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Supporting Information

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Supporting Information

Palladium-Catalyzed Intermolecular Three-Component Coupling of Organic Halides with Alkynes and Alkenes: Efficient Synthesis of Oligoene Compounds

Kana Shibata, Tetsuya Satoh,* and Masahiro Miura*

*Department of Applied Chemistry, Faculty of Engineering, Osaka University, Suita, Osaka
565-0871, Japan*

e-mail: satoh@chem.eng.osaka-u.ac.jp; miura@chem.eng.osaka-u.ac.jp

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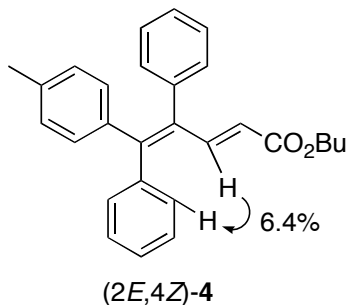
Experimental Section

General. ^1H and ^{13}C NMR spectra were recorded at 400 or 270 MHz and 100 or 68 MHz, respectively, for CDCl_3 solutions. MS data were obtained by EI, unless noted. GC analysis was carried out using a silicon OV-17 column (i. d. 2.6 mm x 1.5 m) or a CBP-1 capillary column (i. d. 0.5 mm x 25 m). GC-MS analysis was carried out using a CBP-1 capillary column (i. d. 0.25 mm x 25 m).

Bromides **7b-h** and **11**^[11b] and diarylacetylenes **2b-d**^[15] and **2e**^[16] were prepared by the method reported previously. Other starting materials were commercially available.

The following experimental procedures may be regarded as typical in methodology and scale.

Palladium-Catalyzed Reaction of 4-Iodotoluene (1b) with Diphenylacetylene (2a) and Butyl Acrylate (3a) (entry 10 in Table 1): A mixture of 4-iodotoluene (**1b**) (1 mmol, 218 mg), diphenylacetylene (**2a**) (4 mmol, 712 mg), butyl acrylate (**3a**) (1 mmol, 128 mg), $\text{Pd}(\text{OAc})_2$ (0.05 mmol, 11 mg), LiCl (0.7 mmol, 30 mg), NaHCO_3 (2 mmol, 168 mg), and 1-methylnaphthalene (ca. 60 mg) as internal standard was stirred in $\text{DMF}/\text{H}_2\text{O}$ (5 mL, 9:1) under nitrogen at 130 °C. After 1 h, the reaction mixture was poured into ether (70 mL), washed by water (100 mL, three times), and dried over sodium sulfate. GC and GC-MS analyses confirmed the formation of **4** and **5** in 72 and 12% yields, respectively. The product **4** (197 mg, 50%) was isolated by column chromatography on silica gel using hexane-ethyl acetate (99.5:0.5, v/v) as eluant. Although the NMR spectra of **4** indicated that it consists of two geometrical isomers ((*2E,4E*):(*2E,4Z*) = 1.2:1), the minor isomer could be isolated by recrystallization from hexane after the column chromatographic separation and was determined to possess (*2E,4Z*)-geometry by NOE experiments as shown below.



Characterization Data of Products.

Butyl (2E)-5-(4-methylphenyl)-4,5-diphenyl-2,4-pentadienoate (4, 4E:4Z = 1.2:1).^[7a] Oil; ¹H NMR (400 MHz, CDCl₃) δ 0.87-0.91 (m, 3H), 1.25-1.36 (m, 2H), 1.53-1.58 (m, 2H), 2.20 (s, 3H, 4Z), 2.39 (s, 3H, 4E), 4.04-4.09 (m, 2H), 5.59 (d, *J* = 15.4 Hz, 1H, 4Z), 5.61 (d, *J* = 15.4 Hz, 1H, 4E), 6.76 (d, *J* = 8.1 Hz, 2H, 4Z), 6.83 (d, *J* = 8.1 Hz, 2H, 4Z), 6.88-6.90 (m, 2H, 4E), 7.01-7.04 (m, 2H, 4E), 7.08-7.37 (m, 10H), 7.70 (d, *J* = 15.4 Hz, 1H, 4Z), 7.76 (d, *J* = 15.4 Hz, 1H, 4E); ¹³C NMR (100 MHz, CDCl₃) δ 13.7 (overlapped), 19.1 (4Z), 19.2 (4E), 21.2 (4Z), 21.3 (4E), 30.68 (4Z), 30.71 (4E), 64.0 (overlapped), 121.3 (4Z), 121.4 (4E), 126.9 (4Z), 127.0 (4E), 127.4 (4E), 128.0 (4Z), 128.10 (4E), 128.12 (4Z), 128.15 (4Z), 128.18(4Z), 128.8 (4E), 130.98 (4Z), 131.03 (overlapped), 131.08 (overlapped), 131.14 (4E), 136.6 (4Z), 136.7 (4E), 137.0 (4Z), 138.2 (4E), 138.5 (4E), 139.0 (4Z), 139.51 (4E), 139.54 (4Z), 141.6 (4Z), 142.1 (4E), 146.4 (4Z), 146.5 (4E), 149.6 (4Z), 149.7 (4E), 167.6 (4Z), 167.7 (4E); MS *m/z* 396 (M⁺). Anal. Calcd for C₂₈H₂₈O₂: C, 84.81, H, 7.12. Found: C, 84.67, H, 7.16.

Butyl (2E)-4,5,5-triphenyl-2,4-pentadienoate (6a).^[7a] Mp 108.5-110 °C; ¹H NMR (400 MHz, CDCl₃) δ 0.89 (t, *J* = 7.3 Hz, 3H), 1.31 (m, 2H), 1.56 (m, 2H), 4.07 (t, *J* = 6.6 Hz, 2H), 5.61 (d, *J* = 15.4 Hz, 1H), 6.88-6.90 (m, 2H), 7.02-7.09 (m, 3H), 7.09 (dd, *J* = 2.2 Hz, 2H), 7.18-7.25 (m, 5H), 7.35-7.40 (m, 3H), 7.72 (d, *J* = 15.4 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 13.7, 19.1, 30.7, 64.1, 121.8, 127.0, 127.1, 127.4, 128.08, 128.14, 128.2, 130.97, 131.04, 131.1, 137.0, 139.3, 141.4, 142.0, 146.2, 149.5, 167.6; MS *m/z* 382 (M⁺). Anal. Calcd for C₂₇H₂₆O₂: C, 84.78, H, 6.85. Found: C, 84.66, H, 6.81.

Butyl (2E)-4,5,5-tri(4-methylphenyl)-2,4-pentadienoate (6b).^[7a] Mp 99-102 °C; ¹H NMR (400 MHz, CDCl₃) δ 0.89 (t, *J* = 7.3 Hz, 3H), 1.31 (m, 2H), 1.55 (m, 2H), 2.21 (s, 3H), 2.30 (s, 3H), 2.38 (s, 3H), 4.06 (t, *J* = 6.9 Hz, 2H), 5.59 (d, *J* = 15.6 Hz, 1H), 6.77 (d, *J* = 8.3 Hz, 2H), 6.84 (d, *J* = 8.0 Hz, 2H), 6.98 (d, *J* = 8.3 Hz, 2H), 7.02 (d, *J* = 8.0 Hz, 2H), 7.09 (d, *J* = 8.0 Hz, 2H), 7.16 (d, *J* = 8.0 Hz, 2H), 7.71 (d, *J* = 15.6 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 13.7, 19.1, 21.2, 21.2, 21.3, 30.7, 64.0, 120.8, 128.1, 128.7,

128.9, 130.95, 131.04, 131.1, 136.2, 136.4, 136.6, 136.8, 138.0, 138.9, 139.3, 147.0, 149.4, 167.8; MS m/z 424 (M^+). Anal. Calcd for $C_{30}H_{32}O_2$: C, 84.87, H, 7.60. Found: C, 84.74, H, 7.55

Butyl (2*E*)-4,5,5-tri(4-chlorophenyl)-2,4-pentadienoate (6c).^[7a] Mp 150-152°C; 1H NMR (400 MHz, $CDCl_3$) δ 0.90 (t, $J = 7.3$ Hz, 3H), 1.33 (m, 2H), 1.57 (m, 2H), 4.09 (t, $J = 6.6$ Hz, 2H), 5.61 (d, $J = 15.4$ Hz, 1H), 6.79 (d, $J = 8.4$ Hz, 2H), 7.02 (d, $J = 8.8$ Hz, 2H), 7.06 (d, $J = 8.4$ Hz, 2H), 7.13 (d, $J = 8.8$ Hz, 2H), 7.23 (d, $J = 8.8$ Hz, 2H), 7.37 (d, $J = 8.8$ Hz, 2H), 7.62 (d, $J = 15.4$ Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.7, 19.1, 30.6, 64.4, 122.9, 128.1, 128.6, 128.8, 132.2, 132.3 (overlapped), 133.5, 133.6, 134.8, 136.6, 137.3, 139.1, 139.6, 145.0, 147.0, 167.1; MS m/z 484, 486, 488 (M^+). Anal. Calcd for $C_{27}H_{23}O_2Cl_3$: C, 66.75, H, 4.77, Cl, 21.89. Found: C, 66.69, H, 4.61, Cl, 21.77.

Butyl (2*E*)-4,5,5-tri(2-thienyl)-2,4-pentadienoate (6d).^[7a] Oil: 1H NMR (400 MHz, $CDCl_3$) δ 0.90 (t, $J = 7.3$ Hz, 3H), 1.34 (m, 2H), 1.58 (m, 2H), 4.08 (t, $J = 6.6$ Hz, 2H), 5.74 (d, $J = 15.4$ Hz, 1H), 6.78 (dd, $J = 1.1, 3.7$ Hz, 1H), 6.83 (dd, $J = 3.7, 5.1$ Hz, 1H), 6.97 (dd, $J = 1.1, 3.3$ Hz, 1H), 7.08 (dd, $J = 3.3, 5.1$ Hz, 1H), 7.12 (dd, $J = 3.7, 5.1$ Hz, 1H), 7.18 (dd, $J = 1.1, 3.7$ Hz, 1H), 7.27 (dd, $J = 1.1, 5.1$ Hz, 1H), 7.44 (dd, $J = 1.1, 5.1$ Hz, 1H), 7.49 (dd, $J = 1.1, 5.1$ Hz, 1H), 7.78 (d, $J = 15.4$ Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.7, 19.13, 30.7, 64.2, 122.0, 126.1, 126.9, 127.5, 127.7, 128.0, 129.6, 129.70, 129.73, 130.7, 132.0, 136.6, 138.9, 141.5, 143.9, 145.8, 167.3; MS m/z 400 (M^+). Anal. Calcd for $C_{21}H_{20}O_2S_3$: C, 62.97, H, 5.03, S, 24.01. Found: C, 62.94, H, 4.92, S, 23.85.

Ethyl (2*E*)-4,5,5-triphenyl-2,4-pentadienoate (6e).^[7a] Mp 181-182 °C; 1H NMR (400 MHz, $CDCl_3$) δ 1.21 (t, $J = 7.3$ Hz, 3H), 4.13 (q, $J = 7.3$ Hz, 2H), 5.62 (d, $J = 15.4$ Hz, 1H), 6.87-6.90 (m, 2H), 7.02-7.05 (m, 3H), 7.09-7.12 (m, 2H), 7.17-7.20 (m, 2H), 7.21-7.25 (m, 3H), 7.26-7.39 (m, 3H), 7.73 (d, $J = 15.4$ Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.2, 60.2, 121.8, 127.0, 127.1, 127.4, 128.10, 128.13, 128.2, 131.0, 131.06, 131.10, 137.0, 139.4, 141.3, 142.0, 146.2, 149.6, 167.5; MS m/z 354 (M^+). Anal. Calcd for $C_{25}H_{22}O_2$: C, 84.72, H, 6.26. Found: C, 84.61, H, 6.14.

(2*E*)-*N,N*-Dimethyl-4,5,5-triphenyl-2,4-pentadienamide (6f).^[7a] Mp 178-179 °C; 1H NMR (400 MHz, $CDCl_3$) δ 2.85 (s, 3H), 2.94 (s, 3H), 6.04 (d, $J = 15.0$ Hz, 1H), 6.87-6.89 (m, 2H), 7.01-7.04 (m, 3H), 7.12-7.19 (m, 2H), 7.17-7.19 (m, 2H), 7.21-7.24 (m,

3H), 7.31-7.38 (m, 3H), 7.67 (d, $J = 15.0$ Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 35.6, 37.2, 121.7, 126.8, 126.8, 127.4, 127.97, 127.99, 128.1, 130.9, 131.0, 131.2, 137.4, 140.1, 141.2, 142.3, 143.7, 148.1, 167.3; MS m/z 353 (M^+). Anal. Calcd for $\text{C}_{25}\text{H}_{23}\text{ON}$: C, 84.95, H, 6.56, N, 3.96. Found: C, 84.81, H, 6.38, N, 3.94.

(2E)-4,5,5-Triphenyl-2,4-pentadienenitrile (6g).^[7a] Mp 136-138 °C; ^1H NMR (400 MHz, CDCl_3) δ 5.08 (d, $J = 16.5$ Hz, 1H), 6.86-6.88 (m, 2H), 7.02-8.07 (m, 3H), 7.08-7.11 (m, 2H), 7.20-7.25 (m, 5H), 7.39-7.44 (m, 4H); ^{13}C NMR (100 MHz, CDCl_3) δ 99.2, 119.0, 127.55, 127.57, 127.60, 128.4, 128.5, 128.7, 130.9 (overlapped), 131.1, 136.4, 137.8, 140.8, 141.2, 150.6, 151.8; MS m/z 307 (M^+). Anal. Calcd for $\text{C}_{23}\text{H}_{17}\text{N}$: C, 89.87, H, 5.57, N, 4.56. Found: C, 89.64, H, 5.57, N, 4.58.

(2E)-1,1,2,4-Tetraphenyl-1,3-butadiene (6h). Mp 145-146 °C (lit.^[17] mp 144-145 °C); ^1H NMR (400 MHz, CDCl_3) δ 6.26 (d, $J = 16.1$ Hz, 1H), 6.89-6.91 (m, 2H), 7.00-7.03 (m, 3H), 7.15-7.23 (m, 12H), 7.30-7.40 (m, 4H); ^{13}C NMR (100 MHz, CDCl_3) δ 126.2, 126.5, 126.7, 127.28, 127.31, 127.4, 127.9, 128.0, 128.5, 131.1, 131.2, 131.3, 131.5, 132.6, 137.8, 138.9, 140.5, 142.5, 142.8, 142.9; MS m/z 358 (M^+).

(2E)-4-(4-Methylphenyl)-1,1,2-triphenyl-1,3-butadiene (6i). Mp 133-134 °C (lit.^[18] 131-132 °C); ^1H NMR (400 MHz, CDCl_3) δ 2.29 (s, 3H), 6.23 (d, $J = 16.12$ Hz, 1H), 6.88-6.91 (m, 2H), 6.99-7.05 (m, 5H), 7.09-7.11 (m, 2H), 7.18-7.22 (m, 5H), 7.30-7.39 (m, 6H); ^{13}C NMR (100 MHz, CDCl_3) δ 21.2, 126.1, 126.4, 126.6, 127.25, 127.28, 127.8, 127.9, 129.2, 130.3, 131.1, 131.3, 131.5, 132.6, 135.0, 137.2, 139.1, 140.6, 142.2, 142.6, 143.0; MS m/z 372 (M^+).

(2E)-4-(4-Chlorophenyl)-1,1,2-triphenyl-1,3-butadiene (6j).^[7a] Mp 167-169 °C; ^1H NMR (400 MHz, CDCl_3) δ 6.20 (d, $J = 16.1$ Hz, 1H), 6.88-6.90 (m, 2H), 7.00-7.03 (m, 3H), 7.10-7.12 (m, 2H), 7.17-7.23 (m, 8H), 7.29-7.31 (m, 2H), 7.34-7.40 (m, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 126.3, 126.8, 127.3, 127.5, 127.6, 127.9, 128.0, 128.7, 131.1, 131.2 (overlapped), 131.4, 131.7, 132.8, 136.3, 138.7, 140.3, 142.4, 142.7, 143.3; MS m/z 392, 394 (M^+). Anal. Calcd for $\text{C}_{28}\text{H}_{21}\text{Cl}$: C, 85.59, H, 5.39, Cl, 9.02. Found: C, 85.53, H, 5.47, Cl, 8.95.

Butyl (2E,4E,6E)-4,5,7-triphenyl-2,4,6-heptatrienoate (8a). Mp 136-138 °C; ^1H NMR (400 MHz, CDCl_3) δ 0.86 (t, $J = 7.33$ Hz, 3H), 1.24-1.30 (m, 2H), 1.47-1.55 (m,

2H), 4.01 (t, $J = 6.59$ Hz, 2H), 5.41 (d, $J = 15.39$ Hz, 1H), 6.19 (d, $J = 15.75$ Hz, 1H), 6.91 (d, $J = 15.75$ Hz, 1H), 7.09-7.21 (m, 5H), 7.28-7.32 (m, 5H), 7.40-7.51 (m, 6H); ^{13}C NMR (100 MHz, CDCl_3) δ 13.7, 19.1, 30.6, 64.0, 121.0, 126.9, 127.7, 127.9, 128.1, 128.3, 128.53, 128.54, 129.7, 130.4, 130.5, 136.0, 137.0, 137.70, 137.73, 138.8, 145.1, 147.1, 167.3; MS m/z 408 (M^+); HRMS (EI) m/z 408.2085 (408.2089 calcd for $\text{C}_{29}\text{H}_{28}\text{O}_2$). Anal. Calcd for $\text{C}_{29}\text{H}_{28}\text{O}_2$: C, 85.26, H, 6.91. Found: C, 85.01, H, 6.97.

Butyl (2E,4E,6E)-7-(4-methylphenyl)-4,5-Diphenyl-2,4,6-heptatrienoate (8b). Mp 110-112 °C; ^1H NMR (400 MHz, CDCl_3) δ 0.86 (t, $J = 7.25$ Hz, 3H), 1.24-1.29 (m, 2H), 1.47-1.54 (m, 2H), 2.27 (s, 3H), 4.00 (t, $J = 6.52$ Hz, 2H), 5.40 (d, $J = 15.58$ Hz, 1H), 6.17 (d, $J = 15.95$ Hz, 1H), 6.86 (d, $J = 15.95$ Hz, 1H), 7.00 (s, 4H), 7.25-7.32 (m, 4H), 7.41-7.50 (m, 7H); ^{13}C NMR (100 MHz, CDCl_3) δ 13.7, 19.1, 21.2, 30.6, 64.0, 120.7, 126.8, 127.6, 127.9, 128.3, 128.5, 128.8, 129.3, 130.4, 130.5, 134.3, 136.1, 137.80, 137.83, 138.2, 138.3, 145.2, 147.4, 167.4; MS m/z 422 (M^+); HRMS (EI) m/z 422.2249 (422.2246 calcd for $\text{C}_{30}\text{H}_{30}\text{O}_2$). Anal. Calcd for $\text{C}_{30}\text{H}_{30}\text{O}_2$: C, 85.27, H, 7.16. Found: C, 85.10, H, 7.00.

Butyl (2E,4E,6E)-7-(4-methoxyphenyl)-4,5-Diphenyl-2,4,6-heptatrienoate (8c). Mp 108-108.5 °C; ^1H NMR (400 MHz, CDCl_3) δ 0.86 (t, $J = 7.25$ Hz, 3H), 1.22-1.31 (m, 2H), 1.47-1.54 (m, 2H), 3.74 (s, 3H), 4.00 (t, $J = 6.52$, 2H), 5.38 (d, $J = 15.58$ Hz, 1H), 6.14 (d, $J = 15.95$, 1H), 6.72-6.74 (m, 2H), 6.78 (d, $J = 15.95$ Hz, 1H), 7.04-7.06 (m, 2H), 7.27-7.32 (m, 4H), 7.40-7.50 (m, 7H); ^{13}C NMR (100 MHz, CDCl_3) δ 13.7, 19.1, 30.6, 55.2, 64.0, 114.0, 120.4, 127.6, 127.7, 127.8, 128.2, 128.3, 128.5, 129.9, 130.4, 130.5, 135.7, 137.85 (overlapped), 137.92, 145.2, 147.5, 159.7, 167.4; MS m/z 438 (M^+); HRMS (EI) m/z 438.2191 (438.2195 calcd for $\text{C}_{30}\text{H}_{30}\text{O}_3$). Anal. Calcd for $\text{C}_{30}\text{H}_{30}\text{O}_3$: C, 82.16, H, 6.89. Found: C, 81.97, H, 6.77.

Butyl (2E,4E,6E)-7-(2-methoxyphenyl)-4,5-Diphenyl-2,4,6-heptatrienoate (8d). Mp 126-127 °C; ^1H NMR (400 MHz, CDCl_3) δ 0.86 (t, $J = 7.25$ Hz, 3H), 1.24-1.30 (m, 2H), 1.47-1.54 (m, 2H), 3.62 (s, 3H), 4.00 (t, $J = 6.52$ Hz, 2H), 5.40 (d, $J = 15.58$ Hz, 1H), 6.48 (d, $J = 15.95$ Hz, 1H), 6.73-6.80 (m, 2H), 7.05 (d, $J = 15.95$ Hz, 1H), 7.09-7.15 (m, 1H), 7.28-7.34 (m, 4H), 7.38-7.50 (m, 8H); ^{13}C NMR (100 MHz, CDCl_3) δ 13.7, 19.1, 30.6, 55.2, 63.9, 110.9, 120.6 (overlapped), 126.1, 127.5, 127.8, 127.9, 128.2, 128.4, 129.1, 130.5 (overlapped), 130.9, 131.6, 137.7, 138.1, 138.4, 145.3, 148.2, 157.2, 167.4; MS m/z

438 (M^+); HRMS (EI) m/z 438.2184 (438.2195 calcd for $C_{30}H_{30}O_3$). Anal. Calcd for $C_{30}H_{30}O_3$: C, 82.16, H, 6.89. Found: C, 81.89, H, 6.81.

