Merging Photoredox and Nickel Catalysis: The Decarboxylative Cross-Coupling of Carboxylic Acids with Vinyl Halides

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Supporting Information

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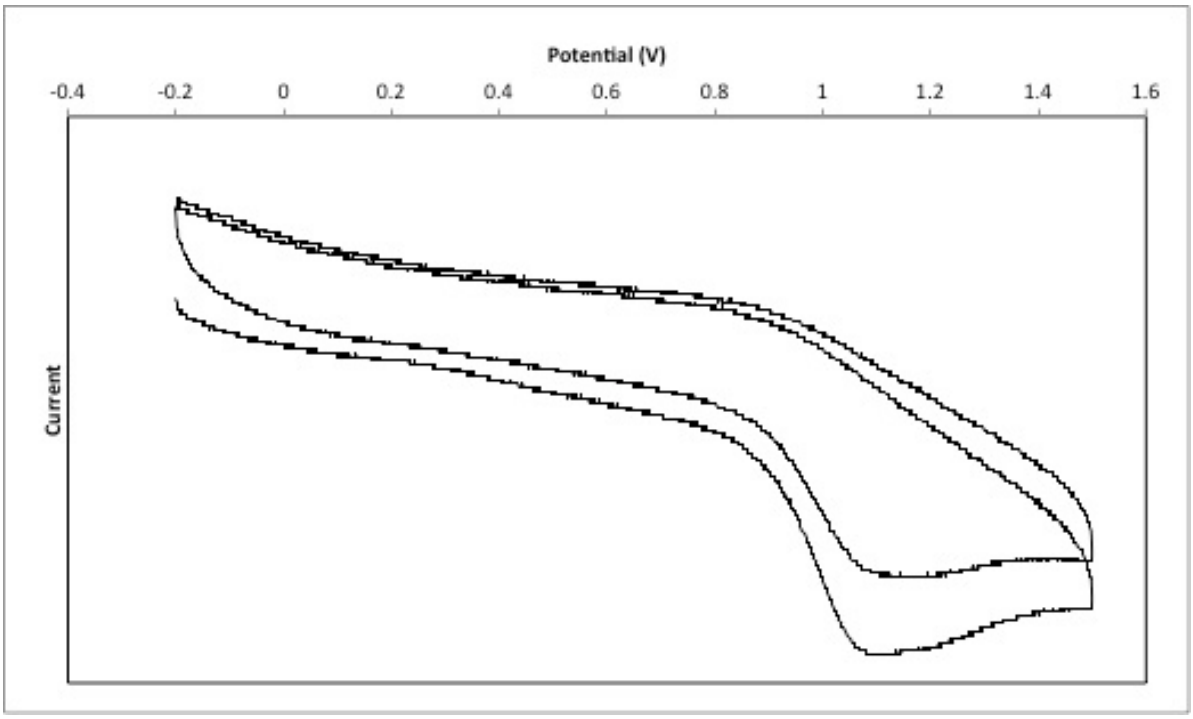
1) General Information

Commercial reagents were purified prior to use following the guidelines of Perrin and Armarego.[1] $Ir[dF(CF_3)ppy]_2(dtbbpy)PF_6$ and $Ir[dF(Me)ppy]_2(dtbbpy)PF_6$ were prepared according to the literature procedures.[2] All solvents were purified according to the method of Grubbs.[3] Non-aqueous reagents were transferred under nitrogen or argon via syringe or cannula. Organic solutions were concentrated under reduced pressure on a Büchi rotary evaporator using a water bath. Chromatographic purification of products was accomplished using forced-flow chromatography on ICN 60 32–64 mesh silica gel 63 or Davisil Grade 643 silica gel according to the method of Still.[4] Thin-layer chromatography (TLC) was performed on Silicycle 0.25 mm silica gel F-254 plates. Visualization of the developed chromatogram was performed by fluorescence quenching or $KMnO_4$ stain. ^1H NMR spectra were recorded on a Bruker UltraShield Plus 500 MHz unless otherwise noted and are internally referenced to residual protio $CDCl_3$ signals (7.27 ppm). Data for ^1H NMR are reported as follows: chemical shift (δ ppm), integration, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, dd = doublet of doublets, dt = doublet of triplets, br = broad), coupling constant (Hz), and assignment. ^{13}C NMR spectra were recorded on a Bruker UltraShield Plus 500 MHz and data are reported in terms of chemical shift relative to $CDCl_3$ (77.0 ppm). IR spectra were recorded on a Perkin Elmer Spectrum 100 FTIR spectrometer and are reported in wavenumbers (cm^{-1}). High Resolution Mass Spectra were obtained from the Princeton University Mass Spectral Facility.

2) Cyclic Voltammetry of Tetrahydrofuran-2-carboxylic acid

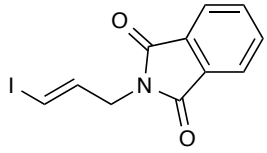
Cyclic voltammetry was performed using a CH Instruments Electrochemical Workstation model CHI600E with a scan rate of 0.5 V/s, 4 sweep segments, a sample interval of 0.001 V, and a sensitivity of 0.001 A/V.

Tetrahydrofuran-2-carboxylic acid (23 mg, 0.2 mmol) and NBu_4PF_6 (774 mg, 2 mmol) were dissolved in MeCN (20 mL) and CsOH (50 wt% in H_2O, 35 μL, 0.2 mmol) was added. The solution was degassed by sparging with N_2 for 15 minutes before the electrochemical measurement. An irreversible oxidation peak was observed at +1.08 V vs. SCE.



3) Preparation of Vinyl Halides

The following vinyl halides were prepared according to literature procedures: (*E*)-(3-iodoallyl)benzene,[5] (*E*)-1-iodo-3, 3-dimethylbut-1-ene,[6] (*E*)-(((4-iodobut-3-en-1-yl)oxy)methyl)benzene,[7] (*E*)-5-chloro-1-iodopent-1-ene,[8] (*Z*)-1-iodo-hept-1-ene,[9] (*E*)-(2-iodovinyl)cyclohexane,[10] 1-bromocyclohept-1-ene,[11] and (*E*)-1-bromooct-1-ene.[12] The following vinyl halides are commercially available: (*E*)-1-iodooct-1-ene, 1-bromo-2-methylprop-1-ene, and (2-bromoallyl)trimethylsilane.



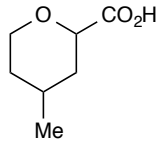
(*E*)-2-(2-Iodoallyl)isoindoline-1, 3-dione

In a glovebox zirconocene chloride hydride (2.90 g, 11 mmol, 1.10 equiv.) was added to 2-(prop-2-yn-1-yl)isoindoline-1,3-dione (1.85 g, 10 mmol, prepared according to a literature procedure).[13] After removal from glovebox dry THF (20 mL, 0.25 M) was added through a septum and the reaction stirred for 2 hours at room temperature. The solution was then cooled to

0 °C and I_2 (1.90 g, 15 mmol, 1.50 equiv.) was added against a positive pressure of N_2. The solution was allowed to warm to room temperature over 1 hour. The reaction was quenched with saturated Na_2SO_3 (5 mL), diluted with EtOAc, and filtered through celite. The filtrate was washed with brine, dried ($MgSO_4$), filtered, and concentrated. The crude product was purified by flash column chromatography (10-30% EtOAc/hexanes) to yield the product (1.4 g, 46%) as a white solid. IR (film) ν_{max} 1765, 1710, 1429, 1395, 1305, 1206, 1036, 929, 716 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 4.24 (2H, dt, *J* = 6.6, 1.3, CH=CHC**H$_2$**), 6.50 (1H, dt, *J* = 14.5, 1.3, IC**H**=CH), 6.61 (1H, dtd, *J* = 14.1, 6.4, 1.3, ICH=C**H**), 7.74 (2H, ddd, *J* = 5.6, 3.0, 1.2, Ar**H**), 7.87 (2H, ddd, *J* = 5.5, 3.1, 1.2, Ar**H**); ^{13}C NMR (126 MHz, $CDCl_3$) δ 41.4, 81.1, 123.5, 131.9, 134.2, 138.6, 167.6; HRMS (ESI-TOF) *m/z* calcd. for $C_{11}H_{29}INO_2$ ($[M+H]^+$) 313.9673, found 313.9669.

4) Preparation of Carboxylic Acids

(*R*)-2-(benxyloxy)-2-((*R*)-2, 2-dimethyl-1, 3-dioxolan-4-yl)acetic acid was prepared according to a literature procedure.[14] The following carboxylic acids are commercially available: tetrahydrofuran-2-carboxylic acid, tetrahydropyran-2-carboxylic acid, (*3*a*S*,4*S*,6*R*,6a*R*)-6-methoxy-2,2-dimethyltetrahydrofuro[3,4-d][1,3]dioxole-4-carboxylic acid, 2-(benzyloxy)propanoic acid, 2-(benzyloxy)acetic acid, *N*-Boc-proline, *N*-Me-Boc-leucine, *N*-Boc-phenylalanine, *N*-Boc-tryptophan, phenylacetic acid, cyclohexanecarboxylic acid, 5-phenylvaleric acid, 4-methoxyphenylacetic acid, and 3-chlorophenylacetic acid.



(*cis*)-4-Methyltetrahydro-2H-pyran-2-carboxylic acid (35)

To a solution of 4-methyl-3, 6-dihydro-2H-pyran-2-carboxylic acid (0.45 g, 3.17 mmol, prepared according to a literature procedure)[15] in EtOAc (15.8 mL, 0.2 M) was added palladium on carbon (0.168 g, 10 wt%). The atmosphere was replaced with hydrogen and the reaction stirred overnight. Filtration of the reaction mixture through celite and concentration provided the product (0.44 g, 97%, 4.8:1 dr). The inseparable diastereomers were characterized as a mixture

IR (film) ν_{max} 2954, 2927, 1732, 1458, 1258, 1174, 1102, 1043, 885 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 1.01 (3H, d, *J* = 6.5), 1.05 (0.6 H, d, *J* = 6.8), 1.14–1.39 (2.4 H, m), 1.56–1.65 (1.2 H, m), 1.65–1.80 (1.4 H, m), 1.88 (0.2 H, dq, *J* = 7.3, 3.7), 1.97–2.06 (0.2 H, m), 2.11 (1H, ddt, *J* = 13.3, 4.0, 2.3), 3.54 (1H, ddd, *J* = 12.4, 11.5, 2.3), 3.82–3.89 (0.4 H, m,), 3.97 (1H, dd, *J* = 12.0, 2.5), 4.15 (1H, ddd, *J* = 11.6, 4.7, 1.5), 4.38 (0.2 H, dd, *J* = 6.4, 4.7); ^{13}C NMR (126 MHz, $CDCl_3$) δ 19.7, 21.9, 25.6, 30.0, 32.3, 33.6, 34.2, 36.7, 64.0, 68.1, 71.7, 75.3, 173.4; HRMS (ESI-TOF) *m/z* calcd. for $C_7H_{12}O$ ($[M+H]^+$) 145.08592, found 145.08606.

(*trans*)-4-Methyltetrahydro-2H-pyran-2-carboxylic acid (36)

In order to selectively access the trans isomer a literature procedure was followed starting from methyl-4-methyl-3,6-dihydro-2H-pyran-2-carboxylate (0.80 g, 5.12 mmol).[17] The product was purified by flash column chromatography (20% EtOAc/hexanes) to afford *trans*-methyl-4-methyltetrahydro-2H-pyran-2-carboxylate (0.50 g, 3.16 mmol, 62%, 20:1 dr). The methyl ester was hydrolyzed using LiOH (0.098 g, 1.3 equiv., 4.11 mmol) in 1:1 THF:H_2O (31 mL, 0.1 M). After stirring 1 hour the pH was adjusted to 3 and 30 mL saturated NH_4Cl solution was added. The solution was extracted with CH_2Cl_2 (3x30 mL), dried (Na_2SO_4), filtered and concentrated. The carboxylic acid was used without further purification.

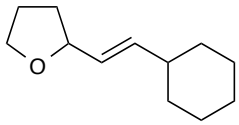
5) General Decarboxylative Vinylation Procedure

General procedure for the decarboxylative vinylation of α-oxy and α-amino acids:

To an oven dried 8 mL vial equipped with a stir bar was added $Ir[dF(Me)ppy]_2(dtbbpy)PF_6$ (5.1 mg, 5.0 μmol, 0.01 equiv.), the carboxylic acid (if solid, 0.80 mmol, 1.6 equiv.), and the vinyl halide (if solid, 0.50 mmol, 1.0 equiv.). The vial was sealed and evacuated then backfilled with nitrogen three times. DMSO (4.0 mL) was added to the vial, followed by the carboxylic acid (if liquid, 0.80 mmol, 1.6 equiv.), 1,8-diazabicycloundec-7-ene (DBU) (0.12 mL, 0.80 mmol, 1.6 equiv.), and the vinyl halide (if liquid, 0.50 mmol, 1.0 equiv.). $NiCl_2$·glyme (2.2 mg, 10 μmol, 0.02 equiv.) and dtbbpy (2.7 mg, 10 μmol, 0.02 equiv.) were added as a 0.01 M solution in DMSO (1.0 mL). The reaction mixture was degassed by sparging with N_2 while stirring for 15 min before sealing the vial with Parafilm. The reaction was stirred and irradiated with a 34 W blue LED lamp until complete consumption of the vinyl halide. The reaction was diluted with

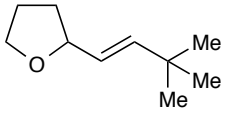
water (15 mL) and brine (5 mL) and the product extracted into Et_2O (3 x 20 mL). The combined organic extracts were combined and washed with water (2 x 15 mL), brine (10 mL), dried ($MgSO_4$), and concentrated *in vacuo*. Purification by flash column chromatography yielded the vinylation product.

6) Experimental Data for Vinylation Products



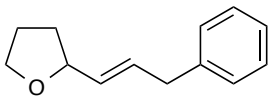
(*E*)-2-(2-Cyclohexylvinyl)tetrahydrofuran (13)

Prepared following the general procedure outlined above using (*E*)-(2-iodovinyl)cyclohexane (118 mg, 0.50 mmol, 1.0 equiv.), tetrahydrofuran-2-carboxylic acid (99 mg, 0.85 mmol, 1.70 equiv.), $Ir[dF(Me)ppy]_2(dtbbpy)PF_6$ (5.1 mg, 5.0 μmol, 0.01 equiv.), $NiCl_2$·glyme (2.2 mg, 10 μmol, 0.02 equiv.), dtbbpy (2.7 mg, 10 μmol, 0.02 equiv.), DBU (129 mg, 0.85 mmol, 1.70 equiv.), and DMSO (5.0 mL). After 18 h, the reaction mixture was subjected to the workup protocol outlined in the general procedure. Purification by flash column chromatography (5% Et_2O/pentane) provided the title compound (81 mg, 0.45 mmol, 90%) as a colorless oil. IR (film) ν_{max} 2970, 2923, 2851, 1449, 1056, 967 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 1.03–1.11 (2H, m, CyC**H$_2$**), 1.16 (1H, tt, *J* = 12.5, 3.1, CyC**H$_2$**), 1.22–1.30 (2H, m, CyC**H$_2$**), 1.54–1.66 (2H, m, $OCH_2(CH_2)_2$ + CyC**H$_2$**), 1.70–1.74 (4H, m, CyC**H$_2$**), 1.84–2.05 (4H, m, $OCH_2(CH_2)_2$ + OCHCH=CHC**H**), 3.76 (1H, td, *J* = 8.0, 6.0, OC**H$_2$**), 3.90 (1H, td, *J* = 7.9, 6.5, OC**H$_2$**), 4.21 (1H, app. q, *J* = 7.2, OC**H**CH=CH), 5.40 (1H, dd, *J* = 15.5, 7.2, OCHC**H**=CH), 5.63 (1H, dd, *J* = 15.5, 6.6, OCHCH=C**H**); ^{13}C NMR (126 MHz, $CDCl_3$) δ 26.0, 26.0, 26.2, 32.3, 32.7, 32.8, 40.3, 67.9, 80.2, 128.0, 138.7; HRMS (ESI-TOF) *m/z* calcd. for $C_{12}H_{21}O$ ($[M+H]^+$) 181.1587, found 181.1587.



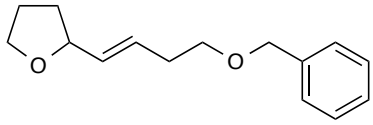
(*E*)-2-(3, 3-dimethylbut-1-en-1-yl)tetrahydrofuran (14)

Prepared following the general procedure outlined above using (*E*)-1-iodo-3, 3-dimethyl-but-1-ene (105 mg, 0.50 mmol, 1.0 equiv.), tetrahydrofuran-2-carboxylic acid (99 mg, 0.85 mmol, 1.70 equiv.), $Ir[dF(Me)ppy]_2(dtbbpy)PF_6$ (5.1 mg, 5.0 μmol, 0.01 equiv.), $NiCl_2$·glyme (2.2 mg, 10 μmol, 0.02 equiv.), dtbbpy (2.7 mg, 10 μmol, 0.02 equiv.), DBU (129 mg, 0.85 mmol, 1.70 equiv.), and DMSO (5.0 mL). After 18 h, the reaction mixture was subjected to the workup protocol outlined in the general procedure. Purification by flash column chromatography (5% Et_2O/pentane) provided the title compound (60 mg, 0.39 mmol, 78%) as a colorless oil. IR (film) ν_{max} 2957, 2905, 2866, 1461, 1362, 1056, 970 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 1.02 (9H, s, $C(CH_3)_3$), 1.55–1.62 (1H, m, $OCH_2(CH_2)_2$), 1.92 (2H, m, $OCH_2(CH_2)_2$), 2.02 (1H, m, $OCH_2(CH_2)_2$), 3.77 (1H, td, *J* = 8.0, 6.2, OCH_2), 3.91 (1H, td, *J* = 8.0, 6.2, OCH_2), 4.21 (1H, app. q, *J* = 7.3, OC**H**CH=CH), 5.36 (1H, dd, *J* = 15.4, 7.3, OCHC**H**=CH), 5.70 (1H, d, *J* = 15.4, OCHCH=C**H**); ^{13}C NMR (126 MHz, $CDCl_3$) δ 26.0, 29.5, 32.4, 32.8, 67.9, 80.4, 125.3, 143.8; HRMS (ESI-TOF) *m*/*z* calcd. for $C_{10}H_{19}O$ ($[M+H]^+$) 155.1430, found 155.1434.



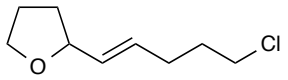
(*E*)-2-(3-phenylprop-1-en-1-yl)tetrahydrofuran (15)

Prepared following the general procedure outlined above using (*E*)-(3-iodoallyl)benzene (78 μL, 0.50 mmol, 1.0 equiv.), tetrahydrofuran-2-carboxylic acid (87 mg, 0.75 mmol, 1.50 equiv.), $Ir[dF(CF_3)ppy]_2(dtbbpy)PF_6$ (5.6 mg, 5.0 μmol, 0.01 equiv.), $NiCl_2$·glyme (11 mg, 50 μmol, 0.10 equiv.), dtbbpy (20 mg, 75 μmol, 0.15 equiv.), Cs_2CO_3 (277 mg, 0.850 mmol, 1.70 equiv.), and DMF (25.0 mL). After 72 h, the reaction mixture was subjected to the workup protocol outlined in the general procedure. Purification by flash column chromatography (10% EtOAc/hexanes) provided the title compound (70 mg, 0.37 mmol, 74%) as a colorless oil. IR (film) ν_{max} 3062, 2867, 1051, 969, 699 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 1.56–1.66 (1H, m, $OCH_2(CH_2)_2$), 1.85–1.99 (2H, m, $OCH_2(CH_2)_2$), 2.00–2.08 (1H, m, $OCH_2(CH_2)_2$), 3.39 (2H, d, *J* = 7.1, CH=CHCH_2), 3.78 (1H, td, *J* = 7.8, 6.2, OCH_2), 3.91 (1H, td, *J* = 7.8, 6.2, OCH_2), 4.28 (1H, q, *J* = 7.1, OC**H**CH=CH), 5.55 (1H, dd, *J* = 15.5, 7.1, OCHC**H**=CH), 5.84 (1H, dt, *J* = 15.5, 6.8, OCHCH=C**H**), 7.17–7.24 (3H, m, Ar**H**), 7.29 (2H, t, *J* = 7.5, Ar**H**); ^{13}C NMR (126 MHz, $CDCl_3$) δ 25.9, 32.2, 38.6, 68.0, 79.6, 126.0, 128.4, 128.6, 130.9, 132.2, 140.1; HRMS (ESI-TOF) *m*/*z* calcd. for $C_{13}H_{17}O$ ($[M+H]^+$) 189.1274, found 189.1274.



(*E*)-2-(4-(benzyloxy)but-1-en-1-yl)tetrahydrofuran (16)

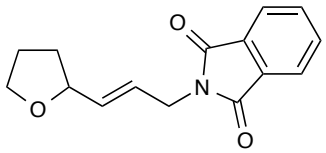
Prepared following the general procedure outlined above using (*E*)-(((4-iodobutyl-3-en-1-yl)oxy)methyl)benzene (144 mg, 0.50 mmol, 1.0 equiv.), tetrahydrofuran-2-carboxylic acid (99 mg, 0.85 mmol, 1.70 equiv.), $Ir[dF(Me)ppy]_2(dtbbpy)PF_6$ (5.1 mg, 5.0 μmol, 0.01 equiv.), $NiCl_2$·glyme (2.2 mg, 10 μmol, 0.02 equiv.), dtbbpy (2.7 mg, 10 μmol, 0.02 equiv.), DBU (129 mg, 0.85 mmol, 1.70 equiv.), and DMSO (5.0 mL). After 18 h, the reaction mixture was subjected to the workup protocol outlined in the general procedure. Purification by flash column chromatography (5% Et_2O/pentane) provided the title compound (89 mg, 0.39 mmol, 77%) as a colorless oil. IR (film) ν_{max} 2858, 1361, 1098, 1051, 967, 912, 731, 697 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 1.56–1.63 (1H, m, $OCH_2(CH_2)_2$), 1.82–2.10 (3H, m, $OCH_2(CH_2)_2$), 2.38 (2H, q, *J* = 6.9, CH=CHC**H**$_2$CH$_2$), 3.52 (2H, td, *J* = 6.9, 1.8, CH=CHCH$_2$C**H**$_2$), 3.77 (1H, q, *J* = 7.5, OC**H**$_2$(CH$_2$)$_2$), 3.90 (1H, q, *J* = 7.3, OC**H**$_2$(CH$_2$)$_2$), 4.25 (1H, q, *J* = 7.1, OC**H**CH=CH), 4.52 (2H, s, OC**H**$_2$Ph), 5.55 (1H, dd, *J* = 15.4, 7.1, OCHC**H**=CH), 5.71 (1H, dt, *J* = 15.4, 6.8, OCHCH=C**H**), 7.28–7.38 (5H, m, Ar**H**).; ^{13}C NMR (126 MHz, $CDCl_3$) δ 25.9, 32.1, 67.9, 69.7, 72.9, 79.7, 127.5, 127.5, 127.6, 128.3, 128.6, 132.7, 138.4; HRMS (ESI-TOF) *m/z* calcd. for $C_{15}H_{21}O_2$ ($[M+H]^+$) 233.1536, found 233.1541.



(*E*)-2-(5-chloropent-1-en-1-yl)tetrahydrofuran (17)

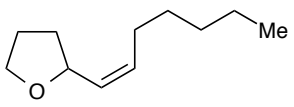
Prepared following the general procedure outlined above using (*E*)-5-chloro-1-iodopent-1-ene (115 mg, 0.50 mmol, 1.0 equiv.), tetrahydrofuran-2-carboxylic acid (99 mg, 0.85 mmol, 1.70 equiv.), $Ir[dF(Me)ppy]_2(dtbbpy)PF_6$ (5.1 mg, 5.0 μmol, 0.010 equiv.), $NiCl_2$·glyme (2.2 mg, 10 μmol, 0.02 equiv.), dtbbpy (2.7 mg, 10 μmol, 0.02 equiv.), DBU (129 mg, 0.850 mmol, 1.7 equiv.), and DMSO (5.0 mL). After 18 h, the reaction mixture was subjected to the workup protocol outlined in the general procedure. Purification by flash column chromatography (5–20% Et_2O/pentane) provided the title compound (59 mg, 0.34 mmol, 68%) as a colorless oil. IR (film)

ν_{max} 2957, 2868, 1444, 1052, 969, 725 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 1.54–1.64 (1H, m, $OCH_2(CH_2)_2$), 1.82–2.08 (5H, m, $OCH_2(CH_2)_2$ + CH_2CH_2Cl), 2.16–2.24 (2H, m,), 3.54 (2H, t, *J* = 6.6, CH_2Cl), 3.77 (1H, td, *J* = 7.9, 6.3, OCH_2), 3.90 (1H, td, *J* = 7.9, 6.3, OCH_2), 4.24 (1H, q, *J* = 6.9, OCHCH=CH), 5.53 (1H, ddt, *J* = 15.4, 7.0, 1.4, OCHCH=CH), 5.65 (1H, dt, *J* = 15.4, 6.9, OCHCH=CH); ^{13}C NMR (126 MHz, $CDCl_3$) δ 25.9, 29.3, 32.2, 44.4, 68.0, 79.7, 130.3, 132.2; HRMS (ESI-TOF) *m/z* calcd. for $C_9H_{16}ClO$ ($[M+H]^+$) 175.0884, found 175.0882.



(*E*)-2-(3-(tetrahydrofuran-2-yl)allyl)isoindoline-1, 3-dione (18)

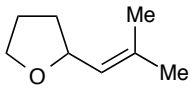
Prepared following the general procedure outlined above using (*E*)-2-(3-iodoallyl)isoindoline-1, 3-dione (157 mg, 0.50 mmol, 1.0 equiv.), tetrahydrofuran-2-carboxylic acid (87 mg, 0.75 mmol, 1.50 equiv.), $Ir[dF(CF_3)ppy]_2(dtbbpy)PF_6$ (5.6 mg, 5.0 μmol, 0.01 equiv.), $NiCl_2$·glyme (22.0 mg, 100 μmol, 0.20 equiv.), dtbbpy (27.0 mg, 100 μmol, 0.20 equiv.), Cs_2CO_3 (277 mg, 0.85 mmol, 1.70 equiv.), and DMF (25.0 mL). After 72 h, the reaction mixture was subjected to the workup protocol outlined in the general procedure. Purification by flash column chromatography (10-30% EtOAc/hexanes) provided the title compound (86 mg, 0.34 mmol, 67%) as a clear oil. IR (film) ν_{max} 2973–2870, 1771, 1712, 1429, 1393, 1054, 721 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 1.55–1.65 (1H, m, $OCH_2(CH_2)_2$), 1.82–1.96 (2H, m, $OCH_2(CH_2)_2$), 1.99–2.05 (1H, m, $OCH_2(CH_2)_2$), 3.76 (1H, td, *J* = 7.9, 6.1, OCH_2), 3.84–3.91 (1H, m, OCH_2), 4.25–4.32 (3H, m, OCHCH=CH + CH=CHCH_2N), 5.71–5.84 (2H, m, OCHCH=CH + OCHCH=CH), 7.72 (2H, dd, *J* = 5.5, 3.0, ArH), 7.85 (2H, dd, *J* = 5.5, 3.0, ArH).; ^{13}C NMR (126 MHz, $CDCl_3$) δ 25.69, 31.95, 38.99, 68.07, 78.57, 123.25, 124.09, 132.13, 133.91, 135.01, 167.86; HRMS (ESI-TOF) *m/z* calcd. for $C_{15}H_{16}NO_3$ ($[M+H]^+$) 258.1125, found 258.1121.



(*Z*)-2-(Hept-1-en-1-yl)tetrahydrofuran (19)

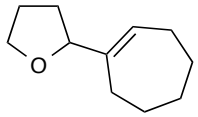
Prepared following the general procedure outlined above using (*Z*)-1-iodohept-1-ene (112 mg, 0.50 mmol, 1.0 equiv., 97:3 *Z*:*E*), tetrahydrofuran-2-carboxylic acid (87 mg, 0.75 mmol, 1.50

equiv.), Ir[dF(CF_3)ppy]$_2$(dtbbpy)PF_6 (5.6 mg, 5.0 μmol, 0.01 equiv.), $NiCl_2$·glyme (11 mg, 50 μmol, 0.10 equiv.), dtbbpy (20 mg, 75 μmol, 0.15 equiv.), Cs_2CO_3 (277 mg, 0.85 mmol, 1.70 equiv.), and DMF (25.0 mL). After 70 h, the reaction mixture was subjected to the workup protocol outlined in the general procedure. Purification by flash column chromatography (0–20% EtOAc/hexanes) provided the title compound (71 mg, 0.42 mmol, 84%, 95:5 *Z*:*E*) as a colorless oil. IR (film) ν_{max} 3015-2858, 1461, 1056 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 0.89 (3H, t, *J* = 6.8, $(CH_2)_3C\mathbf{H_3}$), 1.25–1.44 (6H, m, $(C\mathbf{H_2})_3CH_3$), 1.50–1.58 (1H, m, $OCH_2(C\mathbf{H}_2)_2$), 1.86–2.16 (5H, m, $OCH_2(C\mathbf{H}_2)_2$ + $CH{=}CHC\mathbf{H_2}$), 3.76 (1H, td, *J* = 8.0, 5.9, $OC\mathbf{H}_2$), 3.89–3.94 (1H, m, $OC\mathbf{H}_2$), 4.58 (1H, td, *J* = 8.3, 6.5, O**H**CH=CH), 5.40–5.44 (1H, m, OCHC**H**=CH), 5.51 (1H, dt, *J* = 10.9, 7.4, OCHCH=C**H**); ^{13}C NMR (126 MHz, $CDCl_3$) δ 14.1, 22.5, 26.2, 27.7, 29.4, 31.5, 32.7, 67.9, 74.8, 130.6, 132.6; HRMS (ESI-TOF) *m*/*z* calcd. for $C_{11}H_{21}O$ ($[M+H]^+$) 167.1430, found 167.1430.



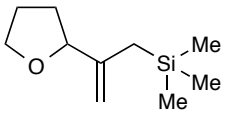
2-(2-Methylprop-1-en-1-yl)tetrahydrofuran (20)

Prepared following the general procedure outlined above using 1-bromo-2-methylprop-1-ene (68 mg, 0.50 mmol, 1.0 equiv.), tetrahydrofuran-2-carboxylic acid (105 mg, 0.90 mmol, 1.80 equiv.), Ir[dF(Me)ppy]$_2$(dtbbpy)PF_6 (5.1 mg, 5.0 μmol, 0.01 equiv.), $NiCl_2$·glyme (2.2 mg, 10 μmol, 0.02 equiv.), dtbbpy (2.7 mg, 10 μmol, 0.02 equiv.), Cs_2CO_3 (293 mg, 0.90 mmol, 1.80 equiv.), *N*-Boc-benzylamine (6.2 mg, 30 μmol, 0.06 equiv.), and DMA (5.0 mL). After 24 h, the reaction mixture was subjected to the workup protocol outlined in the general procedure. Purification by flash column chromatography (10% Et_2O/pentane) provided the title compound (45 mg, 71%) as a colorless oil. IR (film) ν_{max} 2970, 2930, 2870, 1445, 1377, 1050 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 1.52 (1H, dq, *J* = 11.8, 8.2, $OCH_2(C\mathbf{H}_2)_2$), 1.71 (3H, s, $C\mathbf{H_3}$), 1.73 (3H, s, $C\mathbf{H_3}$), 1.85–2.06 (3H, m, $OCH_2(C\mathbf{H}_2)_2$), 3.75 (1H, td, *J* = 8.0, 5.8, $OC\mathbf{H}_2$), 3.90 (1H, q, *J* = 7.3, $OC\mathbf{H}_2$), 4.50 (1H, td, *J* = 8.4, 6.1, O**H**CH=CMe_2), 5.21 (1H, d, *J* = 8.4, C**H**=CMe_2); ^{13}C NMR (126 MHz, $CDCl_3$) δ 18.2, 25.9, 26.3, 32.5, 67.7, 75.8, 125.9, 135.9; HRMS (ESI-TOF) *m*/*z* calcd. for $C_8H_{15}O$ ($[M+H]^+$) 127.1117, found 127.1117.



