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Supporting Information

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Thieme

Supporting Information for:

Copper-Catalyzed Semihydrogenation of Alkynes to (*Z*)-Alkenes

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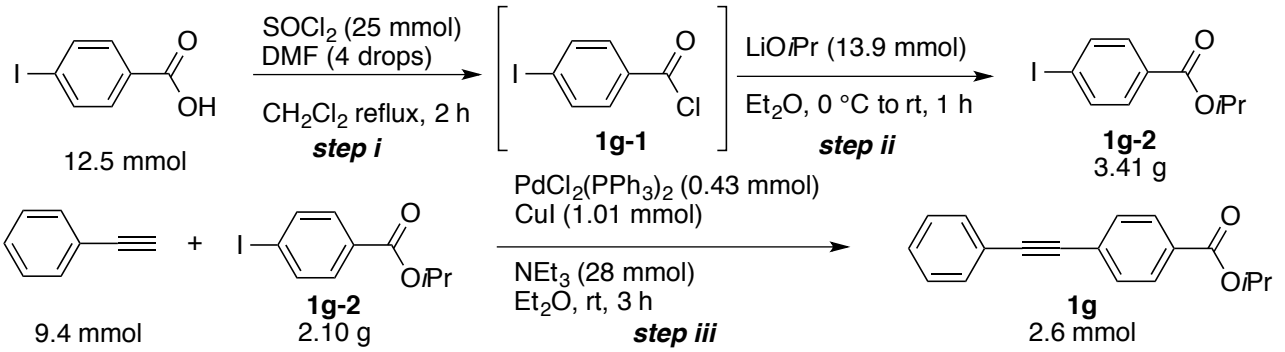
General. All manipulations of oxygen- and moisture-sensitive materials were conducted with a standard schlenk technique under an argon atmosphere or in a glove box under a nitrogen atmosphere. Medium pressure liquid chromatography (MPLC) was performed using Kanto Chemical silica gel 60 (spherical, 40–50 μm) and Biotage® SNAP Ultra. Analytical thin layer chromatography (TLC) was performed on Merck TLC silica gel 60 F_{254} (0.25 mm) plates. Visualization was accomplished with UV light (254 nm). The hydrogenation reactions were performed with the autoclave (EYELA, HIP-7506).

Apparatus. Proton and carbon nuclear magnetic resonance spectra (^{1}H, ^{13}C, and ^{19}F NMR) were recorded on a JEOL ECS-400 (^{1}H NMR, 400MHz; ^{13}C NMR 101 MHz; ^{19}F NMR 376 MHz) spectrometer with solvent resonance as the internal standard (^{1}H NMR, $CHCl_3$ at 7.26 ppm; ^{13}C NMR, $CDCl_3$ at 77.0 ppm; ^{19}F NMR, C_6F_6 at –162 ppm). NMR data are reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, quint = quintet, sext = sextet, sept = septet, br = broad, m = multiplet), coupling constants (Hz), and integration. Medium pressure liquid chromatography (MPLC) was performed with a Shoko Scientific Purif-espoir2 chromatogragh. GC analysis was performed on a Shimadzu GC-2014 equipped with a BP1 column (SGE Analytical Science, 0.25 mm x 30 m, pressure = 149.0 kPa, detector = FID, 290 °C) with helium gas as a carrier. High-resolution mass spectra were obtained with Thermo Scientific Exactive (APCI).

Chemicals. Unless otherwise noted, commercially available chemicals were distilled and degassed before use. $[(PPh_3)CuCl]_4$,[1] **1d**,[2] and **1e**[2] were prepared according to the literature. PPh_3 was purchased from Nacalai Tesque and recrystallized from EtOH before use. The following reagents were purchased from commercial suppliers and used as received: Cu_2O (Aldrich), LiO*t*Bu (Aldrich), LiO*i*Pr (Aldrich), **1c** (Aldrich), **1h** (TCI), CuCl (Strem Chemicals), CuI (Aldrich), CuBr (Nacalai Tesque), CuCN (Nacalai Tesque), CuOAc (Aldrich), anhydrous *i*PrOH (Nacalai Tesque), *i*PrOH-d_8 (99.5 atom% D) (Wako Pure Chemical Industries), 4-iodobenzoic acid (Acros Organics), $PdCl_2(PPh_3)_2$ (TCI), *N*,*N*-dimethylformamide (Nacalai Tesque) and $SOCl_2$ (Nacalai Tesque). Anhydrous toluene, Et_2O, and CH_2Cl_2 was purchased from Kanto Chemical and degassed by purging vigorously with argon for 30 min and further purified by passage through activated alumina under positive argon pressure as described by Grubbs et al.[3]

[1] Jardine, F. H.; Rule, L.; Vohra, A.G. *J. Chem. Soc. A* **1970**, 238.
[2] Park, K.; Bae, G; Moon, J.; Choe, J.; Song, K. H.; Lee, S. L. *J. Org. Chem.* **2010**, *75*, 6244.
[3] Pangborn, A. B.; Giardello, M. A.; Grubbs, R. H.; Rosen, R. K.; Timmers, F. J. *Organometallics* **1996**, *15*, 1518.

Preparation of 1g.

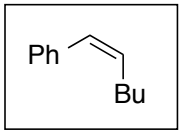


steps i and ii:
A flask was charged with 4-iodobenzoic acid (3.10 g, 12.5 mmol), CH_2Cl_2 (20 mL), $SOCl_2$ (2.0 mL, 25 mmol), and DMF (4 drops) in this order. Then, the mixture was stirred under reflux for 2 h. After removal of all volatiles, the residue was used in the next step without further purification. To a flask containing crude **1g–1** and Et_2O (10 mL), a suspension of LiO*i*Pr (918 mg, 13.9 mmol) in Et_2O (15 mL) was added dropwise at 0 °C. Then, the resulting mixture was stirred at room temperature for 1 h and then it was poured into $NaHCO_3$ aq. and extracted with Et_2O. The combined organic layer was washed with H_2O and dried over $MgSO_4$. After filtration, all of the volatiles were removed in vacuo, and the residue **1g–2** (3.41 g) was used in the next step without further purification.

step iii:
A flask was charged with crude **1g–2** (2.09 g), $PdCl_2(PPh_3)_2$ (300 mg, 0.43 mmol) and CuI (190 mg, 1.01 mmol). The flask was evacuated and refilled with Ar three times, then, to the flask, Et_2O (20 mL), phenylacetylene (1.0 mL, 9.4 mmol) and NEt_3 (3.9 mL, 28 mmol) were added in this order. The mixture was stirred at room temperature for 3 h and then it was poured into NH_4Cl aq. and extracted with Et_2O. The combined organic layer was washed with H_2O and dried over $MgSO_4$. After filtration, all of the volatiles were removed in vacuo. The product was purified by MPLC (160 g of silica gel, eluent: hexane then hexane/EtOAc = 95/5) and further purified by recrystallization from hexane, **1g** was obtained in 36% yield (690 mg, 2.60 mmol) as a yellow solid, R_f = 0.10 (*n*-hexane). ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.02 (d, *J* = 8.1 Hz, 2H), 7.60–7.54 (m, 4H), 7.38–7.36 (m, 3H), 5.26 (sept, *J* = 6.0 Hz, 1H), 1.38 (d, *J* = 6.0 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 165.6, 131.7, 131.4, 130.2, 129.4, 128.7, 128.4, 127.7, 122.7, 92.1, 88.7, 68.6, 21.9. HRMS–APCI(+) (*m*/*z*): $[M+H]^+$ calcd. for $C_{18}H_{17}O_2$, 265.1223; found, 265.1220.

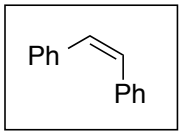
General procedure for Table 1. In a glove box, to a vial, $[(PPh_3)CuCl]_4$ (7.2 mg, 5.0 μmol), toluene (1.0 mL), LiO*t*Bu (40 mg, 0.50 mmol) and *i*PrOH (60 mg, 1.0 mmol) were added in this order. After the resulting mixture was stirred for 1 min at room temperature, **1a** (160 mg, 1.0 mmol) and toluene (2.0 mL) were added. The vial was placed in an autoclave and the autoclave was taken out of the glove box. N_2 in the autoclave was replaced with H_2 by positive pressure of H_2. Then, the mixture was stirred at 100 °C for 3 h under 5 atm of H_2. After cooling to room temperature, H_2 was released and the mixture was diluted with EtOAc. The conversion was determined by GC analysis with *n*-tridecane (18 mg, 0.10 mmol) as an internal standard. The resulting solution was filtered through a pad of silica gel and the yields of the products were determined by ^{1}H NMR analysis with 1,3,5-trimethoxybenzene as an internal standard.

General procedure for entries 3–6 in Table 1. In a glove box, a vial was charged with CuCl (2.0 mg, 0.020 mmol), a phosphine ligand (0.020 mmol) and toluene (1.00 mL), and the resulting solution was stirred for 10 min at room temperature. To this vial, LiO*t*Bu (40 mg, 0.50 mmol) and *i*PrOH (60 mg, 1.0 mmol) were added in this order. After the resulting mixture was stirred for 1 min at room temperature, **1a** (160 mg, 1.0 mmol) and toluene (2.0 mL) were added. The vial was placed in an autoclave and the autoclave was taken out of the glove box. N_2 in the autoclave was replaced with H_2 by positive pressure of H_2. Then, the mixture was stirred at 100 °C for 3 h under 5 atm of H_2. After cooling to room temperature, H_2 was released and the mixture was diluted with EtOAc. The conversion was determined by GC analysis with *n*-tridecane (18 mg, 0.10 mmol) as an internal standard. The resulting solution was filtered through a pad of silica gel and the yields of the products were determined by ^{1}H NMR analysis with 1,3,5-trimethoxybenzene as an internal standard.

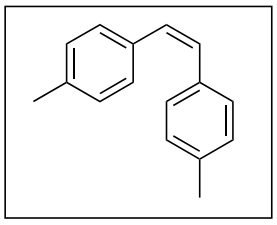


(*Z*)-**2a**. The reaction of **1a** followed by purification by MPLC (16 g of silica gel and Biotage® SNAP Ultra 10 g, *n*-hexane) gave the corresponding products (*Z*)-**2a** (130 mg, 0.80 mmol, 80%) as a colorless oil, R_f = 0.67 (*n*-hexane).^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.37–7.22 (m, 5H), 6.43 (d, *J* = 11.6 Hz, 1H), 5.69 (dt, *J* = 11.6 Hz, 7.3 Hz, 1H), 2.35 (dq, *J* = 7.3 Hz, 1.8 Hz, 2H), 1.48–1.33 (m, 4H), 0.92 (t, *J* = 7.3 Hz, 3H); ^{13}C NMR ($CDCl_3$, 101 MHz): δ 137.8, 133.2, 128.73, 128.68, 128.1, 126.4, 32.2, 28.3, 22.4, 14.0; All the resonances of ^{1}H and ^{13}C NMR spectra were consistent with reported values.[4]

General procedure for Table 2. Similar procedure for Table 1 was employed. The products were purified by MPLC on silica gel to give the corresponding products. In the case of entries 1, 6, 8, and 11, the yields of (*Z*)-**2b**[5a], (*E*)-**2b,**[5a], **3b**,[5b], (*Z*)-**2g**, (*Z*)-**2i**[6], and (*Z*)-5-dodecene[7] were determined by ^{1}H NMR analysis with 1,3,5-trimethoxybenzene as an internal standard.



(*Z*)-**2d**. The reaction of **1d** (180 mg, 1.0 mmol) at 60 °C followed by purification by MPLC (16 g of silica gel and Biotage® SNAP Ultra 10 g, *n*-hexane) gave the corresponding product (160 mg, 0.91 mmol, 91%) as a mixture of (*Z*)-**2c**,[8a] (*E*)-**2d**,[8b] and **3d**[8c] ((*Z*)-**2d**/(*E*)-**2d**/**3d** = 95/5/<1 determined by ^{1}H NMR analysis) as a colorless oil, R_f = 0.53 (hexane). ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.28–7.16 (m, 10H), 6.60 (s, 2H); ^{13}C NMR ($CDCl_3$, 101 MHz): δ 137.2, 130.2, 128.8, 128.2, 127.1. All the resonances of ^{1}H and ^{13}C NMR spectra were consistent with reported values.[8a]



(*Z*)-**2e**. The reaction of **1e** (210 mg, 1.00 mmol) followed by purification by MPLC (16 g of silica gel and Biotage® SNAP Ultra 10 g, *n*-hexane) gave the corresponding product (208 mg, 0.99 mmol, 99%) as a mixture of (*Z*)-**2e**,[9a] (*E*)-**2e**,[9b] and **3e**[9c] ((*Z*)-**2e**/(*E*)-**2e**/**3e** = 98/<1/2 determined by ^{1}H NMR analysis) as a colorless solid, R_f = 0.59 (*n*-hexane). ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.17 (d, *J* = 8.1 Hz, 4H), 7.04 (d, *J* = 8.1 Hz, 4H), 6.50 (s, 2H), 2.32 (s, 6H); ^{13}C NMR ($CDCl_3$, 101 MHz): δ 136.7, 134.5, 129.5, 128.8, 128.7, 21.2. All the resonances of ^{1}H and ^{13}C NMR spectra were consistent with reported values.[9a]

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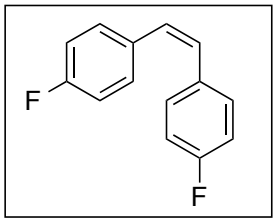
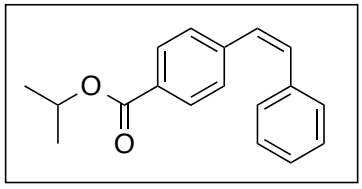
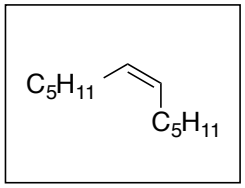
[4] Cahiez, G.; Gager, O.; Lecomte, F. *Org. Lett.* **2008**, *10*, 5255.
[5] (a) Chen, C.; Dugan, T. R.; Brennessel, W. W.; Weix, D. J.; Holland, P. L. *J. Am. Chem. Soc.* **2014**, *136*, 945. (b) Carter, T. S.; Guiet, L.; Frank, D. J.; West, J.; Thomas, S. P. *Adv. Synth. Catal.* **2013**, *355*, 880.
[6] Belger, C.; Neisius. N. M.; Plietker. B. *Chem. Eur. J.* **2010**, *16*, 12214.
[7] Cahiez, G.; Gager, O.; Buendia, J.; Patinote, C. *Chem. Eur. J.* **2012**, *18*, 5860.
[8] (a) Yan, M.; Jin, T.; Ishikawa, Y.; Minato, T.; Fujita, T.; Chen, L.-Y.; Bao, M.; Asao, N.; Chen, M.-W.; Yamamoto, Y. *J. Am. Chem. Soc.* **2012**, 134, 17536. (b) Chen, Z.; Luo, M.; Wen, Y.; Luo, G.; Liu, L. *Org. Lett.* **2014**, *16*, 3020. (c) Kantam. M. L.; Kishore. R.; Yadav, J.; Sudhakar, M.; Venugopal, A. *Adv. Synth. Catal.* **2012**, *354*, 663.
[9] (a) Li. J.; Hua. R.; Liu. T. *J. Org. Chem.* **2010**, *75*, 2966. (b) Kantam, M. L.; Srinivas. P.; Yadav. J.; Likhar. P. R.; Bhargava, S. *J. Org. Chem.* **2009**, *74*, 4882. (c) Chen. T.; Yang, Li. L.; Huang. K.-W. *Tetrahedron* **2012**, *68*, 6152.



