Supporting Information

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Materials and Methods

All reactions were performed under an inert atmosphere of nitrogen with exclusion of moisture from reagents and glassware unless otherwise noted. All Ni-catalyzed coupling reactions were carried out in a glovebox (MBraun Unilab) filled with dry nitrogen. Heating was carried out using a Chemglass dryblock stir plate within the glovebox. $Ni(cod)_2$ and Ligand **1** were purchased from Strem Chemicals (Newburyport, MA) and stored in the glovebox. $Ni(cod)_2$ was stored at -20 °C, and it is crucial that it is a bright yellow color. Samples that appeared yellow-grey gave reduced yields. Toluene, THF, diethyl ether, and dichloromethane were degassed by sparging with nitrogen and dried by passage through a column of activated alumna on an SG Water solvent purification system. Aryl triflates, chlorides, and sulfonates were either purchased from commercial vendors and sparged with nitrogen before use, or prepared using standard procedures[1] then sparged with nitrogen and dried over 3 Å MS. 1-octene, vinylcyclohexane, (–)-β-citronellene, saffrole, and allyl benzene were distilled from Na prior to use. Other alkenes were used as purchased after sparging with nitrogen. Triethylsilyl trifluoromethanesulfonate (TESOTf) was distilled from CaH_2 under reduced pressure prior to use to give best yields. Alternatively, TESOTf can be used directly from the manufacturer though some reduction in yield was obtained, particularly if the solution was a dark brown rather than colorless (indicating purity). All other reagents (including DABCO) were used as received. Commercially available chemicals were purchased from either Sigma-Aldrich Chemical Company (Milwaukee, WI), Alfa Aesar (Ward Hill, MA), Acros Organics (Pittsburgh, PA), or TCI America (Portland, OR). Analytical thin-

[1] (a) Triflates and Mesylates: Kleimark, J.; Hedström, A.; Larsson, P.-F.; Johansson, C.; Norrby, P.-O. *ChemCatChem*, **2009**, *1*, 152–161. (b) 2-napthyl tosylate: Ogata, T.; Hartwig, J. F. *J. Am. Chem. Soc.* **2008**, *130*, 13848–13849. (c) Sulfamate: Quasdorf, K. W.; Andoft-Finch, A.; Liu, P.; Silberstein, A. L.; Komaromi, A.; Blackburn, T.; Ramgren, S. D.; Houk, K. N.; Snieckus, V.; Garg, N. K. *J. Am. Chem. Soc.* **2011**, *133*, 6352–6363.

layer chromatography (TLC) was performed on 0.2 mm coated Science silica gel (EM 60-F254) plates. Visualization was accomplished with UV light (254 nm) and exposure to either ceric ammonium molybdate (CAM), anisaldehyde, or $KMnO_4$ solution followed by heating. Column chromatography was carried out on a Biotage Isolera flash chromatography system using SNAP KP-Sil or HP-Sil columns (silica gel, average particle size 50 μm and 25 μm respectively).

^1H NMR Spectra were obtained on either a Bruker 400 MHz, Bruker 600 MHz, or Varian Inova 500 MHz NMR instrument; ^{13}C spectra were recorded on a Bruker 400 MHz (at 100 MHz) NMR instrument. Chemical shifts (^1H and ^{13}C) are reported in parts per million and referenced to the residual solvent peak (for $CDCl_3$, $\delta = 7.27$ ppm, 77.0ppm respectively). The following designations are used to describe multiplicities: s (singlet), d (doublet), t (triplet), q (quartet), quin (quintet), m (multiplet), br (broad), app (apparent). IR spectra were obtained on an Agilent Cary 630 FT-IR spectrometer equipped with an ATR accessory. High-resolution mass spectrometry data were acquired by the Department of Chemistry Instrumentation Facility, Massachusetts Institute of Technology on a Bruker Daltonics APEXIV 4.7 Tesla FT-ICR Mass Spectrometer. Gas chromatography (GC) was performed on an Agilent 5870 GC (HP-5 column) with a flame ionization detector. GC/MS was performed on an Agilent 5870 GC (HP-5ms column) with an Agilent 5975C MSD. Dodecane (99+%, Alfa Aesar) was used as an internal standard for quantification.

Ni-Catalyzed Heck Reactions

General Procedures

A) For aryl triflates: In a glovebox, Ligand **1** (0.06 mmol, 0.12 equiv) and $Ni(cod)_2$ (0.05 mmol, 0.10 equiv) were weighed into an 8mL vial. THF (1 M), DABCO (1.5 mmol, 3.0 equiv), and alkene (0.75 mmol, 1.5 equiv) were added with stirring. The aryl triflate (0.5 mmol, 1.0 equiv) was then added, the vial was capped, and the reaction mixture was heated to 60 °C for 24 h. When completed, the reaction mixture was removed from the glovebox and 3.0 mL of benzene was added.

B) For aryl chlorides, mesylates, tosylates, and sulfamates: In a glovebox, Ligand **1** (0.06 mmol, 0.12 equiv) and $Ni(cod)_2$ (0.05 mmol, 0.10 equiv) were weighed into an 8mL vial. Half the toluene (0.5 M in total), DABCO (2.5 mmol, 5.0 equiv), and alkene (0.75 mmol, 1.5 equiv), and were added with stirring. The aryl chloride, mesylate, tosylate or sulfamate (0.5 mmol, 1.0 equiv) was then added, and the sides of the vial were rinsed with the remaining toluene. Triethylsilyl trifluoromethanesulfonate (TESOTf, 1.0 mmol, 2.0 equiv) was then added, the vial was capped, and the reaction mixture was heated to 60 °C for 24 h. When completed, the reaction mixture was removed from the glovebox and 400 µL of dry methanol was added to quench the remaining TESOTf and TESCl and allowed to stir at room temperature (capped) for 20 min. If this step is not carried out, water from the SiO_2 will form the disilyl ether, TES_2O, which, for non-polar substrates can be nearly impossible to separate via column chromatography. The silyl ether TESOMe is volatile and can be removed under reduced pressure. After the methanol quench, 3.0 mL of benzene was added.

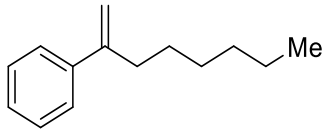
Both A) and B): For reactions analyzed by gas chromatography (GC), the crude mixture was passed through a plug of SiO_2, using a small amount of CH_2Cl_2 to solubilize any precipitate, and eluting with diethyl ether. An external standard (dodecane, 70.2 µL, 1.0 equiv) was then added, the sample was diluted to 50 mL in a volumetric flask and submitted to GC analysis. The desired branched product area was then compared to both the linear product area (br/ln) and to the sum of all isomers, including linear, of the product (rr). The identity of the peak corresponding to the linear product isomer was confirmed by reaction of the substrates with $Ni(cod)_2$ in the absence of Ligand **1**, giving a roughly 1:1 mixture of branched/linear products. Mechanistically, there are two sources of regioisomers: br/ln selectivity is determined in the migratory insertion step, while overall rr can be degraded later in the catalytic cycle by reinsertion of a Ni–H species to the product and subsequent isomerization. As expected, the major isomerized product is the trisubstituted *E*-styrene derivative. When very bulky monodentate phosphines are used, this is the major product. Structure confirmed by ^1H NMR and nOe correlation. GC conditions: 50 °C for 2 min, ramp 20 °C/min to 250 °C, hold at 250 °C for 10 min.

For isolated yields, 15 µL was removed from the mixture diluted in benzene and added to a small amount of hexanes to precipitate out any nickel species. This sample was filtered with a syringe filter into a GC vial, diluted in Et_2O, and put on the GC to determine rr pre-purification. The remaining reaction mixture was passed through a short plug of SiO_2 and then purified via column chromatography. A small amount of the final mixture was then used to prepare another GC sample to determine rr and br/ln post purification.

A Note on Purification

Some substrates, particularly the electron-rich alkenes such as **3b** and **3s** are prone to acid-catalyzed isomerization. This seems to be especially the case if the crude reaction mixture is loaded directly upon a silica column and eluted with hexanes. Running the reaction through a silica plug, eluting with benzene, tends to mitigate most of the isomerization, though not in every case. Therefore, an rr of the reaction mixture pre-purification as noted above was always obtained via GC analysis. Finally, most column chromatography was performed on Et_3N-pretreated columns to avoid any further isomerization on sensitive substrates. When pre-packed silica gel Biotage columns were used for purification, and pretreatment with Et_3N is noted below, columns were flushed with 5-8 column volumes (CV) of a mixture of 2% Et_3N in Hexanes, then equilibrated with pure hexanes for 8 CV in order to purify non-polar products well. Refer to specific substrates below for details of purification.

Characterization of Products



Oct-1-en-2-ylbenzene (3a):

From PhOTf: Following the general procedure **A**, Ligand **1** (34.2 mg, 0.06 mmol, 0.12 equiv), $Ni(cod)_2$ (13.8 mg, 0.05 mmol, 0.10 equiv), THF (500 μL, 1 M), DABCO (168 mg, 1.5 mmol, 3.0 equiv), 1-octene (118 μL, 0.75 mmol, 1.5 equiv), and phenyl trifluoromethanesulfonate (81 μL, 0.5 mmol, 1.0 equiv) were added to an 8 mL vial and stirred at 60 °C for 24 h in a nitrogen-filled

glovebox. When the reaction was done, it was removed from the glovebox and 3.0 mL benzene was added. An aliquot was taken for GC analysis as described above. Complete conversion was obtained, so simple filtration through a silica plug with benzene as the eluent afforded 93.3 mg (99%) **3a** as a colorless oil (rr pre-purification: 38.3, rr post-purification: 32.9, br/ln 87:1 determined by GC analysis).

From PhCl: Following the general procedure **B**, Ligand **1** (34.2 mg, 0.06 mmol, 0.12 equiv), $Ni(cod)_2$ (13.8 mg, 0.05 mmol, 0.10 equiv), Toluene (500 μL), DABCO (280 mg, 2.5 mmol, 5.0 equiv), 1-octene (118 μL, 0.75 mmol, 1.5 equiv), and chlorobenzene (50.9 μL, 0.5 mmol, 1.0 equiv) were added to an 8 mL vial. Toluene (500 μL) was used to rinse down the sides of the vial, and triethylsilyl trifluoromethanesulfonate (226 μL, 1.0 mmol, 2.0 equiv) was added, and the reaction was stirred at 60 °C for 24 h in a nitrogen-filled glovebox. When the reaction was done, it was removed from the glovebox. Dry methanol (400 μL) was added to quench, and the reaction was stirred for 20 min at room temperature (capped). Benzene (3 mL) was then added, and an aliquot was taken for GC analysis as described above. The mixture was passed through a silica plug with benzene as the eluent and concentrated. Column chromatography (Biotage 10g HP-sil, pretreated with Et_3N, 100% hexanes) afforded 76.6 mg (81%) of **3a** as a colorless oil (rr pre-purification: 27.7, rr post purification: 35.7, br/ln: 62:1 determined by GC analysis).

From PhOMs: Following the general procedure **B**, Ligand **1** (34.2 mg, 0.06 mmol, 0.12 equiv), $Ni(cod)_2$ (13.8 mg, 0.05 mmol, 0.10 equiv), Toluene (500 μL), DABCO (280 mg, 2.5 mmol, 5.0 equiv), 1-octene (118 μL, 0.75 mmol, 1.5 equiv), and phenyl methanesulfonate (88.0 mg, 0.5 mmol, 1.0 equiv) were added to an 8 mL vial. Toluene (500 μL) was used to rinse down the sides of the

vial, and triethylsilyl trifluoromethanesulfonate (226 μL, 1.0 mmol, 2.0 equiv) was added, and the reaction was stirred at 60 °C for 24 h in a nitrogen-filled glovebox. When the reaction was done, it was removed from the glovebox. Dry methanol (400 μL) was added to quench, and the reaction was stirred for 20 min at room temperature (capped). Benzene (3 mL) was then added, and an aliquot was taken for GC analysis as described above. The mixture was passed through a silica plug with benzene as the eluent and concentrated. Column chromatography (Biotage 10g HP-sil, pretreated with Et_3N, 100% hexanes) afforded 87.6 mg (91%) of **3a** as a colorless oil (rr pre-purification: 30.0, rr post purification: 30.4, br/ln: 59:1 determined by GC analysis).

From PhOTs: Following the general procedure **B**, Ligand **1** (34.2 mg, 0.06 mmol, 0.12 equiv), $Ni(cod)_2$ (13.8 mg, 0.05 mmol, 0.10 equiv), Toluene (500 μL), DABCO (280 mg, 2.5 mmol, 5.0 equiv), 1-octene (118 μL, 0.75 mmol, 1.5 equiv), and phenyl *p*-toluenesulfonate (121.3 mg, 0.5 mmol, 1.0 equiv) were added to an 8 mL vial. Toluene (500 μL) was used to rinse down the sides of the vial, and triethylsilyl trifluoromethanesulfonate (226 μL, 1.0 mmol, 2.0 equiv) was added, and the reaction was stirred at 60 °C for 24 h in a nitrogen-filled glovebox. When the reaction was done, it was removed from the glovebox. Dry methanol (400 μL) was added to quench, and the reaction was stirred for 20 min at room temperature (capped). Benzene (3 mL) was then added, and an aliquot was taken for GC analysis as described above. The mixture was passed through a silica plug with benzene as the eluent and concentrated. Column chromatography (Biotage 10g HP-sil, pretreated with Et_3N, 100% hexanes) afforded 66.5 mg (72%) of **3a** as a colorless oil (rr pre-purification:17.4, rr post purification: 19.6, br/ln: 37:1 determined by GC analysis).

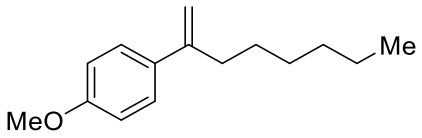
From $PhOSO_2NMe_2$: Following the general procedure **B**, Ligand **1** (34.2 mg, 0.06 mmol, 0.12 equiv), $Ni(cod)_2$ (13.8 mg, 0.05 mmol, 0.10 equiv), Toluene (500 μL), DABCO (280 mg, 2.5 mmol, 5.0 equiv), 1-octene (118 μL, 0.75 mmol, 1.5 equiv), and $PhOSO_2NMe_2$ (82.3 μL, 0.5 mmol, 1.0 equiv) were added to an 8 mL vial. Toluene (500 μL) was used to rinse down the sides of the vial, and triethylsilyl trifluoromethanesulfonate (226 μL, 1.0 mmol, 2.0 equiv) was added, and the reaction was stirred at 60 °C for 24 h in a nitrogen-filled glovebox. When the reaction was done, it was removed from the glovebox. Dry methanol (400 μL) was added to quench, and the reaction was stirred for 20 min at room temperature (capped). Benzene (3 mL) was then added, and an aliquot was taken for GC analysis as described above. The mixture was passed through a silica plug with benzene as the eluent and concentrated. Column chromatography (Biotage 10g HP-sil, pretreated with Et_3N, 100% hexanes) afforded 57.4 mg (61%) of **3a** as a colorless oil (rr pre-purification: 20.1, rr post purification: 19.6, br/ln: 59:1 determined by GC analysis).

^1H NMR (500 MHz, $CDCl_3$) δ 7.44–7.39 (m, 2H), 7.37–7.30 (m, 2H), 7.29–7.24 (m, 1H), 5.27 (app d, J = 1.5 Hz, 1H), 5.06 (app d, J = 1.4 Hz, 1H), 2.50 (t, J = 7.6, 2H), 1.48–1.42 (m, 2H), 1.36–1.23 (m, 6H), 0.88 (t, J = 6.8 Hz, 3H).

^{13}C {^1H} NMR (100 MHz, $CDCl_3$) δ 148.8, 141.5, 128.2, 127.2, 126.1, 112.0, 35.4, 31.7, 29.1, 28.3, 22.7, 14.1.

The ^1H and ^{13}C NMR spectra are in agreement with those reported in the literature.[2]

[2] Blatter, K.; Schlüter, A.-D. *Synthesis*, **1989**, 356–359.



1-methoxy-4-(oct-1-en-2-yl)benzene (3b):

From ArOTf: Following the general procedure **A**, Ligand **1** (34.2 mg, 0.06 mmol, 0.12 equiv), $Ni(cod)_2$ (13.8 mg, 0.05 mmol, 0.10 equiv), THF (500 μL, 1 M), DABCO (168 mg, 1.5 mmol, 3.0 equiv), 1-octene (118 μL, 0.75 mmol, 1.5 equiv), and 4-methoxyphenyl trifluoromethanesulfonate (90.5 μL, 0.5 mmol, 1.0 equiv) were added to an 8 mL vial and stirred at 60 °C for 24 h in a nitrogen-filled glovebox. When the reaction was done, it was removed from the glovebox and 3.0 mL benzene was added. An aliquot was taken for GC analysis as described above. The mixture was passed through a silica plug with benzene as the eluent and concentrated. Column chromatography (Biotage 10g HP-sil, 100% hexanes) afforded 104.3 mg (96%) **3b** as a colorless oil (rr pre-purification: 25.2, rr post-purification: 9.3, br/ln 86:1 determined by GC analysis).

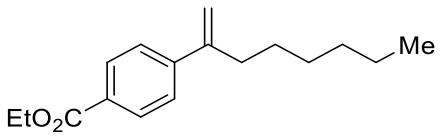
From ArCl: Following the general procedure **B**, Ligand **1** (34.2 mg, 0.06 mmol, 0.12 equiv), $Ni(cod)_2$ (13.8 mg, 0.05 mmol, 0.10 equiv), Toluene (500 μL), DABCO (280 mg, 2.5 mmol, 5.0 equiv), 1-octene (118 μL, 0.75 mmol, 1.5 equiv), and 4-methoxy chlorobenzene (61.2 μL, 0.5 mmol, 1.0 equiv) were added to an 8 mL vial. Toluene (500 μL) was used to rinse down the sides of the vial, and triethylsilyl trifluoromethanesulfonate (226 μL, 1.0 mmol, 2.0 equiv) was added, and the reaction was stirred at 60 °C for 48 h in a nitrogen-filled glovebox. When the reaction was done, it was removed from the glovebox. Dry methanol (400 μL) was added to quench, and the reaction was stirred for 20 min at room temperature (capped). Benzene (3 mL) was then added, and an aliquot was taken for GC analysis as described above. The mixture was passed through a

silica plug with benzene as the eluent and concentrated. Column chromatography (Biotage 25g HP-sil, pretreated with Et_3N, 100% hexanes) afforded 69.6 mg (64%) of **3b** as a colorless oil (rr pre-purification: 26.1, rr post purification: 22.7, br/ln: 84:1 determined by GC analysis).

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.38–7.35 (m, 2H), 6.89–6.85 (m, 2H), 5.20 (d, *J* = 1.6 Hz, 1H), 4.97 (d, *J* = 1.4 Hz, 1H), 3.82 (s, 3H), 2.47 (td, *J* = 7.6, 1.2 Hz, 2H), 1.49–1.40 (m, 2H), 1.35–1.25 (m, 6H), 0.88 (t, *J* = 6.9 Hz, 3H).

^{13}C {^{1}H} NMR (100 MHz, $CDCl_3$) δ 158.9, 148.0, 133.9, 127.1, 113.6, 110.4, 55.2, 35.4, 31.7, 29.0, 28.3, 22.6, 14.1

The ^{1}H and ^{13}C NMR spectra are in agreement with those reported in the literature.[3]



Ethyl 4-(oct-1-en-2-yl)benzoate (3c):

From ArOTf: Following the general procedure **A**, Ligand **1** (34.2 mg, 0.06 mmol, 0.12 equiv), $Ni(cod)_2$ (13.8 mg, 0.05 mmol, 0.10 equiv), THF (500 μL, 1 M), DABCO (168 mg, 1.5 mmol, 3.0

[3] Shirakawa, F.; Imazaki, Y.; Hayashi, T. *Chem. Lett.* **2008**, *37*, 654–655.

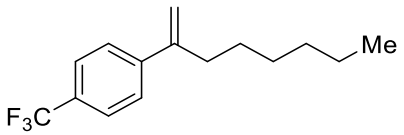
equiv), 1-octene (118 μL, 0.75 mmol, 1.5 equiv), and 4-CO_2Et phenyl trifluoromethanesulfonate (108.1 μL, 0.5 mmol, 1.0 equiv) were added to an 8 mL vial and stirred at 60 °C for 48 h in a nitrogen-filled glovebox. When the reaction was done, it was removed from the glovebox and 3.0 mL benzene was added. An aliquot was taken for GC analysis as described above. The mixture was passed through a silica plug with benzene as the eluent and concentrated. Column chromatography (Biotage 10g HP-sil pretreated with Et_3N, 0–4% ethyl acetate/hexanes) afforded 95.1 mg (73%) **3c** as a colorless oil (rr pre-purification: 46.8, rr post-purification: 38.8, br/ln 83:1 determined by GC analysis).

From ArOMs: Following the general procedure **B**, Ligand **1** (34.2 mg, 0.06 mmol, 0.12 equiv), $Ni(cod)_2$ (13.8 mg, 0.05 mmol, 0.10 equiv), Toluene (500 μL), DABCO (280 mg, 2.5 mmol, 5.0 equiv), 1-octene (118 μL, 0.75 mmol, 1.5 equiv), and 4-CO_2Et phenyl methanesulfonate (123.1 mg, 0.5 mmol, 1.0 equiv) were added to an 8 mL vial. Toluene (500 μL) was used to rinse down the sides of the vial, and triisopropylsilyl trifluoromethanesulfonate (269 μL, 1.0 mmol, 2.0 equiv) was added, and the reaction was stirred at 60 °C for 72 h in a nitrogen-filled glovebox. When the reaction was done, it was removed from the glovebox. Dry methanol (400 μL) was added to quench, and the reaction was stirred for 20 min at room temperature (capped). Benzene (3 mL) was then added, and an aliquot was taken for GC analysis as described above. The mixture was passed through a silica plug with benzene as the eluent and concentrated. Column chromatography (Biotage 25g HP-sil, pretreated with Et_3N, 0–4% ethyl acetate/hexanes) afforded 80.6 mg of a 7.4:1 inseparable mixture of the desired product/4-CO_2EtPhOTIPS (54% adjusted yield) of **3c** as a colorless oil (rr pre-purification: 57.1, rr post purification: 57.1, br/ln: 107:1 determined by GC analysis).

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.04–7.97 (m, 2H), 7.49–7.43 (m, 2H), 5.35 (d, *J* = 1.3 Hz, 1H), 5.15 (q, *J* = 1.4 Hz, 1H), 4.38 (q, *J* = 7.2 Hz, 2H), 2.51 (td, *J* = 7.6, 1.2 Hz, 2H), 1.46–1.41 (m, 2H), 1.40 (t, *J* = 7.1 Hz, 3H), 1.34–1.25 (m, 6H), 0.87 (t, *J* = 6.9 Hz, 3H).

^{13}C {^{1}H} NMR (100 MHz, $CDCl_3$) δ 166.5, 148.0, 146.0, 129.5, 129.2, 126.0, 113.8, 60.8, 35.2, 31.6, 28.9, 28.1, 22.6, 14.3, 14.0.

The ^{1}H and ^{13}C NMR spectra are in agreement with those reported in the literature.[4]



1-(Oct-1-en-2-yl)-4-(trifluoromethyl)benzene (3d):

From ArOTf: Following the general procedure **A**, Ligand **1** (51.3 mg, 0.09 mmol, 0.18 equiv), $Ni(cod)_2$ (20.6 mg, 0.075 mmol, 0.15 equiv), THF (500 μL, 1 M), DABCO (168 mg, 1.5 mmol, 3.0 equiv), 1-octene (236 μL, 1.5 mmol, 3.0 equiv), and 4-trifluoromethylphenyl trifluoromethanesulfonate (96.0 μL, 0.5 mmol, 1.0 equiv) were added to an 8 mL vial and stirred at 60 °C for 48 h in a nitrogen-filled glovebox. When the reaction was done, it was removed from the glovebox and 3.0 mL benzene was added. An aliquot was taken for GC analysis as described

[4] Qin, L.; Ren, X.; Lu, Y.; Li, Y.; Zhou, J. *Angew. Chem., Int. Ed.* **2012**, *51*, 5915–5919.

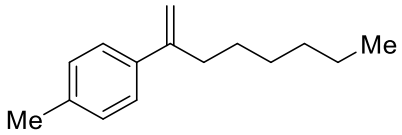
above. The mixture was passed through a silica plug with benzene as the eluent and concentrated. Column chromatography (Biotage 10g HP-sil pretreated with Et_3N, 100% hexanes) afforded 91.0 mg (71%) **3d** as a colorless oil (rr pre-purification: 22.6, rr post-purification: 31.3, br/ln 115:1 determined by GC analysis).

From ArCl: Following the general procedure **B**, Ligand **1** (34.2 mg, 0.06 mmol, 0.12 equiv), $Ni(cod)_2$ (13.8 mg, 0.05 mmol, 0.10 equiv), Toluene (500 µL), DABCO (280 mg, 2.5 mmol, 5.0 equiv), 1-octene (236 µL, 1.5 mmol, 3.0 equiv), and 4-trifluoromethyl chlorobenzene (66.7 µL, 0.5 mmol, 1.0 equiv) were added to an 8 mL vial. Toluene (500 µL) was used to rinse down the sides of the vial, and triisopropylsilyl trifluoromethanesulfonate (269 µL, 1.0 mmol, 2.0 equiv) was added, and the reaction was stirred at 60 °C for 48 h in a nitrogen-filled glovebox. When the reaction was done, it was removed from the glovebox. Dry methanol (400 µL) was added to quench, and the reaction was stirred for 20 min at room temperature (capped). Benzene (3 mL) was then added, and an aliquot was taken for GC analysis as described above. The mixture was passed through a silica plug with benzene as the eluent and concentrated. Column chromatography (Biotage 10g HP-sil, pretreated with Et_3N, 100% hexanes) afforded 85.5g (57%) of **3d** as a colorless oil (rr pre-purification: 19.0, rr post purification: 20.7, br/ln: 164:1 determined by GC analysis). Note: The yield was adjusted to take into account a small amount of TIPSOMe (the result of the methanol quench) remaining inseparable from the product. Although the use of TIPSOTf allows for longer reaction times, the formation of non-volatile TIPSOMe rather than the volatile TESOMe after quenching with MeOH can make purification difficult in the case of very non-polar products.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.58 (d, *J* = 8.2 Hz, 2H), 7.50 (d, *J* = 7.9 Hz, 2H), 5.32 (app s, 1H), 5.16 (d, *J* = 1.4 Hz, 1H), 2.51 (td, *J* = 7.5, 1.2 Hz, 2H), 1.46–1.41 (m, 2H), 1.35–1.21 (m, 6H), 0.88 (t, *J* = 6.9 Hz, 3H).

