 7.46-7.42 (m, 1H), 7.30 (t, J = 7.6 Hz, 1H), 7.13 (t, J = 9.5 Hz, 1H); ^{19}F NMR ($CDCl_3$, 470 MHz): δ -114.0 (m, Ar-F); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 167.1, 164.2, 162.0 (d, $^1J_{C\text{-}F}$ = 256.7 Hz), 134.2, 133.6, 132.9 (d, $^3J_{C\text{-}F}$ = 8.8 Hz), 132.7, 129.0, 128.5, 125.4, 124.6 (d, $^3J_{C\text{-}F}$ = 3.6 Hz), 122.3 (d, $^3J_{C\text{-}F}$ = 7.5 Hz), 121.8 (d, $^2J_{C\text{-}F}$ = 10.7 Hz), 115.6 (d, $^2J_{C\text{-}F}$ = 21.9 Hz); LC-MS (ESI) m/z: 268 $[M+H]^+$.

2.2. Synthesis of *N*-Alkylisothiazol-3(2*H*)-one 1,1-dioxides

N-Alkylisothiazol-3(2*H*)-one 1,1-dioxides **2h**–**2i** are prepared as follows:

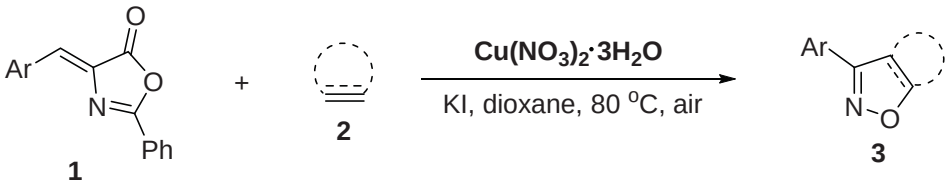


2-Octylisothiazol-3(2*H*)-one 1,1-dioxide (2h): To a solution of 2-octylisothiazol-3(2*H*)-one (410.0 μL, 2.0 mmol) in DCM (10 mL) at 25 °C is added *m*-CPBA (85%, 803.0 mg, 4.6 mmol) and the resulting mixture is stirred at room temperature for 15 h. The mixture was filtered through a pad of silica gel and washed with DCM. A saturated aqueous solution of $NaHCO_3$ was added, the organics were extracted with DCM, dried with Na_2SO_4, filtered and the solvent was removed under vacuum. The given residue was purified by recrystallization from hexane/DCM to give **2h** (380 mg, 78%) as a colorless liquid. IR (KBr, cm^{-1}): 3092, 2927, 2859, 1735, 1320, 1163, 918, 856, 790, 671; ^1H NMR ($CDCl_3$, 500 MHz): δ 7.37 (d, J = 7.2 Hz, 1H), 6.77 (d, J = 7.2 Hz, 1H), 3.64 (t, J = 7.5 Hz, 2H), 1.80-1.73 (m, 2H), 1.36-1.20 (m, 10H), 0.88 (t, J = 6.9 Hz, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 159.2, 138.4, 129.2, 39.8, 31.7, 29.1, 28.9, 28.1, 26.7, 22.6, 14.1; LC-MS (DART Positive) m/z: 246 $[M+H]^+$; HRMS (DART, Positive) calcd for $C_{11}H_{23}O_3N_2S$ $[M+NH_4]^+$ 263.1424, found 263.1422.

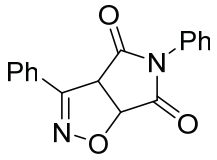


2-Methylisothiazol-3(2*H*)-one 1,1-dioxide (2i): To a solution of 2-methyl-isothiazol-3(2*H*)-one (203.3 mg, 2.0 mmol) in DCM (10 mL) at 25 °C is added *m*-CPBA (85%, 803.0 mg, 4.6 mmol) and the resulting mixture is stirred at room temperature for 15 h. The mixture was filtered through a pad of silica gel and washed with DCM. A saturated aqueous solution of $NaHCO_3$ was added, the organics were extracted with DCM, dried with Na_2SO_4, filtered and the solvent was removed under vacuum. The given residue was purified by recrystallization from hexane/DCM to give **2i** (205.2 mg, 85%) as a white solid. M.p. 106-108 ^oC; IR (KBr, cm^{-1}): 3173, 3094, 1741, 1332, 1300, 1163, 915, 856, 785, 668; ^1H NMR ($CDCl_3$, 500 MHz): δ 7.42 (d, J = 7.4 Hz, 1H), 6.80 (d, J = 7.4 Hz, 1H), 3.15 (s, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 158.8, 138.4, 129.4, 23.5; LC-MS (DART Positive) m/z: 148 [M^+H]; HRMS (ESI) m/z: calcd for $C_4H_6NO_3S$ $[M+H]^+$ 148.0063, found 148.0062.

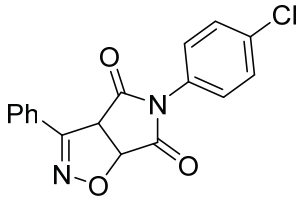
3. Synthesis and Characterization of Products



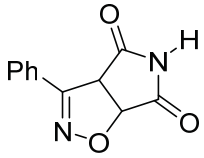
General Procedure: To a test tube were added **1** (0.3 mmol), **2** (0.45 mmol), $Cu(NO_3)_2{\cdot}3H_2O$ (0.6 mmol), KI (0.3 mmol) in dioxane (1.5 mL). The mixture was stirred at 80 ^oC for 4 h under an air atmosphere as monitored by TLC. Upon completion, the reaction mixture was cooled down to room temperature and the solvent was removed under reduced pressure. The residue was purified by column chromatography on silica gel to give product **3**.



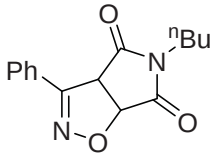
3,5-Diphenyl-3a,6a-dihydro-4*H*-pyrrolo[3,4-*d*]isoxazole-4,6(5*H*)-dione (3a):[8] Following the general procedure, the reaction mixture of **1a** (74.7 mg, 0.3 mmol), 1-phenyl-1*H*-pyrrole-2,5-dione **2a** (78.1 mg, 0.45 mmol), $Cu(NO_3)_2{\cdot}3H_2O$ (145.0 mg, 0.6 mmol), KI (49.8 mg, 0.3 mmol) in dioxane (1.5 mL). After 4 h at 80 °C, purification by column chromatography on silica gel (petroleum ether/ethyl acetate) afforded **3a** (63.6 mg, 73%). M.p. 176-177 ^oC; IR (KBr, cm^{-1}): 3065, 1791, 1719, 1595, 1491, 1448, 1386, 1199; ^1H NMR ($CDCl_3$, 500 MHz): δ 8.03 (dd, J = 7.6, 1.5 Hz, 2H), 7.49-7.39 (m, 6H), 7.28-7.26 (m, 2H), 5.66 (d, J = 9.7 Hz, 1H), 4.97 (d, J = 9.7 Hz, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 170.8, 169.8, 152.8, 131.3, 130.8, 129.3, 129.2, 128.9, 128.1, 126.7, 126.2, 80.4, 54.9; EI-MS m/z: 292 [M^+]; HRMS (EI) m/z: calcd for $C_{17}H_{12}N_2O_3$ [M^+] 292.0848, found 292.0844.



5-(4-Chlorophenyl)-3-phenyl-3a,6a-dihydro-4*H*-pyrrolo[3,4-*d*]isoxazole-4,6(5*H*)-dione (3b): Following the general procedure, the reaction mixture of **1a** (74.7 mg, 0.3 mmol), 1-(4-chlorophenyl)-1*H*-pyrrole-2,5-dione **2b** (93.6 mg, 0.45 mmol), $Cu(NO_3)_2 \cdot 3H_2O$ (145.0 mg, 0.6 mmol), KI (49.8 mg, 0.3 mmol) in dioxane (1.5 mL). After 4 h at 80 °C, purification by column chromatography on silica gel (petroleum ether/ethyl acetate) afforded **3b** (65.7 mg, 67%). M.p. 197-199 ^{o}C; IR (KBr, cm^{-1}): 3000, 2900, 1723, 1497, 1391, 1193, 894, 830, 759; ^{1}H NMR (500 MHz, DMSO-d_6): δ 7.95-7.93(m, 2H), 7.56 (d, J = 8.8 Hz, 2H), 7.51-7.50 (m, 3H), 7.33 (d, J = 8.7 Hz, 2H), 5.74 (d, J = 9.7 Hz, 1H), 5.41 (d, J = 9.7 Hz, 1H); ^{13}C NMR (125 MHz, DMSO-d_6): δ 172.3, 171.2, 153.8, 133.8, 131.3, 131.0, 129.6, 129.3, 129.2, 128.4, 127.6, 81.8, 55.8; EI-MS m/z: 326 [M^+]; HRMS (EI) m/z: calcd for $C_{17}H_{11}ClN_2O_3$ [M^+] 326.0458, found 326.0457.

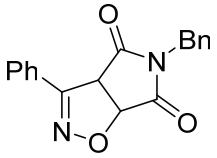


3-Phenyl-3a,6a-dihydro-4*H*-pyrrolo[3,4-*d*]isoxazole-4,6(5*H*)-dione (3c):[9] Following the general procedure, the reaction mixture of **1a** (74.7 mg, 0.3 mmol), 1*H*-pyrrole-2,5-dione **2c** (43.7 mg, 0.45 mmol), $Cu(NO_3)_2 \cdot 3H_2O$ (145.0 mg, 0.6 mmol), KI (49.8 mg, 0.3 mmol) in dioxane (1.5 mL). After 2 h at 80 °C, purification by column chromatography on silica gel (petroleum ether/DCM/ethyl acetate = 5/25/3) afforded **3c** (43.7 mg, 67%). M.p. 220-221 ^{o}C; IR (KBr, cm^{-1}): 3198, 3094, 1793, 1729, 1563, 1336, 1180, 771; ^{1}H NMR ((500 MHz, DMSO-d_6): δ 11.89 (s, 1H), 7.90-7.88 (m, 2H), 7.51-7.48 (m, 3H), 5.52 (d, J = 9.4 Hz, 1H), 5.15 (d, J = 9.4 Hz, 1H); ^{13}C NMR ((125 MHz, DMSO-d_6): δ 174.8, 173.7, 154.0, 131.2, 129.2, 128.3, 127.6, 82.8, 56.6; EI-MS m/z: 216 [M^+]; HRMS (EI) m/z: calcd for $C_{11}H_8N_2O_3$ [M^+] 216.0535, found 216.0541.

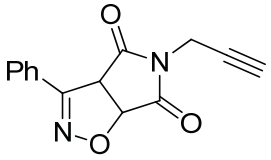


5-Butyl-3-phenyl-3a,6a-dihydro-4*H*-pyrrolo[3,4-*d*]isoxazole-4,6(5*H*)-dione (3d): Following the general procedure, the reaction mixture of **1a** (74.8 mg, 0.3 mmol), 1-butyl-1*H*-pyrrole-2,5-dione **2d** (69.2 mg, 0.45 mmol), $Cu(NO_3)_2 \cdot 3H_2O$ (145.0 mg, 0.6 mmol), KI (49.8 mg, 0.3 mmol) in dioxane (1.5 mL). After 4.5 h at 80 °C, purification by column chromatography on silica gel (petroleum ether/ethyl acetate) afforded **3d** (66.7 mg, 82%). M.p. 82-84 ^{o}C; IR (KBr, cm^{-1}): 3064, 2952, 1783, 1709, 1444, 1402, 1333; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.97-7.95 (m, 2H), 7.47-7.41 (m, 3H), 5.49 (d, J = 9.6 Hz, 1H), 4.81 (d, J = 9.6 Hz, 1H), 3.54-3.45 (m, 2H), 1.54-1.47

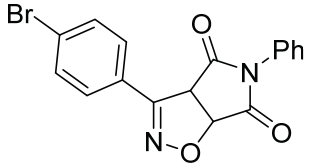
(m, 2H), 1.28-1.20 (m, 2H), 0.86 (t, J = 7.4 Hz, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 172.0, 170.9, 152.8, 131.1, 128.8, 128.0, 126.8, 80.4, 54.9, 39.5, 29.4, 19.9, 13.5; EI-MS m/z: 272 [M^+]; HRMS (EI) m/z: calcd for $C_{15}H_{16}N_2O_3$ [M^+] 272.1161, found 272.1159.



5-Benzyl-3-phenyl-3a,6a-dihydro-4*H*-pyrrolo[3,4-*d*]isoxazole-4,6(5*H*)-dione (3e): Following the general procedure, the reaction mixture of **1a** (74.8 mg, 0.3 mmol), 1-benzyl-1*H*-pyrrole-2,5-dione **2e** (84.3 mg, 0.45 mmol), $Cu(NO_3)_2{\cdot}3H_2O$ (145.0 mg, 0.6 mmol), KI (49.8 mg, 0.3 mmol) in dioxane (1.5 mL). After 4.5 h at 80 °C, purification by column chromatography on silica gel (petroleum ether/ethyl acetate) afforded **3e** (65.1 mg, 71%) as a white solid. M.p. 82-84 ^{o}C; IR (KBr, cm^{-1}): 3064, 2952, 1783, 1709, 1444, 1402, 1333; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.98 (dd, J = 7.7, 1.6 Hz, 2H), 7.48-7.43 (m, 3H), 7.36-7.27 (m, 5H), 5.48 (d, J = 9.6 Hz, 1H), 4.79 (d, J = 9.6 Hz, 1H), 4.70 (d, J = 14.1 Hz, 1H), 4.62 (d, J = 14.1 Hz, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 171.5, 170.6, 152.7, 134.6, 131.2, 128.9, 128.8, 128.4, 128.0, 126.8, 80.5, 54.9, 43.3; EI-MS m/z: 306 [M^+]; HRMS (EI) m/z: calcd for $C_{18}H_{14}N_2O_3$ [M^+] 306.1004, found 306.1006.

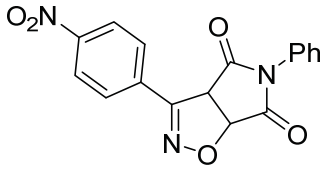


3-Phenyl-5-(prop-2-yn-1-yl)-3a,6a-dihydro-4*H*-pyrrolo[3,4-*d*]isoxazole-4,6(5*H*)-dione (3f): Following the general procedure, the reaction mixture of **1a** (74.7 mg, 0.3 mmol), 1-(prop-2-yn-1-yl)-1*H*-pyrrole-2,5-dione **2f** (61.0 mg, 0.45 mmol), $Cu(NO_3)_2{\cdot}3H_2O$ (145.0 mg, 0.6 mmol), KI (49.8 mg, 0.3 mmol) in dioxane (1.5 mL). After 2.5 h at 60 °C, purification by column chromatography on silica gel (petroleum ether/ethyl acetate) afforded **3f** (31.8 mg, 42%) as a colorless liquid. IR (KBr, cm^{-1}): 3285, 3063, 2974, 1795, 1725, 1425, 1389, 1338, 1180, 1043, 903, 759, 689, 628; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.98 (dd, J = 7.8, 1.4 Hz, 2H), 7.50-7.44 (m, 3H), 5.56 (d, J = 9.7 Hz, 1H), 4.88 (d, J = 9.7 Hz, 1H), 4.33-4.23 (m, 2H), 2.22 (t, J = 2.5 Hz, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 170.3, 169.4, 152.4, 131.3, 128.9, 128.0, 126.6, 80.4, 75.4, 72.5, 55.0, 28.7; LC-MS (DART Positive) m/z: 255 [M+H]$^+$; HRMS (DART Positive) m/z: calcd for $C_{14}H_{11}N_3O_2$ [M+H]$^+$ 255.0764, found 255.0761.

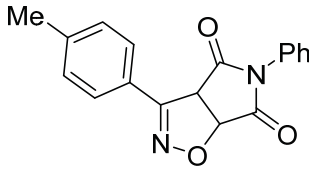


3-(4-Bromophenyl)-5-phenyl-3a,6a-dihydro-4*H*-pyrrolo[3,4-*d*]isoxazole-4,6(5*H*)-dione (3g): Following the general procedure, the reaction mixture of **1f** (98.5 mg, 0.3

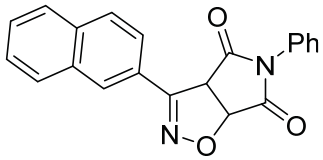
mmol), 1-phenyl-1*H*-pyrrole-2,5-dione **2a** (77.9 mg, 0.45 mmol), $Cu(NO_3)_2 \cdot 3H_2O$ (145.0 mg, 0.6 mmol), KI (49.8 mg, 0.3 mmol) in dioxane (1.5 mL). After 4 h at 80 °C, purification by column chromatography on silica gel (petroleum ether/ethyl acetate) afforded **3g** (89.6 mg, 80%). M.p. 204-206 ^oC; IR (KBr, cm^{-1}): 2923, 1722, 1496, 1496, 1389, 1194, 893, 832, 691, 619; ^1H NMR ($CDCl_3$, 500 MHz): δ 7.91 (d, J = 6.2 Hz, 2H), 7.59 (d, J = 6.9 Hz, 2H), 7.47-7.42 (m, 3H), 7.27-7.26 (m, 2H), 5.68 (d, J = 9.4 Hz, 1H), 4.93 (d, J = 9.4 Hz, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 170.6, 169.7, 152.1, 132.2, 130.7, 129.5, 129.3 (two overlapped peaks), 126.1, 125.9, 125.7, 80.6, 54.7; EI-MS m/z: 370 [M^+ (^{79}Br)], 372 [M^+ (^{81}Br)]; HRMS (EI) m/z: calcd for $C_{17}H_{11}BrN_2O_3$ [M^+] 369.9953, found 369.9955.



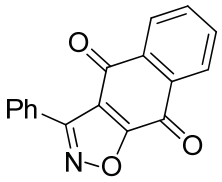
3-(4-Nitrophenyl)-5-phenyl-3a,6a-dihydro-4*H*-pyrrolo[3,4-*d*]isoxazole-4,6(5*H*)-dione (3h):[10] Following the general procedure, the reaction mixture of **1g** (88.5 mg, 0.3 mmol), **2a** (77.9 mg, 0.45 mmol), $Cu(NO_3)_2 \cdot 3H_2O$ (145.0 mg, 0.6 mmol), KI (49.8 mg, 0.3 mmol) in dioxane (1.5 mL). After 4 h at 80 °C, purification by column chromatography on silica gel (petroleum ether/ethyl acetate) afforded **3h** (69.4 mg, 69%). M.p. 251-252 ^oC; IR (KBr, cm^{-1}): 3068, 2956, 1791, 1723, 1518, 1386, 1345, 1205, 910, 856, 744, 688, 624; ^1H NMR (DMSO-d_6, 500 MHz): δ 8.36 (d, J = 8.8 Hz, 2H), 8.20 (d, J = 8.8 Hz, 2H), 7.50-7.42 (m, 3H), 7.29 (d, J = 7.4 Hz, 2H), 5.84 (d, J = 9.8 Hz, 1H), 5.49 (d, J = 9.8 Hz, 1H); ^{13}C NMR (DMSO-d_6, 125 MHz): δ 172.2, 171.3, 153.1, 149.0, 133.8, 132.1, 129.7, 129.5, 129.4, 127.5, 124.4, 82.7, 55.4; EI-MS m/z: 337 [M^+]; HRMS (EI) m/z: calcd for $C_{17}H_{11}N_3O_5$ [M^+] 337.0699, found 337.0695.



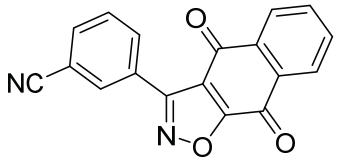
5-Phenyl-3-(*p*-tolyl)-3a,6a-dihydro-4*H*-pyrrolo[3,4-*d*]isoxazole-4,6(5*H*)-dione (3i):[10] Following the general procedure, the reaction mixture of **1h** (79.1 mg, 0.3 mmol), **2a** (78.0 mg, 0.45 mmol), $Cu(NO_3)_2 \cdot 3H_2O$ (145.0 mg, 0.6 mmol), KI (49.8 mg, 0.3 mmol) in dioxane (1.5 mL). After 4 h at 80 °C, purification by column chromatography on silica gel (petroleum ether/ethyl acetate) afforded **3i** (62.2 mg, 68%). M.p. 179-180 ^oC; IR (KBr, cm^{-1}): 3054, 2963, 1724, 1499, 1385, 1193, 892, 854, 695; ^1H NMR ($CDCl_3$, 500 MHz): δ 7.93 (d, J = 8.2 Hz, 2H), 7.48-7.40 (m, 3H), 7.28 (d, J = 7.3 Hz, 4H), 5.65 (d, J = 9.7 Hz, 1H), 4.96 (d, J = 9.7 Hz, 1H), 2.42 (s, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 171.1, 170.0, 152.8, 141.8, 130.9, 129.6, 129.3, 129.2, 128.0, 126.2, 123.9, 80.3, 55.0, 21.6; EI-MS m/z: 306 [M^+]; HRMS (EI) m/z: calcd for $C_{18}H_{14}N_2O_3$ [M^+] 306.1004, found 306.1010.



3-(Naphthalen-2-yl)-5-phenyl-3a,6a-dihydro-4*H*-pyrrolo[3,4-*d*]isoxazole-4,6(5*H*)-dione (3j): Following the general procedure, the reaction mixture of **1i** (89.9 mg, 0.3 mmol), **2a** (77.9 mg, 0.45 mmol), $Cu(NO_3)_2 \cdot 3H_2O$ (145.0 mg, 0.6 mmol), KI (49.8 mg, 0.3 mmol) in dioxane (1.5 mL). After 4 h at 80 °C, purification by column chromatography on silica gel (petroleum ether/ethyl acetate) afforded **3j** (73.7 mg, 72%). M.p. 203-204 ^{o}C; IR (KBr, cm^{-1}): 3059, 1794, 1723, 1594, 1495, 1379, 1193, 899, 745; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 8.53 (s, 1H), 8.08 (d, J = 8.7 Hz, 1H), 7.94 (d, J = 8.7 Hz, 1H), 7.86 (t, J = 9 Hz, 2H), 7.58-7.38 (m, 5H), 7.28 (d, J = 7.6 Hz, 2H), 5.71 (d, J = 9.7 Hz, 1H), 5.07 (d, J = 9.7 Hz, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 170.8, 170.0, 152.9, 134.4, 132.8, 130.8, 129.6, 129.3, 129.2, 129.0, 128.8, 127.9, 127.8, 126.9, 126.2, 124.2, 123.8, 80.6, 55.0; EI-MS m/z: 342 [M^+]; HRMS (EI) m/z: calcd for $C_{21}H_{14}N_2O_3$ [M^+] 342.1004, found 342.1001.

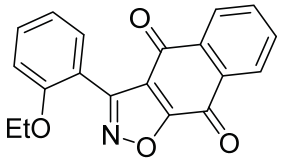


3-Phenylnaphtho[2,3-*d*]isoxazole-4,9-dione (3k):[11] Following the general procedure, Following the general procedure, the reaction mixture of **1a** (74.9 mg, 0.3 mmol), naphthalene-1,4-dione **2g** (71.1 mg, 0.45 mmol), $Cu(NO_3)_2 \cdot 3H_2O$ (145.0 mg, 0.6 mmol), KI (49.8 mg, 0.3 mmol) in dioxane (1.5 mL). After 2 h at 80 °C, purification by column chromatography on silica gel (petroleum ether/ethyl acetate) afforded **3k** (55.9 mg, 68%). M.p. 137-138 ^{o}C; IR (KBr, cm^{-1}): 3072, 1685, 1584, 1433, 1340, 1261, 1204, 1167, 919, 716; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 8.30-8.26 (m, 2H), 8.16 (dd, J = 7.6, 1.4 Hz, 2H), 7.89-7.82 (m, 2H), 7.59-7.53 (m, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 178.7, 173.4, 166.3, 161.0, 135.3, 134.3, 133.8, 131.8, 131.3, 129.4, 128.7, 127.8, 127.3, 126.2, 119.6; EI-MS m/z: 275 [M^+]; HRMS (EI) m/z: calcd for $C_{17}H_9NO_3$ [M^+] 275.0582, found 275.0587.

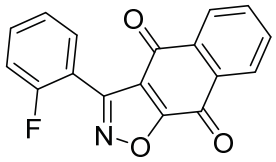


3-(4,9-Dioxo-4,9-dihydronaphtho[2,3-*d*]isoxazol-3-yl)benzonitrile (3l): Following the general procedure, the reaction mixture of **1j** (82.3 mg, 0.3 mmol), naphthalene-1,4-dione **2g** (71.1 mg, 0.45 mmol), $Cu(NO_3)_2 \cdot 3H_2O$ (145.0 mg, 0.6 mmol), KI (49.8 mg, 0.3 mmol) in dioxane (1.5 mL). After 4 h at 80 °C, purification by column chromatography on silica gel (petroleum ether/ethyl acetate) afforded **3l** (68.9 mg, 76%). M.p. 203-205 ^{o}C; IR (KBr, cm^{-1}): 3080, 2231, 1688, 1581, 1461, 1427, 1336, 1204, 929, 804, 721; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 8.58 (s, 1H), 8.47 (d, J = 8.0 Hz, 1H), 8.31-8.28 (m, 2H), 7.95-7.82 (m, 3H), 7.69 (t, J = 7.8 Hz, 1H); ^{13}C

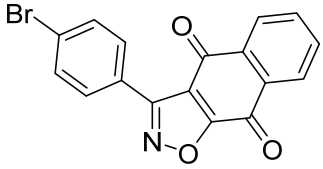
NMR ($CDCl_3$, 125 MHz): δ 178.6, 173.0, 166.6, 159.3, 135.6, 134.7, 134.6, 133.5, 133.4, 133.0, 131.8, 129.7, 127.9, 127.7, 127.5, 119.4, 118.0, 113.3; LC-MS (DART Positive) m/z: 301 $[M+H]^+$; HRMS (DART Positive) m/z: calcd for $C_{18}H_9N_2O_3$ $[M+H]^+$ 301.0608, found 301.0605.



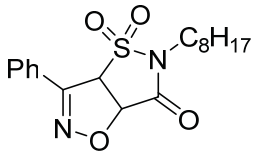
3-(2-Ethoxyphenyl)naphtho[2,3-*d*]isoxazole-4,9-dione (3m): Following the general procedure, the reaction mixture of **1k** (88.1 mg, 0.3 mmol), naphthalene-1,4-dione **2g** (71.1 mg, 0.45 mmol), $Cu(NO_3)_2{\cdot}3H_2O$ (145.0 mg, 0.6 mmol), KI (49.8 mg, 0.3 mmol) in dioxane (1.5 mL). After 4 h at 80 °C, purification by column chromatography on silica gel (petroleum ether/ethyl acetate) afforded **3m** (63.6 mg, 66%). M.p. 193-195 ^oC; IR (KBr, cm^{-1}): 3071, 2981, 2939, 2891, 1687, 1594, 1453, 1336, 1255, 1199, 1037, 918, 762, 721; ^1H NMR ($CDCl_3$, 500 MHz): δ 8.30-8.28 (m, 1H), 8.21-8.17 (m, 1H), 7.85-7.80 (m, 2H), 7.54-7.50 (m, 2H), 7.09-7.04 (m, 2H), 4.09 (q, J = 7.0 Hz, 2H), 1.18 (t, J = 7.0 Hz, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 178.1, 173.6, 165.0, 158.7, 157.5, 135.1, 134.0, 133.8, 132.4, 132.1, 130.6, 127.4, 127.2, 121.5, 120.4, 115.5, 111.9, 63.9, 14.6; LC-MS (DART Positive) m/z: 320 $[M+H]^+$; HRMS (DART Positive) m/z: calcd for $C_{19}H_{14}NO_4$ $[M+H]^+$ 320.0917, found 320.0914.



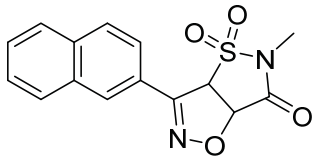
3-(2-Fluorophenyl)naphtho[2,3-*d*]isoxazole-4,9-dione (3n): Following the general procedure, the reaction mixture of **1l** (80.3 mg, 0.3 mmol), **2g** (71.1 mg, 0.45 mmol), $Cu(NO_3)_2{\cdot}3H_2O$ (145.0 mg, 0.6 mmol), KI (49.8 mg, 0.3 mmol) in dioxane (1.5 mL). After 4 h at 80 °C, purification by column chromatography on silica gel (petroleum ether/ethyl acetate) afforded **3n** (58.6 mg, 67%). M.p. 192-194 ^oC; IR (KBr, cm^{-1}): 3077, 1686, 1584, 1514, 1465, 1336, 1197, 918, 758, 716; ^1H NMR ($CDCl_3$, 500 MHz): δ 8.31-8.29 (m, 1H), 8.23-8.21 (m, 1H), 7.87-7.82 (m, 2H), 7.70 (td, J = 7.4, 1.8 Hz, 1H), 7.62-7.57 (m, 1H), 7.35-7.27 (m, 2H); ^{19}F NMR ($CDCl_3$, 470 MHz): δ -111.2 (m, Ar-F); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 178.1, 173.2, 165.5, 160.6 (d, $^1J_{C\text{-}F}$ = 252.8 Hz), 156.4, 135.3, 134.4, 133.6, 133.1 (d, $^3J_{C\text{-}F}$ = 8.4 Hz), 132.0, 131.2 (d, $^4J_{C\text{-}F}$ = 1.2 Hz), 127.5 (d, $^3J_{C\text{-}F}$ = 9.1 Hz), 124.4 (d, $^4J_{C\text{-}F}$ = 3.6 Hz), 120.6, 116.2 (d, $^2J_{C\text{-}F}$ = 21.0 Hz), 114.7 (d, $^2J_{C\text{-}F}$ = 14.4 Hz); EI-MS m/z: 293 $[M^+]$; HRMS (EI) m/z: calcd for $C_{17}H_8FNO_3$ $[M^+]$ 293.0488, found 293.0497.



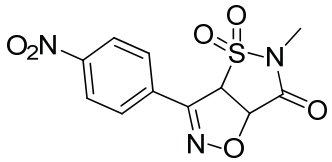
3-(4-Bromophenyl)naphtho[2,3-*d*]isoxazole-4,9-dione (3o): Following the general procedure, the reaction mixture of **1f** (98.6 mg, 0.3 mmol), **2g** (71.1 mg, 0.45 mmol), $Cu(NO_3)_2 \cdot 3H_2O$ (145.0 mg, 0.6 mmol), KI (49.8 mg, 0.3 mmol) in dioxane (1.5 mL). After 4 h at 80 °C, purification by column chromatography on silica gel (petroleum ether/ethyl acetate) afforded **3o** (64.9 mg, 61%). M.p. 178-180 ^oC; IR (KBr, cm^{-1}): 3082, 2924, 1686, 1588, 1443, 1335, 1261, 1195, 1001, 911, 821, 721; ^1H NMR (DMSO-d_6, 500 MHz): δ 8.19-8.15 (m, 2H), 8.04 (d, J = 8.5 Hz, 2H), 7.98-7.94 (m, 2H), 7.83 (d, J = 8.5 Hz, 2H); ^{13}C NMR (DMSO-d_6, 500 MHz): δ 179.1, 173.6, 167.1, 159.9, 135.8, 135.0, 134.0, 132.3, 131.5, 127.5, 127.1, 125.8, 125.5, 119.4; EI-MS m/z: 352 [M^+ (^{79}Br)], 353 [M^+ (^{81}Br)]; HRMS (EI) m/z: calcd for $C_{17}H_8BrNO_3$ [M^+] 352.9688, found 352.9685.



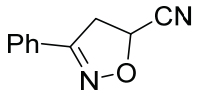
5-Octyl-3-phenyl-3a,6a-dihydroisothiazolo[5,4-*d*]isoxazol-6(5*H*)-one 4,4-dioxide (3p): Following the general procedure, the reaction mixture of **1a** (74.8 mg, 0.3 mmol), **2h** (110.4 mg, 0.45 mmol), $Cu(NO_3)_2 \cdot 3H_2O$ (145.0 mg, 0.6 mmol), KI (49.8 mg, 0.3 mmol) in dioxane (1.5 mL). After 4 h at 80 °C, purification by column chromatography on silica gel (petroleum ether/ethyl acetate) afforded **3p** (55.2 mg, 50%). M.p. 81-83 ^oC; IR (KBr, cm^{-1}): 2924, 2856, 1738, 1458, 1348, 1156, 1077, 917, 772, 689, 554; ^1H NMR ($CDCl_3$, 500 MHz): δ 7.78-7.76 (m, 2H), 7.54-7.46 (m, 3H), 5.72 (d, J = 10.4 Hz, 1H), 5.52 (d, J = 10.4 Hz, 1H), 3.68-3.58 (m, 2H), 1.77-1.71 (m, 2H), 1.31-1.25 (m, 10H), 0.87 (t, J = 6.9 Hz, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 160.1, 151.1, 131.7, 129.3, 127.3, 126.5, 82.7, 69.6, 41.2, 31.7, 29.0, 28.9, 28.0, 26.6, 22.6, 14.1; EI-MS m/z: 364 [M^+]; HRMS (EI) m/z: calcd for $C_{18}H_{24}N_2O_4S$ [M^+] 364.1457, found 364.1458.



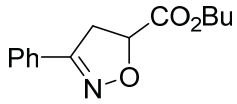
5-Methyl-3-(naphthalen-2-yl)-5,6a-dihydroisothiazolo[5,4-*d*]isoxazol-6(3a*H*)-one 4,4-dioxide (3q): Following the general procedure, the reaction mixture of **1i** (89.8 mg, 0.3 mmol), **2i** (66.4 mg, 0.45 mmol), $Cu(NO_3)_2 \cdot 3H_2O$ (145.0 mg, 0.6 mmol), KI (49.8 mg, 0.3 mmol) in dioxane (1.5 mL). After 4 h at 80 °C, purification by column chromatography on silica gel (petroleum ether/ethyl acetate) afforded **3q** (46.3 mg, 49%). M.p. 198-200 ^oC; IR (KBr, cm^{-1}): 2952, 2923, 1734, 1462, 1340, 1218, 1149, 917, 804, 745, 612; ^1H NMR ($CDCl_3$, 500 MHz): δ 8.07 (d, J = 1.0 Hz, 1H), 7.98-7.86 (m, 4H), 7.62-7.56 (m, 2H), 5.80 (d, J = 10.4 Hz, 1H), 5.68 (d, J = 10.4 Hz, 1H), 3.18 (s, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 159.9, 151.2, 134.6, 132.8, 129.4, 128.9, 128.2 (two overlapped peaks), 128.0, 127.3, 124.0, 123.2, 83.1, 69.6, 25.0; HRMS (ESI) m/z calcd for $C_{15}H_{13}N_2O_4S$ [M^+H] 317.0596, found 317.0582.



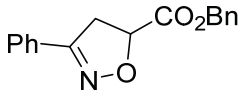
5-Methyl-3-(4-nitrophenyl)-5,6a-dihydroisothiazolo[5,4-*d*]isoxazol-6(3a*H*)-one 4,4-dioxide (3r): Following the general procedure, the reaction mixture of **1g** (88.5 mg, 0.3 mmol), **2i** (66.3 mg, 0.45 mmol), $Cu(NO_3)_2 \cdot 3H_2O$ (145.0 mg, 0.6 mmol), KI (49.8 mg, 0.3 mmol) in dioxane (1.5 mL). After 4 h at 80 °C, purification by column chromatography on silica gel (petroleum ether/ethyl acetate) afforded **3r** (19.7 mg, 21%). M.p. 260-261 ^{o}C; IR (KBr, cm^{-1}): 3086, 2963, 1720, 1520, 1419, 1346, 1263, 1153, 1099, 1025, 925, 803, 740, 690; ^{1}H NMR (DMSO-d_6, 500 MHz): δ 8.40 (d, J = 8.9 Hz, 2H), 8.04 (d, J= 8.9 Hz, 2H), 6.60 (d, J = 10.3 Hz, 1H), 6.12 (d, J = 10.3 Hz, 1H), 3.04 (s, 3H); ^{13}C NMR (DMSO-d_6, 125 MHz): δ 161.1, 151.6, 149.3, 133.3, 128.9, 124.9, 85.2, 69.3, 24.8; EI-MS m/z: 312 $[M+H]^+$; HRMS (EI) m/z: calcd for $C_{11}H_{10}N_3O_6S$ $[M+H]^+$ 312.0285, found 312.0283.



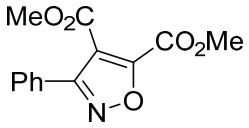
3-Phenyl-4,5-dihydroisoxazole-5-carbonitrile (3s):[12] Following the general procedure, the reaction mixture of **1a** (74.7 mg, 0.3 mmol), acrylonitrile (60.0 μL, 0.9 mmol), $Cu(NO_3)_2 \cdot 3H_2O$ (290.0 mg, 1.2 mmol), KI (99.6 mg, 0.6 mmol) in CH_3CN (1.5 mL). After 4.5 h at 50 °C, purification by column chromatography on silica gel (petroleum ether/ethyl acetate) afforded **3s** (29.1 mg, 56%). M.p. 63-66 ^{o}C; IR (KBr, cm^{-1}): 2990, 2429, 1564, 1444, 1352, 925, 870, 759, 685; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.68-7.66 (m, 2H), 7.51-7.43 (m, 3H), 5.38 (dd, J = 10.5, 6.3 Hz, 1H), 3.81-3.71 (m, 2H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 156.3, 131.3, 129.1, 127.4, 127.1, 117.1, 66.6, 41.2; HRMS (ESI) m/z calcd for $C_{10}H_9N_2O$ $[M^+H]^+$ 173.0715, found 173.0721.



Butyl 3-phenyl-4,5-dihydroisoxazole-5-carboxylate (3t): Following the general procedure, the reaction mixture of **1a** (74.7 mg, 0.3 mmol), *n*-butyl acrylate (66.0 μL, 0.45 mmol), $Cu(NO_3)_2 \cdot 3H_2O$ (145.0 mg, 0.6 mmol), KI (49.7 mg, 0.3 mmol) in CH_3CN (1.5 mL). After 4 h at 80 °C, purification by column chromatography on silica gel (petroleum ether/ethyl acetate) afforded **3t** (42.9 mg, 58%) as a yellow liquid; IR (KBr, cm^{-1}): 3061, 2961, 2873, 1742, 1454, 1352, 1205, 892, 760; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.68-7.66 (m, 2H), 7.43-7.39 (m, 3H), 5.16 (dd, J = 10.5, 7.8, Hz 1H), 4.20 (t, J = 6.8 Hz, 1H), 3.64-3.62 (m, 2H), 1.70-1.63 (m, 2H), 1.43-1.35 (m, 2H), 0.93 (t, J = 7.4 Hz, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 170.3, 156.0, 130.6, 128.8, 128.6, 126.9, 78.1, 65.9, 38.9, 30.5, 19.0, 13.7; EI-MS m/z: 247 $[M^+]$; HRMS (EI) m/z: calcd for $C_{14}H_{17}NO_3$ $[M^+]$ 247.1208, found 247.1215.

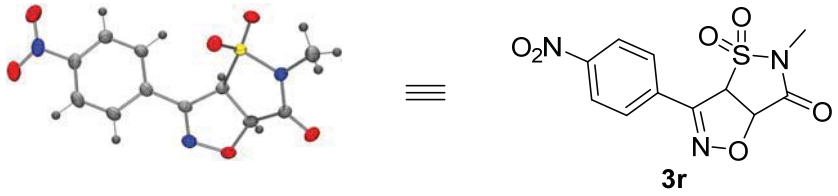


Benzyl 3-phenyl-4,5-dihydroisoxazole-5-carboxylate (3u):[13] Following the general procedure, the reaction mixture of **1a** (74.7 mg, 0.3 mmol), benzyl acrylate (68 μL, 0.45 mmol), $Cu(NO_3)_2 \cdot 3H_2O$ (145.0 mg, 0.6 mmol), KI (49.7 mg, 0.3 mmol) in CH_3CN (1.5 mL). After 4.5 h at 80 °C, purification by column chromatography on silica gel (petroleum ether/ethyl acetate) afforded **3u** (50.7 mg, 60%). M.p. 53-55 ^oC; IR (KBr, cm^{-1}): 3063, 2928, 1749, 1450, 1347, 1197, 1018, 886, 758, 696; ^1H NMR ($CDCl_3$, 500 MHz): δ 7.67 (dd, J = 7.6, 1.6 Hz, 2H), 7.45-7.33 (m, 8H), 5.27-5.19 (m, 3H), 3.69-3.59 (m, 2H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 170.0, 156.0, 135.1, 130.6, 128.8, 128.7, 128.6, 128.5, 128.4, 127.0, 78.1, 67.6, 38.9; EI-MS m/z: 281 [M^+]; HRMS (EI) m/z: calcd for $C_{17}H_{15}NO_3$ [M^+] 281.1052, found 281.1053.



Dimethyl 3-phenylisoxazole-4,5-dicarboxylate (3v):[14] Following the general procedure, the reaction mixture of **1a** (74.7 mg, 0.3 mmol), dimethyl acetylenedicarboxylate (54.0 μL, 0.45 mmol), $Cu(NO_3)_2 \cdot 3H_2O$ (145.0 mg, 0.6 mmol), KI (49.7 mg, 0.3 mmol) in CH_3CN (1.5 mL). After 4 h at 80 °C, purification by column chromatography on silica gel (petroleum ether/ethyl acetate) afforded **3v** (43.4 mg, 55%). M.p. 62-64 ^oC; IR (KBr, cm^{-1}): 3457, 2955, 2923, 1716, 1429, 1222, 1066, 918, 794, 686; ^1H NMR ($CDCl_3$, 500 MHz): δ 7.70-7.68 (m, 2H), 7.53-7.46 (m, 3H), 4.02 (s, 3H), 3.90 (s, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 161.8, 161.3, 159.4, 156.5, 130.7, 128.9, 128.2, 126.9, 116.1, 100.0, 53.4, 53.2; EI-MS m/z: 261 [M^+]; HRMS (EI) m/z: calcd for $C_{13}H_{11}NO_5$ [M^+] 261.0637, found 261.0633.