(*Z*)-**2f**. The reaction of **1f** (214 mg, 1.00 mmol) followed by purification by MPLC (16 g of silica gel and Biotage® SNAP Ultra 10 g, *n*-hexane) gave the corresponding product (206 mg, 0.95 mmol, 95%) as a mixture of (*Z*)-**2f**,[10] (*E*)-**2f**,[10] and **3f**[9c] ((*Z*)-**2f**/(*E*)-**2f**/**3f** = 99/<1/1 determined by ^{1}H NMR analysis) as a colorless solid, R_f = 0.59 (*n*-hexane). ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.20–7.16 (m, 4H), 6.94–6.90 (m, 4H), 6.54 (s, 2H); ^{13}C NMR ($CDCl_3$, 101 MHz): δ 161.8 (d, *J* = 246.3 Hz), 132.9 (d, *J* = 2.9 Hz), 130.5 (d, *J* = 7.8 Hz), 129.1, 115.2 (d, *J* = 22.0 Hz); ^{19}F NMR ($CDCl_3$, 376 MHz): δ –114.7. All the resonances of ^{1}H and ^{13}C spectrum was consistent with reported values.[10]



(*Z*)-**2g**. The reaction of **1g** (79 mg, 0.3 mmol) at 80 °C in toluene (0.8 mL) followed by purification by MPLC (16 g of silica gel and Biotage® SNAP Ultra 10 g, *n*-hexane/EtOAc = 99/1 to 93/7) gave the corresponding products (*Z*)-**2g** (42 mg, 0.16 mmol, 52%) as a pale yellow oil, R_f = 0.35 (*n*-hexane/EtOAc = 95/5). ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.90 (d, *J* = 8.5 Hz, 2H), 7.30 (d, *J* = 8.5 Hz, 2H), 7.25–7.20 (m, 5H), 6.71 (d, *J* = 12.2 Hz, 1H), 6.61 (d, *J* = 12.2 Hz, 1H), 5.24 (sept, *J* = 6.1 Hz, 1H), 1.36 (d, *J* = 6.1 Hz, 6H); ^{13}C NMR ($CDCl_3$, 101 MHz): δ 165.9, 141.8, 136.7, 132.1, 129.4, 129.31, 129.28, 128.83, 128.74, 128.3, 127.4, 68.3, 21.9. HRMS–APCI(+) (*m*/*z*): $[M+H]^+$ calcd. for $C_{18}H_{19}O_2$, 267.1380; found, 267.1375.



(*Z*)-**2h**. The reaction of **1h** (170 mg, 1.0 mmol) followed by purification by MPLC (16 g of silica gel and Biotage® SNAP Ultra 10 g, *n*-hexane) gave the corresponding products (*Z*)-**2h** (130 mg, 0.76 mmol, 76%) as a colorless oil, R_f = 0.92 (*n*-hexane). ^{1}H NMR ($CDCl_3$, 400 MHz): δ 5.36 (m, 2H), 2.03 (q, *J* = 6.4 Hz, 4H), 1.39–1.24 (m, 12H), 0.89 (t, *J* = 6.9 Hz, 6H); ^{13}C NMR ($CDCl_3$, 101 MHz): δ 129.9, 31.6, 29.5, 27.2, 22.6, 14.1. All the resonances of ^{1}H and ^{13}C NMR spectra were consistent with reported values.[11]

[10] Shi, M.; Xu, B. *J. Org. Chem.* **2002**, *67*, 294.
[11] Semba. K.; Fujihara. T.; Xu. T.; Terao. J.; Tsuji. Y. *Adv. Synth. Catal.* **2012**, *354*, 1542.

The procedure for Eq. 1. Similar procedure for Table 1 was employed by using *i*PrOH-d_8 instead of *i*PrOH. The yield and deuterium incorporation were determined by ^{1}H NMR analysis (Figure S1 and S2).

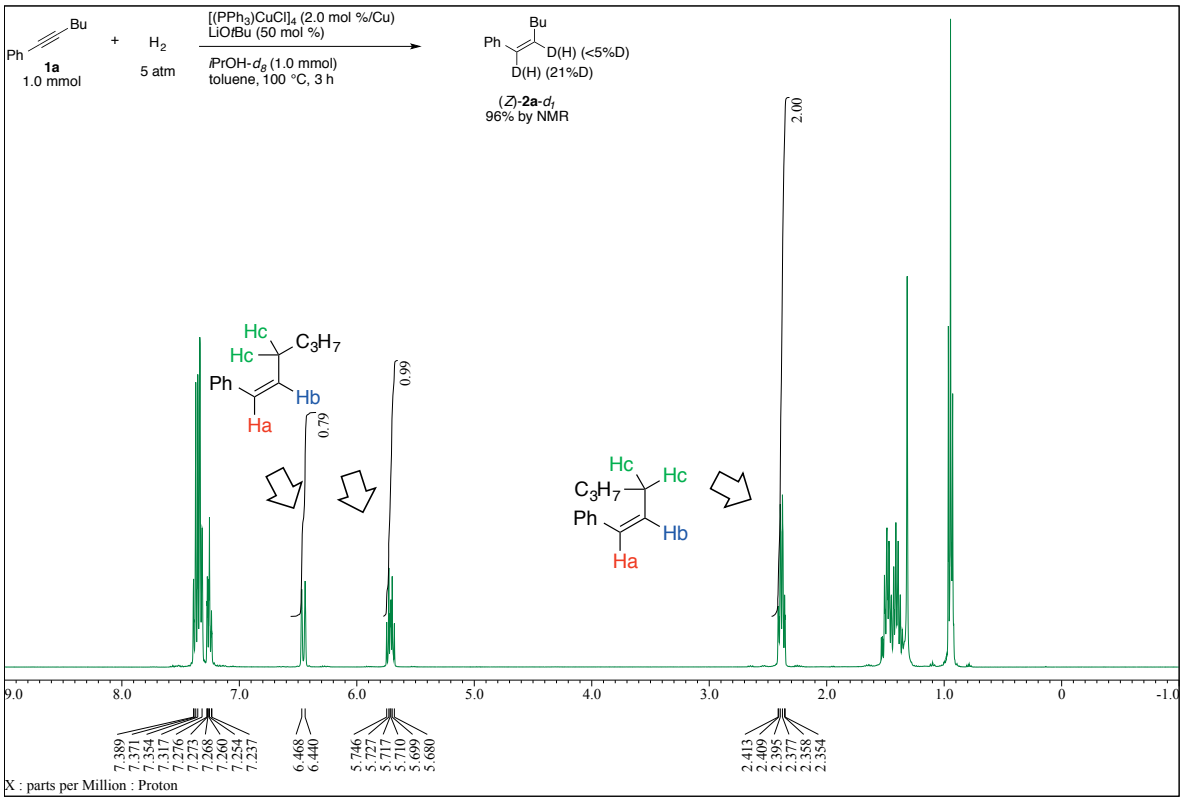


Figure S1.

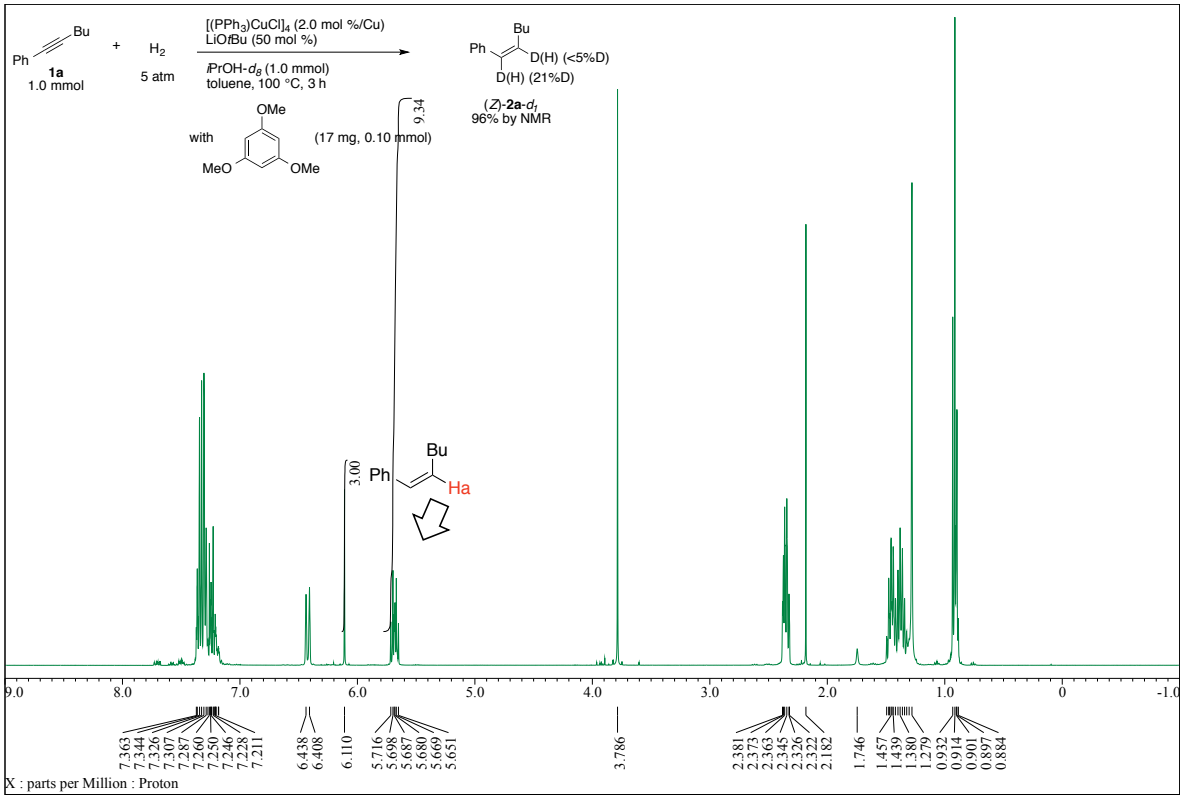
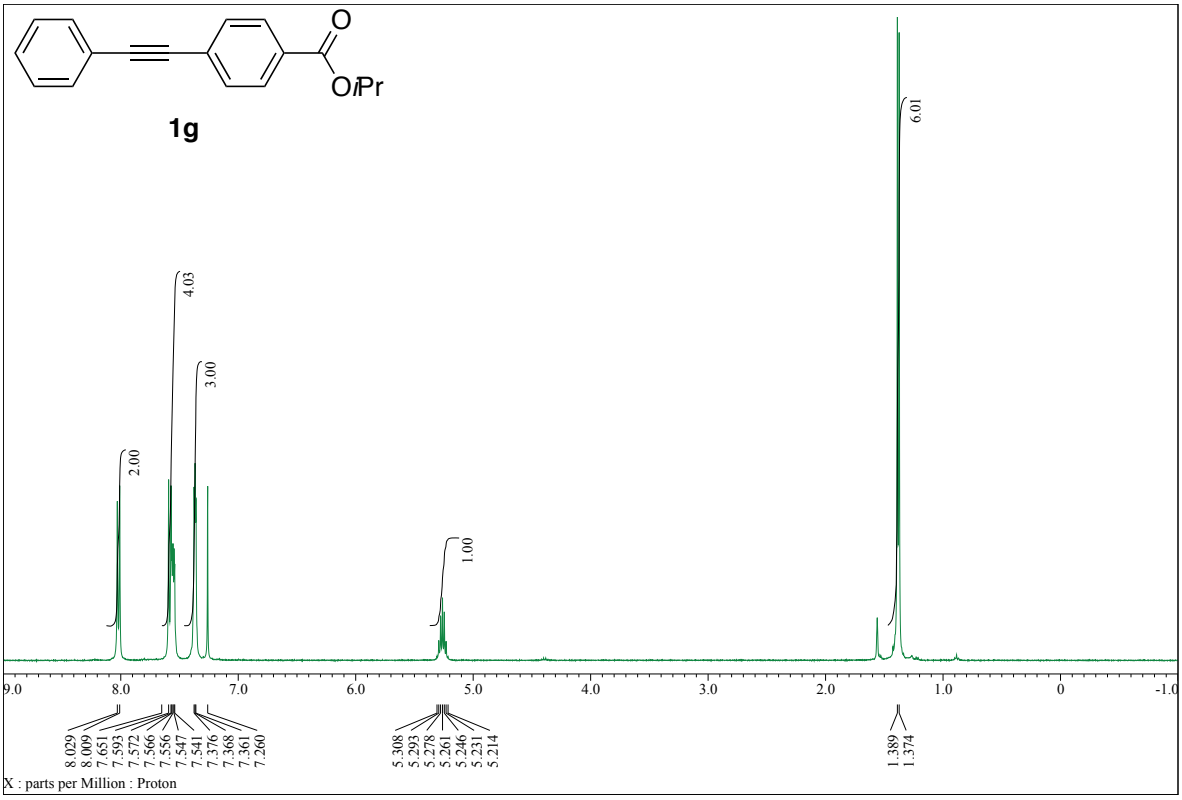


