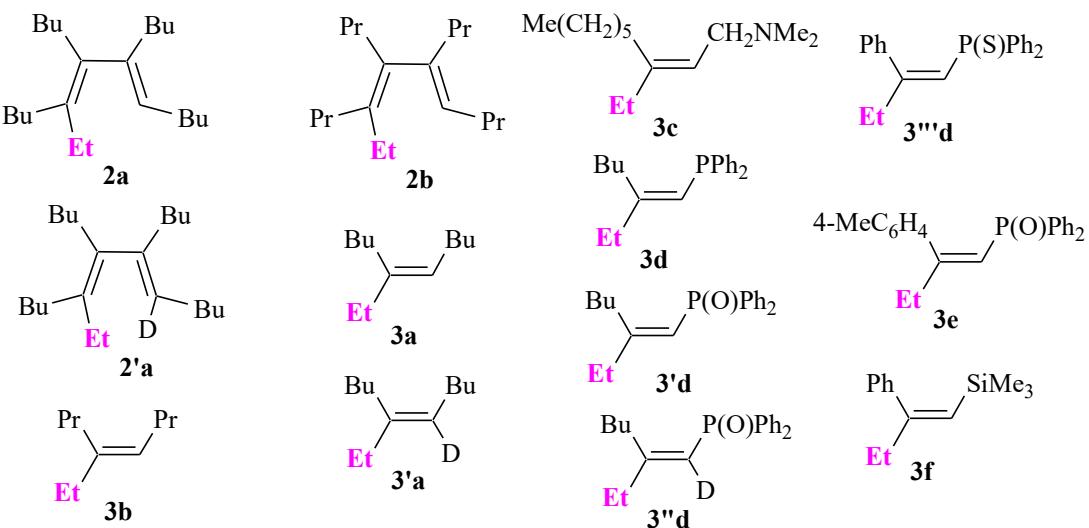


## Tantalum pentachloride-catalyzed reactions of alkynes with Et<sub>3</sub>Al

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**General.** Chromatographic analysis was performed on a Shimadzu GC-9A instrument using a 2000×2 mm column, the SE-30 (5 %) stationary phase on Chromaton N-AW-HMDS (0.125–0.160 mm), helium carrier gas (30 ml/min), temperature programming from 50 to 300 °C at a 8 °C/min rate. The <sup>1</sup>H, <sup>13</sup>C, and <sup>31</sup>P NMR spectra were measured in CDCl<sub>3</sub> on a Bruker Avance-500 spectrometer. The <sup>1</sup>H NMR spectra were recorded at 500 MHz and <sup>13</sup>C-{1H} NMR spectra at 125 MHz in CDCl<sub>3</sub>. Elemental analysis was performed using a Carlo-Erba CHN 1106 elemental analyser. Mass spectra were obtained on a Finnigan 4021 instrument. TLC was performed on Silufol UV-254 plates. For column chromatography, Acros silica gel (0.060–0.200 mm) was used. Reactions with organometallic compounds were performed in a dry argon atmosphere. Toluene was dried over sodium and distilled immediately prior to use. Commercially available 5-decyne, 4-octyne, TaCl<sub>5</sub>, Et<sub>3</sub>Al (93%) were used. *N,N*-Dimethylnon-2-yn-1-amine **1c** were prepared by aminomethylation of 1-octyne with *N,N,N',N'*-tetramethylmethanediamine [S1]. 1-Alkynylphosphines **1d,e** were prepared by direct phosphination of terminal alkynes with chlorophosphanes catalyzed by Ni(acac)<sub>2</sub> [S2].

**Experimental Procedures.** A 50 ml glass reactor equipped with a magnetic stirrer under a dry argon atmosphere at 0 °C, was charged under stirring with toluene (10 ml), TaCl<sub>5</sub> catalyst (143 mg, 0.2 mmol), alkyne **1** (2 mmol) and Et<sub>3</sub>Al (1.2 ml, 8 mmol). The temperature was raised to 120 °C and the mixture was boiled for an additional 18 h. The mixture was cooled under an argon stream to 0 °C. After the addition of Et<sub>2</sub>O (10 ml), the mixture was quenched with a 10% aqueous solution of KOH (10 ml), the organic layer separated, and the aqueous layer extracted with Et<sub>2</sub>O (3 x 15 ml). The combined organics were dried over MgSO<sub>4</sub>. The final products were isolated by column chromatography (**3d-f,3''d**) or by distillation through a micro column (**2a,b,2'a,3a-c,3'a**).



*(5Z,7E)-6,7-Dibutyl-5-ethyldodeca-5,7-diene (2a)*

According to the general procedure, compound **2a** was isolated by distillation through a microcolumn at 1.3 Torr to afford **2a** (356 mg, 58%) as a colorless oil. b.p. 171-173 °C (1.3 Torr).  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  0.89 – 0.96 (m, 15H), 1.26 – 1.37 (m, 16H), 1.99 – 2.08 (m, 10H), 4.99 (t,  $J$  = 7.2 Hz, 1H);  $^{13}\text{C}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  14.07 (3C), 14.14 (2C), 22.44, 22.74, 23.09, 23.17, 25.69, 27.43, 29.42, 29.54, 29.85, 30.37, 30.98, 31.28, 32.32, 127.33, 135.70, 137.66, 139.92. MS (EI): m/z, % = 307 (12) [ $\text{M}^+$ ], 277 (55), 249 (100), 235 (36), 207 (29), 137 (25), 109 (29), 95 (34), 69 (32), 55 (44), 41 (45). Anal. calcd for  $\text{C}_{22}\text{H}_{42}$ , (%): C, 86.19; H, 13.81. Found, %: C, 86.09; H, 13.52.

*(4Z,6E)-4-Ethyl-5,6-dipropyldeca-4,6-diene (2b)*

According to the general procedure, compound **2b** was isolated by distillation through a microcolumn at 1 Torr to afford **2b** (305 mg, 61%) as a colorless oil. b.p. 124-126 °C (1 Torr).  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  0.85 – 0.95 (m, 15H), 1.28 – 1.43 (m, 8H), 1.99 – 2.09 (m, 10H), 5.01 (t,  $J$  = 7.2 Hz, 1H);  $^{13}\text{C}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  13.91, 14.06, 14.09, 14.37, 14.66, 21.39, 21.78, 22.09, 23.24, 25.64, 29.85, 31.88, 32.02, 32.23, 127.26, 135.74, 137.69, 140.04. MS (EI): m/z, % = 250 (34) [ $\text{M}^+$ ], 221 (60), 207 (100), 193 (26), 165 (25), 151 (19), 123 (39), 109 (43), 95 (36), 55 (42), 41 (42). Anal. calcd for  $\text{C}_{18}\text{H}_{34}$ , (%): C, 86.32; H, 13.68. Found, %: C, 86.01; H, 13.43.

*(5Z,7E)-6,7-Dibutyl-8-deutero-5-ethyldodeca-5,7-diene (2'a)*

According to the general procedure compound **2'a** was isolated by distillation through a microcolumn at 1 Torr to afford **2'a** (351 mg, 57%) as a colorless oil. b.p. 170-172 °C (1 mmHg).  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  0.88 – 0.95 (m, 15H), 1.26 – 1.36 (m, 16H), 1.99 – 2.07 (m, 10H);  $^{13}\text{C}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  14.07 (3C), 14.14 (2C), 22.44, 22.73, 23.09, 23.17, 25.69, 27.33, 29.38, 29.54, 29.85, 30.36, 30.98, 31.28, 32.30, 126.77 – 127.26 (m, 1C), 135.69,

137.63, 139.81. MS (EI): m/z, % = 308 (12) [M<sup>+</sup>], 307 (50), 278 (51), 250 (100), 235 (27), 208 (25), 194 (20), 180 (11), 152 (13), 138 (17), 110 (21), 55 (44), 41 (50). Anal. calcd for C<sub>22</sub>H<sub>41</sub>D, (%): C, 85.91. Found, %: C, 86.12.

*(Z)-5-Ethyldec-5-ene (3a)*

According to the general procedure, compound **3a** was isolated by distillation through a microcolumn at 9.7 Torr to afford **3a** (71 mg, 21%) as a colorless oil. b.p. 84-86 °C (9.7 Torr). MS (EI): m/z, % = 168 (18) [M<sup>+</sup>], 139 (2), 111 (10), 97 (25), 69 (100), 55 (68), 41 (50). The spectral properties (<sup>1</sup>H NMR, <sup>13</sup>C NMR) were in good agreement with those that were reported in the literature [S3].

*(Z)-4-Ethyloct-4-ene (3b)*

According to the general procedure, compound **3b** was isolated by distillation through a microcolumn at 25 Torr to afford **3b** (50 mg, 18%) as a colorless oil. b.p. 71-73 °C (25 Torr). MS (EI): m/z, % = 140 (21) [M<sup>+</sup>], 111 (14), 97 (18), 69 (100), 55 (65), 41 (37). Anal. calcd for C<sub>10</sub>H<sub>20</sub>, (%): C, 85.63; H, 14.37. Found, %: C, 85.71; H, 14.17.

*(Z)-6-Deuterio-5-ethyldec-5-ene (3'a)*

According to the general procedure, compound **3'a** was isolated by distillation through a microcolumn at 10 Torr to afford **3'a** (74 mg, 22%) as a colorless oil. b.p. 86-88 °C (10 Torr). <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 0.90 – 0.94 (m, 6H), 0.99 (t, *J* = 7.4 Hz, 3H), 1.32 – 1.36 (m, 8H), 1.98 – 2.04 (m, 6 H); <sup>13</sup>C NMR (500 MHz, CDCl<sub>3</sub>) δ 12.96, 14.09 (2C), 22.46, 22.89, 27.27, 29.50, 29.91, 30.80, 32.42, 122.89 – 123.25 (1C), 140.97. MS (EI): m/z, % = 169 (21) [M<sup>+</sup>], 140 (3), 112 (14), 98 (27), 70 (100), 55 (57), 41 (47). Anal. calcd for C<sub>12</sub>H<sub>23</sub>D, (%): C, 85.12. Found, %: C, 85.31.

*(Z)-3-Ethyl-N,N-dimethylnon-2-en-1-amine (3c)*

According to the general procedure, compound **3c** was isolated by distillation through a microcolumn at 2 Torr to afford **3c** (347 mg, 88%) as a colorless oil. b.p. 88-90 °C (2 Torr). <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 0.87 (t, *J* = 6.2 Hz, 3H), 0.99 (t, *J* = 7.6 Hz, 3H), 1.25 – 1.32 (m, 8H), 1.95-2.10 (m, 2H), 2.10-2.30 (m, 2H), 2.19 (s, 6H), 2.87 (d, *J* = 6.7 Hz, 2H), 5.19 (t, *J* = 6.4 Hz, 1H); <sup>13</sup>C NMR (500 MHz, CDCl<sub>3</sub>) δ 12.69, 14.05, 22.62, 28.44, 29.44, 29.54, 30.56, 31.76, 45.23 (2C), 56.83, 120.51, 144.29. MS (EI): m/z, % = 197 (32) [M<sup>+</sup>], 182 (17), 168 (20), 152 (22), 123 (55), 112 (49), 95 (82), 82 (93), 67 (74), 58 (88), 46 (100). Anal. calcd for C<sub>13</sub>H<sub>27</sub>N, (%): C, 79.11; H, 13.79; N, 7.10. Found, %: C, 79.30; H, 13.66; N, 7.17.