^{13}C {^{1}H} NMR (100 MHz, $CDCl_3$) δ 147.7, 145.1 (q, *J* = 1.3 Hz), 129.2 (q, *J* = 32.4 Hz), 126.4, 125.2 (q, *J* = 3.8 Hz), 124.3 (q, *J* = 271.8 Hz), 113.9, 35.2, 31.6, 28.9, 28.1, 22.6, 14.0.

The ^{1}H and ^{13}C NMR spectra are in agreement with those reported in the literature.[3]



1-Methyl-4-(oct-1-en-2-yl)benzene (3e):

From ArOTf: Following the general procedure **A**, Ligand **1** (34.2 mg, 0.06 mmol, 0.12 equiv), $Ni(cod)_2$ (13.8 mg, 0.05 mmol, 0.10 equiv), THF (500 μL, 2 M), DABCO (168 mg, 1.5 mmol, 3.0 equiv), 1-octene (118 μL, 0.75 mmol, 1.5 equiv), and 4-methylphenyl trifluoromethanesulfonate (89.5 μL, 0.5 mmol, 1.0 equiv) were added to an 8 mL vial and stirred at 60 °C for 24 h in a nitrogen-filled glovebox. When the reaction was done, it was removed from the glovebox and 3.0 mL benzene was added. An aliquot was taken for GC analysis as described above. The mixture was passed through a silica plug with benzene as the eluent and concentrated. Column chromatography (Biotage 10g HP-sil pretreated with Et_3N, 100% hexanes) afforded 71.6 mg (71%)

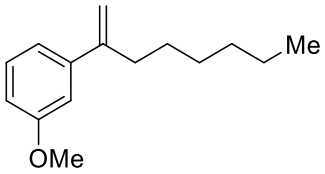
3e as a colorless oil (rr pre-purification: 20.3, rr post-purification: 22.0, br/ln 60:1 determined by GC analysis).

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.32 (d, *J* = 8.2 Hz, 2H), 7.14 (d, *J* = 8.3 Hz, 2H), 5.24 (d, *J* = 1.6 Hz, 1H), 5.01 (d, *J* = 1.5 Hz, 1H), 2.48 (td, *J* = 7.6, 1.3 Hz, 2H), 2.36 (s, 3H), 1.44 (quin, *J* = 7.4 Hz, 2H), 1.35–1.25 (m, 6H), 0.88 (t, *J* = 6.9 Hz, 3H).

^{13}C {^{1}H} NMR (100 MHz, $CDCl_3$) δ 148.6, 138.5, 136.9, 128.9, 126.0, 111.2, 35.4, 31.7, 29.1, 28.3, 22.6, 21.1, 14.1.

IR (ATR, cm^{-1}) 3082, 3025, 2926, 2857, 1625, 1566, 1513, 1457, 1377, 1299, 1186, 1120, 1040, 1019, 890, 823, 733.

HRMS (*m*/*z*) $[M + H]^+$ calcd for $C_{15}H_{22}$, 203.1794; found, 203.1787.



1-Methoxy-3-(oct-1-en-2-yl)benzene (3f):

From ArOTf: Following the general procedure **A**, Ligand **1** (34.2 mg, 0.06 mmol, 0.12 equiv), $Ni(cod)_2$ (13.8 mg, 0.05 mmol, 0.10 equiv), THF (500 μL, 1 M), DABCO (280 mg, 2.5 mmol, 5.0 equiv), 1-octene (199 μL, 1.27 mmol, 2.5 equiv), and 3-methoxy trifluoromethanesulfonate (89.5

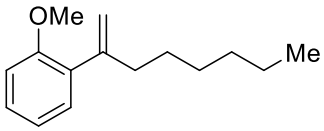
μL, 0.5 mmol, 1.0 equiv) were added to an 8 mL vial and stirred at 60 °C for 24 h in a nitrogen-filled glovebox. When the reaction was done, it was removed from the glovebox and 3.0 mL benzene was added. An aliquot was taken for GC analysis as described above. The mixture was passed through a silica plug with benzene as the eluent and concentrated. Column chromatography (Biotage 25g HP-sil pretreated with Et_3N, 0–1% ethyl acetate/hexanes) afforded 91.2 mg (84%) **3f** as a colorless oil (rr pre-purification: 35.3, rr post-purification: 24.6, br/ln 75:1 determined by GC analysis).

From ArOMs: Following the general procedure **B**, Ligand **1** (34.2 mg, 0.06 mmol, 0.12 equiv), $Ni(cod)_2$ (13.8 mg, 0.05 mmol, 0.10 equiv), Toluene (500 μL), DABCO (280 mg, 2.5 mmol, 5.0 equiv), 1-octene (118 μL, 0.75 mmol, 1.5 equiv), and 3-methoxyphenyl methanesulfonate (77.9 μL, 0.5 mmol, 1.0 equiv) were added to an 8 mL vial. Toluene (500 μL) was used to rinse down the sides of the vial, and triethylsilyl trifluoromethanesulfonate (226 μL, 1.0 mmol, 2.0 equiv) was added, and the reaction was stirred at 60 °C for 48 h in a nitrogen-filled glovebox. When the reaction was done, it was removed from the glovebox. Dry methanol (400 μL) was added to quench, and the reaction was stirred for 20 min at room temperature (capped). Benzene (3 mL) was then added, and an aliquot was taken for GC analysis as described above. The mixture was passed through a silica plug with benzene as the eluent and concentrated. Column chromatography (Biotage 25g HP-sil pretreated with Et_3N, 0–2% ethyl acetate/hexanes) afforded 64.9 mg (59%) **3f** as a colorless oil (rr pre-purification: 37.1, rr post-purification: 34.1, br/ln 112:1 determined by GC analysis).

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.25 (t, *J* = 8.0 Hz, 1H), 7.01 (dt, *J* = 7.7, 1.2 Hz, 1H), 6.95 (dd, *J* = 2.6, 1.7 Hz, 1H), 6.83 (ddd, *J* = 8.2, 2.6, 0.9 Hz, 1H), 5.27 (d, *J* = 1.5 Hz, 1H), 5.06 (d, *J* = 1.5 Hz, 1H), 3.84 (s, 3H), 2.48 (td, *J* = 7.6, 1.2 Hz, 2H), 1.45 (quin, *J* = 7.3 Hz, 2H), 1.35–1.25 (m, 6H), 0.88 (t, *J* = 6.9 Hz, 3H).

^{13}C {^{1}H} NMR (100 MHz, $CDCl_3$) δ 159.5, 148.7, 143.1, 129.1, 118.7, 112.4, 112.14, 112.10, 55.2, 35.4, 31.7, 29.0, 28.2, 22.6, 14.1.

The ^{1}H and ^{13}C NMR spectra are in agreement with those reported in the literature.[3]



1-Methoxy-2-(oct-1-en-2-yl)benzene (3g):

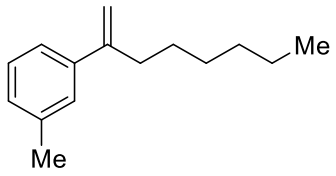
From ArOTf: Following the general procedure **A**, Ligand **1** (34.2 mg, 0.06 mmol, 0.12 equiv), $Ni(cod)_2$ (13.8 mg, 0.05 mmol, 0.10 equiv), THF (500 μL, 1 M), DABCO (168 mg, 1.5 mmol, 3.0 equiv), 1-octene (118 μL, 0.75 mmol, 1.5 equiv), and 2-methoxyphenyl trifluoromethanesulfonate (91.5 μL, 0.5 mmol, 1.0 equiv) were added to an 8 mL vial and stirred at 60 °C for 24 h in a nitrogen-filled glovebox. When the reaction was done, it was removed from the glovebox and 3.0 mL benzene was added. An aliquot was taken for GC analysis as described above. The mixture was passed through a silica plug with benzene as the eluent and concentrated. Column chromatography (Biotage 10g HP-sil, 100% [2% Et_3N in hexanes] to 5% ethyl acetate/95%[2%

Et_3N in hexanes]) afforded 62.7 mg (57%) **3g** as a colorless oil (rr pre-purification: 17.9, rr post-purification: 19.0, br/ln 38:1 determined by GC analysis).

^1H NMR (600 MHz, $CDCl_3$) δ 7.27–7.23 (m, 1H), 7.13 (dd, *J* = 7.4, 1.8 Hz, 1H), 6.92 (td, *J* = 7.4, 1.1 Hz, 1H), 6.88 (dd, *J* = 8.3, 1.0 Hz, 1H), 5.13 (dt, *J* = 2.4, 1.4 Hz, 1H), 5.00 (d, *J* = 2.1 Hz, 1H), 3.83 (s, 3H), 2.47 (t, *J* = 7.6 Hz, 2H), 1.37–1.21 (m, 8H), 0.86 (t, *J* = 7.0 Hz, 3H).

^{13}C {^1H} NMR (100 MHz, $CDCl_3$) δ 156.5, 149.4, 132.4, 130.1, 128.2, 120.4, 113.8, 110.6, 55.4, 36.4, 31.7, 29.0, 28.1, 22.6, 14.1.

The ^1H and ^{13}C NMR spectra are in agreement with those reported in the literature.[3]



1-Methyl-3-(oct-1-en-2-yl)benzene (3h):

From ArOTf: Following the general procedure **A**, Ligand **1** (34.2 mg, 0.06 mmol, 0.12 equiv), $Ni(cod)_2$ (13.8 mg, 0.05 mmol, 0.10 equiv), THF (500 μL, 1 M), DABCO (168 mg, 1.5 mmol, 3.0 equiv), 1-octene (118 μL, 0.75 mmol, 1.5 equiv), and 3-methylphenyl trifluoromethanesulfonate (90.5 μL, 0.5 mmol, 1.0 equiv) were added to an 8 mL vial and stirred at 60 °C for 24 h in a nitrogen-filled glovebox. When the reaction was done, it was removed from the glovebox and 3.0

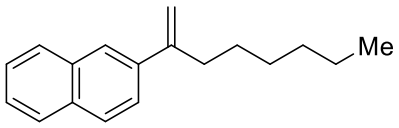
mL benzene was added. An aliquot was taken for GC analysis as described above. The mixture was passed through a silica plug with benzene as the eluent and concentrated. Column chromatography (Biotage 25g HP-sil, pretreated with Et_3N, 100%) afforded 73.7 mg (73%) **3h** as a colorless oil (rr pre-purification: 25.5, rr post-purification: 20.0, br/ln 63:1 determined by GC analysis).

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.25–7.20 (m, 3H), 7.10–7.08 (m, 1H), 5.25 (d, *J* = 1.6 Hz, 1H), 5.04 (d, *J* = 1.5 Hz, 1H), 2.49 (td, *J* = 7.6, 1.2 Hz, 2H), 2.37 (s, 3H), 1.45 (quin, *J* = 7.3 Hz, 2H), 1.36–1.26 (m, 6H), 0.88 (t, *J* = 6.9 Hz, 3H).

^{13}C {^{1}H} NMR (100 MHz, $CDCl_3$) δ 148.9, 141.5, 137.7, 128.1, 128.0, 126.9, 123.2, 111.8, 35.4, 31.7, 29.0, 28.3, 22.6, 21.5, 14.1.

IR (ATR, cm^{-1}) 3028, 2927, 2858, 2360, 2338, 1602, 1582, 1488, 1459, 1426, 1216, 1144, 892, 790, 722.

HRMS (*m*/*z*) $[M + H]^+$ calcd for $C_{15}H_{22}$, 203.1794; found, 203.1794.



2-(Oct-1-en-2-yl)naphthalene (3i):

From ArOTf: Following the general procedure **A**, Ligand **1** (34.2 mg, 0.06 mmol, 0.12 equiv), $Ni(cod)_2$ (13.8 mg, 0.05 mmol, 0.10 equiv), THF (500 μL, 1 M), DABCO (168 mg, 1.5 mmol, 3.0 equiv), 1-octene (118 μL, 0.75 mmol, 1.5 equiv), and 2-naphthyl trifluoromethanesulfonate (136.4 mg, 0.5 mmol, 1.0 equiv) were added to an 8 mL vial and stirred at 60 °C for 24 h in a nitrogen-filled glovebox. When the reaction was done, it was removed from the glovebox and 3.0 mL benzene was added. An aliquot was taken for GC analysis as described above. Complete conversion was obtained, so simple filtration through a silica plug with benzene as the eluent afforded 114.0 mg (97%) **3i** as a colorless oil (rr pre-purification: 17.2, rr post-purification: 24.2, br/ln 57:1 determined by GC analysis).

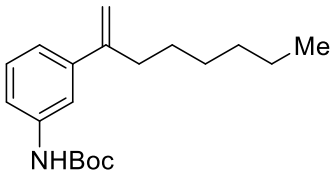
From ArOTs: Following the general procedure **B**, Ligand **1** (34.2 mg, 0.06 mmol, 0.12 equiv), $Ni(cod)_2$ (13.8 mg, 0.05 mmol, 0.10 equiv), Toluene (500 μL), DABCO (280 mg, 2.5 mmol, 5.0 equiv), 1-octene (118 μL, 0.75 mmol, 1.5 equiv), and 2-naphthyl *p*-toluenesulfonate (150.8 mg, 0.5 mmol, 1.0 equiv) were added to an 8 mL vial. Toluene (500 μL) was used to rinse down the sides of the vial, and triethylsilyl trifluoromethanesulfonate (226 μL, 1.0 mmol, 2.0 equiv) was added, and the reaction was stirred at 60 °C for 24 h in a nitrogen-filled glovebox. When the reaction was done, it was removed from the glovebox. Dry methanol (400 μL) was added to quench, and the reaction was stirred for 20 min at room temperature (capped). Benzene (3 mL) was then added, and an aliquot was taken for GC analysis as described above. The mixture was

passed through a silica plug with benzene as the eluent and concentrated. Column chromatography (Biotage 10g HP-sil, pretreated with Et_3N, 100% hexanes) afforded 108.8 mg (72%) of **3i** as a colorless oil (rr pre-purification: 26.1, rr post purification: 23.7, br/ln: 45:1 determined by GC analysis).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87 – 7.81 (m, 4H), 7.61 (dd, *J* = 8.6, 1.8 Hz, 1H), 7.51–7.44 (m, 2H), 5.43 (d, *J* = 1.5 Hz, 1H), 5.18 (d, *J* = 1.4 Hz, 1H), 2.64 (td, *J* = 7.5, 1.2 Hz, 2H), 1.53 (quin, *J* = 7.1 Hz, 2H), 1.42–1.28 (m, 6H), 0.90 (t, *J* = 6.8 Hz, 3H).

^{13}C {^{1}H} NMR (100 MHz, $CDCl_3$) δ 148.6, 138.7, 133.4, 132.7, 128.1, 127.7, 127.5, 126.0, 125.7, 124.7, 124.6, 112.6, 35.4, 31.7, 29.1, 28.3, 22.6, 14.1.

The ^{1}H and ^{13}C NMR spectra are in agreement with those reported in the literature.[2,4]



***Tert*-butyl (3-(oct-1-en-2-yl)phenyl)carbamate (3j):**

From ArOTf: Following the general procedure **A**, Ligand **1** (34.2 mg, 0.06 mmol, 0.12 equiv), $Ni(cod)_2$ (13.8 mg, 0.05 mmol, 0.10 equiv), THF (500 μL, 1 M), DABCO (168 mg, 1.5 mmol, 3.0 equiv), 1-octene (118 μL, 0.75 mmol, 1.5 equiv), and 3-NHBoc-trifluoromethanesulfonate (171.1 mg, 0.50 mmol, 1.0 equiv) were added to an 8 mL vial and stirred at 60 °C for 24 h in a nitrogen-filled glovebox. When the reaction was done, it was removed from the glovebox and 3.0 mL

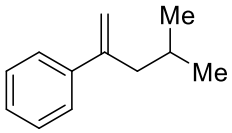
benzene was added. An aliquot was taken for GC analysis as described above. Complete conversion was obtained, so simple filtration through a silica plug with benzene as the eluent and concentrated. Column chromatography (Biotage 10g HP-sil, 4–16% ethyl acetate/hexanes) afforded 77.0 mg (83%) of **3q** as a colorless oil (rr pre-purification: 9.0, rr post purification: 32.5, br/ln: 58:1 determined by GC analysis).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38–7.36 (m, 1H), 7.34–7.29 (m, 1H), 7.25 (t, *J* = 7.8 Hz, 1H), 7.08 (dt, *J* = 7.6, 1.4 Hz, 1H), 6.54 (s, 1H), 5.25 (d, *J* = 1.5 Hz, 1H), 5.05 (dd, *J* = 1.4, 1.4 Hz, 1H), 2.47 (td, *J* = 7.5, 1.2 Hz, 2H), 1.54 (s, 9H), 1.49–1.39 (m, 2H), 1.37–1.22 (m, 6H), 0.88 (t, *J* = 6.8 Hz, 3H).

^{13}C {^{1}H} NMR (100 MHz, $CDCl_3$) δ 152.7, 148.5, 142.5, 138.3, 128.8, 120.9, 117.4, 116.4, 112.3, 80.4, 35.3, 31.6, 29.0, 28.3, 28.2, 22.6, 14.1.

IR (ATR, cm^{-1}) 2929, 2858, 1700, 1606, 1585, 1526, 1489, 1432, 1392, 1367, 1310, 1280, 1233, 1155, 1054, 907, 891, 791, 729.

HRMS (*m*/*z*) $[M + Na]^+$ calcd for $C_{19}H_{29}NO_2$, 326.2091; found, 326.2103.



(4-Methylpent-1-en-2-yl)benzene (3q):

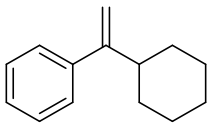
From PhOTf: Following the general procedure **A**, Ligand **1** (34.2 mg, 0.06 mmol, 0.12 equiv), $Ni(cod)_2$ (13.8 mg, 0.05 mmol, 0.10 equiv), THF (500 μL, 1 M), DABCO (168 mg, 1.5 mmol, 3.0 equiv), 4-methyl-1-pentene (94.9 μL, 0.75 mmol, 1.5 equiv), and phenyl trifluoromethanesulfonate (81 μL, 0.5 mmol, 1.0 equiv) were added to an 8 mL vial and stirred at 60 °C for 24 h in a nitrogen-filled glovebox. When the reaction was done, it was removed from the glovebox and 3.0 mL benzene was added. An aliquot was taken for GC analysis as described above. Complete conversion was obtained, so simple filtration through a silica plug with benzene as the eluent afforded 79.8 mg (99%) **3q** as a colorless oil (rr pre-purification: 31.8, rr post-purification: 29.9, br/ln 51:1 determined by GC analysis).

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.40 (m, 2H), 7.33 (app dd, *J* = 8.4, 6.8 Hz, 2H), 7.29–7.25 (m, 1H), 5.27 (d, *J* = 1.8 Hz, 1H), 5.04 (d, *J* = 1.5 Hz, 1H), 2.40 (dd, *J* = 7.3, 1.1 Hz, 2H), 1.67 (app septet, *J* = 6.8 Hz, 1H), 0.88 (d, *J* = 6.6 Hz, 6H).

^{13}C {^{1}H} NMR (100 MHz, $CDCl_3$) δ 147.8, 141.5, 128.2, 127.2, 126.3, 113.4, 45.1, 26.4, 22.4.

The ^{1}H and ^{13}C NMR spectra are in agreement with those reported in the literature.[5]

[6] Limmert, M. E.; Roy, A. H.; Hartwig, J. F. *J. Org. Chem.* **2005**, *70*, 9364–9370.



(1-Cyclohexylvinyl)benzene (3r):

From PhOTf: Following the general procedure **A**, Ligand **1** (34.2 mg, 0.06 mmol, 0.12 equiv), $Ni(cod)_2$ (13.8 mg, 0.05 mmol, 0.10 equiv), THF (500 µL, 1 M), DABCO (168 mg, 1.5 mmol, 3.0 equiv), vinylcyclohexane (103 µL, 0.75 mmol, 1.5 equiv), and phenyl trifluoromethanesulfonate (81 µL, 0.5 mmol, 1.0 equiv) were added to an 8 mL vial and stirred at 60 °C for 48 h in a nitrogen-filled glovebox. When the reaction was done, it was removed from the glovebox and 3.0 mL benzene was added. An aliquot was taken for GC analysis as described above. The mixture was passed through a silica plug with benzene as the eluent and concentrated. Column chromatography (Biotage 10g HP-sil, 100% hexanes) afforded 77.0 mg (83%) of **3r** as a colorless oil (rr pre-purification: 46.1, rr post purification: 57.9, br/ln: 81:1 determined by GC analysis).

From PhOMs: Following the general procedure **B**, Ligand **1** (34.2 mg, 0.06 mmol, 0.12 equiv), $Ni(cod)_2$ (13.8 mg, 0.05 mmol, 0.10 equiv), Toluene (500 µL), DABCO (280 mg, 2.5 mmol, 5.0 equiv), vinylcyclohexane (103 µL, 0.75 mmol, 1.5 equiv), and phenyl methanesulfonate (86.4 mg, 0.5 mmol, 1.0 equiv) were added to an 8 mL vial. Toluene (500 µL) was used to rinse down the sides of the vial, and triethylsilyl trifluoromethanesulfonate (226 µL, 1.0 mmol, 2.0 equiv) was added, and the reaction was stirred at 60 °C for 48 h in a nitrogen-filled glovebox. When the reaction was done, it was removed from the glovebox. Dry methanol (400 µL) was added to quench, and the reaction was stirred for 20 min at room temperature (capped). Benzene (3 mL) was then added, and an aliquot was taken for GC analysis as described above. The mixture was

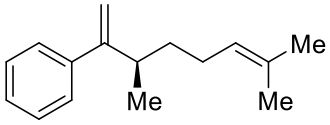
passed through a silica plug with benzene as the eluent and concentrated. Column chromatography (Biotage 10g HP-sil, pretreated with Et_3N, 100% hexanes) afforded 52.9 mg (57%) of **3r** as a colorless oil (rr pre-purification: 28.4, rr post purification: 33.9, br/ln: 55:1 determined by GC analysis).

From PhOTs: Following the general procedure **B**, Ligand **1** (34.2 mg, 0.06 mmol, 0.12 equiv), $Ni(cod)_2$ (13.8 mg, 0.05 mmol, 0.10 equiv), Toluene (500 µL), DABCO (280 mg, 2.5 mmol, 5.0 equiv), vinylcyclohexane (103 µL, 0.75 mmol, 1.5 equiv), and phenyl *p*-toluenesulfonate (123.5 mg, 0.5 mmol, 1.0 equiv) were added to an 8 mL vial. Toluene (500 µL) was used to rinse down the sides of the vial, and triethylsilyl trifluoromethanesulfonate (226 µL, 1.0 mmol, 2.0 equiv) was added, and the reaction was stirred at 60 °C for 24 h in a nitrogen-filled glovebox. When the reaction was done, it was removed from the glovebox. Dry methanol (400 µL) was added to quench, and the reaction was stirred for 20 min at room temperature (capped). Benzene (3 mL) was then added, and an aliquot was taken for GC analysis as described above. The mixture was passed through a silica plug with benzene as the eluent and concentrated. Column chromatography (Biotage 10g HP-sil, pretreated with Et_3N, 100% hexanes) afforded 49.0 mg (53%) of **3r** as a colorless oil (rr pre-purification: 37.8, rr post purification: 33.9, br/ln: 92:1 determined by GC analysis).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38–7.32 (m, 4H), 7.30–7.26 (m, 1H), 5.16 (d, *J* = 1.4 Hz, 1H), 5.03 (app t, *J* = 1.3 Hz, 1H), 2.45 (t, *J* = 11.5 Hz, 1H), 1.92–1.78 (m, 4H), 1.77–1.72 (m, 1H), 1.41–1.14 (m, 5H).

^{13}C {^{1}H} NMR (100 MHz, $CDCl_3$) δ 155.0, 143.0, 128.1, 127.0, 126.6, 110.3, 42.6, 32.7, 26.8, 26.4.

The ^{1}H and ^{13}C NMR spectra are in agreement with those reported in the literature.[6]



(*R*)-(3,7-Dimethylocta-1,6-dien-2-yl)benzene (3s):

From PhOTf: Following the general procedure **A**, Ligand **1** (34.2 mg, 0.06 mmol, 0.12 equiv), $Ni(cod)_2$ (13.8 mg, 0.05 mmol, 0.10 equiv), THF (500 μL, 1 M), DABCO (168 mg, 1.5 mmol, 3.0 equiv), (–)-β-citronellene (136 μL, 0.75 mmol, 1.5 equiv), and phenyl trifluoromethanesulfonate (81 μL, 0.5 mmol, 1.0 equiv) were added to an 8 mL vial and stirred at 60 °C for 48 h in a nitrogen-filled glovebox. When the reaction was done, it was removed from the glovebox and 3.0 mL benzene was added. An aliquot was taken for GC analysis as described above. The mixture was passed through a silica plug with benzene as the eluent and concentrated. Column chromatography (Biotage 10g HP-sil, 100% hexanes) afforded 64.0 mg (60%) of **3s** as a colorless oil (rr pre-purification: 51.2, rr post purification: 57.8, br/ln: 107:1 determined by GC analysis).

[6] Hansen, A. L.; Ebran, J.-P.; Gøgsig, T. M.; Skrydstrup, T. *J. Org. Chem.* **2007**, *72*, 6464–6472.

From PhCl: Following the general procedure **B**, Ligand **1** (34.2 mg, 0.06 mmol, 0.12 equiv), $Ni(cod)_2$ (13.8 mg, 0.05 mmol, 0.10 equiv), Toluene (500 μL), DABCO (280 mg, 2.5 mmol, 5.0 equiv), (–)-β-citronellene (136 μL, 0.75 mmol, 1.5 equiv), and chlorobenzene (50.9 μL, 0.5 mmol, 1.0 equiv) were added to an 8 mL vial. Toluene (500 μL) was used to rinse down the sides of the vial, and triethylsilyl trifluoromethanesulfonate (226 μL, 1.0 mmol, 2.0 equiv) was added, and the reaction was stirred at 60 °C for 48 h in a nitrogen-filled glovebox. When the reaction was done, it was removed from the glovebox. Dry methanol (400 μL) was added to quench, and the reaction was stirred for 20 min at room temperature (capped). Benzene (3 mL) was then added, and an aliquot was taken for GC analysis as described above. The mixture was passed through a silica plug with benzene as the eluent and concentrated. Column chromatography (Biotage 10g HP-sil, pretreated with Et_3N, 100% hexanes) afforded 49.5 mg (49%) of **3s** as a colorless oil (rr pre-purification: 32.5, rr post purification: 43.0, br/ln: 109:1 determined by GC analysis).