2-(Cyclohept-1-en-1-yl)tetrahydrofuran (21)

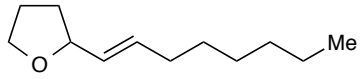
Prepared following the general procedure outlined above using 1-bromocyclohept-1-ene (88 mg, 0.50 mmol, 1.0 equiv.), tetrahydrofuran-2-carboxylic acid (105 mg, 0.90 mmol, 1.80 equiv.), $Ir[dF(Me)ppy]_2(dtbbpy)PF_6$ (5.1 mg, 5.0 μmol, 0.01 equiv.), $NiCl_2$·glyme (2.2 mg, 10 μmol, 0.02 equiv.), dtbbpy (2.7 mg, 10 μmol, 0.02 equiv.), Cs_2CO_3 (293 mg, 0.90 mmol, 1.80 equiv.), *N*-Boc-benzylamine (6.2 mg, 30 μmol, 0.06 equiv.), and DMA (5.0 mL). After 24 h, the reaction mixture was subjected to the workup protocol outlined in the general procedure. Purification by flash column chromatography (10% Et_2O/pentane) provided the title compound (61 mg, 0.37 mmol, 73%) as a colorless oil. IR (film) ν_{max} 2969, 2919, 2849, 1446, 1081, 1054 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 1.43–1.55 (4H, m, C**H**$_2$), 1.59–1.66 (1H, m, $OCH_2(CH_2)_2$), 1.71–1.78 (2H, m, C**H**$_2$), 1.84–1.96 (3H, m, $OCH_2(CH_2)_2$), 2.04–2.18 (4H, m, C**H**$_2$C=CHC**H**$_2$), 3.77–3.81 (1H, m, OC**H**$_2$), 3.90–3.95 (1H, m, OC**H**$_2$), 4.18 (1H, dd, *J* = 8.1, 6.1, OC**H**C=CH), 5.83 (1H, t, *J* = 6.1, OCHC=C**H**); ^{13}C NMR (126 MHz, $CDCl_3$) δ 26.0, 27.0, 27.3, 28.2, 28.3, 30.3, 32.7, 68.4, 84.2, 127.5, 144.2; HRMS (ESI-TOF) *m/z* calcd. for $C_{11}H_{19}O$ ($[M+H]^+$) 167.1434, found 167.1430.



Trimethyl(2-(tetrahydrofuran-2-yl)allyl)silane (22)

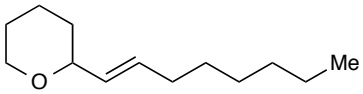
Prepared following the general procedure outlined above using (2-bromoallyl)trimethylsilane (97 mg, 0.50 mmol, 1.0 equiv.), tetrahydrofuran-2-carboxylic acid (87 mg, 0.75 mmol, 1.50 equiv.), $Ir[dF(CF_3)ppy]_2(dtbbpy)PF_6$ (5.6 mg, 5.0 μmol, 0.01 equiv.), $NiCl_2$·glyme (11 mg, 50 μmol, 0.10 equiv.), dtbbpy (20 mg, 75 μmol, 0.15 equiv.), Cs_2CO_3 (277 mg, 0.850 mmol, 1.70 equiv.), and DMF (25.0 mL). After 72 h, the reaction mixture was subjected to the workup protocol outlined in the general procedure. Purification by flash column chromatography (0–20% EtOAc/hexanes) provided the title compound (55 mg, 0.30 mmol, 60%) as a colorless oil. IR (film) ν_{max} 3078, 2954, 2872, 1248, 1161, 1067, 882, 849 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 0.04 (9H, s, $Si(CH_3)_3$), 1.38 (1H, d, *J* = 13.9, C**H**$_2$SiMe$_3$), 1.61 (1H, d, *J* = 14.0, C**H**$_2$SiMe$_3$), 1.63–1.70 (1H,

m, $OCH_2(C\mathbf{H_2})_2$), 1.83–1.96 (2H, m, $OCH_2(C\mathbf{H_2})_2$), 2.01–2.08 (1H, m, $OCH_2(C\mathbf{H_2})_2$), 3.81 (1H, dd, *J* = 7.9, 6.5, $OC\mathbf{H}_2$), 3.95 (1H, dd, *J* = 8.2, 7.2, $OC\mathbf{H}_2$), 4.20 (1H, s, $OC\mathbf{H}C{=}CH_2$), 4.61 (1H, s, $OCHC{=}C\mathbf{H}_2$), 4.91 (1H, t, *J* = 1.6, $OCHC{=}C\mathbf{H}_2$); ^{13}C NMR (126 MHz, $CDCl_3$) δ 0.0, 24.0, 26.8, 32.4, 69.5, 83.1, 107.4, 149.1; HRMS (ESI-TOF) *m/z* calcd. for $C_{10}H_{21}OSi$ ($[M+H]^+$) 185.1351, found 185.1356.



(*E*)-2-(Oct-1-en-1-yl)tetrahydrofuran (23)

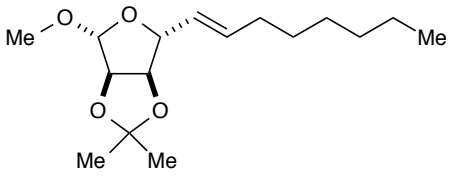
Prepared following the general procedure outlined above using (*E*)-1-iodooct-1-ene (87 μL, 0.50 mmol, 1.0 equiv.), tetrahydrofuran-2-carboxylic acid (99 mg, 0.85 mmol, 1.70 equiv.), $Ir[dF(Me)ppy]_2(dtbbpy)PF_6$ (5.1 mg, 5.0 μmol, 0.01 equiv.), $NiCl_2$·glyme (2.2 mg, 10 μmol, 0.02 equiv.), dtbbpy (2.7 mg, 10 μmol, 0.02 equiv.), DBU (129 mg, 0.85 mmol, 1.70 equiv.), and DMSO (5.0 mL). After 18 h, the reaction mixture was subjected to the workup protocol outlined in the general procedure. Purification by flash column chromatography (5% Et_2O/pentanes) provided the title compound (84 mg, 0.46 mmol, 92%) as a colorless oil. IR (film) ν_{max} 2957–2855, 1461, 1056, 965 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.88 (3H, t, *J* = 6.9, $C\mathbf{H_3}$), 1.23–1.40 (8H, m, $(C\mathbf{H_2})_4CH_3$), 1.57–1.62 (1H, m, $OCH_2(C\mathbf{H_2})_2$), 1.85–2.05 (5H, m, $CH{=}CHC\mathbf{H_2}$ + $OCH_2(C\mathbf{H_2})_2$), 3.76 (1H, td, *J* = 7.9, 6.2, $OC\mathbf{H}_2$), 3.90 (1H, td, *J* = 7.9, 6.2, $OC\mathbf{H}_2$), 4.23 (1H, q, *J* = 7.1, $OC\mathbf{H}CH{=}CH$), 5.45 (1H, dd, *J* = 15.3, 7.2, $OCHC\mathbf{H}{=}CH$), 5.68 (1H, dt, *J* = 15.3, 6.7, $OCHCH{=}C\mathbf{H}$); ^{13}C NMR (126 MHz, $CDCl_3$) δ 14.1, 22.6, 26.0, 28.9, 29.1, 31.7, 32.3, 32.3, 67.9, 80.0, 130.5, 133.0; HRMS (ESI-TOF) *m/z* calcd. for $C_{12}H_{23}O$ ($[M+H]^+$) 183.1742, found 183.1743.



(*E*)-2-(Oct-1-en-1-yl)tetrahydro-2*H*-pyran (24)

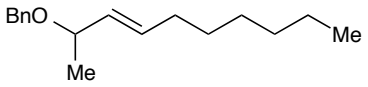
Prepared following the general procedure outlined above using (*E*)-1-iodooct-1-ene (87 μL, 0.50 mmol, 1.0 equiv.), tetrahydro-2*H*-pyran-2-carboxylic acid (195 mg, 1.50 mmol, 3.0 equiv.), $Ir[dF(Me)ppy]_2(dtbbpy)PF_6$ (5.1 mg, 5.0 μmol, 0.010 equiv.), $NiCl_2$·glyme (5.5 mg, 25 μmol, 0.05 equiv.), dtbbpy (6.7 mg, 25 μmol, 0.05 equiv.), Cs_2CO_3 (489 mg, 1.50 mmol, 3.0 equiv.),

and DMSO (20.0 mL). After 24 h, the reaction mixture was subjected to the workup protocol outlined in the general procedure. Purification by flash column chromatography (5% Et_2O/pentane) provided the title compound (73 mg, 0.37 mmol, 74%) as a colorless oil. IR (film) ν_{max} 2928, 2854, 1463, 1440, 1204, 1087, 1050, 1036 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 0.88 (3H, t, *J* = 6.8, C**H**$_3$), 1.23–1.65 (13H, m, C**H**$_2$), 1.82–1.86 (1H, m, C**H**$_2$), 2.00–2.04 (2H, m, C**H**$_2$), 3.48 (1H, td, *J* = 11.6, 2.3, OC**H**$_2$), 3.72–3.76 (1H, m, OC**H**$_2$), 3.99–4.03 (1H, m, OC**H**CH=CH), 5.47 (1H, dd, *J* = 15.5, 6.3, OCHC**H**=CH), 5.64–5.70 (1H, m, OCHCH=C**H**); ^{13}C NMR (126 MHz, $CDCl_3$) δ 14.1, 22.6, 23.5, 25.9, 28.9, 29.1, 31.7, 32.2, 32.4, 68.4, 78.3, 131.1, 132.0; HRMS (ESI-TOF) *m*/*z* calcd. for $C_{13}H_{25}O$ ($[M+H]^+$) 197.1896, found 197.1900.



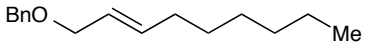
4-Methoxy-2,2-dimethyl-6-((*E*)-oct-1-en-1-yl)tetrahydrofuro[3, 4-*d*][1,3]dioxole (25)

Prepared following the general procedure outlined above using (*E*)-1-iodooct-1-ene (87 μL, 0.50 mmol, 1.0 equiv.), 2, 3-O-isopropylidene-1-O-methyl-D-ribosic acid (164 mg, 0.75 mmol, 1.5 equiv.), $Ir[dF(CF_3)ppy]_2(dtbbpy)PF_6$ (5.6 mg, 5.0 μmol, 0.010 equiv.), $NiCl_2$·glyme (11 mg, 50 μmol, 0.10 equiv.), dtbbpy (20 mg, 75 μmol, 0.15 equiv.), Cs_2CO_3 (277 mg, 0.850 mmol, 1.70 equiv.), and DMF (25.0 mL). After 72 h, the reaction mixture was subjected to the workup protocol outlined in the general procedure. Purification by flash column chromatography (0–20% EtOAc/hexanes) provided the title compound (126 mg, 0.45 mmol, 89%, 18:1 dr) as a colorless oil. IR (film) ν_{max} 2926, 2856, 1462, 1372, 1271, 1209, 1161, 1089, 1104, 1055, 1028, 964, 869, 829, 774 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 0.89 (3H, t, *J* = 6.6, C**H**$_3$), 1.22–1.42 (11H, m, ((C**H**$_2$)$_4$CH$_3$) + OC(C**H**$_3$)$_2$), 1.50 (3H, s, OC(C**H**$_3$)$_2$), 2.02 (2H, q, *J* = 6.9, CH=CHC**H**$_2$), 3.34 (3H, s, OC**H**$_3$), 4.56–4.65 (3H, m, C**H** + C**H** + C**H**), 4.97 (1H, s, C**H**), 5.49 (1H, dd, *J* = 15.4, 8.9, OCHC**H**=CH), 5.69 (1H, dt, *J* = 15.4, 6.7, OCHCH=C**H**); ^{13}C NMR (126 MHz, $CDCl_3$) δ 14.1, 22.6, 25.0, 26.5, 28.8, 28.9, 31.7, 32.1, 54.5, 84.8, 85.7, 88.4, 109.0, 112.2, 129.3, 134.9; HRMS (ESI-TOF) *m*/*z* calcd. for $C_{16}H_{29}O_4$ ($[M+H]^+$) 285.2060, found 285.2062.



(*E*)-((Dec-3-en-2-yloxy)methyl)benzene (26)

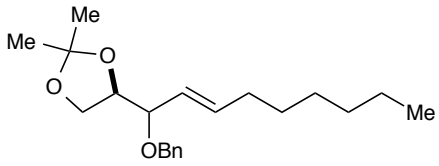
Prepared following the general procedure outlined above using (*E*)-1-iodooct-1-ene (87 μL, 0.50 mmol, 1.0 equiv.), 2-(benzyloxy)propanoic acid (162 mg, 0.90 mmol, 1.80 equiv.), $Ir[dF(Me)ppy]_2(dtbbpy)PF_6$ (5.1 mg, 5.0 μmol, 0.01 equiv.), $NiCl_2$·glyme (2.2 mg, 10 μmol, 0.02 equiv.), dtbbpy (2.7 mg, 10 μmol, 0.02 equiv.), Cs_2CO_3 (293 mg, 0.90 mmol, 1.80 equiv.), *N*-Boc benzylamine (6.2 mg, 30 μmol, 0.06 equiv.), and DMA (5.0 mL). After 18 h, the reaction mixture was subjected to the workup protocol outlined in the general procedure. Purification by flash column chromatography (5% Et_2O/pentane) followed by preparative TLC to remove the ester byproduct, provided the title compound (96 mg, 0.39 mmol, 78%) as a colorless oil. IR (film) ν_{max} 3030, 2958, 2926, 2855, 1454, 1370, 1096, 1071, 1028, 970 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 0.90 (3H, t, *J* = 6.7, CH_2CH_3), 1.27–1.43 (11H, m, $(CH_2)_4CH_3$ + $OCHCH_3$), 2.06 (2H, q, *J* = 7.0, $CH{=}CHCH_2$), 3.83–3.93 (1H, m, $OCHCH_3$), 4.37 (1H, d, *J* = 12.0, OCH_2Ph), 4.56 (1H, d, *J* = 12.0, OCH_2Ph), 5.39 (1H, dd, *J* = 15.4, 8.0, $CH{=}CHCH_2$), 5.62 (1H, dt, *J* = 15.3, 6.7, $CH{=}CHCH_2$), 7.25–7.37 (5H, m, Ar**H**); ^{13}C NMR (126 MHz, $CDCl_3$) δ 14.1, 21.8, 22.7, 28.9, 29.2, 31.7, 32.2, 69.6, 75.9, 127.3, 127.7, 128.3, 131.8, 133.5, 139.0; HRMS (ESI-TOF) *m*/*z* calcd. for $C_{17}H_{26}ONa$ ($[M+Na]^+$) 269.1875, found 269.1876.



(*E*)-((Non-2-en-1-yloxy)methyl)benzene (27)

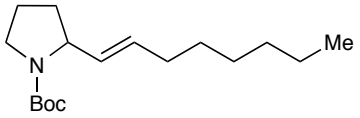
Prepared following the general procedure outlined above using (*E*)-1-iodooct-1-ene (87 μL, 0.50 mmol, 1.0 equiv.), 2-(benzyloxy)acetic acid (107 μL, 0.75 mmol, 1.50 equiv.), $Ir[dF(CF_3)ppy]_2(dtbbpy)PF_6$ (5.6 mg, 5.0 μmol, 0.01 equiv.), $NiCl_2$·glyme (11 mg, 50 μmol, 0.10 equiv.), dtbbpy (20 mg, 75 μmol, 0.15 equiv.), Cs_2CO_3 (277 mg, 0.850 mmol, 1.70 equiv.), and DMF (25.0 mL). After 72 h, the reaction mixture was subjected to the workup protocol outlined in the general procedure. Purification by flash column chromatography (0–20% EtOAc/hexanes) provided the title compound (90 mg, 0.39 mmol, 77%) as a colorless oil. IR (film) ν_{max} 3029, 2956, 2926, 2854, 1455, 1361, 1105, 1070 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 0.89 (3H, t, *J* = 6.9, CH_3), 1.26–1.42 (8H, m, $(CH_2)_4CH_3$), 2.06 (2H, q, *J* = 6.9, $CH{=}CHCH_2$), 3.98 (2H, d, *J* = 6.2, $OCH_2CH{=}CH$), 4.51 (2H, s, OCH_2Ph), 5.57–5.63 (1H, m, $OCH_2CH{=}CH$), 5.70–5.76 (1H,

m, $OCH_2CH{=}C\mathbf{H}$), 7.27–7.38 (5H, m, Ar**H**); ^{13}C NMR (126 MHz, $CDCl_3$) δ 14.1, 22.6, 28.9, 29.1, 31.7, 32.4, 71.0, 71.8, 126.1, 127.5, 127.8, 128.4, 135.2, 138.5; HRMS (ESI-TOF) *m*/*z* calcd. for $C_{16}H_{25}O$ ($[M+H]^+$) 233.1896, found 233.1900.



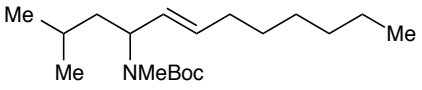
(4*R*)-4-(*E*)-1-(benzyloxy)non-2-en-yl-2,2-dimethyl-1,3-dioxolane (28)

Prepared following the general procedure outlined above using (*E*)-1-bromooct-1-ene (87 μL, 0.50 mmol, 1.0 equiv.), (*R*)-2-(benzyloxy)-2-((*R*)-2,2-dimethyl-1,3-dioxolan-4-yl)acetic acid (240 mg, 0.90 mmol, 1.80 equiv.), $Ir[dF(Me)ppy]_2(dtbbpy)PF_6$ (5.1 mg, 5.0 μmol, 0.01 equiv.), $NiCl_2$·glyme (2.2 mg, 10 μmol, 0.02 equiv.), dtbbpy (2.7 mg, 10 μmol, 0.02 equiv.), Cs_2CO_3 (293 mg, 0.90 mmol, 1.80 equiv.), *N*-Boc benzylamine (6.2 mg, 30 μmol, 0.06 equiv.), and DMA (5.0 mL). After 18 h, the reaction mixture was subjected to the workup protocol outlined in the general procedure. Purification by flash column chromatography (0–10% Et_2O/pentane) provided the title compound (132 mg, 0.40 mmol, 79%, 1.4:1.0 dr) as a colorless oil. The inseparable diastereomers were characterized as a mixture. IR (film) ν_{max} 2927, 2857, 1455, 1370, 1211, 1068, 969, 851, 732, 697 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) For major isomer δ 0.90 (3H, t, *J* = 6.7, $(CH_2)_5\mathbf{CH_3}$), 1.25–1.47 (14H, m, $CH{=}CHCH_2(C\mathbf{H_2})_4CH_3$ + $C(C\mathbf{H_3})_2$), 2.04–2.20 (2H, m, $CH{=}CHC\mathbf{H_2}$), 3.69–4.22 (4H, m, $C\mathbf{H_2}OC\mathbf{H}OC\mathbf{H}CH{=}CH$), 4.39 (1H, d, *J* = 11.9, $OC\mathbf{H_2}Ph$), 4.64 (1H, d, *J* = 11.9, $OC\mathbf{H_2}Ph$), 5.42 (dd, *J* = 15.5, 8.2, $C\mathbf{H}{=}CHCH_2$), 5.72 (dt, *J* = 15.5, 6.8, $CH{=}C\mathbf{H}CH_2$), 7.28–7.38 (5H, m, Ar**H**). For minor isomer δ 0.90 (3H, t, *J* = 6.7, $(CH_2)_5\mathbf{CH_3}$), 1.25–1.47 (14H, m, $CH{=}CHCH_2(C\mathbf{H_2})_4CH_3$ + $C(C\mathbf{H_3})_2$), 2.04–2.20 (2H, m, $CH{=}CHC\mathbf{H_2}$), 3.69–4.22 (4H, m, $C\mathbf{H_2}OC\mathbf{H}OC\mathbf{H}CH{=}CH$), 4.47 (1H, d, *J* = 12.4, $OC\mathbf{H_2}Ph$), 4.68 (1H, d, *J* = 12.4, $OC\mathbf{H_2}Ph$), 5.31 (1H, dd, *J* = 15.5, 8.6, $C\mathbf{H}{=}CHCH_2$), 5.72 (dt, *J* = 15.5, 6.8, $CH{=}C\mathbf{H}CH_2$), 7.28–7.38 (5H, m, Ar**H**).; ^{13}C NMR (126 MHz, $CDCl_3$) δ 14.1, 22.6, 25.4, 25.4, 26.5, 28.8, 29.0, 29.1, 31.6, 31.7, 32.3, 32.4, 66.0, 66.8, 69.6, 70.0, 77.8, 77.9, 80.5, 80.9, 109.4, 109.7, 125.7, 126.4, 127.4, 127.5, 127.7, 127.8, 128.2, 128.3, 137.3, 137.7, 138.4, 138.5; HRMS (ESI-TOF) *m*/*z* calcd. for $C_{21}H_{33}O_3$ ($[M+H]^+$) 333.2424, found 333.2432.



***tert*-Butyl (*E*)-2-(oct-1-en-1-yl)pyrrolidine-1-carboxylate (29)**

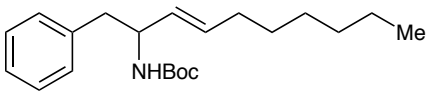
Prepared following the general procedure outlined above using (*E*)-1-iodooct-1-ene (87 μL, 0.50 mmol, 1.0 equiv.), Boc-Pro-OH (183 mg, 0.85 mmol, 1.70 equiv.), $Ir[dF(Me)ppy]_2(dtbbpy)PF_6$ (5.1 mg, 5.0 μmol, 0.01 equiv.), $NiCl_2$·glyme (2.2 mg, 10 μmol, 0.02 equiv.), dtbbpy (2.7 mg, 10 μmol, 0.02 equiv.), DBU (129 mg, 0.850 mmol, 1.70 equiv.), and DMSO (5.0 mL). After 18 h, the reaction mixture was subjected to the workup protocol outlined in the general procedure. Purification by flash column chromatography (10% Et_2O/pentane) provided the title compound (127 mg, 0.45 mmol, 90%) as a colorless oil. IR (film) ν_{max} 2961, 2956, 2856, 1694, 1390, 1364, 1170, 1116 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 0.88 (3H, t, *J* = 6.7, $CH_2C\mathbf{H_3}$), 1.27–1.37 (8H, m, $C\mathbf{H}_2$), 1.44 (9H, s, $OC(C\mathbf{H_3})_3$), 1.64–1.69 (1H, m, $C\mathbf{H}_2$), 1.75–1.89 (2H, m, $C\mathbf{H}_2$), 1.94–2.02 (3H, m, $C\mathbf{H}_2$), 3.32–3.41 (2H, m, $NC\mathbf{H_2}$), 4.22 (1H, br s, $NC\mathbf{H}CH{=}CH$), 5.31 (1H, dd, *J* = 15.3, 6.4, $NCHC\mathbf{H}{=}CH$), 5.46 (1H, dt, *J* = 14.1, 6.8, $NCHCH{=}C\mathbf{H}$); ^{13}C NMR (126 MHz, $CDCl_3$) δ 14.1, 22.6, 23.0, 28.5, 28.8, 29.4, 31.7, 32.1, 32.3, 46.1, 58.5, 78.9, 130.4, 130.4, 154.6; HRMS (ESI-TOF) *m*/*z* calcd. for $C_{17}H_{31}NO_2Na$ ($[M+Na]^+$) 304.2247, found 304.2249.



***tert*-Butyl (*E*)-methyl(2-methyldodec-5-en-4-yl)carbamate (30)**

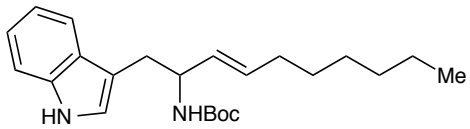
Prepared following the general procedure outlined above using (*E*)-1-iodooct-1-ene (87 μL, 0.50 mmol, 1.0 equiv.), *N*-Me-Boc-Leu-OH (184 mg, 0.75 mmol, 1.50 equiv.), $Ir[dF(CF_3)ppy]_2(dtbbpy)PF_6$ (5.6 mg, 5.0 μmol, 0.01 equiv.), $NiCl_2$·glyme (11 mg, 50 μmol, 0.10 equiv.), dtbbpy (20 mg, 75 μmol, 0.15 equiv.), Cs_2CO_3 (277 mg, 0.850 mmol, 1.70 equiv.), and DMF (25.0 mL). After 38 h, the reaction mixture was subjected to the workup protocol outlined in the general procedure. Purification by flash column chromatography (0–20% EtOAc/hexanes) provided the title compound (139 mg, 0.45 mmol, 89%) as a colorless oil. IR (film) ν_{max} 2957-2856, 1692, 1468, 1455, 1390, 1365, 1321, 1253, 1143 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 0.87–0.93 (9H, m, 3 × $C\mathbf{H_3}$), 1.26–1.37 (9H, m, 4 × $C\mathbf{H_2}$ + $C\mathbf{H}(CH_3)_2$), 1.47 (9H, s, $OC(C\mathbf{H_3})_3$), 1.46–1.50 (2H, m, $C\mathbf{H}_2$), 2.01 (2H, q, *J* = 7.0, $CH{=}CHC\mathbf{H_2}$), 2.64 (3H, s, $NC\mathbf{H_3}$), 4.55–4.72 (1H,

br m, NC**H**CH=CH), 5.34 (1H, dd, *J* = 15.5, 5.6, NCHC**H**=CH), 5.49–5.53 (1H, br m, NCHCH=C**H**); ^{13}C NMR (126 MHz, $CDCl_3$) δ 14.1, 22.1, 22.6, 23.2, 24.6, 28.5, 28.8, 29.3, 31.7, 32.5, 40.8, 54.5, 79.1, 129.1, 132.2, 156.0; HRMS (ESI-TOF) *m/z* calcd. for $C_{19}H_{37}NO_2Na$ ($[M+Na]^+$) 334.2714, found 334.2717.



(*E*)-tert-butyl (1-phenyldec-3-en-2-yl)carbamate (31)

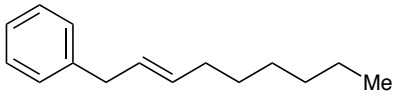
Prepared following the general procedure outlined above using (*E*)-1-bromooct-1-ene (87 μL, 0.50 mmol, 1.0 equiv.), *N*-Boc-Phe-OH (212 mg, 0.8 mmol, 1.60 equiv.), $Ir[dF(Me)ppy]_2(dtbbpy)PF_6$ (5.1 mg, 5.0 μmol, 0.01 equiv.), $NiCl_2$·glyme (1.1 mg, 5 μmol, 0.01 equiv.), dtbbpy (1.3 mg, 5 μmol, 0.01 equiv.), Cs_2CO_3 (261 mg, 0.80 mmol, 1.60 equiv.), and DMA (5.0 mL). After 4 h, the reaction mixture was subjected to the workup protocol outlined in the general procedure. Purification by flash column chromatography (5% Et_2O/pentanes) provided the title compound (159 mg, 0.48 mmol, 96%) as a colorless oil. IR (film) ν_{max} 2958, 2927, 2856, 1697, 1658, 1494, 1366, 1248, 1166, 699 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.89 (3H, t, *J* = 7.0, C**H$_3$**), 1.20–1.34 (8H, m, (C**H$_2$**)$_4$CH$_3$), 1.41 (9H, s, C(C**H$_3$**)$_3$), 1.98 (2H, q, *J* = 7.0, CH=CHC**H$_2$**), 2.79–2.88 (2H, m, C**H$_2$**Ph), 4.35 (1H, br s, C**H**NH), 4.44 (1H, br s, CHN**H**), 5.32–5.39 (1H, m, NHCHC**H**=CH), 5.45–5.53 (1H, m, NHCHCH=C**H**), 7.16–7.24 (3H, m, Ar**H**), 7.26–7.30 (2H, m, Ar**H**); ^{13}C NMR (126 MHz, $CDCl_3$) δ 14.1, 22.6, 28.4, 28.7, 29.1, 31.7, 32.2, 42.0, 53.1, 125.1, 126.3, 128.2, 128.6, 129.6, 131.7, 137.8, 155.1; HRMS (ESI-TOF) *m/z* calcd. for $C_{21}H_{34}NO_2$ ($[M+H]^+$) 332.2584, found 332.2578.



(*E*)-tert-butyl (1-(1*H*-indol-3-yl)dec-3-en-2-yl)carbamate (32)

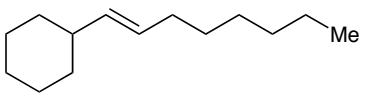
Prepared following the general procedure outlined above using (*E*)-1-bromooct-1-ene (87 μL, 0.50 mmol, 1.0 equiv.), *N*-Boc-Trp-OH (243 mg, 0.80 mmol, 1.60 equiv.), $Ir[dF(Me)ppy]_2(dtbbpy)PF_6$ (5.1 mg, 5.0 μmol, 0.01 equiv.), $NiCl_2$·glyme (1.1 mg, 5 μmol, 0.01 equiv.), dtbbpy (1.3 mg, 5 μmol, 0.01 equiv.), Cs_2CO_3 (261 mg, 0.80 mmol, 1.60 equiv.), and

DMA (5.0 mL). After 4 h, the reaction mixture was subjected to the workup protocol outlined in the general procedure. Purification by flash column chromatography (20% Et_2O/pentanes) provided the title compound (146 mg, 0.40 mmol, 79%) as a colorless oil. IR (film) ν_{max} 3330, 2957, 2925, 2854, 1691, 1497, 1366, 1247, 1167, 1011, 968, 740 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 0.89 (3H, t, *J* = 7.0, C**H**$_3$), 1.22–1.31 (8H, m, (C**H**$_2$)$_4$CH$_3$), 1.42 (9H, s, C(C**H**$_3$)$_3$), 1.97 (2H, q, *J* = 6.9, CH=CHC**H**$_2$), 2.99 (2H, d, *J* = 6.1, C**H**$_2$CHNH), 4.46 (1H, br s, CH$_2$C**H**NH), 4.56 (1H, br s, CH$_2$CHN**H**), 5.43 (1H, dd, *J* = 15.5, 5.7, NHCHC**H**=CH), 5.55 (1H, dt, *J* = 15.5, 6.9, NHCHCH=C**H**), 7.02 (1H, d, *J* = 2.4, C=C**H**NH), 7.13 (1H, app. t, *J* = 7.5, Ar**H**), 7.20 (1H, app. t, *J* = 7.5, Ar**H**), 7.36 (1H, d, *J* = 8.0, Ar**H**), 7.63 (1H, d, *J* = 8.0, Ar**H**), 8.08 (1H, br s, ArN**H**).; ^{13}C NMR (126 MHz, $CDCl_3$) δ 14.1, 22.6, 28.4, 28.8, 29.1, 31.4, 31.7, 32.2, 52.4, 79.1, 111.0, 111.9, 119.2, 119.3, 121.9, 122.7, 128.0, 130.3, 131.2, 136.1, 155.3; HRMS (ESI-TOF) *m/z* calcd. for $C_{23}H_{35}N_2O_2$ ($[M+H]^+$) 371.2693, found 371.2692.



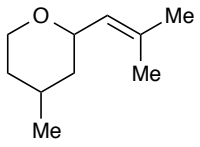
(*E*)-non-2-en-1-ylbenzene (33)

Prepared following the general procedure outlined above using (*E*)-1-iodooct-1-ene (87 μL, 0.50 mmol, 1.0 equiv.), phenylacetic acid (102 mg, 0.75 mmol, 1.50 equiv.), Ir[dF(CF_3)ppy]$_2$(dtbbpy)PF_6 (5.6 mg, 5.0 μmol, 0.01 equiv.), $NiCl_2$·glyme (11 mg, 50 μmol, 0.10 equiv.), dtbbpy (20 mg, 75 μmol, 0.15 equiv.), Cs_2CO_3 (277 mg, 0.850 mmol, 1.70 equiv.), and DMF (25.0 mL). After 72 h, the reaction mixture was subjected to the workup protocol outlined in the general procedure. Purification by flash column chromatography (pentane) provided the title compound (85 mg, 0.42 mmol, 84%) as a colorless oil. IR (film) ν_{max} 2924, 2854, 1494, 1454, 956, 737, 696 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 0.90 (3H, t, *J* = 6.7, (CH$_2$)$_4$C**H**$_3$), 1.26–1.44 (8H, m, (C**H**$_2$)$_4$CH$_3$), 2.04 (2H, q, *J* = 6.9, PhCH$_2$CH=CHC**H**$_2$), 3.35 (2H, d, *J* = 6.0, PhC**H**$_2$), 5.55 (2H, m, C**H**=C**H**), 7.19–7.22 (3H, m, Ar**H**), 7.31 (2H, t, *J* = 7.5, Ar**H**); ^{13}C NMR (126 MHz, $CDCl_3$) δ 14.1, 22.7, 28.9, 29.5, 31.8, 32.5, 39.1, 125.9, 128.3, 128.5, 128.7, 132.2, 141.2; HRMS (ESI-TOF) *m/z* calcd. for $C_{15}H_{22}$ ($[M•]^+$) 201.1638, found 201.1625.



