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Catalytic Intermolecular Allyl–Allyl Cross-Couplings between Alcohols and Boronates

Agustín Jiménez-Aquino, Emmanuel Ferrer Flegeau, Uwe Schneider, and Shū Kobayashi*

Department of Chemistry, School of Science, The University of Tokyo, Hongo, Bunkyo-ku, Tokyo 113-0033, Japan

General and Additional Information

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1 General Experimental

Nuclear Magnetic Resonance (NMR) spectra were recorded on a JEOL ECX–600 spectrometer operating at 600 MHz for ^{1}H NMR, 150 MHz for ^{13}C NMR, and 565 MHz for ^{19}F NMR. Chemical shifts were reported downfield from tetramethyl silane (TMS). Infra Red (IR) spectra were measured using a JASCO FT / IR–610 spectrometer. High Resolution Mass Spectra (HRMS) were recorded using a BRUKER DALTONICS BioTOF II (ESI), a JEOL JMS-T100TD (DART), or a KRATOS ANALYTICAL AXIMA–CFR^{+A} (MALDI–TOF) spectrometer. Preparative thin-layer Chromatography (PTLC) was carried out using Wakogel B–5F from WAKO. Toluene and hexane were freshly distilled from sodium(0) / benzophenone; acetonitrile was distilled from calcium hydride. All solvents were then stored over activated molecular sieves (4 Å) in an argon box. The primary allyl alcohols **1a**,[1] **1c**,[2] and **1j**[3] are literature-known and were prepared accordingly. The secondary allyl alcohols **5a**,[4] **5b**,[5] **5d**,[6] **5e**,[7] **5f**,[8] **5g**,[9] **5h**,[10] **5i**,[11] **5k**,[12] **5l**,[13] **5m**,[14] and **5n**[15] are literature-known and were prepared accordingly. Allyl boronates **2a**[16] and **6**[17] were prepared by slightly modified procedures of reported methods. Tetrakis(triphenylphosphine)nickel(0) was purchased from ALDRICH and stored in an argon box at –30 °C.

2 General Procedure for Catalytic Intermolecular Allyl–Allyl Cross-Couplings

To a flame-dried 5 mL-microwave-type vial with magnetic stirring bar in an argon box were added tetrakis(triphenylphosphine)nickel(0) (0.01–0.02 mmol; 5–10 mol%), the indicated solvent (2 mL; 0.1 M with respect to allyl alcohol substrate; *cf. chapters 3.1 and 3.2*), and the corresponding primary or secondary allyl alcohol **1** *or* **5** (0.20 mmol, 1.0 equiv). After drop-wise addition of the corresponding allyl boronate **2a** *or* **6** (0.24 mmol; 1.2 equiv) *outside of the argon box*, the reaction mixture in the septum-sealed vial was stirred at the indicated temperature until complete conversion of the starting allyl alcohol [thin-layer chromatography (TLC), silica gel]. The crude reaction mixture was sequentially diluted with diethyl ether (6 mL), stirred at open air for 15 min, and filtered through a pad of silica gel. The filtrate was concentrated *in vacuo*, and the residue was purified by preparative thin-layer chromatography (PTLC; eluant: hexane / ethyl acetate) to afford the cross-coupled products **3a**–**n** and **4j** *or* **7** and **8**.

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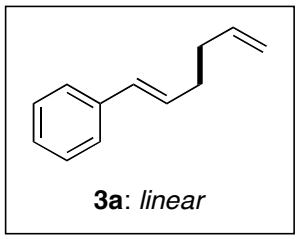
3 Analytical Data for Unknown Compounds

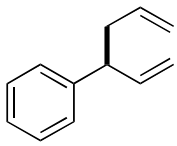
3.1 *Linear* 1,5-Dienes **3a–n** and *Branched* 1,5-Diene **4j**

Analytical data are provided for all unknown 1,5-dienes.

1-[(*E*)-Hexa-1,5-dienyl]benzene (3a)[3]



3a: *linear*



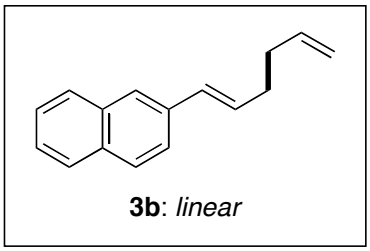
4a: *branched*
not observed

Prepared from the primary *or* secondary allyl alcohol **1a** *or* **5a** and allyl boronate **2a** according to the general procedure (*cf.* chapter 2); *catalyst loading:* 10 mol% *or* 5 mol%; *solvent:* toluene *or* acetonitrile; *temperature:* 25 °C; *time:* 12 h *or* 16 h. *The obtained analytical data of **3a** thus prepared fit accurately with the reported data.*[3]

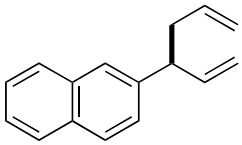
Colorless liquid.

Yield: 94% *or* 85% (ratio **3a**:**4a** = >99:1).

2-[(*E*)-Hexa-1,5-dienyl]naphthalene (3b)



3b: *linear*



4b: *branched*
not observed

Prepared from the primary allyl alcohol **1b** and allyl boronate **2a** according to the general procedure (*cf.* chapter 2); *catalyst loading:* 5 mol%; *solvent:* acetonitrile; *temperature:* 25 °C; *time:* 26 h.

Pale yellow liquid.

Yield: 75% (ratio **3b**:**4b** = >99:1).

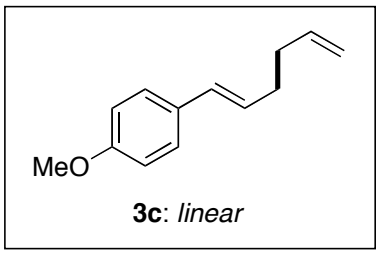
^{1}H NMR ($CDCl_3$, 600 MHz): δ = 2.28–2.41 (m, 4H), 5.04–5.13 (m, 2H), 5.89–5.95 (m, 1H), 6.38 (dt, *J* = 6.8, 15.6 Hz, 1H), 6.58 (d, *J* = 15.6 Hz, 1H), 7.41–7.47 (m, 2H); 7.59–7.60 (m, 1H), 7.69 (s, 1H); 7.77–7.81 (m, 3H) ppm.