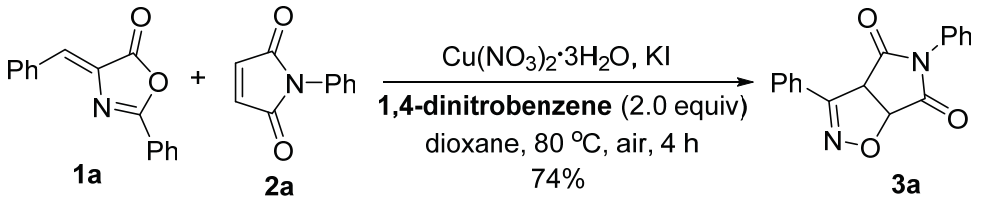
4. X-Ray Crystallographic Analysis for Compound 3r



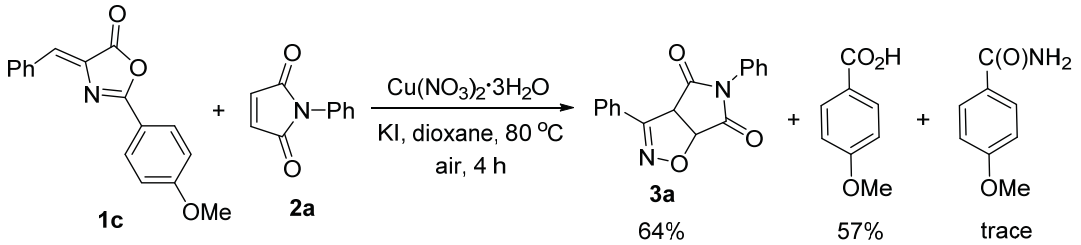
3r

Crystallographic data for **3r**: $C_{11}H_9N_3O_6S$, M = 311.27, orthorhombic, Pbca (No. 61), a = 6.495 (3) Å, b = 17.713 (8) Å, c = 21.586 (10) Å, V = 2483 (2) $Å^3$, Z = 8, Crystal size: 0.25×0.20×0.15 mm, T = 293 K, ρ_{calcd} = 1.665 $g \cdot cm^{-3}$, R_1 = 0.0374 (I>4σ(I)), wR_2 = 0.111 (all data), GOF = 1.031, reflections collected/unique: 14604 / 2926 (Rint = 0.0289), Data: 2306, restraints: 0, parameters: 198. CCDC 1884631 (**3r**) contains the supplementary crystallographic data for this paper. The data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

5. Mechanistic Studies

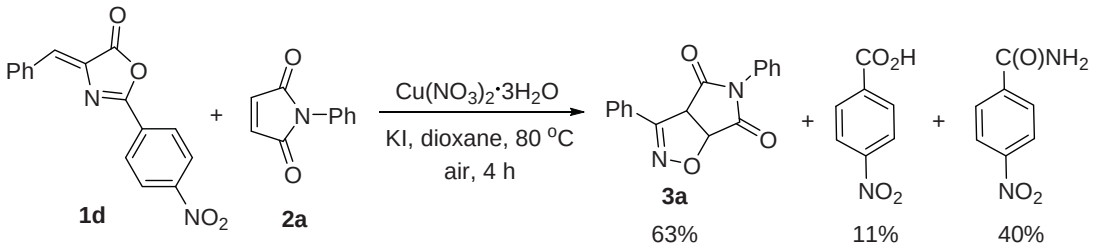


To a test tube were added **1a** (74.7 mg, 0.3 mmol), **2a** (77.9 mg, 0.45 mmol), $Cu(NO_3)_2{\cdot}3H_2O$ (145.0 mg, 0.6 mmol), KI (49.8, 0.3 mmol) and 1,4-dinitrobenzene (101.0 mg, 0.6 mmol) in dioxane (1.5 mL). The mixture was stirred at 80 ^oC for 4 h under an air atmosphere. Upon completion of the reaction, the solution was cooled down to room temperature and the solvent was removed under reduced pressure. The residue was purification by column chromatography on silica gel (petroleum ether/ethyl acetate) afforded **3a** (65 mg, 74%).



To a test tube were added **1c** (83.7 mg, 0.3 mmol), **2a** (77.9 mg, 0.45 mmol), $Cu(NO_3)_2{\cdot}3H_2O$ (145.0 mg, 0.6 mmol) and KI (49.8, 0.3 mmol) in dioxane (1.5 mL). The mixture was stirred at 80 ^oC for 4 h under an air atmosphere. Upon completion of the reaction, the solution was cooled down to room temperature and the solvent was removed under reduced pressure. The residue was purification by column chromatography on silica gel (petroleum ether/ethyl acetate) afforded **3a** (56.3 mg, 64%) and 4-methoxybenzoic acid (26.0 mg, 57%) as a white solid. No observable 4-methoxylbenzamide was isolated.

Characterization of 4-methoxybenzoic acid:[15] ^1H NMR ($CDCl_3$, 500 MHz): δ 8.09 (d, J = 8.9 Hz, 2H), 6.98 (d, J = 8.9 Hz, 2H), 3.91 (s, 3H).

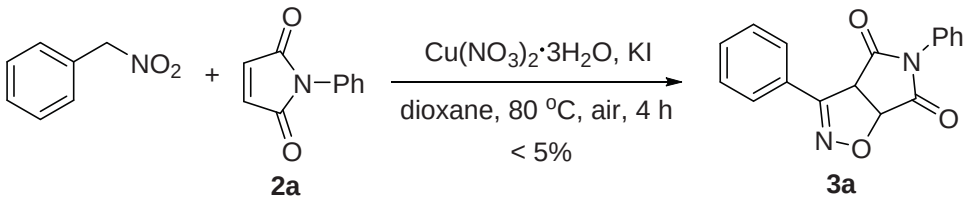


To a test tube were added **1d** (88.3 mg, 0.3 mmol), **2a** (77.9 mg, 0.45 mmol), $Cu(NO_3)_2{\cdot}3H_2O$ (145.0 mg, 0.6 mmol) and KI (49.8, 0.3 mmol) in dioxane (1.5 mL). The mixture was stirred at 80 ^oC for 4 h under an air atmosphere. Upon completion of

the reaction, the solution was cooled down to room temperature and the solvent was removed under reduced pressure. The residue was purification by column chromatography on silica gel (petroleum ether/ethyl acetate) afforded **3a** (55.0 mg, 63%), 4-nitrobenzamide (20.0 mg, 40%) and 4-nitrobenzoic acid (5.6 mg, 11%).

Characterization of 4-nitrobenzamide:[16] ^{1}H NMR (DMSO-d_6, 500 MHz): δ 8.30-8.28 (m, 3H), 8.09 (d, J = 8.7 Hz, 2H), 7.73 (s, 1H).

Characterization of 4-nitrobenzoic acid:[17] ^{1}H NMR (DMSO-d_6, 500 MHz): δ 13.65 (s, 1H), 8.32 (d, J = 8.7 Hz, 2H), 8.17 (d, J = 8.7 Hz, 2H).



2a **3a**

To a test tube were added (nitromethyl)benzene[18] (41 mg, 0.3 mmol), **2a** (77.9 mg, 0.45 mmol), $Cu(NO_3)_2{\cdot}3H_2O$ (145.0 mg, 0.6 mmol) and KI (49.8, 0.3 mmol) in dioxane (1.5 mL). The mixture was stirred at 80 °C for 4 h under an air atmosphere. Upon completion of the reaction, the solution was cooled down to room temperature and the solvent was removed under reduced pressure. The residue was purification by column chromatography on silica gel (petroleum ether/ethyl acetate) afforded **3a** (<5%) and (nitromethyl)benzene (with 95% recovery).

