Electronic Supplementary Material (ESI) for Chemical Science.

Tandem Aza-Heck Suzuki and Carbonylation Reactions of *O*-Phenyl Hydroxamic Ethers: Complex Lactams via Carboamination

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Supporting Information

Table of Contents

1. General Experimental Details. Toluene, dioxane, and acetonitrile were dried on alumina according to a published procedure.[1] Tris(2,2,2-trifluoroethyl) phosphite were purchased from Sigma-Aldrich, distilled from P_2O_5, sparged with N_2, and stored under N_2 in a sealed vessel. (1,5-cyclooctadiene)bis[(trimethylsilyl)methyl]palladium [(COD)Pd($CH_2SiMe_3)_2$] was prepared according to literature procedure,[2] and stored in a sealed container of DrieRite at –30 °C when not in use. 4Å MS powder was activated at 150 °C under vacuum for 1d and stored in the oven when not in use. All hot glassware was oven dried for a minimum of two hours or flame-dried under vacuum prior to use. The following substrates were synthesized according to published procedures: 2-(prop-1-en-2-yl)benzoic acid;[3] 2-(1-phenylvinyl)benzoic acid;[3] 4-chloro-2-(prop-1-en-2-yl)benzoic acid;[3] 4-methylpent-4-enoic acid;[3] 2,2,4-trimethylpent-4-enoic acid;[3] 4-methyl-2-phenylpent-4-enoic acid;[3] 2-(1-cyclopropylvinyl)benzoic acid;[3] 2-(2'-methylallyl)benzoic acid;[3] 1-(2-methylallyl)cyclobutanecarboxylic acid;[4] 2-(2-methylenecyclopentyl)acetic acid;[5] (*Z*)-N-phenoxy-2-styrylbenzamide;[6] (*E*)-N-phenoxy-2-styrylbenzamide.[6] All other substrates and reagents were purchased in highest analytical purity from commercial suppliers and used as received. Reaction optimization was conducted in a glovebox (N_2 atmosphere) on a 0.2 mmol scale in 2-dram vials with Teflon lined caps and heated in an aluminum block with stirring. All NMR chemical yields are reported using 1,3,5-trimethoxybenzene as an internal standard. All other reactions were set up using standard Schlenk technique and heated with stirring in temperature-controlled oil baths.

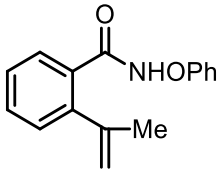
2. Instrumentation and Chromatography

400 MHz ^{1}H, 100 MHz ^{13}C and 376 MHz ^{19}F spectra were obtained on a 400 MHz FT-NMR spectrometer equipped with a Bruker CryoPlatform. 600 MHz ^{1}H, 150 MHz ^{13}C, and 564 MHz ^{19}F spectra were obtained on a 600 MHz FT-NMR spectrometer equipped with a Bruker SMART probe. All samples were analyzed in the indicated deutero-solvent and were recorded at ambient temperatures. All chemical shifts are reported in ppm. ^{1}H NMR spectra were calibrated using the residual protio-signal in deutero-solvents as a standard. ^{13}C NMR spectra were calibrated using the deutero-solvent as a standard. IR spectra were recorded on a Nicolet Magma-IR 560 FT-IR spectrometer as thin films on KBr plates. High resolution MS data was obtained on a Thermo Q-Exactive Orbitrap using electrospray ionization (ESI). Unless otherwise noted, column chromatography was performed either by hand or by use of Isolera 4 Biotage unit with 40-63 μm silica gel, and the eluent reported in parentheses. Analytical thin-layer chromatography (TLC) was performed on precoated glass plates and visualized by UV or by staining ($KMnO_4$ or I_2).

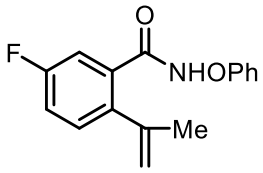
3. Synthesis of the *O*-Phenyl Hydroxamates

Note: All yields in this section are unoptimized

General Protocol: A flame-dried round-bottom flask, equipped with magnetic stir bar and rubber septum was purged with a stream of nitrogen until cool. The septum was quickly removed, and the carboxylic acid (1.0 equiv) was added. Subsequent addition of anhydrous DMF (0.3 M) and diisopropylethyl amine (DIEA, 3.3 equiv) gave a clear-to-pale yellow solution, followed by replacement of the septa. The flask was cooled to 0 °C in an ice bath, and the septa was then briefly removed to allow for solid addition of *O*-(7-azabenzotriazol-1-yl)-*N,N,N′,N′*-tetramethyluronium hexafluorophosphate (HATU, 1.1 equiv). The reaction was stirred at 0 °C for 30 minutes, and *O*-phenylhydroxylamine hydrochloride was added. The reaction was stirred at 0 °C for 4 hours, and then warm up to room temperature upon completion (as monitored by TLC), the reaction was quenched with brine (4X reaction volume) and diluted with EtOAc (4X reaction volume). The resulting biphasic mixture was poured into a separatory funnel and the layers were separated. The organic layer was washed with brine, dried with $MgSO_4$, filtered through a glass frit, and concentrated in vacuo to give crude product. The product was purified by flash column chromatography on silica gel (5-20 μm particle size), recrystallization, or both to yield pure product.

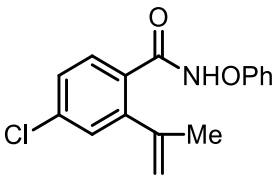


(**1**) According to the general protocol: 2-(prop-1-en-2-yl)benzoic acid (2.44 g, 15 mmol) and DIEA (8.2 mL, 49.5 mmol) were dissolved in DMF (45 mL) and stirred at 0 °C for 10 minutes. HATU (6.28 g, 16.5 mmol) was added, stirred at 0 °C for 30 minutes, and then *O*-phenylhydroxylamine hydrochloride (2.40 g, 16.5 mmol) was added. The reaction stirred for 4 hours, slowly warming to room temperature for overnight, and was worked up according to the general procedure. Purification by chromatography (linear gradient, 5:95 acetone:hexanes to 10:90 acetone:hexanes) and recrystallization in EtOAc afforded **1** (2.72 g, 72%) as a white solid. ^{1}H NMR (600 MHz, DMSO-d_6) δ 12.10 (s, 1H), 7.54-7.49 (m, 2H), 7.42-7.35 (m, 4H), 7.12 (d, J = 7.8 Hz, 2H), 7.06-7.04 (m, 1H), 5.18 (s, 1H), 4.97 (s, 1H), 2.09 (s, 3H); ^{13}C NMR (150 MHz, DMSO-d_6) δ 166.8, 159.6, 143.9, 142.0, 132.4, 130.0, 129.4, 128.1, 127.9, 127.0, 122.3, 115.4, 112.9, 23.8; FTIR (cm^{-1}): 3148, 2968, 1661, 1591, 1489, 1201, 901, 751; mp = 90-92 °C (acetone/hexanes); HRMS (ESI) m/z, calculated for [$C_{16}H_{16}NO_2^+$]: 254.1176; found: 254.1180.

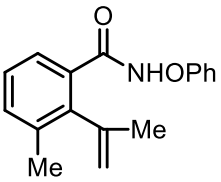


(**S1**) According to the general protocol: 5-fluoro-2-(prop-1-en-2-yl)benzoic acid (1.80 g, 10 mmol) and DIEA (5.5 mL, 33 mmol) were dissolved in DMF (30 mL) and stirred at 0 °C for 10 minutes. HATU (4.18 g, 11 mmol) was added, stirred at 0 °C for 30 minutes, and then *O*-phenylhydroxylamine hydrochloride (1.60 g, 11 mmol) was added. The reaction stirred for 4 hours, slowly warming to room temperature for overnight, and was worked up according to the general procedure. Purification by chromatography (linear gradient, 5:95 EtOAc:hexanes to 40:60 EtOAc:hexanes) afforded **S1** (2.50 g, 92%) as a white solid. ^{1}H NMR (600 MHz, DMSO-d_6) δ 12.21 (s, 1H), 7.45-7.41 (m, 2H), 7.38-7.33 (m, 3H), 7.14 (d, J = 8.4 Hz, 2H), 7.07-7.05 (m, 1H), 5.19 (s, 1H), 4.97 (s, 1H), 2.08 (s, 3H); ^{19}F NMR (565 MHz, DMSO-d_6) δ -114.9 (m); ^{13}C NMR (150 MHz, DMSO-d_6) δ 165.4, 160.5 (d, J = 243.9 Hz), 159.5, 143.0, 138.4, 134.2 (d, J = 6.6 Hz), 130.3 (d, J = 7.8 Hz), 129.4, 122.4, 116.9 (d, J = 20.7 Hz), 115.8, 114.8 (d, J = 30.0 Hz), 113.0, 23.9;

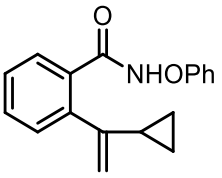
(FTIR cm^{-1}): 3144, 2970, 1662, 1591, 1490, 1215, 1161, 751, 688; mp = 93-95 °C (EtOAc:hexanes); HRMS (ESI) m/z, calculated for $[C_{16}H_{15}FNO_2^+]$: 272.1081; found: 272.1074.



(**S2**) According to the general protocol: 4-chloro-2-(prop-1-en-2-yl)benzoic acid (1.47 g, 7.5 mmol) and DIEA (4.1 mL, 24.8 mmol) were dissolved in DMF (20 mL) and stirred at 0 °C for 10 minutes. HATU (3.14 g, 8.25 mmol) was added, stirred at 0 °C for 30 minutes, and then *O*-phenylhydroxylamine hydrochloride (1.21 g, 8.25 mmol) was added. The reaction stirred for 4 hours, slowly warming to room temperature for overnight, and was worked up according to the general procedure. Purification by chromatography (linear gradient, 4:96 EtOAc:hexanes to 30:70 EtOAc:hexanes) afforded **S2** (1.34 g, 63%) as a white solid. ^{1}H NMR (600 MHz, DMSO-d_6) δ 12.18 (s, 1H), 7.58 (d, J = 7.8 Hz, 1H), 7.49-7.45 (m, 2H), 7.37-7.35 (m, 2H), 7.11 (d, J = 7.8 Hz, 2H), 7.07-7.04 (m, 1H), 5.22 (s, 1H), 5.01 (s, 1H), 2.09 (s, 3H); ^{13}C NMR (150 MHz, DMSO-d_6) δ 165.8, 159.5, 144.1, 142.8, 134.7, 131.2, 129.9, 129.4, 127.9, 127.0, 122.4, 116.3, 112.9, 23.5; FTIR (cm^{-1}): 3141, 2949, 1661, 1590, 1489, 1200, 901, 751; mp = 130-133 °C (EtOAc:hexanes); HRMS (ESI) m/z, calculated for $[C_{16}H_{15}NClO_2^+]$: 288.0786; found: 288.0791.

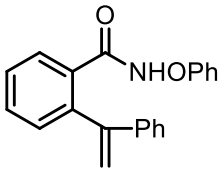


(**S3**) According to the general protocol: 3-methyl-2-(prop-1-en-2-yl)benzoic acid (1.76 g, 10 mmol) and DIEA (5.5 mL, 33 mmol) were dissolved in DMF (30 mL) and stirred at 0 °C for 10 minutes. HATU (4.18 g, 11 mmol) was added, stirred at 0 °C for 30 minutes, and then *O*-phenylhydroxylamine hydrochloride (1.60 g, 11 mmol) was added. The reaction stirred for 4 hours, slowly warming to room temperature for overnight, and was worked up according to the general procedure. Purification by chromatography (linear gradient, 3:97 EtOAc:hexanes to 30:70 EtOAc:hexanes) afforded **S3** (2.23 g, 83%) as a white solid. ^{1}H NMR (600 MHz, DMSO-d_6) δ 12.02 (s, 1H), 7.37-7.34 (m, 4H), 7.30-7.28 (m, 1H), 7.10 (d, J = 6.6 Hz, 2H), 7.06-7.03 (m, 1H), 5.26 (s, 1H), 4.82 (s, 1H), 2.28 (s, 3H), 2.03 (s, 3H); ^{13}C NMR (150 MHz, DMSO-d_6) δ 166.7, 159.6, 144.0, 141.4, 135.2, 133.2, 131.5, 129.4, 126.6, 124.8, 122.2, 115.3, 112.8, 24.2, 18.9; (FTIR cm^{-1}): 3152, 2952, 1662, 1591, 1489, 1208, 751, 688; mp = 115-117 °C (EtOAc:hexanes); HRMS (ESI) m/z, calculated for $[C_{17}H_{18}NO_2^+]$: 268.1332; found: 268.1337.

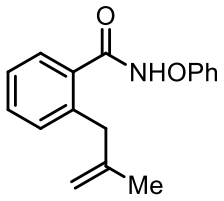


(**S4**) According to the general protocol: 2-(1-cyclopropylvinyl)benzoic acid (1.19 g, 6 mmol) and DIEA (3.3 mL, 19.8 mmol) were dissolved in DMF (20 mL) and stirred at 0 °C for 10 minutes. HATU (2.51 g, 6.6 mmol) was added, stirred at 0 °C for 30 minutes, and then *O*-phenylhydroxylamine hydrochloride (0.96 g, 6.6 mmol) was added. The reaction stirred for 4 hours, slowly warming to room temperature for overnight, and was worked up according to the general procedure. Purification by chromatography (linear gradient, 5:95 EtOAc:hexanes to 35:65 EtOAc:hexanes) afforded **S4** (1.55 g, 93%) as a white solid. ^{1}H NMR (600 MHz, DMSO-d_6) δ 12.05 (s, 1H), 7.54-7.48 (m, 2H), 7.42-7.34 (m, 4H), 7.12 (d, J = 7.8 Hz, 2H), 7.06-7.03 (m, 1H), 5.06 (s, 1H),

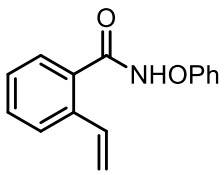
4.93 (s, 1H), 1.69-1.65 (m, 1H), 0.74-0.70 (m, 2H), 0.58-0.55 (m, 2H); ^{13}C NMR (150 MHz, DMSO-d_6) δ 166.6, 159.6, 149.0, 141.1, 132.9, 129.8, 129.4, 128.7, 127.8, 127.0, 122.3, 113.0, 111.9, 17.1, 6.7; FTIR (cm^{-1}): 3151, 3007, 1662, 1592, 1489, 1202, 902, 751, 688; mp = 85-87 °C (EtOAc:hexanes); HRMS (ESI) m/z, calculated for [$C_{18}H_{18}NO_2^+$]: 280.1332; found: 280.1337.



(**S5**) According to the general protocol: 2-(1-phenylvinyl)benzoic acid (2.24 g, 10 mmol) and DIEA (5.5 mL, 33 mmol) were dissolved in DMF (30 mL) and stirred at 0 °C for 10 minutes. HATU (4.18 g, 11 mmol) was added, stirred at 0 °C for 30 minutes, and then *O*-phenylhydroxylamine hydrochloride (1.60 g, 11 mmol) was added. The reaction stirred for 4 hours, slowly warming to room temperature for overnight, and was worked up according to the general procedure. Purification by chromatography (linear gradient, 6:94 EtOAc:hexanes to 50:50 EtOAc:hexanes) afforded **S5** (2.60 g, 83%) as a white solid. ^{1}H NMR (600 MHz, DMSO-d_6) δ 12.02 (d, J = 7.2 Hz, 1H), 7.62-7.20 (m, 11H), 6.96-6.74 (m, 3H), 5.77 (d, J = 14.4 Hz, 1H), 5.26 (d, J = 12.0 Hz, 1H); ^{13}C NMR (150 MHz, DMSO-d_6) δ 166.1, 159.4, 147.4, 140.6, 139.9, 133.4, 130.5, 130.3, 129.3, 128.2, 128.0, 127.8, 127.6, 126.8, 122.1, 115.6, 112.7; FTIR (cm^{-1}): 3163, 2945, 1663, 1591, 1489, 1201, 902, 771; mp = 115-118 °C (EtOAc:hexanes); HRMS (ESI) m/z, calculated for [$C_{21}H_{18}NO_2^+$]: 316.1332; found: 316.1338.

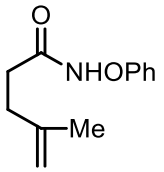


(**S6**) According to the general protocol: 2-(2-methylallyl)benzoic acid (1.76 g, 10 mmol) and DIEA (5.5 mL, 33 mmol) were dissolved in DMF (30 mL) and stirred at 0 °C for 10 minutes. HATU (4.18 g, 11 mmol) was added, stirred at 0 °C for 30 minutes, and then *O*-phenylhydroxylamine hydrochloride (1.60 g, 11 mmol) was added. The reaction stirred for 4 hours, slowly warming to room temperature for overnight, and was worked up according to the general procedure. Purification by chromatography (linear gradient, 5:95 EtOAc:hexanes to 35:65 EtOAc:hexanes) afforded **S6** (1.91 g, 72%) as a white solid. ^{1}H NMR (600 MHz, DMSO-d_6) δ 12.21 (brs, 1H), 7.56-7.46 (m, 2H), 7.36-7.31 (m, 4H), 7.12-7.03 (m, 3H), 4.83 (s, 1H), 4.55 (s, 1H), 1.67 (s, 3H); ^{13}C NMR (150 MHz, DMSO-d_6) δ 159.6, 144.5, 138.0, 133.2, 130.5, 130.3, 129.4, 127.9, 126.2, 122.3, 112.9, 112.1, 40.0, 22.4; FTIR (cm^{-1}): 3151, 2968, 1659, 1591, 1489, 1201, 898, 749; mp = 93-96 °C (EtOAc:hexanes); HRMS (ESI) m/z, calculated for [$C_{17}H_{18}NO_2^+$]: 268.1332; found: 268.1337.

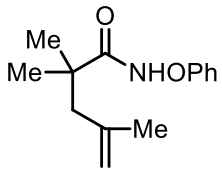


(**S7**) According to the general protocol: 2-vinylbenzoic acid (1.48 g, 10 mmol) and DIEA (5.5 mL, 33 mmol) were dissolved in DMF (30 mL) and stirred at 0 °C for 10 minutes. HATU (4.18 g, 11 mmol) was added, stirred at 0 °C for 30 minutes, and then *O*-phenylhydroxylamine hydrochloride (1.60 g, 11 mmol) was added. The reaction stirred for 4 hours, slowly warming to room temperature for overnight, and was worked up according to the general procedure. Purification by chromatography (linear gradient, 6:94 EtOAc:hexanes to 42:58 EtOAc:hexanes) afforded **S7** (2.07 g, 87%) as a white solid. ^{1}H NMR (600 MHz, DMSO-d_6) δ 12.30 (s, 1H), 7.78 (d, J = 7.8 Hz, 1H), 7.58 (d, J = 7.2 Hz, 1H), 7.55-7.53 (m, 1H), 7.43-7.37 (m, 3H), 7.14 (d, J = 7.2 Hz, 2H), 7.08-

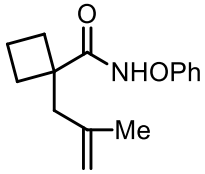
7.06 (m, 1H), 7.02-6.97 (m, 1H), 5,89 (d, *J* = 17.4 Hz), 5.40 (d, *J* = 10.8 Hz); ^{13}C NMR (150 MHz, DMSO-d_6) δ 166.1, 159.5, 135.6, 133.4, 132.2, 130.5, 129.5, 127.9, 127.7, 125.5, 122.4, 116.9, 112.9; FTIR (cm^{-1}): 3145, 2970, 1674, 1595, 1202, 904, 770, 689; mp = 111-114 °C (EtOAc:hexanes); HRMS (ESI) m/z, calculated for [$C_{15}H_{14}NO_2^+$]: 240.1019; found: 240.1023.



(**S8**) According to the general protocol: 4-methylpent-4-enoic acid (2.28 g, 20 mmol) and DIEA (11.0 mL, 66 mmol) were dissolved in DMF (60 mL) and stirred at 0 °C for 10 minutes. HATU (8.37 g, 22 mmol) was added, stirred at 0 °C for 30 minutes, and then *O*-phenylhydroxylamine hydrochloride (3.2 g, 22 mmol) was added. The reaction stirred for 4 hours, slowly warming to room temperature for overnight, and was worked up according to the general procedure. Purification by chromatography (linear gradient, 6:94 EtOAc:hexanes to 60:40 EtOAc:hexanes) and recrystallization in EtOAc afforded **S8** (1.76 g, 43%) as a white solid. ^{1}H NMR (600 MHz, DMSO-d_6) δ 11.71 (s, 1H), 7.32-7.30 (m, 2H), 7.02-6.99 (m, 3H), 4.76 (s, 1H), 4.73 (s, 1H), 2.32-2.30 (m, 4H), 1.73 (s, 3H); ^{13}C NMR (150 MHz, DMSO-d_6) δ 170.0, 160.0, 144.7, 129.8, 122.7, 113.3, 111.0, 33.0, 31.0, 22.7; FTIR (cm^{-1}): 3112, 2904, 1659, 1527, 880, 744, 688; mp = 133-135 °C (EtOAc:hexanes); HRMS (ESI) m/z, calculated for [$C_{12}H_{16}NO_2^+$]: 206.1176; found: 206.1181.

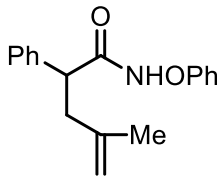


(**S9**) According to the general protocol: 2,2,4-trimethylpent-4-enoic acid (1.42 g, 10 mmol) and DIEA (5.5 mL, 33 mmol) were dissolved in DMF (30 mL) and stirred at 0 °C for 10 minutes. HATU (4.18 g, 11 mmol) was added, stirred at 0 °C for 30 minutes, and then *O*-phenylhydroxylamine hydrochloride (1.60 g, 11 mmol) was added. The reaction stirred for 4 hours, slowly warming to room temperature for overnight, and was worked up according to the general procedure. Purification by chromatography (linear gradient, 5:95 EtOAc:hexanes to 35:65 EtOAc:hexanes) afforded **S9** (2.12 g, 91%) as a white solid. ^{1}H NMR (600 MHz, DMSO-d_6) δ 11.59 (s, 1H), 7.33-7.30 (m, 2H), 7.02-6.98 (m, 3H), 4.83 (s, 1H), 4.70 (s, 1H), 2.30 (s, 2H), 1.67 (s, 3H), 1.18 (s, 6H); ^{13}C NMR (150 MHz, DMSO-d_6) δ 174.6, 159.7, 142.2, 129.3, 122.0, 114.2, 112.8, 47.4, 40.8, 25.3, 23.7; FTIR (cm^{-1}): 3155, 2970, 1656, 1589, 1499, 1205, 755; mp = 84-86 °C (EtOAc:hexanes); HRMS (ESI) m/z, calculated for [$C_{14}H_{20}NO_2^+$]: 234.1489; found: 234.1494.

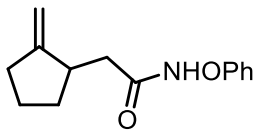


(**S10**) According to the general protocol: 4-methyl-2-phenylpent-4-enoic acid (1.52 g, 10 mmol) and DIEA (5.5 mL, 33 mmol) were dissolved in DMF (30 mL) and stirred at 0 °C for 10 minutes. HATU (4.18 g, 11 mmol) was added, stirred at 0 °C for 30 minutes, and then *O*-phenylhydroxylamine hydrochloride (1.60 g, 11 mmol) was added. The reaction stirred for 4 hours, slowly warming to room temperature for overnight, and was worked up according to the general procedure. Purification by chromatography (linear gradient, 5:95 EtOAc:hexanes to 40:60 EtOAc:hexanes) afforded **S10** (2.11 g, 90%) as a white solid. ^{1}H NMR (600 MHz, DMSO-d_6) δ 11.62 (s, 1H), 7.32-7.30 (m, 2H), 7.01-7.00 (m, 3H), 4.77 (s, 1H), 4.63 (s, 1H), 2.57 (s, 2H), 2.44-2.42 (m, 2H), 1.96-1.82 (m, 4H), 1.67 (s, 3H); ^{13}C NMR (150 MHz, DMSO-d_6) δ 173.6, 159.8, 142.3, 129.3, 122.0, 112.8, 112.3, 46.0, 45.1, 30.3, 23.2, 15.7; FTIR (cm^{-1}): 3192, 2969, 1657, 1592, 1488, 1197, 750, 688; mp = 73-75 °C (EtOAc:hexanes); HRMS (ESI) m/z,

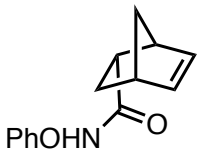
calculated for [$C_{15}H_{20}NO_2^+$]: 246.1489; found: 246.1493.



(**S11**) According to the general protocol: 4-methyl-2-phenylpent-4-enoic acid (1.90 g, 10 mmol) and DIEA (5.5 mL, 33 mmol) were dissolved in DMF (30 mL) and stirred at 0 °C for 10 minutes. HATU (4.18 g, 11 mmol) was added, stirred at 0 °C for 30 minutes, and then *O*-phenylhydroxylamine hydrochloride (1.60 g, 11 mmol) was added. The reaction stirred for 4 hours, slowly warming to room temperature for overnight, and was worked up according to the general procedure. Purification by chromatography (linear gradient, 3:97 EtOAc:hexanes to 30:70 EtOAc:hexanes) and recrystallization in EtOAc afforded **S11** (1.85 g, 66%) as a white solid. ^{1}H NMR (600 MHz, DMSO-d_6) δ 12.03 (s, 1H), 7.40-7.34 (m, 4H), 7.29-723 (m, 3H), 6.99-6.97 (m, 1H), 6.85 (d, *J* = 7.8 Hz, 2H), 4.79 (s, 1H), 4.74 (s, 1H), 3.75-3.72 (m, 1H), 2.79 (dd, *J* = 13.8, 9.6 Hz, 1H), 2.39 (dd, *J* = 14.4, 5.4 Hz, 1H), 1.74 (s, 3H); ^{13}C NMR (150 MHz, DMSO-d_6) δ 169.6, 159.4, 142.5, 139.4, 129.3, 128.3, 127.6, 127.0, 122.3, 112.6, 112.3, 46.6, 40.1, 22.3; FTIR (cm^{-1}): 3158, 2969, 1665, 1591, 1488, 1161, 752, 698; mp = 124-127 °C (EtOAc:hexanes); HRMS (ESI) m/z, calculated for [$C_{18}H_{20}NO_2^+$]: 282.1489; found: 282.1493.



(**S12**) According to the general protocol: 2-(2-methylenecyclopentyl)acetic acid (1.20 g, 8.55 mmol) and DIEA (4.7 mL, 28.2 mmol) were dissolved in DMF (25 mL) and stirred at 0 °C for 10 minutes. HATU (3.58 g, 9.4 mmol) was added, stirred at 0 °C for 30 minutes, and then *O*-phenylhydroxylamine hydrochloride (1.37 g, 9.4 mmol) was added. The reaction stirred for 4 hours, slowly warming to room temperature for overnight, and was worked up according to the general procedure. Purification by chromatography (linear gradient, 6:94 EtOAc:hexanes to 50:50 EtOAc:hexanes) afforded **S12** (1.67 g, 86%) as a white solid. ^{1}H NMR (600 MHz, DMSO-d_6) δ 11.71 (s, 1H), 7.34-7.31 (m, 2H), 7.03-6.99 (m, 3H), 4.90 (s, 1H), 4.86 (s, 1H), 2.78-2.67 (m, 1H), 2.46 (dd, *J* = 14.4, 5.4 Hz, 1H), 2.32-2.78 (m, 2H), 2.09 (dd, *J* = 14.4, 9.0 Hz, 1H), 1.92-1.87 (m, 1H), 1.70-1.68 (m, 1H), 1.56-1.50 (m, 1H), 1.38-1.32 (m, 1H); ^{13}C NMR (150 MHz, DMSO-d_6) δ 169.1, 159.5, 154.9, 129.4, 122.2, 112.8, 105.1, 40.0, 37.0, 32.5, 32.3, 23.5; FTIR (cm^{-1}): 3146, 2954, 1659, 1592, 1488, 1207, 1158, 748, 688; mp = 98-100 °C (EtOAc:hexanes); HRMS (ESI) m/z, calculated for [$C_{14}H_{18}NO_2^+$]: 232.1332; found: 232.1337.

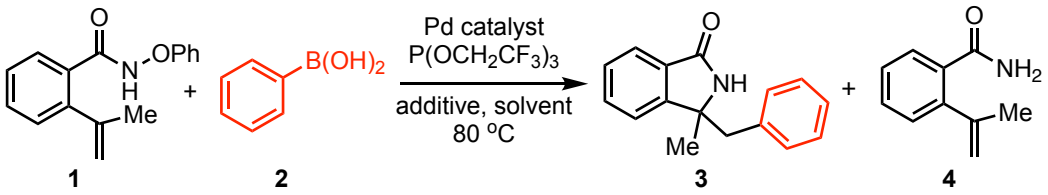


(**35**) According to the general protocol: 4-methyl-2-phenylpent-4-enoic acid (1.52 g, 10 mmol) and DIEA (5.5 mL, 33 mmol) were dissolved in DMF (30 mL) and stirred at 0 °C for 10 minutes. HATU (4.18 g, 11 mmol) was added, stirred at 0 °C for 30 minutes, and then *O*-phenylhydroxylamine hydrochloride (1.60 g, 11 mmol) was added. The reaction stirred for 4 hours, slowly warming to room temperature for overnight, and was worked up according to the general procedure. Purification by chromatography (linear gradient, 6:94 EtOAc:hexanes to 70:30 EtOAc:hexanes) and recrystallization in EtOAc afforded afforded **35** (1.15 g, 50%) as a white solid. ^{1}H NMR (600 MHz, DMSO-d_6) δ 11.59 (s, 1H), 7.33-7.30 (m, 2H), 7.01-6.95 (m, 3H), 6.18 (s, 1H), 5.93 (s, 1H), 3.24 (s, 1H),

2.93-2.87 (m, 2H), 1.87-1.82 (m, 1H), 1.36-1.31 (m, 3H); ^{13}C NMR (150 MHz, DMSO-d_6) δ 171.3, 159.7, 137.3, 132.0, 129.3, 122.0, 112.7, 49.4, 45.6, 41.9, 40.8, 28.2; mp = 170-172 °C (EtOAc:hexanes); FTIR (cm^{-1}): 3164, 2970, 1668, 1513, 1189, 750, 689; HRMS (ESI) m/z, calculated for [$C_{14}H_{16}NO_2^+$]: 230.1176; found: 230.1179.

4. Synthesis of Lactams *via* Aza-Heck-Suzuki Reaction

4.1. Reaction Optimization [a]



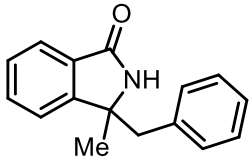
Entry	Pd Catalyst [mol %]	Solvent	Additive	Yield [%] [b]
1	(COD)Pd(CH_2SiMe_3)$_2$ [10]	PhMe	--	18
2	(COD)Pd(CH_2SiMe_3)$_2$ [10]	Dioxane	--	15
3	(COD)Pd(CH_2SiMe_3)$_2$ [10]	*t*-BuOH	--	55
4	(COD)Pd(CH_2SiMe_3)$_2$ [10]	MeCN	--	74
5	(COD)Pd(CH_2SiMe_3)$_2$ [10]	MeCN	4Å MS	81
6	(COD)Pd(CH_2SiMe_3)$_2$ [10]	MeCN	5Å MS	59
7	(COD)Pd(CH_2SiMe_3)$_2$ [10]	MeCN	$MgSO_4$	35
8	Pd(acac)$_2$ [10]	MeCN	4Å MS	72
9	Pd(MeCN)Cl_2 [10]	MeCN	4Å MS	54
10	Pd(OAc)$_2$ [10]	MeCN	4Å MS	81
11[c]	Pd(OAc)$_2$ [5]	MeCN	4Å MS	81

[a] Unless otherwise noted, reactions run with 0.2 mmol **1** and 0.6 mmol **2** at 0.05 M. [b] Yield calculated by ^{1}H NMR with 1,3,5-trimethoxybenzene as internal standard. [c] 5 mol% Pd(OAc)$_2$ at 0.1 M.

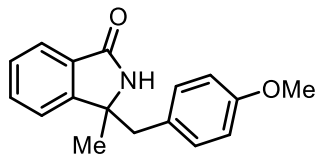
4.2. Synthesis of Lactams *via* Aza-Heck-Suzuki Reaction

General Protocol: A flame-dried Schlenk flask equipped with a magnetic stir bar and rubber septum was attached to a double manifold and cooled under vacuum. The flask was backfilled with N_2, the septum was removed and Pd(OAc)$_2$ or (COD)Pd(CH_2SiMe_3)$_2$ (0.05-0.20 equiv), hydroxamate (1.0 mmol, 1.0 equiv), boronic acid (3.0 mmol, 3.0 equiv) and 4Å MS (500 mg) were added to the flask. The septum was replaced, and the flask was evacuated and backfilled with nitrogen four times. P(OCH_2CF_3)$_3$ (0.15-0.6 equiv), and anhydrous MeCN (10.0 mL) were added sequentially to the flask via syringe. The resulting solution was heated in an oil bath with rapid stirring at the indicated temperature for 24 h. Upon completion, the reaction was cooled to room temperature, opened to air, and the reaction mixture was then diluted with EtOAc, filtered through celite. The

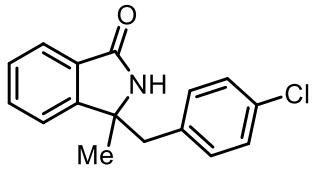
suspension was adsorbed onto Celite via rotary evaporation. The resulting powder was directly chromatographed on silica gel (5-20 μm particle size) to yield the desired product.



(**3**) According to the general protocol: $Pd(OAc)_2$ (11.3 mg, 0.05 mmol, 0.05 equiv), N-phenoxy-2-(prop-1-en-2-yl)benzamide **1** (253.3 mg, 1.00 mmol), phenylboronic acid (366 mg, 3.0 mmol, 3.0 equiv), 4Å MS (500 mg), $P(OCH_2CF_3)_3$ (49.2 mg, 0.15 mmol, 0.15 equiv), and anhydrous MeCN (10.0 mL) were combined under N_2. The reaction was stirred at 80 °C for 24 h under N_2 environment. The reaction was worked up according to the general procedure and purified by chromatography (linear gradient, 12:88 EtOAc:hexanes to 100:0 EtOAc:hexanes) to afford amide **3** (209.8 mg, 88%) as a white solid. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.76 (d, *J* = 7.8 Hz, 1H), 7.59-7.57 (m, 1H), 7.45-7.41 (m, 2H), 7.22-7.21 (m, 3H), 7.08-7.07 (m, 2H), 6.72 (brs, 1H), 3.12 (d, *J* = 13.8 Hz, 1H), 2.97 (d, *J* = 13.8 Hz, 1H), 1.53 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 169.4, 151.7, 135.8, 131.9, 131.1, 130.3, 128.2, 128.1, 127.0, 123.9, 121.4, 61.8, 46.7, 25.4; FTIR (cm^{-1}): 3212, 2917, 1694, 1469, 1455, 1352, 700; mp = 148-150 °C (EtOAc:hexanes); HRMS (ESI) m/z, calculated for [$C_{16}H_{16}NO^+$]: 238.1226; found: 238.1220.

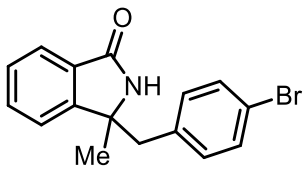


(**5**) According to the general protocol: $Pd(OAc)_2$ (11.3 mg, 0.05 mmol, 0.05 equiv), N-phenoxy-2-(prop-1-en-2-yl)benzamide **1** (253.3 mg, 1.00 mmol), 4-methoxyphenylboronic acid (366 mg, 3.0 mmol, 3.0 equiv), 4Å MS (500 mg), $P(OCH_2CF_3)_3$ (49.2 mg, 0.15 mmol, 0.15 equiv), and anhydrous MeCN (10.0 mL) were combined under N_2. The reaction was stirred at 80 °C for 24 h under N_2 environment. The reaction was worked up according to the general procedure and purified by chromatography (linear gradient, 12:88 EtOAc:hexanes to 100:0 EtOAc:hexanes) to afford amide **5** (212.8 mg, 80%) as a white solid. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.98 (s, 1H), 7.63 (d, *J* = 7.2 Hz, 1H), 7.46-7.43 (m, 1H), 7.31-7.27 (m, 2H), 6.80 (d, *J* = 8.4 Hz, 2H), 6.54 (d, *J* = 8.4 Hz, 2H), 3.57 (s, 3H), 2.97 (d, *J* = 13.8 Hz, 1H), 2.90 (d, *J* = 13.8 Hz, 1H), 1.46 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 170.2, 158.4, 151.6, 131.7, 131.6, 131.3, 128.0, 127.9, 123.7, 121.6, 113.4, 62.5, 55.1, 45.7, 25.7; FTIR (cm^{-1}): 3230, 1693, 1612, 1513, 1249, 1179, 756; mp = 143-145 °C (EtOAc:hexanes); HRMS (ESI) m/z, calculated for [$C_{17}H_{18}NO_2^+$]: 268.1332; found: 268.1336.

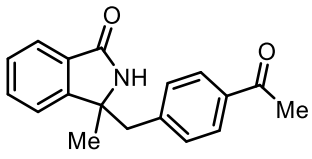


(**6**) According to the general protocol: $Pd(OAc)_2$ (11.3 mg, 0.05 mmol, 0.05 equiv), N-phenoxy-2-(prop-1-en-2-yl)benzamide **1** (253.3 mg, 1.00 mmol), 4-chlorophenylboronic acid (366 mg, 3.0 mmol, 3.0 equiv), 4Å MS (500 mg), $P(OCH_2CF_3)_3$ (49.2 mg, 0.15 mmol, 0.15 equiv), and anhydrous MeCN (10.0 mL) were combined under N_2. The reaction was stirred at 80 °C for 24 h under N_2 environment. The reaction was worked up according to the general procedure and purified by chromatography (linear gradient, 12:88 EtOAc:hexanes to 100:0 EtOAc:hexanes) to afford amide **6** (233 mg, 86%) as a white solid. ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.18 (s, 1H), 7.71 (d, *J* = 7.5 Hz, 1H), 7.58-7.56 (t, *J* = 7.8 Hz, 1H), 7.43-7.40 (m, 2H), 7.04 (d, *J* = 8.4 Hz, 2H), 6.88 (d, *J* = 8.4 Hz, 2H), 3.12 (d, *J* = 13.8 Hz, 1H), 3.04 (d, *J* = 13.8 Hz, 1H), 1.58 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 170.2, 151.0, 134.2, 132.7, 131.8, 131.6, 128.2,

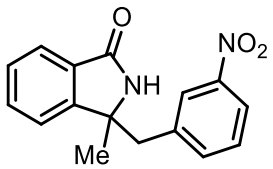
128.1, 123.8, 121.5, 62.3, 45.8, 25.9; FTIR (cm^{-1}): 3215, 1693, 1492, 1352, 1319, 1147, 1016, 762; mp = 155-158 °C (EtOAc:hexanes); HRMS (ESI) m/z, calculated for [$C_{16}H_{15}ClNO^+$]: 272.0837; found: 272.0840.



(**7**) According to the general protocol: $Pd(OAc)_2$ (11.3 mg, 0.05 mmol, 0.05 equiv), N-phenoxy-2-(prop-1-en-2-yl)benzamide **1** (253.3 mg, 1.00 mmol), 4-bromophenylboronic acid (600 mg, 3.0 mmol, 3.0 equiv), 4Å MS (500 mg), $P(OCH_2CF_3)_3$ (49.2 mg, 0.15 mmol, 0.15 equiv), and anhydrous MeCN (10.0 mL) were combined under N_2. The reaction was stirred at 80 °C for 24 h under N_2 environment. The reaction was worked up according to the general procedure and purified by chromatography (linear gradient, 12:88 EtOAc:hexanes to 100:0 EtOAc:hexanes) to afford amide **7** (230 mg, 73%) as a white solid. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.82 (s, 1H), 7.72 (d, *J* = 7.2 Hz, 1H), 7.59-7.57 (m, 1H), 7.44-7.40 (m, 2H), 7.23 (d, *J* = 8.2 Hz, 2H), 6.84 (d, *J* = 8.2 Hz, 2H), 3.10 (d, *J* = 13.2 Hz, 1H), 3.01 (d, *J* = 13.2 Hz, 1H), 1.57 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 170.0, 151.1, 134.7, 131.94, 131.90, 131.4, 131.1, 128.3, 123.9, 121.5, 120.9, 62.1, 45.9, 25.9; FTIR (cm^{-1}): 3217, 2972, 1694, 1592, 1405, 1351, 762, 716; mp = 171-173 °C (EtOAc:hexanes); HRMS (ESI) m/z, calculated for [$C_{16}H_{15}BrNO^+$]: 316.0332; found: 316.0338.

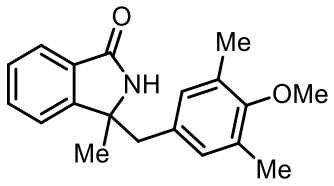


(**8**) According to the general protocol: $Pd(OAc)_2$ (11.3 mg, 0.05 mmol, 0.05 equiv), N-phenoxy-2-(prop-1-en-2-yl)benzamide **1** (253.3 mg, 1.00 mmol), 4-acetylphenylboronic acid (492 mg, 3.0 mmol, 3.0 equiv), 4Å MS (500 mg), $P(OCH_2CF_3)_3$ (49.2 mg, 0.15 mmol, 0.15 equiv), and anhydrous MeCN (10.0 mL) were combined under N_2. The reaction was stirred at 80 °C for 24 h under N_2 environment. The reaction was worked up according to the general procedure and purified by chromatography (linear gradient, 12:88 EtOAc:hexanes to 100:0 EtOAc:hexanes) to afford amide **8** (254.5 mg, 91%) as a white solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.73 (s, 1H), 7.64 (d, *J* = 7.6 Hz, 1H), 7.59 (d, *J* = 8.0 Hz, 2H), 7.56-7.52 (m, 1H), 7.43 (d, *J* = 7.6 Hz, 1H), 7.39-7.35 (m, 1H), 6.98 (d, *J* = 8.0 Hz, 2H), 3.21 (d, *J* = 13.6 Hz, 1H), 3.13 (d, *J* = 13.2 Hz, 1H), 2.40 (s, 3H), 1.58 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 197.9, 170.3, 150.8, 141.4, 135.4, 131.9, 131.6, 130.5, 128.2, 127.8, 123.6, 121.6, 62.4, 46.1, 26.5, 26.2; FTIR (cm^{-1}): 3222, 2973, 1696, 1607, 1357, 1269, 766, 699; mp = 173-175 °C (EtOAc:hexanes); HRMS (ESI) m/z, calculated for [$C_{18}H_{18}NO_2^+$]: 280.1332; found: 280.1336.

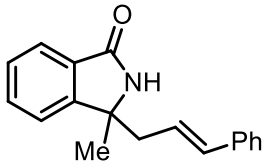


(**9**) According to the general protocol: $Pd(OAc)_2$ (11.3 mg, 0.05 mmol, 0.05 equiv), N-phenoxy-2-(prop-1-en-2-yl)benzamide **1** (253.3 mg, 1.00 mmol), 3-nitrophenylboronic acid (501 mg, 3.0 mmol, 3.0 equiv), 4Å MS (500 mg), $P(OCH_2CF_3)_3$ (49.2 mg, 0.15 mmol, 0.15 equiv), and anhydrous MeCN (10.0 mL) were combined under N_2. The reaction was stirred at 80 °C for 24 h under N_2 environment. The reaction was worked up according to the general procedure and purified by chromatography (linear gradient, 12:88 EtOAc:hexanes to 100:0 EtOAc:hexanes) to afford amide **9** (195.2 mg, 69%) as a white solid. ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.71 (s,

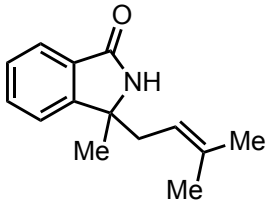
1H), 7.87 (d, *J* = 7.8 Hz, 1H), 7.76 (s, 1H), 7.57-7.52 (m, 2H), 7.42 (d, *J* = 7.2 Hz, 1H), 7.35-7.33 (m, 1H), 7.20 (d, *J* = 7.2 Hz, 1H), 7.16-7.13 (m, 1H), 3.23 (d, *J* = 13.8 Hz, 1H), 3.14 (d, *J* = 13.8 Hz, 1H), 1.58 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 170.5, 150.4, 147.7, 137.6, 136.4, 132.1, 131.5, 128.6, 128.5, 125.2, 123.8, 121.8, 121.4, 62.4, 45.8, 26.0; FTIR (cm^{-1}): 3209, 2974, 1694, 1526, 1350, 764, 700; mp = 165-167 °C (EtOAc:hexanes); HRMS (ESI) m/z, calculated for [$C_{16}H_{15}N_2O_3^+$]: 283.1077; found: 283.1080.



(**10**) According to the general protocol: $Pd(OAc)_2$ (11.3 mg, 0.05 mmol, 0.05 equiv), N-phenoxy-2-(prop-1-en-2-yl)benzamide **1** (253.3 mg, 1.00 mmol), (4-methoxy-3,5-dimethylphenyl)boronic acid (540 mg, 3.0 mmol, 3.0 equiv), 4Å MS (500 mg), $P(OCH_2CF_3)_3$ (49.2 mg, 0.15 mmol, 0.15 equiv), and anhydrous MeCN (10.0 mL) were combined under N_2. The reaction was stirred at 80 °C for 24 h under N_2 environment. The reaction was worked up according to the general procedure and purified by chromatography (linear gradient, 12:88 EtOAc:hexanes to 100:0 EtOAc:hexanes) to afford amide **10** (173.5 mg, 59%) as a white solid. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.73 (d, *J* = 7.8 Hz, 1H), 7.53-7.51 (m, 1H), 7.40-7.38 (m, 1H), 7.35 (d, *J* = 7.2 Hz, 1H), 6.71 (s, 2H), 6.15 (brs, 1H), 3.63 (s, 3H), 2.93 (d, *J* = 13.2 Hz, 1H), 2.69 (d, *J* = 13.2 Hz, 1H), 2.17 (s, 6H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 169.5, 156.0, 152.2, 131.8, 131.3, 131.1, 130.7, 130.6, 128.1, 123.9, 121.4, 61.9, 59.7, 46.1, 25.2, 16.0; FTIR (cm^{-1}): 3204, 2925, 1695, 1469, 1225, 1144, 736; mp = 163-165 °C (EtOAc:hexanes); HRMS (ESI) m/z, calculated for [$C_{19}H_{22}NO_2^+$]: 296.1645; found: 296.1650.

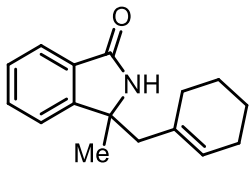


(**11**) According to the general protocol: $Pd(OAc)_2$ (11.3 mg, 0.05 mmol, 0.05 equiv), N-phenoxy-2-(prop-1-en-2-yl)benzamide **1** (253.3 mg, 1.00 mmol), (*E*)-styrylboronic acid (444 mg, 3.0 mmol, 3.0 equiv), 4Å MS (500 mg), $P(OCH_2CF_3)_3$ (49.2 mg, 0.15 mmol, 0.15 equiv), and anhydrous MeCN (10.0 mL) were combined under N_2. The reaction was stirred at 80 °C for 24 h under N_2 environment. The reaction was worked up according to the general procedure and purified by chromatography (linear gradient, 10:90 EtOAc:hexanes to 70:30 EtOAc:hexanes) to afford amide **11** (250.0 mg, 95%) as a colorless oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.75 (d, *J* = 7.2 Hz, 1H), 7.65-7.61 (m, 1H), 7.50-7.48 (m, 1H), 7.39-7.36 (m, 1H), 7.33 (d, *J* = 7.8 Hz, 1H), 7.18-7.16 (m, 4H), 7.12-7.10 (m, 1H), 6.31 (d, *J* = 15.7 Hz, 1H), 5.98 (dt, *J* = 15.4, 7.4 Hz, 1H), 2.65 (dd, *J* = 13.8, 7.8 Hz, 1H), 2.53 (dd, *J* = 13.8, 7.2 Hz, 1H), 1.49 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 170.3, 151.8, 137.0, 134.6, 132.0, 131.4, 128.5, 128.1, 127.4, 126.3, 123.9, 123.9, 121.4, 62.0, 44.1, 25.6; FTIR (cm^{-1}): 3223, 2973, 1692, 1469, 1350, 749, 696; HRMS (ESI) m/z, calculated for [$C_{18}H_{18}NO^+$]: 264.1383; found: 264.1387.

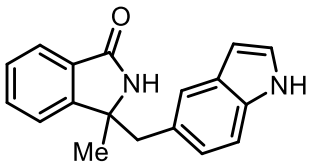


(**12**) According to the general protocol: $Pd(OAc)_2$ (11.3 mg, 0.05 mmol, 0.05 equiv), N-phenoxy-2-(prop-1-en-2-yl)benzamide **1** (253.3 mg, 1.00 mmol), (2-methylprop-1-en-1-yl)boronic acid (300 mg, 3.0 mmol, 3.0 equiv), 4Å MS (500 mg), $P(OCH_2CF_3)_3$ (49.2 mg, 0.15 mmol, 0.15 equiv), and anhydrous MeCN (10.0 mL) were combined under N_2. The reaction was stirred at 80 °C for 24 h under

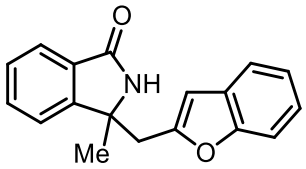
N_2 environment. The reaction was worked up according to the general procedure and purified by chromatography (linear gradient, 10:90 EtOAc:hexanes to 80:20 EtOAc:hexanes) to afford amide **12** (146.1 mg, 65%) as a white solid. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.80 (d, *J* = 7.8 Hz, 1H), 7.54-7.51 (m, 1H), 7.43-7.41(m, 1H), 7.37 (d, *J* = 7.2 Hz, 1H), 7.28 (brs, 1H), 5.03-5.01 (m, 1H), 2.52 (dd, *J* = 14.4, 7.8 Hz, 1H), 2.41 (dd, *J* = 14.4, 7.8 Hz, 1H), 1.64 (s, 3H), 1.53 (s, 3H), 1.52 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 169.8, 152.1, 136.0, 131.7, 131.2, 127.9, 123.7, 121.1, 118.0, 62.0, 38.8, 25.9, 25.4, 17.9; FTIR (cm^{-1}): 3208, 2971, 2927, 1695, 1452, 1319, 760, 699; mp = 78-80 °C (EtOAc:hexanes); HRMS (ESI) m/z, calculated for [$C_{14}H_{18}NO^+$]: 216.1383; found: 216.1387.



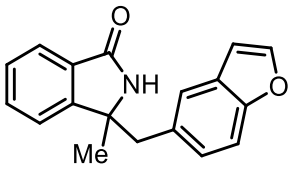
(**13**) According to the general protocol: $Pd(OAc)_2$ (11.3 mg, 0.05 mmol, 0.05 equiv), N-phenoxy-2-(prop-1-en-2-yl)benzamide **1** (253.3 mg, 1.00 mmol), cyclohex-1-en-1-ylboronic acid (378 mg, 3.0 mmol, 3.0 equiv), 4Å MS (500 mg), $P(OCH_2CF_3)_3$ (49.2 mg, 0.15 mmol, 0.15 equiv), and anhydrous MeCN (10.0 mL) were combined under N_2. The reaction was stirred at 80 °C for 24 h under N_2 environment. The reaction was worked up according to the general procedure and purified by chromatography (linear gradient, 10:90 EtOAc:hexanes to 80:20 EtOAc:hexanes) to afford amide **13** (220 mg, 91%) as a white solid. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.75-7.72 (m, 2H), 7.46-7.44 m, 1H), 7.35-7.33 (m, 1H), 7.30 (d, *J* = 7.2 Hz, 1H), 5.28 (s, 1H), 2.46 (d, *J* = 13.2 Hz, 1H), 2.32 (d, *J* = 13.8 Hz, 1H), 1.83-1.70 (m, 3H), 1.52-1,49 (m, 4H), 1.36-1.32 (m, 4H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 170.1, 152.2, 132.7, 131.6, 131.5, 127.8, 127.3, 123.6, 121.5, 62.2, 48.5, 30.5, 26.4, 25.3, 22.9, 21.9; FTIR (cm^{-1}): 3206, 2926, 2835, 1695, 1469, 1372, 761, 700; mp = 112-114 °C (EtOAc:hexanes); HRMS (ESI) m/z, calculated for [$C_{16}H_{20}NO^+$]: 242.1539; found: 242.1542.



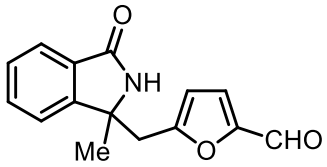
(**14**) According to the general protocol: $(COD)Pd(CH_2SiMe_3)_2$ (38.9 mg, 0.1 mmol, 0.1 equiv), N-phenoxy-2-(prop-1-en-2-yl)benzamide **1** (253.3 mg, 1.00 mmol), (1H-indol-5-yl)boronic acid acid (483 mg, 3.0 mmol, 3.0 equiv), 4Å MS (500 mg), $P(OCH_2CF_3)_3$ (98.4 mg, 0.3 mmol, 0.3 equiv), and anhydrous MeCN (10.0 mL) were combined under N_2. The reaction was stirred at 80 °C for 24 h under N_2 environment. The reaction was worked up according to the general procedure and purified by chromatography (linear gradient, 15:85 EtOAc:hexanes to 100:0 EtOAc:hexanes) to afford amide **14** (198.7 mg, 72%) as a white solid. ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.32 (s, 1H), 7.80 (d, *J* = 7.2 Hz, 1H), 7.61-7.58 (m, 1H), 7.47 -7.43 (m, 3H), 7.31 (d, *J* = 7.8 Hz, 1H), 7.22-7.21 (m, 1H), 6.97 (dd, *J* = 8.4, 1.2 Hz, 1H), 6.50-6.49 (m, 1H), 6.17 (brs, 1H), 3.21 (d, *J* =13.8 Hz, 1H), 2.96 (d, *J* = 13.2 Hz, 1H), 1.49 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 169.2, 152.6, 135.0, 131.9, 130.9, 128.1, 128.1, 127.2, 124.7, 124.5, 124.0, 122.2, 121.4, 110.9, 102.5, 62.0, 46.9, 25.0; FTIR (cm^{-1}): 3287, 1685, 1639, 1412, 1354, 1095, 755, 732; mp = > 200 °C Decompose (EtOAc:hexanes); HRMS (ESI) m/z, calculated for [$C_{18}H_{17}N_2O^+$]: 277.1335; found: 277.1339.



(**15**) According to the general protocol: (COD)Pd(CH_2SiMe_3)$_2$ (38.9 mg, 0.1 mmol, 0.1 equiv), N-phenoxy-2-(prop-1-en-2-yl)benzamide **1** (253.3 mg, 1.00 mmol), benzofuran-2-ylboronic acid (486 mg, 3.0 mmol, 3.0 equiv), 4Å MS (500 mg), P(OCH_2CF_3)$_3$ (98.4 mg, 0.3 mmol, 0.3 equiv), and anhydrous MeCN (10.0 mL) were combined under N_2. The reaction was stirred at 80 °C for 24 h under N_2 environment. The reaction was worked up according to the general procedure and purified by chromatography (linear gradient, 12:88 EtOAc:hexanes to 100:0 EtOAc:hexanes) to afford amide **15** (269.0 mg, 97%) as a white solid. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.73 (s, 1H), 7.70 (d, *J* = 7.8 Hz, 1H), 7.46-7.44 (m, 1H), 7.35-7.31 (m, 2H), 7.28 (d, *J* = 7.2 Hz, 1H), 7.24 (d, *J* = 8.4 Hz, 1H), 7.01-7.03 (m, 2H), 6.30 (s, 1H), 3.19 (d, *J* = 15.0 Hz, 1H), 3.02 (d, *J* = 14.4 Hz, 1H), 1.45 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 169.8, 154.8, 153.9, 151.3, 132.1, 131.2, 128.4, 128.3, 124.0, 123.9, 122.8, 121.4, 120.7, 111.0, 105.7, 61.3, 39.7, 25.5; FTIR (cm^{-1}): 3218, 3070, 1697, 1615, 1469, 1315, 752, 739, 697; mp = 121-123 °C (EtOAc:hexanes); HRMS (ESI) m/z, calculated for [$C_{18}H_{16}NO_2^+$]: 278.1176; found: 278.1180.

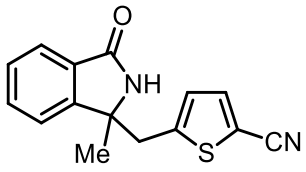


(**16**) According to the general protocol: (COD)Pd(CH_2SiMe_3)$_2$ (38.9 mg, 0.1 mmol, 0.1 equiv), N-phenoxy-2-(prop-1-en-2-yl)benzamide **1** (253.3 mg, 1.00 mmol), benzofuran-5-ylboronic acid (486 mg, 3.0 mmol, 3.0 equiv), 4Å MS (500 mg), P(OCH_2CF_3)$_3$ (98.4 mg, 0.3 mmol, 0.3 equiv), and anhydrous MeCN (10.0 mL) were combined under N_2. The reaction was stirred at 80 °C for 24 h under N_2 environment. The reaction was worked up according to the general procedure and purified by chromatography (linear gradient, 12:88 EtOAc:hexanes to 100:0 EtOAc:hexanes) to afford amide **16** (245.9 mg, 89%) as a white solid. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.66 (d, *J* = 7.8 Hz, 1H), 7.51-7.49 (m, 1H), 7.45 (d, *J* = 1.2 Hz, 1H), 7.36-7.34 (m, 3H), 7.21-7.19 (m, 2H), 6.86 (d, *J* = 8.4 Hz, 1H), 6.47 (d, *J* = 1.2 Hz, 1H), 3.13 (d, *J* = 13.2 Hz, 1H), 2.99 (d, *J* = 13.8 Hz, 1H), 1.46 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 169.8, 154.1, 151.8, 145.2, 131.9, 131.4, 130.3, 128.1, 127.4, 126.6, 123.9, 122.8, 121.5, 110.9, 106.5, 62.3, 46.5, 25.5; FTIR (cm^{-1}): 3208, 1692, 1469, 1412, 1354, 1263, 1200, 759, 737; mp = 162-164 °C (EtOAc:hexanes); HRMS (ESI) m/z, calculated for [$C_{18}H_{16}NO_2^+$]: 278.1176; found: 278.1180.

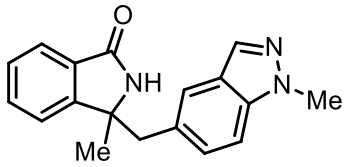


(**17**) According to the general protocol: (COD)Pd(CH_2SiMe_3)$_2$ (38.9 mg, 0.1 mmol, 0.1 equiv), N-phenoxy-2-(prop-1-en-2-yl)benzamide **1** (253.3 mg, 1.00 mmol), (5-formylfuran-2-yl)boronic acid (420 mg, 3.0 mmol, 3.0 equiv), 4Å MS (500 mg), P(OCH_2CF_3)$_3$ (98.4 mg, 0.3 mmol, 0.3 equiv), and anhydrous MeCN (10.0 mL) were combined under N_2. The reaction was stirred at 80 °C for 24 h under N_2 environment. The reaction was worked up according to the general procedure and purified by chromatography (linear gradient, 15:85 EtOAc:hexanes to 100:0 EtOAc:hexanes) to afford amide **17** (244.5 mg, 95%) as a white solid. ^{1}H NMR (600 MHz, $CDCl_3$) δ 9.35 (s, 1H), 8.15 (s, 1H), 7.68 (d, *J* = 7.2 Hz, 1H), 7.52-7.49 (m, 1H), 7.38-7.33 (m, 2H), 6.99 (d, *J* = 3.6 Hz, 1H), 6.04 (s, 1H), 3.29 (d, *J* = 15.0 Hz, 1H), 3.15 (d, *J* = 15.0 Hz, 1H), 1.56 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 177.2,

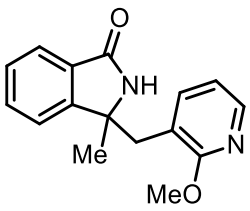
169.9, 157.8, 152.2, 150.6, 132.2, 131.3, 128.5, 123.8, 121.3, 111.5, 61.3, 39.3, 25.8; FTIR (cm^{-1}): 3240, 2795, 1700, 1516, 1469, 1389, 1024, 736, 698; mp = 116-118 °C (EtOAc:hexanes); HRMS (ESI) m/z, calculated for [$C_{15}H_{14}NO_3^+$]: 256.0968; found: 256.0971.



(**18**) According to the general protocol: (COD)Pd(CH_2SiMe_3)$_2$ (38.9 mg, 0.1 mmol, 0.1 equiv), N-phenoxy-2-(prop-1-en-2-yl)benzamide **1** (253.3 mg, 1.00 mmol), (5-cyanothiophen-2-yl)boronic acid (459 mg, 3.0 mmol, 3.0 equiv), 4Å MS (500 mg), P(OCH_2CF_3)$_3$ (98.4 mg, 0.3 mmol, 0.3 equiv), and anhydrous MeCN (10.0 mL) were combined under N_2. The reaction was stirred at 80 °C for 24 h under N_2 environment. The reaction was worked up according to the general procedure and purified by chromatography (linear gradient, 11:89 EtOAc:hexanes to 90:10 EtOAc:hexanes) to afford amide **18** (241.2 mg, 90%) as a white solid. ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.38 (s, 1H), 7.72 (d, *J* = 7.8 Hz, 1H), 7.62-7.60 (m, 1H), 7.48-7.43 (m, 2H), 7.25 (d, *J* = 3.6 Hz, 1H), 6.62 (d, *J* = 3.8 Hz, 1H), 3.48 (d, *J* = 15.0 Hz, 1H), 3.38 (d, *J* = 15.0 Hz, 1H), 1.68 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 170.4, 149.8, 145.5, 136.8, 132.3, 131.7, 128.8, 128.0, 124.0, 121.1, 114.1, 108.5, 61.8, 40.6, 26.3; FTIR (cm^{-1}): 3221, 2975, 2218, 1697, 1469, 1451, 699; mp = 188-190 °C (EtOAc:hexanes); HRMS (ESI) m/z, calculated for [$C_{15}H_{13}N_2OS^+$]: 269.0743; found: 269.0747.

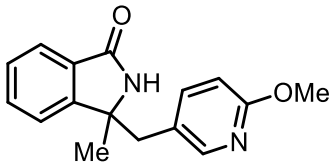


(**19**) According to the general protocol: (COD)Pd(CH_2SiMe_3)$_2$ (38.9 mg, 0.1 mmol, 0.1 equiv), N-phenoxy-2-(prop-1-en-2-yl)benzamide **1** (253.3 mg, 1.00 mmol), (1-methyl-1H-indazol-5-yl)boronic acid (528 mg, 3.0 mmol, 3.0 equiv), 4Å MS (500 mg), P(OCH_2CF_3)$_3$ (98.4 mg, 0.3 mmol, 0.3 equiv), and anhydrous MeCN (10.0 mL) were combined under N_2. The reaction was stirred at 80 °C for 24 h under N_2 environment. The reaction was worked up according to the general procedure and purified by chromatography (linear gradient, 12:88 EtOAc:hexanes to 100:0 EtOAc:hexanes) to afford amide **19** (149.4 mg, 52%) as a white solid. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.81 (s, 1H), 7.69 (d, *J* = 7.2 Hz, 1H), 7.54-7.52 (m, 1H), 7.39-7.36 (m, 3H), 7.21-7.19 (m, 3H), 7.05 (d, *J* = 8.4 Hz, 1H), 6.13 (brs, 1H), 3.98 (s, 3H), 3.15 (d, *J* = 13.8 Hz, 1H), 2.98 (d, *J* = 13.8 Hz, 1H), 1.46 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 169.9, 151.7, 139.1, 132.4, 131.9, 131.5, 128.9, 128.1, 127.9, 124.0, 123.8, 122.3, 121.6, 108.4, 62.5, 46.4, 35.5, 25.6; FTIR (cm^{-1}): 3287, 1685, 1639, 1412, 1095, 755, 732; mp = > 200 °C Decompose (EtOAc:hexanes); HRMS (ESI) m/z, calculated for [$C_{18}H_{18}N_3O^+$]: 292.1444; found: 292.1448.

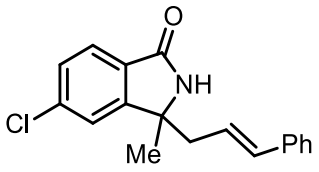


(**21**) According to the general protocol: (COD)Pd(CH_2SiMe_3)$_2$ (38.9 mg, 0.1 mmol, 0.1 equiv), N-phenoxy-2-(prop-1-en-2-yl)benzamide **1** (253.3 mg, 1.00 mmol), (2-methoxypyridin-3-yl)boronic acid acid (459 mg, 3.0 mmol, 3.0 equiv), 4Å MS (500 mg), P(OCH_2CF_3)$_3$ (98.4 mg, 0.3 mmol, 0.3 equiv), and anhydrous MeCN (10.0 mL) were combined under N_2. The reaction was stirred at 80 °C for 24 h under N_2 environment. The reaction was worked up according to the general

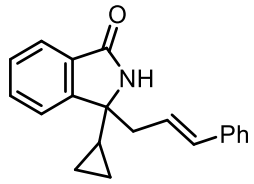
procedure and purified by chromatography (linear gradient, 12:88 EtOAc:hexanes to 100:0 EtOAc:hexanes) to afford amide **21** (207.8 mg, 78%) as a white solid. ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.29 (s, 1H), 7.80 (d, *J* = 3.6 Hz, 1H), 7.57 (d, *J* = 7.2 Hz, 1H), 7.43-7.41 (m, 1H), 7.35 (d, *J* = 7.2 Hz, 1H), 7.27-7.25 (m, 1H), 7.00 (d, *J* = 7.2 Hz, 1H), 6.47-6.45 (m, 1H), 3.67 (s, 3H), 3.14 (d, *J* = 13.8 Hz, 1H), 3.01 (d, *J* = 13.8 Hz, 1H), 1.51 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 170.1, 162.2, 150.9, 145.4, 139.8, 131.6, 131.3, 128.0, 123.3, 121.9, 118.5, 116.3, 62.7, 53.0, 39.0, 26.2; FTIR (cm^{-1}): 3214, 3074, 2975, 1696, 1615, 1586, 1467, 1413, 783, 735; mp = 115-118 °C (EtOAc:hexanes); HRMS (ESI) m/z, calculated for [$C_{16}H_{17}N_2O_2^+$]: 269.1285; found: 269.1289.



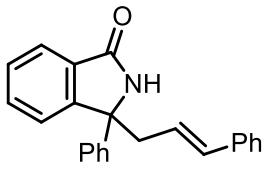
(**22**) According to the general protocol: (COD)Pd(CH_2SiMe_3)$_2$ (38.9 mg, 0.1 mmol, 0.1 equiv), N-phenoxy-2-(prop-1-en-2-yl)benzamide **1** (253.3 mg, 1.00 mmol), (6-methoxypyridin-3-yl)boronic acid (459 mg, 3.0 mmol, 3.0 equiv), 4Å MS (500 mg), P(OCH_2CF_3)$_3$ (98.4 mg, 0.3 mmol, 0.3 equiv), and anhydrous MeCN (10.0 mL) were combined under N_2. The reaction was stirred at 80 °C for 24 h under N_2 environment. The reaction was worked up according to the general procedure and purified by chromatography (linear gradient, 12:88 EtOAc:hexanes to 100:0 EtOAc:hexanes) to afford amide **22** (172.1 mg, 64%) as a white solid. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.79-7.64 (m, 3H), 7.53-7.50 (m, 1H), 7.36-7.35 (m, 2H), 7.11 (d, *J* = 8.4 Hz, 1H), 6.45 (d, *J* = 8.4 Hz, 1H), 3.75 (s, 3H), 3.01 (d, *J* = 13.8 Hz, 1H), 2.94 (d, *J* = 13.8 Hz, 1H), 1.54 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 170.5, 163.0, 150.7, 147.8, 140.4, 131.9, 131.8, 128.2, 123.8, 123.7, 121.4, 109.8, 62.5, 53.2, 42.6, 26.0; FTIR (cm^{-1}): 3219, 2975, 1695, 1609, 1493, 1392, 1028, 762; mp = 165-167 °C (EtOAc:hexanes); HRMS (ESI) m/z, calculated for [$C_{16}H_{17}N_2O_2^+$]: 269.1285; found: 269.1289.



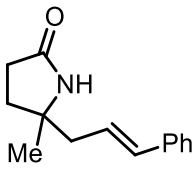
(**23**) According to the general protocol: Pd(OAc)$_2$ (11.3 mg, 0.05 mmol, 0.05 equiv), 4-chloro-N-phenoxy-2-(prop-1-en-2-yl)benzamide **S2** (287.8 mg, 1.00 mmol), (*E*)-styrylboronic acid (444 mg, 3.0 mmol, 3.0 equiv), 4Å MS (500 mg), P(OCH_2CF_3)$_3$ (49.2 mg, 0.15 mmol, 0.15 equiv), and anhydrous MeCN (10.0 mL) were combined under N_2. The reaction was stirred at 80 °C for 24 h under N_2 environment. The reaction was worked up according to the general procedure and purified by chromatography (linear gradient, 10:90 EtOAc:hexanes to 80:20 EtOAc:hexanes) to afford amide **23** (288.6 mg, 97%) as a white solid. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.96 (s, 1H), 7.73 (d, *J* = 7.8 Hz, 1H), 7.43 (dd, *J* = 8.4, 1.2 Hz, 1H), 7.40 (s, 1H), 7.25-7.24 (m, 4H), 7.21-7.18 (m, 1H), 6.39 (d, *J* = 16.2 Hz, 1H), 6.04-5.94 (m, 1H), 2.72 (dd, *J* = 13.8, 7.8 Hz, 1H), 2.58 (dd, *J* = 13.8, 7.2 Hz, 1H), 1.56 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 169.0, 153.3, 138.3, 136.8, 135.0, 129.8, 128.7, 128.4, 127.5, 126.2, 125.1, 123.1, 121.8, 61.8, 43.8, 25.4; FTIR (cm^{-1}): 3202, 3060, 1696, 1611, 1450, 1421, 742, 693; mp = 177-179 °C (EtOAc:hexanes); HRMS (ESI) m/z, calculated for [$C_{18}H_{17}ClNO^+$]: 298.0993; found: 298.0998.



(**24**) According to the general protocol: $Pd(OAc)_2$ (11.3 mg, 0.05 mmol, 0.05 equiv), 2-(1-cyclopropylvinyl)-N-phenoxybenzamide **S4** (279.3 mg, 1.00 mmol), (*E*)-styrylboronic acid (444 mg, 3.0 mmol, 3.0 equiv), 4Å MS (500 mg), $P(OCH_2CF_3)_3$ (49.2 mg, 0.15 mmol, 0.15 equiv), and anhydrous MeCN (10.0 mL) were combined under N_2. The reaction was stirred at 80 °C for 24 h under N_2 environment. The reaction was worked up according to the general procedure and purified by chromatography (linear gradient, 5:95 EtOAc:hexanes to 70:30 EtOAc:hexanes) to afford amide **24** (158.9 mg, 55%) as a white solid. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.64 (d, *J* = 7.2 Hz, 1H), 7.59 (s, 1H), 7.42-7.40 (m, 1H), 7.31-7.28 (m, 2H), 7.11-7.02 (m, 5H), 6.22 (d, *J* = 15.6 Hz, 1H), 5.97-5.92 (m, 1H), 2.71 (dd, *J* = 13.8, 7.2 Hz, 1H), 2.58 (dd, *J* = 13.8, 7.2 Hz, 1H), 1.26-1.21 (m, 1H), 0.45-0.41 (m, 1H), 0.26-0.21 (m, 1H), 0.14-0.09 (m, 1H), 0.02--0.03 (m, 1H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 168.6, 148.3, 134.7, 131.7, 129.5, 129.1, 126.0, 125.7, 124.8, 123.7, 121.5, 121.3, 119.2, 61.8, 40.8, 16.0, 0.0, -2.3; FTIR (cm^{-1}): 3199, 3078, 1693, 1468, 1344, 747, 693; mp = 151-153 °C (EtOAc:hexanes); HRMS (ESI) m/z, calculated for [$C_{20}H_{20}NO^+$]: 290.1539; found: 290.1543.

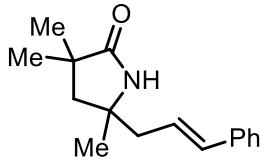


(**25**) According to the general protocol: $Pd(OAc)_2$ (11.3 mg, 0.05 mmol, 0.05 equiv), N-phenoxy-2-(1-phenylvinyl)benzamide **S5** (316 mg, 1.00 mmol), (*E*)-styrylboronic acid (444 mg, 3.0 mmol, 3.0 equiv), 4Å MS (500 mg), $P(OCH_2CF_3)_3$ (49.2 mg, 0.15 mmol, 0.15 equiv), and anhydrous MeCN (10.0 mL) were combined under N_2. The reaction was stirred at 80 °C for 24 h under N_2 environment. The reaction was worked up according to the general procedure and purified by chromatography (linear gradient, 10:90 EtOAc:hexanes to 80:20 EtOAc:hexanes) to afford amide **25** (289.7 mg, 90%) as a white solid. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.81 (d, *J* = 7.2 Hz, 1H), 7.54-7.51 (m, 3H), 7.44-7.42 (m, 2H), 7.37-7.35 (m, 3H), 7.30-7.28 (m, 1H), 7.23-7.17 (m, 5H), 6.44 (d, *J* = 15.6 Hz, 1H), 5.96-5.91 (m, 1H), 3.44 (dd, *J* = 13.8, 6.0 Hz, 1H), 2.94 (dd, *J* = 13.8, 7.8 Hz, 1H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 170.4, 151.1, 140.9, 136.7, 135.0, 132.3, 130.3, 129.0, 128.4, 128.3, 127.8, 127.5, 126.2, 125.5, 124.0, 123.3, 122.3, 66.8, 43.0; FTIR (cm^{-1}): 3196, 3059, 1695, 1612, 1447, 966, 764, 695; mp = 190-192 °C (EtOAc:hexanes); HRMS (ESI) m/z, calculated for [$C_{23}H_{20}NO^+$]: 326.1539; found: 326.1544.

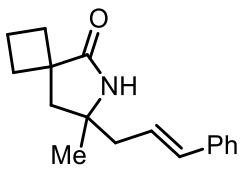


(**26**) According to the general protocol: $(COD)Pd(CH_2SiMe_3)_2$ (38.9 mg, 0.1 mmol, 0.1 equiv), 4-methyl-N-phenoxypent-4-enamide **S8** (205.2 mg, 1.00 mmol), (*E*)-styrylboronic acid (444 mg, 3.0 mmol, 3.0 equiv), 4Å MS (500 mg), $P(OCH_2CF_3)_3$ (98.4 mg, 0.3 mmol, 0.3 equiv), and anhydrous MeCN (10.0 mL) were combined under N_2. The reaction was stirred at 100 °C for 24 h in sealed tube. The reaction was worked up according to the general procedure and purified by chromatography (linear gradient, 16:84 EtOAc:hexanes to 100:0 EtOAc:hexanes) to afford amide **26** (197.5 mg, 92%) as a white solid. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.35 (d, *J* = 7.8 Hz, 2H), 7.30-7.28 (m, 2H), 7.22-7.20 m, 1H), 7.02 (s, 1H), 6.45 (d, *J* = 15.6 Hz, 1H), 6.20-6.15 (m, 1H), 2.43-2.36 (m, 4H), 2.06-2.01 (m, 1H), 1.87-1.82 (m, 1H), 1.30 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 177.3, 137.0, 134.0, 128.4, 127.3, 126.1, 124.4, 59.3, 45.5, 32.9, 30.5, 27.4; FTIR (cm^{-1}): 3207, 2966,

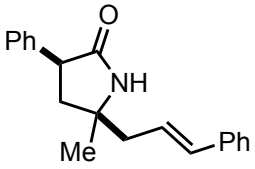
1692, 969, 744, 694; mp = 74-77 °C (EtOAc:hexanes); HRMS (ESI) m/z, calculated for $[C_{16}H_{17}N_2O_2^+]$: 216.1383; found: 216.1388.



(**27**) According to the general protocol: (COD)Pd(CH_2SiMe_3)$_2$ (38.9 mg, 0.1 mmol, 0.1 equiv), 2,2,4-trimethyl-N-phenoxypent-4-enamide **S9** (233.3 mg, 1.00 mmol), (*E*)-styrylboronic acid (444 mg, 3.0 mmol, 3.0 equiv), 4Å MS (500 mg), P(OCH_2CF_3)$_3$ (98.4 mg, 0.3 mmol, 0.3 equiv), and anhydrous MeCN (10.0 mL) were combined under N_2. The reaction was stirred at 100 °C for 24 h in sealed tube. The reaction was worked up according to the general procedure and purified by chromatography (linear gradient, 12:88 EtOAc:hexanes to 100:0 EtOAc:hexanes) to afford amide **27** (222.5 mg, 91%) as a white solid. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.36 (d, *J* = 7.2 Hz, 2H), 7.32-7.30 (m, 2H), 7.24-7.22 (m, 1H), 6.46 (d, *J* = 15.6 Hz, 1H), 6.19-6.14 (m, 1H), 6.00 (s, 1H), 2.45-2.42 (m, 1H), 2.39-2.36 (m, 1H), 2.04 (d, *J* = 13.2 Hz, 1H), 1.85 (d, *J* = 13.2 Hz, 1H), 1.34 (s, 3H), 1.26 (s, 3H), 1.23 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 181.9, 137.0, 134.0, 128.4, 127.3, 126.1, 124.8, 55.7, 48.0, 47.0, 40.8, 29.2, 27.7, 27.1; FTIR (cm^{-1}): 3196, 2965, 1687, 1468, 1449, 1362, 974, 745, 694; mp = 153-155 °C (EtOAc:hexanes); HRMS (ESI) m/z, calculated for $[C_{16}H_{22}NO^+]$: 244.1696; found: 244.1699.