*(Z)-(2-Ethylhex-1-en-1-yl)diphenylphosphane (3d)*

According to the general procedure, crude product **3d** was placed into NMR tube without purification by column chromatography. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 0.98 (t, *J* = 7.3 Hz, 3H),

1.19 (t,  $J = 7.3$  Hz, 3H), 1.39 – 1.44 (m, 2H), 1.49 – 1.55 (m, 2H), 2.31 – 2.36 (m, 2H), 2.65 (t,  $J = 7.7$ , 2H), 6.03 (br. s, 1H), 7.33 – 7.39 (m, 6H), 7.50 (t,  $J = 7.0$  Hz, 4H);  $^{13}\text{C}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  12.74, 14.14, 22.82, 31.04, 31.11 (d,  $J = 6.8$  Hz, 1C), 33.71 (d,  $J = 23.7$  Hz, 1C), 120.83 (d,  $J = 5.3$  Hz, 1C), 128.08 (2C), 128.39 (d,  $J = 6.2$  Hz, 4C), 132.61 (d,  $J = 18.7$  Hz, 4C), 140.62 (d,  $J = 9.5$  Hz, 2C), 161.54 (d,  $J = 24.3$  Hz, 1C).  $^{31}\text{P}$  NMR ( $\delta$ , ppm): 30.31.

*(Z)-(1-Deuterio-2-ethylhex-1-en-1-yl)diphenylphosphine oxide (3''d)*

A 50 ml glass reactor equipped with a magnetic stirrer under a dry argon atmosphere at 0 °C, was charged under stirring with toluene (10 ml),  $\text{TaCl}_5$  catalyst (143 mg, 0.2 mmol), (hex-1-yn-1-yl)(diphenyl)phosphane **1d** (450 mg, 2 mmol) and  $\text{Et}_3\text{Al}$  (1.2 ml, 8 mmol). The temperature was raised to 120 °C, and the mixture was boiled for 18 h. The mixture was cooled under an argon stream to 0 °C. After the addition of  $\text{Et}_2\text{O}$  (10 ml),  $\text{D}_2\text{O}$  (3 ml) was added dropwise while cooling the reactor flask in an ice bath, the organic layer separated, and the aqueous layer extracted with  $\text{Et}_2\text{O}$  (3 x 15 ml). The combined organics were dried over  $\text{MgSO}_4$ . The reaction mixture was filtered through a filter paper and concentrated *in vacuo*. A 30% hydrogen peroxide solution (0.35 ml, 3 mmol) was slowly added dropwise with vigorous stirring to a solution of the crude residue in chloroform (5 ml). The reaction mixture was stirred for 8 h and washed with water (3 x 5 ml), the organic layer was dried over  $\text{MgSO}_4$ . Evaporation of solvent and purification of the residue by column chromatography (hexane : ethyl acetate : methanol = 5 : 2 : 1) gave **3''d** (495 mg, 79%) as yellow oil.  $R_f$  0.30.  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  0.69 (t,  $J = 7.4$  Hz, 3H), 1.04 (t,  $J = 7.4$  Hz, 3H), 1.07 – 1.12 (m, 2H), 1.21 – 1.27 (m, 2H), 2.19 – 2.23 (q,  $J = 7.4$  Hz,  $J = 14.8$  Hz, 2H), 2.47 (t,  $J = 7.5$  Hz, 2H), 7.35 – 7.40 (m, 6H), 7.69 – 7.73 (m, 4H);  $^{13}\text{C}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  12.10, 13.80, 22.74, 30.29, 31.14 (d,  $J = 16.3$  Hz, 1C), 33.89 (d,  $J = 7.6$  Hz, 1C), 128.39 (d,  $J = 11.9$  Hz, 6C), 130.85 (d,  $J = 9.7$  Hz, 4C), 131.19 (d,  $J = 2.3$  Hz, 2C), 135.37 (d,  $J = 103.7$  Hz, 2C), 169.79.  $^{31}\text{P}$  NMR ( $\delta$ , ppm): 20.49. MS (EI): m/z, % = 313 (51) [ $\text{M}^+$ ], 284 (75), 202 (100), 183 (9), 155 (9), 77 (13), 47 (16). Anal. calcd for  $\text{C}_{20}\text{H}_{24}\text{DOP}$ , (%): C, 76.65. Found, %: C, 76.75.

*(Z)-(2-Ethylhex-1-en-1-yl)diphenylphosphine oxide (3'd)*

Using the procedure described above, (hex-1-yn-1-yl)(diphenyl)phosphane **1d** (450 mg, 2 mmol) and  $\text{H}_2\text{O}$  instead of  $\text{D}_2\text{O}$  gave crude product that was purified by column chromatography (hexane : ethyl acetate : methanol = 5 : 2 : 1) to afford **3'd** (518 mg, 83%) as yellow oil.  $R_f$  0.30.  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  0.69 (t,  $J = 7.4$  Hz, 3H), 1.03 (t,  $J = 7.4$  Hz, 3H), 1.06 – 1.13 (m, 2H), 1.21 – 1.26 (m, 2H), 2.18 – 2.22 (q,  $J = 7.4$  Hz,  $J = 14.8$  Hz, 2H), 2.47 (t,  $J = 7.4$  Hz, 2H), 5.82 (d,  $J = 25.1$  Hz, 1H), 7.33 – 7.39 (m, 6H), 7.68 – 7.72 (m, 4H);  $^{13}\text{C}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  12.11, 13.79, 22.73, 30.29, 31.19 (d,  $J = 16.2$  Hz, 1C), 33.90 (d,  $J = 7.6$  Hz, 1C), 114.75 (d,  $J = 106.1$  Hz, 1C), 128.38 (d,  $J = 11.8$  Hz, 6C), 130.85 (d,  $J = 9.6$  Hz, 4C), 131.18 (d,

$J = 2.4$  Hz, 2C), 135.41 (d,  $J = 103.4$  Hz, 2C), 169.83. MS (EI): m/z, % = 312 (51) [M<sup>+</sup>], 297 (7), 283 (98), 202 (100), 183 (10), 125 (11), 77 (13), 47 (15). Anal. calcd for C<sub>20</sub>H<sub>25</sub>OP, (%): C, 76.90; H, 8.07. Found, %: C, 76.71; H, 7.85.

*(Z)-(2-Ethylhex-1-en-1-yl)diphenylphosphine sulfide (3'''d)*

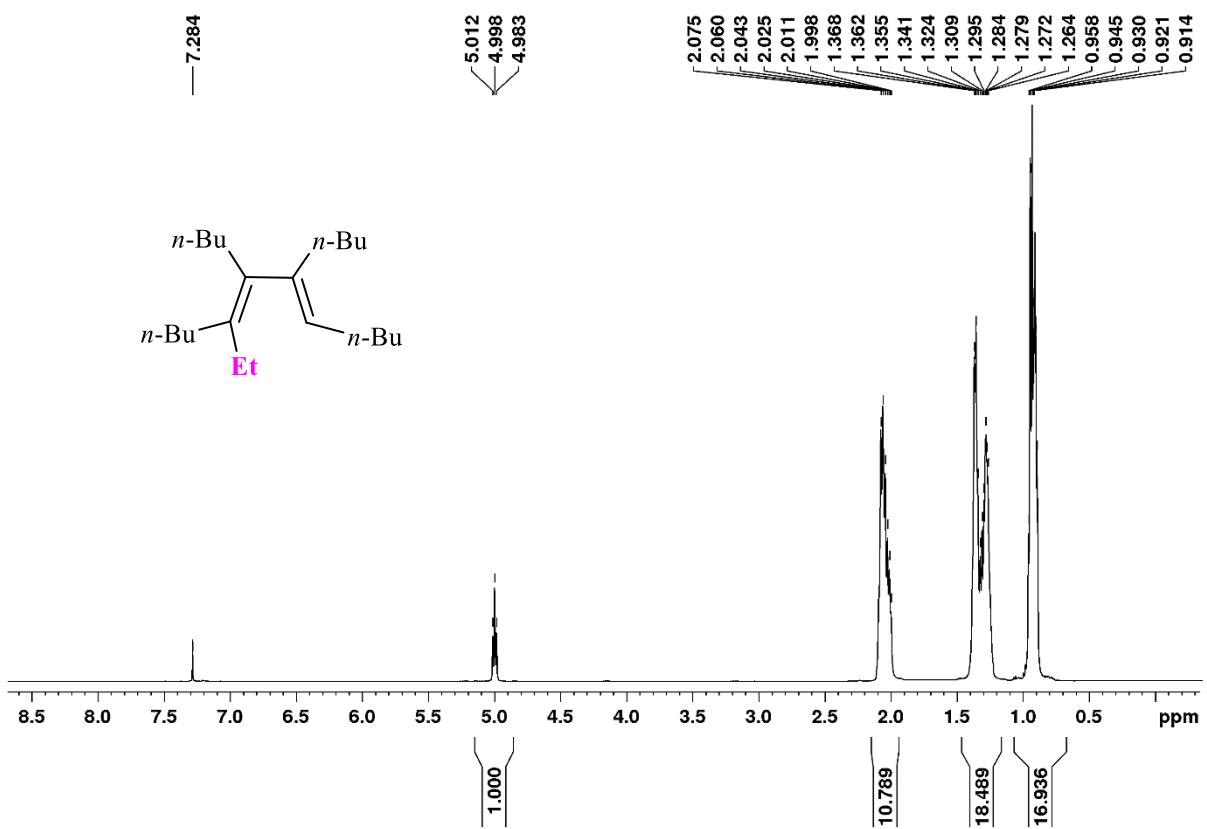
A 50 ml glass reactor equipped with a magnetic stirrer under a dry argon atmosphere at 0 °C, was charged under stirring with toluene (10 ml), TaCl<sub>5</sub> catalyst (143 mg, 0.2 mmol), (hex-1-yn-1-yl)(diphenyl)phosphane **1d** (450 mg, 2 mmol) and Et<sub>3</sub>Al (1.2 ml, 8 mmol). The temperature was raised to 120 °C and the mixture was boiled for an additional 18 h. After boiled for 18 h, elemental sulfur (580 mg, 18 mmol) was added at room temperature. The reaction mixture was stirred for 18 h and then was diluted with hexane (5 ml). Water (3 ml) was added dropwise while cooling the reactor flask in an ice bath. The precipitate was filtered on a filter paper, and the aqueous layer was extracted with diethyl ether (3×5 ml). The combined organic layers were washed with brine (10 ml) and dried over anhydrous CaCl<sub>2</sub>. Evaporation of the solvent and purification of the residue by column chromatography (hexane : ethyl acetate : methanol = (5 : 2 : 1) as eluent) gave **3'''d** (485 mg, 74%) as yellow oil. R<sub>f</sub> 0.44. <sup>1</sup>H NMR ( $\delta$ , ppm, J/Hz): 0.72 (t,  $J = 7.3$ , 3H, C(12)H<sub>3</sub>), 1.13 (t,  $J = 7.4$ , 3H, C(8)H<sub>3</sub>), 1.20-1.35 (m, 2H, C(11)H<sub>2</sub>), 1.35-1.45 (m, 2H, C(10)H<sub>2</sub>), 2.28 (q,  $J = 7.4$ , 2H, C(7)H<sub>2</sub>), 2.37 (t,  $J = 8.0$ , 2H, C(9)H<sub>2</sub>), 6.03 (d,  $J = 23.4$ , 1H, C(5)H<sub>1</sub>), 7.40-7.97 (m, 10H, Ph). <sup>13</sup>C NMR ( $\delta$ , ppm, J/Hz): 12.16 (C(8)), 13.78 (C(12)), 22.80 (C(11)), 29.48 (C(10)), 31.21 (d,  $J = 16.5$ , C(7)), 33.90 (d,  $J = 9.2$ , C(9)), 115.99 (d,  $J = 89.3$ , C(5)), 128.42 (d,  $J = 12.3$ , 4C, C(3)), 130.99 (d,  $J = 2.8$ , 2C, C(4)), 131.19 (d,  $J = 10.6$ , 4C, C(2)), 135.22 (d,  $J = 84.3$ , 2C, C(1)), 168.19 (C(6)). <sup>31</sup>P NMR ( $\delta$ , ppm): 28.67. MS (m/z, %): 328 (50) [M]<sup>+</sup>, 299 (16) [M-Et]<sup>+</sup>, 286 (5), 253 (1), 218 (100) [Ph<sub>2</sub>PS], 183 (27), 139 (28), 108 (20), 91 (10), 63 (10) [PS], 41 (10). The spectral properties (<sup>1</sup>H NMR, <sup>13</sup>C NMR) were in good agreement with those that were reported in the literature [S4].