Butyl (2*E*,4*E*,6*E*)-4,5-diphenyl-7-(3,4,5-trimethoxyphenyl)-2,4,6-heptatrienoate (8e). Mp 119.5-120 °C; 1H NMR (400 MHz, $CDCl_3$) δ 0.86 (t, J = 7.25 Hz, 3H), 1.24-1.30 (m, 2H), 1.48-1.53 (m, 2H), 3.72 (s, 6H), 3.79 (s, 3H), 4.01 (t, J = 6.52 Hz, 2H), 5.45 (d, J = 15.58, 1H), 6.11 (d, J = 15.94, 1H), 6.32 (s, 2H), 6.80 (d, J = 15.58 Hz, 1H), 7.29-7.33 (m, 4H), 7.40-7.52 (m, 7H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.6, 19.0, 30.6, 55.9, 60.9, 64.0, 103.9, 121.0, 127.6, 127.9, 128.3, 128.4, 129.5, 130.4, 130.5, 132.7, 135.5, 137.6, 137.8, 138.4, 138.7, 144.8, 146.9, 153.2, 167.3; MS m/z 498 (M^+); HRMS (EI) m/z 498.2399 (498.2406 calcd for $C_{32}H_{34}O_5$).

Butyl (2*E*,4*E*,6*E*)-4,5-diphenyl-7-(2-thienyl)-2,4,6-heptatrienoate (8f). Mp 107-109 °C; 1H NMR (270 MHz, $CDCl_3$) δ 0.86 (t, J = 7.29 Hz, 3H), 1.19-1.33 (m, 2H), 1.45-1.57 (m, 2H), 4.00 (t, J = 6.75 Hz, 2H), 5.42 (d, J = 15.39 Hz, 1H), 6.29 (d, J = 15.66 Hz, 1H), 6.71 (d, J = 15.66 Hz, 1H), 6.77-6.87 (m, 2H), 7.09 (d, J = 5.4 Hz, 1H), 7.24-7.53 (m, 11H); ^{13}C NMR (68 MHz, $CDCl_3$) δ 13.8, 19.2, 30.7, 64.0, 120.9, 125.6, 127.0, 127.7, 127.9, 128.3, 128.4 (overlapped), 128.6, 129.3, 130.3, 130.4, 137.4, 137.5, 138.5, 142.7, 144.8, 146.7, 167.2; MS m/z 414 (M^+); HRMS (EI) m/z 414.1647 (414.1654 calcd for $C_{27}H_{26}O_2S$).

Butyl (2*E*,4*E*,6*E*,8*E*)-4,5,9-triphenyl-2,4,6,8-nonatetraenoate (8g). Mp 157-158 °C; 1H NMR (400 MHz, $CDCl_3$) δ 0.85 (t, J = 7.25 Hz, 3H), 1.21-1.34 (m, 2H), 1.46-1.53 (m, 2H), 4.00 (t, J = 6.53 Hz, 2H), 5.38 (d, J = 15.59 Hz, 1H), 6.05 (dd, J = 15.22, 10.87 Hz, 1H), 6.38-6.46 (m, 2H), 6.65-6.72 (m, 1H), 7.14-7.49 (m, 16H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.7, 19.1, 30.6, 63.9, 120.8, 126.5, 127.6, 127.8, 127.9, 128.3, 128.6, 128.7, 129.2, 130.3, 130.4, 133.8, 134.9, 136.8, 137.0, 137.6, 137.8, 138.5, 145.0, 147.4, 167.3; MS m/z 434 (M^+). Anal. Calcd for $C_{31}H_{30}O_2$: C, 85.68, H, 6.96. Found: C, 85.64, H, 7.17.

Butyl (2*E*,4*E*,6*E*)-4,5-di(4-methoxyphenyl)-7-phenyl-2,4,6-heptatrienoate (8h). Mp 157-158 °C; 1H NMR (270 MHz, $CDCl_3$) δ 0.87 (t, J = 7.29 Hz, 3H), 1.25-1.36 (m, 2H), 1.48-1.58 (m, 2H), 3.89 (s, 6H), 4.02 (t, J = 6.48 Hz, 2H), 5.45 (d, J = 15.39 Hz, 1H), 6.24 (d, J = 15.93 Hz, 1H), 6.39-7.01 (m, 5H), 7.12-7.28 (m, 9H), 7.48 (d, J = 15.39 Hz, 1H); ^{13}C NMR (68 MHz, $CDCl_3$) δ 13.8, 19.2, 30.8, 55.3, 55.4, 64.0, 113.7, 113.8, 120.6,

126.8, 127.9, 128.5, 129.97, 130.04, 130.1, 131.67, 131.71, 135.6, 137.0, 138.3, 145.6, 146.8, 158.9, 159.2, 167.4; MS m/z 468 (M^+). Anal. Calcd for $C_{31}H_{32}O_4$: C, 79.46, H, 6.88. Found: C, 79.18, H, 6.90.

Butyl (2*E*,4*E*,6*E*)-4,5-di(4-chlorophenyl)-7-phenyl-2,4,6-heptatrienoate (8i). Mp 176-177 °C; 1H NMR (270 MHz, $CDCl_3$) δ 0.88 (t, $J = 7.29$ Hz, 3H), 1.25-1.36 (m, 2H), 1.48-1.56 (m, 2H), 4.03 (t, $J = 6.75$ Hz, 2H), 5.37 (d, $J = 15.66$ Hz, 1H), 6.18 (d, $J = 15.93$ Hz, 1H), 6.84 (d, $J = 15.66$ Hz, 1H), 7.11-7.48 (m, 14H); ^{13}C NMR (68 MHz, $CDCl_3$) δ 13.8, 19.2, 30.7, 64.2, 121.5, 126.9, 128.4, 128.6, 128.7, 128.77, 128.84, 131.66, 131.73, 132.2, 133.8, 135.80, 135.84, 136.4, 136.7, 137.5, 144.2, 146.0, 166.9; MS m/z 476, 478, 480 (M^+). Anal. Calcd for $C_{29}H_{26}O_2Cl_2$: C, 72.96, H, 5.49, Cl, 14.85. Found: C, 72.69, H, 5.46, Cl, 14.94.

Ethyl (2*E*,4*E*,6*E*)-4,5,7-triphenyl-2,4,6-heptatrienoate (8j). Oil: 1H NMR (400 MHz, $CDCl_3$) δ 1.17 (t, $J = 6.96$ Hz, 3H), 4.07 (q, $J = 6.96$ Hz, 2H), 5.42 (d, $J = 15.39$ Hz, 1H), 6.18 (d, $J = 15.75$ Hz, 1H), 6.90 (d, $J = 15.76$ Hz, 1H), 7.09-7.19 (m, 6H), 7.27-7.32 (m, 3H), 7.42-7.50 (m, 7H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.2, 60.1, 121.1, 126.9, 127.7, 128.0, 128.1, 128.4, 128.5 (overlapped), 129.7, 130.45, 130.48, 136.0, 137.0, 137.7, 137.8, 138.8, 145.1, 147.1, 167.3; MS m/z 380 (M^+); HRMS (EI) m/z 380.1770 (380.1776 calcd for $C_{27}H_{24}O_2$).

(2*E*,4*E*,6*E*)-*N,N*-Dimethyl-4,5,7-triphenyl-2,4,6-heptatrienamide (8k). Oil: 1H NMR (400 MHz, $CDCl_3$) δ 2.80 (s, 3H), 2.89 (s, 3H), 5.82 (d, $J = 15.02$ Hz, 1H), 6.15 (d, $J = 15.75$ Hz, 1H), 6.89 (d, $J = 15.75$ Hz, 1H), 7.08-7.18 (m, 5H), 7.29-7.48 (m, 11H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 35.5, 37.1, 121.2, 126.7, 127.6, 127.8, 127.9, 128.37, 128.40, 128.5, 130.0, 130.45, 130.53, 135.0, 137.2, 137.8, 138.5, 139.2, 142.7, 145.7, 167.1; MS m/z 379 (M^+); HRMS (EI) m/z 379.1928 (379.1936 calcd for $C_{27}H_{25}NO$).

1,2,3,4,6-Pentaphenylfulvene (10a). Mp 200-201 °C (lit.^[13] mp 202 °C); 1H NMR (400 MHz, $CDCl_3$) δ 6.78-7.05 (m, 21H), 7.24-7.29 (m, 4H), 7.42 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 125.4, 126.25, 126.30, 126.5, 127.0, 127.1, 127.2, 127.3 (overlapped), 127.6, 127.8, 130.3, 130.46, 130.52, 130.9, 131.2, 131.7, 135.30, 135.34, 135.7, 136.1, 136.7, 141.3, 141.7, 144.4, 146.0; MS m/z 458 (M^+). Anal. Calcd for $C_{36}H_{26}$: C, 94.29, H, 5.71. Found: C, 94.01, H, 5.71.

6-(4-Methylphenyl)-1,2,3,4-tetraphenylfulvene (10b). Mp 169-170 °C (lit.^[19] mp 182 °C); ¹H NMR (400 MHz, CDCl₃) δ 2.18 (s, 3H), 6.69 (d, *J* = 8.06 Hz, 2H), 6.79-6.87 (m, 11H), 6.99-7.06 (m, 6H), 7.22-7.30 (m, 5H), 7.39 (s, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 21.2, 125.3, 126.16, 126.22, 126.4, 127.1, 127.3 (overlapped), 127.7, 127.9, 130.3, 130.6, 130.7, 130.9, 131.2, 131.7, 132.8, 135.4, 135.5 (overlapped), 136.2, 136.9, 137.9, 141.4, 141.7, 143.6, 145.7; MS *m/z* 472 (M⁺); HRMS (EI) *m/z* 472.2186 (472.2191 calcd for C₃₇H₂₈).

6-(4-Methoxyphenyl)-1,2,3,4-tetraphenylfulvene (10c). Mp 201-202 °C (lit.^[20] mp 199-199.5 °C); ¹H NMR (400 MHz, CDCl₃) δ 3.69 (s, 3H), 6.41-6.43 (m, 2H), 6.79-7.05 (m, 17H), 7.22-7.28 (m, 5H), 7.36 (s, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 55.2, 112.7, 125.4, 126.1, 126.2, 126.4, 127.1, 127.2, 127.4, 127.7, 128.2, 130.4, 130.6, 131.0 (overlapped), 131.7, 132.6, 135.5, 135.6 (overlapped), 136.3, 137.1, 141.0, 141.5, 142.7, 145.5, 159.6; MS *m/z* 488 (M⁺); HRMS (EI) *m/z* 488.2133 (488.2140 calcd for C₃₇H₂₈O).

6-(2-Methoxyphenyl)-1,2,3,4-tetraphenylfulvene (10d). Mp 223-224 °C (lit.^[21] mp 210 °C); ¹H NMR (400 MHz, CDCl₃) δ 3.72 (s, 3H), 6.24-6.28 (m, 1H), 6.63-6.65 (m, 1H), 6.69-6.71 (m, 1H), 6.78-6.86 (m, 9H), 6.96-7.06 (m, 7H), 7.23-7.27 (m, 5H), 7.53 (s, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 55.3, 109.3, 119.2, 125.0, 125.3 (overlapped), 126.1, 126.2, 126.3, 127.0, 127.1, 127.3, 127.6, 129.5, 130.4, 130.6, 130.8, 131.6, 131.8, 133.0, 135.45, 135.54, 135.9, 136.8, 138.0, 141.4, 144.0, 145.6, 157.4; MS *m/z* 488 (M⁺); HRMS (EI) *m/z* 488.2127 (488.2140 calcd for C₃₇H₂₈O). Anal. Calcd for C₃₇H₂₈O: C, 90.95, H, 5.78. Found: C, 90.74, H, 5.76.

1,2,3,4-Tetraphenyl-6-(3,4,5-trimethoxyphenyl)fulvene (10e). Mp 118-120 °C; ¹H NMR (400 MHz, CDCl₃) δ 3.55 (s, 6H), 3.74 (s, 3H), 6.23 (s, 2H), 6.77-6.93 (m, 9H), 7.00-7.07 (m, 6H), 7.23-7.30 (m, 5H), 7.38 (s, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 55.7, 60.7, 108.0, 125.6, 126.3, 126.4, 126.5, 127.2, 127.26, 127.27, 127.8, 130.3, 130.5, 130.7, 130.8, 131.3, 131.7, 135.2, 135.3, 135.4, 136.3, 136.8, 138.1, 141.5, 141.7, 143.6, 146.4, 152.1; MS *m/z* 548 (M⁺); HRMS (EI) *m/z* 548.2343 (548.2351 calcd for C₃₉H₃₂O₃).

1,2,3,4-Tetraphenyl-6-(2-thienyl)fulvene (10f). Mp 213-215 °C; ¹H NMR (400 MHz, CDCl₃) δ 6.49 (m, 1H), 6.62 (dd, *J* = 5.07, 3.52 Hz, 1H), 6.81-6.84 (m, 4H), 7.01-7.11 (m, 11H), 7.18-7.31 (m, 7H); ¹³C NMR (100 MHz, CDCl₃) δ 126.16, 126.23,

126.3, 126.5, 127.09, 127.14, 127.2, 127.8, 127.9, 130.3, 130.48 (overlapped), 130.52, 131.1, 131.7, 132.4, 133.6, 135.4 (overlapped), 135.4, 136.6, 137.0, 138.9, 141.2, 142.3, 146.0; MS m/z 464 (M^+); HRMS (EI) m/z 464.1593 (464.1599 calcd for $C_{34}H_{24}S$). Anal. Calcd for $C_{34}H_{24}S$: C, 87.89, H, 5.21, S, 6.90. Found: C, 87.60, H, 5.22, S, 6.93.

1,2,3,4-Tetraphenyl-6-(2-phenylethenyl)fulvene (10g). Mp 191-194 °C (lit.^[13] mp 197 °C); 1H NMR (400 MHz, $CDCl_3$) δ 6.63 (d, J = 15.23 Hz, 1H), 6.80 (dd, J = 15.23, 11.96 Hz, 1H), 6.85-6.88 (m, 2H), 6.90-7.05 (m, 11H), 7.19-7.38 (m, 13H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 125.8, 126.2, 126.3, 126.4, 126.7, 127.1, 127.2, 127.3, 127.9, 128.1, 128.6, 128.8, 130.2, 130.34, 131.4, 131.5 (overlapped), 134.9, 135.4, 135.5, 135.6, 136.6, 138.3, 139.3, 140.8, 141.4, 144.1, 144.5; MS m/z 484 (M^+). Anal. Calcd for $C_{38}H_{28}$: C, 94.18, H, 5.82. Found: C, 93.92, H, 5.91.

6-(1-Naphthyl)-1,2,3,4-tetraphenylfulvene (10h). Mp 218-220 °C (lit.^[13] mp 217 °C); 1H NMR (270 MHz, $CDCl_3$) δ 6.55-7.09 (m, 17H), 7.27-7.35 (m, 5H), 7.42-7.50 (m, 3H), 7.71-7.75 (m, 1H), 7.83-7.86 (m, 1H), 7.90 (s, 1H); ^{13}C NMR (68 MHz, $CDCl_3$) δ 124.4, 124.8, 125.2, 125.6, 126.1, 126.2 (overlapped), 126.5, 126.7, 127.0, 127.2, 127.8, 128.0, 128.2, 129.8, 130.2, 130.3, 130.4, 131.4, 131.5, 131.8, 132.6, 132.8, 135.1, 135.2, 135.5, 136.3, 139.0 (overlapped), 141.8, 145.8, 145.9; MS m/z 508 (M^+); HRMS (EI) m/z 508.2198 (508.2191 calcd for $C_{40}H_{28}$).

1,2,3,4-Tetra(4-methoxyphenyl)-6-phenylfulvene (10i). Mp 205-206 °C; 1H NMR (270 MHz, $CDCl_3$) δ 3.66 (s, 3H), 3.70 (s, 3H), 3.71 (s, 3H), 3.80 (s, 3H), 6.39-6.42 (m, 2H), 6.56-6.61 (m, 4H), 6.70-6.92 (m, 13 H), 7.13-7.17 (m, 2H), 7.33 (s, 1H); ^{13}C NMR (68 MHz, $CDCl_3$) δ 55.02, 55.03, 55.2 (overlapped), 112.6, 112.7, 112.9 (overlapped), 113.3, 126.9, 127.2, 127.8, 127.9, 129.3, 129.9, 130.3, 131.5, 131.7, 131.8, 132.6, 134.8, 135.9, 139.7, 140.8, 144.7, 145.1, 157.4, 157.7, 157.8, 158.1; MS m/z 578 (M^+); HRMS (EI) m/z 578.2455 (578.2457 calcd for $C_{40}H_{34}O_4$).

1,2,3,4-Tetra(4-chlorophenyl)-6-phenylfulvene (10j). Mp 219-220 °C; 1H NMR (270 MHz, $CDCl_3$) δ 6.65-6.75 (m, 6H), 6.80-6.85 (m, 2H), 6.89-6.97 (m, 4H), 7.01-7.15 (m, 7H), 7.56-7.30 (m, 2H), 7.40 (s, 1H); ^{13}C NMR (68 MHz, $CDCl_3$) δ 127.2, 127.6, 127.8, 127.9, 128.2 (overlapped), 130.3, 130.6, 131.3, 131.5, 131.7, 131.8, 132.56, 132.61, 132.7, 132.9 (overlapped), 133.0, 134.5, 135.0 (overlapped), 135.5, 140.3, 142.3, 143.5,

144.4; MS m/z 594, 596, 598, 600 (M^+); HRMS (EI) m/z 594.0484 (594.0476 calcd for $C_{36}H_{22}Cl_4$).

(1E)-1-Bromo-3,4,4-triphenyl-1,3-butadiene (11). Mp 139-140 °C; 1H NMR (270 MHz, $CDCl_3$) δ 6.01 (d, J = 13.5 Hz, 1H), 6.84-6.89 (m, 2H), 6.97-7.07 (m, 3H), 7.11-7.40 (m, 11H); ^{13}C NMR (68 MHz, $CDCl_3$) δ 111.6, 126.4, 126.9, 127.3, 127.5, 128.01, 128.04, 130.7 (overlapped), 131.0, 136.7, 138.95, 139.04, 141.6, 141.8, 142.8; MS m/z 360, 362 (M^+). Anal. Calcd for $C_{22}H_{17}Br$: C, 73.14, H, 4.74, Br, 22.12. Found: C, 72.99, H, 4.74, Br, 21.98.

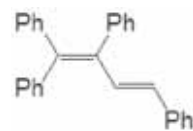
Butyl (2E,4E,6E,8E)-4,5,8,9,9-pentaphenyl-2,4,6,8-nonatetraenoate (12). Mp 187-188 °C; 1H NMR (270 MHz, $CDCl_3$) δ 0.83 (t, J = 7.29 Hz, 3H), 1.20-1.28 (m, 2H), 1.45-1.52 (m, 2H), 3.98 (t, J = 6.48 Hz, 2H), 5.38 (d, J = 15.39 Hz, 1H), 6.01 (d, J = 15.66 Hz, 1H), 6.27 (d, J = 15.66 Hz, 1H), 6.76-6.80 (m, 2H), 6.86-7.06 (m, 12H), 7.14-7.34 (m, 11H), 7.40 (d, J = 15.39 Hz, 1H); ^{13}C NMR (68 MHz, $CDCl_3$) δ 13.8, 19.2, 30.7, 63.9, 120.4, 126.2, 126.3, 127.1 (overlapped), 127.1, 127.46, 127.52 (overlapped), 127.98, 128.01, 130.1 (overlapped), 130.8, 130.9, 131.0, 133.8, 137.3, 137.5, 138.3, 138.5, 139.2, 139.5, 141.7, 142.4, 144.3, 144.9, 147.8, 167.2; MS m/z 586 (M^+). Anal. Calcd for $C_{43}H_{38}O_2$: C, 88.02, H, 6.53. Found: C, 87.68, H, 6.52.

1,2,3,4-Tetraphenyl-6-(1,2,2-triphenylethenyl)fulvene (13). Mp 238-239 °C; 1H NMR (270 MHz, $CDCl_3$) δ 5.29 (s, 1H), 6.46-6.49 (m, 2H), 6.70-6.89 (m, 14H), 6.93-7.06 (m, 9H), 7.09 (s, 5H), 7.28-7.39 (m, 5H); ^{13}C NMR (68 MHz, $CDCl_3$) δ 124.9, 125.2, 125.8, 125.9 (overlapped), 126.6, 126.7, 126.8, 126.9, 127.1, 127.2, 127.3, 127.6, 127.9, 129.5, 130.1 (overlapped), 130.3, 131.0, 131.1, 131.2, 131.6, 135.2, 135.3, 135.4, 135.7, 136.7, 137.1, 140.9, 141.2, 142.1, 142.9, 144.1, 145.1, 145.3, 146.7; MS m/z 636 (M^+); HRMS (EI) m/z 636.2808 (636.2817 calcd for $C_{50}H_{36}$).