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.36–7.31 (m, 4H), 7.29–7.25 (m, 1H), 5.20 (d, *J* = 1.2 Hz, 1H), 5.09 (ddt, *J* = 8.6, 5.7, 1.5 Hz, 1H), 5.05 (t, *J* = 1.3 Hz, 1H), 2.70 (sextet, *J* = 6.9 Hz, 1H), 2.01 (br s, 2H), 1.68 (d, *J* = 1.3 Hz, 3H), 1.57 (s, 3H) 1.59–1.54 (m, 1H), 1.40–1.29 (m, 1H), 1.14 (d, *J* = 6.8 Hz, 3H).

^{13}C {^{1}H} NMR (100 MHz, $CDCl_3$) δ 154.7, 143.0, 131.4, 128.1, 127.0, 126.7, 124.6, 111.0, 37.5, 36.1, 25.8, 25.7, 20.1, 17.6.

IR (ATR, cm^{-1}) 2964, 2923, 2856, 1625, 1574, 1492, 1451, 1375, 895, 776, 698.

HRMS (*m*/*z*) $[M + H]^+$ calcd for $C_{16}H_{22}$, 215.1794; found, 215.1799.

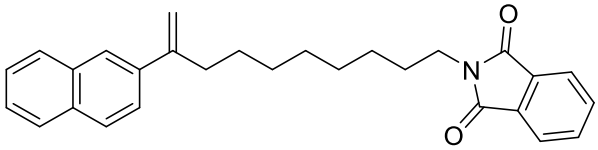
$[\alpha]^{22}_D = -0.107$ (*c* = 0.088, $CHCl_3$)

Conservation of Chirality: Enantiomers were separated by GC Varian Capillary Column CP-Chirasil-Dex CB, 25 m/0.25 mm/0.25 μm. Method: hold 2 min at 50 °C; increase 5 °C/min to 180 °C; hold 15 min at 180 °C

(–)-β-citronellene: 3.59:1 (R)/(S)

Product **3s**: 3.48:1 (R)/(S)

Therefore, there is **97% conservation of chirality** from starting alkene to product



2-(9-(Naphthalen-2-yl)dec-9-en-1-yl)isoindoline-1,3-dione (3t):

From ArOTf: Following the general procedure **A**, Ligand **1** (34.2 mg, 0.06 mmol, 0.12 equiv), $Ni(cod)_2$ (13.8 mg, 0.05 mmol, 0.10 equiv), THF (500 μL, 1 M), DABCO (168 mg, 1.5 mmol, 3.0 equiv), 2-(dec-9-en-1-yl)isoindoline-1,3-dione (207 μL, 0.75 mmol, 1.5 equiv), and 2-naphthyl trifluoromethanesulfonate (138.0 mg, 0.5 mmol, 1.0 equiv) were added to an 8 mL vial and stirred at 60 °C for 24 h in a nitrogen-filled glovebox. When the reaction was done, it was removed from the glovebox and 3.0 mL benzene was added. An aliquot was taken for GC analysis as described

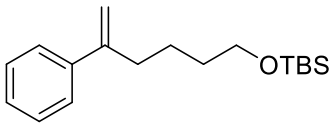
above. The mixture was passed through a silica plug with benzene as the eluent and concentrated. Column chromatography (Biotage 25g HP-sil, 1–10% ethyl acetate with [2% Et_3N in hexanes] as an eluent) afforded 188.5 mg (92%) **3t** as a white solid (rr pre-purification 23.2, rr post-purification: 9.3, br/ln 43:1 determined by GC analysis). Note: Because of the high molecular weight of the product, the following GC method was used: 50 °C for 2 min, 20 °C/min to 320 °C, hold at 320 °C for 25 min.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.86–7.79 (m, 6H), 7.71 (dd, *J* = 5.5, 3.0 Hz, 2H), 7.58 (dd, *J* = 8.4, 1.7 Hz, 1H), 7.46 (tt, *J* = 8.0, 6.6 Hz, 2H), 5.41 (app s, 1H), 5.15 (app s, 1H), 3.67 (t, *J* = 7.3 Hz, 2H), 2.61 (t, *J* = 7.6 Hz, 2H), 1.66 (quin, *J* = 7.5 Hz, 2H), 1.49 (quin, *J* = 7.4 Hz, 2H), 1.39–1.25 (m, 8H).

^{13}C {^{1}H} NMR (100 MHz, $CDCl_3$) δ 168.4, 148.5, 138.6, 133.8, 133.4, 132.7, 132.1, 128.1, 127.7, 127.5, 126.0, 125.6, 124.7, 124.6, 123.1, 112.6, 38.0, 35.3, 29.22, 29.19, 29.1, 28.5, 28.2, 26.8.

IR (ATR, cm^{-1}) 3057, 2925, 2853, 1771, 1706, 1617, 1506, 1465, 1432, 1393, 1365, 1058, 1015, 888, 858, 819, 751, 717.

HRMS (*m*/*z*) $[M + H]^+$ calcd for $C_{28}H_{29}NO_2$, 412.2271; found, 412.2286.



***tert*-Butyldimethyl((5-phenylhex-5-en-1-yl)oxy)silane (3u):**

From PhOTf: Following the general procedure **A**, Ligand **1** (34.2 mg, 0.06 mmol, 0.12 equiv), $Ni(cod)_2$ (13.8 mg, 0.05 mmol, 0.10 equiv), THF (500 µL, 1 M), DABCO (168 mg, 1.5 mmol, 3.0 equiv), the TBS-protected alkenol (199 µL, 0.75 mmol, 1.5 equiv), and phenyl trifluoromethanesulfonate (81 µL, 0.5 mmol, 1.0 equiv) were added to an 8 mL vial and stirred at 60 °C for 24 h in a nitrogen-filled glovebox. When the reaction was done, it was removed from the glovebox and 3.0 mL benzene was added. An aliquot was taken for GC analysis as described above. The mixture was passed through a silica plug with benzene as the eluent and concentrated. Column chromatography (Biotage 25g HP-sil, pretreated with Et_3N, 100% hexanes then subsequent resubjection of mixed fractions to same column to separate product from remaining alkene) afforded 114.8 mg (79%) of **3u** as a colorless oil (rr pre-purification 31.2, rr post purification: 35.6, br/ln: 77:1 determined by GC analysis).

From PhCl: Following the general procedure **B**, Ligand **1** (34.2 mg, 0.06 mmol, 0.12 equiv), $Ni(cod)_2$ (13.8 mg, 0.05 mmol, 0.10 equiv), Toluene (500 µL), DABCO (280 mg, 2.5 mmol, 5.0 equiv), the TBS-protected alkenol (199 µL, 0.75 mmol, 1.5 equiv), and chlorobenzene (50.9 µL, 0.5 mmol, 1.0 equiv) were added to an 8 mL vial. Toluene (500 µL) was used to rinse down the sides of the vial, and triethylsilyl trifluoromethanesulfonate (226 µL, 1.0 mmol, 2.0 equiv) was added, and the reaction was stirred at 60 °C for 24 h in a nitrogen-filled glovebox. When the reaction was done, it was removed from the glovebox. Dry methanol (400 µL) was added to

quench, and the reaction was stirred for 20 min at room temperature (capped). Benzene (3 mL) was then added, and an aliquot was taken for GC analysis as described above. The mixture was passed through a silica plug with benzene as the eluent and concentrated. Column chromatography (Biotage 25g HP-sil, pretreated with Et_3N, 100% hexanes then subsequent resubjection of mixed fractions to same column to separate product from remaining alkene) afforded 91.6 mg (63%) of **3u** as a colorless oil (rr pre-purification: 25.7, rr post purification: 29.8, br/ln: 59:1 determined by GC analysis).

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.42–7.40 (m, 2H), 7.33 (ddd, *J* = 7.7, 6.8, 1.1 Hz, 2H), 7.28–7.25 (m, 1H), 5.28 (d, *J* = 1.5 Hz, 1H), 5.07 (app q, *J* = 1.4 Hz, 1H), 3.60 (t, *J* = 6.4 Hz, 2H), 2.53 (td, *J* = 7.4, 1.3 Hz, 2H), 1.60–1.46 (m, 4H), 0.88 (s, 9H), 0.04 (s, 6H).

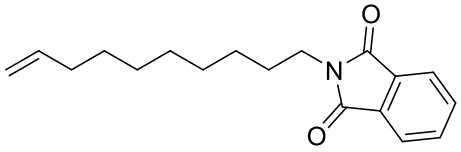
^{13}C {^{1}H} NMR (100 MHz, $CDCl_3$) δ 148.5, 141.3, 128.2, 127.2, 126.1, 112.2, 63.0, 35.1, 32.4, 26.0, 24.4, 18.3, –5.3.

IR (ATR, cm^{-1}) 2929, 2857, 2359, 1627, 1495, 1495, 1472, 1388, 1361, 1253, 1098, 1006, 975, 893, 833, 773, 701, 661.

HRMS (*m*/*z*) $[M + H]^+$ calcd for $C_{18}H_{30}SiO$, 291.2139; found, 291.2137.

Synthesis of Substrates and Authentic Product Samples

Non-Commercially Available Alkenes:



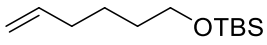
2-(Dec-9-en-1-yl)isoindoline-1,3-dione (S12):[7] To a suspension of 9-decen-1-ol (1.4 mL, 7.85 mmol, 1.0 equiv), phthalimide (1.15 g, 7.85 mmol, 1.0 equiv) and triphenlyphosphine (2.06 g, 7.85 mmol, 1.0 equiv) in THF (24 mL, 0.33 M) at 0 °C was added dropwise diisopropyl azodicarboxylate (DIAD) (1.55 mL, 7.85 mmol, 1.0 equiv). The reaction mixture was allowed to warm to room temperature and stirred overnight. 100 mL Et_2O was added and the slurry was filtered to remove triphenylphosphine oxide. The filtrate was concentrated. Column chromatography (Biotage 50 g HP-sil, 1–18% ethyl acetate/hexanes) afforded 1.60 g (71%) of **S12** as a clear oil. The product was transferred to a vial, sparged with N_2 for 20 min, and dried over 3Å mol sieves overnight before using in the Heck reaction.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.85 (dd, *J* = 5.4, 3.0 Hz, 2H), 7.71 (dd, *J* = 5.4, 3.0 Hz, 2H), 5.80 (ddt, *J* = 16.9, 10.1, 6.7 Hz, 1H), 4.99 (ddt, *J* = 17.2, 1.8, 1.7 Hz, 1H), 4.92 (ddt, *J* = 10.2, 2.2, 1.3 Hz, 1H), 3.68 (t, *J* = 7.3 Hz, 2H), 2.06–2.00 (m, 2H), 1.67 (app quin, *J* = 7.3 Hz, 2H), 1.38–1.25 (m, 10H).

[7] Procedure adopted from: Hattori, K.; Sajiki, H.; Hirota, K. *Tetrahedron* **2000**, *56*, 8433–8441.

^{13}C {^{1}H} NMR (100 MHz, $CDCl_3$) δ 168.4, 139.1, 133.7, 132.1, 123.1, 114.1, 38.0, 33.7, 29.3, 29.1, 29.0, 28.8, 28.5, 26.8.

The ^{1}H and ^{13}C NMR spectra are in agreement with those reported in the literature.[8]



tert-Butyl(hex-5-en-1-yloxy)dimethylsilane (S13): To a solution of 5-hexen-1-ol (2.70 mL, 22.5 mmol, 1.0 equiv) in DMF (22.5 mL, 1 M) was added imidazole (4.59 g, 67.4 mmol, 3.0 equiv) followed by *tert*-butyldimethylsilyl chloride (4.41 g, 29.2 mmol, 1.3 equiv). The reaction mixture was stirred at room temperature overnight. The reaction was quenched by the addition of 20 mL saturated aq. NH_4Cl. Et_2O (80 mL) and H_2O (70 mL) were added, and the layers were separated. The aqueous layer was extracted with Et_2O (2x70 mL), and the combined organic layers were washed with brine (100 mL), dried over $MgSO_4$, filtered, and concentrated. Column chromatography (Biotage 100 g HP-Sil, 0–6% ethyl acetate/hexanes) afforded 3.38 g (70%) of **S13** as a clear oil. The product was transferred to a vial, sparged with N_2 for 20 min, and dried over 3Å mol sieves overnight before using in the Heck reaction.

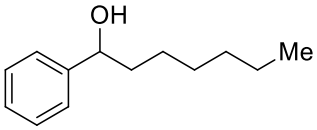
[8] Makado, G.; Morimoto, T.; Sugimoto, Y.; Tsutsumi, K.; Kagawa, N.; Kakiuchi, K. *Adv. Synth. Catal.* **2010**, *352*, 299–304.

^{1}H NMR (600 MHz, $CDCl_3$) δ 5.82 (ddt, *J* = 16.9, 10.1, 6.7 Hz, 1H), 5.01 (ddt, *J* = 17.1, 2.2, 1.6 Hz, 1H), 4.95 (ddt, *J* = 10.2, 2.4, 1.3 Hz, 1H), 3.62 (t, *J* = 6.5 Hz, 2H), 2.07 (tdt, *J* = 7.9, 6.7, 1.4 Hz, 2H), 1.60–1.49 (m, 2H), 1.48–1.39 (m, 2H), 0.90 (s, 9H), 0.06 (s, 6H).

^{13}C {^{1}H} NMR (100 MHz, $CDCl_3$) δ 138.9, 114.3, 63.1, 33.5, 32.3, 26.0, 25.3, 18.4, –5.28.

The ^{1}H and ^{13}C NMR spectra are in agreement with those reported in the literature.[9]

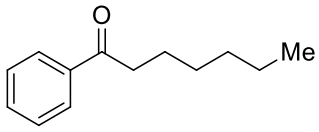
Independent Synthesis of Isomerically Pure Branched Product 3a:



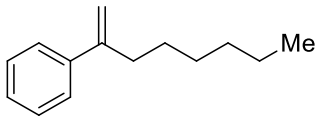
1-Phenylheptan-1-ol (S14): To a solution of 1-heptanal (1.27 mL, 9.12 mmol, 1.0 equiv) in Et_2O (29.4 mL) at 0 °C under nitrogen was added a solution of phenyl lithium (1.8 M in *n*-butyl ether, 7.60 mL, 13.7 mmol, 1.5 equiv). The reaction mixture was stirred at 0 °C for 4.5 h. Saturated aq. NH_4Cl (9 mL) was added and stirred for 15 min. Additional Et_2O (50 mL) and H_2O (50 mL) were added, and the layers were separated. The aqueous layer was extracted with Et_2O (2x70 mL), and the combined organic layers were washed with brine (100 mL), dried over $MgSO_4$, filtered, and

[9] Lebel, H.; Paquet, V. *J. Am. Chem. Soc.* **2004**, *126*, 320–328.

concentrated. Column chromatography (Biotage 50 g HP-Sil, 1–10% ethyl acetate/hexanes) afforded 1.32 g (75%) of the desired alcohol as a clear oil.



1-Phenylheptan-1-one (S15): To a solution of **S14** (1.32g, 6.87 mmol, 1.0 equiv) in CH_2Cl_2 (6.90 mL) was added 2,2,6,6-tetramethyl-1-piperidinyloxy (TEMPO) (109 mg, 0.69 mmol, 0.1 equiv) followed by (diacetoxyiodo)benzene (2.44 g, 7.56 mmol, 1.1 equiv). The reaction was stirred at room temperature for 20 h, after which was added saturated aq. $Na_2S_2O_3$ (5 mL) and allowed to stir an additional 15 min. Additional CH_2Cl_2 (50 mL) and H_2O (50 mL) were added, and the layers were separated. The aqueous layer was extracted with CH_2Cl_2 (2x70 mL), and the combined organic layers were washed with brine (100 mL), dried over $MgSO_4$, filtered, and concentrated. Column chromatography (Biotage 50 g HP-sil, 1–10% ethyl acetate/hexanes) afforded 1.31 g (quant) of the desired ketone as a clear oil.

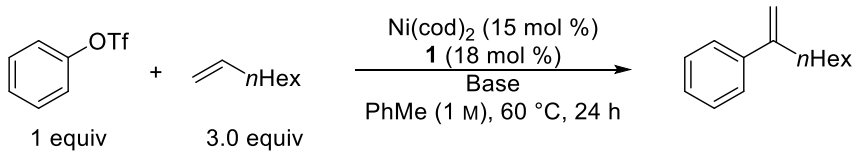


Oct-1-en-2-ylbenzene (3a): To a solution of methyltriphenylphosphonium bromide (2.45 g, 6.87 mmol, 1.0 equiv) in Et_2O (21.5 mL) under nitrogen at room temperature was added *n*-butyl lithium (1.6 M in hexanes, 4.3 mL, 6.87 mmol, 1.0 equiv). The bright orange reaction mixture was stirred

at room temperature for 40 min. **S14**, which had been azeotroped from benzene to remove H_2O was then added slowly, rinsing with an additional amount of Et_2O (4 mL), and stirred at room temperature for 21 h. The reaction mixture was then poured into H_2O (70 mL), and extracted with Et_2O (2x50 mL). The combined organic layers were washed with brine (50 mL), dried over $MgSO_4$, filtered, and concentrated. Column chromatography (25 g HP-Sil Biotage column, 100% hexanes) afforded 931 mg (72%) of the desired alkene **3a** as a clear oil. Characterization details listed above.

Further Reaction Optimization Tables

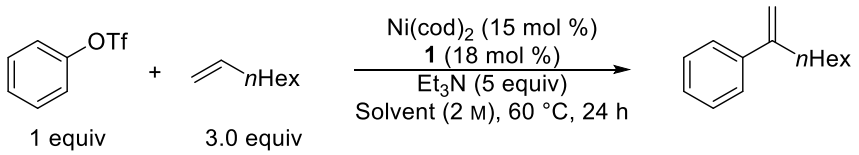
Table S1. Evaluation of different bases.[a]



Entry	Base	Equivalents	Conversion PhOTf (%)	Yield (%)	br/ln	rr
1	Et_3N	5.0	89	86	>100:1	7.5
2	$NMeCy_2$	5.0	84	75	75:1	4.3
3	Morpholine	5.0	46	39	43:1	17.3
4	Et_2NH	5.0	73	67	73:1	24.3
5	DABCO	3.0	99	100	>100:1	32.3
6	Quinuclidine	3.0	99	100	>100:1	37.7
7	$^nBu_3N$	5.0	39	39	64:1	3.0
8	Hünig's Base (DIPEA)	5.0	22	21	90:1	4.8
9	DBU	5.0	23	–[b]	–[b]	–[b]
10	Urotropine	3.0	10	11	61:1	10.5
11	Proton Sponge	3.0	13	16	12:1	7.6
12	Bu_4NOAc	3.0	73	0	–	–
13	$NaHCO_3$	3.0	7	2	–	–
14	K_2CO_3	3.0	7	1	–	–
15	K_3PO_4	3.0	5	2	–	–
16	Cs_2CO_3[c]	3.0	16	0	–	–
17	KO^tBu[c]	5.0	99	0	–	–
18	KHMDS[c]	3.0	99	0	–	–

[a] Conversions and yields determined by GC with dodecane as internal standard [b] Product overlapped with base in chromatogram [c] with dcypb instead of Ligand **1**

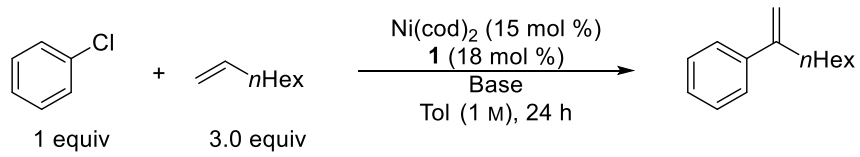
Table S2. Evaluation of different solvents.[a]



Entry	Solvent	Conversion PhOTf (%)	Yield (%)	br/ln	rr
1	Neat	75	79	>100:1	10.7
2	Toluene	89	86	>100:1	7.5
3	Benzene	96	91	>100:1	5.4
4	THF	96	93	>100:1	7.4
5	1,4-Dioxane	97	92	98:1	6.6
6	CH_3CN	8	1	–	–
7	DMF	52	57	>100:1	21.1
8	Et_2O, rt	35	37	>100:1	30.2
9	CH_2Cl_2	2	1	–	–
10	$CHCl_3$[b]	0	0	–	–
11	THF[b]	97	100	82:1	33.7
12	1,4-Dioxane[b]	90	89	52:1	13:1
13	DMF[b]	38	40	71:1	32.8
14	Et_2O[b]	34	33	122:1	45.7

[a] Conversions and yields determined by GC with dodecane as internal standard
[b] DABCO (3 equiv) rather than Et_3N

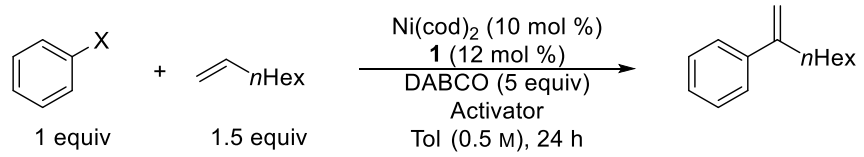
Table S3. Evaluation of counterion exchange reagents for chlorobenzene.[a]



Entry	Additive (1.75 equiv)	Ligand	Base	Temp (°C)	Conversion PhCl (%)	Yield (%)	br/ln
1	TESOTf	PCy_2Ph	Et_3N	rt	49	18	12:1
2	$NaClO_4$	PCy_2Ph	Et_3N	rt	28	4	1.7:1
3	NaOTf	PCy_2Ph	Et_3N	rt	30	3	1.4:1
4	AgOTf	PCy_2Ph	Et_3N	rt	9	0	–
5	TESOTf	PCy_2Ph	Et_3N	60	29	14	23:1
6	$NaClO_4$	dippp	Et_3N	rt	17	10	–
7	NaOTf	dippp	Et_3N	rt	17	11	–
8	AgOTf	dippp	Et_3N	rt	6	0	–
9	$NaBF_4$	dippp	Et_3N	rt	13	0	–
10	$NaPF_6$	dippp	Et_3N	rt	17	12	–
11	$NaBPh_4$	dippp	Et_3N	rt	20	11	–
12	$NaSbF_6$	dippp	Et_3N	rt	15	7	–
13	NaBARF	dippp	Et_3N	rt	17	10	39:1
14	none	dcypb	Et_3N	60	16	1	–
15	TESOTf	dcypb	Et_3N	60	29	21	–
16	TMSOTf	dcypb	Et_3N	60	17	14	–
17	TESOTf	Ligand **1**	DABCO	60	61	64	59:1
18	TESOTf	Ligand **1**	Et_3N	60	22	20	>100:1
19	NaOTf	Ligand **1**	DABCO	60	11	1	–
20	TMSOTf	Ligand **1**	DABCO	60	14	4	–
21	TESOTf (optimized conds[b])	Ligand **1**	DABCO	60	82	87	63:1
22	TESOTf (undistilled – dark brown/ black)	Ligand **1**	DABCO	60	71	72	42:1

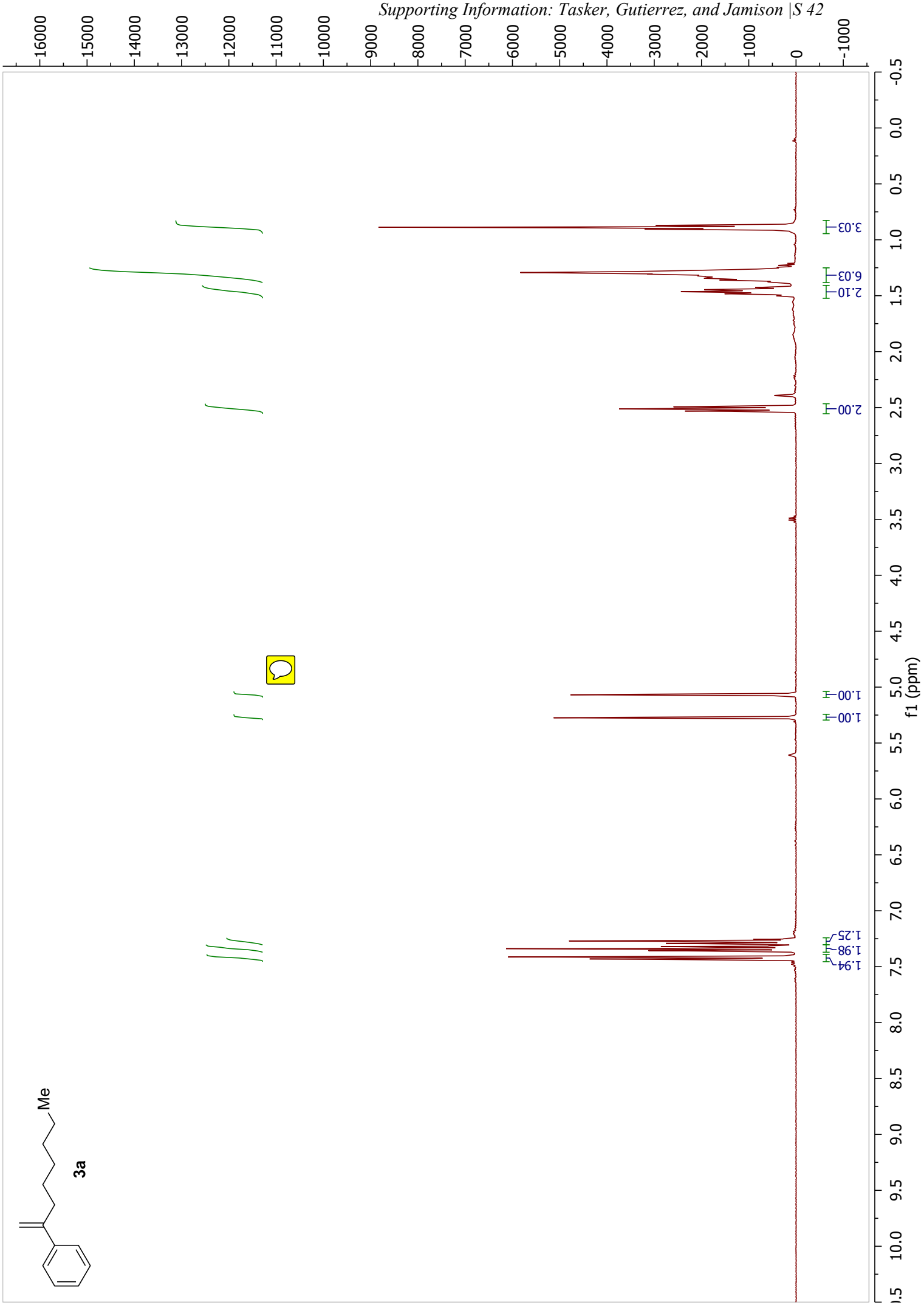
[a] Conversions and yields determined by GC with dodecane as internal standard [b] Optimized conditions: $Ni(cod)_2$ (10 mol %), **1** (12 mol %), DABCO (5 equiv), TESOTf (2 equiv), PhMe (0.5 M), 60 °C, 24 h