*(Z)-Diphenyl(2-p-tolylbut-1-en-1-yl)phosphine oxide (3e)*

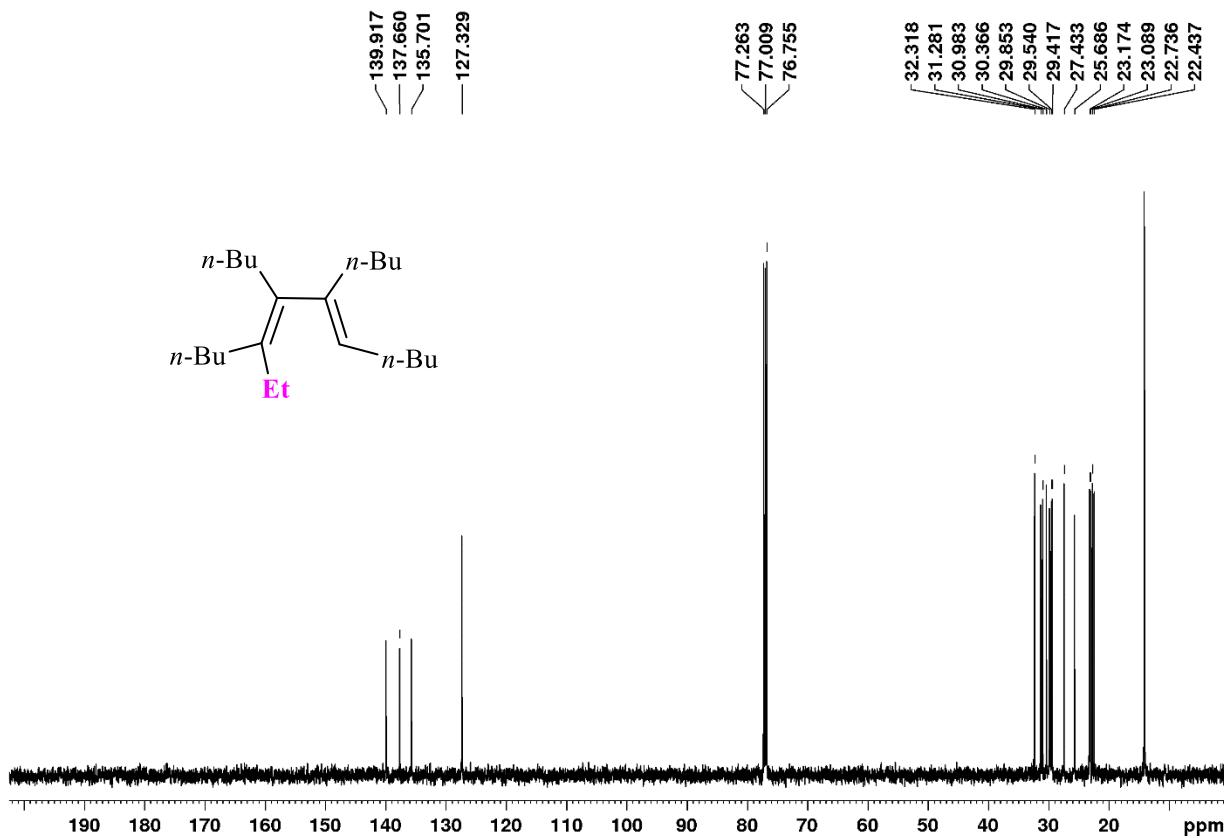
According to the procedure for (Z)-(1-deuterio-2-ethylhex-1-en-1-yl)(diphenyl)phosphine oxide **3'''d**, compound **3e** was isolated by column chromatography (hexane : ethyl acetate : methanol = (5 : 2 : 1) as eluent), which provided **3e** as a yellow oil (484 mg, 70 %). <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  1.09 (t,  $J = 7.4$  Hz, 3H), 2.18 (s, 2H), 2.56 – 2.60 (q,  $J = 7.3$  Hz,  $J = 14.7$  Hz, 2H), 6.26 (d,  $J = 19.5$  Hz, 1H), 6.84 (d,  $J = 7.8$  Hz, 2H), 7.09 (d,  $J = 7.9$  Hz, 2H), 7.23 – 7.34 (m, 6H), 7.59 – 7.62 (m, 4H); <sup>13</sup>C NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  12.55, 21.10, 34.64 (d,  $J = 15.6$  Hz, 1C), 118.05 (d,  $J = 104.6$  Hz, 1C), 127.99 (s, 4C), 128.17 (d,  $J = 22.8$  Hz, 4C), 130.76 (d,  $J = 2.6$  Hz, 2C), 130.86 (d,  $J = 9.5$  Hz, 2H), 134.53 (d,  $J = 105.2$  Hz, 2C), 137.81, 166.58. Anal. Calcd for C<sub>23</sub>H<sub>23</sub>OP, (%): C, 79.75; H, 6.69. Found, %: C, 79.89; H, 6.77.

*(Z)-Trimethyl(2-phenylbut-1-en-1-yl)silane (3f)*

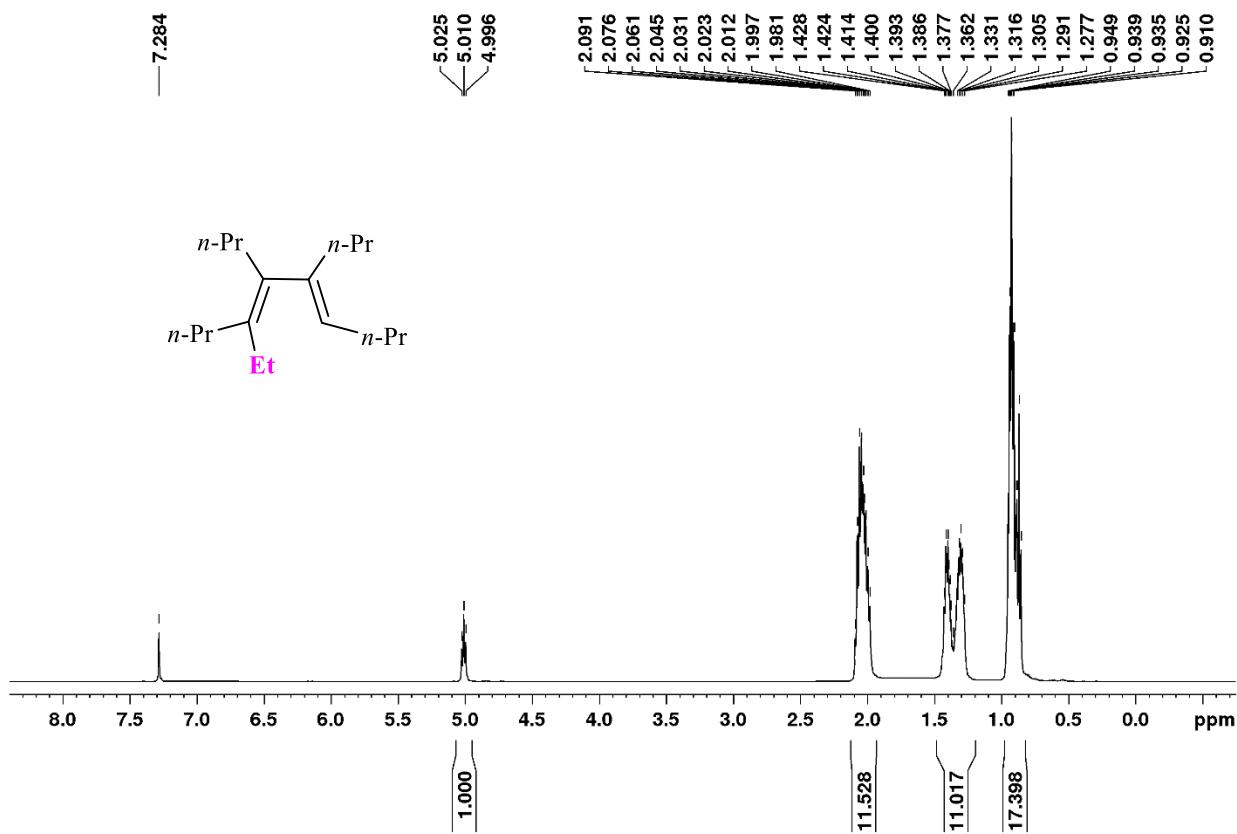
According to the general procedure, compound **3f** was isolated by distillation through a microcolumn at 10 Torr to afford **3f** (74 mg, 22%) as a colorless oil. b.p. 114-116 °C (10 Torr). <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 0.24 (s, 9H), 1.03 (t, *J* = 7.3 Hz, 3H), 2.67 – 2.71 (q, *J* = 7.6 Hz, *J* = 14.9 Hz, 2H), 5.78 (s, 1H), 2.31 – 2.36 (m, 2H), 7.27 – 7.30 (m, 1H), 7.35 (t, *J* = 7.5 Hz, 2H), 7.44 (d, *J* = 7.7 Hz, 2H). <sup>13</sup>C NMR (500 MHz, CDCl<sub>3</sub>) δ 0.27 (3C), 14.14, 27.89, 126.25 (2C), 127.22, 127.25, 128.17 (2C), 143.33, 159.06. MS (EI): m/z, % = 205 (14), 204 (1) [M<sup>+</sup>], 190 (70), 174 (4), 135 (100), 105 (3), 73 (16), 43 (7). Anal. calcd for C<sub>13</sub>H<sub>20</sub>Si, (%): C, 76.40; H, 9.86. Found, %: C, 76.53; H, 9.94.



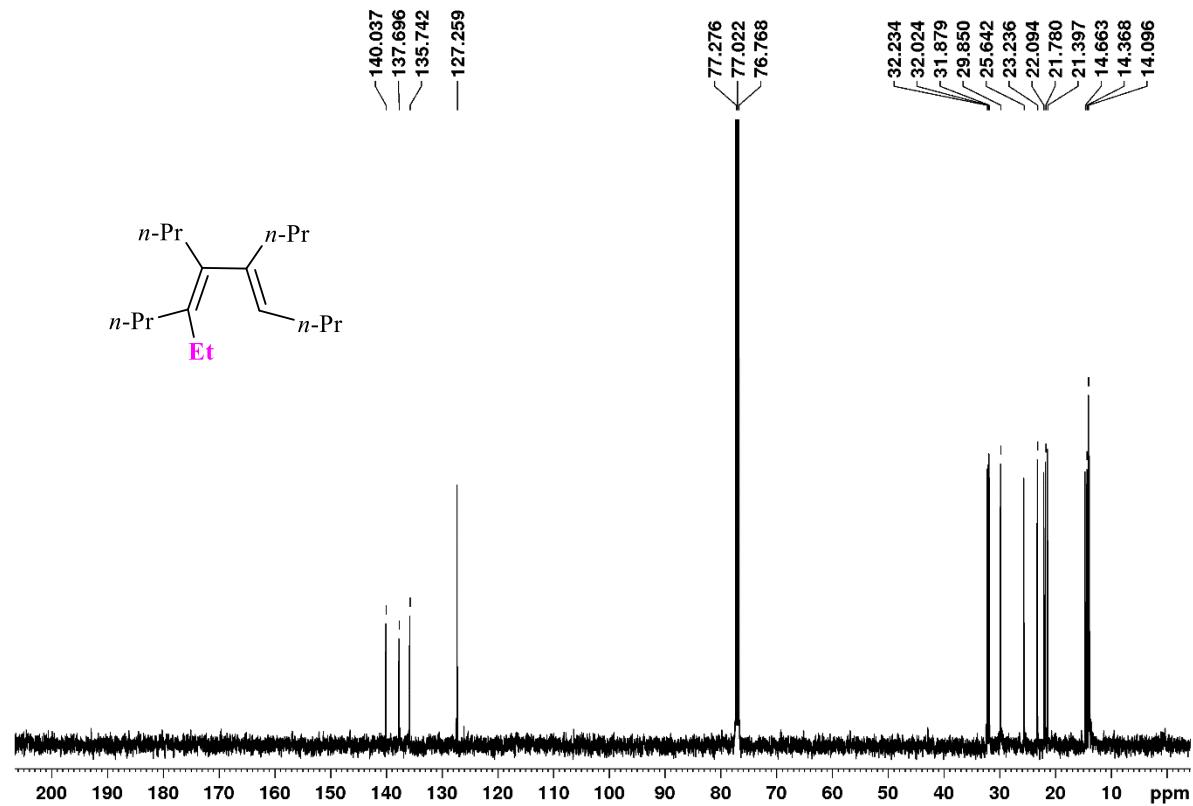
<sup>1</sup>H NMR spectrum of (5Z,7E)-6,7-dibutyl-5-ethyldodeca-5,7-diene (**2a**)