6. Reference

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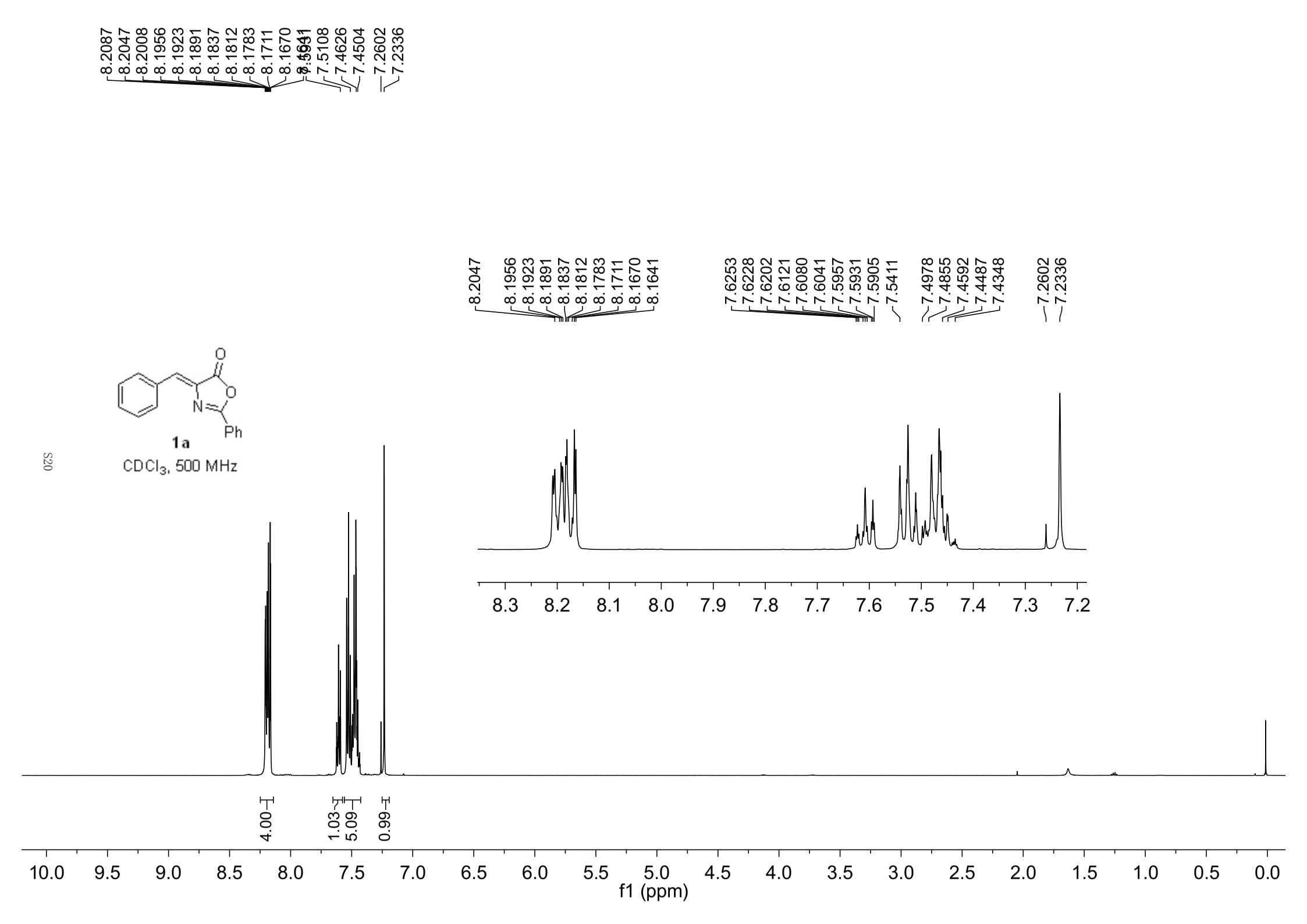
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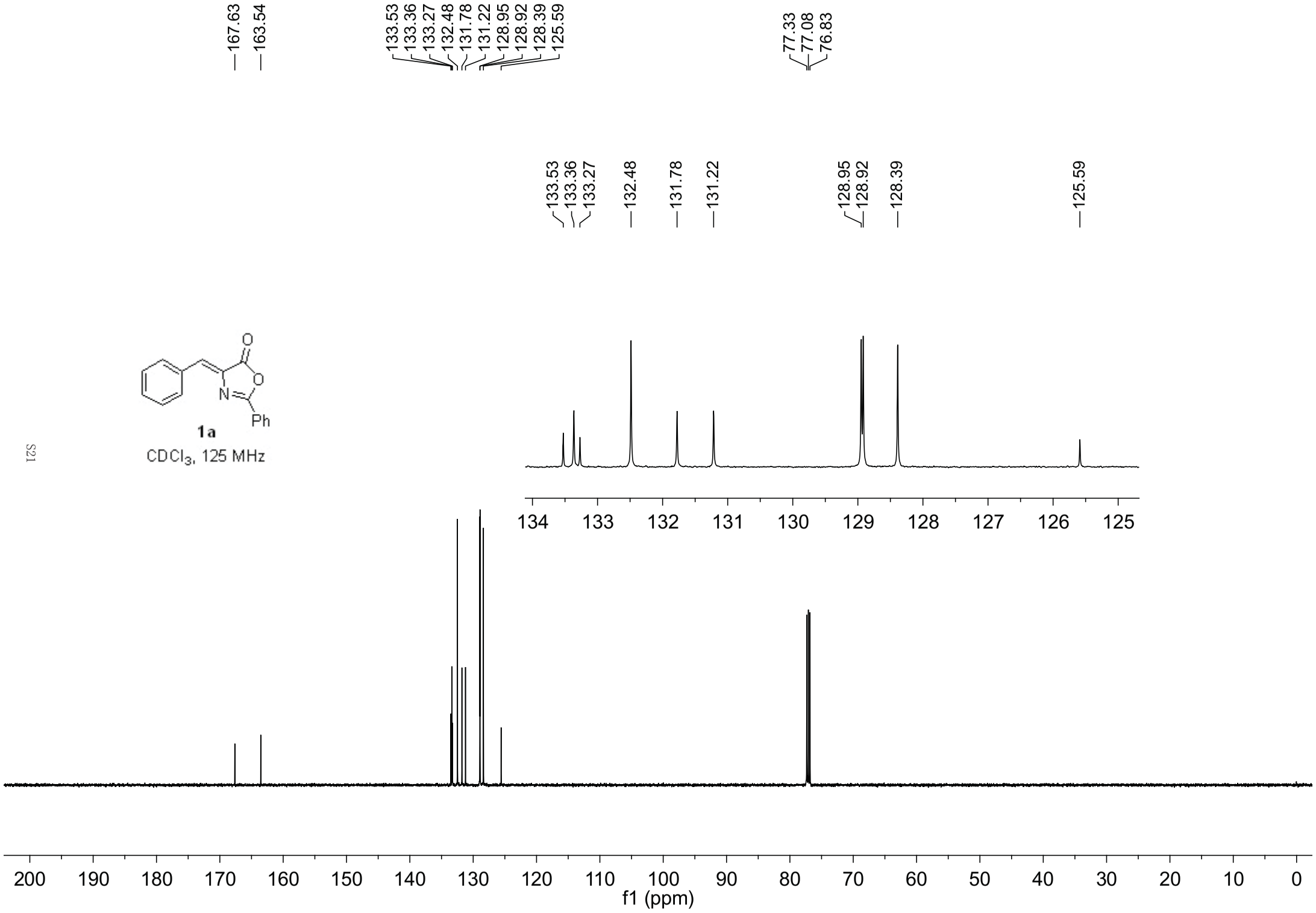
8.1923
8.1891
8.1837
8.1812
8.1783
8.1711
8.1670
7.5108
7.4626
7.4504
7.2602
7.2336
8.2047
8.1956
8.1923
8.1891
8.1837
8.1812
8.1783
8.1711
8.1670
8.1641
7.6253
7.6228
7.6202
7.6121
7.6080
7.6041
7.5957
7.5931
7.5905
7.5411
7.4978
7.4855
7.4592
7.4487
7.4348
7.2602
7.2336
O
O
N
Ph
1a
CDCl3, 500 MHz
S20
4.00
1.03
5.09
0.99
8.3
8.2
8.1
8.0
7.9
7.8
7.7
7.6
7.5
7.4
7.3
7.2
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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133.53
133.36
133.27
132.48
131.78
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128.92
128.39
125.59
77.33
77.08
76.83
O
O
N
Ph
1a
CDCl3, 125 MHz
134
133
132
131
130
129
128
127
126
125
200
190
180
170
160
150
140
130
120
110
100
90
80
70
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50
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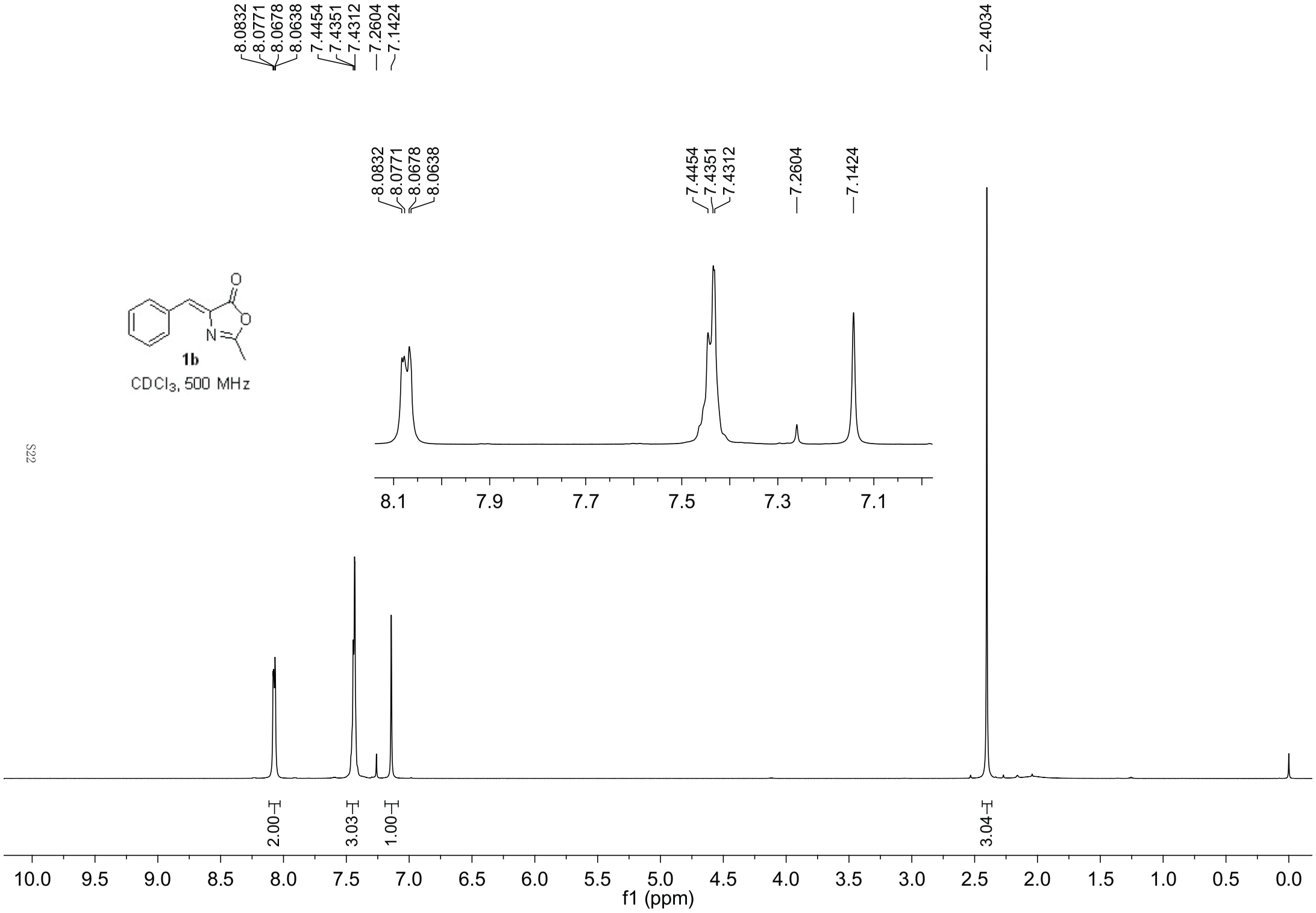
30
20
10
0
f1 (ppm)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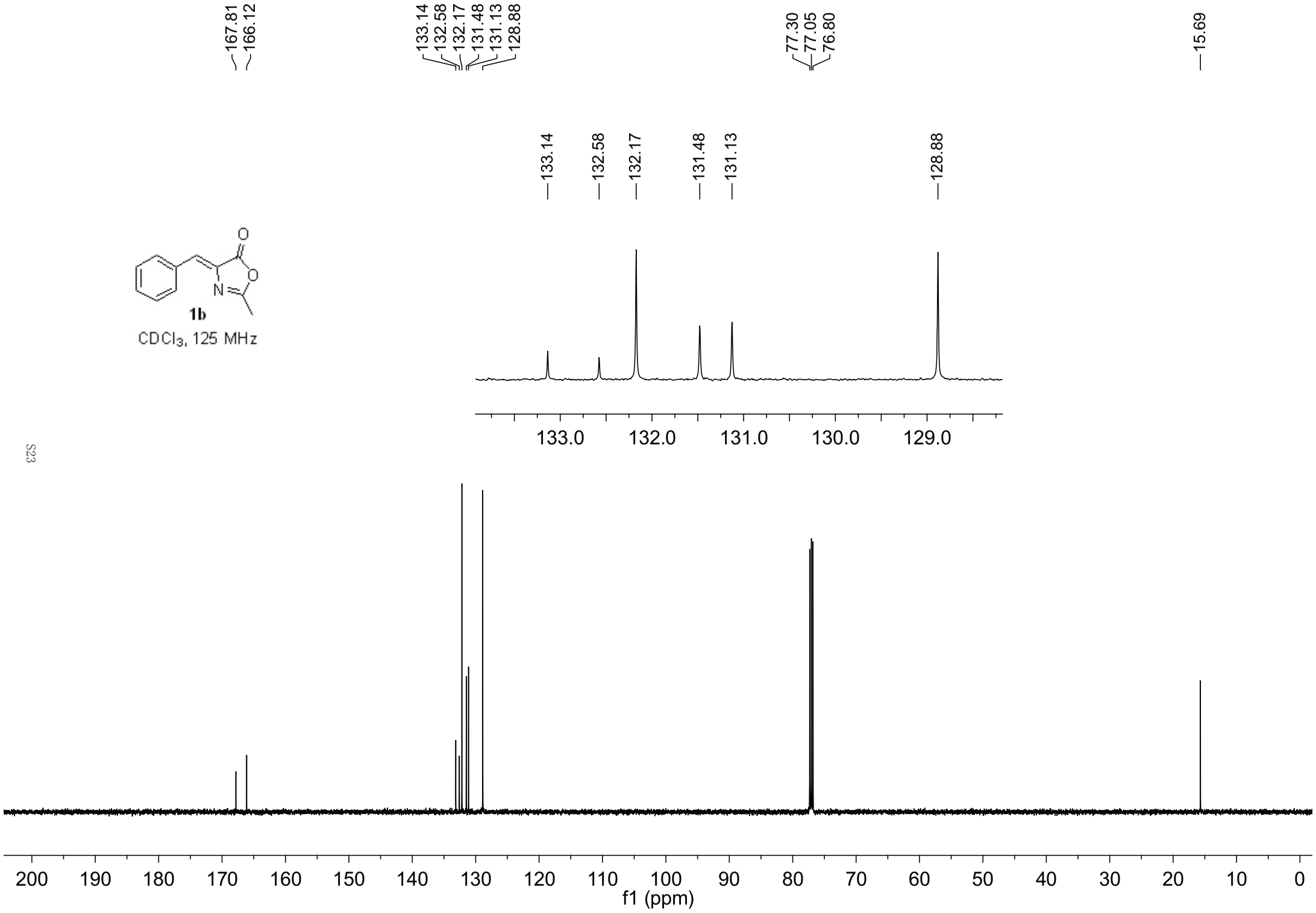
3.03
1.00
3.04
f1 (ppm)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132.58
132.17
131.48
131.13
128.88
77.30
77.05
76.80
15.69
O
O
N
1b
CDCl3, 125 MHz
133.0
132.0
131.0
130.0
129.0
200
190
180
170
160
150
140
130
120
110
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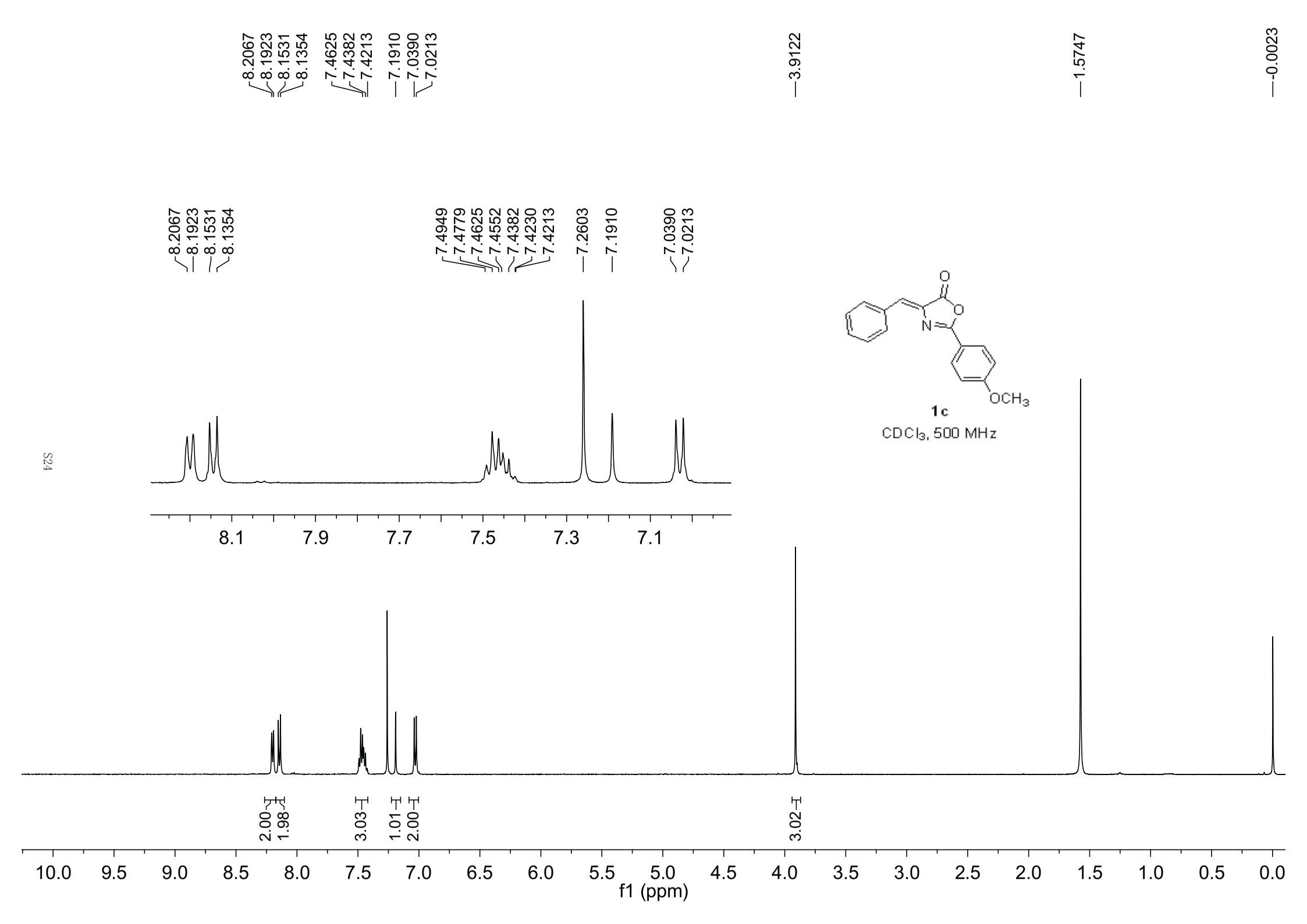
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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8.1354
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3.9122
1.5747
-0.0023
7.4949
7.4779
7.4552
7.4230
7.2603
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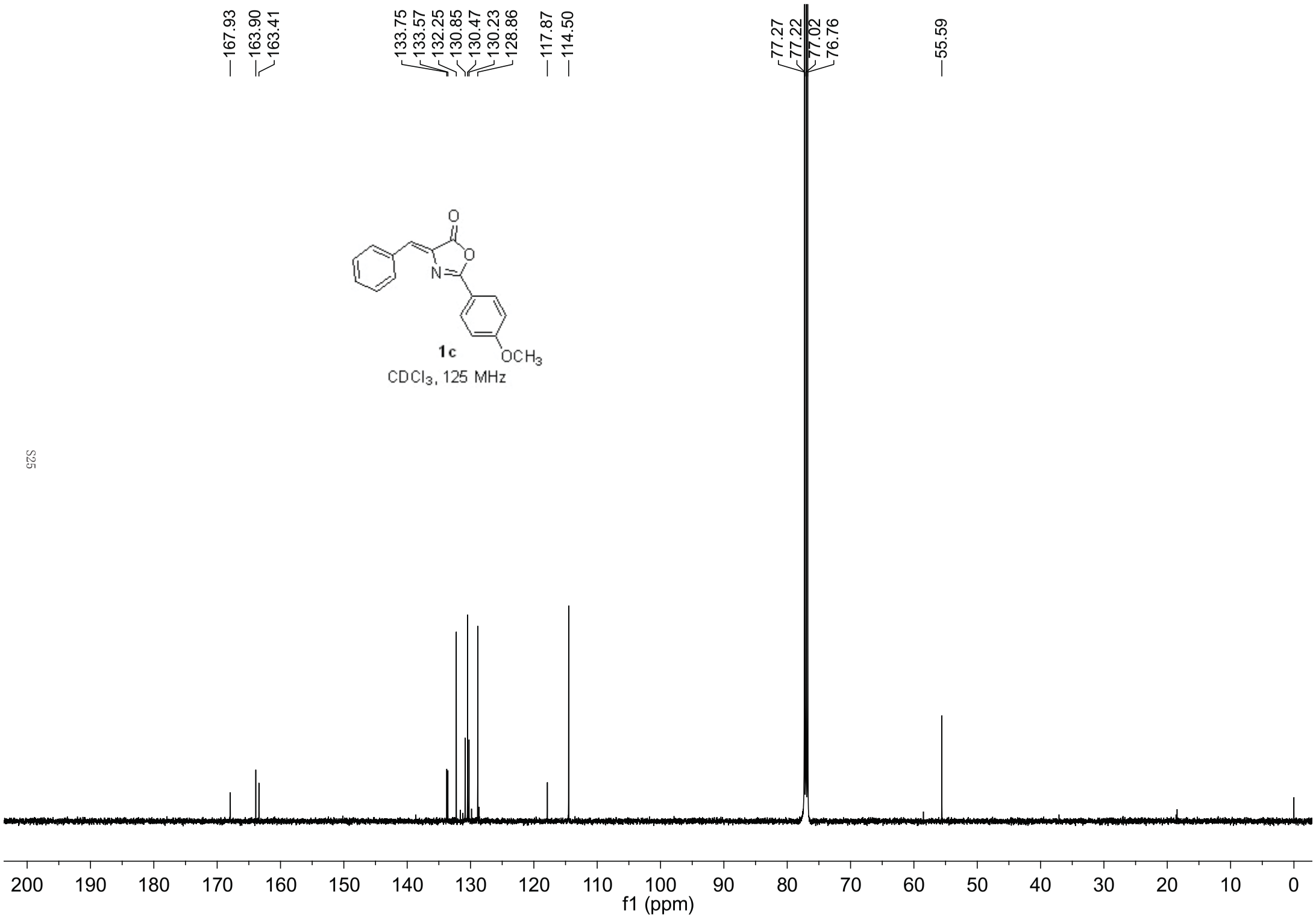
N
OCH3
1c
CDCl3, 500 MHz
2.00
1.98
3.03
1.01
2.00
3.02
f1 (ppm)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130.47
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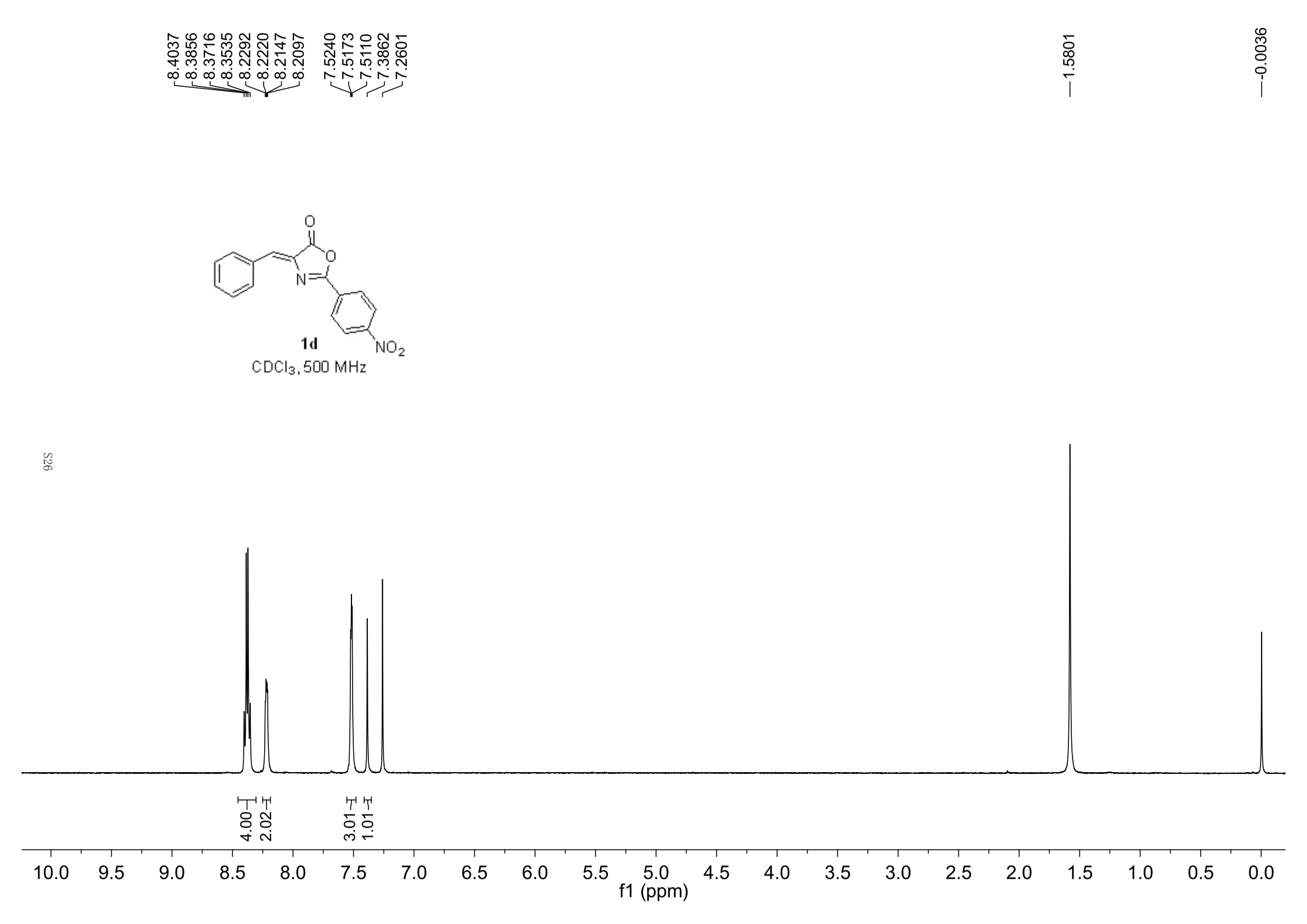
117.87
114.50
77.27
77.22
77.02
76.76
55.59
O
O
N
OCH3
1c
CDCl3, 125 MHz
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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8.3535
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8.2220
8.2147
8.2097
7.5240
7.5173
7.5110
7.3862
7.2601
1.5801
-0.0036
O
O
N
NO2
1d
CDCl3, 500 MHz
S26
4.00
2.02
3.01
1.01
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
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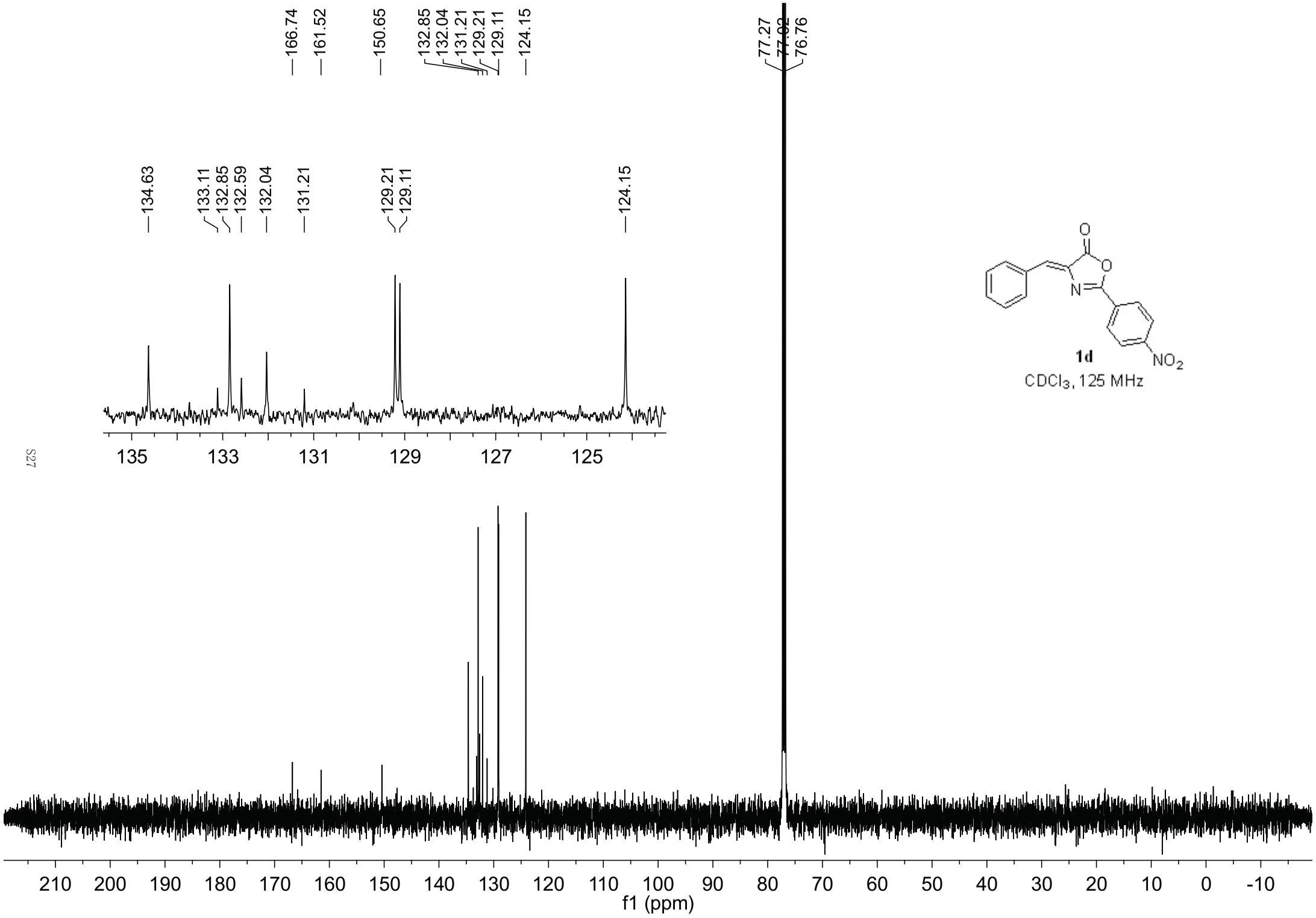
0.0
f1 (ppm)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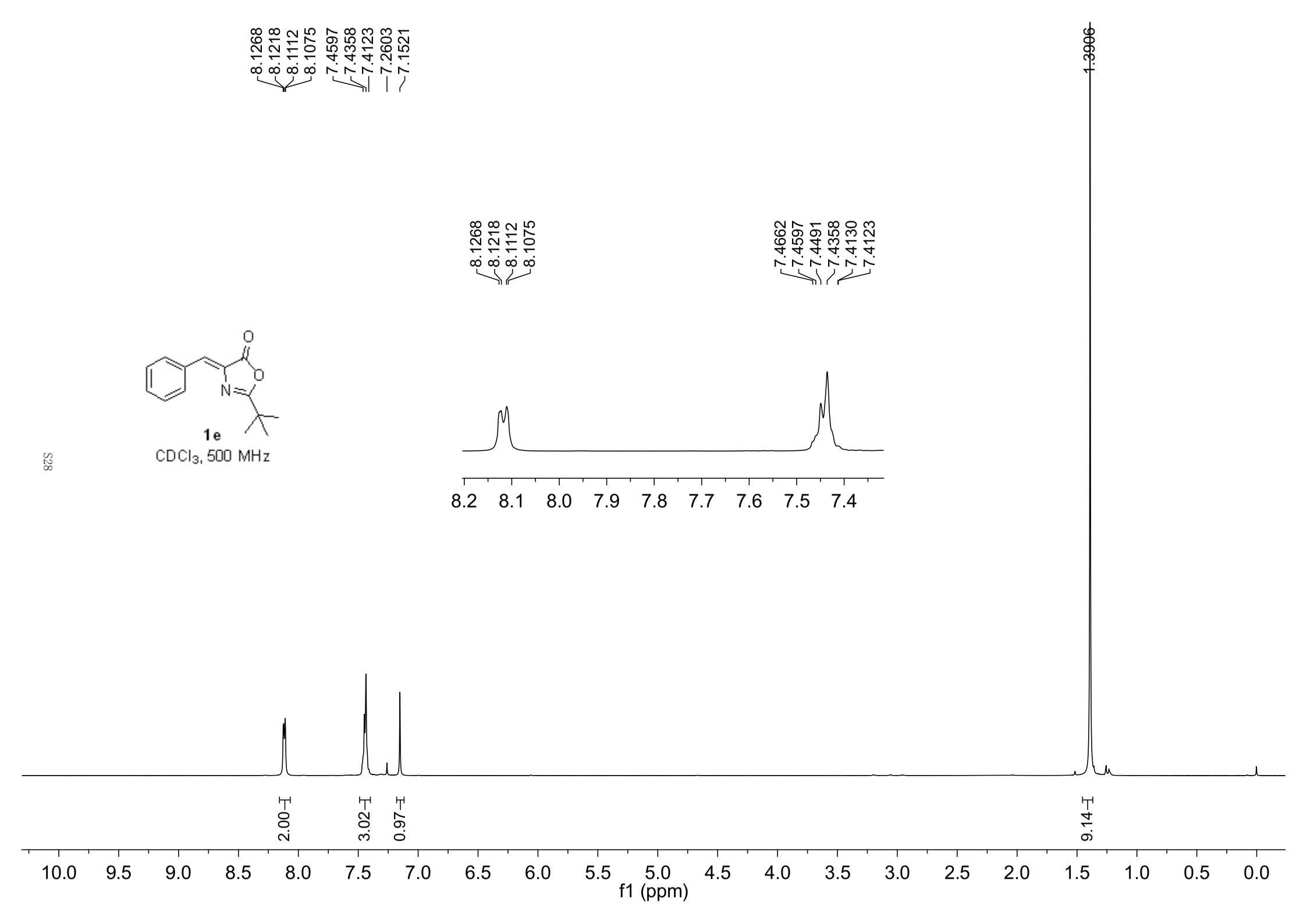
129.21
129.11
124.15
77.27
77.02
76.76
134.63
133.11
132.85
132.59
132.04
131.21
129.21
129.11
124.15
135
133
131
129
127
125
O
O
N
1d
NO2
CDCl3, 125 MHz
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)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7.1521
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8.1218
8.1112
8.1075
7.4662
7.4597
7.4491
7.4358
7.4130
7.4123
O
O
N
1e
CDCl3, 500 MHz
8.2
8.1
8.0
7.9
7.8
7.7
7.6
7.5
7.4
2.00
3.02
0.97
9.14
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
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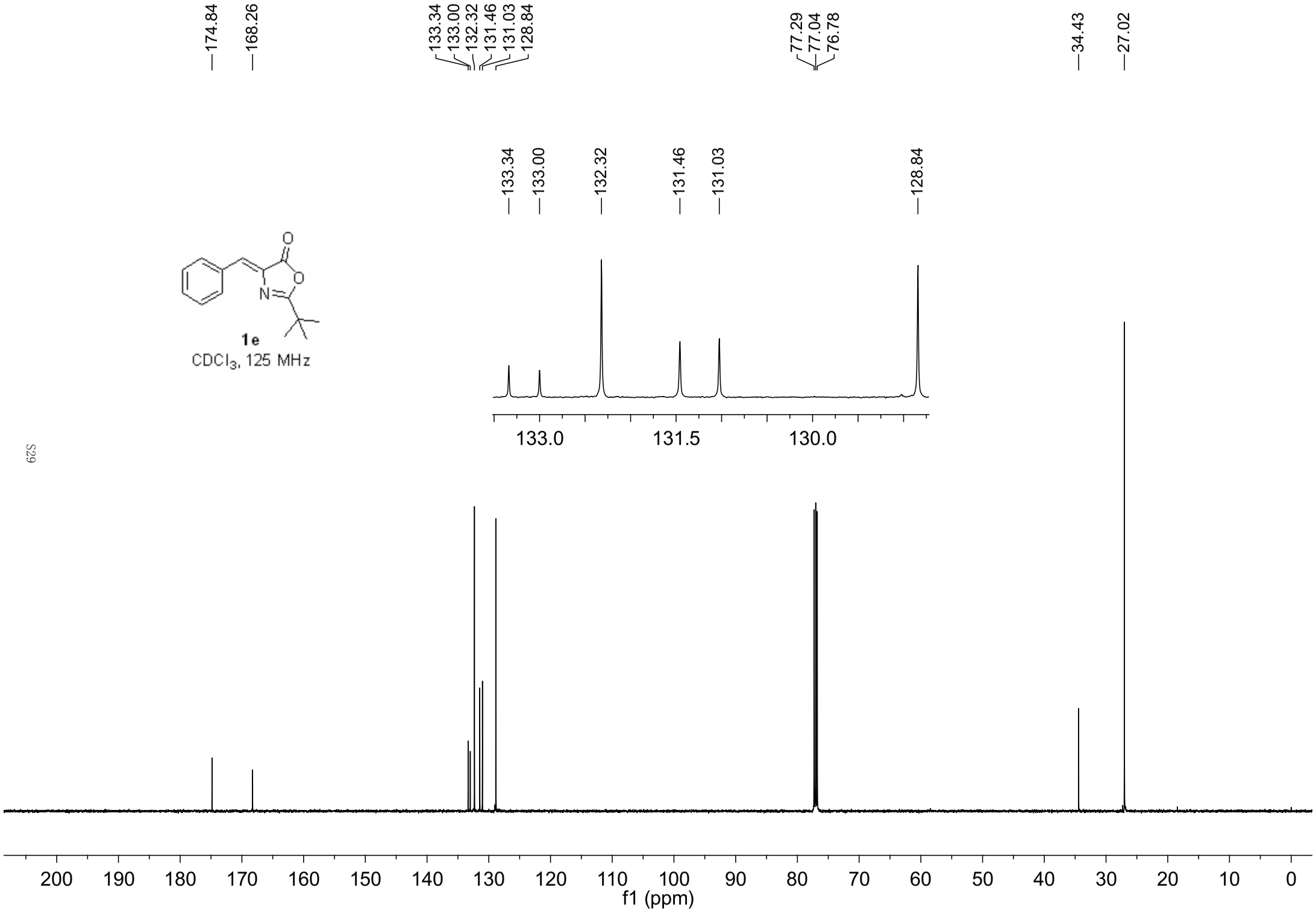
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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133.34
133.00
132.32
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131.03
128.84
77.29
77.04
76.78
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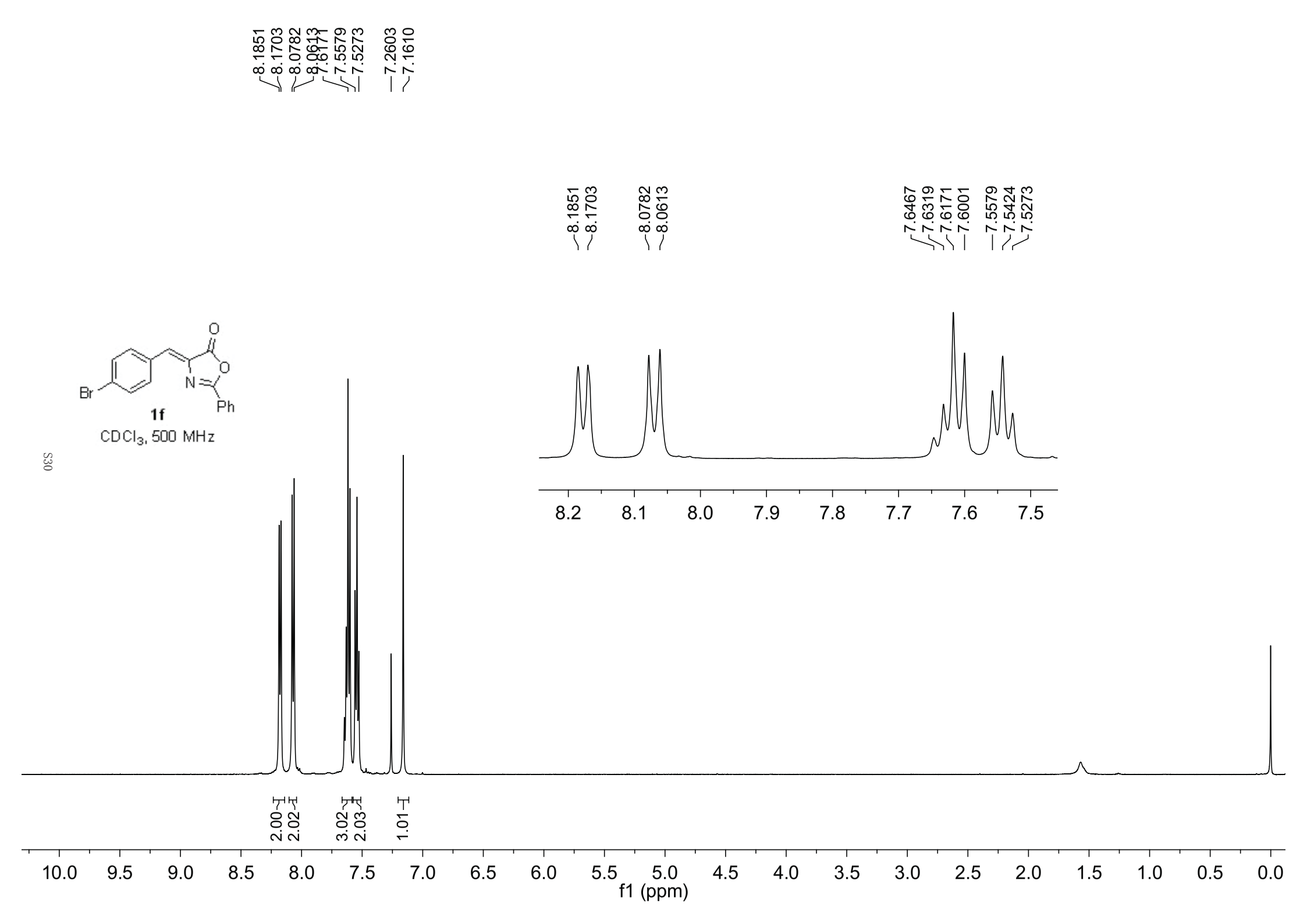
27.02
O
O
N
1e
CDCl3, 125 MHz
133.0
131.5
130.0
f1 (ppm)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7.5273
7.2603
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8.1703
8.0782
8.0613
7.6467
7.6319
7.6171
7.6001
7.5579
7.5424
7.5273
O
O
N
Br
Ph
1f
CDCl3, 500 MHz
S30
8.2
8.1
8.0
7.9
7.8
7.7
7.6
7.5
2.00
2.02
3.02
2.03
1.01
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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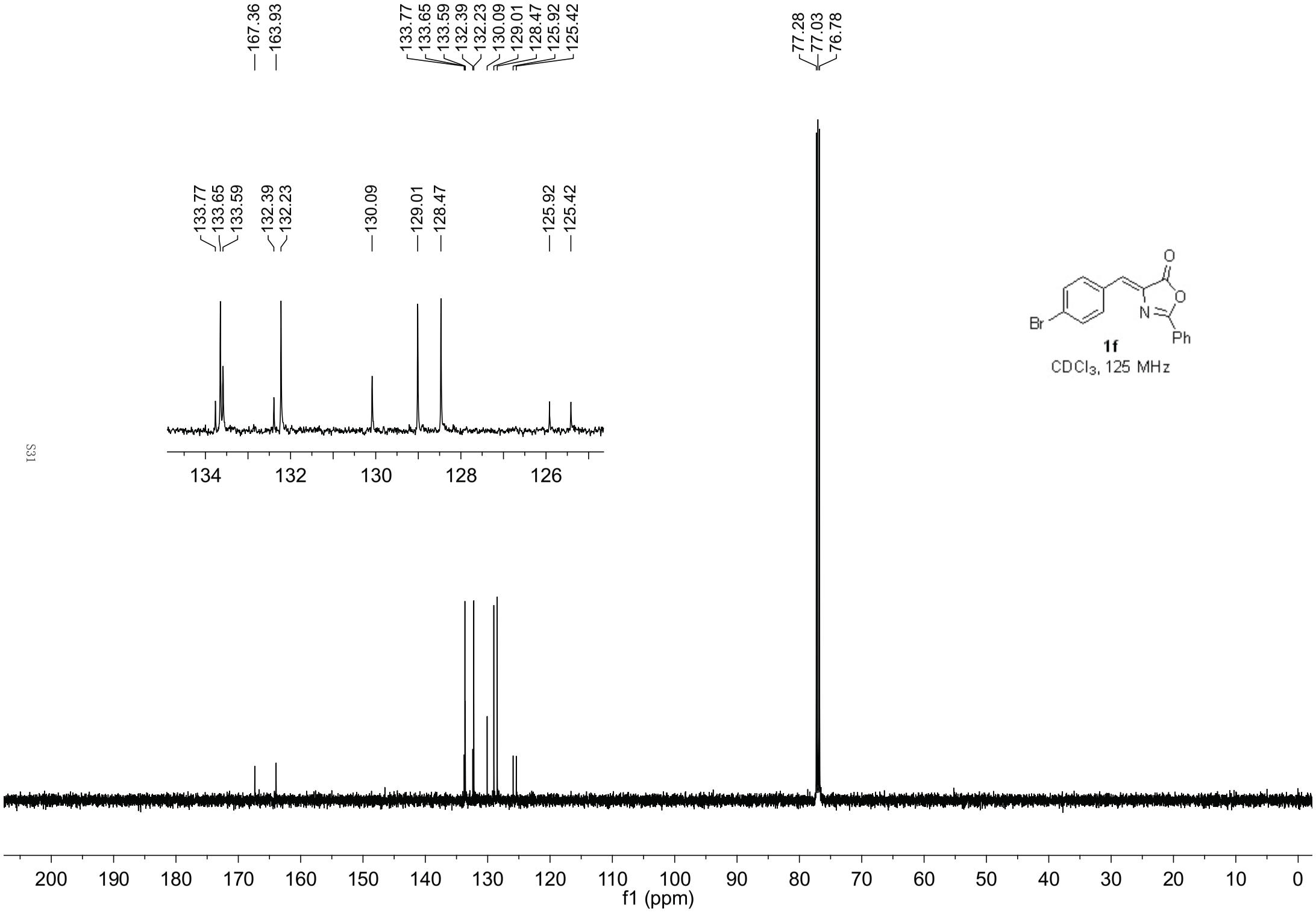
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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130.09
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128.47
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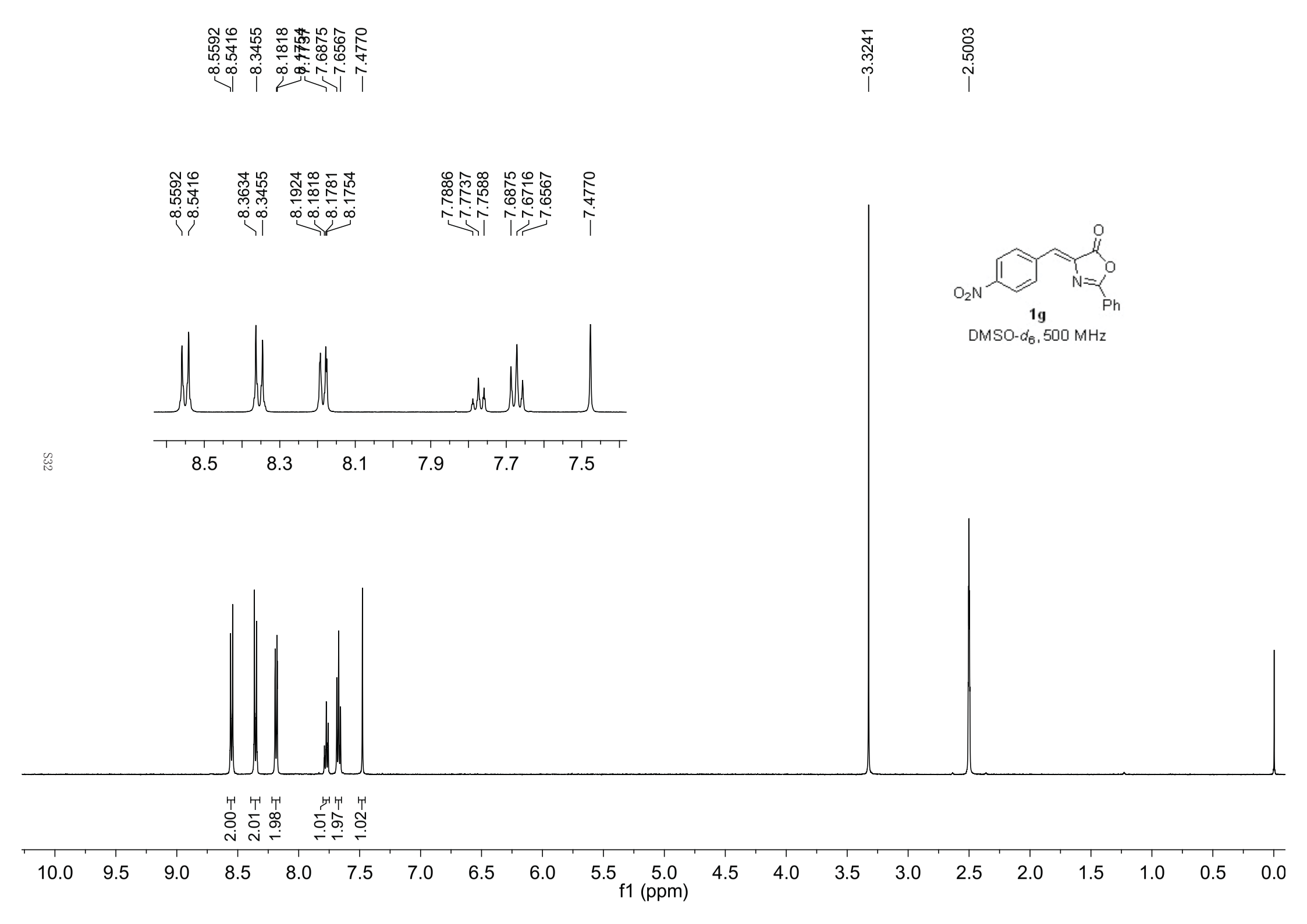
77.28
77.03
76.78
Br
O
O
N
Ph
1f
CDCl3, 125 MHz
f1 (ppm)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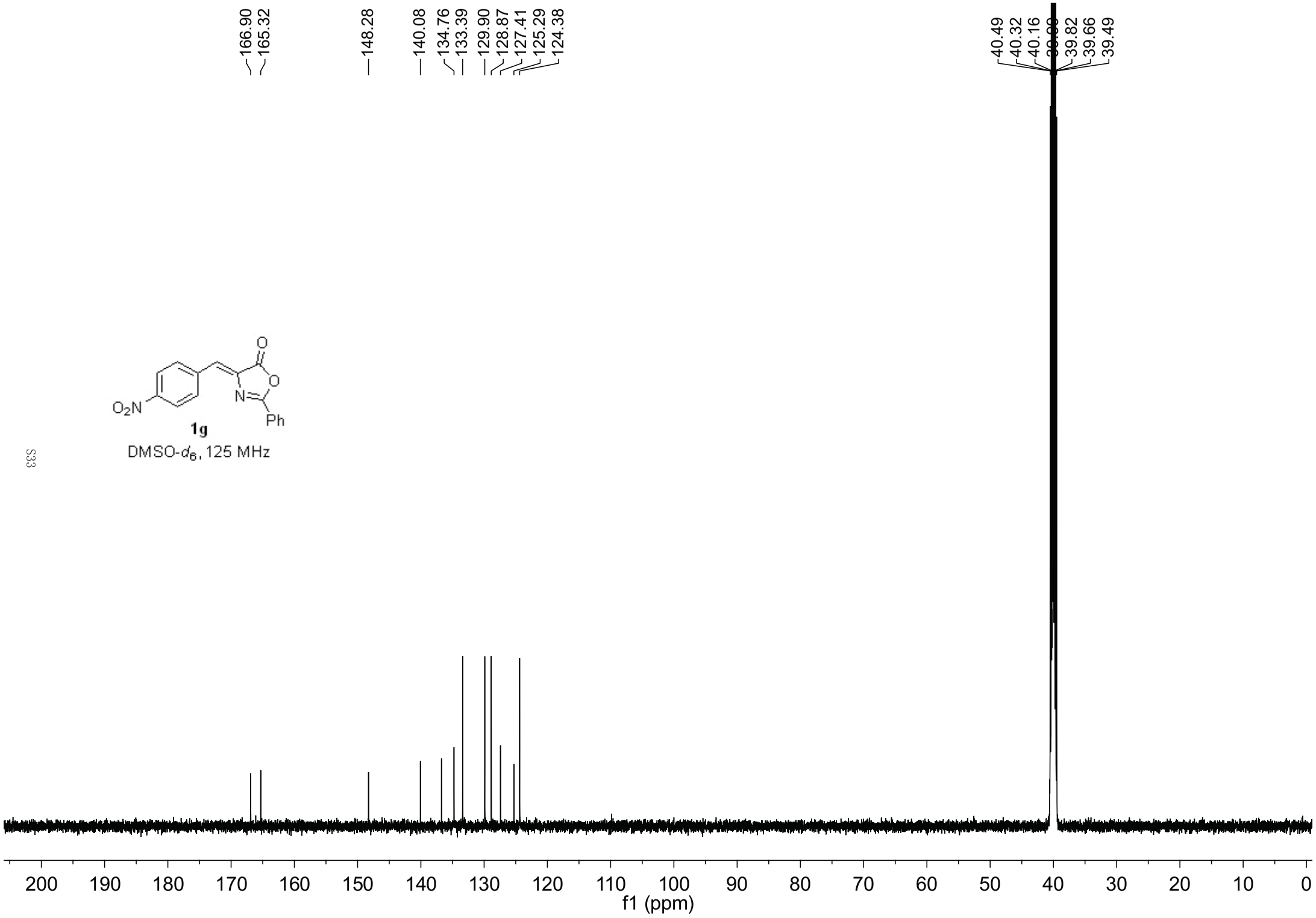
8.5416
8.3634
8.3455
8.1924
8.1818
8.1781
8.1754
7.7886
7.7737
7.7588
7.6875
7.6716
7.6567
7.4770
O
O
N
O2N
Ph
1g
DMSO-d6, 500 MHz
S32
2.00
2.01
1.98
1.01
1.97
1.02
f1 (ppm)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128.87
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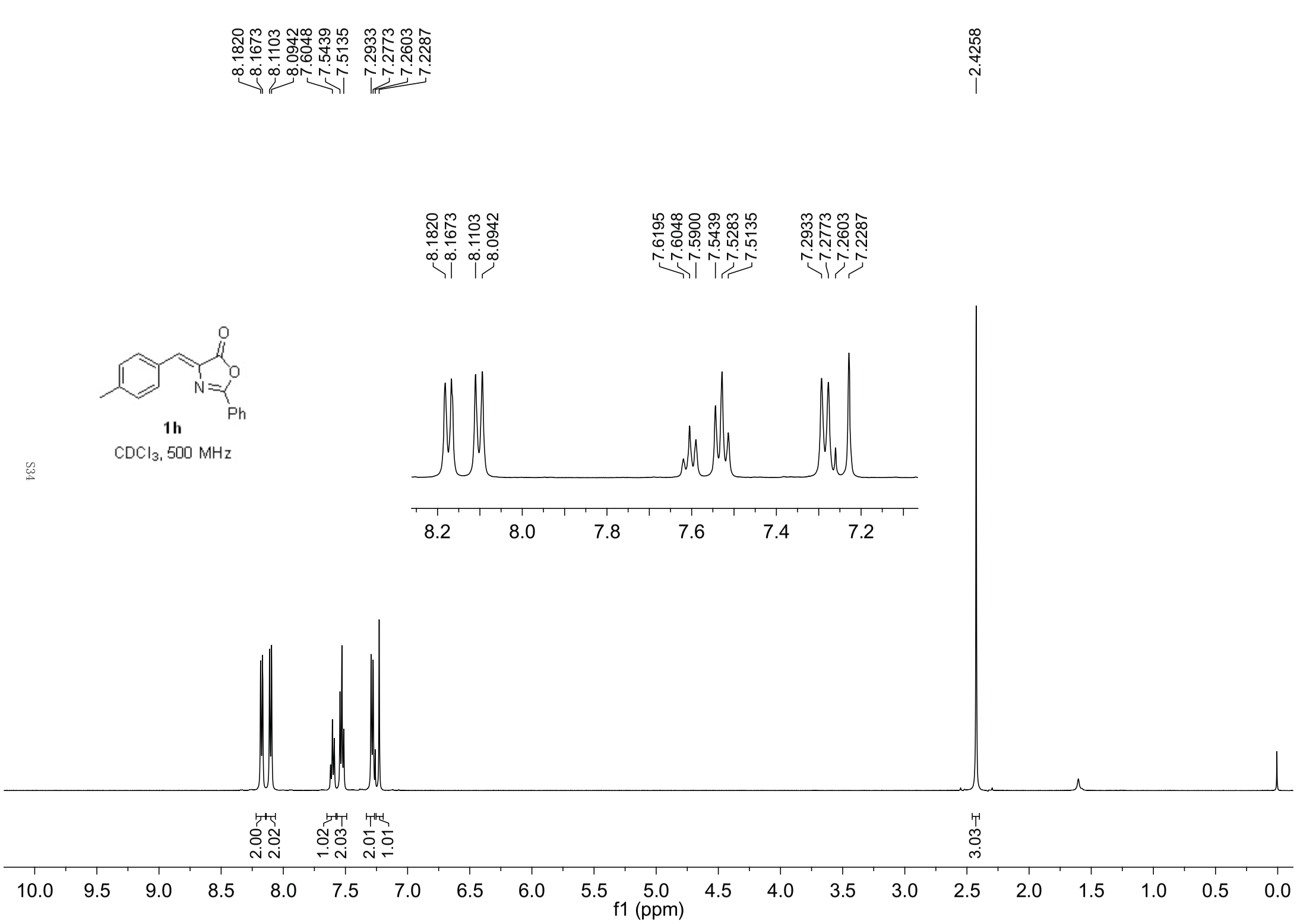
125.29
124.38
40.49
40.32
40.16
39.82
39.66
39.49
O
O
N
O2N
Ph
1g
DMSO-d6, 125 MHz
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.5135
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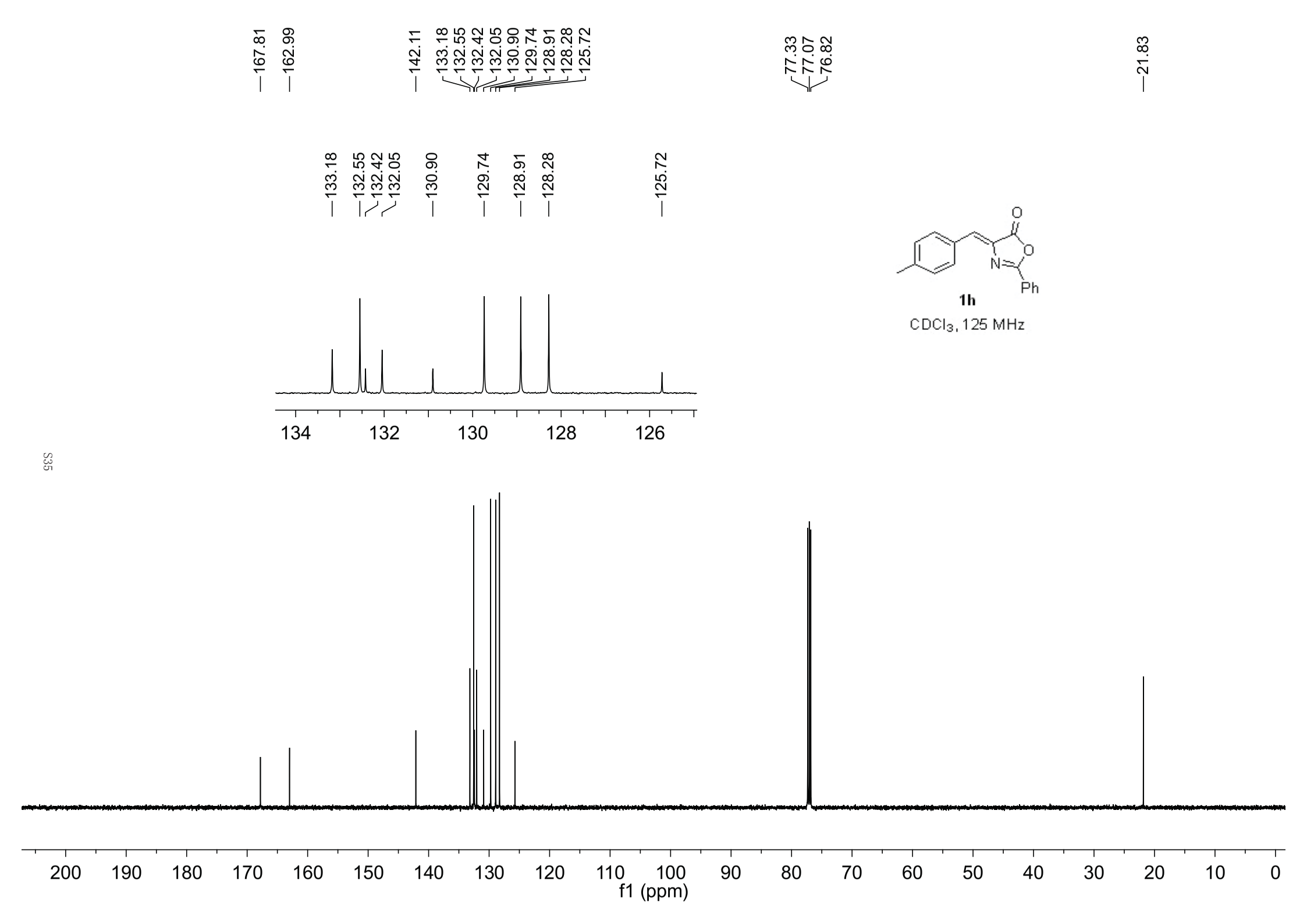
7.2773
7.2603
7.2287
2.4258
8.1820
8.1673
8.1103
8.0942
7.6195
7.6048
7.5900
7.5439
7.5283
7.5135
7.2933
7.2773
7.2603
7.2287
O
O
N
Ph
1h
CDCl3, 500 MHz
8.2
8.0
7.8
7.6
7.4
7.2
2.00
2.02
1.02
2.03
2.01
1.01
3.03
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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129.74
128.91
128.28
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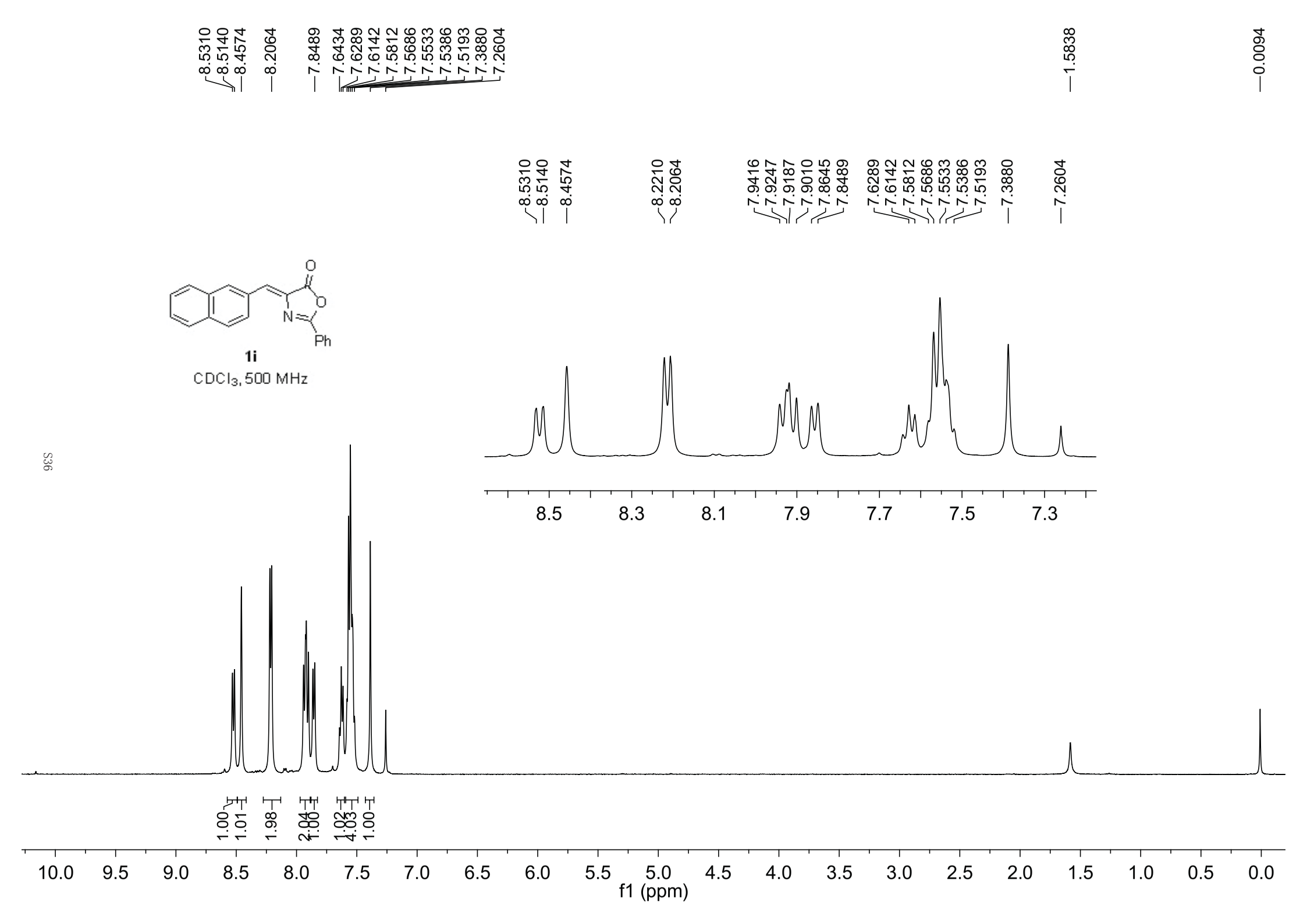
77.33
77.07
76.82
21.83
O
O
N
Ph
1h
CDCl3, 125 MHz
f1 (ppm)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7.6289
7.6142
7.5812
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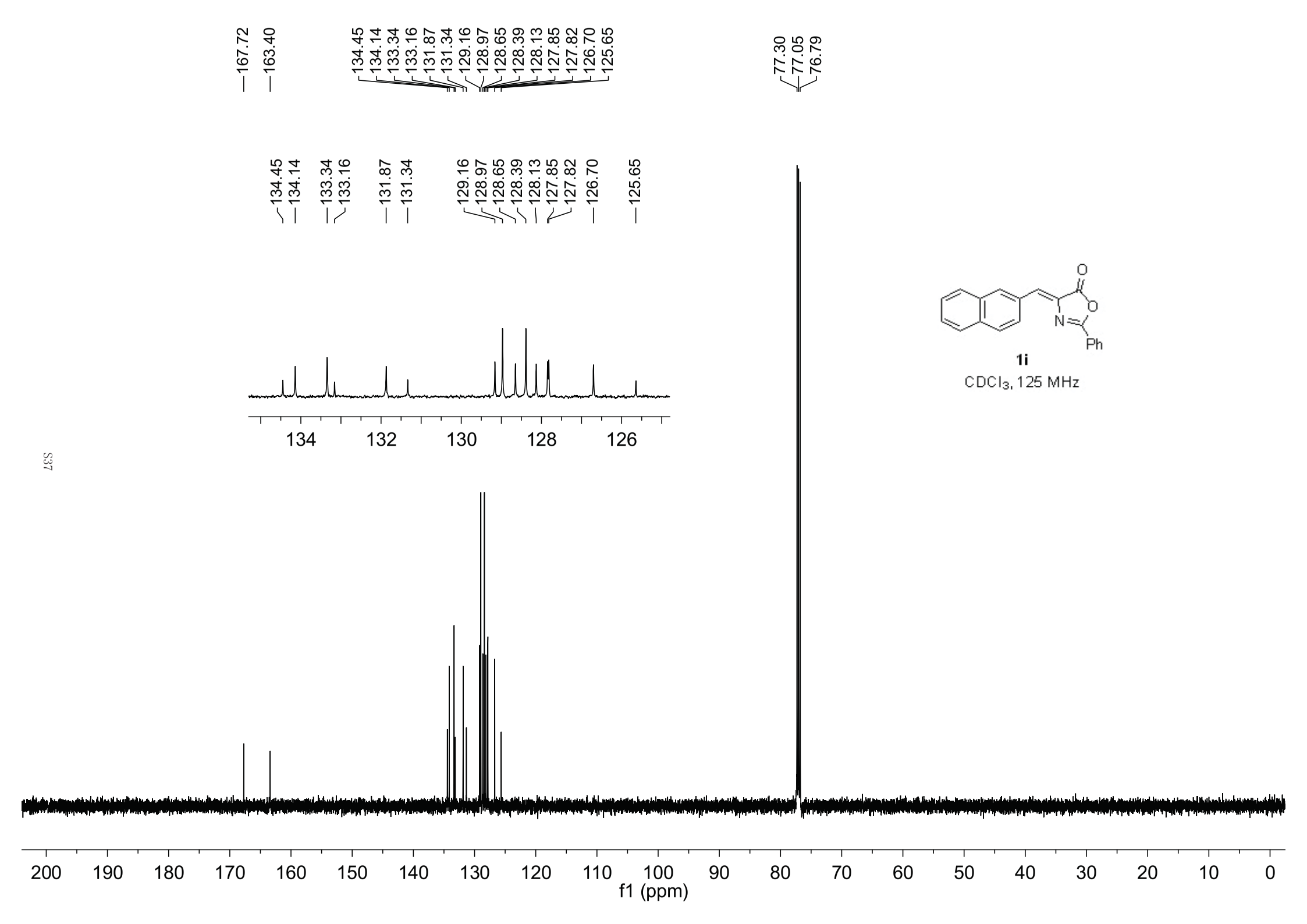
7.5533
7.5386
7.5193
7.3880
7.2604
1.5838
0.0094
8.5310
8.5140
8.4574
8.2210
8.2064
7.9416
7.9247
7.9187
7.9010
7.8645
7.8489
7.6289
7.6142
7.5812
7.5686
7.5533
7.5386
7.5193
7.3880
7.2604
O
O
N
Ph
1i
CDCl3, 500 MHz
S36
8.5
8.3
8.1
7.9
7.7
7.5
7.3
1.00
1.01
1.98
2.04
1.00
1.02
4.03
1.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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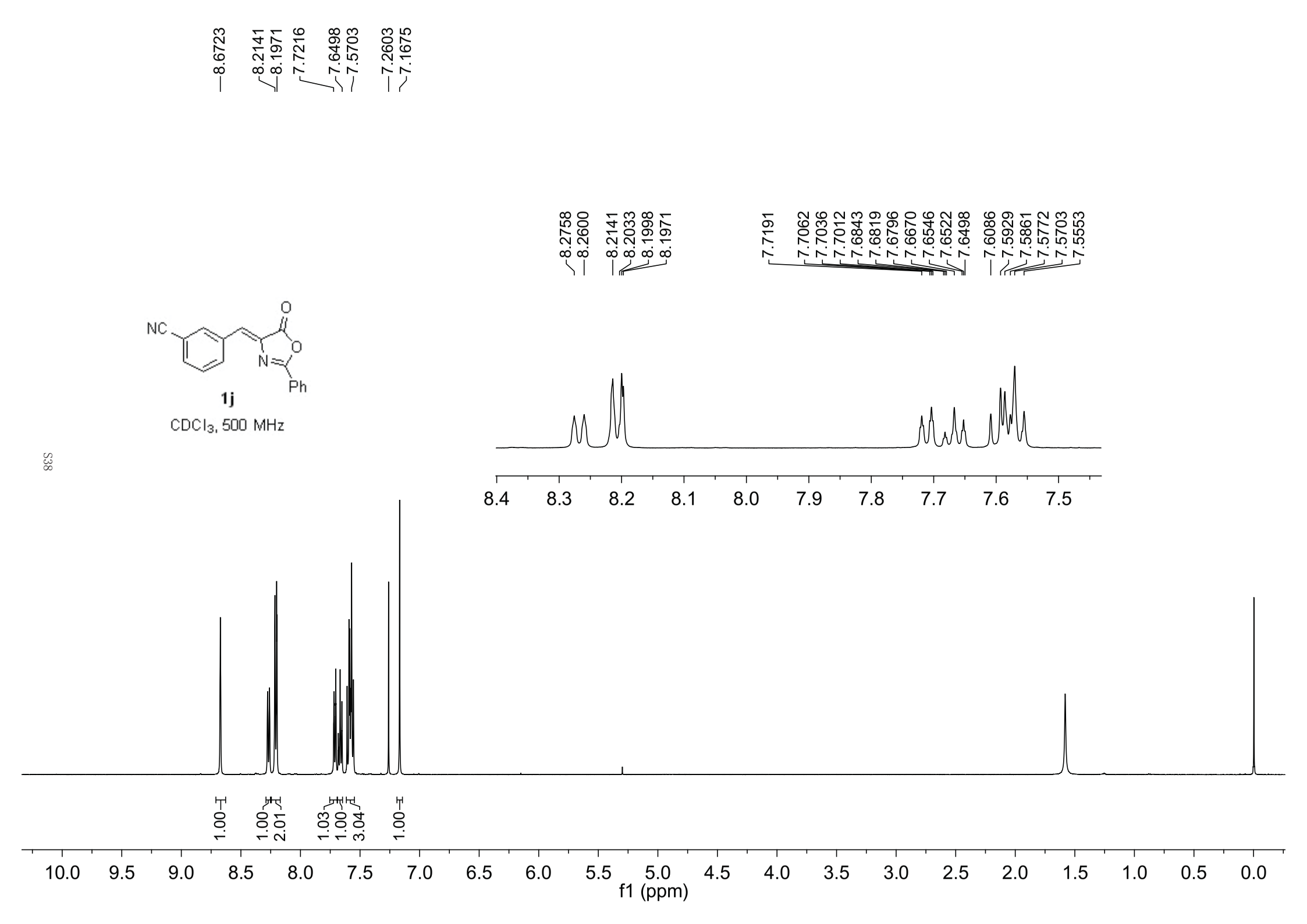
131.87
131.34
129.16
128.97
128.65
128.39
128.13
127.85
127.82
126.70
125.65
77.30
77.05
76.79
O
O
N
Ph
1i
CDCl3, 125 MHz
f1 (ppm)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7.6498
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7.2603
7.1675
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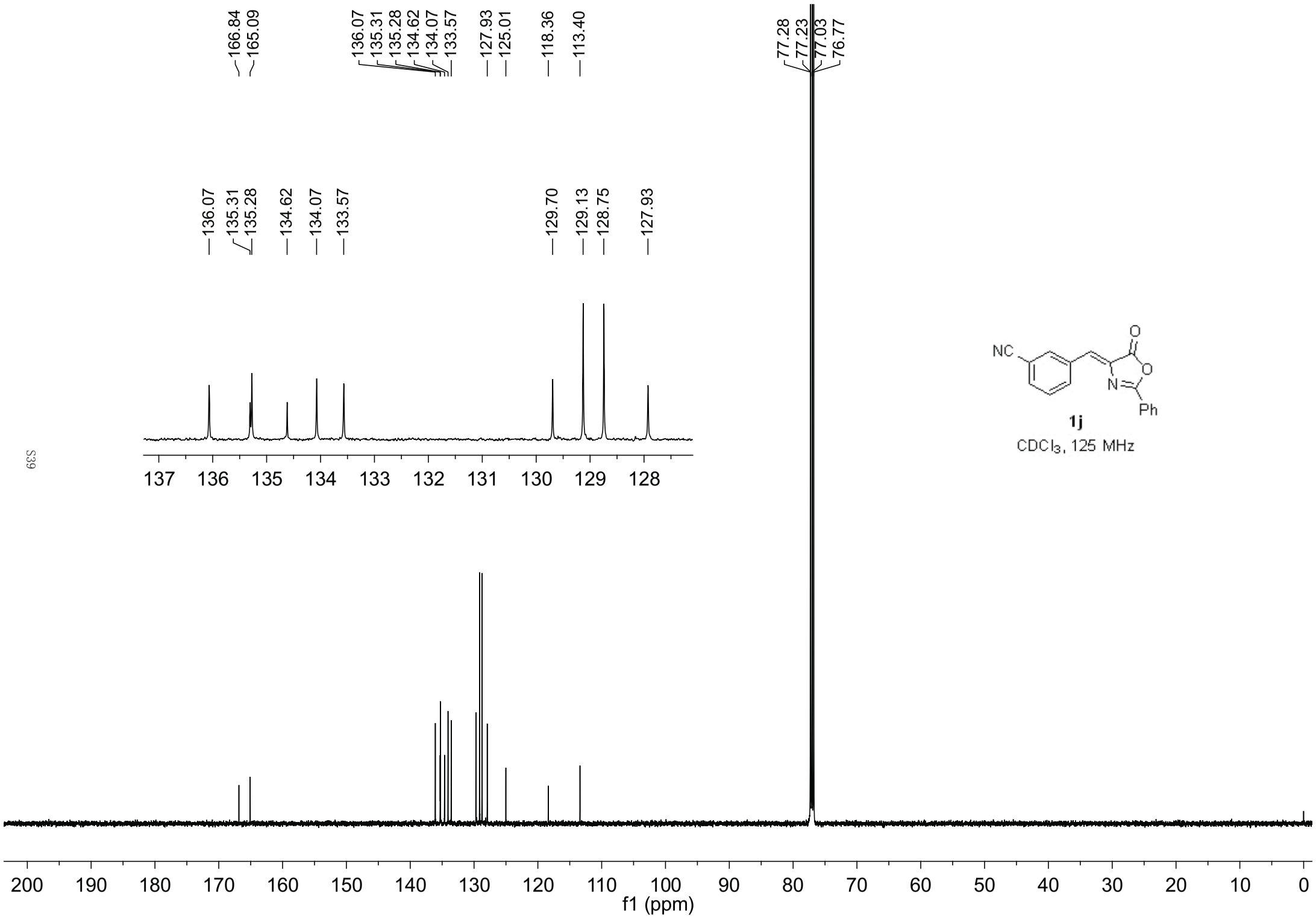
8.2600
8.2141
8.2033
8.1998
8.1971
7.7191
7.7062
7.7036
7.7012
7.6843
7.6819
7.6796
7.6670
7.6546
7.6522
7.6498
7.6086
7.5929
7.5861
7.5772
7.5703
7.5553
NC
O
O
N
Ph
1j
CDCl3, 500 MHz
8.4
8.3
8.2
8.1
8.0
7.9
7.8
7.7
7.6
7.5
1.00
1.00
2.01
1.03
1.00
3.04
1.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