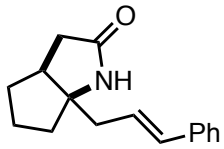


(**28**) According to the general protocol: (COD)Pd(CH_2SiMe_3)$_2$ (38.9 mg, 0.1 mmol, 0.1 equiv), 1-(2-methylallyl)-N-phenoxycyclobutanecarboxamide **S10** (245.3 mg, 1.00 mmol), (*E*)-styrylboronic acid (444 mg, 3.0 mmol, 3.0 equiv), 4Å MS (500 mg), P(OCH_2CF_3)$_3$ (98.4 mg, 0.3 mmol, 0.3 equiv), and anhydrous MeCN (10.0 mL) were combined under N_2. The reaction was stirred at 100 °C for 24 h in sealed tube. The reaction was worked up according to the general procedure and purified by chromatography (linear gradient, 12:88 EtOAc:hexanes to 100:0 EtOAc:hexanes) to afford amide **28** (218.2 mg, 86%) as a white solid. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.36 (d, *J* = 7.2 Hz, 2H), 7.31-7.28 (m, 2H), 7.22-7.20 (m, 1H), 6.65 (s, 1H), 6.43 (d, *J* = 15.6 Hz, 1H), 6.20-6.15 (m, 1H), 2.54-2.49 (m, 2H), 2.39-2.31 (m, 2H), 2.20 (d, *J* = 12.6 Hz, 1H), 2.11-2.06 (m, 1H), 2.03 (d, *J* = 13.2 Hz, 1H), 1.97-1.90 (mz, 3H), 1.26 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 180.6, 137.0, 134.0, 128.5, 127.3, 126.1, 124.7, 56.9, 48.1, 46.1, 46.0, 32.1, 31.7, 27.9, 16.6; FTIR (cm^{-1}): 3192, 2927, 1696, 1369, 972, 746, 694; mp = 128-130 °C (EtOAc:hexanes); HRMS (ESI) m/z, calculated for $[C_{17}H_{22}NO^+]$: 256.1696; found: 256.1699.

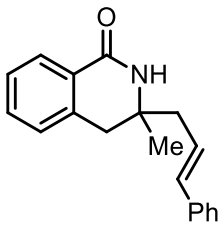


(**29**) According to the general protocol: (COD)Pd(CH_2SiMe_3)$_2$ (38.9 mg, 0.1 mmol, 0.1 equiv), 4-methyl-N-phenoxy-2-phenylpent-4-enamide **S11** (281.4 mg, 1.00 mmol), (*E*)-styrylboronic acid (444 mg, 3.0 mmol, 3.0 equiv), 4Å MS (500 mg), P(OCH_2CF_3)$_3$ (98.4 mg, 0.3 mmol, 0.3 equiv), and anhydrous MeCN (10.0 mL) were combined under N_2. The reaction was stirred at 100 °C for 24 h in sealed tube. The reaction was worked up according to the general procedure and purified by chromatography (linear gradient, 12:88 EtOAc:hexanes to 100:0 EtOAc:hexanes) to afford amide **29** (220 mg, 76%) as a white solid. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.52 (s, 1H), 7.22-7.08 (m, 10H), 6.31

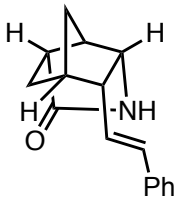
(d, *J* = 15.6 Hz, 1H), 6.14-609 (m, 1H), 3.74 (t, *J* = 9.6 Hz, 1H), 2.38-2.30 (m, 2H), 2.25-2.21 (m, 1H), 2.05-2.01 m, 1H), 1.22 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 177.0, 139.5, 136.9, 134.0, 128.5, 128.4, 128.1, 127.2, 126.8, 126.0, 124.4, 57.1, 47.6, 45.8, 43.2, 26.8; FTIR (cm^{-1}): 3195, 3028, 2966, 1693, 1497, 1449, 971, 748, 695; mp = 149-151 °C (EtOAc:hexanes); HRMS (ESI) m/z, calculated for [$C_{20}H_{22}NO^+$]: 292.1696; found: 292.1699.



(**30**) According to the general protocol: (COD)Pd(CH_2SiMe_3)$_2$ (38.9 mg, 0.1 mmol, 0.1 equiv), 2-(2-methylenecyclopentyl)-N-phenoxyacetamide **S12** (231.3 mg, 1.00 mmol), (*E*)-styrylboronic acid (444 mg, 3.0 mmol, 3.0 equiv), 4Å MS (500 mg), P(OCH_2CF_3)$_3$ (98.4 mg, 0.3 mmol, 0.3 equiv), and anhydrous MeCN (10.0 mL) were combined under N_2. The reaction was stirred at 100 °C for 24 h in sealed tube. The reaction was worked up according to the general procedure and purified by chromatography (linear gradient, 12:88 EtOAc:hexanes to 100:0 EtOAc:hexanes) to afford amide **30** (197.7 mg, 82%) as a white solid. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.27-7.19 (m, 4H), 7.14-7.12 (m, 1H), 6.86 (d, *J* = 6.0 Hz, 1H), 6.39 (d, *J* = 15.6 Hz, 1H), 6.13-6.08 (m, 1H), 2.58-2.54 (m, 1H), 2.45 (dd, *J* = 13.8, 6.0 Hz, 1H), 2.39-2.34 (m, 2H), 1.97 (dd, *J* = 17.4, 2.4 Hz, 1H), 1.83-1.77 (m, 1H), 1.70-1.63 (m, 2H), 1.57-1.52 (m, 2H), 1.43-1.41 (m, 1H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 177.7, 137.2, 134.0, 128.6, 127.4, 126.2, 124.8, 70.6, 44.4, 42.2, 39.2, 38.6, 34.9, 24.5; FTIR (cm^{-1}): 3197, 2951, 2866, 1689, 1422, 968, 748, 695; mp = 117-120 °C (EtOAc:hexanes); HRMS (ESI) m/z, calculated for [$C_{16}H_{20}NO^+$]: 242.1539; found: 242.1543.



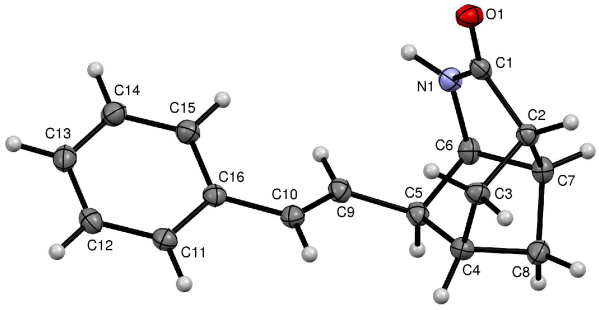
(**31**) According to the general protocol: Pd(OAc)$_2$ (11.3 mg, 0.05 mmol, 0.05 equiv), 2-(2-methylallyl)-N-phenoxybenzamide **S6** (267.4 mg, 1.00 mmol), (*E*)-styrylboronic acid (444 mg, 3.0 mmol, 3.0 equiv), 4Å MS (500 mg), P(OCH_2CF_3)$_3$ (49.2 mg, 0.15 mmol, 0.15 equiv), and anhydrous MeCN (10.0 mL) were combined under N_2. The reaction was stirred at 80 °C for 24 h under N_2 environment. The reaction was worked up according to the general procedure and purified by chromatography (linear gradient, 10:90 EtOAc:hexanes to 80:20 EtOAc:hexanes) to afford amide **31** (157.1 mg, 55%) as a white solid. ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.00 (d, *J* = 7.8 Hz, 1H), 7.38 (m, 1H), 7.29-7.11 (m, 7H), 6.34 (d, *J* = 15.6 Hz, 1H), 6.14-6.09 (m, 2H), 2.96 (d, *J* = 15.6 Hz, 1H), 2.85 (d, *J* = 15.6 Hz, 1H), 2.40-2.39 (m, 2H), 1.27 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 165.5, 137.3, 136.9, 134.7, 132.5, 128.6, 128.1, 128.0, 127.9, 127.6, 127.1, 126.2, 123.8, 54.7, 44.9, 40.0, 26.7; FTIR (cm^{-1}): 3193, 3060, 1664, 1578, 1462, 1392, 742; mp = 120-122 °C (EtOAc:hexanes); HRMS (ESI) m/z, calculated for [$C_{19}H_{20}NO^+$]: 278.1539; found: 278.1544.

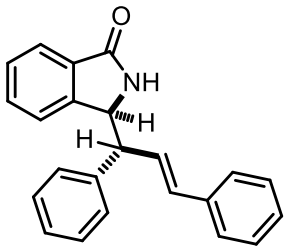


(**36**) According to the general protocol: (COD)Pd(CH_2SiMe_3)$_2$ (77.8 mg, 0.2 mmol, 0.2 equiv), N-phenoxybicyclo[2.2.1]hept-5-ene-2-carboxamide **35** (229.3 mg, 1.00 mmol), (*E*)-styrylboronic acid (444 mg, 3.0 mmol, 3.0 equiv), 4Å MS (500 mg), P(OCH_2CF_3)$_3$ (196.8 mg, 0.6 mmol, 0.6 equiv), and anhydrous MeCN (10.0 mL) were combined under N_2. The reaction was stirred at 120 °C for 24 h in sealed tube. The reaction was worked up

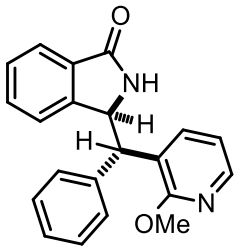
according to the general procedure and purified by chromatography (linear gradient, 18:82 EtOAc:hexanes to 100:0 EtOAc:hexanes) to afford amide **36** (118.4 mg, 49%) as a white solid. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.26 (d, *J* = 7.2 Hz, 2H), 7.20-7.18 (m, 2H), 7.12-7.10 (m, 1H), 6.39-6.36 (m, 2H), 6.17 (dd, *J* = 15.6, 8.4 Hz, 1H), 3.60 (t, *J* = 6.0 Hz, 1H), 3.05 (t, *J* = 4.2 Hz, 1H), 2.36-2.33 (m, 1H), 2.28 (s, 1H), 2.20 (dd, *J* = 10.8, 4.2 Hz, 1H), 1.89 (d, *J* = 13.2 Hz, 1H), 1.67-1.65 (m, 1H), 1.55 (d, *J* = 10.2 Hz, 1H), 1.47 (d, *J* = 10.8 Hz, 1H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 182.9, 137.1, 132.8, 128.3, 127.2, 126.5, 126.1, 57.1, 48.4, 47.7, 42.9, 41.5, 37.1, 28.6; FTIR (cm^{-1}): 3218, 2956, 2875, 1698, 1449, 1257, 967, 747, 694; mp = 145-148 °C (EtOAc:hexanes); HRMS (ESI) m/z, calculated for [$C_{16}H_{18}NO^{+}$]: 240.1383; found: 240.1386.

A small sample of compound **36** was dissolved in EtOAc under air and recrystallized *via* slow vapor diffusion of hexanes at room temperature to give an X-ray quality crystal. See below for full crystallographic details.





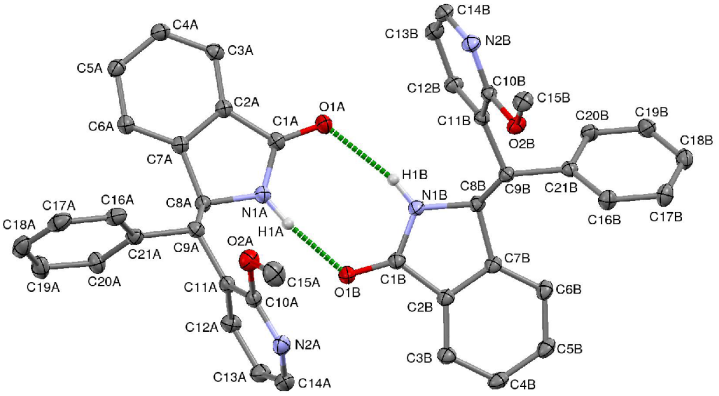
(**41**) According to the general protocol: $Pd(OAc)_2$ (11.3 mg, 0.05 mmol, 0.05 equiv), (*E*)-N-phenoxy-2-styrylbenzamide (315.4 mg, 1.00 mmol), (*E*)-styrylboronic acid (444 mg, 3.0 mmol, 3.0 equiv), 4Å MS (500 mg), $P(OCH_2CF_3)_3$ (49.2 mg, 0.15 mmol, 0.15 equiv), and anhydrous MeCN (10.0 mL) were combined under N_2. The reaction was stirred at 80 °C for 24 h under N_2 environment. The reaction was worked up according to the general procedure and purified by chromatography (linear gradient, 7:93 EtOAc:hexanes to 60:40 EtOAc:hexanes) to afford amide **41** (234.1 mg, 72%) as a colorless oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.73 (d, *J* = 7.2 Hz, 1H), 7.34-7.32 (m, 3H), 7.29-7.18 (m, 8H), 7.15-7.13 (m, 1H), 6.71 (brs, 1H), 6.46-6.24 (m, 2H), 6.35 (dd, *J* = 15.6, 8.4 Hz, 1H), 4.84 (d, *J* = 8.4 Hz, 1H), 3.47 (t, *J* = 8.4 Hz, 1H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 170.3, 145.4, 140.2, 136.4, 133.3, 132.1, 131.4, 129.0, 128.5, 128.4, 128.3, 128.2, 127.8, 127.6, 126.4, 123.7, 123.6, 60.6, 54.6; FTIR (cm^{-1}): 3208, 1696, 1468, 1028, 744, 695; HRMS (ESI) m/z, calculated for [$C_{23}H_{20}NO^{+}$]: 326.1539; found: 326.1543.

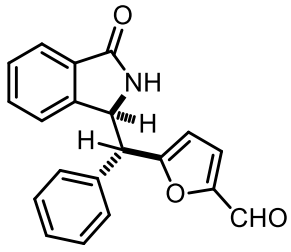


(**42**) According to the general protocol: $(COD)Pd(CH_2SiMe_3)_2$ (38.9 mg, 0.1 mmol, 0.1 equiv), (*E*)-N-phenoxy-2-styrylbenzamide (315.4 mg, 1.00 mmol), (2-methoxypyridin-3-yl)boronic acid acid (459 mg, 3.0 mmol, 3.0 equiv), 4Å MS (500 mg), $P(OCH_2CF_3)_3$ (98.4 mg, 0.3 mmol, 0.3 equiv), and anhydrous MeCN (10.0 mL) were combined under N_2. The reaction was stirred at 80 °C for 24 h under N_2 environment. The reaction was worked up according to the general procedure and

purified by chromatography (linear gradient, 12:88 EtOAc:hexanes to 100:0 EtOAc:hexanes) to afford amide **42** (179.8 mg, 54%) as a white solid. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.96 (d, *J* = 3.6 Hz, 1H), 7.70 (d, *J* = 7.2 Hz, 1H), 7.61 (d, *J* = 7.2 Hz, 1H), 7.32-7.17 (m, 7H), 6.80-6.78 (m, 1H), 6.60 (s, 1H), 6.31 (d, *J* = 7.8 Hz, 1H), 5.40 (d, *J* = 10.2 Hz, 1H), 4.17 (d, *J* = 10.2 Hz, 1H), 3.81 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 170.4, 161.4, 146.0, 145.6, 139.9, 136.2, 132.0, 131.3, 128.84, 128.81, 128.4, 127.5, 124.0, 123.8, 123.7, 117.0, 58.7, 53.6, 50.1; FTIR (cm^{-1}): 3203, 3061, 1697, 1585, 1463, 1260, 748, 700; mp = 198-200 °C (EtOAc:hexanes); HRMS (ESI) m/z, calculated for [$C_{21}H_{19}N_2O_2^+$]: 331.1441; found: 331.1446.

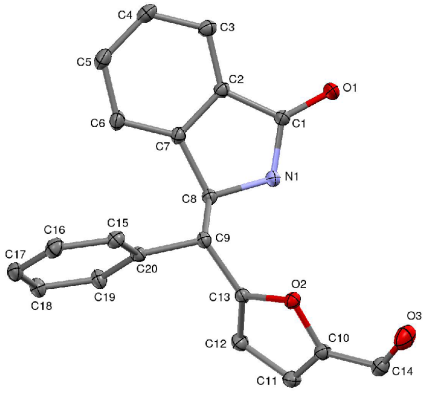
A small sample of compound **42** was dissolved in EtOAc under air and recrystallized *via* slow vapor diffusion of hexanes at room temperature to give an X-ray quality crystal (See below for full crystallographic details).

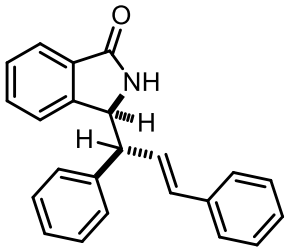




(**43**) According to the general protocol: (COD)Pd(CH_2SiMe_3)$_2$ (38.9 mg, 0.1 mmol, 0.1 equiv), (*E*)-N-phenoxy-2-styrylbenzamide (315.4 mg, 1.00 mmol), (5-formylfuran-2-yl)boronic acid (420 mg, 3.0 mmol, 3.0 equiv), 4Å MS (500 mg), P(OCH_2CF_3)$_3$ (98.4 mg, 0.3 mmol, 0.3 equiv), and anhydrous MeCN (10.0 mL) were combined under N_2. The reaction was stirred at 80 °C for 24 h under N_2 environment. The reaction was worked up according to the general procedure and purified by chromatography (linear gradient, 12:88 EtOAc:hexanes to 100:0 EtOAc:hexanes) to afford amide **43** (152.2 mg, 48%) as a white solid. ^{1}H NMR (600 MHz, $CDCl_3$) δ 9.51 (s, 1H), 7.71 (d, *J* = 7.8 Hz, 1H), 7.36-7.22 (m, 7H), 7.13-7.09 (m, 2H), 6.33 (d, *J* = 7.8 Hz, 1H), 6.30 (d, *J* = 3.6 Hz, 1H), 5.24 (d, *J* = 9.0 Hz, 1H), 4.10 (d, *J* = 9.0 Hz, 1H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 177.5, 170.6, 160.6, 152.5, 144.7, 136.8, 132.2, 131.5, 129.1, 128.9, 128.7, 128.4, 123.8, 123.7, 110.7, 59.4, 51.1; FTIR (cm-1): 3205, 1698, 1678, 1513, 1469, 759, 702; mp = 182-184 °C (EtOAc:hexanes); HRMS (ESI) m/z, calculated for [$C_{20}H_{16}NO_3^+$]: 318.1125; found: 29318.1129.

A small sample of compound **43** was dissolved in EtOAc under air and recrystallized *via* slow vapor diffusion of hexanes at room temperature to give an X-ray quality crystal (See below for full crystallographic details).

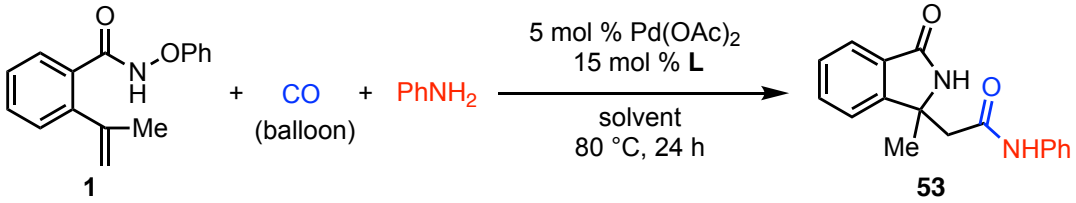




(**45**) According to the general protocol: $Pd(OAc)_2$ (11.3 mg, 0.05 mmol, 0.05 equiv), (*Z*)-N-phenoxy-2-styrylbenzamide (315.4 mg, 1.00 mmol), (*E*)-styrylboronic acid (444 mg, 3.0 mmol, 3.0 equiv), 4Å MS (500 mg), $P(OCH_2CF_3)_3$ (49.2 mg, 0.15 mmol, 0.15 equiv), and anhydrous MeCN (10.0 mL) were combined under N_2. The reaction was stirred at 80 °C for 24 h under N_2 environment. The reaction was worked up according to the general procedure and purified by chromatography (linear gradient, 7:93 EtOAc:hexanes to 60:40 EtOAc:hexanes) to afford amide **45** (162.6 mg, 50%) as a white solid. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.74 (d, *J* = 6.0 Hz, 1H), 7.39-7.18 (m, 13H), 6.48-6.41 (m, 2H), 6.36 (s, 1H), 4.89 (d, *J* = 8.4 Hz, 1H), 3.52 (t, *J* = 8.4 Hz, 1H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 170.1, 145.6, 140.2, 136.6, 133.3, 132.2, 131.3, 129.1, 128.6, 128.4, 128.3, 127.9, 127.8, 127.4, 126.4, 124.1, 123.8, 60.3, 54.6; FTIR (cm^{-1}): 2923, 1669, 1494, 1340, 1261, 697; mp = 153-155 °C (EtOAc:hexanes); HRMS (ESI) m/z, calculated for $[C_{23}H_{20}NO^+]$: 326.1539; found: 326.1543.

5. Synthesis of Lactams *via* Aza-Heck-Carbonylation Reaction

5.1. Reaction Optimization of Aniline [a]



Entry	L	Solvent	Yield [%] [b]
1	$P(OCH_2CF_3)_3$	MeCN	40
2	$P(OCH_2CF_3)_3$	dioxane	2
3	$P(OCH_2CF_3)_3$	DCE	15
4	$P(OCH_2CF_3)_3$	*t*-BuOH	21
5	$P(OCH_2CF_3)_3$	PhMe	43
6	$P(OCH_2CF_3)_3$	Ph-F	27
7	$P(OCH_2CF_3)_3$	Ph-CF_3	58
8	P(O*i*-Pr)$_3$	Ph-CF_3	68

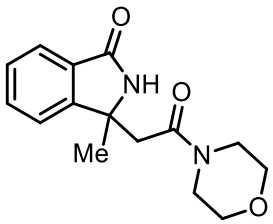
[a] Unless otherwise noted, reactions run with 0.2 mmol **1** and 0.3 mmol $PhNH_2$. [b] Yield calculated by ^{1}H NMR with 1,3,5-trimethoxybenzene as internal standard.

5.2. Synthesis of Lactams *via* Aza-Heck-Carbonylation Reaction

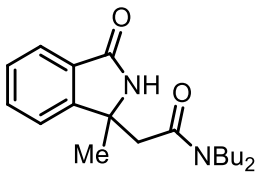
NOTE: CARBON MONOXIDE IS A HIGHLY TOXIC GAS. THESE PROCEDURES SHOULD ONLY BE CARRIED OUT BY A HIGHLY TRAINED EXPERIMENTALIST WITH EXPERTISE IN HANDLING TOXIC GASES. ADDITIONALLY, PROPER PERSONAL PROTECTIVE GEAR AND CARBON MONOXIDE MONITORS SHOULD BE EMPLOYED WHEN PREFORMING THESE PROCESSES. PLEASE CONSULT WITH YOUR LOCAL SAFETY OFFICALS BEFORE ATTEMPTING.

General Protocol: A flame-dried Schlenk flask equipped with a magnetic stir bar and rubber septum was attached to a double manifold and cooled under vacuum. The flask was backfilled with N_2, the septum was removed and $Pd(OAc)_2$ (0.05 equiv), hydroxamate (1.0 mmol, 1.0 equiv) were added to the flask. The septum was replaced, and the flask was evacuated and backfilled with nitrogen four times. Then anhydrous solvent (10.0 mL), $P(OCH_2CF_3)_3$ (0.15 equiv), and amine (1.5 mmol, 1.5 equiv) were added sequentially to the flask via syringe. The resulting solution was purged with CO which was introduced *via* a needle attached balloon for 30 s, then heated in an oil bath with rapid stirring at the indicated temperature for 24 h under CO which was introduced *via* a needle attached balloon. Upon completion, the reaction was cooled to room temperature, opened to air, and the reaction mixture was then diluted with EtOAc, filtered through celite. The suspension was adsorbed onto Celite via rotary evaporation. The resulting powder was

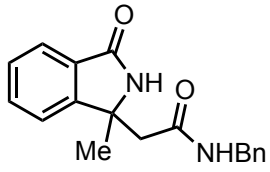
directly chromatographed on silica gel (5-20 μm particle size) to yield the desired product.



(**47**) According to the general protocol: $Pd(OAc)_2$ (11.3 mg, 0.05 mmol, 0.05 equiv), N-phenoxy-2-(prop-1-en-2-yl)benzamide **1** (253.3 mg, 1.00 mmol), morpholine (131 mg, 1.5 mmol, 1.5 equiv), $P(OCH_2CF_3)_3$ (49.2 mg, 0.15 mmol, 0.15 equiv), and anhydrous dioxane (10.0 mL) were combined under N_2. The resulting solution was purged with a CO balloon for 30 s, then heated in an oil bath with rapid stirring at 80 °C for 24 h under a CO balloon. The reaction was worked up according to the general procedure and purified by chromatography (linear gradient, 50:50 EtOAc:hexanes to 100:0 EtOAc:hexanes) to afford amide **47** (250.2 mg, 91%) as a white solid. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.74 (d, *J* = 7.2 Hz, 1H), 7.57 (s, 1H), 7.50-7.48 (m, 1H), 7.39-7.35 (m, 2H), 3.64-3.55 (m, 4H), 3.53-3.49 (m, 2H), 3.39-3.34 (m, 1H), 3.32-3.28 (m, 1H), 2.95 (d, *J* = 16.2 Hz, 1H), 2.32 (d, *J* = 16.2 Hz, 1H), 1.59 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 168.5, 168.4, 151.9, 131.8, 130.7, 128.2, 123.9, 120.9, 66.5, 66.1, 59.3, 45.7, 41.9, 41.6, 24.8; FTIR (cm^{-1}): 3424, 3305, 2970, 2858, 1698, 1637, 1467, 1232, 1115, 1038, 734, 699; mp = 153-155 °C (EtOAc:hexanes); HRMS (ESI) m/z, calculated for [$C_{15}H_{19}N_2O_3^+$]: 275.1390; found: 275.1393.

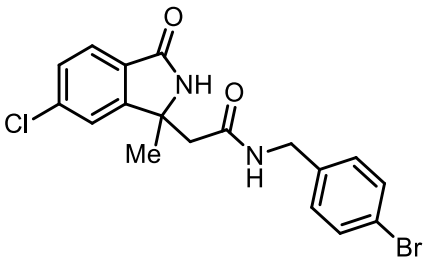


(**48**) According to the general protocol: $Pd(OAc)_2$ (11.3 mg, 0.05 mmol, 0.05 equiv), N-phenoxy-2-(prop-1-en-2-yl)benzamide **1** (253.3 mg, 1.00 mmol), dibutylamine (194 mg, 1.5 mmol, 1.5 equiv), $P(OCH_2CF_3)_3$ (49.2 mg, 0.15 mmol, 0.15 equiv), and anhydrous dioxane (10.0 mL) were combined under N_2. The resulting solution was purged with a CO balloon for 30 s, then heated in an oil bath with rapid stirring at 80 °C for 24 h under a CO balloon. The reaction was worked up according to the general procedure and purified by chromatography (linear gradient, 25:75 EtOAc:hexanes to 100:0 EtOAc:hexanes) to afford amide **48** (264.3 mg, 84%) as a white solid. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.79 (d, *J* = 7.8 Hz, 1H), 7.59 (s, 1H), 7.54-7.51 (m, 1H), 7.43-4.40 (m, 1H), 7.36 (d, *J* = 7.8 Hz, 1H), 3.33-3.25 (m, 2H), 3.14-3.11 (m, 2H), 2.96 (d, *J* = 16.2 Hz, 1H), 2.28 (d, *J* = 15.6 Hz, 1H), 1.59 (s, 3H), 1.51-1.44 (m, 4H), 1.31-1.23 (m, 4H), 0.91-0.87 (m, 6H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 169.3, 168.4, 152.2, 131.8, 131.1, 128.2, 124.0, 120.9, 59.5, 47.6, 45.8, 42.1, 31.0, 29.7, 24.7, 20.2, 19.9, 13.7, 13.6. FTIR (cm^{-1}): 3425, 3290, 2959, 2932, 2873, 1703, 1631, 1468, 1375, 1216, 1140, 764, 697; mp = 82-84 °C (EtOAc:hexanes); HRMS (ESI) m/z, calculated for [$C_{19}H_{29}N_2O_2^+$]: 317.2224; found: 317.2227.

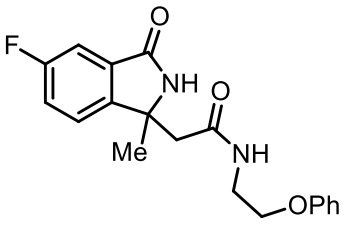


(**49**) According to the general protocol: $Pd(OAc)_2$ (11.3 mg, 0.05 mmol, 0.05 equiv), N-phenoxy-2-(prop-1-en-2-yl)benzamide **1** (253.3 mg, 1.00 mmol), phenylmethanamine (161 mg, 1.5 mmol, 1.5 equiv), $P(OCH_2CF_3)_3$ (49.2 mg, 0.15 mmol, 0.15 equiv), and anhydrous dioxane (10.0 mL) were combined under N_2. The resulting solution was purged with a CO balloon for 30 s, then heated in an oil bath with rapid stirring at 80 °C for 24 h under a CO balloon. The reaction was worked up according to the general procedure and purified by chromatography (linear gradient, 7:93 EtOAc:hexanes to 40:60 EtOAc:hexanes) to afford amide **49** (258.2 mg, 88%) as a

colorless oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.64 (d, *J* = 7.2 Hz, 1H), 7.58 (s, 1H), 7.44 (td, *J* = 7.8, 1.2 Hz, 1H), 7.32 (td, *J* = 7.2, 0.6 Hz, 1H), 7.27 (d, *J* = 7.2 Hz, 1H), 7.21-7.19 (m, 2H), 7.17-7.14 (m, 1H), 7.11 (d, *J* = 7.2 Hz, 2H), 6.53 (t, *J* = 4.8 Hz, 1H), 4.34 (dd, *J* = 15.0, 6.0 Hz, 1H), 4.27 (dd, *J* = 14.0, 5.4 Hz, 1H), 2.73 (d, *J* = 15.0 Hz, 1H), 2.36 (d, *J* = 14.4 Hz, 1H), 1.50 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 169.6, 169.1, 151.4, 138.0, 132.0, 130.7, 128.4, 128.2, 127.5, 127.2, 123.7, 121.2, 59.9, 45.5, 43.3, 25.1; FTIR (cm^{-1}): 3289, 3064, 2975, 1692, 1615, 1551, 1469, 1349, 735, 698; HRMS (ESI) m/z, calculated for [$C_{18}H_{19}N_2O_2^+$]: 295.1441; found: 295.1445.

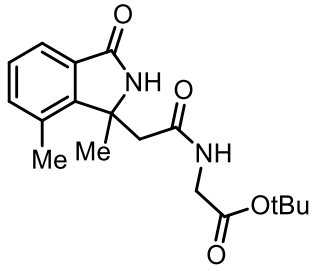


(**50**) According to the general protocol: $Pd(OAc)_2$ (11.3 mg, 0.05 mmol, 0.05 equiv), 4-chloro-N-phenoxy-2-(prop-1-en-2-yl)benzamide **S2** (287.8 mg, 1.00 mmol), (4-bromophenyl)methanamine (279.2 mg, 1.5 mmol, 1.5 equiv), $P(OCH_2CF_3)_3$ (49.2 mg, 0.15 mmol, 0.15 equiv), and anhydrous dioxane (10.0 mL) were combined under N_2. The resulting solution was purged with a CO balloon for 30 s, then heated in an oil bath with rapid stirring at 80 °C for 24 h under a CO balloon. The reaction was worked up according to the general procedure and purified by chromatography (linear gradient, 12:88 EtOAc:hexanes to 90:10 EtOAc:hexanes) to afford amide **50** (184.1mg, 45%) as a white solid. ^{1}H NMR (600 MHz, CD_3OD) δ 7.67 (d, *J* = 7.8 Hz, 1H), 7.60 (d, *J* = 1.2 Hz, 1H), 7.49 (dd, *J* = 7.8, 1.8 Hz, 1H), 7.43-7.41 (m, 2H), 7.04 (d, *J* = 8.4 Hz, 2H), 4.24 (d, *J* = 15.0 Hz, 1H), 4.20 (d, *J* = 15.0 Hz, 1H), 2.81 (d, *J* = 14.4 Hz, 1H), 2.70 (d, *J* = 14.4 Hz, 1H), 1.59 (s, 3H); ^{13}C NMR (150 MHz, CD_3OD) δ 171.0, 170.3, 154.4, 139.6, 139.1, 132.6, 131.0, 130.5, 130.1, 126.0, 123.8, 121.9, 61.6, 45.8, 43.3, 26.3; FTIR (cm^{-1}): 3262, 1691, 1650, 1553, 1406, 834, 784; mp = > 200 °C Decompose (EtOAc:hexanes); HRMS (ESI) m/z, calculated for [$C_{18}H_{17}ClBrN_2O_2^+$]: 407.0156; found: 407.0165.

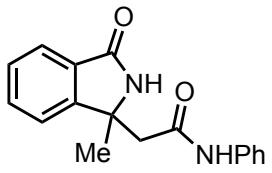


(**51**) According to the general protocol: $Pd(OAc)_2$ (11.3 mg, 0.05 mmol, 0.05 equiv), 5-fluoro-N-phenoxy-2-(prop-1-en-2-yl)benzamide **S1** (271.3 mg, 1.00 mmol), 2-phenoxyethanamine (205.8 mg, 1.5 mmol, 1.5 equiv), $P(OCH_2CF_3)_3$ (49.2 mg, 0.15 mmol, 0.15 equiv), and anhydrous dioxane (10.0 mL) were combined under N_2. The resulting solution was purged with a CO balloon for 30 s, then heated in an oil bath with rapid stirring at 80 °C for 24 h under a CO balloon. The reaction was worked up according to the general procedure and purified by chromatography (linear gradient, 8:92 EtOAc:hexanes to 70:30 EtOAc:hexanes) to afford amide **51** (210.1mg, 61%) as a white solid. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.81 (s, 1H), 7.41 (dd, *J* = 7.8, 2.4 Hz, 1H), 7.34 (dd, *J* = 8.4, 4.2 Hz, 1H), 7.27-7.24 (m, 2H), 7.20 (td, *J* = 9.0, 2.4 Hz, 1H), 6.96-6.93 (m, 1H), 6.84 (d, *J* = 7.8 Hz, 2H), 6.51-6.49 (m, 1H), 4.01-3.95 (m, 2H), 3.68-3.60 (m, 2H), 2.79 (d, *J* = 14.4 Hz, 1H), 2.44 (d, *J* = 14.4 Hz, 1H), 1.57 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 169.5, 167.8 (d, *J* = 3.3 Hz), 162.9 (d, *J* = 246.5 Hz), 158.3, 146. 9 (d, *J* = 2.2 Hz), 133.2 (d, *J* = 8.4 Hz), 129.5, 122.7 (d, *J* = 8.4 Hz), 121.3, 119.5 (d, *J* = 23.6 Hz), 114.4, 110.7(d, *J* = 23.3 Hz), 66.4, 59.6, 45.8, 39.0, 25.2; ^{19}F NMR (565 MHz, $CDCl_3$) δ -112.6 (m); FTIR (cm^{-1}): 3300, 3069, 2935, 1698, 1657, 1487, 1243, 1223,

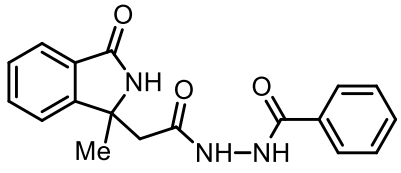
755, 692; mp = 134-136 °C (EtOAc:hexanes); HRMS (ESI) m/z, calculated for [$C_{19}H_{20}FN_2O_3^+$]: 343.1452; found: 343.1455.



(**52**) According to the general protocol: $Pd(OAc)_2$ (11.3 mg, 0.05 mmol, 0.05 equiv), 3-methyl-N-phenoxy-2-(prop-1-en-2-yl)benzamide **S3** (267.3 mg, 1.00 mmol), tert-butyl-2-aminoacetate (196.8 mg, 1.5 mmol, 1.5 equiv), $P(OCH_2CF_3)_3$ (49.2 mg, 0.15 mmol, 0.15 equiv), and anhydrous dioxane (10.0 mL) were combined under N_2. The resulting solution was purged with a CO balloon for 30 s, then heated in an oil bath with rapid stirring at 80 °C for 24 h under a CO balloon. The reaction was worked up according to the general procedure and purified by chromatography (linear gradient, 8:92 EtOAc:hexanes to 70:30 EtOAc:hexanes) to afford amide **52** (310.1mg, 93%) as a white solid. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.92 (s, 1H), 7.50-7.48 (m, 1H), 7.33-7.30 (m, 1H), 7.25-7.24 (m, 2H), 3.97 (dd, *J* = 18.0, 6.0 Hz, 1H), 3.73 (dd, *J* = 18.0, 5.4 Hz, 1H), 3.08 (d, *J* = 13.8 Hz, 1H), 2.45 (d, *J* = 13.8 Hz, 1H), 2.43 (s, 3H), 1.63 (s, 3H), 1.38 (s, 9H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 170.0, 169.5, 169.0, 148.6, 134.4, 132.2, 131.4, 128.4, 121.5, 82.2, 60.7, 43.7, 42.1, 27.9, 23.0, 18.6; FTIR (cm^{-1}): 3299, 2980, 2935, 1743, 1695, 1553, 1368, 1226, 1156, 735; mp = 143-145 °C (EtOAc:hexanes); HRMS (ESI) m/z, calculated for [$C_{18}H_{25}FNO_4^+$]: 333.1809; found: 333.1811.

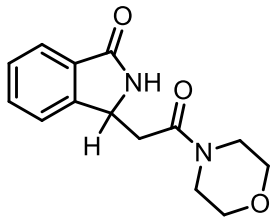


(**53**) According to the general protocol: $Pd(OAc)_2$ (11.3 mg, 0.05 mmol, 0.05 equiv), N-phenoxy-2-(prop-1-en-2-yl)benzamide **1** (253.3 mg, 1.00 mmol), aniline (140 mg, 1.5 mmol, 1.5 equiv), P(O*i*-Pr)$_3$ (31.3 mg, 0.15 mmol, 0.15 equiv), and anhydrous Ph-CF_3 (10.0 mL) were combined under N_2. The resulting solution was purged with a CO balloon for 30 s, then heated in an oil bath with rapid stirring at 80 °C for 24 h under a CO balloon. The reaction was worked up according to the general procedure and purified by chromatography (linear gradient, 20:80 EtOAc:hexanes to 100:0 EtOAc:hexanes) to afford amide **53** (207.2 mg, 77%) as a white solid. ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.30 (s, 1H), 7.75 (s, 1H), 7.66 (d, *J* = 7.2 Hz, 1H), 7.47-7.45 (m, 1H), 7.39 (d, *J* = 7.8 Hz, 2H), 7.34-7.30 (m, 2H), 7.18-7.15 (m, 2H), 7.00-6.98 (m, 1H), 2.89 (d, *J* = 14.4 Hz, 1H), 2.50 (d, *J* = 15.0 Hz, 1H), 1.55 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 169.4, 168.1, 151.5, 137.6, 132.3, 130.7, 128.9, 128.5, 124.5, 124.0, 121.3, 120.2, 60.1, 46.6, 25.2; FTIR (cm^{-1}): 3293, 3057, 1669, 1600, 1548, 1443, 1308, 755, 696; mp = 159-161 °C (EtOAc:hexanes); HRMS (ESI) m/z, calculated for [$C_{17}H_{17}N_2O_2^+$]: 281.1285; found: 281.1289.

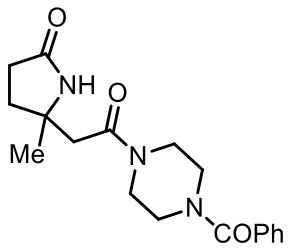


(**54**) According to the general protocol: $Pd(OAc)_2$ (11.3 mg, 0.05 mmol, 0.05 equiv), N-phenoxy-2-(prop-1-en-2-yl)benzamide **1** (253.3 mg, 1.00 mmol), benzohydrazide (204.3 mg, 1.5 mmol, 1.5 equiv), $P(OCH_2CF_3)_3$ (49.2 mg, 0.15 mmol, 0.15 equiv), and anhydrous dioxane (10.0 mL) were combined under N_2. The resulting solution was purged with a CO balloon for 30 s, then heated in an oil bath with rapid stirring at 80 °C for 24 h under a CO balloon. The reaction was worked up according to the general

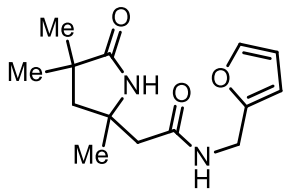
procedure and purified by chromatography (linear gradient, 10:90 EtOAc:hexanes to 90:10 EtOAc:hexanes) to afford amide **54** (202.6 mg, 63%) as a white solid. ^{1}H NMR (600 MHz, CD_3OD) δ 7.89-7.88 (m, 2H), 7.76 (d, *J* = 7.8 Hz, 1H), 7.67-7.64 (m, 2H), 7.59-7.56 (m, 1H), 7.53-7.47 (m, 3H), 2.93 (d, *J* = 14.4 Hz, 1H), 2.60 (d, *J* = 14.4 Hz, 1H), 1.68 (s, 3H); ^{13}C NMR (150 MHz, CD_3OD) δ 171.4, 171.2, 169.3, 153.2, 133.7, 133.4, 131.8, 129.7, 128.7, 124.6, 123.1, 61.5, 44.5, 25.6; FTIR (cm^{-1}): 3240, 1690, 1605, 1310, 695; mp = > 200 °C Decompose (EtOAc:hexanes); HRMS (ESI) m/z, calculated for [$C_{18}H_{18}N_3O_3^+$]: 324.1343; found: 324.1346.



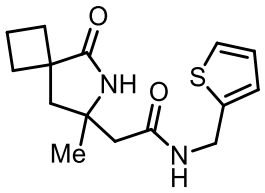
(**55**) According to the general protocol: $Pd(OAc)_2$ (11.3 mg, 0.05 mmol, 0.05 equiv), N-phenoxy-2-vinylbenzamide **S7** (240 mg, 1.00 mmol), morpholine (131 mg, 1.5 mmol, 1.5 equiv), $P(OCH_2CF_3)_3$ (49.2 mg, 0.15 mmol, 0.15 equiv), and anhydrous dioxane (10.0 mL) were combined under N_2. The resulting solution was purged with CO for 30 s, then heated in an oil bath with rapid stirring at 80 °C for 24 h under 20 psi CO. The reaction was worked up according to the general procedure and purified by chromatography (linear gradient, 80:20 EtOAc:hexanes to 100:0 EtOAc:hexanes) to afford amide **55** (160.8 mg, 62%) as a colorless oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.83 (d, *J* = 7.8 Hz, 1H), 7.55-7.53 (m, 1H), 7.47-7.44 (m, 1H), 7.41 (d, *J* = 7.2 Hz, 1H), 7.11 (s, 1H), 5.01 (dd, *J* = 10.8, 3.0 Hz, 1H), 3.71-3.59 (m, 6H), 3.42-3.35 (m, 2H), 3.00 (dd, *J* = 16.2, 3.0 Hz, 1H), 2.38 (dd, *J* = 16.2, 10.2 Hz, 1H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 169.7, 168.7, 146.3, 132.2, 131.7, 128.4, 124.0, 122.2, 66.7, 66.3, 53.1, 45.7, 42.0, 38.8. FTIR (cm^{-1}): 3428, 3290, 2859, 1702, 1640, 1467, 1273, 1115, 1031, 733, 697; HRMS (ESI) m/z, calculated for [$C_{14}H_{17}N_2O_3^+$]: 261.1234; found: 261.1237.



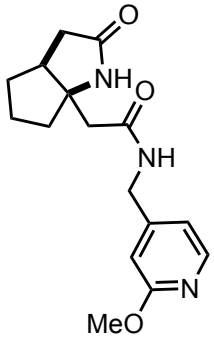
(**56**) According to the general protocol: $Pd(OAc)_2$ (11.3 mg, 0.05 mmol, 0.05 equiv), 4-methyl-N-phenoxypent-4-enamide **S8** (205.3 mg, 1.00 mmol), phenyl(piperazin-1-yl)methanone (286 mg, 1.5 mmol, 1.5 equiv), $P(OCH_2CF_3)_3$ (49.2 mg, 0.15 mmol, 0.15 equiv), and anhydrous dioxane (10.0 mL) were combined under N_2. The resulting solution was purged with a CO balloon for 30 s, then heated in an oil bath with rapid stirring at 100 °C for 24 h under a CO balloon. The reaction was worked up according to the general procedure and purified by chromatography (linear gradient, 2:98 acetone:hexanes to 16:84 acetone:hexanes) to afford amide **56** (212.1mg, 64%) as a white solid. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.42-7.35 (m, 5H), 6.87 (s, 1H), 3.70-3.31 (m, 8H), 2.59 (brs, 1H), 2.45 (d, *J* = 15.6 Hz, 1H), 2.39-2.26 (m, 2H), 2.00-1.93 (m, 2H), 1.36 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 176.2, 170.5, 168.9, 134.9, 130.0, 128.5, 126.9, 57.4, 43.6, 35.0, 29.4, 26.9; FTIR (cm^{-1}): 3413, 3269, 2969, 1694, 1633, 1429, 1008, 732, 710; mp = > 200 °C Decompose (acetone:hexanes); HRMS (ESI) m/z, calculated for [$C_{18}H_{24}N_3O_3^+$]: 330.1812; found: 330.1815.



(**57**) According to the general protocol: $Pd(OAc)_2$ (11.3 mg, 0.05 mmol, 0.05 equiv), 2,2,4-trimethyl-N-phenoxypent-4-enamide **S9** (233.4 mg, 1.00 mmol), furan-2-ylmethanamine (145.8 mg, 1.5 mmol, 1.5 equiv), $P(OCH_2CF_3)_3$ (49.2 mg, 0.15 mmol, 0.15 equiv), and anhydrous dioxane (10.0 mL) were combined under N_2. The resulting solution was purged with a CO balloon for 30 s, then heated in an oil bath with rapid stirring at 100 °C for 24 h under a CO balloon. The reaction was worked up according to the general procedure and purified by chromatography (linear gradient, 80:20 EtOAc:hexanes to 100:0 EtOAc:hexanes) to afford amide **57** (160.6, 61%) as a white solid. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.32 (d, *J* = 1.2 Hz, 1H), 6.90 (s, 1H), 6.60 (s, 1H), 6.28 (dd, *J* = 3.0, 1.8 Hz, 1H), 6.19 (d, *J* = 3.0 Hz, 1H), 4.42-4.35 (m, 2H), 2.40 (s, 2H), 2.02 (d, *J* = 13.2 Hz, 1H), 1.88 (d, *J* = 13.2 Hz, 1H), 1.34 (s, 3H), 1.20 (s, 3H), 1.14 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 181.2, 169.9, 151.1, 142.1, 110.4, 107.4, 54.2, 49.2, 48.9, 40.2, 36.3, 28.9, 27.5, 27.1; FTIR (cm^{-1}): 3279, 2967, 2870, 1656, 1549, 1410, 1254, 733; mp = 175-178 °C (EtOAc:hexanes); HRMS (ESI) m/z, calculated for [$C_{14}H_{21}N_2O_3^+$]: 265.1547; found: 265.1550.



(**58**) According to the general protocol: $Pd(OAc)_2$ (11.3 mg, 0.05 mmol, 0.05 equiv), 1-(2-methylallyl)-N-phenoxycyclobutanecarboxamide **S10** (245.3mg, 1.00 mmol), thiophen-2-ylmethanamine (169.8 mg, 1.5 mmol, 1.5 equiv), $P(OCH_2CF_3)_3$ (49.2 mg, 0.15 mmol, 0.15 equiv), and anhydrous dioxane (10.0 mL) were combined under N_2. The resulting solution was purged with a CO balloon for 30 s, then heated in an oil bath with rapid stirring at 100 °C for 24 h under a CO balloon. The reaction was worked up according to the general procedure and purified by chromatography (linear gradient, 80:20 EtOAc:hexanes to 100:0 EtOAc:hexanes) to afford amide **58** (195.4, 67%) as a white solid. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.16 (dd, *J* = 4.8, 1.2 Hz, 1H), 6.93-6-89 (m, 3H), 6.84 (s, 1H), 4.57 (dd, *J* = 15.6, 6.0 Hz, 1H), 4.51 (dd, *J* = 15.6, 6.0 Hz, 1H), 2.44-2.39 (m, 3H), 2.31 (d, *J* = 14.4 Hz, 1H), 2.15 (d, *J* = 12.6 Hz, 1H), 2.10 (d, *J* = 12.6 Hz, 1H), 2.03-1.98 (m, 1H), 1.96-1.83 (m, 3H), 1.25 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 180.0, 169.8, 141.0, 126.8, 125.8, 125.0, 55.4, 48.8, 47.9, 45.3, 38.1, 32.5, 30.8, 27.6, 16.6. FTIR (cm^{-1}): 3281, 3072, 2930, 1650, 1549, 1264, 1161, 700; mp = 101-103 °C (EtOAc:hexanes); HRMS (ESI) m/z, calculated for [$C_{15}H_{21}SN_2O^+$]: 293.1318; found: 293.1321.



(**59**) According to the general protocol: $Pd(OAc)_2$ (11.3 mg, 0.05 mmol, 0.05 equiv), 2-(2-methylenecyclopentyl)-N-phenoxyacetamide **S12** (231.3 mg, 1.00 mmol), (2-methoxypyridin-4-yl)methanamine (207.3 mg, 1.5 mmol, 1.5 equiv), $P(OCH_2CF_3)_3$ (49.2 mg, 0.15 mmol, 0.15 equiv), and anhydrous dioxane (10.0 mL) were combined under N_2. The resulting solution was purged with a CO balloon for 30 s, then heated in an oil bath with rapid stirring at 100 °C for 24 h under a CO balloon. The reaction was worked up according to the general procedure and purified by chromatography (linear gradient, 25:75 EtOAc:hexanes to 100:0 EtOAc:hexanes) to afford amide **59** (212.1 mg, 66%) as a colorless oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.01 (d, *J* = 4.8 Hz, 1H), 7.31-7.29 (m, 1H), 6.91 (s, 1H),

6.68 (d, *J* = 4.8 Hz, 1H), 6.53 (s, 1H), 4.30-4.22 (m, 2H), 3.84 (s, 3H), 2.57-2.49 (m, 4H), 1.89 (d, *J* = 15.0 Hz, 1H), 1.81-1.78 (m, 2H), 1.60-1.57 (m, 2H), 1.52-1.47 (m, 1H), 1.42-1.40 (m, 1H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 177.1, 170.7, 164.5, 150.2, 147.0, 115.5, 108.7, 68.4, 53.3, 46.2, 42.6, 42.1, 39.4, 38.0, 34.4, 24.1; FTIR (cm^{-1}): 3278, 3061, 2950, 2868, 1665, 1614, 1565, 1399, 1225, 1046, 734; HRMS (ESI) m/z, calculated for [$C_{16}H_{22}N_3O_3^+$]: 304.1656; found: 304.1659.

6. Crystallographic Details

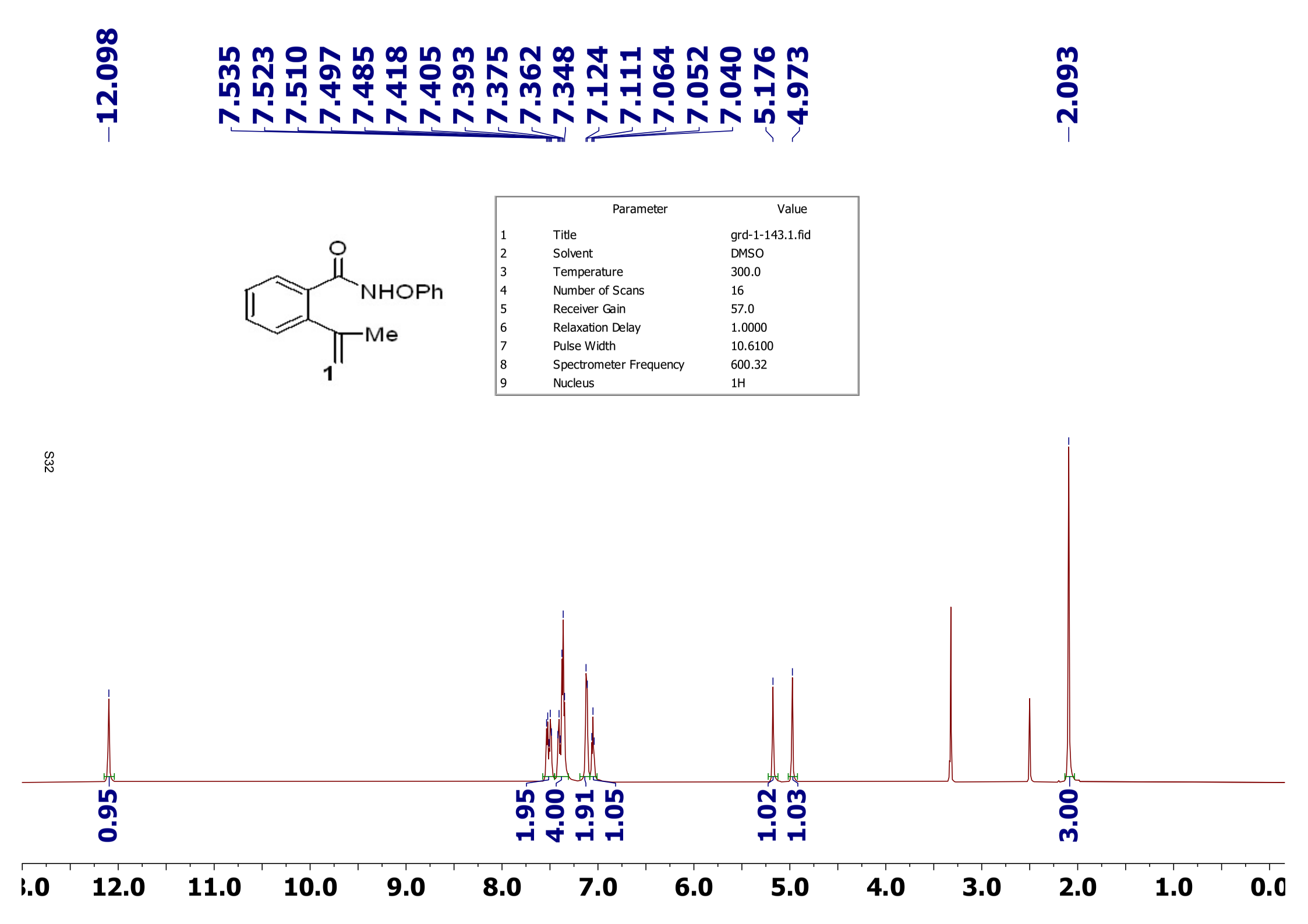
X-ray structural analysis for **36**: Crystals were mounted using viscous oil onto a plastic mesh and cooled to the data collection temperature (100K). Data were collected on a Bruker-AXS APEX II DUO CCD diffractometer with Cu-Kα radiation (λ = 1.54178 Å) focused Mo-Kα radiation (λ = 0.71073 Å). Unit cell parameters were obtained from 36 to 48 data frames, 0.5º ω, from different sections of the Ewald sphere. The unit-cell dimensions, equivalent reflections and systematic absences in the diffraction data are consistent, uniquely, with $P2_1/c$. The data were treated with multi-scan absorption corrections.[7] Structures were solved using intrinsic phasing methods[8] and refined with full-matrix, least-squares procedures on F^2.[9] The structures have been deposited at the Cambridge Structural Database under the following CCDC deposition numbers: 2071160.

X-ray structural analysis for **42**: Crystals were mounted using viscous oil onto a plastic mesh and cooled to the data collection temperature (150K). Data were collected on a Bruker-AXS APEX II DUO CCD diffractometer with Cu-Kα radiation (λ = 1.54178 Å) focused with Goebel mirrors. Unit cell parameters were obtained from 36 to 48 data frames, 0.5º ω, from different sections of the Ewald sphere. No symmetry higher than triclinic was observed and refinement in the centrosymmetric space group option, *P*-1, yielded chemically reasonable and computationally stable results of refinement. The data were treated with multi-scan absorption corrections.[7] Structures were solved using intrinsic phasing methods[8] and refined with full-matrix, least-squares procedures on F^2.[9] Two symmetry-unique but chemically identical compound molecules were found in the asymmetric unit (i.e. Z = 2 and Z' = 2) together with a chloroform solvent molecule, disordered in two positions, with chemically equivalent atoms in non-crystallographical symmetry restrained disordered contributions treated with equal atomic displacement parameter restraints, having 88/12 refined site occupancy ratio. Non-hydrogen atoms were refined with anisotropic displacement parameters. Other than the H-atom on each of the disordered chloroform solvent molecule locations, treated as idealized contributions with geometrically calculated positions and with U_{iso} equal to 1.2 U_{eq} of the attached carbon atom, all other H-atoms were located from the difference map and refined independently with isotropic parameters. Atomic scattering factors are contained in the SHELXTL program library.[9] The structures have been deposited at the Cambridge Structural Database under the following CCDC deposition numbers: 2071161.

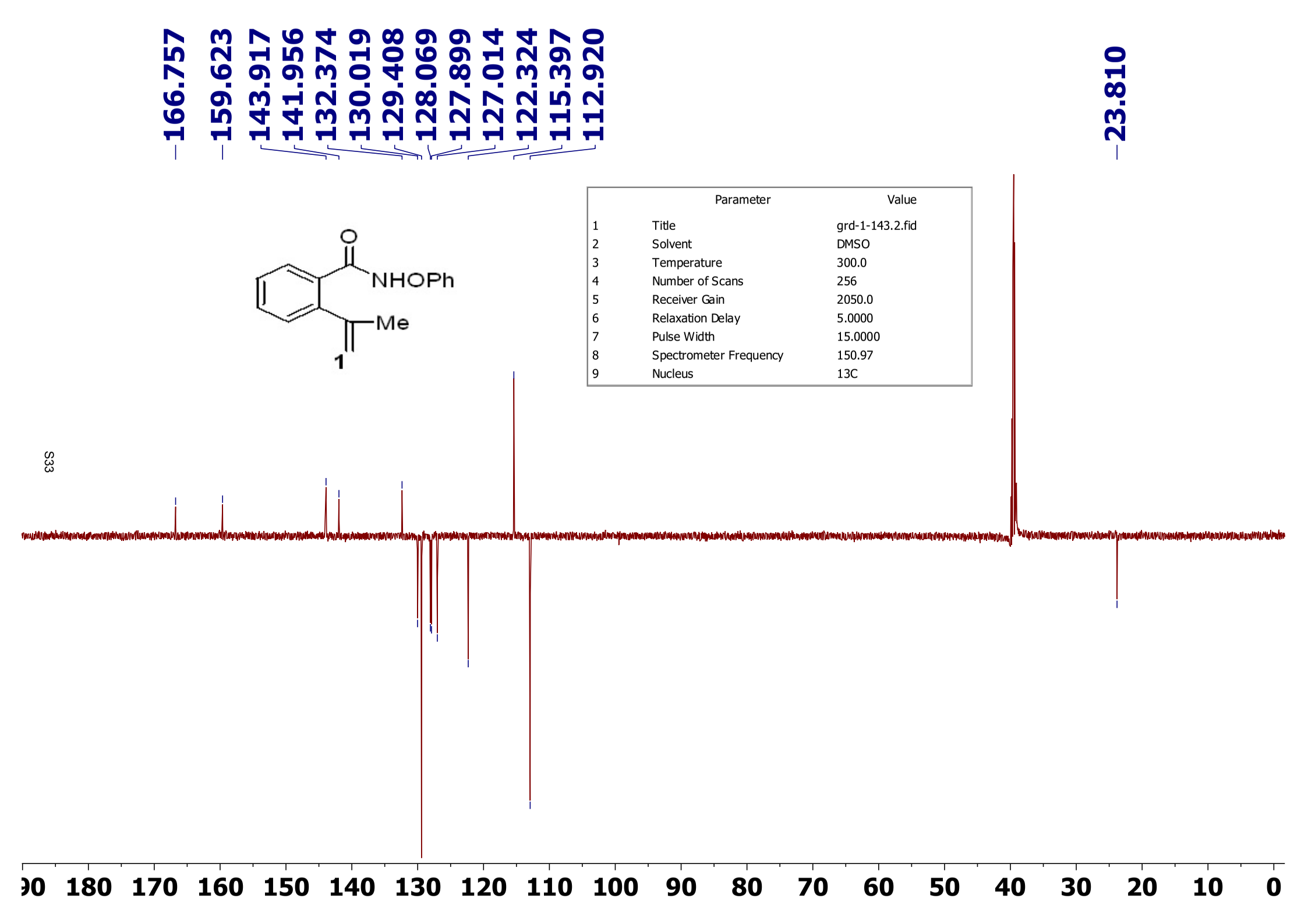
X-ray structural analysis for **43**: Crystals were mounted using viscous oil onto a plastic mesh and cooled to the data collection temperature (150K). Data were collected on a Bruker-AXS APEX II DUO CCD diffractometer with Cu-Kα radiation (λ = 1.54178 Å) focused Mo-Kα radiation (λ = 0.71073 Å). Unit cell parameters were obtained from 36 to 48 data frames, 0.5º ω, from different sections of the Ewald sphere. The unit-cell dimensions, equivalent reflections and systematic absences in the diffraction data are consistent, uniquely, with $P2_1/n$. The data were treated with multi-scan absorption corrections.[7] Structures were solved using intrinsic phasing methods[8] and refined with full-matrix, least-squares procedures on F^2.[9] The structures have been deposited at the Cambridge Structural Database under the following CCDC deposition numbers: 2071162.

7. References

(1) A. B. Pangborn, M. A. Giardello, R. H. Grubbs, R. K. Rosen and F. J. Timmers, *Organometallics*, 1996, **15**, 1518
(2) J. R. McAtee, S. E. S. Martin, D. T. Ahneman, K. A. Johnson and D. A. Watson, *Angew. Chem. Int. Ed.*, 2012, **51**, 3663
(3) B. N. Hemric, K. Shen and Q. Wang, *J. Am. Chem. Soc.*, 2016, **138**, 5813
(4) Z. Pan, S. Wang, J. T. Brethorst and C. J. Douglas, *J. Am. Chem. Soc.,* 2018, **140**, 3331
(5) A. B. Smith III, B. H. Toder, S. J. Branca and R. K. Dieter, *J. Am. Chem. Soc.*, 1981, **103**, 1996
(6) S. A. Shuler, G. Yin, S. B. Krause, C. M. Vesper and D. A. Watson, *J. Am. Chem. Soc*., 2016, **138**, 13830
(7) Apex3, Bruker AXS Inc.: Madison, WI, 2015
(8) G. M. Sheldrick, *Acta. Cryst*., **2015**, *A71*, 3
(9) G. M. Sheldrick, *Acta. Cryst*., **2015**, *C71*, 3



	Parameter	Value
1	Title	grd-1-143.1.fid
2	Solvent	DMSO
3	Temperature	300.0
4	Number of Scans	16
5	Receiver Gain	57.0
6	Relaxation Delay	1.0000
7	Pulse Width	10.6100
8	Spectrometer Frequency	600.32
9	Nucleus	1H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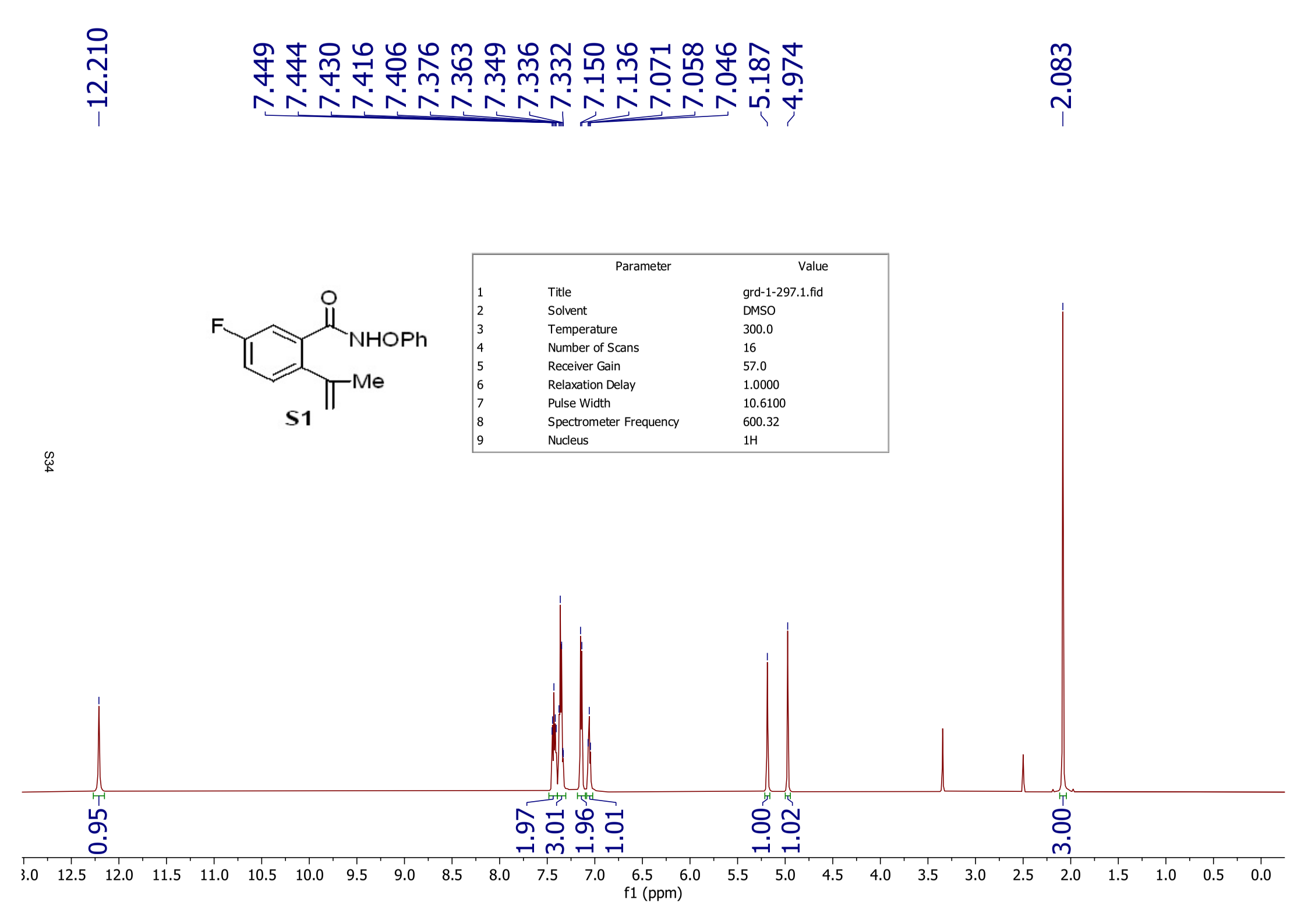
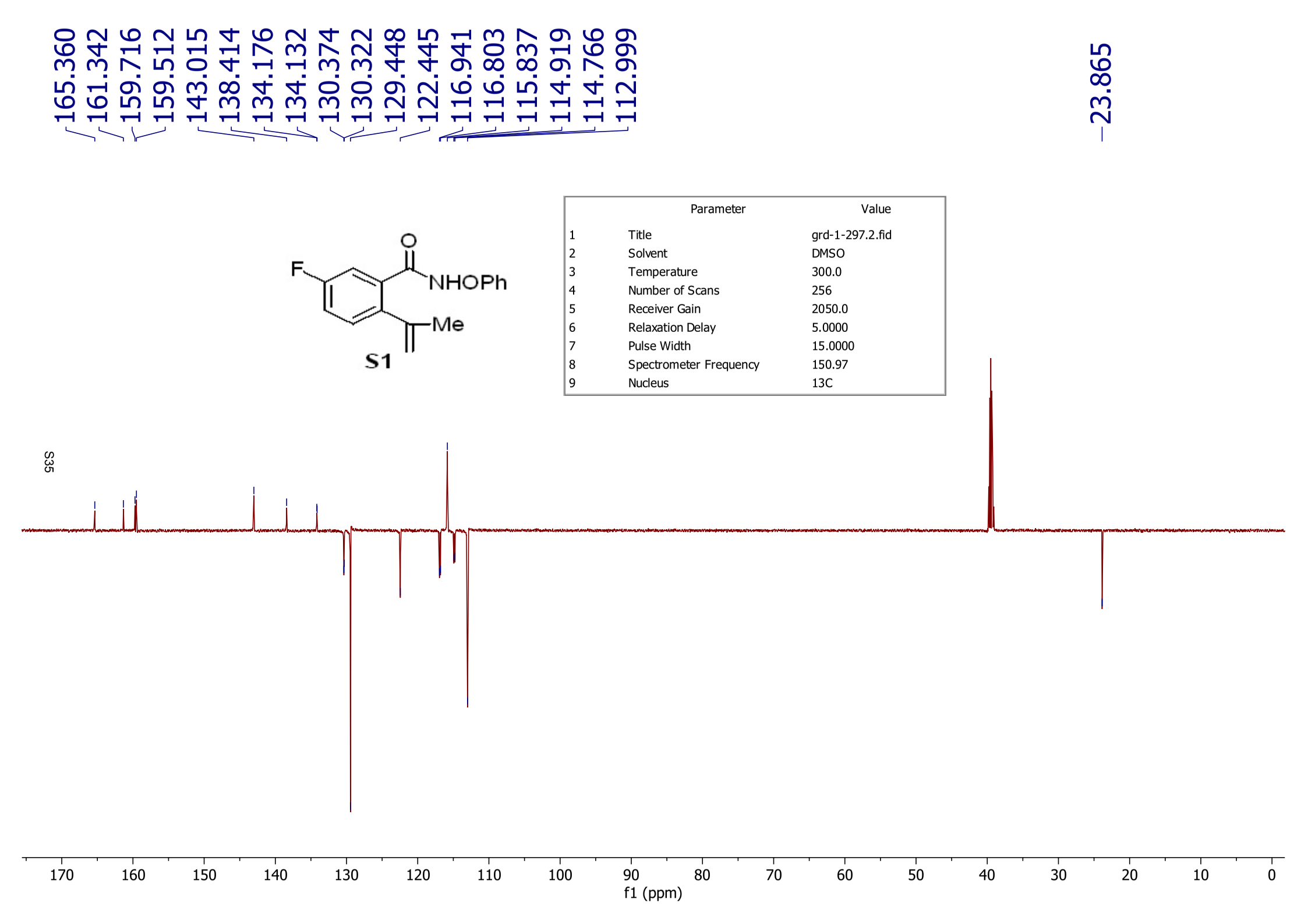
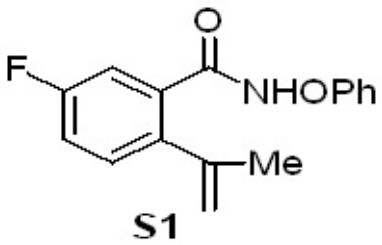
159.623
143.917
141.956
132.374
130.019
129.408
128.069
127.899
127.014
122.324
115.397
112.920
23.810
NHOPh
Me
1
Parameter Value
1 Title grd-1-143.2.fid
2 Solvent DMSO
3 Temperature 300.0
4 Number of Scans 256
5 Receiver Gain 2050.0
6 Relaxation Delay 5.0000
7 Pulse Width 15.0000
8 Spectrometer Frequency 150.97
9 Nucleus 13C
S33
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0


12.210
7.449
7.444
7.430
7.416
7.406
7.376
7.363
7.349
7.336
7.332
7.150
7.136
7.071
7.058
7.046
5.187
4.974
2.083
F
O
NHOPh
Me
S1
Parameter
Value
1 Title grd-1-297.1.fid
2 Solvent DMSO
3 Temperature 300.0
4 Number of Scans 16
5 Receiver Gain 57.0
6 Relaxation Delay 1.0000
7 Pulse Width 10.6100
8 Spectrometer Frequency 600.32
9 Nucleus 1H
0.95
1.97
3.01
1.96
1.01
1.00
1.02
3.00
f1 (ppm)

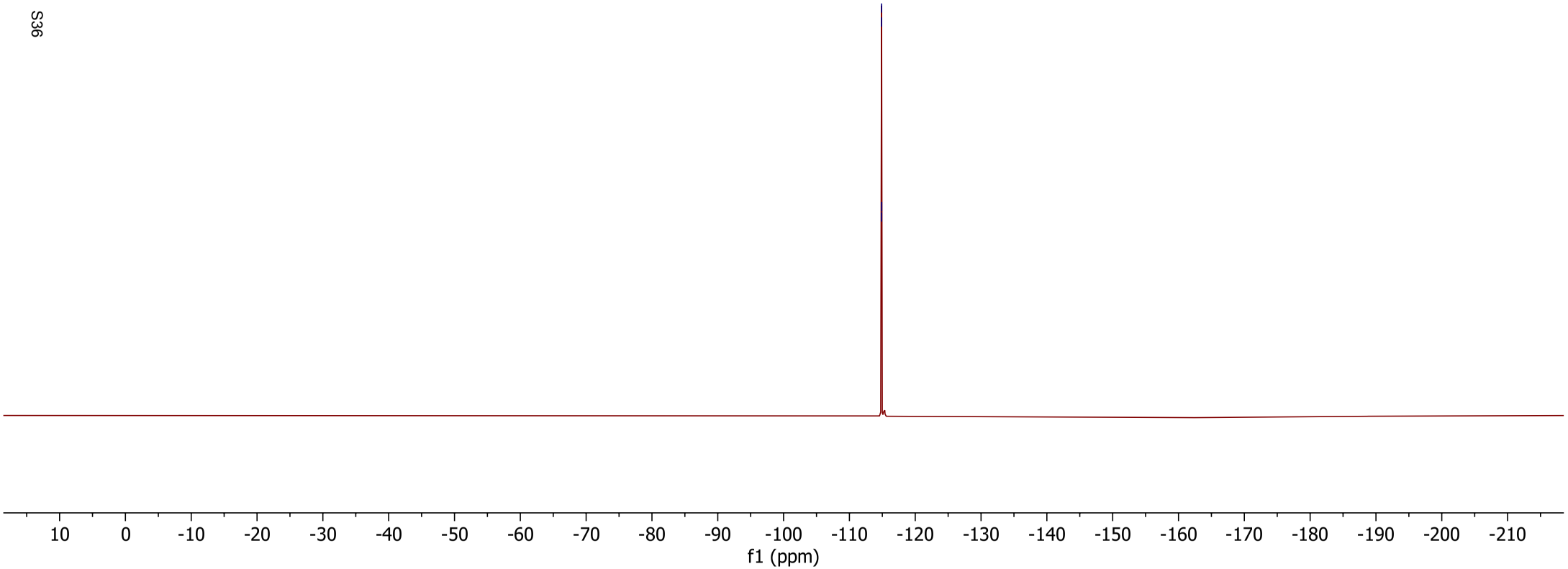

165.360
161.342
159.716
159.512
143.015
138.414
134.176
134.132
130.374
130.322
129.448
122.445
116.941
116.803
115.837
114.919
114.766
112.999
23.865
F
O
NHOPh
Me
S1
Parameter
Value
1 Title grd-1-297.2.fid
2 Solvent DMSO
3 Temperature 300.0
4 Number of Scans 256
5 Receiver Gain 2050.0
6 Relaxation Delay 5.0000
7 Pulse Width 15.0000
8 Spectrometer Frequency 150.97
9 Nucleus 13C
S35
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

