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1. General method

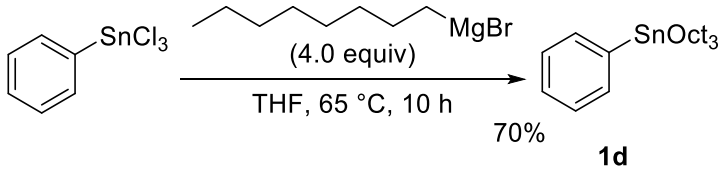
All anaerobic and moisture-sensitive manipulations were carried out with standard Schlenk techniques under dry nitrogen gas. NMR spectra were recorded on a Bruker AV 500MHz NMR, BBI probe (500 MHz for ^{1}H, 125 MHz for ^{13}C), Bruker AV 400MHz NMR, QNP probe (400 MHz for ^{1}H, 100 MHz for ^{13}C, 61 MHz for ^{2}H), Bruker AVIII 400MHz NMR, BBFO Probe (400 MHz for ^{1}H, 100 MHz for ^{13}C), JEOL ECA 400 MHz NMR (400 MHz for ^{1}H, 100 MHz for ^{13}C), or Bruker AV 300MHz NMR, QNP probe (300 MHz for ^{1}H, 75 MHz for ^{13}C). Chemical shifts are reported in δ (ppm) referenced to the residual peaks of $CDCl_3$ (δ 7.26) for ^{1}H NMR, $CDCl_3$ (δ 77.00) for ^{13}C NMR and acetone (δ 2.05) for ^{2}H NMR. The following abbreviations are used to describe the multiplicities; s: singlet, d: doublet, t: triplet, q: quartet, quint: quintet, sext: sextet, m: multiplet, br: broad. High resolution mass spectra (HRMS) were obtained with a Waters Q-Tof Premier mass spectrometer. For thin layer chromatography (TLC), Merck pre-coated TLC plates (Merck 60 F254) were used, and compounds were visualized with a UV light at 254 nm. Further visualization was achieved by basic aqueous $KMnO_4$ solution stain. Flash column chromatography was performed with Silica gel 60 (Merk). The products were further purified by GPC (Gel Permeation Chromatography) if necessary.

2. Materials

Alkynes (**2a**–**2c**, **2e**–**2h**, **2k** and **2l**), aryl halides, bisphosphine ligands, n-BuLi, D_2O, CuI, CuCl, AgOTf, KOH, Mg, $ZnCl_2$, $ZnBr_2$, ZnI_2, cyclohex-2-enone, trifluoroacetic anhydride, tributyltin chloride, $PhSnCl_3$, $PhSnBu_3$ (**1a**), $PhSnMe_3$ (**1b**), 4-$MeOC_6H_4SnBu_3$ (**1i**), 4-$FC_6H_4SnBu_3$ (**1j**), 4-$ClC_6H_4SnBu_3$ (**1k**), 1-chloromethyl-4-fluoro-1,4-diazoniabicyclo[2.2.2]octane bis(tetrafluoroborate), 1-bromooctane, 1-bromopropane, 2-bromo-1,1,2-triphenylethylene, $[IrCl(coe)_2]_2$, $CoCl_2$(xantphos), $PdCl_2(PPh_3)_2$, $RhCl(PPh_3)_3$ and iodine were purchased and used as received. Dioxane and THF were distilled over benzophenone ketyl under N_2.

$[RhCl(coe)_2]_2$,[1] $[RhCl(cod)]_2$,[2] RhPh(binap)(PPh_3) (**5**) [434314-09-7],[3] 4-$BrC_6H_4SnBu_3$ (**1l**) [17151-49-4],[4] 4-$NCC_6H_4SnBu_3$ (**1m**) [79048-30-9],[4] 4-$MeOOCC_6H_4SnBu_3$ (**1n**) [91734-76-8],[5] and but-2-yn-1-ylcyclohexane (**2d**) [57497-07-1][6] were prepared according to the reported procedures.

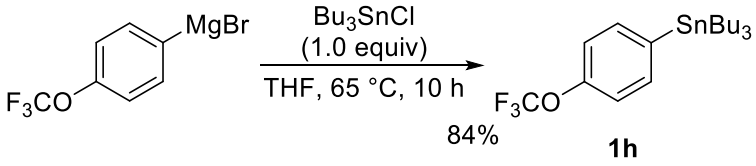
3. Preparation of trioctyl(phenyl)stannane (1d) [143363-50-2]



To a solution of octylmagnesium bromide in THF (25.0 mL), prepared from magnesium turnings (593 mg, 24 mmol) and 1-bromooctane (3.86 g, 20.0 mmol), $PhSnCl_3$ (1.51 g, 5.0 mmol) was added dropwise over 15 min at room temperature. The mixture was stirred at 65 °C for 10 h before H_2O (10.0 mL) was added. The mixture was extracted with ethyl acetate, and the combined organic layer was dried over $MgSO_4$ and concentrated under reduced pressure. The residue was subjected to chromatography on silica gel (pre-treated with 1% Et_3N in hexanes) to give trioctyl(phenyl)stannane (**1d**) [143363-50-2] (70%, 1.87 g, 3.5 mmol) as colorless oil. The spectral data are in agreement with reported literature values.[7]

$PhSnPr_3$ (**1c**) [55335-05-2] was prepared in the same manner as above using propylmagnesium bromide, and the spectral data are in agreement with reported literature values.[8]

4. Preparation of tributyl(4-(trifluoromethoxy)phenyl)stannane (1h) and tributyl(penta-deuterophenyl)stannane (1a-d_5)



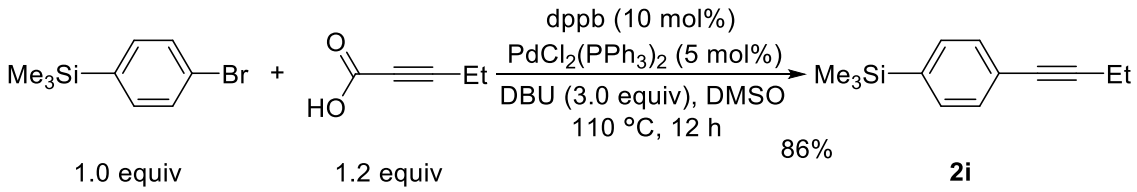
To a solution of 4-(trifluoromethoxy)phenylmagnesium bromide in THF (10.0 mL), prepared from magnesium turnings (288 mg, 12 mmol) and 4-bromotrifluoromethoxybenzene (2.41 g, 10.0 mmol), Bu_3SnCl (3.26 g, 10.0 mmol) was added dropwise over 15 min at room temperature. The mixture was stirred at 65 °C for 10 h before H_2O (5.0 mL) was added. The mixture was extracted with ethyl acetate, and the combined organic layer was dried over $MgSO_4$ and concentrated under reduced pressure. The residue was subjected to chromatography on silica gel (pre-treated with 1% Et_3N in hexanes) to give tributyl(4-(trifluoromethoxy)phenyl)stannane (**1h**) (84%, 3.79 g, 8.4 mmol) as colorless oil. R_f = 0.7 (hexanes). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 0.92 (t, $J_{H,H}$ = 7.3 Hz, 9H), 1.09 (t, $J_{H,H}$ = 8.1 Hz, $J_{H,^{119}Sn}$ = 51.2 Hz, 6H), 1.36 (sext, $J_{H,H}$ = 7.3 Hz, 6H), 1.45–1.65 (m, 6H), 7.19 (d, $J_{H,H}$ = 7.6 Hz, 2H), 7.50 (d, $J_{H,H}$ = 8.1 Hz, $J_{H,^{119}Sn}$ = 35.7 Hz, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 9.7 ($J_{C,^{119}Sn}$ = 343.4 Hz, $J_{C,^{117}Sn}$ = 328.2 Hz), 13.6, 27.4 ($J_{C,^{119}Sn}$ = 55.9 Hz), 29.0 ($J_{C,^{119}Sn}$ = 20.3 Hz), 120.3 ($J_{C,^{119}Sn}$ = 41.7 Hz), 120.6 (q, $J_{C,^{19}F}$ = 256.8

Hz), 137.6 ($J_{C,^{119}Sn}$ = 33.5 Hz), 140.6 ($J_{C,^{119}Sn}$ = 368.7 Hz, $J_{C,^{117}Sn}$ = 352.7 Hz), 149.5 (q, $J_{C,^{19}F}$ = 1.4 Hz). **HRMS** (ESI) calcd for $C_{19}H_{31}OF_3Na^{120}Sn$ $[M+Na]^+$ 475.1247, found 475.1272.

$C_6D_5SnBu_3$ (**1a**-d_5) was prepared in the same manner as above using pentadeuterophenylmagnesium bromide and Bu_3SnCl. Thus, the reaction of a solution of pentadeuterophenylmagnesium bromides in THF (10.0 mL), prepared from magnesium turnings (299 mg, 12 mmol) and pentadeuterophenyl bromide (1.62g, 10.0 mmol), with Bu_3SnCl (3.26 g, 10.0 mmol) at 65 °C for 10 h gave tributyl(pentadeuterophenyl)stannane (**1a**-d_5) (83% yield, 3.09 g, 8.3 mmol) as colorless oil. R_f = 0.9 (hexanes). **^{1}H NMR** (300 MHz, $CDCl_3$) δ 0.93 (t, $J_{H,H}$ = 7.3 Hz, 9H), 1.10 (t, $J_{H,H}$ = 8.1 Hz, $J_{H,^{119}Sn}$ = 51.1 Hz, 6H), 1.37 (sext, $J_{H,H}$ = 7.3 Hz, 6H), 1.44–1.75 (m, 6H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 9.5 ($J_{C,^{119}Sn}$ = 339.6 Hz, $J_{C,^{117}Sn}$ = 324.6 Hz), 13.7, 27.4 ($J_{C,^{119}Sn}$ = 56.6 Hz), 29.1 ($J_{C,^{119}Sn}$ = 20.1 Hz), 127.4 (t, $J_{C,^2H}$ = 24.1 Hz), 136.0 (t, $J_{C,^2H}$ = 24.0 Hz), 141.7. **HRMS** (ESI) calcd for $C_{18}H_{27}D_5Na^{120}Sn$ $[M+Na]^+$ 396.1738, found 396.1733.

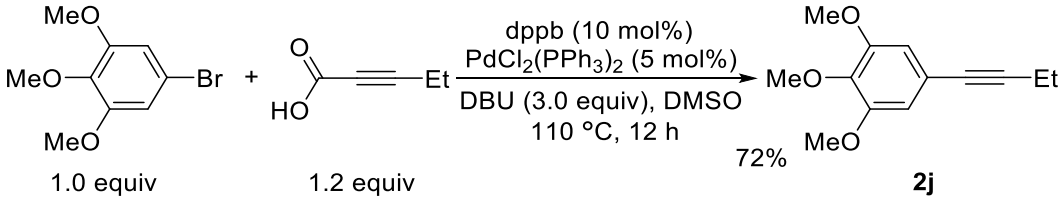
4-$MeC_6H_4SnBu_3$ (**1e**) [31614-66-1],[7] 4-$PhC_6H_4SnBu_3$ (**1f**) [51533-89-2],[4] 4-$Me_3SiC_6H_4SnBu_3$ (**1g**) [38860-01-4],[9] 4-$CF_3C_6H_4SnBu_3$ (**1o**) [86487-19-6],[5] 3-$MeC_6H_4SnBu_3$ (**1p**) [68971-88-0],[5] 3-$Me_3SiC_6H_4SnBu_3$ (**1q**) [1026787-60-9],[10] 3-$CF_3C_6H_4SnBu_3$ (**1r**) [53566-38-4],[11] 2-naphthylSnBu$_3$ (**1s**) [972-11-2],[11] and 2-$MeC_6H_4SnBu_3$ (**1t**) [68971-87-9][5] were prepared in the same manner as above from the corresponding arylmagnesium bromides and Bu_3SnCl. Their spectral data are in agreement with reported literature values.

5. Preparation of 1-(4-trimethylsilylphenyl)but-1-yne (2i) and 1-(3,4,5-trimethoxyphenyl)but-1-yne (2j)



$PdCl_2(PPh_3)_2$ (351.0 mg, 0.50 mmol, 5.0 mol%) and 1,4-bis(diphenylphosphino)butane (426.5 mg, 1.0 mmol, 10.0 mol%) were placed in a 100 mL flask under nitrogen. DMSO (25 mL) was added and the mixture was stirred at room temperature for 10 min. To the mixure, 1-bromo-4-(trimethylsilyl)benzene (2.29 g, 10.0 mmol), 2-pentynoic acid (1.18 g, 12.0 mmol) and DBU (4.58 g, 30.0 mmol) were added. The mixture was heated at 110 °C for 12 h. The mixture was diluted with 20.0 mL of diethyl ether, and it was passed through a short pad of silica gel with ethyl acetate as an eluent. The solution was washed with H_2O and brine, dried over anhydrous $MgSO_4$, and concentrated under reduced pressure. The residue was subjected to chromatography on silica gel to give 1-(4-trimethylsilylphenyl)but-1-yne (**2i**) (86%, 1.74 g, 8.6 mmol) as pale yellow oil. R_f = 0.7 (hexanes). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 0.25 (s, 9H), 1.24 (t, $J_{H,H}$ = 7.5 Hz, 3H), 2.42 (q, $J_{H,H}$ = 7.5 Hz, 2H), 7.36 (d,

$J_{H,H}$ = 8.1 Hz, 2H), 7.43 (d, $J_{H,H}$ = 8.1 Hz, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ –1.2, 13.1, 13.9, 80.0, 92.1, 124.3, 130.6, 133.1, 139.9. **HRMS** (ESI) calcd for $C_{13}H_{19}Si$ $[M+H]^+$ 203.1256, found 203.1218.



The alkyne, 1-(3,4,5-trimethoxyphenyl)but-1-yne (**2j**), was prepared in the same manner as above using 5-bromo-1,2,3-trimethoxybenzene. Thus, the reaction of $PdCl_2(PPh_3)_2$ (351.0 mg, 0.50 mmol, 5.0 mol%), 1,4-bis(diphenylphosphino)butane (426.5 mg, 1.0 mmol, 10.0 mol%), 5-bromo-1,2,3-trimethoxybenzene (2.47 g, 10.0 mmol), 2-pentynoic acid (1.18 g, 12.0 mmol) and DBU (4.58 g, 30.0 mmol) at 110 °C for 12 h gave **2j** (72% yield, 1.59 g, 7.2 mmol) as colorless solid. R_f = 0.1 (hexanes). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 1.24 (t, $J_{H,H}$ = 7.5 Hz, 3H), 2.41 (q, $J_{H,H}$ = 7.5 Hz, 2H), 3.83 (s, 3H), 3.84 (s, 6H), 6.63 (s, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 12.9, 13.8, 55.9, 60.8, 79.7, 90.6, 108.6, 119.0, 138.1, 152.8. **HRMS** (ESI) calcd for $C_{13}H_{17}O_3$ $[M+H]^+$ 221.1178, found 221.1190.

6. A typical procedure for migratory arylstannylation of alkynes catalysed by rhodium complex and zinc chloride (Table 1, entry 1)

$[RhCl(coe)_2]_2$ (3.58 mg, 0.0050 mmol, 5.0 mol% of Rh), binap (6.85 mg, 0.0110 mmol, 5.5 mol%), $ZnCl_2$ (27.3 mg, 0.20 mmol) and $PhSnBu_3$ (**1a**) (146.9 mg, 0.40 mmol) were placed in a 10 mL Schlenk tube under nitrogen. Dioxane (1.0 mL) was added and the mixture was stirred at room temperature for 10 min. Before the tube was sealed, 4-octyne (**2a**) (22.0 mg, 0.20 mmol) was added. The tube was placed in a preheated oil bath at 130 °C and the mixture was stirred for 16 h. The reaction mixture was passed through a short pad of basic aluminum oxide with ethyl acetate as the eluent. The solvent was removed on a rotary evaporator. The residue was subjected to chromatography on silica gel (pre-treated with 1% Et_3N in hexanes) to give (*E*)-4-(2-tributylstannylphenyl)oct-4-ene (**3aa**) (85%, 81.2 mg, 0.17 mmol) as colorless oil.

The procedures shown above in Section 6 for the reaction of $PhSnBu_3$ (**1a**) with 4-octyne (**2a**) (Table 1, entry 1) were used for the reaction of $ArSnR_3$ (**1c**–**1h**, **1j**–**1n** and **1p**–**1t**) with 4-octyne (**2a**) (Table 2, entries 3–8, 10, 11, 13, 15, 17 and 20–24), and that of $PhSnBu_3$ (**1a**) with unfunctionalised alkynes (**2b**–**2l**) (Table 3).

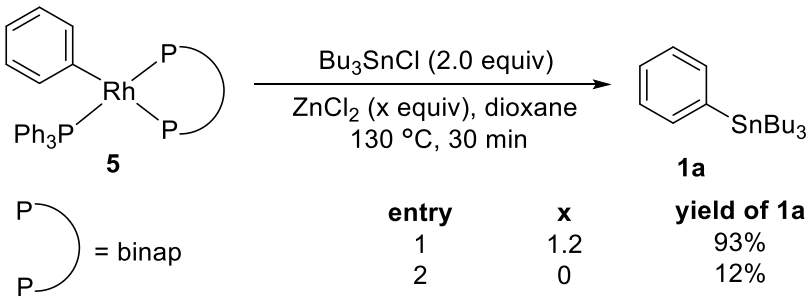
In the reaction of $ArSnR_3$ (**1k**–**1o**) with 4-octyne (**2a**) (Table 2, entries 12, 14, 16, 18 and 19), the procedures shown in Section 6 were modified by use of segphos instead of binap.

In Table 2 entry 2, the procedures shown in Section 6 were modified by using THF as solvent and decreasing the reaction temperature to 90 °C. Thus, the reaction of $[RhCl(coe)_2]_2$ (3.58 mg, 0.0050 mmol,

5.0 mol% of Rh), binap (6.85 mg, 0.0110 mmol, 5.5 mol%), $ZnCl_2$ (27.3 mg, 0.20 mmol), $PhSnMe_3$ (**1b**) (96.4 mg, 0.40 mmol) and 4-octyne (**2a**, 22.0 mg, 0.20 mmol) in THF (1.0 mL) at 90 °C for 16 h gave (*E*)-4-(2-trimethylstannylphenyl)oct-4-ene (**3ba**) (77% yield, 54.1 mg, 0.15 mmol) as colorless oil. The same procedures for the reaction of $PhSnMe_3$ (**1b**) with 4-octyne (**2a**) were also applied to the reaction of 4-$MeOC_6H_4SnBu_3$ (**1i**) with 4-octyne (**2a**) (Table 2, entry 9).

7 Reactions to support the catalytic cycle (Scheme 3)

Scheme 3a

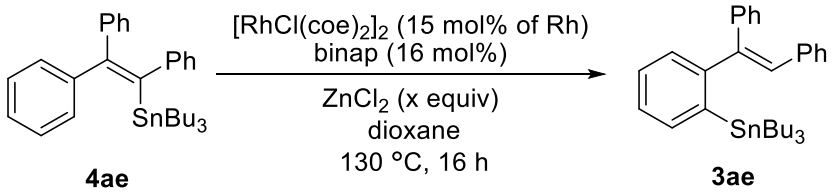


entry	x	yield of 1a
1	1.2	93%
2	0	12%

Scheme 3a, entry 1: In an oven-dried Schlenk tube, $RhPh(PPh_3)$(binap) (**5**) (106.0 mg, 0.10 mmol) and $ZnCl_2$ (16.4 mg, 0.12 mmol) were placed under nitrogen. Dioxane (1.0 mL) and Bu_3SnCl (65.3 mg, 0.20 mmol) were added and the mixture was heated at 130 °C for 30 min. The reaction was quenched with saturated aqueous solution of Na_2CO_3 (1.0 mL). The mixture was extracted with diethyl ether, and the combined organic layer was dried over $MgSO_4$ and concentrated under reduced pressure. The residue was subjected to short chromatography on silica gel (pre-treated with 1% Et_3N in hexanes) to give tributyl(phenyl)stannane (**1a**) (93%, 34.5 mg, 0.09 mmol) as colorless oil. R_f= 0.9 (hexanes). The spectral data are in agreement with reported literature values.[10b]

Scheme 3a, entry 2: The procedures for entry 1 in Scheme 3a were modified by removing $ZnCl_2$. The reaction of $RhPh(PPh_3)$(binap) (107.1 mg, 0.10 mmol) and Bu_3SnCl (66.0 mg, 0.20 mmol) in dioxane (1.0 mL) at 130 °C for 30 min gave tributyl(phenyl)stannane (**1a**) (12%, 4.5 mg, 0.01 mmol) as colorless oil.

Scheme 3b



4ae **3ae**

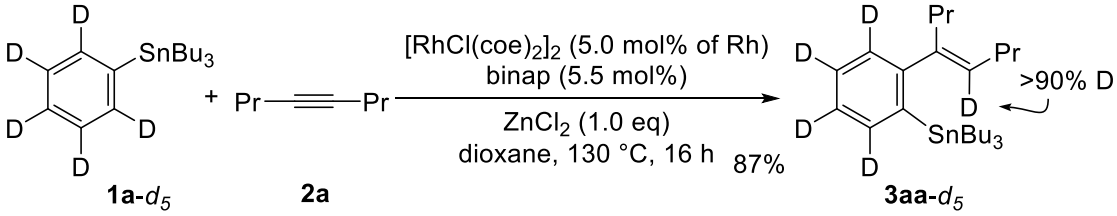
entry	x	yield of 3ae
1	3	16%
2	0	0%

Preparation of compound 4ae: To a solution of 2-bromo-1,1,2-triphenylethylene (3.35 g, 10.0 mmol) in diethyl ether (20.0 mL), n-BuLi (5.0 mL, 10.0 mmol, 2.0 M in cyclohexane) was added dropwise at –78 °C over 30 min. The mixture was allowed to warm to 0 °C and stirred for 2 h. Bu_3SnCl (3.30 g, 10.1 mmol) was added to the mixture at 0 °C. The mixture was stirred at room temperature for 12 h before H_2O (5.0 mL) was added. The mixture was extracted with ethyl acetate, and the combined organic layer was dried over $MgSO_4$ and concentrated under reduced pressure. The residue was subjected to chromatography on silica gel (pre-treated with 1% Et_3N in hexanes) to give **4ae** (88%, 4.78 g, 8.8 mmol) as pale yellow oil. R_f = 0.8 (hexanes). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 0.53 (t, $J_{H,H}$ = 8.2 Hz, $J_{H,^{119}Sn}$ = 50.8 Hz, 6H), 0.80 (t, $J_{H,H}$ = 7.2 Hz, 9H), 1.16 (sext, $J_{H,H}$ = 7.2 Hz, 6H), 1.21–1.32 (m, 6H), 6.87–7.03 (m, 8H), 7.12 (t, $J_{H,H}$ = 7.6 Hz, 2H), 7.28–7.35 (m, 5H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 11.6 ($J_{C,^{119}Sn}$ = 334.2 Hz, $J_{C,^{117}Sn}$ = 319.6 Hz), 13.6, 27.3 ($J_{C,^{119}Sn}$ = 61.6 Hz), 29.0 ($J_{C,^{119}Sn}$ = 19.3 Hz), 124.4, 125.9, 127.1, 127.2, 127.6, 128.0 ($J_{C,^{119}Sn}$ = 15.6 Hz), 128.1, 129.4, 130.0, 142.5 ($J_{C,^{119}Sn}$ = 49.2 Hz), 145.9 ($J_{C,^{119}Sn}$ = 26.0 Hz), 146.2 ($J_{C,^{119}Sn}$ = 25.4 Hz), 148.3, 152.7 ($J_{C,^{119}Sn}$ = 24.7 Hz). **HRMS** (ESI) calcd for $C_{32}H_{42}Na^{120}Sn$ $[M+Na]^+$ 569.2206, found 569.2206.

Scheme 3b, entry 1: In an oven-dried Schlenk tube, $[RhCl(coe)_2]_2$ (10.9 mg, 0.015 mmol, 15 mol% of Rh), binap (19.9 mg, 0.032 mmol, 16 mol%), $ZnCl_2$ (81.5 mg, 0.60 mmol) and compound **4ae** (110.2 mg, 0.20 mmol) were placed under nitrogen. Before the tube was sealed, dioxane (1.0 mL) was added. The reaction mixture was heated at 130 °C for 16 h. The reaction mixture was passed through a short pad of basic aluminum oxide with ethyl acetate as the eluent. The solvent was removed on a rotary evaporator. The residue was subjected to chromatography on silica gel (pre-treated with 1% Et_3N in hexanes) to give **3ae** (16%, 17.6 mg, 0.32 mmol) as colorless oil.

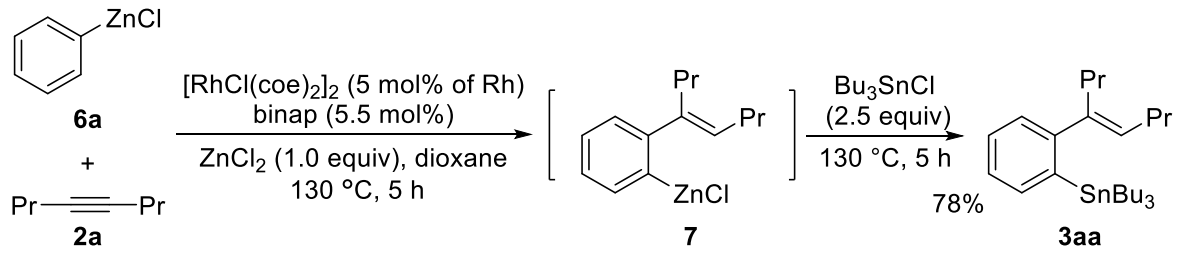
Scheme 3b, entry 2: The procedures for entry 2 in Scheme 3b were modified by removing $ZnCl_2$. The reaction of compound **4ae** (112.2 mg, 0.21 mmol), $[RhCl(coe)_2]_2$ (10.5 mg, 0.015 mmol, 15 mol% of Rh) and binap (19.5 mg, 0.032 mmol, 16 mol%) in dioxane (1.0 mL) at 130 °C for 16 h did not give **3ae**. Compound **3ae** was not detected by ^{1}H NMR or GC.

Scheme 3c



Scheme 3c: The procedures shown in Section 6 for the reaction of $PhSnBu_3$ (**1a**) with 4-octyne (**2a**) (Table 1, entry 1) were used for the reaction of $C_6D_5SnBu_3$ (**1a**-d_5) with 4-octyne (**2a**). Thus, the reaction of $[RhCl(coe)_2]_2$ (3.61 mg, 0.0050 mmol, 5.0 mol% of Rh), binap (6.92 mg, 0.0110 mmol, 5.5 mol%), $ZnCl_2$ (27.6 mg, 0.20 mmol), $C_6D_5SnBu_3$ (**1a**-d_5) (148.9 mg, 0.40 mmol) and 4-octyne (**2a**) (22.3 mg, 0.20 mmol) in dioxane (1.0 mL) at 130 °C for 16 h gave **3aa**-d_5 (87% yield, 83.9 mg, 0.17 mmol) as colorless oil.

Scheme 3d

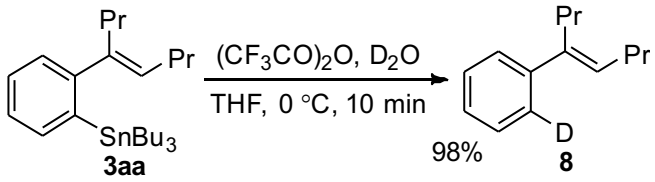


Preparation of PhZnCl (6a): An oven-dried Schlenk flask equipped with a stir bar was charged with a solution of bromobenzene (3.39 g, 22.0 mmol) in THF (12.0 mL). The solution was cooled down to –80 °C and n-BuLi (7.4 mL, 20.0 mmol, 2.69 M in hexanes) was added dropwise over 30 min. Precipitates were formed immediately. The reaction mixture was stirred at –80 °C for 90 min. To the THF solution of phenyllithium thus generated, the THF solution of $ZnCl_2$ (30.0 mL, 30 mmol, 1.0 M) was added dropwise at –80 °C. The reaction mixture was allowed to warm to room temperature to give a solution of phenylzinc chloride (**6a**) (0.4 M) and $ZnCl_2$ (0.2 M) in THF.

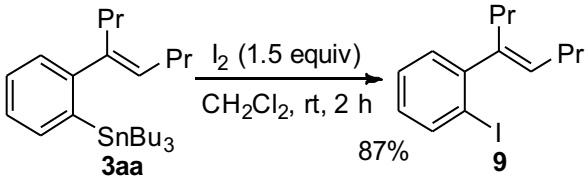
Scheme 3d: In an oven-dried Schlenk tube, $[RhCl(coe)_2]_2$ (3.58 mg, 0.0050 mmol, 5.0 mol% of Rh) and binap (6.85 mg, 0.0110 mmol, 5.5 mol%) were placed under nitrogen. Dioxane (1.2 mL) was added and the mixture was stirred at room temperature for 10 min. To the mixture, 4-octyne (**2a**) (22.3 mg, 0.20 mmol) and 1.0 mL of the THF solution containing phenylzinc chloride (**6a**) (0.40 mmol, 0.4 M) and $ZnCl_2$ (0.4 mmol, 0.2 M), whose preparation is shown above, were added at room temperature. After the mixture was concentrated to *ca.* 1.0 mL under the flow of dry N_2, the reaction mixture was heated at 130 °C for 5 h. Bu_3SnCl (163.1 mg, 0.50 mmol) was added to the mixture at room temperature and the mixture was heated at 130 °C for another 5 h. The reaction mixture was passed through a short pad of basic aluminum oxide with ethyl acetate as the eluent. The solvent was removed on a rotary evaporator.

The residue was subjected to chromatography on silica gel (pre-treated with 1% Et_3N in hexanes) to give (*E*)-4-(2-tributylstannylphenyl)oct-4-ene (**3aa**) (78%, 74.6 mg, 0.16 mmol) as colorless oil.

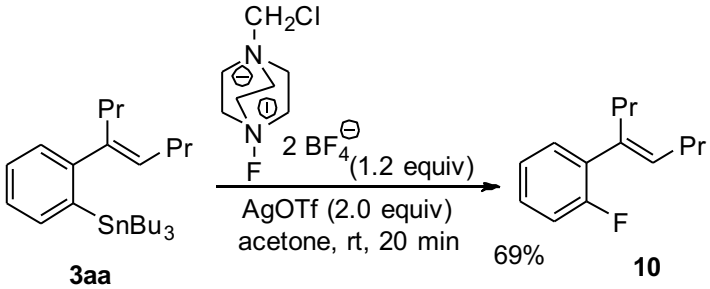
8. Transformations of (*E*)-4-(2-tributylstannylphenyl)oct-4-ene (3aa) (Scheme 4)



Deuterodestannylation of **3aa** was carried out according to a reported procedure.[12] To a mixture of trifluoroacetic anhydride (3.02g, 14.4 mmol) and **3aa** (85.9 mg, 0.18 mmol) in THF (1.0 mL) at 0 °C was added deuterium oxide (0.27 g, 13.5 mmol). The mixture was stired at 0 °C for 10 min and was neutralized by addition of 6 N aqueous sodium hydroxide. The mixture was extracted with Et_2O, and the combined organic layer was dried over $MgSO_4$ and concentrated under reduced pressure. The residue was subjected to chromatography on silica gel to give (*E*)-4-(2-deuteriophenyl)oct-4-ene (**8**) (98%, 33.5 mg, 0.18 mmol) as colorless oil.

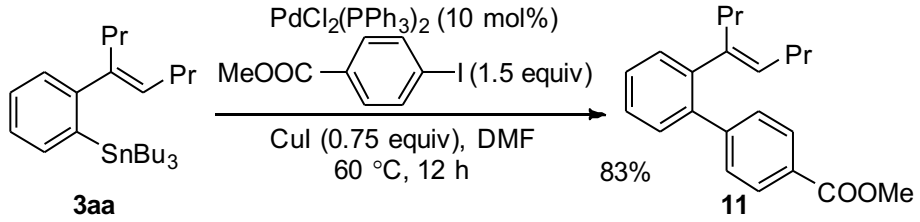


Iododestannylation of **3aa** was carried out according to a reported procedure[11] with minor modifications. An oven-dried flask was charged with **3aa** (114.7 mg, 0.24 mmol), I_2 (91.4 mg, 0.36 mmol) and CH_2Cl_2 (5.0 mL). The mixture was stirred at room temperatue for 2 h before saturated aqueous solution of $Na_2S_2O_3$ (3.0 mL) was added. The mixture was extracted with Et_2O, and the combined organic layer was dried over $MgSO_4$ and concentrated under reduced pressure. The residue was subjected to chromatography on silica gel (pre-treated with 1% Et_3N in hexanes) to give (*E*)-4-(2-iodophenyl)oct-4-ene (**9**) (87%, 65.5 mg, 0.21 mmol) as colorless oil.

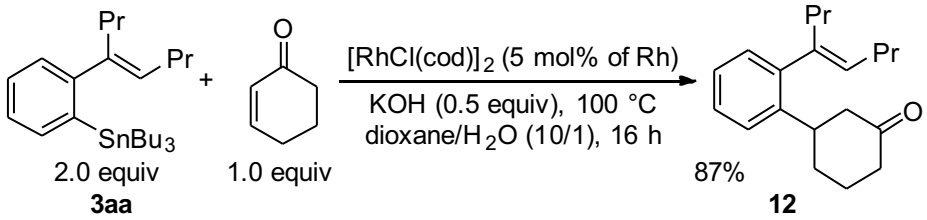


Fluorodestannylation of **3aa** was carried out according to a reported procedure.[13] Under nitrogen at room temperature, to a solution of **3aa** (90.6 mg, 0.19 mmol) in dry acetone (2.0 mL) was added silver triflate (97.6 mg, 0.38 mmol) and 1-chloromethyl-4-fluoro-1,4-diazoniabicyclo[2.2.2]octane

bis(tetrafluoroborate) (80.9 mg, 0.23 mmol). The mixture was stirred at room temperature for 20 min, and it was passed through a short pad of silica gel with ethyl acetate as the eluent. The solvent was removed on a rotary evaporator. The residue was subjected to chromatography on silica gel to give (*E*)-4-(2-fluorophenyl)oct-4-ene (**10**) (69%, 27.1 mg, 0.13 mmol) as colorless oil.

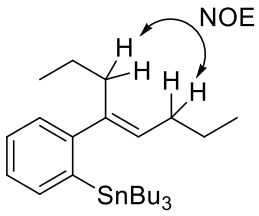


The cross-coupling of **3aa** was performed according to a reported procedure[14] with some modifications. Under nitrogen, **3aa** (95.5 mg, 0.20 mmol), methyl 4-iodobenzoate (78.6 mg, 0.30 mmol), $PdCl_2(PPh_3)_2$ (14.0 mg, 0.020 mmol, 10 mol%) and CuI (28.6 mg, 0.15 mmol) were placed in a 10 mL Schlenk tube. DMF (4.0 mL) was added by syringe and the mixture was allowed to stir at 60 °C for 12 h. The mixture was passed through a short pad of silica gel with ethyl acetate as the eluent. The solvent was removed on a rotary evaporator. The residue was subjected to chromatography on silica gel to give (*E*)-4-(2-(4-methoxycarbonylphenyl)phenyl)oct-4-ene (**11**) (83%, 53.5 mg, 0.17 mmol) as pale yellow oil.



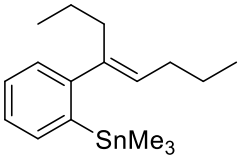
$[RhCl(cod)]_2$ (2.46 mg, 0.0050 mmol, 5 mol% of Rh), **3aa** (190.9 mg, 0.40 mmol), 2-cyclohexenone (19.2 mg, 0.20 mmol) and KOH (5.61 mg, 0.10 mmol) were placed in a 10 mL Schlenk tube under nitrogen. Dioxane (1.0 mL) and water (0.1 mL) were added by syringe, and the mixture was heated at 100 °C for 16 h. The mixture was passed through a short pad of silica gel with ethyl acetate as the eluent. The solvent was removed on a rotary evaporator. The residue was subjected to chromatography on silica gel to give the product **12** (87%, 49.5 mg, 0.17 mmol) as colorless oil.

9. Characterization of the products



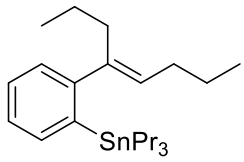
3aa

Compound 3aa. (Table 2, entry 1, 85% yield, colorless oil, R_f = 0.9 (hexanes)). The *E* geometry was assigned by NOESY NMR study. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 0.88 (t, $J_{H,H}$ = 7.3 Hz, 9H), 0.90 (t, $J_{H,H}$ = 7.2 Hz, 3H), 0.97 (t, $J_{H,H}$ = 7.3 Hz, 3H), 0.99 (t, $J_{H,H}$ = 8.0 Hz, $J_{H,^{119}Sn}$ = 51.4 Hz, 6H), 1.32 (sext, $J_{H,H}$ = 7.2 Hz, 6H+2H), 1.42–1.54 (m, 8H), 2.14 (q, $J_{H,H}$ = 7.5 Hz, 2H), 2.32 (t, $J_{H,H}$ = 8.0 Hz, 2H), 5.27 (t, $J_{H,H}$ = 7.0 Hz, 1H), 7.15 (d, $J_{H,H}$ = 7.4 Hz, 1H), 7.18 (td, $J_{H,H}$ = 7.1 Hz, 1.5 Hz, 1H), 7.23 (td, $J_{H,H}$ = 7.3 Hz, 1.6 Hz, 1H), 7.42 (dd, $J_{H,H}$ = 7.0 Hz, 1.2 Hz, $J_{H,^{119}Sn}$ = 42.9 Hz, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 10.9 ($J_{C,^{119}Sn}$ = 339.7 Hz, $J_{C,^{117}Sn}$ = 324.5 Hz), 13.6, 14.1, 14.4, 21.6, 22.9, 27.5 ($J_{C,^{119}Sn}$ = 60.9 Hz), 29.2 ($J_{C,^{119}Sn}$ = 18.9 Hz), 30.6, 34.9, 125.6 ($J_{C,^{119}Sn}$ = 42.4 Hz), 127.4 ($J_{C,^{119}Sn}$ = 9.6 Hz), 127.9 ($J_{C,^{119}Sn}$ = 35.0 Hz), 129.5, 136.9 ($J_{C,^{119}Sn}$ = 32.7 Hz), 140.7 ($J_{C,^{119}Sn}$ = 412.3 Hz, $J_{C,^{117}Sn}$ = 393.8 Hz), 144.3 ($J_{C,^{119}Sn}$ = 14.6 Hz), 152.7 ($J_{C,^{119}Sn}$ = 28.4 Hz). **HRMS** (ESI) calcd for $C_{26}H_{46}Na^{120}Sn$ $[M+Na]^+$ 501.2519, found 501.2537.



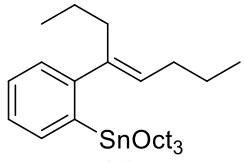
3ba

Compound 3ba. (Table 2, entry 2, 77% yield, colorless oil, R_f = 0.9 (hexanes)). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 0.24 (t, $J_{H,^{119}Sn}$ = 53.0 Hz, 9H), 0.91 (t, $J_{H,H}$ = 7.3 Hz, 3H), 0.97 (t, $J_{H,H}$ = 7.4 Hz, 3H), 1.37 (sext, $J_{H,H}$ = 7.8 Hz, 2H), 1.46 (sext, $J_{H,H}$ = 7.4 Hz, 2H), 2.15 (q, $J_{H,H}$ = 7.4 Hz, 2H), 2.34 (t, $J_{H,H}$ = 8.1 Hz, 2H), 5.31 (t, $J_{H,H}$ = 7.1 Hz, 1H), 7.19 (d, $J_{H,H}$ = 7.5 Hz, 1H), 7.22 (t, $J_{H,H}$ = 6.9 Hz, 1H), 7.24–7.28 (m, 1H), 7.47 (d, $J_{H,H}$ = 6.9 Hz, $J_{H,^{119}Sn}$ = 49.4 Hz, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ –7.4 ($J_{C,^{119}Sn}$ = 350.4 Hz, $J_{C,^{117}Sn}$ = 334.8 Hz), 14.1, 14.5, 21.6, 22.9, 30.5, 34.8, 125.9 ($J_{C,^{119}Sn}$ = 47.8 Hz), 127.6 ($J_{C,^{119}Sn}$ = 38.2 Hz), 127.8 ($J_{C,^{119}Sn}$ = 9.5 Hz), 130.0, 136.3 ($J_{C,^{119}Sn}$ = 38.6 Hz), 141.0 ($J_{C,^{119}Sn}$ = 497.6 Hz, $J_{C,^{117}Sn}$ = 475.1 Hz), 144.2 ($J_{C,^{119}Sn}$ = 16.4 Hz), 152.4 ($J_{C,^{119}Sn}$ = 31.1 Hz). **HRMS** (ESI) calcd for $C_{17}H_{28}Na^{120}Sn$ $[M+Na]^+$ 375.1111, found 375.1118.



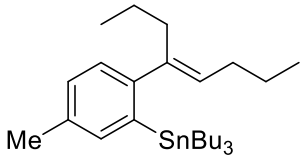
3ca

Compound 3ca. (Table 2, entry 3, 89% yield, colorless oil, R_f = 0.9 (hexanes)). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 0.90 (t, $J_{H,H}$ = 7.3 Hz, 3H), 0.96 (t, $J_{H,H}$ = 7.2 Hz, 9H), 0.98 (t, $J_{H,H}$ = 7.4 Hz, 3H), 1.00 (t, $J_{H,H}$ = 8.3 Hz, $J_{H,^{119}Sn}$ = 50.8 Hz, 6H),1.36 (sext, $J_{H,H}$ = 7.6 Hz, 2H), 1.47 (sext, $J_{H,H}$ = 7.5 Hz, 2H), 1.55 (sext, $J_{H,H}$ = 7.7 Hz, 6H), 2.14 (q, $J_{H,H}$ = 7.4 Hz, 2H), 2.32 (t, $J_{H,H}$ = 8.0 Hz, 2H), 5.27 (t, $J_{H,H}$ = 7.0 Hz, 1H), 7.16 (dd, $J_{H,H}$ = 7.6 Hz, 1.3 Hz, 1H), 7.18 (td, $J_{H,H}$ = 7.1 Hz, 1.6 Hz, 1H), 7.23 (td, $J_{H,H}$ = 7.3 Hz, 1.6 Hz, 1H), 7.43 (dd, $J_{H,H}$ = 7.0 Hz, 1.4 Hz, $J_{H,^{119}Sn}$ = 43.0 Hz, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 13.9 ($J_{C,^{119}Sn}$ = 339.0 Hz, $J_{C,^{117}Sn}$ = 323.8 Hz), 14.1, 14.4, 19.1 ($J_{C,^{119}Sn}$ = 62.6 Hz, $J_{C,^{117}Sn}$ = 59.2 Hz), 20.4 ($J_{C,^{119}Sn}$ = 18.6 Hz), 21.6, 22.9, 30.6, 34.9, 125.6 ($J_{C,^{119}Sn}$ = 41.0 Hz), 127.5 ($J_{C,^{119}Sn}$ = 9.5 Hz), 128.0 ($J_{C,^{119}Sn}$ = 35.1 Hz), 129.5, 136.9 ($J_{C,^{119}Sn}$ = 32.4 Hz), 140.7 ($J_{C,^{119}Sn}$ = 412.6 Hz, $J_{C,^{117}Sn}$ = 394.1 Hz), 144.3 ($J_{C,^{119}Sn}$ = 14.7 Hz), 152.7 ($J_{C,^{119}Sn}$ = 27.7 Hz). **HRMS** (ESI) calcd for $C_{23}H_{40}Na^{120}Sn$ $[M+Na]^+$ 459.2059, found 459.2050.



3da

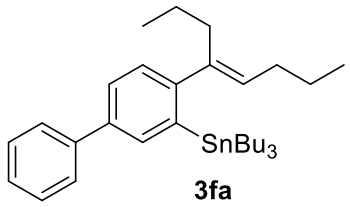
Compound 3da. (Table 2, entry 4, 78% yield, colorless oil, R_f = 0.9 (hexanes)). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 0.88 (t, $J_{H,H}$ = 7.0 Hz, 9H), 0.90 (t, $J_{H,H}$ = 7.2 Hz, 3H), 0.97 (t, $J_{H,H}$ = 7.2 Hz, 3H), 0.98 (t, $J_{H,H}$ = 8.3 Hz, $J_{H,^{119}Sn}$ = 50.7 Hz, 6H), 1.20–1.40 (br m, 32H), 1.41–1.60 (m, 8H), 2.14 (q, $J_{H,H}$ = 7.4 Hz, 2H), 2.31 (t, $J_{H,H}$ = 8.0 Hz, 2H), 5.26 (t, $J_{H,H}$ = 7.0 Hz, 1H), 7.15 (d, $J_{H,H}$ = 7.6 Hz, 1H), 7.17 (td, $J_{H,H}$ = 7.1 Hz, 1.5 Hz, 1H), 7.22 (td, $J_{H,H}$ = 7.3 Hz, 1.6 Hz, 1H), 7.41 (dd, $J_{H,H}$ = 7.0 Hz, 1.3 Hz, $J_{H,^{119}Sn}$ = 42.8 Hz, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 11.2 ($J_{C,^{119}Sn}$ = 339.2 Hz, $J_{C,^{117}Sn}$ = 323.0 Hz), 14.09, 14.13, 14.4, 21.6, 22.7, 23.0, 26.9 ($J_{C,^{119}Sn}$ = 18.6 Hz), 29.2, 29.3, 30.6, 31.9, 34.5 ($J_{C,^{119}Sn}$ = 58.1 Hz), 34.9, 125.6 ($J_{C,^{119}Sn}$ = 41.7 Hz), 127.4, 127.9, 129.5, 136.9 ($J_{C,^{119}Sn}$ = 32.9 Hz), 140.8, 144.3, 152.7. **HRMS** (ESI) calcd for $C_{38}H_{71}{}^{120}Sn$ $[M+H]^+$ 647.4578, found 647.4565.



3ea

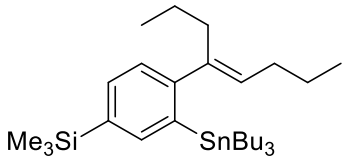
Compound 3ea. (Table 2, entry 5, 87% yield, colorless oil, R_f = 0.9 (hexanes)). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 0.88 (t, $J_{H,H}$ = 7.3 Hz, 9H), 0.89 (t, $J_{H,H}$ = 7.3 Hz, 3H), 0.96 (t, $J_{H,H}$ = 7.3 Hz, 3H), 0.98

(t, $J_{H,H}$ = 7.8 Hz, $J_{H,^{119}Sn}$ = 49.6 Hz, 6H), 1.32 (sext, $J_{H,H}$ = 7.3 Hz, 6H), 1.28–1.40 (m, 2H), 1.41–1.54 (m, 8H), 2.12 (q, $J_{H,H}$ = 7.4 Hz, 2H), 2.30 (t, $J_{H,H}$ = 8.2 Hz, 2H), 2.32 (s, 3H), 5.25 (t, $J_{H,H}$ = 7.0 Hz, 1H), 7.02–7.08 (m, 2H), 7.21 (s, $J_{H,^{119}Sn}$ = 44.3 Hz, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 10.8 ($J_{C,^{119}Sn}$ = 338.7 Hz, $J_{C,^{117}Sn}$ = 323.7 Hz), 13.6, 14.1, 14.4, 21.1, 21.6, 23.0, 27.5 ($J_{C,^{119}Sn}$ = 62.3 Hz, $J_{C,^{117}Sn}$ = 59.4 Hz), 29.2 ($J_{C,^{119}Sn}$ = 18.7 Hz), 30.6, 35.0, 127.7 ($J_{C,^{119}Sn}$ = 37.0 Hz), 128.3, 129.4, 134.8, 137.6, 140.5, 144.1, 149.8. **HRMS** (ESI) calcd for $C_{27}H_{48}Na^{120}Sn$ $[M+Na]^+$ 515.2676, found 515.2684.



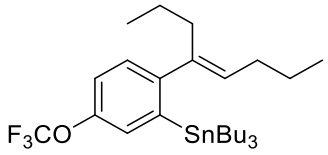
3fa

Compound 3fa. (Table 2, entry 6, 91% yield, colorless oil, R_f = 0.8 (hexanes)). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 0.89 (t, $J_{H,H}$ = 7.3 Hz, 9H), 0.92 (t, $J_{H,H}$ = 7.4 Hz, 3H), 0.98 (t, $J_{H,H}$ = 7.4 Hz, 3H), 1.02 (t, $J_{H,H}$ = 8.3 Hz, $J_{H,^{119}Sn}$ = 47.2 Hz, 6H), 1.33 (sext, $J_{H,H}$ = 7.3 Hz, 6H), 1.32–1.42 (m, 2H), 1.44–1.55 (m, 8H), 2.16 (q, $J_{H,H}$ = 7.4 Hz, 2H), 2.35 (t, $J_{H,H}$ = 8.0 Hz, 2H), 5.32 (t, $J_{H,H}$ = 7.0 Hz, 1H), 7.23 (d, $J_{H,H}$ = 8.0 Hz, $J_{H,^{119}Sn}$ = 16.0 Hz, 1H), 7.32 (t, $J_{H,H}$ = 7.3 Hz, 1H), 7.43 (t, $J_{H,H}$ = 7.7 Hz, 2H), 7.45 (d, $J_{H,H}$ = 8.0 Hz, 1H), 7.58 (d, $J_{H,H}$ = 7.1 Hz, 2H), 7.63 (d, $J_{H,H}$ = 1.9 Hz, $J_{H,^{119}Sn}$ = 43.6 Hz, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 11.0 ($J_{C,^{119}Sn}$ = 339.7 Hz, $J_{C,^{117}Sn}$ = 324.9 Hz), 13.7, 14.1, 14.5, 21.7, 23.0, 27.5 ($J_{C,^{119}Sn}$ = 61.4 Hz), 29.2 ($J_{C,^{119}Sn}$ = 18.7 Hz), 30.6, 34.9, 126.4 ($J_{C,^{119}Sn}$ = 9.7 Hz), 126.9, 127.1, 128.1 ($J_{C,^{119}Sn}$ = 36.1 Hz), 128.7, 129.7, 135.6 ($J_{C,^{119}Sn}$ = 32.9 Hz), 138.2, 141.3, 141.5, 144.0 ($J_{C,^{119}Sn}$ = 13.9 Hz), 151.8. **HRMS** (ESI) calcd for $C_{32}H_{50}Na^{120}Sn$ $[M+Na]^+$ 577.2832, found 577.2823.



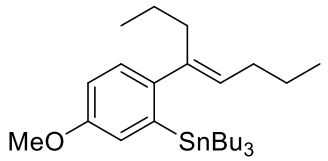
3ga

Compound 3ga. (Table 2, entry 7, 83% yield, colorless oil, R_f = 0.9 (hexanes)). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 0.26 (s, 9H), 0.89 (t, $J_{H,H}$ = 7.2 Hz, 9H+3H), 0.96 (t, $J_{H,H}$ = 7.4 Hz, 3H), 0.99 (t, $J_{H,H}$ = 8.4 Hz, $J_{H,^{119}Sn}$ = 48.8 Hz, 6H), 1.33 (sext, $J_{H,H}$ = 7.3 Hz, 6H+2H), 1.43–1.53 (m, 8H), 2.14 (q, $J_{H,H}$ = 7.4 Hz, 2H), 2.32 (t, $J_{H,H}$ = 8.0 Hz, 2H), 5.27 (t, $J_{H,H}$ = 7.0 Hz, 1H), 7.15 (d, $J_{H,H}$ = 7.5 Hz, $J_{H,^{119}Sn}$ = 15.0 Hz, 1H), 7.39 (dd, $J_{H,H}$ = 7.5 Hz, 1.4 Hz, 1H), 7.58 (s, $J_{H,^{119}Sn}$ = 42.3 Hz, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ –1.0, 11.0 ($J_{C,^{119}Sn}$ = 337.9 Hz, $J_{C,^{117}Sn}$ = 322.1 Hz), 13.7, 14.1, 14.4, 21.7, 22.9, 27.5 ($J_{C,^{119}Sn}$ = 60.1 Hz), 29.2 ($J_{C,^{119}Sn}$ = 19.5 Hz), 30.6, 34.8, 127.2, 129.7, 132.6, 136.7, 139.7, 142.2, 144.3, 153.1. **HRMS** (ESI) calcd for $C_{29}H_{54}NaSi^{120}Sn$ $[M+Na]^+$ 573.2914, found 573.2903.