^{13}C NMR ($CDCl_3$, 150 MHz): δ = 32.6, 33.6, 115.1, 123.6, 125.5, 126.2, 127.7, 127.9, 128.1, 130.4, 130.6, 132.8, 133.8, 135.3, 138.2 ppm.

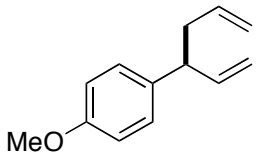
IR (film): ν = 3056, 2923, 1638, 1440, 963, 911, 858, 809, 744 cm^{-1}.

HRMS (ESI): calculated for $C_{16}H_{16}^+$ = $[M]^+$: *m*/*z* = 208.1252; *mass spectroscopic analyses (DART, ESI, MALDI) failed to give the desired molecular signal, resulting in fragmentation.*

1-[(*E*)-Hexa-1,5-dienyl]-4-methoxybenzene (3c)[3]



3c: *linear*



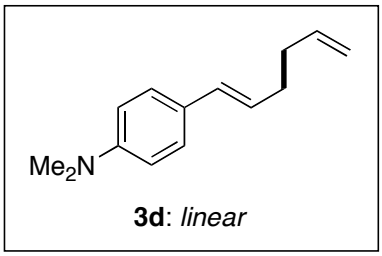
4c: *branched*
not observed

Prepared from the primary allyl alcohol **1c** and allyl boronate **2a** according to the general procedure (*cf.* chapter 2); *catalyst loading:* 10 mol%; *solvent:* toluene; *temperature:* 30 °C; *time:* 14 h. *The obtained analytical data of **3c** thus prepared fit accurately with the reported data.*[3]

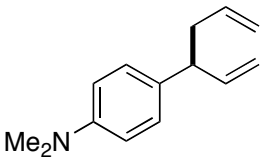
Colorless liquid.

Yield: 89% (ratio **3c**:**4c** = >99:1).

4-[(*E*)-Hexa-1,5-dienyl]-*N,N*-dimethylbenzenamine (3d)



3d: *linear*



4d: *branched*
not observed

Prepared from the secondary allyl alcohol **5d** and allyl boronate **2a** according to the general procedure (*cf.* chapter 2); *catalyst loading:* 5 mol%; *solvent:* acetonitrile; *temperature:* 25 °C; *time:* 26 h.

Orange-red liquid.

Yield: 86% (ratio **3d**:**4d** = >99:1).

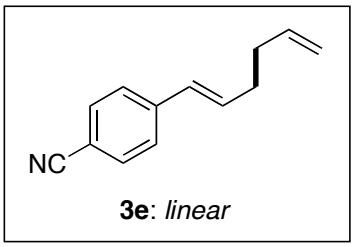
^{1}H NMR ($CDCl_3$, 600 MHz): δ = 2.26–2.38 (m, 4H), 2.98 (s, 6H), 5.04–5.14 (m, 2H), 5.90–5.97 (m, 1H), 6.08 (dt, *J* = 6.7, 15.8 Hz, 1H), 6.37–6.40 (m, 1H), 6.73 (d, *J* = 8.8 Hz, 2H), 7.29 (d, *J* = 8.8 Hz, 2H) ppm.

^{13}C NMR ($CDCl_3$, 150 MHz): δ = 32.7, 34.1, 40.8, 112.8, 114.9, 126.0, 126.7, 127.0, 130.1, 138.6, 149.9 ppm.

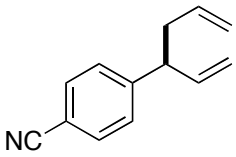
IR (film): ν = 2977, 2919, 2844, 1610, 1522, 1444, 1351, 1165, 963, 911, 802 cm^{-1}.

HRMS (ESI): calculated for $C_{14}H_{19}N^+$ = $[M]^+$: *m/z* = 201.1517; *mass spectroscopic analyses (DART, ESI, MALDI) failed to give the desired molecular signal, resulting in fragmentation.*

4-[(*E*)-Hexa-1,5-dienyl]benzonitrile (3e)[3]



3e: *linear*



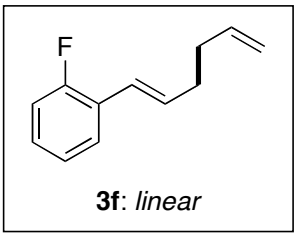
4e: *branched*
not observed

Prepared from the secondary allyl alcohol **5e** and allyl boronate **2a** according to the general procedure (*cf.* chapter 2); *catalyst loading:* 10 mol%; *solvent:* acetonitrile; *temperature:* 25 °C; *time:* 12 h. *The obtained analytical data of **3e** thus prepared fit accurately with the reported data.*[3]

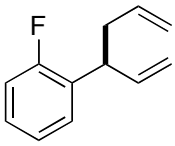
Colorless liquid.

Yield: 87% (ratio **3e**:**4e** = >99:1).

1-fluoro-[(*E*)-Hexa-1,5-dienyl]benzene (3f)



3f: *linear*



4f: *branched*
not observed

Prepared from the secondary allyl alcohol **5f** and allyl boronate **2a** according to the general procedure (*cf.* chapter 2); *catalyst loading:* 10 mol%; *solvent:* hexane; *temperature:* 40 °C; *time:* 19 h.

Colorless liquid.

Yield: 86% (ratio **3f**:**4f** = >99:1).

^{1}H NMR ($CDCl_3$, 600 MHz): δ = 2.24–2.35 (m, 4H), 5.00–5.09 (m, 2H), 5.92 (ddt, *J* = 6.5, 10.2, 16.8 Hz, 1H), 6.31 (dt, *J* = 6.9, 16.0 Hz, 1H), 6.55–6.58 (m, 1H), 6.99–7.18 (m, 3H), 7.42 (td, *J* = 1.8, 7.7 Hz, 1H) ppm.

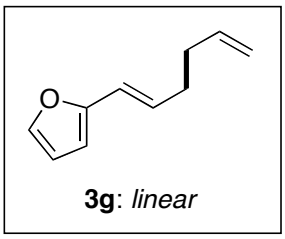
^{13}C NMR ($CDCl_3$, 150 MHz): δ = 32.9, 33.5, 115.1, 115.7 (d, *J* = 22.3 Hz), 122.6 (d, *J* = 3.6 Hz), 124.0 (d, *J* = 3.6 Hz), 125.5 (d, *J* = 12.4 Hz), 127.1 (d, *J* = 4.1 Hz), 128.1 (d, *J* = 8.3 Hz), 132.9 (d, *J* = 4.4 Hz), 138.1, 160.0 (d, *J* = 248.4 Hz) ppm.