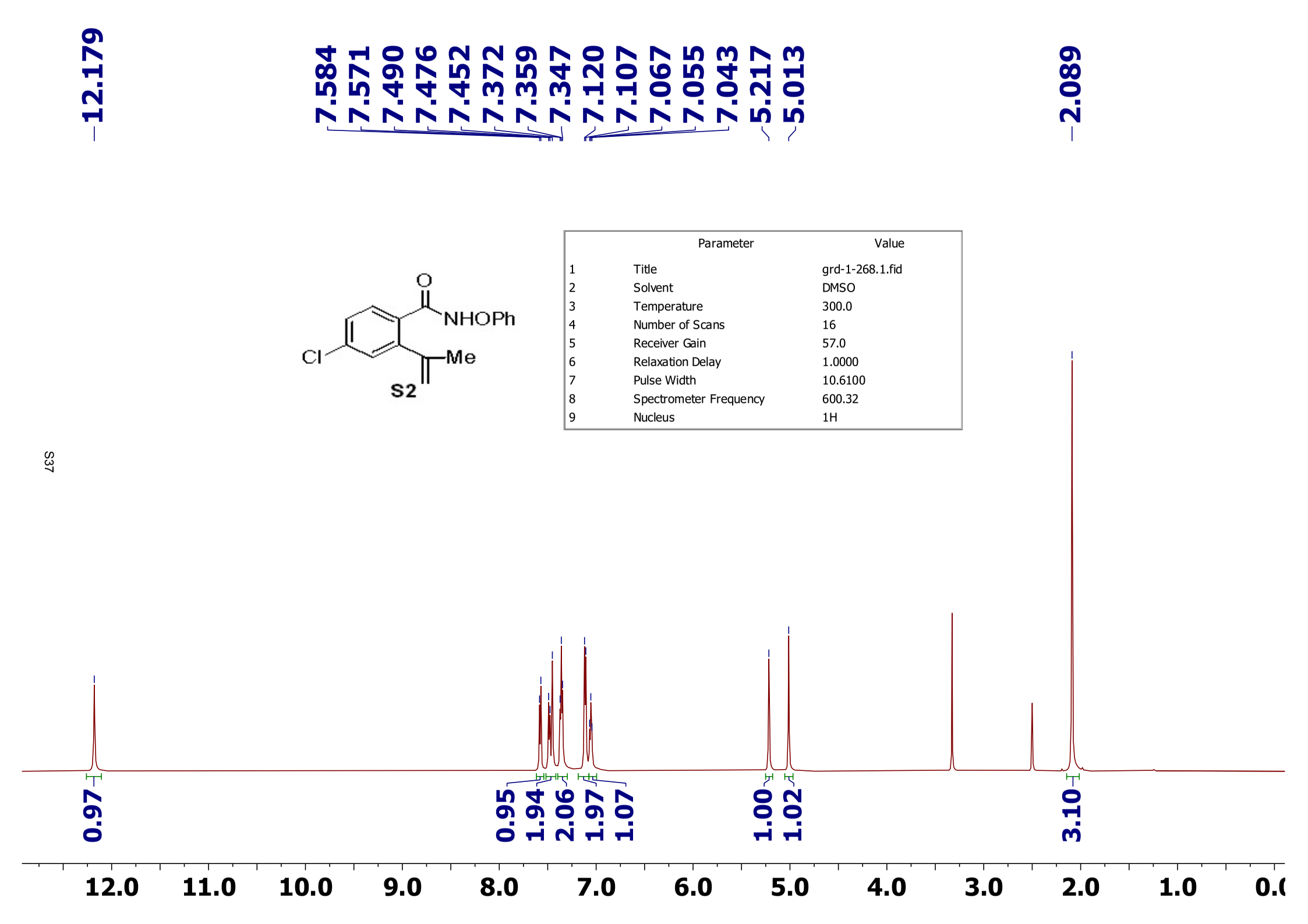
-114.859
-114.873
-114.887
-114.901

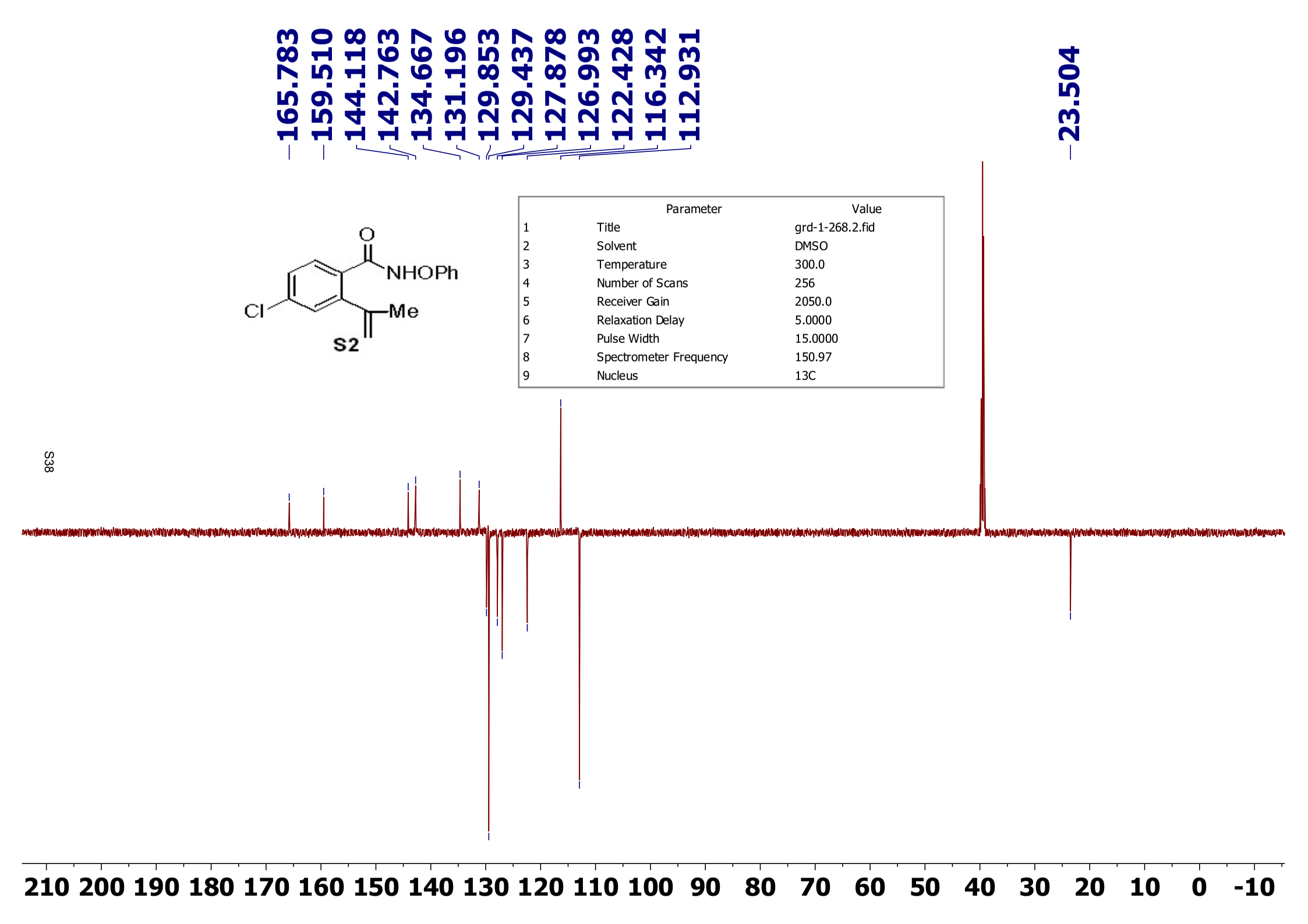


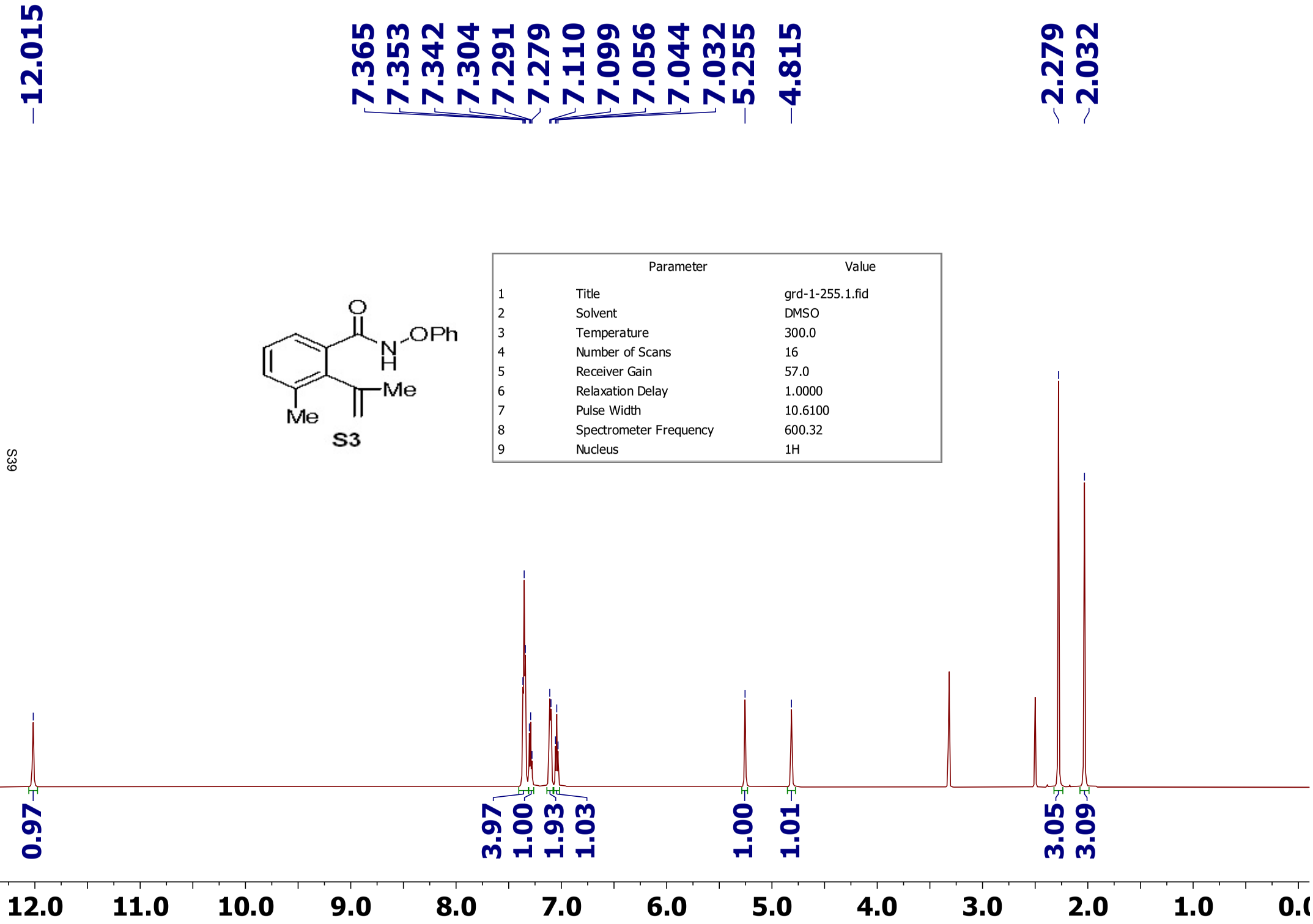
S1

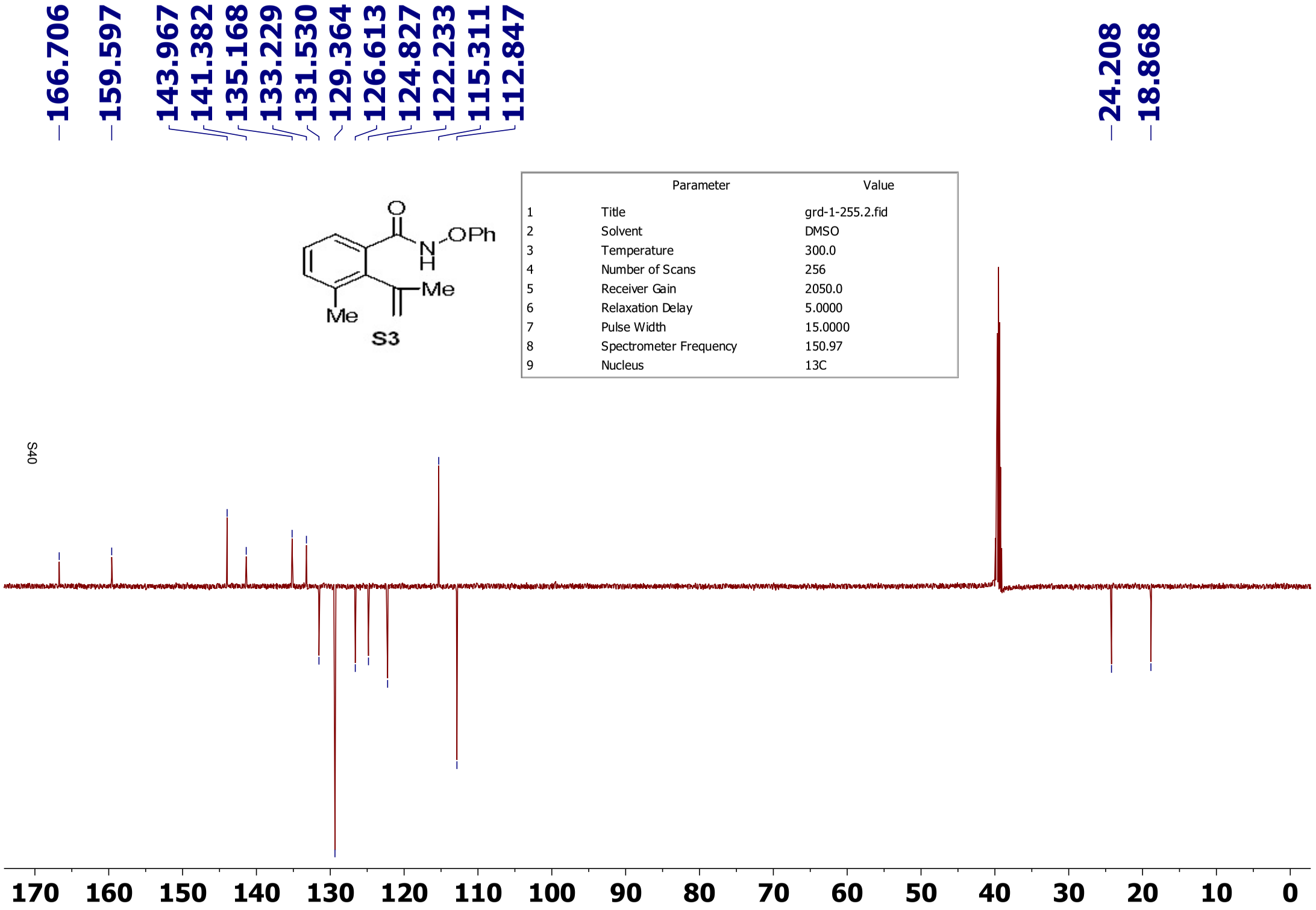
	Parameter	Value
1	Title	grd-1-297.3.fid
2	Solvent	DMSO
3	Temperature	300.0
4	Number of Scans	32
5	Receiver Gain	144.0
6	Relaxation Delay	3.0000
7	Pulse Width	11.4000
8	Spectrometer Frequency	564.81
9	Nucleus	19F

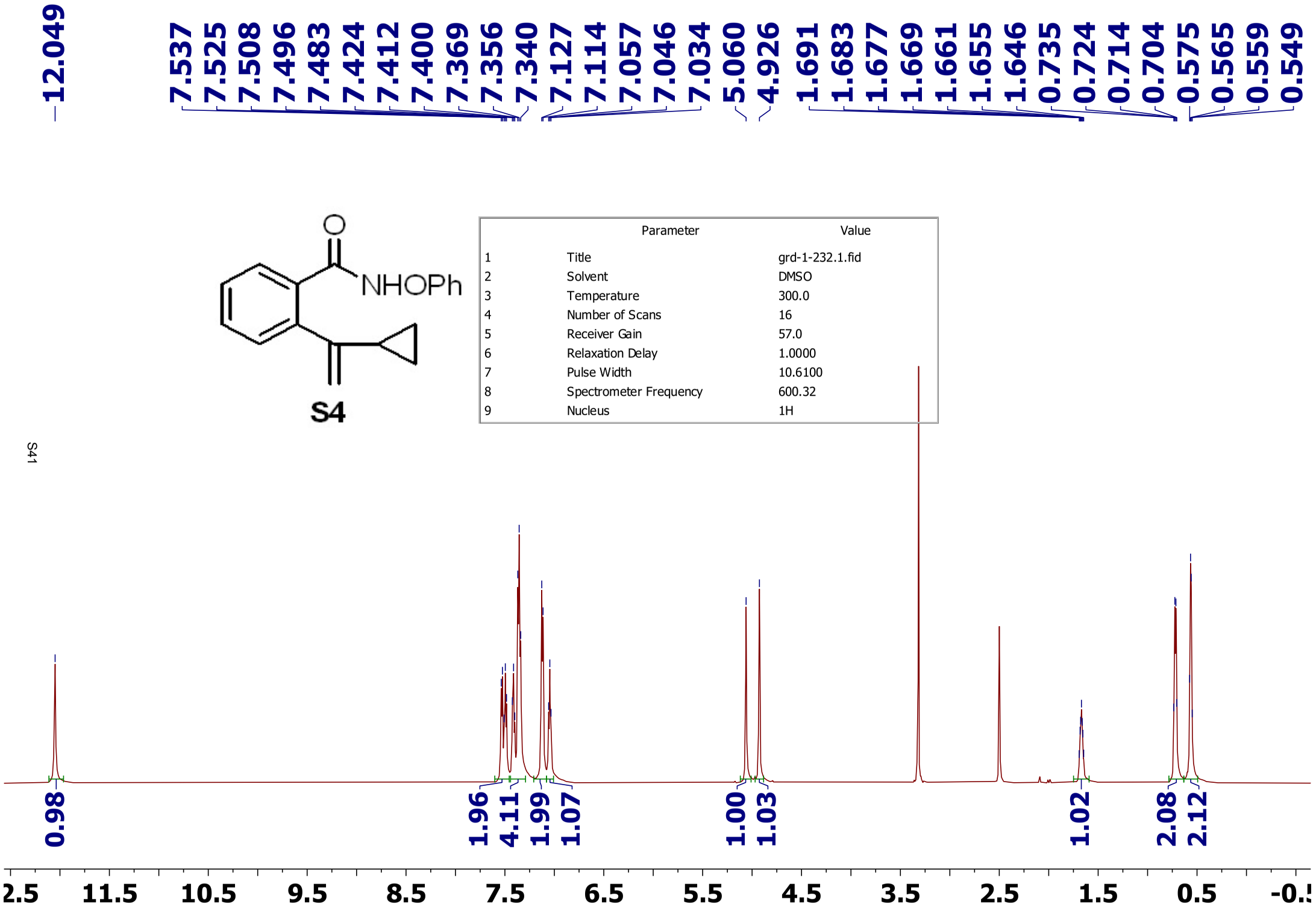


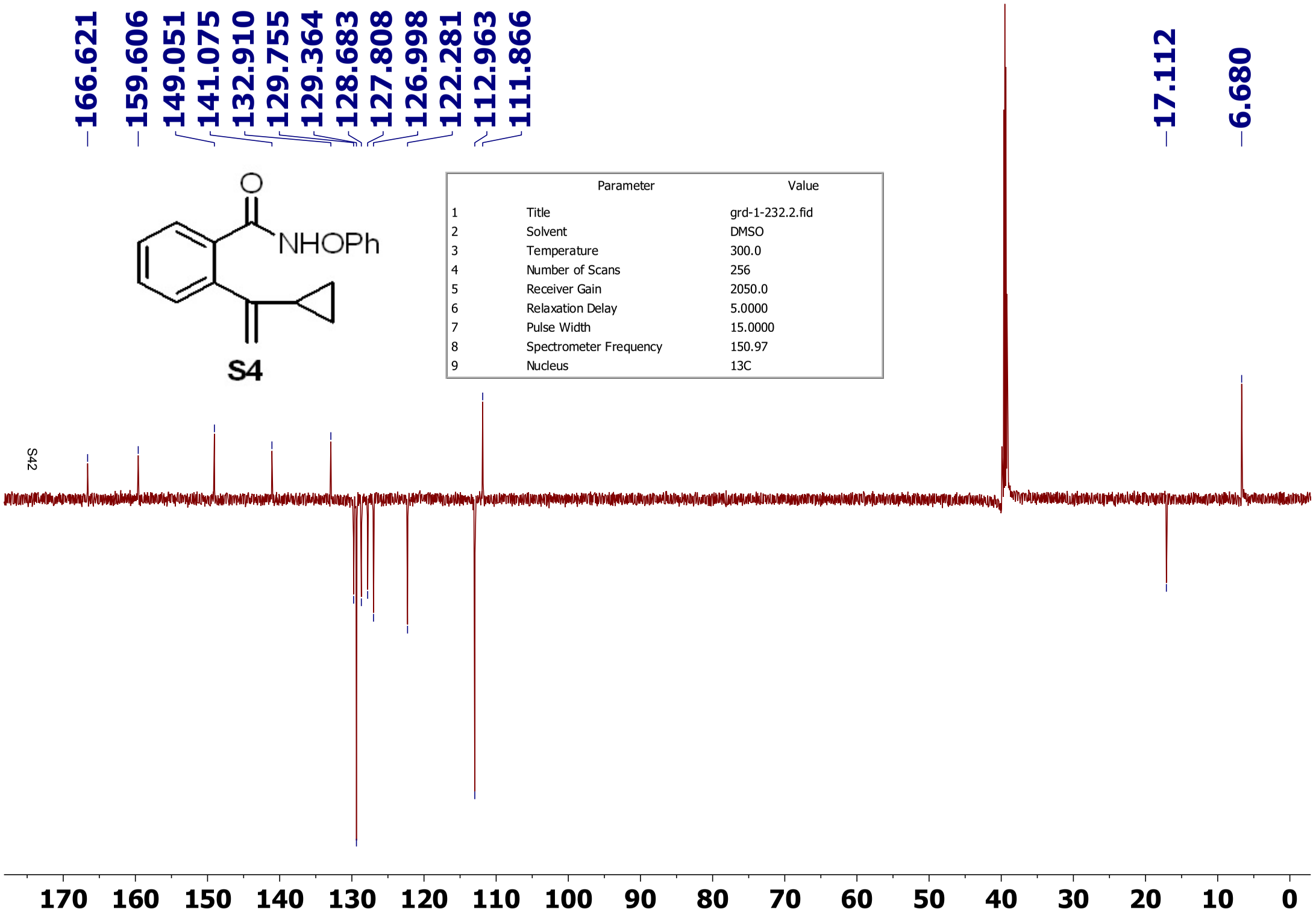

12.179
7.584
7.571
7.490
7.476
7.452
7.372
7.359
7.347
7.120
7.107
7.067
7.055
7.043
5.217
5.013
2.089
O
NHOPh
Cl
Me
S2
Parameter
Value
1 Title grd-1-268.1.fid
2 Solvent DMSO
3 Temperature 300.0
4 Number of Scans 16
5 Receiver Gain 57.0
6 Relaxation Delay 1.0000
7 Pulse Width 10.6100
8 Spectrometer Frequency 600.32
9 Nucleus 1H
S37
0.97
0.95
1.94
2.06
1.97
1.07
1.00
1.02
3.10
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0


165.783
159.510
144.118
142.763
134.667
131.196
129.853
129.437
127.878
126.993
122.428
116.342
112.931
23.504
O
NHOPh
Cl
Me
S2
Parameter Value
1 Title grd-1-268.2.fid
2 Solvent DMSO
3 Temperature 300.0
4 Number of Scans 256
5 Receiver Gain 2050.0
6 Relaxation Delay 5.0000
7 Pulse Width 15.0000
8 Spectrometer Frequency 150.97
9 Nucleus 13C
S38
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10

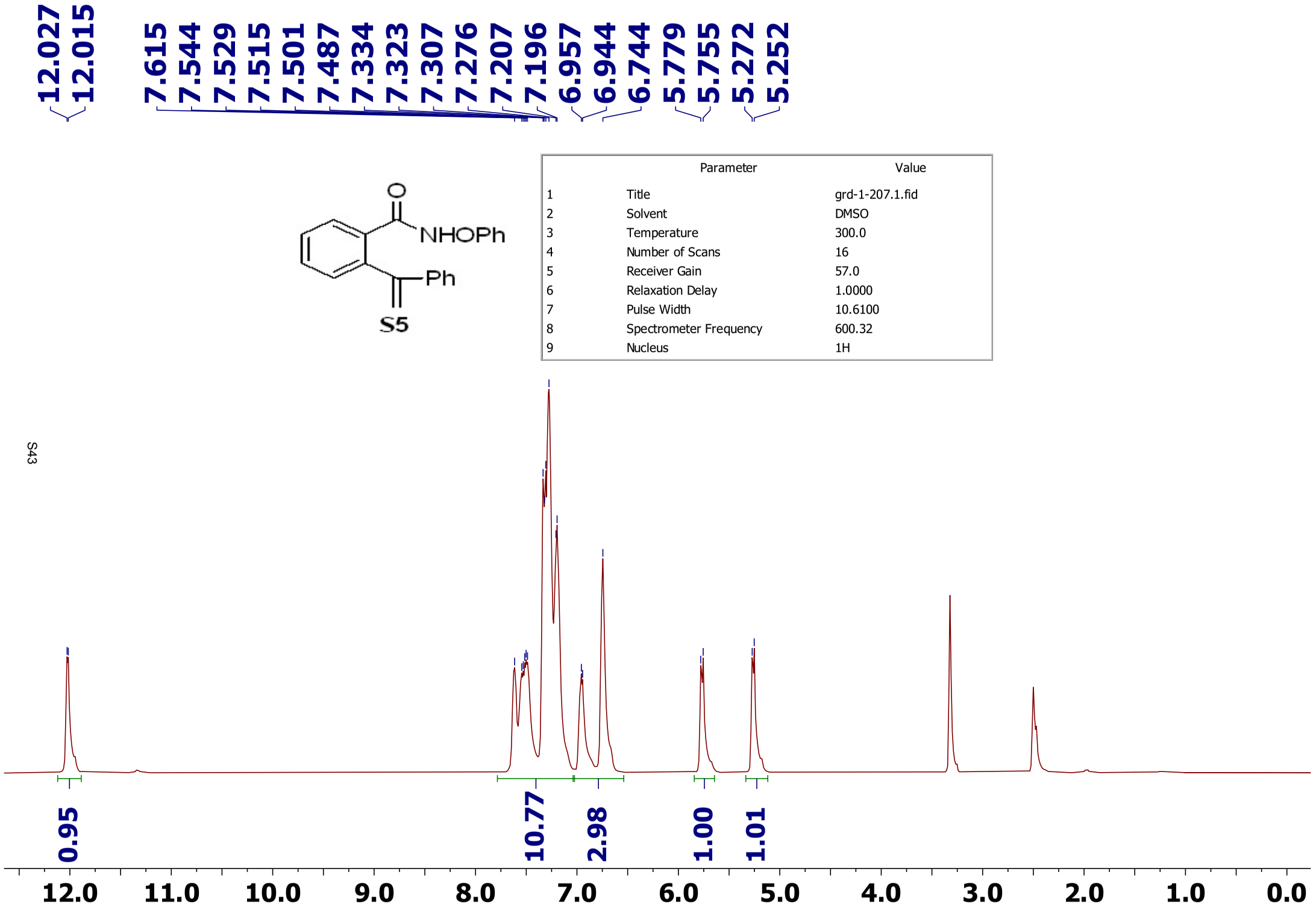

12.015
7.365
7.353
7.342
7.304
7.291
7.279
7.110
7.099
7.056
7.044
7.032
5.255
4.815
2.279
2.032
S3
Parameter Value
1 Title grd-1-255.1.fid
2 Solvent DMSO
3 Temperature 300.0
4 Number of Scans 16
5 Receiver Gain 57.0
6 Relaxation Delay 1.0000
7 Pulse Width 10.6100
8 Spectrometer Frequency 600.32
9 Nucleus 1H
S39
0.97
3.97
1.00
1.93
1.03
1.00
1.01
3.05
3.09
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0


166.706
159.597
143.967
141.382
135.168
133.229
131.530
129.364
126.613
124.827
122.233
115.311
112.847
24.208
18.868
O
N
H
OPh
Me
Me
S3
Parameter
Value
1 Title grd-1-255.2.fid
2 Solvent DMSO
3 Temperature 300.0
4 Number of Scans 256
5 Receiver Gain 2050.0
6 Relaxation Delay 5.0000
7 Pulse Width 15.0000
8 Spectrometer Frequency 150.97
9 Nucleus 13C
S40
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0


12.049
7.537
7.525
7.508
7.496
7.483
7.424
7.412
7.400
7.369
7.356
7.340
7.127
7.114
7.057
7.046
7.034
5.060
4.926
1.691
1.683
1.677
1.669
1.661
1.655
1.646
0.735
0.724
0.714
0.704
0.575
0.565
0.559
0.549
NHOPh
S4
Parameter Value
1 Title grd-1-232.1.fid
2 Solvent DMSO
3 Temperature 300.0
4 Number of Scans 16
5 Receiver Gain 57.0
6 Relaxation Delay 1.0000
7 Pulse Width 10.6100
8 Spectrometer Frequency 600.32
9 Nucleus 1H
S41
0.98
1.96
4.11
1.99
1.07
1.00
1.03
1.02
2.08
2.12
2.5
11.5
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5



	Parameter	Value
1	Title	grd-1-232.2.fid
2	Solvent	DMSO
3	Temperature	300.0
4	Number of Scans	256
5	Receiver Gain	2050.0
6	Relaxation Delay	5.0000
7	Pulse Width	15.0000
8	Spectrometer Frequency	150.97
9	Nucleus	13C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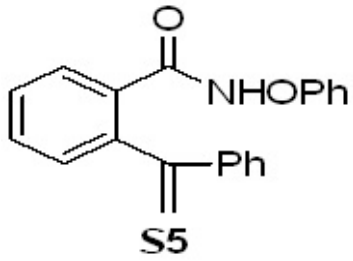
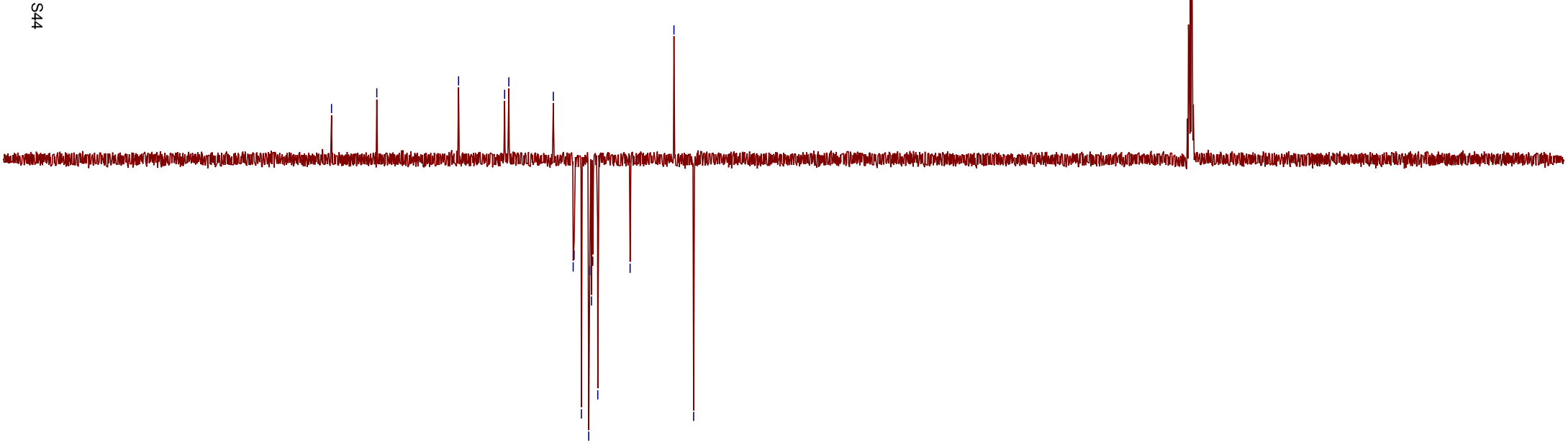
12.015
7.615
7.544
7.529
7.515
7.501
7.487
7.334
7.323
7.307
7.276
7.207
7.196
6.957
6.944
6.744
5.779
5.755
5.272
5.252
NHOPh
Ph
S5
Parameter
Value
1 Title grd-1-207.1.fid
2 Solvent DMSO
3 Temperature 300.0
4 Number of Scans 16
5 Receiver Gain 57.0
6 Relaxation Delay 1.0000
7 Pulse Width 10.6100
8 Spectrometer Frequency 600.32
9 Nucleus 1H
S43
0.95
10.77
2.98
1.00
1.01
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0

166.081
159.408
147.355
140.570
139.947
133.390
130.463
130.330
129.253
128.177
128.020
127.767
127.575
126.840
122.074
115.612
112.713

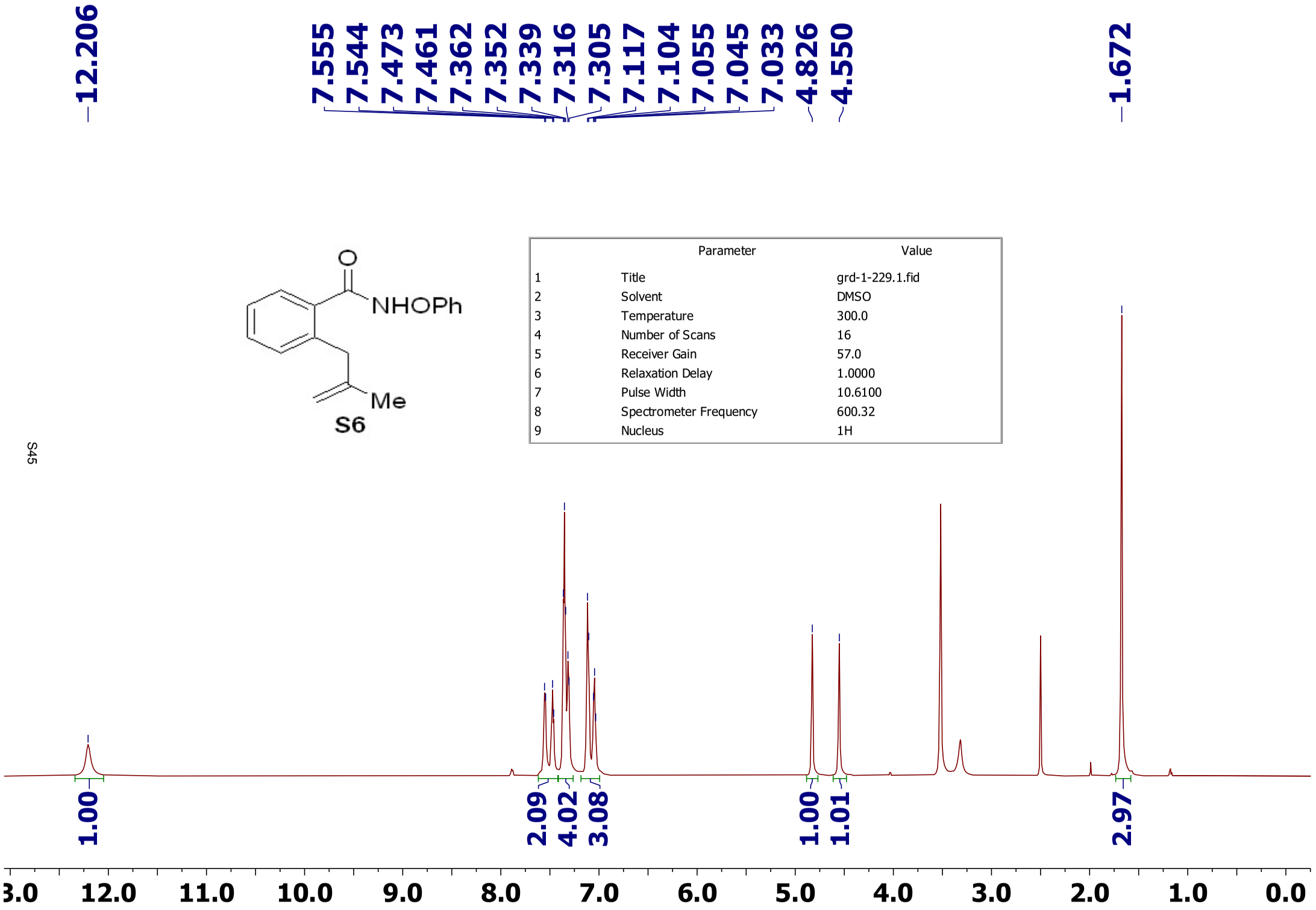


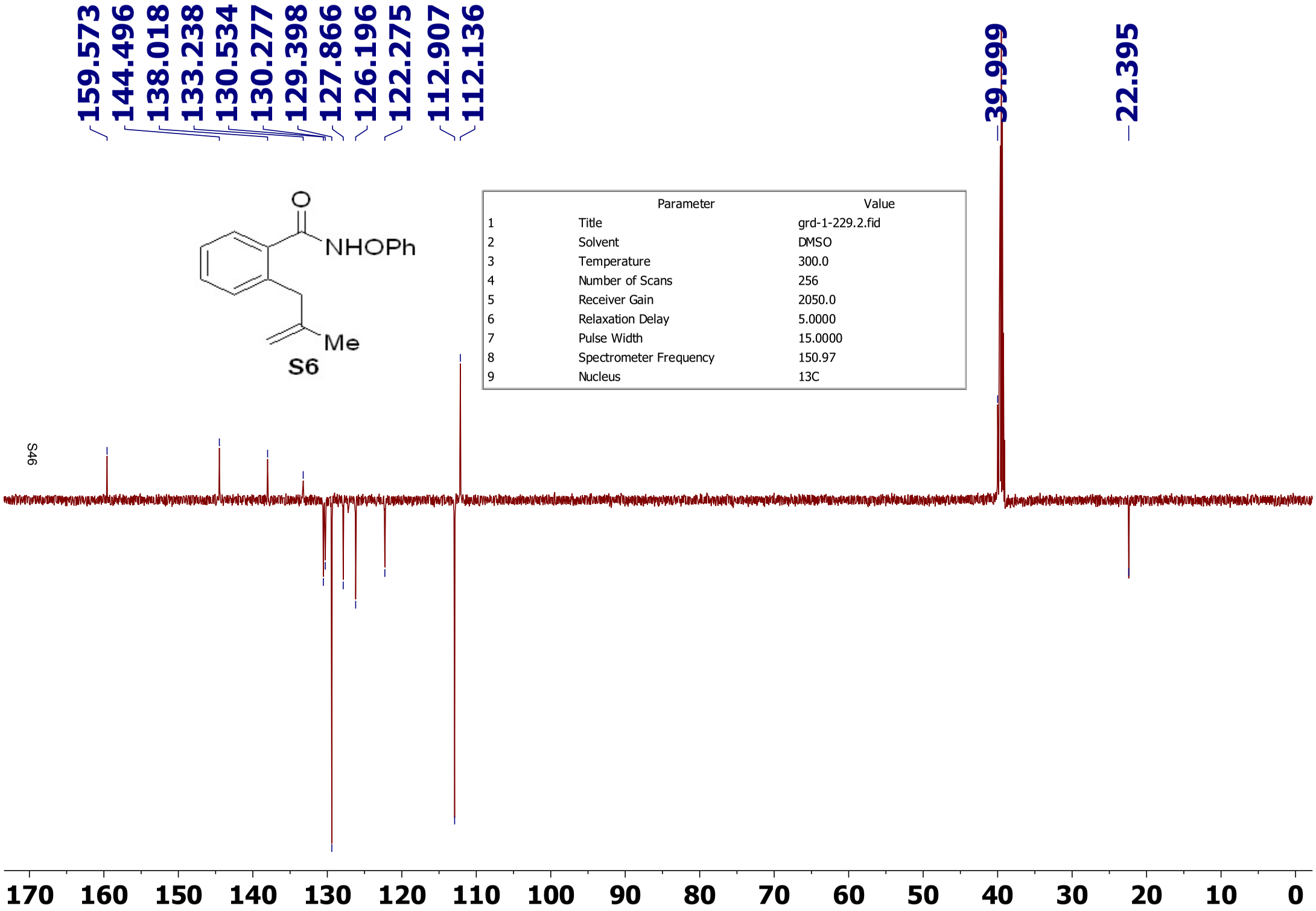
S5

	Parameter	Value
1	Title	grd-1-207.2.fid
2	Solvent	DMSO
3	Temperature	300.0
4	Number of Scans	256
5	Receiver Gain	2050.0
6	Relaxation Delay	5.0000
7	Pulse Width	15.0000
8	Spectrometer Frequency	150.97
9	Nucleus	13C

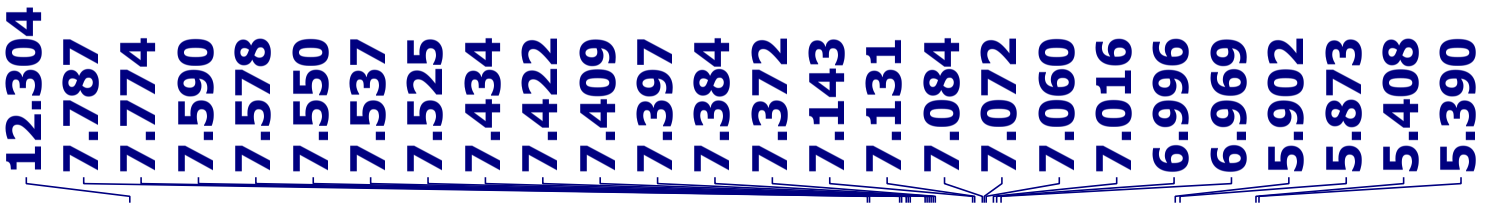


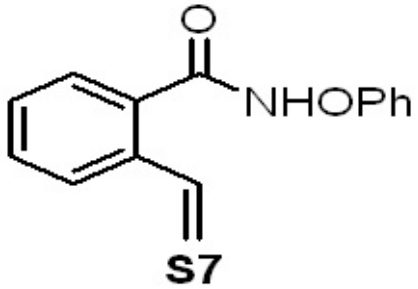
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10


12.206
7.555
7.544
7.473
7.461
7.362
7.352
7.339
7.316
7.305
7.117
7.104
7.055
7.045
7.033
4.826
4.550
1.672
O
NHOPh
Me
S6
Parameter
Value
1 Title grd-1-229.1.fid
2 Solvent DMSO
3 Temperature 300.0
4 Number of Scans 16
5 Receiver Gain 57.0
6 Relaxation Delay 1.0000
7 Pulse Width 10.6100
8 Spectrometer Frequency 600.32
9 Nucleus 1H
S45
1.00
2.09
4.02
3.08
1.00
1.01
2.97
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
0.0

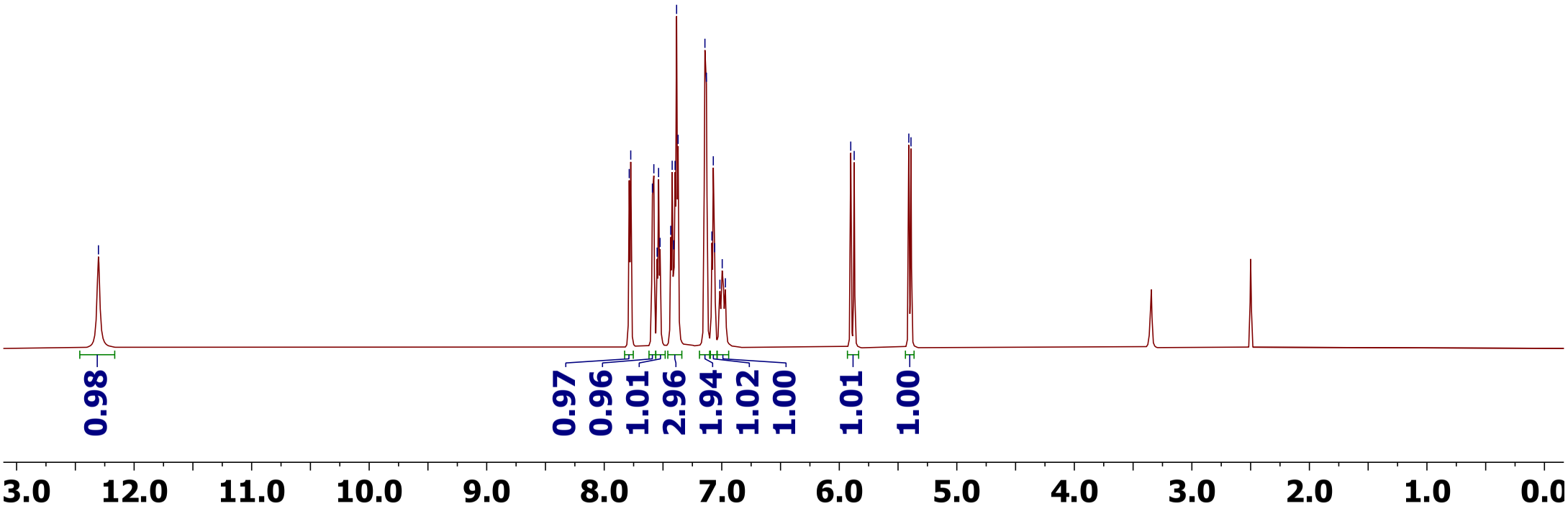


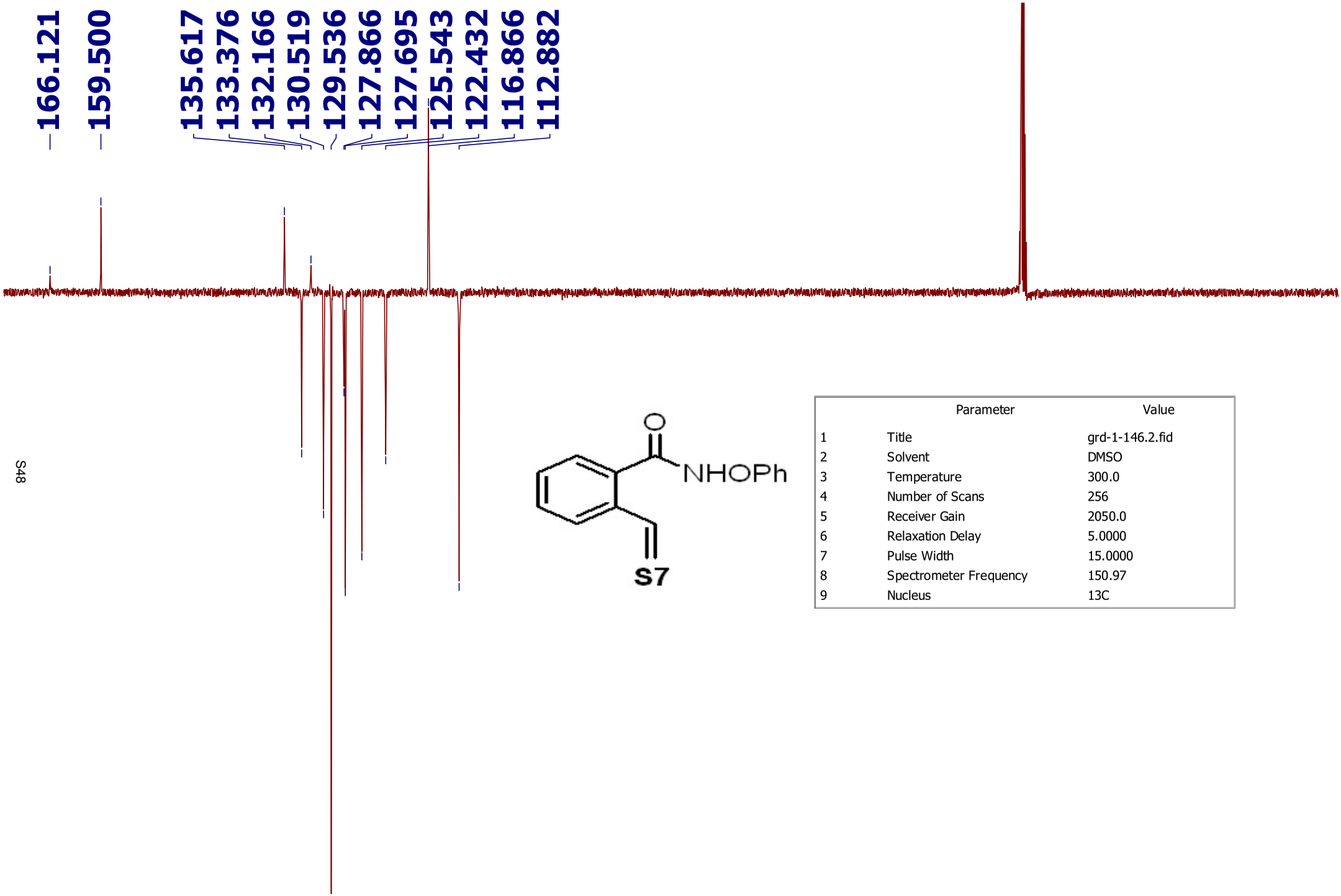
	Parameter	Value
1	Title	grd-1-229.2.fid
2	Solvent	DMSO
3	Temperature	300.0
4	Number of Scans	256
5	Receiver Gain	2050.0
6	Relaxation Delay	5.0000
7	Pulse Width	15.0000
8	Spectrometer Frequency	150.97
9	Nucleus	13C



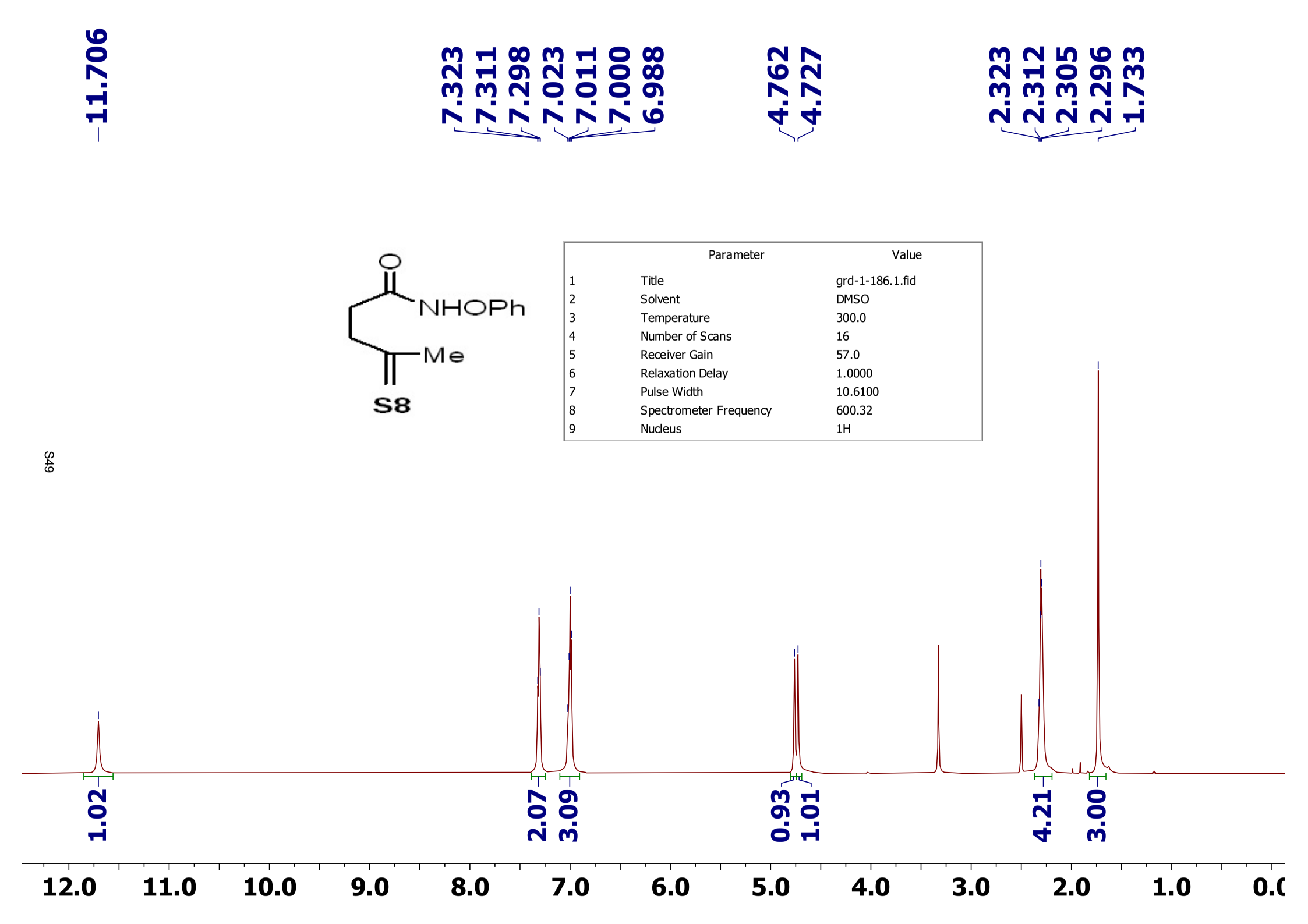


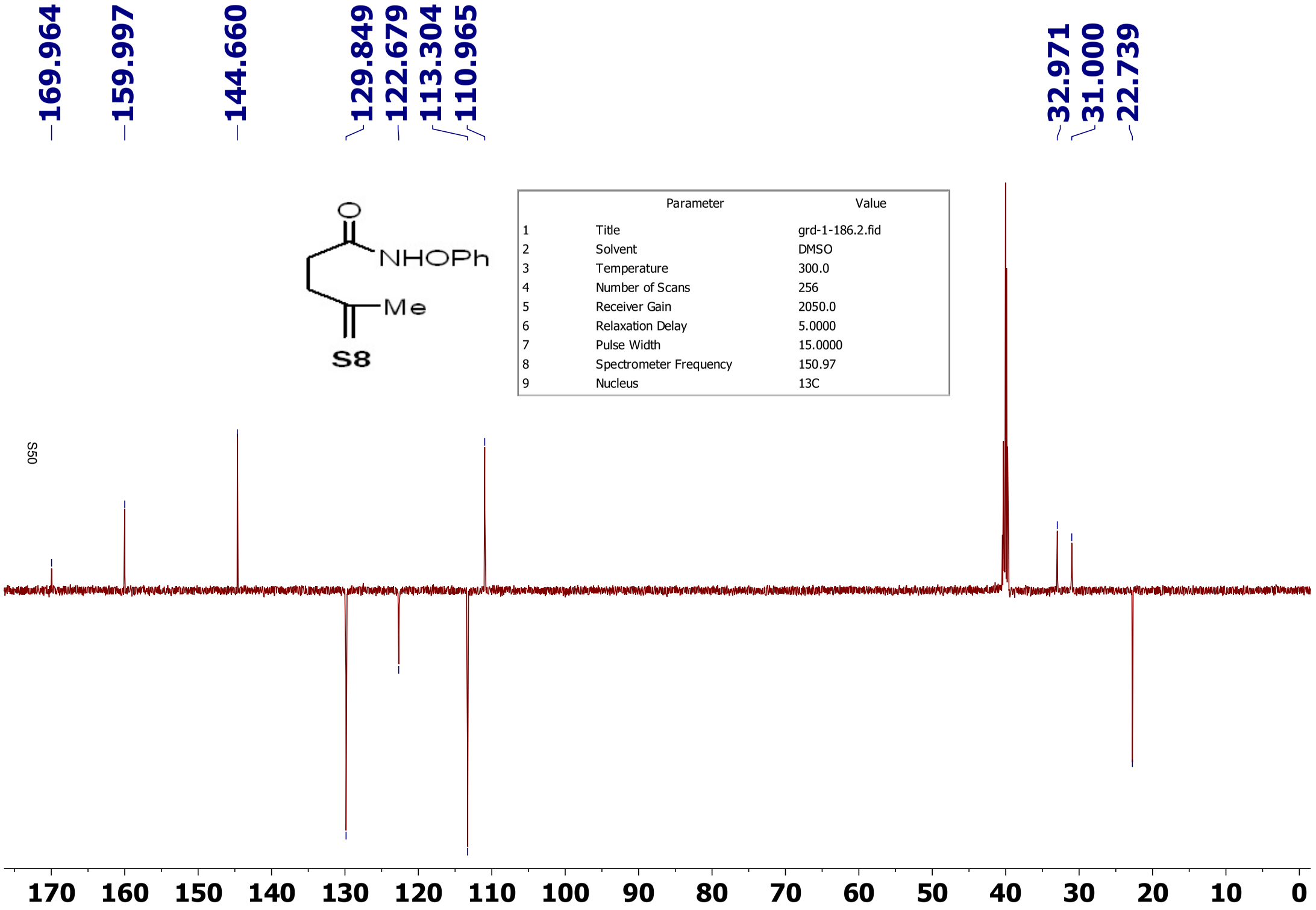
	Parameter	Value
1	Title	grd-1-146.1.fid
2	Solvent	DMSO
3	Temperature	300.0
4	Number of Scans	16
5	Receiver Gain	57.0
6	Relaxation Delay	1.0000
7	Pulse Width	10.6100
8	Spectrometer Frequency	600.32
9	Nucleus	1H



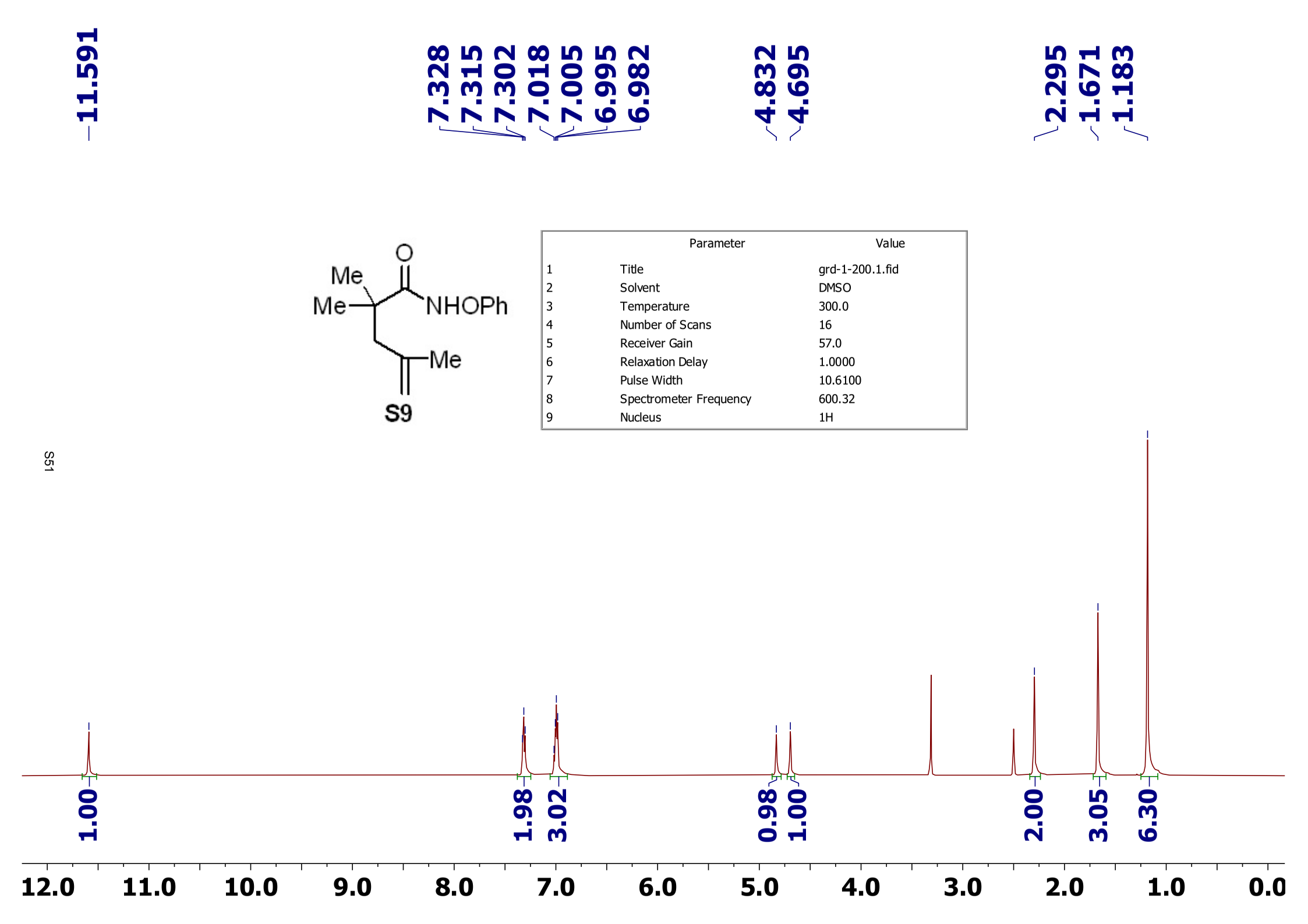


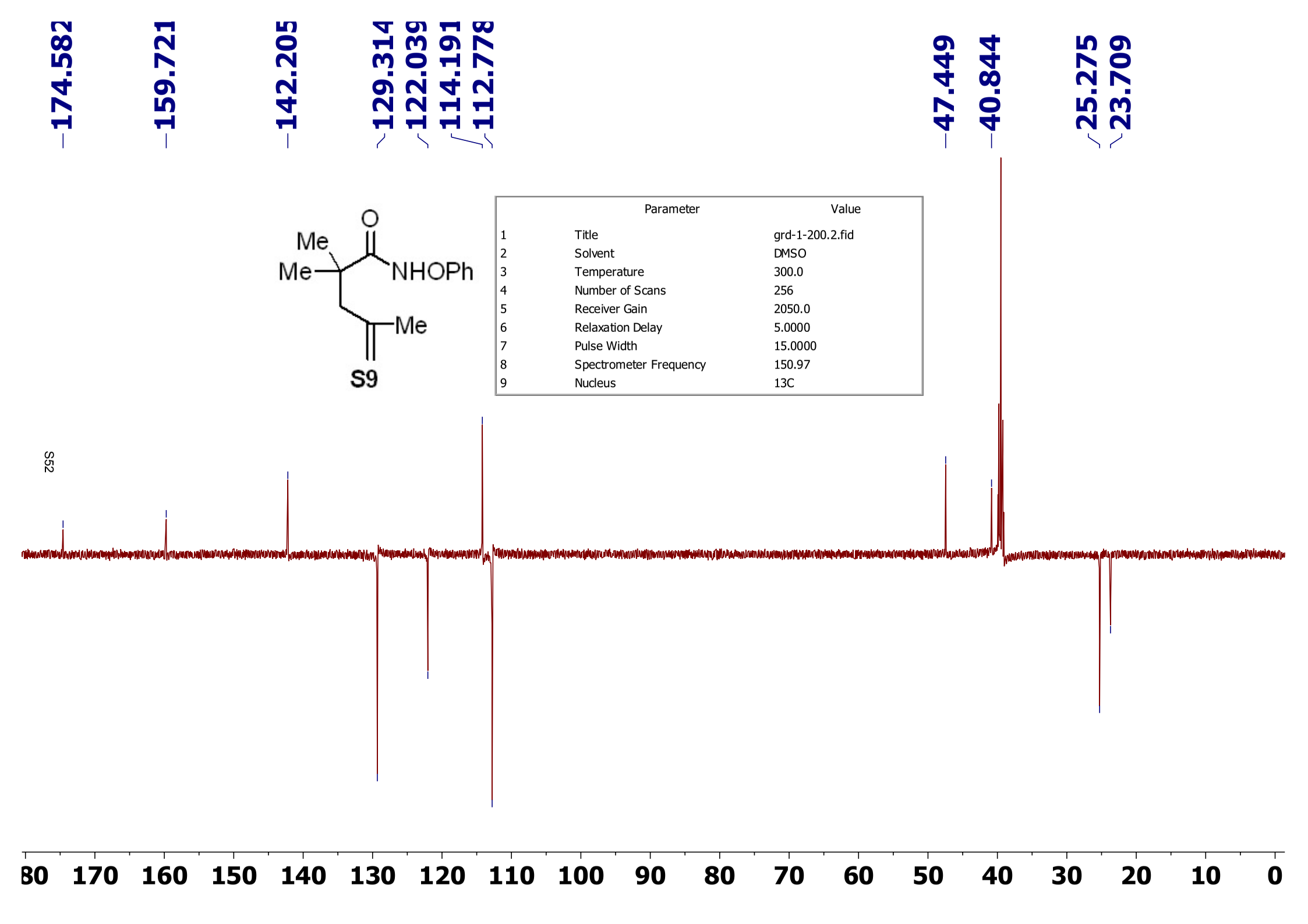
	Parameter	Value
1	Title	grd-1-146.2.fid
2	Solvent	DMSO
3	Temperature	300.0
4	Number of Scans	256
5	Receiver Gain	2050.0
6	Relaxation Delay	5.0000
7	Pulse Width	15.0000
8	Spectrometer Frequency	150.97
9	Nucleus	13C

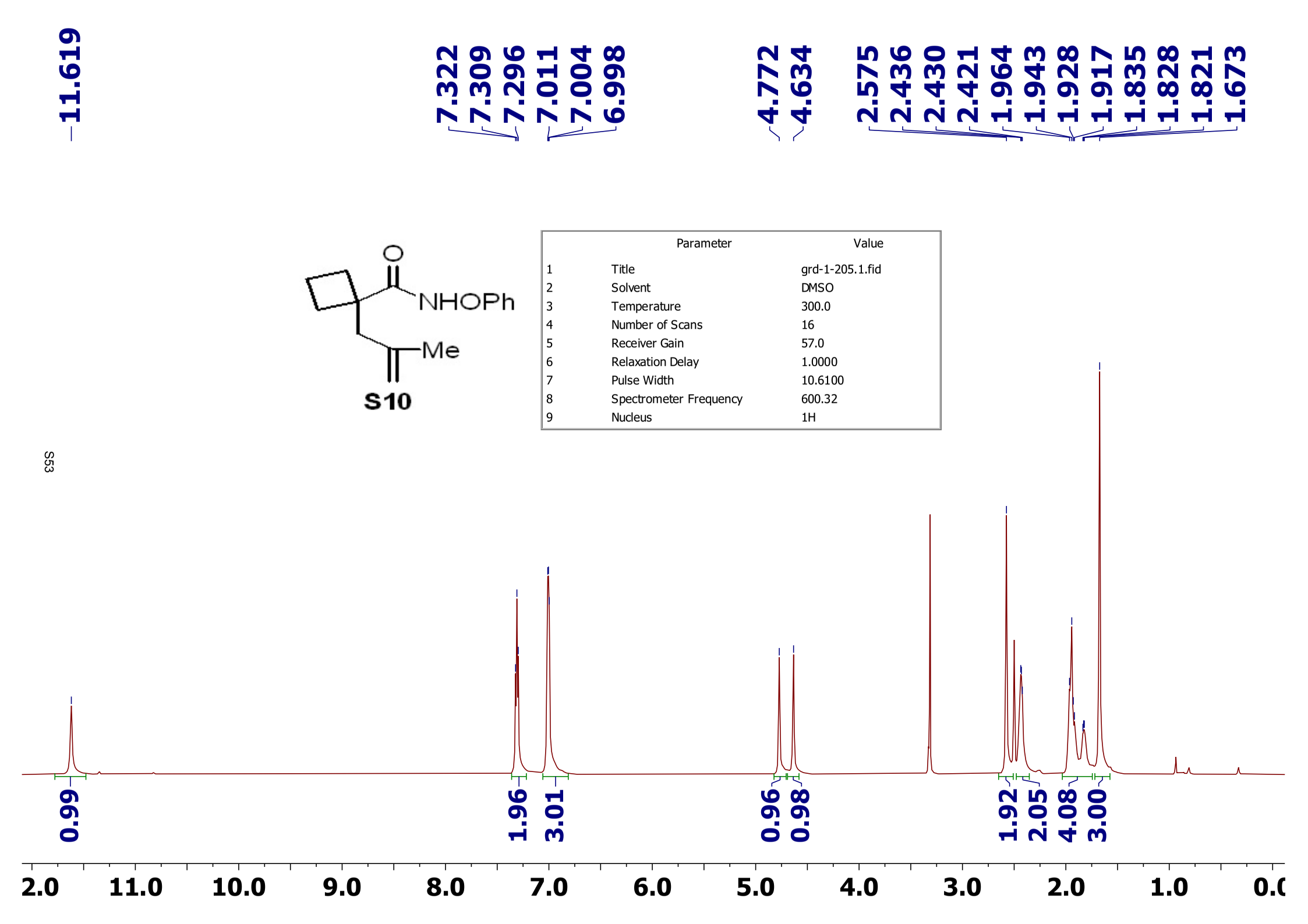

11.706
7.323
7.311
7.298
7.023
7.011
7.000
6.988
4.762
4.727
2.323
2.312
2.305
2.296
1.733
NHOPh
Me
S8
Parameter
Value
1 Title grd-1-186.1.fid
2 Solvent DMSO
3 Temperature 300.0
4 Number of Scans 16
5 Receiver Gain 57.0
6 Relaxation Delay 1.0000
7 Pulse Width 10.6100
8 Spectrometer Frequency 600.32
9 Nucleus 1H
1.02
2.07
3.09
0.93
1.01
4.21
3.00
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0

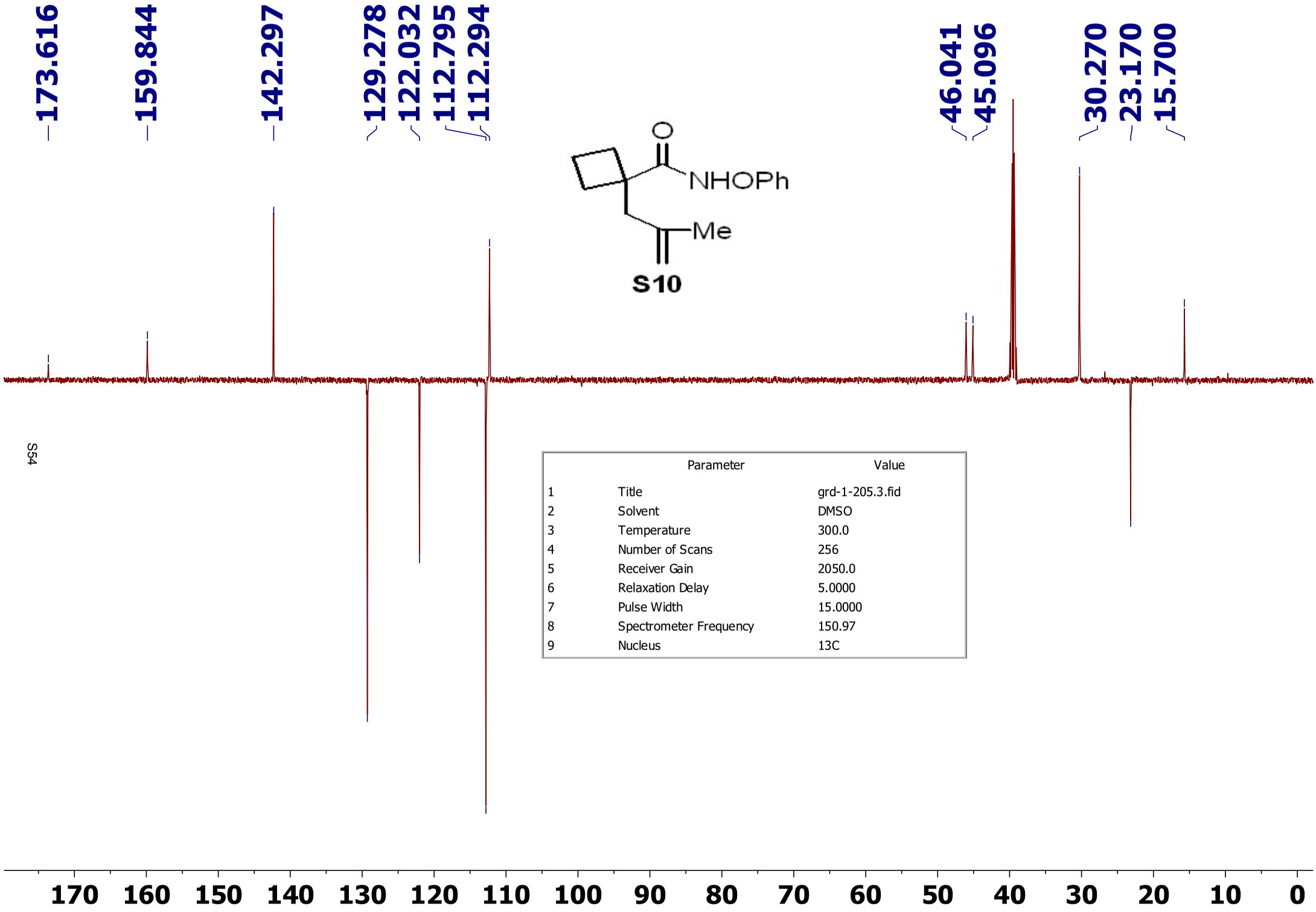


	Parameter	Value
1	Title	grd-1-186.2.fid
2	Solvent	DMSO
3	Temperature	300.0
4	Number of Scans	256
5	Receiver Gain	2050.0
6	Relaxation Delay	5.0000
7	Pulse Width	15.0000
8	Spectrometer Frequency	150.97
9	Nucleus	13C

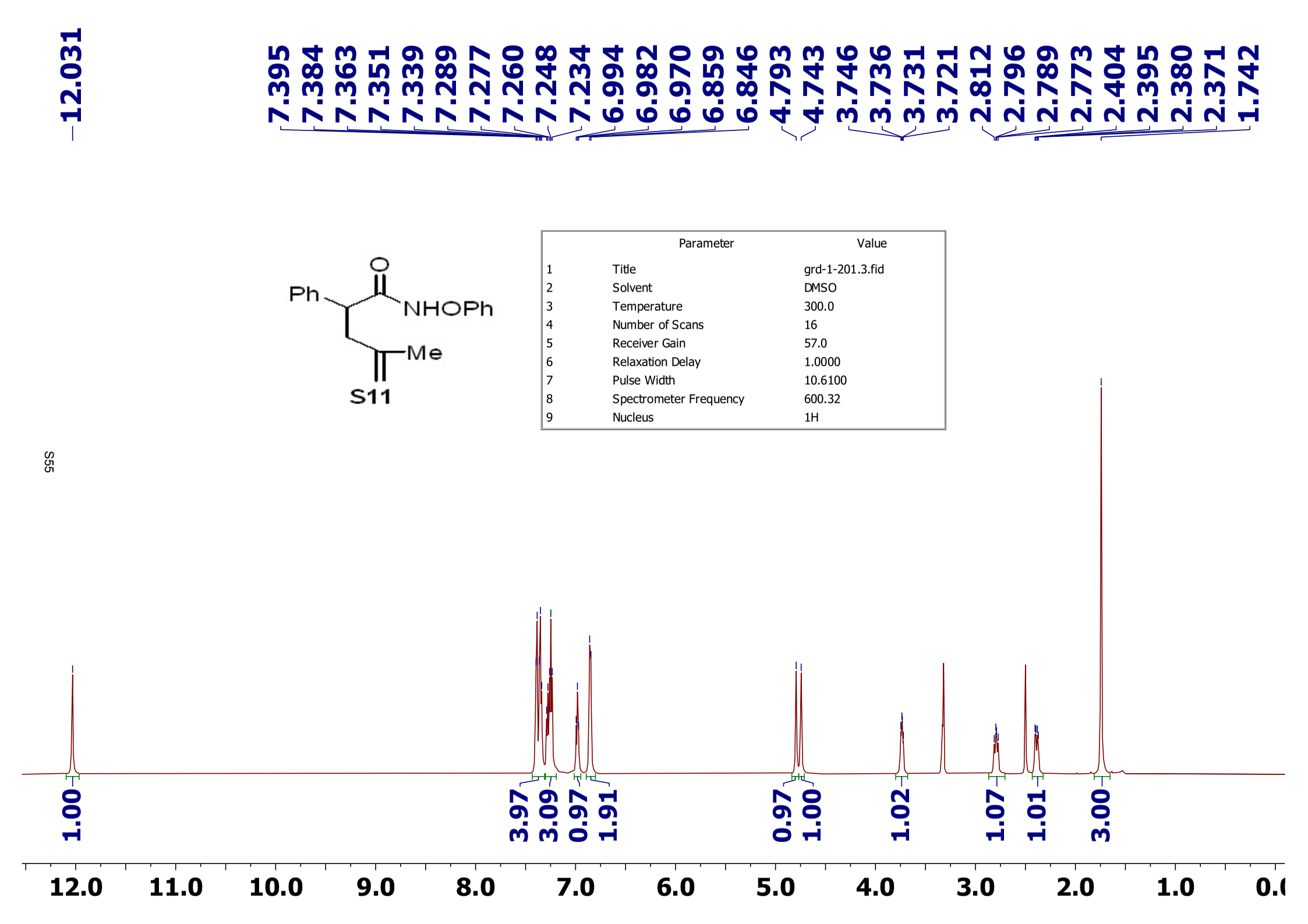

11.591
7.328
7.315
7.302
7.018
7.005
6.995
6.982
4.832
4.695
2.295
1.671
1.183
Me
Me
O
NHOPh
Me
S9
Parameter
Value
1 Title grd-1-200.1.fid
2 Solvent DMSO
3 Temperature 300.0
4 Number of Scans 16
5 Receiver Gain 57.0
6 Relaxation Delay 1.0000
7 Pulse Width 10.6100
8 Spectrometer Frequency 600.32
9 Nucleus 1H
S51
1.00
1.98
3.02
0.98
1.00
2.00
3.05
6.30
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0


174.582
159.721
142.205
129.314
122.039
114.191
112.778
47.449
40.844
25.275
23.709
O
Me
Me
NHOPh
Me
S9
Parameter Value
1 Title grd-1-200.2.fid
2 Solvent DMSO
3 Temperature 300.0
4 Number of Scans 256
5 Receiver Gain 2050.0
6 Relaxation Delay 5.0000
7 Pulse Width 15.0000
8 Spectrometer Frequency 150.97
9 Nucleus 13C
S52
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0


11.619
7.322
7.309
7.296
7.011
7.004
6.998
4.772
4.634
2.575
2.436
2.430
2.421
1.964
1.943
1.928
1.917
1.835
1.828
1.821
1.673
NHOPh
Me
S10
Parameter
Value
1 Title grd-1-205.1.fid
2 Solvent DMSO
3 Temperature 300.0
4 Number of Scans 16
5 Receiver Gain 57.0
6 Relaxation Delay 1.0000
7 Pulse Width 10.6100
8 Spectrometer Frequency 600.32
9 Nucleus 1H
S53
0.99
1.96
3.01
0.96
0.98
1.92
2.05
4.08
3.00
2.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0