Figure S2.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4.03
3.00
2.00
1.00
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
-1.0
8.029
8.009
7.651
7.593
7.572
7.566
7.556
7.547
7.541
7.376
7.368
7.361
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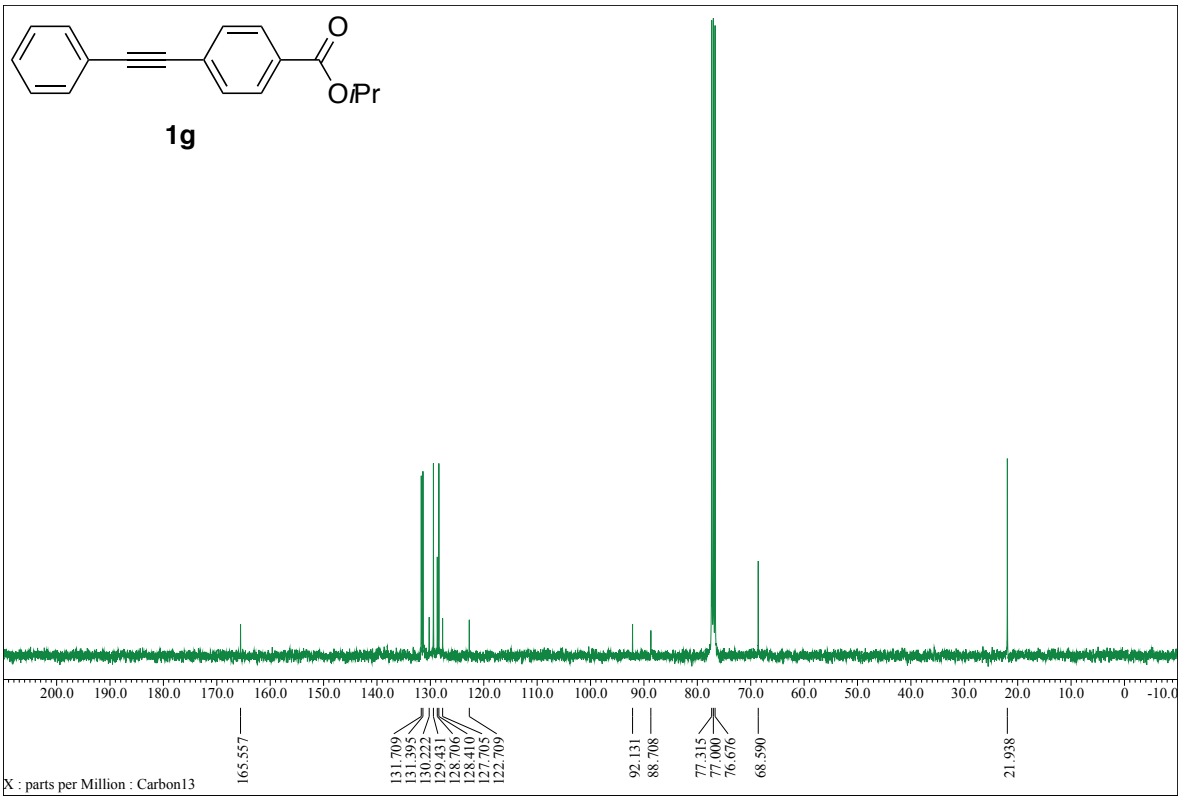
5.231
5.214
1.389
1.374
X : parts per Million : Proton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160.0
150.0
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120.0
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100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
-10.0
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131.709
131.395
130.222
129.431
128.706
128.410
127.705
122.709
92.131
88.708
77.315
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76.676
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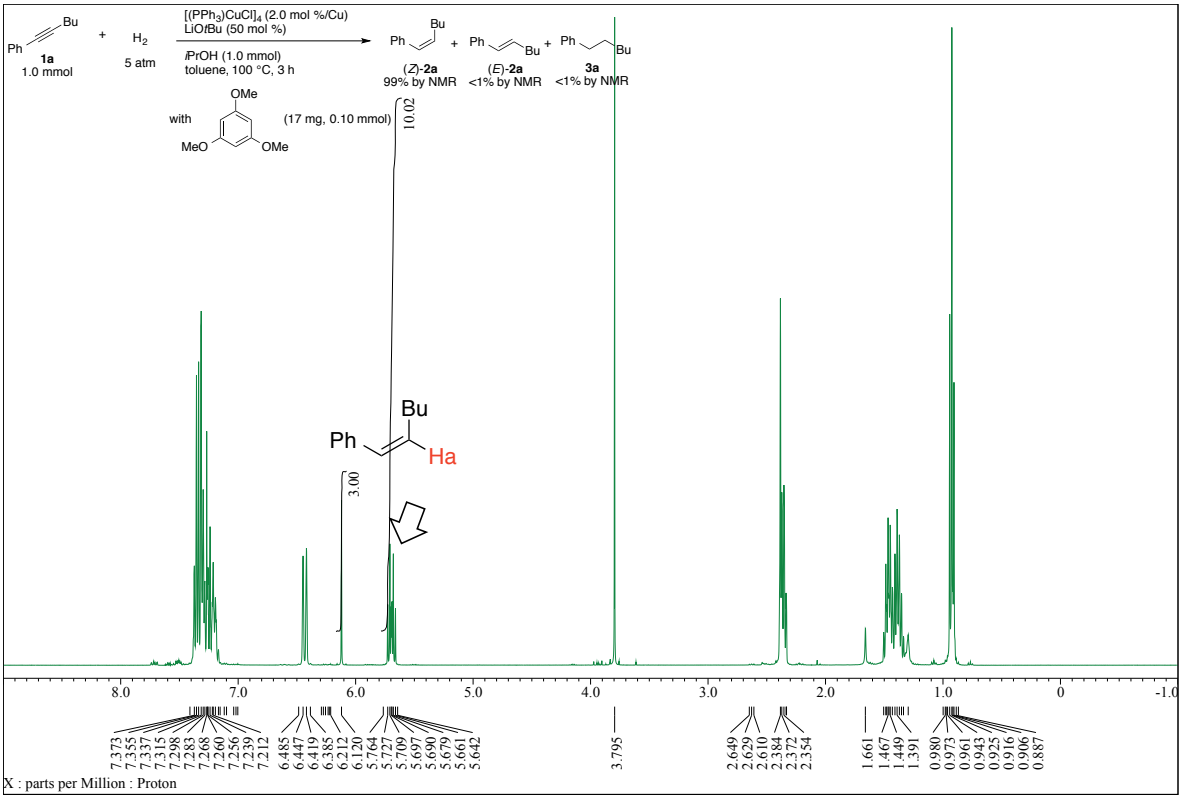
21.938
X : parts per Million : Carbon13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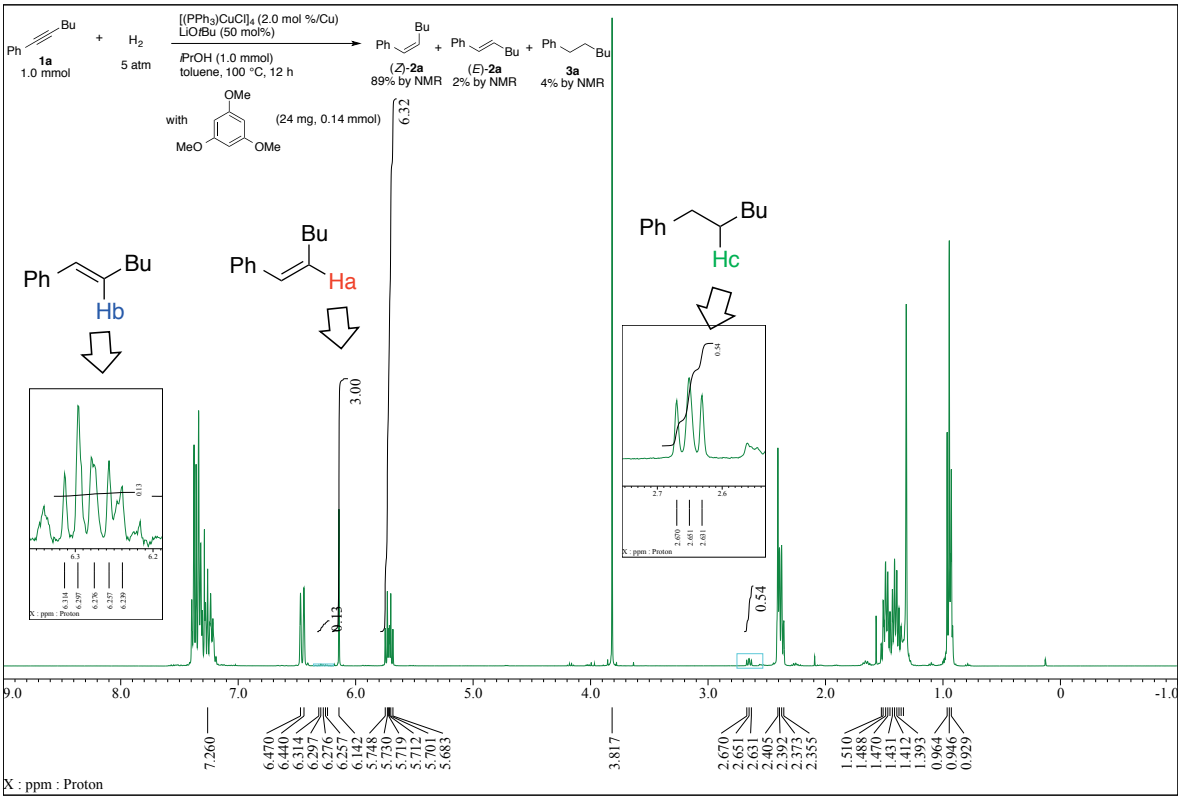
1.0 mmol
5 atm
(Z)-2a
99% by NMR
(E)-2a
<1% by NMR
3a
<1% by NMR
with
(17 mg, 0.10 mmol)
Ha
10.02
3.00
X : parts per Million : Proton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LiOtBu (50 mol%)