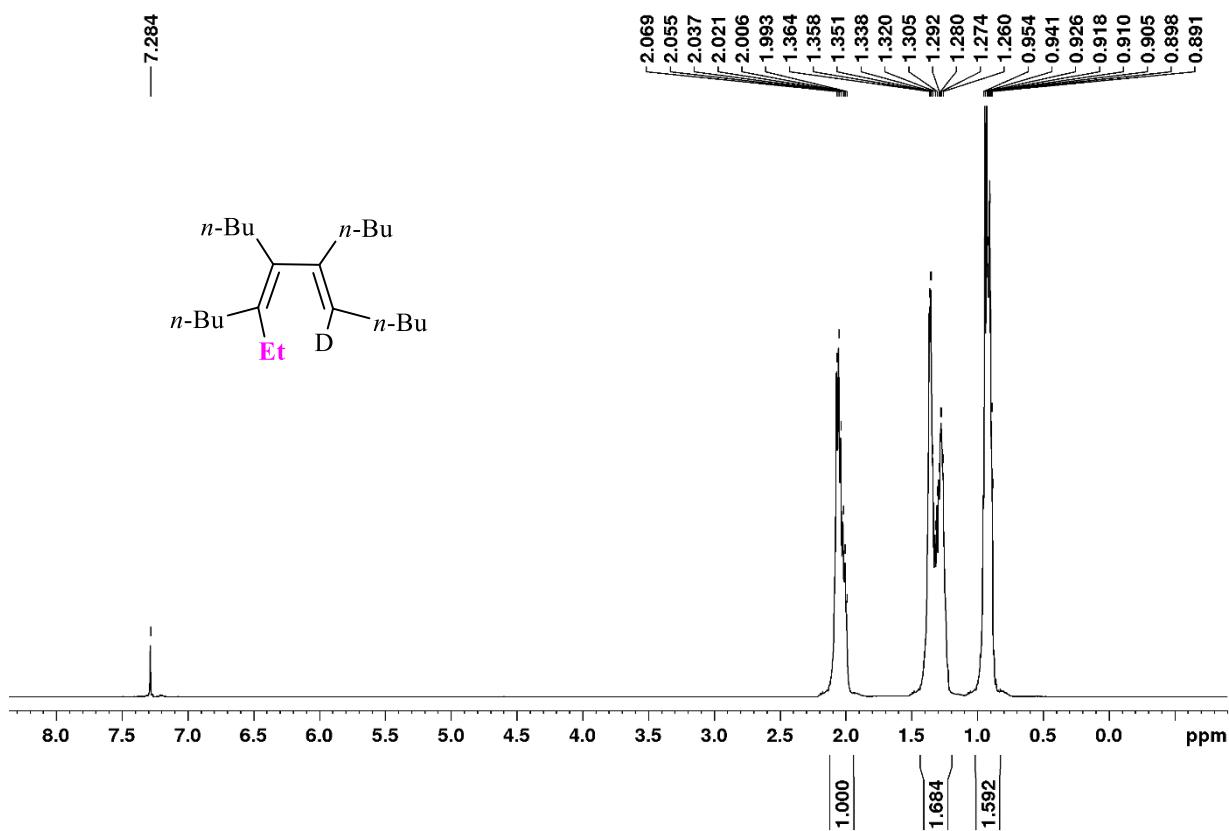
<sup>13</sup>C NMR spectrum of (5Z,7E)-6,7-dibutyl-5-ethyldodeca-5,7-diene (**2a**)



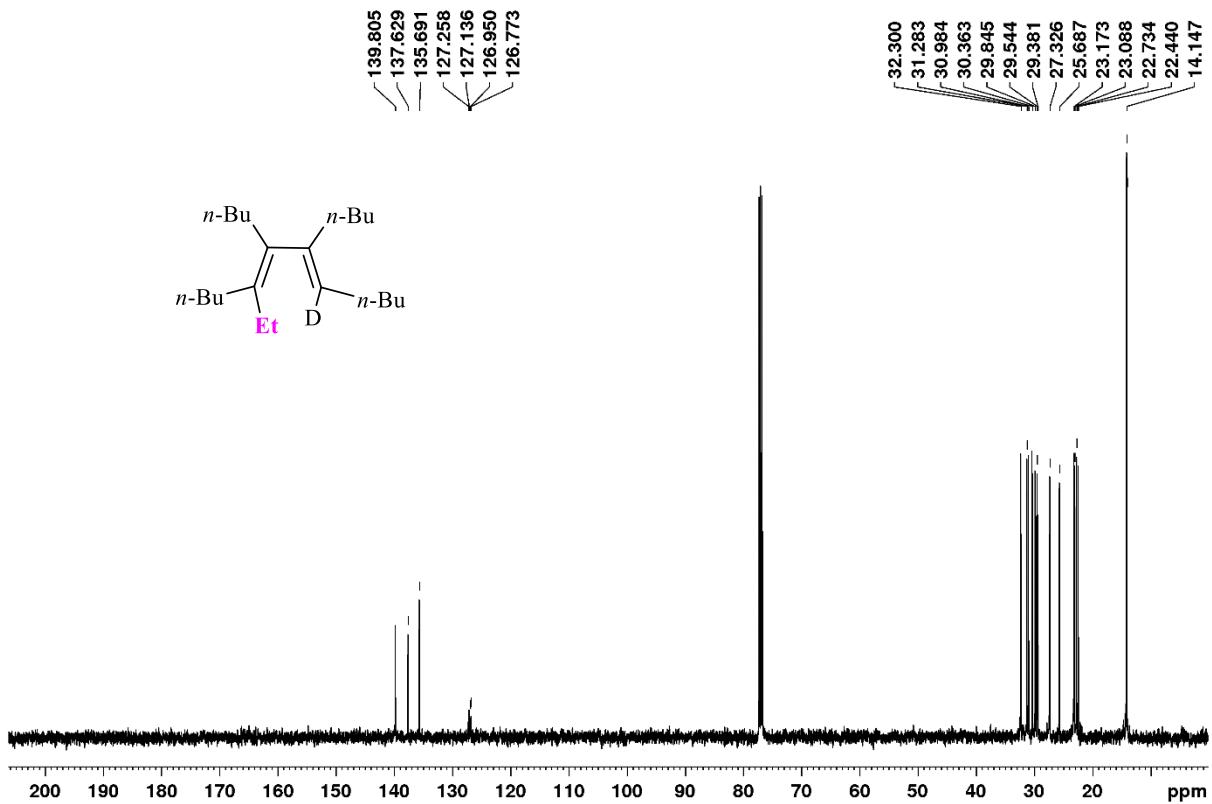
<sup>1</sup>H NMR spectrum of (4Z,6E)-4-ethyl-5,6-dipropyldeca-4,6-diene (**2b**)



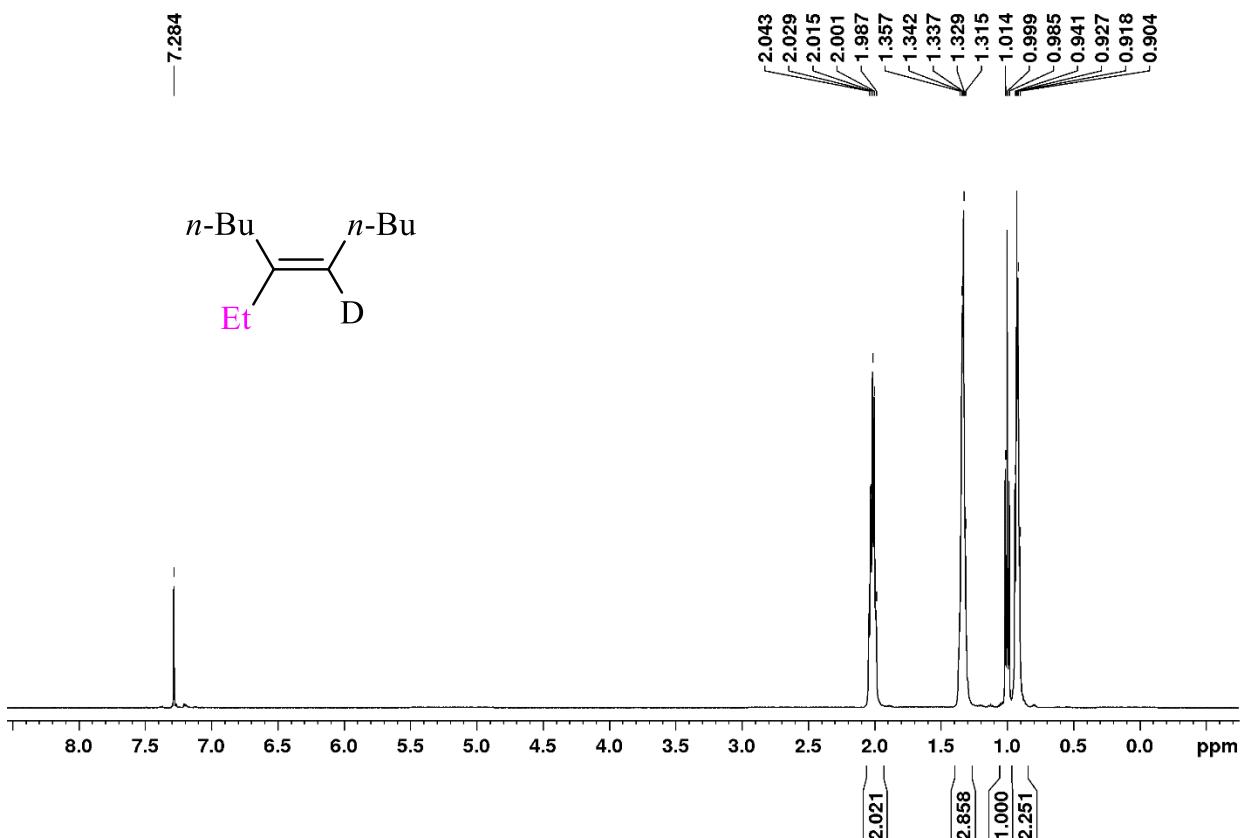
<sup>13</sup>C NMR spectrum of (4Z,6E)-4-ethyl-5,6-dipropyldeca-4,6-diene (**2b**)