^{19}F NMR ($CDCl_3$, 565 MHz): δ = –118.8 ppm.

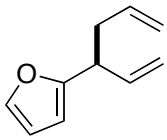
IR (film): ν = 3077, 3041, 3001, 2979, 2925, 2846, 1642, 1577, 1488, 1452, 1229, 1199, 970, 913, 753 cm^{-1}.

HRMS (ESI): calculated for $C_{12}H_{13}F^{+}$ = $[M]^{+}$: *m*/*z* = 176.1001; *mass spectroscopic analyses (DART, ESI, MALDI) failed to give the desired molecular signal, resulting in fragmentation.*

2-[(*E*)-Hexa-1,5-dienyl]furan (3g)



3g: *linear*



4g: *branched*
not observed

Prepared from the secondary allyl alcohol **5g** and allyl boronate **2a** according to the general procedure (*cf.* chapter 2); *catalyst loading:* 10 mol%; *solvent:* toluene; *temperature:* 25 °C; *time:* 24 h.

Pale yellow liquid.

Yield: 89% (ratio **3g**:**4g** = >99:1).

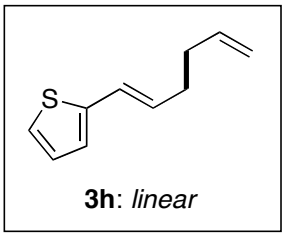
^{1}H NMR ($CDCl_3$, 600 MHz): δ = 2.20–2.29 (m, 4H), 4.98–5.07 (m, 2H), 5.85 (ddt, *J* = 6.5, 10.1, 16.8 Hz, 1H), 6.12–6.23 (m, 3H), 6.33–6.34 (m, 1H), 7.29 (d, *J* = 1.7 Hz, 1H) ppm.

^{13}C NMR ($CDCl_3$, 150 MHz): δ = 32.3, 33.5, 106.2, 111.2, 115.1, 119.0, 129.2, 138.1, 141.4, 153.3 ppm.

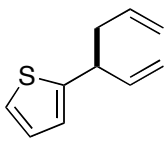
IR (film): ν = 3078, 2978, 2924, 2845, 1641, 1491, 1442, 1254, 1150, 1011, 960, 915, 792, 732 cm^{-1}.

HRMS (ESI): calculated for $C_{10}H_{12}O^+$ = $[M]^+$: *m*/*z* = 148.0888; *mass spectroscopic analyses (DART, ESI, MALDI) failed to give the desired molecular signal, resulting in fragmentation*.

2-[(*E*)-Hexa-1,5-dienyl]thiophene (3h)[3]



3h: *linear*



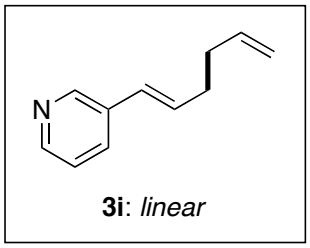
4h: *branched*
not observed

Prepared from the secondary allyl alcohol **5h** and allyl boronate **2a** according to the general procedure (*cf.* chapter 2); *catalyst loading:* 10 mol%; *solvent:* toluene; *temperature:* 25 °C; *time:* 14 h. *The obtained analytical data of **3h** thus prepared fit accurately with the reported data.*[3]

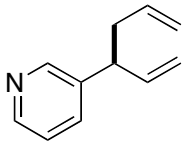
Pale yellow liquid.

Yield: 88% (ratio **3h**:**4h** = >99:1).

3-[(*E*)-Hexa-1,5-dienyl]pyridine (3i)[3]



3i: *linear*



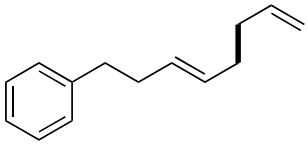
4i: *branched*
not observed

Prepared from the secondary allyl alcohol **5i** and allyl boronate **2a** according to the general procedure (*cf.* chapter 2); *catalyst loading:* 10 mol%; *solvent:* toluene; *temperature:* 25 °C; *time:* 24 h. *The obtained analytical data of **3i** thus prepared fit accurately with the reported data*.[3]

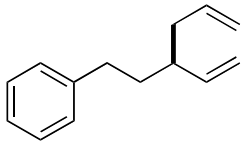
Pale yellow liquid.

Yield: 91% (ratio **3i**:**4i** = >99:1).

1-[(*E*)-Octa-3,7-dienyl]benzene (3j)[3]



3j: *linear*



4j: *branched*

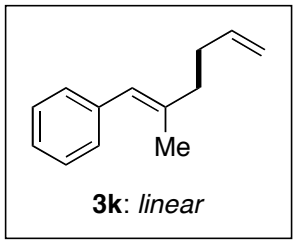
ratio **3j**:**4j** = 4:1

Prepared from the primary allyl alcohol **1j** and allyl boronate **2a** according to the general procedure (*cf.* chapter 2); *catalyst loading:* 10 mol%; *solvent:* hexane; *temperature:* 40 °C; *time:* 36 h. *The obtained analytical data of **3j** and **4j** thus prepared fit accurately with the reported data*.[3]

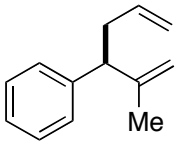
Colorless liquid.

Yield: 78% (ratio **3j**:**4j** = 4:1).

1-[(*E*)-2-Methylhexa-1,5-dienyl]benzene (3k)[3]



3k: *linear*



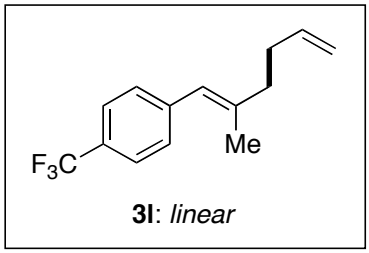
4k: *branched*
not observed

Prepared from the secondary allyl alcohol **5k** and allyl boronate **2a** according to the general procedure (*cf.* chapter 2); *catalyst loading:* 10 mol%; *solvent:* acetonitrile; *temperature:* 40 °C; *time:* 36 h. *The obtained analytical data of **3k** thus prepared fit accurately with the reported data.*[3]

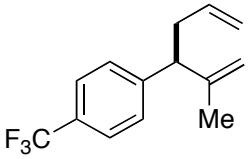
Yellow liquid.