iPrOH (1.0 mmol)
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1a
1.0 mmol
5 atm
(Z)-2a
89% by NMR
(E)-2a
2% by NMR
3a
4% by NMR
with
(24 mg, 0.14 mmol)
Hb
Ha
Hc
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3.00
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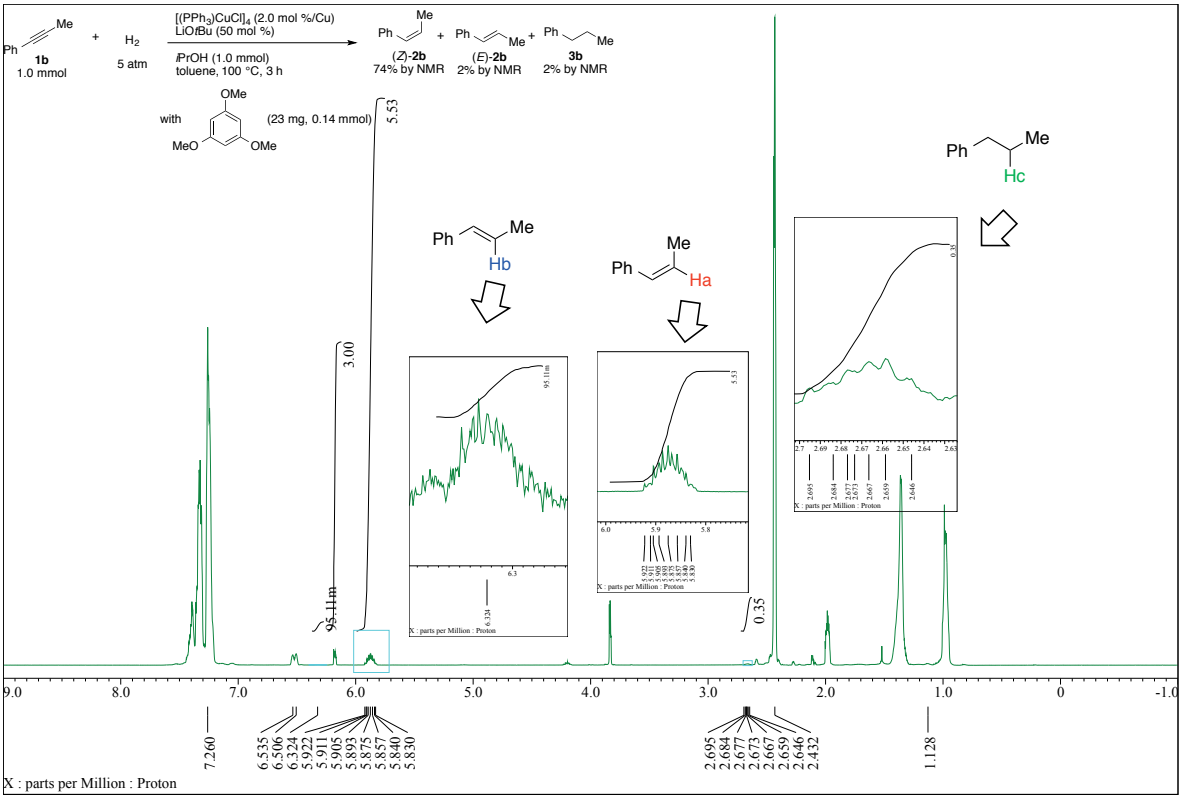
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LiOtBu (50 mol %)
iPrOH (1.0 mmol)
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1b
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5 atm
(Z)-2b
74% by NMR
(E)-2b
2% by NMR
3b
2% by NMR
with
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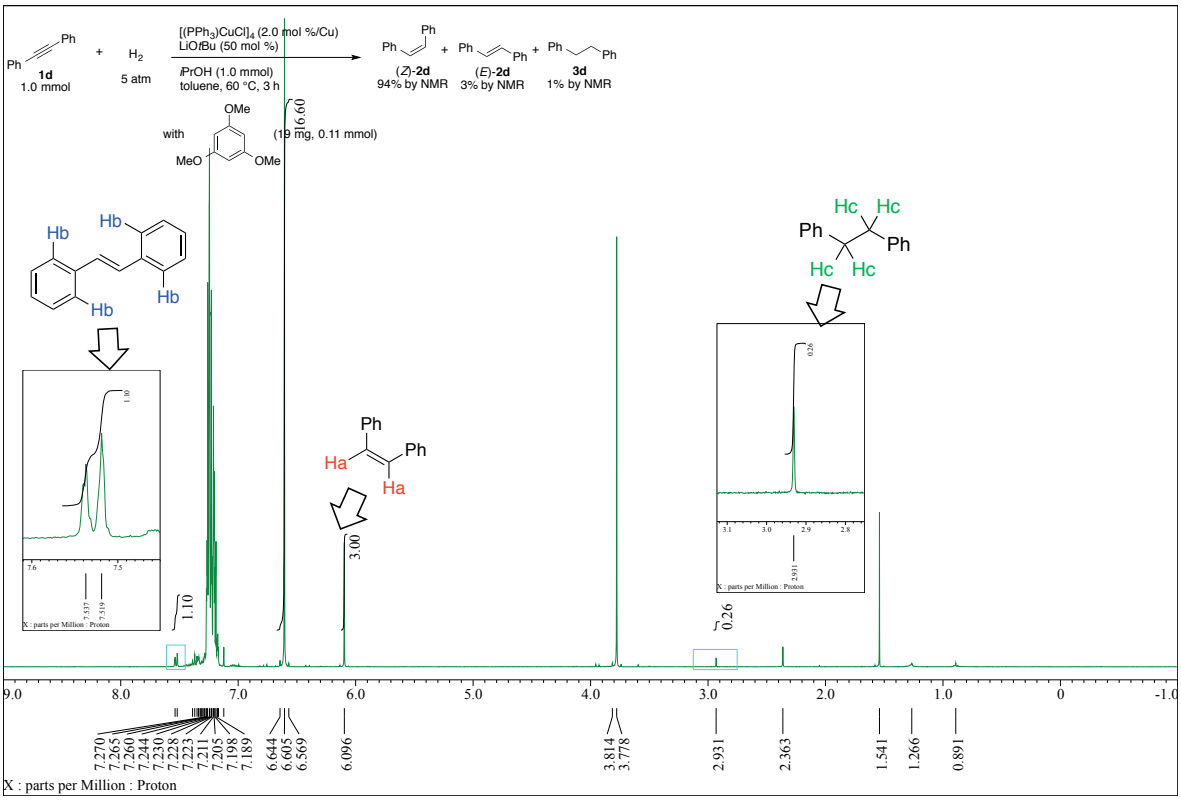
3.00
95.11m
0.35
Hb
Ha
Hc
X : parts per Million : Proton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LiOtBu (50 mol %)
iPrOH (1.0 mmol)
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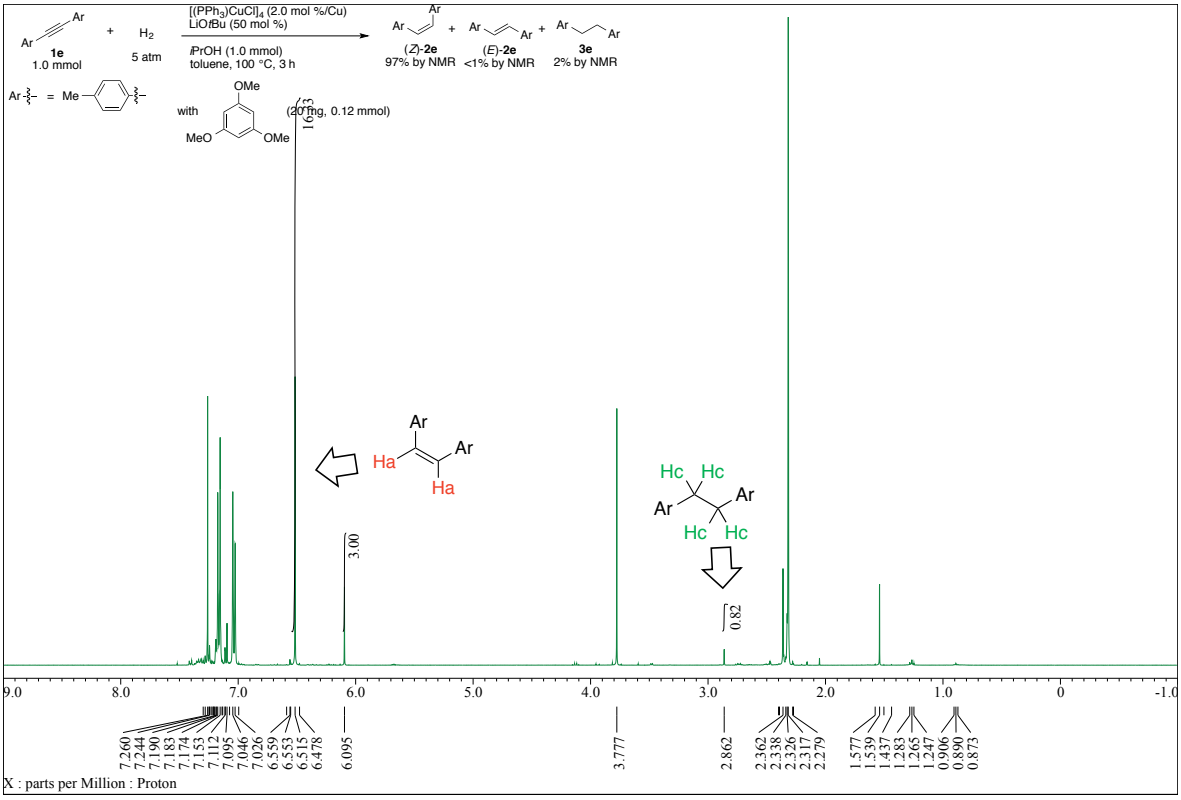
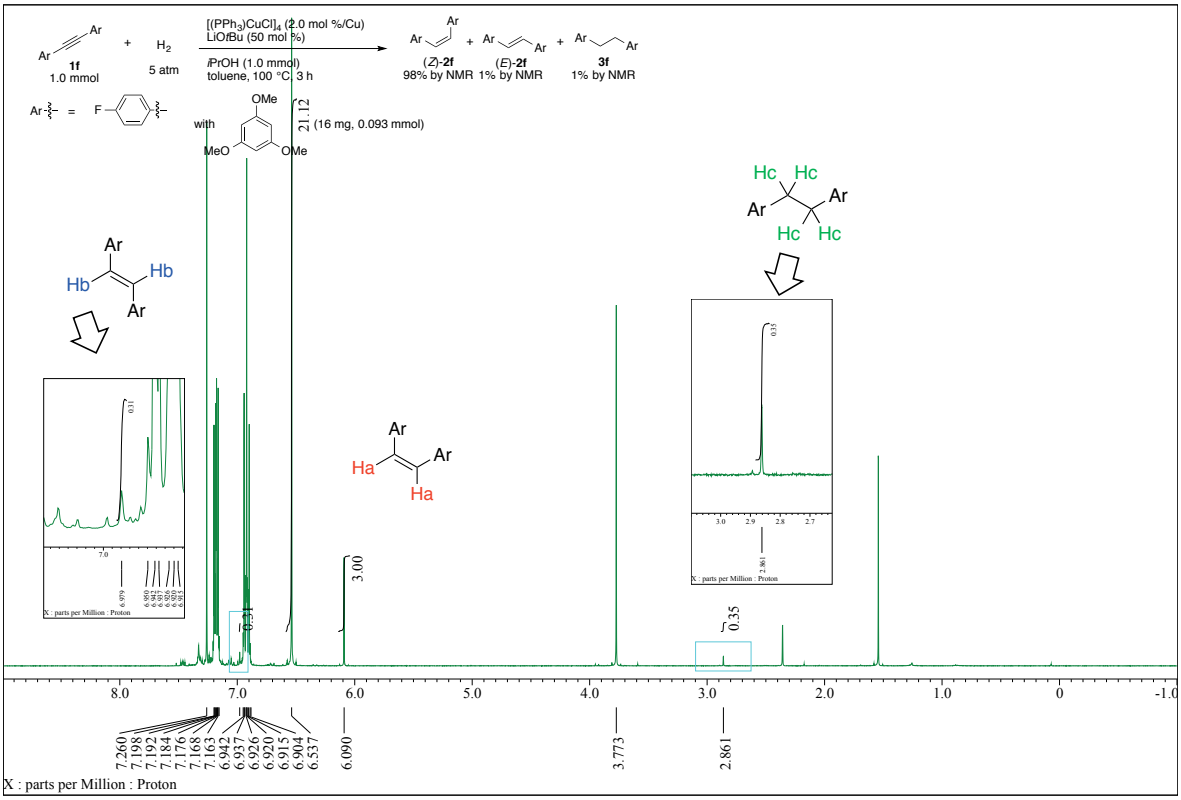
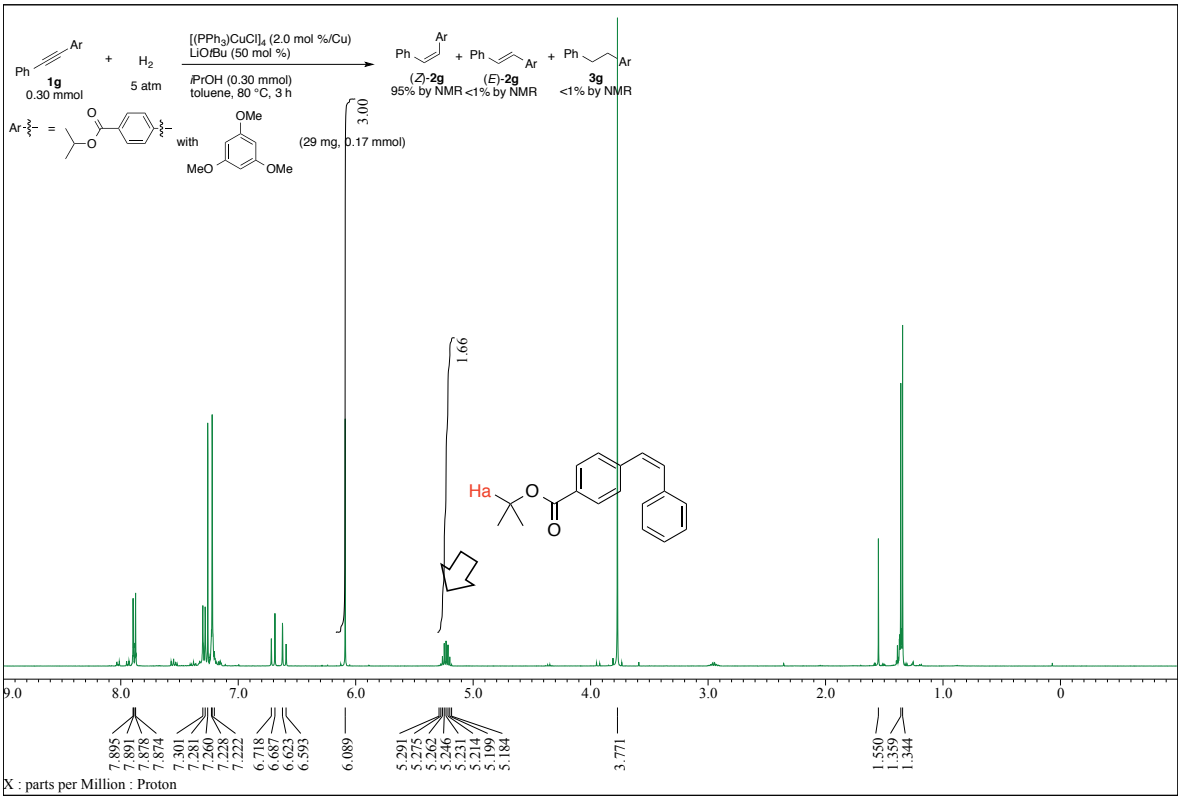
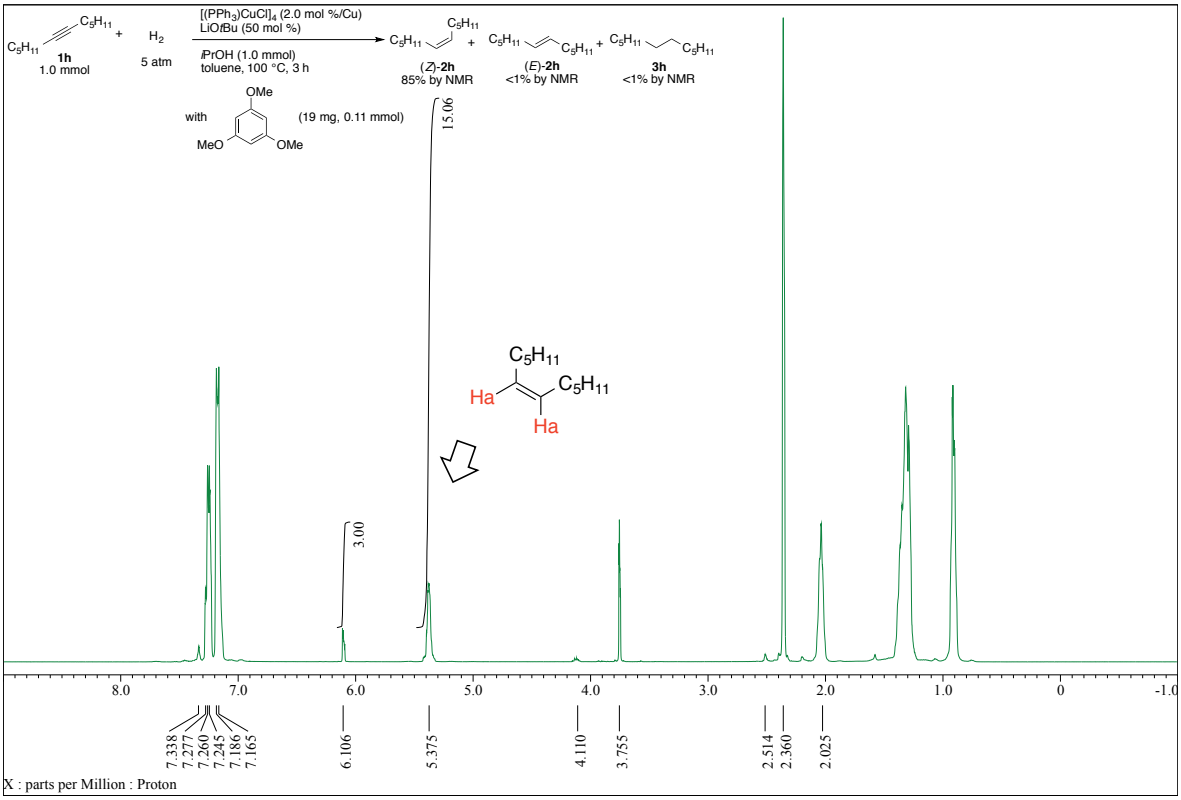
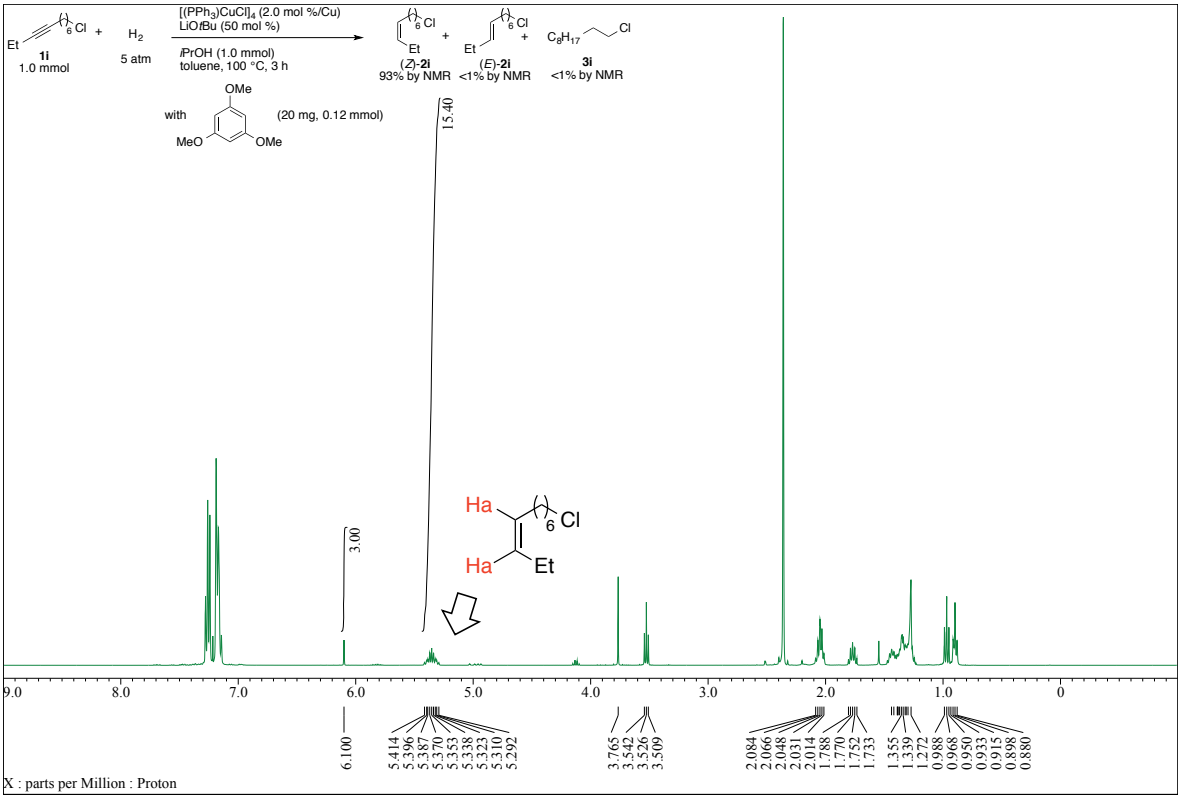
1d
1.0 mmol
H2
5 atm
(Z)-2d
94% by NMR
(E)-2d
3% by NMR
3d
1% by NMR
with
(19 mg, 0.11 mmol)
6.60
1.10
3.00
0.26
Hb
Ha
Hc
X : parts per Million : Proton