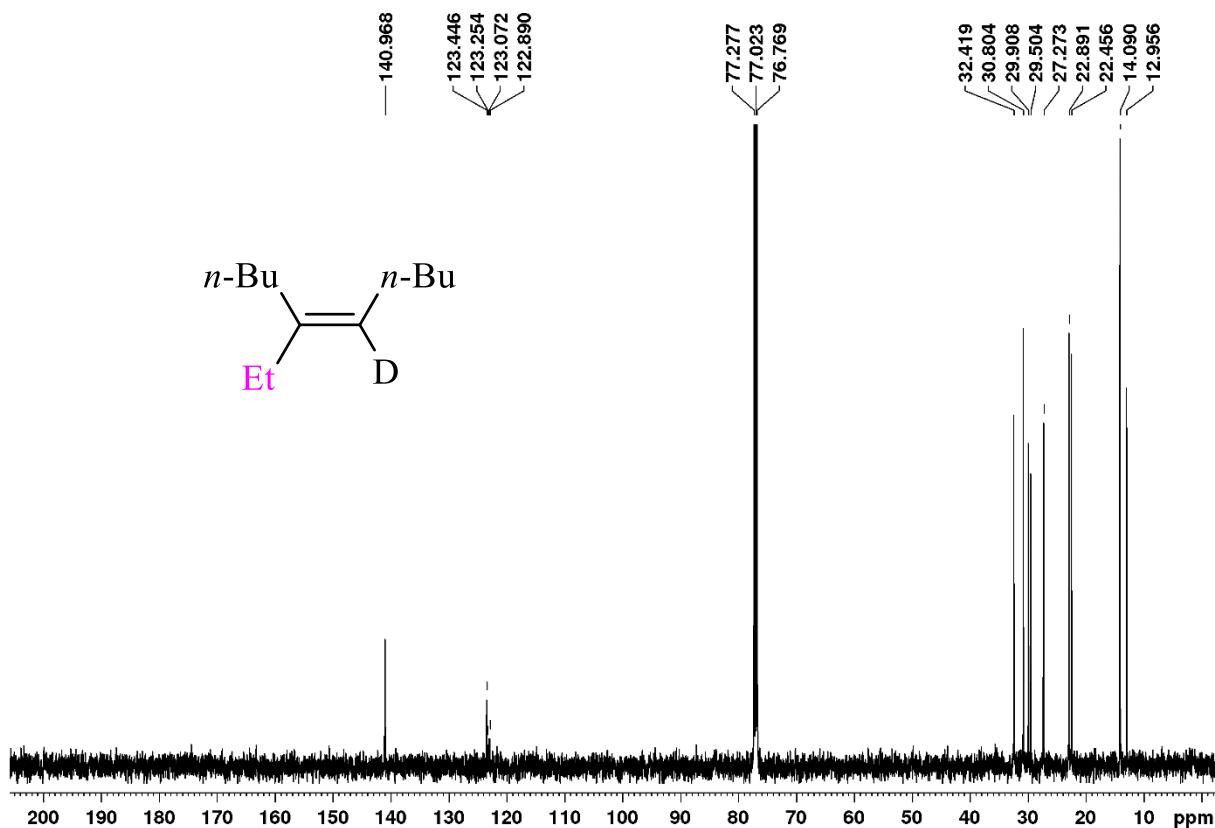
<sup>1</sup>H NMR spectrum of (5Z,7E)-6,7-dibutyl-8-deuterio-5-ethyldodeca-5,7-diene (**2'a**)



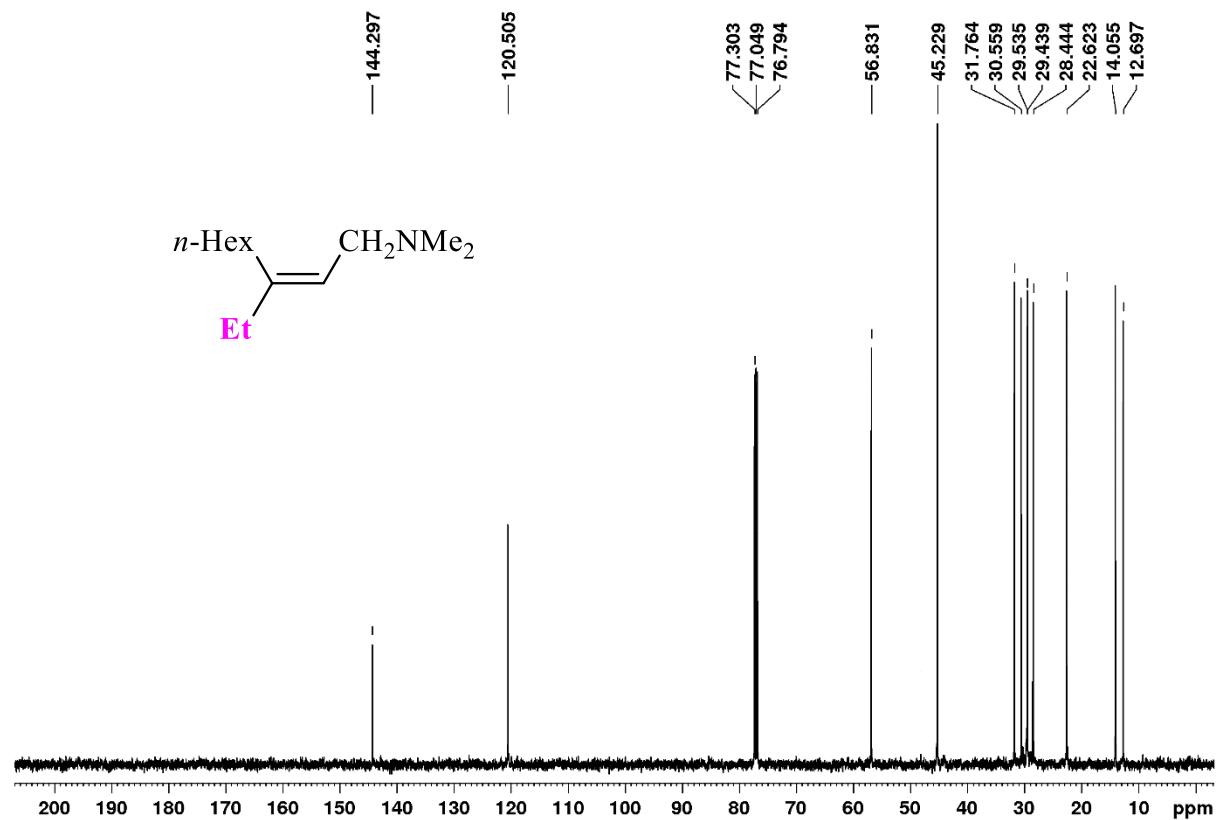
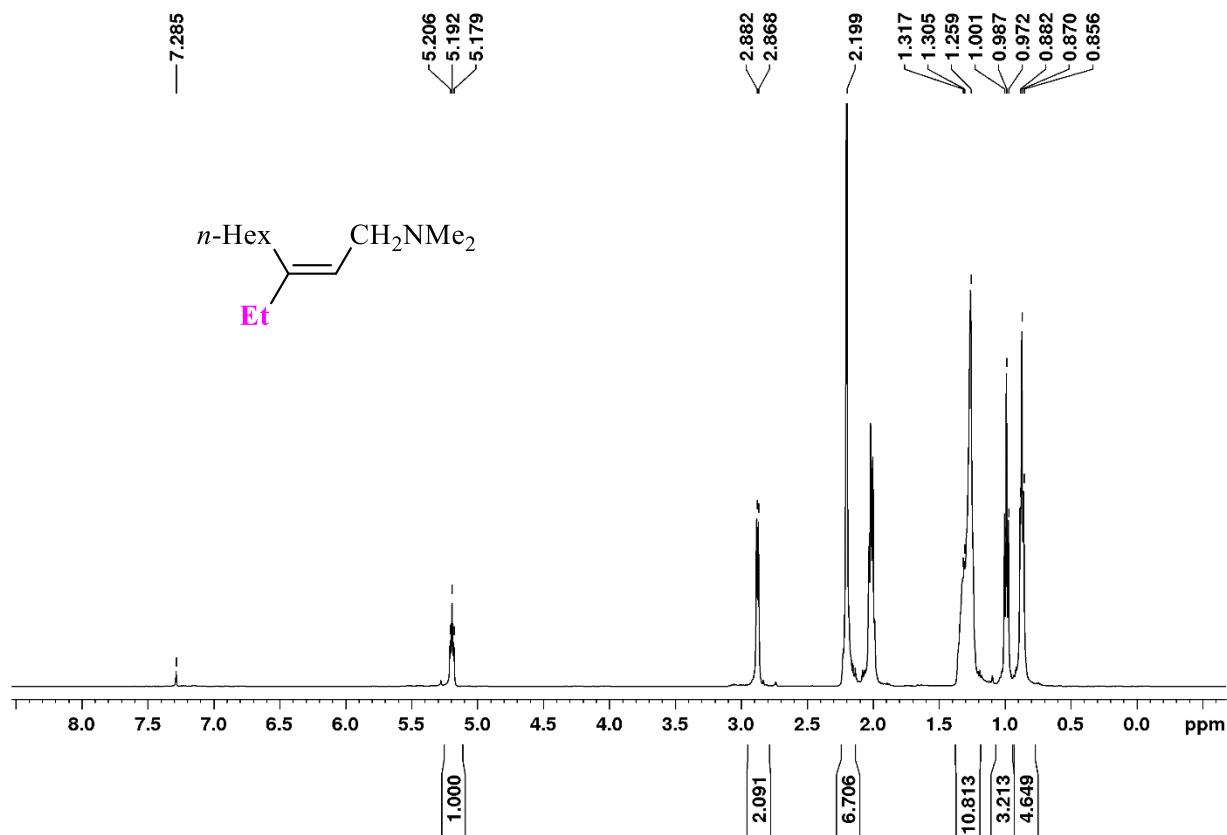
<sup>13</sup>C NMR spectrum of (5Z,7E)-6,7-dibutyl-8-deuterio-5-ethyldodeca-5,7-diene (**2'a**)

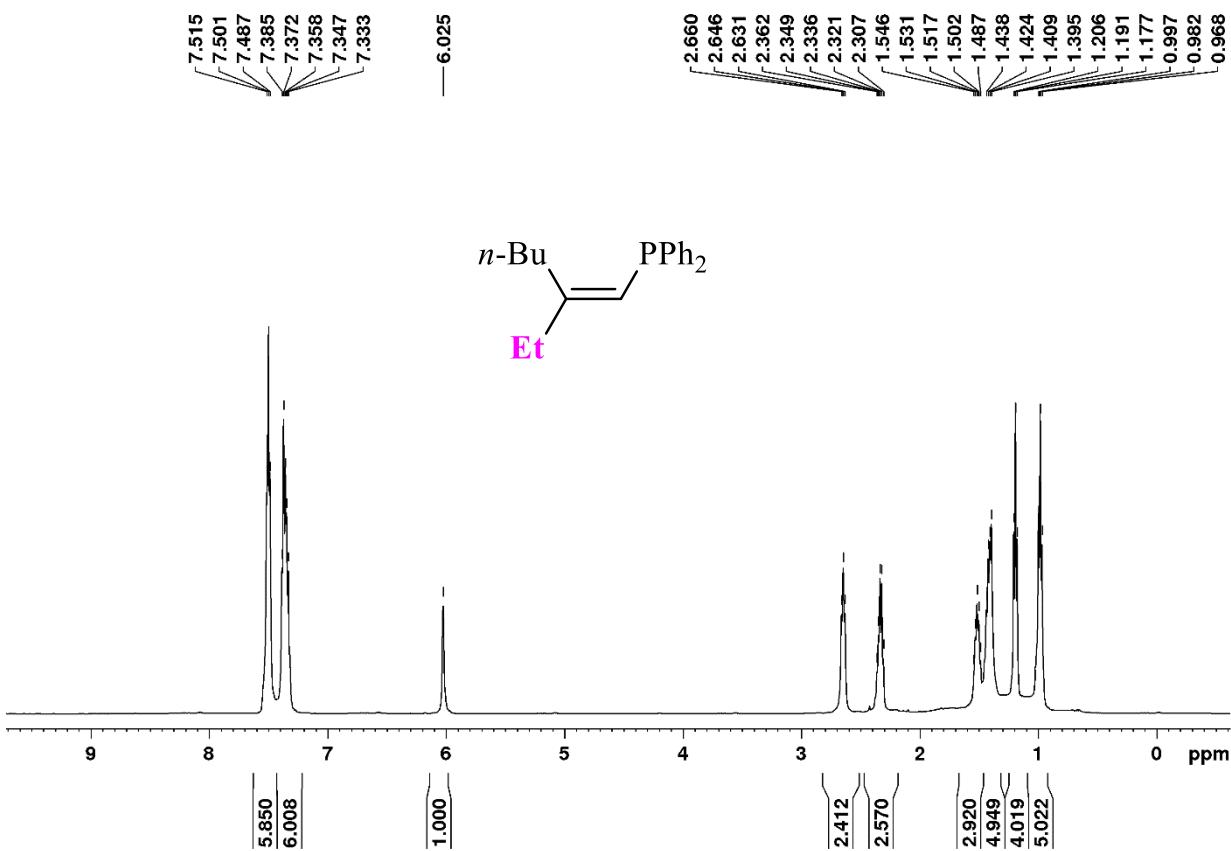


$^1\text{H}$  NMR spectrum of (*Z*)-6-deuterio-5-ethyldec-5-ene (**3'a**)