166.84
165.09
136.07
135.31
135.28
134.62
134.07
133.57
127.93
125.01
118.36
113.40
77.28
77.23
77.03
76.77
136.07
135.31
135.28
134.62
134.07
133.57
129.70
129.13
128.75
127.93
137 136 135 134 133 132 131 130 129 128
NC
O
O
N
Ph
1j
CDCl3, 125 MHz
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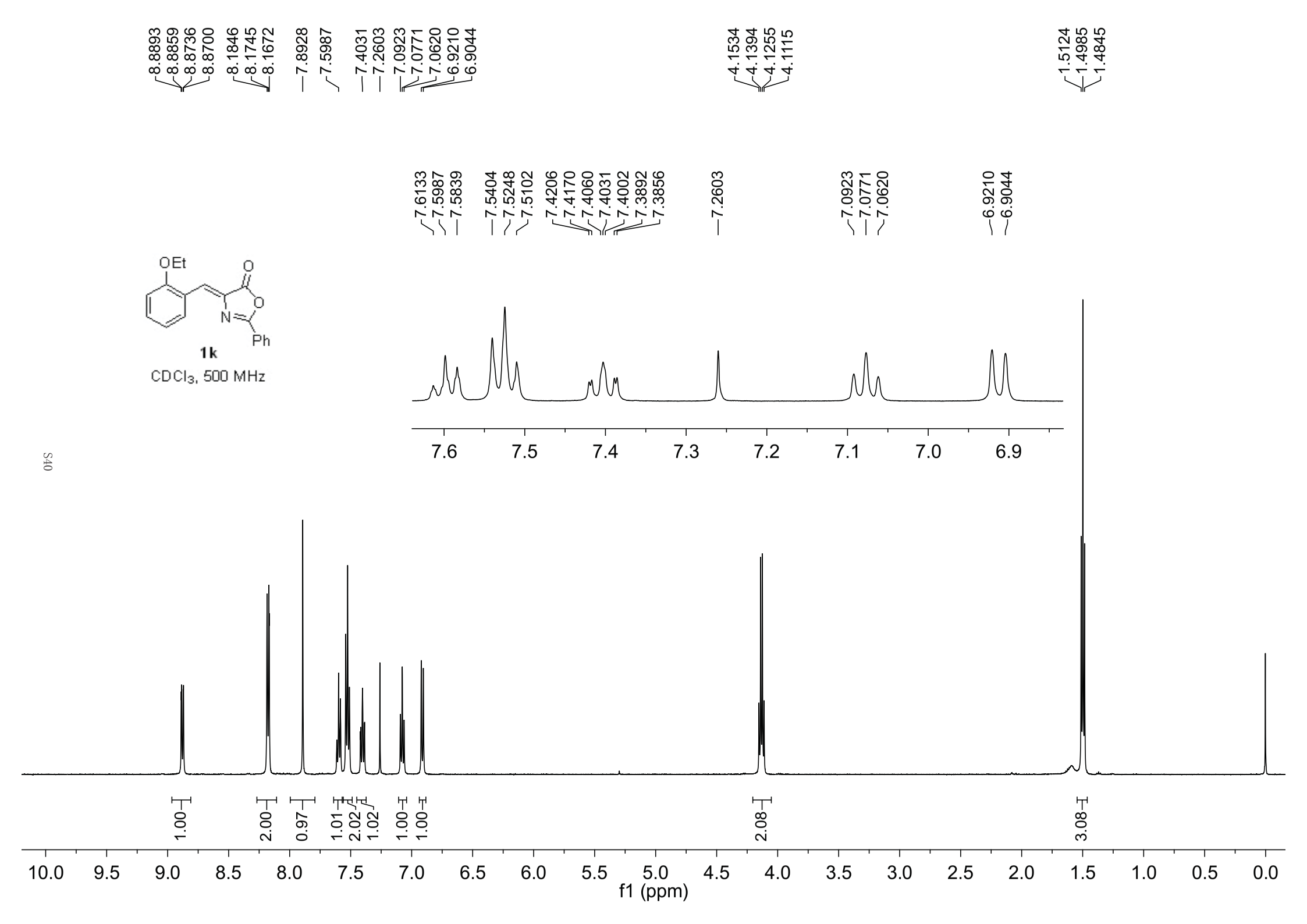
f1 (ppm)


8.8893
8.8859
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8.1745
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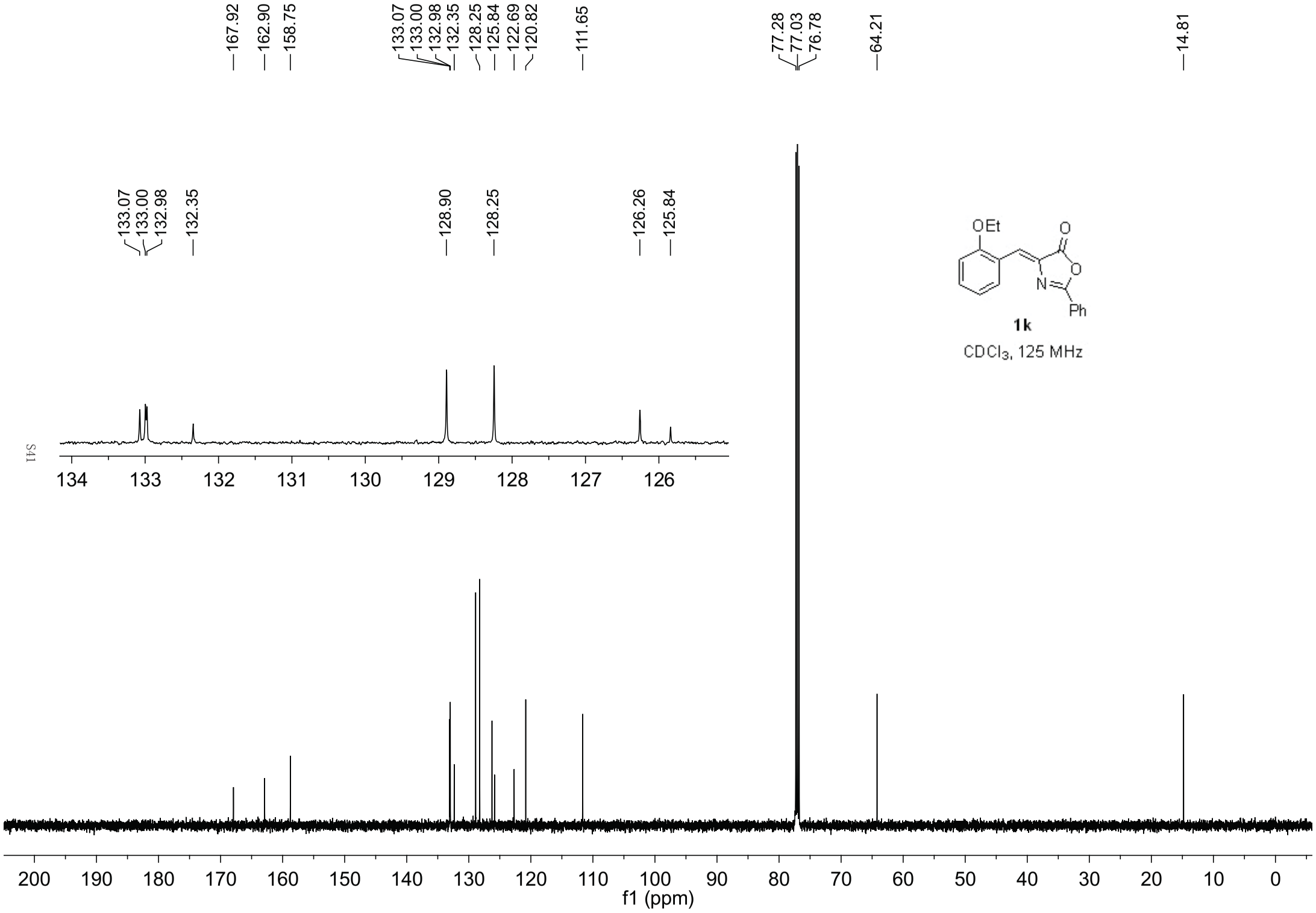
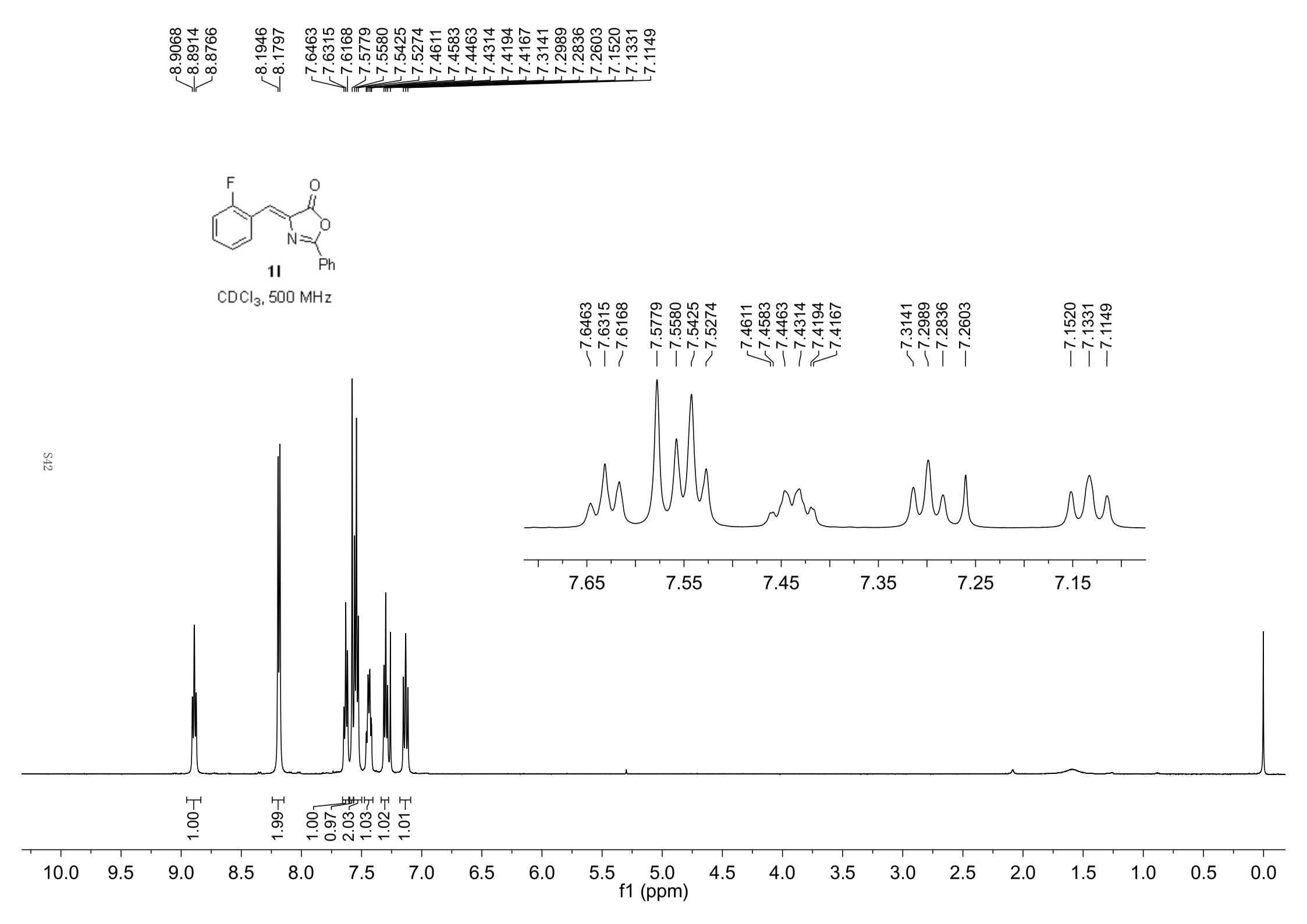
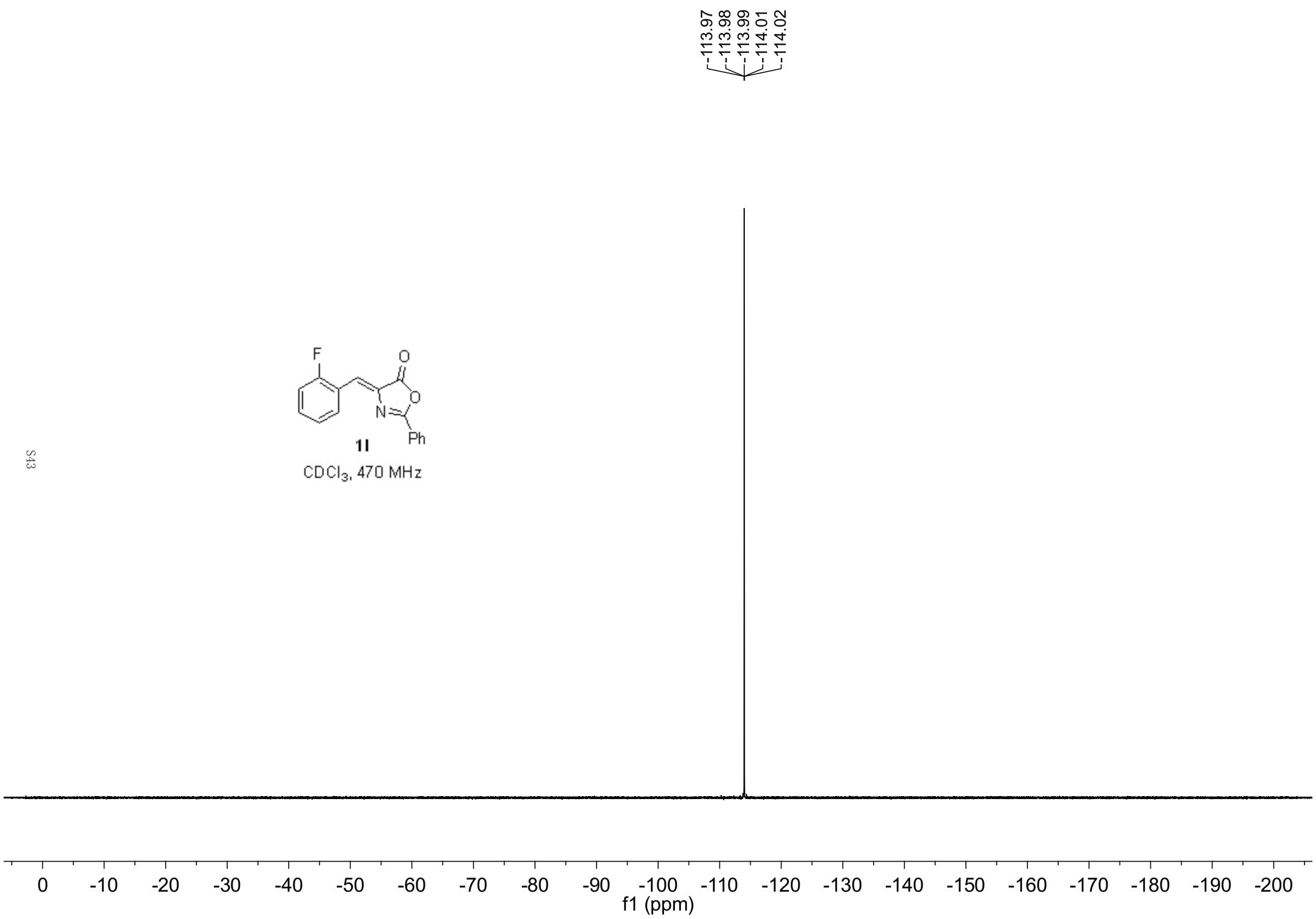
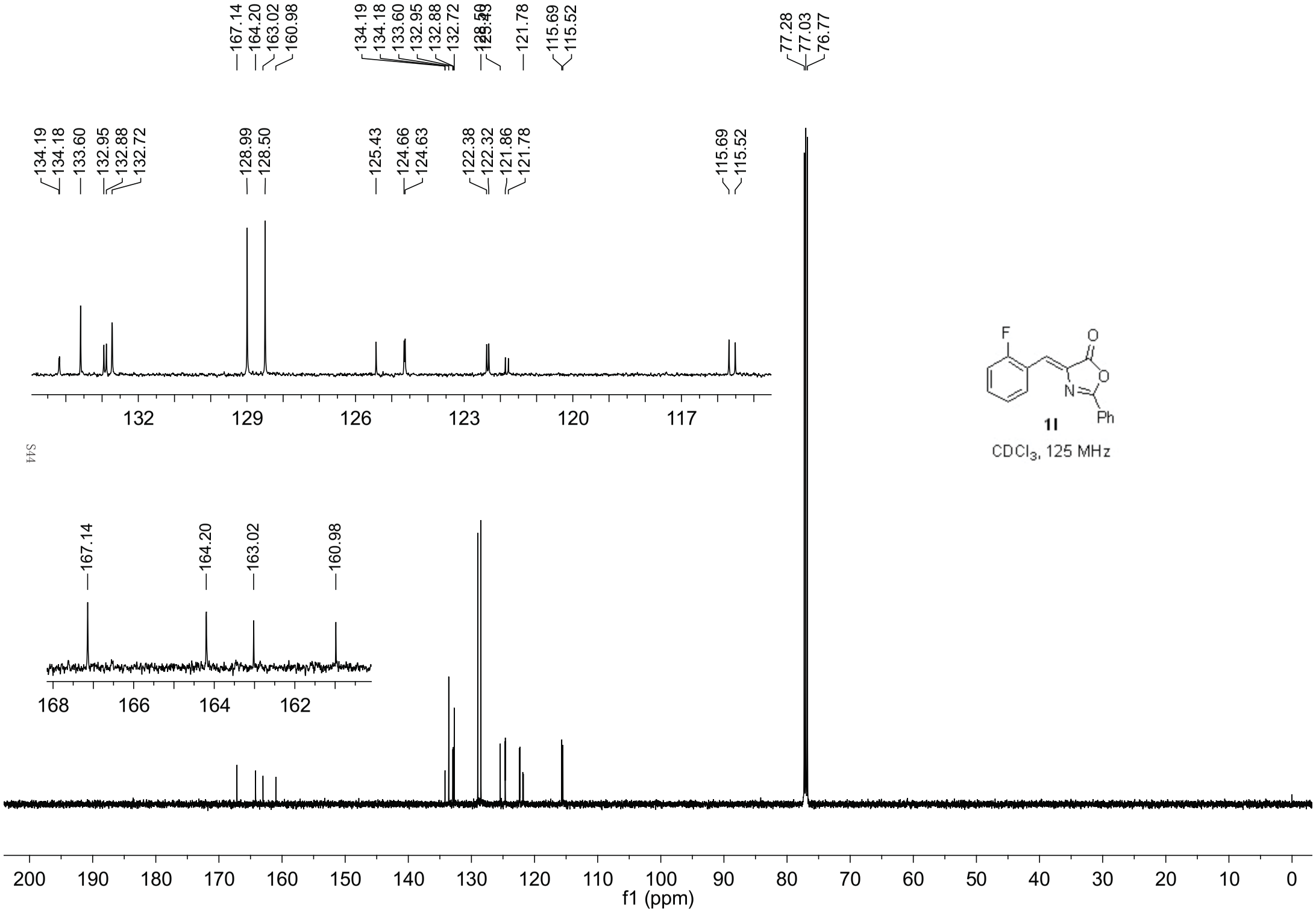
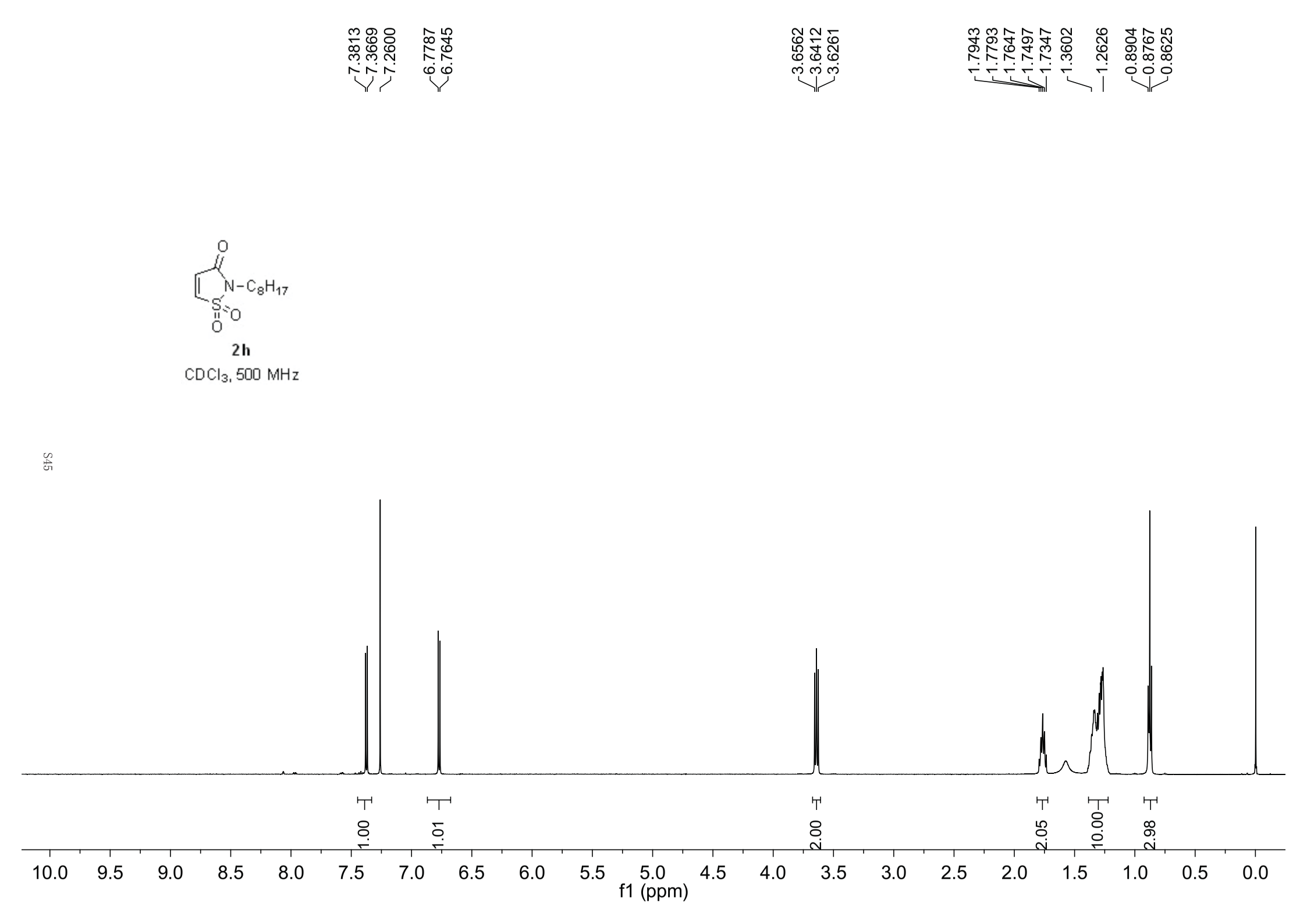
7.4031
7.2603
7.0923
7.0771
7.0620
6.9210
6.9044
4.1534
4.1394
4.1255
4.1115
1.5124
1.4985
1.4845
7.6133
7.5987
7.5839
7.5404
7.5248
7.5102
7.4206
7.4170
7.4060
7.4031
7.4002
7.3892
7.3856
7.2603
7.0923
7.0771
7.0620
6.9210
6.9044
OEt
O
O
N
Ph
1k
CDCl3, 500 MHz
7.6
7.5
7.4
7.3
7.2
7.1
7.0
6.9
1.00
2.00
0.97
1.01
2.02
1.02
1.00
1.00
2.08
3.08
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

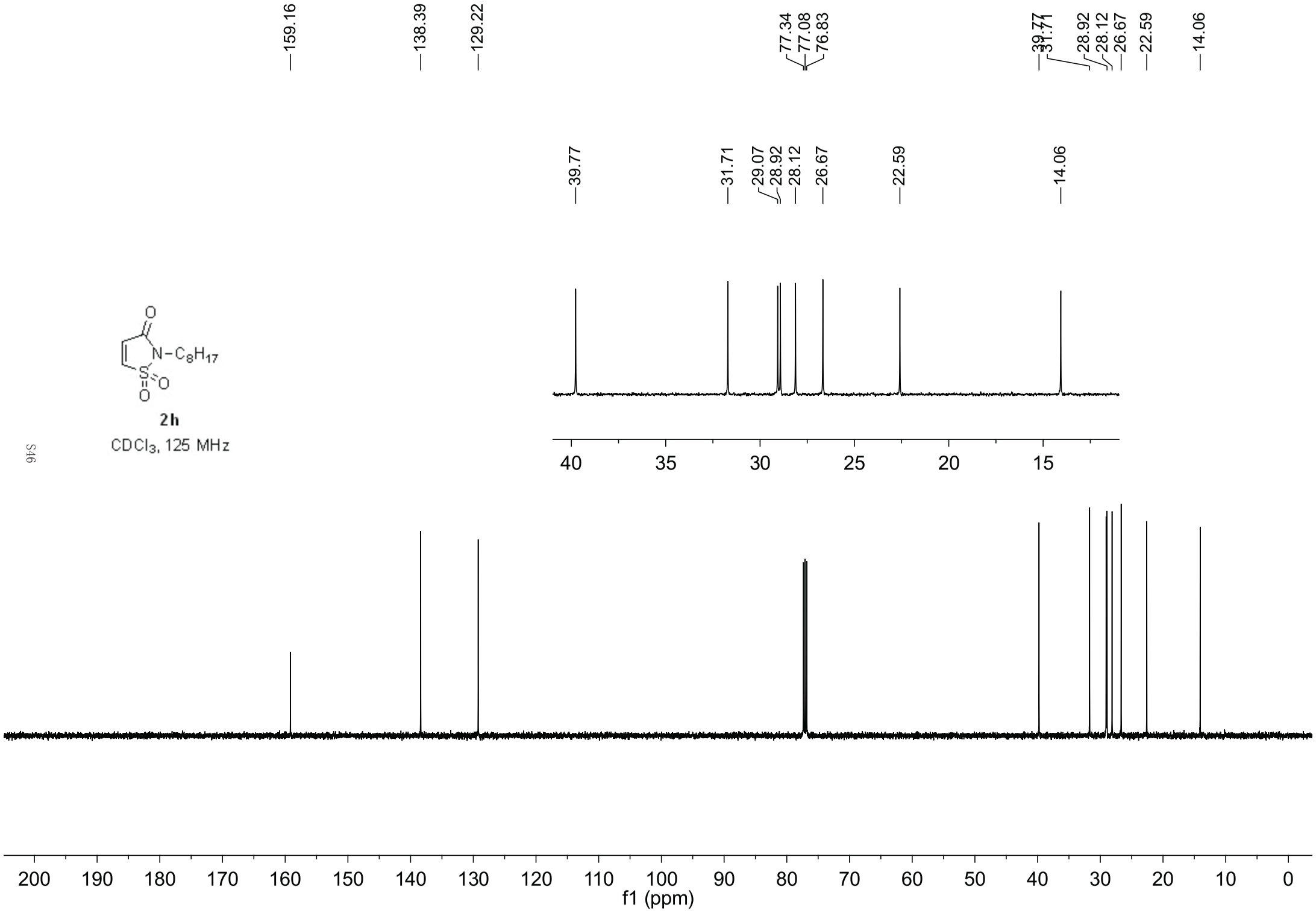

167.92
162.90
158.75
133.07
133.00
132.98
132.35
128.25
125.84
122.69
120.82
111.65
77.28
77.03
76.78
64.21
14.81
133.07
133.00
132.98
132.35
128.90
128.25
126.26
125.84
OEt
O
O
N
Ph
1k
CDCl3, 125 MHz
134
133
132
131
130
129
128
127
126
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

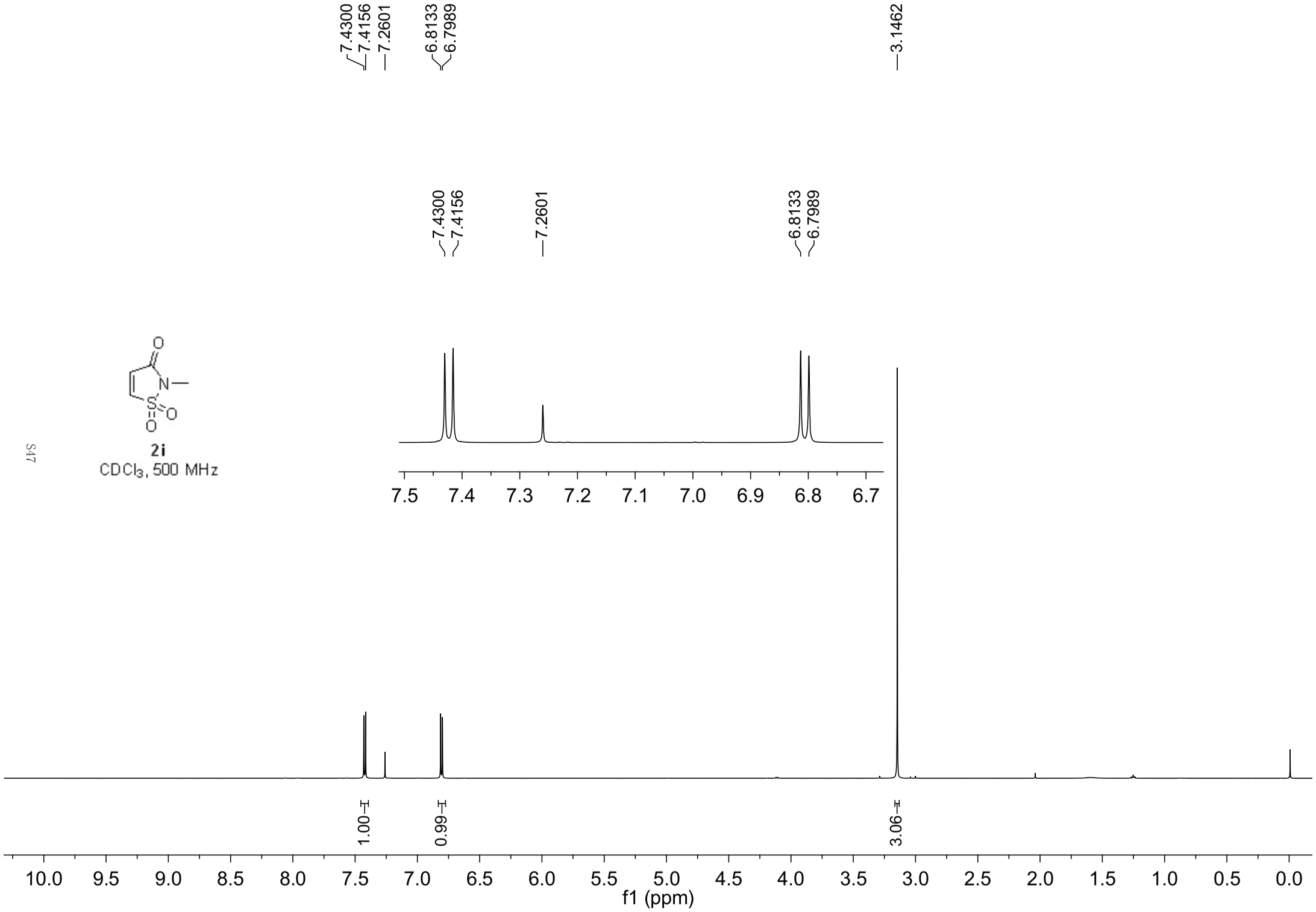

8.9068
8.8914
8.8766
8.1946
8.1797
7.6463
7.6315
7.6168
7.5779
7.5580
7.5425
7.5274
7.4611
7.4583
7.4463
7.4314
7.4194
7.4167
7.3141
7.2989
7.2836
7.2603
7.1520
7.1331
7.1149
F
O
O
N
Ph
1l
CDCl3, 500 MHz
1.00
1.99
1.00
0.97
2.03
1.03
1.02
1.01
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)
7.65
7.55
7.45
7.35
7.25
7.15

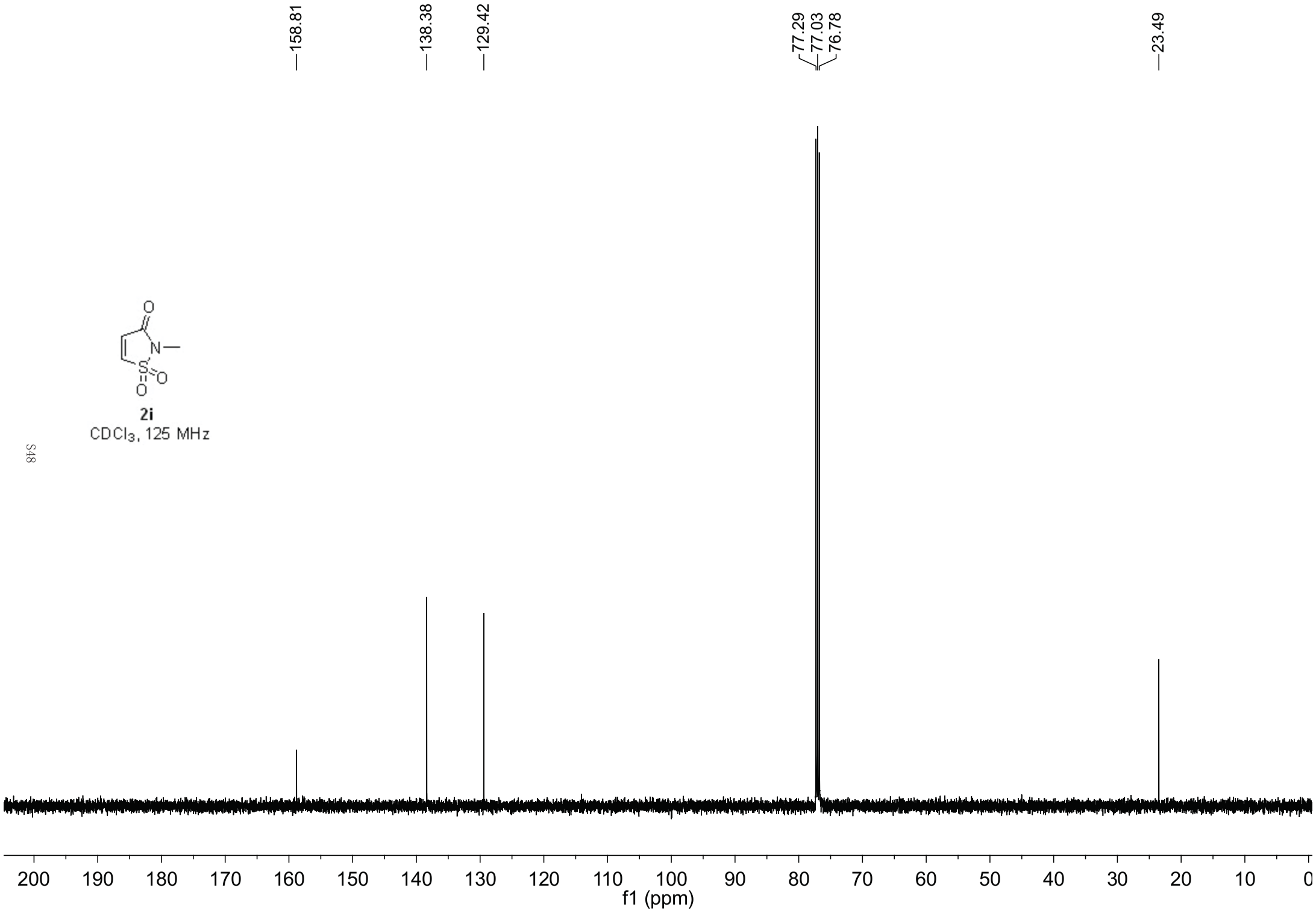

-113.97
-113.98
-113.99
-114.01
-114.02
F
O
O
N
Ph
1l
CDCl3, 470 MHz
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
f1 (ppm)

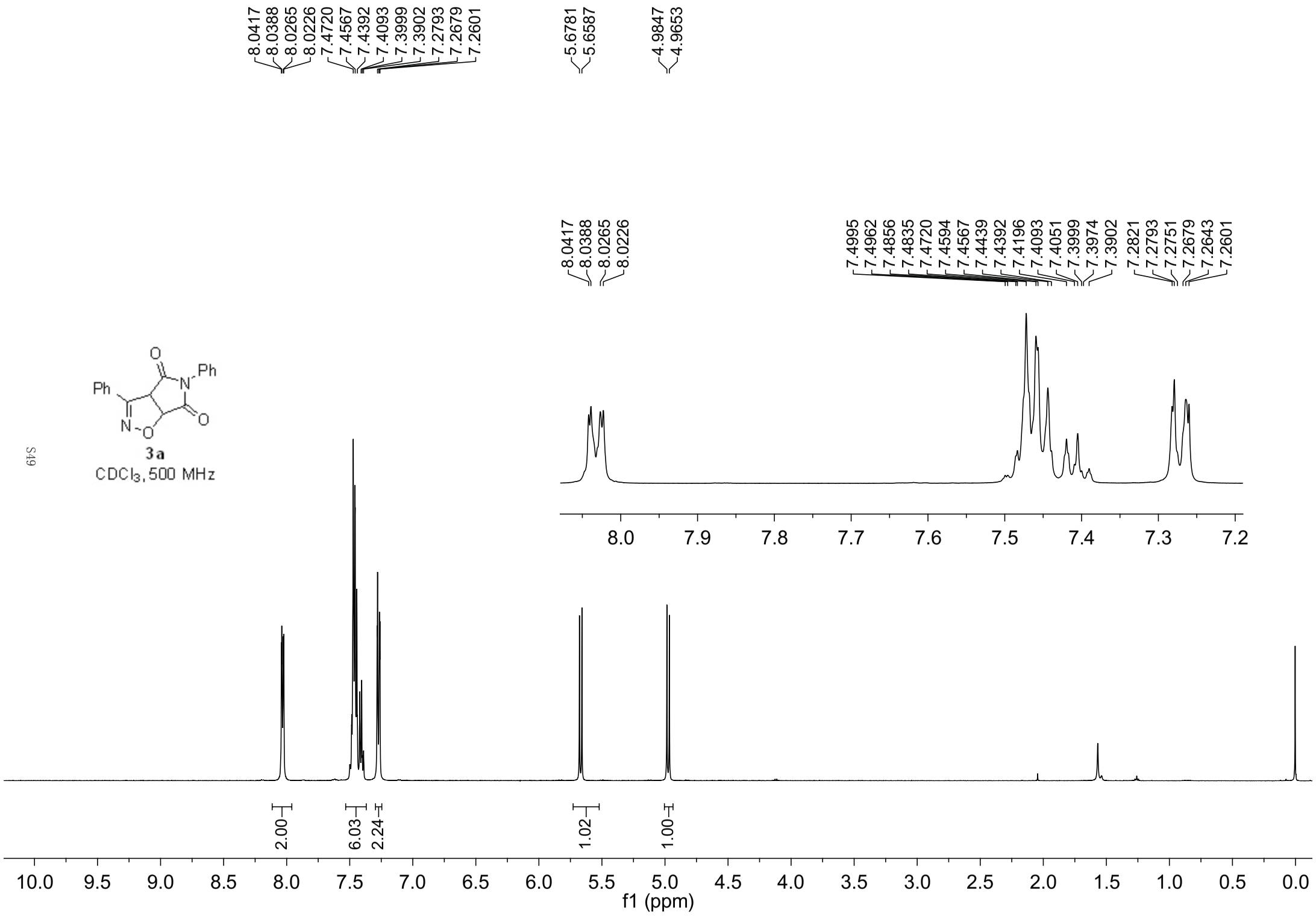

167.14
164.20
163.02
160.98
134.19
134.18
133.60
132.95
132.88
132.72
128.99
128.50
125.43
124.66
124.63
122.38
122.32
121.86
121.78
115.69
115.52
77.28
77.03
76.77
F
O
O
N
Ph
1l
CDCl3, 125 MHz
f1 (ppm)

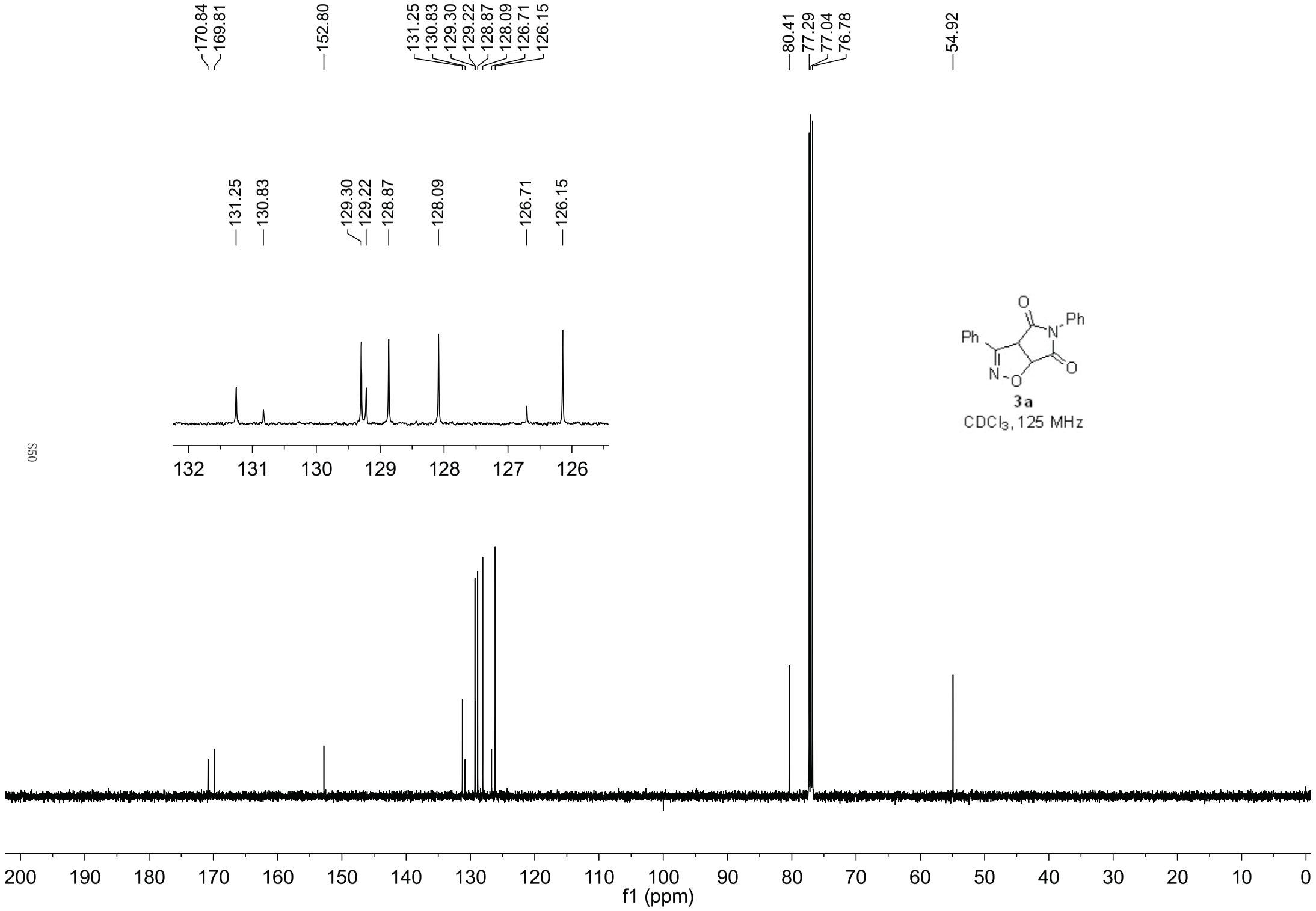

7.3813
7.3669
7.2600
6.7787
6.7645
3.6562
3.6412
3.6261
1.7943
1.7793
1.7647
1.7497
1.7347
1.3602
1.2626
0.8904
0.8767
0.8625
O
N-C8H17
S
O
O
2h
CDCl3, 500 MHz
1.00
1.01
2.00
2.05
10.00
2.98
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