Yield: 86% (ratio **3k**:**4k** = >99:1).

1-(Trifluoromethyl)-4-[(*E*)-2-methylhexa-1,5-dienyl]benzene (3l)[3]



3l: *linear*



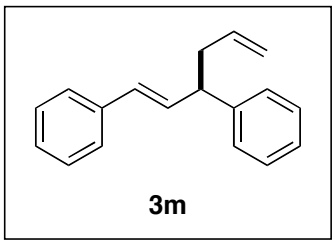
4l: *branched*
not observed

Prepared from the secondary allyl alcohol **5l** and allyl boronate **2a** according to the general procedure (*cf.* chapter 2); *catalyst loading:* 10 mol%; *solvent:* acetonitrile; *temperature:* 25 °C; *time:* 24 h. *The obtained analytical data of **3l** thus prepared fit accurately with the reported data.*[3]

Yellow liquid.

Yield: 87% (ratio **3l**:**4l** = >99:1).

(*E*)-1,3-Diphenylhexa-1,5-diene (3m)[3]



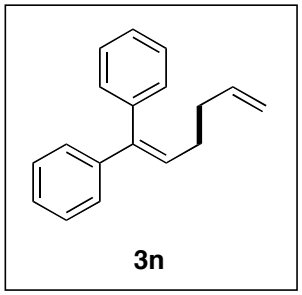
3m

Prepared from the secondary allyl alcohol **5m** and allyl boronate **2a** according to the general procedure (*cf.* chapter 2); *catalyst loading:* 10 mol%; *solvent:* toluene; *temperature:* 60 °C; *time:* 36 h. *The obtained analytical data of **7** and **8** thus prepared fit accurately with the reported data.*[3]

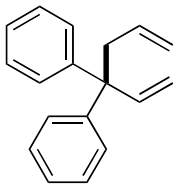
Pale yellow liquid.

Yield: 83%.

1,1-Diphenylhexa-1,5-diene (3n)



3n



4n: *branched*
not observed

Prepared from the secondary allyl alcohol **5n** and allyl boronate **2a** according to the general procedure (*cf.* chapter 2); *catalyst loading:* 10 mol%; *solvent:* MeCN; *temperature:* 60 °C; *time:* 36 h.

Pale yellow liquid.

Yield: 79% (ratio **3n**:**4n** = >99:1).

^{1}H NMR ($CDCl_3$, 600 MHz): δ = 2.23–2.29 (m, 4H), 5.00–5.07 (m, 2H), 5.80–5.86 (m, 1H), 6.12 (t, J = 7 Hz, 1H), 7.21–7.47 (m, 10H) ppm.

^{13}C NMR ($CDCl_3$, 150 MHz): δ = 29.3, 34.2, 115.1, 127.0, 127.1, 127.4, 128.2, 128.3, 129.3, 130.0, 138.2, 140.3, 142.1, 142.9 ppm.

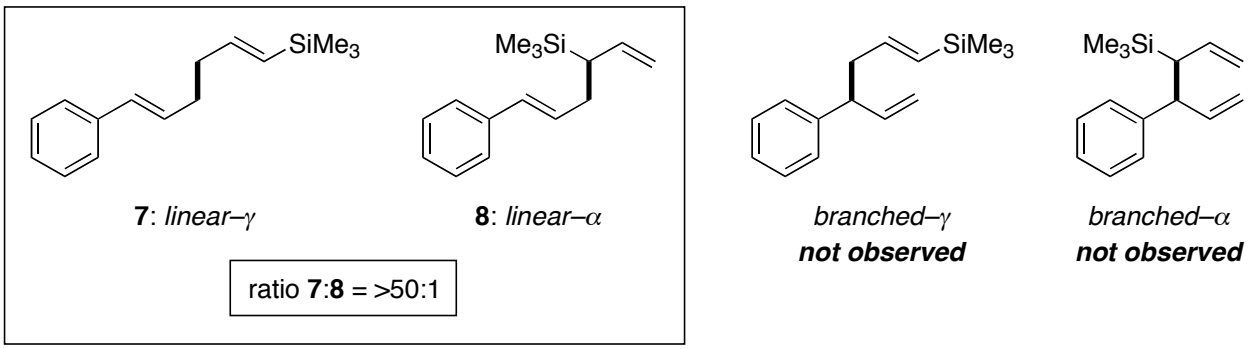
IR (film): ν = 3058, 3024, 2919, 1493, 1442, 994, 913, 762, 698 cm^{-1}.

HRMS (ESI): calculated for $C_{18}H_{18}^+$ = $[M]^+$: m/z = 234.1409; *mass spectroscopic analyses (DART, ESI, MALDI) failed to give the desired molecular signal, resulting in fragmentation.*

3.2 *Linear* Vinyl Silanes **7** and **8**

Trimethyl-[(1*E*,5*E*)-6-phenylhexa-1,5-dienyl]silane (7)[3]

Trimethyl-[(*E*)-6-phenylhexa-1,5-dien-3-yl]silane (8)[3]



Prepared from the primary allyl alcohol **1a** and α-silylallyl boronate **6** according to the general procedure (*cf.* chapter 2); *catalyst loading:* 10 mol%; *solvent:* hexane; *temperature:* 30 °C; *time:* 24 h. *The obtained analytical data of **7** and **8** thus prepared fit accurately with the reported data.*[3]

Pale yellow liquid.

Yield: 88% (ratio **7**:**8** = >50:1).