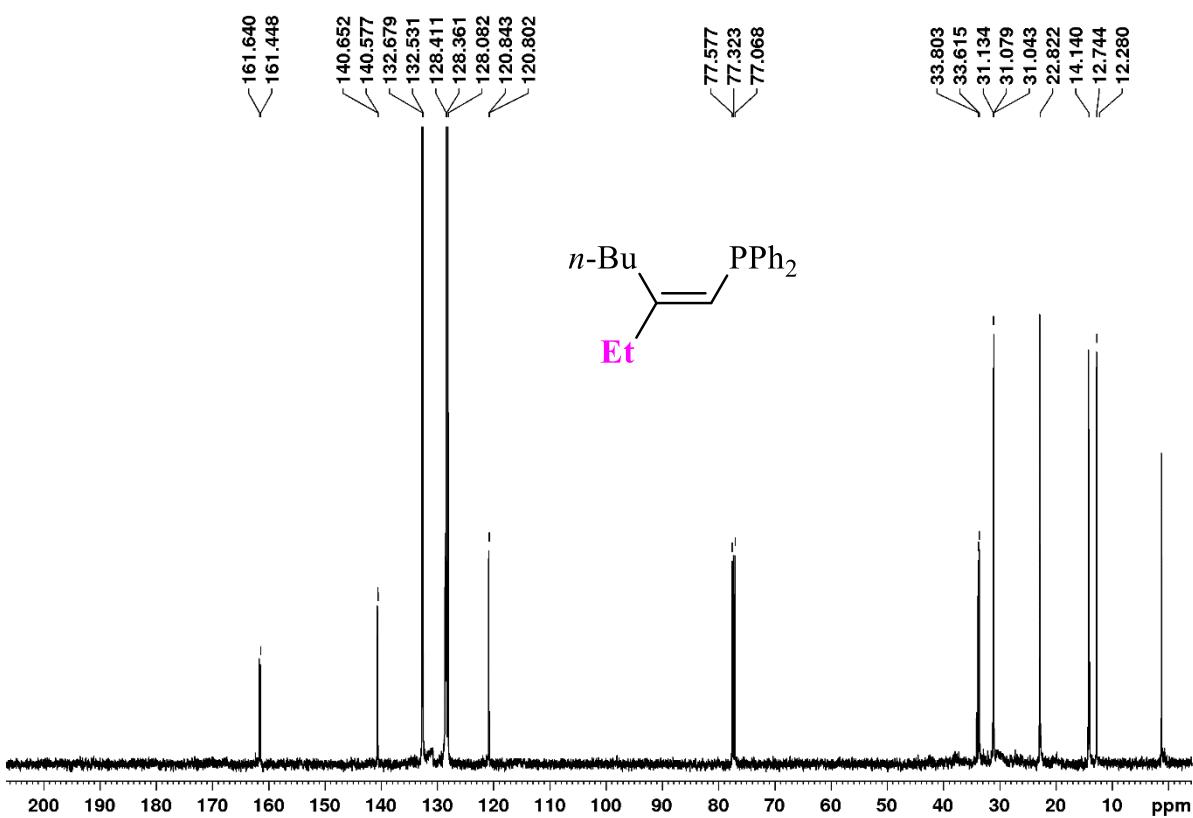


$^{13}\text{C}$  NMR spectrum of (*Z*)-6-deuterio-5-ethyldec-5-ene (**3'a**)

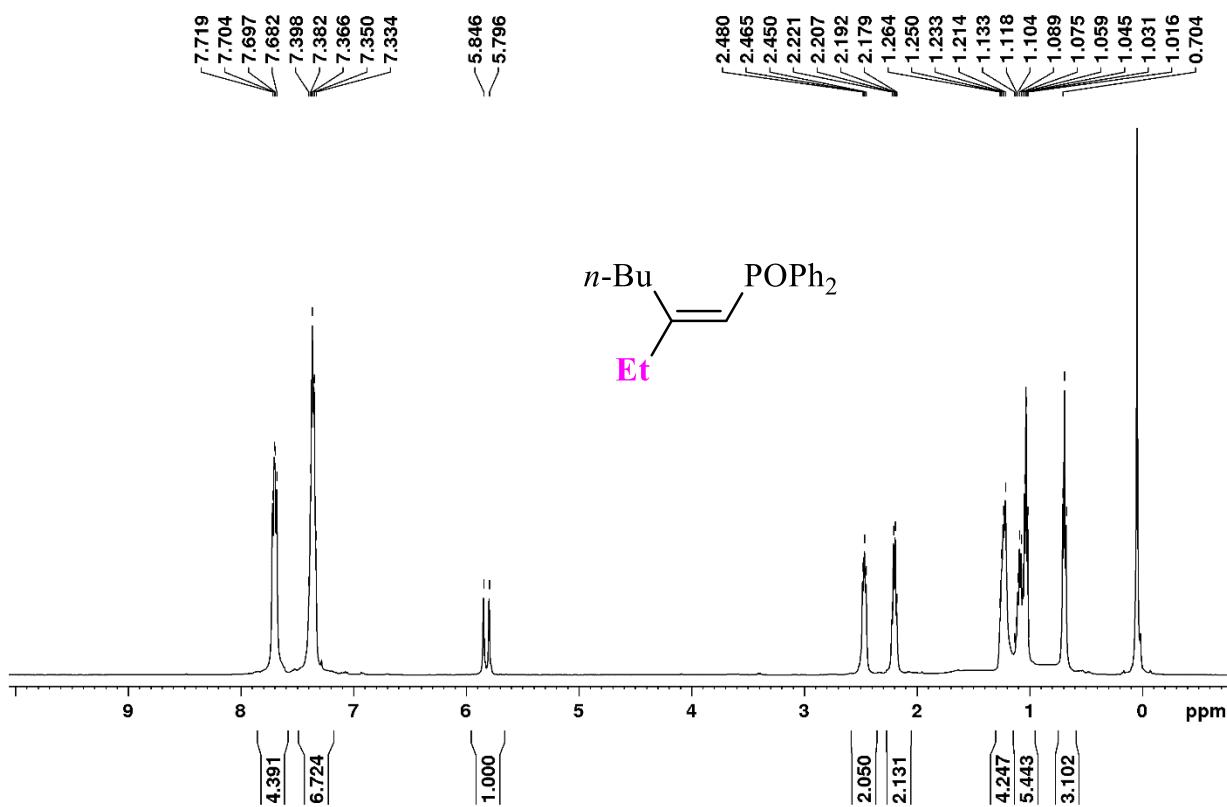




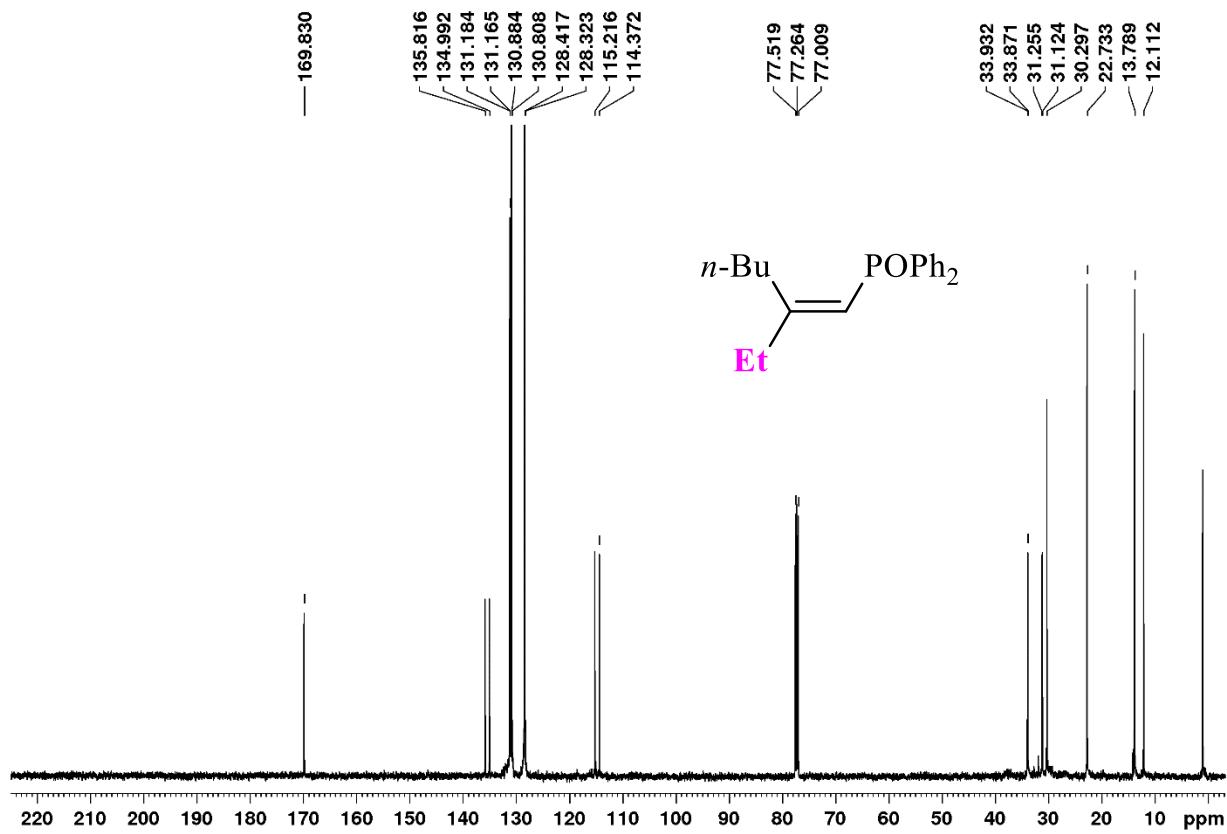
<sup>1</sup>H NMR spectrum of (Z)-(2-ethylhex-1-en-1-yl)diphenylphosphane (**3d**)



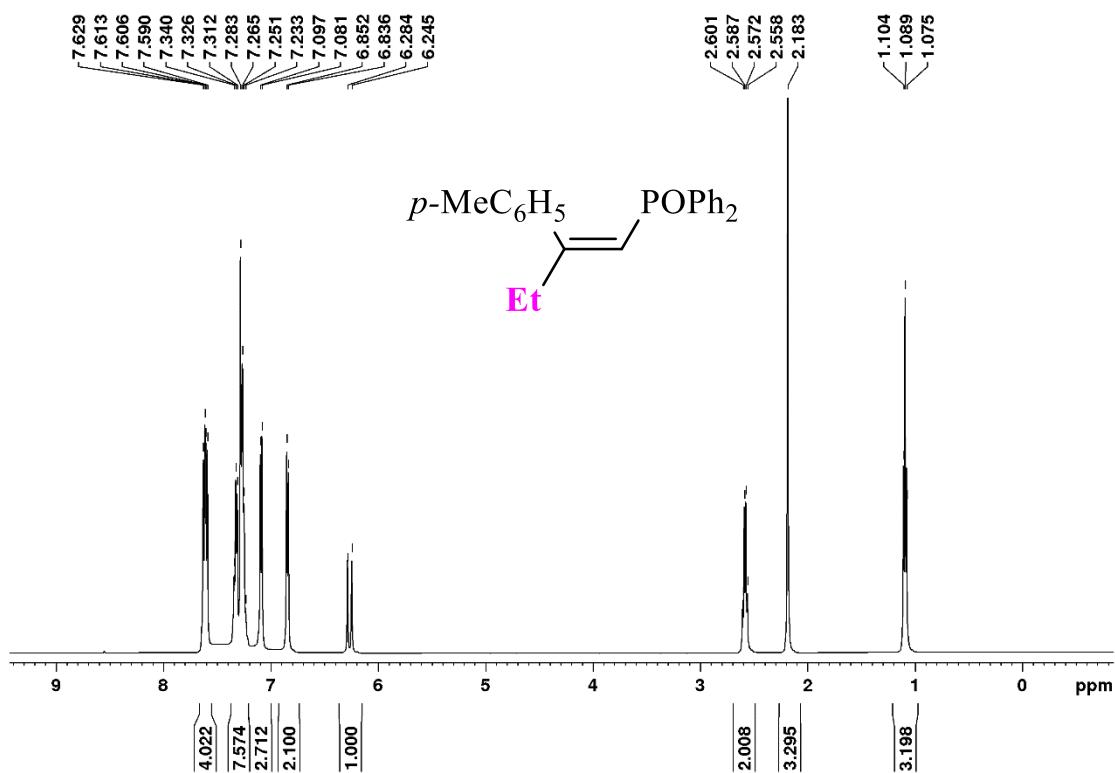
<sup>13</sup>C NMR spectrum of (Z)-(2-ethylhex-1-en-1-yl)diphenylphosphane (**3d**)