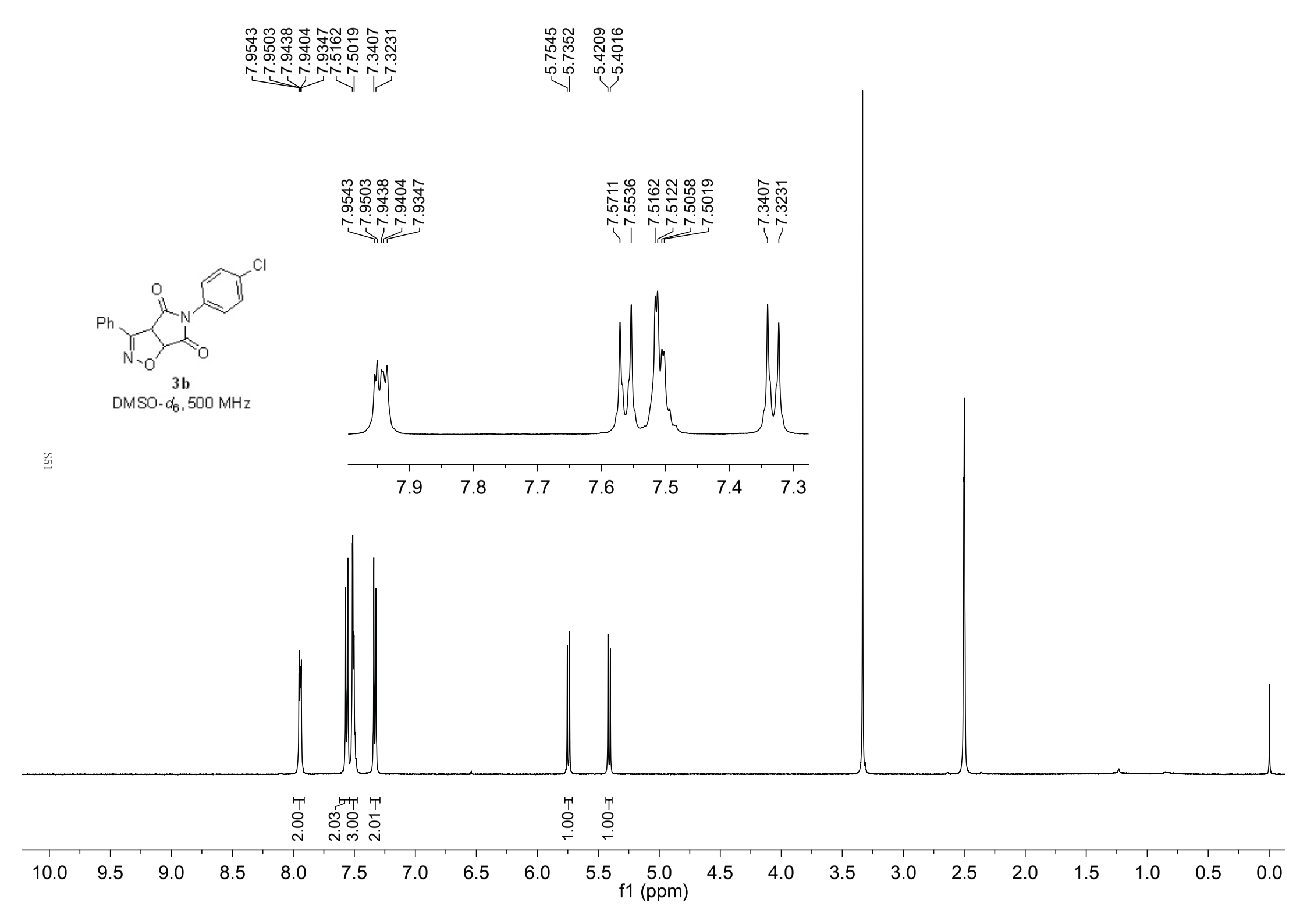

159.16
138.39
129.22
77.34
77.08
76.83
39.77
31.71
28.92
28.12
26.67
22.59
14.06
39.77
31.71
29.07
28.92
28.12
26.67
22.59
14.06
40
35
30
25
20
15
O
N-C8H17
S
O
O
2h
CDCl3, 125 MHz
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

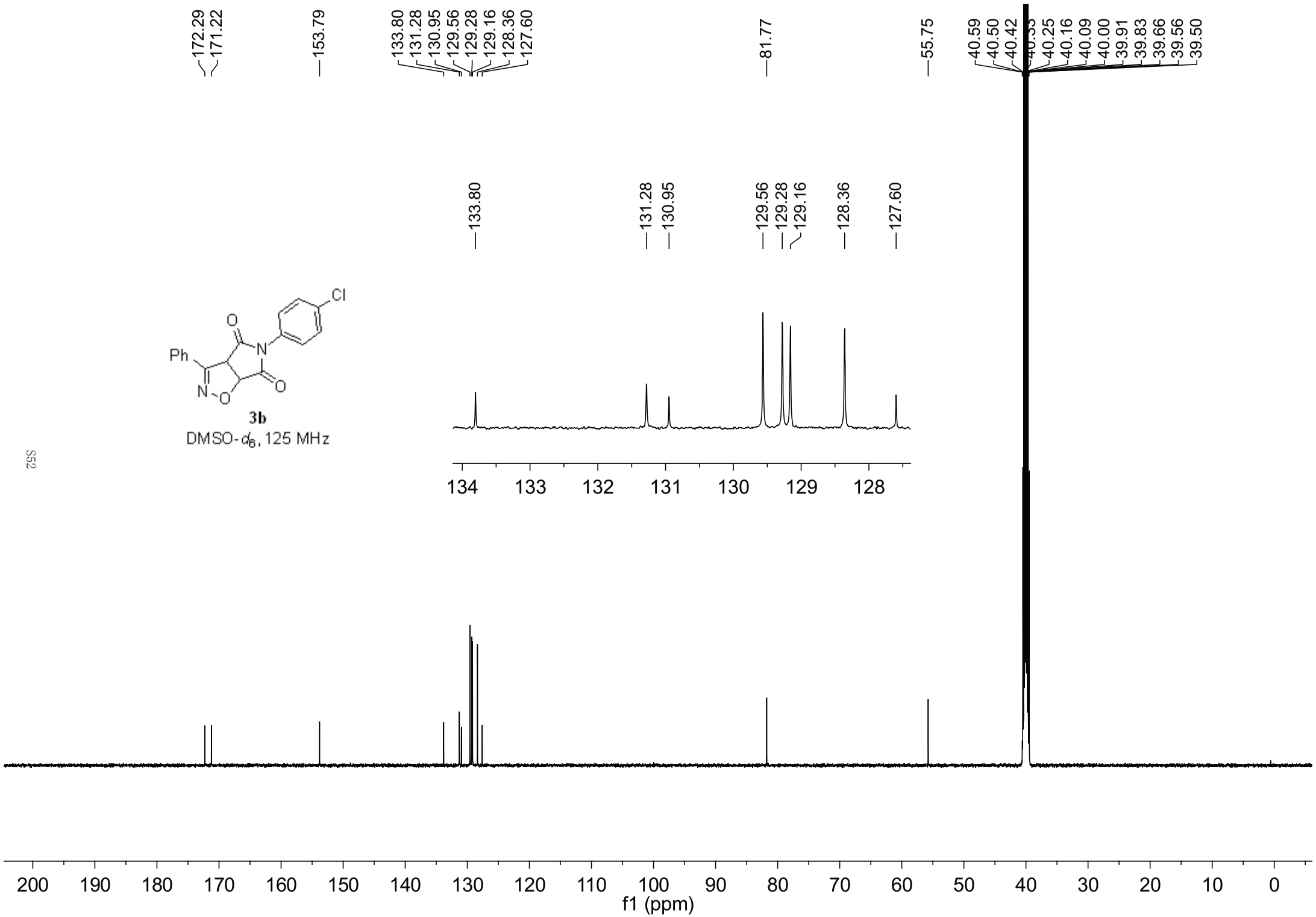

7.4300
7.4156
7.2601
6.8133
6.7989
3.1462
2i
CDCl3, 500 MHz
1.00
0.99
3.06
f1 (ppm)

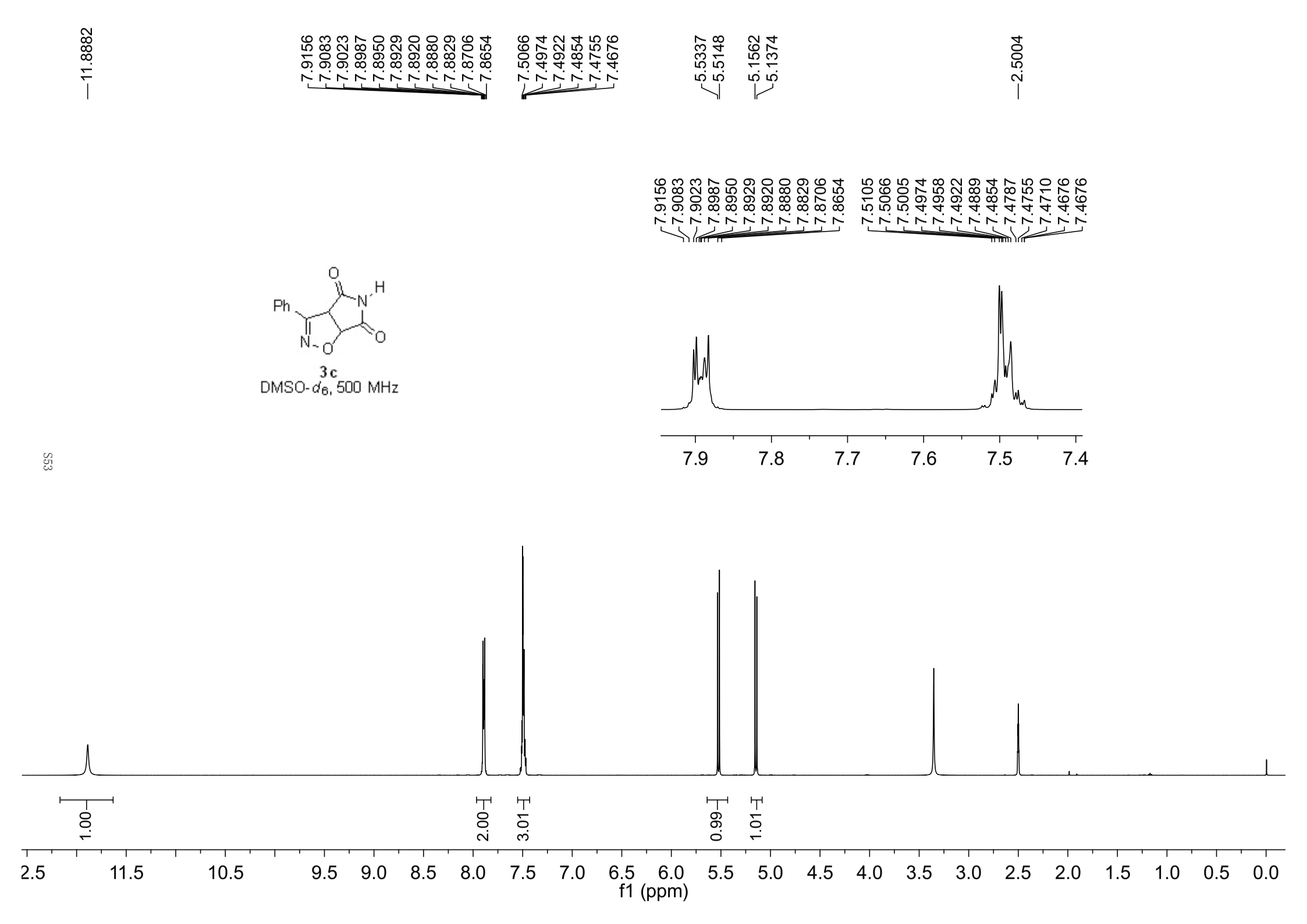

158.81
138.38
129.42
77.29
77.03
76.78
23.49
O
N
S
O
O
2i
CDCl3, 125 MHz
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

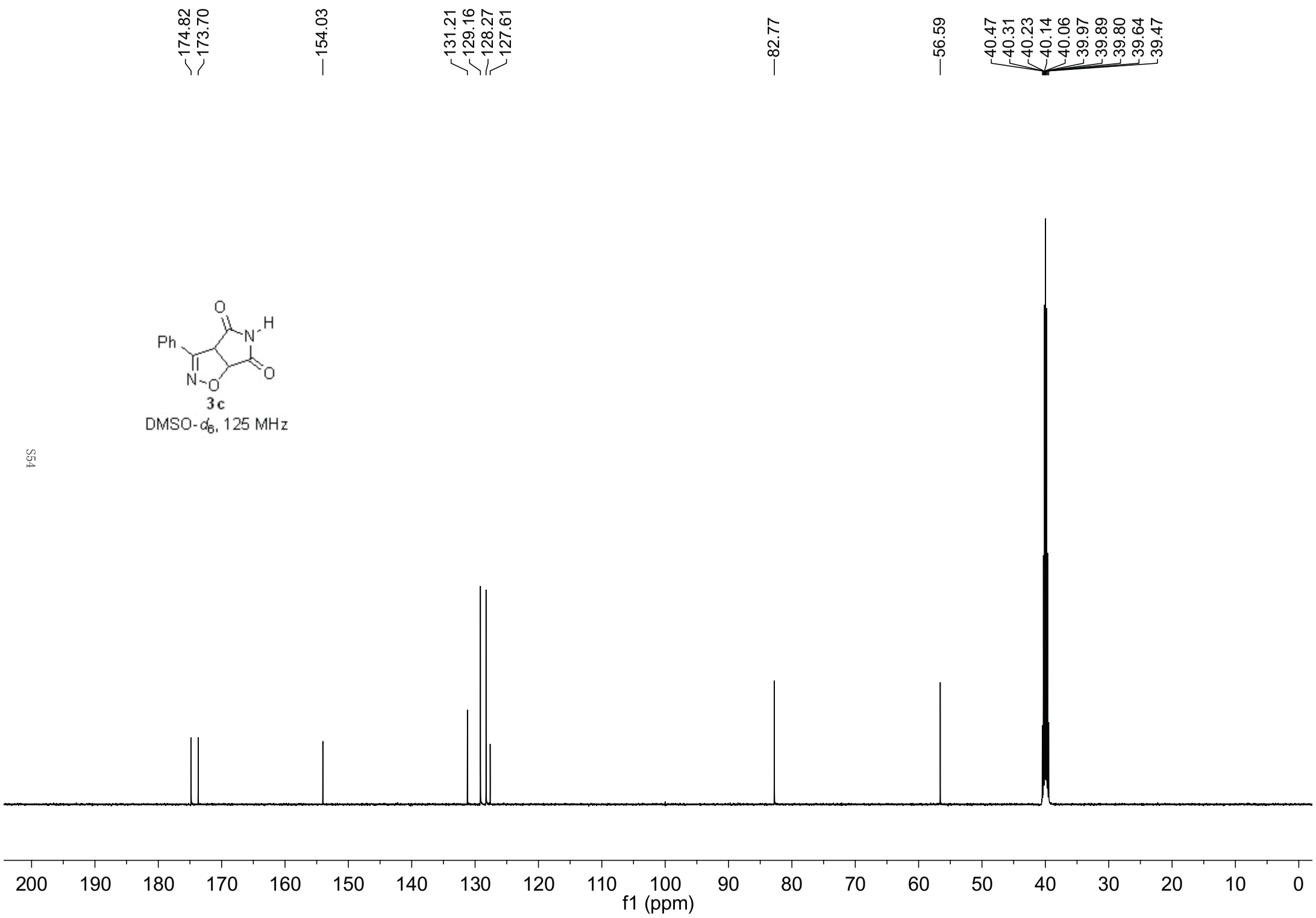

8.0417
8.0388
8.0265
8.0226
7.4720
7.4567
7.4392
7.4093
7.3999
7.3902
7.2793
7.2679
7.2601
5.6781
5.6587
4.9847
4.9653
7.4995
7.4962
7.4856
7.4835
7.4720
7.4594
7.4567
7.4439
7.4392
7.4196
7.4093
7.4051
7.3999
7.3974
7.3902
7.2821
7.2793
7.2751
7.2679
7.2643
7.2601
O
Ph
Ph
N
O
N
O
3a
CDCl3, 500 MHz
2.00
6.03
2.24
1.02
1.00
f1 (ppm)

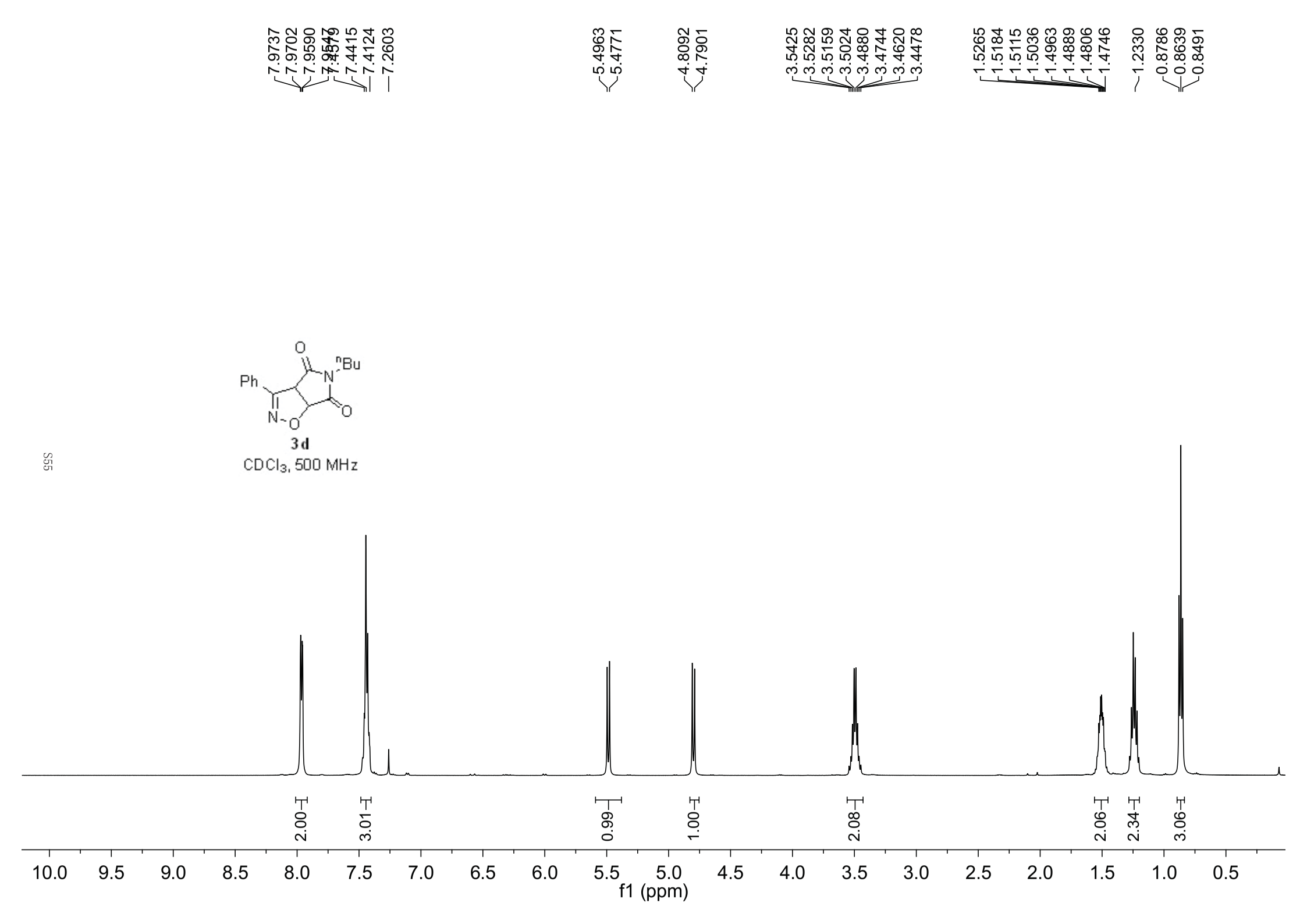

170.84
169.81
152.80
131.25
130.83
129.30
129.22
128.87
128.09
126.71
126.15
80.41
77.29
77.04
76.78
54.92
Ph
Ph
O
N
O
N
O
O
3a
CDCl3, 125 MHz
f1 (ppm)

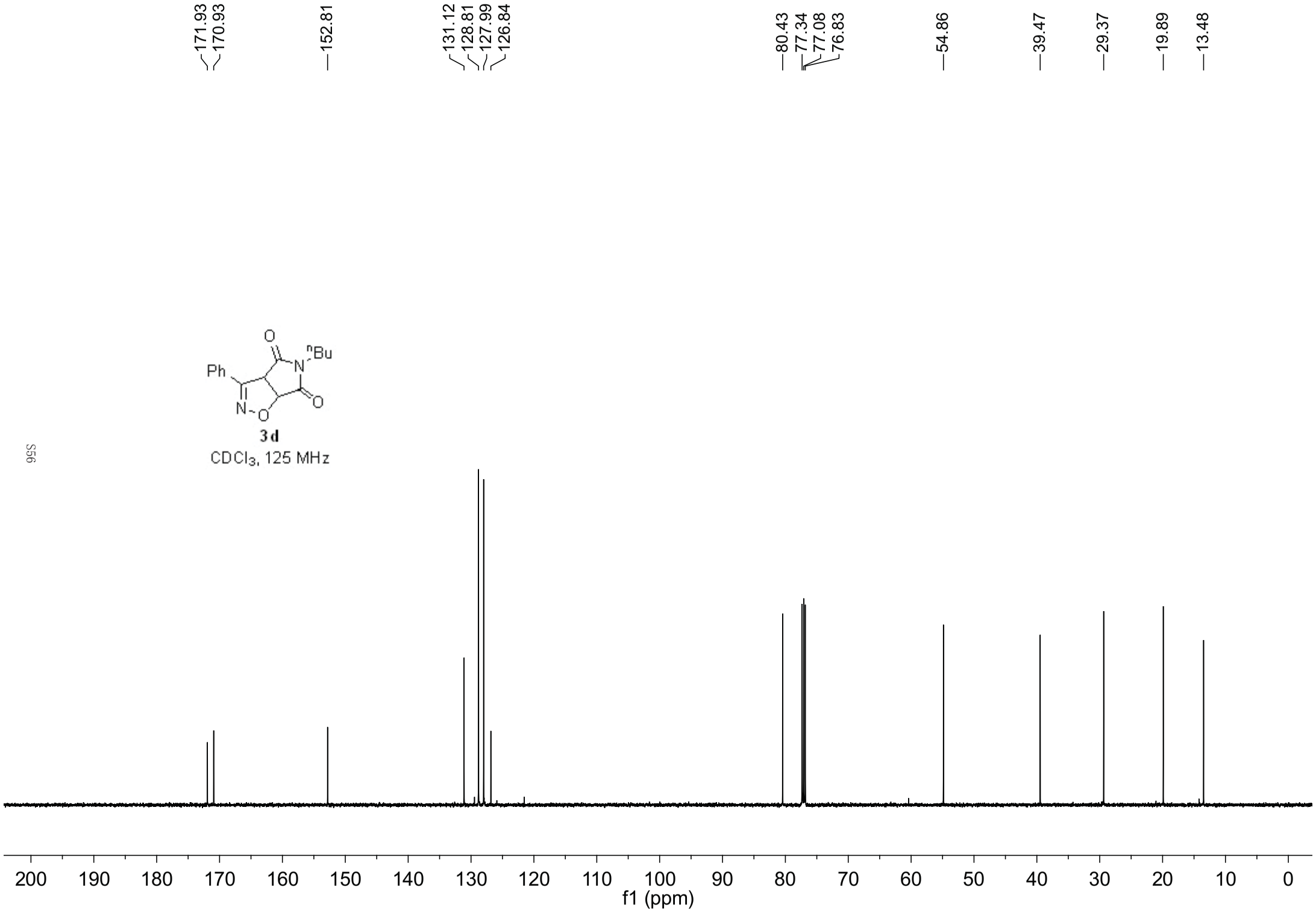

7.9543
7.9503
7.9438
7.9404
7.9347
7.5162
7.5019
7.3407
7.3231
5.7545
5.7352
5.4209
5.4016
Cl
O
N
Ph
O
N
O
3b
DMSO-d_6, 500 MHz
7.9543
7.9503
7.9438
7.9404
7.9347
7.5711
7.5536
7.5162
7.5122
7.5058
7.5019
7.3407
7.3231
7.9
7.8
7.7
7.6
7.5
7.4
7.3
2.00
2.03
3.00
2.01
1.00
1.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

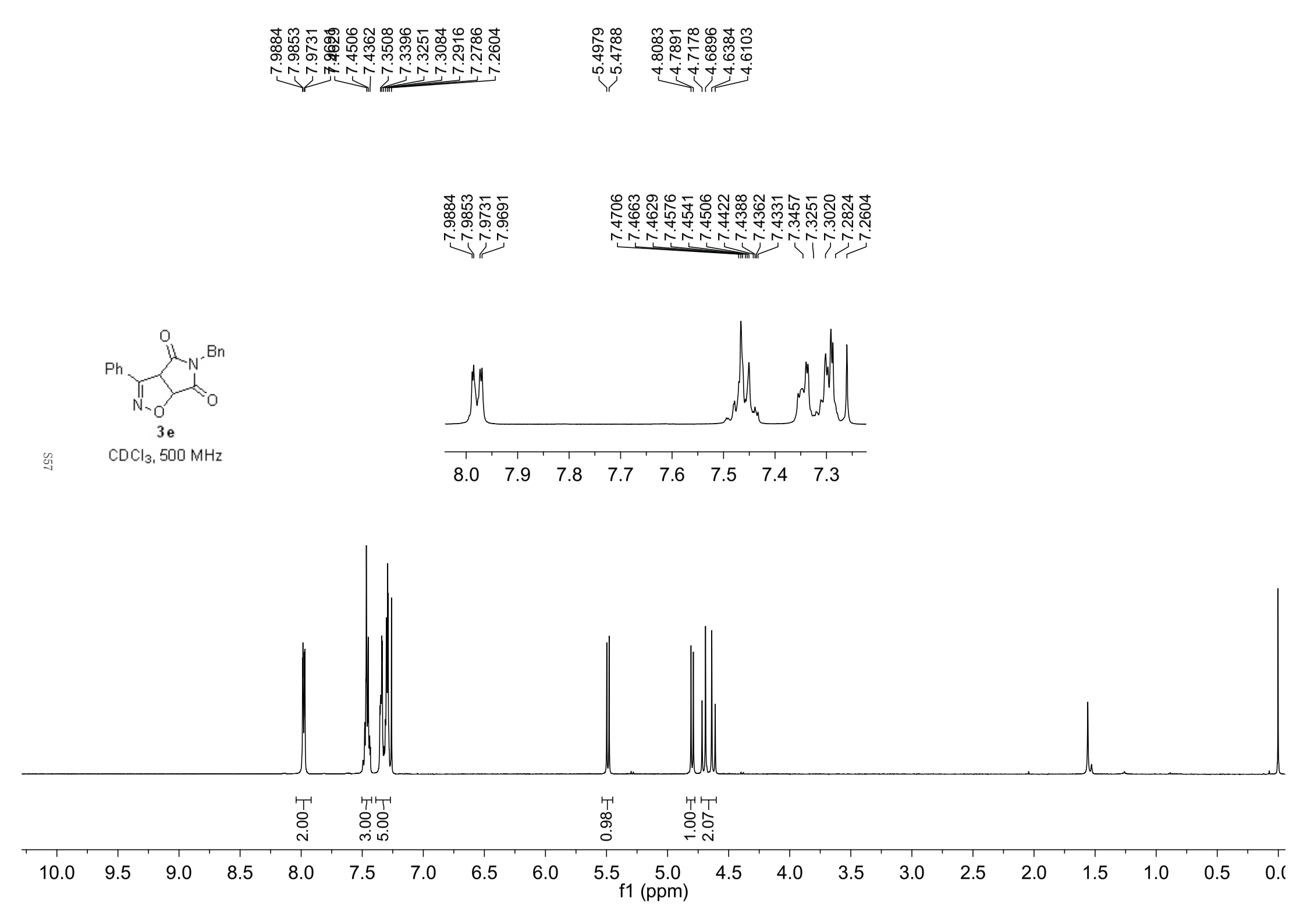

172.29
171.22
153.79
133.80
131.28
130.95
129.56
129.28
129.16
128.36
127.60
81.77
55.75
40.59
40.50
40.42
40.25
40.16
40.09
40.00
39.91
39.83
39.66
39.56
39.50
Cl
O
N
Ph
N
O
O
3b
DMSO-d_6, 125 MHz
134
133
132
131
130
129
128
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

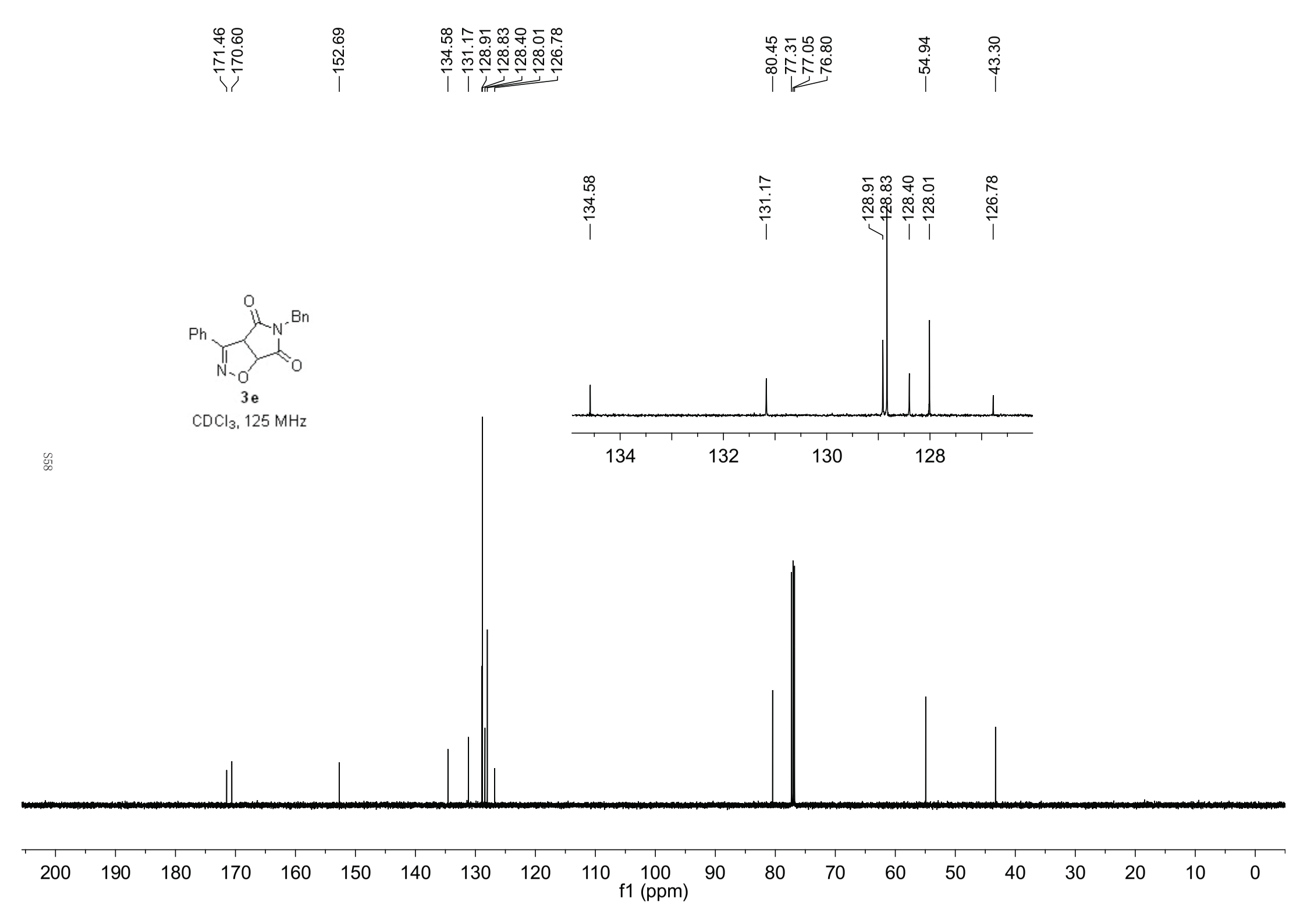

11.8882
7.9156
7.9083
7.9023
7.8987
7.8950
7.8929
7.8920
7.8880
7.8829
7.8706
7.8654
7.5066
7.4974
7.4922
7.4854
7.4755
7.4676
5.5337
5.5148
5.1562
5.1374
2.5004
7.9156
7.9083
7.9023
7.8987
7.8950
7.8929
7.8920
7.8880
7.8829
7.8706
7.8654
7.5105
7.5066
7.5005
7.4974
7.4958
7.4922
7.4889
7.4854
7.4787
7.4755
7.4710
7.4676
7.4676
Ph
N
H
O
O
N
O
3c
DMSO-d6, 500 MHz
7.9
7.8
7.7
7.6
7.5
7.4
1.00
2.00
3.01
0.99
1.01
2.5
11.5
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

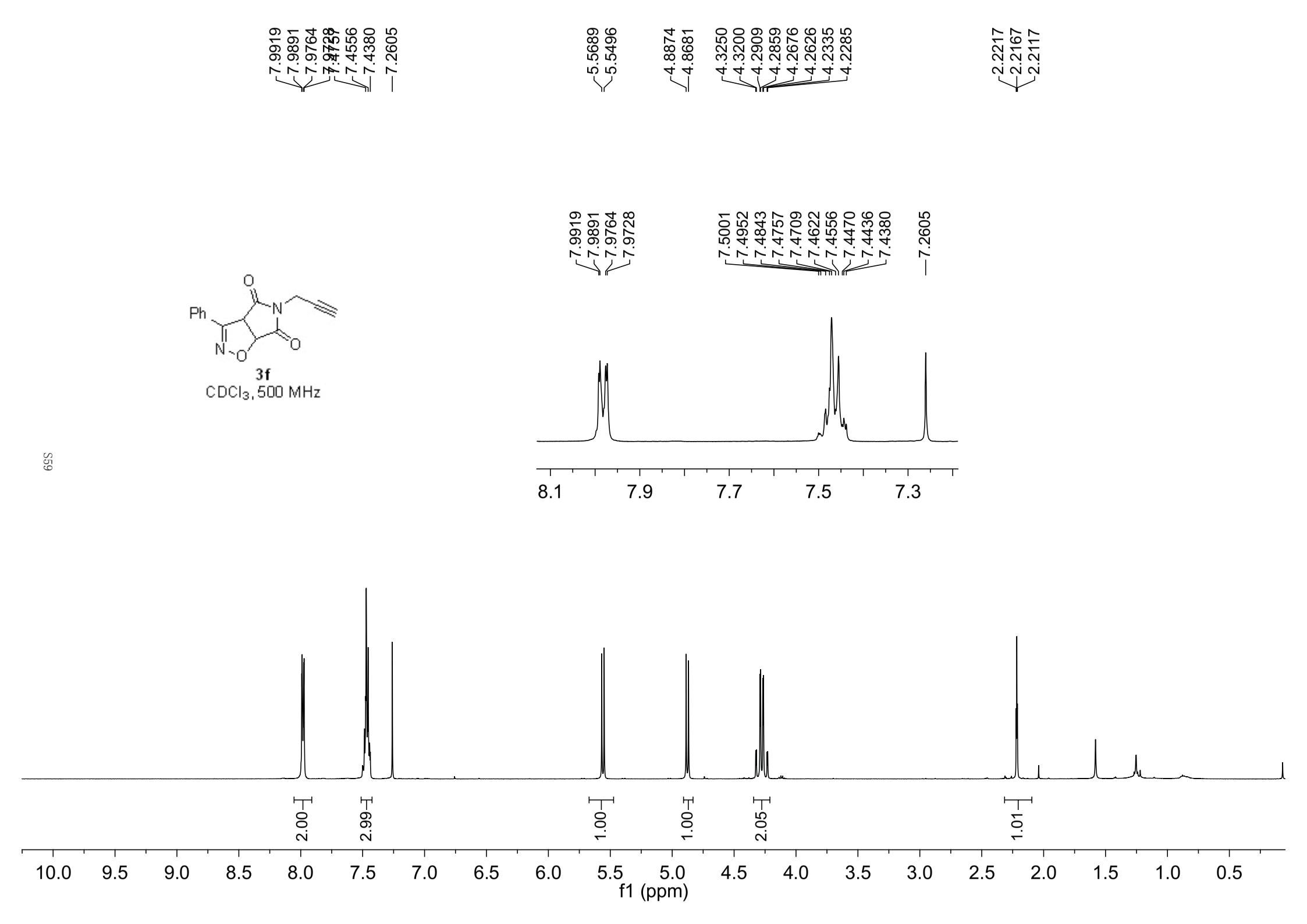

174.82
173.70
154.03
131.21
129.16
128.27
127.61
82.77
56.59
40.47
40.31
40.23
40.14
40.06
39.97
39.89
39.80
39.64
39.47
Ph
O
N
H
O
N
O
3c
DMSO-d_6, 125 MHz
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

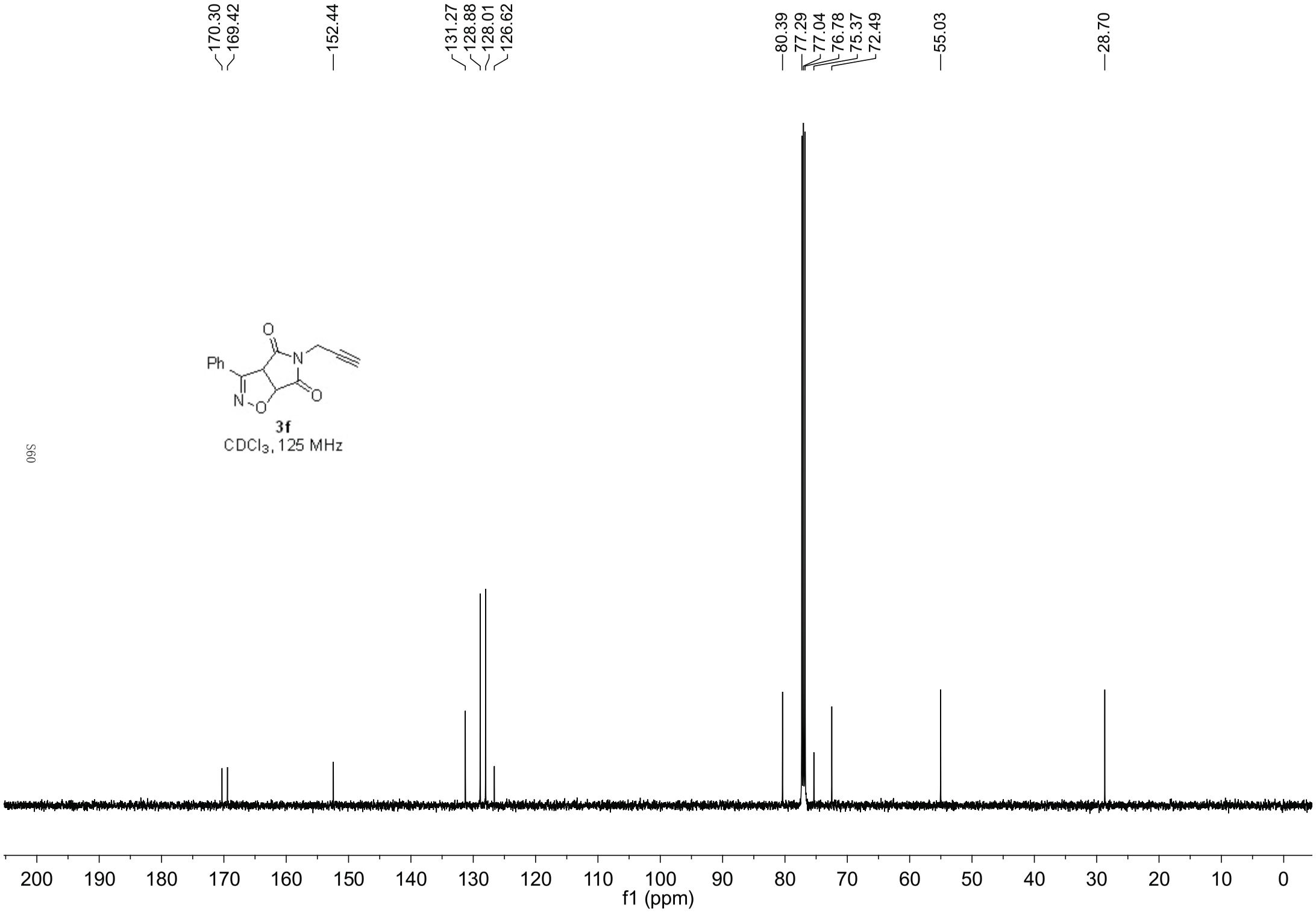

7.9737
7.9702
7.9590
7.4415
7.4124
7.2603
5.4963
5.4771
4.8092
4.7901
3.5425
3.5282
3.5159
3.5024
3.4880
3.4744
3.4620
3.4478
1.5265
1.5184
1.5115
1.5036
1.4963
1.4889
1.4806
1.4746
1.2330
0.8786
0.8639
0.8491
O
nBu
N
Ph
N
O
O
3d
CDCl3, 500 MHz
2.00
3.01
0.99
1.00
2.08
2.06
2.34
3.06
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

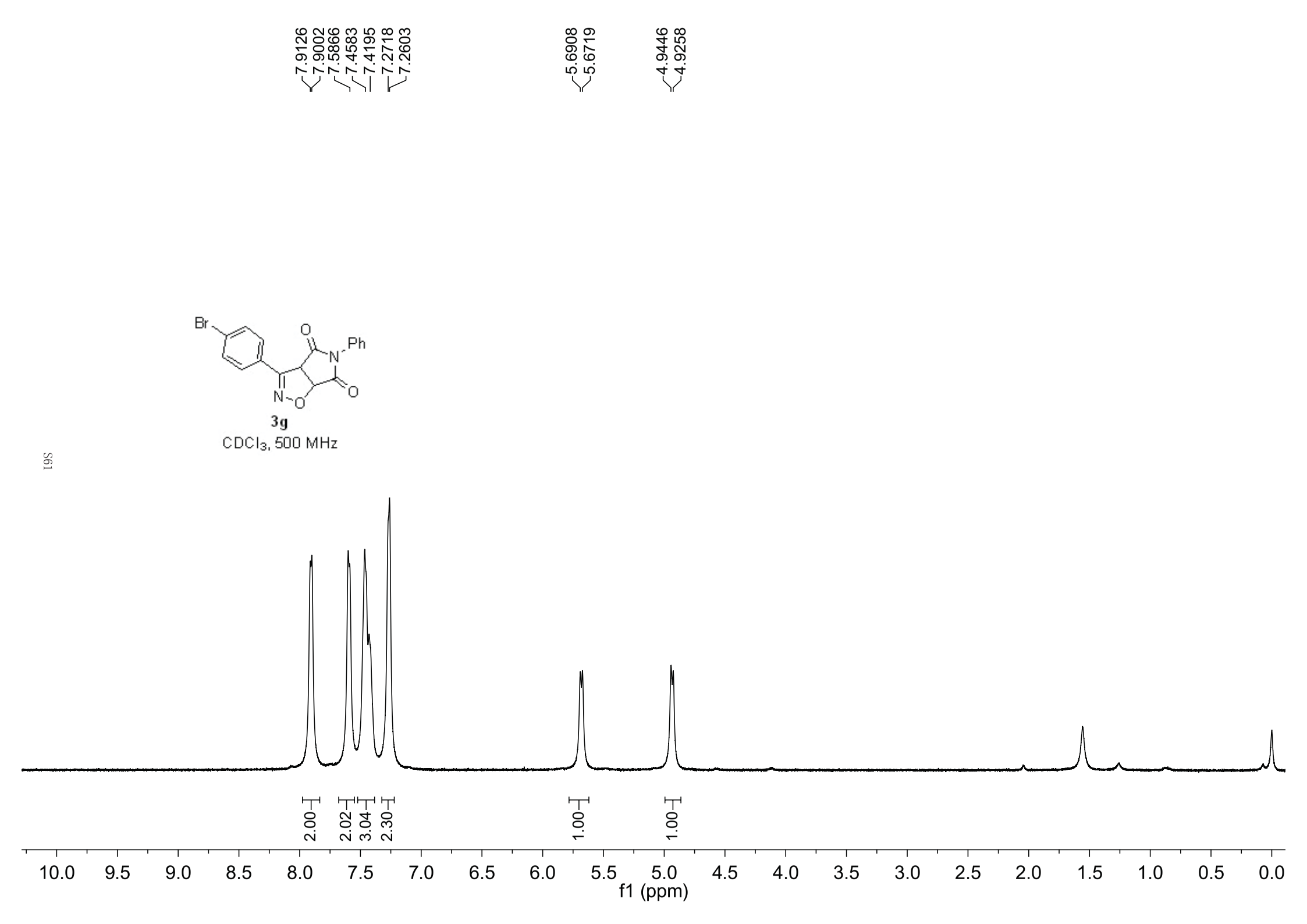

171.93
170.93
152.81
131.12
128.81
127.99
126.84
80.43
77.34
77.08
76.83
54.86
39.47
29.37
19.89
13.48
O
nBu
Ph
N
O
N
O
3d
CDCl3, 125 MHz
S56
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