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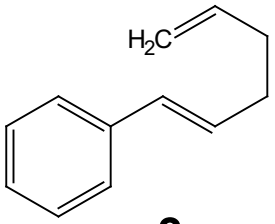

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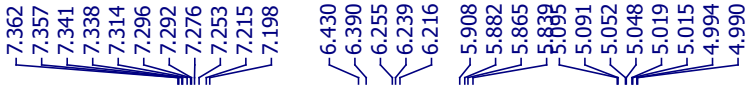

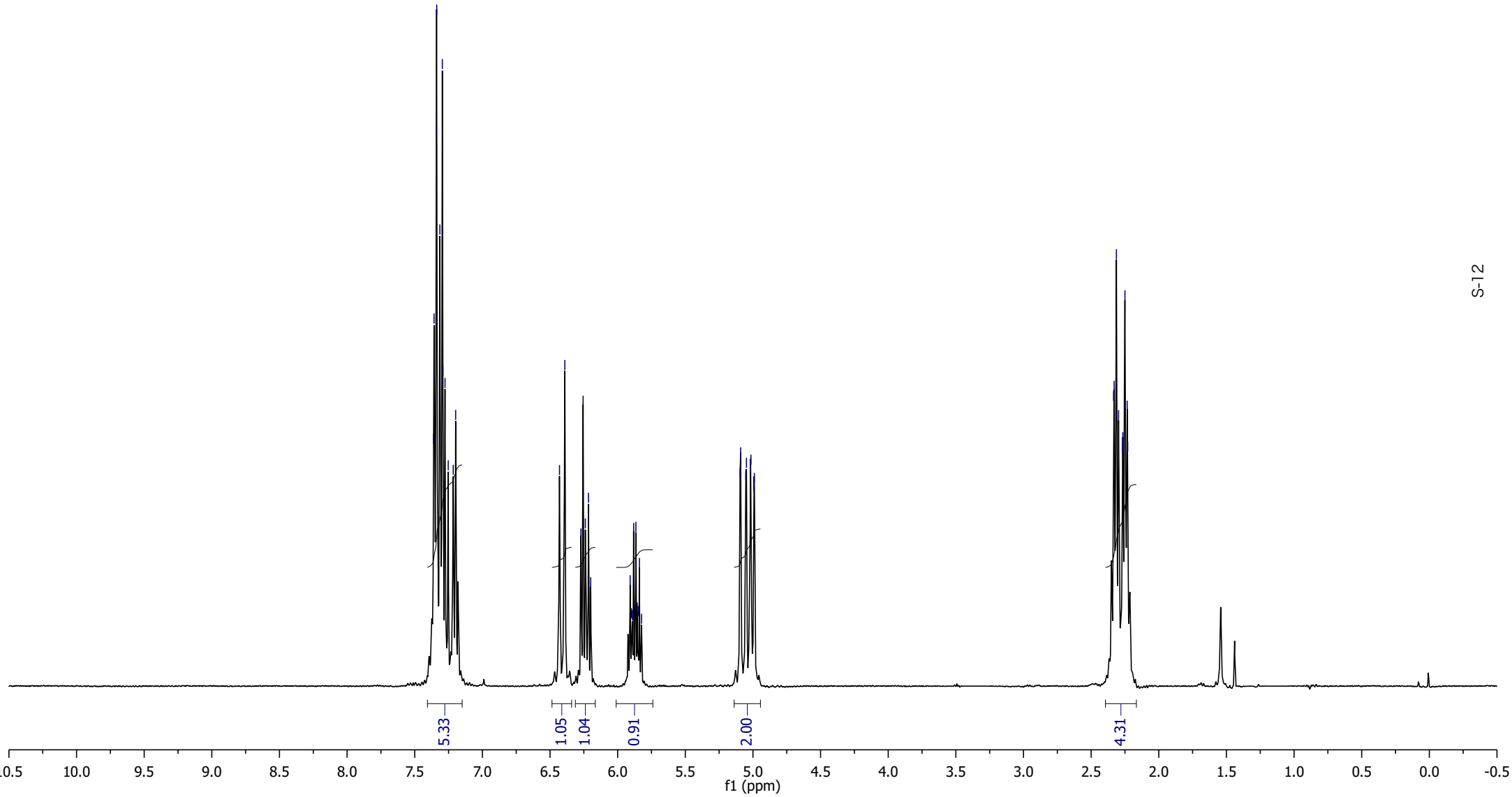

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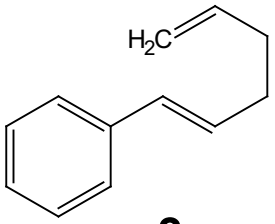

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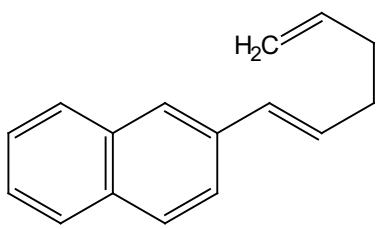

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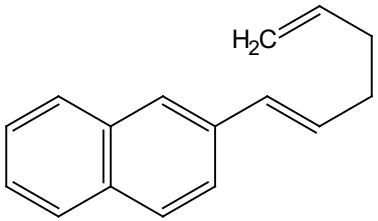

3b

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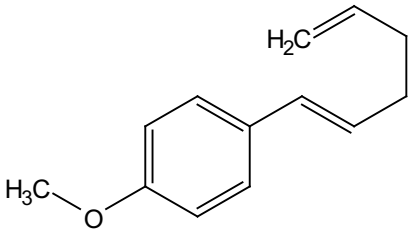

3c

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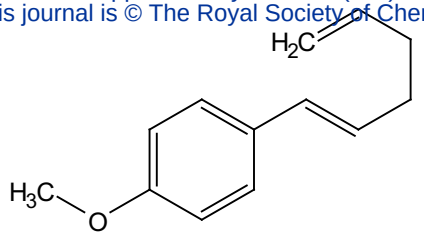

158.797
138.328
130.707
129.624
128.044
127.134
114.927
114.005
55.368
33.810
32.520

3c

200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

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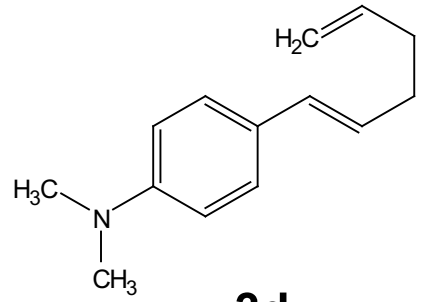