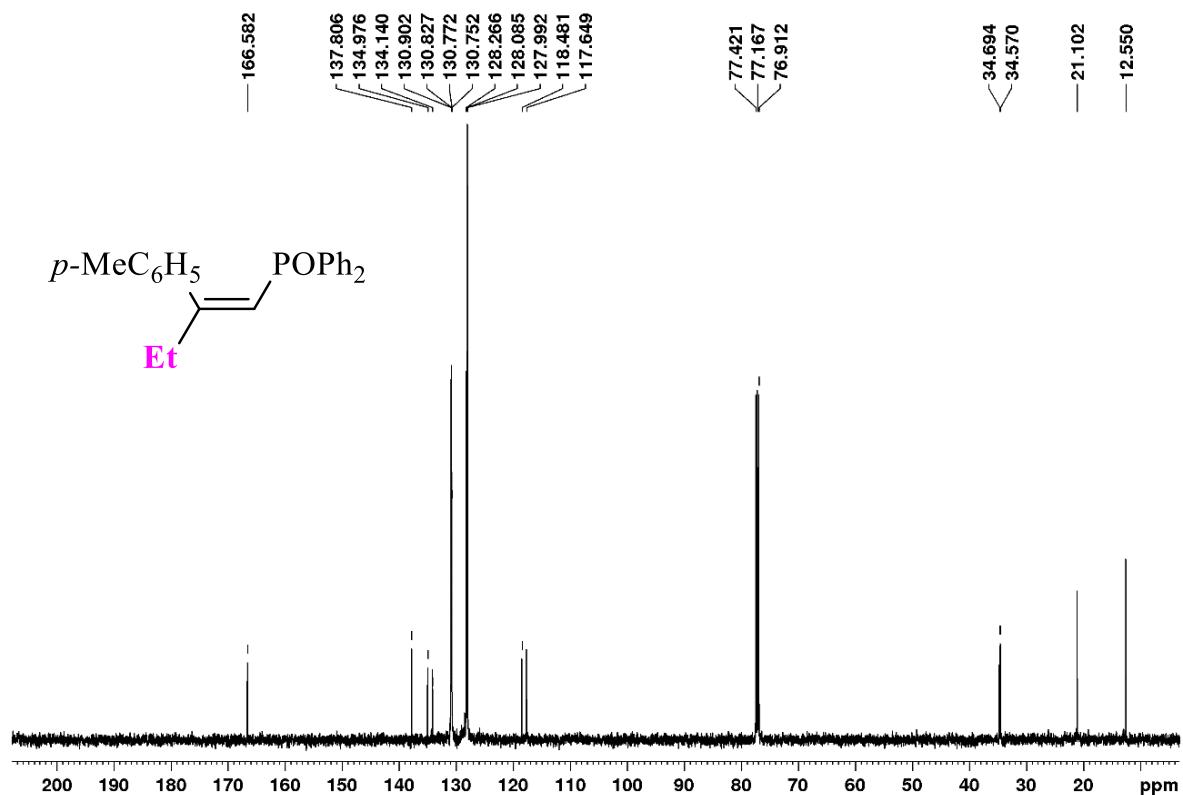
<sup>1</sup>H NMR spectrum of (Z)-(2-ethylhex-1-en-1-yl)diphenylphosphine oxide (**3'd**)



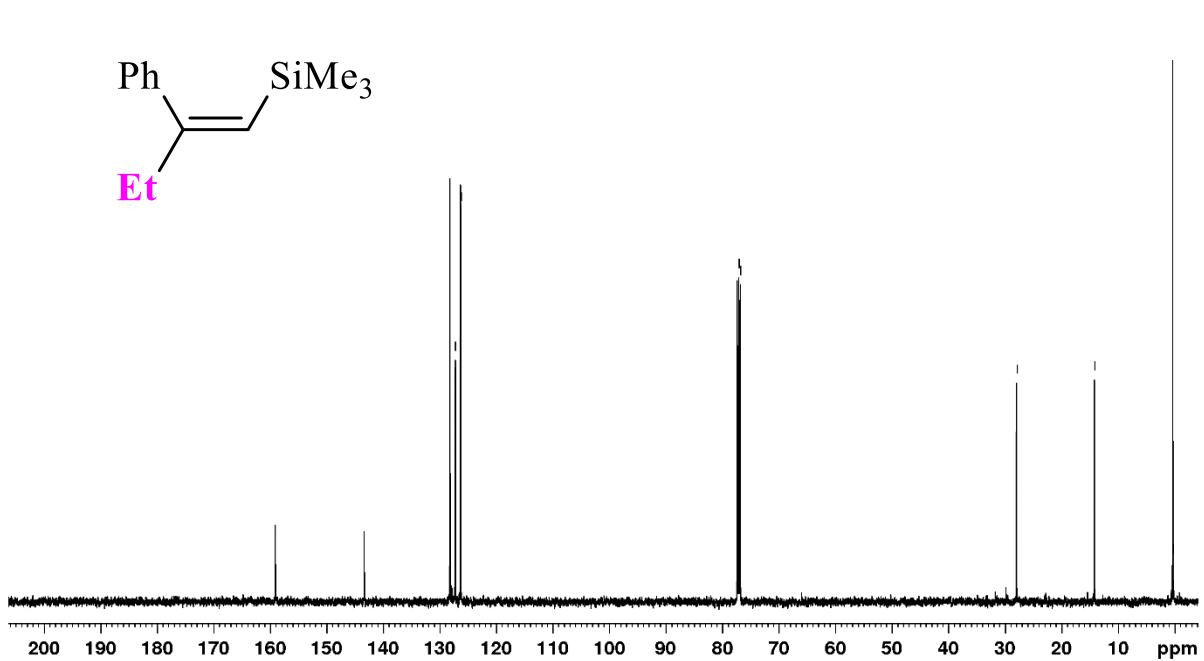
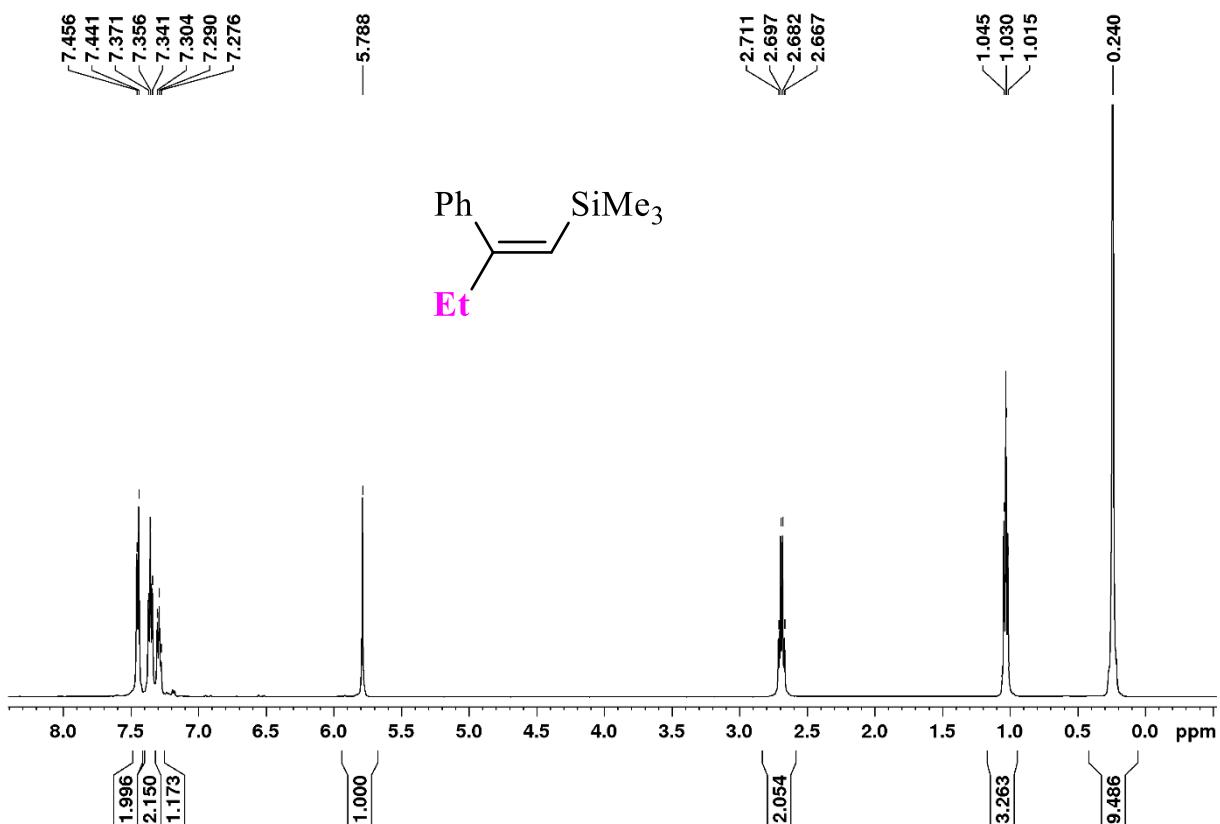
<sup>13</sup>C NMR spectrum of (Z)-(2-ethylhex-1-en-1-yl)diphenylphosphine oxide (**3'd**)

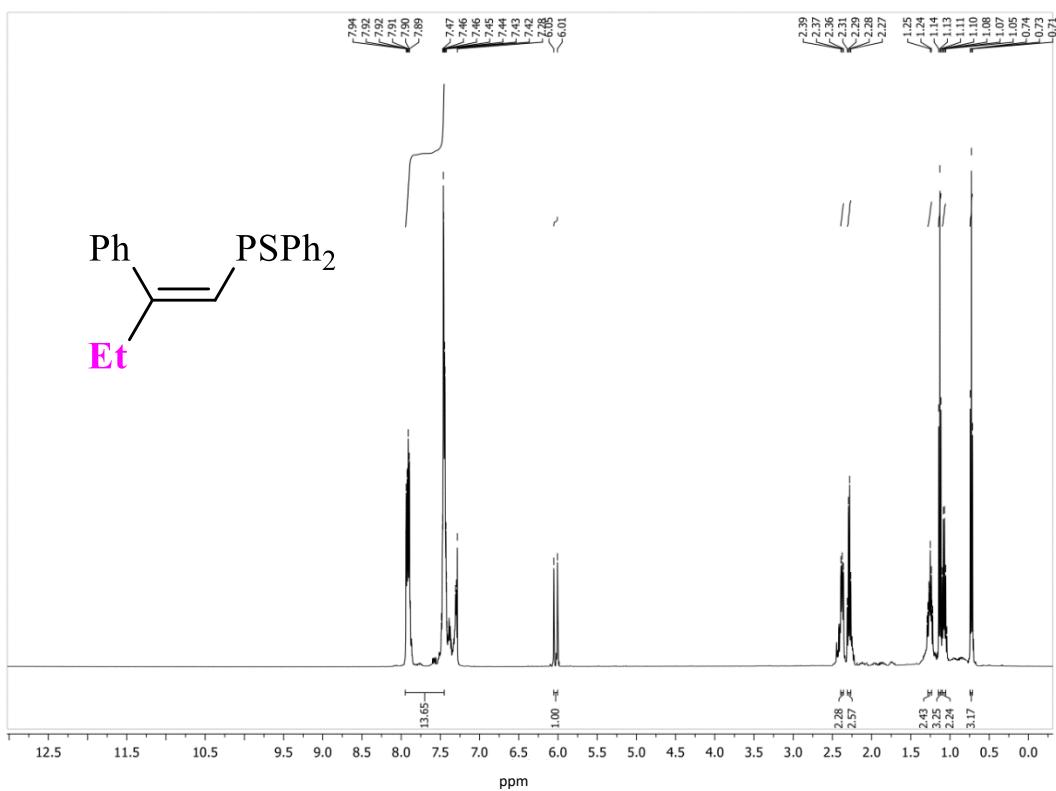


<sup>1</sup>H NMR spectrum of (Z)-diphenyl(2-*p*-tolylbut-1-en-1-yl)phosphine oxide (**3e**)