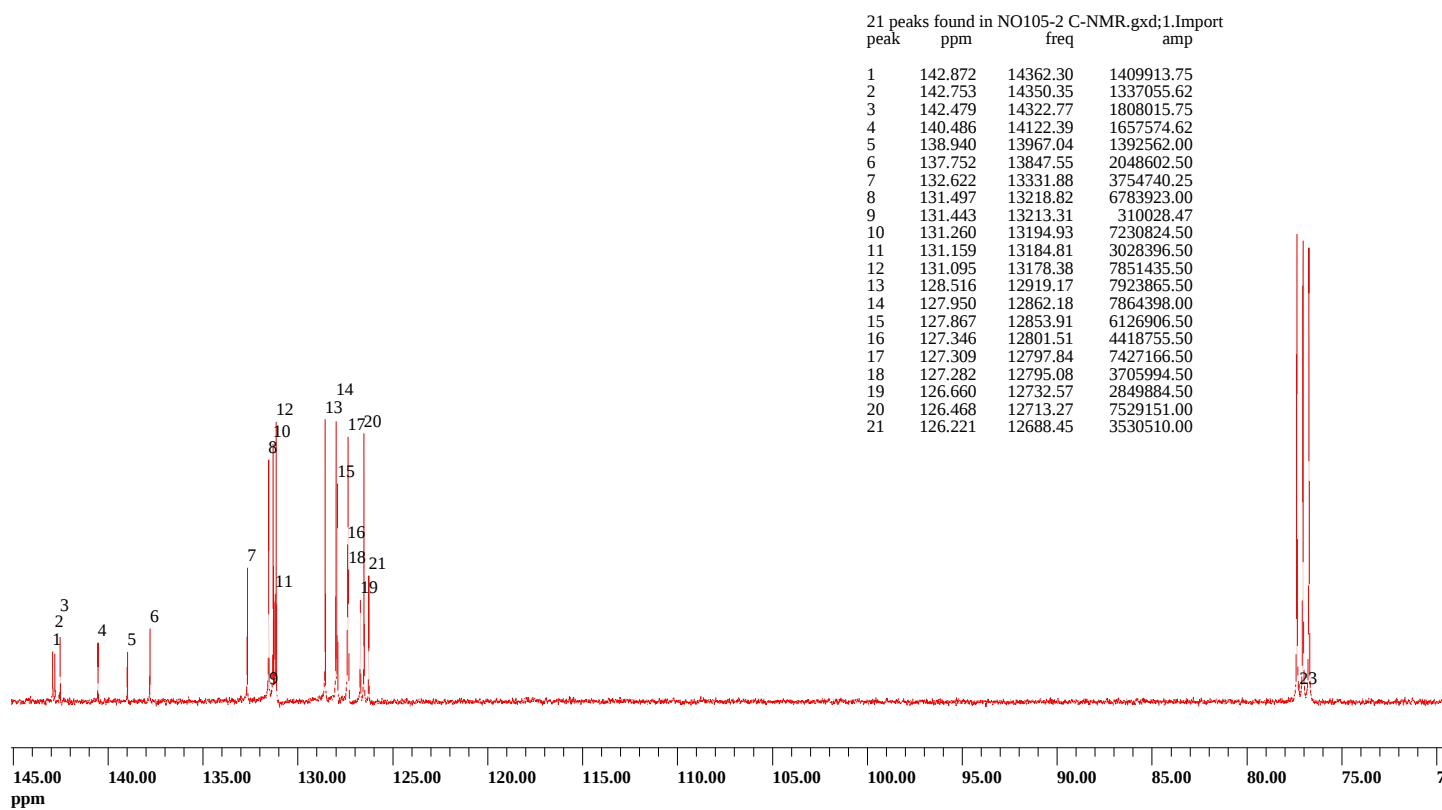
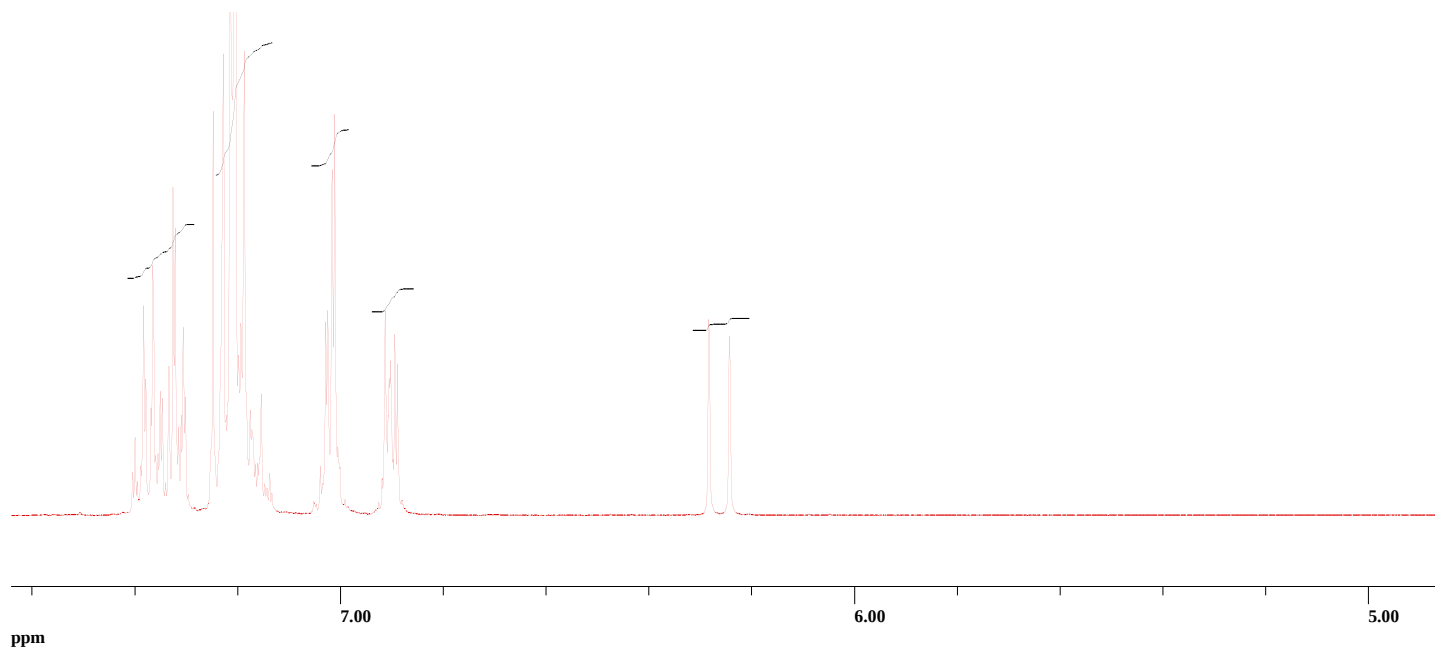
References

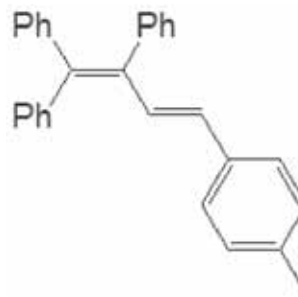
- [7] a) K. Shibata, T. Satoh, M. Miura, *Org. Lett.* **2005**, 7, 1781.
- [11] b) J. Prakash Das, S. Roy, *J. Org. Chem.* **2002**, 67, 7861.
- [13] D. Braun, R. Langendorf, *J. Prakt. Chem.* **2000**, 342, 80.
- [15] M. J. Mio, L. C. Kopel, J. B. Braun, T. L. Gadzikwa, K. L. Hull, R. G. Brisbois,

- C. J. Markworth, P. A. Grieco, *Org. Lett.* **2002**, 4, 3199.
- [16] M. S. Mohamed Ahmed, A. Sekiguchi, K. Masui, A. Mori, *Bull. Chem. Soc. Jpn.* **2005**, 78, 160.
- [17] X. Zhang, R. C. Larock, *Org. Lett.* **2003**, 5, 2993.
- [18] M. Regitz, H. Eckes, *Tetrahedron*, **1981**, 37, 1039
- [19] W. Ried, H. Knorr, H. Guercan, *Justus Liebigs Ann. Chem.* **1976**, 1415.
- [20] D. Taber, N. Picus, E. I. Becker, P. E. Spoerri, *J. Am. Chem. Soc.* **1955**, 77, 1010.
- [21] D. Hellwinkel, K. Goeke, R. Karle, *Synthesis* **1994**, 49, 973.

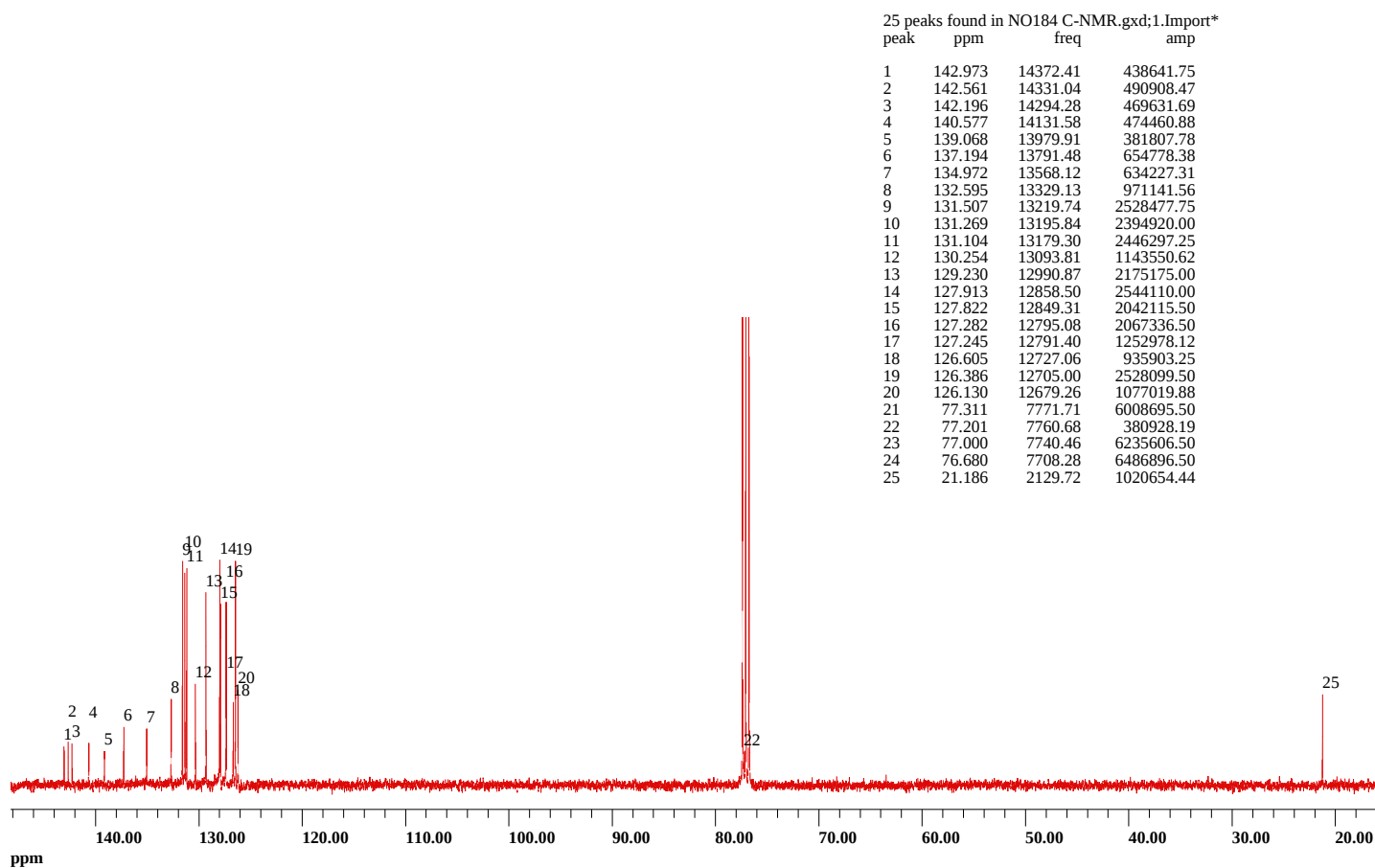
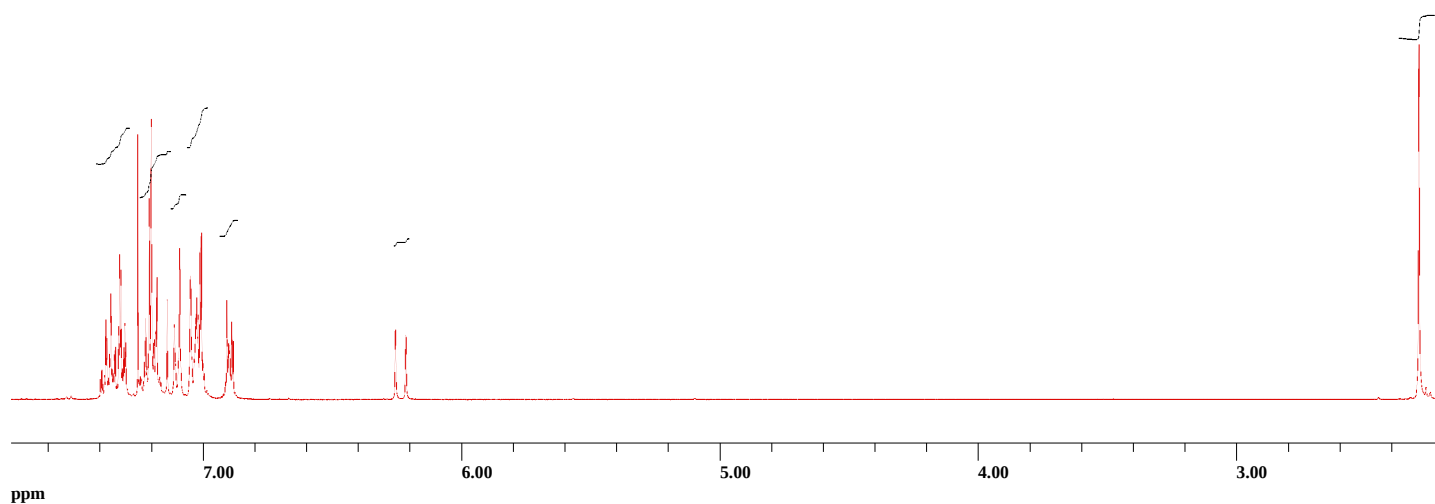


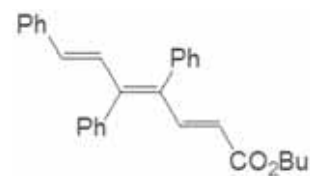
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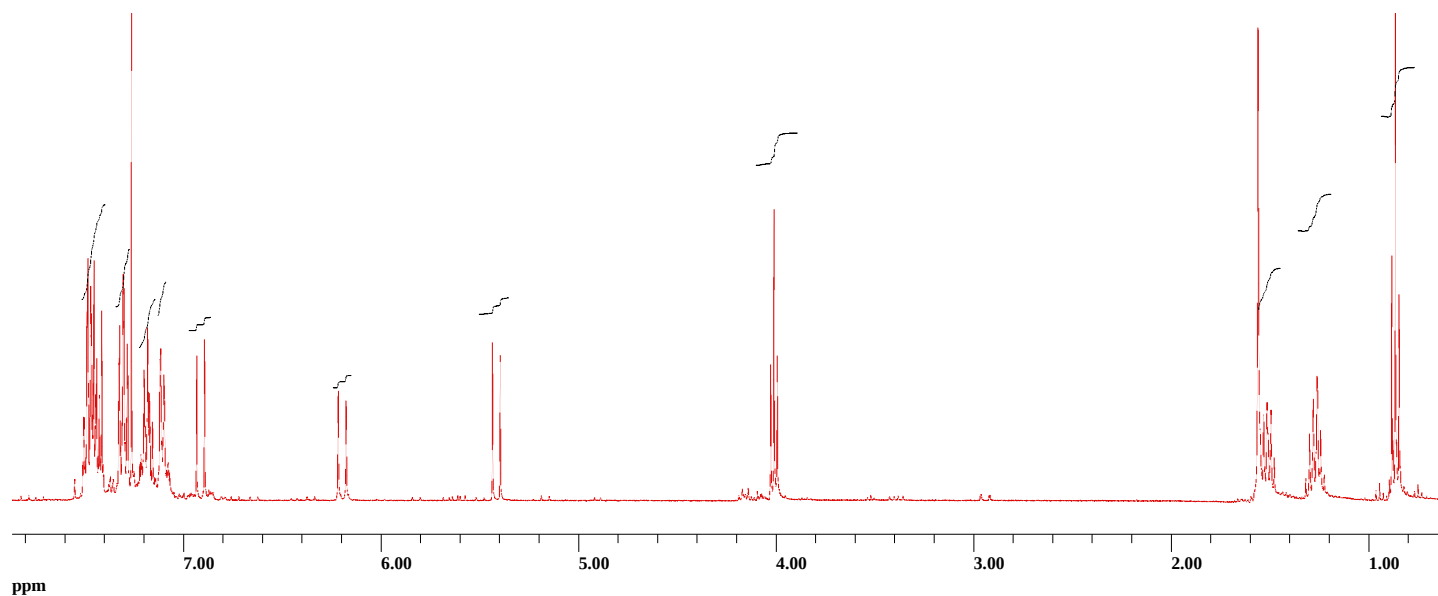


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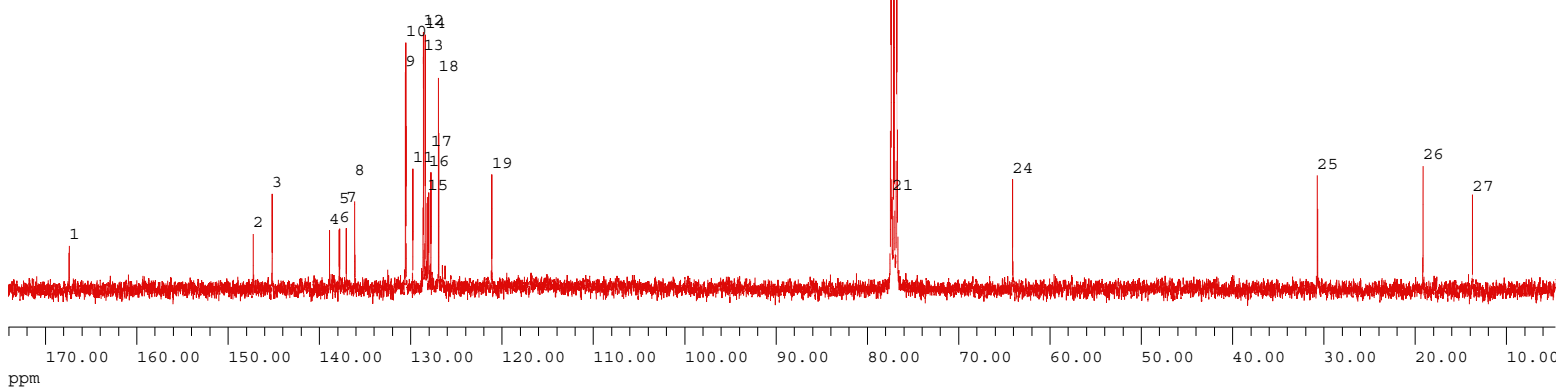