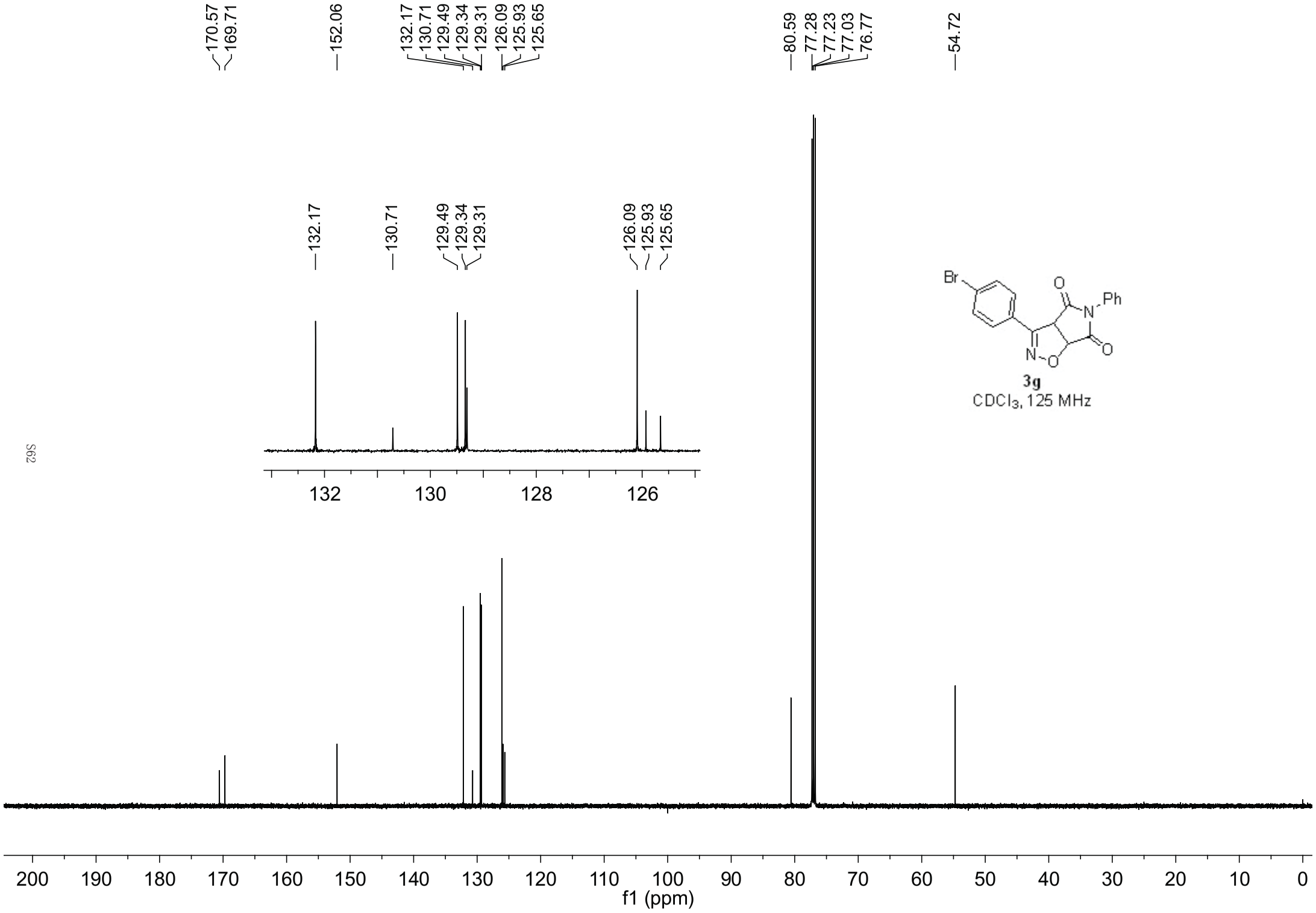

7.9884
7.9853
7.9731
7.4506
7.4362
7.3508
7.3396
7.3251
7.3084
7.2916
7.2786
7.2604
5.4979
5.4788
4.8083
4.7891
4.7178
4.6896
4.6384
4.6103
7.9884
7.9853
7.9731
7.9691
7.4706
7.4663
7.4629
7.4576
7.4541
7.4506
7.4422
7.4388
7.4362
7.4331
7.3457
7.3251
7.3020
7.2824
7.2604
Ph
Bn
O
N
O
N
O
3e
CDCl3, 500 MHz
8.0
7.9
7.8
7.7
7.6
7.5
7.4
7.3
2.00
3.00
5.00
0.98
1.00
2.07
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

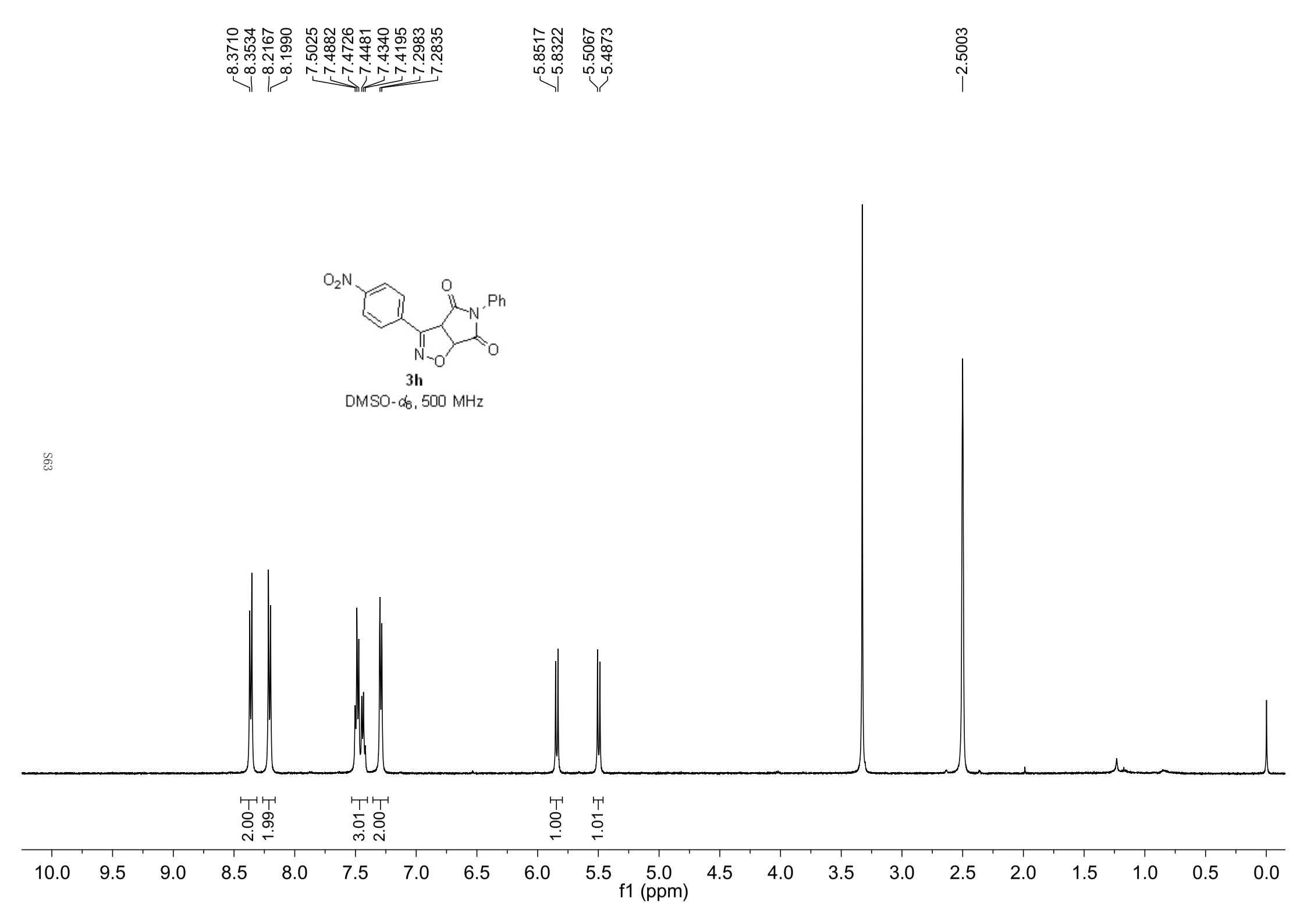

171.46
170.60
152.69
134.58
131.17
128.91
128.83
128.40
128.01
126.78
80.45
77.31
77.05
76.80
54.94
43.30
Ph
Bn
O
N
O
N
O
O
3e
CDCl3, 125 MHz
S58
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

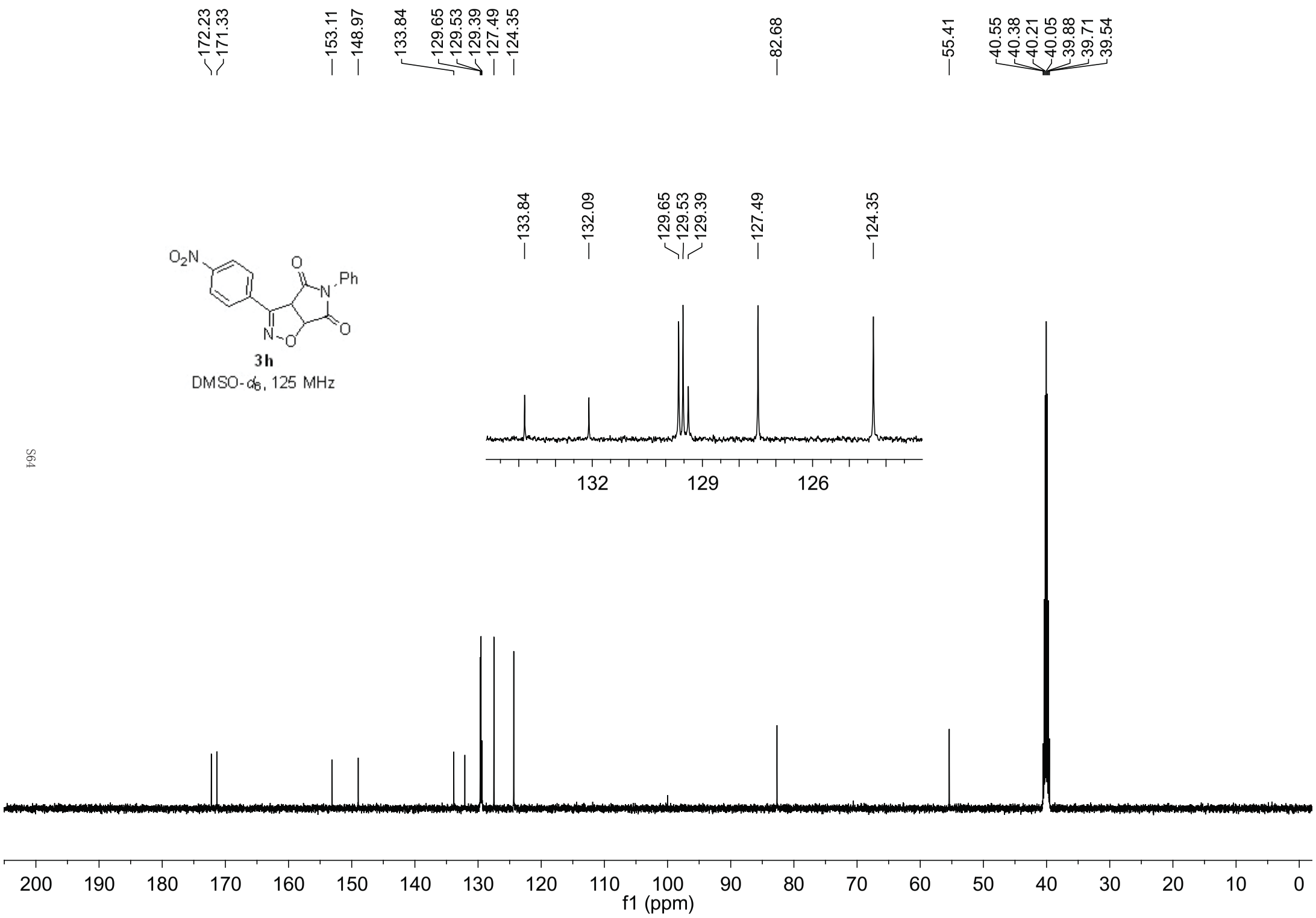

7.9919
7.9891
7.9764
7.9728
7.4757
7.4556
7.4380
7.2605
5.5689
5.5496
4.8874
4.8681
4.3250
4.3200
4.2909
4.2859
4.2676
4.2626
4.2335
4.2285
2.2217
2.2167
2.2117
7.9919
7.9891
7.9764
7.9728
7.5001
7.4952
7.4843
7.4757
7.4709
7.4622
7.4556
7.4470
7.4436
7.4380
7.2605
8.1
7.9
7.7
7.5
7.3
Ph
O
N
O
N
O
3f
CDCl3, 500 MHz
2.00
2.99
1.00
1.00
2.05
1.01
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

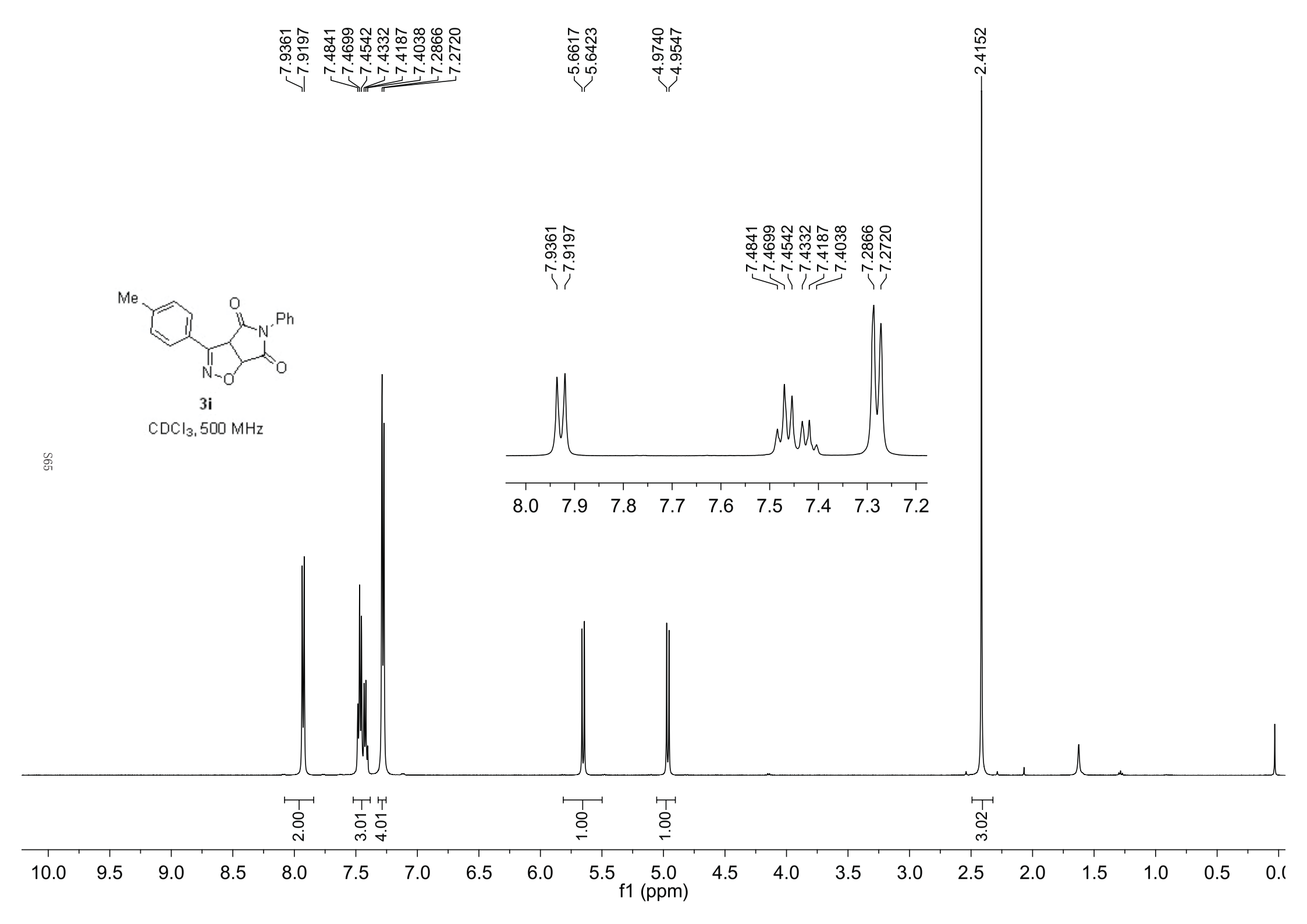

170.30
169.42
152.44
131.27
128.88
128.01
126.62
80.39
77.29
77.04
76.78
75.37
72.49
55.03
28.70
Ph
O
N
O
N
O
O
3f
CDCl3, 125 MHz
S60
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

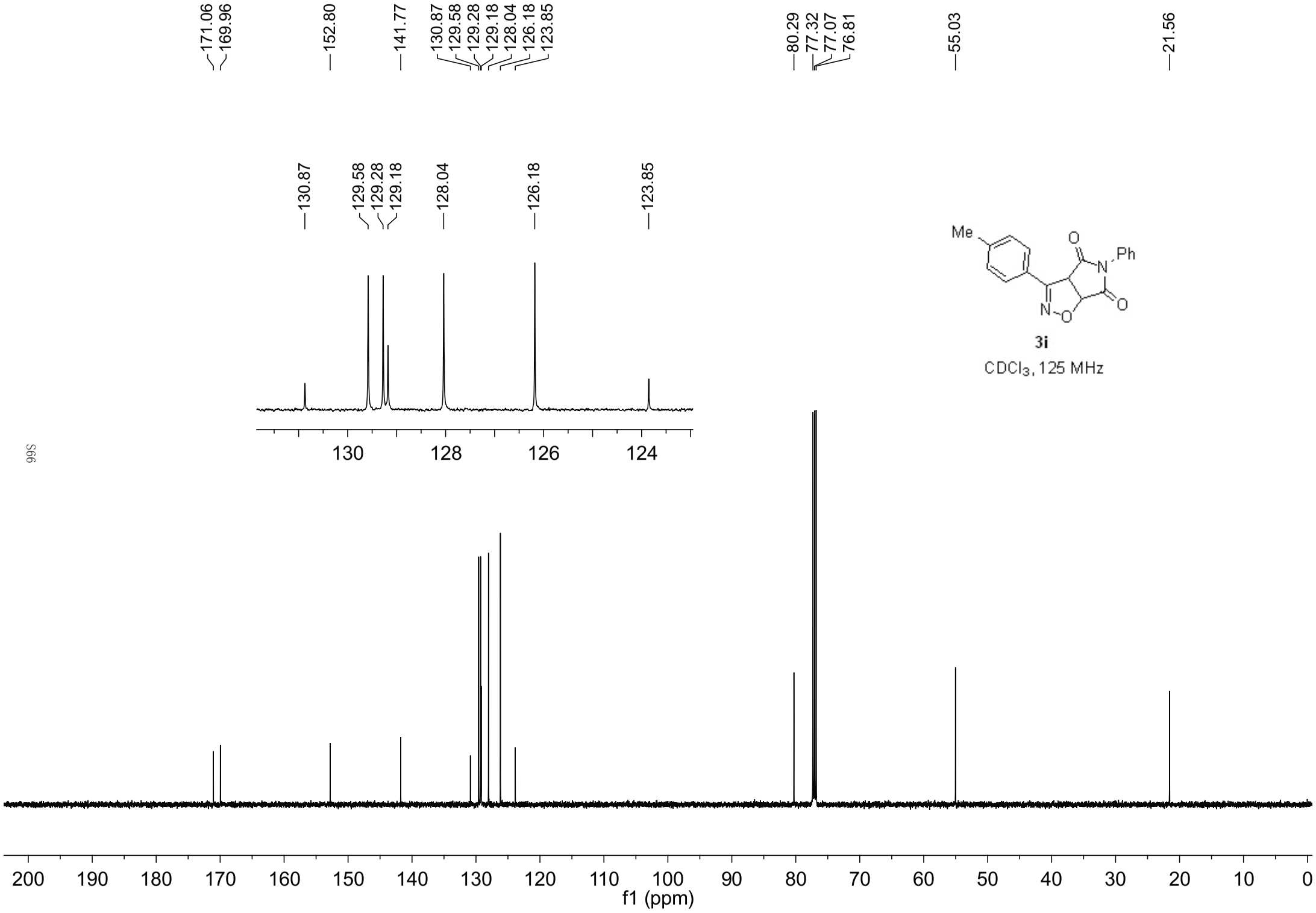

7.9126
7.9002
7.5866
7.4583
7.4195
7.2718
7.2603
5.6908
5.6719
4.9446
4.9258
Br
O
N
Ph
O
N
O
3g
CDCl3, 500 MHz
2.00
2.02
3.04
2.30
1.00
1.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

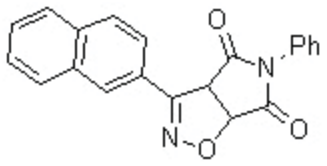

170.57
169.71
152.06
132.17
130.71
129.49
129.34
129.31
126.09
125.93
125.65
80.59
77.28
77.23
77.03
76.77
54.72
132.17
130.71
129.49
129.34
129.31
126.09
125.93
125.65
132
130
128
126
Br
O
N
Ph
O
N
O
3g
CDCl3, 125 MHz
S62
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


8.3710
8.3534
8.2167
8.1990
7.5025
7.4882
7.4726
7.4481
7.4340
7.4195
7.2983
7.2835
5.8517
5.8322
5.5067
5.4873
2.5003
O2N
O
N
Ph
O
N
O
3h
DMSO-d6, 500 MHz
S63
2.00
1.99
3.01
2.00
1.00
1.01
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


172.23
171.33
153.11
148.97
133.84
129.65
129.53
129.39
127.49
124.35
82.68
55.41
40.55
40.38
40.21
40.05
39.88
39.71
39.54
O2N
O
N
Ph
O
N
O
3h
DMSO-d6, 125 MHz
133.84
132.09
129.65
129.53
129.39
127.49
124.35
132
129
126
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

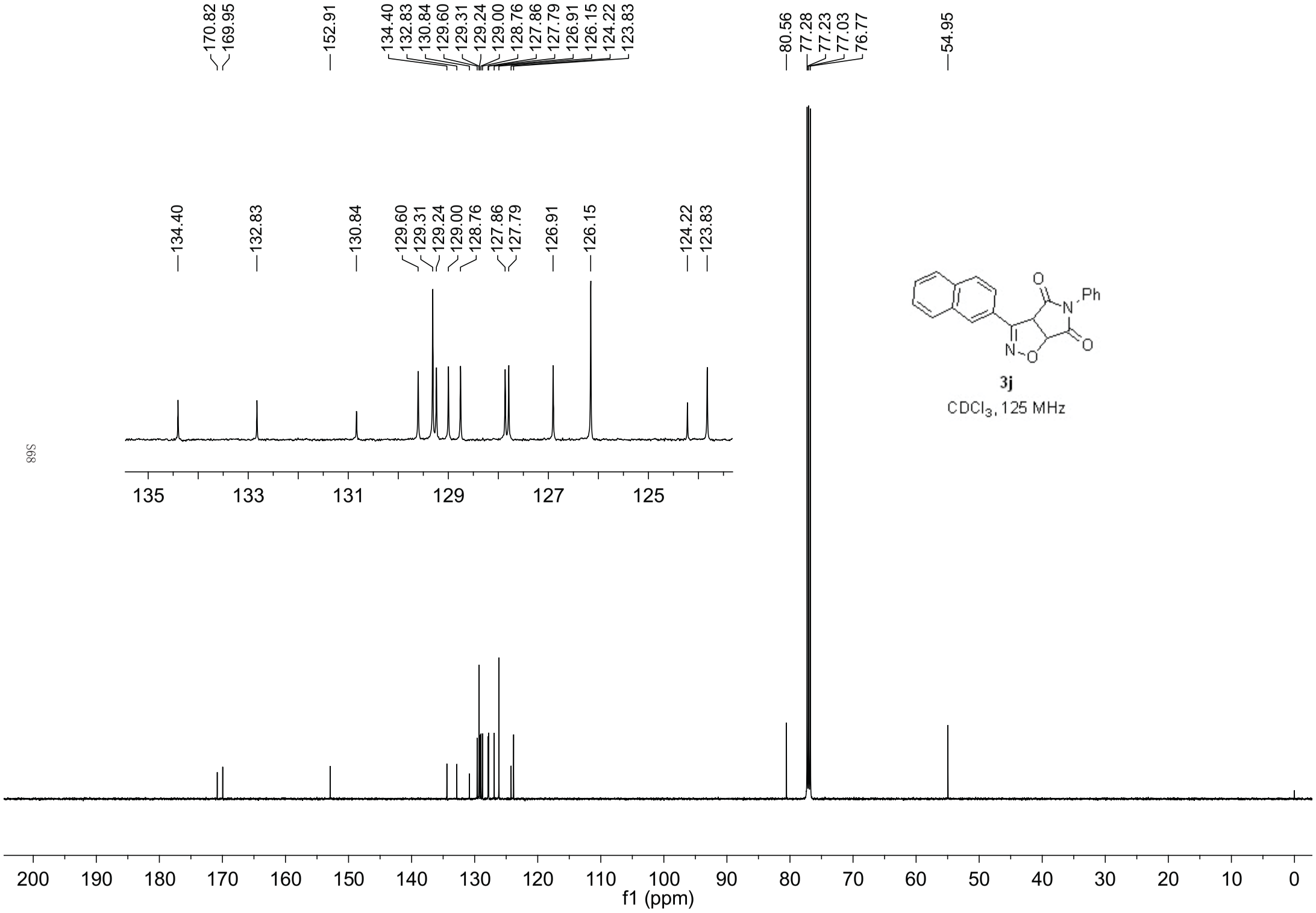

7.9361
7.9197
7.4841
7.4699
7.4542
7.4332
7.4187
7.4038
7.2866
7.2720
5.6617
5.6423
4.9740
4.9547
2.4152
Me
O
N
Ph
O
N
O
3i
CDCl3, 500 MHz
S65
8.0
7.9
7.8
7.7
7.6
7.5
7.4
7.3
7.2
2.00
3.01
4.01
1.00
1.00
3.02
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

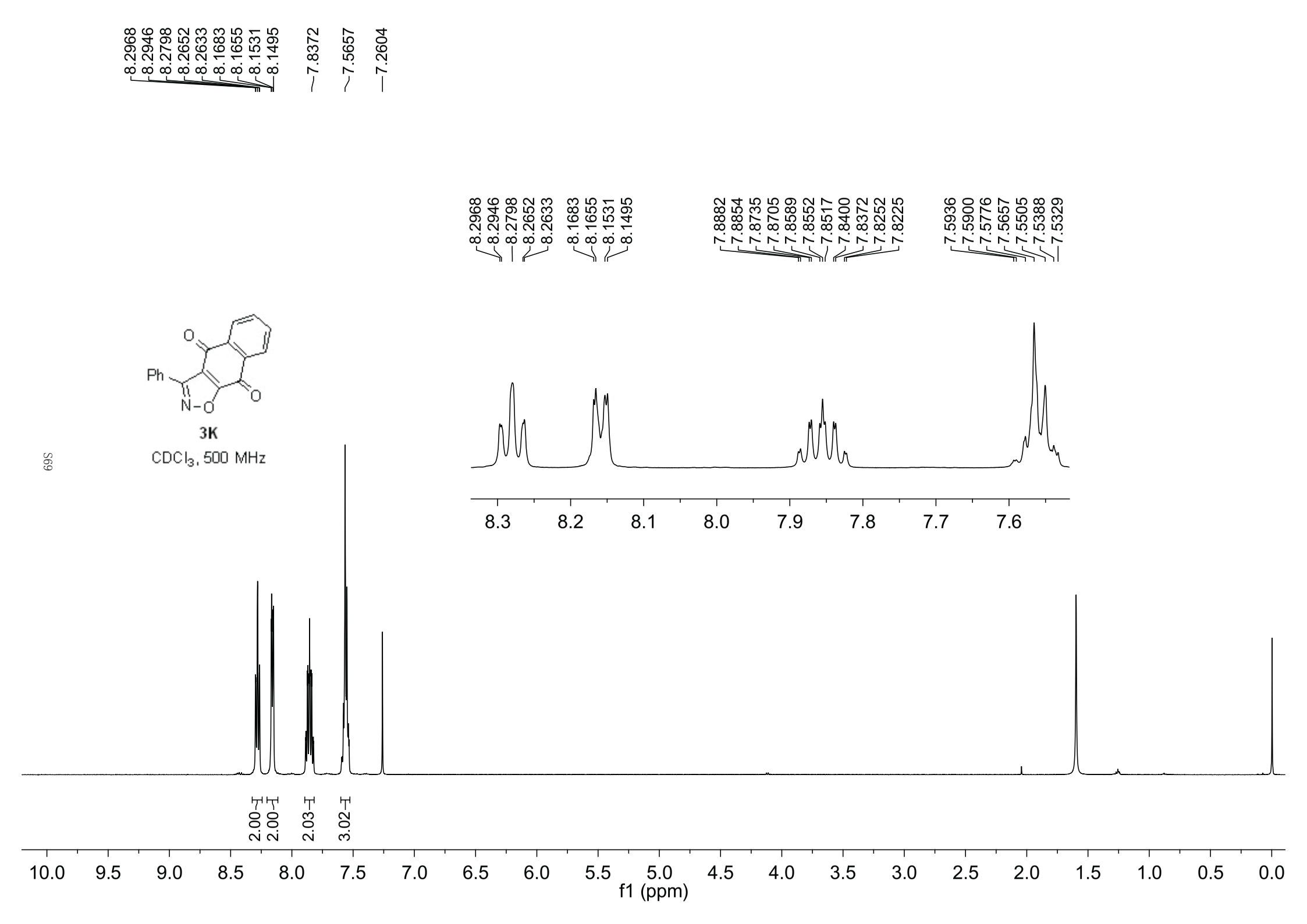

171.06
169.96
152.80
141.77
130.87
129.58
129.28
129.18
128.04
126.18
123.85
80.29
77.32
77.07
76.81
55.03
21.56
Me
O
N
Ph
O
N
O
3i
CDCl3, 125 MHz
S66
f1 (ppm)

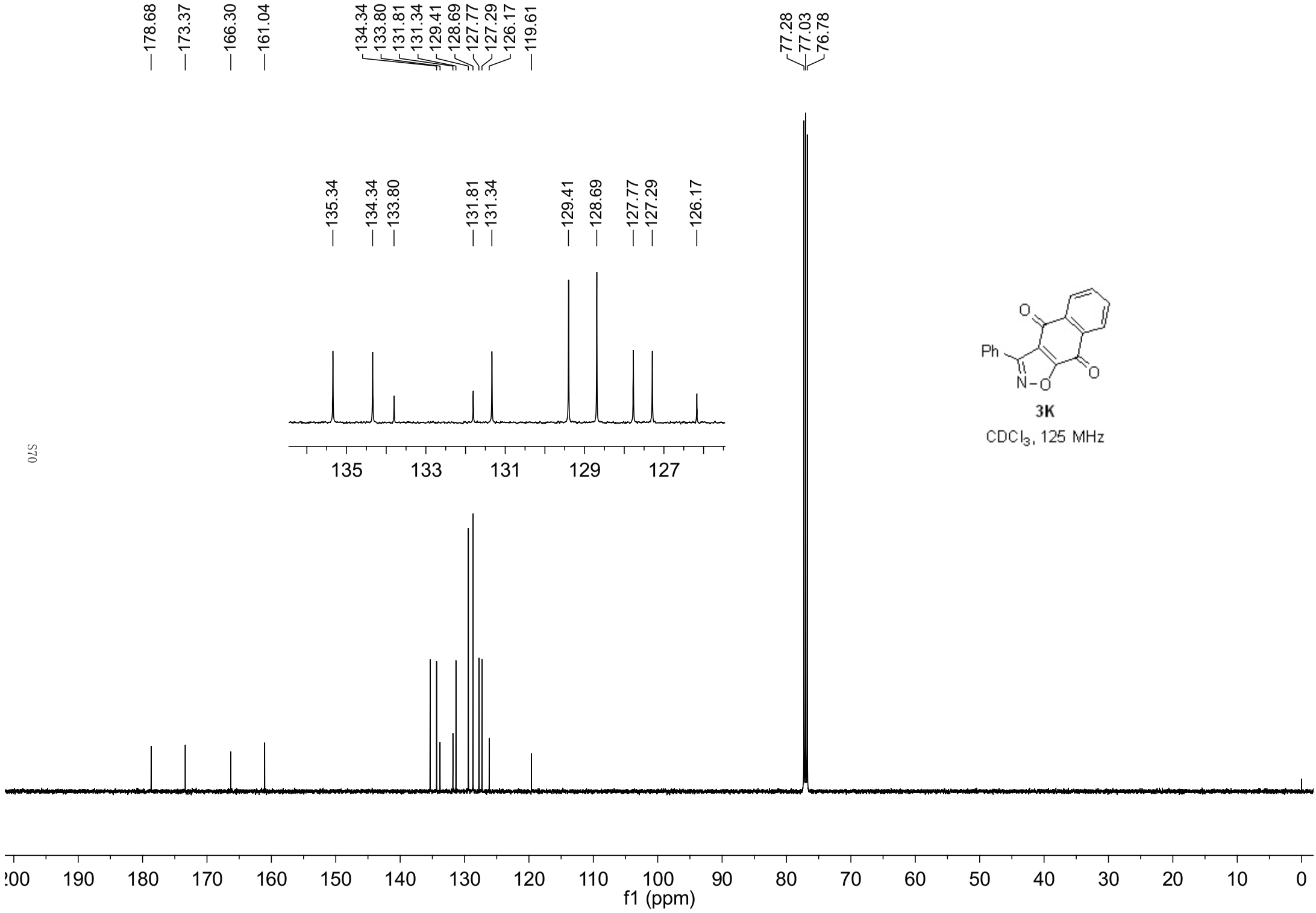


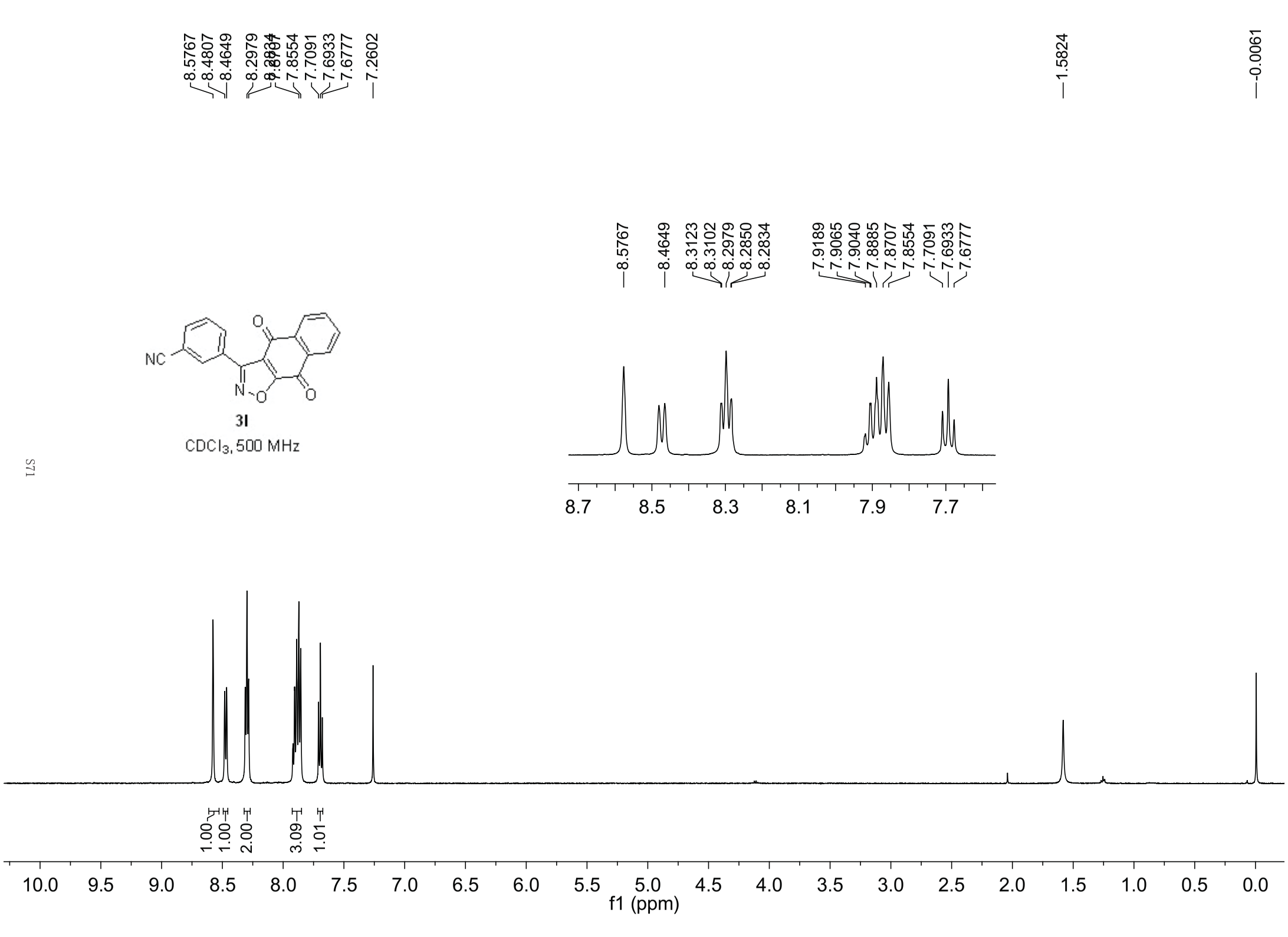
3j

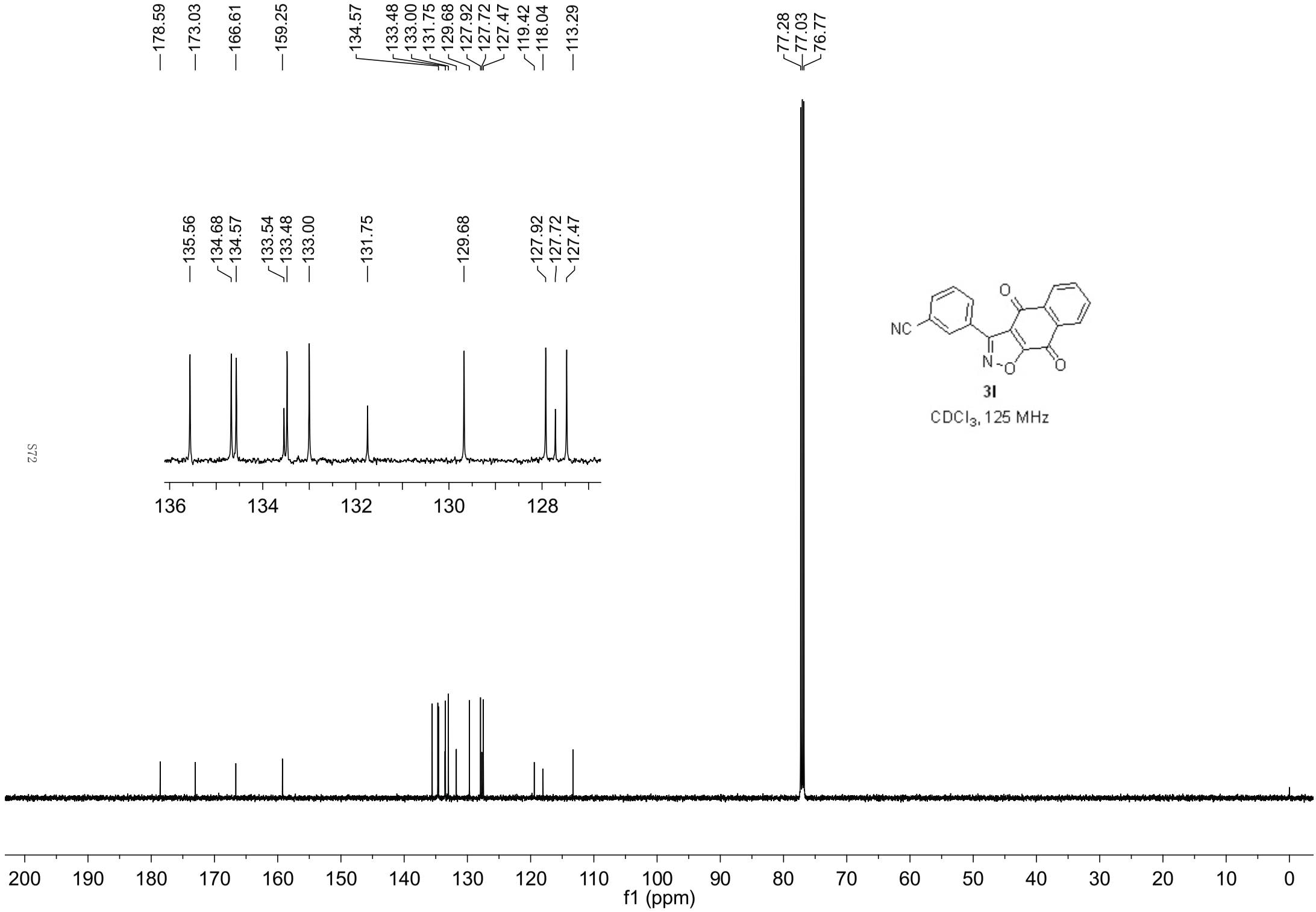
$CDCl_3$, 500 MHz

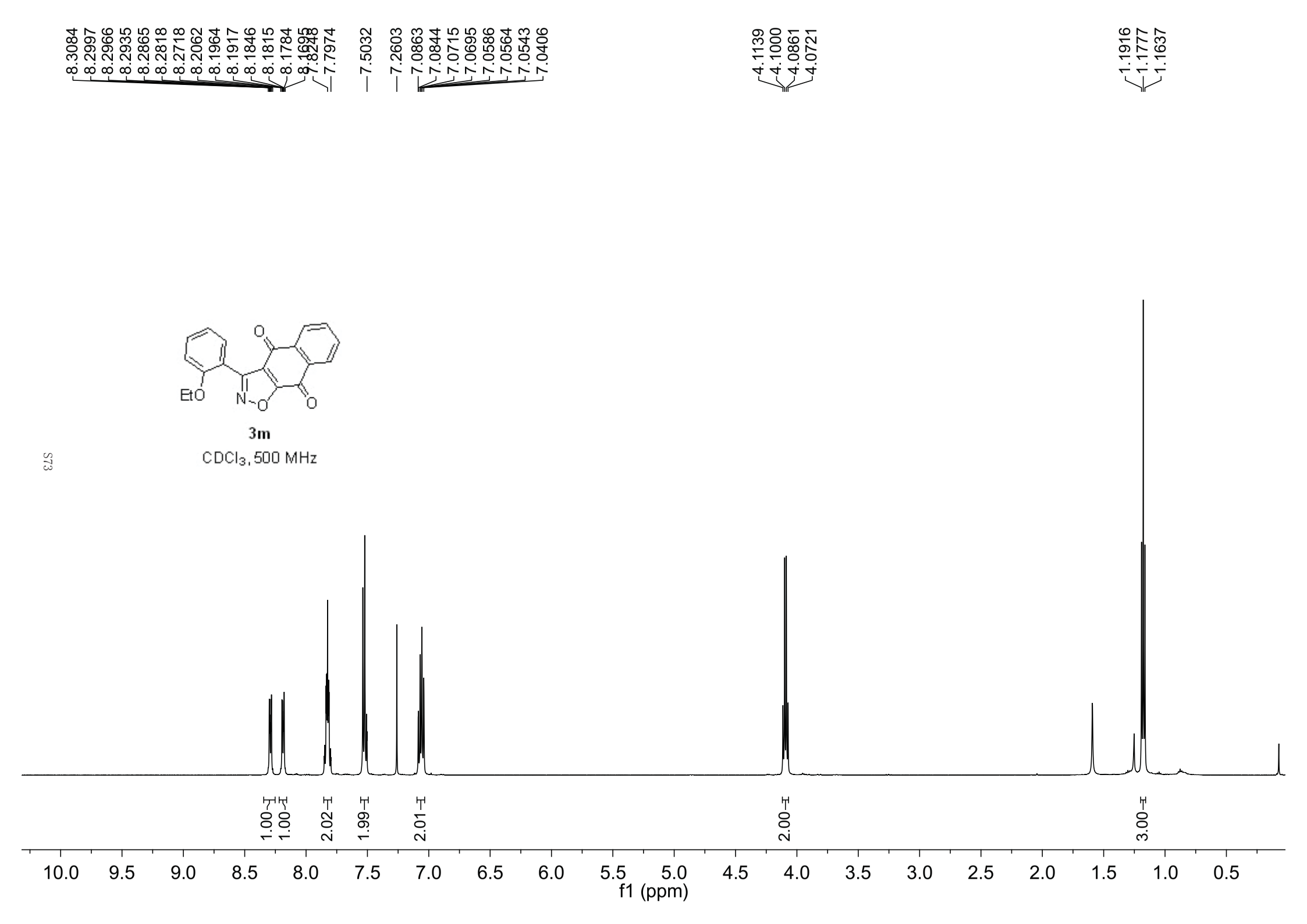

170.82
169.95
152.91
134.40
132.83
130.84
129.60
129.31
129.24
129.00
128.76
127.86
127.79
126.91
126.15
124.22
123.83
80.56
77.28
77.23
77.03
76.77
54.95
Ph
O
N
O
N
O
3j
CDCl3, 125 MHz
135
133
131
129
127
125
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