(*E*)-oct-1-en-1-ylcyclohexane (34)

Prepared following the general procedure outlined above using (*E*)-1-iodooct-1-ene (87 μL, 0.50 mmol, 1.0 equiv.), cyclohexanecarboxylic acid (192 mg, 1.50 mmol, 3.0 equiv.), $Ir[dF(CF_3)ppy]_2(dtbbpy)PF_6$ (5.6 mg, 5.0 μmol, 0.01 equiv.), $NiCl_2$·glyme (5.5 mg, 25 μmol, 0.05 equiv.), dtbbpy (6.7 mg, 25 μmol, 0.05 equiv.), Cs_2CO_3 (489 mg, 1.5 mmol, 3.0 equiv.), and DMSO (20.0 mL). After 8 h, the reaction mixture was subjected to the workup protocol outlined in the general procedure. Purification by flash column chromatography (pentane) provided the title compound (76 mg, 0.39 mmol, 78%) as a colorless oil. IR (film) ν_{max} 2956, 2921, 2852, 1448, 966 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 0.89 (3H, t, *J* = 6.8, $(CH_2)_5CH_3$), 1.04–1.16 (3H, m, CH_2), 1.21–1.36 (11H, m, CH_2), 1.59–1.75 (4H, m, CH_2), 1.84–1.93 (1H, m, CHCH=CH), 1.94–2.00 (2H, m, CH=CHCH_2), 5.31–5.40 (2H, m, CHCH=CH + CHCH=CH); ^{13}C NMR (126 MHz, $CDCl_3$) δ 14.1, 22.6, 26.1, 26.2, 26.9, 28.8, 29.7, 30.2, 31.8, 32.7, 33.3, 40.7, 127.7, 136.4; HRMS (ESI-TOF) *m*/*z* calcd. for $C_{14}H_{27}$ ($[M+H]^+$) 195.2107, found 195.2108.

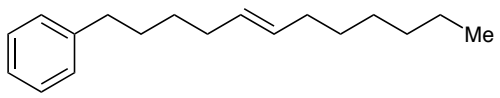


***trans*-rose oxide**

Prepared following the general procedure outlined above using 1-bromo-2-methylprop-1-ene (52 μL, 0.50 mmol, 1.0 equiv.), 4-methyltetrahydro-2*H*-pyran-2-carboxylic acid (144 mg, 1.0 mmol, 2.0 equiv.), $Ir[dF(Me)ppy]_2(dtbbpy)PF_6$ (5.1 mg, 5.0 μmol, 0.010 equiv.), $NiCl_2$·glyme (5.5 mg, 25 μmol, 0.05 equiv.), dtbbpy (6.7 mg, 25 μmol, 0.05 equiv.), Cs_2CO_3 (326 mg, 1.0 mmol, 2.0 equiv.), and DMSO (20.0 mL). After 18 h, the reaction mixture was subjected to the workup protocol outlined in the general procedure. Purification by flash column chromatography (5% Et_2O/pentane) provided the title compound (61 mg, 0.40 mmol, 79%, 5:1 d.r.). The inseparable diastereomers were characterized as a mixture. IR (film) ν_{max} 2954, 2919, 2849, 1669, 1453, 1377, 1184, 1078, 1053, 881, 833, 733 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 0.83–0.99 (0.8H, m), 1.07 (3H, d, *J* = 7.0), 1.19–1.30 (1.2 H, m), 1.34–1.41 (1H, m), 1.50–1.64 (2.4H, m), 1.68–1.81 (7.2H, m), 1.97–2.07 (1H, m), 3.39–3.51 (0.4H, m), 3.67–3.79 (2H, m), 3.95–4.07 (0.4H, m), 4.37 (1H, td, *J* = 8.2, 3.3), 5.17 (0.2 H, dt, *J* = 8.3, 1.6), 5.29 (1H, dt, *J* = 7.9, 1.5); ^{13}C NMR (126 MHz, $CDCl_3$) δ 18.3, 19.1, 22.3, 24.9, 25.7, 25.8, 30.3, 32.4, 34.4, 38.2, 40.8, 62.2, 67.9, 68.4,

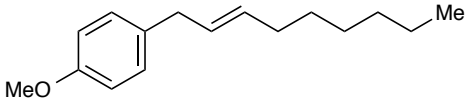
69.1, 74.6, 80.1, 125.4, 126.3, 135.6; HRMS (ESI-TOF) *m/z* calcd. for $C_{10}H_{19}O$ ($[M+H]^+$) 155.1430, found 155.1432.

7) Experimental Data for Additional Examples



(*E*)-dodec-5-en-1-ylbenzene (S1)

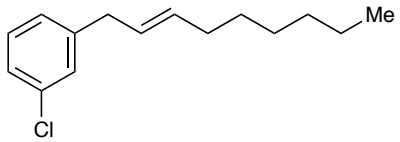
Prepared following the general procedure outlined above using (*E*)-1-iodooct-1-ene (87 μL, 0.50 mmol, 1.0 equiv.), 5-phenylvaleric acid (267 mg, 1.5 mmol, 3.0 equiv.), $Ir[dF(CF_3)ppy]_2(dtbbpy)PF_6$ (5.6 mg, 5.0 μmol, 0.010 equiv.), $NiCl_2$·glyme (5.5 mg, 25 μmol, 0.05 equiv.), dtbbpy (6.7 mg, 25 μmol, 0.05 equiv.), Cs_2CO_3 (489 mg, 1.5 mmol, 3.0 equiv.), and DMSO (20.0 mL). After 24 h, the reaction mixture was subjected to the workup protocol outlined in the general procedure. Purification by flash column chromatography (pentane) provided the title compound (14 mg, 0.06 mmol, 11%) as a colorless oil. IR (film) ν_{max} 2926, 2854, 1454 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 0.89 (3H, t, *J* = 6.7, $(CH_2)_5CH_3$), 1.22–1.37 (8H, m, CH_2), 1.36–1.45 (2H, m, CH_2), 1.59–1.67 (2H, m, CH_2), 1.93–2.05 (4H, m, $CH_2CH{=}CHCH_2$), 2.61 (2H, t, *J* = 7.8, CH_2Ph), 5.39 (2H, m, *J* = 4.7, CH=CH), 7.17–7.30 (5H, m, ArH); ^{13}C NMR (126 MHz, $CDCl_3$) δ 14.1, 22.7, 28.9, 29.3, 29.6, 31.0, 31.8, 32.5, 32.6, 35.9, 125.6, 128.2, 128.4, 130.0, 130.7, 142.9; HRMS (ESI-TOF) *m/z* calcd. for $C_{18}H_{29}$ ($[M+H]^+$) 245.2264, found 245.2256.



(*E*)-1-methoxy-4-(non-2-en-1-yl)benzene (S2)

Prepared following the general procedure outlined above using (*E*)-1-iodooct-1-ene (87 μL, 0.50 mmol, 1.0 equiv.), 2-(4-methoxyphenyl)acetic acid (125 mg, 0.75 mmol, 1.50 equiv.), $Ir[dF(CF_3)ppy]_2(dtbbpy)PF_6$ (5.6 mg, 5.0 μmol, 0.010 equiv.), $NiCl_2$·glyme (11 mg, 50 μmol, 0.10 equiv.), dtbbpy (20 mg, 75 μmol, 0.15 equiv.), Cs_2CO_3 (277 mg, 0.85 mmol, 1.70 equiv.), and DMF (25.0 mL). After 72 h, the reaction mixture was subjected to the workup protocol

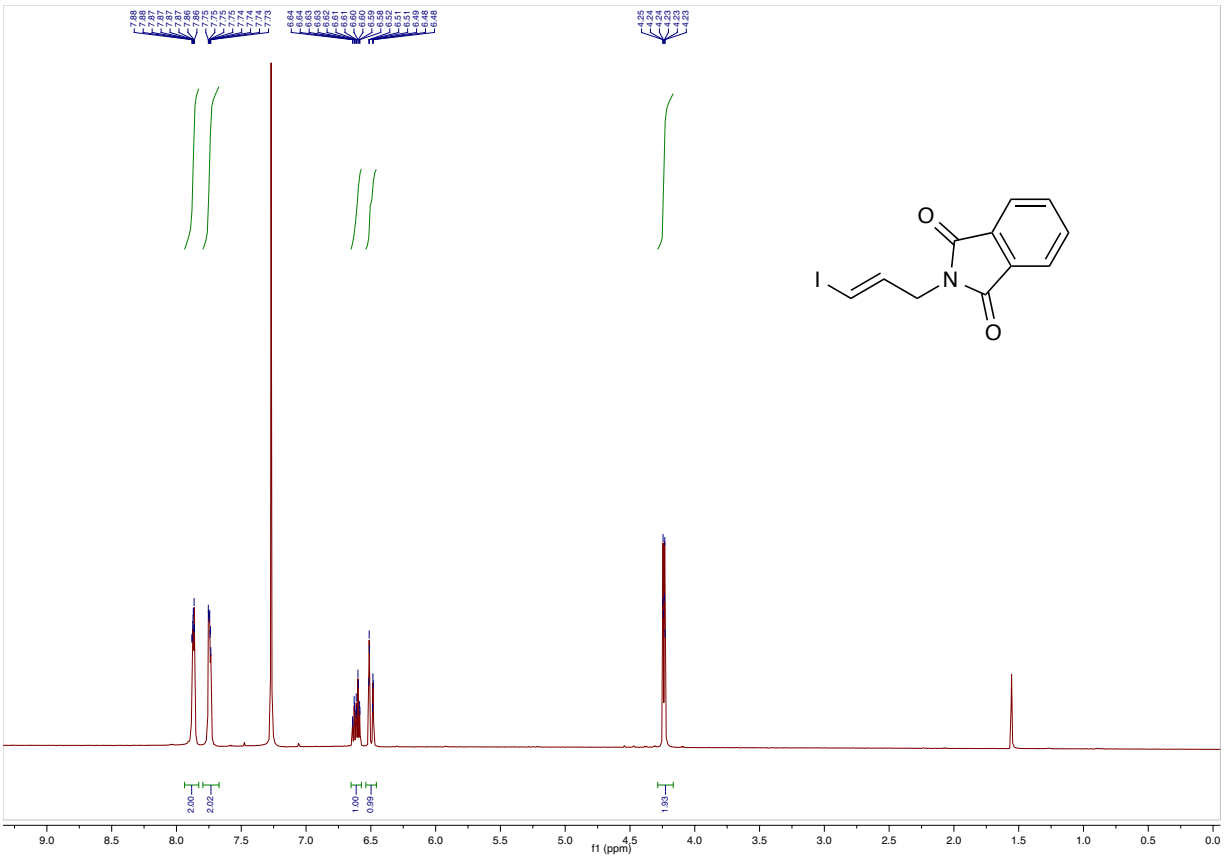
outlined in the general procedure. Purification by flash column chromatography (0–5% Et_2O/pentane) provided the title compound (112 mg, 0.482 mmol, 96%) as a colorless oil. IR (film) ν_{max} 2955, 2924, 2854, 1510, 1243, 1175, 1038, 966, 818 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 0.89 (3H, t, *J* = 6.7, C**H**$_3$), 1.21–1.43 (8H, m, (C**H**$_2$)$_4$CH$_3$), 2.02 (2H, q, *J* = 6.8, CH=CHC**H**$_2$), 3.28 (2H, d, *J* = 6.0, C**H**$_2$Ph), 3.80 (3H, s, OC**H**$_3$), 5.43–5.60 (2H, m, C**H**=C**H**), 6.82–6.86 (2H, m, Ar**H**), 7.07–7.15 (2H, m, Ar**H**); ^{13}C NMR (126 MHz, $CDCl_3$) δ 14.1, 22.6, 28.9, 29.5, 31.7, 32.5, 38.1, 55.3, 113.7, 129.1, 129.3, 131.8, 133.2, 157.8; HRMS (ESI-TOF) *m/z* calcd. for $C_{15}H_{21}$ ($[M+H]^+$) 233.1900, found 233.1896.



(*E*)-1-chloro-3-(non-2-en-1-yl)benzene (S3)

Prepared following the general procedure outlined above using (*E*)-1-iodooct-1-ene (87 μL, 0.50 mmol, 1.0 equiv.), 2-(3-chlorophenyl)acetic acid (128 mg, 0.75 mmol, 1.50 equiv.), $Ir[dF(CF_3)ppy]_2(dtbbpy)PF_6$ (5.6 mg, 5.0 μmol, 0.010 equiv.), $NiCl_2$·glyme (11 mg, 50 μmol, 0.10 equiv.), dtbbpy (20 mg, 75 μmol, 0.15 equiv.), Cs_2CO_3 (277 mg, 0.85 mmol, 1.70 equiv.), and DMF (25.0 mL). After 72 h, the reaction mixture was subjected to the workup protocol outlined in the general procedure. Purification by flash column chromatography (0–5% Et_2O/pentane) provided the title compound (80 mg, 0.338 mmol, 68%) as a clear oil. IR (film) ν_{max} 2956, 2924, 2854, 1597, 1431, 1078, 966, 776, 682 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 0.89 (3H, t, *J* = 6.6, C**H**$_3$), 1.21–1.45 (8H, m, (C**H**$_2$)$_4$CH$_3$), 2.01–2.05 (2H, m, CH=CHC**H**$_2$), 3.31 (2H, d, *J* = 4.1, C**H**$_2$Ar), 5.47–5.60 (2H, m, C**H**=C**H**), 7.05–7.24 (4H, m, Ar**H**); ^{13}C NMR (126 MHz, $CDCl_3$) δ 14.1, 22.6, 28.9, 29.3, 31.7, 32.5, 38.7, 126.0, 126.6, 127.7, 128.6, 129.5, 132.9, 134.1, 143.2; HRMS (ESI-TOF) *m/z* calcd. for $C_{15}H_{21}$ ($[M+H]^+$) 237.1405, found 237.1409.

8) Spectral Data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6.49
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6.48
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4.25
4.24
4.24
4.23
4.23
4.23
4.23
O
N
I
O
2.00
2.02
1.00
0.99
1.93
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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4.5
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3.0
2.5
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1.0
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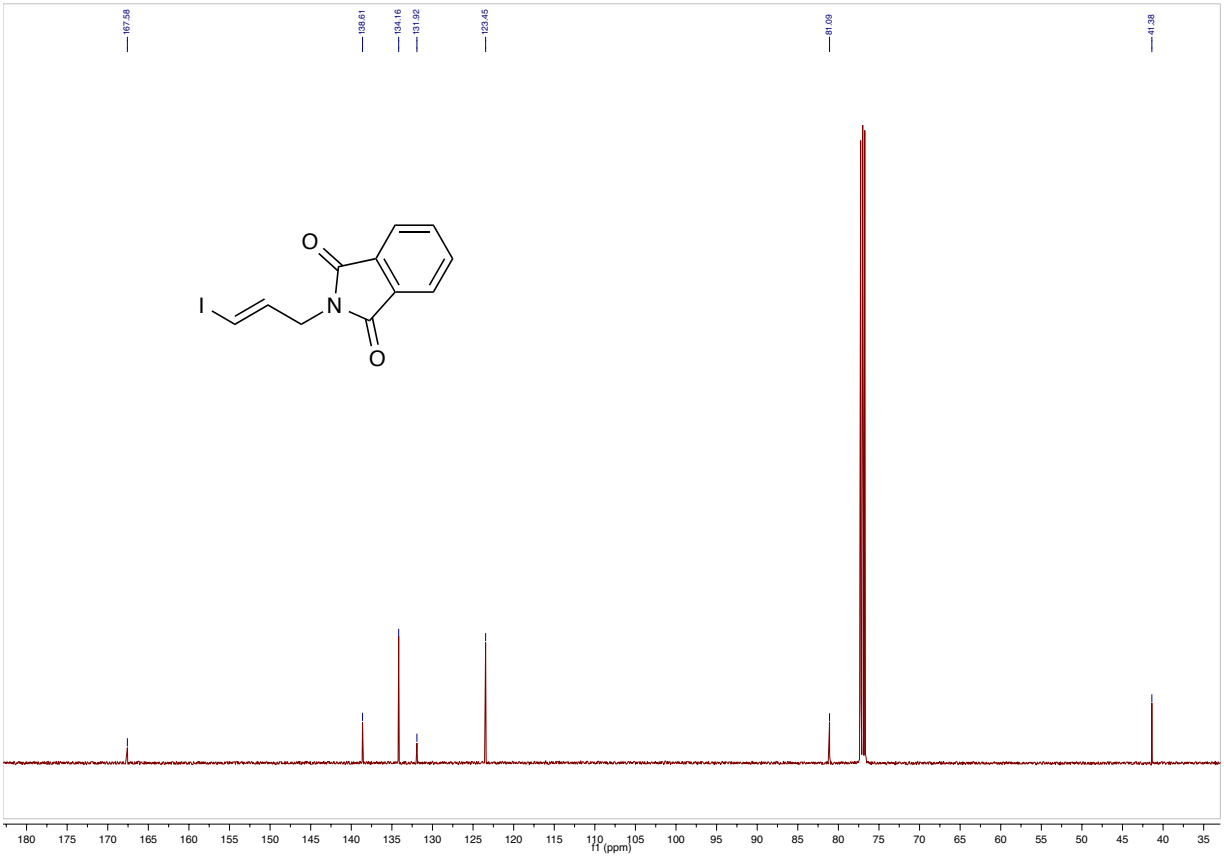
0.0
f1 (ppm)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131.92
123.45
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I
O
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175
170
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150
145
140
135
130
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60
55
50
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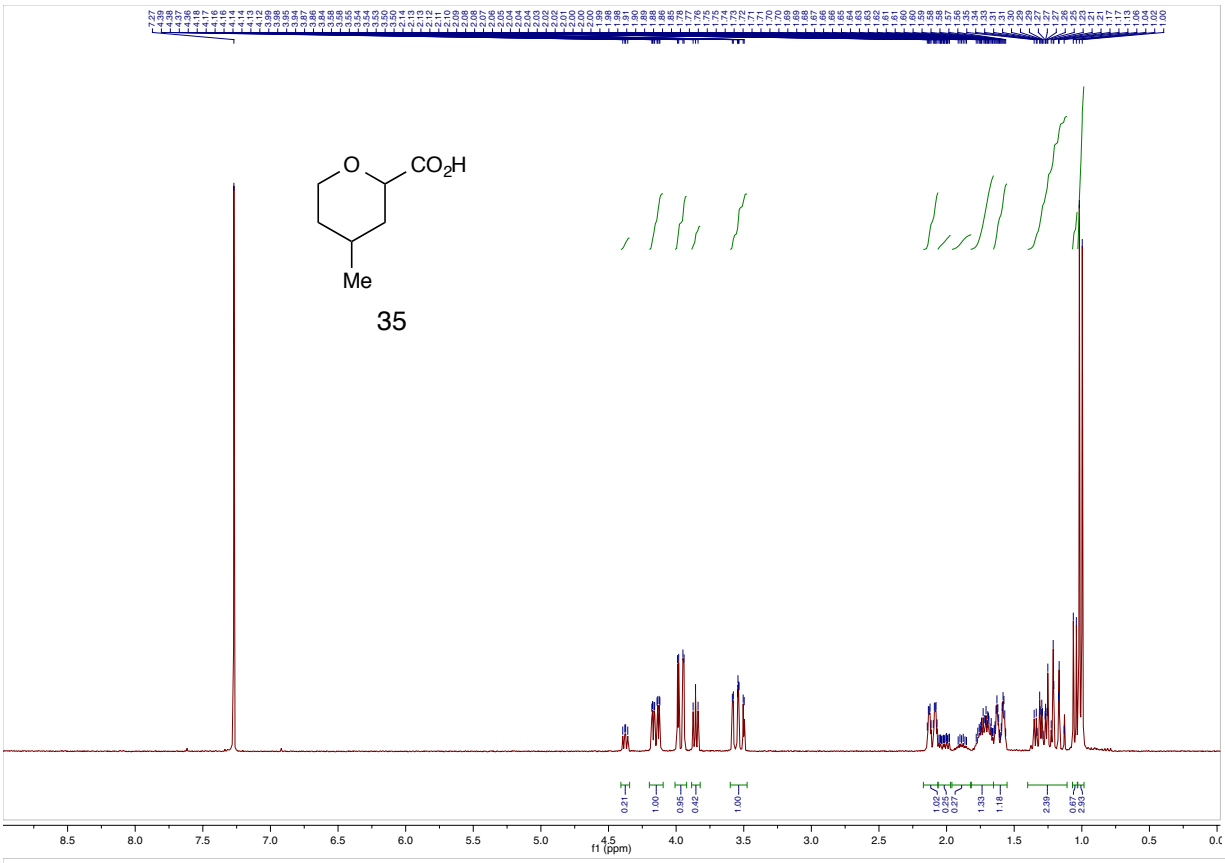
35
f1 (ppm)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CO2H
Me
35
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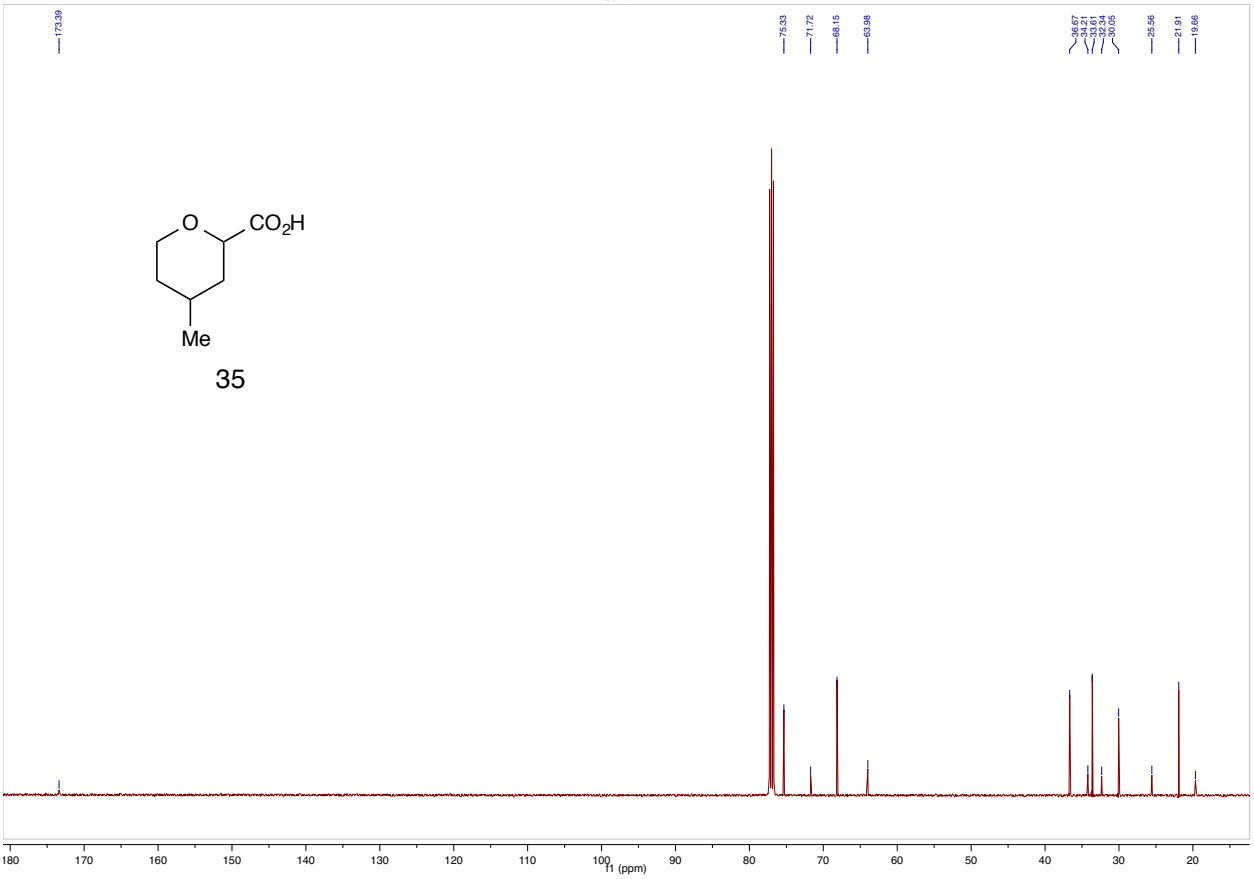
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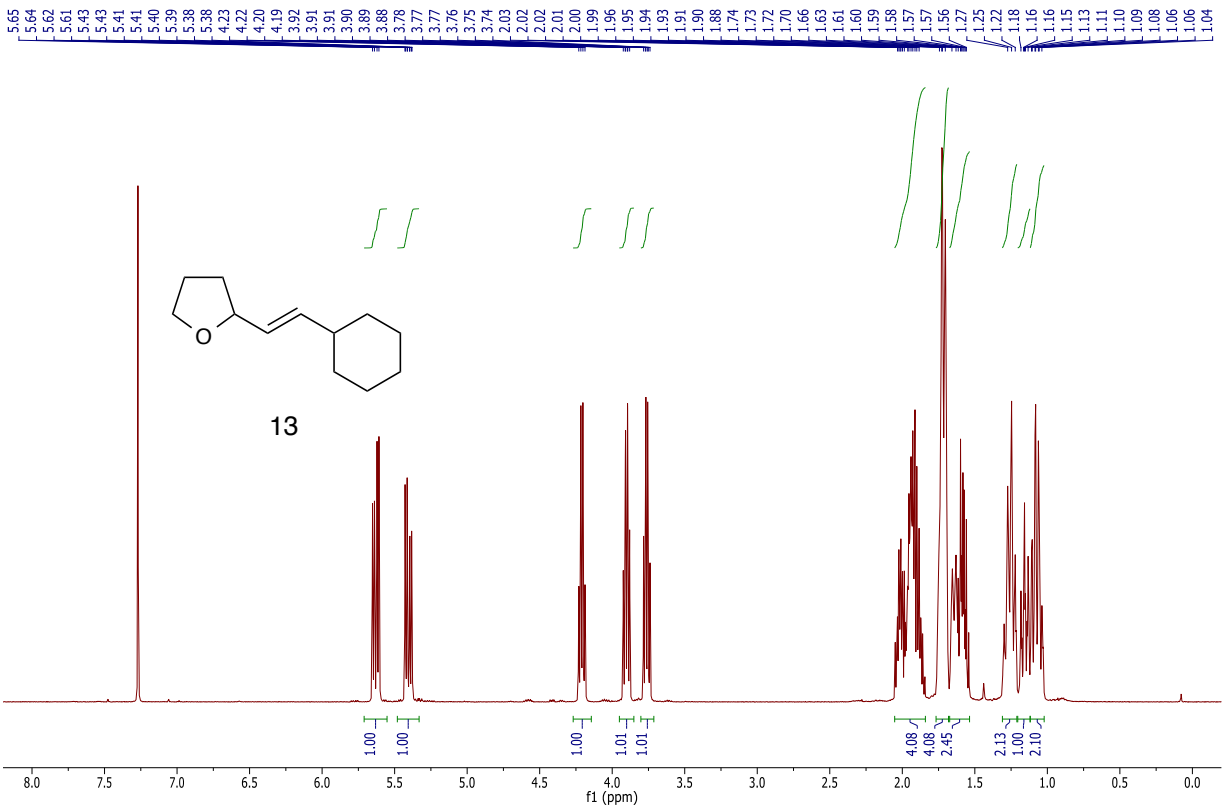
CO2H
Me
35
f1 (ppm)

AN-2-639



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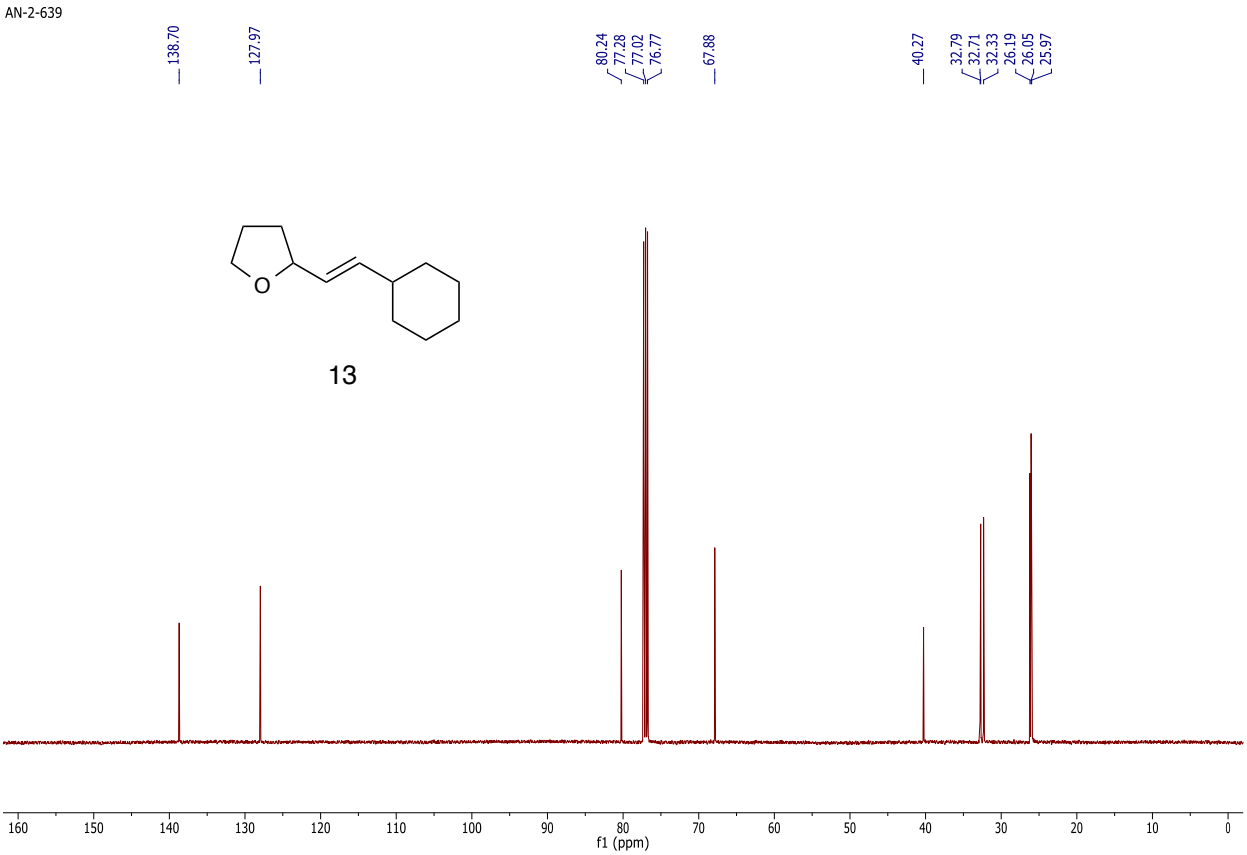