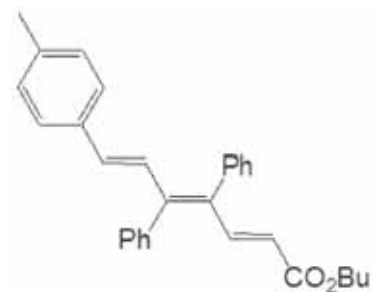
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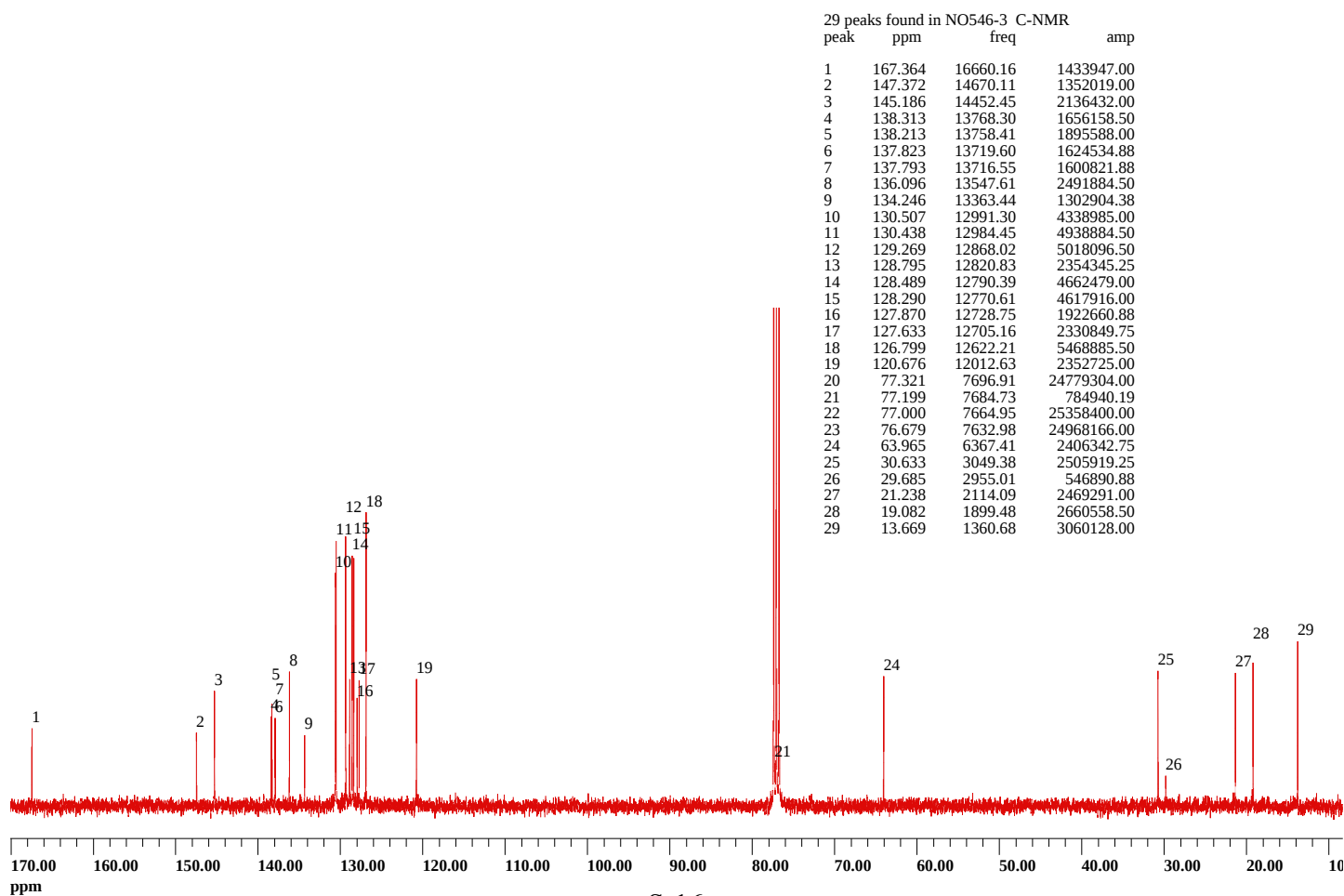
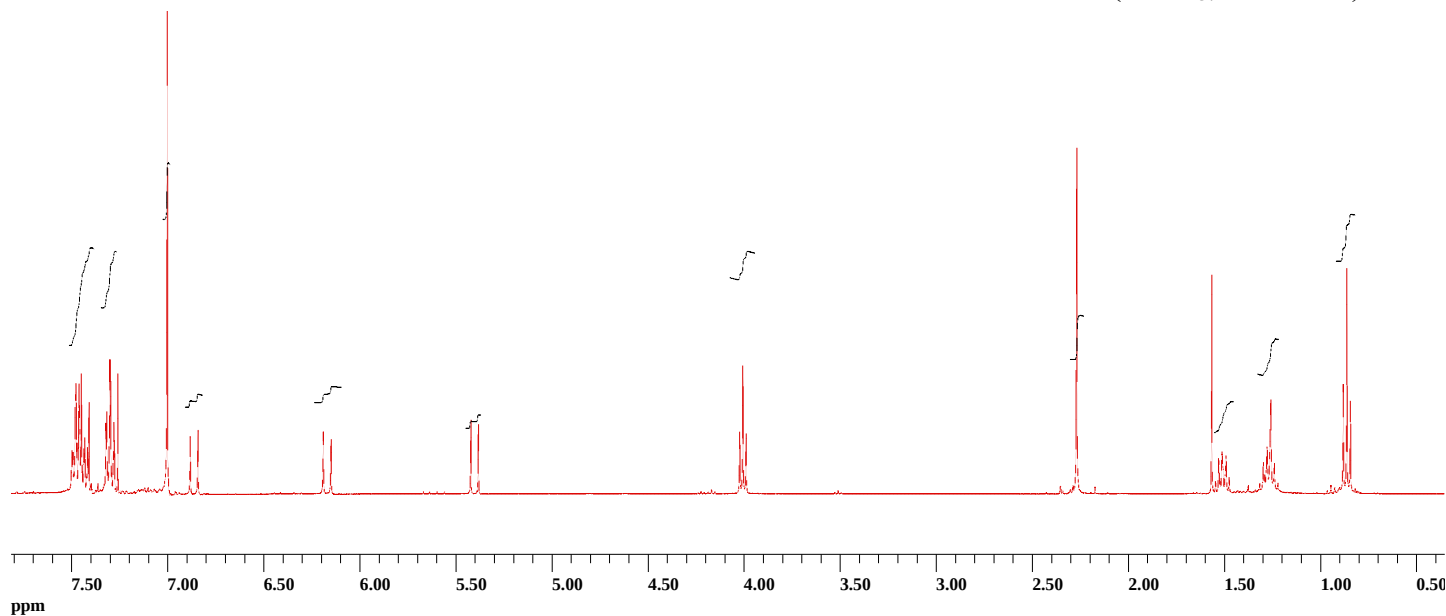
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peak ppm freq amp

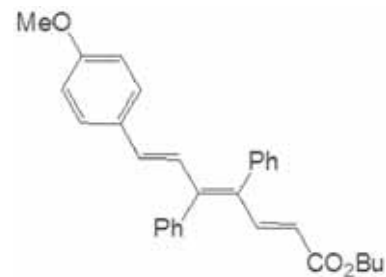
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16	127.940	12861.26	1247344.88
17	127.712	12838.28	1511989.38
18	126.861	12752.80	2744123.75
19	121.037	12167.27	1483633.38
20	77.320	7772.63	23668214.00
21	77.201	7760.68	1190271.75
22	77.000	7740.46	25151026.00
23	76.680	7708.28	24523264.00
24	63.997	6433.37	1424555.00
25	30.632	3079.25	1476581.25
26	19.092	1919.23	1592579.62
27	13.670	1374.15	1187895.75



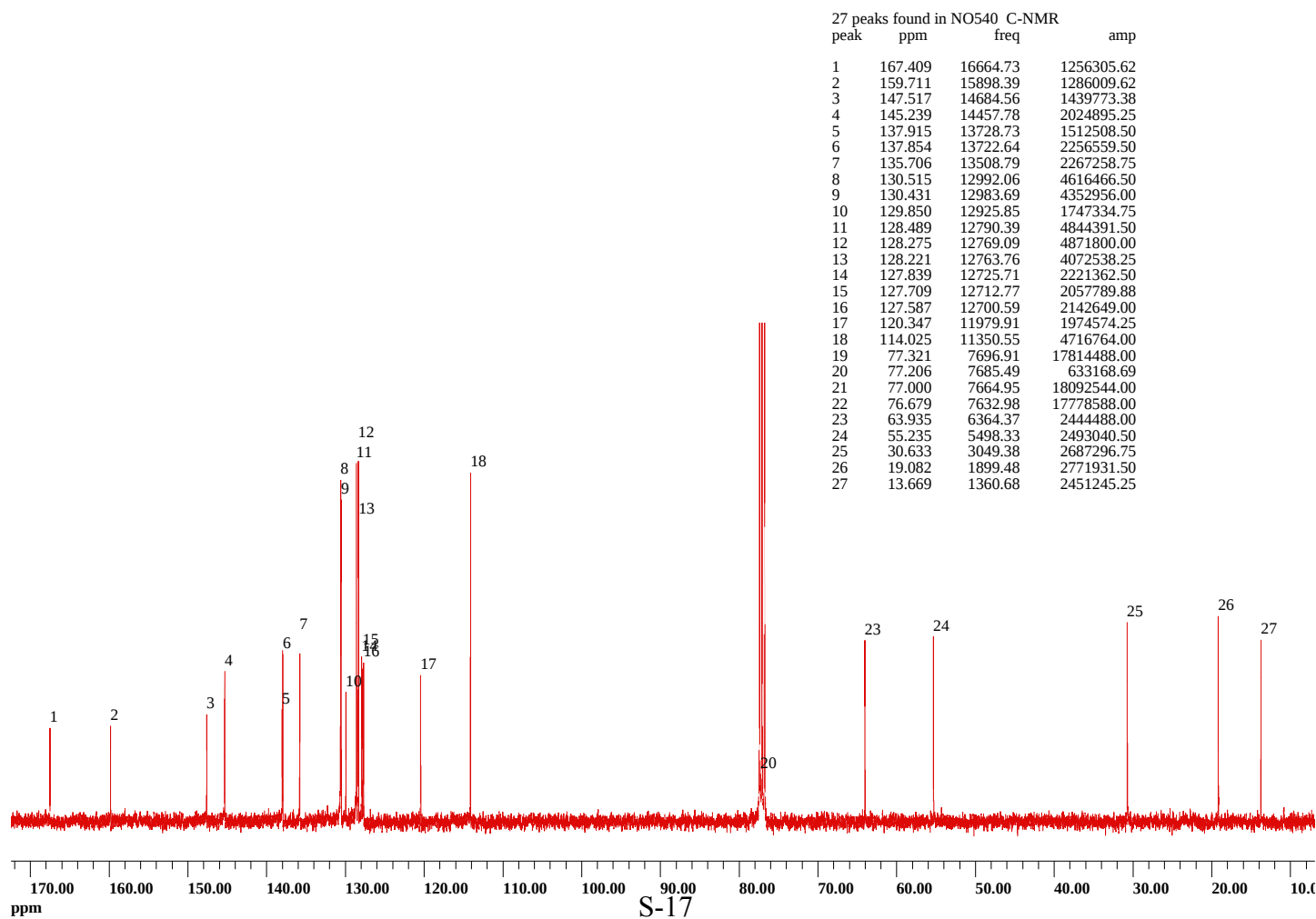
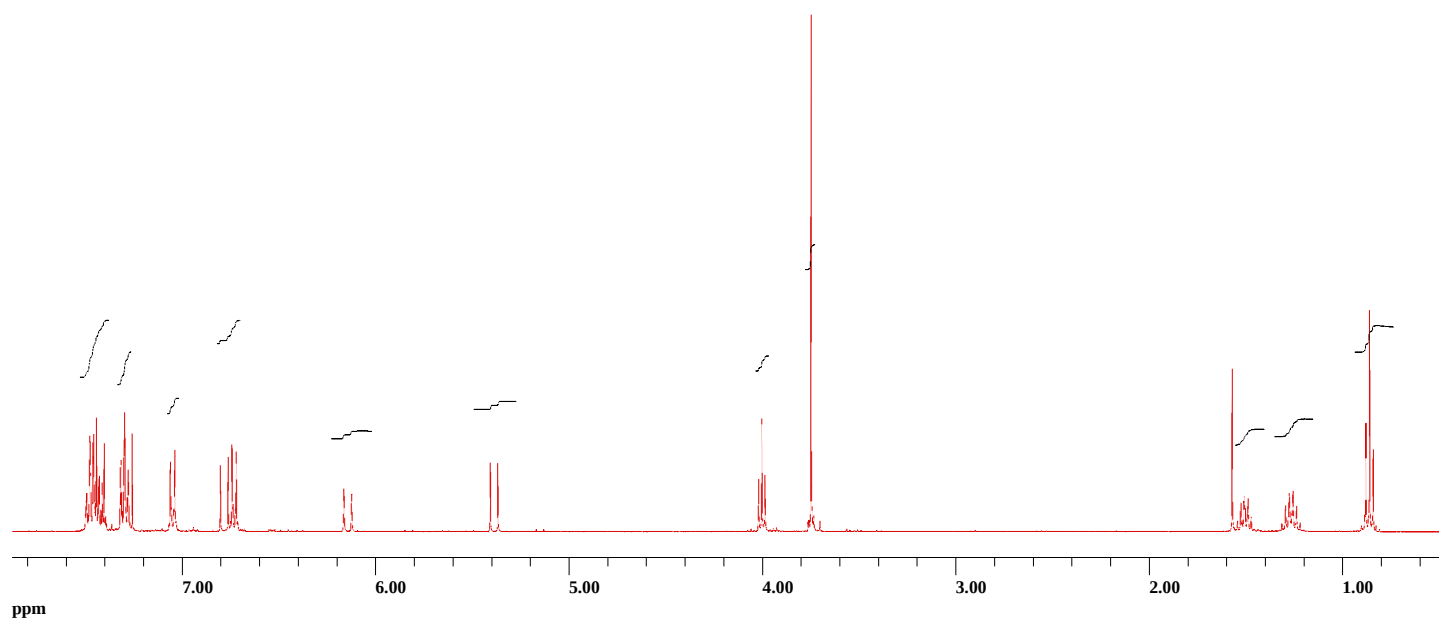


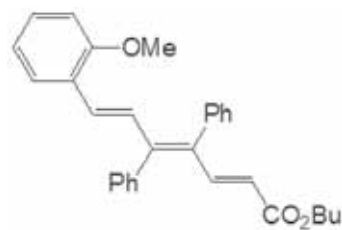
8b (CDCl₃, 400 MHz)



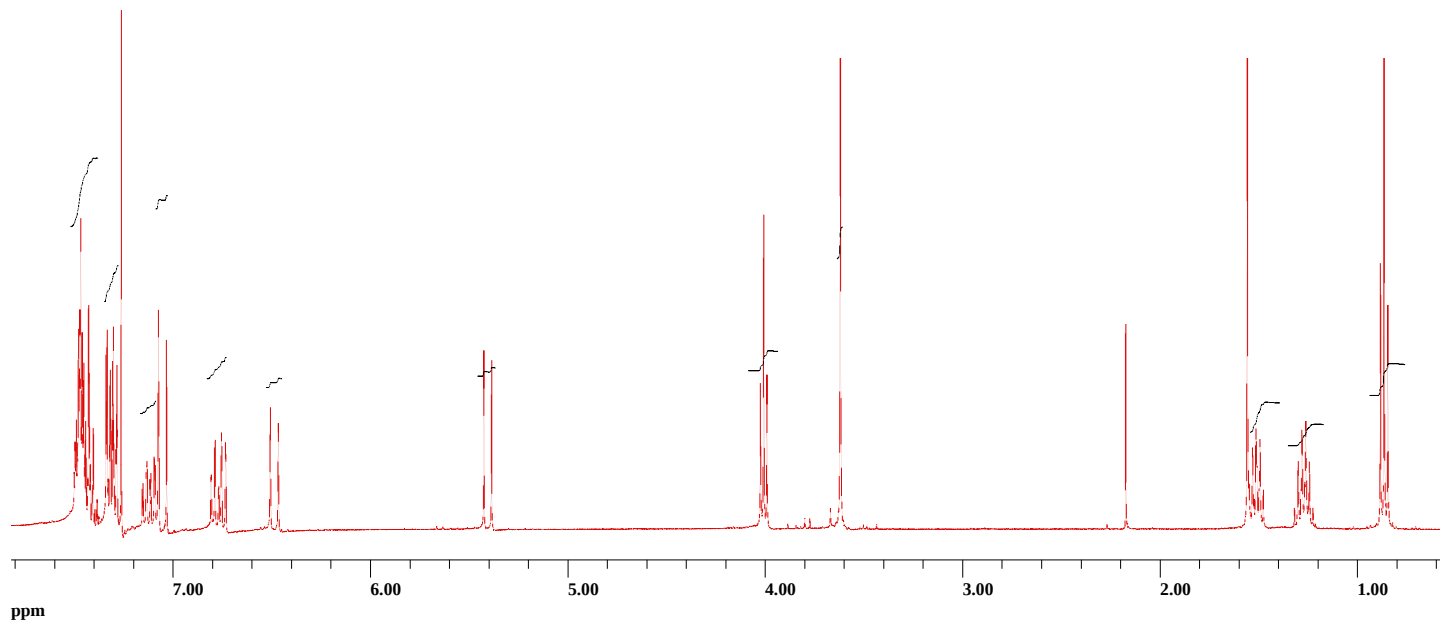


8c (CDCl₃, 400 MHz)



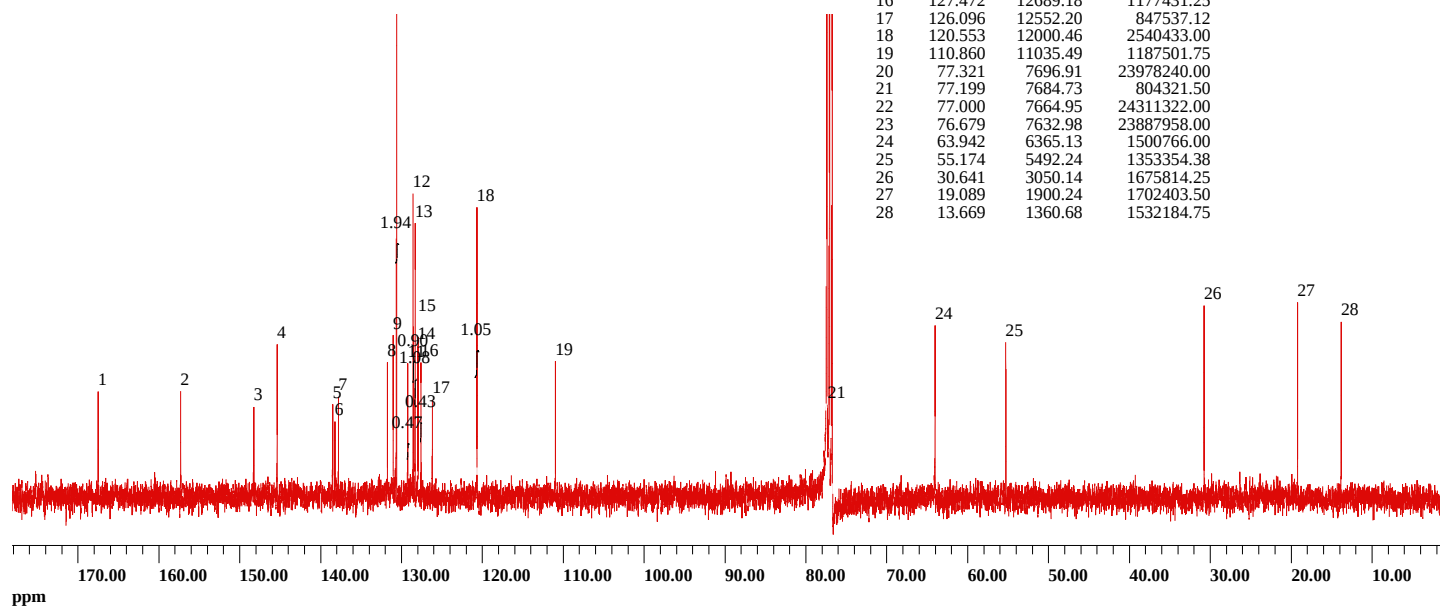


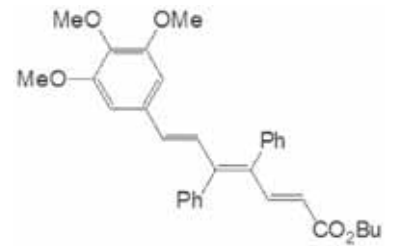
Butyl(2*E*,4*E*,6*E*)-7-(2-methoxyphenyl)-4,5-diphenyl-2,4,6-heptatrienoate (8d)
(CDCl₃, 400 MHz)