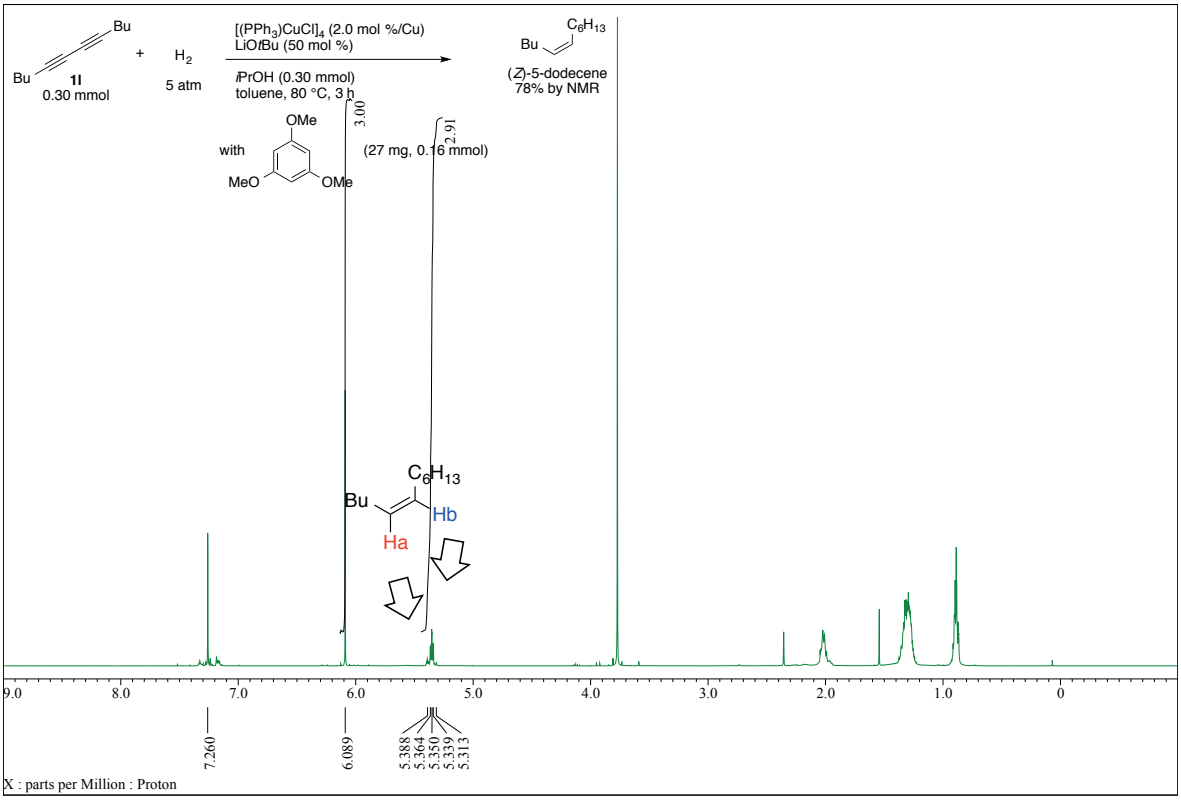

[(PPh3)CuCl]4 (2.0 mol %/Cu)
LiOtBu (50 mol %)
iPrOH (1.0 mmol)
toluene, 100 °C, 3 h
1e
1.0 mmol
5 atm
(Z)-2e
97% by NMR
(E)-2e
<1% by NMR
3e
2% by NMR
Ar = Me
with
(20 mg, 0.12 mmol)
16.33
3.00
0.82
Ha
Hc
X : parts per Million : Proton

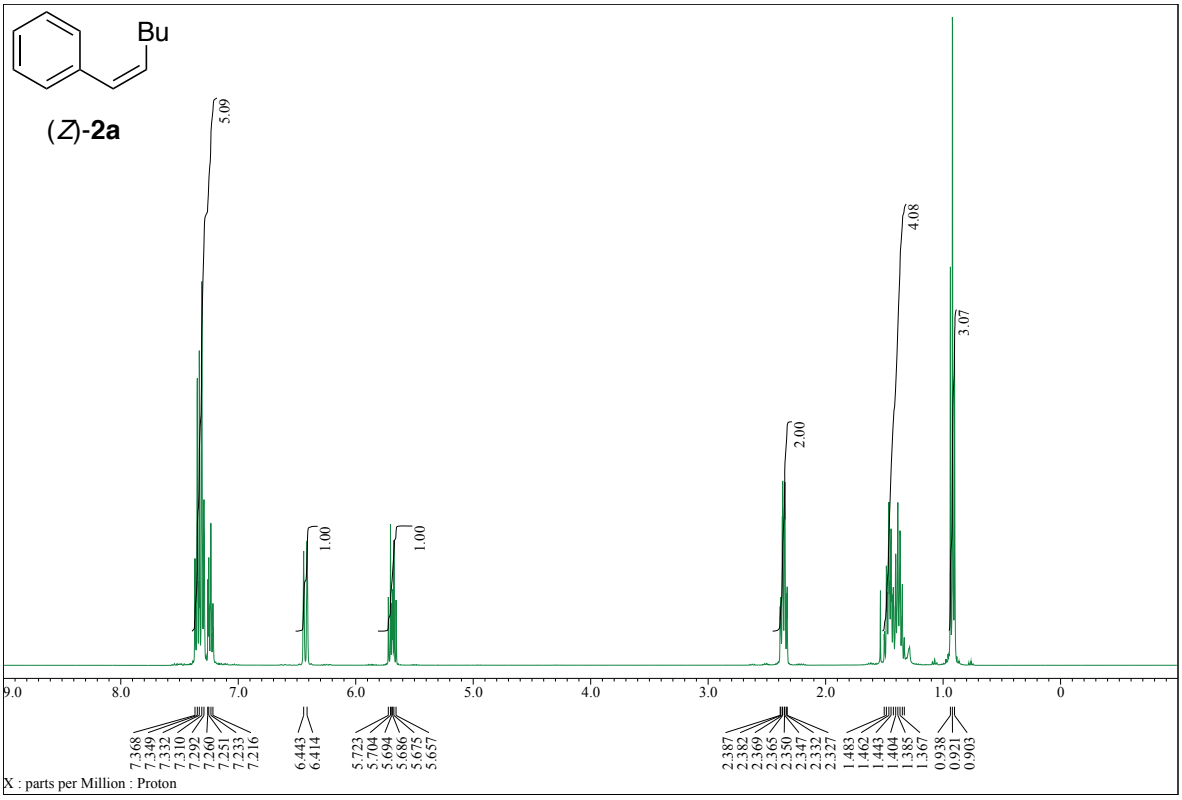

[(PPh3)CuCl]4 (2.0 mol %/Cu)
LiOtBu (50 mol %)
iPrOH (1.0 mmol)
toluene, 100 °C, 3 h
1f
1.0 mmol
5 atm
(Z)-2f
98% by NMR
(E)-2f
1% by NMR
3f
1% by NMR
Ar = F
with
(16 mg, 0.093 mmol)
21.12
3.00
0.31
0.35
Ha
Hb
Hc
X : parts per Million : Proton