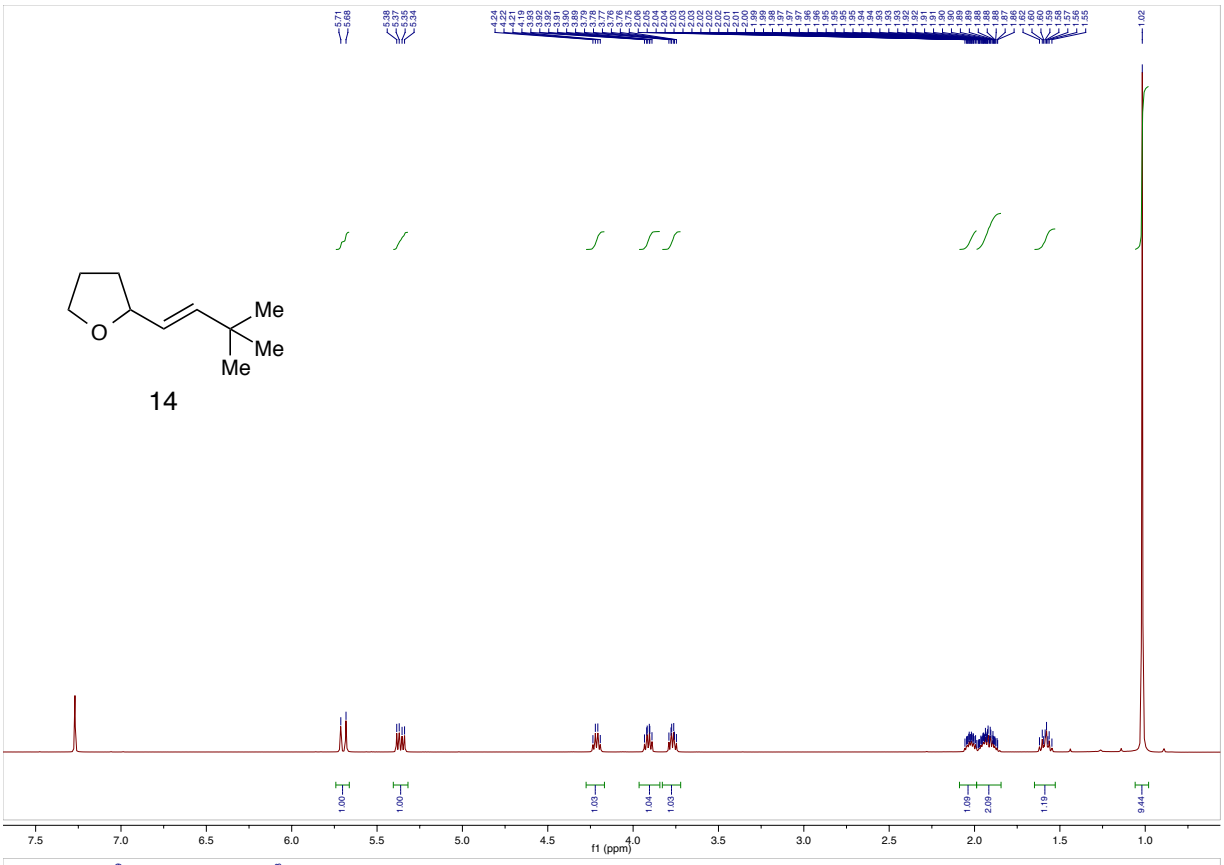

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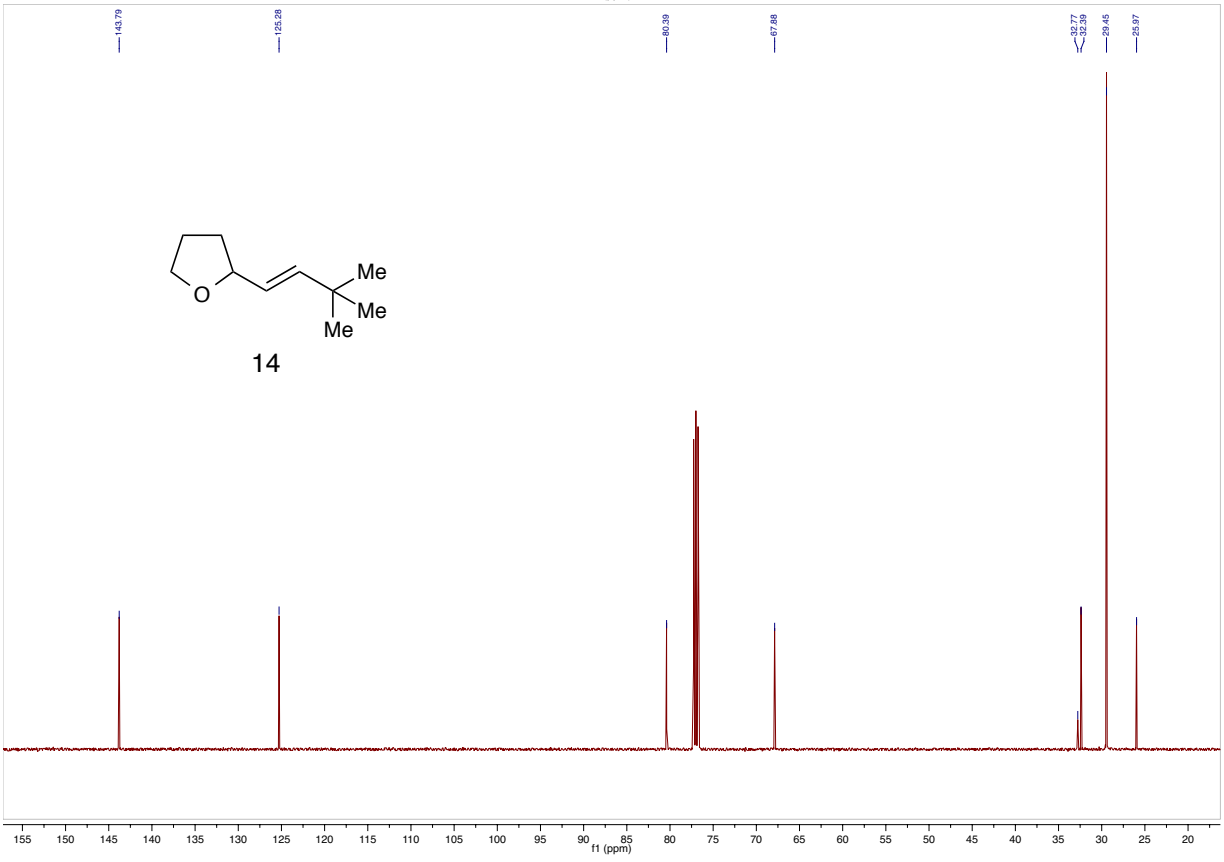

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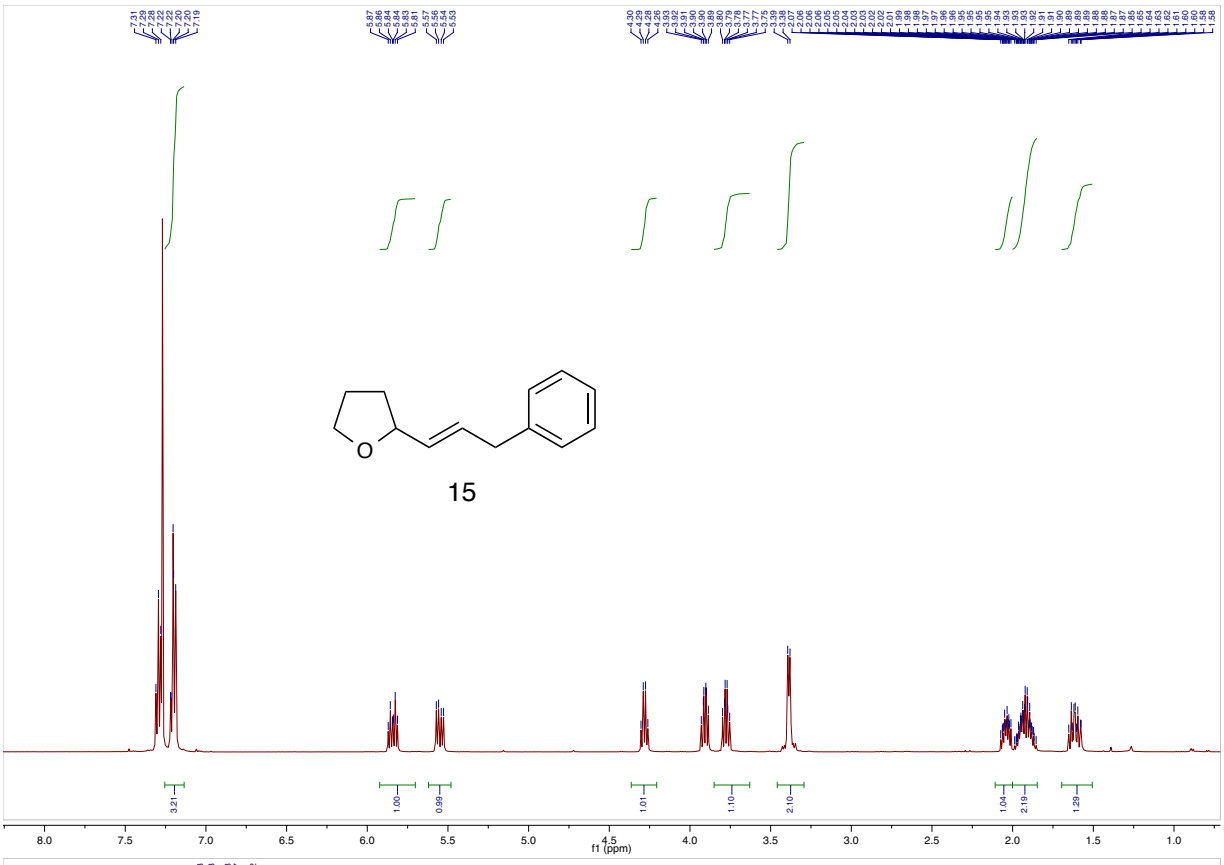
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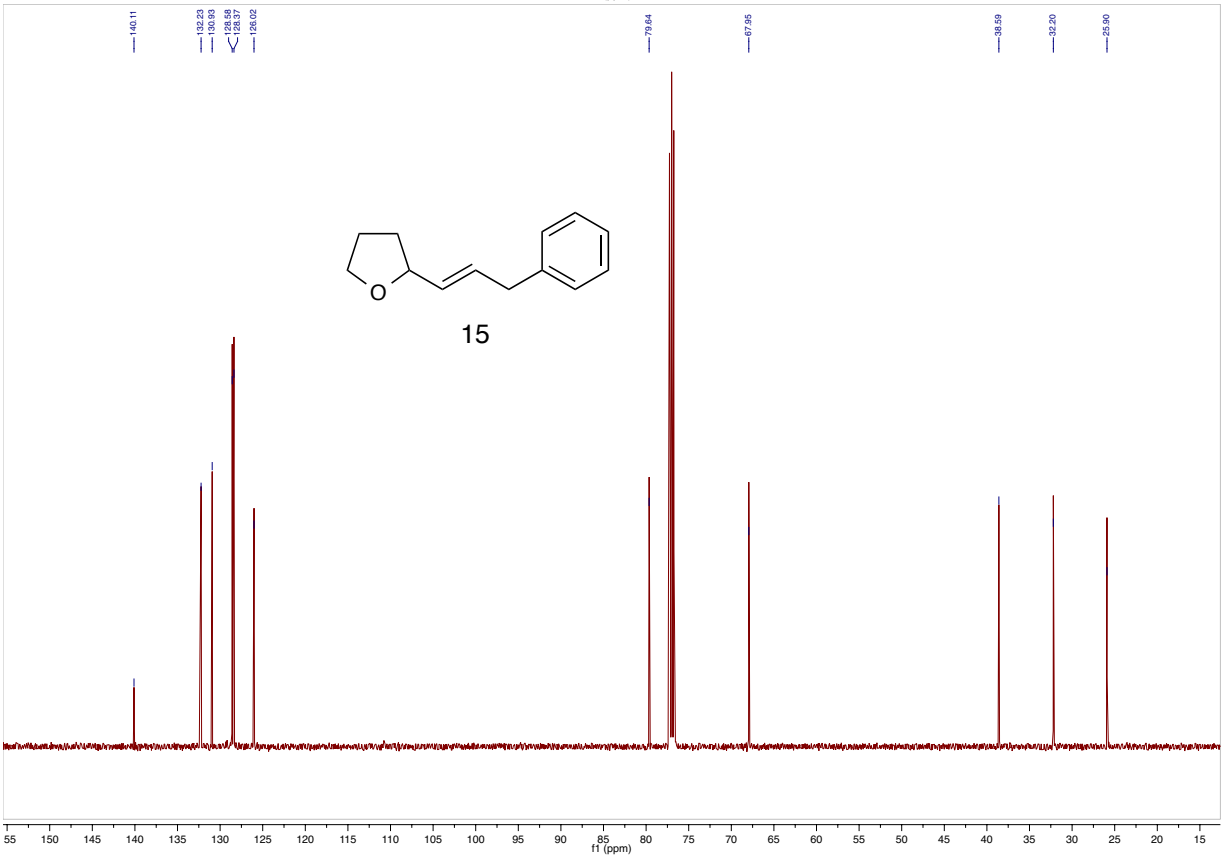
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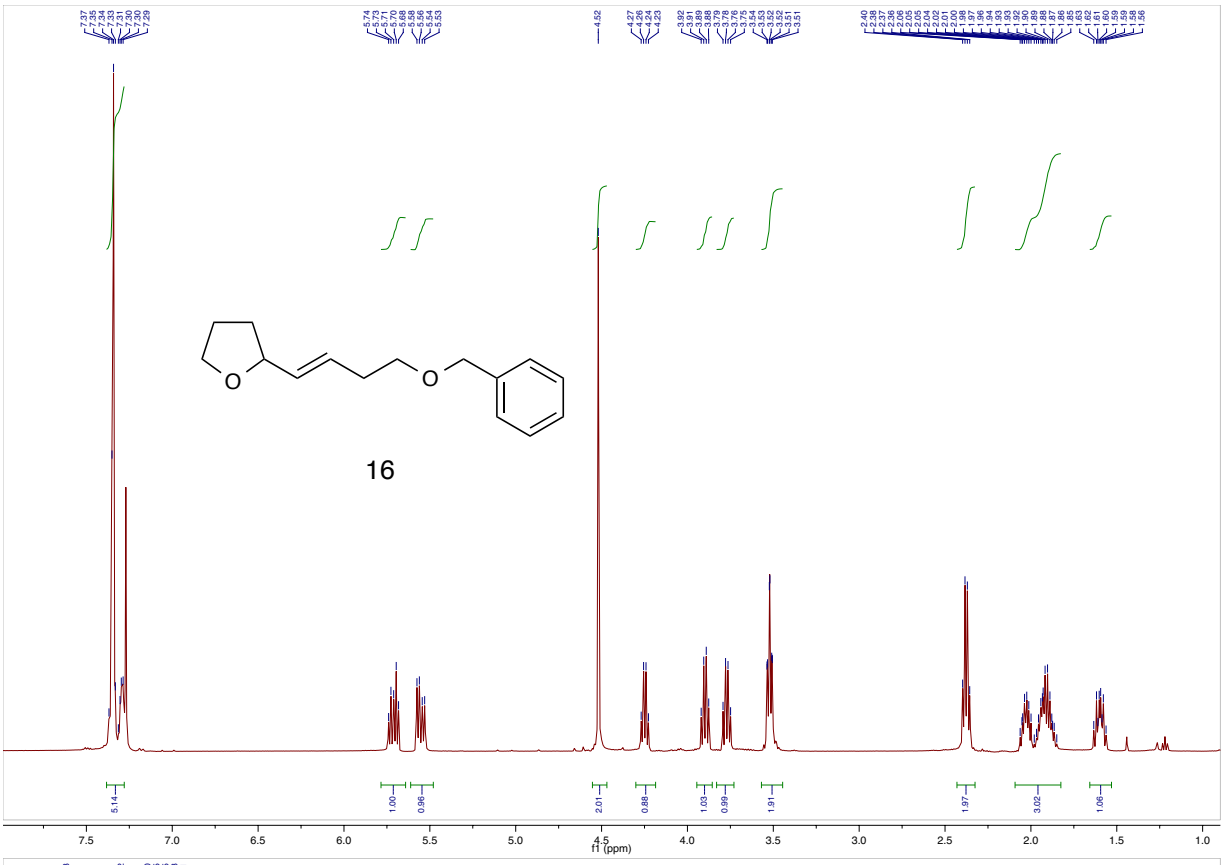
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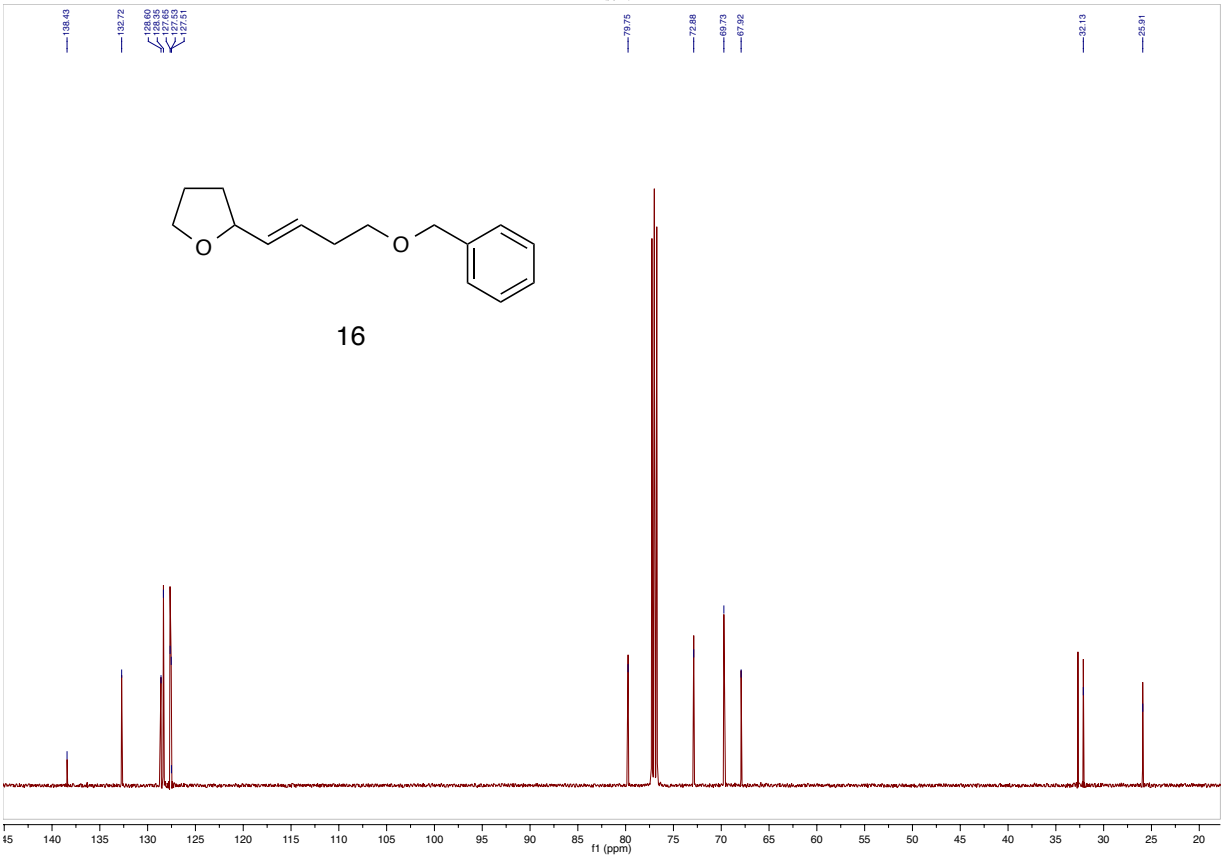
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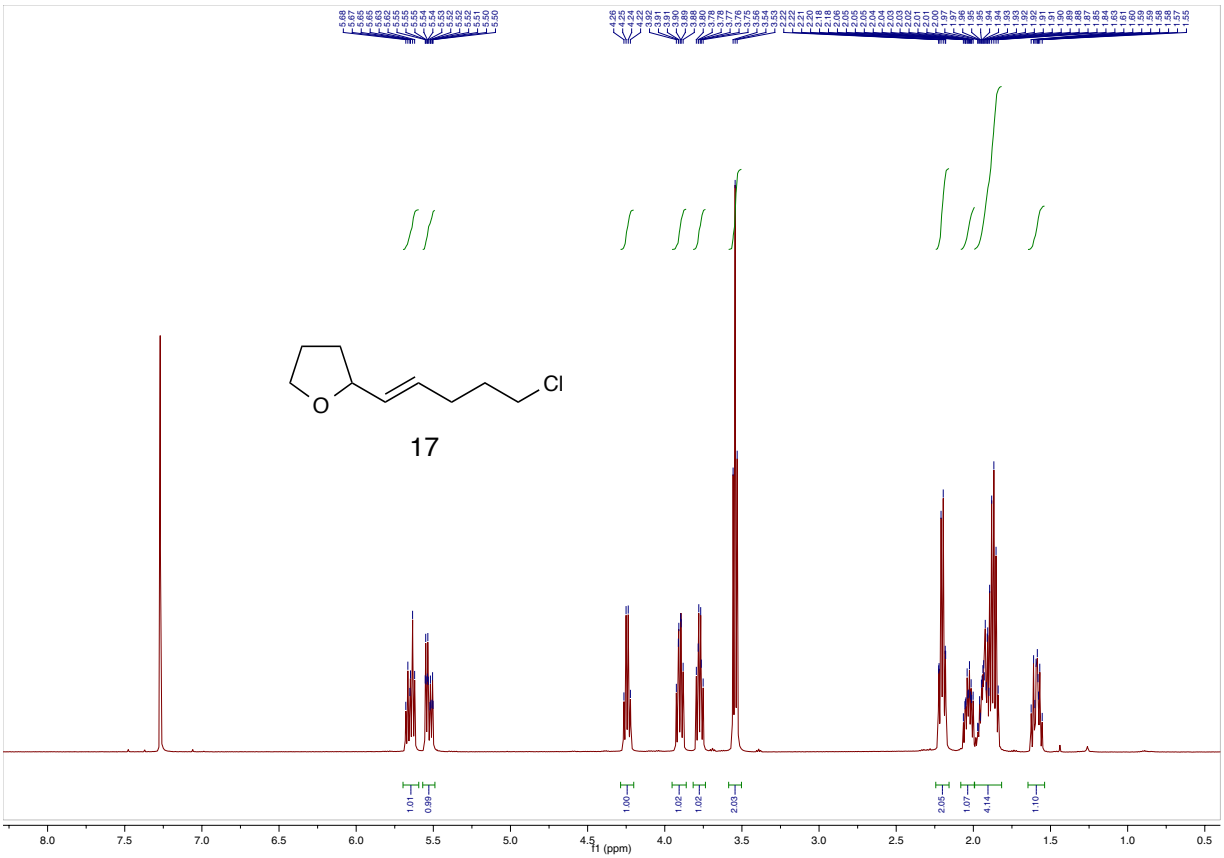
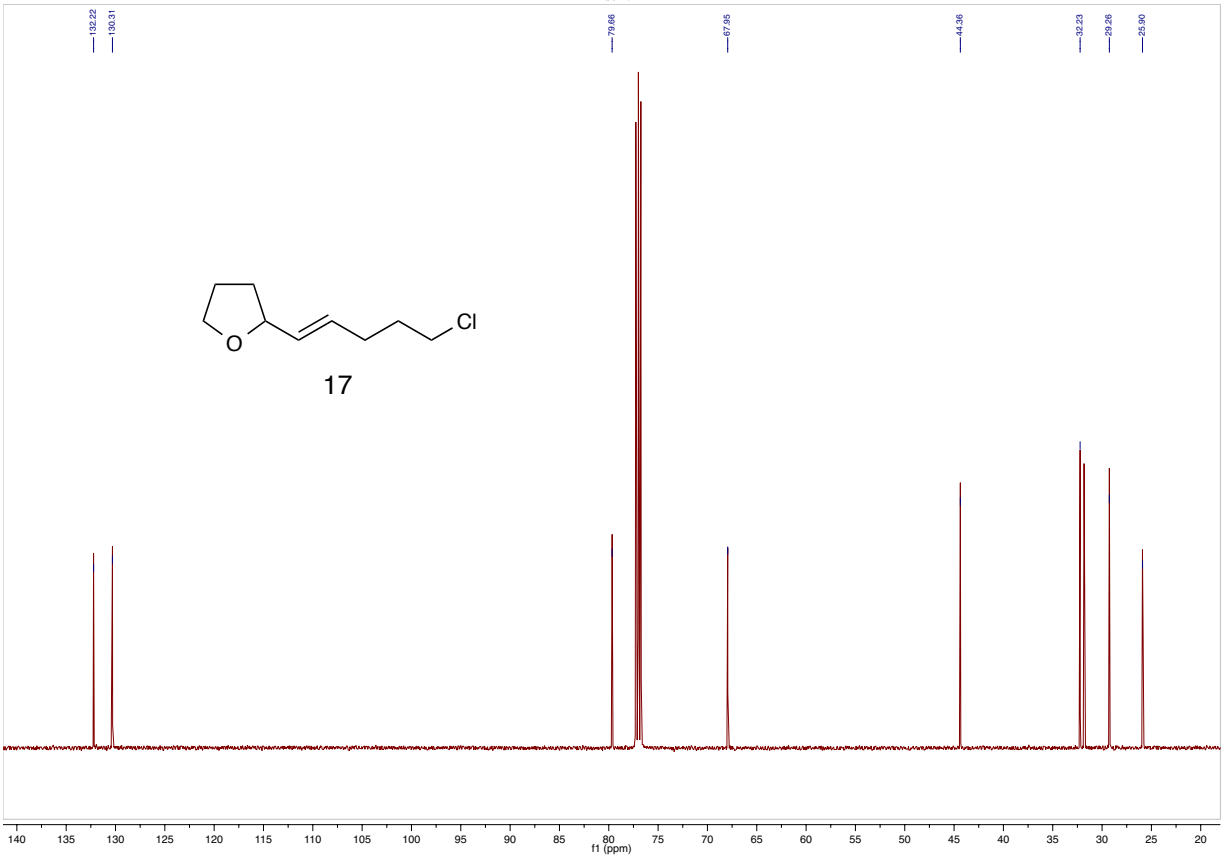
f1 (ppm)