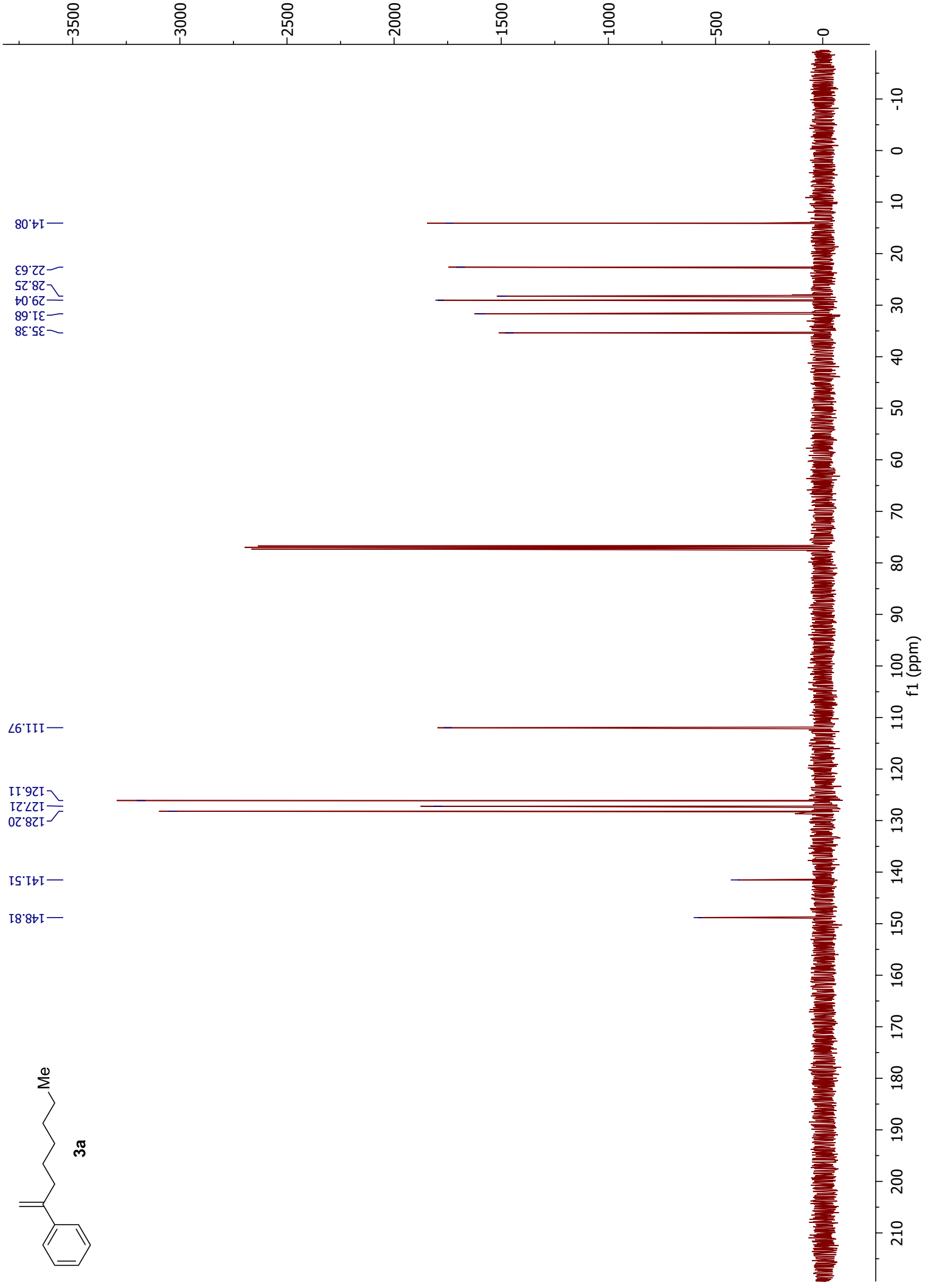
Table S4. Investigation of electrophiles.[a]

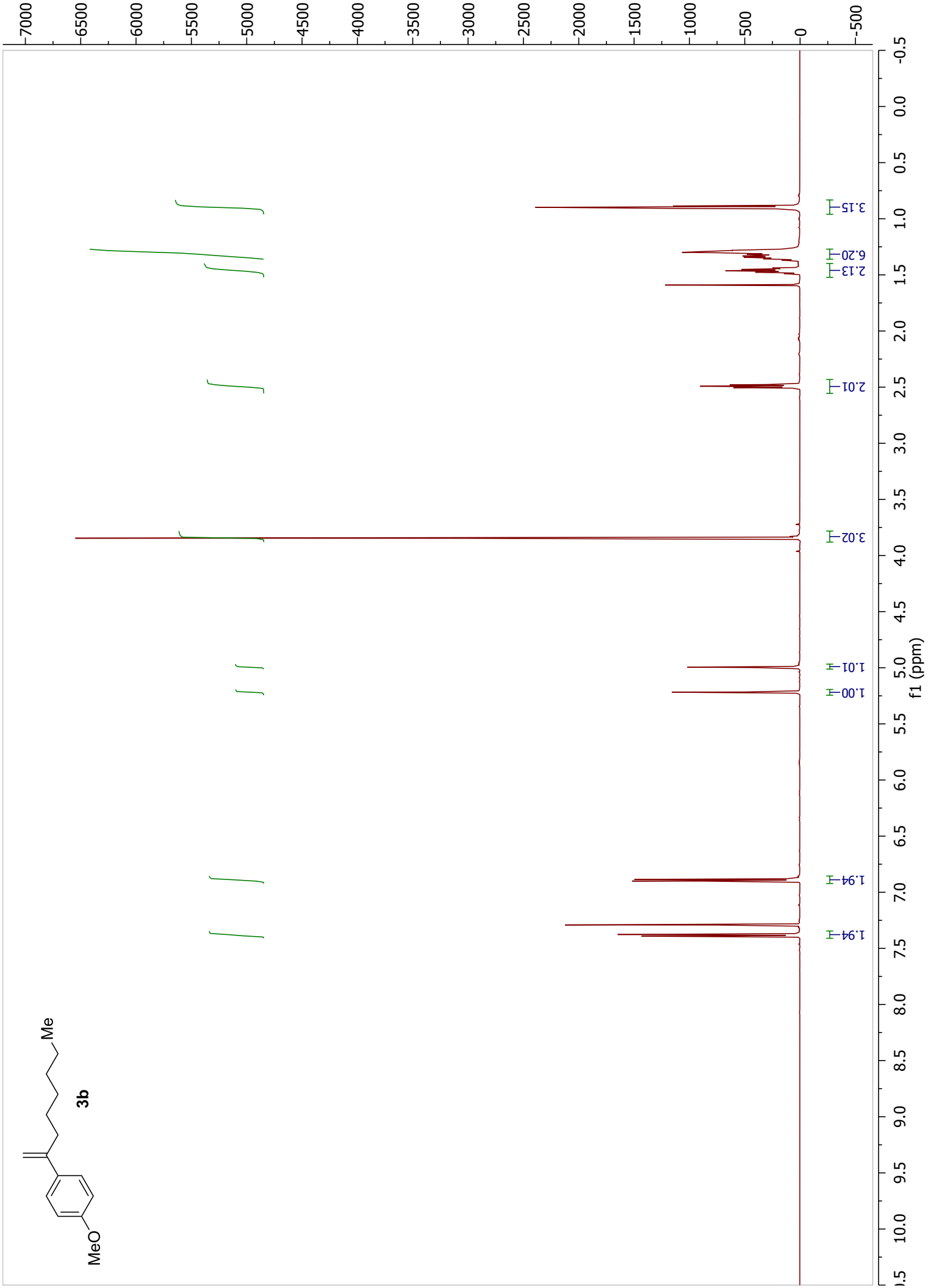


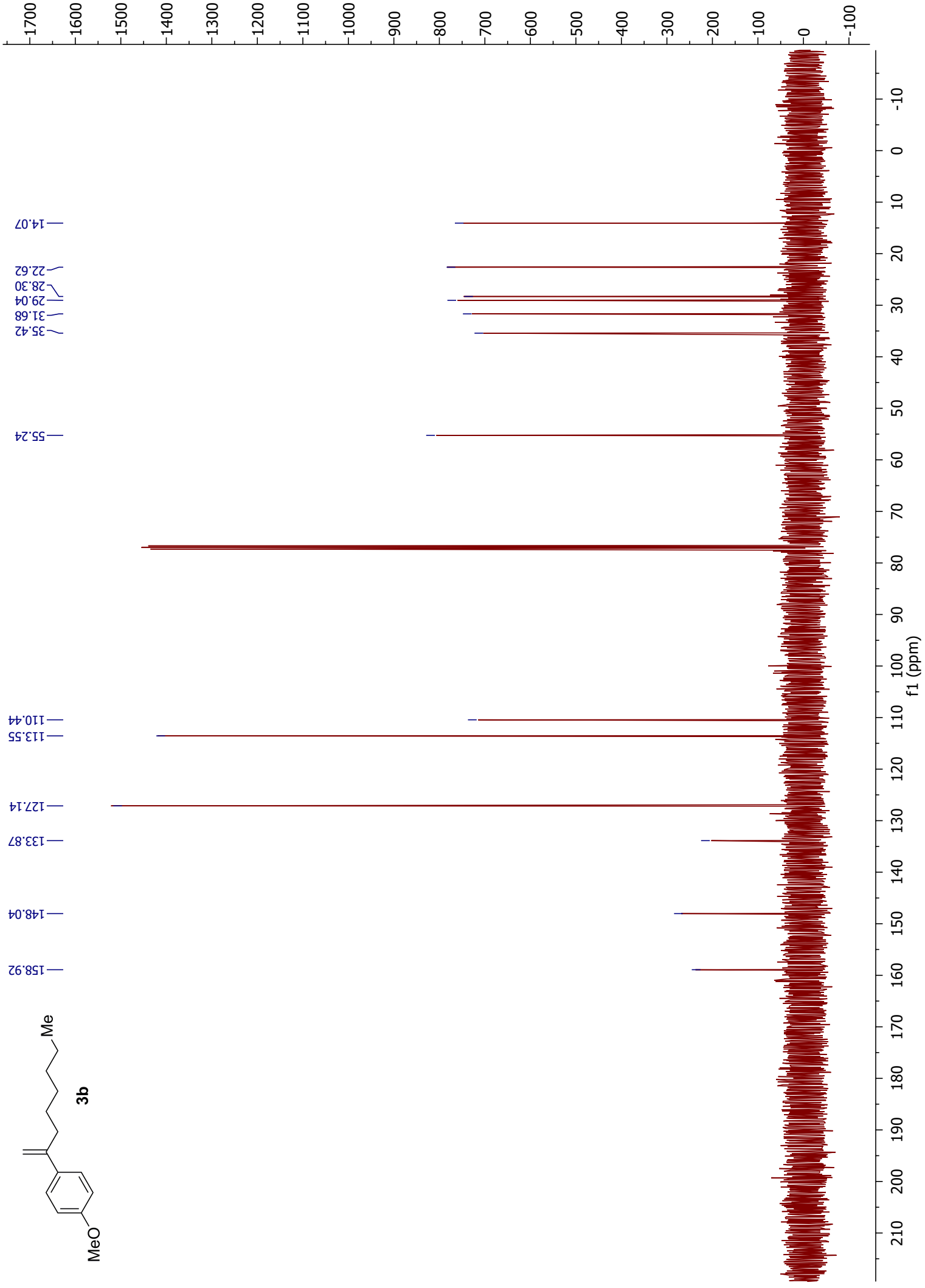
Entry	X	Activator	Yield (%)	br/ln
1	Cl	TESOTf	87	63:1
2	OMs	none	25	–
3	OMs	TESOTf	81	60:1
4	OTs	none	33	83:1
5	OTs	TESOTf	75	39:1
6	OSO_2NMe_2	none	8	–
7	OSO_2NMe_2	TESOTf	65	59:1
8	Br	none	0	–
9	Br	TESOTf	4	13:1
10	Br	Zn dust	1	–
11	I	none	0	–
12	I	TESOTf	2	11:1
13	I	AgOTf	3	2.7:1
14	OMe	TESOTf	0	–
15	OAc	TESOTf	1	–
16	$OCO_2{}^tBu$	TESOTf	0	–
17	F	TESOTf	0	–
18	$OCOCF_3$	TESOTf	4	–

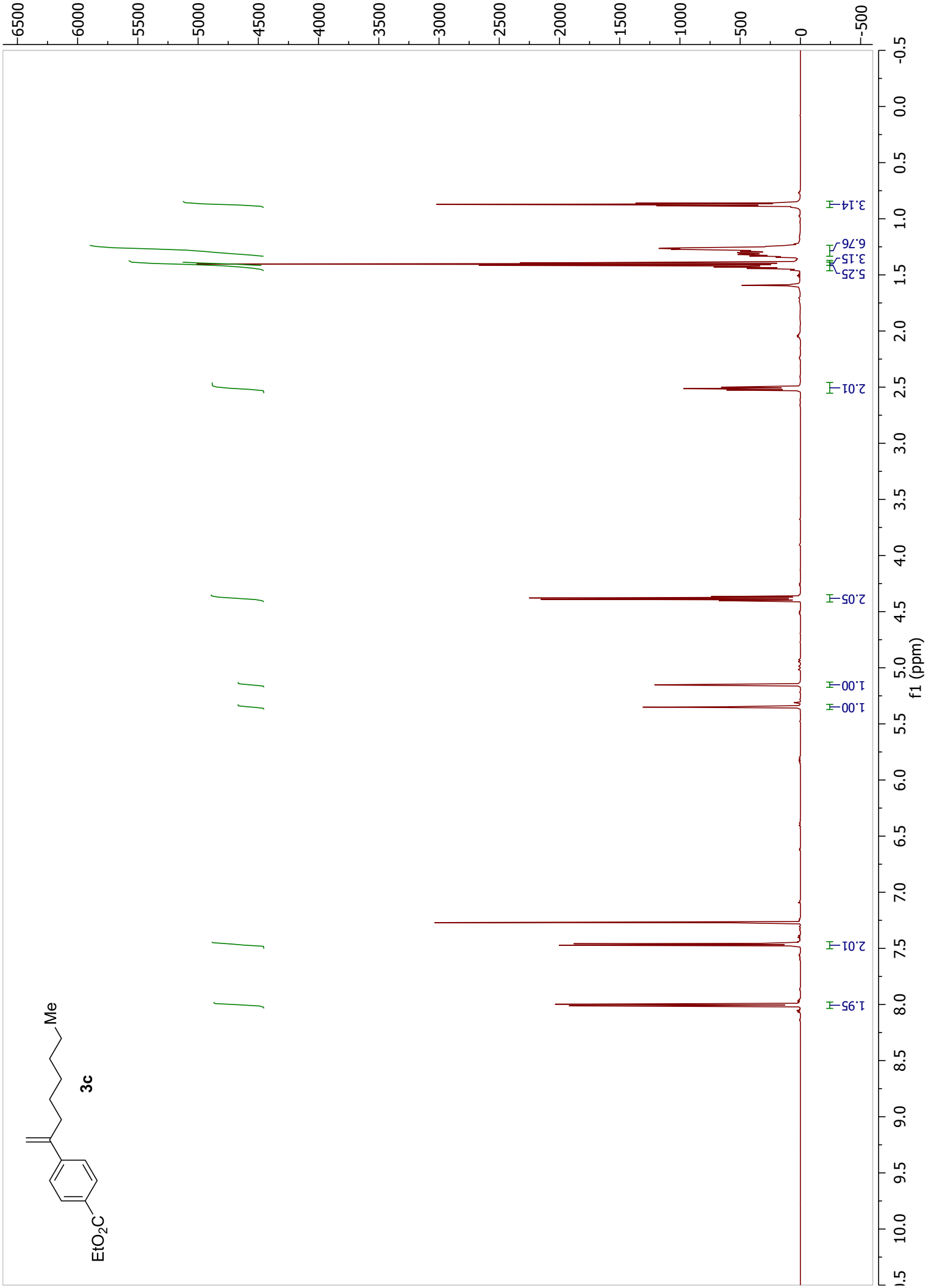
[a]Conversions and yields determined by GC with dodecane as internal standard

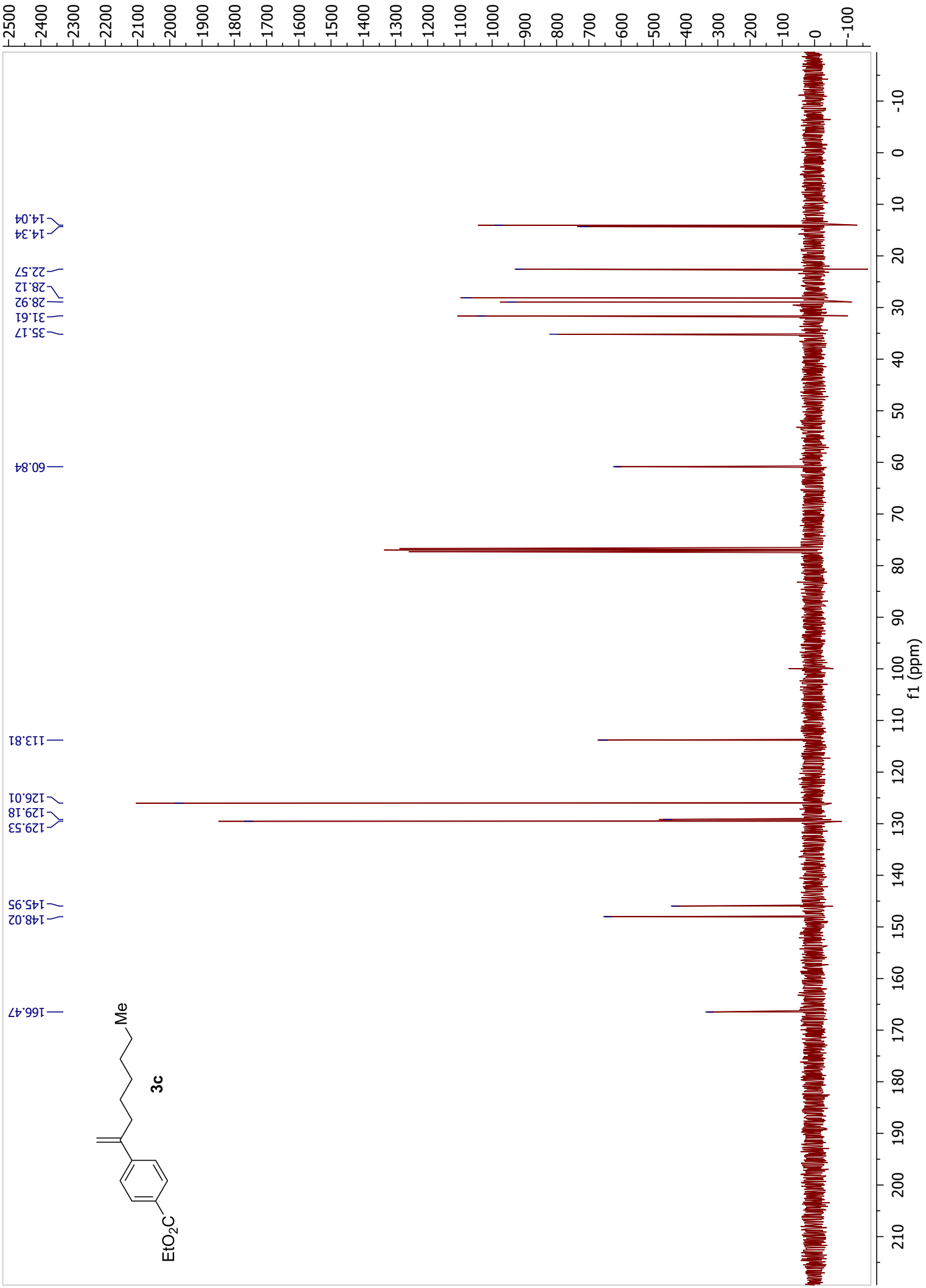

3.03
6.03
2.10
2.00
1.00
1.00
1.25
1.98
1.94
f1 (ppm)
Me
3a

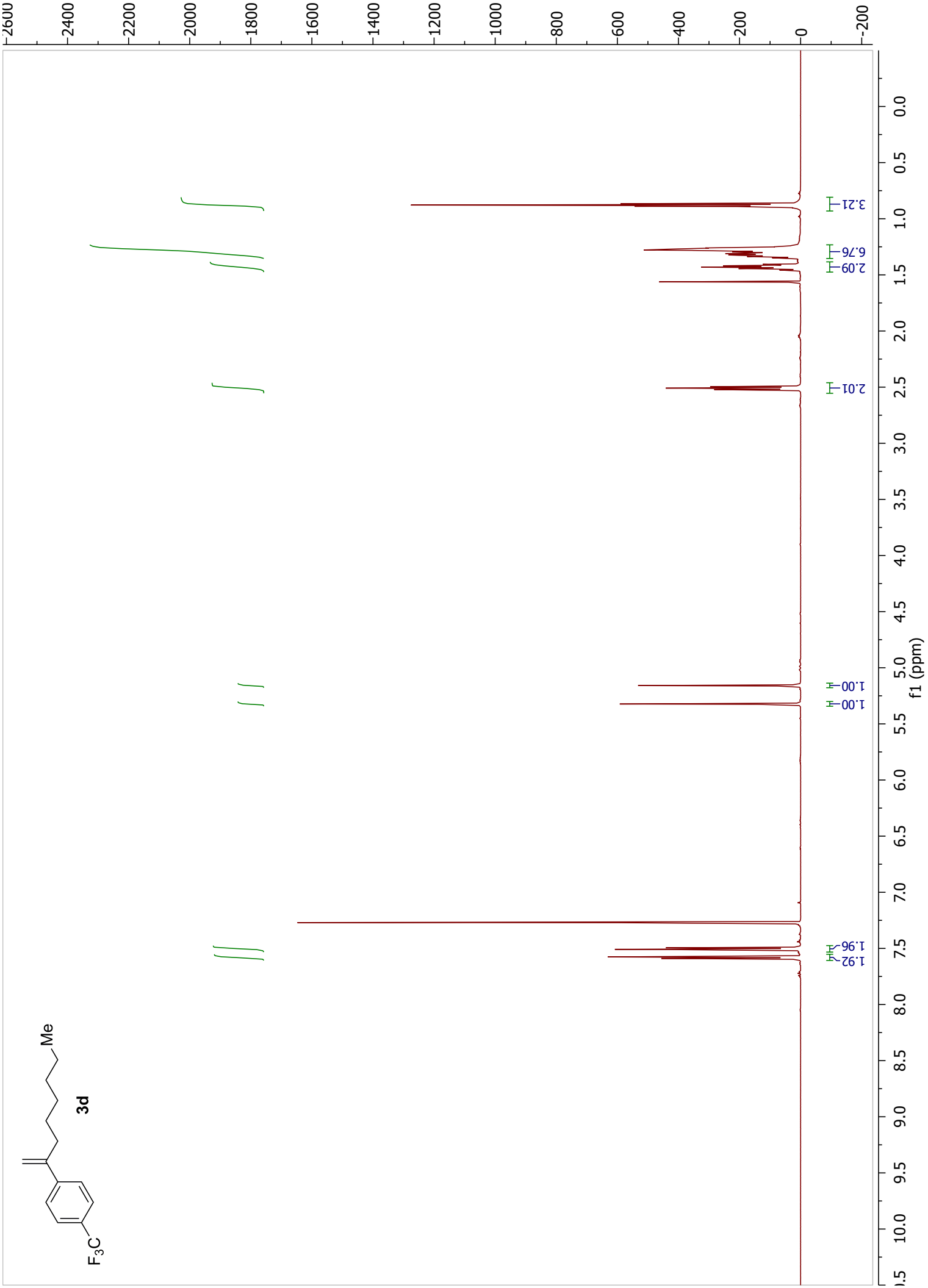

14.08
22.63
28.25
29.04
31.68
35.38
111.97
126.11
127.21
128.20
141.51
148.81
Me
3a
f1 (ppm)