	Parameter	Value
1	Title	grd-1-205.3.fid
2	Solvent	DMSO
3	Temperature	300.0
4	Number of Scans	256
5	Receiver Gain	2050.0
6	Relaxation Delay	5.0000
7	Pulse Width	15.0000
8	Spectrometer Frequency	150.97
9	Nucleus	13C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7.384
7.363
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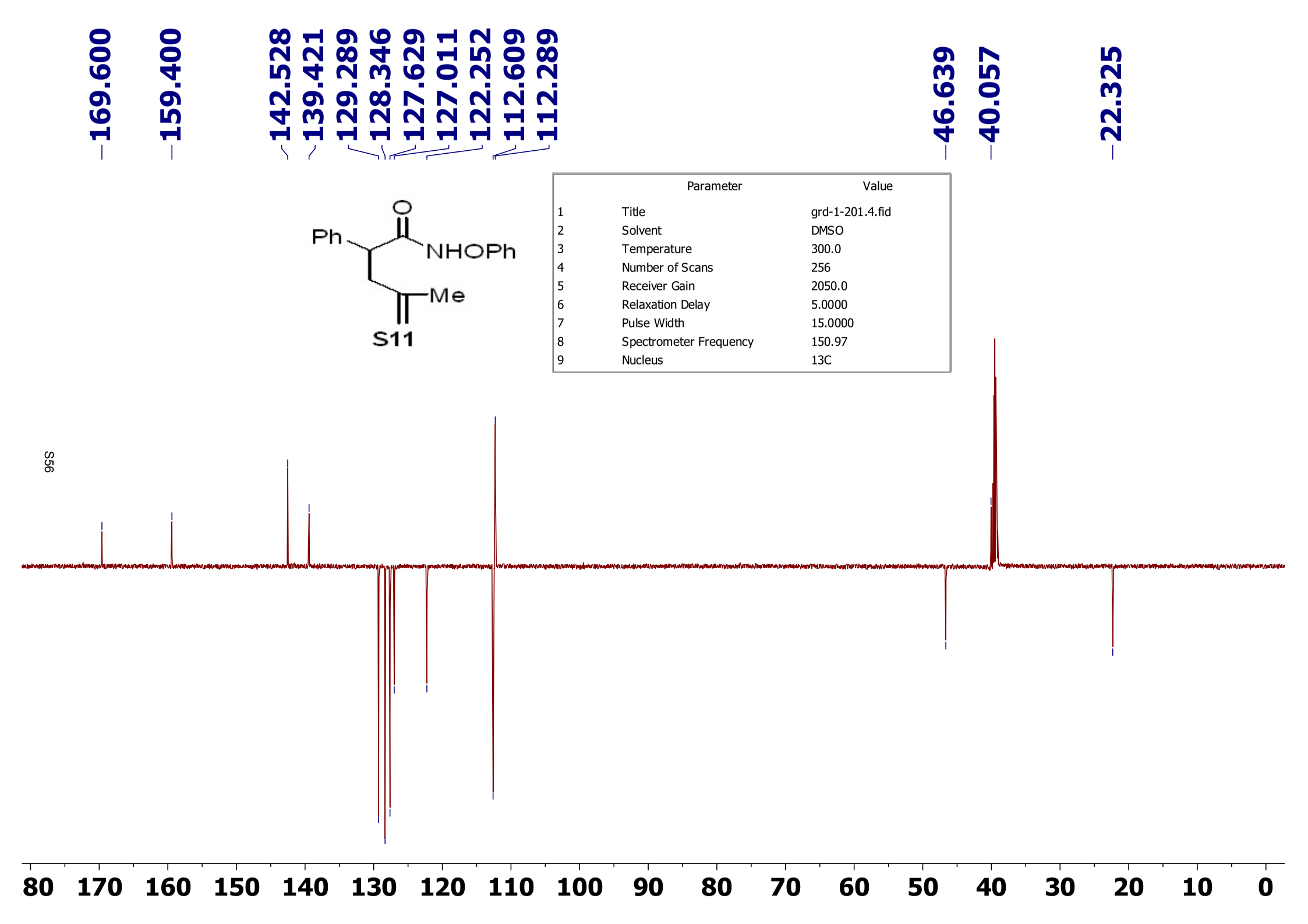
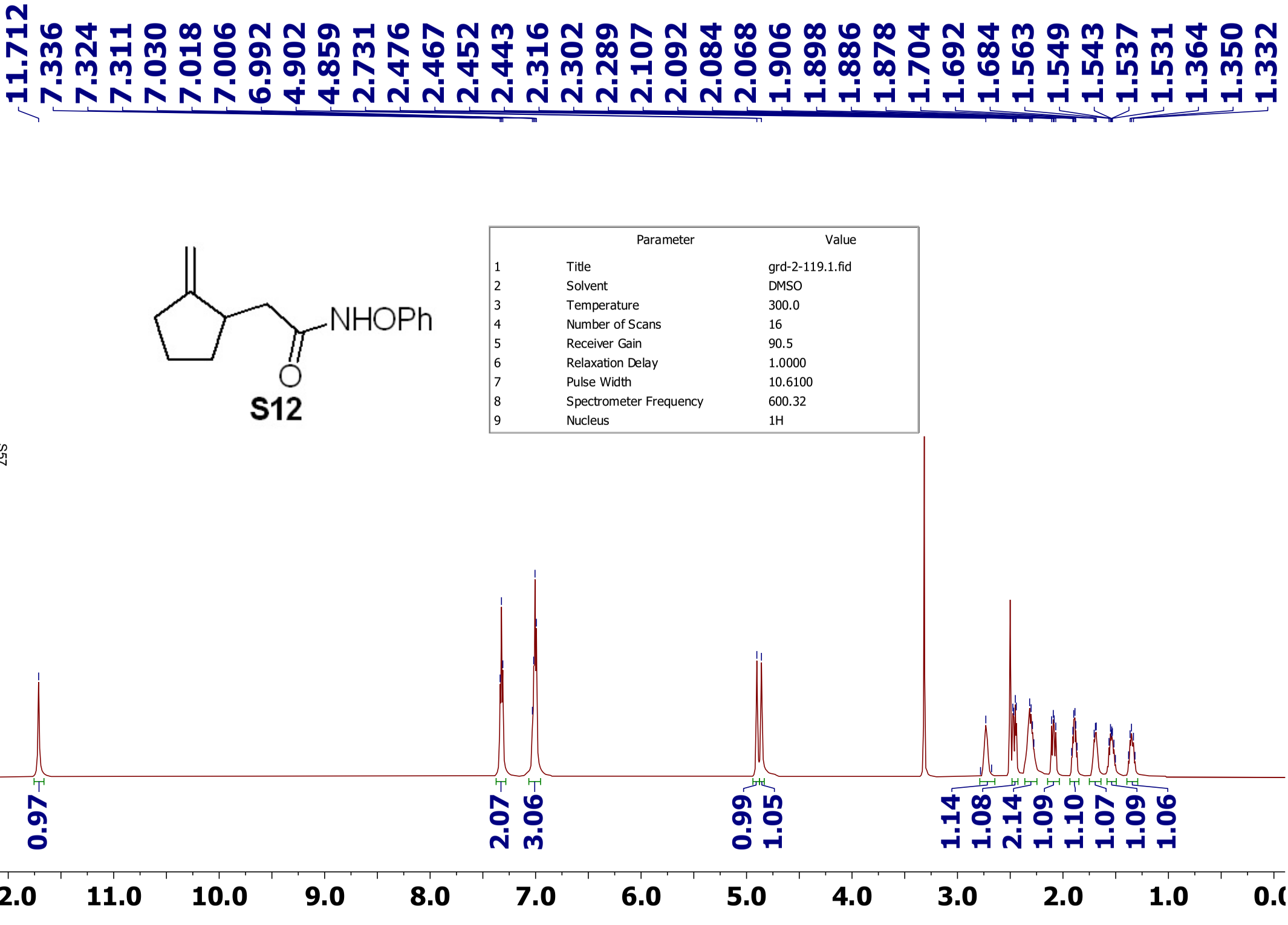
7.339
7.289
7.277
7.260
7.248
7.234
6.994
6.982
6.970
6.859
6.846
4.793
4.743
3.746
3.736
3.731
3.721
2.812
2.796
2.789
2.773
2.404
2.395
2.380
2.371
1.742
Ph
NHOPh
Me
S11
Parameter Value
1 Title grd-1-201.3.fid
2 Solvent DMSO
3 Temperature 300.0
4 Number of Scans 16
5 Receiver Gain 57.0
6 Relaxation Delay 1.0000
7 Pulse Width 10.6100
8 Spectrometer Frequency 600.32
9 Nucleus 1H
S55
1.00
3.97
3.09
0.97
1.91
0.97
1.00
1.02
1.07
1.01
3.00
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0



	Parameter	Value
1	Title	grd-1-201.4.fid
2	Solvent	DMSO
3	Temperature	300.0
4	Number of Scans	256
5	Receiver Gain	2050.0
6	Relaxation Delay	5.0000
7	Pulse Width	15.0000
8	Spectrometer Frequency	150.97
9	Nucleus	13C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7.311
7.030
7.018
7.006
6.992
4.902
4.859
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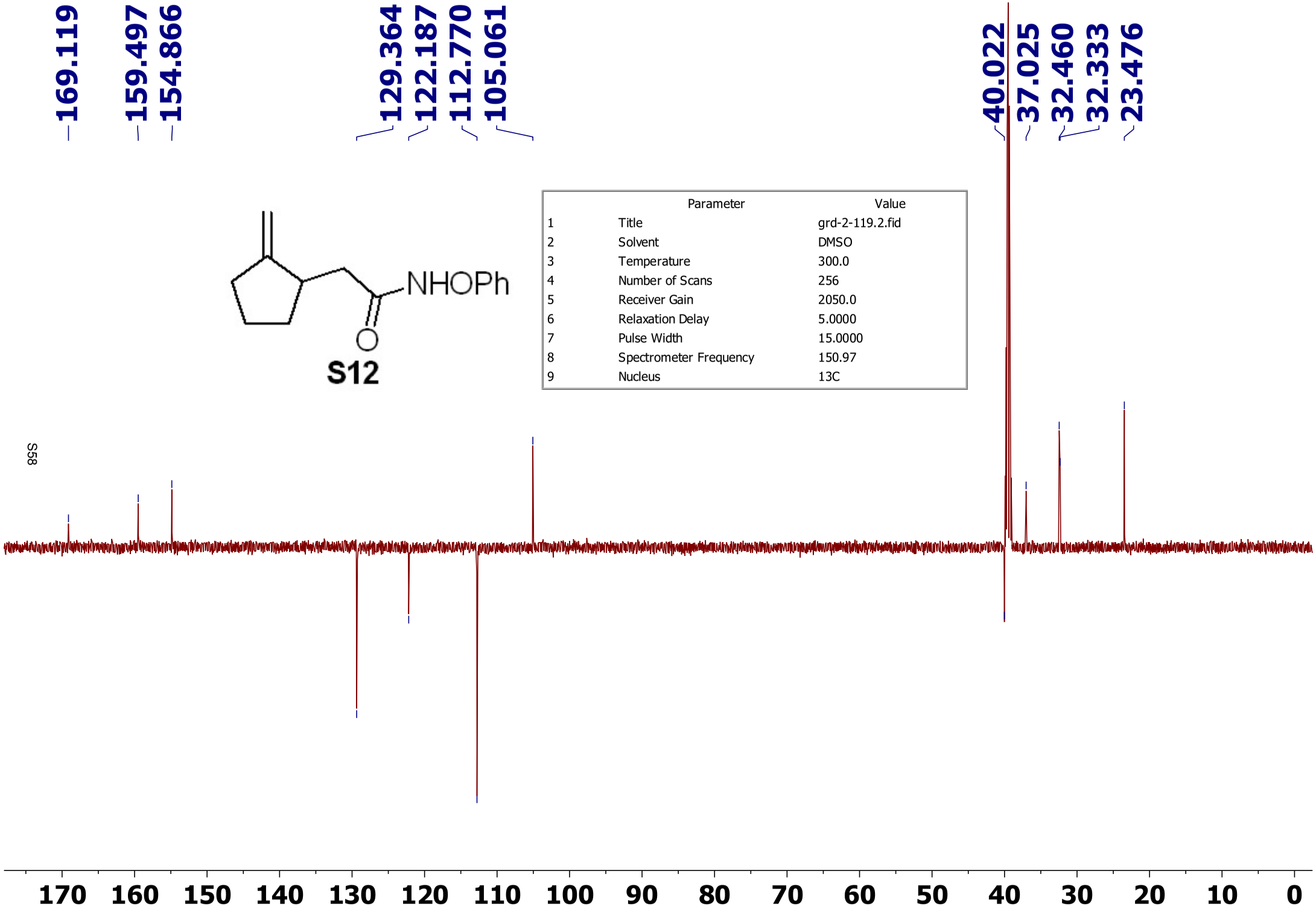
2.476
2.467
2.452
2.443
2.316
2.302
2.289
2.107
2.092
2.084
2.068
1.906
1.898
1.886
1.878
1.704
1.692
1.684
1.563
1.549
1.543
1.537
1.531
1.364
1.350
1.332
NHOPh
O
S12
Parameter Value
1 Title grd-2-119.1.fid
2 Solvent DMSO
3 Temperature 300.0
4 Number of Scans 16
5 Receiver Gain 90.5
6 Relaxation Delay 1.0000
7 Pulse Width 10.6100
8 Spectrometer Frequency 600.32
9 Nucleus 1H
S57
0.97
2.07
3.06
0.99
1.05
1.14
1.08
2.14
1.09
1.10
1.07
1.09
1.06
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0

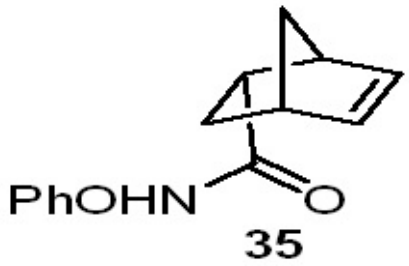


	Parameter	Value
1	Title	grd-2-119.2.fid
2	Solvent	DMSO
3	Temperature	300.0
4	Number of Scans	256
5	Receiver Gain	2050.0
6	Relaxation Delay	5.0000
7	Pulse Width	15.0000
8	Spectrometer Frequency	150.97
9	Nucleus	13C

11.585

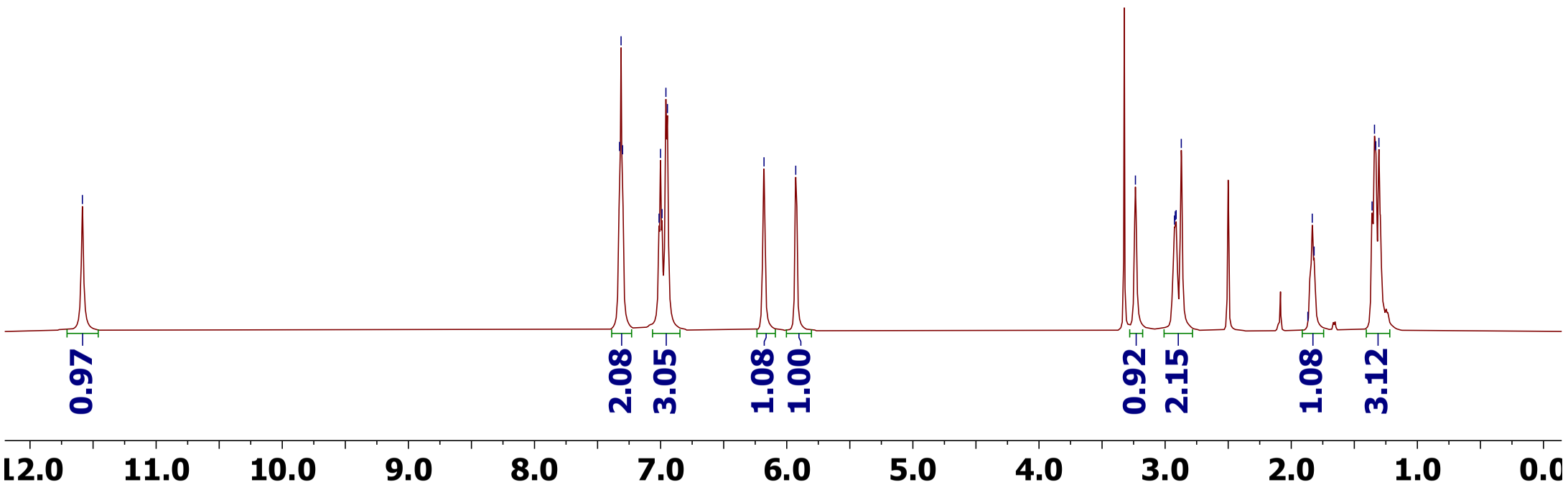
7.326 7.314 7.302 7.013 7.001 6.989 6.958 6.946 6.180 5.929

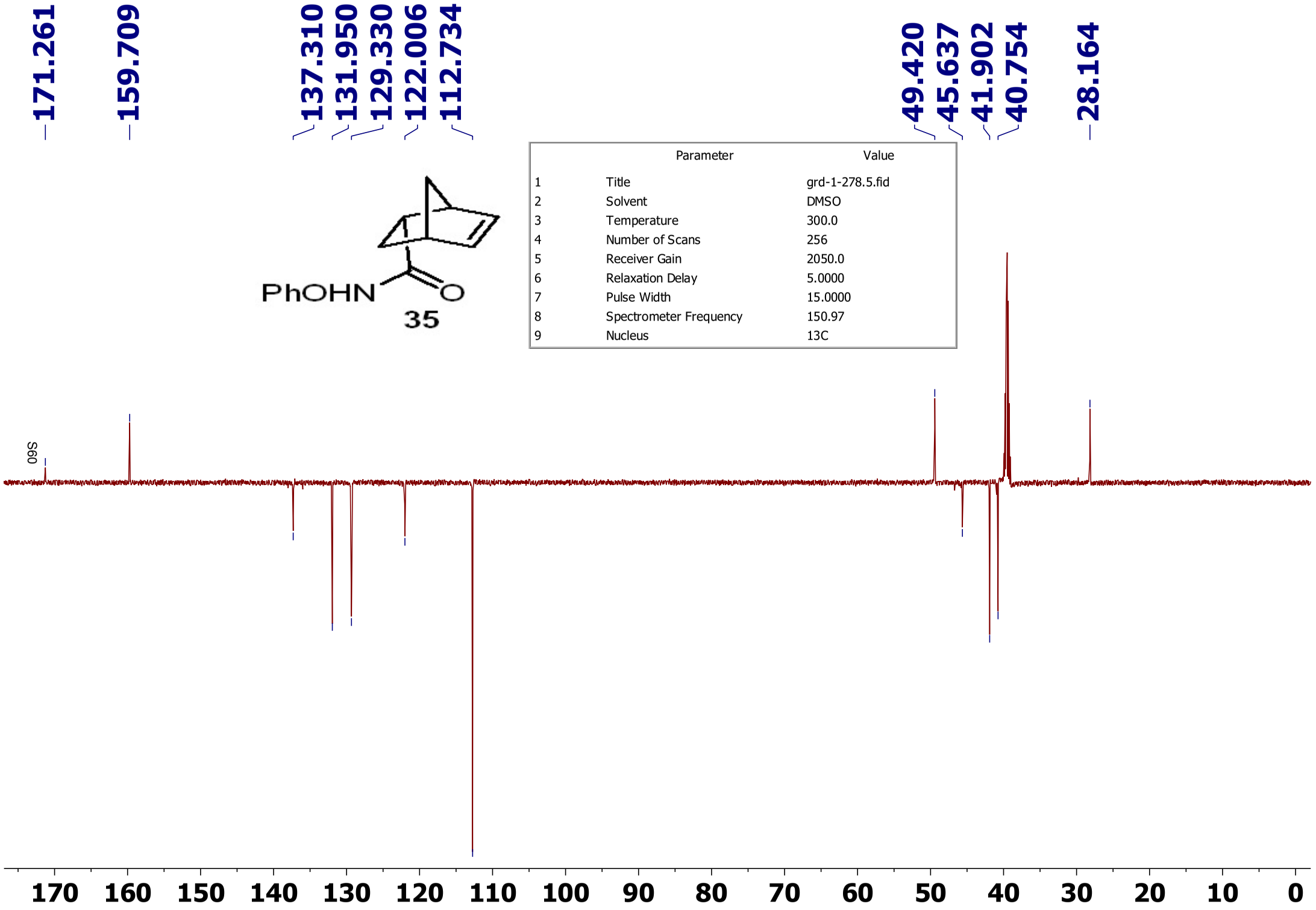
3.235 2.926 2.920 2.913 2.872 1.868 1.834 1.819 1.359 1.340 1.330 1.305



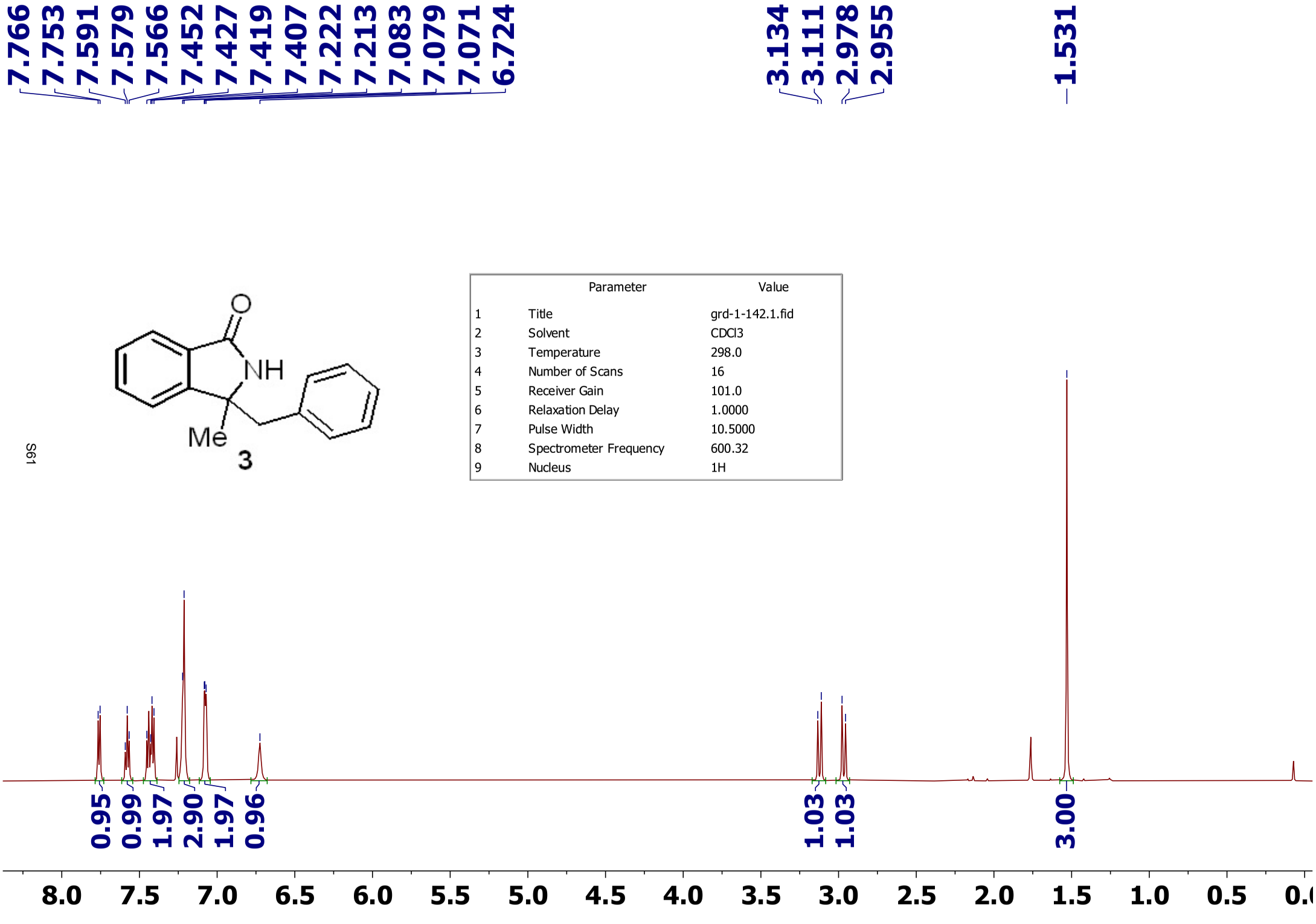
35

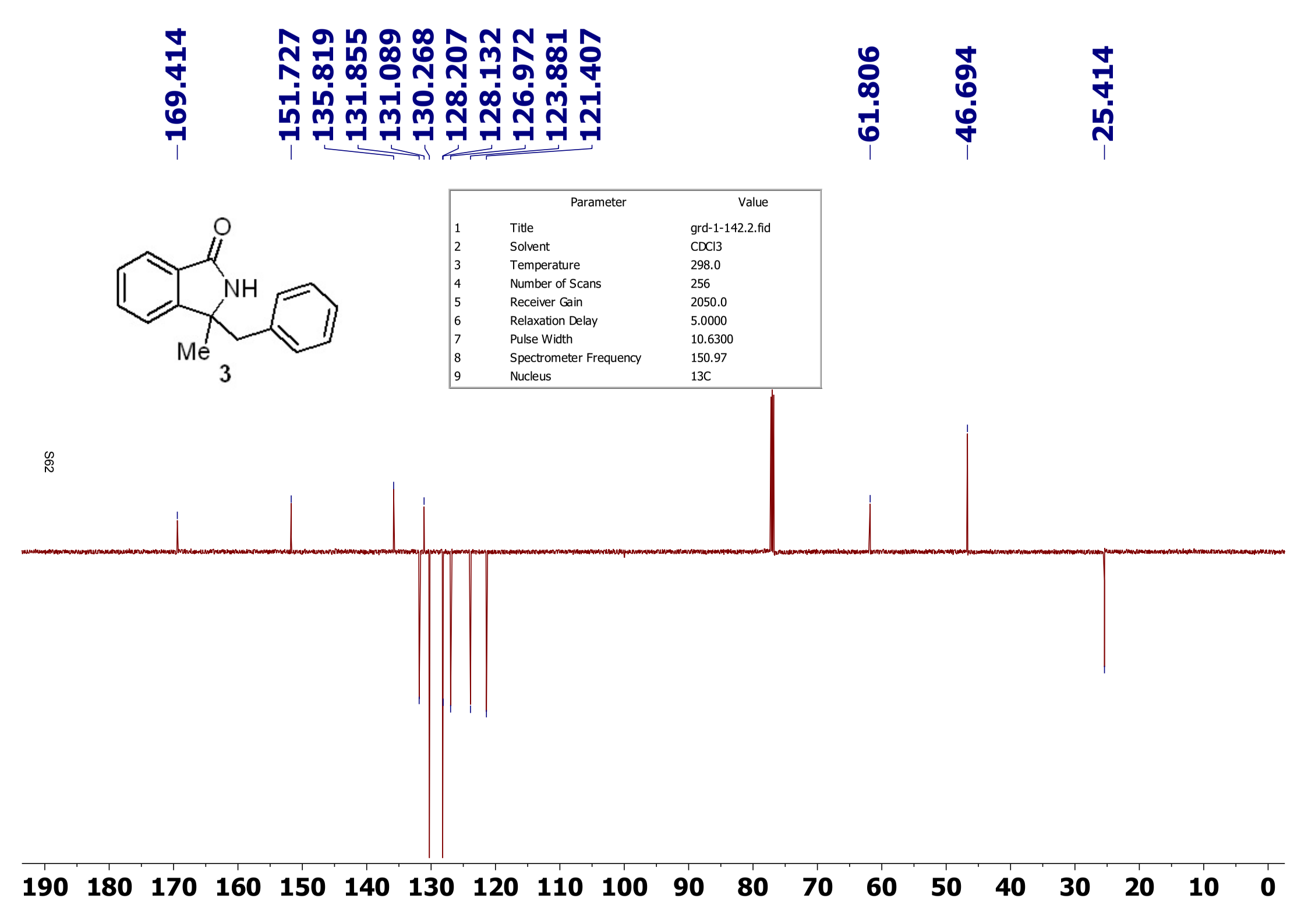
	Parameter	Value
1	Title	grd-1-278.3.fid
2	Solvent	DMSO
3	Temperature	300.0
4	Number of Scans	16
5	Receiver Gain	57.0
6	Relaxation Delay	1.0000
7	Pulse Width	10.6100
8	Spectrometer Frequency	600.32
9	Nucleus	1H

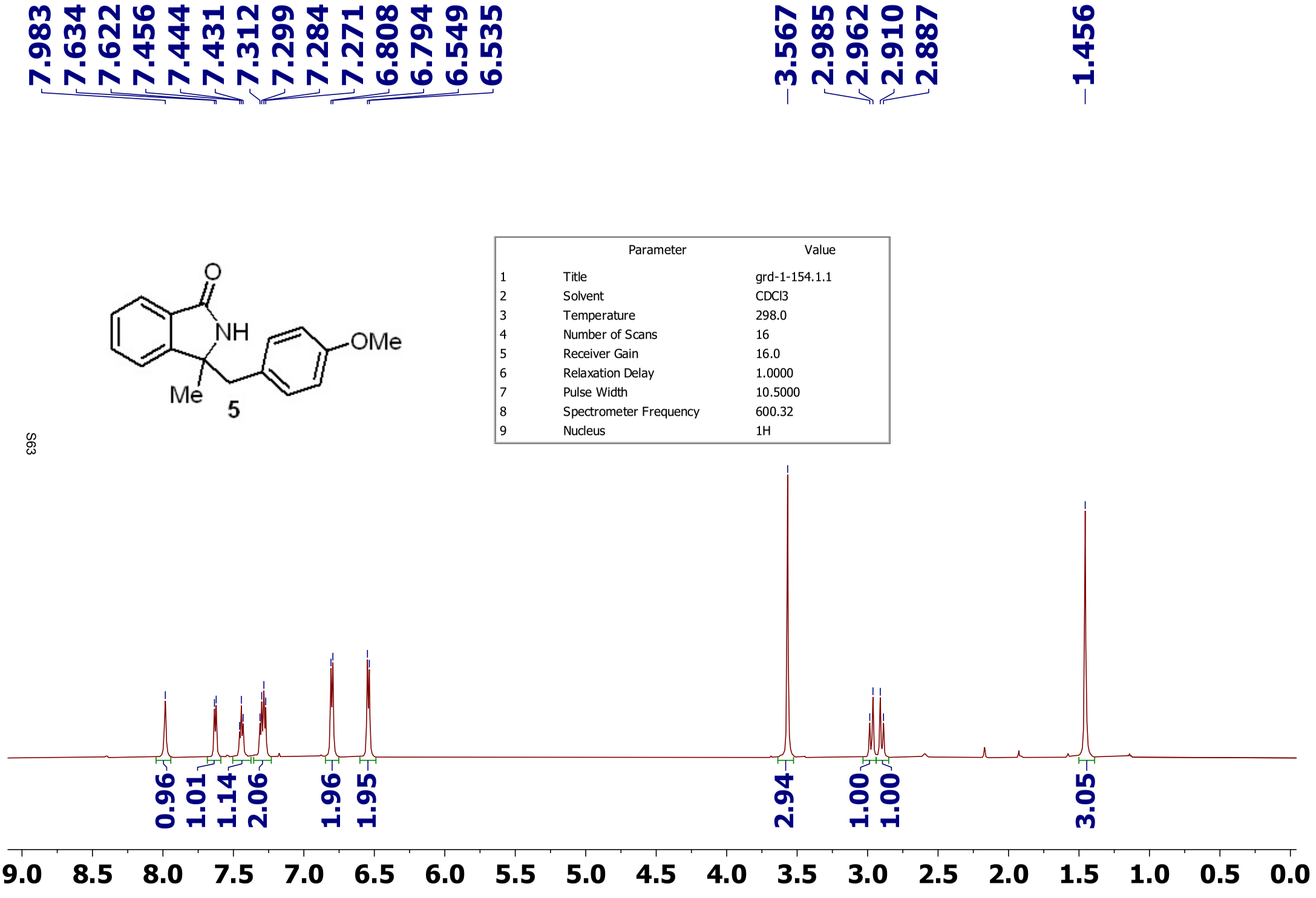




	Parameter	Value
1	Title	grd-1-278.5.fid
2	Solvent	DMSO
3	Temperature	300.0
4	Number of Scans	256
5	Receiver Gain	2050.0
6	Relaxation Delay	5.0000
7	Pulse Width	15.0000
8	Spectrometer Frequency	150.97
9	Nucleus	13C


7.766
7.753
7.591
7.579
7.566
7.452
7.427
7.419
7.407
7.222
7.213
7.083
7.079
7.071
6.724
3.134
3.111
2.978
2.955
1.531
O
NH
Me
3
S61
Parameter Value
1 Title grd-1-142.1.fid
2 Solvent CDCl3
3 Temperature 298.0
4 Number of Scans 16
5 Receiver Gain 101.0
6 Relaxation Delay 1.0000
7 Pulse Width 10.5000
8 Spectrometer Frequency 600.32
9 Nucleus 1H
0.95
0.99
1.97
2.90
1.97
0.96
1.03
1.03
3.00
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


169.414
151.727
135.819
131.855
131.089
130.268
128.207
128.132
126.972
123.881
121.407
61.806
46.694
25.414
O
NH
Me
3
Parameter Value
1 Title grd-1-142.2.fid
2 Solvent CDCl3
3 Temperature 298.0
4 Number of Scans 256
5 Receiver Gain 2050.0
6 Relaxation Delay 5.0000
7 Pulse Width 10.6300
8 Spectrometer Frequency 150.97
9 Nucleus 13C
S62
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0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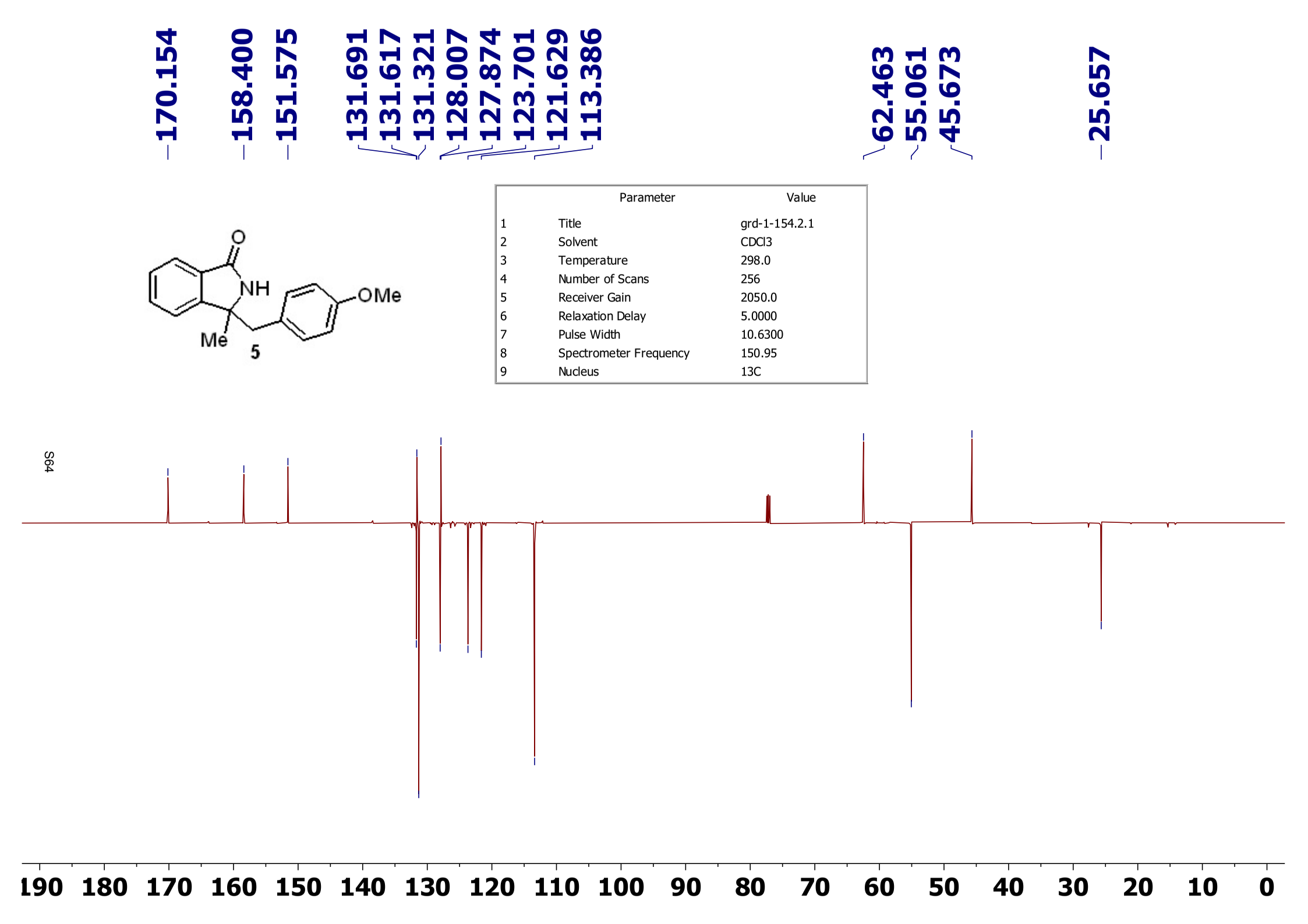
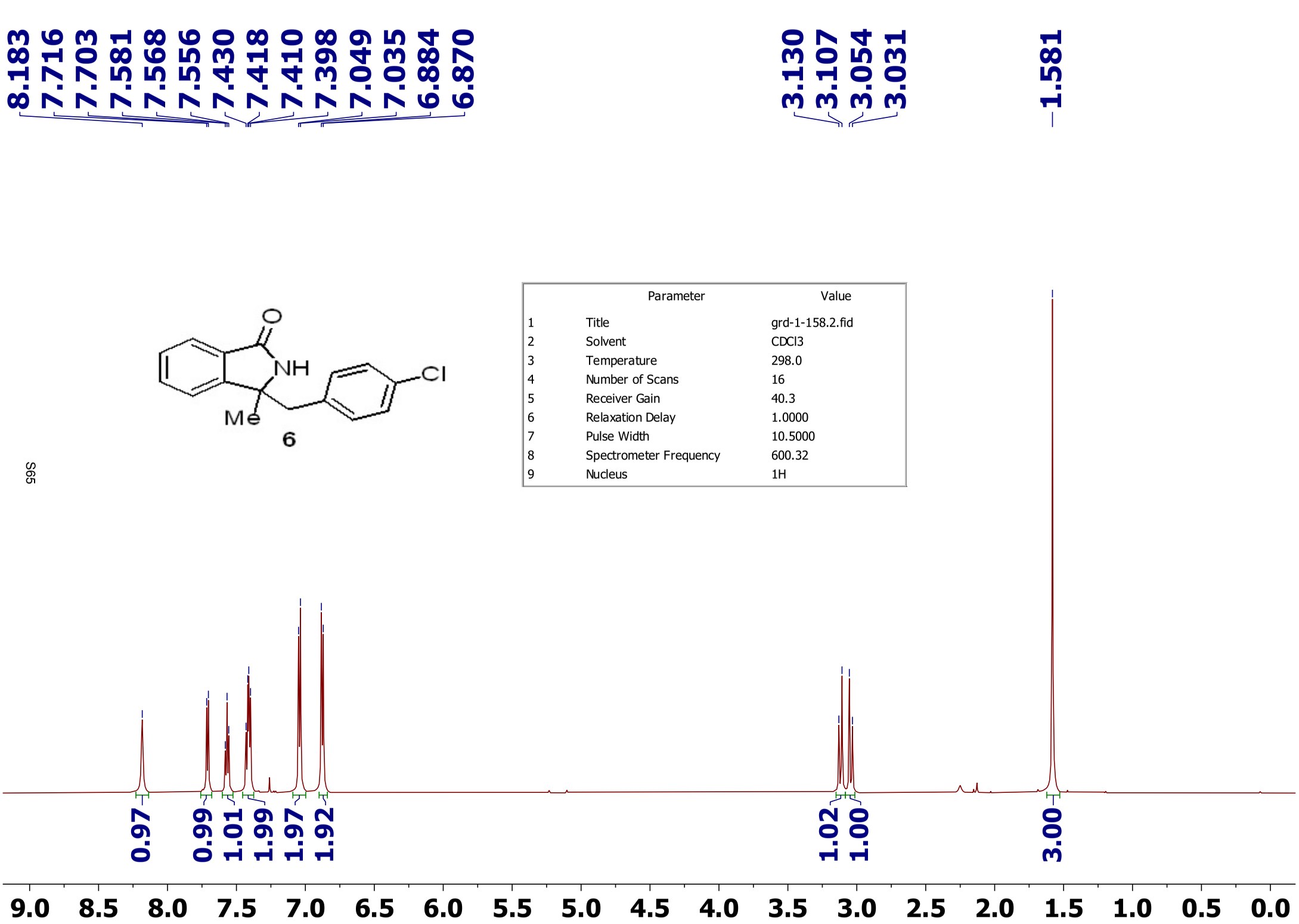
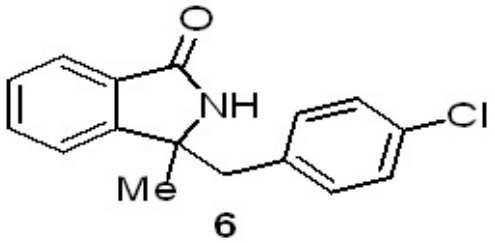
7.622
7.456
7.444
7.431
7.312
7.299
7.284
7.271
6.808
6.794
6.549
6.535
3.567
2.985
2.962
2.910
2.887
1.456
O
NH
OMe
Me
5
Parameter Value
1 Title grd-1-154.1.1
2 Solvent CDCl3
3 Temperature 298.0
4 Number of Scans 16
5 Receiver Gain 16.0
6 Relaxation Delay 1.0000
7 Pulse Width 10.5000
8 Spectrometer Frequency 600.32
9 Nucleus 1H
S63
0.96
1.01
1.14
2.06
1.96
1.95
2.94
1.00
1.00
3.05
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


170.154
158.400
151.575
131.691
131.617
131.321
128.007
127.874
123.701
121.629
113.386
62.463
55.061
45.673
25.657
O
NH
OMe
Me
5
Parameter Value
1 Title grd-1-154.2.1
2 Solvent CDCl3
3 Temperature 298.0
4 Number of Scans 256
5 Receiver Gain 2050.0
6 Relaxation Delay 5.0000
7 Pulse Width 10.6300
8 Spectrometer Frequency 150.95
9 Nucleus 13C
S64
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0



	Parameter	Value
1	Title	grd-1-158.2.fid
2	Solvent	CDCl3
3	Temperature	298.0
4	Number of Scans	16
5	Receiver Gain	40.3
6	Relaxation Delay	1.0000
7	Pulse Width	10.5000
8	Spectrometer Frequency	600.32
9	Nucleus	1H

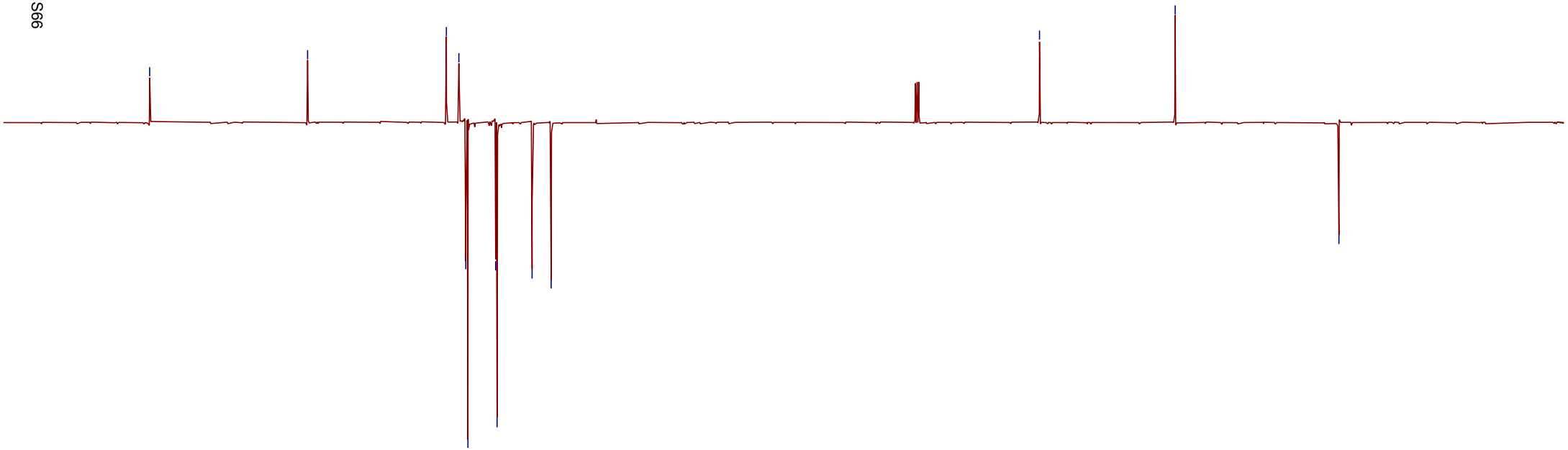
170.187
151.034
134.201
132.685
131.849
131.583
128.223
128.051
123.802
121.490
62.256
45.801
25.936



6

	Parameter	Value
1	Title	grd-1-158.3.fid
2	Solvent	CDCl3
3	Temperature	298.0
4	Number of Scans	256
5	Receiver Gain	2050.0
6	Relaxation Delay	5.0000
7	Pulse Width	10.6300
8	Spectrometer Frequency	150.97
9	Nucleus	13C

S66

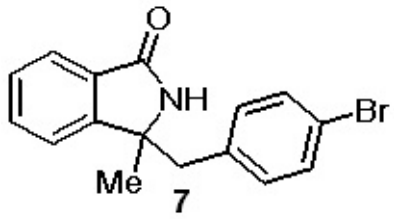


180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

7.815 7.728 7.716 7.589 7.577 7.565 7.443 7.431 7.418 7.413 7.400 7.233 7.219 6.848 6.834

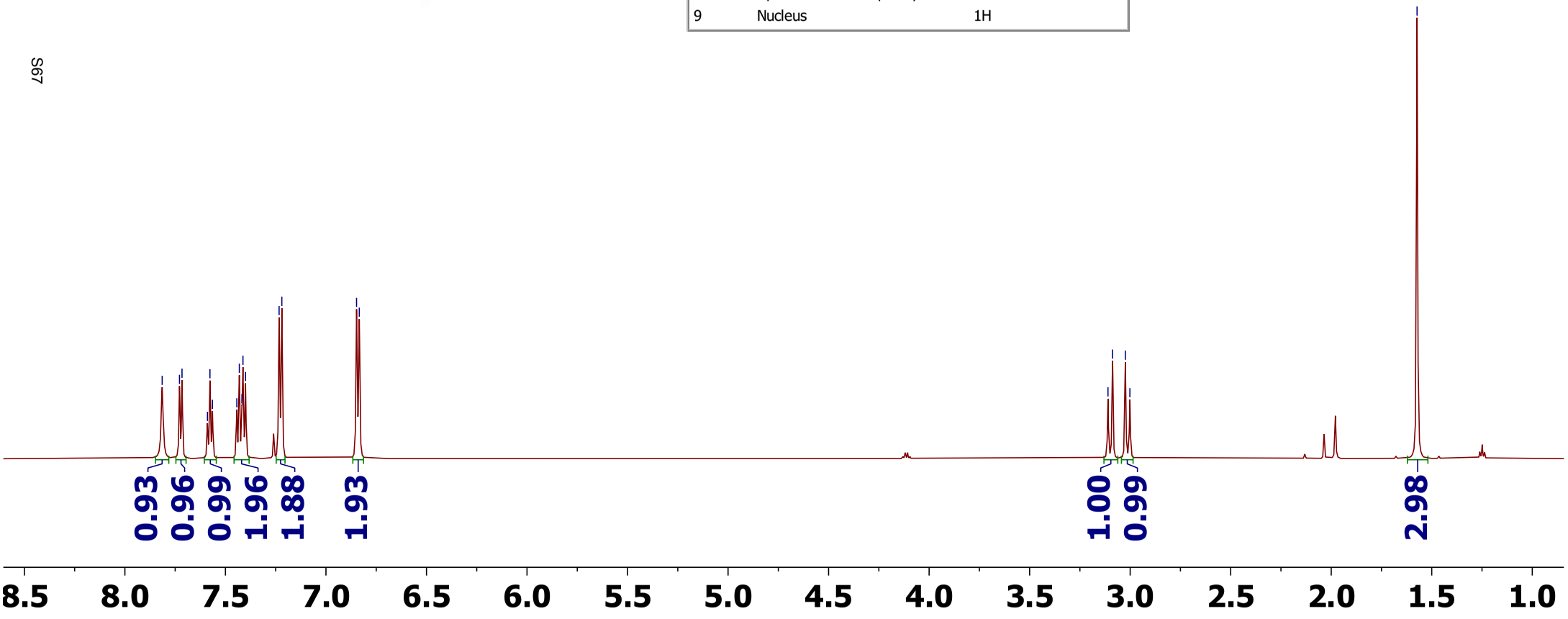
3.110 3.088 3.024 3.001

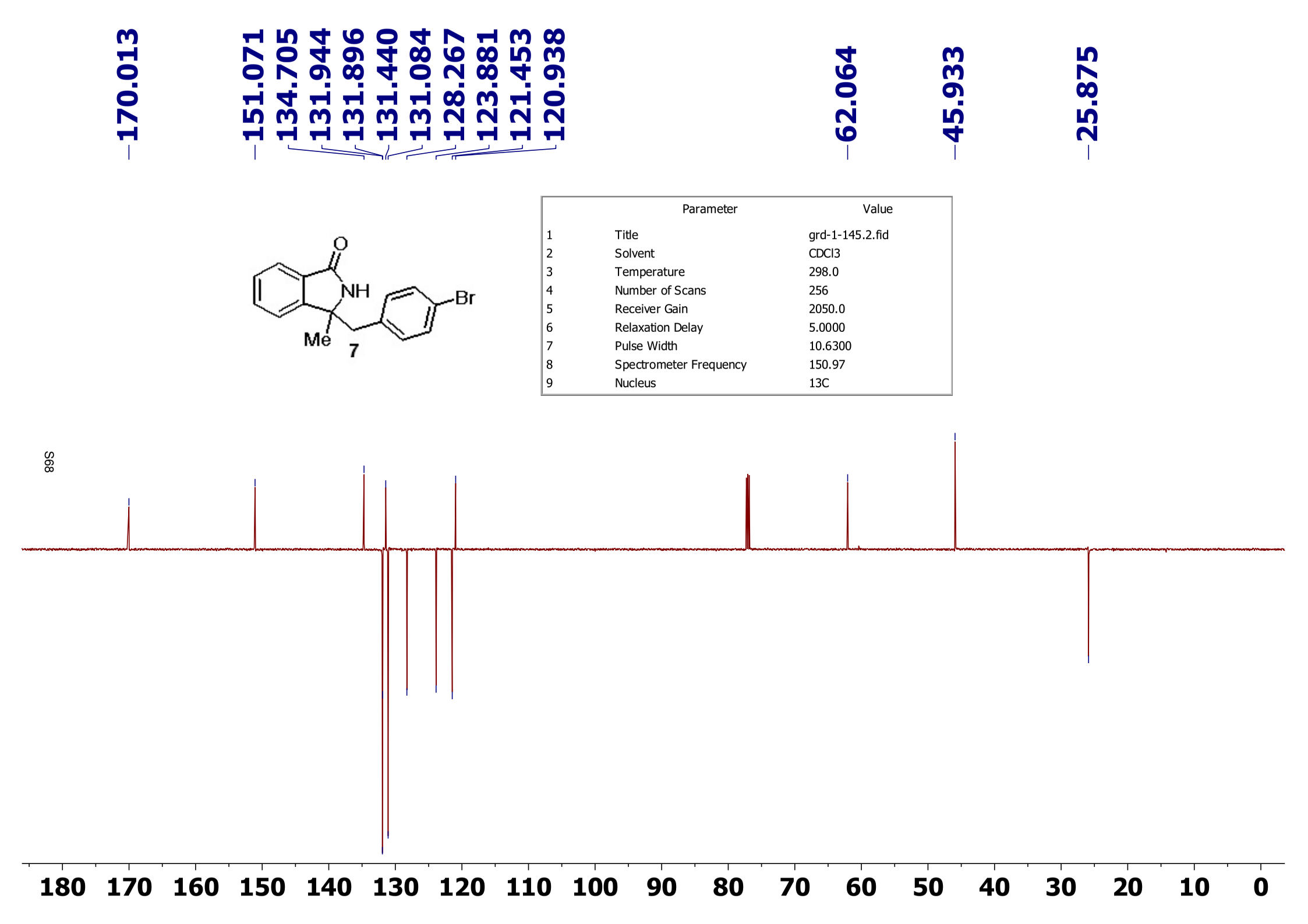
1.573



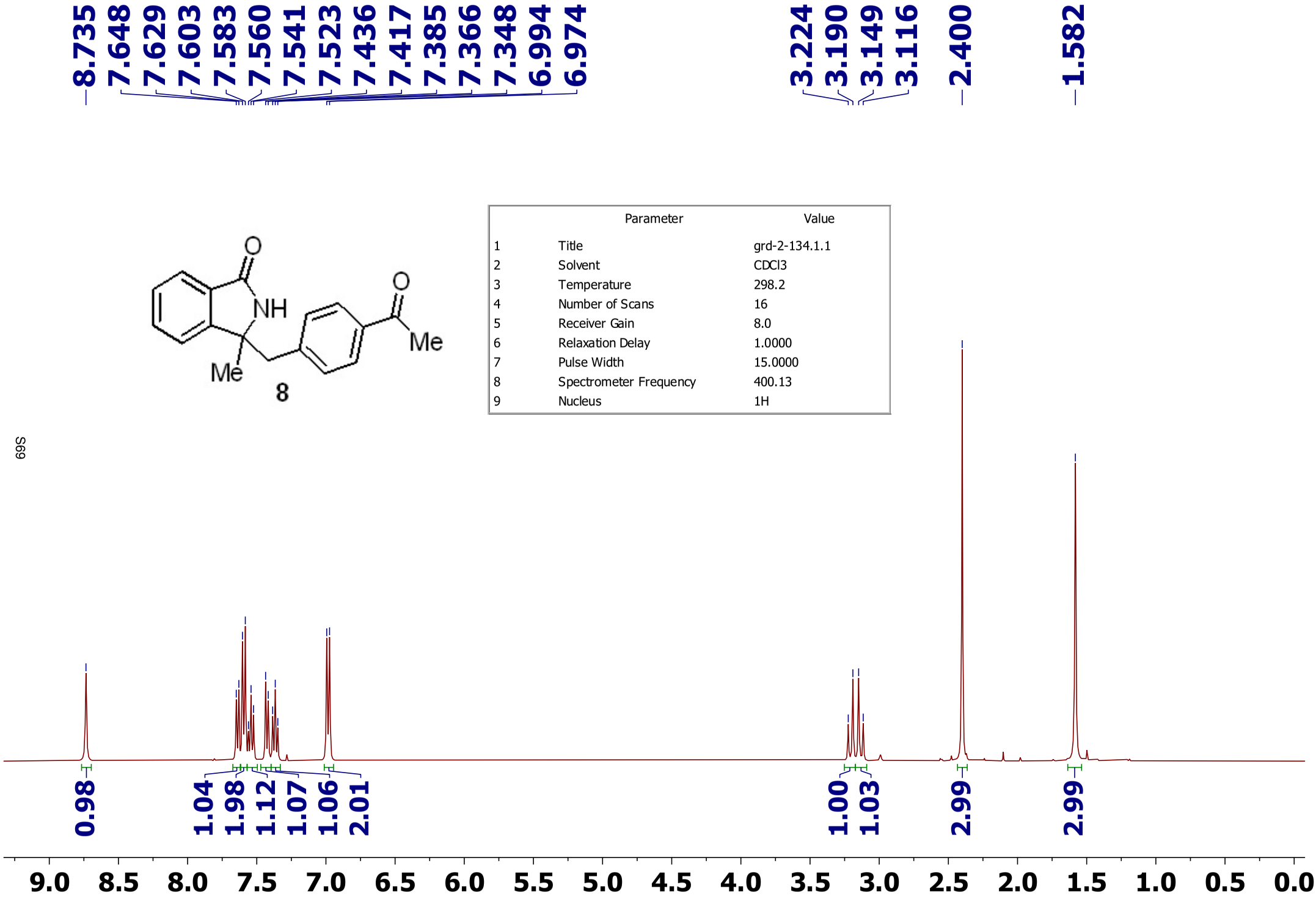
7

	Parameter	Value
1	Title	grd-1-145.1.fid
2	Solvent	CDCl3
3	Temperature	298.0
4	Number of Scans	16
5	Receiver Gain	57.0
6	Relaxation Delay	1.0000
7	Pulse Width	10.5000
8	Spectrometer Frequency	600.32
9	Nucleus	1H





	Parameter	Value
1	Title	grd-1-145.2.fid
2	Solvent	CDCl3
3	Temperature	298.0
4	Number of Scans	256
5	Receiver Gain	2050.0
6	Relaxation Delay	5.0000
7	Pulse Width	10.6300
8	Spectrometer Frequency	150.97
9	Nucleus	13C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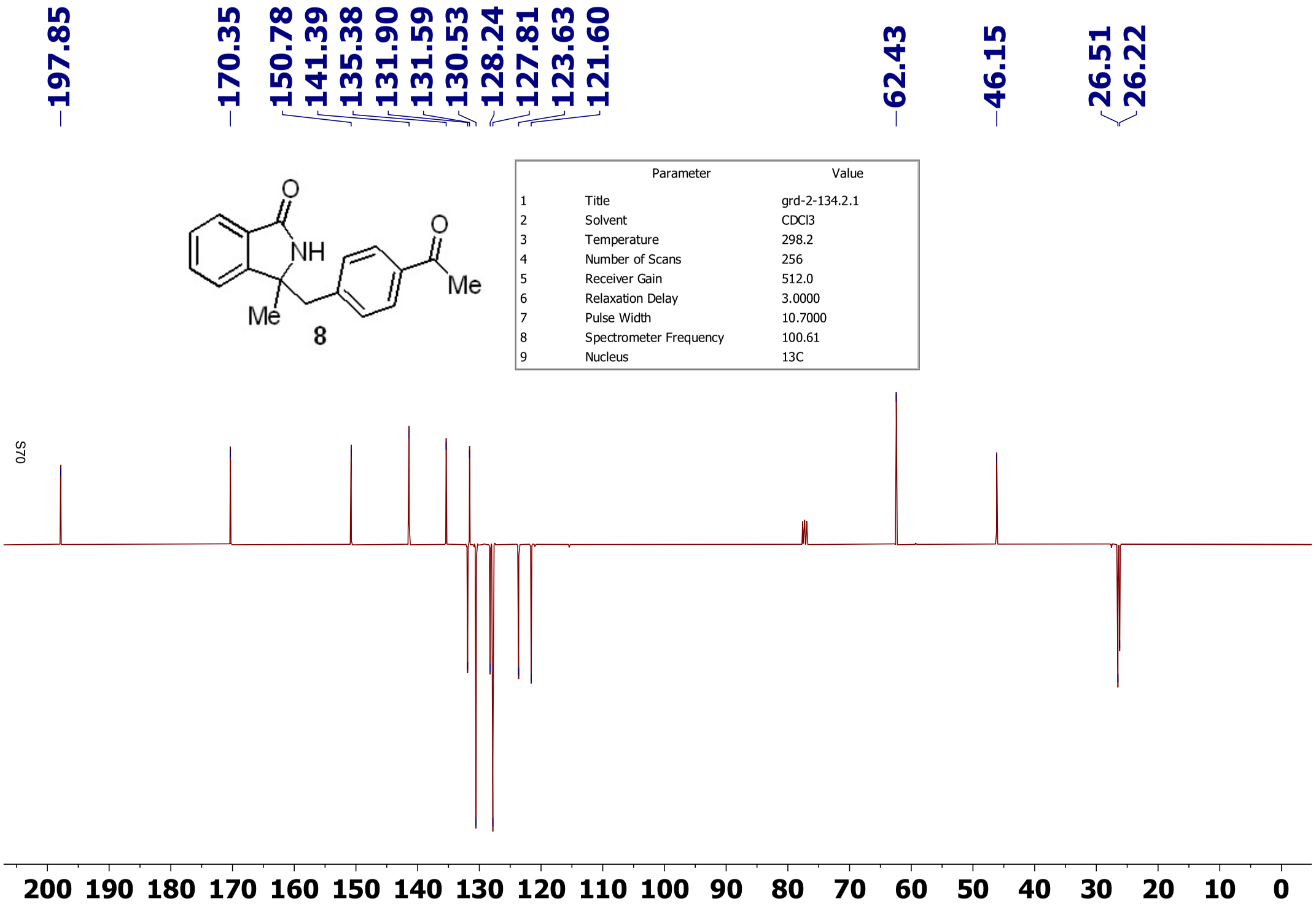
7.583
7.560
7.541
7.523
7.436
7.417
7.385
7.366
7.348
6.994
6.974
3.224
3.190
3.149
3.116
2.400
1.582
O
NH
O
Me
Me
8
Parameter Value
1 Title grd-2-134.1.1
2 Solvent CDCl3
3 Temperature 298.2
4 Number of Scans 16
5 Receiver Gain 8.0
6 Relaxation Delay 1.0000
7 Pulse Width 15.0000
8 Spectrometer Frequency 400.13
9 Nucleus 1H
S69
0.98
1.04
1.98
1.12
1.07
1.06
2.01
1.00
1.03
2.99
2.99
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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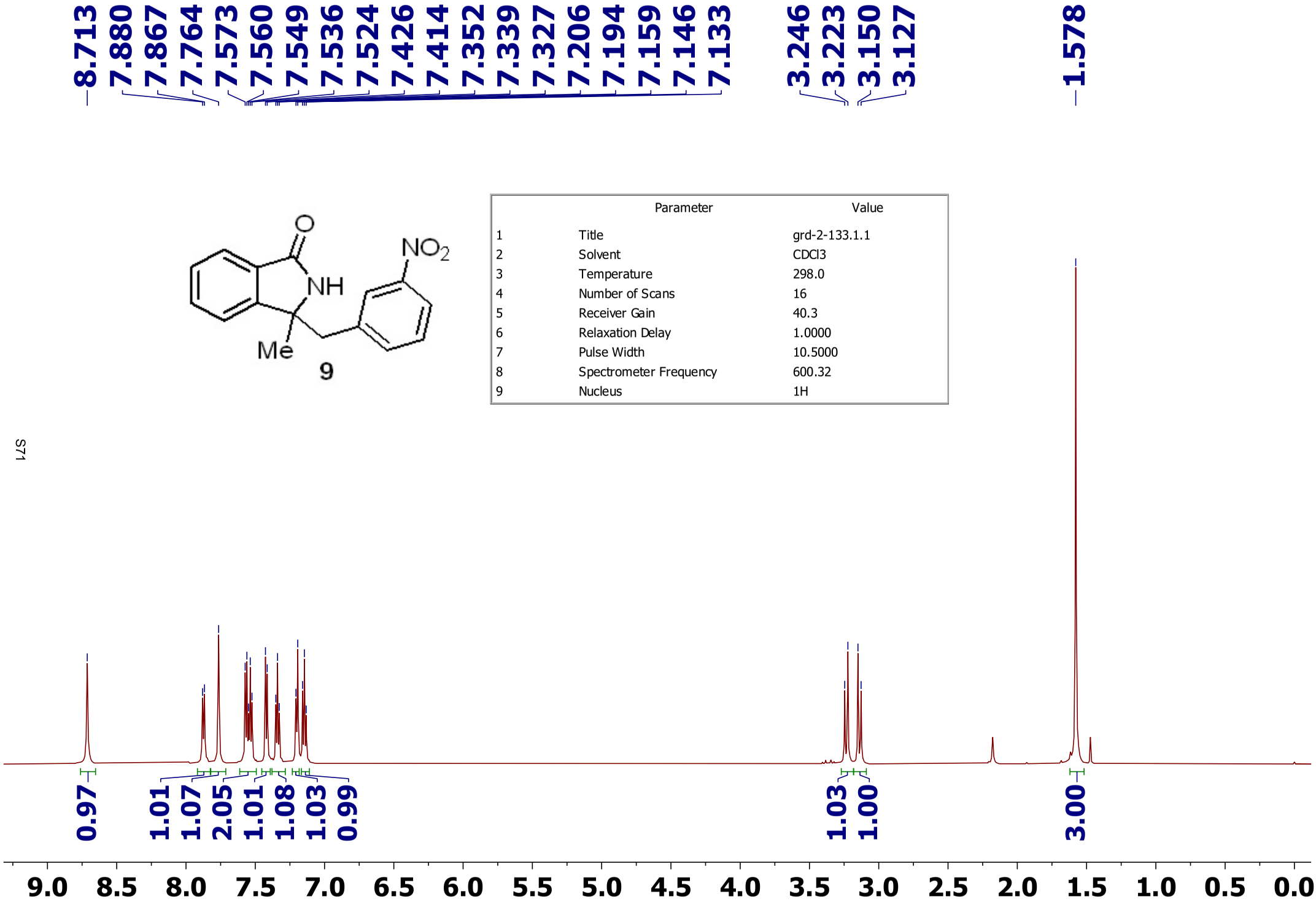
150.78
141.39
135.38
131.90
131.59
130.53
128.24
127.81
123.63
121.60
62.43
46.15
26.51
26.22
O
NH
O
Me
Me
8
Parameter Value
1 Title grd-2-134.2.1
2 Solvent CDCl3
3 Temperature 298.2
4 Number of Scans 256
5 Receiver Gain 512.0
6 Relaxation Delay 3.0000
7 Pulse Width 10.7000
8 Spectrometer Frequency 100.61
9 Nucleus 13C
S70
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0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7.560
7.549
7.536
7.524
7.426
7.414
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7.339
7.327
7.206
7.194
7.159
7.146
7.133
3.246
3.223
3.150
3.127
1.578
O
NH
NO2
Me
9
Parameter
Value
1 Title grd-2-133.1.1
2 Solvent CDCl3
3 Temperature 298.0
4 Number of Scans 16
5 Receiver Gain 40.3
6 Relaxation Delay 1.0000
7 Pulse Width 10.5000
8 Spectrometer Frequency 600.32
9 Nucleus 1H
S71
0.97
1.01
1.07
2.05
1.01
1.08
1.03
0.99
1.03
1.00
3.00
9.0
8.5
8.0
7.5
7.0
6.5
6.0
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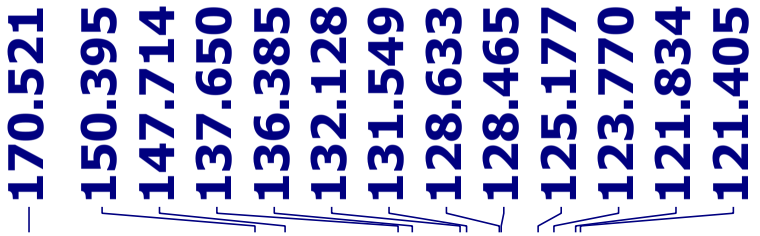
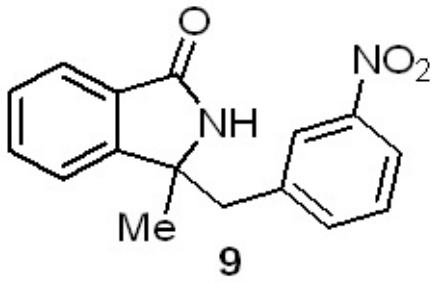
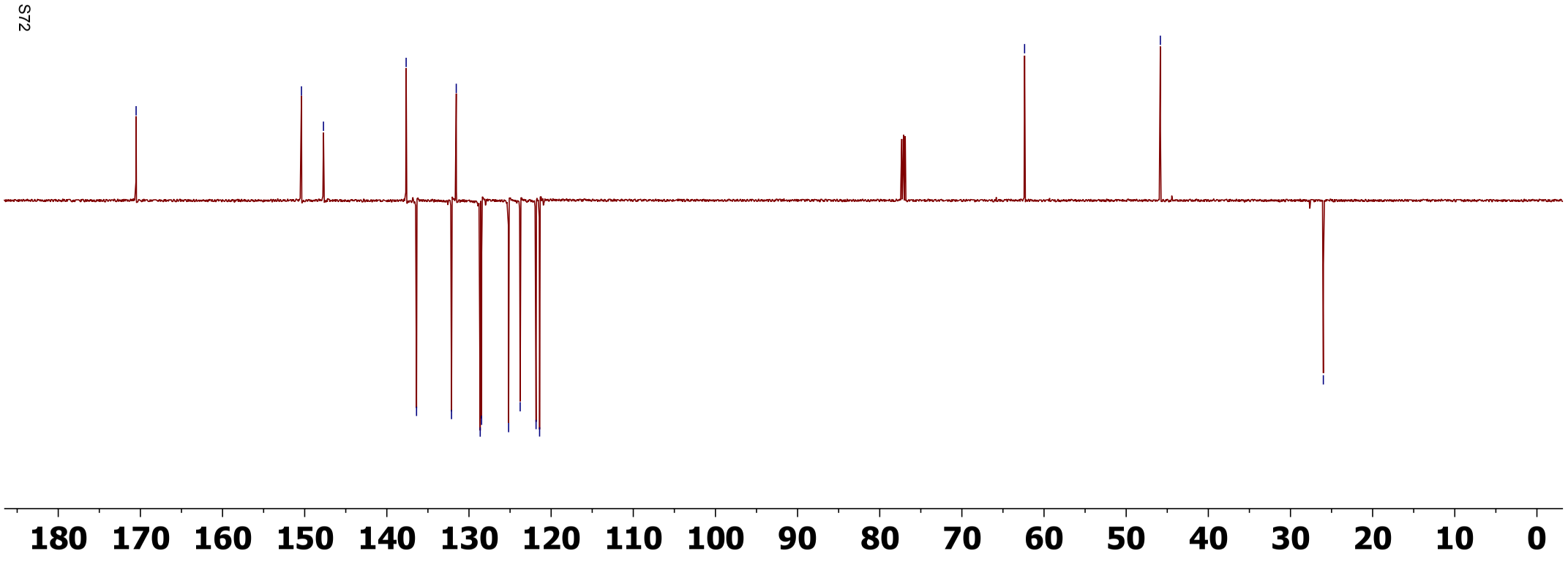




9

	Parameter	Value
1	Title	grd-2-133.2.1
2	Solvent	CDCl3
3	Temperature	298.0
4	Number of Scans	256
5	Receiver Gain	2050.0
6	Relaxation Delay	5.0000
7	Pulse Width	10.6300
8	Spectrometer Frequency	150.95
9	Nucleus	13C



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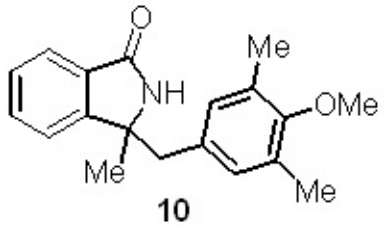
3.632

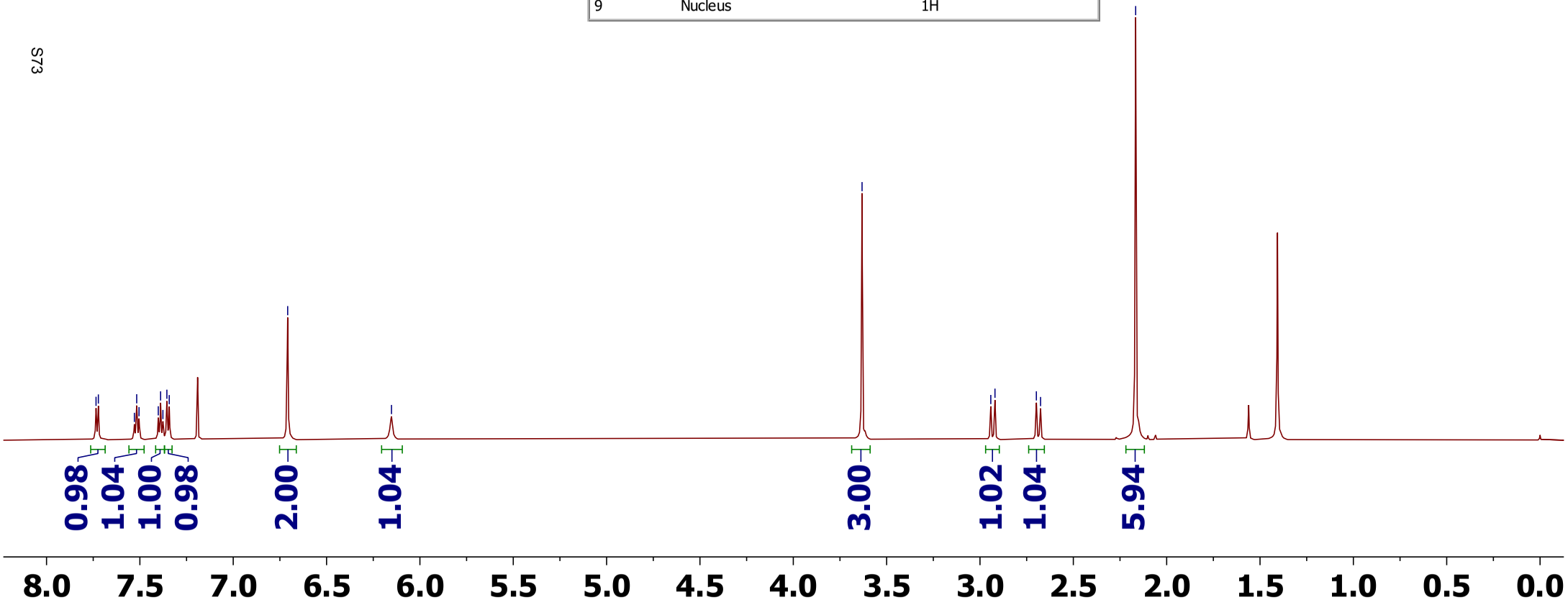
2.942 2.920 2.698 2.676

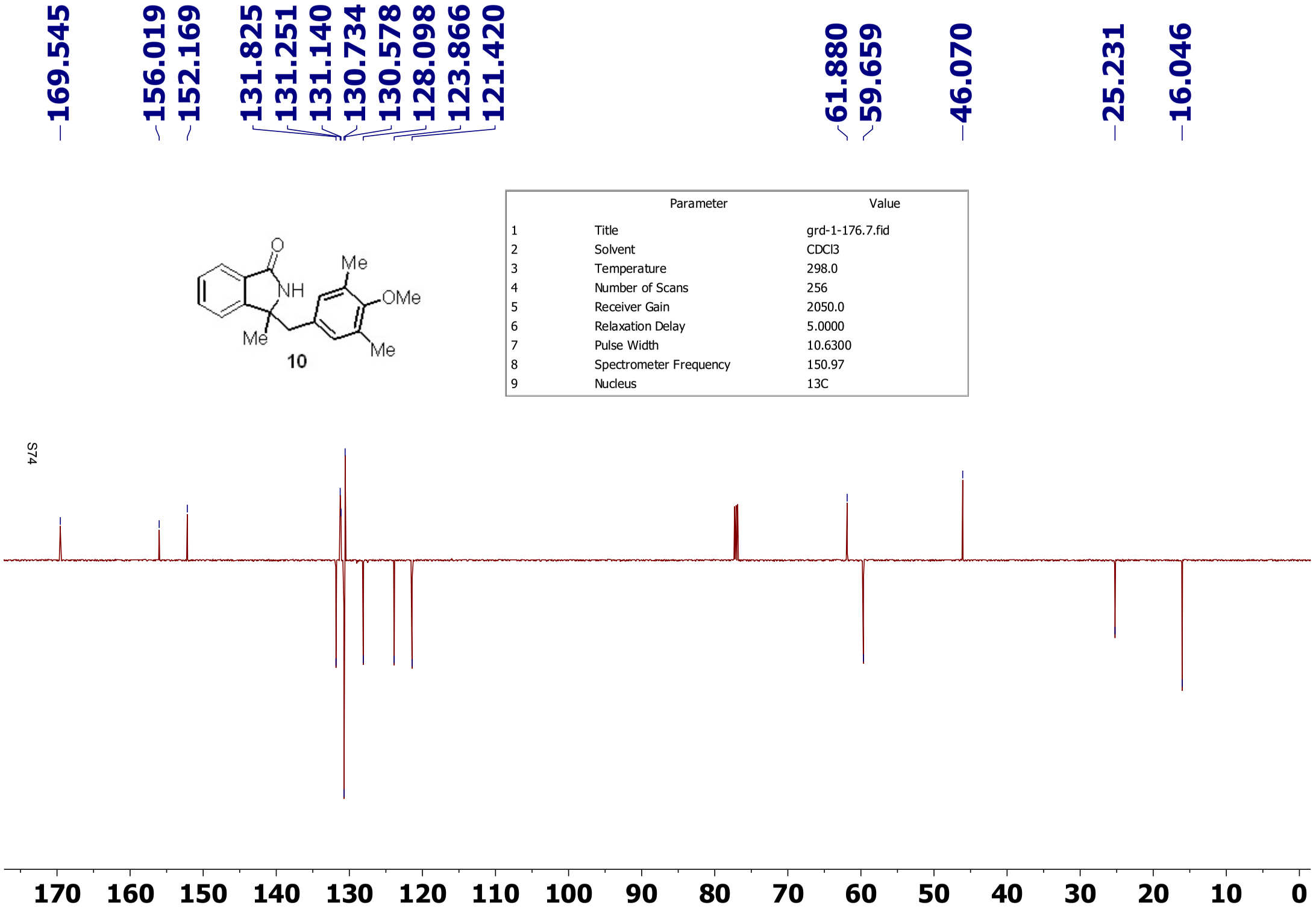
2.166



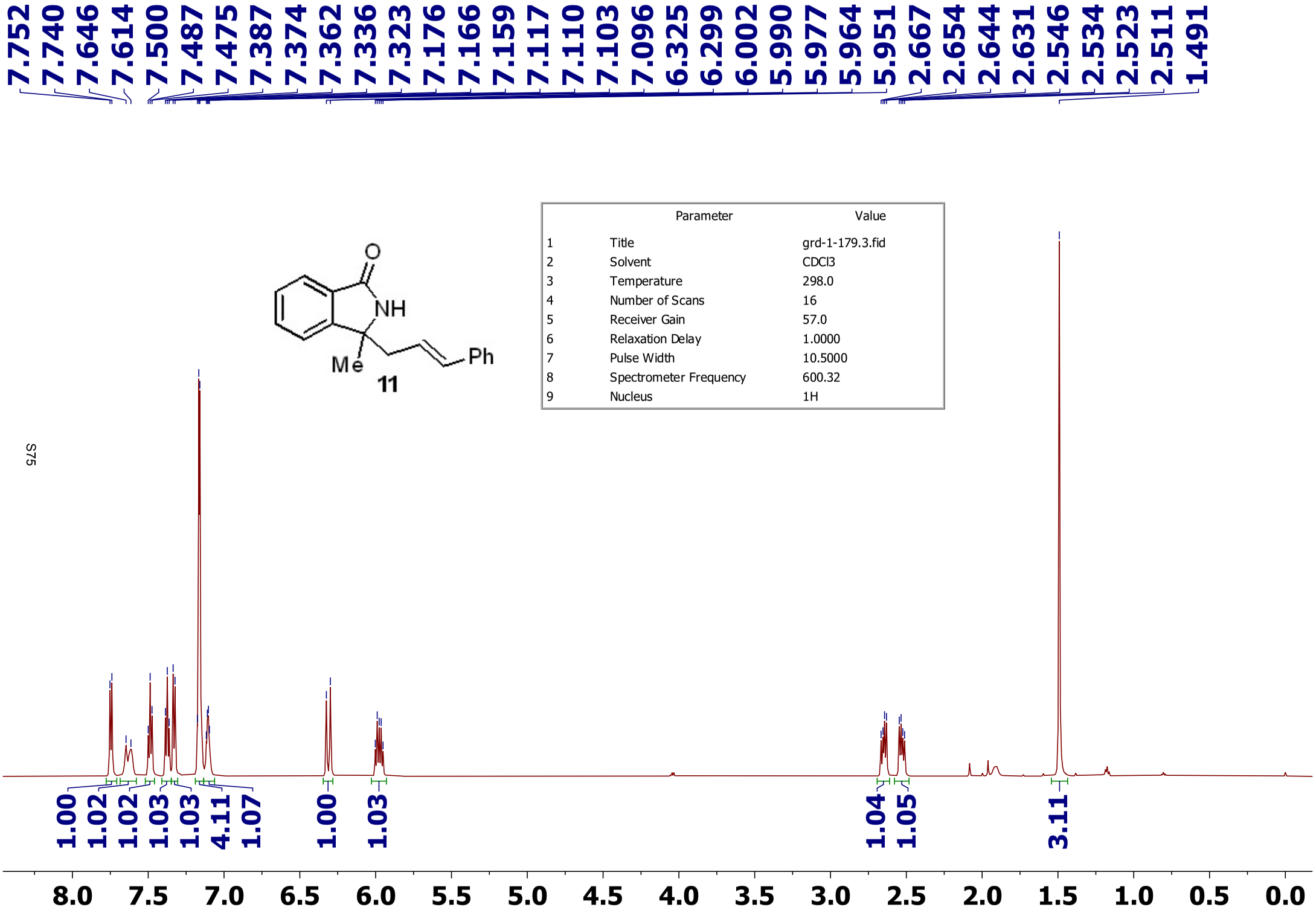
10

	Parameter	Value
1	Title	grd-1-176.8.fid
2	Solvent	CDCl3
3	Temperature	298.0
4	Number of Scans	16
5	Receiver Gain	128.0
6	Relaxation Delay	1.0000
7	Pulse Width	10.5000
8	Spectrometer Frequency	600.32
9	Nucleus	1H