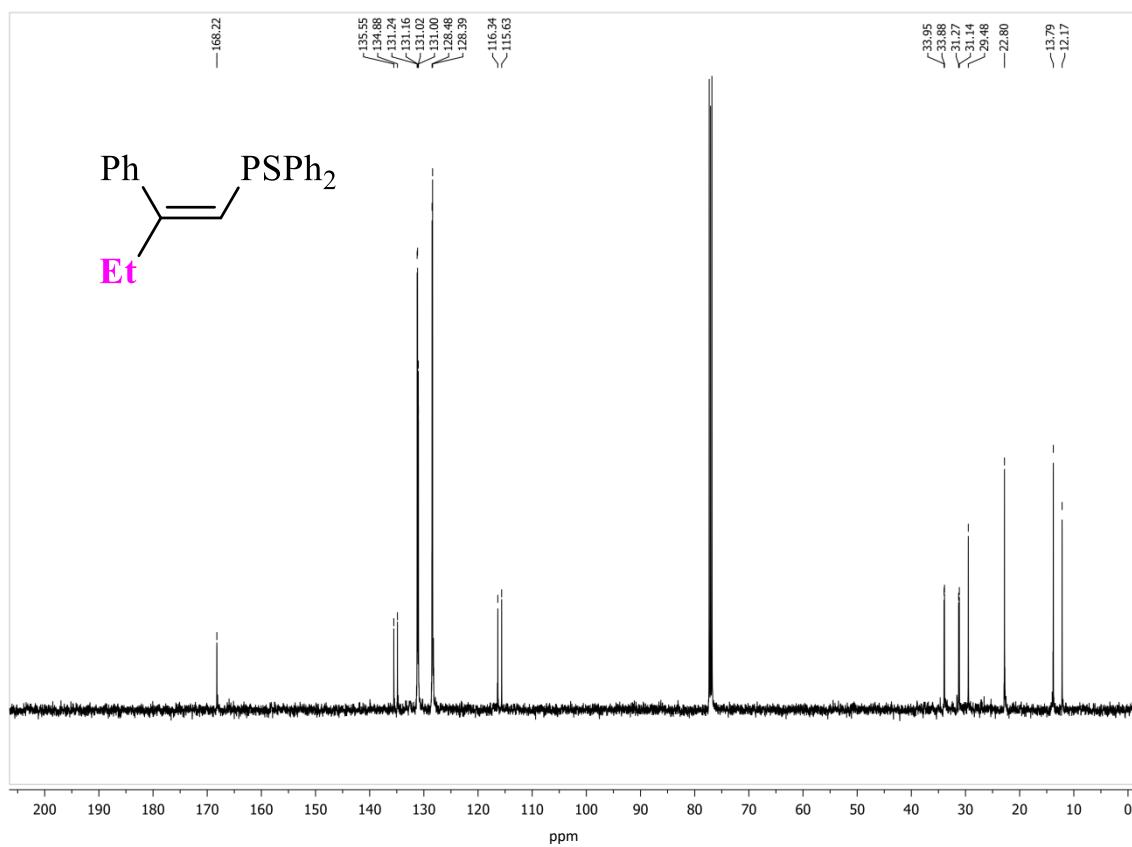


<sup>13</sup>C NMR spectrum of (Z)-diphenyl(2-*p*-tolylbut-1-en-1-yl)phosphine oxide (**3e**)

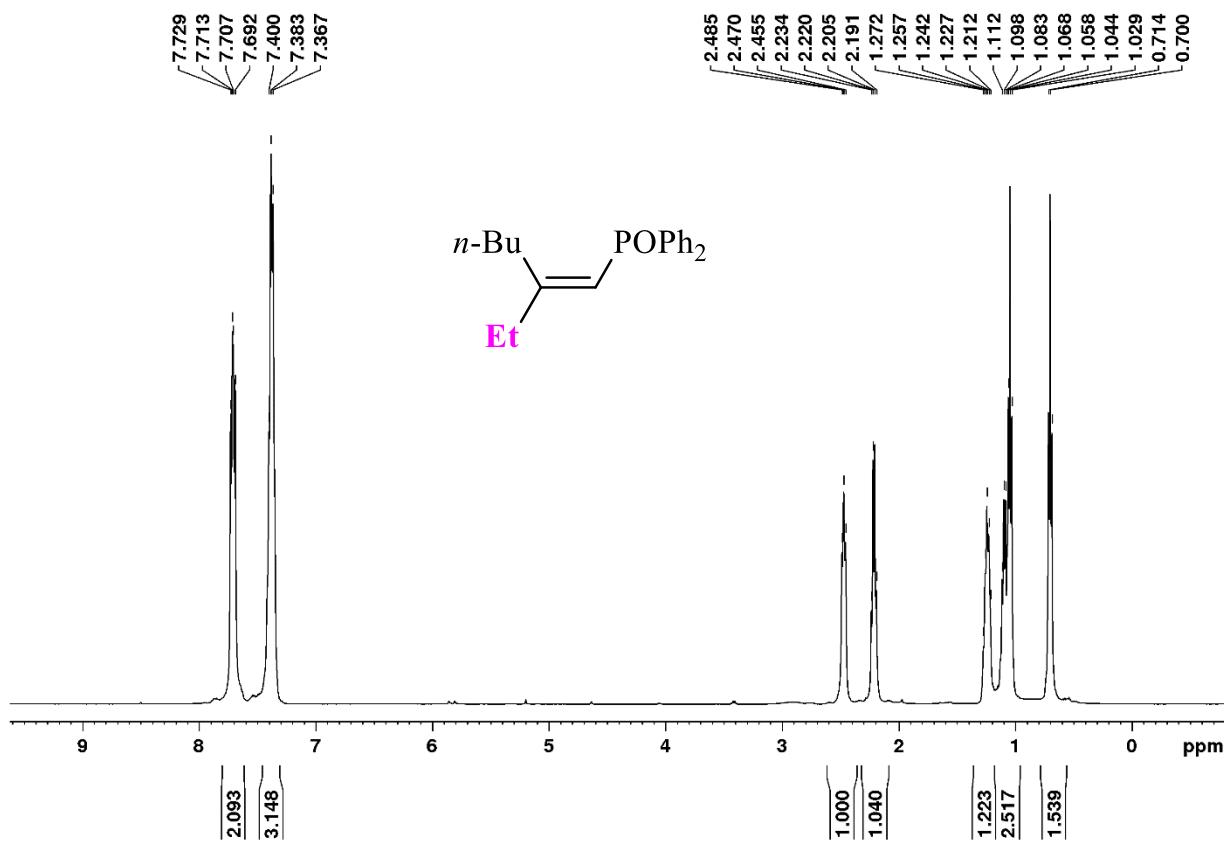




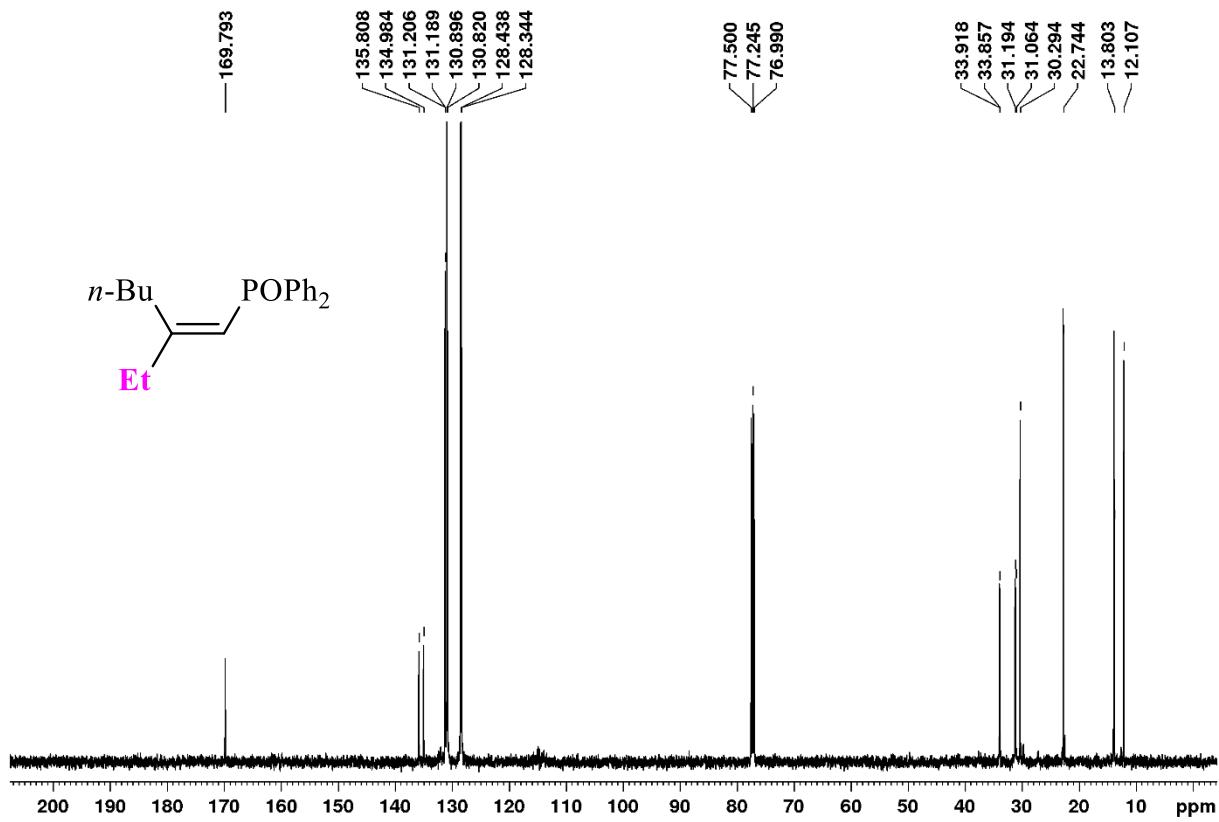
$^1\text{H}$  NMR spectrum of (Z)-(2-ethylhex-1-en-1-yl)(diphenyl)phosphine sulfide (**3'''d**)



$^{13}\text{C}$  NMR spectrum of (Z)-(2-ethylhex-1-en-1-yl)(diphenyl)phosphine sulfide (**3'''d**)



$^1\text{H}$  NMR spectrum of (Z)-(1-deutero-2-ethylhex-1-en-1-yl)(diphenyl)phosphine oxide (**3''d**)



$^{13}\text{C}$  NMR spectrum of (Z)-(1-deutero-2-ethylhex-1-en-1-yl)(diphenyl)phosphine oxide (**3''d**)

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