Cl
O
17
f1 (ppm)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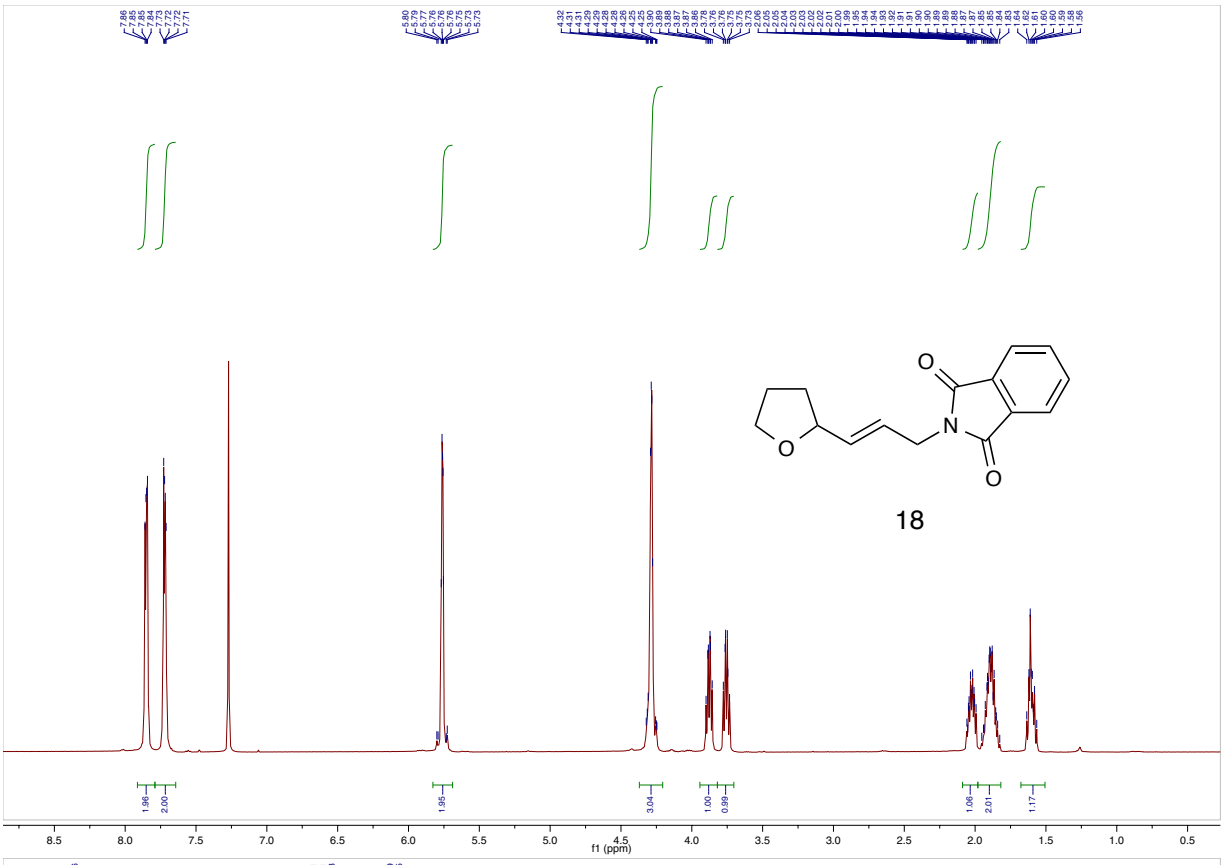
25.90
Cl
O
17
f1 (ppm)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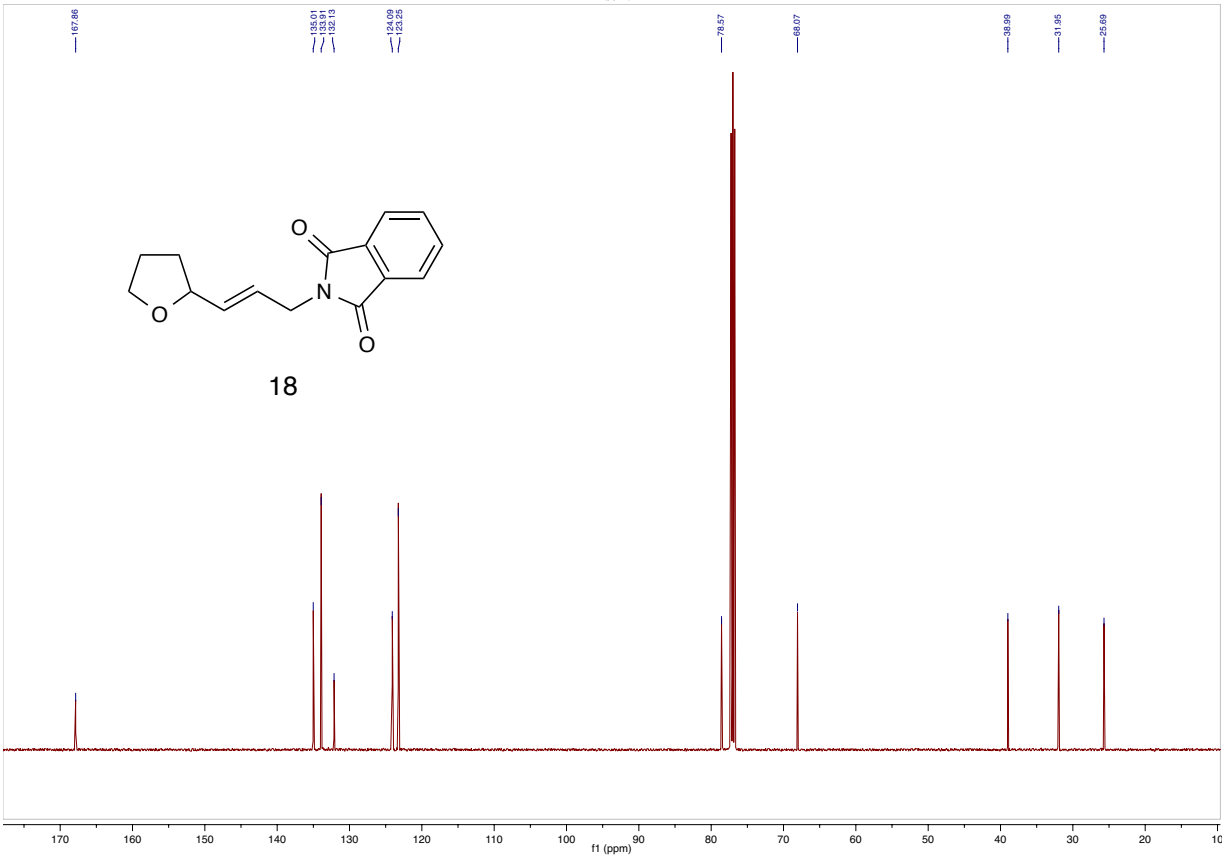
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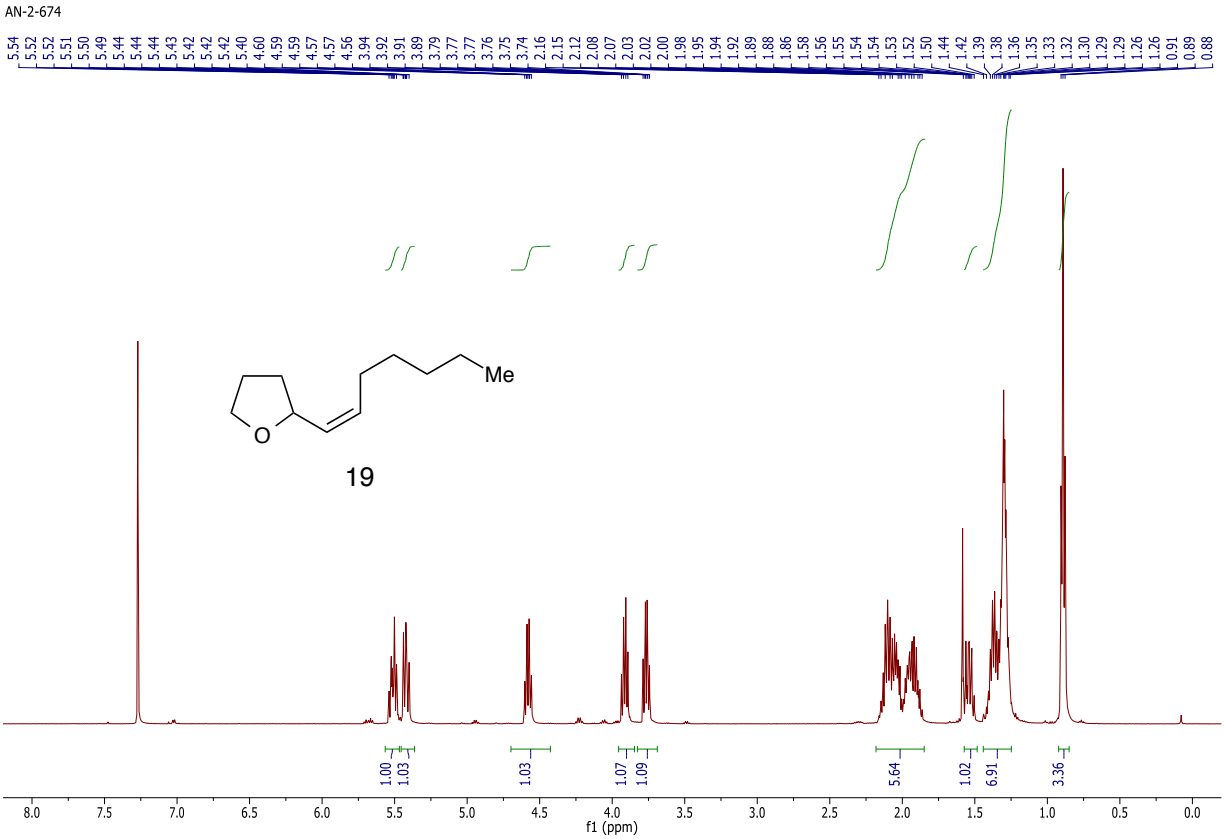
AN-2-674
Me
O
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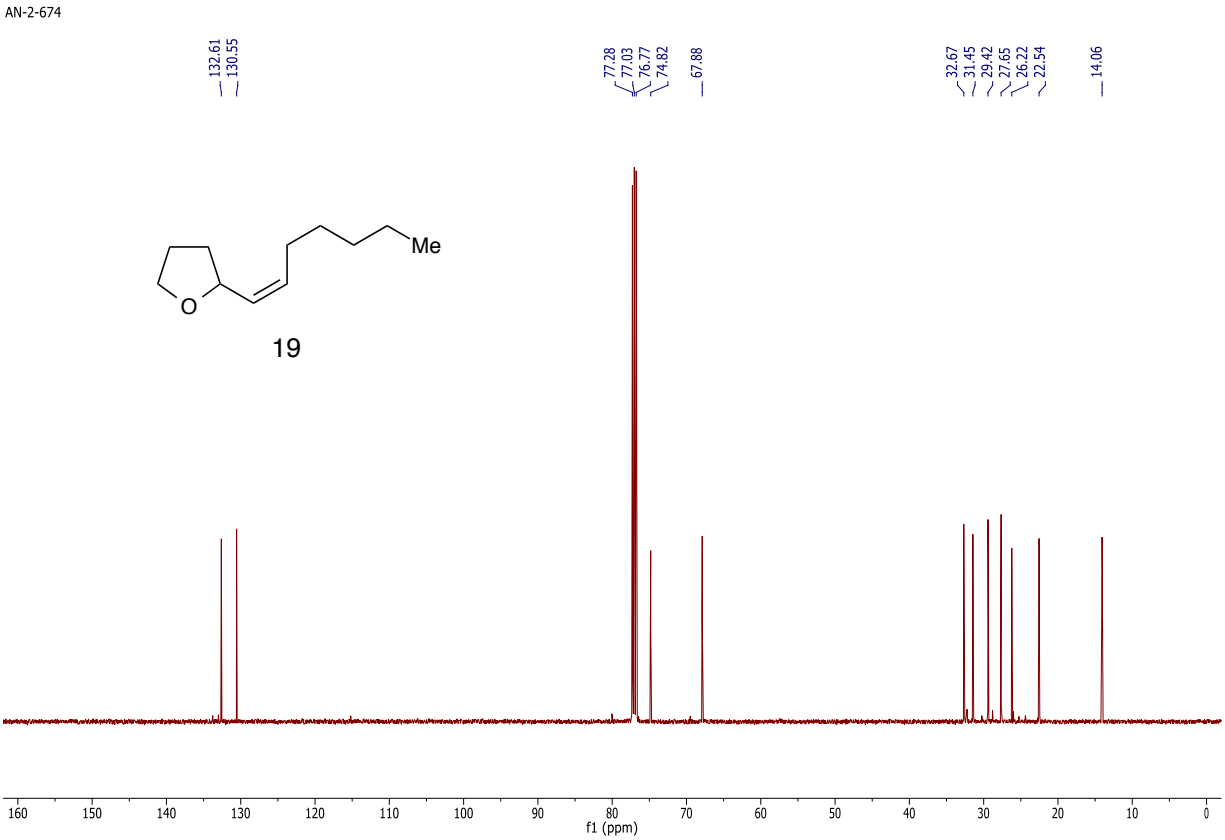
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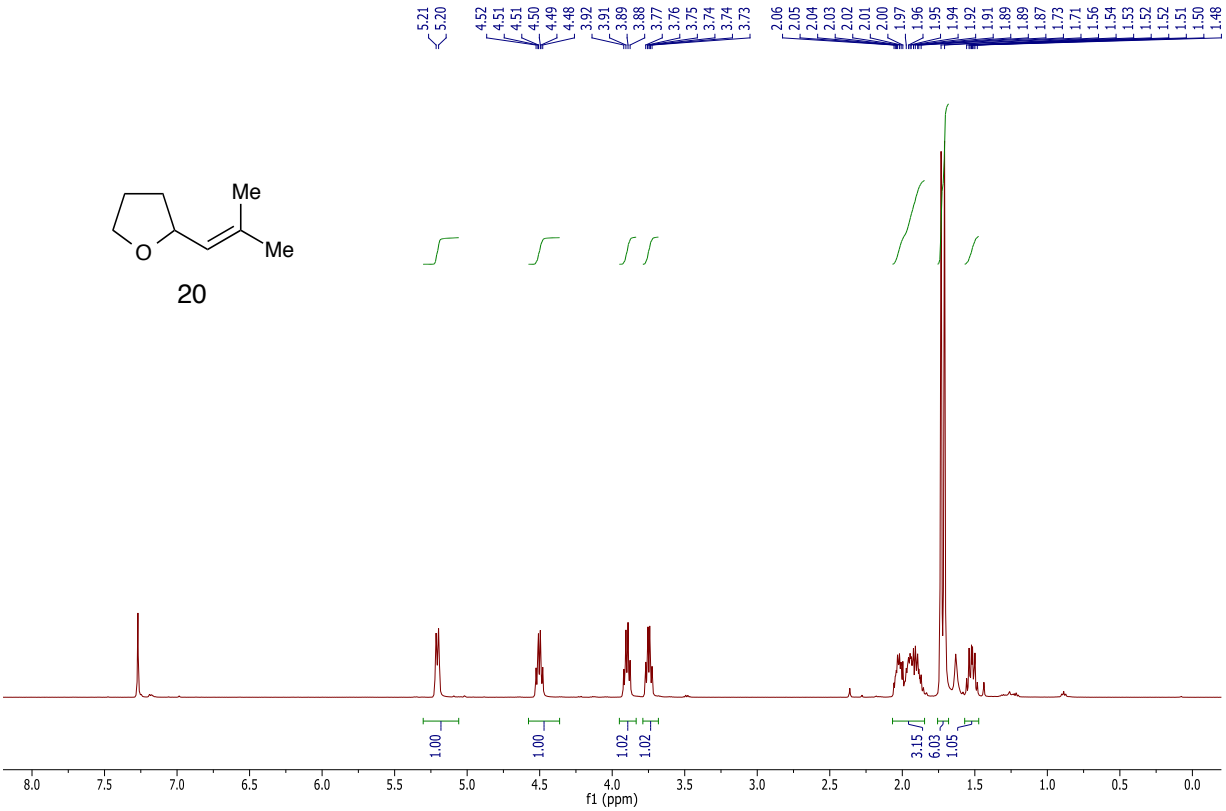
Me
O
19
f1 (ppm)

AN-2-653



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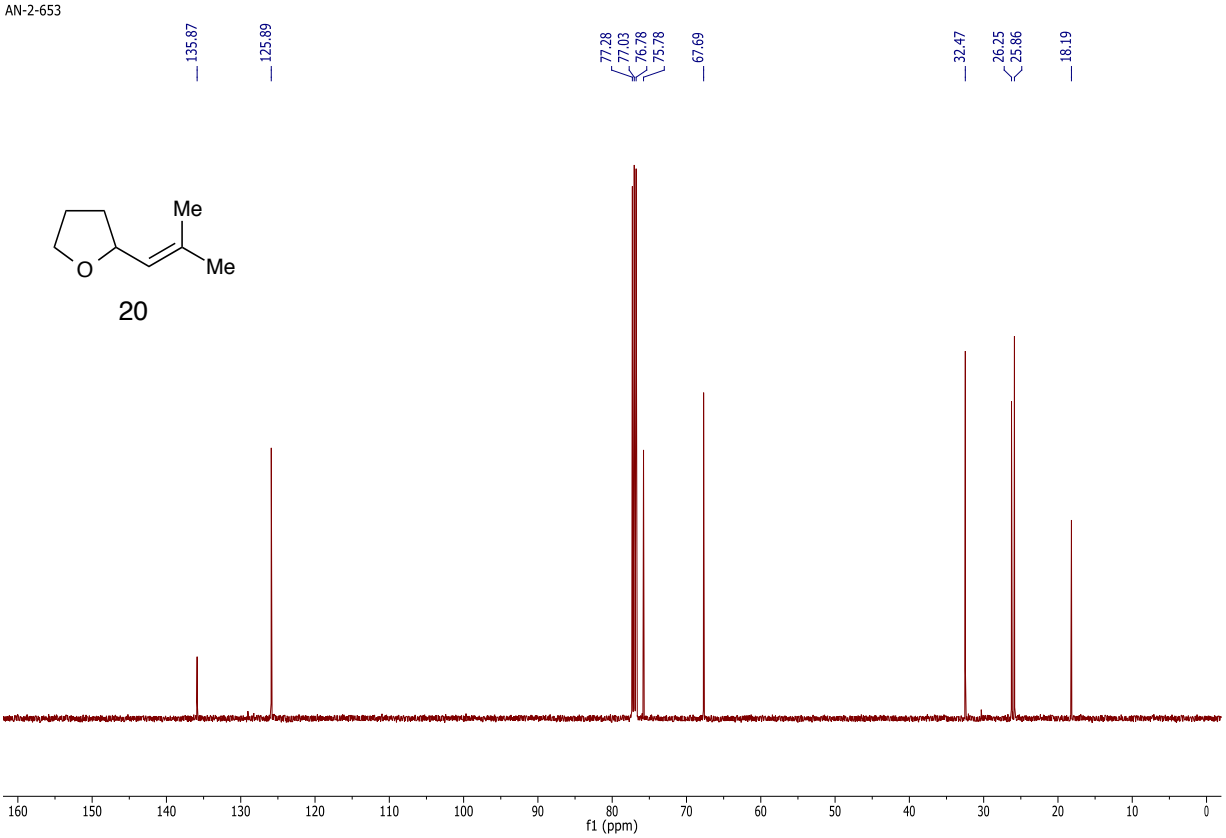

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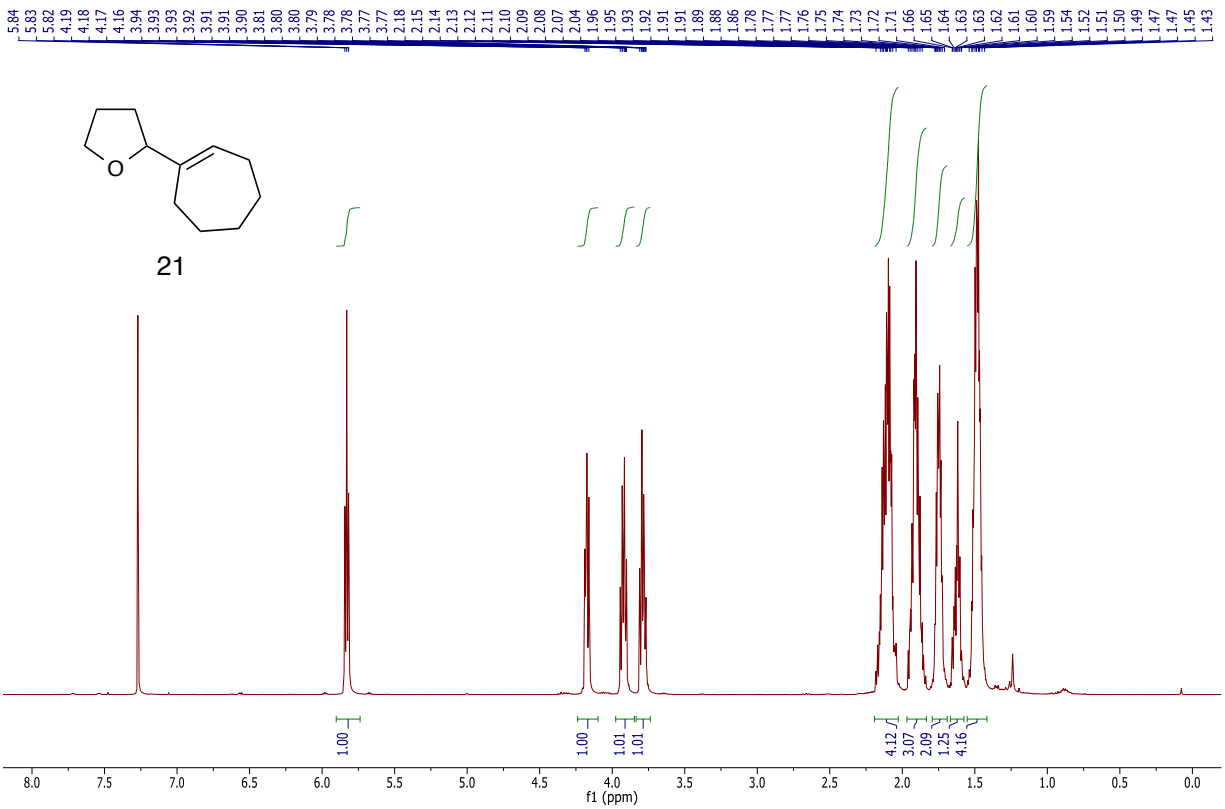

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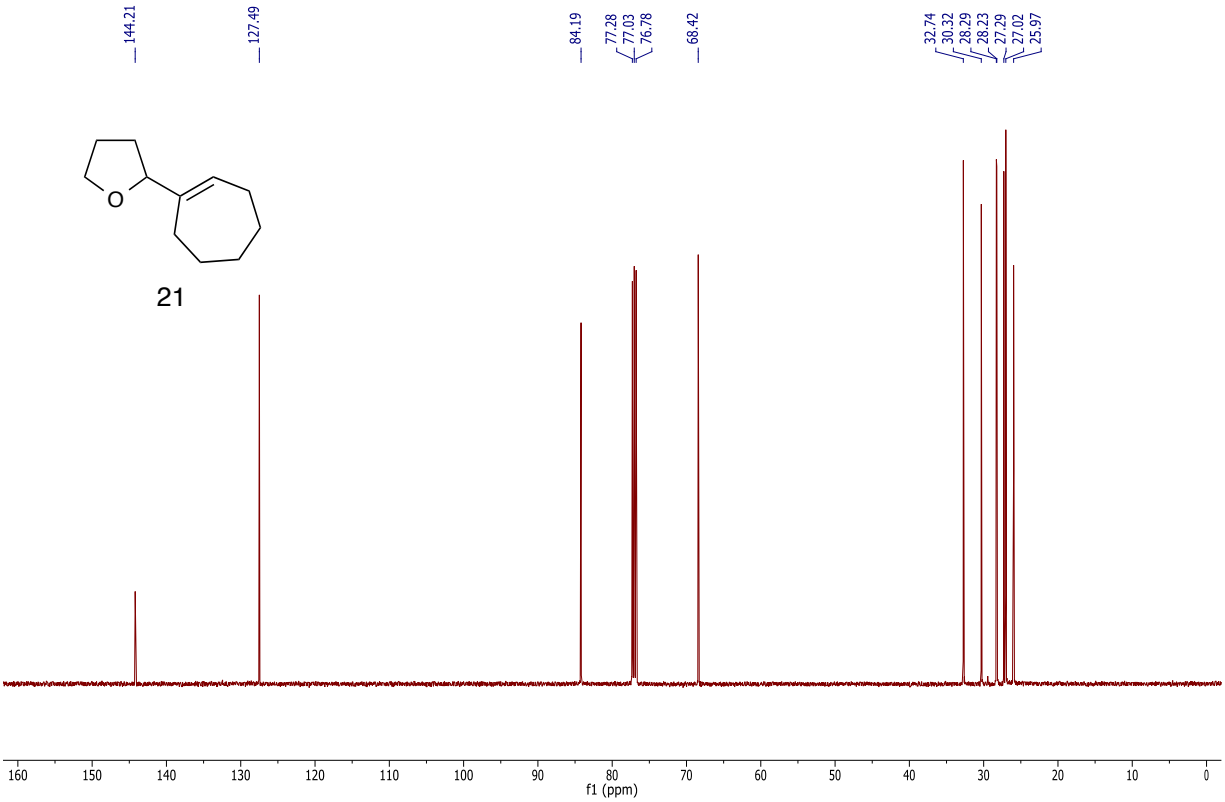

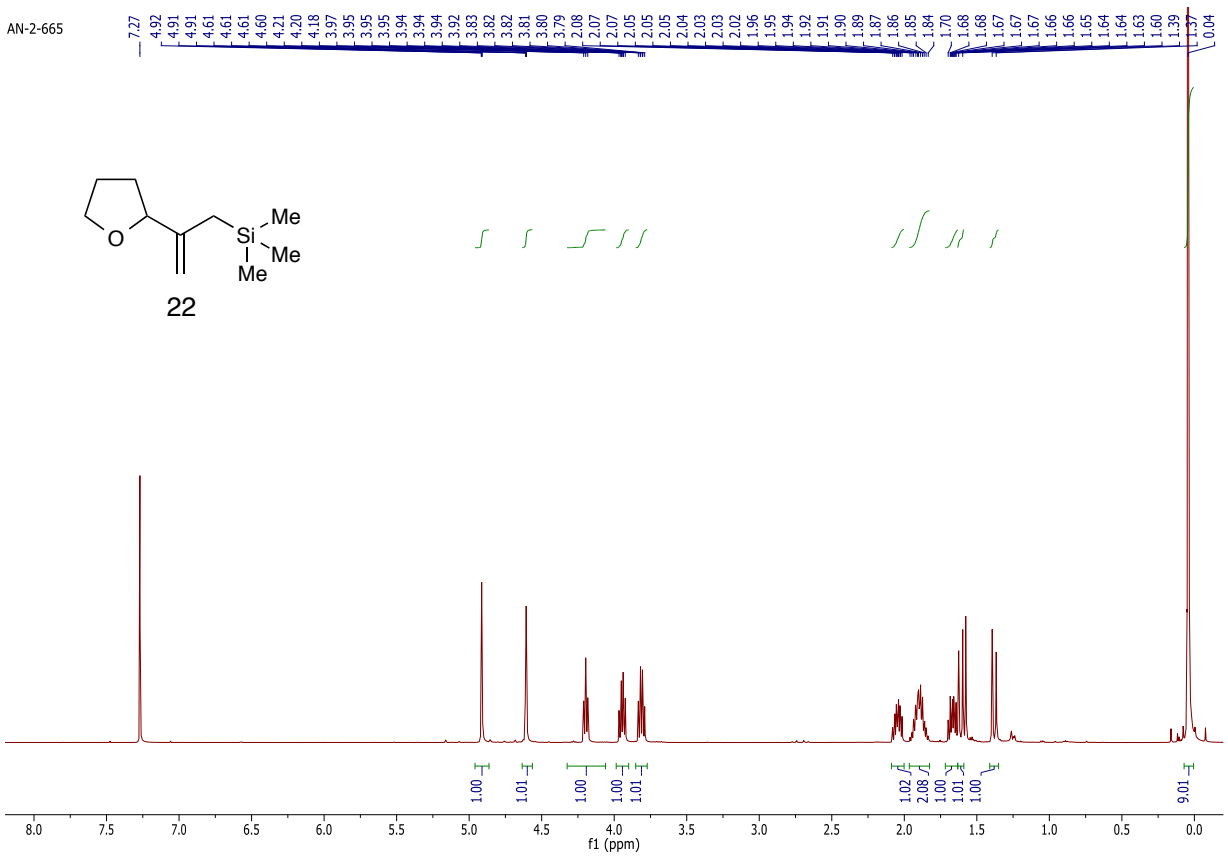
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Si
Me
Me
O
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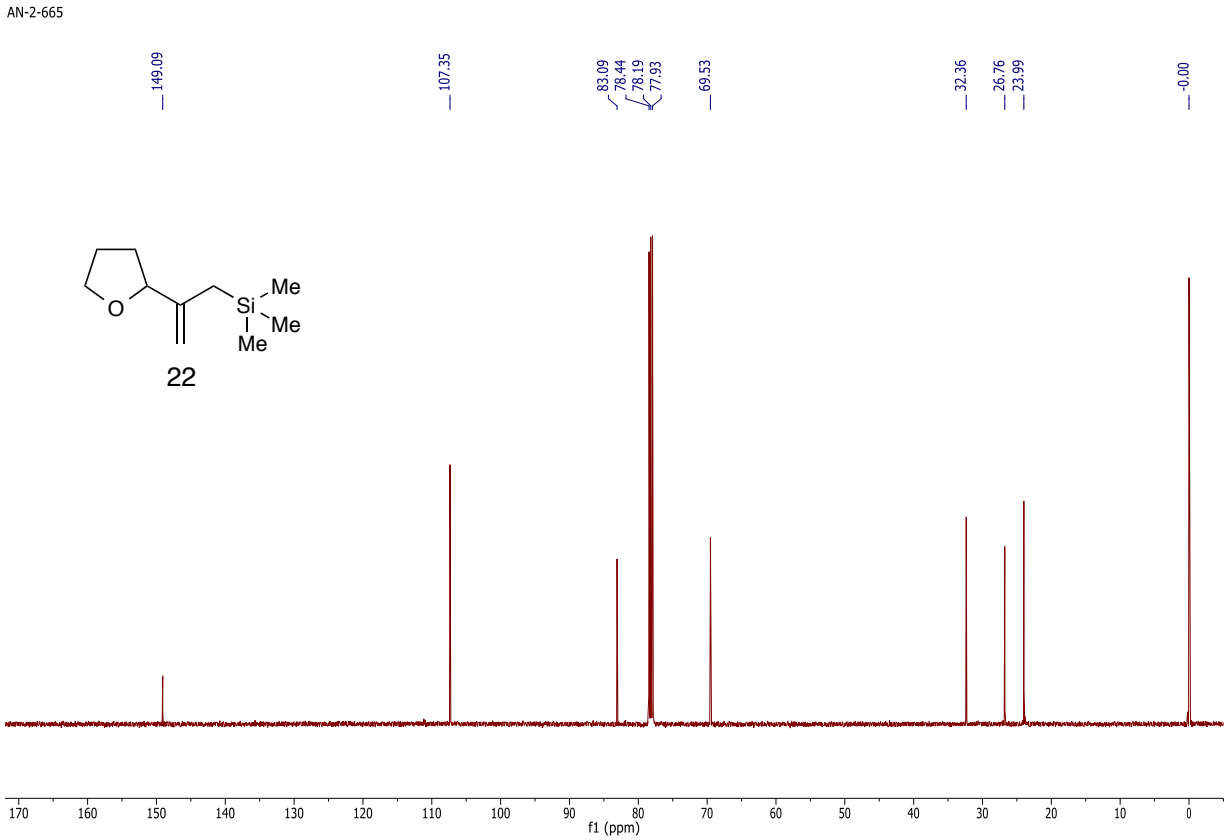
f1 (ppm)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O
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f1 (ppm)

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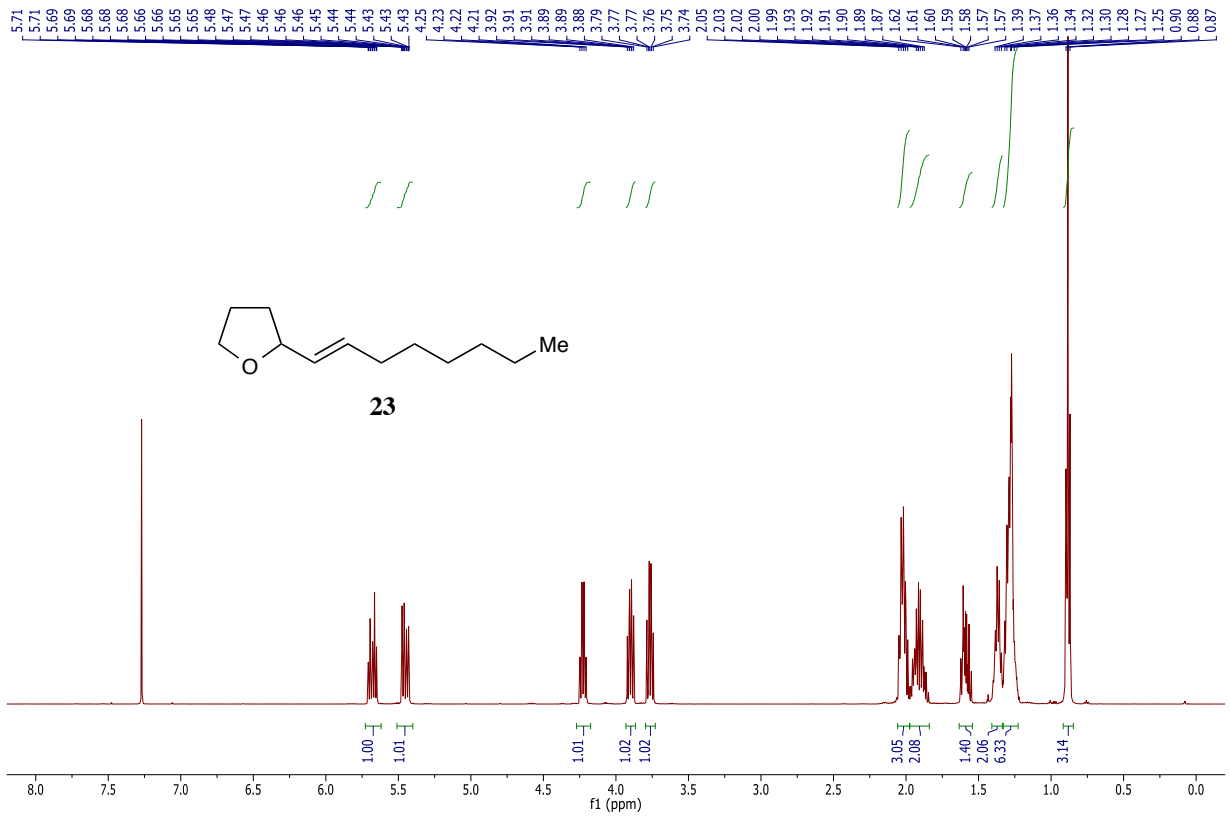

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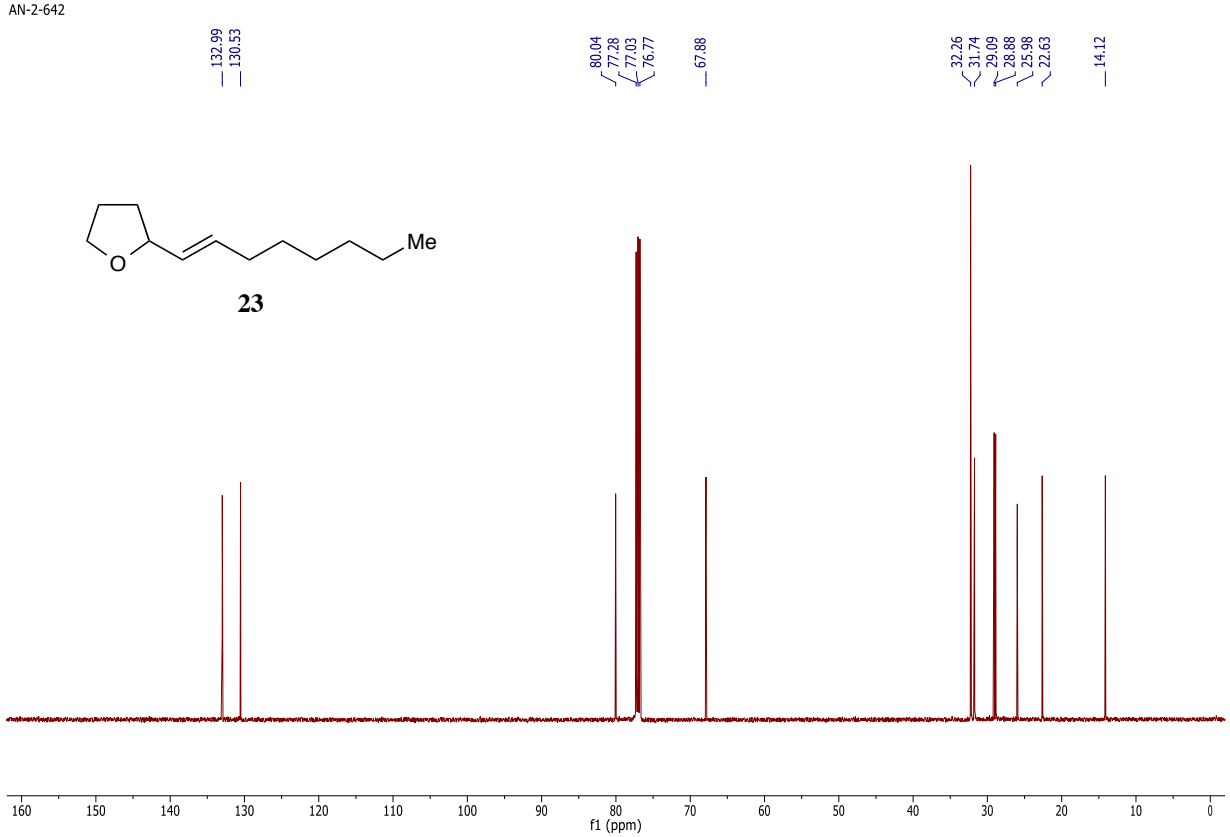