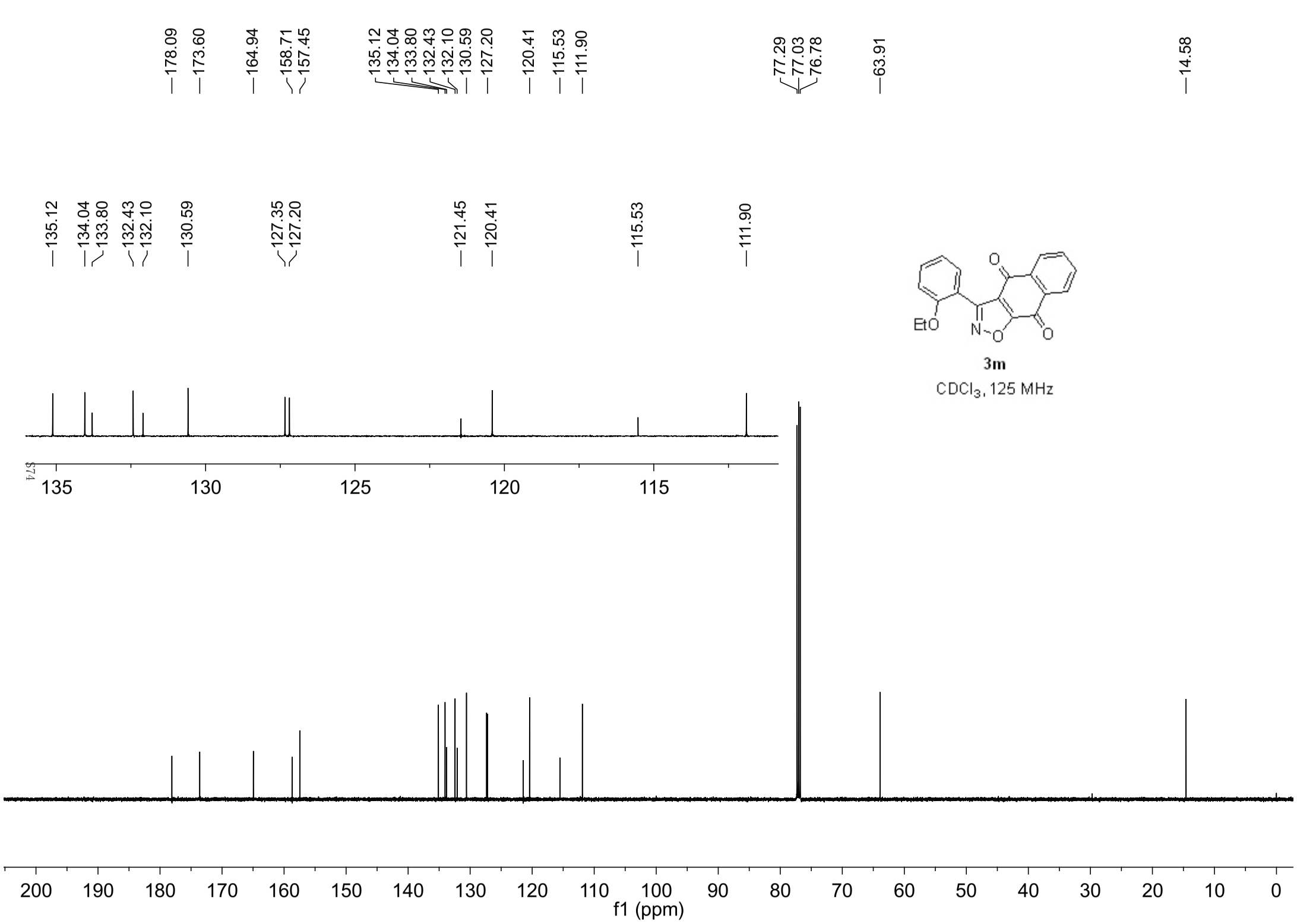

8.2968
8.2946
8.2798
8.2652
8.2633
8.1683
8.1655
8.1531
8.1495
7.8372
7.5657
7.2604
8.2968
8.2946
8.2798
8.2652
8.2633
8.1683
8.1655
8.1531
8.1495
7.8882
7.8854
7.8735
7.8705
7.8589
7.8552
7.8517
7.8400
7.8372
7.8252
7.8225
7.5936
7.5900
7.5776
7.5657
7.5505
7.5388
7.5329
Ph
O
O
N–O
3K
CDCl3, 500 MHz
S69
8.3
8.2
8.1
8.0
7.9
7.8
7.7
7.6
2.00
2.00
2.03
3.02
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

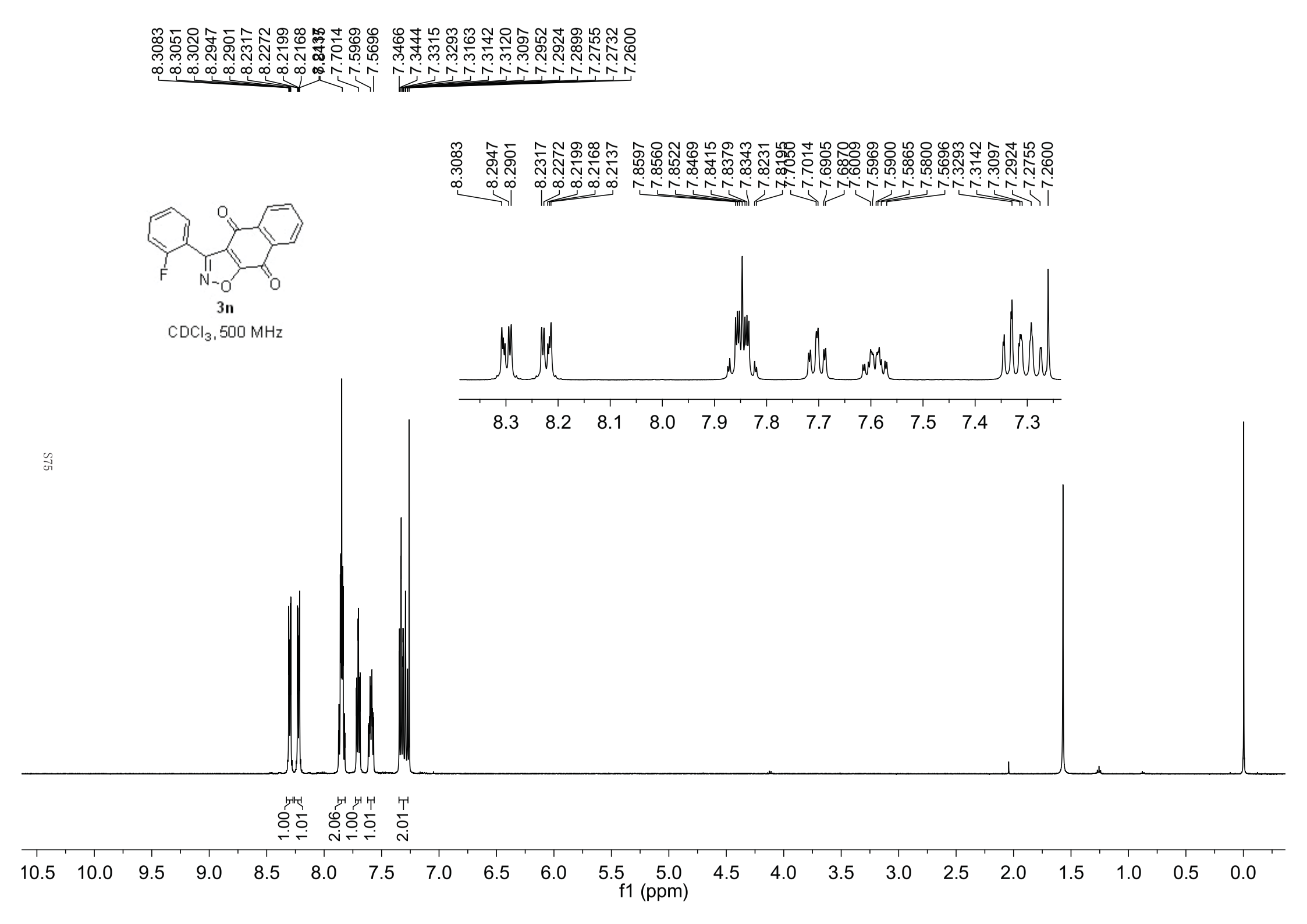

178.68
173.37
166.30
161.04
134.34
133.80
131.81
131.34
129.41
128.69
127.77
127.29
126.17
119.61
77.28
77.03
76.78
135.34
134.34
133.80
131.81
131.34
129.41
128.69
127.77
127.29
126.17
135
133
131
129
127
Ph
O
O
N
O
3K
CDCl3, 125 MHz
f1 (ppm)

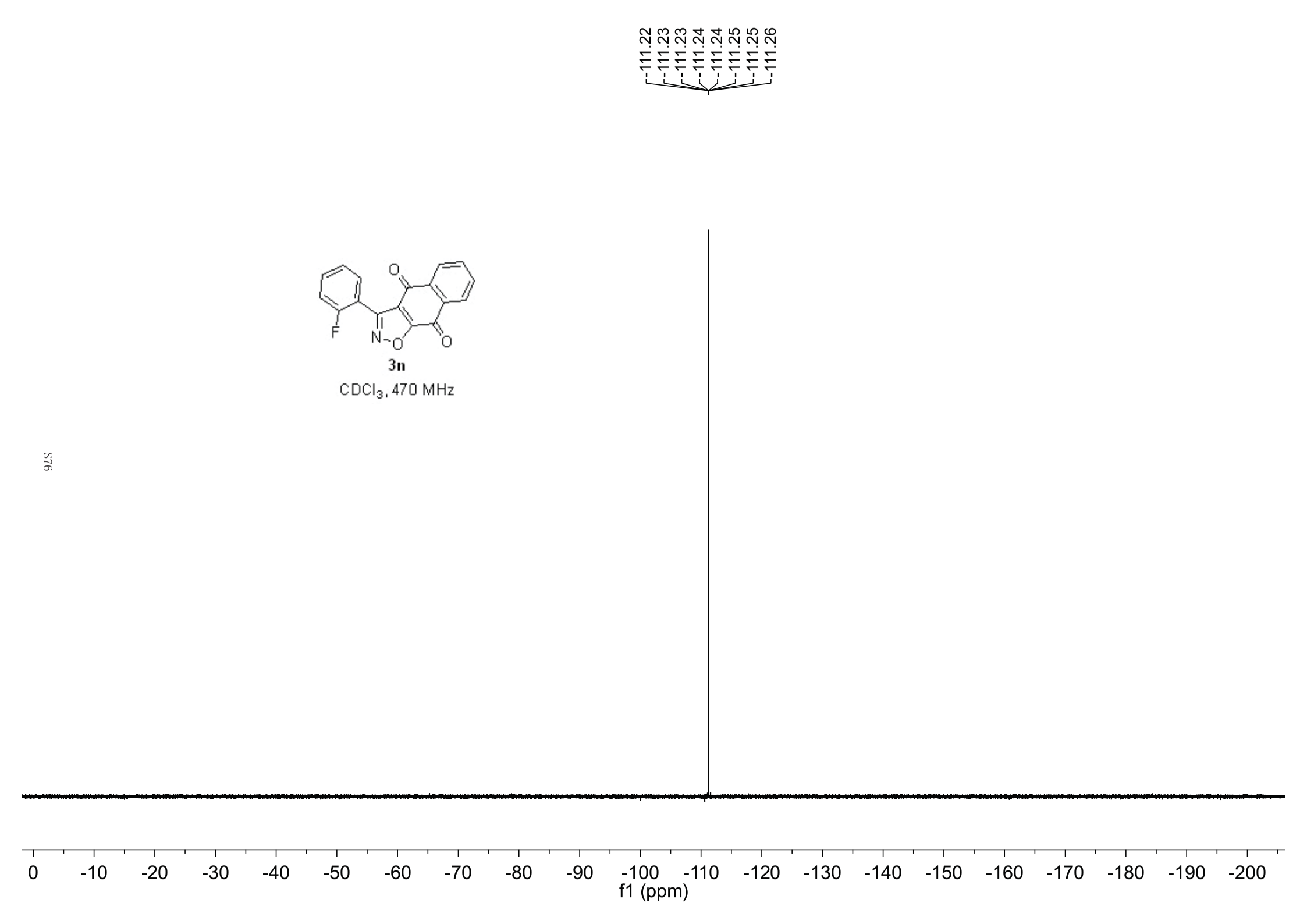

8.5767
8.4807
8.4649
8.2979
7.8554
7.7091
7.6933
7.6777
7.2602
1.5824
-0.0061
8.5767
8.4649
8.3123
8.3102
8.2979
8.2850
8.2834
7.9189
7.9065
7.9040
7.8885
7.8707
7.8554
7.7091
7.6933
7.6777
NC
O
N
O
O
3l
CDCl3, 500 MHz
8.7
8.5
8.3
8.1
7.9
7.7
1.00
1.00
2.00
3.09
1.01
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

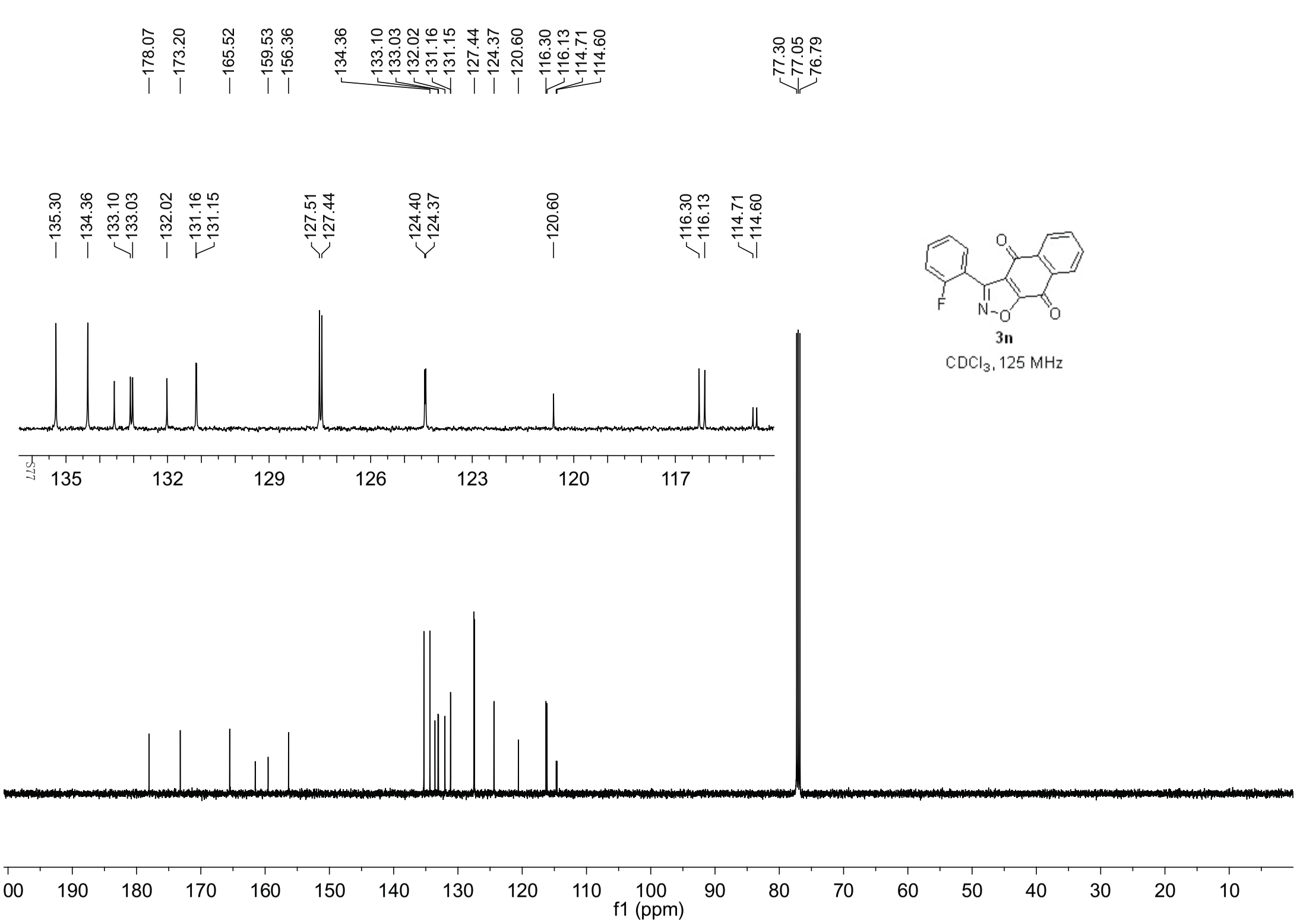

178.59
173.03
166.61
159.25
134.57
133.48
133.00
131.75
129.68
127.92
127.72
127.47
119.42
118.04
113.29
77.28
77.03
76.77
135.56
134.68
134.57
133.54
133.48
133.00
131.75
129.68
127.92
127.72
127.47
136
134
132
130
128
NC
O
N
O
O
3l
CDCl3, 125 MHz
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

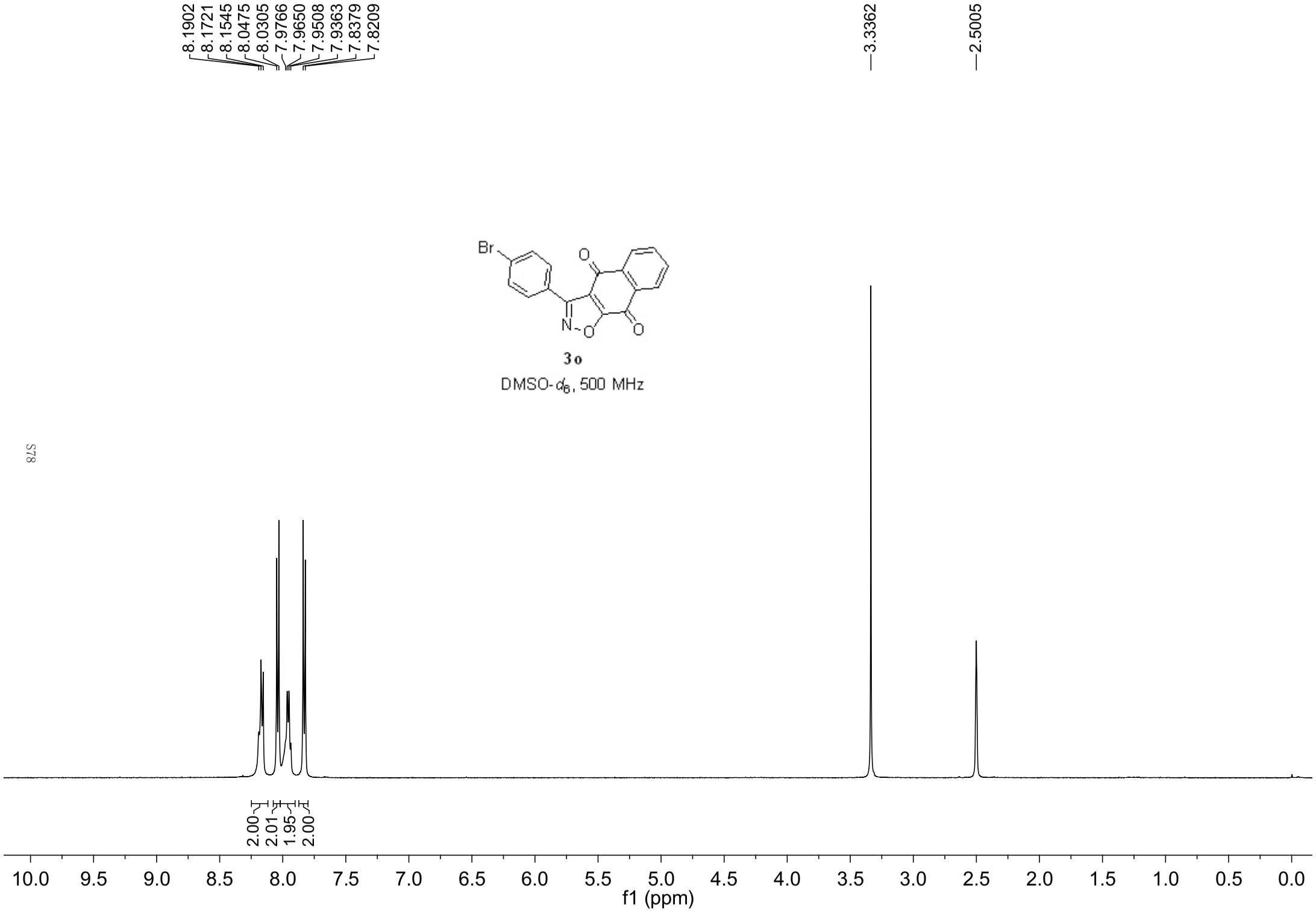

8.3084
8.2997
8.2966
8.2935
8.2865
8.2818
8.2718
8.2062
8.1964
8.1917
8.1846
8.1815
8.1784
7.7974
7.5032
7.2603
7.0863
7.0844
7.0715
7.0695
7.0586
7.0564
7.0543
7.0406
4.1139
4.1000
4.0861
4.0721
1.1916
1.1777
1.1637
EtO
N
O
O
O
3m
CDCl3, 500 MHz
1.00
1.00
2.02
1.99
2.01
2.00
3.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

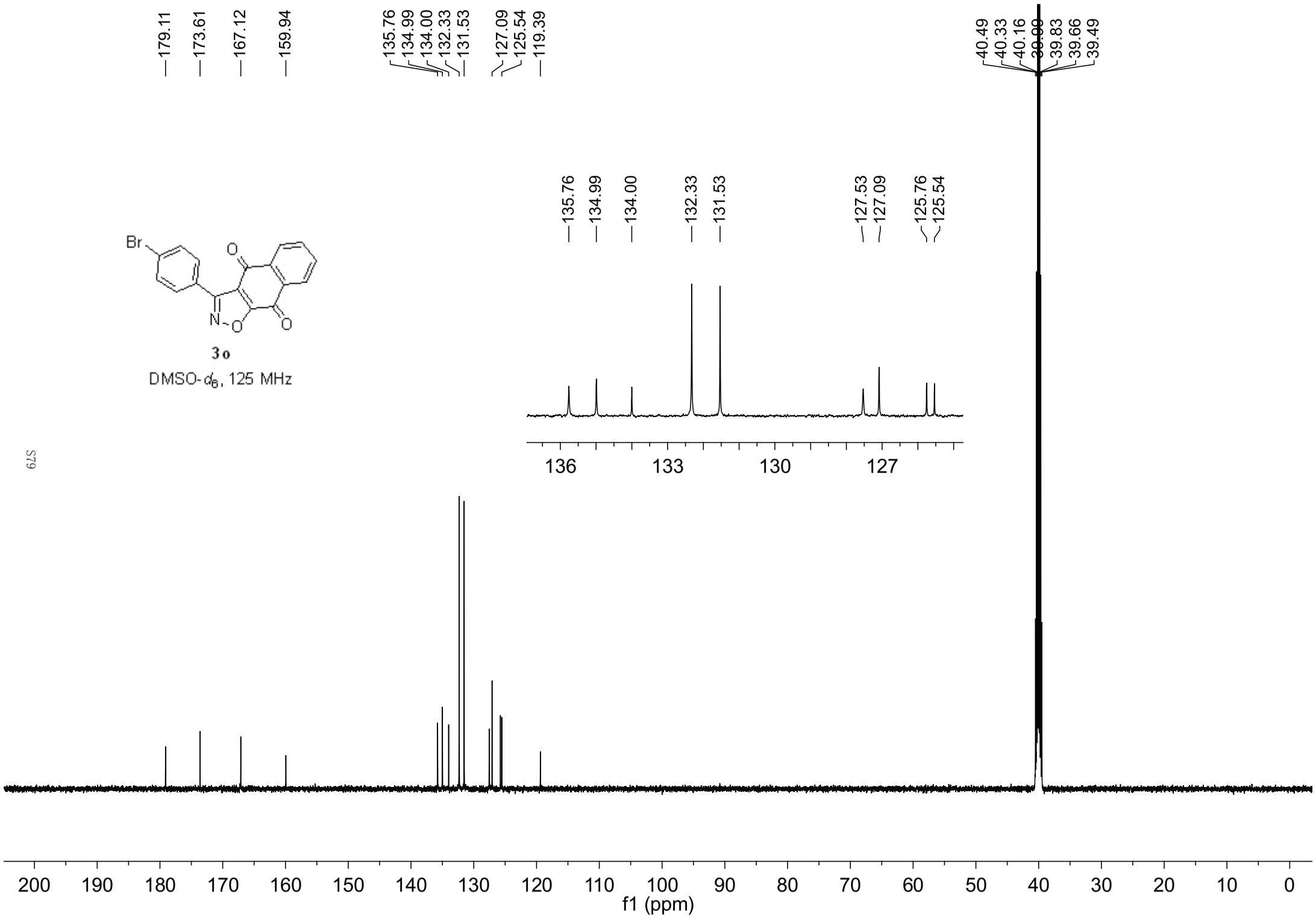

178.09
173.60
164.94
158.71
157.45
135.12
134.04
133.80
132.43
132.10
130.59
127.20
120.41
115.53
111.90
77.29
77.03
76.78
63.91
14.58
135.12
134.04
133.80
132.43
132.10
130.59
127.35
127.20
121.45
120.41
115.53
111.90
EtO
N
O
O
O
3m
CDCl3, 125 MHz
574
135
130
125
120
115
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

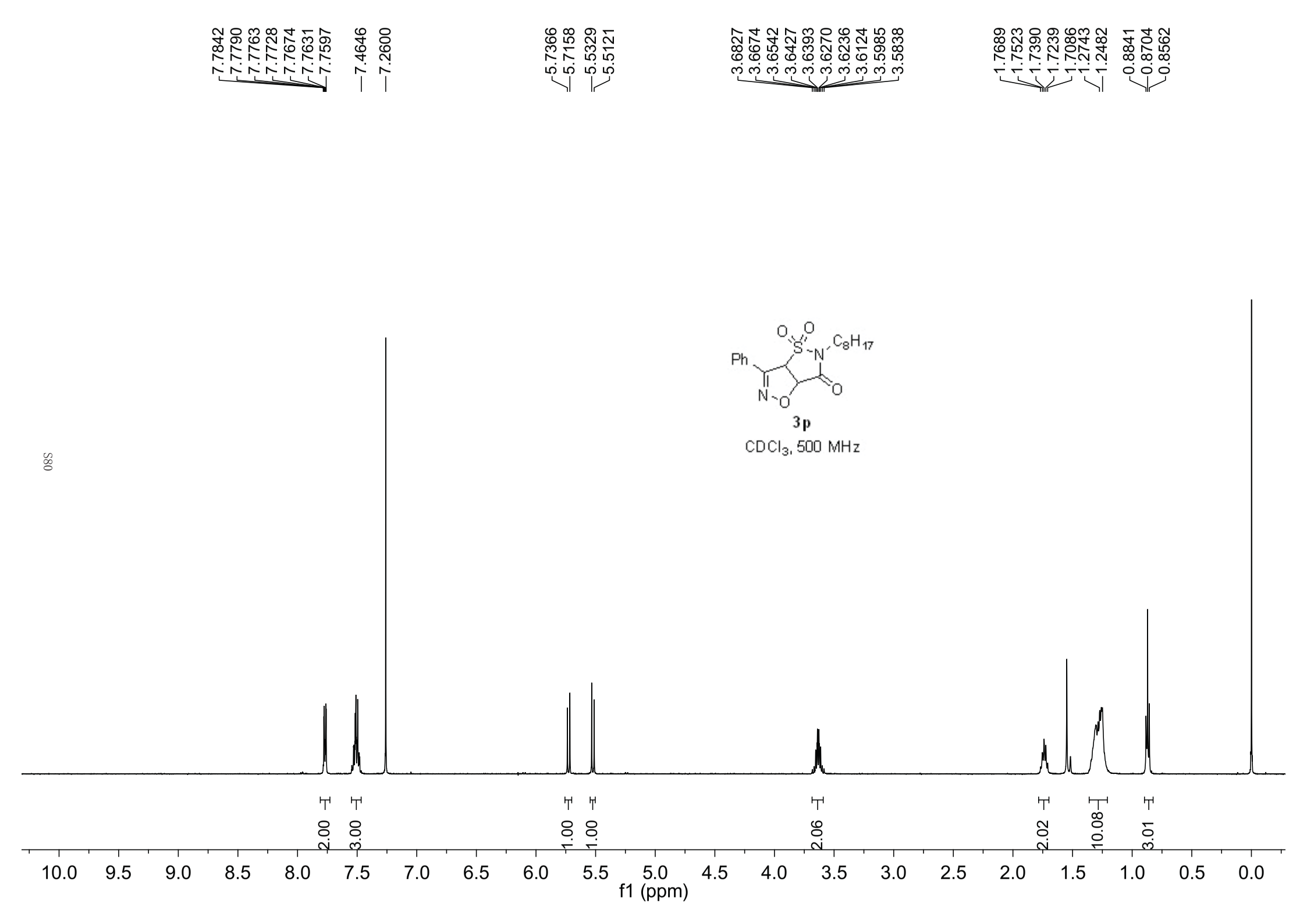

8.3083
8.3051
8.3020
8.2947
8.2901
8.2317
8.2272
8.2199
8.2168
7.7014
7.5969
7.5696
7.3466
7.3444
7.3315
7.3293
7.3163
7.3142
7.3120
7.3097
7.2952
7.2924
7.2899
7.2755
7.2732
7.2600
F
N
O
O
O
3n
CDCl3, 500 MHz
8.3083
8.2947
8.2901
8.2317
8.2272
8.2199
8.2168
8.2137
7.8597
7.8560
7.8522
7.8469
7.8415
7.8379
7.8343
7.8231
7.7014
7.6905
7.6870
7.6009
7.5969
7.5900
7.5865
7.5800
7.5696
7.3293
7.3142
7.3097
7.2924
7.2755
7.2600
8.3 8.2 8.1 8.0 7.9 7.8 7.7 7.6 7.5 7.4 7.3
1.00
1.01
2.06
1.00
1.01
2.01
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

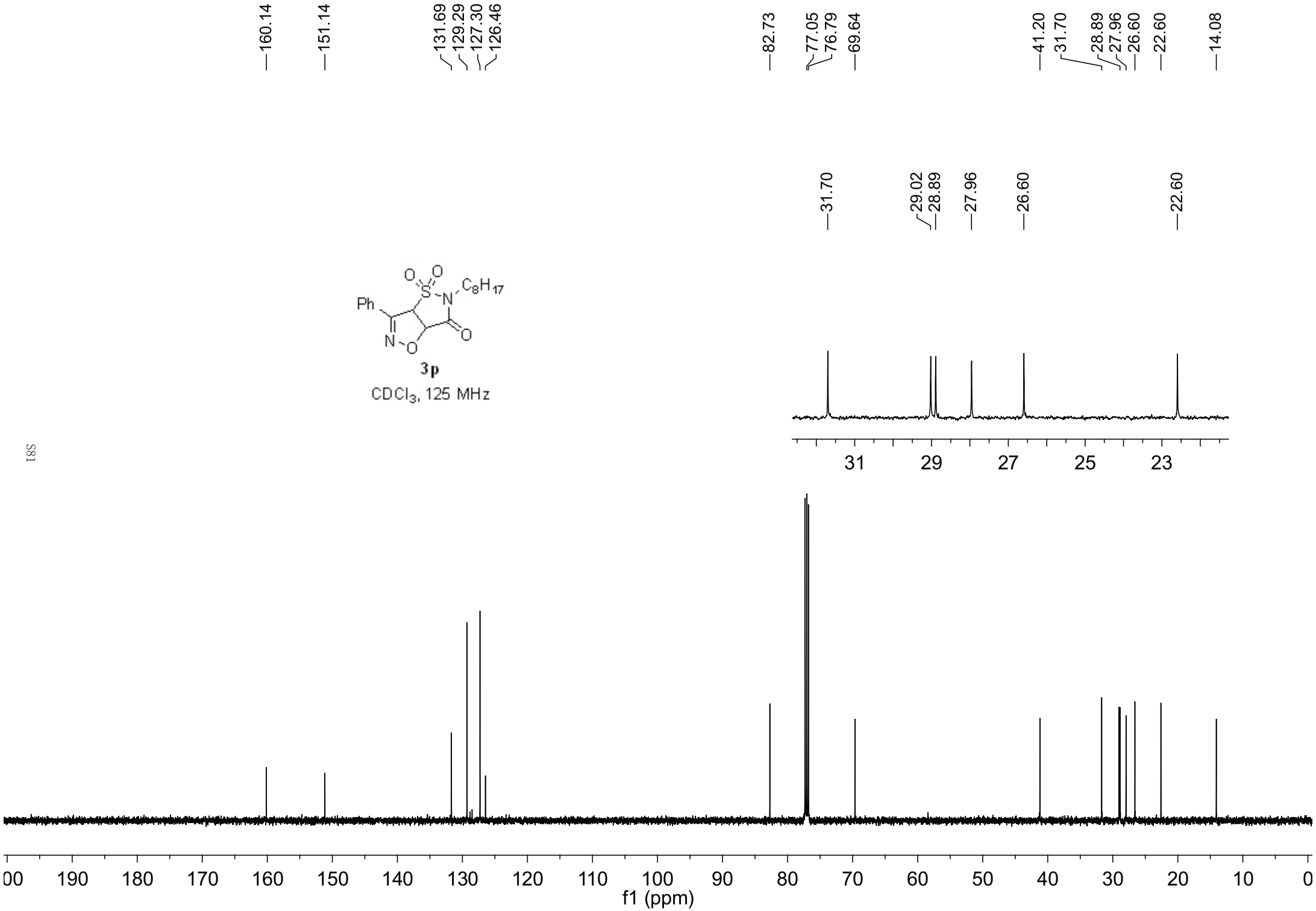

-111.22
-111.23
-111.23
-111.24
-111.24
-111.25
-111.25
-111.26
O
F
N
O
O
3n
CDCl3, 470 MHz
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
f1 (ppm)

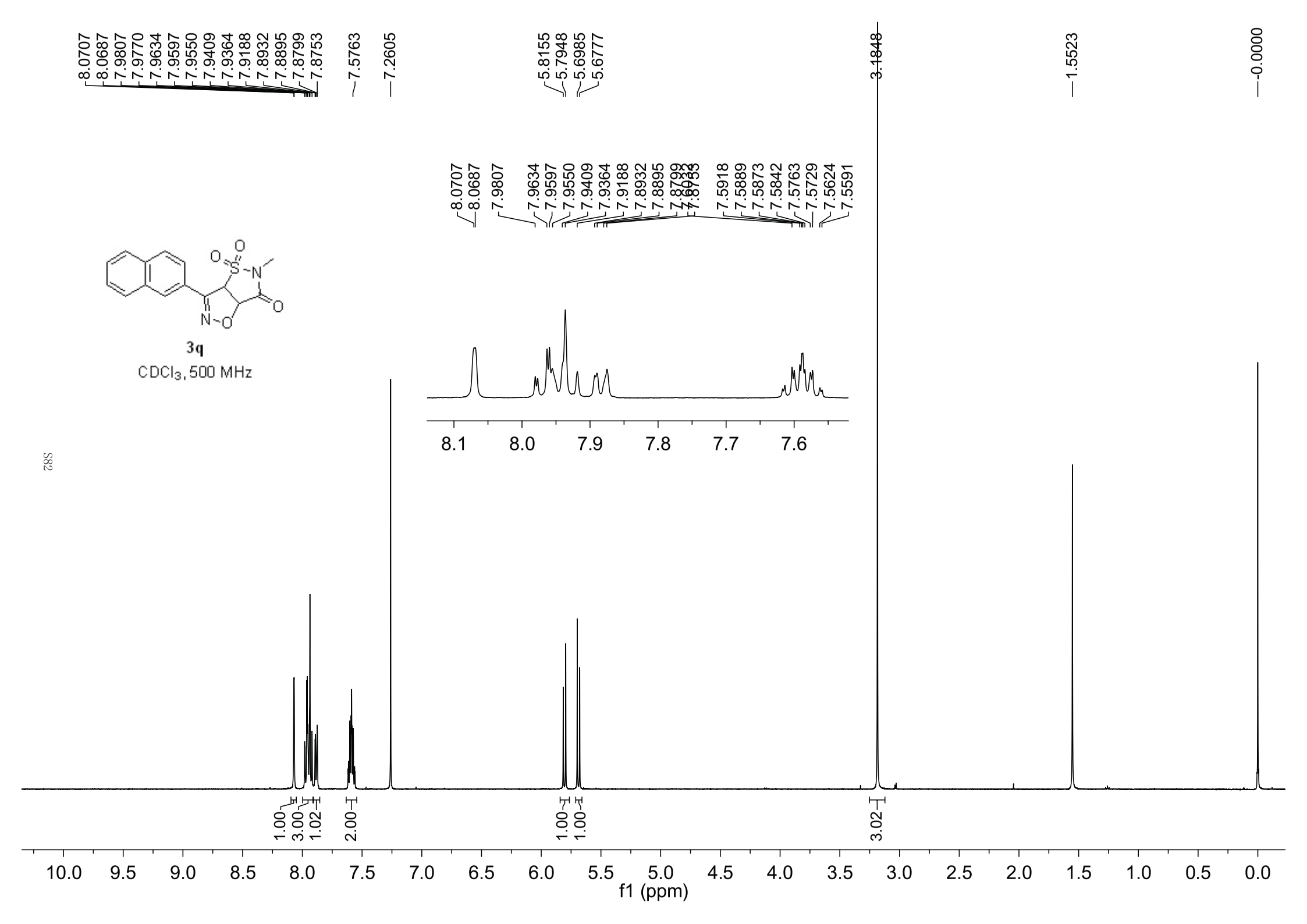

178.07
173.20
165.52
159.53
156.36
134.36
133.10
133.03
132.02
131.16
131.15
127.44
124.37
120.60
116.30
116.13
114.71
114.60
77.30
77.05
76.79
135.30
134.36
133.10
133.03
132.02
131.16
131.15
127.51
127.44
124.40
124.37
120.60
116.30
116.13
114.71
114.60
135
132
129
126
123
120
117
F
O
N
O
O
3n
CDCl3, 125 MHz
00
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

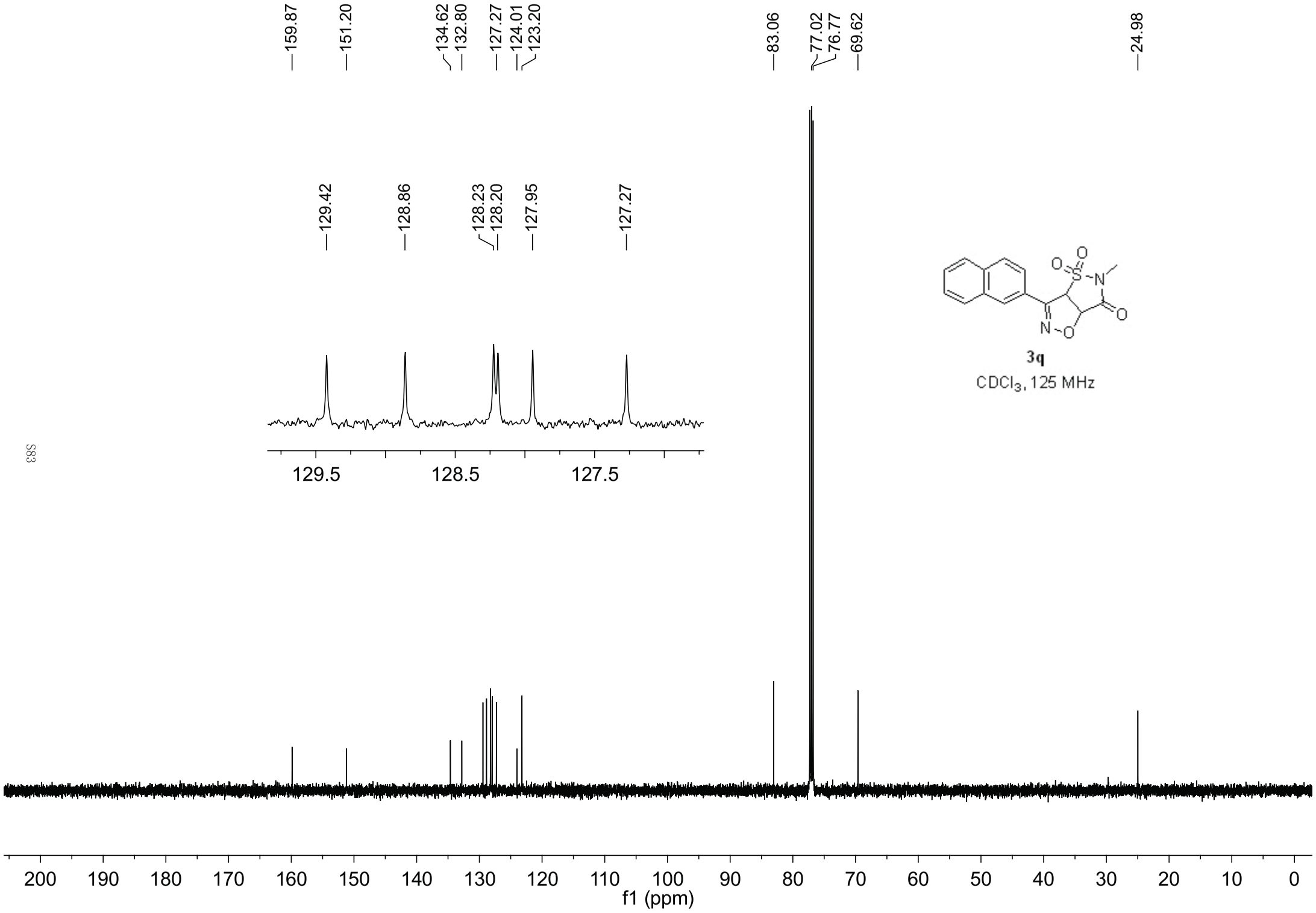

8.1902
8.1721
8.1545
8.0475
8.0305
7.9766
7.9650
7.9508
7.9363
7.8379
7.8209
3.3362
2.5005
Br
O
N
O
O
3o
DMSO-d_6, 500 MHz
2.00
2.01
1.95
2.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