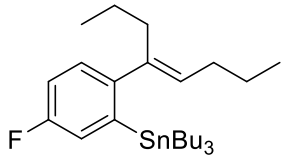
3ha

Compound 3ha. (Table 2, entry 8, 83% yield, colorless oil, R_f = 0.8 (hexanes)). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 0.88 (t, $J_{H,H}$ = 7.2 Hz, 9H), 0.90 (t, $J_{H,H}$ = 7.1 Hz, 3H), 0.97 (t, $J_{H,H}$ = 7.4 Hz, 3H), 1.01 (t, $J_{H,H}$ = 8.2 Hz, $J_{H,^{119}Sn}$ = 49.9 Hz, 6H), 1.32 (sext, $J_{H,H}$ = 7.2 Hz, 6H+2H), 1.43–1.54 (m, 8H), 2.13 (q, $J_{H,H}$ = 7.4 Hz, 2H), 2.30 (t, $J_{H,H}$ = 8.0 Hz, 2H), 5.27 (t, $J_{H,H}$ = 7.1 Hz, 1H), 7.04 (dd, $J_{H,H}$ = 8.4 Hz, 1.4 Hz, 1H), 7.14 (d, $J_{H,H}$ = 8.4 Hz, $J_{H,^{119}Sn}$ = 16.5 Hz, 1H), 7.23 (d, $J_{H,H}$ = 1.5 Hz, $J_{H,^{119}Sn}$ = 43.4 Hz, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 11.0 ($J_{C,^{119}Sn}$ = 343.7 Hz, $J_{C,^{117}Sn}$ = 328.4 Hz), 13.6, 14.1, 14.3, 21.5, 22.9, 27.4 ($J_{C,^{119}Sn}$ = 62.7 Hz, $J_{C,^{117}Sn}$ = 59.2 Hz), 29.0 ($J_{C,^{119}Sn}$ = 19.2 Hz), 30.6, 34.9, 119.7, 120.6 (q, $J_{C,^{19}F}$ = 256.5 Hz), 128.6 ($J_{C,^{119}Sn}$ = 34.4 Hz), 128.9 ($J_{C,^{119}Sn}$ = 37.0 Hz), 130.2, 143.3 ($J_{C,^{119}Sn}$ = 13.1 Hz), 143.4 ($J_{C,^{119}Sn}$ = 380.6 Hz, $J_{C,^{117}Sn}$ = 343.7 Hz), 147.3, 151.2 ($J_{C,^{119}Sn}$ = 25.0 Hz). **HRMS** (ESI) calcd for $C_{27}H_{45}F_3ONa^{120}Sn$ $[M+Na]^+$ 585.2342, found 585.2335.



3ia

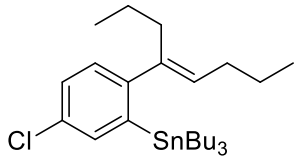
Compound 3ia. (Table 2, entry 9, 65% yield, colorless oil, R_f = 0.8 (hexanes)). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 0.88 (t, $J_{H,H}$ = 7.3 Hz, 9H), 0.89 (t, $J_{H,H}$ = 7.3 Hz, 3H), 0.96 (t, $J_{H,H}$ = 7.4 Hz, 3H), 0.98 (t, $J_{H,H}$ = 8.4 Hz, $J_{H,^{119}Sn}$ = 50.5 Hz, 6H), 1.32 (sext, $J_{H,H}$ = 7.3 Hz, 6H+2H), 1.42–1.52 (m, 8H), 2.11 (q, $J_{H,H}$ = 7.4 Hz, 2H), 2.29 (t, $J_{H,H}$ = 8.0 Hz, 2H), 3.80 (s, 3H), 5.24 (t, $J_{H,H}$ = 7.0 Hz, 1H), 6.76 (dd, $J_{H,H}$ = 8.4 Hz, 2.8 Hz, 1H), 6.97 (d, $J_{H,H}$ = 2.8 Hz, $J_{H,^{119}Sn}$ = 46.6 Hz, 1H), 7.09 (d, $J_{H,H}$ = 8.4 Hz, $J_{H,^{119}Sn}$ = 17.6 Hz, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 10.9 ($J_{C,^{119}Sn}$ = 340.5 Hz, $J_{C,^{117}Sn}$ = 325.5 Hz), 13.6, 14.1, 14.4, 21.6, 23.0, 27.5 ($J_{C,^{119}Sn}$ = 60.6 Hz), 29.1 ($J_{C,^{119}Sn}$ = 18.9 Hz), 30.6, 35.0, 55.1, 112.2, 122.4, 128.6, 129.3, 142.2, 143.8, 145.2, 157.2. **HRMS** (ESI) calcd for $C_{27}H_{48}ONa^{120}Sn$ $[M+Na]^+$ 531.2625, found 531.2617.



3ja

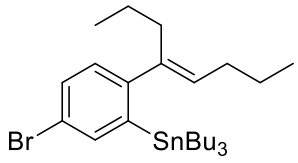
Compound 3ja. (Table 2, entry 10, 77% yield, colorless oil, R_f = 0.8 (hexanes)). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 0.886 (t, $J_{H,H}$ = 7.3 Hz, 9H), 0.894 (t, $J_{H,H}$ = 7.3 Hz, 3H), 0.97 (t, $J_{H,H}$ = 7.4 Hz, 3H), 0.99 (t, $J_{H,H}$ = 8.3 Hz, $J_{H,^{119}Sn}$ = 50.9 Hz, 6H), 1.32 (sext, $J_{H,H}$ = 7.3 Hz, 6H), 1.27–1.37 (m, 2H), 1.41–1.53 (m, 8H), 2.12 (q, $J_{H,H}$ = 7.4 Hz, 2H), 2.28 (t, $J_{H,H}$ = 8.0 Hz, 2H), 5.24 (t, $J_{H,H}$ = 7.0 Hz, 1H), 6.88

(td, $J_{H,H} = J_{H,^{19}F} = 8.6$ Hz, 2.8 Hz, 1H), 7.09 (dd, $J_{H,H} = 2.0$ Hz, $J_{H,^{19}F} = 8.4$ Hz, $J_{H,^{119}Sn} = 43.6$ Hz, 1H), 7.10 (d, $J_{H,H} = 8.3$ Hz, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 10.9 ($J_{C,^{119}Sn} = 343.0$ Hz, $J_{C,^{117}Sn} = 327.7$ Hz), 13.6, 14.1, 14.4, 21.5, 22.9, 27.4 ($J_{C,^{119}Sn} = 61.7$ Hz), 29.1 ($J_{C,^{119}Sn} = 19.1$ Hz), 30.6, 35.0, 114.0 (d, $J_{C,^{19}F} = 20.9$ Hz, $J_{C,^{119}Sn} = 8.7$ Hz), 122.7 (d, $J_{C,^{19}F} = 17.5$ Hz, $J_{C,^{119}Sn} = 34.1$ Hz), 129.2 (d, $J_{C,^{19}F} = 6.5$ Hz, $J_{C,^{119}Sn} = 39.4$ Hz), 129.9, 143.4 ($J_{C,^{119}Sn} = 13.2$ Hz), 143.6 (d, $J_{C,^{19}F} = 2.1$ Hz), 148.4 (d, $J_{C,^{19}F} = 3.1$ Hz, $J_{C,^{119}Sn} = 25.8$ Hz), 161.1 (d, $J_{C,^{19}F} = 248.6$ Hz). **HRMS** (ESI) calcd for $C_{26}H_{45}FNa^{120}Sn$ $[M+Na]^+$ 519.2425, found 519.2407.



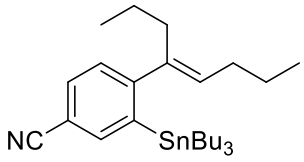
3ka

Compound 3ka. (Table 2, entry 12, 84% yield, colorless oil, $R_f = 0.8$ (hexanes)). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 0.89 (t, $J_{H,H} = 7.3$ Hz, 9H+3H), 0.96 (t, $J_{H,H} = 7.3$ Hz, 3H), 1.00 (t, $J_{H,H} = 8.3$ Hz, $J_{H,^{119}Sn} = 51.7$ Hz, 6H), 1.32 (sext, $J_{H,H} = 7.3$ Hz, 6H+2H), 1.42–1.51 (m, 8H), 2.12 (q, $J_{H,H} = 7.4$ Hz, 2H), 2.28 (t, $J_{H,H} = 8.0$ Hz, 2H), 5.25 (t, $J_{H,H} = 7.0$ Hz, 1H), 7.06 (d, $J_{H,H} = 8.2$ Hz, $J_{H,^{119}Sn} = 16.2$ Hz, 1H), 7.18 (dd, $J_{H,H} = 8.2$ Hz, 2.3 Hz, 1H), 7.34 (d, $J_{H,H} = 2.3$ Hz, $J_{H,^{119}Sn} = 42.1$ Hz, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 10.9 ($J_{C,^{119}Sn} = 342.6$ Hz, $J_{C,^{117}Sn} = 327.6$ Hz), 13.6, 14.1, 14.4, 21.5, 22.9, 27.4 ($J_{C,^{119}Sn} = 62.1$ Hz), 29.1 ($J_{C,^{119}Sn} = 19.0$ Hz), 30.5, 34.8, 127.4 ($J_{C,^{119}Sn} = 8.5$ Hz), 129.2 ($J_{C,^{119}Sn} = 35.7$ Hz), 130.0, 132.0 ($J_{C,^{119}Sn} = 53.4$ Hz), 136.1 ($J_{C,^{119}Sn} = 34.6$ Hz), 143.3($J_{C,^{119}Sn} = 13.0$ H), 143.4, 150.8 ($J_{C,^{119}Sn} = 25.4$ Hz). **HRMS** (ESI) calcd for $C_{26}H_{45}NaCl^{120}Sn$ $[M+Na]^+$ 535.2129, found 535.2140.



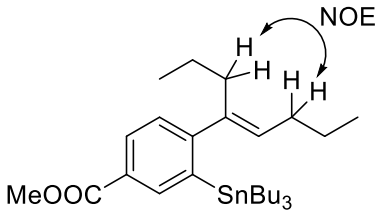
3la

Compound 3la. (Table 2, entry 14, 78% yield, colorless oil, $R_f = 0.8$ (hexanes)). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 0.89 (t, $J_{H,H} = 7.3$ Hz, 9H+3H), 0.96 (t, $J_{H,H} = 7.3$ Hz, 3H), 1.00 (t, $J_{H,H} = 8.3$ Hz, $J_{H,^{119}Sn} = 51.2$ Hz, 6H), 1.32 (sext, $J_{H,H} = 7.3$ Hz, 6H+2H), 1.42–1.51 (m, 8H), 2.12 (q, $J_{H,H} = 7.3$ Hz, 2H), 2.28 (t, $J_{H,H} = 8.0$ Hz, 2H), 5.26 (t, $J_{H,H} = 7.0$ Hz, 1H), 7.00 (d, $J_{H,H} = 8.1$ Hz, $J_{H,^{119}Sn} = 16.1$ Hz, 1H), 7.32 (dd, $J_{H,H} = 8.2$ Hz, 2.2 Hz, 1H), 7.48 (d, $J_{H,H} = 2.1$ Hz, $J_{H,^{119}Sn} = 41.4$ Hz, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 11.0 ($J_{C,^{119}Sn} = 343.0$ Hz, $J_{C,^{117}Sn} = 327.1$ Hz), 13.6, 14.1, 14.4, 21.5, 22.9, 27.4 ($J_{C,^{119}Sn} = 61.5$ Hz), 29.1 ($J_{C,^{119}Sn} = 19.2$ Hz), 30.5, 34.8, 120.7, 129.7, 130.0, 130.3, 139.0, 143.3, 144.1, 151.2. **HRMS** (ESI) calcd for $C_{26}H_{45}NaBr^{120}Sn$ $[M+Na]^+$ 579.1624, found 579.1625.



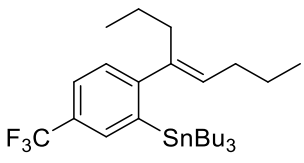
3ma

Compound 3ma. (Table 2, entry 16, 67% yield, colorless oil, R_f = 0.6 (hexanes)). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 0.886 (t, $J_{H,H}$ = 7.3 Hz, 9H), 0.892 (t, $J_{H,H}$ = 7.3 Hz, 3H), 0.97 (t, $J_{H,H}$ = 7.4 Hz, 3H), 1.01 (t, $J_{H,H}$ = 8.3 Hz, $J_{H,^{119}Sn}$ = 51.2 Hz, 6H), 1.31 (sext, $J_{H,H}$ = 7.3 Hz, 6H), 1.25–1.36 (m, 2H), 1.41–1.50 (m, 8H), 2.14 (q, $J_{H,H}$ = 7.4 Hz, 2H), 2.31 (t, $J_{H,H}$ = 8.0 Hz, 2H), 5.28 (t, $J_{H,H}$ = 7.1 Hz, 1H), 7.20 (d, $J_{H,H}$ = 7.9 Hz, $J_{H,^{119}Sn}$ = 14.4 Hz, 1H), 7.50 (dd, $J_{H,H}$ = 7.9 Hz, 1.8 Hz, 1H), 7.66 (d, $J_{H,H}$ = 1.7 Hz, $J_{H,^{119}Sn}$ = 39.2 Hz, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 11.0 ($J_{C,^{119}Sn}$ = 345.3 Hz, $J_{C,^{117}Sn}$ = 329.8 Hz), 13.6, 14.1, 14.3, 21.6, 22.8, 27.3 ($J_{C,^{119}Sn}$ = 62.3 Hz), 29.0 ($J_{C,^{119}Sn}$ = 19.3 Hz), 30.5, 34.5, 109.6 ($J_{C,^{119}Sn}$ = 44.8 Hz), 119.8, 128.2 ($J_{C,^{119}Sn}$ = 30.9 Hz), 131.0, 131.1 ($J_{C,^{119}Sn}$ = 8.5 Hz), 140.3 ($J_{C,^{119}Sn}$ = 35.6 Hz), 142.9, 143.5 ($J_{C,^{119}Sn}$ = 12.2 H), 157.2 ($J_{C,^{119}Sn}$ = 24.8 Hz). **HRMS** (ESI) calcd for $C_{27}H_{45}NNa^{120}Sn$ $[M+Na]^+$ 526.2472, found 526.2480.



3na

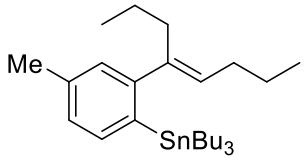
Compound 3na. (Table 2, entry 18, 82% yield, colorless oil, R_f = 0.5 (hexanes)). The *E* geometry was assigned by NOESY NMR study. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 0.88 (t, $J_{H,H}$ = 7.3 Hz, 9H), 0.89 (t, $J_{H,H}$ = 7.3 Hz, 3H), 0.97 (t, $J_{H,H}$ = 7.4 Hz, 3H), 1.02 (t, $J_{H,H}$ = 8.3 Hz, $J_{H,^{119}Sn}$ = 49.9 Hz, 6H), 1.32 (sext, $J_{H,H}$ = 7.3 Hz, 6H+2H), 1.42–1.53 (m, 8H), 2.14 (q, $J_{H,H}$ = 7.4 Hz, 2H), 2.33 (t, $J_{H,H}$ = 8.0 Hz, 2H), 3.90 (s, 3H), 5.30 (t, $J_{H,H}$ = 7.0 Hz, 1H), 7.20 (d, $J_{H,H}$ = 8.0 Hz, $J_{H,^{119}Sn}$ = 15.0 Hz, 1H), 7.87 (dd, $J_{H,H}$ = 8.0 Hz, 1.8 Hz, 1H), 8.09 (d, $J_{H,H}$ = 1.8 Hz, $J_{H,^{119}Sn}$ = 43.2 Hz, 1H); **^{13}C NMR** (75 MHz, $CDCl_3$) δ 10.9 ($J_{C,^{119}Sn}$ = 342.4 Hz, $J_{C,^{117}Sn}$ = 327.3 Hz), 13.6, 14.1, 14.3, 21.6, 22.9, 27.4 ($J_{C,^{119}Sn}$ = 62.3 Hz, $J_{C,^{117}Sn}$ = 59.6 Hz), 29.1 ($J_{C,^{119}Sn}$ = 19.1 Hz), 30.6, 34.6, 51.9, 127.1 ($J_{C,^{119}Sn}$ = 41.3 Hz), 127.8 ($J_{C,^{119}Sn}$ = 32.9 Hz), 128.8 ($J_{C,^{119}Sn}$ = 8.8 Hz), 130.3, 138.1 ($J_{C,^{119}Sn}$ = 35.4 Hz), 141.2, 143.9 ($J_{C,^{119}Sn}$ = 13.1 Hz), 157.5 ($J_{C,^{119}Sn}$ = 27.5 Hz), 167.6. **HRMS** (ESI) calcd for $C_{28}H_{48}O_2Na^{120}Sn$ $[M+Na]^+$ 559.2574, found 559.2605.



3oa

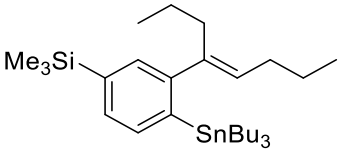
Compound 3oa. (Table 2, entry 19, 69% yield, colorless oil, R_f = 0.8 (hexanes)). **^{1}H NMR** (400

MHz, $CDCl_3$) δ 0.89 (t, $J_{H,H}$ = 7.3 Hz, 9H), 0.90 (t, $J_{H,H}$ = 7.2 Hz, 3H), 0.97 (t, $J_{H,H}$ = 7.3 Hz, 3H), 1.02 (t, $J_{H,H}$ = 8.3 Hz, $J_{H,^{119}Sn}$ = 52.2 Hz, 6H), 1.32 (sext, $J_{H,H}$ = 7.3 Hz, 6H), 1.27–1.37 (m, 2H), 1.42–1.52 (m, 8H), 2.15 (q, $J_{H,H}$ = 7.4 Hz, 2H), 2.32 (t, $J_{H,H}$ = 8.0 Hz, 2H), 5.29 (t, $J_{H,H}$ = 7.1 Hz, 1H), 7.23 (d, $J_{H,H}$ = 7.9 Hz, $J_{H,^{119}Sn}$ = 14.5 Hz, 1H), 7.46 (dd, $J_{H,H}$ = 8.0 Hz, 1.5 Hz, 1H), 7.63 (s, $J_{H,^{119}Sn}$ = 42.4 Hz, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 10.9 ($J_{C,^{119}Sn}$ = 343.6 Hz, $J_{C,^{117}Sn}$ = 328.7 Hz), 13.6, 14.1, 14.4, 21.6, 22.9, 27.4 ($J_{C,^{119}Sn}$ = 62.0 Hz), 29.1 ($J_{C,^{119}Sn}$ = 19.2 Hz), 30.5, 34.7, 124.4 (q, $J_{C,^{19}F}$ = 3.8 Hz), 124.7 (q, $J_{C,^{19}F}$ = 272.2 Hz), 127.5 (q, $J_{C,^{19}F}$ = 31.6 Hz), 127.9, 130.5, 133.2 (q, $J_{C,^{19}F}$ = 3.4 Hz), 142.0, 143.6, 156.2. **HRMS** (ESI) calcd for $C_{27}H_{45}F_3Na^{120}Sn$ $[M+Na]^+$ 569.2393, found 569.2371.



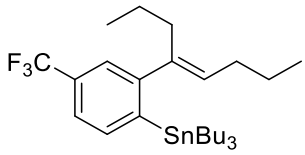
3pa

Compound 3pa. (Table 2, entry 20, 77% yield, colorless oil, R_f = 0.9 (hexanes)). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 0.88 (t, $J_{H,H}$ = 7.3 Hz, 9H+3H), 0.97 (t, $J_{H,H}$ = 8.2 Hz, $J_{H,^{119}Sn}$ = 50.2 Hz, 6H), 0.97 (t, $J_{H,H}$ = 8.2 Hz, 3H), 1.32 (sext, $J_{H,H}$ = 7.4 Hz, 6H), 1.29–1.38 (m, 2H) 1.43–1.53 (m, 8H), 2.13 (q, $J_{H,H}$ = 7.4 Hz, 2H), 2.31 (t, $J_{H,H}$ = 8.2 Hz, 2H), 2.32 (s, 3H), 5.26 (t, $J_{H,H}$ = 7.0 Hz, 1H), 6.99 (s, 1H), 7.02 (d, $J_{H,H}$ = 7.5 Hz, 1H), 7.31 (d, $J_{H,H}$ = 7.4 Hz, $J_{H,^{119}Sn}$ = 42.9 Hz, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 10.8 ($J_{C,^{119}Sn}$ = 339.9 Hz, $J_{C,^{117}Sn}$ = 324.5 Hz), 13.6, 14.1, 14.4, 21.3, 21.7, 23.0, 27.5 ($J_{C,^{119}Sn}$ = 61.0 Hz), 29.2 ($J_{C,^{119}Sn}$ = 18.6 Hz), 30.6, 34.9, 126.6 ($J_{C,^{119}Sn}$ = 44.5 Hz), 128.8 ($J_{C,^{119}Sn}$ = 36.6 Hz), 129.4, 136.7, 136.9, 137.1, 144.4 ($J_{C,^{119}Sn}$ = 14.5 Hz), 152.7 ($J_{C,^{119}Sn}$ = 29.1 Hz). **HRMS** (ESI) calcd for $C_{27}H_{48}Na^{120}Sn$ $[M+Na]^+$ 515.2676, found 515.2698.



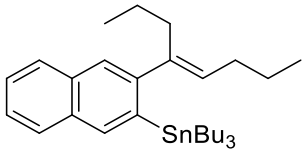
3qa

Compound 3qa. (Table 2, entry 21, 84% yield, colorless oil, R_f = 0.9 (hexanes)). **^{1}H NMR** (500 MHz, $CDCl_3$) δ 0.27 (s, 9H), 0.90 (t, $J_{H,H}$ = 7.3 Hz, 9H), 0.92 (t, $J_{H,H}$ = 7.4 Hz, 3H), 0.987 (t, $J_{H,H}$ = 7.2 Hz, 3H), 0.993 (t, $J_{H,H}$ = 8.4 Hz, $J_{H,^{119}Sn}$ = 50.7 Hz, 6H), 1.33 (sext, $J_{H,H}$ = 7.3 Hz, 6H), 1.33–1.41 (m, 2H), 1.44–1.52 (m, 8H), 2.15 (q, $J_{H,H}$ = 7.4 Hz, 2H), 2.33 (t, $J_{H,H}$ = 8.0 Hz, 2H), 5.28 (t, $J_{H,H}$ = 7.0 Hz, 1H), 7.28 (s, $J_{H,^{119}Sn}$ = 16.4 Hz, 1H), 7.34 (d, $J_{H,H}$ = 7.2 Hz, 1H), 7.43 (d, $J_{H,H}$ = 7.1 Hz, $J_{H,^{119}Sn}$ = 43.0 Hz, 1H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ –1.1, 10.8 ($J_{C,^{119}Sn}$ = 339.0 Hz, $J_{C,^{117}Sn}$ = 323.9 Hz), 13.6, 14.2, 14.4, 21.6, 23.0, 27.5 ($J_{C,^{119}Sn}$ = 60.4 Hz), 29.2 ($J_{C,^{119}Sn}$ = 18.5 Hz), 30.6, 35.0, 129.5, 130.5, 132.6, 136.3, 139.1, 141.6, 144.5, 151.8. **HRMS** (ESI) calcd for $C_{29}H_{54}NaSi^{120}Sn$ $[M+Na]^+$ 573.2914, found 573.2917.



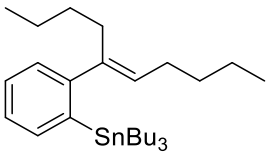
3ra

Compound 3ra. (Table 2, entry 22, 67% yield, colorless oil, R_f = 0.9 (hexanes)). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 0.89 (t, $J_{H,H}$ = 7.3 Hz, 9H), 0.91 (t, $J_{H,H}$ = 7.4 Hz, 3H), 0.98 (t, $J_{H,H}$ = 7.4 Hz, 3H), 1.02 (t, $J_{H,H}$ = 8.2 Hz, $J_{H,^{119}Sn}$ = 50.0 Hz, 6H), 1.32 (sext, $J_{H,H}$ = 7.3 Hz, 6H), 1.26–1.42 (m, 2H), 1.42–1.51 (m, 8H), 2.15 (q, $J_{H,H}$ = 7.4 Hz, 2H), 2.32 (t, $J_{H,H}$ = 8.0 Hz, 2H), 5.29 (t, $J_{H,H}$ = 7.0 Hz, 1H), 7.34 (s, 1H), 7.40 (d, $J_{H,H}$ = 7.6 Hz, 1H), 7.53 (d, $J_{H,H}$ = 7.6 Hz, $J_{H,^{119}Sn}$ = 40.0 Hz, 1H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 10.9 ($J_{C,^{119}Sn}$ = 343.1 Hz, $J_{C,^{117}Sn}$ = 328.1 Hz), 13.6, 14.1, 14.4, 21.6, 22.9, 27.4 ($J_{C,^{119}Sn}$ = 61.7 Hz), 29.1 ($J_{C,^{119}Sn}$ = 19.2 Hz), 30.6, 34.8, 121.8 (q, $J_{C,^{19}F}$ = 3.7 Hz, $J_{C,^{119}Sn}$ = 41.0 Hz), 124.1 (q, $J_{C,^{19}F}$ = 3.6 Hz, $J_{C,^{119}Sn}$ = 34.0 Hz), 124.4 (q, $J_{C,^{19}F}$ = 272.2 Hz), 129.6 (q, $J_{C,^{19}F}$ = 31.8 Hz), 130.6, 137.1 ($J_{C,^{119}Sn}$ = 33.0 Hz), 143.4 ($J_{C,^{119}Sn}$ = 13.0 Hz), 146.3, 153.2 ($J_{C,^{119}Sn}$ = 28.2 Hz). **HRMS** (ESI) calcd for $C_{27}H_{45}F_3Na^{120}Sn$ $[M+Na]^+$ 569.2393, found 569.2393.



3sa

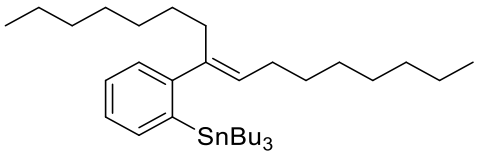
Compound 3sa. (Table 2, entry 23, 83% yield, colorless oil, R_f = 0.9 (hexanes)). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 0.89 (t, $J_{H,H}$ = 7.3 Hz, 9H+3H), 1.00 (t, $J_{H,H}$ = 7.4 Hz, 3H), 1.06 (t, $J_{H,H}$ = 8.3 Hz, $J_{H,^{119}Sn}$ = 50.8 Hz, 6H), 1.34 (sext, $J_{H,H}$ = 7.3 Hz, 6H), 1.25–1.41 (m, 2H), 1.47–1.54 (m, 8H), 2.19 (q, $J_{H,H}$ = 7.4 Hz, 2H), 2.39 (t, $J_{H,H}$ = 7.9 Hz, 2H), 5.38 (t, $J_{H,H}$ = 7.0 Hz, 1H), 7.40–7.45 (m, 2H), 7.56 (s, $J_{H,^{119}Sn}$ = 14.3 Hz, 1H), 7.73–7.80 (m, 2H), 7.89 (s, $J_{H,^{119}Sn}$ = 48.3 Hz, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 11.0 ($J_{C,^{119}Sn}$ = 340.0 Hz, $J_{C,^{117}Sn}$ = 325.2 Hz), 13.7, 14.2, 14.3, 21.6, 23.0, 27.5 ($J_{C,^{119}Sn}$ = 62.1 Hz), 29.2 ($J_{C,^{119}Sn}$ = 19.0 Hz), 30.6, 34.8, 125.2, 125.7, 125.9, 127.3, 127.5, 129.9, 131.7, 133.0, 137.1 ($J_{C,^{119}Sn}$ = 30.6 Hz), 140.0, 144.1($J_{C,^{119}Sn}$ = 11.6 Hz), 149.3. **HRMS** (ESI) calcd for $C_{30}H_{48}Na^{120}Sn$ $[M+Na]^+$ 551.2676, found 551.2660.



3ab

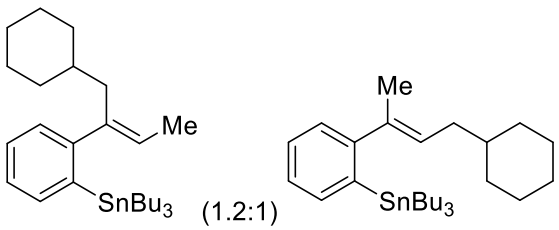
Compound 3ab. (Table 3, 84% yield, colorless oil, R_f = 0.9 (hexanes)). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 0.88 (t, $J_{H,H}$ = 7.0 Hz, 3H), 0.89 (t, $J_{H,H}$ = 7.3 Hz, 9H), 0.94 (t, $J_{H,H}$ = 7.1 Hz, 3H), 0.99 (t, $J_{H,H}$ = 8.3 Hz, $J_{H,^{119}Sn}$ = 50.6 Hz, 6H), 1.28–1.52 (m, 20H), 2.16 (q, $J_{H,H}$ = 7.1 Hz, 2H), 2.34 (t, $J_{H,H}$ = 7.5 Hz,

2H), 5.25 (t, $J_{H,H}$ = 7.0 Hz, 1H), 7.16 (d, $J_{H,H}$ = 7.4 Hz, 1.3 Hz, 1H), 7.19 (td, $J_{H,H}$ = 7.1 Hz, 1.6 Hz, 1H), 7.24 (td, $J_{H,H}$ = 7.3 Hz, 1.7 Hz, 1H), 7.42 (dd, $J_{H,H}$ = 7.0 Hz, 1.3 Hz, $J_{H,^{119}Sn}$ = 43.0 Hz, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 10.9 ($J_{C,^{119}Sn}$ = 339.7 Hz, $J_{C,^{117}Sn}$ = 324.5 Hz), 13.6, 13.9, 14.0, 22.6, 23.1, 27.5 ($J_{C,^{119}Sn}$ = 61.0 Hz), 28.1, 29.2 ($J_{C,^{119}Sn}$ = 18.9 Hz), 30.6, 32.0, 32.5, 125.6 ($J_{C,^{119}Sn}$ = 42.6 Hz), 127.5 ($J_{C,^{119}Sn}$ = 9.5 Hz), 127.9 ($J_{C,^{119}Sn}$ = 34.8 Hz), 129.6, 136.9 ($J_{C,^{119}Sn}$ = 30.5 Hz), 140.7 ($J_{C,^{119}Sn}$ = 413.7 Hz, $J_{C,^{117}Sn}$ = 395.4 Hz), 144.4 ($J_{C,^{119}Sn}$ = 14.4 Hz), 152.7 ($J_{C,^{119}Sn}$ = 28.3 Hz). **HRMS** (ESI) calcd for $C_{28}H_{50}Na^{120}Sn$ $[M+Na]^+$ 529.2832, found 529.2819.



3ac

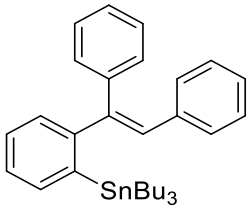
Compound 3ac. (Table 3, 83% yield, colorless oil, R_f = 0.8 (hexanes)). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 0.89 (t, $J_{H,H}$ = 7.2 Hz, 9H), 0.85–0.92 (m, 6H), 0.99 (t, $J_{H,H}$ = 8.3 Hz, $J_{H,^{119}Sn}$ = 50.8 Hz, 6H), 1.18–1.40 (m, 24H), 1.42–1.54 (m, 8H), 2.15 (q, $J_{H,H}$ = 7.2 Hz, 2H), 2.33 (t, $J_{H,H}$ = 7.5 Hz, 2H), 5.25 (t, $J_{H,H}$ = 7.0 Hz, 1H), 7.16 (d, $J_{H,H}$ = 7.4 Hz, 1H), 7.19 (td, $J_{H,H}$ = 7.1 Hz, 1.5 Hz, 1H), 7.24 (td, $J_{H,H}$ = 7.3 Hz, 1.6 Hz, 1H), 7.42 (dd, $J_{H,H}$ = 7.0 Hz, 1.5 Hz, $J_{H,^{119}Sn}$ = 42.8 Hz, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 10.9 ($J_{C,^{119}Sn}$ = 339.6 Hz, $J_{C,^{117}Sn}$ = 324.6 Hz), 13.7, 14.06, 14.09, 22.6, 22.7, 27.5 ($J_{C,^{119}Sn}$ = 62.1 Hz), 28.5, 29.16, 29.17, 29.3, 29.6, 29.8, 30.0, 31.9, 32.8, 125.6 ($J_{C,^{119}Sn}$ = 42.3 Hz), 127.4 ($J_{C,^{119}Sn}$ = 9.4 Hz), 127.9 ($J_{C,^{119}Sn}$ = 34.9 Hz), 129.6, 136.9 ($J_{C,^{119}Sn}$ = 32.4 Hz), 140.7 ($J_{C,^{119}Sn}$ = 414.0 Hz, $J_{C,^{117}Sn}$ = 395.7 Hz), 144.4 ($J_{C,^{119}Sn}$ = 14.5 Hz), 152.7 ($J_{C,^{119}Sn}$ = 28.1 Hz). **HRMS** (ESI) calcd for $C_{34}H_{62}Na^{120}Sn$ $[M+Na]^+$ 613.3771, found 613.3762.



3ad **3ad'**

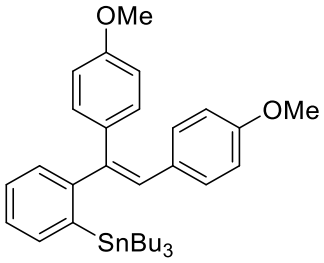
Compound 3ad:3ad' (1.2:1). (Table 3, 84% yield, colorless oil, R_f = 0.9 (hexanes)). **^{1}H NMR** (400 MHz, $CDCl_3$) for **3ad** δ 0.88 (t, $J_{H,H}$ = 7.3 Hz, 9H), 0.83–0.91 (m, 2H), 0.98 (t, $J_{H,H}$ = 8.3 Hz, 6H), 1.32 (sext, $J_{H,H}$ = 7.3 Hz, 6H), 1.10–1.41 (m, 4H), 1.43–1.60 (m, 6H), 1.73 (d, $J_{H,H}$ = 6.8 Hz, 3H), 1.60–1.83 (m, 5H), 2.22 (d, $J_{H,H}$ = 7.1 Hz, 2H), 5.39 (q, $J_{H,H}$ = 6.8 Hz, 1H), 7.10 (dd, $J_{H,H}$ = 7.4 Hz, 1.3 Hz, 1H), 7.16–7.29 (m, 2H), 7.41 (dd, $J_{H,H}$ = 6.9 Hz, 1.6 Hz, 1H); **3ad'** δ 0.89 (t, $J_{H,H}$ = 7.3 Hz, 9H), 0.83–0.91 (m, 2H), 0.97 (t, $J_{H,H}$ = 8.3 Hz, 6H), 1.32 (sext, $J_{H,H}$ = 7.3 Hz, 6H), 1.10–1.40 (m, 4H), 1.43–1.60 (m, 6H), 1.60–1.83 (m, 5H), 1.96 (s, 3H), 2.03 (t, $J_{H,H}$ = 7.0 Hz, 2H), 5.31 (td, $J_{H,H}$ = 7.3 Hz, 1.4 Hz, 1H), 7.16–7.29 (m, 3H), 7.43 (dd, $J_{H,H}$ = 7.0 Hz, 1.4 Hz, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) for **3ad** δ

10.7 ($J_{C,^{119}Sn}$ = 339.4 Hz, $J_{C,^{117}Sn}$ = 323.9 Hz), 13.6, 14.1, 26.3, 26.6, 27.5 ($J_{C,^{119}Sn}$ = 60.7 Hz), 29.2 ($J_{C,^{119}Sn}$ = 19.0 Hz), 33.6, 36.4, 38.4, 125.5, 127.0, 127.5 ($J_{C,^{119}Sn}$ = 10.0 Hz), 128.2, 136.8, 140.5, 143.7, 153.2; **3ad'** δ 10.8 ($J_{C,^{119}Sn}$ = 340.9 Hz, $J_{C,^{117}Sn}$ = 326.5 Hz), 13.6, 18.9, 26.4, 26.6, 27.5 ($J_{C,^{119}Sn}$ = 60.7 Hz), 29.2 ($J_{C,^{119}Sn}$ = 19.0 Hz), 33.4, 36.6, 40.1, 124.6, 125.8, 127.6 ($J_{C,^{119}Sn}$ = 10.0 Hz), 128.3, 137.0, 140.4, 140.7, 154.0. **HRMS** (ESI) calcd for $C_{28}H_{48}Na^{120}Sn$ $[M+Na]^+$ 527.2676, found 527.2703.



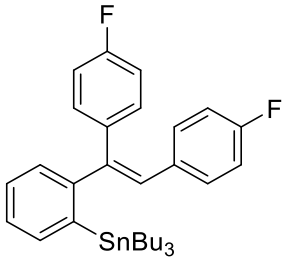
3ae

Compound 3ae. (Table 3, 72% yield, colorless oil, R_f = 0.8 (hexanes)). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 0.83 (t, $J_{H,H}$ = 7.3 Hz, 9H), 0.98 (t, $J_{H,H}$ = 8.3 Hz, $J_{H,^{119}Sn}$ = 50.6 Hz, 6H), 1.27 (sext, $J_{H,H}$ = 7.3 Hz, 6H), 1.4–1.5 (m, 6H), 6.58 (s, 1H), 7.01 (dd, $J_{H,H}$ = 7.6 Hz, 1.0 Hz, 1H), 7.05–7.30 (m, 12H), 7.54 (dd, $J_{H,H}$ = 7.2 Hz, 1.1 Hz, $J_{H,^{119}Sn}$ = 41.6 Hz, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 11.0 ($J_{C,^{119}Sn}$ = 342.2 Hz, $J_{C,^{117}Sn}$ = 327.0 Hz), 13.6, 27.4 ($J_{C,^{119}Sn}$ = 61.6 Hz), 29.2 ($J_{C,^{119}Sn}$ = 18.9 Hz), 126.7, 126.8, 127.5, 127.6 ($J_{C,^{119}Sn}$ = 9.3 Hz), 128.0, 128.2, 129.4, 129.5, 129.6, 130.6, 137.2, 137.4, 140.8, 142.0, 146.3 ($J_{C,^{119}Sn}$ = 14.3 Hz), 152.4 ($J_{C,^{119}Sn}$ = 25.1 Hz). **HRMS** (ESI) calcd for $C_{32}H_{42}Na^{120}Sn$ $[M+Na]^+$ 569.2206, found 569.2222.



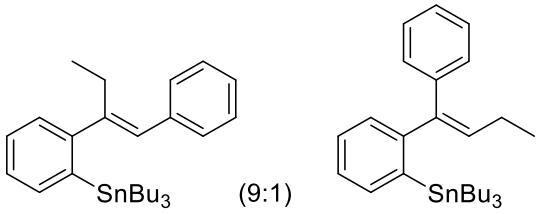
3af

Compound 3af. (Table 3, 78% yield, colorless oil, R_f = 0.3 (hexanes)). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 0.83 (t, $J_{H,H}$ = 7.3 Hz, 9H), 0.96 (t, $J_{H,H}$ = 7.3 Hz, $J_{H,^{119}Sn}$ = 50.6 Hz, 6H), 1.26 (sext, $J_{H,H}$ = 7.3 Hz, 6H), 1.40–1.48 (m, 6H), 3.78 (s, 3H), 3.81 (s, 3H), 6.43 (s, 1H), 6.72 (d, $J_{H,H}$ = 8.8 Hz, 2H), 6.78 (d, $J_{H,H}$ = 8.8 Hz, 2H), 7.00 (dd, $J_{H,H}$ = 7.6 Hz, 1.1 Hz, 1H), 7.06 (d, $J_{H,H}$ = 8.6 Hz, 2H), 7.11 (d, $J_{H,H}$ = 8.8 Hz, 2H), 7.19 (td, $J_{H,H}$ = 7.4 Hz, 1.5 Hz, 1H), 7.24 (td, $J_{H,H}$ = 7.2 Hz, 1.4 Hz, 1H), 7.52 (dd, $J_{H,H}$ = 7.2 Hz, 1.1 Hz, $J_{H,^{119}Sn}$ = 41.7 Hz, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 11.0 ($J_{C,^{119}Sn}$ = 341.9 Hz, $J_{C,^{117}Sn}$ = 326.7 Hz), 13.6, 27.5 ($J_{C,^{119}Sn}$ = 61.7 Hz), 29.2 ($J_{C,^{119}Sn}$ = 18.9 Hz), 55.1, 55.2, 113.4, 113.6, 126.5, 127.5 ($J_{C,^{119}Sn}$ = 9.4 Hz), 128.3, 129.5, 130.2, 130.6, 131.8, 133.3, 137.3 ($J_{C,^{119}Sn}$ = 32.1 Hz), 142.0, 144.1 ($J_{C,^{119}Sn}$ = 14.6 Hz), 152.8, 158.3, 158.9. **HRMS** (ESI) calcd for $C_{34}H_{46}O_2Na^{120}Sn$ $[M+Na]^+$ 629.2417, found 629.2411.



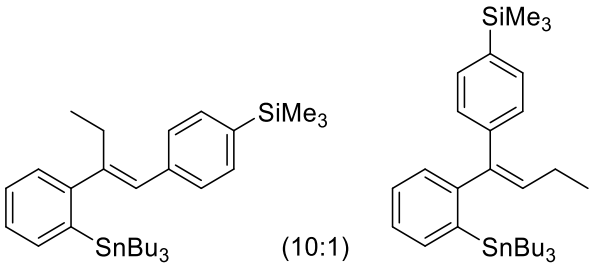
3ag

Compound 3ag. (Table 3, 74% yield, colorless solid, R_f = 0.7 (hexanes)). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 0.83 (t, $J_{H,H}$ = 7.3 Hz, 9H), 0.96 (t, $J_{H,H}$ = 8.3 Hz, $J_{H,^{119}Sn}$ = 52.1 Hz, 6H), 1.26 (sext, $J_{H,H}$ = 7.3 Hz, 6H), 1.40–1.48 (m, 6H), 6.51 (s, 1H), 6.88 (t, $J_{H,H}$ = $J_{H,^{19}F}$ = 8.7 Hz, 2H), 6.94 (t, $J_{H,H}$ = $J_{H,^{19}F}$ = 8.7 Hz, 2H), 6.97 (dd, $J_{H,H}$ = 7.1 Hz, 1.0 Hz, 1H), 7.06 (dd, $J_{H,H}$ = 8.6 Hz, $J_{H,^{19}F}$ = 5.5 Hz, 2H), 7.13 (dd, $J_{H,H}$ = 8.7 Hz, $J_{H,^{19}F}$ = 5.5 Hz, 2H), 7.20 (td, $J_{H,H}$ = 7.5 Hz, 1.5 Hz, 1H), 7.27 (td, $J_{H,H}$ = 7.3 Hz, 1.4 Hz, 1H), 7.54 (dd, $J_{H,H}$ = 7.2 Hz, 1.2 Hz, $J_{H,^{119}Sn}$ = 41.5 Hz, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 11.1 ($J_{C,^{119}Sn}$ = 343.4 Hz, $J_{C,^{117}Sn}$ = 326.9 Hz), 13.6, 27.4 ($J_{C,^{119}Sn}$ = 61.8 Hz), 29.1 ($J_{C,^{119}Sn}$ = 18.9 Hz), 115.2 (d, $J_{C,^{19}F}$ = 21.3 Hz), 115.4 (d, $J_{C,^{19}F}$ = 21.4 Hz), 126.9, 127.7, 128.4, 129.5, 130.9 (d, $J_{C,^{19}F}$ = 7.9 Hz), 132.3 (d, $J_{C,^{19}F}$ = 7.9 Hz), 133.2 (d, $J_{C,^{19}F}$ = 3.2 Hz), 136.4 (d, $J_{C,^{19}F}$ = 2.6 Hz), 137.5, 142.0, 145.1, 151.9, 161.6 (d, $J_{C,^{19}F}$ = 247.4 Hz), 162.3 (d, $J_{C,^{19}F}$ = 247.7 Hz). **HRMS** (ESI) calcd for $C_{32}H_{40}F_2Na^{120}Sn$ $[M+Na]^+$ 605.2018, found 605.2023. The structure of compound **3ag** was confirmed by single crystal X-ray diffraction (Section 9).



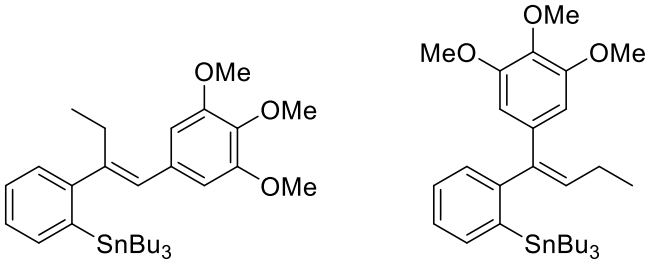
3ah **3ah'**

Compound 3ah:3ah' (9:1). (Table 3, 73% yield, colorless oil, R_f = 0.8 (hexanes)). **^{1}H NMR** (400 MHz, $CDCl_3$) for **3ah** δ 0.83 (t, $J_{H,H}$ = 7.3 Hz, 9H), 1.00 (t, $J_{H,H}$ = 8.4 Hz, 6H), 1.04 (t, $J_{H,H}$ = 7.8 Hz, 3H), 1.26 (sext, $J_{H,H}$ = 7.3 Hz, 6H), 1.40–1.50 (m, 6H), 2.68 (q, $J_{H,H}$ = 7.5 Hz, 2H), 6.33 (s, 1H), 7.15–7.38 (m, 8H), 7.49 (d, $J_{H,H}$ = 7.0 Hz, $J_{H,^{119}Sn}$ = 42.7 Hz, 1H), (**3ah'** δ 1.12 (t, $J_{H,H}$ = 7.5 Hz, 3H), 2.31 (quint, $J_{H,H}$ = 7.5 Hz, 2H), 5.63 (t, $J_{H,H}$ = 7.5 Hz, 1H)); **^{13}C NMR** (100 MHz, $CDCl_3$) for **3ah** δ 11.0 ($J_{C,^{119}Sn}$ = 338.4 Hz, $J_{C,^{117}Sn}$ = 323.7 Hz), 12.9, 13.6, 25.9, 27.4 ($J_{C,^{119}Sn}$ = 61.2 Hz), 29.1 ($J_{C,^{119}Sn}$ = 18.8 Hz), 126.2, 126.5, 127.4, 127.6, 128.2, 128.7, 129.9, 137.3, 137.8, 141.1, 148.4, 151.7. **HRMS** (ESI) calcd for $C_{26}H_{46}Na^{120}Sn$ $[M+Na]^+$ 521.2206, found 521.2206.



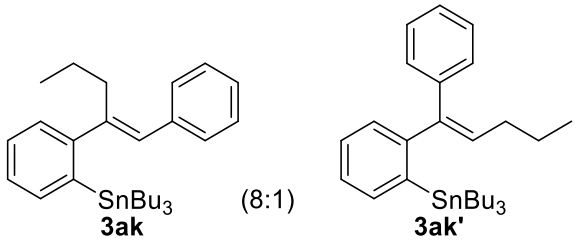
3ai **3ai'**

Compound 3ai:3ai' (10:1). (Table 3, 77% yield, colorless oil, R_f = 0.8 (hexanes)). **^{1}H NMR** (400 MHz, $CDCl_3$) for **3ai** δ 0.29 (s, 9H), 0.83 (t, $J_{H,H}$ = 7.3 Hz, 9H), 1.01 (t, $J_{H,H}$ = 8.4 Hz, $J_{H,^{119}Sn}$ = 50.3 Hz, 6H), 1.05 (t, $J_{H,H}$ = 7.5 Hz, 3H), 1.28 (sext, $J_{H,H}$ = 7.3 Hz, 6H), 1.41–1.50 (m, 6H), 2.70 (q, $J_{H,H}$ = 7.5 Hz, 2H), 6.33 (s, 1H), 7.23–7.30 (m, 3H), 7.32 (d, $J_{H,H}$ = 7.8 Hz, 2H), 7.50 (d, $J_{H,H}$ = 7.0 Hz, $J_{H,^{119}Sn}$ = 41.6 Hz, 1H), 7.53 (d, $J_{H,H}$ = 8.0 Hz, 2H), (**3ai'** δ 0.26 (s, 9H), 1.13 (t, $J_{H,H}$ = 7.5 Hz, 3H), 2.34 (quint, $J_{H,H}$ = 7.5 Hz, 2H), 5.63 (t, $J_{H,H}$ = 7.5 Hz, 1H)); **^{13}C NMR** (100 MHz, $CDCl_3$) for **3ai** δ –1.1, 11.1 ($J_{C,^{119}Sn}$ = 339.9 Hz, $J_{C,^{117}Sn}$ = 325.0 Hz), 12.9, 13.6, 26.0, 27.4 ($J_{C,^{119}Sn}$ = 61.9 Hz), 29.1 ($J_{C,^{119}Sn}$ = 19.0 Hz), 126.2 ($J_{C,^{119}Sn}$ = 40.5 Hz), 127.4, 127.6 ($J_{C,^{119}Sn}$ = 9.5 Hz), 128.0, 128.7, 133.3, 137.3 ($J_{C,^{119}Sn}$ = 31.6 Hz), 138.2, 138.5, 141.1, 148.6 ($J_{C,^{119}Sn}$ = 13.9 Hz), 151.7. **HRMS** (ESI) calcd for $C_{31}H_{50}NaSi^{120}Sn$ $[M+Na]^+$ 593.2601, found 593.2617.



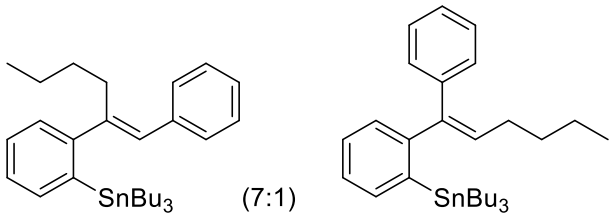
3aj **3aj'**

Compound 3aj:3aj' (17:1). (Table 3, 76% yield, colorless oil, R_f = 0.3 (hexanes)). **^{1}H NMR** (400 MHz, $CDCl_3$) for **3aj** δ 0.84 (t, $J_{H,H}$ = 7.3 Hz, 9H), 1.02 (t, $J_{H,H}$ = 8.4 Hz, 6H), 1.07 (t, $J_{H,H}$ = 7.5 Hz, 3H), 1.29 (sext, $J_{H,H}$ = 7.3 Hz, 6H), 1.43–1.51 (m, 6H), 2.71 (q, $J_{H,H}$ = 7.6 Hz, 2H), 3.87 (s, 6H), 3.88 (s, 3H), 6.27 (s, 1H), 6.57 (s, 2H), 7.20–7.37 (m, 3H), 7.49 (d, $J_{H,H}$ = 7.0 Hz, $J_{H,^{119}Sn}$ = 42.5 Hz, 1H), (**3aj'** δ 1.34 (t, $J_{H,H}$ = 7.5 Hz, 3H), 2.33 (quint, $J_{H,H}$ = 7.5 Hz, 2H), 5.60 (t, $J_{H,H}$ = 7.5 Hz, 1H)); **^{13}C NMR** (100 MHz, $CDCl_3$) for **3aj** δ 11.1 ($J_{C,^{119}Sn}$ = 340.4 Hz, $J_{C,^{117}Sn}$ = 325.2 Hz), 12.9, 13.6, 26.1, 27.5, 29.1 ($J_{C,^{119}Sn}$ = 18.7 Hz), 56.0, 60.9, 105.9, 126.3, 127.4, 127.6, 128.7, 133.5, 136.9, 137.3, 141.1, 148.1, 151.6, 153.0. **HRMS** (ESI) calcd for $C_{31}H_{48}O_3Na^{120}Sn$ $[M+Na]^+$ 611.2523, found 611.2550.



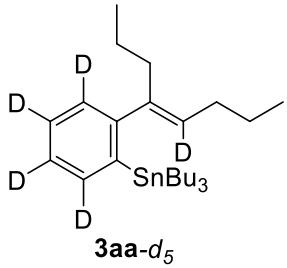
3ak **3ak'**

Compound 3ak:3ak' (8:1). (Table 3, 71% yield, colorless oil, R_f = 0.8 (hexanes)). **^{1}H NMR** (400 MHz, $CDCl_3$) for **3ak** δ 0.84 (t, $J_{H,H}$ = 7.3 Hz, 9H), 0.91 (t, $J_{H,H}$ = 7.4 Hz, 3H), 1.01 (t, $J_{H,H}$ = 8.4 Hz, $J_{H,^{119}Sn}$ = 51.4 Hz, 6H), 1.28 (sext, $J_{H,H}$ = 7.3 Hz, 6H), 1.41–1.50 (m, 8H), 2.61 (deformed t, $J_{H,H}$ = 8.2 Hz, 2H), 6.35 (s, 1H), 7.18–7.39 (m, 8H), 7.49 (d, $J_{H,H}$ = 7.0 Hz, $J_{H,^{119}Sn}$ = 41.8 Hz, 1H), (**3ak'** δ 2.28 (q, $J_{H,H}$ = 7.5 Hz, 2H), 5.68 (t, $J_{H,H}$ = 7.4 Hz, 1H)); **^{13}C NMR** (100 MHz, $CDCl_3$) for **3ak** δ 11.0 ($J_{C,^{119}Sn}$ = 340.1 Hz, $J_{C,^{117}Sn}$ = 325.1 Hz), 13.6, 14.5, 21.7, 27.4 ($J_{C,^{119}Sn}$ = 62.1 Hz), 29.1 ($J_{C,^{119}Sn}$ = 19.1 Hz), 35.3, 126.1, 126.5, 127.5, 127.6, 128.2, 128.7, 129.0, 137.2, 137.9, 140.8, 147.3, 152.2. **HRMS** (ESI) calcd for $C_{29}H_{44}Na^{120}Sn$ $[M+Na]^+$ 535.2363, found 535.2394.