3d

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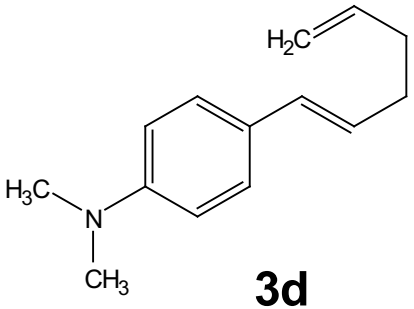

3d

149.875
138.567
130.099
126.972
126.689
126.028
114.856
112.778
40.775
34.102
32.700

f1 (ppm)

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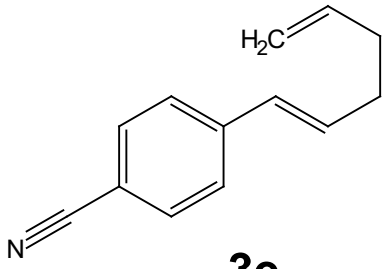

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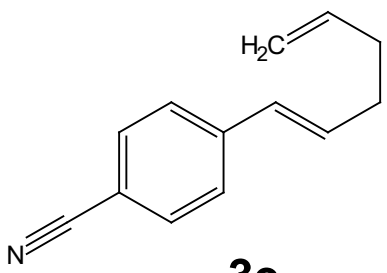

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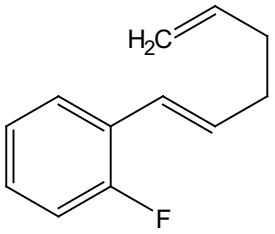

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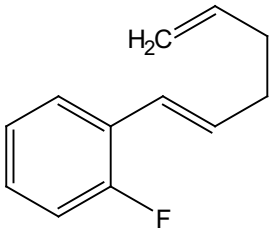

3f

160.866
159.220
138.064
132.906
128.180
127.132
127.105
124.081
124.057
122.670
115.770
115.622
115.114
33.527
32.885

S-23

200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

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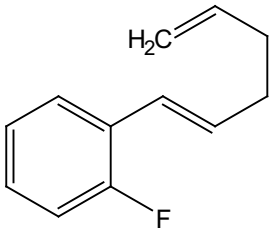

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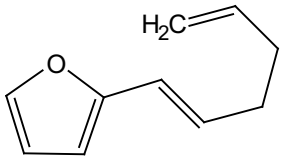

3g

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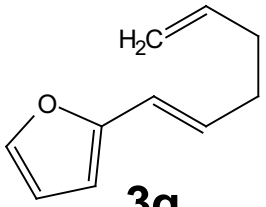

3g

153.265
141.361
138.065
129.201
118.991
115.055
111.157
106.213
33.461
32.270

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f1 (ppm)

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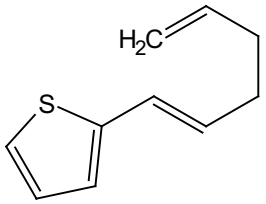

3h

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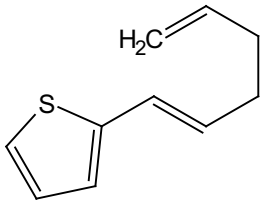

3h

143.059
138.036
130.175
127.296
124.418
123.555
123.267
115.109
33.484
32.330

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f1 (ppm)

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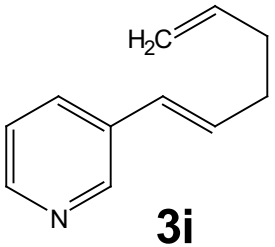

3i

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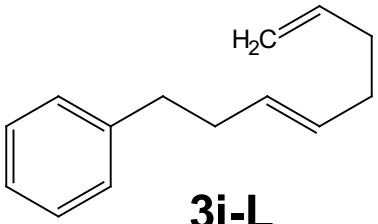
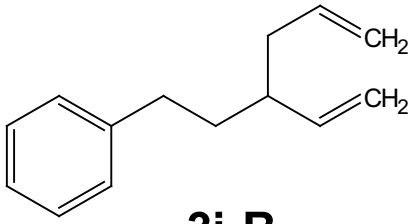


3j-L



3j-B

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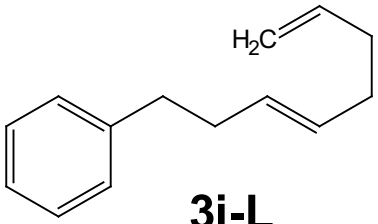
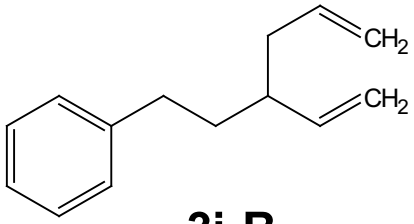


3j-L



3j-B

142.767
142.359
142.216
138.544
136.883
130.299
129.981
128.572
128.385
128.323
115.937
114.959
114.606
43.355
39.588
36.200
36.057
34.494
33.846
33.498
32.071

S-31

190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

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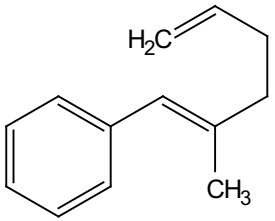

3k

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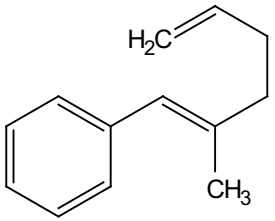

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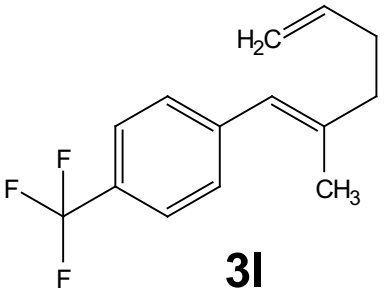