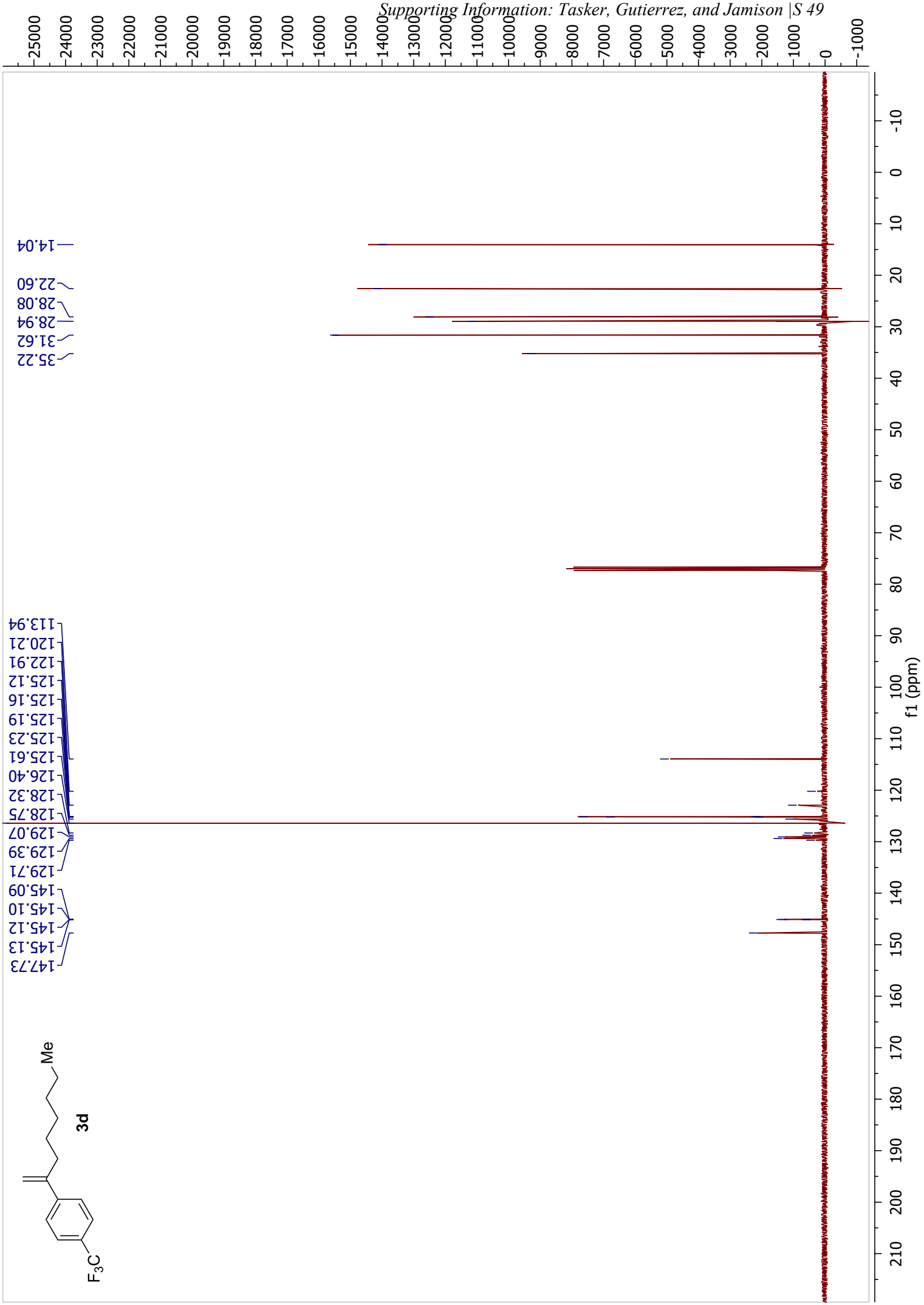

3.15
6.20
2.13
2.01
3.02
1.01
1.00
1.94
1.94
f1 (ppm)
Me
MeO
3b

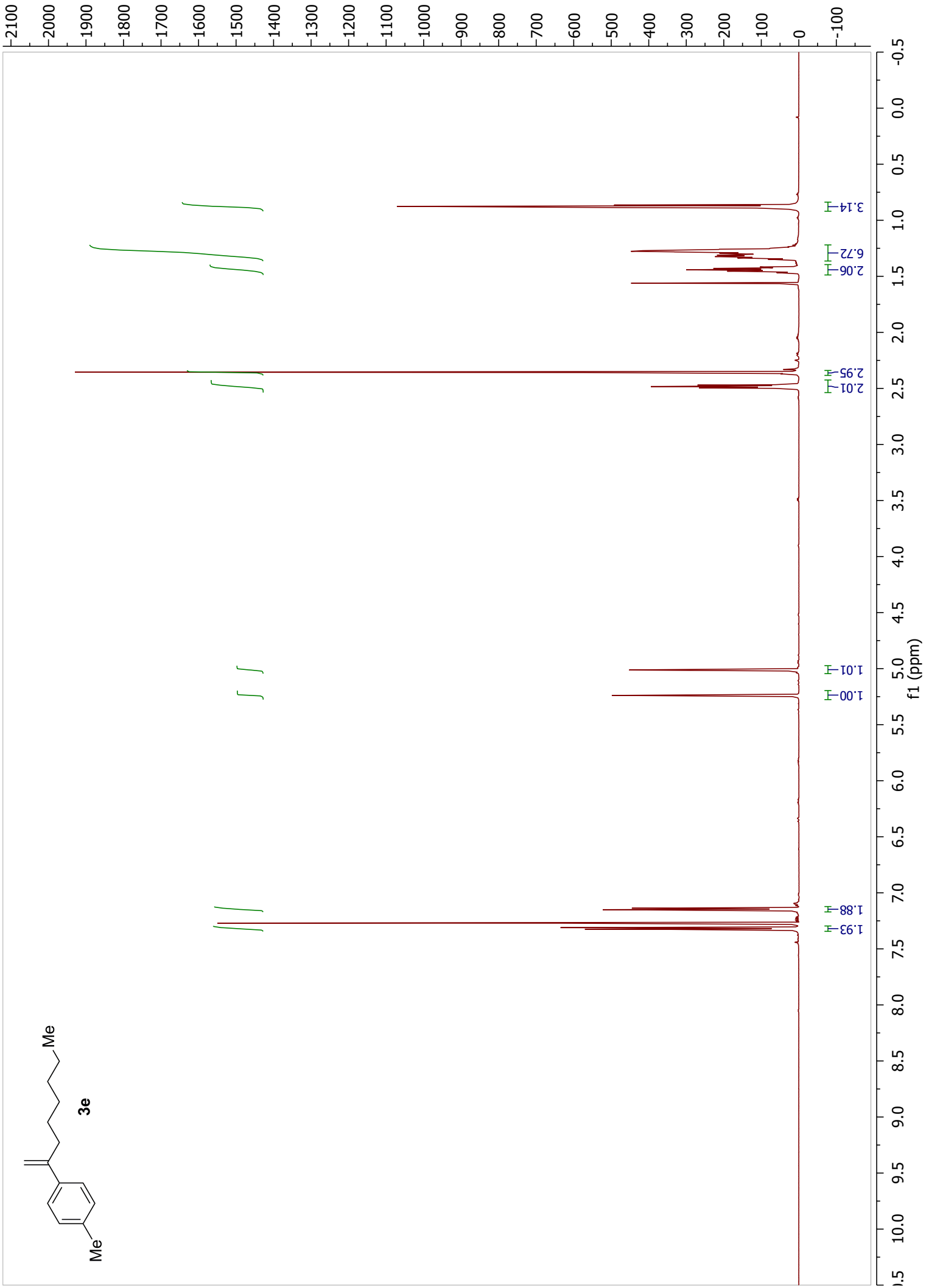

14.07
22.62
28.30
29.04
31.68
35.42
55.24
110.44
113.55
127.14
133.87
148.04
158.92
Me
3b
MeO
f1 (ppm)

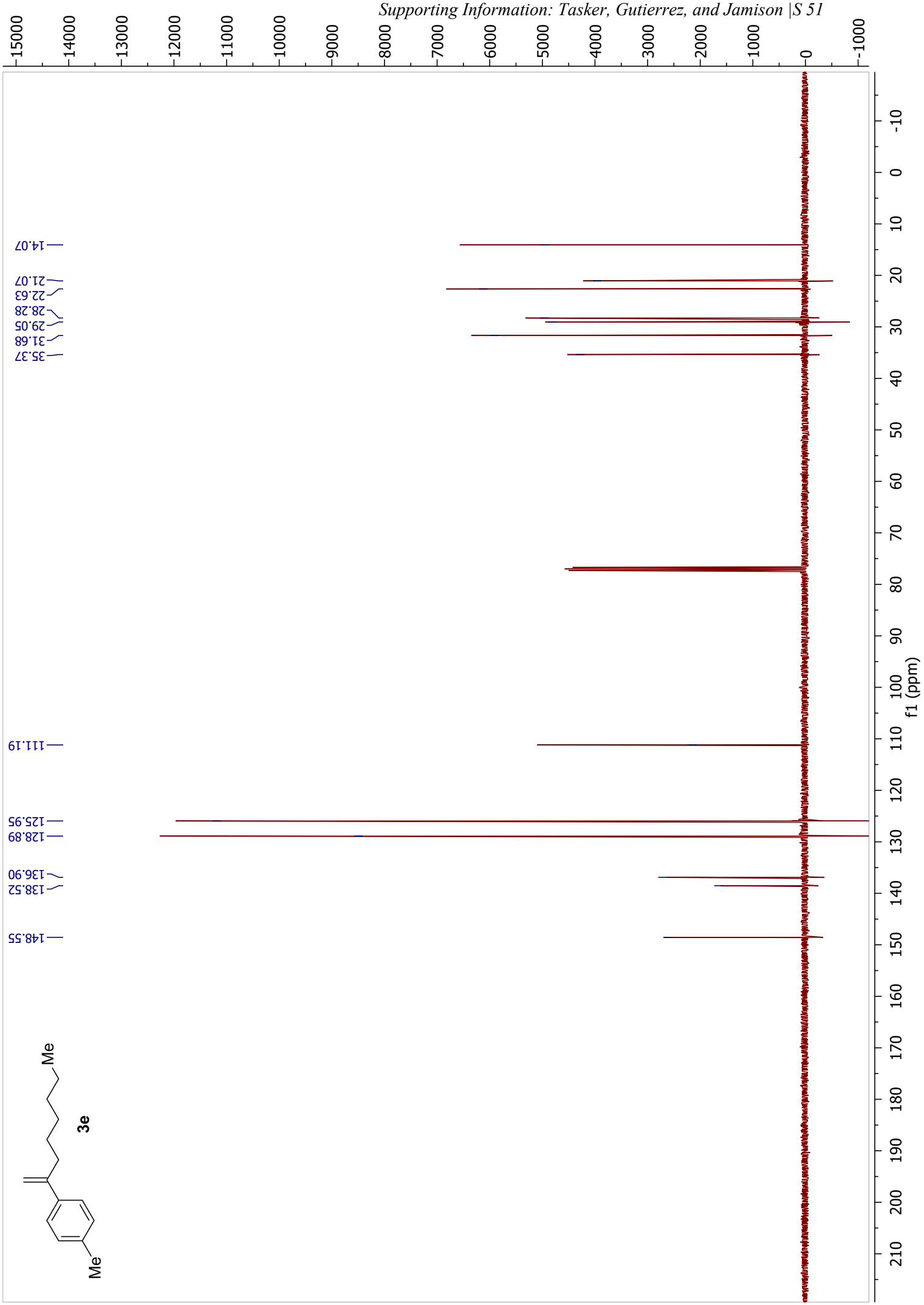

3.14
6.76
3.15
5.25
2.01
2.05
1.00
1.00
2.01
1.95
f1 (ppm)
Me
3c
EtO2C

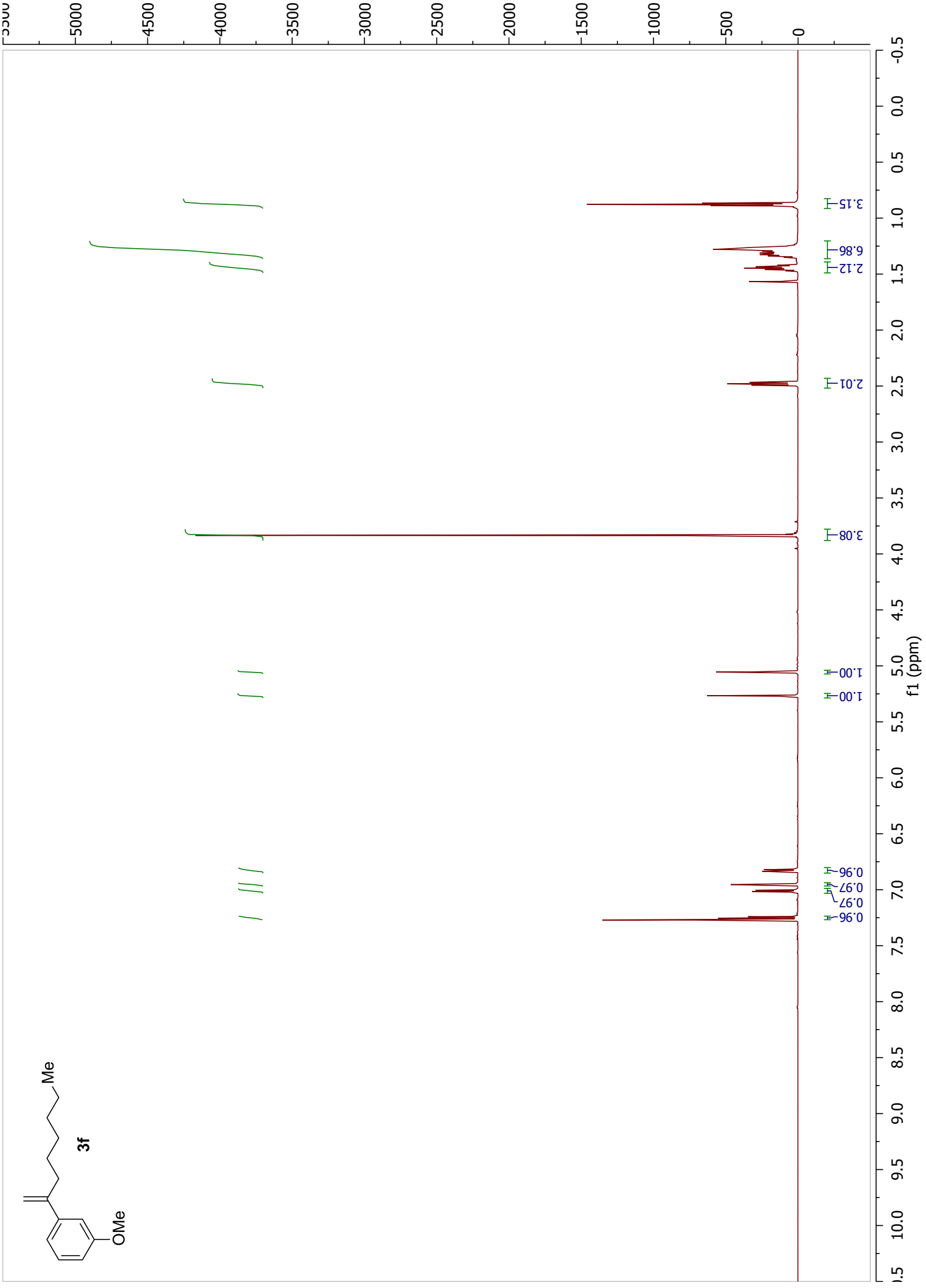

14.04
14.34
22.57
28.12
28.92
31.61
35.17
60.84
113.81
126.01
129.18
129.53
145.95
148.02
166.47
Me
3c
EtO2C
f1 (ppm)

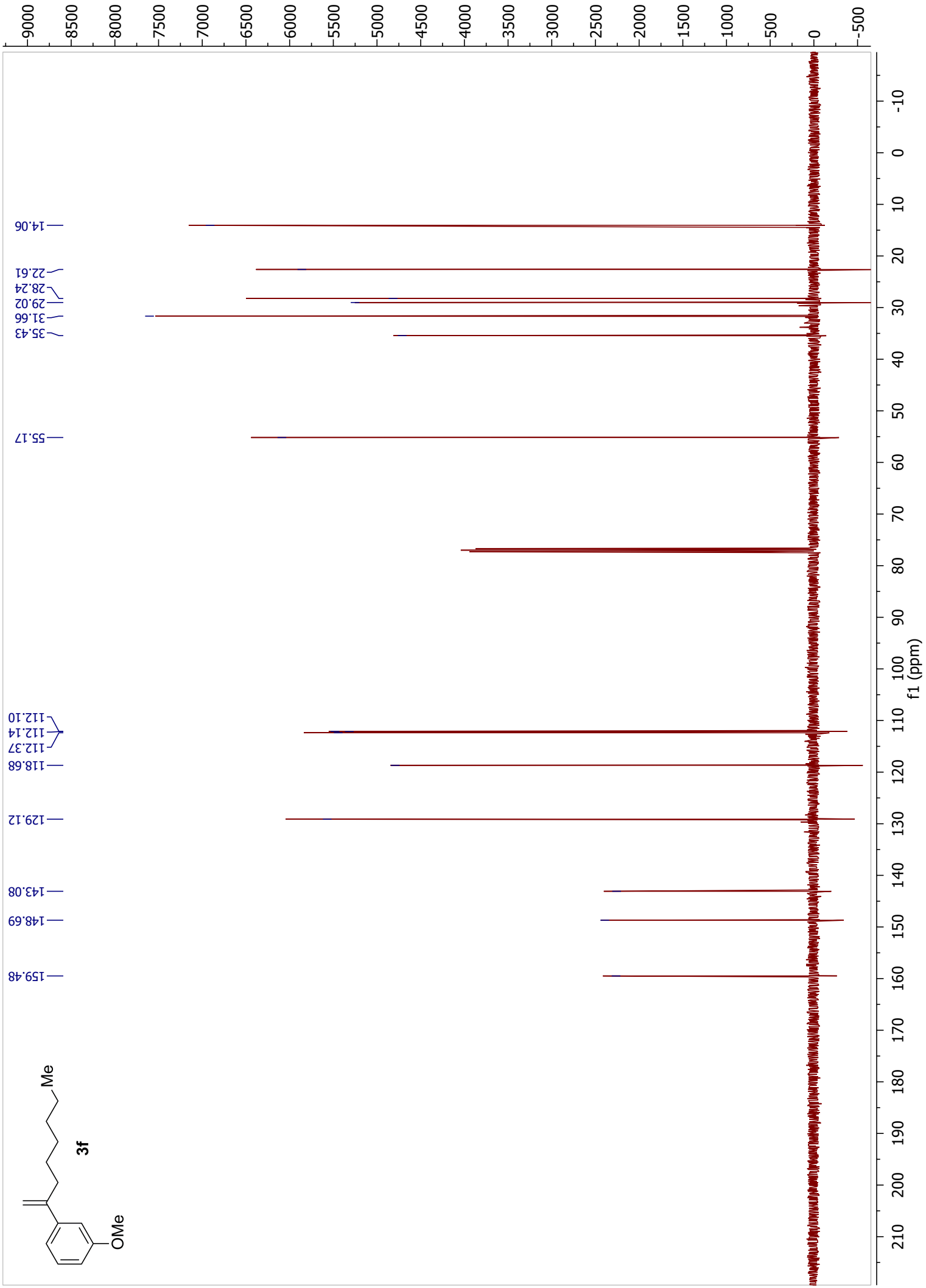

3.21
6.76
2.09
2.01
1.00
1.00
1.96
1.92
f1 (ppm)
Me
3d
F3C

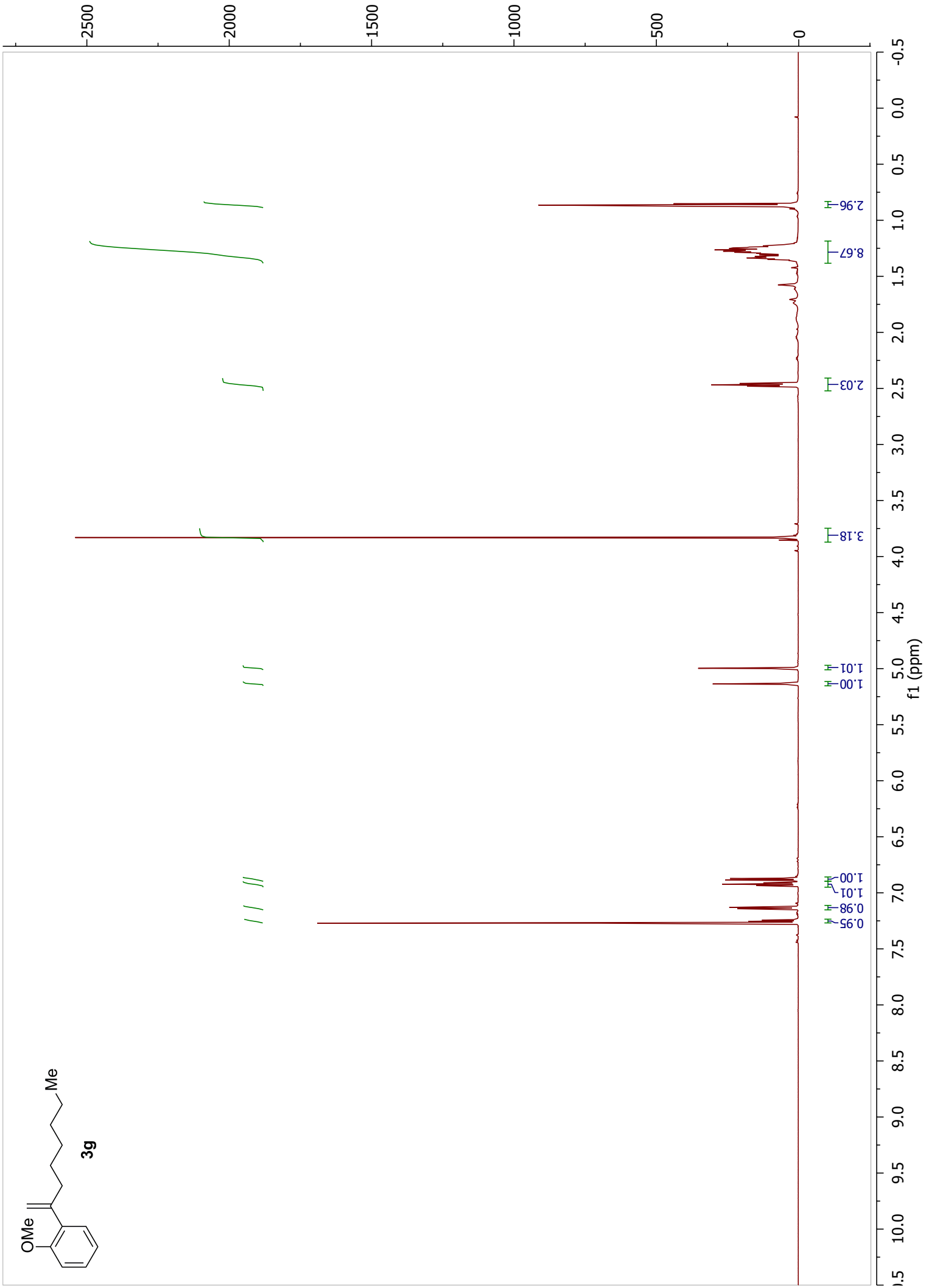

14.04
22.60
28.08
28.94
31.62
35.22
113.94
120.21
122.91
125.12
125.16
125.19
125.23
125.61
126.40
128.32
128.75
129.07
129.39
129.71
145.09
145.10
145.12
145.13
147.73
Me
3d
F3C
f1 (ppm)

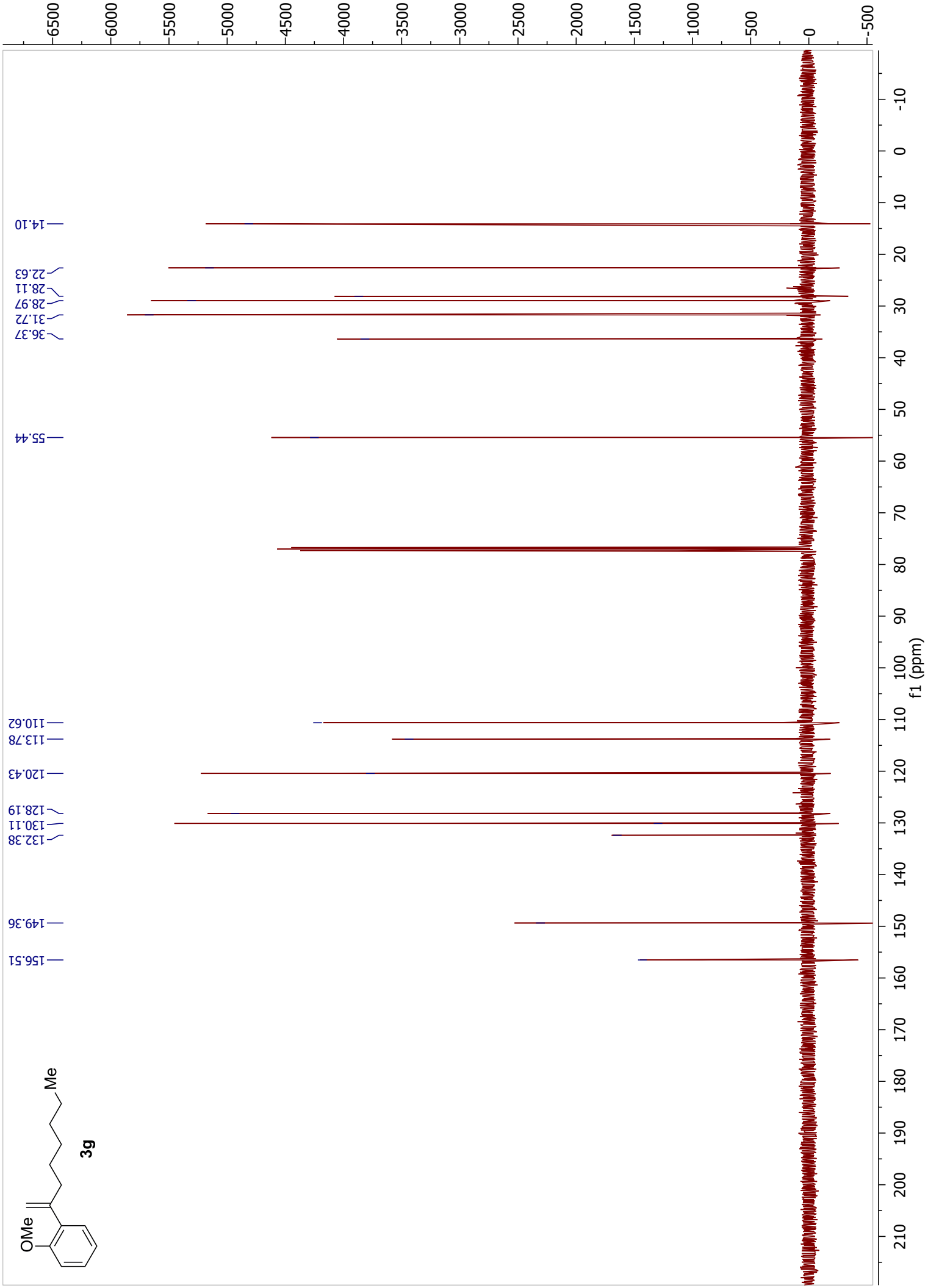

3.14
6.72
2.06
2.95
2.01
1.01
1.00
1.88
1.93
f1 (ppm)
Me
Me
3e

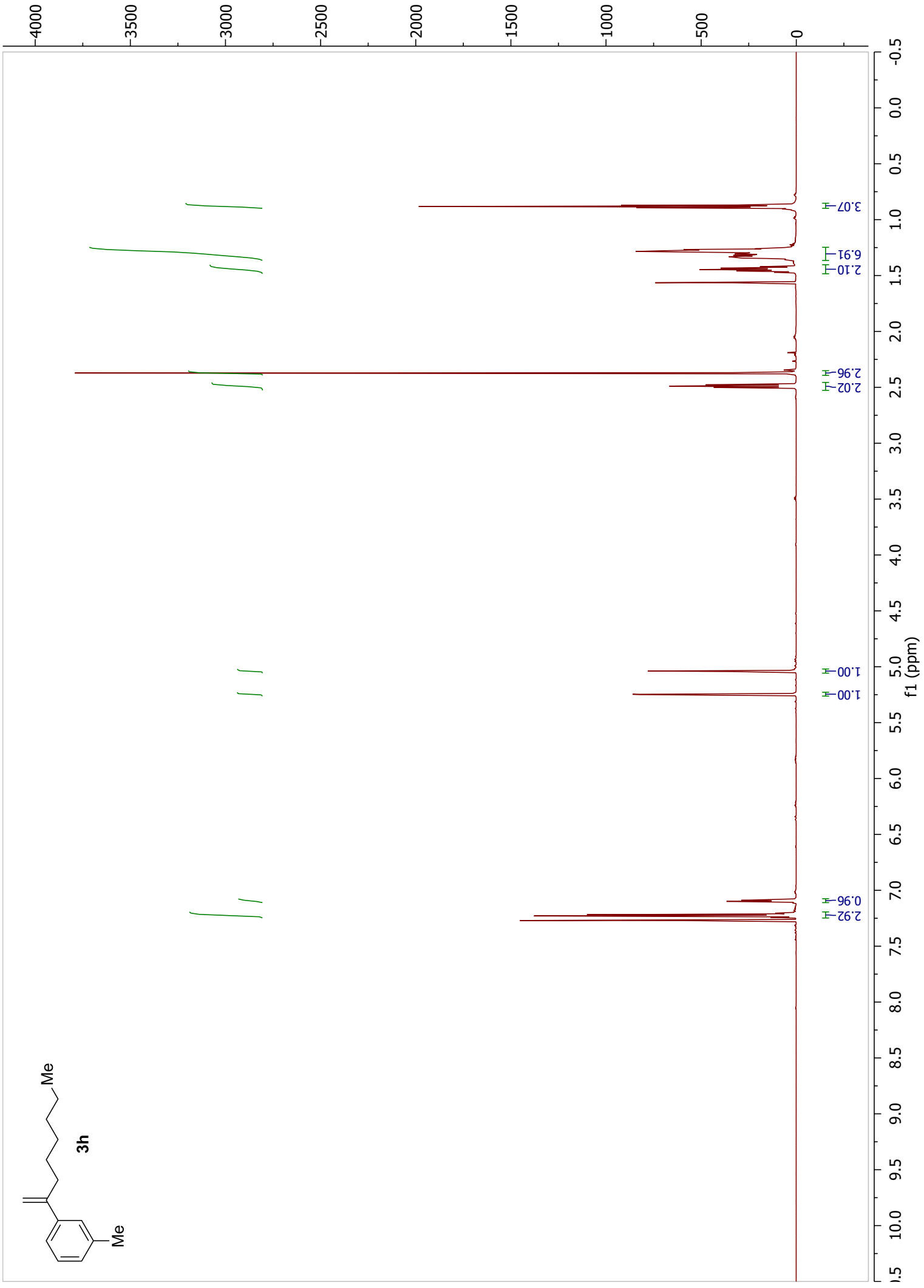

14.07
21.07
22.63
28.28
29.05
31.68
35.37
111.19
125.95
128.89
136.90
138.52
148.55
Me
3e
Me
f1 (ppm)