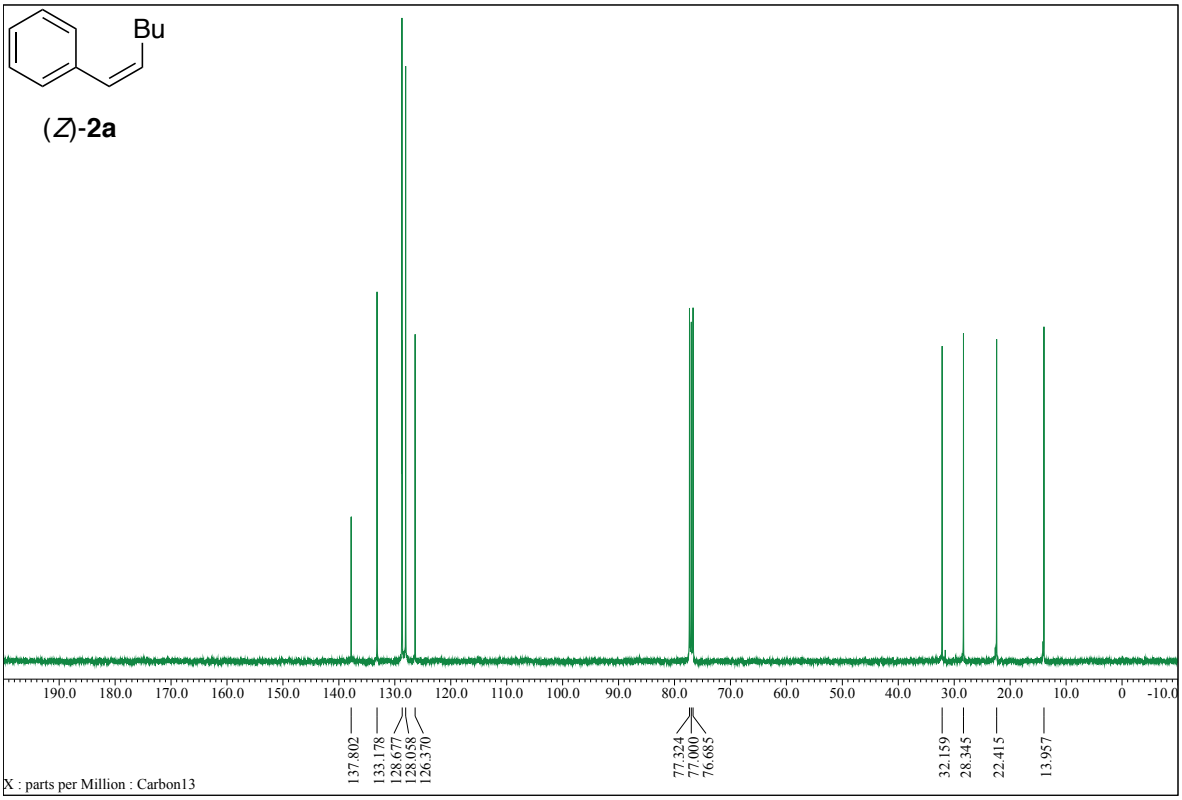

[(PPh3)CuCl]4 (2.0 mol %/Cu)
LiOtBu (50 mol %)
iPrOH (0.30 mmol)
toluene, 80 °C, 3 h
1g
0.30 mmol
5 atm
with
(29 mg, 0.17 mmol)
(Z)-2g
95% by NMR
(E)-2g
<1% by NMR
3g
<1% by NMR
3.00
1.66
Ha
7.895
7.891
7.878
7.874
7.301
7.281
7.260
7.228
7.222
6.718
6.687
6.623
6.593
6.089
5.291
5.275
5.262
5.246
5.231
5.214
5.199
5.184
3.771
1.550
1.359
1.344
X : parts per Million : Proton

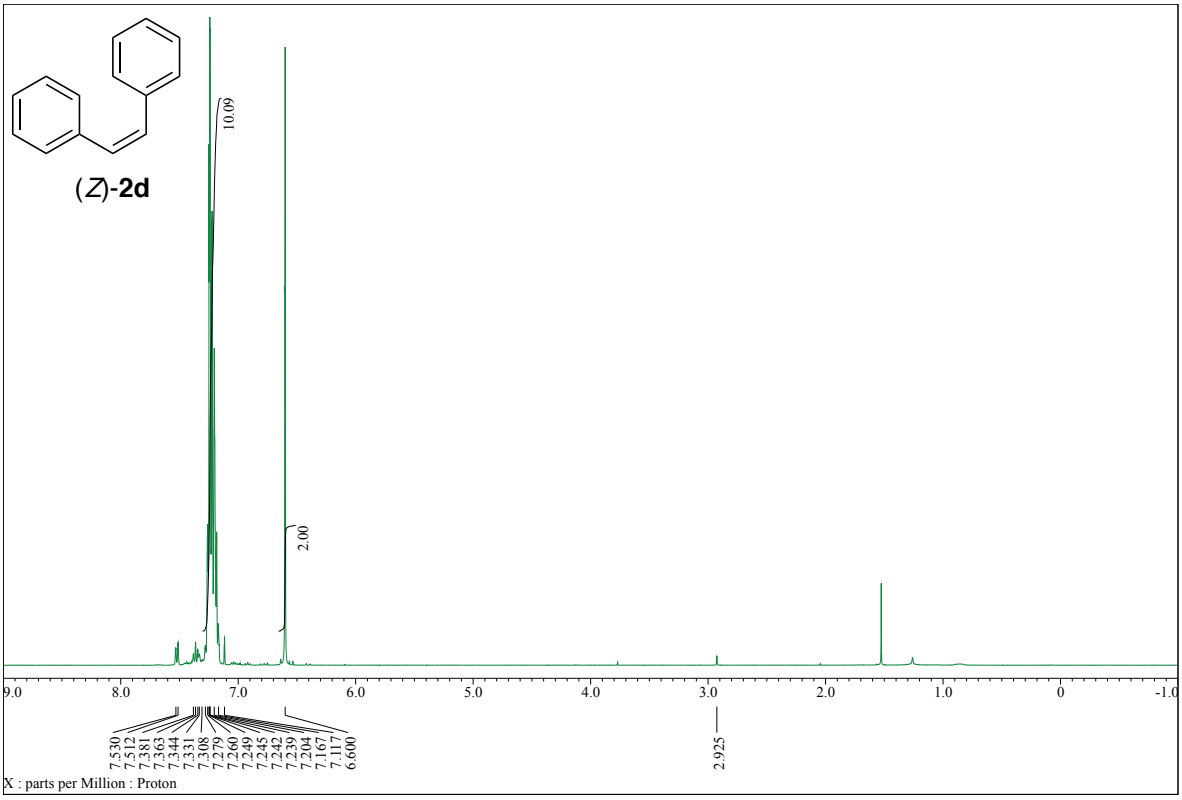

[(PPh3)CuCl]4 (2.0 mol %/Cu)
LiOtBu (50 mol %)
iPrOH (1.0 mmol)
toluene, 100 °C, 3 h
1h
1.0 mmol
5 atm
with
(19 mg, 0.11 mmol)
(Z)-2h
85% by NMR
(E)-2h
<1% by NMR
3h
<1% by NMR
15.06
3.00
Ha
Ha
7.338
7.277
7.260
7.245
7.186
7.165
6.106
5.375
4.110
3.755
2.514
2.360
2.025
X : parts per Million : Proton

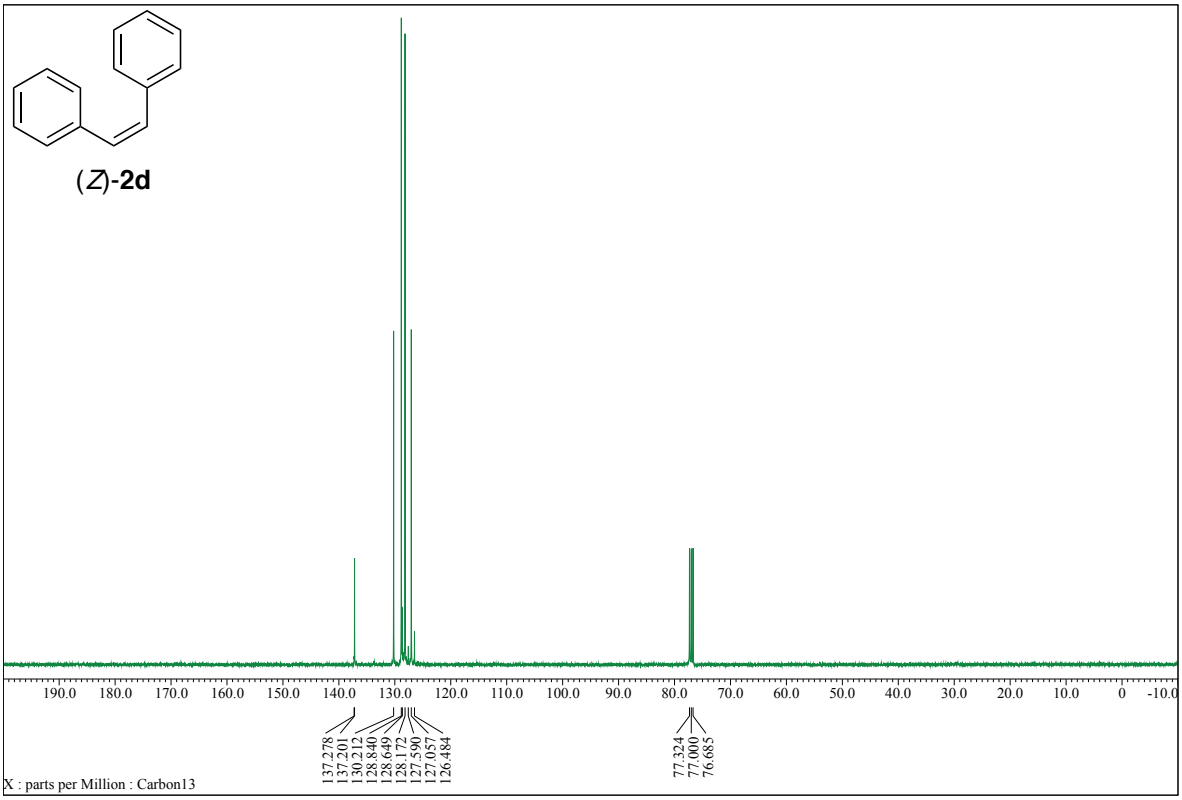

[(PPh3)CuCl]4 (2.0 mol %/Cu)
LiOtBu (50 mol %)
iPrOH (1.0 mmol)
toluene, 100 °C, 3 h
1i
1.0 mmol
5 atm
(Z)-2i
93% by NMR
(E)-2i
<1% by NMR
3i
<1% by NMR
with
(20 mg, 0.12 mmol)
OMe
MeO
OMe
15.40
3.00
Ha
Cl
Ha
Et
X : parts per Million : Proton

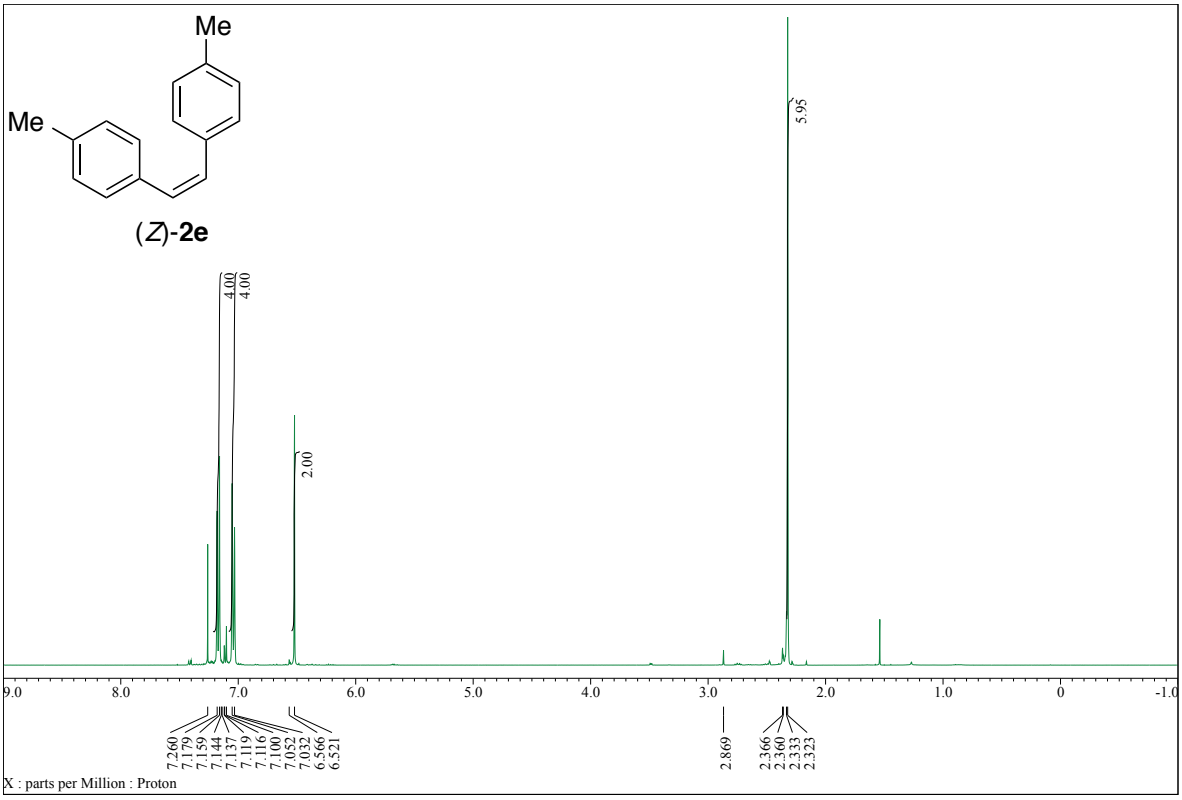

[(PPh3)CuCl]4 (2.0 mol %/Cu)
LiOtBu (50 mol %)
iPrOH (0.30 mmol)
toluene, 80 °C, 3 h
1l
0.30 mmol
5 atm
(Z)-5-dodecene
78% by NMR
with
(27 mg, 0.16 mmol)
OMe
MeO
OMe
3.00
2.91
C6H13
Bu
Hb
Ha
X : parts per Million : Proton