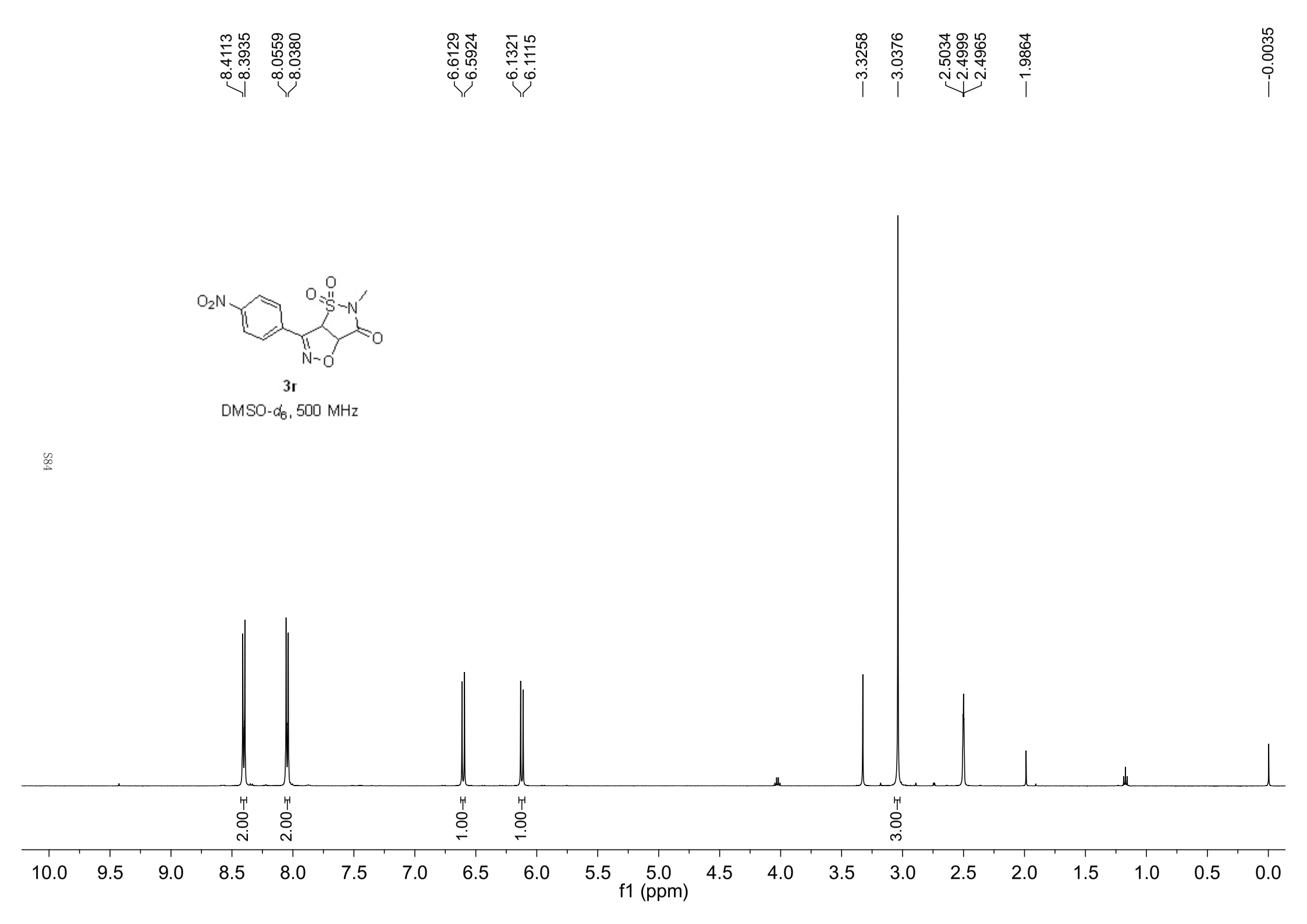

179.11
173.61
167.12
159.94
135.76
134.99
134.00
132.33
131.53
127.09
125.54
119.39
40.49
40.33
40.16
39.83
39.66
39.49
Br
O
N
O
O
3o
DMSO-d_6, 125 MHz
135.76
134.99
134.00
132.33
131.53
127.53
127.09
125.76
125.54
136
133
130
127
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

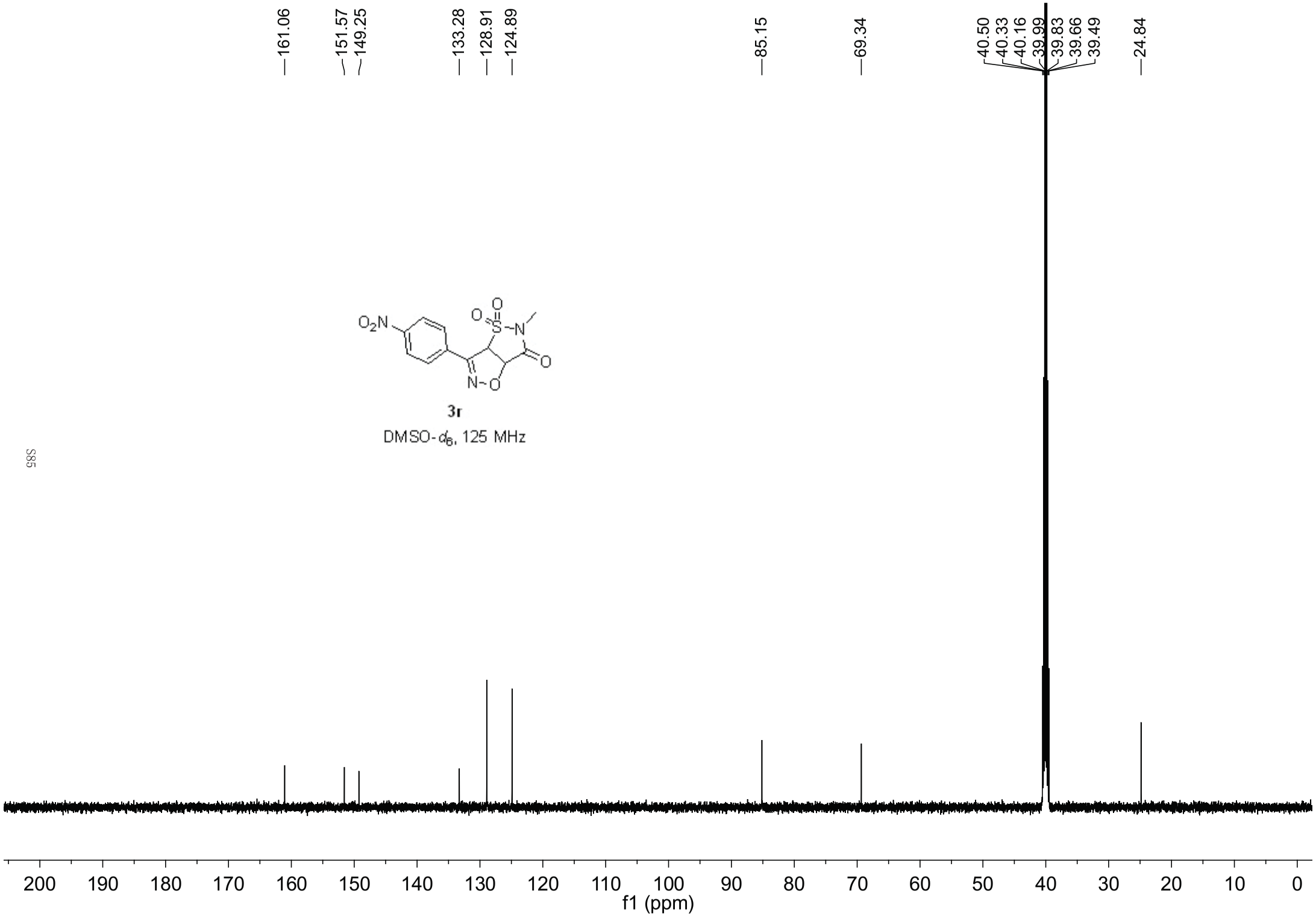

7.7842
7.7790
7.7763
7.7728
7.7674
7.7631
7.7597
7.4646
7.2600
5.7366
5.7158
5.5329
5.5121
3.6827
3.6674
3.6542
3.6427
3.6393
3.6270
3.6236
3.6124
3.5985
3.5838
1.7689
1.7523
1.7390
1.7239
1.7086
1.2743
1.2482
0.8841
0.8704
0.8562
Ph
C8H17
3p
CDCl3, 500 MHz
S80
2.00
3.00
1.00
1.00
2.06
2.02
10.08
3.01
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

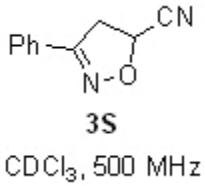

160.14
151.14
131.69
129.29
127.30
126.46
82.73
77.05
76.79
69.64
41.20
31.70
28.89
27.96
26.60
22.60
14.08
31.70
29.02
28.89
27.96
26.60
22.60
Ph
C8H17
3p
CDCl3, 125 MHz
31
29
27
25
23
f1 (ppm)


8.0707
8.0687
7.9807
7.9770
7.9634
7.9597
7.9550
7.9409
7.9364
7.9188
7.8932
7.8895
7.8799
7.8753
7.5763
7.2605
5.8155
5.7948
5.6985
5.6777
3.1848
1.5523
-0.0000
8.0707
8.0687
7.9807
7.9634
7.9597
7.9550
7.9409
7.9364
7.9188
7.8932
7.8895
7.8799
7.8733
7.8733
7.5918
7.5889
7.5873
7.5842
7.5763
7.5729
7.5624
7.5591
8.1
8.0
7.9
7.8
7.7
7.6
3q
CDCl3, 500 MHz
S82
1.00
3.00
1.02
2.00
1.00
1.00
3.02
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


159.87
151.20
134.62
132.80
127.27
124.01
123.20
83.06
77.02
76.77
69.62
24.98
129.42
128.86
128.23
128.20
127.95
127.27
129.5
128.5
127.5
3q
CDCl3, 125 MHz
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


8.4113
8.3935
8.0559
8.0380
6.6129
6.5924
6.1321
6.1115
3.3258
3.0376
2.5034
2.4999
2.4965
1.9864
-0.0035
O2N
O
O
S
N
O
N
O
3r
DMSO-d6, 500 MHz
2.00
2.00
1.00
1.00
3.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


161.06
151.57
149.25
133.28
128.91
124.89
85.15
69.34
40.50
40.33
40.16
39.99
39.83
39.66
39.49
24.84
O2N
O
S
O
N
O
N
O
3r
DMSO-d6, 125 MHz
S85
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)



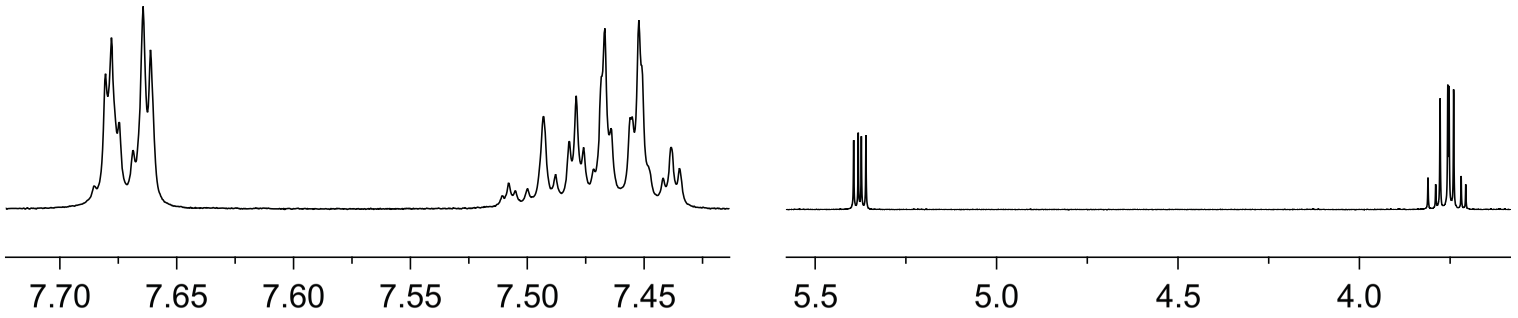
3S

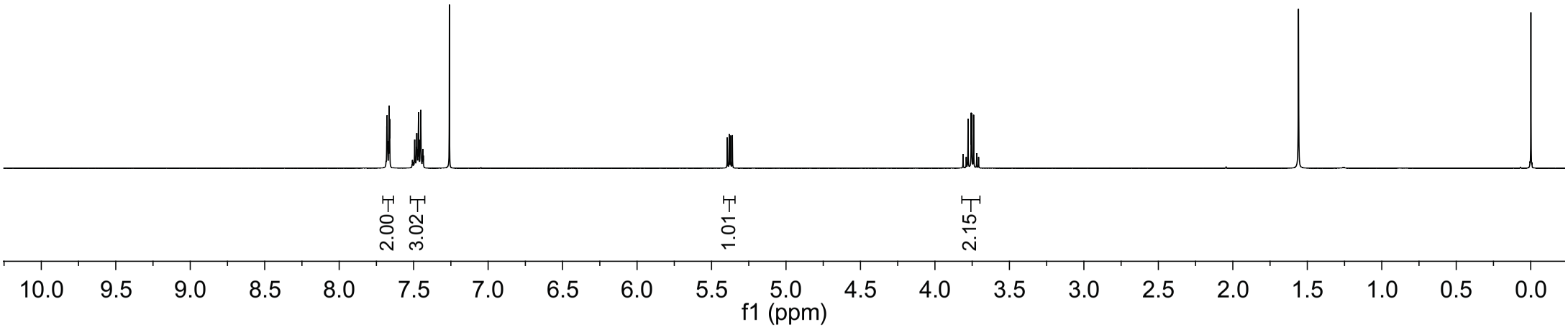
$CDCl_3$, 500 MHz

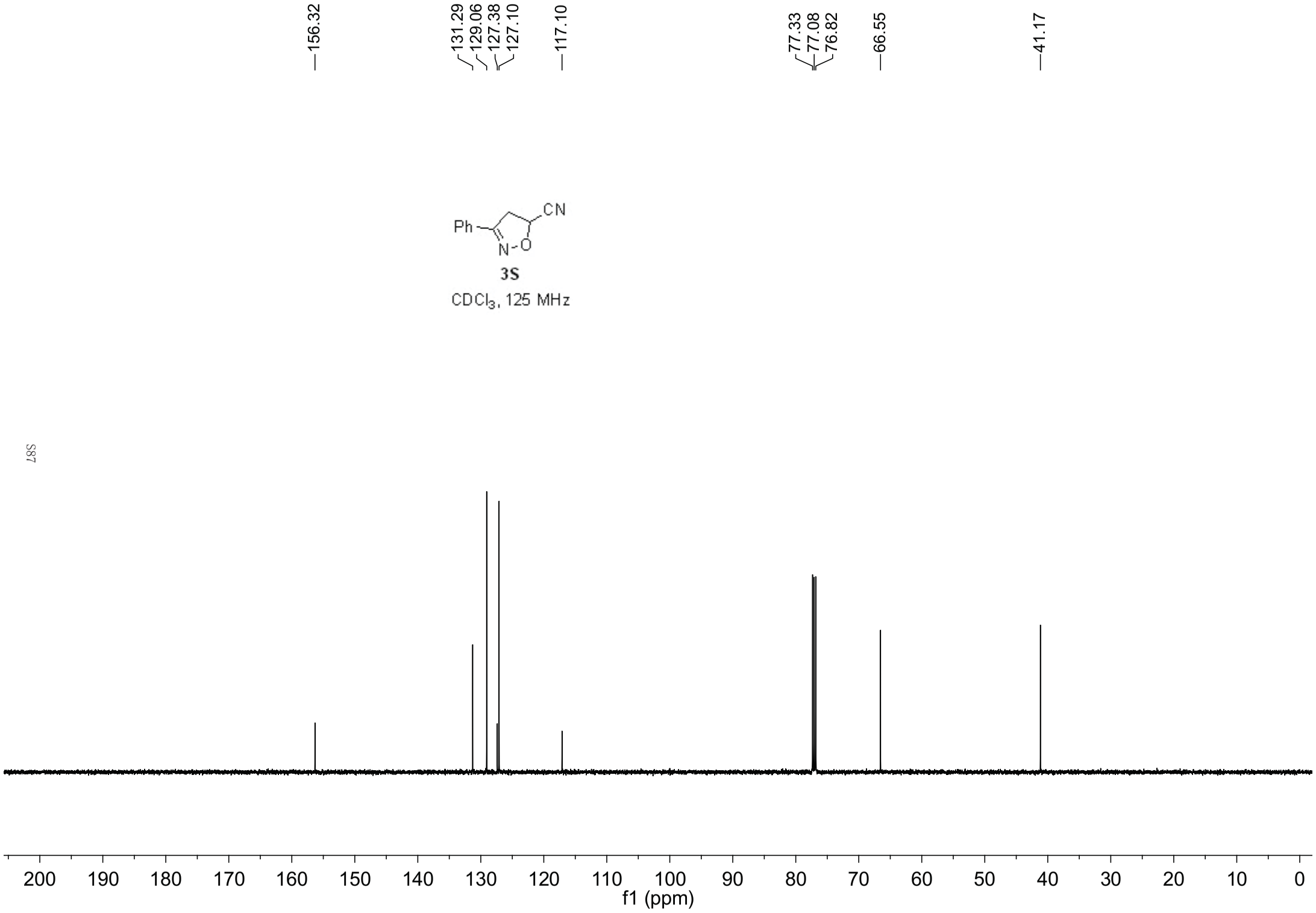
7.6854 7.6806 7.6779 7.6745 7.6688 7.6644 7.6611 7.4523 7.2601

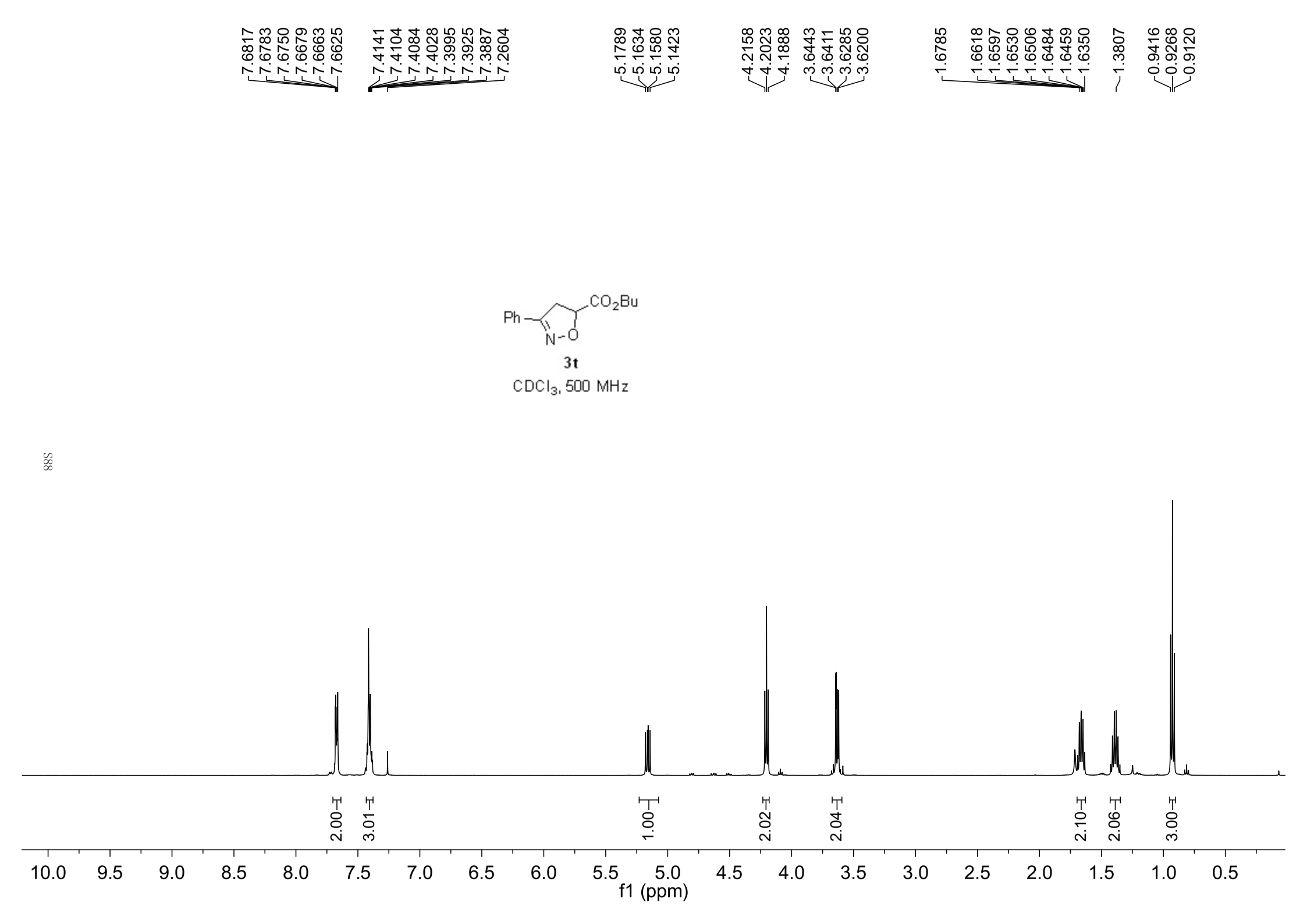
5.3939 5.3813 5.3727 5.3601

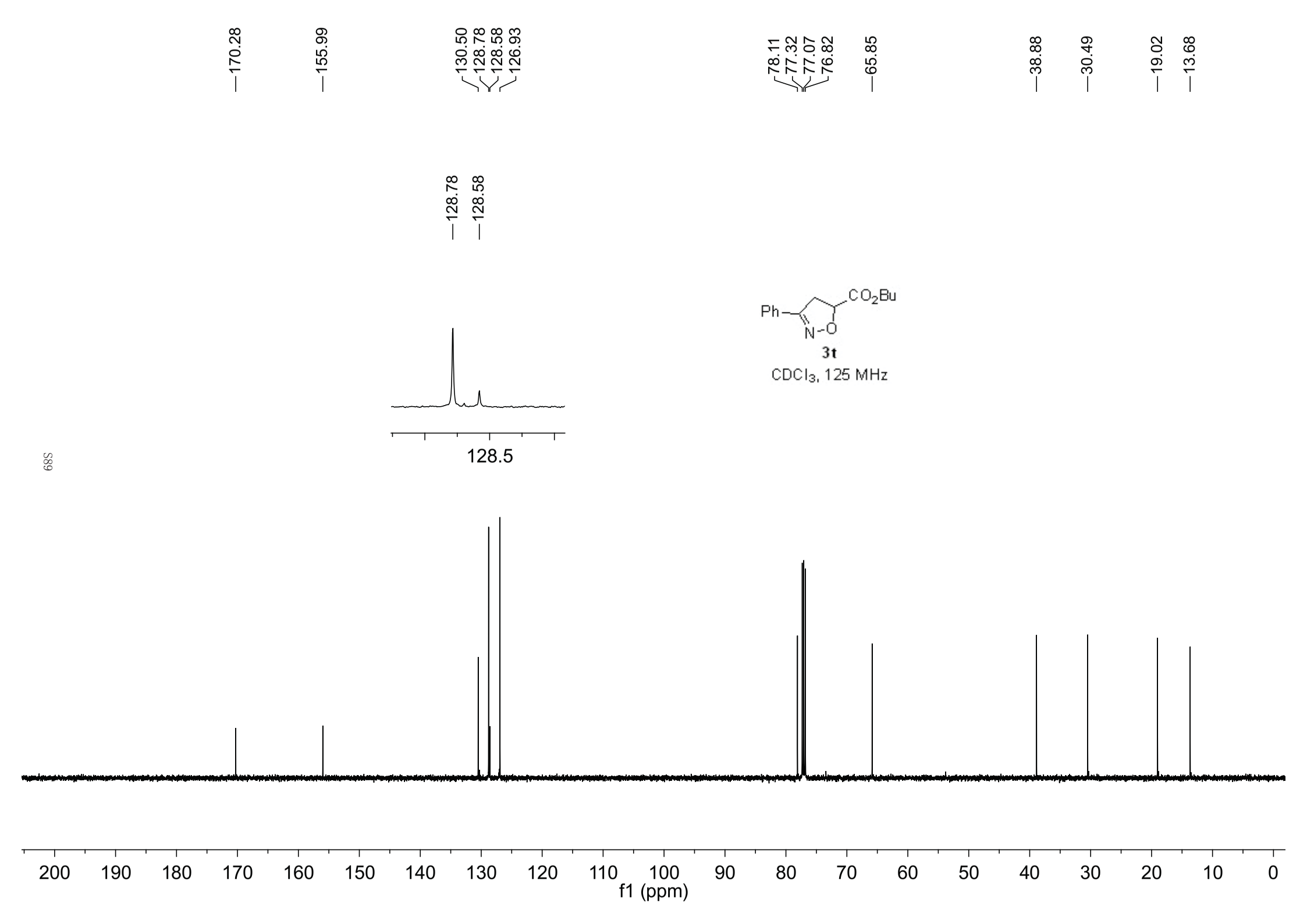
3.8110 3.7898 3.7777 3.7565 3.7529 3.7489 3.7403 3.7196 3.7070

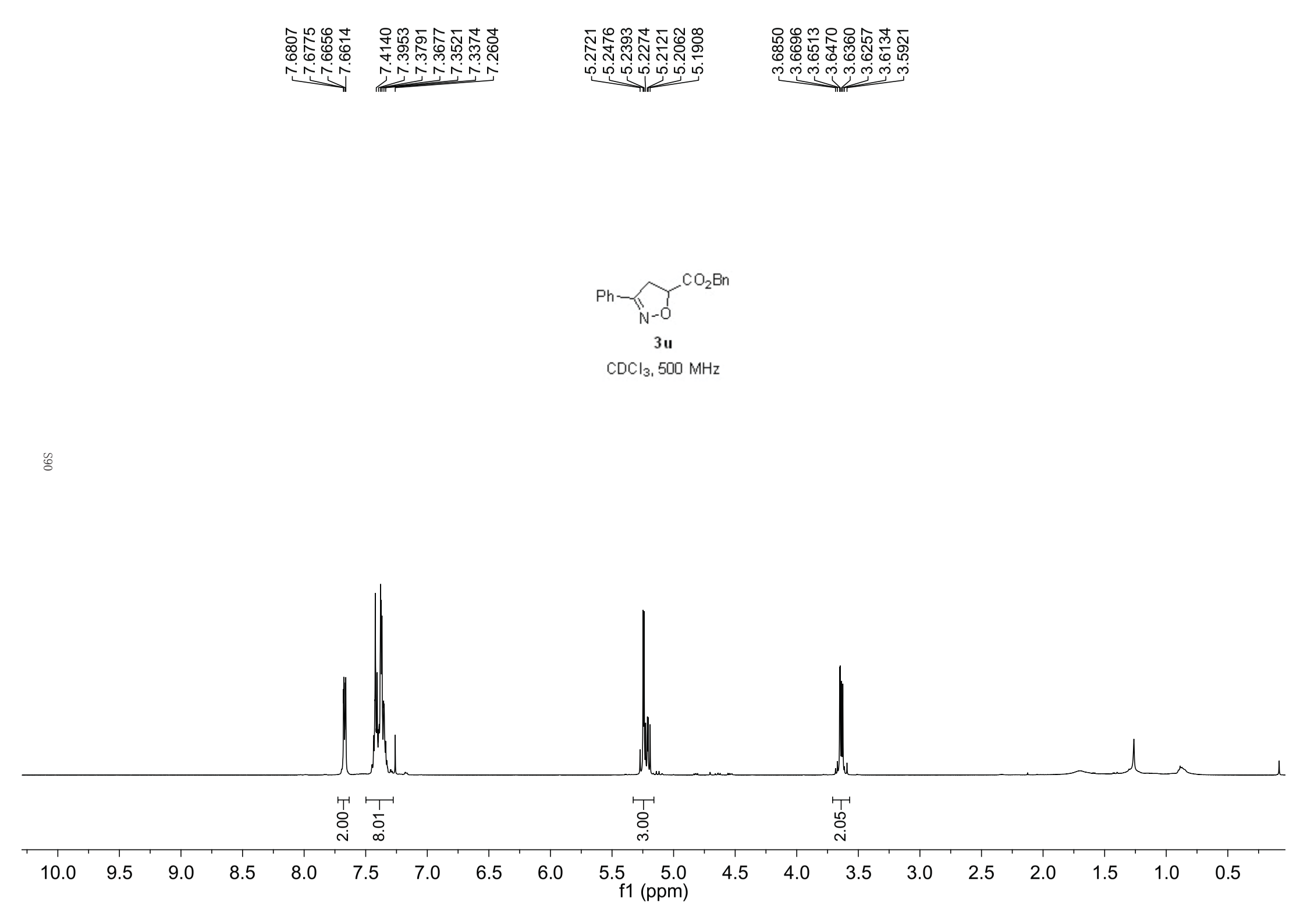


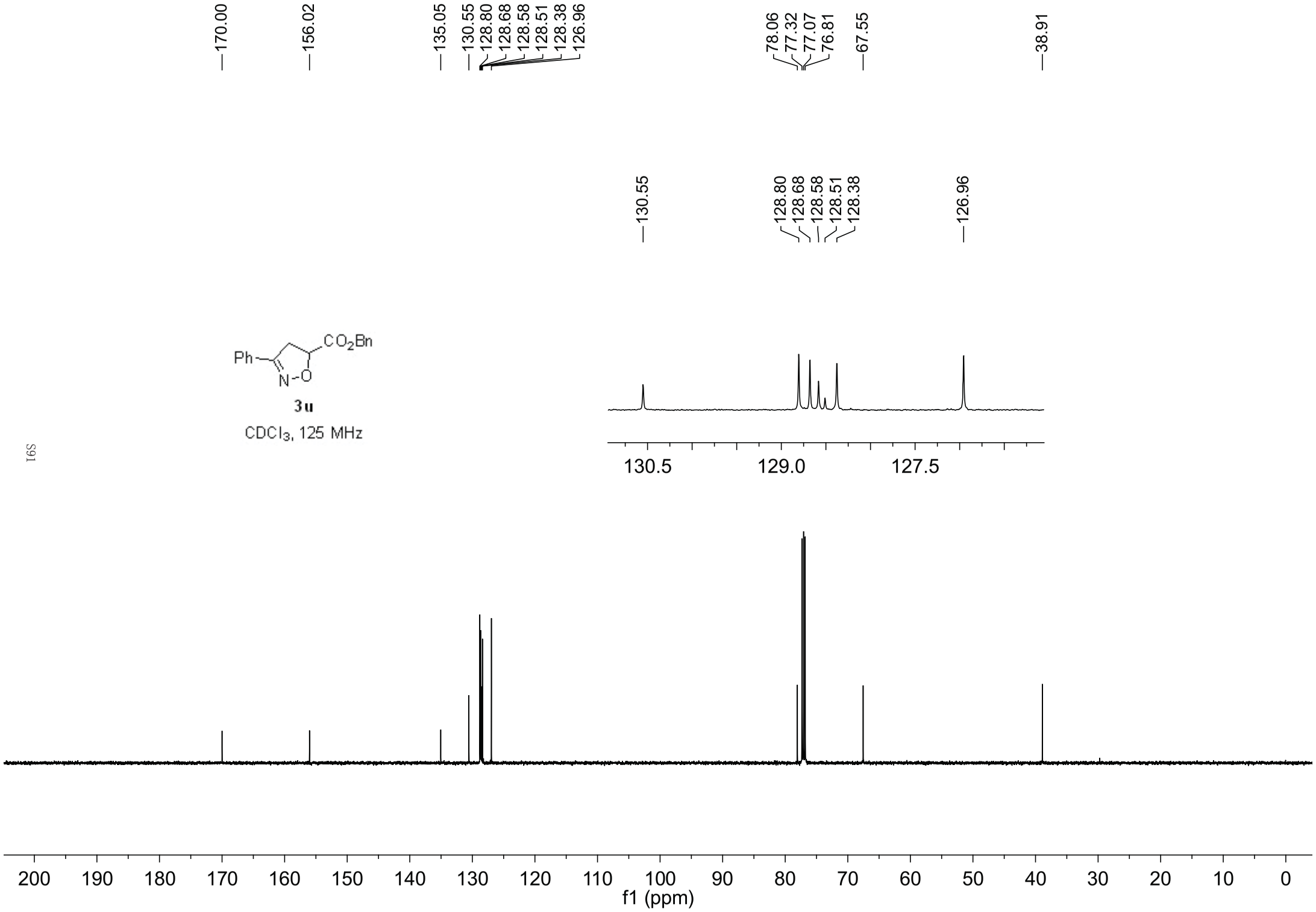


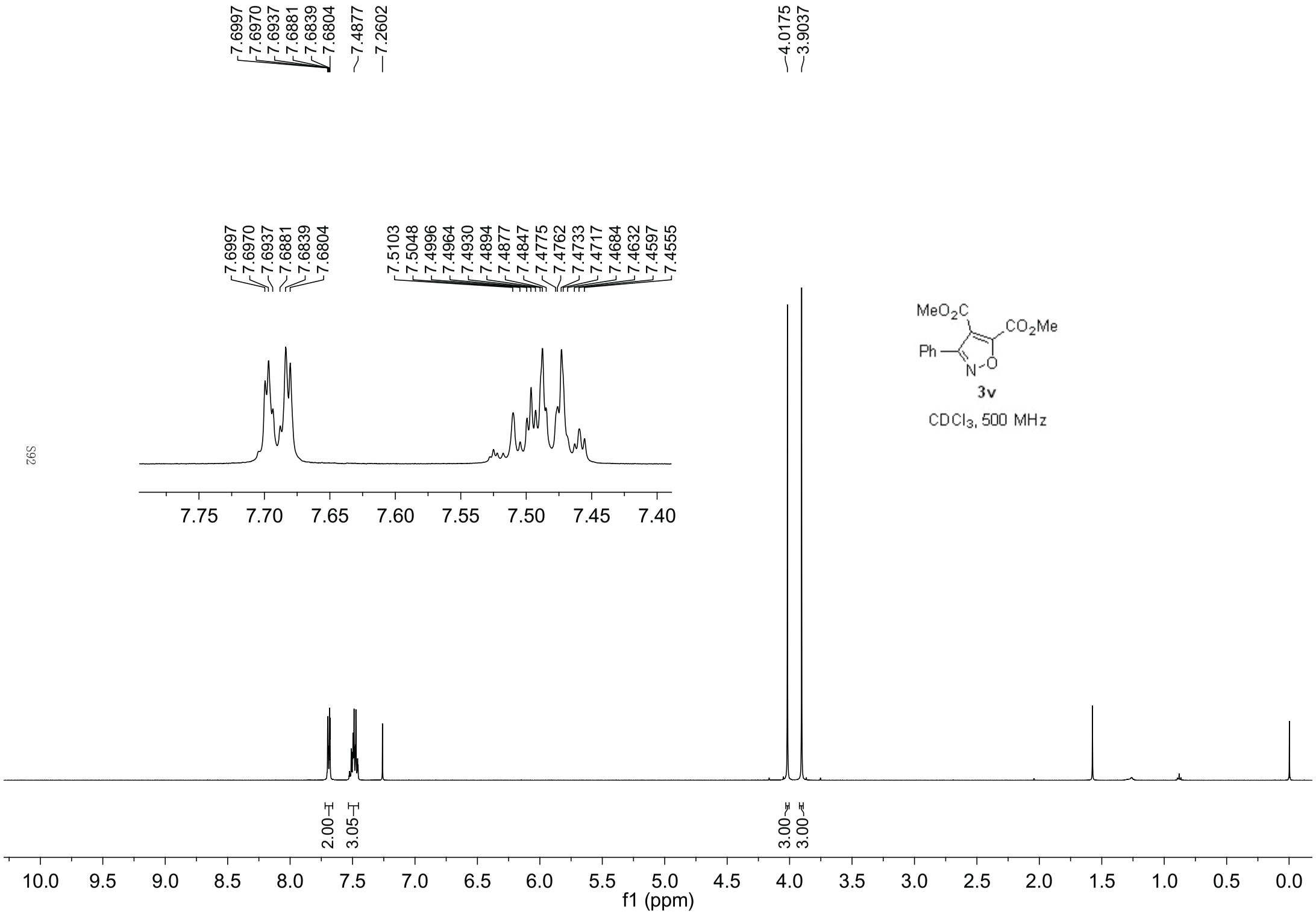

156.32
131.29
129.06
127.38
127.10
117.10
77.33
77.08
76.82
66.55
41.17
Ph
CN
N
O
3S
CDCl3, 125 MHz
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

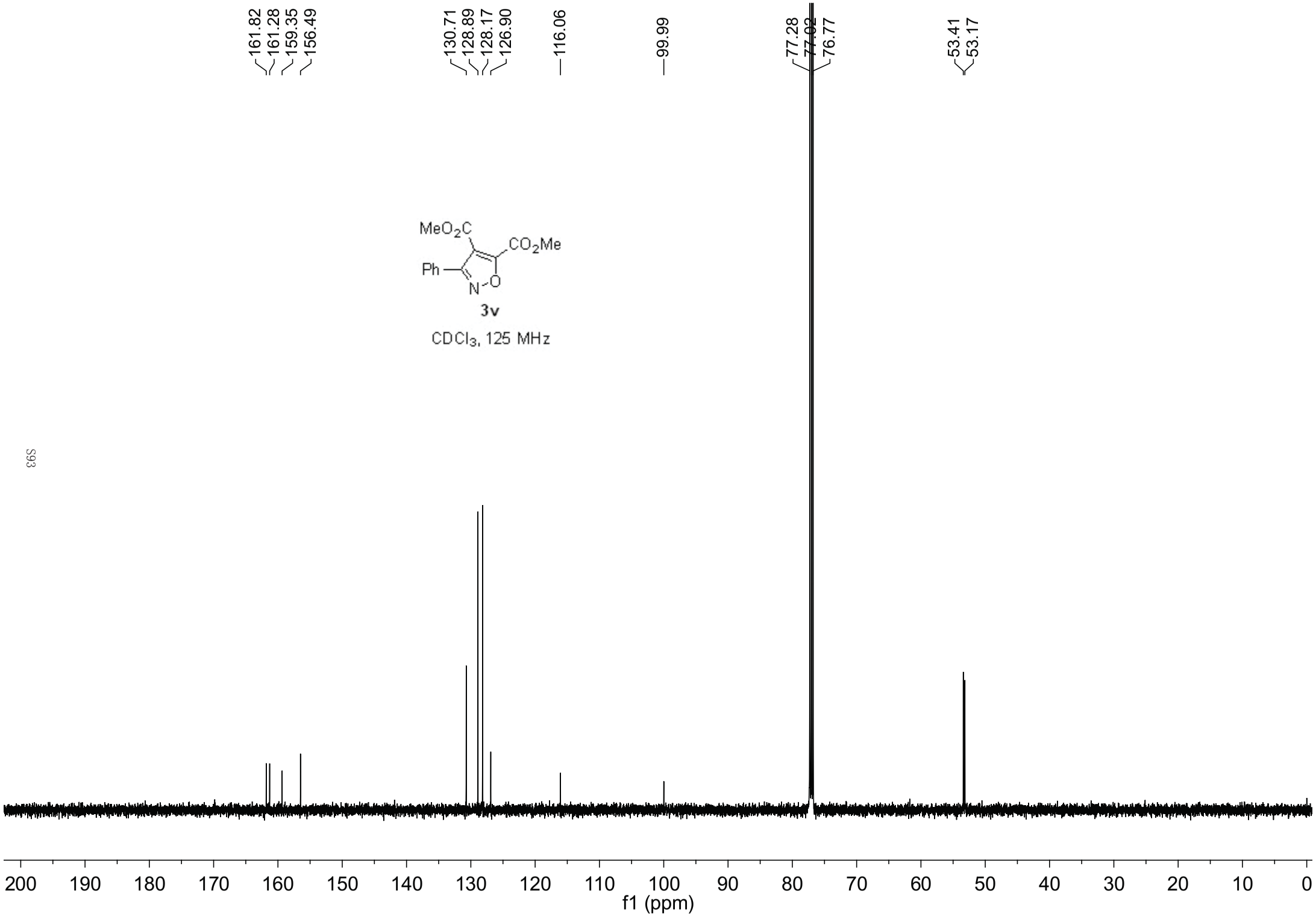

7.6817
7.6783
7.6750
7.6679
7.6663
7.6625
7.4141
7.4104
7.4084
7.4028
7.3995
7.3925
7.3887
7.2604
5.1789
5.1634
5.1580
5.1423
4.2158
4.2023
4.1888
3.6443
3.6411
3.6285
3.6200
1.6785
1.6618
1.6597
1.6530
1.6506
1.6484
1.6459
1.6350
1.3807
0.9416
0.9268
0.9120
Ph
CO2Bu
N
O
3t
CDCl3, 500 MHz
S88
2.00
3.01
1.00
2.02
2.04
2.10
2.06
3.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)


170.28
155.99
130.50
128.78
128.58
126.93
78.11
77.32
77.07
76.82
65.85
38.88
30.49
19.02
13.68
128.78
128.58
128.5
Ph
CO2Bu
N
O
3t
CDCl3, 125 MHz
S89
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.6807
7.6775
7.6656
7.6614
7.4140
7.3953
7.3791
7.3677
7.3521
7.3374
7.2604
5.2721
5.2476
5.2393
5.2274
5.2121
5.2062
5.1908
3.6850
3.6696
3.6513
3.6470
3.6360
3.6257
3.6134
3.5921
Ph
CO2Bn
N
O
3u
CDCl3, 500 MHz
2.00
8.01
3.00
2.05
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)


170.00
156.02
135.05
130.55
128.80
128.68
128.58
128.51
128.38
126.96
78.06
77.32
77.07
76.81
67.55
38.91
130.55
128.80
128.68
128.58
128.51
128.38
126.96
Ph
CO2Bn
N
O
3u
CDCl3, 125 MHz
130.5
129.0
127.5
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.6997
7.6970
7.6937
7.6881
7.6839
7.6804
7.4877
7.2602
4.0175
3.9037
7.5103
7.5048
7.4996
7.4964
7.4930
7.4894
7.4877
7.4847
7.4775
7.4762
7.4733
7.4717
7.4684
7.4632
7.4597
7.4555
7.75
7.70
7.65
7.60
7.55
7.50
7.45
7.40
MeO2C
CO2Me
Ph
N
O
3v
CDCl3, 500 MHz
2.00
3.05
3.00
3.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


161.82
161.28
159.35
156.49
130.71
128.89
128.17
126.90
116.06
99.99
77.28
77.02
76.77
53.41
53.17
MeO2C
CO2Me
Ph
N
O
3v
CDCl3, 125 MHz
f1 (ppm)