3l

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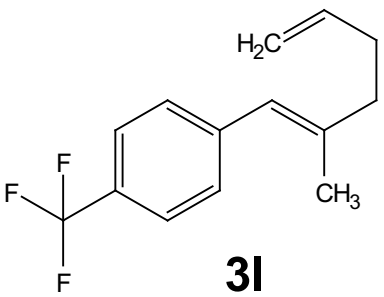
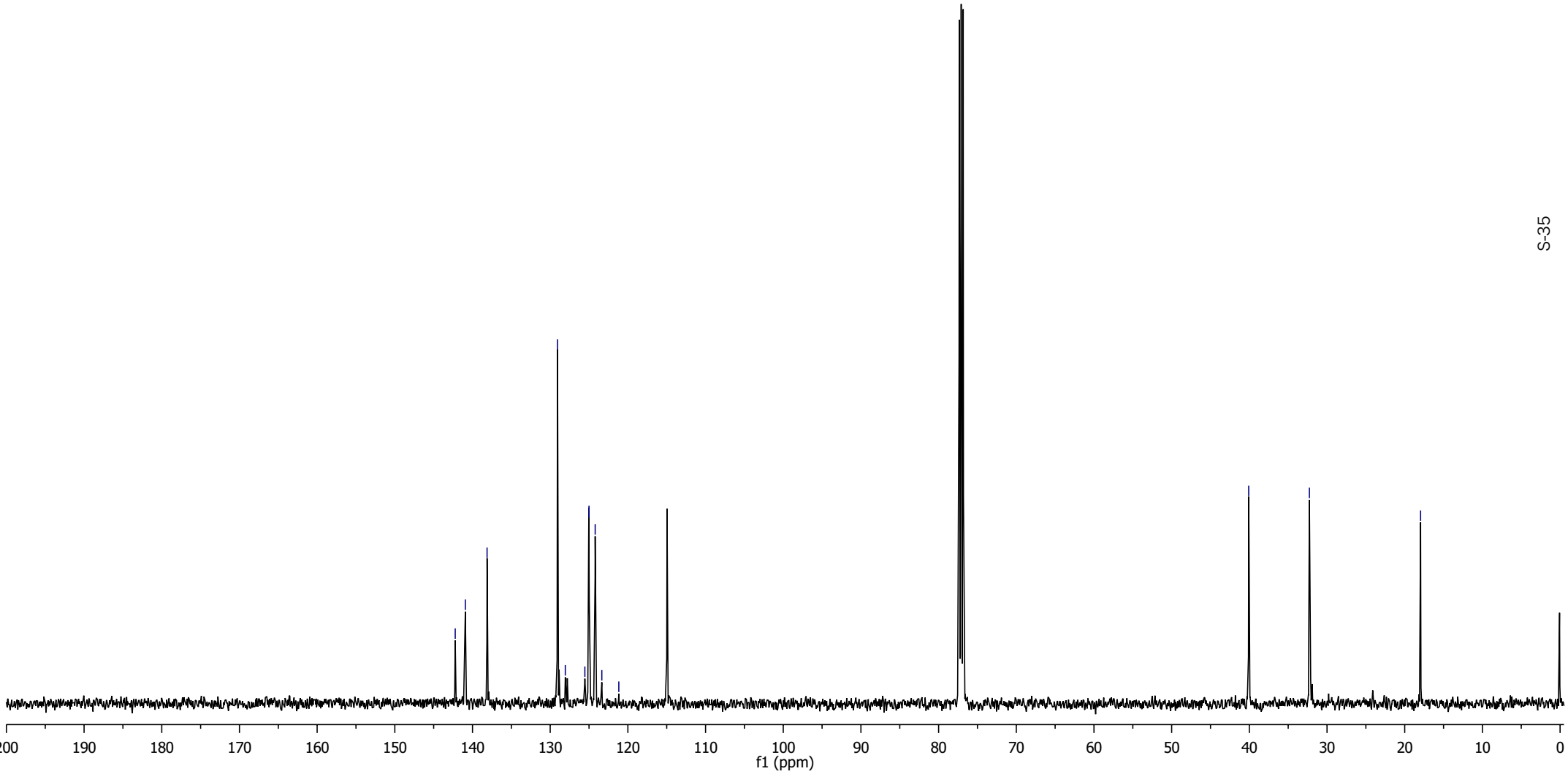


3l

142.226
140.923
138.126
129.062
128.055
125.536
125.033
125.002
124.208
123.353
121.165
40.080
32.271
17.955



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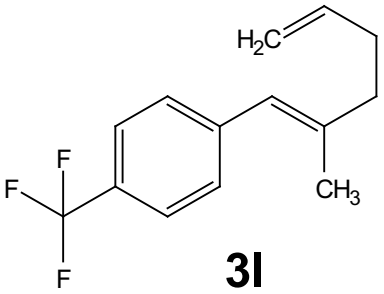

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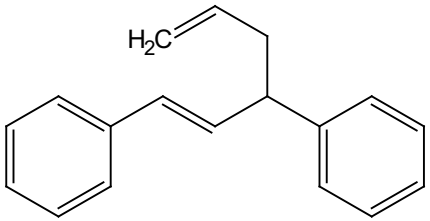

3m

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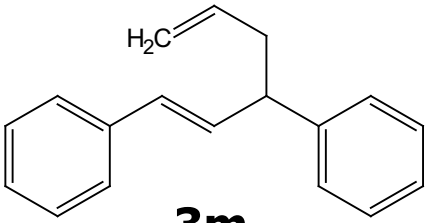

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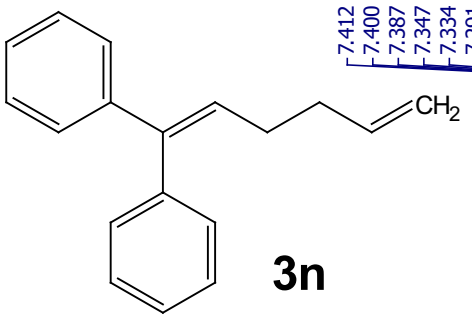

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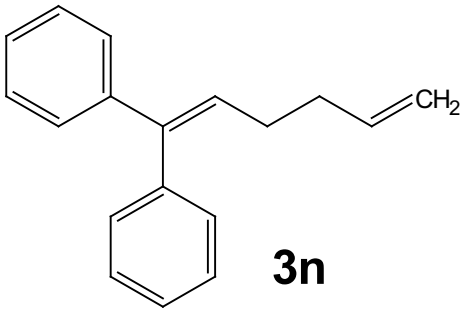
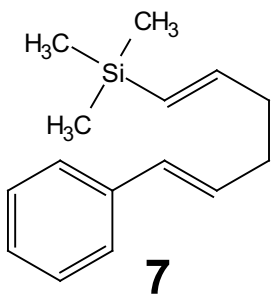