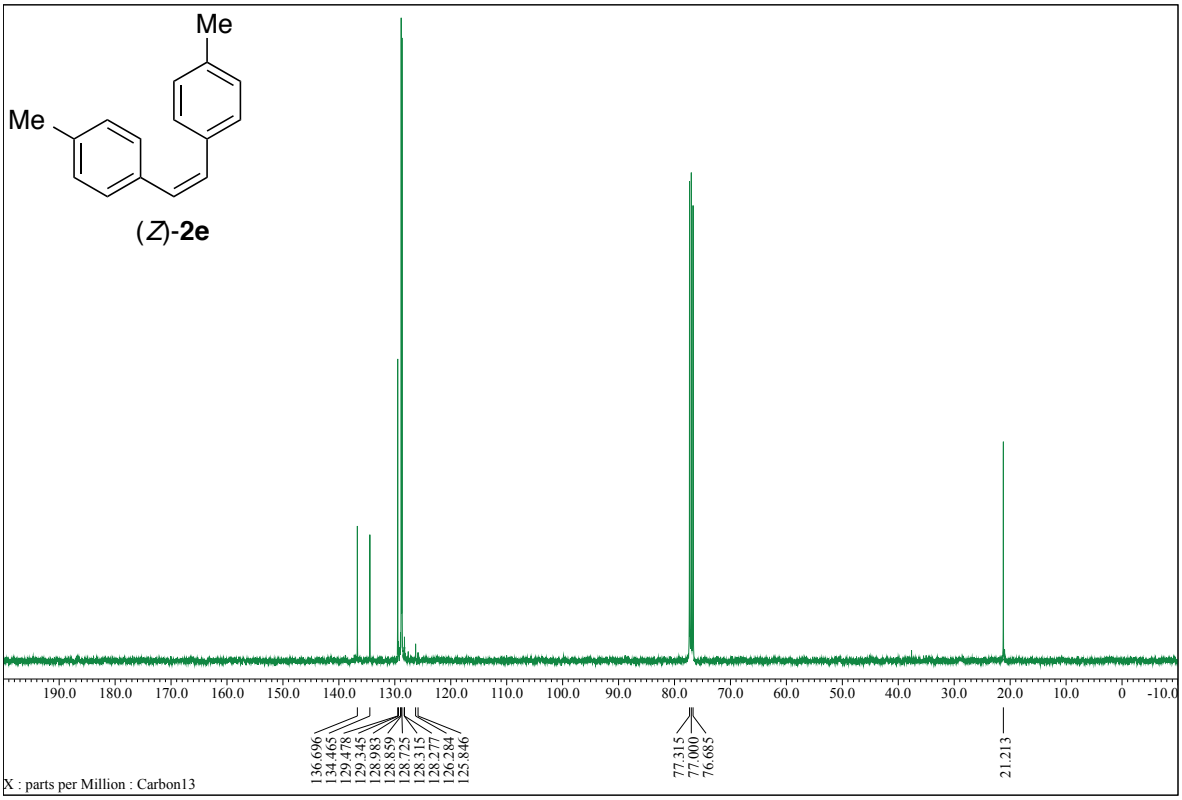

Bu
(Z)-2a
5.09
1.00
1.00
2.00
4.08
3.07
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.368
7.349
7.332
7.310
7.292
7.260
7.251
7.233
7.216
6.443
6.414
5.723
5.704
5.694
5.686
5.675
5.657
2.387
2.382
2.369
2.365
2.350
2.347
2.332
2.327
1.483
1.462
1.443
1.404
1.385
1.367
0.938
0.921
0.903
X : parts per Million : Proton

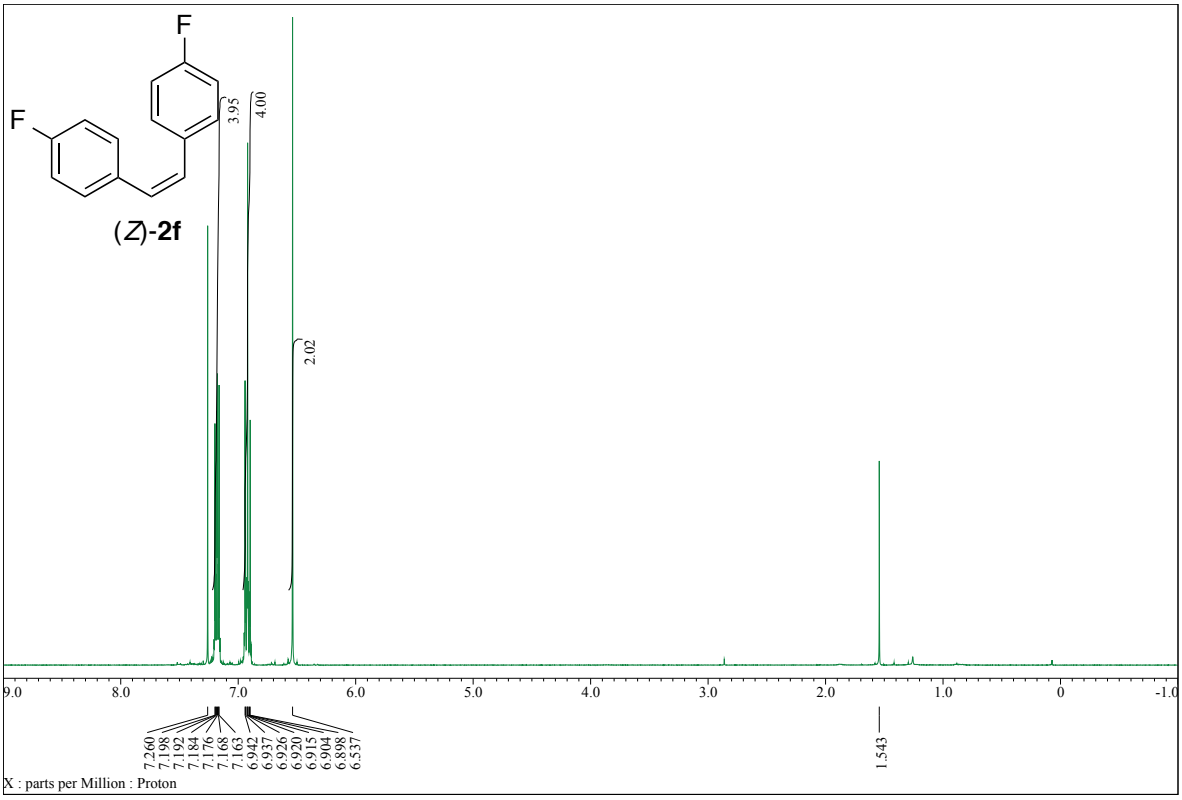

Bu
(Z)-2a
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
-10.0
137.802
133.178
128.677
128.058
126.370
77.324
77.000
76.685
32.159
28.345
22.415
13.957
X : parts per Million : Carbon13

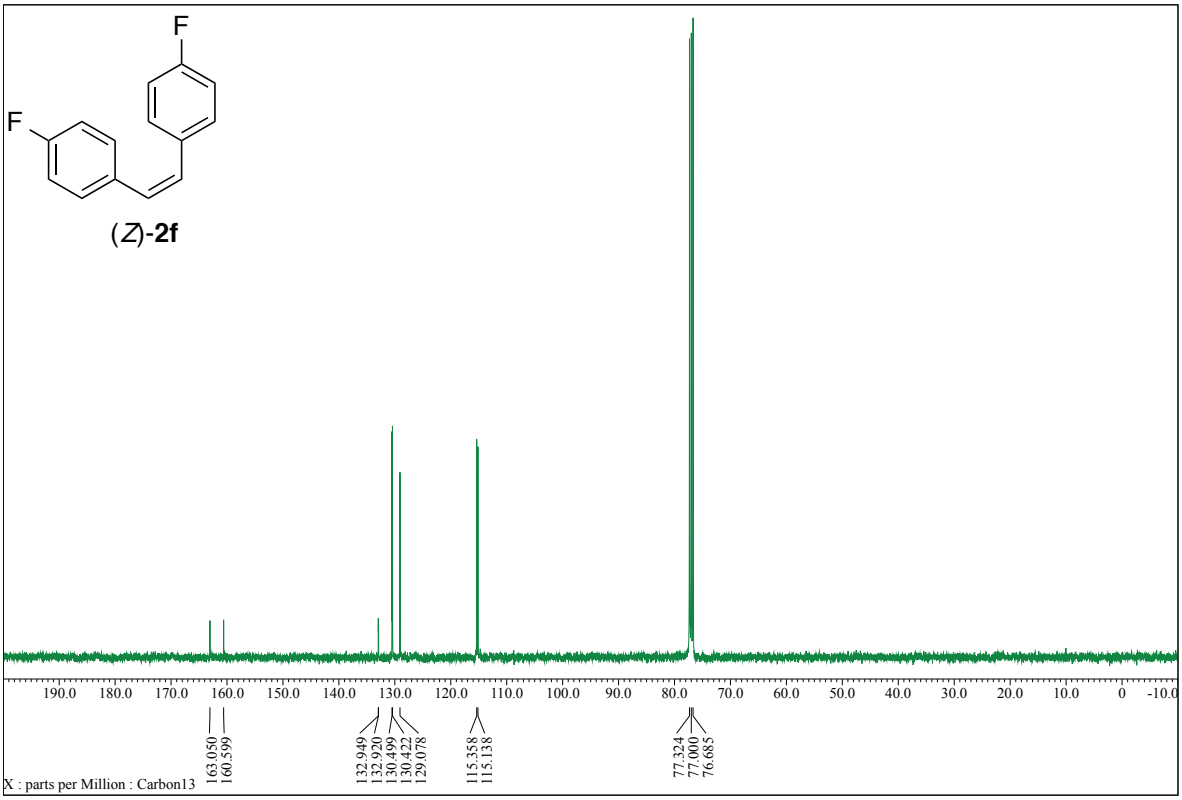

(Z)-2d
10.09
2.00
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
-1.0
7.530
7.512
7.381
7.363
7.344
7.331
7.308
7.279
7.260
7.249
7.245
7.242
7.239
7.204
7.167
7.117
6.600
2.925
X : parts per Million : Proton

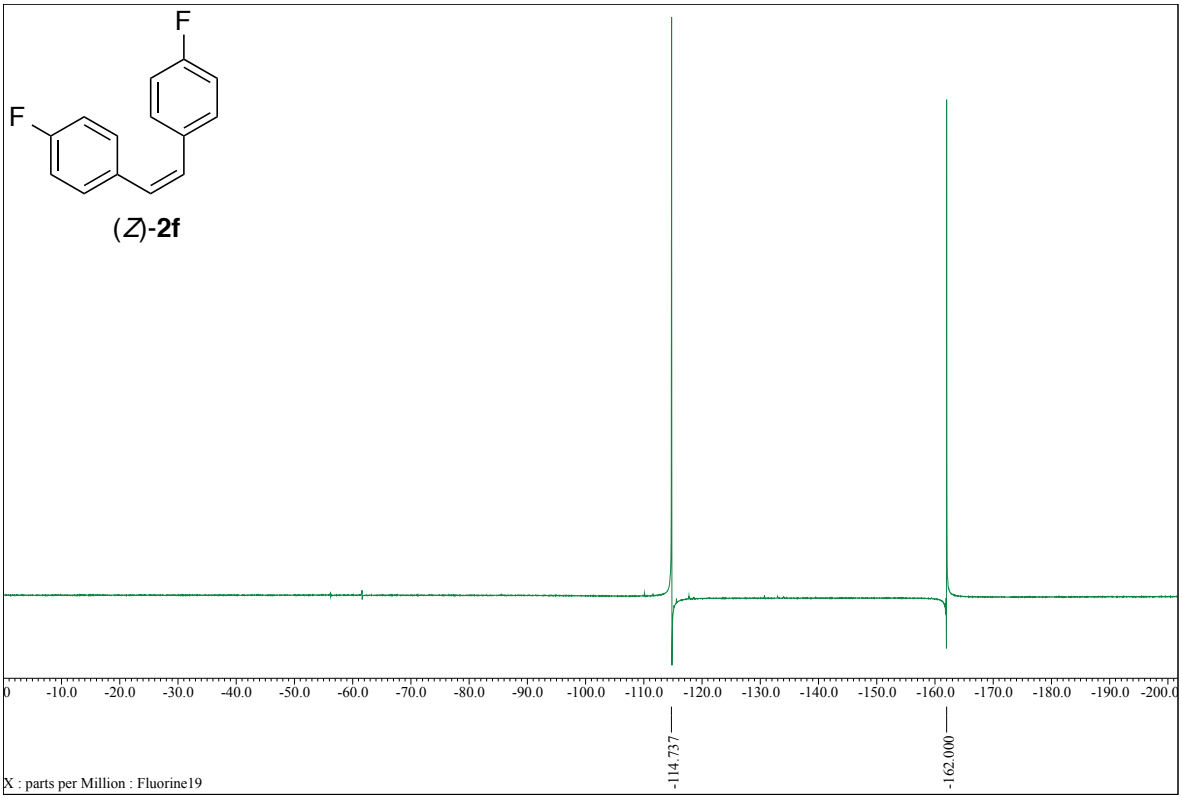

(Z)-2d
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
-10.0
137.278
137.201
130.212
128.840
128.649
128.172
127.590
127.057
126.484
77.324
77.000
76.685
X : parts per Million : Carbon13