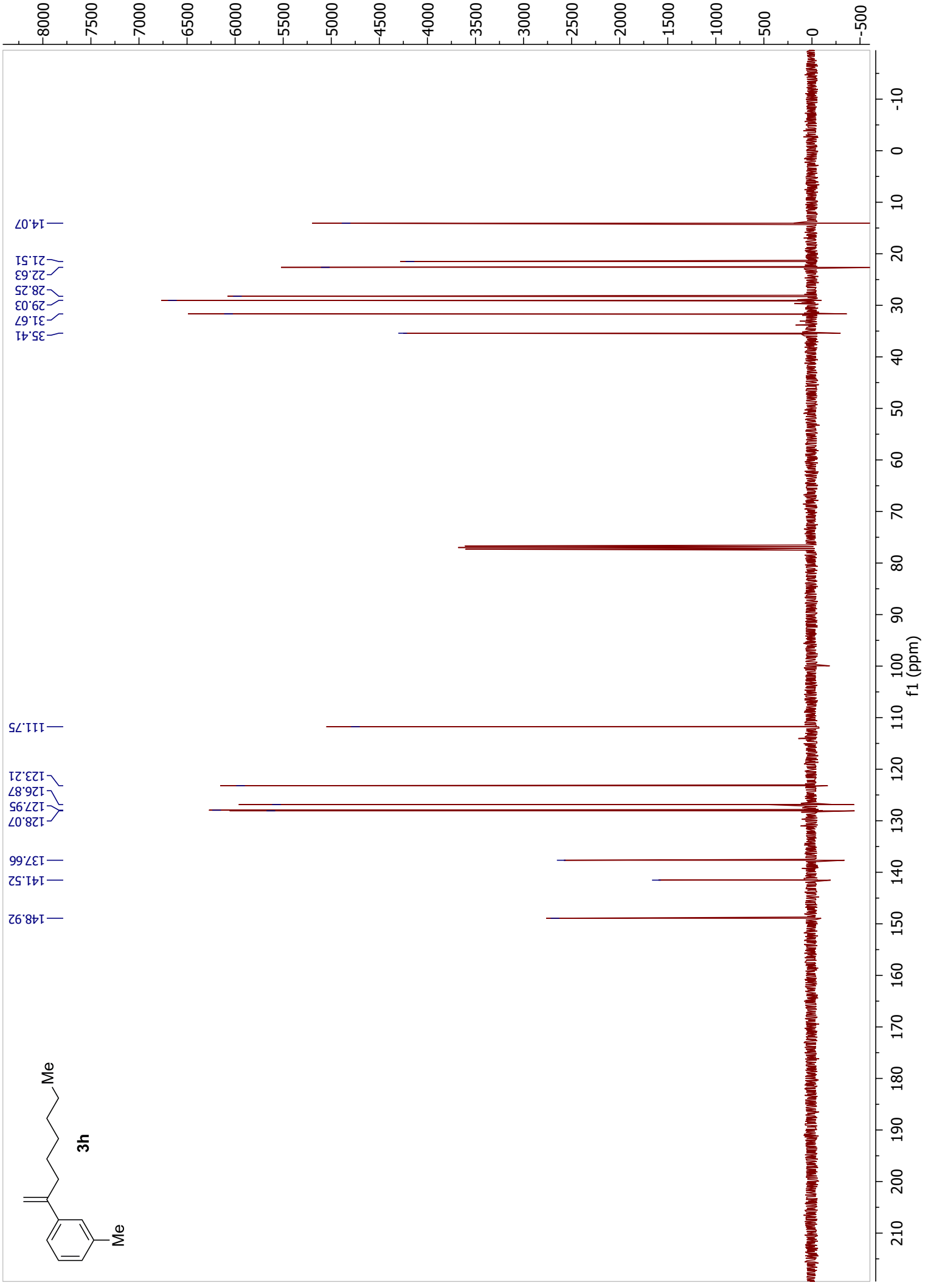

3.15
6.86
2.12
2.01
3.08
1.00
1.00
0.96
0.97
0.97
0.96
f1 (ppm)
Me
3f
OMe

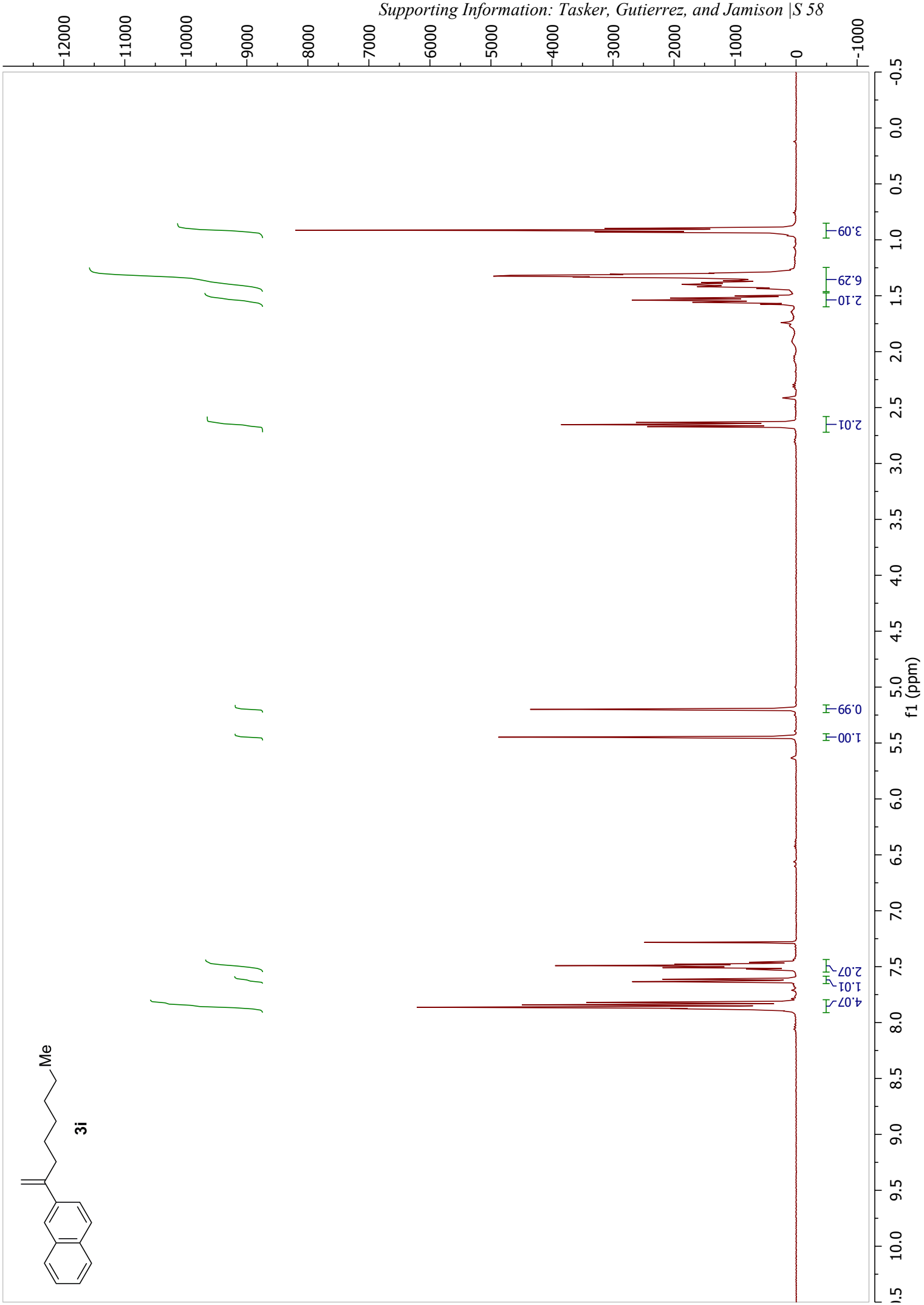

14.06
22.61
28.24
29.02
31.66
35.43
55.17
112.10
112.14
112.37
118.68
129.12
143.08
148.69
159.48
Me
3f
OMe
f1 (ppm)

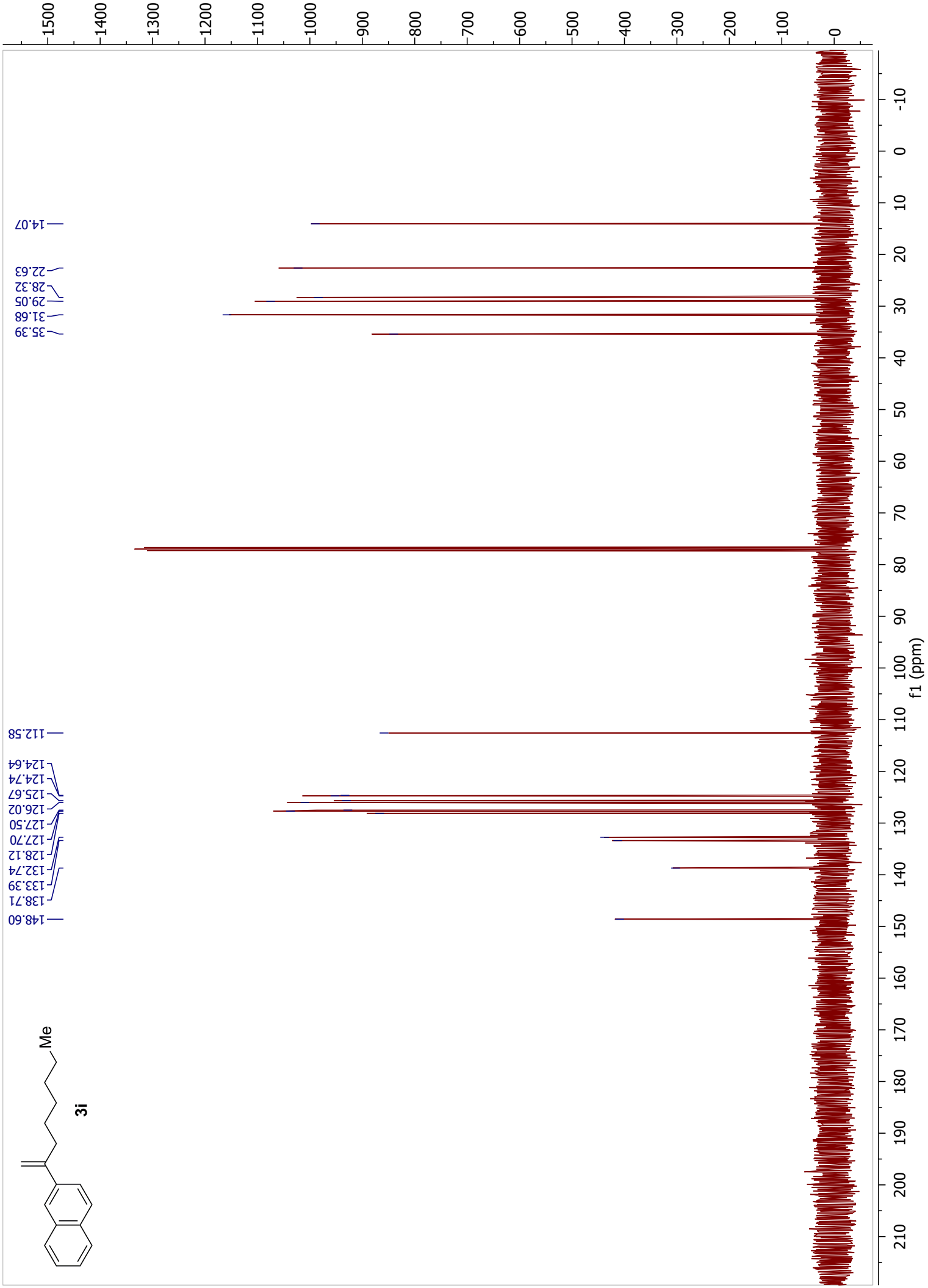

2.96
8.67
2.03
3.18
1.01
1.00
1.00
1.01
0.98
0.95
f1 (ppm)
OMe
Me
3g

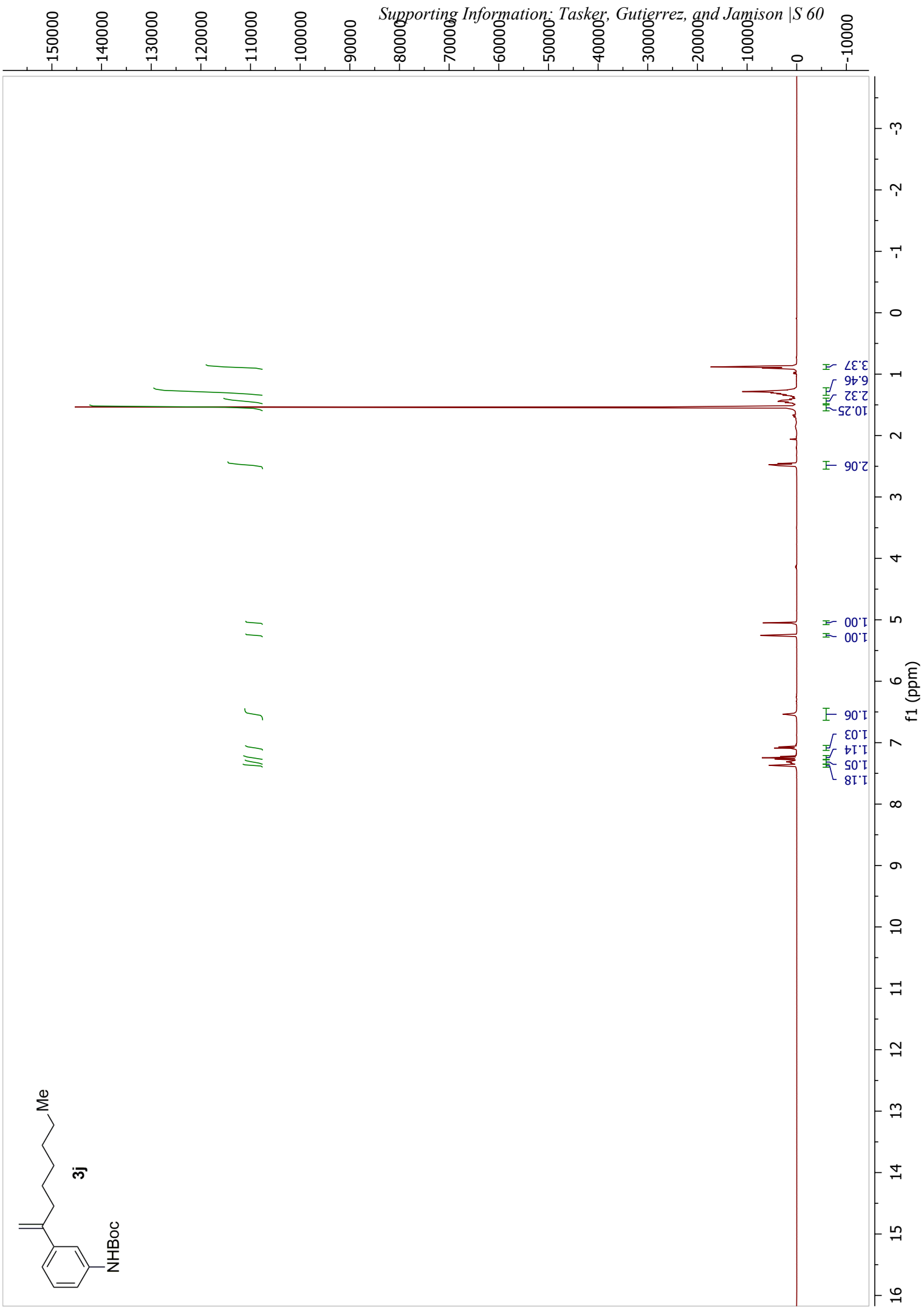

14.10
22.63
28.11
28.97
31.72
36.37
55.44
110.62
113.78
120.43
128.19
130.11
132.38
149.36
156.51
OMe
Me
3g
f1 (ppm)

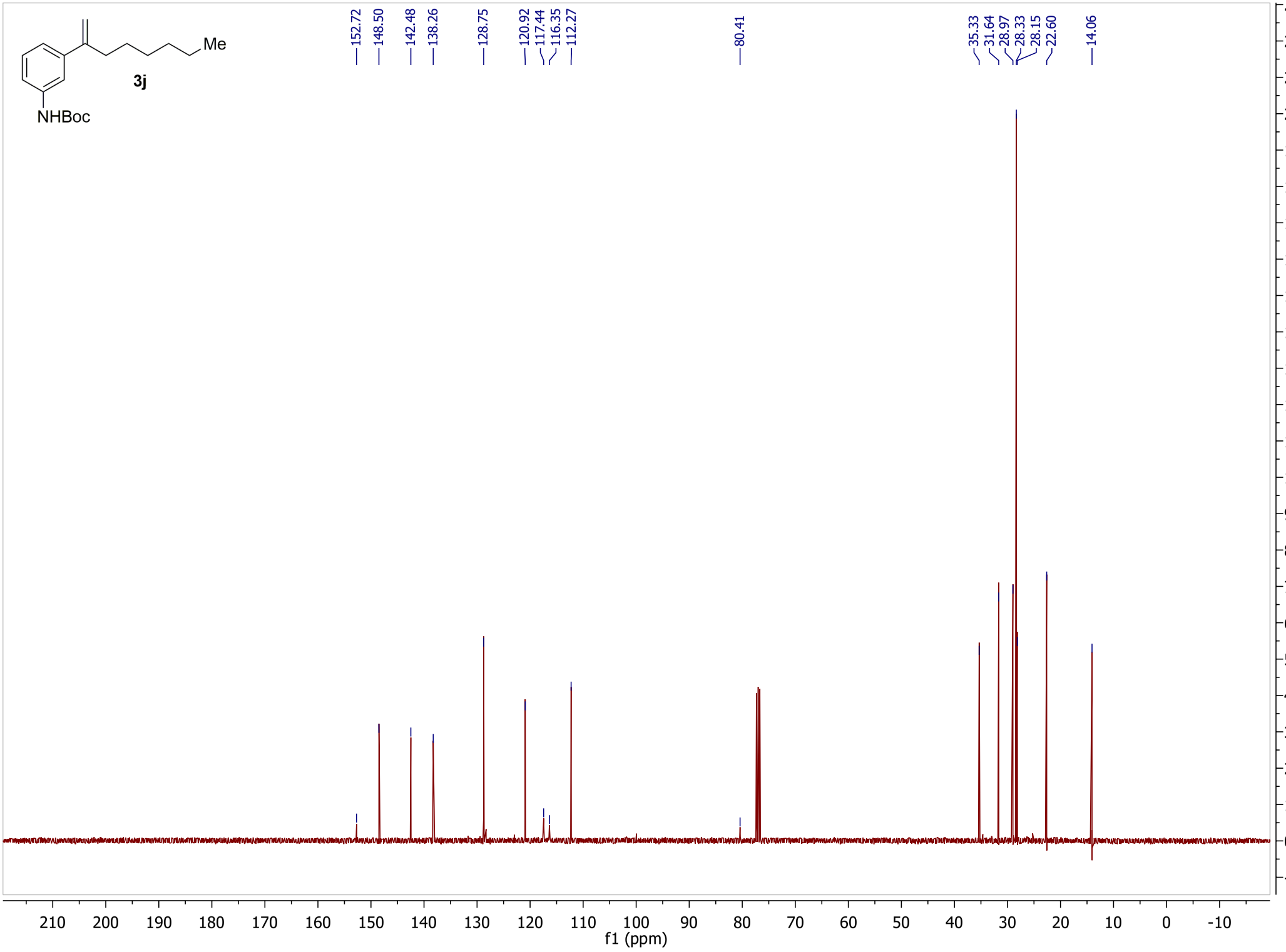

3.07
6.91
2.10
2.96
2.02
1.00
1.00
0.96
2.92
f1 (ppm)
Me
3h
Me

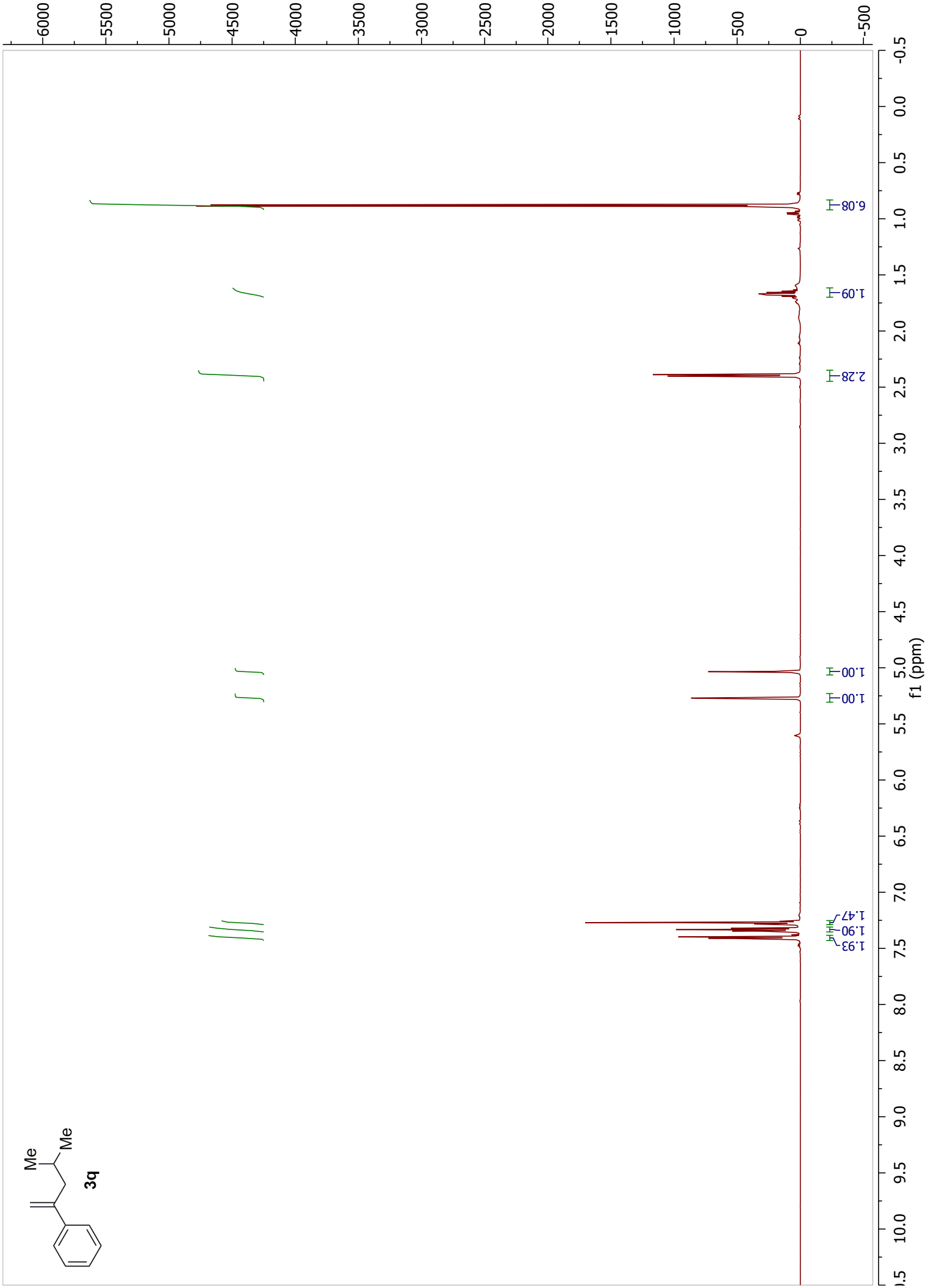

14.07
21.51
22.63
28.25
29.03
31.67
35.41
111.75
123.21
126.87
127.95
128.07
137.66
141.52
148.92
Me
3h
Me
f1 (ppm)

