Enantioselective Synthesis of Isochromans and Tetrahydroisoquinolines by C–H Insertion of Donor/Donor Carbenes

Supporting Information

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EXPERIMENTAL DETAILS

General Comments. Chemicals were purchased and used without further purification unless otherwise specified. All reactions using anhydrous solvents were carried out under an atmosphere of industrial argon in flame-dried glassware with magnetic stirring. Anhydrous solvent was dispensed from a solvent purification system that passes solvent through two columns of dry neutral alumina. Reactions were monitored by thin layer chromatography (TLC, Merck), and detected by examination under UV light (254 nm and 365 nm). Flash column chromatography was performed using silica gel [230−400 mesh (40−63 µm)]. Extracts were concentrated *in vacuo* using both a rotary evaporator (bath temperatures up to 40 °C) at a pressure of ≥10 torr (diaphragm pump). High vacuum procedures were carried out at room temperature at a pressure of 1 mtorr (diaphragm pump) or ≥1000 mtorr (oil pump). ^{1}H and proton-decoupled ^{13}C spectra were measured in $CDCl_3$ at 400, or 600 MHz, and 101 or 151 MHz respectively unless otherwise noted. All spectra in $CDCl_3$ were referenced at TMS = 0 ppm. Multiplicities are given as: s (singlet), d (doublet), t (triplet), q (quartet), p (pentet), m (multiplet), or combinations of these signals. Apparent signals are indicated with *app.* and are used when signals with multiple couplings appear to form a certain peak type. High-resolution mass spectrometry was performed on positive mode and ESI/Orbitrap™, ESI/TOF, and CI/TOF techniques were generally used. For substrates **15b**, **17a**, **76**, **80**, high-resolution mass spectrometry using the aforementioned techniques was not achieved; low-resolution mass spectrometry using an Advion© ASAP-APCI-MS was achieved and the corresponding data is reported for those samples. Melting points were taken on an EZ-melting apparatus and were uncorrected. Infrared spectra were taken on a Bruker Tensor 27 spectrometer. All microwave experiments were run in a Biotage Initiator EXP EU 400W microwave synthesizer 2.0 serial number 11031. NOTE: it is necessary that the MnO_2 used for the oxidation of hydrazones be ~85% pure with an average particle size of 2 microns, appearing as a fine black powder (e.g. Oakwood Chemical, CAS #: 1313-13-9, cat. #: 094454, lot #: 094454K03K).

General Procedure A (alkylation of alcohols). Following a modified literature procedure,[1] NaH (60% suspension in mineral oil, 1.5-3.0 equiv) and anhydrous THF (0.56 M with NaH) were added to a flame-dried round bottom flask. The suspension of NaH in THF was stirred and cooled to 0 °C. To the flask was added a solution of the desired alcohol (1.0 equiv) in anhydrous THF (0.78 M) dropwise and stirred for 2 h. The respective bromide (1.1-1.7 equiv) was added dropwise and the reaction was warmed to rt and stirred for 2-16 hours. Upon completion by TLC, the reaction was quenched at 0 °C with sat. aq. NH_4Cl or H_2O (20 mL) dropwise. The crude mixture was extracted with EtOAc (3 X 40 mL) and the combined organic layers were washed with brine (1 X 30 mL). The organic layer was dried over Na_2SO_4, filtered, and concentrated *in vacuo*. The crude reaction mixture was purified by flash column chromatography to yield the desired ether.

General Procedure B (alkylation of amides). Following a modified literature procedure,[2] NaH (60% suspension in mineral oil, 1.5 equiv) and anhydrous THF (3.5 M with NaH) were added to a flame-dried round bottom flask. The suspension of NaH in THF was stirred and cooled to 0 °C. To the flask was added a solution of the desired amide (1.0 equiv) in THF (0.67 M) dropwise and the mixture was allowed to warm to rt over 2 h. Aryl Bromide **57** (1.1 equiv) was added to the mixture at rt and the reaction was stirred for 16 h. Upon completion by TLC, the reaction was quenched with H_2O (30 mL) and extracted with EtOAc (3 X 40 mL). The combined organic layers were washed with brine (1 X 30 mL), dried over Na_2SO_4, filtered, and concentrated *in vacuo*. The crude reaction mixture was purified by flash column chromatography to yield the desired amide.

General Procedure C (alkylation of alcohols). To a flame-dried round bottom flask was added NaH (60% suspension in mineral oil, 3.0 equiv) and anhydrous THF (1.2 M with NaH). The suspension of NaH in THF was stirred for 15 min and cooled to 0 °C. To the flask was added a solution of the respective alcohol (2.5 equiv) in anhydrous THF (6.0 M) and stirred for 1 h. To the flask was added a solution of the desired alkyl/aryl halide (1.0 equiv) in THF (0.8 M) followed by tetrabutyl ammonium iodide (0.14 equiv). The reaction was allowed to warm to rt overnight. Upon completion by TLC, the reaction was quenched with sat. aq. NH_4Cl (20-50 mL) and the crude mixture was extracted with EtOAc (3 X 40 mL). The combined organic layers were washed with H_2O (1 X 30 mL) and brine (1 X 30 mL). The organic layer was dried over Na_2SO_4, filtered, and concentrated *in vacuo*. The crude reaction mixture was purified by flash column chromatography to yield the desired ether.

General Procedure D (synthesis of morpholine amide). Following a modified literature procedure,[3] $Pd(OAc)_2$ (0.025 equiv), Xantphos (0.05 equiv), and potassium phosphate tribasic (K_3PO_4, 3 equiv) were added to a dry 20 mL μW tube or a 25 mL round bottom flask under argon and the tube was sealed. A solution of the relevant aryl halide (1 equiv) in toluene (0.5 M) was added to the tube followed by morpholine (1.5 equiv) which had been freshly filtered through basic alumina. A vent needle was placed in the septum of the μW tube and the headspace was purged with CO (g) from a balloon for 30 sec. The vent needle was removed and the reaction was kept under an atmosphere of CO with the balloon in the headspace. The reaction was heated to 100 °C overnight. Upon completion by TLC, the reaction was diluted with EtOAc (10 mL) and filtered through Celite. The crude reaction mixture was purified by flash column chromatography to yield the desired morpholine amide. *Morpholine amides often showed significant rotamer distortion in the ^1H and ^{13}C NMR.*

General Procedure E (addition to morpholine amide). To a flame-dried round bottom flask was added the desired morpholine amide (1.0 equiv) in THF (0.1-0.17 M) and the solution was cooled to -78 °C. The respective aryl/alkyl lithium reagent (2 equiv) was added dropwise and the reaction was allowed to warm to rt overnight. The reaction was quenched at -78 °C with sat. NH_4Cl in CH_3OH (10 mL) and the mixture was allowed to warm to rt. H_2O (10 mL) was added and the crude mixture was extracted with EtOAc (3 X 30 mL) and the combined organic layers were washed with brine (1 X 20 mL). The organic layer was dried over Na_2SO_4, filtered, and concentrated *in vacuo*. The crude reaction mixture was purified by flash column chromatography to yield the desired ketone.

General Procedure F (addition to morpholine amide). To a flame-dried round bottom flask was added the desired morpholine amide (1.0 equiv) in THF (0.1 M) and the solution was cooled to -78 °C. The respective aryl lithium (2 equiv) was added dropwise and reacted for 10 min. Then the reaction was allowed to warm to rt for 1 h. The reaction was quenched at -78 °C with sat. aq. NH_4Cl (10 mL) and the crude mixture was extracted with EtOAc (3 X 30 mL) and the combined organic layers were washed with brine (1 X 20 mL). The organic layer was dried over Na_2SO_4, filtered, and concentrated *in vacuo*. The crude reaction mixture was purified by flash column chromatography to yield the desired ketone.

General Procedure G (addition to morpholine amide). To a flame-dried round bottom flask was added the desired aryl halide (1.5 equiv) in THF (0.26 M) and the solution was cooled to -78 °C. To the solution was added *n*-butyllithium (1.6 equiv) dropwise and the solution was stirred for 2 h at -78 °C. The respective morpholine amide (1.0 equiv) in THF (0.44 M) was added to the solution and the reaction was allowed to warm to rt overnight. The reaction was quenched at -78 °C with sat. NH_4Cl in CH_3OH (10 mL) and the mixture was allowed to warm to rt. H_2O (10 mL) was added and the crude mixture was extracted with EtOAc (3 X 30 mL) and the combined organic layers were washed with brine (1 X 20 mL). The organic layer was dried over Na_2SO_4, filtered, and concentrated *in vacuo*. The crude reaction mixture was purified by flash column chromatography to yield the desired ketone.

General Procedure H (synthesis of Weinreb amide). Following a modified literature procedure,[4] the desired benzoic acid (1.0 equiv) in CH_2Cl_2 (0.5 M) was added to a flame-dried round bottom flask under argon and equipped with a vent needle. Oxalyl chloride (2.0 equiv) was added dropwise to the solution followed by addition of *N*, *N*-dimethylformamide (1.3 equiv) and the solution was stirred at rt for 1 h. The mixture was concentrated *in vacuo* and the resulting acid chloride was diluted with CH_2Cl_2 (0.1 M) and Et_3N (3.0 equiv) and *N*,*O*-dimethylhydroxylamine hydrochloride (1.5 equiv) were added. After stirring for 2.5 h the reaction was quenched with sat. aq. $NaHCO_3$ (175 mL) and the mixture was extracted with CH_2Cl_2 (3 X 175 mL) and the combined organic layers were washed with $NaHCO_3$ (2 X 175 mL) and brine (175 mL). The organic layer was dried over Na_2SO_4, filtered, and concentrated *in vacuo*. The crude reaction mixture was purified by flash column chromatography to yield the desired Weinreb amide.

General Procedure I (synthesis of Weinreb amide). Following a modified literature procedure, to a flame-dried round bottom flask were added *N*,*O*-dimethylhydroxylamine hydrochloride (1.0 equiv) and CH_2Cl_2 (0.4 M). Et_3N (3.0 equiv) was added slowly at 0 °C. Benzoyl chloride (1.0 equiv) was then added dropwise into the mixture the solution was stirred at rt overnight. Upon completion, the reaction was quenched with sat. aq. $NaHCO_3$ (10 mL) and the mixture was extracted with CH_2Cl_2 (3 X 30 mL) and the combined organic layers were washed with H_2O (20 mL) and brine (20 mL). The organic layer was dried over Na_2SO_4, filtered, and concentrated *in vacuo*. The crude reaction mixture was purified by flash column chromatography to yield the desired Weinreb amide.

General Procedure J (addition to Weinreb amide). Following a modified literature procedure, the desired aryl halide (1.0 equiv) in anhydrous THF (0.1 M) was added to a flame-dried round bottom flask under argon and cooled to -78 °C. To the solution was added *n*-butyllithium in hexanes (1.1 equiv) dropwise and the resulting mixture was stirred at -78 °C for 1-2 h. The relevant Weinreb amide (1.5 equiv) in THF (0.2-1 M) was added to the mixture and the reaction was allowed to warm to rt overnight. The reaction was quenched at -78 ºC with sat. aq. NH_4Cl and was extracted with EtOAc (3 X 20 mL). The combined organic layers were washed with H_2O (1 X 20 mL) and brine (1 X 20 mL), dried over Na_2SO_4, filtered, and concentrated *in vacuo*. The crude reaction mixture was purified by flash column chromatography to yield the desired ketone.

General Procedure K (Reduction of Ketone). To a flame-dried round bottom flask were added 1-(2-bromophenyl)ethan-1-one (1.0 equiv) and anhydrous EtOH (0.25 M with ketone). The solution was cooled to 0 °C. To the flask was added $NaBH_4$ (4.0 equiv) in one portion and stirred for 10 min. The reaction was allowed to warm to rt overnight. Upon completion by TLC, the reaction was quenched with sat. aq. NH_4Cl (20 mL) and the crude mixture was extracted with EtOAc (3 X 40 mL). The combined organic layers were washed with H_2O (1 X 30 mL) and brine (1 X 30 mL). The organic layer was dried over Na_2SO_4, filtered, and concentrated *in vacuo*. The crude reaction mixture was purified by flash column chromatography to yield the desired alcohol.

General Procedure L (Alkylation of Alcohol). To a flame-dried round bottom flask were added 1-(2-bromophenyl)ethan-1-ol (1.0 equiv) and anhydrous THF/DMF (91:9 v/v, 0.18 M with alcohol). NaH (60% suspension in mineral oil, 2.5 equiv) was added portion-wise at 0 °C and the mixture was stirred for 10 min. The respective chloride (1.1 equiv) and TBAI (0.1 equiv) were added to the flask. The reaction was allowed to warm to rt overnight. Upon completion by TLC, the reaction was quenched with H_2O (20 mL) and the crude mixture was extracted with EtOAc (3 X 40 mL). The combined organic layers were washed with brine (1 X 30 mL), dried over Na_2SO_4, filtered, and concentrated *in vacuo*. The crude reaction mixture was purified by flash column chromatography to yield the desired ether.

General Procedure M (addition to Weinreb amide). Following a modified literature procedure,[5] the desired aryl halide (1.0 equiv) in anhydrous THF (0.31 M) was added to a flame-dried round bottom flask under argon and cooled to -78 °C. To the solution was added *n*-butyllithium in hexanes (1.3 equiv) dropwise and the resulting mixture was stirred at -78 °C for 2 h. The relevant Weinreb amide (1.2 equiv) in THF (1.2 M) was added to the mixture and the reaction was allowed to warm to rt overnight. The reaction was quenched with sat. NH_4Cl in CH_3OH at -78 °C and upon warming to rt the reaction was extracted with diethyl ether (3 X 20 mL). The combined organic layers were dried over Na_2SO_4, filtered, and concentrated *in vacuo*. The crude reaction mixture was purified by flash column chromatography to yield the desired ketone.

General Procedure N (carbonylation with benzaldehyde). Following a modified literature procedure,[6] the desired aryl halide (1.0 equiv), palladium (II) acetate (0.05 equiv), silver (I) oxide (1.2 equiv), benzaldehyde (8.0 equiv), and 70% *tert*-butyl hydroperoxide in H_2O (5.0 equiv) were added to a microwave vial with a stir bar and the microwave vial was sealed. An argon line (or balloon) was added to the vial and the system was heated to 118 °C. When the temperature was reached the argon line/balloon was removed and the mixture was heated overnight. The reaction mixture was cooled to rt, diluted with EtOAc, filtered through Celite with a small pad of silica, and concentrated *in vacuo*. The crude product was purified using flash column chromatography to yield the desired ketone.

General Procedure O (microwave hydrazone formation). Following a modified literature procedure,[7] the respective ketone (1.0 equiv) in anhydrous EtOH (0.1 M) was added to a flame-dried 5-25 mL argon-backfilled microwave vial. The solution was sparged with argon for 10-30 min after which AcOH (1.0-1.3 equiv) and anhydrous hydrazine (6-13 equiv) were added and the solution was sparged for an additional 1 min. The vial was heated in a microwave reactor at 150-170 °C for 2-10 hours. The reaction mixture was then concentrated *in vacuo,* dissolved in Et_2O (50 mL), and washed with H_2O (3 X 30 mL). The organic layer was dried over Na_2SO_4, filtered, concentrated *in vacuo*, and purified by flash column chromatography to yield the desired hydrazone. *Hydrazones were often isolated as a mixture of E/Z isomers and were used without further purification. As such ^1H NMR peaks have been reported only for selected examples. Due to restricted rotation, the hydrazone NMRs often indicated diastereotopic protons.*

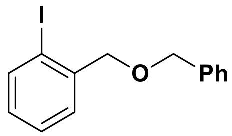
General Procedure P (hydrazone formation). Following literature precedent,[8] the desired ketone (1 equiv) in anhydrous EtOH (0.06 M) was added to a flame-dried round bottom flask under argon. To the flask was added anhydrous hydrazine (10 equiv.) and glacial acetic acid (0.5 to 2.0 equiv). The reaction was heated to 80 °C for 18-120 h. Upon completion by TLC, the reaction was allowed to cool and EtOH was removed *in vacuo*. The residue was dissolved in diethyl ether (30 mL) and washed with H_2O (2 X 20 mL) and brine (1 X 20 mL). The organic layer was dried over Na_2SO_4, filtered, and concentrated *in vacuo*, and purified by flash column chromatography to yield the desired hydrazone. *Hydrazones were often isolated as a mixture of E/Z isomers or were used without further purification. As such ^1H NMR peaks have been reported only for selected examples. Due to restricted rotation, the hydrazone NMRs often indicated diastereotopic protons.*

General Procedure Q (one-pot insertion). Following literature precedent,[7] the desired hydrazone (1.0 equiv) was added to a flame-dried scintillation vial under argon followed by anhydrous CH_2Cl_2 or CH_3CN (0.015 M). The vial was cooled to 0 °C and manganese (IV) oxide (MnO_2, 8.0 equiv) and the desired rhodium catalyst (0.01 equiv) were added. The resulting dark suspension was warmed to room temperature and allowed to stir from 10 min to 12 h. The crude reaction mixture was filtered over Celite, concentrated *in vacuo*, and purified by flash column chromatography to yield the desired insertion product.

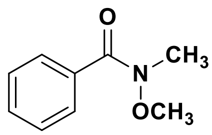
General Procedure R (sequential one-pot insertion). Following literature precedent,[7] the desired hydrazone (1.0 equiv) was added to a flame-dried scintillation vial under argon followed by anhydrous CH_2Cl_2 or CH_3CN (0.015-0.017 M). To the vial was added manganese (IV) oxide (MnO_2, 8.0 equiv) and the resulting dark suspension was stirred until full conversion of the starting material was observed by TLC. Upon pausing stirring, a color change from clear to magenta was observed from formation of the diazo. The vial was then cooled to 0 °C and the desired rhodium catalyst was added (0.01 equiv). The reaction mixture was allowed to warm to rt for 10 min to 24 h. The crude reaction mixture was filtered over Celite to remove MnO_2, concentrated *in vacuo*, and purified by flash column chromatography to yield the desired insertion product.

General Procedure S (two-pot insertion). Following literature precedent,[7] the desired hydrazone (1.0 equiv) was added to a flame-dried scintillation vial under argon followed by anhydrous CH_2Cl_2 or CH_3CN (0.015 M). To the vial was added manganese (IV) oxide (MnO_2, 8.0 equiv) and the resulting dark suspension was stirred until full conversion of the starting material was observed by TLC. The reaction mixture was filtered over Celite into a new flame-dried, argon backfilled 20 mL scintillation vial using the same solvent. The magenta solution was cooled to 0 °C and the desired rhodium catalyst was added (0.01 equiv). The reaction mixture was allowed to warm to rt for 10 min to 16 h. The crude reaction mixture was concentrated *in vacuo* and purified by flash column chromatography to yield the desired insertion product.

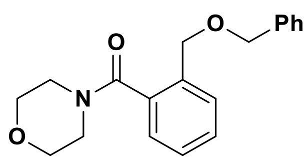
ISOCHROMANS



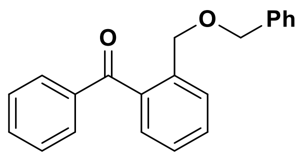
1-((benzyloxy)methyl)-2-iodobenzene (30) was synthesized according to General Procedure A using 2-iodobenzyl alcohol (1.000 g, 4.273 mmol) in THF (5.5 mL), NaH (0.292 g, 7.30 mmol) in THF (13 mL), and benzyl bromide (0.87 mL, 7.3 mmol).The crude product was extracted from NH_4Cl (20 mL) with EtOAc (2 X 40 mL) and washed with brine (1 X 20 mL). The crude material was purified by flash column chromatography (99:1 to 90:10, hexanes:EtOAc) affording **30** as a yellow oil (1.320 g, 95%).^{1}H NMR (600 MHz, $CDCl_3$) δ 7.83 (dd, *J* = 8.0, 1.3 Hz, 1H), 7.49 (dd, *J* = 7.6, 1.7 Hz, 1H), 7.43 – 7.39 (m, 2H), 7.39 – 7.33 (m, 3H), 7.33 – 7.29 (m, 1H), 7.02 – 6.96 (m, 1H), 4.64 (s, 2H), 4.55 (s, 2H).. ^{1}H NMR data was consistent with reported literature values.[9]



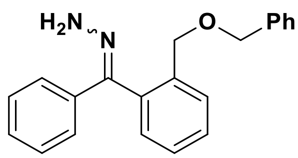
***N*-methoxy-*N*-methylbenzamide (31)** was synthesized according to General Procedure H using benzoic acid (2.000 g, 16.37 mmol) in CH_2Cl_2 (3.3 mL), oxalyl chloride (2.8 mL, 33 mmol), *N*, *N*-dimethylformamide (1.64 mL, 21.3 mmol), Et_3N (6.3 mL, 49 mmol), and *N*,*O*-dimethylhydroxylamine hydrochloride (2.400 g, 24.60 mmol) in CH_2Cl_2 (164 mL). The crude product was extracted from $NaHCO_3$ (100 mL) with CH_2Cl_2 (3 X 60 mL) and washed with $NaHCO_3$ (2 X 70 mL) and brine (1 X 70 mL). The crude product was purified by flash column chromatography (90:10 to 70:30, hexanes:EtOAc) affording **31** as a clear oil (1.937 g, 71%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.67 (d, *J* = 7.3 Hz, 2H), 7.47 – 7.43 (m, 1H), 7.42 – 7.38 (m, 2H), 3.55 (s, 3H), 3.36 (s, 3H). ^{1}H NMR data was consistent with literature values.[10]



(2-((benzyloxy)methyl)phenyl)(morpholino)methanone (32) was synthesized according to General Procedure D using ether **30** (0.500 g, 1.54 mmol), morpholine (0.20 mL, 2.3 mmol), palladium (II) acetate (0.008 g, 0.02 mmol), Xantphos (0.045 g, 0.077 mmol), potassium phosphate tribasic (0.981 g, 4.62 mmol), CO (1 balloon), in toluene (3 mL). The crude product was purified using flash column chromatography (40:60, hexanes:EtOAc) affording **32** as a yellow oil (0.403 g, 84%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46 (d, *J* = 7.6 Hz, 1H), 7.41 – 7.28 (m, 7H), 7.19 (d, *J* = 7.4 Hz, 1H), 4.73 – 4.39 (m, 4H), 3.79 – 3.58 (m, 4H), 3.47 (s, 2H), 3.31 – 3.09 (m, 2H). ^{1}H NMR was taken at rt and shows a mixture of rotamers.



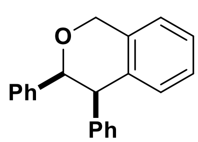
(2-((benzyloxy)methyl)phenyl)(phenyl)methanone (33). To a flame-dried vial was added bromobenzene (0.30 mL, 2.8 mmol) in THF (3 mL) and the solution was cooled to -78 °C. A 1.7 M solution of *tert*-butyllithium (3.3 mL, 5.68 mmol) was added dropwise to the solution and the mixture was stirred at -78 °C for 1 h. A solution of morpholine amide **32** (0.295 g, 0.947 mmol) in THF (5.6 mL) was added dropwise to the solution and the reaction was allowed to warm to rt overnight. The reaction was quenched with sat. NH_4Cl in MeOH (6 mL) at -78 °C. The quenched solution was warmed to rt and diluted with water (20 mL). The resulting mixture was extracted with EtOAc (3 X 30 mL), washed with brine (1 X 30 mL), dried over Na_2SO_4, filtered, and concentrated *in vacuo*. The crude product was purified using flash column chromatography (90:10, hexanes:EtOAc) affording **33** as a yellow oil (0.144 g, 50%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.79 (d, *J* = 7.8 Hz, 2H), 7.60 – 7.51 (m, 2H), 7.49 – 7.43 (m, 1H), 7.40 (t, *J* = 7.6 Hz, 2H), 7.37 – 7.29 (m, 2H), 7.23 – 7.17 (m, 3H), 7.17 – 7.11 (m, 2H), 4.65 (s, 2H), 4.39 (s, 2H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 197.91, 137.98, 137.92, 137.89, 137.60, 133.09, 130.45, 130.17, 128.79, 128.65, 128.40, 128.26, 127.66, 127.52, 127.04, 72.82, 70.00; AMM (ESI-TOF) *m/z* calcd for $C_{21}H_{18}NaO_2^+$ $[M+Na]^+$ 325.1205, found 325.1200.



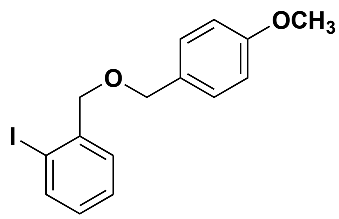
((2-((benzyloxy)methyl)phenyl)(phenyl)methylene)hydrazine (10a) was synthesized according to General Procedure O using ketone **33** (0.280 g, 0.926 mmol), hydrazine (0.29 mL, 9.3 mmol), acetic acid (0.06 mL, 1 mmol), and EtOH (9.3 mL) at 150 °C for 2 h. The crude product was purified by flash column chromatography (100:0 to 70:30, hexanes:EtOAc) affording **10a** as a white foam (0.172 g, 58%).

Major stereoisomer **10a-1***:* ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.69 (d, *J* = 7.6 Hz, 1H), 7.51 – 7.41 (m, 4H), 7.30 – 7.20 (m, 8H), 7.18 (d, *J* = 7.3 Hz, 1H), 5.34 (s, 2H), 4.46 (d, *J* = 11.8 Hz, 1H), 4.43 (d, *J* = 11.7 Hz, 1H), 4.40 (d, *J* = 11.9 Hz, 1H), 4.35 (d, *J* = 12.0 Hz, 1H).

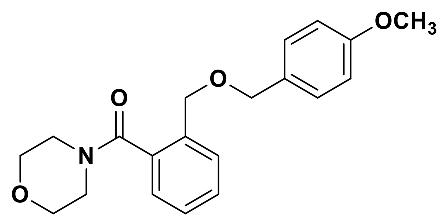
Minor stereoisomer **10a-2***:* (0.028 g, 9%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.60 (d, *J* = 7.7 Hz, 1H), 7.48 – 7.41 (m, 2H), 7.40 – 7.24 (m, 9H), 7.21 (t, *J* = 7.5 Hz, 1H), 7.14 (d, *J* = 7.7 Hz, 1H), 5.53 (s, 2H), 4.67 (s, 2H), 4.50 (s, 2H).



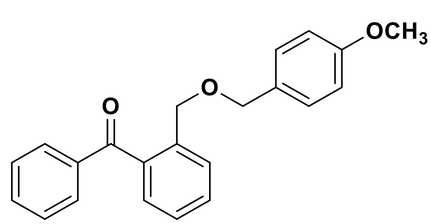
(3*S*,4*R*)-3,4-diphenylisochromane (11a) was synthesized according to General Procedure R using hydrazone **10a-1** (0.050 g, 0.16 mmol), MnO_2 (0.111 g, 1.28 mmol), and Rh_2(*R*-PTAD)$_4$ (0.002 mg, 0.002 mmol) in CH_2Cl_2 (11 mL). The crude product was purified by flash column chromatography (100:0 to 97:3, hexanes: EtOAc) affording **11a** as white crystals (0.028 g, 61%, >95:5 dr, 97:3 er). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.27 – 7.22 (m, 1H), 7.21 – 7.10 (m, 5H), 7.08 – 6.99 (m, 4H), 6.99 – 6.93 (m, 2H), 6.78 – 6.73 (m, 2H), 5.24 (d, *J* = 15.2 Hz, 1H), 5.12 (d, *J* = 15.3 Hz, 1H), 5.10 (d, J = 3.3 Hz, 1H), 4.13 (d, J = 3.2 Hz, 1H).; ^{13}C NMR (151 MHz, $CDCl_3$) δ 140.8, 140.3, 137.3, 134.3, 130.4, 130.2, 127.8, 127.4, 127.0, 127.0, 126.8, 126.2, 126.0, 124.2, 80.1, 69.2, 50.4; AMM (CI-TOF) *m/z* calcd for $C_{21}H_{22}NO^+$ $[M+H]^+$ 304.1696, found 304.1690; $[\alpha]_D^{22} = -33.4$ (c = 0.086, $CHCl_3$).



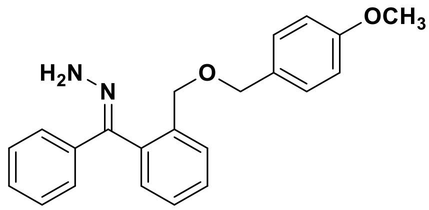
2-(4-methoxybenzyloxymethyl)iodobenzene (34) was synthesized according to General Procedure C, using sodium hydride (0.352 g, 8.55 mmol) in THF (10.7 mL), iodobenzyl alcohol (1.001 g, 4.273 mmol) in THF (5.3 mL), tetrabutylammonium iodide (0.224 g, 0.607 mmol), and 4-methoxybenzyl chloride (0.64 mL, 4.7 mmol), reacting for 2 h at 50 °C. The crude product was purified using flash column chromatography (60:40, hexanes:CH_2Cl_2), affording **34** as a yellow oil (1.344 g, 89%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.82 (d, J = 7.9 Hz, 1H), 7.47 (d, J = 7.1 Hz, 1H), 7.38 – 7.30 (m, 3H), 6.98 (t, J = 7.6 Hz, 1H), 6.93 – 6.86 (m, 2H), 4.57 (s, 2H), 4.52 (s, 2H), 3.81 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 159.0, 140.4, 138.8, 129.8, 129.1, 128.9, 128.6, 127.9, 113.5, 97.7, 75.5, 72.1, 54.9; AMM (ESI-TOF) *m/z* calcd for $C_{15}H_{15}INaO_2^+$ $[M+Na]^+$ 377.0009, found 377.0015.



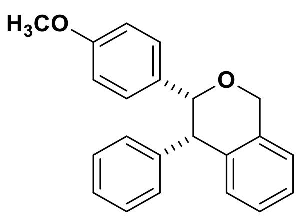
((4-methoxybenzyl)oxymethyl-phenyl)-morpholin-4-yl-methanone (35) was synthesized according to General Procedure D, using $Pd(OAc)_2$ (0.012 g, 0.052 mmol), Xantphos (0.058 g, 0.10 mmol), and potassium phosphate tribasic (1.280 g, 6.031 mmol), morpholine (0.26 mL, 3.0 mmol), and **34** (0.711 g, 2.00 mmol) in toluene (4.0 mL), reacting at 100 °C for 6.5 h. The crude product was purified using flash column chromatography (20:80, hexanes:EtOAc), affording **35** as a clear oil (0.572 g, 84%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.44 (d, J = 7.6 Hz, 1H), 7.37 (t, J = 7.5 Hz, 1H), 7.32 (t, J = 7.5 Hz, 1H), 7.30 – 7.27 (m, 2H), 7.19 (d, J = 7.4 Hz, 1H), 6.91 – 6.89 (m, 1H), 6.89 – 6.87 (m, 1H), 4.64 (br s, 1H), 4.51 (s, 2H), 4.44 (br s, 1H), 3.81 (s, 3H), 3.77 – 3.73 (m, 2H), 3.73 – 3.60 (m, 2H), 3.48 (br s, 2H), 3.29 – 3.08 (m, 2H); AMM (ESI) *m/z* calcd for $C_{20}H_{24}NO_4^+$ $[M+H]^+$ 342.1700, found 342.1704; IR (neat): 2855, 1635, 1514, 1429, 1114 cm^{-1}. ^{1}H NMR was taken at rt and shows a mixture of rotamers.



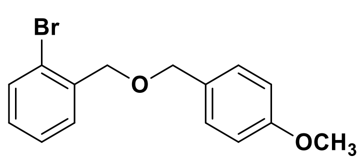
2-(4-methoxybenzyloxymethyl)benzophenone (36) was synthesized according to General Procedure E, using **35** (0.406 g, 1.19 mmol) in THF (11.7 mL), and 1.9 M phenyllithium in dibutyl ether (1.25 mL, 2.38 mmol), reacting for 2 h. The crude product was purified using flash column chromatography (90:10, hexanes:EtOAc), affording **36** as a yellow oil (0.330 g, 84%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.80 (d, *J* = 7.2 Hz, 2H), 7.59 – 7.55 (m, 2H), 7.48 (t, *J* = 7.3 Hz, 1H), 7.44 (t, *J* = 7.8 Hz, 2H), 7.38 – 7.32 (m, 2H), 7.08 (d, *J* = 8.6 Hz, 2H), 6.76 (d, *J* = 8.6 Hz, 2H), 4.62 (s, 2H), 4.33 (s, 2H), 3.77 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 198.1, 159.2, 138.2, 138.0, 137.7, 133.1, 130.5, 130.3, 130.1, 129.4, 128.8, 128.8, 128.5, 127.1, 113.7, 72.6, 69.8, 55.4; AMM (ESI) *m/z* calcd for $C_{22}H_{21}O_3^+$ $[M+H]^+$ 333.1485, found 333.1484; IR (neat): 2930, 2855, 1665, 1514, 1271 cm^{-1}.



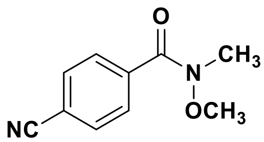
((2-(((4-methoxy)benzyloxy)methyl)phenyl)(phenyl)methylene)hydrazine (10b) was synthesized according to General Procedure O, using **36** (0.124 g, 0.372 mmol) in EtOH (3.7 mL), glacial acetic acid (0.03 mL, 0.5 mmol), and hydrazine (0.15 mL, 4.8 mmol), reacting for 3 h at 170 °C. The crude product was purified using flash column chromatography (70:30, hexanes:EtOAc), affording **10b** as a yellow oil (0.079 g, 61%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.66 (d, *J* = 7.1 Hz, 1H), 7.51 – 7.40 (m, 4H), 7.27 (dd, *J* = 5.1, 1.9 Hz, 3H), 7.19 – 7.11 (m, 3H), 6.81 (d, *J* = 8.6 Hz, 2H), 5.34 (s, 2H), 4.39 – 4.29 (m, 4H), 3.78 (s, 3H); AMM (ESI) *m/z* calcd for $C_{22}H_{23}N_2O_2^+$ $[M+H]^+$ 347.1754, found 347.1750.



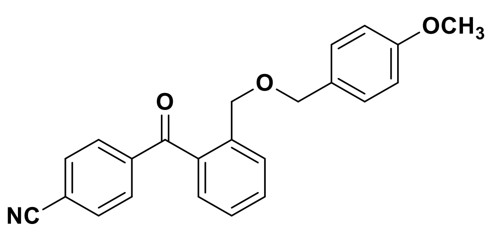
(3*R*,4*S*)-3-(4-methoxy)phenyl-4-phenylisochromane (11b) was synthesized according to General Procedure R, using MnO_2 (0.021 g, 0.24 mmol) and **10b** (0.027 g, 0.078 mmol) in CH_2Cl_2 (5.2 mL), reacting for 20 min. Rh_2(*S*-PTAD)$_4$ (0.001 g, 0.7 μmol) was added, reacting for 30 h. The crude product was purified using flash column chromatography (90:10, hexanes:EtOAc), affording **11b** as a white crystalline solid (0.016 g, >95:5 dr, >99.5:0.5 er, 69%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.26 – 7.21 (m, 1H), 7.19 – 7.11 (m, 2H), 7.08 – 6.98 (m, 4H), 6.85 (d, *J* = 8.5 Hz, 2H), 6.80 – 6.72 (m, 2H), 6.71 – 6.62 (m, 2H), 5.22 (d, *J* = 15.3 Hz, 1H), 5.10 (d, *J* = 15.3 Hz, 1H), 5.04 (d, *J* = 3.2 Hz, 1H), 4.07 (d, *J* = 2.8 Hz, 1H), 3.72 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 158.6, 141.0, 137.4, 134.4, 132.6, 130.4, 130.3, 127.4, 127.3, 126.9, 126.7, 126.2, 124.2, 113.2, 79.9, 69.3, 55.3, 50.5; AMM (CI-TOF) *m/z* calcd for $C_{22}H_{20}O_2^+$ $[M]^+$ 316.1463, found 316.1455; m.p. 167-168 °C; $[\alpha]_D^{21} = 523.9$ (c = 0.19, $CHCl_3$).



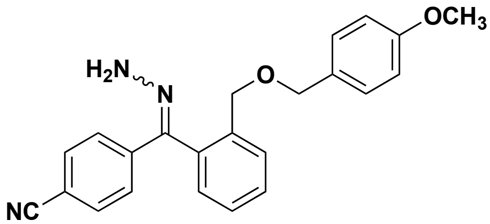
1-bromo-2-(((4-methoxybenzyl)oxy)methyl)benzene (37) was synthesized according to General Procedure C using 2-bromobenzyl bromide (3.000 g, 12.00 mmol) in THF (15 mL), *p*-methoxybenzyl alcohol (3.7 mL, 30 mmol) in THF (5 mL), NaH (1.440 g, 36.00 mmol) in THF (30 mL), and tetrabutylammonium iodide (0.621 g, 1.68 mmol). The crude product was quenched with sat. aq. NH_4Cl (50 mL) and extracted with EtOAc (3 X 40 mL), the combined organic layers were washed with H_2O (1 X 40 mL) and brine (1 X 40 mL). The crude product was purified by flash column chromatography (90:10, hexanes:EtOAc) affording **37** as a clear oil (3.424 g, 93%). ^1H NMR (400 MHz, $CDCl_3$) δ 7.52 (t, *J* = 8.0 Hz, 2H), 7.36 – 7.28 (m, 3H), 7.14 (t, *J* = 7.7 Hz, 1H), 6.90 (d, *J* = 8.1 Hz, 2H), 4.60 (s, 2H), 4.57 (s, 2H), 3.81 (s, 3H). ^1H NMR data was consistent with literature values.[11]



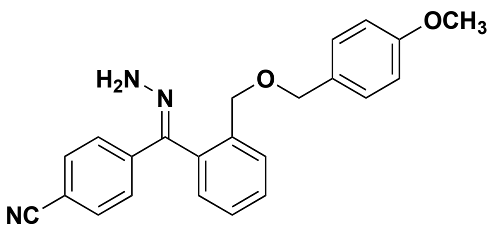
4-cyano-*N*-methoxy-*N*-methylbenzamide (38) was synthesized according to General Procedure H using 4-cyano-benzoic acid (1.863 g, 12.66 mmol) in CH_2Cl_2 (25 mL), oxalyl chloride (12.66 mL, 25.32 mmol), DMF (1.3 mL, 16 mmol), Et_3N (5.3 mL, 28 mmol), and *N*,*O*-dimethylhydroxylamine hydrochloride (1.852 g, 18.99 mmol) in CH_2Cl_2 (164 mL). The crude product was extracted from $NaHCO_3$ (100 mL) with CH_2Cl_2 (3 X 60 mL) and the combined organic layers were washed with $NaHCO_3$ (2 X 70 mL) and brine (1 X 70 mL). The crude product was purified by flash column chromatography (90:10, hexanes:EtOAc) affording **38** as a clear oil (1.858 g, 77%). ^1H NMR (600 MHz, $CDCl_3$) δ 7.78 (d, *J* = 8.1 Hz, 2H), 7.71 (d, *J* = 8.1 Hz, 2H), 3.53 (s, 3H), 3.39 (s, 3H). ^1H NMR data was consistent with literature values.[12]



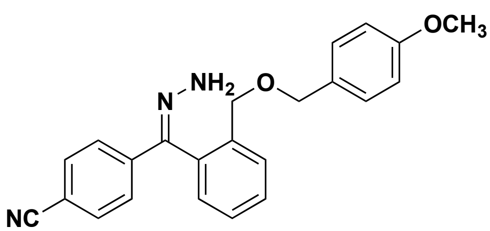
4-(2-(((4-methoxybenzyl)oxy)methyl)benzoyl)benzonitrile (39) was synthesized according to General Procedure J using **37** (0.538 g, 1.75 mmol), 2.5 M *n*-butyllithium in hexanes (0.85 mL, 2.1 mmol), **38** (0.500 g, 2.63 mmol), and THF (10.0 mL). The crude product was purified using flash column chromatography (80:20, hexanes:EtOAc), affording **39** as a white solid (0.455 g, 73%). ^1H NMR (600 MHz, $CDCl_3$) δ 7.83 (d, *J* = 8.4 Hz, 2H), 7.70 (d, *J* = 8.3 Hz, 2H), 7.57 – 7.48 (m, 2H), 7.41 – 7.36 (m, 1H), 7.31 (d, *J* = 7.2 Hz, 1H), 7.03 (d, *J* = 8.6 Hz, 2H), 6.75 (d, *J* = 8.6 Hz, 2H), 4.60 (s, 2H), 4.30 (s, 2H), 3.79 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 196.4, 159.2, 140.9, 138.2, 136.9, 132.1, 130.9, 130.2, 129.6, 129.3, 128.8, 128.5, 127.3, 118.0, 116.0, 113.6, 72.6, 69.7, 55.2; AMM (ESI-TOF) *m/z* calcd for $C_{23}H_{19}NaNO_3^+$ $[M+Na]^+$ 380.1263, found 380.1262.



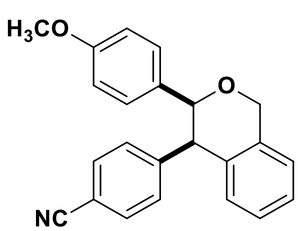
(*E/Z*)-4-(hydrazineylidene(2-(((4-methoxybenzyl)oxy)methyl)phenyl)methyl)benzonitrile (10c) was synthesized according to General Procedure P using **39** (0.357 g, 1.00 mmol), hydrazine (0.31 mL, 10 mmol), acetic acid (0.07 mL, 1 mmol), and EtOH (6.7 mL). The reaction was heated at 90 °C for 2 days. The crude product was purified using flash column chromatography (80:20, hexanes:EtOAc), affording **10c-1** as a colorless liquid (0.040 g, 11%), and **10c-2** as a colorless liquid (0.181 g, 49%)



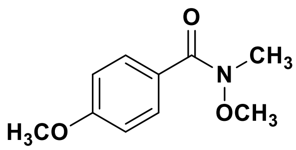
(*E*)-4-(hydrazineylidene(2-(((4-methoxybenzyl)oxy)methyl)phenyl)methyl)benzonitrile (10c-1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.69 (d, *J* = 7.8 Hz, 2H), 7.57 – 7.50 (m, 1H), 7.47 (d, *J* = 7.9 Hz, 2H), 7.32 (t, *J* = 7.5 Hz, 1H), 7.26 – 7.19 (m, 3H), 7.07 (d, *J* = 7.6 Hz, 1H), 6.86 (d, *J* = 8.1 Hz, 2H), 5.55 (s, 2H), 4.57 (s, 2H), 4.41 (s, 2H), 3.80 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 159.2, 146.5, 138.3, 137.2, 137.2, 132.7, 130.3, 129.9, 129.7, 129.5, 129.0, 128.4, 127.4, 118.3, 113.7, 112.5, 72.1, 70.1, 55.3; AMM (ESI) *m/z* calcd for $C_{23}H_{22}N_3O_2^+$ $[M+H]^+$ 372.1707, found 372.1703.



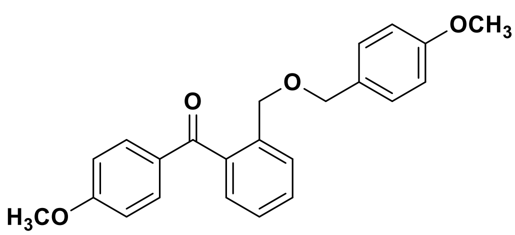
(*Z*)-4-(hydrazineylidene(2-(((4-methoxybenzyl)oxy)methyl)phenyl)methyl)benzonitrile (10c-2). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.66 (d, *J* = 7.3 Hz, 1H), 7.59 – 7.42 (m, 6H), 7.13 (d, *J* = 7.1 Hz, 1H), 7.08 (d, *J* = 8.1 Hz, 2H), 6.80 (d, *J* = 8.0 Hz, 2H), 5.58 (s, 2H), 4.35 – 4.30 (m, 2H), 4.29 – 4.23 (m, 2H), 3.79 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 159.2, 145.6, 142.3, 137.4, 132.0, 131.1, 129.9, 129.8, 129.7, 129.3, 129.2, 128.9, 126.2, 119.1, 113.7, 110.8, 72.7, 69.7, 55.3; AMM (ESI) *m/z* calcd for $C_{23}H_{22}N_3O_2^+$ $[M+H]^+$ 372.1707, found 372.1701.



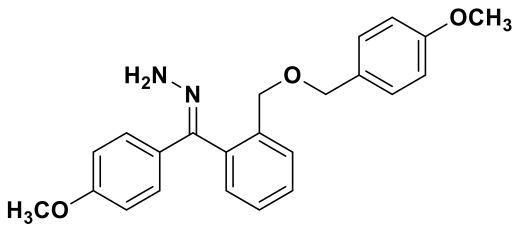
4-((3*S*,4*R*)-3-(4-methoxyphenyl)isochroman-4-yl)benzonitrile (11c) was synthesized according to General Procedure R using hydrazone **10c-2** (0.025 g, 0.067 mmol), MnO_2 (0.047 g, 0.54 mmol), and Rh_2(*R*-PTAD)$_4$ (0.001 g, 0.0007 mmol) in CH_3CN (4.5 mL). The crude product was purified by flash column chromatography (83:17, hexanes:EtOAc), affording **11c** as white crystals (0.0223 g, 97%, >95:5 dr, 99:1 er). ^1H NMR (600 MHz, $CDCl_3$) δ 7.33 (d, *J* = 8.2 Hz, 2H), 7.29 (t, *J* = 7.5 Hz, 1H), 7.21 – 7.17 (m, 2H), 6.99 (d, *J* = 7.8 Hz, 1H), 6.89 – 6.83 (m, 4H), 6.70 (d, *J* = 8.8 Hz, 2H), 5.23 (d, *J* = 15.4 Hz, 1H), 5.12 (d, *J* = 15.4 Hz, 1H), 5.08 (d, *J* = 3.4 Hz, 1H), 4.14 (d, *J* = 3.4 Hz, 1H), 3.75 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 158.6, 146.5, 135.7, 134.2, 131.7, 131.1, 130.8, 130.1, 127.2, 127.1, 126.8, 124.4, 119.1, 113.3, 109.9, 79.3, 69.1, 55.2, 50.5; AMM (ESI-TOF) *m/z* calcd for $C_{23}H_{19}NaNO_2^+$ $[M+Na]^+$ 364.1313, found 364.1318; m.p. 163-164 °C; $[\alpha]_D^{22} = -487$ (c = 0.11, $CHCl_3$).



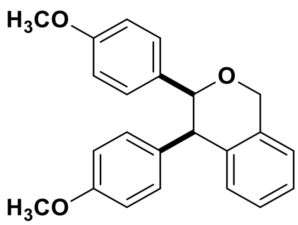
***N*,4-dimethoxy-*N*-methylbenzamide (40)** was synthesized according to General Procedure H using 4-methoxybenzoic acid (3.041 g, 20.00 mmol) in CH_2Cl_2 (40 mL), 2 M oxalyl chloride in CH_2Cl_2 (20.0 mL, 40.0 mmol), DMF (2.0 mL, 26 mmol), Et_3N (8.4 mL, 60 mmol), and *N*,*O*-dimethylhydroxylamine hydrochloride (2.926 g, 30.00 mmol) in CH_2Cl_2 (200.0 mL). The crude product was extracted from $NaHCO_3$ (200 mL) with CH_2Cl_2 (3 X 120 mL) and the combined organic layers were washed with $NaHCO_3$ (2 X 70 mL) and brine (1 X 70 mL). The crude product was purified by flash column chromatography (90:10, hexanes:EtOAc) affording **40** as a clear oil (3.360, 86%). ^1H NMR (400 MHz, $CDCl_3$) δ 7.73 (d, *J* = 8.4 Hz, 2H), 6.90 (d, *J* = 8.5 Hz, 2H), 3.85 (s, 3H), 3.56 (s, 3H), 3.36 (s, 3H). ^1H NMR data was consistent with literature values.[13]



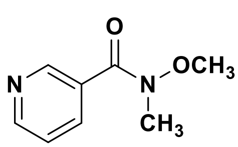
(2-(((4-methoxybenzyl)oxy)methyl)phenyl)(4-methoxyphenyl)methanone (41) was synthesized according to General Procedure J using **37** (1.536 g, 5.000 mmol), 2.5 M *n*-BuLi in hexanes (2.4 mL, 6.0 mmol), **40** (1.464 g, 7.500 mmol), and THF (30 mL). The crude product was purified using flash column chromatography (80:20, hexanes:EtOAc), affording **41** as a white solid (1.464 g, 84%). ^1H NMR (600 MHz, $CDCl_3$) δ 7.79 (d, *J* = 8.2 Hz, 2H), 7.56 (d, *J* = 7.7 Hz, 1H), 7.49 – 7.43 (m, 1H), 7.36 – 7.32 (m, 2H), 7.08 (d, *J* = 8.1 Hz, 2H), 6.91 (d, *J* = 8.2 Hz, 2H), 6.76 (d, *J* = 8.1 Hz, 2H), 4.60 (s, 2H), 4.34 (s, 2H), 3.87 (s, 3H), 3.77 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 196.6, 163.6, 159.0, 138.5, 137.5, 132.5, 130.5, 130.1, 130.0, 129.5, 129.3, 128.6, 128.2, 126.9, 113.6, 72.4, 69.6, 55.5, 55.2; AMM (ESI) *m/z* calcd for $C_{23}H_{23}O_4^+$ $[M+H]^+$ 363.1591, found 363.1586.



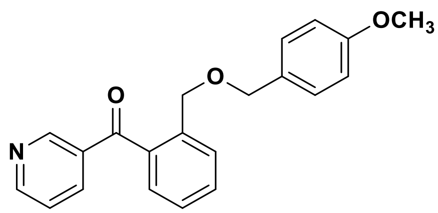
((2-(((4-methoxybenzyl)oxy)methyl)phenyl)(4-methoxyphenyl)methylene)hydrazine (10d) was synthesized according to General Procedure P using **41** (0.500 g, 1.38 mmol), hydrazine (0.45 mL, 14 mmol), acetic acid (0.14 mL, 2.8 mmol), and ethanol (9.2 mL). The reaction was heated at 90 °C for 4 days. The crude product was purified using flash column chromatography (80:20, hexanes:EtOAc), affording **10d** as a colorless liquid (0.421 g, 81%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.66 (d, *J* = 7.2 Hz, 1H), 7.45 (p, *J* = 7.5 Hz, 2H), 7.37 (d, *J* = 8.3 Hz, 2H), 7.15 (m, 3H), 6.88 – 6.73 (m, 4H) 5.21 (s, 2H), 4.50 – 4.27 (m, 4H), 3.79 (s, 3H), 3.78 (s, 3H) ;^{13}C NMR (151 MHz, $CDCl_3$) δ 159.8, 159.1, 148.5, 137.2, 132.4, 130.8, 130.2, 129.4, 129.2, 129.2, 128.8, 128.7, 127.3, 113.7, 113.6, 72.6, 69.7, 55.3, 55.2; AMM (ESI) *m/z* calcd for $C_{23}H_{25}N_2O_3^+$ [M+H]$^+$ 377.1860, found 377.1855.



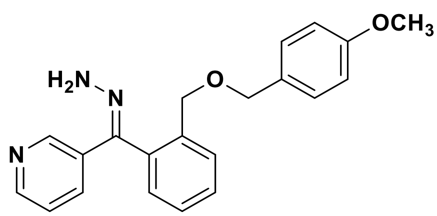
(3*S*,4*R*)-3,4-bis(4-methoxyphenyl)isochromane (11d) was synthesized according to General Procedure R using **10d** (0.025 g, 0.066 mmol), MnO_2 (0.046 g, 0.53 mmol), and Rh_2(*R*-PTAD)$_4$ (0.001 g, 0.0007 mmol) in CH_2Cl_2 (4.5 mL). The crude product was purified by flash column chromatography (83:17, hexanes:EtOAc), affording **11d** as white crystals (0.023 g, 98%, >95:5 dr, 99:1 er). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.27 – 7.21 (m, 1H), 7.19 – 7.13 (m, 2H), 7.04 (d, *J* = 7.5 Hz, 1H), 6.85 (d, *J* = 8.3 Hz, 2H), 6.70 (d, *J* = 8.3 Hz, 2H), 6.67 (d, *J* = 8.3 Hz, 2H), 6.59 (d, *J* = 8.3 Hz, 2H), 5.21 (d, *J* = 15.3 Hz, 1H), 5.10 (d, *J* = 15.2 Hz, 1H), 5.01 (d, *J* = 3.3 Hz, 1H), 4.03 (d, *J* = 3.2 Hz, 1H), 3.75 (s, 3H), 3.70 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 158.4, 157.8, 137.6, 134.2, 133.1, 132.6, 131.0, 130.2, 127.2, 126.8, 126.5, 124.0, 113.0, 112.7, 79.8, 69.1, 55.2, 55.1, 49.5; m.p. 159-160 °C ; AMM (ESI) *m/z* calcd for [M+Na]$^+$ $C_{23}H_{22}NaO_3^+$ 369.1467, found 369.1464; $[\alpha]_D^{22} = -352$ (c = 0.12, $CHCl_3$).



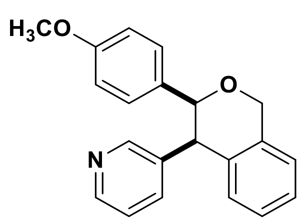
***N*-methoxy-*N*-methyl-3-nicotinamide (42)** was synthesized by preparing a suspension of *N*-methoxy-*N*-methylamine•HCl (1.317 g, 13.50 mmol, 1.2 equiv.) in CH_2Cl_2 (45.0 mL), following a modified literature procedure.[13] This suspension was cooled to 0 °C and triethylamine (6.25 mL, 44.8 mmol, 3.9 equiv) was added slowly over 20 minutes, the resulting solution was cooled to 0 °C. To the solution was added nicotinoyl chloride•HCl (2.032 g, 11.41 mmol, 1.0 equiv) and the mixture was allowed to warm to rt and stirred for 23 h, The reaction mixture was quenched with sat. aq. $NaHCO_3$ (10 mL), and the organic layer was separated. The aqueous layer was then neutralized with 1 M HCl and extracted with CH_2Cl_2 (3 X 50 mL); the combined organic layers were washed with brine (2 X 100 mL) and dried over Na_2SO_4. The resulting solution was concentrated *in vacuo* and the crude product was purified using flash column chromatography (95:5, CH_2Cl_2:CH_3OH), affording **42** as a yellow oil (1.511 g, 80%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.96 (s, 1H), 8.69 (d, *J* = 4.7 Hz, 1H), 8.03 (d, *J* = 7.8 Hz, 1H), 7.37 (dd, *J* = 7.8, 4.9 Hz, 1H), 3.56 (s, 3H), 3.40 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 167.4, 151.4, 149.3, 136.1, 129.8, 123.0, 61.3, 33.1. ^{1}H NMR and ^{13}C NMR data were consistent with literature values.[13]



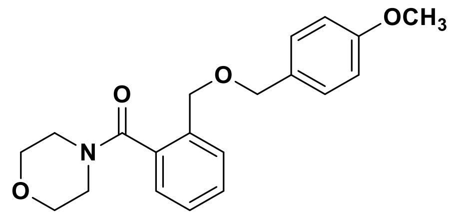
(2-(((4-methoxybenzyl)oxy)methyl)phenyl)(pyridin-3-yl)methanone (43) was synthesized according to General Procedure J, using **37** (1.008 g, 3.282 mmol) in THF (12.2 mL), 2.13 M *n*-butyllithium in hexanes (2.0 mL, 4.3 mmol), and **42** (0.649 g, 3.91 mmol) in THF (4.9 mL), reacting for 16 h. The crude product was purified using flash column chromatography (60:40, Et_2O:hexanes), affording **43** as a white oil (0.974 g, 89%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.94 (s, 1H), 8.77 (d, *J* = 4.7 Hz, 1H), 8.07 (d, *J* = 7.9 Hz, 1H), 7.57 (d, *J* = 7.6 Hz, 1H), 7.52 (t, *J* = 7.2 Hz, 1H), 7.42 – 7.33 (m, 3H), 7.08 (d, *J* = 8.4 Hz, 2H), 6.77 (d, *J* = 8.4 Hz, 2H), 4.64 (s, 2H), 4.33 (s, 2H), 3.77 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 196.6, 159.2, 153.3, 151.5, 138.5, 137.2, 137.0, 133.2, 131.1, 129.9, 129.5, 129.0, 128.9, 127.4, 123.4, 113.8, 72.7, 69.8, 55.4; AMM (ESI) *m/z* calcd for $C_{21}H_{20}NO_3^+$ $[M+H]^+$ 334.1438, found 334.1443.



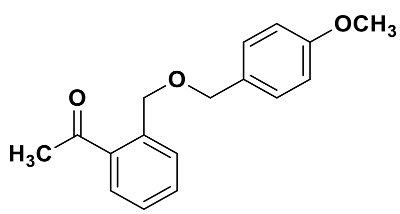
((2-(((4-methoxybenzyl)oxy)methyl)phenyl)(pyridin-3-yl)methylene)hydrazine (10e) was synthesized according to General Procedure O, using **43** (0.321 g, 0.963 mmol) in anhydrous EtOH (9.6 mL), hydrazine (0.17 mL, 5.4 mmol), and glacial acetic acid (0.06 mL, 1 mmol), reacting for 4.5 h at 155 °C. The crude product was purified using flash column chromatography (40:60, hexanes:EtOAc), affording **10e** as a dense yellow oil (0.204 g, 61%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.63 (s, 1H), 8.49 (d, *J* = 4.7 Hz, 1H), 7.74 (d, *J* = 8.1 Hz, 1H), 7.66 (d, *J* = 7.5 Hz, 1H), 7.53 – 7.44 (m, 2H), 7.19 (dd, *J* = 8.0, 4.8 Hz, 1H), 7.17 (d, *J* = 7.2 Hz, 1H), 7.14 (d, *J* = 8.5 Hz, 2H), 6.82 (d, *J* = 8.6 Hz, 2H), 5.48 (s, 2H), 4.40 – 4.35 (m, 2H), 4.35 – 4.29 (m, 2H), 3.79 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 159.3, 149.0, 147.7, 145.5, 137.4, 133.8, 133.0, 131.2, 130.0, 129.8, 129.5, 129.2, 129.0, 123.2, 113.9, 72.8, 69.9, 55.4, 14.3.



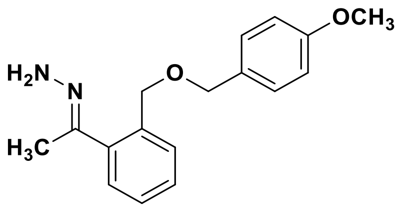
(3*S*,4*S*)-3-(4-methoxy)phenyl-4-(pyridin-3-yl)lisochromane (11e) was synthesized according to General Procedure R, using MnO_2 (0.050 g, 0.58 mmol), **10e** (0.025 g, 0.072 mmol) in CH_3CN (4.8 mL), reacting for 1 h. Rh_2(*R*-PTAD)$_4$ (0.001 g, 0.8 μmol) was added, reacting for 38 h. The crude product was purified using flash column chromatography (40:60, hexanes:EtOAc), affording **11e** as a white solid (0.019 g, >95:5 dr, 94:6 er, 82%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.29 (d, *J* = 4.5 Hz, 1H), 7.95 (s, 1H), 7.30 – 7.25 (m, 1H), 7.21 – 7.16 (m, 2H), 7.13 (d, *J* = 7.7 Hz, 1H), 7.06 – 6.96 (m, 2H), 6.87 (d, *J* = 8.4 Hz, 2H), 6.70 (d, *J* = 8.5 Hz, 2H), 5.23 (d, *J* = 15.3 Hz, 1H), 5.12 (d, *J* = 15.3 Hz, 1H), 5.09 (d, *J* = 2.9 Hz, 1H), 4.10 (d, *J* = 2.3 Hz, 1H), 3.73 (s, 3H); ^{13}C NMR (151 MHz, CD_2Cl_2) δ 158.5, 150.7, 147.3, 137.1, 136.7, 136.1, 134.4, 132.1, 129.9, 126.9, 126.9, 126.8, 124.2, 122.4, 113.0, 78.9, 69.0, 55.0, 47.7; AMM (CI-TOF) *m/z* calcd $C_{21}H_{20}NO_2^+$ [M+H]$^+$ 318.1494, found 318.1490; m.p. 181-183 °C; $[\alpha]_D^{20} = -217.1$ (c = 0.57, $CHCl_3$).



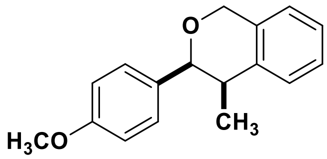
(2-(((4-methoxybenzyl)oxy)methyl)phenyl)(morpholino)methanone (44) was synthesized using General Procedure D using ether **34** (1.000 g, 3.255 mmol), morpholine (0.43 mL, 4.9 mmol), palladium (II) acetate (0.018 g, 0.081 mmol), Xantphos (0.094 g, 0.16 mmol), potassium phosphate tribasic (2.073 g, 9.765 mmol), CO (1 balloon), in toluene (6.5 mL). The crude product was purified using flash column chromatography (40:60, hexanes:EtOAc) affording **44** as a pale yellow oil (0.800 g, 84%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.44 (d, *J* = 7.6 Hz, 1H), 7.40 – 7.26 (m, 4H), 7.18 (d, *J* = 7.1 Hz, 1H), 6.94 – 6.85 (m, 2H), 4.51 (s, 2H), 3.81 (s, 3H), 3.78 – 3.61 (m, 4H), 3.55 – 3.41 (m, 2H), 3.19 (s, 2H), 1.62 (s, 2H). ^{1}H NMR was taken at rt and shows a mixture of rotamers.



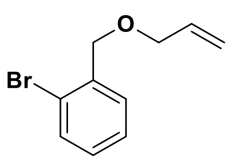
1-(2-(((4-methoxybenzyl)oxy)methyl)phenyl)ethan-1-one (45) was synthesized according to General Procedure E using morpholine amide **44** (0.552 g, 1.62 mmol) and methyllithium (1.52 M in diethyl ether, 1.28 mL, 1.94 mmol) in THF (9.5 mL). The crude product was purified using flash column chromatography (80:20 hexanes: EtOAc) affording **45** as a yellow oil (0.267 g, 61%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.72 (d, *J* = 7.9 Hz, 2H), 7.50 (t, *J* = 7.6 Hz, 1H), 7.39 – 7.28 (m, 3H), 6.92 – 6.85 (m, 2H), 4.85 (s, 2H), 4.55 (s, 2H), 3.80 (s, 3H), 2.57 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 201.7, 159.3, 139.7, 136.6, 132.0, 130.6, 129.5, 129.4, 128.1, 127.0, 113.9, 72.8, 70.4, 55.4, 29.3; AMM (ESI-TOF) *m/z* calcd for $C_{17}H_{18}NaO_3^+$ [M+Na]$^+$ 293.1157, found 293.1157.



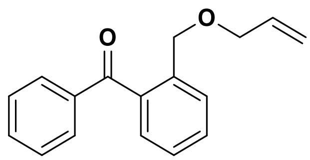
(1-(2-(((4-methoxybenzyl)oxy)methyl)phenyl)ethylidene)hydrazine (10f) was synthesized according to General Procedure O using ketone **45** (0.267 g, 0.988 mmol), hydrazine (0.31 mL, 9.8 mmol), acetic acid (0.07 mL, 1 mmol) and ethanol (6.6 mL). The crude product was purified using flash column chromatography (100:0 to 60:40 hexanes:EtOAc) affording **10f** as a yellow oil (0.0447 g, 16%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.60 – 7.50 (m, 1H), 7.42 – 7.33 (m, 2H), 7.32 – 7.23 (m, 2H), 7.13 – 7.03 (m, 1H), 6.88 (d, *J* = 8.0 Hz, 2H), 4.88 (s, 2H), 4.50 (s, 2H), 4.42 (s, 2H), 3.80 (s, 3H), 2.14 (s, 3H).



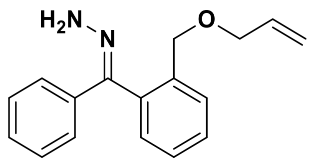
(3*S*,4*R*)-3-(4-methoxyphenyl)-4-methylisochromane (11f) was synthesized according to General Procedure R using **10f** (0.022 g, 0.077 mmol), MnO_2 (0.054 g, 0.62 mmol), Rh_2(*R*-PTAD)$_4$ (0.001 g, 0.0008 mmol) and CH_2Cl_2 (5 mL). The crude product was purified using flash column chromatography (90:10 to 80:20 hexanes:EtOAc) affording **11f** as a clear oil (0.011 g, 56%, >95:5 dr, 60:40 er). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.29 (d, *J* = 8.2 Hz, 2H), 7.23 – 7.14 (m, 3H), 7.08 – 7.02 (m, 1H), 6.92 (d, *J* = 8.3 Hz, 2H), 5.05 (d, *J* = 15.1 Hz, 1H), 4.98 (d, *J* = 15.2 Hz, 1H), 4.86 (d, *J* = 2.8 Hz, 1H), 3.82 (s, 3H), 3.03 – 2.91 (m, 1H), 0.97 (d, *J* = 7.0 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 158.6, 140.5, 134.0, 133.3, 129.0, 126.9, 126.6, 126.3, 124.2, 113.7, 78.4, 69.0, 55.4, 38.1, 17.2; AMM (CI-TOF) *m/z* calcd for $C_{17}H_{18}O_2^+$ [M]$^+$ 254.1307, found 253.1304; $[\alpha]_D^{22} = -3.4$ (c = 0.07, $CHCl_3$).



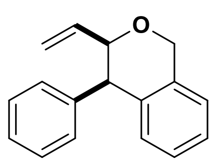
2-((allyloxy)methyl)bromobenzene (46) was synthesized according to General Procedure A, using NaH (1.216 g, 30.31 mmol) in THF (37.9 mL), 2-bromobenzyl bromide (3.003 g, 12.02 mmol) in THF (15.0 mL), and allyl alcohol (1.03 mL, 14.7 mmol), reacting for 47 h at 60 °C. The crude product was purified using flash column chromatography (95:5, hexanes:EtOAc), affording **46** as a clear oil (2.251 g, 82%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.53 (d, *J* = 7.9 Hz, 1H), 7.50 (d, *J* = 7.3 Hz, 1H), 7.31 (t, *J* = 7.5 Hz, 1H), 7.14 (t, *J* = 7.6 Hz, 1H), 5.98 (ddt, *J* = 16.3, 10.6, 5.5 Hz, 1H), 5.39 – 5.31 (m, 1H), 5.23 (d, *J* = 10.4 Hz, 1H), 4.58 (s, 2H), 4.11 (d, *J* = 5.5 Hz, 2H).; ^{13}C NMR (151 MHz, $CDCl_3$) δ 137.8, 134.6, 132.6, 129.0, 128.9, 127.5, 122.7, 117.4, 71.8, 71.5; AMM (CI-TOF) *m/z* calcd for $C_{10}H_{11}BrO^+$ [M]$^+$ 225.9993, found 225.9993.



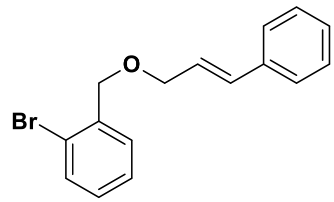
2-((allyloxy)methyl)benzophenone (47) was synthesized according to General Procedure J, using **46** (0.501 g, 2.21 mmol) in THF (8.2 mL), 2.13 M *n*-butyllithium in hexanes (1.34 mL, 2.86 mmol), and **31** (0.436 g, 2.64 mmol) in THF (3.3 mL), were combined and allowed to react for 20 h. The crude product was purified using flash column chromatography (90:10, hexanes:EtOAc), affording **47** as a clear oil (0.482 g, 87%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.80 (d, *J* = 7.3 Hz, 2H), 7.57 (t, *J* = 6.6 Hz, 2H), 7.49 (t, *J* = 7.3 Hz, 1H), 7.45 (t, *J* = 7.7 Hz, 2H), 7.38 – 7.33 (m, 2H), 5.74 (ddt, *J* = 16.1, 10.8, 5.4 Hz, 1H), 5.18 – 5.11 (m, 1H), 5.10 – 5.03 (m, 1H), 4.60 (s, 2H), 3.87 (d, *J* = 5.5 Hz, 2H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 198.0, 138.2, 137.8, 137.7, 134.4, 133.1, 130.5, 130.2, 128.9, 128.5, 128.4, 127.0, 117.1, 71.7, 69.8; AMM (ESI) *m/z* calcd for $C_{17}H_{17}O_2^+$ [M+H]$^+$ 253.1223, found 252.1226.



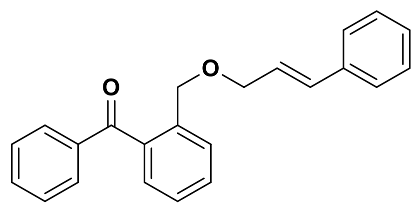
((2-((allyloxy)methyl)phenyl)(phenyl)methylene)hydrazine (12a) was synthesized according to General Procedure P in a sealed microwave vial, using ketone **47** (0.251 g, 0.996 mmol) in anhydrous EtOH (10.0 mL), hydrazine (0.19 mL, 6.0 mmol), and glacial acetic acid (0.07 mL, 1 mmol) reacting for 5 d at 90 °C. The crude product was purified using flash column chromatography (80:20, hexanes:EtOAc), affording **12a** as a yellow oil (0.132 g, 50%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.67 (d, *J* = 7.4 Hz, 1H), 7.53 – 7.40 (m, 4H), 7.32 – 7.23 (m, 3H), 7.17 (d, *J* = 7.2 Hz, 1H), 5.82 (ddt, *J* = 16.7, 10.6, 5.5 Hz, 1H), 5.36 (s, 2H), 5.19 (d, *J* = 17.3 Hz, 1H), 5.11 (d, *J* = 10.4 Hz, 1H), 4.39 – 4.25 (m, 2H), 3.92 (d, *J* = 5.5 Hz, 2H).



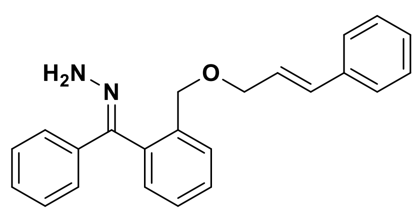
(3*R*,4*R*)-4-phenyl-3-vinylisochromane (13a) was synthesized according to General Procedure R, using MnO_2 (0.065 g, 0.75 mmol) and **12a** (0.026 g, 0.099 mmol) in CH_3CN (6.6 mL), reacting for 0.25 h. Rh_2(*R*-PTAD)$_4$ (0.002 g, 1 μmol) was added, reacting for 3 h. The crude product was purified using flash column chromatography (90:10, hexanes:EtOAc), affording **13a** as a white crystalline solid (0.017 g, >95:5 dr, 97:3 er, 73%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.27 – 7.16 (m, 4H), 7.16 – 7.07 (m, 4H), 7.02 (d, *J* = 7.5 Hz, 1H), 5.57 – 5.44 (m, 1H), 5.24 (d, *J* = 17.3 Hz, 1H), 5.13 – 5.03 (m, 2H), 4.99 (d, *J* = 15.2 Hz, 1H), 4.50 – 4.40 (m, 1H), 3.96 – 3.89 (m, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 141.7, 137.1, 137.0, 134.1, 130.3, 130.1, 128.0, 127.0, 126.6, 126.5, 124.2, 116.4, 79.3, 68.5, 48.7; AMM (CI-TOF) *m/z* calcd for $C_{17}H_{20}NO^+$ [M+NH$_4$]$^+$ 254.1545, found 254.1554; m.p. 83-86 °C; $[\alpha]_D^{22} = -207.6$ (c = 0.26, $CHCl_3$).



2-((cinnamyloxy)methyl)bromobenzene (48) was synthesized according to General Procedure A, using NaH (0.961 g, 24.0 mmol) in THF (30 mL), 2-bromobenzyl bromide (2.011 g, 8.047 mmol) in THF (10.1 mL), *trans*-cinnamyl alcohol (1.623 g, 12.09 mmol), reacting for 20 h. The crude product was purified using flash column chromatography (95:5, hexanes:EtOAc), affording **48** as a clear oil (1.793 g, 74%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.57 – 7.50 (m, 2H), 7.41 (d, *J* = 7.4 Hz, 2H), 7.32 (t, *J* = 7.6 Hz, 3H), 7.25 (t, *J* = 7.3 Hz, 1H), 7.15 (t, *J* = 8.2 Hz, 1H), 6.67 (d, *J* = 15.9 Hz, 1H), 6.36 (dt, *J* = 15.9, 6.0 Hz, 1H), 4.64 (s, 2H), 4.28 (d, *J* = 6.0 Hz, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 137.8, 136.8, 132.8, 132.7, 129.3, 129.1, 128.7, 127.9, 127.6, 126.7, 126.0, 122.9, 71.6, 71.5; AMM (CI-TOF) *m/z* calcd for $C_{16}H_{15}BrO^+$ $[M]^+$ 302.0306, found 302.0291.

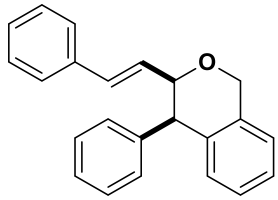


2-((cinnamyloxy)methyl)benzophenone (49) was synthesized according to General Procedure J, using **48** (1.005 g, 3.30 mmol) in THF (12.2 mL), 2.13 M *n*-butyllithium in hexanes (2.01 mL, 4.29 mmol), and **31** (0.654 g, 3.96 mmol) in THF (5.0 mL), reacting for 36 h. The crude product was purified using flash column chromatography (80:20, hexanes:EtOAc), affording **49** as a yellow oil (0.530 g, 49%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.81 (d, *J* = 7.7 Hz, 2H), 7.57 (t, *J* = 7.7 Hz, 2H), 7.53 – 7.46 (m, 1H), 7.43 (t, *J* = 7.5 Hz, 2H), 7.40 – 7.31 (m, 2H), 7.32 – 7.17 (m, 5H), 6.44 (d, *J* = 15.9 Hz, 1H), 6.08 (dt, *J* = 15.8, 5.9 Hz, 1H), 4.65 (s, 2H), 4.03 (d, *J* = 5.9 Hz, 2H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 198.1, 138.1, 138.0, 137.7, 136.8, 133.2, 132.4, 130.5, 130.2, 128.8, 128.7, 128.6, 128.5, 127.7, 127.2, 126.6, 125.7, 71.4, 70.0; AMM (ESI) *m/z* calcd for $C_{23}H_{21}O_2^+$ $[M+H]^+$ 329.1536, found 329.1533. NOTE: compound was found to degrade over several days and should be used immediately.

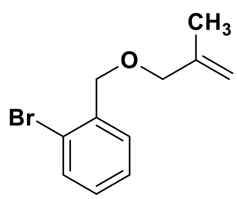


((2-((cinnamyloxy)methyl)phenyl)(phenyl)methylene)hydrazine (12b) was synthesized according to General Procedure P using a sealed microwave vial, using **49** (0.309 g, 0.939 mmol) in anhydrous EtOH (9.4 mL), hydrazine (0.19 mL, 6.0 mmol), and glacial acetic acid (0.07 mL, 1 mmol), reacting for 3 d at 100 °C. The crude product was purified using flash column chromatography (80:20, hexanes:EtOAc), affording **12b** as a yellow amorphous solid (0.224 g, 70%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.69 (d, *J* = 7.3 Hz, 1H), 7.56 – 7.39 (m, 4H), 7.34 – 7.22 (m, 8H), 7.18 (d, *J* =

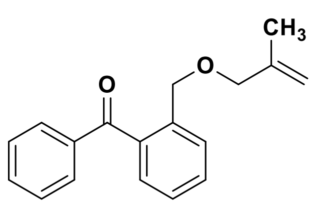
7.2 Hz, 1H), 6.50 (d, J = 16.0 Hz, 1H), 6.16 (dt, J = 15.8, 6.0 Hz, 1H), 5.37 (s, 2H), 4.46 – 4.27 (m, 2H), 4.07 (d, J = 5.9 Hz, 2H); AMM (ESI) m/z calcd for $C_{24}H_{25}N_2O^+$ $[M+H]^+$ 343.1805, found 343.1814.



(3*R*,4*R*)-4-phenyl-3-((*E*)-styryl)isochromane (13b) was synthesized according to General Procedure R, using MnO_2 (0.051 g, 0.58 mmol) and **12b** (0.022 g, 0.065 mmol) in CH_3CN (4.3 mL), reacting for 3 h. Rh_2(*R*-PTAD)$_4$ (0.001 g, 0.7 µmol) was added, reacting for 14 h. The crude product was purified using flash column chromatography (90:10, hexanes:EtOAc), affording **13b** as a white crystalline solid (0.019 g, >95:5 dr, 98:2 er, 95%). **13b** was crystallized out of hexanes:*i*-PrOH (90:10) with a small amount of CH_2Cl_2 to dissolve fully followed by slow evaporation over 24 h. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.28 – 7.16 (m, 9H), 7.16 – 7.10 (m, 4H), 7.04 (d, J = 7.5 Hz, 1H), 6.57 (d, J = 16.0 Hz, 1H), 5.81 (dd, J = 15.9, 6.9 Hz, 1H), 5.13 (d, J = 15.2 Hz, 1H), 5.04 (d, J = 15.2 Hz, 1H), 4.61 (dd, J = 6.4, 2.9 Hz, 1H), 4.02 (d, J = 2.6 Hz, 1H).; ^{13}C NMR (101 MHz, $CDCl_3$) δ 141.6, 137.0, 137.0, 134.2, 131.6, 130.3, 130.1, 128.6, 128.6, 128.0, 127.7, 127.0, 126.7, 126.7, 126.6, 124.2, 79.1, 68.6, 49.2; AMM (CI-TOF) m/z calcd for $C_{23}H_{20}O^+$ $[M]^+$ 312.1514, found 312.1526; m.p. 106-107 °C; $[\alpha]_D^{21} = -431.0$ (c = 0.22, $CHCl_3$).

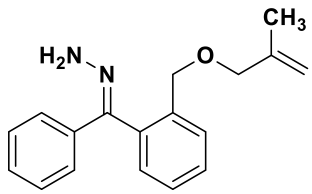


2-(((2-methylallyl)oxy)methyl)bromobenzene (50) was synthesized according to General Procedure A, using NaH (0.960 g, 24.0 mmol) in THF (30 mL), 2-bromobenzyl bromide (2.008 g, 8.033 mmol) in THF (10.0 mL), and 2-methallyl alcohol (1.01 mL, 12.0 mmol), reacting for 48 h at 60 °C. The crude product was purified using flash column chromatography (80:20, hexanes:CH_2Cl_2), affording **50** as a clear oil (1.438 g, 75%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.53 (d, J = 8.0 Hz, 1H), 7.51 (d, J = 7.8 Hz, 1H), 7.32 (t, J = 7.5 Hz, 1H), 7.14 (t, J = 7.6 Hz, 1H), 5.04 (s, 1H), 4.95 (s, 1H), 4.56 (s, 2H), 4.02 (s, 2H), 1.79 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 142.2, 138.0, 132.6, 129.1, 128.9, 127.5, 122.7, 112.6, 74.8, 71.3, 19.7; AMM (CI-TOF) m/z calcd for $C_{11}H_{14}BrO^+$ $[M+H]^+$ 241.0228, found 241.0224.

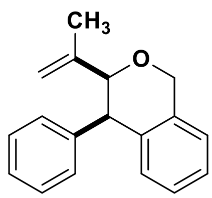


2-(((2-methylallyl)oxy)methyl)benzophenone (51) was synthesized according to General Procedure J, using **50** (1.913 g, 4.200 mmol) in THF (15.6 mL), 2.13 M *n*-butyllithium in hexanes (2.53 mL, 5.33 mmol), and **31** (0.822 g, 4.98 mmol) in THF (6.2 mL) were combined and allowed to react for 18 h. The crude product was purified using flash column chromatography (95:5, hexanes:EtOAc), affording **51** as a clear oil (0.844 g, 75%). ^{1}H NMR (600 MHz,

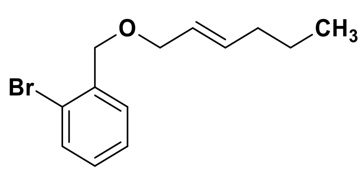
$CDCl_3$) δ 7.80 (d, *J* = 7.5 Hz, 2H), 7.61 – 7.54 (m, 2H), 7.49 (t, *J* = 7.9 Hz, 1H), 7.45 (t, *J* = 7.7 Hz, 2H), 7.39 – 7.32 (m, 2H), 4.84 (s, 1H), 4.80 (s, 1H), 4.59 (s, 2H), 3.79 (s, 2H), 1.61 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 198.0, 142.0, 138.3, 137.8, 137.7, 133.1, 130.6, 130.2, 128.9, 128.5, 128.4, 127.0, 112.3, 74.8, 69.7, 19.5; AMM (ESI) *m/z* calcd for $C_{18}H_{19}O_2^+$ [M+H]$^+$ 267.1380, found 267.1380.



((2-(((2-methallyl)oxy)methyl)phenyl)(phenyl)methylene)hydrazine (12c) was synthesized according to General Procedure P in a sealed microwave vial, using **51** (0.308 g, 1.16 mmol) in anhydrous EtOH (11.6 mL), hydrazine (0.21 mL, 6.8 mmol), and glacial acetic acid (0.08 mL, 1 mmol), reacting for 3.5 d at 100 °C. The crude product was purified using flash column chromatography (80:20, hexanes:EtOAc), affording **12c** as a yellow oil (0.251 g, 77%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.68 (d, *J* = 7.4 Hz, 1H), 7.53 – 7.40 (m, 4H), 7.32 – 7.23 (m, 3H), 7.16 (d, *J* = 7.2 Hz, 1H), 5.36 (s, 2H), 4.89 (s, 1H), 4.83 (s, 1H), 4.32 (d, *J* = 12.2 Hz, 1H), 4.26 (d, *J* = 12.2 Hz, 1H), 3.83 (s, 2H), 1.67 (s, 3H); AMM (ESI) *m/z* calcd for $C_{18}H_{21}N_2O^+$ [M+H]$^+$ 281.1648, found 281.1646.

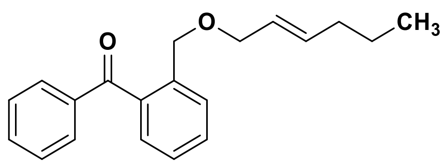


(3*S*,4*R*)-4-phenyl-3-((1-methyl)vinyl)isochromane (13c) was synthesized according to General Procedure R, using MnO_2 (0.064 g, 0.71 mmol) and **12c** (0.022 g, 0.078 mmol) in CH_3CN (5.2 mL), reacting for 1.5 h. Rh_2(*R*-PTAD)$_4$ (0.001 g, 0.9 µmol) was added, reacting for 16 h. The crude product was purified using flash column chromatography (90:10, hexanes:EtOAc), affording **13c** as a white solid (0.018 g, >95:5 dr, 95:5 er, 90%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.23 – 7.13 (m, 6H), 7.13 – 7.07 (m, 2H), 7.03 (d, *J* = 7.5 Hz, 1H), 5.13 (d, *J* = 15.2 Hz, 1H), 4.98 (d, *J* = 15.2 Hz, 1H), 4.81 (s, 1H), 4.72 (s, 1H), 4.39 (s, 1H), 4.06 – 3.97 (m, 1H), 1.55 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 143.3, 141.9, 137.4, 134.1, 130.4, 129.8, 127.8, 126.9, 126.6, 126.5, 124.2, 111.4, 80.8, 68.9, 47.5, 19.7; AMM (CI-TOF) *m/z* calcd for $C_{18}H_{22}NO^+$ [M+NH$_4$]$^+$ 268.1701, found 268.1702; m.p. 88-90 °C; $[\alpha]_D^{22} = -225.1$ (c = 0.46, $CHCl_3$).

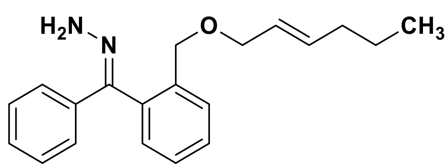


2-((*E*-hex-2-en-1-yloxy)methyl)bromobenzene (52) was synthesized according to General Procedure A, using NaH (1.616 g, 40.40 mmol) in THF (50.5 mL), 2-bromobenzyl bromide (2.017 g, 8.071 mmol) in THF (10.1 mL), and *E*-hexen-1-ol (1.42 mL, 12.0 mmol), reacting for 20 h at rt. The crude product was purified using flash column chromatography (90:10, hexanes:EtOAc), affording **52** as a yellow oil (2.057 g, 95%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.53 (d, *J* = 8.0 Hz, 1H), 7.49 (d, *J* = 7.6 Hz, 1H), 7.31 (t, *J* = 7.5 Hz, 1H), 7.14 (t, *J* = 7.7 Hz, 1H), 5.75 (dt, *J* = 14.5, 6.7

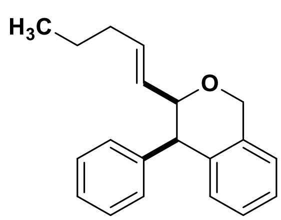
Hz, 1H), 5.66 – 5.59 (m, 1H), 4.56 (s, 2H), 4.05 (d, *J* = 6.2 Hz, 2H), 2.05 (app. q, *J* = 14.6, 7.2 Hz, 2H), 1.43 (sextet, *J* = 7.4 Hz, 2H), 0.92 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 138.0, 135.2, 132.6, 129.2, 128.9, 127.5, 126.3, 122.8, 71.7, 71.2, 34.6, 22.4, 13.9; AMM (CI-TOF) *m/z* calcd for $C_{13}H_{17}BrO^+$ $[M]^+$ 268.0463, found 268.0468.



2-((*E*-hex-2-en-1-yloxy)methyl)benzophenone (53) was synthesized according to General Procedure J, using **52** (1.002 g, 3.723 mmol) in THF (13.8 mL), 2.13 M *n*-butyllithium in hexanes (2.27 mL, 4.83 mmol), and **31** (0.736 g, 4.46 mmol) in THF (5.6 mL), reacting for 18 h. The crude product was purified using flash column chromatography (95:5, hexanes:EtOAc), affording **53** as a clear oil (0.756 g, 71%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.80 (d, *J* = 7.6 Hz, 2H), 7.60 – 7.54 (m, 2H), 7.51 – 7.42 (m, 3H), 7.38 – 7.32 (m, 2H), 5.54 (dt, *J* = 14.1, 6.7 Hz, 1H), 5.36 (dt, *J* = 14.6, 6.2 Hz, 1H), 4.56 (s, 2H), 3.81 (d, *J* = 6.2 Hz, 2H), 1.92 (app. q, *J* = 14.2, 7.1 Hz, 2H), 1.31 (sextet, *J* = 7.4 Hz, 2H), 0.84 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 198.1, 138.3, 138.0, 137.8, 134.8, 133.1, 130.5, 130.2, 128.8, 128.6, 128.5, 127.0, 126.0, 71.5, 69.5, 34.5, 22.3, 13.8; AMM (ESI) *m/z* calcd for $C_{20}H_{23}O_2^+$ $[M+H]^+$ 295.1693, found 295.1694.

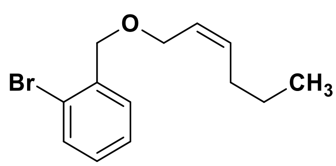


((2-((*E*-hex-2-en-1-yloxy)methyl)phenyl)(phenyl)methylene)hydrazine (12d) was synthesized according to General Procedure O, using **53** (0.301 g, 1.02 mmol) in anhydrous EtOH (10.2 mL), hydrazine (0.20 mL, 6.1 mmol), and glacial acetic acid (0.07 mL, 1 mmol) reacting for 5 h at 160 °C. The crude product was purified using flash column chromatography (80 hexanes :20 EtOAc), affording **12d** as a white solid (0.244 g, 77%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.66 (d, *J* = 7.2 Hz, 1H), 7.51 – 7.40 (m, 4H), 7.29 – 7.25 (m, 3H), 7.16 (dd, *J* = 7.3, 1.4 Hz, 1H), 5.65 – 5.54 (m, 1H), 5.49 – 5.40 (m, 1H), 5.36 (s, 2H), 4.35 – 4.21 (m, 2H), 3.86 (d, *J* = 6.1 Hz, 2H), 1.95 (app. q, *J* = 7.5, 7.2 Hz, 2H), 1.34 (sextet, *J* = 7.3 Hz, 2H), 0.86 (t, *J* = 7.4 Hz, 3H).

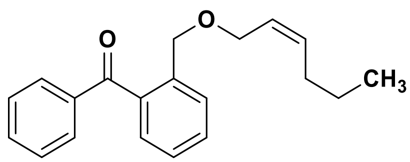


(3*R*,4*R*)-3-(*E*-pent-1-en-1-yl)-4-phenylisochromane (13d) was synthesized according to General Procedure R, using MnO_2 (0.059 g, 0.68 mmol) and **12d** (0.020 g, 0.069 mmol) in CH_3CN (4.6 mL), reacting for 1.5 h. $Rh_2(R\text{-PTAD})_4$ (0.001 g, 0.8 μmol) was added, reacting for 16 h. The crude product was purified using flash column chromatography (90:10, hexanes:EtOAc), affording **13d** as a clear oil (0.019 g, >95:5 dr, 97:3 er, 97%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.24 – 7.20 (m, 2H), 7.20 – 7.14 (m, 2H), 7.14 – 7.07 (m, 4H), 7.01 (d, *J* = 7.6 Hz, 1H), 5.68 (dt, *J* = 14.4, 6.8 Hz, 1H), 5.11 – 5.04 (m, 2H), 4.98 (d, *J* = 15.2 Hz, 1H), 4.40 (dd, *J* = 7.1, 3.2 Hz, 1H), 3.89 (d, *J* = 2.7 Hz, 1H), 1.91 (app.

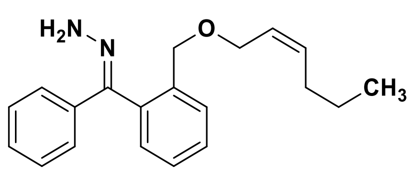
q, J = 14.2, 7.1 Hz, 2H), 1.29 (sextet, J = 14.6, 7.6 Hz, 2H), 0.80 (t, J = 7.4 Hz, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 141.9, 137.3, 134.2, 133.6, 130.3, 130.2, 128.9, 127.9, 126.9, 126.5, 126.4, 124.1, 79.2, 68.5, 49.1, 34.5, 22.3, 13.8; AMM (CI-TOF) *m/z* calcd for $C_{20}H_{26}NO^+$ $[M+NH_4]^+$ 296.2014, found 296.2002; $[\alpha]_D^{23} = -88.3$ (c = 0.52, $CHCl_3$).



2-((*Z*-hex-2-en-1-yloxy)methyl)bromobenzene (54) was synthesized according to General Procedure A, using NaH (1.611 g, 40.28 mmol) in THF (50.4), 2-bromobenzyl bromide (2.009 g, 8.036 mmol) in THF (10.0 mL), and *Z*-hexen-1-ol (1.42 mL, 12.0 mmol), reacting for 27 h at room temperature. The crude product was purified using flash column chromatography (95:5, hexanes:EtOAc), affording **54** as a clear oil (1.826g, 84%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.53 (d, J = 8.0 Hz, 1H), 7.49 (d, J = 7.6 Hz, 1H), 7.31 (t, J = 7.5 Hz, 1H), 7.14 (t, J = 7.6 Hz, 1H), 5.68 – 5.58 (m, 2H), 4.57 (s, 2H), 4.16 (d, J = 4.9 Hz, 2H), 2.06 (m, 2H), 1.41 (sextet, J = 7.3 Hz, 2H), 0.91 (t, J = 7.4 Hz, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 138.0, 134.1, 132.6, 129.2, 128.9, 127.5, 126.0, 122.8, 71.5, 66.5, 29.8, 22.8, 13.9; AMM (CI-TOF) *m/z* calcd for $C_{13}H_{17}BrO^+$ $[M]^+$ 268.0463, found 268.0475.

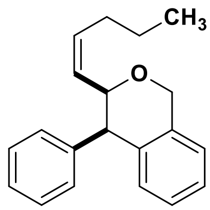


2-((*Z*-hex-2-en-1-yloxy)methyl)benzophenone (55) was synthesized according to General Procedure J, using **54** (1.002 g, 3.721 mmol) in THF (13.8 mL), 2.13 M *n*-butyllithium in hexanes (2.3 mL, 3.0 mmol), and **31** (0.737 g, 4.46 mmol) in THF (5.6 mL), reacting for 21 h. The crude product was purified using flash column chromatography (95:5, hexanes:EtOAc), affording **55** as a clear oil (0.826 g, 75%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.80 (d, J = 7.9 Hz, 2H), 7.60 – 7.54 (m, 2H), 7.48 (t, J = 7.2 Hz, 1H), 7.45 (t, J = 7.6 Hz, 2H), 7.38 – 7.32 (m, 2H), 5.52 – 5.45 (m, 1H), 5.41 – 5.35 (m, 1H), 4.58 (s, 2H), 3.93 (d, J = 6.5 Hz, 2H), 1.93 (app. q, J = 14.7, 7.3 Hz, 2H), 1.31 (sextet, J = 7.3 Hz, 2H), 0.85 (t, J = 7.4 Hz, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 198.0, 138.3, 137.9, 137.8, 133.6, 133.1, 130.5, 130.2, 128.9, 128.6, 128.5, 127.0, 125.9, 69.8, 66.5, 29.7, 22.8, 13.8; AMM (ESI-TOF) *m/z* calcd for $C_{20}H_{23}O_2^+$ $[M+H]^+$ 295.1693, found 295.1689.

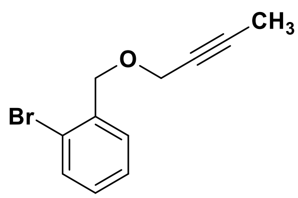


((2-((*Z*-hex-2-en-1-yloxy)methyl)phenyl)(phenyl)methylene)hydrazine (12e) was synthesized according to General Procedure O, using **55** (0.302 g, 1.03 mmol) in anhydrous EtOH (10.3 mL), hydrazine (0.22 mL, 7.0 mmol), and glacial acetic acid (0.08 mL, 1 mmol) reacting for 5.5 h at 150 °C. The crude product was purified using flash column chromatography (80:20, hexanes:EtOAc), affording **12e** as a clear oil (0.245 g, 77%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.65 (d, J = 7.6 Hz, 1H), 7.47 (t, J = 7.5 Hz, 1H), 7.46 – 7.38 (m, 3H), 7.31 – 7.22 (m, 3H), 7.15 (d, J = 7.3 Hz, 1H), 5.55 – 5.41 (m, 2H), 5.36 (s, 2H), 4.36 – 4.22 (m, 2H), 4.01 – 3.90 (m, 2H), 1.93 (app. q, J = 14.5, 7.3 Hz, 2H), 1.32

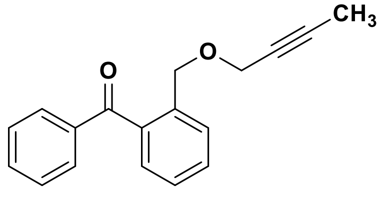
(sextet, J = 7.4 Hz, 2H), 0.84 (t, J = 7.4 Hz, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 148.3, 137.9, 137.4, 133.5, 131.9, 129.3, 129.0, 128.7, 128.6, 128.2, 128.1, 125.9, 125.9, 69.7, 66.5, 29.5, 22.6, 13.7.



(3*R*,4*R*)-3-(*Z*-pent-1-en-1-yl)-4-phenylisochromane (13e) was synthesized according to General Procedure R, using MnO_2 (0.056 g, 0.65 mmol), **12e** (0.021 g, 0.068 mmol) in CH_3CN (4.5 mL), reacting for 0.5 h. $Rh_2(R\text{-PTAD})_4$ (0.001 g, 0.8 µmol) was added, reacting for 2.5 h. The crude product was purified using flash column chromatography (90:10, hexanes:EtOAc), affording **13e** as a clear oil (0.017 g, >95:5 dr, 95:5 er, 92%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.25 – 7.16 (m, 4H), 7.16 – 7.08 (m, 4H), 7.02 (d, J = 7.6 Hz, 1H), 5.53 – 5.41 (m, 1H), 5.07 (d, J = 15.2 Hz, 1H), 5.01 (d, J = 15.2 Hz, 1H), 4.96 (dd, J = 9.6, 8.6 Hz, 1H), 4.76 (dd, J = 8.3, 3.0 Hz, 1H), 3.85 (d, J = 2.7 Hz, 1H), 2.16 – 2.06 (m, 2H), 1.44 (sextet, J = 7.1 Hz, 2H), 0.94 (t, J = 7.4 Hz, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 141.8, 137.3, 134.2, 132.4, 130.3, 130.2, 128.7, 127.9, 126.9, 126.6, 126.5, 124.2, 74.3, 68.4, 48.4, 30.4, 22.9, 14.0; AMM (CI-TOF) *m/z* calcd for $C_{20}H_{26}NO^+$ $[M+NH_4]^+$ 296.2014, found 296.2024; $[\alpha]_D^{22} = -146.2$ (c = 0.45, $CHCl_3$).

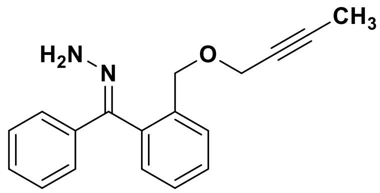


2-((but-2-yn-1-yloxy)methyl)bromobenzene (56) was synthesized according to General Procedure A, using NaH (1.216 g, 30.40 mmol) in THF (38.0 mL), 2-bromobenzyl bromide (3.001 g, 12.01 mmol) in THF (15.0 mL), and but-2-yn-1-ol (1.14 mL, 15.2 mmol), reacting for 15 h at 65 °C. The crude product was purified using flash column chromatography (95:5, hexanes:EtOAc), affording **56** as a clear oil (2.473 g, 86%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.54 (d, J = 7.9 Hz, 1H), 7.49 (d, J = 7.6 Hz, 1H), 7.31 (t, J = 7.5 Hz, 1H), 7.14 (t, J = 7.6 Hz, 1H), 4.65 (s, 2H), 4.22 (s, 2H), 1.88 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 137.3, 132.6, 129.4, 129.1, 127.5, 123.0, 83.1, 75.0, 71.0, 58.6, 3.8; AMM (CI-TOF) *m/z* calcd for $C_{11}H_{15}BrNO^+$ $[M+NH_4]^+$ 256.0332, found 256.0327.

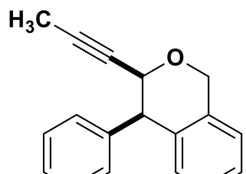


2-((but-2-yn-1-yloxy)methyl)benzophenone (57) was synthesized according to General Procedure J, using **56** (0.202 g, 0.846 mmol) in THF (3.1 mL), 2.13 M *n*-butyllithium in hexanes (0.51 mL, 1.1 mmol), and **31** (0.166 g, 1.01 mmol) in THF (1.3 mL), reacting for 21 h. The crude product was purified using flash column chromatography (90:10, hexanes:EtOAc), affording **57** as a clear oil (0.132 g, 59%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.80 (d, J = 7.9 Hz, 2H), 7.63

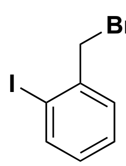
– 7.54 (m, 2H), 7.54 – 7.42 (m, 3H), 7.39 – 7.31 (m, 2H), 4.67 (s, 2H), 4.00 (s, 2H), 1.76 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 198.0, 138.0, 137.8, 137.8, 133.1, 130.7, 130.3, 129.0, 128.8, 128.5, 127.1, 82.9, 74.9, 69.1, 58.4, 3.7; AMM (ESI) *m/z* calcd for $C_{18}H_{17}O_2^+$ [M+H]$^+$ 265.1223, found 265.1235.



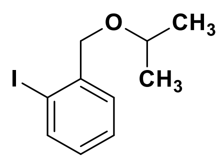
((2-((but-2-yn-1-yloxy)methyl)phenyl)(phenyl)methylene)hydrazine (12f) was synthesized according to General Procedure P, using **57** (0.132 g, 0.501 mmol) in anhydrous EtOH (5.0 mL), hydrazine (0.10 mL, 3.2 mmol), and glacial acetic acid (0.03 mL, 0.6 mmol) reacting for 3 d at 90 °C. The crude product was purified using flash column chromatography (80:20, hexanes:EtOAc), affording **12f** as a yellow oil (0.108 g, 78%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.67 (d, *J* = 7.3 Hz, 1H), 7.51 – 7.41 (m, 4H), 7.31 – 7.24 (m, 3H), 7.17 (d, *J* = 7.2 Hz, 1H), 5.37 (s, 2H), 4.44 – 4.30 (m, 2H), 4.04 (s, 2H), 1.76 (s, 3H).



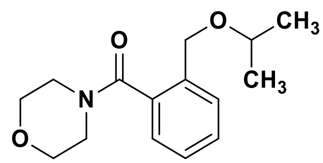
(3*S*,4*R*)-4-phenyl-3-(prop-1-yn-1-yl)isochromane (13f) was synthesized according to General Procedure R, using MnO_2 (0.063 g, 0.72 mmol), **12f** (0.025 g, 0.090 mmol) in CH_3CN (6.0 mL), reacting for 0.75 h. Rh_2(*R*-PTAD)$_4$ (0.001 g, 0.9 μmol) was added, reacting for 4 h. The crude product was purified using flash column chromatography (90:10, hexanes:EtOAc), affording **13f** as a clear oil (0.014 g, >95:5 dr, 92:8 er, 64%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31 – 7.17 (m, 6H), 7.13 (t, *J* = 7.4 Hz, 1H), 7.07 (d, *J* = 7.5 Hz, 1H), 6.99 (d, *J* = 7.6 Hz, 1H), 5.12 (d, *J* = 15.2 Hz, 1H), 4.91 (d, *J* = 15.2 Hz, 1H), 4.77 (br s, 1H), 4.13 (d, *J* = 3.5 Hz, 1H), 1.70 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 140.9, 135.8, 134.0, 130.4, 129.9, 127.8, 126.9, 126.9, 126.7, 124.1, 85.2, 75.9, 69.8, 67.4, 48.6, 3.7; AMM (ESI) *m/z* calcd for $C_{18}H_{17}O^+$ [M+H]$^+$ 249.1274, found 249.1275; $[\alpha]_D^{20} = -46.7$ (c = 0.69, $CHCl_3$).



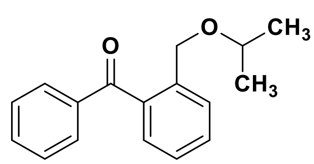
1-(bromomethyl)-2-iodobenzene (58). Following a literature procedure,[18] phosphorus tribromide (0.72 mL, 7.7 mmol) was added to a solution of 2-iodobenzyl alcohol (3.000 g, 12.82 mmol) in THF (64 mL) at 0 °C under argon. The reaction was stirred for one hour at 0 °C and then was concentrated *in vacuo* (NOTE: concentration *in vacuo* should be performed in a fume hood). The crude product was purified using flash column chromatography (85:15, hexanes:EtOAc) affording **58** as white crystals (3.685 g, 97%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.86 (d, *J* = 8.0 Hz, 1H), 7.48 (d, *J* = 7.6 Hz, 1H), 7.34 (t, *J* = 7.5 Hz, 1H), 6.98 (t, *J* = 7.7 Hz, 1H), 4.60 (s, 2H). ^{1}H NMR data matched literature values.[18]



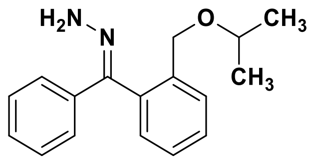
2-((isopropoxy)methyl)iodobenzene (59) was synthesized according to General Procedure A, using NaH (0.410 g, 17.1 mmol) in THF (21.4 mL), **58** (1.005 g, 3.385 mmol) in THF (4.2 mL), and *i*-PrOH (0.64 mL, 11 mmol, previously dried over activated 3 Å molecular sieves for a minimum of 24 h), reacting at 50 °C for 24 h. The crude product was purified using flash column chromatography (50:50, hexanes:CH_2Cl_2), affording **59** as a yellow oil (0.617 g, 66%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.80 (d, J = 7.9 Hz, 1H), 7.46 (d, J = 7.4 Hz, 1H), 7.34 (t, J = 7.5 Hz, 1H), 6.96 (t, J = 8.2 Hz, 1H), 4.47 (s, 2H), 3.75 (hept, J = 6.1 Hz, 1H), 1.26 (d, J = 6.1 Hz, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 141.3, 139.1, 129.0, 128.8, 128.3, 97.8, 74.2, 71.8, 22.3; AMM (CI-TOF) *m/z* calcd for $C_{10}H_{13}IO^+$ $[M]^+$ 276.0011, found 276.0015.



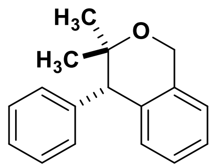
(isopropoxymethyl-phenyl)-morpholin-4-yl-methanone (60) was synthesized according to General Procedure D, using $Pd(OAc)_2$ (0.012 g, 0.052 mmol), Xantphos (0.058 g, 0.10 mmol), and potassium phosphate tribasic (1.271 g, 5.989 mmol), morpholine (0.26 mL, 3.0 mmol), and **59** (0.557 g, 2.00 mmol) in *m*-xylene (4.0 mL), reacting at 100 °C for 6.5 h. The crude product was purified using flash column chromatography (40:60, hexanes:EtOAc), affording **60** as a yellow oil (0.439 g, 83%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.45 (d, J = 7.5 Hz, 1H), 7.36 (t, J = 7.4 Hz, 1H), 7.30 (t, J = 7.4 Hz, 1H), 7.18 (d, J = 7.4 Hz, 1H), 4.61 (br s, 1H), 4.42 (s, 1H), 3.97 – 3.64 (m, 5H), 3.59 (s, 2H), 3.26 (s, 2H), 1.21 (d, J = 6.1 Hz, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.9, 136.2, 135.1, 129.4, 129.2, 127.7, 126.0, 71.9, 67.9, 66.8, 47.7, 42.0, 22.2; AMM (ESI) *m/z* calcd for $C_{15}H_{22}NO_3^+$ $[M+H]^+$ 264.1594, found 264.1594 . ^{1}H NMR and ^{13}C NMR were taken at rt and show a mixture of rotamers.



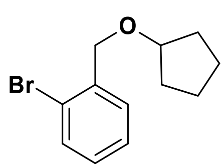
2-(isopropoxymethyl)benzophenone (61) was synthesized according to General Procedure E, using **60** (0.552 g, 2.10 mmol) in THF (21.0 mL), and 1.9 M phenyllithium in dibutyl ether (2.21 mL, 4.20 mmol), reacting for 2 h. The crude product was purified using flash column chromatography (90:10 to 80:20, hexanes:EtOAc), affording **61** as a yellow oil (0.385 g, 72%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.82 (d, J = 8.0 Hz, 2H), 7.60 – 7.53 (m, 2H), 7.50 – 7.42 (m, 3H), 7.33 (d, J = 3.8 Hz, 2H), 4.58 (s, 2H), 3.50 (hept, J = 6.1 Hz, 1H), 1.00 (d, J = 6.1 Hz, 6H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 198.1, 138.7, 138.1, 137.8, 133.1, 130.4, 130.3, 128.6, 128.6, 128.4, 126.9, 71.9, 67.9, 21.9; AMM (ESI) *m/z* calcd for $C_{17}H_{19}O_2^+$ $[M+H]^+$ 255.1380, found 255.1378.



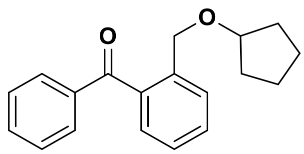
((2-((isopropyloxy)methyl)phenyl)(phenyl)methylene)hydrazine (14a) was synthesized according to General Procedure O, using **61** (0.385 g, 1.51 mmol) in anhydrous EtOH (15.1 mL), glacial acetic acid (0.10 mL, 1.8 mmol), and hydrazine (0.62 mL, 20 mmol), reacting at 170 °C for 3h. The crude product was purified using flash column chromatography (80:20, hexanes:EtOAc), affording **14a** as a yellow oil (0.308 g, 76%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.65 (d, *J* = 7.4 Hz, 1H), 7.51 – 7.39 (m, 4H), 7.32 – 7.22 (m, 3H), 7.16 (d, *J* = 7.2 Hz, 1H), 5.36 (s, 2H), 4.31 (d, *J* = 11.8 Hz, 1H), 4.26 (d, *J* = 11.8 Hz, 1H), 3.53 (hept, *J* = 6.1 Hz, 1H), 1.09 (d, *J* = 6.1 Hz, 3H), 1.04 (d, *J* = 6.1 Hz, 3H); AMM (ESI) *m/z* calcd for $C_{17}H_{21}N_2O^+$ $[M+H]^+$ 269.1648, found 269.1657; IR (neat): 3400, 2971, 2930, 1126, 1068 cm^{-1}.



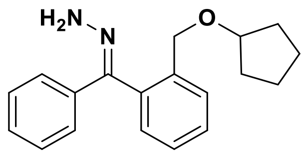
(*S*)-3,3-dimethyl-4-phenylisochromane (15a) was synthesized according to General Procedure R, using MnO_2 (0.130 g, 1.49 mmol) and **14a** (0.055 g, 0.19 mmol) in CH_3CN (12.7 mL), reacting for 30 min. Rh_2(*S*-PTAD)$_4$ (0.003 g, 2 μmol) was added, reacting for 4 h. The crude product was purified using flash column chromatography (50:50, hexanes:CH_2Cl_2), affording **15a** as a white crystalline solid (0.046 g, 96:4 er, 95%). ^{1}H NMR (600 MHz, CD_2Cl_2) δ 7.26 (s, 2H), 7.21 – 7.13 (m, 4H), 7.10 (t, *J* = 7.0 Hz, 2H), 6.95 (d, *J* = 7.8 Hz, 1H), 4.97 (d, *J* = 15.9 Hz, 1H), 4.90 (d, *J* = 15.9 Hz, 1H), 3.74 (s, 1H), 1.32 (s, 3H), 0.97 (s, 3H); ^{13}C NMR (101 MHz, CD_3CN) δ 143.7, 137.0, 133.6, 130.2, 129.6, 127.8, 126.4, 126.2, 126.0, 123.9, 73.0, 62.5, 52.7, 26.3, 24.6; AMM (CI-TOF) *m/z* calcd for $C_{17}H_{22}NO^+$ $[M+NH_4]^+$ 256.1701, found 256.1706; m.p. 105-106 °C; $[\alpha]_D^{22} = 119.2$ (c = 0.29, $CHCl_3$).



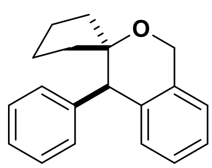
2-((cyclopentyloxy)methyl)bromobenzene (62) was synthesized according to General Procedure A, using NaH (0.960 g, 24.0 mmol) in THF (30.0 mL), 2-bromobenzyl bromide (2.002 g, 8.010 mmol) in THF (10.0 mL), and cyclopentanol (1.09 mL, 12.0 mmol, previously dried over activated 3 Å molecular sieves for a minimum of 24 h), reacting for 53 h. The crude product was purified using flash column chromatography (95:5, hexanes:EtOAc), affording **62** as a yellow oil (1.903 g, 93%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.51 (d, *J* = 8.0 Hz, 1H), 7.49 (d, *J* = 7.5 Hz, 1H), 7.30 (t, *J* = 7.5 Hz, 1H), 7.12 (t, *J* = 7.6 Hz, 1H), 4.52 (s, 2H), 4.09 – 4.02 (m, 1H), 1.81 – 1.68 (m, 6H), 1.61 – 1.50 (m, 2H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 138.5, 132.5, 129.1, 128.7, 127.5, 122.6, 81.7, 70.2, 32.5, 23.8; AMM (CI-TOF) *m/z* calcd for $C_{12}H_{15}BrO^+$ $[M]^+$ 254.0306, found 254.0305.



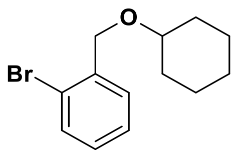
2-(cylopentyloxymethyl)benzophenone (63) was synthesized according to General Procedure J, using **62** (0.376 g, 1.25 mmol) in THF (4.6 mL), 1.68 M *n*-butyllithium in hexanes (0.96 mL, 1.7 mmol), and **31** (0.247 g, 1.50 mmol) in THF (1.9 mL), reacting for 20h. The crude product was purified using flash column chromatography (90:10, hexanes:EtOAc), affording **63** as a clear oil (0.248 g, 71%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.81 (d, *J* = 7.7 Hz, 2H), 7.62 – 7.50 (m, 2H), 7.50 – 7.39 (m, 3H), 7.33 (d, *J* = 4.0 Hz, 2H), 4.53 (s, 2H), 3.85 – 3.75 (m, 1H), 1.63 – 1.30 (m, 8H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 198.1, 138.6, 138.1, 137.7, 133.1, 130.3, 130.2, 128.6, 128.5, 128.4, 126.9, 81.7, 68.5, 32.0, 23.6; AMM (ESI-TOF) *m/z* calcd for $C_{19}H_{21}O_2^+$ $[M+H]^+$ 281.1536, found 281.1552.



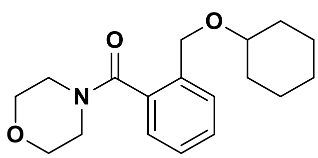
((2-((cyclopropyloxy)methyl)phenyl)(phenyl)methylene)hydrazine (14b) was synthesized according to General Procedure O, using **63** (0.303 g, 1.08 mmol) in anhydrous EtOH (10.8 mL), hydrazine (0.20 mL, 6.4 mmol), and glacial acetic acid (0.07 mL, 1 mmol), reacting for 8 hr at 170 °C. The crude product was purified using flash column chromatography (80:20, hexanes:EtOAc), affording **14b** as a clear oil (0.318 g, 100%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.63 (d, *J* = 7.3 Hz, 1H), 7.49 – 7.40 (m, 4H), 7.31 – 7.23 (m, 3H), 7.15 (d, *J* = 8.1 Hz, 1H), 5.36 (s, 2H), 4.24 (q, *J* = 11.9 Hz, 2H), 3.90 – 3.80 (m, 1H), 1.66 – 1.50 (m, 6H), 1.49 – 1.38 (m, 2H).



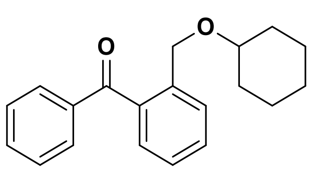
(4*R*)-spiro[cyclopentane-1,3'-(4-phenyl)isochromane] (15b) was synthesized according to General Procedure R, using MnO_2 (0.060 g, 0.69 mmol) and **14b** (0.023 g, 0.077 mmol) in CH_3CN (5.1 mL), reacting for 1 h. Rh_2(*R*-PTAD)$_4$ (0.001 g, 0.8 µmol) was added, reacting for 2 h. The crude product was purified using flash column chromatography (80:20, hexanes:EtOAc), affording **15b** as a clear oil (0.018 g, 97:3 er, 90%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.28 – 7.12 (m, 6H), 7.08 (t, *J* = 8.2 Hz, 2H), 6.99 (d, *J* = 7.5 Hz, 1H), 4.99 (d, *J* = 16.0 Hz, 1H), 4.91 (d, *J* = 16.0 Hz, 1H), 3.72 (s, 1H), 2.10 – 1.97 (m, 1H), 1.88 – 1.55 (m, 4H), 1.55 – 1.36 (m, 2H), 1.36 – 1.20 (m, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 143.9, 137.2, 133.6, 130.3, 129.5, 128.1, 126.6, 126.4, 126.3, 124.0, 85.4, 63.1, 52.2, 37.3, 34.4, 23.9, 23.1; Low-resolution MS (Advion ASAP-APCI) *m/z* calcd for $C_{19}H_{21}O^+$ $[M+H]^+$ 265.1587, found 265.0; $[\alpha]_D^{22} = -34.9$ (c = 0.20, $CHCl_3$).



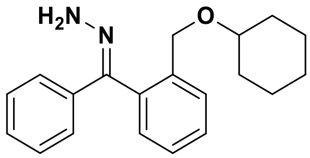
2-((cyclohexyloxy)methyl)bromobenzene (64) was synthesized according to General Procedure A, using NaH (0.480 g, 12.0 mmol) in THF (15.0 mL), 2-bromobenzyl bromide (1.034 g, 3.139 mmol) in THF (3.9 mL), and cyclohexanol (0.53 mL, 5.1 mmol, previously dried over activated 3 Å molecular sieves for a minimum of 24 h), reacting at 50 °C for 14 h. The crude product was purified using flash column chromatography (80:20, hexanes:EtOAc), affording **64** as a clear oil (0.832 g, 87%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.55 – 7.49 (m, 2H), 7.30 (t, *J* = 7.5 Hz, 1H), 7.11 (t, *J* = 7.7 Hz, 1H), 4.59 (s, 2H), 3.44 – 3.35 (m, 1H), 2.03 – 1.94 (m, 2H), 1.81 – 1.73 (m, 2H), 1.58 – 1.50 (m, 1H), 1.45 – 1.36 (m, 2H), 1.32 – 1.18 (m, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 138.7, 132.5, 129.0, 128.7, 127.4, 122.6, 77.8, 69.3, 32.4, 26.0, 24.2; AMM (CI-TOF) *m/z* calcd for $C_{13}H_{17}BrO^+$ $[M]^+$ 268.0463, found 268.0463.



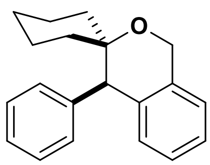
(cyclohexyloxymethyl-phenyl)-morpholin-4-yl-methanone (65) was synthesized according to General Procedure D, using $Pd(OAc)_2$ (0.010 g, 0.043 mmol), Xantphos (0.046 g, 0.080 mmol), and potassium phosphate tribasic (1.015 g, 4.780 mmol), morpholine (0.21 mL, 2.4 mmol), and **64** (0.430 g, 1.60 mmol) in *m*-xylene (3.2 mL), reacting at 100 °C for 17 h. The crude product was purified using flash column chromatography (30:70, hexanes:EtOAc), affording **65** as a clear oil (0.411 g, 47%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.45 (d, *J* = 7.6 Hz, 1H), 7.36 (t, *J* = 7.5 Hz, 1H), 7.30 (t, *J* = 7.5 Hz, 1H), 7.18 (d, *J* = 7.5 Hz, 1H), 4.65 (br s, 1H), 4.46 (br s, 1H), 3.97 – 3.65 (m, 4H), 3.60 (br d, *J* = 14.5 Hz, 2H), 3.42 – 3.33 (m, 1H), 3.27 (br d, *J* = 13.6 Hz, 2H), 1.98 (d, *J* = 9.6 Hz, 2H), 1.81 – 1.71 (m, 2H), 1.60 – 1.52 (m, 1H), 1.37 – 1.15 (m, 5H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 170.1, 136.4, 135.2, 129.5, 127.7, 126.1, 78.1, 67.7, 66.9, 47.8, 42.1, 32.5, 25.9, 24.3; AMM (ESI) *m/z* calcd for $C_{18}H_{26}NO_3^+$ $[M+H]^+$ 304.1907, found 304.1902; IR (neat): 2930, 2855, 1639, 1428, 1115 cm^{-1}. ^{1}H NMR and ^{13}C NMR were taken at rt and show a mixture of rotamers.



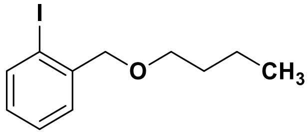
2-(cylohexyloxymethyl)benzophenone (66) was synthesized according to General Procedure E, using **65** (0.427 g, 1.41 mmol) in THF (14.1 mL), and 1.9 M phenyllithium in dibutyl ether (1.48 mL, 2.82 mmol), reacting for 2 h. The crude product was purified using flash column chromatography (90:10, hexanes:EtOAc), affording **66** as a yellow oil (0.339 g, 82%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.84 – 7.78 (m, 2H), 7.60 – 7.54 (m, 2H), 7.49 – 7.42 (m, 3H), 7.33 (d, *J* = 4.4 Hz, 2H), 4.61 (s, 2H), 3.22 – 3.14 (m, 1H), 1.76 – 1.67 (m, 2H), 1.65 – 1.55 (m, 2H), 1.48 – 1.40 (m, 1H), 1.19 – 1.06 (m, 5H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 198.1, 138.9, 138.1, 137.8, 133.1, 130.4, 130.3, 128.6, 128.6, 128.4, 126.8, 77.8, 67.6, 31.9, 25.9, 24.1; AMM (ESI) *m/z* calcd for $C_{20}H_{23}O_2^+$ $[M+H]^+$ 295.1693, found 295.1690; IR (neat): 2932, 2856, 1667, 1449, 1272 cm^{-1}.



((2-((cyclohexyloxy)methyl)phenyl)(phenyl)methylene)hydrazine (14c) was synthesized according to General Procedure O, using **66** (0.306 g, 1.04 mmol) in anhydrous EtOH (10.4 mL), glacial acetic acid (0.07 mL, 1 mmol), and hydrazine (0.19 mL, 6.1 mmol), reacting for 6 h at 170 °C. The crude product was purified using flash column chromatography (80:20, hexanes:EtOAc), affording **14c** as a yellow oil (0.312 g, 97%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.67 (d, *J* = 7.6 Hz, 1H), 7.49 – 7.40 (m, 4H), 7.31 – 7.22 (m, 3H), 7.15 (d, *J* = 7.3 Hz, 1H), 5.37 (s, 2H), 4.38 – 4.26 (m, 2H), 3.23 – 3.14 (m, 1H), 1.85 – 1.78 (m, 1H), 1.78 – 1.73 (m, 1H), 1.70 – 1.61 (m, 2H), 1.48 (s, 1H), 1.27 – 1.11 (m, 5H); AMM (ESI) *m/z* calcd for $C_{20}H_{25}N_2O^+$ $[M+H]^+$ 309.1961, found 309.1957.

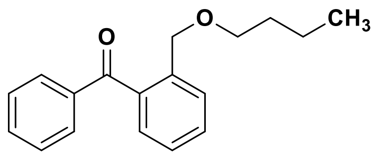


(*R*)-spiro[cyclohexane-1,3'-(4-phenyl)isochromane] (15c) was synthesized according to General Procedure S, using MnO_2 (0.056 g, 0.73 mmol) and **14c** (0.026 g, 0.08 mmol) in CH_2Cl_2 (5.3 mL), reacting for 2.5 h. $Rh_2(R\text{-PTAD})_4$ (0.001 g, 1 μmol) was added, reacting for 1.5 h. The crude product was purified using flash column chromatography (90:10, hexanes:EtOAc), affording **15c** as a white solid (0.019 g, 95:5 er, 80%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.26 – 7.20 (m, 2H), 7.19 – 7.10 (m, 4H), 7.10 – 7.04 (m, 2H), 6.95 (d, *J* = 7.6 Hz, 1H), 4.95 (d, *J* = 15.9 Hz, 1H), 4.87 (d, *J* = 15.9 Hz, 1H), 3.69 (s, 1H), 1.97 (d, *J* = 13.1 Hz, 1H), 1.62 – 1.51 (m, 4H), 1.47 – 1.40 (m, 1H), 1.35 (t, *J* = 11.1 Hz, 1H), 1.26 – 1.16 (m, 3H); ^{13}C NMR (101 MHz, CD_2Cl_2) δ 143.10, 136.77, 133.71, 130.46, 129.76, 127.76, 126.42, 126.12, 125.89, 123.79, 73.73, 62.01, 53.01, 34.96, 33.07, 25.83, 22.29, 21.57.; AMM (CI-TOF) *m/z* calcd for $C_{20}H_{26}NO^+$ $[M+NH_4]^+$ 296.2014, found 296.2029; m.p. 109-112 °C; $[\alpha]_D^{22} = -48.3$ (c = 0.20, $CHCl_3$). Note: compound appears to degrade rapidly on silica gel during purification, a very small silica gel plug was used to remove the catalyst from the reaction mixture.

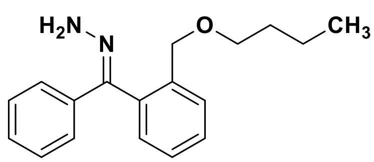


1-(butoxymethyl)-2-iodobenzene (67) was synthesized according to General Procedure C using 2-iodobenzyl alcohol (2.000 g, 8.546 mmol) in THF (4 mL), NaH (1.712 g, 42.80 mmol) in THF (10 mL), *n*-butyl bromide (4.58 mL, 42.8 mmol), and tetrabutylammonium iodide (0.380 g, 10.3 mmol). The crude product was purified using flash column chromatography (95:5 hexanes:EtOAc) affording **67** as a clear oil (2.257 g, 91%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.76 (d, *J* = 7.8 Hz, 1H), 7.42 (d, *J* = 7.5 Hz, 1H), 7.29 (t, *J* = 7.5 Hz, 1H), 6.95 – 6.87 (m, 1H), 4.43 (s, 2H), 3.52 (t, *J* =

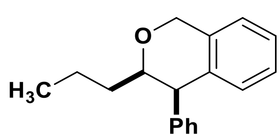
6.5 Hz, 2H), 1.67 – 1.57 (m, 2H), 1.48 – 1.35 (m, 2H), 0.92 (t, *J* = 7.4 Hz, 3H). ^{1}H NMR data was consistent with literature values.[14]



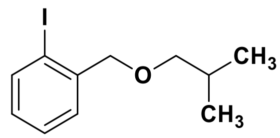
(2-(butoxymethyl)phenyl)(phenyl)methanone (68) was synthesized according to General Procedure N using **67** (0.300 g, 1.03 mmol), palladium (II) acetate (0.012 g, 0.052 mmol), silver (I) oxide (0.288 g, 1.24 mmol), benzaldehyde (0.84 mL, 8.3 mmol), and 70% *tert*-butyl hydroperoxide in H_2O (0.73 mL, 5.3 mmol). The crude product was purified using flash column chromatography (90:10 to 0:100 hexanes:CH_2Cl_2) affording **68** as a yellow oil (0.133 g, 48%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84 – 7.77 (m, 2H), 7.60 – 7.52 (m, 2H), 7.51 – 7.41 (m, 3H), 7.38 – 7.31 (m, 2H), 4.57 (s, 2H), 3.32 (t, *J* = 6.6 Hz, 2H), 1.44 – 1.33 (m, 2H), 1.27 – 1.15 (m, 2H), 0.79 (t, *J* = 7.3 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 198.0, 138.5, 137.9, 137.8, 133.1, 130.4, 130.2, 128.7, 128.5, 128.4, 126.9, 70.9, 70.6, 31.7, 19.3, 14.0; AMM (ESI) *m/z* calcd for $C_{18}H_{21}O_2^+$ [M+H]$^+$ 269.1536, found 269.1535; IR (neat): ν_{max} 2957, 1667, 1270 cm^{-1}.



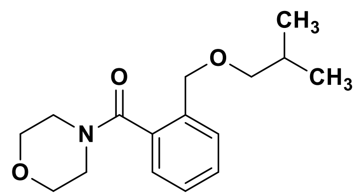
((2-(butoxymethyl)phenyl)(phenyl)methylene)hydrazine (14d) was synthesized according to General Procedure O using **68** (0.120 g, 0.447 mmol), hydrazine (0.14 mL, 4.5 mmol), acetic acid (0.03 mL, 0.5 mmol) and anhydrous ethanol (4.5 mL). The crude product was purified using flash column chromatography (95:5 to 80:20 hexanes:EtOAc) affording **14d** as a yellow oil (0.114 g, 90%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.65 (d, *J* = 7.3 Hz, 1H), 7.52 – 7.40 (m, 4H), 7.31 – 7.26 (m, 3H), 7.16 (dd, *J* = 7.3, 1.6 Hz, 1H), 5.36 (s, 2H), 4.32 (d, *J* = 12.2 Hz, 1H), 4.26 (d, *J* = 12.2 Hz, 1H), 3.36 (t, *J* = 6.6 Hz, 2H), 1.53 – 1.41 (m, 2H), 1.37 – 1.22 (m, 2H), 0.86 (t, *J* = 7.4 Hz, 3H).



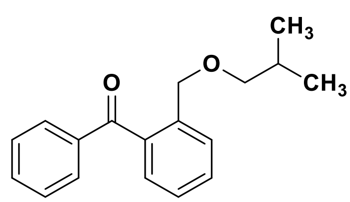
(3*R*,4*R*)-4-phenyl-3-propylisochromane (15d) was synthesized according to General Procedure R using **14d** (0.050 g, 0.18 mmol), manganese (IV) oxide (0.123 g, 0.708 mmol), Rh_2(*R*-PTAD)$_4$ (0.003 g, 0.01 mmol), and CH_2Cl_2 (12 mL). The crude product was purified using flash column chromatography (97:3 hexanes:EtOAc) affording **15d** as a clear oil (0.024 g, 54%, >95:5 dr, >99.5:0.5 er). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.27 – 7.20 (m, 2H), 7.20 – 7.13 (m, 4H), 7.13 – 7.05 (m, 2H), 7.00 (d, *J* = 7.6 Hz, 1H), 5.04 (d, *J* = 15.1 Hz, 1H), 4.91 (d, *J* = 15.1 Hz, 1H), 3.91 – 3.82 (m, 1H), 3.81 (d, *J* = 3.2 Hz, 1H), 1.58 – 1.31 (m, 2H), 1.31 – 1.20 (m, 1H), 1.20 – 1.08 (m, 1H), 0.86 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 142.3, 137.8, 134.4, 130.4, 129.9, 128.1, 126.8, 126.4, 126.4, 124.2, 78.0, 68.9, 48.1, 35.8, 19.3, 14.2; AMM (CI-TOF) *m/z* calcd for $C_{18}H_{24}NO^+$ [M+NH$_4$]$^+$ 270.1852, found 270.1866; IR (neat): ν_{max} 3024, 2958, 1493, 742 cm^{-1}; $[\alpha]_D^{21} = -21.4$ (c = 2.4, $CHCl_3$).



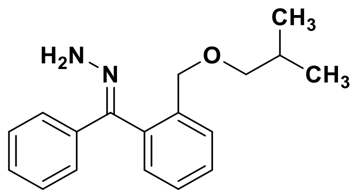
1-iodo-2-(isobutoxymethyl)benzene (69) was synthesized according to General Procedure C using 2-iodobenzyl alcohol (1.000 g, 4.273 mmol) in THF (2 mL), NaH (0.855 g, 21.4 mmol) in THF (1.5 mL), 1-bromo-2-methylpropane (2.32 mL, 21.4 mmol), and tetrabutylammonium iodide (0.221 g, 0.598 mmol). The crude product was purified using flash column chromatography (99:1 to 98:2, hexanes:CH_2Cl_2) affording **69** as a clear oil (0.493 g, 40%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.81 (d, *J* = 7.9 Hz, 1H), 7.45 (d, *J* = 7.4 Hz, 1H), 7.34 (t, *J* = 7.5 Hz, 1H), 7.01 – 6.93 (m, 1H), 4.47 (s, 2H), 3.32 (d, *J* = 6.7 Hz, 2H), 2.02 – 1.90 (m, 1H), 0.96 (d, *J* = 6.7 Hz, 6H); ^{13}C NMR (151 MHz, $(CD_3)_2CO$) δ 141.9, 139.8, 130.0, 129.5, 129.0, 97.9, 78.1, 77.1, 29.3, 19.7.; AMM (CI-TOF) *m/z* calcd for $C_{11}H_{15}IO^+$ $[M]^+$ 290.0168, found 290.0178.



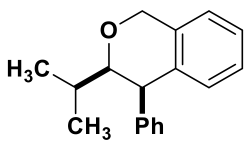
(2-(isobutoxymethyl)phenyl)(morpholino)methanone (70) was synthesized according to General Procedure D using **69** (0.290 g, 0.999 mmol), palladium (II) acetate (0.006 g, 0.02 mmol), morpholine (0.13 mL, 1.5 mmol), Xantphos (0.029 g, 0.050 mmol), potassium phosphate tribasic (0.637 g, 3.00 mmol), and toluene (2 mL). The crude product was purified using flash column chromatography (90:10 to 50:50, hexanes:EtOAc) affording **70** as a red oil (0.259 g, 93%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46 (d, *J* = 7.7 Hz, 1H), 7.37 (t, *J* = 7.5 Hz, 1H), 7.30 (t, *J* = 7.5 Hz, 1H), 7.18 (d, *J* = 7.4 Hz, 1H), 4.71 – 4.31 (m, 2H), 3.90 – 3.67 (m, 4H), 3.64 – 3.53 (m, 2H), 3.35 – 3.17 (m, 4H), 1.96 – 1.85 (m, 1H), 0.92 (d, *J* = 6.7 Hz, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.9, 136.0, 135.0, 129.3, 129.1, 127.7, 126.1, 78.3, 70.6, 67.0, 47.8, 42.1, 28.6, 19.6. ^{1}H NMR was taken at rt and shows a mixture of rotamers.



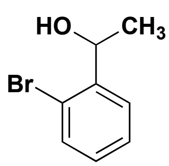
(2-(isobutoxymethyl)phenyl)(phenyl)methanone (71) was synthesized according to General Procedure E using **70** (0.250 g, 0.901 mmol), 1.9 M phenyllithium solution in dibutyl ether (0.95 mL, 1.8 mmol), and THF (9 mL). The crude product was purified using flash column chromatography (99:1 to 98:2, hexanes:EtOAc) affording **71** as a yellow oil (0.200 g, 83%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.80 (d, *J* = 7.9 Hz, 2H), 7.60 – 7.53 (m, 2H), 7.52 – 7.41 (m, 3H), 7.39 – 7.30 (m, 2H), 4.58 (s, 2H), 3.11 (d, *J* = 6.6 Hz, 2H), 1.77 – 1.65 (m, 1H), 0.77 (d, *J* = 6.7 Hz, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 198.0, 138.6, 137.8, 137.8, 133.1, 130.5, 130.3, 128.8, 128.5, 128.4, 126.9, 78.0, 70.7, 28.5, 19.4; AMM (ESI) *m/z* calcd for $C_{18}H_{21}O_2^+$ $[M+H]^+$ 269.1536, found 269.1532.



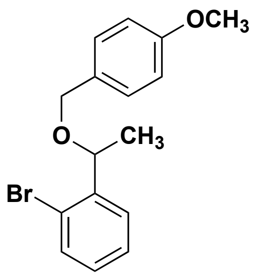
((2-(isobutoxymethyl)phenyl)(phenyl)methylene)hydrazine (14e) was synthesized according to General Procedure O using **71** (0.184 g, 0.684 mmol), hydrazine (0.22 mL, 6.8 mmol), acetic acid (0.05 mL, 0.8 mmol), and anhydrous ethanol (6.8 mL). The crude product was purified using flash column chromatography (99:1 to 90:10, hexanes:EtOAc) affording **14e** as an orange oil (0.145 g, 75%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.67 (d, *J* = 7.7 Hz, 1H), 7.53 – 7.39 (m, 4H), 7.32 – 7.26 (m, 3H), 7.16 (d, *J* = 7.3 Hz, 1H), 5.36 (s, 2H), 4.32 (d, *J* = 12.4 Hz, 1H), 4.25 (d, *J* = 12.4 Hz, 1H), 3.14 (d, *J* = 6.6 Hz, 2H), 1.85 – 1.72 (m, 1H), 0.85 (d, *J* = 6.6 Hz, 6H).



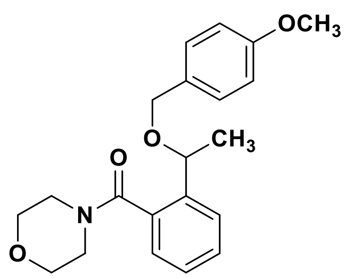
(3*R*,4*R*)-3-isopropyl-4-phenylisochromane (15e) was synthesized according to General Procedure R using **14e** (0.050 g, 0.18 mmol), manganese (IV) oxide (0.123 g, 1.42 mmol), Rh_2(*R*-PTAD)$_4$ (0.003 g, 0.002 mmol), and CH_3CN (12 mL). The crude product was purified using flash column chromatography (80:20 to 70:30, hexanes:CH_2Cl_2) affording **15e** as white crystals (0.027 g, 62%, >95:5 dr, 96:4 er). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31 (d, *J* = 7.1 Hz, 2H), 7.27 – 7.19 (m, 2H), 7.20 – 7.10 (m, 2H), 7.07 (t, *J* = 6.7 Hz, 2H), 7.02 (d, *J* = 7.5 Hz, 1H), 5.07 (d, *J* = 15.1 Hz, 1H), 4.89 (d, *J* = 15.2 Hz, 1H), 3.96 (d, *J* = 2.6 Hz, 1H), 3.37 (dd, *J* = 9.9, 2.9 Hz, 1H), 1.45 – 1.31 (m, 1H), 0.98 (d, *J* = 6.5 Hz, 3H), 0.92 (d, *J* = 6.6 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 142.5, 138.2, 134.2, 130.3, 129.9, 128.2, 126.8, 126.4, 126.3, 124.2, 84.6, 69.2, 46.0, 30.2, 19.8, 18.8; AMM (CI-TOF) *m/z* calcd for $C_{18}H_{24}NO^+$ $[M+NH_4]^+$ 270.1852, found 270.1852; m.p. 100-104 °C; $[\alpha]_D^{22} = -36.3$ (c = 0.2, $CHCl_3$).



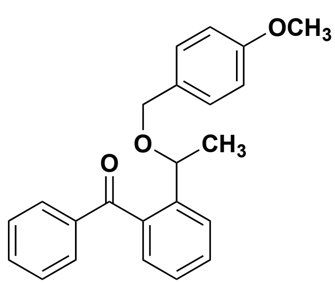
1-(2-bromophenyl)ethan-1-ol (72) was synthesized according to the General Procedure K using 1-(2-bromophenyl)ethan-1-one (0.995 g, 5.00 mmol), $NaBH_4$ (0.757 g, 20.0 mmol) in EtOH (20 mL, 0.25 M). The crude product was purified by flash column chromatography (91:9, hexanes:EtOAc) affording **72** as a clear oil (0.924 g, 92%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.60 (d, *J* = 7.7 Hz, 1H), 7.51 (d, *J* = 8.0 Hz, 1H), 7.39 – 7.30 (m, 1H), 7.17 – 7.08 (m, 1H), 5.25 (q, *J* = 6.4 Hz, 1H), 1.97 (s, 1H), 1.49 (d, *J* = 6.4 Hz, 3H). ^{1}H NMR data of the crude material was consistent with the reported literature values.[15]



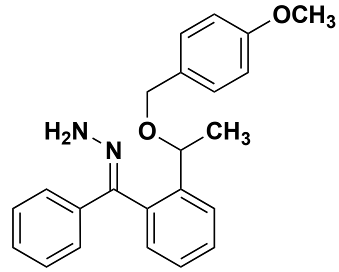
1-bromo-2-(1-((4-methoxybenzyl)oxy)ethyl)benzene (73) was synthesized according to the General Procedure L using **72** (0.608 g, 3.02 mmol), 1-(chloromethyl)-4-methoxybenzene (0.45 mL, 3.3 mmol), 60% NaH in mineral oil (0.300 g, 7.50 mmol), TBAI (0.111 g, 0.301 mmol) in THF (20 mL) and DMF (2 mL). The crude product was purified by flash column chromatography (91:9, hexanes:EtOAc) affording **73** as a clear oil (1.000 g, 100%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.59 (d, *J* = 7.7 Hz, 1H), 7.53 (d, *J* = 8.0 Hz, 1H), 7.41 – 7.32 (m, 1H), 7.25 (d, *J* = 8.3 Hz, 2H), 7.19 – 7.10 (m, 1H), 6.88 (d, *J* = 8.3 Hz, 2H), 4.90 (q, *J* = 6.4 Hz, 1H), 4.38 (d, *J* = 11.2 Hz, 1H), 4.25 (d, *J* = 11.2 Hz, 1H), 3.81 (s, 3H), 1.42 (d, *J* = 6.4 Hz, 3H). ^{1}H NMR data of the crude material was consistent with the reported literature values.[16]



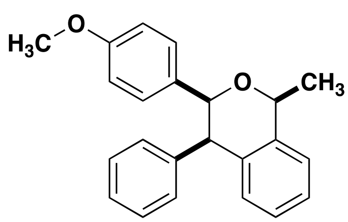
(2-(1-((4-methoxybenzyl)oxy)ethyl)phenyl)(morpholino)methanone (74) was synthesized according to General Procedure D using **73** (0.964 mg, 3.00 mmol), morpholine (0.39 mL, 4.5 mmol), $Pd(OAc)_2$ (0.017 g, 0.0757 mmol), Xantphos (0.087 g, 0.15 mmol) and potassium phosphate tribasic (1.91 g, 9.00 mmol) in toluene (6 mL, 0.5 M). The crude product was purified by flash column chromatography (67:33, hexanes:EtOAc) affording **74** as a pale yellow oil (0.769 g, 72%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.69 – 7.62 (m, 1H), 7.49 – 7.40 (m, 1H), 7.34 – 7.27 (m, 1H), 7.24 (d, *J* = 8.2 Hz, 2H), 7.15 (d, *J* = 7.6 Hz, 1H), 6.86 (d, *J* = 7.9 Hz, 2H), 4.84 – 4.50 (m, 1H), 4.38 – 4.24 (m, 2H), 3.92 – 3.40 (m, 9H), 3.37 – 3.06 (m, 2H), 1.56 – 1.34 (m, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.5, 159.2, 159.1, 141.4, 134.5, 134.2, 130.8, 130.3, 129.7, 129.4, 129.1, 127.4, 126.7, 126.4, 125.7, 113.9, 113.7, 77.3, 74.4, 73.5, 70.4, 66.9, 66.8, 55.3, 47.6, 42.0, 24.0, 23.4. AMM (ESI) *m/z* calcd for $C_{21}H_{26}NO_4^+$ $[M+H]^+$ 356.1856, found 356.1861. ^{1}H NMR was taken at rt and shows a mixture of rotamers. IR (neat): ν_{max} 1633, 1514, 1250, 1114 cm^{-1}.



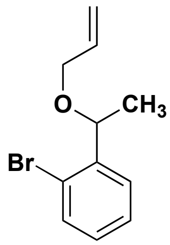
(2-(1-((4-methoxybenzyl)oxy)ethyl)phenyl)(phenyl)methanone (75) was synthesized according to General Procedure F using **74** (0.340 g, 0.957 mmol), phenyllithium (1.9 M in Bu_2O, 1.0 mL, 1.9 mmol) in THF (10 mL, 0.1 M). The crude product was purified by flash column chromatography (91:9, hexanes:EtOAc) affording **75** as a clear oil (0.294 g, 88%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.81 (d, *J* = 7.7 Hz, 2H), 7.75 (d, *J* = 7.9 Hz, 1H), 7.63 – 7.52 (m, 2H), 7.50 – 7.41 (m, 2H), 7.37 – 7.28 (m, 2H), 7.10 (d, *J* = 8.3 Hz, 2H), 6.76 (d, *J* = 8.5 Hz, 2H), 4.73 (q, *J* = 6.4 Hz, 1H), 4.28 (d, *J* = 11.1 Hz, 1H), 4.15 (d, *J* = 11.1 Hz, 1H), 3.76 (s, 3H), 1.46 (d, *J* = 6.4 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 198.0, 159.0, 143.6, 137.9, 137.7, 133.3, 130.7, 130.4, 130.3, 129.3, 128.4, 128.0, 126.6, 126.5, 113.7, 73.6, 70.4, 55.2, 24.0. AMM (ESI) *m/z* calcd for $C_{23}H_{22}NaO_3^+$ [M+Na]$^+$ 369.1467, found 369.1469.



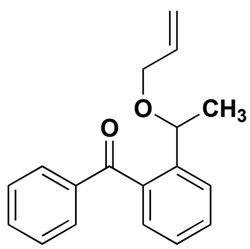
((2-(1-((4-methoxybenzyl)oxy)ethyl)phenyl)(phenyl)methylene)hydrazine (**16a**) was synthesized according to General Procedure O using **75** (0.144 g, 0.415 mmol), hydrazine (0.17 mL, 5.4 mmol), acetic acid (0.03 mL, 0.5 mmol), and EtOH (3 mL) at 160 °C for 3 h. The crude product was purified by flash column chromatography (91:9 to 84:16, hexanes:EtOAc) affording **16a** as a clear oil (0.106 g, 71%). Compound was isolated as a mixture of isomers; ^{1}HNMR spectral data for this mixture is complex and the mixture was carried on to the next step.