	Parameter	Value
1	Title	grd-1-176.7.fid
2	Solvent	CDCl3
3	Temperature	298.0
4	Number of Scans	256
5	Receiver Gain	2050.0
6	Relaxation Delay	5.0000
7	Pulse Width	10.6300
8	Spectrometer Frequency	150.97
9	Nucleus	13C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7.500
7.487
7.475
7.387
7.374
7.362
7.336
7.323
7.176
7.166
7.159
7.117
7.110
7.103
7.096
6.325
6.299
6.002
5.990
5.977
5.964
5.951
2.667
2.654
2.644
2.631
2.546
2.534
2.523
2.511
1.491
O
NH
Me
Ph
11
Parameter Value
1 Title grd-1-179.3.fid
2 Solvent CDCl3
3 Temperature 298.0
4 Number of Scans 16
5 Receiver Gain 57.0
6 Relaxation Delay 1.0000
7 Pulse Width 10.5000
8 Spectrometer Frequency 600.32
9 Nucleus 1H
S75
1.00
1.02
1.02
1.03
1.03
4.11
1.07
1.00
1.03
1.04
1.05
3.11
8.0
7.5
7.0
6.5
6.0
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4.0
3.5
3.0
2.5
2.0
1.5
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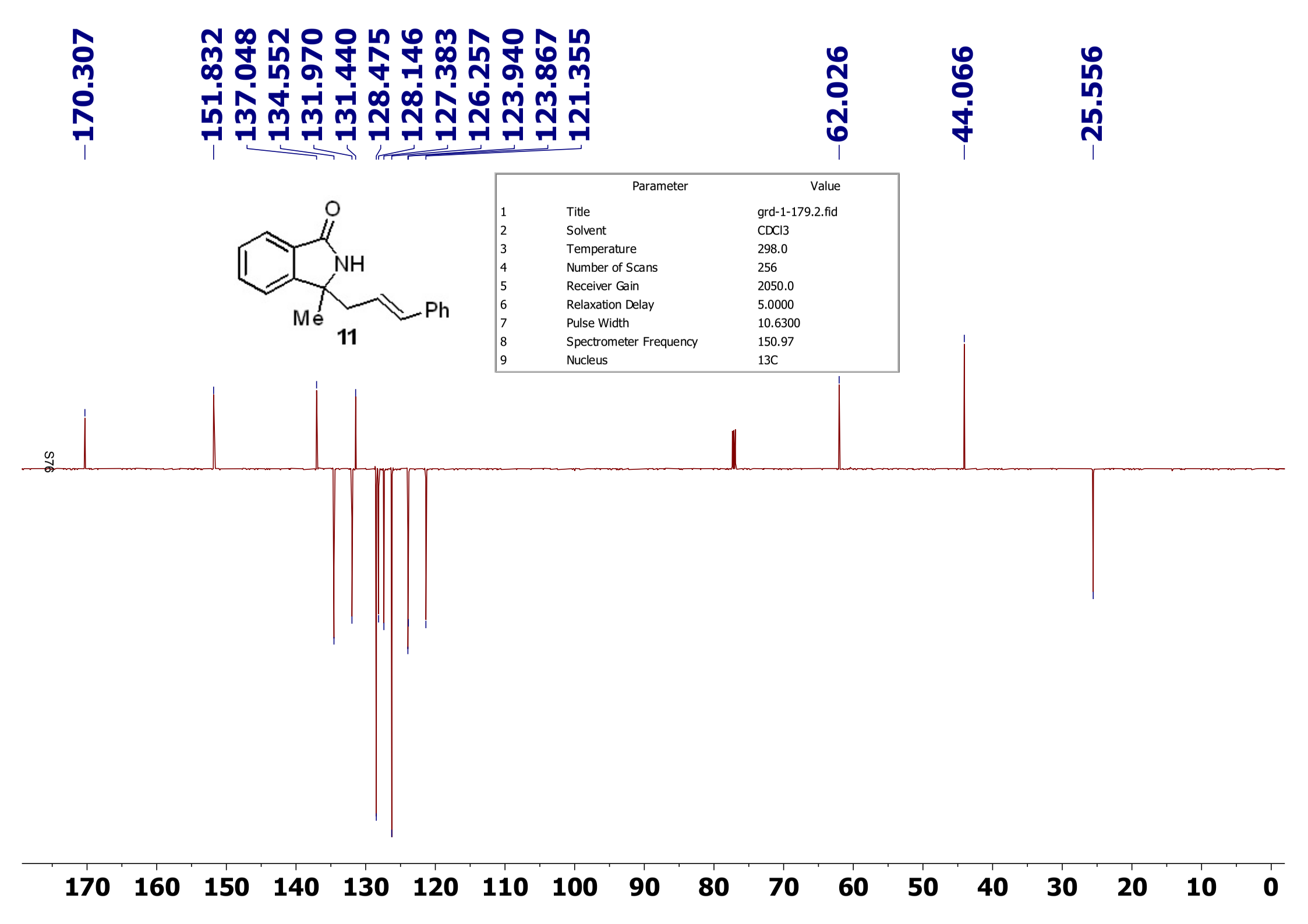
0.0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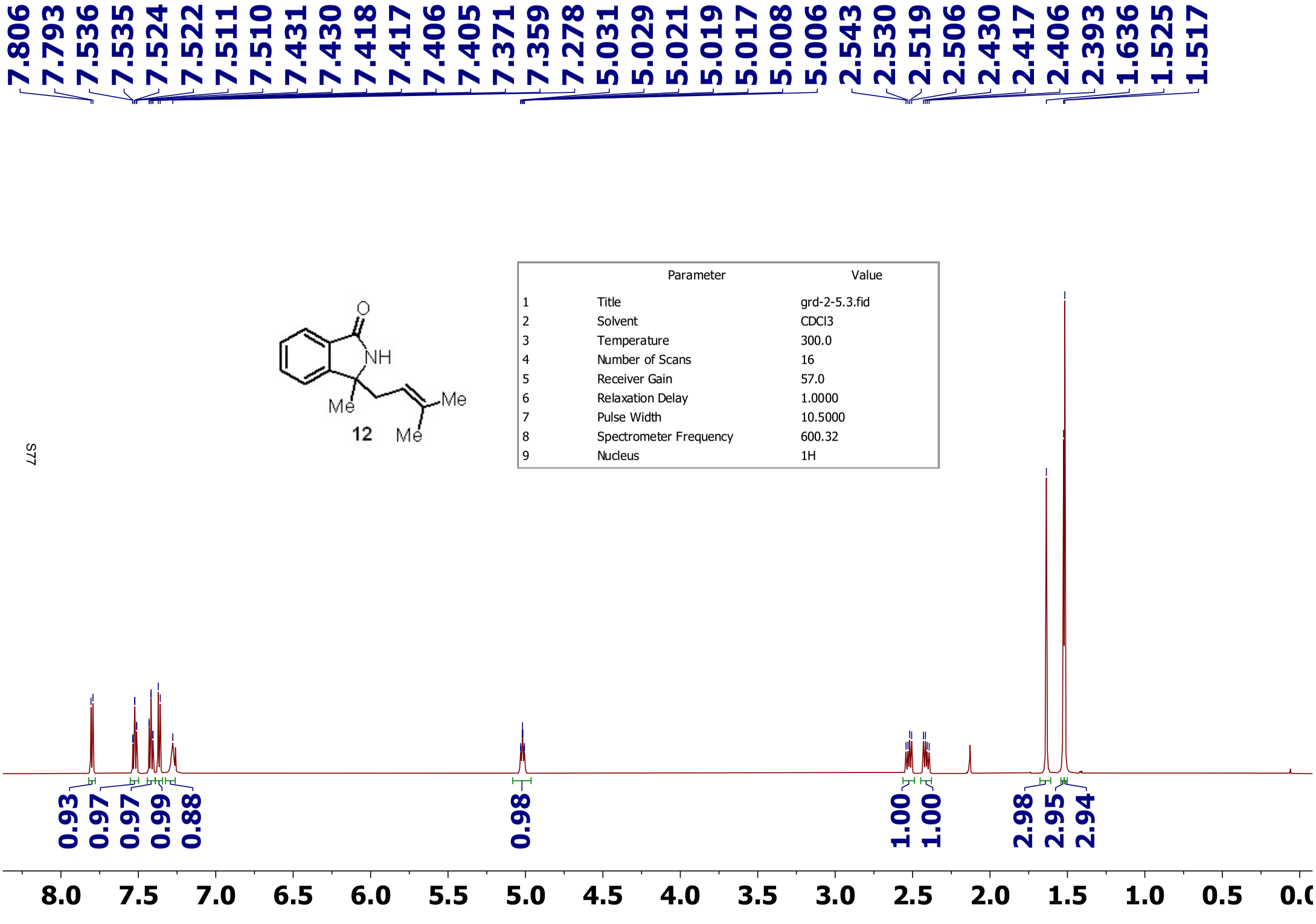
151.832
137.048
134.552
131.970
131.440
128.475
128.146
127.383
126.257
123.940
123.867
121.355
62.026
44.066
25.556
O
NH
Me
Ph
11
Parameter Value
1 Title grd-1-179.2.fid
2 Solvent CDCl3
3 Temperature 298.0
4 Number of Scans 256
5 Receiver Gain 2050.0
6 Relaxation Delay 5.0000
7 Pulse Width 10.6300
8 Spectrometer Frequency 150.97
9 Nucleus 13C
S76
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0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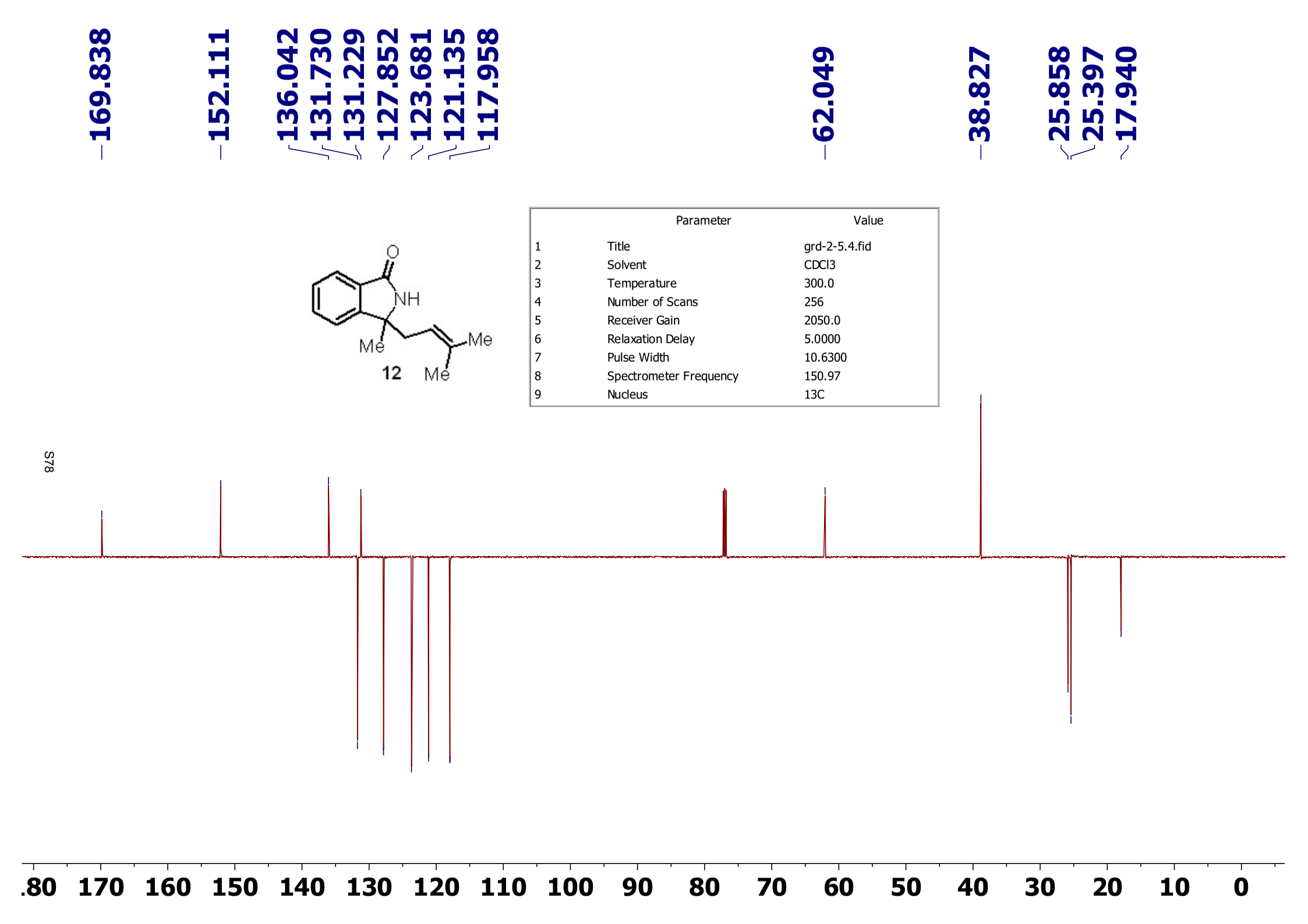
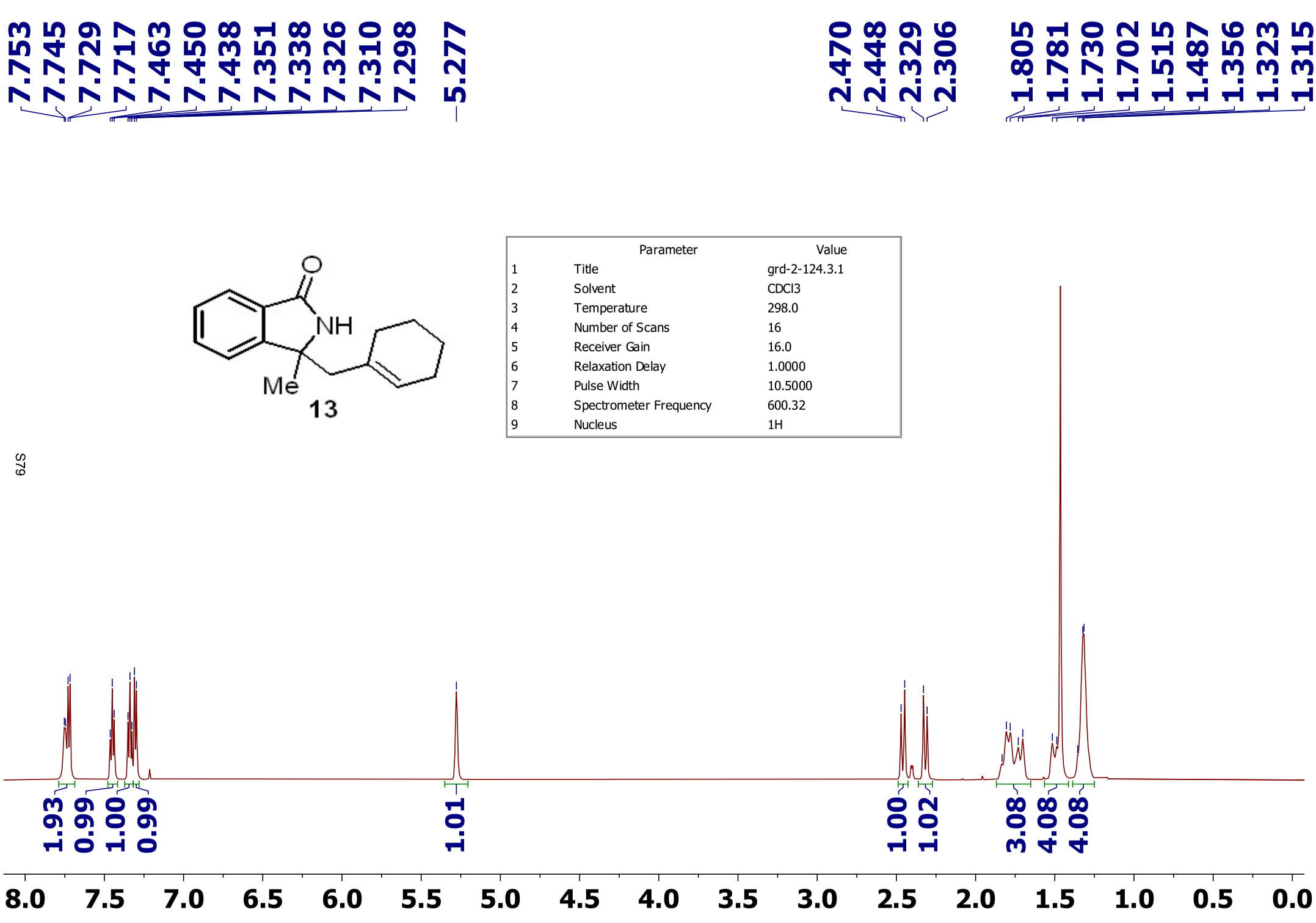
7.793
7.536
7.535
7.524
7.522
7.511
7.510
7.431
7.430
7.418
7.417
7.406
7.405
7.371
7.359
7.278
5.031
5.029
5.021
5.019
5.017
5.008
5.006
2.543
2.530
2.519
2.506
2.430
2.417
2.406
2.393
1.636
1.525
1.517
O
NH
Me
Me
Me
12
Parameter
Value
1 Title grd-2-5.3.fid
2 Solvent CDCl3
3 Temperature 300.0
4 Number of Scans 16
5 Receiver Gain 57.0
6 Relaxation Delay 1.0000
7 Pulse Width 10.5000
8 Spectrometer Frequency 600.32
9 Nucleus 1H
S77
0.93
0.97
0.97
0.99
0.88
0.98
1.00
1.00
2.98
2.95
2.94
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


169.838
152.111
136.042
131.730
131.229
127.852
123.681
121.135
117.958
62.049
38.827
25.858
25.397
17.940
O
NH
Me
Me
Me
12
Parameter
Value
1 Title grd-2-5.4.fid
2 Solvent CDCl3
3 Temperature 300.0
4 Number of Scans 256
5 Receiver Gain 2050.0
6 Relaxation Delay 5.0000
7 Pulse Width 10.6300
8 Spectrometer Frequency 150.97
9 Nucleus 13C
S78
.80 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0


7.753
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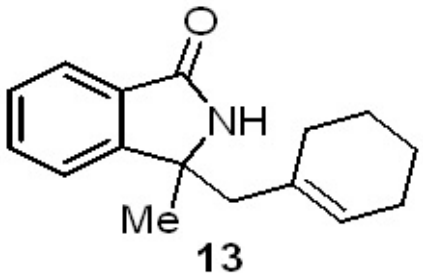
7.729
7.717
7.463
7.450
7.438
7.351
7.338
7.326
7.310
7.298
5.277
2.470
2.448
2.329
2.306
1.805
1.781
1.730
1.702
1.515
1.487
1.356
1.323
1.315
O
NH
Me
13
Parameter Value
1 Title grd-2-124.3.1
2 Solvent CDCl3
3 Temperature 298.0
4 Number of Scans 16
5 Receiver Gain 16.0
6 Relaxation Delay 1.0000
7 Pulse Width 10.5000
8 Spectrometer Frequency 600.32
9 Nucleus 1H
S79
1.93
0.99
1.00
0.99
1.01
1.00
1.02
3.08
4.08
4.08
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

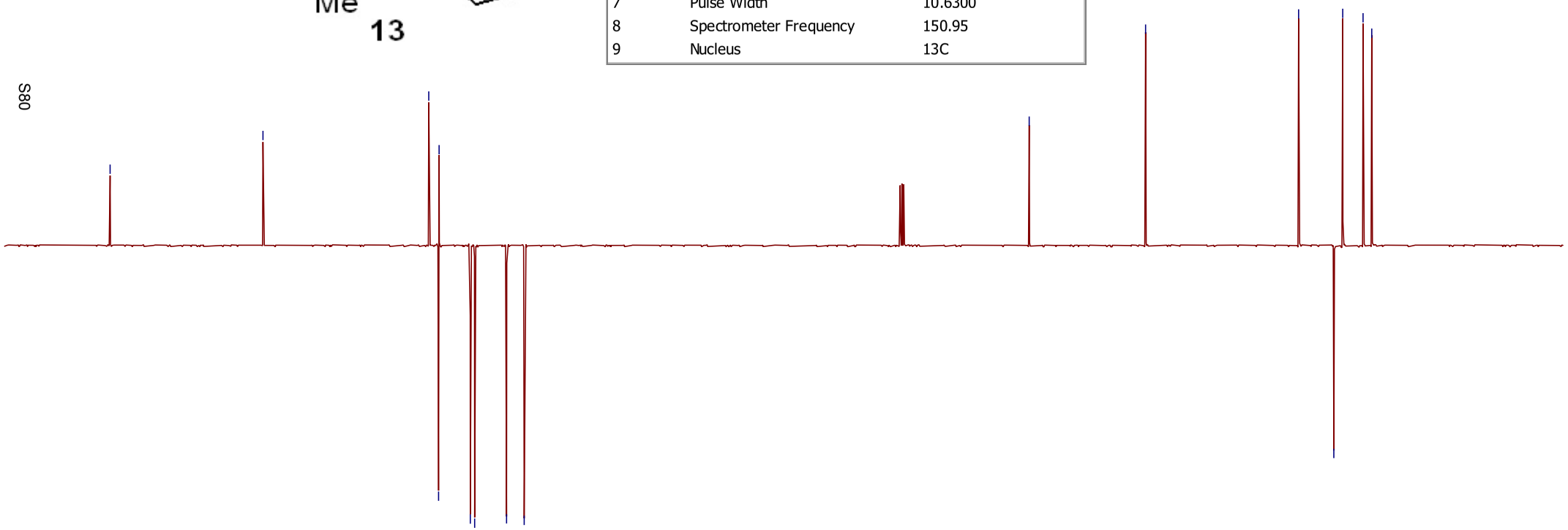
170.150
152.190
132.711
131.558
131.496
127.829
127.318
123.571
121.499
62.166
48.491
30.512
26.375
25.328
22.941
21.919



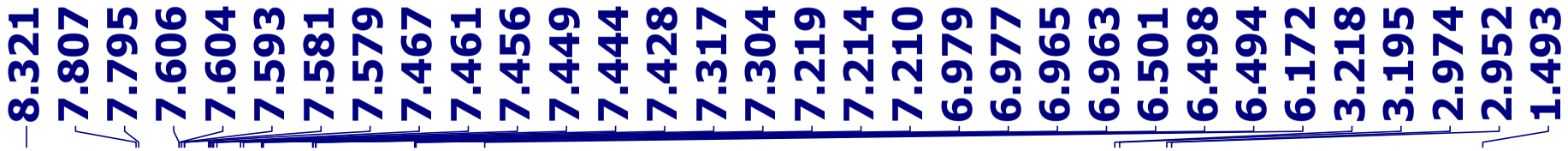
13

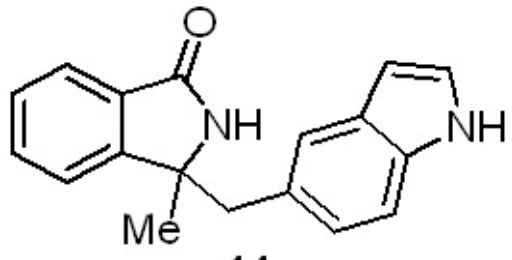
	Parameter	Value
1	Title	grd-2-124.4.1
2	Solvent	CDCl3
3	Temperature	298.0
4	Number of Scans	256
5	Receiver Gain	2050.0
6	Relaxation Delay	5.0000
7	Pulse Width	10.6300
8	Spectrometer Frequency	150.95
9	Nucleus	13C

S80



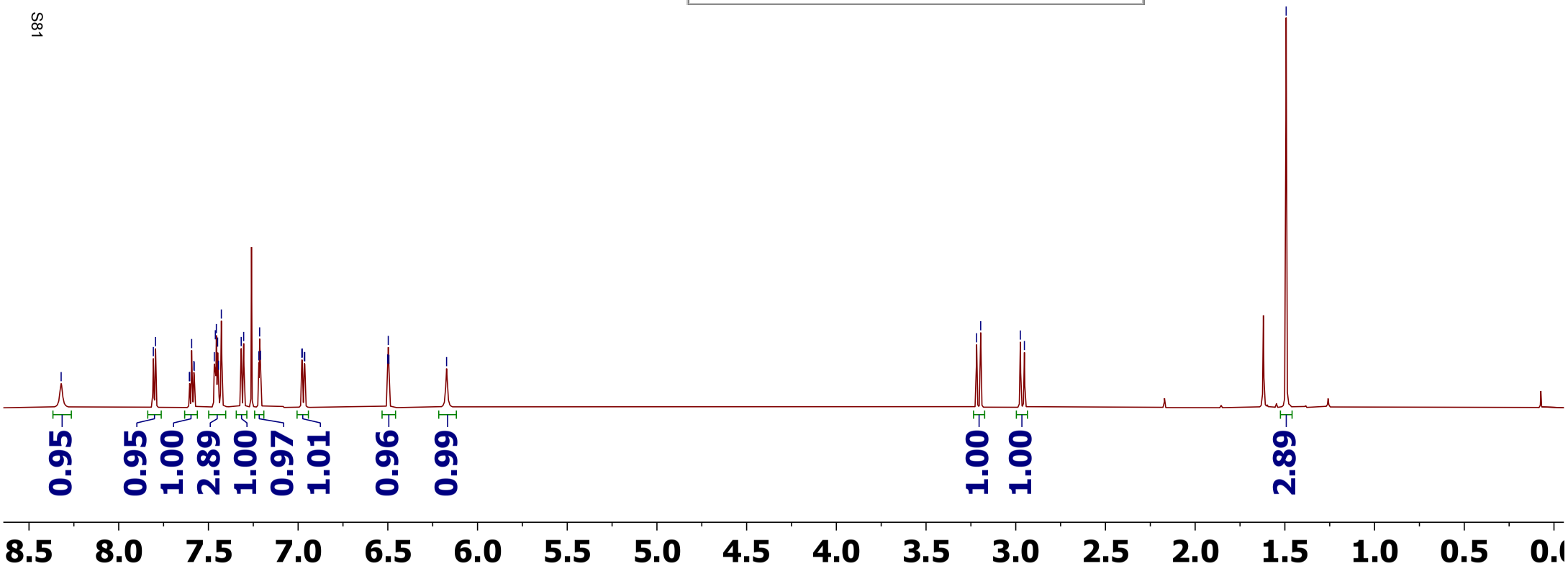
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

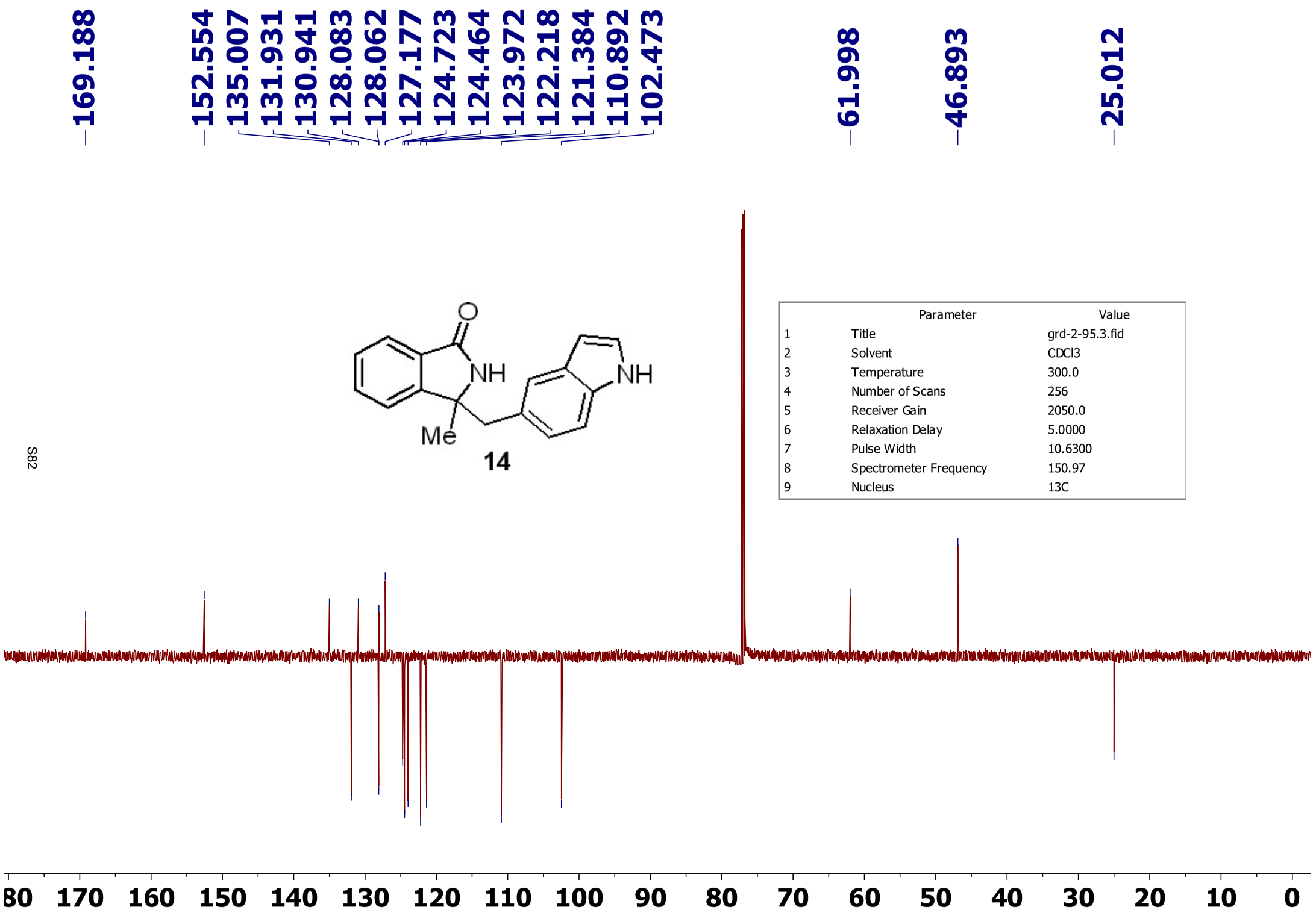




14

	Parameter	Value
1	Title	grd-2-95.2.fid
2	Solvent	CDCl3
3	Temperature	300.0
4	Number of Scans	16
5	Receiver Gain	144.0
6	Relaxation Delay	1.0000
7	Pulse Width	10.5000
8	Spectrometer Frequency	600.32
9	Nucleus	1H

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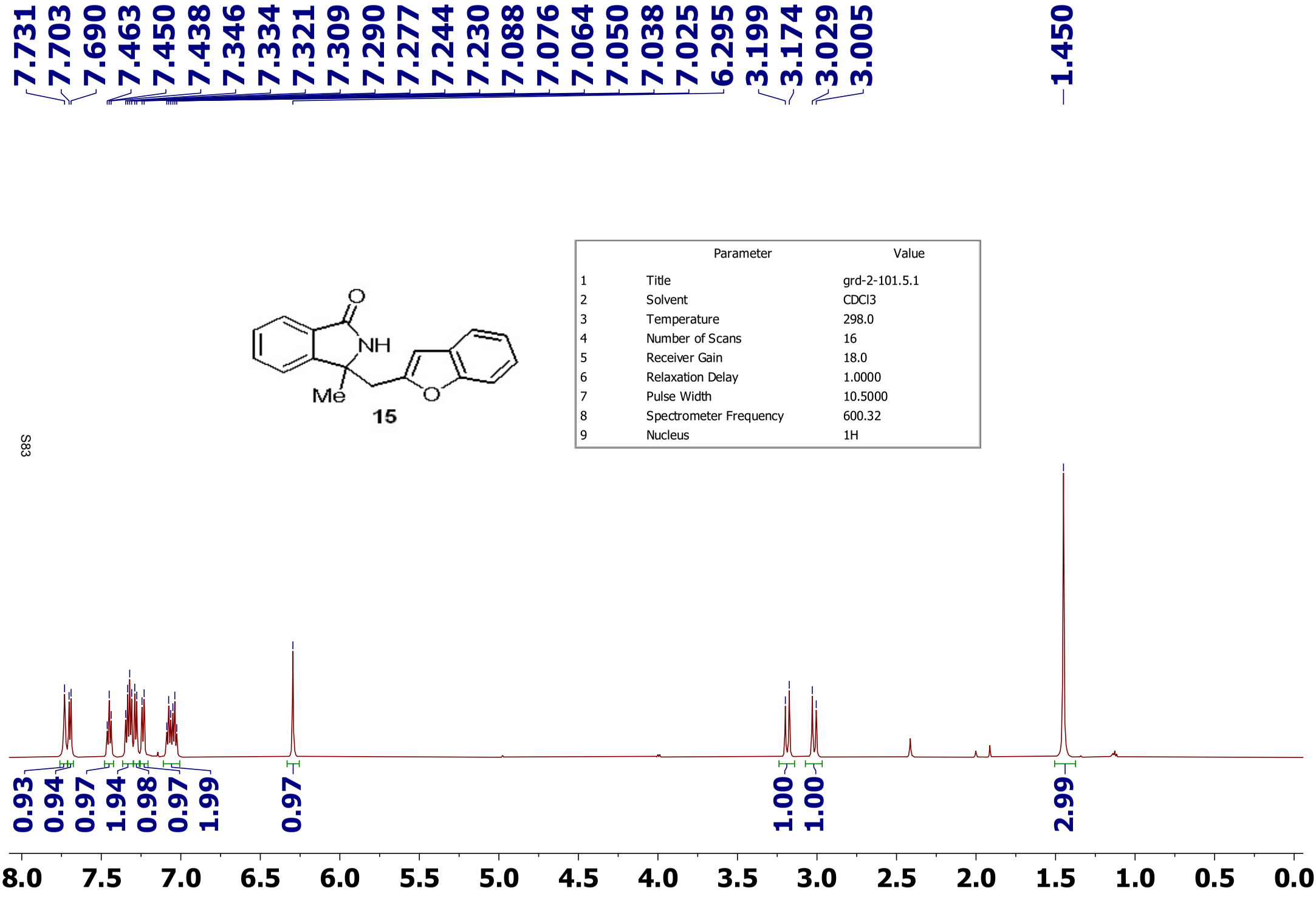
130.941
128.083
128.062
127.177
124.723
124.464
123.972
122.218
121.384
110.892
102.473
61.998
46.893
25.012
O
NH
NH
Me
14
S82
Parameter Value
1 Title grd-2-95.3.fid
2 Solvent CDCl3
3 Temperature 300.0
4 Number of Scans 256
5 Receiver Gain 2050.0
6 Relaxation Delay 5.0000
7 Pulse Width 10.6300
8 Spectrometer Frequency 150.97
9 Nucleus 13C
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0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7.703
7.690
7.463
7.450
7.438
7.346
7.334
7.321
7.309
7.290
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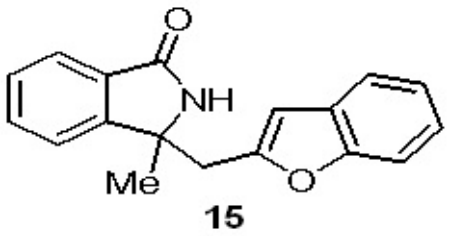
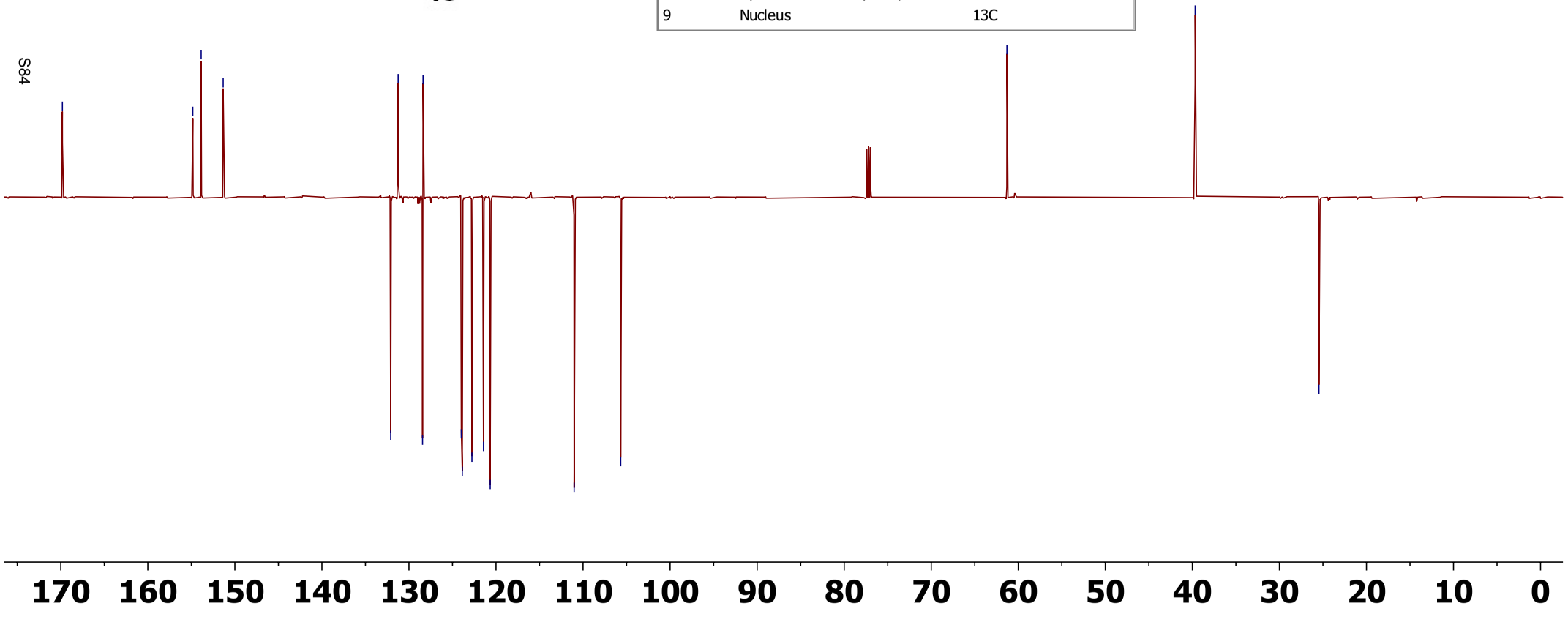
7.244
7.230
7.088
7.076
7.064
7.050
7.038
7.025
6.295
3.199
3.174
3.029
3.005
1.450
NH
Me
O
O
15
Parameter Value
1 Title grd-2-101.5.1
2 Solvent CDCl3
3 Temperature 298.0
4 Number of Scans 16
5 Receiver Gain 18.0
6 Relaxation Delay 1.0000
7 Pulse Width 10.5000
8 Spectrometer Frequency 600.32
9 Nucleus 1H
S83
0.93
0.94
0.97
1.94
0.98
0.97
1.99
0.97
1.00
1.00
2.99
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

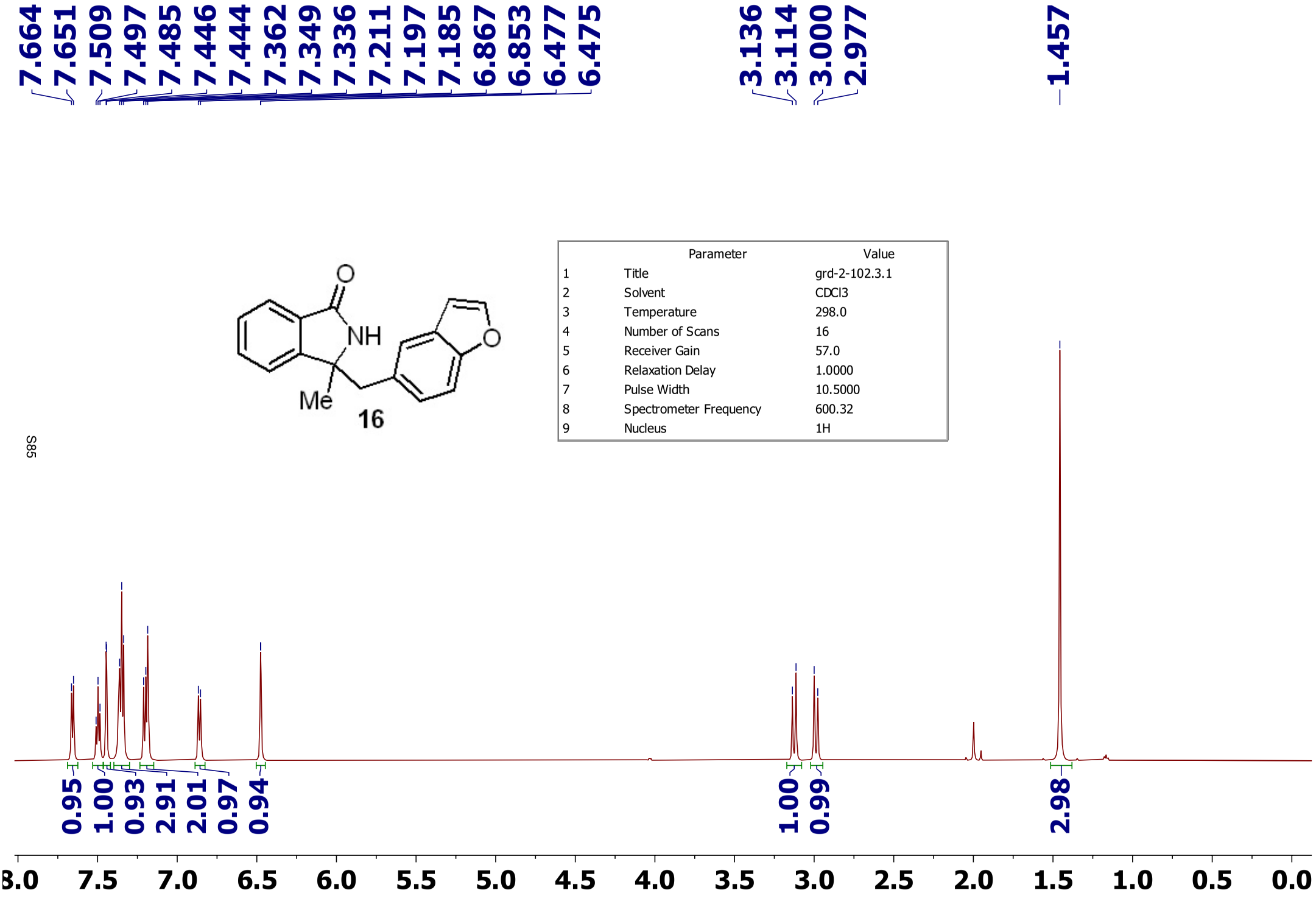
169.82
154.83
153.87
151.34
132.10
131.24
128.44
128.38
123.98
123.87
122.77
121.44
120.66
111.02
105.67
61.31
39.68
25.46



15

	Parameter	Value
1	Title	grd-2-101.6.1
2	Solvent	CDCl3
3	Temperature	298.0
4	Number of Scans	256
5	Receiver Gain	2050.0
6	Relaxation Delay	5.0000
7	Pulse Width	10.6300
8	Spectrometer Frequency	150.95
9	Nucleus	13C

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7.509
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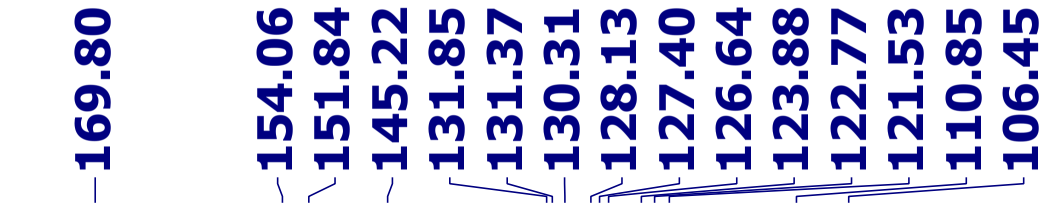
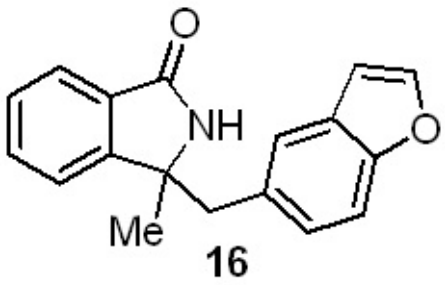
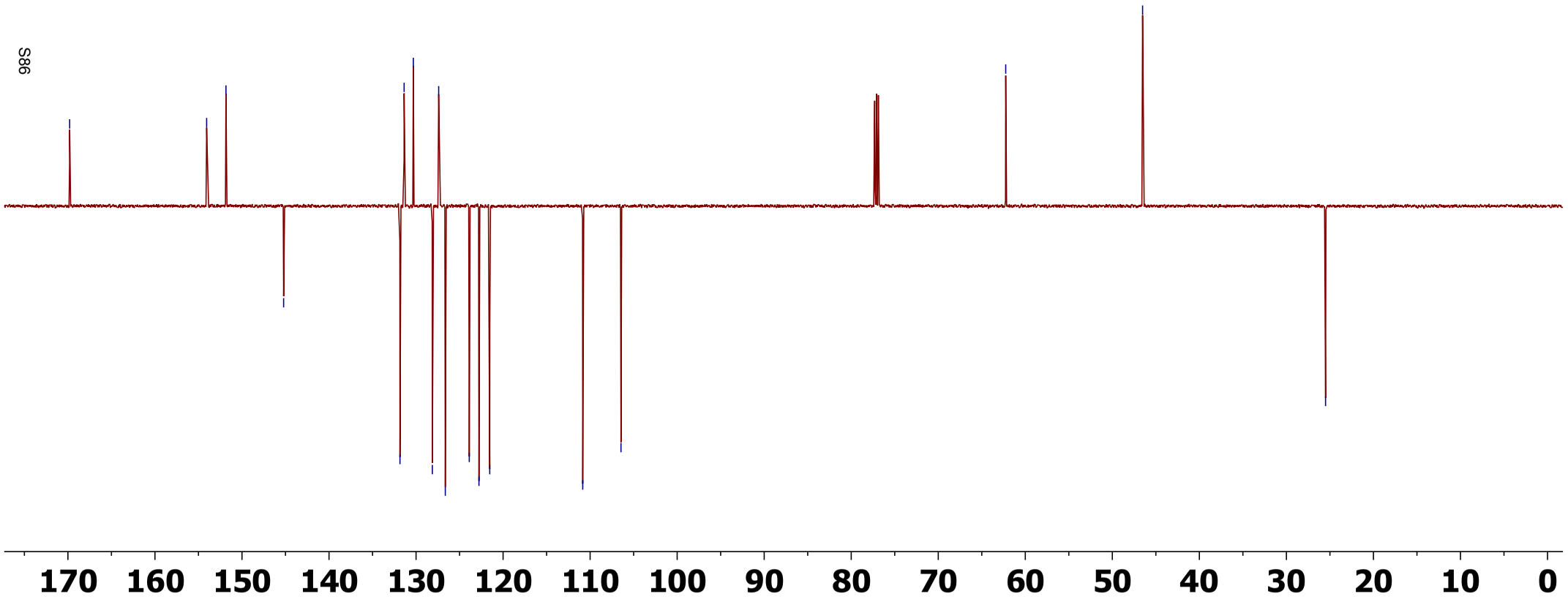
7.444
7.362
7.349
7.336
7.211
7.197
7.185
6.867
6.853
6.477
6.475
3.136
3.114
3.000
2.977
1.457
O
NH
O
Me
16
Parameter Value
1 Title grd-2-102.3.1
2 Solvent CDCl3
3 Temperature 298.0
4 Number of Scans 16
5 Receiver Gain 57.0
6 Relaxation Delay 1.0000
7 Pulse Width 10.5000
8 Spectrometer Frequency 600.32
9 Nucleus 1H
S85
0.95
1.00
0.93
2.91
2.01
0.97
0.94
1.00
0.99
2.98
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

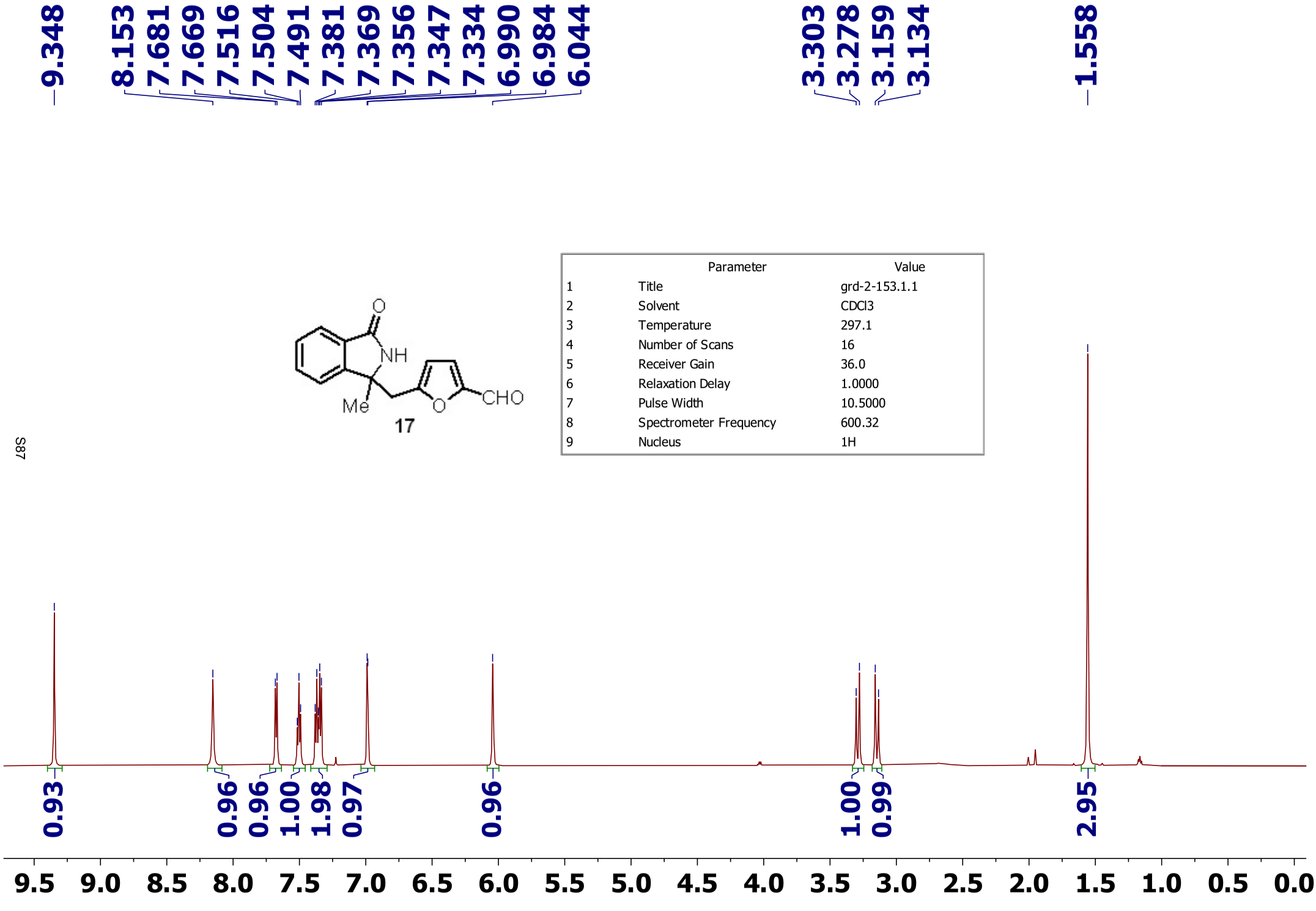




16

	Parameter	Value
1	Title	grd-2-102.4.1
2	Solvent	CDCl3
3	Temperature	298.0
4	Number of Scans	256
5	Receiver Gain	2050.0
6	Relaxation Delay	5.0000
7	Pulse Width	10.6300
8	Spectrometer Frequency	150.95
9	Nucleus	13C

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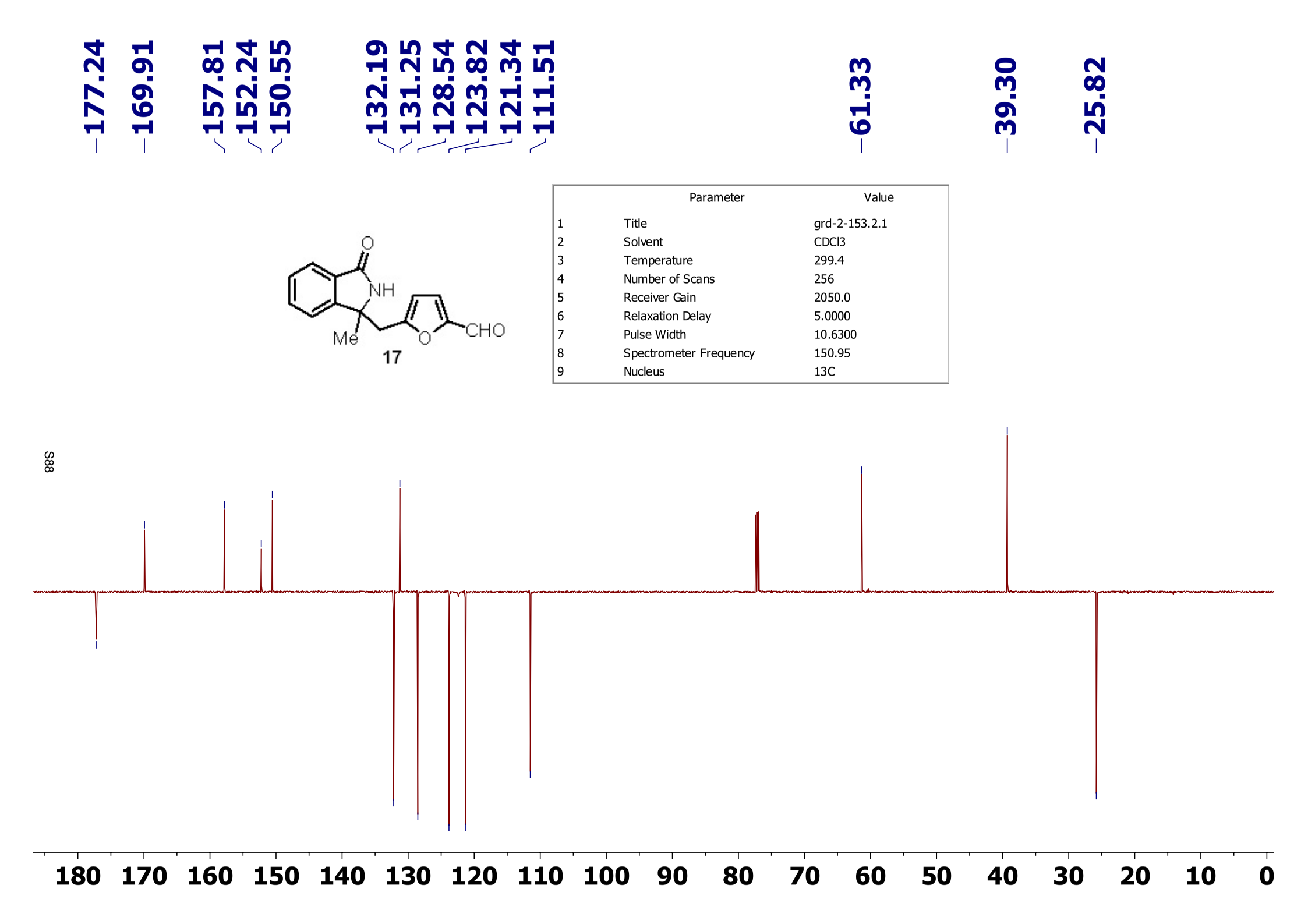
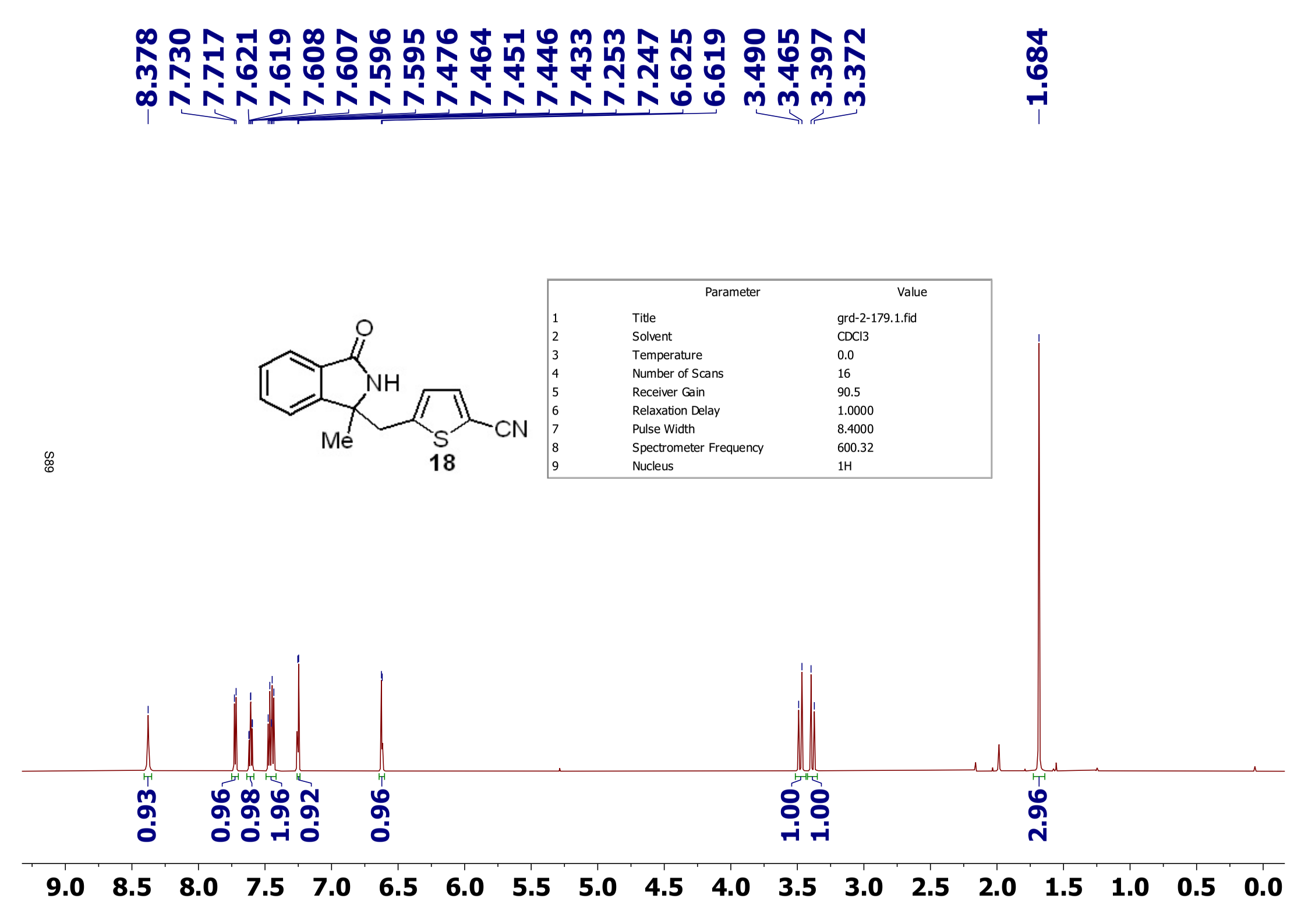
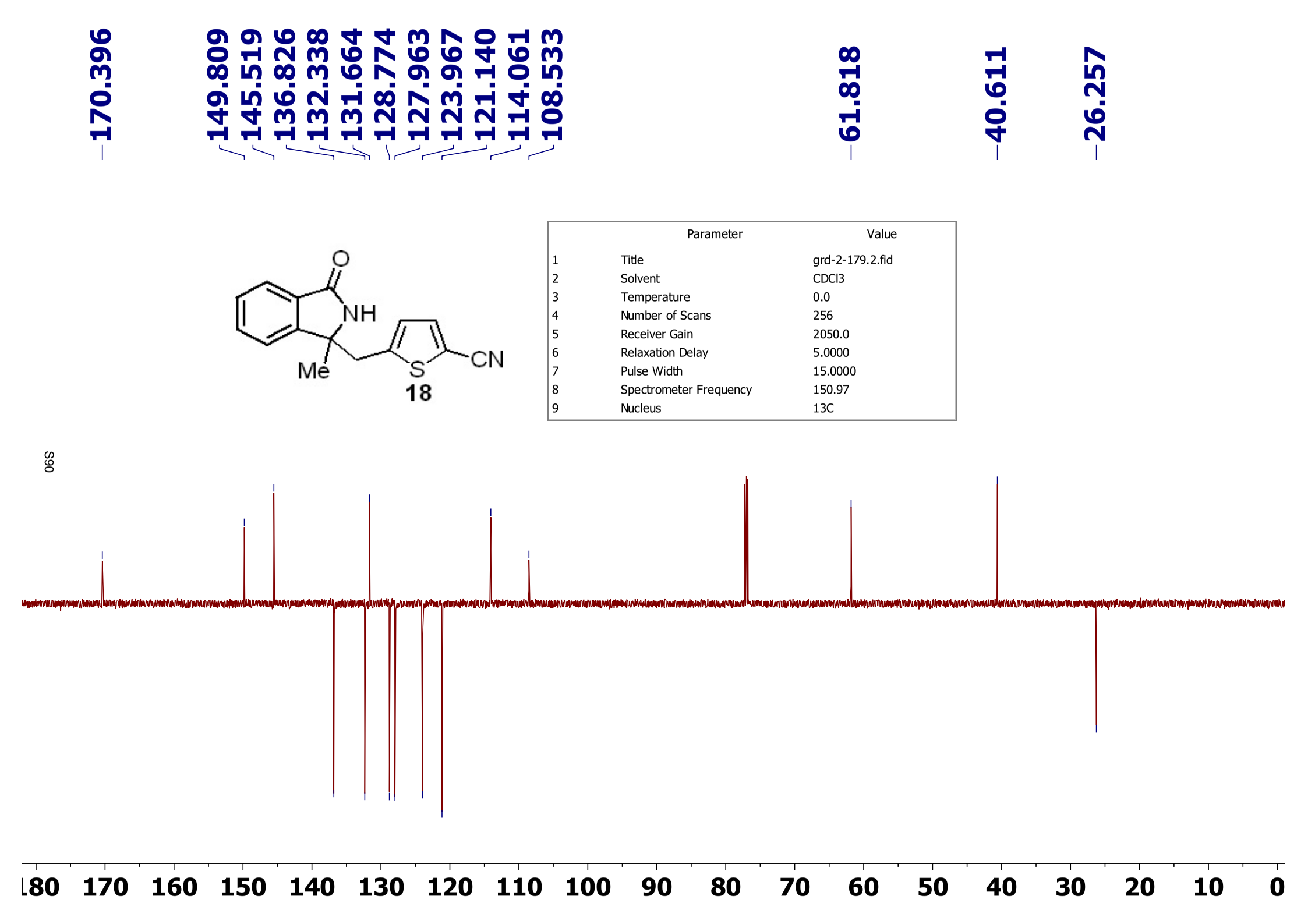
7.504
7.491
7.381
7.369
7.356
7.347
7.334
6.990
6.984
6.044
3.303
3.278
3.159
3.134
1.558
O
NH
Me
O
CHO
17
Parameter Value
1 Title grd-2-153.1.1
2 Solvent CDCl3
3 Temperature 297.1
4 Number of Scans 16
5 Receiver Gain 36.0
6 Relaxation Delay 1.0000
7 Pulse Width 10.5000
8 Spectrometer Frequency 600.32
9 Nucleus 1H
S87
0.93
0.96
0.96
1.00
1.98
0.97
0.96
1.00
0.99
2.95
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



	Parameter	Value
1	Title	grd-2-153.2.1
2	Solvent	CDCl3
3	Temperature	299.4
4	Number of Scans	256
5	Receiver Gain	2050.0
6	Relaxation Delay	5.0000
7	Pulse Width	10.6300
8	Spectrometer Frequency	150.95
9	Nucleus	13C



	Parameter	Value
1	Title	grd-2-179.1.fid
2	Solvent	CDCl3
3	Temperature	0.0
4	Number of Scans	16
5	Receiver Gain	90.5
6	Relaxation Delay	1.0000
7	Pulse Width	8.4000
8	Spectrometer Frequency	600.32
9	Nucleus	1H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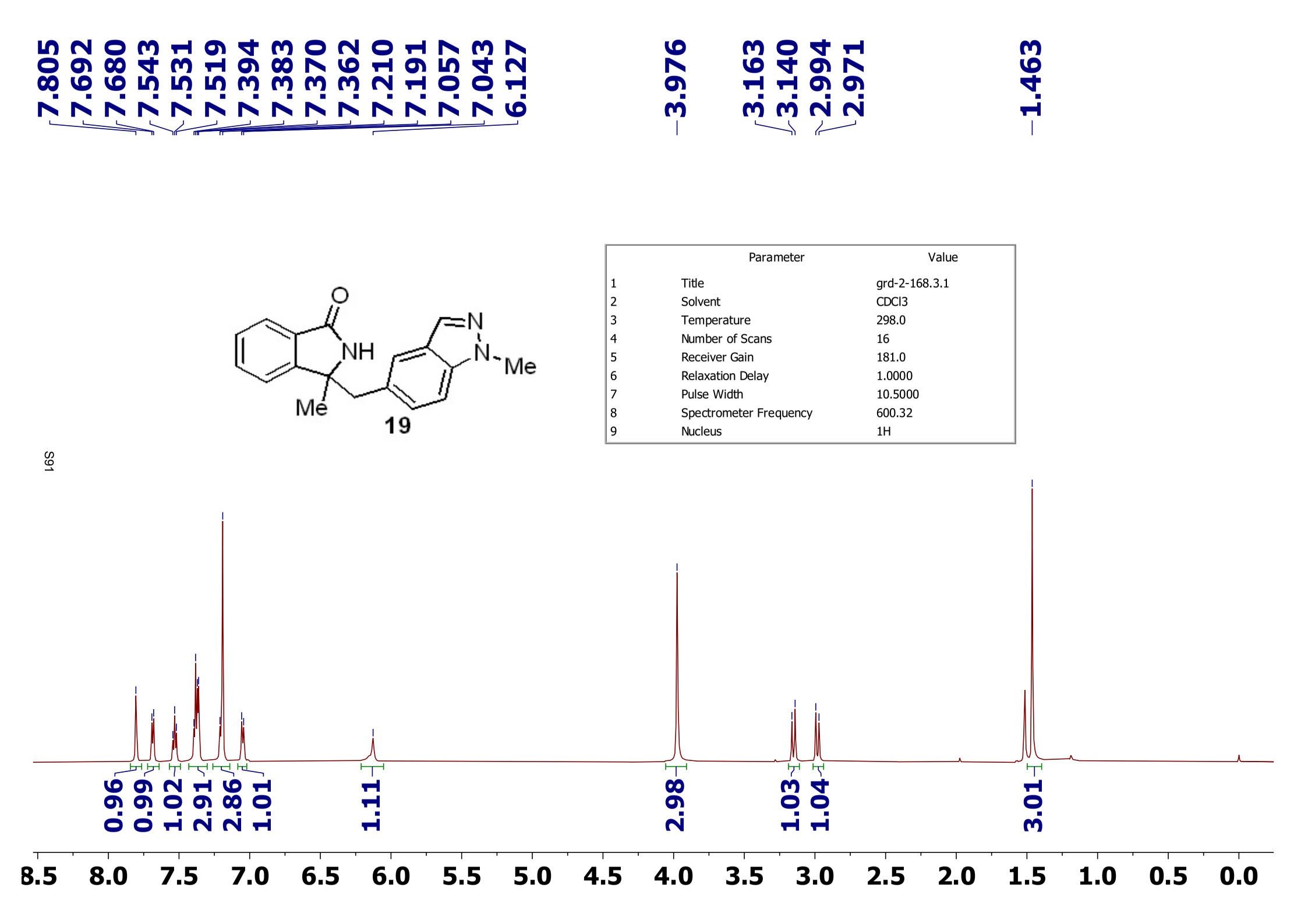
127.963
123.967
121.140
114.061
108.533
61.818
40.611
26.257
O
NH
Me
S
CN
18
Parameter
Value
1 Title grd-2-179.2.fid
2 Solvent CDCl3
3 Temperature 0.0
4 Number of Scans 256
5 Receiver Gain 2050.0
6 Relaxation Delay 5.0000
7 Pulse Width 15.0000
8 Spectrometer Frequency 150.97
9 Nucleus 13C
S90
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0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7.394
7.383
7.370
7.362
7.210
7.191
7.057
7.043
6.127
3.976
3.163
3.140
2.994
2.971
1.463
O
NH
N
N
Me
Me
19
Parameter
Value
1 Title grd-2-168.3.1
2 Solvent CDCl3
3 Temperature 298.0
4 Number of Scans 16
5 Receiver Gain 181.0
6 Relaxation Delay 1.0000
7 Pulse Width 10.5000
8 Spectrometer Frequency 600.32
9 Nucleus 1H
S91
0.96
0.99
1.02
2.91
2.86
1.01
1.11
2.98
1.03
1.04
3.01
8.5
8.0
7.5
7.0
6.5
6.0
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4.0
3.5
3.0
2.5
2.0
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1.0
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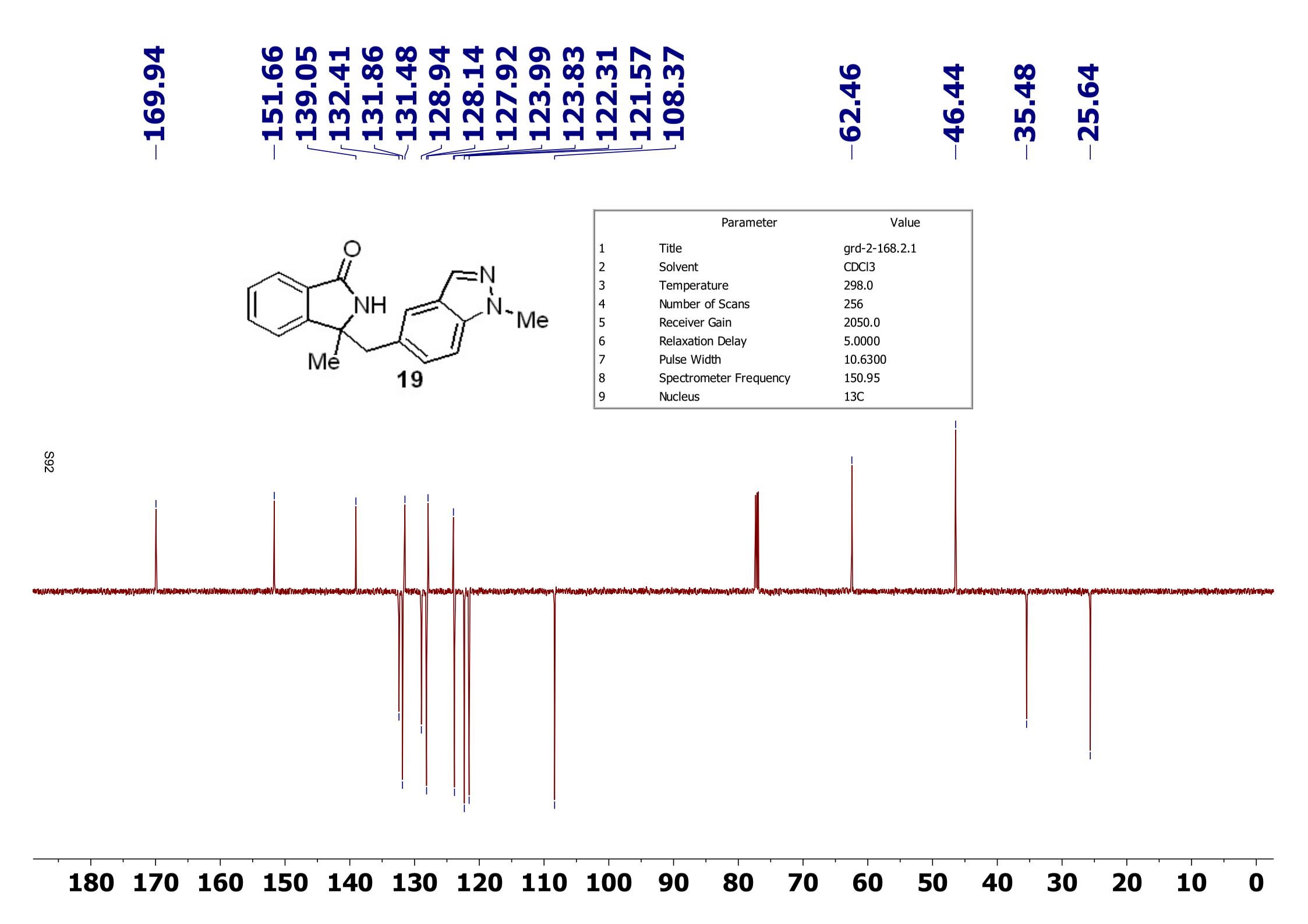
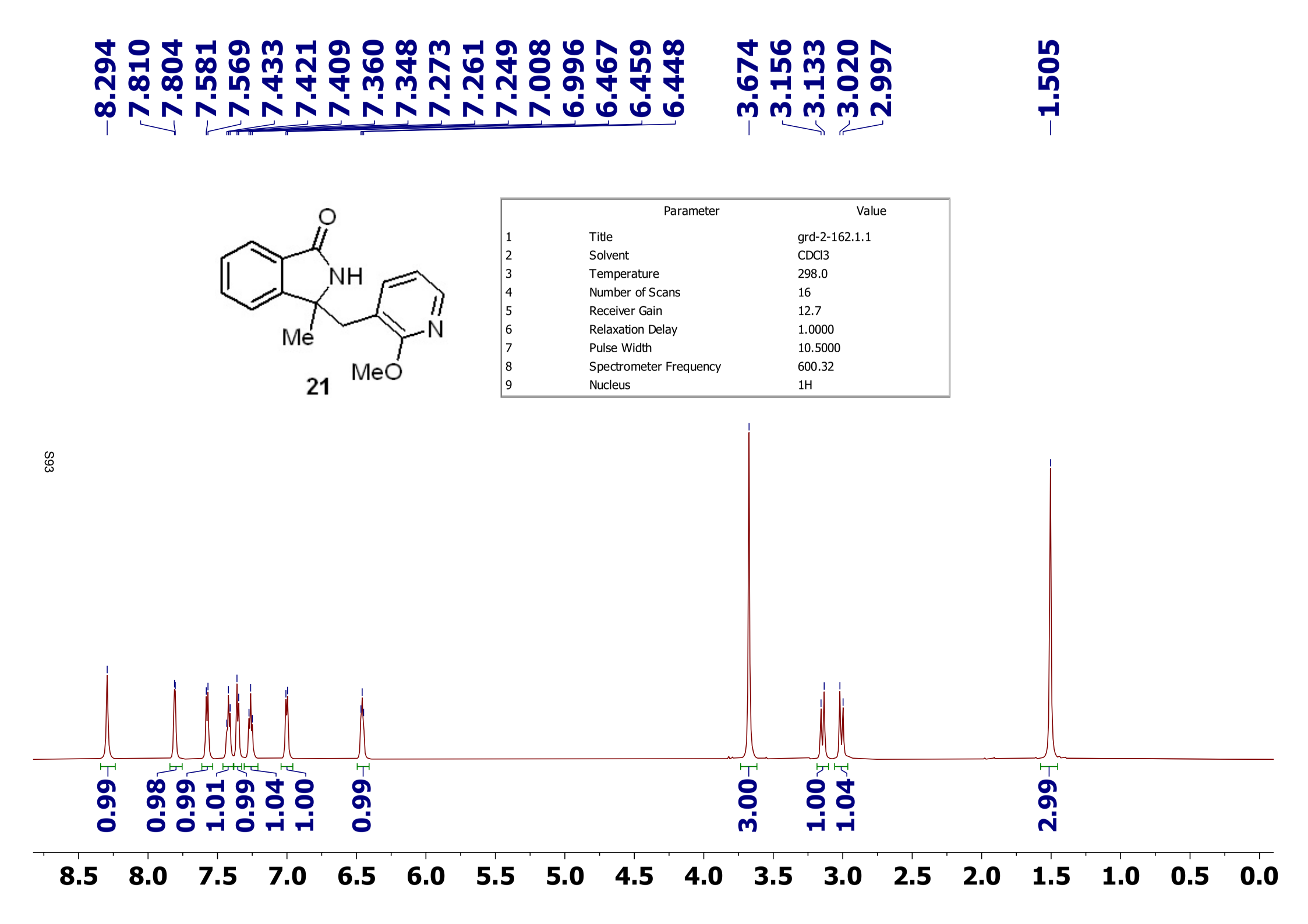
0.0