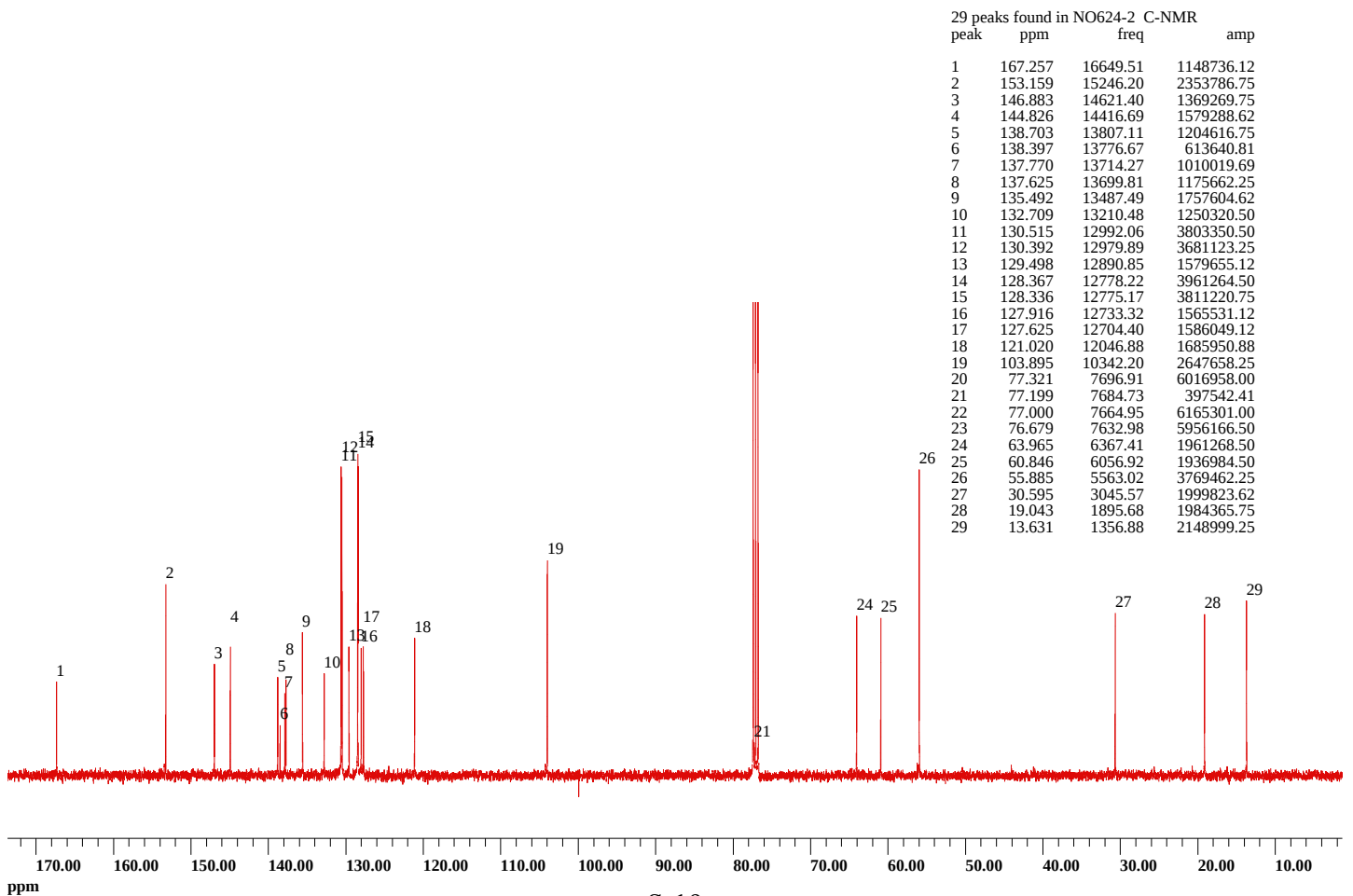
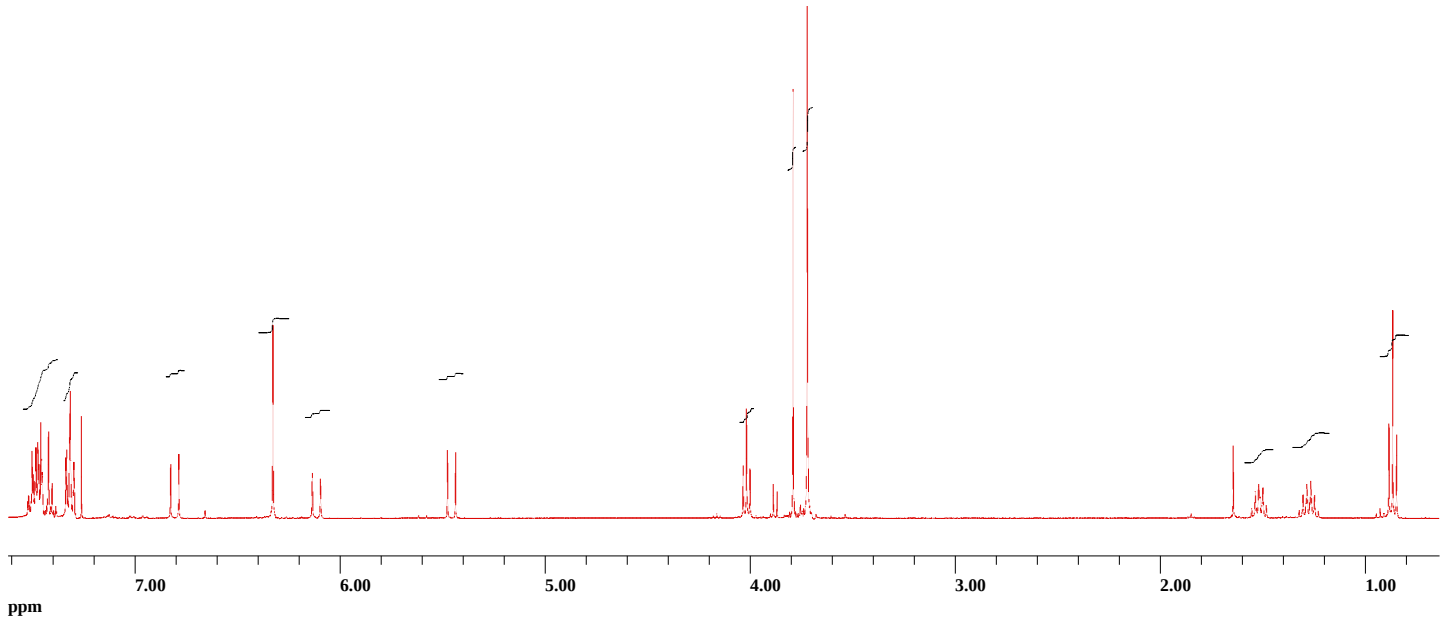
28 peaks found in NO527 C-NMR

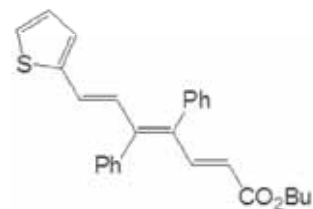
peak	ppm	freq	amp
1	167.409	16664.73	919785.62
2	157.180	15646.49	922105.69
3	148.152	14747.73	782585.12
4	145.270	14460.83	1333297.88
5	138.397	13776.67	806580.81
6	138.122	13749.27	658083.06
7	137.701	13707.42	873042.31
8	131.623	13102.41	1176877.00
9	130.905	13030.87	1415671.12
10	130.507	12991.30	4428953.50
11	129.123	12853.56	1167058.88
12	128.443	12785.83	2659793.75
13	128.214	12763.00	2400177.00
14	127.862	12727.99	1323753.75
15	127.816	12723.42	1397461.75
16	127.472	12689.18	1177431.25
17	126.096	12552.20	847537.12
18	120.553	12000.46	2540433.00
19	110.860	11035.49	1187501.75
20	77.321	7696.91	23978240.00
21	77.199	7684.73	804321.50
22	77.000	7664.95	24311322.00
23	76.679	7632.98	23887958.00
24	63.942	6365.13	1500766.00
25	55.174	5492.24	1353354.38
26	30.641	3050.14	1675814.25
27	19.089	1900.24	1702403.50
28	13.669	1360.68	1532184.75



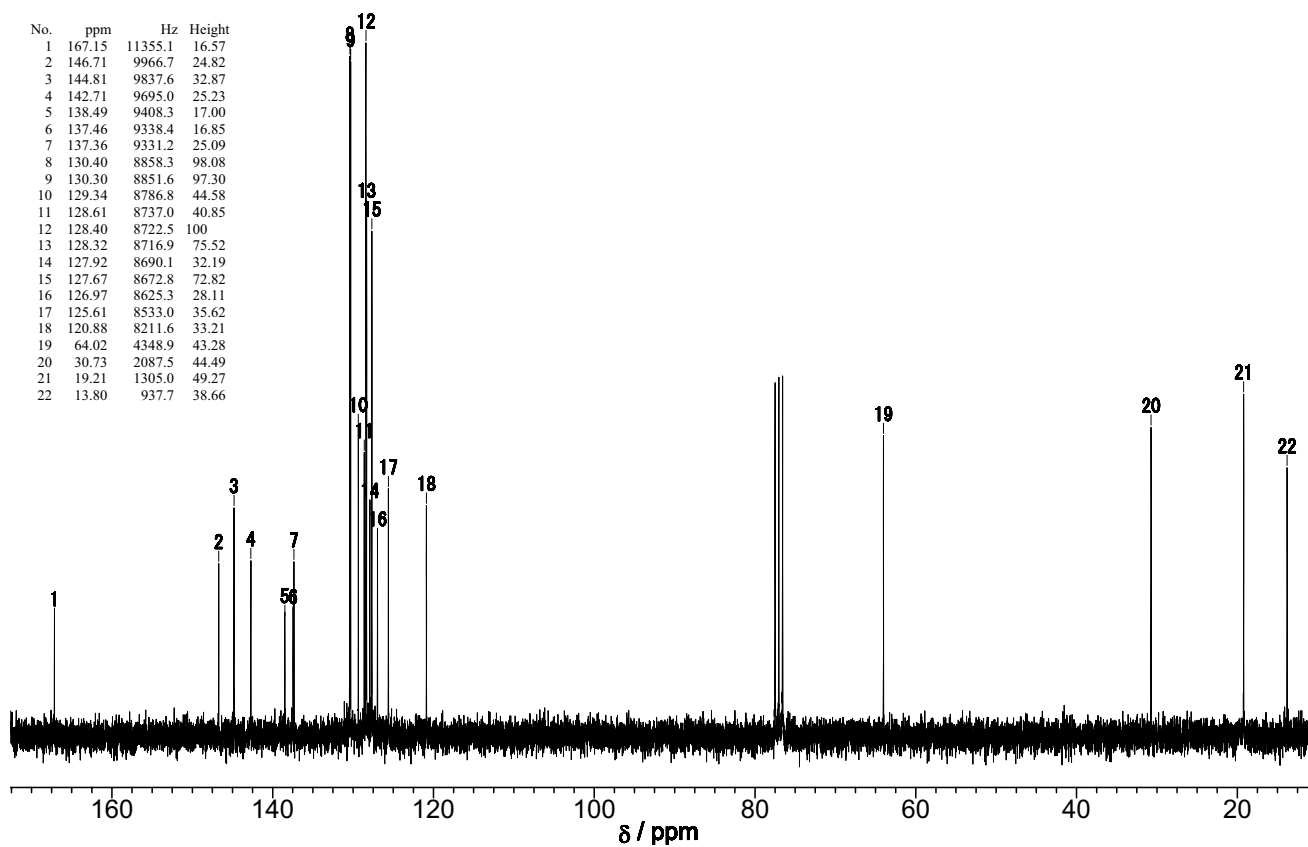
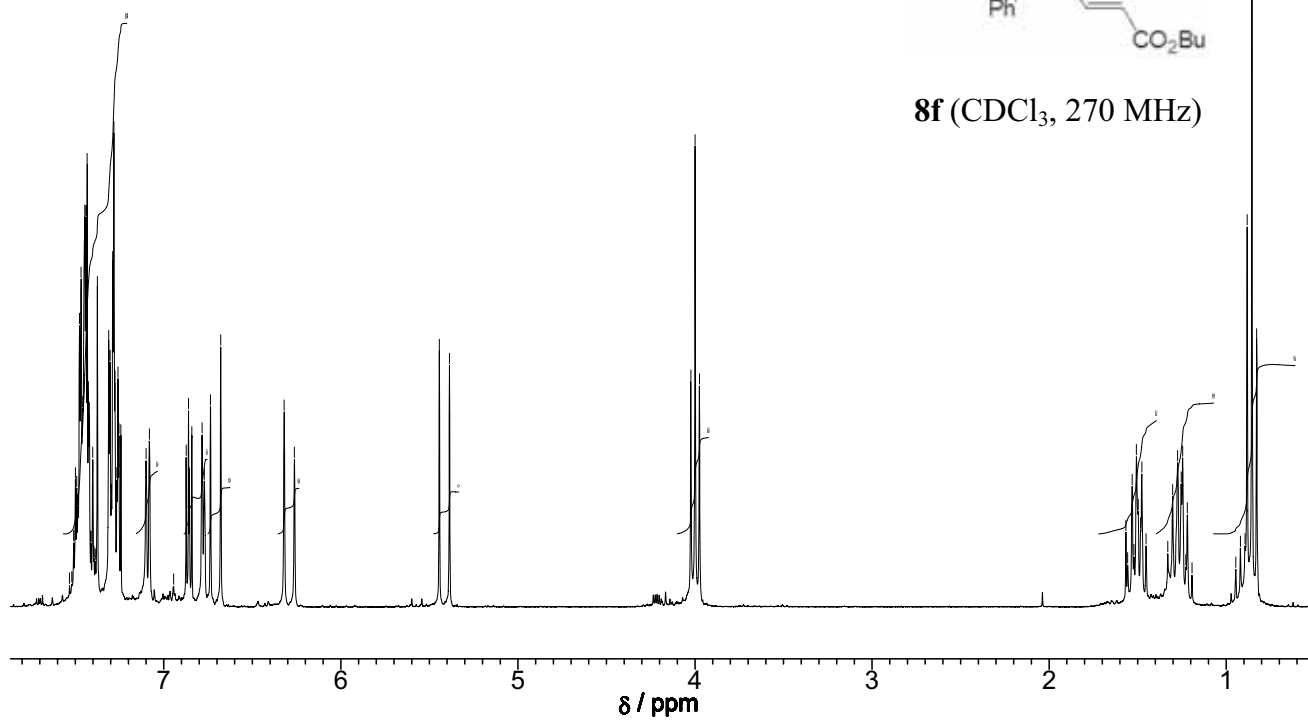


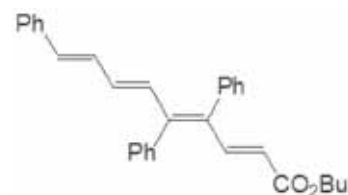
8e (CDCl₃, 400 MHz)



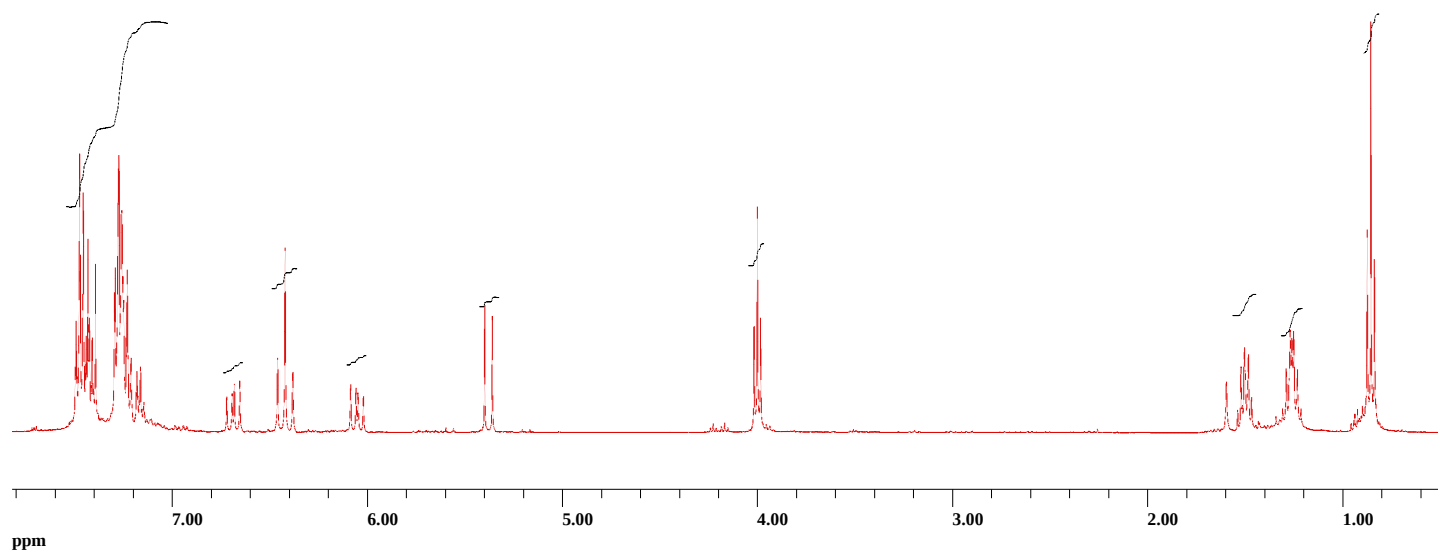


8f (CDCl₃, 270 MHz)



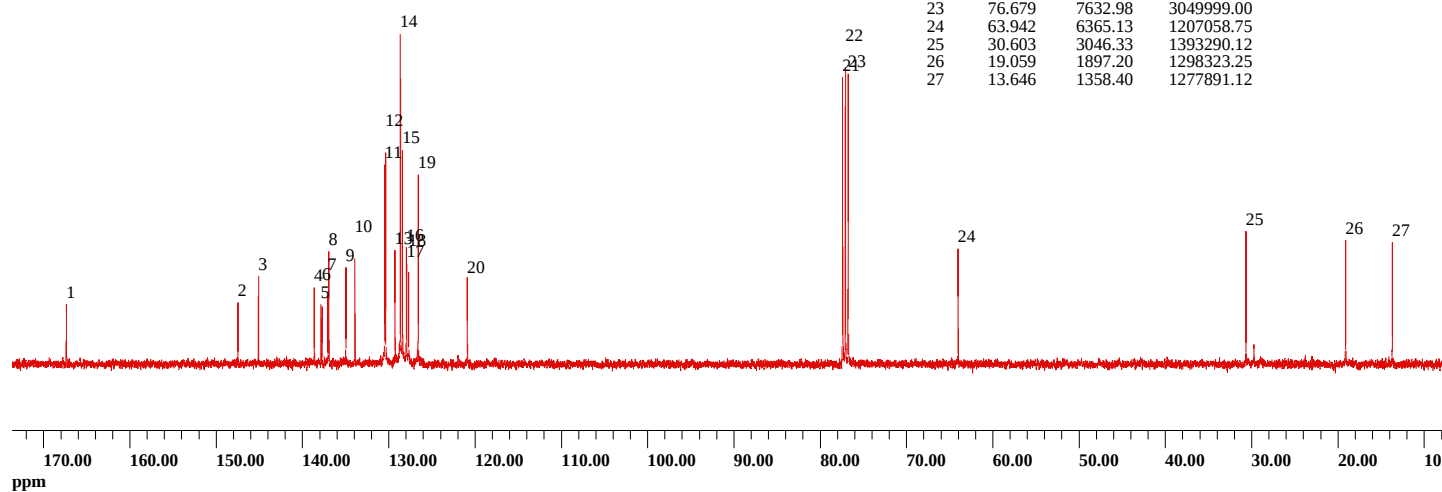


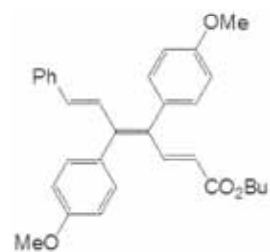
8g (CDCl₃, 400 MHz)



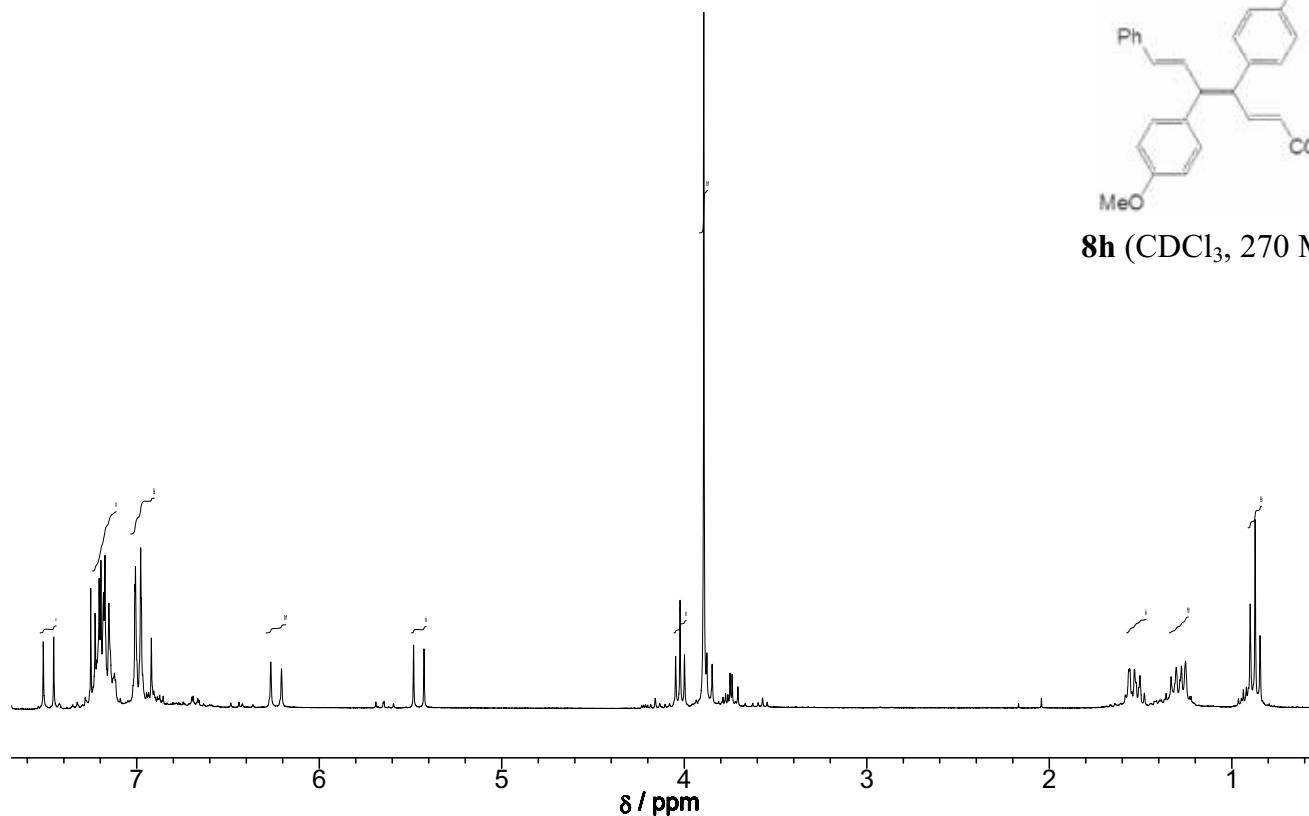
27 peaks found in NO560-6 C-NMR

peak	ppm	freq	amp
1	167.272	16651.03	624764.81
2	147.387	14671.63	643759.12
3	145.002	14434.19	920766.06
4	138.534	13790.37	799970.88
5	137.755	13712.75	622726.19
6	137.625	13699.81	598479.06
7	136.952	13632.84	714168.88
8	136.837	13621.42	1180955.38
9	134.865	13425.08	1014182.25
10	133.817	13320.82	1104198.12
11	130.354	12976.08	2092566.88
12	130.270	12967.71	2221805.00
13	129.177	12858.89	1193240.38
14	128.550	12796.48	3469428.75
15	128.313	12772.89	2249495.50
16	127.854	12727.23	1221697.25
17	127.831	12724.95	1048942.25
18	127.602	12702.12	963327.31
19	126.478	12590.25	1987648.62
20	120.798	12024.81	890262.44
21	77.321	7696.91	3013105.75
22	77.000	7664.95	3121691.75
23	76.679	7632.98	3049999.00
24	63.942	6365.13	1207058.75
25	30.603	3046.33	1393290.12
26	19.059	1897.20	1298323.25
27	13.646	1358.40	1277891.12