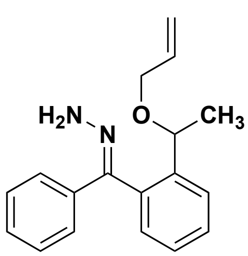
3-(4-methoxyphenyl)-1-methyl-4-phenylisochromane (17a) was synthesized according to General Procedure R using **16a** (0.031 g, 0.085 mmol), MnO_2 (0.059 g, 0.68 mmol), and $Rh_2(mes)_4$ (0.001 g, 0.0008 mmol) in CH_3CN (5 mL, 0.017 M). The crude product was purified by flash column chromatography (97:3, hexanes:EtOAc) affording **17a** as a clear oil (0.017 g, 60%, >95:5:Σ other isomers dr). ^{1}H NMR (599 MHz, $CDCl_3$) δ 7.21 – 7.17 (m, 3H), 7.12 – 7.06 (m, 1H), 7.00 – 6.94 (m, 4H), 6.81 (d, *J* = 8.3 Hz, 2H), 6.72 – 6.67 (m, 2H), 6.61 (d, *J* = 8.5 Hz, 2H), 5.09 (q, *J* = 6.5 Hz, 1H), 5.00 (d, *J* = 3.1 Hz, 1H), 4.01 (d, *J* = 3.2 Hz, 1H), 3.66 (s, 3H), 1.71 (d, *J* = 6.5 Hz, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 158.6, 140.9, 139.4, 137.4, 132.9, 130.5, 130.3, 127.4, 127.4, 126.90, 126.87, 126.1, 124.6, 113.1, 79.2, 74.4, 55.3, 51.2, 22.4. Low-resolution MS (Advion ASAP-APCI) *m/z* calcd for $C_{23}H_{23}O_2^+$ [M+H]$^+$ 331.1693, found 331.0.



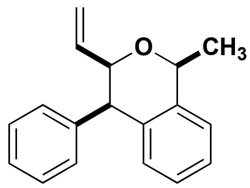
1-(1-(allyloxy)ethyl)-2-bromobenzene (76) was synthesized according to the General Procedure L using **72** (0.468 g, 2.33 mmol), 3-bromoprop-1-ene (0.3 mL, 3 mmol), 60% NaH in mineral oil (0.23 g, 5.8 mmol) in THF (10 mL) and DMF (1 mL). The crude product was purified using flash column chromatography (91:9, hexanes:EtOAc) affording **76** as a clear oil (0.481 g, 87%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.56 – 7.48 (m, 2H), 7.34 (t, *J* = 7.6 Hz, 1H), 7.16 – 7.09 (m, 1H), 5.97 – 5.87 (m, 1H), 5.27 (d, *J* = 17.2 Hz, 1H), 5.18 (d, *J* = 10.4 Hz, 1H), 4.87 (q, *J* = 6.4 Hz, 1H), 3.91 (dd, *J* = 12.7, 5.2 Hz, 1H), 3.82 (dd, *J* = 12.7, 5.9 Hz, 1H), 1.42 (d, *J* = 6.4 Hz, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 143.0, 134.8, 132.7, 128.8, 128.0, 127.3, 122.6, 117.1, 76.0, 69.9, 22.9; Low-resolution MS (Advion ASAP-APCI) *m/z* calcd for $C_{11}H_{14}BrO^+$ $[M+H]^+$ 241.0223, found 240.9.



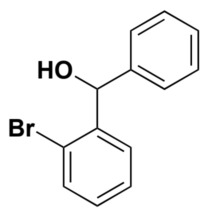
(2-(1-(allyloxy)ethyl)phenyl)(phenyl)methanone (77) was synthesized according to the General Procedure J using **76** (0.241 g, 1.00 mmol), **31** (0.165 g, 1.00 mmol), 2.13 M *n*-butyllithium in hexanes (0.52 mL, 1.1 mmol) and THF (20 mL). The crude product was purified using flash column chromatography (95:5, hexanes:EtOAc) affording **77** as a clear oil (0.109 g, 41%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.81 (d, *J* = 7.6 Hz, 2H), 7.69 (d, *J* = 7.9 Hz, 1H), 7.60 (t, *J* = 7.7 Hz, 1H), 7.53 (t, *J* = 7.6 Hz, 1H), 7.46 (t, *J* = 7.6 Hz, 2H), 7.33 (t, *J* = 7.4 Hz, 1H), 7.29 (d, *J* = 7.8 Hz, 1H), 5.81 – 5.71 (m, 1H), 5.12 (d, *J* = 17.2 Hz, 1H), 5.04 (d, *J* = 10.4 Hz, 1H), 4.68 (q, *J* = 6.4 Hz, 1H), 3.81 (dd, *J* = 12.7, 5.0 Hz, 1H), 3.73 (dd, *J* = 13.1, 6.2 Hz, 1H), 1.46 (d, *J* = 6.3 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 198.2, 143.7, 137.9, 137.8, 134.8, 133.4, 130.8, 130.4, 128.6, 128.1, 126.7, 126.6, 116.9, 73.8, 69.9, 24.0; AMM (ESI) *m/z* calcd for $C_{18}H_{19}O_2^+$ $[M+H]^+$ 267.1385, found 267.1374.



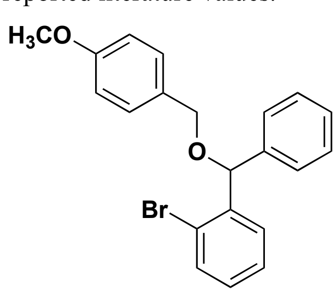
((2-(1-(allyloxy)ethyl)phenyl)(phenyl)methylene)hydrazine (**16b**) was synthesized according to General Procedure O using **77** (0.053 g, 0.20 mmol), hydrazine (0.080 mL, 2.6 mmol), acetic acid (0.014 mL, 0.24 mmol), and EtOH (2 mL) at 160 °C for 3 h. The crude product was purified by flash column chromatography (91:9 to 84:16, hexanes:EtOAc) affording **16b** as a clear oil (0.029 g, 51%). Compound was isolated as a mixture of isomers; ^{1}HNMR spectral data for this mixture is complex and the mixture was carried on to the next step.



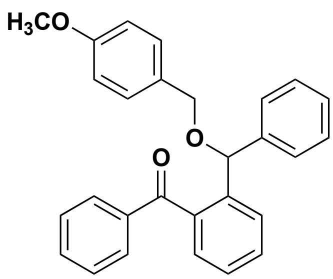
(1*S*,3*R*,4*R*)-1-methyl-4-phenyl-3-vinylisochromane (17b) was synthesized according to General Procedure R using **16b** (0.037 g, 0.13 mmol), MnO_2 (0.091 g, 1.0 mmol), and Rh_2(*R*-TCPTTL)$_4$ (0.003 g, 0.001 mmol) in CH_3CN (7.7 mL). The crude product was purified using flash column chromatography (99:1, hexanes:EtOAc) affording **17b** as a clear oil (0.025 g, 76%, >95:Σ other isomers dr). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.24 – 7.12 (m, 8H), 7.01 (d, *J* = 7.7 Hz, 1H), 5.56 – 5.45 (m, 1H), 5.25 (d, *J* = 17.3 Hz, 1H), 5.10 – 5.00 (m, 2H), 4.51 – 4.45 (m, 1H), 3.95 – 3.89 (m, 1H), 1.72 (d, *J* = 6.5 Hz, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 141.6, 139.0, 137.3, 137.1, 130.3, 130.2, 127.9, 126.9, 126.8, 126.5, 124.5, 116.4, 78.8, 73.6, 49.5, 22.2; AMM (CI) *m/z* calcd for $C_{18}H_{18}O^+$ $[M+H]^+$ 250.1358, found 250.1359; IR (neat): ν_{max} 1490, 1451, 1098, 722, 698 cm^{-1}.



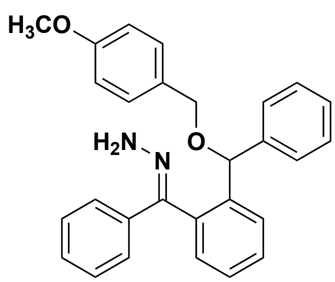
(2-bromophenyl)(phenyl)methanol (78) To a flame-dried flask was added 2-bromobenzaldehyde (0.92 g, 5.0 mmol) and Et_2O (15 mL). Phenylmagnesium bromide (1.0 M in THF) (7.5 mL, 7.5 mmol) was then added dropwise at 0 °C. The reaction was allowed to warm to rt overnight. Upon completion by TLC, the reaction was quenched with sat. aq. NH_4Cl (10 mL) and the crude mixture was extracted with Et_2O (3 X 10 mL). The combined organic layers were washed with H_2O (1 X 10 mL) and brine (1 X 10 mL). The organic layer was dried over Na_2SO_4, filtered, and concentrated *in vacuo*. The crude reaction mixture was purified using flash column chromatography (91:9, hexanes:EtOAc), affording **78** as a clear oil (1.3 g, 100%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.58 (d, *J* = 7.7 Hz, 1H), 7.54 (d, *J* = 8.0 Hz, 1H), 7.41 (d, *J* = 7.5 Hz, 2H), 7.34 (t, *J* = 7.3 Hz, 3H), 7.29 (d, *J* = 7.3 Hz, 1H), 7.15 (t, *J* = 7.6 Hz, 1H), 6.21 (d, *J* = 3.5 Hz, 1H), 2.35 (d, *J* = 3.8 Hz, 1H). ^{1}H NMR data of the crude material was consistent with the reported literature values.[17]



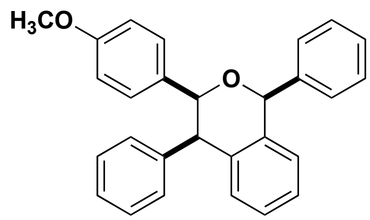
1-bromo-2-(((4-methoxybenzyl)oxy)(phenyl)methyl)benzene (79) was synthesized according to the General Procedure L using **78** (0.789 g, 3.00 mmol), 1-(chloromethyl)-4-methoxybenzene (0.45 mL, 3.3 mmol), 60% NaH in mineral oil (0.300 g, 7.50 mmol), TBAI (0.111 g, 0.301 mmol) in THF (20 mL) and DMF (2 mL). The crude product was purified using flash column chromatography (95:5, hexanes:EtOAc) affording **79** as a clear oil (1.17 g, 100%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.60 (dd, *J* = 7.9, 1.7 Hz, 1H), 7.53 (d, *J* = 8.0 Hz, 1H), 7.40 (d, *J* = 7.6 Hz, 2H), 7.36 – 7.20 (m, 6H), 7.12 (td, *J* = 7.7, 1.7 Hz, 1H), 6.87 (d, *J* = 8.6 Hz, 2H), 5.86 (s, 1H), 4.51 (d, *J* = 11.3 Hz, 1H), 4.43 (d, *J* = 11.2 Hz, 1H), 3.79 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 159.4, 141.3, 140.9, 132.9, 130.3, 129.7, 129.1, 129.0, 128.4, 127.9, 127.7, 127.6, 123.8, 113.9, 80.8, 70.8, 55.4; AMM (CI) *m/z* calcd for $C_{21}H_{19}BrO_2^+$ [M]$^+$ 382.0569, found 382.0564.



(2-(1-((4-methoxybenzyl)oxy)ethyl)phenyl)(phenyl)methanone (80) was synthesized according to the General Procedure J using **79** (0.387 g, 1.01 mmol), **31** (0.165 g, 1.00 mmol), 2.13 M *n*-butyllithium in hexane (0.52 mL, 1.1 mmol) and THF (15 mL). The crude product was purified using flash column chromatography (95:5, hexanes:EtOAc) affording **80** as a clear oil (0.23 g, 56%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.73 (d, *J* = 7.7 Hz, 2H), 7.54 (t, *J* = 7.5 Hz, 1H), 7.43 – 7.20 (m, 11H), 6.96 (d, *J* = 8.5 Hz, 2H), 6.68 (d, *J* = 8.6 Hz, 2H), 5.86 (s, 1H), 4.29 (d, *J* = 10.8 Hz, 1H), 4.23 (d, *J* = 10.8 Hz, 1H), 3.74 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 198.3, 159.1, 141.7, 141.0, 138.7, 137.8, 133.2, 130.4, 130.2, 130.2, 129.5, 128.5, 128.4, 128.1, 128.0, 128.0, 127.7, 126.8, 113.6, 79.0, 71.0, 55.4; Low-resolution MS (Advion ASAP-APCI) *m/z* calcd for $C_{28}H_{25}O_3^+$ [M+H]$^+$ 409.1798, found 409.0.

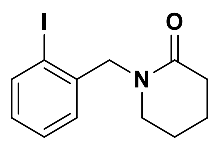


((2-(((4-methoxybenzyl)oxy)(phenyl)methyl)phenyl)(phenyl)methylene)hydrazine (**16c**) was synthesized according to General Procedure O **80** (0.123 g, 0.300 mmol), hydrazine (0.12 mL, 3.9 mmol), acetic acid (0.021 mL, 0.36 mmol), and anhydrous EtOH (3 mL) were heated in a microwave reactor at 160 °C for 3 h. After cooling down to room temperature, hydrazine (0.12 mL, 3.9 mmol) and acetic acid (0.021 mL, 0.36 mmol) were added again and the mixture was heated in a microwave reactor at 160 °C for another 3 hours. The crude product was purified by flash column chromatography (91:9 to 84:16, hexanes:EtOAc) affording **16c** as a clear oil (0.088 g, 69%). Compound was isolated as a mixture of isomers; ^{1}HNMR spectral data for this mixture is complex and the mixture was carried on to the next step.

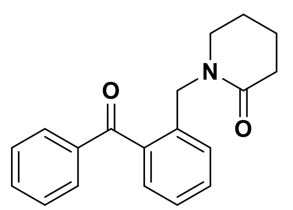


(1*S*,3*S*,4*R*)-3-(4-methoxyphenyl)-1,4-diphenylisochromane (17c) was synthesized according to General Procedure R using **16c** (0.037 g, 0.087 mmol), MnO_2 (0.060 g, 0.69 mmol), and Rh_2(*R*-TCPTTL)$_4$ (0.002 g, 0.0009 mmol) in CH_3CN (5.1 mL). The crude product was purified using flash column chromatography (99:1, hexanes:EtOAc), affording **17c** as a white solid (0.024 g, 70%, >95:Σ other isomers). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.58 (d, *J* = 7.6 Hz, 2H), 7.47 – 7.41 (m, 2H), 7.41 – 7.35 (m, 1H), 7.18 – 7.11 (m, 2H), 7.10 – 7.01 (m, 4H), 6.96 – 6.83 (m, 5H), 6.66 (d, *J* = 8.7 Hz, 2H), 5.98 (s, 1H), 5.31 (d, *J* = 3.6 Hz, 1H), 4.24 (d, *J* = 3.6 Hz, 1H), 3.72 (s, 3H); ^{13}C NMR (201 MHz, $CDCl_3$) δ 158.6, 141.8, 141.3, 137.8, 137.5, 132.7, 130.5, 130.5, 129.1, 128.7, 128.4, 127.5, 127.4, 127.2, 126.7, 126.5, 126.2, 113.1, 82.4, 80.3, 55.3, 50.8; m.p. 157-158 °C; AMM (ESI) *m/z* calcd for $C_{28}H_{25}O_2^+$ [M+Na]$^+$ 415.1674, found 415.1676; IR (neat): ν_{max} 1516, 1252, 1033, 706 cm^{-1}.

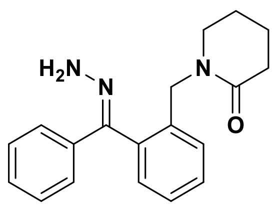
TETRAHYDROISOQUINOLINES



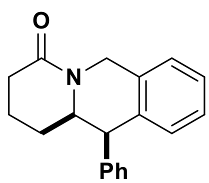
1-(2-iodobenzyl)piperidin-2-one (81) was synthesized according to General Procedure B using NaH (0.309 g, 7.56 mmol) in THF (7.5 mL), 2-piperidinone (0.500 g, 5.04 mmol) in THF (7.5 mL), and **58** (1.645 g, 5.540 mmol). After addition of the **58** the reaction was stirred overnight at rt. The crude product was purified using flash column chromatography (30:70, hexanes:EtOAc) affording **81** as a clear oil (1.585 g, 99%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.83 (d, *J* = 7.8 Hz, 1H), 7.32 (t, *J* = 7.5 Hz, 1H), 7.14 (d, *J* = 7.6 Hz, 1H), 6.96 (t, *J* = 7.6 Hz, 1H), 4.66 (s, 2H), 3.23 (t, *J* = 5.8 Hz, 2H), 2.51 (t, *J* = 6.4 Hz, 2H), 1.89 – 1.78 (m, 4H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 170.2, 139.6, 139.0, 129.1, 128.7, 128.0, 99.1, 54.9, 47.9, 32.6, 23.4, 21.5. AMM (ESI) *m/z* calcd for $C_{12}H_{15}INO^+$ [M+H]$^+$ 316.0193, found 316.0192.



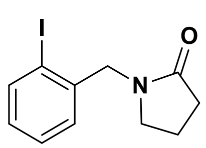
1-(2-benzoylbenzyl)piperidin-2-one (82) was synthesized according to General Procedure N using **81** (0.600 g, 1.90 mmol), benzaldehyde (1.55 mL, 15.2 mmol), palladium (II) acetate (0.021 g, 0.095 mmol), silver (I) oxide (0.533 g, 2.30 mmol), and 70% *tert*-butyl hydroperoxide in H_2O (1.29 mL, 9.5 mmol). The crude product was purified using flash column chromatography (20:80, hexanes:EtOAc) affording **82** as a pink oil (0.325 g, 58%).^{1}H NMR (600 MHz, $CDCl_3$) δ 7.84 – 7.78 (m, 2H), 7.63 – 7.57 (m, 1H), 7.51 – 7.44 (m, 3H), 7.41 (d, *J* = 7.8 Hz, 1H), 7.36 – 7.31 (m, 2H), 4.66 (s, 2H), 3.17 (t, J = 5.7 Hz, 2H), 2.35 (t, J = 6.3 Hz, 2H), 1.76 – 1.66 (m, 4H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 198.1, 170.3, 138.5, 137.6, 136.8, 133.6, 130.8, 130.3, 128.7, 128.7, 128.7, 126.7, 48.0, 47.7, 32.5, 23.2, 21.4; AMM (ESI) *m/z* calcd for $C_{19}H_{20}NO_2^+$ $[M+H]^+$ 294.1489, found 294.1485.



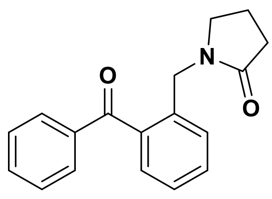
1-(2-(hydrazineylidene(phenyl)methyl)benzyl)piperidin-2-one (22a) was synthesized according to General Procedure P using **82** (0.168 g, 0.573 mmol), hydrazine (0.18 mL, 5.7 mmol), acetic acid (0.02 mL, 0.3 mmol), and ethanol (5.7 mL). The reaction was heated at 80 °C for 3 days. The crude product was purified using flash column chromatography (100% EtOAc) affording **22a** as a white foam (0.107 g, 61%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.52 – 7.32 (m, 5H), 7.33 – 7.21 (m, 3H), 7.15 (d, J = 7.1 Hz, 1H), 5.44 (s, 2H), 4.48 (d, J = 15.4 Hz, 1H), 4.32 (d, J = 15.3 Hz, 1H), 3.24 – 2.94 (m, 2H), 2.46 – 2.26 (m, 2H), 1.83 – 1.57 (m, 4H).



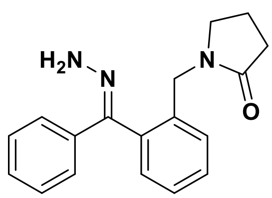
(11*R*,11a*R*)-11-phenyl-1,2,3,6,11,11a-hexahydro-4H-pyrido[1,2-b]isoquinolin-4-one (23a) was synthesized according to General Procedure S using **22a** (0.072 g, 0.23 mmol), MnO_2 (0.162 g, 1.86 mmol), Rh_2(*R*-PTAD)$_4$ (0.003 g, 0.002 mmol), and CH_2Cl_2 (16 mL). The oxidation was conducted in CH_2Cl_2 (2 mL) for 5 h at which time the MnO_2 was filtered off and the resulting diazo was diluted with CH_2Cl_2 to 16 mL. The catalyst was added to the solution at -20 °C and the reaction was left at -20 °C until the fuchsia color of the diazo was gone and the solution was a pale green (30 min). The crude product was purified using flash column chromatography (97:3, CH_2Cl_2:MeOH) affording **23a** as white crystals (0.035 g, 54%, >95:5 dr, 99:1 er). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.29 – 7.19 (m, 5H), 7.12 (t, J = 7.4 Hz, 1H), 7.07 – 6.97 (m, 3H), 5.56 (d, J = 18.2 Hz, 1H), 4.41 (d, J = 18.2 Hz, 1H), 4.03 – 3.95 (m, 2H), 2.31 (dt, J = 17.2, 5.4 Hz, 1H), 2.06 – 1.95 (m, 2H), 1.59 – 1.45 (m, 2H), 1.28 – 1.18 (m, 1H); ^{13}C NMR (151 MHz, $(CD_3)_2CO$) δ 170.3, 142.8, 138.8, 133.2, 130.4, 130.2, 129.0, 127.6, 127.5, 127.4, 127.2, 56.7, 51.6, 45.7, 33.2, 27.5, 19.1.; AMM (ESI) *m/z* calcd for $C_{19}H_{20}NO^+$ $[M+H]^+$ 278.1539, found 278.1544; m.p. 125.129 °C ; $[\alpha]_D^{20} = -193.2$ (c = 0.25, $CHCl_3$).



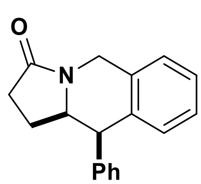
1-(2-iodobenzyl)pyrrolidin-2-one (83) was synthesized according to General Procedure B, using NaH (0.703 g, 17.2 mmol) THF (5 mL), and 2-pyrrolidinone (1.15 mL, 15.2 mmol) in THF (23 mL) and the reaction was allowed to warm to rt over 2 h. **58** (1.000 g, 10.08 mmol) was added to the mixture and the reaction was stirred at rt overnight. Upon completion by TLC, the reaction was quenched with water (30 mL) and extracted with EtOAc (3 X 40 mL). The combined organic layers were washed with brine (1 X 30 mL), dried over Na_2SO_4, filtered, and concentrated *in vacuo*. The crude product was purified using flash column chromatography (30:70, hexanes:EtOAc) affording **83** as a yellow oil (0.983 g, 33%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.84 (d, *J* = 7.9 Hz, 1H), 7.33 (t, *J* = 7.5 Hz, 1H), 7.21 (d, *J* = 7.6 Hz, 1H), 6.98 (t, *J* = 7.6 Hz, 1H), 4.54 (s, 2H), 3.31 (t, *J* = 7.0 Hz, 2H), 2.46 (t, *J* = 8.1 Hz, 2H), 2.08 – 2.00 (m, 2H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 175.3, 139.7, 138.7, 129.4, 128.9, 128.8, 99.0, 51.2, 47.0, 30.8, 18.0; AMM (ESI) *m/z* calcd for $C_{11}H_{13}INO^+$ $[M+H]^+$ 302.0036, found 302.0030.



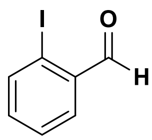
1-(2-benzoylbenzyl)pyrrolidin-2-one (84) was synthesized according to General Procedure N using **83** (0.480 g, 1.59 mmol), benzaldehyde (1.29 mL, 12.7 mmol), palladium (II) acetate (0.018 g, 0.080 mmol), silver (I) oxide (0.443 g, 1.91 mmol), and 70% *tert*-butyl hydroperoxide in H_2O (1.1 mL, 7.9 mmol). The crude product was purified using flash column chromatography (20:80, hexanes:EtOAc) affording **84** as a yellow oil (0.287 g, 65%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.83 – 7.77 (m, 2H), 7.63 – 7.57 (m, 1H), 7.51 – 7.44 (m, 3H), 7.42 (d, *J* = 7.7 Hz, 1H), 7.37 – 7.33 (m, 2H), 4.52 (s, 2H), 3.23 (t, *J* = 7.1 Hz, 2H), 2.31 (t, *J* = 8.1 Hz, 2H), 1.92 – 1.81 (m, 2H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 197.6, 175.2, 138.2, 137.3, 136.0, 133.5, 130.7, 130.1, 129.3, 128.7, 128.6, 126.9, 47.0, 43.7, 30.7, 17.7; AMM (ESI) *m/z* calcd for $C_{18}H_{18}NO_2^+$ $[M+H]^+$ 280.1332, found 280.1332.



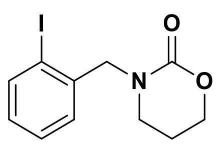
1-(2-(hydrazineylidene(phenyl)methyl)benzyl)pyrrolidin-2-one (22b) was synthesized according to General Procedure P using **84** (0.464 g, 1.66 mmol), hydrazine (0.52 mL, 17 mmol), acetic acid (0.05 mL, 0.8 mmol) and ethanol (16 mL). The reaction was heated at 80 °C for 4 d. The crude product was purified using flash column chromatography (100:0 to 0:100, hexanes:EtOAc) affording **22b** as a white foam (0.248 g, 51%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.49 – 7.38 (m, 5H), 7.31 – 7.23 (m, 3H), 7.19 – 7.14 (m, 1H), 5.44 (s, 2H), 4.32 – 4.23 (m, 2H), 3.15 (td, *J* = 9.1, 5.8 Hz, 1H), 3.02 (td, *J* = 9.1, 5.6 Hz, 1H), 2.37 – 2.28 (m, 1H), 2.28 – 2.20 (m, 1H), 1.91 – 1.73 (m, 2H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 175.2, 147.3, 137.8, 135.8, 132.1, 129.6, 129.0, 128.8, 128.5, 128.3, 128.2, 125.7, 47.1, 43.9, 30.6, 17.6; AMM (ESI) *m/z* calcd for $C_{18}H_{20}N_3O^+$ $[M+H]^+$ 294.1601, found 294.1601.



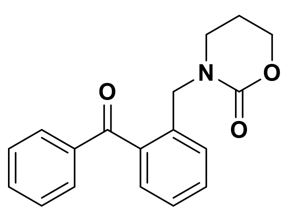
(10*R*,10a*R*)-10-phenyl-1,5,10,10a-tetrahydropyrrolo[1,2-b]isoquinolin-3(2H)-one (23b) was synthesized according to General Procedure S using **22b** (0.024 g, 0.082 mmol), MnO_2 (0.059 g, 0.68 mmol), $Rh_2(R\text{-PTAD})_4$ (0.001 g, 0.0008 mmol), and CH_2Cl_2 (6 mL). The oxidation was conducted in CH_2Cl_2 (1 mL) for 2 h at which time the MnO_2 was filtered off and the resulting diazo was diluted with CH_2Cl_2 to 6 mL. The catalyst was added to the solution at -20 °C and the reaction was left at -20 °C until the fuchsia color of the diazo was gone and the solution was a pale green (1 h). The crude product was purified using flash column chromatography (25:75, hexanes:EtOAc) affording **23b** as a clear oil (0.018 g, 81%, >95:5 dr, 94:6 er). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.30 – 7.19 (m, 5H), 7.19 – 7.13 (m, 1H), 7.08 (d, J = 7.7 Hz, 1H), 6.97 – 6.92 (m, 2H), 5.24 (d, J = 17.7 Hz, 1H), 4.39 (d, J = 17.7 Hz, 1H), 4.20 – 4.14 (m, 1H), 4.07 (d, J = 3.9 Hz, 1H), 2.20 – 2.12 (m, 1H), 2.12 – 2.02 (m, 1H), 1.82 – 1.75 (m, 1H), 1.35 – 1.27 (m, 1H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 174.8, 139.8, 136.7, 131.4, 130.5, 129.3, 128.5, 127.2, 127.1, 127.0, 126.6, 57.1, 49.5, 42.5, 29.5, 21.1; AMM (ESI) *m/z* calcd for $C_{18}H_{18}NO^+$ $[M+H]^+$ 264.1383, found 264.1380; IR (neat): ν_{max} 3060, 2925, 1764, 1678, 1284 cm^{-1}; $[\alpha]_D^{22} = -11.3$ (c = 0.14, $CHCl_3$).



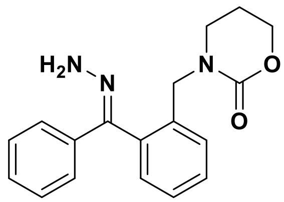
2-iodobenzaldehyde (85). Following a modified literature procedure,[19] 2-iodobenzyl alcohol (2.000 g, 8.546 mmol) and CH_2Cl_2 (24 mL) were added to a flame-dried round bottom flask under argon. Manganese (IV) oxide (11.154 g, 128.30 mmol) was added to the solution and the mixture was refluxed for 16 h. The mixture was cooled to rt, filtered through Celite, and the filtrate was concentrated *in vacuo* affording **85** as a white solid (1.960 g, 98%) with no purification needed. ^{1}H NMR (600 MHz, $CDCl_3$) δ 10.03 (s, 1H), 7.91 (d, J = 7.8 Hz, 1H), 7.84 (d, J = 7.4 Hz, 1H), 7.44 (t, J = 7.4 Hz, 1H), 7.26 (t, J = 7.5 Hz, 1H). ^{1}H NMR data was consistent with literature values.[19]



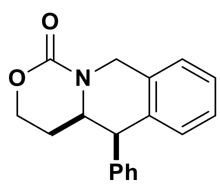
3-(2-iodobenzyl)-1,3-oxazinan-2-one (86) was synthesized according to General Procedure B using NaH (0.606 g, 14.8 mmol) in THF (4 mL), 1,3-oxazinan-2-one (1.000 g, 9.891 mmol) in THF (15 mL), and **58** (3.231 g, 10.88 mmol). The crude product was purified using flash column chromatography (100:0 to 30:70, hexanes:EtOAc) affording **86** as white crystals (1.935 g, 62%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.82 (dd, J = 7.9, 1.3 Hz, 1H), 7.34 (td, J = 7.5, 1.3 Hz, 1H), 7.27 – 7.21 (m, 1H), 6.98 (td, J = 7.6, 1.8 Hz, 1H), 4.59 (s, 2H), 4.30 (t, J = 5.3 Hz, 2H), 3.24 (t, J = 6.2 Hz, 2H), 2.09 – 2.01 (m, 2H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 153.7, 139.4, 138.1, 129.1, 128.5, 127.8, 98.6, 66.5, 56.9, 44.8, 22.0; AMM (ESI) *m/z* calcd for $C_{11}H_{13}INO_2^+$ $[M+H]^+$ 317.9985, found 317.9985; m.p. 103-105 °C.



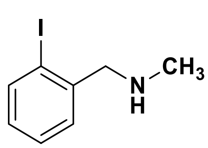
3-(2-benzoylbenzyl)-1,3-oxazinan-2-one (87) was synthesized according to General Procedure N using **86** (1.848 g, 5.827 mmol), benzaldehyde (4.7 mL, 46.6 mmol), palladium (II) acetate (0.065 g, 0.29 mmol), silver (I) oxide (1.620 g, 6.992 mmol), and *tert*-butyl hydroperoxide (3.9 mL, 29.1 mmol). The crude product was purified using flash column chromatography (90:10, CH_2Cl_2:CH_3CN) affording **87** as a pale, yellow oil (0.671 g, 39%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.80 (d, *J* = 7.8 Hz, 2H), 7.60 (t, *J* = 7.4 Hz, 1H), 7.55 – 7.42 (m, 4H), 7.40 – 7.32 (m, 2H), 4.63 (s, 2H), 4.12 (t, *J* = 5.3 Hz, 2H), 3.21 (t, *J* = 6.3 Hz, 2H), 1.96 – 1.90 (m, 2H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 197.9, 154.0, 138.2, 137.3, 136.2, 133.5, 130.8, 130.1, 128.7, 128.7, 128.6, 126.8, 66.4, 49.9, 45.2, 22.2; AMM (ESI) *m/z* calcd for $C_{18}H_{18}NO_3^+$ [M+H]$^+$ 296.1281, found 296.1286.



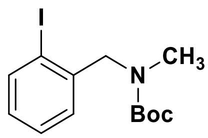
3-(2-(hydrazineylidene(phenyl)methyl)benzyl)-1,3-oxazinan-2-one (22c) was synthesized according to General Procedure P using **87** (0.567 g, 1.92 mmol), hydrazine (0.60 mL, 19 mmol), acetic acid (0.05 mL, 0.9 mmol) and ethanol (19 mL). The reaction was heated at 80 °C for 5 d. The crude product was purified using flash column chromatography (20:80, hexanes:EtOAc) affording **22c** as a pale yellow foam (0.322 g, 54%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.51 (d, *J* = 7.6 Hz, 1H), 7.49 – 7.39 (m, 4H), 7.34 – 7.25 (m, 3H), 7.15 (d, *J* = 7.4 Hz, 1H), 5.45 (s, 2H), 4.40 (d, *J* = 15.7 Hz, 1H), 4.30 (d, *J* = 15.7 Hz, 1H), 4.16 – 4.03 (m, 2H), 3.13 (dt, *J* = 12.1, 6.2 Hz, 1H), 3.04 (dt, *J* = 12.0, 6.3 Hz, 1H), 1.93 – 1.81 (m, 2H).



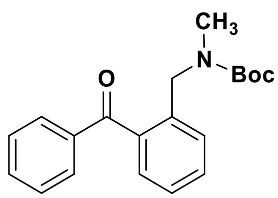
(4a*R*,5*R*)-5-phenyl-4,4a,5,10-tetrahydro-1*H*,3*H*-[1,3]oxazino[3,4-*b*]isoquinolin-1-one (23c) was synthesized according to General Procedure S using **22c** (0.100 g, 0.323 mmol), MnO_2 (0.224 g, 2.58 mmol), Rh_2(*R*-PTAD)$_4$ (0.005 g, 0.003 mmol), and CH_2Cl_2 (22 mL). The oxidation was conducted in CH_2Cl_2 (2 mL) for 4 h at which time the MnO_2 was filtered off and the resulting diazo was diluted with CH_2Cl_2 to 22 mL. The catalyst was added to the solution at -20 °C and the reaction was left at -20 °C until the fuchsia color of the diazo was gone and the solution was a pale green (1-3 h). The crude product was purified using flash column chromatography (95:5, CH_2Cl_2:CH_3CN) affording **23c** as white foam (0.042 g, 52%, >95:5 dr, 99:1 er). **23c** was crystallized out of hexanes:*i*-PrOH (90:10) with a small amount of CH_2Cl_2 to dissolve fully followed by slow evaporation over 1 h. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.32 – 7.20 (m, 5H), 7.19 – 7.10 (m, 1H), 7.07 – 7.00 (m, 3H), 5.33 (d, *J* = 17.5 Hz, 1H), 4.54 (d, *J* = 17.5 Hz, 1H), 4.06 – 3.96 (m, 2H), 3.85 (dt, *J* = 10.4, 4.8 Hz, 1H), 3.09 (td, *J* = 10.2, 3.1 Hz, 1H), 2.26 – 2.14 (m, 1H), 1.95 – 1.84 (m, 1H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 154.4, 140.1, 136.6, 131.8, 129.7, 129.6, 128.7, 127.5, 127.3, 127.1, 126.5, 63.2, 53.3, 50.7, 47.5, 25.5; AMM (ESI) *m/z* calcd for $C_{18}H_{18}NO_2^+$ [M+H]$^+$ 280.1332, found 280.1328; $[\alpha]_D^{22} = -202.3$ (c = 0.66, $CHCl_3$); m.p. 182-183 °C.



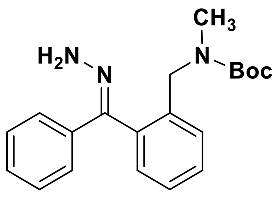
1-(2-iodophenyl)-N-methylmethanamine (88). To a flame-dried round bottom flask was added **85** (0.842 g, 3.63 mmol) in anhydrous ethanol (12 mL) and a solution of 33% methylamine in ethanol (5.8 mL, 46 mmol) followed by acetic acid (0.02 mL, 0.4 mmol). The reaction was stirred overnight at rt after which time the reaction was cooled to 0 °C and $NaBH_4$ (0.272 g, 7.19 mmol) was added. The reaction was stirred for 2 h and then quenched with $NaHCO_3$ (5 mL) and the mixture was extracted with EtOAc (3 X 30 mL) and the combined organic extracts were dried over Na_2SO_4, filtered, and concentrated *in vacuo* affording **88** as a clear oil (0.890 g, 99%) with no purification needed. ^1H NMR (600 MHz, $CDCl_3$) δ 7.83 (d, J = 7.9 Hz, 1H), 7.36 (d, J = 7.3 Hz, 1H), 7.32 (t, J = 7.4 Hz, 1H), 6.96 (t, J = 7.5 Hz, 1H), 3.77 (s, 2H), 2.46 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 142.1, 139.6, 129.8, 128.9, 128.4, 99.8, 60.3, 36.0; AMM (ESI) m/z calcd for $C_8H_{11}IN^+$ $[M+H]^+$ 247.9931, found 247.9926.



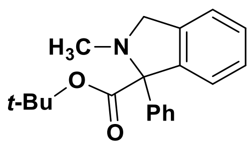
***tert*-butyl (2-iodobenzyl)(methyl)carbamate (89)**. Following a modified literature procedure,[20] di-*tert*-butyl decarbonate (1.69 mL, 7.34 mmol) was added to a solution of **88** (0.906 g, 3.67 mmol) in THF (24 mL) and the reaction was heated to 40 °C overnight. The reaction was cooled to rt and imidazole (0.500 g, 7.34 mmol) was added and the reaction was stirred for 30 min after which time the reaction was concentrated *in vacuo*. The residue was dissolved in $CHCl_3$ (50 mL) and washed with 1 M HCl (2 X 25 mL), water (2 X 25 mL), and brine (1 X 25 mL). The organic layer was dried over Na_2SO_4, filtered, and concentrated *in vacuo*. The crude product was purified using flash column chromatography (100:0 to 85:15, hexanes:EtOAc) affording **89** as a clear oil (1.092 g, 84%). ^1H NMR (600 MHz, $CDCl_3$) δ 7.83 (d, J = 7.8 Hz, 1H), 7.34 (t, J = 7.5 Hz, 1H), 7.15 – 7.08 (m, 1H), 7.01 – 6.92 (m, 1H), 4.48 (s, 2H), 2.85 (s, 3H), 1.51 (s, 9H). ^1H NMR data was consistent with literature values.[21] ^1H NMR was taken at rt and shows a mixture of rotamers.



***tert*-butyl (2-benzoylbenzyl)(methyl)carbamate (90)** was synthesized according to General Procedure N using **89** (0.500 g, 1.44 mmol), benzaldehyde (1.17 mL, 11.5 mmol), palladium (II) acetate (0.016 g, 0.072 mmol), silver (I) oxide (0.401 g, 1.73 mmol), and 70% *tert*-butyl hydroperoxide in H_2O (0.97 mL, 7.2 mmol). The crude product was purified using flash column chromatography (90:10, hexanes:EtOAc) affording **90** as a clear oil (0.282 g, 60%). ^1H NMR (600 MHz, $CDCl_3$) δ 7.80 (d, J = 7.6 Hz, 2H), 7.63 – 7.56 (m, 1H), 7.52 – 7.43 (m, 3H), 7.43 – 7.30 (m, 3H), 4.53 (s, 2H), 2.84 – 2.70 (m, 3H), 1.46 – 1.36 (m, 9H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 198.0, 156.0, 138.1, 137.6, 133.5, 130.8, 130.3, 129.1, 128.8, 128.6, 128.3, 127.5, 126.4, 79.9, 79.7, 50.4, 49.5, 34.5, 28.5, 28.4; AMM (ESI-TOF) m/z calcd for $C_{20}H_{23}NNaO_3^+$ $[M+Na]^+$ 348.1570, found 348.1571. NMRs were taken at rt and show a mixture of rotamers.



***tert*-butyl-(2-(hydrazineylidene(phenyl)methyl)benzyl)(methyl)carbamate (24)** was synthesized according to General Procedure P using **90** (0.282 g, 0.866 mmol), hydrazine (0.27 mL, 8.7 mmol), acetic acid (0.02 mL, 0.4 mmol), and ethanol (9 mL). The reaction was heated for 3 d with incomplete conversion. The crude product was purified using flash column chromatography (80:20, hexanes:EtOAc) affording **24** as a yellow oil (0.171 g, 58%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.51 – 7.32 (m, 5H), 7.31 – 7.23 (m, 3H), 7.15 (d, J = 7.4 Hz, 1H), 5.40 (s, 2H), 4.41 – 4.24 (m, 1H), 4.22 – 4.16 (m, 1H), 2.75 (s, 3H), 1.39 (s, 9H). ^{1}H NMR was taken at rt and shows a mixture of rotamers.



***tert*-butyl 1-methyl-1-phenylisoindoline-2-carboxylate (26)** was synthesized according to a modified version of General Procedure S using **24** (0.322 g, 0.949 mmol), MnO_2 (0.659 g, 7.59 mmol), $Rh_2(TFA)_4$ (0.006 g, 0.01 mmol), and CH_3CN (63 mL). The hydrazone was oxidized in CH_3CN (4 mL) and upon completion by TLC the MnO_2 was filtered off and the diazo was diluted to 63 mL with CH_3CN. To the solution was added $Rh_2(TFA)_4$ and the magenta solution was heated to reflux for 19 h at which point the solution was a dark teal color. The crude product was purified using flash column chromatography (93:7, hexanes:EtOAc) affording **26** as a clear oil (0.150 g, 50%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.34 – 7.24 (m, 6H), 7.24 – 7.18 (m, 1H), 7.12 (d, J = 7.2 Hz, 2H), 4.27 (d, J = 12.5 Hz, 1H), 4.01 (d, J = 12.5 Hz, 1H), 2.45 (s, 3H), 1.45 (s, 9H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 171.3, 143.4, 140.7, 139.7, 128.2, 128.0, 127.8, 127.4, 127.0, 124.6, 122.1, 81.9, 79.9, 58.6, 35.6, 28.2; ^{1}H NMR (600 MHz, C_6D_6) δ 7.53 – 7.49 (m, 2H), 7.38 – 7.34 (m, 1H), 7.20 – 7.17 (m, 2H), 7.12 – 6.96 (m, 4H), 4.21 (d, J = 12.2 Hz, 1H), 4.03 (d, J = 12.2 Hz, 1H), 2.59 (s, 3H), 1.26 (s, 9H); ^{13}C NMR (151 MHz, C_6D_6) δ 171.0, 144.9, 141.3, 141.2, 128.9, 128.2, 127.7, 127.5, 127.1, 124.9, 122.2, 81.0, 80.4, 58.7, 35.1, 28.1; AMM (ESI) *m/z* calcd for $C_{20}H_{24}NO_2^+$ $[M+H]^+$ 310.1802, found 310.1799; IR (neat): ν_{max} 2974, 2791, 1720, 1599, 1366 cm^{-1}. All spectroscopic data was consistent with literature data.[22]

COMPUTATIONAL DETAILS

General comments. Gas phase DFT calculations provide evidence that nucleophilic attack of the Rh-carbene by the carbamate N is energetically feasible at experimental conditions (a ~13.4 kcal mol^{-1} free energy barrier), which subsequently undergoes a facile Stevens [1,2]-rearrangement[23,24] that is 2.8 kcal mol^{-1} higher in free energy relative to a free ylide intermediate, i.e. with the Rh catalyst unattached to the carbene carbon. For simplicity, the dirhodium tetraacetate complex was used to approximate the Rh catalyst. While computing the Stevens rearrangement transition state structure (TSS) with the catalyst bound to the substrate, we could only identify an optimized TSS in which the catalyst is not explicitly *bound* to the carbene carbon, that is, a free ylide structure. After the free ylide is formed, the low-energy, concerted Stevens rearrangement step leads to the final product. We also computed the free ylide without a metal involved to compare energy barriers; the barrier for this process is 2.6 kcal mol^{-1} at the uB3LYP/LANL2DZ[6-31G(d)] level of theory. This barrier is 0.2 kcal/mol lower in free energy than that with the catalyst present, which provides further evidence that the metal catalyst may not be essential in this step.

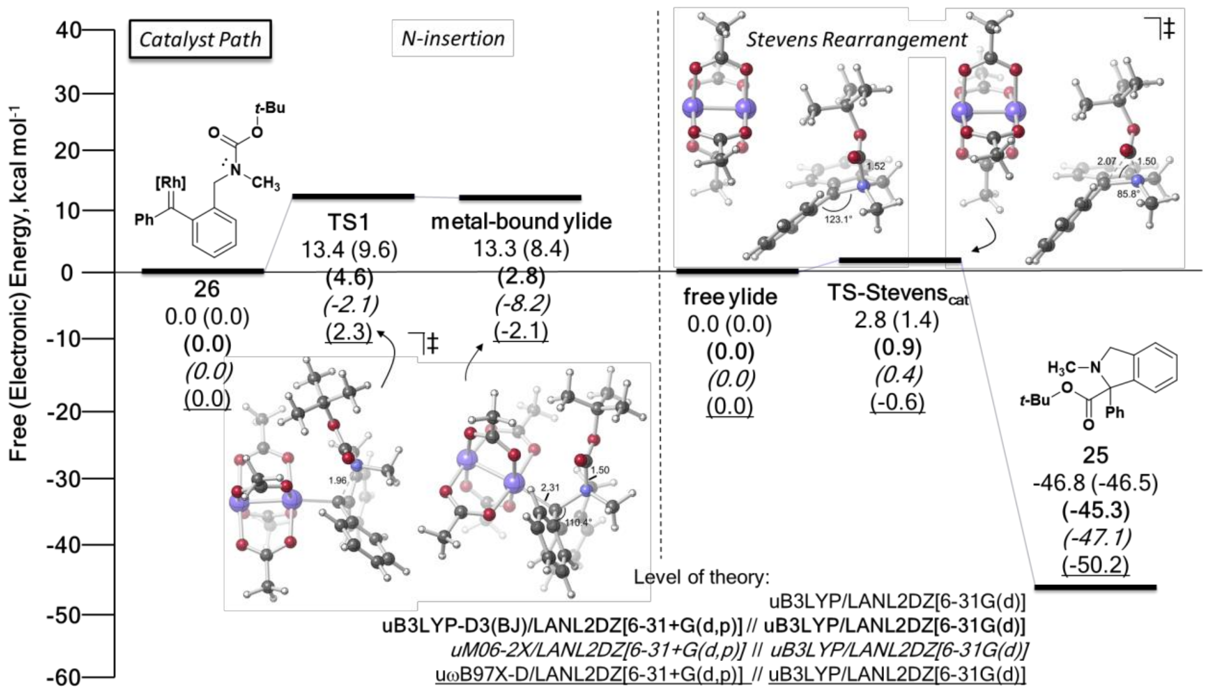


Figure S1. Energetic profile for *N*-attack on the Rh carbene and Stevens rearrangement with Rh catalyst present.

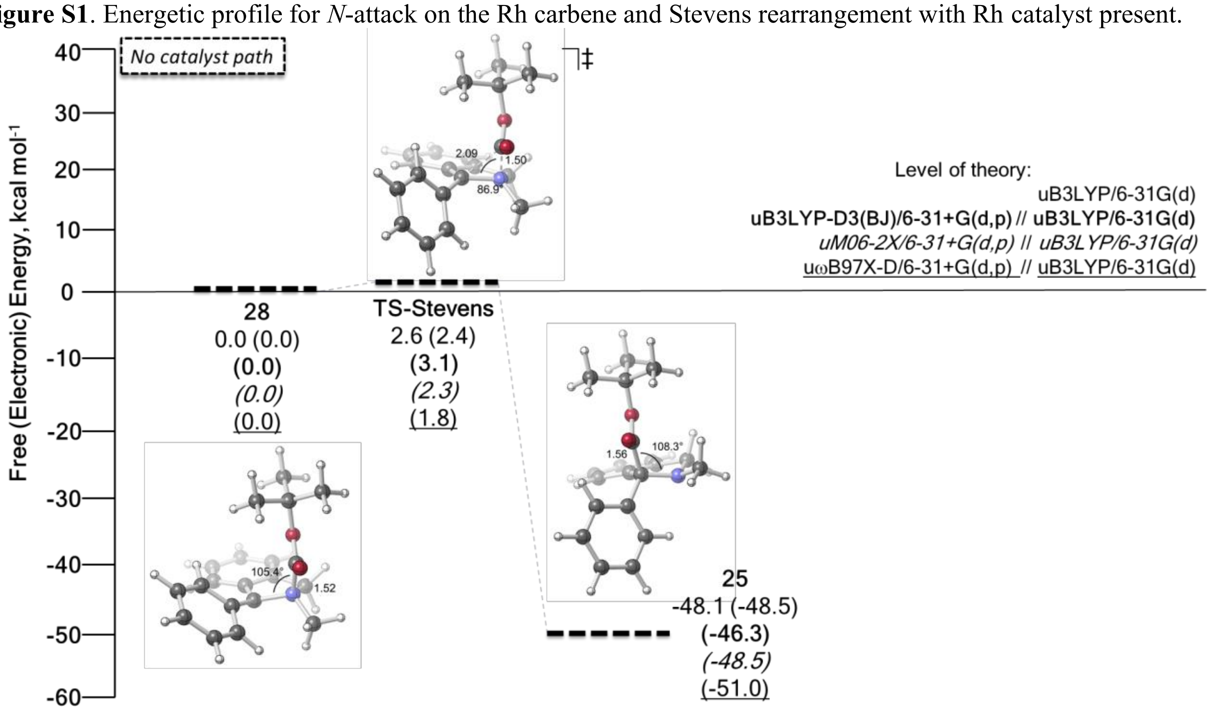


Figure S2. Energetic profile Stevens rearrangement step *without* Rh catalyst.

Recent studies[25,26] highlight that whether Rh catalysts are bound at each step of Rh(II)-catalyzed reactions remains unanswered. In previous theoretical work,[27] we suggested that the Rh catalyst is not involved at every mechanistic step of a Rh(II)-promoted indole formation from vinyl/azidoarenes.

The DFT results in the current study suggest that the concerted Stevens rearrangement energetically prefers a free ylide pathway. Although we cannot definitively say whether the Rh catalyst is present or absent at each step, our results are consistent with the catalyst being involved in the *N*-attack step, but subsequently falling off before the Stevens rearrangement step. Our future computational work will investigate other experimentally relevant examples in which metal catalysts may or may not be involved at every mechanistic step.

Nitrogen attack of the carbamate on to the Rh-carbene is consistent with recent literature demonstrating that the N-H group is more nucleophilic than the carbonyl oxygen.[28] Attack of the carbamate oxygen was also computed, but no productive pathways to the product could be identified.

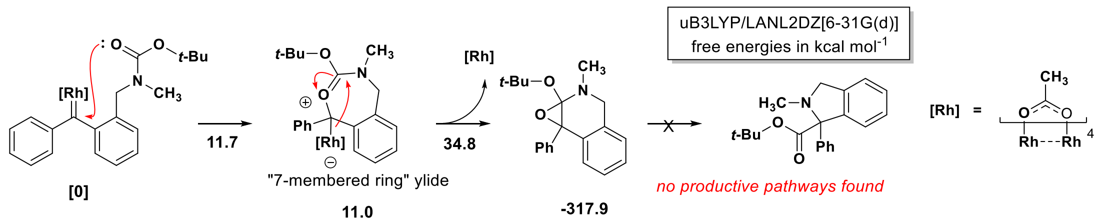


Figure S3. Computed Free energy pathway for O attack. Formation of epoxide is energetically viable given the experimental conditions, but significantly exergonic. No further viable pathways to the product could be found.

Computational Methods. All Density Functional Theory (DFT) calculations were carried out using the *Gaussian 09* suite of programs.[29] DFT methods were chosen over single-determinant *ab initio* or multiconfigurational methods for practical reasons and because DFT methods have been in reported in the literature as being sufficient for modeling Stevens rearrangements—see below for literature precedent. Additionally, as discussed in more detail below, the need for modeling radical character of the Stevens rearrangement transition state structure (TSS) was ruled out based on wavefunction stability tests on the restricted and unrestricted DFT-optimized TSS's, which support evidence of a stable, non-radical Stevens TSS. Structures are provided with this SI document in the form of a mol2 file. TSS's and minima were optimized using the unrestricted B3LYP (uB3LYP) functional[30] with the 6-31G(d) basis set[31] for C, H, O, and N and the LANL2DZ[32] basis set and effective core potential (ECP) for Rh (i.e. uB3LYP/LANL2DZ[6-31G(d)]). These values are reported in the main manuscript. The "guess=(mix,always)" keyword in *Gaussian 09* was used to allow for singlet diradical species. A wavefunction stability test using the "stable" keyword in *Gaussion 09* on the Stevens rearrangement TSS resulted in a stable wavefunction with an <S^2> eigenvalue = 0.00.

UB3LYP – optimizes to a "stable" singlet wavefunction

```
SCF Done:  E(UB3LYP) =  -981.108141993     A.U. after    1 cycles
           NFock=  1  Conv=0.50D-08     -V/T= 2.0096
<Sx>= 0.0000 <Sy>= 0.0000 <Sz>= 0.0000 <S**2>= 0.0000 S= 0.0000
<L.S>= 0.000000000000E+00
Annihilation of the first spin contaminant:
S**2 before annihilation     0.0000,   after    -0.0000
```

```
SavETr:  write IOETrn=   770 NScale= 10 NData=  16 NLR=1 NState=    1 LETran=
The wavefunction is stable under the perturbations considered.
The wavefunction is already stable.
```

RB3LYP – singlet wavefunction is "stable"

```
SavETr:  write IOETrn=   770 NScale= 10 NData=  16 NLR=1 NState=    1 LETran=
The wavefunction is stable under the perturbations considered.
The wavefunction is already stable.
```

Single-point calculations were carried out at the uB3LYP/6-31G(d) geometries using uB3LYP with Grimme's D3 version of dispersion correction[33] with Becke-Johnson (BJ) damping[34] (uB3LYP-D3(BJ)), uM06-2X,[35] and uωB97X-D[36] functionals with the 6-31+G(d,p) basis set for verification that computed energies were reasonable. These results yielded energies that exhibit the typical variation one might see in a DFT study, thus validating our uB3LYP geometries (see Tables below). Transition state structures and minima were verified as such by frequency calculations: identification of exactly one imaginary frequency and no imaginary frequencies was diagnostic of TSSs and minima, respectively. All uB3LYP coordinates are contained in the concatenated ".mol2" file that comes with the SI.

Supplemental References of DFT Methods on Stevens Rearrangements.

(1) Computational Study of Stevens and Sommelet-Hauser Rearrangements: G. Ghigo and coworkers, *J. Org. Chem.*, **2010**, *75*, 3608.

(2) Direct dynamics study of [2,3]- and [1,2]-sigmatropic rearrangements: D. A. Singleton and coworkers, *J. Am. Chem. Soc.,* **2014**, *136*, 3740. (notably, the authors thoroughly scrutinized DFT methods against more accurate UBD(T) methods. Although DFT methods do not satisfactorily match the UBD(T) results, they provided a less computationally intensive method to obtain reasonable results.)

(3) A [2,3]-Stevens Rearrangement: S. C. Schmid et al., *ACS Catal.*, **2018**, *8*, 7907-7914.

(4) A similar, yet different, DFT study with a Rh(II)-catalyzed [1,5]-sigmatropic rearrangement: J. G. Harrison et al., *J. Am. Chem. Soc.*, **2016**, *138*, 487-490.

Comments on the Stevens [1,2] rearrangement mechanism. The mechanism of the Stevens [1,2] rearrangement has been a topic of debate for a long time.[37,38] The rearrangement mechanism can be described as a net 4-electron [1,2]-sigmatropic rearrangement. Thus, according to the Woodward-Hoffmann rules,[39] a concerted rearrangement would result in an inversion of configuration (due to the requirement that one of the orbital components should be antarafacial) at the migrating carbon. However, experimentalists have repeatedly observed *retention* of configuration in Stevens rearrangements;[40] this observation is often rationalized by invoking a non-concerted, radical-pair type mechanism, which has experimental support from the observation of the CIDNP (Chemically Induced Dynamic Nuclear Polarization) effect during some Stevens rearrangements. Ghigo et al.[24] studied this mechanism theoretically in 2010 and reported diradical pathways in most rearrangements studied. The authors discovered that the phenyltrimethylammonium ylide was the one exception. Their computational (DFT) work supported a concerted rearrangement due to the ability of the migrating group, a phenyl group in their case, to delocalize the negative charge and the lack of hydrogens at the same atomic center. Our computed Stevens rearrangement TSS is confirmed to correspond to a concerted process by Intrinsic Reaction Coordinate (IRC) calculations. This is observation is consistent with the carbamate group (presumably) being able to delocalize the negative charge. See Figures S4 and S5 for IRC plots.

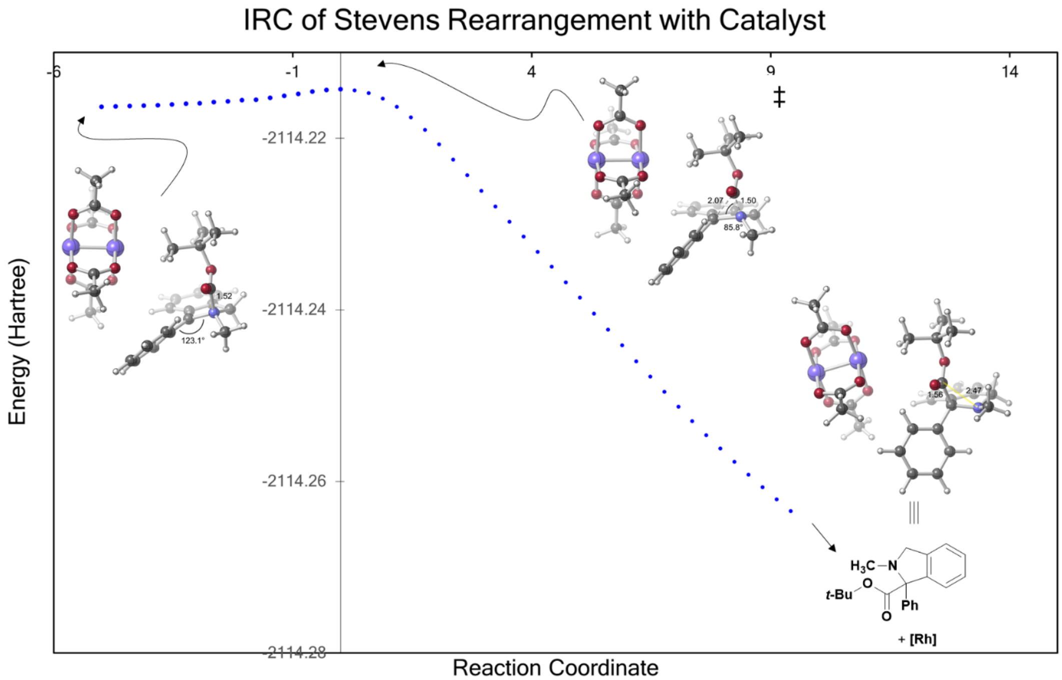


Figure S4. Intrinsic Reaction Coordinate (IRC) plot of Stevens Rearrangement step with Rh catalyst present.

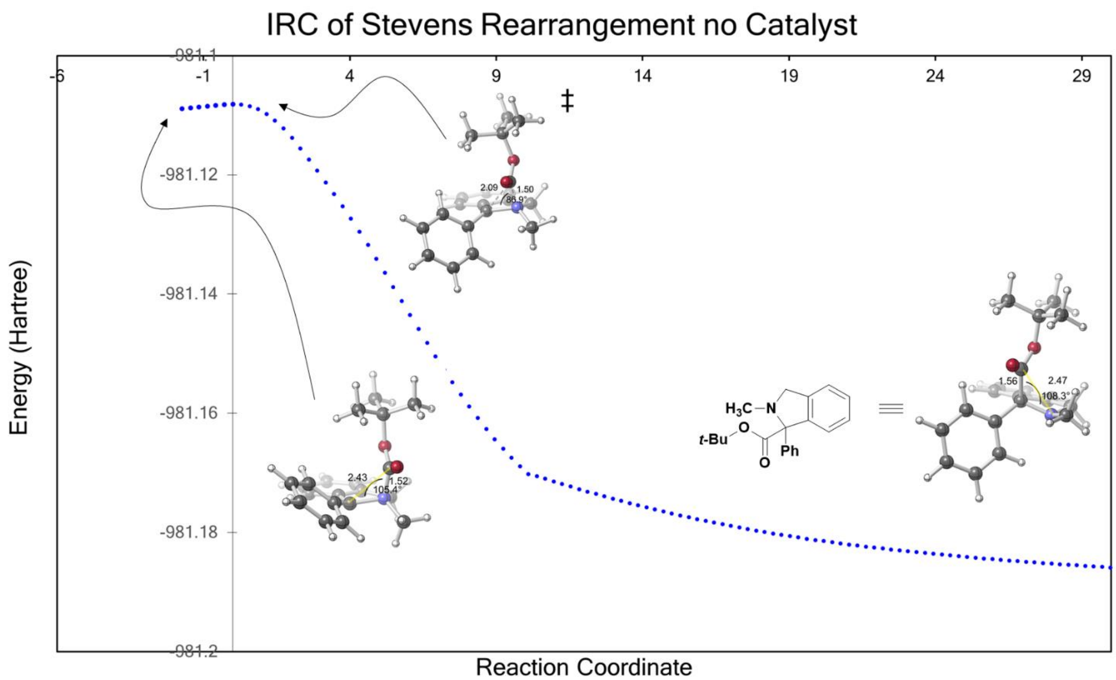


Figure S5. Intrinsic Reaction Coordinate (IRC) plot of Stevens Rearrangement step with Rh catalyst absent.

Energy and Frequencies.

uB3LYP/LANL2DZ[6-31G(d)]

Table S1. Free Energies, Electronic Energies, and Frequencies for computed structures for *N*-attack in which the metal catalyst is involved optimized at uB3LYP/LANL2DZ[6-31G(d)]. File number refers to ordering as found in concatenated .mol2 file.

System	File number	Electronic + thermal Free Energies, G (Hartree)	Electronic Energy, E (Hartree)	ΔG	ΔE	Lowest Frequency
TSS – N attacks (TS1)	1	-2113.721793	-2114.24535	13.4	9.6	-165.03
Reactant (26)	2	-2113.743079	-2114.260587	[0]	[0]	4.07
Metal-bound ylide (27)	7	-2113.721905	-2114.247219	13.3	8.4	24.01

Table S2. Free Energies, Electronic Energies, and Frequencies for computed structures for the Stevens rearrangement in which the metal catalyst is involved optimized at uB3LYP/LANL2DZ[6-31G(d)]. File number refers to ordering as found in concatenated .mol2 file.

System	File number	Electronic + thermal Free Energies, G (Hartree)	Electronic Energy, E (Hartree)	ΔG	ΔE	Lowest Frequency
Stevens rearrangement TSS ($TS\text{-}Stevens_{cat}$)	5	-2113.695073	-2114.214303	2.8	1.4	-236.48
Free ylide (28)	8	-2113.699459	-2114.216499	[0]	[0]	9.63
Final Product (25)	3	-2113.774012	-2114.290523	-46.8	-46.5	10.80

Table S3. Free Energies, Electronic Energies, and Frequencies for computed structures in which the metal catalyst is *absent* optimized at uB3LYP/LANL2DZ[6-31G(d)]. File number refers to ordering as found in concatenated .mol2 file.

System	File number	Electronic + thermal Free Energies, G (Hartree)	Electronic Energy, E (Hartree)	ΔG	ΔE	Lowest Frequency
Stevens rearrangement TSS (TS-Stevens)	6	-980.769834	-981.108142	2.6	2.4	-202.92
Ylide (28)	9	-980.774042	-981.1119932	[0]	[0]	8.68
Final Product (25)	4	-980.850635	-981.1892336	-48.1	-48.5	18.48

uB3LYP-D3(BJ) single-point calculations

Table S4. Single-point calculation Electronic Energies for computed structures for *N*-attack in which the metal catalyst is involved at the uB3LYP-D3(BJ)/LANL2DZ[6-31G(d)] level of theory. Single-point energies were computed at the uB3LYP/LANL2DZ[6-31G(d)] geometry.

System	Electronic Energy, E (Hartree)	ΔE
TSS – N attacks (TS1)	-2114.557684	4.6
Reactant (26)	-2114.565084	[0]

Metal-bound ylide (27)	-2114.560645	2.8

Table S5. Single-point calculation Electronic Energies for computed structures for the Stevens rearrangement in which the metal catalyst is involved at the uB3LYP-D3(BJ)/LANL2DZ[6-31G(d)] level of theory. Single-point energies were computed at the uB3LYP/LANL2DZ[6-31G(d)] geometry.

System	Electronic Energy, E (Hartree)	ΔE
Stevens rearrangement TSS (TS-Stevens$_{cat}$)	-2114.509369	0.9
Free ylide (28)	-2114.510764	[0]
Final Product (25)	-2114.582915	-45.3

Table S6. Single-point calculation Electronic Energies for computed structures in which the metal catalyst is *absent* at the uB3LYP-D3(BJ)/LANL2DZ[6-31G(d)] level of theory. Single-point energies were computed at the uB3LYP/LANL2DZ[6-31G(d)] geometry.

System	Electronic Energy, E (Hartree)	ΔE
Stevens rearrangement TSS (TS-Stevens)	-981.2777339	3.1
Ylide (28)	-981.2827422	[0]
Final Product (25)	-981.3565186	-46.3

uM06-2X single-point calculations

Table S7. Single-point calculation Electronic Energies for computed structures for *N*-attack in which the metal catalyst is involved at the uM06-2X/LANL2DZ[6-31G(d)] level of theory. Single-point energies were computed at the uB3LYP/LANL2DZ[6-31G(d)] geometry.

System	Electronic Energy, E (Hartree)	ΔE
TSS – N attacks (TS1)	-2113.309886	-2.1
Reactant (26)	-2113.306573	[0]
Metal-bound ylide (27)	-2113.319651	-8.2

Table S8. Single-point calculation Electronic Energies for computed structures for the Stevens rearrangement in which the metal catalyst is involved at the uM06-2X/LANL2DZ[6-31G(d)] level of theory. Single-point energies were computed at the uB3LYP/LANL2DZ[6-31G(d)] geometry.

System	Electronic Energy, E (Hartree)	ΔE
Stevens rearrangement TSS (TS-Stevens$_{cat}$)	-2113.26417	0.4
Free ylide (28)	-2113.264739	[0]
Final Product (25)	-2113.339804	-47.1

Table S9. Single-point calculation Electronic Energies for computed structures in which the metal catalyst is *absent* at the uM06-2X/LANL2DZ[6-31G(d)] level of theory. Single-point energies were computed at the uB3LYP/LANL2DZ[6-31G(d)] geometry.

System	Electronic Energy, E (Hartree)	ΔE
Stevens rearrangement TSS (TS-Stevens)	-980.744731	2.3
Ylide (28)	-980.7483685	[0]
Final Product (25)	-980.8255832	-48.5

uωB97X-D single-point calculations

Table S10. Single-point calculation Electronic Energies for computed structures for *N*-attack in which the metal catalyst is involved at the uωB97X-D/LANL2DZ[6-31G(d)] level of theory. Single-point energies were computed at the uB3LYP/LANL2DZ[6-31G(d)] geometry.

System	Electronic Energy, E (Hartree)	ΔE
TSS – N attacks (TS1)	-2113.750742	2.3
Reactant (26)	-2113.754343	[0]
Metal-bound ylide (27)	-2113.757618	-2.1

Table S11. Single-point calculation Electronic Energies for computed structures for the Stevens rearrangement in which the metal catalyst is involved at the uωB97X-D /LANL2DZ[6-31G(d)] level of theory. Single-point energies were computed at the uB3LYP/LANL2DZ[6-31G(d)] geometry.

System	Electronic Energy, E (Hartree)	ΔE
Stevens rearrangement TSS (TS-Stevens$_{cat}$)	-2113.704963	-0.6
Free ylide (28)	-2113.704079	[0]
Final Product (25)	-2113.784142	-50.2

Table S12. Single-point calculation Electronic Energies for computed structures in which the metal catalyst is *absent* at the uωB97X-D/LANL2DZ[6-31G(d)] level of theory. Single-point energies were computed at the uB3LYP/LANL2DZ[6-31G(d)] geometry.

System	Electronic Energy, E (Hartree)	ΔE
Stevens rearrangement TSS (TS-Stevens)	-980.8627539	1.8
Ylide (28)	-980.8655603	[0]
Final Product (25)	-980.9468588	-51.0

X-RAY DATA

ISOCHROMAN 11c

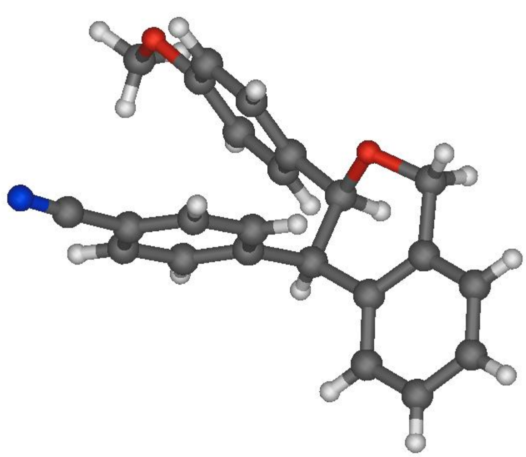


Table 1. Crystal data and structure refinement for [$C_{23}H_{19}NO_2$].

Identification code	JF2831FMI	
Empirical formula	C23 H19 N O2	
Formula weight	341.39	
Temperature	100(2) K	
Wavelength	1.54178 Å	
Crystal system	Orthorhombic	
Space group	$P2_12_12_1$	
Unit cell dimensions	a = 5.5431(2) Å	α= 90°.
	b = 17.2727(6) Å	β= 90°.
	c = 17.7127(6) Å	γ = 90°.
Volume	1695.89(10) Å^3	
Z	4	
Density (calculated)	1.337 Mg/m^3	
Absorption coefficient	0.675 mm^{-1}	
F(000)	720	
Crystal size	0.214 x 0.106 x 0.076 mm^3	
Crystal color and habit	Colorless Block	
Diffractometer	Bruker Photon100 CMOS	
Theta range for data collection	3.574 to 72.081°.	
Index ranges	-5<=h<=6, -21<=k<=21, -21<=l<=21	
Reflections collected	10237	
Independent reflections	3335 [R(int) = 0.0184]	
Observed reflections (I > 2sigma(I))	3267	
Completeness to theta = 67.679°	100.0 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.9370 and 0.8744	
Solution method	SHELXT (Sheldrick, 2014)	
Refinement method	SHELXL-2018/3 (Sheldrick, 2018) Full-matrix least-squares on F^2	
Data / restraints / parameters	3335 / 0 / 312	
Goodness-of-fit on F^2	1.043	
Final R indices [I>2sigma(I)]	R1 = 0.0250, wR2 = 0.0645	
R indices (all data)	R1 = 0.0258, wR2 = 0.0651	
Absolute structure parameter	-0.08(6)	
Extinction coefficient	0.0042(4)	
Largest diff. peak and hole	0.195 and -0.160 e.Å^{-3}	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for JF2831FMI. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	7252(3)	4451(1)	1222(1)	14(1)
O(1)	4745(2)	4644(1)	1209(1)	15(1)
C(2)	8445(3)	4838(1)	1920(1)	14(1)
C(3)	7938(3)	5703(1)	1883(1)	14(1)
C(4)	5973(3)	5982(1)	1475(1)	14(1)
C(5)	4419(3)	5441(1)	1013(1)	16(1)
C(6)	5462(3)	6774(1)	1471(1)	16(1)
C(7)	6926(3)	7284(1)	1862(1)	16(1)
C(8)	8923(3)	7012(1)	2260(1)	16(1)
C(9)	9410(3)	6224(1)	2271(1)	15(1)
C(10)	7526(3)	3582(1)	1172(1)	14(1)
C(11)	5795(3)	3079(1)	1470(1)	16(1)
C(12)	6061(3)	2284(1)	1402(1)	17(1)
C(13)	8056(3)	1975(1)	1033(1)	14(1)
O(13)	8166(2)	1183(1)	1005(1)	17(1)
C(14)	9804(3)	2468(1)	729(1)	16(1)
C(15)	9510(3)	3267(1)	803(1)	16(1)
C(16)	10348(3)	853(1)	719(1)	20(1)
C(17)	7634(3)	4494(1)	2667(1)	14(1)
C(18)	5549(3)	4750(1)	3034(1)	17(1)
C(19)	4844(3)	4428(1)	3717(1)	17(1)
C(20)	6217(3)	3838(1)	4042(1)	16(1)
C(21)	8327(3)	3579(1)	3691(1)	16(1)
C(22)	9006(3)	3914(1)	3008(1)	15(1)
C(23)	5379(3)	3480(1)	4734(1)	18(1)
N(23)	4648(3)	3193(1)	5272(1)	25(1)

Table 3. Bond lengths [Å] and angles [°] for JF2831FMI.

C(1)-O(1)	1.4294(18)	C(17)-C(22)	1.396(2)
C(1)-C(10)	1.5113(19)	C(17)-C(18)	1.397(2)
C(1)-C(2)	1.553(2)	C(18)-C(19)	1.388(2)
C(1)-H(1)	1.021(19)	C(18)-H(18)	0.99(2)
O(1)-C(5)	1.4307(17)	C(19)-C(20)	1.396(2)
C(2)-C(17)	1.520(2)	C(19)-H(19)	0.96(2)
C(2)-C(3)	1.521(2)	C(20)-C(21)	1.397(2)
C(2)-H(2)	0.93(2)	C(20)-C(23)	1.450(2)
C(3)-C(4)	1.393(2)	C(21)-C(22)	1.392(2)
C(3)-C(9)	1.396(2)	C(21)-H(21)	0.99(2)
C(4)-C(6)	1.397(2)	C(22)-H(22)	0.96(2)
C(4)-C(5)	1.511(2)	C(23)-N(23)	1.147(2)
C(5)-H(5A)	1.00(2)		
C(5)-H(5B)	0.992(19)	O(1)-C(1)-C(10)	109.16(12)
C(6)-C(7)	1.384(2)	O(1)-C(1)-C(2)	109.10(12)
C(6)-H(6)	0.95(2)	C(10)-C(1)-C(2)	115.59(12)
C(7)-C(8)	1.393(2)	O(1)-C(1)-H(1)	107.9(10)
C(7)-H(7)	0.96(2)	C(10)-C(1)-H(1)	108.8(10)
C(8)-C(9)	1.387(2)	C(2)-C(1)-H(1)	106.0(10)
C(8)-H(8)	0.95(2)	C(1)-O(1)-C(5)	110.55(11)
C(9)-H(9)	0.94(2)	C(17)-C(2)-C(3)	111.54(12)
C(10)-C(15)	1.390(2)	C(17)-C(2)-C(1)	113.49(12)
C(10)-C(11)	1.399(2)	C(3)-C(2)-C(1)	108.04(12)
C(11)-C(12)	1.387(2)	C(17)-C(2)-H(2)	108.9(12)
C(11)-H(11)	0.97(2)	C(3)-C(2)-H(2)	110.2(12)
C(12)-C(13)	1.391(2)	C(1)-C(2)-H(2)	104.3(12)
C(12)-H(12)	0.97(2)	C(4)-C(3)-C(9)	119.30(14)
C(13)-O(13)	1.3711(18)	C(4)-C(3)-C(2)	120.42(14)
C(13)-C(14)	1.397(2)	C(9)-C(3)-C(2)	120.27(14)
O(13)-C(16)	1.430(2)	C(3)-C(4)-C(6)	119.96(14)
C(14)-C(15)	1.396(2)	C(3)-C(4)-C(5)	120.84(13)
C(14)-H(14)	0.96(2)	C(6)-C(4)-C(5)	119.17(14)
C(15)-H(15)	0.95(2)	O(1)-C(5)-C(4)	113.08(12)
C(16)-H(16A)	0.98(2)	O(1)-C(5)-H(5A)	108.1(11)
C(16)-H(16B)	0.98(2)	C(4)-C(5)-H(5A)	109.1(12)
C(16)-H(16C)	0.99(2)	O(1)-C(5)-H(5B)	108.7(10)

C(4)-C(5)-H(5B)	109.2(11)
H(5A)-C(5)-H(5B)	108.6(16)
C(7)-C(6)-C(4)	120.15(15)
C(7)-C(6)-H(6)	119.9(12)
C(4)-C(6)-H(6)	119.9(12)
C(6)-C(7)-C(8)	120.29(14)
C(6)-C(7)-H(7)	120.0(12)
C(8)-C(7)-H(7)	119.7(12)
C(9)-C(8)-C(7)	119.48(15)
C(9)-C(8)-H(8)	120.8(12)
C(7)-C(8)-H(8)	119.7(12)
C(8)-C(9)-C(3)	120.80(15)
C(8)-C(9)-H(9)	119.4(12)
C(3)-C(9)-H(9)	119.8(12)
C(15)-C(10)-C(11)	118.45(14)
C(15)-C(10)-C(1)	119.74(14)
C(11)-C(10)-C(1)	121.78(14)
C(12)-C(11)-C(10)	120.69(15)
C(12)-C(11)-H(11)	119.3(11)
C(10)-C(11)-H(11)	120.0(11)
C(11)-C(12)-C(13)	120.30(15)
C(11)-C(12)-H(12)	120.4(12)
C(13)-C(12)-H(12)	119.3(12)
O(13)-C(13)-C(12)	115.73(14)
O(13)-C(13)-C(14)	124.30(14)
C(12)-C(13)-C(14)	119.97(14)
C(13)-O(13)-C(16)	116.61(12)
C(15)-C(14)-C(13)	119.02(14)
C(15)-C(14)-H(14)	119.7(12)
C(13)-C(14)-H(14)	121.3(12)
C(10)-C(15)-C(14)	121.56(14)
C(10)-C(15)-H(15)	119.2(14)
C(14)-C(15)-H(15)	119.2(14)
O(13)-C(16)-H(16A)	112.0(13)
O(13)-C(16)-H(16B)	108.8(13)
H(16A)-C(16)-H(16B)	109.5(19)
O(13)-C(16)-H(16C)	105.8(14)
H(16A)-C(16)-H(16C)	107.2(19)
H(16B)-C(16)-H(16C)	113.6(17)
C(22)-C(17)-C(18)	118.45(14)
C(22)-C(17)-C(2)	119.80(14)
C(18)-C(17)-C(2)	121.74(13)
C(19)-C(18)-C(17)	120.76(14)
C(19)-C(18)-H(18)	118.0(13)
C(17)-C(18)-H(18)	121.2(13)
C(18)-C(19)-C(20)	119.92(15)
C(18)-C(19)-H(19)	119.9(13)
C(20)-C(19)-H(19)	120.2(13)
C(19)-C(20)-C(21)	120.39(14)
C(19)-C(20)-C(23)	119.08(15)
C(21)-C(20)-C(23)	120.50(14)
C(22)-C(21)-C(20)	118.69(14)
C(22)-C(21)-H(21)	120.9(13)
C(20)-C(21)-H(21)	120.4(12)
C(21)-C(22)-C(17)	121.78(15)
C(21)-C(22)-H(22)	119.8(12)
C(17)-C(22)-H(22)	118.4(12)
N(23)-C(23)-C(20)	177.88(19)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x 10^3$) for JF2831FMI. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}

C(1)	14(1)	15(1)	15(1)	1(1)	0(1)	-1(1)
O(1)	13(1)	12(1)	21(1)	0(1)	-2(1)	0(1)
C(2)	12(1)	13(1)	16(1)	0(1)	0(1)	0(1)
C(3)	14(1)	15(1)	13(1)	2(1)	3(1)	0(1)
C(4)	14(1)	15(1)	14(1)	1(1)	2(1)	-1(1)
C(5)	16(1)	15(1)	18(1)	1(1)	-3(1)	1(1)
C(6)	16(1)	16(1)	16(1)	2(1)	-1(1)	0(1)
C(7)	20(1)	11(1)	18(1)	2(1)	2(1)	0(1)
C(8)	19(1)	16(1)	15(1)	0(1)	0(1)	-4(1)
C(9)	15(1)	18(1)	13(1)	2(1)	0(1)	-1(1)
C(10)	16(1)	14(1)	12(1)	0(1)	-3(1)	-1(1)
C(11)	14(1)	18(1)	16(1)	-1(1)	1(1)	1(1)
C(12)	17(1)	17(1)	16(1)	1(1)	1(1)	-4(1)
C(13)	17(1)	14(1)	12(1)	-1(1)	-4(1)	-1(1)
O(13)	18(1)	14(1)	20(1)	-1(1)	1(1)	-1(1)
C(14)	16(1)	17(1)	15(1)	1(1)	2(1)	1(1)
C(15)	15(1)	17(1)	16(1)	2(1)	1(1)	-2(1)
C(16)	23(1)	15(1)	22(1)	-1(1)	5(1)	3(1)
C(17)	14(1)	12(1)	15(1)	-1(1)	-2(1)	-2(1)
C(18)	17(1)	14(1)	20(1)	2(1)	-1(1)	2(1)
C(19)	14(1)	18(1)	19(1)	-2(1)	2(1)	1(1)
C(20)	17(1)	15(1)	15(1)	-1(1)	-1(1)	-4(1)
C(21)	18(1)	13(1)	16(1)	0(1)	-2(1)	1(1)
C(22)	14(1)	14(1)	17(1)	-2(1)	1(1)	1(1)
C(23)	18(1)	18(1)	18(1)	-2(1)	-1(1)	0(1)
N(23)	26(1)	29(1)	20(1)	4(1)	4(1)	1(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for JF2831FMI.

	x	y	z	U(eq)
H(1)	8040(30)	4697(10)	760(10)	8(4)

H(2)	10090(40)	4745(11)	1857(11)	12(4)
H(5A)	2690(40)	5576(11)	1094(11)	19(5)
H(5B)	4810(40)	5509(10)	470(10)	10(4)
H(6)	4140(40)	6963(11)	1190(11)	18(5)
H(7)	6570(40)	7828(12)	1860(11)	16(5)
H(8)	9930(40)	7368(11)	2522(11)	19(5)
H(9)	10740(40)	6042(11)	2544(12)	19(5)
H(11)	4390(40)	3285(11)	1728(11)	17(5)
H(12)	4880(40)	1936(12)	1625(12)	19(5)
H(14)	11180(40)	2268(12)	466(11)	20(5)
H(15)	10690(50)	3604(13)	592(12)	29(6)
H(16A)	11770(40)	1047(13)	984(13)	27(6)
H(16B)	10480(40)	975(12)	182(12)	25(5)
H(16C)	10240(40)	290(13)	825(12)	26(5)
H(18)	4550(40)	5171(12)	2822(12)	21(5)
H(19)	3400(40)	4604(12)	3959(12)	25(5)
H(21)	9300(40)	3162(12)	3922(12)	21(5)
H(22)	10470(40)	3748(11)	2763(11)	18(5)

Table 6. Torsion angles [°] for JF2831FMI.

C(10)-C(1)-O(1)-C(5)	-161.47(11)	C(1)-O(1)-C(5)-C(4)	-49.74(16)
C(2)-C(1)-O(1)-C(5)	71.35(14)	C(3)-C(4)-C(5)-O(1)	16.4(2)
O(1)-C(1)-C(2)-C(17)	68.87(15)	C(6)-C(4)-C(5)-O(1)	-165.47(13)
C(10)-C(1)-C(2)-C(17)	-54.56(18)	C(3)-C(4)-C(6)-C(7)	1.0(2)
O(1)-C(1)-C(2)-C(3)	-55.36(15)	C(5)-C(4)-C(6)-C(7)	-177.09(14)
C(10)-C(1)-C(2)-C(3)	-178.80(13)	C(4)-C(6)-C(7)-C(8)	0.2(2)
C(17)-C(2)-C(3)-C(4)	-102.21(16)	C(6)-C(7)-C(8)-C(9)	-1.0(2)
C(1)-C(2)-C(3)-C(4)	23.18(19)	C(7)-C(8)-C(9)-C(3)	0.6(2)
C(17)-C(2)-C(3)-C(9)	76.41(18)	C(4)-C(3)-C(9)-C(8)	0.6(2)
C(1)-C(2)-C(3)-C(9)	-158.19(13)	C(2)-C(3)-C(9)-C(8)	-178.01(14)
C(9)-C(3)-C(4)-C(6)	-1.4(2)	O(1)-C(1)-C(10)-C(15)	147.26(14)
C(2)-C(3)-C(4)-C(6)	177.20(14)	C(2)-C(1)-C(10)-C(15)	-89.34(17)
C(9)-C(3)-C(4)-C(5)	176.65(13)	O(1)-C(1)-C(10)-C(11)	-30.97(19)
C(2)-C(3)-C(4)-C(5)	-4.7(2)	C(2)-C(1)-C(10)-C(11)	92.43(18)

C(15)-C(10)-C(11)-C(12)	0.3(2)	C(1)-C(2)-C(17)-C(22)	94.98(16)
C(1)-C(10)-C(11)-C(12)	178.57(14)	C(3)-C(2)-C(17)-C(18)	36.5(2)
C(10)-C(11)-C(12)-C(13)	-0.2(2)	C(1)-C(2)-C(17)-C(18)	-85.85(17)
C(11)-C(12)-C(13)-O(13)	179.14(14)	C(22)-C(17)-C(18)-C(19)	-0.7(2)
C(11)-C(12)-C(13)-C(14)	0.0(2)	C(2)-C(17)-C(18)-C(19)	-179.89(14)
C(12)-C(13)-O(13)-C(16)	-171.99(13)	C(17)-C(18)-C(19)-C(20)	-0.5(2)
C(14)-C(13)-O(13)-C(16)	7.1(2)	C(18)-C(19)-C(20)-C(21)	1.4(2)
O(13)-C(13)-C(14)-C(15)	-179.00(14)	C(18)-C(19)-C(20)-C(23)	-176.62(15)
C(12)-C(13)-C(14)-C(15)	0.1(2)	C(19)-C(20)-C(21)-C(22)	-1.0(2)
C(11)-C(10)-C(15)-C(14)	-0.3(2)	C(23)-C(20)-C(21)-C(22)	176.93(15)
C(1)-C(10)-C(15)-C(14)	-178.54(14)	C(20)-C(21)-C(22)-C(17)	-0.2(2)
C(13)-C(14)-C(15)-C(10)	0.1(2)	C(18)-C(17)-C(22)-C(21)	1.1(2)
C(3)-C(2)-C(17)-C(22)	-142.70(14)	C(2)-C(17)-C(22)-C(21)	-179.75(14)

Symmetry transformations used to generate equivalent atoms:

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Table 1. Crystal data and structure refinement for [$C_{23}H_{20}O$].

Identification code	JF2822FMI (BDB11-11-018)	
Empirical formula	C23 H20 O	
Formula weight	312.39	
Temperature	90(2) K	
Wavelength	1.54178 Å	
Crystal system	Monoclinic	
Space group	I2	
Unit cell dimensions	a = 17.3724(8) Å	α= 90°.
	b = 5.5677(3) Å	β= 111.6606(19)°.
	c = 18.8919(12) Å	γ = 90°.
Volume	1698.27(16) Å^3	
Z	4	
Density (calculated)	1.222 Mg/m^3	
Absorption coefficient	0.562 mm^{-1}	
F(000)	664	
Crystal size	0.240 x 0.167 x 0.062 mm^3	
Crystal color and habit	Colorless Plate	

Diffractometer	Bruker APEX-II CCD
Theta range for data collection	2.956 to 69.279°.
Index ranges	-20<=h<=20, -6<=k<=6, -22<=l<=22
Reflections collected	4368
Independent reflections	2996 [R(int) = 0.0156]
Observed reflections (I > 2sigma(I))	2931
Completeness to theta = 67.679°	99.1 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9369 and 0.8379
Solution method	SHELXT (Sheldrick, 2014)
Refinement method	SHELXL-2018/3 (Sheldrick, 2018) Full-matrix least-squares on F^2
Data / restraints / parameters	2996 / 1 / 297
Goodness-of-fit on F^2	1.040
Final R indices [I>2sigma(I)]	R1 = 0.0301, wR2 = 0.0792
R indices (all data)	R1 = 0.0308, wR2 = 0.0800
Absolute structure parameter	0.05(15)
Largest diff. peak and hole	0.372 and -0.151 e.Å^{-3}

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^{2}x 10^3) for JF2822FMI. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	8822(1)	5106(3)	5479(1)	26(1)
O(1)	8862(1)	7507(3)	5754(1)	26(1)
C(2)	9278(1)	7543(4)	6560(1)	26(1)
C(3)	8904(1)	5850(3)	6964(1)	23(1)
C(4)	9062(1)	6145(4)	7738(1)	28(1)
C(5)	8767(1)	4529(4)	8130(1)	30(1)
C(6)	8292(1)	2573(4)	7747(1)	29(1)
C(7)	8116(1)	2306(4)	6973(1)	26(1)
C(8)	8421(1)	3916(3)	6575(1)	23(1)
C(9)	8217(1)	3586(3)	5726(1)	23(1)
C(10)	8602(1)	5221(4)	4633(1)	28(1)
C(11)	8889(1)	3643(4)	4265(1)	27(1)
C(12)	8746(1)	3555(3)	3446(1)	25(1)

C(13)	9074(1)	1629(4)	3179(1)	29(1)
C(14)	8981(1)	1463(4)	2419(1)	29(1)
C(15)	8561(1)	3248(4)	1911(1)	28(1)
C(16)	8233(1)	5179(4)	2170(1)	29(1)
C(17)	8322(1)	5329(4)	2927(1)	27(1)
C(18)	7312(1)	4108(3)	5267(1)	23(1)
C(19)	6848(1)	2462(4)	4726(1)	28(1)
C(20)	6011(1)	2858(4)	4302(1)	30(1)
C(21)	5632(1)	4930(4)	4412(1)	28(1)
C(22)	6091(1)	6609(4)	4946(1)	27(1)
C(23)	6923(1)	6195(4)	5371(1)	25(1)

Table 3. Bond lengths [Å] and angles [°] for JF2822FMI.

C(1)-O(1)	1.427(2)	C(10)-C(11)	1.326(3)
C(1)-C(10)	1.501(2)	C(10)-H(10)	0.96(3)
C(1)-C(9)	1.549(2)	C(11)-C(12)	1.475(2)
C(1)-H(1)	0.99(2)	C(11)-H(11)	1.01(3)
O(1)-C(2)	1.425(2)	C(12)-C(13)	1.393(3)
C(2)-C(3)	1.502(3)	C(12)-C(17)	1.395(3)
C(2)-H(2A)	0.99(2)	C(13)-C(14)	1.387(3)
C(2)-H(2B)	1.01(3)	C(13)-H(13)	0.99(2)
C(3)-C(4)	1.395(3)	C(14)-C(15)	1.388(3)
C(3)-C(8)	1.396(2)	C(14)-H(14)	0.98(3)
C(4)-C(5)	1.380(3)	C(15)-C(16)	1.387(3)
C(4)-H(4)	0.98(3)	C(15)-H(15)	0.95(2)
C(5)-C(6)	1.396(3)	C(16)-C(17)	1.384(3)
C(5)-H(5)	0.99(2)	C(16)-H(16)	1.00(2)
C(6)-C(7)	1.386(2)	C(17)-H(17)	0.89(3)
C(6)-H(6)	0.96(2)	C(18)-C(19)	1.388(3)
C(7)-C(8)	1.395(3)	C(18)-C(23)	1.394(3)
C(7)-H(7)	1.01(2)	C(19)-C(20)	1.394(3)
C(8)-C(9)	1.522(2)	C(19)-H(19)	0.97(2)
C(9)-C(18)	1.517(2)	C(20)-C(21)	1.382(3)
C(9)-H(9)	1.00(2)	C(20)-H(20)	0.95(3)

C(21)-C(22)	1.390(3)	C(3)-C(8)-C(9)	120.54(16)
C(21)-H(21)	1.01(2)	C(18)-C(9)-C(8)	111.56(14)
C(22)-C(23)	1.389(3)	C(18)-C(9)-C(1)	113.53(15)
C(22)-H(22)	0.93(2)	C(8)-C(9)-C(1)	109.40(14)
C(23)-H(23)	0.98(3)	C(18)-C(9)-H(9)	108.4(11)
		C(8)-C(9)-H(9)	108.7(11)
O(1)-C(1)-C(10)	107.87(15)	C(1)-C(9)-H(9)	104.9(11)
O(1)-C(1)-C(9)	110.45(14)	C(11)-C(10)-C(1)	122.15(17)
C(10)-C(1)-C(9)	114.17(15)	C(11)-C(10)-H(10)	122.0(15)
O(1)-C(1)-H(1)	108.4(13)	C(1)-C(10)-H(10)	115.8(15)
C(10)-C(1)-H(1)	108.4(12)	C(10)-C(11)-C(12)	128.43(17)
C(9)-C(1)-H(1)	107.4(12)	C(10)-C(11)-H(11)	117.7(14)
C(2)-O(1)-C(1)	109.87(14)	C(12)-C(11)-H(11)	113.9(14)
O(1)-C(2)-C(3)	112.95(15)	C(13)-C(12)-C(17)	118.09(16)
O(1)-C(2)-H(2A)	108.3(12)	C(13)-C(12)-C(11)	118.21(17)
C(3)-C(2)-H(2A)	109.8(13)	C(17)-C(12)-C(11)	123.67(17)
O(1)-C(2)-H(2B)	105.6(12)	C(14)-C(13)-C(12)	121.26(18)
C(3)-C(2)-H(2B)	111.1(13)	C(14)-C(13)-H(13)	119.0(14)
H(2A)-C(2)-H(2B)	109.1(18)	C(12)-C(13)-H(13)	119.7(14)
C(4)-C(3)-C(8)	119.32(17)	C(13)-C(14)-C(15)	119.95(18)
C(4)-C(3)-C(2)	120.22(16)	C(13)-C(14)-H(14)	119.1(13)
C(8)-C(3)-C(2)	120.42(16)	C(15)-C(14)-H(14)	120.9(13)
C(5)-C(4)-C(3)	121.24(18)	C(16)-C(15)-C(14)	119.37(17)
C(5)-C(4)-H(4)	121.9(14)	C(16)-C(15)-H(15)	121.5(14)
C(3)-C(4)-H(4)	116.9(15)	C(14)-C(15)-H(15)	119.1(14)
C(4)-C(5)-C(6)	119.69(17)	C(17)-C(16)-C(15)	120.51(18)
C(4)-C(5)-H(5)	121.6(14)	C(17)-C(16)-H(16)	119.2(14)
C(6)-C(5)-H(5)	118.7(14)	C(15)-C(16)-H(16)	120.2(14)
C(7)-C(6)-C(5)	119.28(18)	C(16)-C(17)-C(12)	120.81(18)
C(7)-C(6)-H(6)	120.4(13)	C(16)-C(17)-H(17)	119.9(15)
C(5)-C(6)-H(6)	120.2(13)	C(12)-C(17)-H(17)	119.3(15)
C(6)-C(7)-C(8)	121.32(18)	C(19)-C(18)-C(23)	118.28(16)
C(6)-C(7)-H(7)	118.6(13)	C(19)-C(18)-C(9)	119.44(17)
C(8)-C(7)-H(7)	120.1(13)	C(23)-C(18)-C(9)	122.27(16)
C(7)-C(8)-C(3)	119.13(16)	C(18)-C(19)-C(20)	121.14(19)
C(7)-C(8)-C(9)	120.33(16)	C(18)-C(19)-H(19)	119.1(12)

C(20)-C(19)-H(19)	119.7(12)	C(23)-C(22)-C(21)	120.25(18)
C(21)-C(20)-C(19)	120.00(18)	C(23)-C(22)-H(22)	117.9(13)
C(21)-C(20)-H(20)	120.5(15)	C(21)-C(22)-H(22)	121.9(13)
C(19)-C(20)-H(20)	119.5(15)	C(22)-C(23)-C(18)	120.82(17)
C(20)-C(21)-C(22)	119.49(17)	C(22)-C(23)-H(23)	120.3(14)
C(20)-C(21)-H(21)	120.6(14)	C(18)-C(23)-H(23)	118.9(14)
C(22)-C(21)-H(21)	119.9(14)		

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2$x 10^3) for JF2822FMI. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	22(1)	29(1)	26(1)	1(1)	10(1)	3(1)
O(1)	25(1)	28(1)	25(1)	1(1)	9(1)	-1(1)
C(2)	21(1)	29(1)	26(1)	-1(1)	6(1)	-1(1)
C(3)	18(1)	25(1)	24(1)	0(1)	5(1)	3(1)
C(4)	23(1)	30(1)	25(1)	-2(1)	3(1)	1(1)
C(5)	29(1)	39(1)	21(1)	0(1)	7(1)	5(1)
C(6)	30(1)	31(1)	28(1)	6(1)	12(1)	4(1)
C(7)	24(1)	26(1)	27(1)	0(1)	9(1)	0(1)
C(8)	19(1)	24(1)	23(1)	1(1)	6(1)	4(1)
C(9)	22(1)	25(1)	23(1)	-2(1)	8(1)	1(1)
C(10)	24(1)	33(1)	28(1)	2(1)	11(1)	1(1)
C(11)	22(1)	31(1)	26(1)	1(1)	8(1)	1(1)
C(12)	19(1)	30(1)	27(1)	-2(1)	9(1)	-3(1)
C(13)	28(1)	32(1)	27(1)	3(1)	9(1)	5(1)
C(14)	30(1)	32(1)	28(1)	-2(1)	12(1)	4(1)
C(15)	27(1)	35(1)	24(1)	1(1)	11(1)	-1(1)
C(16)	26(1)	30(1)	30(1)	6(1)	11(1)	2(1)
C(17)	25(1)	27(1)	33(1)	-2(1)	14(1)	0(1)
C(18)	23(1)	27(1)	19(1)	1(1)	9(1)	-2(1)
C(19)	26(1)	29(1)	26(1)	-3(1)	8(1)	1(1)
C(20)	27(1)	34(1)	26(1)	-7(1)	6(1)	-3(1)

C(21)	23(1)	37(1)	23(1)	0(1)	7(1)	0(1)
C(22)	26(1)	32(1)	24(1)	-3(1)	11(1)	4(1)
C(23)	25(1)	28(1)	21(1)	-4(1)	8(1)	-2(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^{3}) for JF2822FMI.

	x	y	z	U(eq)
H(1)	9380(13)	4370(40)	5713(11)	21(5)
H(2A)	9866(14)	7120(40)	6680(11)	24(5)
H(2B)	9244(13)	9260(50)	6719(12)	25(5)
H(4)	9379(15)	7570(50)	7984(13)	36(6)
H(5)	8886(13)	4710(40)	8681(12)	27(5)
H(6)	8071(13)	1470(40)	8011(12)	22(5)
H(7)	7764(14)	890(40)	6699(12)	28(5)
H(9)	8334(12)	1880(40)	5630(11)	16(5)
H(10)	8238(15)	6510(50)	4373(14)	39(6)
H(11)	9263(15)	2330(50)	4574(13)	37(6)
H(13)	9385(14)	370(50)	3536(13)	29(6)
H(14)	9216(15)	70(50)	2249(13)	35(6)
H(15)	8507(13)	3120(40)	1394(13)	28(6)
H(16)	7918(14)	6460(50)	1808(13)	31(6)
H(17)	8113(14)	6580(50)	3089(13)	29(6)
H(19)	7108(13)	980(40)	4659(12)	26(5)
H(20)	5703(15)	1670(50)	3949(14)	38(6)
H(21)	5025(15)	5240(40)	4107(13)	32(6)
H(22)	5856(13)	8030(40)	5035(12)	24(5)
H(23)	7245(14)	7370(50)	5746(13)	35(6)

Table 6. Torsion angles [°] for JF2822FMI.

C(10)-C(1)-O(1)-C(2)	-164.53(13)	C(1)-O(1)-C(2)-C(3)	-54.09(19)
C(9)-C(1)-O(1)-C(2)	70.07(18)	O(1)-C(2)-C(3)-C(4)	-160.97(16)

O(1)-C(2)-C(3)-C(8)	21.4(2)	C(10)-C(11)-C(12)-C(13)	-176.18(19)
C(8)-C(3)-C(4)-C(5)	1.7(3)	C(10)-C(11)-C(12)-C(17)	5.7(3)
C(2)-C(3)-C(4)-C(5)	-176.03(17)	C(17)-C(12)-C(13)-C(14)	-0.2(3)
C(3)-C(4)-C(5)-C(6)	-0.8(3)	C(11)-C(12)-C(13)-C(14)	-178.39(17)
C(4)-C(5)-C(6)-C(7)	-0.9(3)	C(12)-C(13)-C(14)-C(15)	0.5(3)
C(5)-C(6)-C(7)-C(8)	1.7(3)	C(13)-C(14)-C(15)-C(16)	-0.3(3)
C(6)-C(7)-C(8)-C(3)	-0.8(3)	C(14)-C(15)-C(16)-C(17)	-0.2(3)
C(6)-C(7)-C(8)-C(9)	-179.87(16)	C(15)-C(16)-C(17)-C(12)	0.5(3)
C(4)-C(3)-C(8)-C(7)	-0.8(2)	C(13)-C(12)-C(17)-C(16)	-0.3(3)
C(2)-C(3)-C(8)-C(7)	176.85(16)	C(11)-C(12)-C(17)-C(16)	177.78(17)
C(4)-C(3)-C(8)-C(9)	178.21(16)	C(8)-C(9)-C(18)-C(19)	-130.14(17)
C(2)-C(3)-C(8)-C(9)	-4.1(2)	C(1)-C(9)-C(18)-C(19)	105.7(2)
C(7)-C(8)-C(9)-C(18)	70.0(2)	C(8)-C(9)-C(18)-C(23)	49.7(2)
C(3)-C(8)-C(9)-C(18)	-109.04(18)	C(1)-C(9)-C(18)-C(23)	-74.5(2)
C(7)-C(8)-C(9)-C(1)	-163.56(16)	C(23)-C(18)-C(19)-C(20)	-0.9(3)
C(3)-C(8)-C(9)-C(1)	17.4(2)	C(9)-C(18)-C(19)-C(20)	178.94(17)
O(1)-C(1)-C(9)-C(18)	75.86(19)	C(18)-C(19)-C(20)-C(21)	0.7(3)
C(10)-C(1)-C(9)-C(18)	-45.9(2)	C(19)-C(20)-C(21)-C(22)	0.1(3)
O(1)-C(1)-C(9)-C(8)	-49.47(18)	C(20)-C(21)-C(22)-C(23)	-0.7(3)
C(10)-C(1)-C(9)-C(8)	-171.22(15)	C(21)-C(22)-C(23)-C(18)	0.5(3)
O(1)-C(1)-C(10)-C(11)	146.39(18)	C(19)-C(18)-C(23)-C(22)	0.3(3)
C(9)-C(1)-C(10)-C(11)	-90.4(2)	C(9)-C(18)-C(23)-C(22)	-179.52(17)
C(1)-C(10)-C(11)-C(12)	-178.39(17)		

Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for JF2822FMI [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
C(9)-H(9)...O(1)#1	1.00(2)	2.58(2)	3.560(2)	165.3(15)

Symmetry transformations used to generate equivalent atoms:

#1 x,y-1,z

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Table 1. Crystal data and structure refinement for [$C_{28}H_{24}O_2$].

Identification code	JF2849FMI (MG-2-184)	
Empirical formula	C28 H24 O2	
Formula weight	392.47	
Temperature	90(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 9.5034(17) Å	α= 96.945(3)°.
	b = 10.6505(19) Å	β= 102.069(3)°.
	c = 21.889(4) Å	γ = 99.440(3)°.
Volume	2109.3(7) Å^3	
Z, Z'	4, 2	
Density (calculated)	1.236 Mg/m^3	
Absorption coefficient	0.076 mm^{-1}	
F(000)	832	
Crystal size	0.781 x 0.652 x 0.320 mm^3	
Crystal color and habit	Colorless Block	
Diffractometer	Bruker APEX-II CCD	
Theta range for data collection	1.928 to 27.500°.	
Index ranges	-12<=h<=12, -13<=k<=13, -28<=l<=28	
Reflections collected	19292	
Independent reflections	9689 [R(int) = 0.0110]	
Observed reflections (I > 2sigma(I))	8901	
Completeness to theta = 25.242°	100.0 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.9673 and 0.8536	
Solution method	SHELXT (Sheldrick, 2014)	
Refinement method	SHELXL-2018/3 (Sheldrick, 2018) Full-matrix least-squares on F^2	
Data / restraints / parameters	9689 / 0 / 733	
Goodness-of-fit on F^2	1.033	
Final R indices [I>2sigma(I)]	R1 = 0.0361, wR2 = 0.0932	
R indices (all data)	R1 = 0.0389, wR2 = 0.0959	
Largest diff. peak and hole	0.377 and -0.199 e.Å^{-3}	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^{2}x 10^3)

for JF2849FMI. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	8948(1)	9403(1)	7555(1)	15(1)
O(1)	8398(1)	8243(1)	7773(1)	16(1)
C(2)	8142(1)	10481(1)	7747(1)	15(1)
C(3)	8186(1)	10582(1)	8449(1)	15(1)
C(4)	8442(1)	9560(1)	8777(1)	16(1)
C(5)	8841(1)	8364(1)	8447(1)	16(1)
C(6)	8416(1)	9676(1)	9418(1)	19(1)
C(7)	8168(1)	10803(1)	9734(1)	20(1)
C(8)	7934(1)	11827(1)	9409(1)	20(1)
C(9)	7928(1)	11708(1)	8770(1)	17(1)
C(10)	8801(1)	9101(1)	6850(1)	16(1)
C(11)	9741(1)	9857(1)	6568(1)	20(1)
C(12)	9635(1)	9615(1)	5921(1)	22(1)
C(13)	8587(1)	8588(1)	5546(1)	20(1)
O(13)	8595(1)	8401(1)	4914(1)	30(1)
C(14)	7630(1)	7829(1)	5817(1)	20(1)
C(15)	7737(1)	8101(1)	6467(1)	18(1)
C(16)	7786(1)	7210(1)	4544(1)	31(1)
C(17)	6574(1)	10299(1)	7358(1)	16(1)
C(18)	6222(1)	11104(1)	6916(1)	23(1)
C(19)	4795(1)	10948(1)	6552(1)	29(1)
C(20)	3703(1)	9983(1)	6624(1)	26(1)
C(21)	4037(1)	9176(1)	7063(1)	22(1)
C(22)	5457(1)	9337(1)	7430(1)	19(1)
C(23)	8168(1)	7117(1)	8631(1)	16(1)
C(24)	6731(1)	6502(1)	8341(1)	22(1)
C(25)	6109(1)	5381(1)	8528(1)	25(1)
C(26)	6909(1)	4863(1)	9009(1)	22(1)
C(27)	8345(1)	5460(1)	9294(1)	23(1)
C(28)	8979(1)	6578(1)	9103(1)	20(1)
C(31)	10312(1)	5326(1)	2419(1)	17(1)
O(31)	9509(1)	6304(1)	2242(1)	18(1)

C(32)	9229(1)	4032(1)	2356(1)	16(1)
C(33)	8163(1)	3770(1)	1710(1)	16(1)
C(34)	7977(1)	4746(1)	1343(1)	16(1)
C(35)	8860(1)	6116(1)	1581(1)	17(1)
C(36)	6978(1)	4466(1)	752(1)	20(1)
C(37)	6145(1)	3235(1)	530(1)	23(1)
C(38)	6312(1)	2262(1)	897(1)	23(1)
C(39)	7315(1)	2529(1)	1479(1)	19(1)
C(40)	11244(1)	5812(1)	3079(1)	17(1)
C(41)	12523(1)	5348(1)	3289(1)	21(1)
C(42)	13411(1)	5810(1)	3889(1)	23(1)
C(43)	13052(1)	6772(1)	4286(1)	20(1)
O(43)	14058(1)	7234(1)	4850(1)	26(1)
C(44)	11756(1)	7213(1)	4093(1)	21(1)
C(45)	10860(1)	6722(1)	3493(1)	20(1)
C(46)	13898(1)	8405(1)	5201(1)	29(1)
C(47)	8443(1)	4009(1)	2894(1)	16(1)
C(48)	9085(1)	3629(1)	3461(1)	20(1)
C(49)	8398(1)	3640(1)	3964(1)	24(1)
C(50)	7046(1)	4013(1)	3906(1)	26(1)
C(51)	6396(1)	4382(1)	3344(1)	24(1)
C(52)	7091(1)	4385(1)	2843(1)	19(1)
C(53)	7915(1)	7122(1)	1482(1)	17(1)
C(54)	7041(1)	7376(1)	1904(1)	20(1)
C(55)	6094(1)	8235(1)	1799(1)	23(1)
C(56)	6016(1)	8855(1)	1272(1)	25(1)
C(57)	6901(1)	8626(1)	856(1)	26(1)
C(58)	7857(1)	7767(1)	962(1)	22(1)

Table 3. Bond lengths [Å] and angles [°] for JF2849FMI.

C(1)-O(1)	1.4353(11)	O(1)-C(5)	1.4309(11)
C(1)-C(10)	1.5090(13)	C(2)-C(3)	1.5187(13)
C(1)-C(2)	1.5447(12)	C(2)-C(17)	1.5234(13)
C(1)-H(1)	1.002(12)	C(2)-H(2)	0.997(12)

C(3)-C(9)	1.3996(13)	C(21)-C(22)	1.3909(14)
C(3)-C(4)	1.4001(13)	C(21)-H(21)	0.971(15)
C(4)-C(6)	1.3999(13)	C(22)-H(22)	0.990(13)
C(4)-C(5)	1.5292(12)	C(23)-C(24)	1.3933(14)
C(5)-C(23)	1.5135(13)	C(23)-C(28)	1.3956(13)
C(5)-H(5)	0.998(12)	C(24)-C(25)	1.3899(14)
C(6)-C(7)	1.3913(14)	C(24)-H(24)	0.972(14)
C(6)-H(6)	0.979(13)	C(25)-C(26)	1.3894(15)
C(7)-C(8)	1.3937(14)	C(25)-H(25)	0.995(16)
C(7)-H(7)	0.970(13)	C(26)-C(27)	1.3858(16)
C(8)-C(9)	1.3876(14)	C(26)-H(26)	0.976(15)
C(8)-H(8)	0.982(14)	C(27)-C(28)	1.3944(15)
C(9)-H(9)	0.977(13)	C(27)-H(27)	0.987(14)
C(10)-C(15)	1.3914(14)	C(28)-H(28)	0.981(14)
C(10)-C(11)	1.3946(13)	C(31)-O(31)	1.4319(11)
C(11)-C(12)	1.3868(14)	C(31)-C(40)	1.5085(13)
C(11)-H(11)	0.982(14)	C(31)-C(32)	1.5506(13)
C(12)-C(13)	1.3949(15)	C(31)-H(31)	0.995(12)
C(12)-H(12)	0.969(15)	O(31)-C(35)	1.4260(12)
C(13)-O(13)	1.3760(12)	C(32)-C(33)	1.5219(13)
C(13)-C(14)	1.3896(14)	C(32)-C(47)	1.5226(13)
O(13)-C(16)	1.4237(14)	C(32)-H(32)	0.996(13)
C(14)-C(15)	1.3971(14)	C(33)-C(34)	1.3990(13)
C(14)-H(14)	0.971(14)	C(33)-C(39)	1.4037(13)
C(15)-H(15)	0.976(14)	C(34)-C(36)	1.4019(14)
C(16)-H(16A)	0.995(16)	C(34)-C(35)	1.5283(13)
C(16)-H(16B)	0.995(16)	C(35)-C(53)	1.5141(13)
C(16)-H(16C)	0.991(16)	C(35)-H(35)	1.006(13)
C(17)-C(18)	1.3921(14)	C(36)-C(37)	1.3879(14)
C(17)-C(22)	1.3981(13)	C(36)-H(36)	0.979(14)
C(18)-C(19)	1.3944(15)	C(37)-C(38)	1.3946(15)
C(18)-H(18)	0.960(15)	C(37)-H(37)	0.978(15)
C(19)-C(20)	1.3837(17)	C(38)-C(39)	1.3884(15)
C(19)-H(19)	0.983(15)	C(38)-H(38)	0.979(14)
C(20)-C(21)	1.3869(16)	C(39)-H(39)	0.975(13)
C(20)-H(20)	0.984(14)	C(40)-C(45)	1.3923(13)

C(40)-C(41)	1.3968(14)		
C(41)-C(42)	1.3867(15)	O(1)-C(1)-C(10)	108.54(7)
C(41)-H(41)	0.985(14)	O(1)-C(1)-C(2)	109.94(7)
C(42)-C(43)	1.3954(14)	C(10)-C(1)-C(2)	113.46(7)
C(42)-H(42)	0.979(15)	O(1)-C(1)-H(1)	108.5(7)
C(43)-O(43)	1.3738(12)	C(10)-C(1)-H(1)	109.0(7)
C(43)-C(44)	1.3914(14)	C(2)-C(1)-H(1)	107.3(7)
O(43)-C(46)	1.4309(14)	C(5)-O(1)-C(1)	111.57(7)
C(44)-C(45)	1.3941(14)	C(3)-C(2)-C(17)	111.05(7)
C(44)-H(44)	0.984(14)	C(3)-C(2)-C(1)	109.03(7)
C(45)-H(45)	0.959(14)	C(17)-C(2)-C(1)	113.56(8)
C(46)-H(46A)	1.008(15)	C(3)-C(2)-H(2)	108.8(7)
C(46)-H(46B)	0.976(16)	C(17)-C(2)-H(2)	107.3(7)
C(46)-H(46C)	0.994(15)	C(1)-C(2)-H(2)	106.8(7)
C(47)-C(52)	1.3939(13)	C(9)-C(3)-C(4)	119.36(9)
C(47)-C(48)	1.3999(14)	C(9)-C(3)-C(2)	119.20(8)
C(48)-C(49)	1.3914(15)	C(4)-C(3)-C(2)	121.42(8)
C(48)-H(48)	0.956(14)	C(6)-C(4)-C(3)	119.49(9)
C(49)-C(50)	1.3914(16)	C(6)-C(4)-C(5)	120.28(8)
C(49)-H(49)	0.986(14)	C(3)-C(4)-C(5)	120.12(8)
C(50)-C(51)	1.3876(16)	O(1)-C(5)-C(23)	107.55(7)
C(50)-H(50)	0.975(15)	O(1)-C(5)-C(4)	111.53(7)
C(51)-C(52)	1.3931(14)	C(23)-C(5)-C(4)	113.59(8)
C(51)-H(51)	0.981(15)	O(1)-C(5)-H(5)	108.3(7)
C(52)-H(52)	0.988(13)	C(23)-C(5)-H(5)	108.8(7)
C(53)-C(58)	1.3946(14)	C(4)-C(5)-H(5)	106.9(7)
C(53)-C(54)	1.3972(14)	C(7)-C(6)-C(4)	120.60(9)
C(54)-C(55)	1.3904(14)	C(7)-C(6)-H(6)	119.4(8)
C(54)-H(54)	0.990(12)	C(4)-C(6)-H(6)	120.0(8)
C(55)-C(56)	1.3923(16)	C(6)-C(7)-C(8)	119.90(9)
C(55)-H(55)	0.974(14)	C(6)-C(7)-H(7)	120.8(8)
C(56)-C(57)	1.3894(16)	C(8)-C(7)-H(7)	119.3(8)
C(56)-H(56)	0.982(14)	C(9)-C(8)-C(7)	119.74(9)
C(57)-C(58)	1.3971(15)	C(9)-C(8)-H(8)	120.2(8)
C(57)-H(57)	0.972(15)	C(7)-C(8)-H(8)	120.0(8)
C(58)-H(58)	0.976(14)	C(8)-C(9)-C(3)	120.88(9)

C(8)-C(9)-H(9)	121.1(7)	C(19)-C(20)-C(21)	119.56(10)
C(3)-C(9)-H(9)	118.0(7)	C(19)-C(20)-H(20)	120.1(8)
C(15)-C(10)-C(11)	118.46(9)	C(21)-C(20)-H(20)	120.4(8)
C(15)-C(10)-C(1)	122.13(8)	C(20)-C(21)-C(22)	120.32(10)
C(11)-C(10)-C(1)	119.40(8)	C(20)-C(21)-H(21)	119.9(8)
C(12)-C(11)-C(10)	121.10(9)	C(22)-C(21)-H(21)	119.8(8)
C(12)-C(11)-H(11)	119.6(8)	C(21)-C(22)-C(17)	120.71(9)
C(10)-C(11)-H(11)	119.3(8)	C(21)-C(22)-H(22)	119.5(8)
C(11)-C(12)-C(13)	119.71(9)	C(17)-C(22)-H(22)	119.8(8)
C(11)-C(12)-H(12)	121.6(9)	C(24)-C(23)-C(28)	119.02(9)
C(13)-C(12)-H(12)	118.7(9)	C(24)-C(23)-C(5)	120.83(8)
O(13)-C(13)-C(14)	124.36(9)	C(28)-C(23)-C(5)	120.15(9)
O(13)-C(13)-C(12)	115.47(9)	C(25)-C(24)-C(23)	120.31(9)
C(14)-C(13)-C(12)	120.17(9)	C(25)-C(24)-H(24)	119.6(8)
C(13)-O(13)-C(16)	117.11(8)	C(23)-C(24)-H(24)	120.0(8)
C(13)-C(14)-C(15)	119.29(9)	C(26)-C(25)-C(24)	120.50(10)
C(13)-C(14)-H(14)	120.9(8)	C(26)-C(25)-H(25)	119.6(9)
C(15)-C(14)-H(14)	119.8(8)	C(24)-C(25)-H(25)	119.9(9)
C(10)-C(15)-C(14)	121.23(9)	C(27)-C(26)-C(25)	119.54(10)
C(10)-C(15)-H(15)	119.5(8)	C(27)-C(26)-H(26)	121.1(8)
C(14)-C(15)-H(15)	119.2(8)	C(25)-C(26)-H(26)	119.3(8)
O(13)-C(16)-H(16A)	109.1(9)	C(26)-C(27)-C(28)	120.18(9)
O(13)-C(16)-H(16B)	105.5(9)	C(26)-C(27)-H(27)	120.4(8)
H(16A)-C(16)-H(16B)	110.7(13)	C(28)-C(27)-H(27)	119.5(8)
O(13)-C(16)-H(16C)	111.1(9)	C(27)-C(28)-C(23)	120.43(9)
H(16A)-C(16)-H(16C)	111.2(13)	C(27)-C(28)-H(28)	120.6(8)
H(16B)-C(16)-H(16C)	109.1(12)	C(23)-C(28)-H(28)	118.9(8)
C(18)-C(17)-C(22)	118.31(9)	O(31)-C(31)-C(40)	107.15(7)
C(18)-C(17)-C(2)	120.00(8)	O(31)-C(31)-C(32)	109.62(7)
C(22)-C(17)-C(2)	121.70(8)	C(40)-C(31)-C(32)	113.98(8)
C(17)-C(18)-C(19)	120.91(10)	O(31)-C(31)-H(31)	108.4(7)
C(17)-C(18)-H(18)	118.7(9)	C(40)-C(31)-H(31)	109.9(7)
C(19)-C(18)-H(18)	120.3(8)	C(32)-C(31)-H(31)	107.6(7)
C(20)-C(19)-C(18)	120.18(10)	C(35)-O(31)-C(31)	112.89(7)
C(20)-C(19)-H(19)	120.5(9)	C(33)-C(32)-C(47)	112.25(8)
C(18)-C(19)-H(19)	119.3(9)	C(33)-C(32)-C(31)	109.10(8)

C(47)-C(32)-C(31)	112.31(8)	C(43)-C(42)-H(42)	118.1(9)
C(33)-C(32)-H(32)	108.9(7)	O(43)-C(43)-C(44)	124.60(9)
C(47)-C(32)-H(32)	107.5(7)	O(43)-C(43)-C(42)	115.60(9)
C(31)-C(32)-H(32)	106.6(7)	C(44)-C(43)-C(42)	119.80(9)
C(34)-C(33)-C(39)	118.79(9)	C(43)-O(43)-C(46)	116.81(9)
C(34)-C(33)-C(32)	121.58(8)	C(43)-C(44)-C(45)	119.36(9)
C(39)-C(33)-C(32)	119.61(9)	C(43)-C(44)-H(44)	120.8(8)
C(33)-C(34)-C(36)	119.83(9)	C(45)-C(44)-H(44)	119.8(8)
C(33)-C(34)-C(35)	120.58(8)	C(40)-C(45)-C(44)	121.41(9)
C(36)-C(34)-C(35)	119.60(8)	C(40)-C(45)-H(45)	118.4(8)
O(31)-C(35)-C(53)	106.56(7)	C(44)-C(45)-H(45)	120.1(8)
O(31)-C(35)-C(34)	111.45(8)	O(43)-C(46)-H(46A)	111.1(9)
C(53)-C(35)-C(34)	112.15(8)	O(43)-C(46)-H(46B)	105.0(9)
O(31)-C(35)-H(35)	108.9(7)	H(46A)-C(46)-H(46B)	112.0(12)
C(53)-C(35)-H(35)	109.5(7)	O(43)-C(46)-H(46C)	111.5(9)
C(34)-C(35)-H(35)	108.3(7)	H(46A)-C(46)-H(46C)	107.7(12)
C(37)-C(36)-C(34)	120.78(9)	H(46B)-C(46)-H(46C)	109.6(12)
C(37)-C(36)-H(36)	119.8(8)	C(52)-C(47)-C(48)	118.40(9)
C(34)-C(36)-H(36)	119.4(8)	C(52)-C(47)-C(32)	121.32(9)
C(36)-C(37)-C(38)	119.61(10)	C(48)-C(47)-C(32)	120.27(9)
C(36)-C(37)-H(37)	119.7(9)	C(49)-C(48)-C(47)	120.81(10)
C(38)-C(37)-H(37)	120.7(9)	C(49)-C(48)-H(48)	119.4(8)
C(39)-C(38)-C(37)	119.90(9)	C(47)-C(48)-H(48)	119.8(8)
C(39)-C(38)-H(38)	120.5(8)	C(50)-C(49)-C(48)	120.24(10)
C(37)-C(38)-H(38)	119.6(8)	C(50)-C(49)-H(49)	120.9(8)
C(38)-C(39)-C(33)	121.08(9)	C(48)-C(49)-H(49)	118.9(8)
C(38)-C(39)-H(39)	120.2(8)	C(51)-C(50)-C(49)	119.34(10)
C(33)-C(39)-H(39)	118.7(8)	C(51)-C(50)-H(50)	120.2(8)
C(45)-C(40)-C(41)	118.39(9)	C(49)-C(50)-H(50)	120.5(8)
C(45)-C(40)-C(31)	121.45(9)	C(50)-C(51)-C(52)	120.47(10)
C(41)-C(40)-C(31)	120.16(9)	C(50)-C(51)-H(51)	121.1(9)
C(42)-C(41)-C(40)	120.78(9)	C(52)-C(51)-H(51)	118.4(9)
C(42)-C(41)-H(41)	120.6(8)	C(51)-C(52)-C(47)	120.74(9)
C(40)-C(41)-H(41)	118.6(8)	C(51)-C(52)-H(52)	119.7(7)
C(41)-C(42)-C(43)	120.12(9)	C(47)-C(52)-H(52)	119.5(7)
C(41)-C(42)-H(42)	121.7(9)	C(58)-C(53)-C(54)	119.23(9)

C(58)-C(53)-C(35)	121.14(9)	C(57)-C(56)-H(56)	119.7(8)
C(54)-C(53)-C(35)	119.59(9)	C(55)-C(56)-H(56)	120.5(8)
C(55)-C(54)-C(53)	120.52(9)	C(56)-C(57)-C(58)	120.24(10)
C(55)-C(54)-H(54)	120.3(7)	C(56)-C(57)-H(57)	120.2(9)
C(53)-C(54)-H(54)	119.2(7)	C(58)-C(57)-H(57)	119.6(9)
C(54)-C(55)-C(56)	120.05(10)	C(53)-C(58)-C(57)	120.15(10)
C(54)-C(55)-H(55)	120.0(8)	C(53)-C(58)-H(58)	119.7(8)
C(56)-C(55)-H(55)	120.0(8)	C(57)-C(58)-H(58)	120.1(8)
C(57)-C(56)-C(55)	119.78(10)		

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x 10^3$) for JF2849FMI. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	14(1)	15(1)	18(1)	4(1)	5(1)	3(1)
O(1)	20(1)	14(1)	15(1)	3(1)	6(1)	4(1)
C(2)	14(1)	13(1)	18(1)	3(1)	5(1)	2(1)
C(3)	12(1)	15(1)	18(1)	2(1)	4(1)	2(1)
C(4)	14(1)	14(1)	18(1)	1(1)	3(1)	2(1)
C(5)	16(1)	15(1)	16(1)	3(1)	4(1)	5(1)
C(6)	22(1)	16(1)	18(1)	3(1)	4(1)	4(1)
C(7)	24(1)	19(1)	17(1)	1(1)	5(1)	4(1)
C(8)	21(1)	16(1)	22(1)	-1(1)	5(1)	4(1)
C(9)	16(1)	14(1)	22(1)	3(1)	4(1)	3(1)
C(10)	15(1)	16(1)	18(1)	4(1)	6(1)	6(1)
C(11)	18(1)	19(1)	22(1)	2(1)	7(1)	1(1)
C(12)	21(1)	23(1)	23(1)	6(1)	10(1)	0(1)
C(13)	22(1)	23(1)	17(1)	4(1)	6(1)	4(1)
O(13)	36(1)	32(1)	17(1)	1(1)	10(1)	-7(1)
C(14)	19(1)	20(1)	20(1)	3(1)	3(1)	1(1)
C(15)	17(1)	19(1)	20(1)	6(1)	6(1)	2(1)
C(16)	34(1)	35(1)	20(1)	-3(1)	7(1)	-3(1)
C(17)	15(1)	16(1)	17(1)	1(1)	5(1)	5(1)

C(18)	20(1)	27(1)	25(1)	11(1)	8(1)	5(1)
C(19)	25(1)	41(1)	26(1)	15(1)	6(1)	12(1)
C(20)	17(1)	37(1)	22(1)	-1(1)	2(1)	8(1)
C(21)	16(1)	20(1)	29(1)	-3(1)	7(1)	2(1)
C(22)	18(1)	14(1)	26(1)	3(1)	7(1)	4(1)
C(23)	19(1)	14(1)	16(1)	1(1)	6(1)	6(1)
C(24)	20(1)	19(1)	25(1)	9(1)	2(1)	5(1)
C(25)	21(1)	21(1)	32(1)	9(1)	2(1)	2(1)
C(26)	26(1)	17(1)	25(1)	7(1)	8(1)	6(1)
C(27)	28(1)	21(1)	21(1)	8(1)	2(1)	8(1)
C(28)	21(1)	18(1)	20(1)	3(1)	1(1)	5(1)
C(31)	14(1)	16(1)	21(1)	2(1)	6(1)	3(1)
O(31)	18(1)	15(1)	20(1)	2(1)	3(1)	4(1)
C(32)	15(1)	14(1)	20(1)	2(1)	5(1)	4(1)
C(33)	15(1)	17(1)	18(1)	1(1)	7(1)	4(1)
C(34)	16(1)	16(1)	18(1)	1(1)	8(1)	4(1)
C(35)	17(1)	17(1)	19(1)	3(1)	7(1)	3(1)
C(36)	24(1)	20(1)	18(1)	3(1)	7(1)	6(1)
C(37)	25(1)	23(1)	18(1)	-2(1)	3(1)	5(1)
C(38)	25(1)	18(1)	24(1)	-2(1)	6(1)	2(1)
C(39)	22(1)	16(1)	22(1)	2(1)	7(1)	4(1)
C(40)	15(1)	16(1)	21(1)	3(1)	6(1)	0(1)
C(41)	18(1)	21(1)	24(1)	-1(1)	6(1)	5(1)
C(42)	18(1)	26(1)	25(1)	2(1)	4(1)	7(1)
C(43)	20(1)	22(1)	18(1)	3(1)	6(1)	0(1)
O(43)	25(1)	30(1)	20(1)	-2(1)	2(1)	5(1)
C(44)	22(1)	20(1)	22(1)	2(1)	10(1)	4(1)
C(45)	17(1)	20(1)	23(1)	3(1)	7(1)	4(1)
C(46)	31(1)	27(1)	25(1)	-4(1)	6(1)	-1(1)
C(47)	16(1)	13(1)	19(1)	2(1)	4(1)	1(1)
C(48)	16(1)	19(1)	24(1)	6(1)	2(1)	2(1)
C(49)	26(1)	25(1)	20(1)	6(1)	3(1)	1(1)
C(50)	30(1)	25(1)	22(1)	2(1)	12(1)	3(1)
C(51)	23(1)	27(1)	27(1)	3(1)	10(1)	9(1)
C(52)	19(1)	20(1)	21(1)	4(1)	5(1)	6(1)
C(53)	17(1)	13(1)	21(1)	2(1)	4(1)	1(1)

C(54)	22(1)	15(1)	22(1)	2(1)	6(1)	3(1)
C(55)	23(1)	18(1)	29(1)	1(1)	9(1)	5(1)
C(56)	23(1)	16(1)	36(1)	6(1)	4(1)	5(1)
C(57)	27(1)	22(1)	30(1)	11(1)	5(1)	4(1)
C(58)	24(1)	21(1)	25(1)	7(1)	9(1)	4(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^{2}x 10 ^{3}) for JF2849FMI.

	x	y	z	U(eq)
H(1)	10011(13)	9697(11)	7770(5)	13(3)
H(2)	8697(13)	11302(11)	7669(5)	13(3)
H(5)	9931(13)	8474(11)	8570(5)	15(3)
H(6)	8590(14)	8965(13)	9650(6)	22(3)
H(7)	8146(14)	10885(12)	10178(6)	23(3)
H(8)	7755(15)	12622(13)	9631(6)	26(3)
H(9)	7731(14)	12400(12)	8530(6)	21(3)
H(11)	10508(15)	10556(13)	6833(6)	25(3)
H(12)	10293(16)	10128(14)	5722(7)	32(4)
H(14)	6884(15)	7121(13)	5560(6)	28(3)
H(15)	7065(15)	7567(13)	6657(6)	27(3)
H(16A)	8147(17)	6487(15)	4734(7)	37(4)
H(16B)	7986(18)	7222(15)	4116(8)	43(4)
H(16C)	6720(17)	7143(14)	4510(7)	37(4)
H(18)	6978(16)	11777(14)	6870(7)	31(3)
H(19)	4570(17)	11540(15)	6250(7)	38(4)
H(20)	2696(16)	9876(13)	6367(7)	31(4)
H(21)	3274(16)	8498(14)	7116(7)	30(3)
H(22)	5673(15)	8773(13)	7751(6)	25(3)
H(24)	6157(15)	6858(13)	8007(6)	28(3)
H(25)	5079(17)	4952(14)	8323(7)	36(4)
H(26)	6441(16)	4095(14)	9145(7)	31(3)
H(27)	8927(15)	5100(13)	9634(7)	31(4)

H(28)	10005(15)	6990(13)	9294(6)	28(3)
H(31)	10949(13)	5198(11)	2118(6)	16(3)
H(32)	9830(14)	3349(12)	2382(6)	21(3)
H(35)	9660(14)	6261(12)	1346(6)	24(3)
H(36)	6857(15)	5152(13)	497(6)	27(3)
H(37)	5442(16)	3059(14)	119(7)	34(4)
H(38)	5712(15)	1398(14)	744(6)	28(3)
H(39)	7426(14)	1856(13)	1740(6)	22(3)
H(41)	12781(15)	4682(14)	3002(7)	29(3)
H(42)	14328(16)	5516(14)	4036(7)	34(4)
H(44)	11479(15)	7883(13)	4370(6)	26(3)
H(45)	9979(15)	7037(13)	3351(6)	27(3)
H(46A)	13832(16)	9101(15)	4928(7)	37(4)
H(46B)	14763(17)	8632(14)	5553(7)	38(4)
H(46C)	12998(17)	8279(14)	5368(7)	34(4)
H(48)	10010(16)	3364(13)	3506(6)	29(3)
H(49)	8873(16)	3359(14)	4355(7)	32(4)
H(50)	6552(16)	4006(14)	4254(7)	32(4)
H(51)	5438(17)	4637(14)	3288(7)	35(4)
H(52)	6616(14)	4645(12)	2445(6)	22(3)
H(54)	7106(13)	6940(12)	2281(6)	19(3)
H(55)	5487(15)	8406(13)	2096(7)	29(3)
H(56)	5338(16)	9452(14)	1191(7)	31(4)
H(57)	6860(16)	9063(14)	489(7)	32(4)
H(58)	8486(15)	7614(13)	672(6)	27(3)

Table 6. Torsion angles [°] for JF2849FMI.

C(10)-C(1)-O(1)-C(5)	164.91(7)	C(1)-C(2)-C(3)-C(9)	161.19(8)
C(2)-C(1)-O(1)-C(5)	-70.46(9)	C(17)-C(2)-C(3)-C(4)	105.38(10)
O(1)-C(1)-C(2)-C(3)	50.72(10)	C(1)-C(2)-C(3)-C(4)	-20.48(11)
C(10)-C(1)-C(2)-C(3)	172.47(7)	C(9)-C(3)-C(4)-C(6)	0.82(14)
O(1)-C(1)-C(2)-C(17)	-73.67(10)	C(2)-C(3)-C(4)-C(6)	-177.51(8)
C(10)-C(1)-C(2)-C(17)	48.08(10)	C(9)-C(3)-C(4)-C(5)	-175.38(8)
C(17)-C(2)-C(3)-C(9)	-72.94(10)	C(2)-C(3)-C(4)-C(5)	6.30(13)

C(1)-O(1)-C(5)-C(23)	178.09(7)	C(18)-C(19)-C(20)-C(21)	0.25(17)
C(1)-O(1)-C(5)-C(4)	52.93(10)	C(19)-C(20)-C(21)-C(22)	0.26(16)
C(6)-C(4)-C(5)-O(1)	162.95(8)	C(20)-C(21)-C(22)-C(17)	-0.93(15)
C(3)-C(4)-C(5)-O(1)	-20.88(12)	C(18)-C(17)-C(22)-C(21)	1.05(14)
C(6)-C(4)-C(5)-C(23)	41.22(12)	C(2)-C(17)-C(22)-C(21)	-178.99(9)
C(3)-C(4)-C(5)-C(23)	-142.61(9)	O(1)-C(5)-C(23)-C(24)	-41.37(11)
C(3)-C(4)-C(6)-C(7)	-1.27(14)	C(4)-C(5)-C(23)-C(24)	82.55(11)
C(5)-C(4)-C(6)-C(7)	174.93(9)	O(1)-C(5)-C(23)-C(28)	139.62(9)
C(4)-C(6)-C(7)-C(8)	0.37(15)	C(4)-C(5)-C(23)-C(28)	-96.45(10)
C(6)-C(7)-C(8)-C(9)	0.97(15)	C(28)-C(23)-C(24)-C(25)	1.16(15)
C(7)-C(8)-C(9)-C(3)	-1.42(15)	C(5)-C(23)-C(24)-C(25)	-177.86(9)
C(4)-C(3)-C(9)-C(8)	0.52(14)	C(23)-C(24)-C(25)-C(26)	0.28(17)
C(2)-C(3)-C(9)-C(8)	178.88(9)	C(24)-C(25)-C(26)-C(27)	-1.05(17)
O(1)-C(1)-C(10)-C(15)	26.90(12)	C(25)-C(26)-C(27)-C(28)	0.38(16)
C(2)-C(1)-C(10)-C(15)	-95.62(11)	C(26)-C(27)-C(28)-C(23)	1.08(16)
O(1)-C(1)-C(10)-C(11)	-154.26(8)	C(24)-C(23)-C(28)-C(27)	-1.84(15)
C(2)-C(1)-C(10)-C(11)	83.22(11)	C(5)-C(23)-C(28)-C(27)	177.19(9)
C(15)-C(10)-C(11)-C(12)	-0.71(15)	C(40)-C(31)-O(31)-C(35)	165.67(7)
C(1)-C(10)-C(11)-C(12)	-179.59(9)	C(32)-C(31)-O(31)-C(35)	-70.17(9)
C(10)-C(11)-C(12)-C(13)	-1.00(16)	O(31)-C(31)-C(32)-C(33)	49.41(10)
C(11)-C(12)-C(13)-O(13)	-178.11(9)	C(40)-C(31)-C(32)-C(33)	169.48(8)
C(11)-C(12)-C(13)-C(14)	1.61(16)	O(31)-C(31)-C(32)-C(47)	-75.70(10)
C(14)-C(13)-O(13)-C(16)	-13.47(16)	C(40)-C(31)-C(32)-C(47)	44.37(11)
C(12)-C(13)-O(13)-C(16)	166.24(10)	C(47)-C(32)-C(33)-C(34)	108.11(10)
O(13)-C(13)-C(14)-C(15)	179.20(10)	C(31)-C(32)-C(33)-C(34)	-17.03(12)
C(12)-C(13)-C(14)-C(15)	-0.50(15)	C(47)-C(32)-C(33)-C(39)	-70.26(11)
C(11)-C(10)-C(15)-C(14)	1.85(14)	C(31)-C(32)-C(33)-C(39)	164.60(8)
C(1)-C(10)-C(15)-C(14)	-179.30(9)	C(39)-C(33)-C(34)-C(36)	-1.28(13)
C(13)-C(14)-C(15)-C(10)	-1.25(15)	C(32)-C(33)-C(34)-C(36)	-179.66(8)
C(3)-C(2)-C(17)-C(18)	128.24(9)	C(39)-C(33)-C(34)-C(35)	179.04(8)
C(1)-C(2)-C(17)-C(18)	-108.46(10)	C(32)-C(33)-C(34)-C(35)	0.66(13)
C(3)-C(2)-C(17)-C(22)	-51.71(11)	C(31)-O(31)-C(35)-C(53)	173.54(7)
C(1)-C(2)-C(17)-C(22)	71.59(11)	C(31)-O(31)-C(35)-C(34)	50.90(10)
C(22)-C(17)-C(18)-C(19)	-0.54(15)	C(33)-C(34)-C(35)-O(31)	-15.77(12)
C(2)-C(17)-C(18)-C(19)	179.50(10)	C(36)-C(34)-C(35)-O(31)	164.55(8)
C(17)-C(18)-C(19)-C(20)	-0.11(18)	C(33)-C(34)-C(35)-C(53)	-135.15(9)

C(36)-C(34)-C(35)-C(53)	45.17(12)	C(33)-C(32)-C(47)-C(52)	-31.26(12)
C(33)-C(34)-C(36)-C(37)	1.43(14)	C(31)-C(32)-C(47)-C(52)	92.10(11)
C(35)-C(34)-C(36)-C(37)	-178.89(9)	C(33)-C(32)-C(47)-C(48)	150.04(9)
C(34)-C(36)-C(37)-C(38)	-0.58(15)	C(31)-C(32)-C(47)-C(48)	-86.59(11)
C(36)-C(37)-C(38)-C(39)	-0.39(16)	C(52)-C(47)-C(48)-C(49)	-0.64(14)
C(37)-C(38)-C(39)-C(33)	0.52(15)	C(32)-C(47)-C(48)-C(49)	178.09(9)
C(34)-C(33)-C(39)-C(38)	0.32(14)	C(47)-C(48)-C(49)-C(50)	0.91(16)
C(32)-C(33)-C(39)-C(38)	178.73(9)	C(48)-C(49)-C(50)-C(51)	-0.43(16)
O(31)-C(31)-C(40)-C(45)	24.83(12)	C(49)-C(50)-C(51)-C(52)	-0.29(17)
C(32)-C(31)-C(40)-C(45)	-96.62(11)	C(50)-C(51)-C(52)-C(47)	0.56(16)
O(31)-C(31)-C(40)-C(41)	-155.36(9)	C(48)-C(47)-C(52)-C(51)	-0.09(15)
C(32)-C(31)-C(40)-C(41)	83.19(11)	C(32)-C(47)-C(52)-C(51)	-178.81(9)
C(45)-C(40)-C(41)-C(42)	-1.84(15)	O(31)-C(35)-C(53)-C(58)	139.94(9)
C(31)-C(40)-C(41)-C(42)	178.35(9)	C(34)-C(35)-C(53)-C(58)	-97.85(11)
C(40)-C(41)-C(42)-C(43)	-1.54(16)	O(31)-C(35)-C(53)-C(54)	-42.58(11)
C(41)-C(42)-C(43)-O(43)	-175.21(9)	C(34)-C(35)-C(53)-C(54)	79.62(11)
C(41)-C(42)-C(43)-C(44)	3.79(16)	C(58)-C(53)-C(54)-C(55)	1.71(15)
C(44)-C(43)-O(43)-C(46)	-12.78(15)	C(35)-C(53)-C(54)-C(55)	-175.81(9)
C(42)-C(43)-O(43)-C(46)	166.16(10)	C(53)-C(54)-C(55)-C(56)	-0.32(15)
O(43)-C(43)-C(44)-C(45)	176.30(9)	C(54)-C(55)-C(56)-C(57)	-0.87(16)
C(42)-C(43)-C(44)-C(45)	-2.60(15)	C(55)-C(56)-C(57)-C(58)	0.67(17)
C(41)-C(40)-C(45)-C(44)	3.05(15)	C(54)-C(53)-C(58)-C(57)	-1.91(15)
C(31)-C(40)-C(45)-C(44)	-177.15(9)	C(35)-C(53)-C(58)-C(57)	175.58(9)
C(43)-C(44)-C(45)-C(40)	-0.84(15)	C(56)-C(57)-C(58)-C(53)	0.73(16)

Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for JF2849FMI [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
C(12)-H(12)...O(13)#1	0.969(15)	2.472(15)	3.4189(13)	165.6(12)

Symmetry transformations used to generate equivalent atoms:

#1 -x+2,-y+2,-z+1

TETRAHYDROISOQUINOLINE 23c

Table 1. Crystal data and structure refinement for [C18 H17 N O2].

Identification code	JF2851CuFMI	
Empirical formula	C18 H17 N O2	
Formula weight	279.32	
Temperature	90(2) K	
Wavelength	1.54178 Å	
Crystal system	Orthorhombic	
Space group	$P2_12_12_1$	
Unit cell dimensions	a = 9.0262(4) Å	α= 90°.
	b = 10.7097(4) Å	β= 90°.
	c = 14.6781(6) Å	γ = 90°.
Volume	1418.90(10) $Å^3$	
Z	4	
Density (calculated)	1.308 Mg/m^3	
Absorption coefficient	0.679 mm^{-1}	
F(000)	592	
Crystal size	0.764 x 0.170 x 0.115 mm^3	
Crystal color and habit	Colorless Rod	
Diffractometer	Bruker APEX-II CCD	
Theta range for data collection	5.112 to 72.141°.	
Index ranges	-10<=h<=11, -13<=k<=13, -18<=l<=17	
Reflections collected	7991	
Independent reflections	2743 [R(int) = 0.0192]	
Observed reflections (I > 2sigma(I))	2718	
Completeness to theta = 67.679°	99.9 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.8416 and 0.7268	
Solution method	SHELXT (Sheldrick, 2014)	
Refinement method	SHELXL-2018/3 (Sheldrick, 2018) Full-matrix least-squares on F^2	
Data / restraints / parameters	2743 / 0 / 259	
Goodness-of-fit on F^2	1.065	
Final R indices [I>2sigma(I)]	R1 = 0.0262, wR2 = 0.0696	
R indices (all data)	R1 = 0.0263, wR2 = 0.0697	

Absolute structure parameters	Flack: 0.03(5), Parsons: 0.02(5), Hooft: 0.01(4)
Extinction coefficient	0.0028(5)
Largest diff. peak and hole	0.252 and -0.156 e.Å^{-3}

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^{2}x 10^3) for JF2851CUFMI. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	4352(2)	4795(2)	2561(1)	20(1)
N(1)	4914(1)	5433(1)	3279(1)	18(1)
O(1)	3344(1)	4045(1)	2633(1)	27(1)
C(2)	5906(2)	6058(2)	1605(1)	23(1)
O(2)	4935(1)	4991(1)	1724(1)	25(1)
C(3)	7035(2)	6077(1)	2359(1)	20(1)
C(4)	6239(2)	6233(1)	3261(1)	17(1)
C(5)	7234(2)	5947(1)	4090(1)	16(1)
C(6)	6339(2)	6123(1)	4952(1)	18(1)
C(7)	7008(2)	6565(1)	5746(1)	21(1)
C(8)	6194(2)	6717(1)	6539(1)	24(1)
C(9)	4690(2)	6414(1)	6545(1)	24(1)
C(10)	4016(2)	5996(1)	5755(1)	22(1)
C(11)	4830(2)	5847(1)	4952(1)	18(1)
C(12)	4048(2)	5342(1)	4124(1)	20(1)
C(13)	7998(2)	4682(1)	4067(1)	17(1)
C(14)	7227(2)	3561(1)	4164(1)	18(1)
C(15)	7961(2)	2418(1)	4145(1)	20(1)
C(16)	9491(2)	2379(2)	4038(1)	21(1)
C(17)	10279(2)	3485(2)	3955(1)	22(1)
C(18)	9536(2)	4626(2)	3965(1)	19(1)

Table 3. Bond lengths [Å] and angles [°] for JF2851CUFMI.