	Parameter	Value
1	Title	grd-2-168.2.1
2	Solvent	CDCl3
3	Temperature	298.0
4	Number of Scans	256
5	Receiver Gain	2050.0
6	Relaxation Delay	5.0000
7	Pulse Width	10.6300
8	Spectrometer Frequency	150.95
9	Nucleus	13C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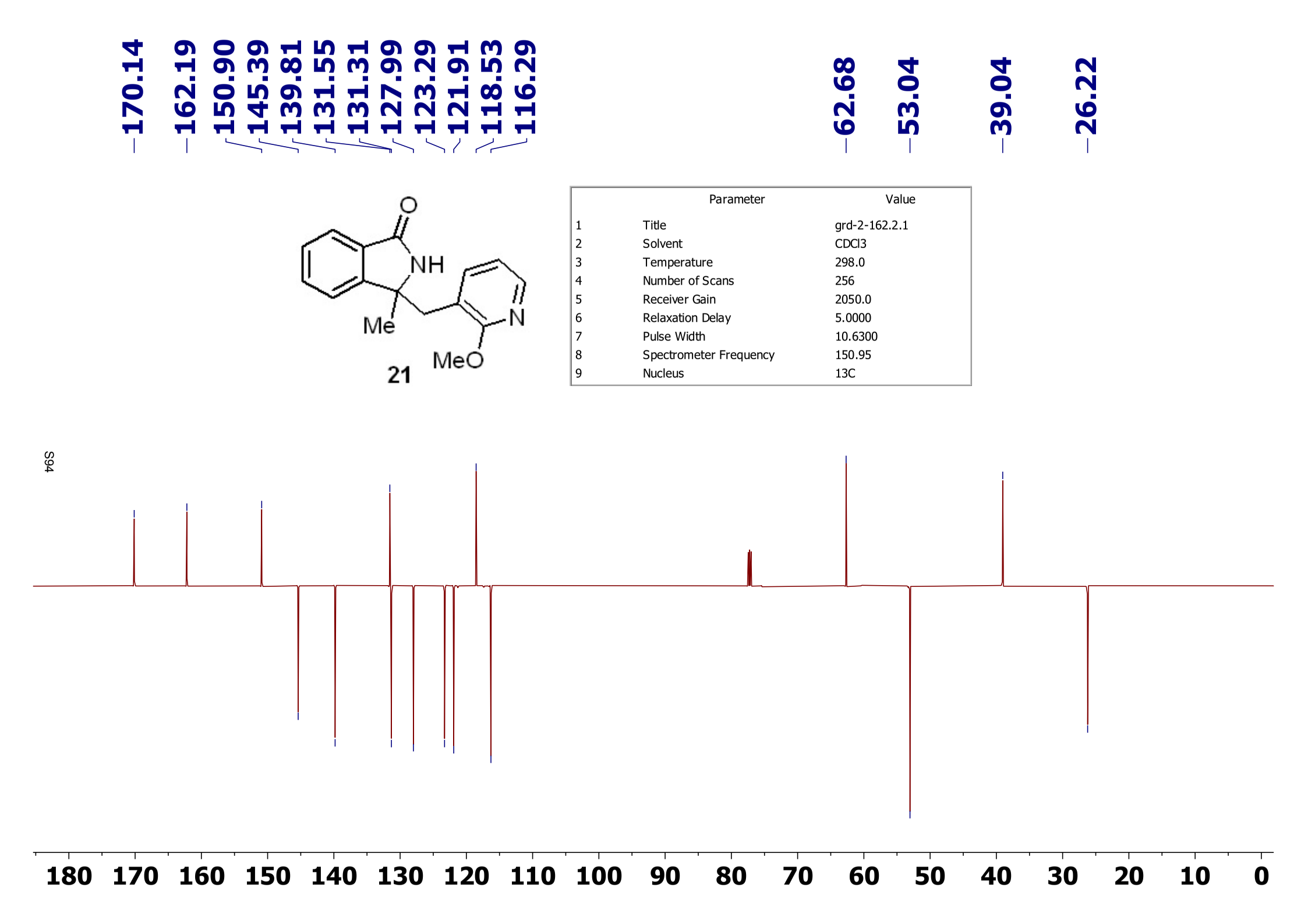
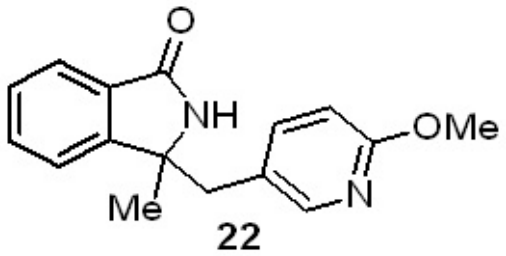
7.348
7.273
7.261
7.249
7.008
6.996
6.467
6.459
6.448
3.674
3.156
3.133
3.020
2.997
1.505
O
NH
Me
N
MeO
21
Parameter
Value
1 Title grd-2-162.1.1
2 Solvent CDCl3
3 Temperature 298.0
4 Number of Scans 16
5 Receiver Gain 12.7
6 Relaxation Delay 1.0000
7 Pulse Width 10.5000
8 Spectrometer Frequency 600.32
9 Nucleus 1H
S93
0.99
0.98
0.99
1.01
0.99
1.04
1.00
0.99
3.00
1.00
1.04
2.99
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



	Parameter	Value
1	Title	grd-2-162.2.1
2	Solvent	CDCl3
3	Temperature	298.0
4	Number of Scans	256
5	Receiver Gain	2050.0
6	Relaxation Delay	5.0000
7	Pulse Width	10.6300
8	Spectrometer Frequency	150.95
9	Nucleus	13C

7.794 7.750 7.684 7.649 7.636 7.527 7.514 7.502 7.358 7.346 7.120 7.106 6.456 6.442

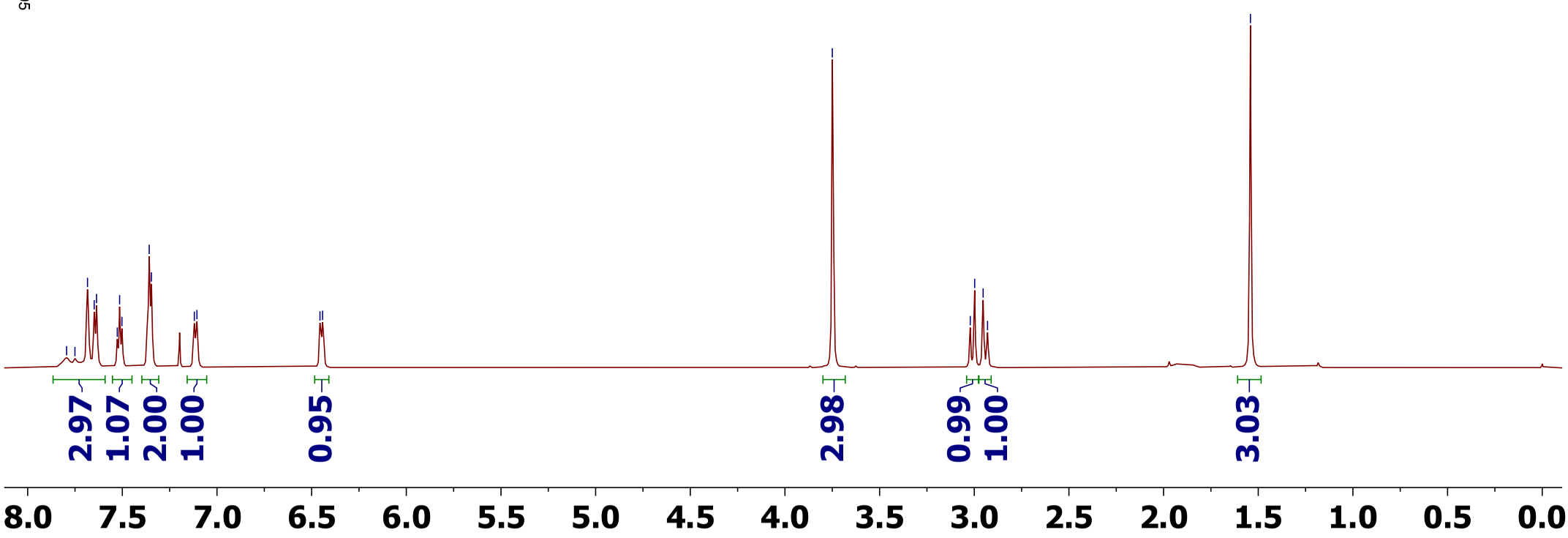
3.750 3.021 2.998 2.953 2.930 1.541



22

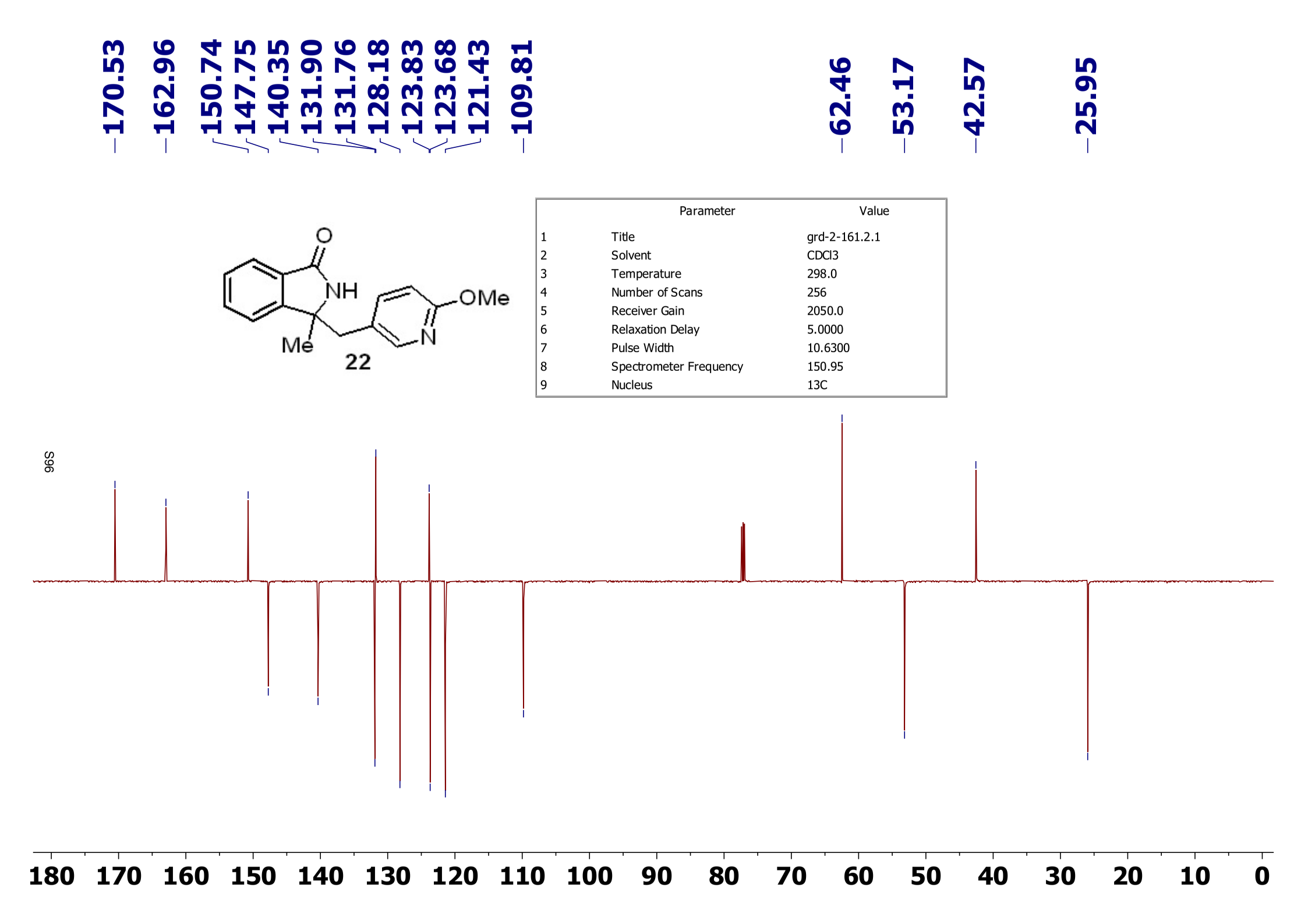
	Parameter	Value
1	Title	grd-2-161.4.1
2	Solvent	CDCl3
3	Temperature	298.0
4	Number of Scans	16
5	Receiver Gain	57.0
6	Relaxation Delay	1.0000
7	Pulse Width	10.5000
8	Spectrometer Frequency	600.32
9	Nucleus	1H

S95



2.97 1.07 2.00 1.00 0.95 2.98 0.99 1.00 3.03

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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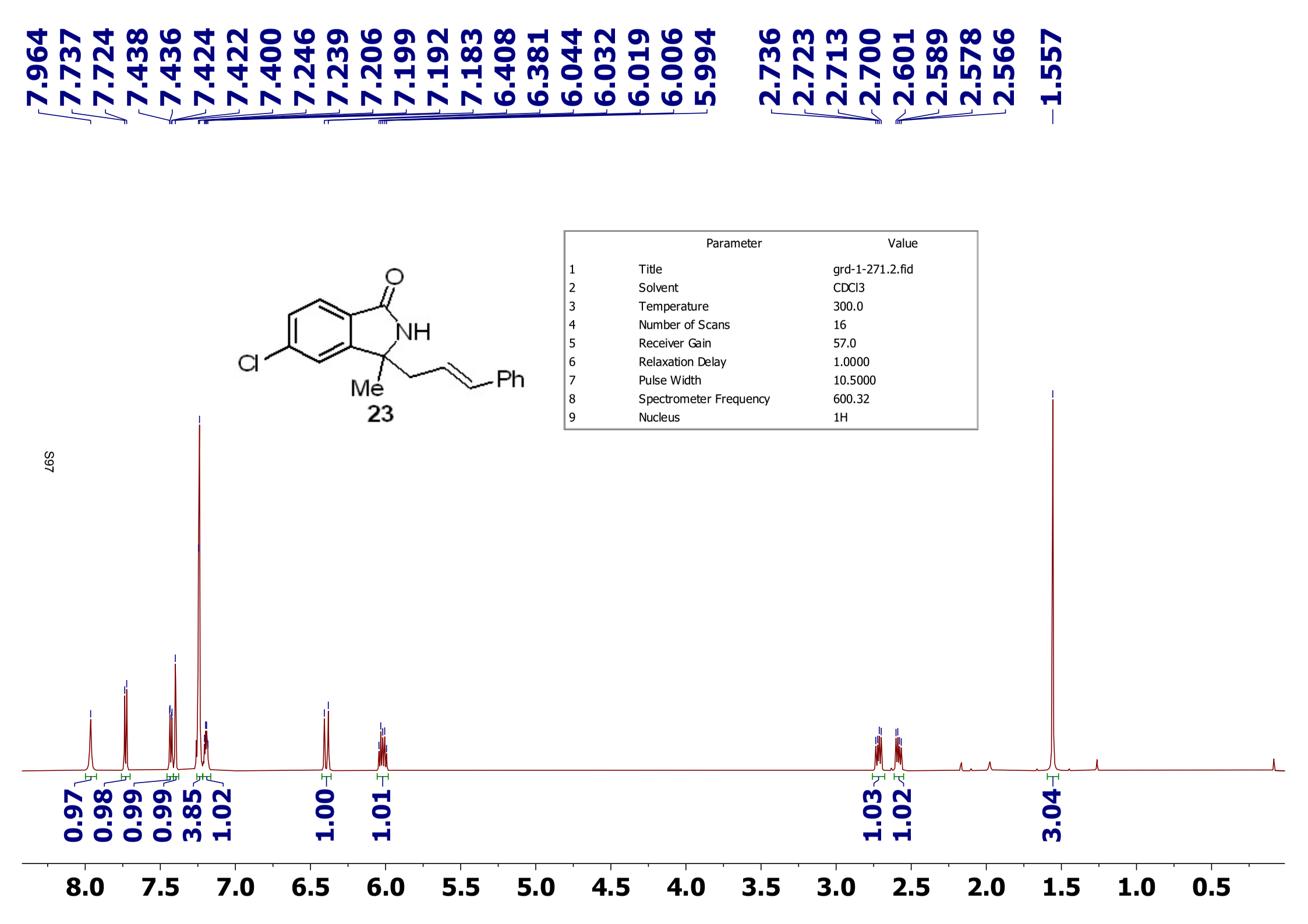
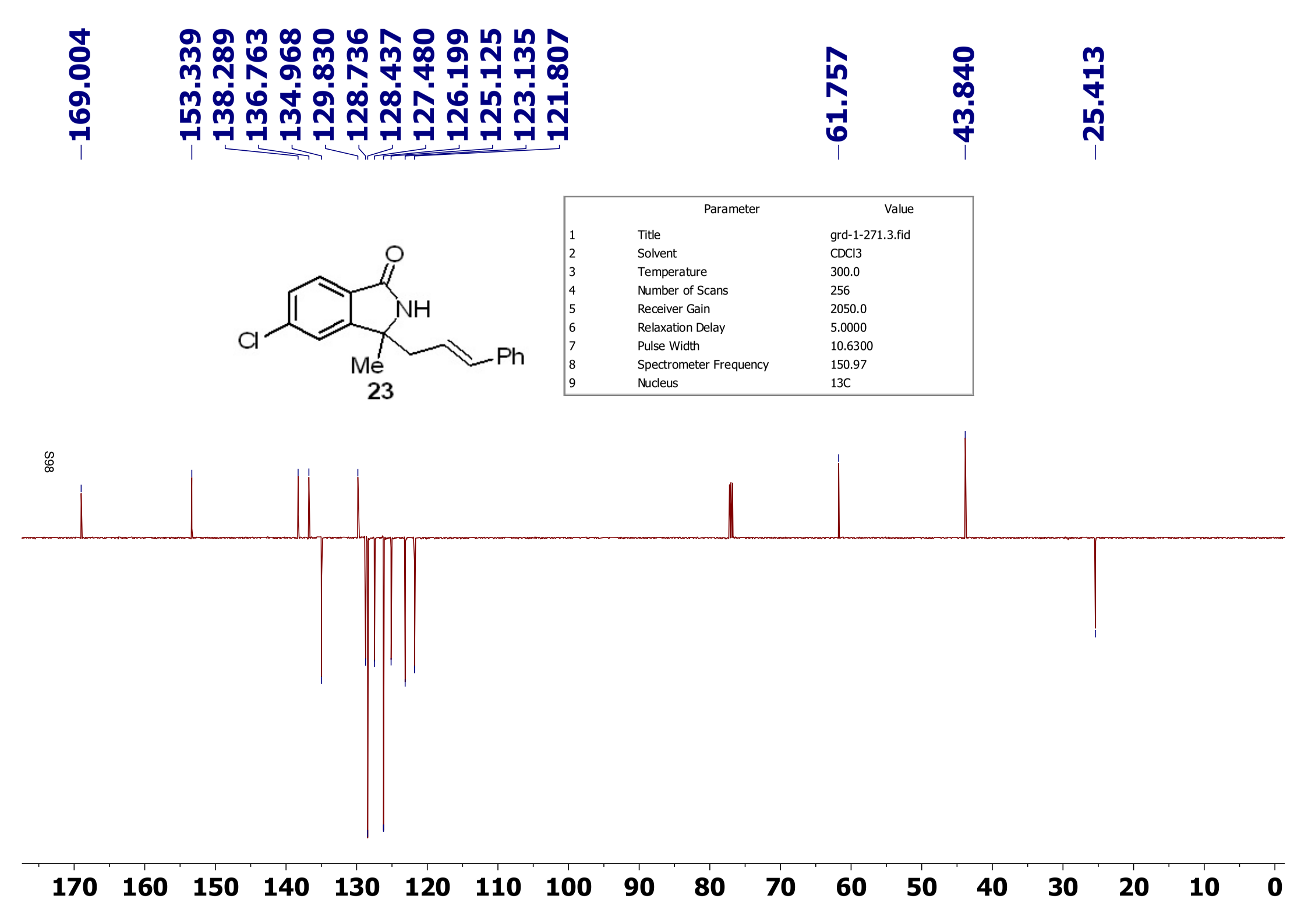
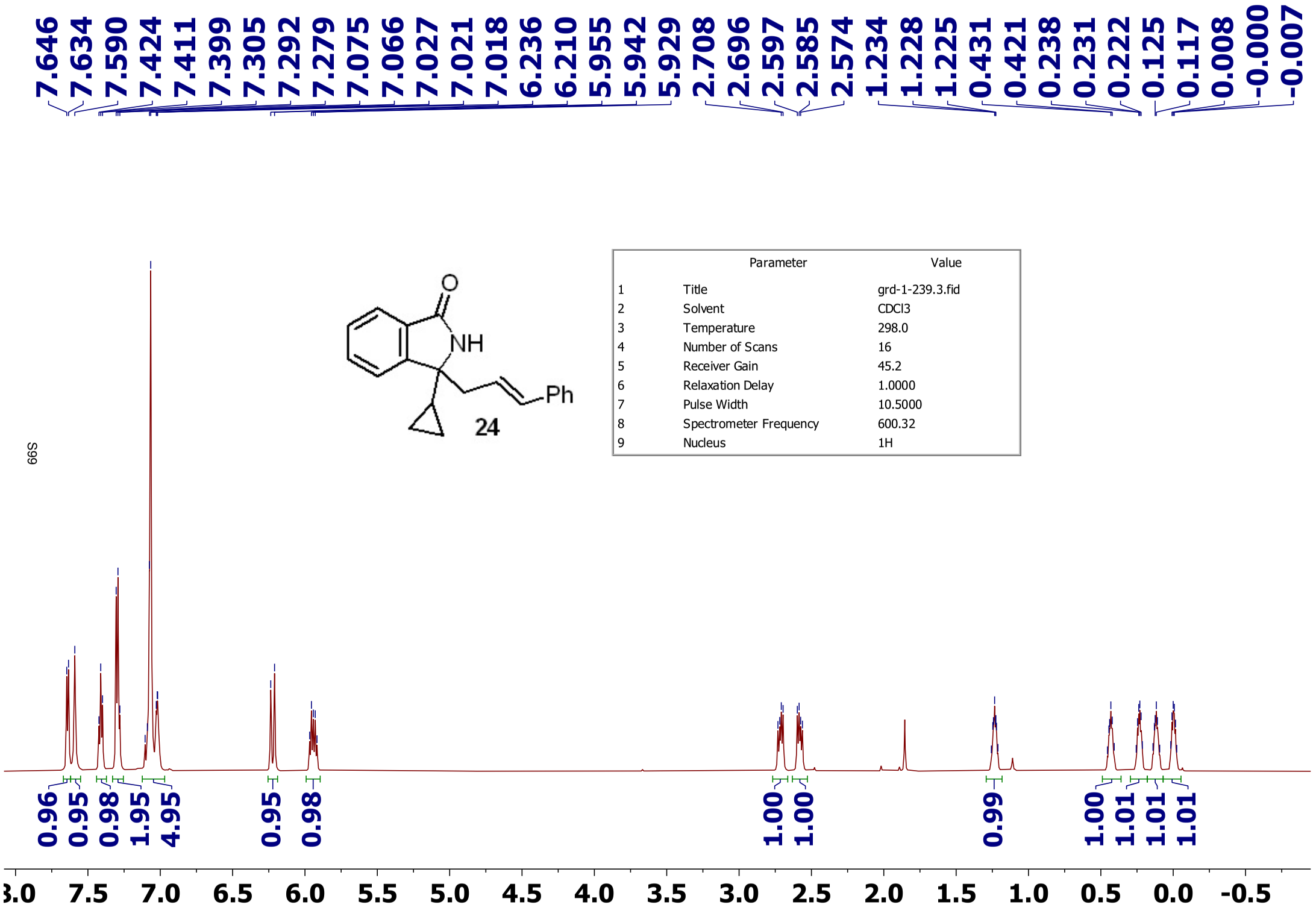
150.74
147.75
140.35
131.90
131.76
128.18
123.83
123.68
121.43
109.81
62.46
53.17
42.57
25.95
O
NH
OMe
N
Me
22
Parameter Value
1 Title grd-2-161.2.1
2 Solvent CDCl3
3 Temperature 298.0
4 Number of Scans 256
5 Receiver Gain 2050.0
6 Relaxation Delay 5.0000
7 Pulse Width 10.6300
8 Spectrometer Frequency 150.95
9 Nucleus 13C
S96
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0


7.964
7.737
7.724
7.438
7.436
7.424
7.422
7.400
7.246
7.239
7.206
7.199
7.192
7.183
6.408
6.381
6.044
6.032
6.019
6.006
5.994
2.736
2.723
2.713
2.700
2.601
2.589
2.578
2.566
1.557
O
NH
Cl
Me
Ph
23
Parameter Value
1 Title grd-1-271.2.fid
2 Solvent CDCl3
3 Temperature 300.0
4 Number of Scans 16
5 Receiver Gain 57.0
6 Relaxation Delay 1.0000
7 Pulse Width 10.5000
8 Spectrometer Frequency 600.32
9 Nucleus 1H
S97
0.97
0.98
0.99
0.99
3.85
1.02
1.00
1.01
1.03
1.02
3.04
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



	Parameter	Value
1	Title	grd-1-271.3.fid
2	Solvent	CDCl3
3	Temperature	300.0
4	Number of Scans	256
5	Receiver Gain	2050.0
6	Relaxation Delay	5.0000
7	Pulse Width	10.6300
8	Spectrometer Frequency	150.97
9	Nucleus	13C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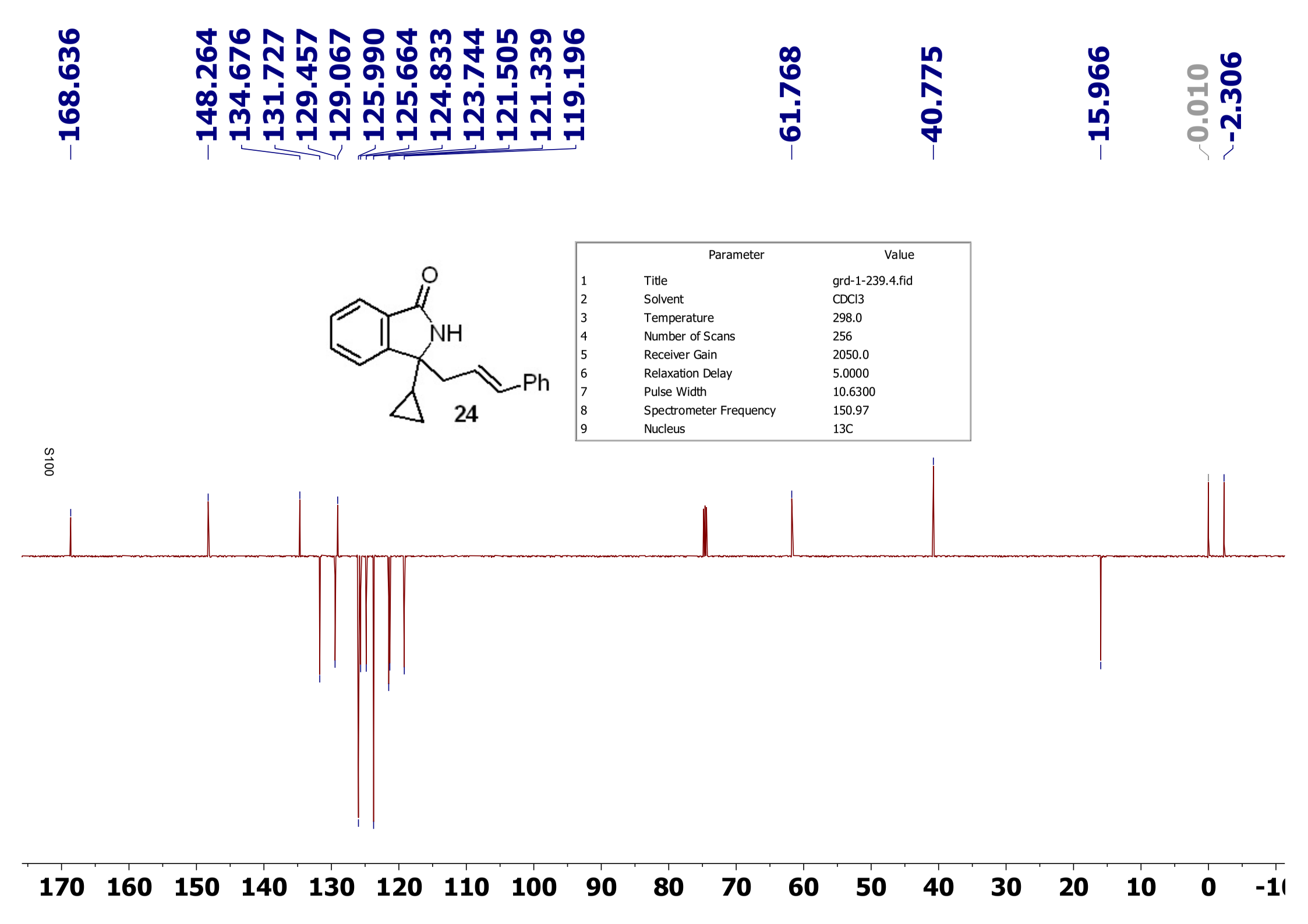
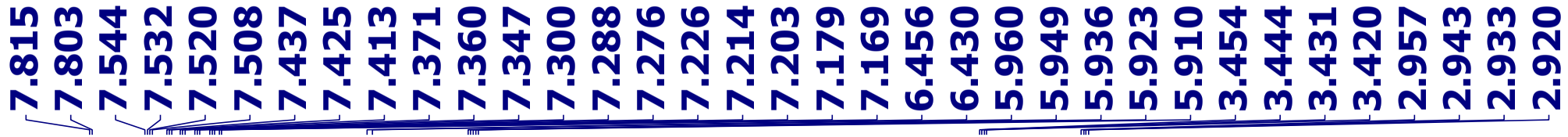
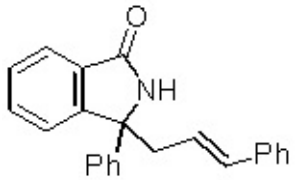
7.634
7.590
7.424
7.411
7.399
7.305
7.292
7.279
7.075
7.066
7.027
7.021
7.018
6.236
6.210
5.955
5.942
5.929
2.708
2.696
2.597
2.585
2.574
1.234
1.228
1.225
0.431
0.421
0.238
0.231
0.222
0.125
0.117
0.008
-0.000
-0.007
O
NH
Ph
24
Parameter Value
1 Title grd-1-239.3.fid
2 Solvent CDCl3
3 Temperature 298.0
4 Number of Scans 16
5 Receiver Gain 45.2
6 Relaxation Delay 1.0000
7 Pulse Width 10.5000
8 Spectrometer Frequency 600.32
9 Nucleus 1H
S99
0.96
0.95
0.98
1.95
4.95
0.95
0.98
1.00
1.00
0.99
1.00
1.01
1.01
1.01
3.0
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
-0.5



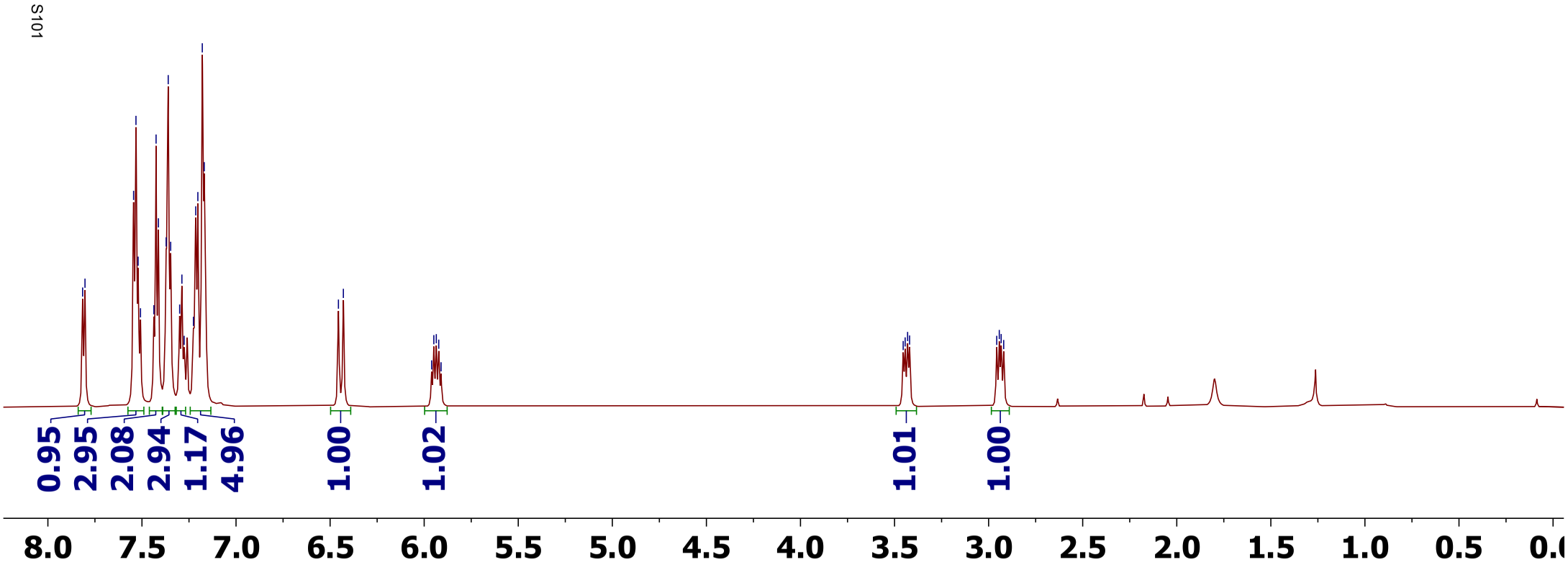
	Parameter	Value
1	Title	grd-1-239.4.fid
2	Solvent	CDCl3
3	Temperature	298.0
4	Number of Scans	256
5	Receiver Gain	2050.0
6	Relaxation Delay	5.0000
7	Pulse Width	10.6300
8	Spectrometer Frequency	150.97
9	Nucleus	13C

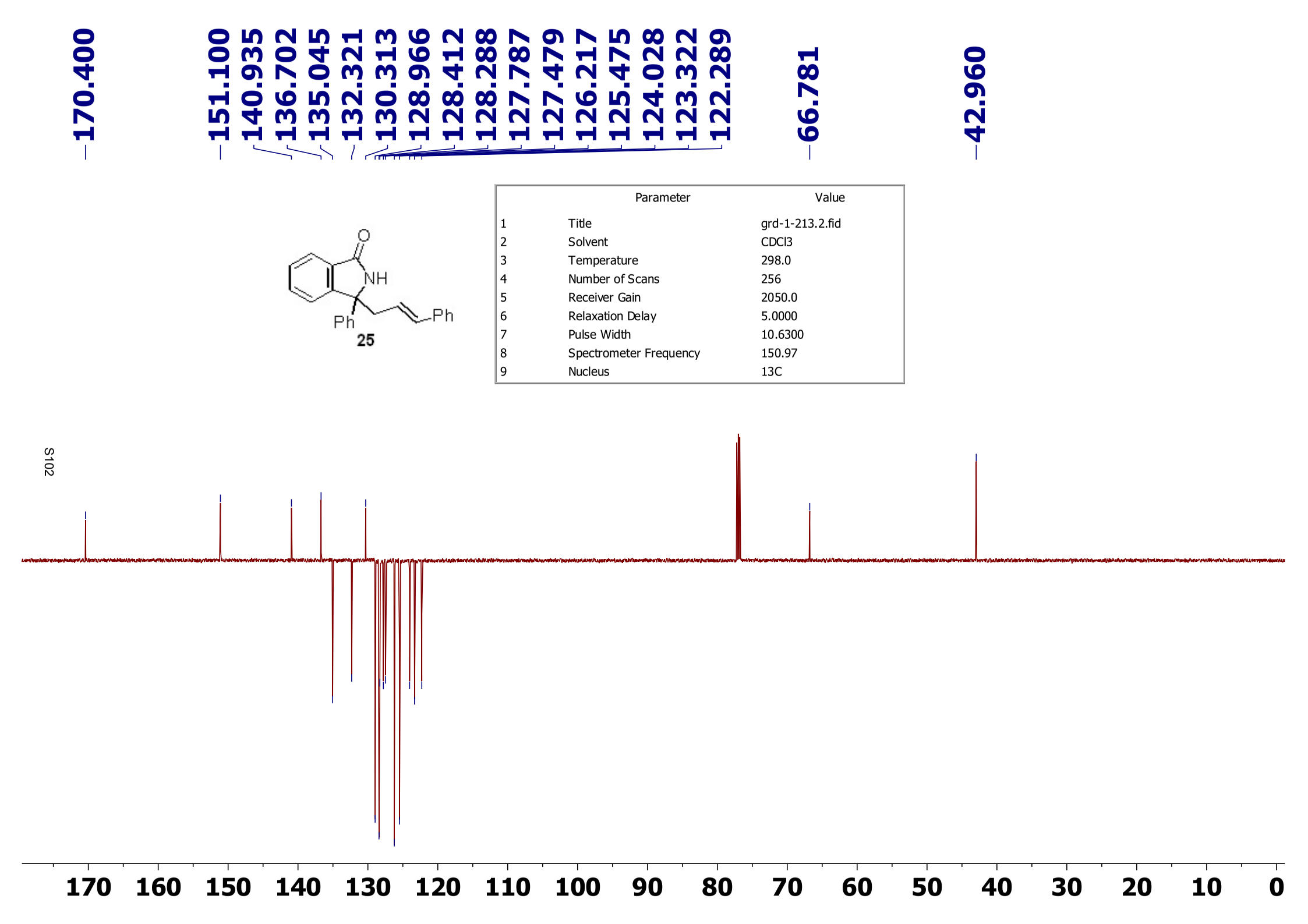




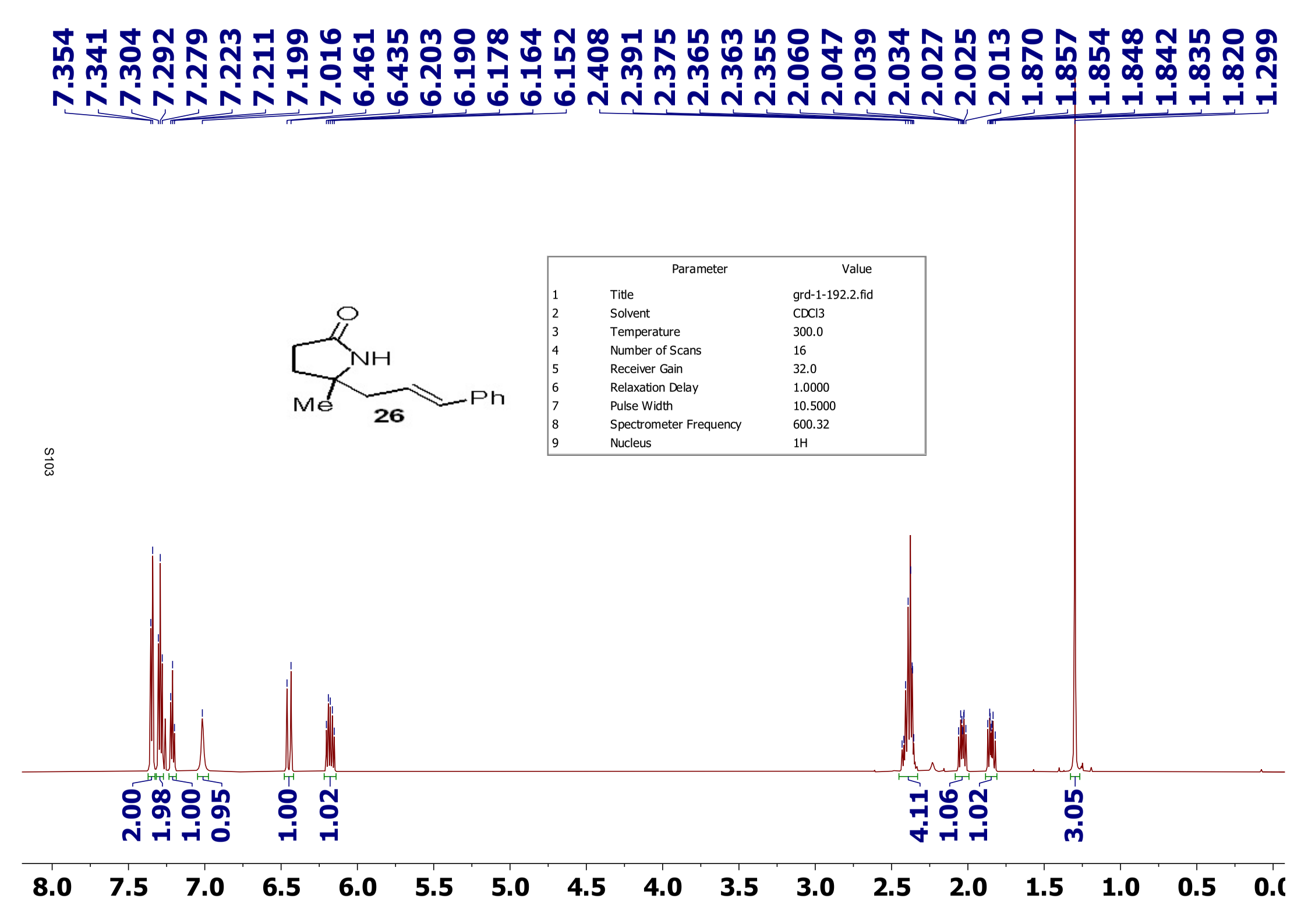
25

	Parameter	Value
1	Title	grd-1-213.1.fid
2	Solvent	CDCl3
3	Temperature	298.0
4	Number of Scans	16
5	Receiver Gain	57.0
6	Relaxation Delay	1.0000
7	Pulse Width	10.5000
8	Spectrometer Frequency	600.32
9	Nucleus	1H





	Parameter	Value
1	Title	grd-1-213.2.fid
2	Solvent	CDCl3
3	Temperature	298.0
4	Number of Scans	256
5	Receiver Gain	2050.0
6	Relaxation Delay	5.0000
7	Pulse Width	10.6300
8	Spectrometer Frequency	150.97
9	Nucleus	13C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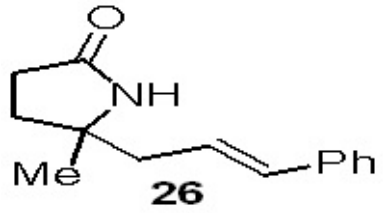
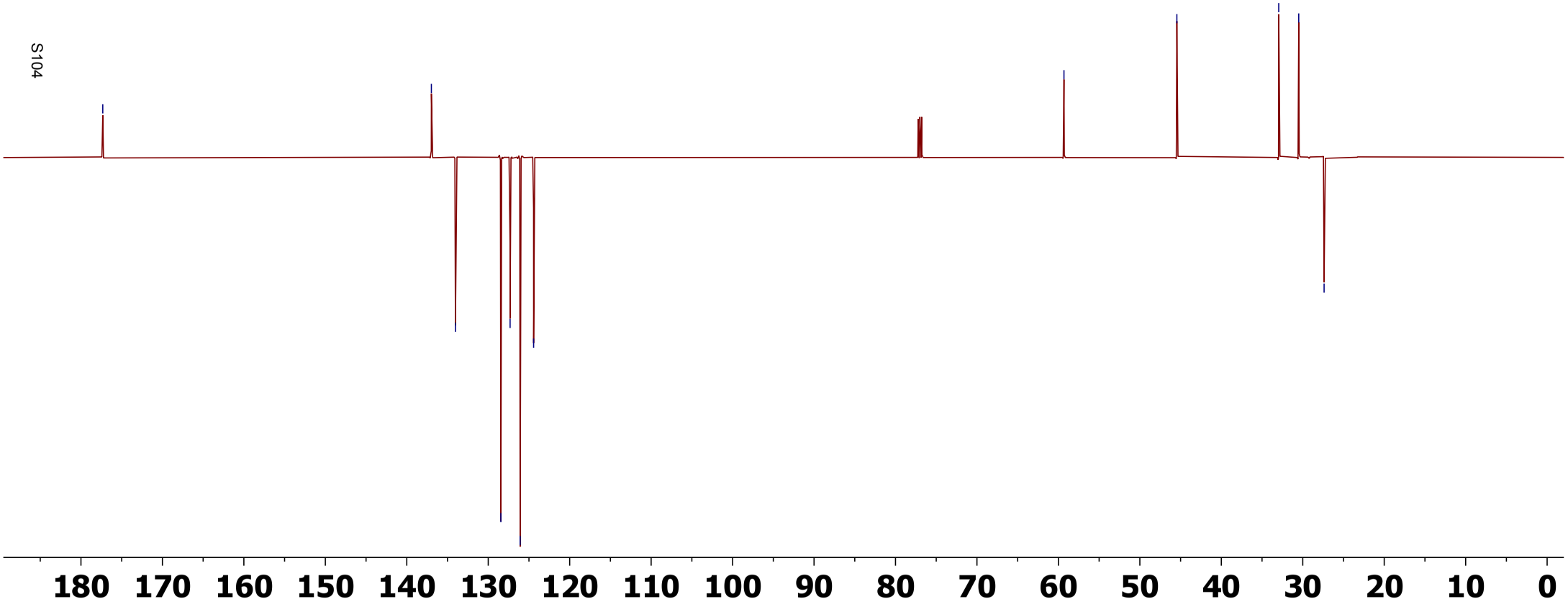
7.304
7.292
7.279
7.223
7.211
7.199
7.016
6.461
6.435
6.203
6.190
6.178
6.164
6.152
2.408
2.391
2.375
2.365
2.363
2.355
2.060
2.047
2.039
2.034
2.027
2.025
2.013
1.870
1.857
1.854
1.848
1.842
1.835
1.820
1.299
NH
Me
Ph
26
Parameter Value
1 Title grd-1-192.2.fid
2 Solvent CDCl3
3 Temperature 300.0
4 Number of Scans 16
5 Receiver Gain 32.0
6 Relaxation Delay 1.0000
7 Pulse Width 10.5000
8 Spectrometer Frequency 600.32
9 Nucleus 1H
S103
2.00
1.98
1.00
0.95
1.00
1.02
4.11
1.06
1.02
3.05
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

177.321
136.979
134.027
128.449
127.315
126.082
124.427
59.317
45.456
32.949
30.493
27.381



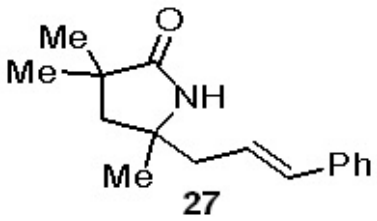
26

	Parameter	Value
1	Title	grd-1-192.3.fid
2	Solvent	CDCl3
3	Temperature	300.0
4	Number of Scans	256
5	Receiver Gain	2050.0
6	Relaxation Delay	5.0000
7	Pulse Width	10.6300
8	Spectrometer Frequency	150.97
9	Nucleus	13C



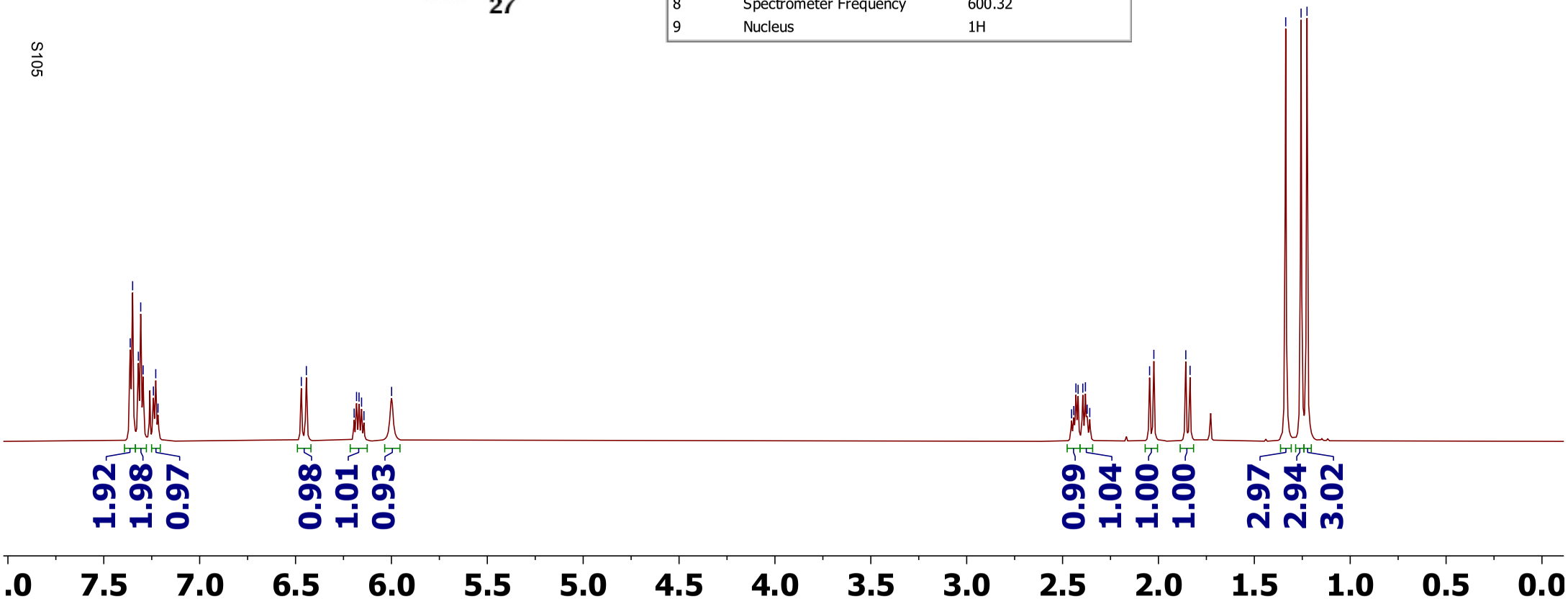
7.362 7.350 7.320 7.307 7.295 7.241 7.229 7.217 6.470 6.444 6.194 6.182 6.169 6.156 6.143 5.999

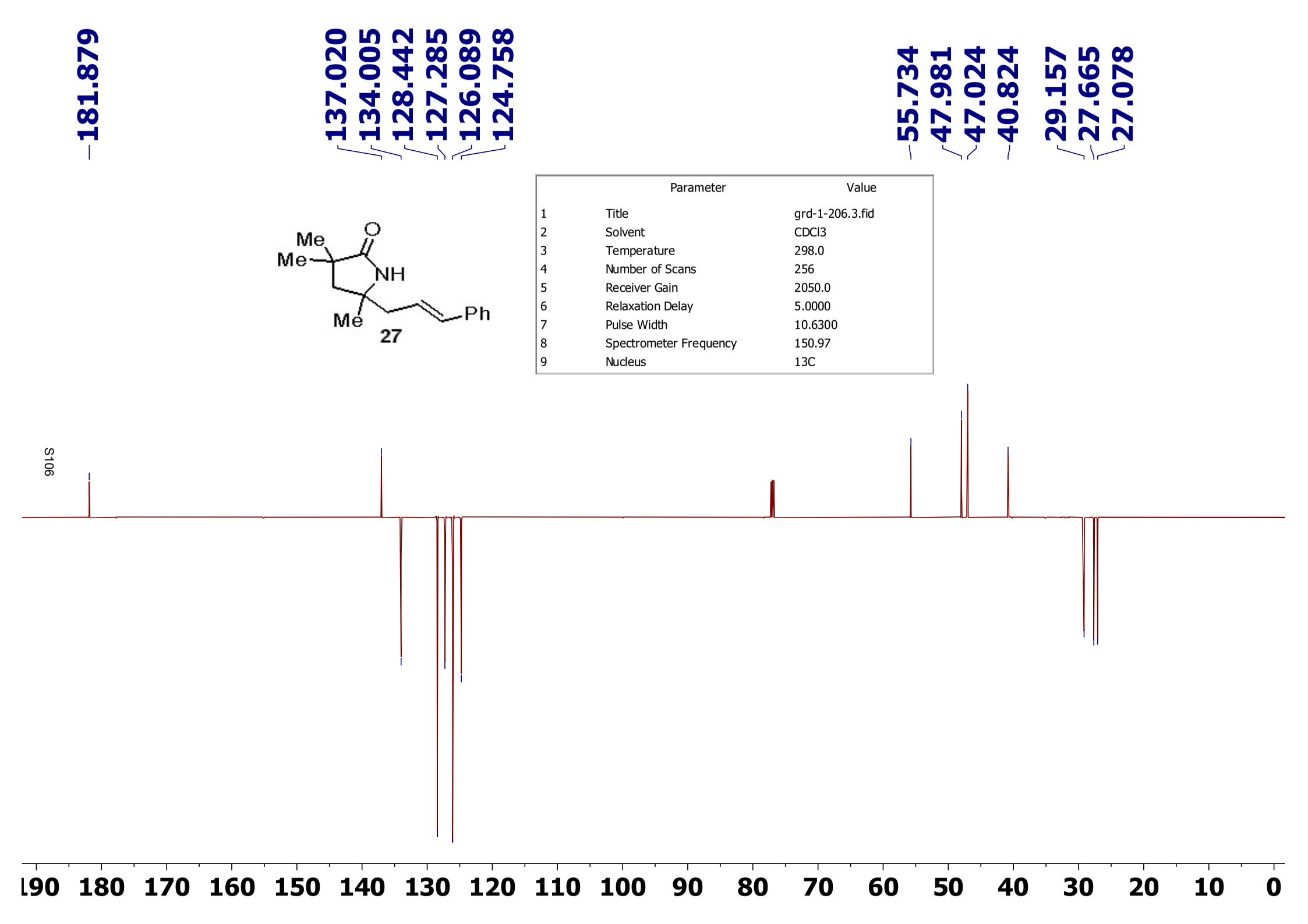
2.453 2.442 2.430 2.419 2.394 2.381 2.372 2.358 2.046 2.024 1.857 1.835 1.336 1.256 1.225



27

	Parameter	Value
1	Title	grd-1-206.4.fid
2	Solvent	CDCl3
3	Temperature	298.0
4	Number of Scans	16
5	Receiver Gain	57.0
6	Relaxation Delay	1.0000
7	Pulse Width	10.5000
8	Spectrometer Frequency	600.32
9	Nucleus	1H

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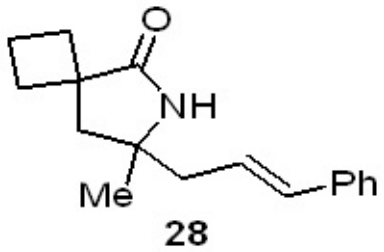
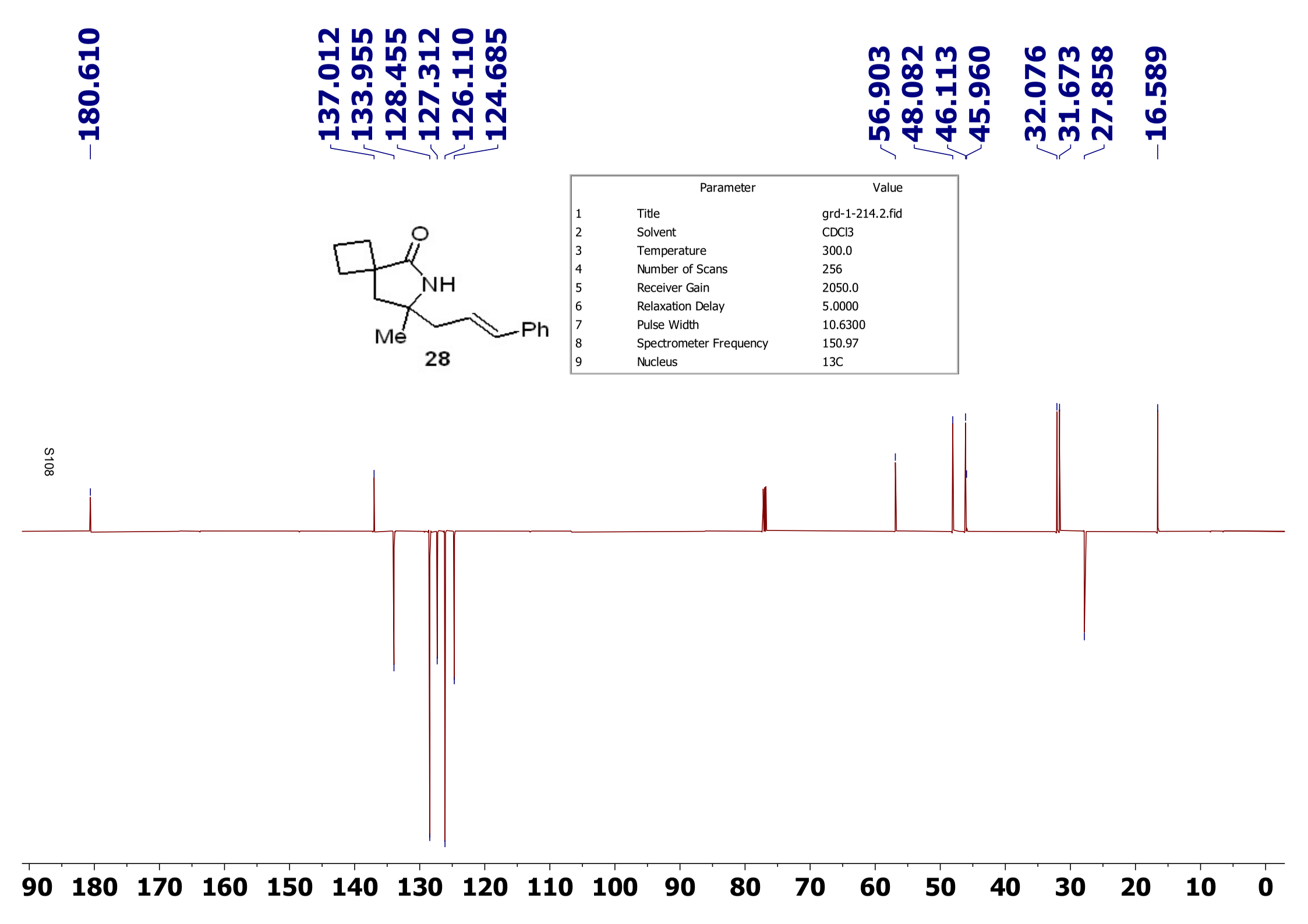
134.005
128.442
127.285
126.089
124.758
55.734
47.981
47.024
40.824
29.157
27.665
27.078
Me
Me
O
NH
Me
Ph
27
Parameter Value
1 Title grd-1-206.3.fid
2 Solvent CDCl3
3 Temperature 298.0
4 Number of Scans 256
5 Receiver Gain 2050.0
6 Relaxation Delay 5.0000
7 Pulse Width 10.6300
8 Spectrometer Frequency 150.97
9 Nucleus 13C
S106
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0



28

	Parameter	Value
1	Title	grd-1-214.1.fid
2	Solvent	CDCl3
3	Temperature	300.0
4	Number of Scans	16
5	Receiver Gain	20.2
6	Relaxation Delay	1.0000
7	Pulse Width	10.5000
8	Spectrometer Frequency	600.32
9	Nucleus	1H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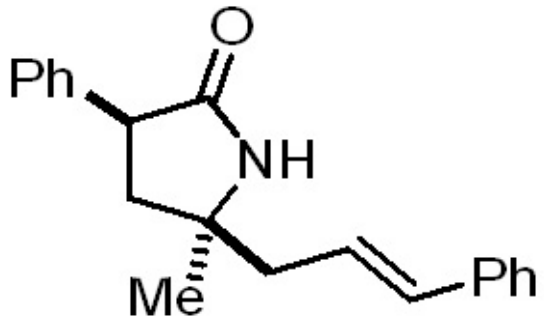
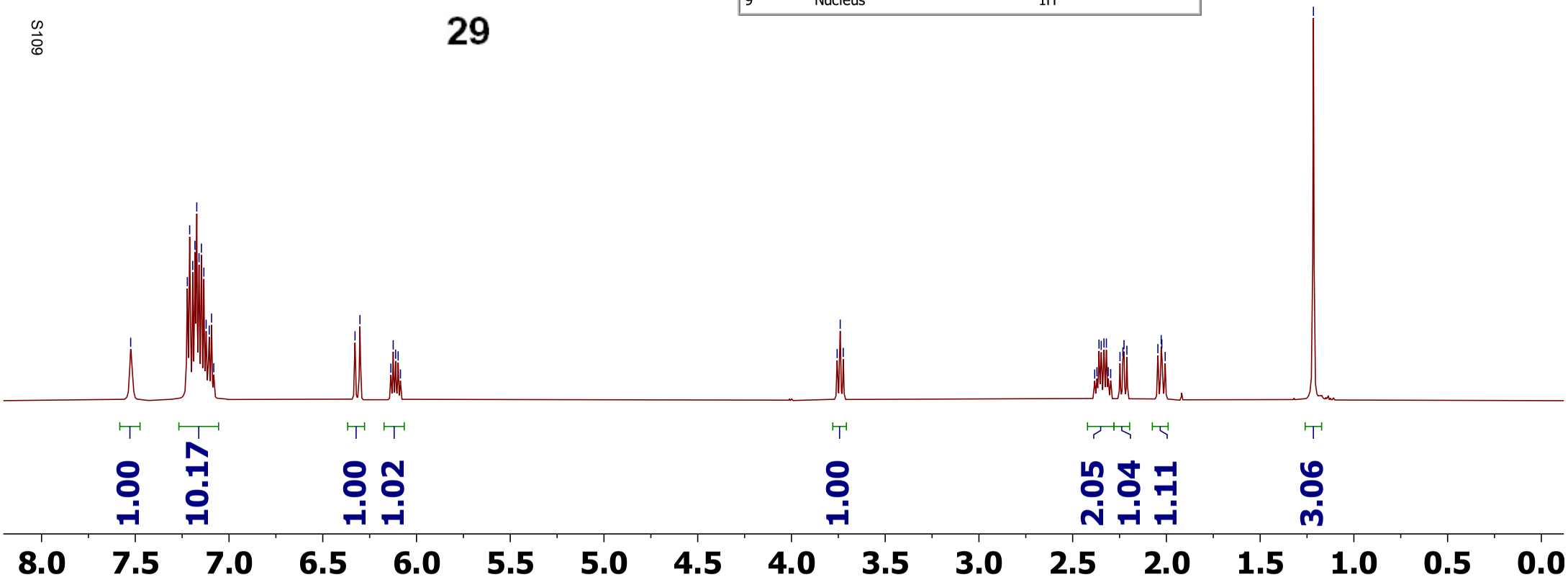
128.455
127.312
126.110
124.685
56.903
48.082
46.113
45.960
32.076
31.673
27.858
16.589
O
NH
Me
Ph
28
Parameter Value
1 Title grd-1-214.2.fid
2 Solvent CDCl3
3 Temperature 300.0
4 Number of Scans 256
5 Receiver Gain 2050.0
6 Relaxation Delay 5.0000
7 Pulse Width 10.6300
8 Spectrometer Frequency 150.97
9 Nucleus 13C
S108
90 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

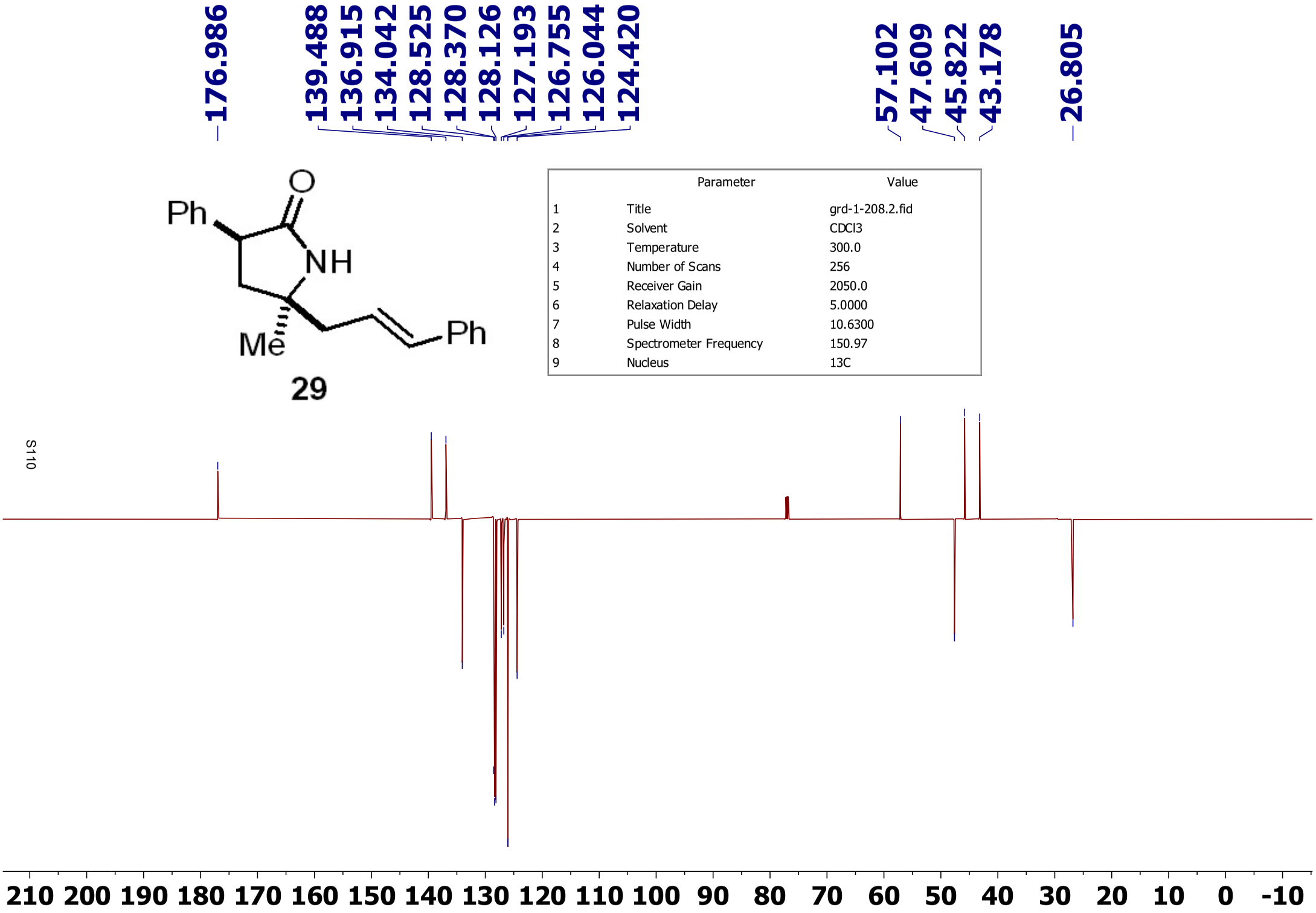
7.525 7.222 7.210 7.194 7.182 7.172 7.160 7.147 7.134 7.122 7.106 7.093 7.081 6.328 6.302 6.137 6.124 6.111 6.098 3.756 3.740 3.724 2.371 2.359 2.348 2.333 2.320 2.310 2.248 2.232 2.226 2.211 2.046 2.028 2.024 2.007 1.216

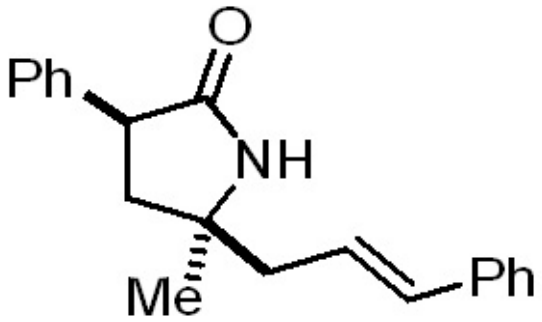


29

	Parameter	Value
1	Title	grd-1-208.1.fid
2	Solvent	CDCl3
3	Temperature	300.0
4	Number of Scans	16
5	Receiver Gain	12.7
6	Relaxation Delay	1.0000
7	Pulse Width	10.5000
8	Spectrometer Frequency	600.32
9	Nucleus	1H

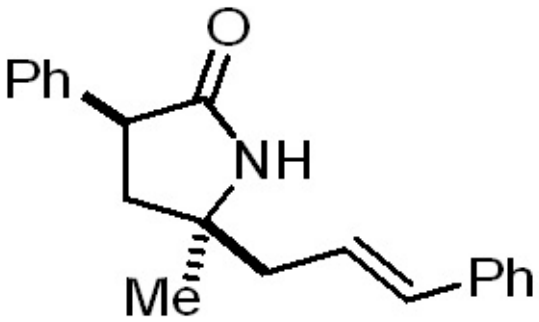



176.986
139.488
136.915
134.042
128.525
128.370
128.126
127.193
126.755
126.044
124.420
57.102
47.609
45.822
43.178
26.805
O
Ph
NH
Me
Ph
29
Parameter Value
1 Title grd-1-208.2.fid
2 Solvent CDCl3
3 Temperature 300.0
4 Number of Scans 256
5 Receiver Gain 2050.0
6 Relaxation Delay 5.0000
7 Pulse Width 10.6300
8 Spectrometer Frequency 150.97
9 Nucleus 13C
S110
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10



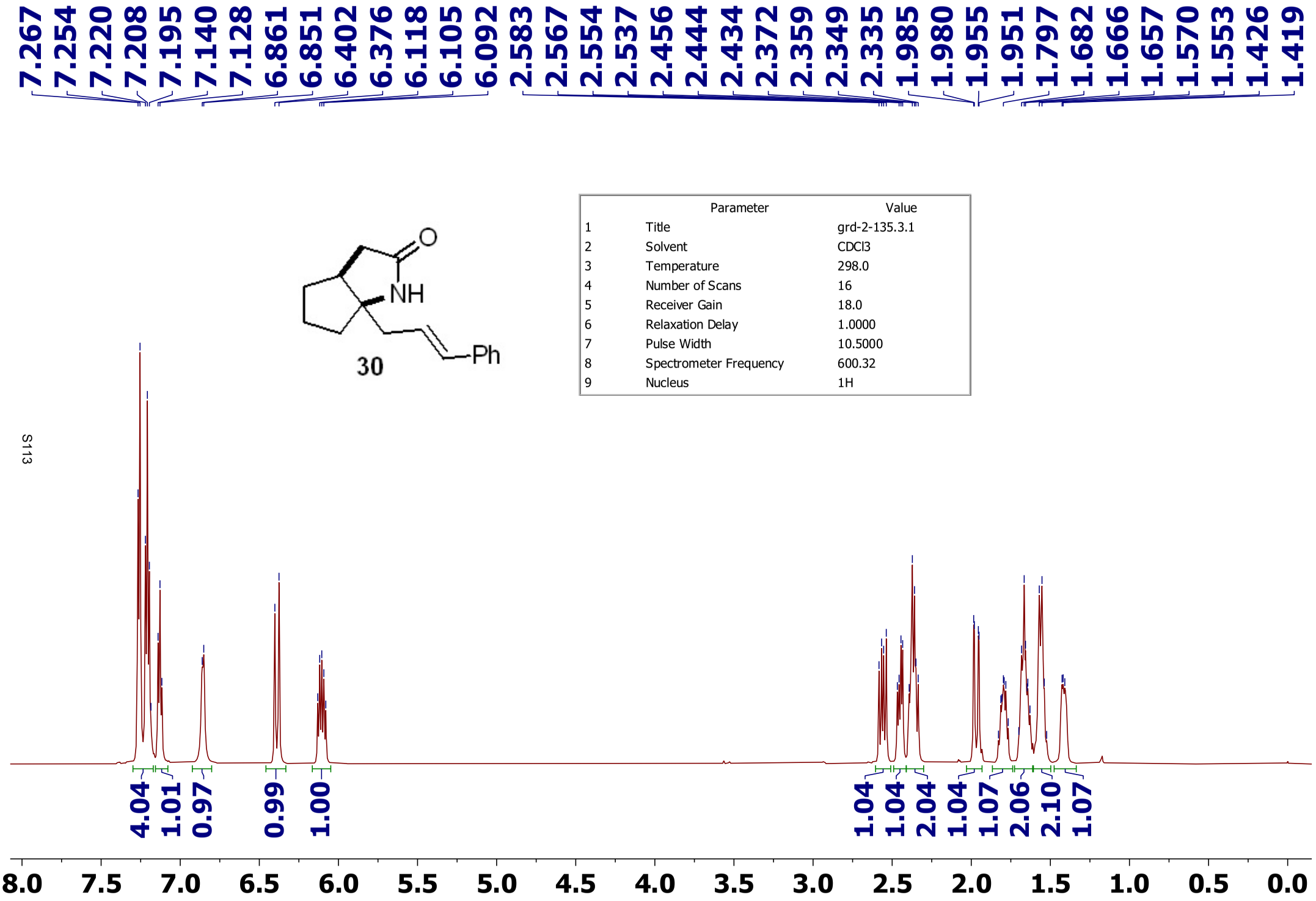
29

	Parameter	Value
1	Title	grd-1-208.6.fid
2	Solvent	CDCl3
3	Temperature	298.2
4	Number of Scans	64
5	Receiver Gain	18.0
6	Relaxation Delay	2.0000
7	Pulse Width	15.0000
8	Spectrometer Frequency	400.13
9	Nucleus	1H