3al **3al'**

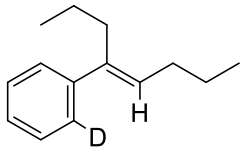
Compound 3al:3al' (7:1). (Table 3, 73% yield, colorless oil, R_f = 0.9 (hexanes)). **^{1}H NMR** (400 MHz, $CDCl_3$) for **3al** δ 0.84 (t, $J_{H,H}$ = 7.3 Hz, 9H), 0.87 (t, $J_{H,H}$ = 7.2 Hz, 3H), 1.01 (t, $J_{H,H}$ = 8.4 Hz, $J_{H,^{119}Sn}$ = 51.2 Hz, 6H), 1.24 (t, $J_{H,H}$ = 7.3 Hz, 6H), 1.27–1.35 (m, 2H), 1.39–1.51 (m, 8H), 2.64 (deformed t, $J_{H,H}$ = 8.1 Hz, 2H), 6.34 (s, 1H), 7.16–7.39 (m, 8H), 7.50 (d, $J_{H,H}$ = 7.0 Hz, $J_{H,^{119}Sn}$ = 41.9 Hz, 1H), (**3al'** δ 2.30 (q, $J_{H,H}$ = 7.5 Hz, 2H), 5.67 (t, $J_{H,H}$ = 7.4 Hz, 1H)); **^{13}C NMR** (100 MHz, $CDCl_3$) for **3al** δ 11.0 ($J_{C,^{119}Sn}$ = 340.3 Hz, $J_{C,^{117}Sn}$ = 325.1 Hz), 13.6, 13.9, 23.1, 27.4 ($J_{C,^{119}Sn}$ = 62.0 Hz), 29.1 ($J_{C,^{119}Sn}$ = 19.7 Hz), 30.6, 32.9, 126.2, 126.5, 127.5, 127.6, 128.2, 128.7, 128.9, 137.3, 137.9, 140.9, 147.4, 152.2. **HRMS** (ESI) calcd for $C_{26}H_{46}Na^{120}Sn$ $[M+Na]^+$ 549.2519, found 549.2546.



3aa-d_5

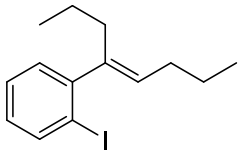
Compound 3aa-d_5. (equotion 1, 87% yield, colorless oil, R_f = 0.9 (hexanes)). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 0.89 (t, $J_{H,H}$ = 7.1 Hz, 9H+3H), 0.96 (t, $J_{H,H}$ = 6.7 Hz, 3H), 0.99 (t, $J_{H,H}$ = 8.1 Hz, $J_{H,^{119}Sn}$ = 51.0 Hz, 6H), 1.32 (sext, $J_{H,H}$ = 7.1 Hz, 6H+2H), 1.42–1.51 (m, 8H), 2.14 (t, $J_{H,H}$ = 7.5 Hz, 2H), 2.32 (t, $J_{H,H}$ =

8.0 Hz, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 10.8 ($J_{C,^{119}Sn}$ = 339.6 Hz, $J_{C,^{117}Sn}$ = 324.7 Hz), 13.6, 14.1, 14.4, 21.6, 22.9, 27.5 ($J_{C,^{119}Sn}$ = 61.0 Hz), 29.2 ($J_{C,^{119}Sn}$ = 18.9 Hz), 30.5, 34.9, 125.1 (t, $J_{C,^{2}H}$ = 23.8 Hz), 126.9 (t, $J_{C,^{2}H}$ = 23.9 Hz), 127.5 (t, $J_{C,^{2}H}$ = 23.2 Hz), 129.2 (t, $J_{C,^{2}H}$ = 22.8 Hz), 136.5 (t, $J_{C,^{2}H}$ = 23.9 Hz), 140.5, 144.2 ($J_{C,^{119}Sn}$ = 14.6 Hz), 152.6 ($J_{C,^{119}Sn}$ = 28.6 Hz); **^{2}H{^{1}H} NMR** (61 MHz, acetone) δ 5.26 (s, 1H), 7.19 (s, 2H), 7.32 (s, 1H), 7.43 (s, 1H). **HRMS** (ESI) calcd for $C_{26}H_{41}D_5Na^{120}Sn$ $[M+Na]^+$ 506.2833, found 506.2838.



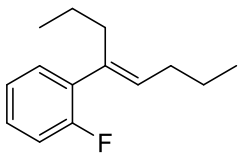
8

Compound 8 [374107-00-3]. (Scheme 3, 98% yield, colorless oil, R_f = 0.9 (hexanes)). **^{1}H NMR** (500 MHz, $CDCl_3$) δ 0.89 (t, $J_{H,H}$ = 7.4 Hz, 3H), 0.97 (t, $J_{H,H}$ = 7.4 Hz, 3H), 1.37 (sext, $J_{H,H}$ = 7.6 Hz, 2H), 1.48 (sext, $J_{H,H}$ = 7.4 Hz, 2H), 2.18 (q, $J_{H,H}$ = 7.3 Hz, 2H), 2.48 (t, $J_{H,H}$ = 7.7 Hz, 2H), 5.67 (t, $J_{H,H}$ = 7.3 Hz, 1H), 7.21 (td, $J_{H,H}$ = 7.4 Hz, 1.3 Hz, 1H), 7.30 (t, $J_{H,H}$ = 6.9 Hz, 1H), 7.30 (d, $J_{H,H}$ = 6.7 Hz, 1H), 7.34 (d, $J_{H,H}$ = 7.7 Hz, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 13.9, 14.0, 21.8, 23.1, 30.6, 31.7, 126.0 (t, $J_{C,^{2}H}$ = 24.1 Hz), 126.3, 128.0, 128.1, 129.2, 140.0, 143.5. The spectral data are in agreement with reported literature values.[15]



9

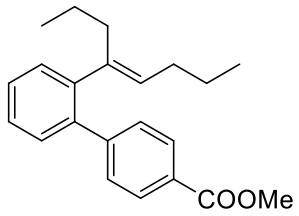
Compound 9. (Scheme 3, 96% yield, colorless oil, R_f = 0.8 (hexanes)). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 0.98 (t, $J_{H,H}$ = 7.3 Hz, 3H), 1.07 (t, $J_{H,H}$ = 7.4 Hz, 3H), 1.39 (sext, $J_{H,H}$ = 7.8 Hz, 2H), 1.57 (sext, $J_{H,H}$ = 7.3 Hz, 2H), 2.25 (q, $J_{H,H}$ = 7.3 Hz, 2H), 2.46 (t, $J_{H,H}$ = 7.8 Hz, 2H), 5.37 (t, $J_{H,H}$ = 7.3 Hz, 1H), 6.95 (td, $J_{H,H}$ = 7.7 Hz, 1.7 Hz, 1H), 7.17 (dd, $J_{H,H}$ = 7.5 Hz, 1.7 Hz, 1H), 7.31 (td, $J_{H,H}$ = 7.5 Hz, 1.2 Hz, 1H), 7.89 (dd, $J_{H,H}$ = 7.9 Hz, 1.1 Hz, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 14.0, 14.2, 21.2, 22.7, 30.0, 33.5, 99.4, 127.5, 127.8, 129.6, 131.3, 139.0, 143.3, 149.1. **HRMS** (ESI) calcd for $C_{14}H_{20}I$ $[M+H]^+$ 315.0610, found 315.0613.



10

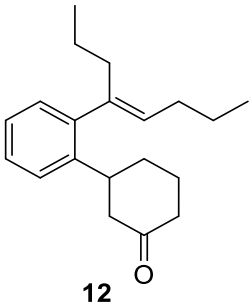
Compound 10 [1092526-15-2]. (Scheme 3, 69% yield, colorless oil, R_f = 0.9 (hexanes)). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 0.86 (t, $J_{H,H}$ = 7.3 Hz, 3H), 0.96 (t, $J_{H,H}$ = 7.4 Hz, 3H), 1.30 (sext, $J_{H,H}$ = 7.4 Hz, 2H), 1.47 (sext, $J_{H,H}$ = 7.4 Hz, 2H), 2.18 (q, $J_{H,H}$ = 7.3 Hz, 2H), 2.44 (t, $J_{H,H}$ = 7.6 Hz, 2H), 5.50

(t, $J_{H,H}$ = 7.2 Hz, 1H), 6.99 (dd, $J_{H,H}$ = 8.4 Hz, $J_{H,^{19}F}$ = 10.6 Hz, 1H), 7.06 (dd, $J_{H,H}$ = 7.4 Hz, 1.2 Hz, 1H), 7.17 (d, $J_{H,H}$ = 7.2 Hz, 1H), 7.19–7.23 (m, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 13.8, 21.5, 22.9, 30.3, 32.5, 115.4 (d, $J_{C,^{19}F}$ = 23.1 Hz), 123.7 (d, $J_{C,^{19}F}$ = 3.2 Hz), 126.4, 127.9 (d, $J_{C,^{19}F}$ = 8.0 Hz), 130.6 (d, $J_{C,^{19}F}$ = 4.6 Hz), 131.9 (d, $J_{C,^{19}F}$ = 14.6 Hz), 136.1, 160.0 (d, $J_{C,^{19}F}$ = 245.8 Hz). The spectral data are in agreement with reported literature values.[16]



11

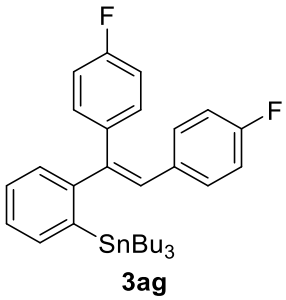
Compound 11. (Scheme 3, 83% yield, pale yellow oil, R_f = 0.4 (hexanes)). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 0.69 (t, $J_{H,H}$ = 7.3 Hz, 3H), 0.91 (t, $J_{H,H}$ = 7.4 Hz, 3H), 1.10 (sext, $J_{H,H}$ = 7.5 Hz, 2H), 1.40 (sext, $J_{H,H}$ = 7.3 Hz, 2H), 1.79 (t, $J_{H,H}$ = 7.7 Hz, 2H), 2.06 (q, $J_{H,H}$ = 7.3 Hz, 2H), 3.93 (s, 3H), 5.48 (t, $J_{H,H}$ = 7.3 Hz, 1H), 7.20–7.33 (m, 4H), 7.50 (d, $J_{H,H}$ = 8.3 Hz, 2H), 8.01 (d, $J_{H,H}$ = 8.3 Hz, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 13.9, 21.5, 22.8, 30.3, 32.9, 52.0, 126.8, 127.6, 128.3, 129.1, 129.2, 129.7, 130.4, 132.2, 138.6, 141.2, 143.6, 147.2, 167.2. **HRMS** (ESI) calcd for $C_{22}H_{27}O_2$ $[M+H]^+$ 323.2011, found 323.1995.



12

Compound 12. (Scheme 3, 87% yield, colorless oil, R_f = 0.4 (hexanes)). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 0.87 (t, $J_{H,H}$ = 7.3 Hz, 3H), 0.94 (t, $J_{H,H}$ = 7.4 Hz, 3H), 1.27 (sext d, $J_{H,H}$ = 7.8 Hz, 2.2 Hz, 2H), 1.43 (sext, $J_{H,H}$ = 7.3 Hz, 2H), 1.69 (qt, $J_{H,H}$ = 12.8 Hz, 4.5 Hz, 1H), 1.82 (qd, $J_{H,H}$ = 12.3 Hz, 3.2 Hz, 1H), 1.91 (br d, $J_{H,H}$ = 13.2 Hz, 1H), 2.14 (q, $J_{H,H}$ = 7.4 Hz, 2H), 2.11–2.20 (m, 1H), 2.22–2.31 (m, 2H), 2.38 (td, $J_{H,H}$ = 14.5 Hz, 6.6 Hz, 1H), 2.4–2.5 (m, 1H), 2.45 (br d, $J_{H,H}$ = 14.1 Hz, 1H), 2.53 (t, $J_{H,H}$ = 13.8 Hz, 1H), 3.23 (tt, $J_{H,H}$ = 12.2 Hz, 3.9 Hz, 1H), 5.21 (t, $J_{H,H}$ = 7.3 Hz, 1H), 7.05 (dd, $J_{H,H}$ = 7.5 Hz, 1.0 Hz, 1H), 7.15 (td, $J_{H,H}$ =7.3 Hz, 1.6 Hz, 1H), 7.25 (td, $J_{H,H}$ = 7.8 Hz, 1.2 Hz, 1H), 7.29 (dd, $J_{H,H}$ = 7.7 Hz, 1.4 Hz, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 13.9, 14.2, 21.3, 23.0, 25.9, 30.1, 33.4, 34.9, 40.6, 41.2, 49.3, 125.6, 125.8, 126.8, 129.5, 130.4, 139.6, 141.7, 143.8, 211.1. **HRMS** (ESI) calcd for $C_{20}H_{29}O$ $[M+H]^+$ 285.2218, found 285.2221.

10. Single crystal X-ray diffraction data for compound 3ag (CCDC 1841177)



3ag

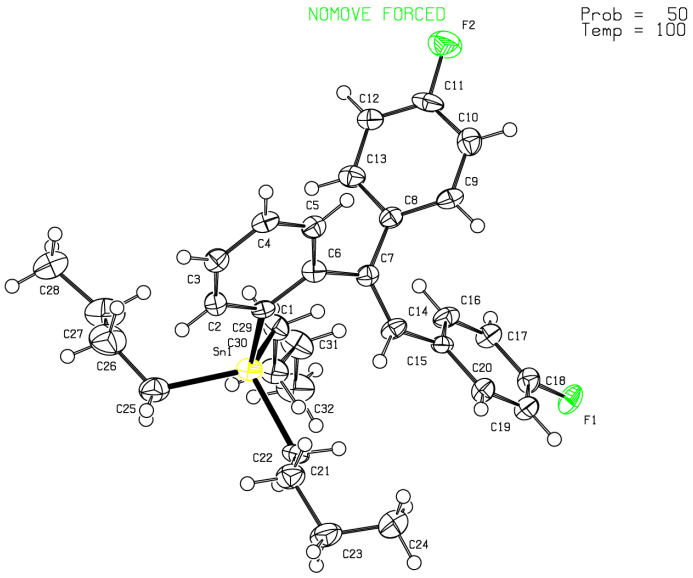


Table S1. Crystal data and structure refinement for compound 3ag

Empirical formula	$C_{32}H_{40}F_2Sn$	
Formula weight	581.33 g/mol	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal size	0.020 x 0.040 x 0.320 mm	
Crystal habit	colorless needle	
Crystal system	Monoclinic	
Space group	P 1 21/c 1	
Unit cell dimensions	a = 17.1715(13) Å	α = 90°.
	b = 17.8894(14) Å	β = 101.309(3)°.
	c = 9.4518(7) Å	γ = 90°.
Volume	2847.1(4) $Å^3$	
Z	4	
Density (calculated)	1.356 g/cm^3	
Absorption coefficient	0.928 mm^{-1}	
F(000)	1200	
Theta range for data collection	2.48 to 27.94°.	
Index ranges	–22<=h<=20, –22<=k<=23, –12<=l<=11	
Reflections collected	16876	
Independent reflections	6610 [R(int) = 0.0945]	

Coverage of independent reflections	96.5%	
Absorption correction	Multi-Scan	
Max. and min. transmission	0.9820 and 0.7560	
Structure solution technique	direct methods	
Structure solution program	XT, VERSION 2014/5	
Refinement method	Full-matrix least-squares on F^2	
Refinement program	SHELXL-2016/6 (Sheldrick, 2016)	
Function minimized	$\Sigma\, w(F_o^2 - F_c^2)^2$	
Data / restraints / parameters	6610 / 0 / 319	
Goodness-of-fit on F^2	1.014	
Final R indices	3523 data; $I>2\sigma(I)$	R1 = 0.0663, wR2 = 0.1362
	all data	R1 = 0.1468, wR2 = 0.1726
Weighting scheme	$w=1/[\sigma^2(F_o^2)+(0.0685P)^2]$	
	where $P=(F_o^2+2F_c^2)/3$	
Largest diff. peak and hole	1.424 and –1.680 $eÅ^{-3}$	
R.M.S. deviation from mean	0.155 $eÅ^{-3}$	

11. Reference

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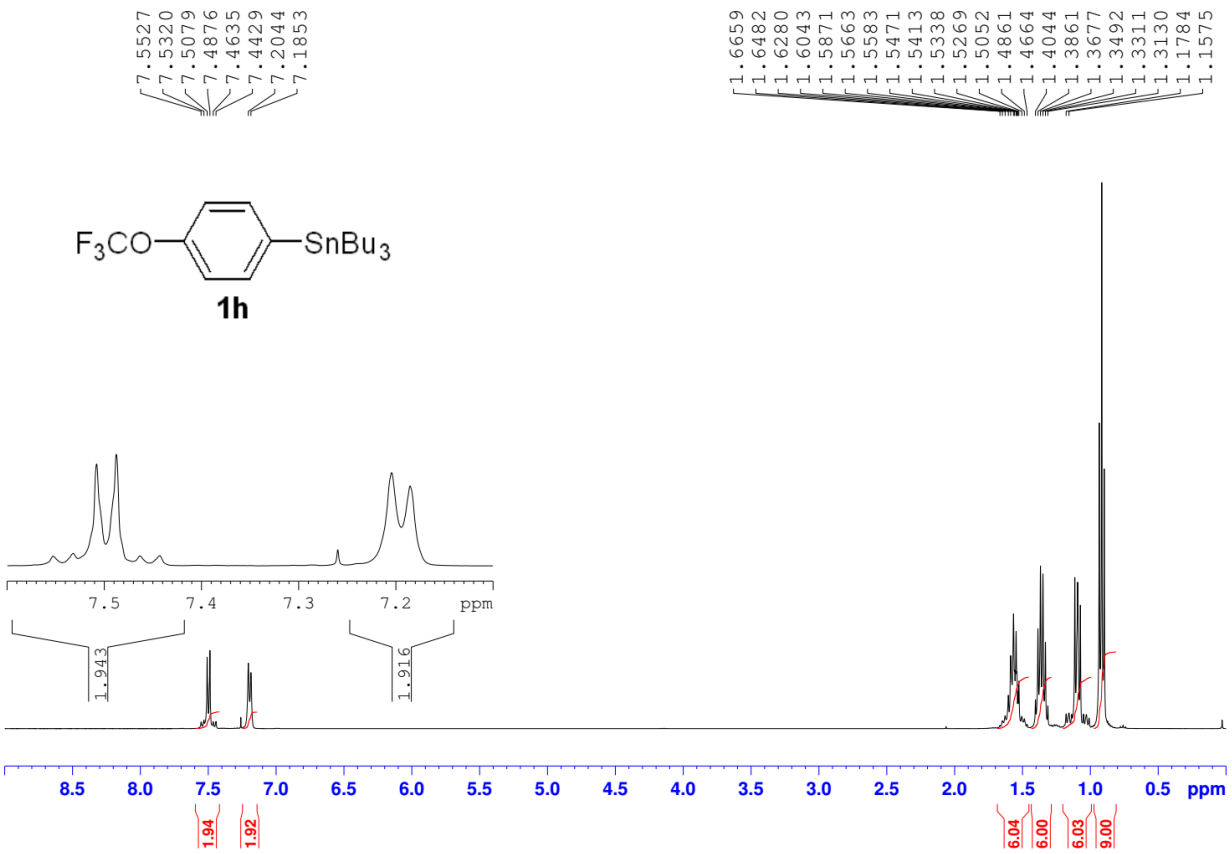
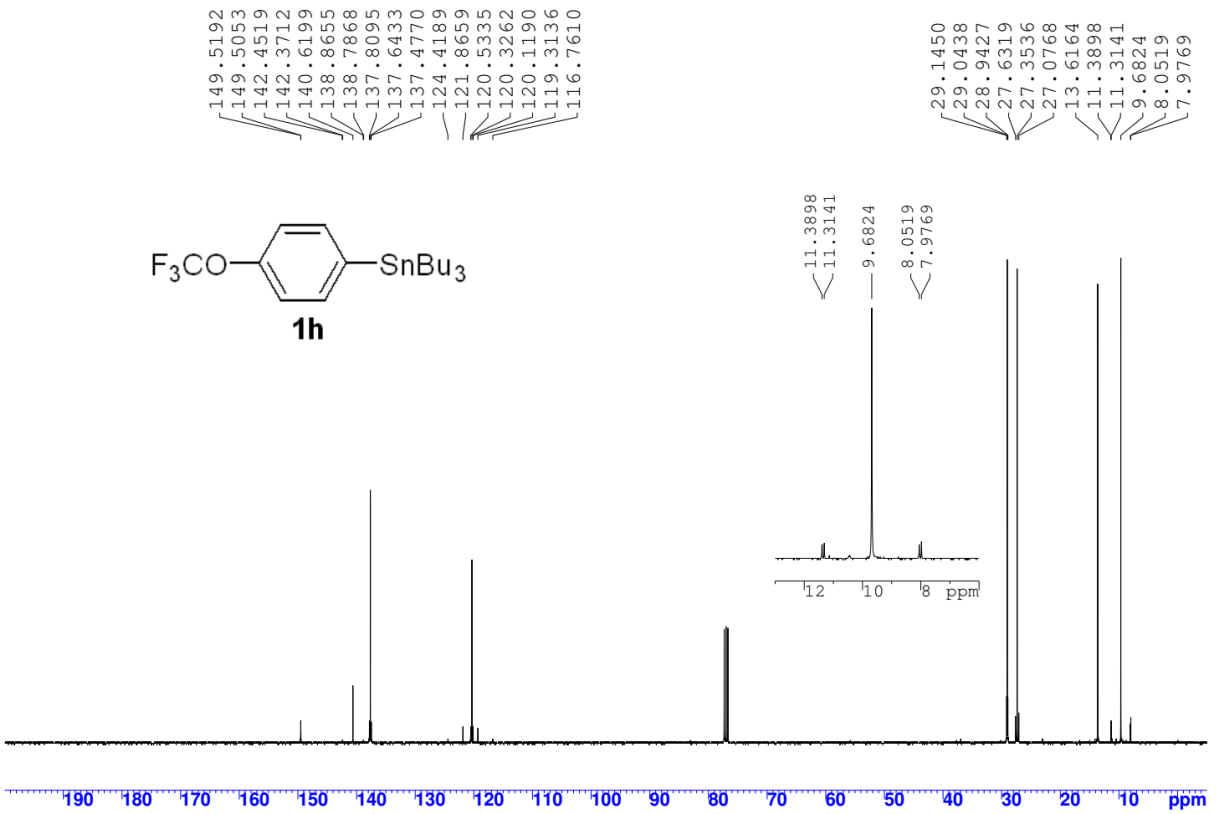
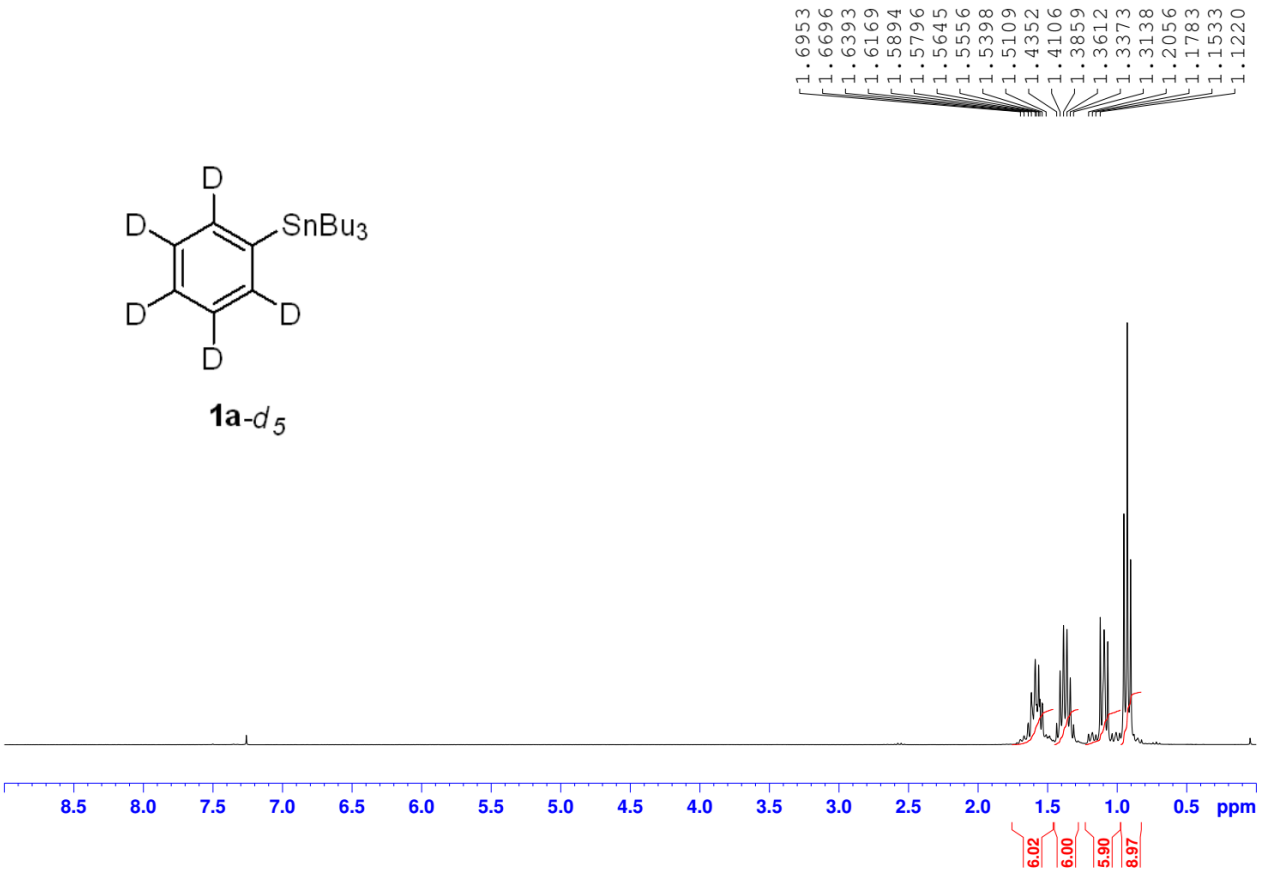
12. NMR spectra