C(1)-O(1)	1.2180(19)	C(1)-N(1)	1.354(2)
C(1)-O(2)	1.3534(19)	N(1)-C(12)	1.4697(19)

N(1)-C(4)	1.4712(18)	C(18)-H(18)	0.95(2)
C(2)-O(2)	1.4511(19)		
C(2)-C(3)	1.505(2)	O(1)-C(1)-O(2)	118.09(14)
C(2)-H(2A)	0.98(2)	O(1)-C(1)-N(1)	123.04(14)
C(2)-H(2B)	0.98(2)	O(2)-C(1)-N(1)	118.86(13)
C(3)-C(4)	1.515(2)	C(1)-N(1)-C(12)	115.12(12)
C(3)-H(3A)	1.01(2)	C(1)-N(1)-C(4)	125.73(13)
C(3)-H(3B)	0.97(2)	C(12)-N(1)-C(4)	119.08(12)
C(4)-C(5)	1.544(2)	O(2)-C(2)-C(3)	109.33(12)
C(4)-H(4)	0.976(19)	O(2)-C(2)-H(2A)	108.9(11)
C(5)-C(6)	1.513(2)	C(3)-C(2)-H(2A)	110.2(11)
C(5)-C(13)	1.520(2)	O(2)-C(2)-H(2B)	106.3(12)
C(5)-H(5)	0.939(19)	C(3)-C(2)-H(2B)	111.1(12)
C(6)-C(11)	1.393(2)	H(2A)-C(2)-H(2B)	111.0(16)
C(6)-C(7)	1.396(2)	C(1)-O(2)-C(2)	117.80(12)
C(7)-C(8)	1.385(2)	C(2)-C(3)-C(4)	108.86(13)
C(7)-H(7)	0.97(2)	C(2)-C(3)-H(3A)	110.7(11)
C(8)-C(9)	1.396(3)	C(4)-C(3)-H(3A)	108.9(11)
C(8)-H(8)	0.99(2)	C(2)-C(3)-H(3B)	109.5(12)
C(9)-C(10)	1.384(2)	C(4)-C(3)-H(3B)	111.7(12)
C(9)-H(9)	1.00(2)	H(3A)-C(3)-H(3B)	107.1(15)
C(10)-C(11)	1.397(2)	N(1)-C(4)-C(3)	109.73(12)
C(10)-H(10)	0.96(2)	N(1)-C(4)-C(5)	110.07(11)
C(11)-C(12)	1.506(2)	C(3)-C(4)-C(5)	113.01(12)
C(12)-H(12A)	0.962(19)	N(1)-C(4)-H(4)	108.4(11)
C(12)-H(12B)	0.97(2)	C(3)-C(4)-H(4)	108.5(11)
C(13)-C(14)	1.395(2)	C(5)-C(4)-H(4)	107.0(11)
C(13)-C(18)	1.398(2)	C(6)-C(5)-C(13)	111.83(12)
C(14)-C(15)	1.391(2)	C(6)-C(5)-C(4)	108.86(12)
C(14)-H(14)	0.93(2)	C(13)-C(5)-C(4)	115.04(12)
C(15)-C(16)	1.391(2)	C(6)-C(5)-H(5)	108.4(11)
C(15)-H(15)	1.00(2)	C(13)-C(5)-H(5)	106.7(11)
C(16)-C(17)	1.387(2)	C(4)-C(5)-H(5)	105.6(11)
C(16)-H(16)	0.98(2)	C(11)-C(6)-C(7)	119.64(14)
C(17)-C(18)	1.393(2)	C(11)-C(6)-C(5)	119.76(13)
C(17)-H(17)	0.96(2)	C(7)-C(6)-C(5)	120.60(14)

C(8)-C(7)-C(6)	120.76(15)	H(12A)-C(12)-H(12B)	106.6(16)
C(8)-C(7)-H(7)	120.6(12)	C(14)-C(13)-C(18)	118.00(14)
C(6)-C(7)-H(7)	118.7(12)	C(14)-C(13)-C(5)	122.59(12)
C(7)-C(8)-C(9)	119.58(15)	C(18)-C(13)-C(5)	119.40(14)
C(7)-C(8)-H(8)	118.7(13)	C(15)-C(14)-C(13)	121.14(13)
C(9)-C(8)-H(8)	121.7(13)	C(15)-C(14)-H(14)	118.3(12)
C(10)-C(9)-C(8)	119.86(14)	C(13)-C(14)-H(14)	120.4(12)
C(10)-C(9)-H(9)	118.2(11)	C(16)-C(15)-C(14)	120.14(14)
C(8)-C(9)-H(9)	121.9(11)	C(16)-C(15)-H(15)	120.3(12)
C(9)-C(10)-C(11)	120.77(15)	C(14)-C(15)-H(15)	119.5(12)
C(9)-C(10)-H(10)	118.7(12)	C(17)-C(16)-C(15)	119.51(15)
C(11)-C(10)-H(10)	120.5(12)	C(17)-C(16)-H(16)	120.3(12)
C(6)-C(11)-C(10)	119.37(14)	C(15)-C(16)-H(16)	120.2(12)
C(6)-C(11)-C(12)	122.25(13)	C(16)-C(17)-C(18)	120.10(13)
C(10)-C(11)-C(12)	118.33(14)	C(16)-C(17)-H(17)	120.3(12)
N(1)-C(12)-C(11)	114.12(12)	C(18)-C(17)-H(17)	119.6(12)
N(1)-C(12)-H(12A)	107.1(11)	C(17)-C(18)-C(13)	121.11(14)
C(11)-C(12)-H(12A)	109.5(11)	C(17)-C(18)-H(18)	120.6(12)
N(1)-C(12)-H(12B)	109.5(11)	C(13)-C(18)-H(18)	118.3(12)
C(11)-C(12)-H(12B)	109.8(11)		

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x 10^3$) for JF2851CUFMI. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	18(1)	19(1)	23(1)	-2(1)	-1(1)	2(1)
N(1)	18(1)	17(1)	19(1)	0(1)	0(1)	-1(1)
O(1)	25(1)	26(1)	32(1)	-6(1)	0(1)	-7(1)
C(2)	28(1)	24(1)	17(1)	1(1)	1(1)	0(1)
O(2)	28(1)	27(1)	20(1)	-4(1)	-1(1)	-4(1)
C(3)	22(1)	19(1)	19(1)	1(1)	3(1)	0(1)
C(4)	17(1)	14(1)	19(1)	1(1)	1(1)	-2(1)
C(5)	17(1)	15(1)	18(1)	0(1)	1(1)	-3(1)

C(6)	22(1)	12(1)	19(1)	1(1)	2(1)	2(1)
C(7)	25(1)	18(1)	22(1)	1(1)	-2(1)	3(1)
C(8)	33(1)	20(1)	19(1)	-1(1)	-2(1)	5(1)
C(9)	34(1)	19(1)	19(1)	2(1)	6(1)	9(1)
C(10)	23(1)	16(1)	26(1)	3(1)	5(1)	5(1)
C(11)	22(1)	12(1)	21(1)	3(1)	2(1)	3(1)
C(12)	16(1)	21(1)	23(1)	0(1)	4(1)	0(1)
C(13)	18(1)	19(1)	13(1)	0(1)	0(1)	-1(1)
C(14)	17(1)	19(1)	19(1)	2(1)	1(1)	-1(1)
C(15)	22(1)	19(1)	21(1)	2(1)	0(1)	-2(1)
C(16)	23(1)	22(1)	19(1)	2(1)	-1(1)	4(1)
C(17)	16(1)	28(1)	21(1)	1(1)	-1(1)	3(1)
C(18)	18(1)	21(1)	18(1)	0(1)	1(1)	-3(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for JF2851CUFMI.

	x	y	z	U(eq)
H(2A)	5310(20)	6821(18)	1619(13)	23(5)
H(2B)	6380(20)	5951(19)	1011(14)	26(5)
H(3A)	7740(20)	6792(19)	2276(13)	21(5)
H(3B)	7610(20)	5320(20)	2342(14)	25(5)
H(4)	5920(20)	7100(18)	3317(12)	19(4)
H(5)	7990(20)	6552(17)	4078(13)	17(4)
H(7)	8060(20)	6760(20)	5735(14)	27(5)
H(8)	6700(20)	7030(20)	7090(15)	33(5)
H(9)	4080(20)	6480(18)	7108(14)	21(4)
H(10)	2990(20)	5790(18)	5772(13)	22(5)
H(12A)	3820(20)	4474(18)	4216(12)	17(4)
H(12B)	3110(20)	5766(18)	4046(13)	20(4)
H(14)	6220(20)	3563(17)	4279(13)	24(5)
H(15)	7380(20)	1630(20)	4191(14)	28(5)
H(16)	10010(20)	1571(18)	4033(13)	24(5)

H(17)	11340(20)	3471(18)	3906(13)	24(5)
H(18)	10070(20)	5390(19)	3916(12)	24(5)

Table 6. Torsion angles [°] for JF2851CUFMI.

O(1)-C(1)-N(1)-C(12)	-11.1(2)	C(7)-C(8)-C(9)-C(10)	-1.7(2)
O(2)-C(1)-N(1)-C(12)	169.20(12)	C(8)-C(9)-C(10)-C(11)	1.4(2)
O(1)-C(1)-N(1)-C(4)	172.15(14)	C(7)-C(6)-C(11)-C(10)	-1.2(2)
O(2)-C(1)-N(1)-C(4)	-7.6(2)	C(5)-C(6)-C(11)-C(10)	179.10(13)
O(1)-C(1)-O(2)-C(2)	167.33(14)	C(7)-C(6)-C(11)-C(12)	-178.71(14)
N(1)-C(1)-O(2)-C(2)	-12.9(2)	C(5)-C(6)-C(11)-C(12)	1.6(2)
C(3)-C(2)-O(2)-C(1)	48.10(18)	C(9)-C(10)-C(11)-C(6)	0.1(2)
O(2)-C(2)-C(3)-C(4)	-62.50(15)	C(9)-C(10)-C(11)-C(12)	177.65(13)
C(1)-N(1)-C(4)-C(3)	-9.14(19)	C(1)-N(1)-C(12)-C(11)	168.95(12)
C(12)-N(1)-C(4)-C(3)	174.18(12)	C(4)-N(1)-C(12)-C(11)	-14.03(18)
C(1)-N(1)-C(4)-C(5)	-134.12(14)	C(6)-C(11)-C(12)-N(1)	-13.0(2)
C(12)-N(1)-C(4)-C(5)	49.21(16)	C(10)-C(11)-C(12)-N(1)	169.48(12)
C(2)-C(3)-C(4)-N(1)	42.77(15)	C(6)-C(5)-C(13)-C(14)	55.33(18)
C(2)-C(3)-C(4)-C(5)	166.03(12)	C(4)-C(5)-C(13)-C(14)	-69.53(18)
N(1)-C(4)-C(5)-C(6)	-56.69(14)	C(6)-C(5)-C(13)-C(18)	-123.24(15)
C(3)-C(4)-C(5)-C(6)	-179.76(12)	C(4)-C(5)-C(13)-C(18)	111.90(15)
N(1)-C(4)-C(5)-C(13)	69.70(15)	C(18)-C(13)-C(14)-C(15)	-1.1(2)
C(3)-C(4)-C(5)-C(13)	-53.37(16)	C(5)-C(13)-C(14)-C(15)	-179.69(14)
C(13)-C(5)-C(6)-C(11)	-94.97(15)	C(13)-C(14)-C(15)-C(16)	0.8(2)
C(4)-C(5)-C(6)-C(11)	33.25(17)	C(14)-C(15)-C(16)-C(17)	0.2(2)
C(13)-C(5)-C(6)-C(7)	85.36(16)	C(15)-C(16)-C(17)-C(18)	-0.9(2)
C(4)-C(5)-C(6)-C(7)	-146.42(13)	C(16)-C(17)-C(18)-C(13)	0.6(2)
C(11)-C(6)-C(7)-C(8)	0.9(2)	C(14)-C(13)-C(18)-C(17)	0.4(2)
C(5)-C(6)-C(7)-C(8)	-179.40(13)	C(5)-C(13)-C(18)-C(17)	179.02(13)
C(6)-C(7)-C(8)-C(9)	0.5(2)		

Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for JF2851CUFMI [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
C(3)-H(3A)...O(1)#1	1.01(2)	2.61(2)	3.1964(19)	117.2(13)
C(4)-H(4)...O(1)#1	0.976(19)	2.592(19)	3.3063(18)	130.2(14)

Symmetry transformations used to generate equivalent atoms:

#1 -x+1,y+1/2,-z+1/2

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Experimental Details

HPLC Sample Preparation: Approximately 4-5 mgs of the compound to be analyzed was weighed into a vial. The sample was dissolved in a mixture of 10% isopropanol/hexanes solution such that the concentration was 1 mg/mL. The solution was mixed vigorously using a combination of sonication and vortexing. About 2 mL of the solution was taken up in a syringe, which was then fitted with a filter prior to loading a 1.5 mL HPLC vial with the sample.

HPLC Methods and Materials: Enantiomer ratios were determined by chiral HPLC with one of the following columns (denoted as AD, AS, or OD on individual HPLC traces): AD = Phenomenex Lux®, 5µm Amylose-1 LC Column, 250x4.6mm, part #: 00G-4732-E0; AS = Daicel Chiralpak®, AS-H 5µm, 250x4.6mm, column #: ASH0CE-LF002; OD = Phenomenex Lux®, 5µm Cellulose-1 LC Column, 250x4.6mm, part #: 00G-4459-E0. Isocratic solvent mixture (hexanes: isopropanol), flow rate, injection volume, and analysis-wavelength are denoted on individual HPLC traces. Note: unless otherwise specified, 1mL/min is the default HPLC method flow-rate.

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==== HPLC Analysis Report ====

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Sample ID : LNII-162A
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Vail # : 45
Injection Volume : 5 uL
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Method File Name : OD-98_2_Hexane_IPA_25min.lcm
Batch File Name : 6.23.17.lcb
Report File Name : UCD Default.lcr
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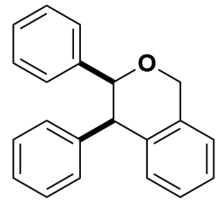
Data Processed : 6/23/2017 1:06:09 PM



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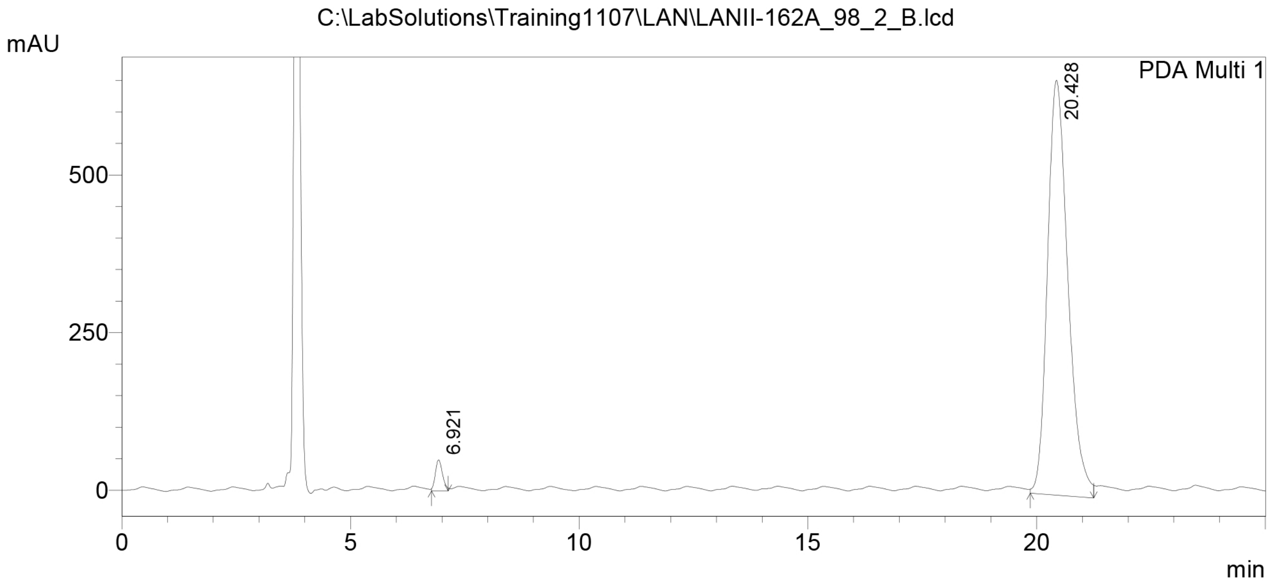

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Peak#	Ret. Time	Area	Height	Area %	Height %
1	6.921	540858	49287	2.570	6.963
2	20.428	20502736	658547	97.430	93.037
Total		21043594	707835	100.000	100.000

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==== HPLC Analysis Report ====

C:\LabSolutions\Training1107\LAN\LANII-163A_98_2_B.lcd

Acquired by : Admin
Sample Name : LNII-163A
Sample ID : LNII-163A
Tray# : 1
Vail # : 44
Injection Volume : 5 uL
Data File Name : LANII-163A_98_2_B.lcd
Method File Name : OD-98_2_Hexane_IPA_25min.lcm
Batch File Name : 6.23.17.lcb
Report File Name : UCD Default.lcr
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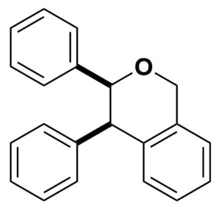

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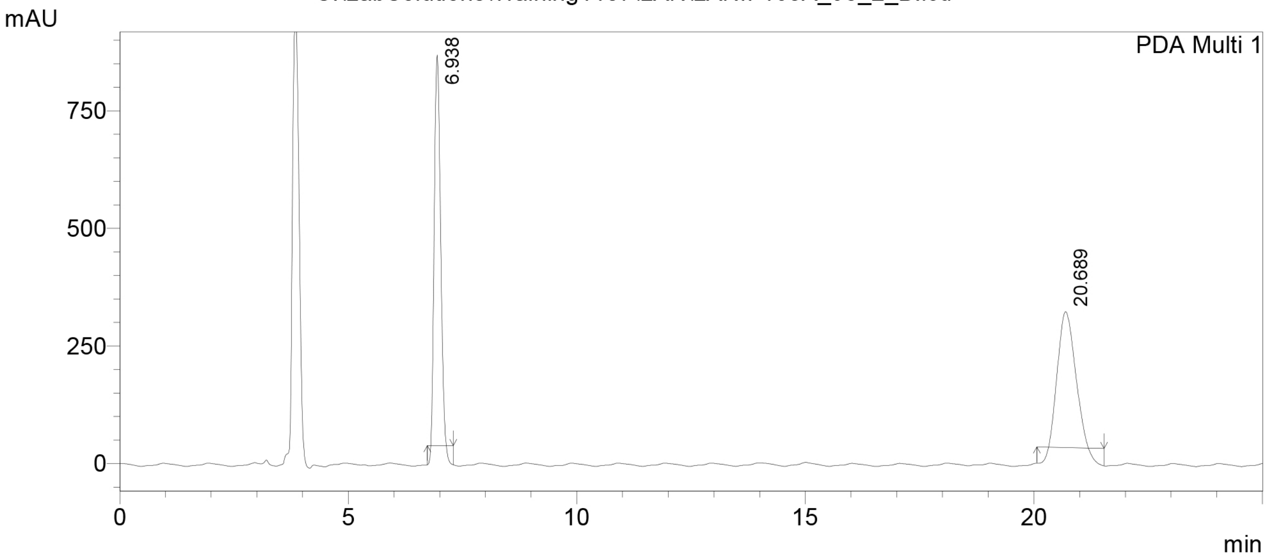

1 PDA Multi 1/214nm 4nm

< Peak Table >

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PDA Ch1 214nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	6.938	7393048	830123	52.208	74.184
2	20.689	6767795	288881	47.792	25.816
Total		14160843	1119004	100.000	100.000

6/23/2017 1 / 1 C:\LabSolutions\Training1107\LAN\LANII-163A_98_2_B.lcd

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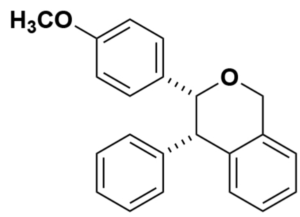
==== HPLC Analysis Report ====

C:\LabSolutions\Training1107\Ben B\BDB_I_033_2.lcd

Acquired by	: Admin
Sample Name	: 1
Sample ID	: 1
Tray#	: 1
Vail #	: 92
Injection Volume	: 20 uL
Data File Name	: BDB_I_033_2.lcd
Method File Name	: OD-98_2_Hexane_IPA_35min.lcm
Batch File Name	: 10JUN2018.lcb
Report File Name	: UCD Default.lcr
Data Acquired	: 6/10/2018 11:22:26 AM
Data Processed	: 6/10/2018 11:57:29 AM



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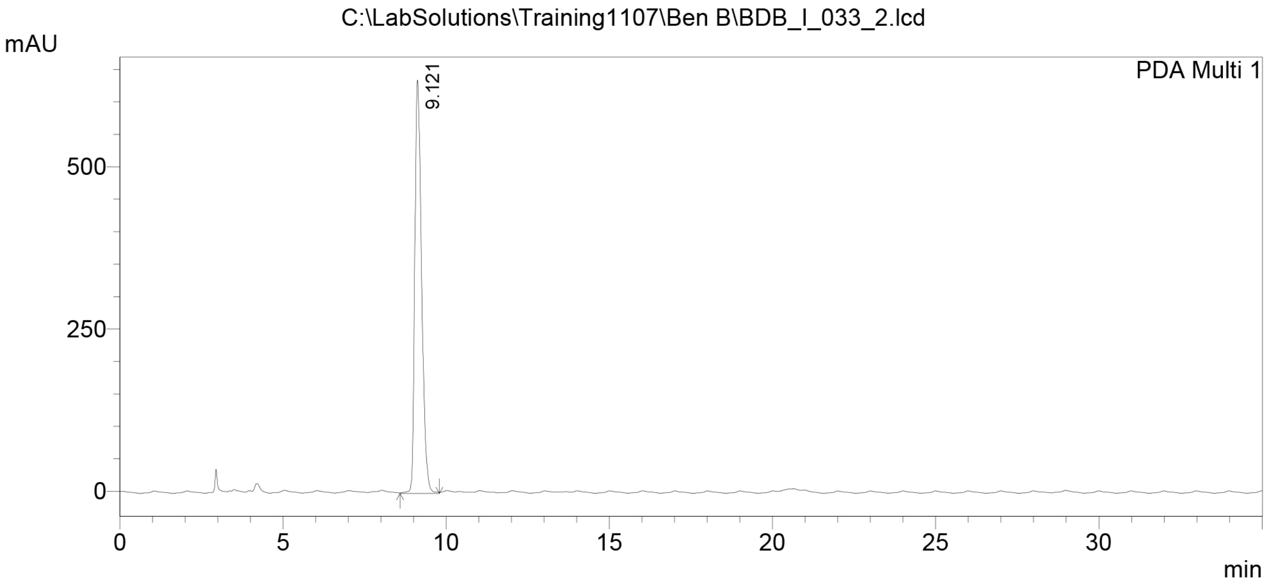

1 PDA Multi 1/224nm 4nm

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Peak#	Ret. Time	Area	Height	Area %	Height %
1	9.121	9280631	636863	100.000	100.000
Total		9280631	636863	100.000	100.000

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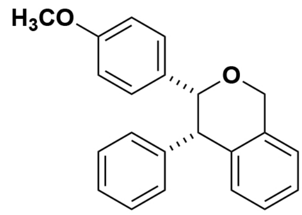
==== HPLC Analysis Report ====

C:\LabSolutions\Training1107\Ben B\BDB_I_011_2.lcd

Acquired by : Admin
Sample Name : 1
Sample ID : 1
Tray# : 1
Vail # : 91
Injection Volume : 20 uL
Data File Name : BDB_I_011_2.lcd
Method File Name : OD-98_2_Hexane_IPA_35min.lcm
Batch File Name : 10JUN2018.lcb
Report File Name : UCD Default.lcr
Data Acquired : 6/10/2018 10:46:59 AM
Data Processed : 6/10/2018 11:22:03 AM



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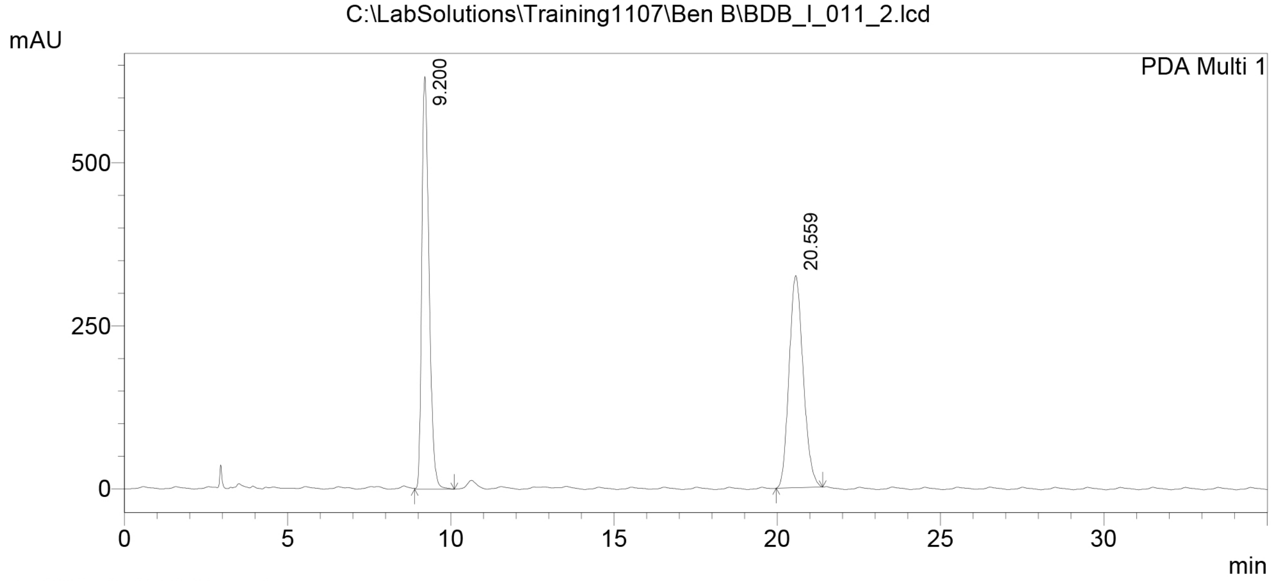

1 PDA Multi 1/224nm 4nm

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PDA Ch1 224nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	9.200	9807717	633380	50.002	66.057
2	20.559	9806928	325458	49.998	33.943
Total		19614645	958839	100.000	100.000

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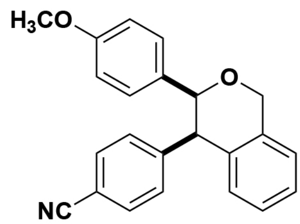
==== HPLC Analysis Report ====

C:\LabSolutions\Training1107\Jack Shiue\JS_I_010_AS_98_2_0.8mL_20min.lcd

Acquired by	: Admin
Sample Name	: 1
Sample ID	: 1
Tray#	: 1
Vail #	: 33
Injection Volume	: 2 uL
Data File Name	: JS_I_010_AS_98_2_0.8mL_20min.lcd
Method File Name	: AS-80_20_Hexane_IPA_20min_0.8ml.lcm
Batch File Name	: 20FEB2019_1.lcb
Report File Name	: UCD Default.lcr
Data Acquired	: 2/21/2019 12:45:27 PM
Data Processed	: 2/21/2019 1:55:28 PM



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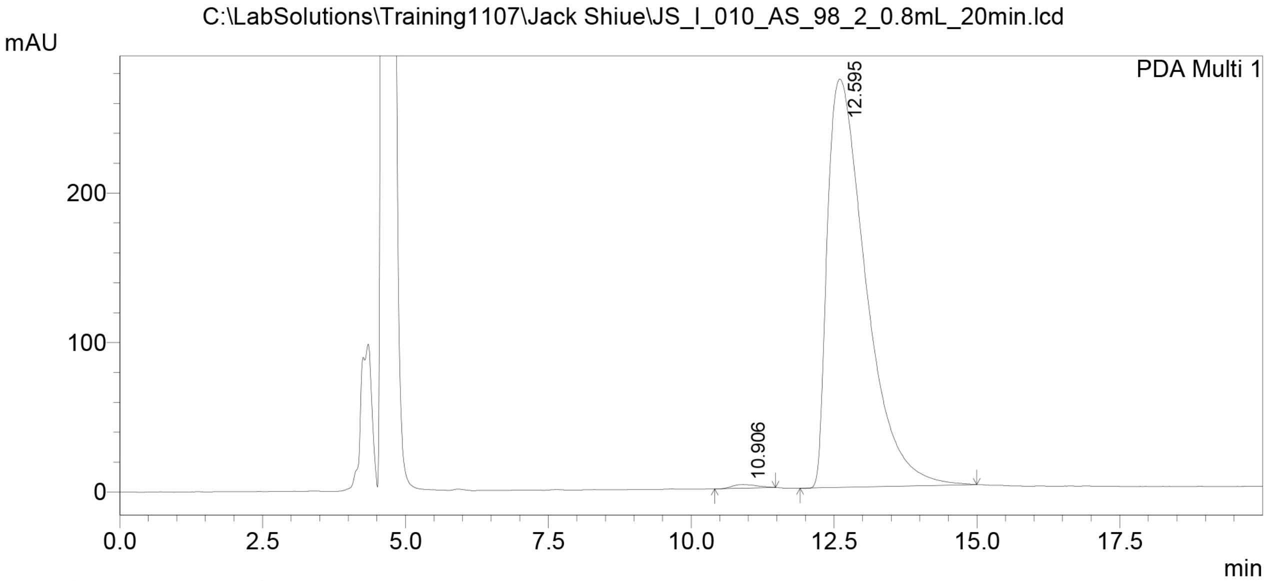

1 PDA Multi 1/205nm 4nm

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PDA Ch1 205nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	10.906	73366	2518	0.571	0.913
2	12.595	12784238	273261	99.429	99.087
Total		12857604	275779	100.000	100.000

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==== HPLC Analysis Report ====

C:\LabSolutions\Training1107\Jack Shiue\JS_I_011_AS_98_2_0.8mL_20min.lcd

Acquired by	: Admin
Sample Name	: 1
Sample ID	: 1
Tray#	: 1
Vail #	: 4
Injection Volume	: 2 uL
Data File Name	: JS_I_011_AS_98_2_0.8mL_20min.lcd
Method File Name	: AS-80_20_Hexane_IPA_20min_0.8ml.lcm
Batch File Name	: 20FEB2019_1.lcb
Report File Name	: UCD Default.lcr
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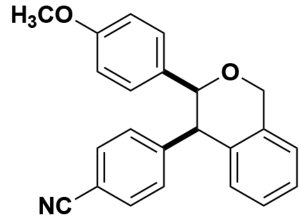
Data Processed	: 2/20/2019 10:57:38 AM



11c
racemic

<Chromatogram>

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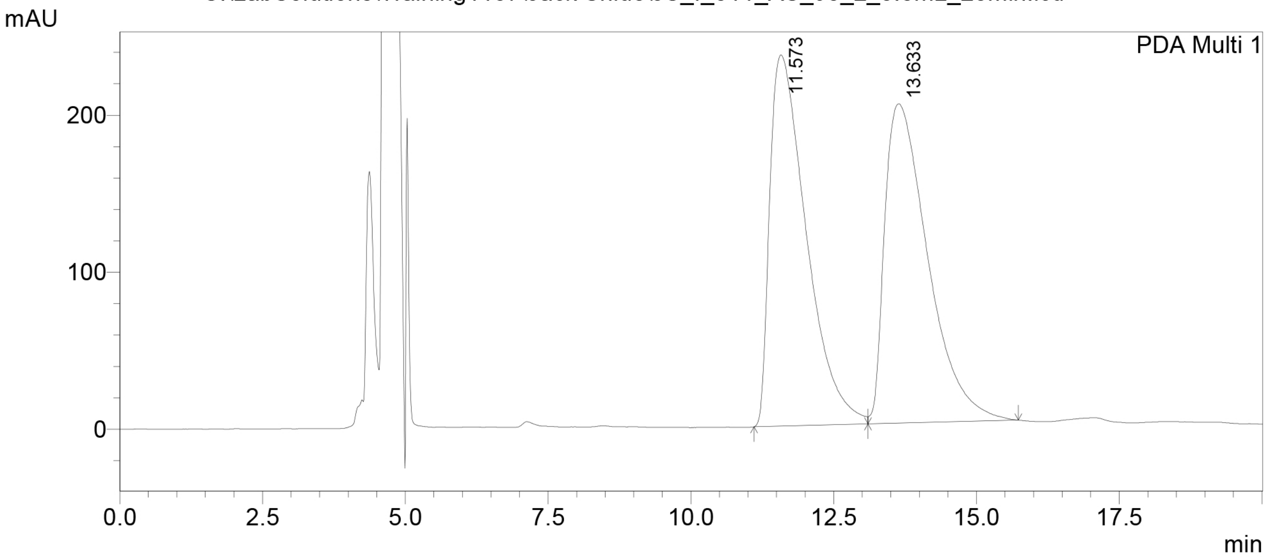

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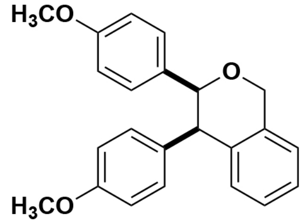
PDA Ch1 204nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	11.573	10549823	236414	49.610	53.745
2	13.633	10715667	203464	50.390	46.255
Total		21265490	439878	100.000	100.000

==== HPLC Analysis Report ====

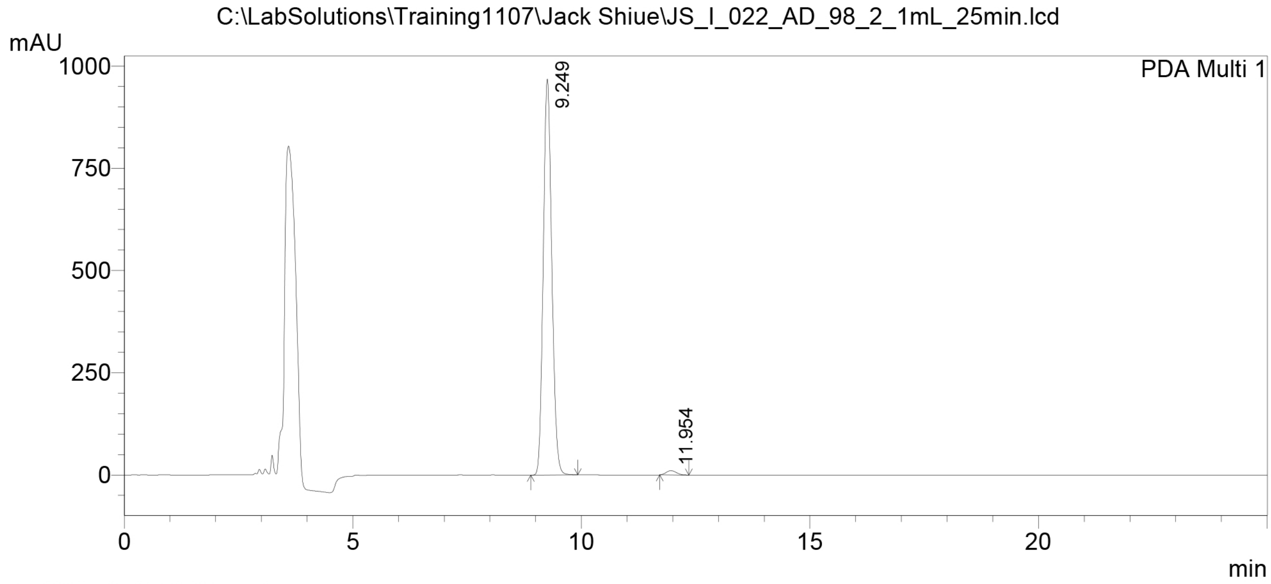
C:\LabSolutions\Training1107\Jack Shiue\JS_I_022_AD_98_2_1mL_25min.lcd

Acquired by : Admin
Sample Name : 1
Sample ID : 1
Tray# : 1
Vail # : 22
Injection Volume : 2 uL
Data File Name : JS_I_022_AD_98_2_1mL_25min.lcd
Method File Name : AD-80_20_Hexane_IPA_25min.lcm
Batch File Name : 19FEB2019_1.lcb
Report File Name : UCD Default.lcr
Data Acquired : 4/7/2019 2:50:45 PM
Data Processed : 4/7/2019 3:26:13 PM



11d
chiral

<Chromatogram>



1 PDA Multi 1/205nm 4nm

< Peak Table >

PeakTable C:\LabSolutions\Training1107\Jack Shiue\JS_I_022_AD_98_2_1mL_25min.lcd
PDA Ch1 205nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	9.249	12566488	968228	98.706	98.917
2	11.954	164696	10605	1.294	1.083
Total		12731183	978832	100.000	100.000

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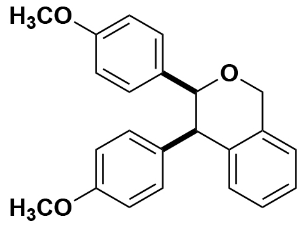
==== HPLC Analysis Report ====

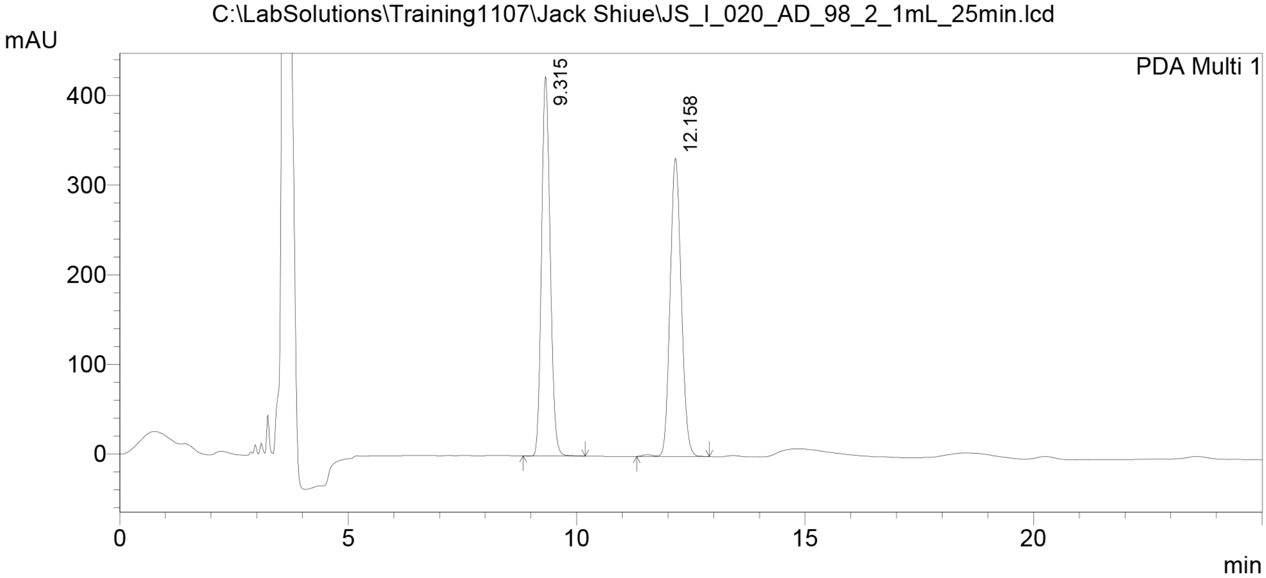
C:\LabSolutions\Training1107\Jack Shiue\JS_I_020_AD_98_2_1mL_25min.lcd

Acquired by : Admin
Sample Name : 1
Sample ID : 1
Tray# : 1
Vail # : 22
Injection Volume : 2 uL
Data File Name : JS_I_020_AD_98_2_1mL_25min.lcd
Method File Name : AD-80_20_Hexane_IPA_25min.lcm
Batch File Name : 19FEB2019_1.lcb
Report File Name : UCD Default.lcr
Data Acquired : 4/7/2019 1:03:58 PM
Data Processed : 4/7/2019 1:29:01 PM



11d
racemic

<Chromatogram>



1 PDA Multi 1/204nm 4nm

< Peak Table >

PeakTable C:\LabSolutions\Training1107\Jack Shiue\JS_I_020_AD_98_2_1mL_25min.lcd
PDA Ch1 204nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	9.315	5576576	423824	49.652	55.999
2	12.158	5654798	333012	50.348	44.001
Total		11231374	756836	100.000	100.000

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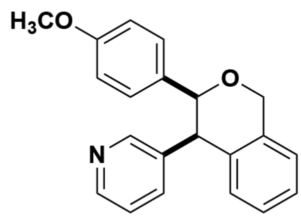
==== HPLC Analysis Report ====

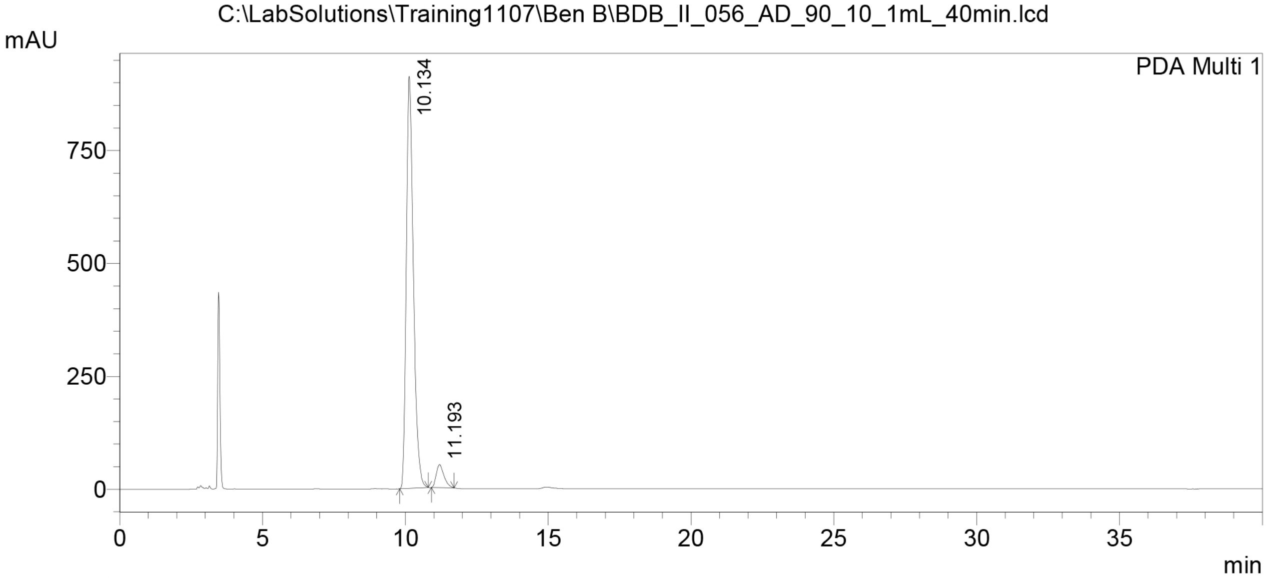
C:\LabSolutions\Training1107\Ben B\BDB_II_056_AD_90_10_1mL_40min.lcd

Acquired by	: Admin
Sample Name	: 1
Sample ID	: 1
Tray#	: 1
Vail #	: 66
Injection Volume	: 3 uL
Data File Name	: BDB_II_056_AD_90_10_1mL_40min.lcd
Method File Name	: AD-100%IPA-40min.lcm
Batch File Name	: 04APR2019.lcb
Report File Name	: UCD Default.lcr
Data Acquired	: 4/3/2019 7:46:29 PM
Data Processed	: 4/3/2019 8:26:33 PM



11e
chiral

<Chromatogram>



1 PDA Multi 1/206nm 4nm

< Peak Table >

PeakTable C:\LabSolutions\Training1107\Ben B\BDB_II_056_AD_90_10_1mL_40min.lcd

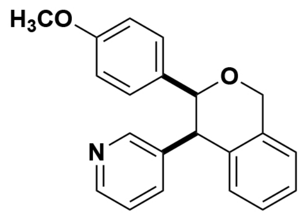
PDA Ch1 206nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	10.134	15740198	912013	94.260	94.689
2	11.193	958460	51150	5.740	5.311
Total		16698658	963163	100.000	100.000

==== HPLC Analysis Report ====

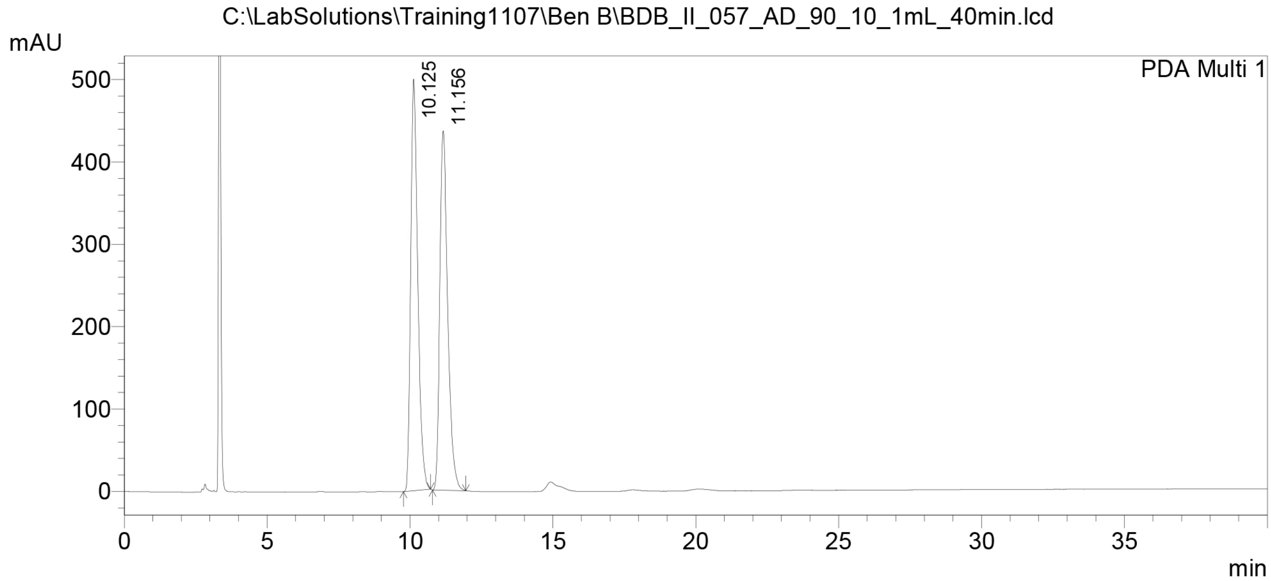
C:\LabSolutions\Training1107\Ben B\BDB_II_057_AD_90_10_1mL_40min.lcd

Acquired by	: Admin
Sample Name	: 1
Sample ID	: 1
Tray#	: 1
Vail #	: 67
Injection Volume	: 3 uL
Data File Name	: BDB_II_057_AD_90_10_1mL_40min.lcd
Method File Name	: AD-100%IPA-40min.lcm
Batch File Name	: 04APR2019.lcb
Report File Name	: UCD Default.lcr
Data Acquired	: 4/3/2019 7:06:04 PM
Data Processed	: 4/3/2019 7:46:07 PM



11e
racemic

<Chromatogram>



1 PDA Multi 1/206nm 4nm

< Peak Table >

PeakTable C:\LabSolutions\Training1107\Ben B\BDB_II_057_AD_90_10_1mL_40min.lcd

PDA Ch1 206nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	10.125	8594930	499964	50.607	53.391
2	11.156	8388726	436456	49.393	46.609
Total		16983656	936421	100.000	100.000

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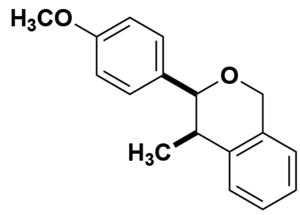
==== HPLC Analysis Report ====

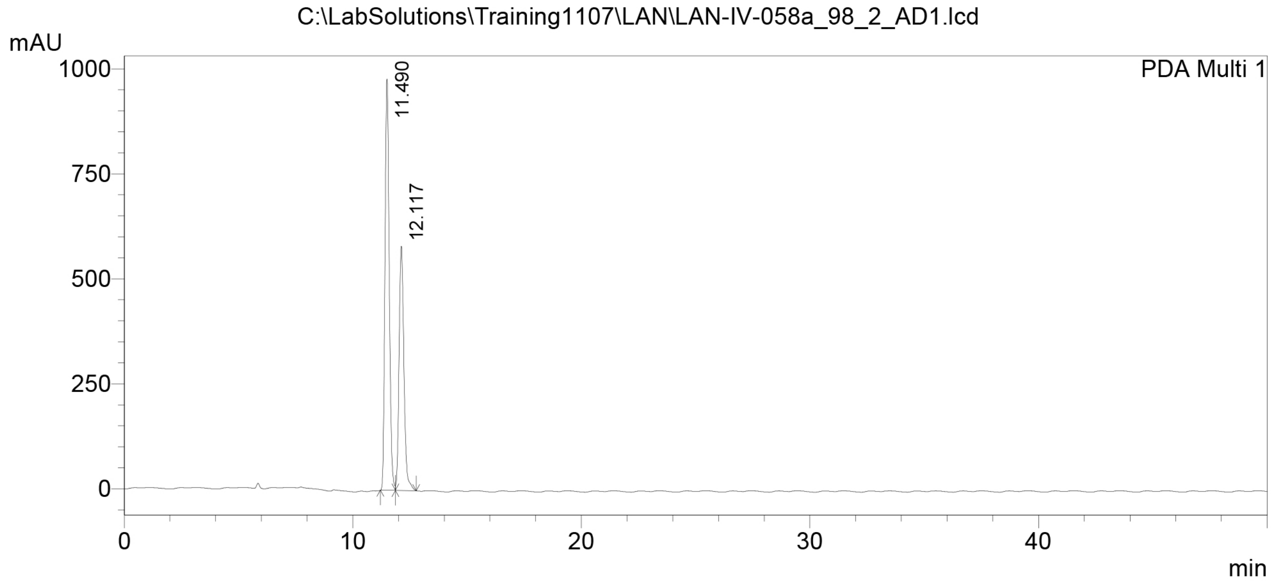
C:\LabSolutions\Training1107\LAN\LAN-IV-058a_98_2_AD1.lcd

Acquired by : Admin
Sample Name : LAN-IV-062A
Sample ID : LAN-IV-062A
Tray# : 1
Vail # : 76
Injection Volume : 10 uL
Data File Name : LAN-IV-058a_98_2_AD1.lcd
Method File Name : AD-98_2_Hexane_IPA_50min-0.5ml.lcm
Batch File Name : 10.24.18_2.lcb
Report File Name : UCD Default.lcr
Data Acquired : 10/24/2018 5:59:53 PM
Data Processed : 10/25/2018 11:15:08 AM



11f
chiral

<Chromatogram>



1 PDA Multi 1/223nm 4nm

< Peak Table >

PeakTable C:\LabSolutions\Training1107\LAN\LAN-IV-058a_98_2_AD1.lcd
PDA Ch1 223nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	11.490	12679851	978653	59.677	62.717
2	12.117	8567717	581764	40.323	37.283
Total		21247568	1560417	100.000	100.000

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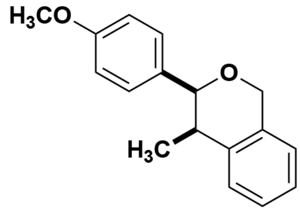
==== HPLC Analysis Report ====

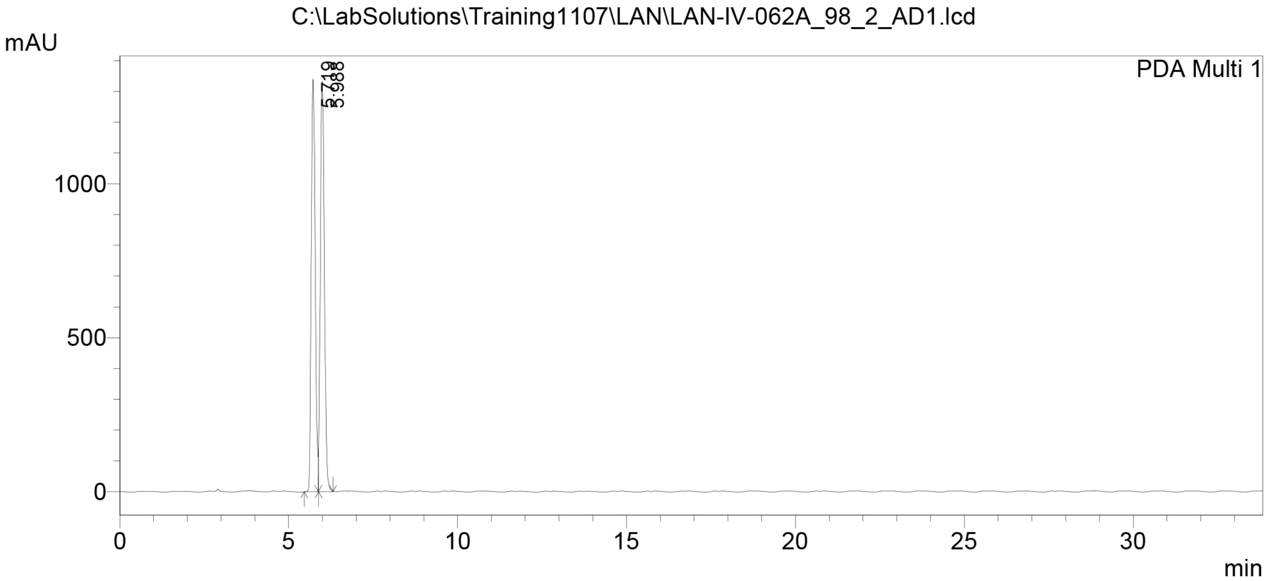
C:\LabSolutions\Training1107\LAN\LAN-IV-062A_98_2_AD1.lcd

Acquired by	: Admin
Sample Name	: LAN-IV-062A
Sample ID	: LAN-IV-062A
Tray#	: 1
Vail #	: 92
Injection Volume	: 10 uL
Data File Name	: LAN-IV-062A_98_2_AD1.lcd
Method File Name	: AD-98_2_Hexane_IPA_60min.lcm
Batch File Name	: 10.24.18.lcb
Report File Name	: UCD Default.lcr
Data Acquired	: 10/24/2018 3:22:10 PM
Data Processed	: 10/24/2018 3:56:02 PM



11f
racemic

<Chromatogram>



1 PDA Multi 1/223nm 4nm

< Peak Table >

PeakTable C:\LabSolutions\Training1107\LAN\LAN-IV-062A_98_2_AD1.lcd

PDA Ch1 223nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	5.719	11006329	1340656	49.815	50.139
2	5.988	11088090	1333208	50.185	49.861
Total		22094419	2673864	100.000	100.000

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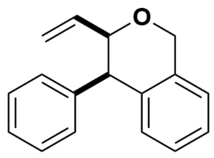
==== HPLC Analysis Report ====

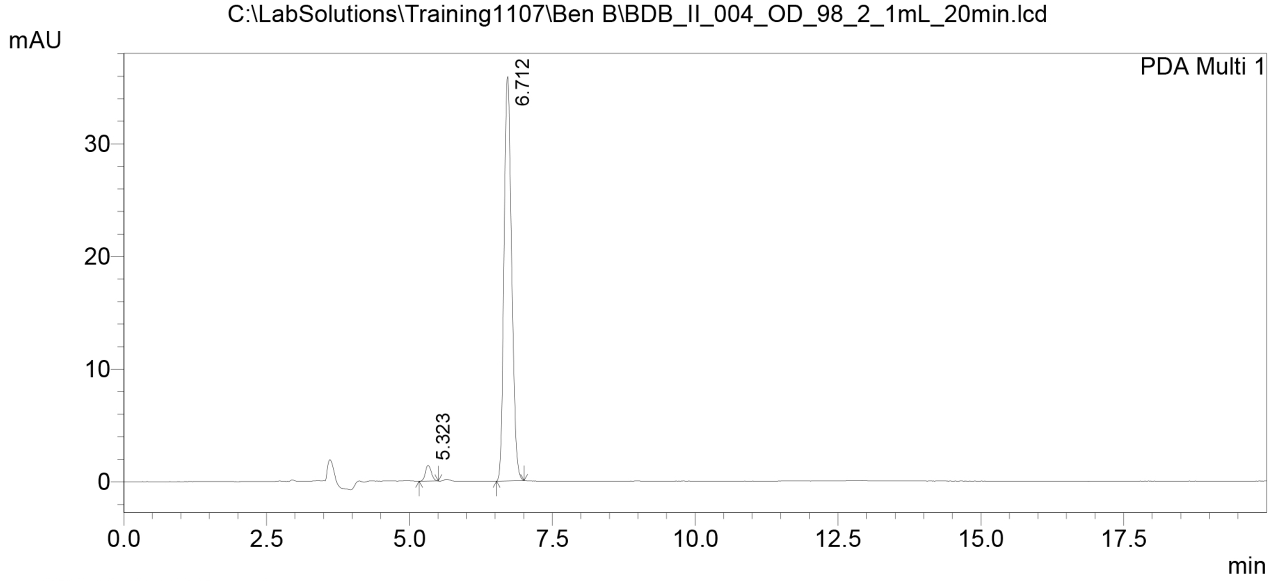
C:\LabSolutions\Training1107\Ben B\BDB_II_004_OD_98_2_1mL_20min.lcd

Acquired by : Admin
Sample Name : 1
Sample ID : 1
Tray# : 1
Vail # : 92
Injection Volume : 2 uL
Data File Name : BDB_II_004_OD_98_2_1mL_20min.lcd
Method File Name : OD-100_0_Hexane_IPA_20min.lcm
Batch File Name : 21DEC2018_3.lcb
Report File Name : UCD Default.lcr
Data Acquired : 12/21/2018 4:07:21 PM
Data Processed : 12/21/2018 4:27:22 PM



13a
chiral

<Chromatogram>



1 PDA Multi 1/254nm 4nm

< Peak Table >

PeakTable C:\LabSolutions\Training1107\Ben B\BDB_II_004_OD_98_2_1mL_20min.lcd
PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	5.323	10193	1390	2.975	3.730
2	6.712	332390	35888	97.025	96.270
Total		342583	37278	100.000	100.000

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==== HPLC Analysis Report ====

C:\LabSolutions\Training1107\Ben B\BDB_II_005_OD_98_2_1mL_20min.lcd

Acquired by	: Admin
Sample Name	: 1
Sample ID	: 1
Tray#	: 1
Vail #	: 91
Injection Volume	: 2 uL
Data File Name	: BDB_II_005_OD_98_2_1mL_20min.lcd
Method File Name	: OD-100_0_Hexane_IPA_20min.lcm
Batch File Name	: 21DEC2018_3.lcb
Report File Name	: UCD Default.lcr
Data Acquired	: 12/21/2018 3:46:53 PM
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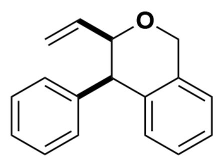
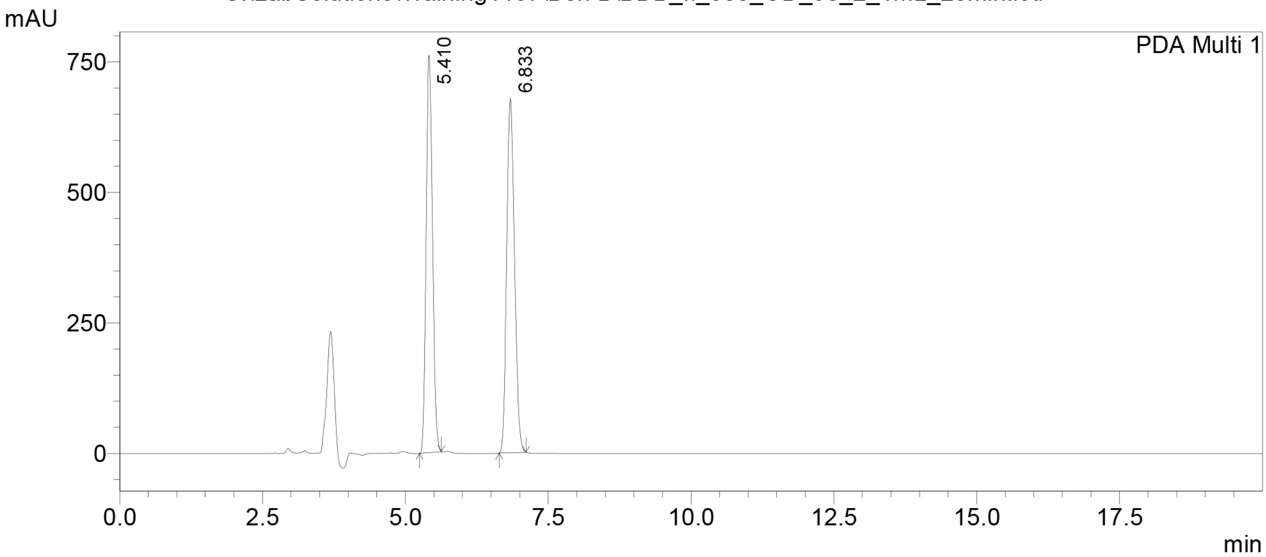


13a
racemic

<Chromatogram>

C:\LabSolutions\Training1107\Ben B\BDB_II_005_OD_98_2_1mL_20min.lcd



1 PDA Multi 1/206nm 4nm

< Peak Table >

PeakTable C:\LabSolutions\Training1107\Ben B\BDB_II_005_OD_98_2_1mL_20min.lcd

PDA Ch1 206nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	5.410	5880365	761664	47.575	52.851
2	6.833	6479833	679479	52.425	47.149
Total		12360198	1441144	100.000	100.000

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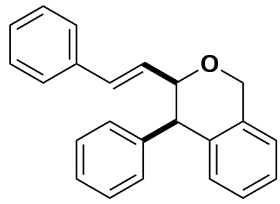
==== HPLC Analysis Report ====

C:\LabSolutions\Training1107\Ben B\BDB_II_018_AD_98_2_0.5mL_20min.lcd

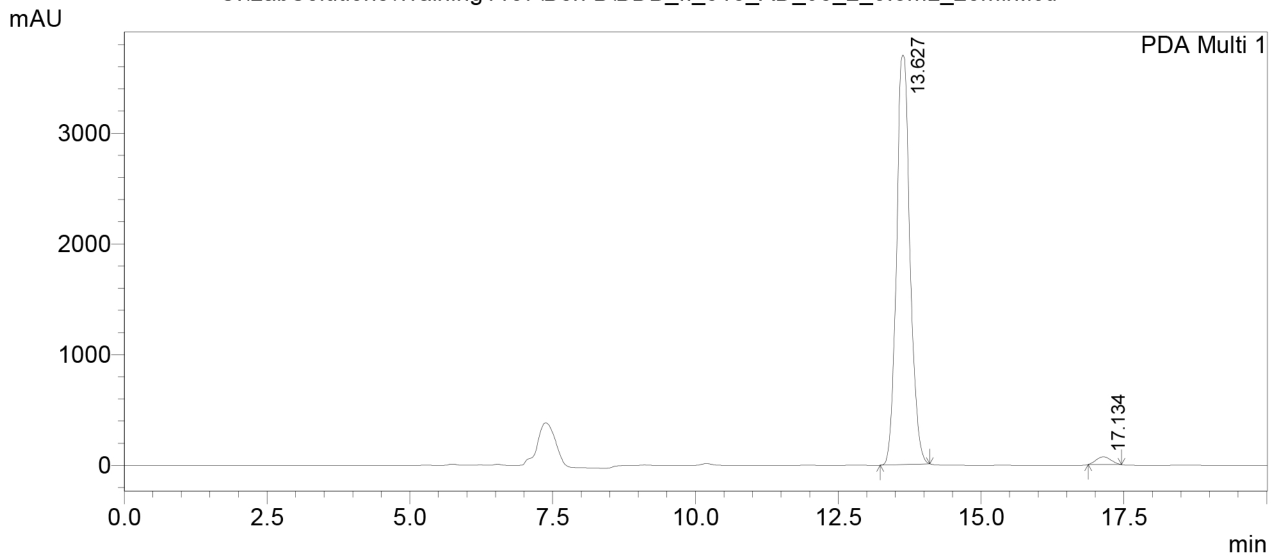
Acquired by : Admin
Sample Name : 1
Sample ID : 1
Tray# : 1
Vail # : 98
Injection Volume : 4 uL
Data File Name : BDB_II_018_AD_98_2_0.5mL_20min.lcd
Method File Name : AD-100%_Hexanes_20min_0.5mL.lcm
Batch File Name : 16JAN2019.lcb
Report File Name : UCD Default.lcr
Data Acquired : 1/16/2019 6:41:50 PM
Data Processed : 1/16/2019 7:01:52 PM



13b
chiral

<Chromatogram>

C:\LabSolutions\Training1107\Ben B\BDB_II_018_AD_98_2_0.5mL_20min.lcd



1 PDA Multi 1/205nm 4nm

< Peak Table >

PeakTable C:\LabSolutions\Training1107\Ben B\BDB_II_018_AD_98_2_0.5mL_20min.lcd
PDA Ch1 205nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	13.627	60649099	3698079	97.986	98.137
2	17.134	1246615	70216	2.014	1.863
Total		61895714	3768295	100.000	100.000

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==== HPLC Analysis Report ====

C:\LabSolutions\Training1107\Ben B\BDB_II_019_AD_98_2_0.5mL_20min.lcd

Acquired by : Admin
Sample Name : 1
Sample ID : 1
Tray# : 1
Vail # : 99
Injection Volume : 4 uL
Data File Name : BDB_II_019_AD_98_2_0.5mL_20min.lcd
Method File Name : AD-100%_Hexanes_20min_0.5mL.lcm
Batch File Name : 16JAN2019.lcb
Report File Name : UCD Default.lcr
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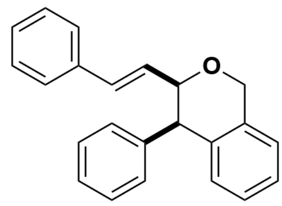
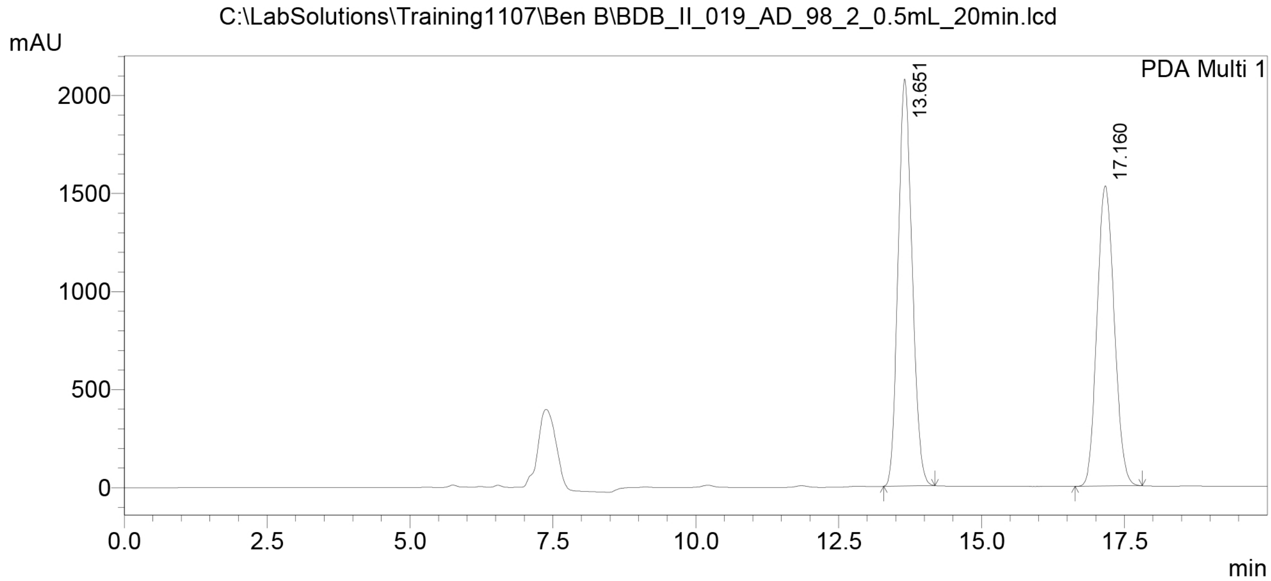
Data Processed : 1/16/2019 6:41:29 PM



13b
racemic

<Chromatogram>



1 PDA Multi 1/205nm 4nm

< Peak Table >

PeakTable C:\LabSolutions\Training1107\Ben B\BDB_II_019_AD_98_2_0.5mL_20min.lcd
PDA Ch1 205nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	13.651	35348389	2075808	52.335	57.542
2	17.160	32194431	1531650	47.665	42.458
Total		67542820	3607458	100.000	100.000

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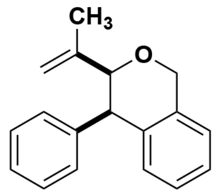
==== HPLC Analysis Report ====

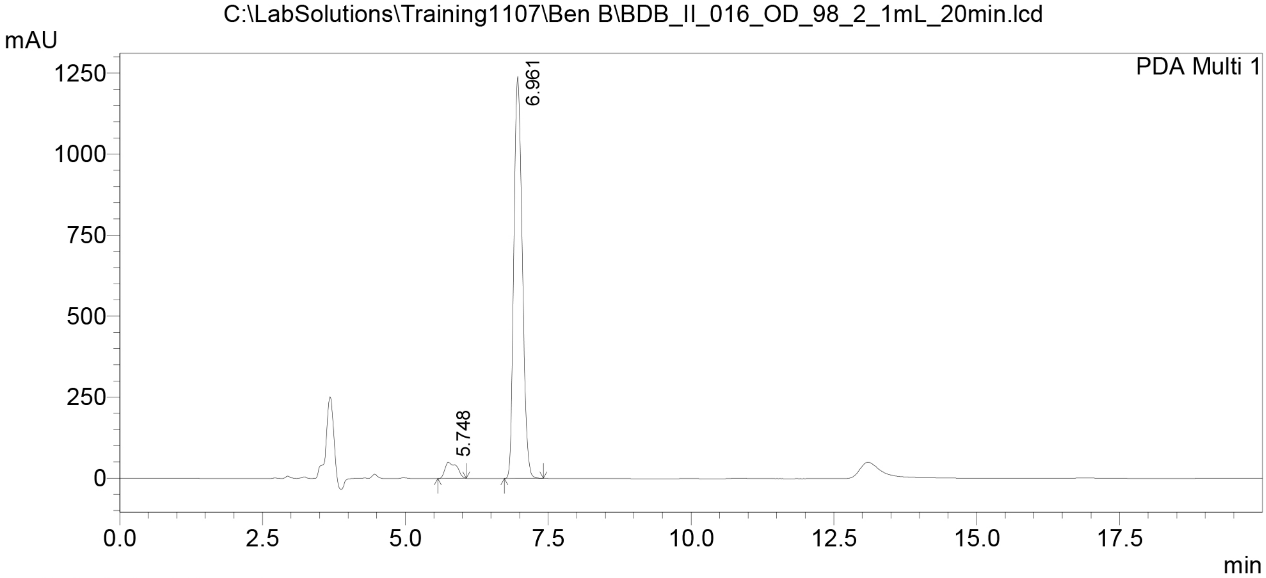
C:\LabSolutions\Training1107\Ben B\BDB_II_016_OD_98_2_1mL_20min.lcd

Acquired by	: Admin
Sample Name	: 1
Sample ID	: 1
Tray#	: 1
Vail #	: 96
Injection Volume	: 2 uL
Data File Name	: BDB_II_016_OD_98_2_1mL_20min.lcd
Method File Name	: OD-100_0_Hexane_IPA_20min.lcm
Batch File Name	: 17JAN2019_2.lcb
Report File Name	: UCD Default.lcr
Data Acquired	: 1/17/2019 11:48:34 AM
Data Processed	: 1/17/2019 12:08:37 PM



13c
chiral

<Chromatogram>



1 PDA Multi 1/205nm 4nm

< Peak Table >

PeakTable C:\LabSolutions\Training1107\Ben B\BDB_II_016_OD_98_2_1mL_20min.lcd

PDA Ch1 205nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	5.748	727354	49891	5.400	3.865
2	6.961	12743099	1241006	94.600	96.135
Total		13470453	1290897	100.000	100.000

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==== HPLC Analysis Report ====

C:\LabSolutions\Training1107\Ben B\BDB_II_017_OD_98_2_1mL_20min.lcd

Acquired by	: Admin
Sample Name	: 1
Sample ID	: 1
Tray#	: 1
Vail #	: 97
Injection Volume	: 2 uL
Data File Name	: BDB_II_017_OD_98_2_1mL_20min.lcd
Method File Name	: OD-100_0_Hexane_IPA_20min.lcm
Batch File Name	: 17JAN2019.lcb
Report File Name	: UCD Default.lcr
Data Acquired	: 1/17/2019 10:57:29 AM
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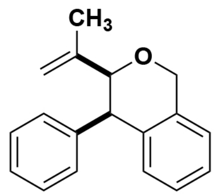
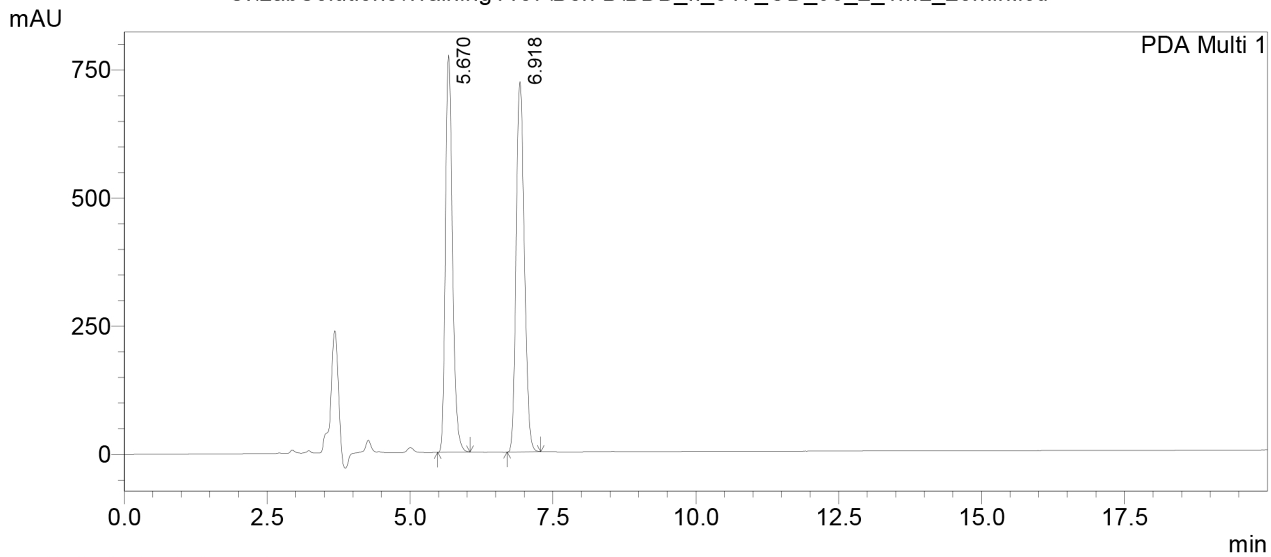


13c
racemic

<Chromatogram>

C:\LabSolutions\Training1107\Ben B\BDB_II_017_OD_98_2_1mL_20min.lcd



1 PDA Multi 1/205nm 4nm

< Peak Table >

PeakTable C:\LabSolutions\Training1107\Ben B\BDB_II_017_OD_98_2_1mL_20min.lcd
PDA Ch1 205nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	5.670	6625039	775188	47.905	51.750
2	6.918	7204425	722749	52.095	48.250
Total		13829465	1497937	100.000	100.000

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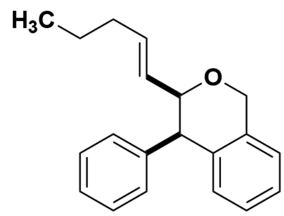
==== HPLC Analysis Report ====

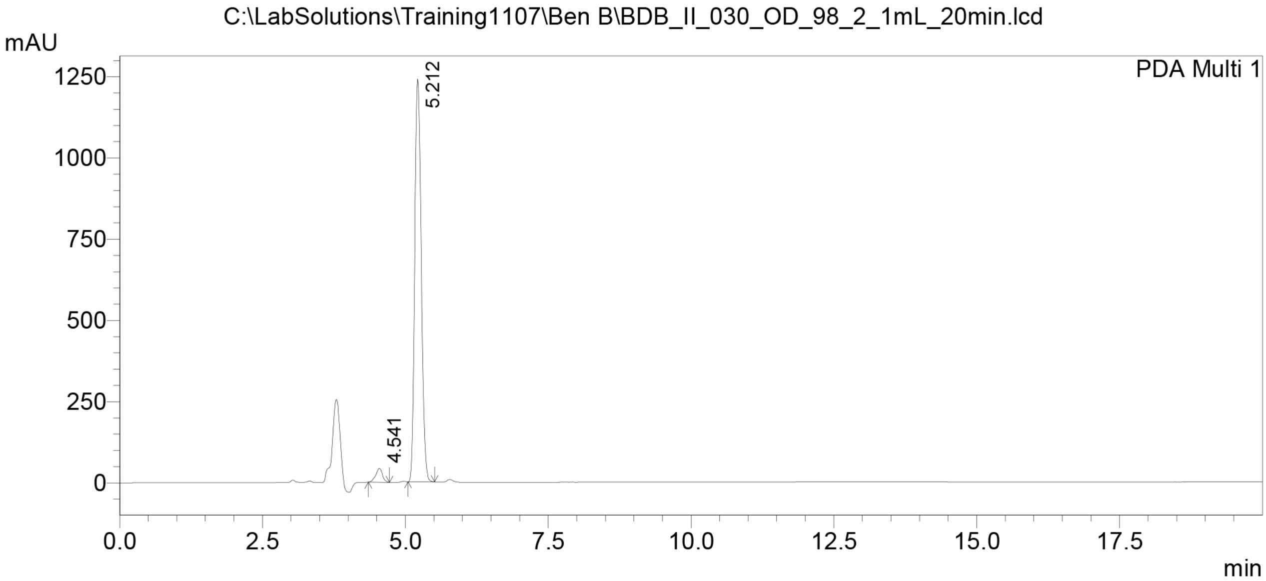
C:\LabSolutions\Training1107\Ben B\BDB_II_030_OD_98_2_1mL_20min.lcd

Acquired by	: Admin
Sample Name	: 1
Sample ID	: 1
Tray#	: 1
Vail #	: 93
Injection Volume	: 2 uL
Data File Name	: BDB_II_030_OD_98_2_1mL_20min.lcd
Method File Name	: OD-100_0_Hexane_IPA_20min.lcm
Batch File Name	: 24JAN2019_2.lcb
Report File Name	: UCD Default.lcr
Data Acquired	: 1/24/2019 1:21:35 PM
Data Processed	: 1/24/2019 1:41:38 PM



13d
chiral

<Chromatogram>



1 PDA Multi 1/205nm 4nm

< Peak Table >

PeakTable C:\LabSolutions\Training1107\Ben B\BDB_II_030_OD_98_2_1mL_20min.lcd
PDA Ch1 205nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	4.541	348015	42837	3.427	3.339
2	5.212	9806386	1240053	96.573	96.661
Total		10154401	1282890	100.000	100.000

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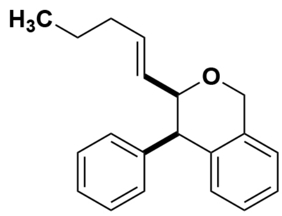
==== HPLC Analysis Report ====

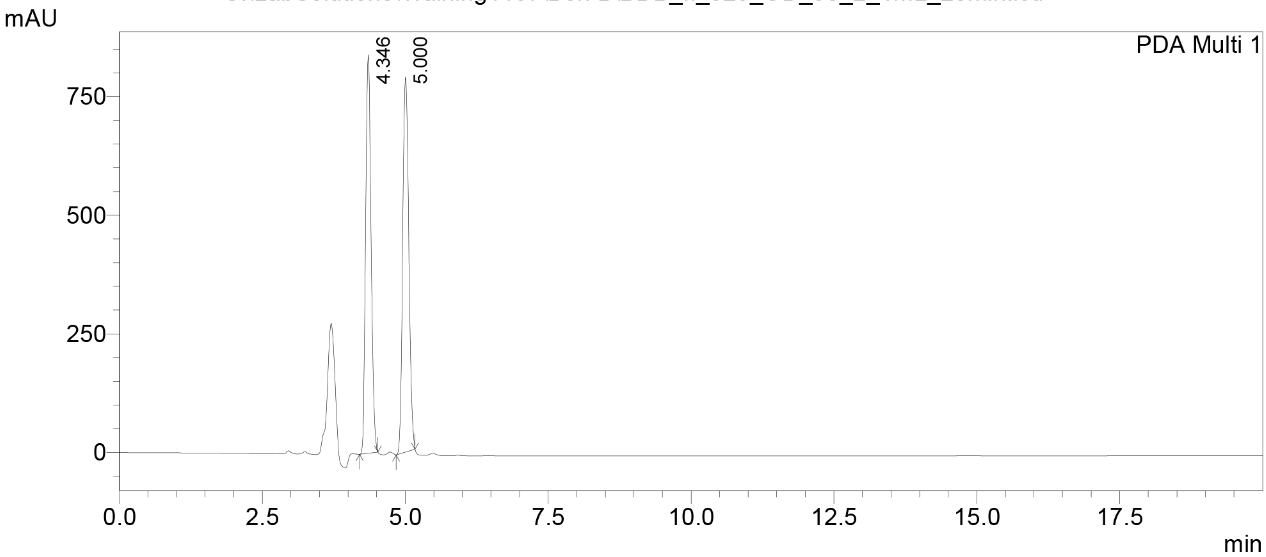
C:\LabSolutions\Training1107\Ben B\BDB_II_029_OD_98_2_1mL_20min.lcd

Acquired by	: Admin
Sample Name	: 1
Sample ID	: 1
Tray#	: 1
Vail #	: 94
Injection Volume	: 2 uL
Data File Name	: BDB_II_029_OD_98_2_1mL_20min.lcd
Method File Name	: OD-100_0_Hexane_IPA_20min.lcm
Batch File Name	: 24JAN2019.lcb
Report File Name	: UCD Default.lcr
Data Acquired	: 1/24/2019 12:38:55 PM
Data Processed	: 1/24/2019 12:58:56 PM



13d
racemic

<Chromatogram>



1 PDA Multi 1/205nm 4nm

< Peak Table >

PeakTable C:\LabSolutions\Training1107\Ben B\BDB_II_029_OD_98_2_1mL_20min.lcd
PDA Ch1 205nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	4.346	5560310	839786	48.727	51.518
2	5.000	5850778	790282	51.273	48.482
Total		11411088	1630068	100.000	100.000

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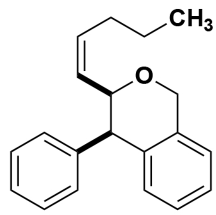
==== HPLC Analysis Report ====

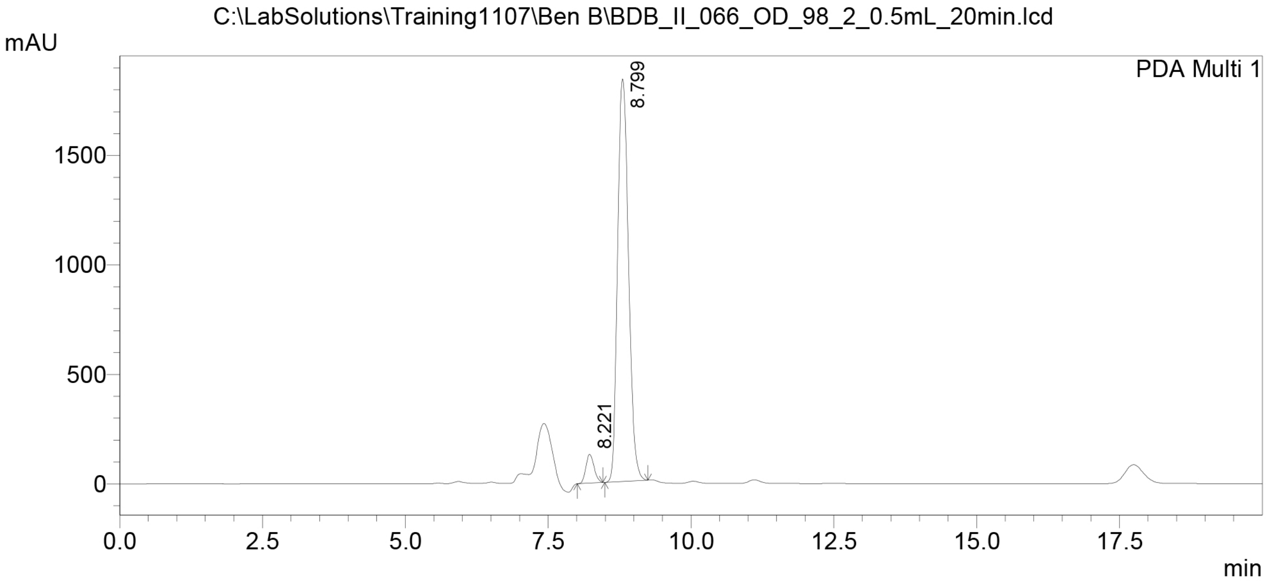
C:\LabSolutions\Training1107\Ben B\BDB_II_066_OD_98_2_0.5mL_20min.lcd

Acquired by	: Admin
Sample Name	: 1
Sample ID	: 1
Tray#	: 1
Vail #	: 62
Injection Volume	: 3 uL
Data File Name	: BDB_II_066_OD_98_2_0.5mL_20min.lcd
Method File Name	: OD-80_20_Hexane_IPA_20min_0.5mL.lcm
Batch File Name	: 04-10-19.lcb
Report File Name	: UCD Default.lcr
Data Acquired	: 4/10/2019 12:03:03 PM
Data Processed	: 4/10/2019 12:23:05 PM



13e
chiral

<Chromatogram>



1 PDA Multi 1/204nm 4nm

< Peak Table >

PeakTable C:\LabSolutions\Training1107\Ben B\BDB_II_066_OD_98_2_0.5mL_20min.lcd
PDA Ch1 204nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	8.221	1305504	130913	5.065	6.647
2	8.799	24467588	1838643	94.935	93.353
Total		25773092	1969555	100.000	100.000

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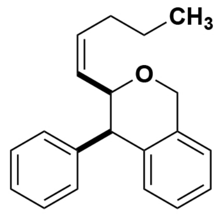
==== HPLC Analysis Report ====

C:\LabSolutions\Training1107\Ben B\BDB_II_065_OD_98_2_0.5mL_20min.lcd

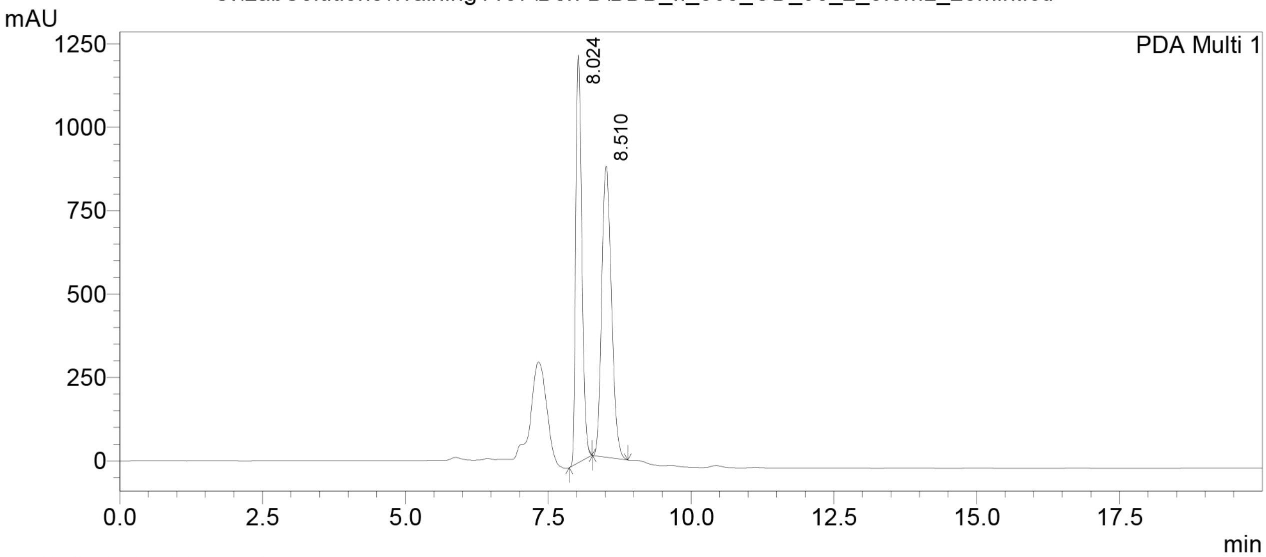
Acquired by	: Admin
Sample Name	: 1
Sample ID	: 1
Tray#	: 1
Vail #	: 62
Injection Volume	: 3 uL
Data File Name	: BDB_II_065_OD_98_2_0.5mL_20min.lcd
Method File Name	: OD-80_20_Hexane_IPA_20min_0.5mL.lcm
Batch File Name	: 08APR2019.lcb
Report File Name	: UCD Default.lcr
Data Acquired	: 4/8/2019 5:25:09 PM
Data Processed	: 4/8/2019 5:45:10 PM



13e
racemic

<Chromatogram>

C:\LabSolutions\Training1107\Ben B\BDB_II_065_OD_98_2_0.5mL_20min.lcd



1 PDA Multi 1/206nm 4nm

< Peak Table >

PeakTable C:\LabSolutions\Training1107\Ben B\BDB_II_065_OD_98_2_0.5mL_20min.lcd
PDA Ch1 206nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	8.024	9034375	1223049	46.507	58.365
2	8.510	10391516	872484	53.493	41.635
Total		19425891	2095533	100.000	100.000

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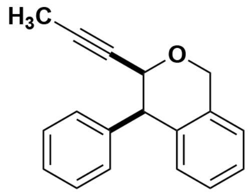
==== HPLC Analysis Report ====

C:\LabSolutions\Training1107\Ben B\BDB_II_006_AD_98_2_0.5mL.lcd

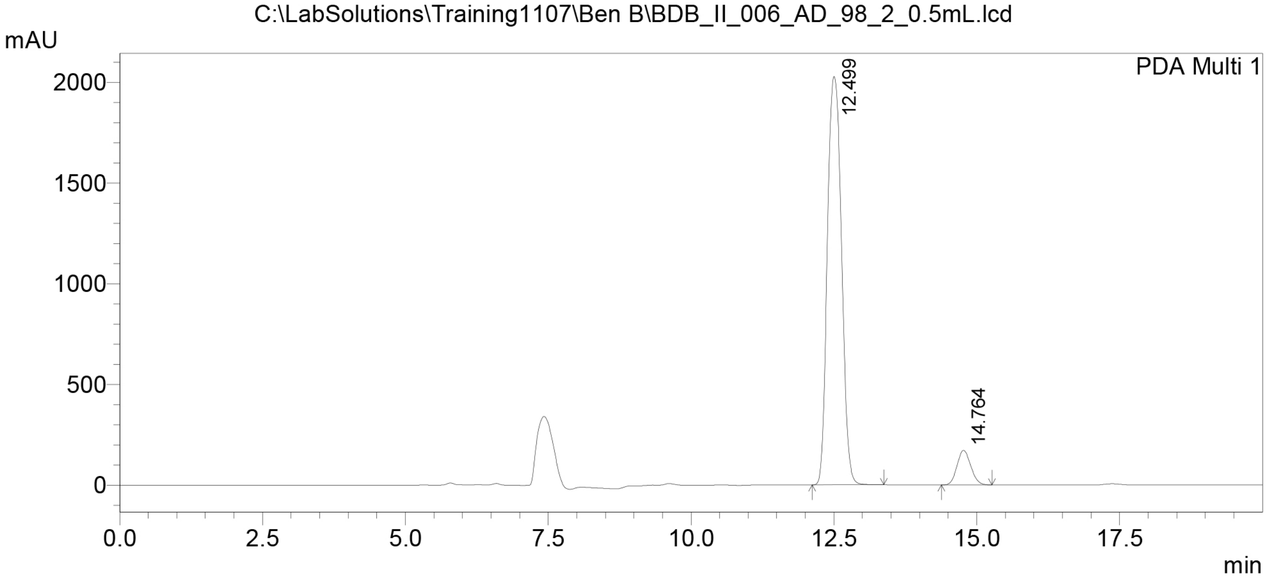
Acquired by	: Admin
Sample Name	: 1
Sample ID	: 1
Tray#	: 1
Vail #	: 94
Injection Volume	: 3 uL
Data File Name	: BDB_II_006_AD_98_2_0.5mL.lcd
Method File Name	: AD-100%_Hexanes_20min_0.5mL.lcm
Batch File Name	: 21DEC2018.lcb
Report File Name	: UCD Default.lcr
Data Acquired	: 12/21/2018 10:51:07 AM
Data Processed	: 12/21/2018 11:11:09 AM



13f
chiral

<Chromatogram>

C:\LabSolutions\Training1107\Ben B\BDB_II_006_AD_98_2_0.5mL.lcd



1 PDA Multi 1/206nm 4nm

< Peak Table >

PeakTable C:\LabSolutions\Training1107\Ben B\BDB_II_006_AD_98_2_0.5mL.lcd

PDA Ch1 206nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	12.499	34930795	2027919	92.406	92.215
2	14.764	2870640	171193	7.594	7.785
Total		37801435	2199112	100.000	100.000

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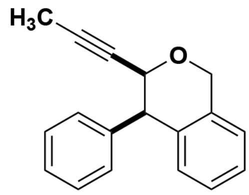
==== HPLC Analysis Report ====

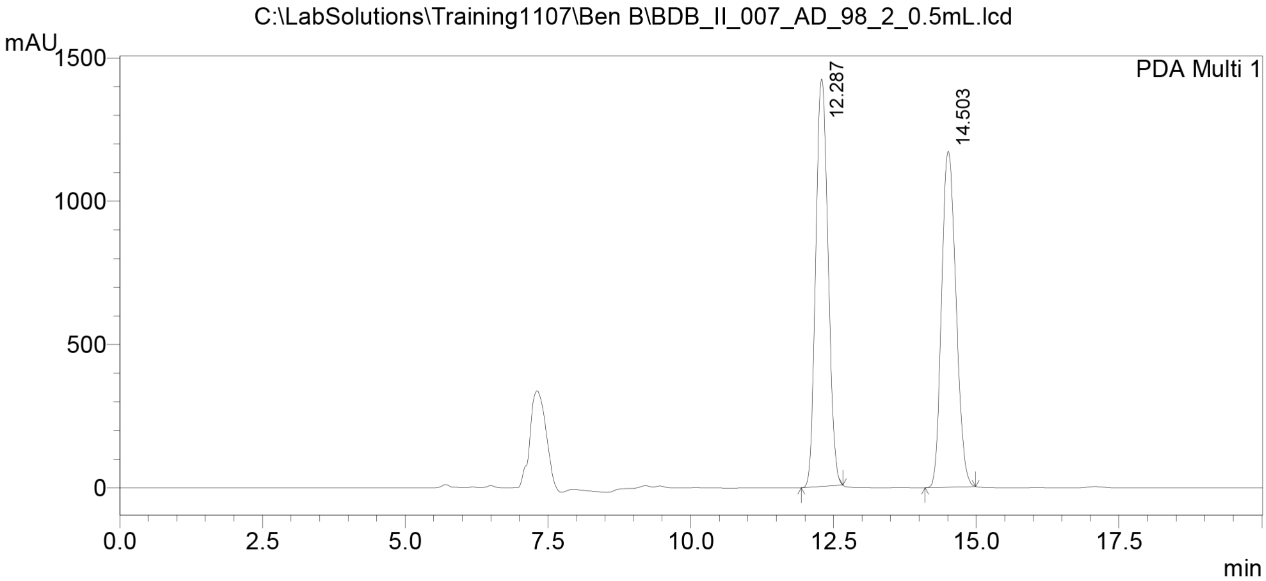
C:\LabSolutions\Training1107\Ben B\BDB_II_007_AD_98_2_0.5mL.lcd

Acquired by	: Admin
Sample Name	: 1
Sample ID	: 1
Tray#	: 1
Vail #	: 92
Injection Volume	: 3 uL
Data File Name	: BDB_II_007_AD_98_2_0.5mL.lcd
Method File Name	: AD-100%_Hexanes_20min_0.5mL.lcm
Batch File Name	: 20DEC2018.lcb
Report File Name	: UCD Default.lcr
Data Acquired	: 12/20/2018 4:34:30 PM
Data Processed	: 12/20/2018 4:54:30 PM



13f
racemic

<Chromatogram>



1 PDA Multi 1/206nm 4nm

< Peak Table >

PeakTable C:\LabSolutions\Training1107\Ben B\BDB_II_007_AD_98_2_0.5mL.lcd

PDA Ch1 206nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	12.287	21424492	1421636	51.024	54.800
2	14.503	20564510	1172595	48.976	45.200
Total		41989002	2594231	100.000	100.000

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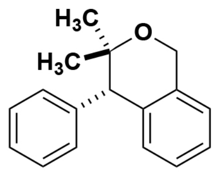
==== HPLC Analysis Report ====

C:\LabSolutions\Training1107\Ben B\BDB_I_106_AD2.lcd

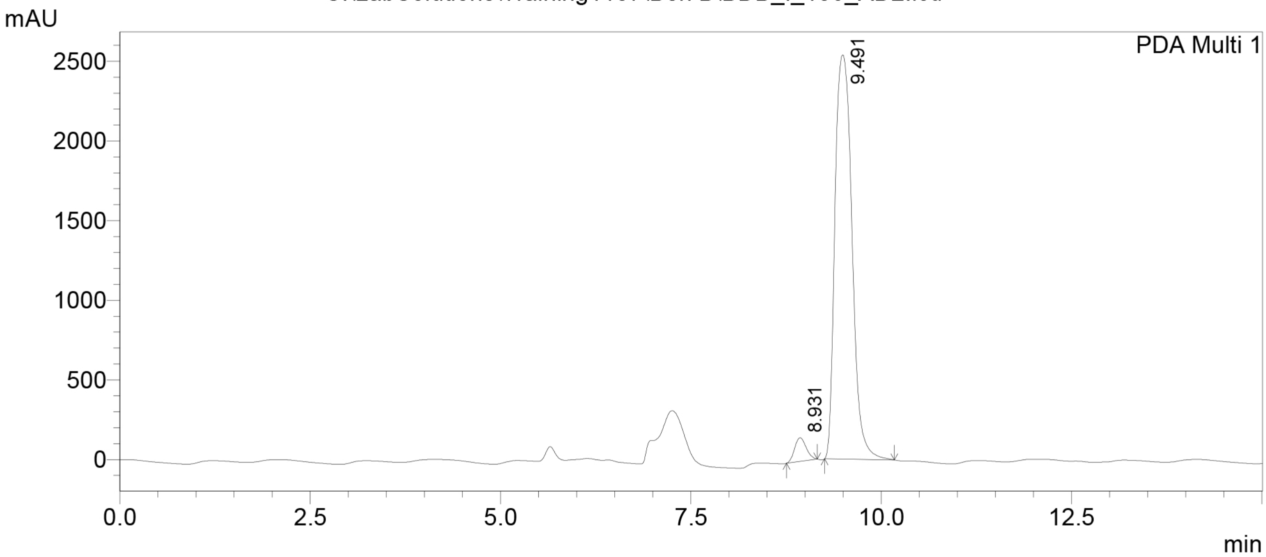
Acquired by : Admin
Sample Name : 1
Sample ID : 1
Tray# : 1
Vail # : 96
Injection Volume : 10 uL
Data File Name : BDB_I_106_AD2.lcd
Method File Name : AD-98_2_Hexane_IPA_15min_0.5mL.lcm
Batch File Name : 30JUL2018_3.lcb
Report File Name : UCD Default.lcr
Data Acquired : 7/30/2018 9:06:49 AM
Data Processed : 7/30/2018 9:21:50 AM



15a
chiral

<Chromatogram>

C:\LabSolutions\Training1107\Ben B\BDB_I_106_AD2.lcd



1 PDA Multi 1/206nm 4nm

< Peak Table >

PeakTable C:\LabSolutions\Training1107\Ben B\BDB_I_106_AD2.lcd
PDA Ch1 206nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	8.931	1596198	149975	3.887	5.582
2	9.491	39471429	2536622	96.113	94.418
Total		41067627	2686596	100.000	100.000

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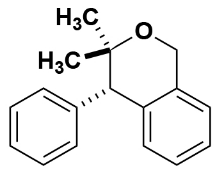
==== HPLC Analysis Report ====

C:\LabSolutions\Training1107\Ben B\BDB_I_107_AD3.lcd

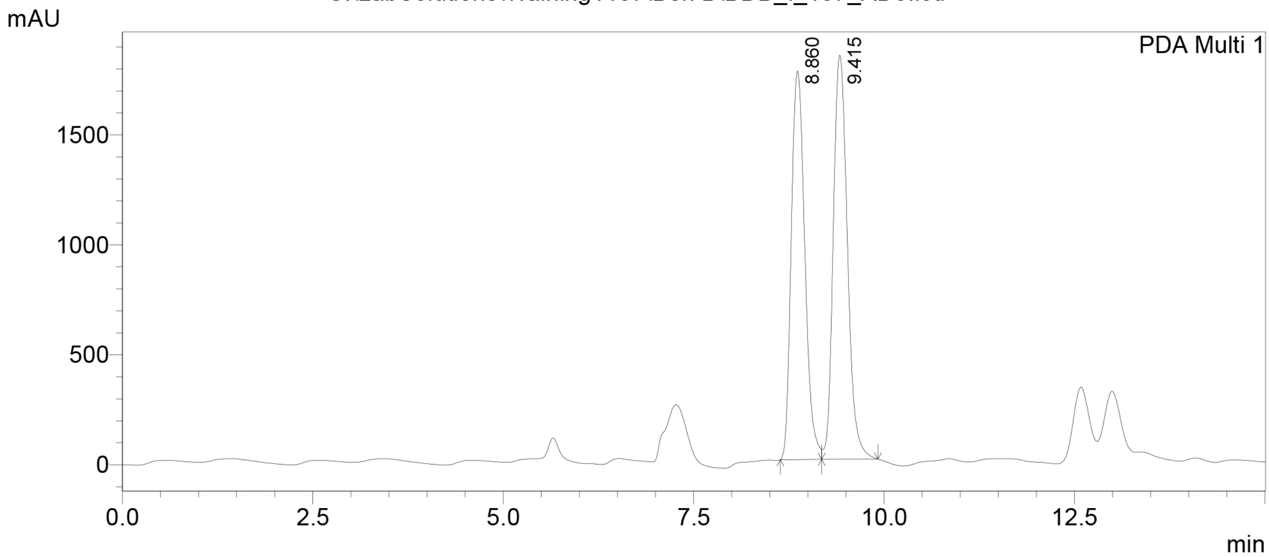
Acquired by : Admin
Sample Name : 1
Sample ID : 1
Tray# : 1
Vail # : 97
Injection Volume : 10 uL
Data File Name : BDB_I_107_AD3.lcd
Method File Name : AD-98_2_Hexane_IPA_15min_0.5mL.lcm
Batch File Name : 30JUL2018_3.lcb
Report File Name : UCD Default.lcr
Data Acquired : 7/30/2018 8:51:22 AM
Data Processed : 7/30/2018 9:06:24 AM



15a
racemic

<Chromatogram>

C:\LabSolutions\Training1107\Ben B\BDB_I_107_AD3.lcd



1 PDA Multi 1/206nm 4nm

< Peak Table >

PeakTable C:\LabSolutions\Training1107\Ben B\BDB_I_107_AD3.lcd

PDA Ch1 206nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	8.860	21883165	1767860	48.004	49.022
2	9.415	23703300	1838388	51.996	50.978
Total		45586465	3606248	100.000	100.000

==== HPLC Analysis Report ====

C:\LabSolutions\Training1107\Ben B\BDB_II_110_OD_99_1_1mL_30min.lcd

Acquired by : Admin
Sample Name : 1
Sample ID : 1
Tray# : 1
Vail # : 4
Injection Volume : 2 uL
Data File Name : BDB_II_110_OD_99_1_1mL_30min.lcd
Method File Name : OD-90_10_Hexane_IPA_30min.lcm
Batch File Name : 01JUN2019.lcb
Report File Name : UCD Default.lcr
Data Acquired : 6/1/2019 12:48:19 PM
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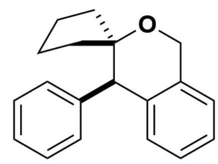
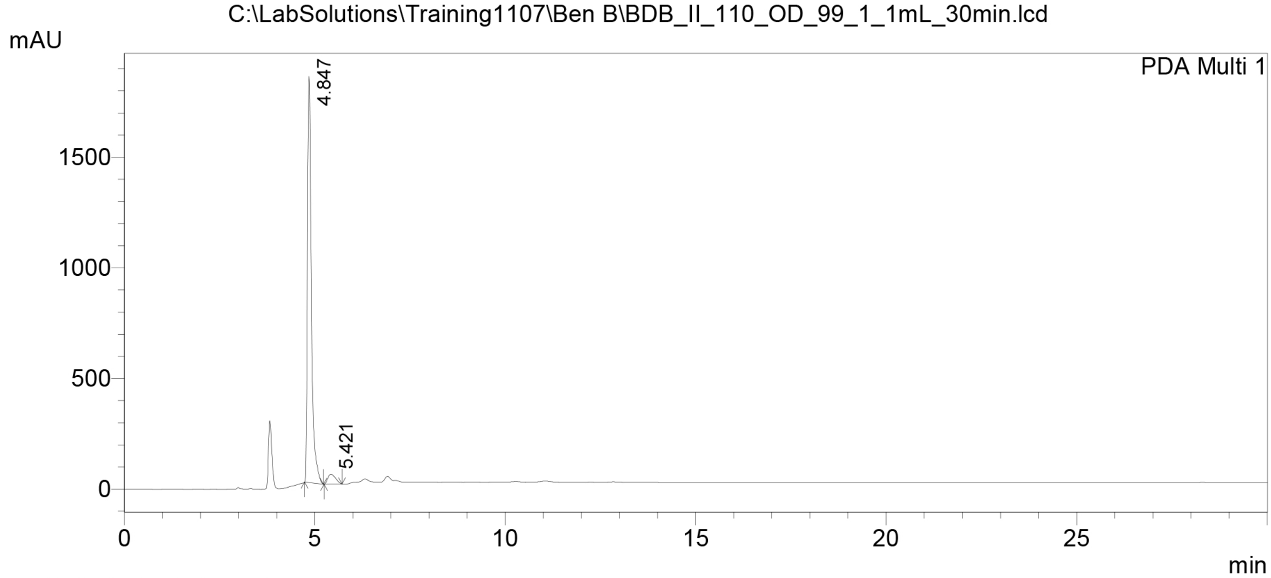


15b
chiral

<Chromatogram>



1 PDA Multi 1/205nm 4nm

< Peak Table >

PeakTable C:\LabSolutions\Training1107\Ben B\BDB_II_110_OD_99_1_1mL_30min.lcd
PDA Ch1 205nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	4.847	13133076	1835936	95.400	97.636
2	5.421	633188	44454	4.600	2.364
Total		13766264	1880390	100.000	100.000

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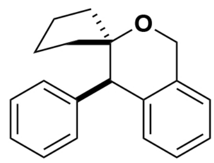
==== HPLC Analysis Report ====

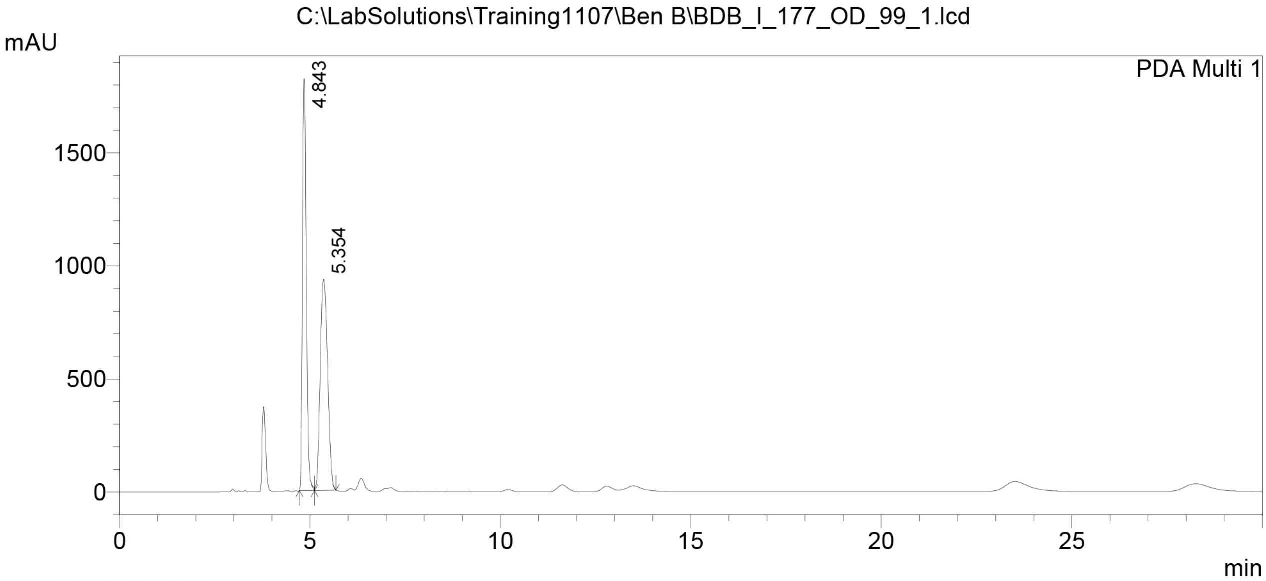
C:\LabSolutions\Training1107\Ben B\BDB_I_177_OD_99_1.lcd

Acquired by : Admin
Sample Name : 1
Sample ID : 1
Tray# : 1
Vail # : 70
Injection Volume : 5 uL
Data File Name : BDB_I_177_OD_99_1.lcd
Method File Name : OD-100_0_Hexane_IPA_30min.lcm
Batch File Name : 03DEC2018.lcb
Report File Name : UCD Default.lcr
Data Acquired : 12/3/2018 11:55:11 PM
Data Processed : 12/4/2018 12:25:13 AM



15b
racemic

<Chromatogram>



1 PDA Multi 1/205nm 4nm

< Peak Table >

PeakTable C:\LabSolutions\Training1107\Ben B\BDB_I_177_OD_99_1.lcd

PDA Ch1 205nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	4.843	12918307	1821349	50.490	66.116
2	5.354	12667469	933445	49.510	33.884
Total		25585776	2754794	100.000	100.000

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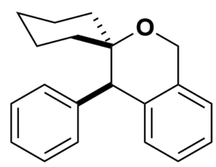
==== HPLC Analysis Report ====

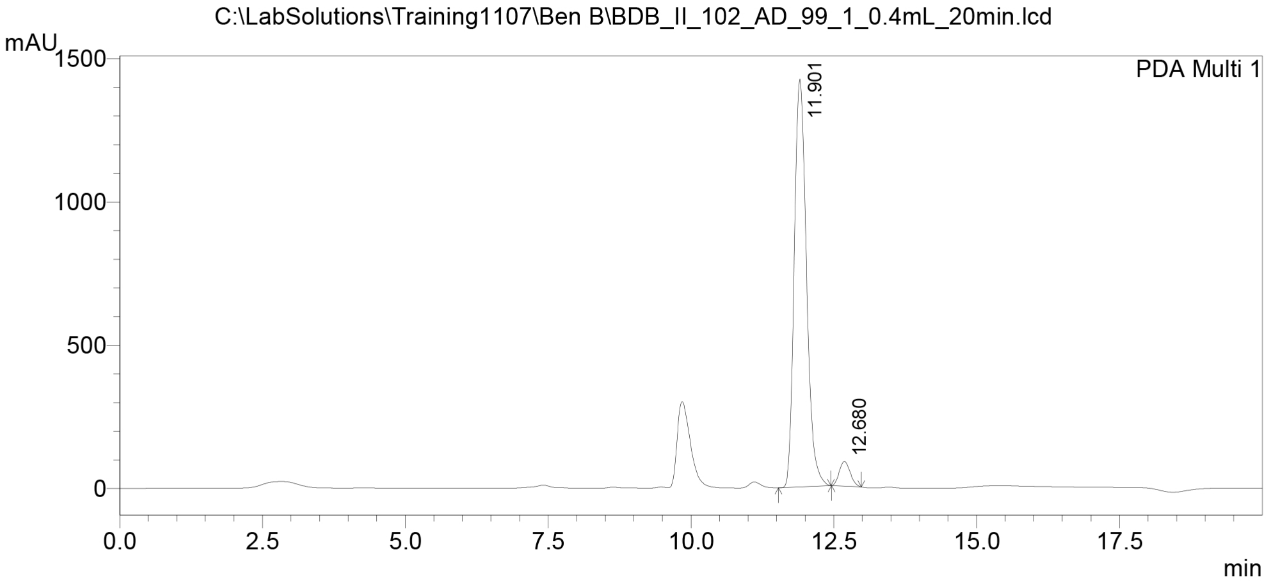
C:\LabSolutions\Training1107\Ben B\BDB_II_102_AD_99_1_0.4mL_20min.lcd

Acquired by	: Admin
Sample Name	: 1
Sample ID	: 1
Tray#	: 1
Vail #	: 2
Injection Volume	: 2 uL
Data File Name	: BDB_II_102_AD_99_1_0.4mL_20min.lcd
Method File Name	: AD-90_10_Hexane_IPA_20min_0.4mL.lcm
Batch File Name	: 31MAY2019.lcb
Report File Name	: UCD Default.lcr
Data Acquired	: 5/31/2019 8:30:03 PM
Data Processed	: 5/31/2019 8:50:04 PM



15c
chiral

<Chromatogram>



1 PDA Multi 1/204nm 4nm

< Peak Table >

PeakTable C:\LabSolutions\Training1107\Ben B\BDB_II_102_AD_99_1_0.4mL_20min.lcd
PDA Ch1 204nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	11.901	21205026	1423918	94.860	94.265
2	12.680	1148958	86626	5.140	5.735
Total		22353985	1510545	100.000	100.000

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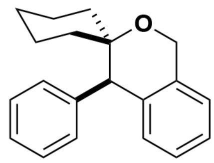
==== HPLC Analysis Report ====

C:\LabSolutions\Training1107\Ben B\BDB_II_103_AD_99_1_0.4mL_20min.lcd

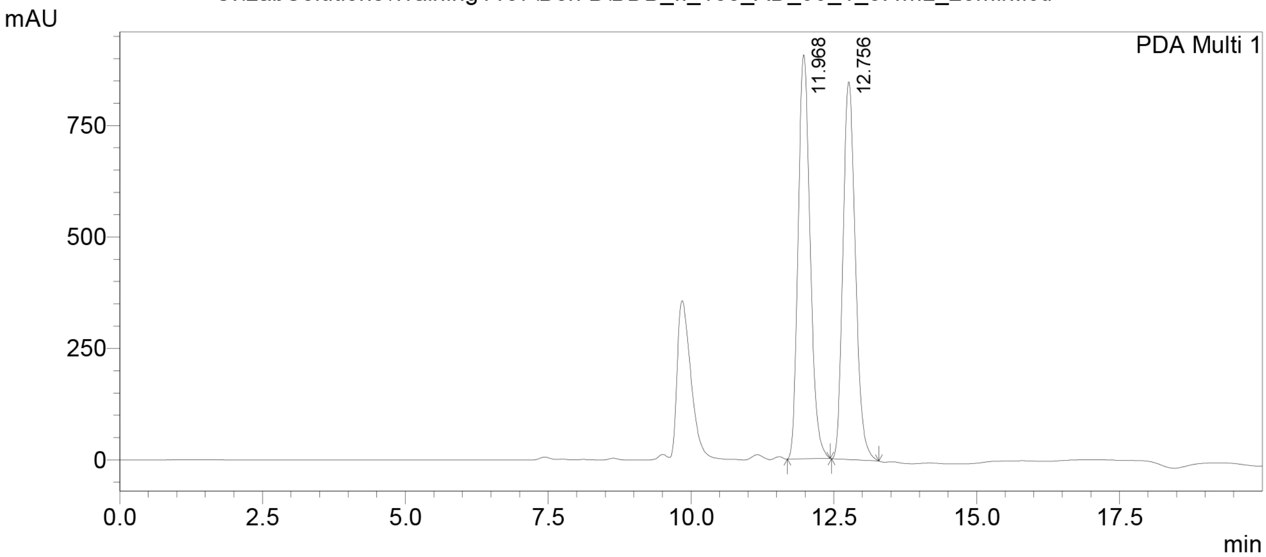
Acquired by	: Admin
Sample Name	: 1
Sample ID	: 1
Tray#	: 1
Vail #	: 3
Injection Volume	: 2 uL
Data File Name	: BDB_II_103_AD_99_1_0.4mL_20min.lcd
Method File Name	: AD-90_10_Hexane_IPA_20min_0.4mL.lcm
Batch File Name	: 31MAY2019.lcb
Report File Name	: UCD Default.lcr
Data Acquired	: 5/31/2019 8:09:36 PM
Data Processed	: 5/31/2019 8:29:39 PM



15c
racemic

<Chromatogram>

C:\LabSolutions\Training1107\Ben B\BDB_II_103_AD_99_1_0.4mL_20min.lcd



1 PDA Multi 1/204nm 4nm

< Peak Table >

PeakTable C:\LabSolutions\Training1107\Ben B\BDB_II_103_AD_99_1_0.4mL_20min.lcd
PDA Ch1 204nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	11.968	13259527	905585	50.711	51.655
2	12.756	12887700	847559	49.289	48.345
Total		26147227	1753144	100.000	100.000

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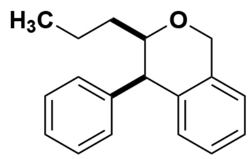
==== HPLC Analysis Report ====

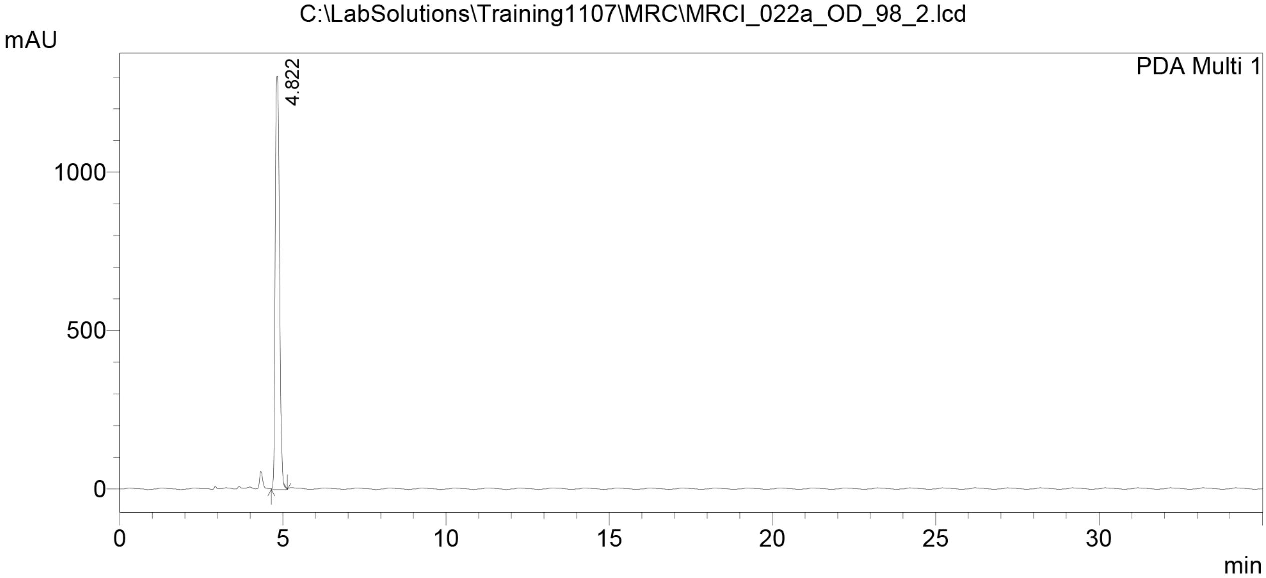
C:\LabSolutions\Training1107\MRC\MRCI_022a_OD_98_2.lcd

Acquired by	: Admin
Sample Name	: 1
Sample ID	: 1
Tray#	: 1
Vail #	: 63
Injection Volume	: 10 uL
Data File Name	: MRCI_022a_OD_98_2.lcd
Method File Name	: OD-98_2_Hexane_IPA_35min.lcm
Batch File Name	: 7-18-18.lcb
Report File Name	: UCD Default.lcr
Data Acquired	: 7/18/2018 12:13:59 PM
Data Processed	: 7/18/2018 12:49:02 PM



15d
chiral

<Chromatogram>



1 PDA Multi 1/222nm 4nm

< Peak Table >

PeakTable C:\LabSolutions\Training1107\MRC\MRCI_022a_OD_98_2.lcd

PDA Ch1 222nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	4.822	11988170	1304698	100.000	100.000
Total		11988170	1304698	100.000	100.000

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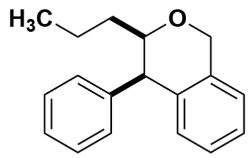
==== HPLC Analysis Report ====

C:\LabSolutions\Training1107\MRC\MRCI_021a_98_2_OD.lcd

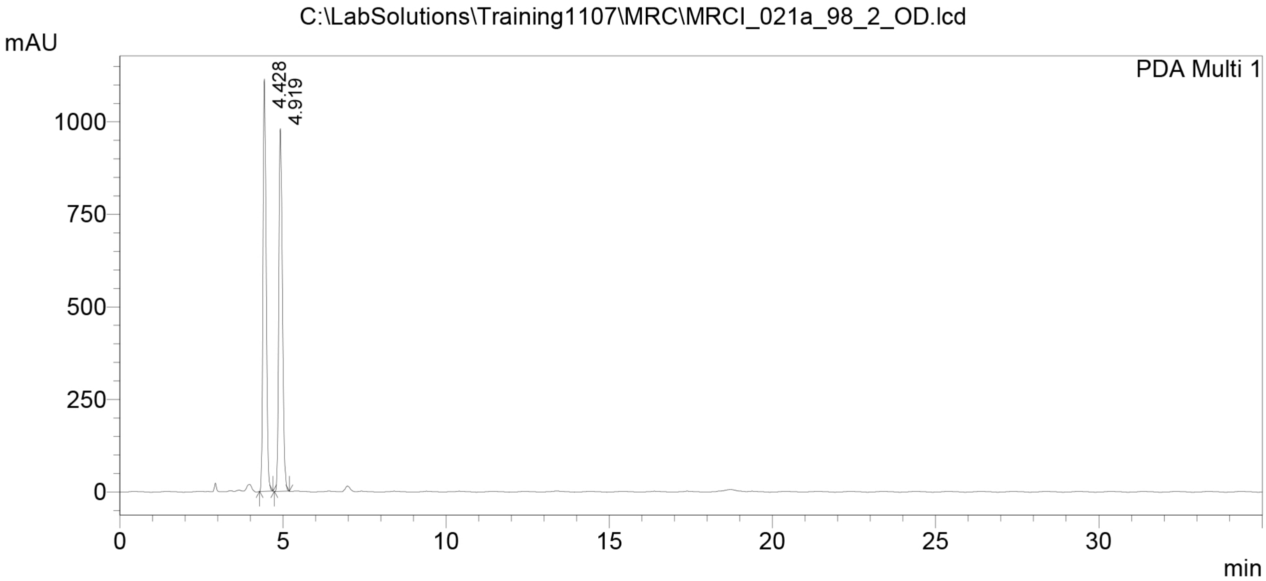
Acquired by	: Admin
Sample Name	: 1
Sample ID	: 1
Tray#	: 1
Vail #	: 91
Injection Volume	: 10 uL
Data File Name	: MRCI_021a_98_2_OD.lcd
Method File Name	: OD-98_2_Hexane_IPA_35min.lcm
Batch File Name	: 6-29-18.lcb
Report File Name	: UCD Default.lcr
Data Acquired	: 6/29/2018 8:02:17 AM
Data Processed	: 6/29/2018 8:37:19 AM



15d

racemic

<Chromatogram>



1 PDA Multi 1/215nm

< Peak Table >

PeakTable C:\LabSolutions\Training1107\MRC\MRCI_021a_98_2_OD.lcd

PDA Ch1 215nm 0nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	4.428	7613902	1113876	49.853	53.186
2	4.919	7658908	980426	50.147	46.814
Total		15272810	2094302	100.000	100.000

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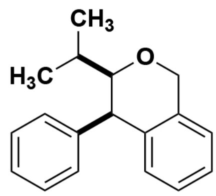
==== HPLC Analysis Report ====

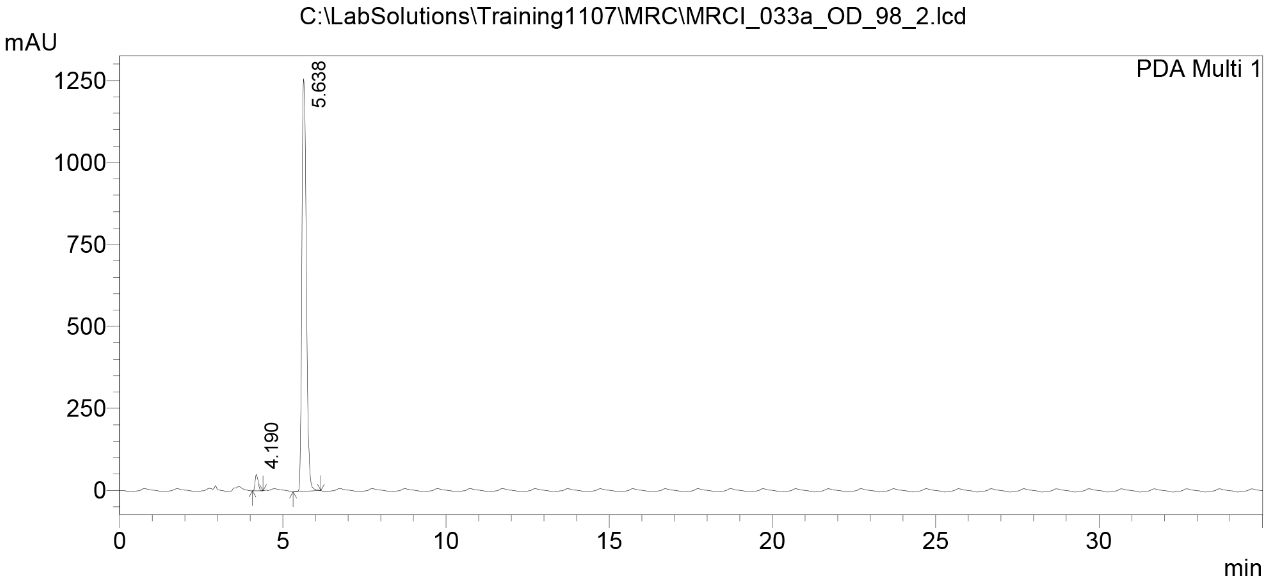
C:\LabSolutions\Training1107\MRC\MRCI_033a_OD_98_2.lcd

Acquired by	: Admin
Sample Name	: 1
Sample ID	: 1
Tray#	: 1
Vail #	: 61
Injection Volume	: 10 uL
Data File Name	: MRCI_033a_OD_98_2.lcd
Method File Name	: OD-98_2_Hexane_IPA_35min.lcm
Batch File Name	: 05SEP2018_2.lcb
Report File Name	: UCD Default.lcr
Data Acquired	: 9/6/2018 12:57:23 PM
Data Processed	: 9/6/2018 1:32:23 PM



15e
chiral

<Chromatogram>



1 PDA Multi 1/222nm 4nm

< Peak Table >

PeakTable C:\LabSolutions\Training1107\MRC\MRCI_033a_OD_98_2.lcd

PDA Ch1 222nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	4.190	317905	49680	2.579	3.799
2	5.638	12008642	1258172	97.421	96.201
Total		12326547	1307853	100.000	100.000

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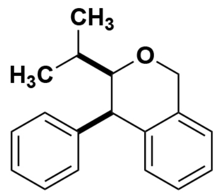
==== HPLC Analysis Report ====

C:\LabSolutions\Training1107\MRC\MRCI_032a_OD_98_2.lcd

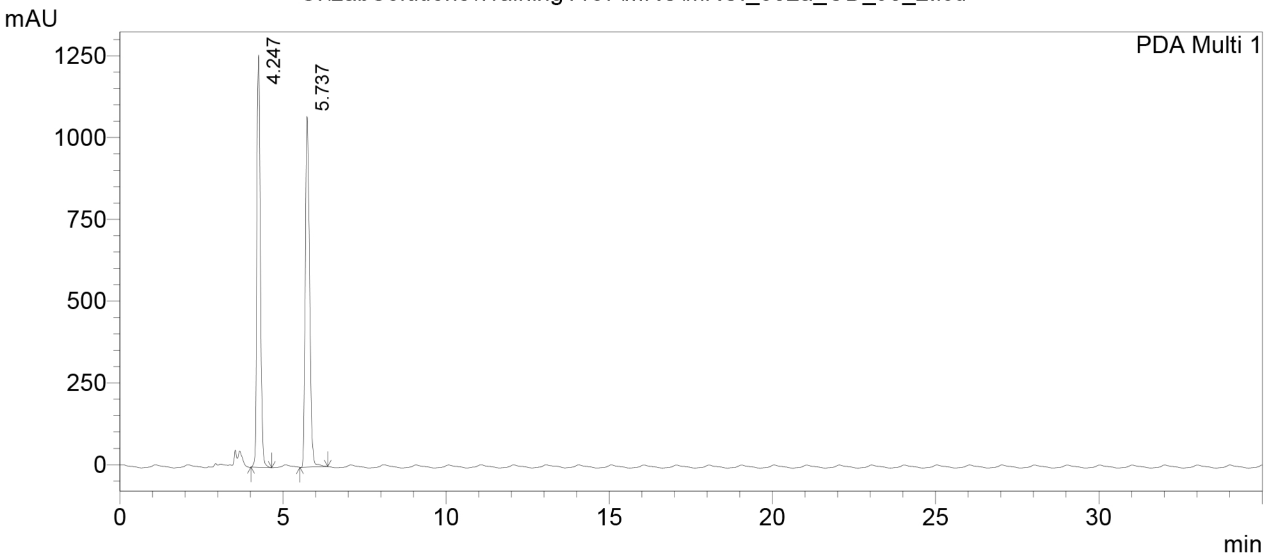
Acquired by	: Admin
Sample Name	: 1
Sample ID	: 1
Tray#	: 1
Vail #	: 46
Injection Volume	: 10 uL
Data File Name	: MRCI_032a_OD_98_2.lcd
Method File Name	: OD-98_2_Hexane_IPA_35min.lcm
Batch File Name	: 05SEP2018.lcb
Report File Name	: UCD Default.lcr
Data Acquired	: 9/6/2018 11:31:47 AM
Data Processed	: 9/6/2018 12:06:48 PM



15e
racemic

<Chromatogram>

C:\LabSolutions\Training1107\MRC\MRCI_032a_OD_98_2.lcd



1 PDA Multi 1/222nm 4nm

< Peak Table >

PeakTable C:\LabSolutions\Training1107\MRC\MRCI_032a_OD_98_2.lcd

PDA Ch1 222nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	4.247	9376189	1260561	48.750	54.028
2	5.737	9857158	1072622	51.250	45.972
Total		19233348	2333183	100.000	100.000

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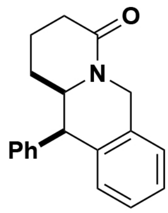
==== HPLC Analysis Report ====

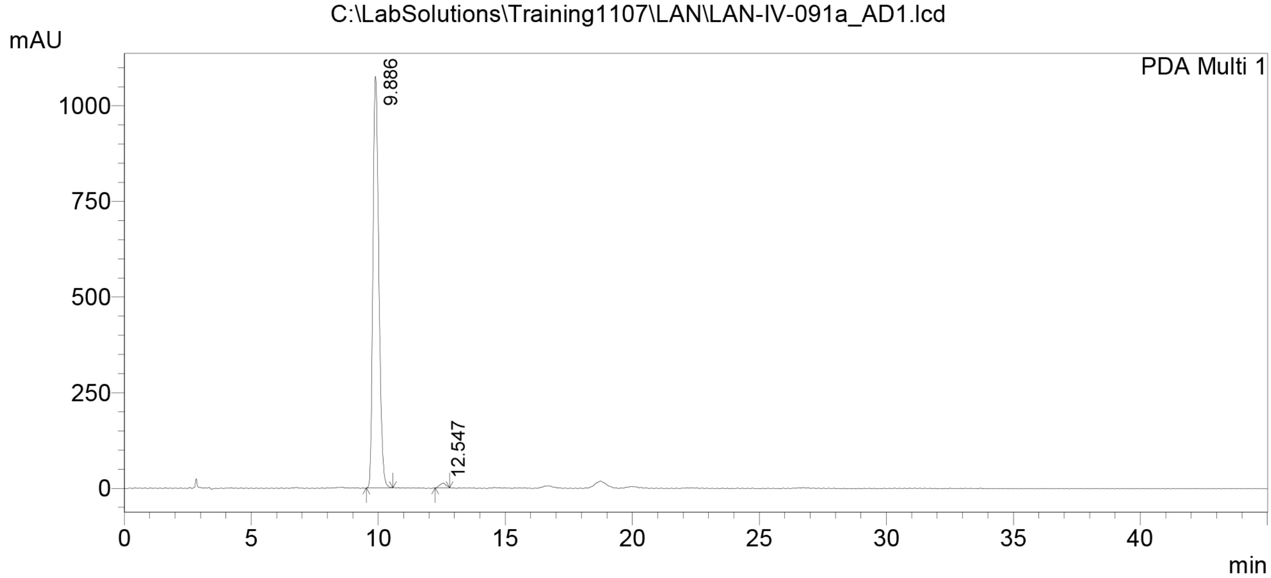
C:\LabSolutions\Training1107\LAN\LAN-IV-091a_AD1.lcd

Acquired by	: Admin
Sample Name	: LAN-IV-091a
Sample ID	: LAN-IV-091a
Tray#	: 1
Vail #	: 92
Injection Volume	: 5 uL
Data File Name	: LAN-IV-091a_AD1.lcd
Method File Name	: AD-90_10_Hexane_IPA_45min.lcm
Batch File Name	: 12.18.18.lcb
Report File Name	: UCD Default.lcr
Data Acquired	: 12/18/2018 12:50:22 PM
Data Processed	: 12/18/2018 1:41:38 PM



23a
chiral

<Chromatogram>



1 PDA Multi 1/207nm 4nm

< Peak Table >

PeakTable C:\LabSolutions\Training1107\LAN\LAN-IV-091a_AD1.lcd

PDA Ch1 207nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	9.886	17324845	1075560	98.816	98.892
2	12.547	207596	12048	1.184	1.108
Total		17532441	1087608	100.000	100.000

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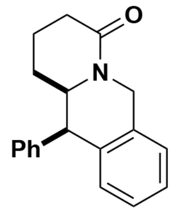
==== HPLC Analysis Report ====

C:\LabSolutions\Training1107\LAN\LAN-IV-095a_AD1.lcd

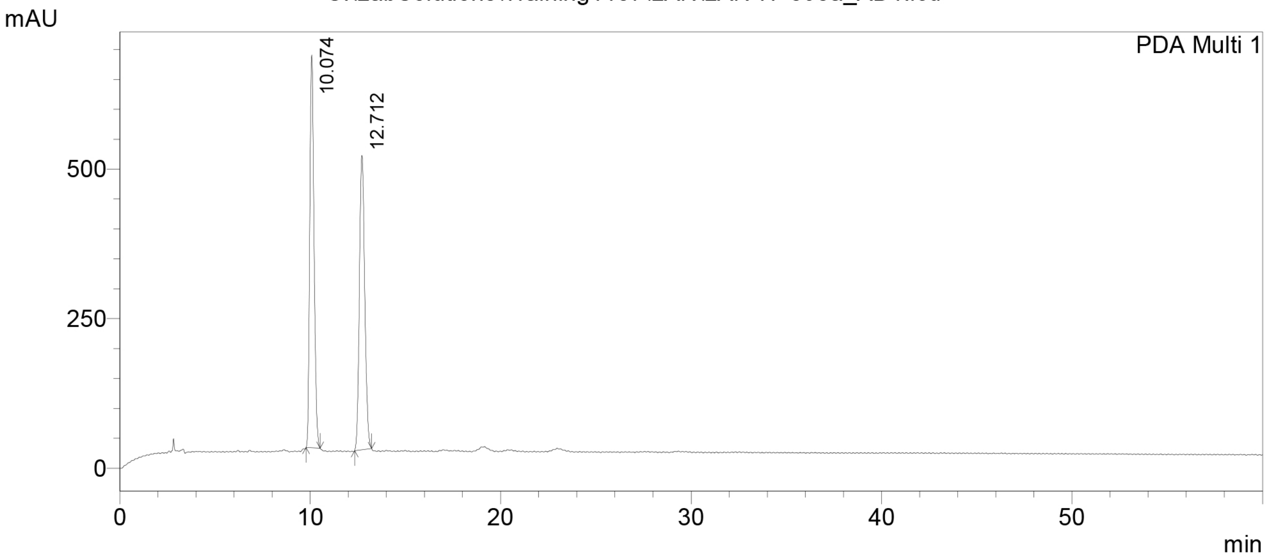
Acquired by : Admin
Sample Name : LAN-IV-095a
Sample ID : LAN-IV-095a
Tray# : 1
Vail # : 91
Injection Volume : 5 uL
Data File Name : LAN-IV-095a_AD1.lcd
Method File Name : AD-90_10_Hexane_IPA_60min_1mLmin.lcm
Batch File Name : 12.18.18.lcb
Report File Name : UCD Default.lcr
Data Acquired : 12/18/2018 11:39:46 AM
Data Processed : 12/18/2018 1:42:03 PM



23a
racemic

<Chromatogram>

C:\LabSolutions\Training1107\LAN\LAN-IV-095a_AD1.lcd



1 PDA Multi 1/207nm 4nm

< Peak Table >

PeakTable C:\LabSolutions\Training1107\LAN\LAN-IV-095a_AD1.lcd

PDA Ch1 207nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	10.074	10266411	656606	51.641	57.142
2	12.712	9613764	492476	48.359	42.858
Total		19880175	1149082	100.000	100.000

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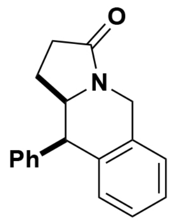
==== HPLC Analysis Report ====

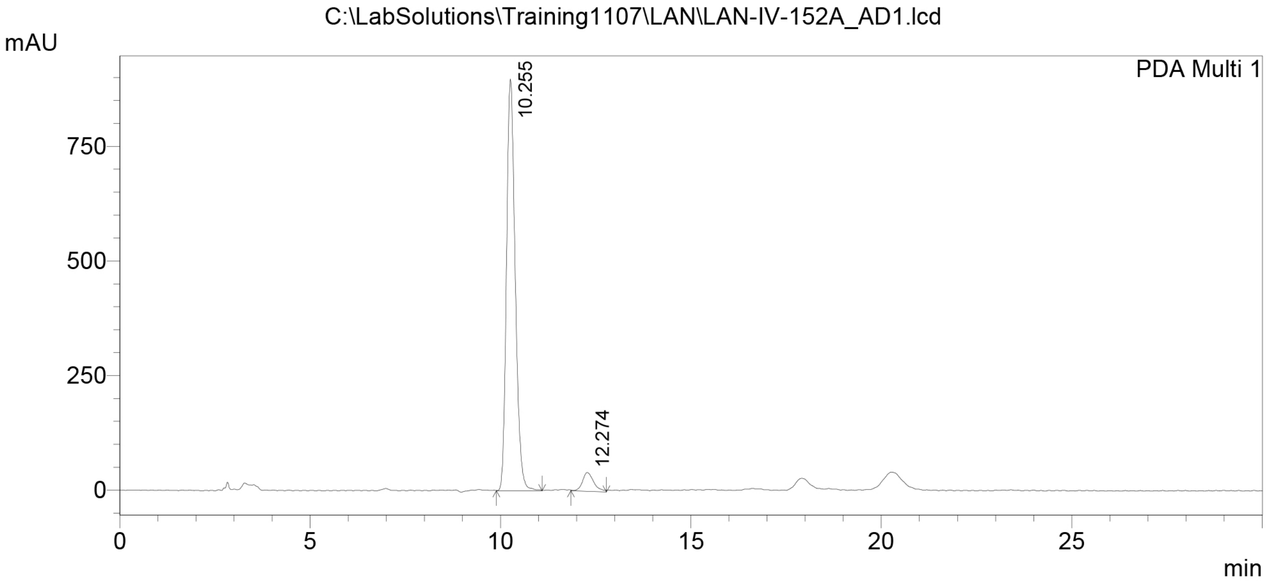
C:\LabSolutions\Training1107\LAN\LAN-IV-152A_AD1.lcd

Acquired by : Admin
Sample Name : LAN-IV-152A
Sample ID : LAN-IV-152A
Tray# : 1
Vail # : 63
Injection Volume : 5 uL
Data File Name : LAN-IV-152A_AD1.lcd
Method File Name : AD-90_10_Hexane_IPA_30min.lcm
Batch File Name : 5.2.19.lcb
Report File Name : UCD Default.lcr
Data Acquired : 5/2/2019 3:23:44 PM
Data Processed : 5/2/2019 3:53:46 PM



23b
chiral

<Chromatogram>



1 PDA Multi 1/209nm 4nm

< Peak Table >

PeakTable C:\LabSolutions\Training1107\LAN\LAN-IV-152A_AD1.lcd

PDA Ch1 209nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	10.255	14291014	897949	94.365	95.627
2	12.274	853337	41065	5.635	4.373
Total		15144351	939015	100.000	100.000

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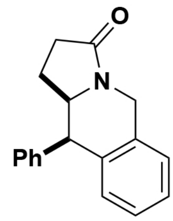
==== HPLC Analysis Report ====

C:\LabSolutions\Training1107\LAN\LAN-IV-159B_AD1.lcd

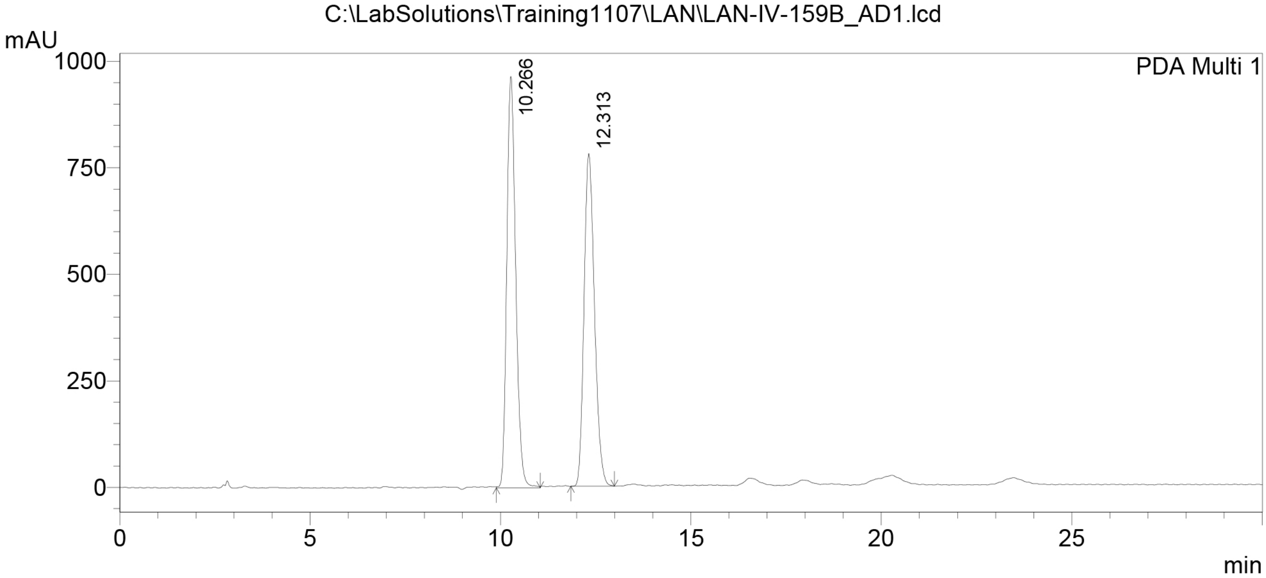
Acquired by	: Admin
Sample Name	: LAN-IV-159B
Sample ID	: LAN-IV-159B
Tray#	: 1
Vail #	: 62
Injection Volume	: 5 uL
Data File Name	: LAN-IV-159B_AD1.lcd
Method File Name	: AD-90_10_Hexane_IPA_30min.lcm
Batch File Name	: 5.2.19.lcb
Report File Name	: UCD Default.lcr
Data Acquired	: 5/2/2019 2:43:07 PM
Data Processed	: 5/2/2019 3:43:10 PM