29

	Parameter	Value
1	Title	grd-1-208.7.fid
2	Solvent	CDCl3
3	Temperature	298.2
4	Number of Scans	64
5	Receiver Gain	22.6
6	Relaxation Delay	2.0000
7	Pulse Width	15.0000
8	Spectrometer Frequency	400.13
9	Nucleus	1H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7.140
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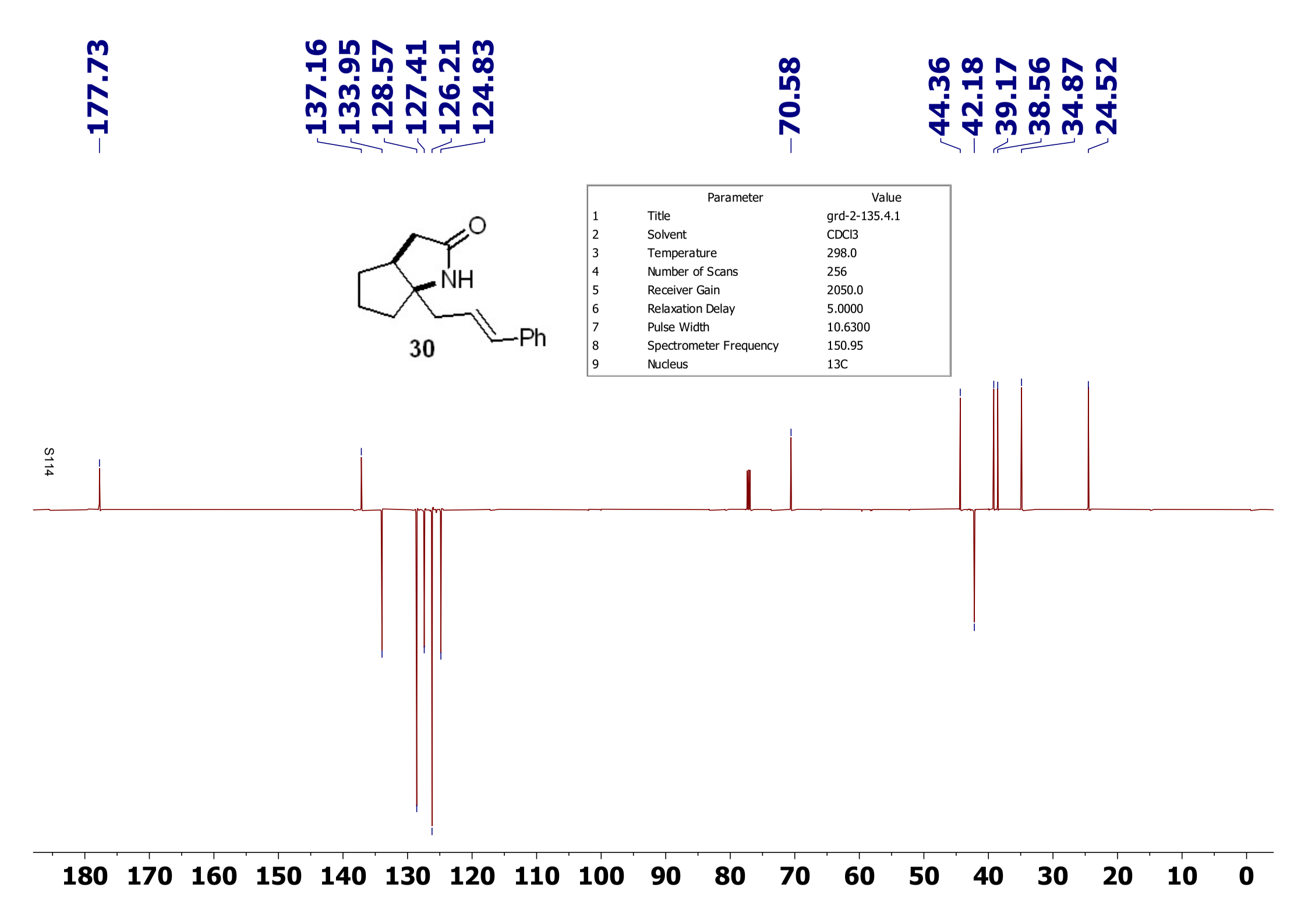
6.851
6.402
6.376
6.118
6.105
6.092
2.583
2.567
2.554
2.537
2.456
2.444
2.434
2.372
2.359
2.349
2.335
1.985
1.980
1.955
1.951
1.797
1.682
1.666
1.657
1.570
1.553
1.426
1.419
O
NH
Ph
30
Parameter Value
1 Title grd-2-135.3.1
2 Solvent CDCl3
3 Temperature 298.0
4 Number of Scans 16
5 Receiver Gain 18.0
6 Relaxation Delay 1.0000
7 Pulse Width 10.5000
8 Spectrometer Frequency 600.32
9 Nucleus 1H
S113
4.04
1.01
0.97
0.99
1.00
1.04
1.04
2.04
1.04
1.07
2.06
2.10
1.07
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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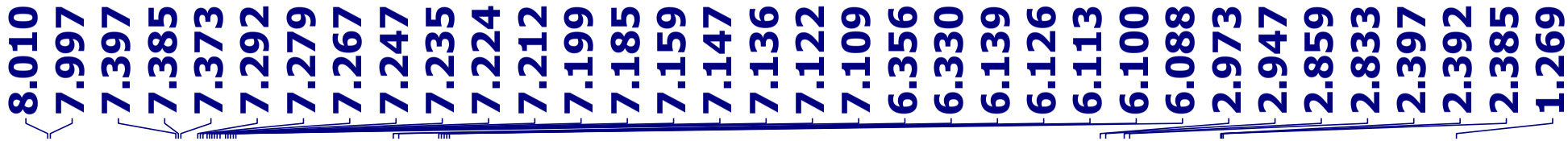
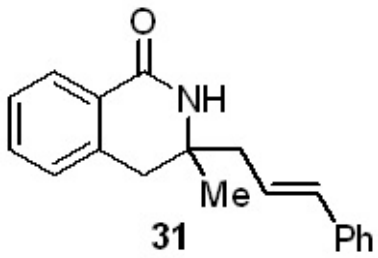
133.95
128.57
127.41
126.21
124.83
70.58
44.36
42.18
39.17
38.56
34.87
24.52
NH
O
Ph
30
Parameter Value
1 Title grd-2-135.4.1
2 Solvent CDCl3
3 Temperature 298.0
4 Number of Scans 256
5 Receiver Gain 2050.0
6 Relaxation Delay 5.0000
7 Pulse Width 10.6300
8 Spectrometer Frequency 150.95
9 Nucleus 13C
S114
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

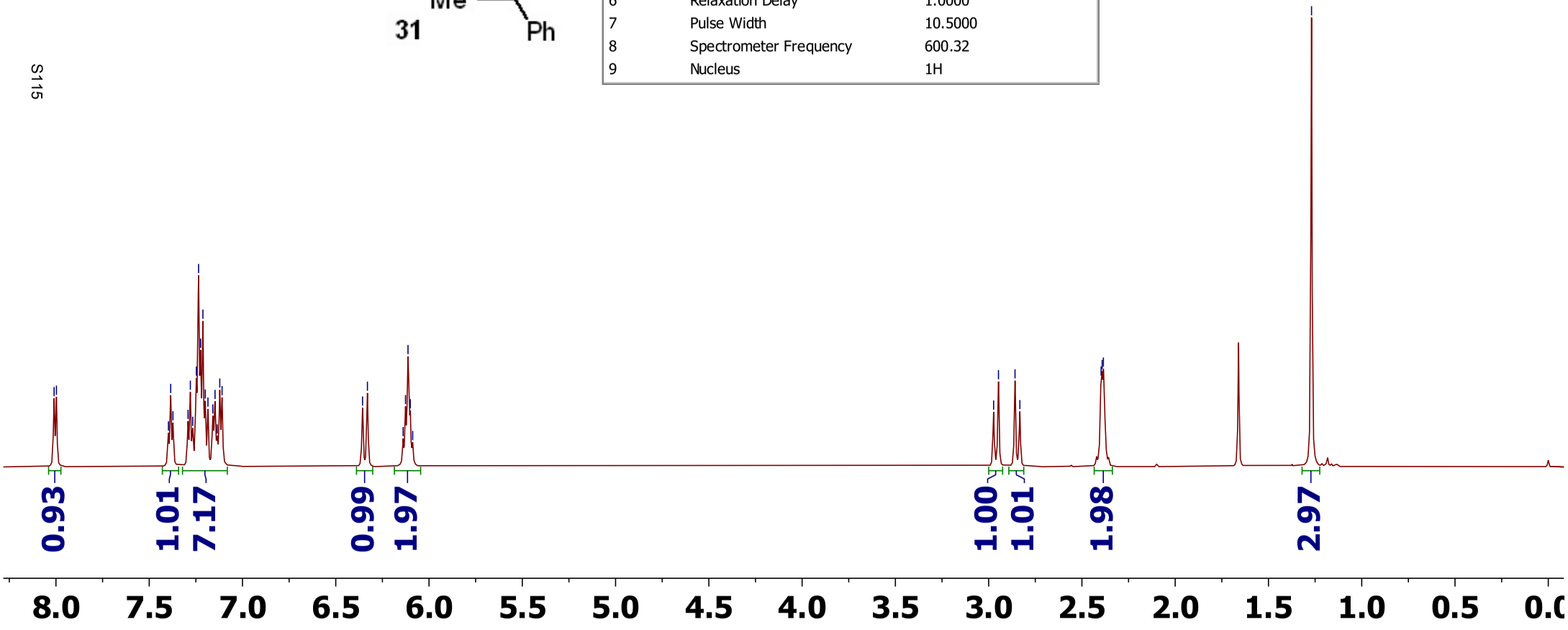


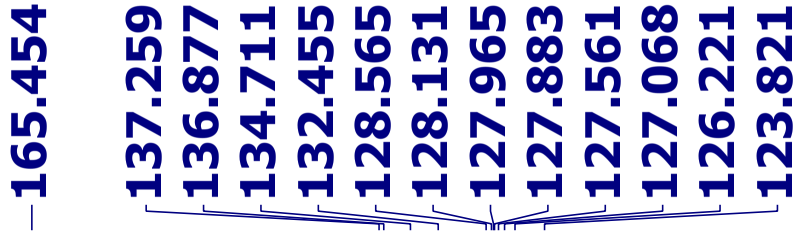


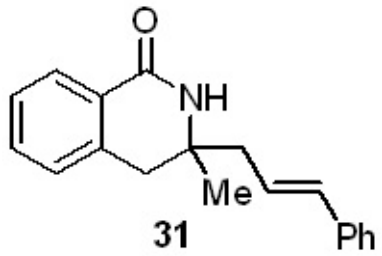
31

	Parameter	Value
1	Title	grd-1-238.3.fid
2	Solvent	CDCl3
3	Temperature	298.0
4	Number of Scans	16
5	Receiver Gain	90.5
6	Relaxation Delay	1.0000
7	Pulse Width	10.5000
8	Spectrometer Frequency	600.32
9	Nucleus	1H

S115

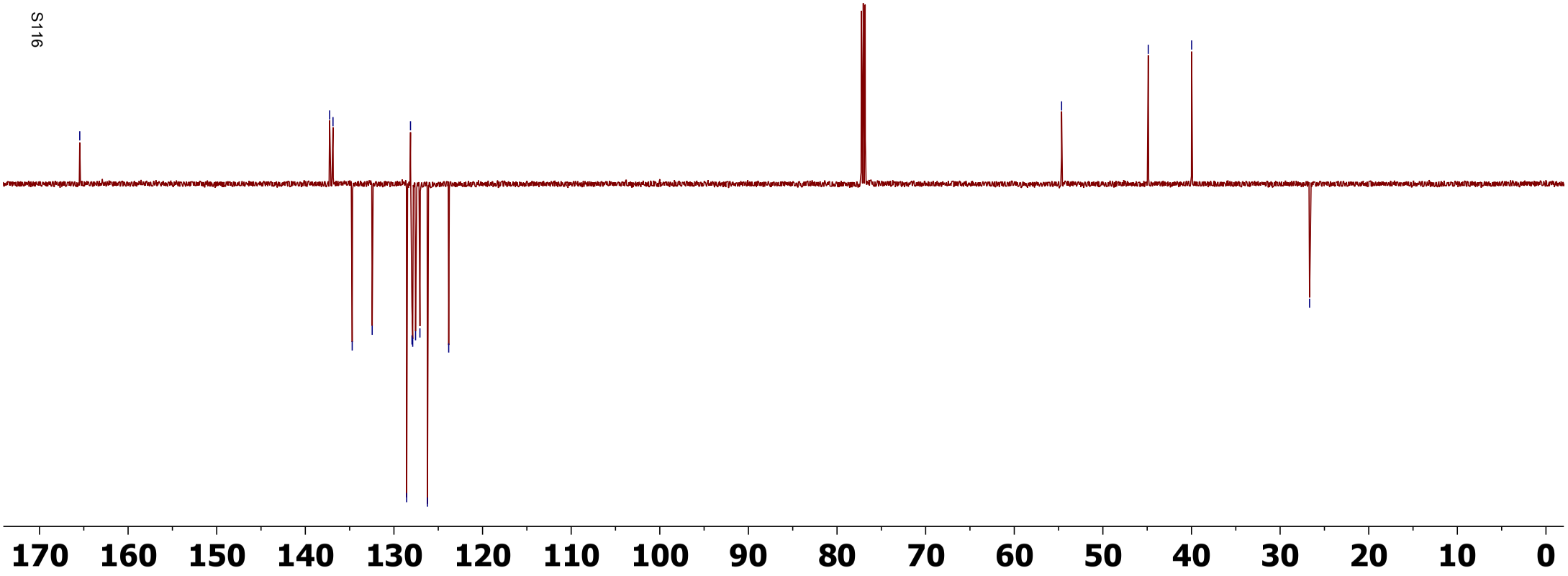




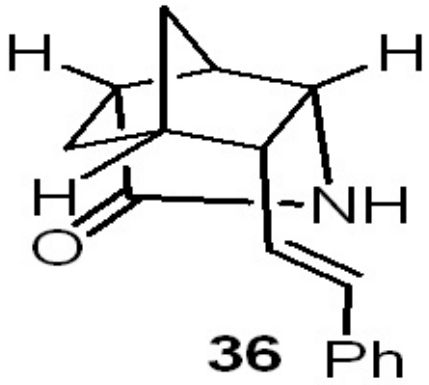


31

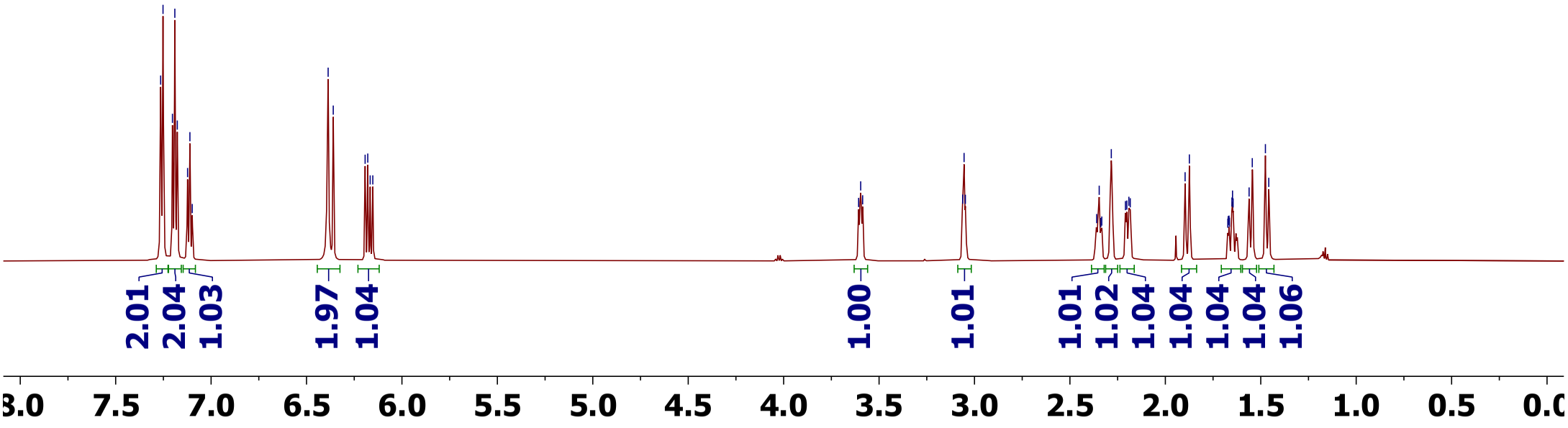
	Parameter	Value
1	Title	grd-1-238.4.fid
2	Solvent	CDCl3
3	Temperature	298.0
4	Number of Scans	256
5	Receiver Gain	2050.0
6	Relaxation Delay	5.0000
7	Pulse Width	10.6300
8	Spectrometer Frequency	150.97
9	Nucleus	13C

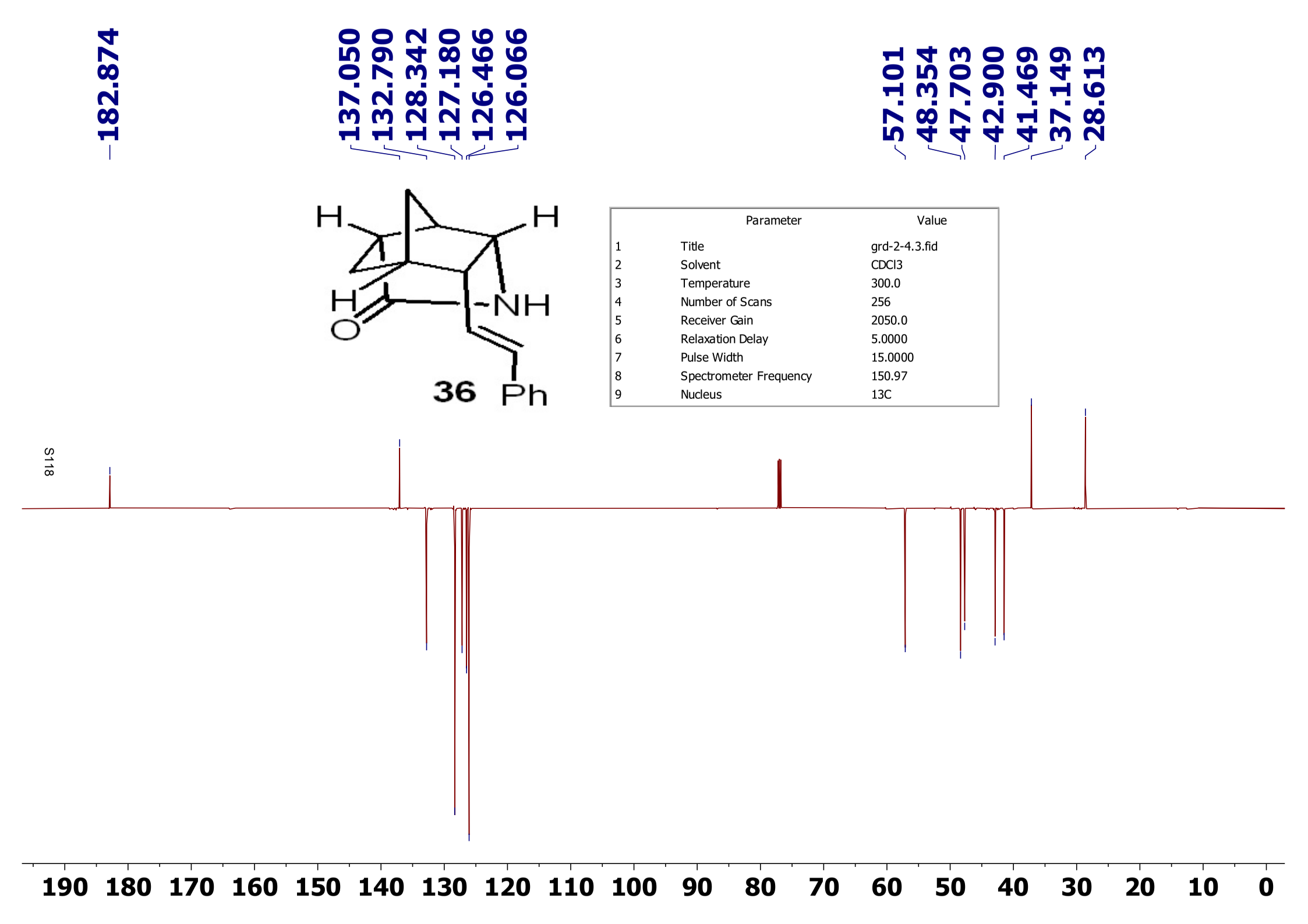


7.264 7.252 7.203 7.190 7.177 7.123 7.111 7.099 6.387 6.360 6.193 6.179 6.167 6.153 3.607 3.596 3.586 3.062 3.055 3.048 2.360 2.347 2.283 2.209 2.202 2.191 2.184 1.896 1.874 1.666 1.651 1.648 1.645 1.561 1.544 1.476 1.458

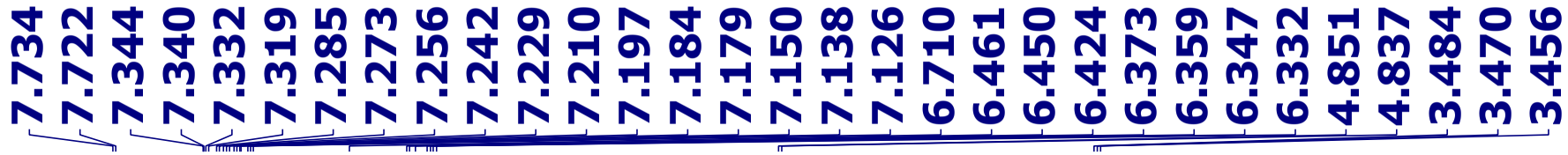


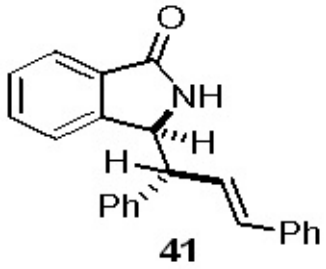
	Parameter	Value
1	Title	grd-2-4.2.fid
2	Solvent	CDCl3
3	Temperature	300.0
4	Number of Scans	16
5	Receiver Gain	18.0
6	Relaxation Delay	1.0000
7	Pulse Width	10.6100
8	Spectrometer Frequency	600.32
9	Nucleus	1H





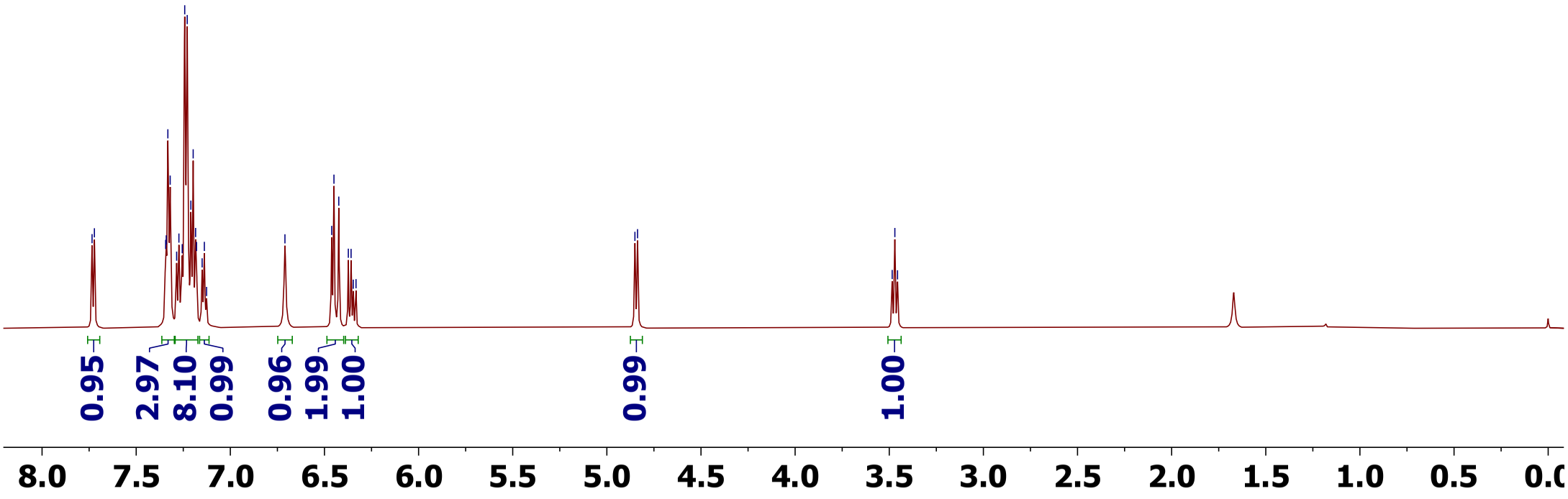
	Parameter	Value
1	Title	grd-2-4.3.fid
2	Solvent	CDCl3
3	Temperature	300.0
4	Number of Scans	256
5	Receiver Gain	2050.0
6	Relaxation Delay	5.0000
7	Pulse Width	15.0000
8	Spectrometer Frequency	150.97
9	Nucleus	13C

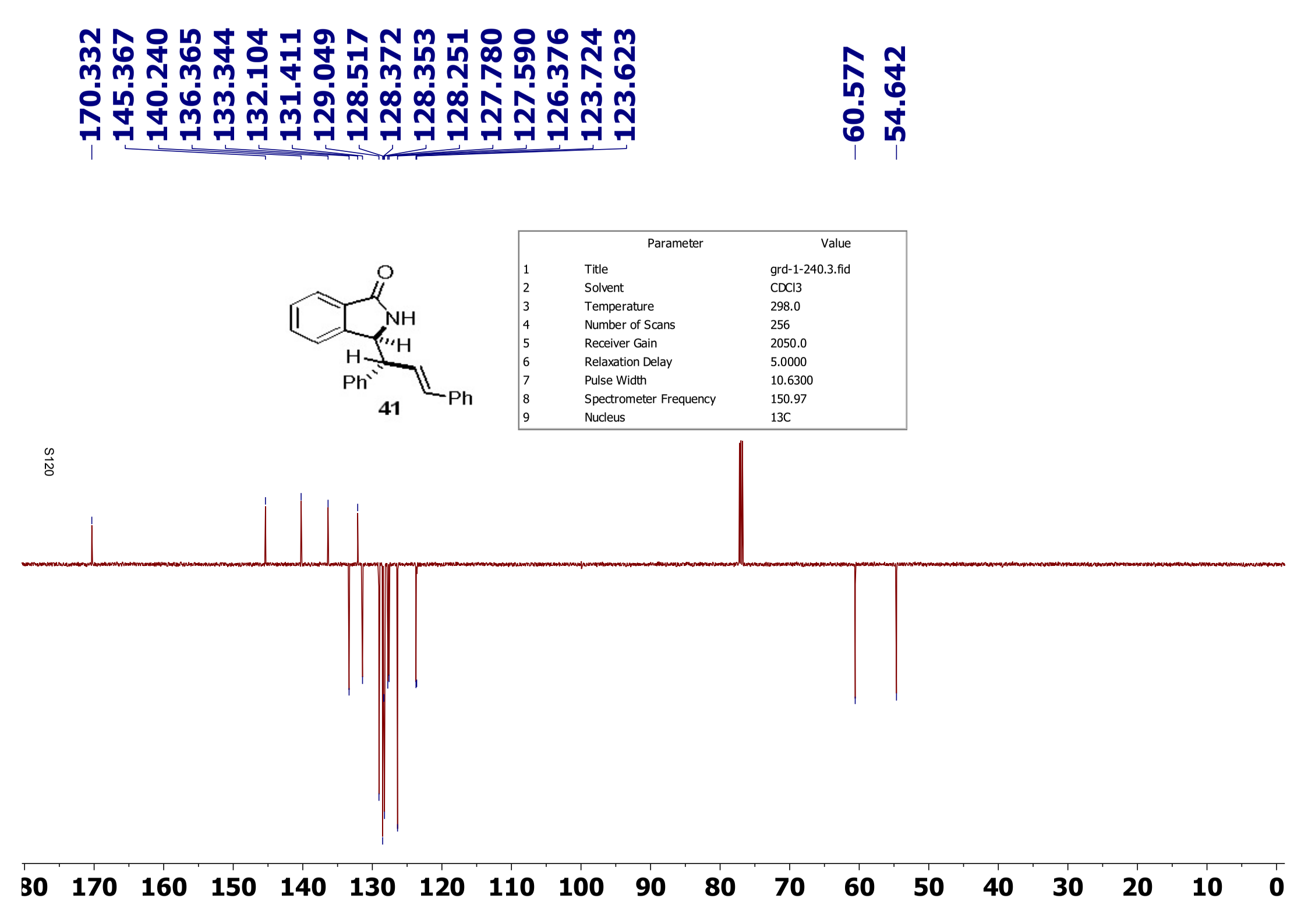


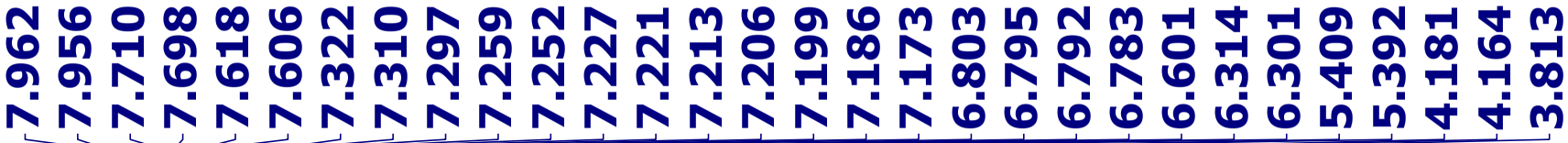


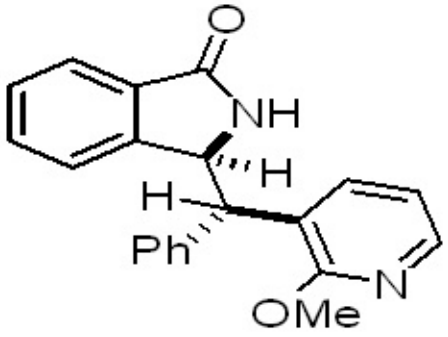
41

	Parameter	Value
1	Title	grd-1-240.2.fid
2	Solvent	CDCl3
3	Temperature	298.0
4	Number of Scans	16
5	Receiver Gain	57.0
6	Relaxation Delay	1.0000
7	Pulse Width	10.5000
8	Spectrometer Frequency	600.32
9	Nucleus	1H



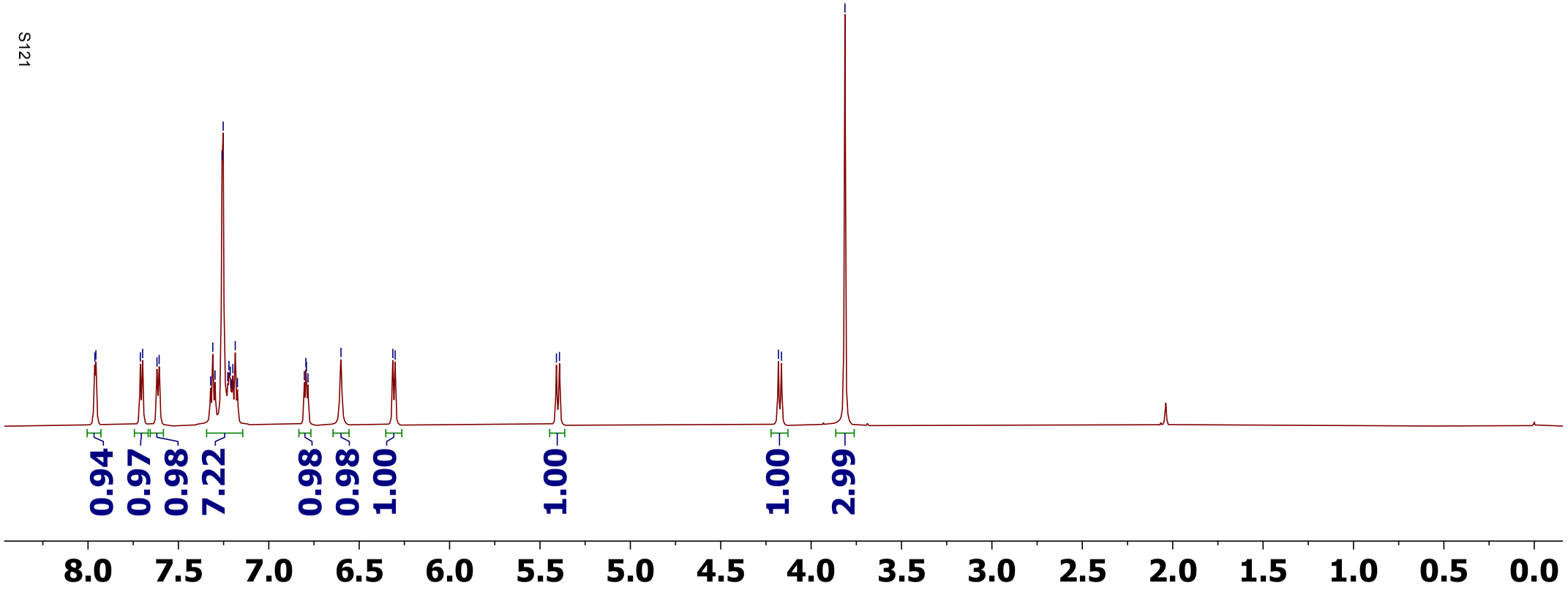

170.332
145.367
140.240
136.365
133.344
132.104
131.411
129.049
128.517
128.372
128.353
128.251
127.780
127.590
126.376
123.724
123.623
60.577
54.642
NH
H
H
Ph
Ph
41
Parameter Value
1 Title grd-1-240.3.fid
2 Solvent CDCl3
3 Temperature 298.0
4 Number of Scans 256
5 Receiver Gain 2050.0
6 Relaxation Delay 5.0000
7 Pulse Width 10.6300
8 Spectrometer Frequency 150.97
9 Nucleus 13C
S120
80 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

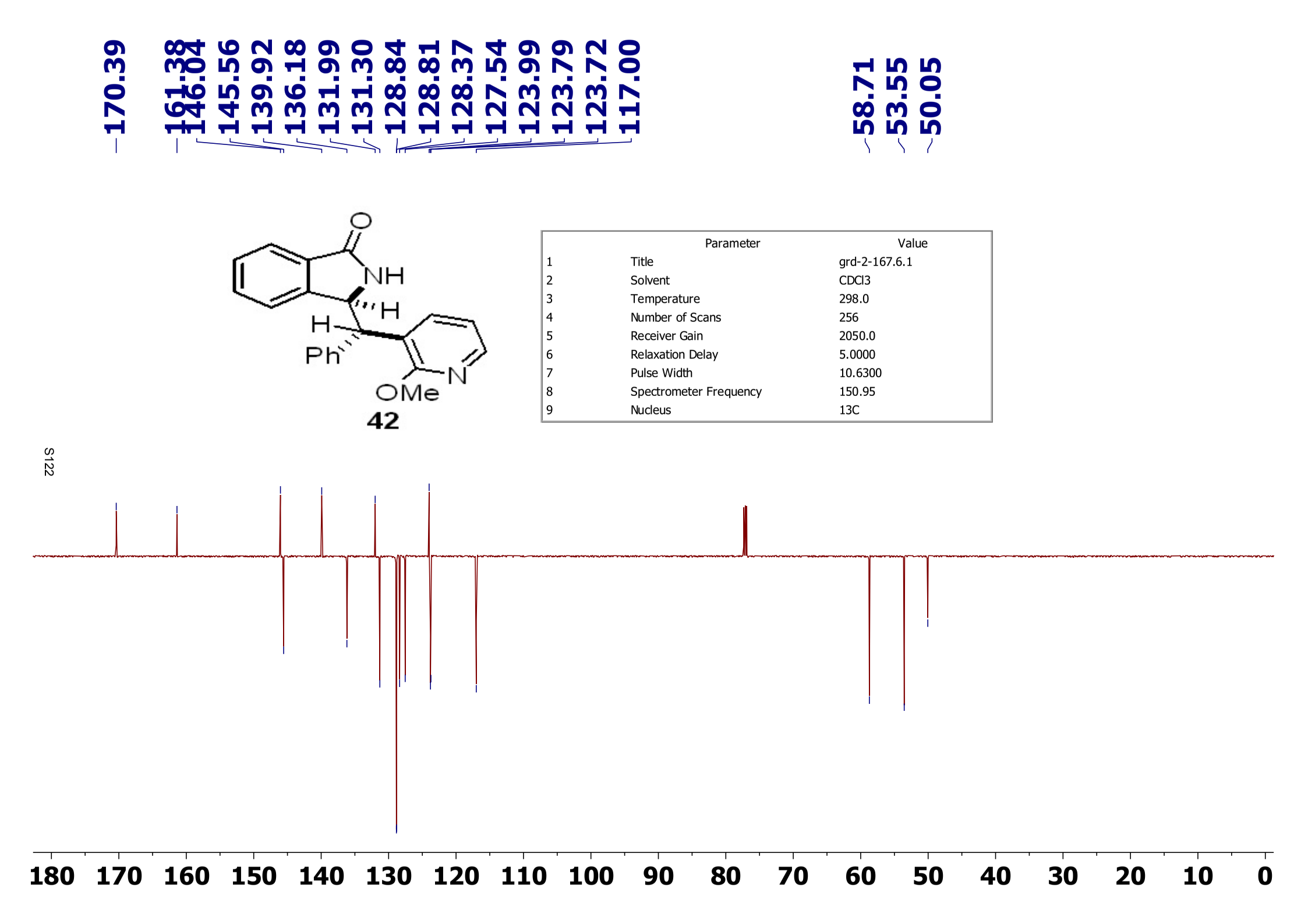




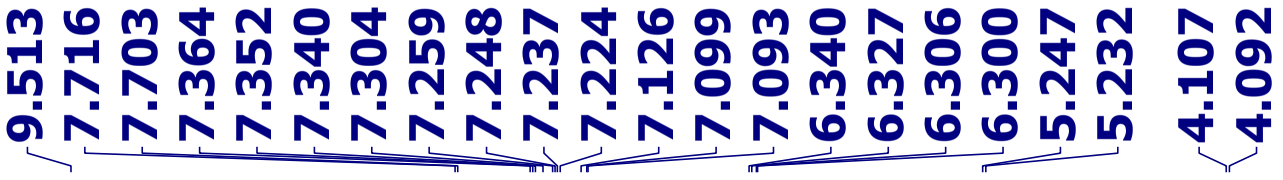
42

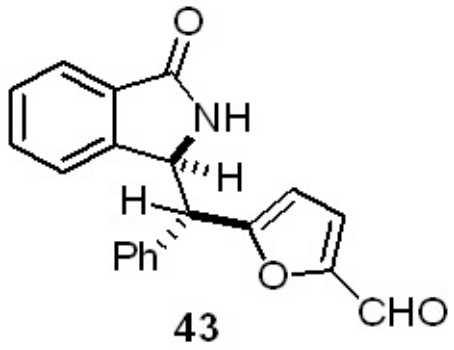
	Parameter	Value
1	Title	grd-2-167.5.1
2	Solvent	CDCl3
3	Temperature	298.0
4	Number of Scans	16
5	Receiver Gain	40.3
6	Relaxation Delay	1.0000
7	Pulse Width	10.5000
8	Spectrometer Frequency	600.32
9	Nucleus	1H





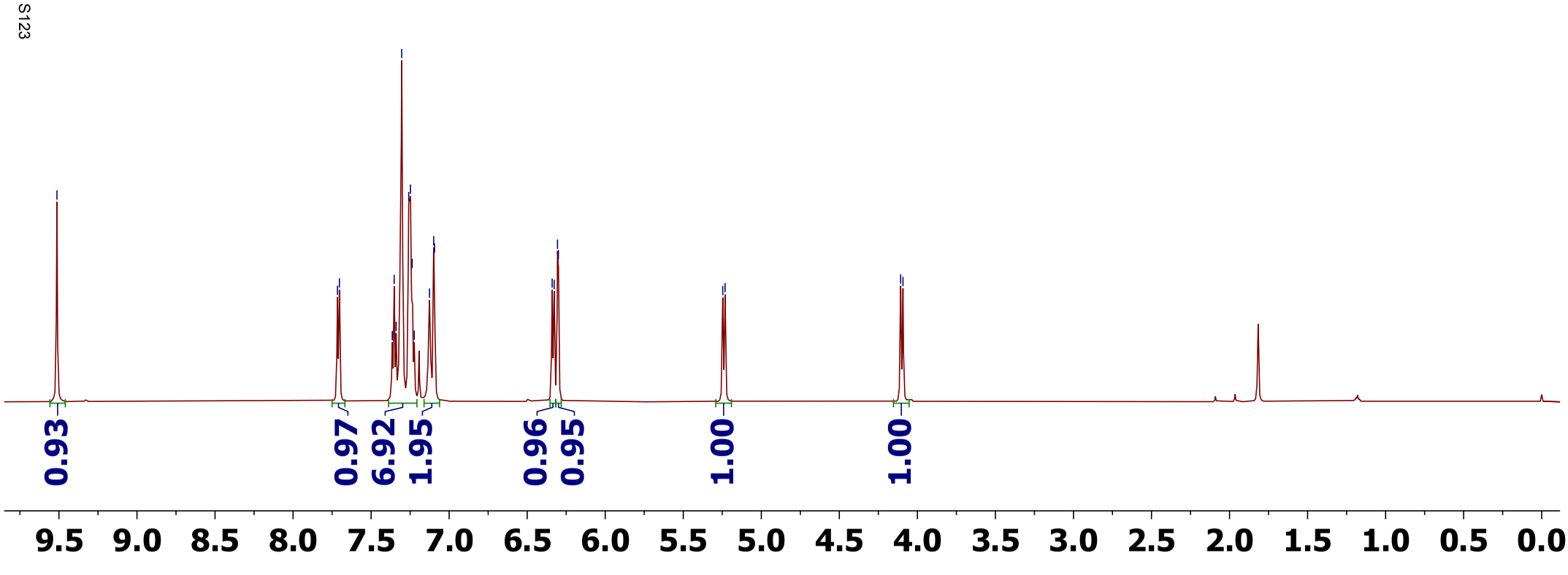
	Parameter	Value
1	Title	grd-2-167.6.1
2	Solvent	CDCl3
3	Temperature	298.0
4	Number of Scans	256
5	Receiver Gain	2050.0
6	Relaxation Delay	5.0000
7	Pulse Width	10.6300
8	Spectrometer Frequency	150.95
9	Nucleus	13C



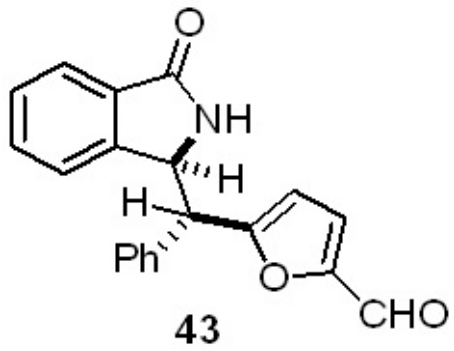


43

	Parameter	Value
1	Title	grd-2-172.3.1
2	Solvent	CDCl3
3	Temperature	298.0
4	Number of Scans	16
5	Receiver Gain	57.0
6	Relaxation Delay	1.0000
7	Pulse Width	10.5000
8	Spectrometer Frequency	600.32
9	Nucleus	1H

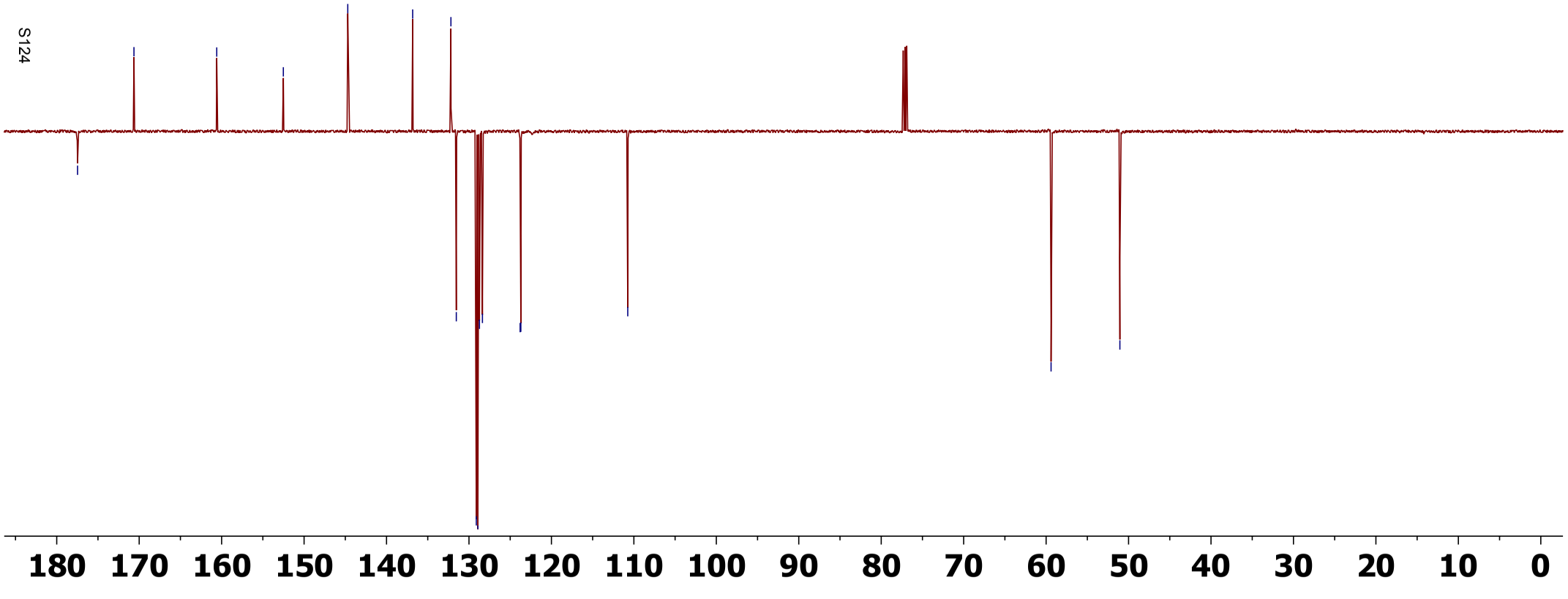


177.48
170.63
160.59
152.52
144.70
136.83
132.19
131.54
129.11
128.92
128.73
128.37
123.80
123.71
110.74
59.40
51.05

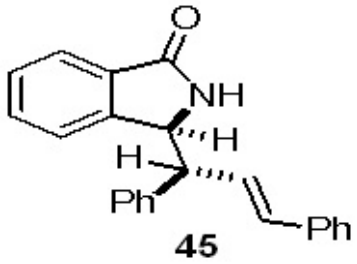


43

	Parameter	Value
1	Title	grd-2-172.2.1
2	Solvent	CDCl3
3	Temperature	298.0
4	Number of Scans	256
5	Receiver Gain	2050.0
6	Relaxation Delay	5.0000
7	Pulse Width	10.6300
8	Spectrometer Frequency	150.95
9	Nucleus	13C



7.744 7.734 7.385 7.380 7.374 7.361 7.353 7.341 7.307 7.295 7.271 7.258 7.245 7.230 7.216 7.203 7.187 7.179 7.175 6.481 6.467 6.455 6.441 6.431 6.405 6.357 4.895 4.881 3.529 3.515 3.501



45

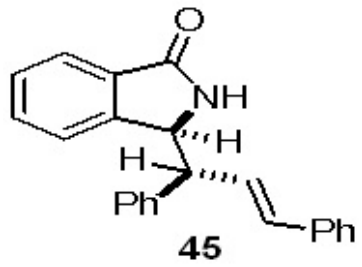
	Parameter	Value
1	Title	grd-2-43.5.fid
2	Solvent	CDCl3
3	Temperature	300.0
4	Number of Scans	16
5	Receiver Gain	57.0
6	Relaxation Delay	1.0000
7	Pulse Width	10.5000
8	Spectrometer Frequency	600.32
9	Nucleus	1H

S125

0.95 13.29 2.05 1.00 1.00 1.02

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

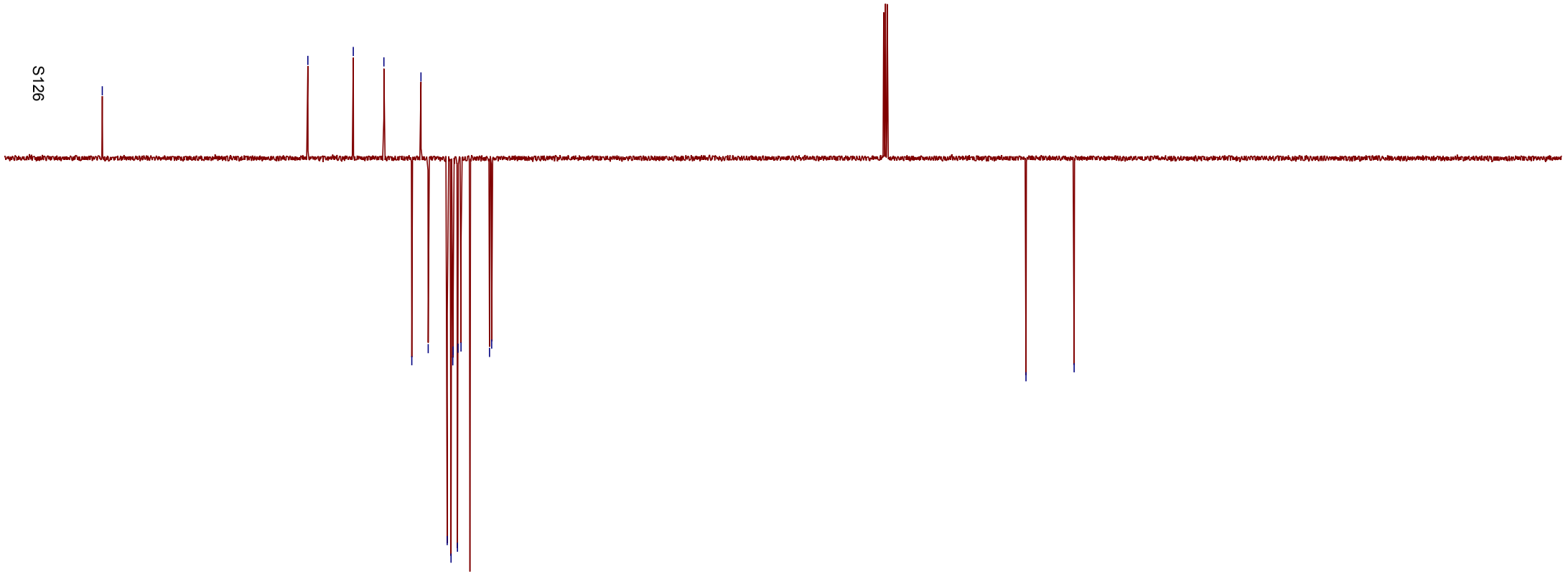
170.073
145.642
140.245
136.605
133.289
132.202
131.341
129.081
128.626
128.418
128.381
127.866
127.800
127.442
126.381
124.055
123.798
60.301
54.580



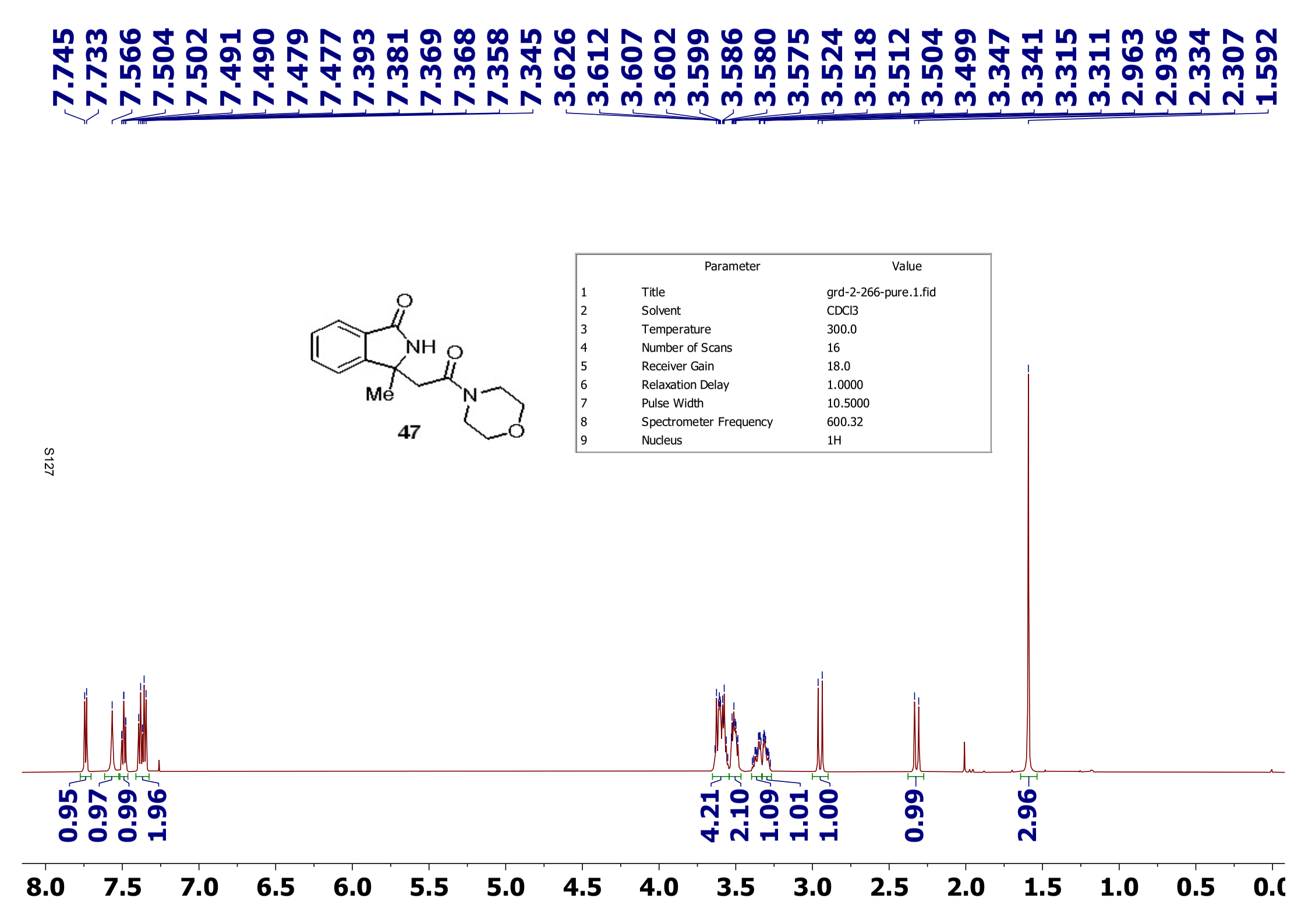
45

	Parameter	Value
1	Title	grd-2-43.6.fid
2	Solvent	CDCl3
3	Temperature	300.0
4	Number of Scans	256
5	Receiver Gain	2050.0
6	Relaxation Delay	5.0000
7	Pulse Width	10.6300
8	Spectrometer Frequency	150.97
9	Nucleus	13C

S126



.80 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0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7.566
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7.502
7.491
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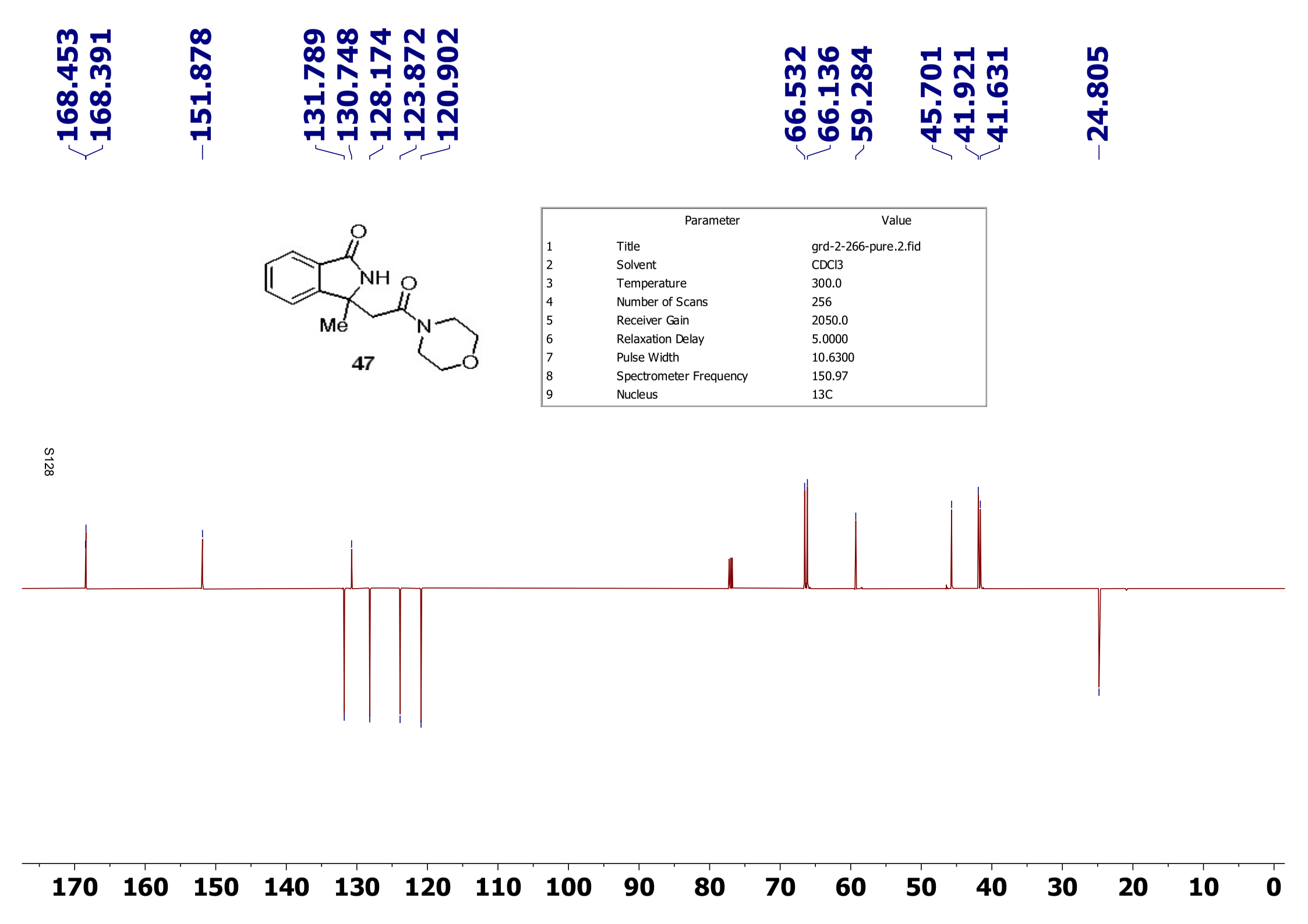
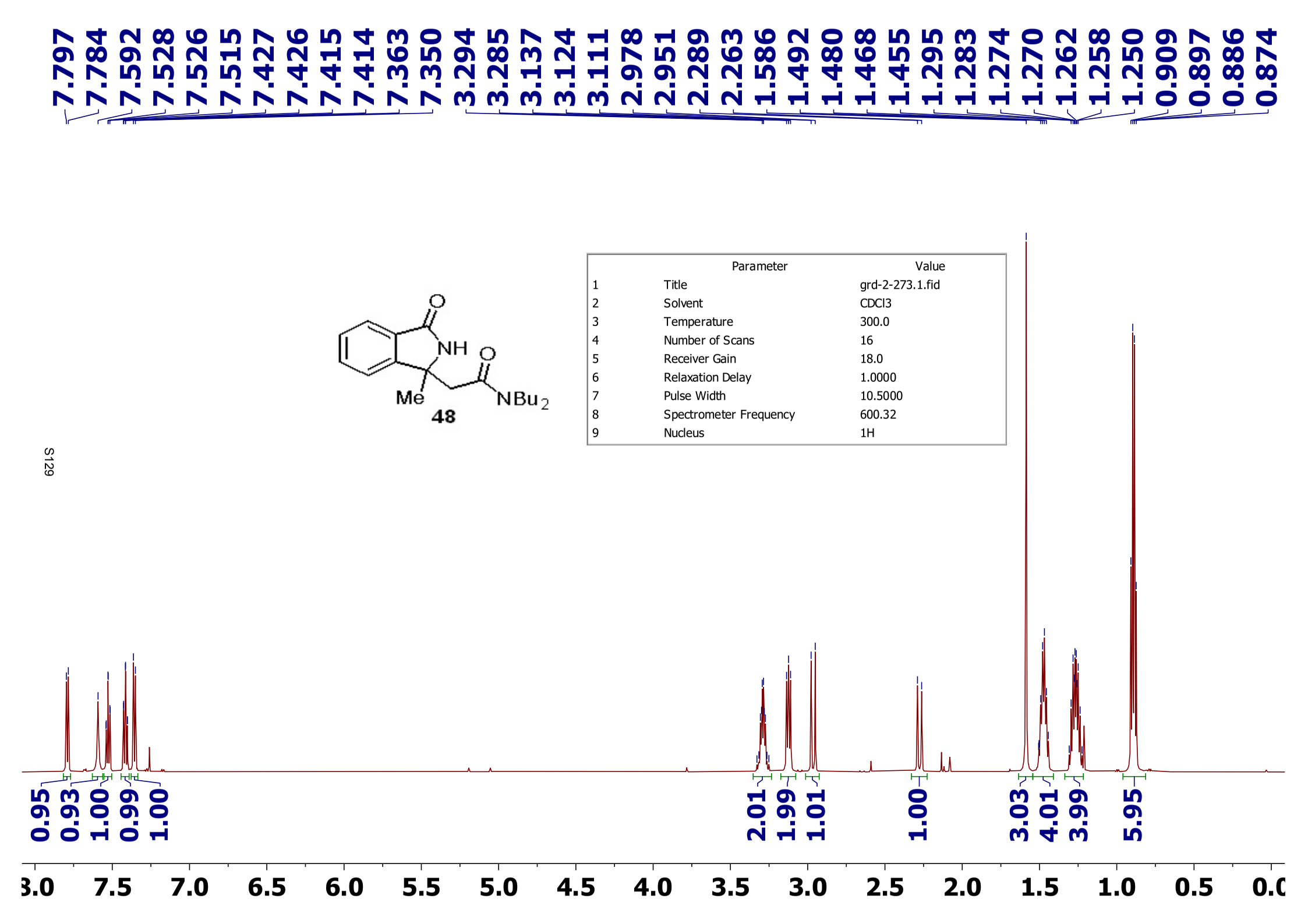
7.479
7.477
7.393
7.381
7.369
7.368
7.358
7.345
3.626
3.612
3.607
3.602
3.599
3.586
3.580
3.575
3.524
3.518
3.512
3.504
3.499
3.347
3.341
3.315
3.311
2.963
2.936
2.334
2.307
1.592
NH
O
O
N
O
Me
47
Parameter
Value
1 Title grd-2-266-pure.1.fid
2 Solvent CDCl3
3 Temperature 300.0
4 Number of Scans 16
5 Receiver Gain 18.0
6 Relaxation Delay 1.0000
7 Pulse Width 10.5000
8 Spectrometer Frequency 600.32
9 Nucleus 1H
S127
0.95
0.97
0.99
1.96
4.21
2.10
1.09
1.01
1.00
0.99
2.96
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



	Parameter	Value
1	Title	grd-2-266-pure.2.fid
2	Solvent	CDCl3
3	Temperature	300.0
4	Number of Scans	256
5	Receiver Gain	2050.0
6	Relaxation Delay	5.0000
7	Pulse Width	10.6300
8	Spectrometer Frequency	150.97
9	Nucleus	13C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7.784
7.592
7.528
7.526
7.515
7.427
7.426
7.415
7.414
7.363
7.350
3.294
3.285
3.137
3.124
3.111
2.978
2.951
2.289
2.263
1.586
1.492
1.480
1.468
1.455
1.295
1.283
1.274
1.270
1.262
1.258
1.250
0.909
0.897
0.886
0.874
NH
O
O
Me
NBu2
48
Parameter
Value
1 Title grd-2-273.1.fid
2 Solvent CDCl3
3 Temperature 300.0
4 Number of Scans 16
5 Receiver Gain 18.0
6 Relaxation Delay 1.0000
7 Pulse Width 10.5000
8 Spectrometer Frequency 600.32
9 Nucleus 1H
S129
0.95
0.93
1.00
0.99
1.00
2.01
1.99
1.01
1.00
3.03
4.01
3.99
5.95
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

169.303
168.404
152.179
131.767
131.064
128.163
124.029
120.852
59.548
47.648
45.778
42.072
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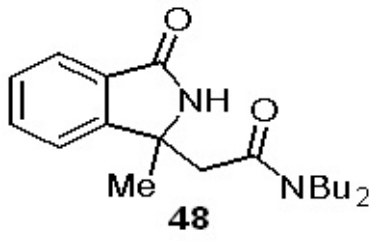
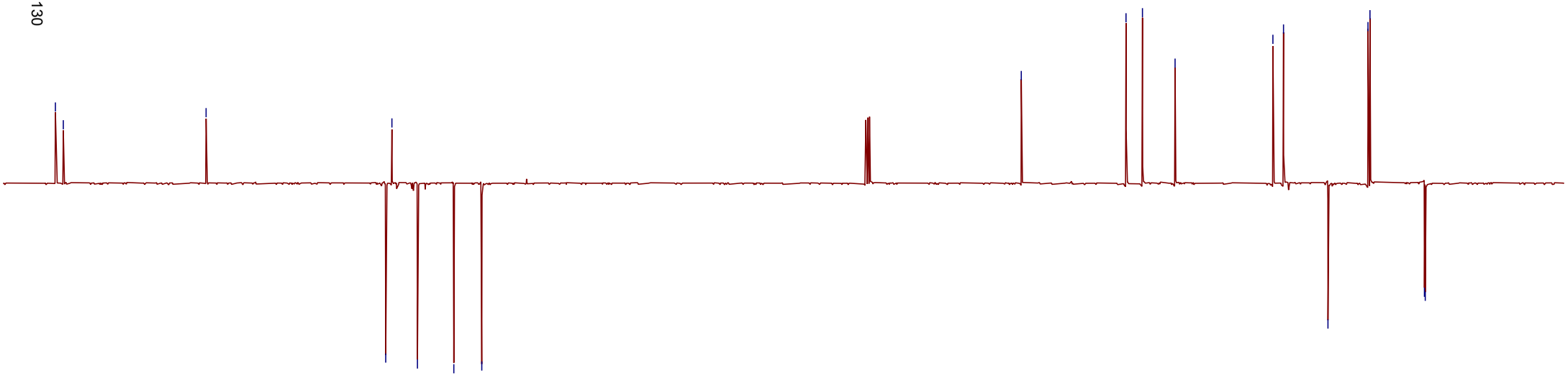
24.699
20.151
19.926
13.738
13.643



48

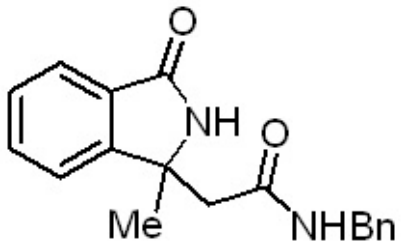
	Parameter	Value
1	Title	grd-2-273.2.fid
2	Solvent	CDCl3
3	Temperature	300.0
4	Number of Scans	256
5	Receiver Gain	2050.0
6	Relaxation Delay	5.0000
7	Pulse Width	10.6300
8	Spectrometer Frequency	150.97
9	Nucleus	13C

S130



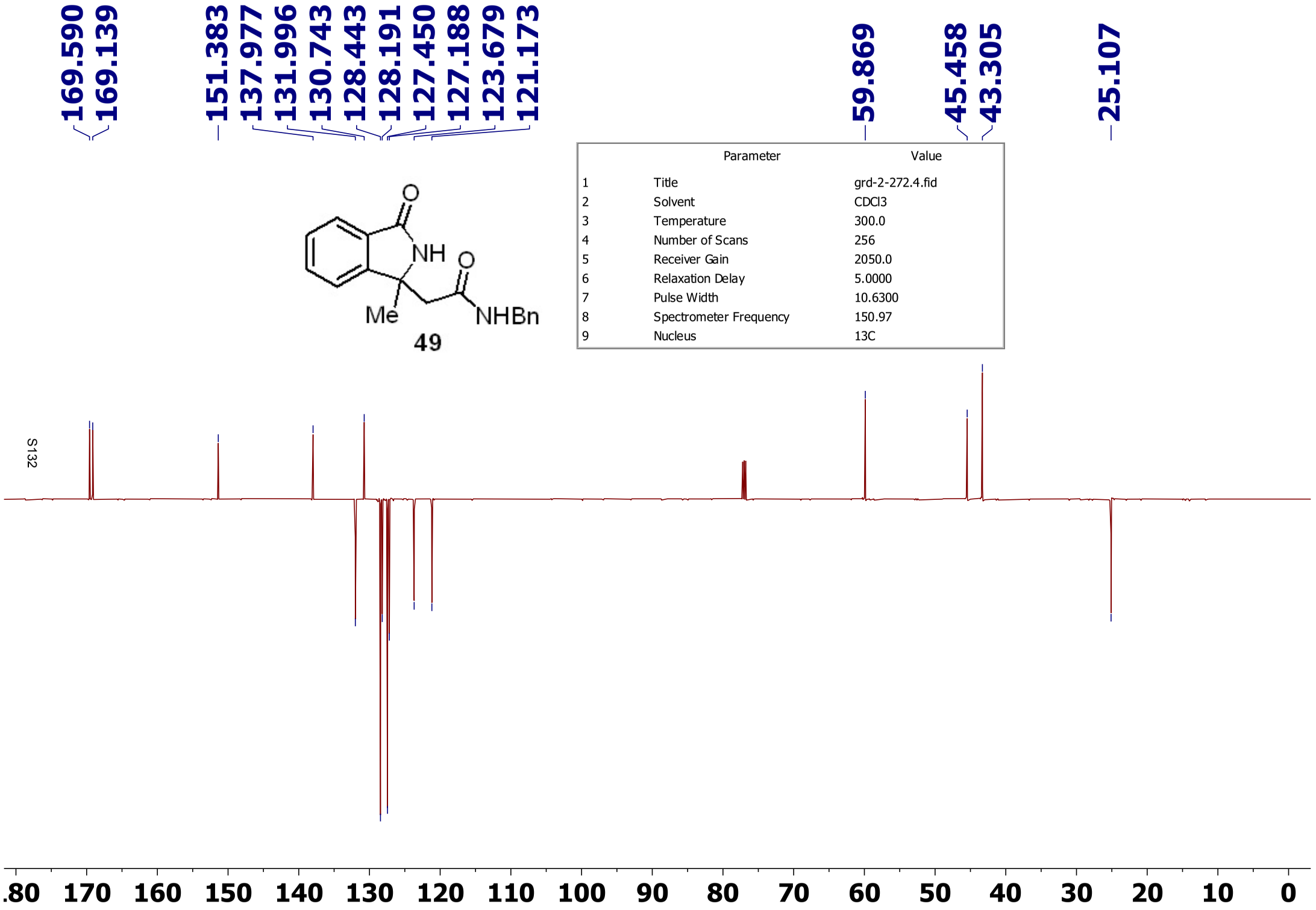
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

7.645 7.633 7.580 7.454 7.452 7.441 7.440 7.429 7.427 7.344 7.343 7.332 7.331 7.319 7.318 7.274 7.262 7.210 7.199 7.189 7.186 7.166 7.154 7.116 7.104 6.530 4.352 4.337 4.327 4.291 4.282 4.266 2.746 2.721 2.369 2.345 1.500

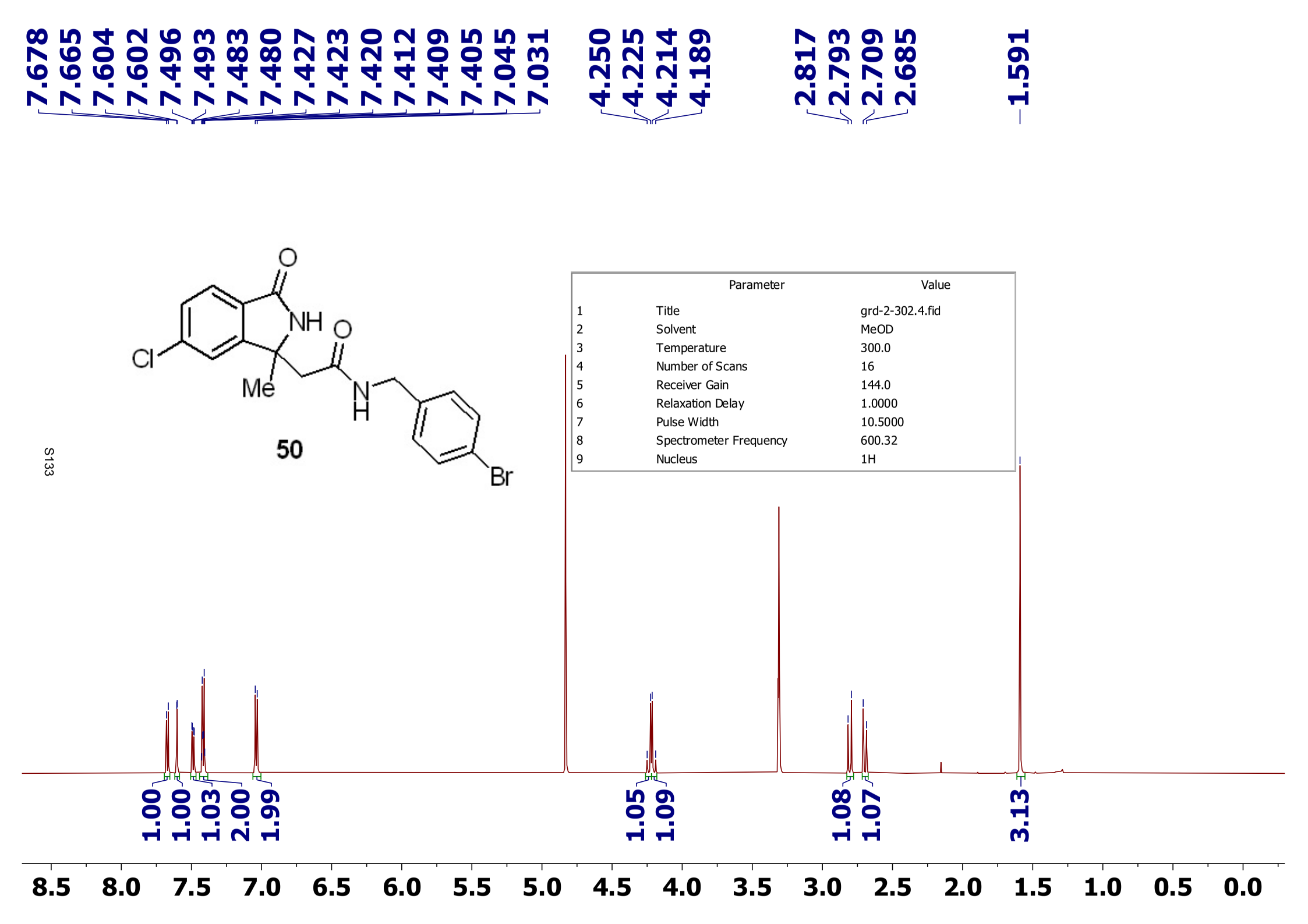


49

	Parameter	Value
1	Title	grd-2-272.5.fid
2	Solvent	CDCl3
3	Temperature	300.0
4	Number of Scans	16
5	Receiver Gain	57.0
6	Relaxation Delay	1.0000
7	Pulse Width	10.5000
8	Spectrometer Frequency	600.32
9	Nucleus	1H