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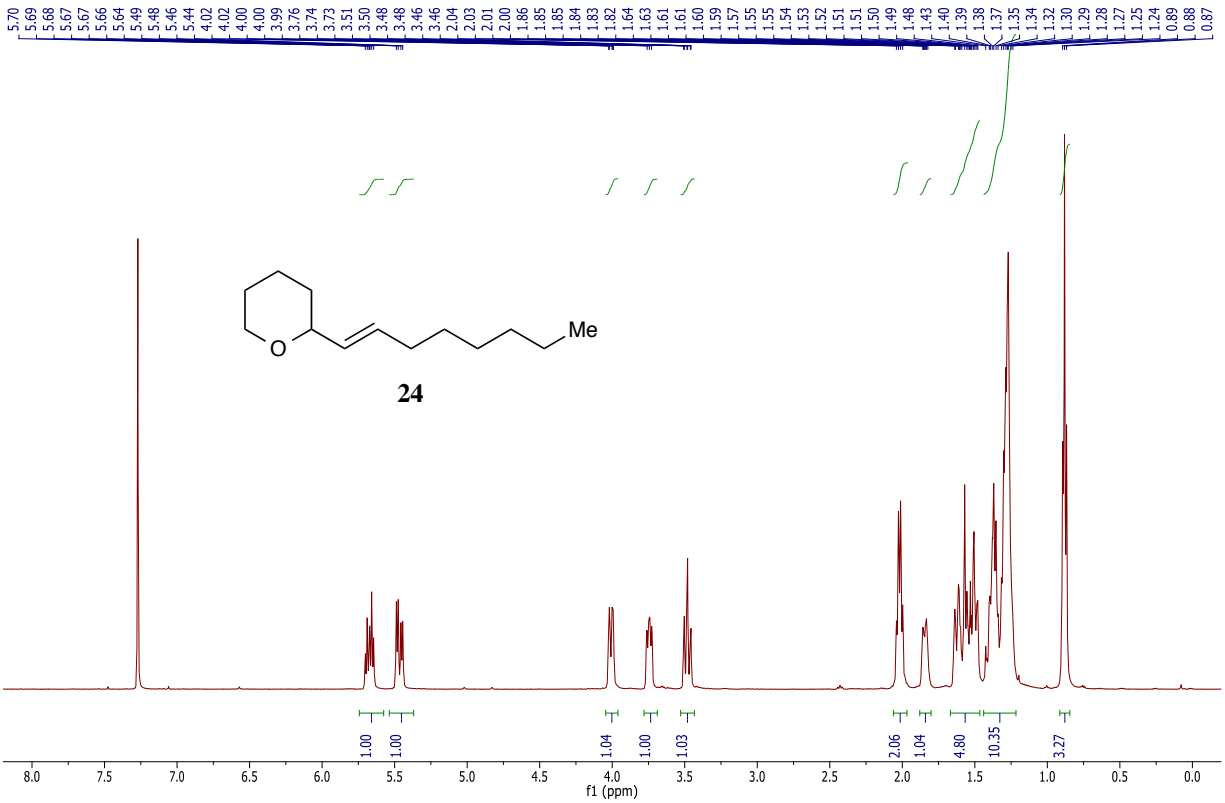

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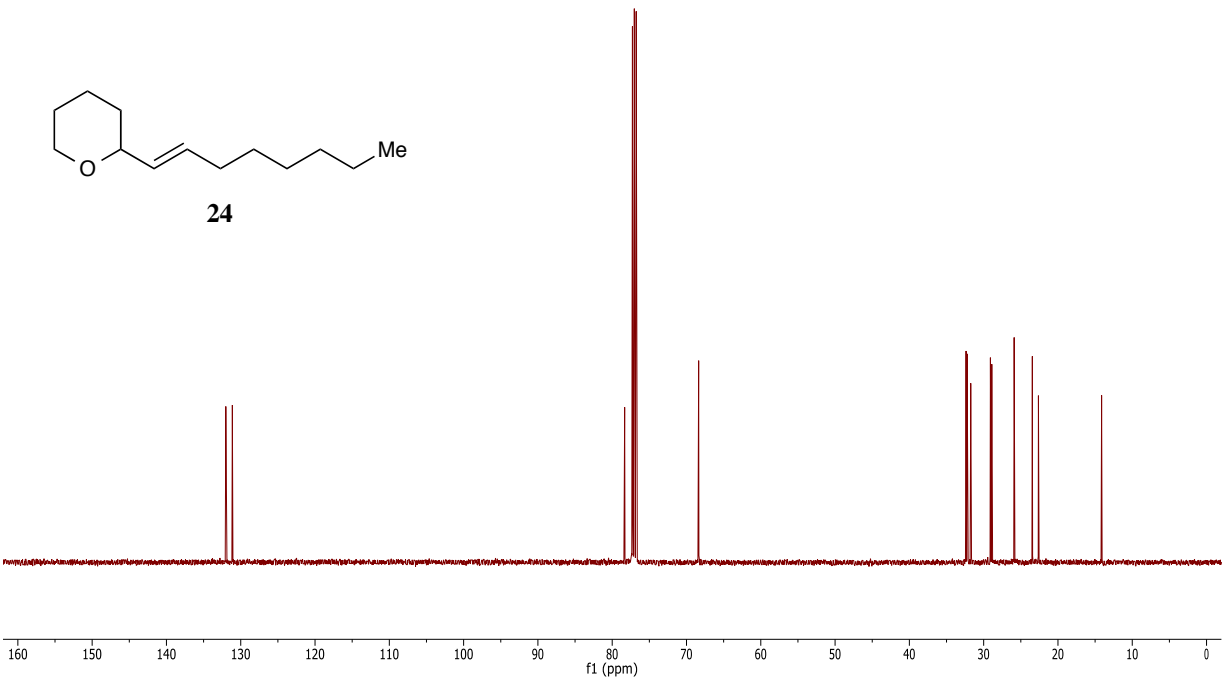

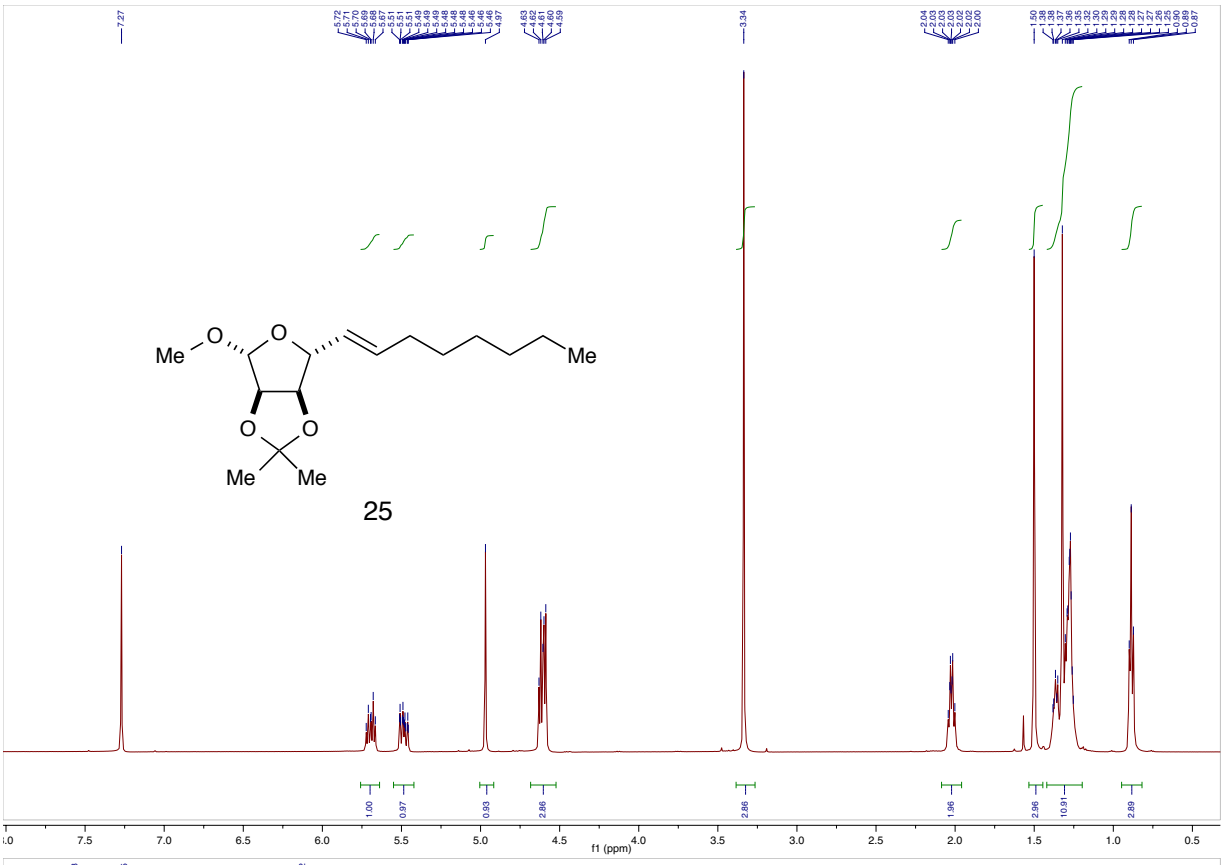
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Me
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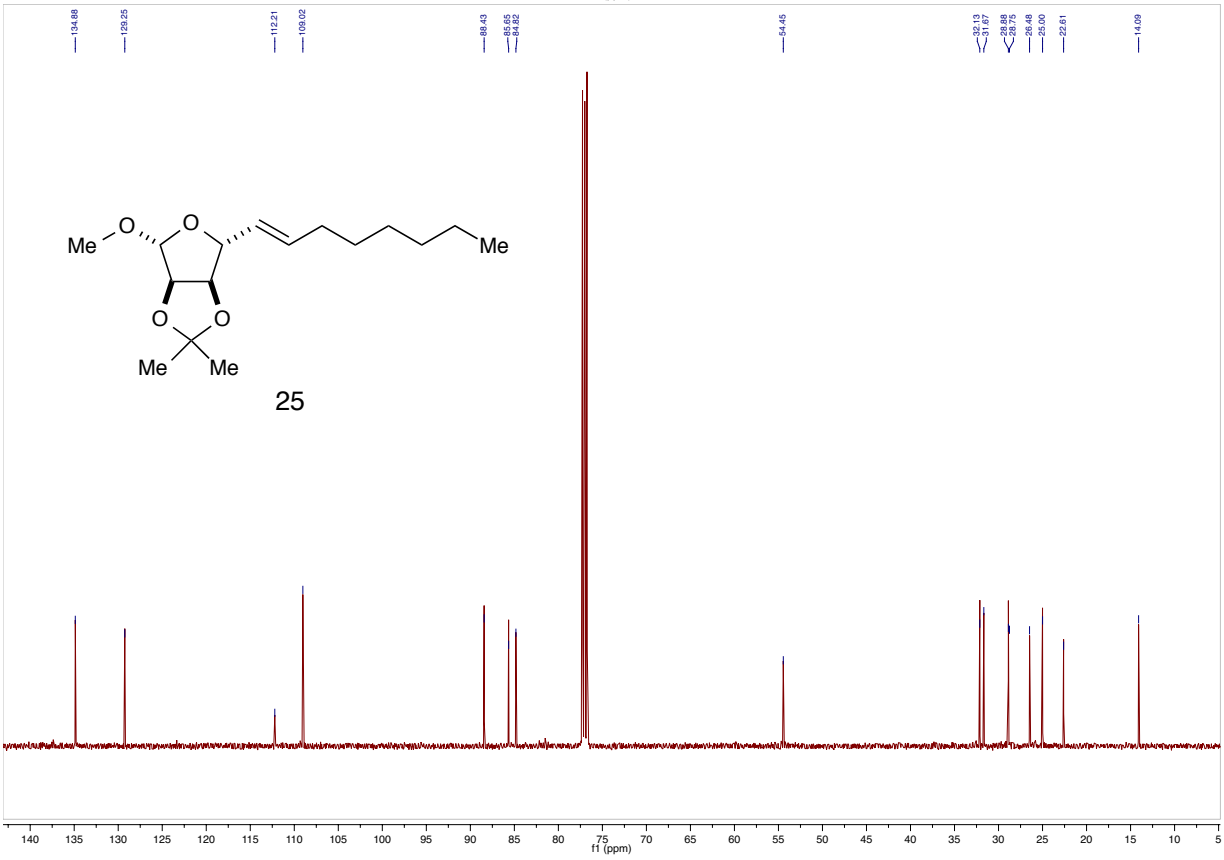
25
f1 (ppm)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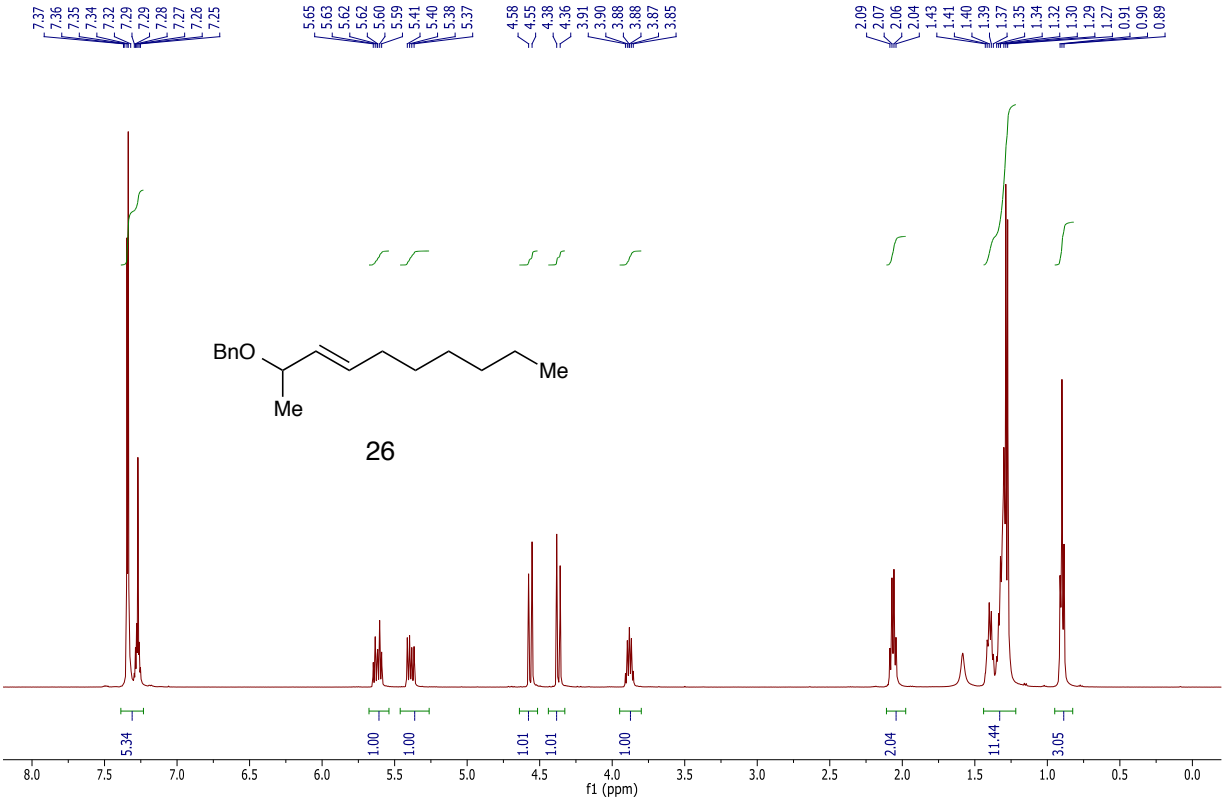

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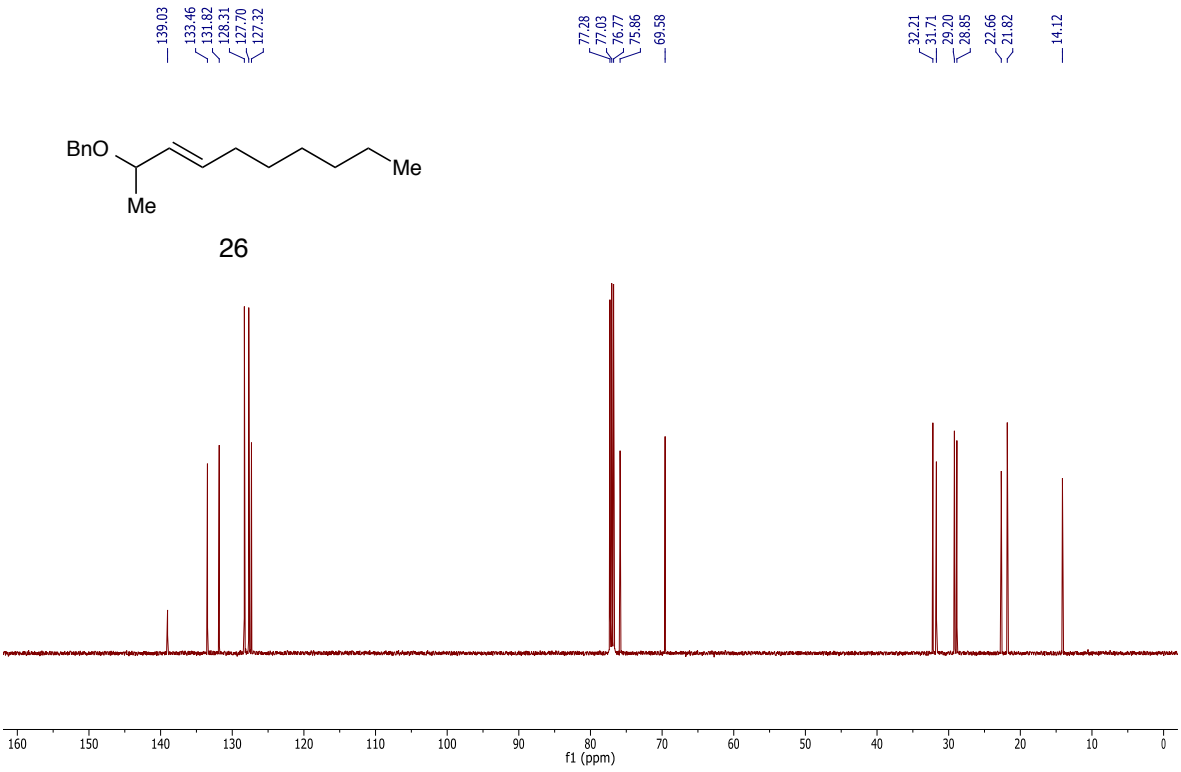

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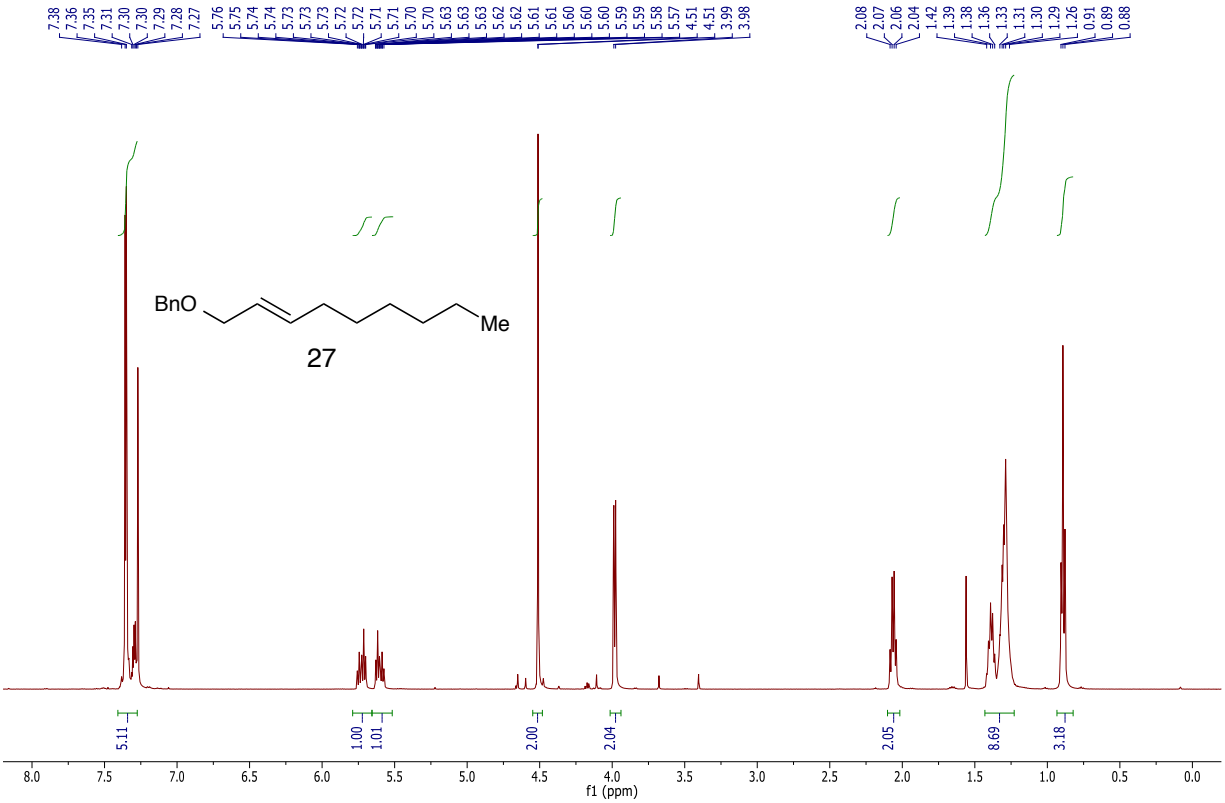

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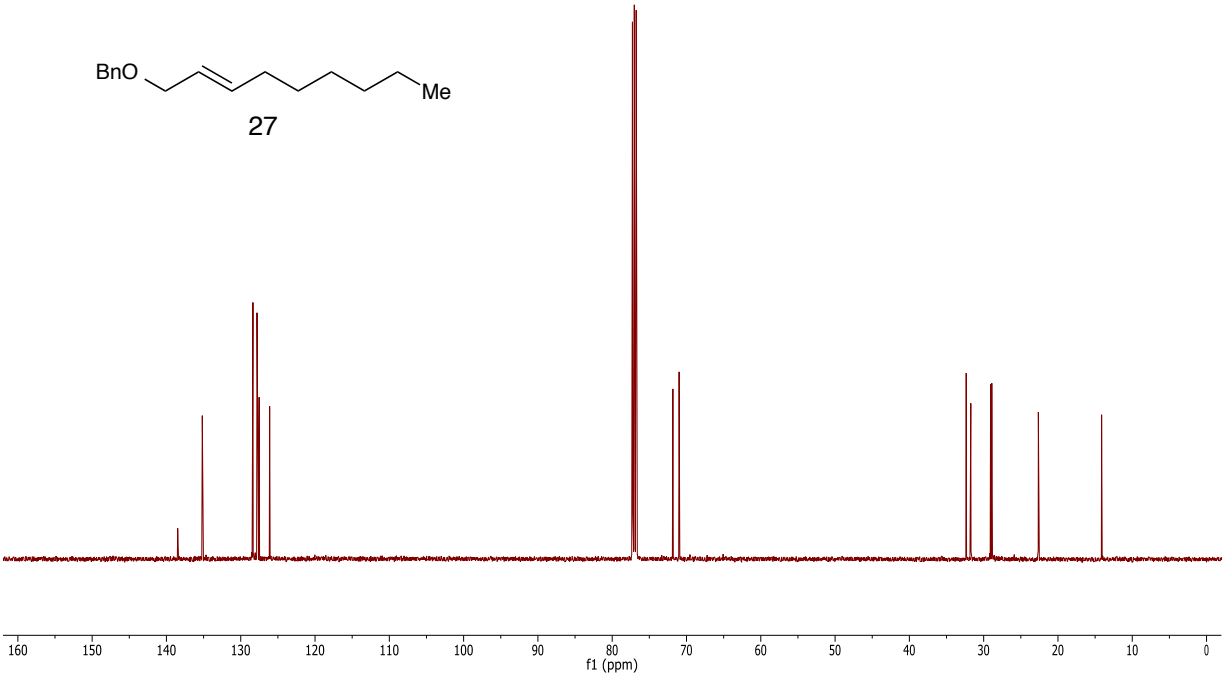
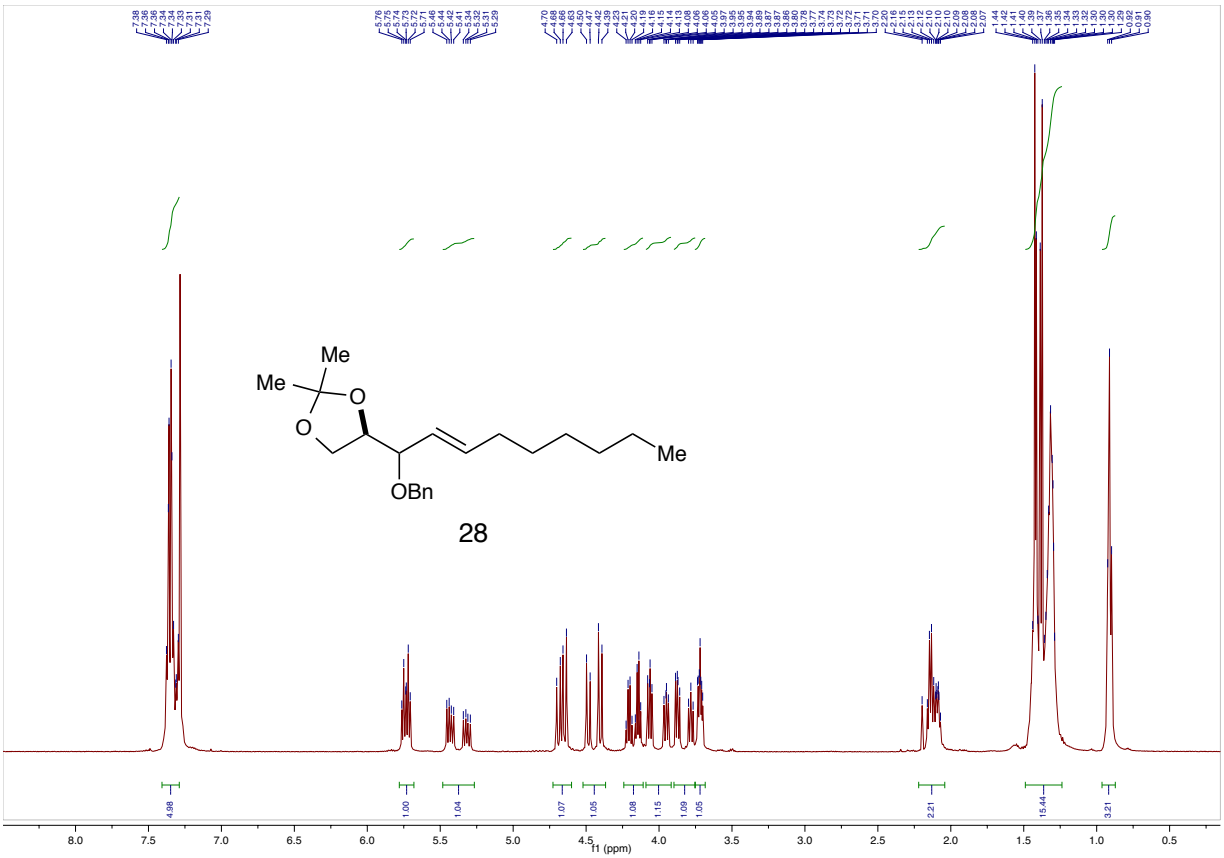
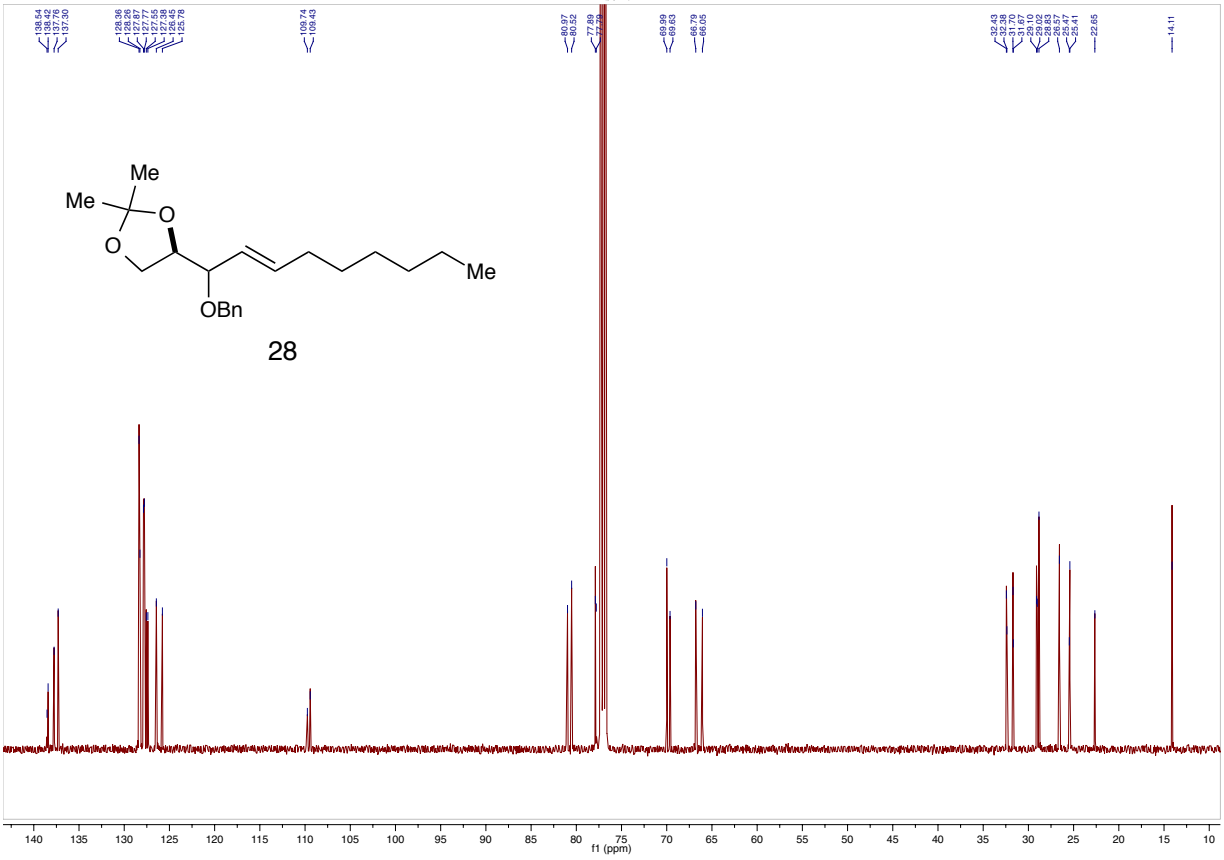