23b
racemic

<Chromatogram>

C:\LabSolutions\Training1107\LAN\LAN-IV-159B_AD1.lcd



1 PDA Multi 1/207nm 4nm

< Peak Table >

PeakTable C:\LabSolutions\Training1107\LAN\LAN-IV-159B_AD1.lcd

PDA Ch1 207nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	10.266	15724723	965545	51.325	55.275
2	12.313	14913040	781263	48.675	44.725
Total		30637763	1746808	100.000	100.000

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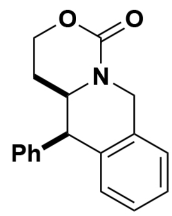
==== HPLC Analysis Report ====

C:\LabSolutions\Training1107\LAN\LAN-IV-178a_AD2.lcd

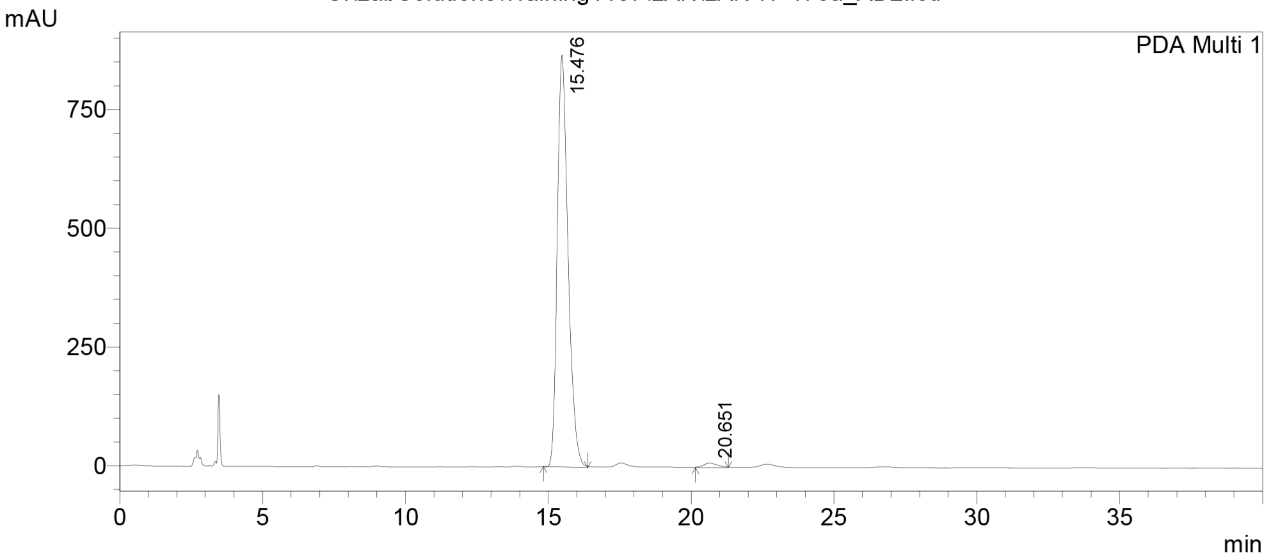
Acquired by	: Admin
Sample Name	: LAN-IV-178a_AD1.lcd
Sample ID	: LAN-IV-178a_AD7.lcd
Tray#	: 1
Vail #	: 63
Injection Volume	: 5 uL
Data File Name	: LAN-IV-178a_AD2.lcd
Method File Name	: AD-100%IPA-40min.lcm
Batch File Name	: 5.31.19.lcb
Report File Name	: UCD Default.lcr
Data Acquired	: 5/31/2019 3:39:50 PM
Data Processed	: 5/31/2019 4:19:54 PM



23c
chiral

<Chromatogram>

C:\LabSolutions\Training1107\LAN\LAN-IV-178a_AD2.lcd



1 PDA Multi 1/206nm 4nm

< Peak Table >

PeakTable C:\LabSolutions\Training1107\LAN\LAN-IV-178a_AD2.lcd

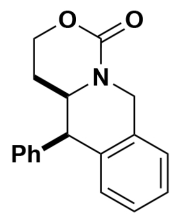
PDA Ch1 206nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	15.476	22796550	867573	98.580	98.916
2	20.651	328491	9511	1.420	1.084
Total		23125040	877084	100.000	100.000

==== HPLC Analysis Report ====

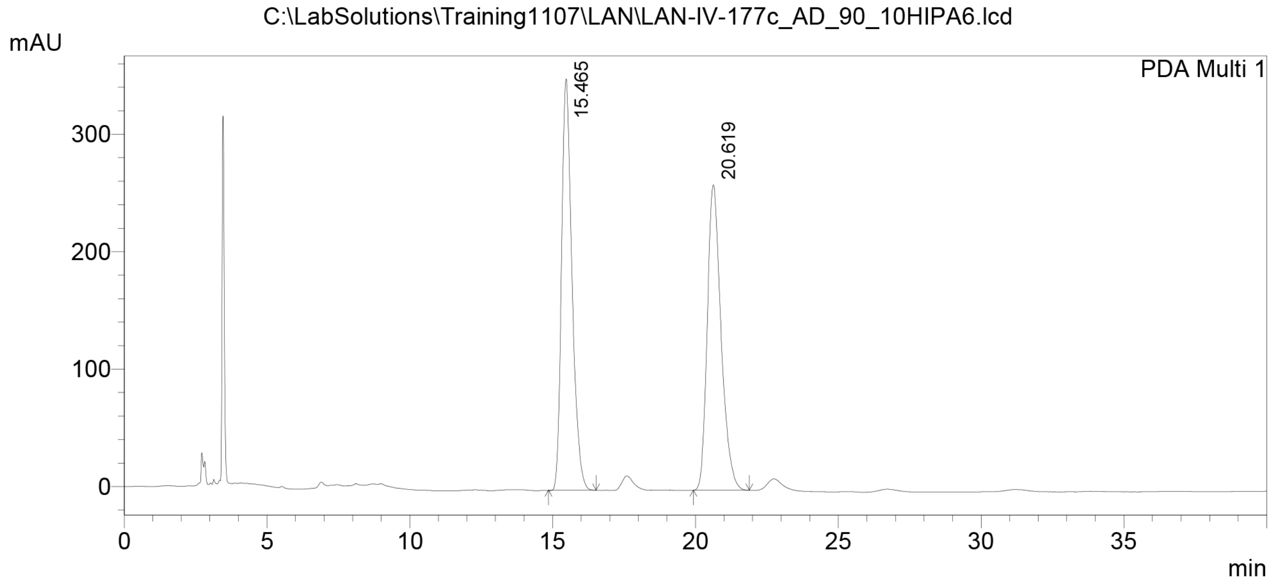
C:\LabSolutions\Training1107\LAN\LAN-IV-177c_AD_90_10HIPA6.lcd

Acquired by	: Admin
Sample Name	: LAN-IV-177c_AD1.lcd
Sample ID	: LAN-IV-177c_AD1.lcd
Tray#	: 1
Vail #	: 62
Injection Volume	: 5 uL
Data File Name	: LAN-IV-177c_AD_90_10HIPA6.lcd
Method File Name	: AD-100%IPA-40min.lcm
Batch File Name	: 5.31.19.lcb
Report File Name	: UCD Default.lcr
Data Acquired	: 5/31/2019 1:03:35 PM
Data Processed	: 5/31/2019 1:43:38 PM



23c
racemic

<Chromatogram>

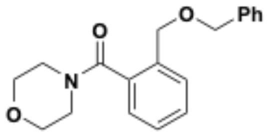


1 PDA Multi 1/206nm 4nm

< Peak Table >

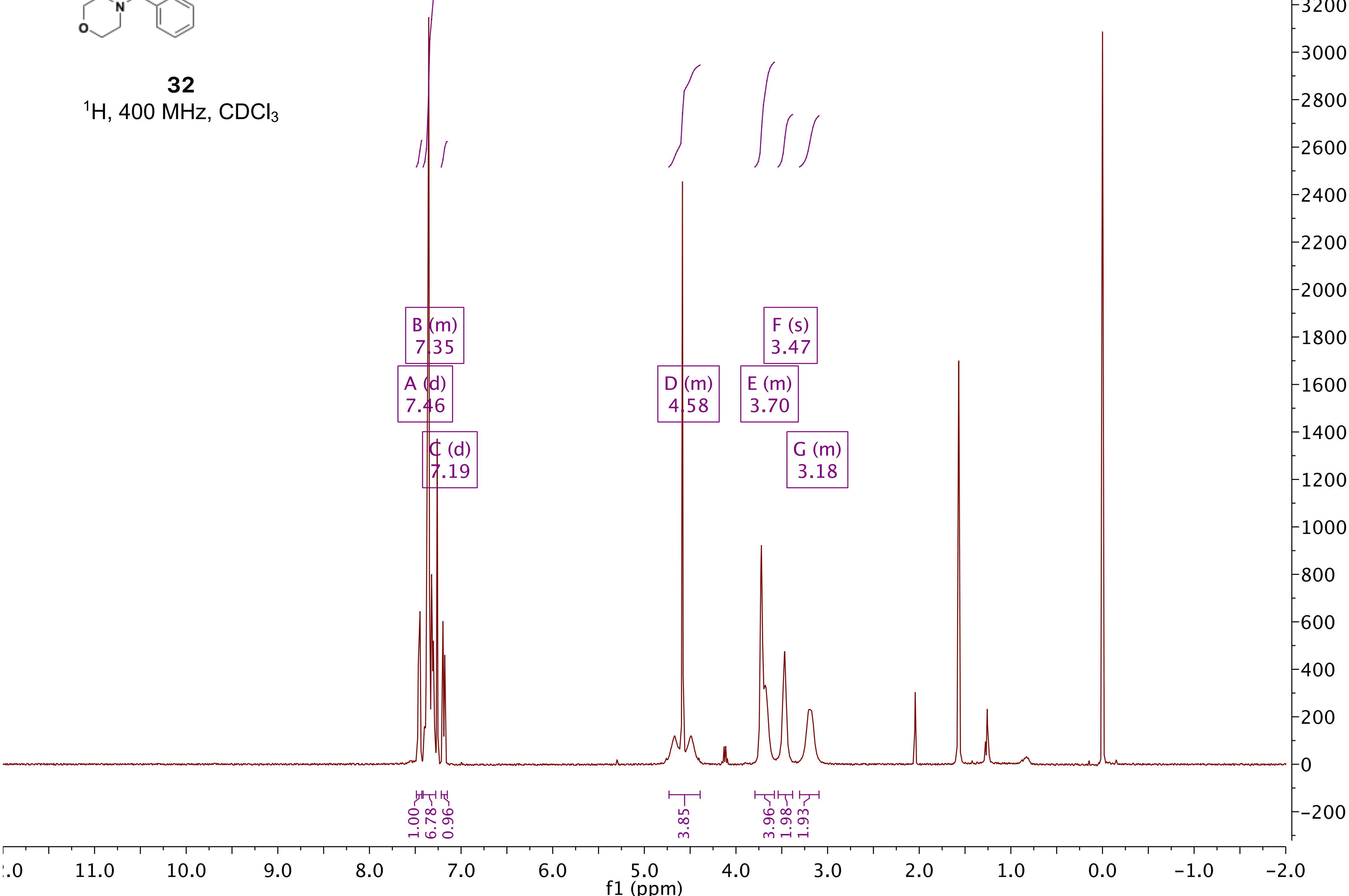
PeakTable C:\LabSolutions\Training1107\LAN\LAN-IV-177c_AD_90_10HIPA6.lcd
PDA Ch1 206nm 4nm

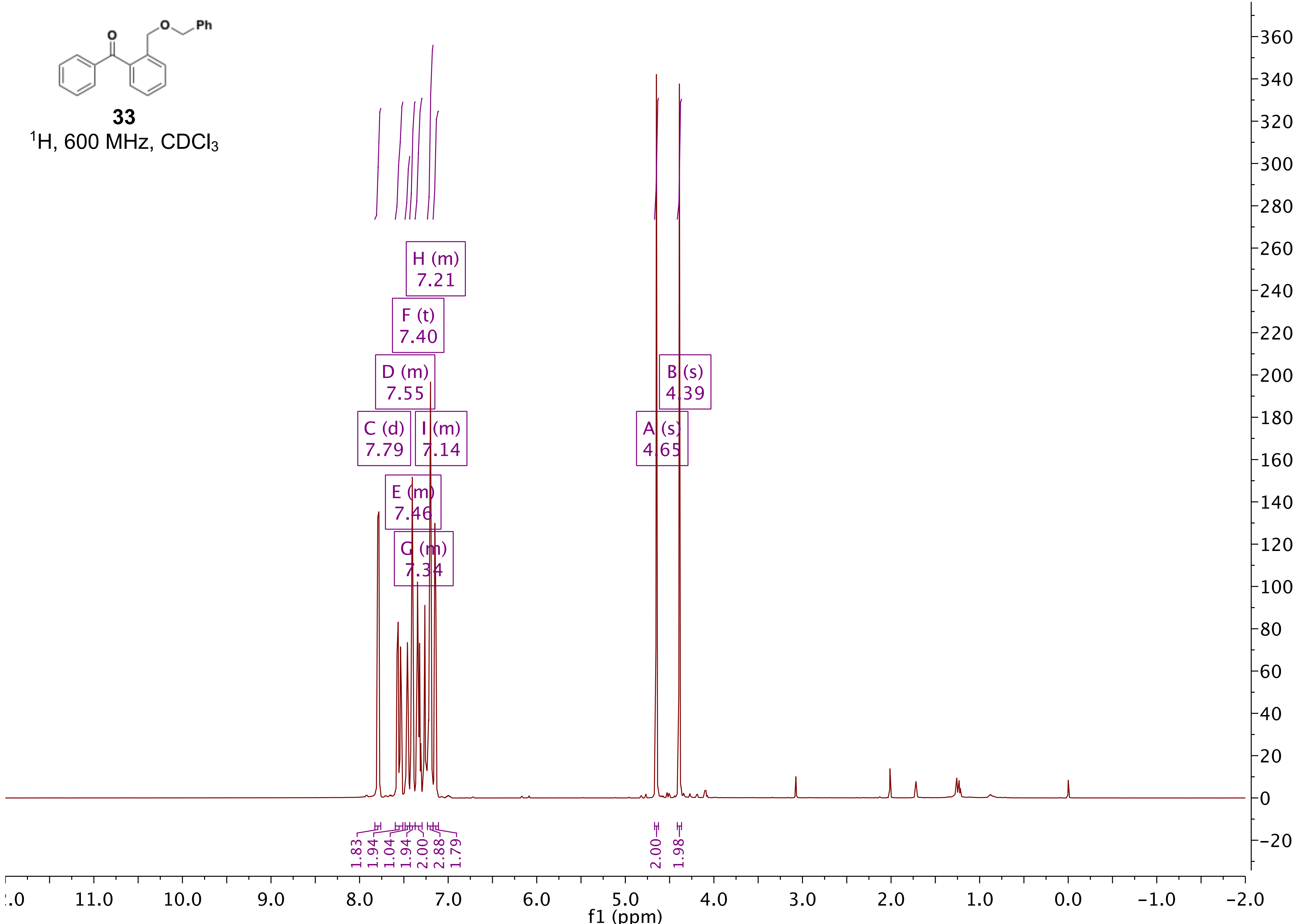
Peak#	Ret. Time	Area	Height	Area %	Height %
1	15.465	8921007	350105	50.563	57.376
2	20.619	8722206	260091	49.437	42.624
Total		17643213	610196	100.000	100.000

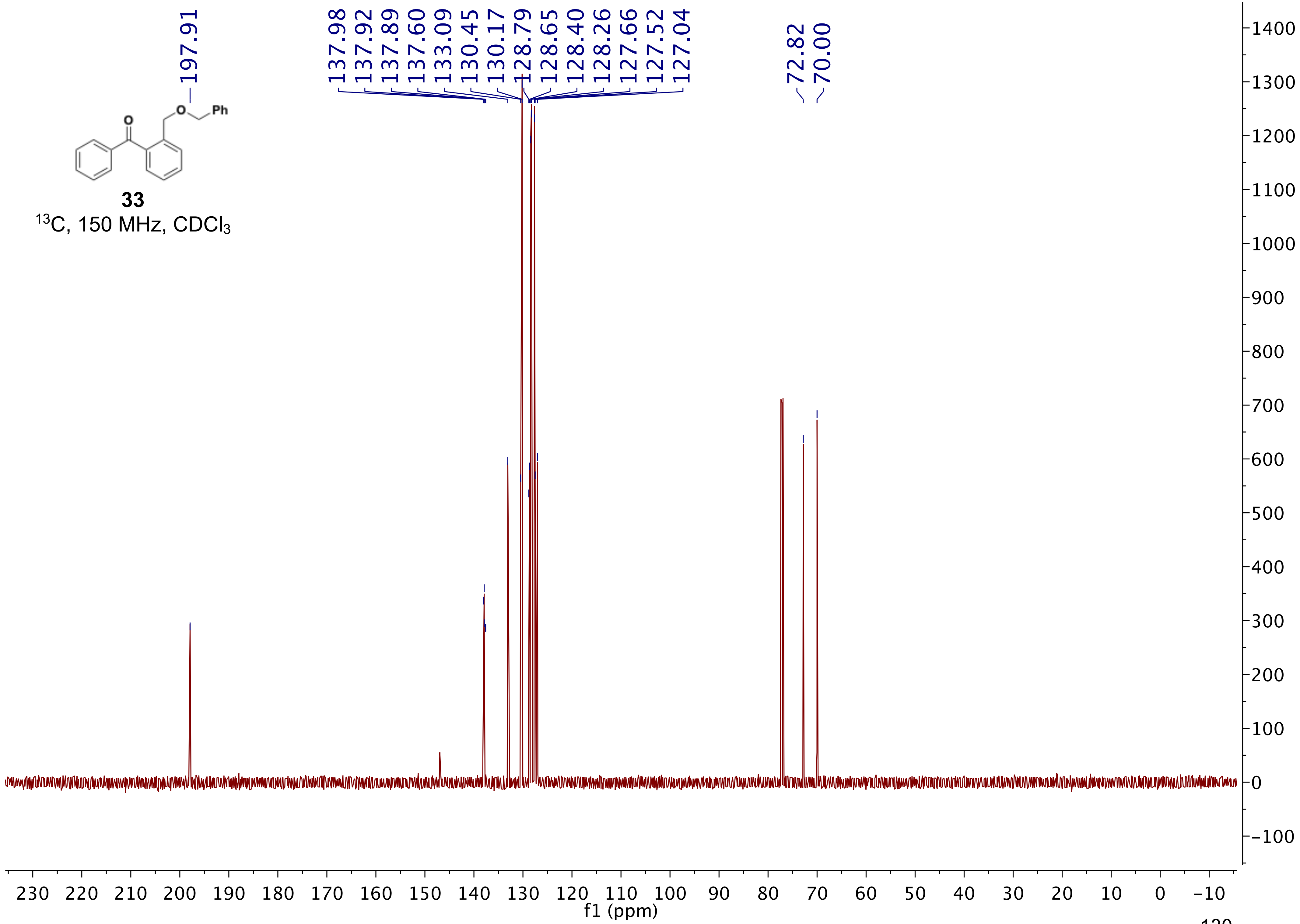


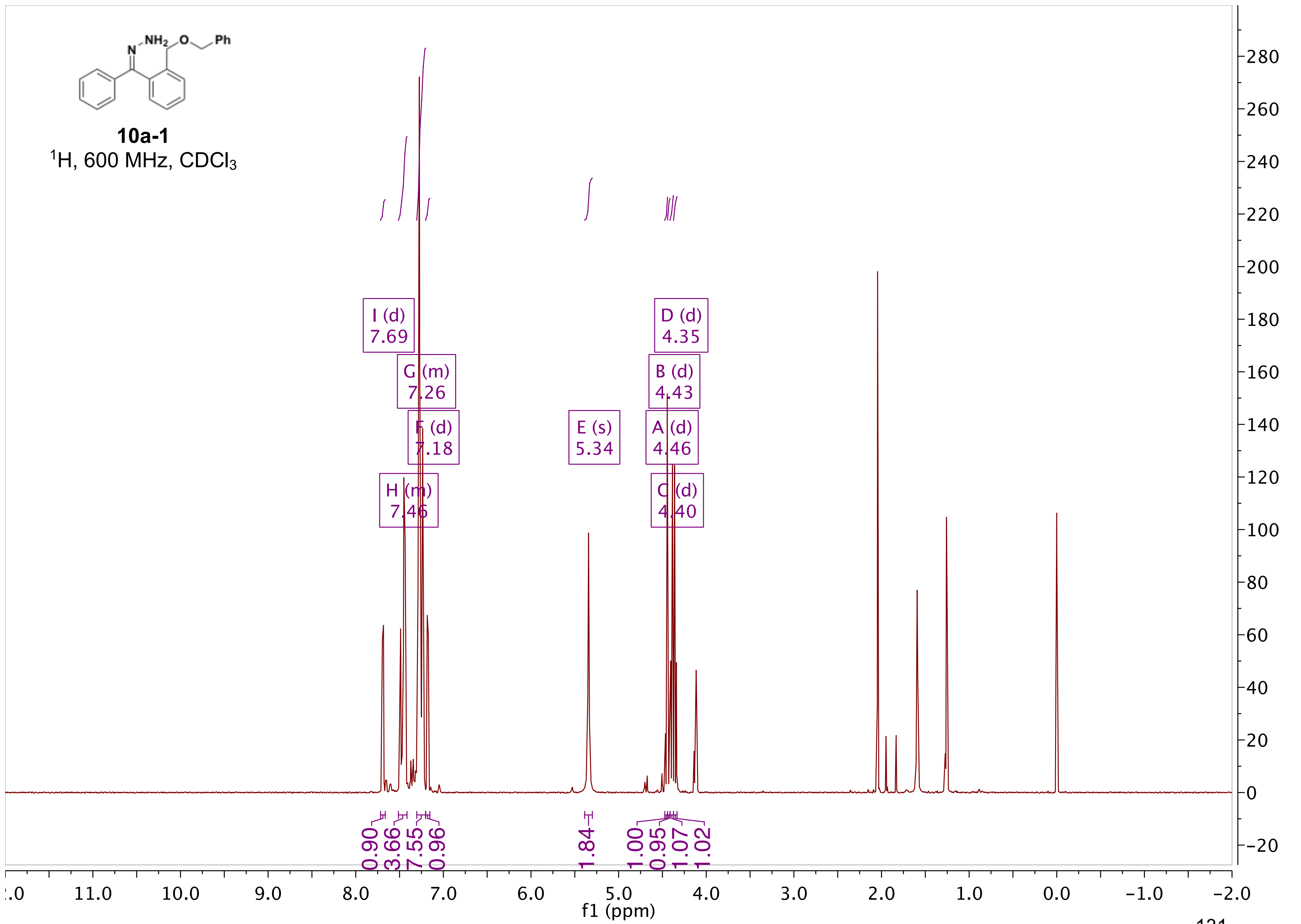
32

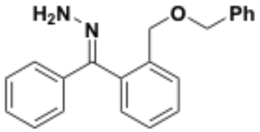
^1H, 400 MHz, $CDCl_3$




Ph
O
O
33
¹H, 600 MHz, CDCl3
H (m) 7.21
F (t) 7.40
D (m) 7.55
C (d) 7.79
I (m) 7.14
E (m) 7.46
G (m) 7.34
B (s) 4.39
A (s) 4.65
1.83
1.94
1.04
1.94
2.00
2.88
1.79
2.00
1.98
f1 (ppm)

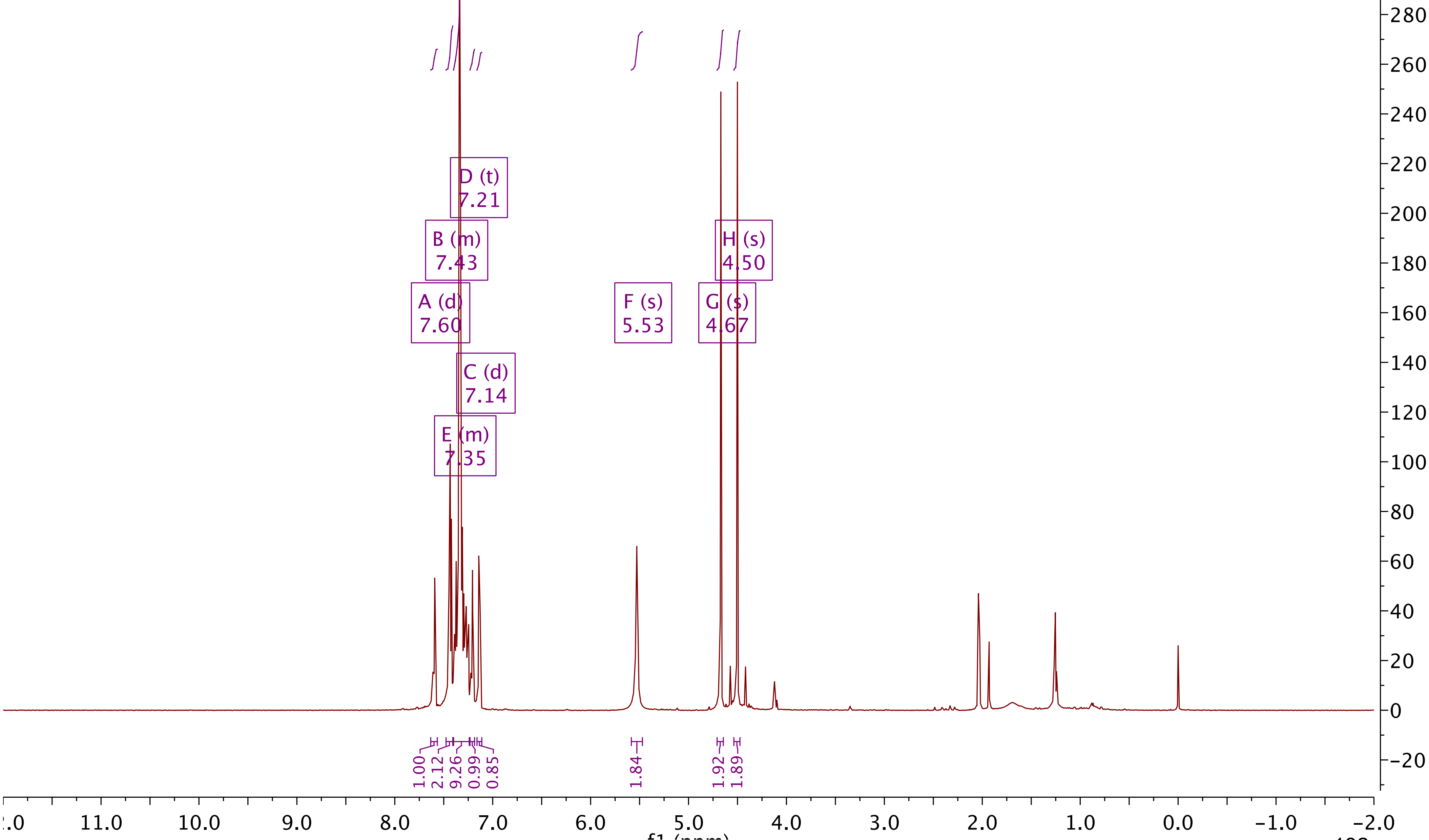

197.91
137.98
137.92
137.89
137.60
133.09
130.45
130.17
128.79
128.65
128.40
128.26
127.66
127.52
127.04
72.82
70.00
O
O
Ph
33
13C, 150 MHz, CDCl3
f1 (ppm)

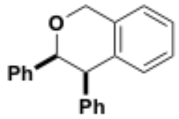

10a-1
1H, 600 MHz, CDCl3
I (d) 7.69
G (m) 7.26
F (d) 7.18
H (m) 7.46
E (s) 5.34
D (d) 4.35
B (d) 4.43
A (d) 4.46
C (d) 4.40
0.90
3.66
7.55
0.96
1.84
1.00
0.95
1.07
1.02
f1 (ppm)



10a-2

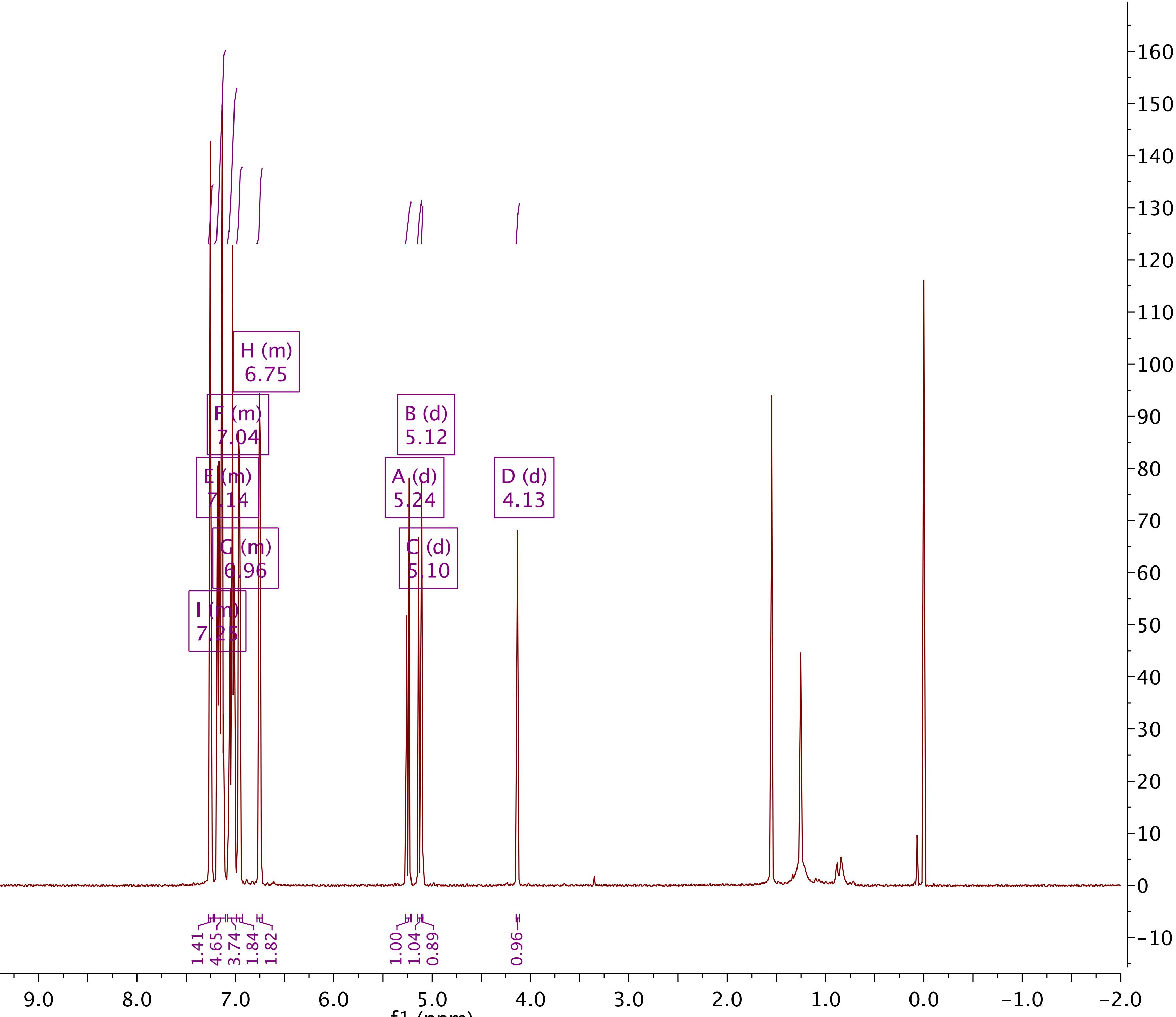
^{1}H, 600 MHz, $CDCl_3$

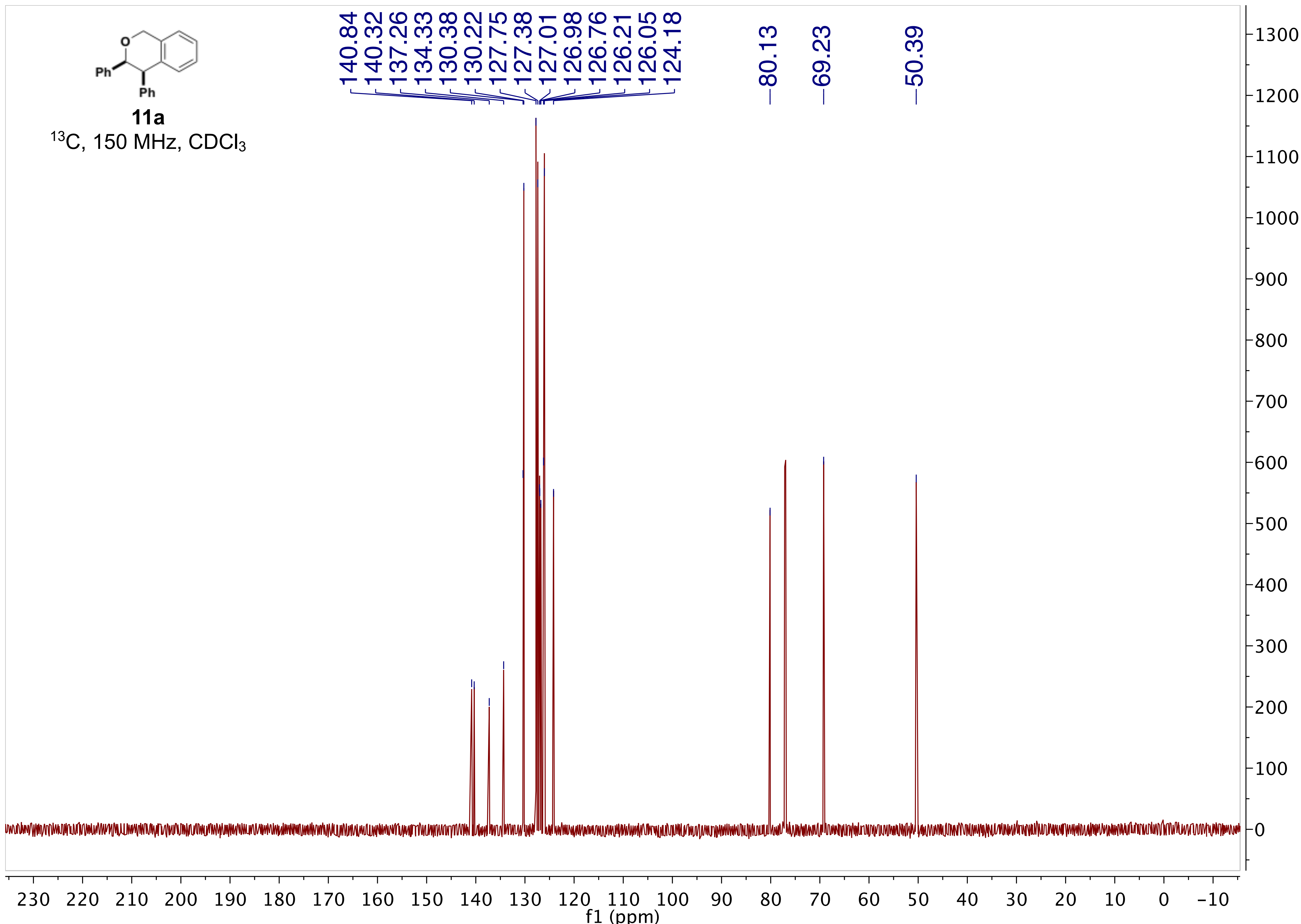


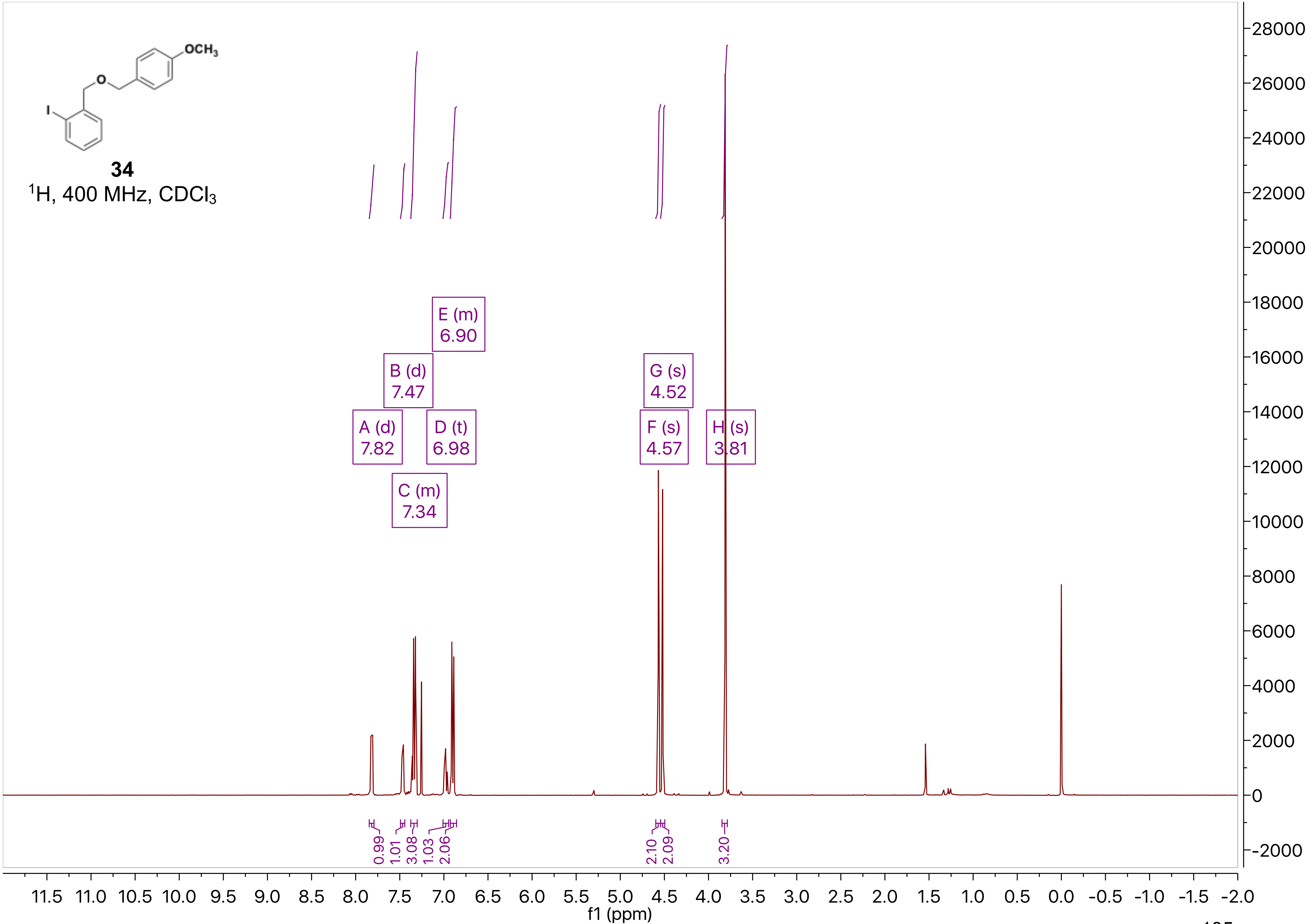


11a

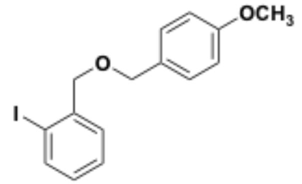
^{1}H, 600 MHz, $CDCl_3$




11a
13C, 150 MHz, CDCl3
140.84
140.32
137.26
134.33
130.38
130.22
127.75
127.38
127.01
126.98
126.76
126.21
126.05
124.18
80.13
69.23
50.39
f1 (ppm)

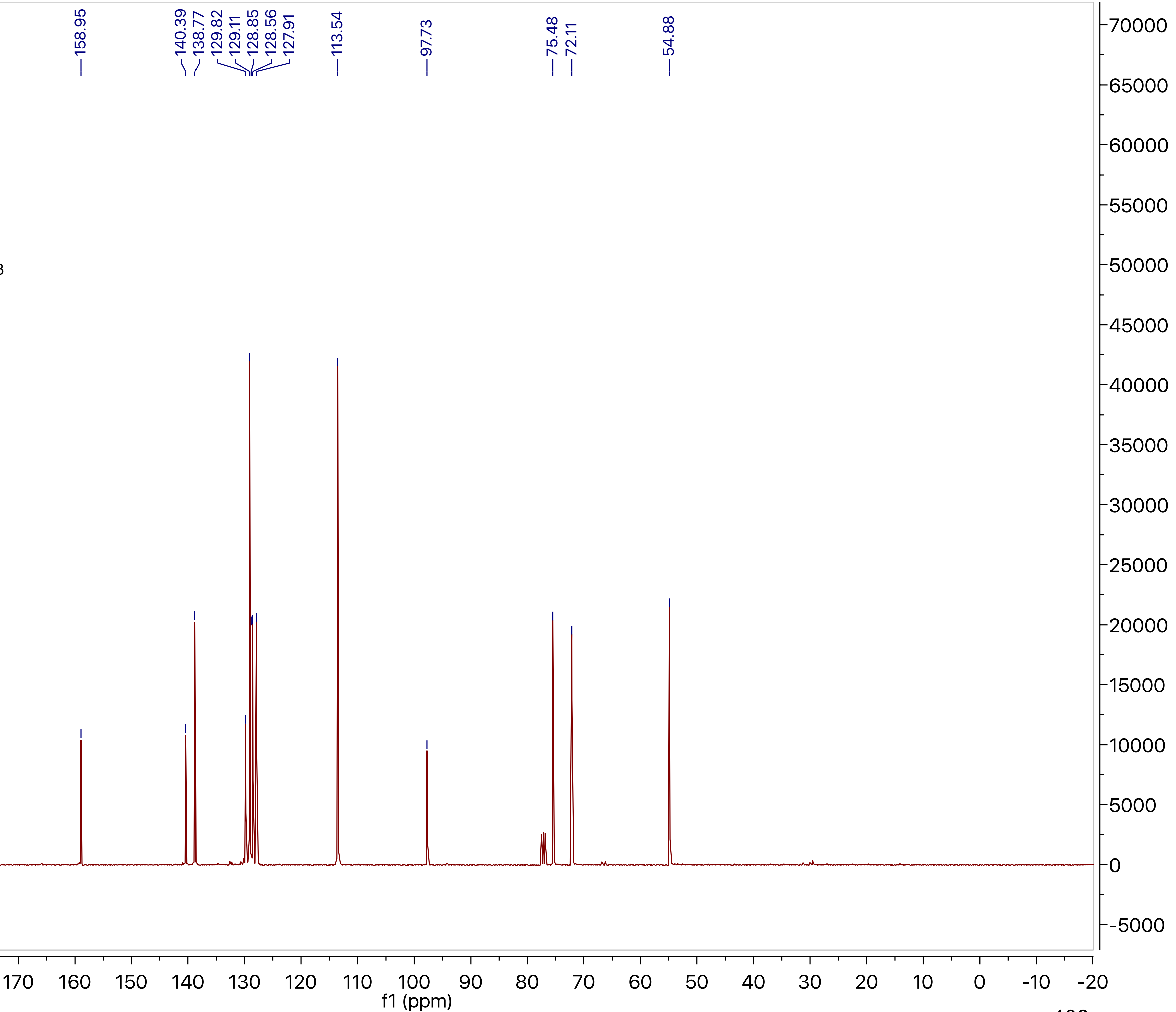


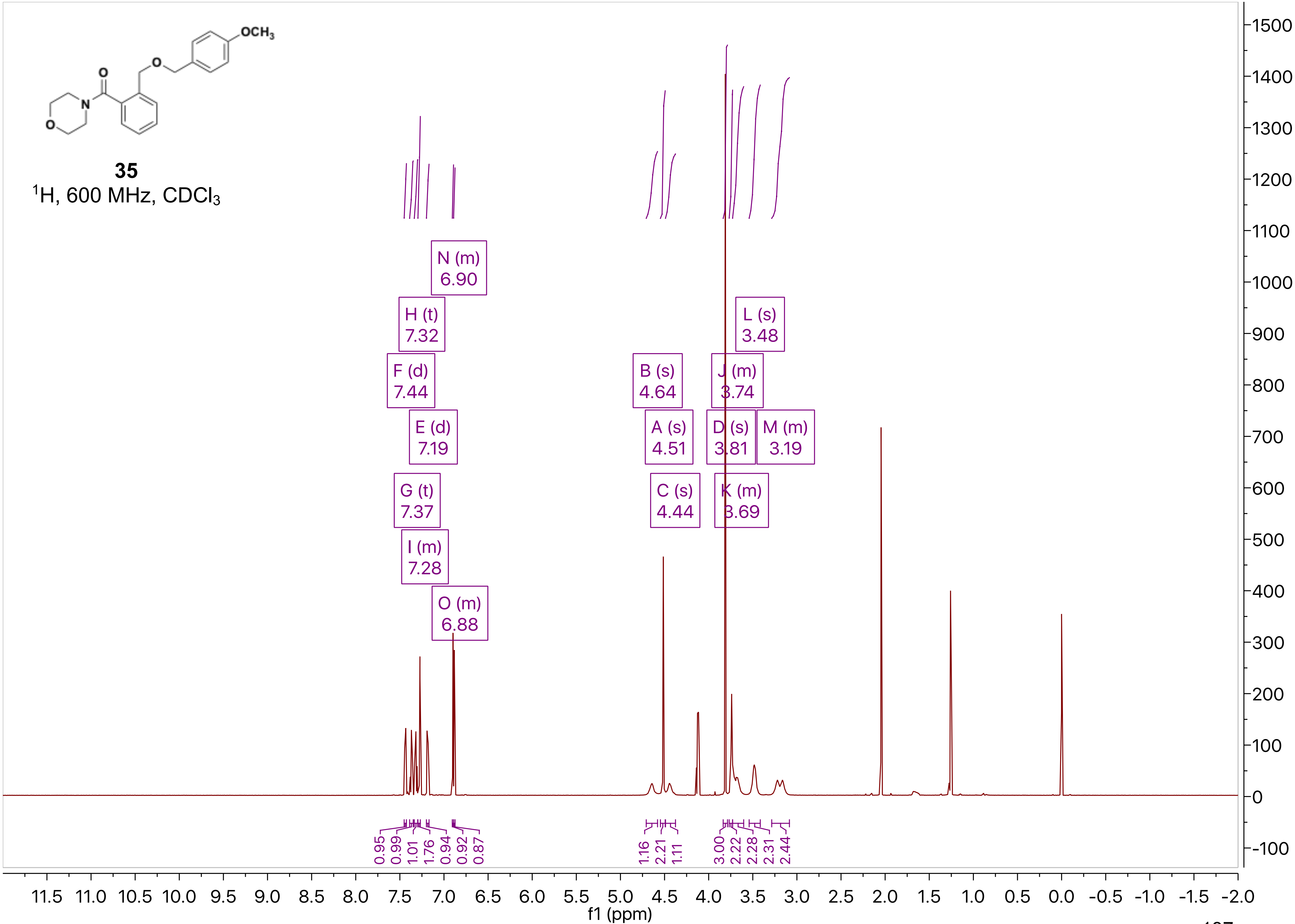
OCH3
O
I
34
1H, 400 MHz, CDCl3
E (m)
6.90
B (d)
7.47
G (s)
4.52
A (d)
7.82
D (t)
6.98
F (s)
4.57
H (s)
3.81
C (m)
7.34
0.99
1.01
3.08
1.03
2.06
2.10
2.09
3.20
f1 (ppm)

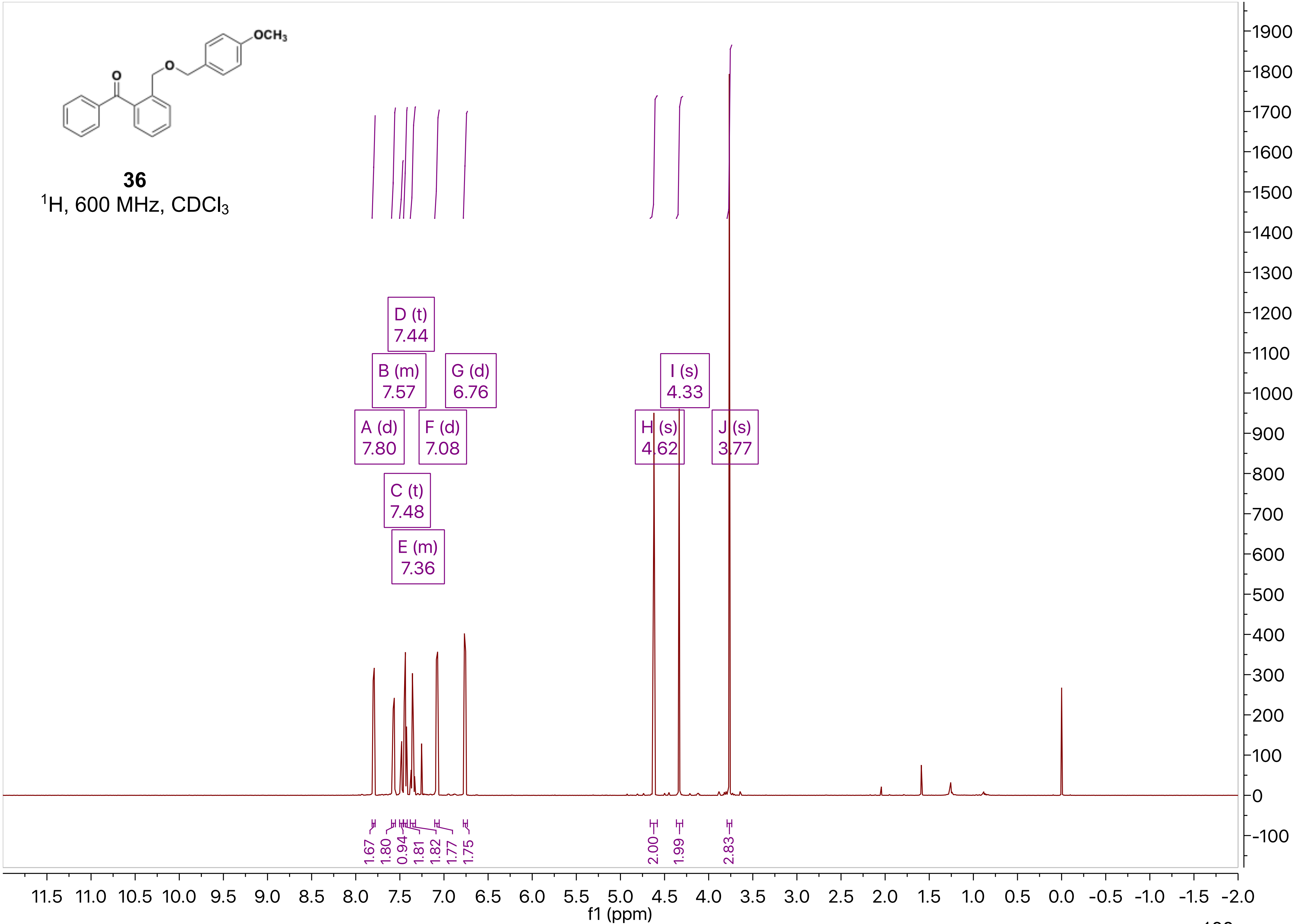


34

^{13}C, 100 MHz, $CDCl_3$

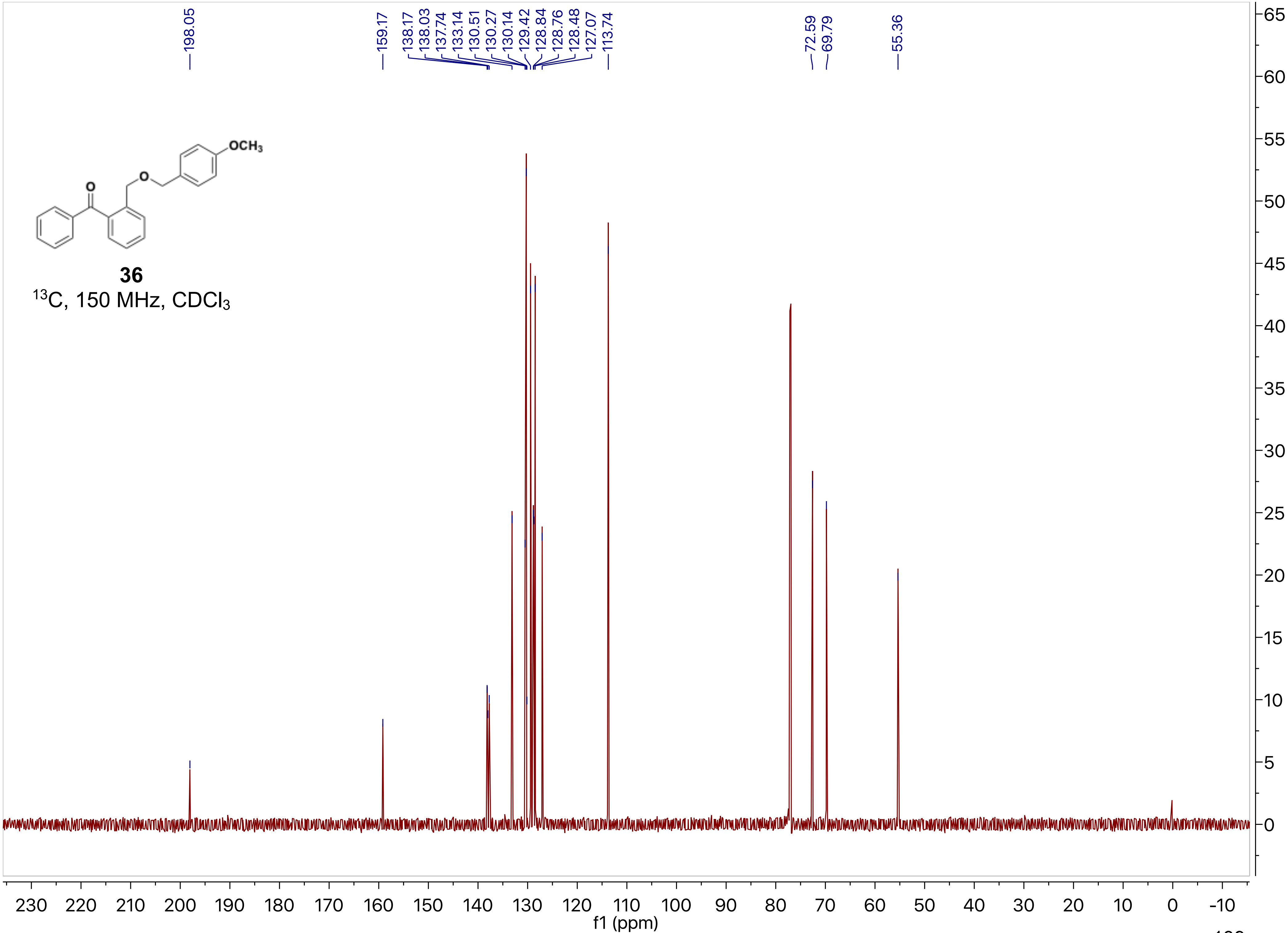


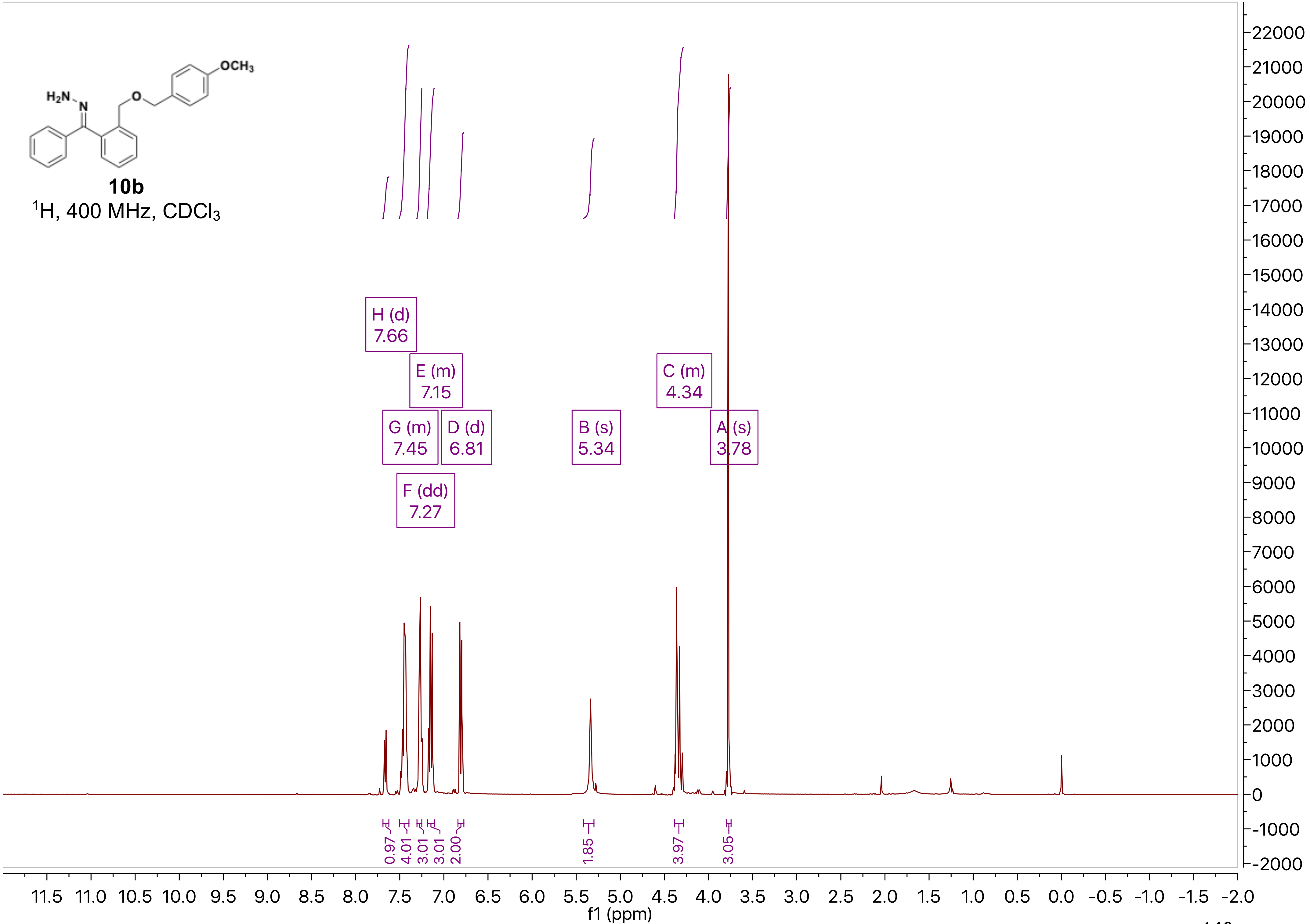

OCH3
O
N
O
O
35
1H, 600 MHz, CDCl3
N (m) 6.90
H (t) 7.32
F (d) 7.44
E (d) 7.19
G (t) 7.37
I (m) 7.28
O (m) 6.88
B (s) 4.64
A (s) 4.51
C (s) 4.44
L (s) 3.48
J (m) 3.74
D (s) 3.81
M (m) 3.19
K (m) 3.69
0.95 0.99 1.01 1.76 0.94 0.92 0.87
1.16 2.21 1.11
3.00 2.22 2.28 2.31 2.44
f1 (ppm)

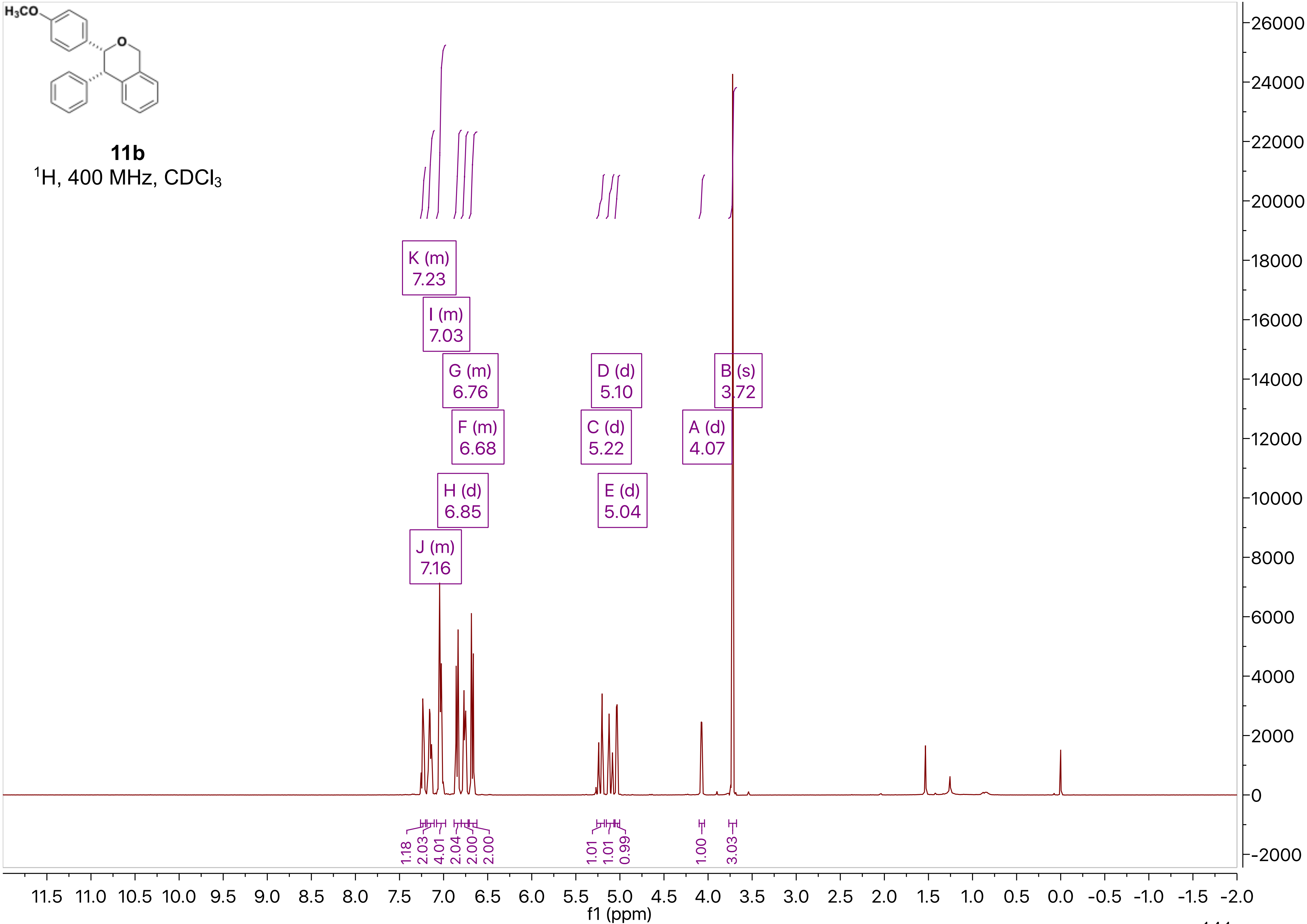


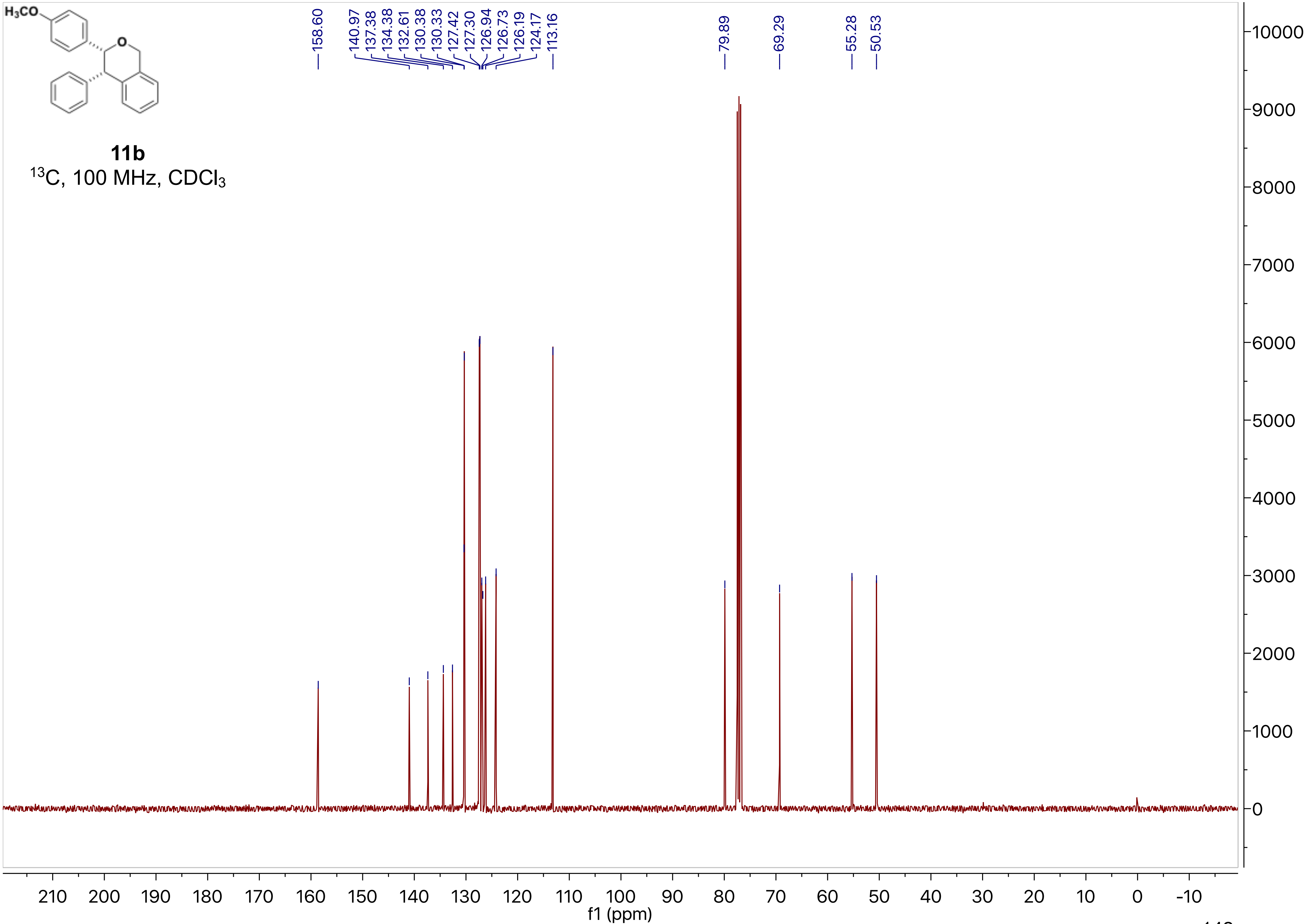
36

^{1}H, 600 MHz, $CDCl_3$


OCH3
O
O
36
13C, 150 MHz, CDCl3
198.05
159.17
138.17
138.03
137.74
133.14
130.51
130.27
130.14
129.42
128.84
128.76
128.48
127.07
113.74
72.59
69.79
55.36
f1 (ppm)

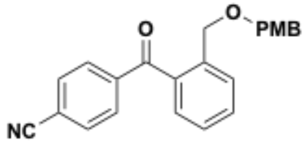

10b
¹H, 400 MHz, CDCl₃
H (d) 7.66
E (m) 7.15
G (m) 7.45
D (d) 6.81
F (dd) 7.27
B (s) 5.34
C (m) 4.34
A (s) 3.78
0.97
4.01
3.01
3.01
2.00
1.85
3.97
3.05
f1 (ppm)


H3CO
O
11b
1H, 400 MHz, CDCl3
K (m) 7.23
I (m) 7.03
G (m) 6.76
F (m) 6.68
H (d) 6.85
J (m) 7.16
D (d) 5.10
C (d) 5.22
E (d) 5.04
A (d) 4.07
B (s) 3.72
1.18
2.03
4.01
2.04
2.00
2.00
1.01
1.01
0.99
1.00
3.03
f1 (ppm)



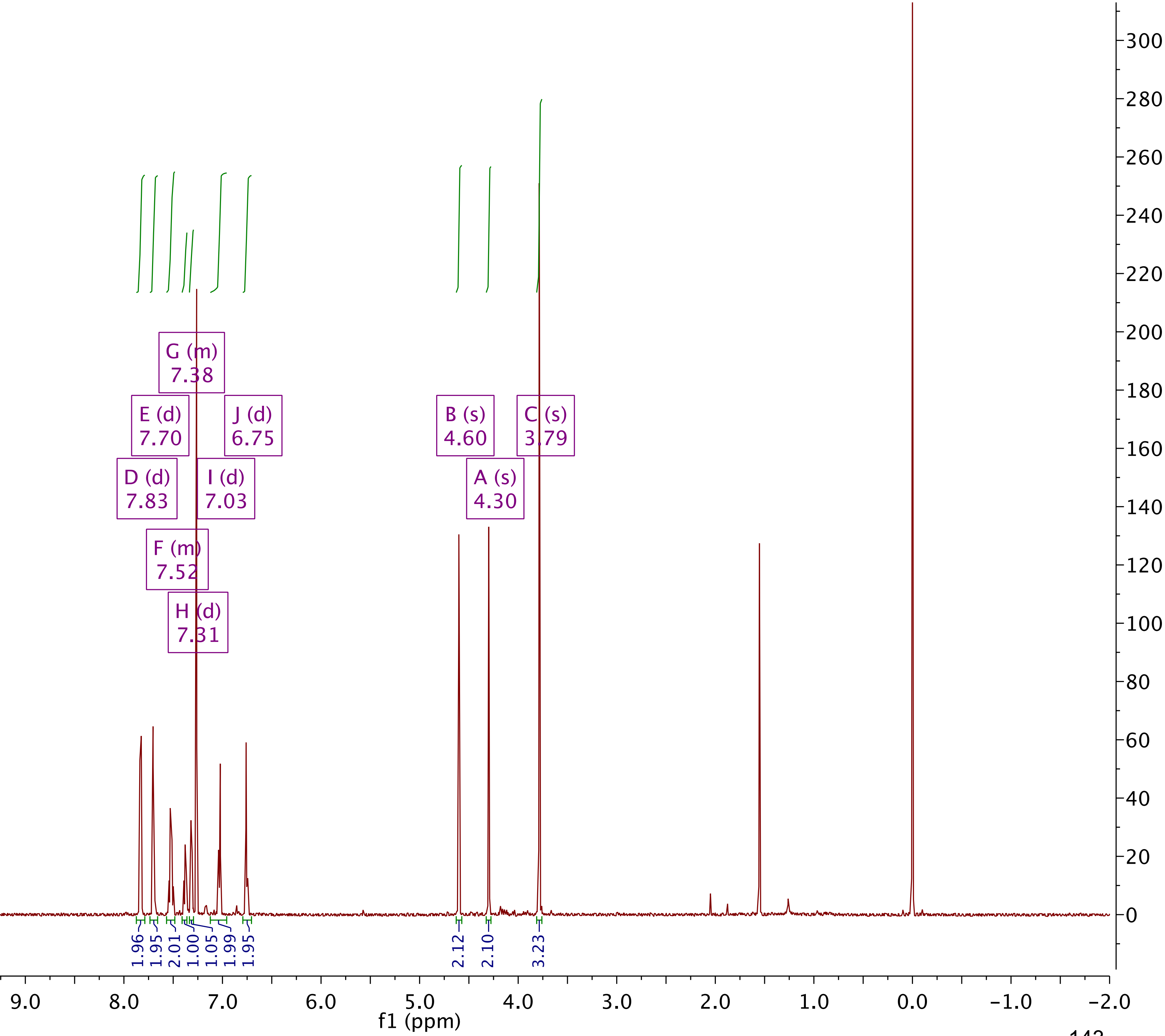
11b

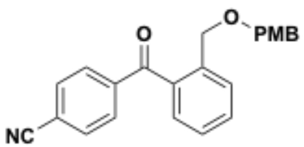
^{13}C, 100 MHz, $CDCl_3$



39

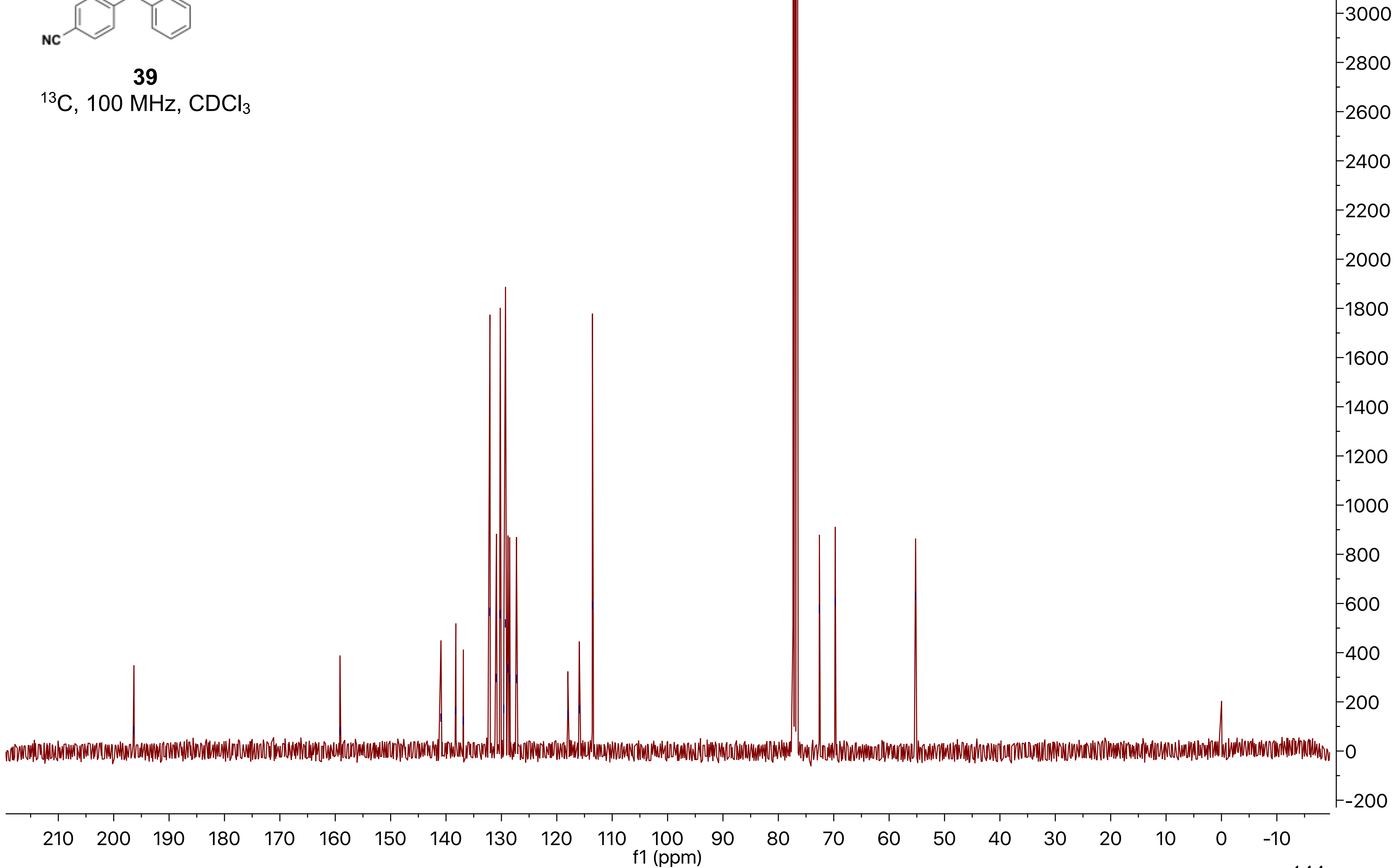
^{1}H, 600 MHz, $CDCl_3$

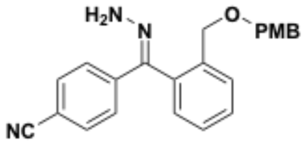




39

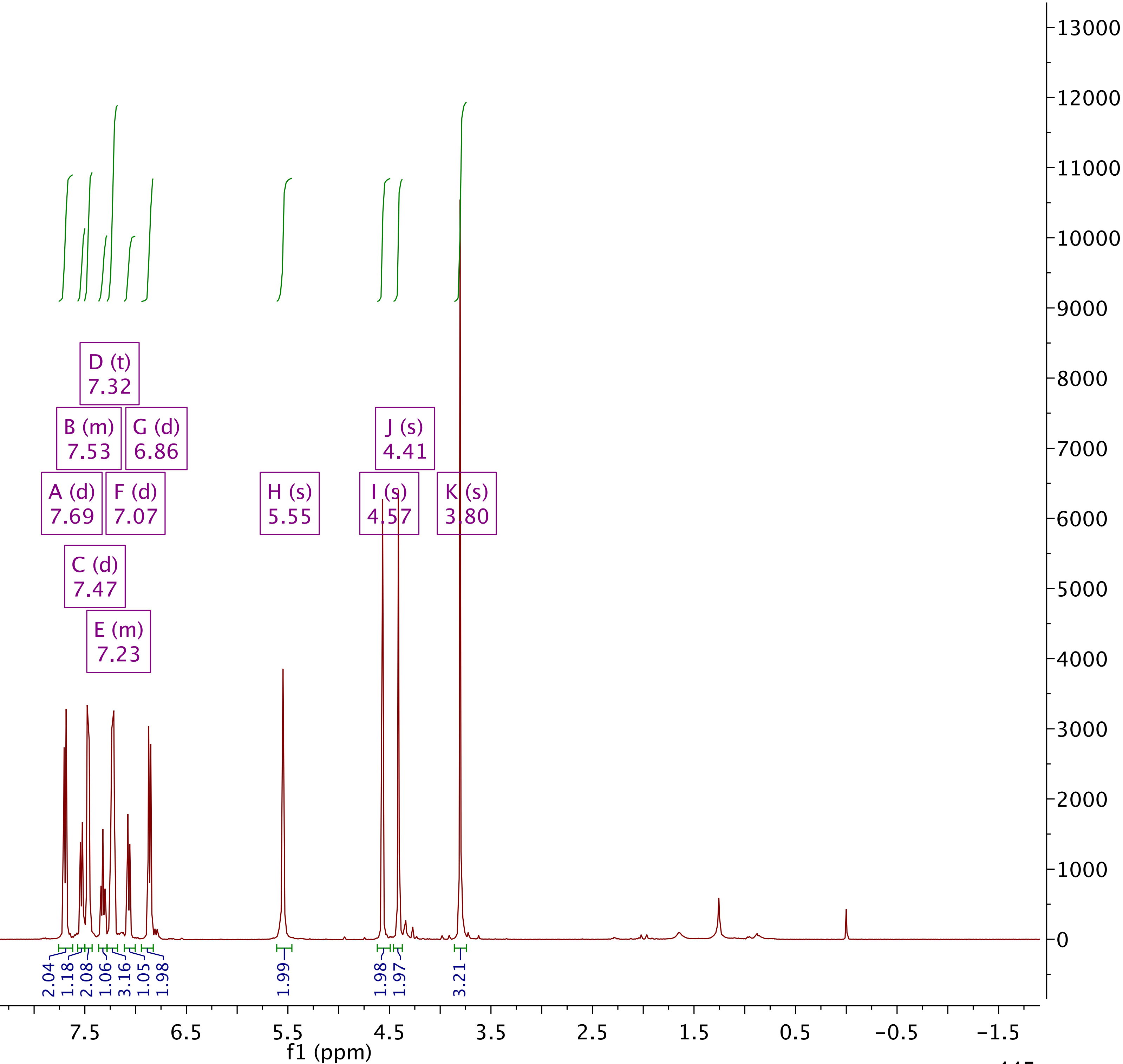
^{13}C, 100 MHz, $CDCl_3$

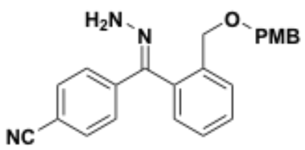




10c-1

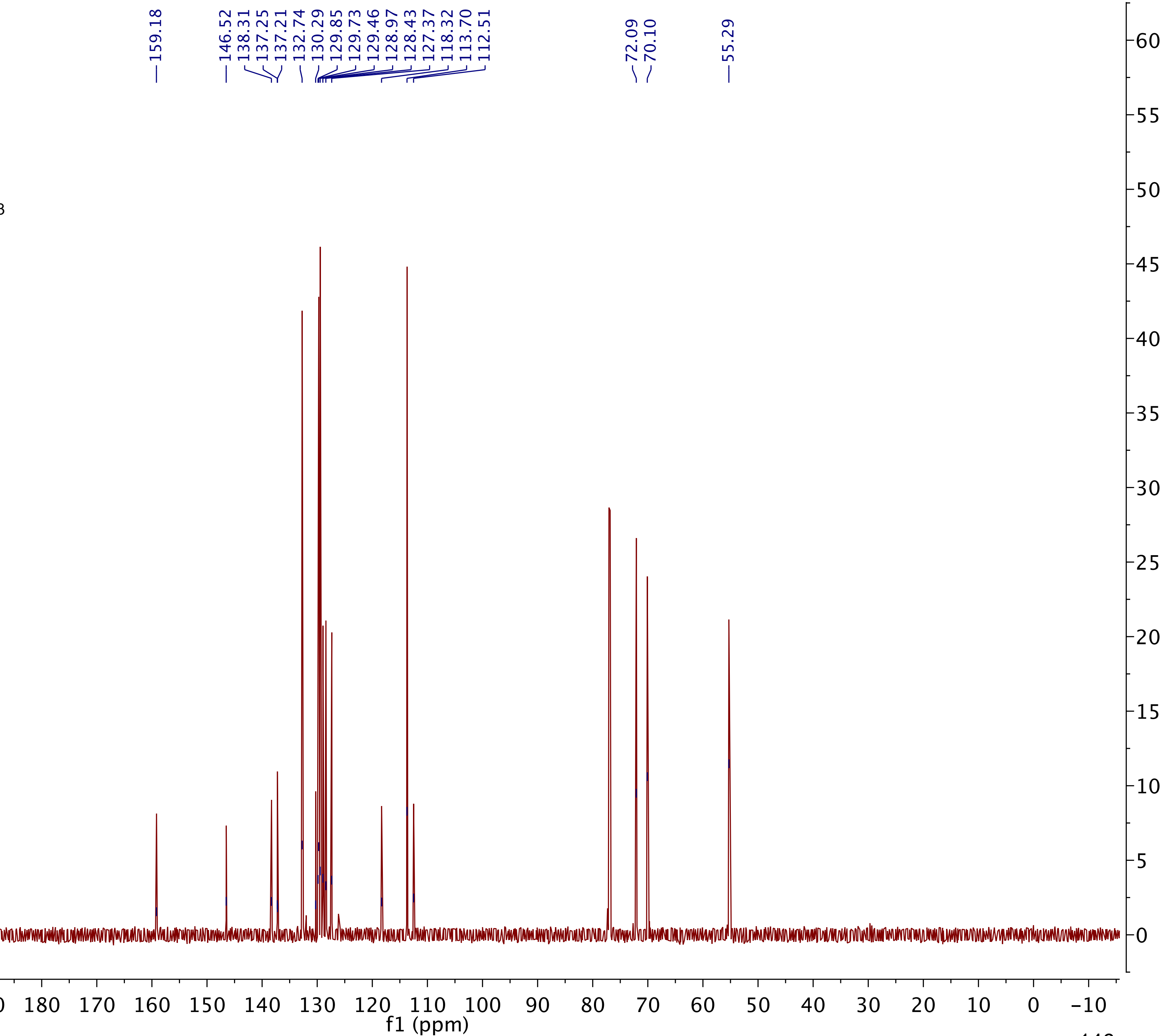
^{1}H, 600 MHz, $CDCl_3$

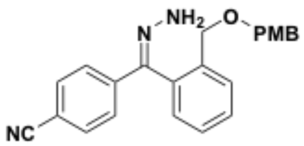




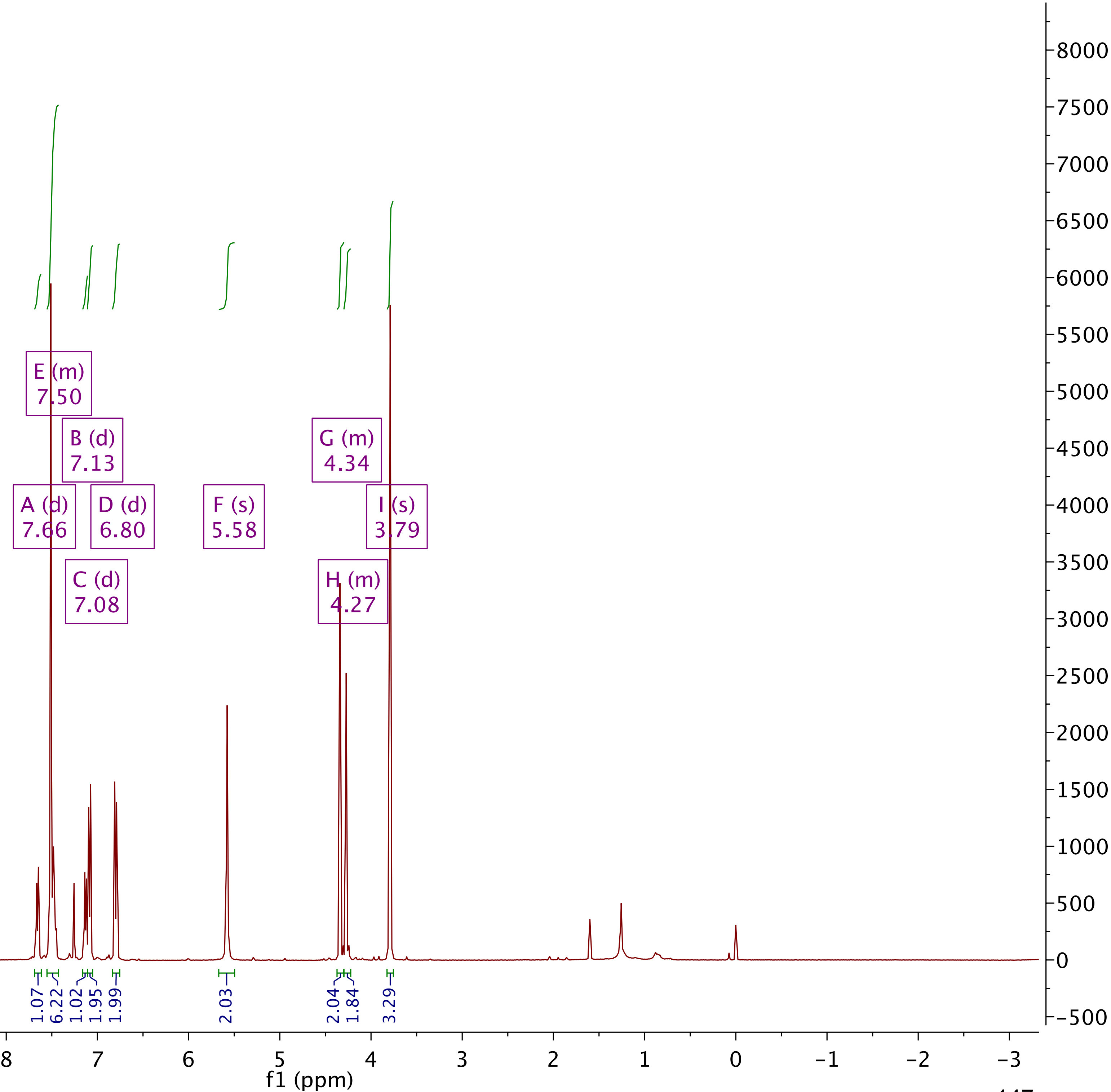
10c-1

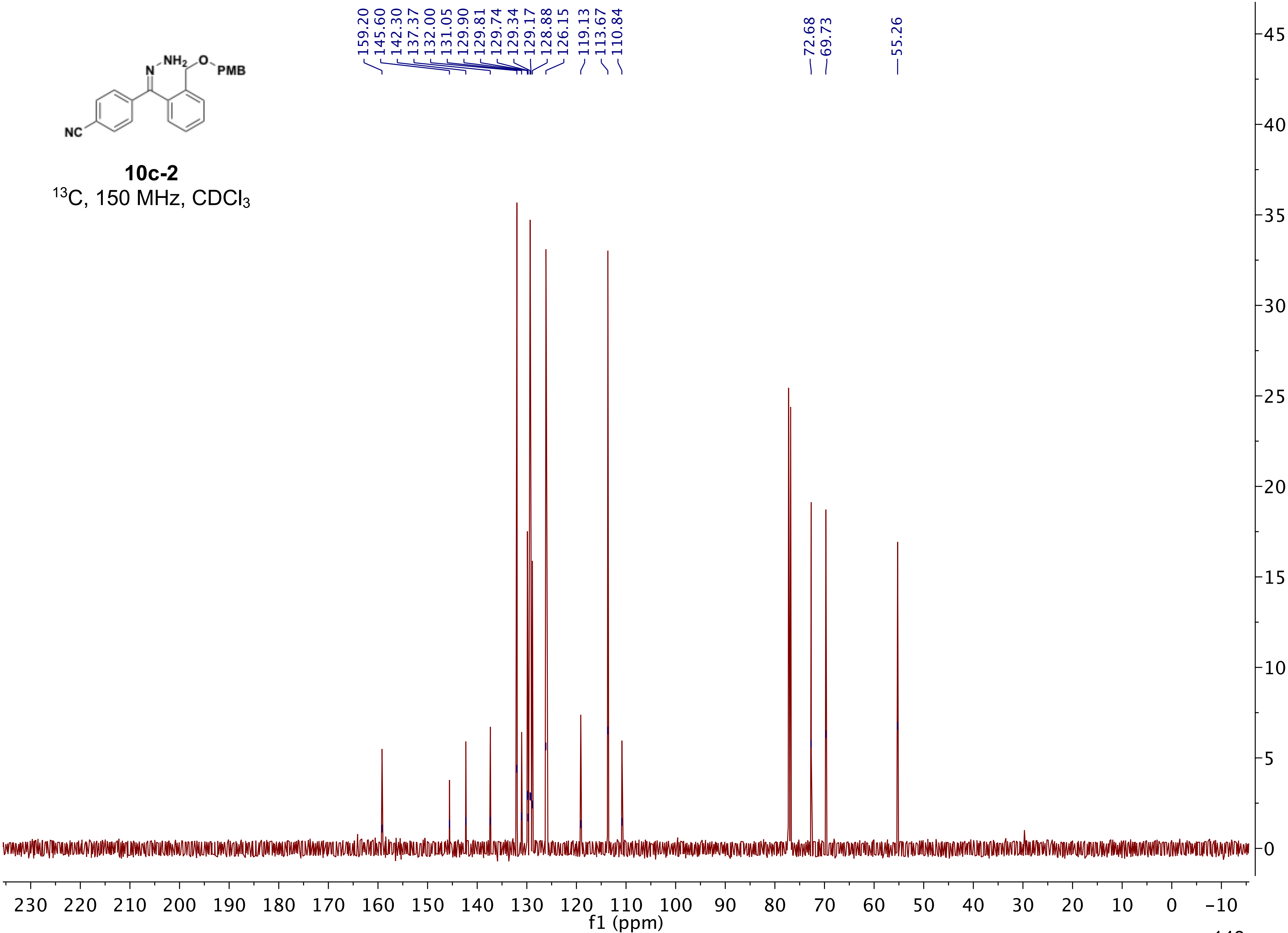
^{13}C, 150 MHz, $CDCl_3$



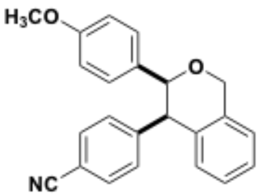


10c-2
^{1}H, 400 MHz, $CDCl_3$



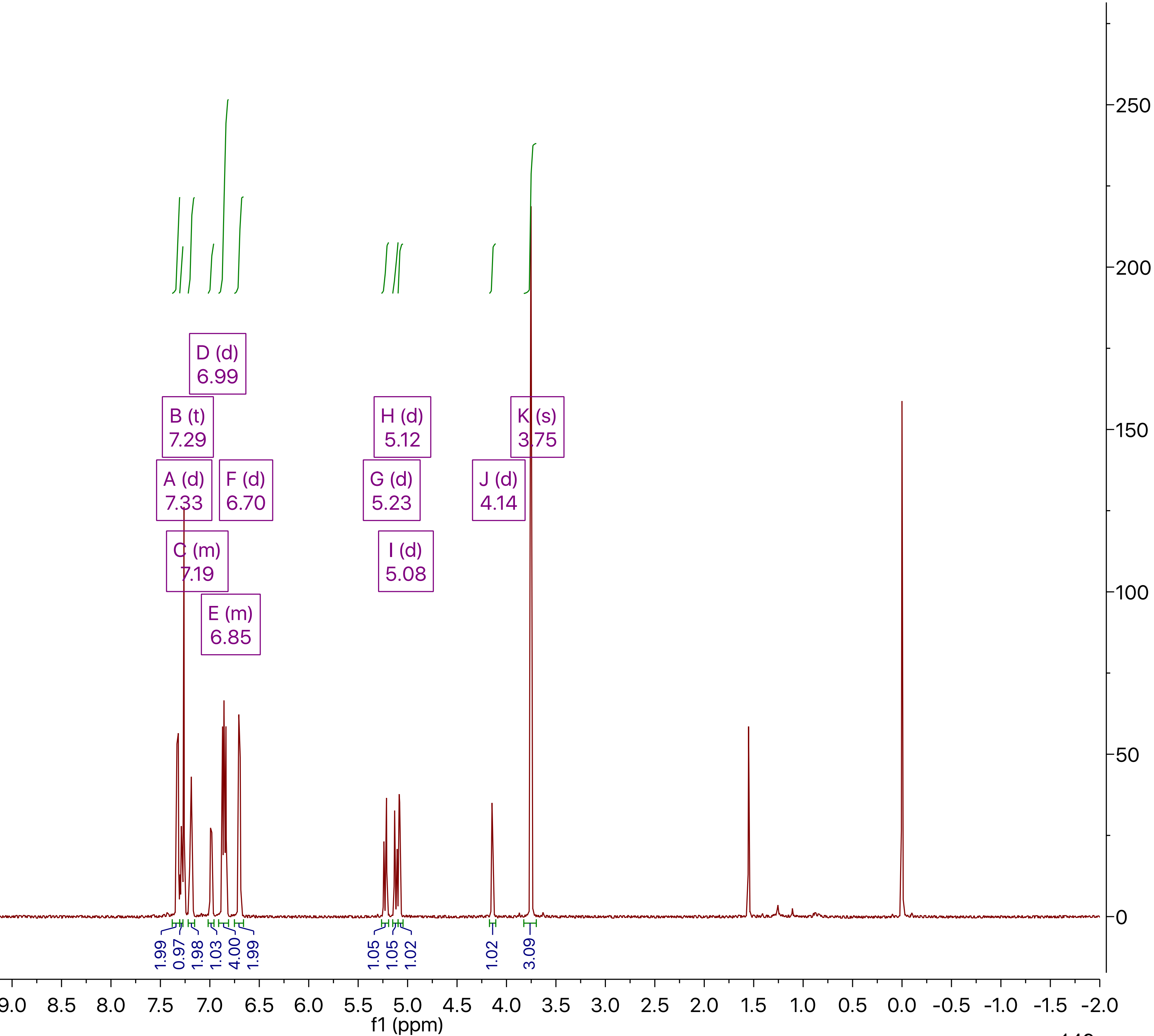


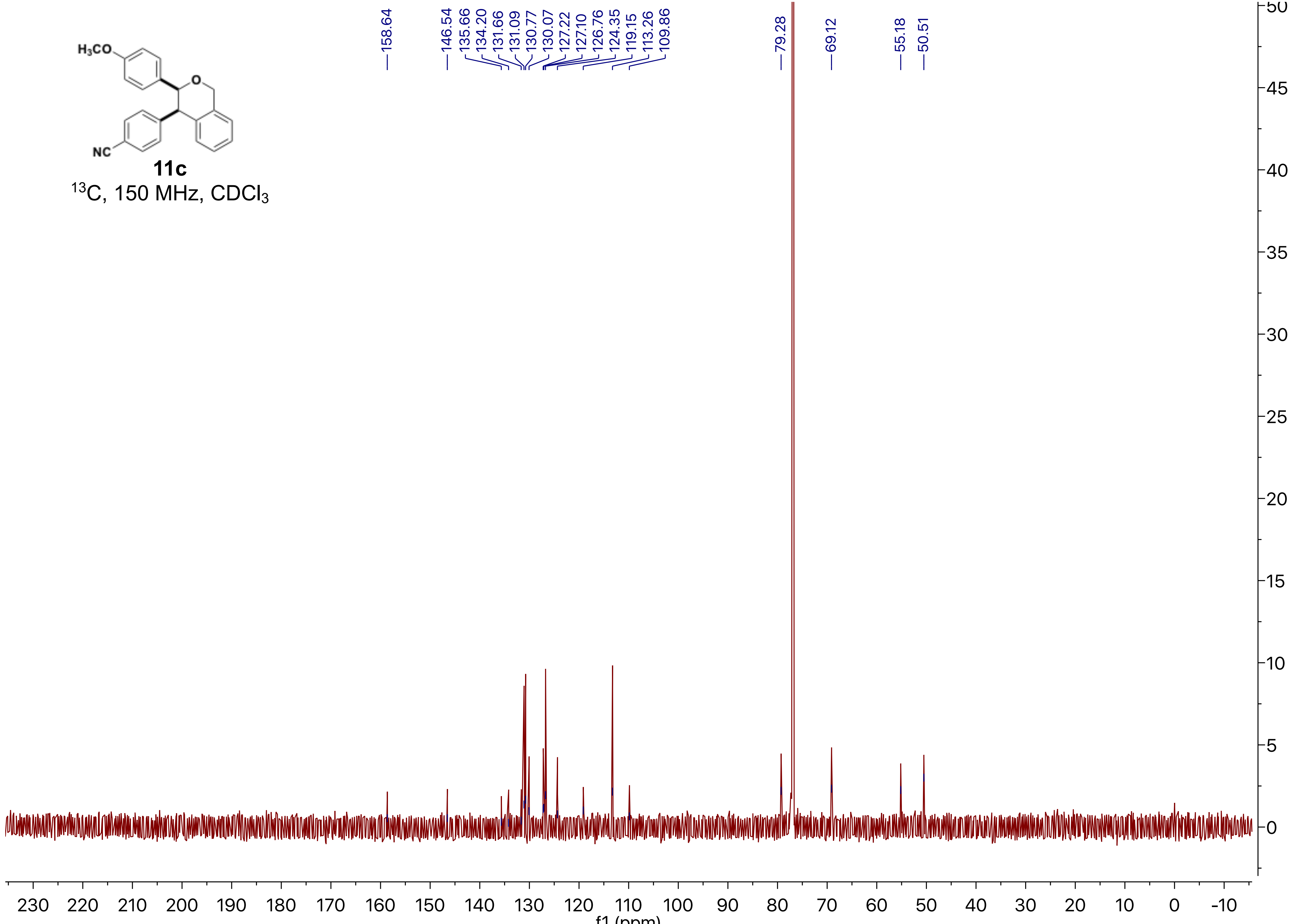
^{13}C, 150 MHz, $CDCl_3$



11c

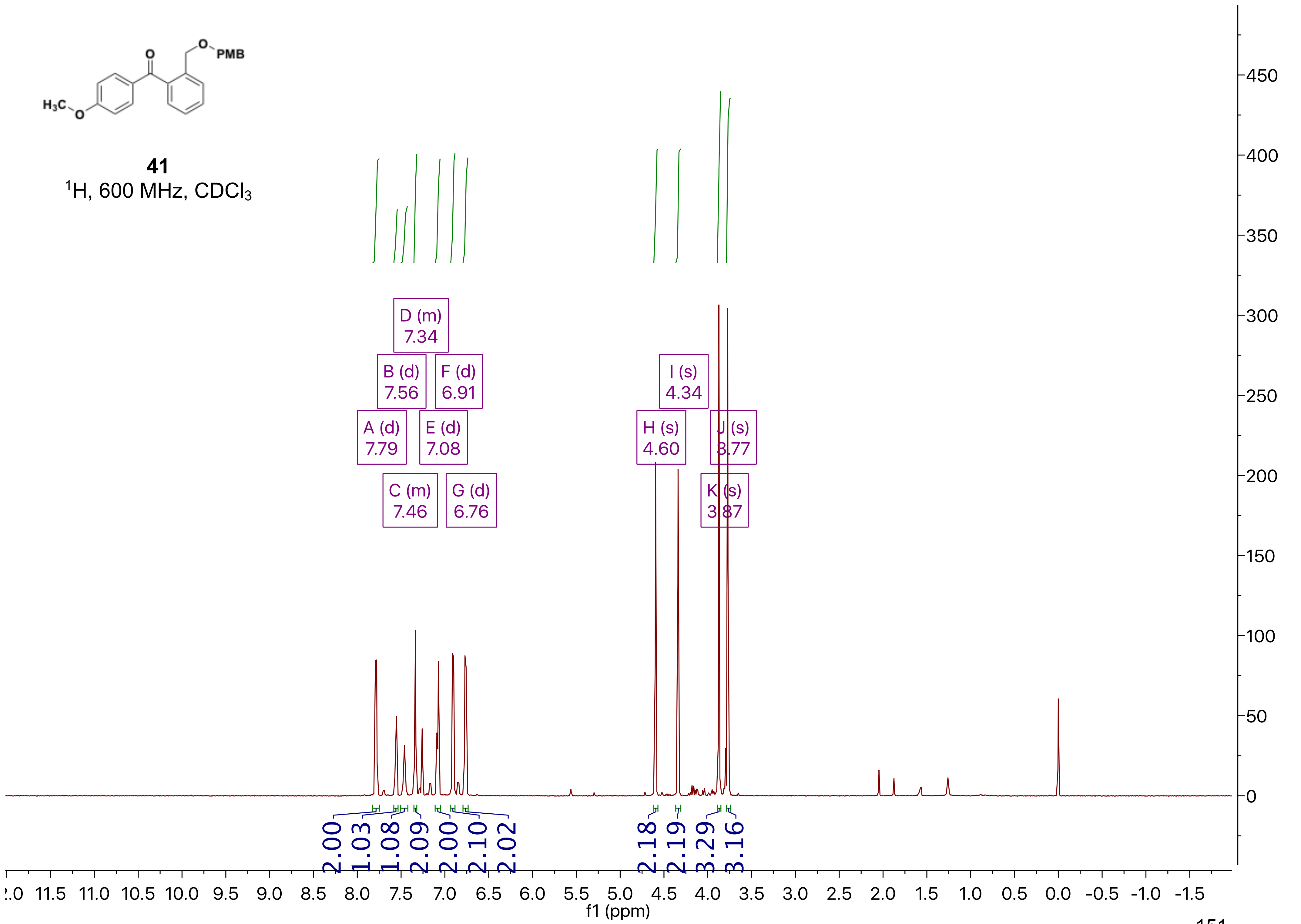
^{1}H, 600 MHz, $CDCl_3$



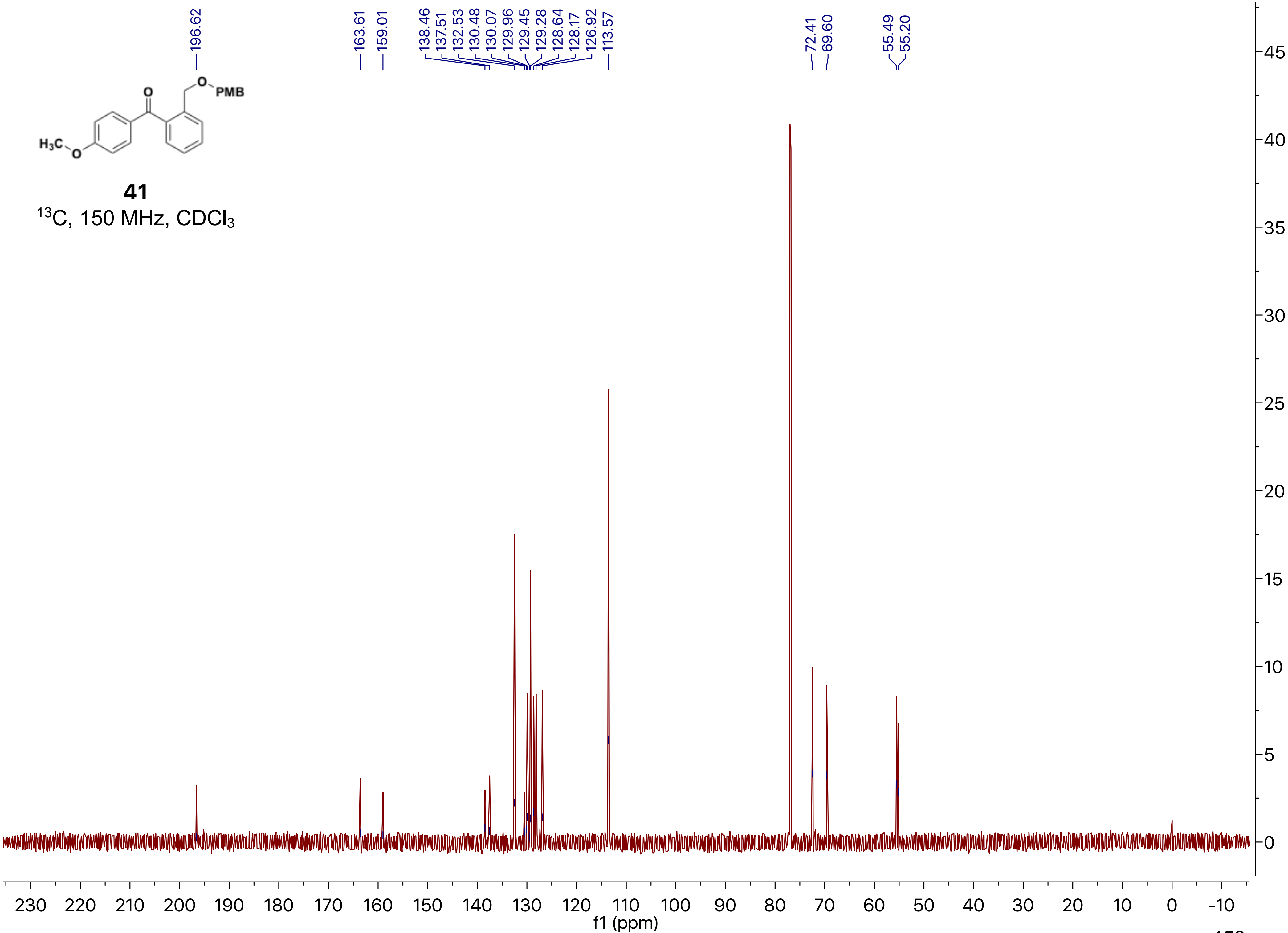


11c

^{13}C, 150 MHz, CDCl$_3$

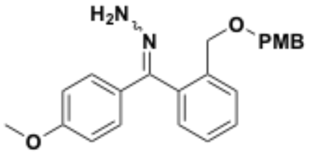


O
O
PMB
H3C
O
41
1H, 600 MHz, CDCl3
D (m)
7.34
B (d)
7.56
F (d)
6.91
A (d)
7.79
E (d)
7.08
C (m)
7.46
G (d)
6.76
I (s)
4.34
H (s)
4.60
J (s)
3.77
K (s)
3.87
2.00
1.03
1.08
2.09
2.00
2.10
2.02
2.18
2.19
3.29
3.16
f1 (ppm)



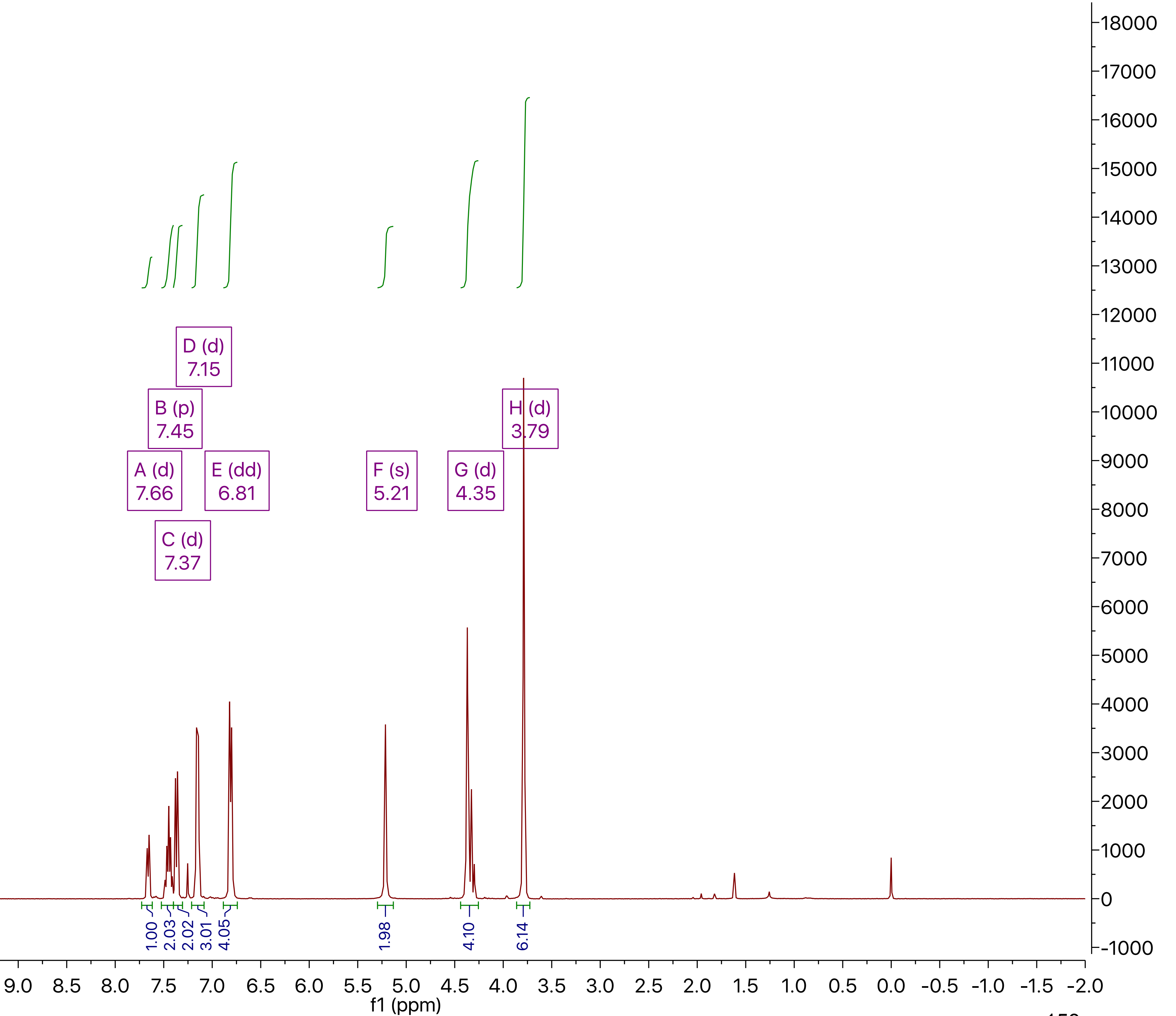
41

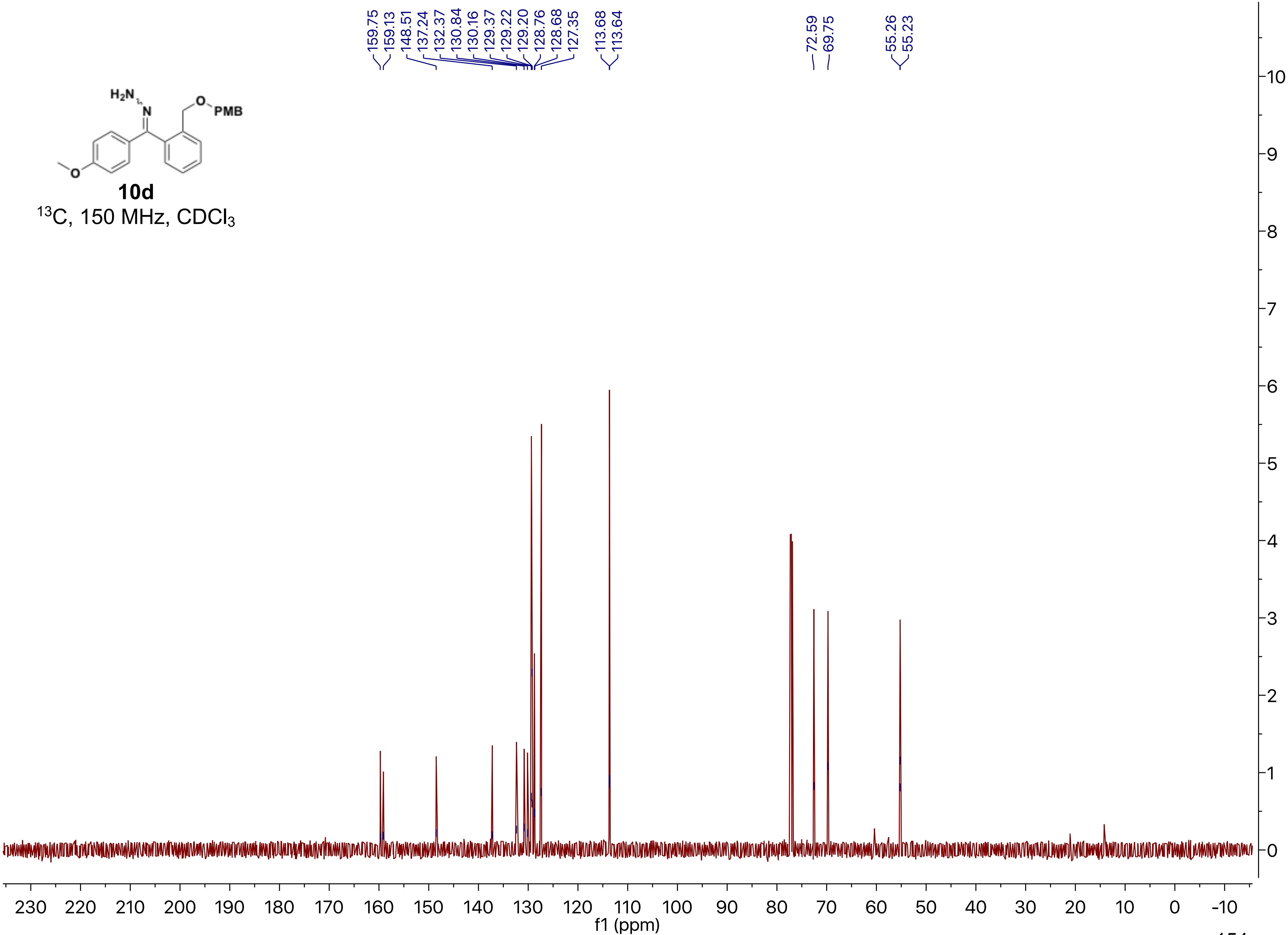
^{13}C, 150 MHz, $CDCl_3$



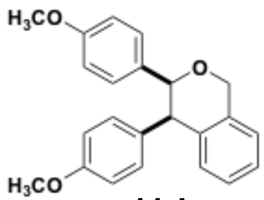
10d

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



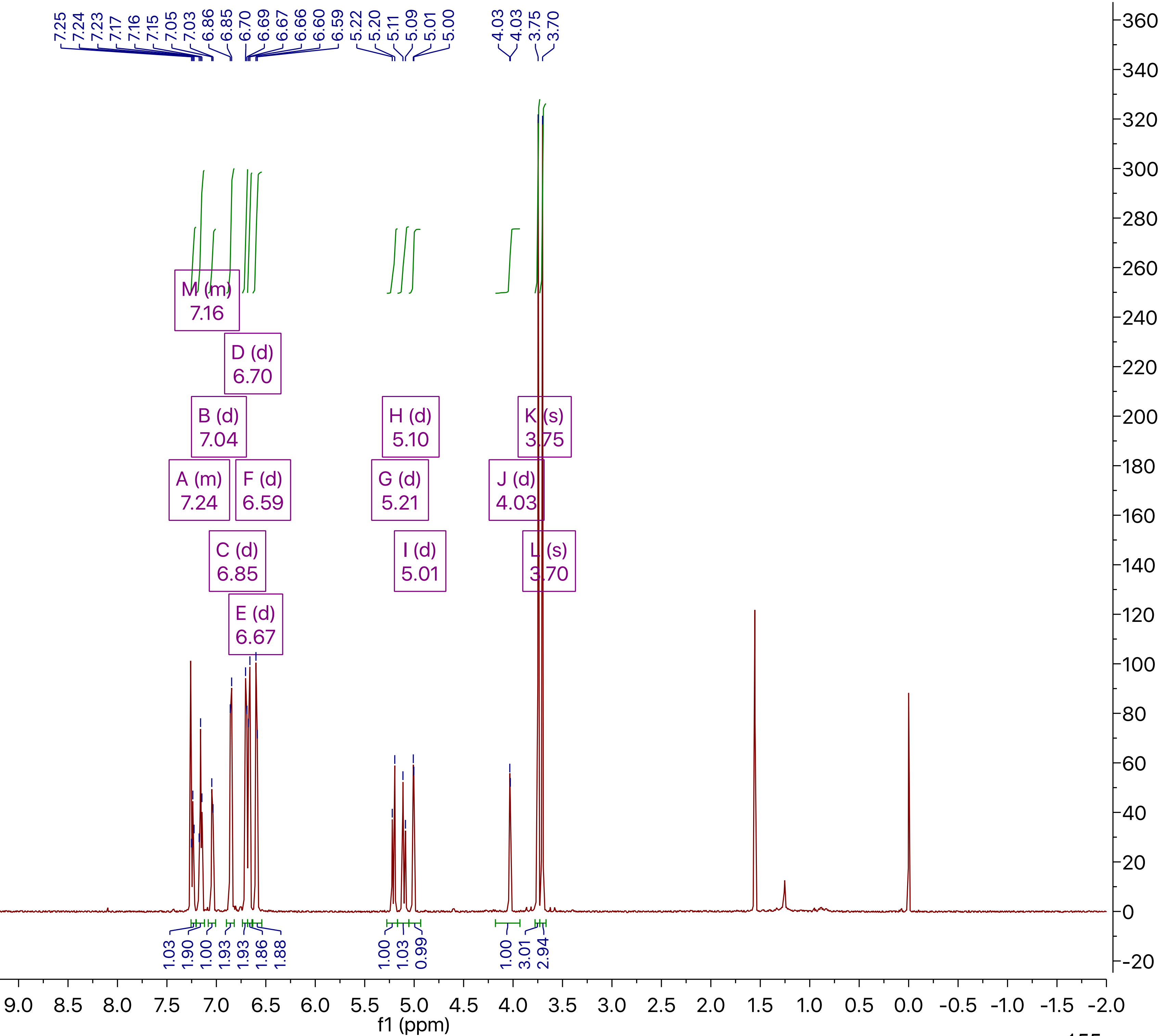


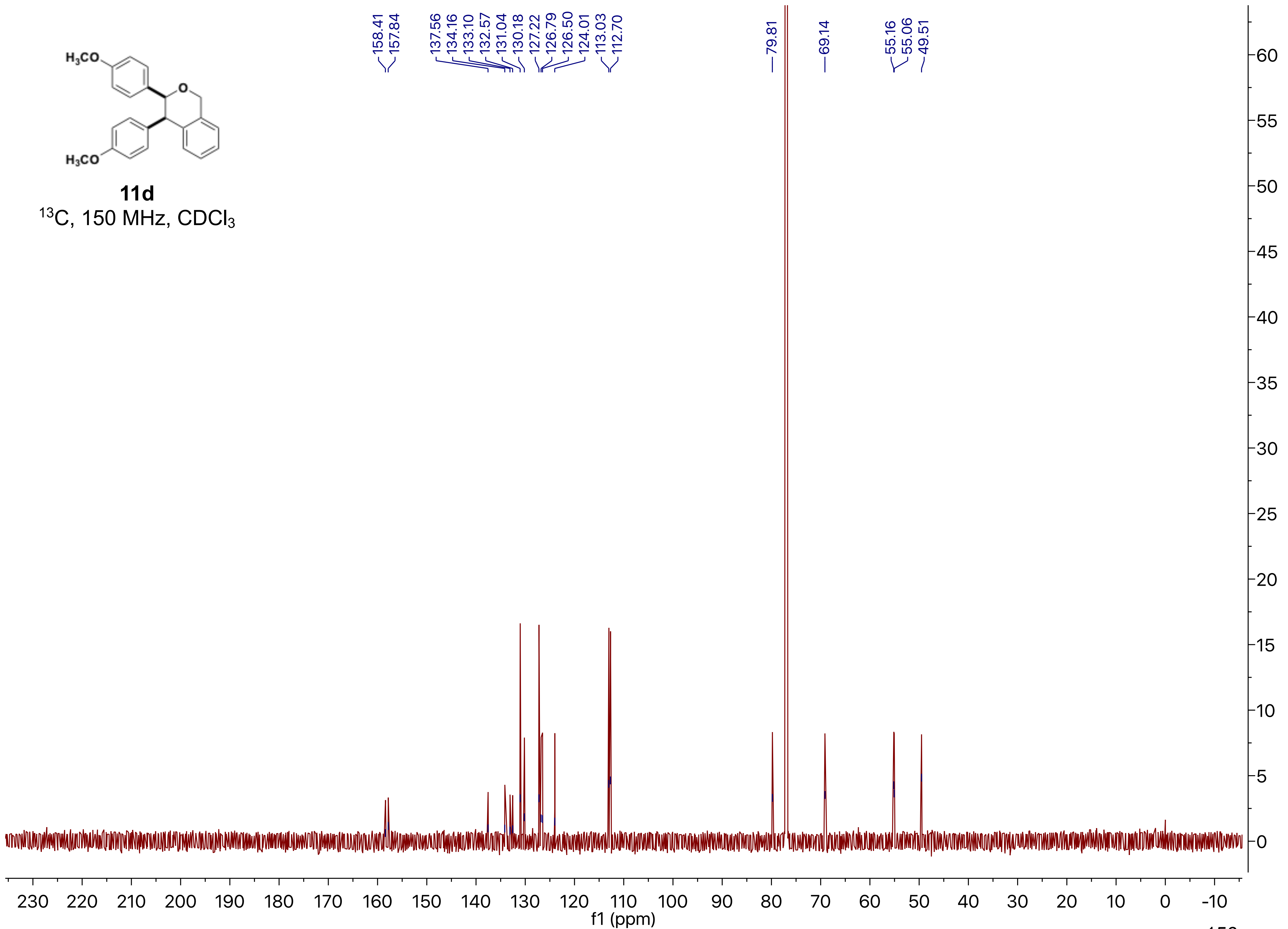
159.75
159.13
148.51
137.24
132.37
130.84
130.16
129.37
129.22
129.20
128.76
128.68
127.35
113.68
113.64
72.59
69.75
55.26
55.23
H2N
N
O
PMB
O
10d
13C, 150 MHz, CDCl3
f1 (ppm)



11d

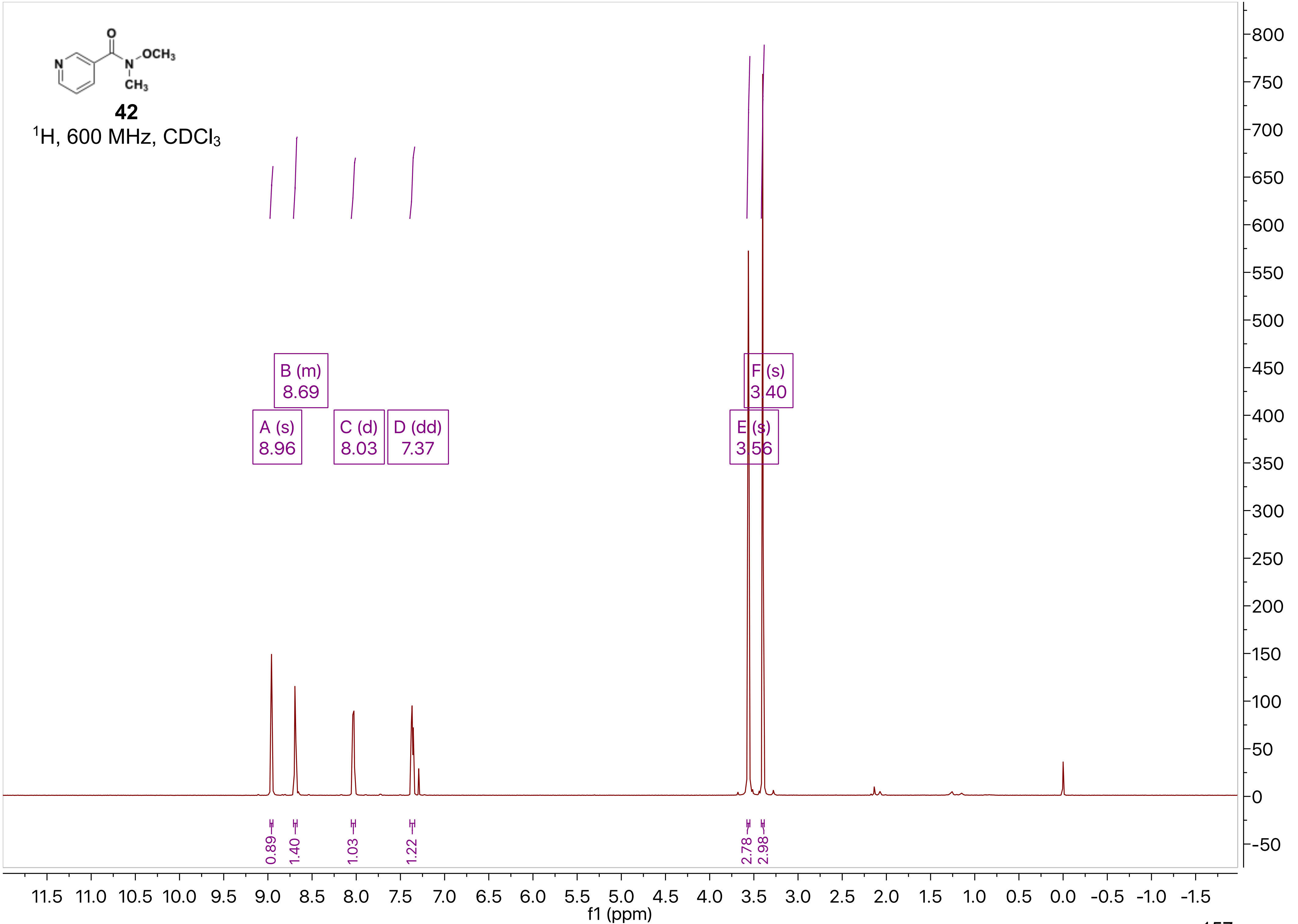
^{1}H, 600 MHz, $CDCl_3$

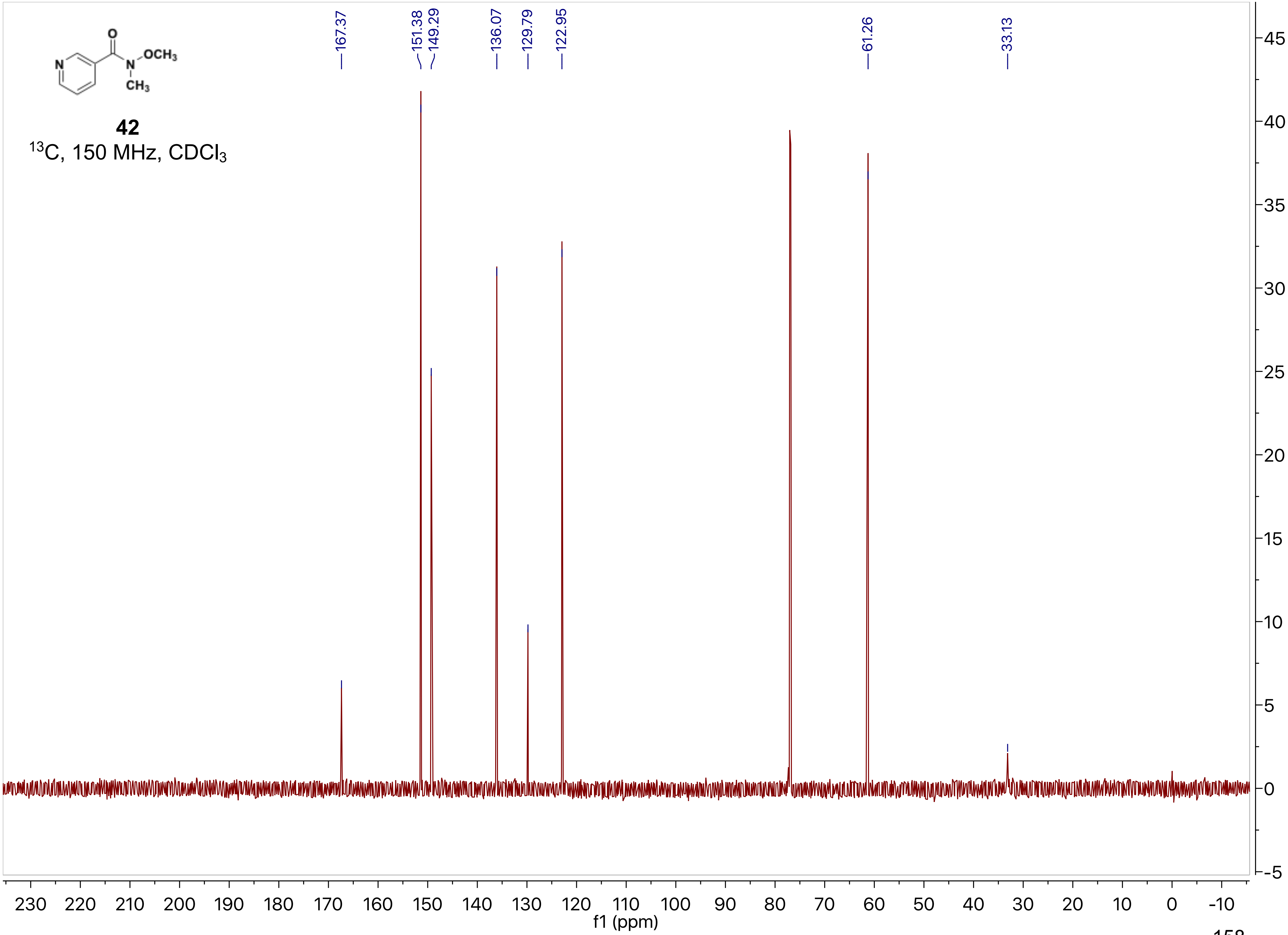


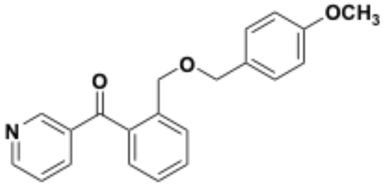


11d

^{13}C, 150 MHz, $CDCl_3$

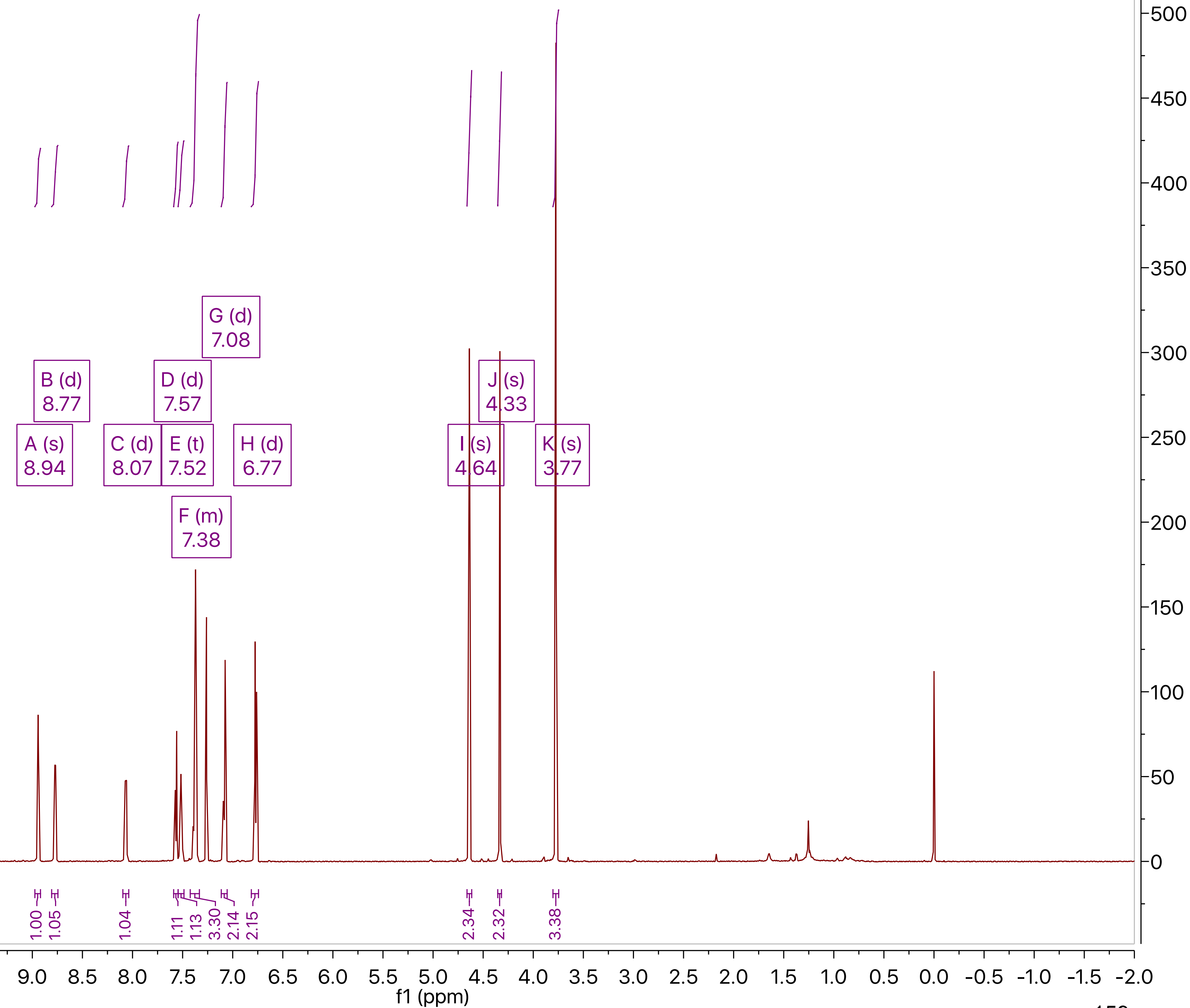

42
1H, 600 MHz, CDCl3
A (s)
8.96
B (m)
8.69
C (d)
8.03
D (dd)
7.37
E (s)
3.56
F (s)
3.40
0.89
1.40
1.03
1.22
2.78
2.98
f1 (ppm)

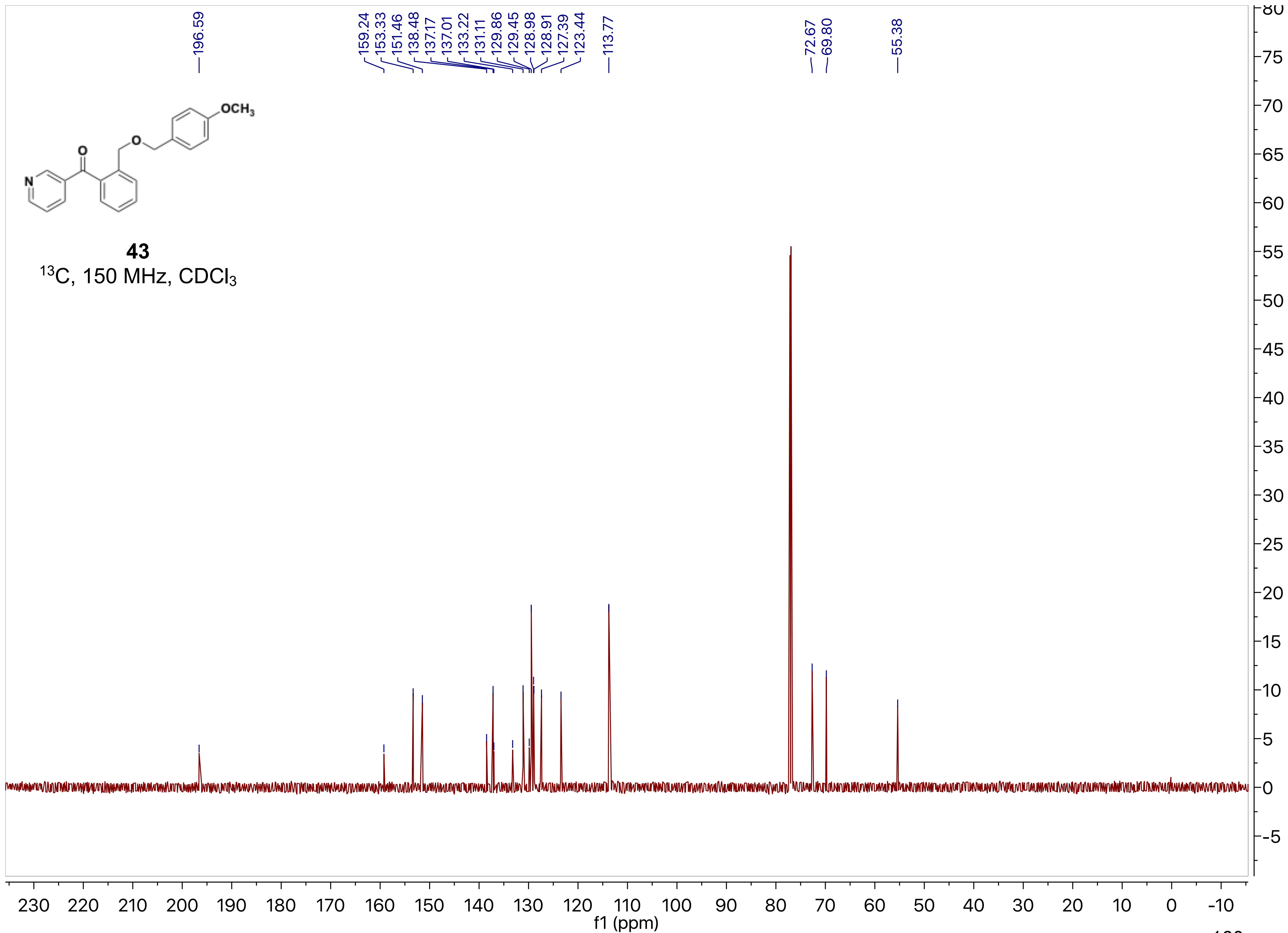

42
^{13}C, 150 MHz, CDCl$_3$
167.37
151.38
149.29
136.07
129.79
122.95
61.26
33.13
f1 (ppm)

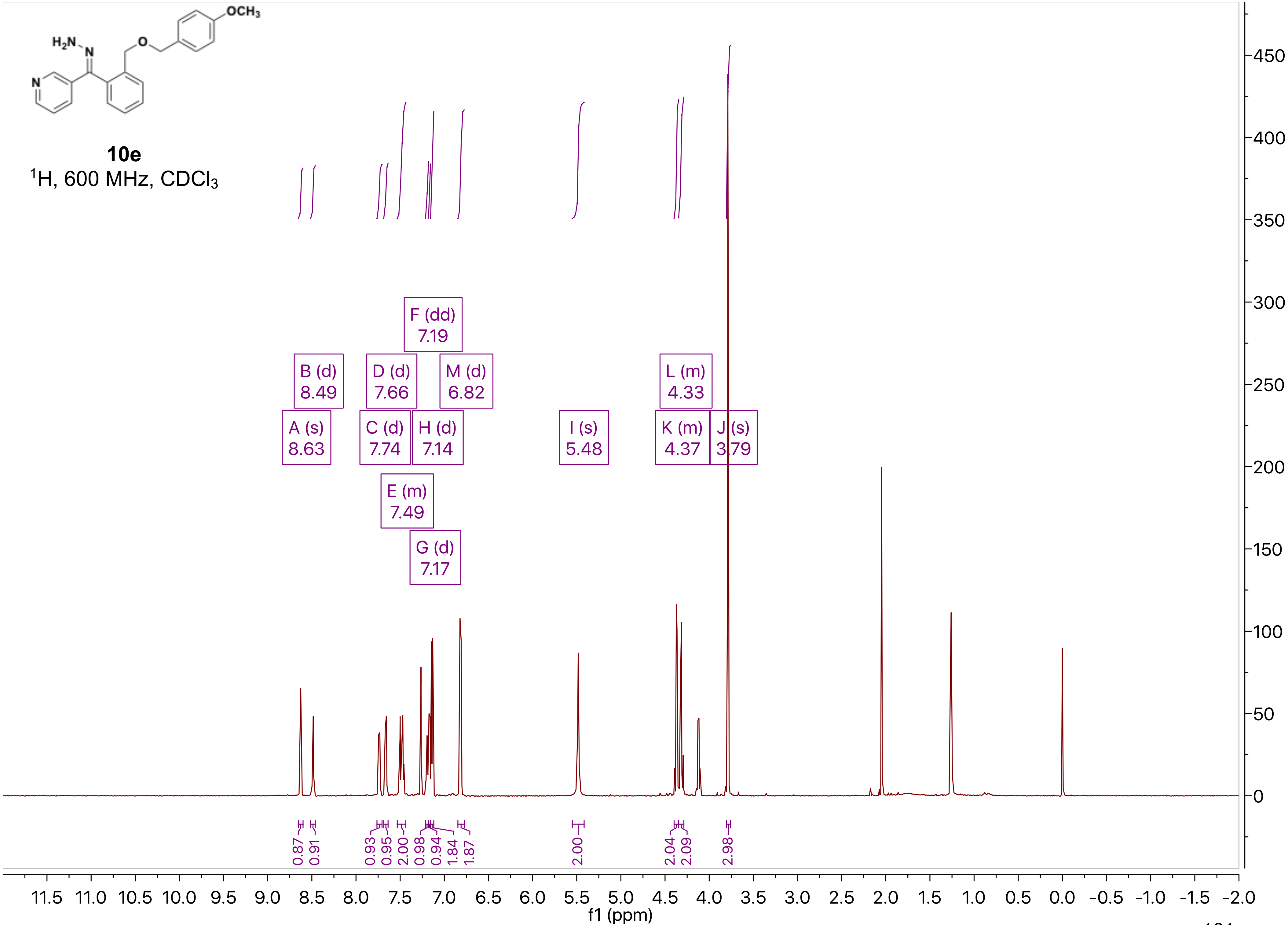


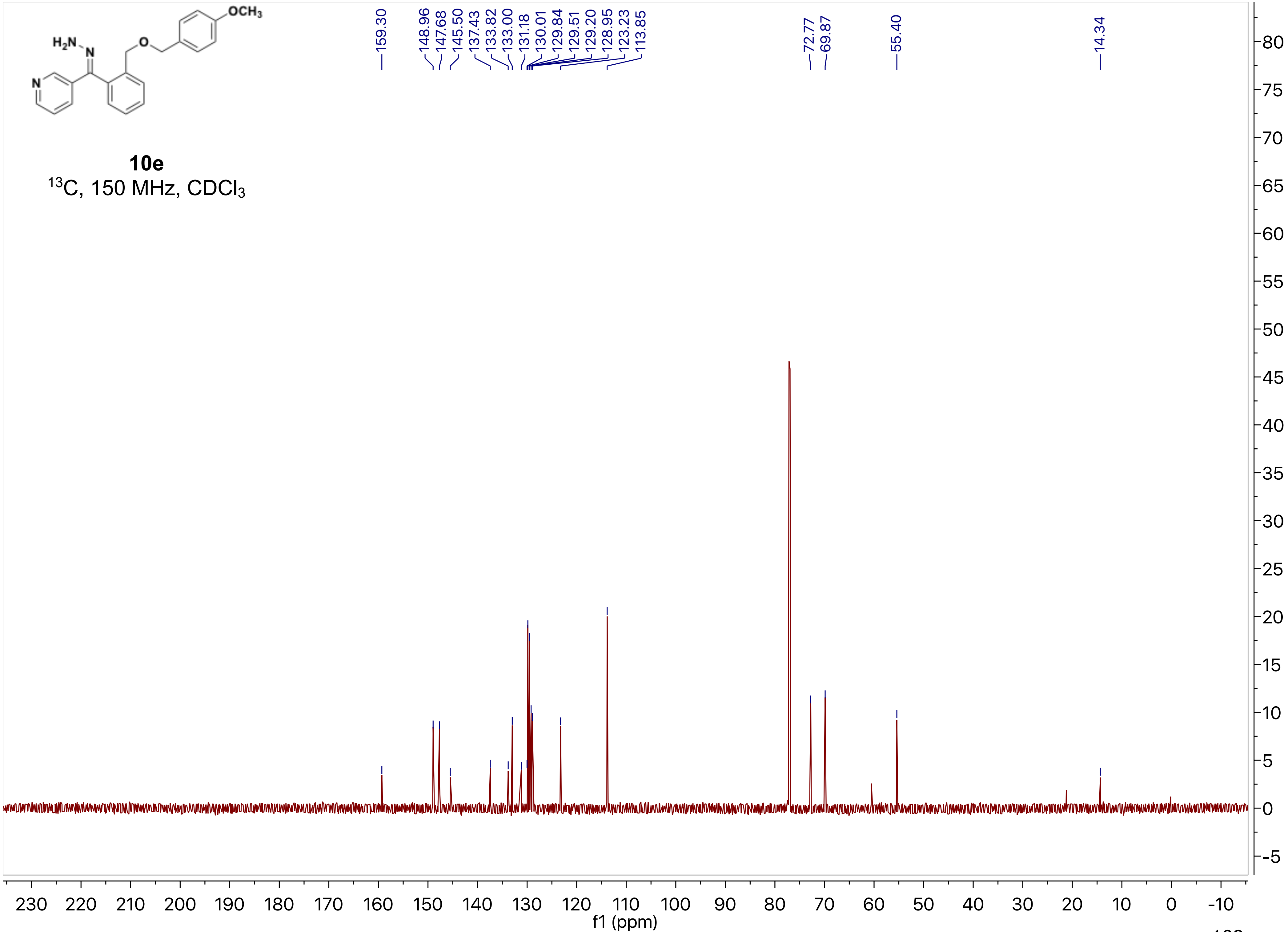
43

^1H, 600 MHz, $CDCl_3$



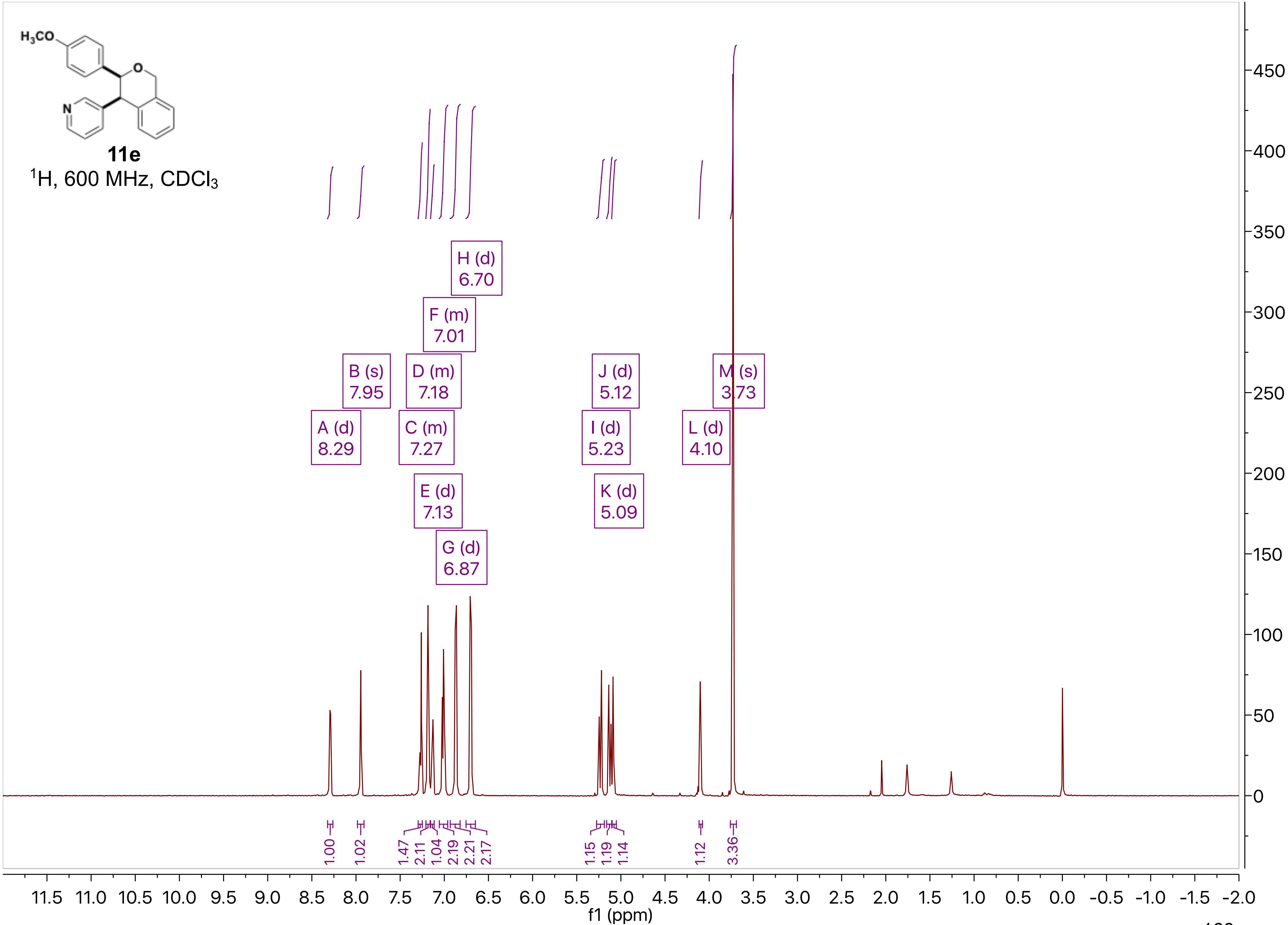

196.59
159.24
153.33
151.46
138.48
137.17
137.01
133.22
131.11
129.86
129.45
128.98
128.91
127.39
123.44
113.77
72.67
69.80
55.38
OCH3
O
N
43
13C, 150 MHz, CDCl3
f1 (ppm)