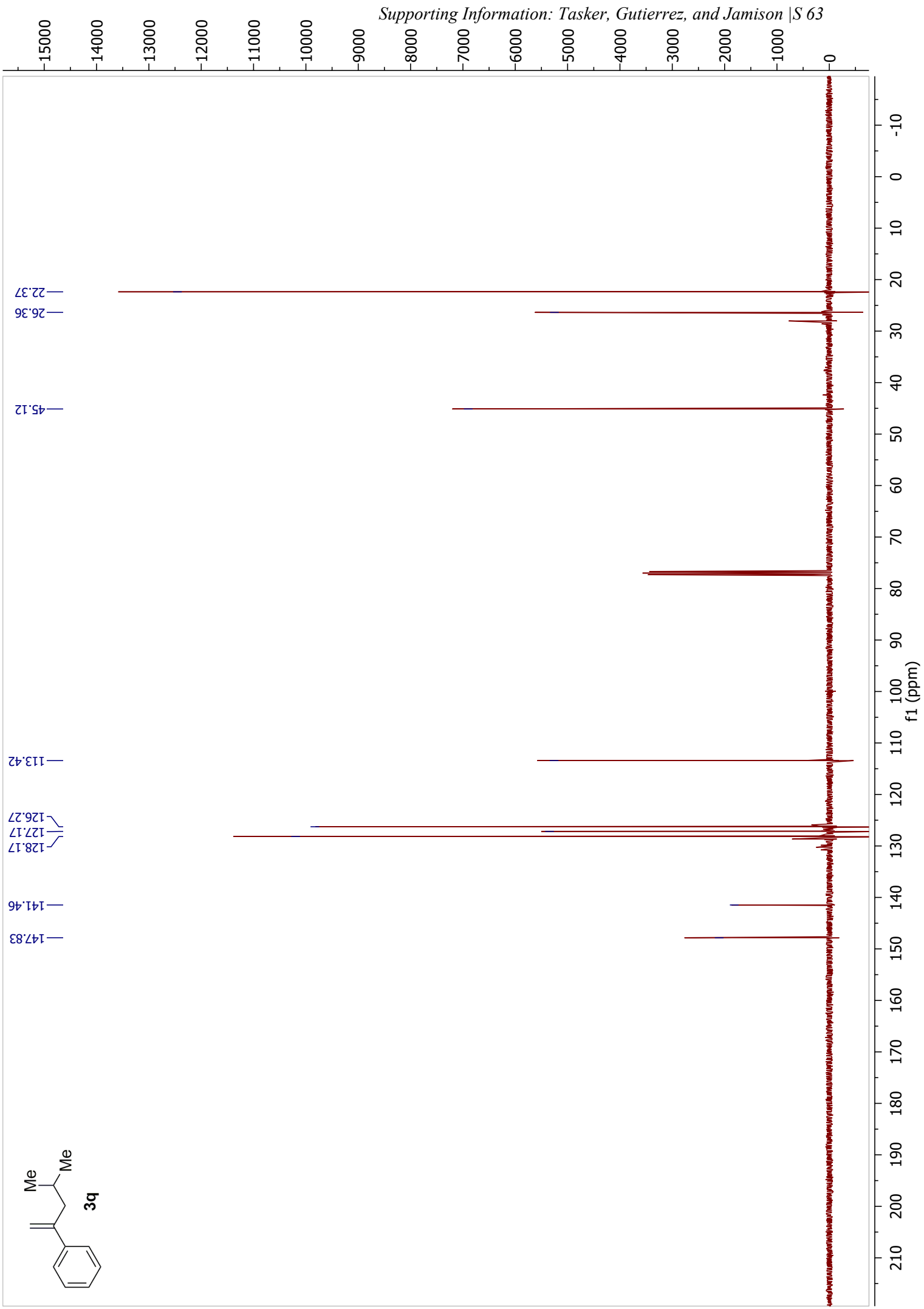

3.09
6.29
2.10
2.01
0.99
1.00
2.07
1.01
4.07
f1 (ppm)
Me
3i

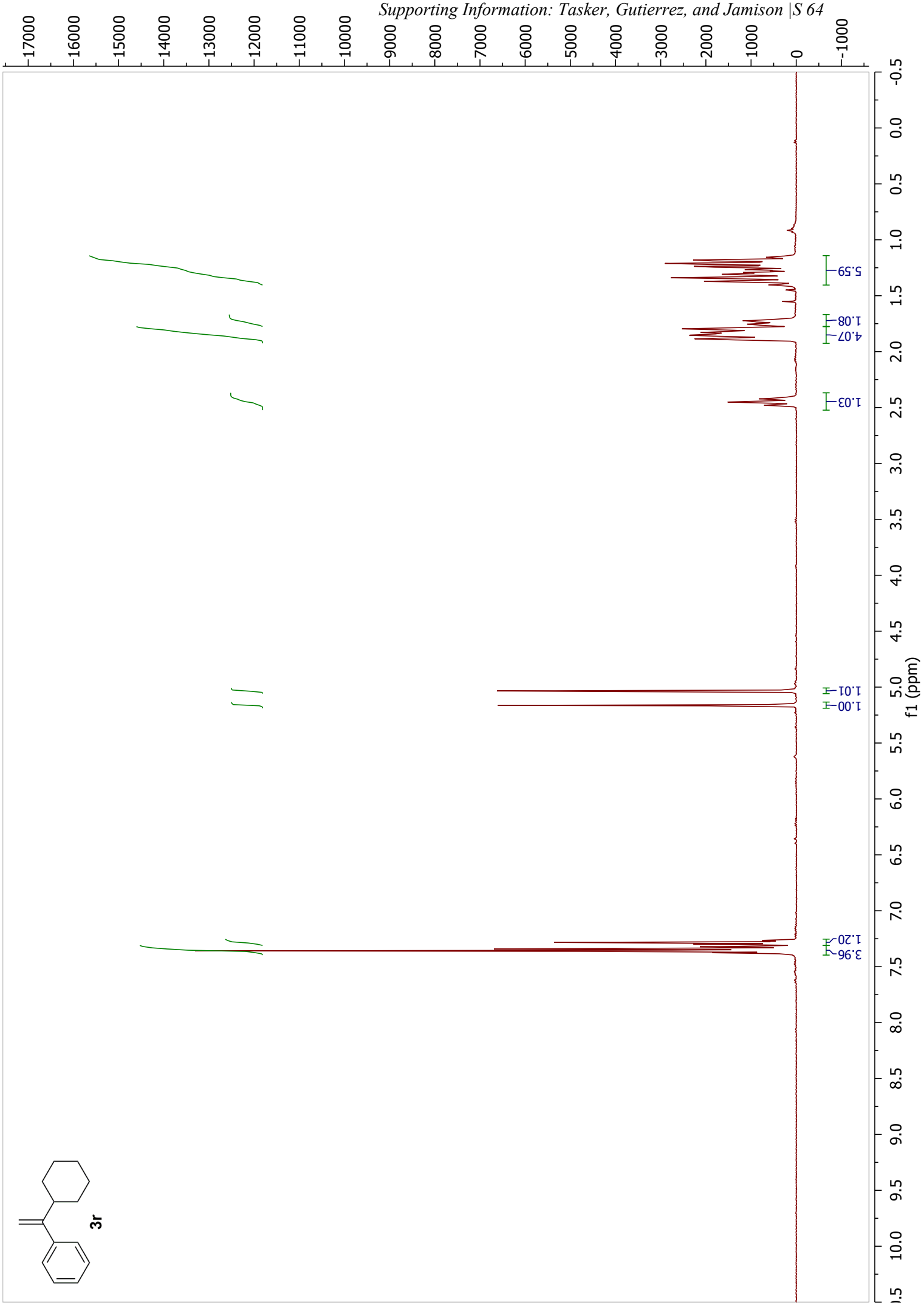

14.07
22.63
28.32
29.05
31.68
35.39
112.58
124.64
124.74
125.67
126.02
127.50
127.70
128.12
132.74
133.39
138.71
148.60
Me
3i
f1 (ppm)

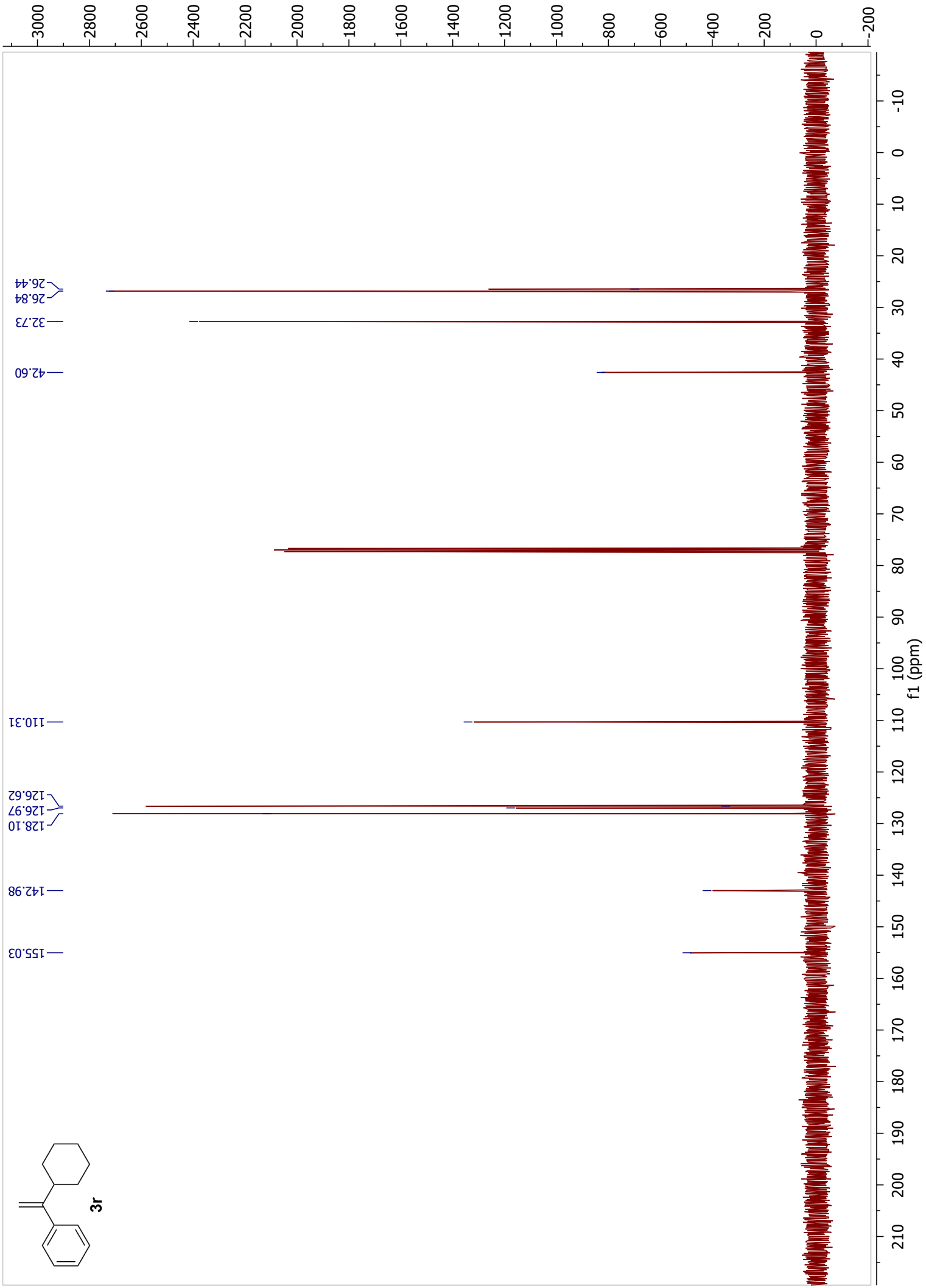

Me
3j
NHBoc
3.37
6.46
2.32
10.25
2.06
1.00
1.00
1.06
1.03
1.14
1.05
1.18
f1 (ppm)

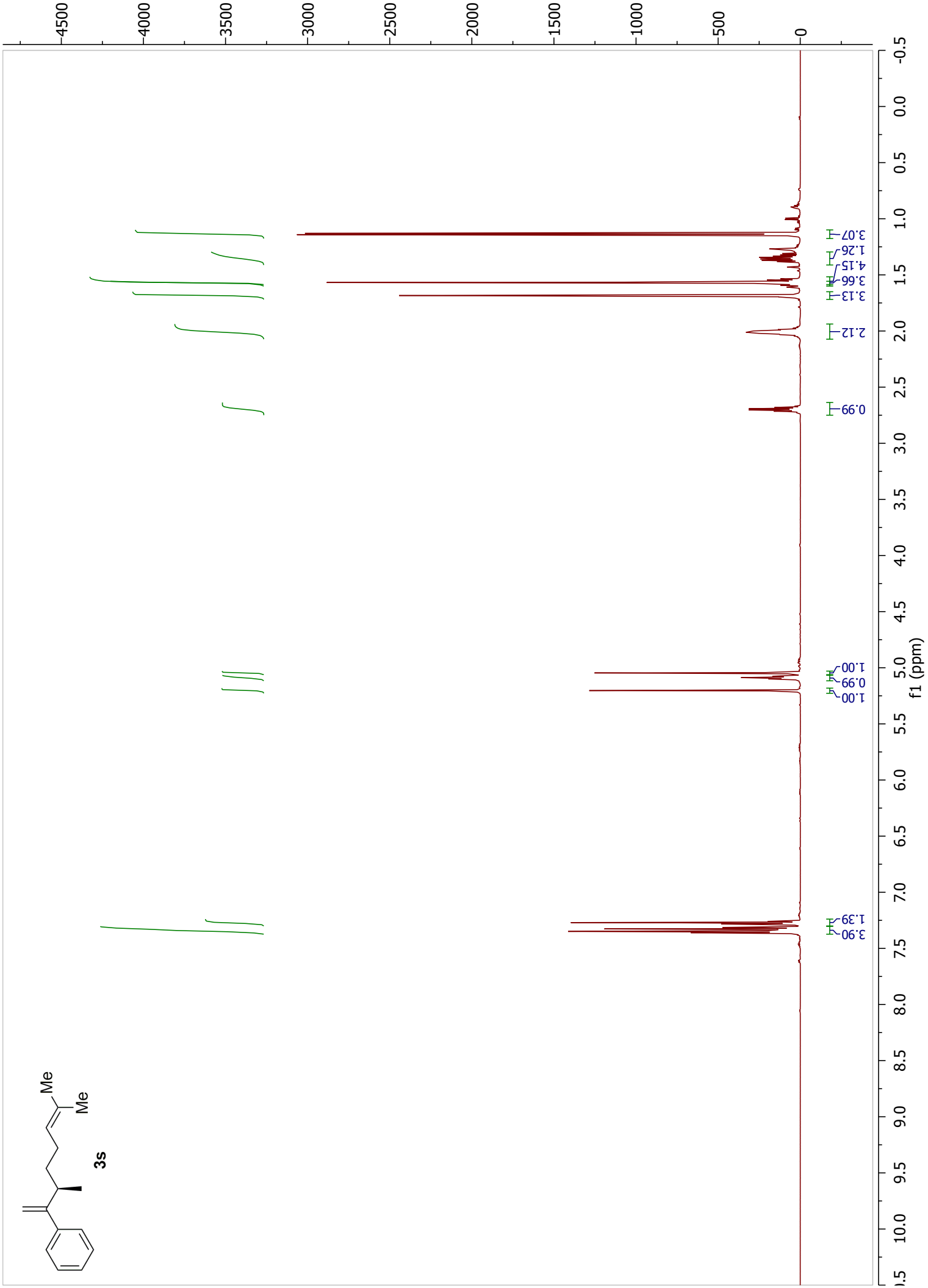

Me
3j
NHBoc
152.72
148.50
142.48
138.26
128.75
120.92
117.44
116.35
112.27
80.41
35.33
31.64
28.97
28.33
28.15
22.60
14.06
f1 (ppm)

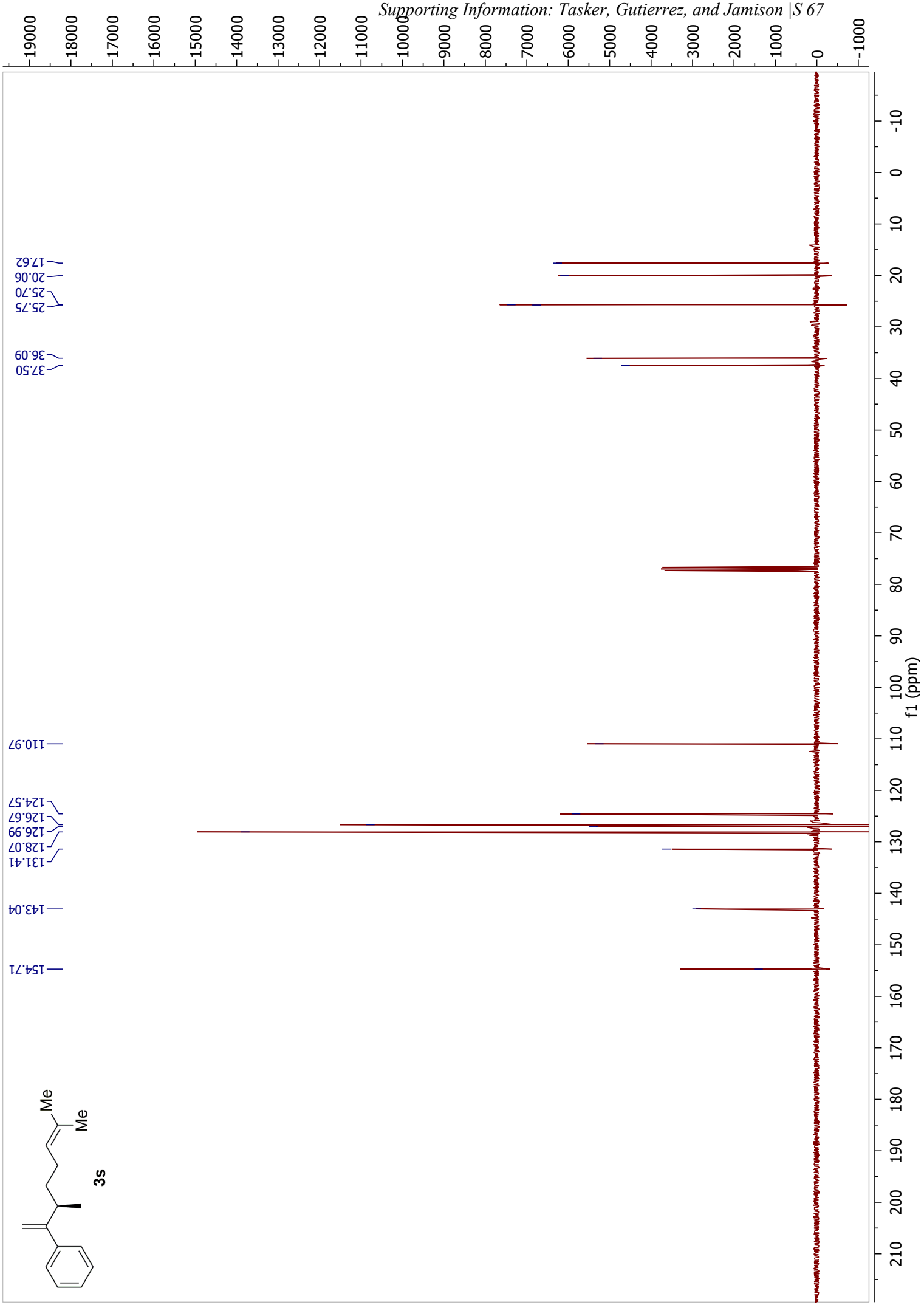

6.08
1.09
2.28
1.00
1.00
1.47
1.90
1.93
f1 (ppm)
Me
Me
3q

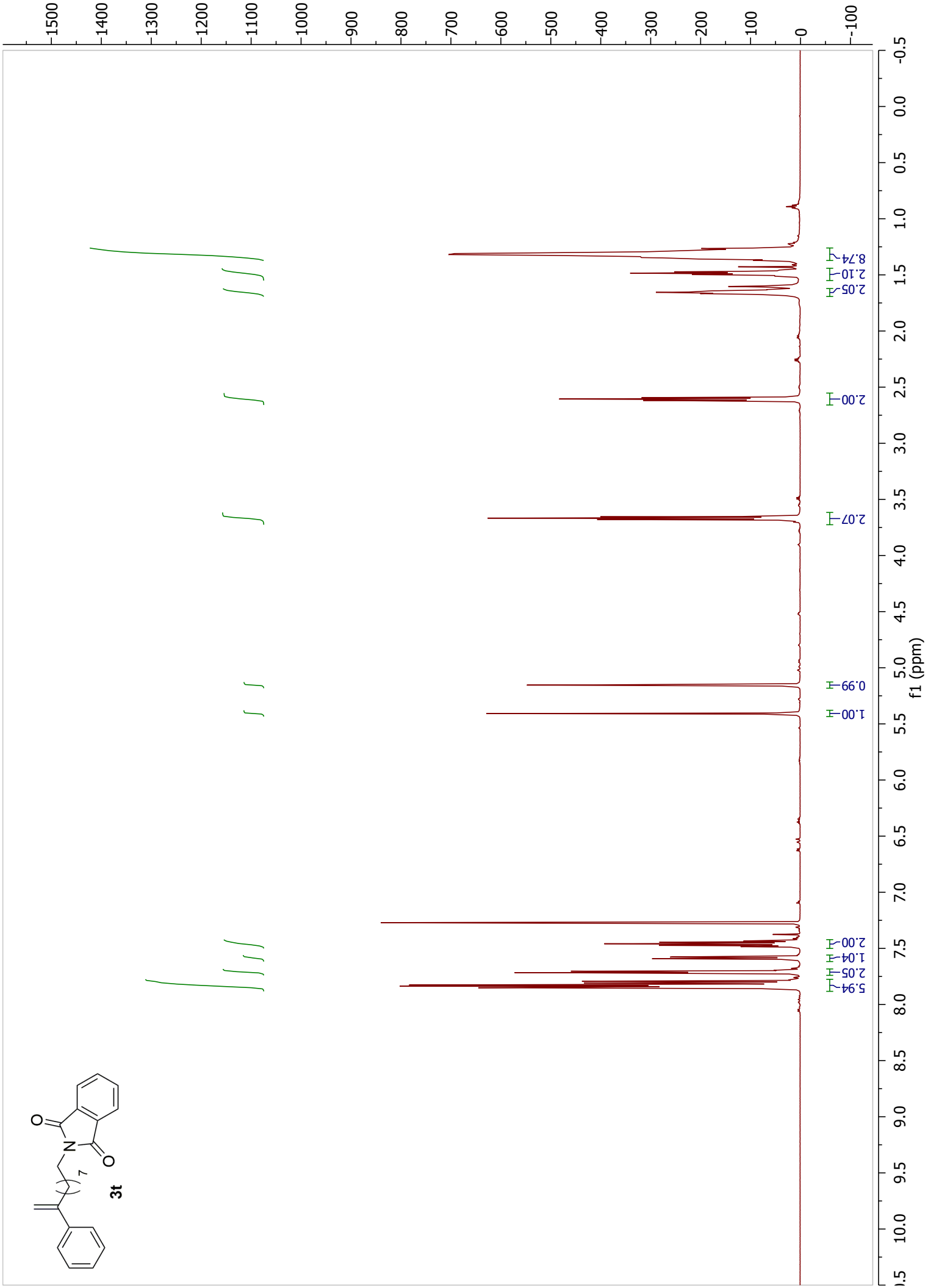

22.37
26.36
45.12
113.42
126.27
127.17
128.17
141.46
147.83
Me
Me
3q
f1 (ppm)