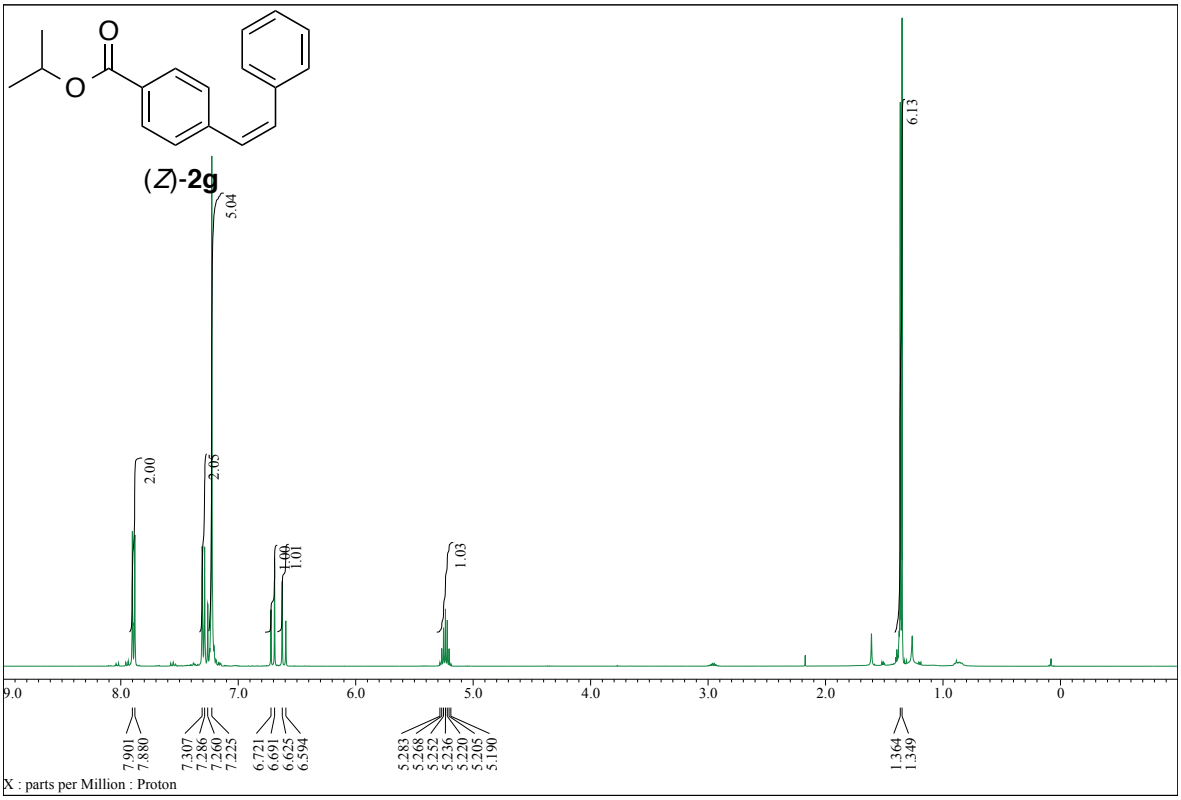

Me
Me
(Z)-2e
5.95
4.00
4.00
2.00
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
-1.0
7.260
7.179
7.159
7.144
7.137
7.119
7.116
7.100
7.052
7.032
6.566
6.521
2.869
2.366
2.360
2.333
2.323
X : parts per Million : Proton

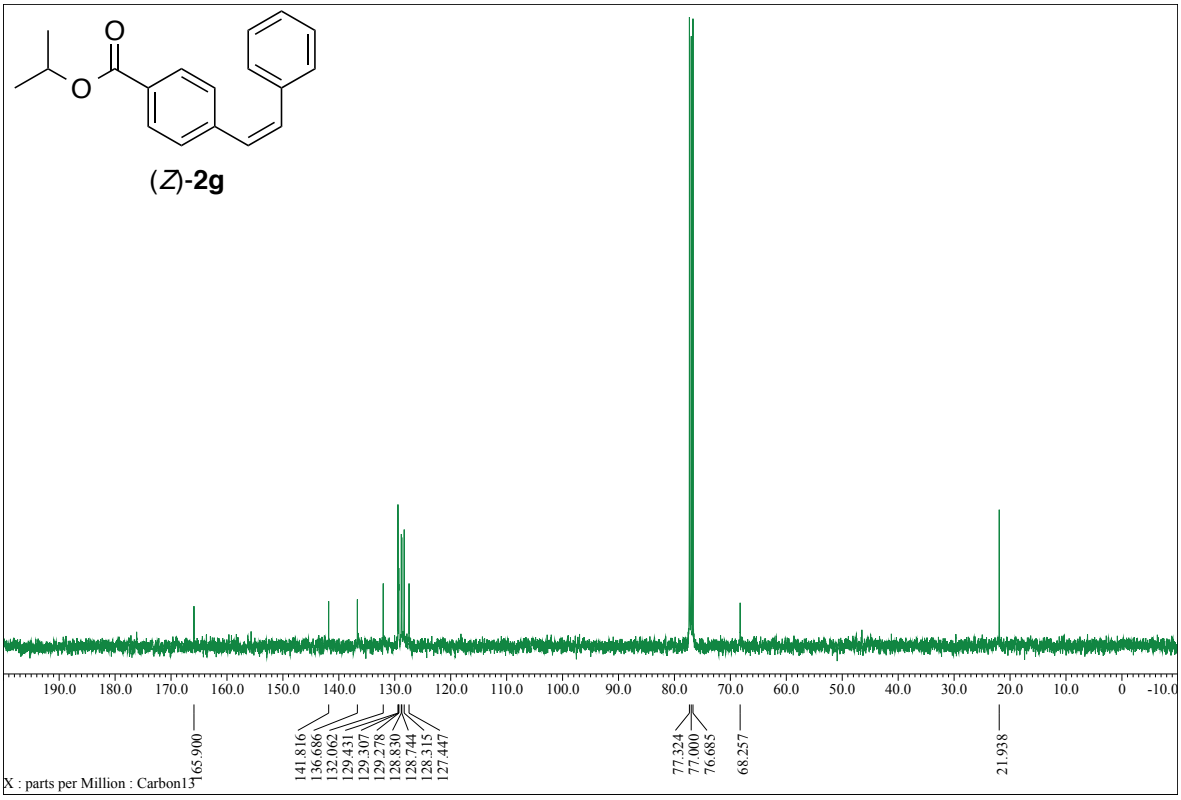

Me
Me
(Z)-2e
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
-10.0
136.696
134.465
129.478
129.345
128.983
128.859
128.725
128.315
128.277
126.284
125.846
77.315
77.000
76.685
21.213
X : parts per Million : Carbon13

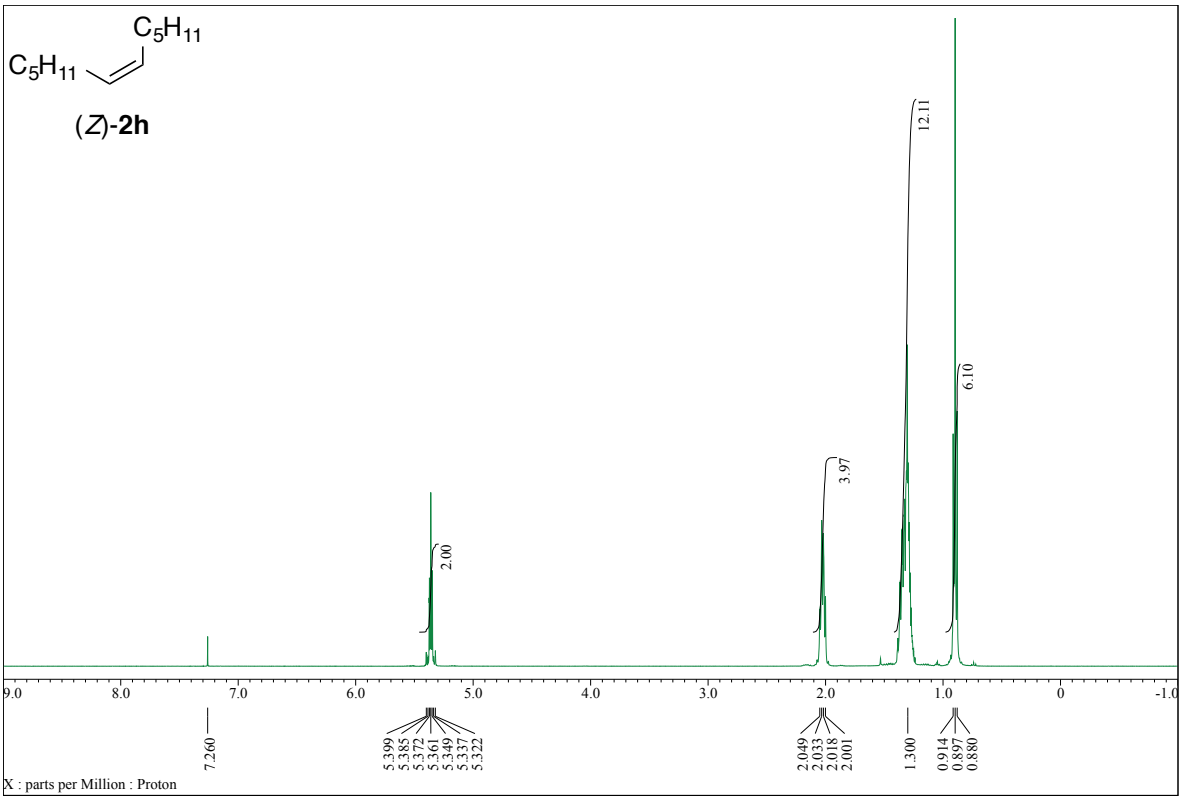

F
F
(Z)-2f
3.95
4.00
2.02
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
-1.0
7.260
7.198
7.192
7.184
7.176
7.168
7.163
6.942
6.937
6.926
6.920
6.915
6.904
6.898
6.537
1.543
X : parts per Million : Proton

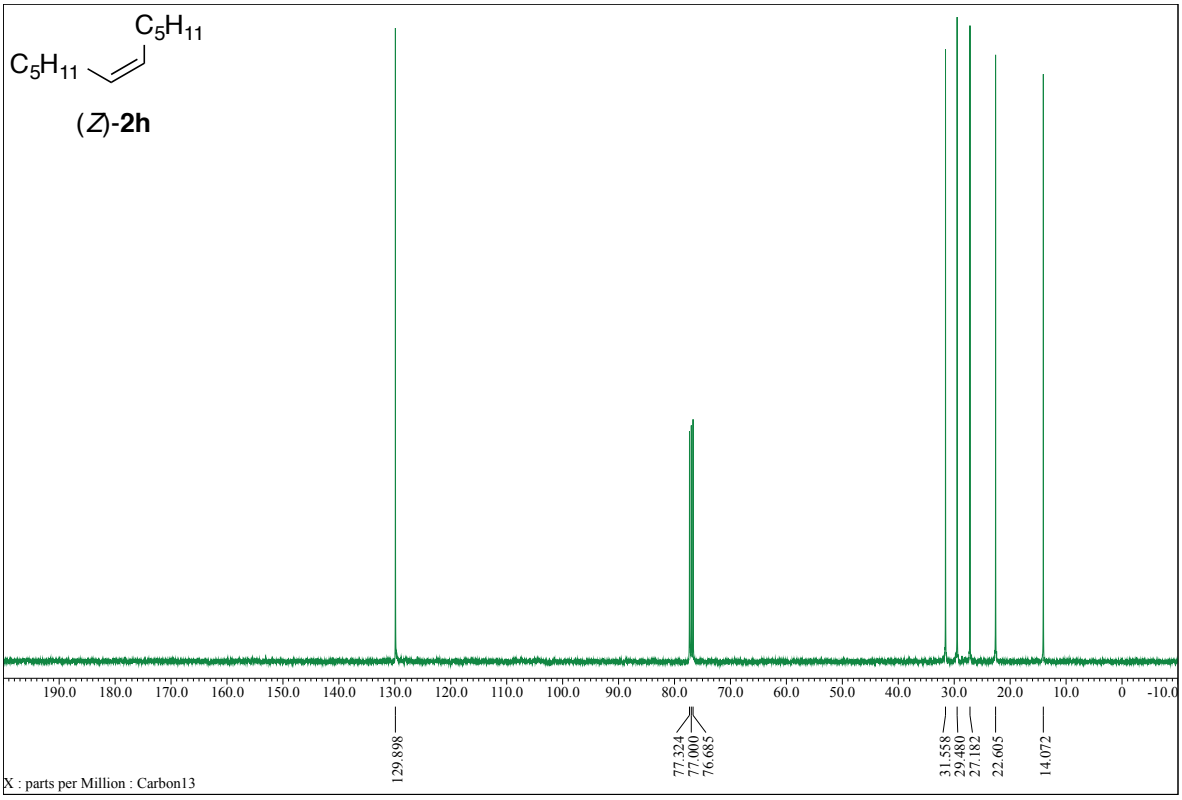

F
F
(Z)-2f
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
-10.0
163.050
160.599
132.949
132.920
130.499
130.422
129.078
115.358
115.138
77.324
77.000
76.685
X : parts per Million : Carbon13


F
F
(Z)-2f
-114.737
-162.000
X : parts per Million : Fluorine19


O
O
(Z)-2g
6.13
5.04
2.00
2.03
1.00
1.01
1.03
7.901
7.880
7.307
7.286
7.260
7.225
6.721
6.691
6.625
6.594
5.283
5.268
5.252
5.236
5.220
5.205
5.190
1.364
1.349
X : parts per Million : Proton


(Z)-2g
190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 -10.0
165.900
141.816
136.686
132.062
129.431
129.307
129.278
128.830
128.744
128.315
127.447
77.324
77.000
76.685
68.257
21.938
X : parts per Million : Carbon13


C5H11
C5H11
(Z)-2h
12.11
6.10
3.97
2.00
9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0 -1.0
7.260
5.399
5.385
5.372
5.361
5.349
5.337
5.322
2.049
2.033
2.018
2.001
1.300
0.914
0.897
0.880
X : parts per Million : Proton


C5H11
C5H11
(Z)-2h
190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 -10.0
129.898
77.324
77.000
76.685
31.558
29.480
27.182
22.605
14.072
X : parts per Million : Carbon13