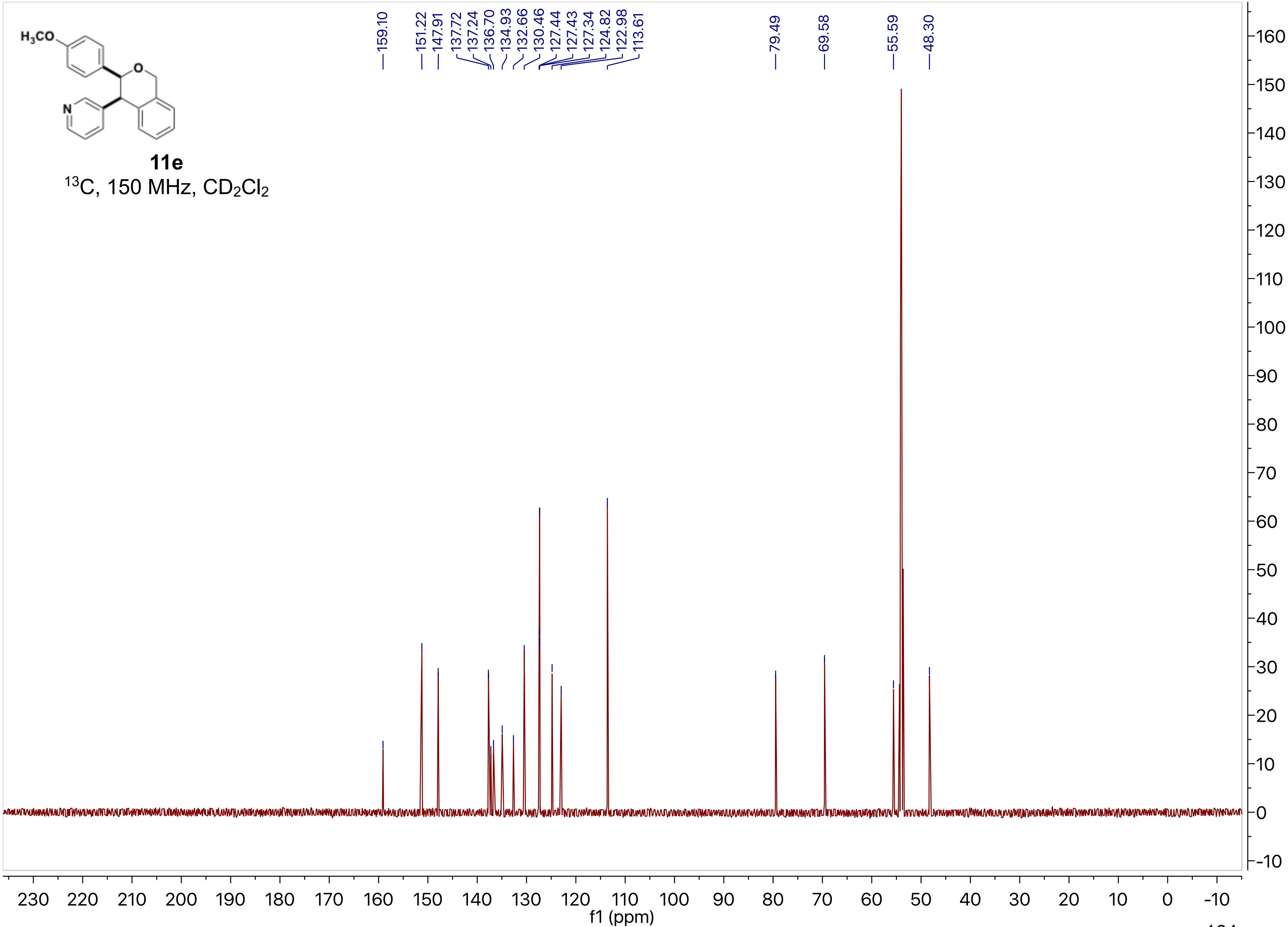

OCH3
H2N
N
O
N
10e
1H, 600 MHz, CDCl3
F (dd)
7.19
B (d)
8.49
D (d)
7.66
M (d)
6.82
L (m)
4.33
A (s)
8.63
C (d)
7.74
H (d)
7.14
I (s)
5.48
K (m)
4.37
J (s)
3.79
E (m)
7.49
G (d)
7.17
0.87
0.91
0.93
0.95
2.00
0.98
0.94
1.84
1.87
2.00
2.04
2.09
2.98
f1 (ppm)

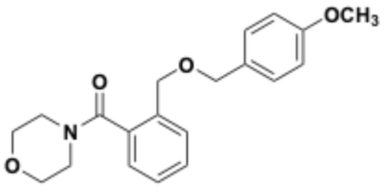


10e

^{13}C, 150 MHz, $CDCl_3$

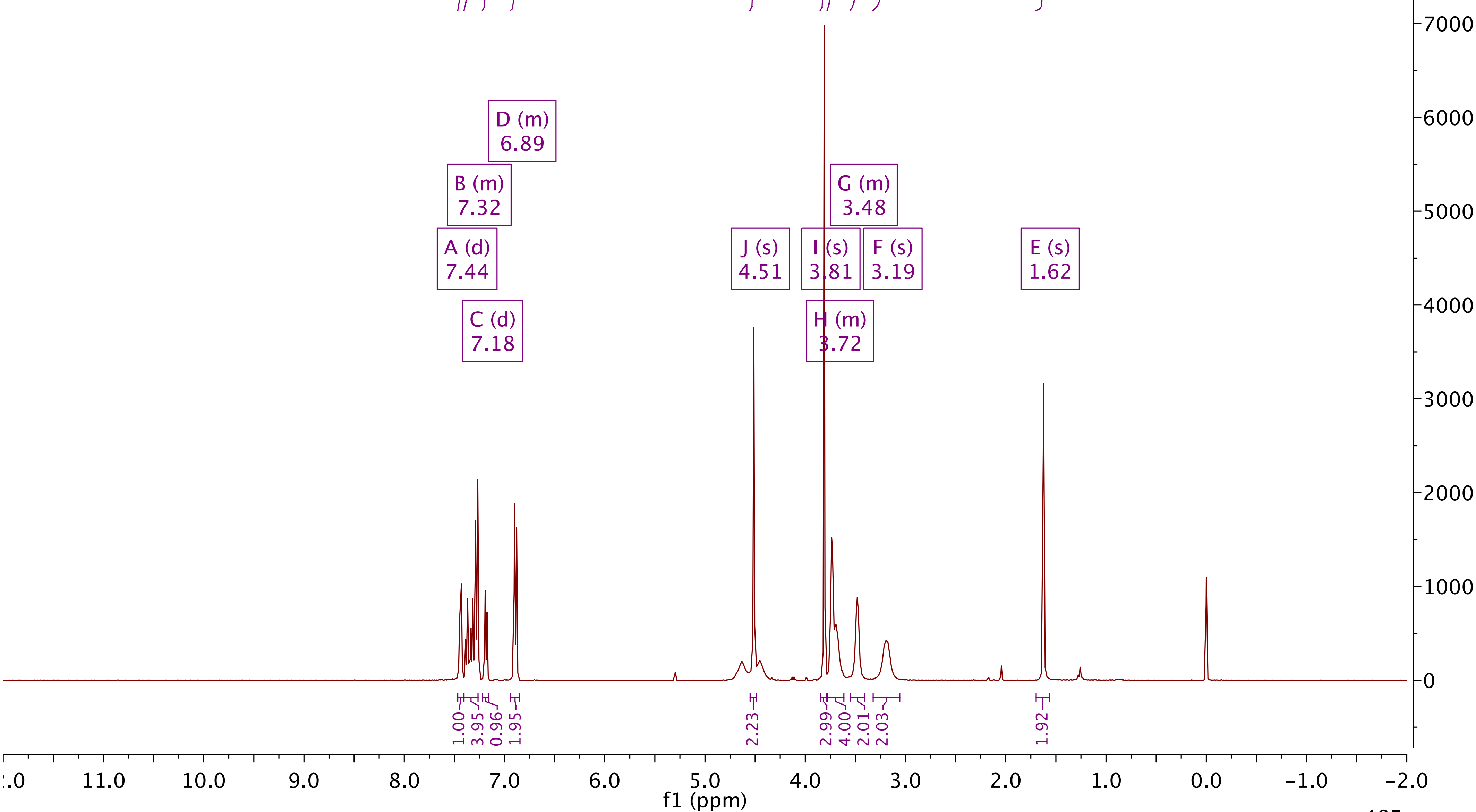

H3CO
O
N
11e
1H, 600 MHz, CDCl3
H (d) 6.70
F (m) 7.01
B (s) 7.95
D (m) 7.18
J (d) 5.12
M (s) 3.73
A (d) 8.29
C (m) 7.27
I (d) 5.23
L (d) 4.10
E (d) 7.13
K (d) 5.09
G (d) 6.87
1.00
1.02
1.47
2.11
1.04
2.19
2.21
2.17
1.15
1.19
1.14
1.12
3.36
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5 -2.0
f1 (ppm)
450
400
350
300
250
200
150
100
50
0

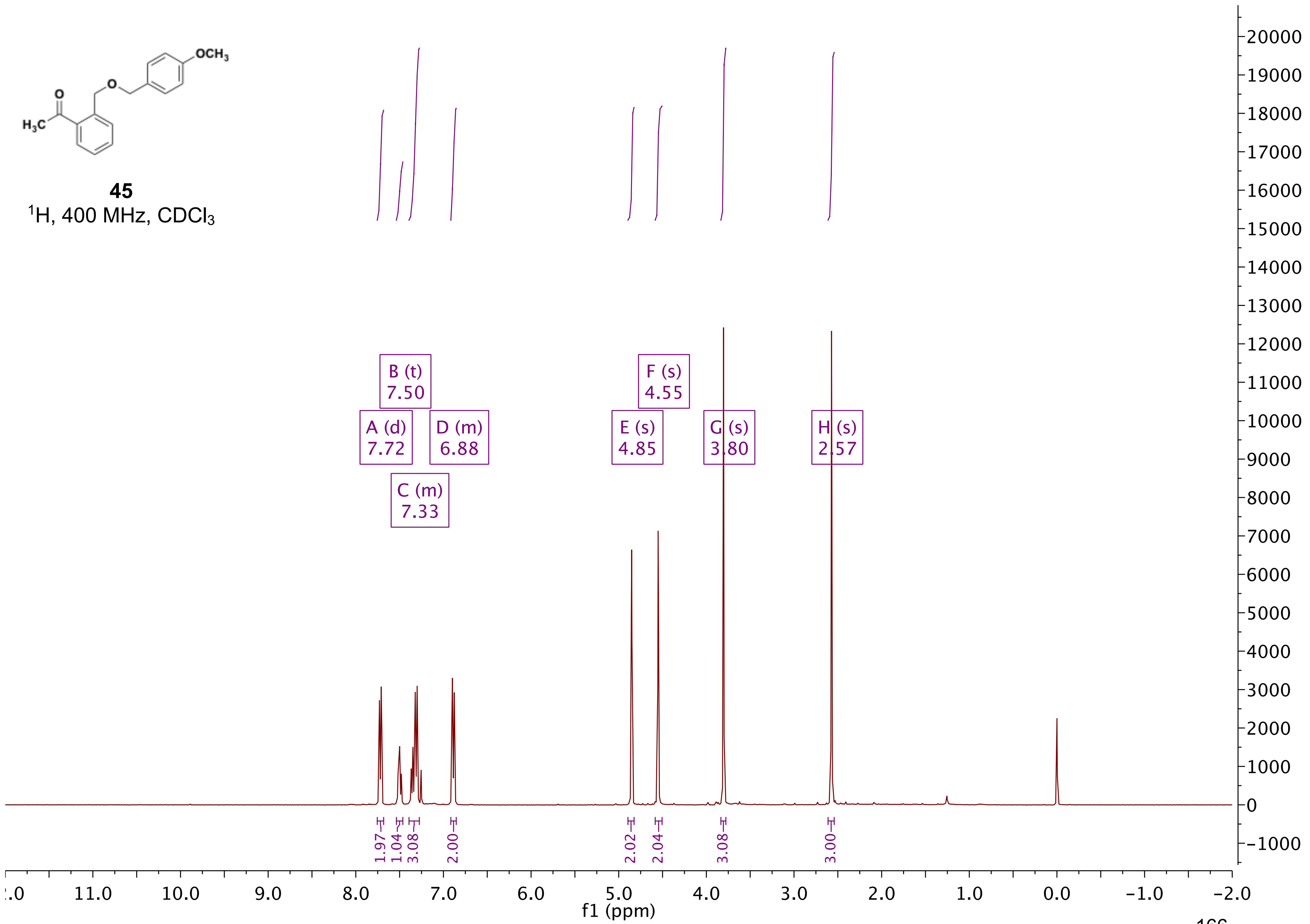

H3CO
N
O
11e
13C, 150 MHz, CD2Cl2
159.10
151.22
147.91
137.72
137.24
136.70
134.93
132.66
130.46
127.44
127.43
127.34
124.82
122.98
113.61
79.49
69.58
55.59
48.30
f1 (ppm)



44

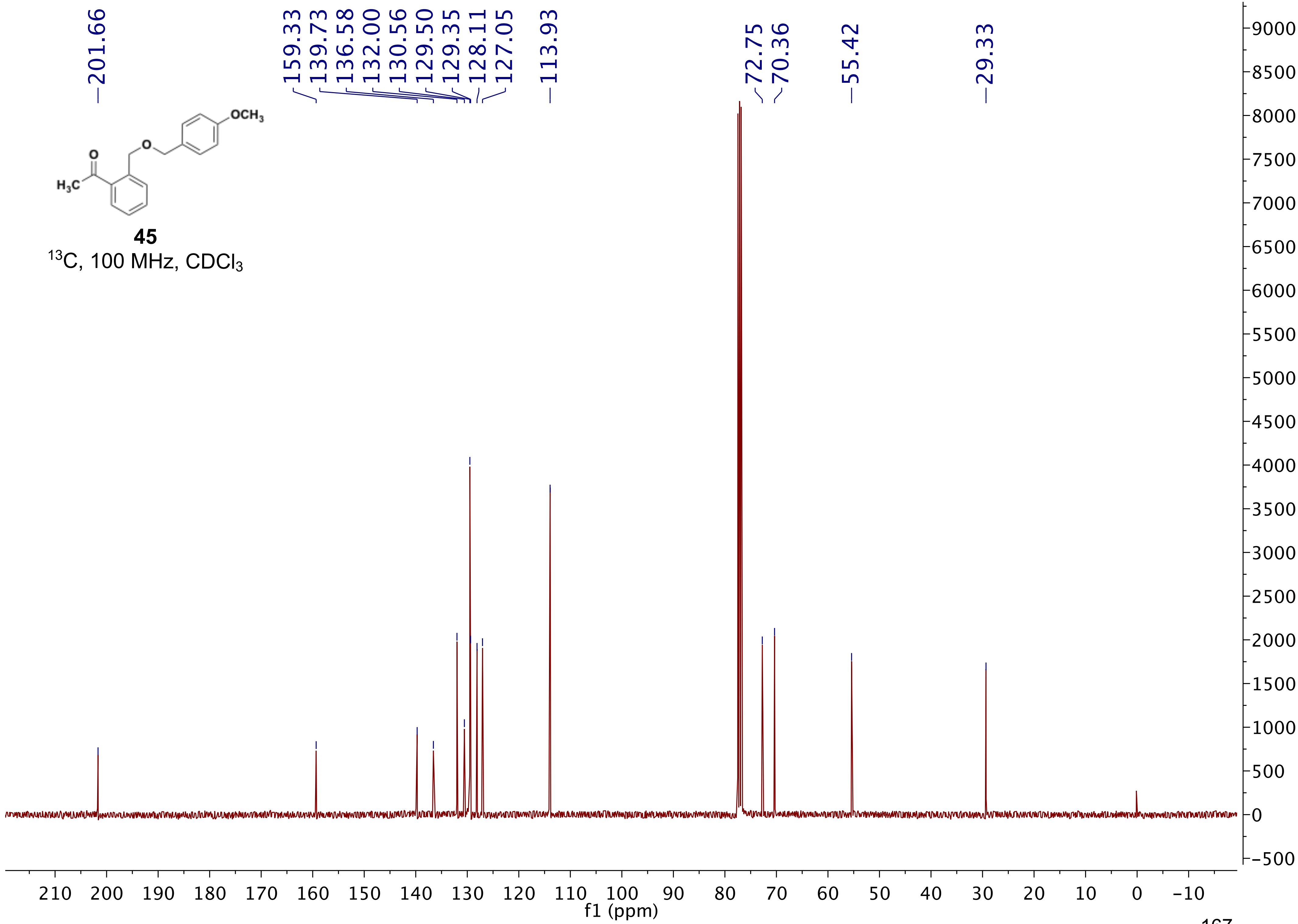
^{1}H, 400 MHz, $CDCl_3$





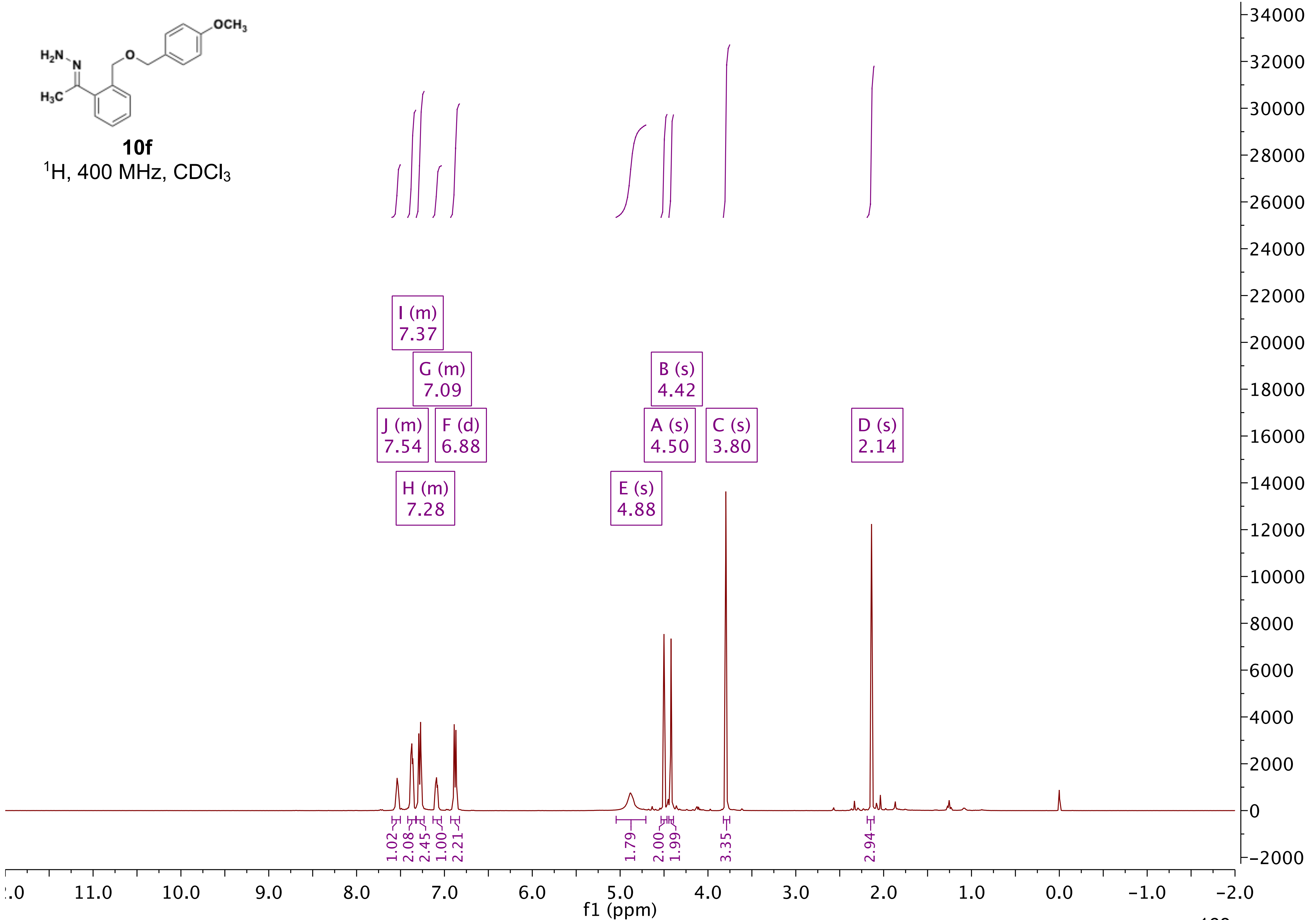
45

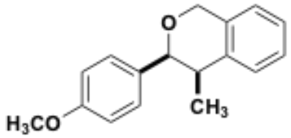
^{1}H, 400 MHz, $CDCl_3$



45

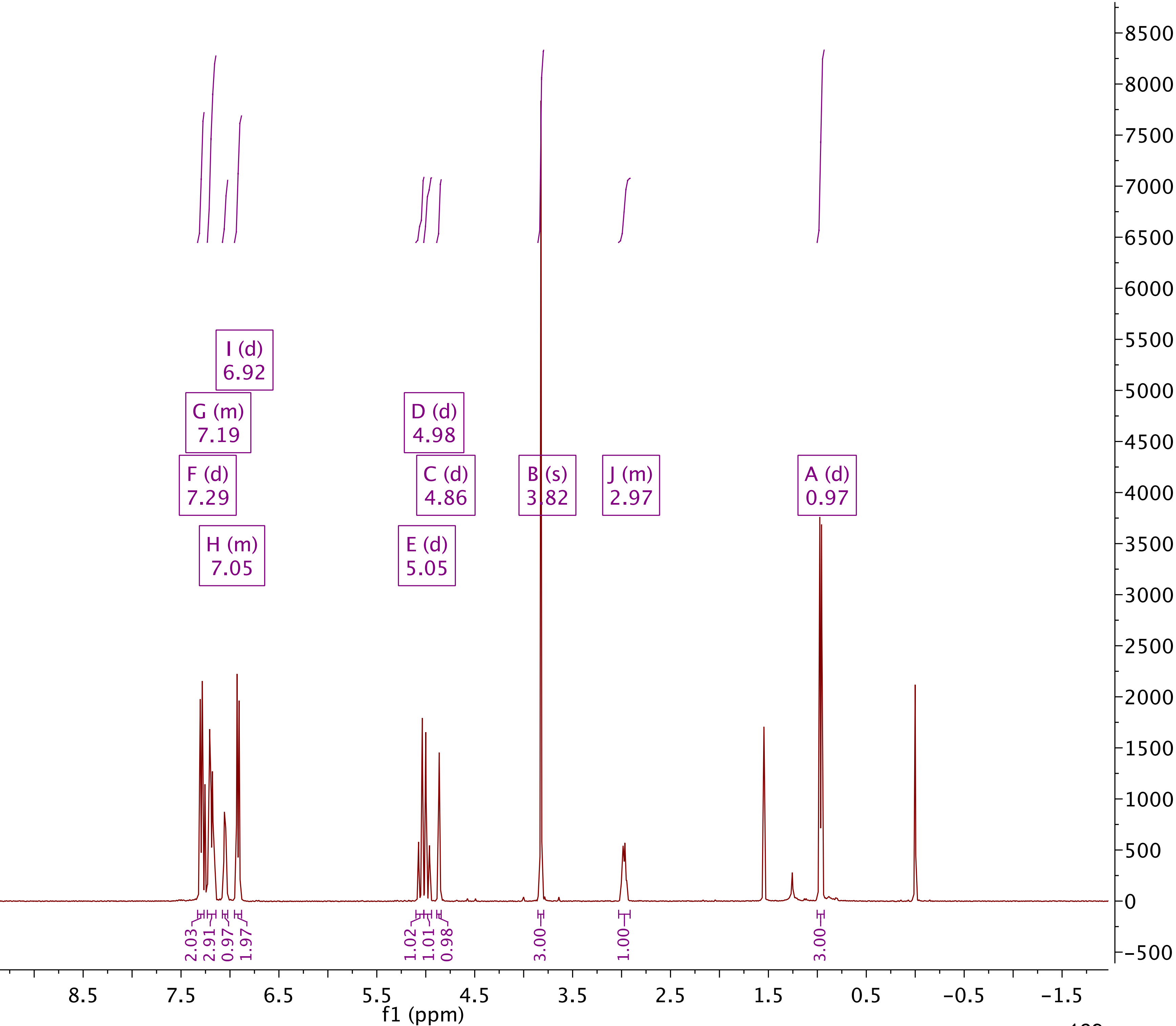
^{13}C, 100 MHz, $CDCl_3$

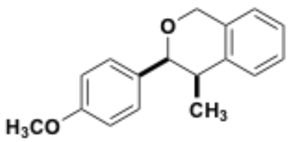

10f
1H, 400 MHz, CDCl3
I (m) 7.37
G (m) 7.09
B (s) 4.42
J (m) 7.54
F (d) 6.88
A (s) 4.50
C (s) 3.80
D (s) 2.14
H (m) 7.28
E (s) 4.88
1.02
2.08
2.45
1.00
2.21
1.79
2.00
1.99
3.35
2.94
f1 (ppm)



11f

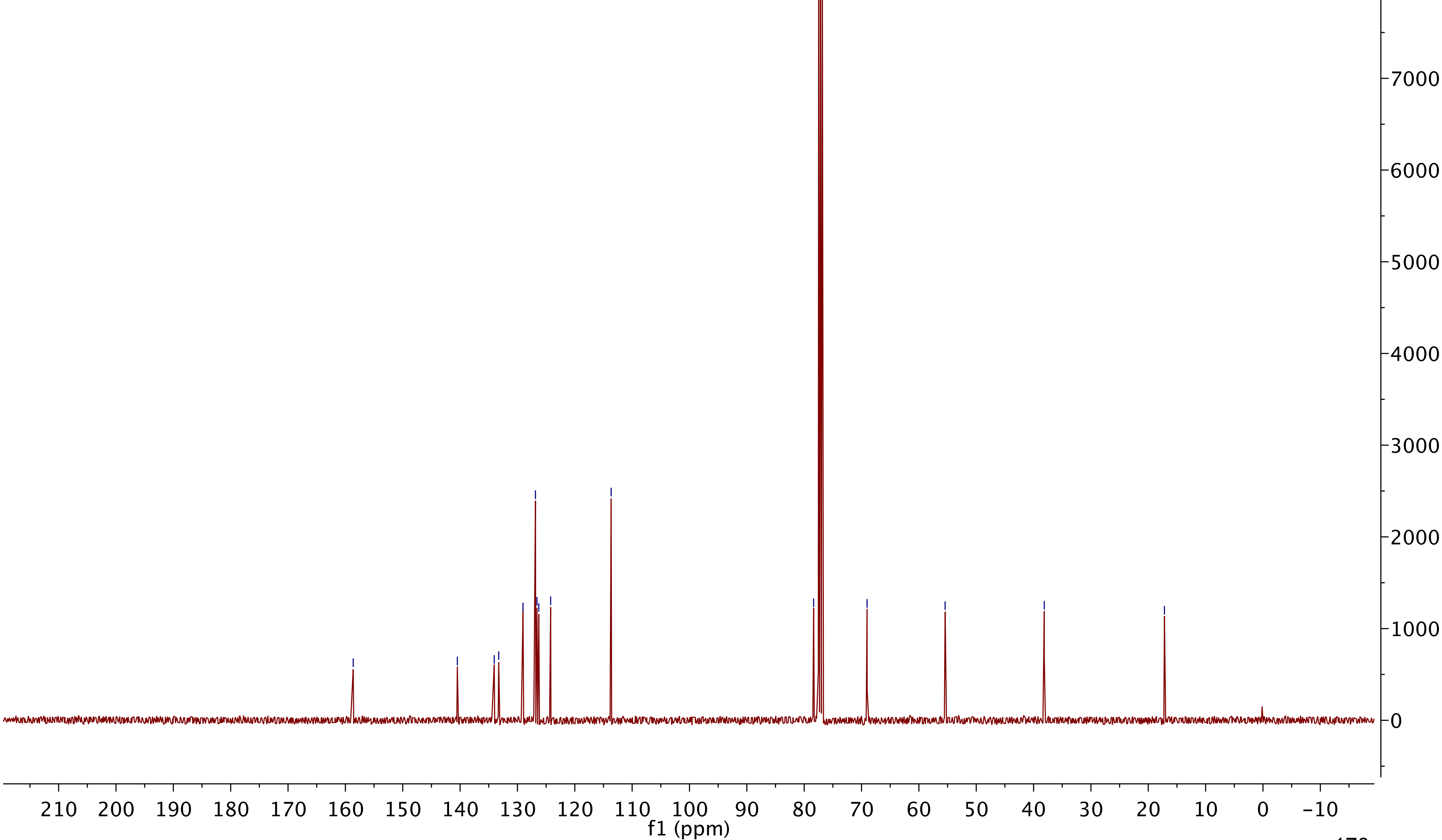
^{1}H, 400 MHz, $CDCl_3$

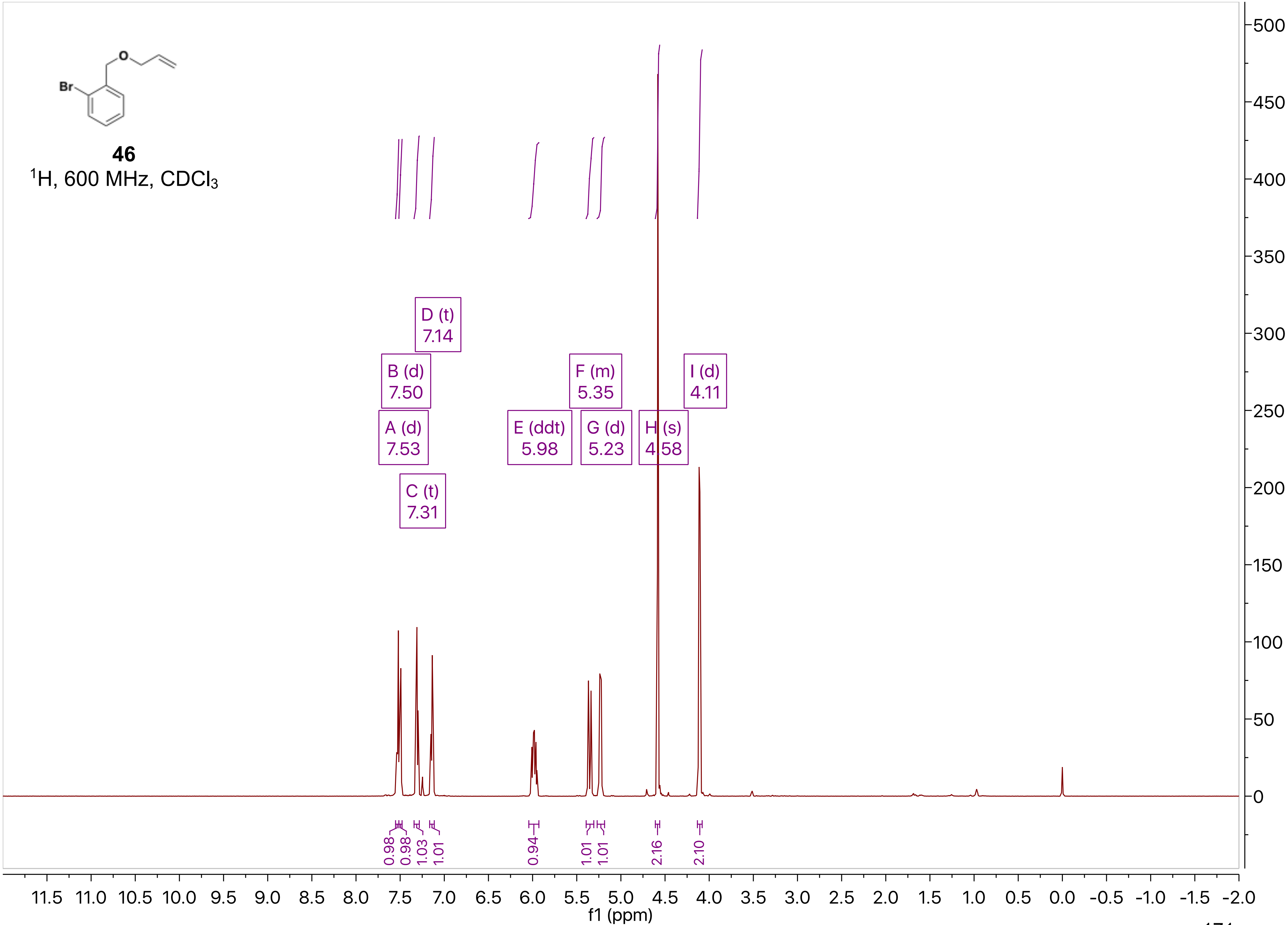




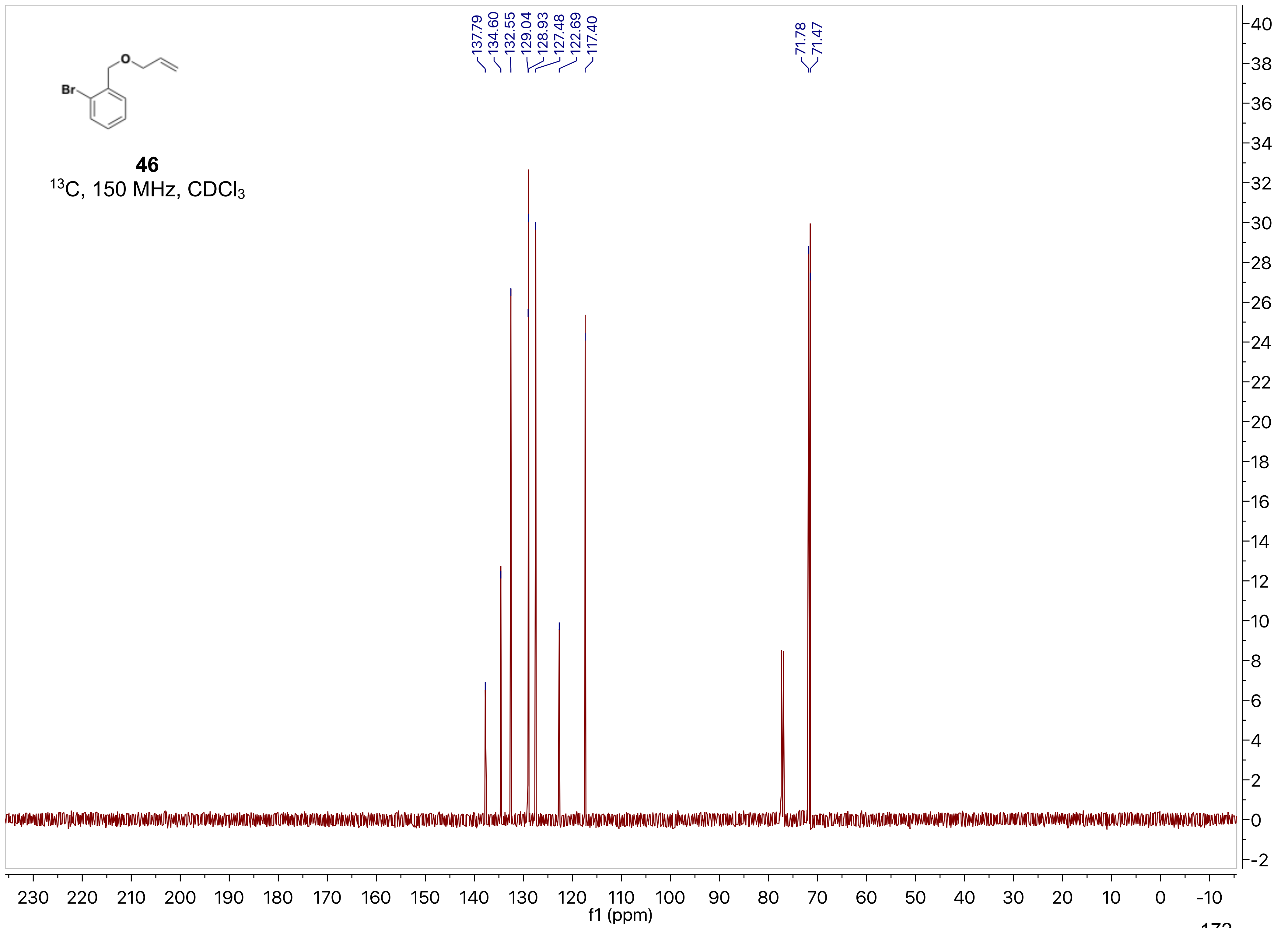
11f

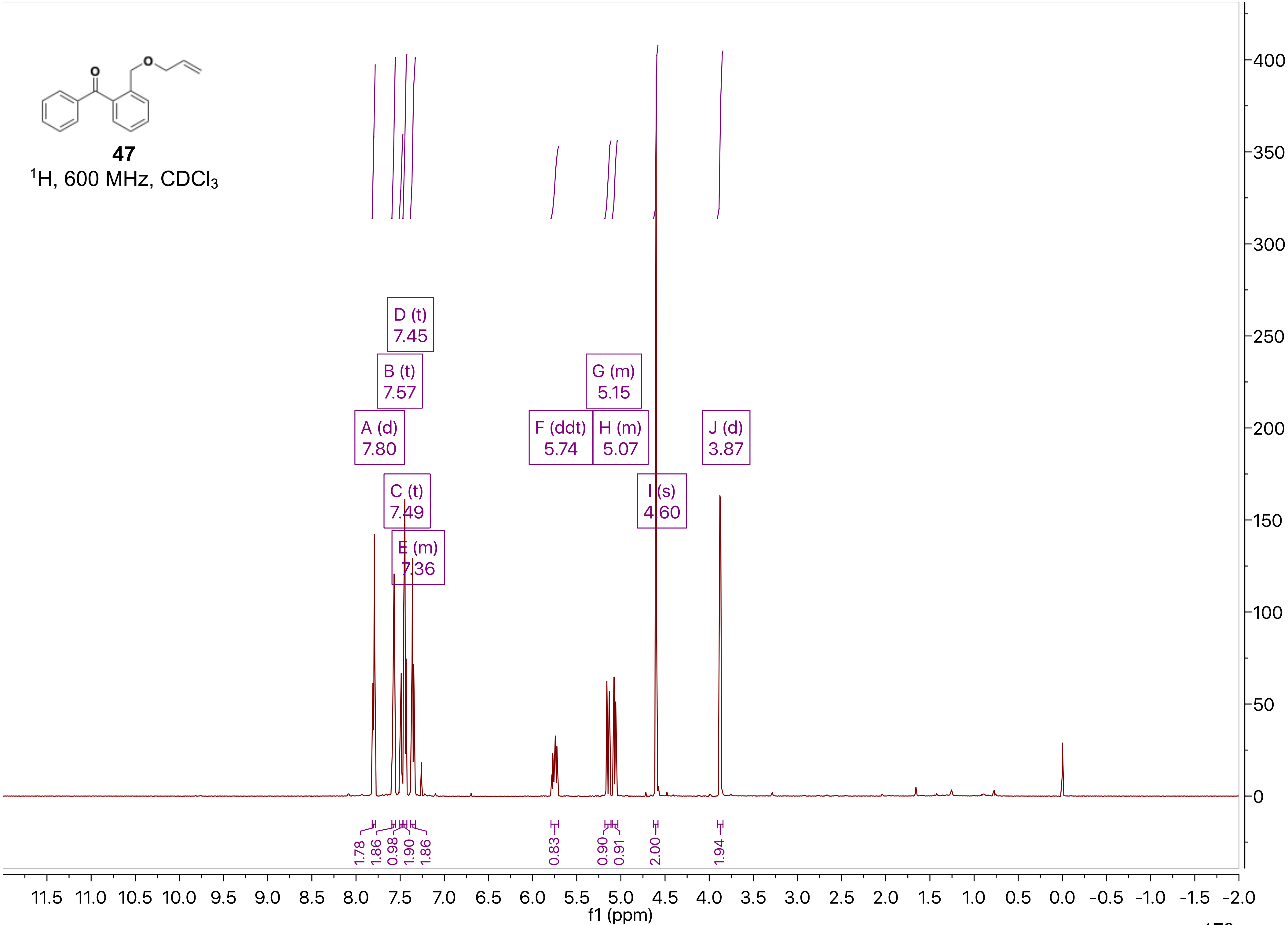
^{13}C, 100 MHz, $CDCl_3$



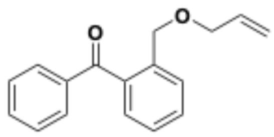


Br
O
46
1H, 600 MHz, CDCl3
D (t)
7.14
B (d)
7.50
A (d)
7.53
C (t)
7.31
E (ddt)
5.98
F (m)
5.35
G (d)
5.23
H (s)
4.58
I (d)
4.11
0.98
0.98
1.03
1.01
0.94
1.01
1.01
2.16
2.10
f1 (ppm)


137.79
134.60
132.55
129.04
128.93
127.48
122.69
117.40
71.78
71.47
Br
O
46
13C, 150 MHz, CDCl3
f1 (ppm)

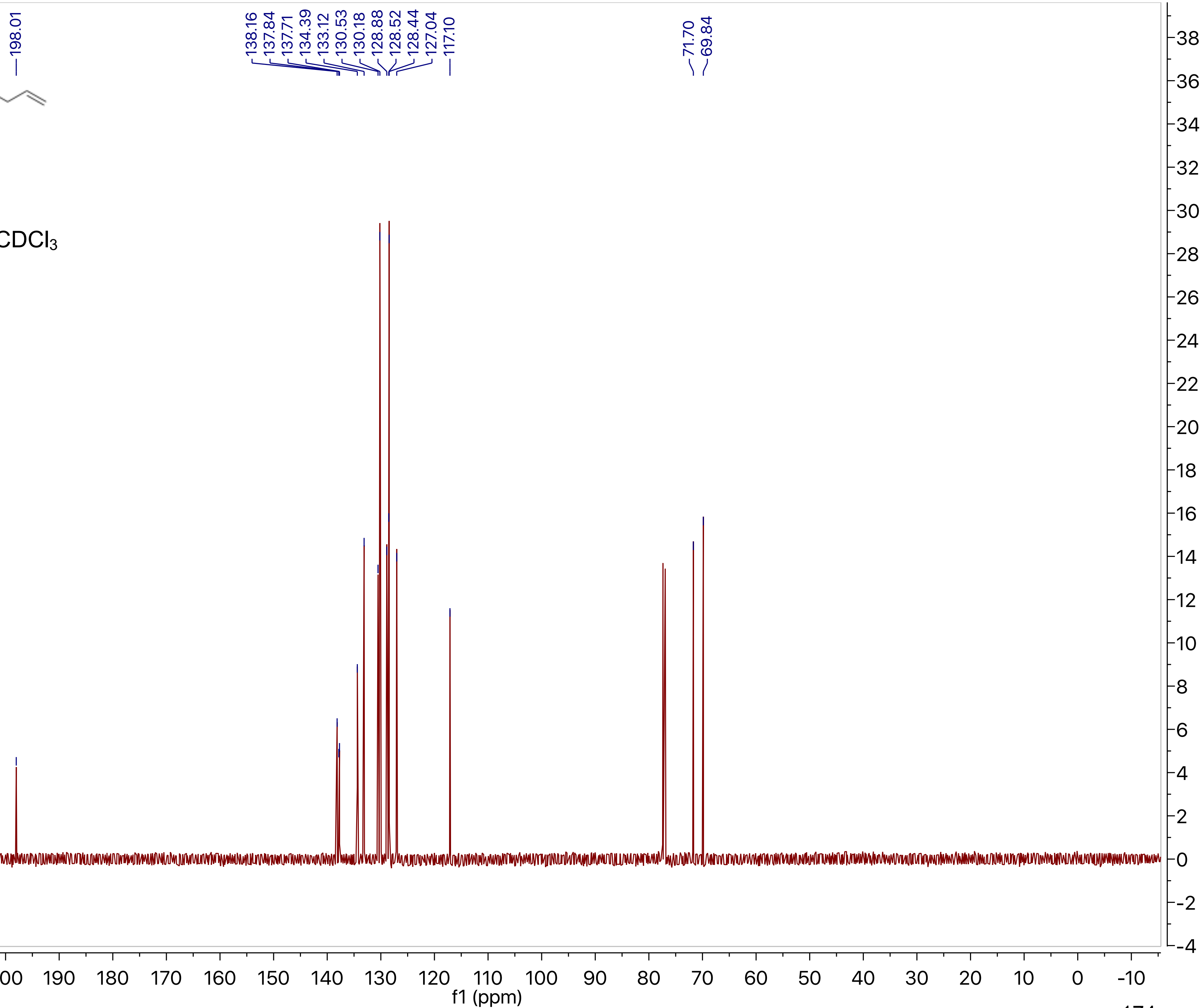


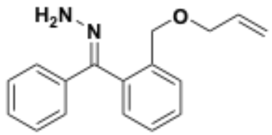
47
1H, 600 MHz, CDCl3
D (t)
7.45
B (t)
7.57
A (d)
7.80
C (t)
7.49
E (m)
7.36
F (ddt)
5.74
G (m)
5.15
H (m)
5.07
I (s)
4.60
J (d)
3.87
1.78
1.86
0.98
1.90
1.86
0.83
0.90
0.91
2.00
1.94
f1 (ppm)



47

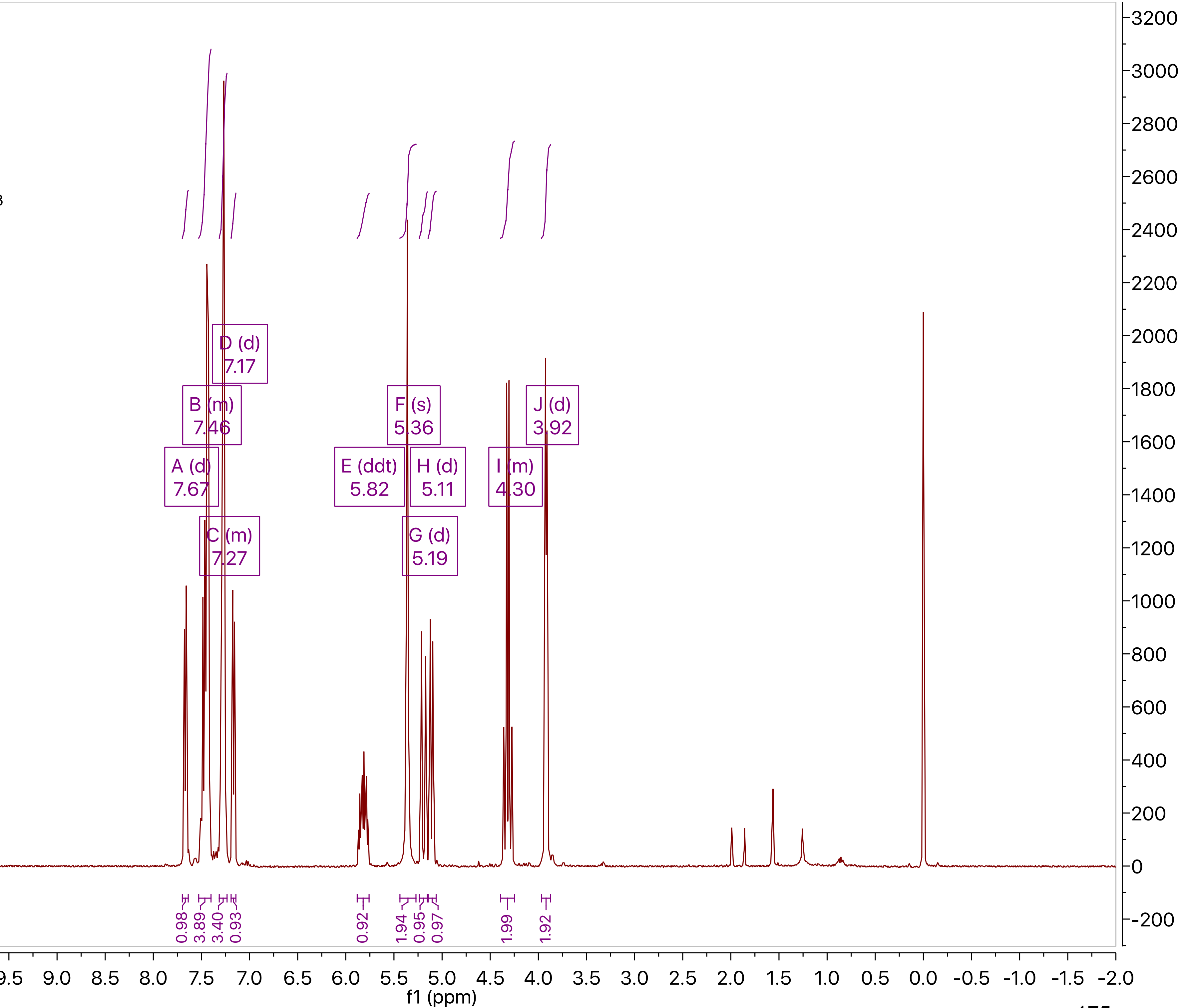
^{13}C, 150 MHz, $CDCl_3$

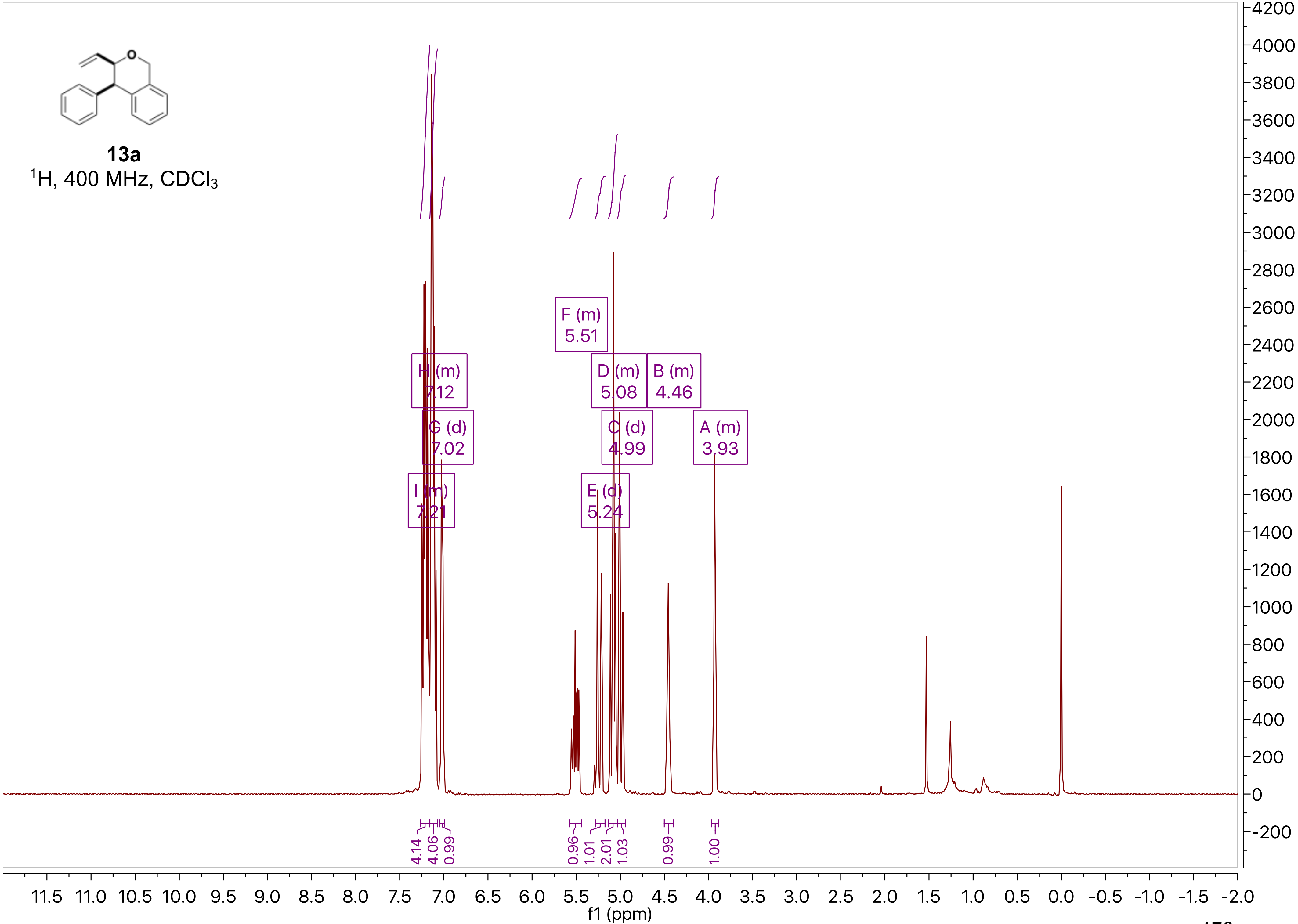


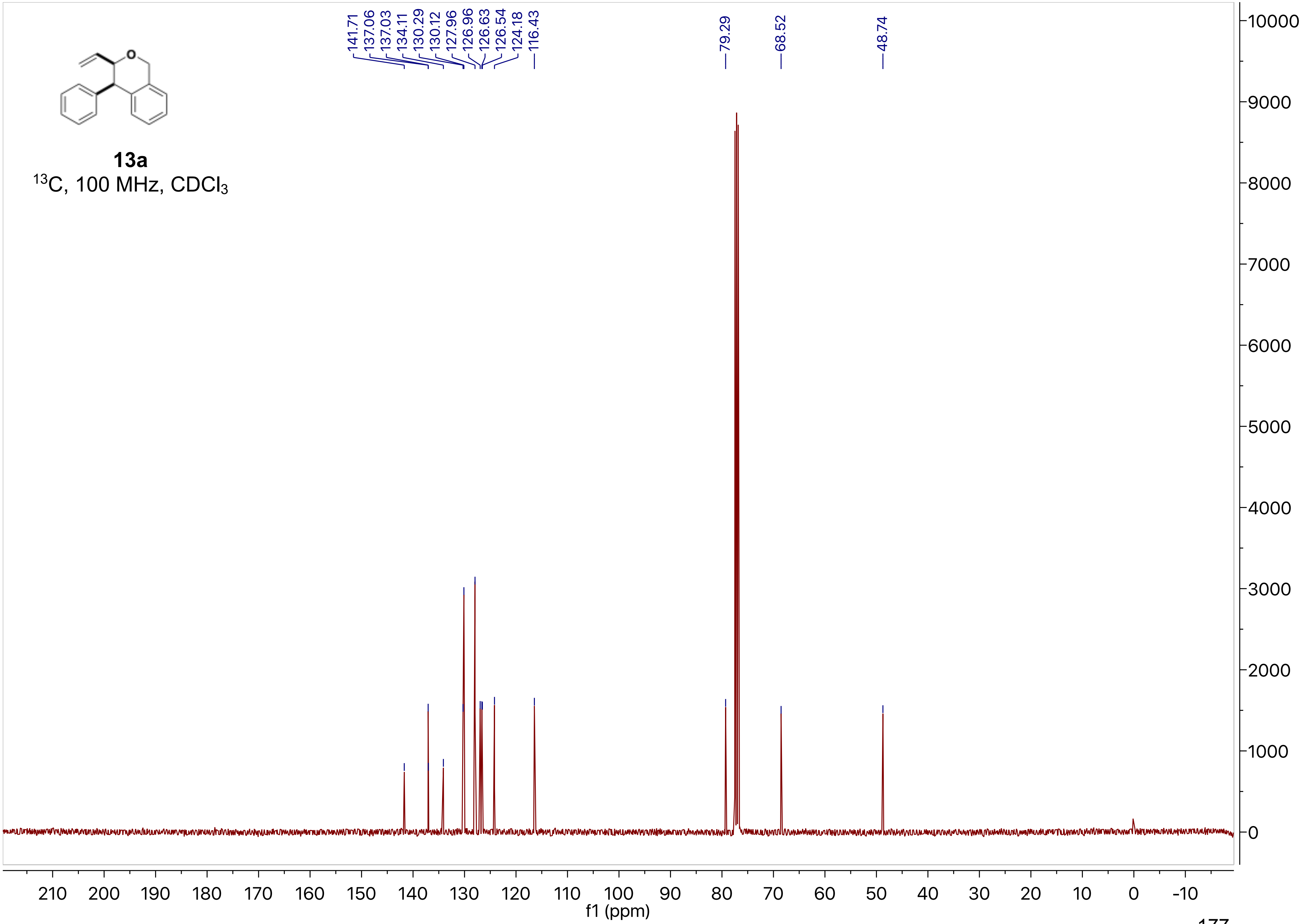


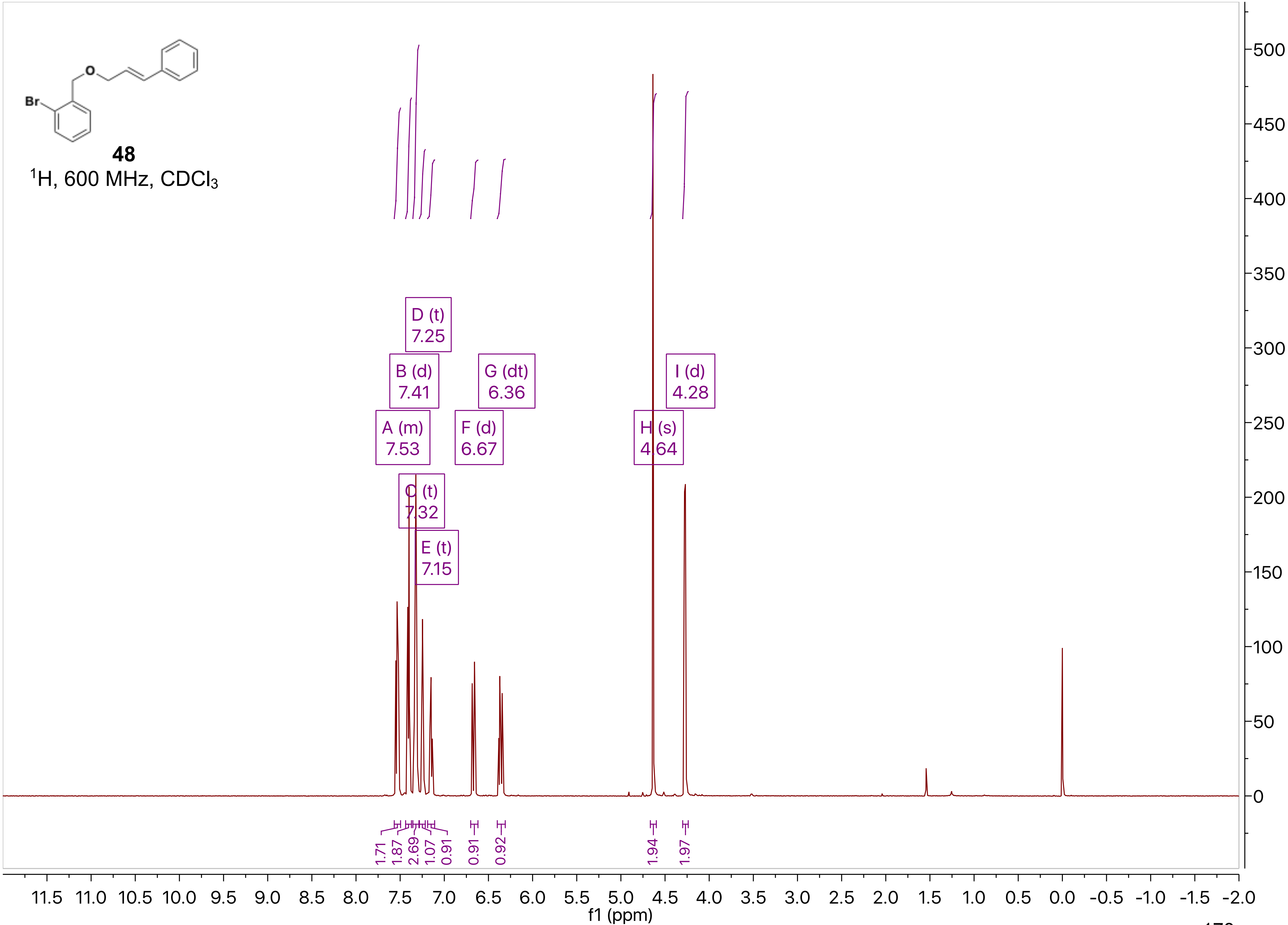
12a

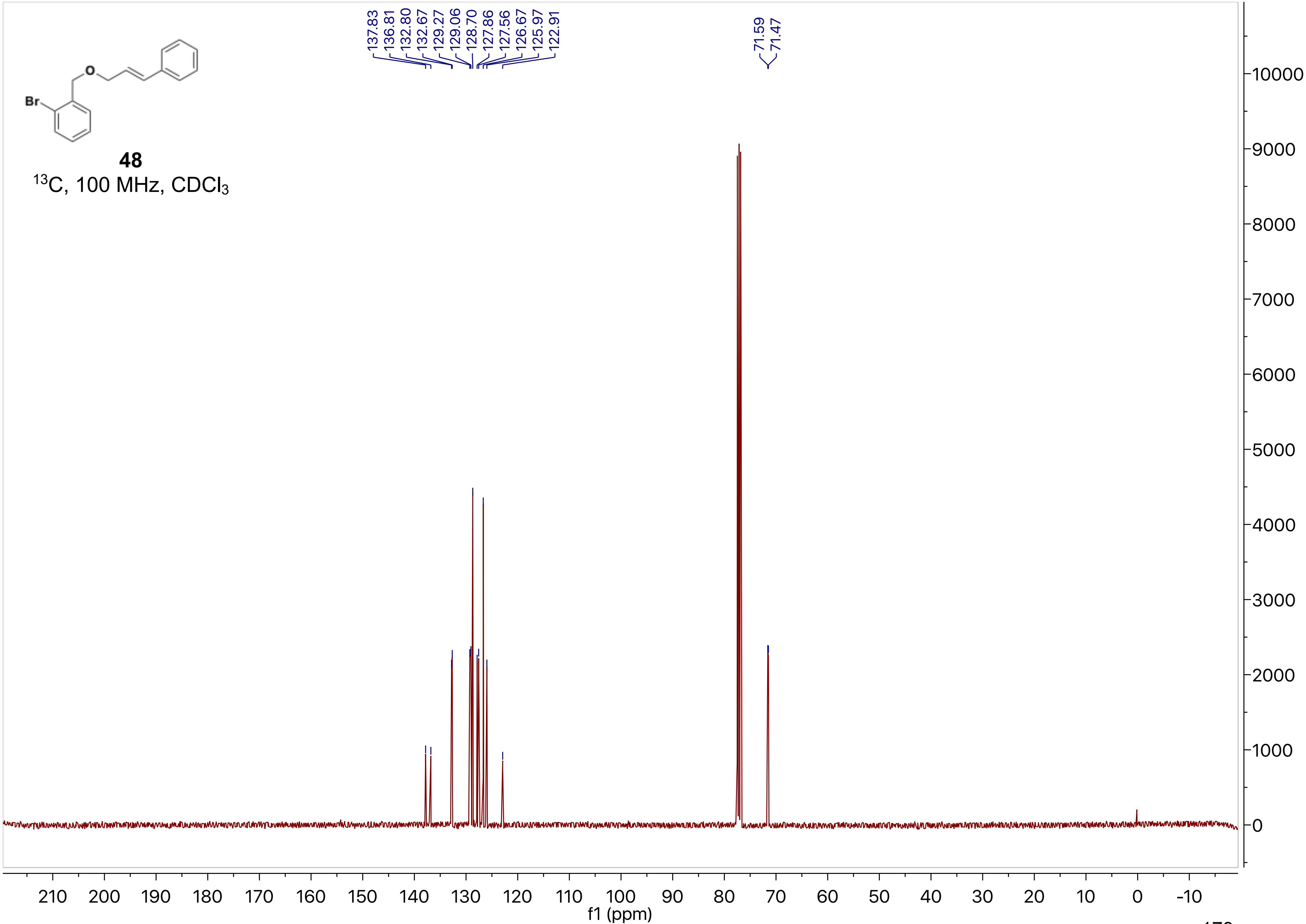
^{1}H, 400 MHz, $CDCl_3$




13a
1H, 400 MHz, CDCl3
F (m) 5.51
H (m) 7.12
D (m) 5.08
B (m) 4.46
G (d) 7.02
C (d) 4.99
A (m) 3.93
I (m) 7.21
E (d) 5.24
4.14
4.06
0.99
0.96
1.01
2.01
1.03
0.99
1.00
f1 (ppm)

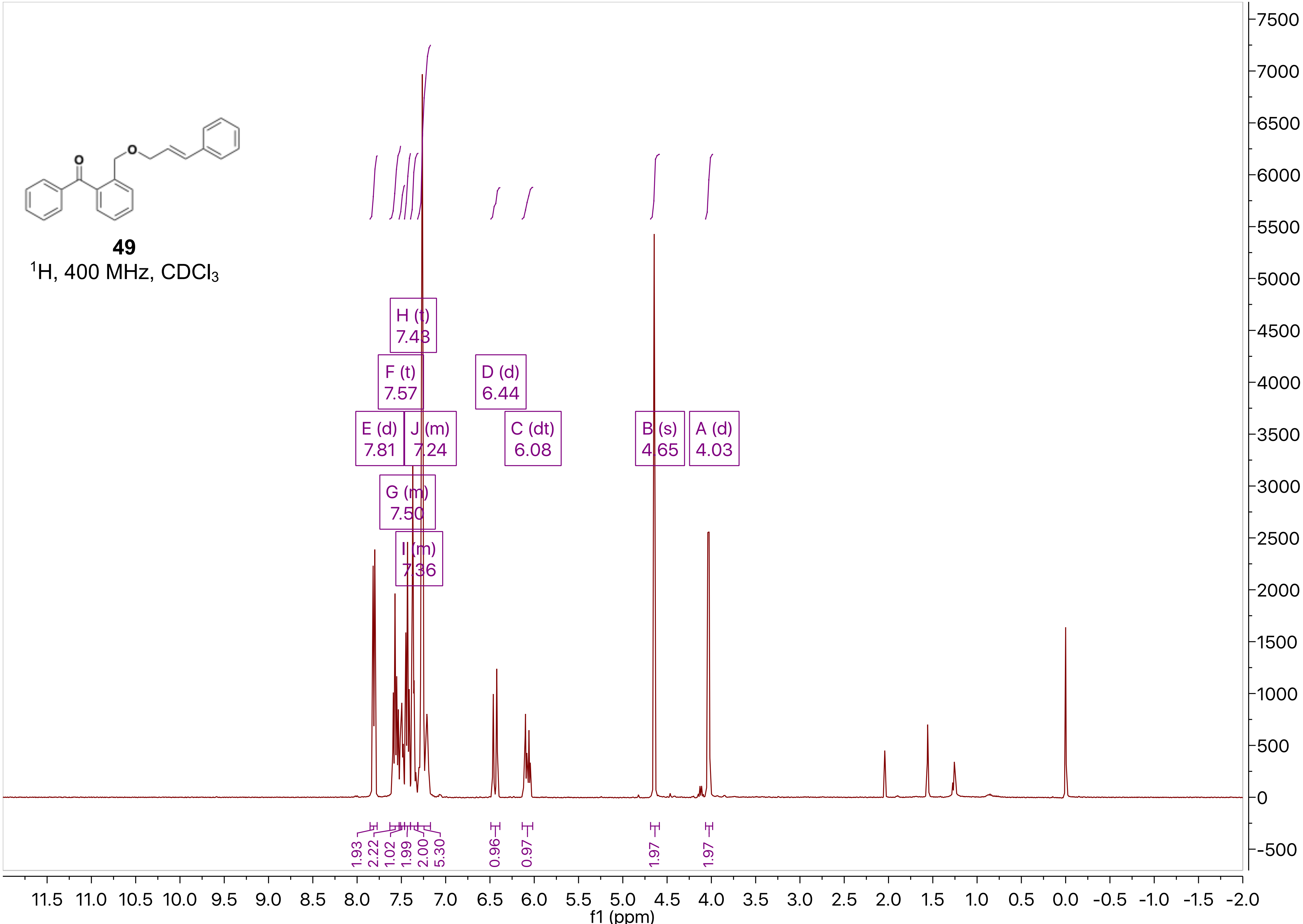

13a
^{13}C, 100 MHz, $CDCl_3$
141.71
137.06
137.03
134.11
130.29
130.12
127.96
126.96
126.63
126.54
124.18
116.43
79.29
68.52
48.74
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

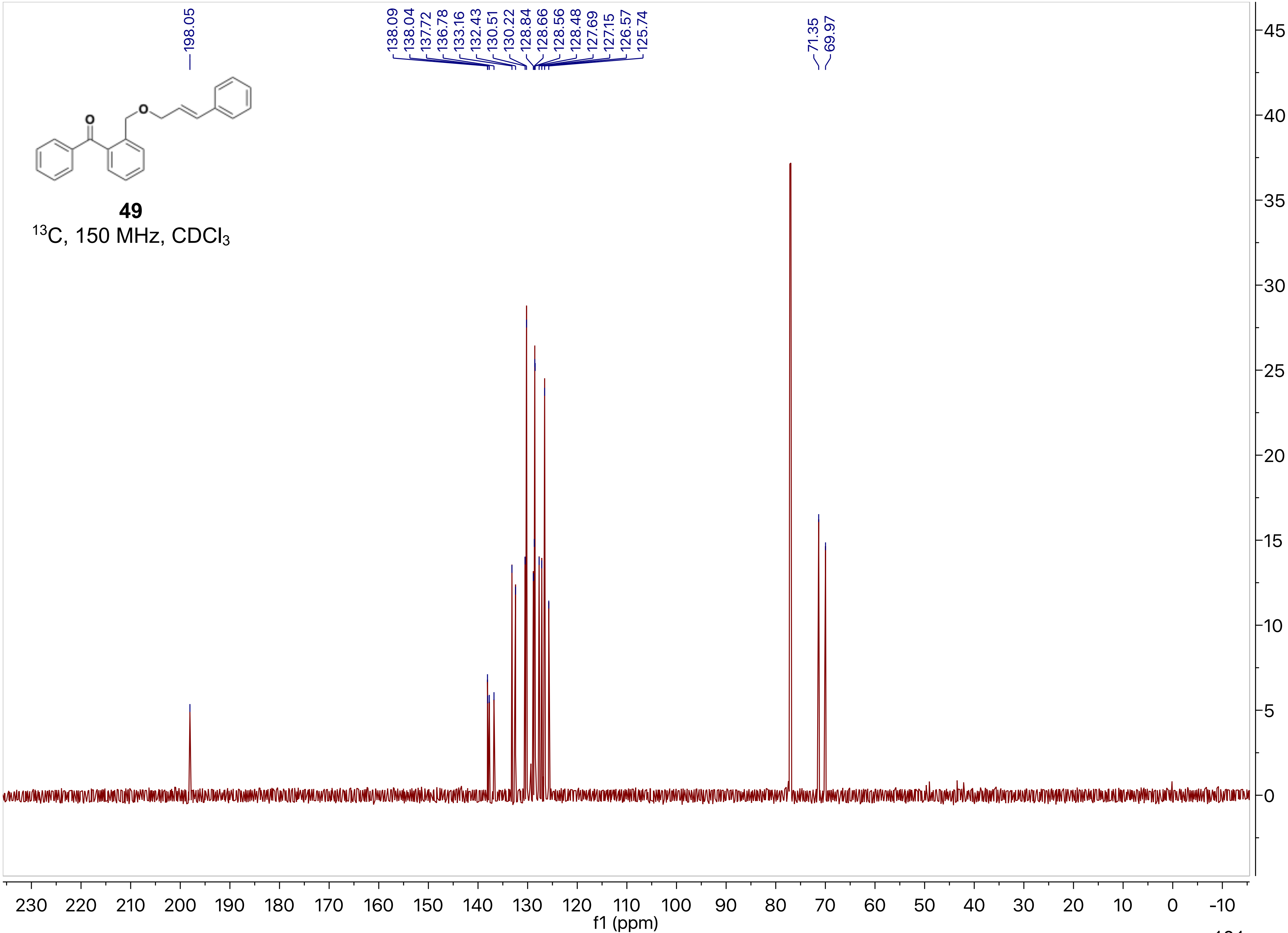

Br
O
48
1H, 600 MHz, CDCl3
D (t)
7.25
B (d)
7.41
G (dt)
6.36
I (d)
4.28
A (m)
7.53
F (d)
6.67
H (s)
4.64
C (t)
7.32
E (t)
7.15
1.71
1.87
2.69
1.07
0.91
0.91
0.92
1.94
1.97
f1 (ppm)



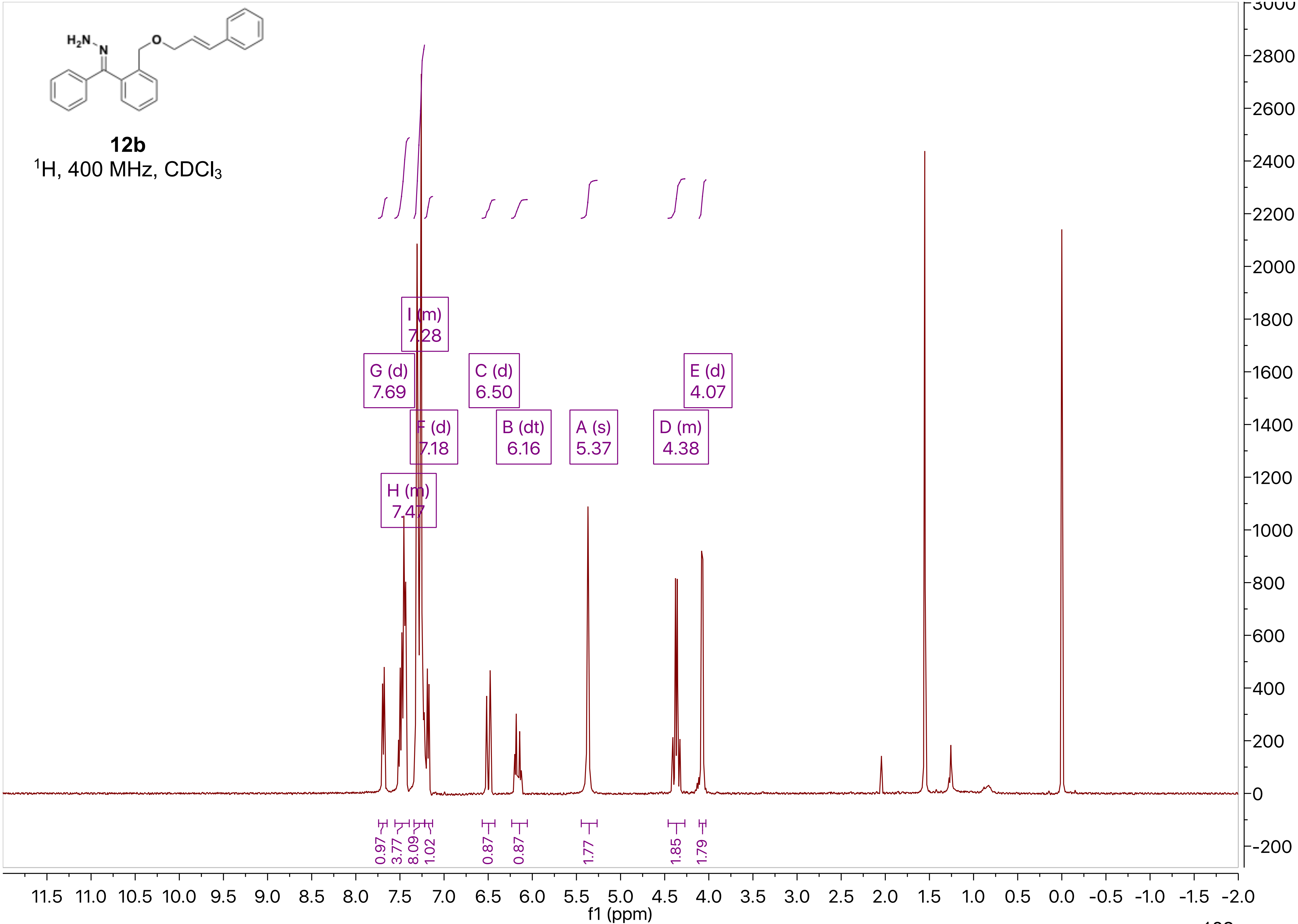
48

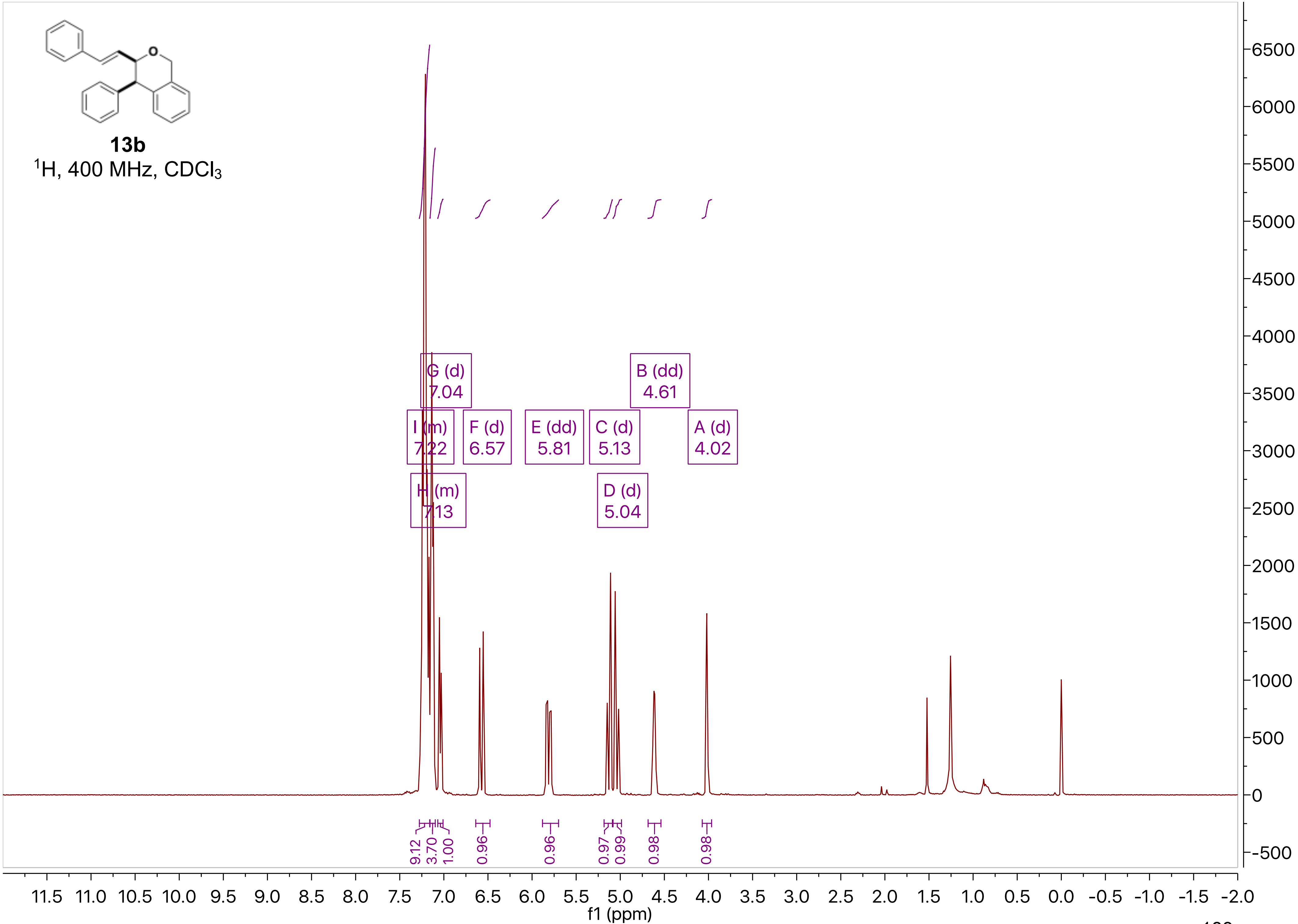
^{13}C, 100 MHz, $CDCl_3$

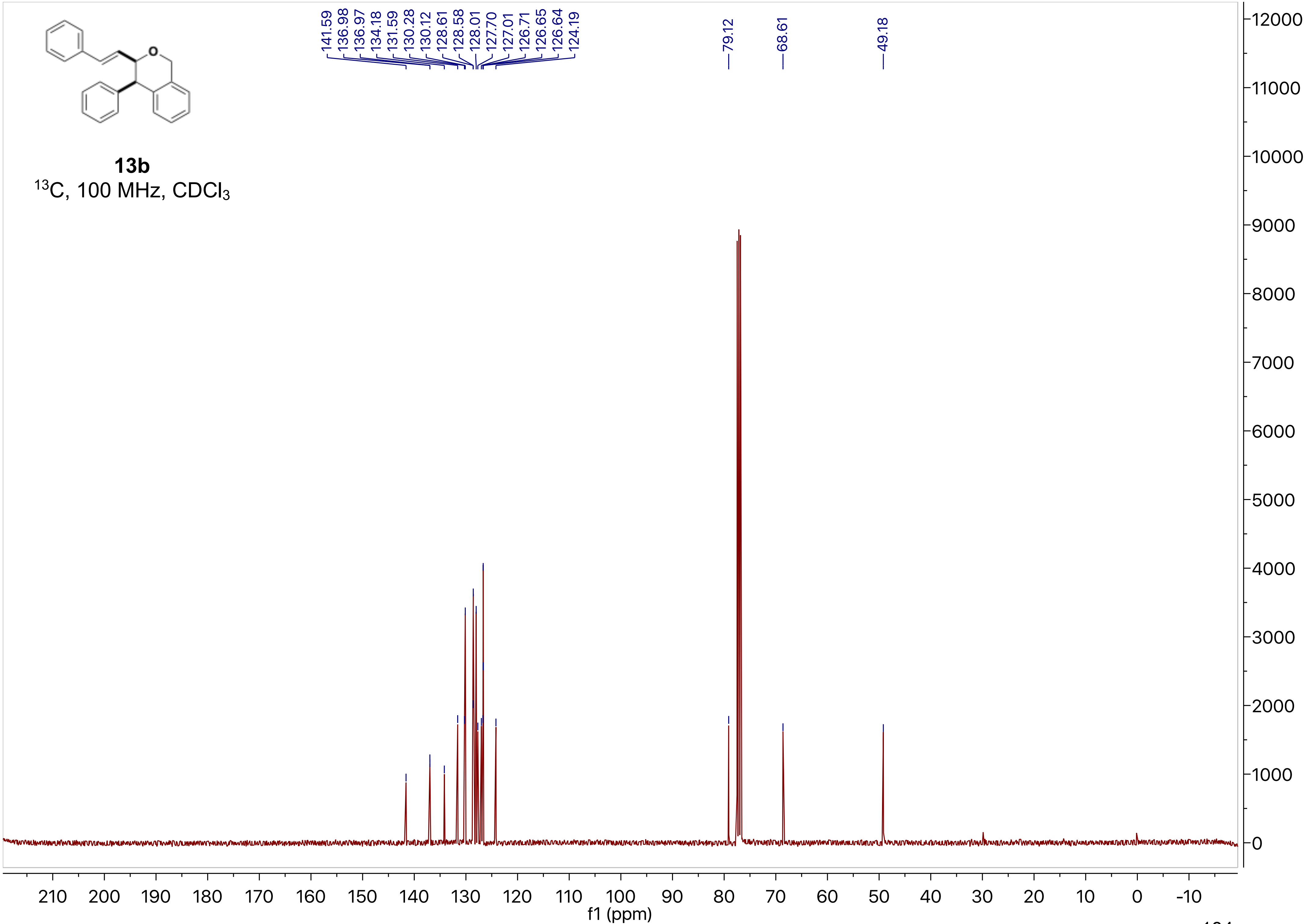

49
^{1}H, 400 MHz, $CDCl_3$
H (t) 7.43
F (t) 7.57
D (d) 6.44
E (d) 7.81
J (m) 7.24
C (dt) 6.08
B (s) 4.65
A (d) 4.03
G (m) 7.50
I (m) 7.36
1.93
2.22
1.02
1.99
2.00
5.30
0.96
0.97
1.97
1.97
f1 (ppm)

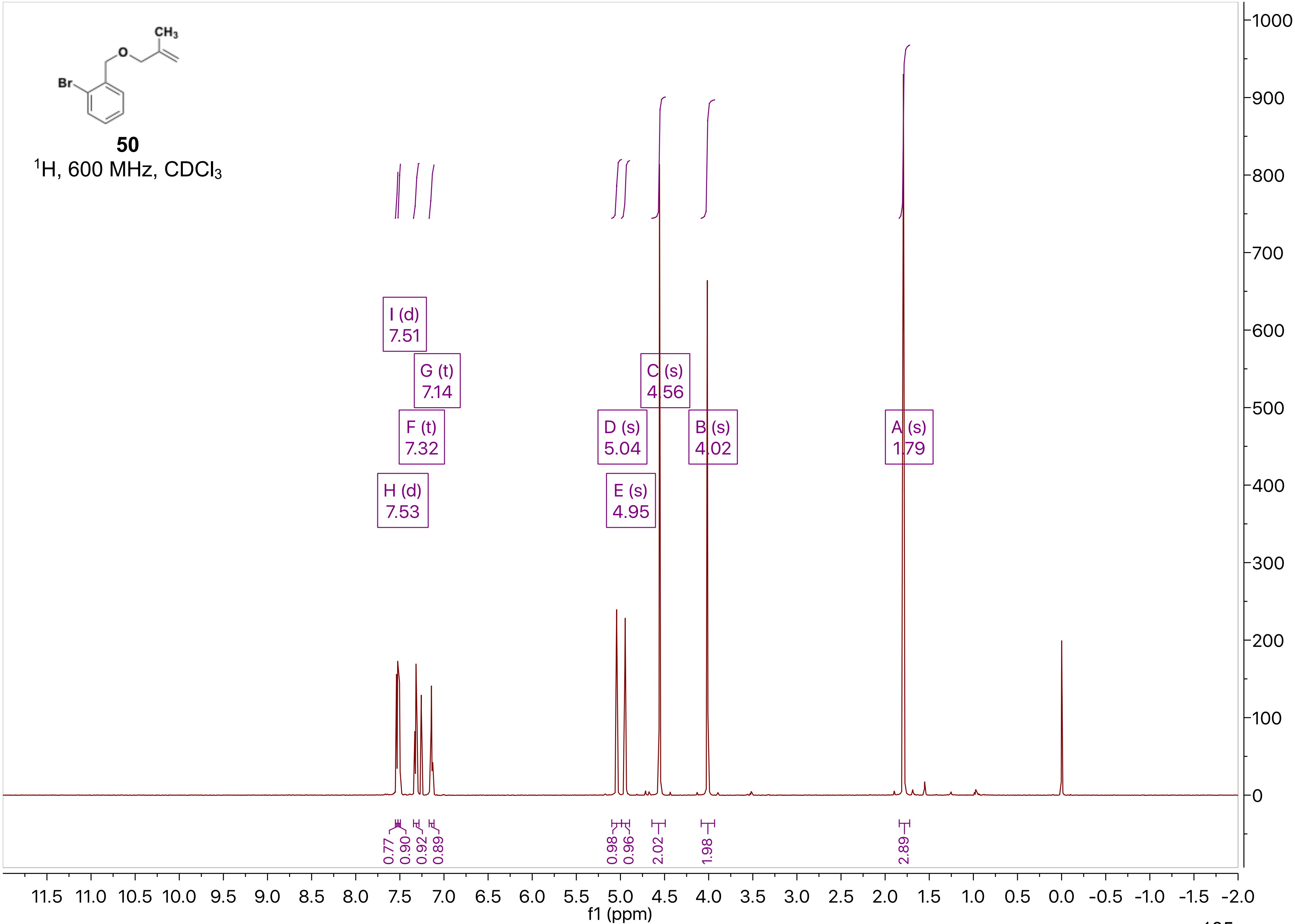


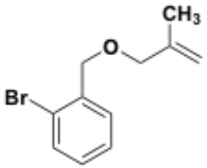
198.05
138.09
138.04
137.72
136.78
133.16
132.43
130.51
130.22
128.84
128.66
128.56
128.48
127.69
127.15
126.57
125.74
71.35
69.97
49
13C, 150 MHz, CDCl3
f1 (ppm)


12b
¹H, 400 MHz, CDCl₃
I (m) 7.28
G (d) 7.69
C (d) 6.50
E (d) 4.07
F (d) 7.18
B (dt) 6.16
A (s) 5.37
D (m) 4.38
H (m) 7.47
0.97
3.77
8.09
1.02
0.87
0.87
1.77
1.85
1.79
f1 (ppm)


13b
1H, 400 MHz, CDCl3
G (d) 7.04
B (dd) 4.61
I (m) 7.22
F (d) 6.57
E (dd) 5.81
C (d) 5.13
A (d) 4.02
H (m) 7.13
D (d) 5.04
9.12
3.70
1.00
0.96
0.96
0.97
0.99
0.98
0.98
f1 (ppm)

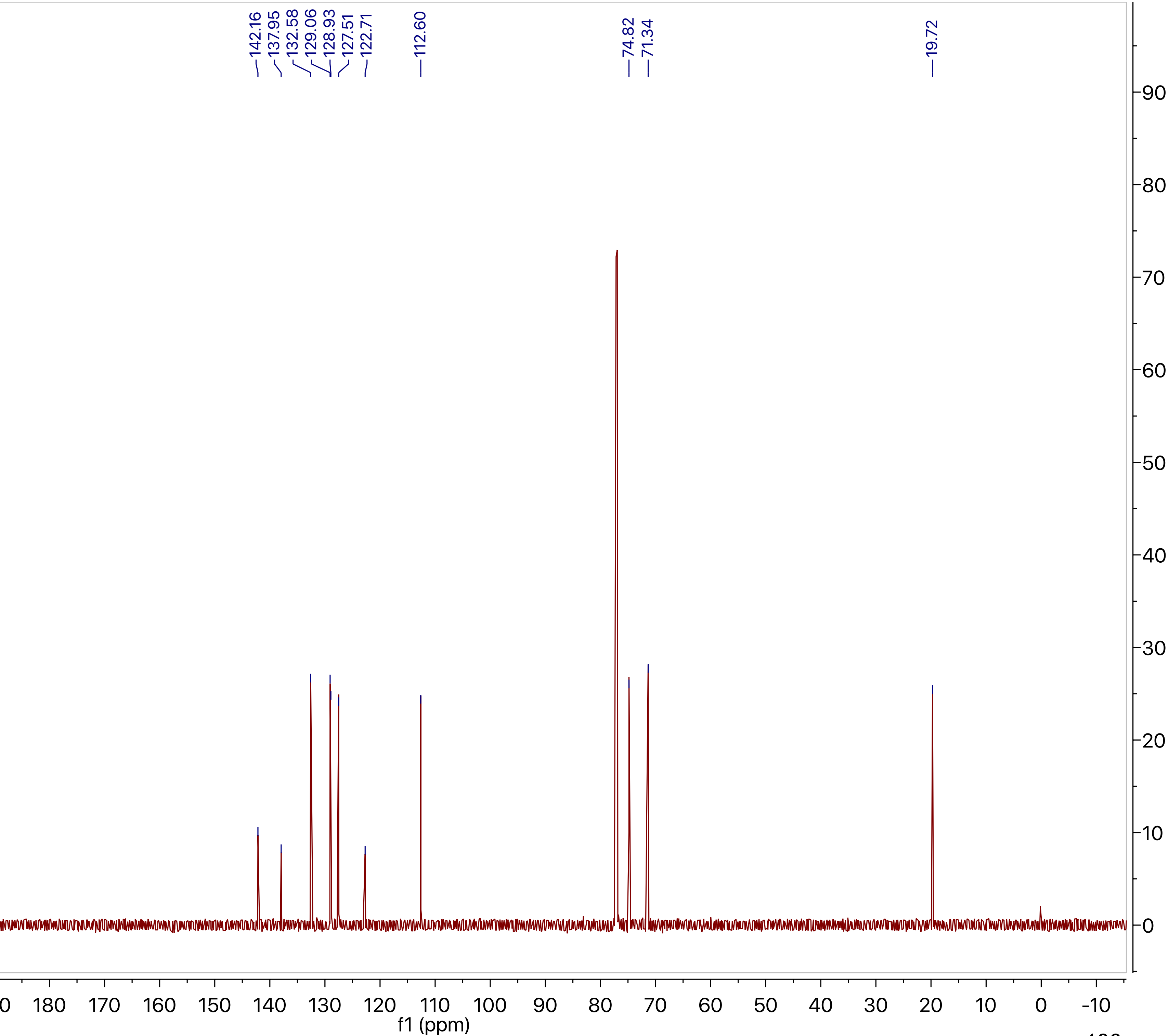

13b
13C, 100 MHz, CDCl3
141.59
136.98
136.97
134.18
131.59
130.28
130.12
128.61
128.58
128.01
127.70
127.01
126.71
126.65
126.64
124.19
79.12
68.61
49.18
f1 (ppm)

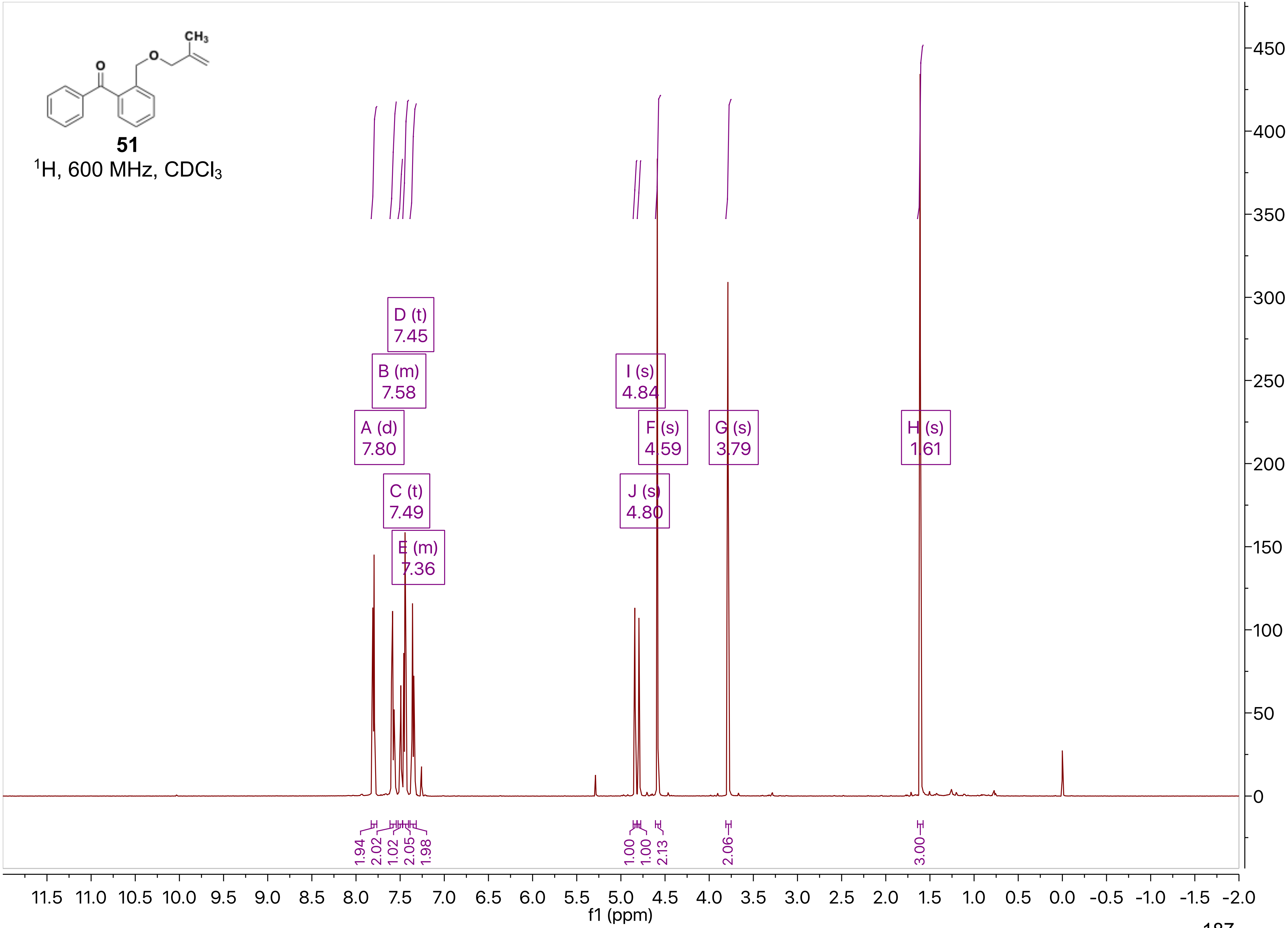

CH3
O
Br
50
1H, 600 MHz, CDCl3
I (d)
7.51
G (t)
7.14
F (t)
7.32
H (d)
7.53
D (s)
5.04
E (s)
4.95
C (s)
4.56
B (s)
4.02
A (s)
1.79
0.77
0.90
0.92
0.89
0.98
0.96
2.02
1.98
2.89
f1 (ppm)



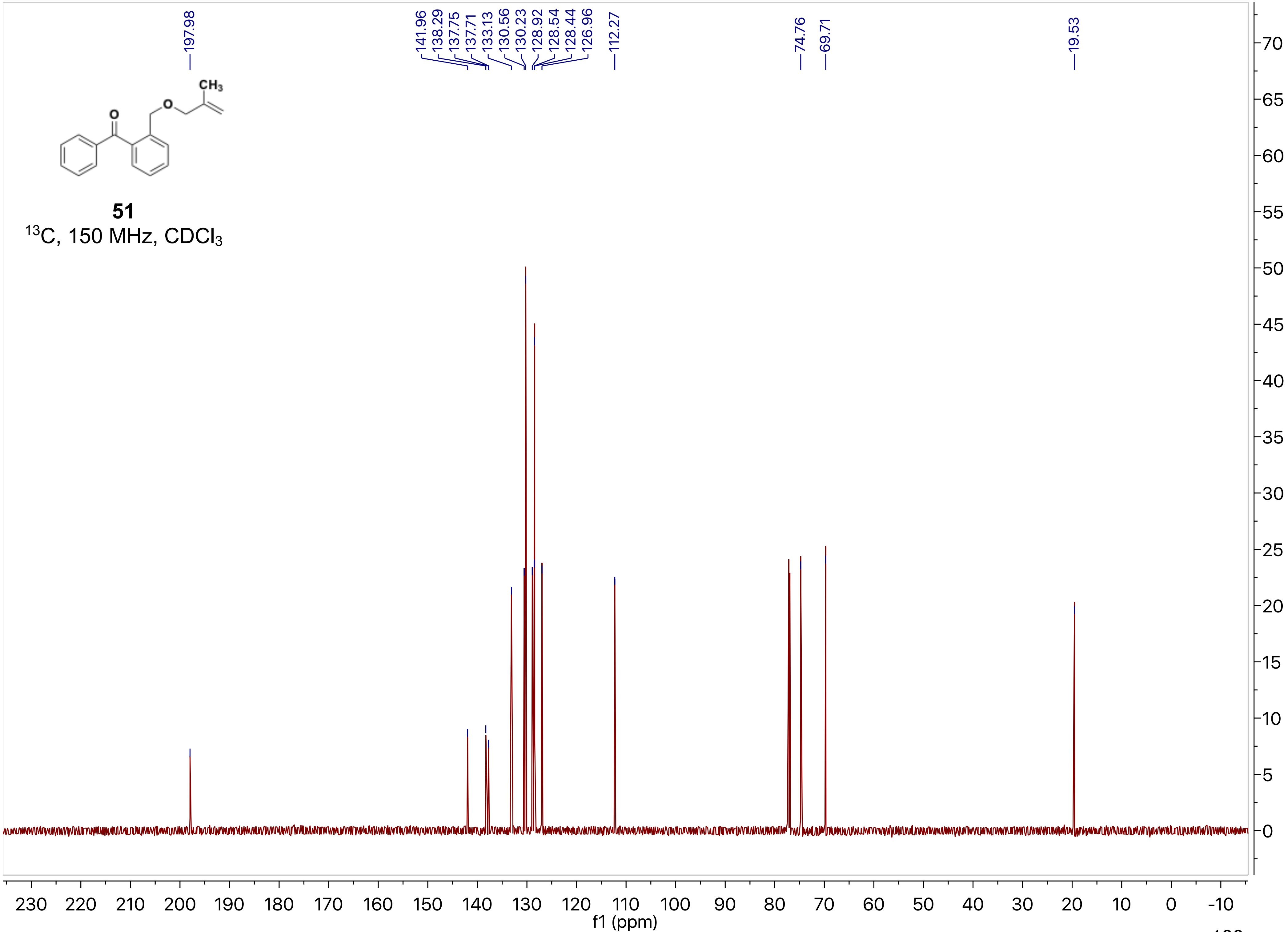
50

^{13}C, 150 MHz, $CDCl_3$

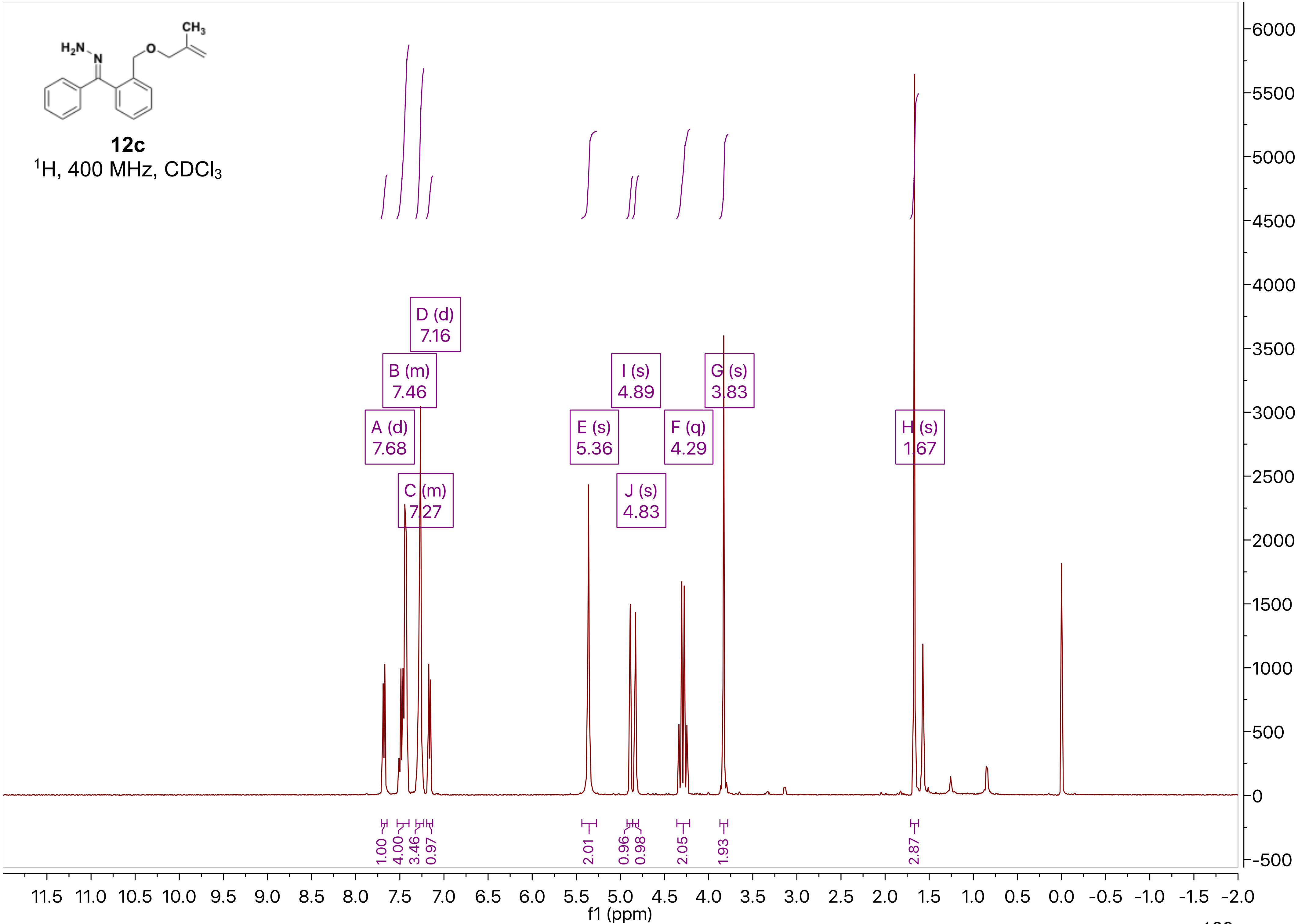




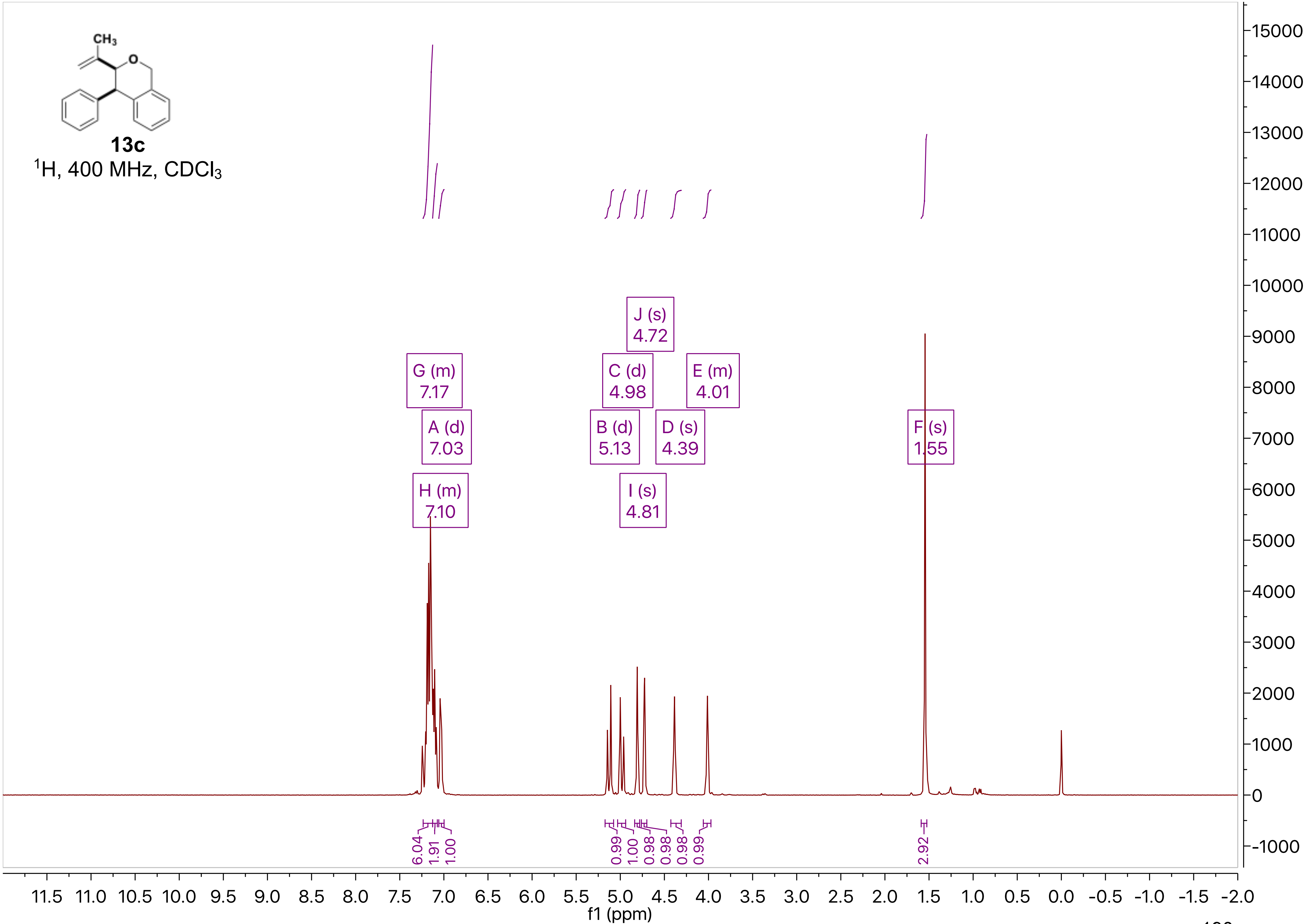
CH3
O
O
51
1H, 600 MHz, CDCl3
D (t) 7.45
B (m) 7.58
A (d) 7.80
C (t) 7.49
E (m) 7.36
I (s) 4.84
F (s) 4.59
J (s) 4.80
G (s) 3.79
H (s) 1.61
1.94
2.02
1.02
2.05
1.98
1.00
1.00
2.13
2.06
3.00
f1 (ppm)

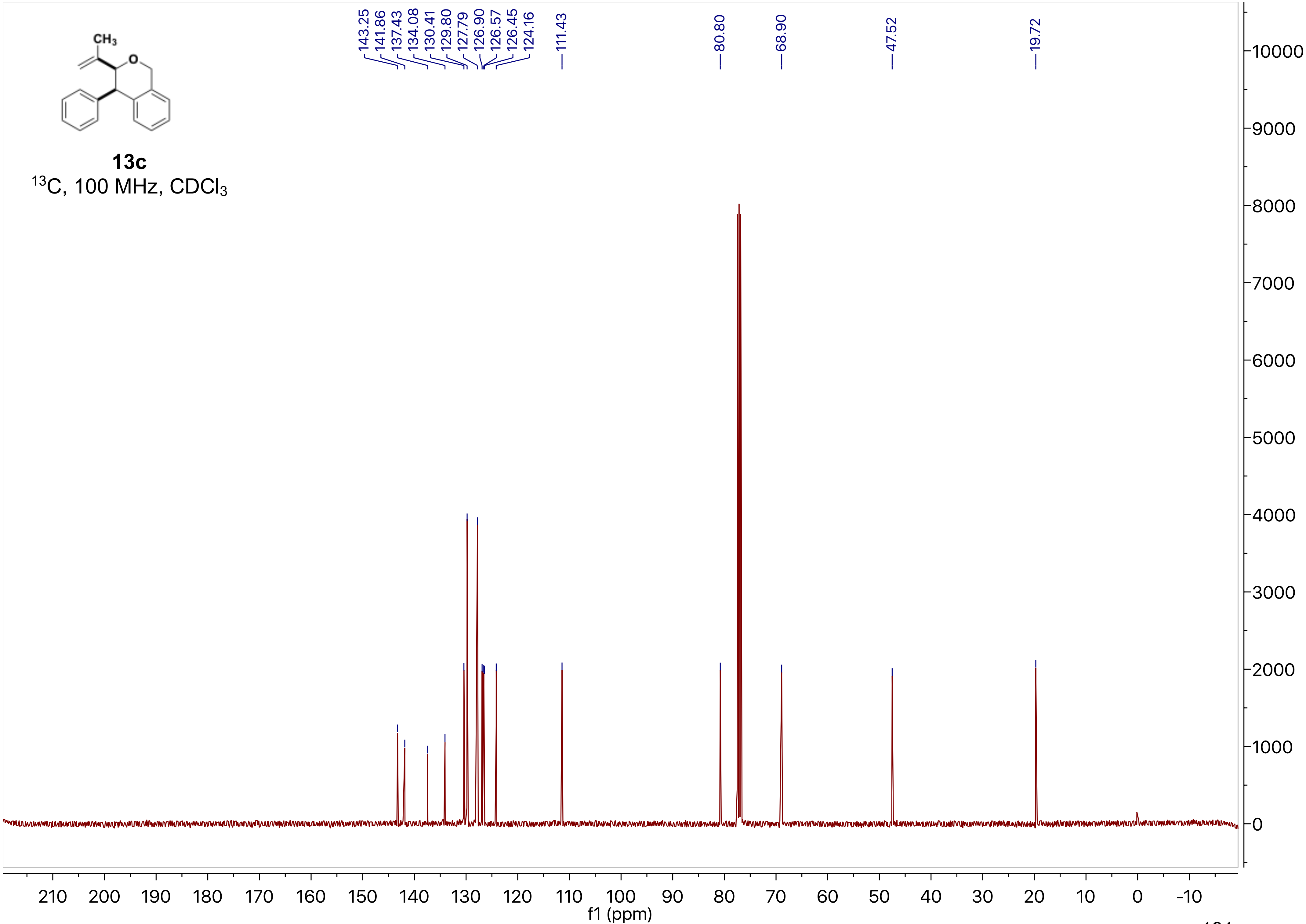


CH3
O
O
51
13C, 150 MHz, CDCl3
197.98
141.96
138.29
137.75
137.71
133.13
130.56
130.23
128.92
128.54
128.44
126.96
112.27
74.76
69.71
19.53
f1 (ppm)

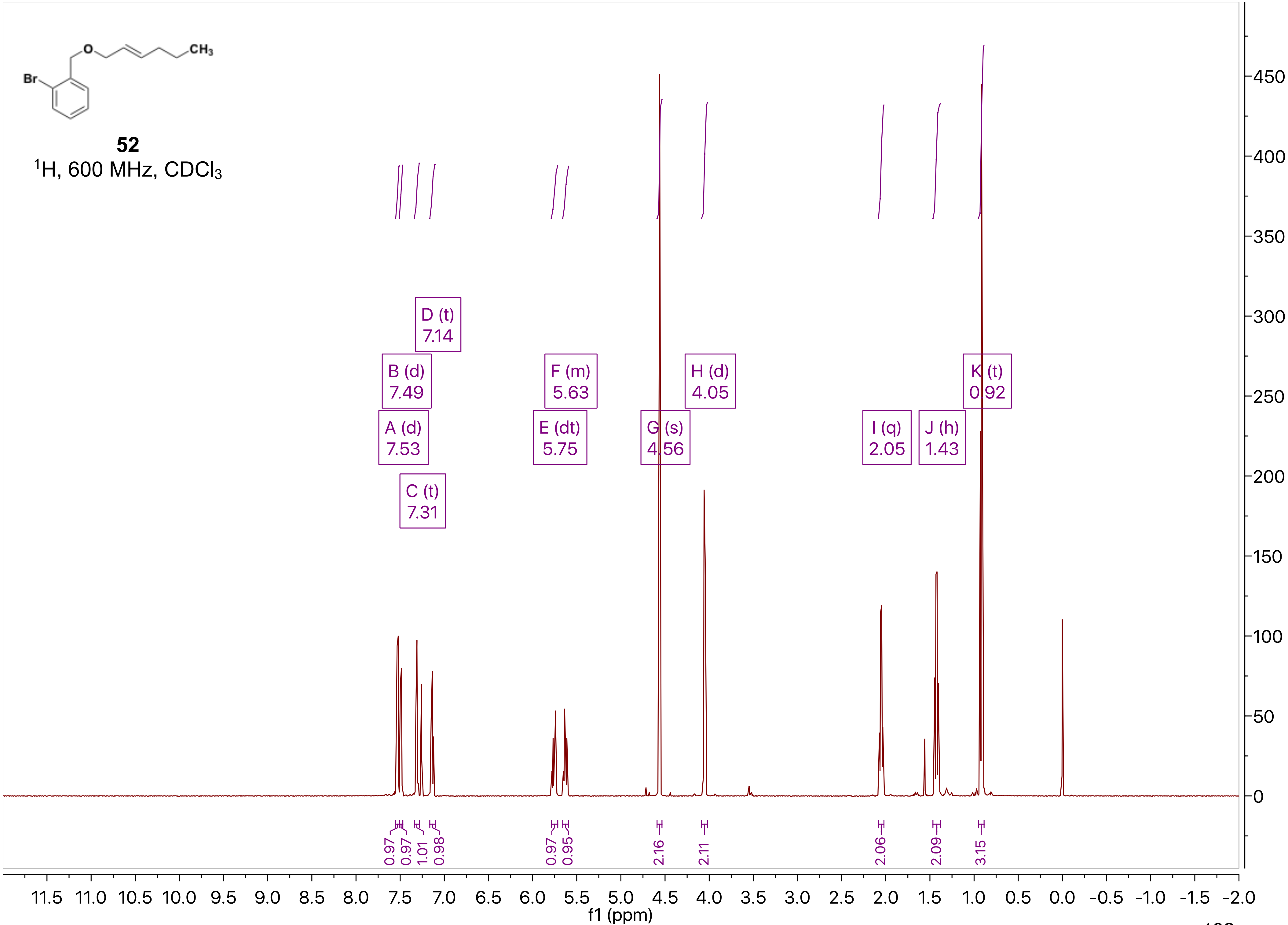


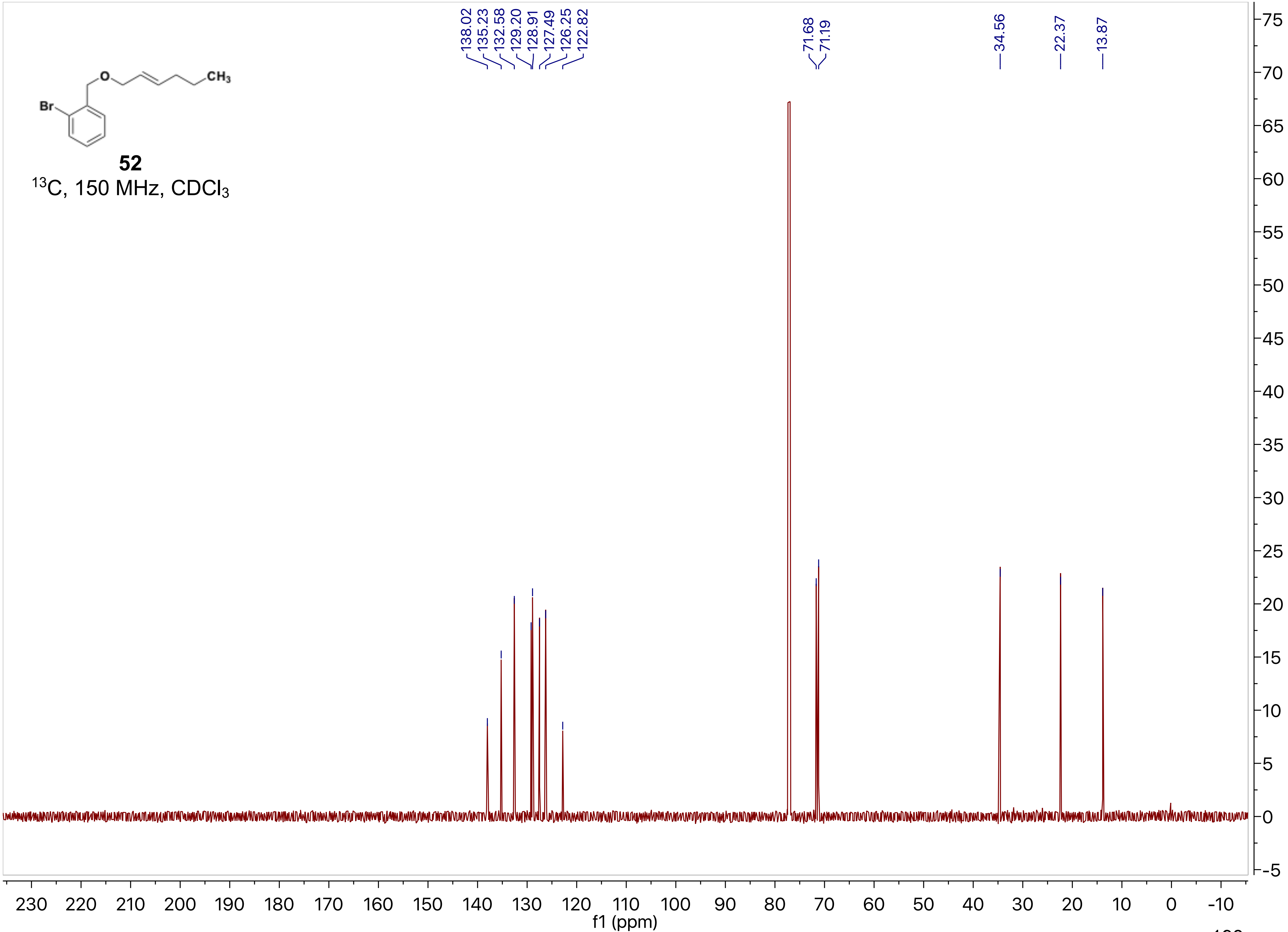
12c
^{1}H, 400 MHz, $CDCl_3$
A (d) 7.68
B (m) 7.46
C (m) 7.27
D (d) 7.16
E (s) 5.36
I (s) 4.89
J (s) 4.83
F (q) 4.29
G (s) 3.83
H (s) 1.67
1.00
4.00
3.46
0.97
2.01
0.96
0.98
2.05
1.93
2.87
f1 (ppm)

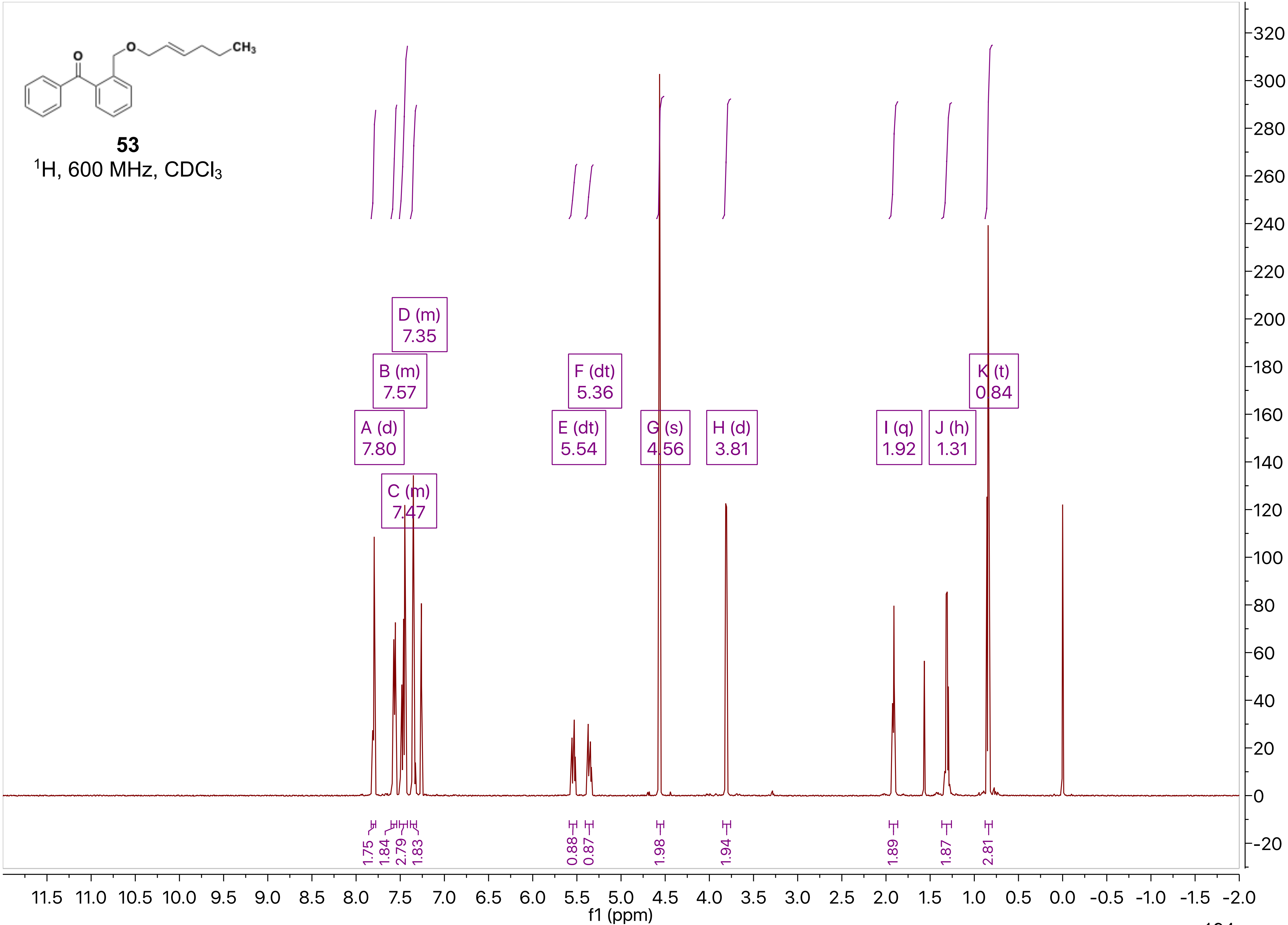

CH3
13c
1H, 400 MHz, CDCl3
G (m) 7.17
A (d) 7.03
H (m) 7.10
J (s) 4.72
C (d) 4.98
B (d) 5.13
I (s) 4.81
E (m) 4.01
D (s) 4.39
F (s) 1.55
6.04
1.91
1.00
0.99
1.00
0.98
0.98
0.98
0.99
2.92
f1 (ppm)

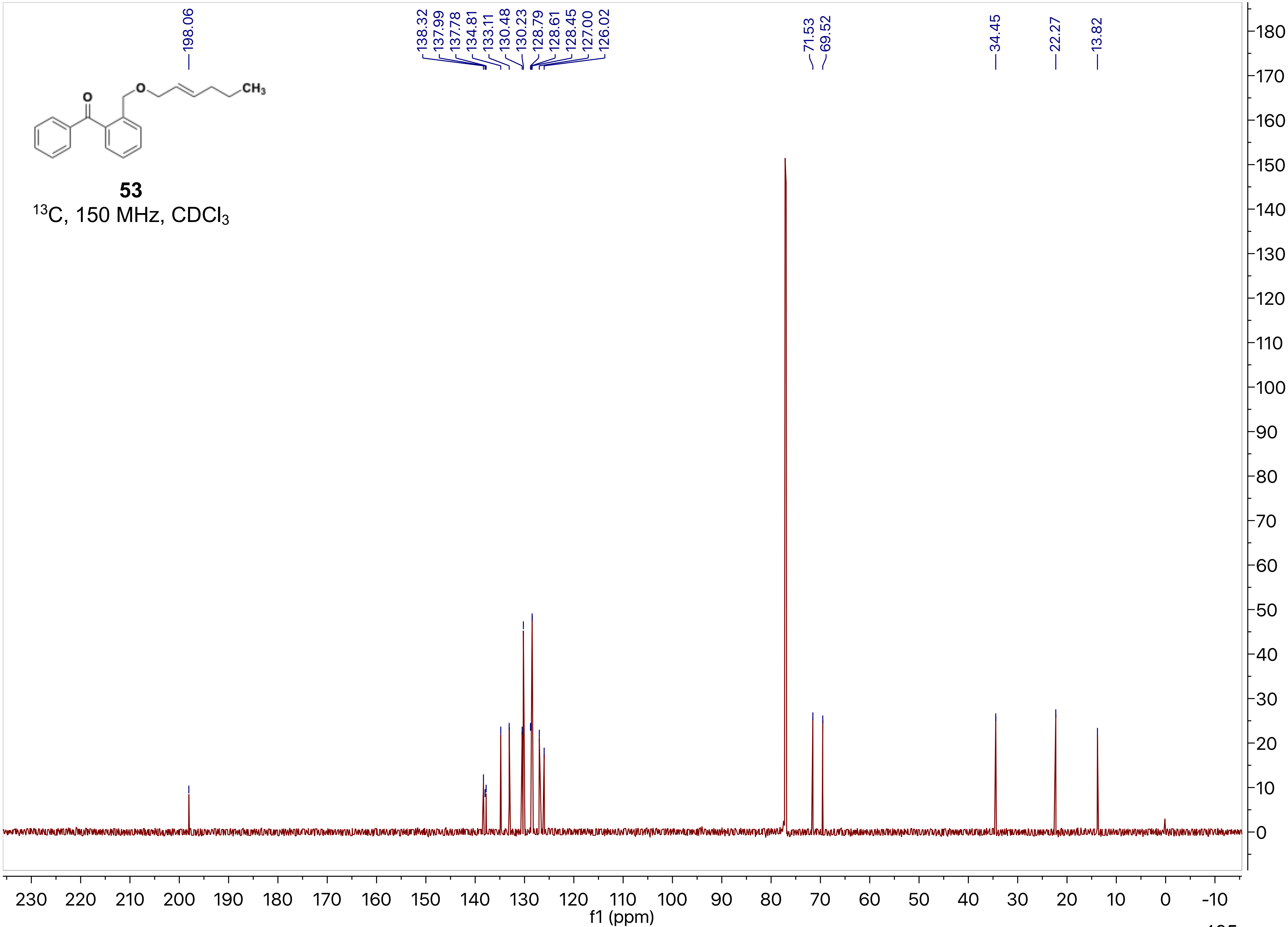


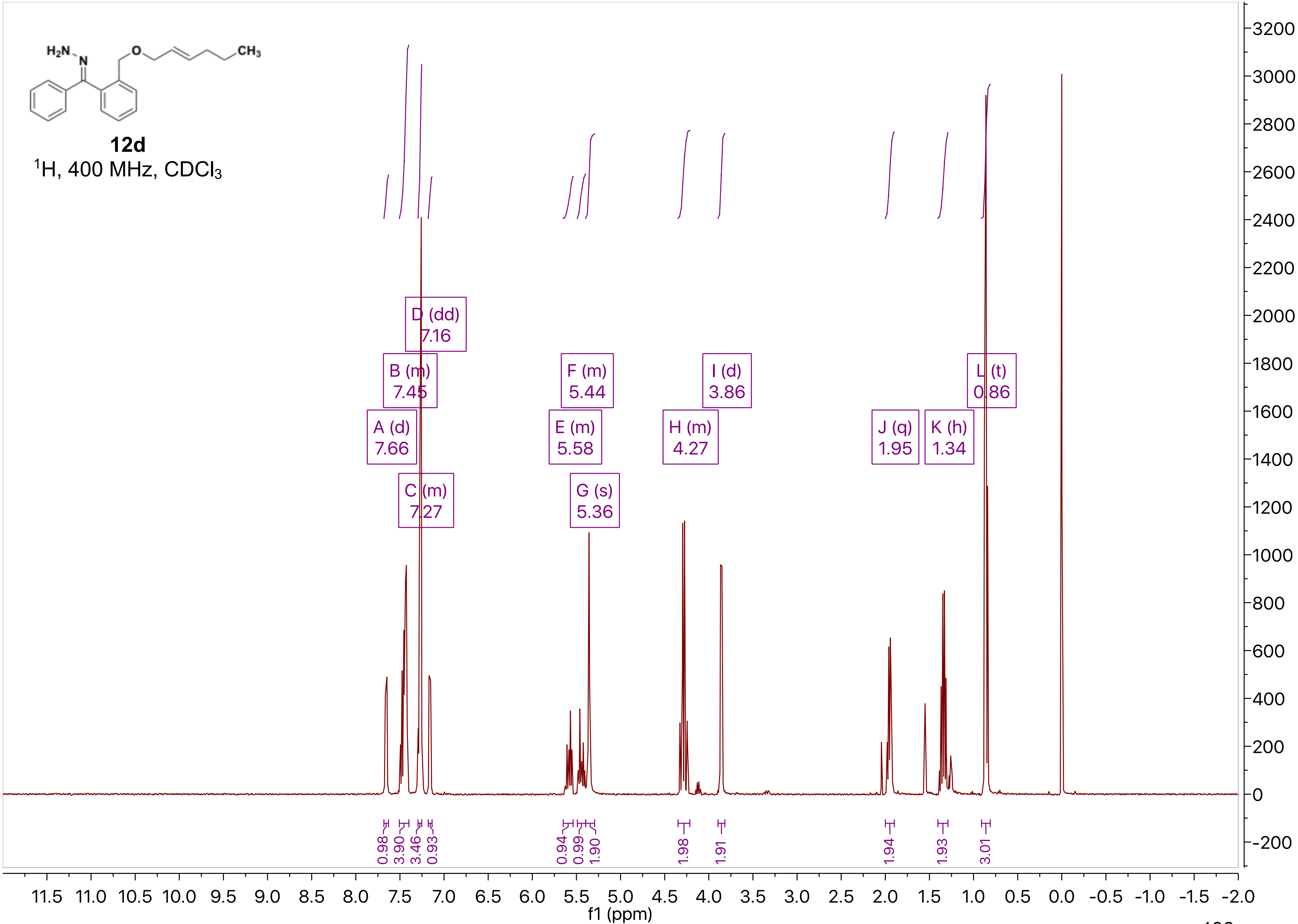
13c
^{13}C, 100 MHz, $CDCl_3$
143.25
141.86
137.43
134.08
130.41
129.80
127.79
126.90
126.57
126.45
124.16
111.43
80.80
68.90
47.52
19.72
f1 (ppm)


Br
O
CH3
52
1H, 600 MHz, CDCl3
D (t) 7.14
B (d) 7.49
A (d) 7.53
C (t) 7.31
F (m) 5.63
E (dt) 5.75
G (s) 4.56
H (d) 4.05
I (q) 2.05
J (h) 1.43
K (t) 0.92
0.97
0.97
1.01
0.98
0.97
0.95
2.16
2.11
2.06
2.09
3.15
f1 (ppm)

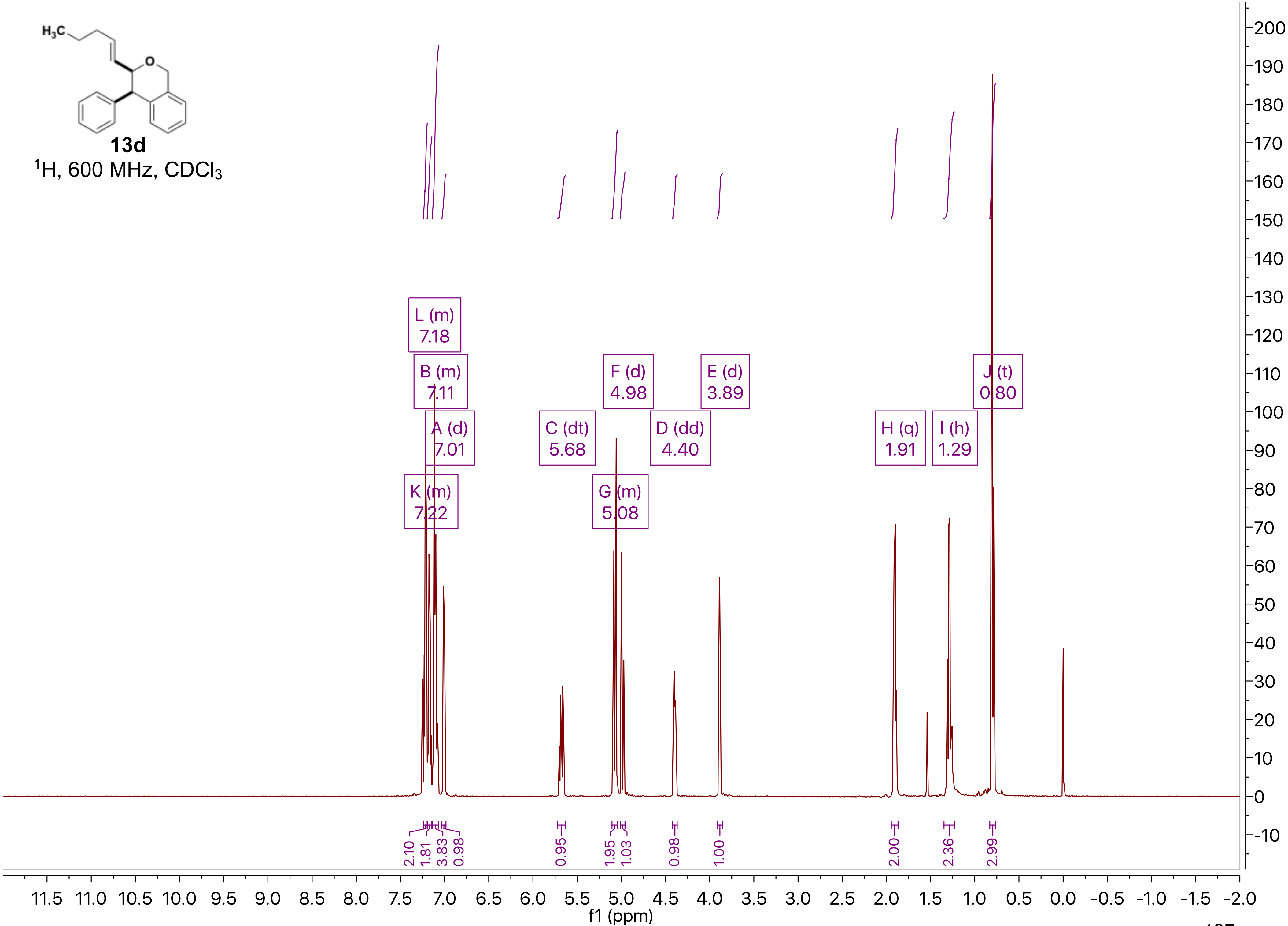

Br
O
CH3
52
13C, 150 MHz, CDCl3
138.02
135.23
132.58
129.20
128.91
127.49
126.25
122.82
71.68
71.19
34.56
22.37
13.87
f1 (ppm)


53
1H, 600 MHz, CDCl3
A (d) 7.80
B (m) 7.57
C (m) 7.47
D (m) 7.35
E (dt) 5.54
F (dt) 5.36
G (s) 4.56
H (d) 3.81
I (q) 1.92
J (h) 1.31
K (t) 0.84
1.75
1.84
2.79
1.83
0.88
0.87
1.98
1.94
1.89
1.87
2.81
f1 (ppm)

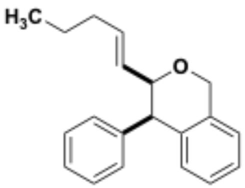

198.06
138.32
137.99
137.78
134.81
133.11
130.48
130.23
128.79
128.61
128.45
127.00
126.02
71.53
69.52
34.45
22.27
13.82
CH3
O
53
13C, 150 MHz, CDCl3
f1 (ppm)



12d
[1]H, 400 MHz, CDCl3
D (dd) 7.16
B (m) 7.45
A (d) 7.66
C (m) 7.27
F (m) 5.44
E (m) 5.58
G (s) 5.36
H (m) 4.27
I (d) 3.86
J (q) 1.95
K (h) 1.34
L (t) 0.86
0.98
3.90
3.46
0.93
0.94
0.99
1.90
1.98
1.91
1.94
1.93
3.01
f1 (ppm)