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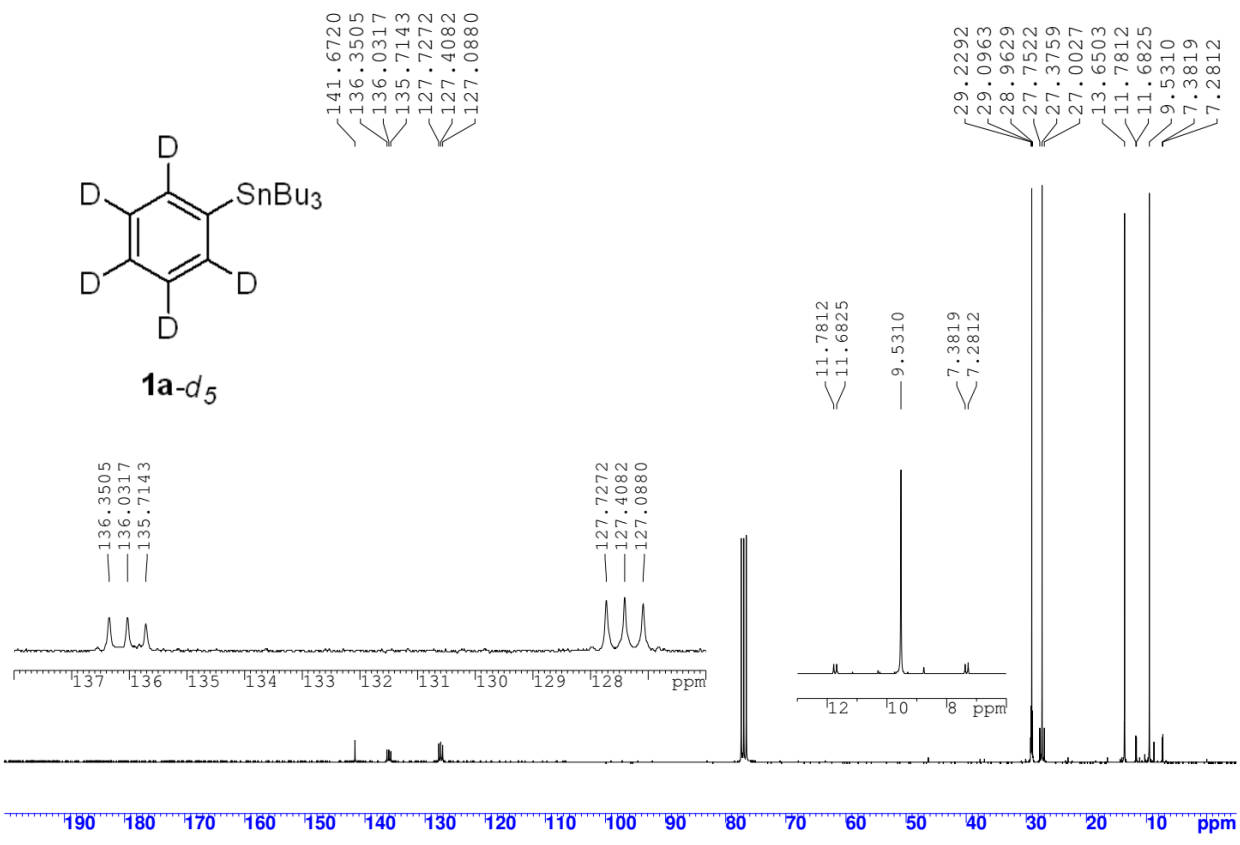
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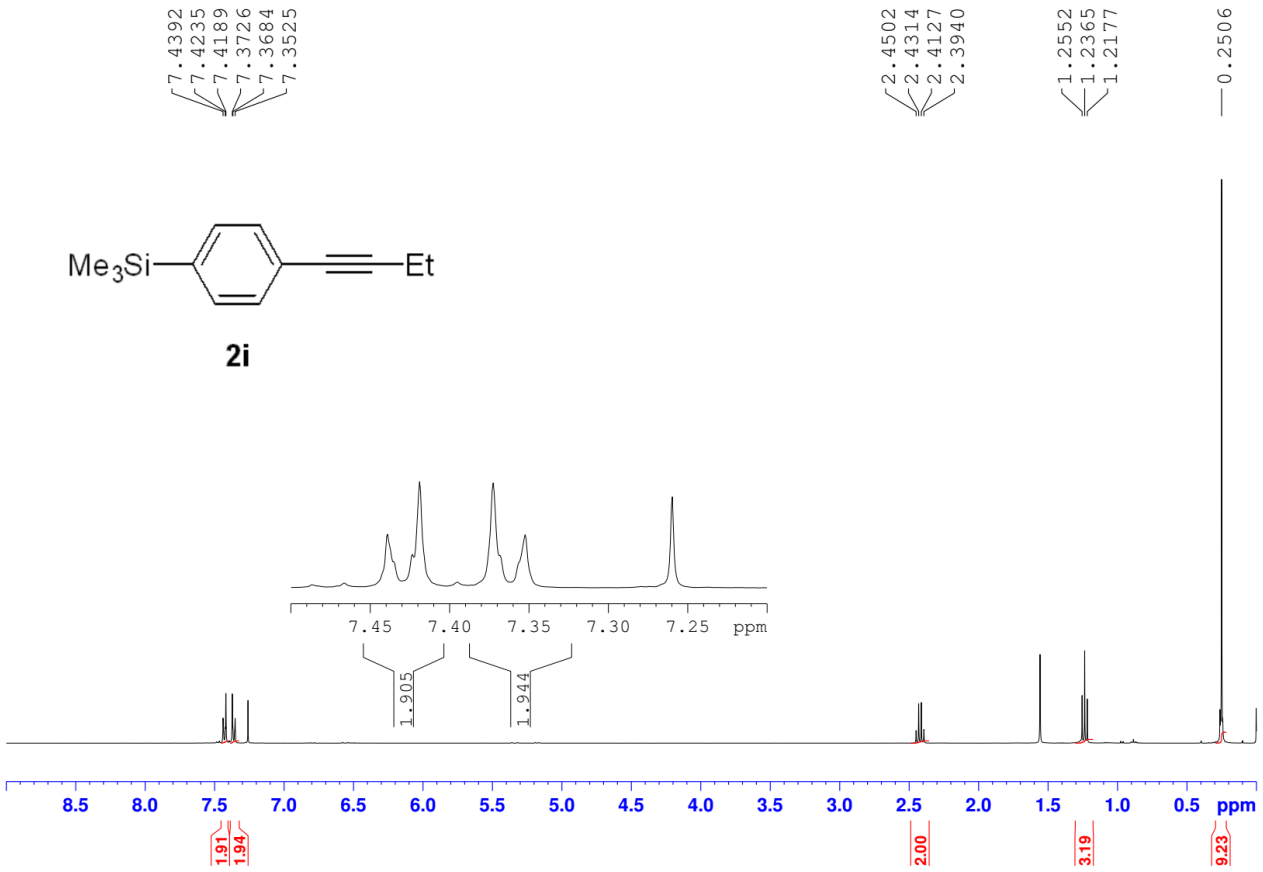
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7.3726
7.3684
7.3525
2.4502
2.4314
2.4127
2.3940
1.2552
1.2365
1.2177
0.2506
Me3Si
Et
2i
7.45
7.40
7.35
7.30
7.25
ppm
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1.944
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
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ppm
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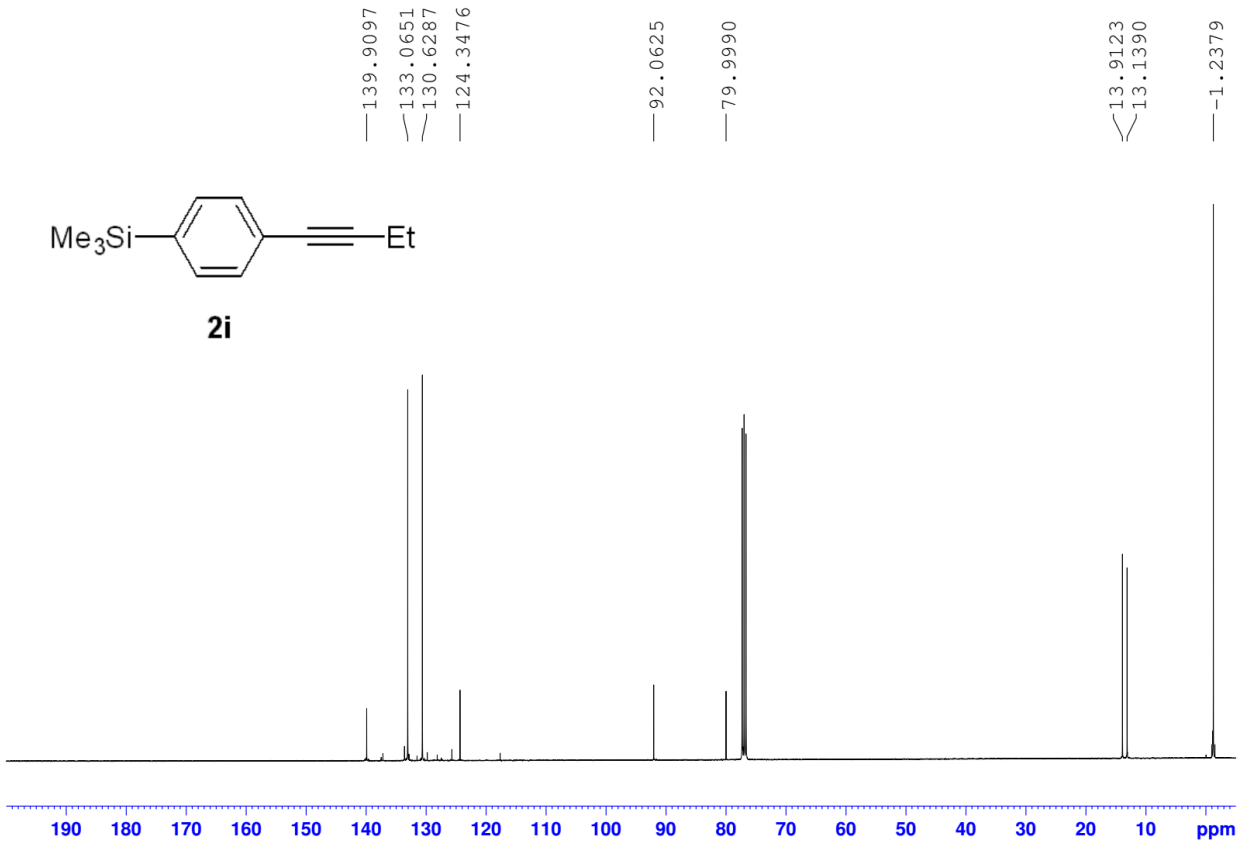
1.94
2.00
3.19
9.23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Me3Si
Et
2i
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110
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80
70
60
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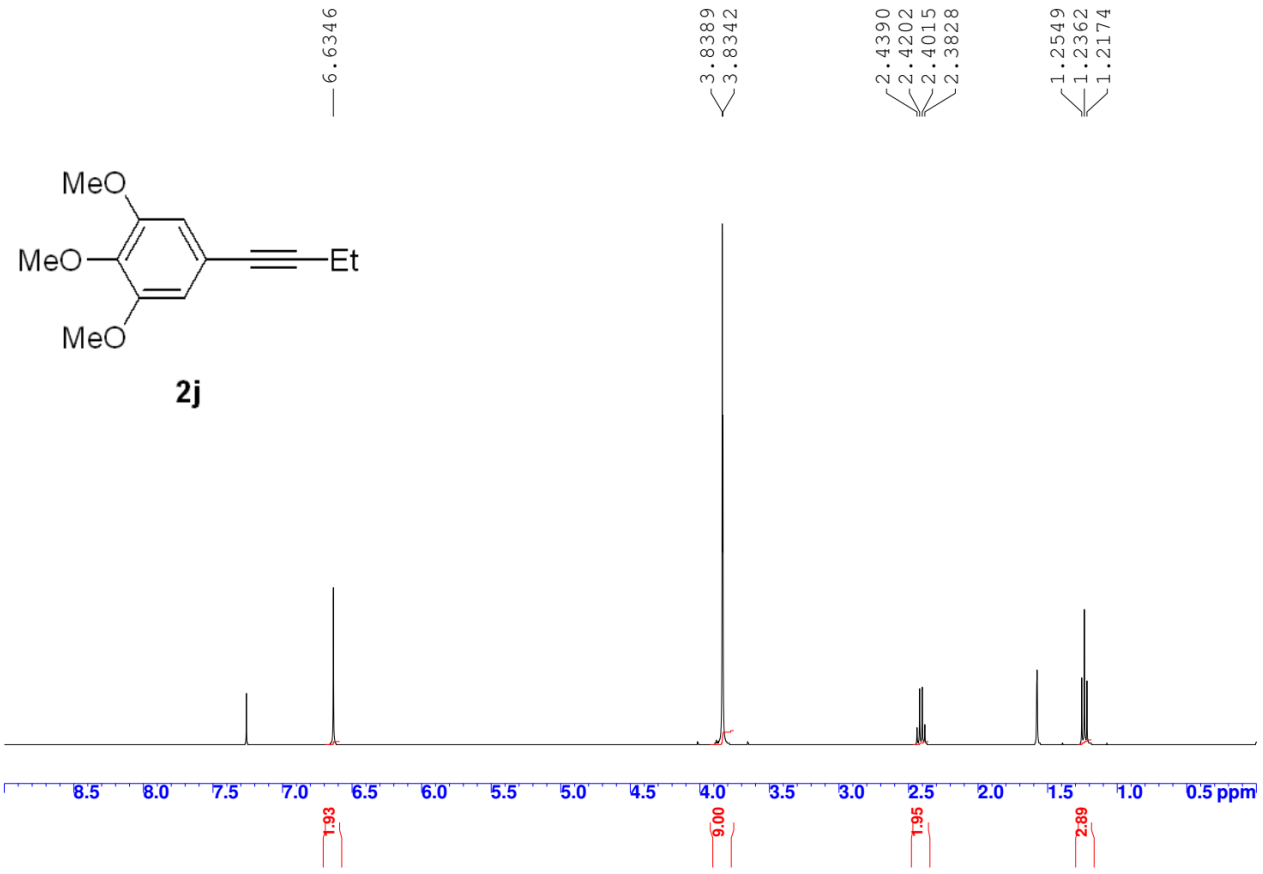
40
30
20
10
ppm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1.2362
1.2174
MeO
MeO
MeO
Et
2j
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
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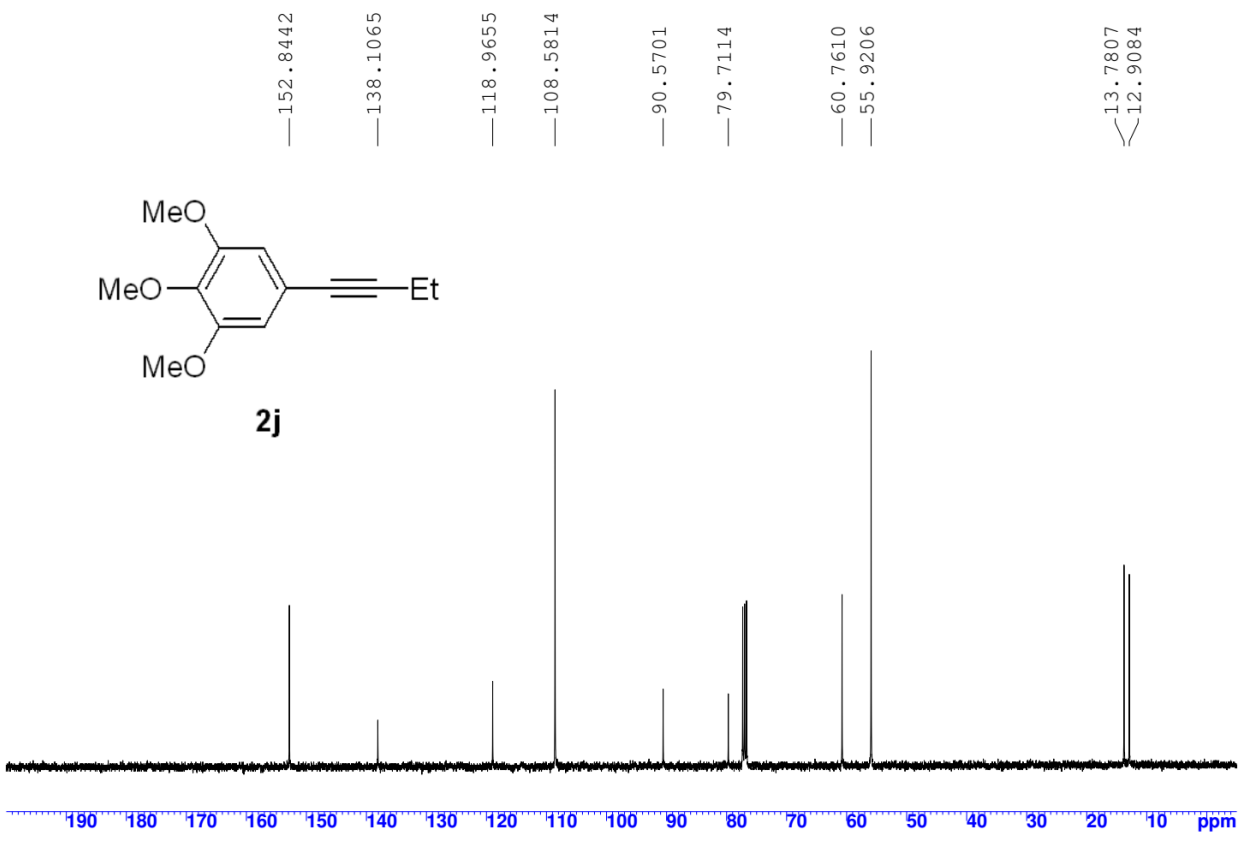
1.95
2.89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79.7114
60.7610
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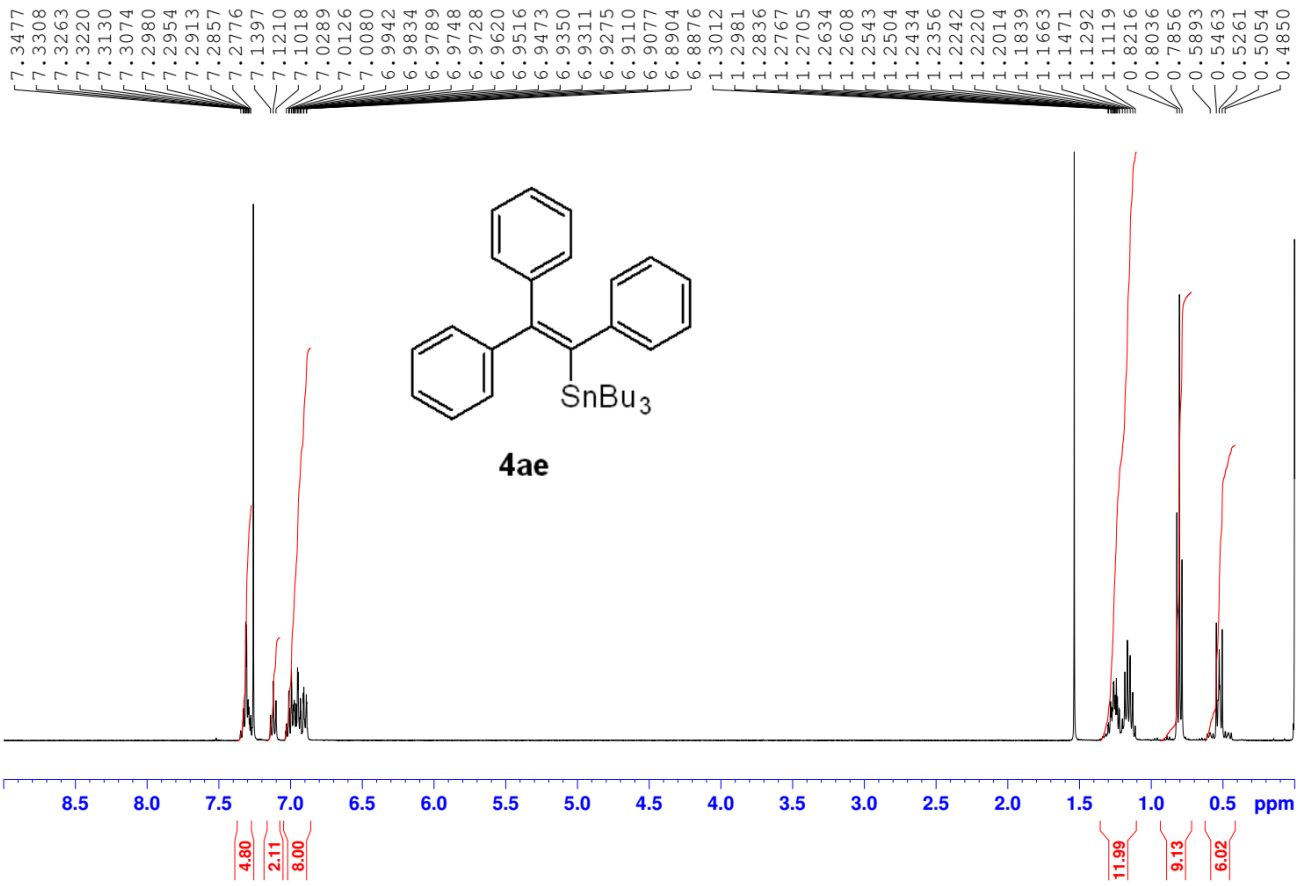
12.9084
MeO
MeO
MeO
Et
2j
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm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7.2776
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7.1210
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7.0126
7.0080
6.9942
6.9834
6.9789
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6.9728
6.9620
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6.9350
6.9311
6.9275
6.9110
6.9077
6.8904
6.8876
1.3012
1.2981
1.2836
1.2767
1.2705
1.2634
1.2608
1.2543
1.2504
1.2434
1.2356
1.2242
1.2220
1.2014
1.1839
1.1663
1.1471
1.1292
1.1119
0.8216
0.8036
0.7856
0.5893
0.5463
0.5261
0.5054
0.4850
SnBu3
4ae
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
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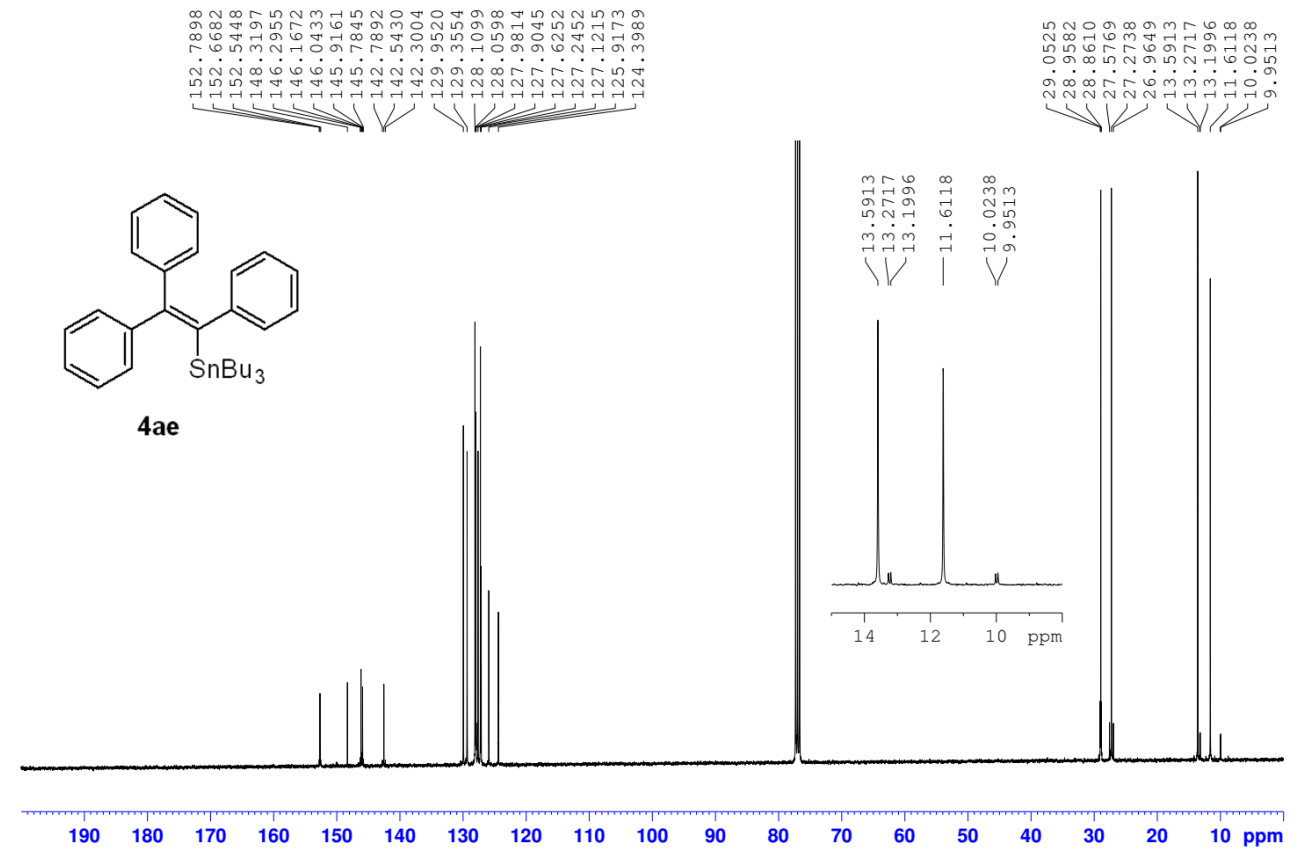
6.02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125.9173
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29.0525
28.9582
28.8610
27.5769
27.2738
26.9649
13.5913
13.2717
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SnBu3
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14 12 10 ppm
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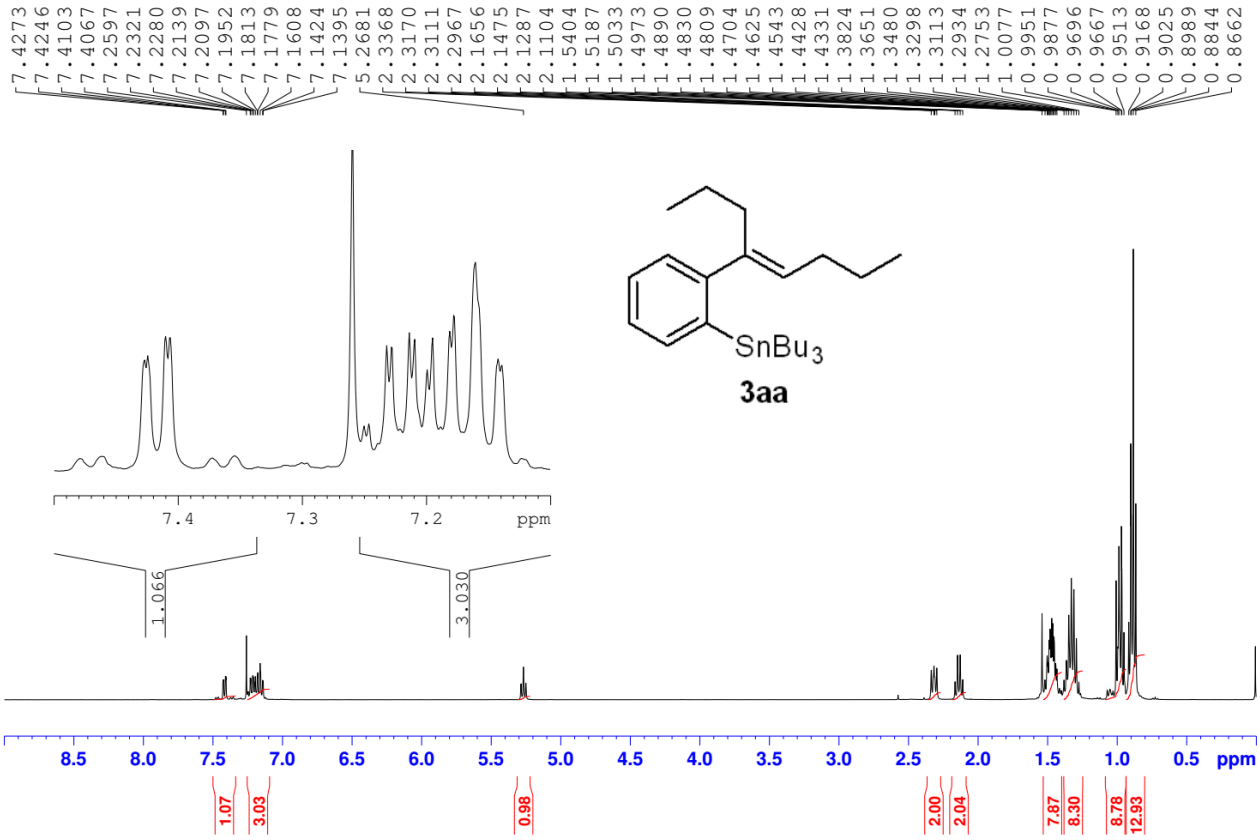
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7.1779
7.1608
7.1424
7.1395
5.2681
2.3368
2.3170
2.3111
2.2967
2.1656
2.1475
2.1287
2.1104
1.5404
1.5187
1.5033
1.4973
1.4890
1.4830
1.4809
1.4704
1.4625
1.4543
1.4428
1.4331
1.3824
1.3651
1.3480
1.3298
1.3113
1.2934
1.2753
1.0077
0.9951
0.9877
0.9696
0.9667
0.9513
0.9168
0.9025
0.8989
0.8844
0.8662
SnBu3
3aa
7.4
7.3
7.2
ppm
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3.030
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
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2.04
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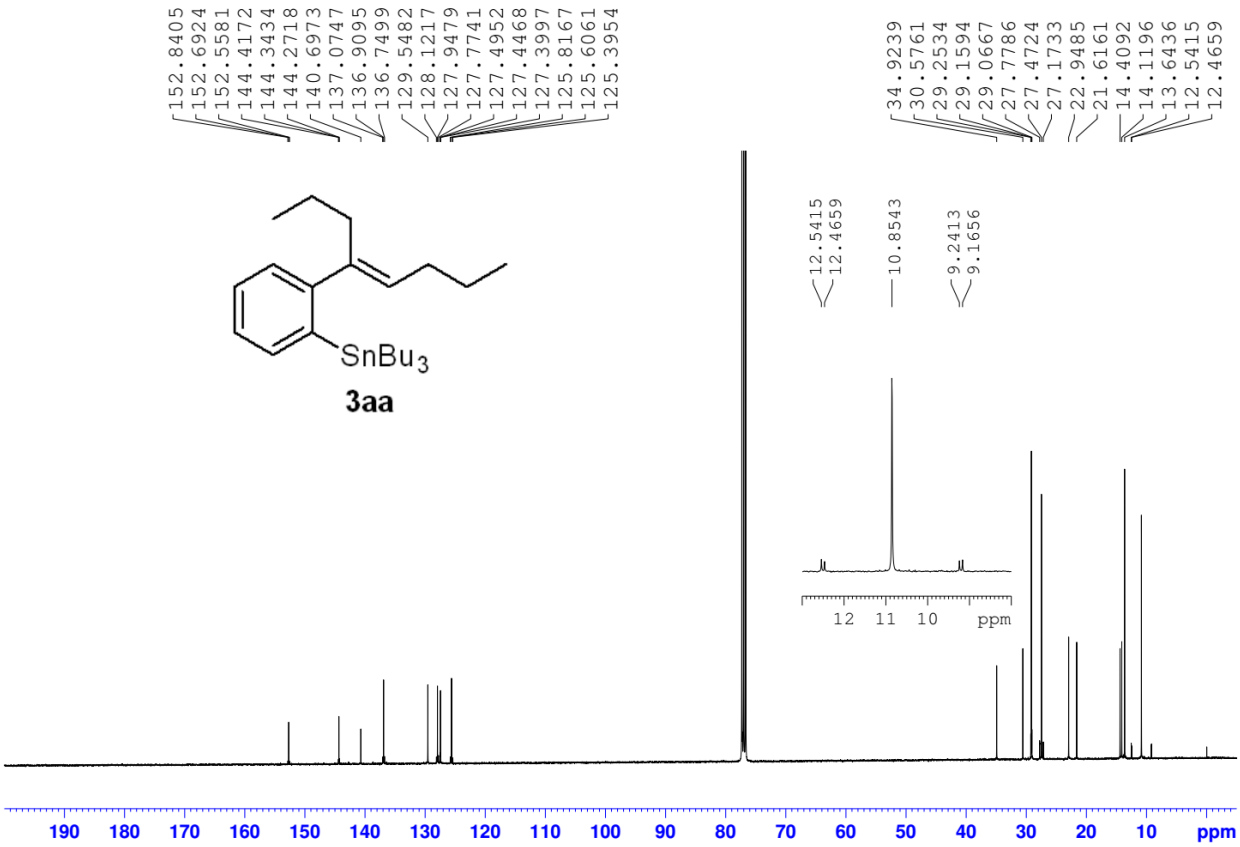
12.93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29.2534
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29.0667
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27.4724
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22.9485
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14.4092
14.1196
13.6436
12.5415
12.4659
SnBu3
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10.8543
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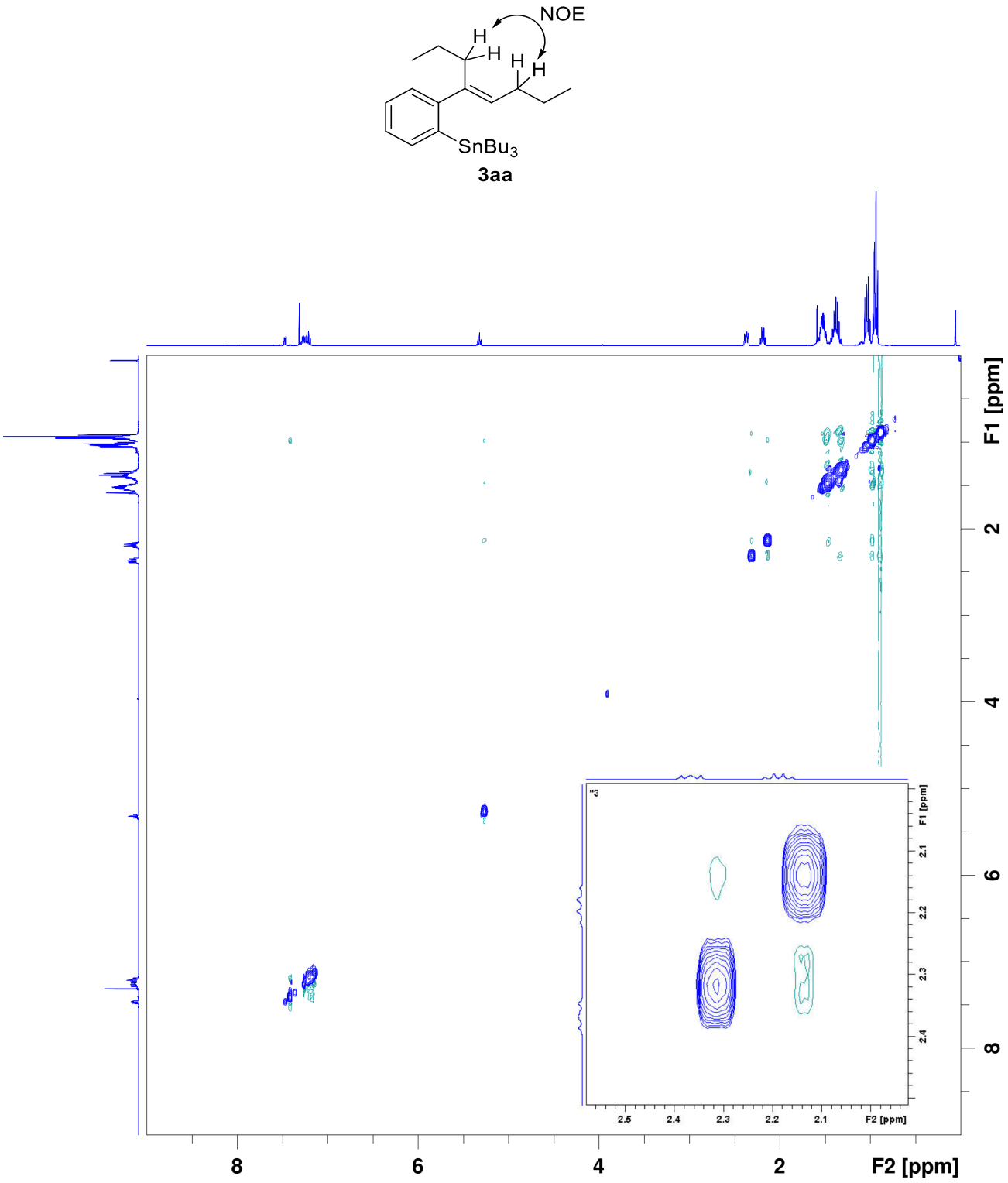
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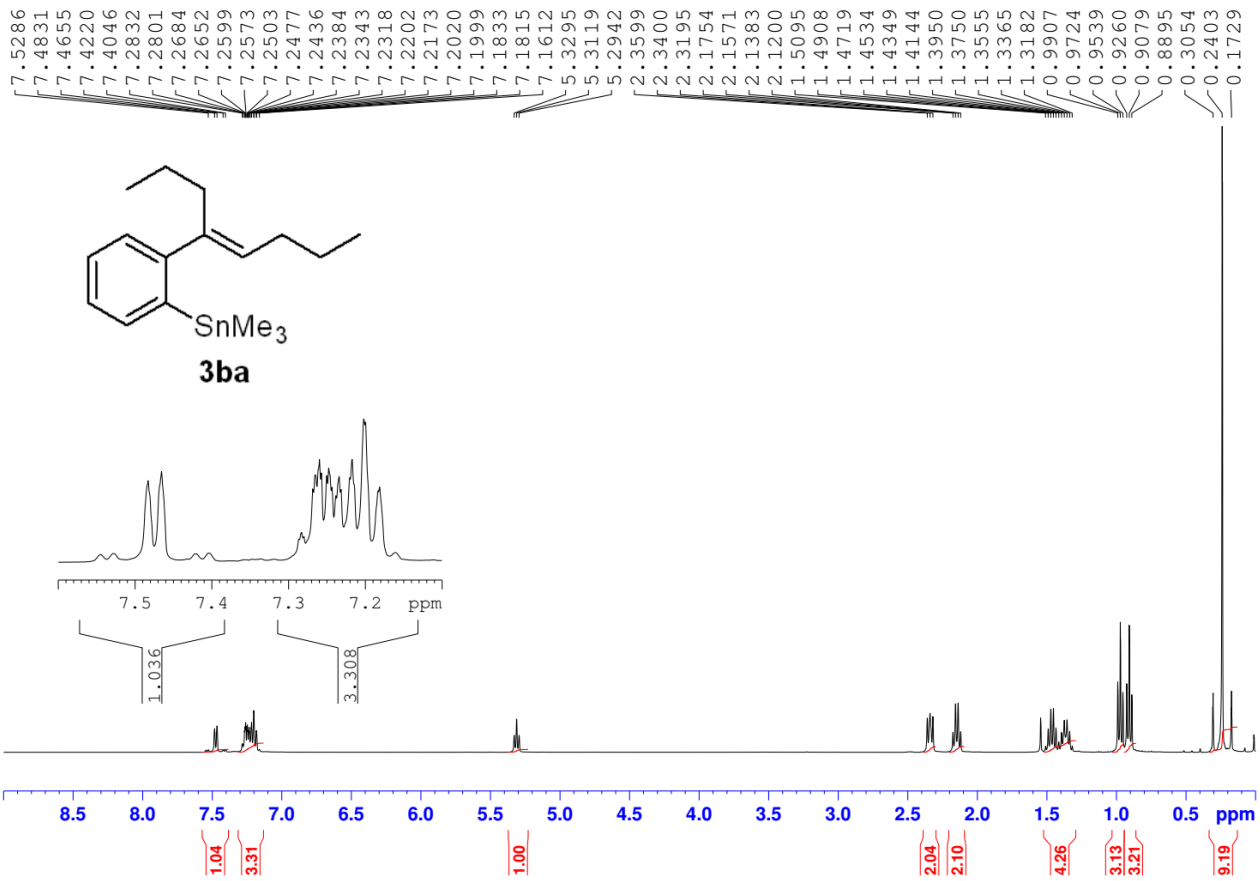
H
H
H
H
SnBu3
3aa
F1 [ppm]
F2 [ppm]
8
6
4
2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7.2801
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7.2652
7.2599
7.2573
7.2503
7.2477
7.2436
7.2384
7.2343
7.2318
7.2202
7.2173
7.2020
7.1999
7.1833
7.1815
7.1612
5.3295
5.3119
5.2942
2.3599
2.3400
2.3195
2.1754
2.1571
2.1383
2.1200
1.5095
1.4908
1.4719
1.4534
1.4349
1.4144
1.3950
1.3750
1.3555
1.3365
1.3182
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0.9724
0.9539
0.9260
0.9079
0.8895
0.3054
0.2403
0.1729
SnMe3
3ba
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7.4
7.3
7.2
ppm
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7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
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ppm
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1.00
2.04
2.10
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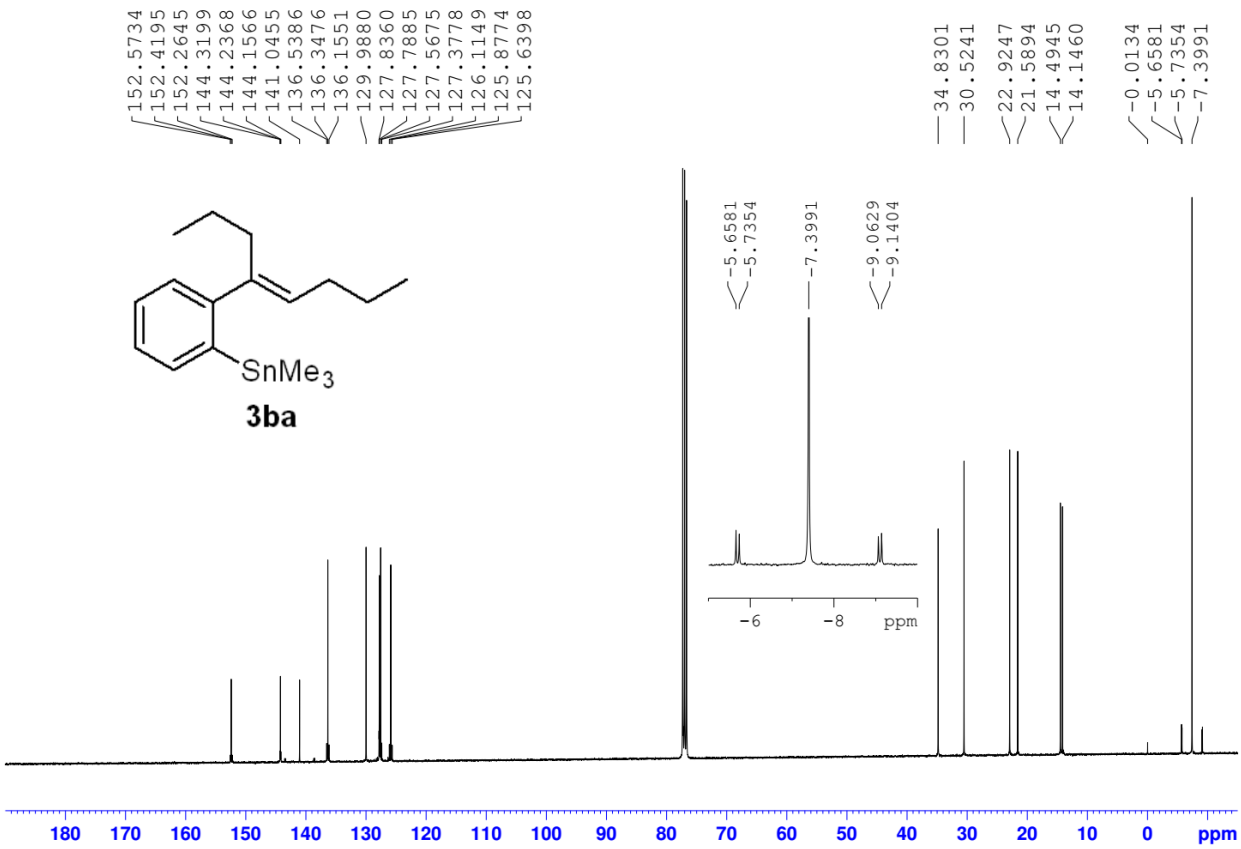
3.13
3.21
9.19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126.1149
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30.5241
22.9247
21.5894
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14.1460
-0.0134
-5.6581
-5.7354
-7.3991
-5.6581
-5.7354
-7.3991
-9.0629
-9.1404
SnMe3
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-8
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140
130
120
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80
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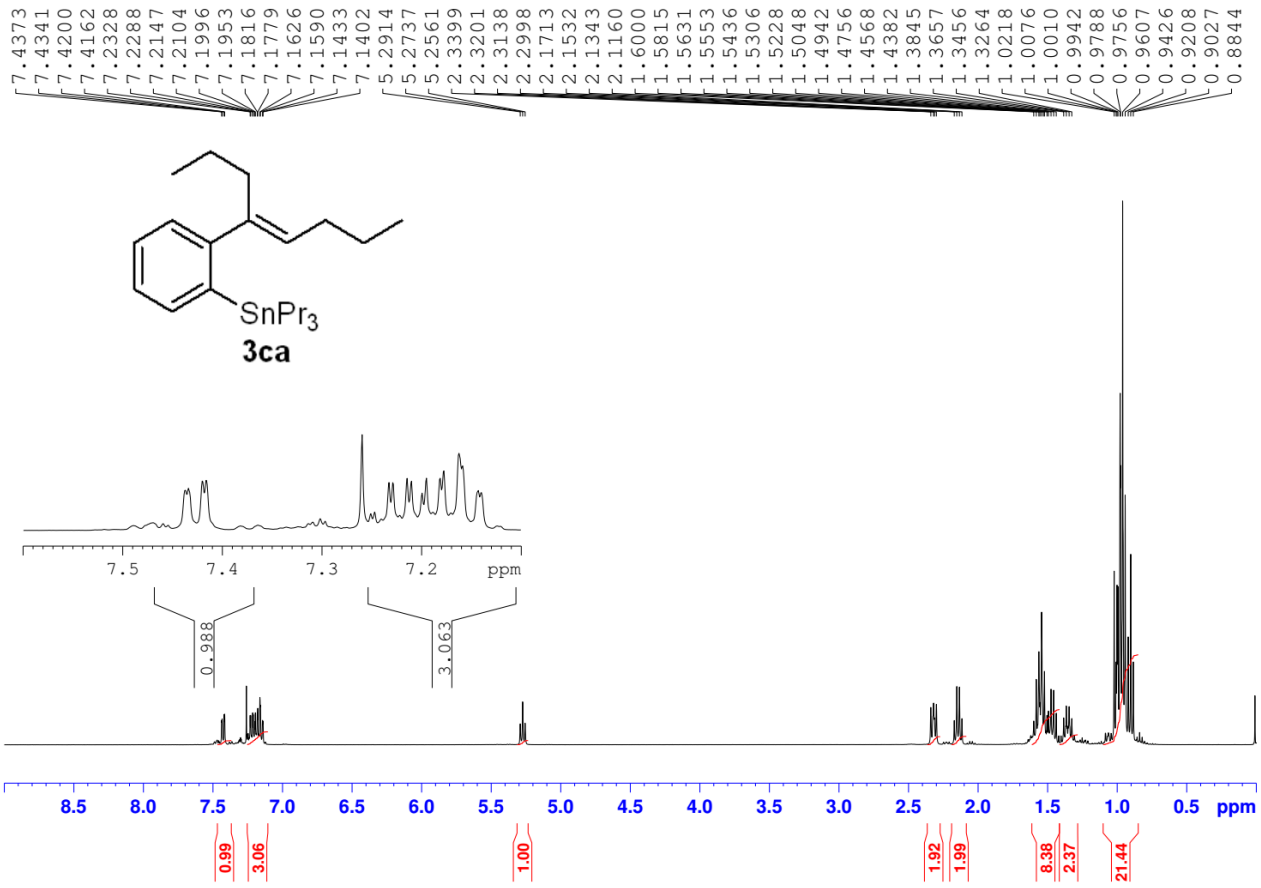
60
50
40
30
20
10
0
ppm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7.1626
7.1590
7.1433
7.1402
5.2914
5.2737
5.2561
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2.3201
2.3138
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2.1713
2.1532
2.1343
2.1160
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1.5815
1.5631
1.5553
1.5436
1.5306
1.5228
1.5048
1.4942
1.4756
1.4568
1.4382
1.3845
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1.3456
1.3264
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1.0076
1.0010
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0.9788
0.9756
0.9607
0.9426
0.9208
0.9027
0.8844
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7.3
7.2
ppm
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4.5
4.0
3.5
3.0
2.5
2.0
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0.5
ppm
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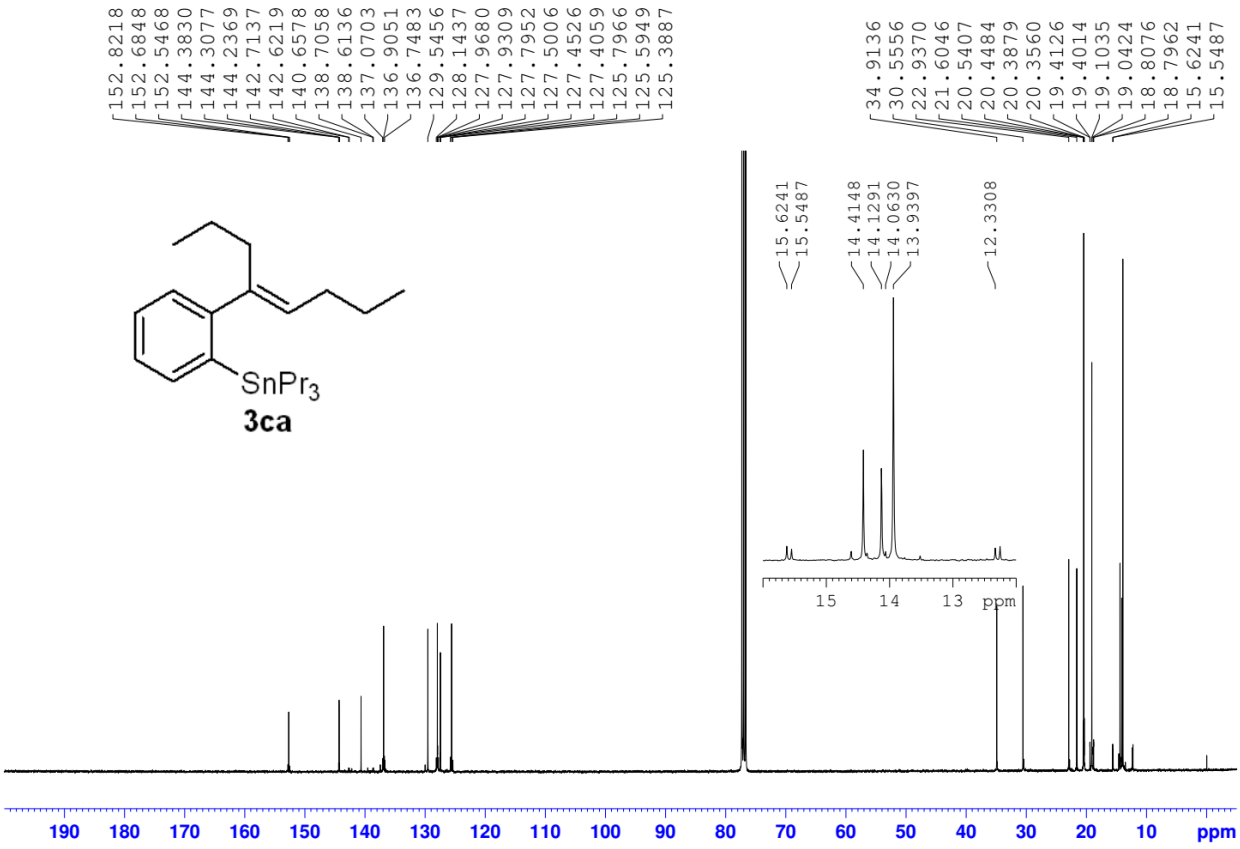
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136.7483
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127.9309
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127.4059
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22.9370
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20.4484
20.3879
20.3560
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19.4014
19.1035
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SnPr3
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15
14
13
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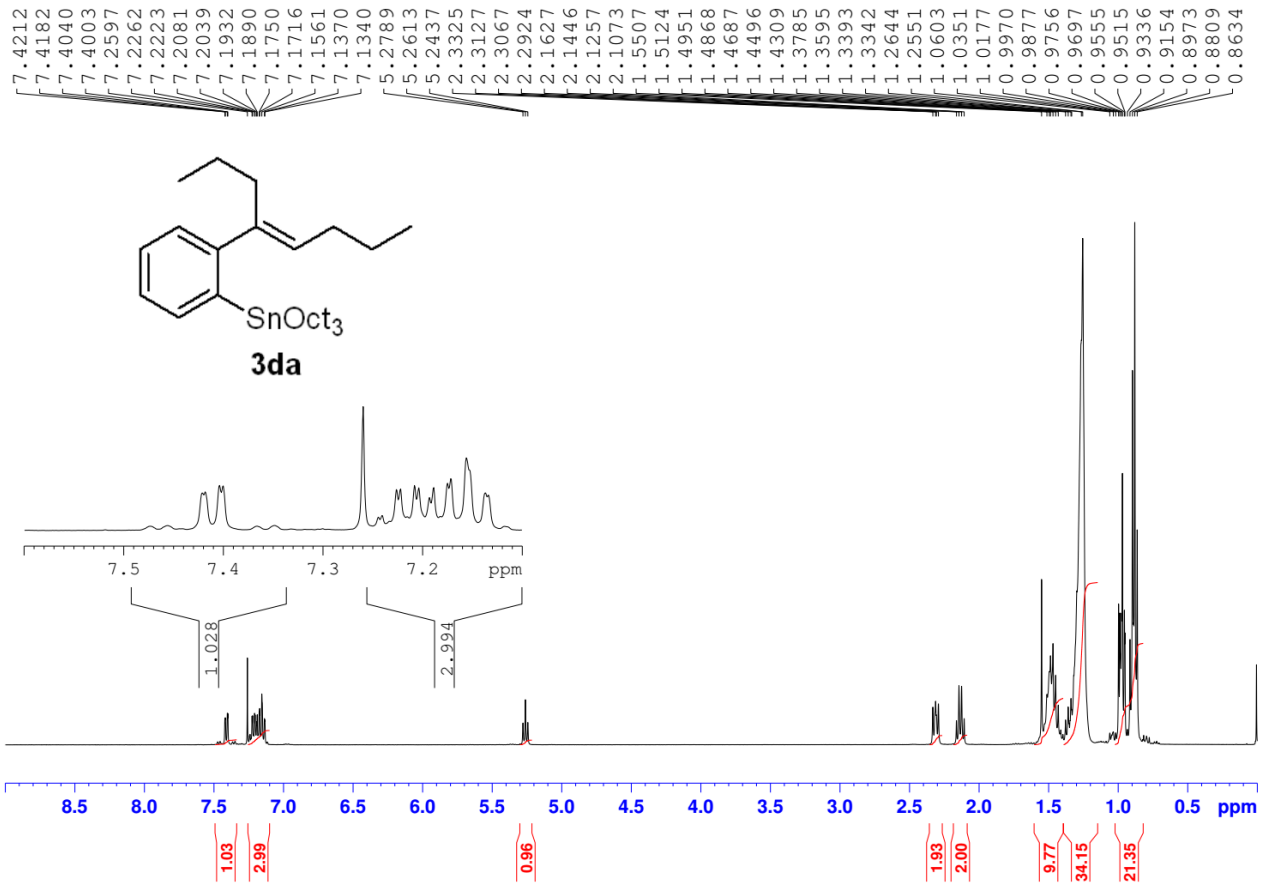
30
20
10
ppm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7.1750
7.1716
7.1561
7.1370
7.1340
5.2789
5.2613
5.2437
2.3325
2.3127
2.3067
2.2924
2.1627
2.1446
2.1257
2.1073
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1.4868
1.4687
1.4496
1.4309
1.3785
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1.3393
1.3342
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0.9697
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0.9154
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0.8809
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SnOct3
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7.4
7.3
7.2
ppm
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6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
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1.93
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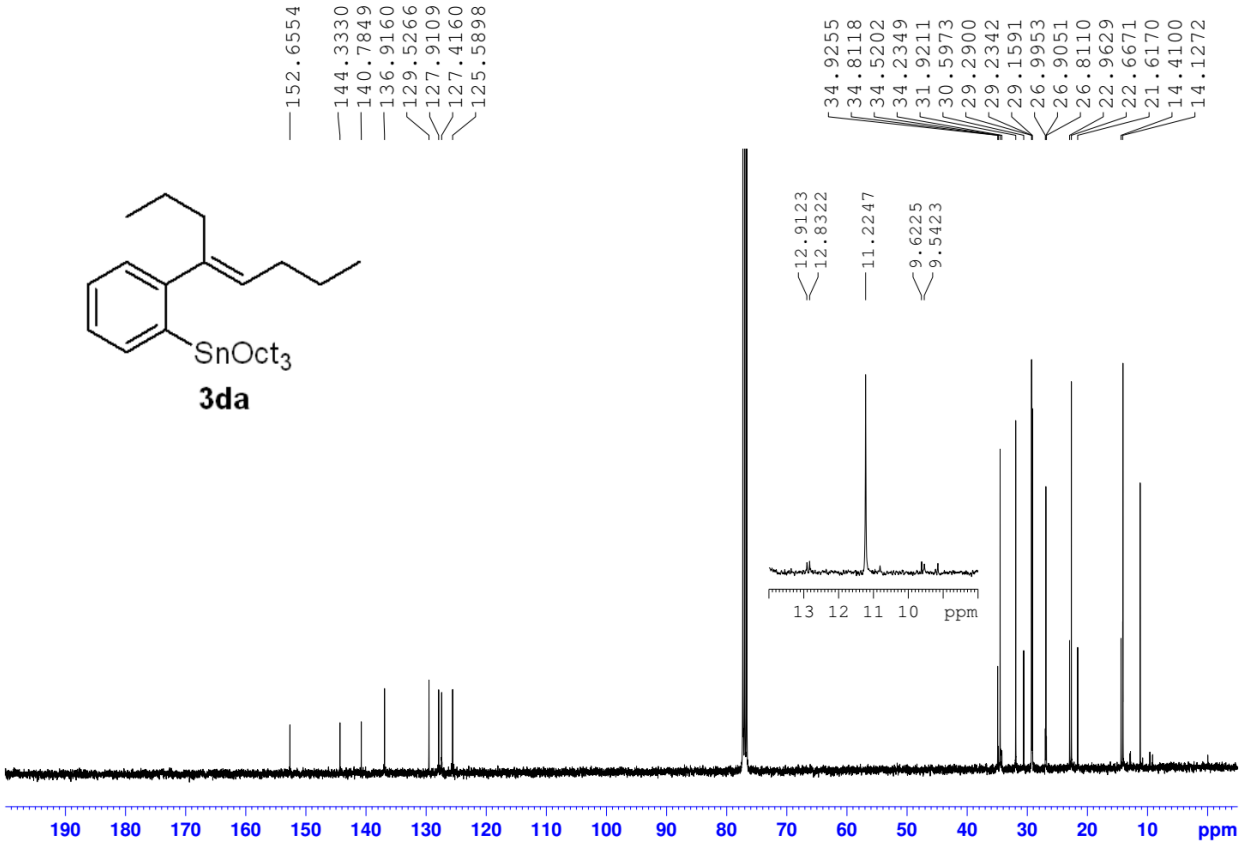
21.35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26.9953
26.9051
26.8110
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22.6671
21.6170
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12.8322
11.2247
9.6225
9.5423
SnOct3
3da
13 12 11 10 ppm
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140
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110
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70
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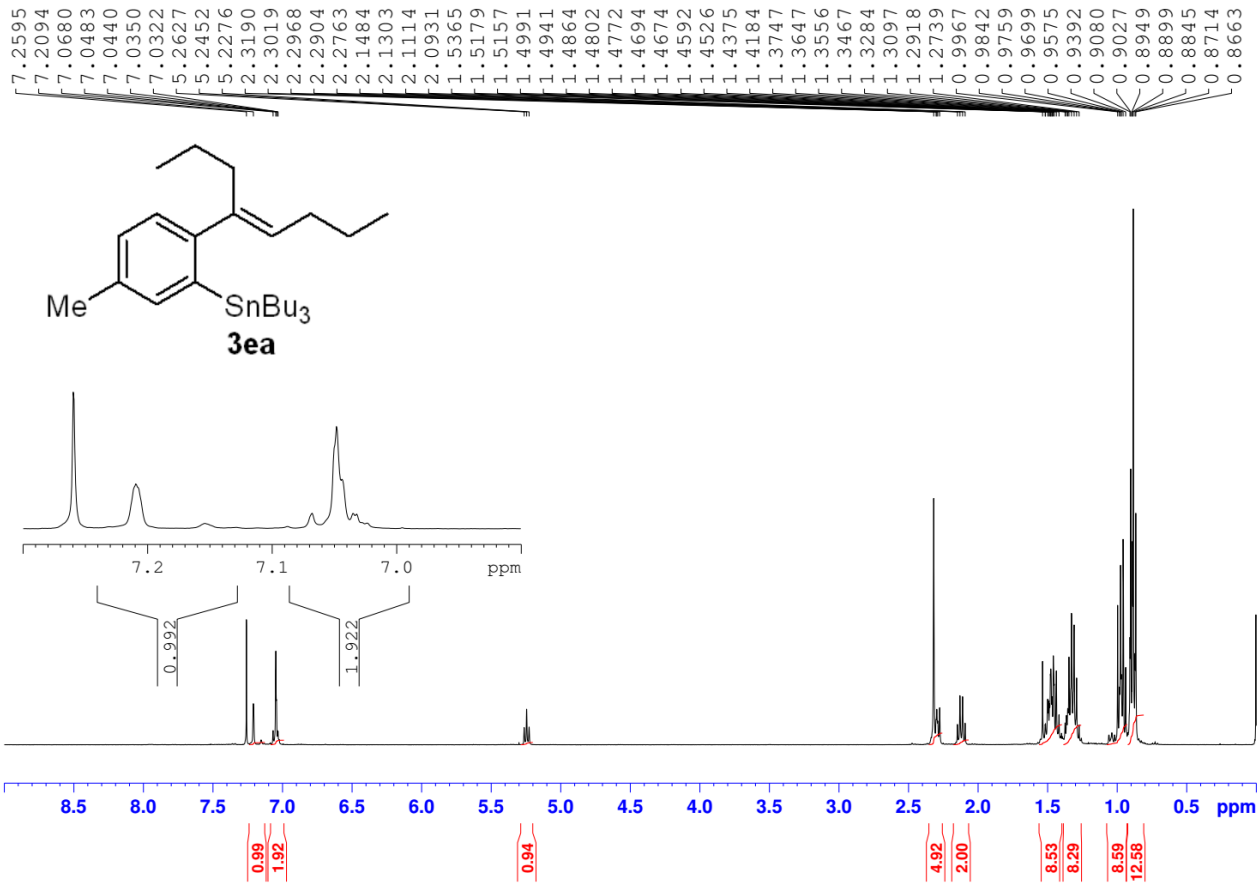
10
ppm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1.4694
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1.4375
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1.3467
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1.2739
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0.8899
0.8845
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0.8663
Me
SnBu3
3ea
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7.1
7.0
ppm
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6.0
5.5
5.0
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4.0
3.5
3.0
2.5
2.0
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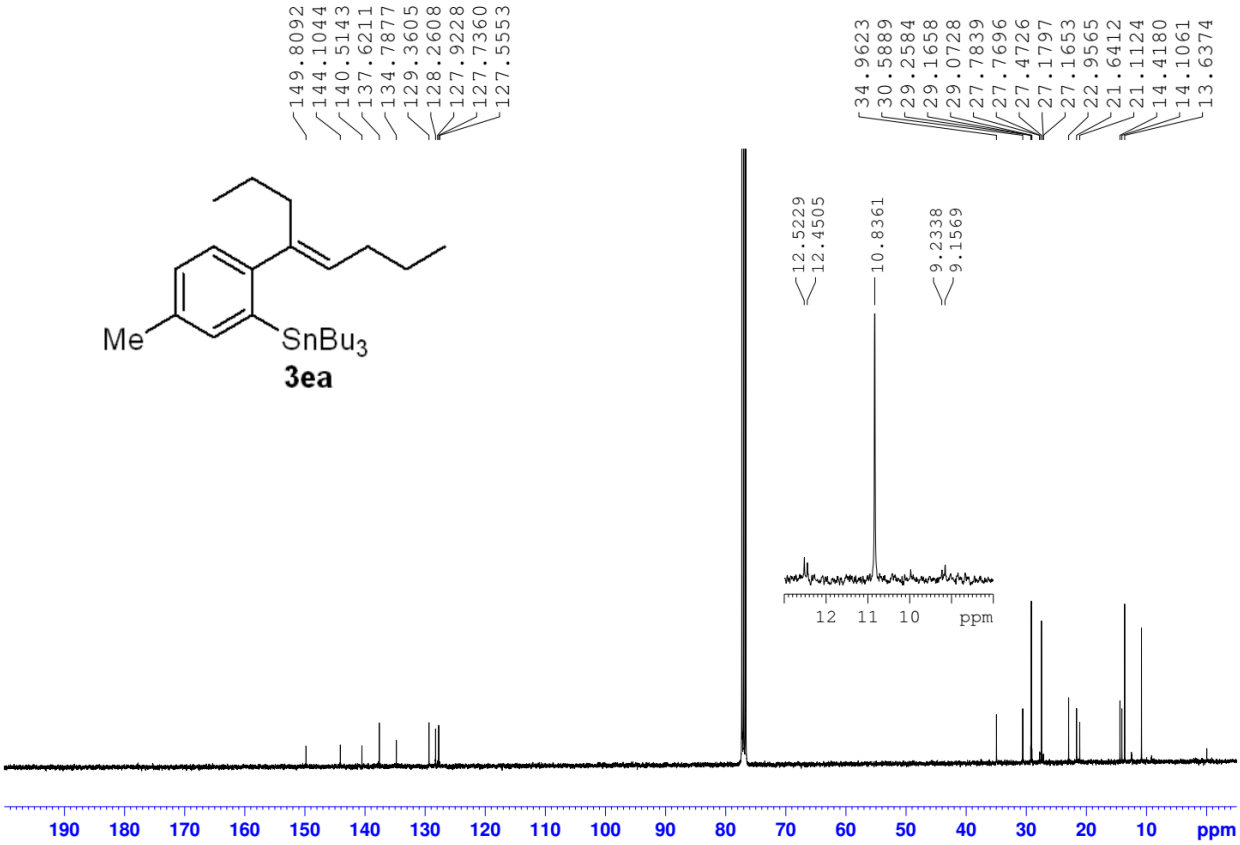
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27.4726
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27.1653
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21.6412
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13.6374
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12.4505
10.8361
9.2338
9.1569
Me
SnBu3
3ea
12
11
10
ppm
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150
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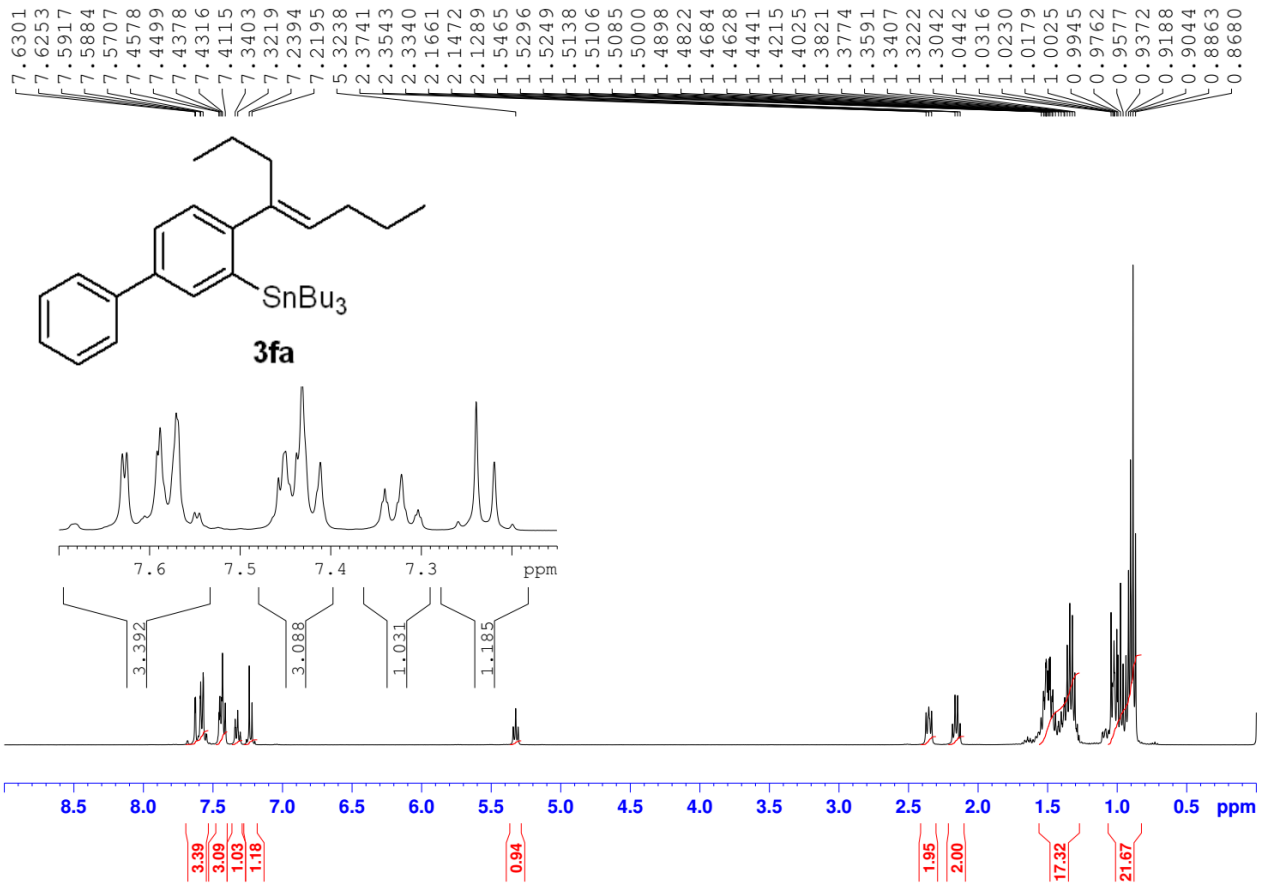
20
10
ppm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1.4025
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1.0179
1.0025
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SnBu3
3fa
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7.4
7.3
ppm
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7.0
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6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
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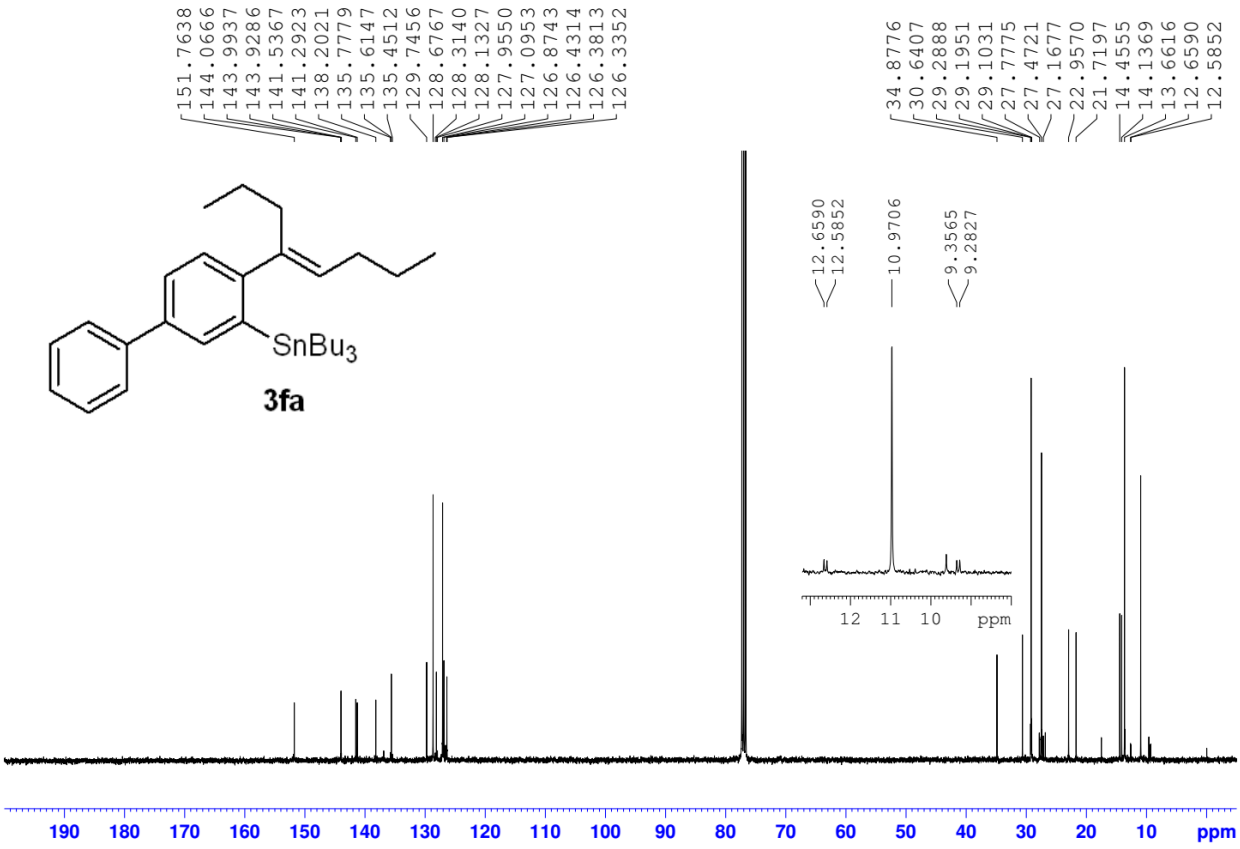
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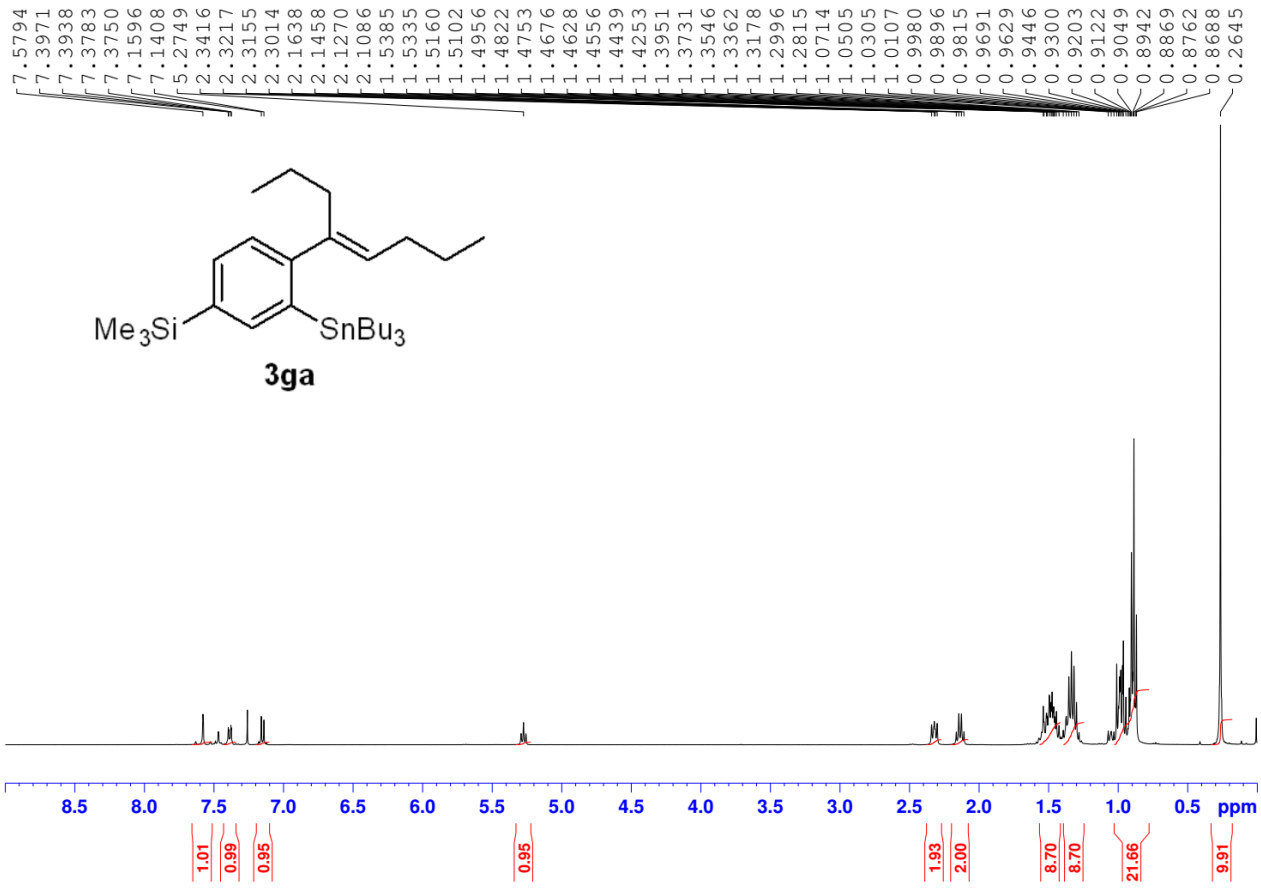
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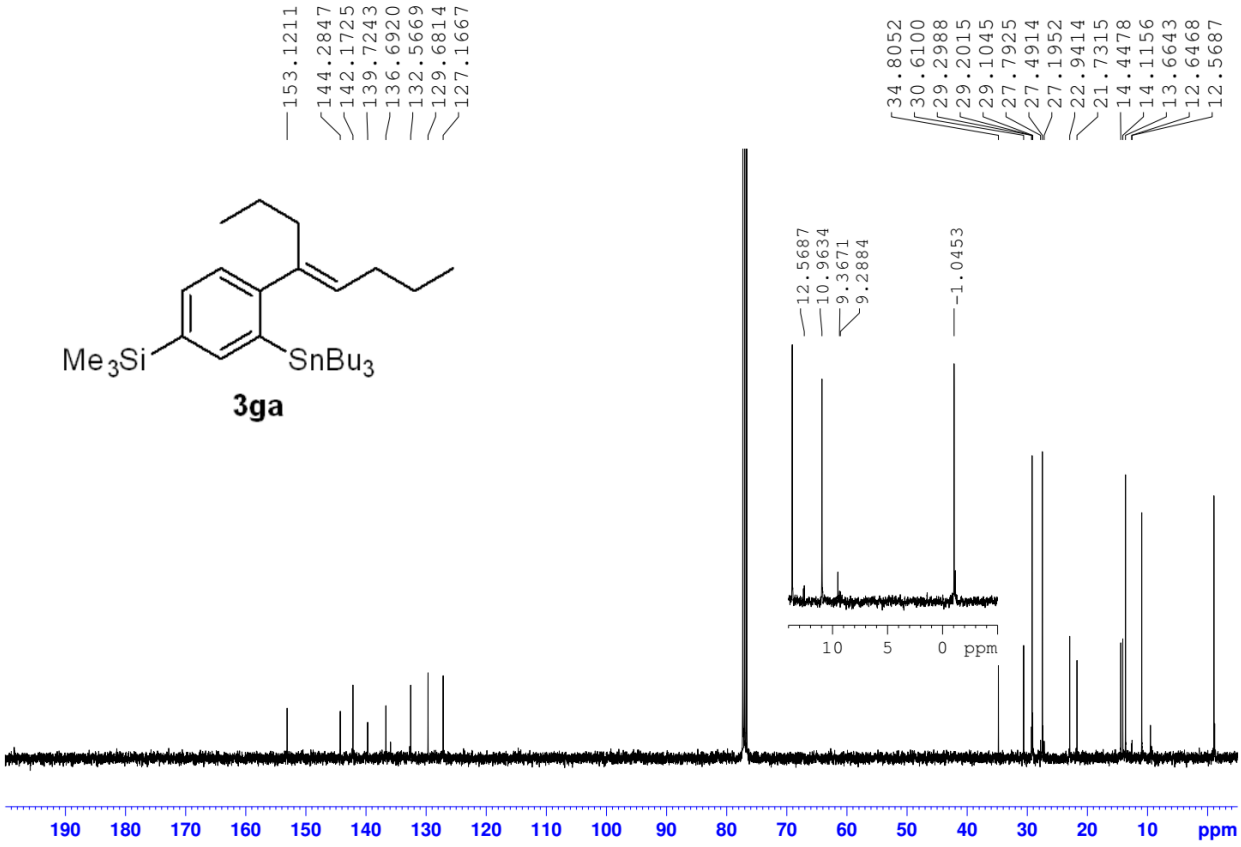
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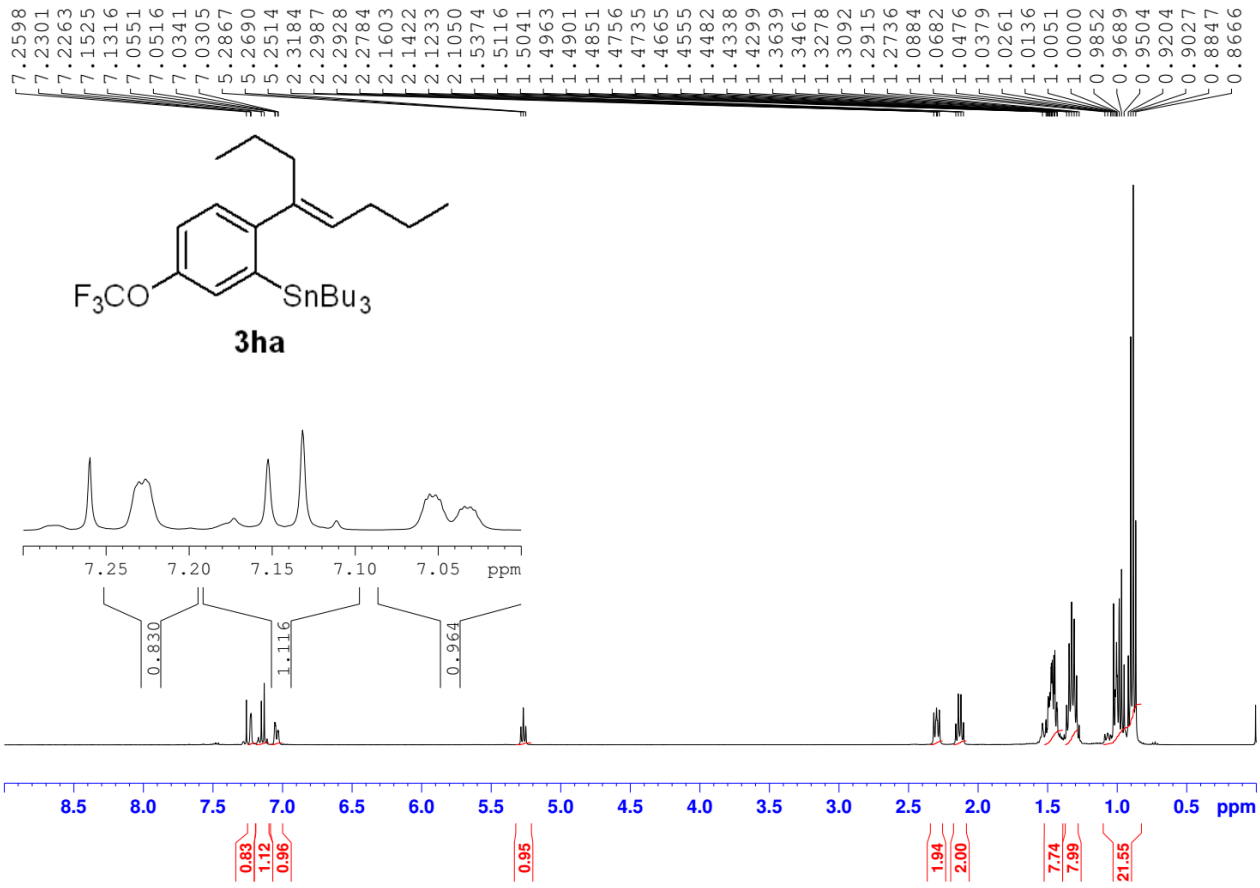
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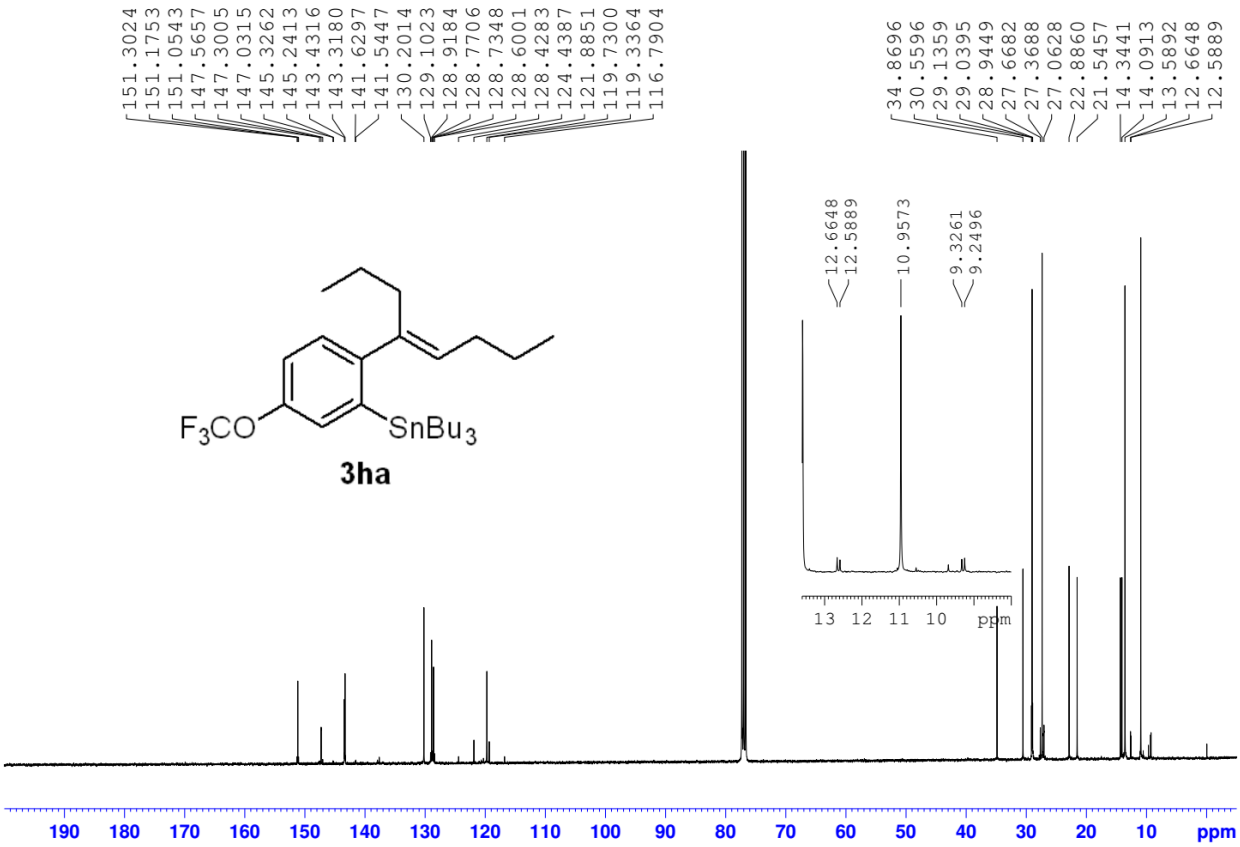
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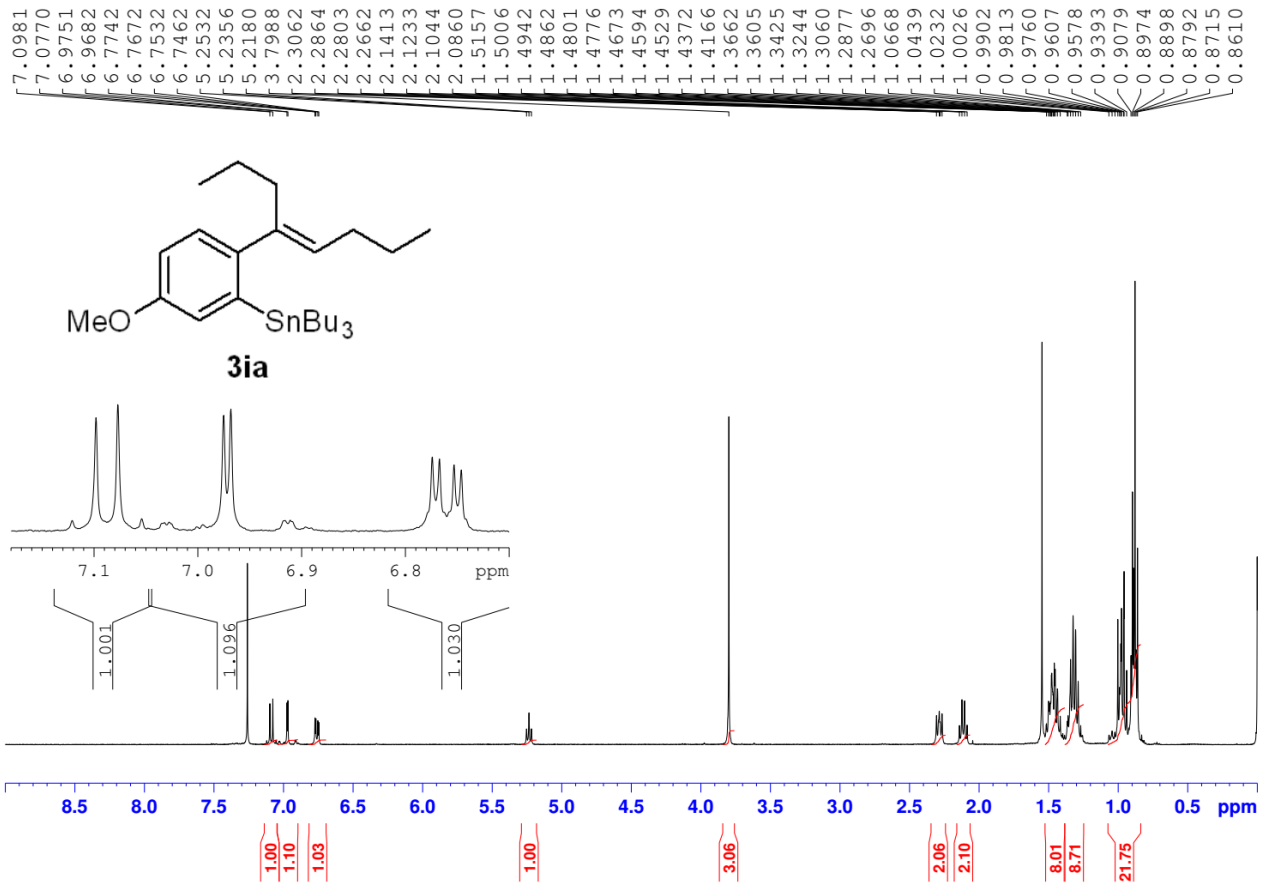
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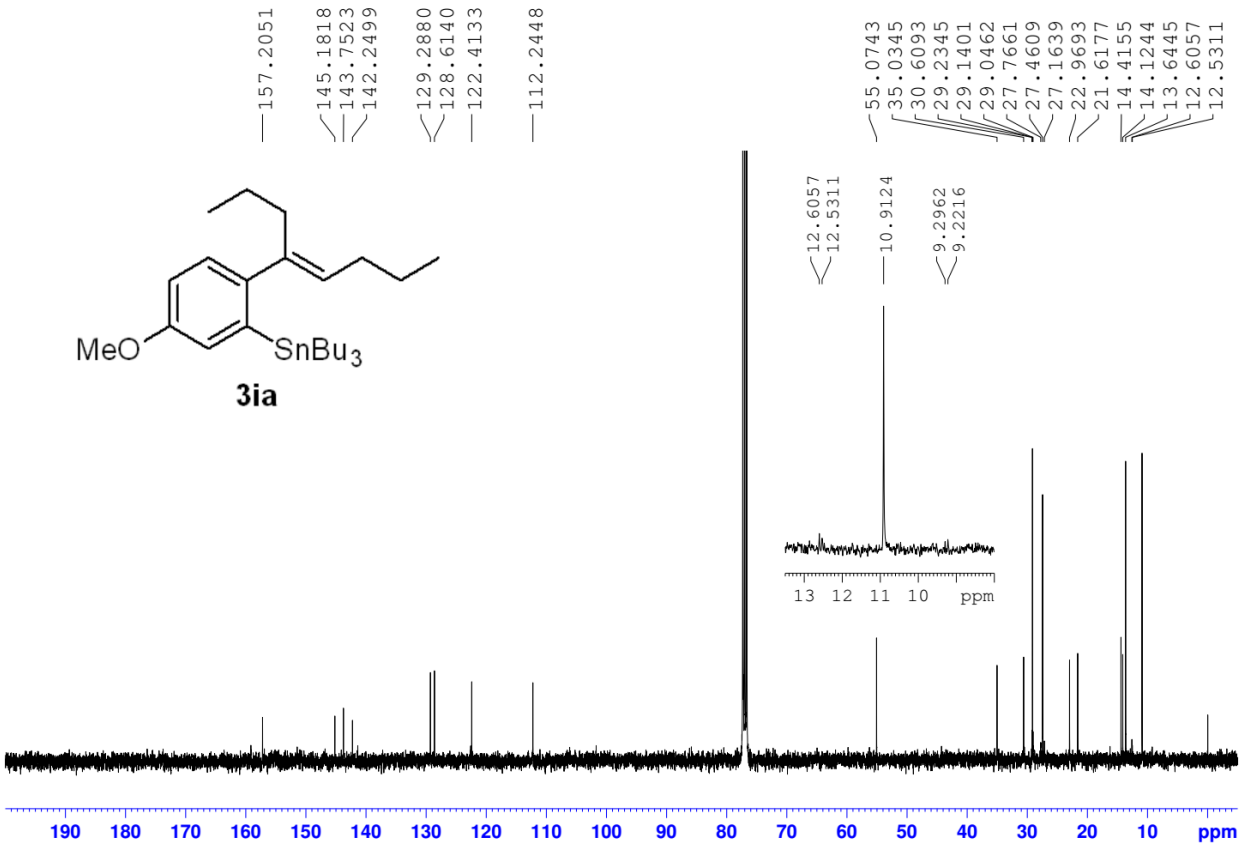
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12
11
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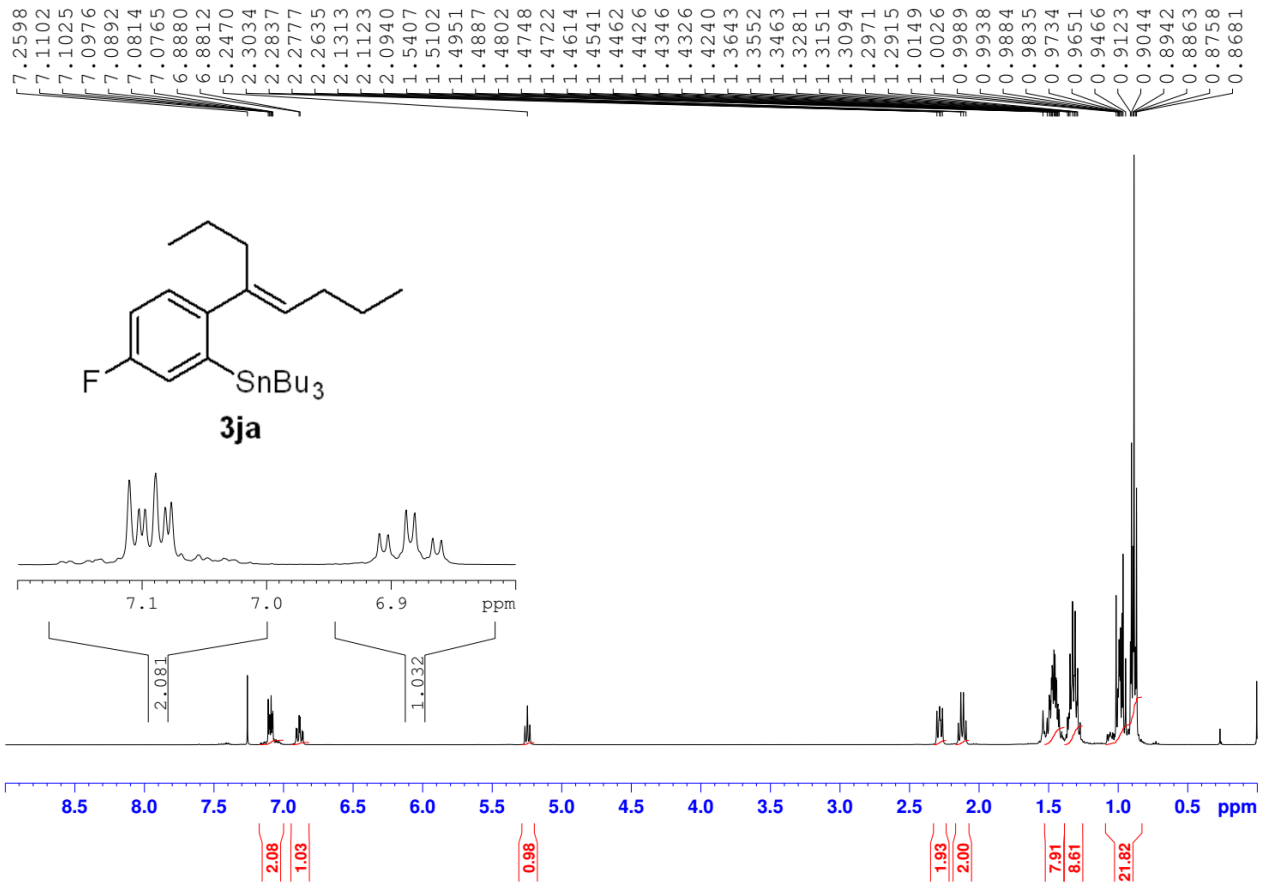
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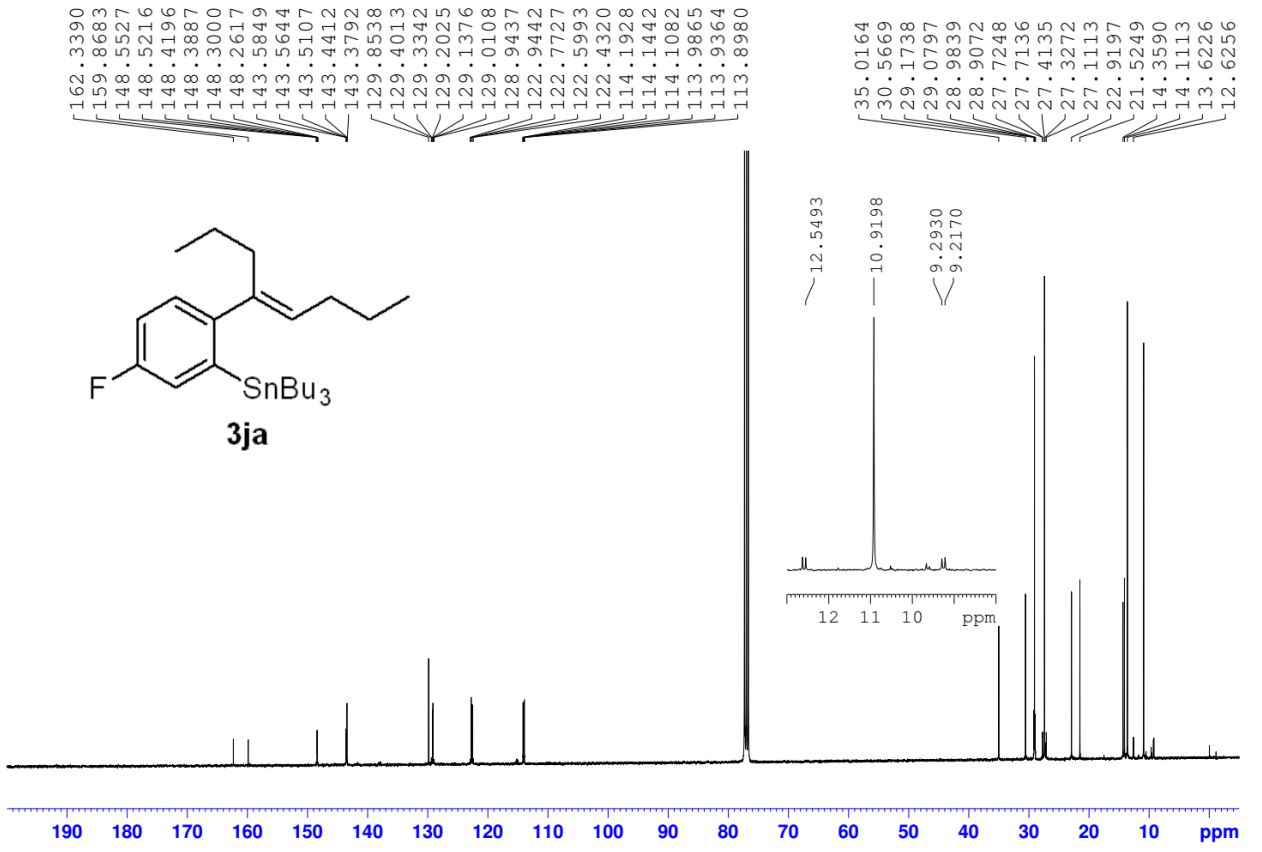
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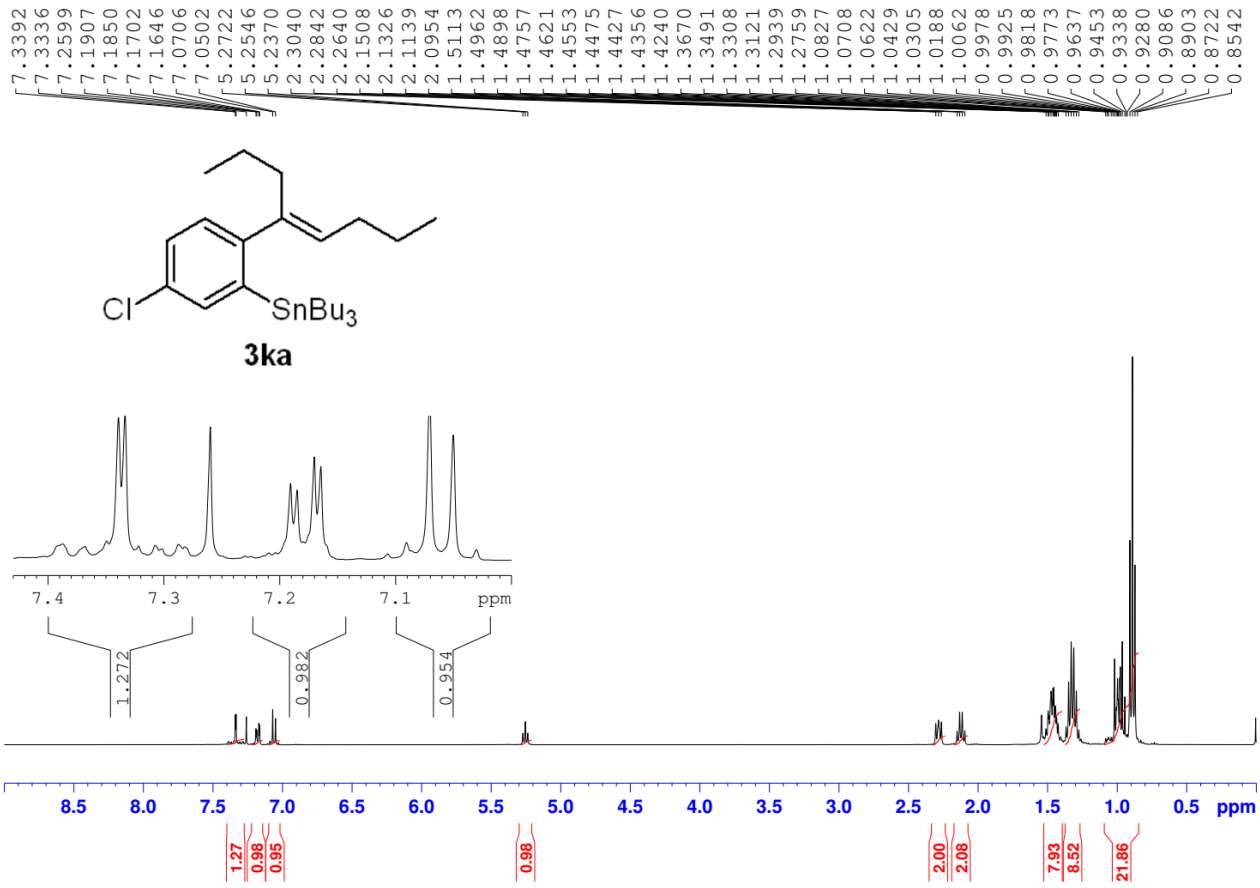
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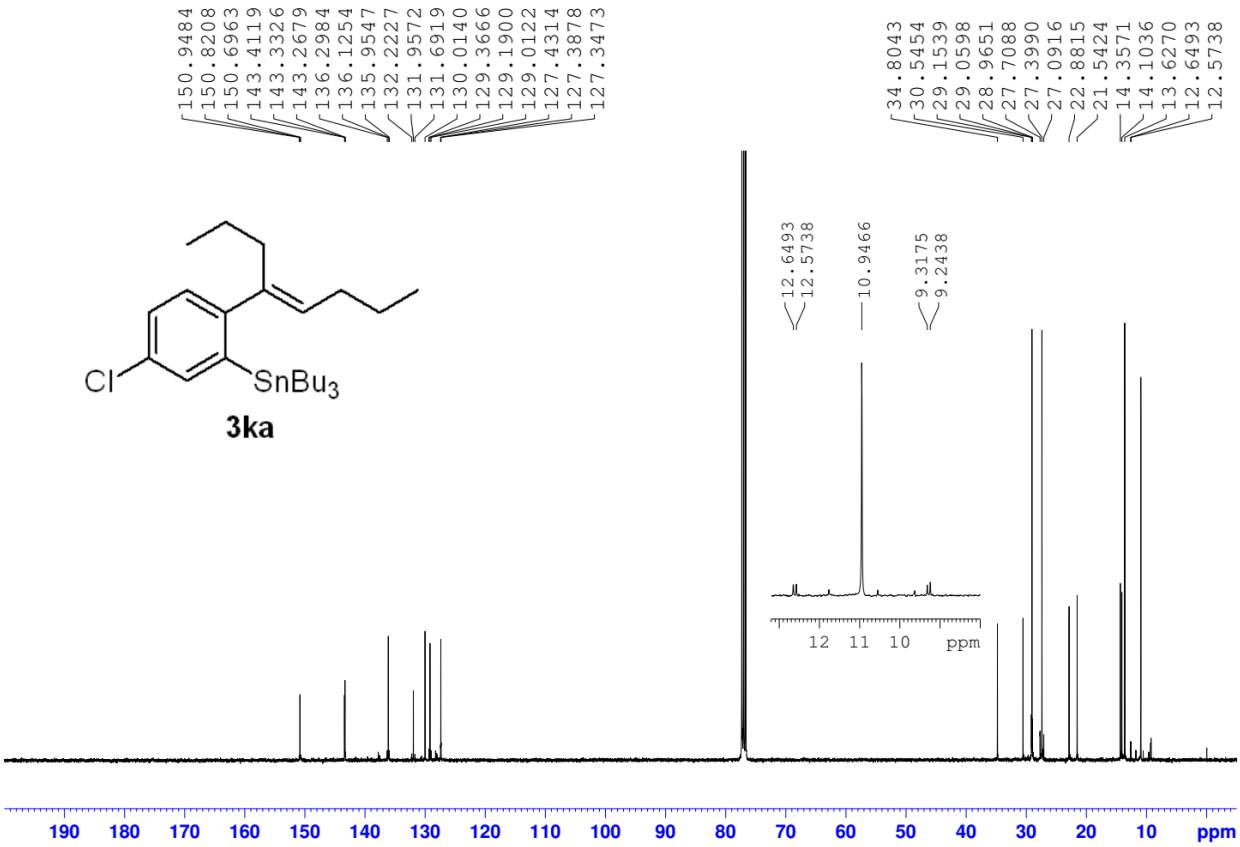
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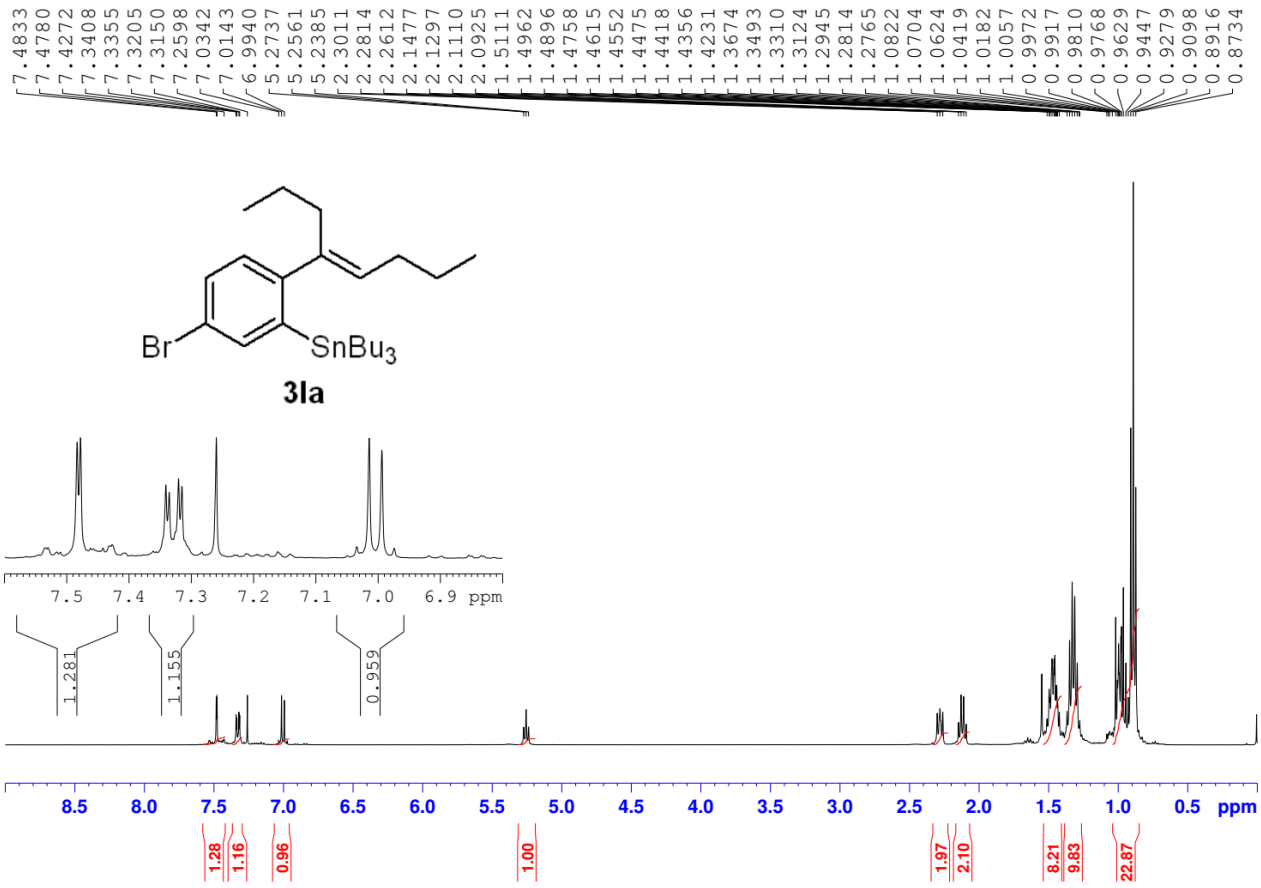
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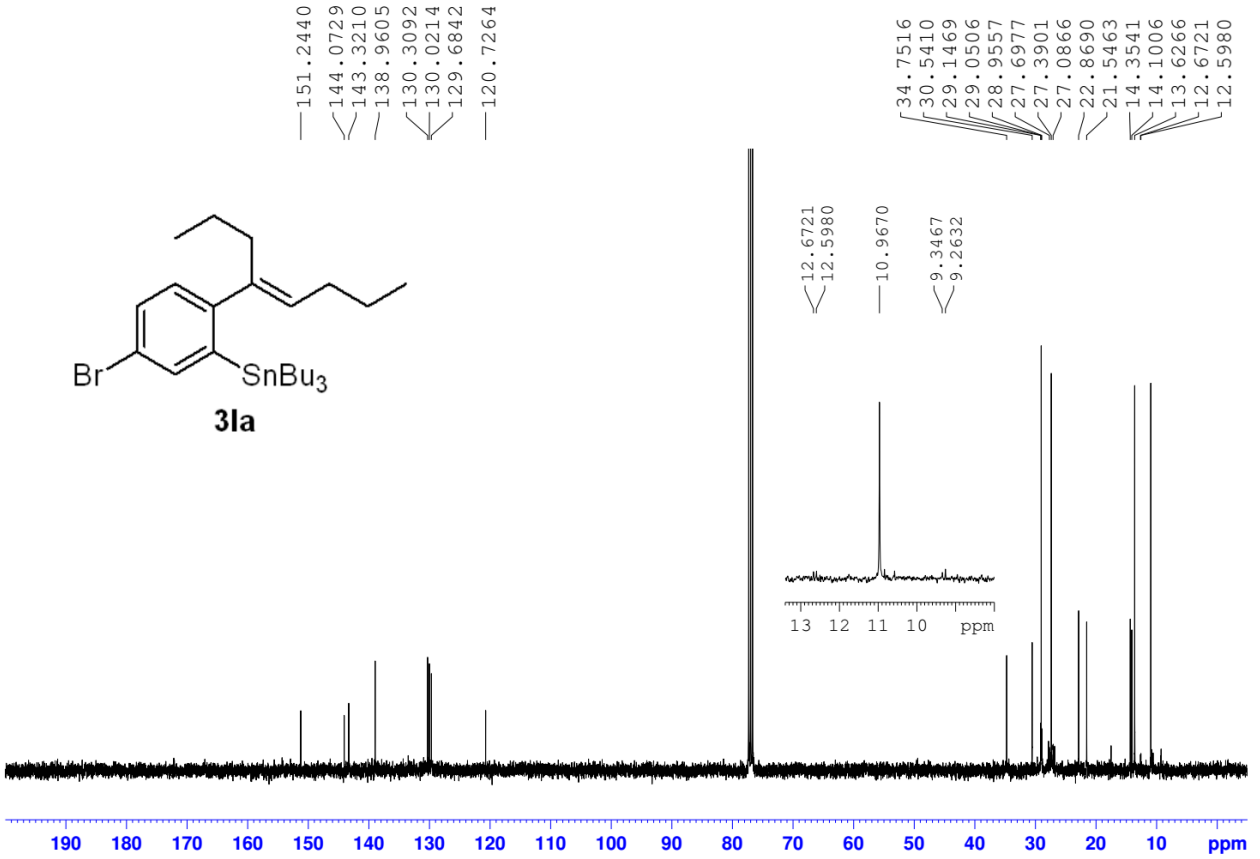
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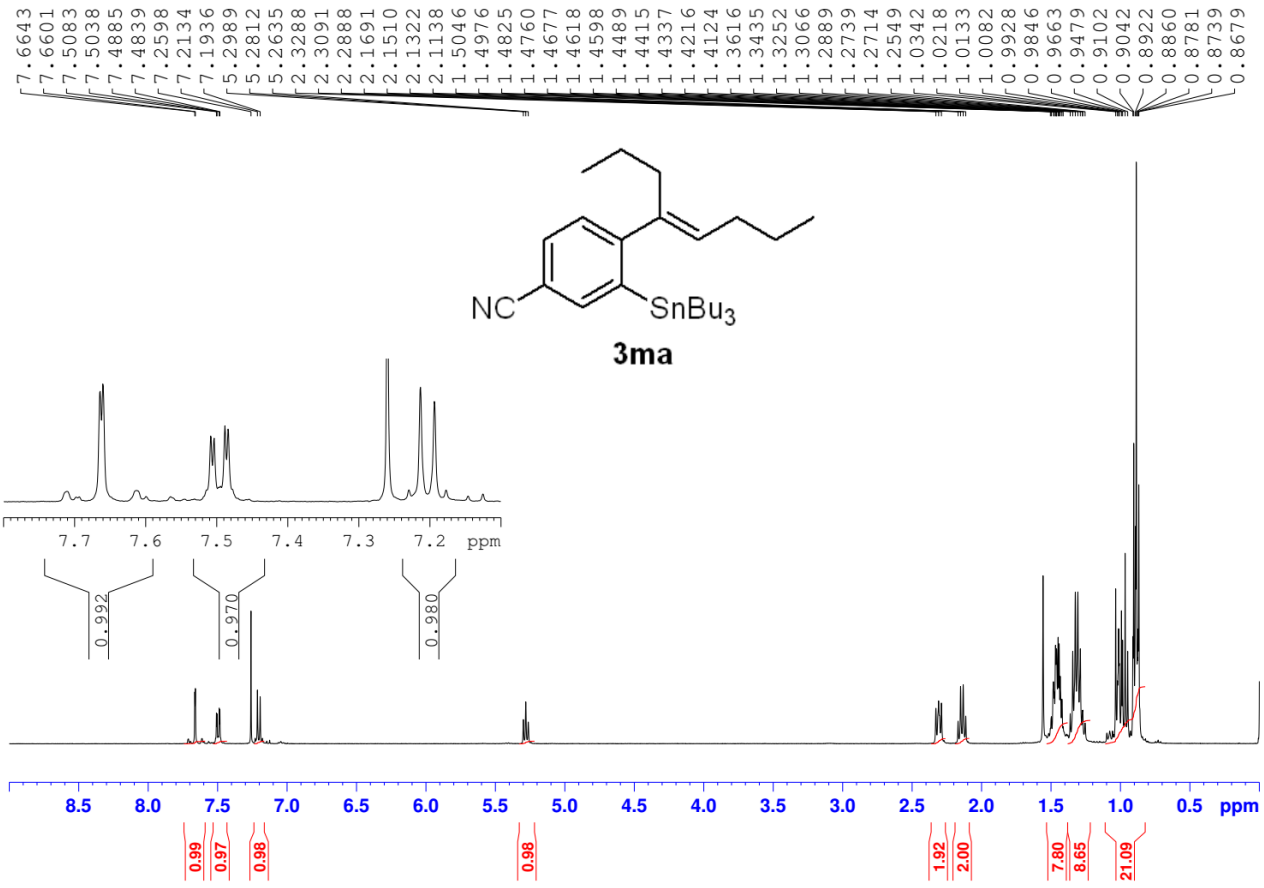
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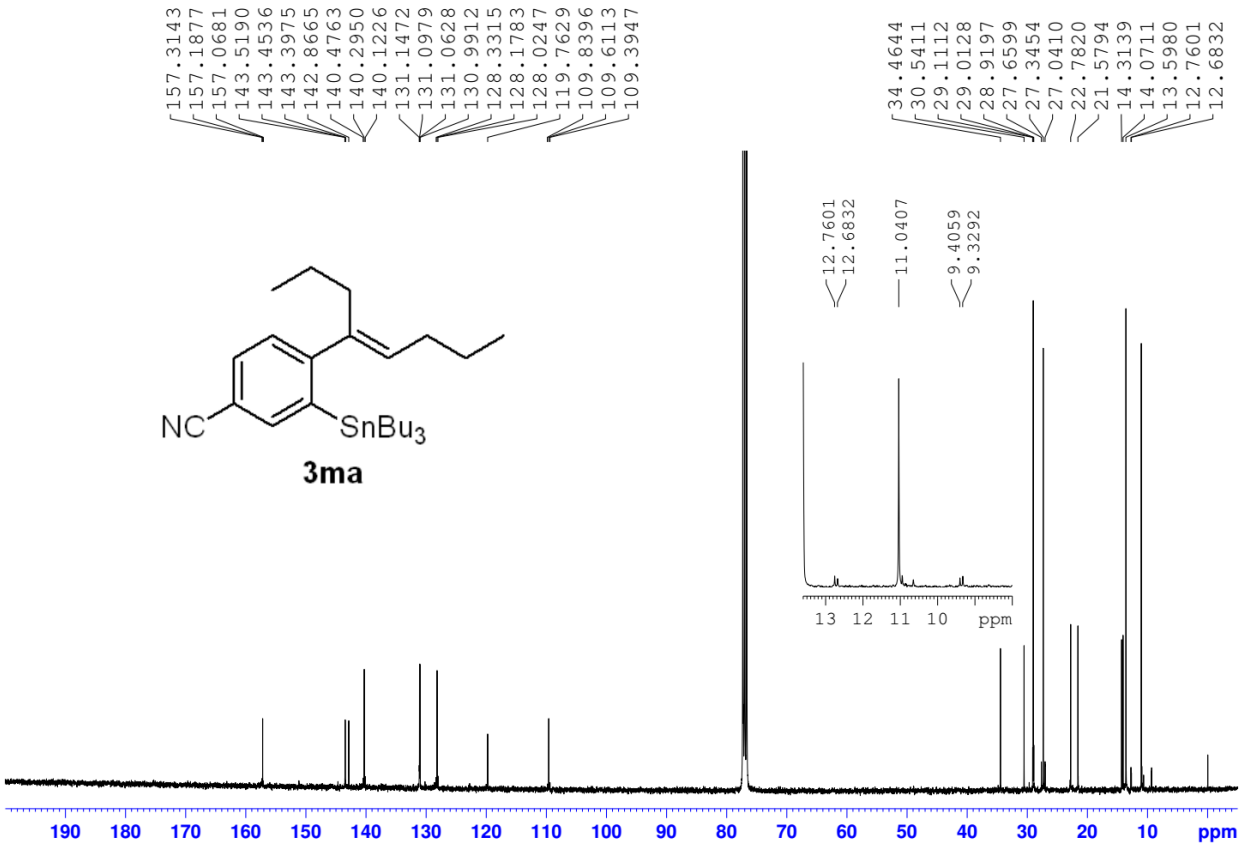
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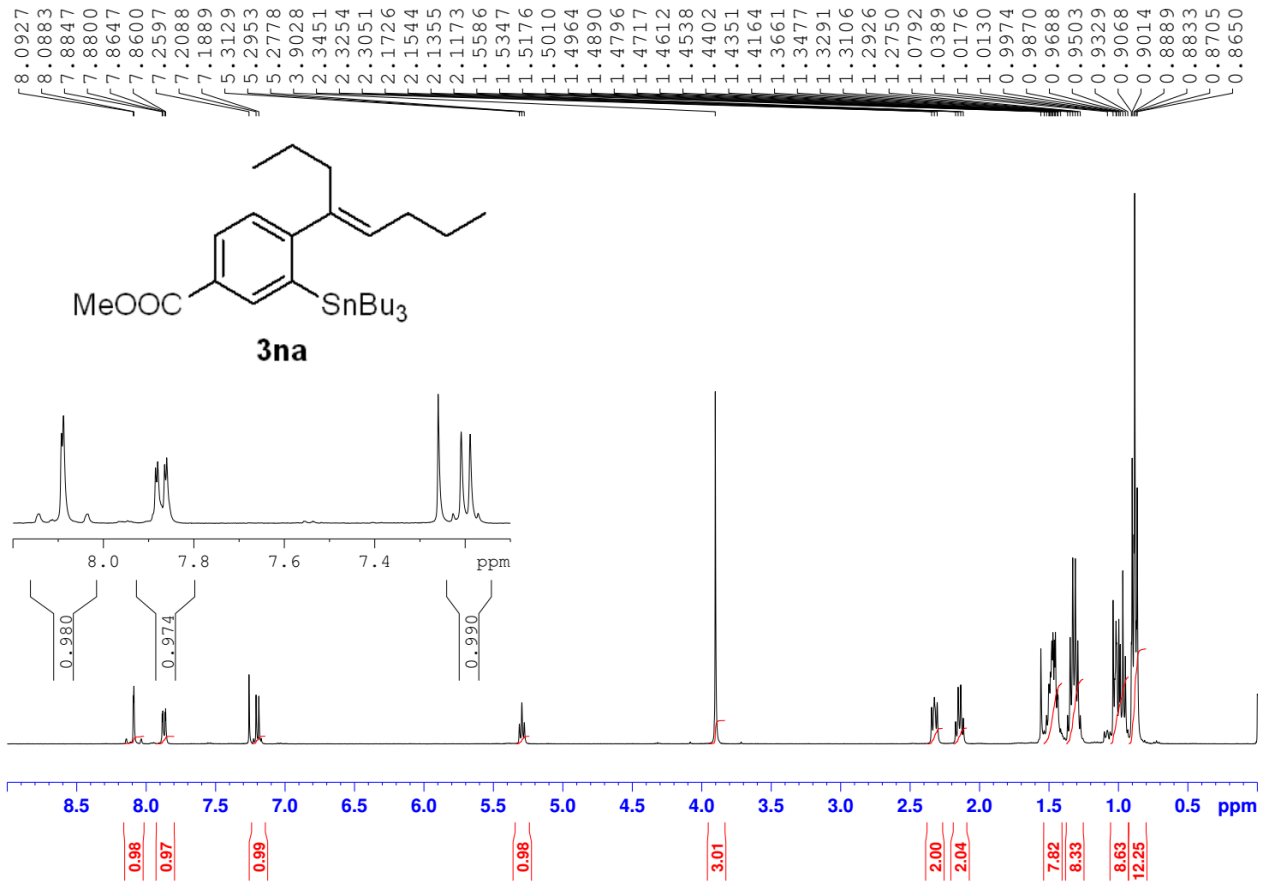
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MeOOC
SnBu3
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7.4
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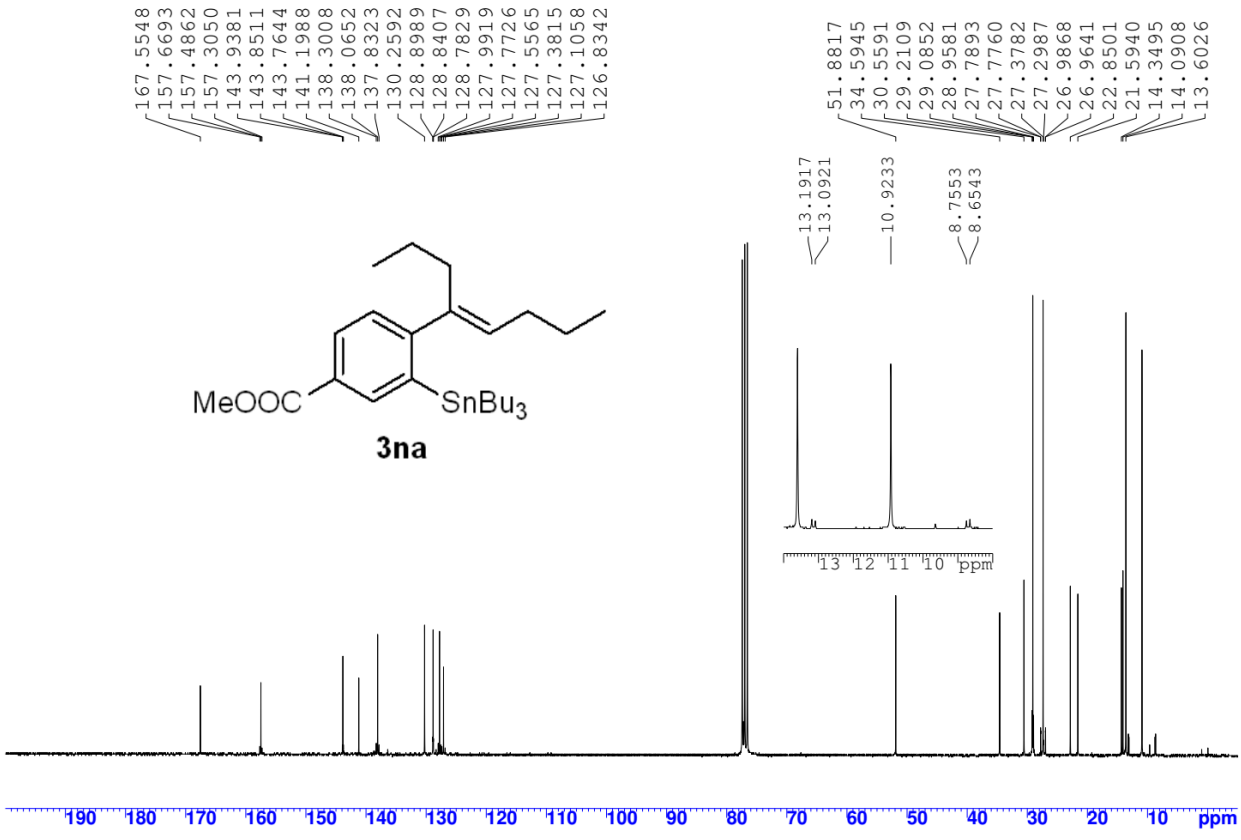
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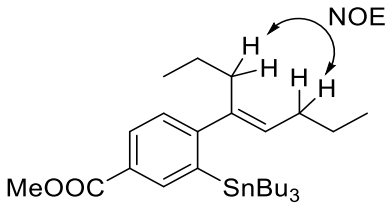