28



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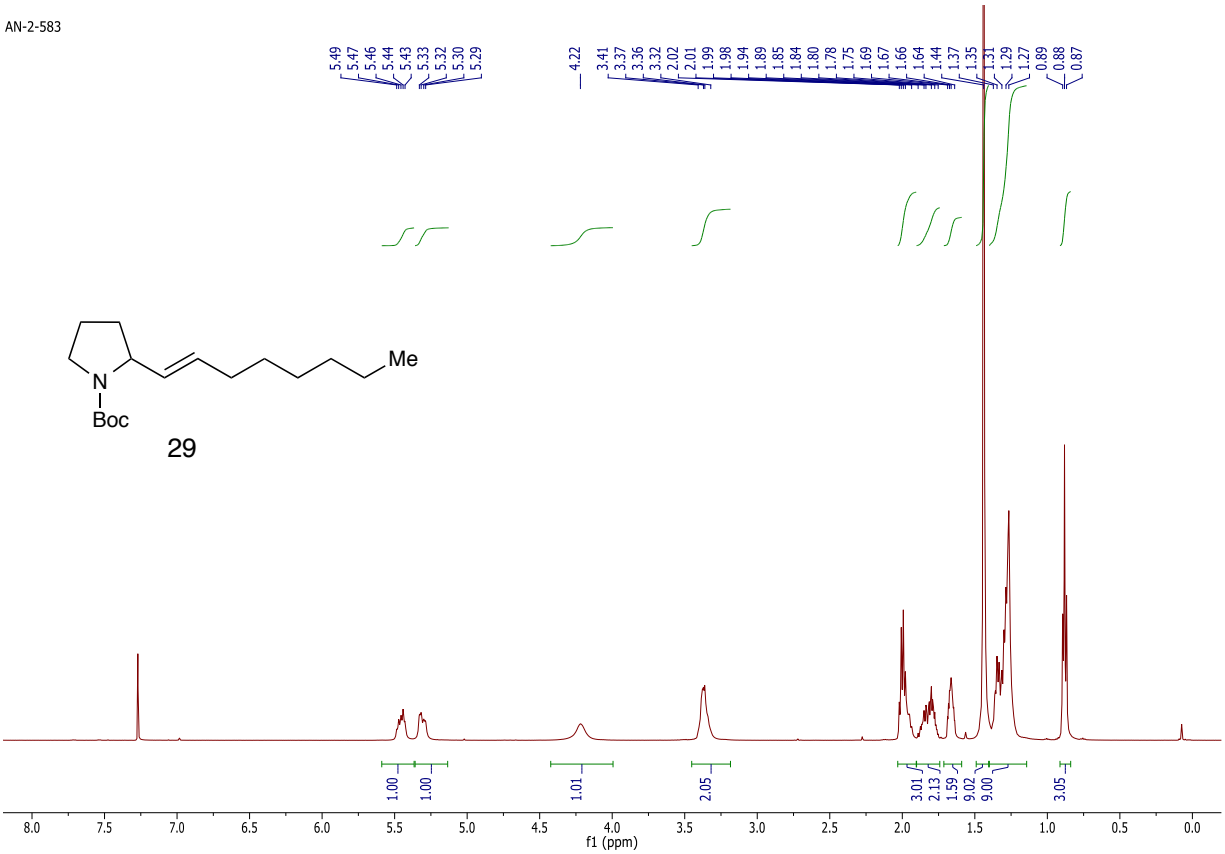
AN-2-583
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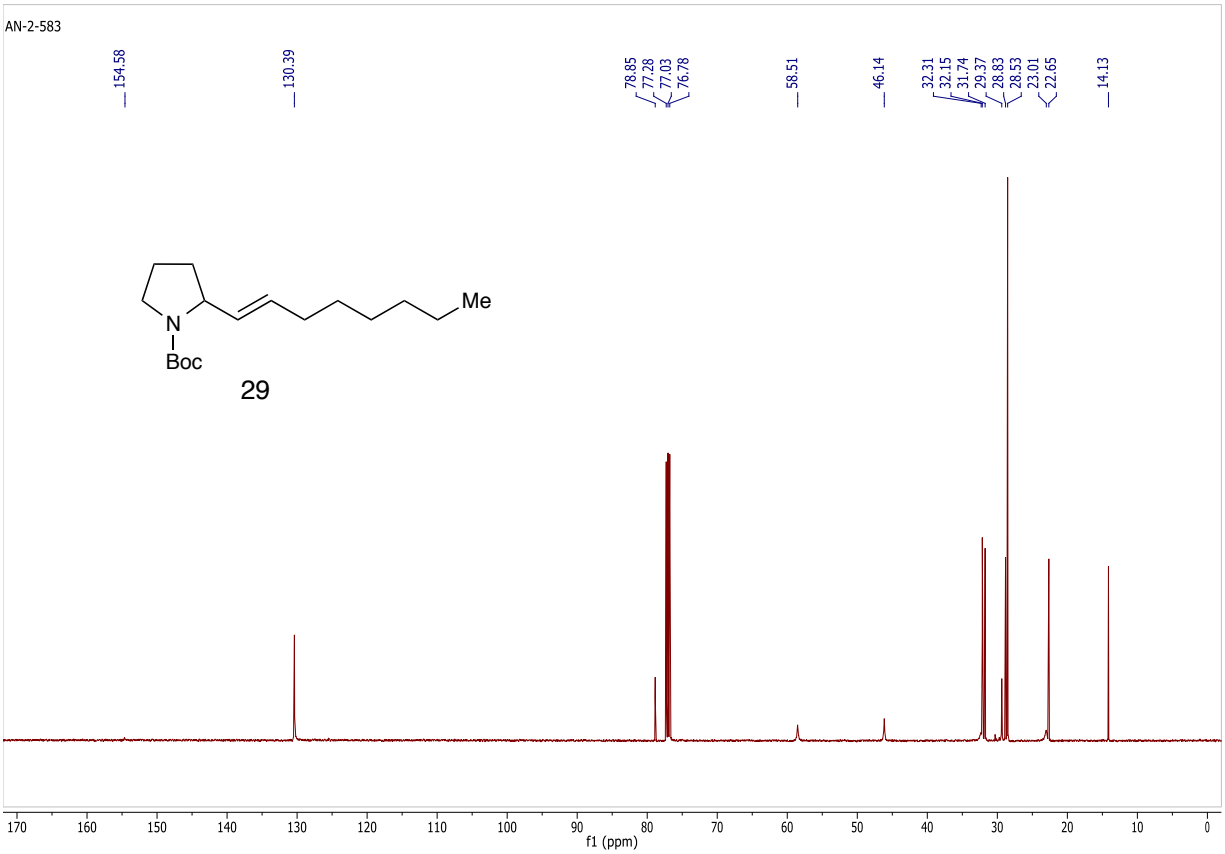
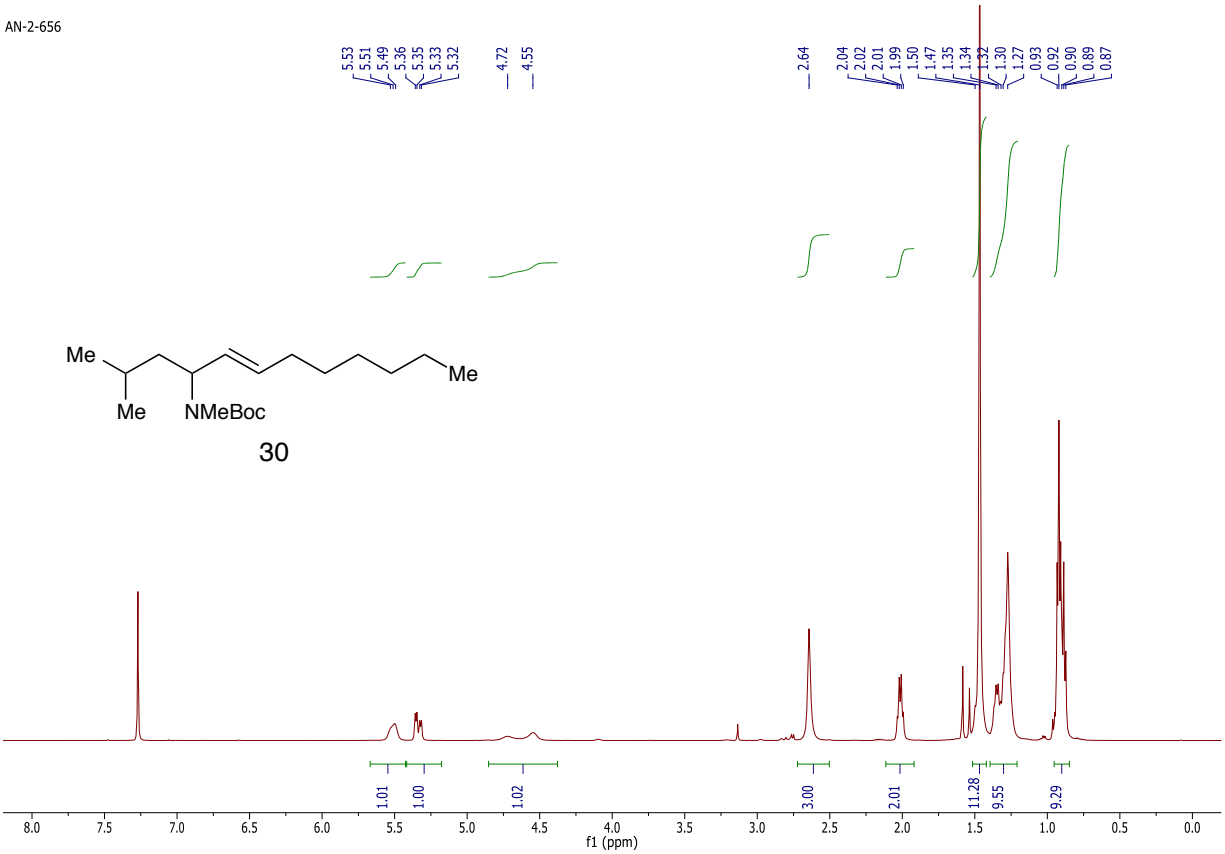
5.47
5.46
5.44
5.43
5.33
5.32
5.30
5.29
4.22
3.41
3.37
3.36
3.32
2.02
2.01
1.99
1.98
1.94
1.89
1.85
1.84
1.80
1.78
1.75
1.69
1.67
1.66
1.64
1.44
1.37
1.35
1.31
1.29
1.27
0.89
0.88
0.87
Me
N
Boc
29
1.00
1.00
1.01
2.05
3.01
2.13
1.59
9.02
9.00
3.05
f1 (ppm)


AN-2-583
154.58
130.39
78.85
77.28
77.03
76.78
58.51
46.14
32.31
32.15
31.74
29.37
28.83
28.53
23.01
22.65
14.13
Me
N
Boc
29
f1 (ppm)


AN-2-656
5.53
5.51
5.49
5.36
5.35
5.33
5.32
4.72
4.55
2.64
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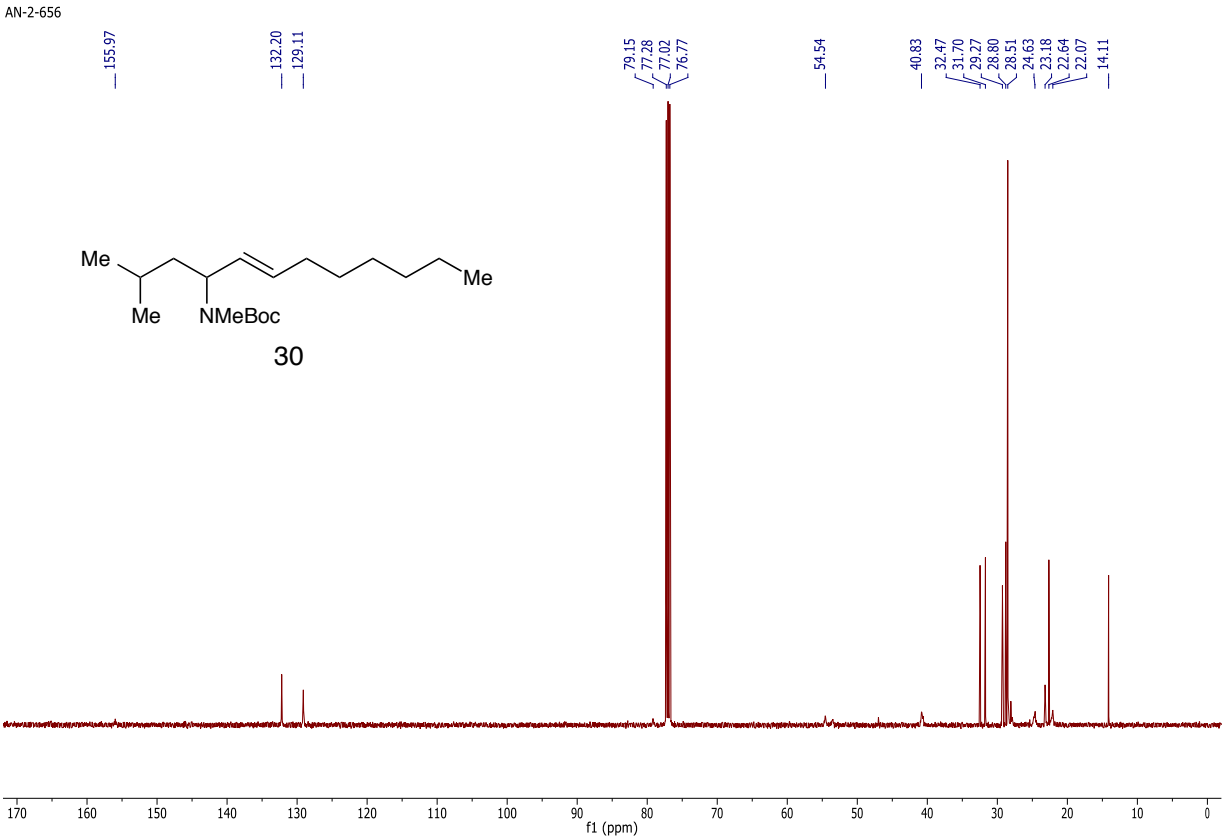
2.02
2.01
1.99
1.50
1.47
1.35
1.34
1.32
1.30
1.27
0.93
0.92
0.90
0.89
0.87
Me
Me
NMeBoc
Me
30
1.01
1.00
1.02
3.00
2.01
11.28
9.55
9.29
f1 (ppm)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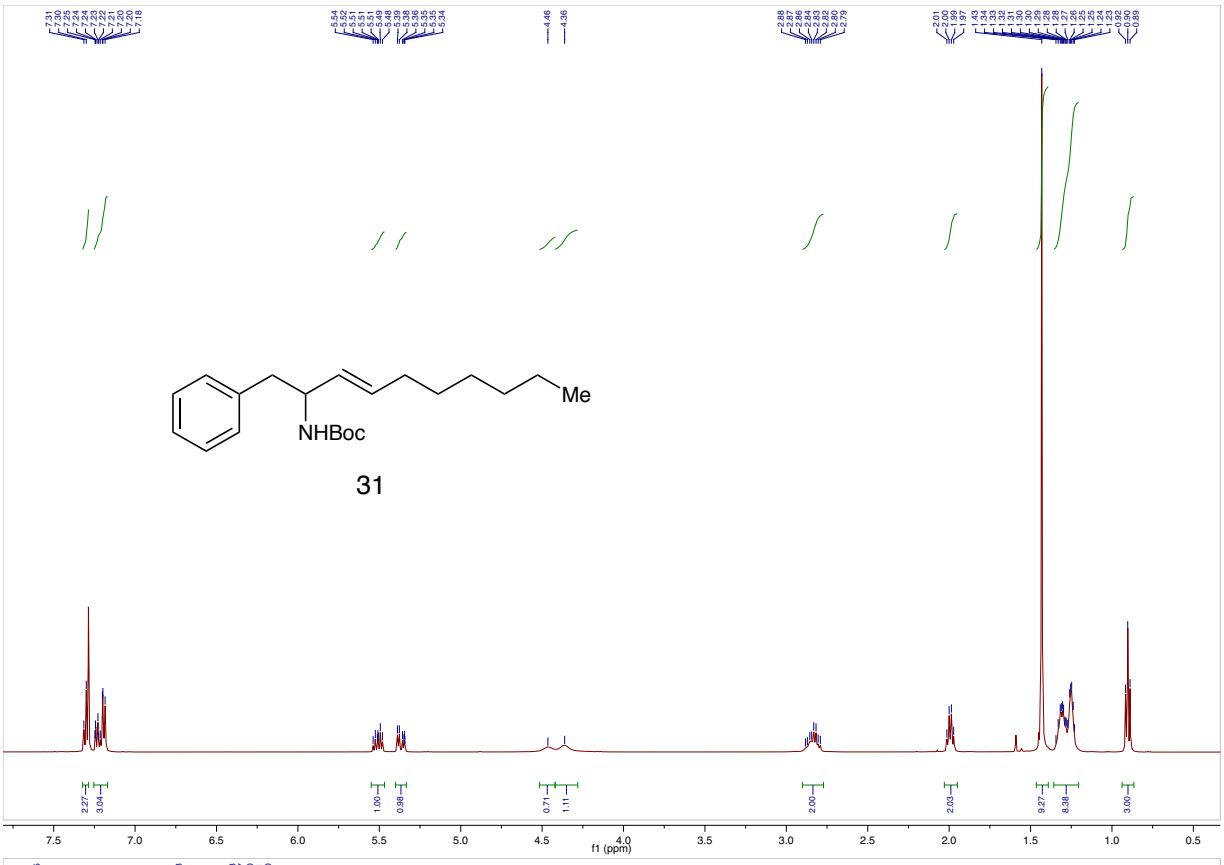
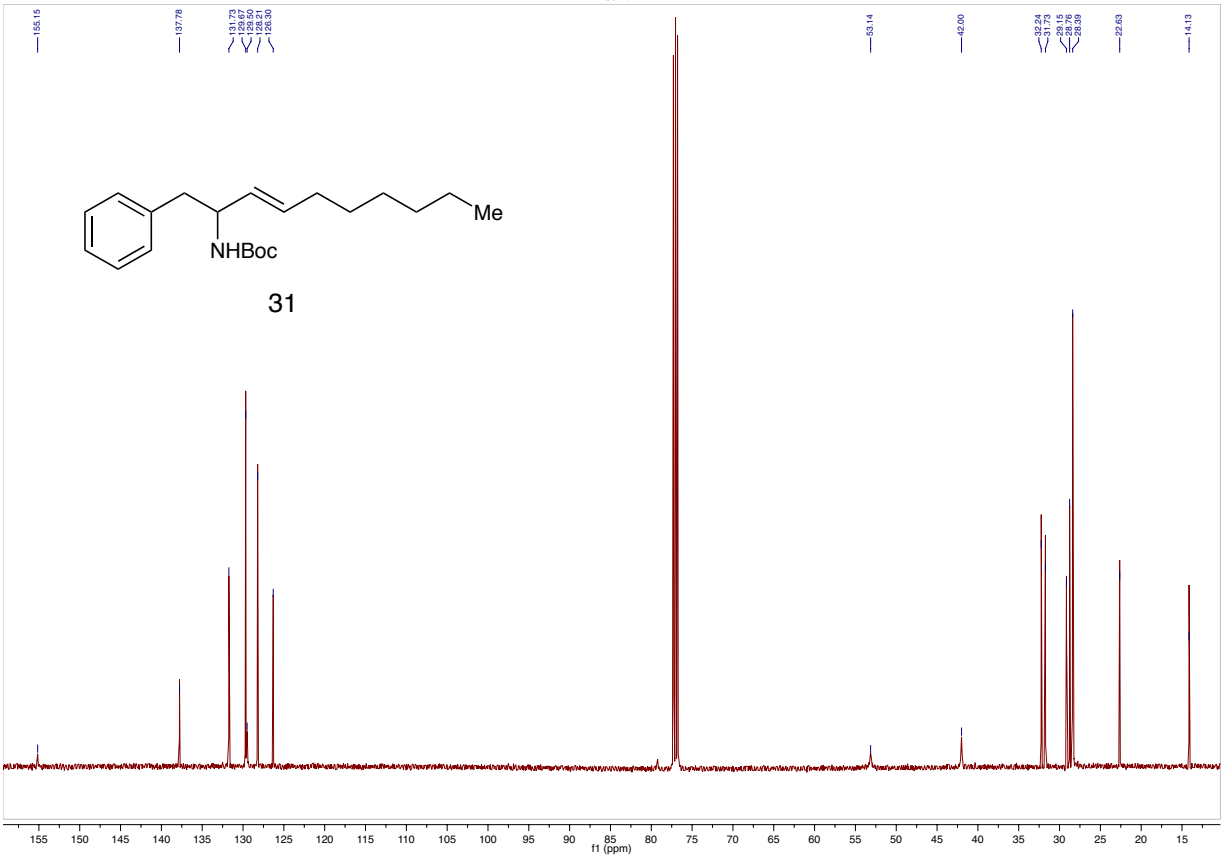
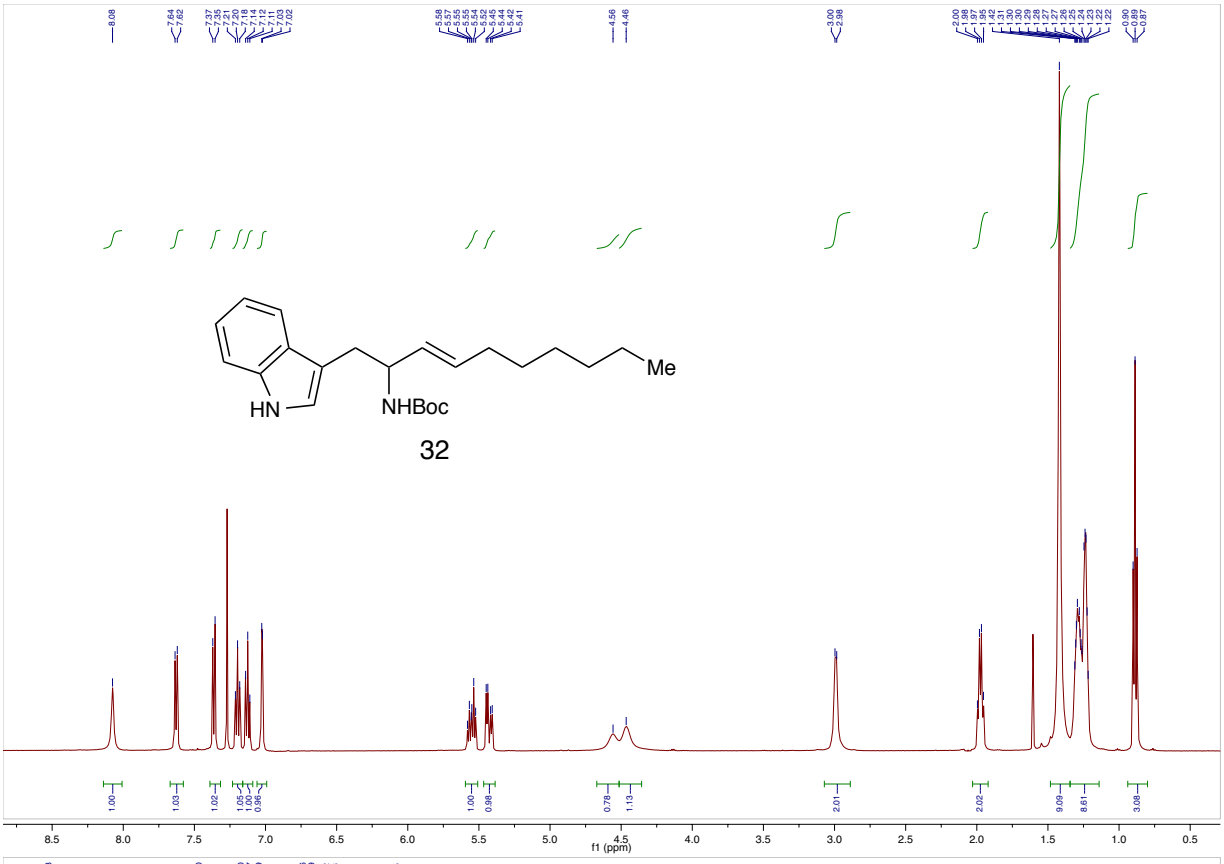
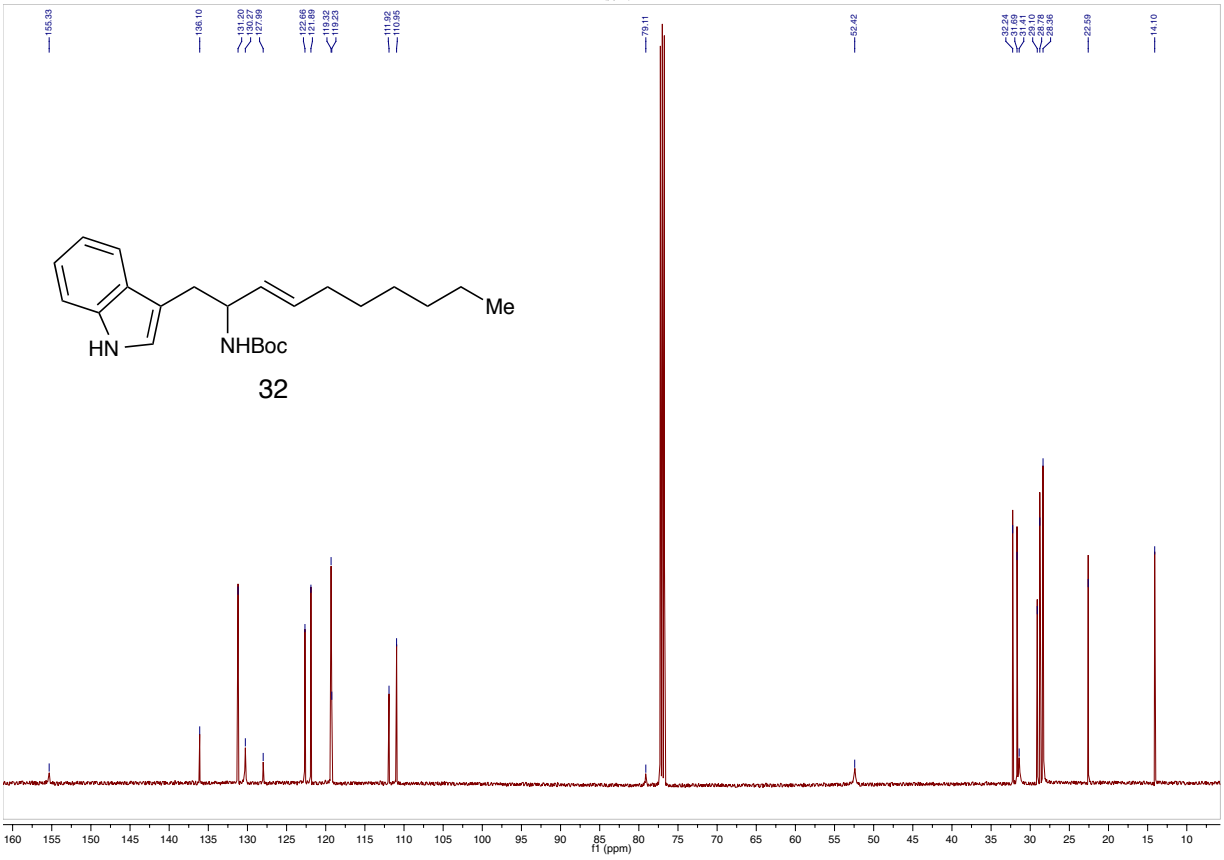
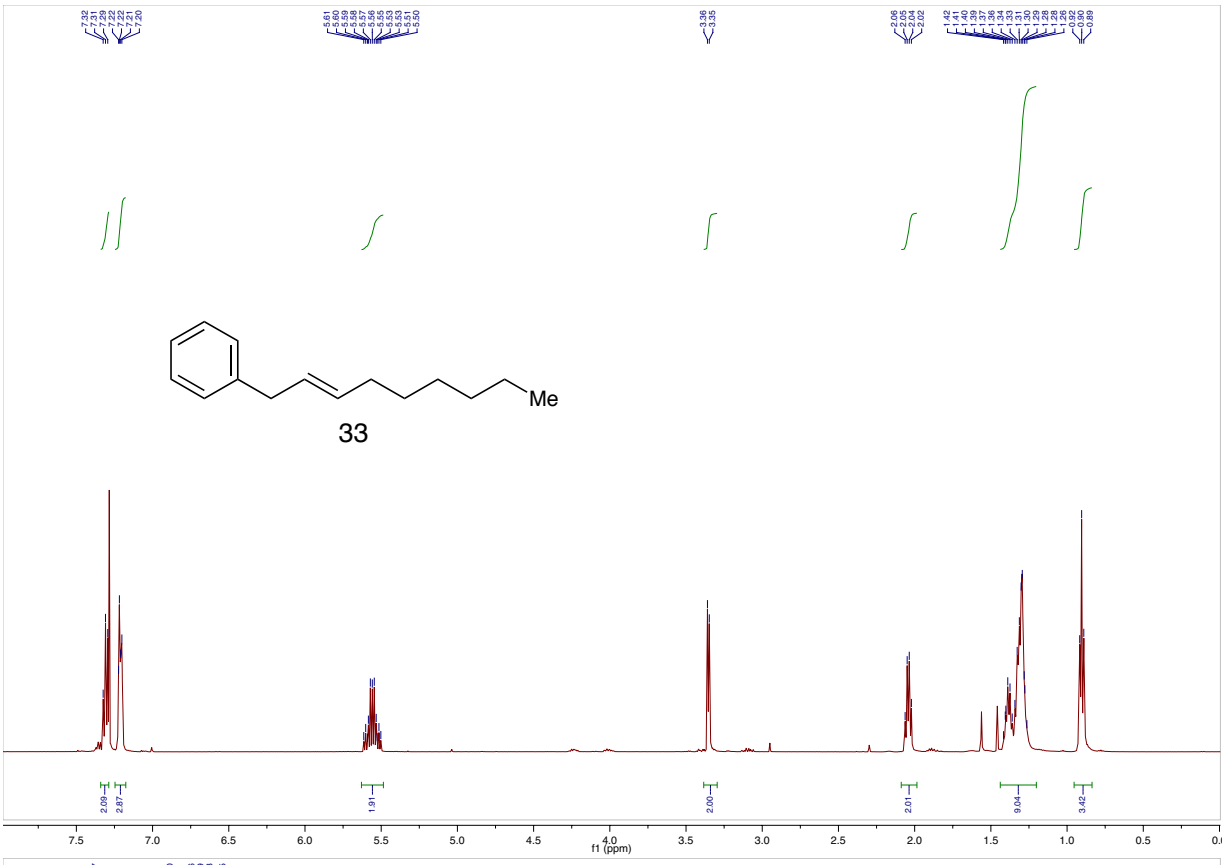
155.97
132.20
129.11
79.15
77.28
77.02
76.77
54.54
40.83
32.47
31.70
29.27
28.80
28.51
24.63
23.18
22.64
22.07
14.11
Me
Me
NMeBoc
Me
30
f1 (ppm)

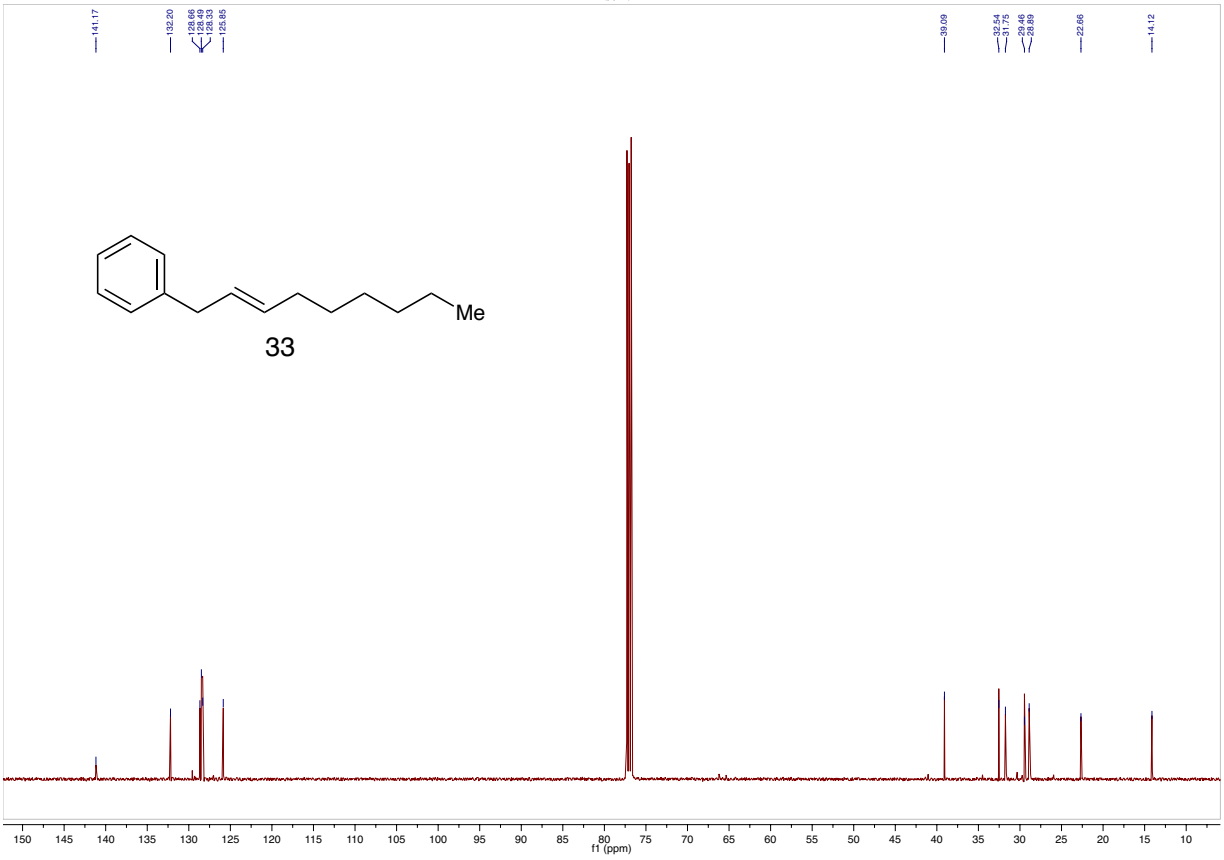

Me
NHBoc
31
f1 (ppm)