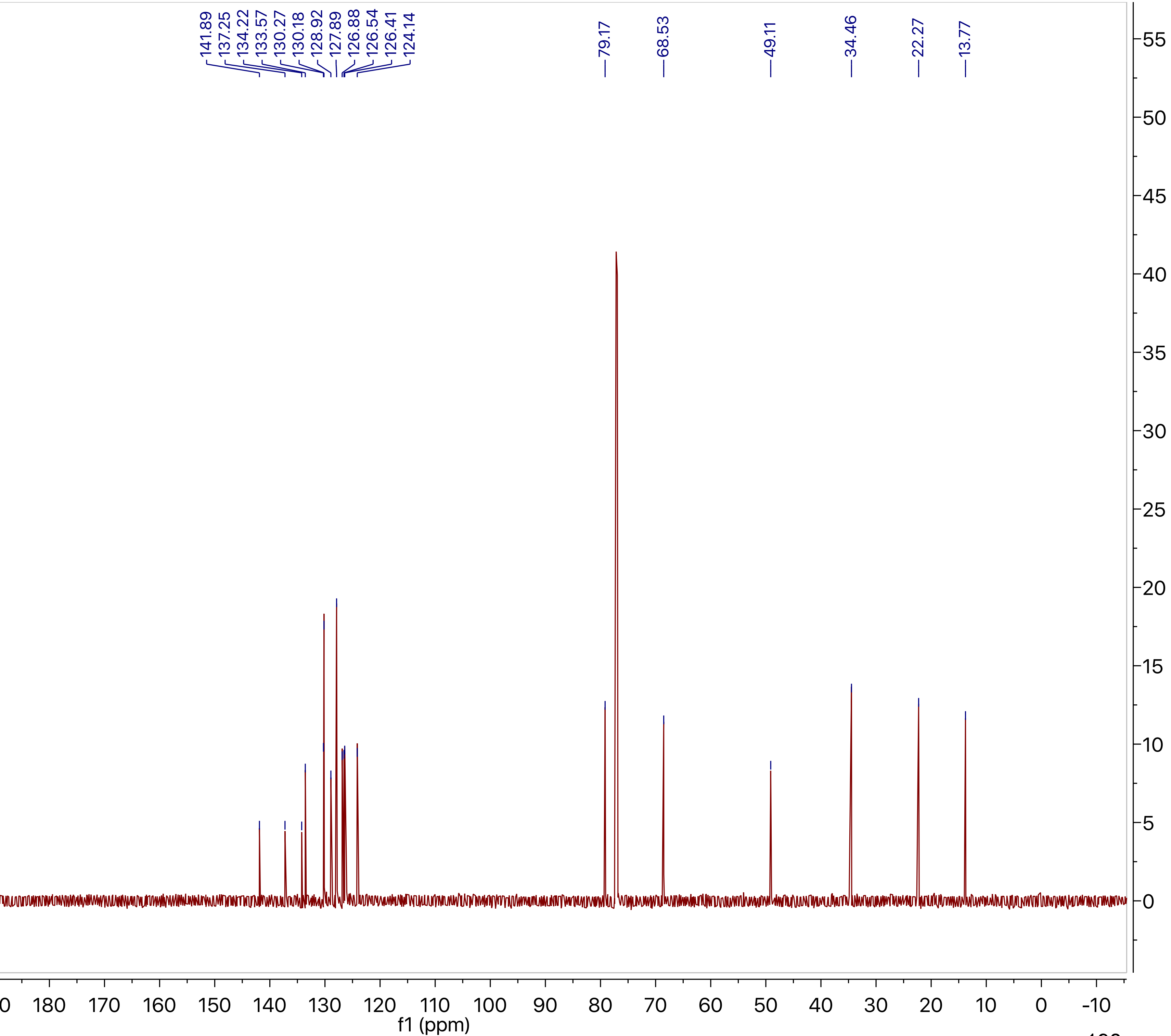


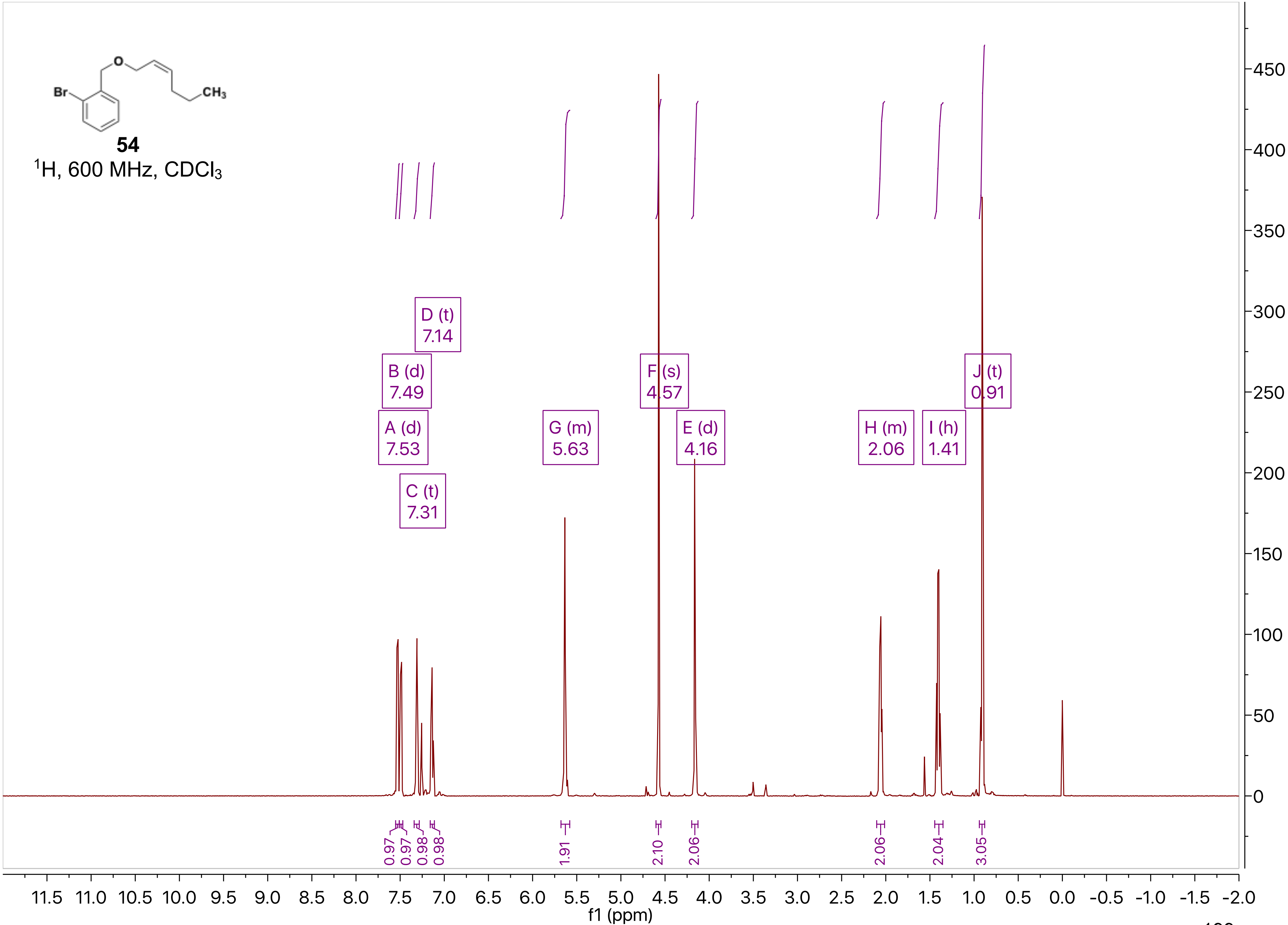
13d
1H, 600 MHz, CDCl3
L (m) 7.18
B (m) 7.11
A (d) 7.01
K (m) 7.22
C (dt) 5.68
F (d) 4.98
G (m) 5.08
D (dd) 4.40
E (d) 3.89
H (q) 1.91
I (h) 1.29
J (t) 0.80
2.10
1.81
3.83
0.98
0.95
1.95
1.03
0.98
1.00
2.00
2.36
2.99
f1 (ppm)

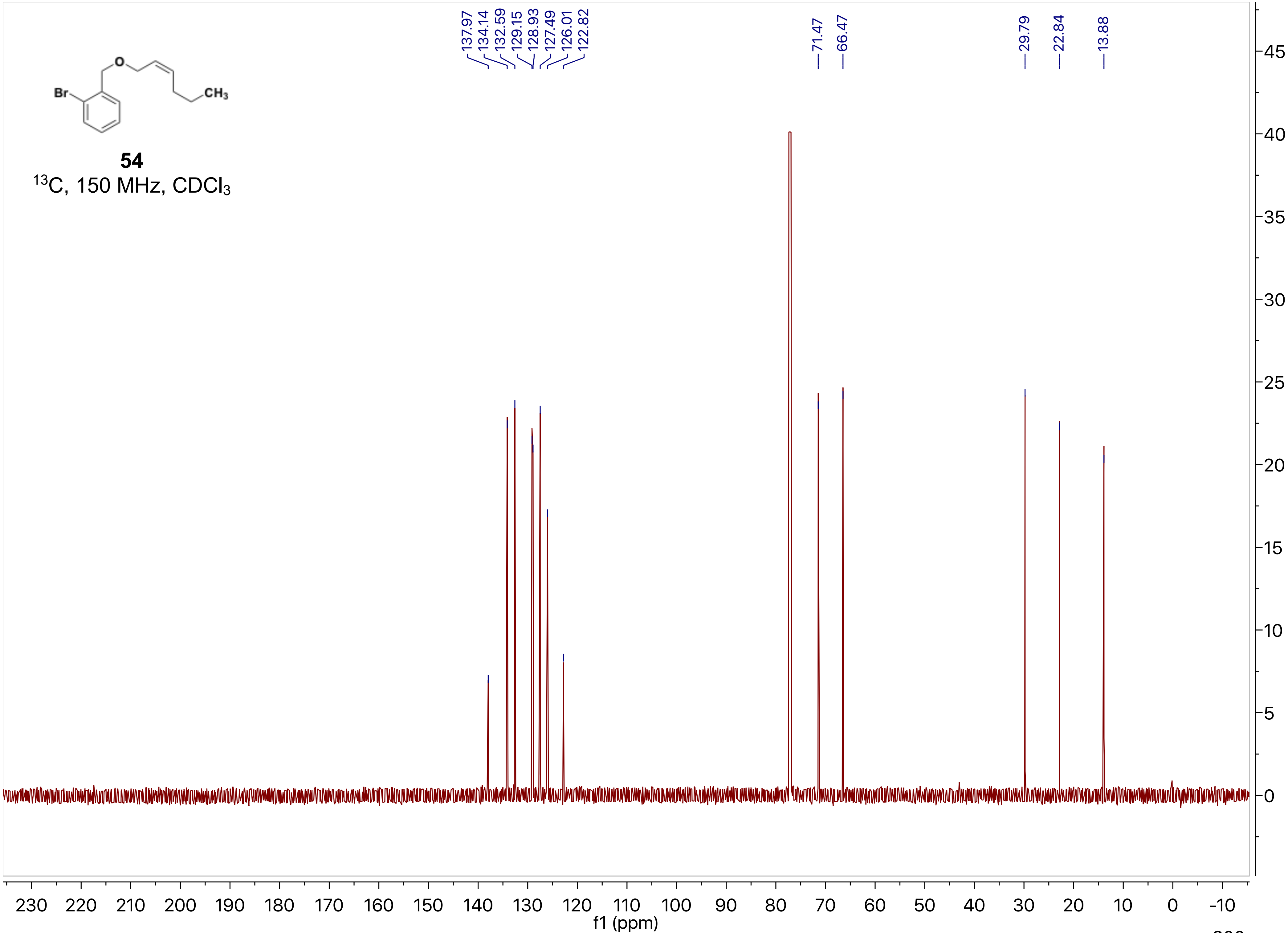


13d

^{13}C, 150 MHz, $CDCl_3$

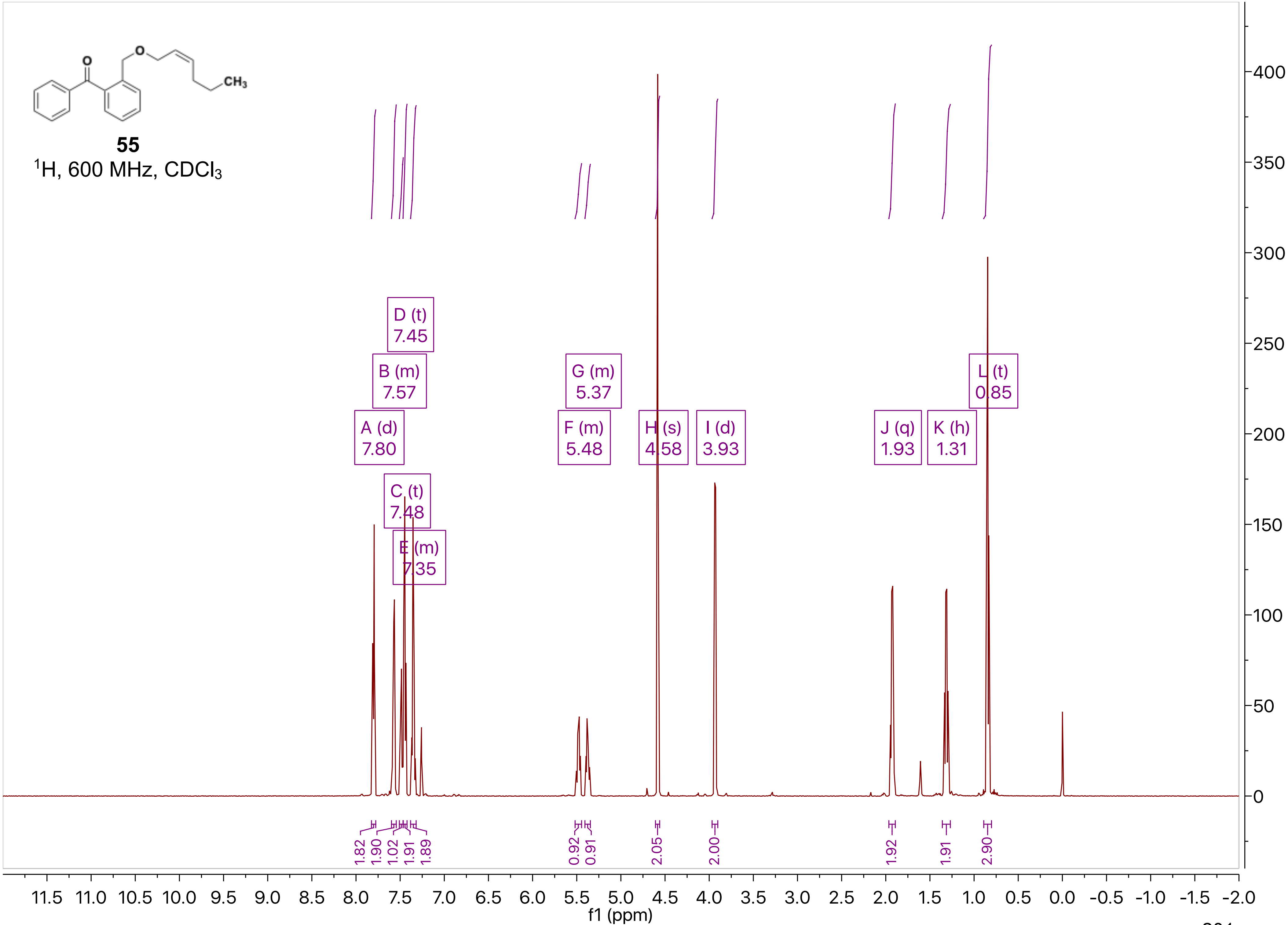



Br
O
CH3
54
1H, 600 MHz, CDCl3
D (t)
7.14
B (d)
7.49
A (d)
7.53
C (t)
7.31
G (m)
5.63
F (s)
4.57
E (d)
4.16
H (m)
2.06
I (h)
1.41
J (t)
0.91
0.97
0.97
0.98
0.98
1.91
2.10
2.06
2.06
2.04
3.05
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5 -2.0
f1 (ppm)
450
400
350
300
250
200
150
100
50
0

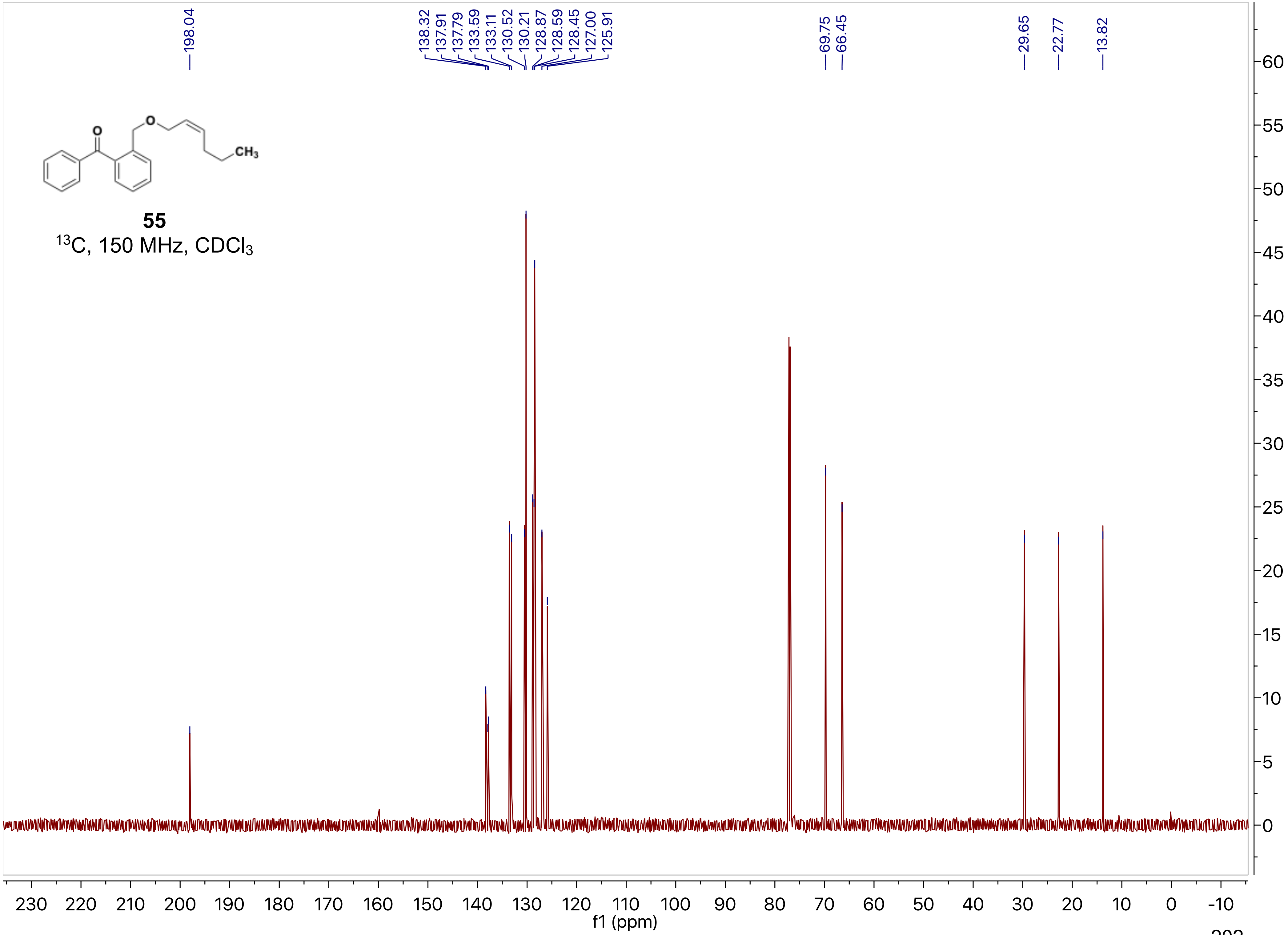


54

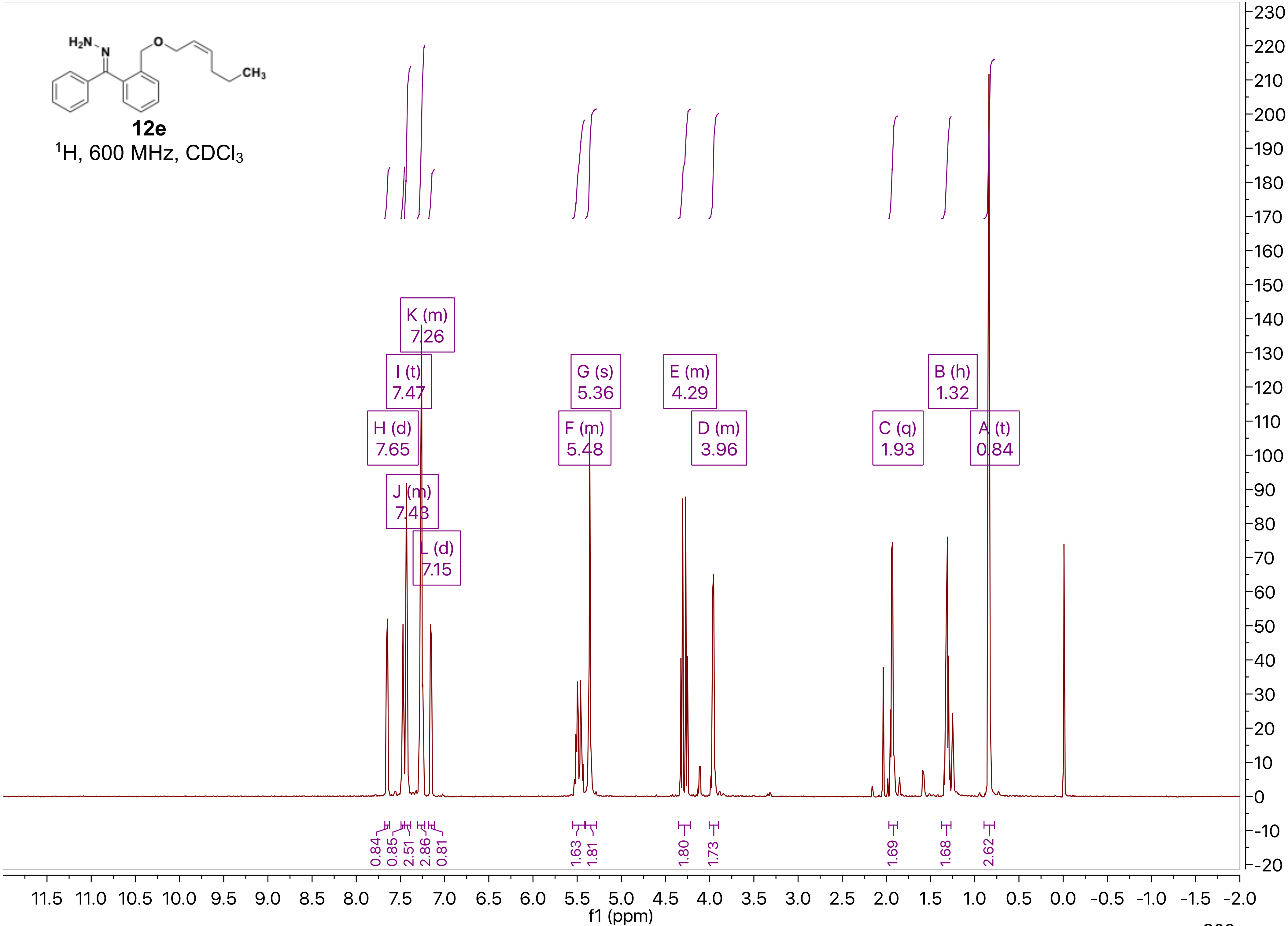
^{13}C, 150 MHz, $CDCl_3$



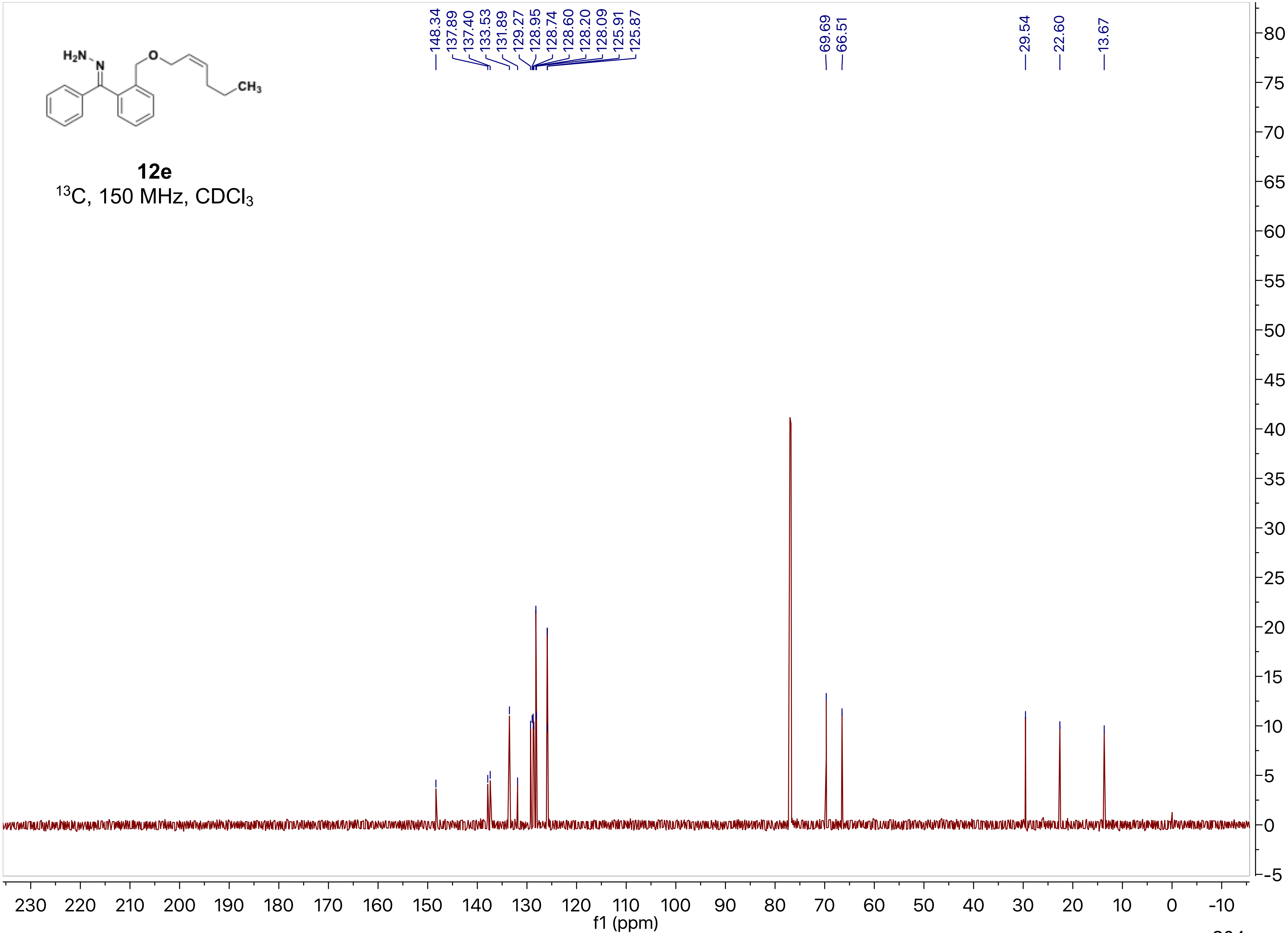
55
1H, 600 MHz, CDCl3
A (d) 7.80
B (m) 7.57
C (t) 7.48
D (t) 7.45
E (m) 7.35
F (m) 5.48
G (m) 5.37
H (s) 4.58
I (d) 3.93
J (q) 1.93
K (h) 1.31
L (t) 0.85
1.82
1.90
1.02
1.91
1.89
0.92
0.91
2.05
2.00
1.92
1.91
2.90
f1 (ppm)



198.04
138.32
137.91
137.79
133.59
133.11
130.52
130.21
128.87
128.59
128.45
127.00
125.91
69.75
66.45
29.65
22.77
13.82
O
O
CH3
55
13C, 150 MHz, CDCl3
f1 (ppm)

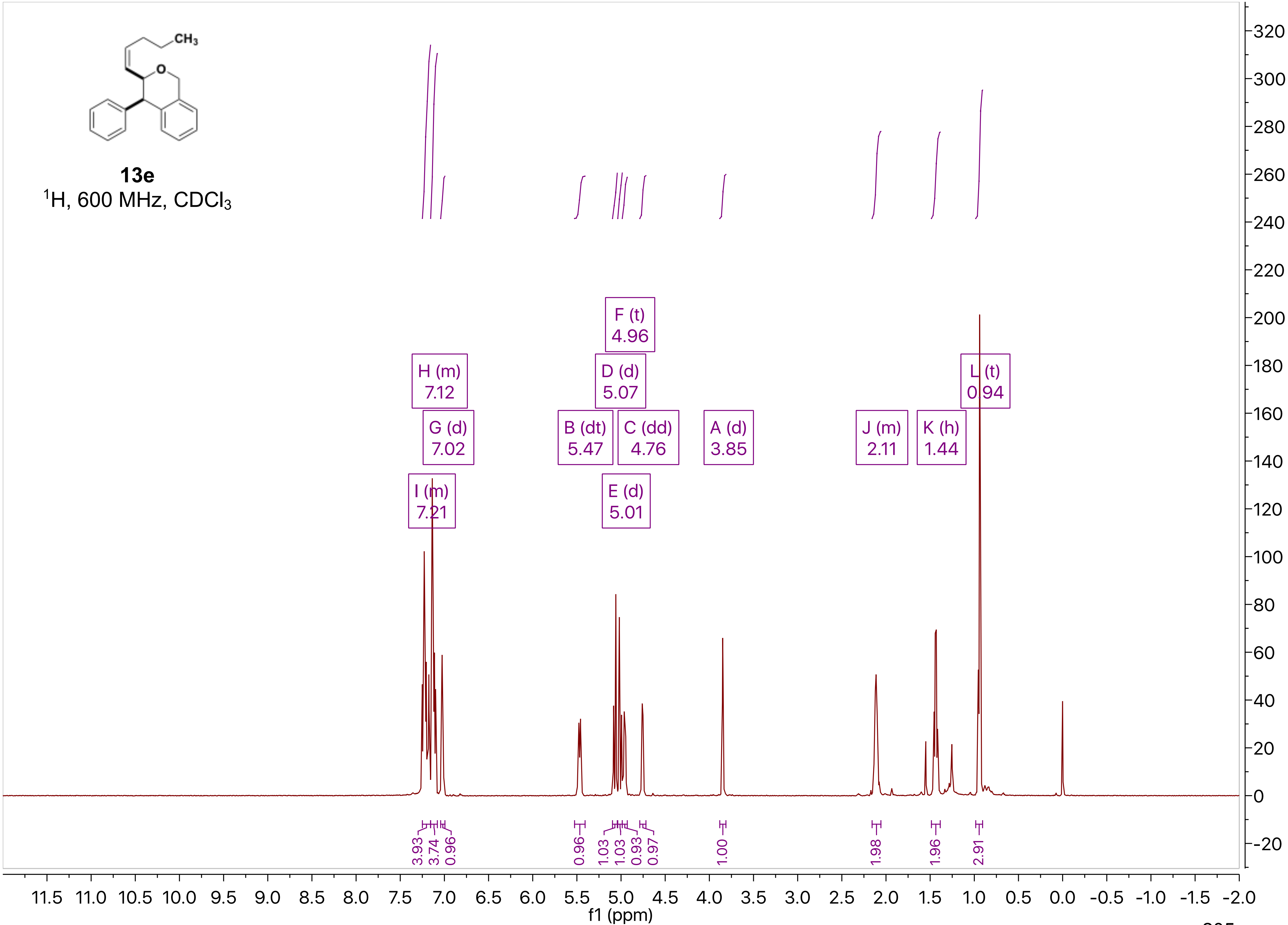


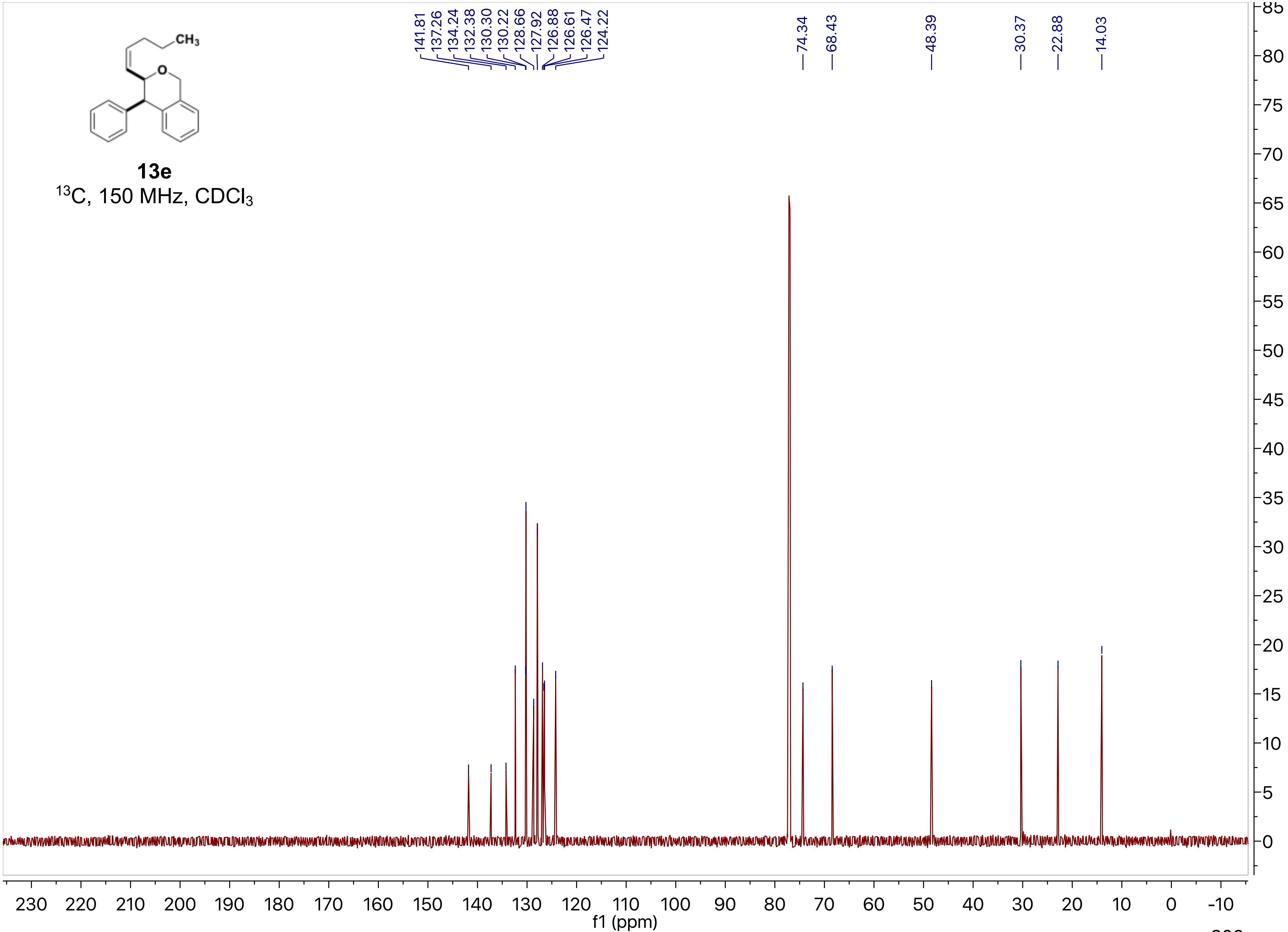
H₂N
N
O
CH₃
12e
¹H, 600 MHz, CDCl₃
K (m) 7.26
I (t) 7.47
H (d) 7.65
J (m) 7.43
L (d) 7.15
G (s) 5.36
F (m) 5.48
E (m) 4.29
D (m) 3.96
C (q) 1.93
B (h) 1.32
A (t) 0.84
0.84 0.85 2.51 2.86 0.81 1.63 1.81 1.80 1.73 1.69 1.68 2.62
f1 (ppm)



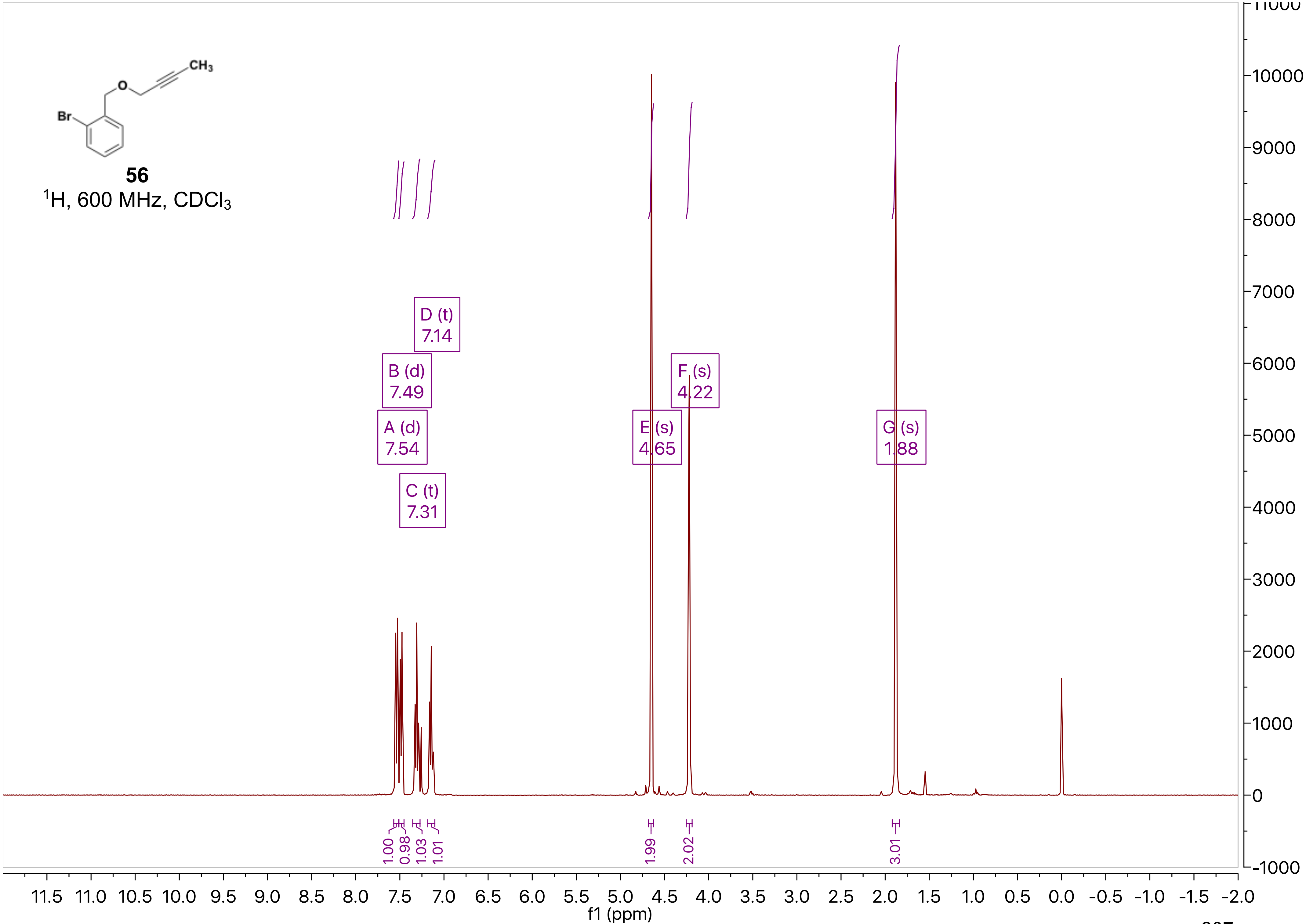
12e

^{13}C, 150 MHz, $CDCl_3$

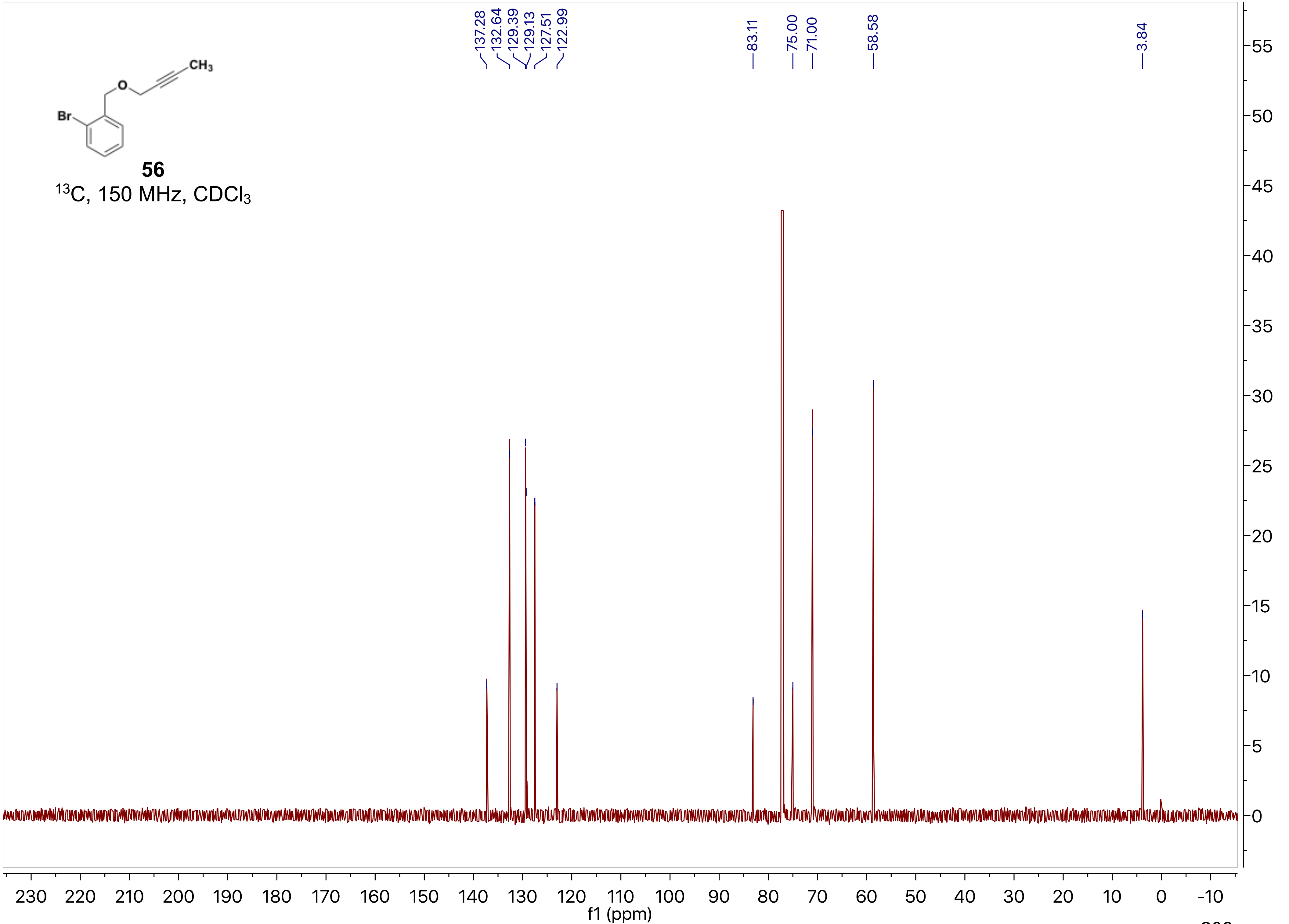

CH3
O
13e
1H, 600 MHz, CDCl3
F (t)
4.96
H (m)
7.12
D (d)
5.07
L (t)
0.94
G (d)
7.02
B (dt)
5.47
C (dd)
4.76
A (d)
3.85
J (m)
2.11
K (h)
1.44
I (m)
7.21
E (d)
5.01
3.93
3.74
0.96
0.96
1.03
1.03
0.93
0.97
1.00
1.98
1.96
2.91
f1 (ppm)

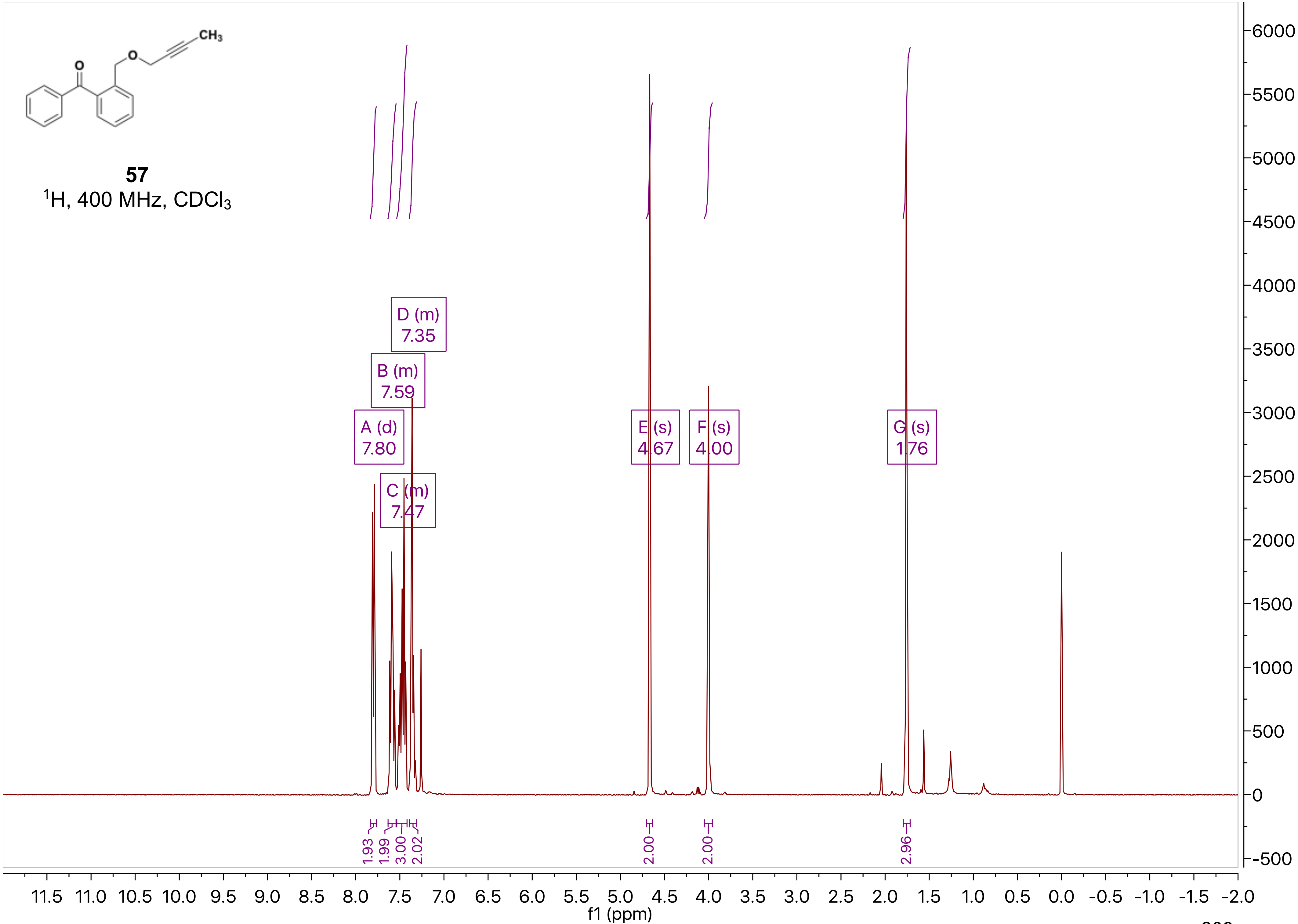


13e
^{13}C, 150 MHz, $CDCl_3$

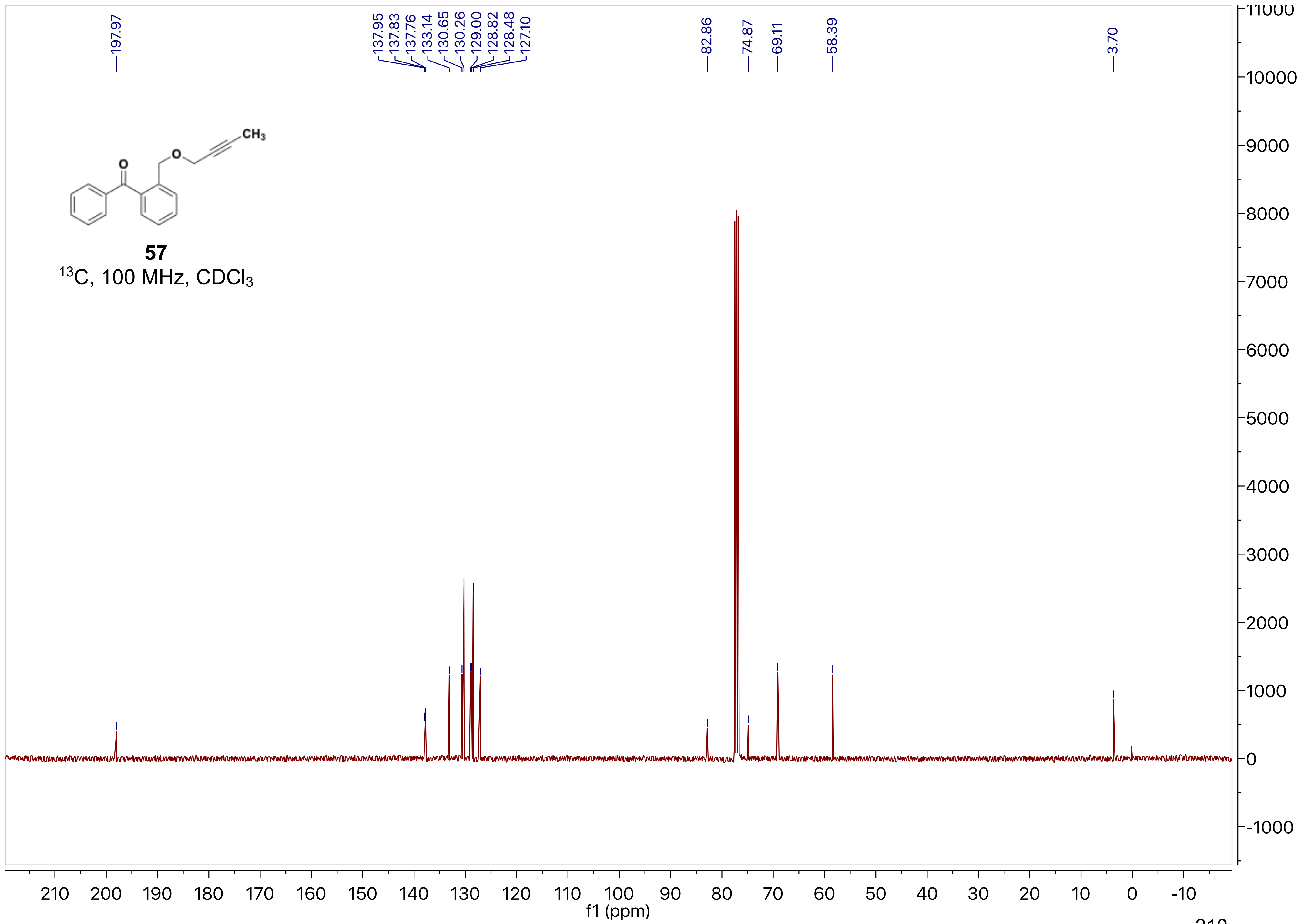


CH3
O
Br
56
1H, 600 MHz, CDCl3
D (t)
7.14
B (d)
7.49
A (d)
7.54
C (t)
7.31
E (s)
4.65
F (s)
4.22
G (s)
1.88
1.00
0.98
1.03
1.01
1.99
2.02
3.01
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5 -2.0
f1 (ppm)

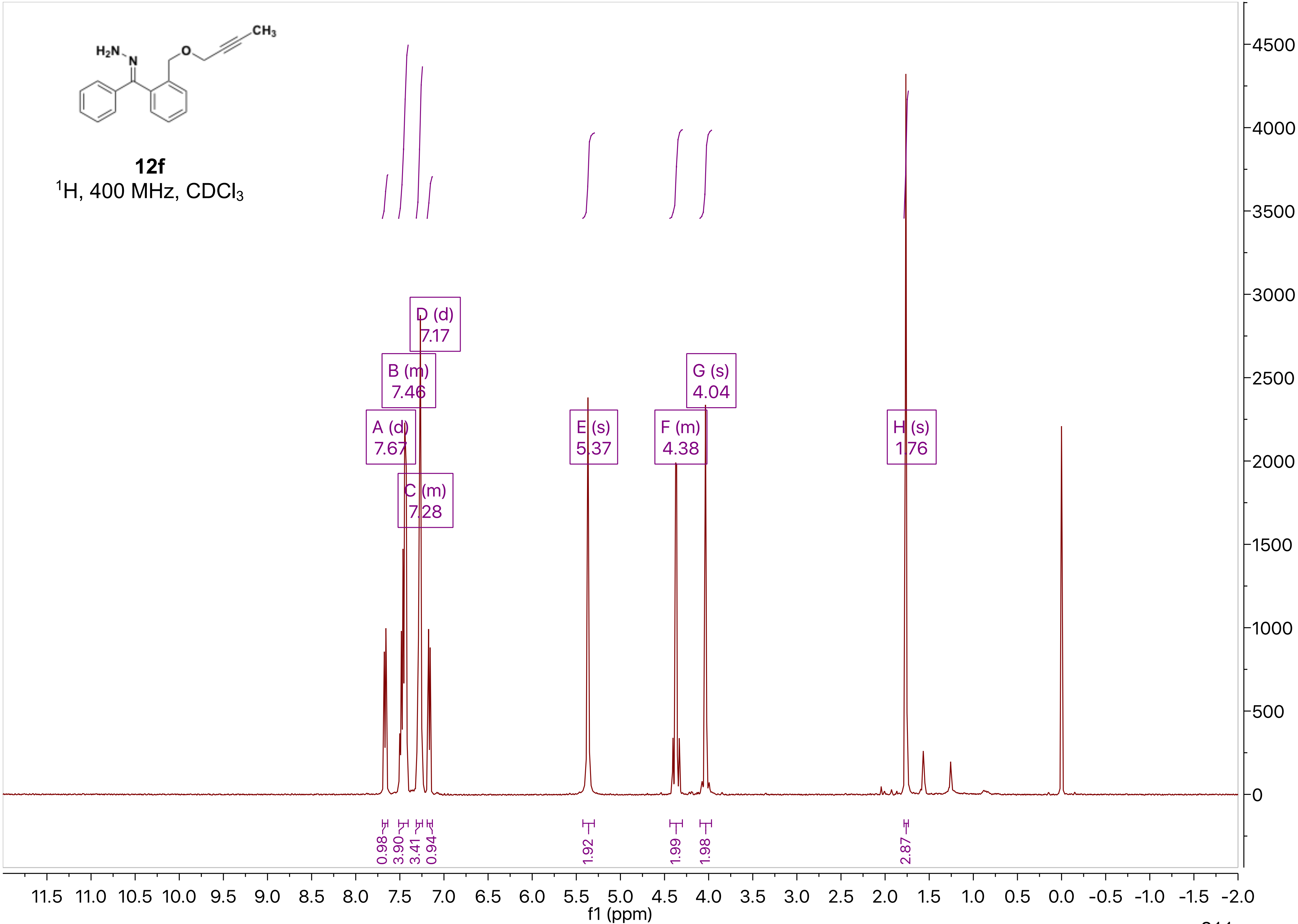

CH3
O
Br
56
13C, 150 MHz, CDCl3
137.28
132.64
129.39
129.13
127.51
122.99
83.11
75.00
71.00
58.58
3.84
55
50
45
40
35
30
25
20
15
10
5
0
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

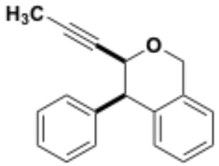


57
1H, 400 MHz, CDCl3
A (d) 7.80
B (m) 7.59
C (m) 7.47
D (m) 7.35
E (s) 4.67
F (s) 4.00
G (s) 1.76
1.93
1.99
3.00
2.02
2.00
2.00
2.96
f1 (ppm)



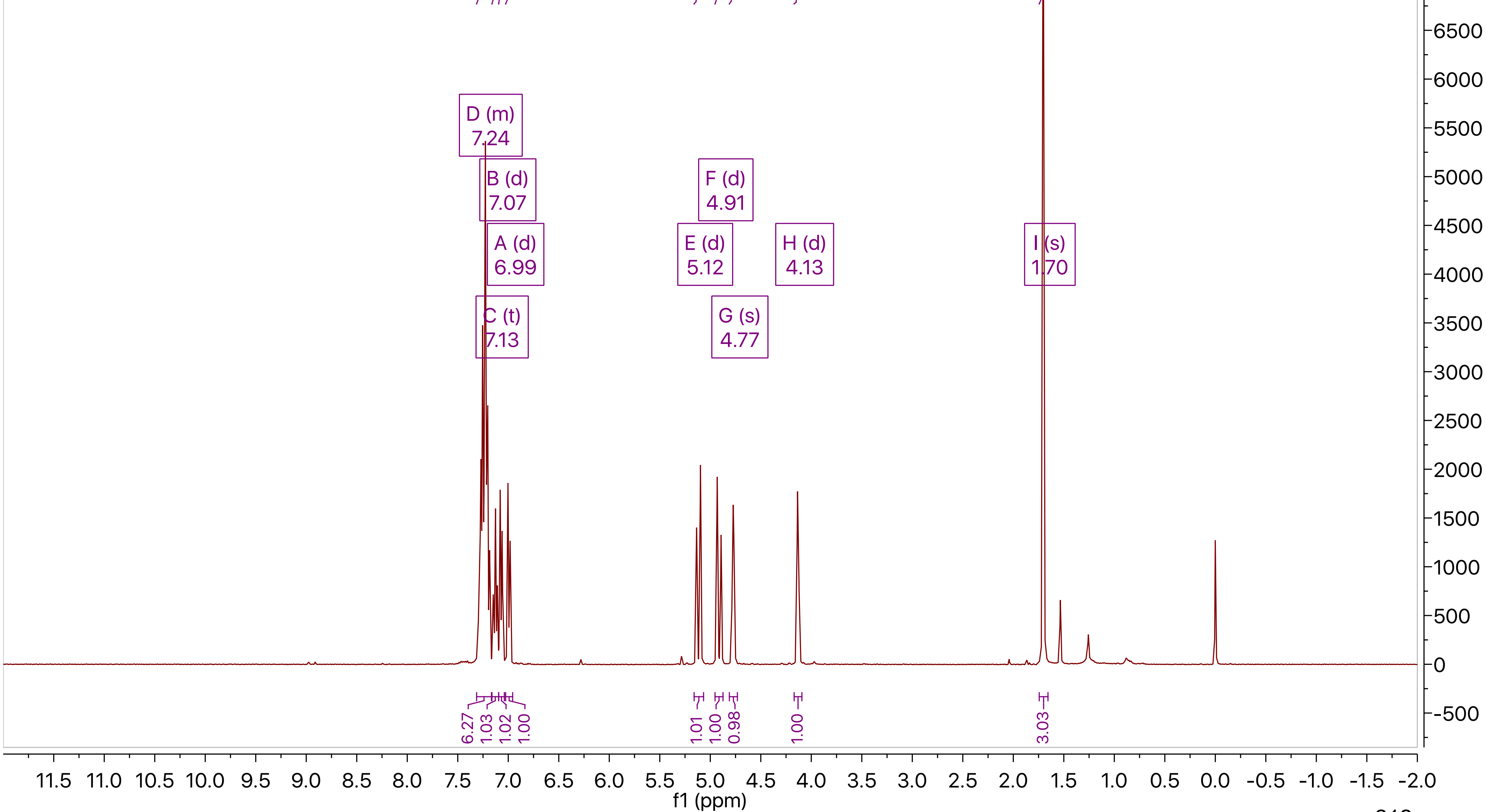
197.97
137.95
137.83
137.76
133.14
130.65
130.26
129.00
128.82
128.48
127.10
82.86
74.87
69.11
58.39
3.70
CH3
O
O
57
13C, 100 MHz, CDCl3
f1 (ppm)

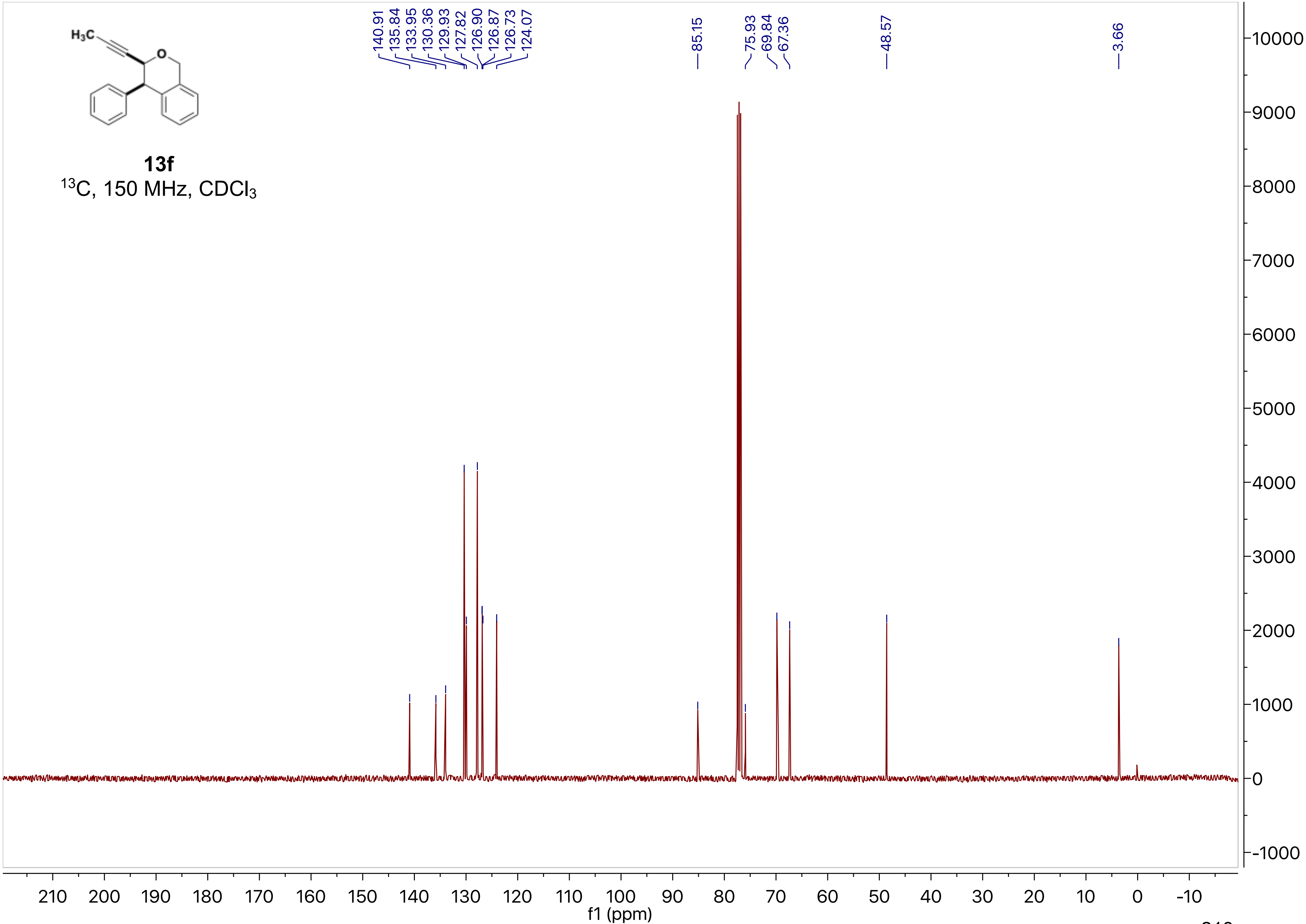

12f
^{1}H, 400 MHz, $CDCl_3$
A (d) 7.67
B (m) 7.46
C (m) 7.28
D (d) 7.17
E (s) 5.37
F (m) 4.38
G (s) 4.04
H (s) 1.76
0.98
3.90
3.41
0.94
1.92
1.99
1.98
2.87
f1 (ppm)

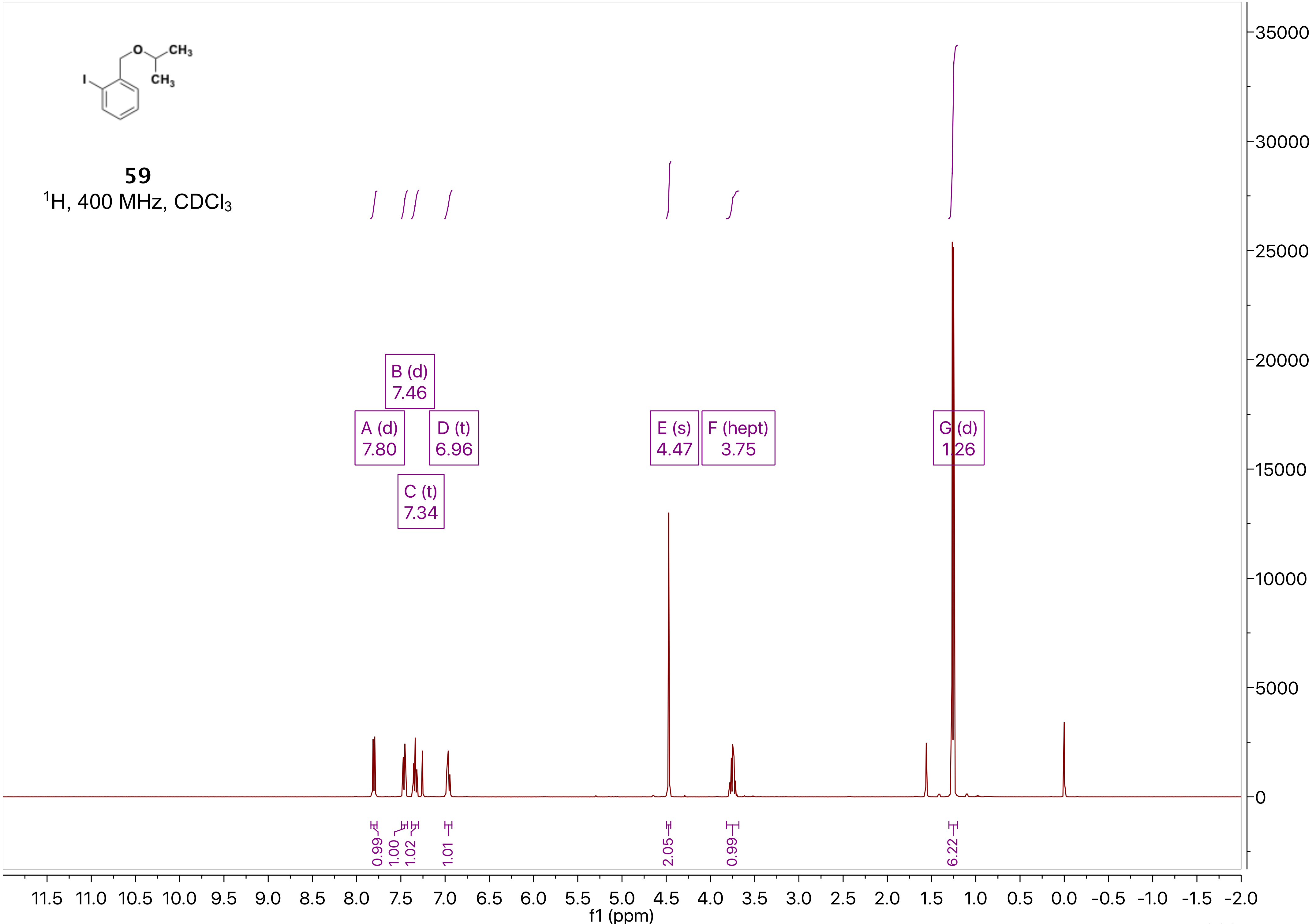


13f

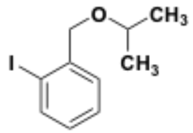
^{1}H, 600 MHz, $CDCl_3$




H3C
O
13f
13C, 150 MHz, CDCl3
140.91
135.84
133.95
130.36
129.93
127.82
126.90
126.87
126.73
124.07
85.15
75.93
69.84
67.36
48.57
3.66
f1 (ppm)

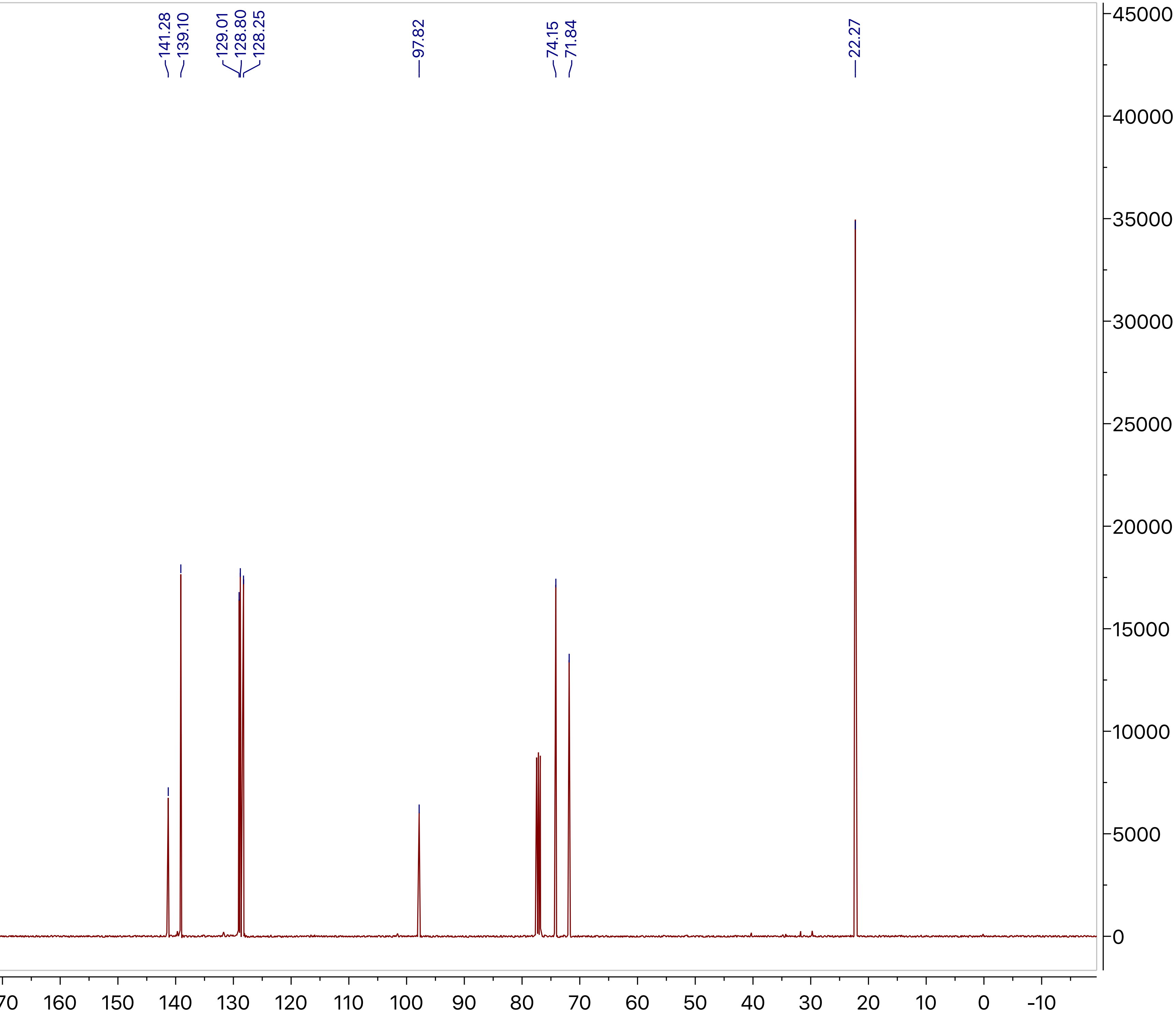


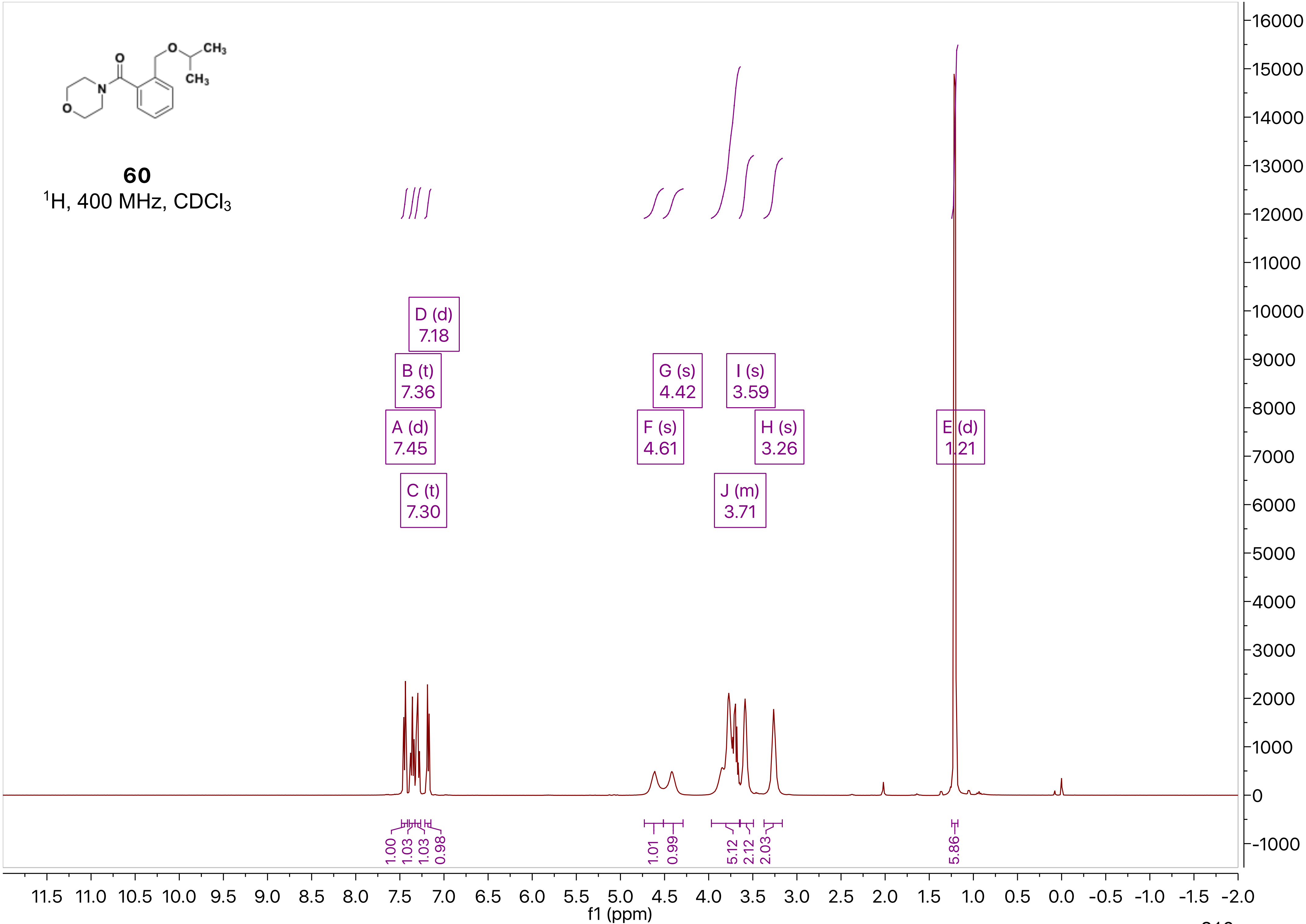
59
1H, 400 MHz, CDCl3
A (d)
7.80
B (d)
7.46
C (t)
7.34
D (t)
6.96
E (s)
4.47
F (hept)
3.75
G (d)
1.26
0.99
1.00
1.02
1.01
2.05
0.99
6.22
f1 (ppm)

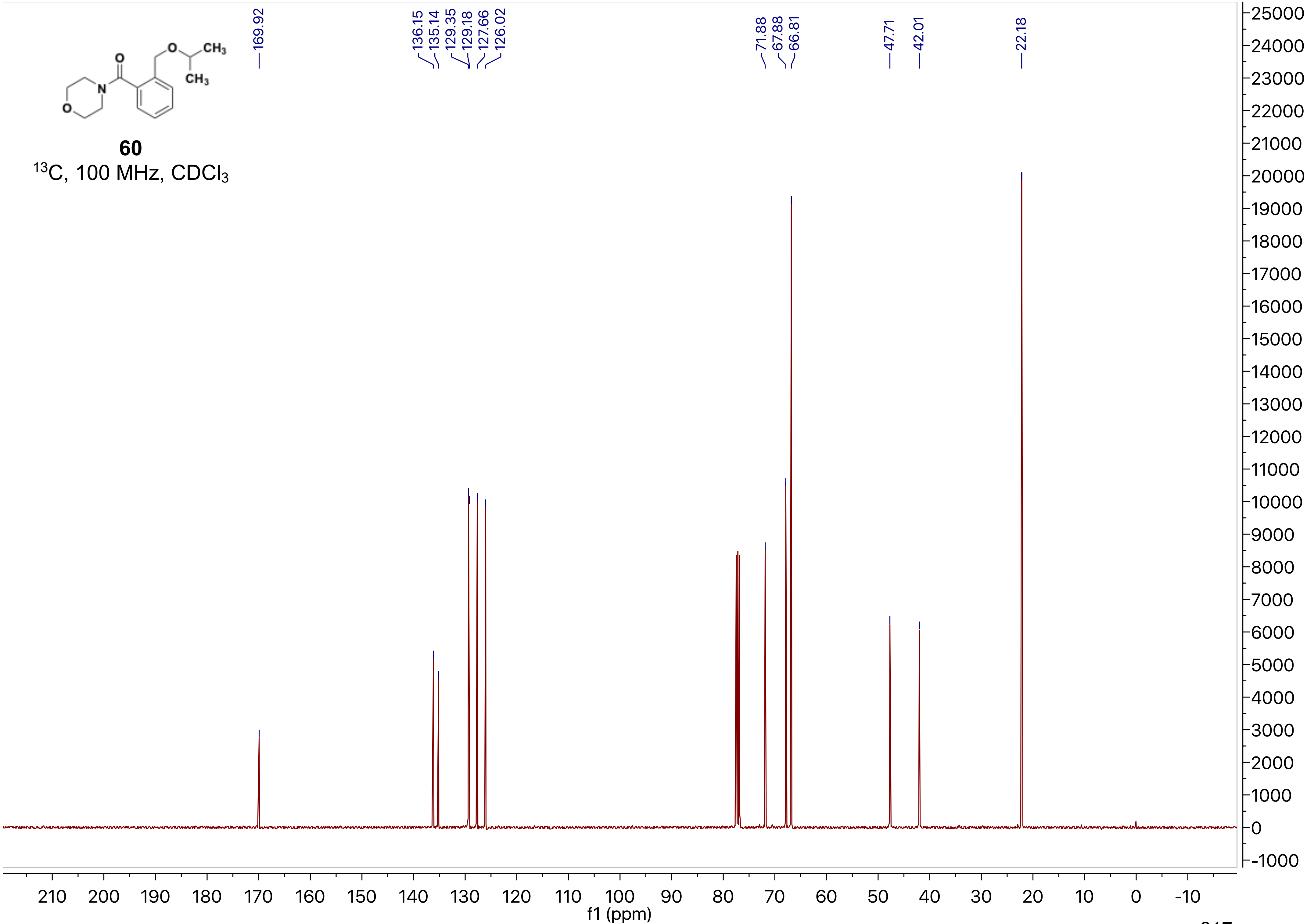


59

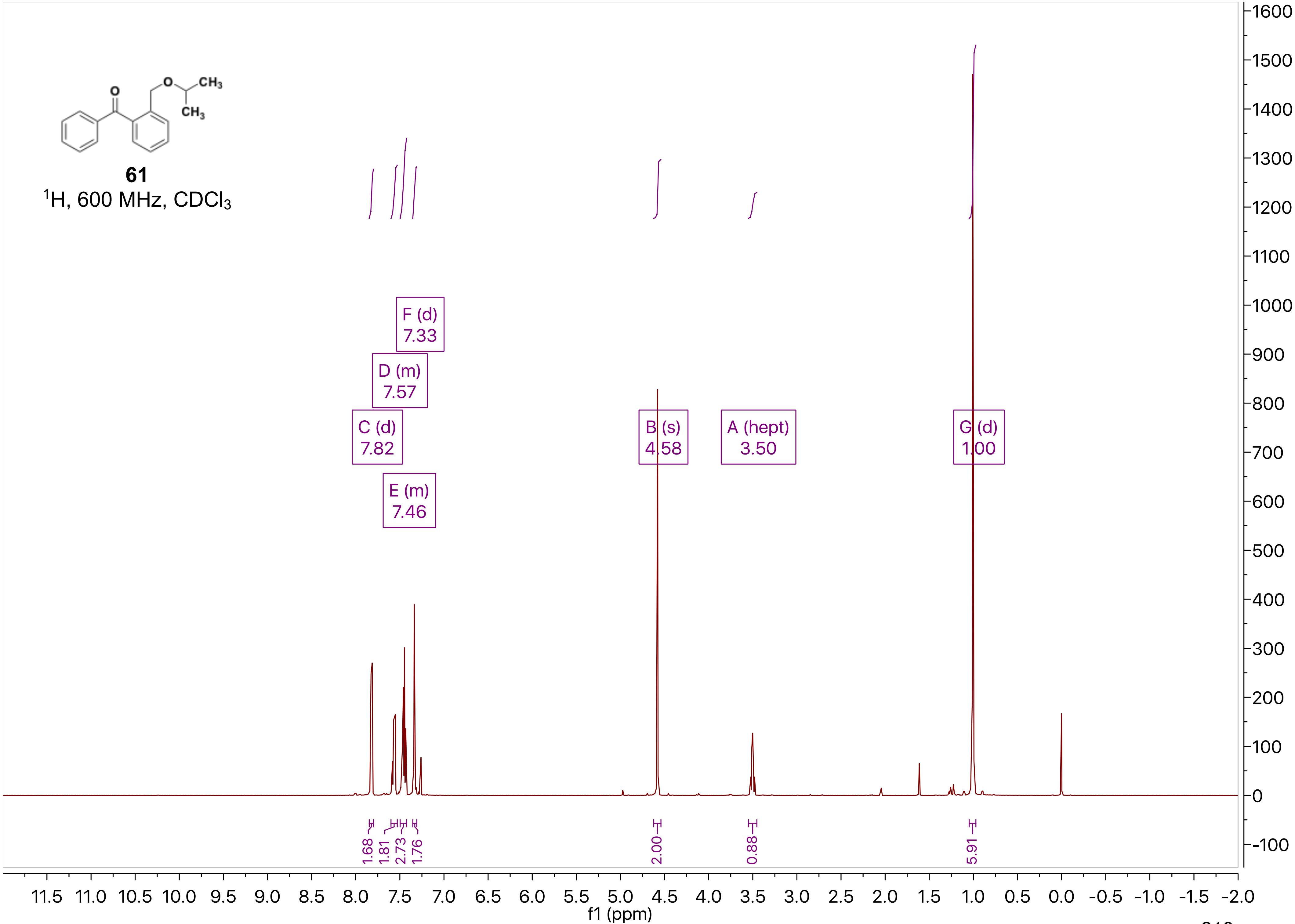
^{13}C, 100 MHz, $CDCl_3$

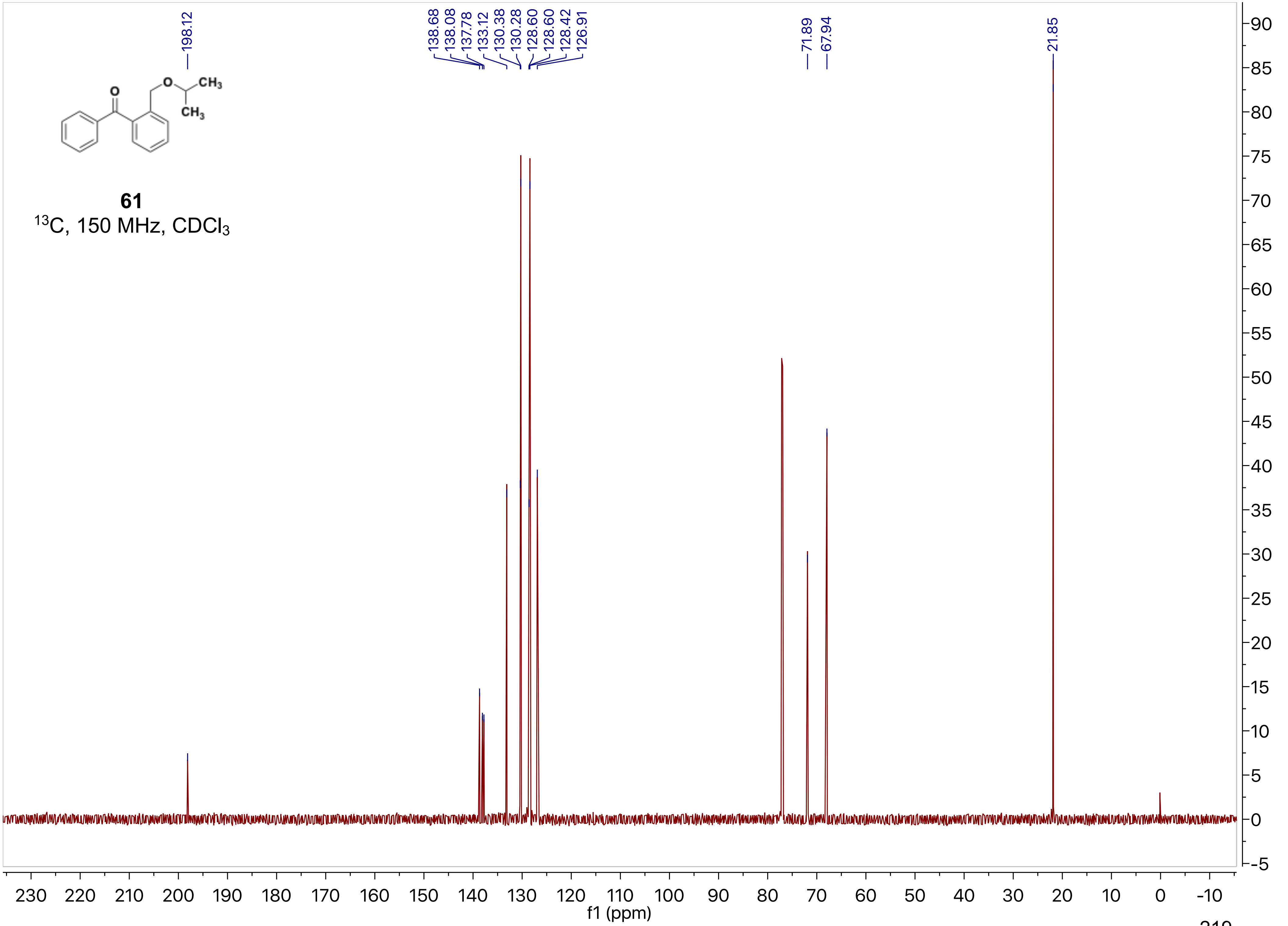



60
1H, 400 MHz, CDCl3
D (d) 7.18
B (t) 7.36
A (d) 7.45
C (t) 7.30
G (s) 4.42
F (s) 4.61
I (s) 3.59
H (s) 3.26
J (m) 3.71
E (d) 1.21
1.00
1.03
1.03
0.98
1.01
0.99
5.12
2.12
2.03
5.86
f1 (ppm)



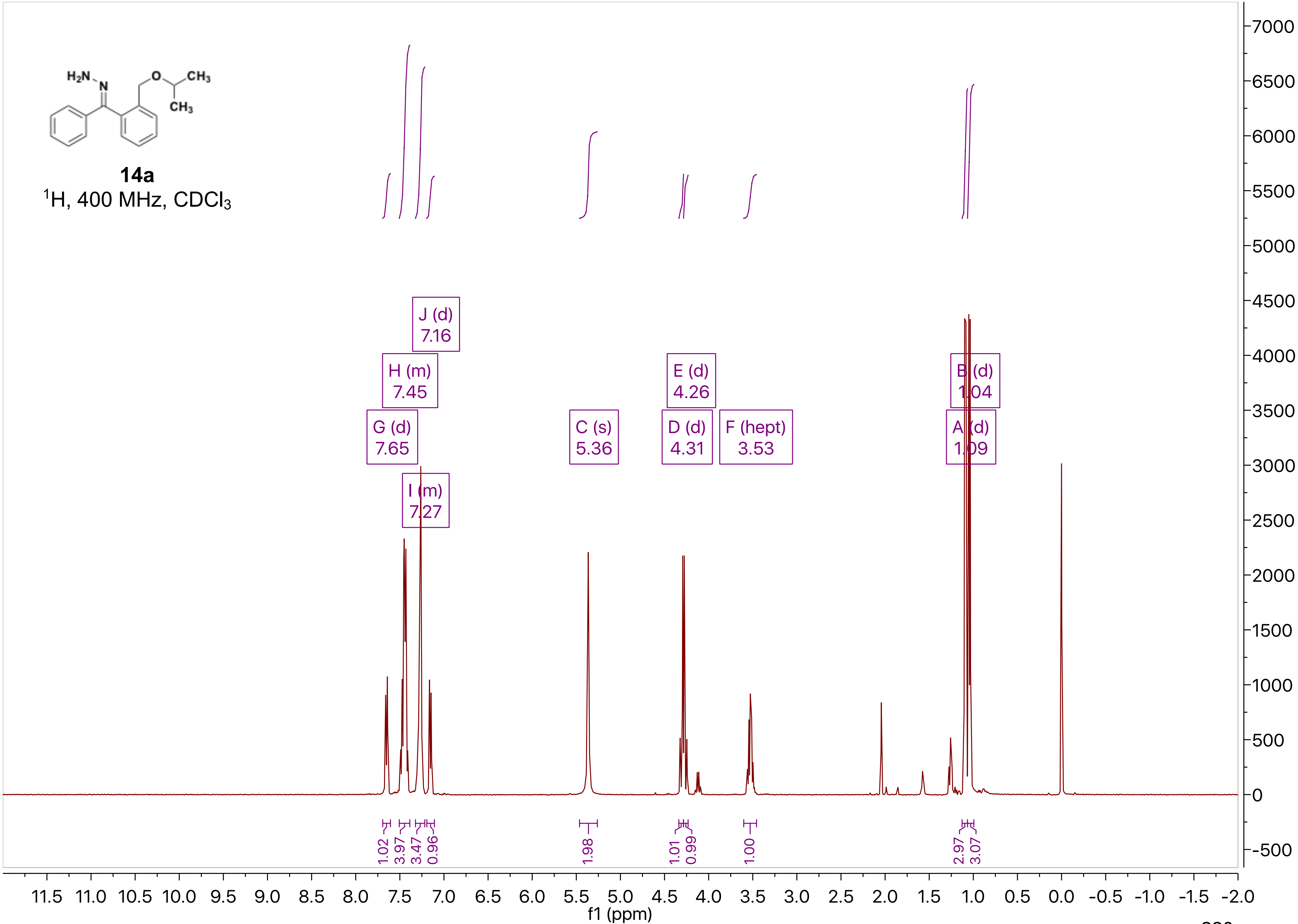
60
^{13}C, 100 MHz, $CDCl_3$
169.92
136.15
135.14
129.35
129.18
127.66
126.02
71.88
67.88
66.81
47.71
42.01
22.18
f1 (ppm)


61
¹H, 600 MHz, CDCl₃
F (d) 7.33
D (m) 7.57
C (d) 7.82
E (m) 7.46
B (s) 4.58
A (hept) 3.50
G (d) 1.00
1.68
1.81
2.73
1.76
2.00
0.88
5.91
f1 (ppm)

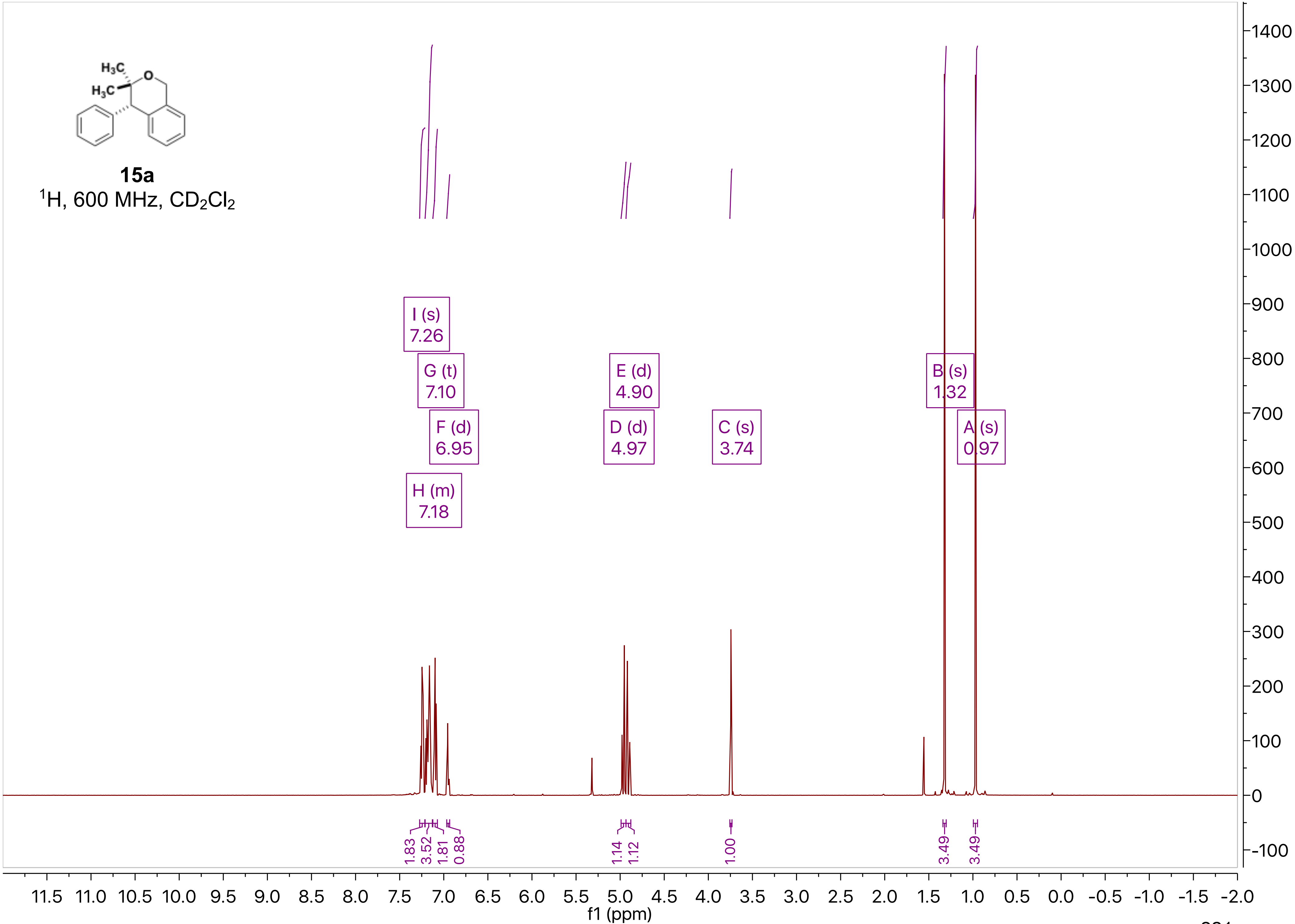


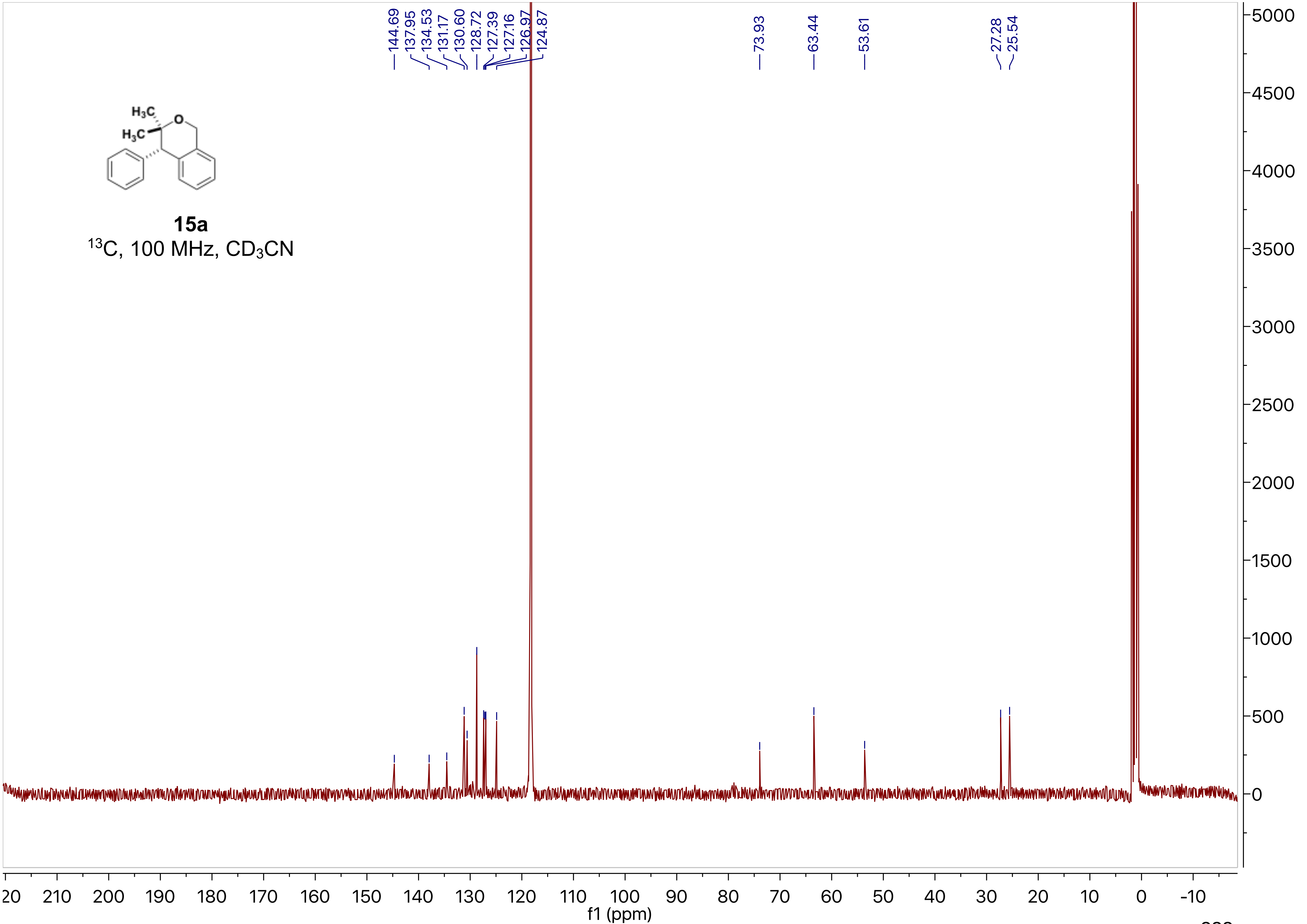
61

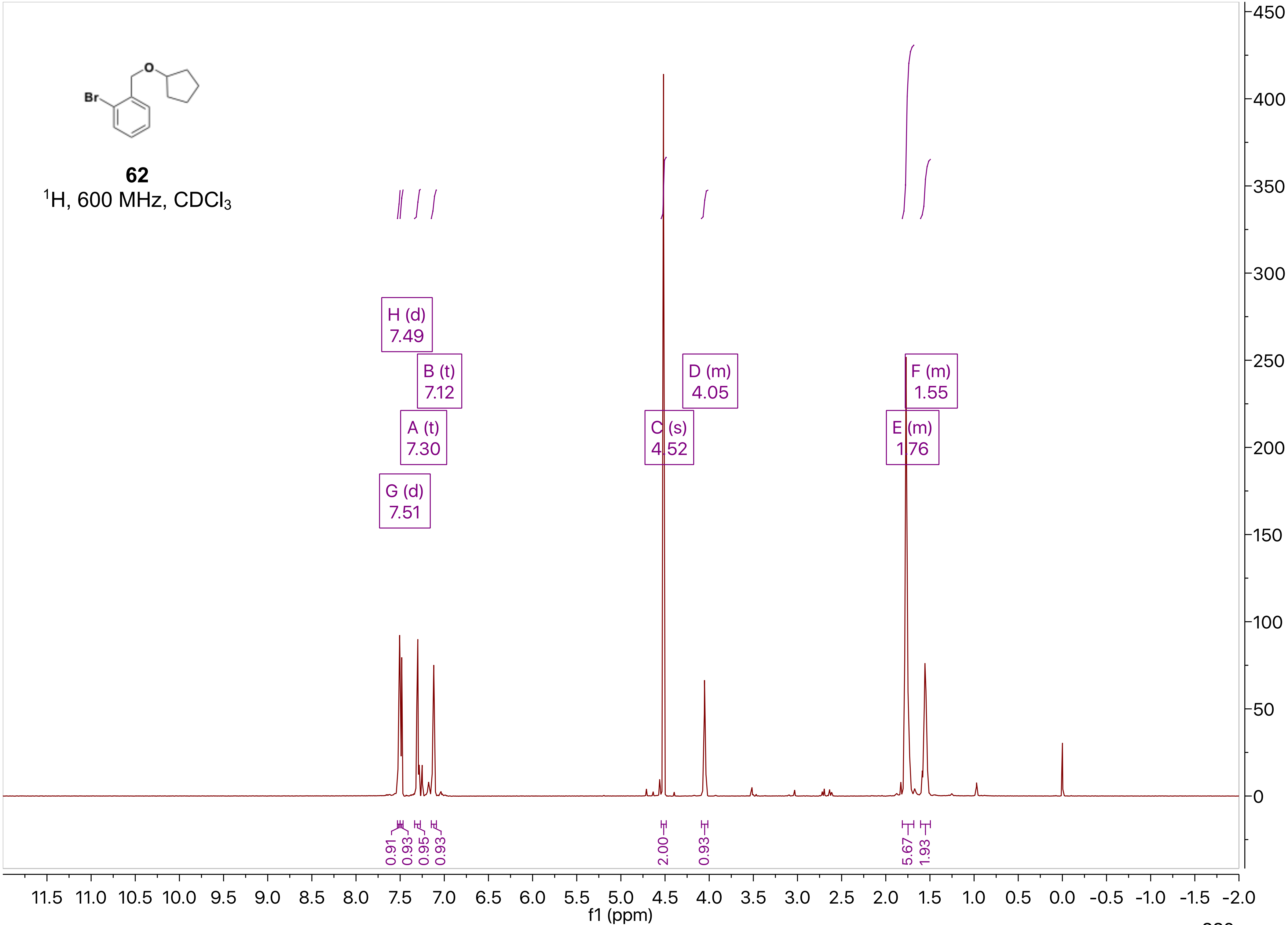
^{13}C, 150 MHz, $CDCl_3$



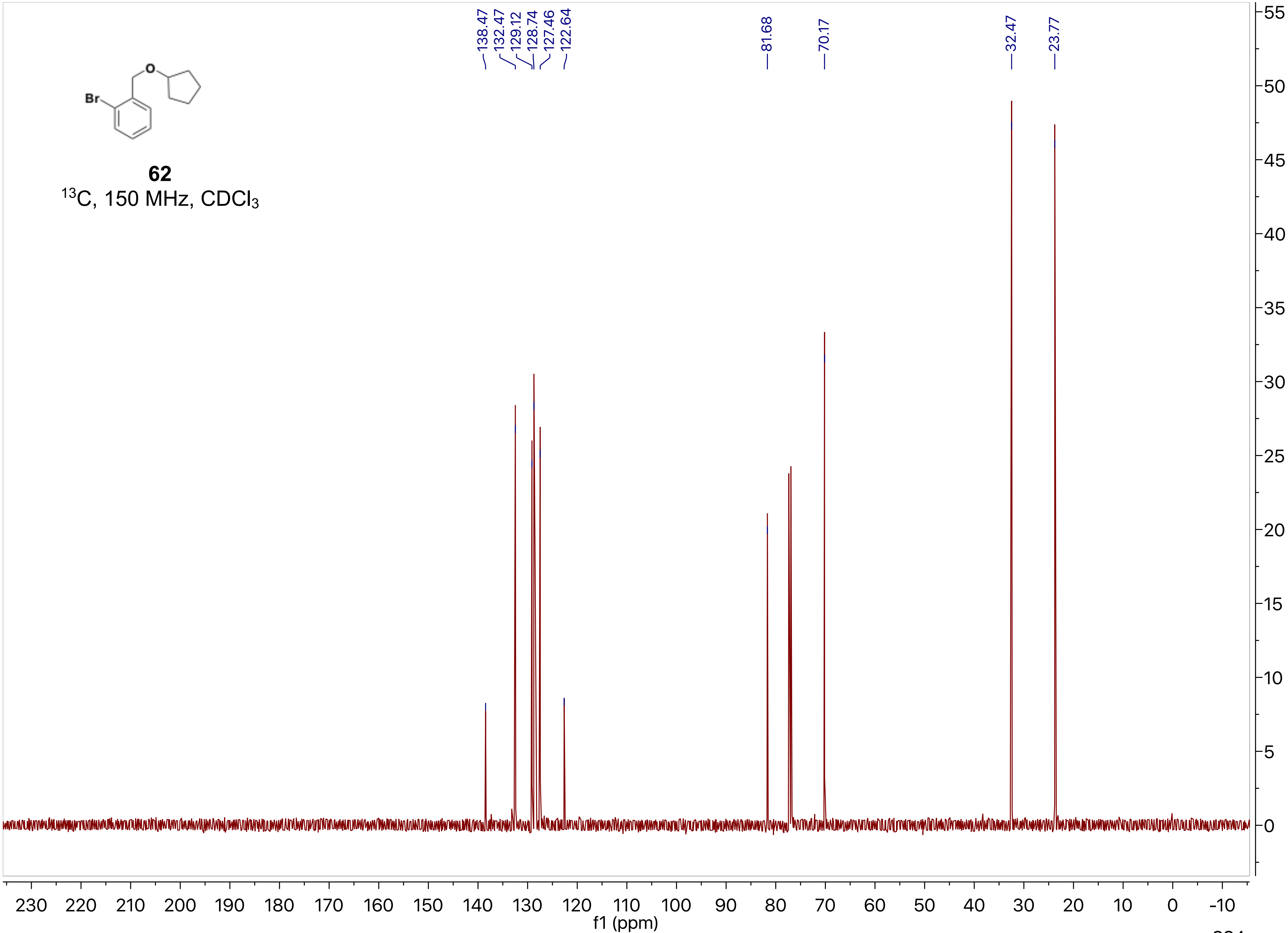
H2N N O CH3 CH3
14a
1H, 400 MHz, CDCl3
J (d) 7.16
H (m) 7.45
G (d) 7.65
I (m) 7.27
C (s) 5.36
E (d) 4.26
D (d) 4.31
F (hept) 3.53
B (d) 1.04
A (d) 1.09
1.02 3.97 3.47 0.96
1.98
1.01 0.99
1.00
2.97 3.07
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5 -2.0
f1 (ppm)
7000 6500 6000 5500 5000 4500 4000 3500 3000 2500 2000 1500 1000 500 0 -500


15a
¹H, 600 MHz, CD2Cl2
I (s)
7.26
G (t)
7.10
F (d)
6.95
H (m)
7.18
E (d)
4.90
D (d)
4.97
C (s)
3.74
B (s)
1.32
A (s)
0.97
1.83
3.52
1.81
0.88
1.14
1.12
1.00
3.49
3.49
f1 (ppm)

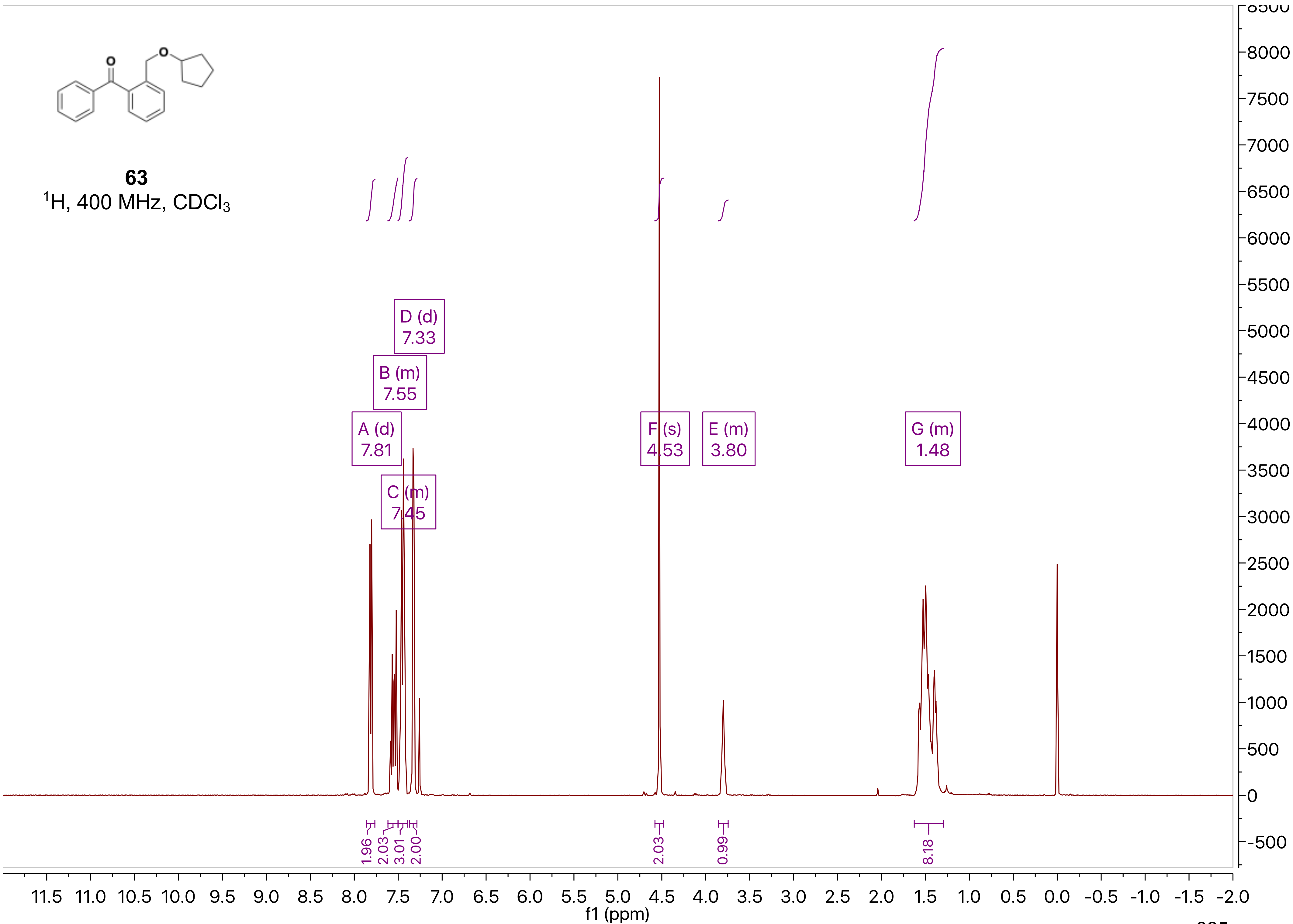

144.69
137.95
134.53
131.17
130.60
128.72
127.39
127.16
126.97
124.87
73.93
63.44
53.61
27.28
25.54
15a
13C, 100 MHz, CD3CN
f1 (ppm)

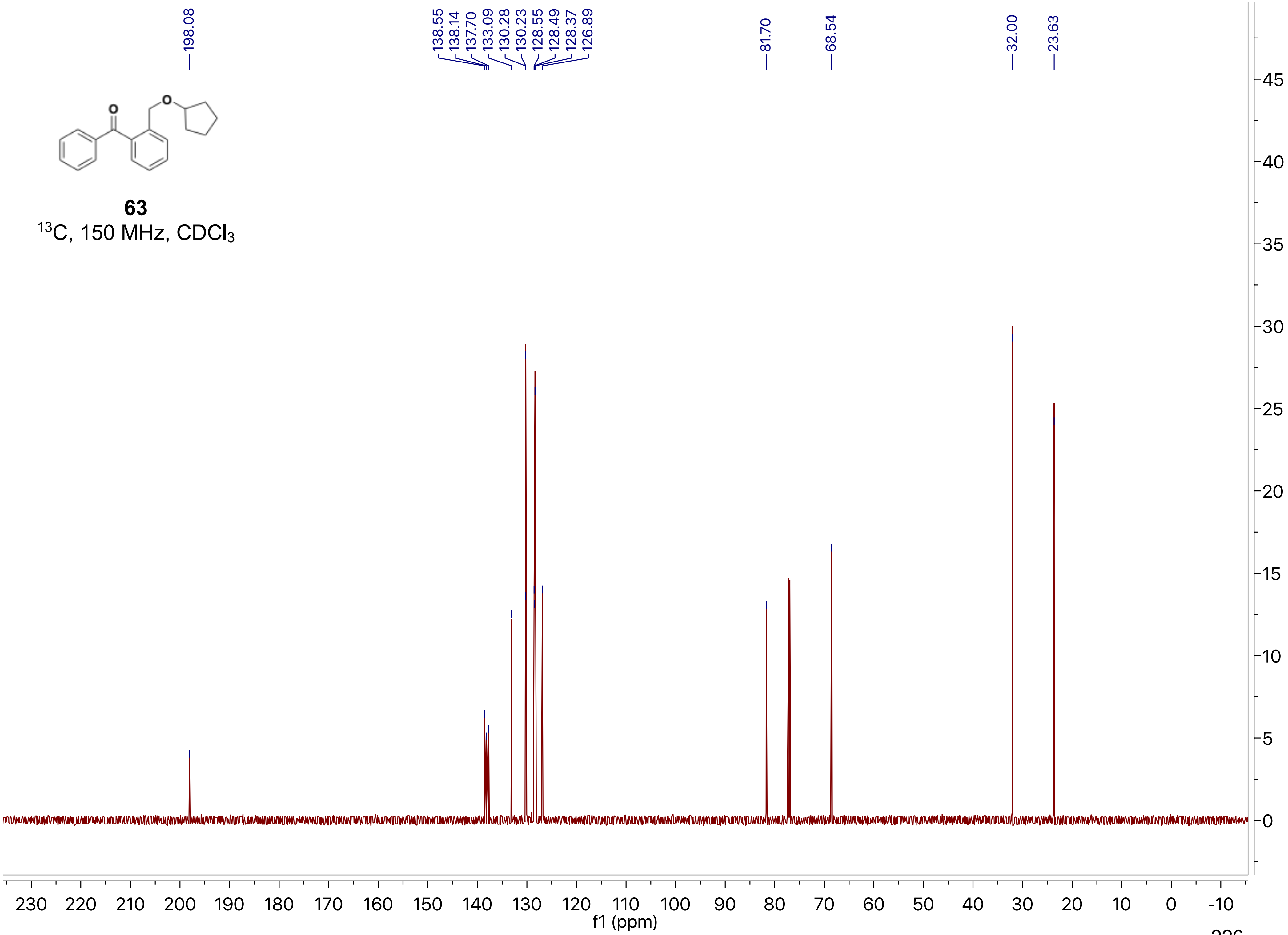


Br
O
62
^{1}H, 600 MHz, $CDCl_3$
H (d) 7.49
B (t) 7.12
A (t) 7.30
G (d) 7.51
C (s) 4.52
D (m) 4.05
E (m) 1.76
F (m) 1.55
0.91
0.93
0.95
0.93
2.00
0.93
5.67
1.93
f1 (ppm)



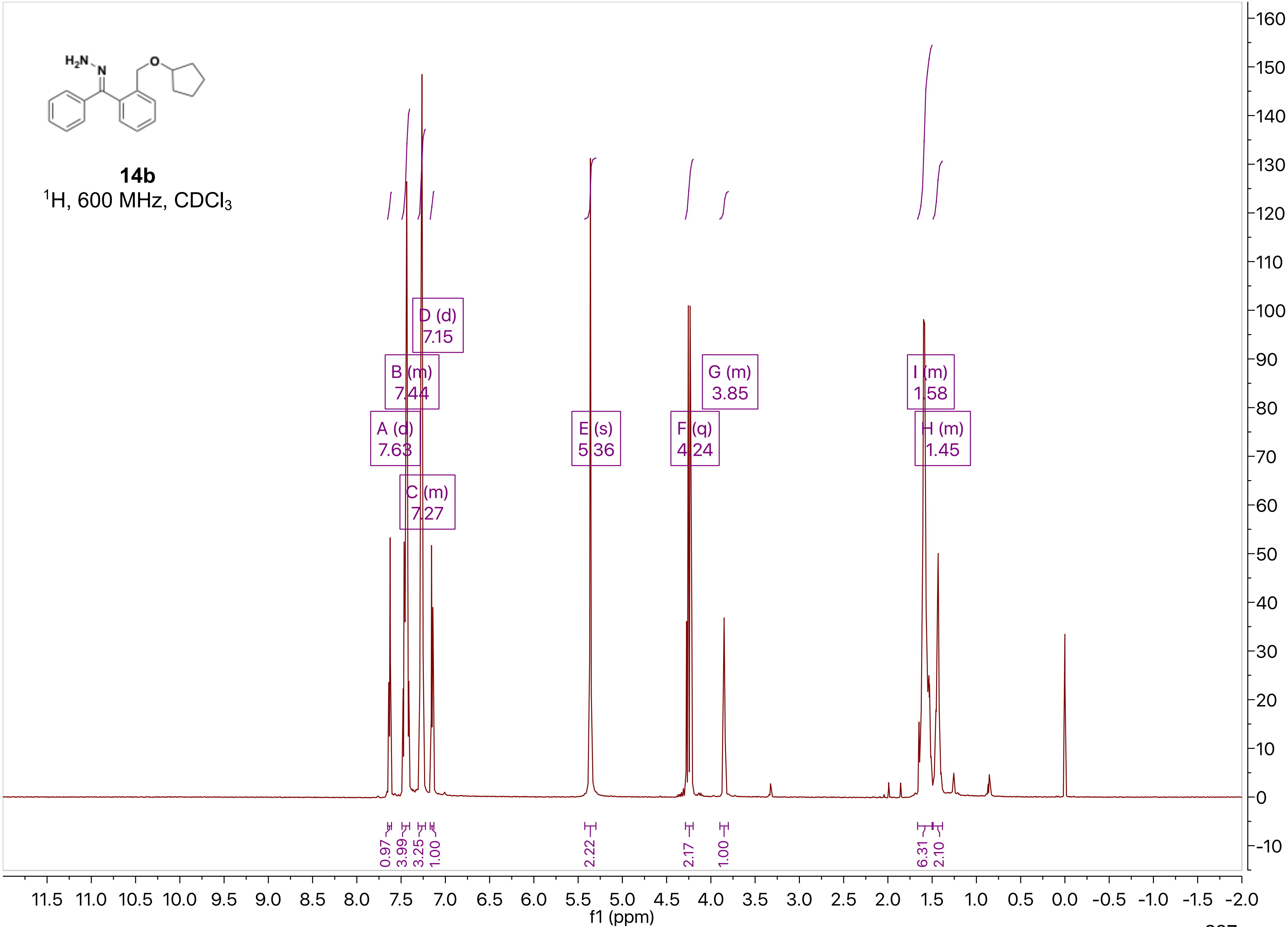
Br
O
62
13C, 150 MHz, CDCl3
138.47
132.47
129.12
128.74
127.46
122.64
81.68
70.17
32.47
23.77
f1 (ppm)


63
1H, 400 MHz, CDCl3
D (d) 7.33
B (m) 7.55
A (d) 7.81
C (m) 7.45
F (s) 4.53
E (m) 3.80
G (m) 1.48
1.96
2.03
3.01
2.00
2.03
0.99
8.18
f1 (ppm)

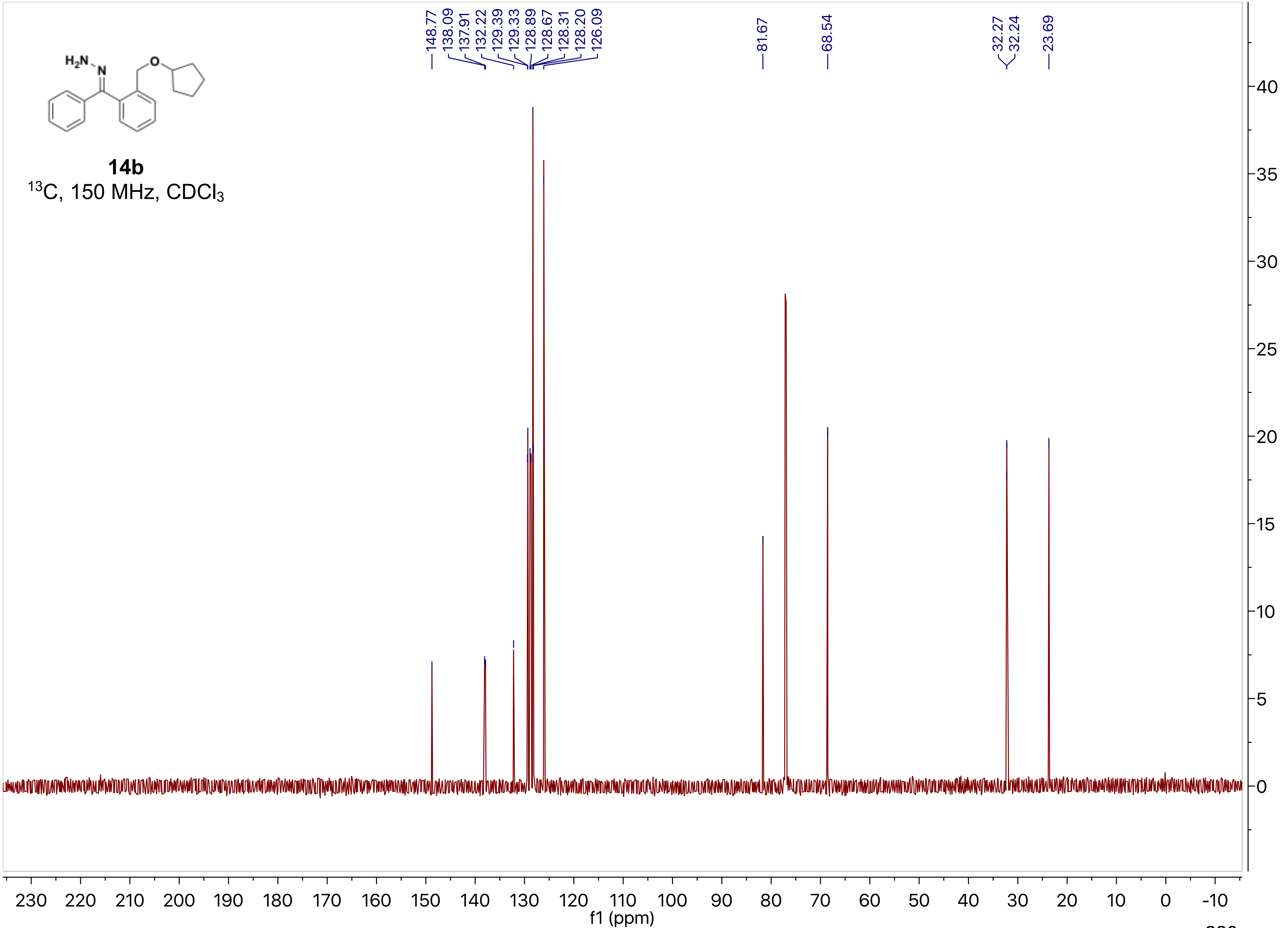


63

^{13}C, 150 MHz, $CDCl_3$

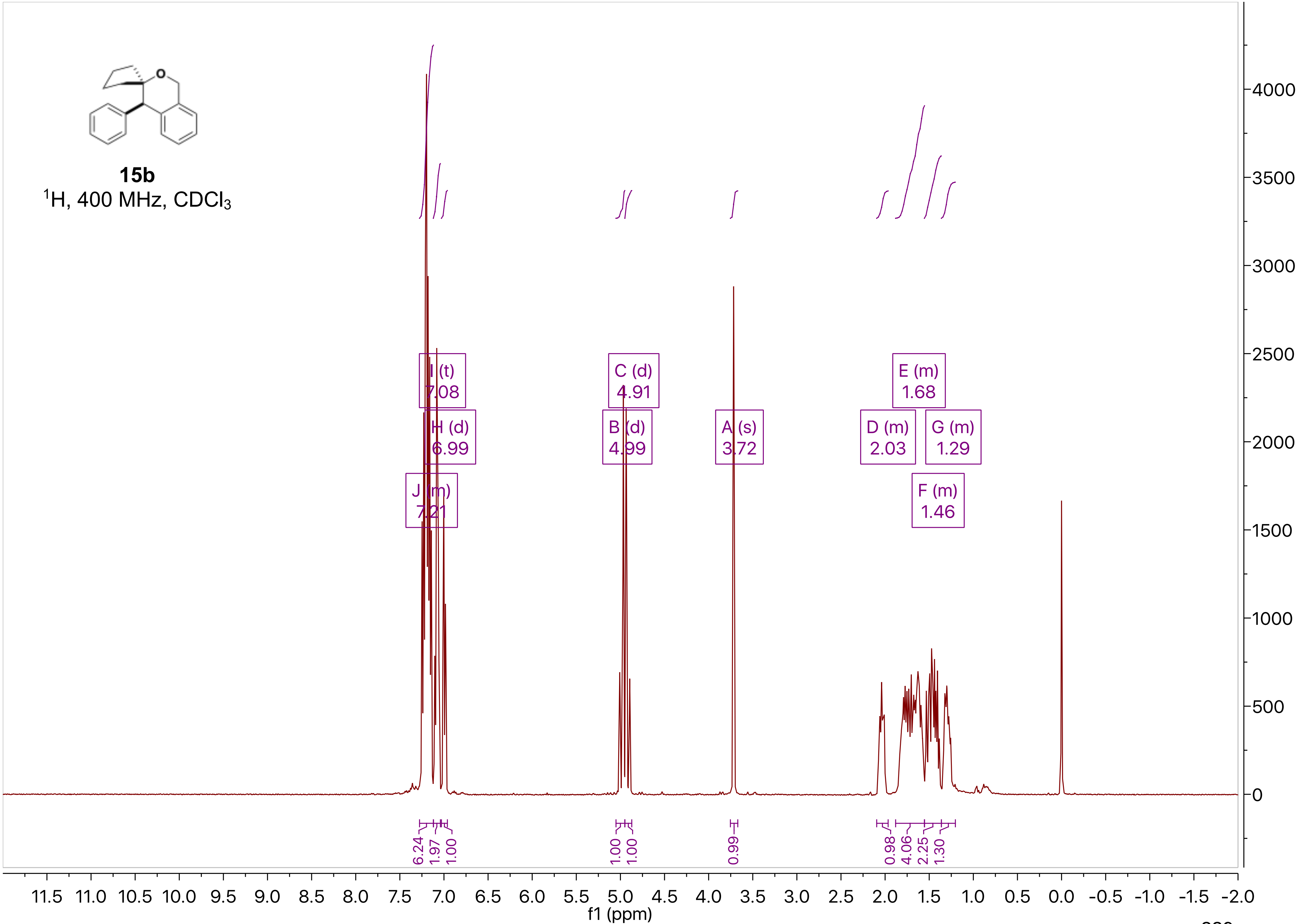


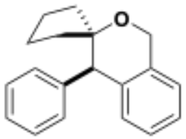
14b
¹H, 600 MHz, CDCl₃
A (d) 7.63
B (m) 7.44
C (m) 7.27
D (d) 7.15
E (s) 5.36
F (q) 4.24
G (m) 3.85
I (m) 1.58
H (m) 1.45
0.97
3.99
3.25
1.00
2.22
2.17
1.00
6.31
2.10
f1 (ppm)



14b

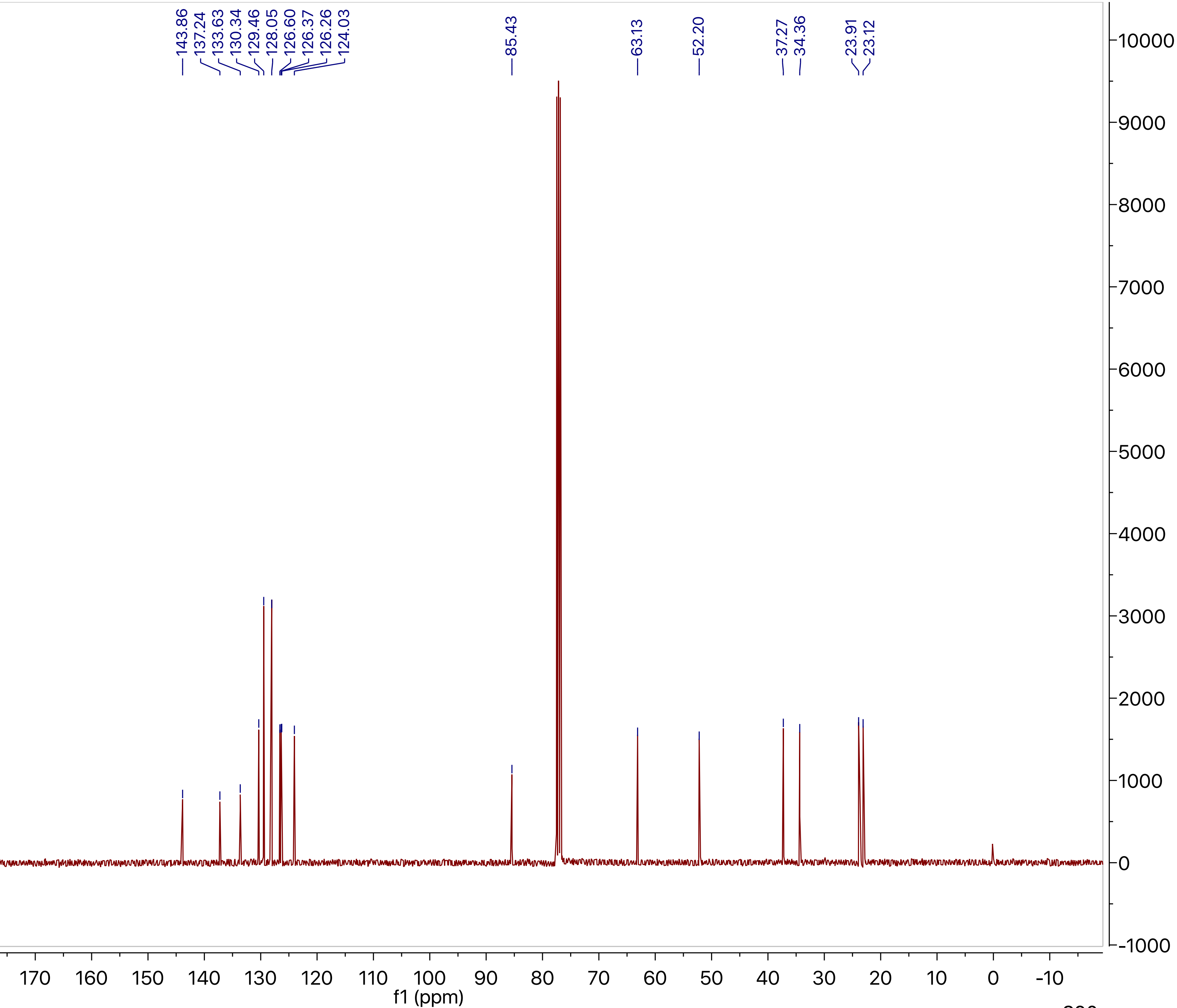
^{13}C, 150 MHz, $CDCl_3$

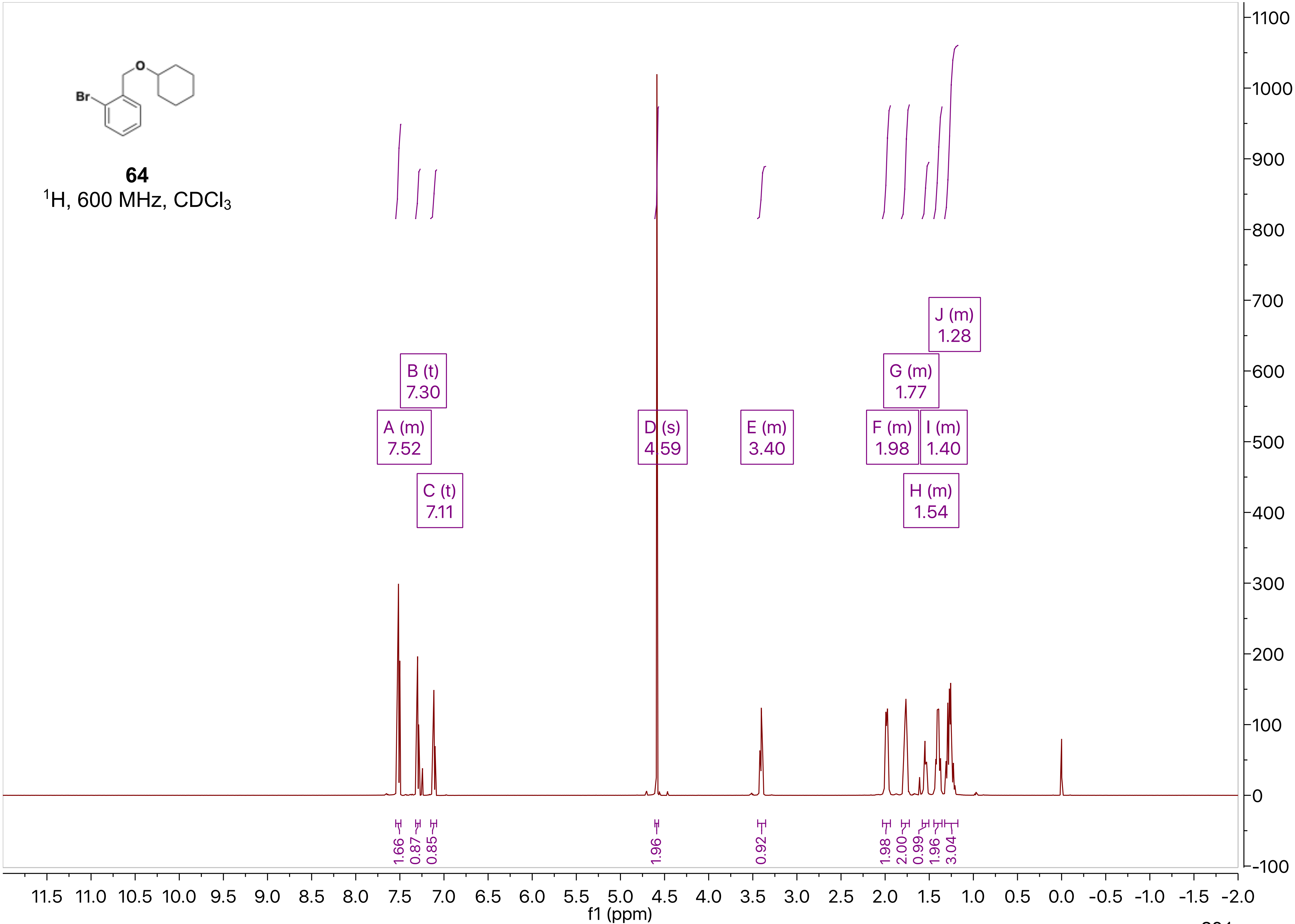

15b
1H, 400 MHz, CDCl3
I (t)
7.08
H (d)
6.99
J (m)
7.21
C (d)
4.91
B (d)
4.99
A (s)
3.72
E (m)
1.68
D (m)
2.03
G (m)
1.29
F (m)
1.46
6.24
1.97
1.00
1.00
1.00
0.99
0.98
4.06
2.25
1.30
f1 (ppm)

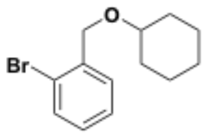


15b

^{13}C, 100 MHz, $CDCl_3$

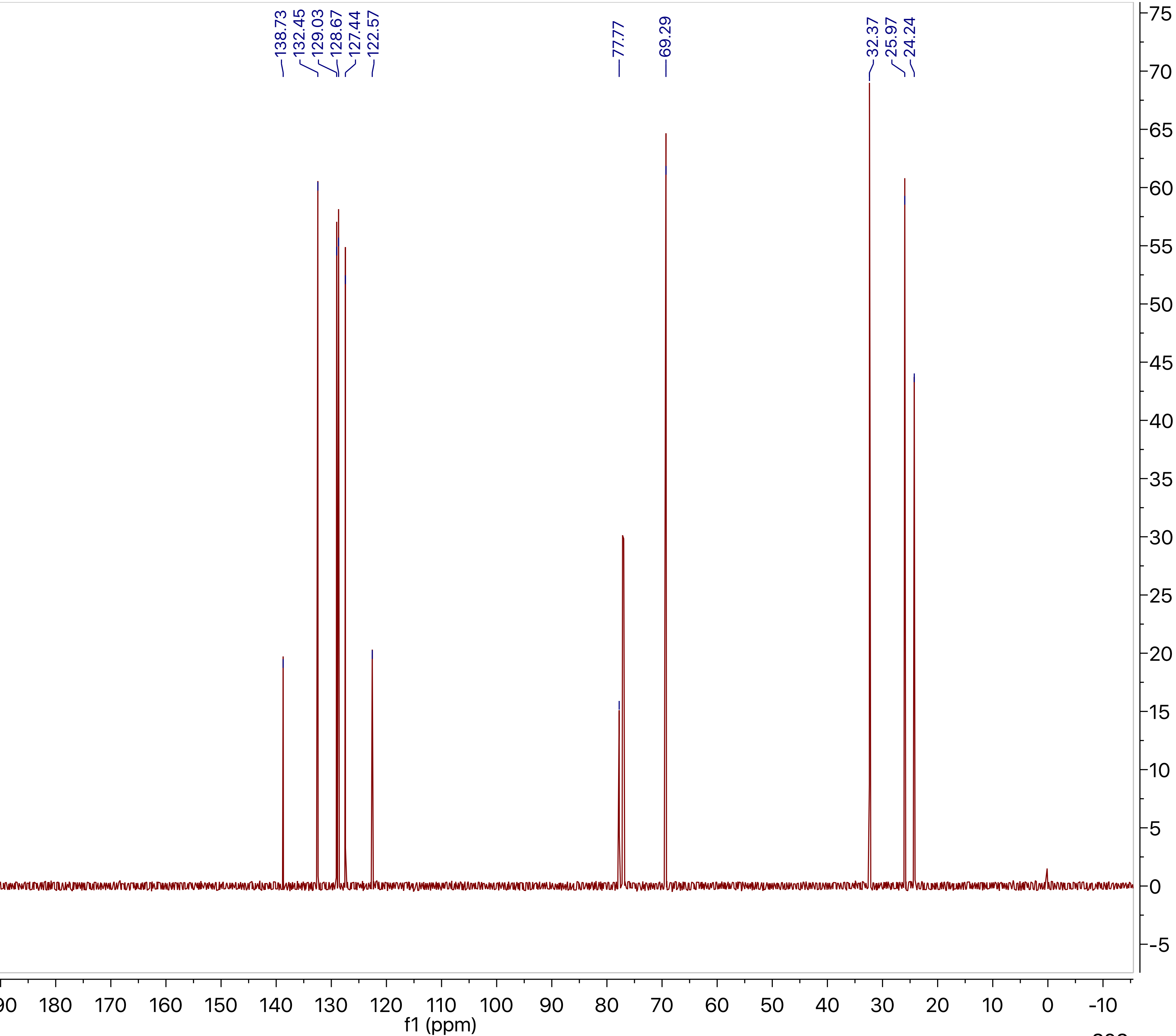


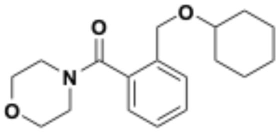

Br
O
64
^{1}H, 600 MHz, $CDCl_3$
A (m) 7.52
B (t) 7.30
C (t) 7.11
D (s) 4.59
E (m) 3.40
F (m) 1.98
G (m) 1.77
H (m) 1.54
I (m) 1.40
J (m) 1.28
1.66
0.87
0.85
1.96
0.92
1.98
2.00
0.99
1.96
3.04
f1 (ppm)