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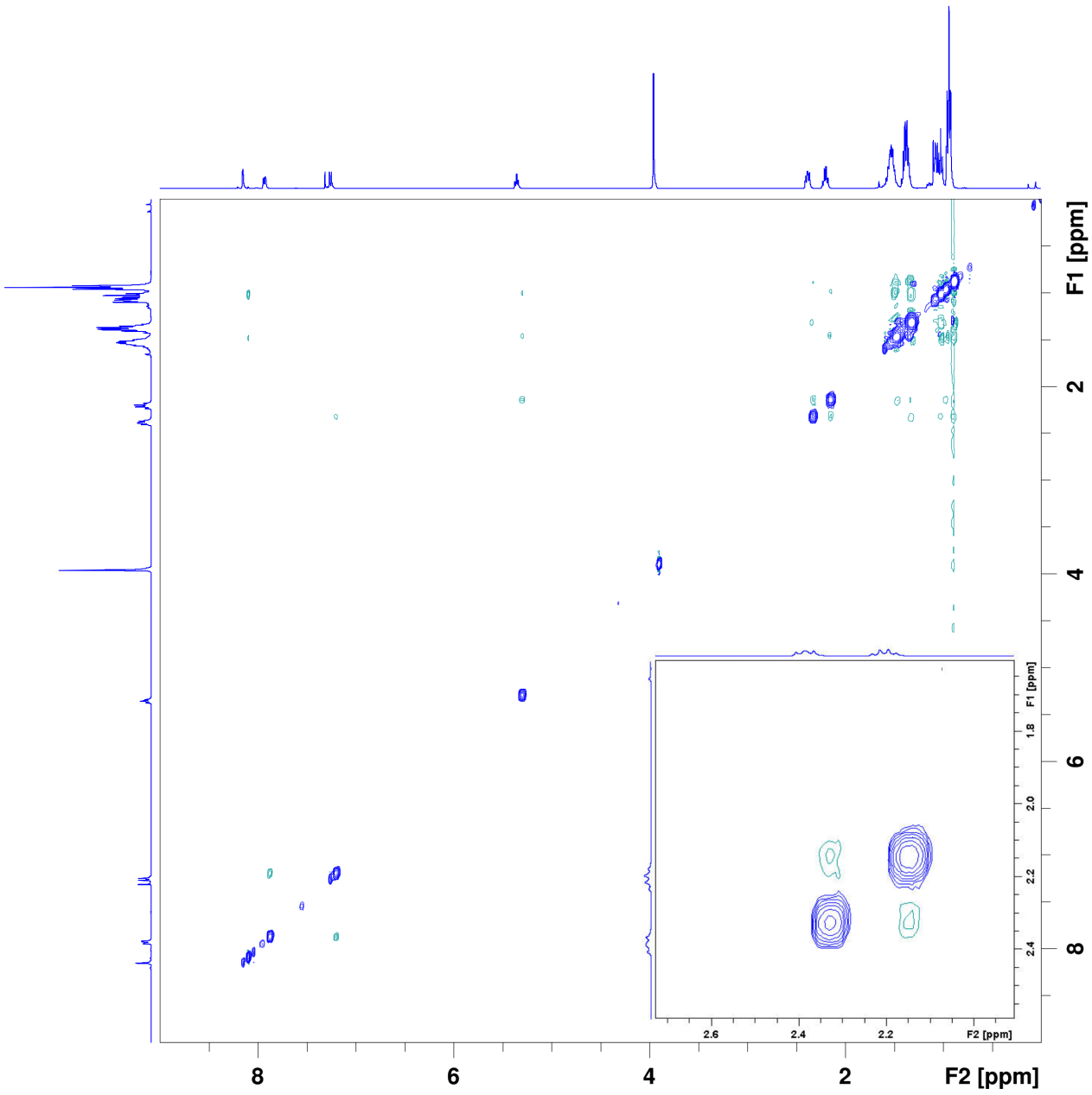