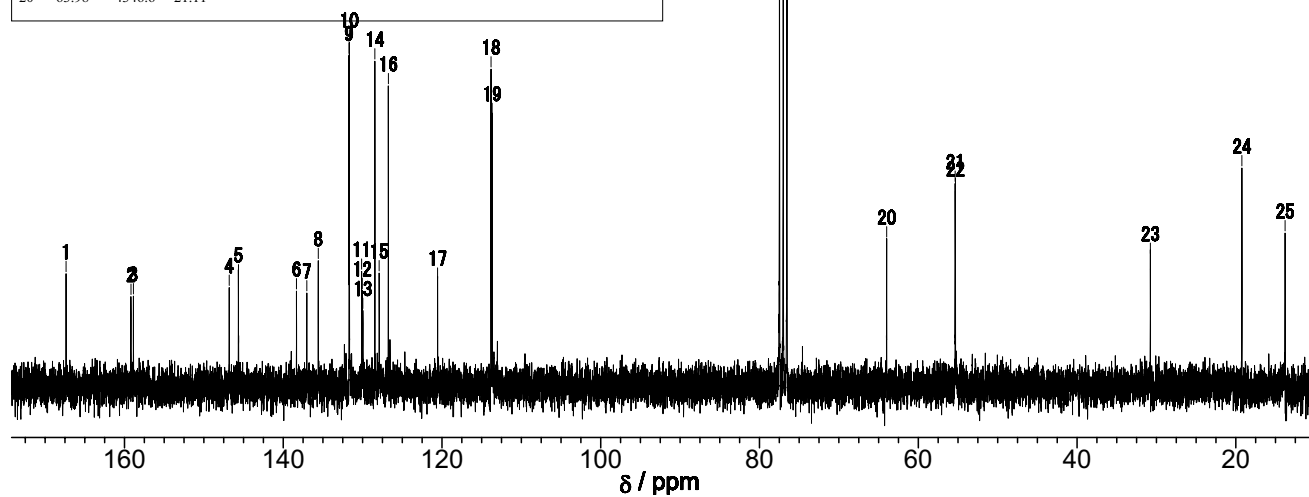


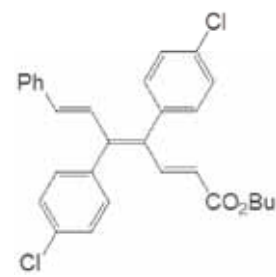


8h (CDCl₃, 270 MHz)

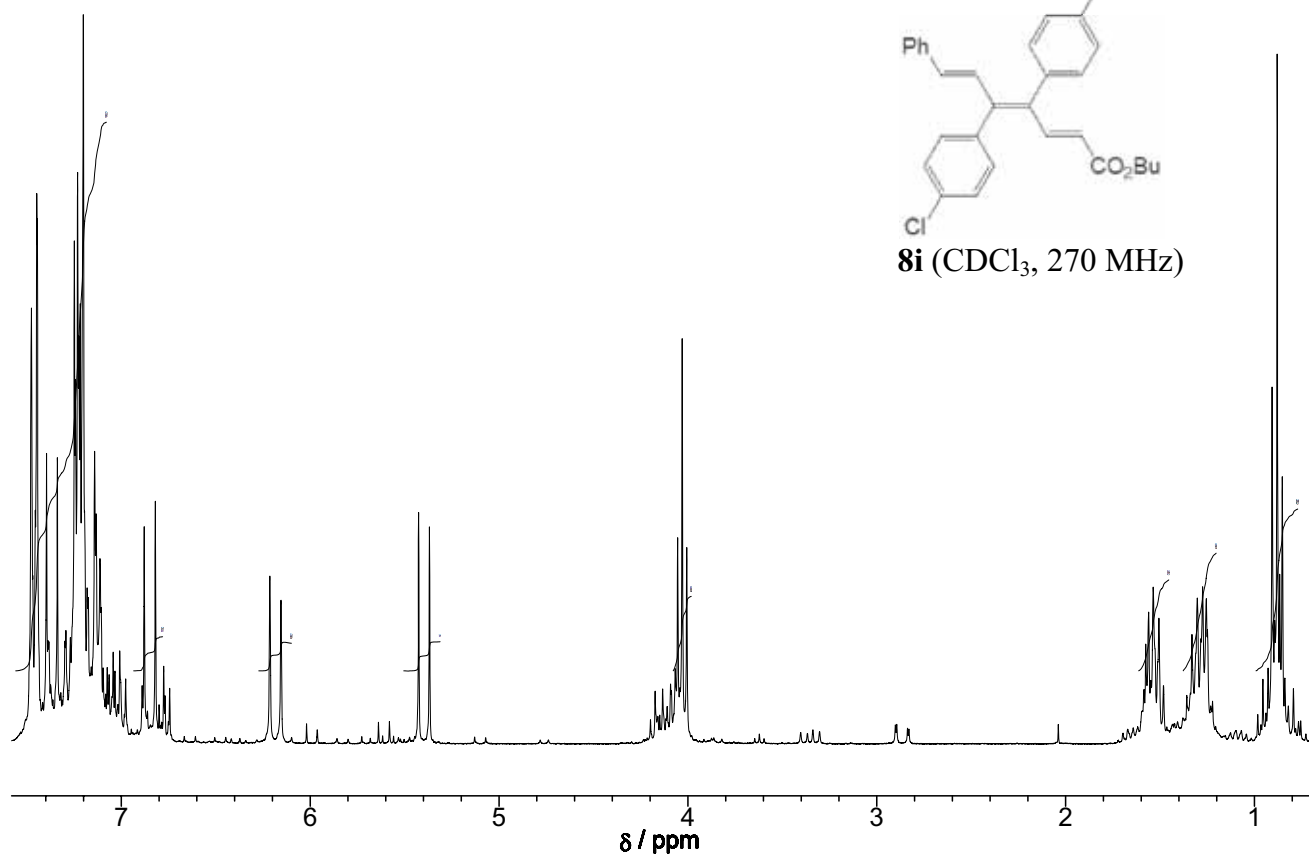


No.	ppm	Hz	Height	No.	ppm	Hz	Height
1	167.36	11369.0	16.12	21	55.35	3760.3	29.15
2	159.19	10814.0	12.82	22	55.32	3758.1	28.06
3	158.87	10792.8	12.90	23	30.77	2090.2	18.74
4	146.80	9972.8	14.13	24	19.23	1306.6	31.39
5	145.64	9894.0	15.65	25	13.80	937.2	22.00
6	138.33	9397.1	13.62				
7	137.03	9308.8	13.34				
8	135.59	9211.0	18.00				
9	131.71	8947.2	47.64				
10	131.67	8945.0	49.58				
11	130.13	8839.9	16.47				
12	130.04	8834.3	13.66				
13	129.97	8829.3	10.81				
14	128.46	8726.4	46.81				
15	127.91	8689.5	16.23				
16	126.76	8611.3	43.24				
17	120.56	8189.8	15.17				
18	113.83	7732.6	45.67				
19	113.68	7722.6	38.95				
20	63.98	4346.6	21.11				

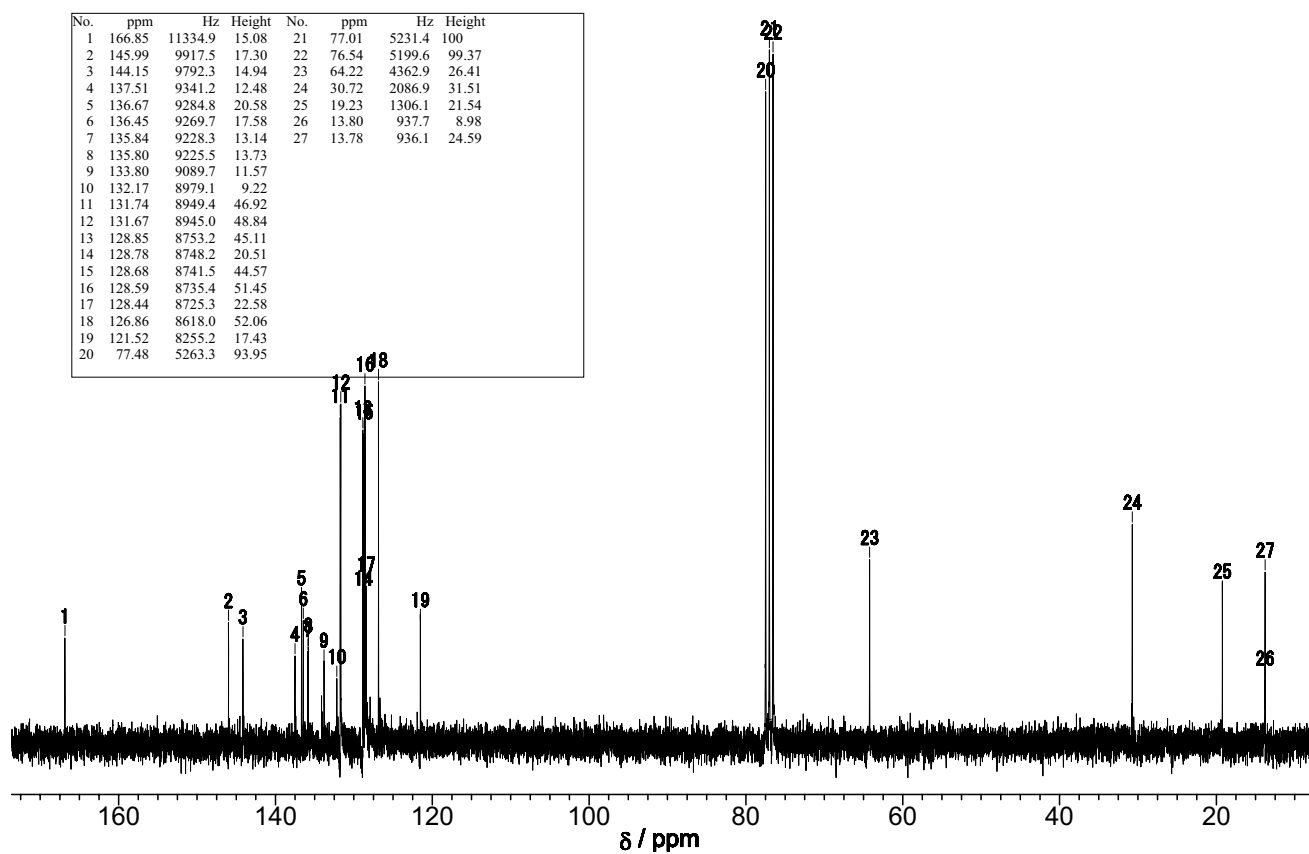


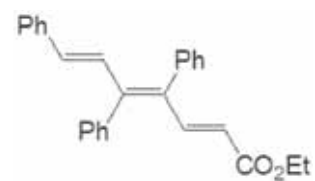


8i (CDCl₃, 270 MHz)

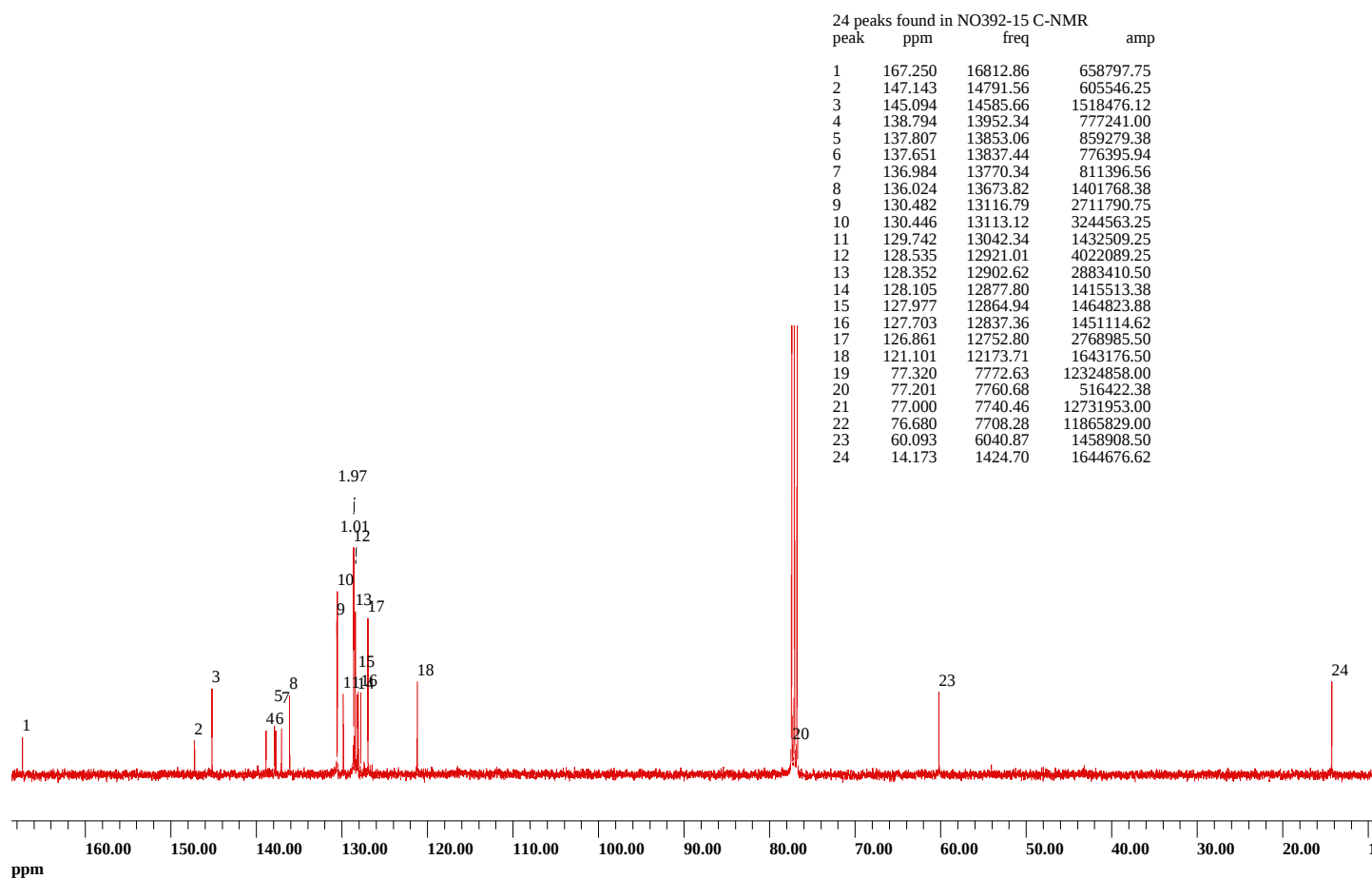
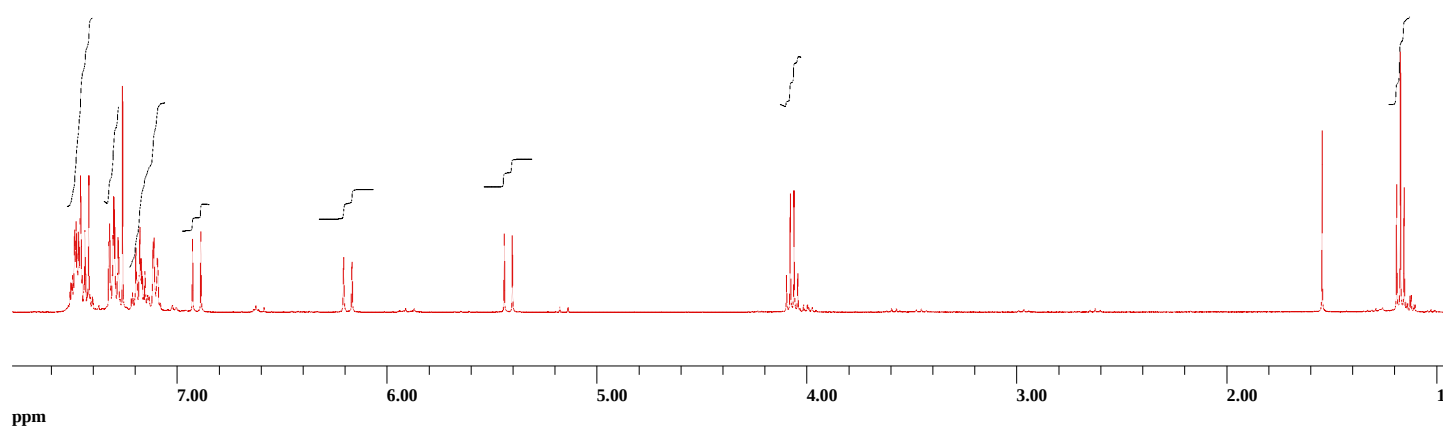


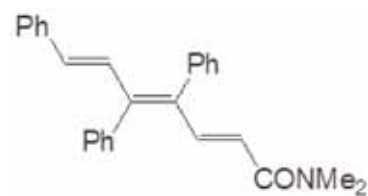
No.	ppm	Hz	Height	No.	ppm	Hz	Height
1	166.85	11334.9	15.08	21	77.01	5231.4	100
2	145.99	9917.5	17.30	22	76.54	5199.6	99.37
3	144.15	9792.3	14.94	23	64.22	4362.9	26.41
4	137.51	9341.2	12.48	24	30.72	2086.9	31.51
5	136.67	9284.8	20.58	25	19.23	1306.1	21.54
6	136.45	9269.7	17.58	26	13.80	937.7	8.98
7	135.84	9228.3	13.14	27	13.78	936.1	24.59
8	135.80	9225.5	13.73				
9	133.80	9089.7	11.57				
10	132.17	8979.1	9.22				
11	131.74	8949.4	46.92				
12	131.67	8945.0	48.84				
13	128.85	8753.2	45.11				
14	128.78	8748.2	20.51				
15	128.68	8741.5	44.57				
16	128.59	8735.4	51.45				
17	128.44	8725.3	22.58				
18	126.86	8618.0	52.06				
19	121.52	8255.2	17.43				
20	77.48	5263.3	93.95				



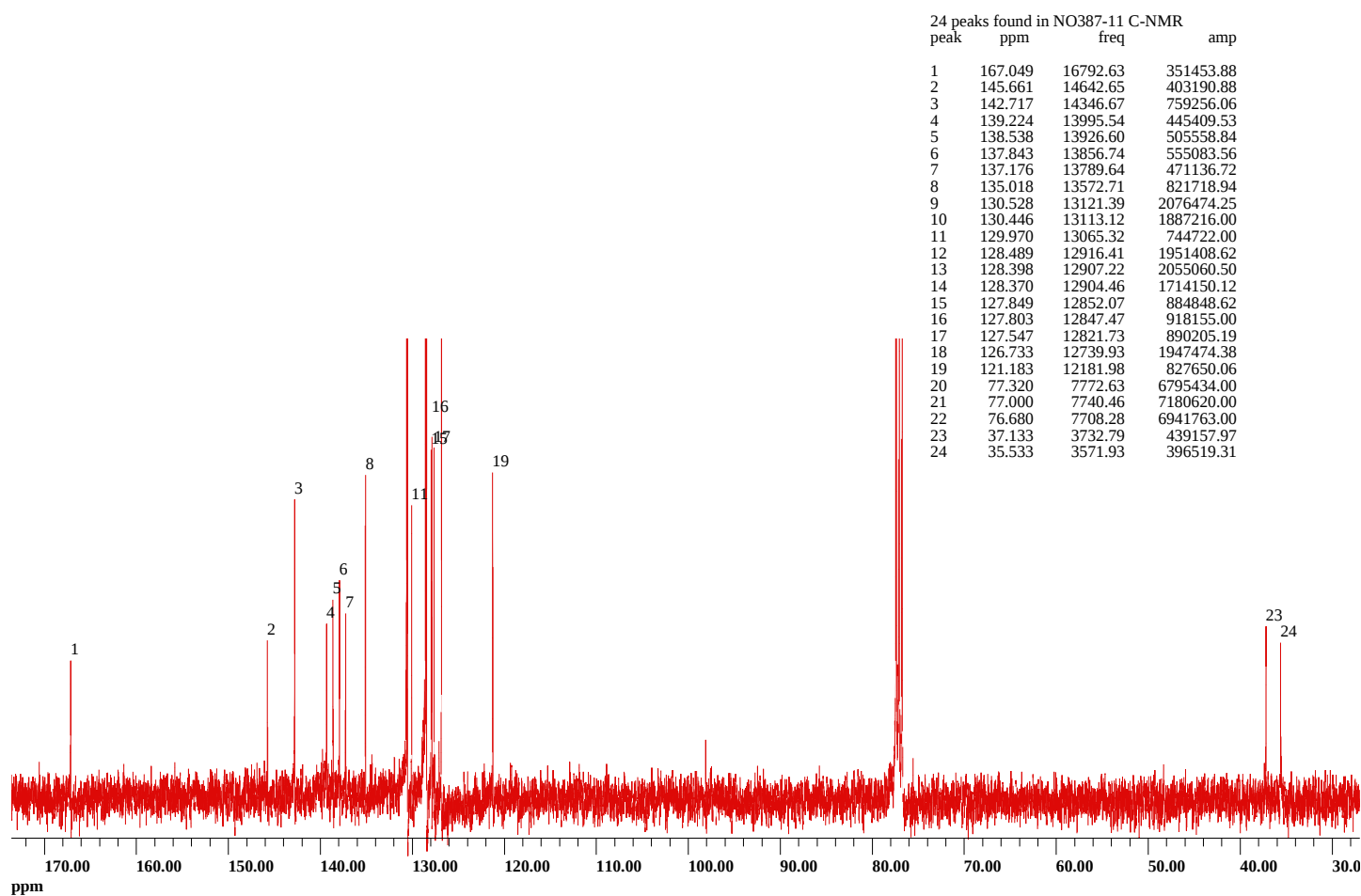
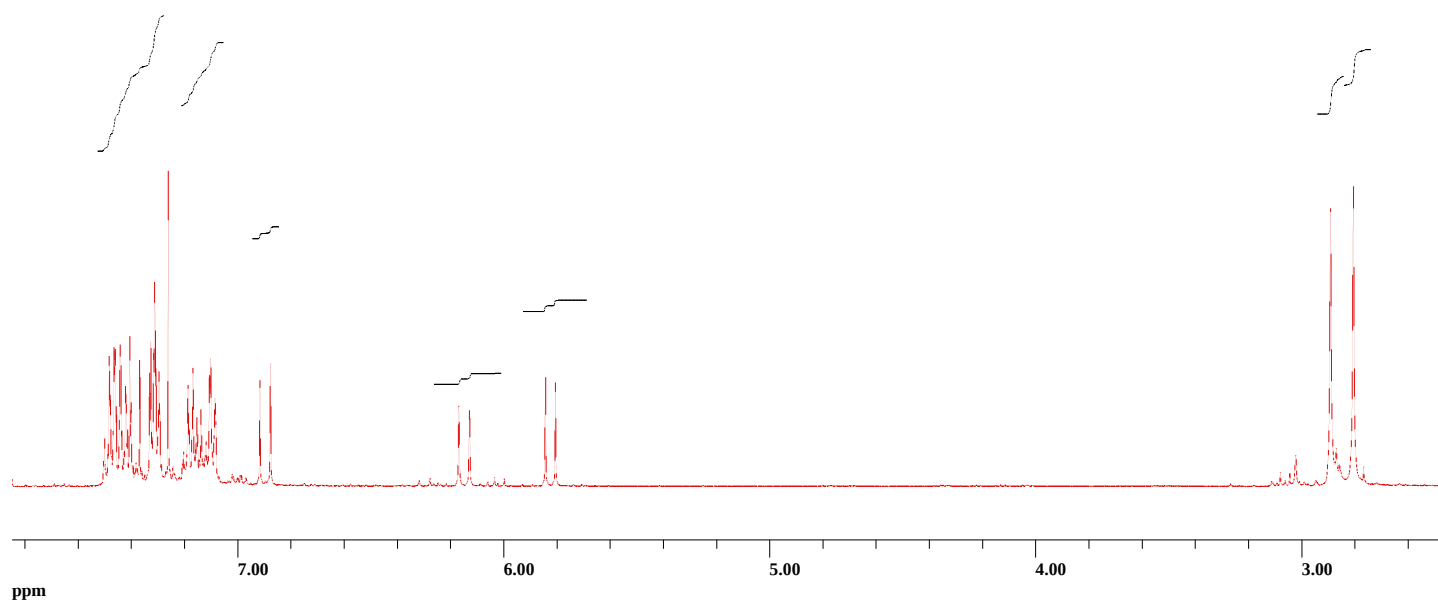