64

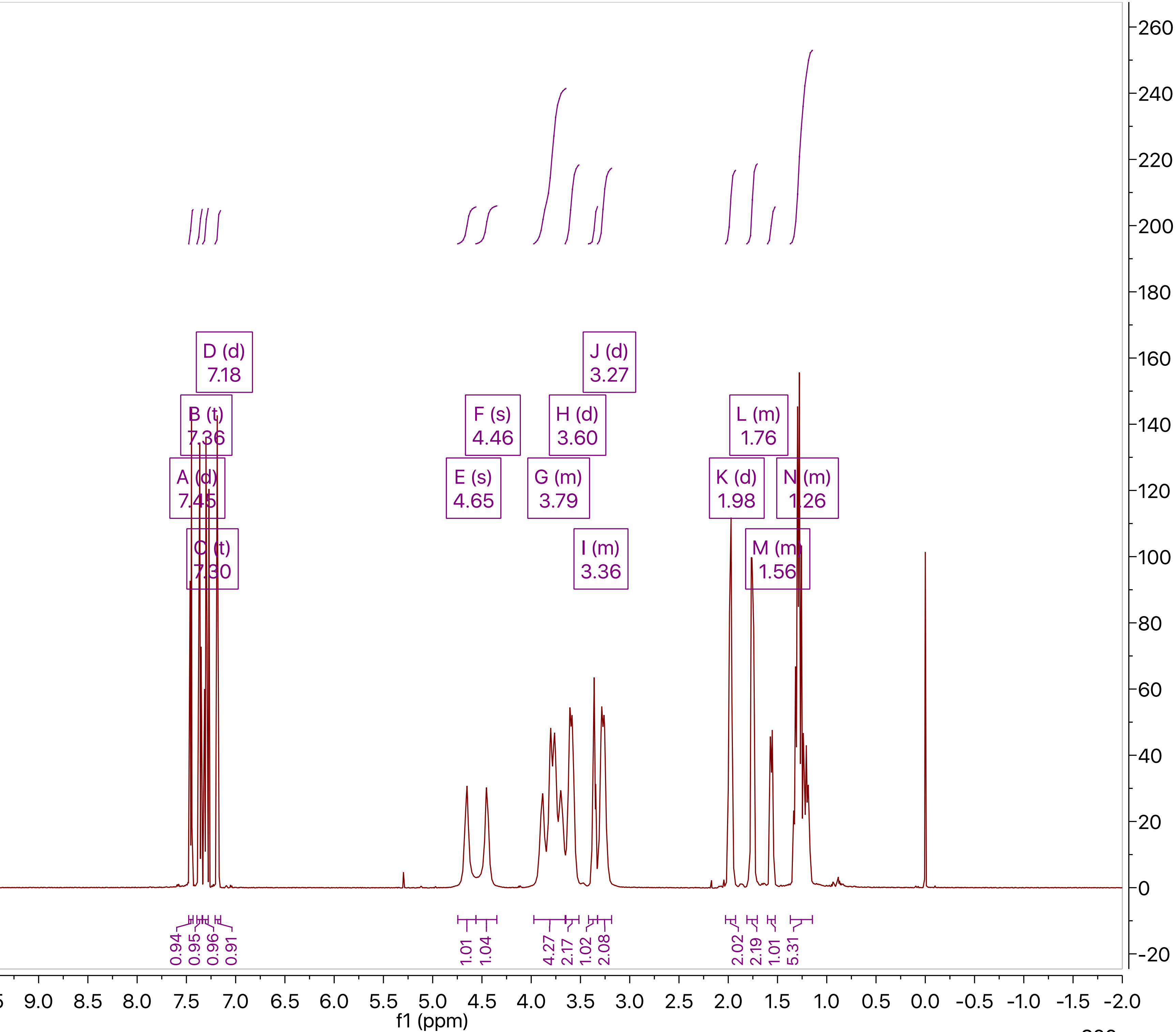
^{13}C, 150 MHz, $CDCl_3$

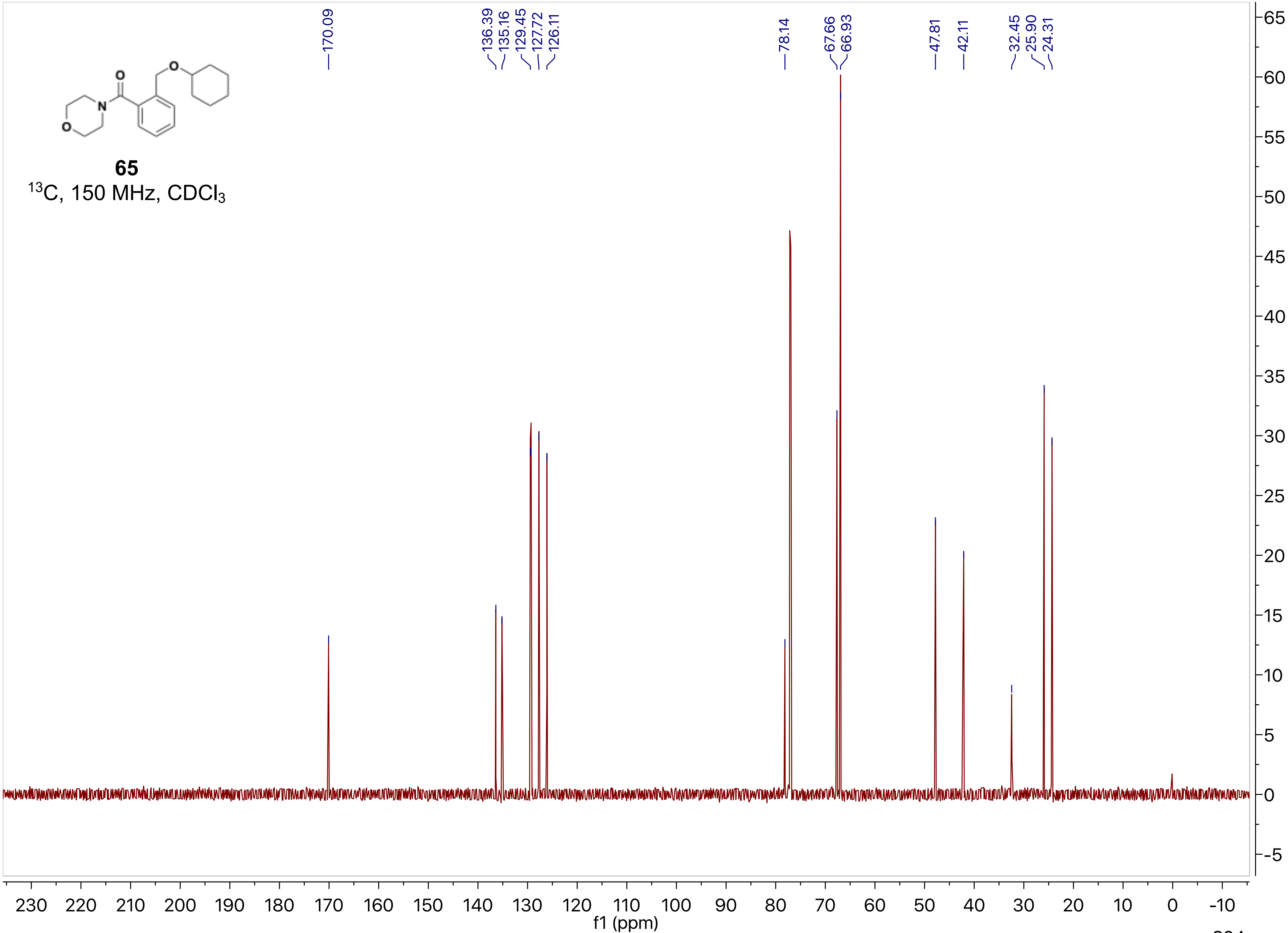


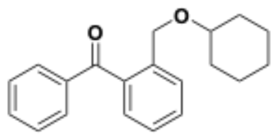


65

^{1}H, 600 MHz, $CDCl_3$

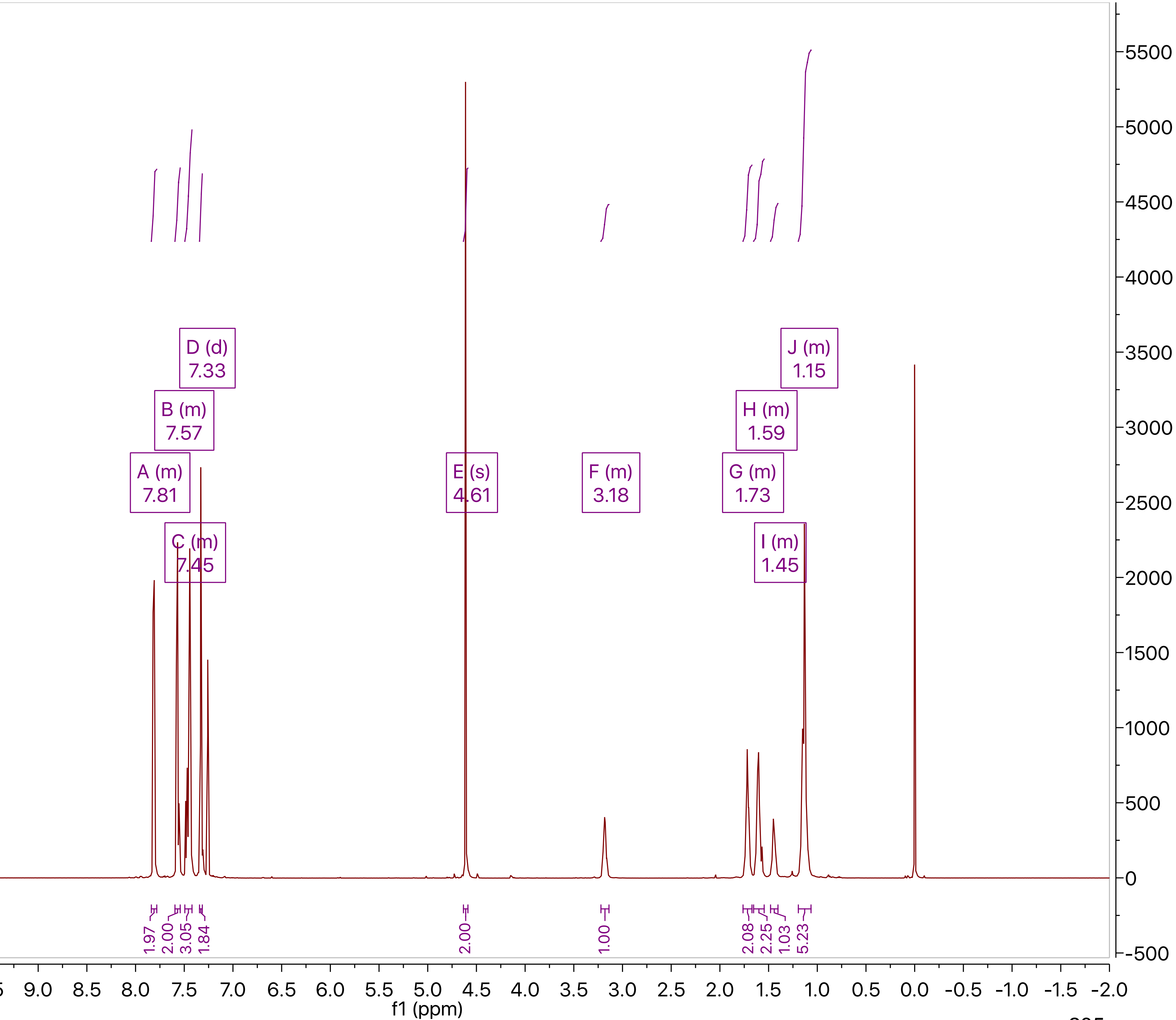


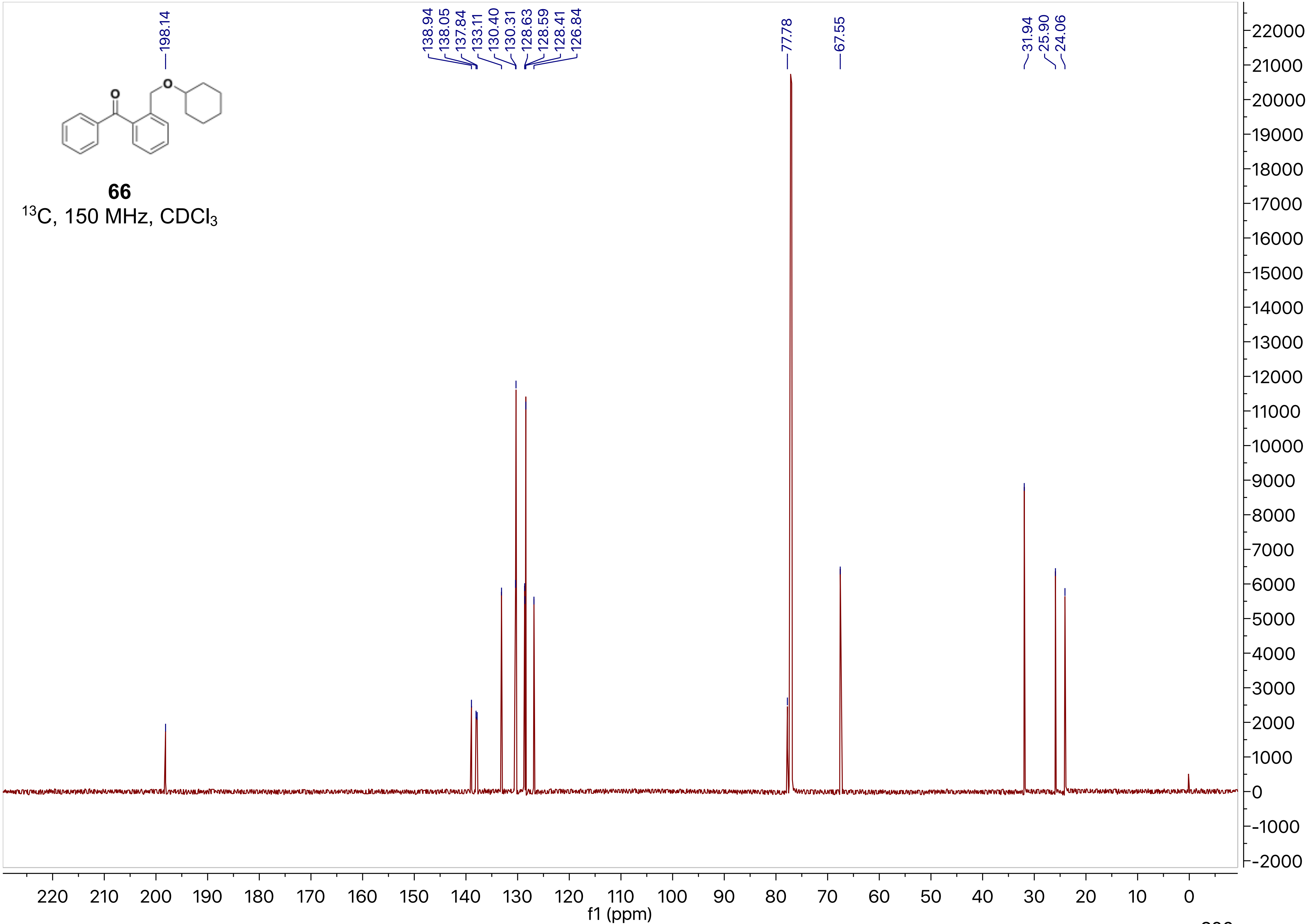

65
^{13}C, 150 MHz, $CDCl_3$
170.09
136.39
135.16
129.45
127.72
126.11
78.14
67.66
66.93
47.81
42.11
32.45
25.90
24.31
f1 (ppm)

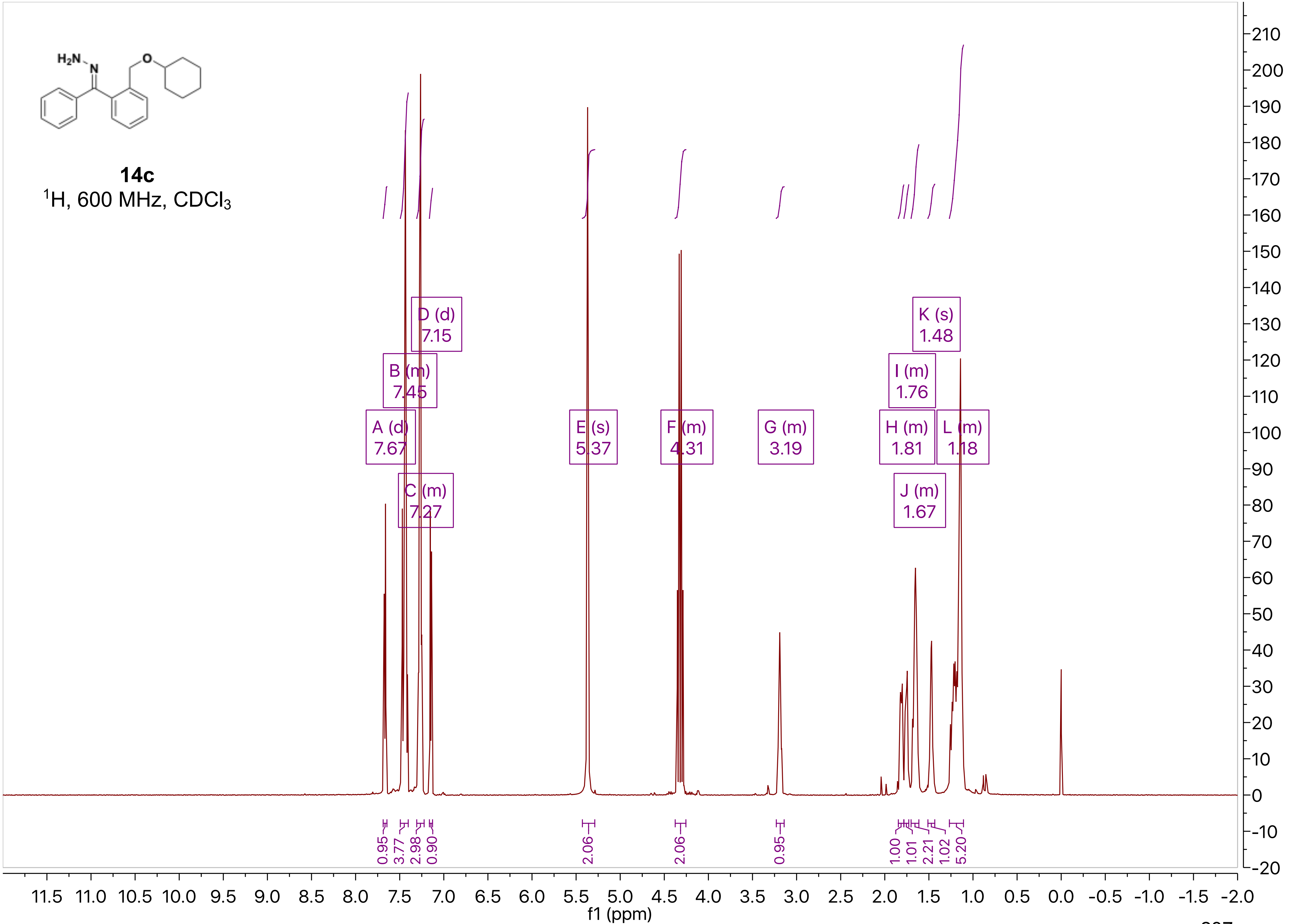


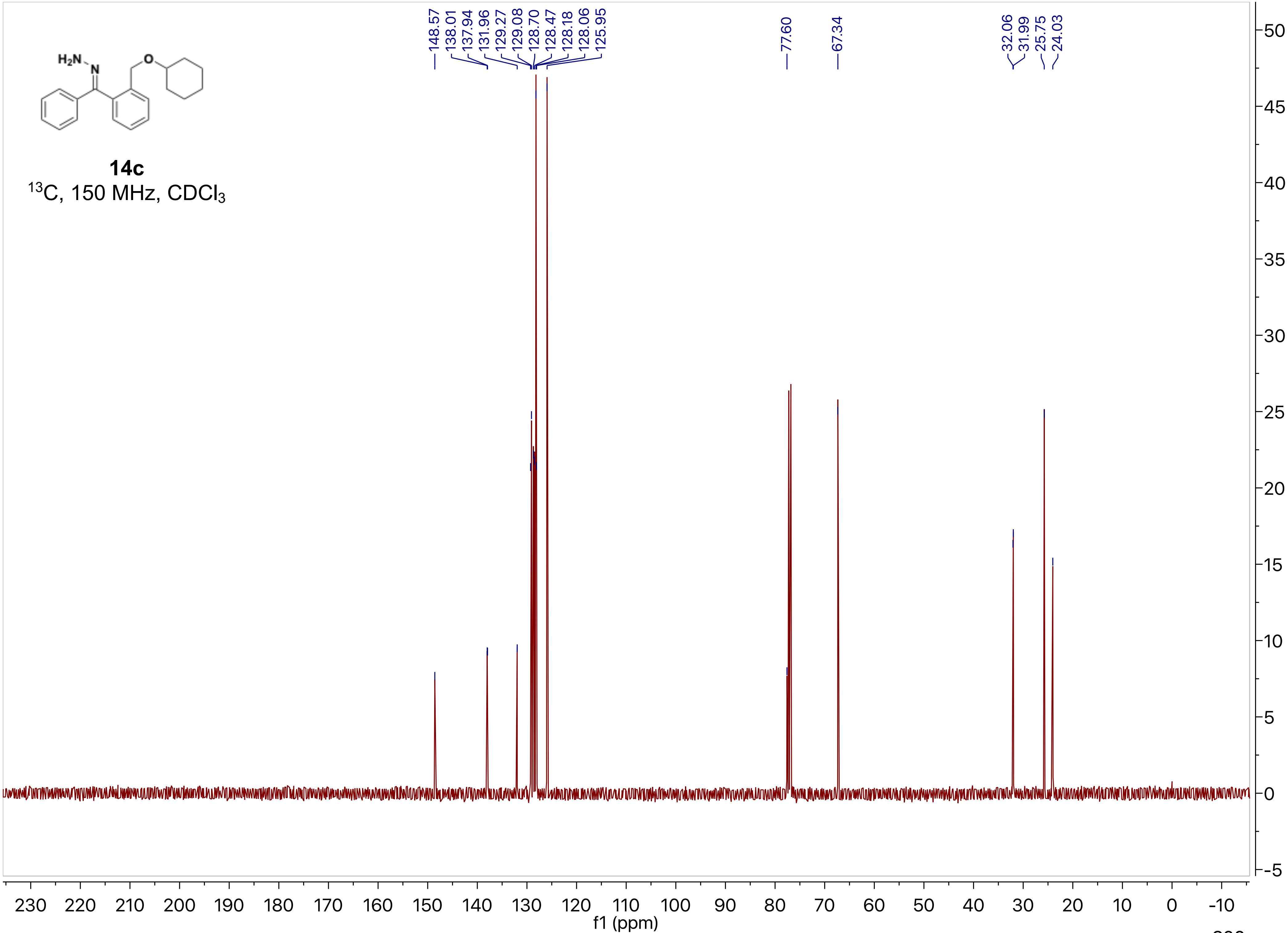
66

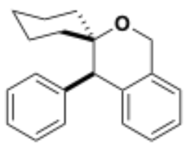
^1H, 600 MHz, $CDCl_3$




198.14
138.94
138.05
137.84
133.11
130.40
130.31
128.63
128.59
128.41
126.84
77.78
67.55
31.94
25.90
24.06
66
^{13}C, 150 MHz, CDCl$_3$
f1 (ppm)

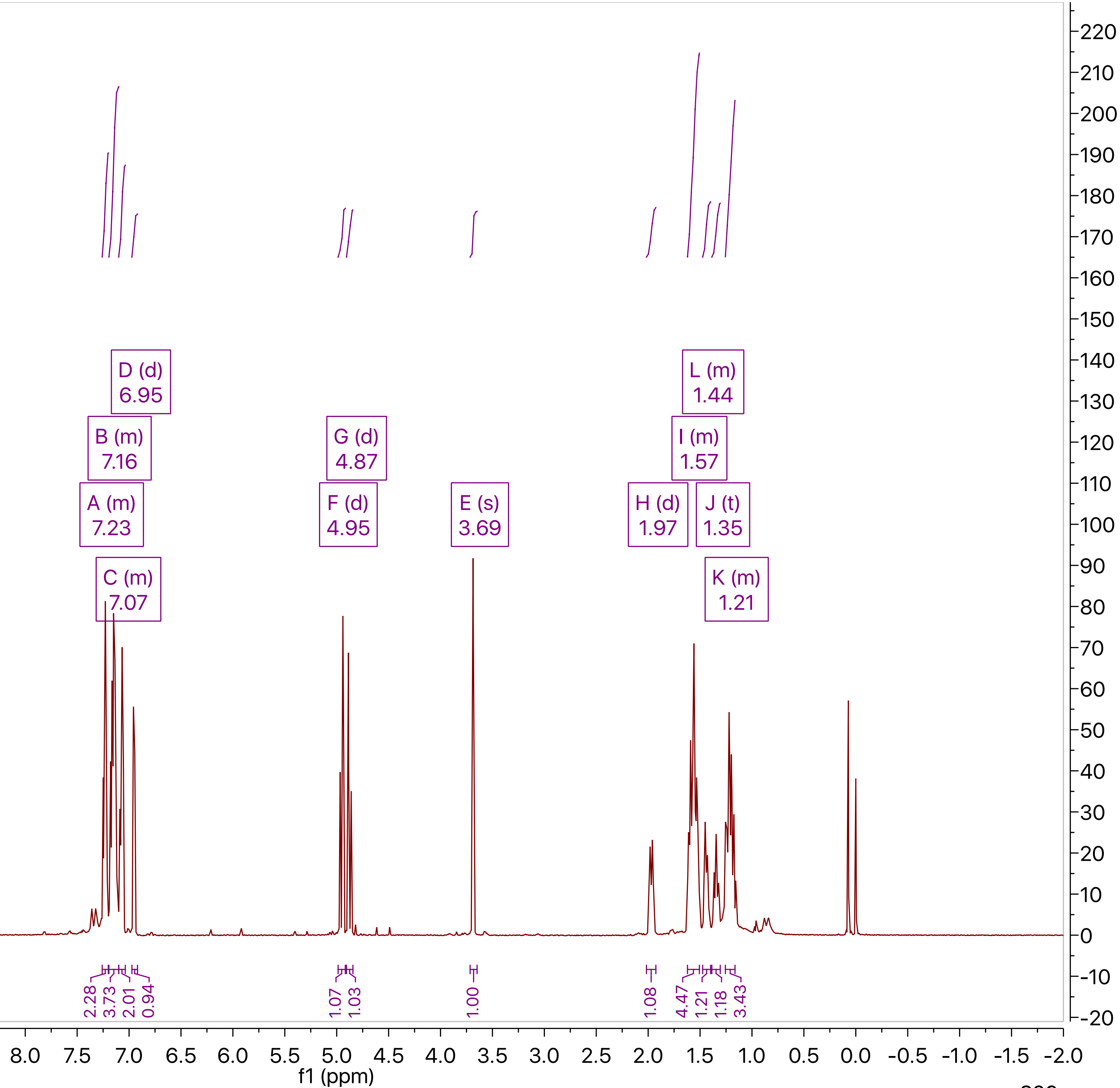

14c
1H, 600 MHz, CDCl3
H2N
N
O
A (d) 7.67
B (m) 7.45
C (m) 7.27
D (d) 7.15
E (s) 5.37
F (m) 4.31
G (m) 3.19
H (m) 1.81
I (m) 1.76
J (m) 1.67
K (s) 1.48
L (m) 1.18
0.95
3.77
2.98
0.90
2.06
2.06
0.95
1.00
1.01
2.21
1.02
5.20
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5 -2.0
f1 (ppm)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20

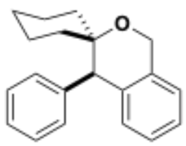

14c
13C, 150 MHz, CDCl3
148.57
138.01
137.94
131.96
129.27
129.08
128.70
128.47
128.18
128.06
125.95
77.60
67.34
32.06
31.99
25.75
24.03
f1 (ppm)



15c

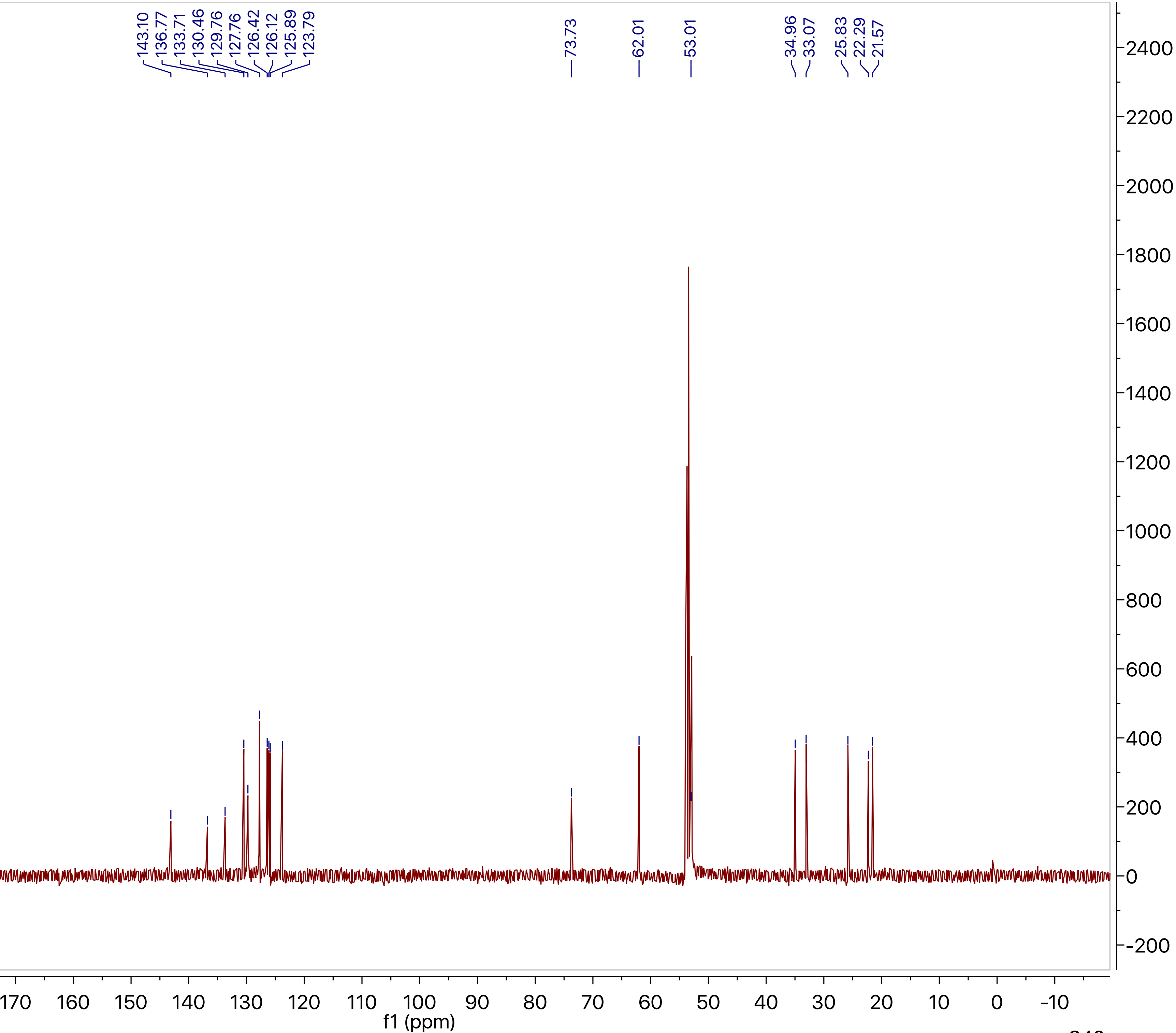
^{1}H, 400 MHz, $CDCl_3$

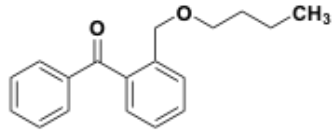




15c

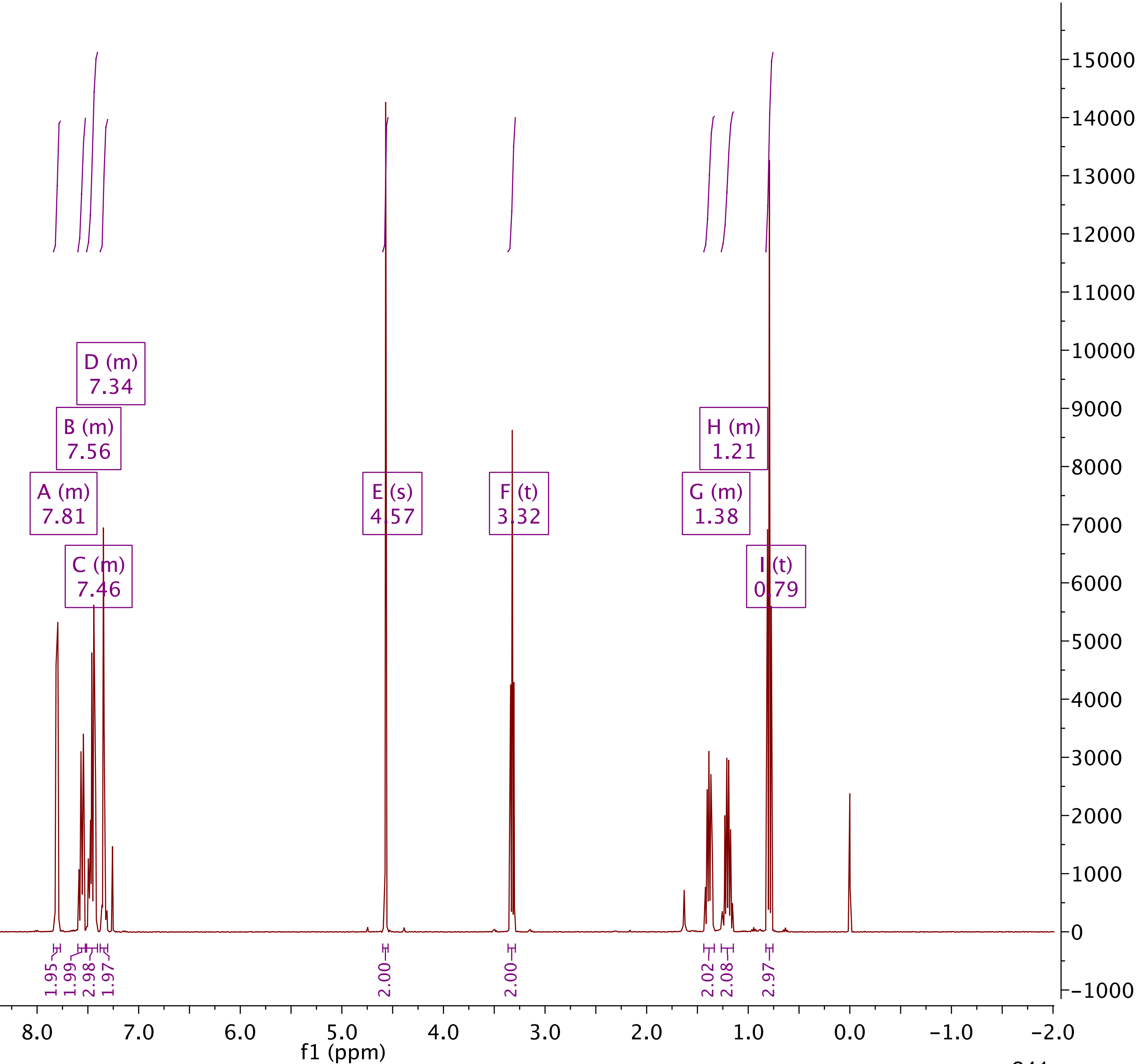
^{13}C, 100 MHz, CD_2Cl_2

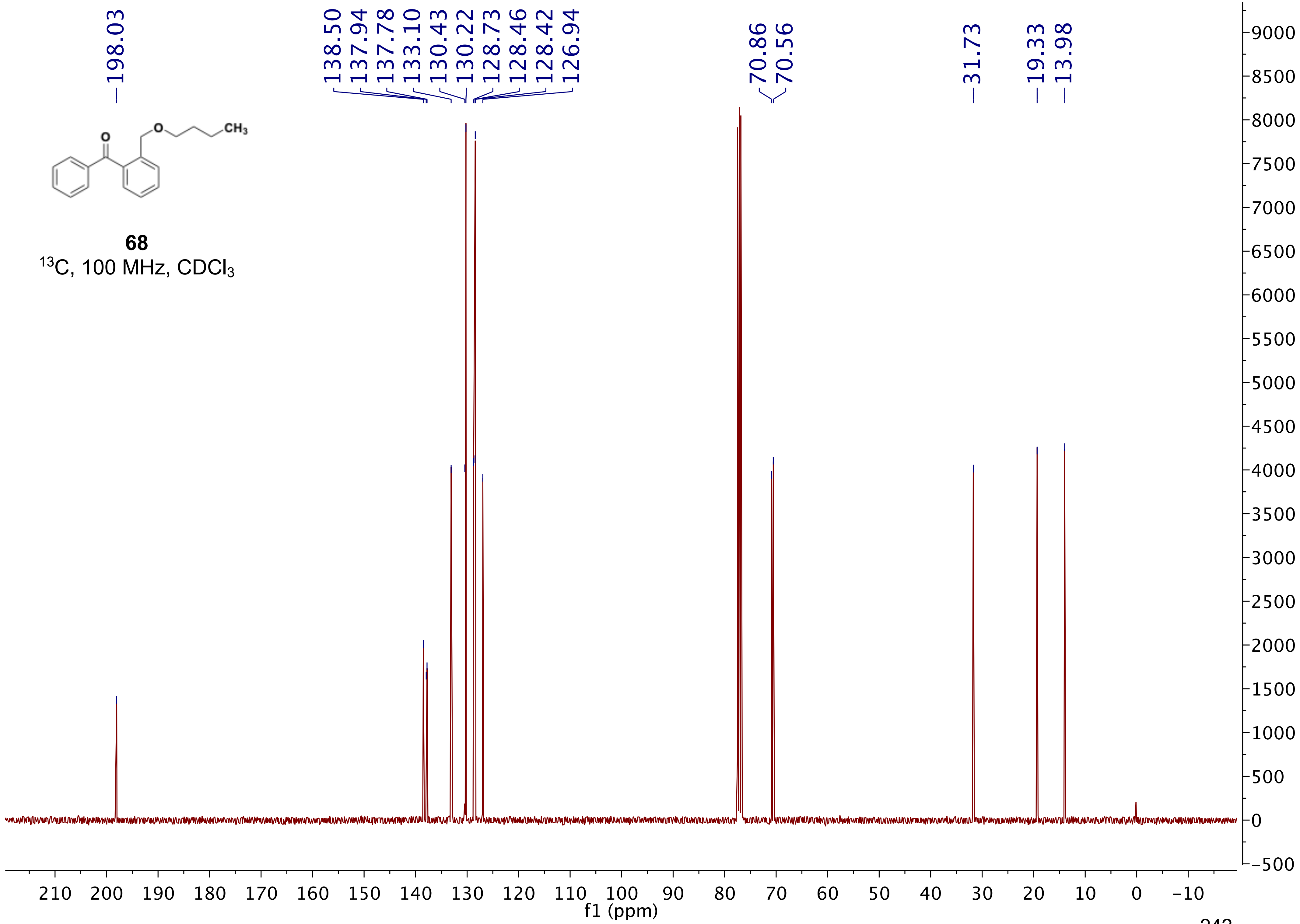




68

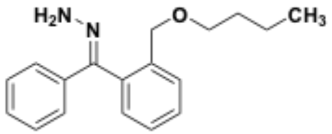
^{1}H, 400 MHz, $CDCl_3$





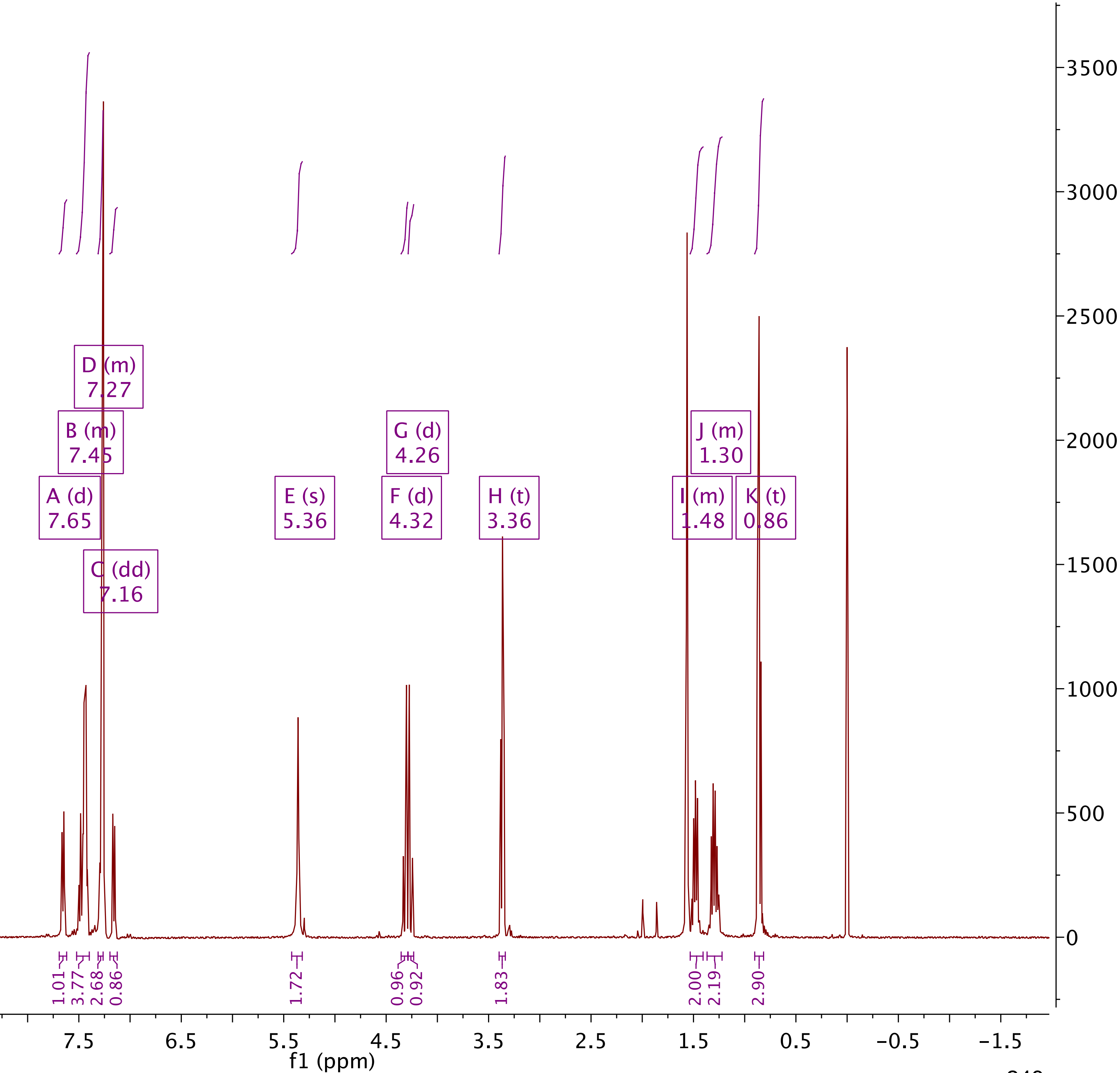
68

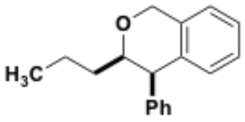
^{13}C, 100 MHz, $CDCl_3$



14d

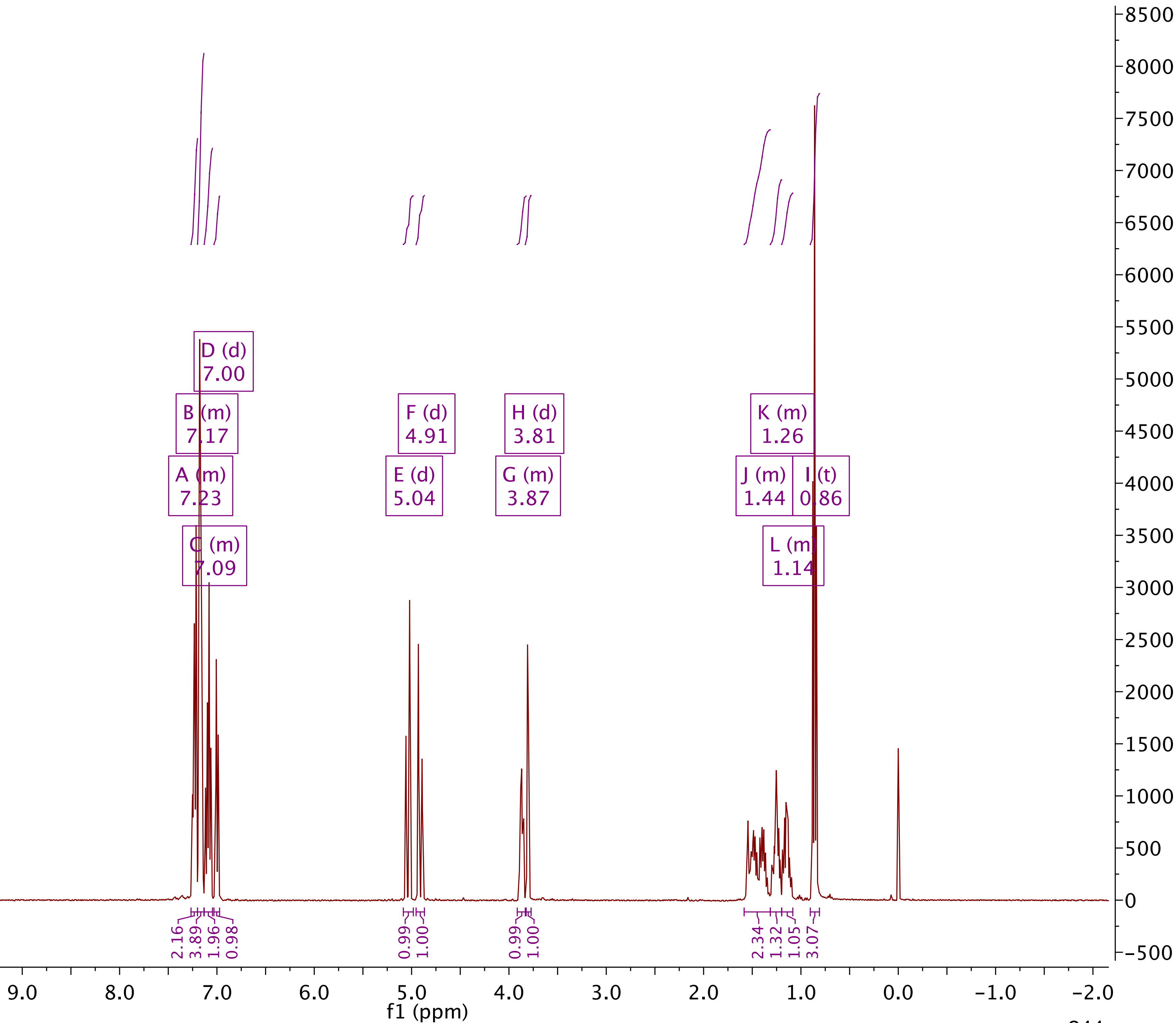
^{1}H, 400 MHz, $CDCl_3$

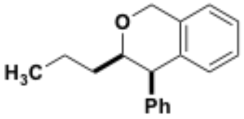




15d

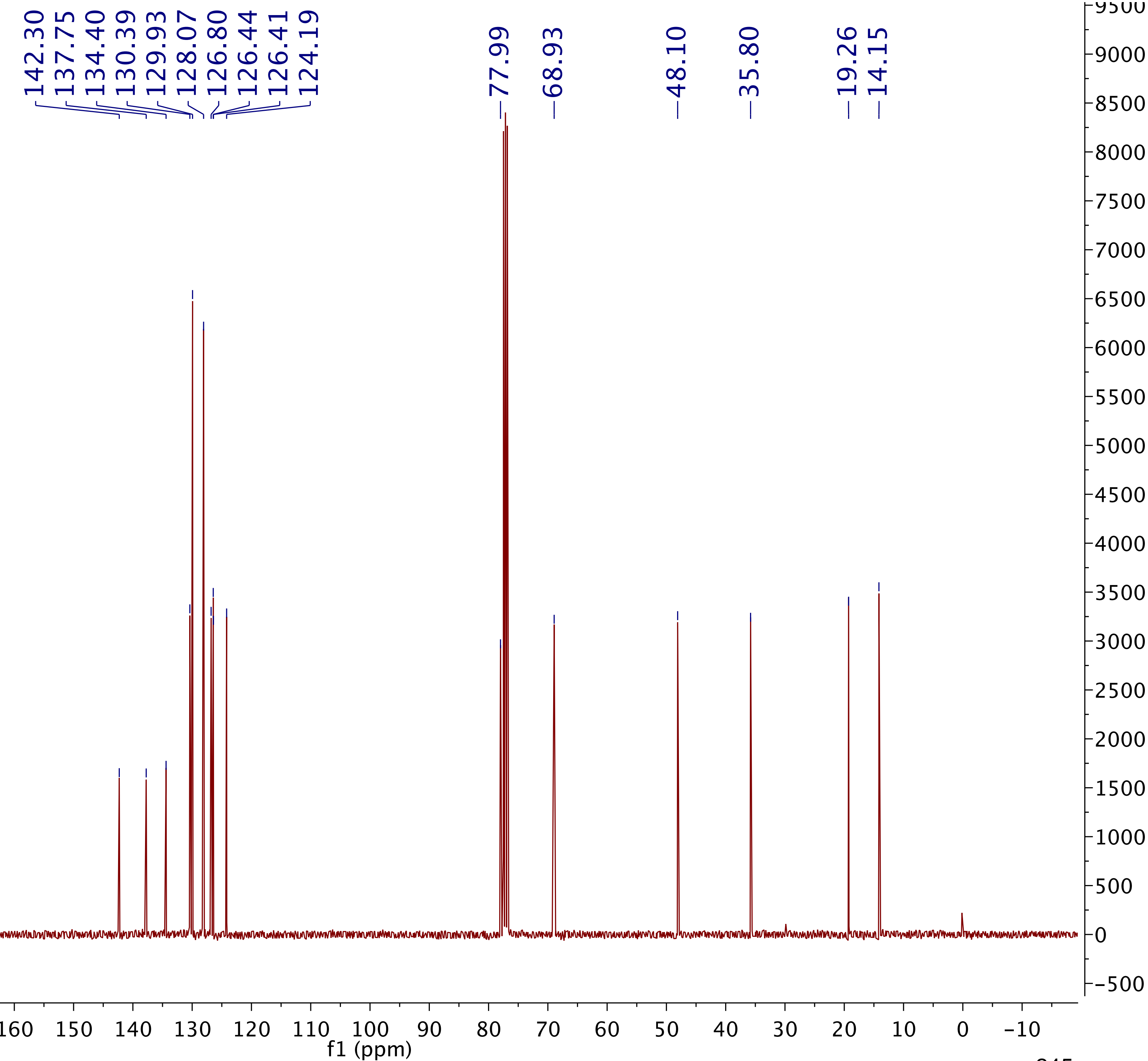
^{1}H, 400 MHz, $CDCl_3$

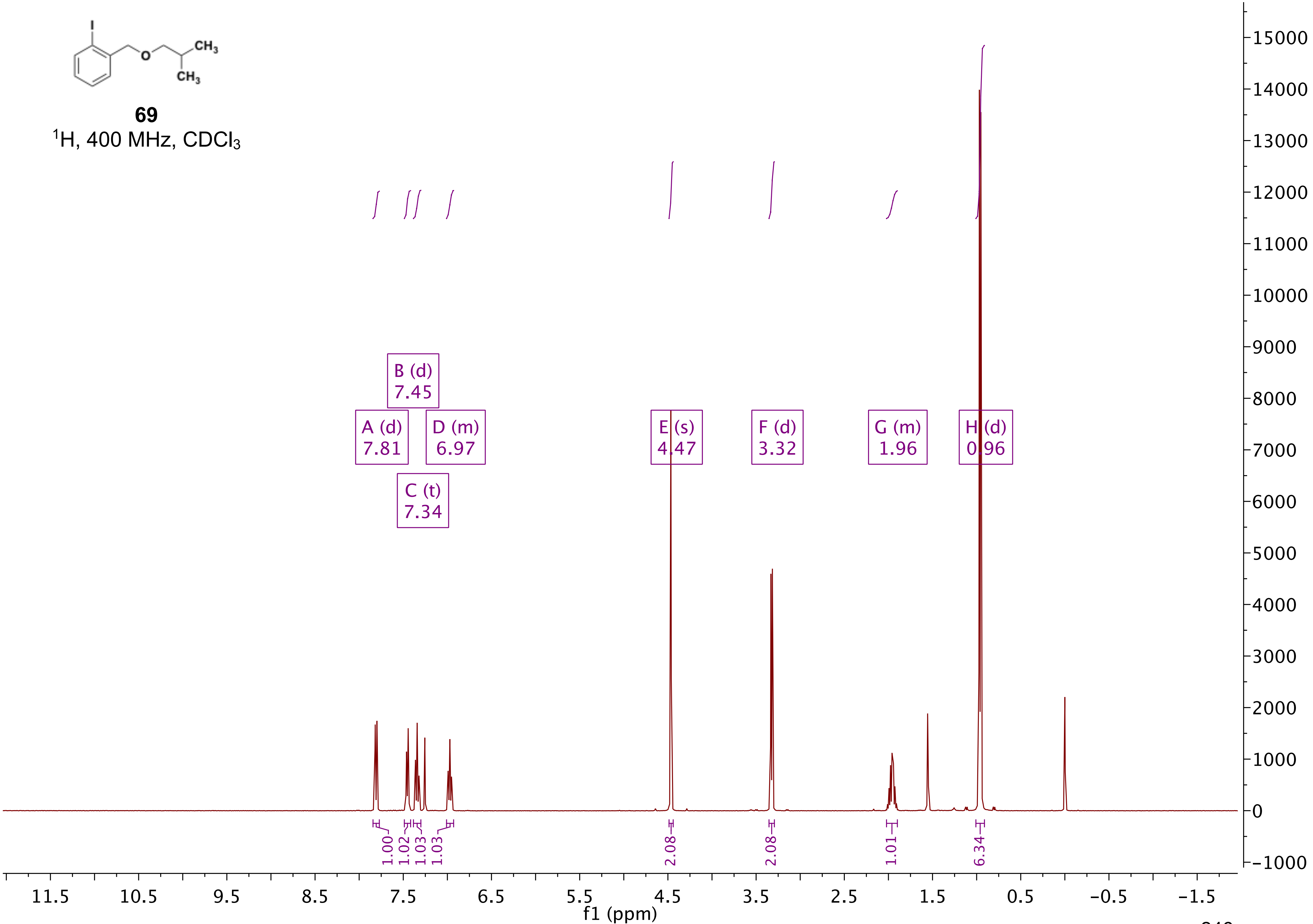




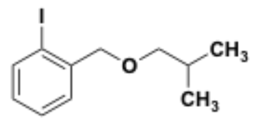
15d

^{13}C, 100 MHz, $CDCl_3$



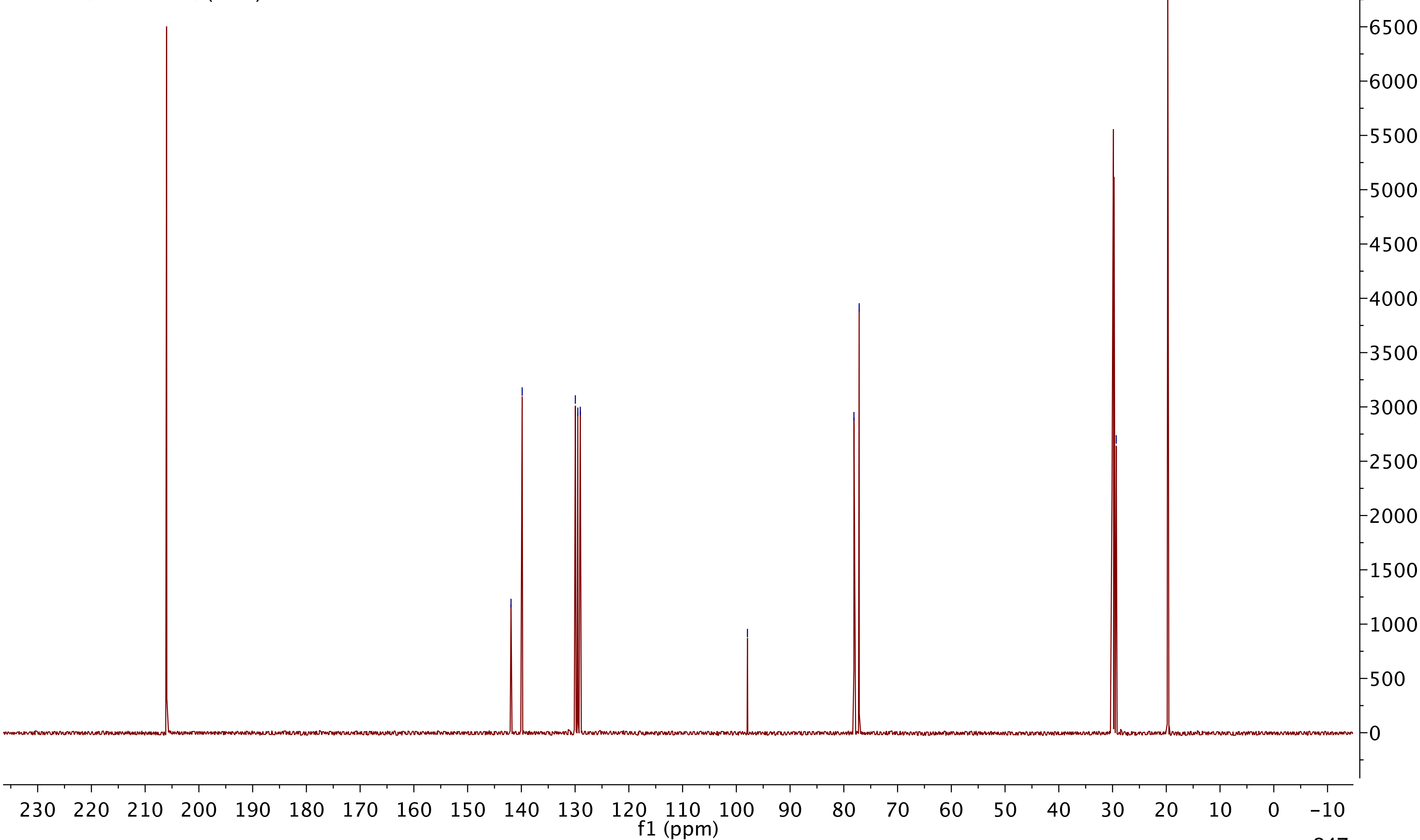


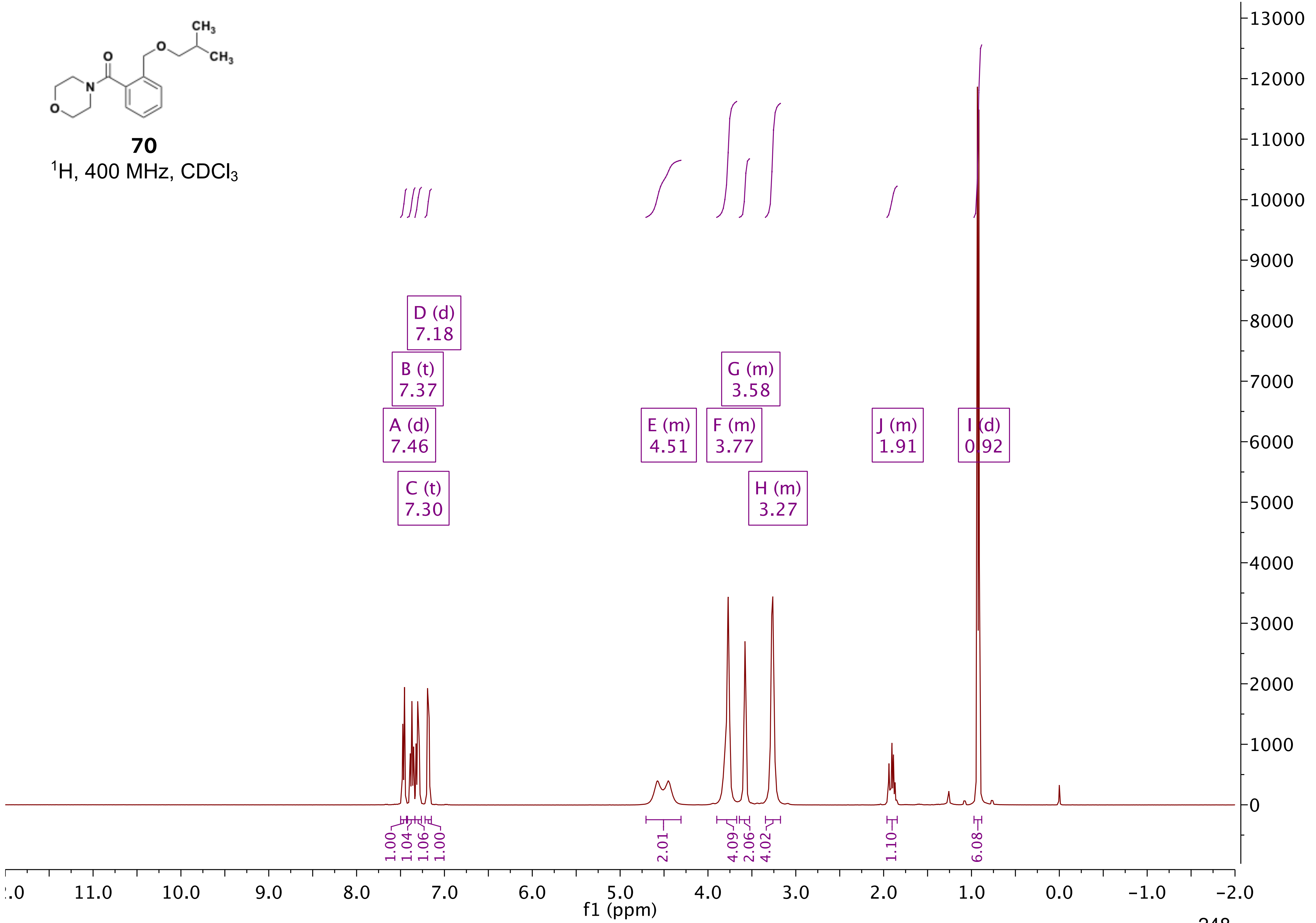
69
[1]H, 400 MHz, CDCl3
A (d) 7.81
B (d) 7.45
C (t) 7.34
D (m) 6.97
E (s) 4.47
F (d) 3.32
G (m) 1.96
H (d) 0.96
1.00
1.02
1.03
1.03
2.08
2.08
1.01
6.34
f1 (ppm)



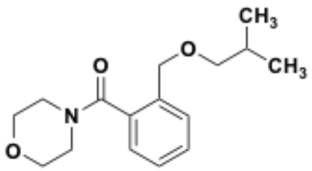
69

^{13}C, 150 MHz, $(CD_3)_2CO$



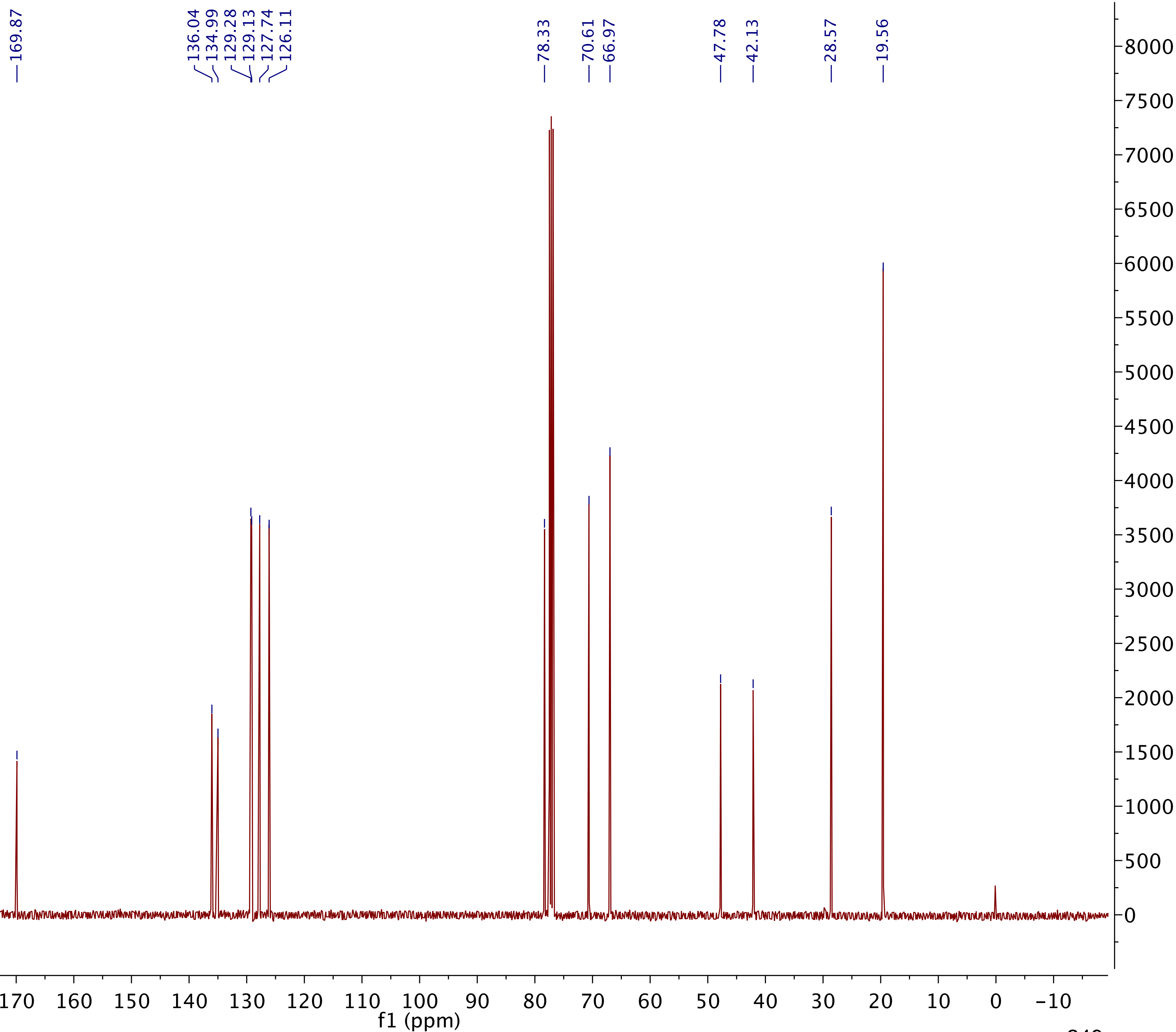


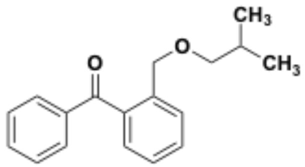
70
¹H, 400 MHz, CDCl₃
D (d) 7.18
B (t) 7.37
A (d) 7.46
C (t) 7.30
E (m) 4.51
F (m) 3.77
G (m) 3.58
H (m) 3.27
J (m) 1.91
I (d) 0.92
1.00
1.04
1.06
1.00
2.01
4.09
2.06
4.02
1.10
6.08
f1 (ppm)



70

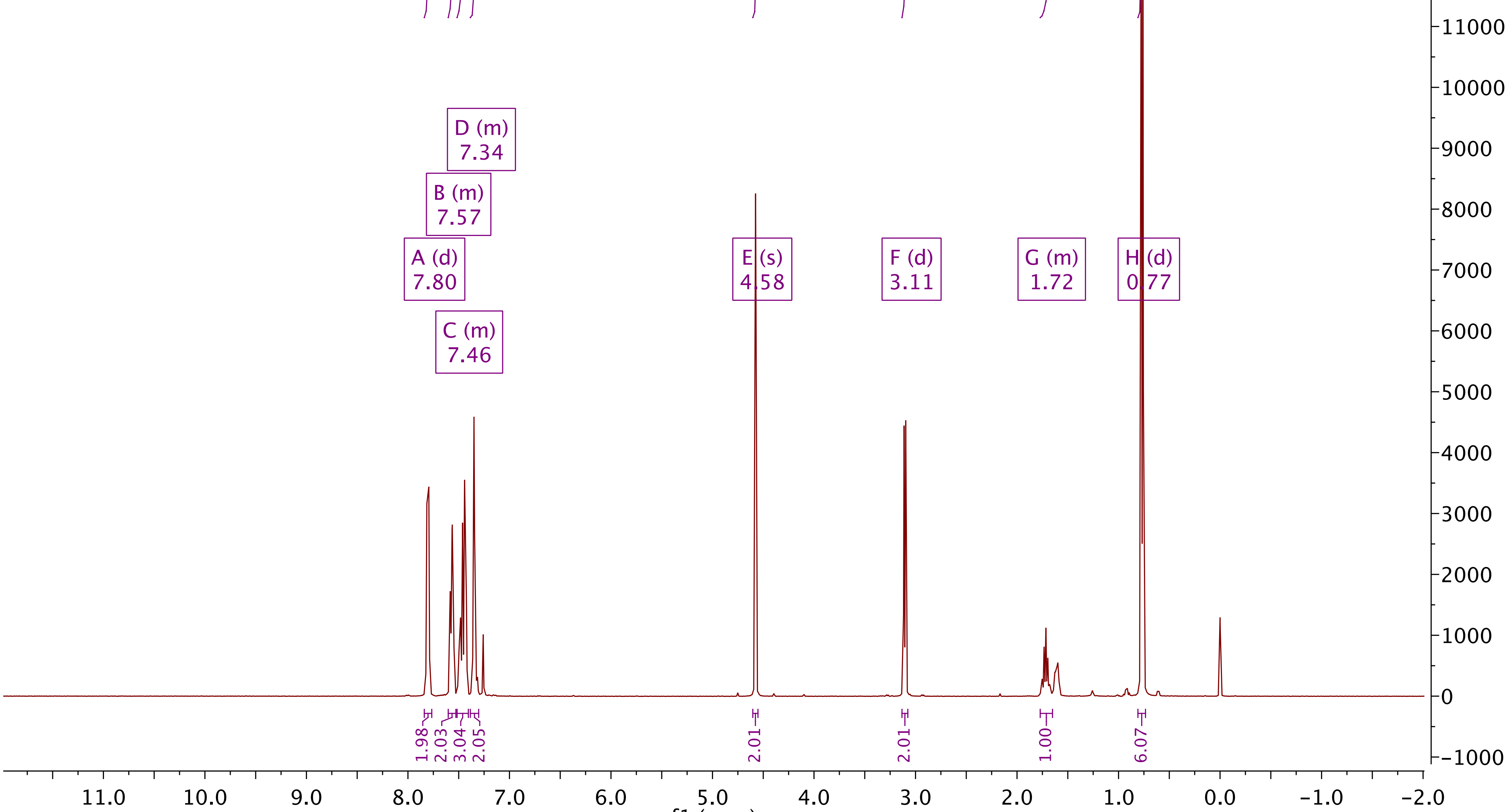
^{13}C, 100 MHz, $CDCl_3$

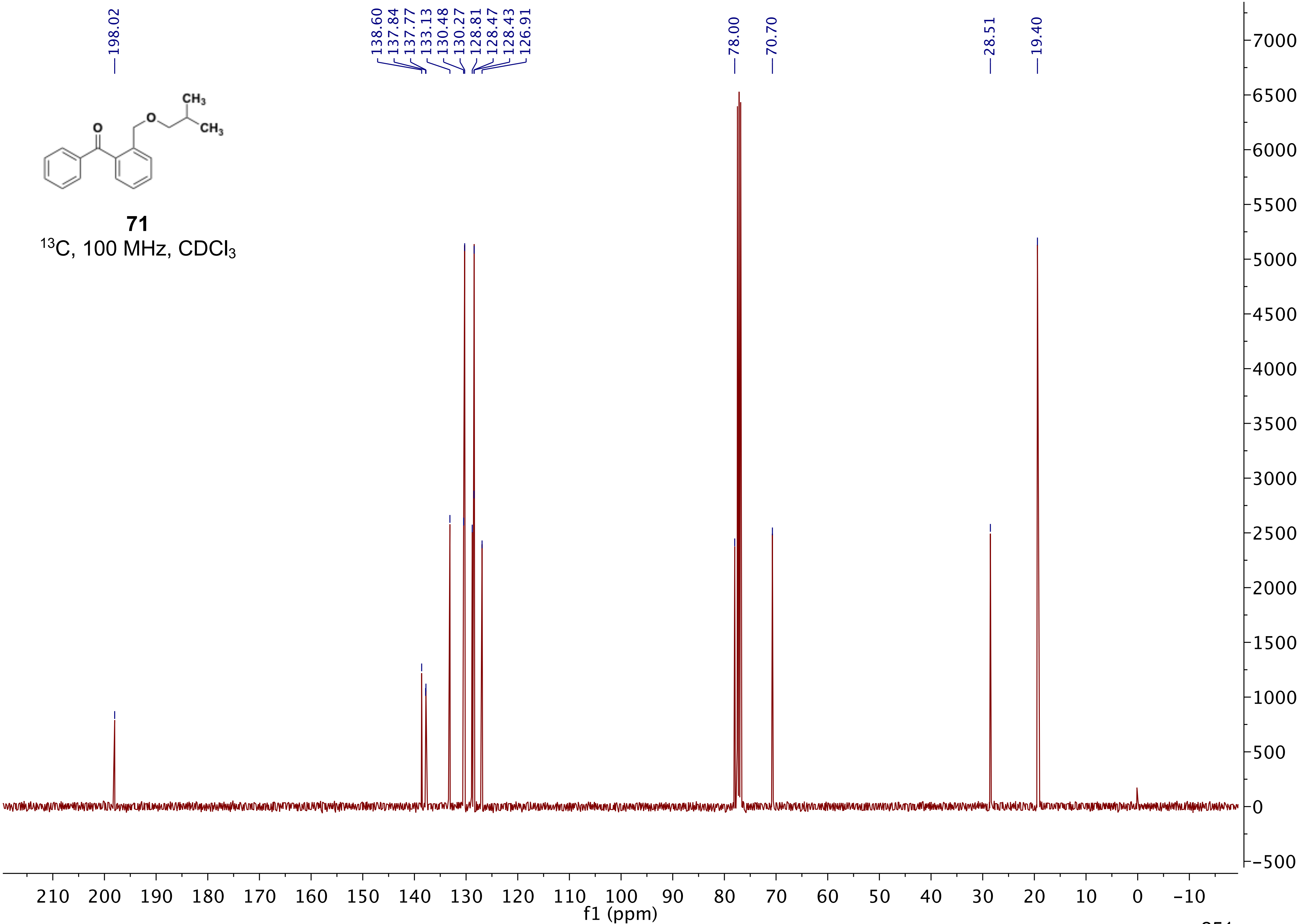




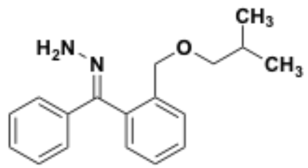
71

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

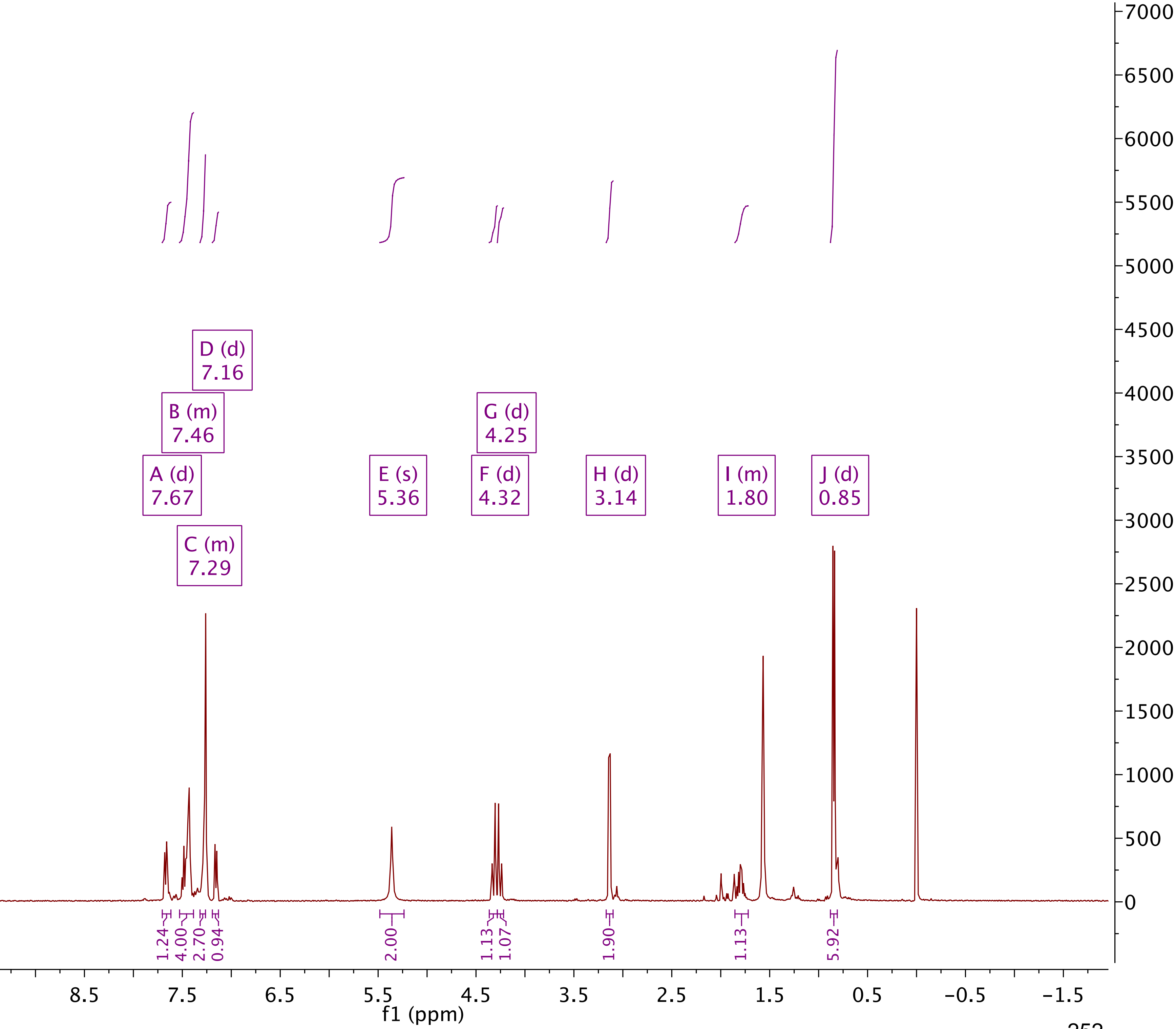


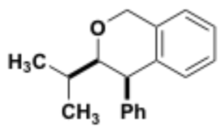


198.02
138.60
137.84
137.77
133.13
130.48
130.27
128.81
128.47
128.43
126.91
78.00
70.70
28.51
19.40
O
O
CH3
CH3
71
13C, 100 MHz, CDCl3
f1 (ppm)



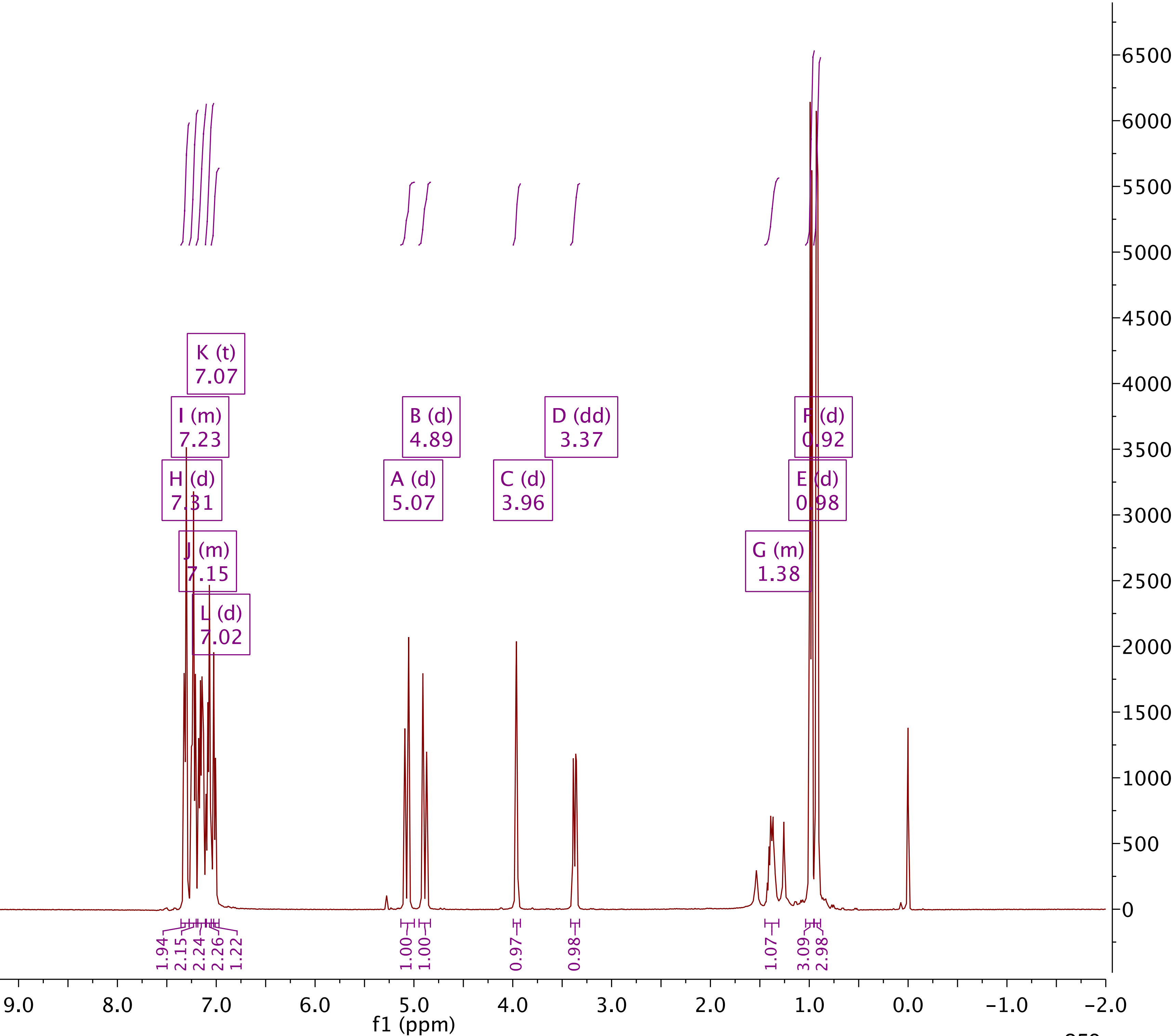
14e
^{1}H, 400 MHz, $CDCl_3$

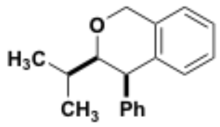




15e

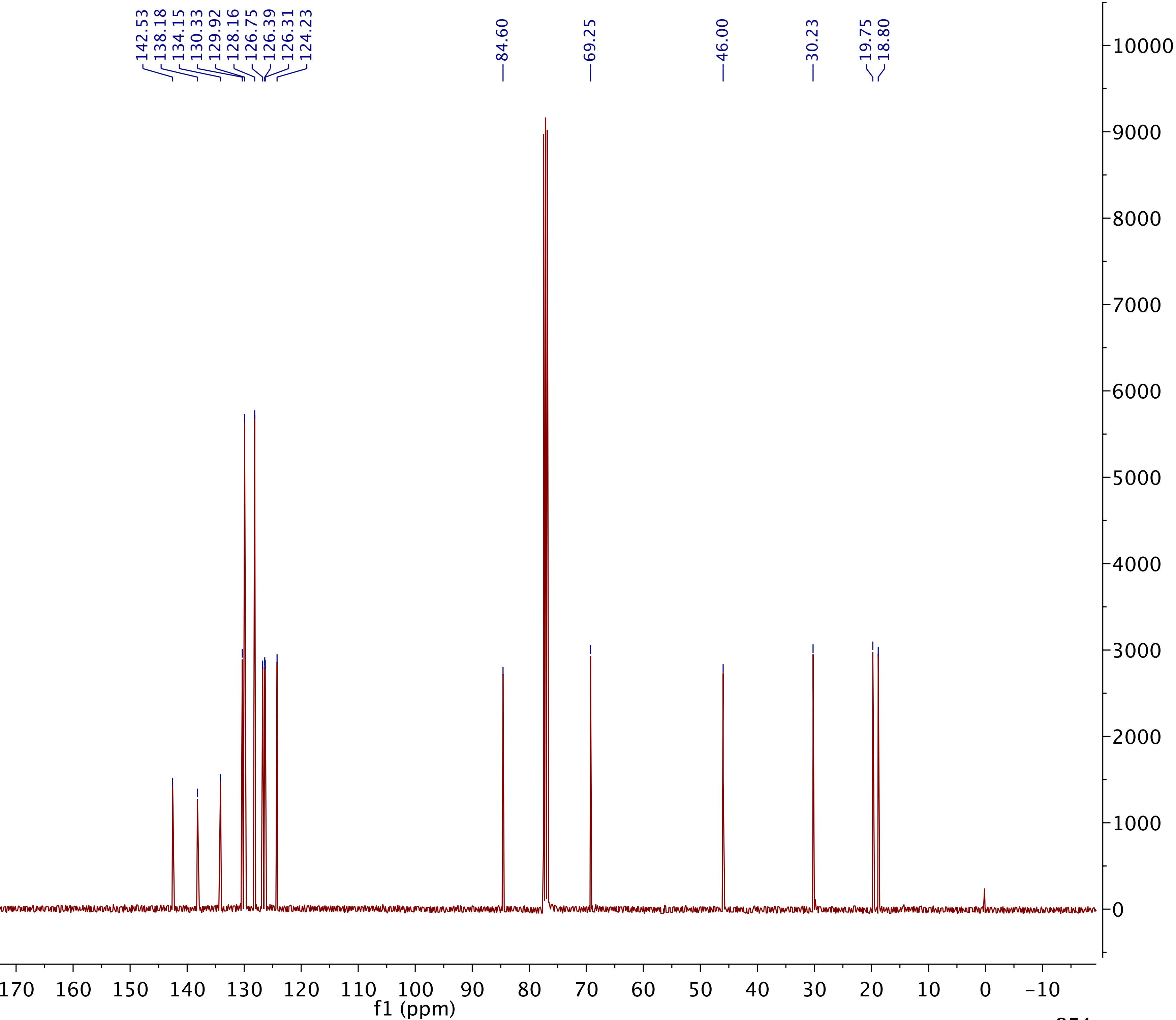
^{1}H, 400 MHz, $CDCl_3$

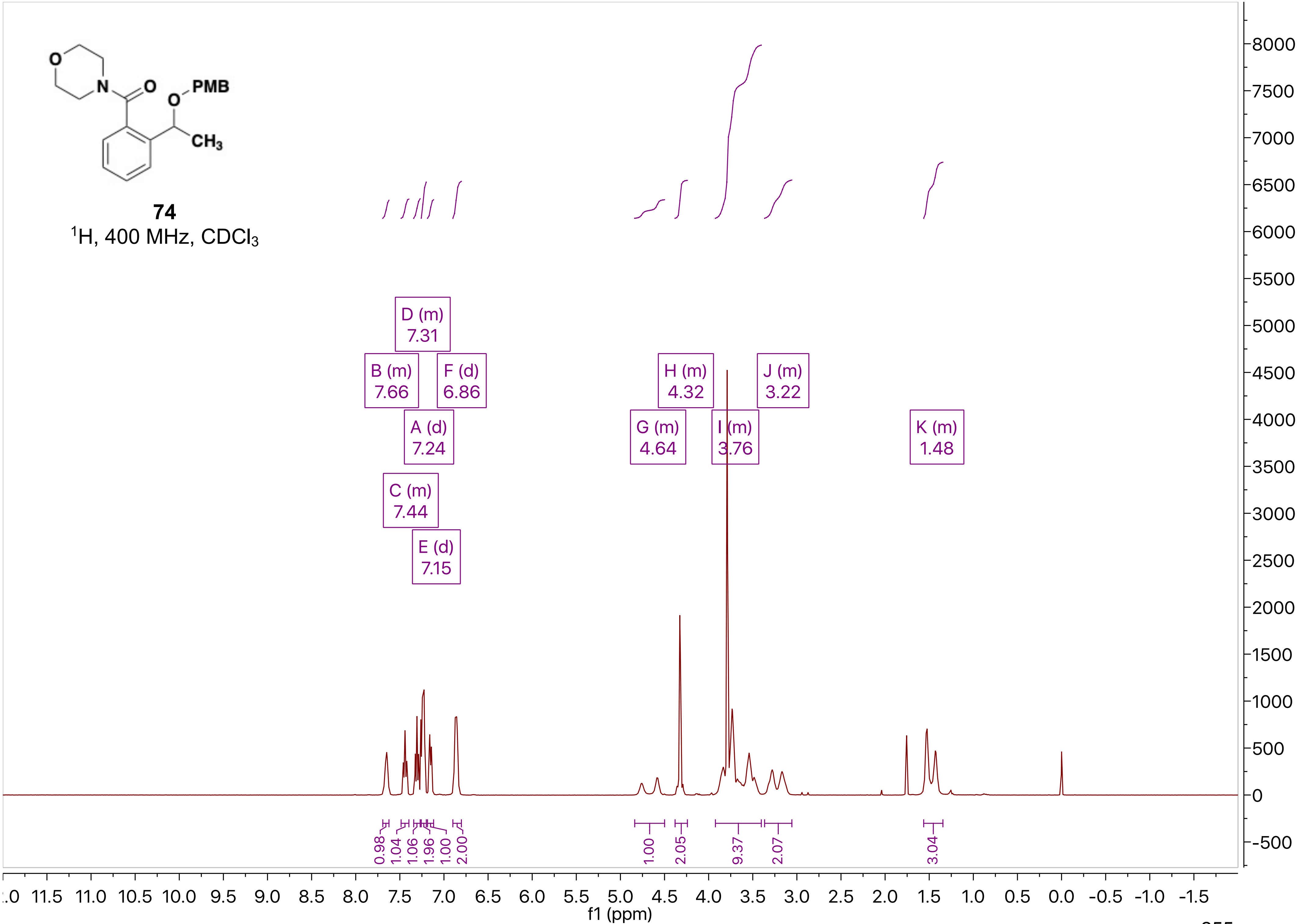




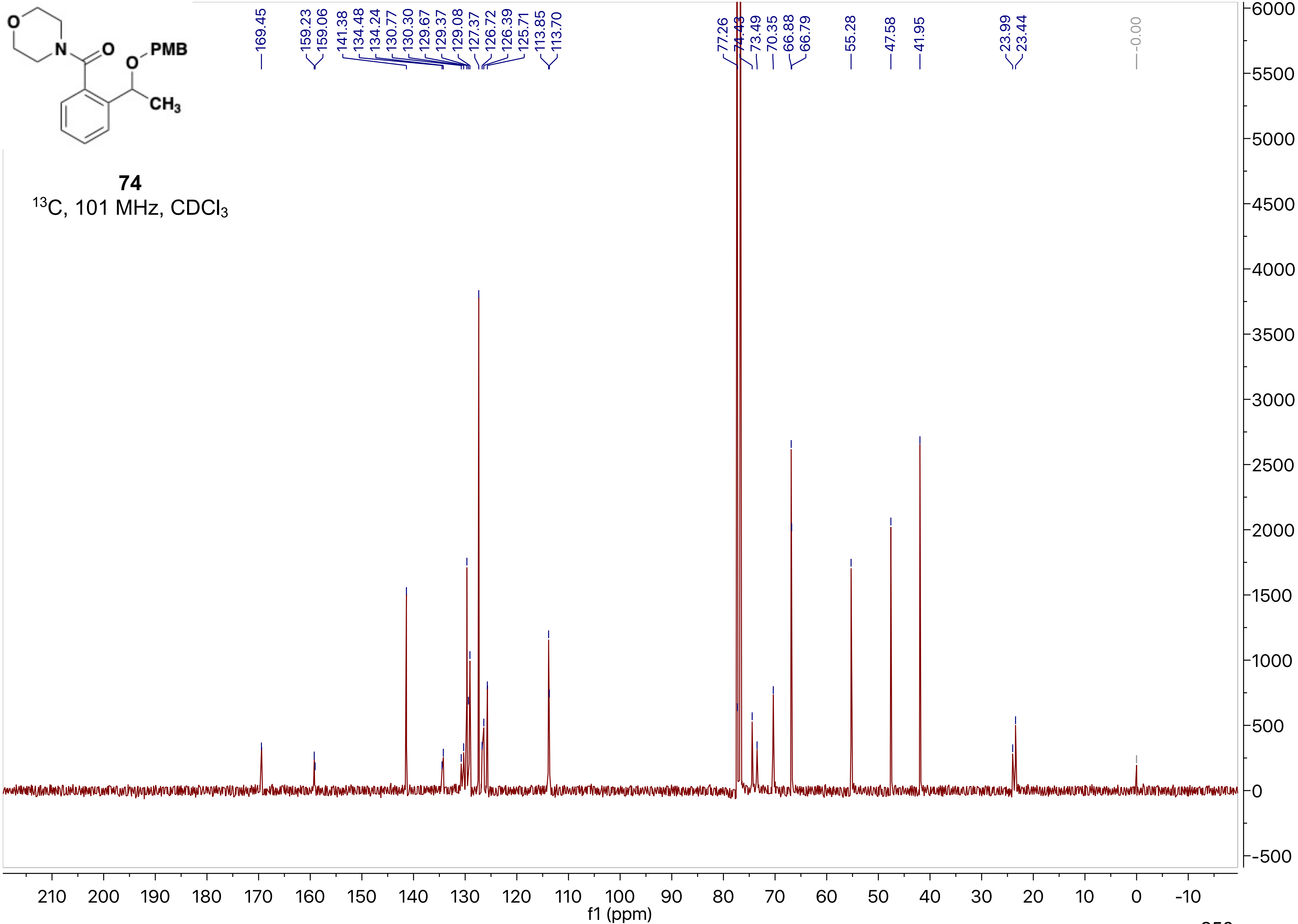
15e

^{13}C, 100 MHz, $CDCl_3$

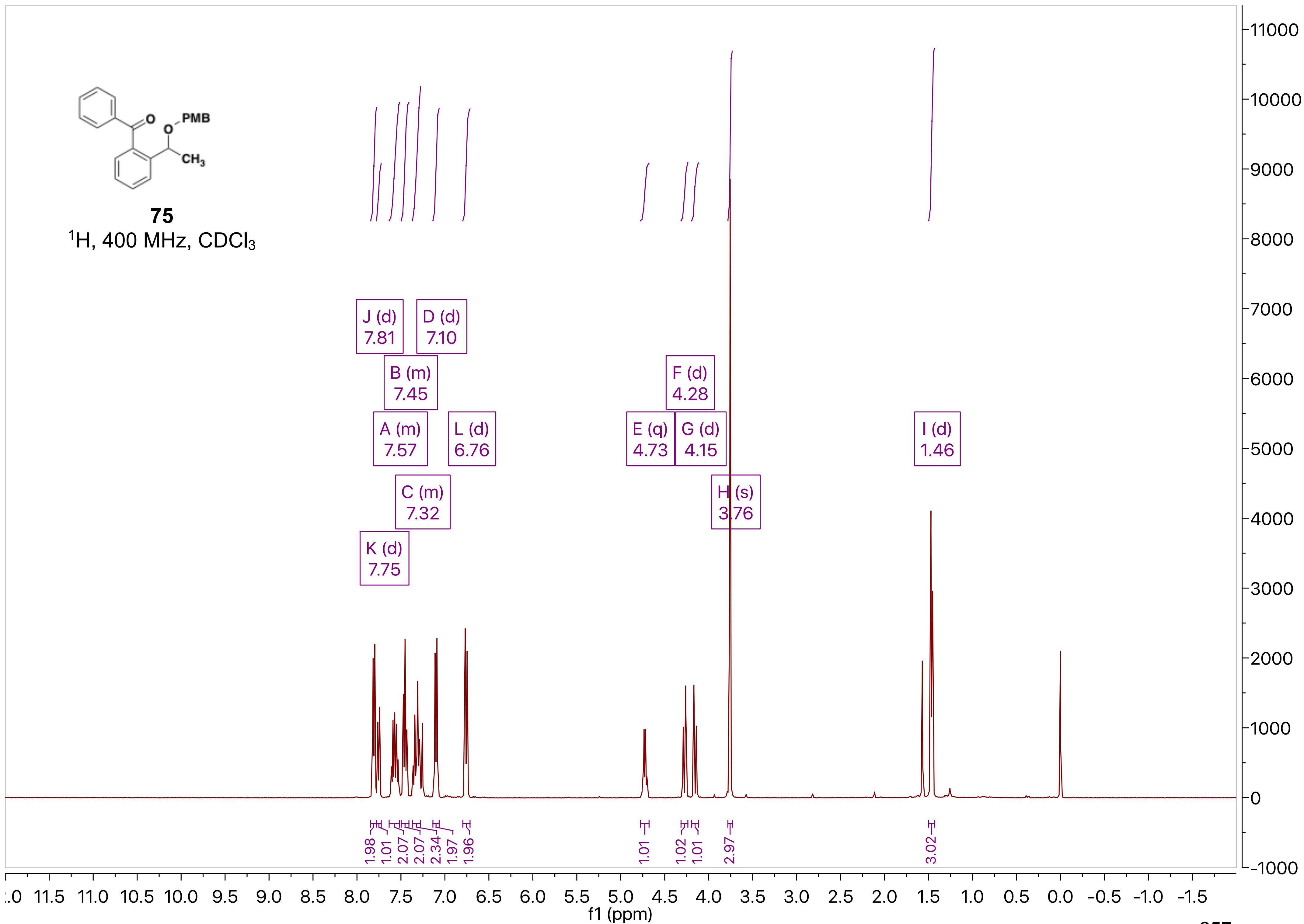


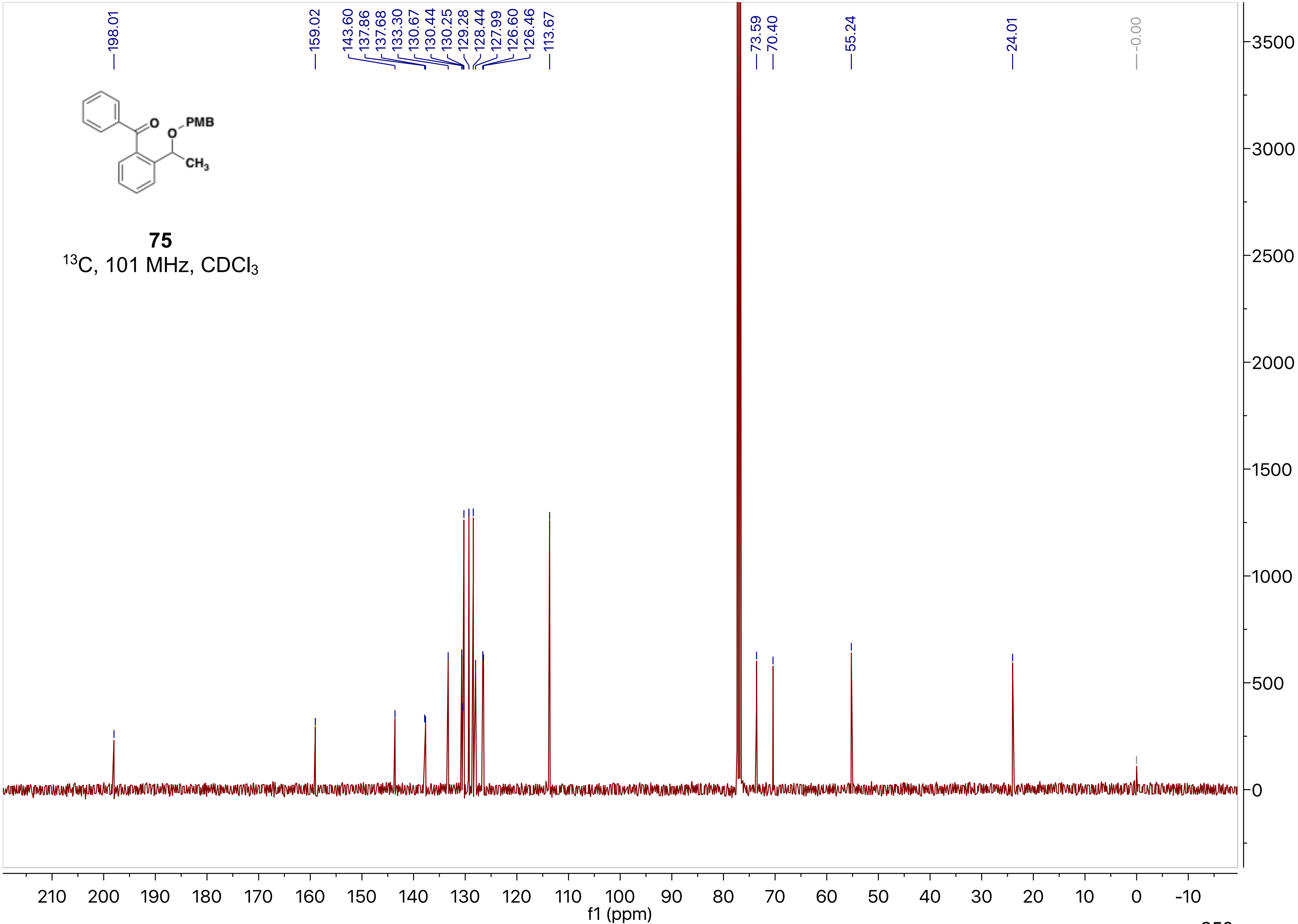


O
N
O
O
PMB
CH3
74
1H, 400 MHz, CDCl3
D (m) 7.31
B (m) 7.66
F (d) 6.86
A (d) 7.24
C (m) 7.44
E (d) 7.15
H (m) 4.32
J (m) 3.22
G (m) 4.64
I (m) 3.76
K (m) 1.48
0.98
1.04
1.06
1.96
1.00
2.00
1.00
2.05
9.37
2.07
3.04
f1 (ppm)

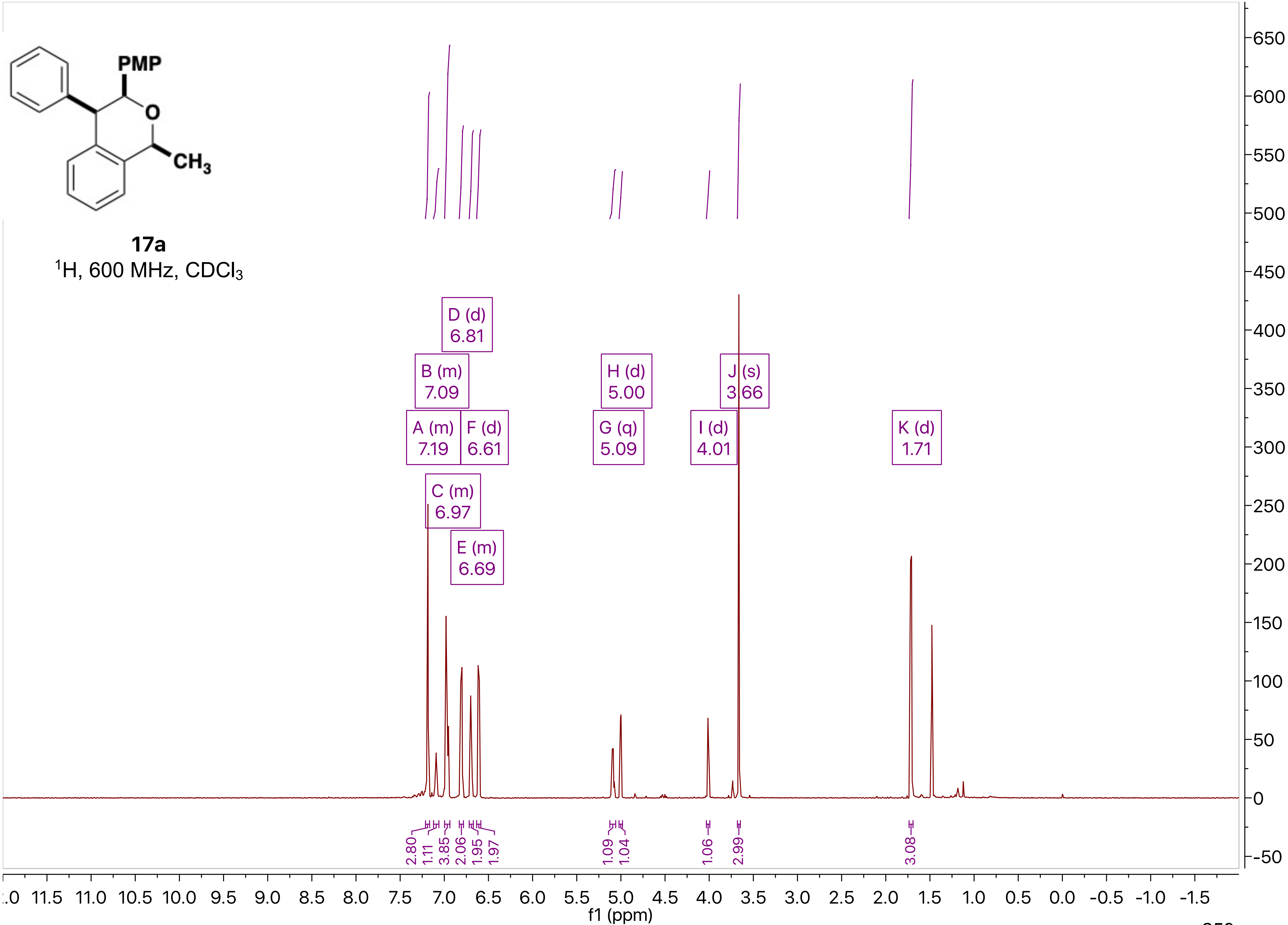


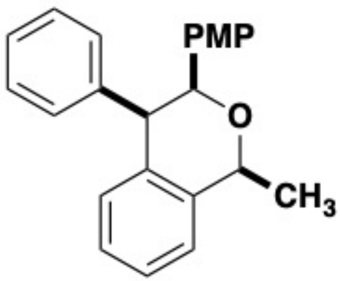
O
N
O
O
PMB
CH3
74
13C, 101 MHz, CDCl3
169.45
159.23
159.06
141.38
134.48
134.24
130.77
130.30
129.67
129.37
129.08
127.37
126.72
126.39
125.71
113.85
113.70
77.26
74.43
73.49
70.35
66.88
66.79
55.28
47.58
41.95
23.99
23.44
-0.00
6000
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


PMB
CH3
75
1H, 400 MHz, CDCl3
J (d) 7.81
D (d) 7.10
B (m) 7.45
A (m) 7.57
L (d) 6.76
C (m) 7.32
K (d) 7.75
F (d) 4.28
E (q) 4.73
G (d) 4.15
H (s) 3.76
I (d) 1.46
1.98 1.01 2.07 2.07 2.34 1.97 1.96
1.01 1.02 1.01 2.97 3.02
f1 (ppm)



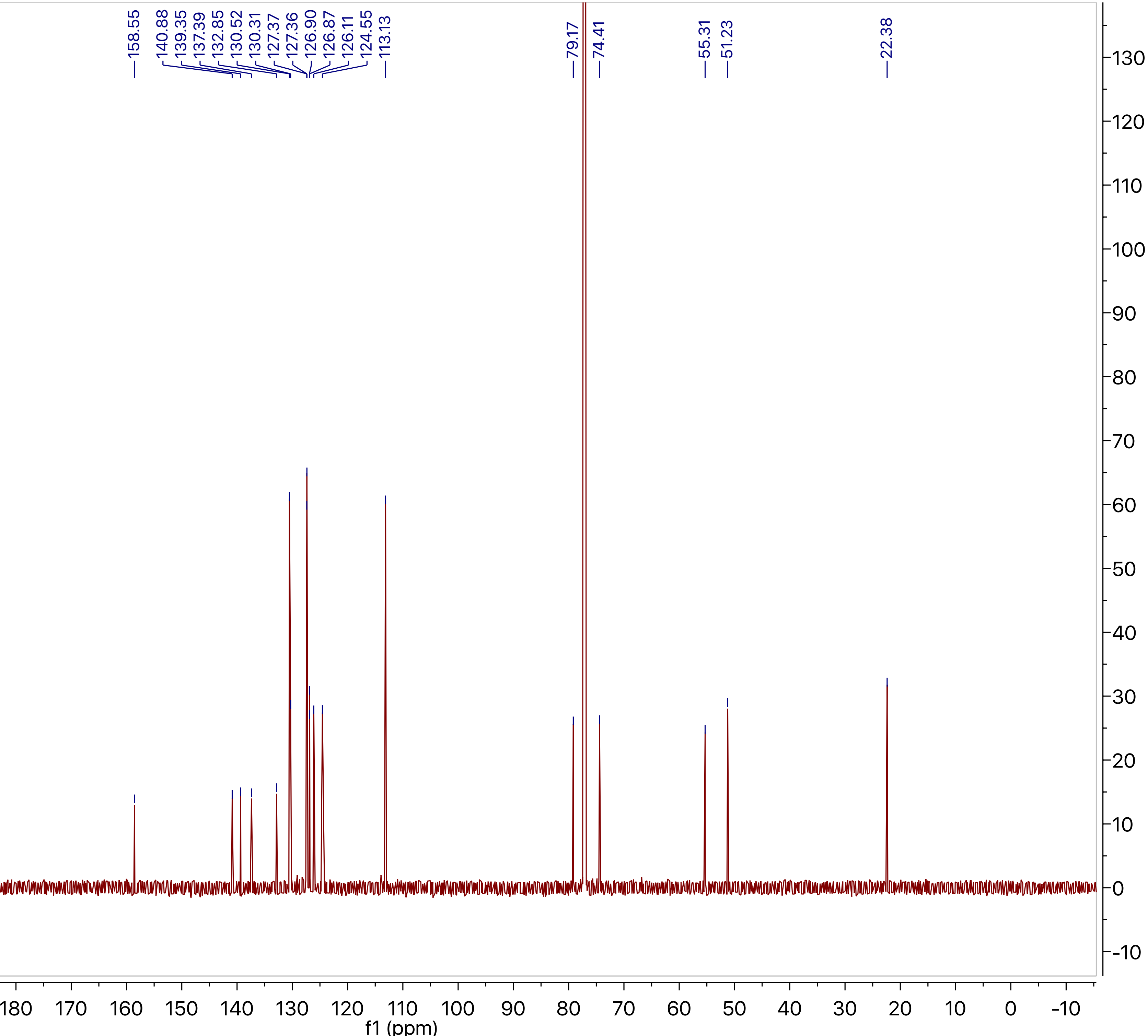
198.01
159.02
143.60
137.86
137.68
133.30
130.67
130.44
130.25
129.28
128.44
127.99
126.60
126.46
113.67
73.59
70.40
55.24
24.01
-0.00
PMB
O
CH3
75
13C, 101 MHz, CDCl3
f1 (ppm)

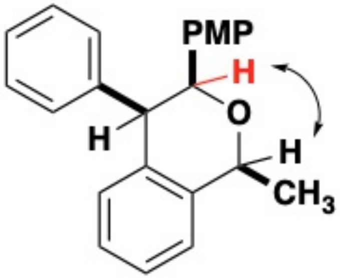

PMP
O
CH3
17a
1H, 600 MHz, CDCl3
D (d) 6.81
B (m) 7.09
A (m) 7.19
F (d) 6.61
C (m) 6.97
E (m) 6.69
H (d) 5.00
G (q) 5.09
I (d) 4.01
J (s) 3.66
K (d) 1.71
2.80
1.11
3.85
2.06
1.95
1.97
1.09
1.04
1.06
2.99
3.08
f1 (ppm)



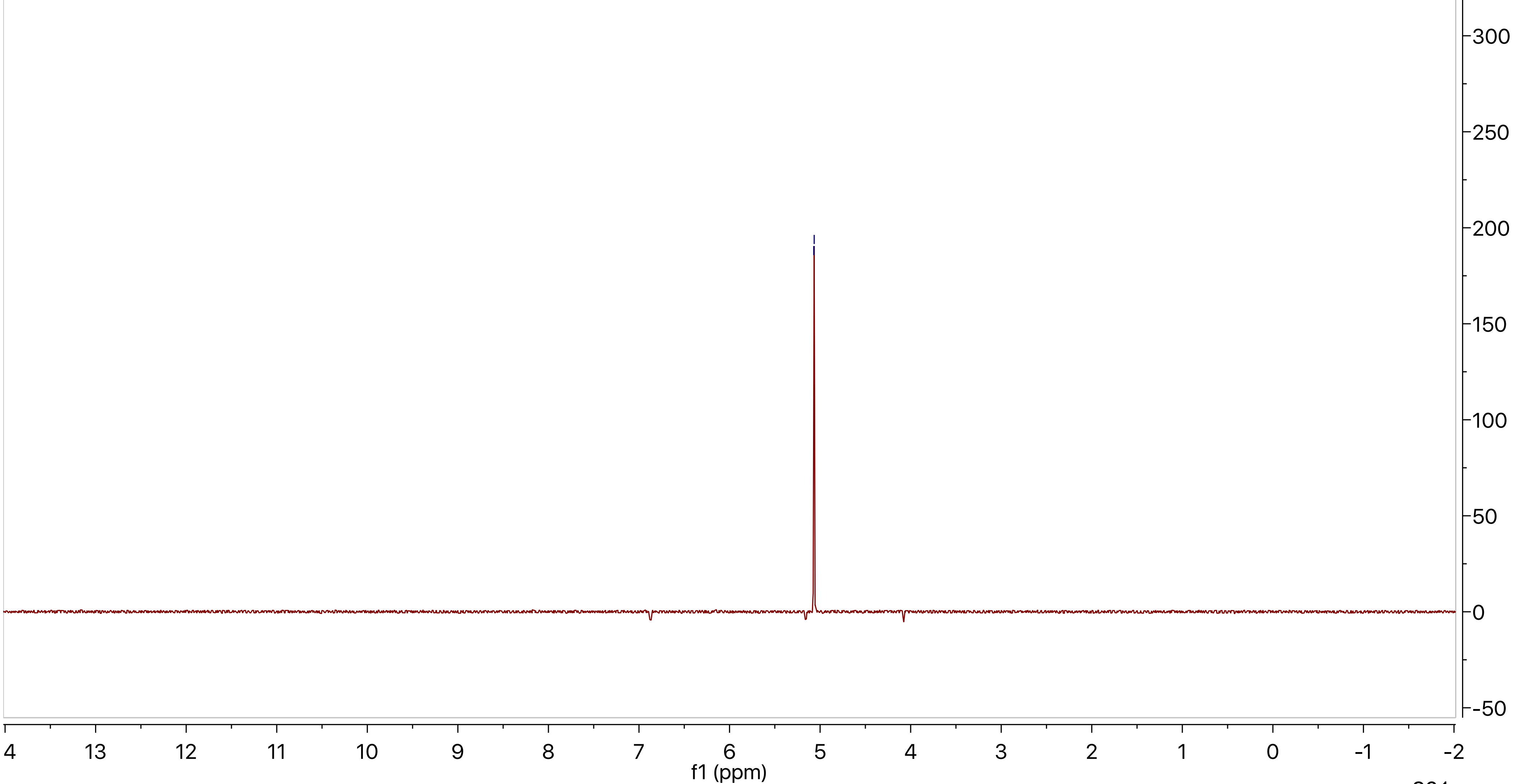
17a

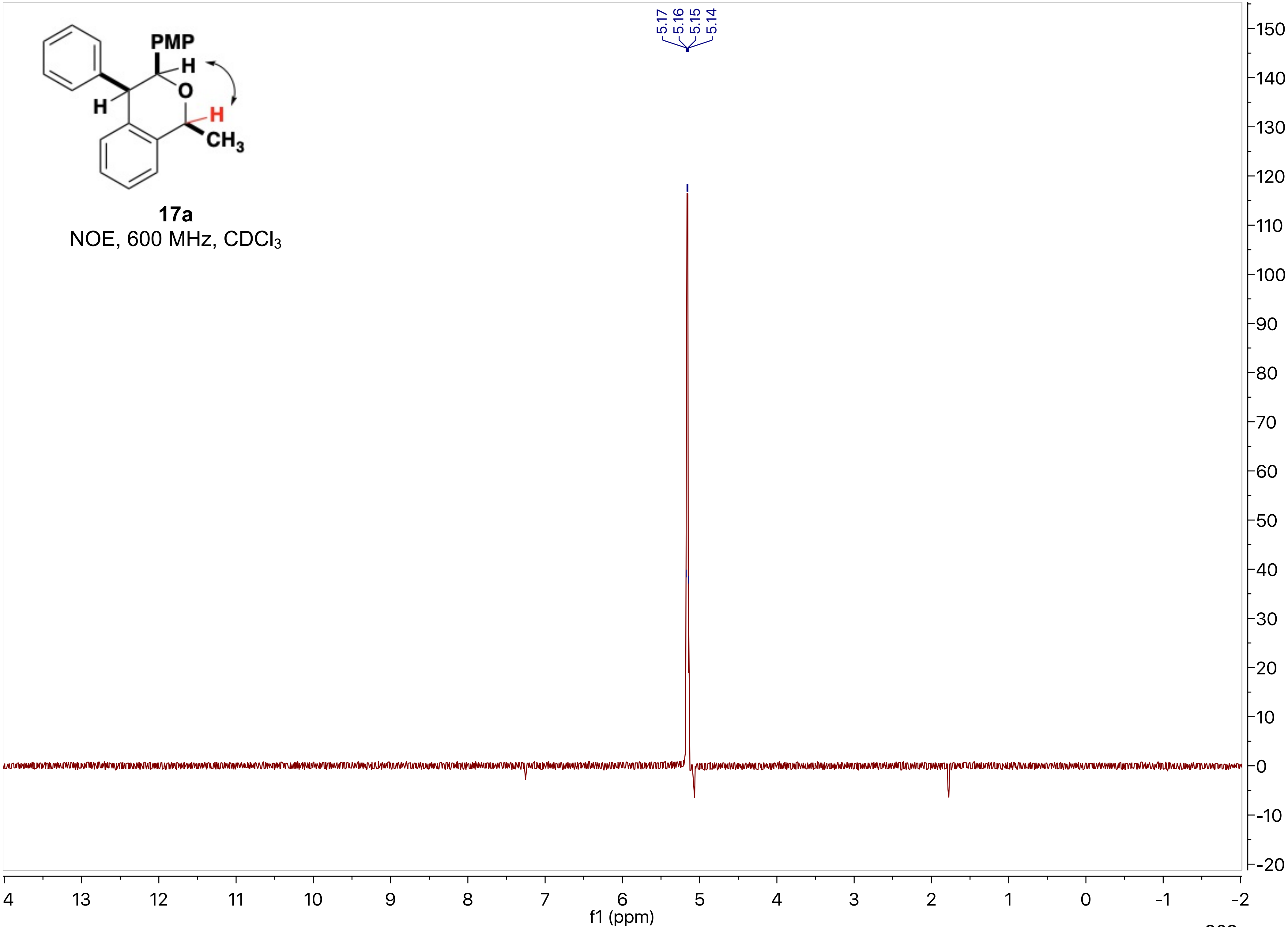
^{13}C, 151 MHz, $CDCl_3$



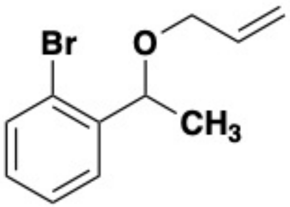


17a
NOE, 600 MHz, $CDCl_3$



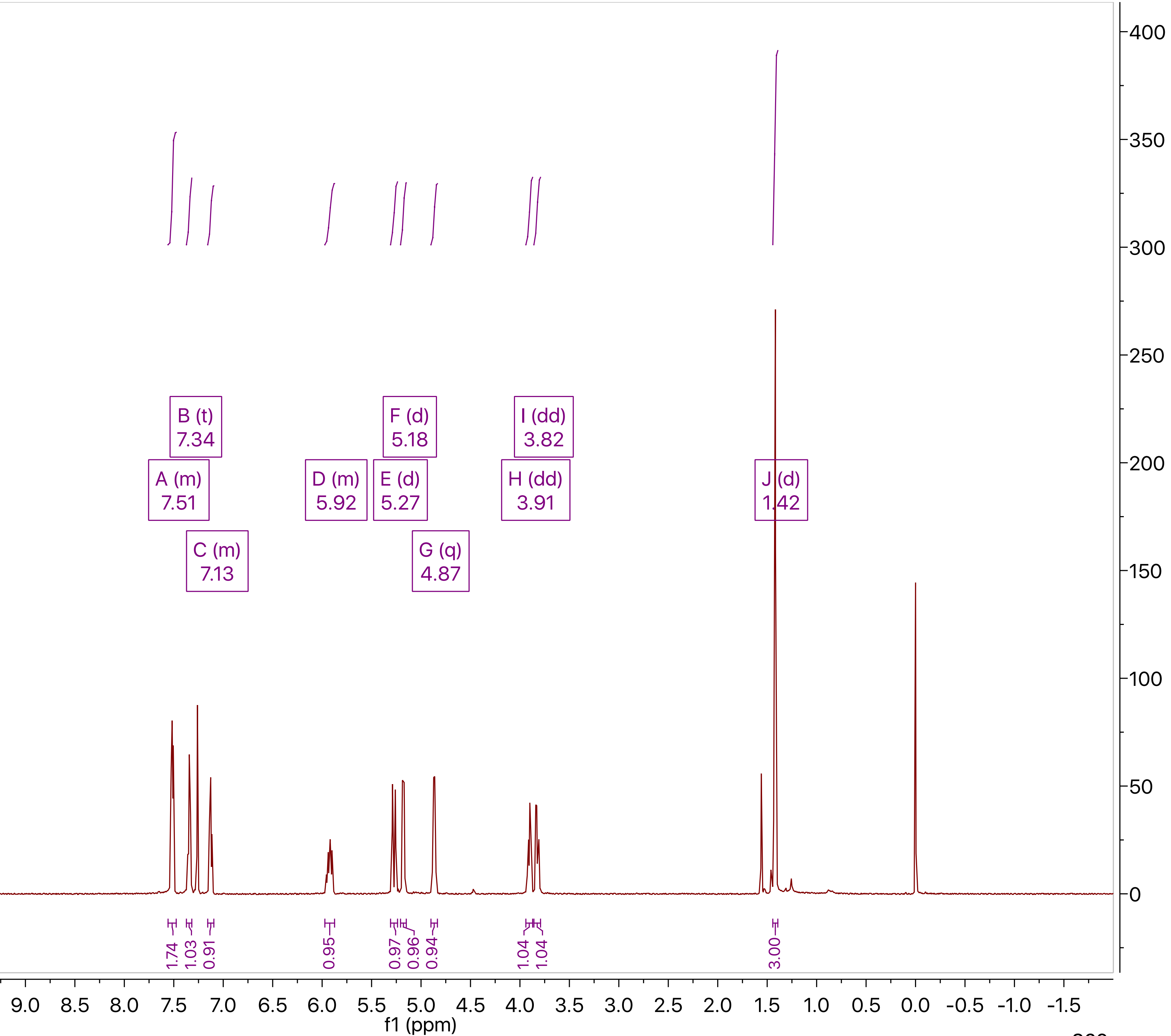


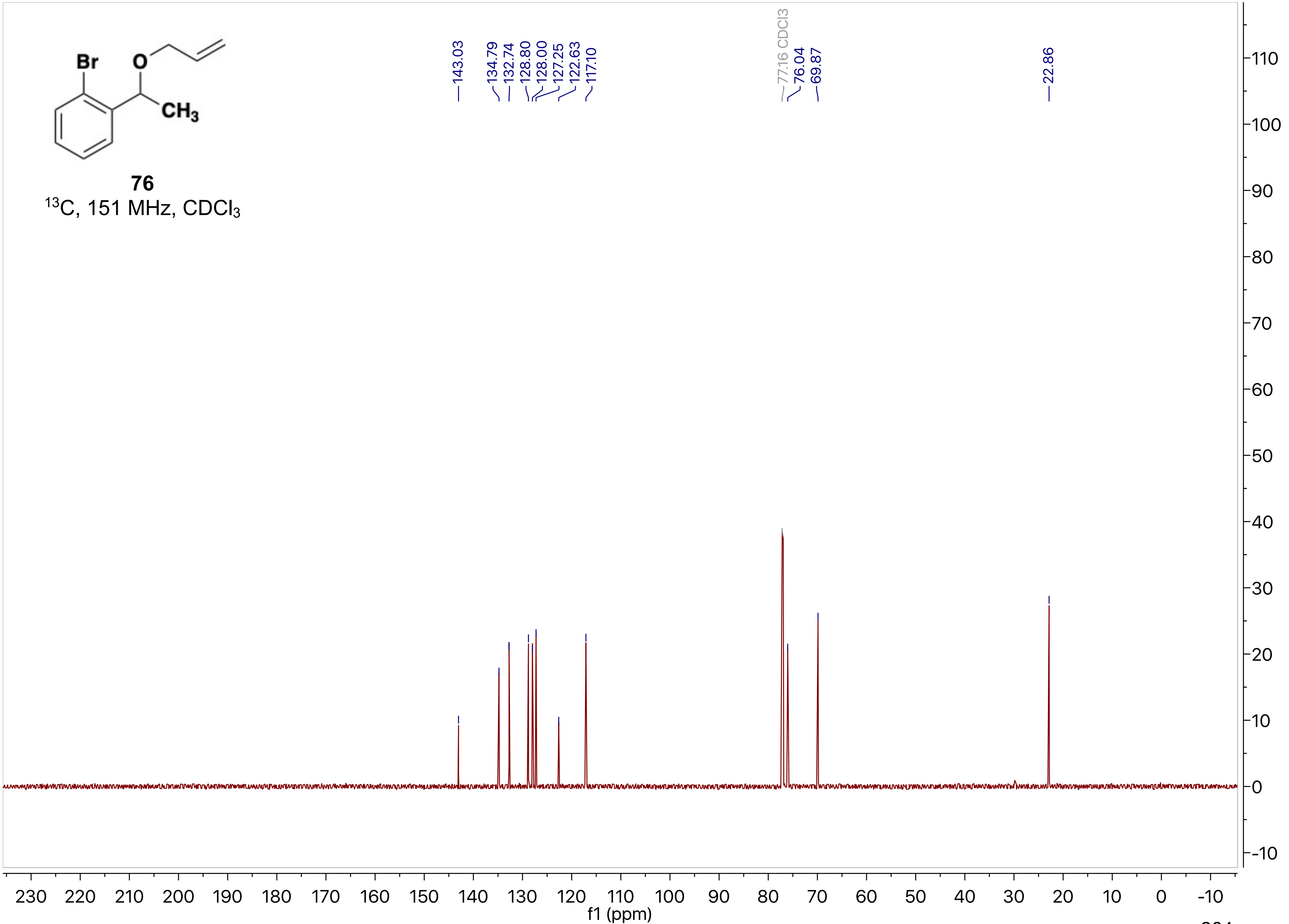
17a
NOE, 600 MHz, $CDCl_3$

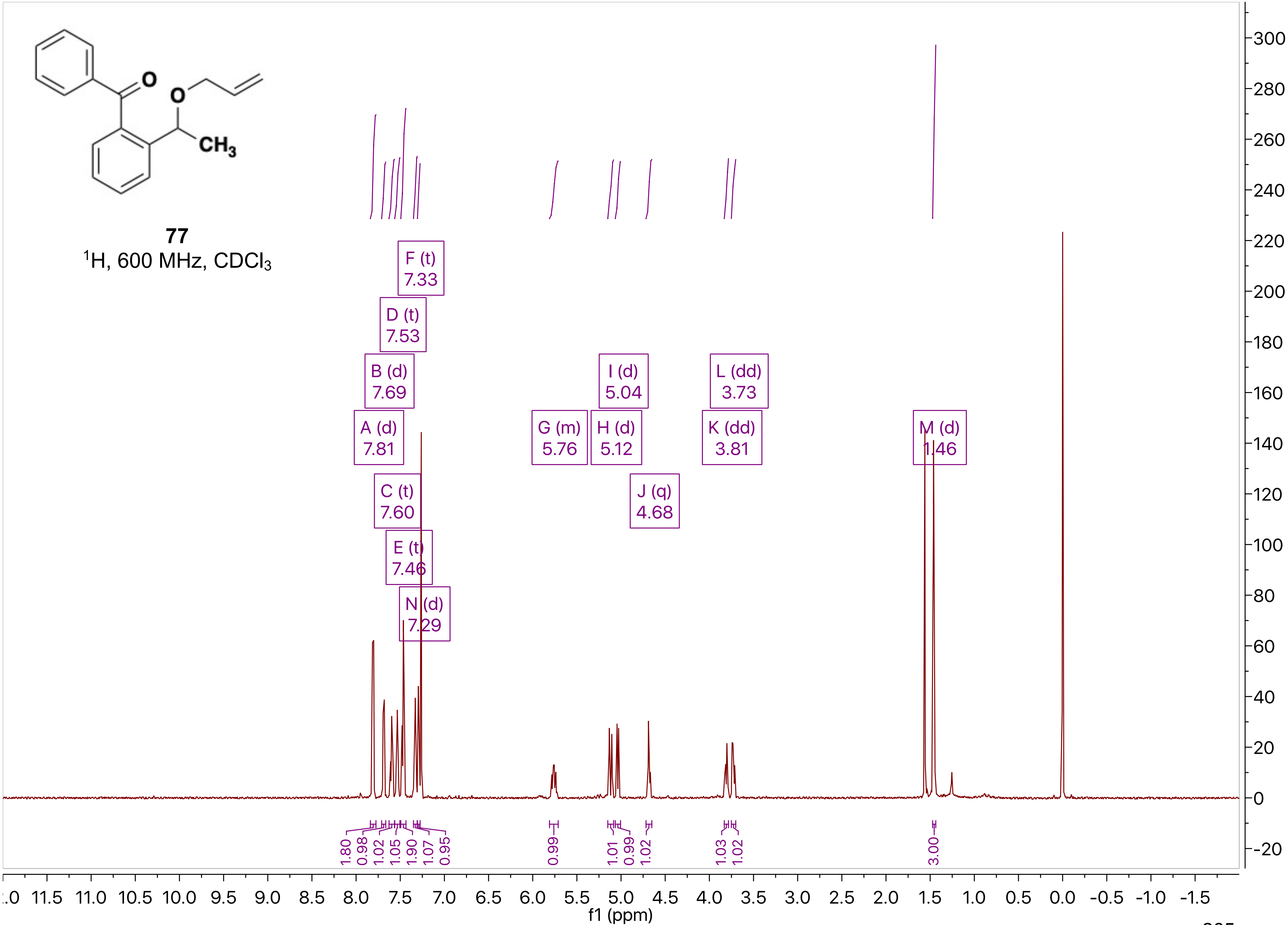


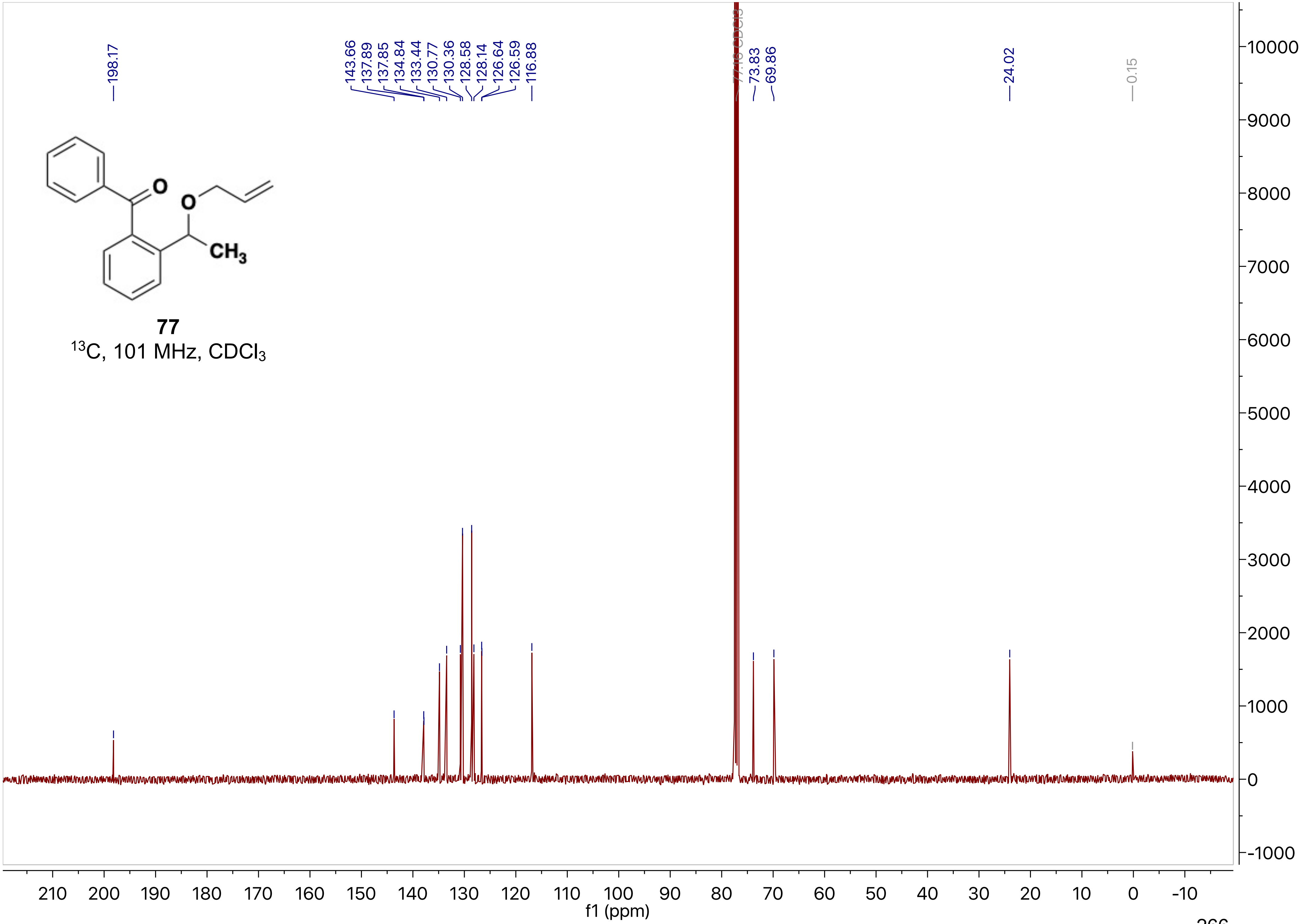
76

^{1}H, 600 MHz, $CDCl_3$



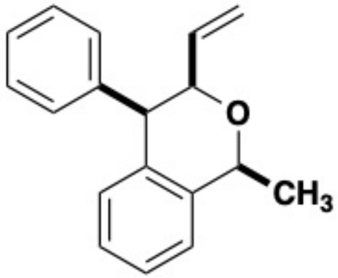

Br
O
CH3
76
13C, 151 MHz, CDCl3
143.03
134.79
132.74
128.80
128.00
127.25
122.63
117.10
77.16 CDCl3
76.04
69.87
22.86
f1 (ppm)


77
¹H, 600 MHz, CDCl₃
F (t) 7.33
D (t) 7.53
B (d) 7.69
A (d) 7.81
C (t) 7.60
E (t) 7.46
N (d) 7.29
G (m) 5.76
I (d) 5.04
H (d) 5.12
J (q) 4.68
L (dd) 3.73
K (dd) 3.81
M (d) 1.46
1.80
0.98
1.02
1.05
1.90
1.07
0.95
0.99
1.01
0.99
1.02
1.03
1.02
3.00
f1 (ppm)



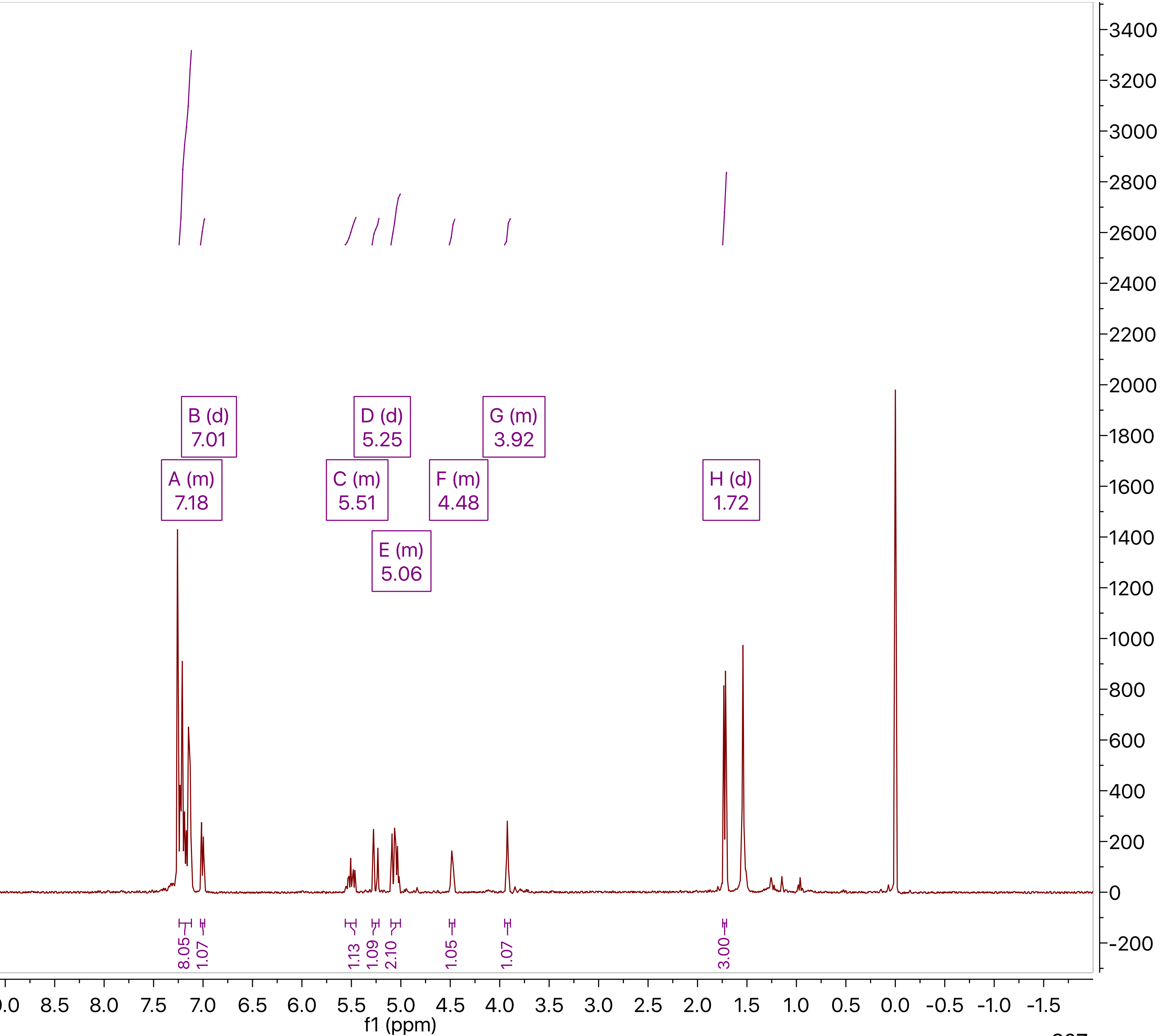
77

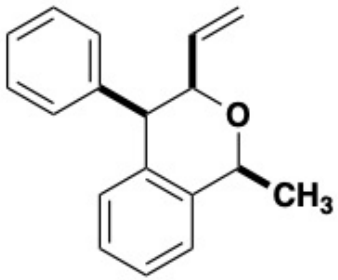
^{13}C, 101 MHz, $CDCl_3$



17b

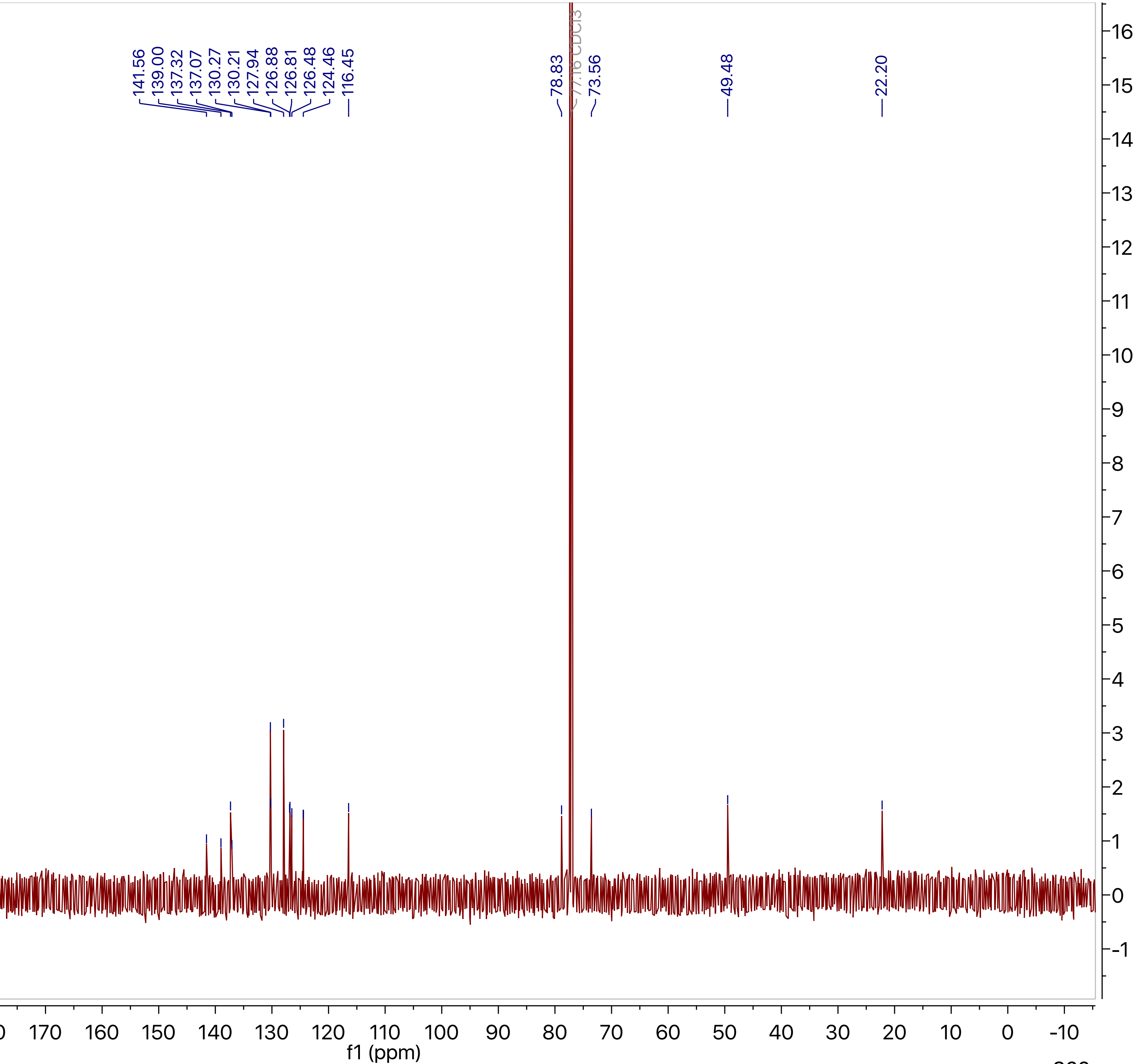
^{1}H, 400 MHz, $CDCl_3$

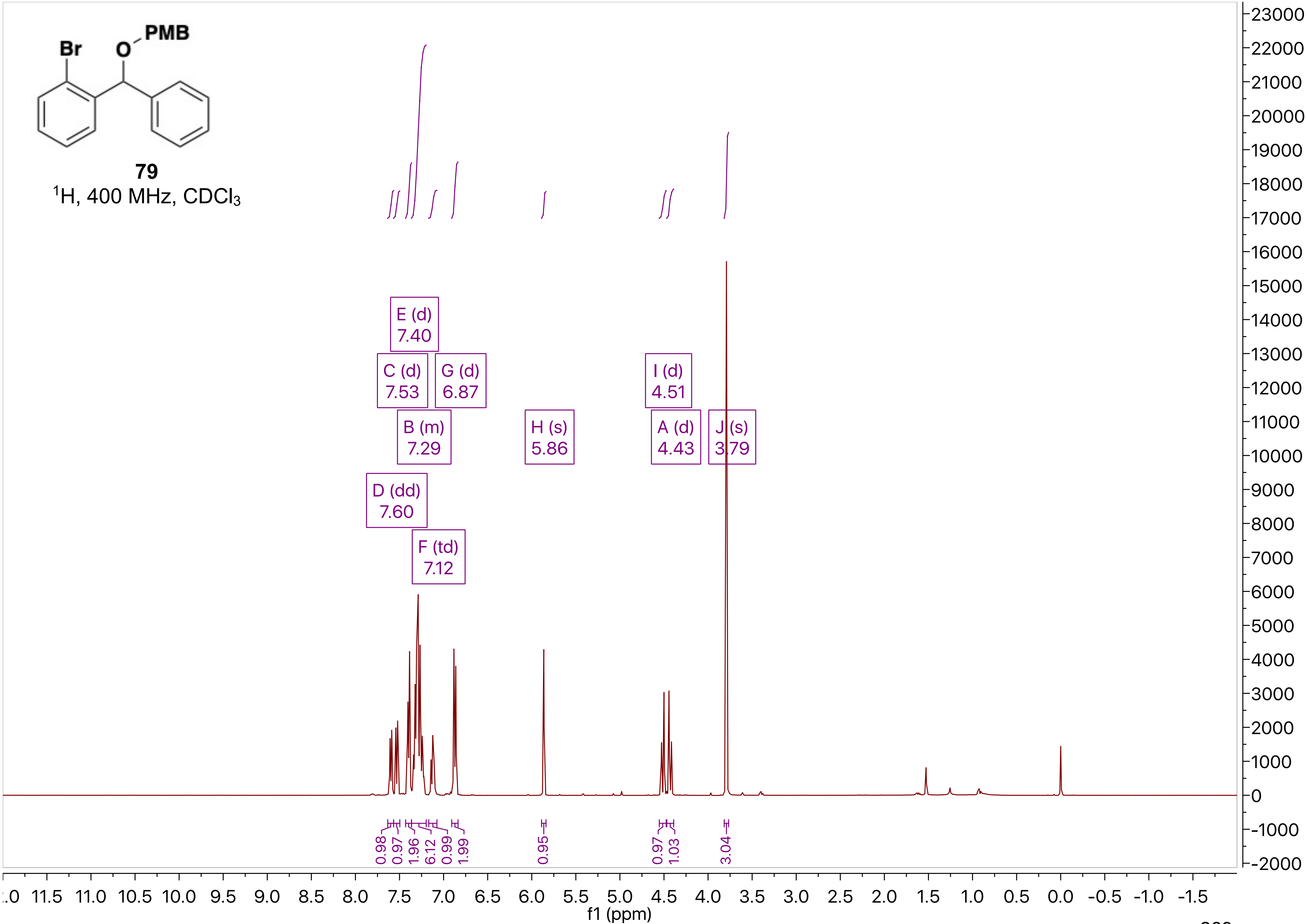




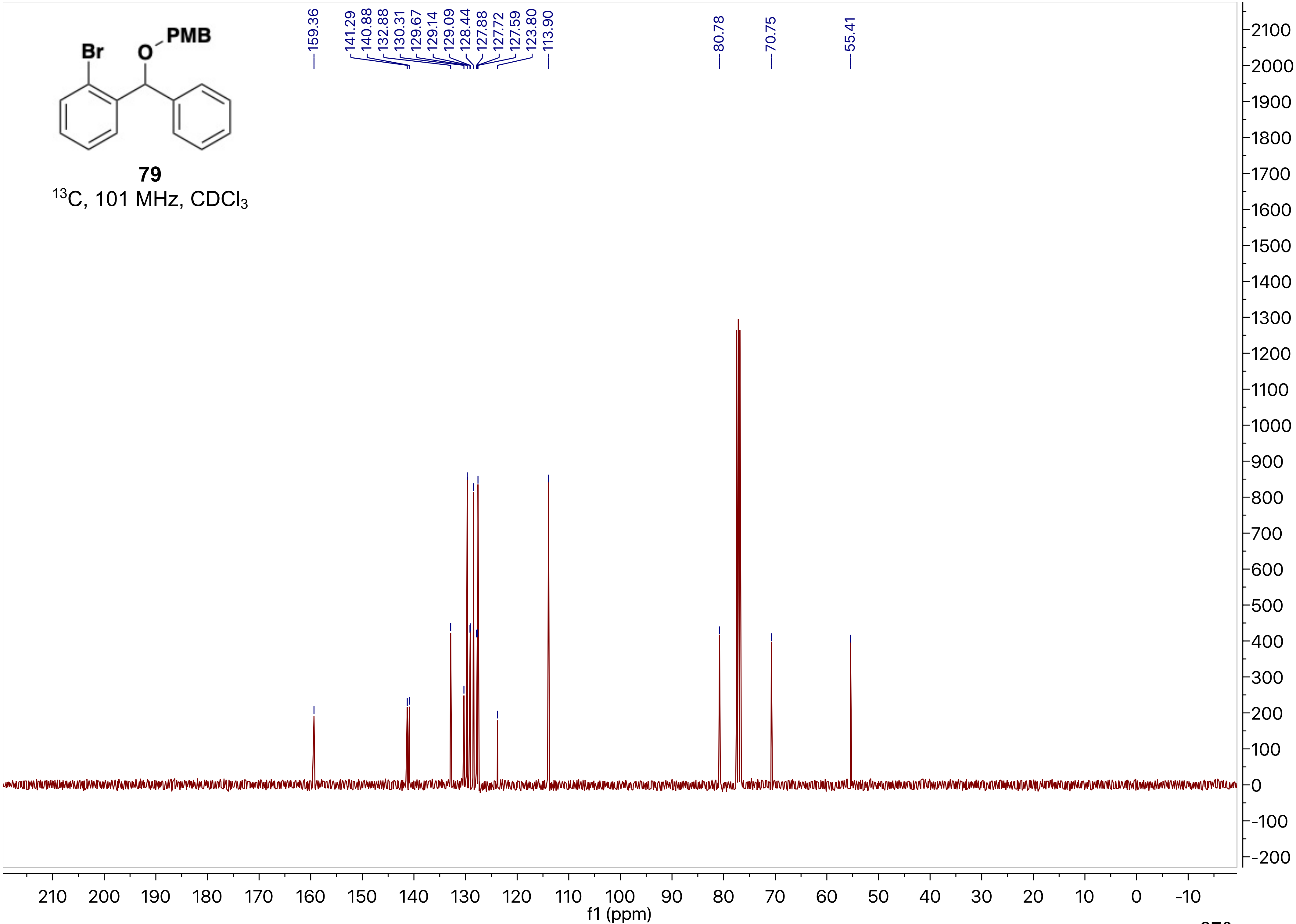
17b

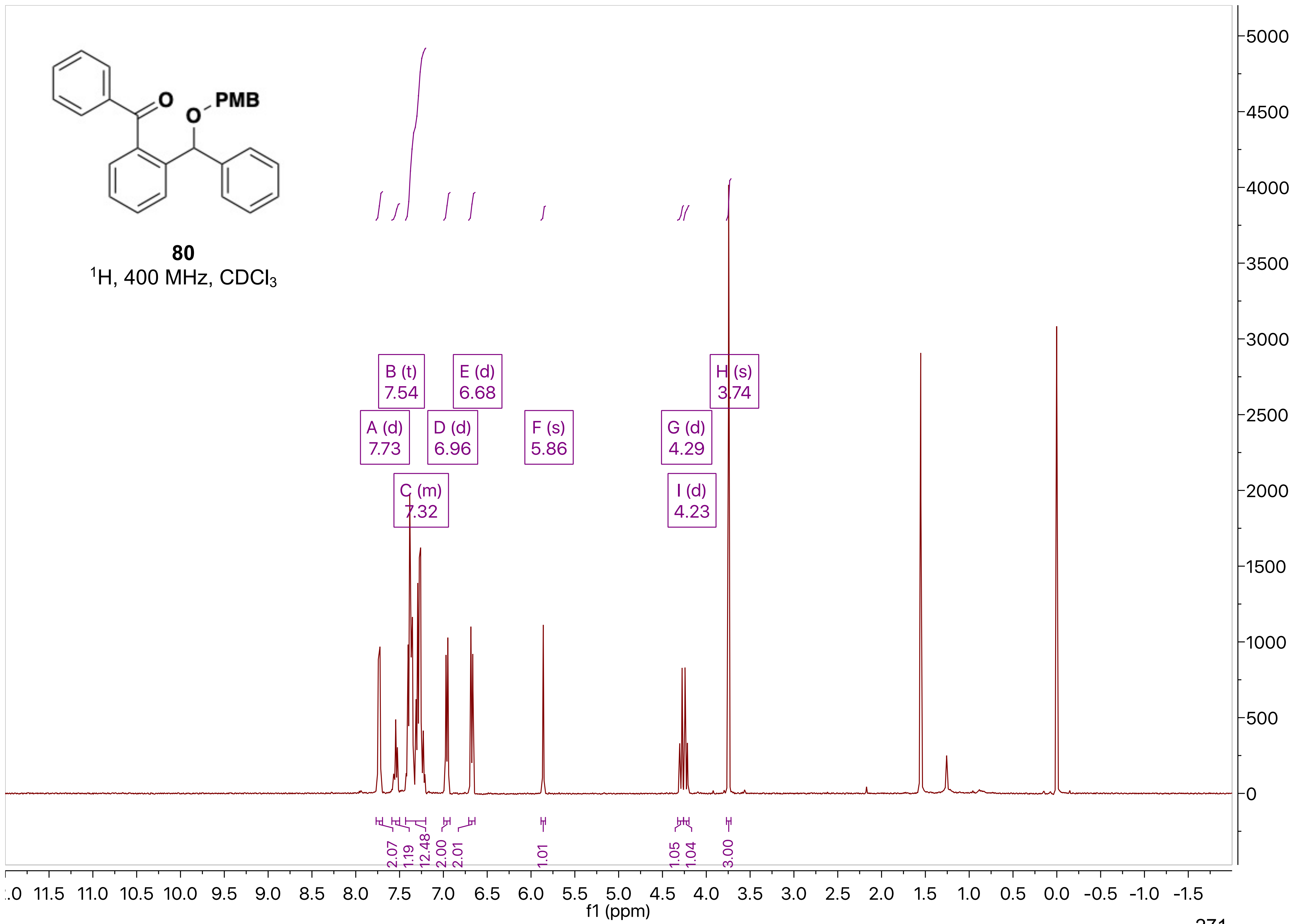
^{13}C, 151 MHz, $CDCl_3$

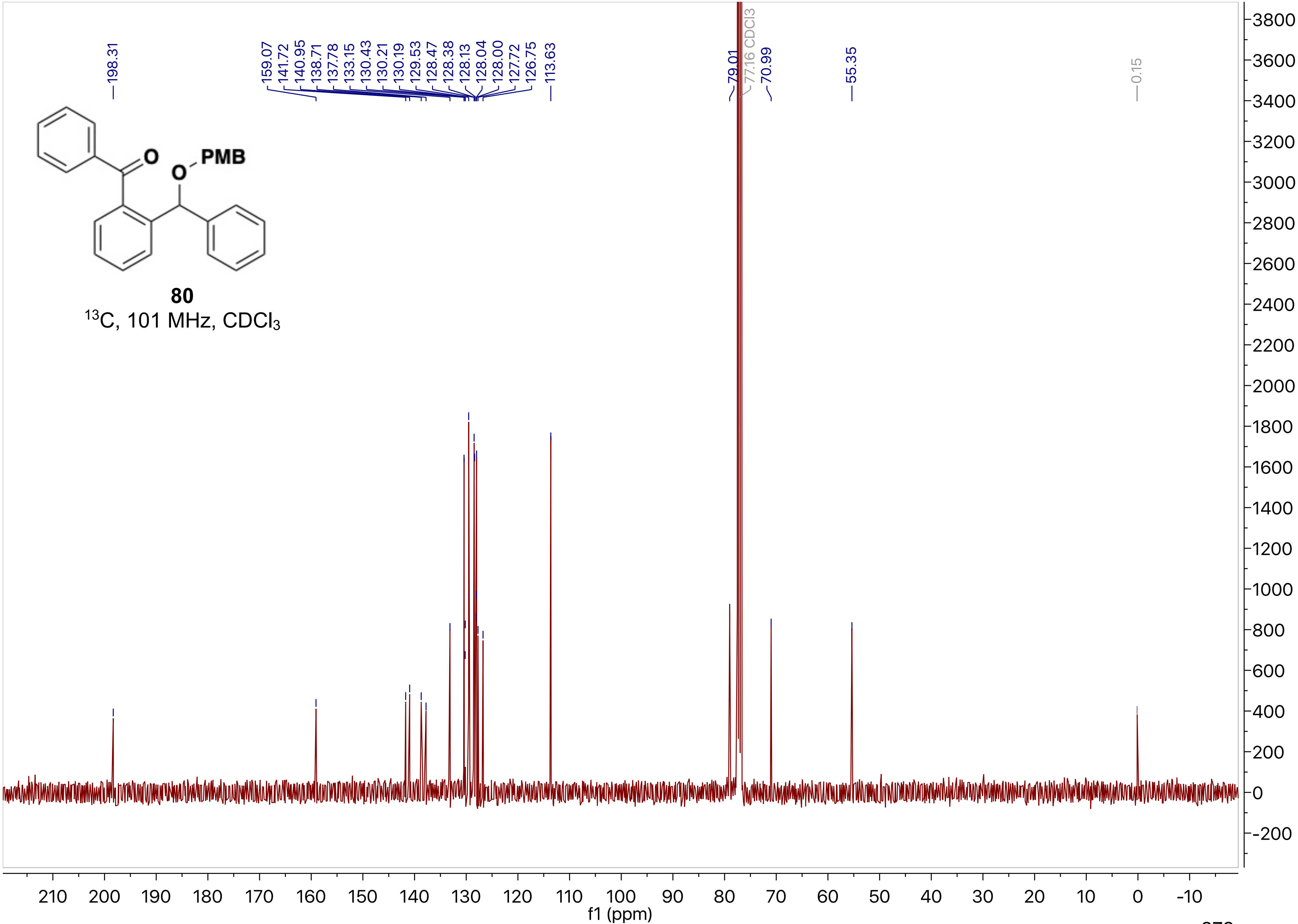


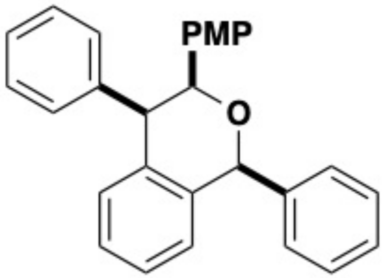


Br
O
PMB
79
¹H, 400 MHz, CDCl₃
E (d) 7.40
C (d) 7.53
G (d) 6.87
B (m) 7.29
D (dd) 7.60
F (td) 7.12
H (s) 5.86
I (d) 4.51
A (d) 4.43
J (s) 3.79
0.98
0.97
1.96
6.12
0.99
1.99
0.95
0.97
1.03
3.04
f1 (ppm)