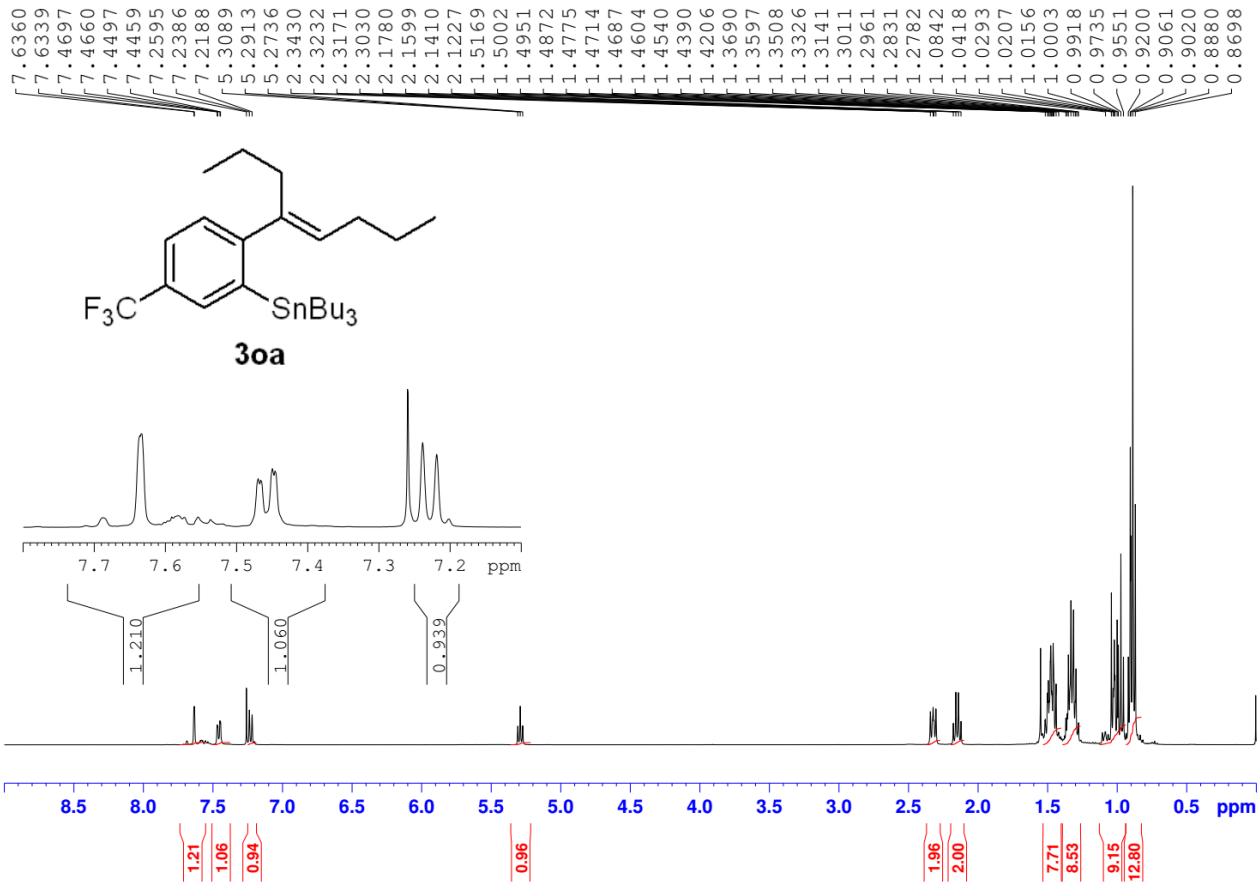
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7.3
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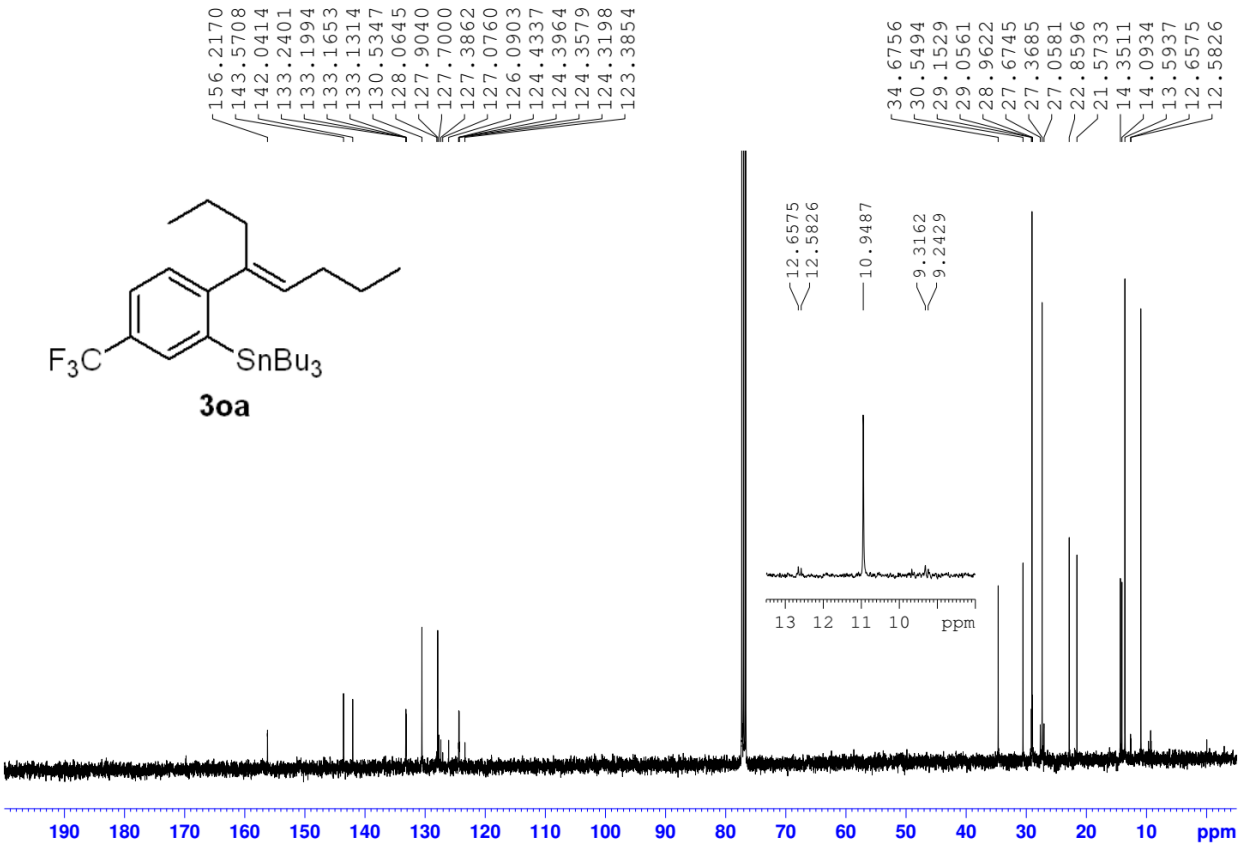
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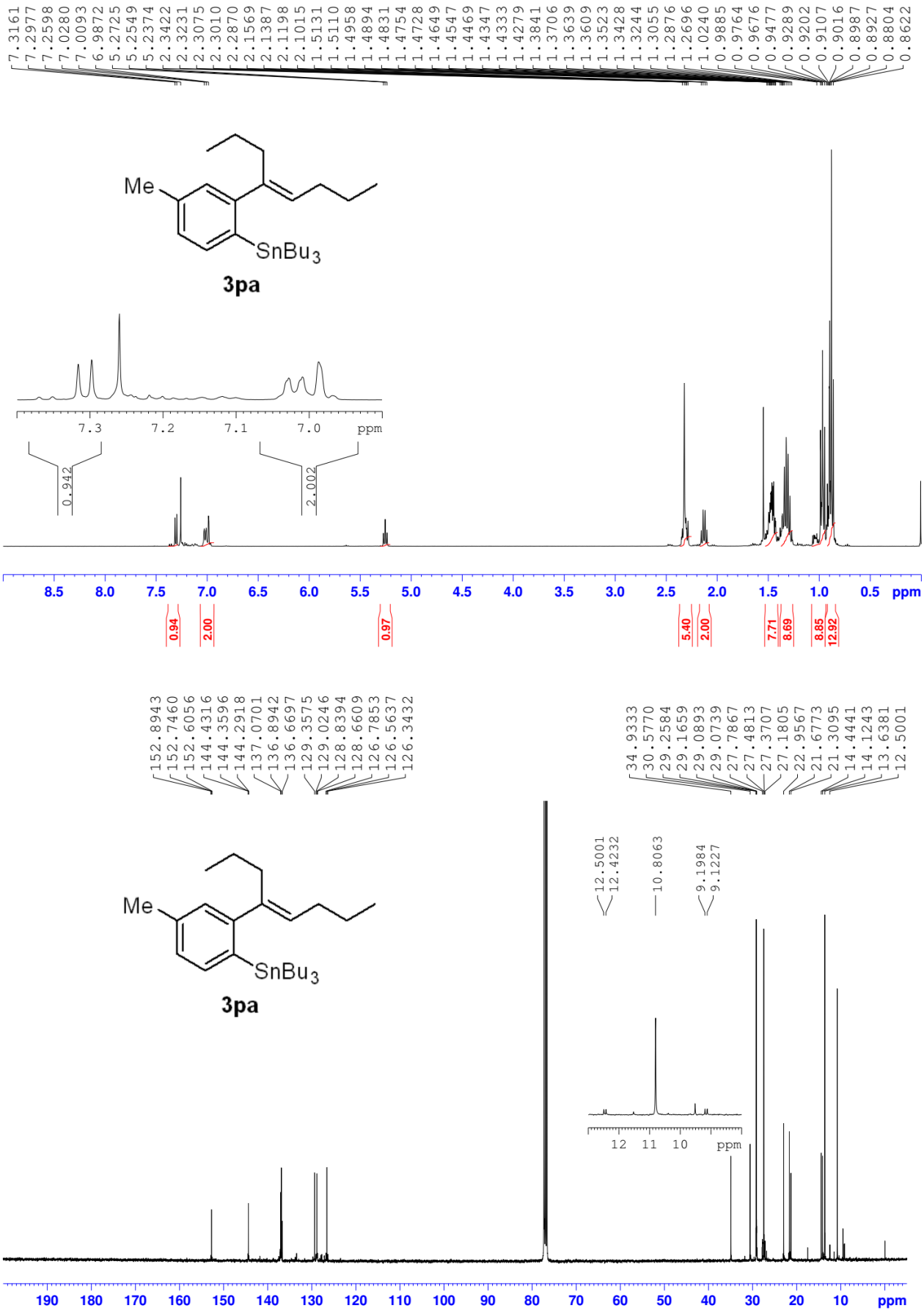
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Me
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1.5
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10
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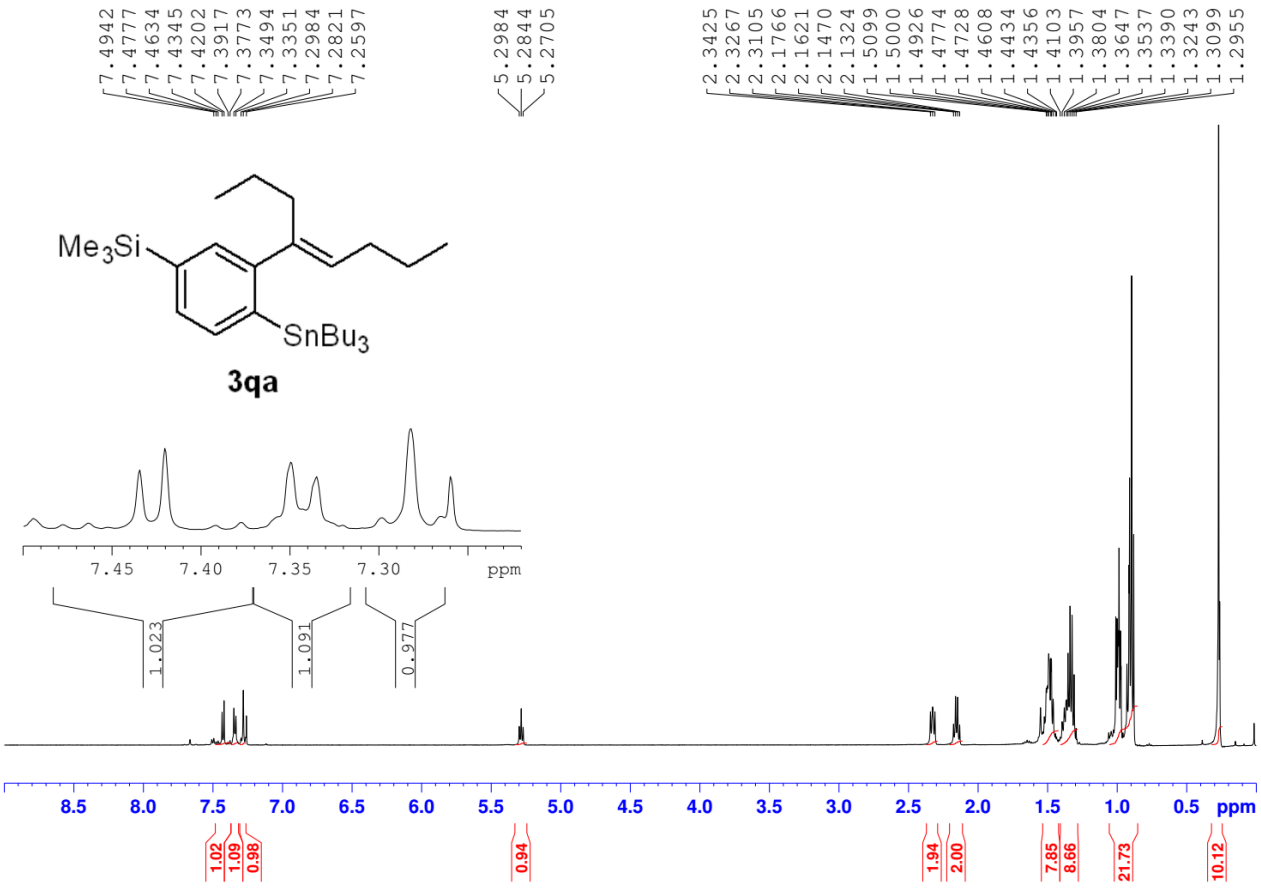
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5.2844
5.2705
2.3425
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2.1324
1.5099
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1.3099
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SnBu3
3qa
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7.35
7.30
ppm
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7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
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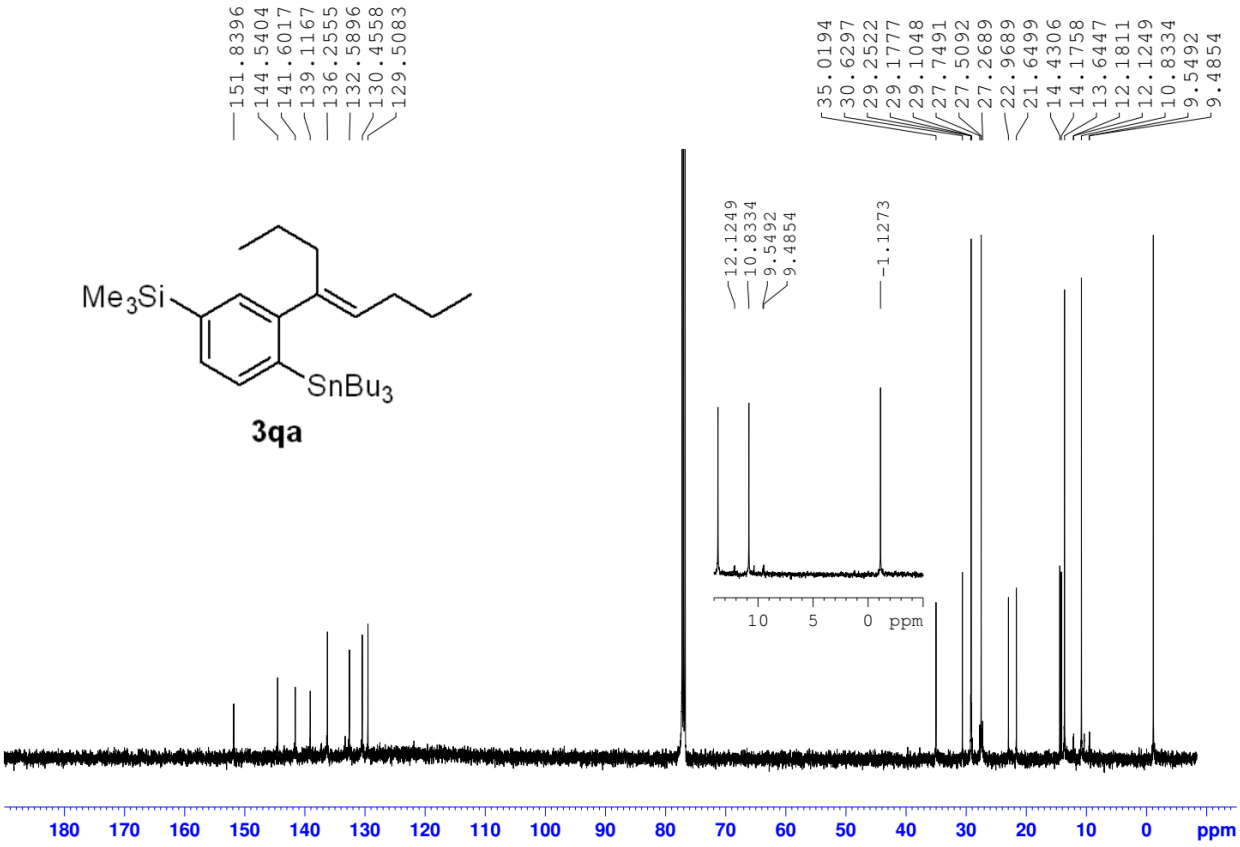
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SnBu3
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10
5
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20
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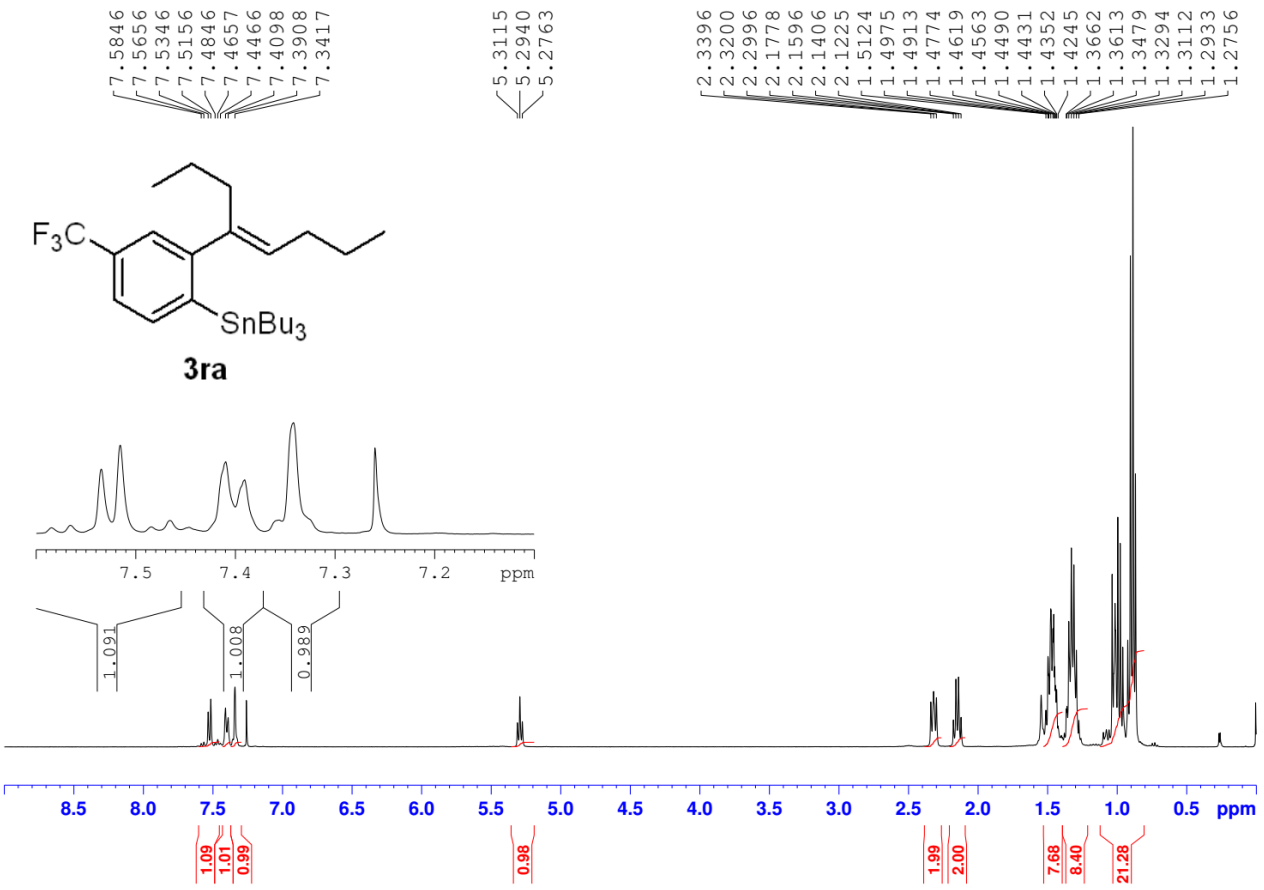
ppm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F3C
SnBu3
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7.4
7.3
7.2
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6.0
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4.0
3.5
3.0
2.5
2.0
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ppm
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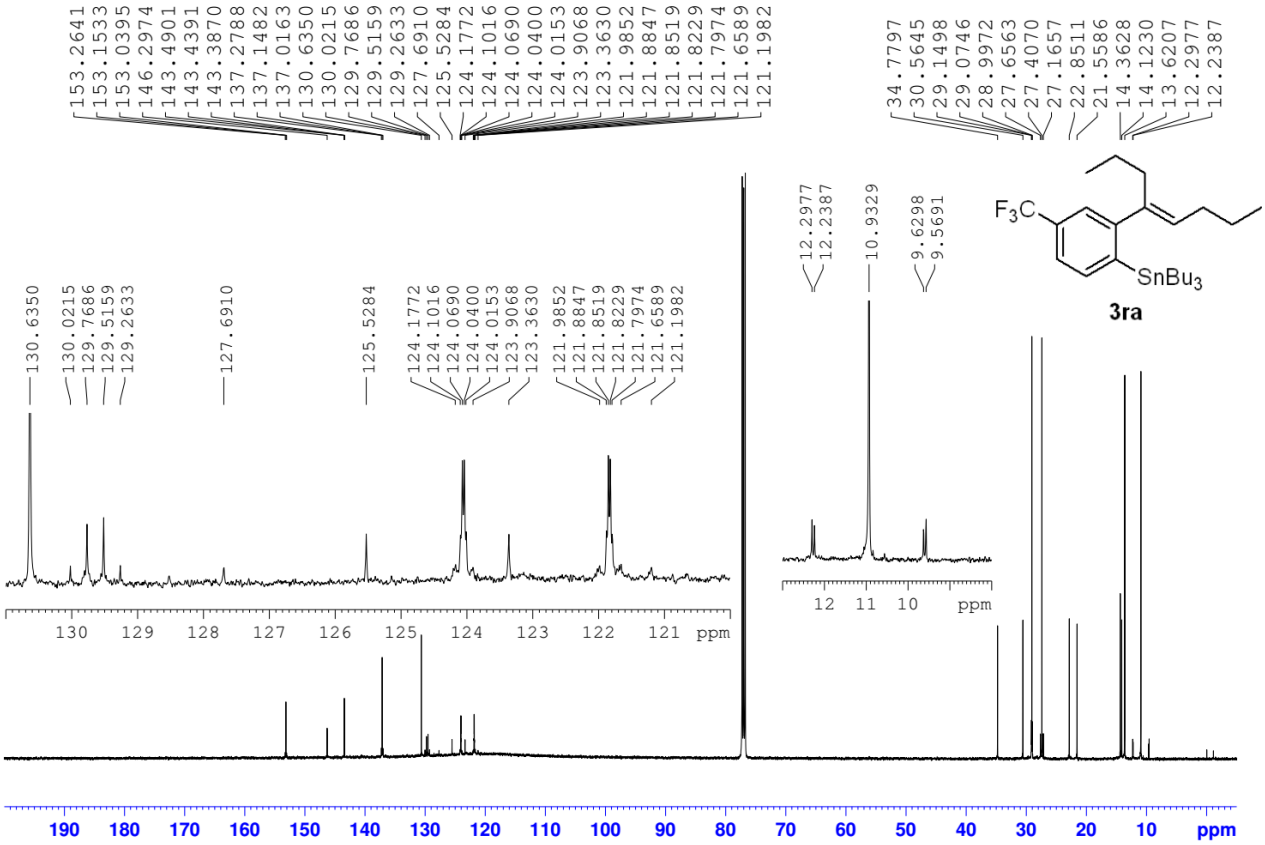
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128
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124
123
122
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10
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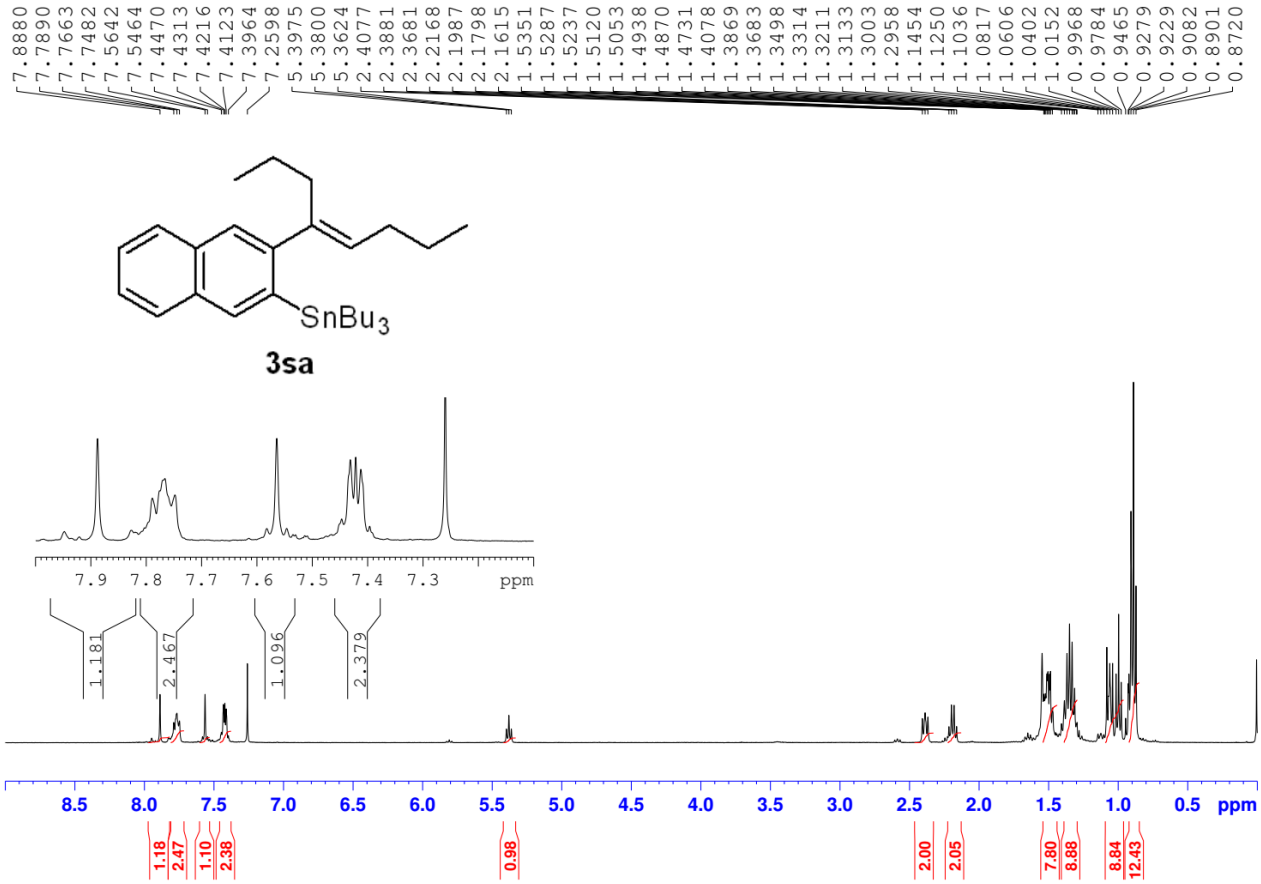
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0.8720
SnBu3
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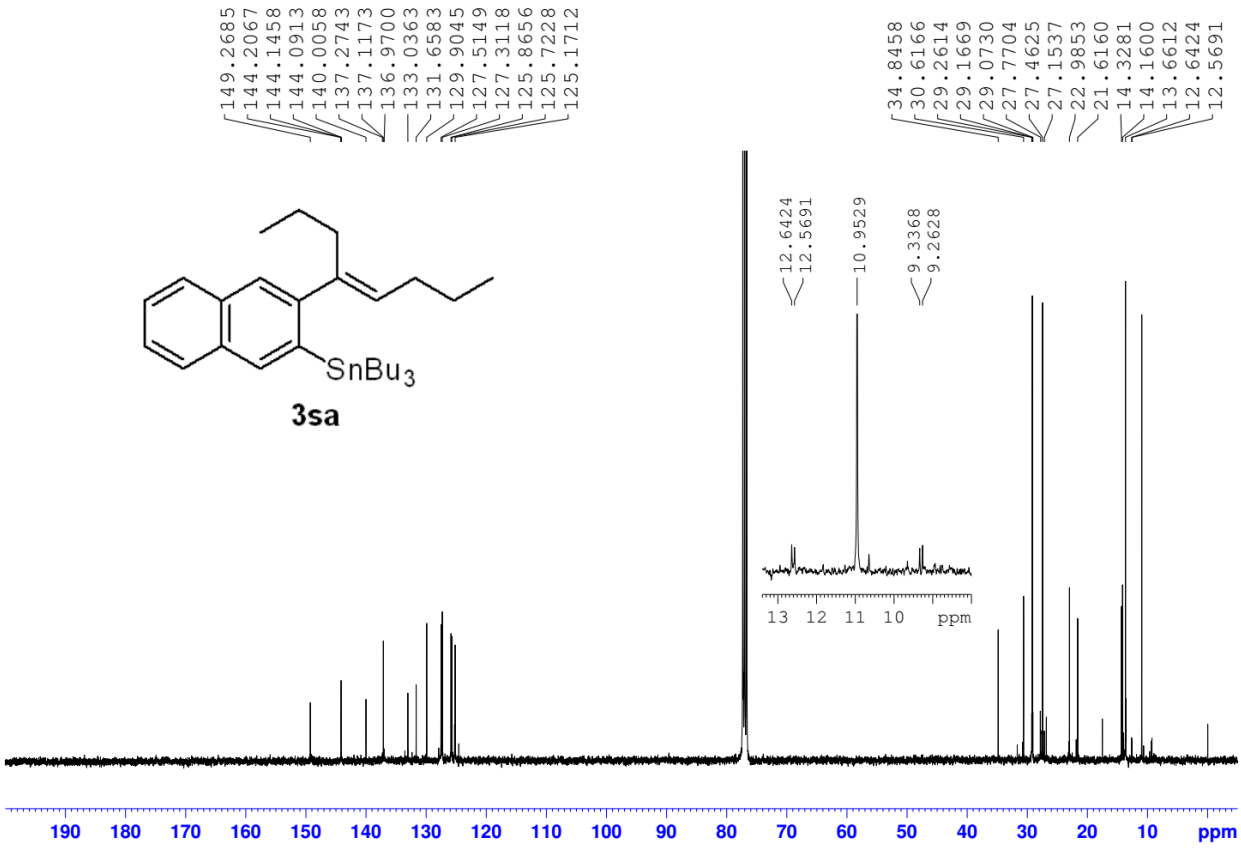
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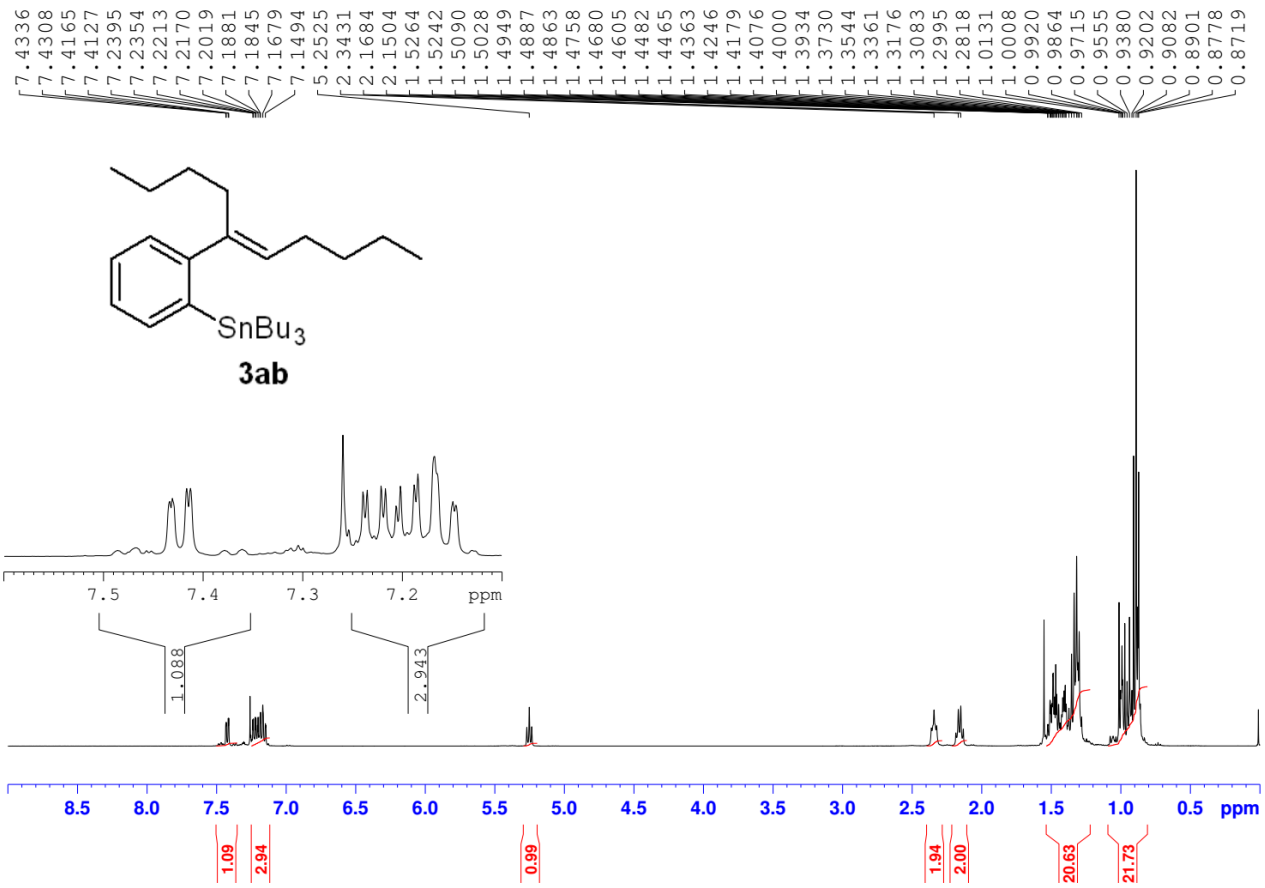
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7.2
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5.0
4.5
4.0
3.5
3.0
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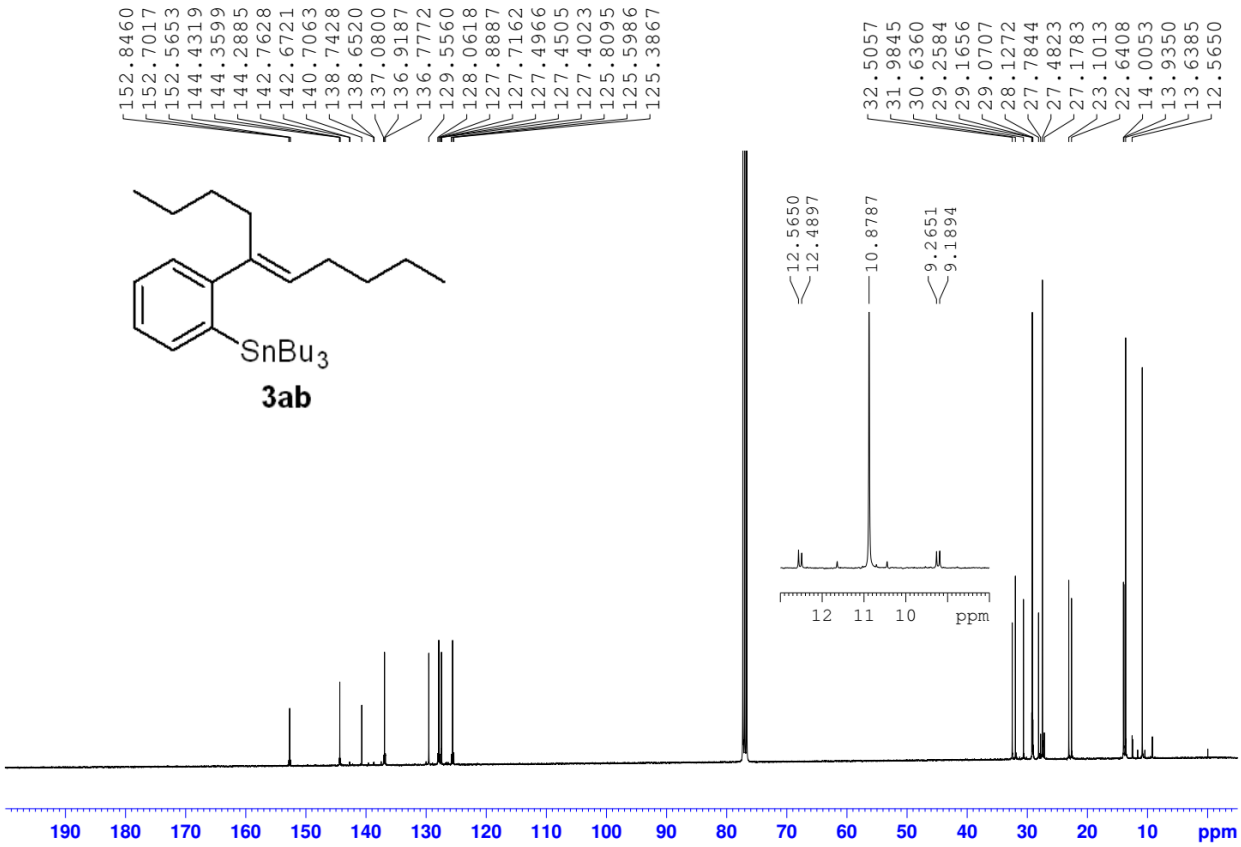
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10
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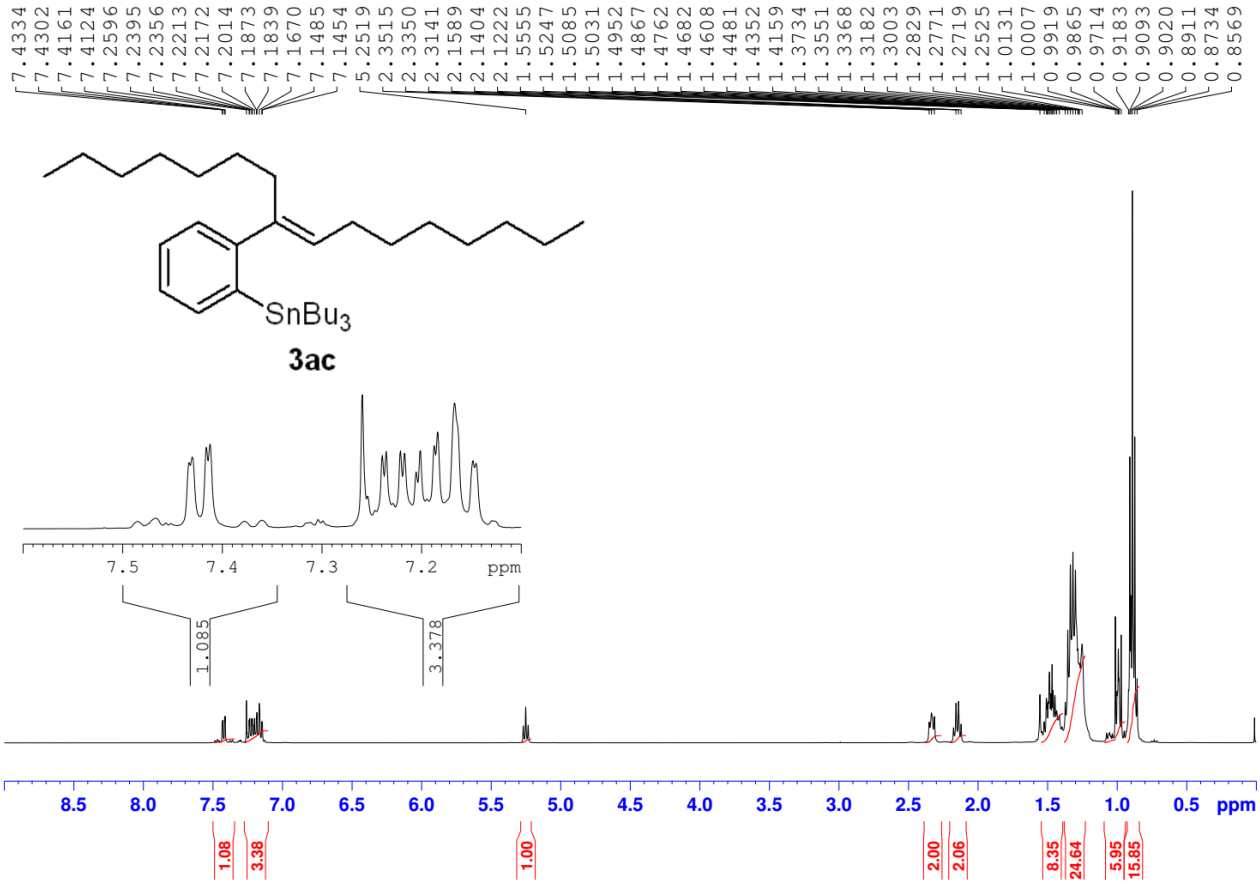
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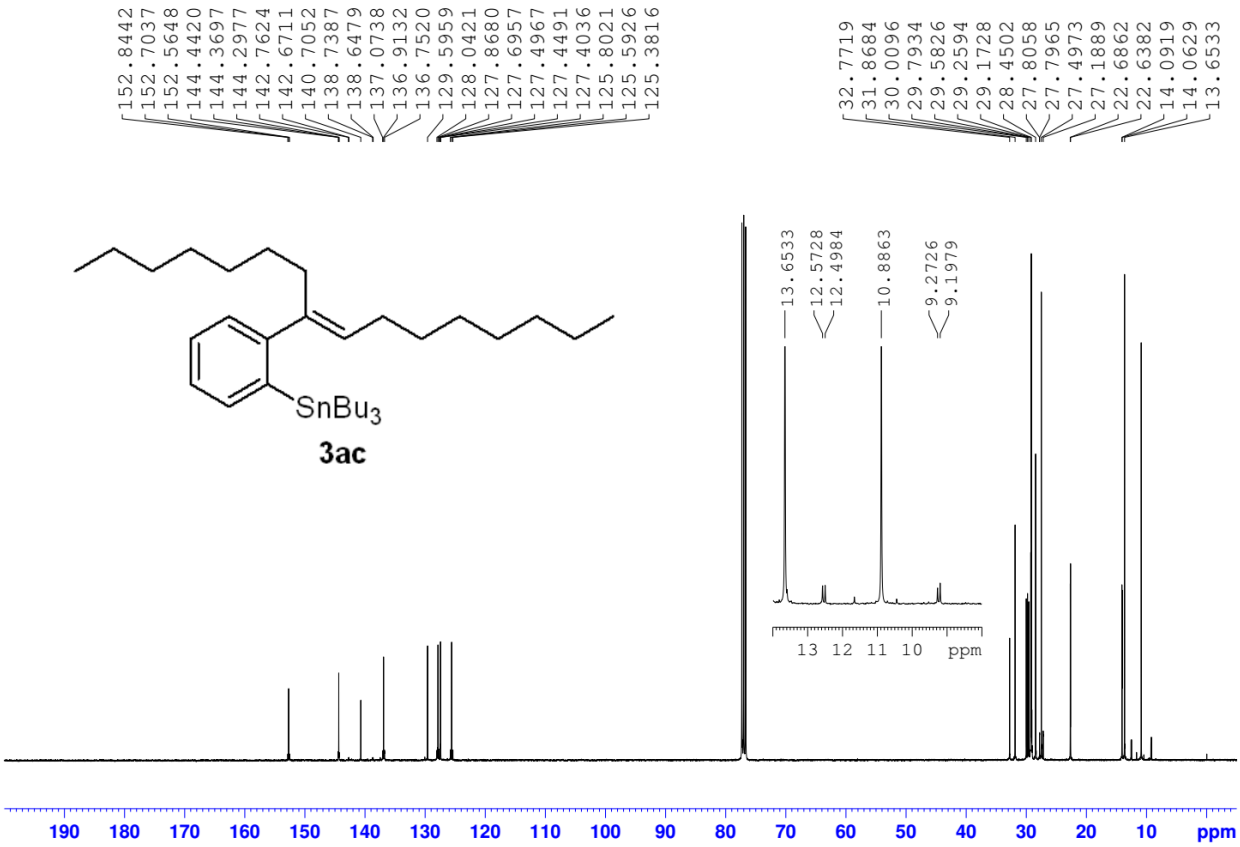
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29.2594
29.1728
28.4502
27.8058
27.7965
27.4973
27.1889
22.6862
22.6382
14.0919
14.0629
13.6533
SnBu3
3ac
13.6533
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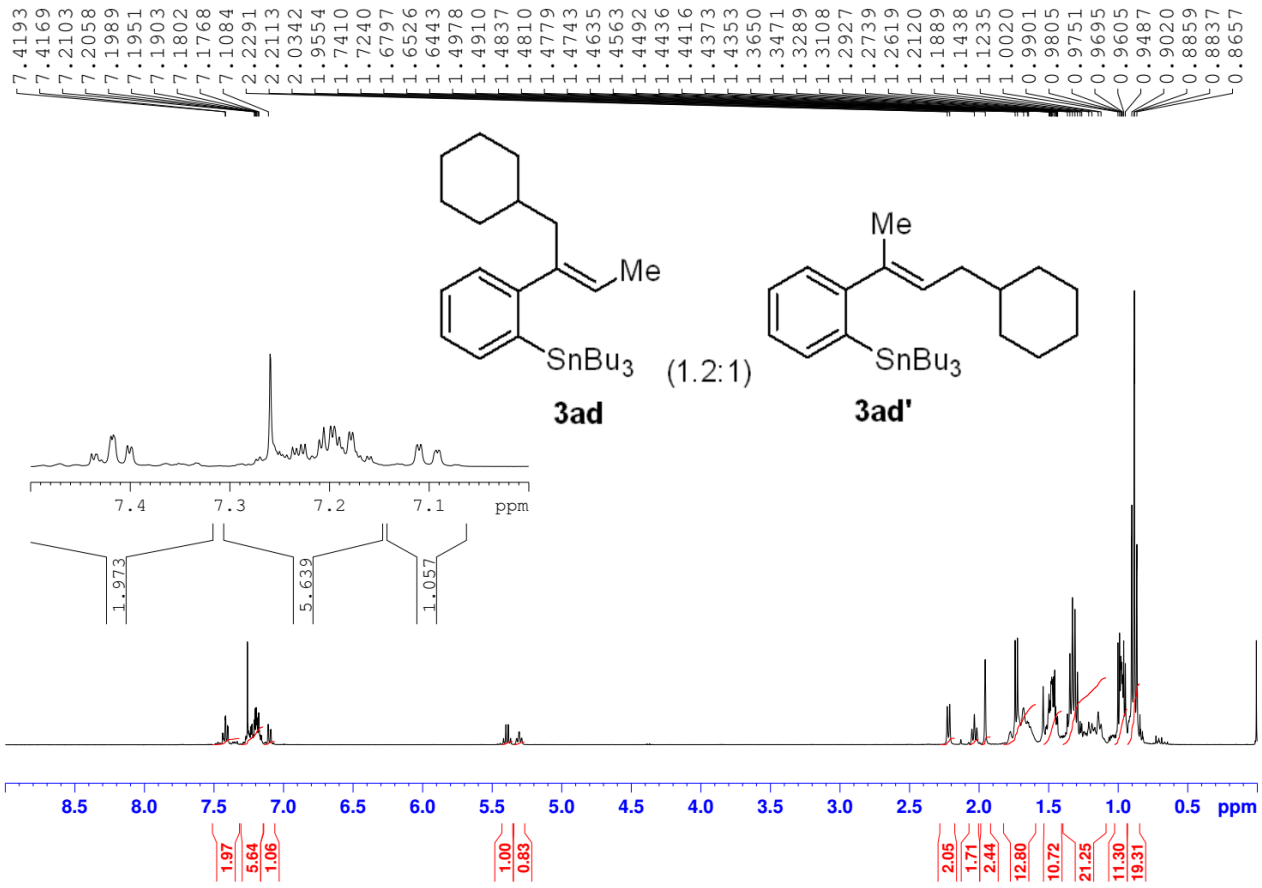
10.8863
9.2726
9.1979
13 12 11 10 ppm
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm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2.2113
2.0342
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1.7410
1.7240
1.6797
1.6526
1.6443
1.4978
1.4910
1.4837
1.4810
1.4779
1.4743
1.4635
1.4563
1.4492
1.4436
1.4416
1.4373
1.4353
1.3650
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1.3289
1.3108
1.2927
1.2739
1.2619
1.2120
1.1889
1.1438
1.1235
1.0020
0.9901
0.9805
0.9751
0.9695
0.9605
0.9487
0.9020
0.8859
0.8837
0.8657
Me
SnBu3
3ad
(1.2:1)
Me
SnBu3
3ad'
7.4
7.3
7.2
7.1
ppm
1.973
5.639
1.057
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
1.97
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2.44
12.80
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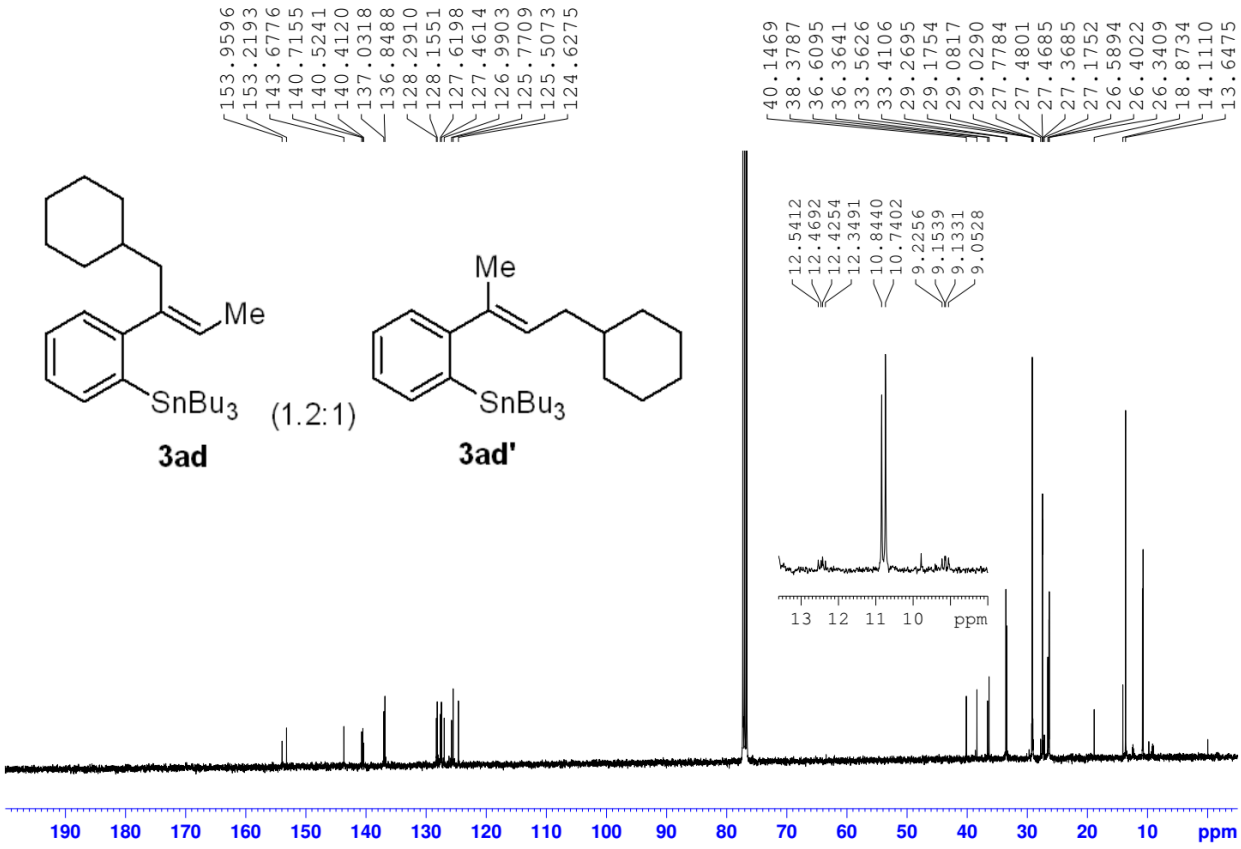
19.31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126.9903
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125.5073
124.6275
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38.3787
36.6095
36.3641
33.5626
33.4106
29.2695
29.1754
29.0817
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27.7784
27.4801
27.4685
27.3685
27.1752
26.5894
26.4022
26.3409
18.8734
14.1110
13.6475
Me
SnBu3
3ad
(1.2:1)
Me
SnBu3
3ad'
12.5412
12.4692
12.4254
12.3491
10.8440
10.7402
9.2256
9.1539
9.1331
9.0528
13
12
11
10
ppm
190
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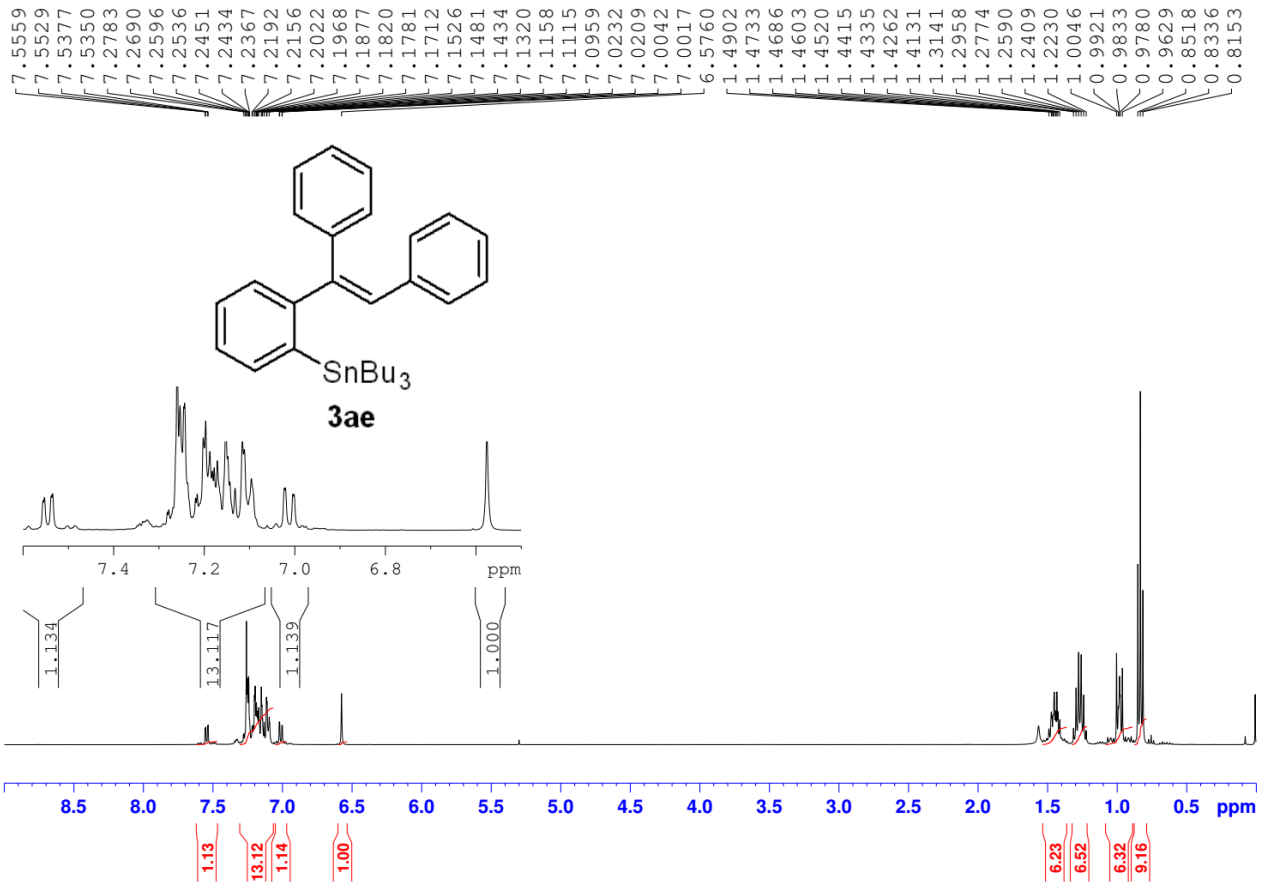
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm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7.2783
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7.2596
7.2536
7.2451
7.2434
7.2367
7.2192
7.2156
7.2022
7.1968
7.1877
7.1820
7.1781
7.1712
7.1526
7.1481
7.1434
7.1320
7.1158
7.1115
7.0959
7.0232
7.0209
7.0042
7.0017
6.5760
1.4902
1.4733
1.4686
1.4603
1.4520
1.4415
1.4335
1.4262
1.4131
1.3141
1.2958
1.2774
1.2590
1.2409
1.2230
1.0046
0.9921
0.9833
0.9780
0.9629
0.8518
0.8336
0.8153
SnBu3
3ae
7.4
7.2
7.0
6.8
ppm
1.134
13.117
1.139
1.000
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
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1.0
0.5
ppm
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1.00
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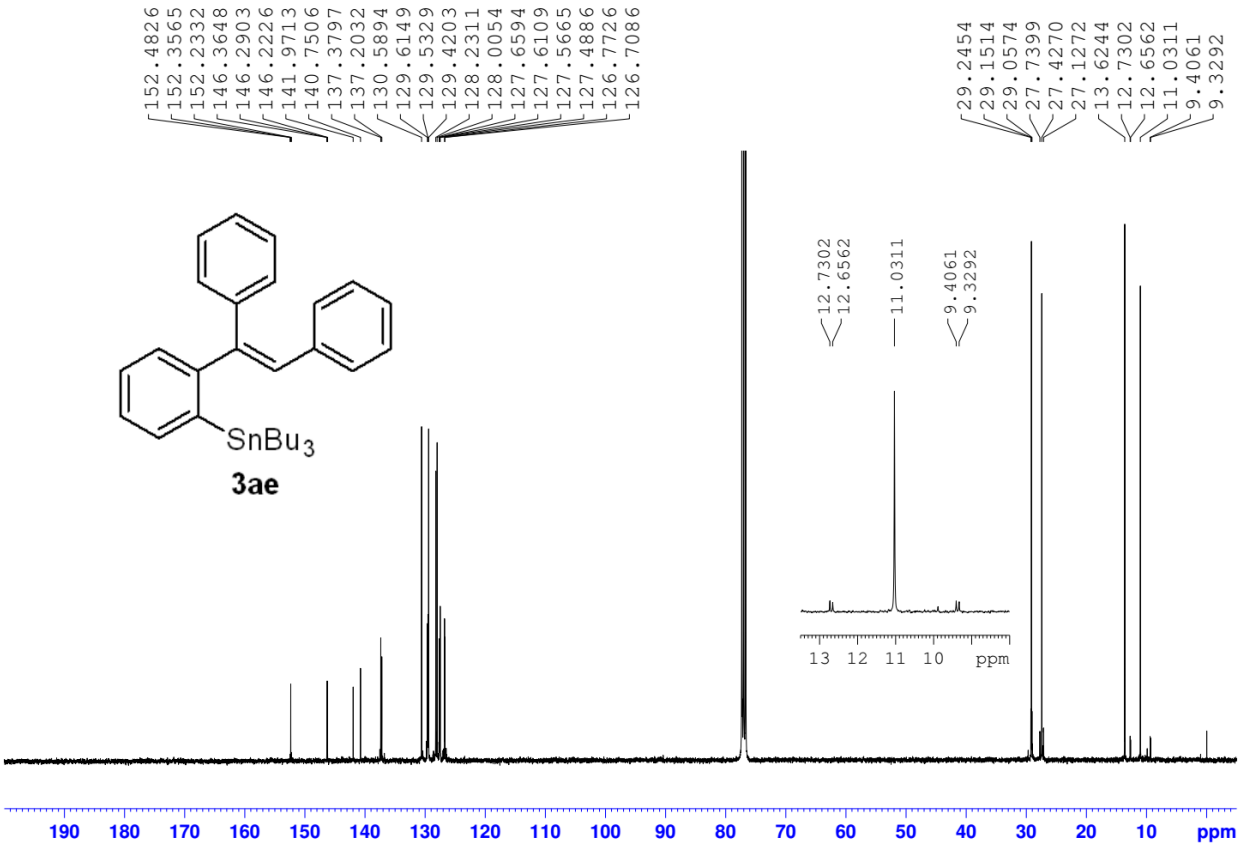
6.32
9.16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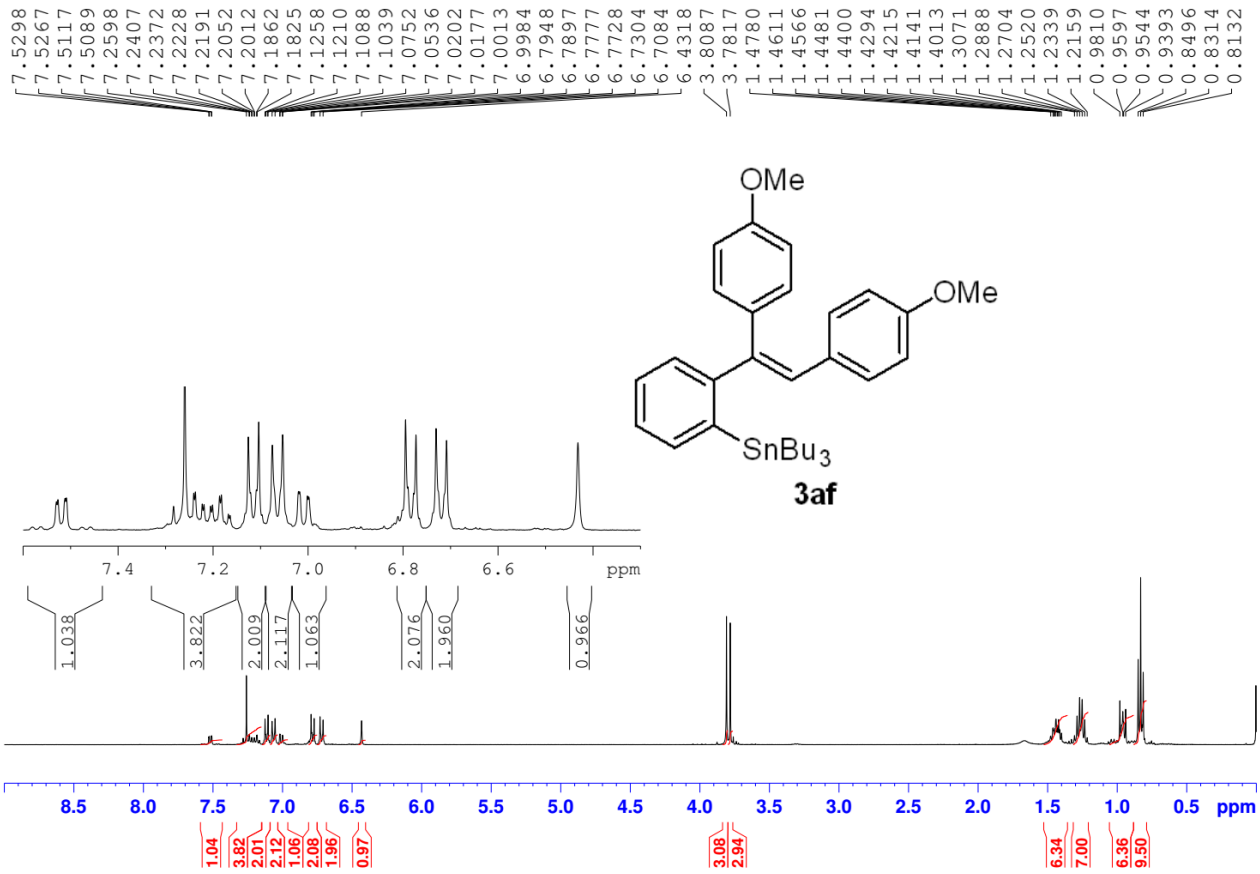
146.2903
146.2226
141.9713
140.7506
137.3797
137.2032
130.5894
129.6149
129.5329
129.4203
128.2311
128.0054
127.6594
127.6109
127.5665
127.4886
126.7726
126.7086
29.2454
29.1514
29.0574
27.7399
27.4270
27.1272
13.6244
12.7302
12.6562
11.0311
9.4061
9.3292
SnBu3
3ae
12.7302
12.6562
11.0311
9.4061
9.3292
13
12
11
10
ppm
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm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7.5267
7.5117
7.5089
7.2598
7.2407
7.2372
7.2228
7.2191
7.2052
7.2012
7.1862
7.1825
7.1258
7.1210
7.1088
7.1039
7.0752
7.0536
7.0202
7.0177
7.0013
6.9984
6.7948
6.7897
6.7777
6.7728
6.7304
6.7084
6.4318
3.8087
3.7817
1.4780
1.4611
1.4566
1.4481
1.4400
1.4294
1.4215
1.4141
1.4013
1.3071
1.2888
1.2704
1.2520
1.2339
1.2159
0.9810
0.9597
0.9544
0.9393
0.8496
0.8314
0.8132
OMe
OMe
SnBu3
3af
7.4
7.2
7.0
6.8
6.6
ppm
1.038
3.822
2.009
2.117
1.063
2.076
1.960
0.966
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
1.04
3.82
2.01
2.12
1.06
2.08
1.96
0.97
3.08
2.94
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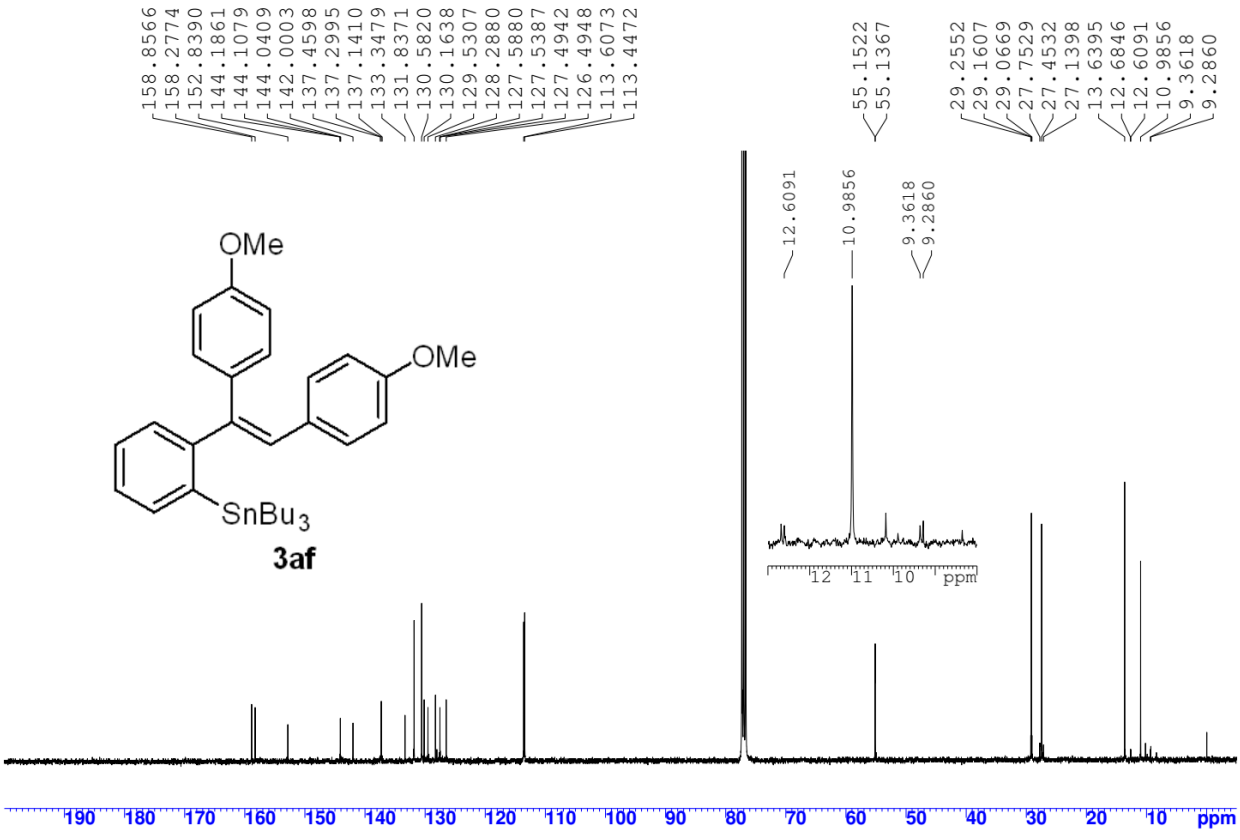
7.00
6.36
9.50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142.0003
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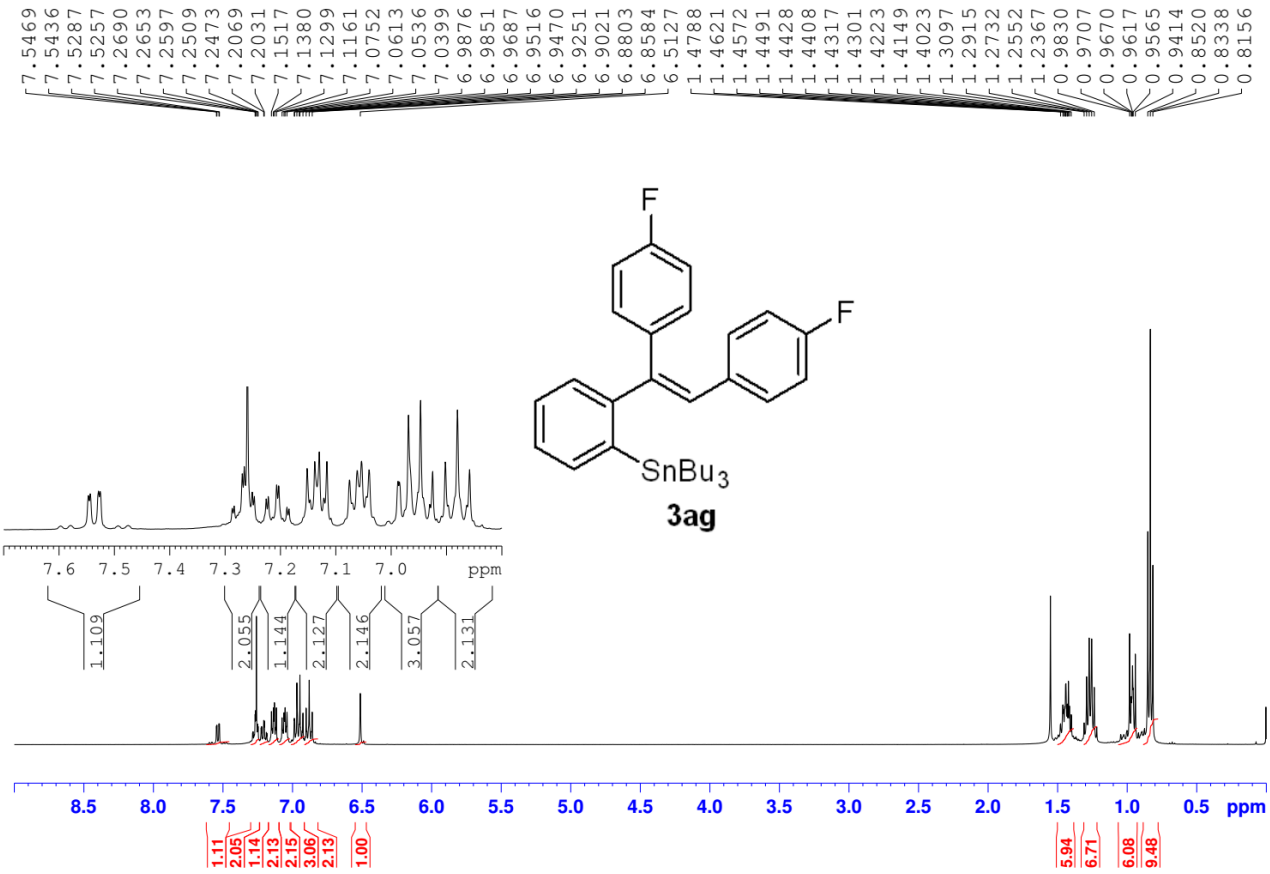
137.2995
137.1410
133.3479
131.8371
130.5820
130.1638
129.5307
128.2880
127.5880
127.5387
127.4942
126.4948
113.6073
113.4472
55.1522
55.1367
29.2552
29.1607
29.0669
27.7529
27.4532
27.1398
13.6395
12.6846
12.6091
10.9856
9.3618
9.2860
OMe
OMe
SnBu3
3af
12.6091
10.9856
9.3618
9.2860
12
11
10
ppm
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm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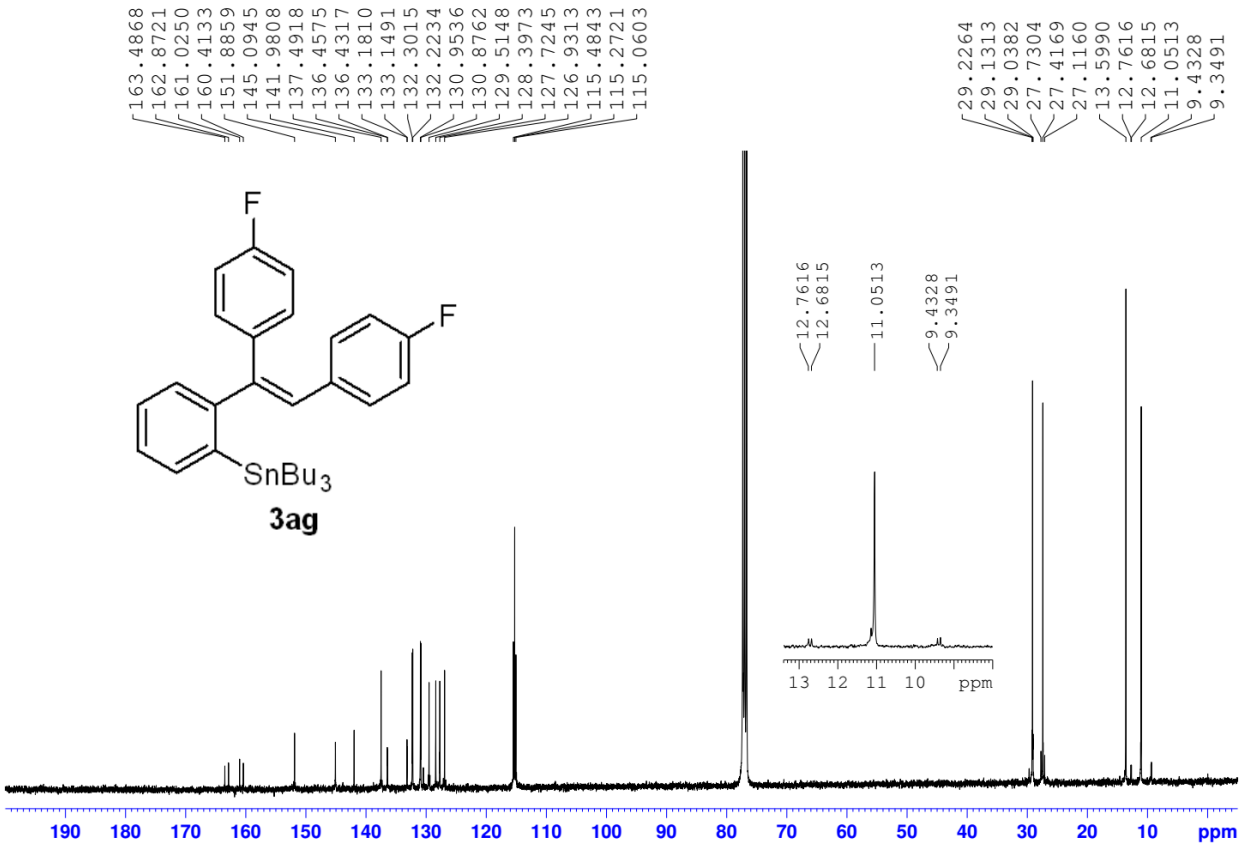
7.5257
7.2690
7.2653
7.2597
7.2509
7.2473
7.2069
7.2031
7.1517
7.1380
7.1299
7.1161
7.0752
7.0613
7.0536
7.0399
6.9876
6.9851
6.9687
6.9516
6.9470
6.9251
6.9021
6.8803
6.8584
6.5127
1.4788
1.4621
1.4572
1.4491
1.4428
1.4408
1.4317
1.4301
1.4223
1.4149
1.4023
1.3097
1.2915
1.2732
1.2552
1.2367
0.9830
0.9707
0.9670
0.9617
0.9565
0.9414
0.8520
0.8338
0.8156
F
F
SnBu3
3ag
7.6 7.5 7.4 7.3 7.2 7.1 7.0 ppm
1.109
2.055
1.144
2.127
2.146
3.057
2.131
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.11
2.05
1.14
2.13
2.15
3.06
2.13
1.00
5.94
6.71
6.08
9.48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162.8721
161.0250
160.4133
151.8859
145.0945
141.9808
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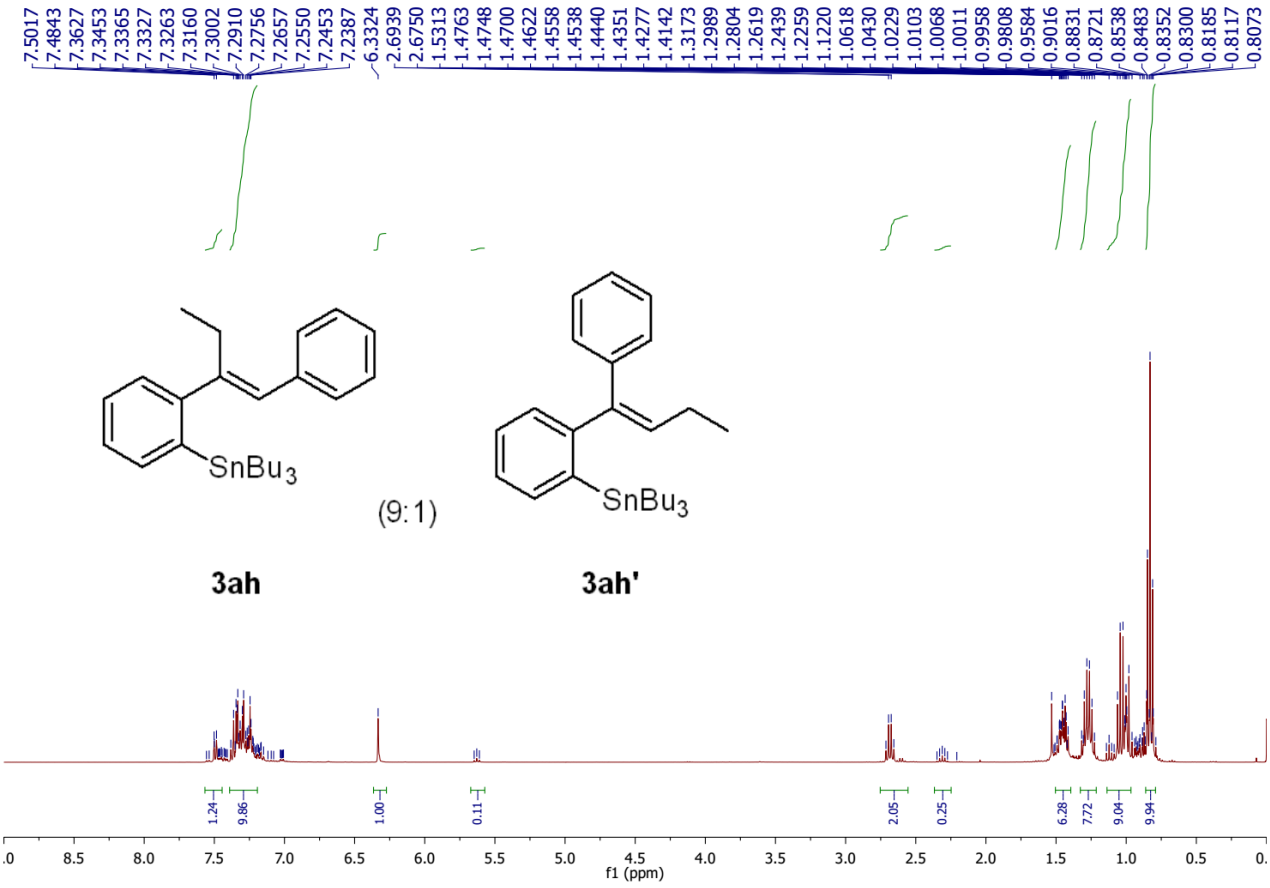
136.4575
136.4317
133.1810
133.1491
132.3015
132.2234
130.9536
130.8762
129.5148
128.3973
127.7245
126.9313
115.4843
115.2721
115.0603
29.2264
29.1313
29.0382
27.7304
27.4169
27.1160
13.5990
12.7616
12.6815
11.0513
9.4328
9.3491
F
F
SnBu3
3ag
12.7616
12.6815
11.0513
9.4328
9.3491
13 12 11 10 ppm
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