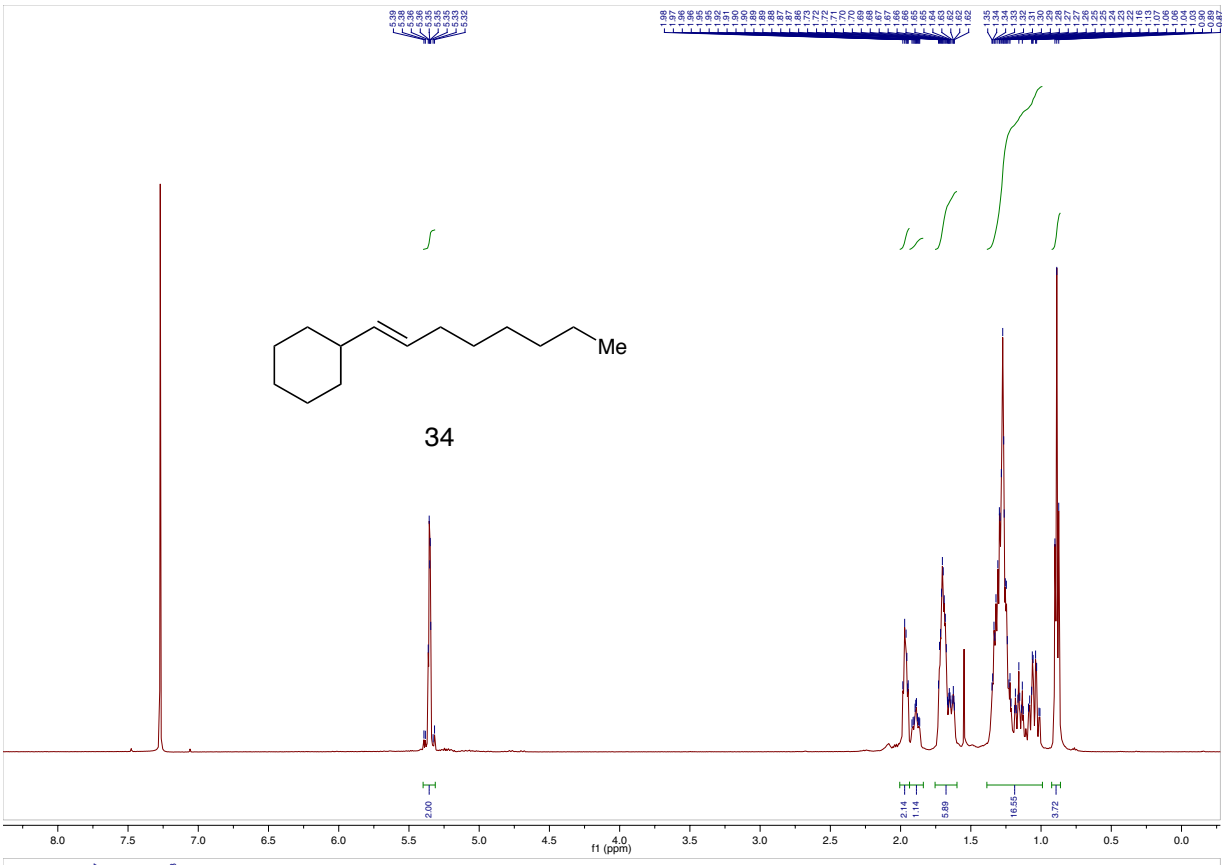

Me
NHBoc
31
f1 (ppm)

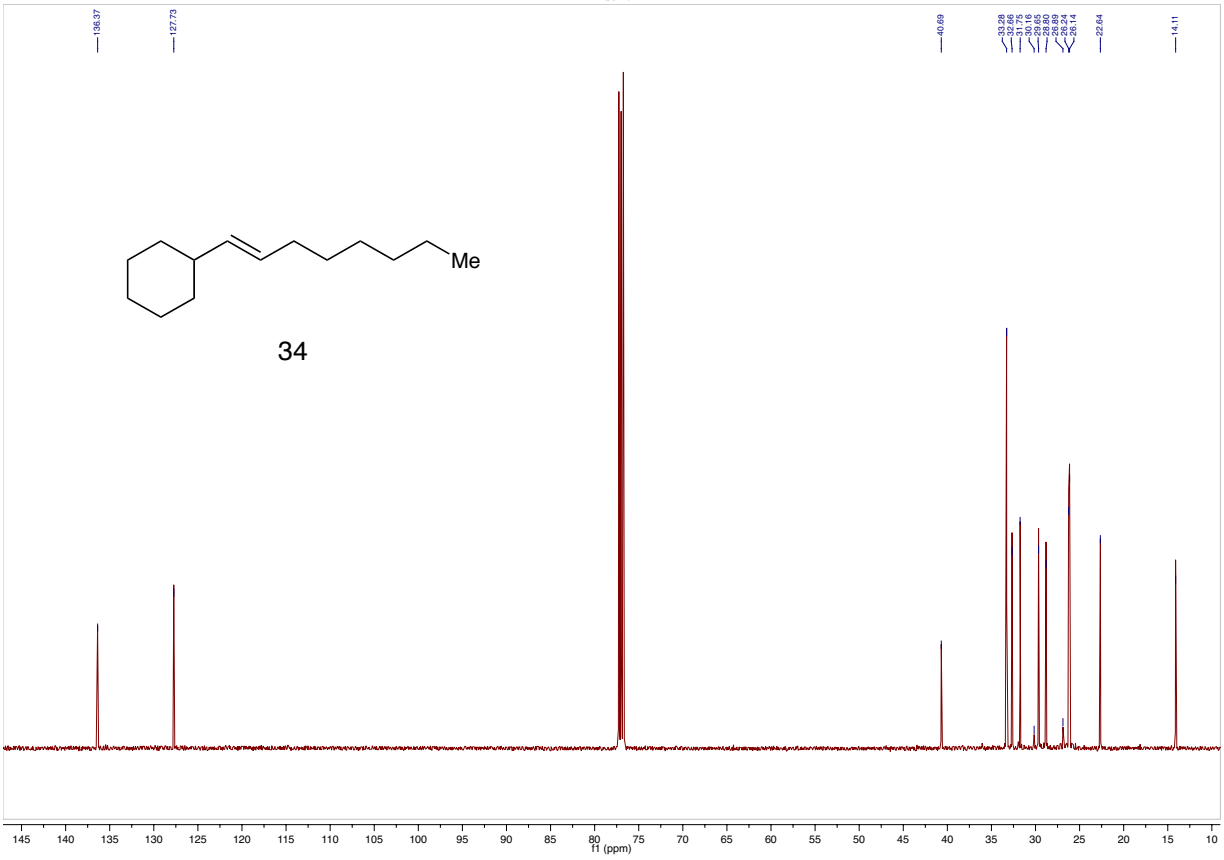

HN
NHBoc
Me
32
f1 (ppm)

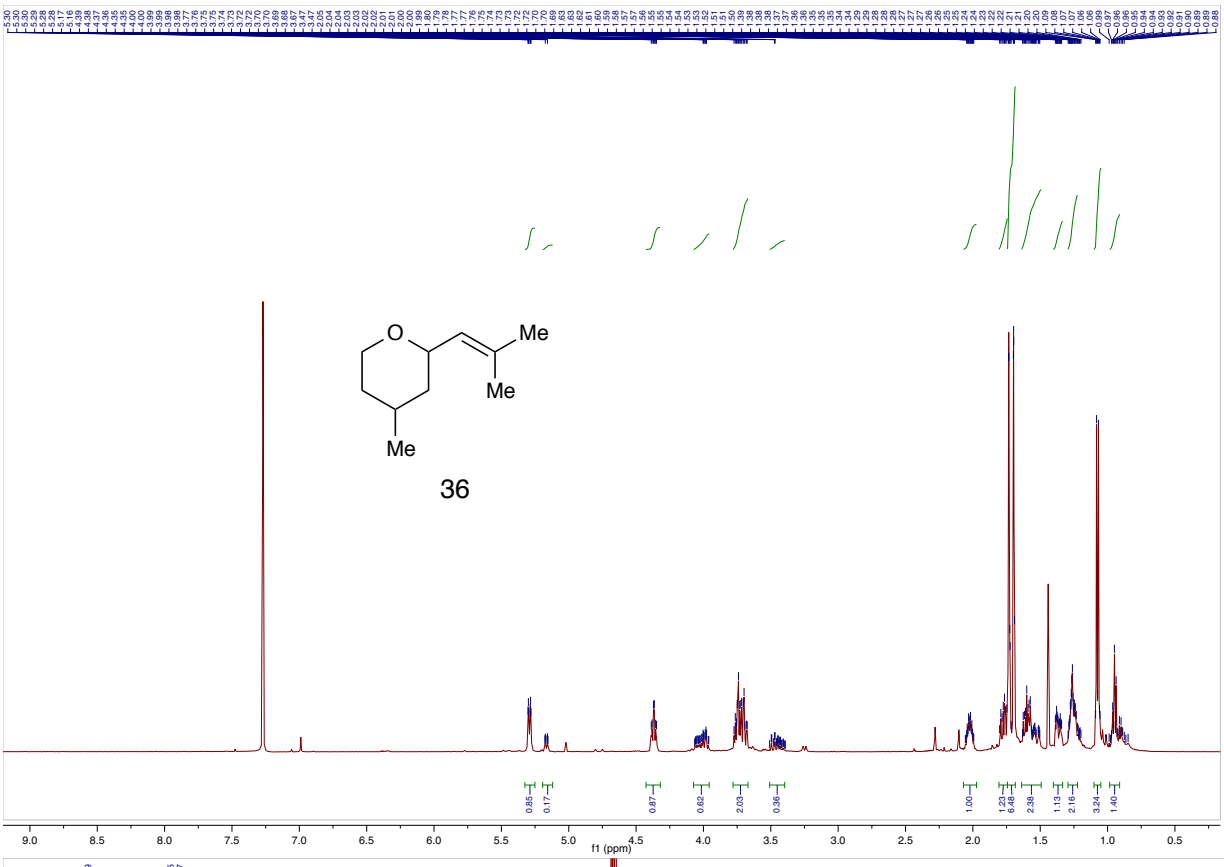

HN
NHBoc
Me
32
f1 (ppm)

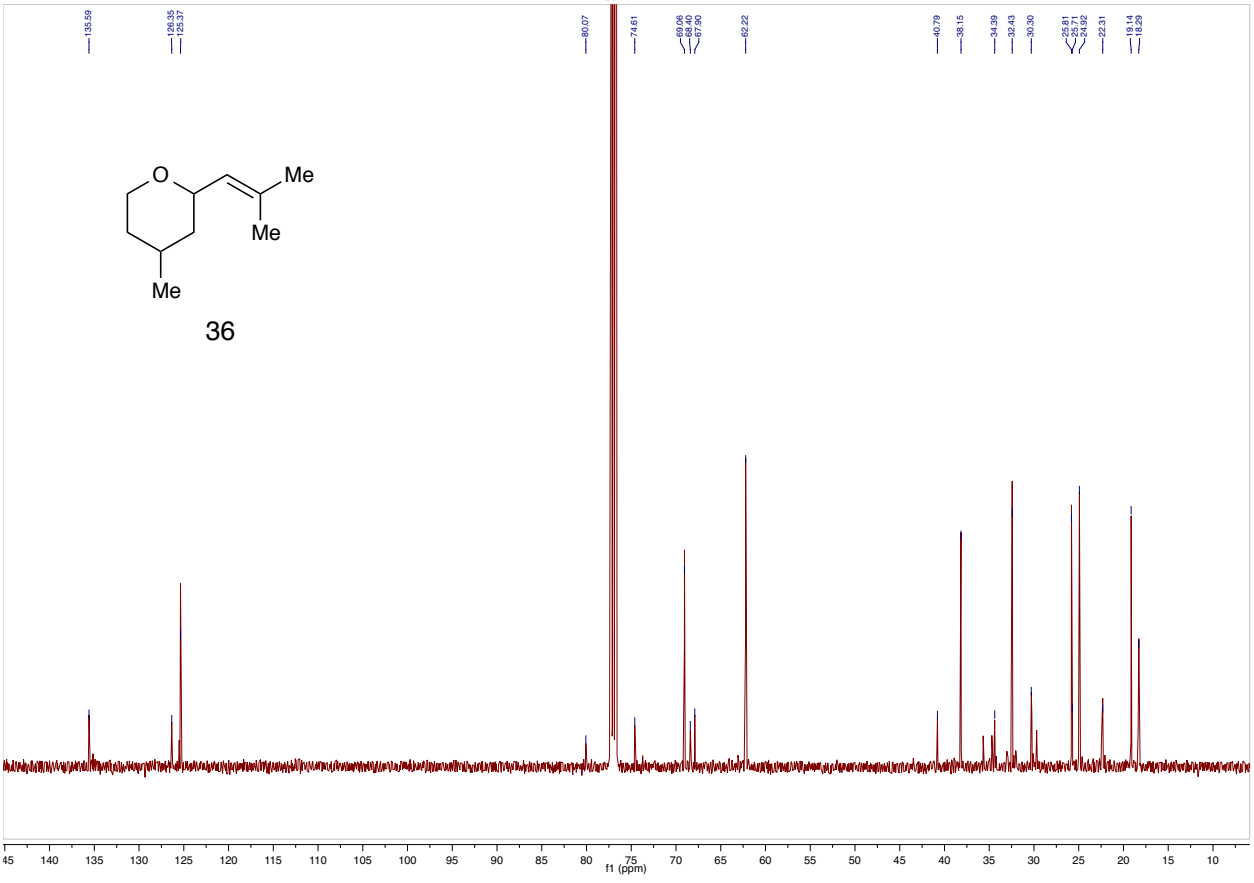

Me
33
f1 (ppm)

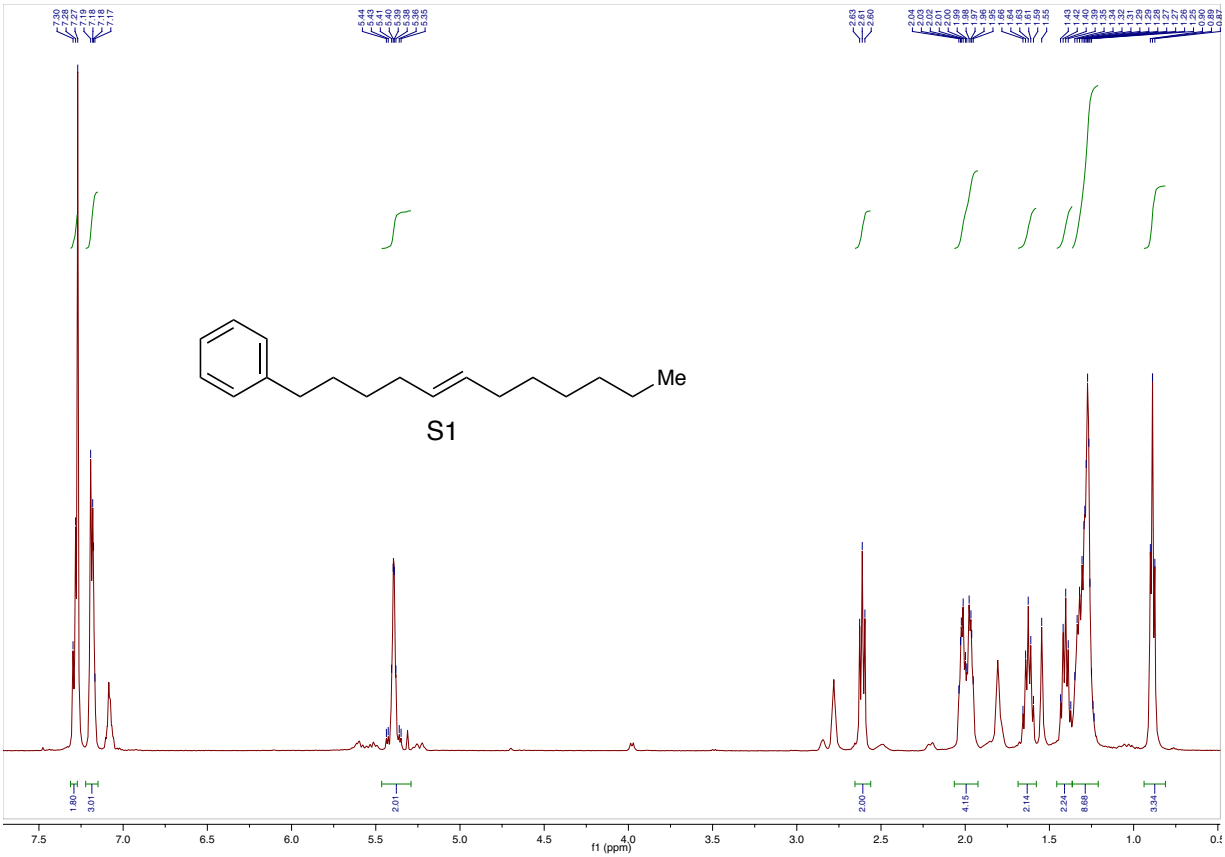

Me
33
f1 (ppm)

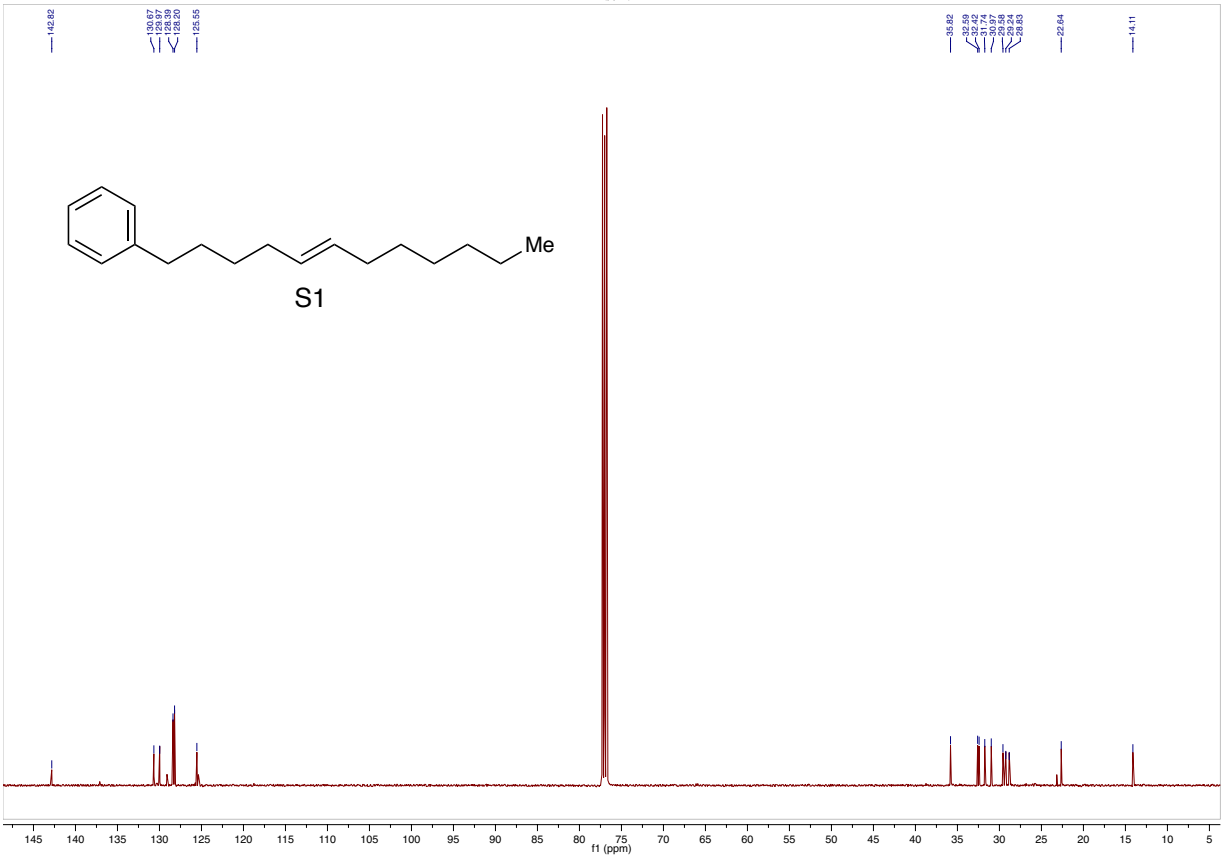

Me
34
f1 (ppm)

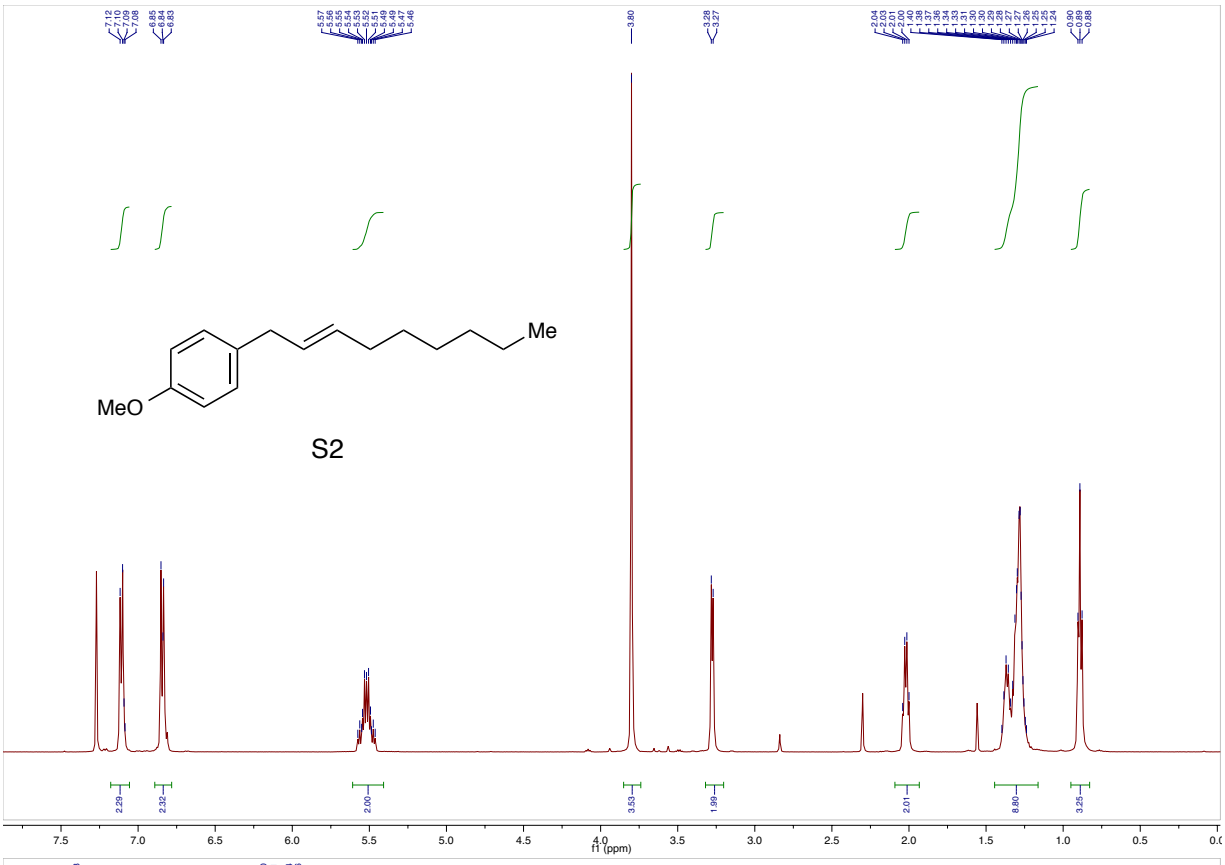

Me
34
f1 (ppm)

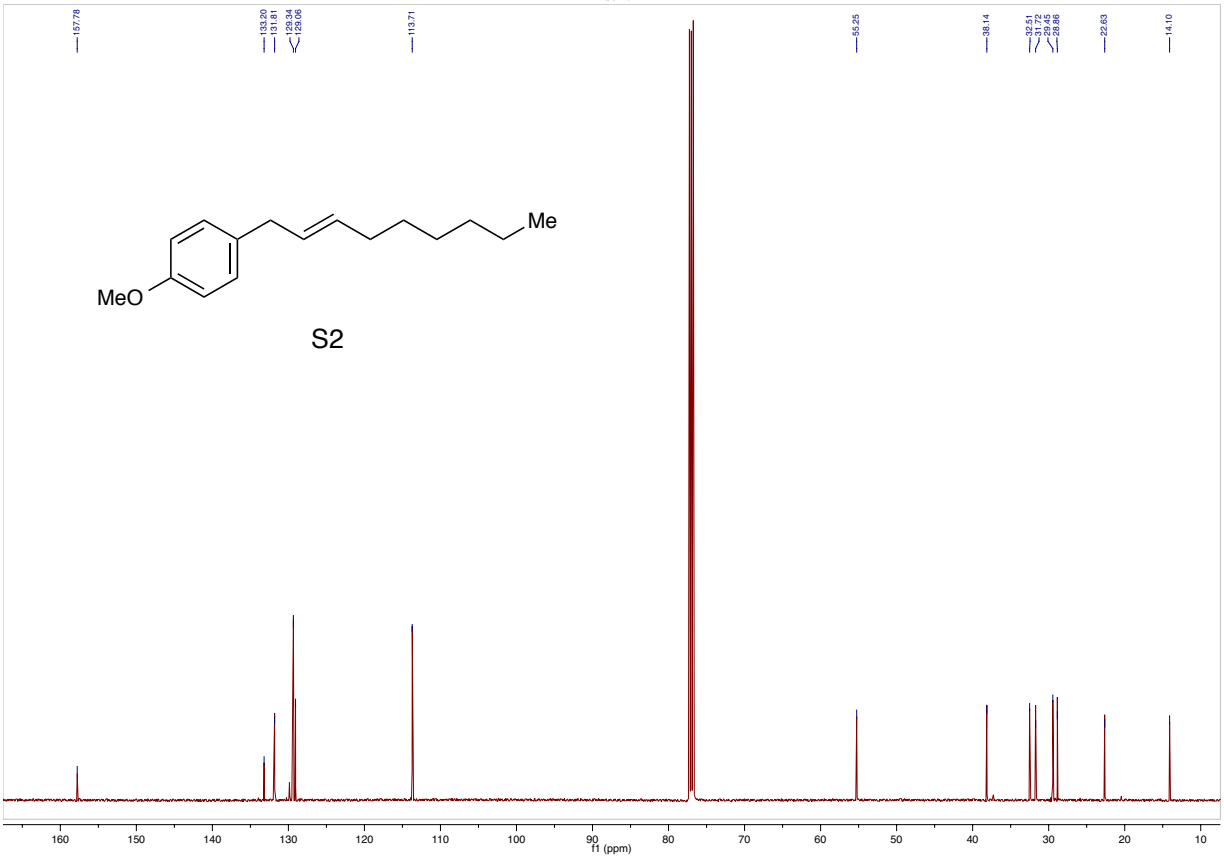

O
Me
Me
Me
36
f1 (ppm)

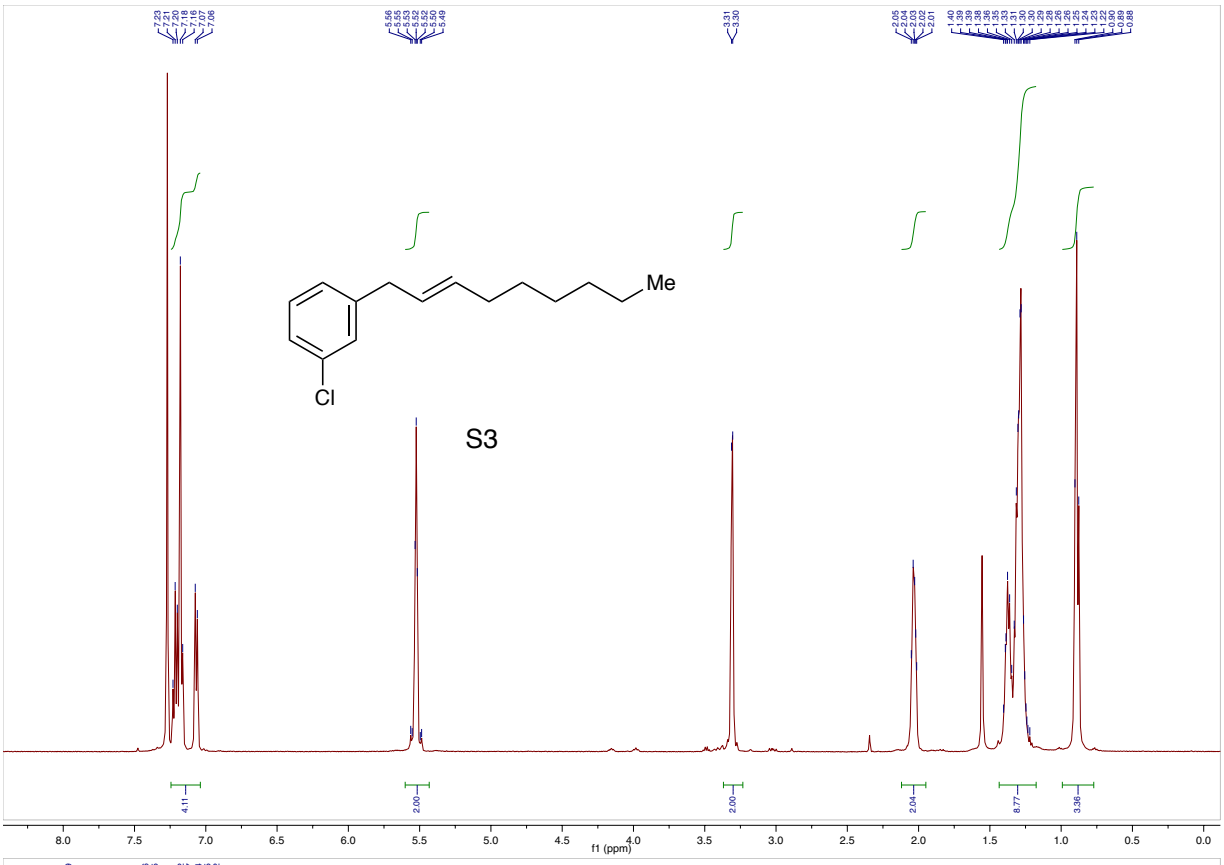

O
Me
Me
Me
36
f1 (ppm)

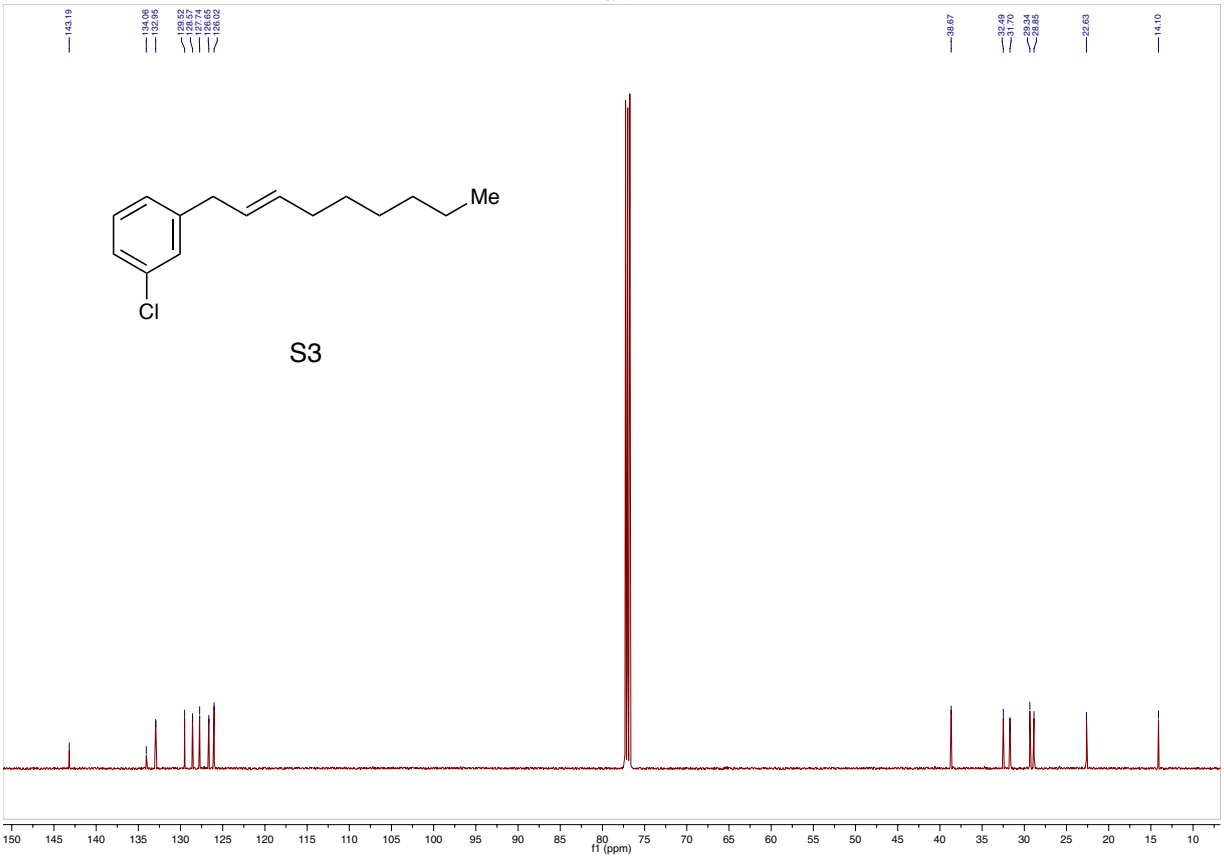

Me
S1
f1 (ppm)


Me
S1
f1 (ppm)


Me
MeO
S2
f1 (ppm)


Me
MeO
S2
f1 (ppm)


Me
Cl
S3
f1 (ppm)


Me
Cl
S3
f1 (ppm)

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