Br
O
PMB
79
^{13}C, 101 MHz, $CDCl_3$
159.36
141.29
140.88
132.88
130.31
129.67
129.14
129.09
128.44
127.88
127.72
127.59
123.80
113.90
80.78
70.75
55.41
f1 (ppm)

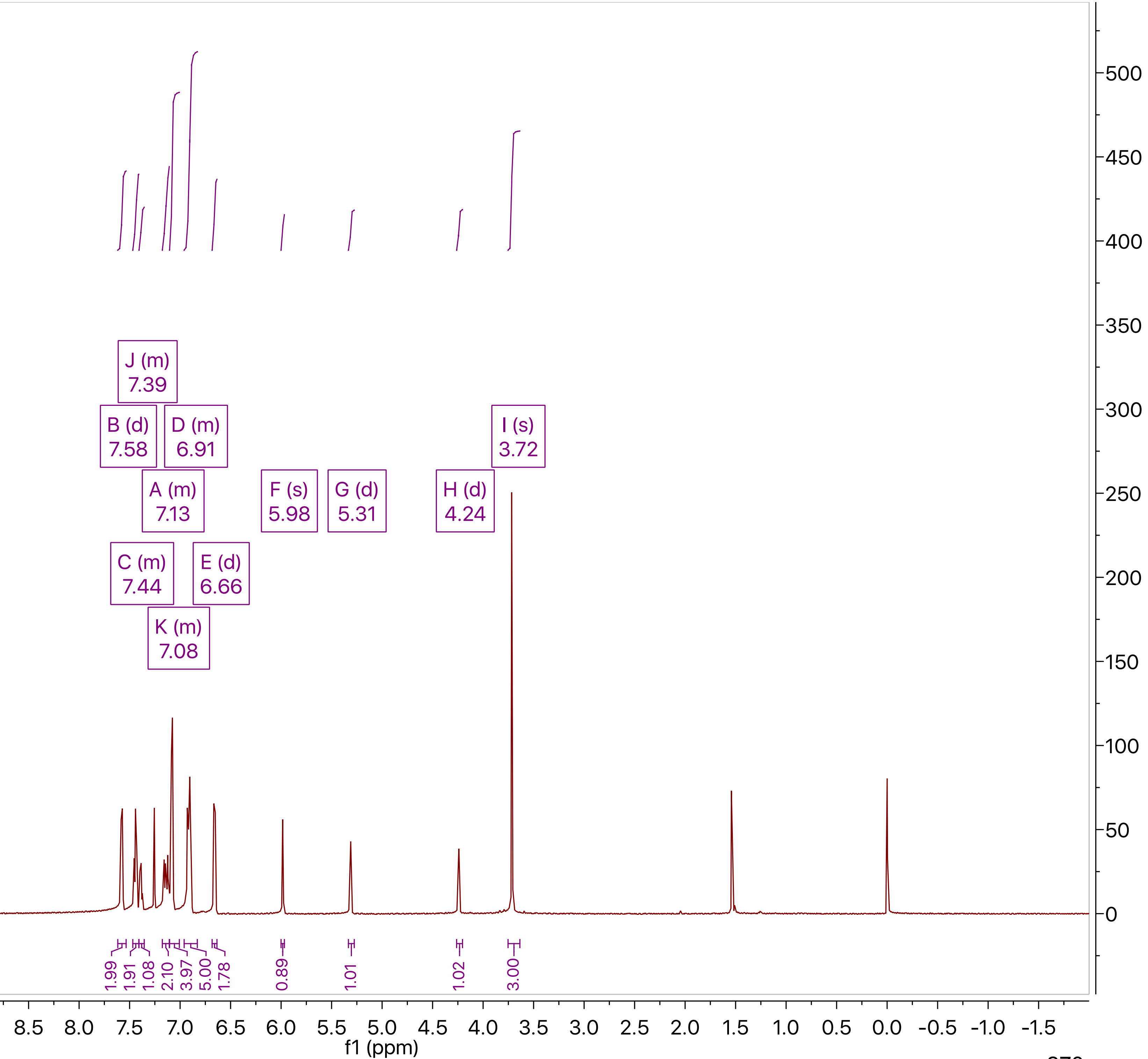

PMB
O
O
80
¹H, 400 MHz, CDCl₃
A (d) 7.73
B (t) 7.54
C (m) 7.32
D (d) 6.96
E (d) 6.68
F (s) 5.86
G (d) 4.29
H (s) 3.74
I (d) 4.23
2.07
1.19
12.48
2.00
2.01
1.01
1.05
1.04
3.00
f1 (ppm)

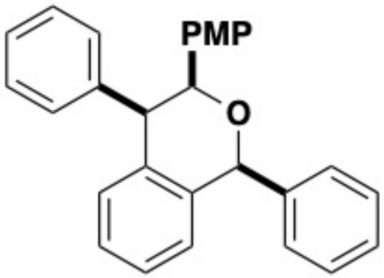

198.31
159.07
141.72
140.95
138.71
137.78
133.15
130.43
130.21
130.19
129.53
128.47
128.38
128.13
128.04
128.00
127.72
126.75
113.63
79.01
77.16 CDCl3
70.99
55.35
0.15
O
O
PMB
80
13C, 101 MHz, CDCl3
f1 (ppm)



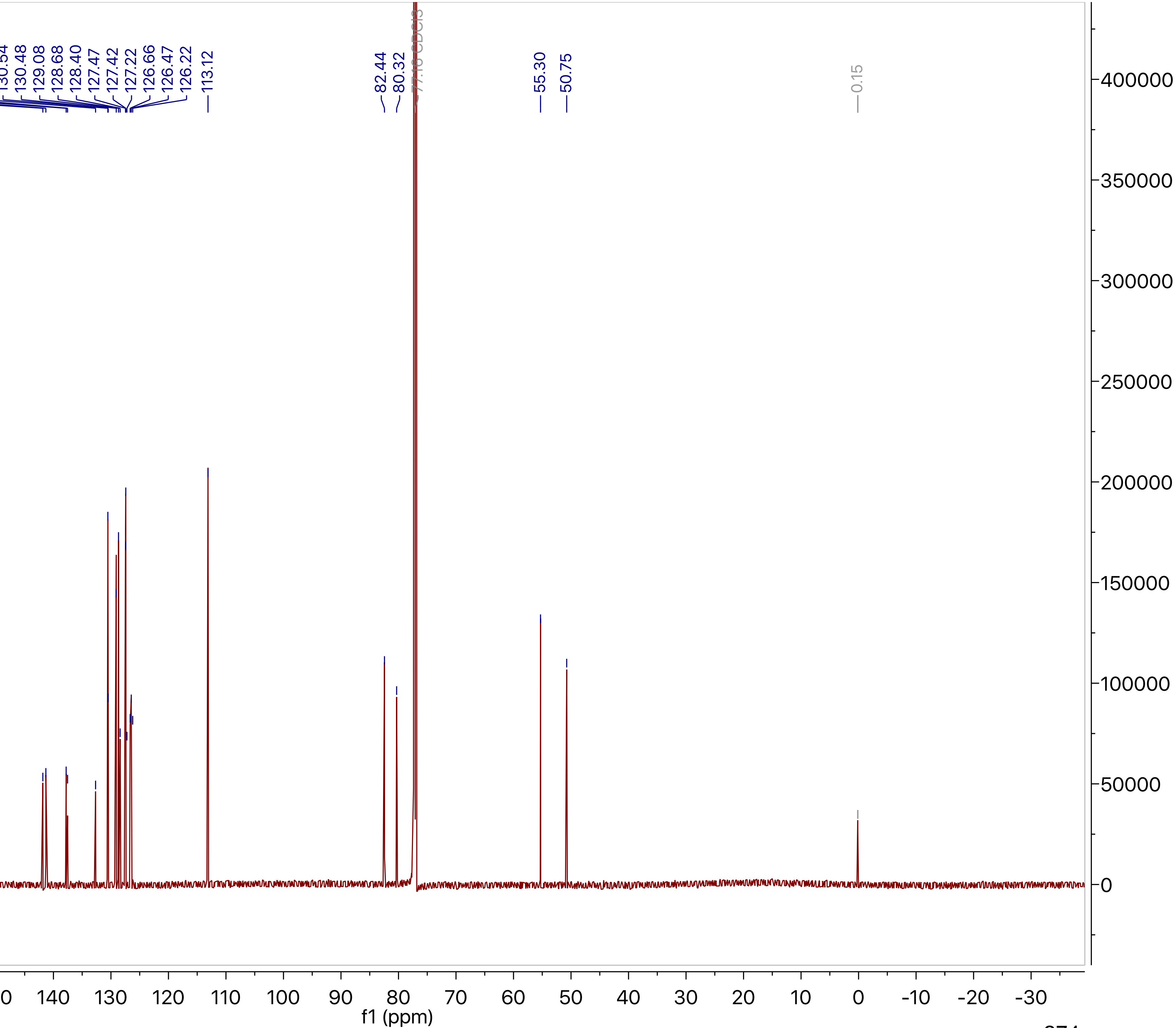
17c

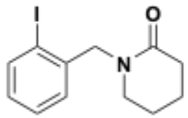
^{1}H, 600 MHz, $CDCl_3$





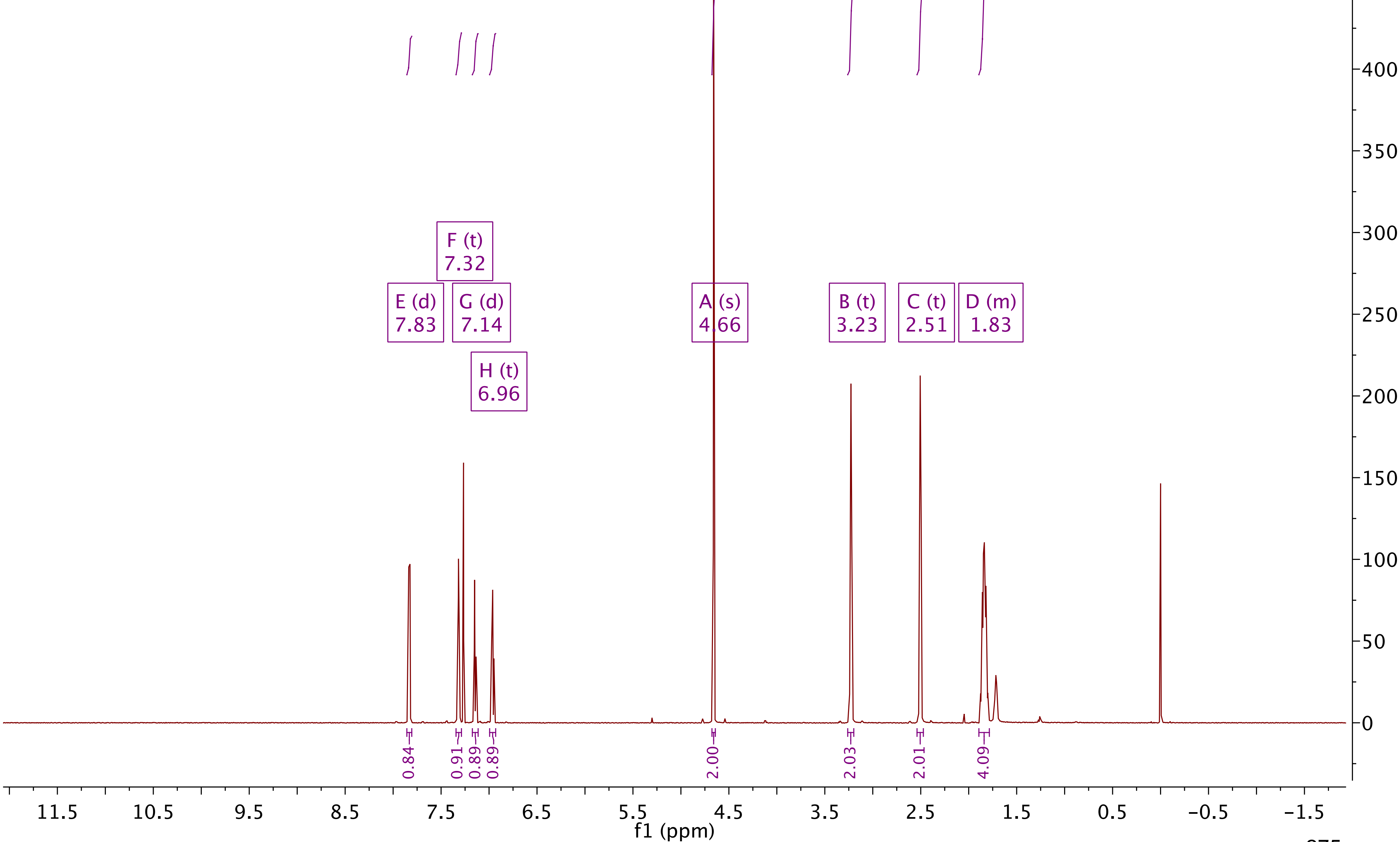
17c
^{13}C, 200 MHz, $CDCl_3$

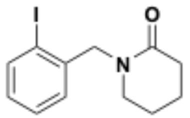




81

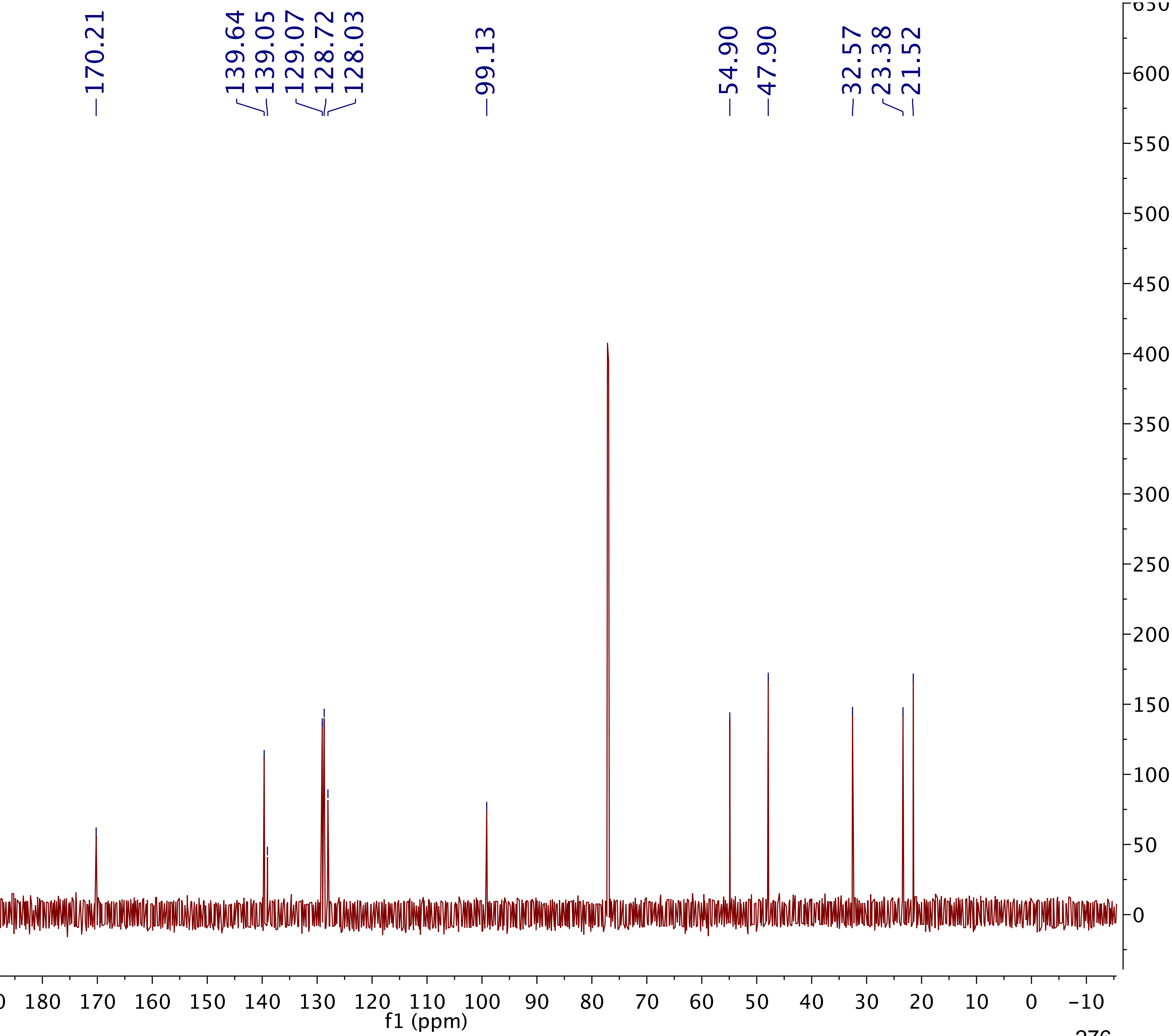
^{1}H, 600 MHz, $CDCl_3$

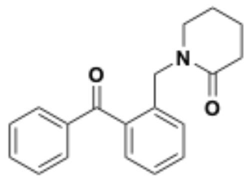




81

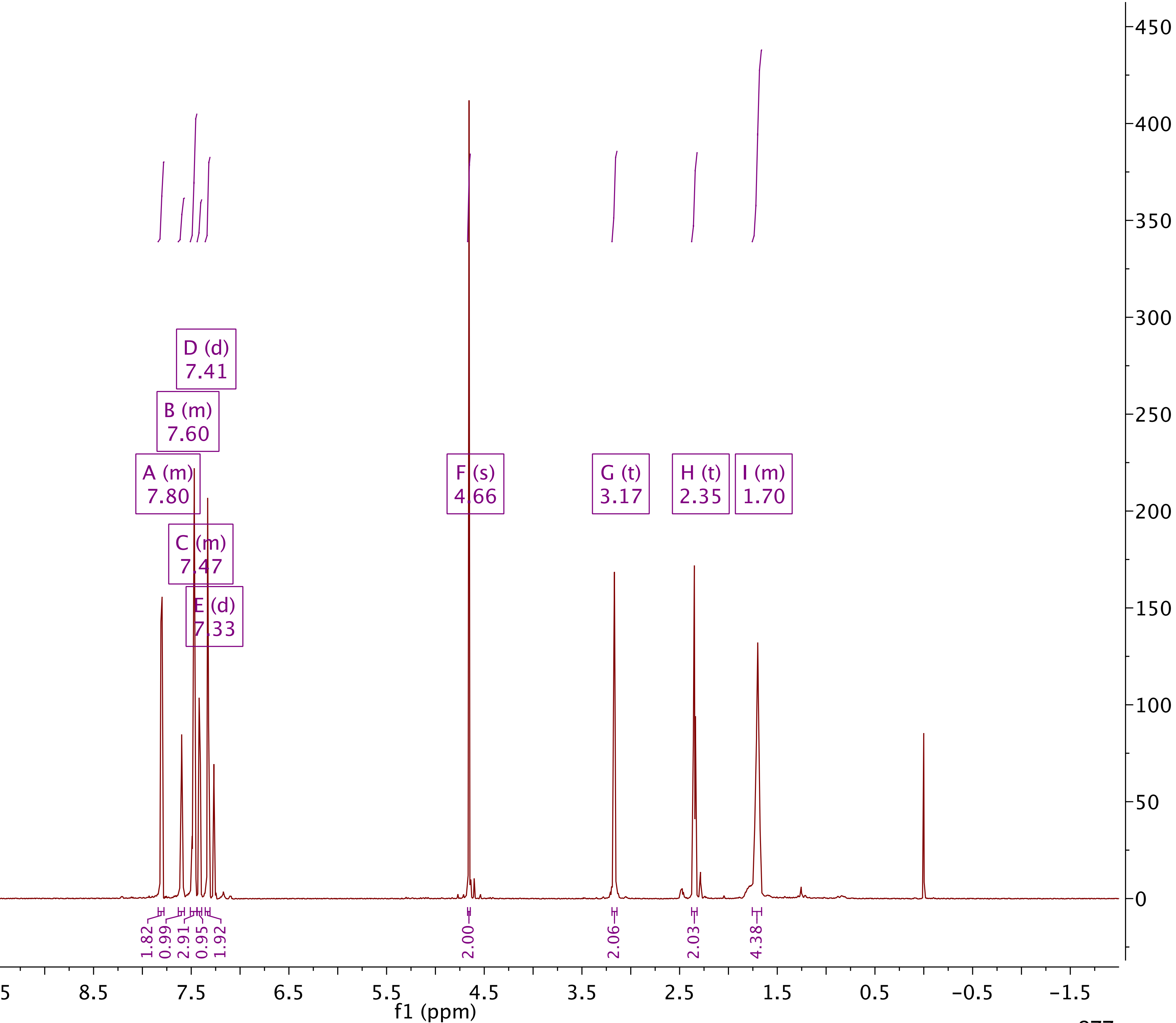
^{13}C, 150 MHz, $CDCl_3$

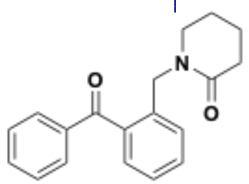




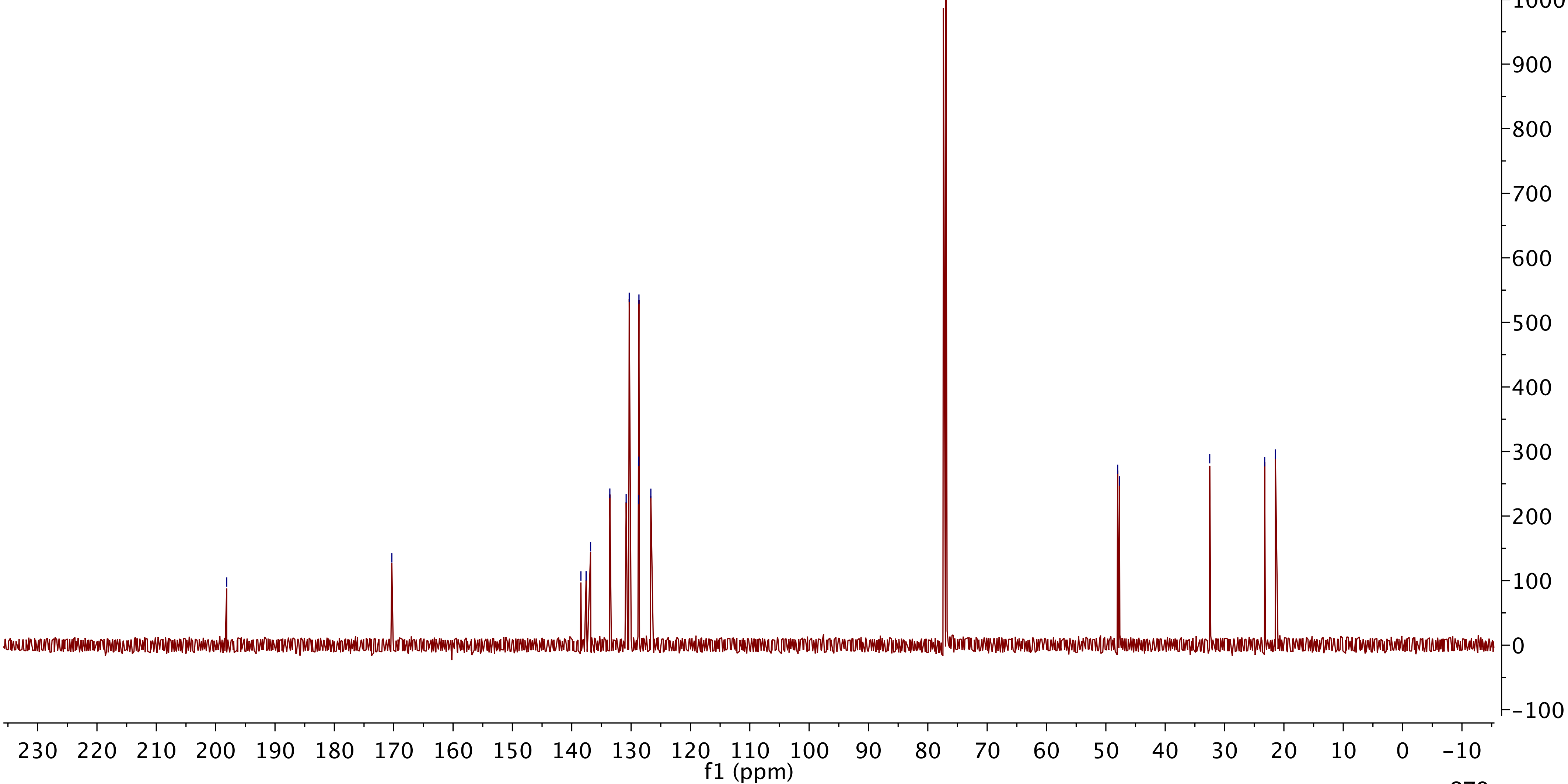
82

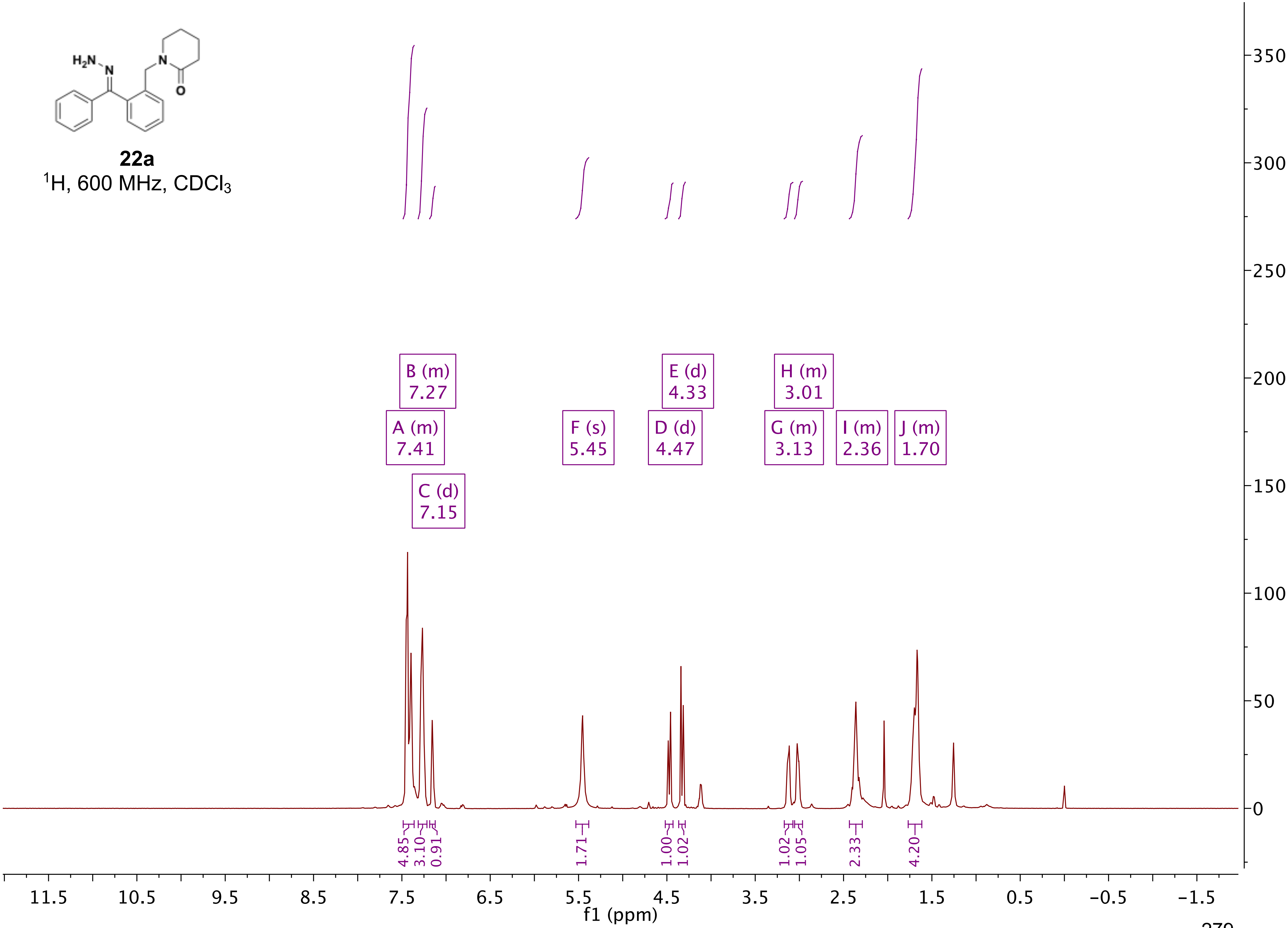
^{1}H, 600 MHz, $CDCl_3$

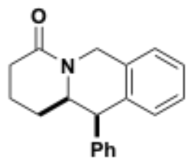




82
^{13}C, 150 MHz, $CDCl_3$

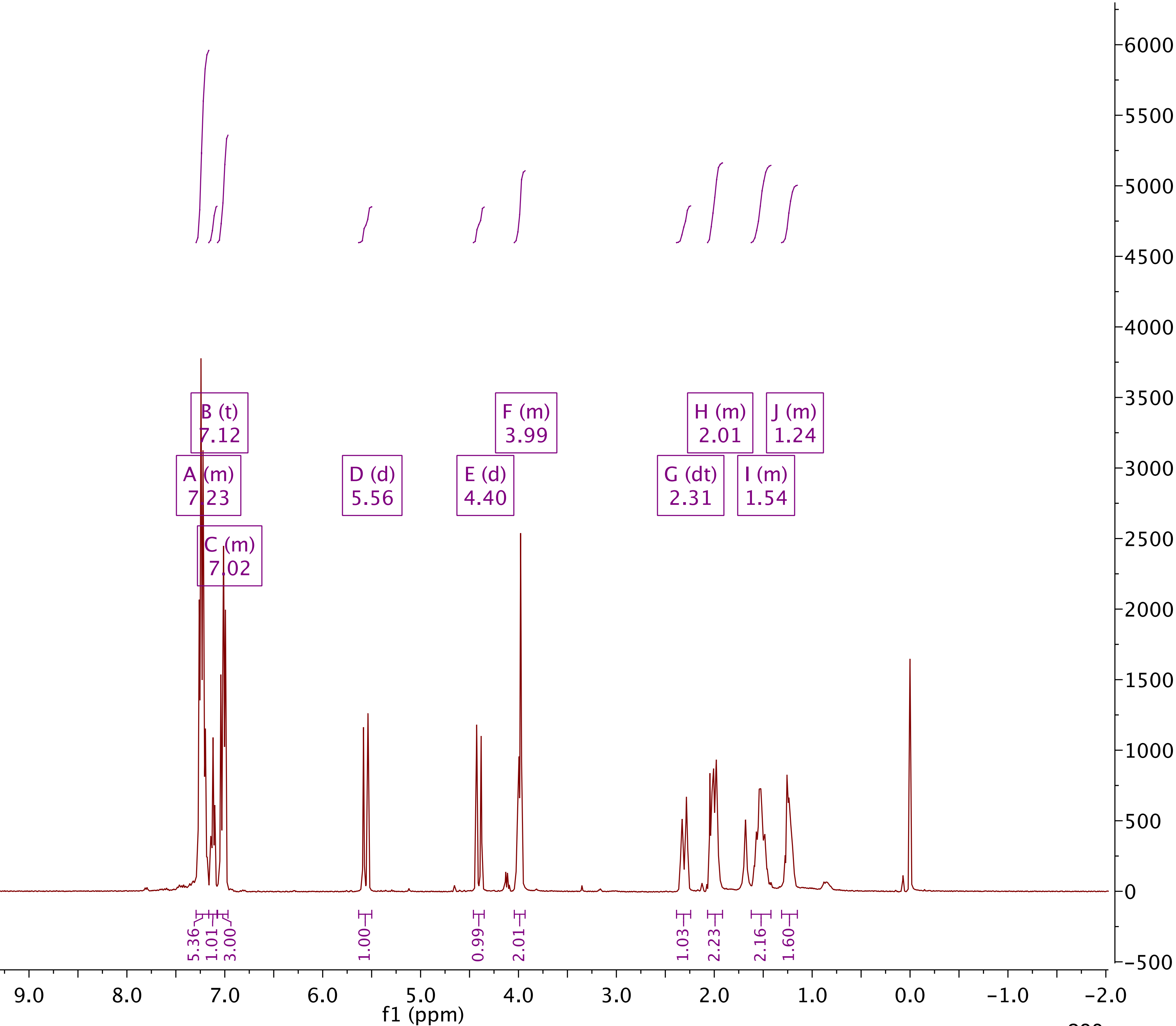


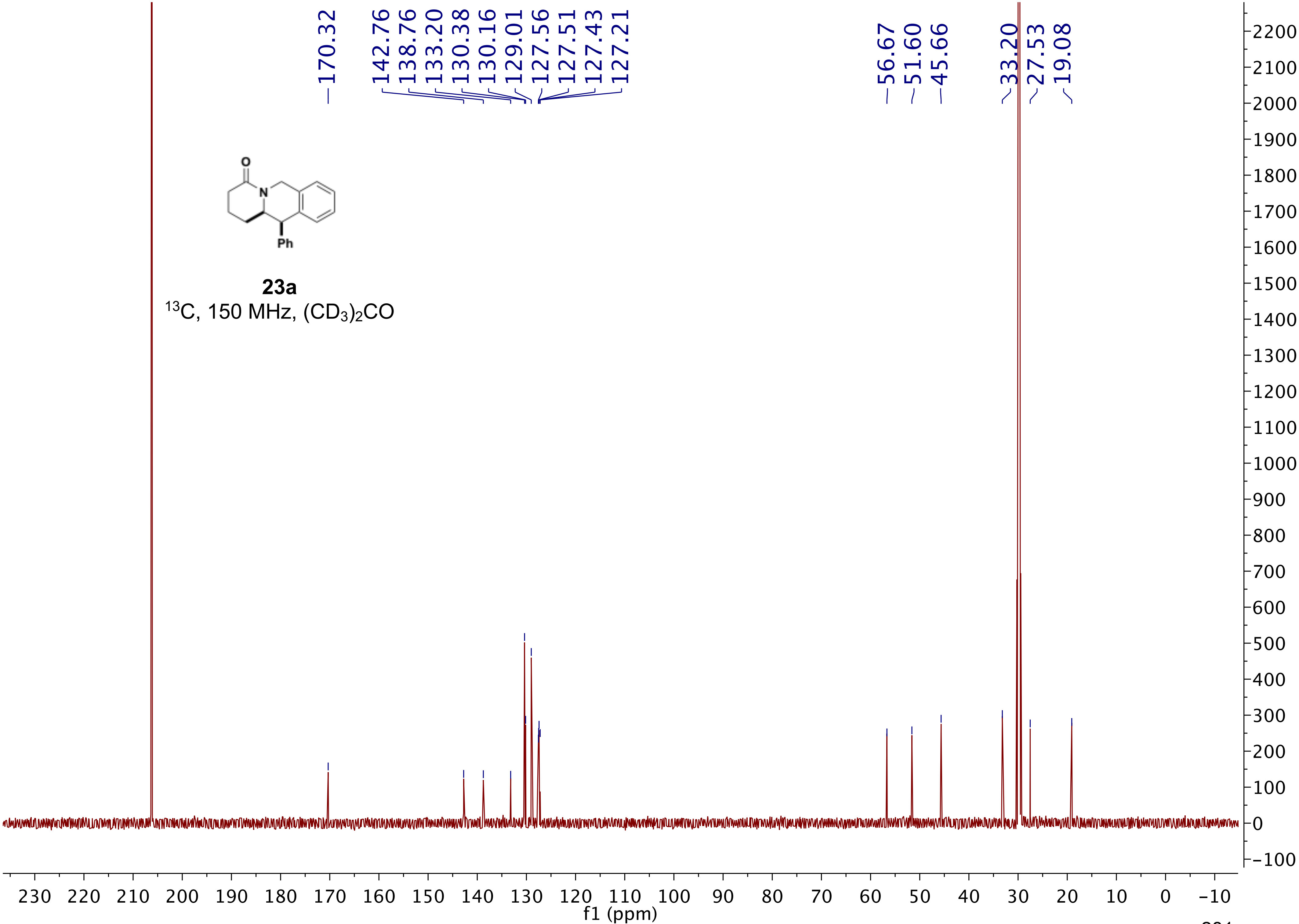

22a
1H, 600 MHz, CDCl3
B (m) 7.27
E (d) 4.33
H (m) 3.01
A (m) 7.41
F (s) 5.45
D (d) 4.47
G (m) 3.13
I (m) 2.36
J (m) 1.70
C (d) 7.15
4.85
3.10
0.91
1.71
1.00
1.02
1.02
1.05
2.33
4.20
f1 (ppm)

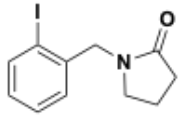


23a

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

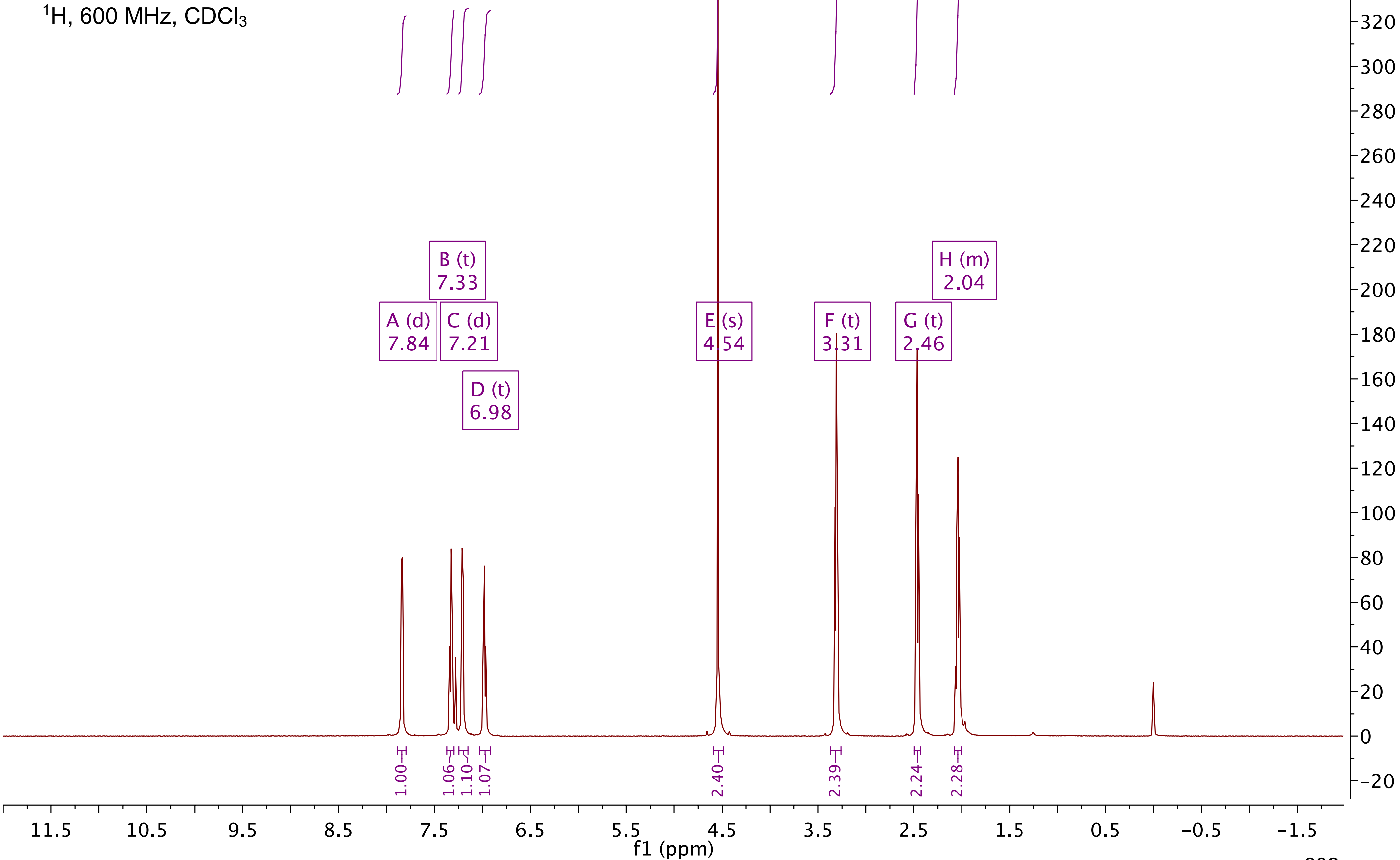


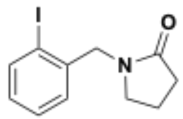

170.32
142.76
138.76
133.20
130.38
130.16
129.01
127.56
127.51
127.43
127.21
56.67
51.60
45.66
33.20
27.53
19.08
Ph
O
N
23a
^{13}C, 150 MHz, $(CD_3)_2CO$
f1 (ppm)



83

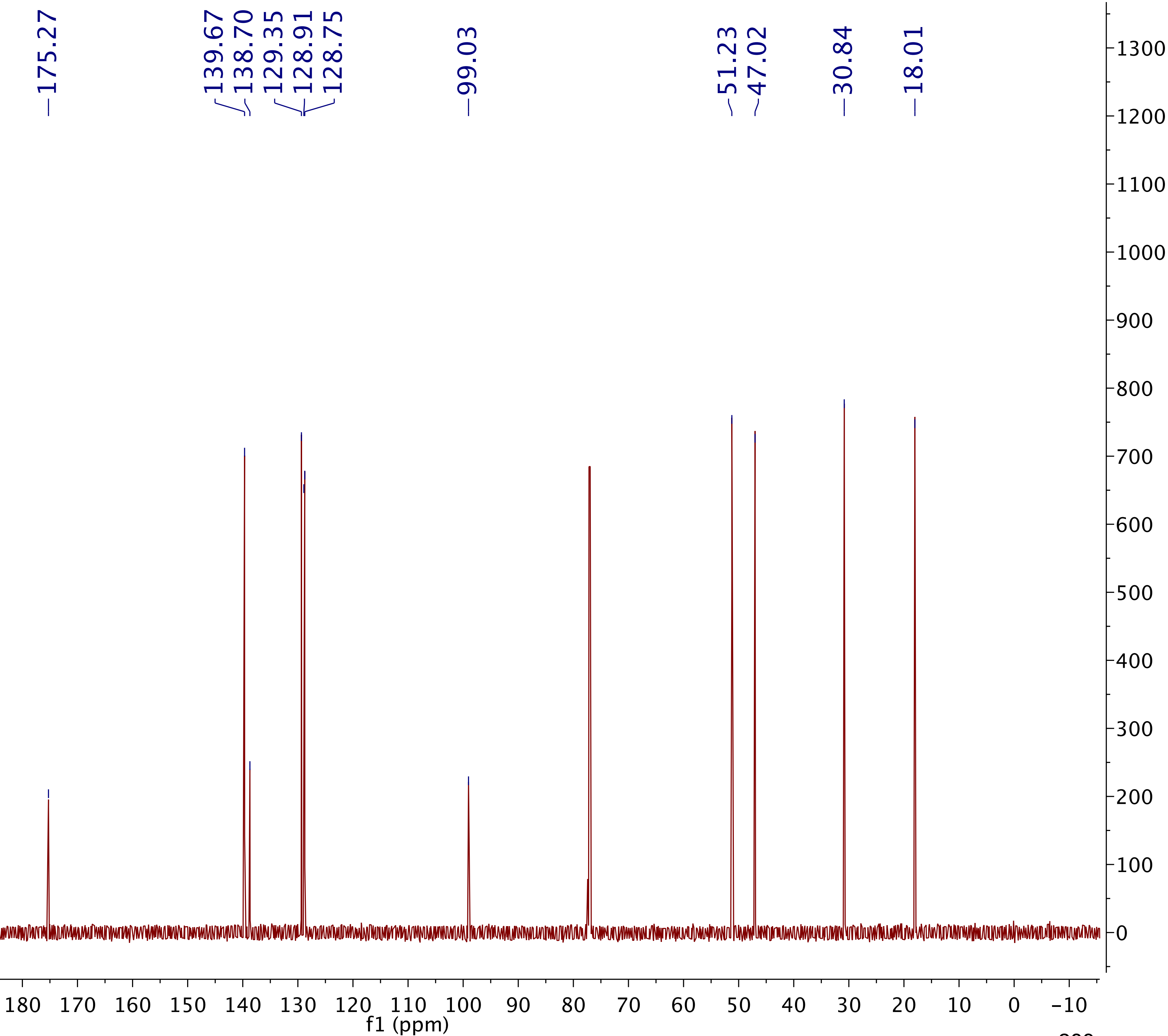
^{1}H, 600 MHz, $CDCl_3$

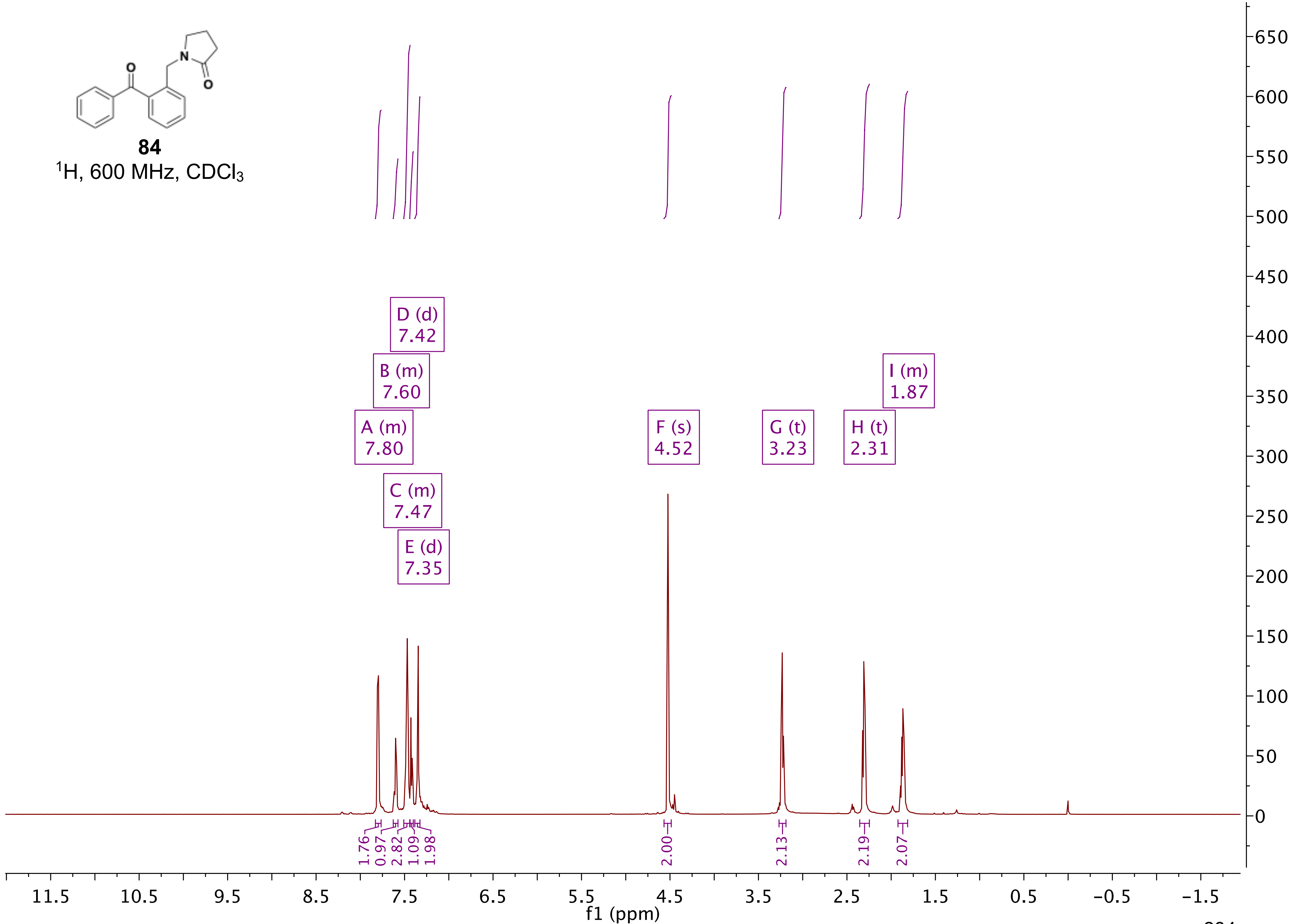


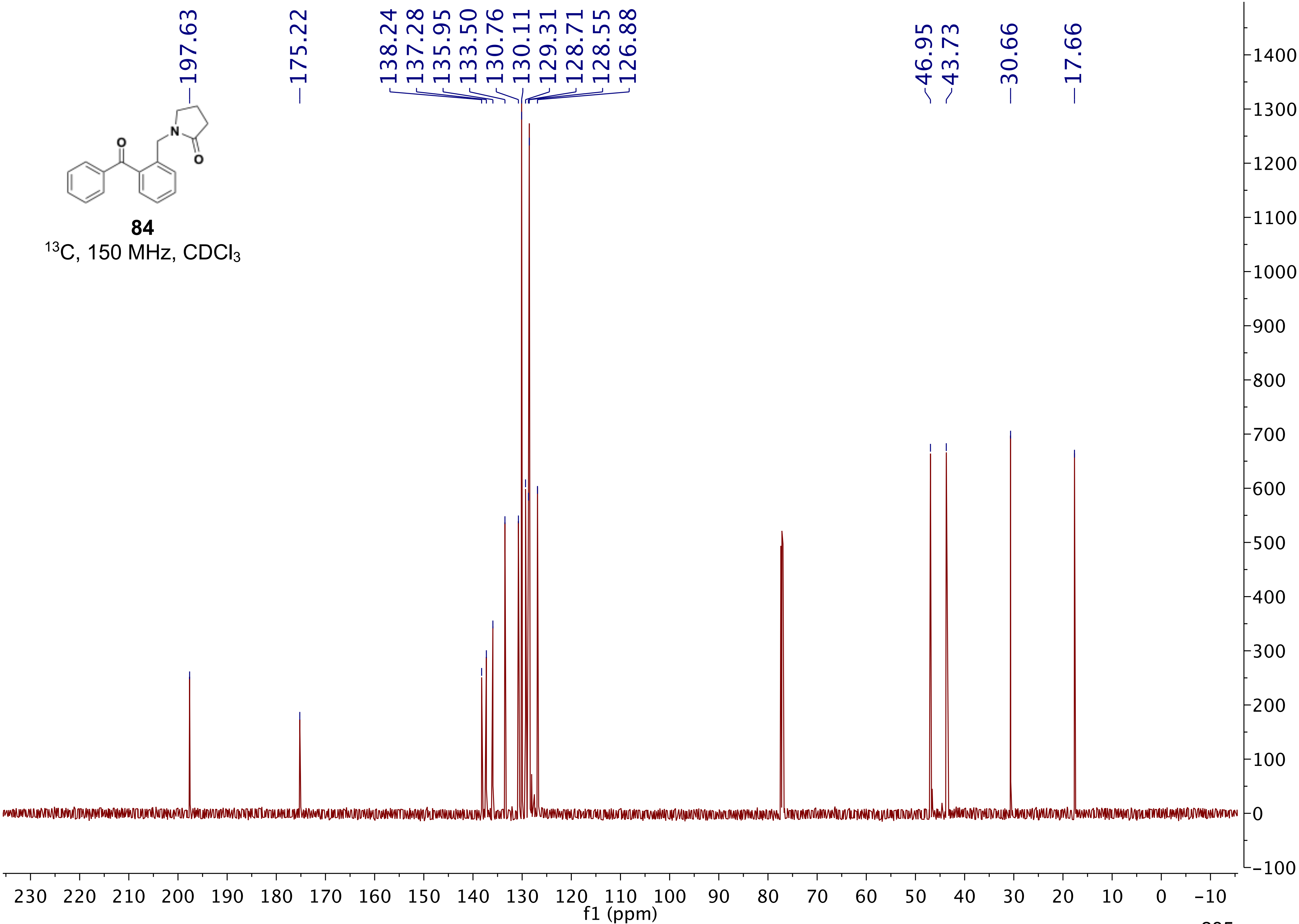


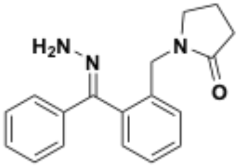
83

^{13}C, 150 MHz, $CDCl_3$



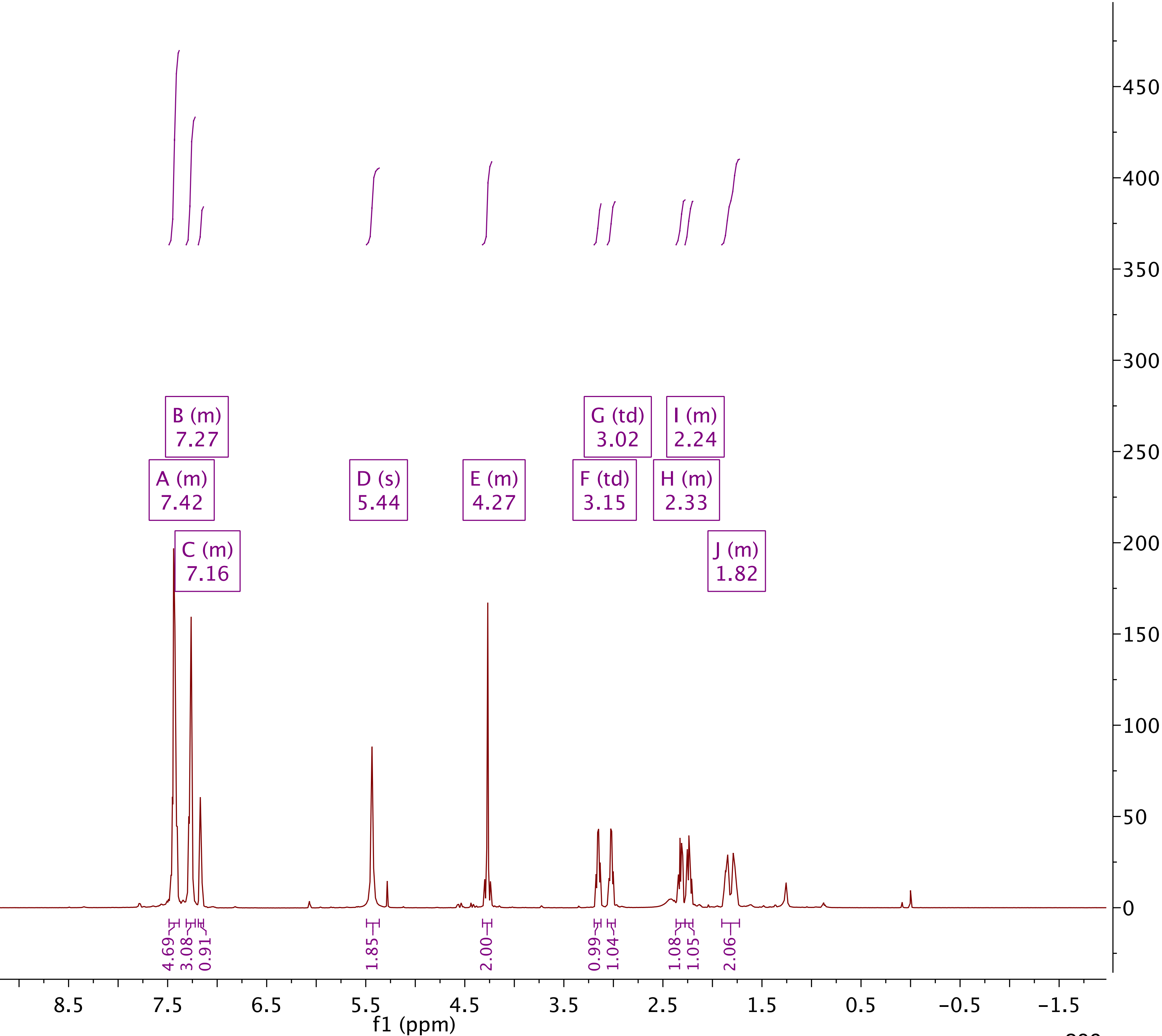

84
1H, 600 MHz, CDCl3
D (d) 7.42
B (m) 7.60
A (m) 7.80
C (m) 7.47
E (d) 7.35
F (s) 4.52
G (t) 3.23
H (t) 2.31
I (m) 1.87
1.76
0.97
2.82
1.09
1.98
2.00
2.13
2.19
2.07
f1 (ppm)

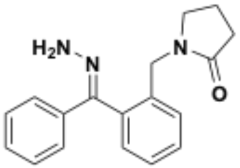

197.63
175.22
138.24
137.28
135.95
133.50
130.76
130.11
129.31
128.71
128.55
126.88
46.95
43.73
30.66
17.66
N
O
O
84
^{13}C, 150 MHz, $CDCl_3$
f1 (ppm)



22b

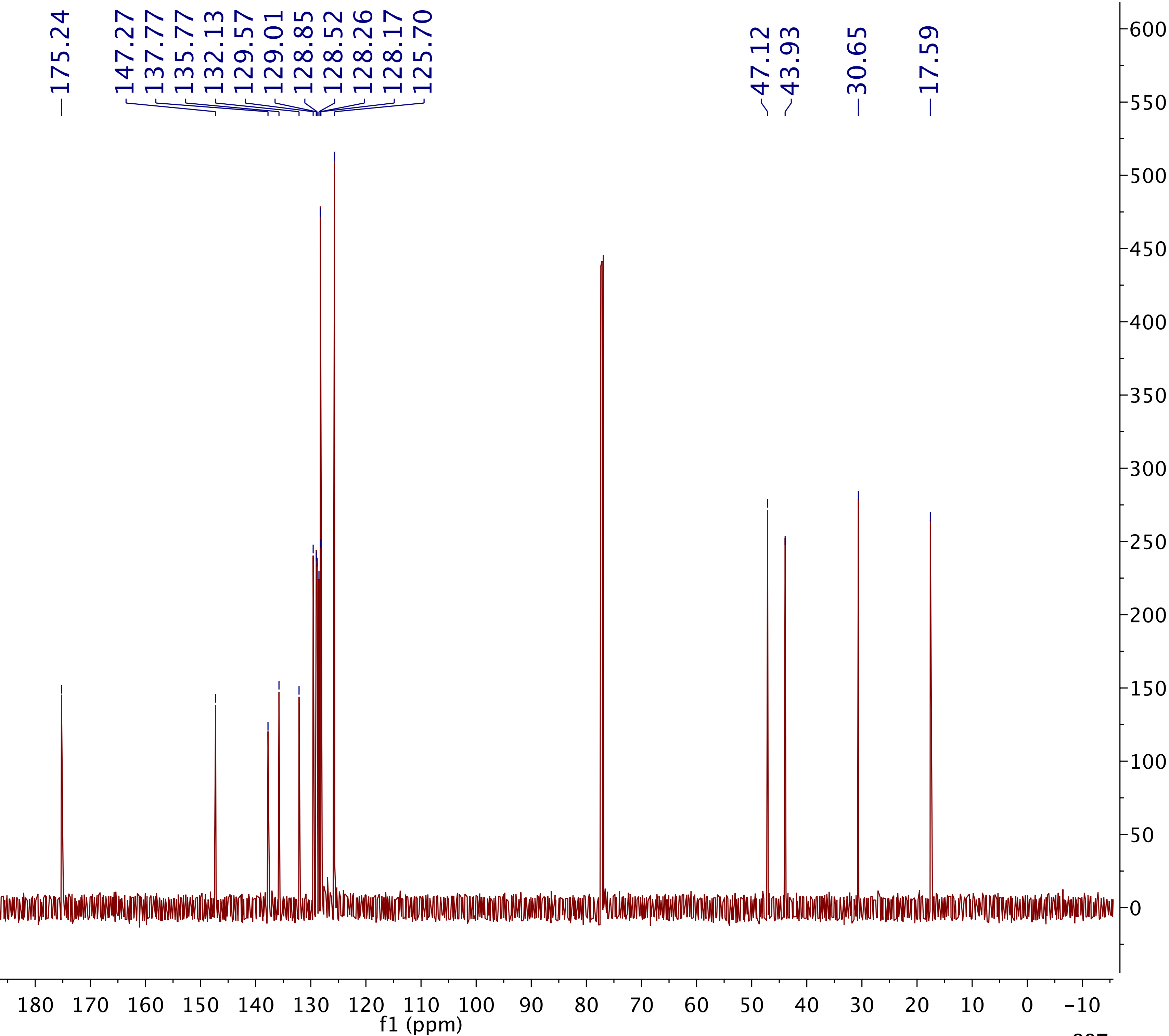
^{1}H, 600 MHz, $CDCl_3$

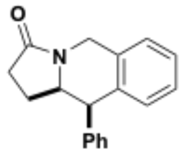




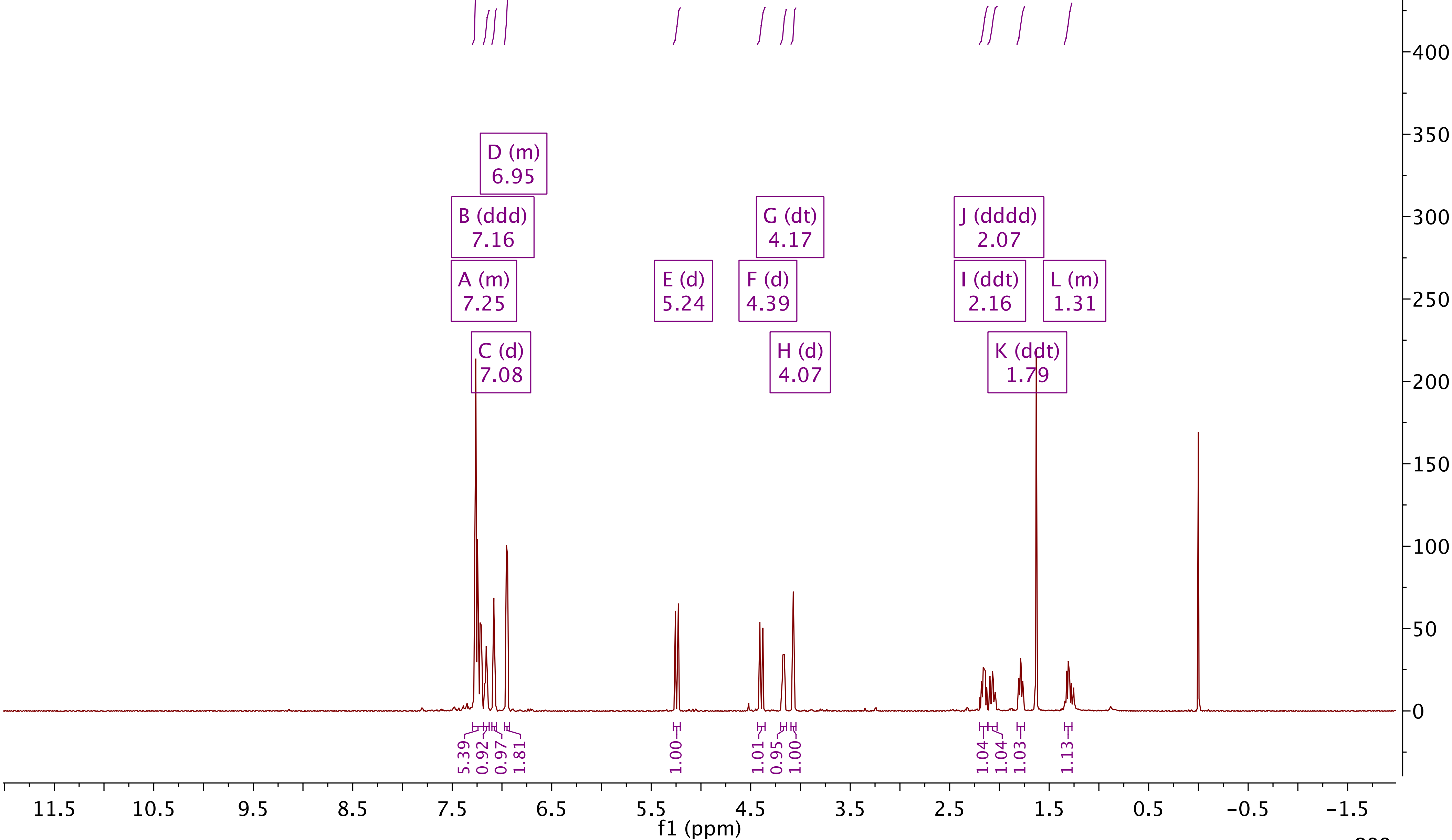
22b

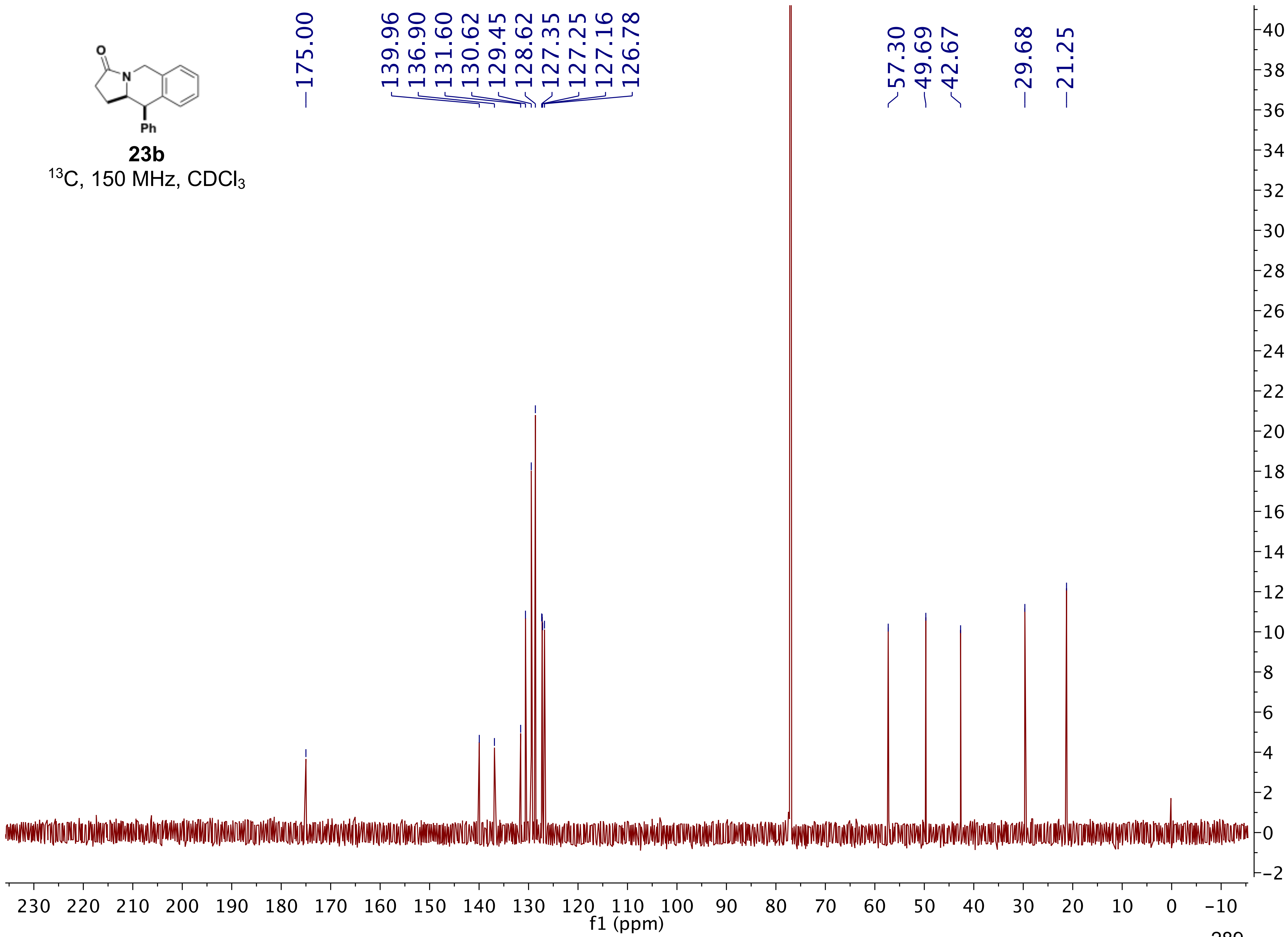
^{13}C, 150 MHz, $CDCl_3$

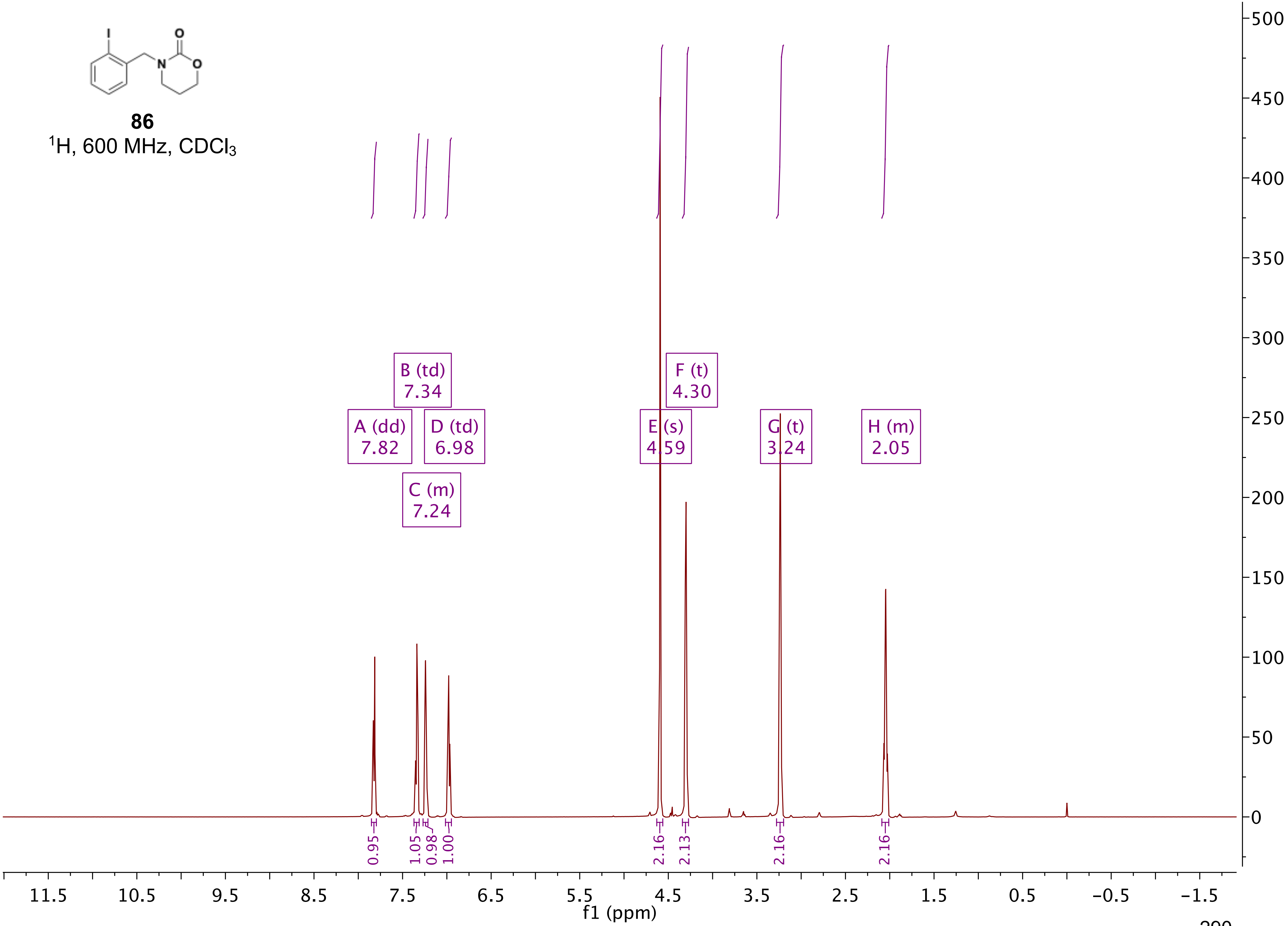


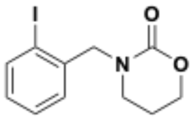


23b
^1H, 600 MHz, $CDCl_3$



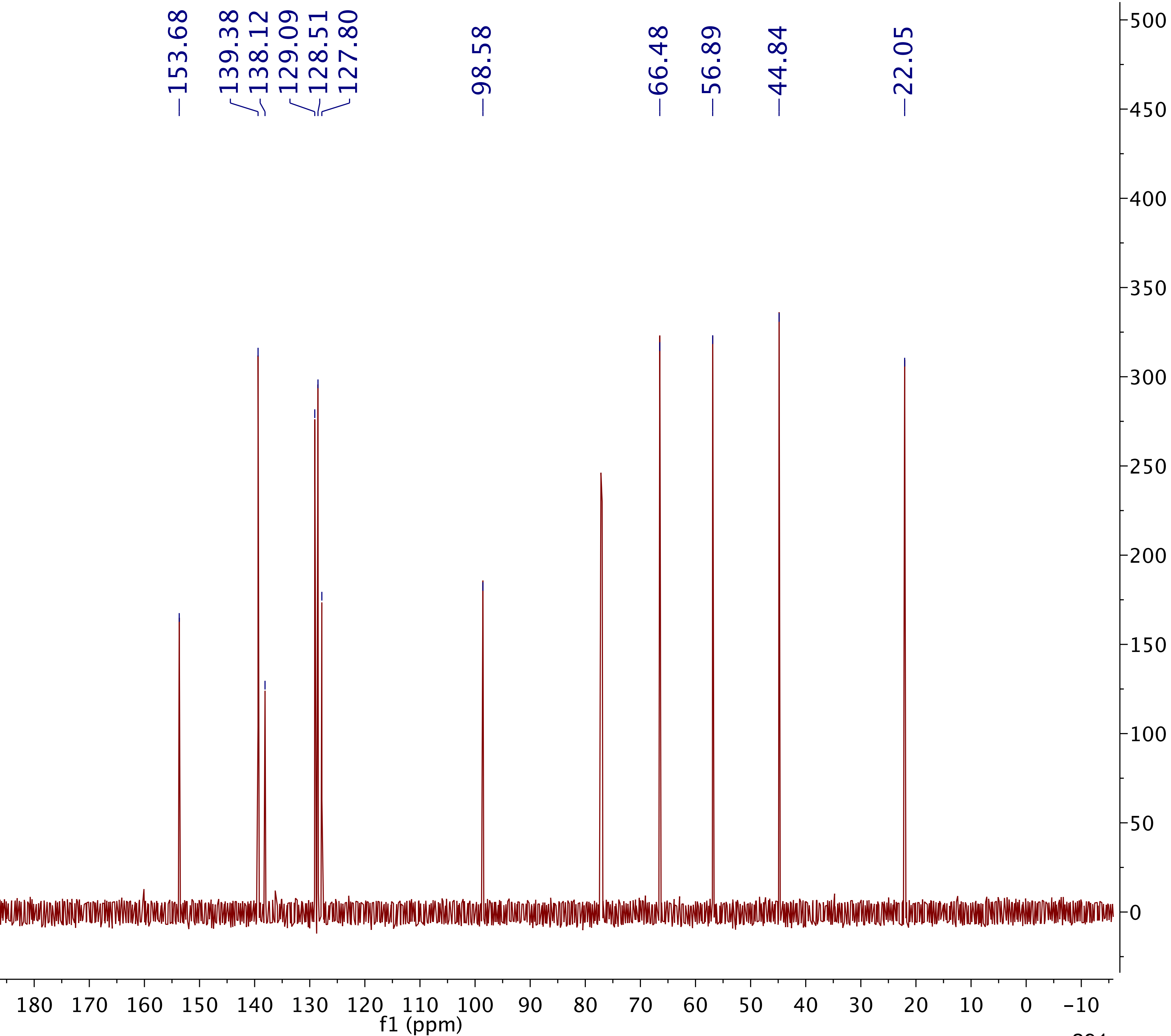

23b
^{13}C, 150 MHz, CDCl$_3$
175.00
139.96
136.90
131.60
130.62
129.45
128.62
127.35
127.25
127.16
126.78
57.30
49.69
42.67
29.68
21.25
f1 (ppm)

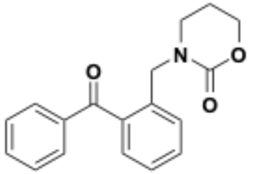

86
1H, 600 MHz, CDCl3
A (dd) 7.82
B (td) 7.34
C (m) 7.24
D (td) 6.98
E (s) 4.59
F (t) 4.30
G (t) 3.24
H (m) 2.05
0.95
1.05
0.98
1.00
2.16
2.13
2.16
2.16
f1 (ppm)



86

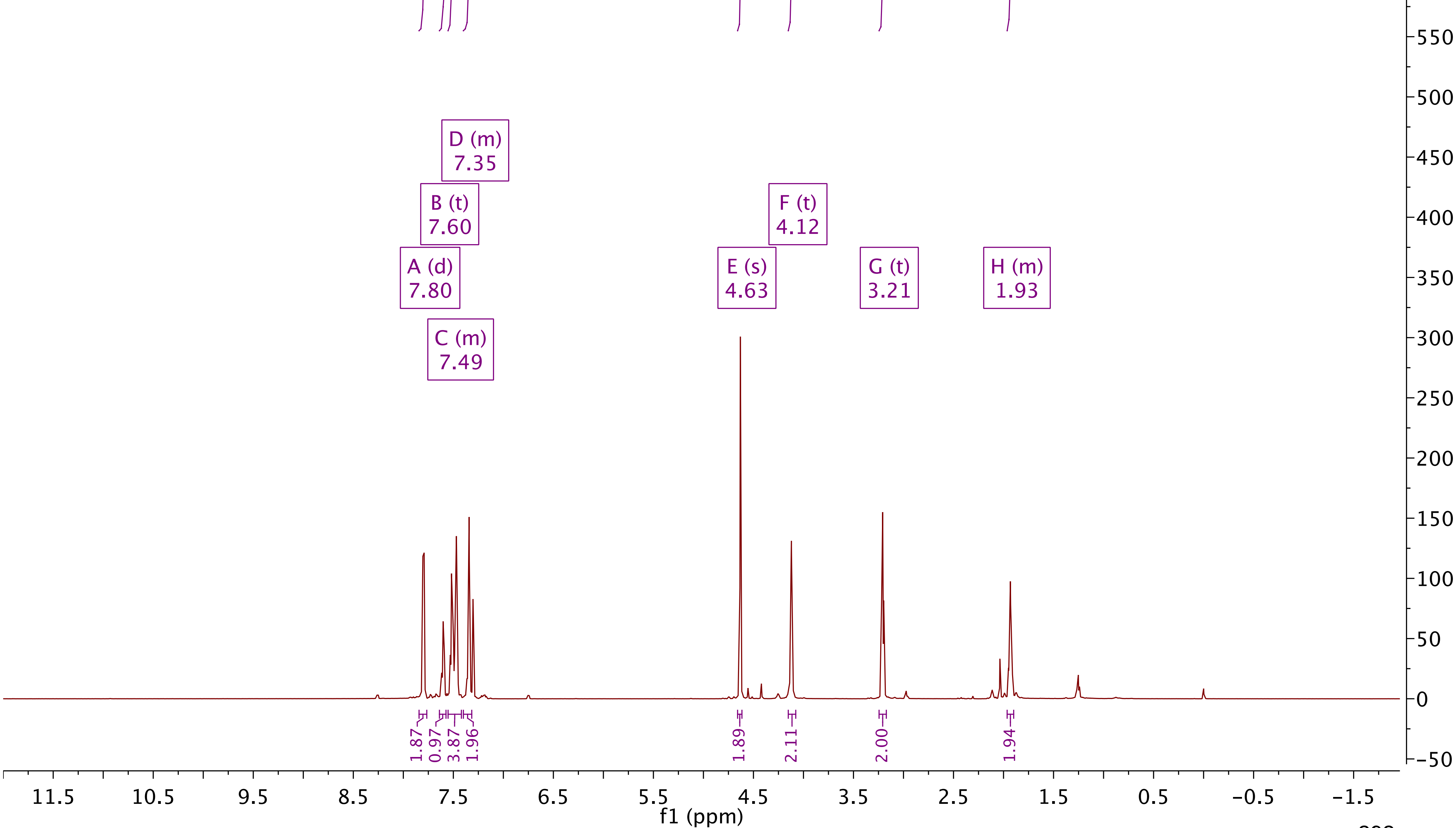
^{13}C, 150 MHz, $CDCl_3$

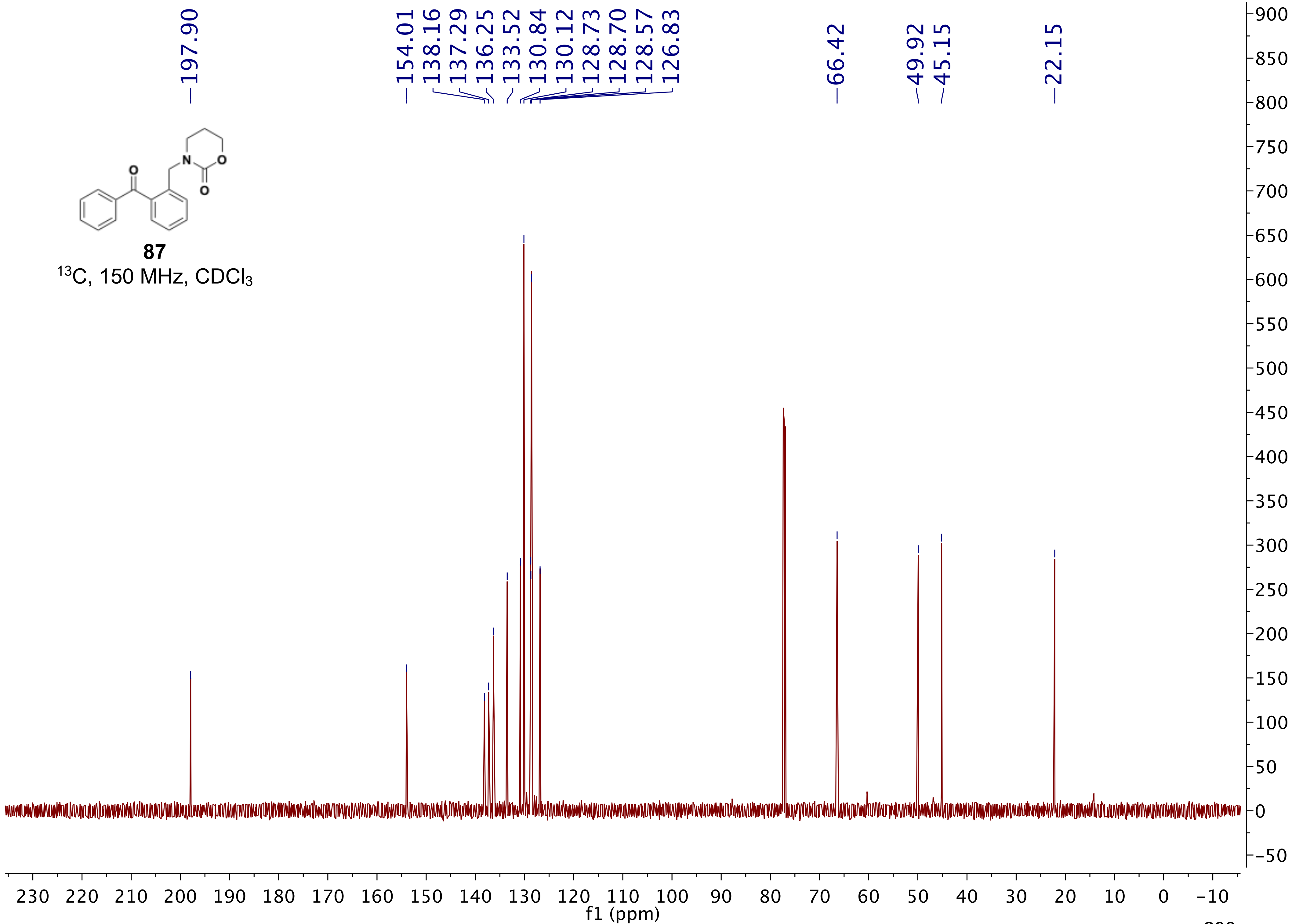




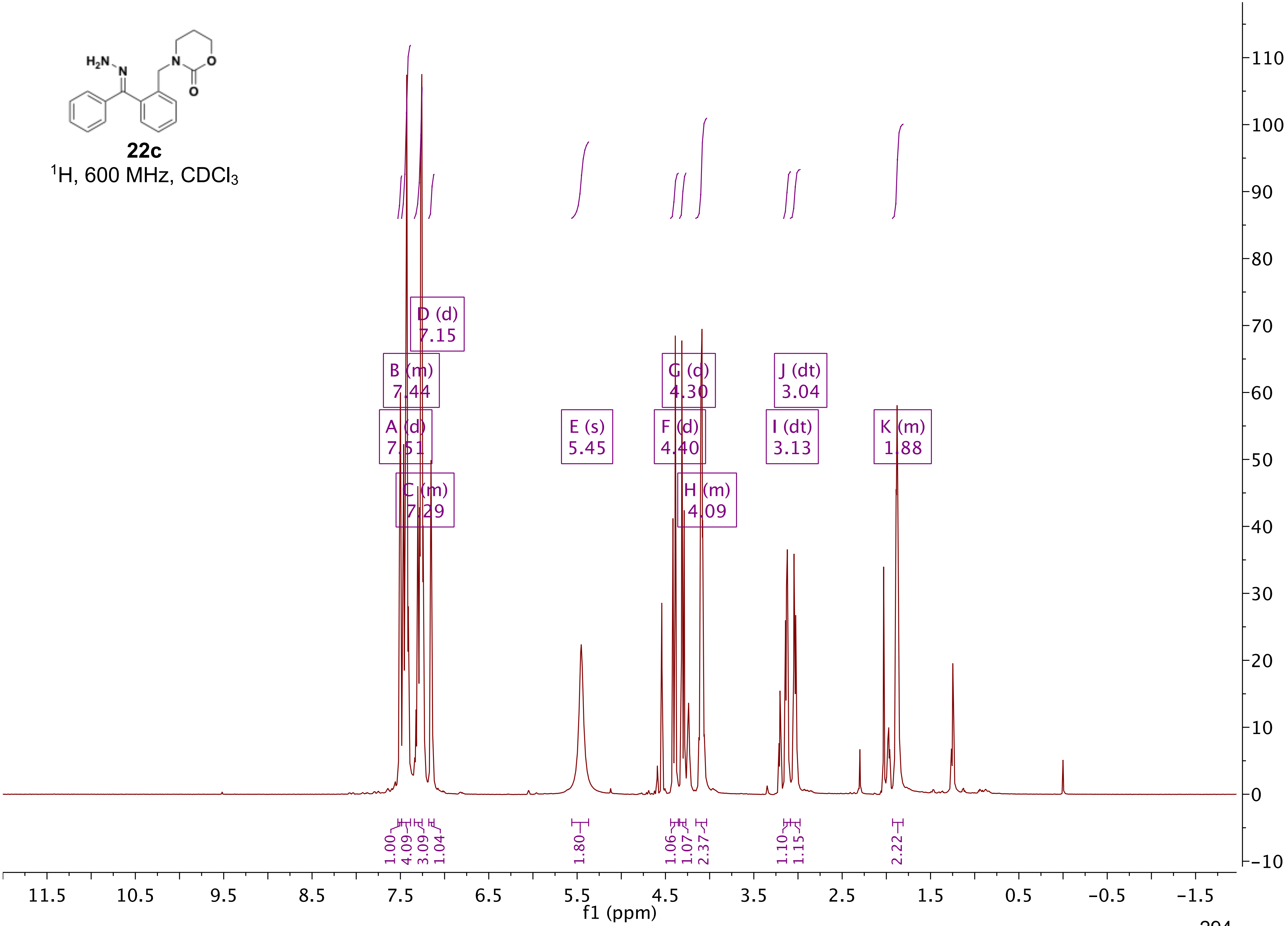
87

^{1}H, 600 MHz, $CDCl_3$



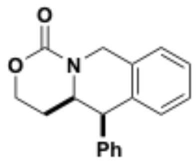


197.90
154.01
138.16
137.29
136.25
133.52
130.84
130.12
128.73
128.70
128.57
126.83
66.42
49.92
45.15
22.15
87
^{13}C, 150 MHz, $CDCl_3$
f1 (ppm)



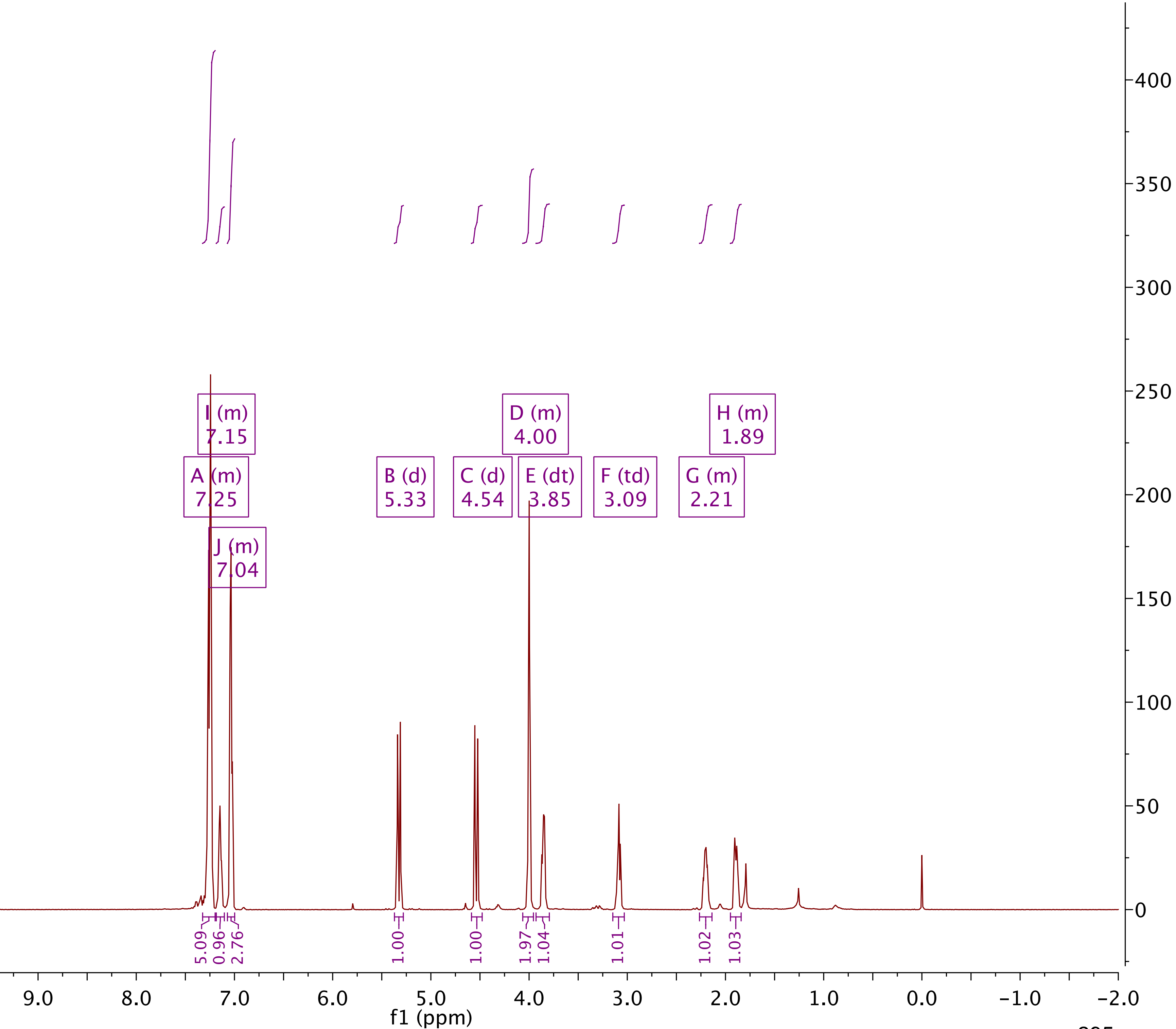
22c

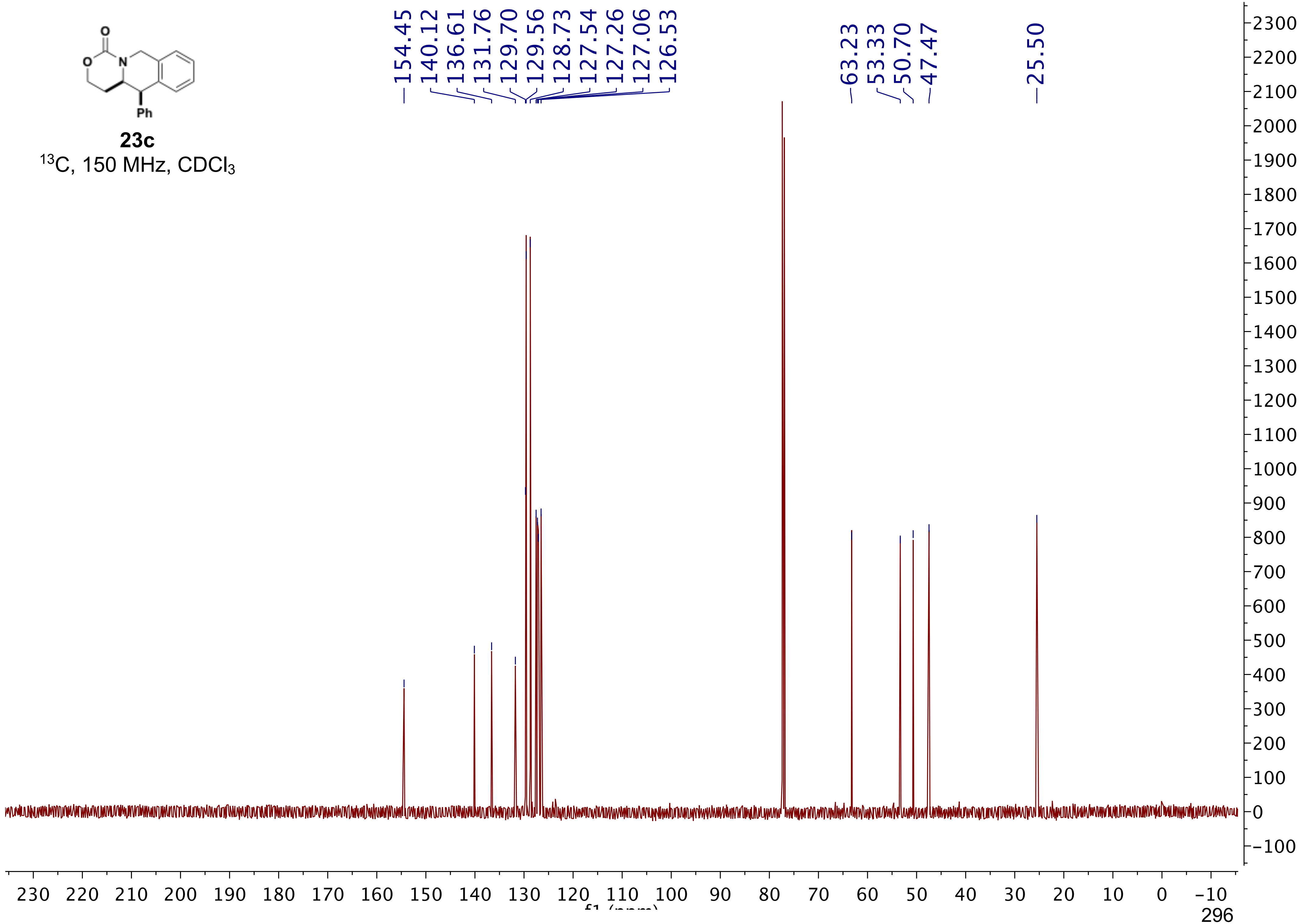
^{1}H, 600 MHz, $CDCl_3$

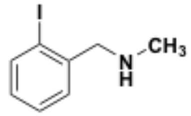


23c

^{1}H, 600 MHz, $CDCl_3$

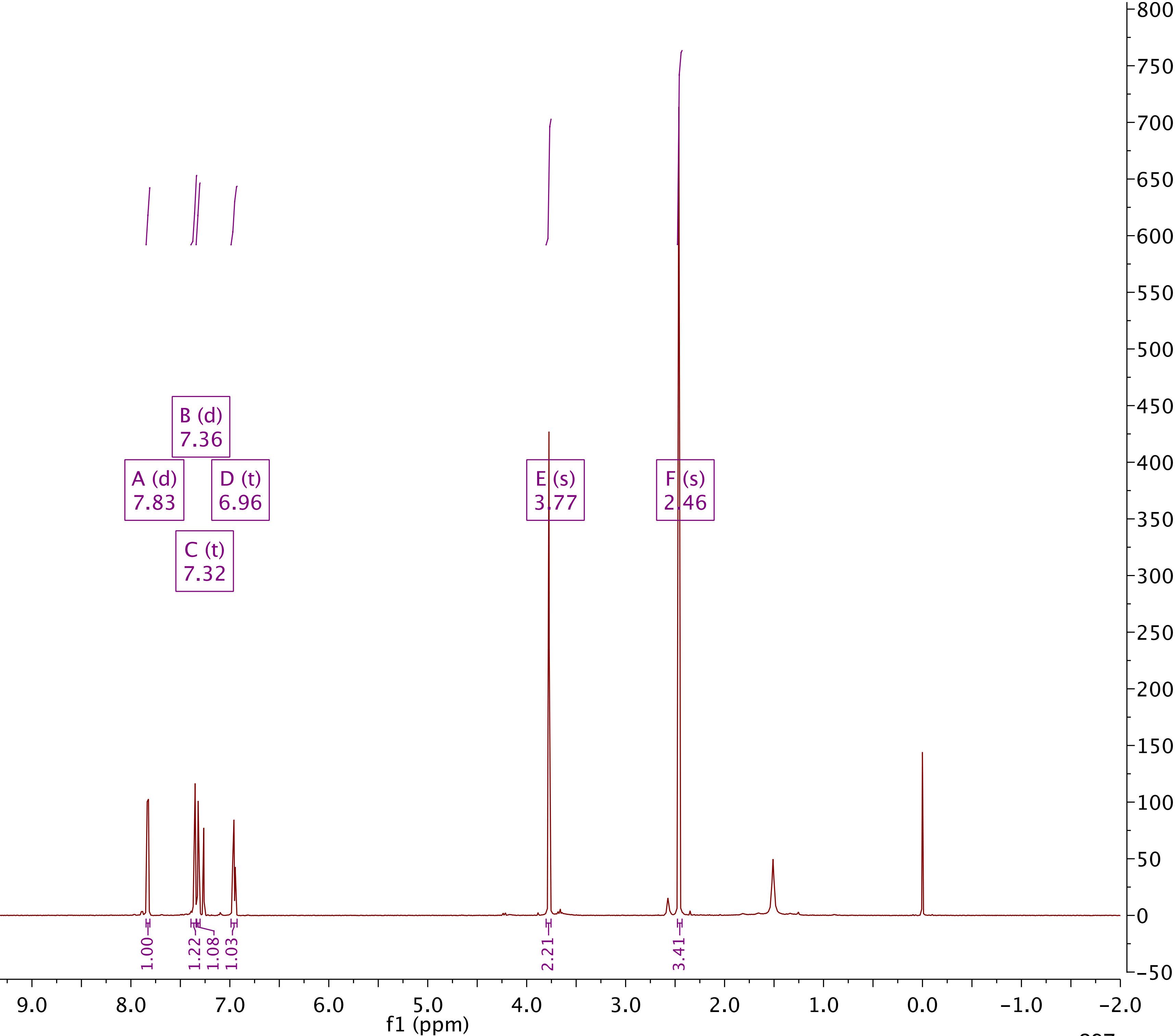



23c
^{13}C, 150 MHz, $CDCl_3$
154.45
140.12
136.61
131.76
129.70
129.56
128.73
127.54
127.26
127.06
126.53
63.23
53.33
50.70
47.47
25.50
f1 (ppm)



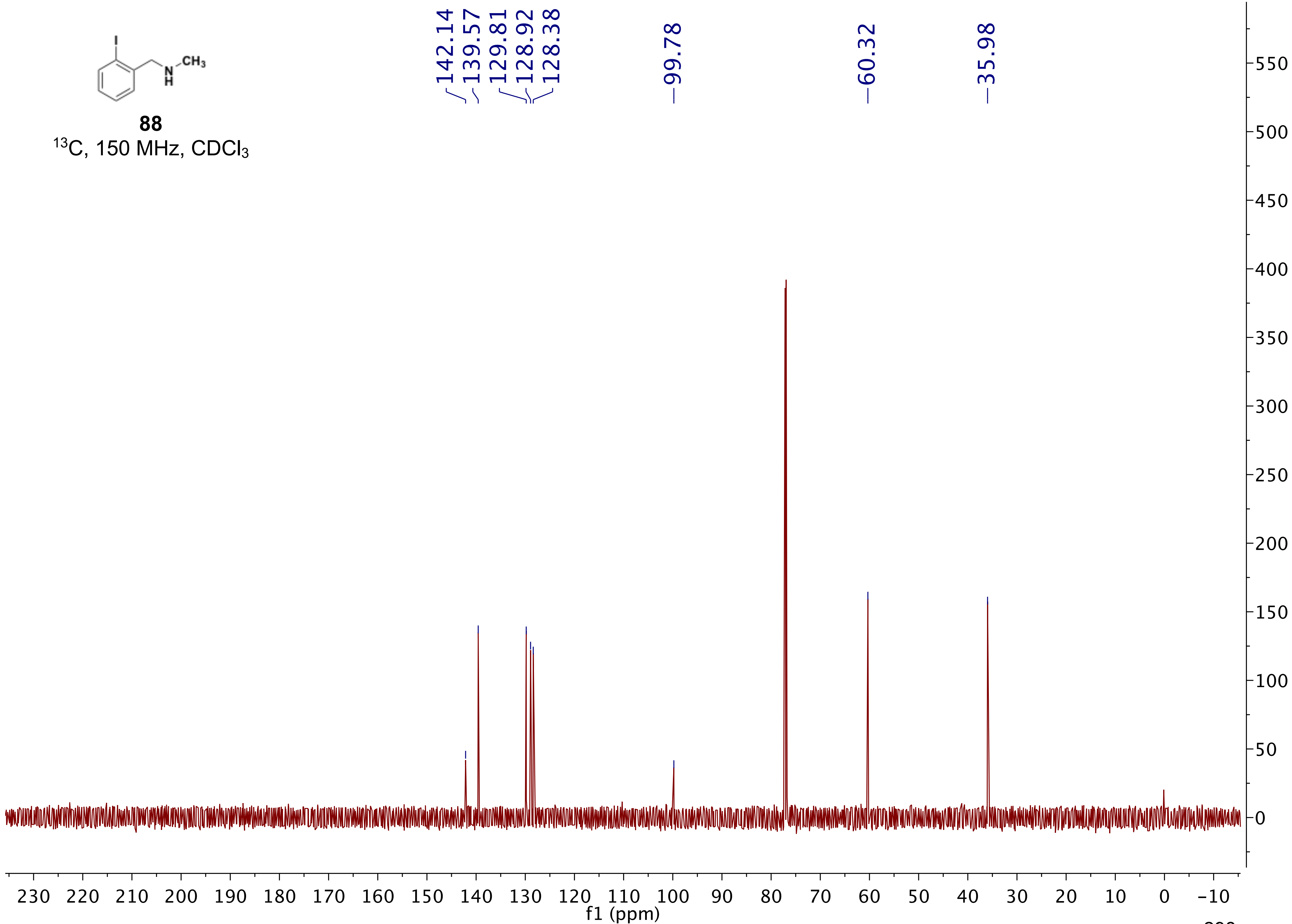
88

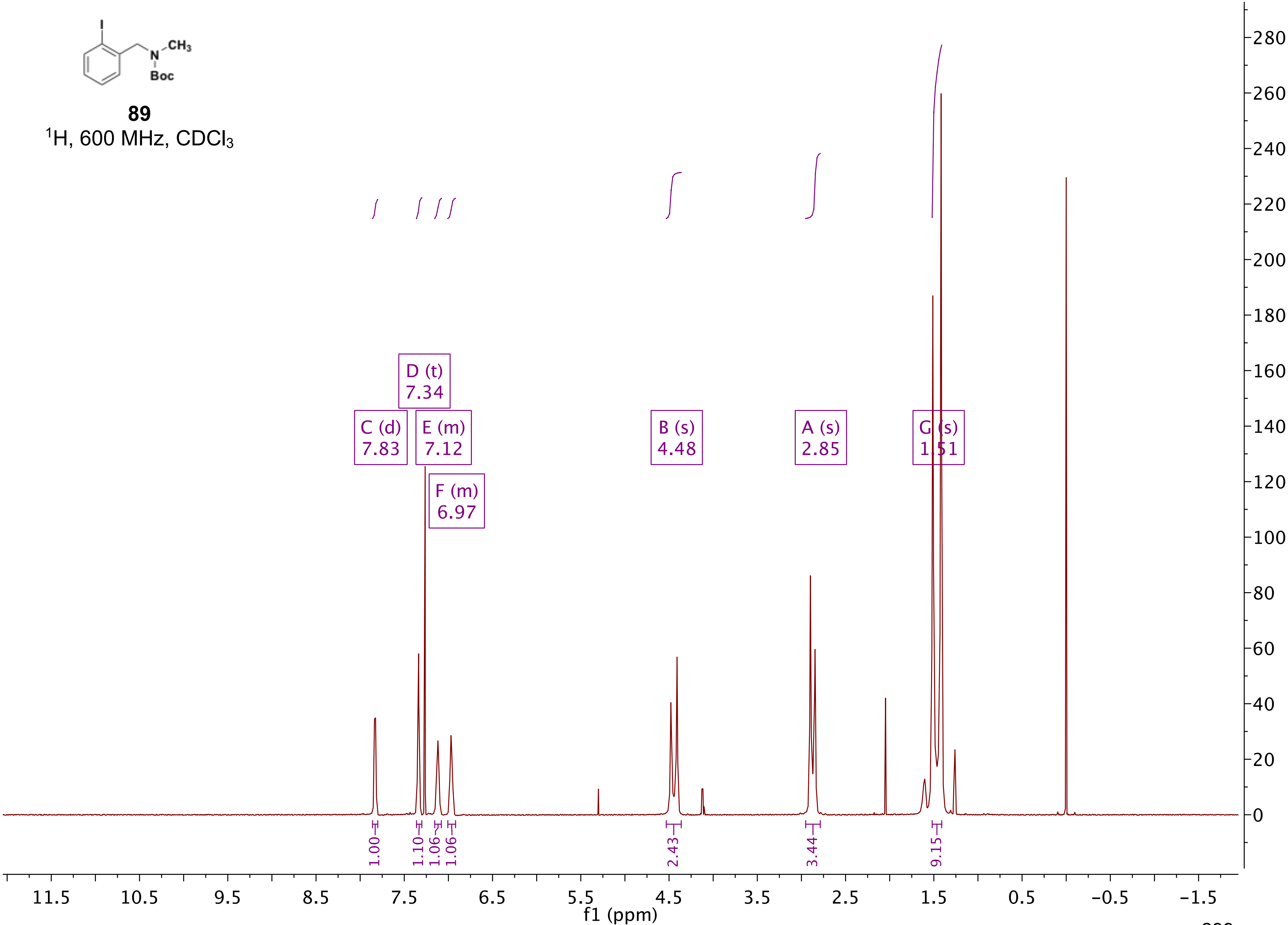
^{1}H, 600 MHz, $CDCl_3$

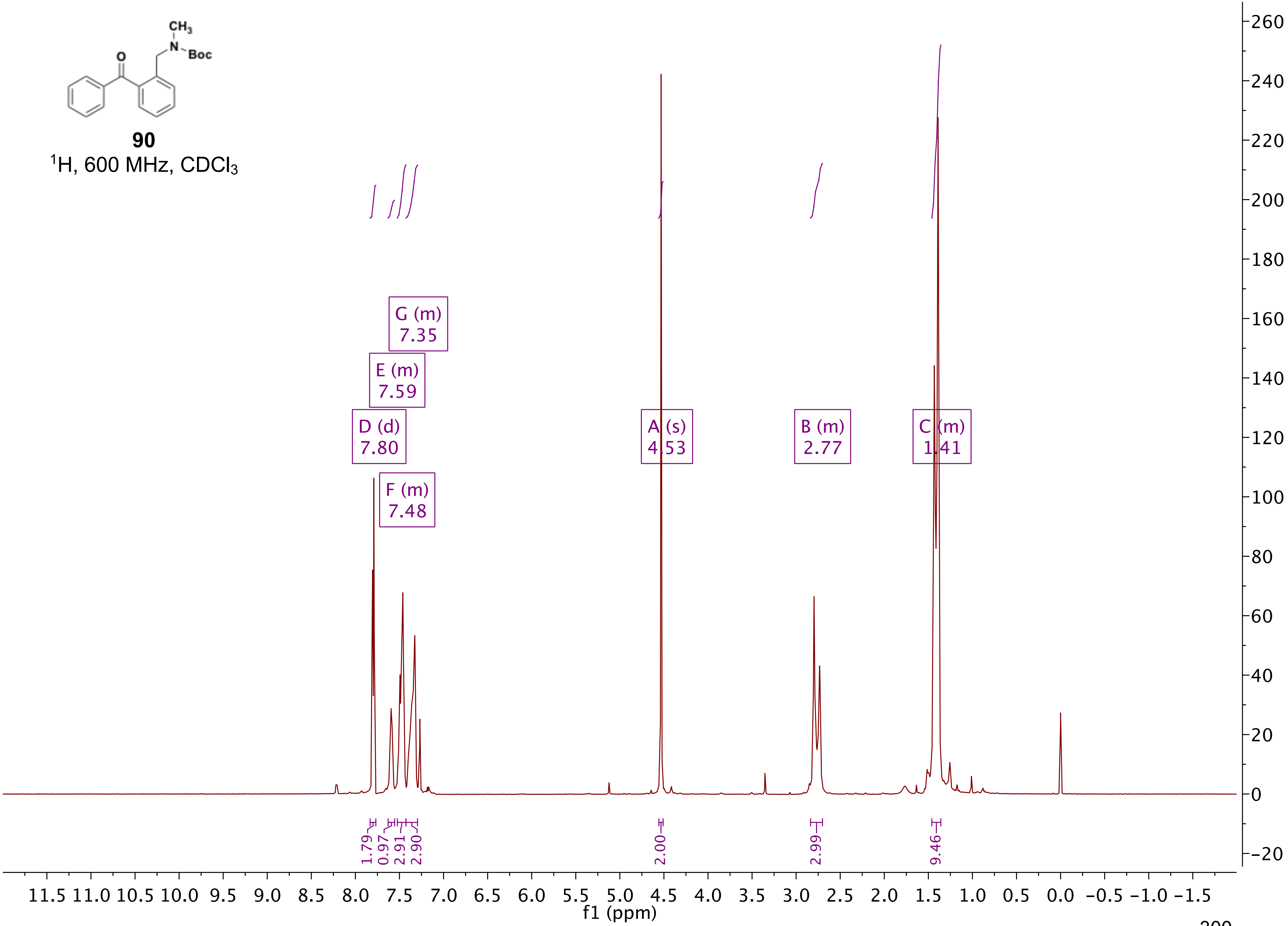


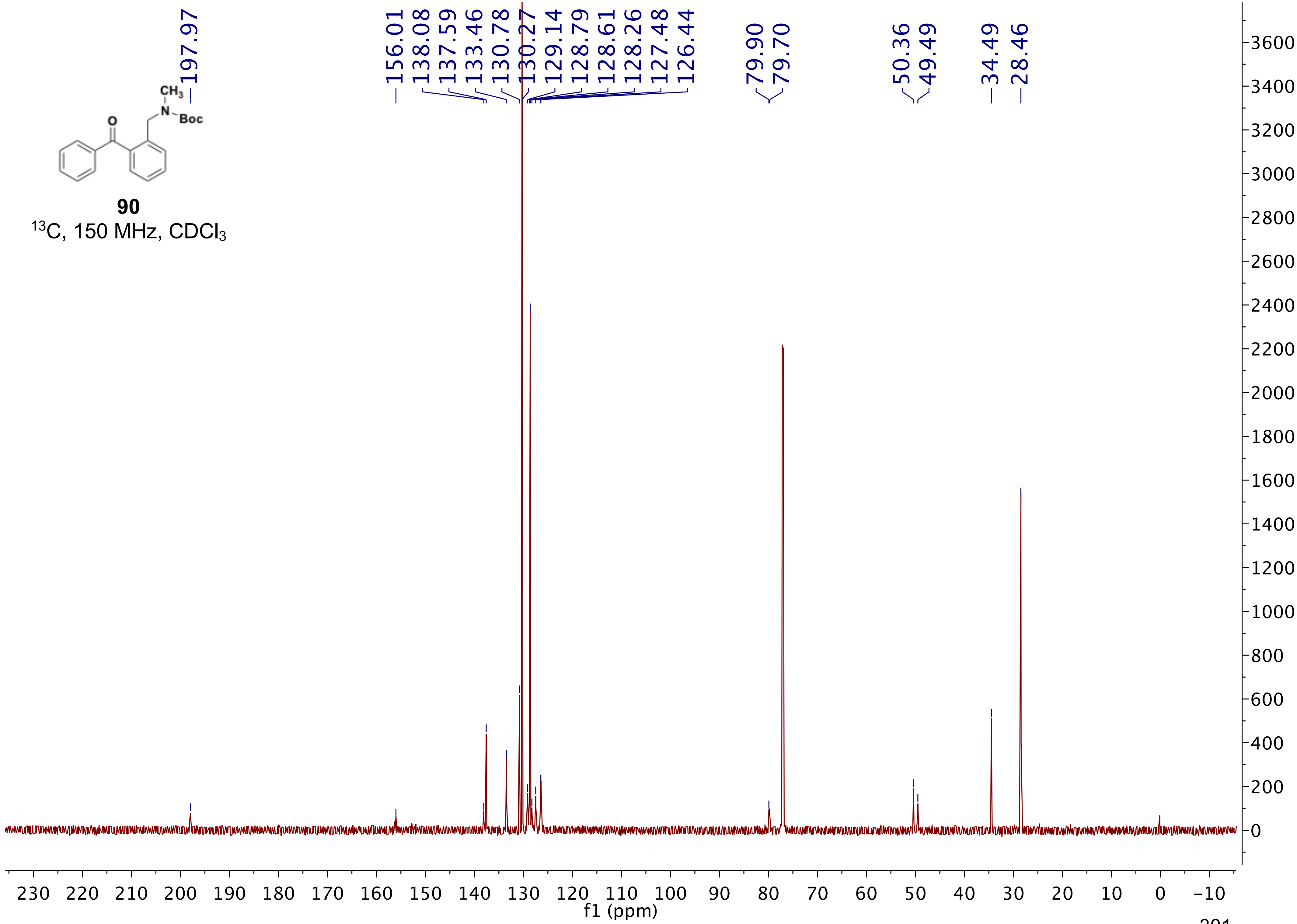
88

^{13}C, 150 MHz, $CDCl_3$

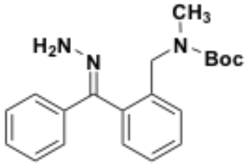



I
N
CH3
Boc
89
1H, 600 MHz, CDCl3
D (t)
7.34
C (d)
7.83
E (m)
7.12
F (m)
6.97
B (s)
4.48
A (s)
2.85
G (s)
1.51
1.00
1.10
1.06
1.06
2.43
3.44
9.15
11.5
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
−0.5
−1.5
f1 (ppm)
280
260
240
220
200
180
160
140
120
100
80
60
40
20
0

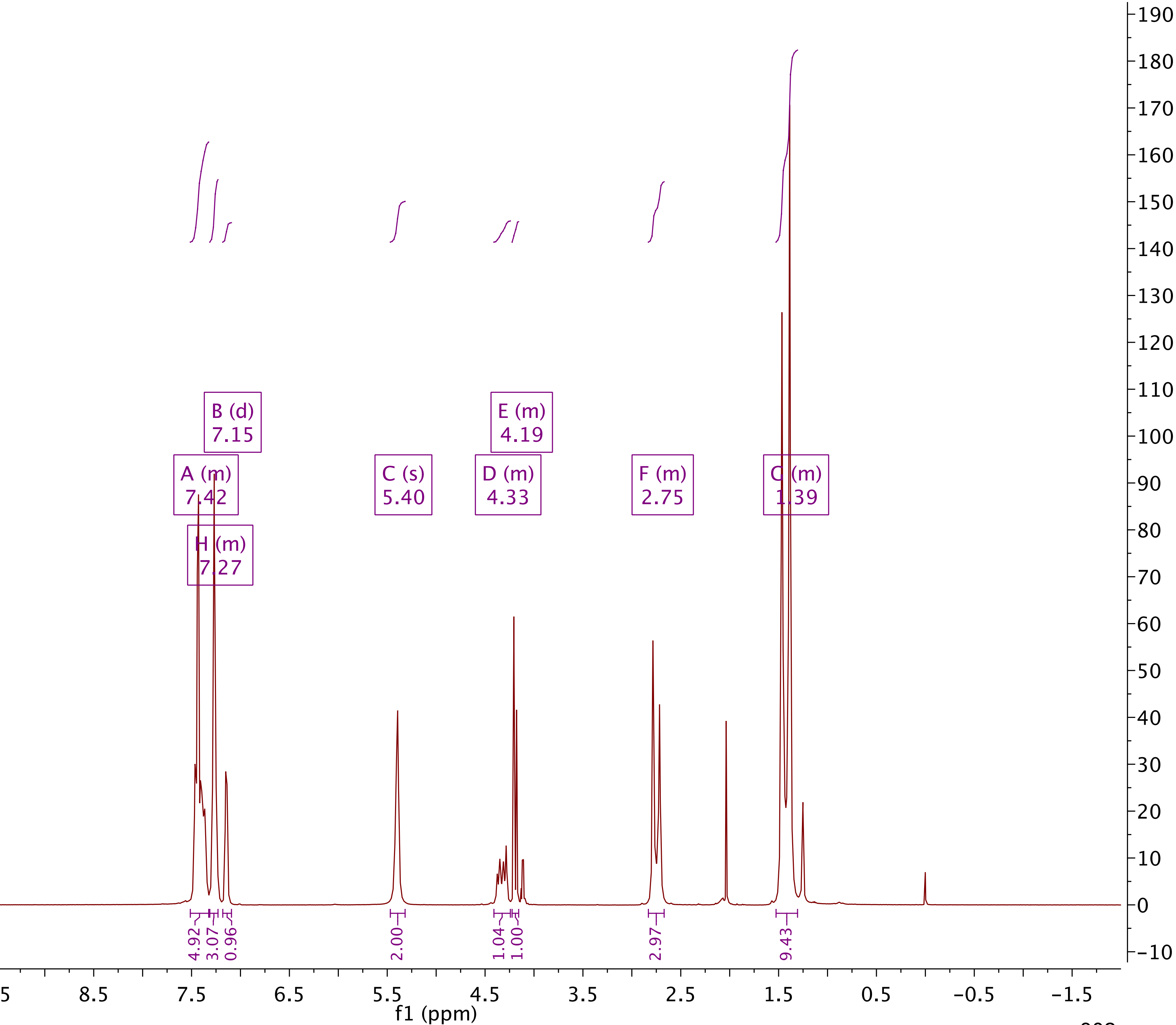

CH3
N
Boc
O
90
1H, 600 MHz, CDCl3
G (m) 7.35
E (m) 7.59
D (d) 7.80
F (m) 7.48
A (s) 4.53
B (m) 2.77
C (m) 1.41
1.79
0.97
2.91
2.90
2.00
2.99
9.46
f1 (ppm)

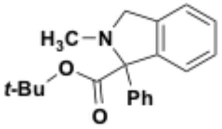


197.97
156.01
138.08
137.59
133.46
130.78
130.27
129.14
128.79
128.61
128.26
127.48
126.44
79.90
79.70
50.36
49.49
34.49
28.46
CH3
N
Boc
O
90
13C, 150 MHz, CDCl3
f1 (ppm)

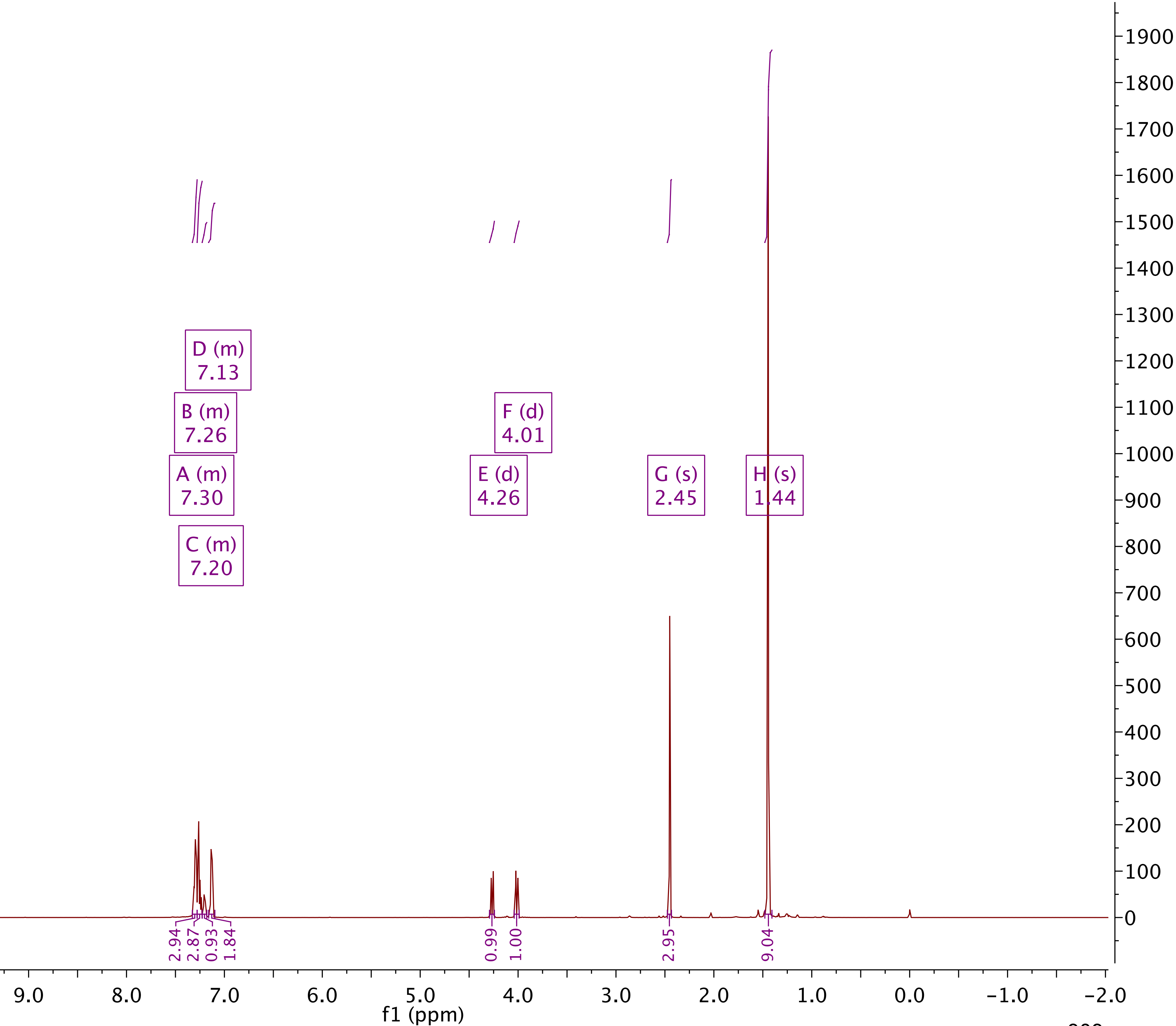


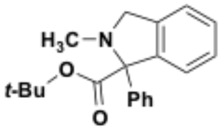
24
^{1}H, 600 MHz, $CDCl_3$





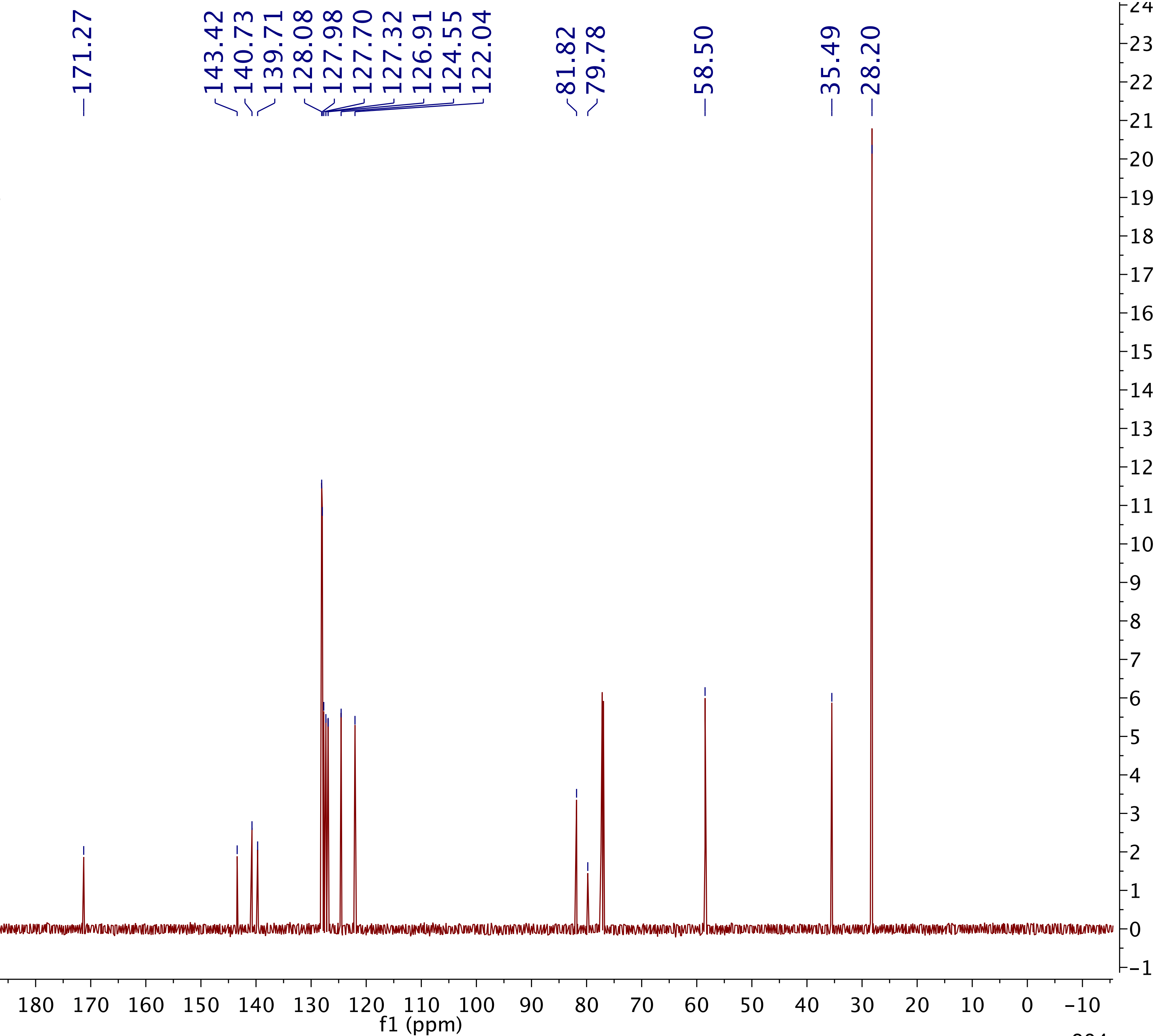
26
^1H, 600 MHz, $CDCl_3$

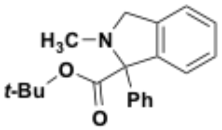




26

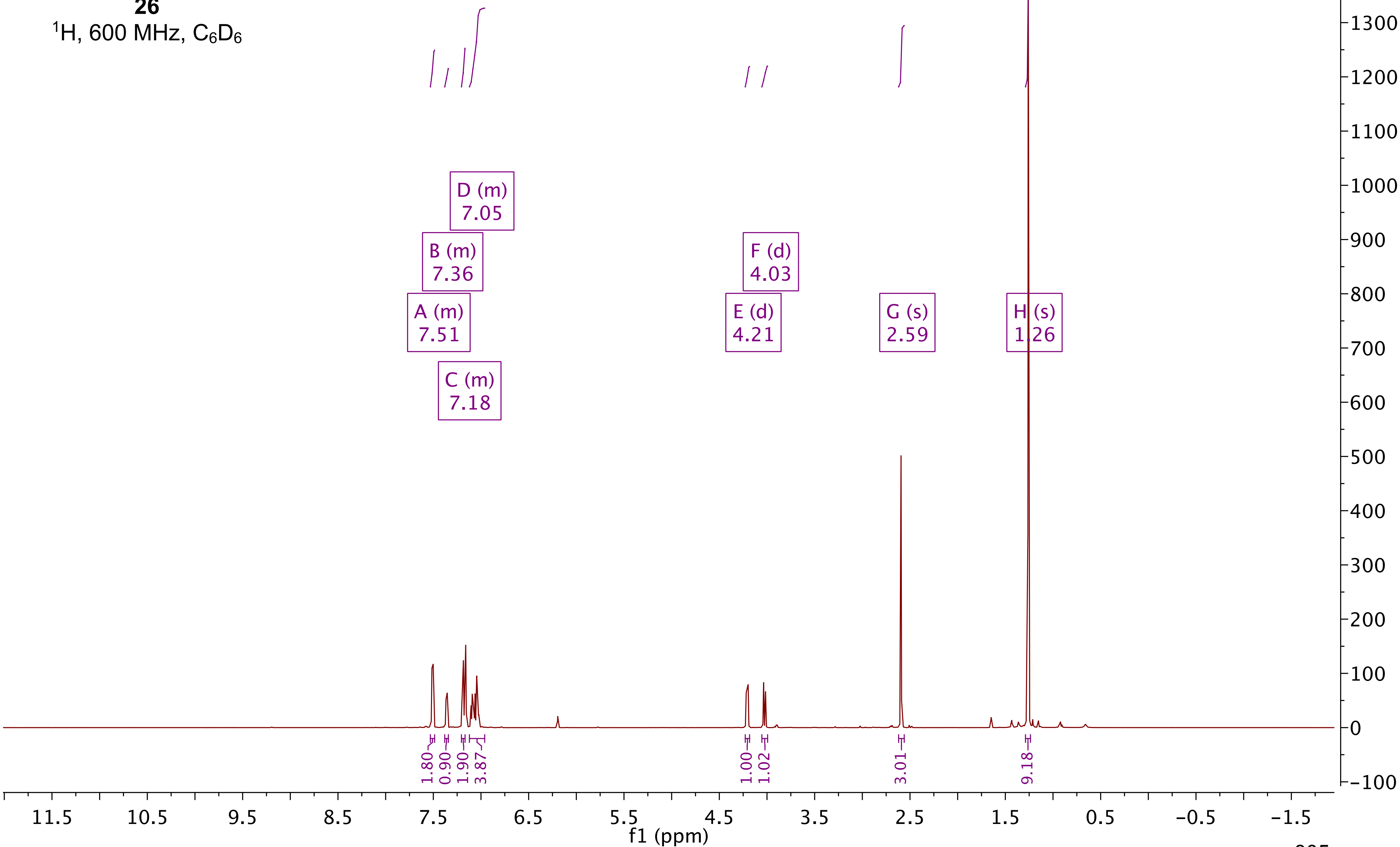
^{13}C, 150 MHz, $CDCl_3$

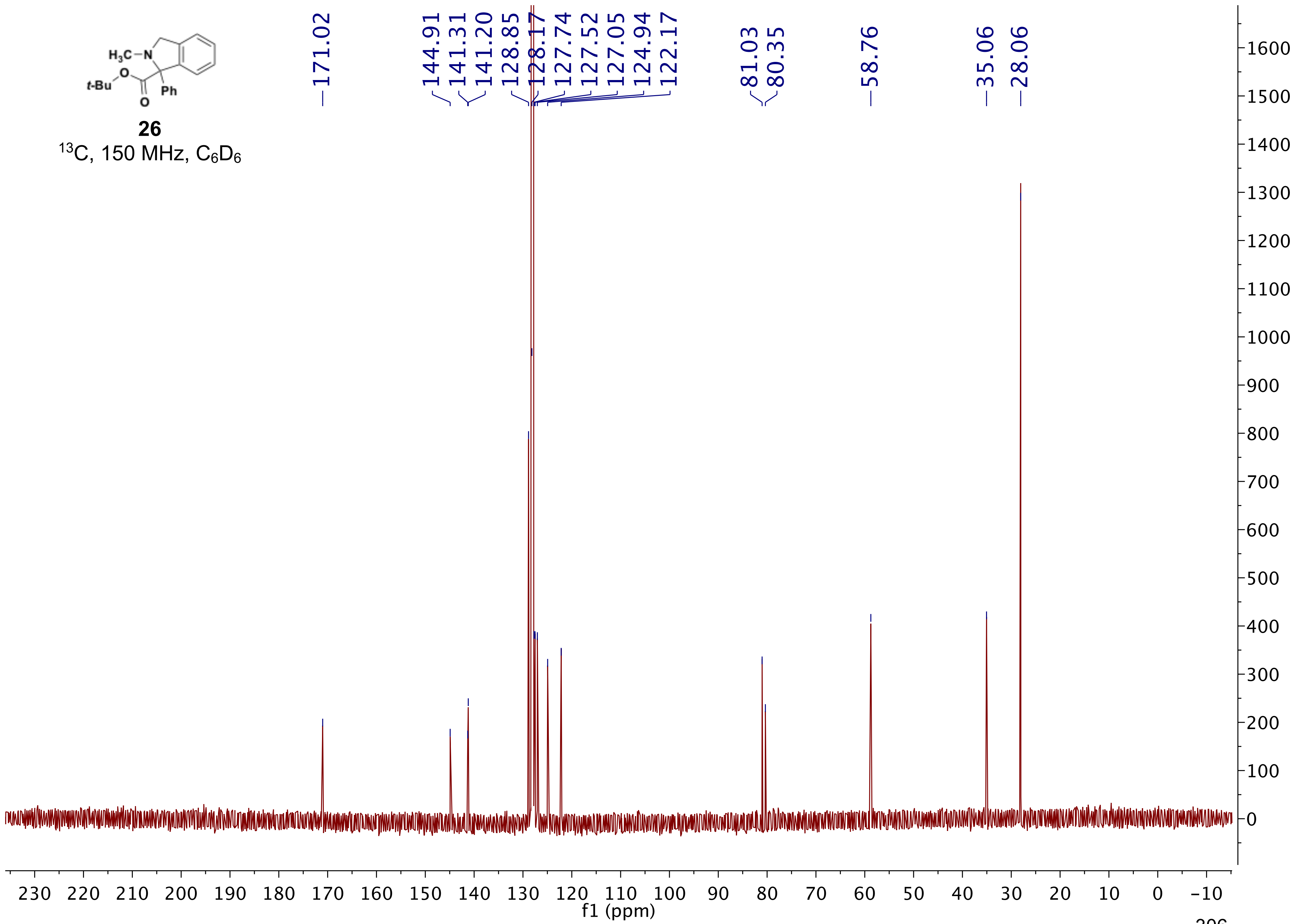




26

^{1}H, 600 MHz, C_6D_6




26
^{13}C, 150 MHz, C_6D_6
171.02
144.91
141.31
141.20
128.85
128.17
127.74
127.52
127.05
124.94
122.17
81.03
80.35
58.76
35.06
28.06
f1 (ppm)