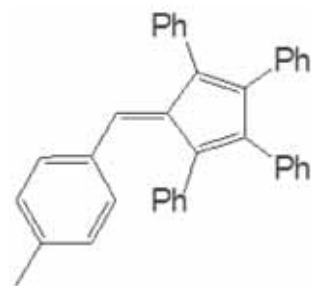
8j (CDCl₃, 400 MHz)



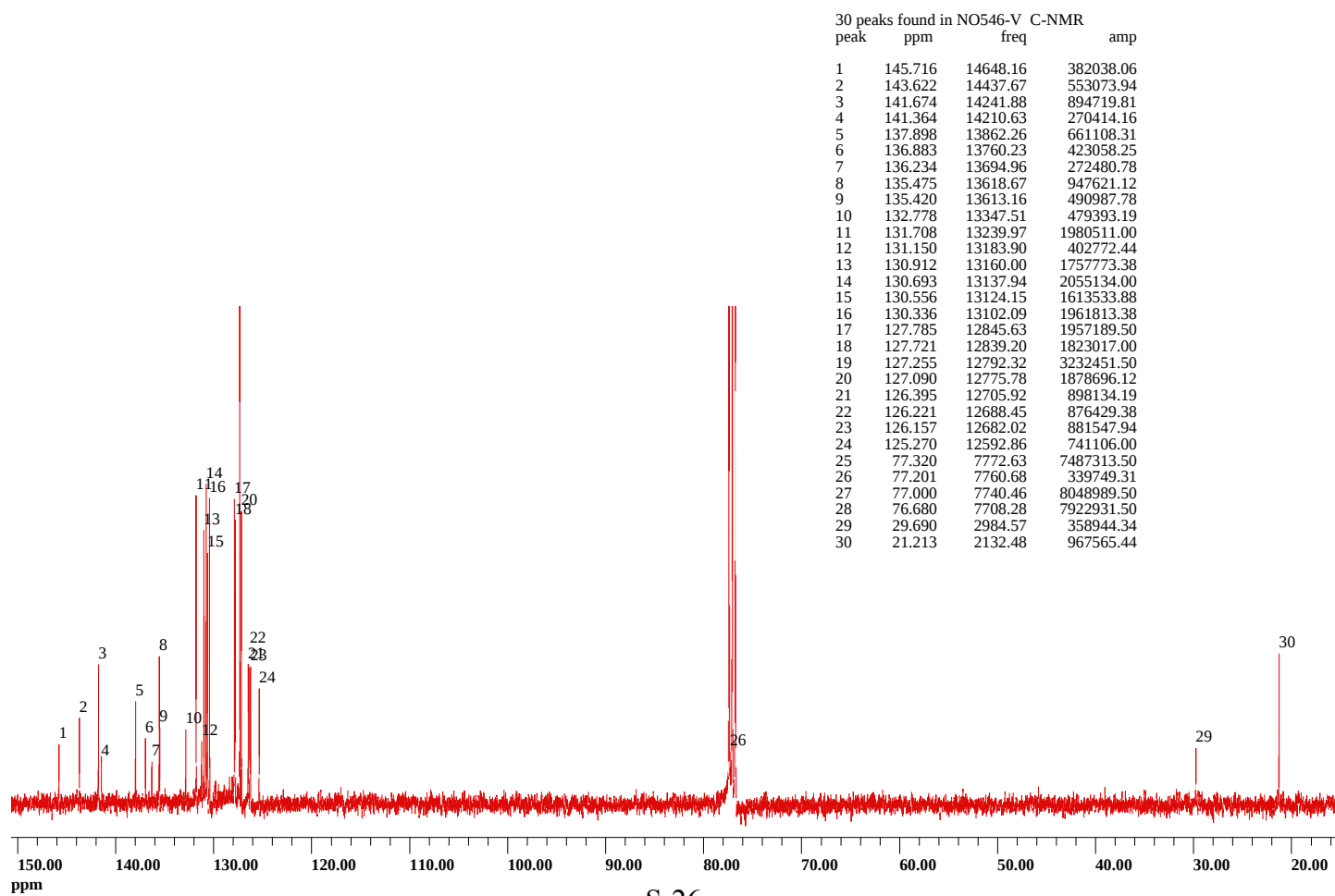
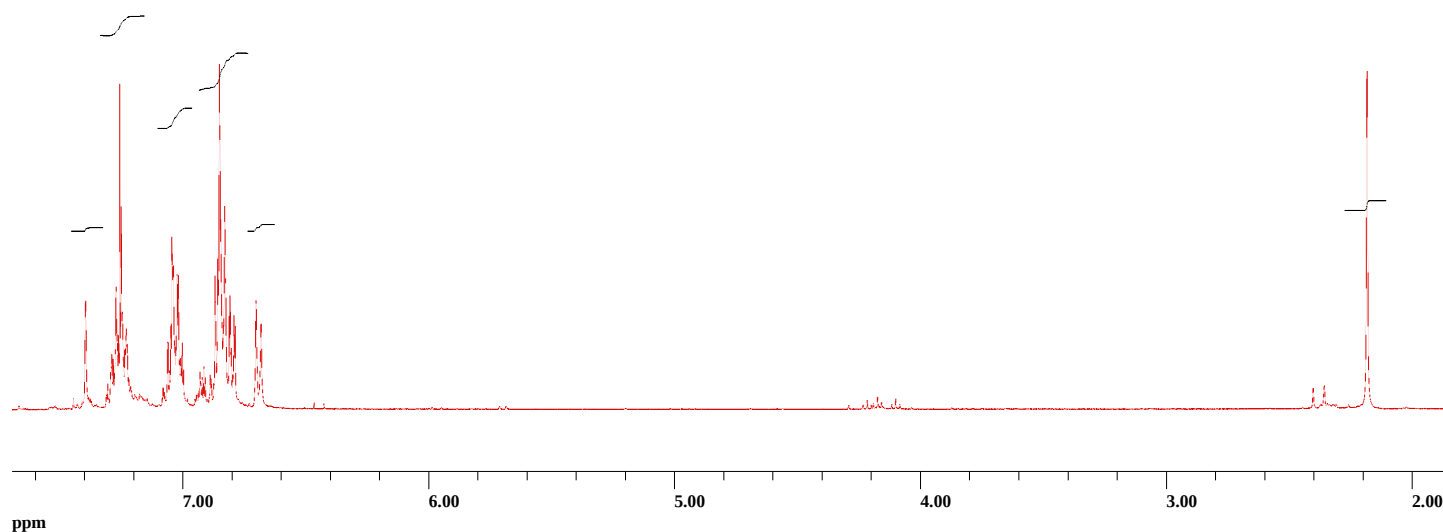


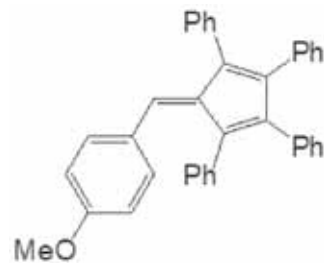
8k (CDCl₃, 400 MHz)



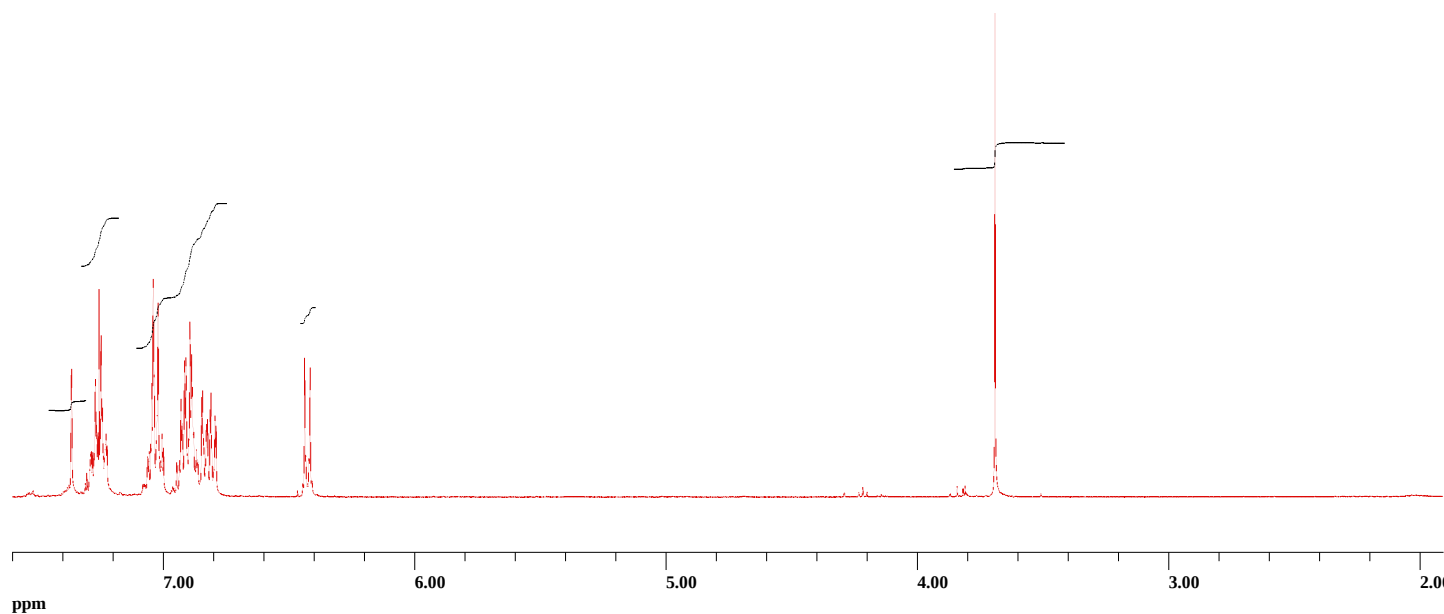


10b (CDCl₃, 400 MHz)



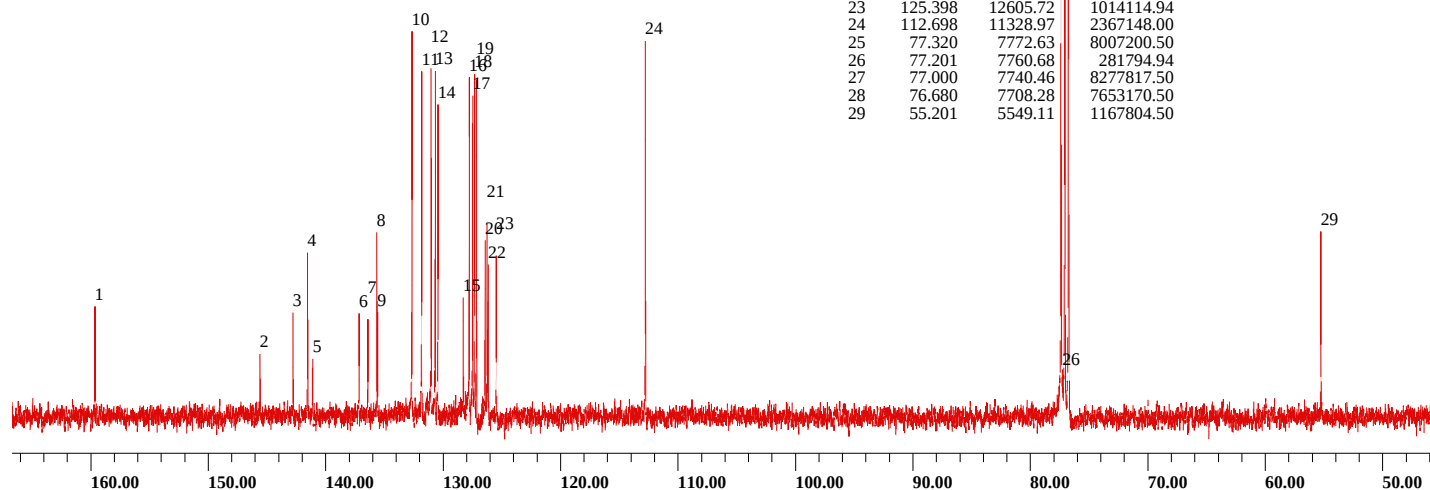


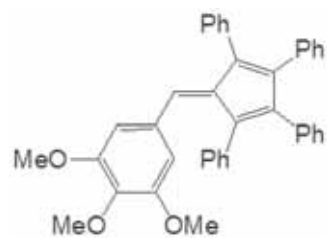
10c (CDCl₃, 400 MHz)



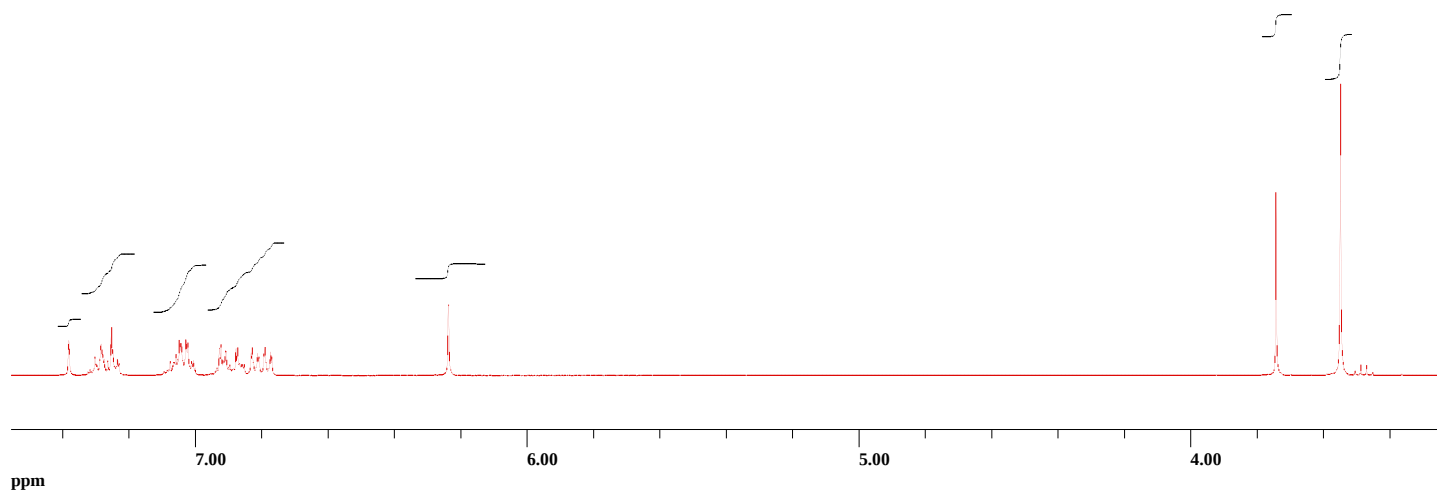
29 peaks found in NO540-V C-NMR

peak	ppm	freq	amp
1	159.569	16040.74	693862.06
2	145.515	14627.94	394238.66
3	142.699	14344.83	655090.25
4	141.455	14219.82	1031929.81
5	141.034	14177.54	364173.06
6	137.075	13779.53	650476.12
7	136.334	13705.08	612292.25
8	135.566	13627.86	1160053.25
9	135.493	13620.51	658564.69
10	132.576	13327.29	2429474.25
11	131.744	13243.64	2174871.50
12	130.949	13163.67	2196250.00
13	130.601	13128.74	2177381.75
14	130.364	13104.84	1967115.88
15	128.206	12887.92	748820.56
16	127.694	12836.44	2140362.50
17	127.410	12807.95	2021898.50
18	127.236	12790.48	2160279.50
19	127.081	12774.86	2120834.25
20	126.359	12702.24	1110783.00
21	126.176	12683.86	1217199.12
22	126.093	12675.58	958443.06
23	125.398	12605.72	1014114.94
24	112.698	11328.97	2367148.00
25	77.320	7772.63	8007200.50
26	77.201	7760.68	281794.94
27	77.000	7740.46	8277817.50
28	76.680	7708.28	7653170.50
29	55.201	5549.11	1167804.50



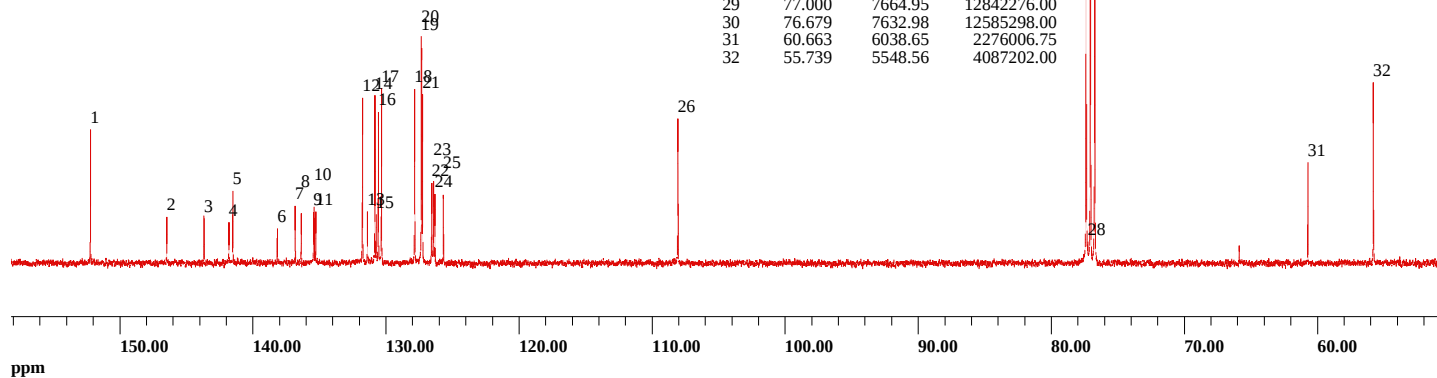


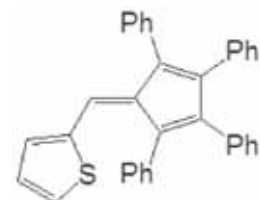
10e (CDCl₃, 400 MHz)