7.5017
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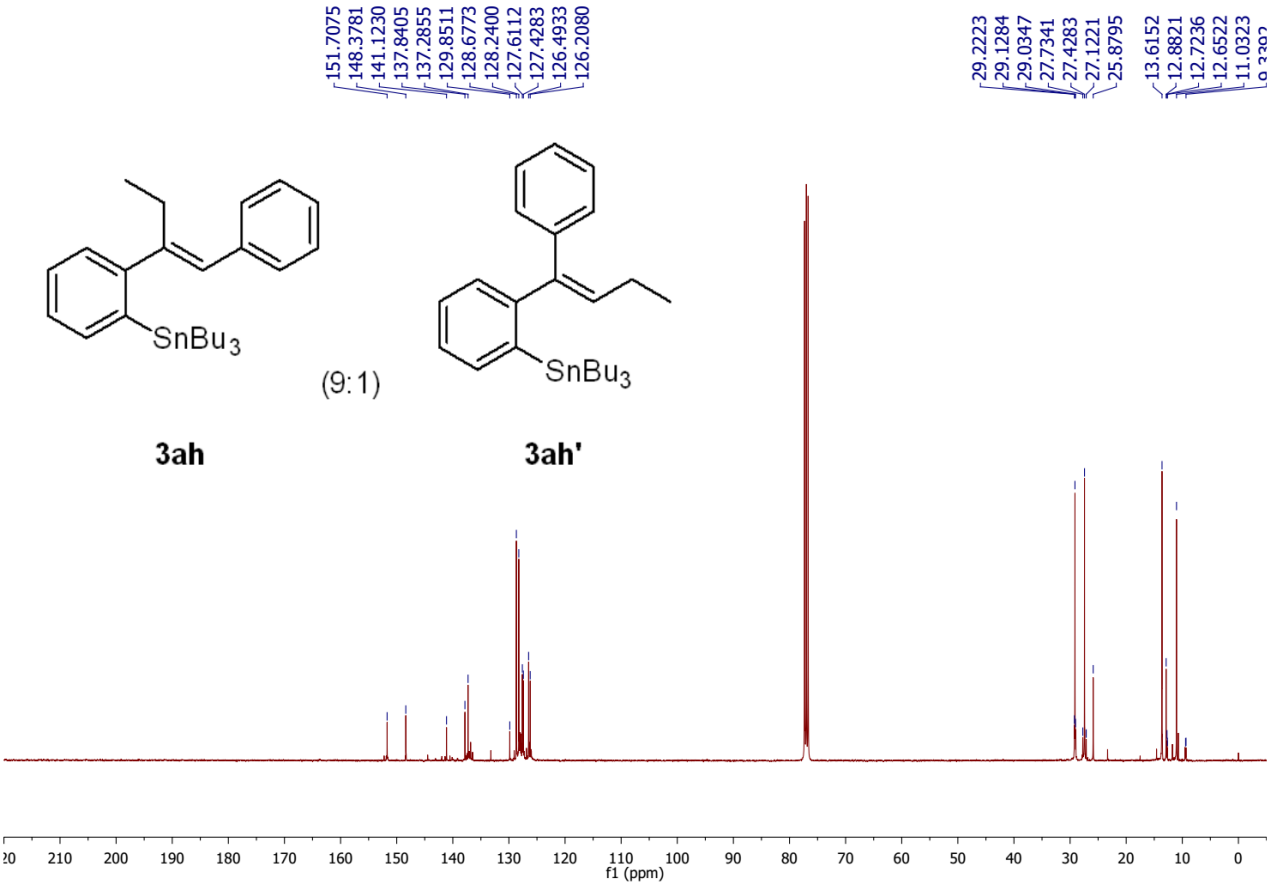
7.3627
7.3453
7.3365
7.3327
7.3263
7.3160
7.3002
7.2910
7.2756
7.2657
7.2550
7.2453
7.2387
6.3324
2.6939
2.6750
2.5313
1.4763
1.4748
1.4700
1.4622
1.4558
1.4538
1.4440
1.4351
1.4277
1.4142
1.3173
1.2989
1.2804
1.2619
1.2439
1.2259
1.1220
1.0618
1.0430
1.0229
1.0103
1.0068
1.0011
0.9958
0.9808
0.9584
0.9016
0.8831
0.8721
0.8538
0.8483
0.8352
0.8300
0.8185
0.8117
0.8073
SnBu3
(9:1)
SnBu3
3ah
3ah'
1.24
9.86
1.00
0.11
2.05
0.25
6.28
7.72
9.04
9.94
f1 (ppm)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137.2855
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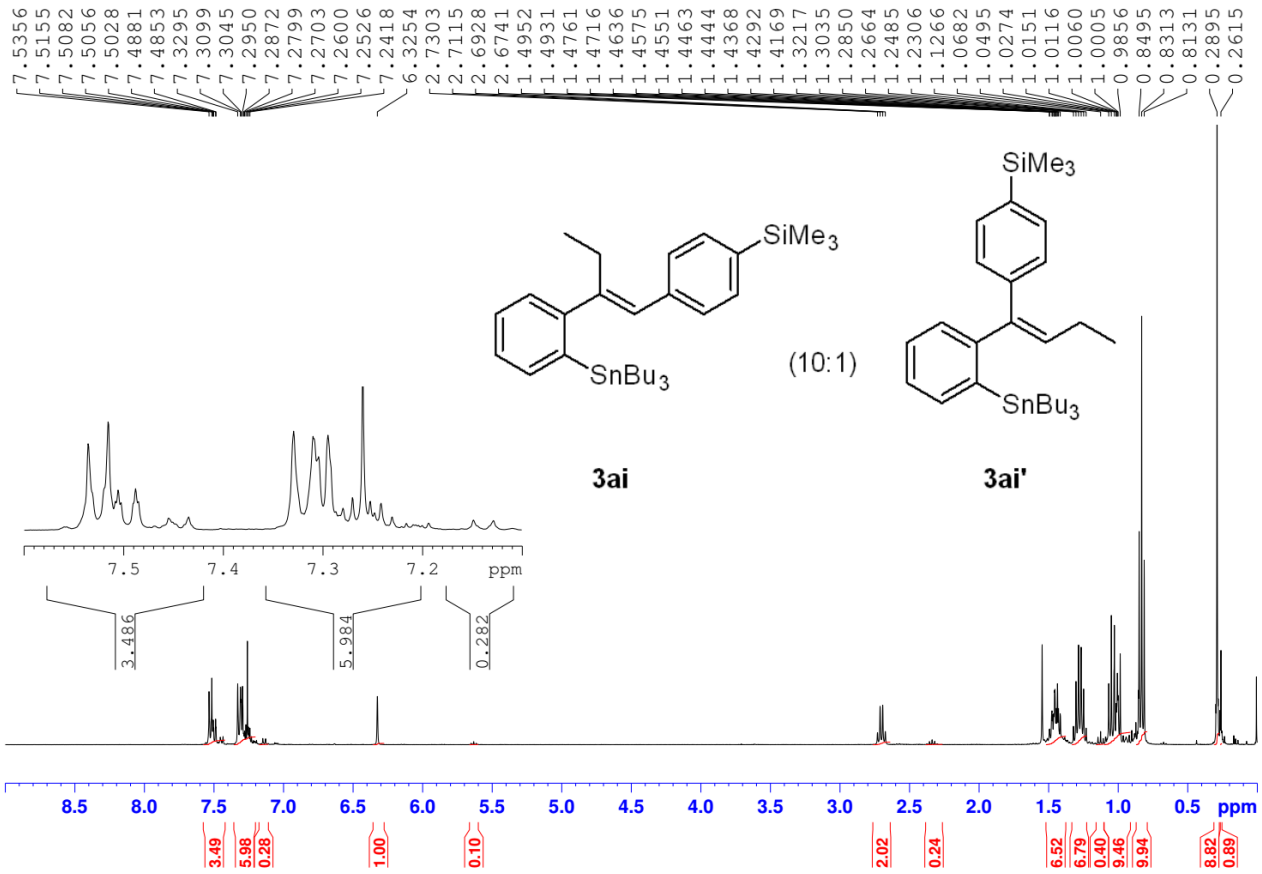
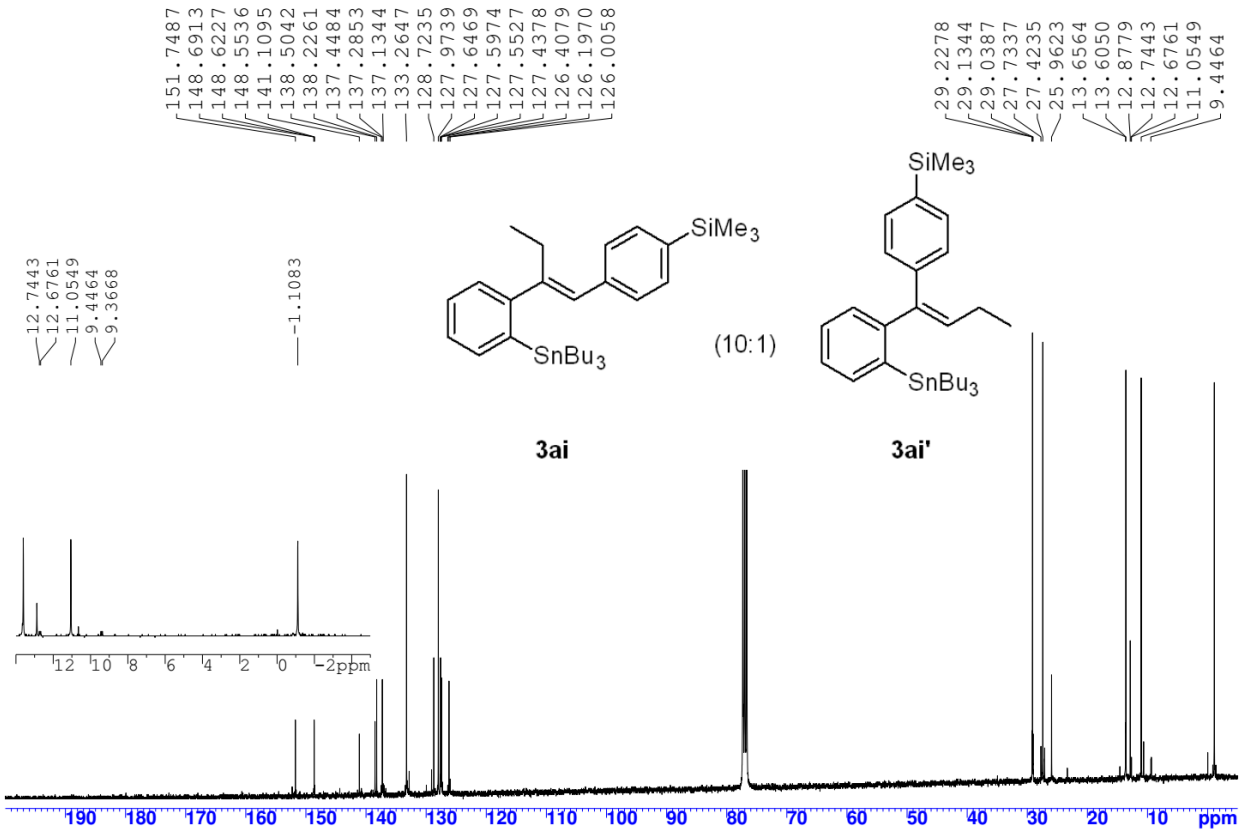
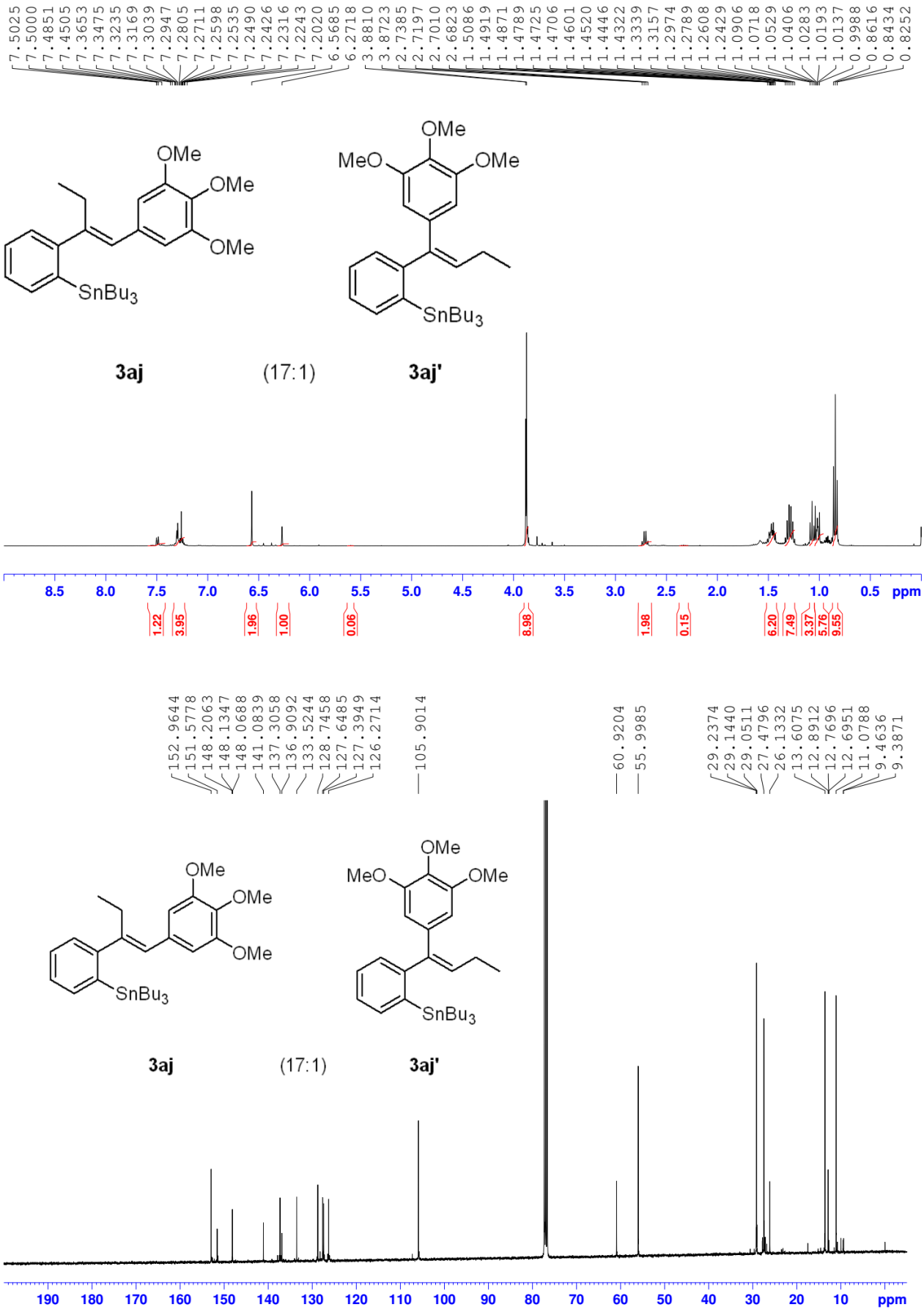
128.6773
128.2400
127.6112
127.4283
126.4933
126.2080
29.2223
29.1284
29.0347
27.7341
27.4283
27.1221
25.8795
13.6152
12.8821
12.7236
12.6522
11.0323
SnBu3
(9:1)
SnBu3
3ah
3ah'
f1 (ppm)