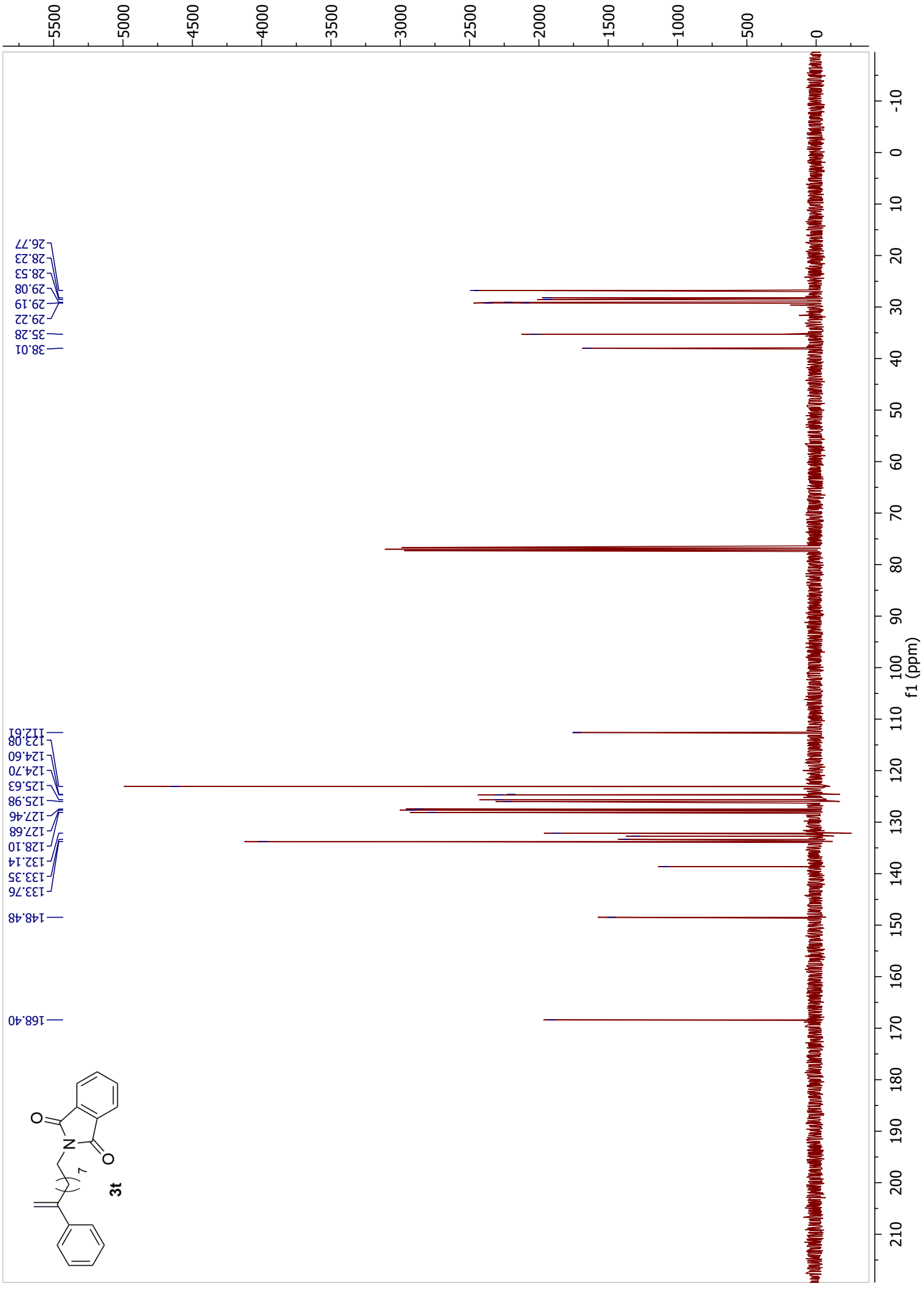

5.59
1.08
4.07
1.03
1.01
1.00
1.20
3.96
f1 (ppm)
3r

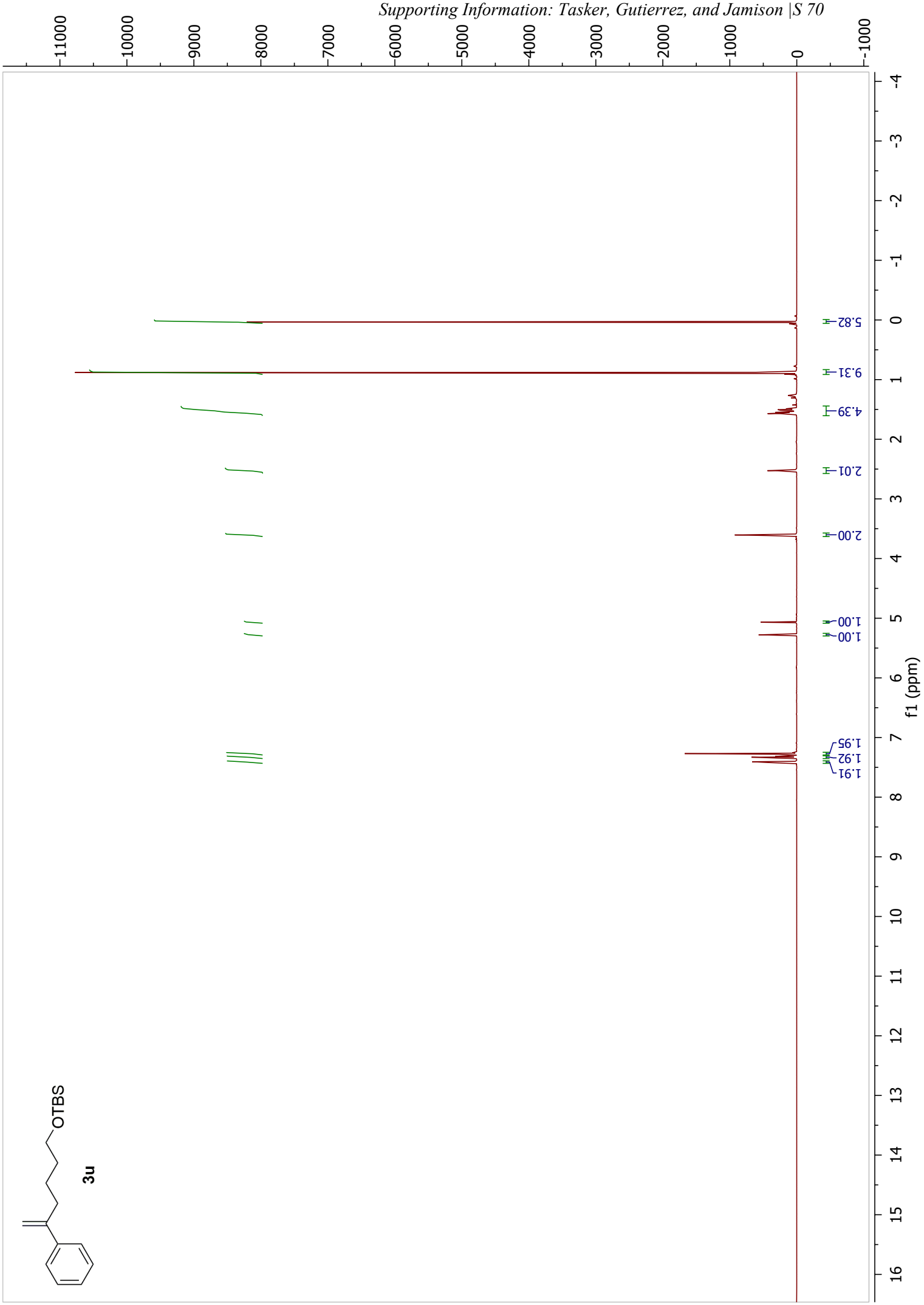

26.44
26.84
32.73
42.60
110.31
126.62
126.97
128.10
142.98
155.03
3r
f1 (ppm)

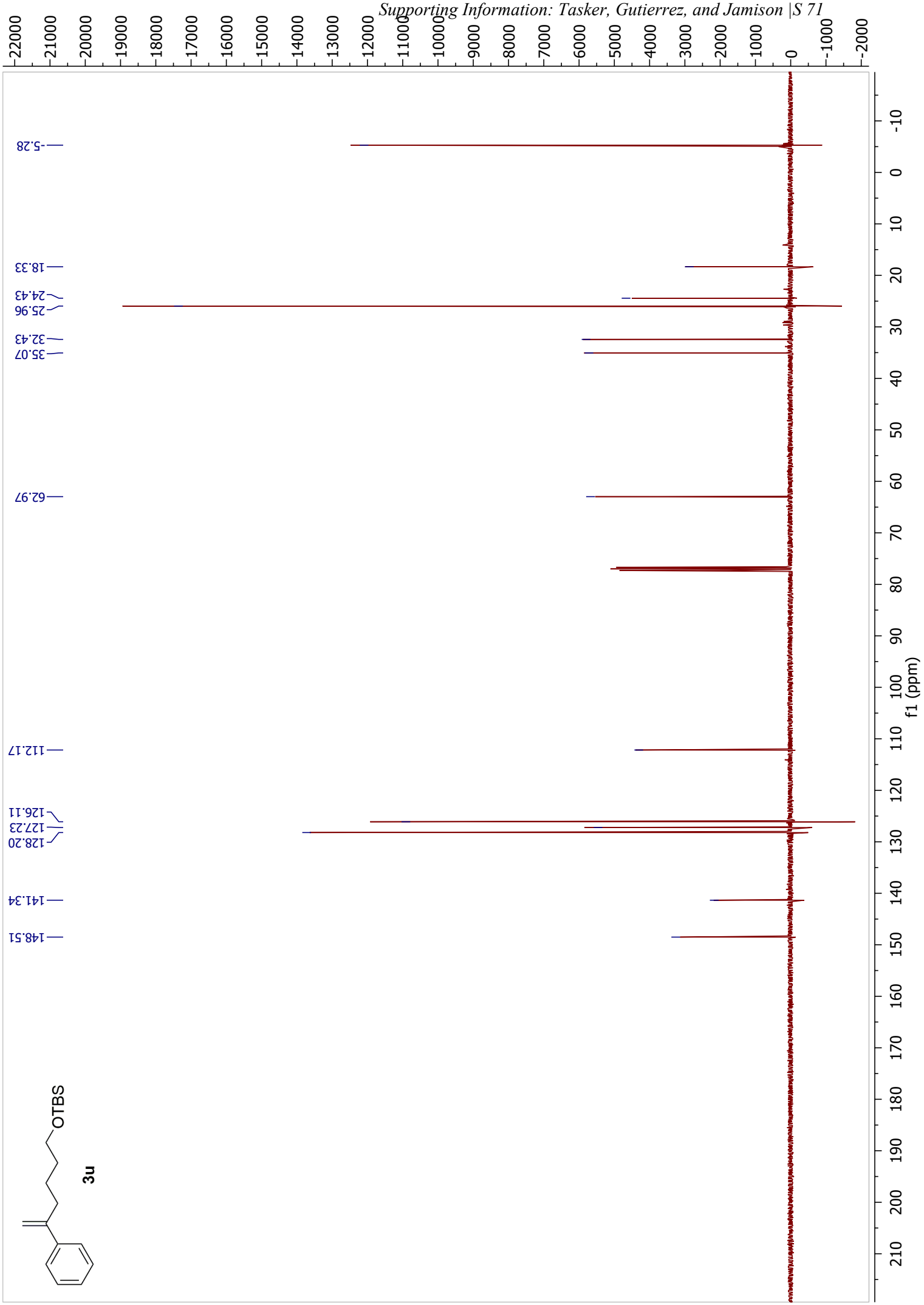

3.07
1.26
4.15
3.66
3.13
2.12
0.99
1.00
0.99
1.00
3.90
1.39
f1 (ppm)
Me
Me
3s

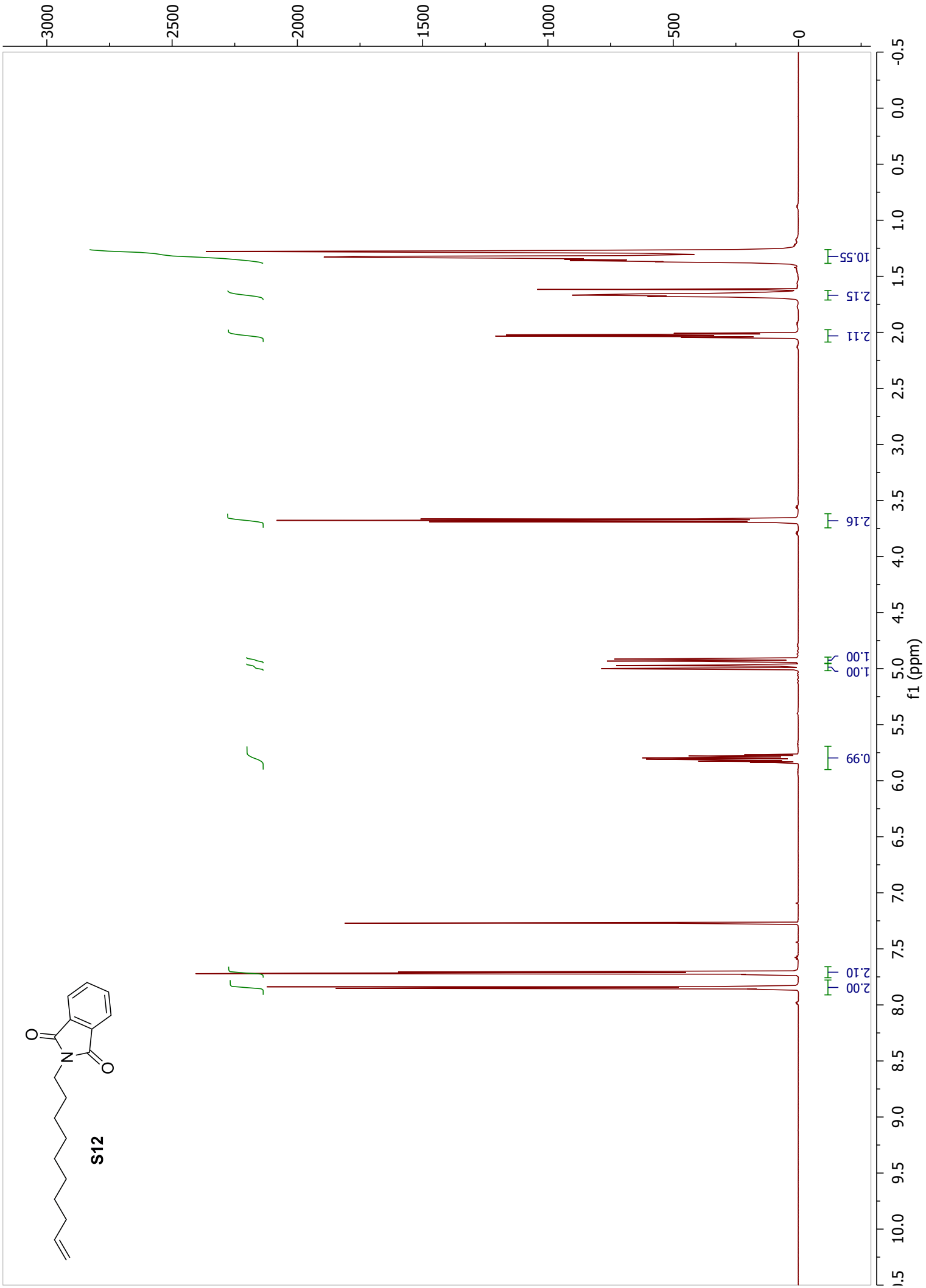

17.62
20.06
25.70
25.75
36.09
37.50
110.97
124.57
126.67
126.99
128.07
131.41
143.04
154.71
Me
Me
3s
f1 (ppm)

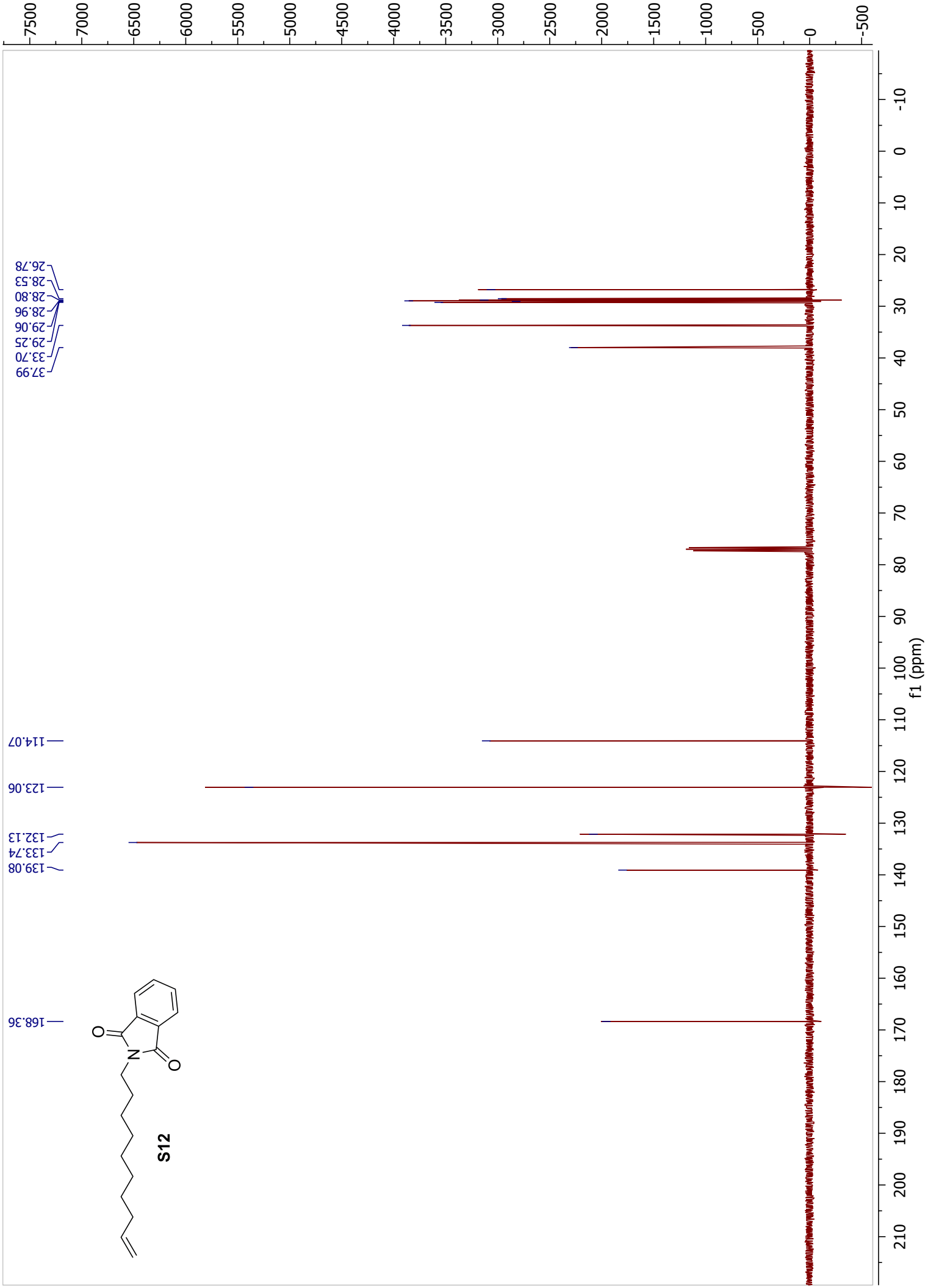

8.74
2.10
2.05
2.00
2.07
0.99
1.00
2.00
1.04
2.05
5.94
f1 (ppm)
3t

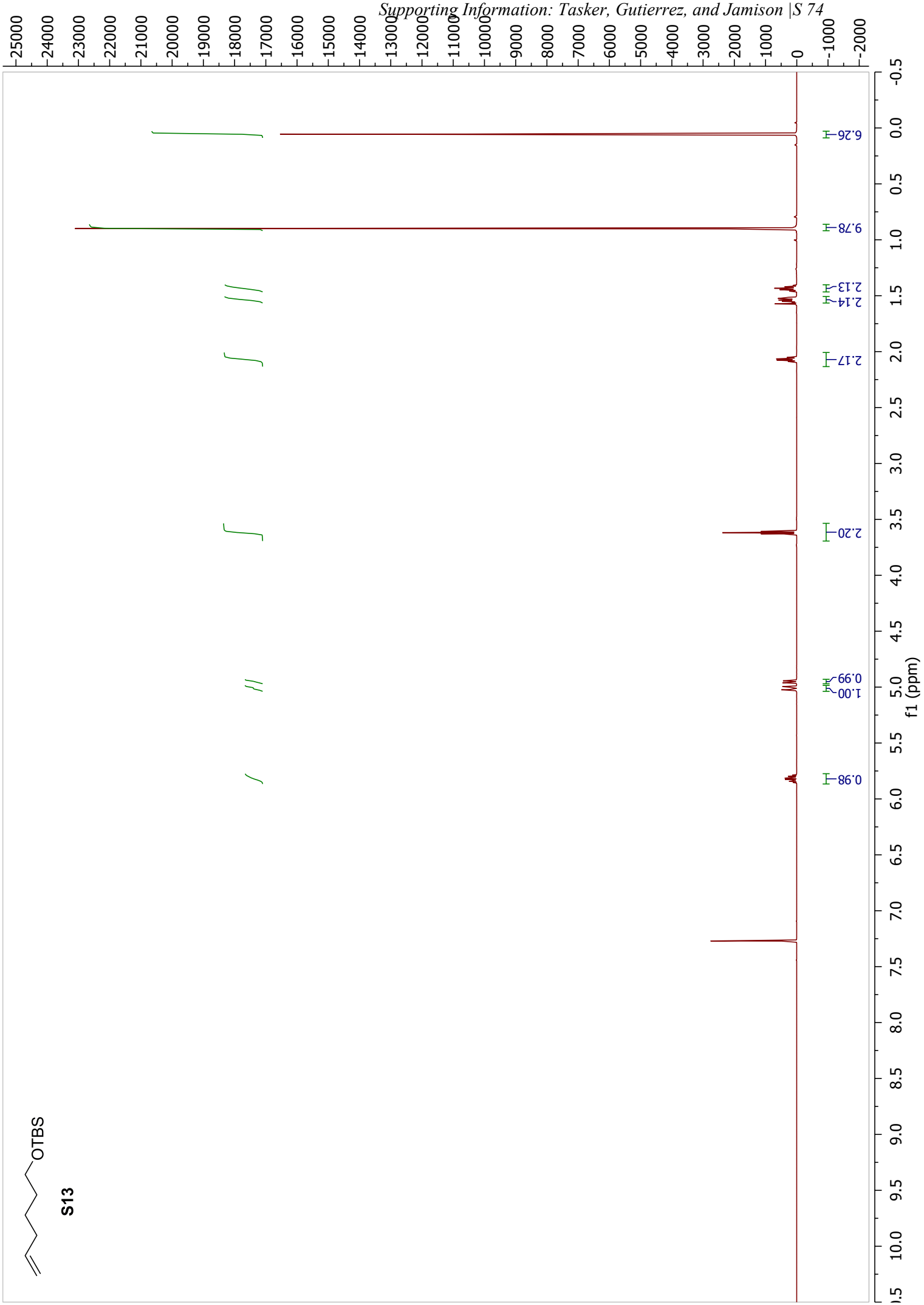

168.40
148.48
133.76
133.35
132.14
128.10
127.68
127.46
125.98
125.63
124.70
124.60
123.08
112.61
38.01
35.28
29.22
29.19
29.08
28.53
28.23
26.77
f1 (ppm)
3t

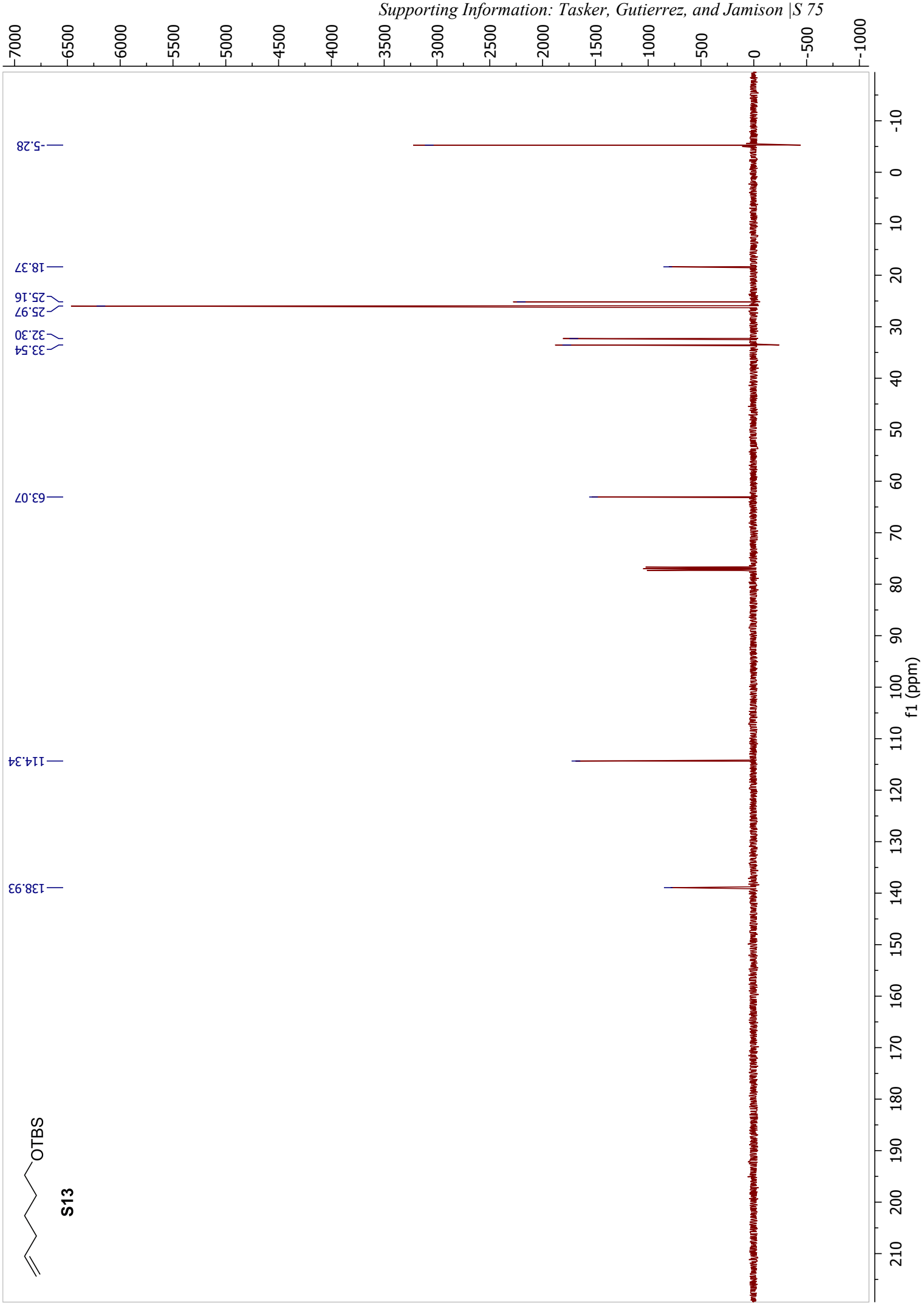

OTBS
3u
5.82
9.31
4.39
2.01
2.00
1.00
1.00
1.95
1.92
1.91
f1 (ppm)


-5.28
18.33
24.43
25.96
32.43
35.07
62.97
112.17
126.11
127.23
128.20
141.34
148.51
OTBS
3u
f1 (ppm)


10.55
2.15
2.11
2.16
1.00
1.00
0.99
2.10
2.00
f1 (ppm)
S12


168.36
139.08
133.74
132.13
123.06
114.07
37.99
33.70
29.25
29.06
28.96
28.80
28.53
26.78
S12
f1 (ppm)


OTBS
S13
6.26
9.78
2.13
2.14
2.17
2.20
0.99
1.00
0.98
f1 (ppm)


-5.28
18.37
25.16
25.97
32.30
33.54
63.07
114.34
138.93
OTBS
S13
f1 (ppm)