	Parameter	Value
1	Title	grd-2-272.4.fid
2	Solvent	CDCl3
3	Temperature	300.0
4	Number of Scans	256
5	Receiver Gain	2050.0
6	Relaxation Delay	5.0000
7	Pulse Width	10.6300
8	Spectrometer Frequency	150.97
9	Nucleus	13C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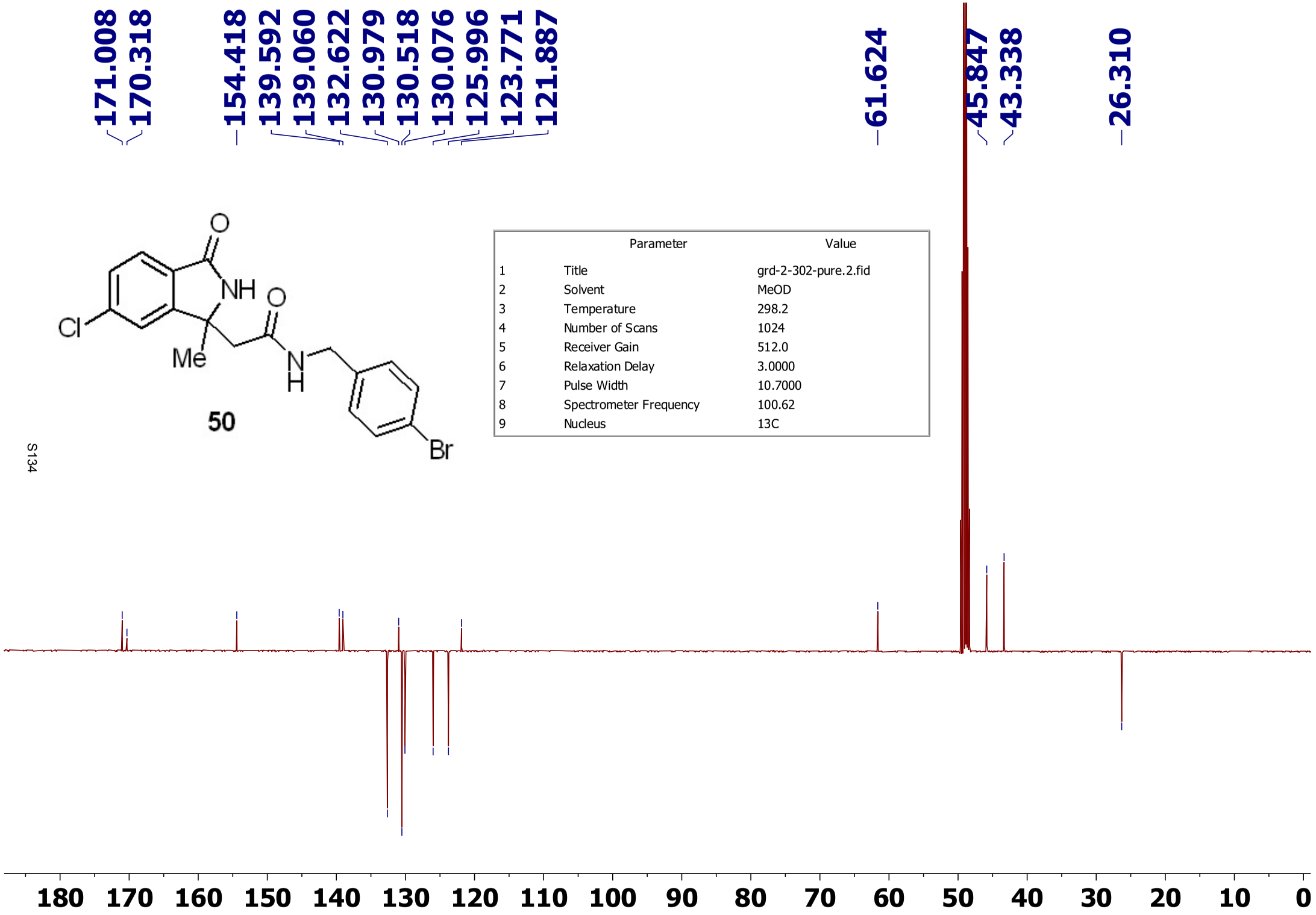
7.602
7.496
7.493
7.483
7.480
7.427
7.423
7.420
7.412
7.409
7.405
7.045
7.031
4.250
4.225
4.214
4.189
2.817
2.793
2.709
2.685
1.591
O
NH
O
Cl
Me
N
H
Br
50
S133
Parameter Value
1 Title grd-2-302.4.fid
2 Solvent MeOD
3 Temperature 300.0
4 Number of Scans 16
5 Receiver Gain 144.0
6 Relaxation Delay 1.0000
7 Pulse Width 10.5000
8 Spectrometer Frequency 600.32
9 Nucleus 1H
1.00
1.00
1.03
2.00
1.99
1.05
1.09
1.08
1.07
3.13
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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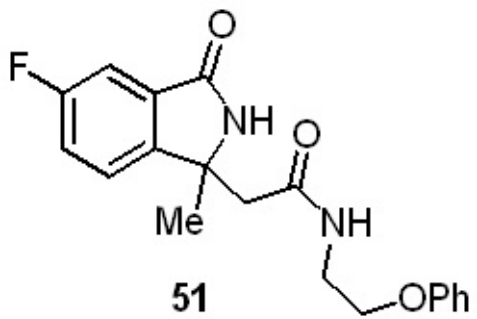
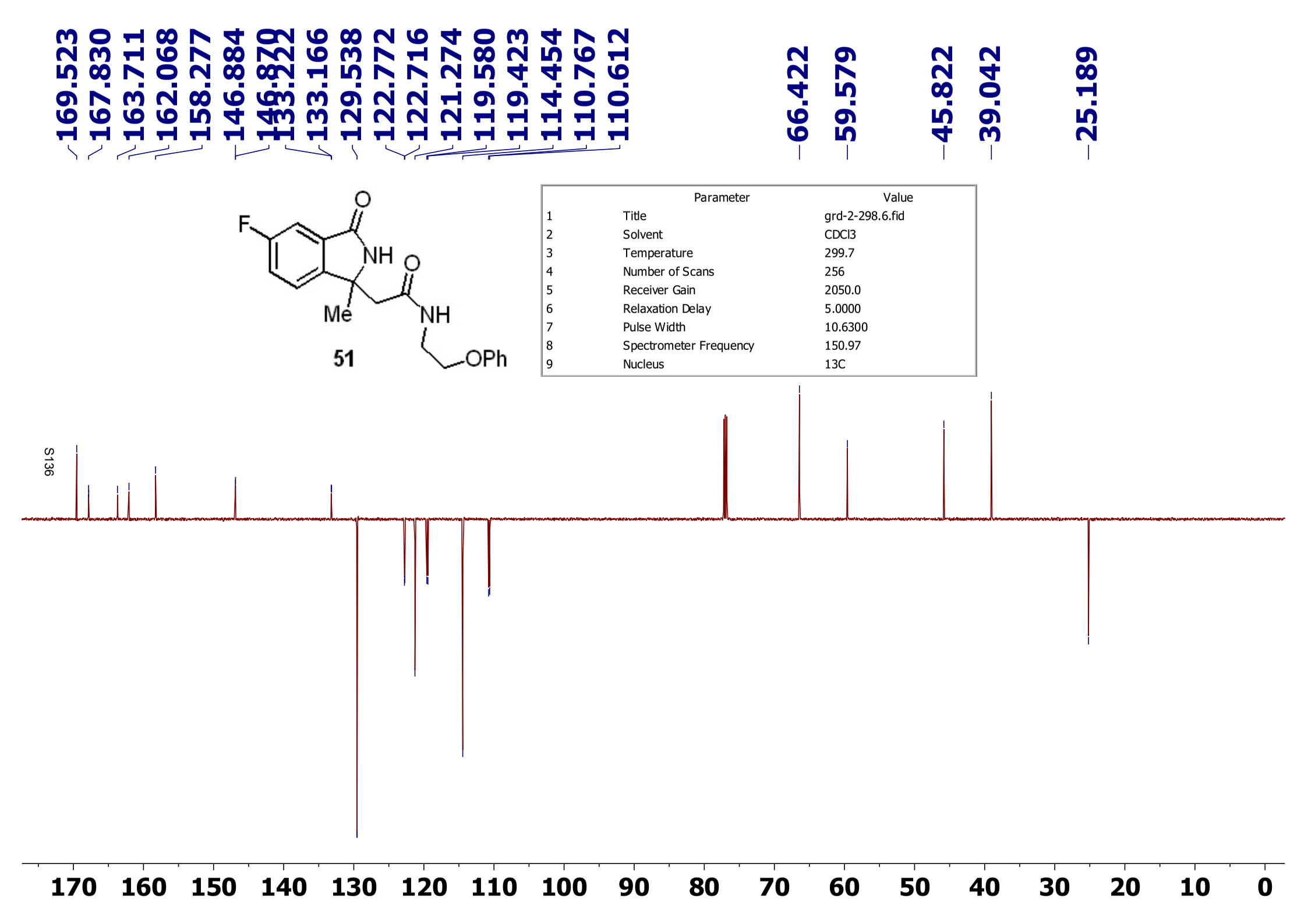
170.318
154.418
139.592
139.060
132.622
130.979
130.518
130.076
125.996
123.771
121.887
61.624
45.847
43.338
26.310
O
NH
O
Cl
Me
N
H
Br
50
S134
Parameter Value
1 Title grd-2-302-pure.2.fid
2 Solvent MeOD
3 Temperature 298.2
4 Number of Scans 1024
5 Receiver Gain 512.0
6 Relaxation Delay 3.0000
7 Pulse Width 10.7000
8 Spectrometer Frequency 100.62
9 Nucleus 13C
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

7.806 7.417 7.413 7.404 7.401 7.351 7.344 7.337 7.330 7.267 7.255 7.241 7.216 7.212 7.201 7.198 6.958 6.945 6.933 6.848 6.835 6.498 3.994 3.986 3.983 3.976 3.973 3.965 3.657 3.648 3.641 3.634 2.799 2.775 2.449 2.425 1.565



51

	Parameter	Value
1	Title	grd-2-298.5.fid
2	Solvent	CDCl3
3	Temperature	297.3
4	Number of Scans	16
5	Receiver Gain	57.0
6	Relaxation Delay	1.0000
7	Pulse Width	10.5000
8	Spectrometer Frequency	600.32
9	Nucleus	1H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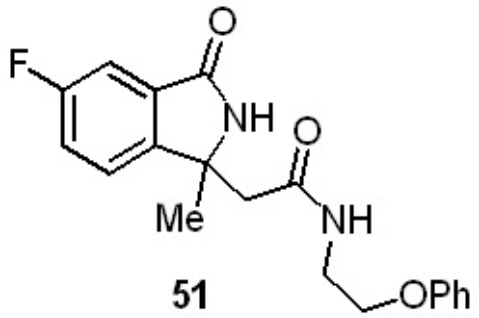
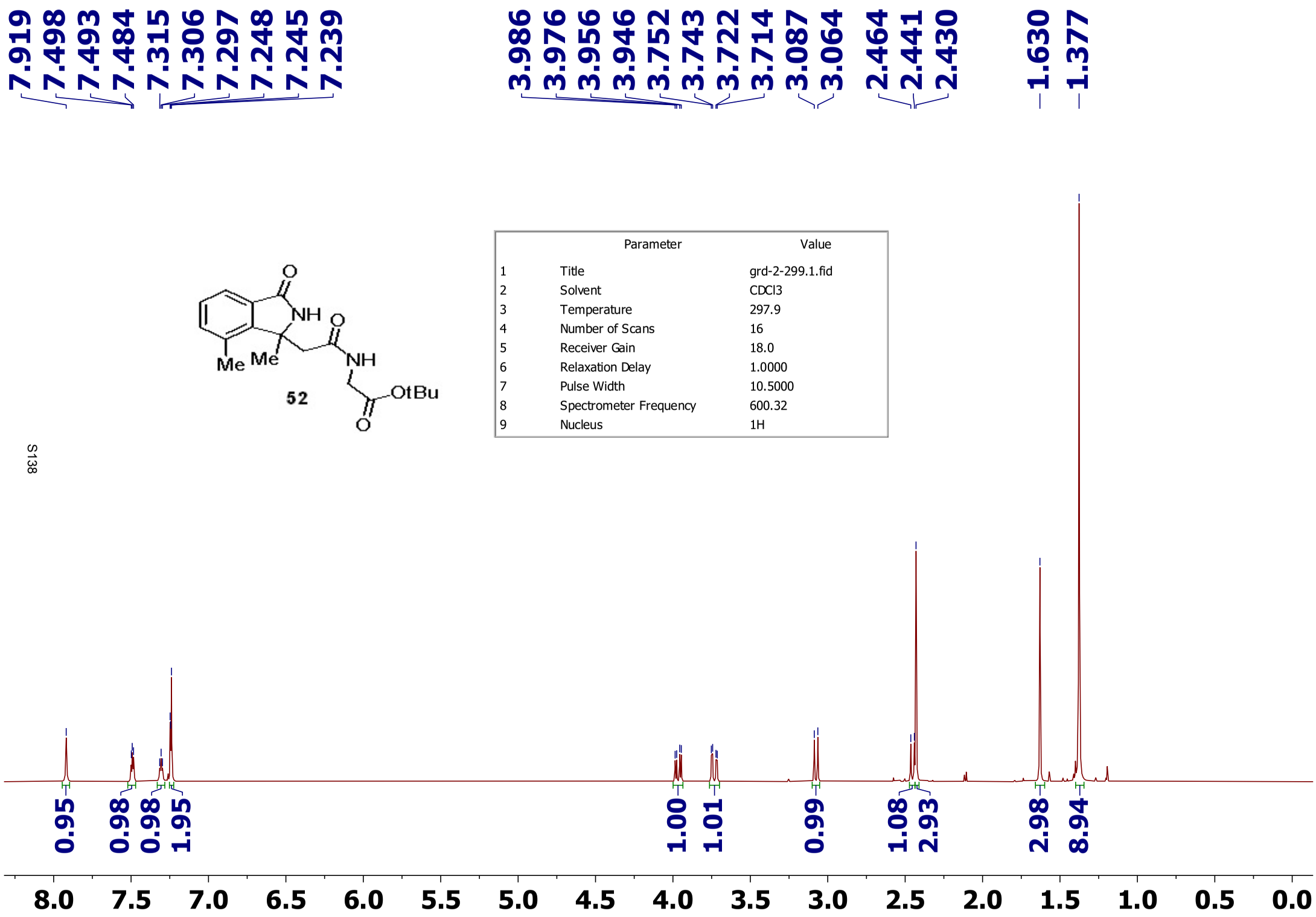
163.711
162.068
158.277
146.884
146.870
133.222
133.166
129.538
122.772
122.716
121.274
119.580
119.423
114.454
110.767
110.612
66.422
59.579
45.822
39.042
25.189
F
O
NH
O
Me
NH
OPh
51
Parameter
Value
1 Title grd-2-298.6.fid
2 Solvent CDCl3
3 Temperature 299.7
4 Number of Scans 256
5 Receiver Gain 2050.0
6 Relaxation Delay 5.0000
7 Pulse Width 10.6300
8 Spectrometer Frequency 150.97
9 Nucleus 13C
S136
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

-112.550
-112.564
-112.571
-112.585

	Parameter	Value
1	Title	grd-2-298.2.fid
2	Solvent	CDCl3
3	Temperature	298.1
4	Number of Scans	32
5	Receiver Gain	181.0
6	Relaxation Delay	3.0000
7	Pulse Width	11.4000
8	Spectrometer Frequency	564.81
9	Nucleus	19F



51


7.919
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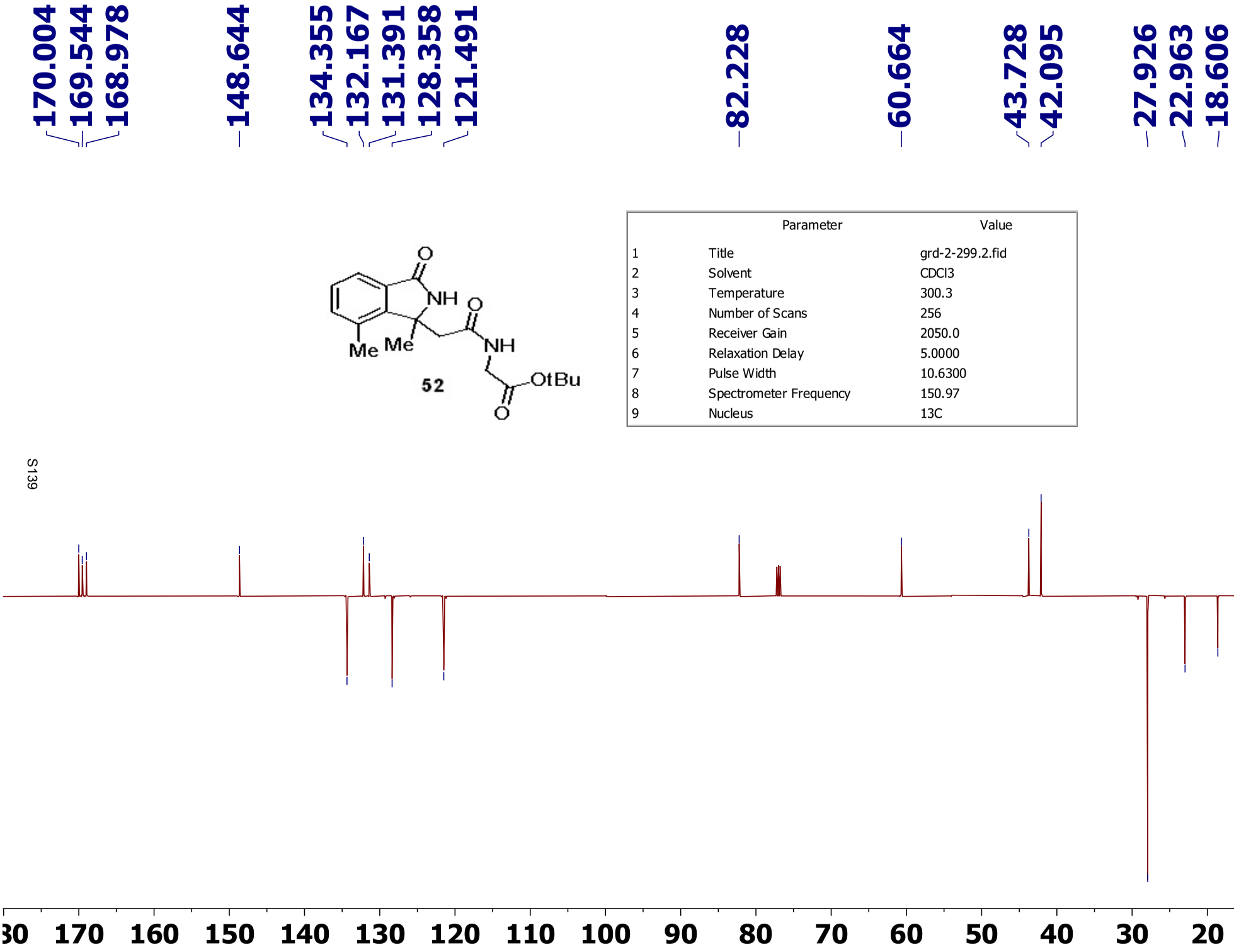
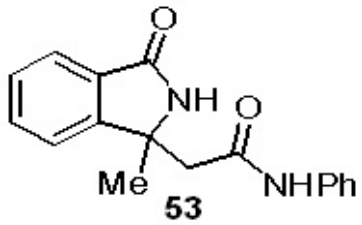
7.493
7.484
7.315
7.306
7.297
7.248
7.245
7.239
3.986
3.976
3.956
3.946
3.752
3.743
3.722
3.714
3.087
3.064
2.464
2.441
2.430
1.630
1.377
Me
Me
NH
NH
O
O
O
O
OtBu
52
S138
Parameter Value
1 Title grd-2-299.1.fid
2 Solvent CDCl3
3 Temperature 297.9
4 Number of Scans 16
5 Receiver Gain 18.0
6 Relaxation Delay 1.0000
7 Pulse Width 10.5000
8 Spectrometer Frequency 600.32
9 Nucleus 1H
0.95
0.98
0.98
1.95
1.00
1.01
0.99
1.08
2.93
2.98
8.94
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

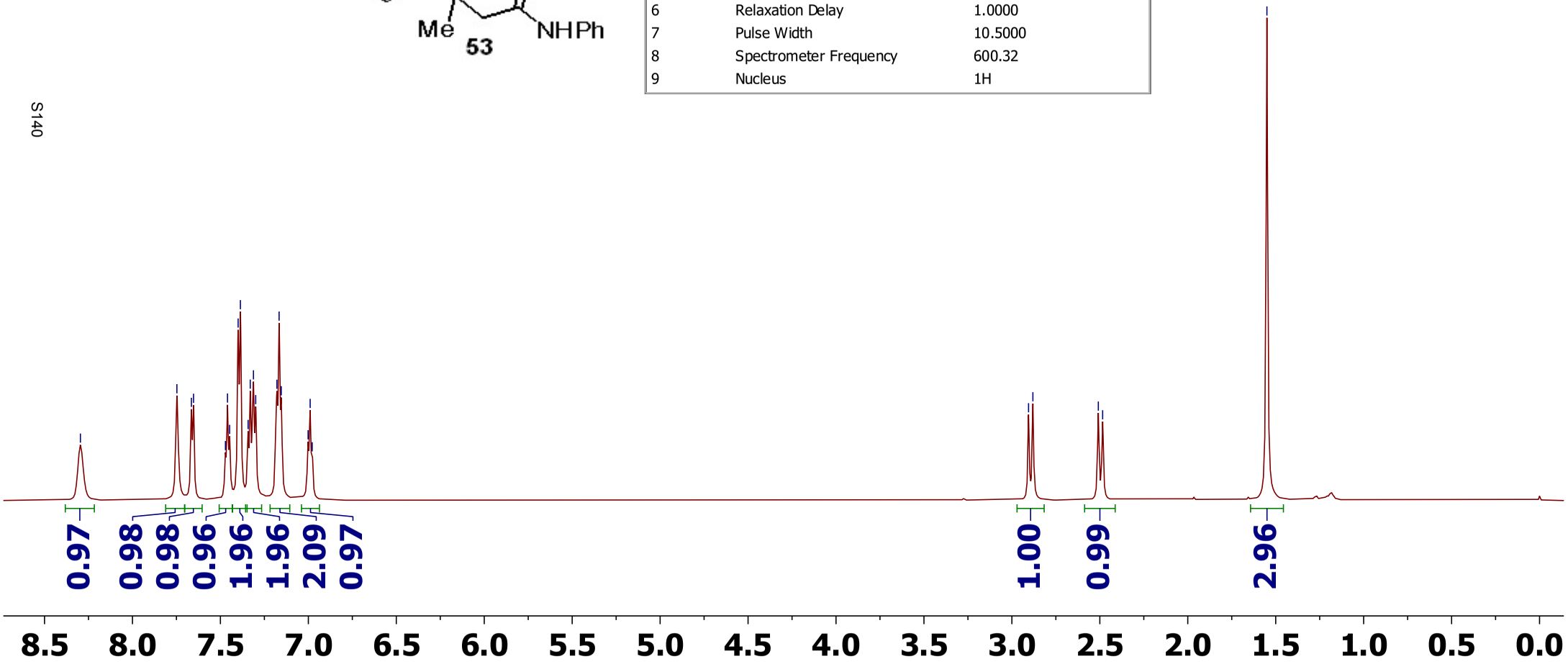


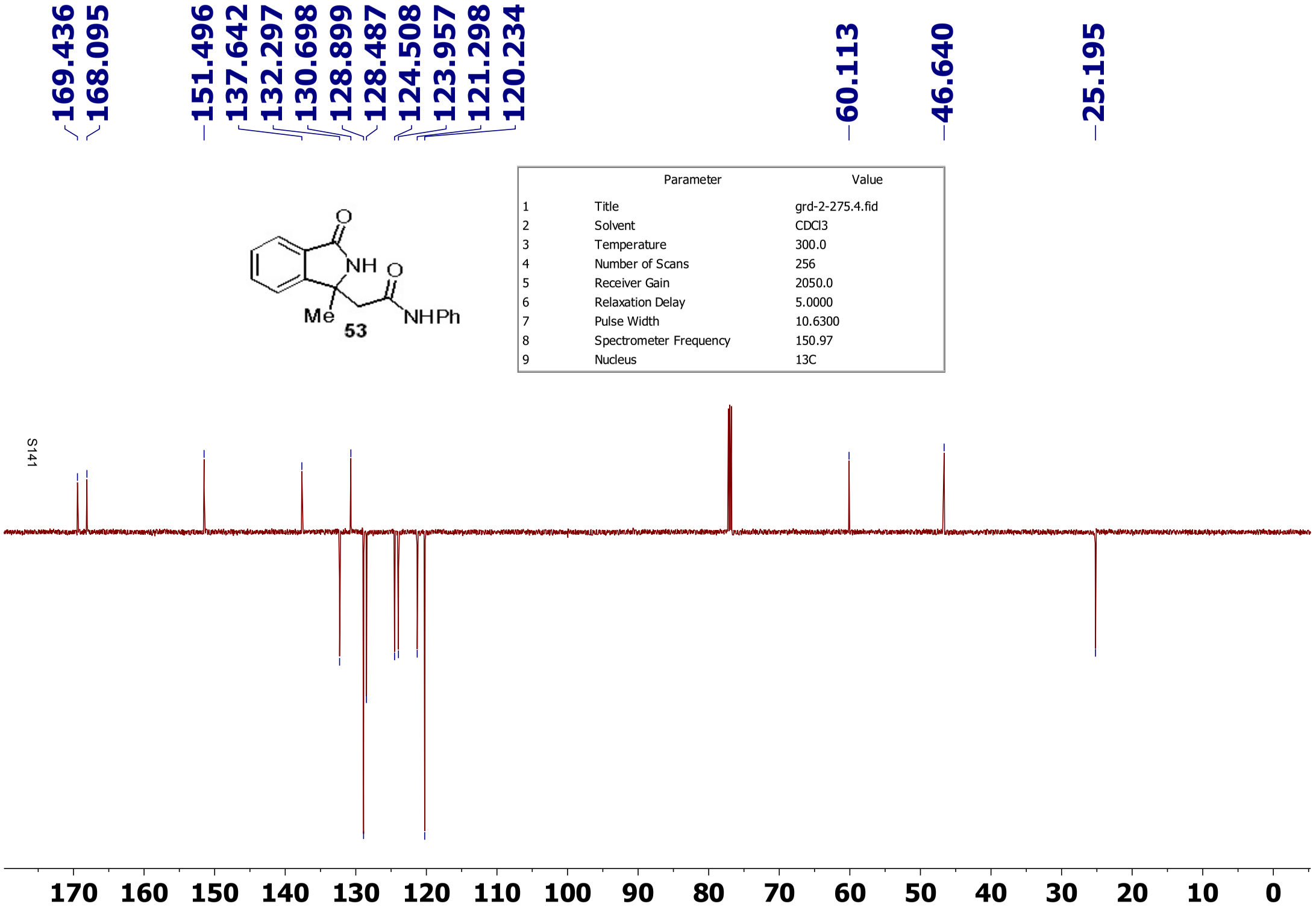
	Parameter	Value
1	Title	grd-2-299.2.fid
2	Solvent	CDCl3
3	Temperature	300.3
4	Number of Scans	256
5	Receiver Gain	2050.0
6	Relaxation Delay	5.0000
7	Pulse Width	10.6300
8	Spectrometer Frequency	150.97
9	Nucleus	13C



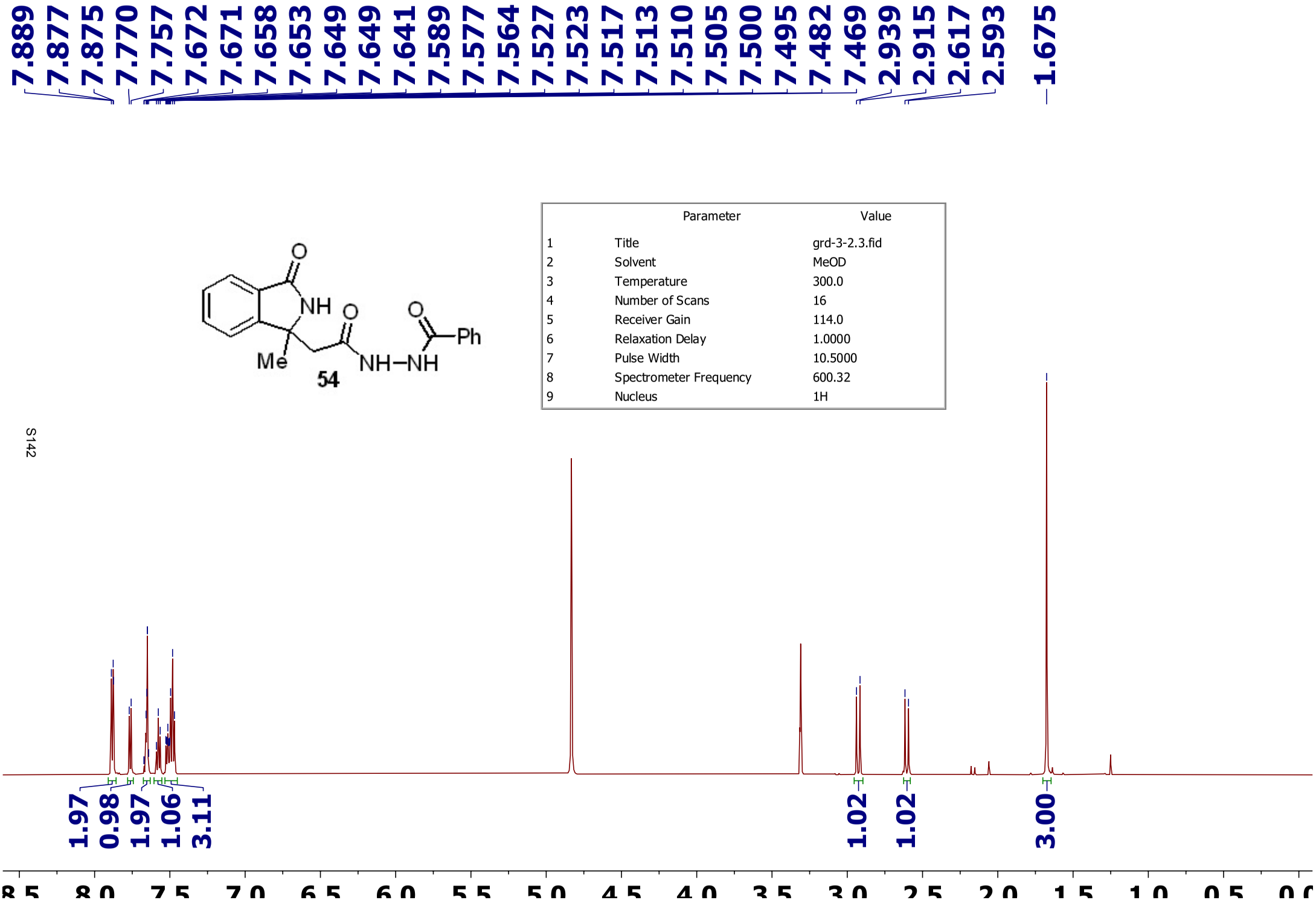
53

	Parameter	Value
1	Title	grd-2-275.2.fid
2	Solvent	CDCl3
3	Temperature	300.0
4	Number of Scans	16
5	Receiver Gain	57.0
6	Relaxation Delay	1.0000
7	Pulse Width	10.5000
8	Spectrometer Frequency	600.32
9	Nucleus	1H





	Parameter	Value
1	Title	grd-2-275.4.fid
2	Solvent	CDCl3
3	Temperature	300.0
4	Number of Scans	256
5	Receiver Gain	2050.0
6	Relaxation Delay	5.0000
7	Pulse Width	10.6300
8	Spectrometer Frequency	150.97
9	Nucleus	13C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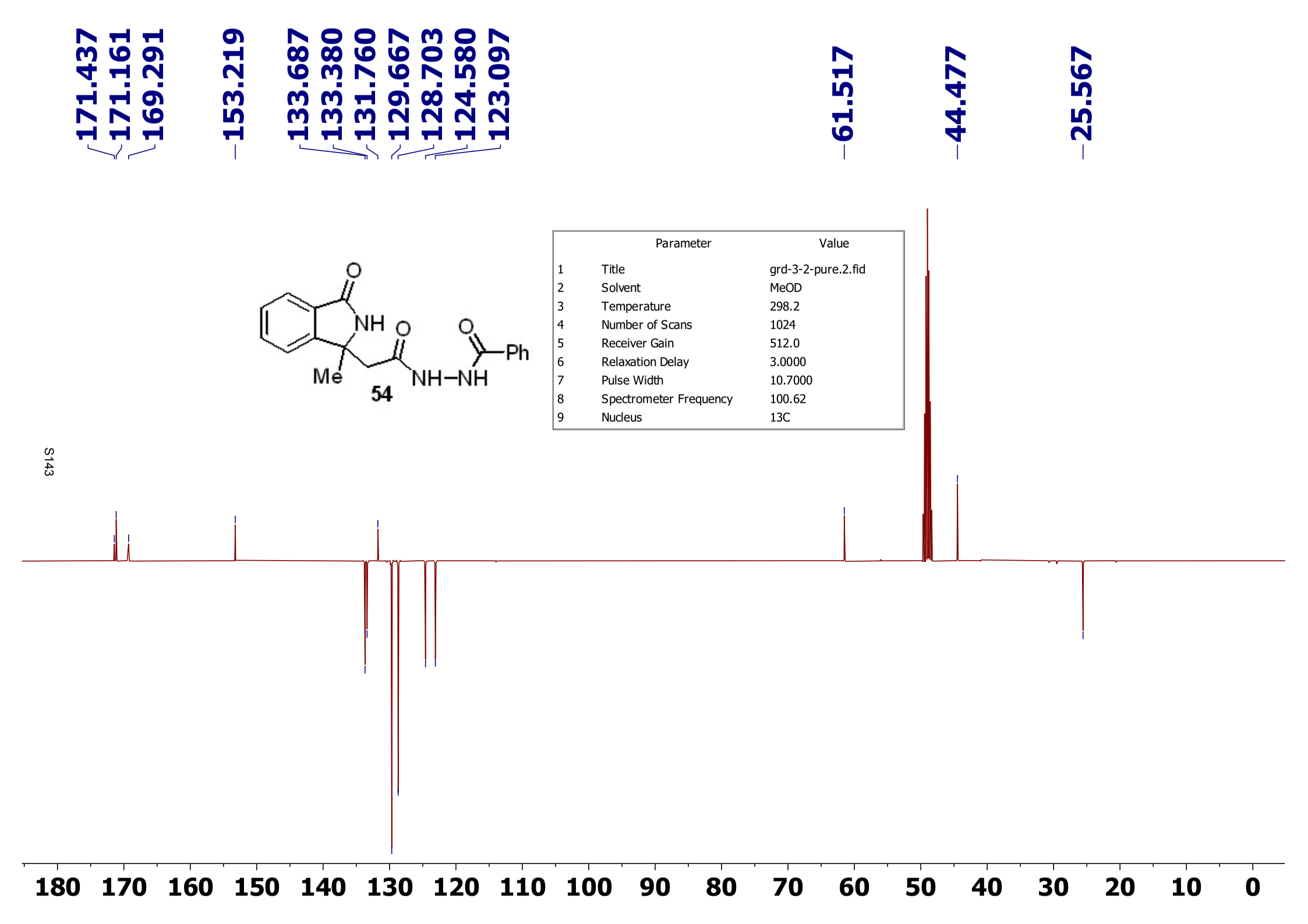
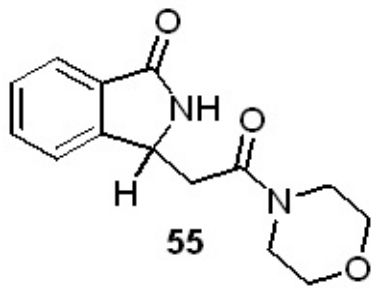
7.877
7.875
7.770
7.757
7.672
7.671
7.658
7.653
7.649
7.649
7.641
7.589
7.577
7.564
7.527
7.523
7.517
7.513
7.510
7.505
7.500
7.495
7.482
7.469
2.939
2.915
2.617
2.593
1.675
O
NH
O
O
Ph
Me
NH–NH
54
Parameter Value
1 Title grd-3-2.3.fid
2 Solvent MeOD
3 Temperature 300.0
4 Number of Scans 16
5 Receiver Gain 114.0
6 Relaxation Delay 1.0000
7 Pulse Width 10.5000
8 Spectrometer Frequency 600.32
9 Nucleus 1H
S142
1.97
0.98
1.97
1.06
3.11
1.02
1.02
3.00
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



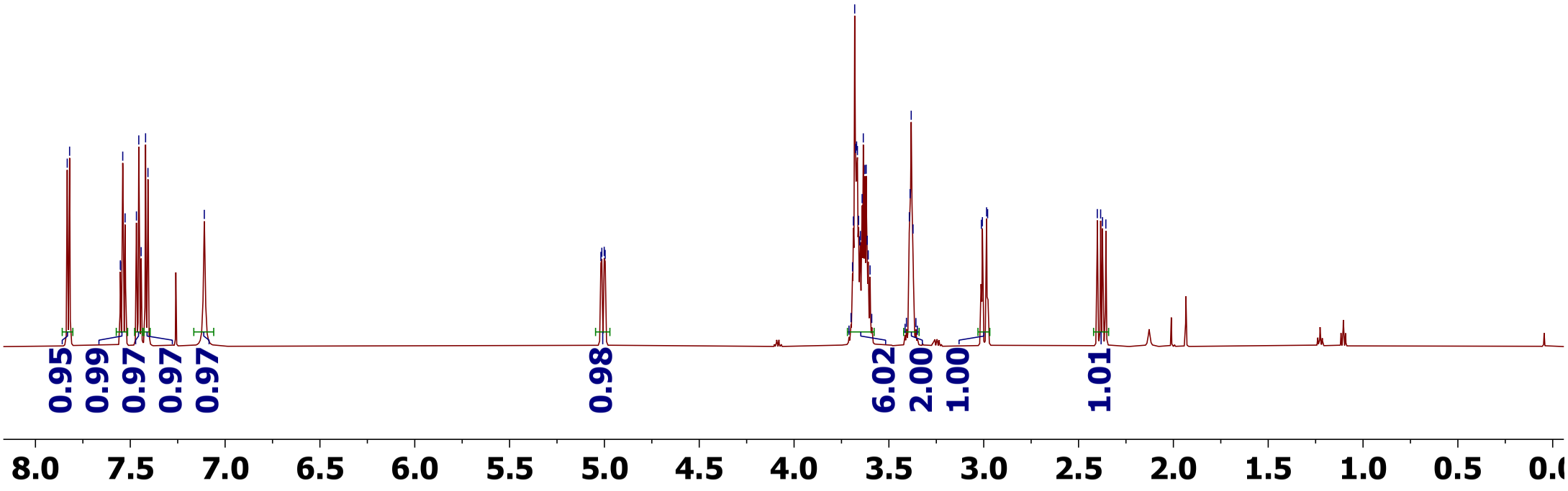
	Parameter	Value
1	Title	grd-3-2-pure.2.fid
2	Solvent	MeOD
3	Temperature	298.2
4	Number of Scans	1024
5	Receiver Gain	512.0
6	Relaxation Delay	3.0000
7	Pulse Width	10.7000
8	Spectrometer Frequency	100.62
9	Nucleus	13C

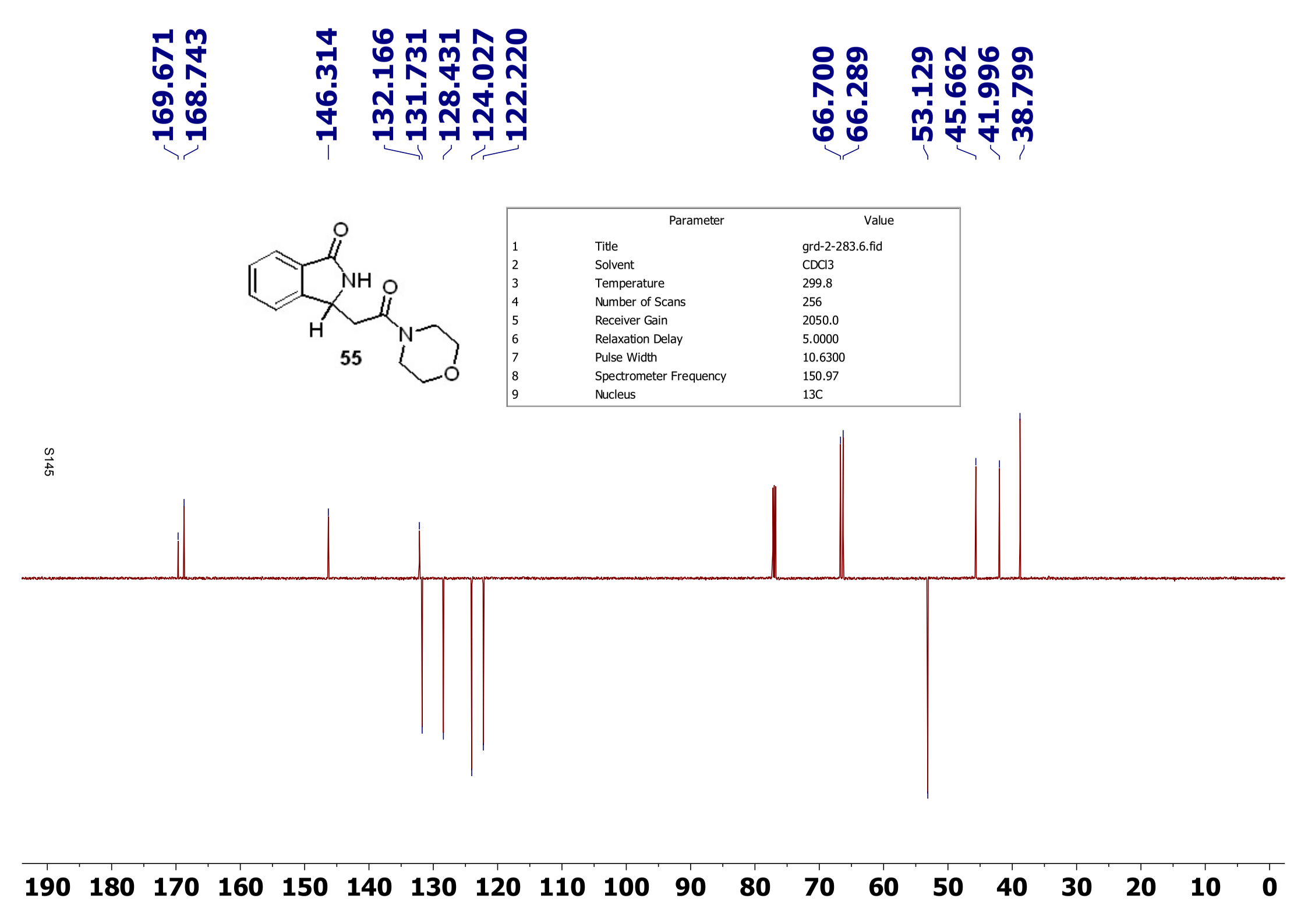
7.833 7.820 7.540 7.527 7.468 7.455 7.443 7.419 7.407 7.110 5.014 5.001 3.687 3.680 3.675 3.670 3.666 3.660 3.653 3.650 3.641 3.635 3.627 3.620 3.614 3.392 3.388 3.382 3.373 3.012 3.007 2.985 2.979 2.401 2.384 2.374 2.356



55

	Parameter	Value
1	Title	grd-2-283.5.fid
2	Solvent	CDCl3
3	Temperature	297.3
4	Number of Scans	16
5	Receiver Gain	57.0
6	Relaxation Delay	1.0000
7	Pulse Width	10.5000
8	Spectrometer Frequency	600.32
9	Nucleus	1H

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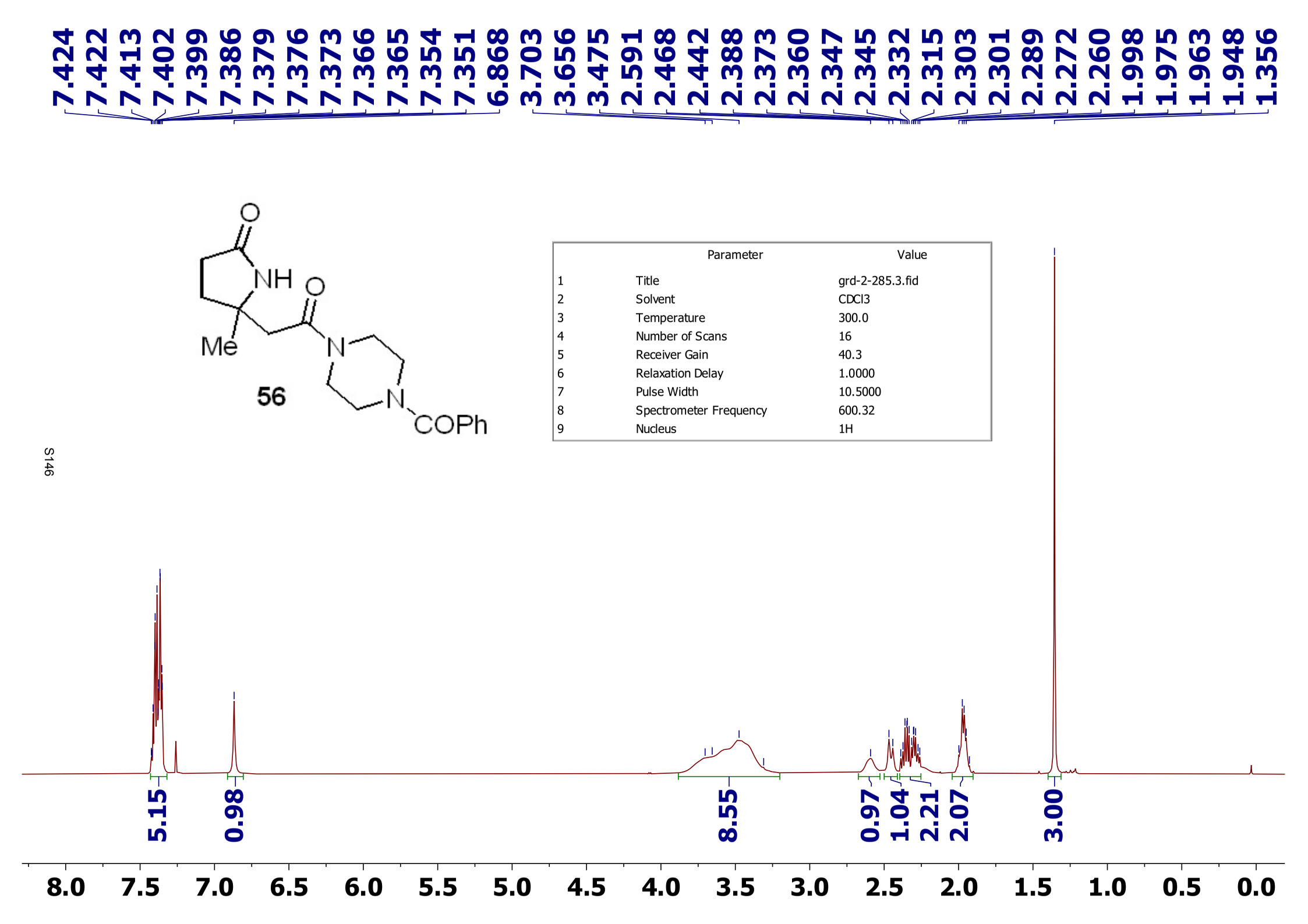
146.314
132.166
131.731
128.431
124.027
122.220
66.700
66.289
53.129
45.662
41.996
38.799
O
NH
O
H
N
O
55
Parameter
Value
1 Title grd-2-283.6.fid
2 Solvent CDCl3
3 Temperature 299.8
4 Number of Scans 256
5 Receiver Gain 2050.0
6 Relaxation Delay 5.0000
7 Pulse Width 10.6300
8 Spectrometer Frequency 150.97
9 Nucleus 13C
S145
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0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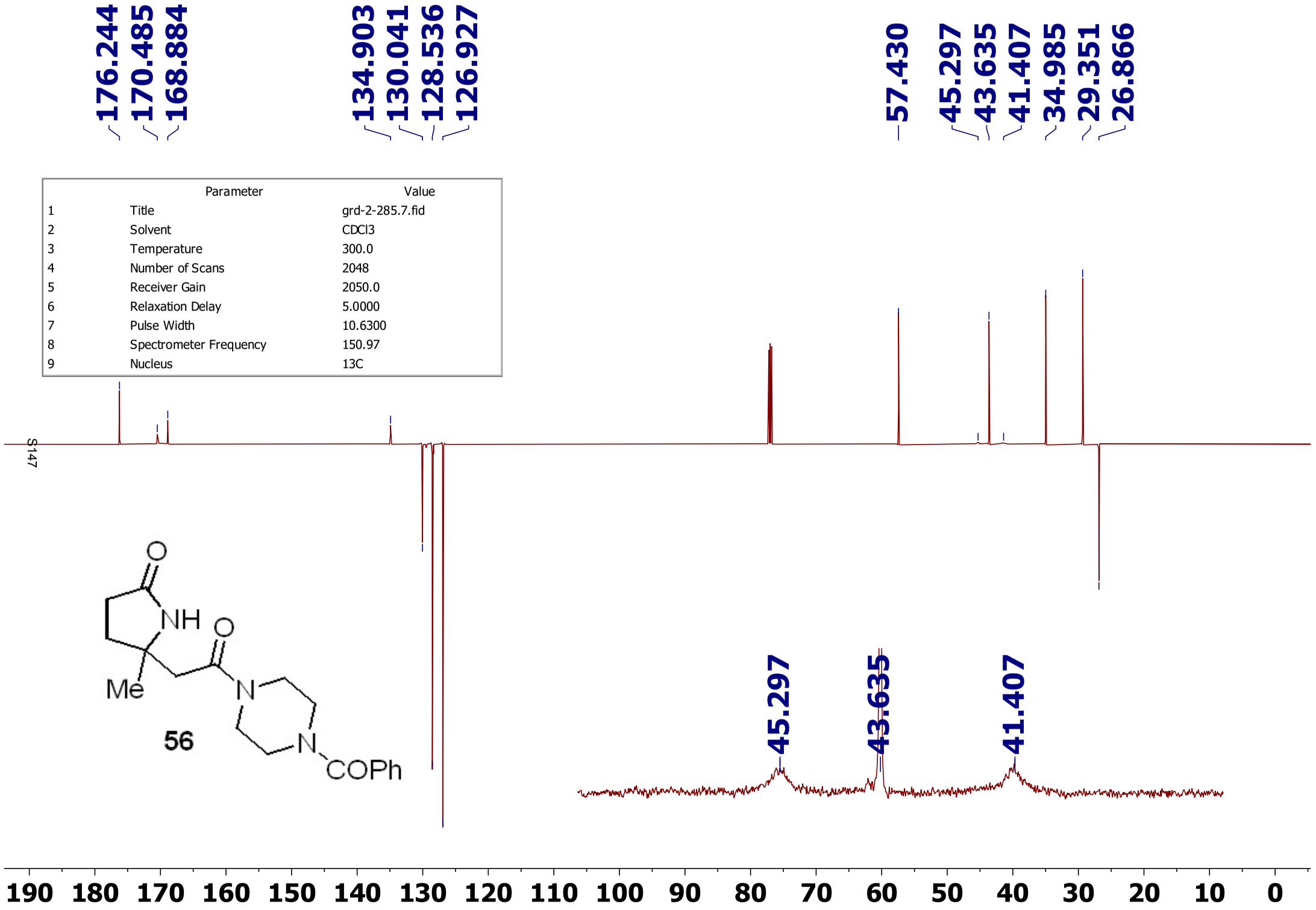
7.422
7.413
7.402
7.399
7.386
7.379
7.376
7.373
7.366
7.365
7.354
7.351
6.868
3.703
3.656
3.475
2.591
2.468
2.442
2.388
2.373
2.360
2.347
2.345
2.332
2.315
2.303
2.301
2.289
2.272
2.260
1.998
1.975
1.963
1.948
1.356
O
NH
O
Me
N
N
COPh
56
Parameter
Value
1 Title grd-2-285.3.fid
2 Solvent CDCl3
3 Temperature 300.0
4 Number of Scans 16
5 Receiver Gain 40.3
6 Relaxation Delay 1.0000
7 Pulse Width 10.5000
8 Spectrometer Frequency 600.32
9 Nucleus 1H
5.15
0.98
8.55
0.97
1.04
2.21
2.07
3.00
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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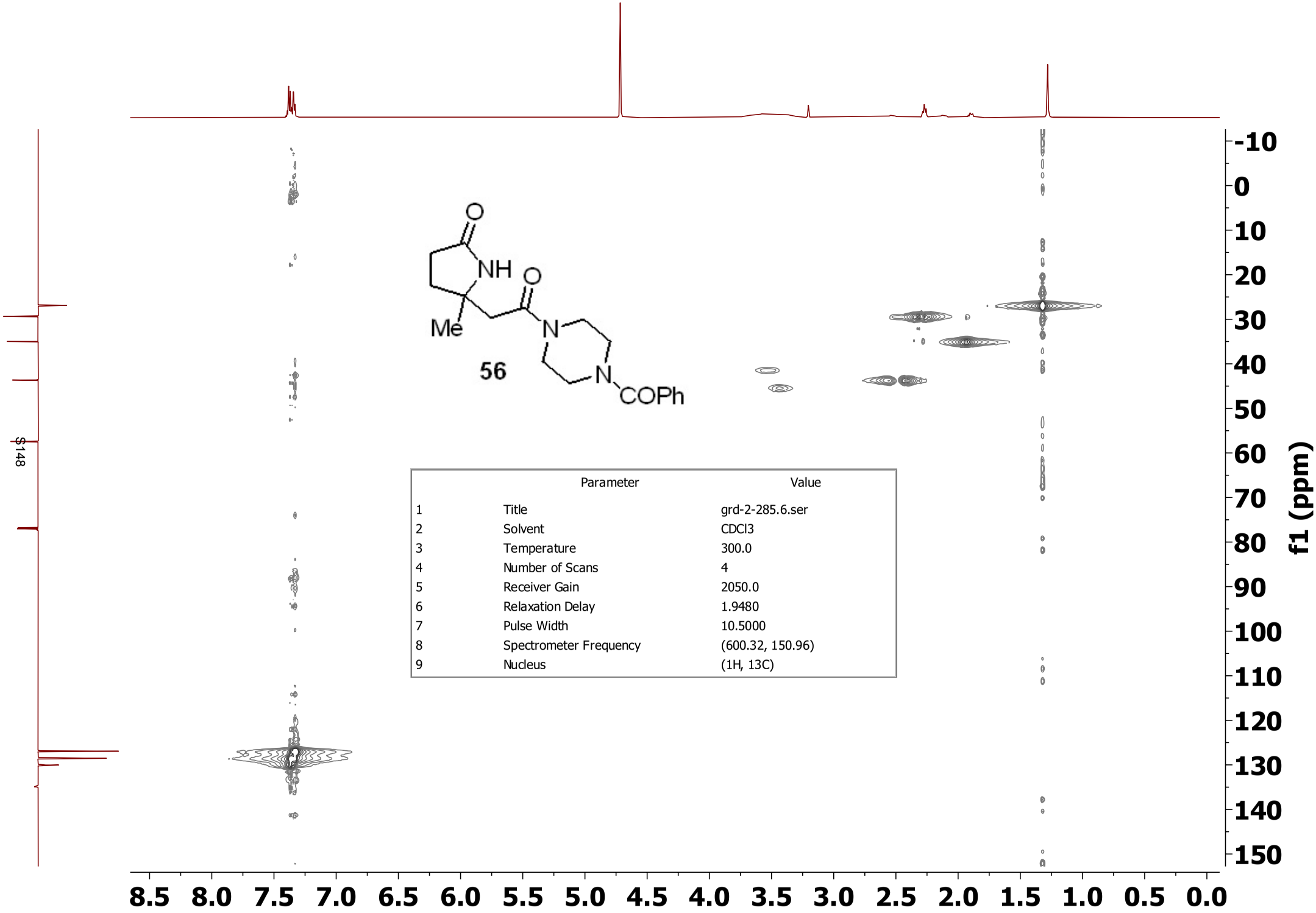
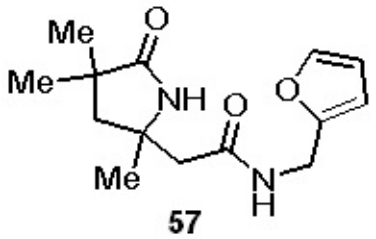
170.485
168.884
134.903
130.041
128.536
126.927
57.430
45.297
43.635
41.407
34.985
29.351
26.866
Parameter Value
1 Title grd-2-285.7.fid
2 Solvent CDCl3
3 Temperature 300.0
4 Number of Scans 2048
5 Receiver Gain 2050.0
6 Relaxation Delay 5.0000
7 Pulse Width 10.6300
8 Spectrometer Frequency 150.97
9 Nucleus 13C
S147
O
NH
O
Me
N
N
COPh
56
45.297
43.635
41.407
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

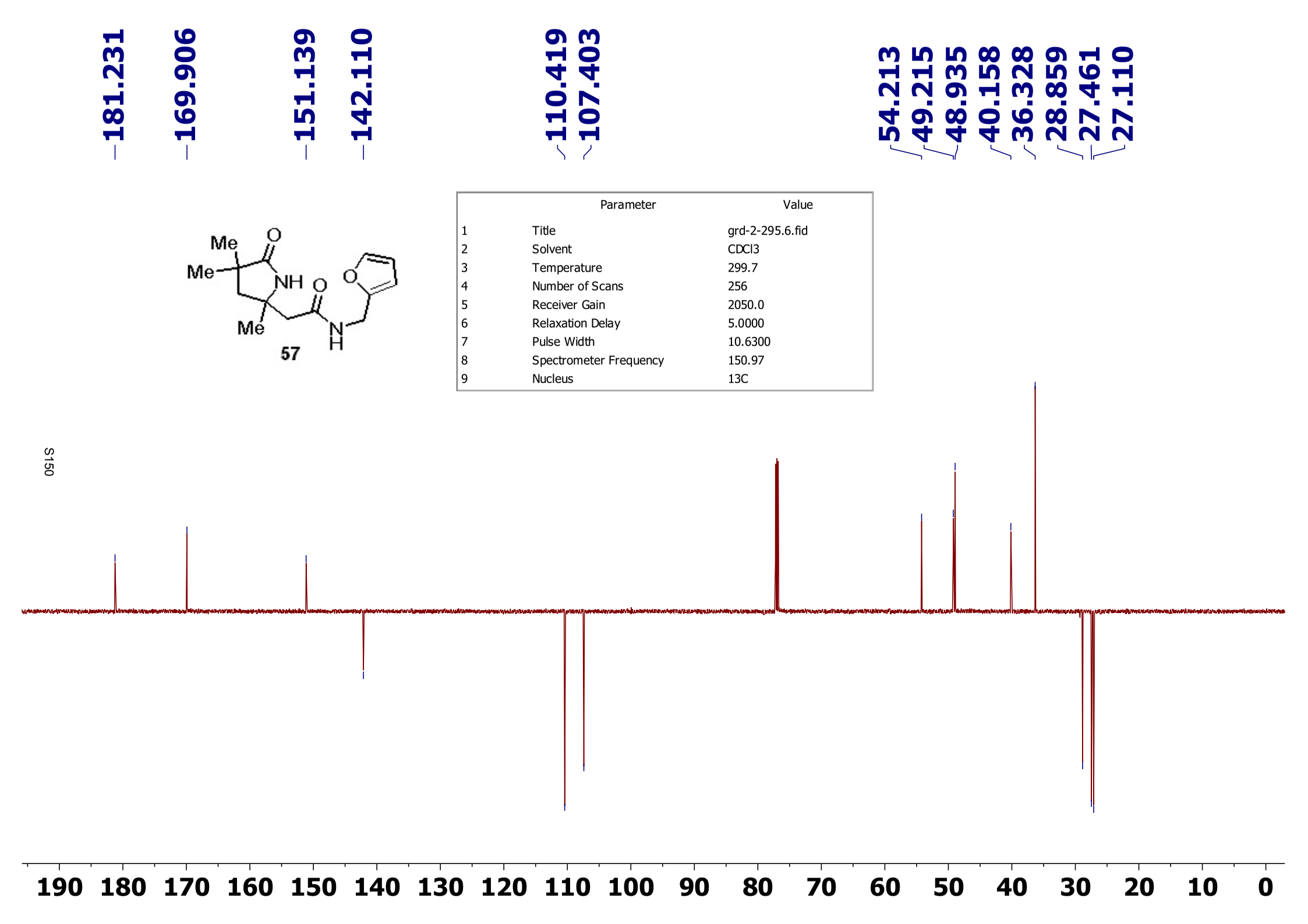


	Parameter	Value
1	Title	grd-2-285.6.ser
2	Solvent	CDCl3
3	Temperature	300.0
4	Number of Scans	4
5	Receiver Gain	2050.0
6	Relaxation Delay	1.9480
7	Pulse Width	10.5000
8	Spectrometer Frequency	(600.32, 150.96)
9	Nucleus	(1H, 13C)



57

	Parameter	Value
1	Title	grd-2-295.5.fid
2	Solvent	CDCl3
3	Temperature	297.2
4	Number of Scans	16
5	Receiver Gain	57.0
6	Relaxation Delay	1.0000
7	Pulse Width	10.5000
8	Spectrometer Frequency	600.32
9	Nucleus	1H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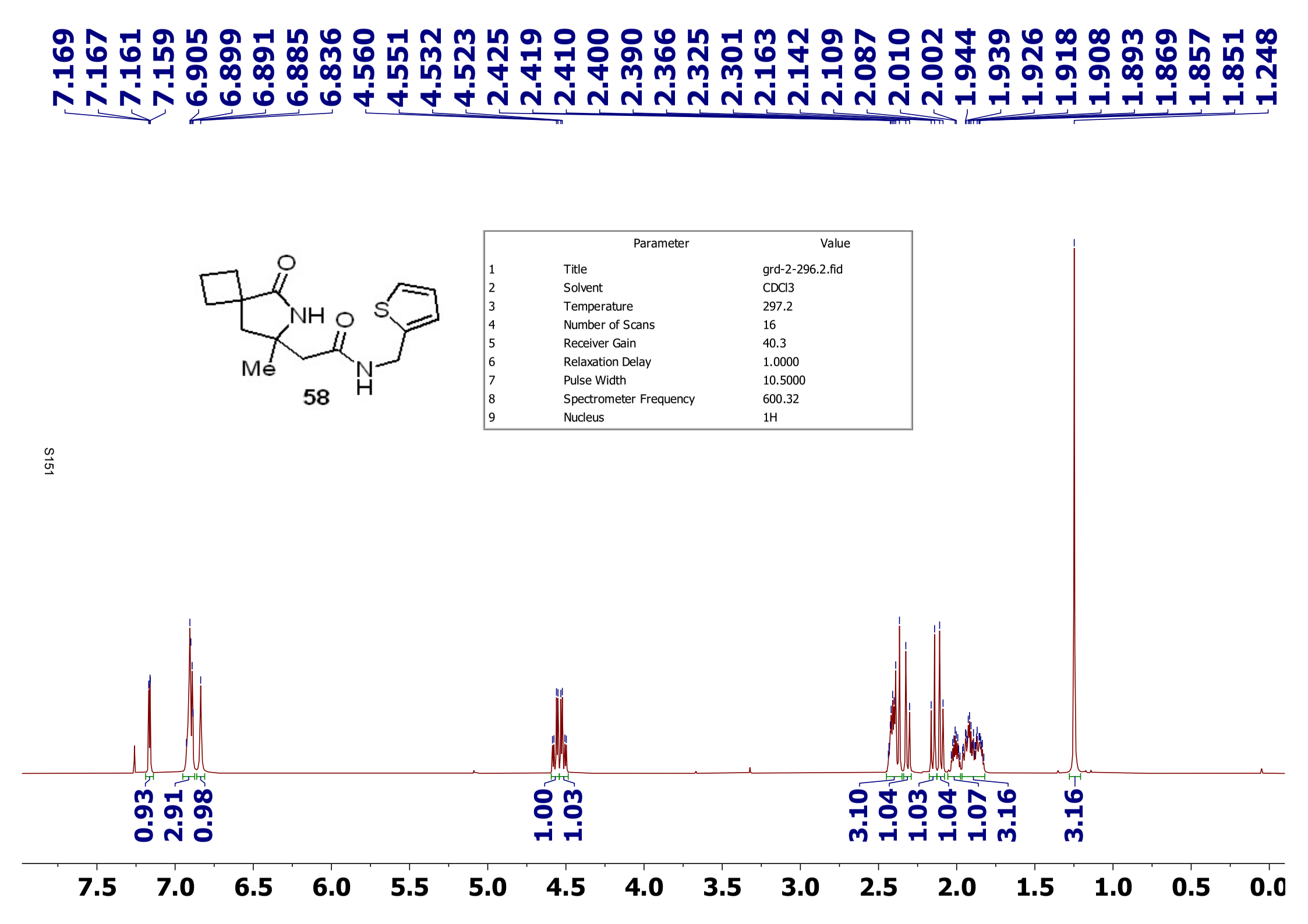
142.110
110.419
107.403
54.213
49.215
48.935
40.158
36.328
28.859
27.461
27.110
Me
Me
Me
O
NH
O
N
H
O
57
Parameter Value
1 Title grd-2-295.6.fid
2 Solvent CDCl3
3 Temperature 299.7
4 Number of Scans 256
5 Receiver Gain 2050.0
6 Relaxation Delay 5.0000
7 Pulse Width 10.6300
8 Spectrometer Frequency 150.97
9 Nucleus 13C
S150
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0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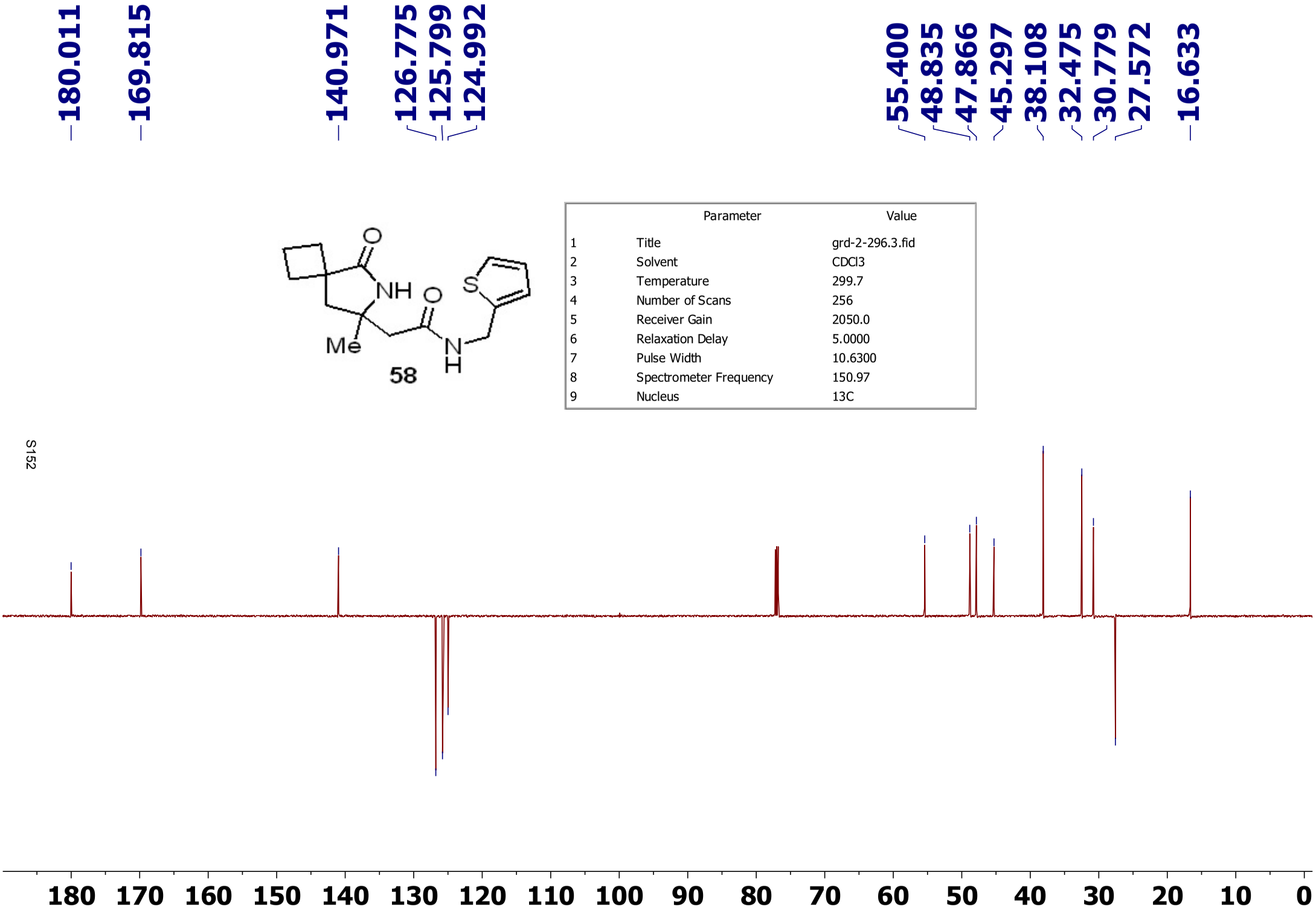
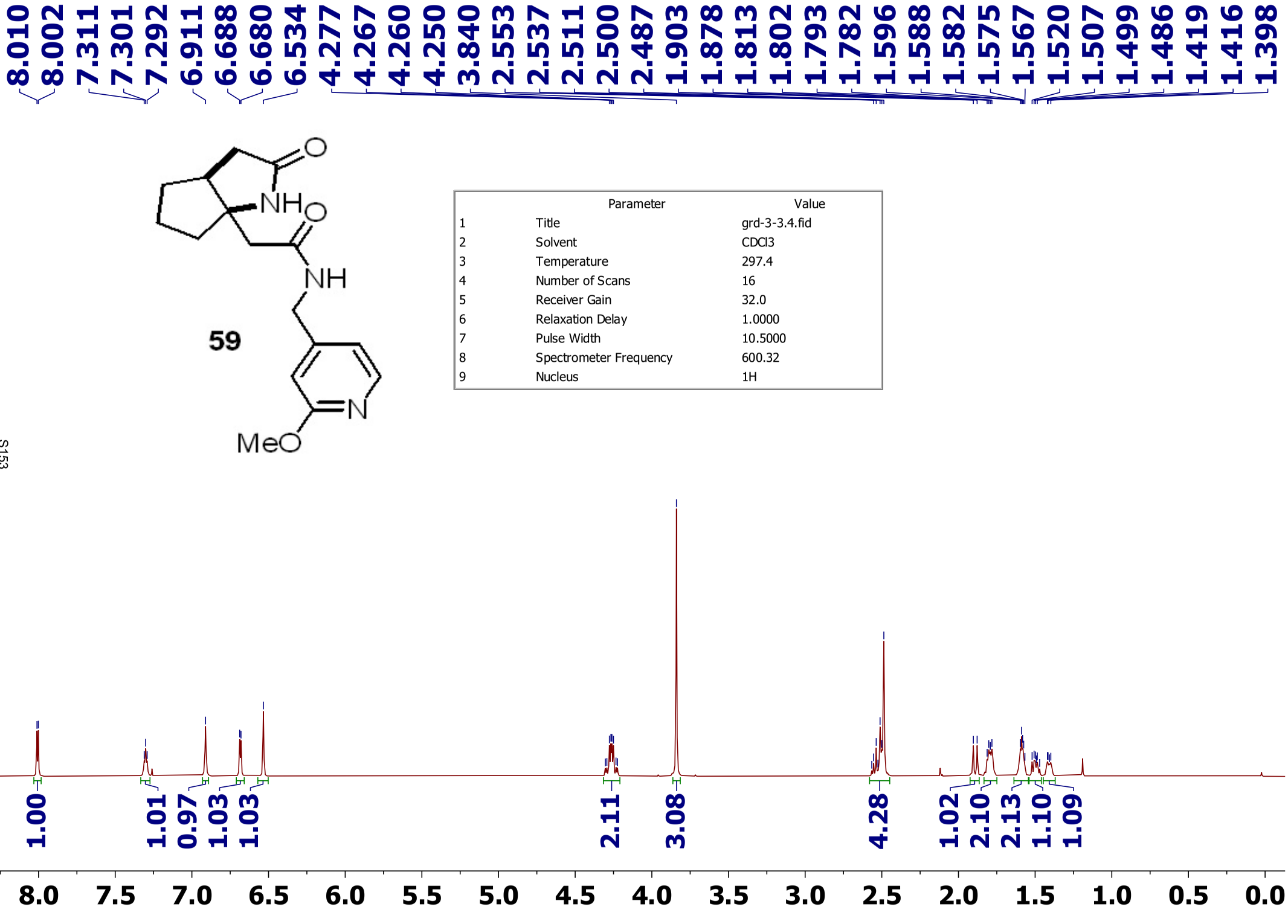
7.159
6.905
6.899
6.891
6.885
6.836
4.560
4.551
4.532
4.523
2.425
2.419
2.410
2.400
2.390
2.366
2.325
2.301
2.163
2.142
2.109
2.087
2.010
2.002
1.944
1.939
1.926
1.918
1.908
1.893
1.869
1.857
1.851
1.248
O
NH
O
S
Me
N
H
58
Parameter Value
1 Title grd-2-296.2.fid
2 Solvent CDCl3
3 Temperature 297.2
4 Number of Scans 16
5 Receiver Gain 40.3
6 Relaxation Delay 1.0000
7 Pulse Width 10.5000
8 Spectrometer Frequency 600.32
9 Nucleus 1H
S151
0.93
2.91
0.98
1.00
1.03
3.10
1.04
1.03
1.04
1.07
3.16
3.16
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



	Parameter	Value
1	Title	grd-2-296.3.fid
2	Solvent	CDCl3
3	Temperature	299.7
4	Number of Scans	256
5	Receiver Gain	2050.0
6	Relaxation Delay	5.0000
7	Pulse Width	10.6300
8	Spectrometer Frequency	150.97
9	Nucleus	13C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8.002
7.311
7.301
7.292
6.911
6.688
6.680
6.534
4.277
4.267
4.260
4.250
3.840
2.553
2.537
2.511
2.500
2.487
1.903
1.878
1.813
1.802
1.793
1.782
1.596
1.588
1.582
1.575
1.567
1.520
1.507
1.499
1.486
1.419
1.416
1.398
O
NH
O
NH
59
N
MeO
Parameter Value
1 Title grd-3-3.4.fid
2 Solvent CDCl3
3 Temperature 297.4
4 Number of Scans 16
5 Receiver Gain 32.0
6 Relaxation Delay 1.0000
7 Pulse Width 10.5000
8 Spectrometer Frequency 600.32
9 Nucleus 1H
1.00
1.01
0.97
1.03
1.03
2.11
3.08
4.28
1.02
2.10
2.13
1.10
1.09
8.0
7.5
7.0
6.5
6.0
5.5
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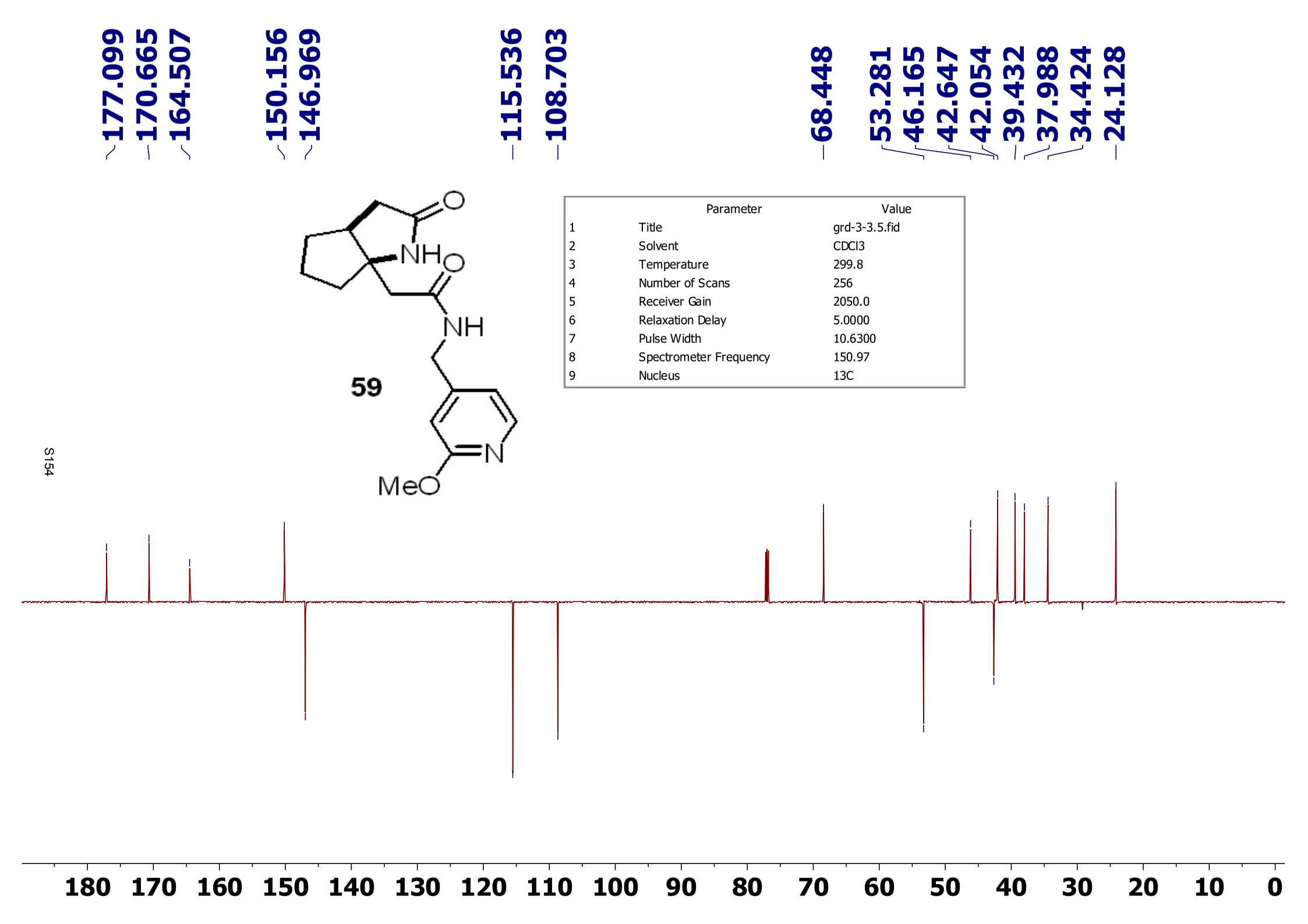
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0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170.665
164.507
150.156
146.969
115.536
108.703
68.448
53.281
46.165
42.647
42.054
39.432
37.988
34.424
24.128
O
NH
O
NH
N
MeO
59
Parameter Value
1 Title grd-3-3.5.fid
2 Solvent CDCl3
3 Temperature 299.8
4 Number of Scans 256
5 Receiver Gain 2050.0
6 Relaxation Delay 5.0000
7 Pulse Width 10.6300
8 Spectrometer Frequency 150.97
9 Nucleus 13C
S154
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0