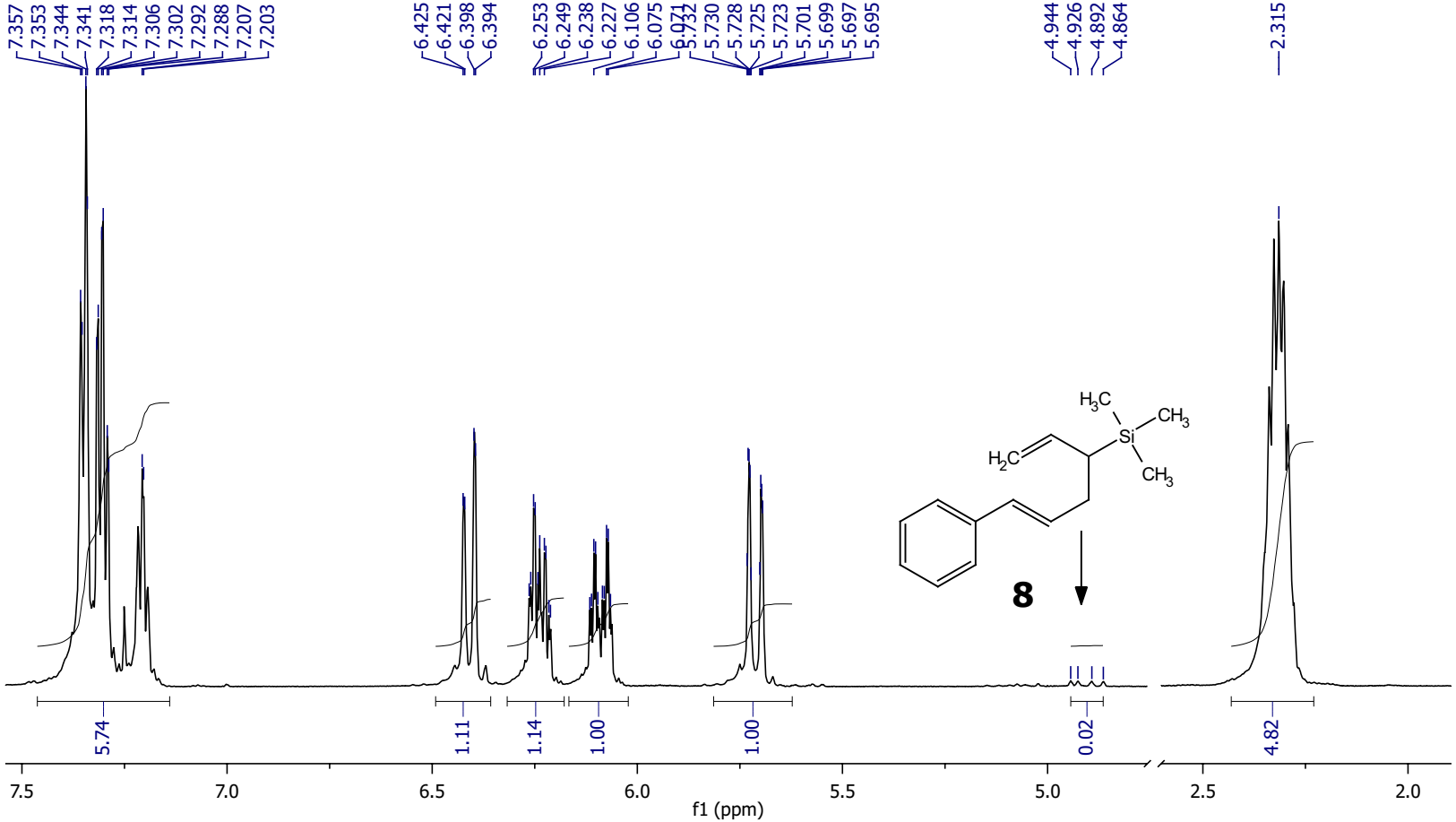
3n

142.910
142.081
140.315
138.236
130.043
129.311
128.288
128.202
127.386
127.036
126.983
115.073
34.176
29.265

200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

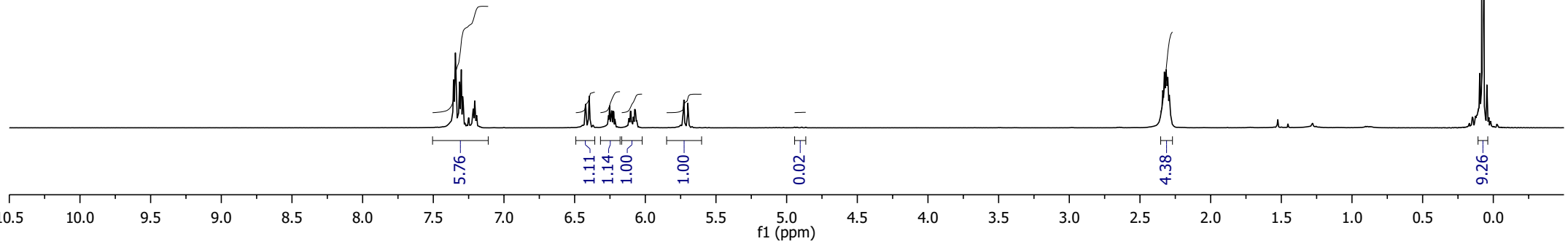

H3C
CH3
Si
H3C

7


7.357
7.353
7.344
7.341
7.318
7.314
7.306
7.302
7.292
7.288
7.207
7.203
6.425
6.421
6.398
6.394
6.253
6.249
6.238
6.227
6.106
6.075
6.071
5.732
5.730
5.728
5.725
5.723
5.701
5.699
5.697
5.695
4.944
4.926
4.892
4.864
2.315
0.076
0.072
5.74
1.11
1.14
1.00
1.00
0.02
4.82
7.5
7.0
6.5
6.0
5.5
5.0
2.5
2.0
f1 (ppm)

H3C
CH3
Si
H2C
CH3

8


5.76
1.11
1.14
1.00
1.00
0.02
4.38
9.26
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

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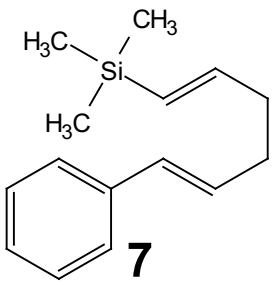

7