SiMe3
SnBu3
(10:1)
3ai
3ai'
ppm


SiMe3
SnBu3
(10:1)
3ai
3ai'
ppm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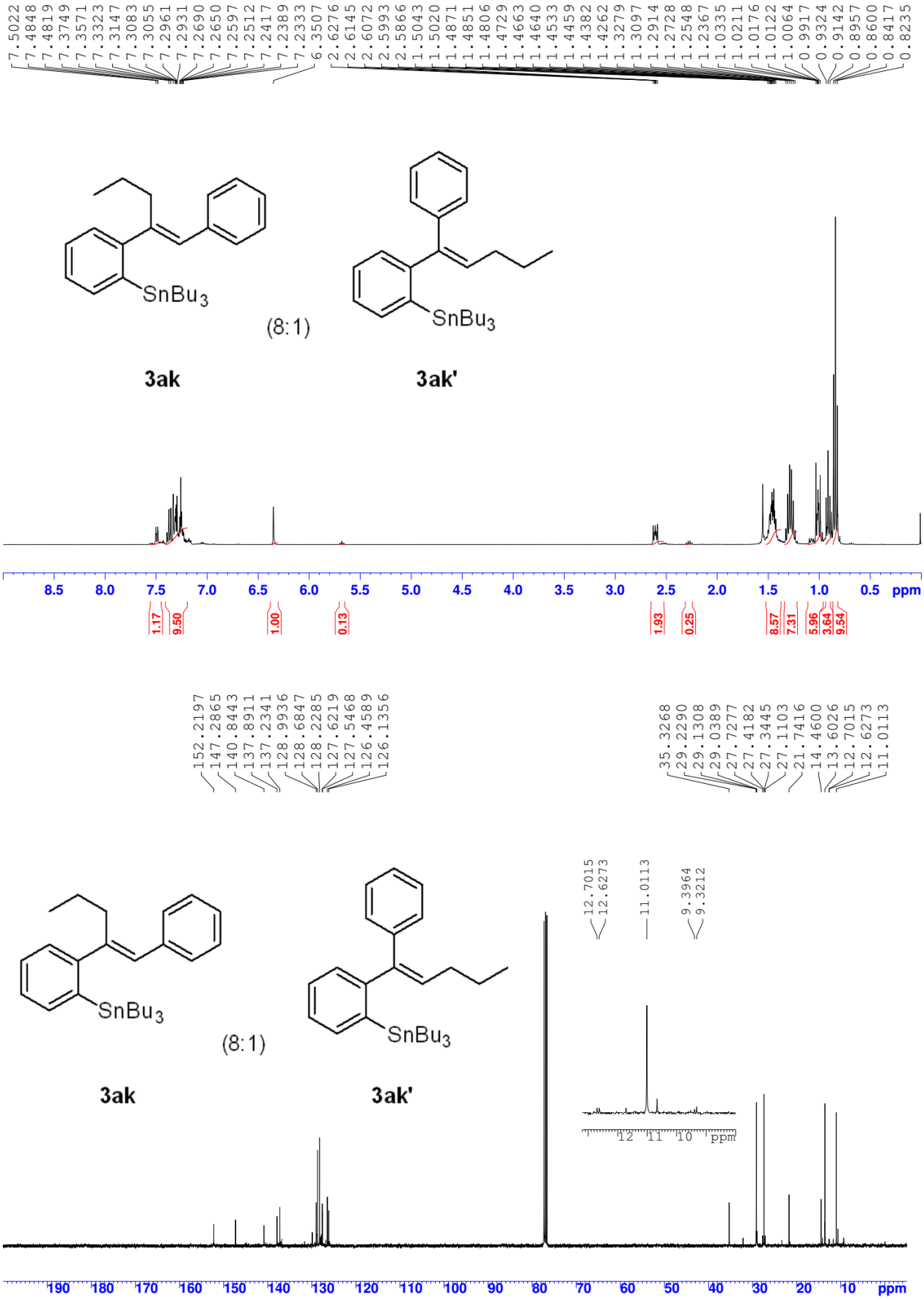
7.3039
7.2947
7.2805
7.2711
7.2598
7.2535
7.2490
7.2426
7.2316
7.2243
7.2020
6.5685
6.2718
3.8810
3.8723
2.7385
2.7197
2.7010
2.6823
1.5086
1.4919
1.4871
1.4789
1.4725
1.4706
1.4601
1.4520
1.4446
1.4322
1.3339
1.3157
1.2974
1.2789
1.2608
1.2429
1.0906
1.0718
1.0529
1.0406
1.0283
1.0193
1.0137
0.9988
0.8616
0.8434
0.8252
OMe
OMe
OMe
SnBu3
MeO
OMe
OMe
SnBu3
3aj
(17:1)
3aj'
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
1.22
3.95
1.96
1.00
0.06
8.98
1.98
0.15
6.20
7.49
3.37
5.76
9.55
152.9644
151.5778
148.2063
148.1347
148.0688
141.0839
137.3058
136.9092
133.5244
128.7458
127.6485
127.3949
126.2714
105.9014
60.9204
55.9985
29.2374
29.1440
29.0511
27.4796
26.1332
13.6075
12.8912
12.7696
12.6951
11.0788
9.4636
9.3871
OMe
OMe
OMe
SnBu3
MeO
OMe
OMe
SnBu3
3aj
(17:1)
3aj'
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm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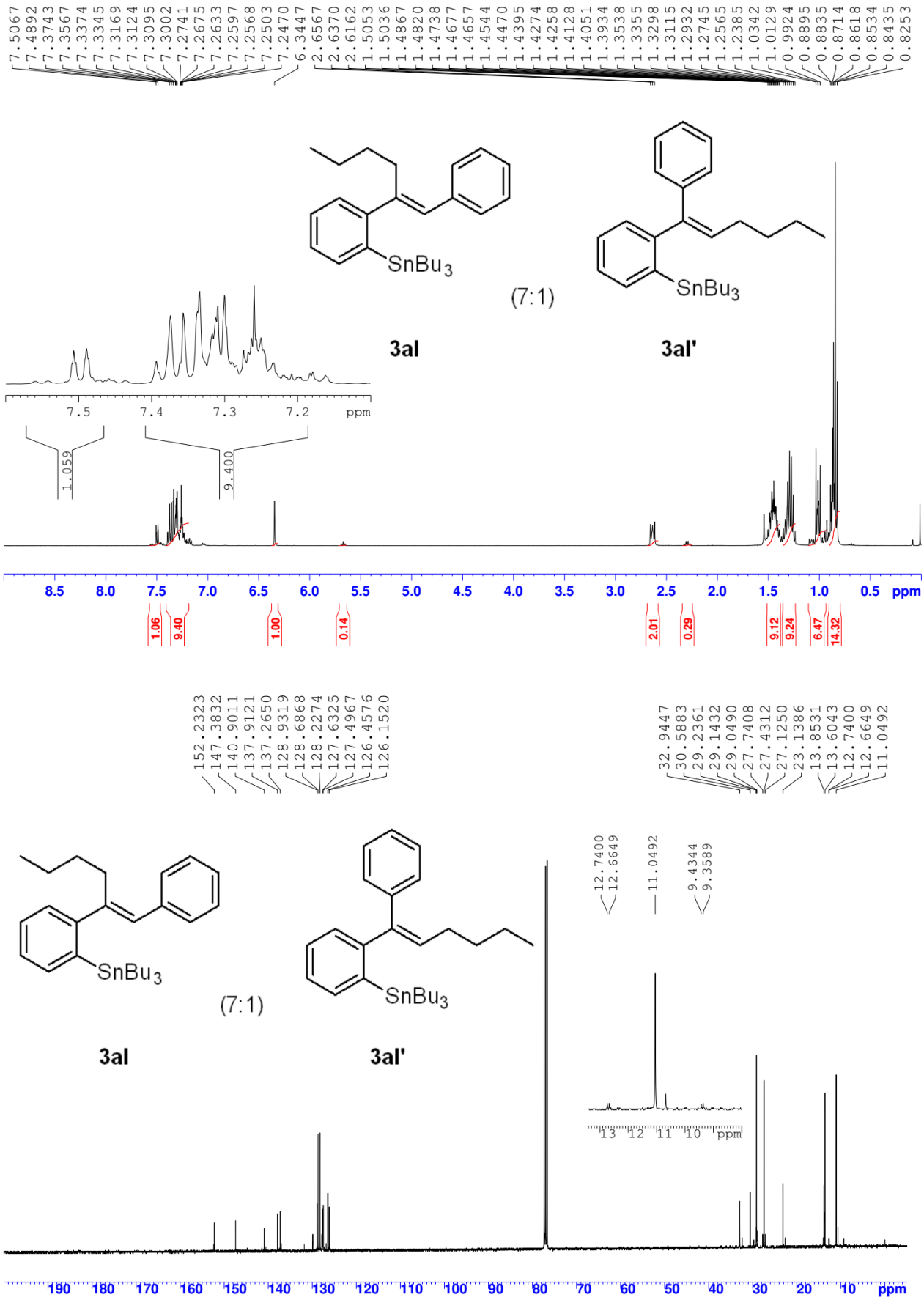
7.3055
7.2961
7.2931
7.2690
7.2650
7.2597
7.2512
7.2417
7.2389
7.2333
6.3507
2.6276
2.6145
2.6072
2.5993
2.5866
1.5043
1.5020
1.4871
1.4851
1.4806
1.4729
1.4663
1.4640
1.4533
1.4459
1.4382
1.4262
1.3279
1.3097
1.2914
1.2728
1.2548
1.2367
1.0335
1.0211
1.0176
1.0122
1.0064
0.9917
0.9324
0.9142
0.8957
0.8600
0.8417
0.8235
SnBu3
(8:1)
SnBu3
3ak
3ak'
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.17
9.50
1.00
0.13
1.93
0.25
8.57
7.31
5.96
3.64
9.54
152.2197
147.2865
140.8443
137.8911
137.2341
128.9936
128.6847
128.2285
127.6219
127.5468
126.4589
126.1356
35.3268
29.2290
29.1308
29.0389
27.7277
27.4182
27.3445
27.1103
21.7416
14.4600
13.6026
12.7015
12.6273
11.0113
12.7015
12.6273
11.0113
9.3964
9.3212
SnBu3
(8:1)
SnBu3
3ak
3ak'
12 11 10 ppm
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm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7.3567
7.3374
7.3345
7.3169
7.3124
7.3095
7.3002
7.2741
7.2675
7.2633
7.2597
7.2568
7.2503
7.2470
6.3447
2.6567
2.6370
2.6162
1.5053
1.5036
1.4867
1.4820
1.4738
1.4677
1.4657
1.4544
1.4470
1.4395
1.4274
1.4258
1.4128
1.4051
1.3934
1.3538
1.3355
1.3298
1.3115
1.2932
1.2745
1.2565
1.2385
1.0342
1.0129
0.9924
0.8895
0.8835
0.8714
0.8618
0.8534
0.8435
0.8253
SnBu3
SnBu3
(7:1)
3al
3al'
7.5
7.4
7.3
7.2
ppm
1.059
9.400
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
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0.14
2.01
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9.24
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137.2650
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126.1520
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29.2361
29.1432
29.0490
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27.4312
27.1250
23.1386
13.8531
13.6043
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12.6649
11.0492
12.7400
12.6649
11.0492
9.4344
9.3589
SnBu3
SnBu3
(7:1)
3al
3al'
13
12
11
10
ppm
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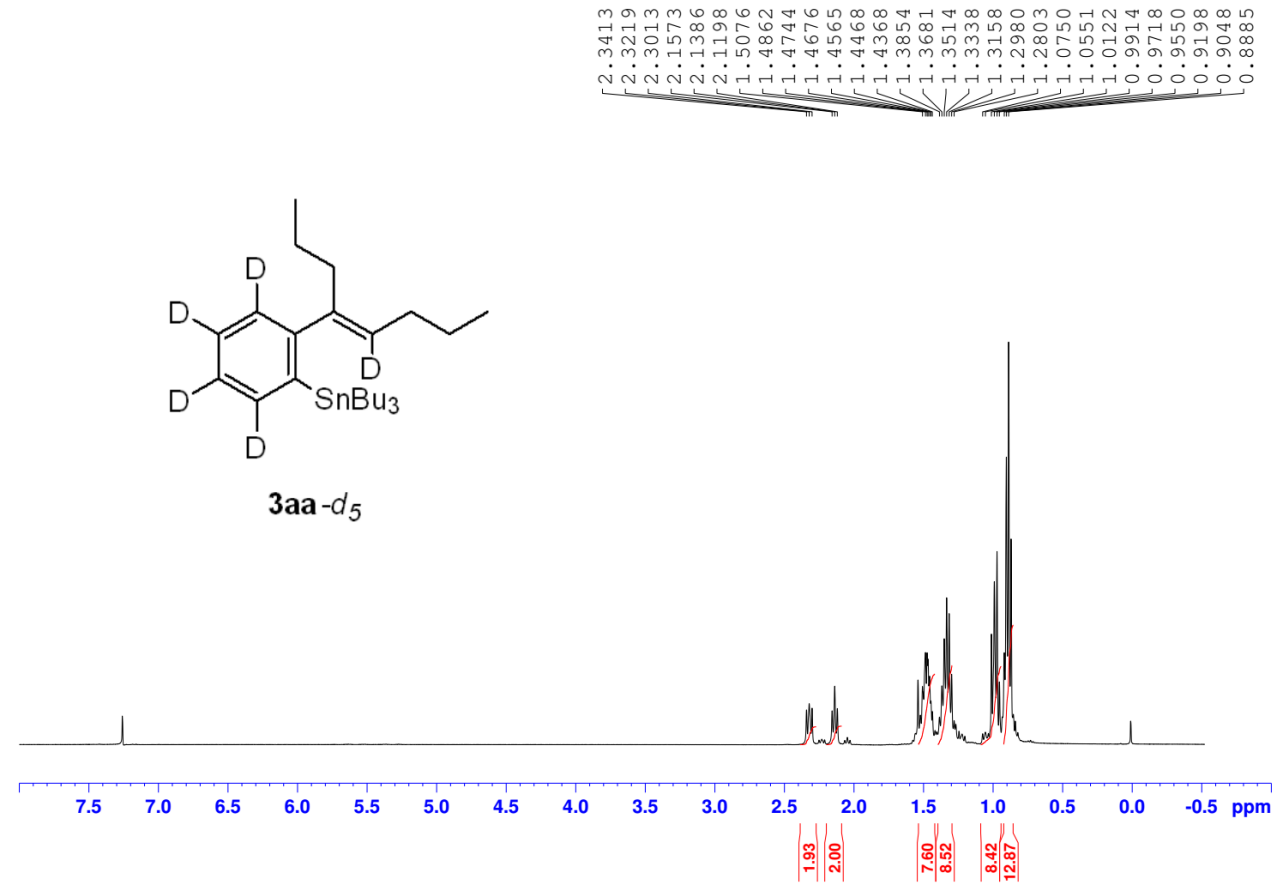
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1.0750
1.0551
1.0122
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0.9718
0.9550
0.9198
0.9048
0.8885
D
D
D
D
D
SnBu3
3aa-d5
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 ppm
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2.00
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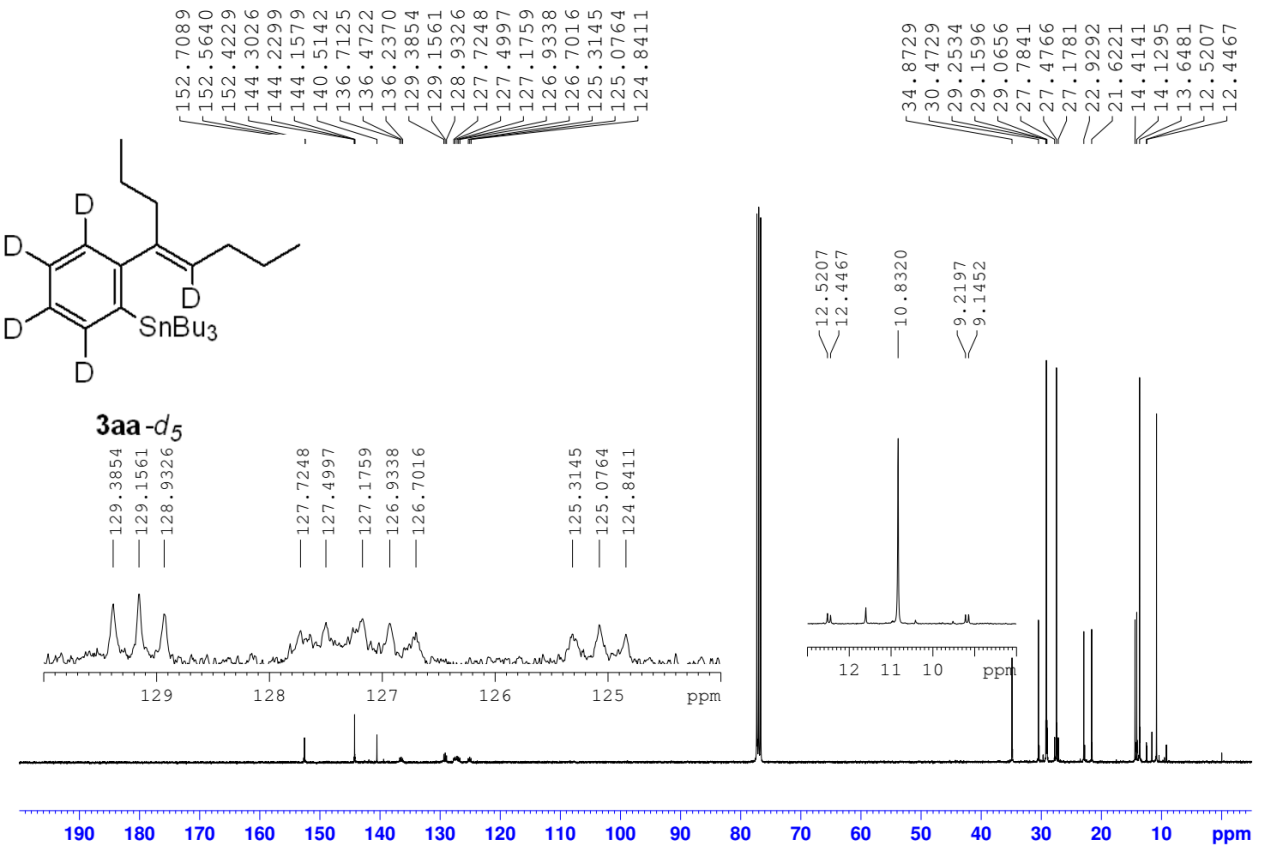
12.87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129.1561
128.9326
127.7248
127.4997
127.1759
126.9338
126.7016
125.3145
125.0764
124.8411
34.8729
30.4729
29.2534
29.1596
29.0656
27.7841
27.4766
27.1781
22.9292
21.6221
14.4141
14.1295
13.6481
12.5207
12.4467
D
D
D
D
D
SnBu3
3aa-d5
12.5207
12.4467
10.8320
9.2197
9.1452
129.3854
129.1561
128.9326
127.7248
127.4997
127.1759
126.9338
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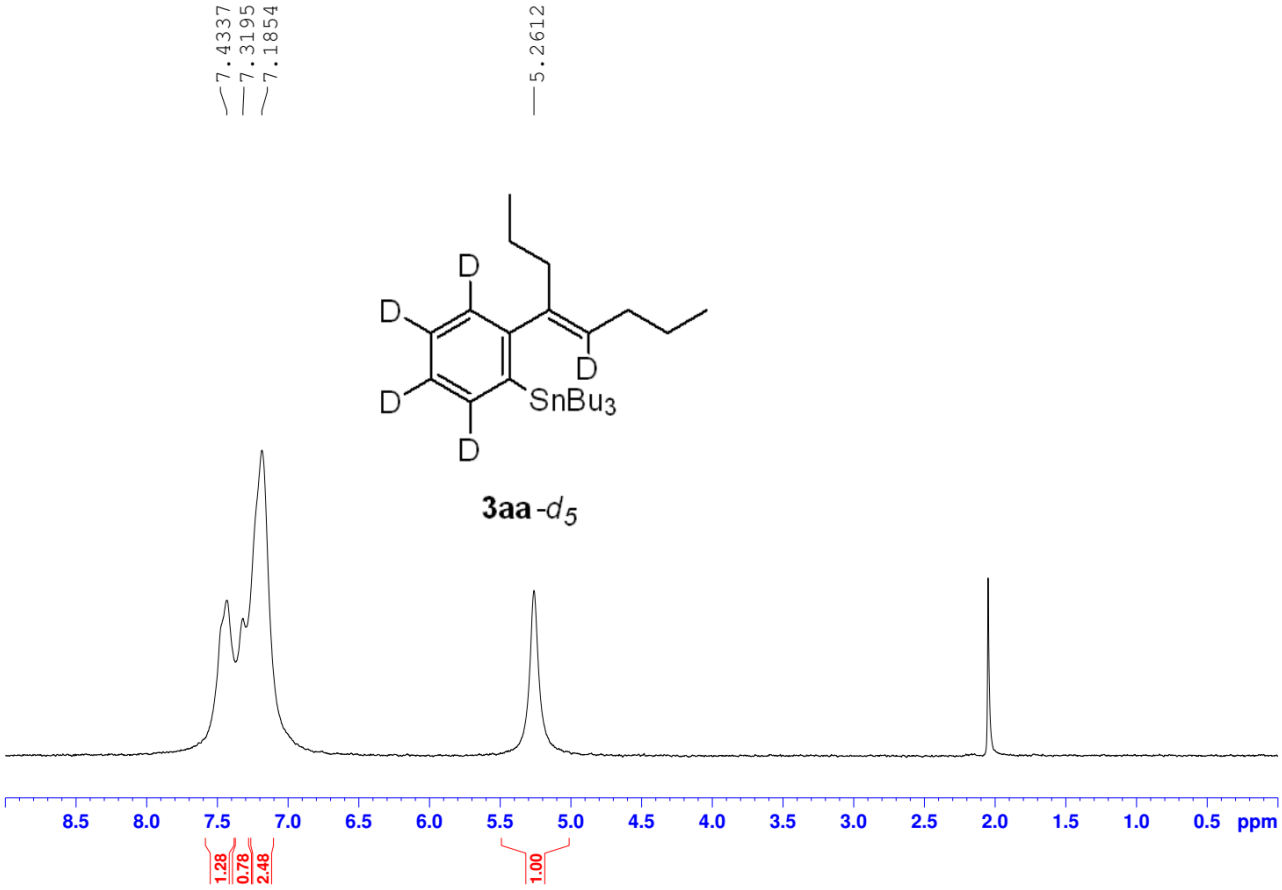
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D
D
D
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7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
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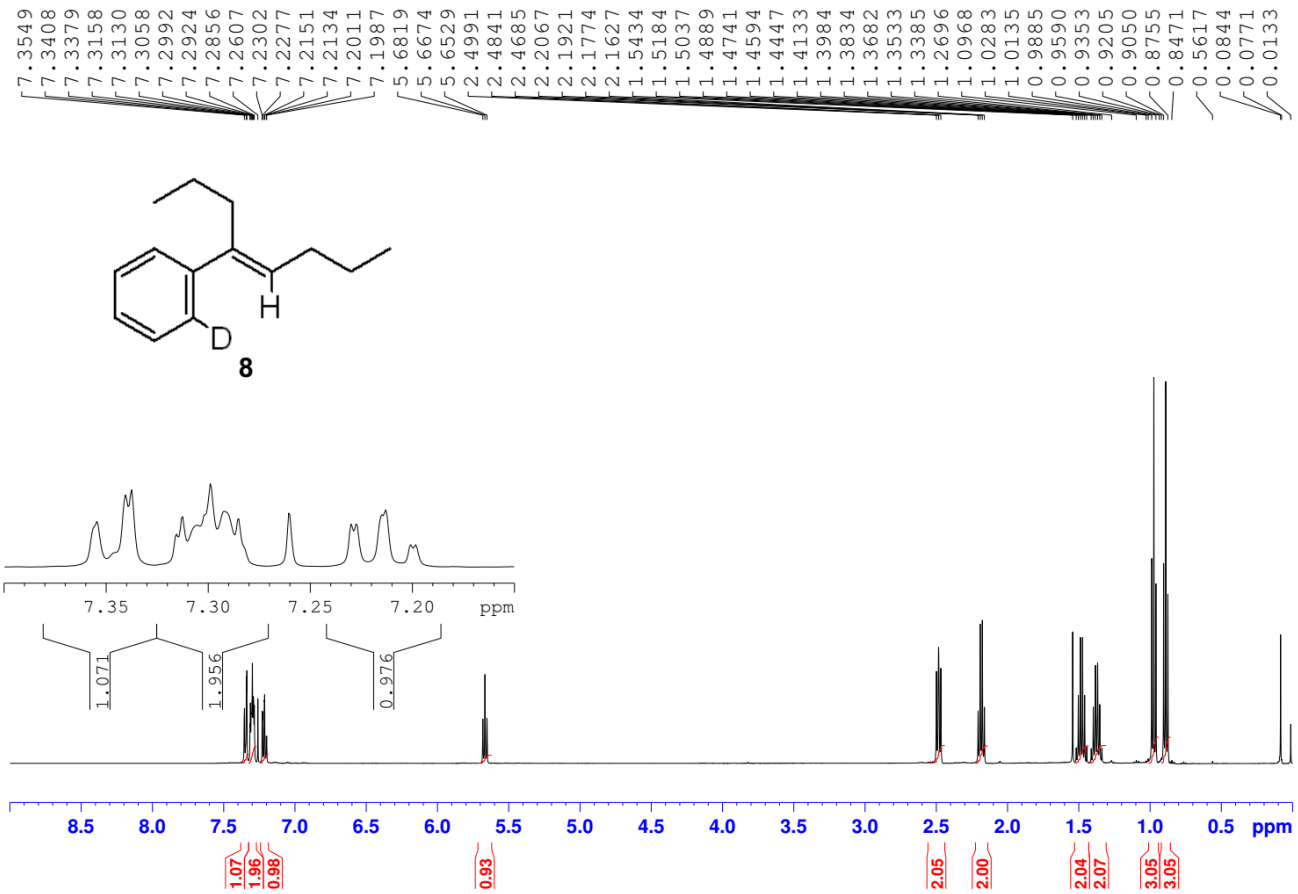
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7.2277
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7.2134
7.2011
7.1987
5.6819
5.6674
5.6529
2.4991
2.4841
2.4685
2.2067
2.1921
2.1774
2.1627
1.5434
1.5184
1.5037
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1.4741
1.4594
1.4447
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0.9590
0.9353
0.9205
0.9050
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0.0844
0.0771
0.0133
D
H
8
7.35
7.30
7.25
7.20
ppm
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8.0
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7.0
6.5
6.0
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5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
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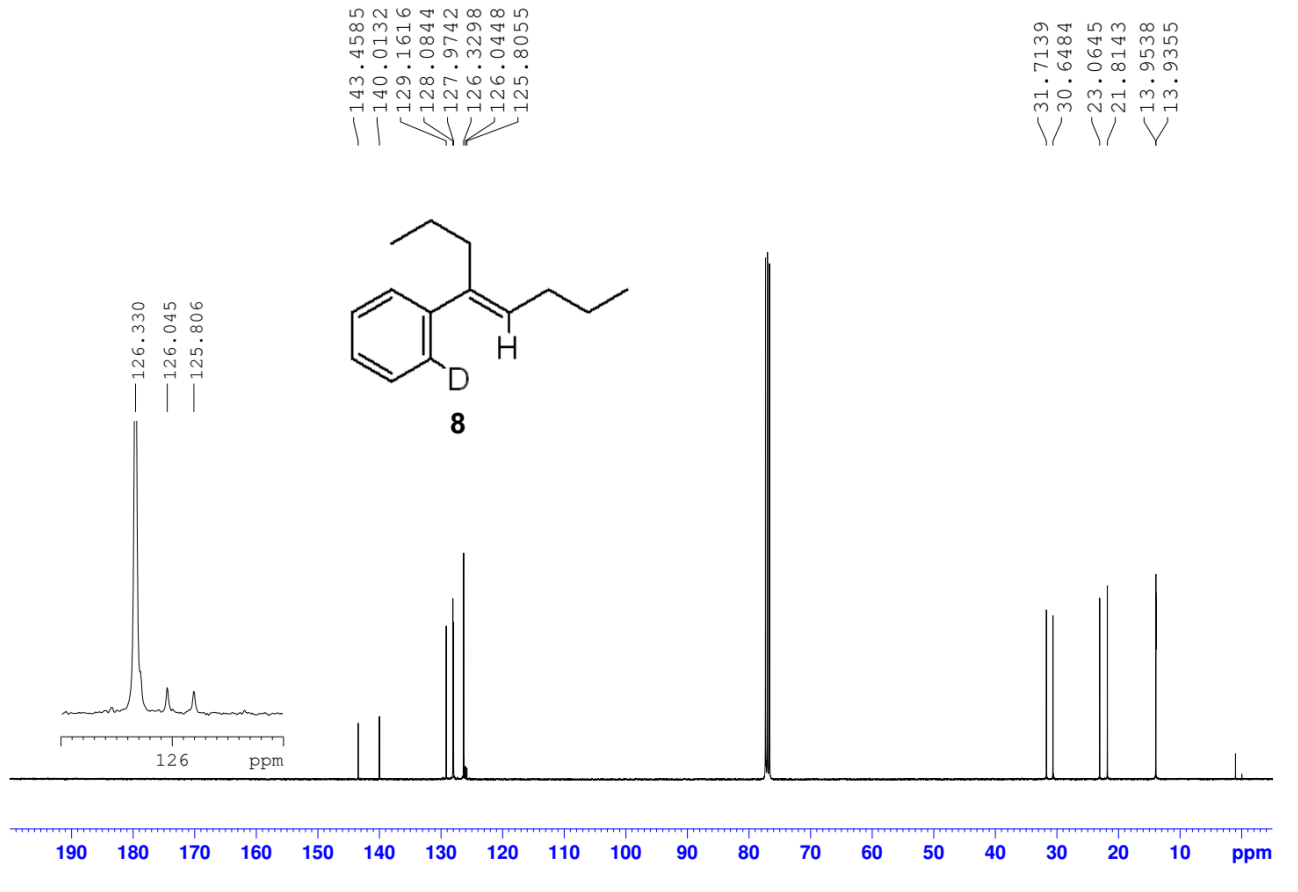
2.07
3.05
3.05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30.6484
23.0645
21.8143
13.9538
13.9355
126.330
126.045
125.806
D
H
8
126
ppm
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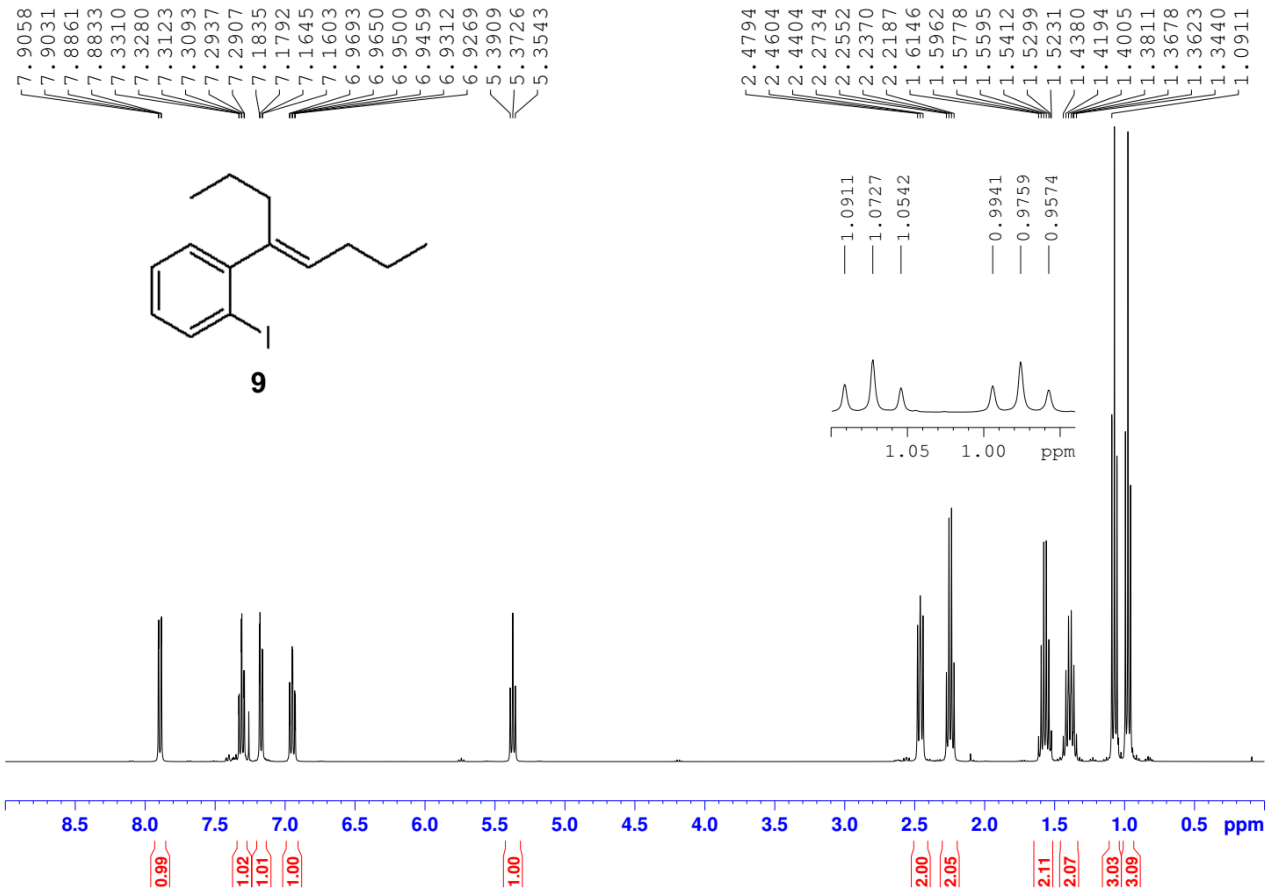
10
ppm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7.1792
7.1645
7.1603
6.9693
6.9650
6.9500
6.9459
6.9312
6.9269
5.3909
5.3726
5.3543
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2.4404
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2.2552
2.2370
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1.4005
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1.3678
1.3623
1.3440
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1.0911
1.0727
1.0542
0.9941
0.9759
0.9574
1.05
1.00
ppm
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7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
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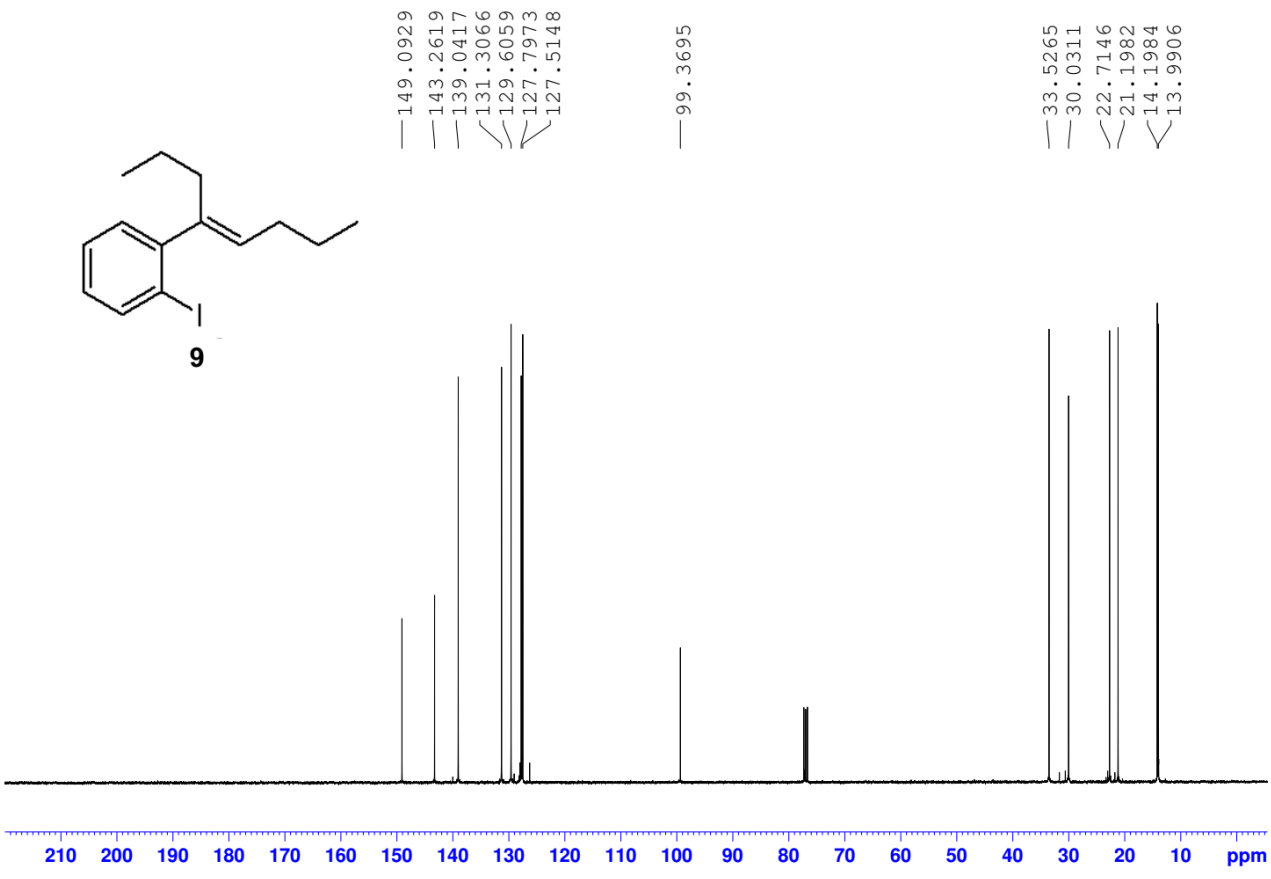
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21.1982
14.1984
13.9906
9
210
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150
140
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120
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30
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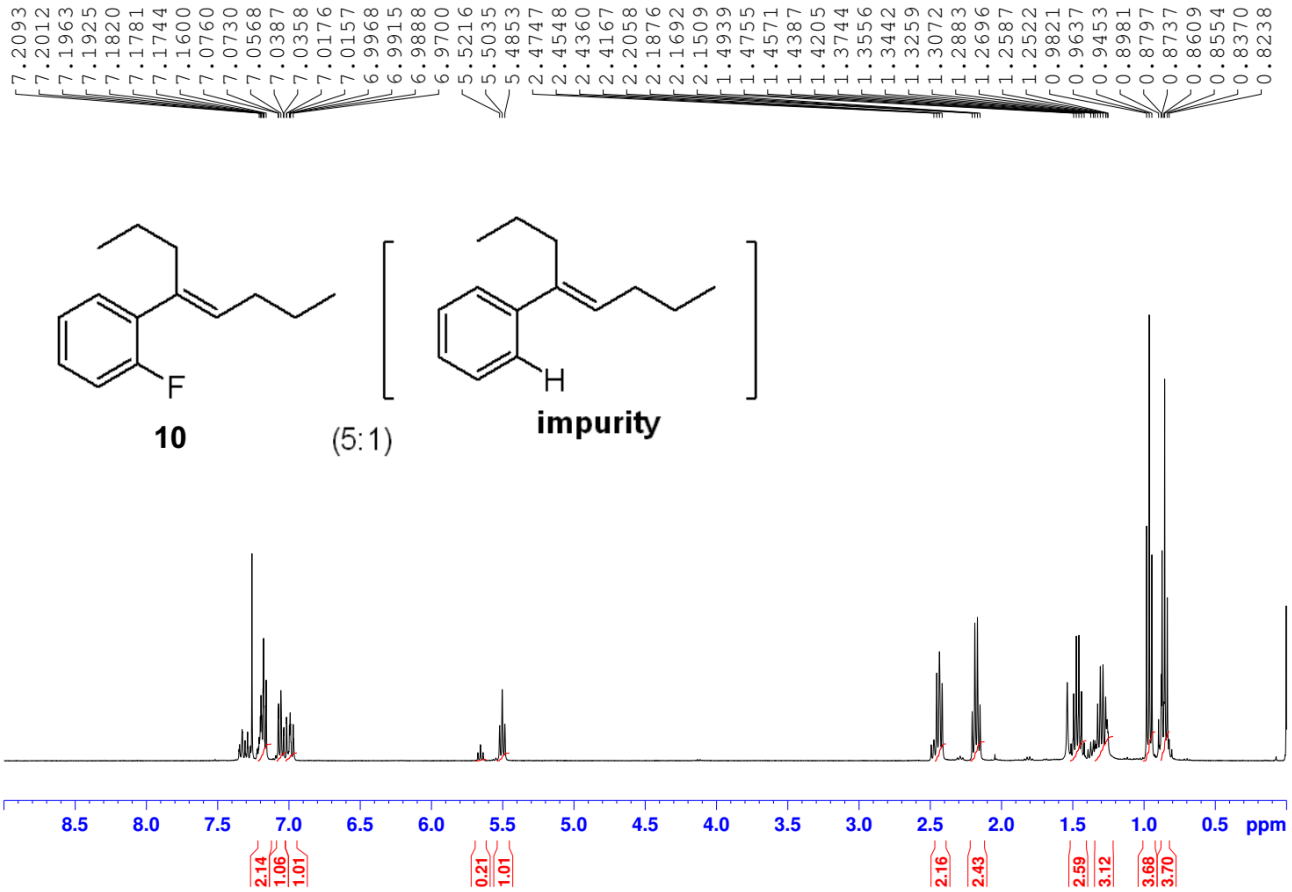
10
ppm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7.0157
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6.9915
6.9888
6.9700
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5.5035
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2.4548
2.4360
2.4167
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2.1876
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2.1509
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1.4205
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0.8981
0.8797
0.8737
0.8609
0.8554
0.8370
0.8238
F
10
(5:1)
H
impurity
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
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1.01
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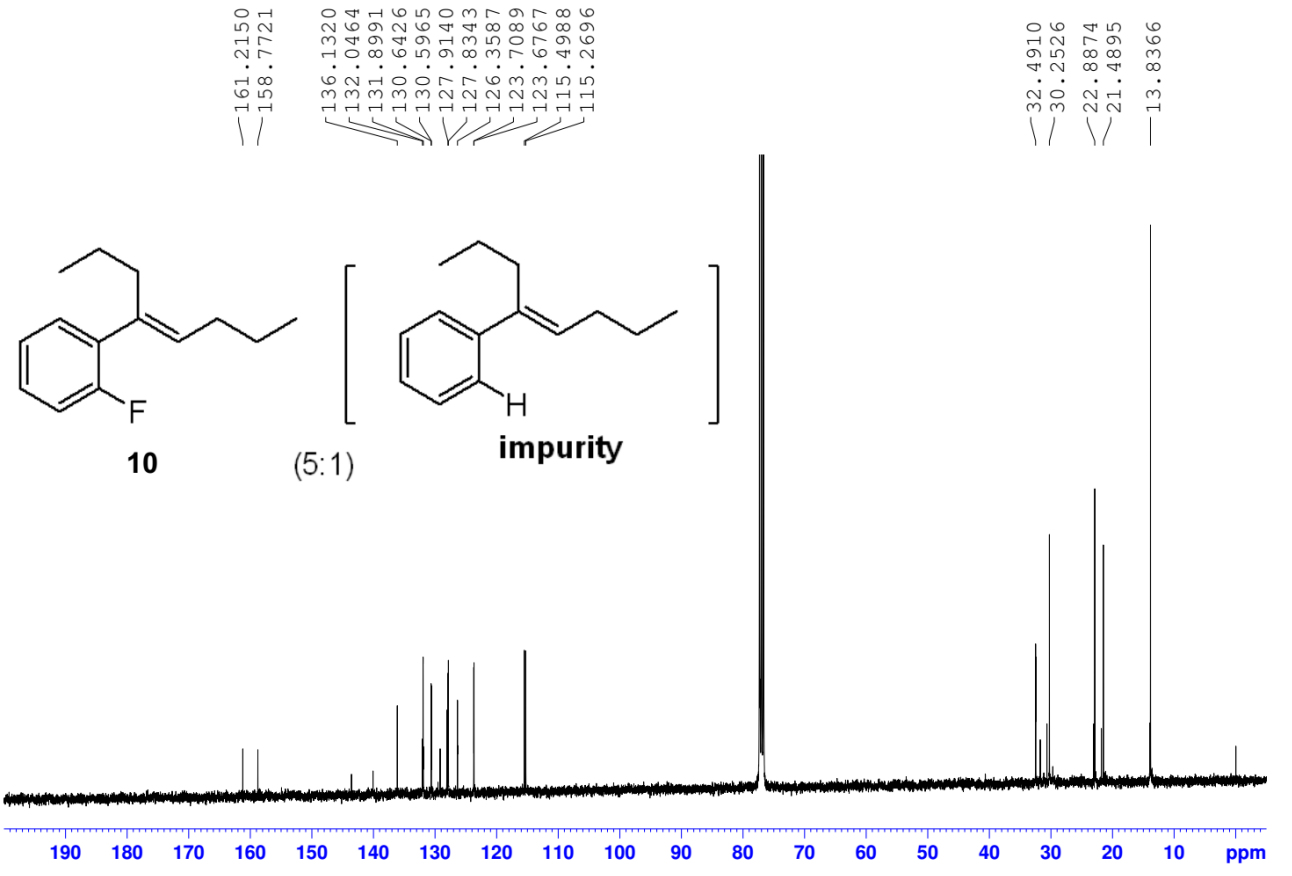
3.70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13.8366
F
10
(5:1)
H
impurity
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150
140
130
120
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100
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80
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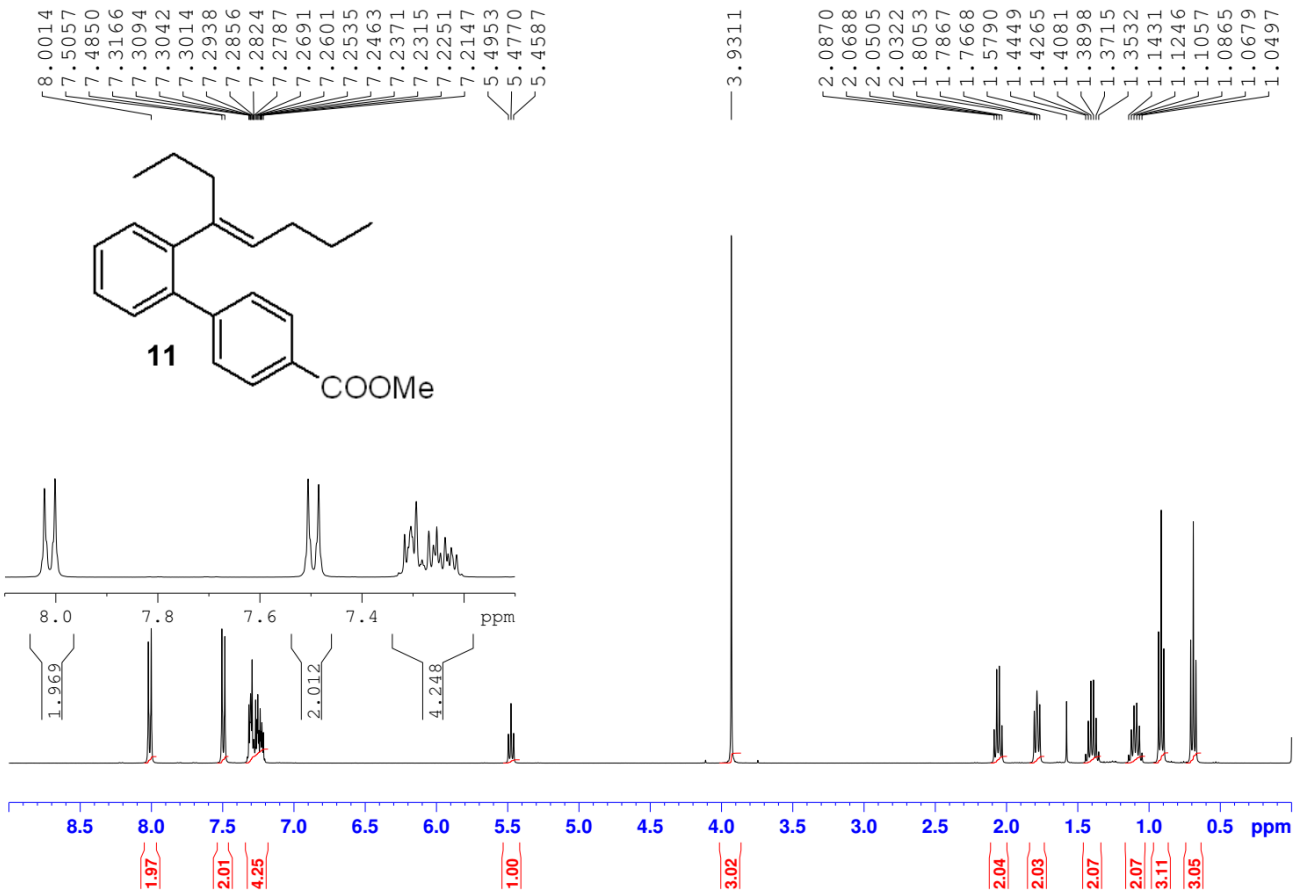
20
10
ppm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5.4587
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1.1431
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1.0679
1.0497
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COOMe
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7.4
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7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
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2.03
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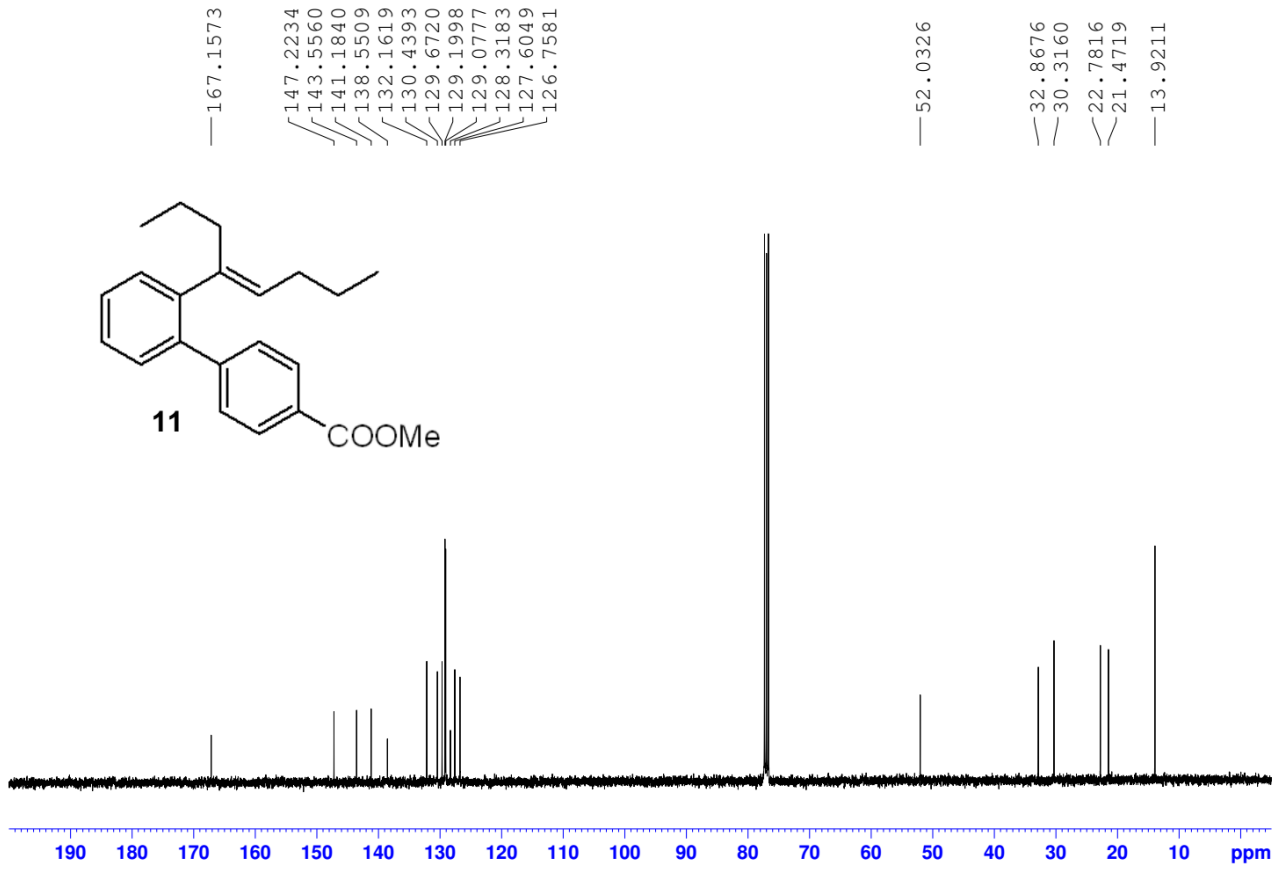
3.05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32.8676
30.3160
22.7816
21.4719
13.9211
11
COOMe
190
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150
140
130
120
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70
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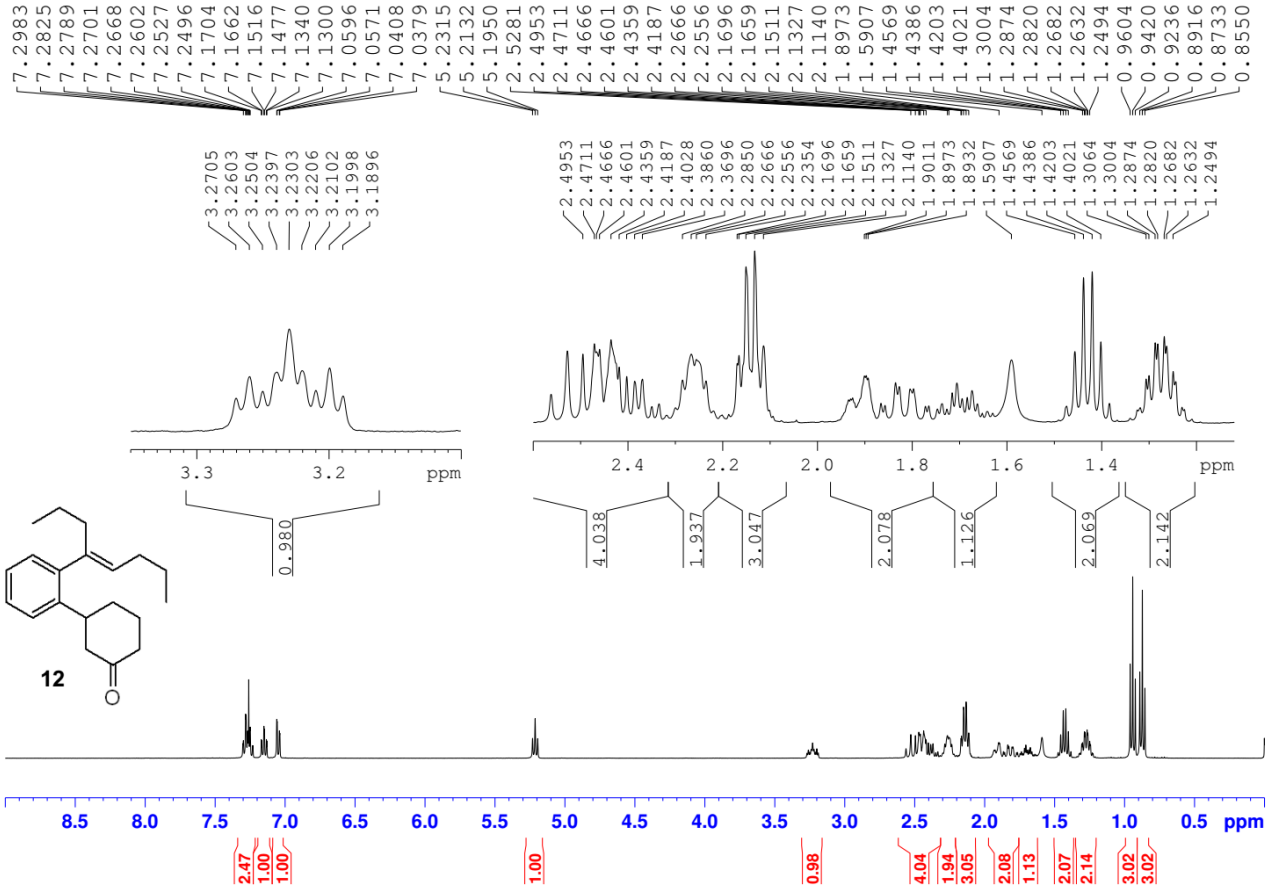
10
ppm


12
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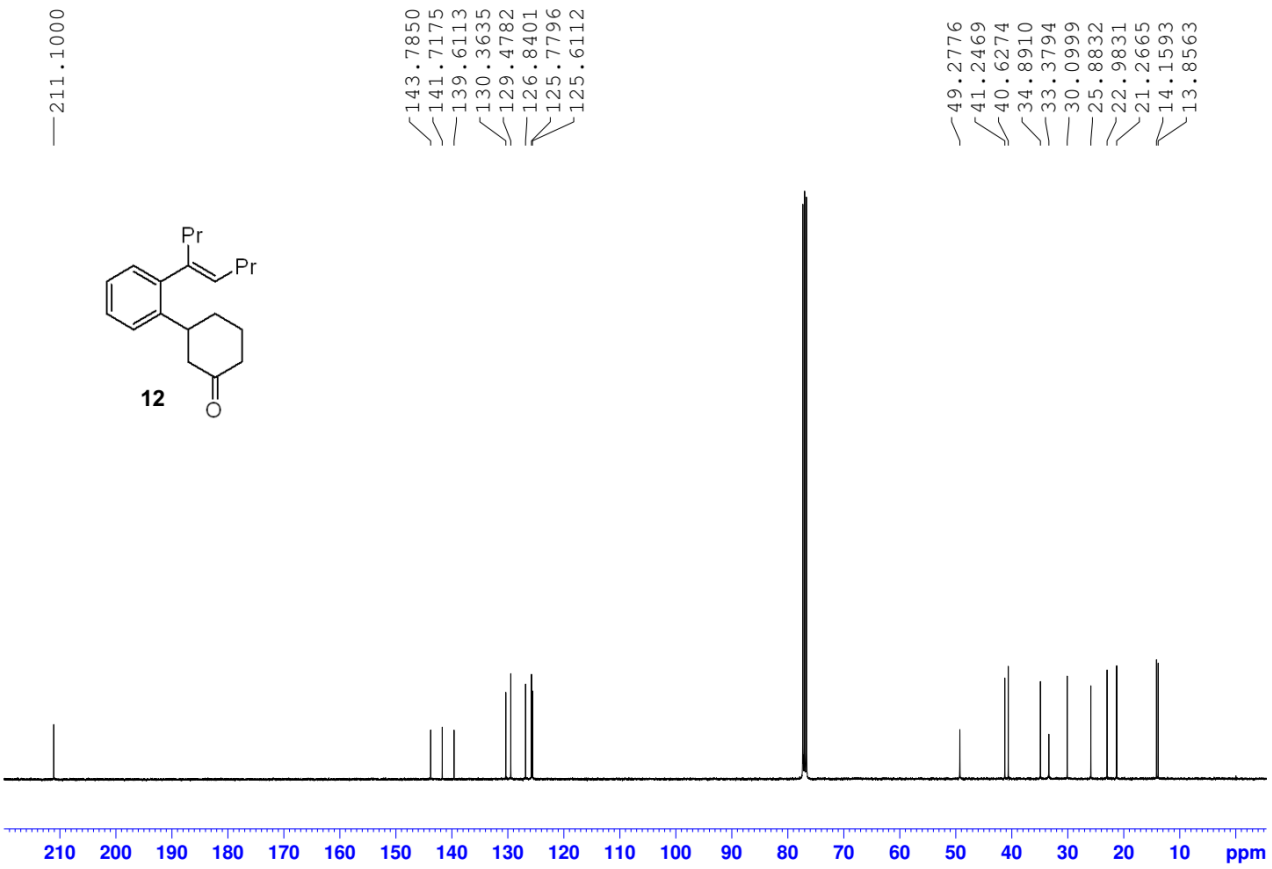
12
Pr
Pr
ppm