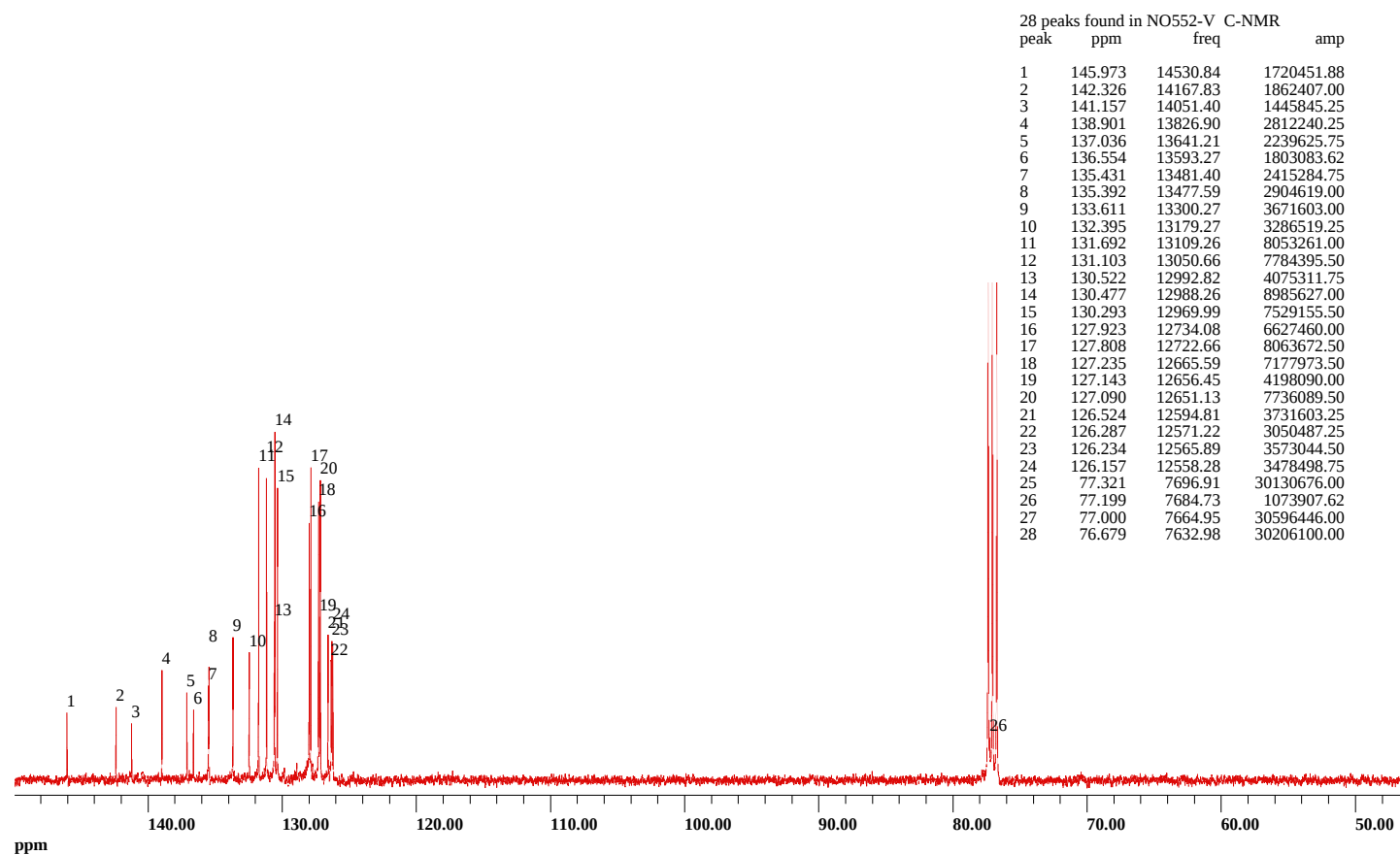
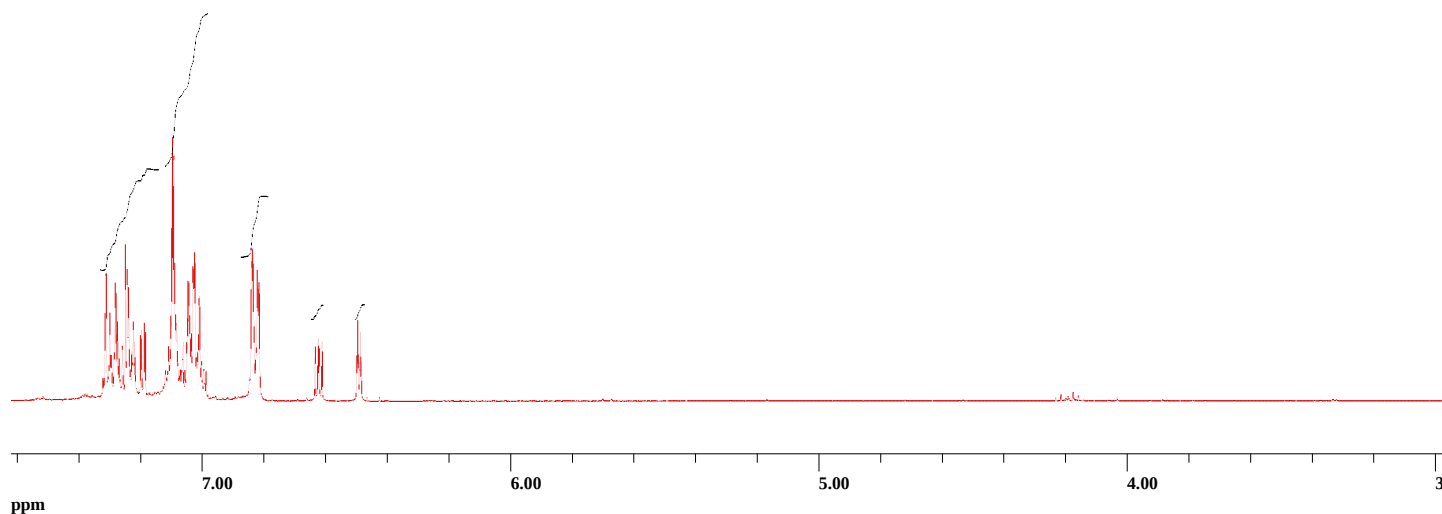
32 peaks found in NO573-V' C-NMR

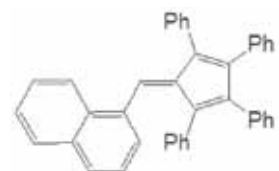
peak	ppm	freq	amp
1	152.142	15144.98	3017474.50
2	146.409	14574.22	1032924.94
3	143.603	14294.92	1004663.62
4	141.738	14109.24	915882.31
5	141.447	14080.32	1631233.12
6	138.099	13746.99	772530.81
7	136.761	13613.81	1290788.25
8	136.302	13568.15	1112276.50
9	135.377	13476.07	1148928.12
10	135.323	13470.74	1261558.12
11	135.209	13459.33	1153524.88
12	131.700	13110.02	3743366.75
13	131.325	13072.73	1159043.00
14	130.767	13017.18	3794788.75
15	130.683	13008.81	1095862.50
16	130.507	12991.30	3423469.25
17	130.278	12968.47	3963306.75
18	127.778	12719.62	3937486.00
19	127.273	12669.39	5136578.50
20	127.258	12667.87	4870207.00
21	127.189	12661.02	3820507.00
22	126.494	12591.77	1801906.00
23	126.364	12578.83	1844271.00
24	126.257	12568.18	1560336.00
25	125.622	12505.01	1539022.12
26	108.000	10750.87	3263696.50
27	77.321	7696.91	12590301.00
28	77.199	7684.73	478433.22
29	77.000	7664.95	12842276.00
30	76.679	7632.98	12585298.00
31	60.663	6038.65	2276006.75
32	55.739	5548.56	4087202.00



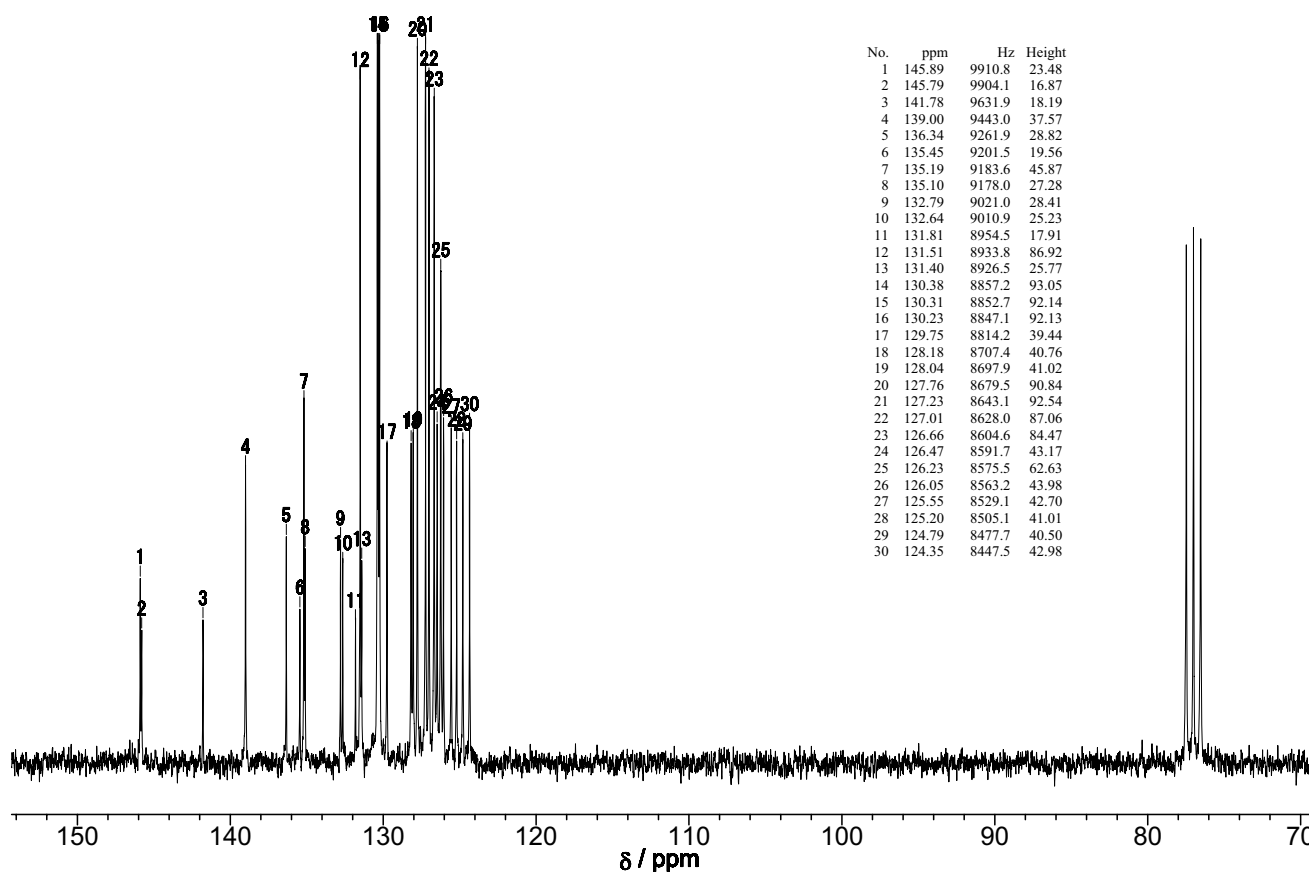
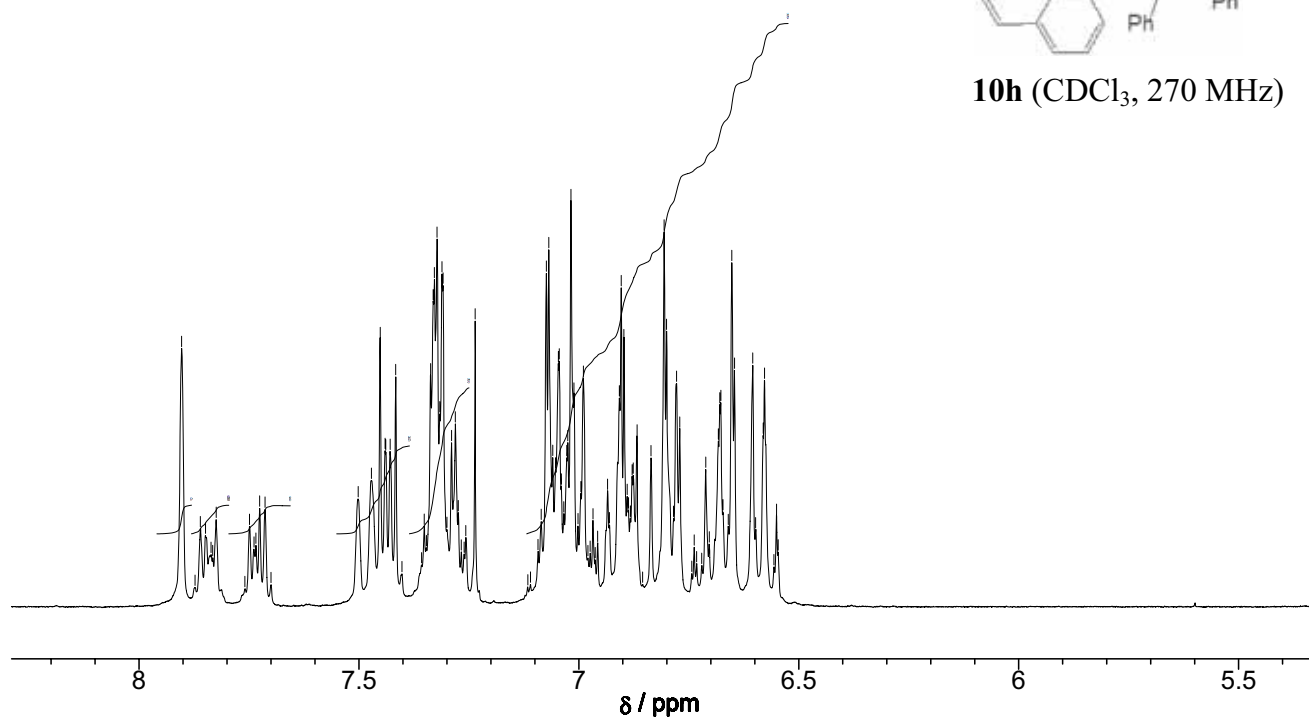


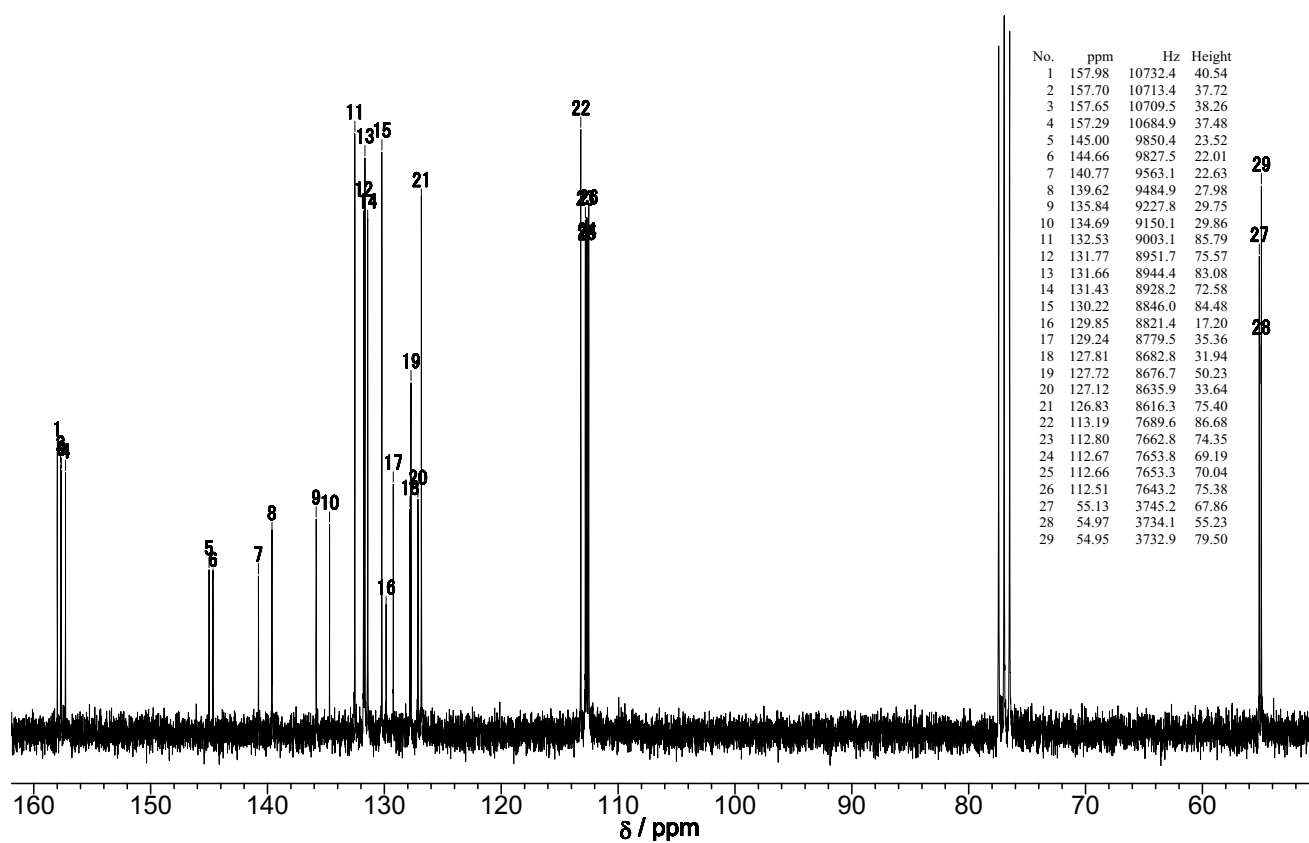
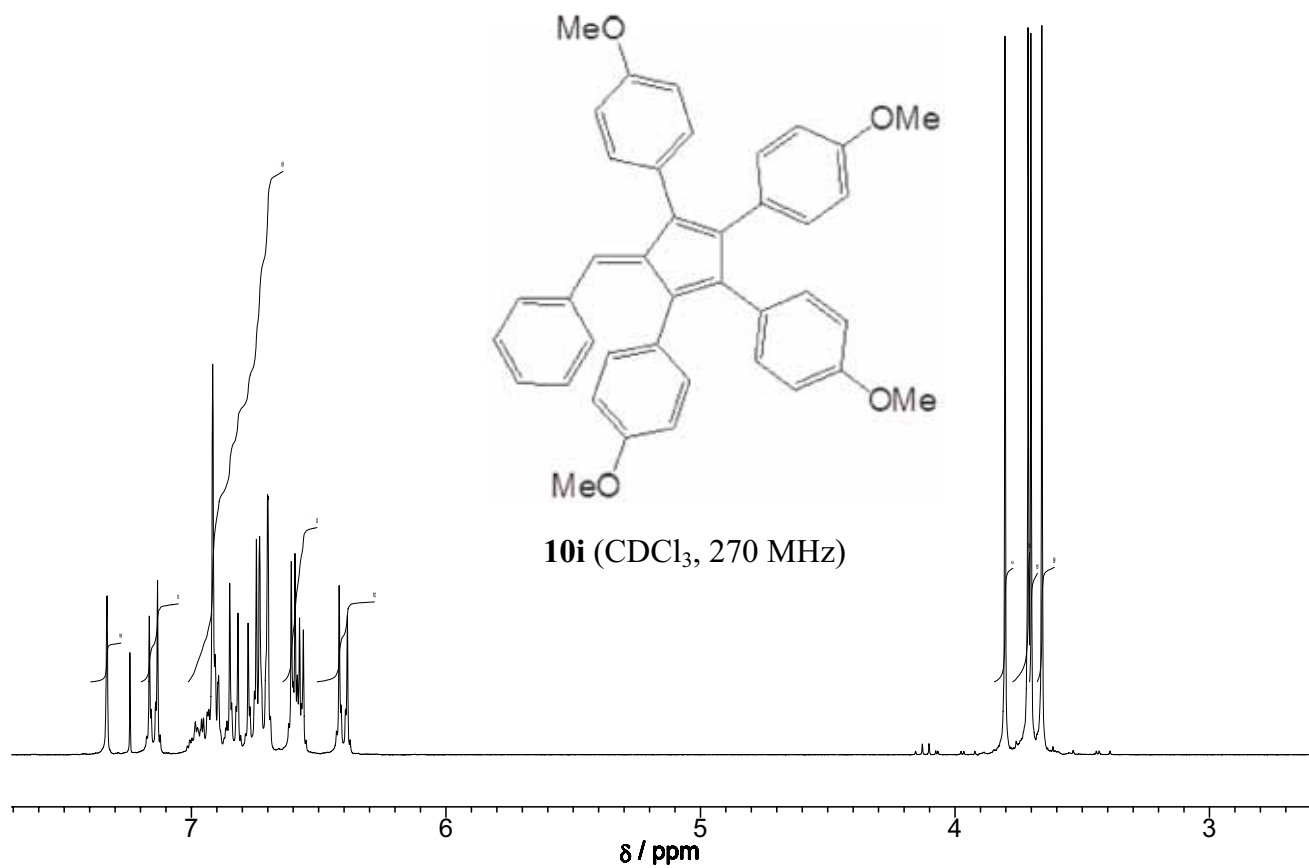
10f (CDCl₃, 400 MHz)

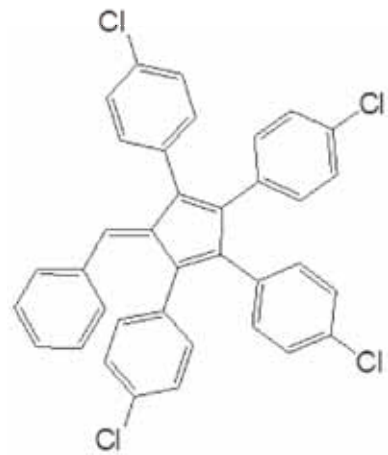




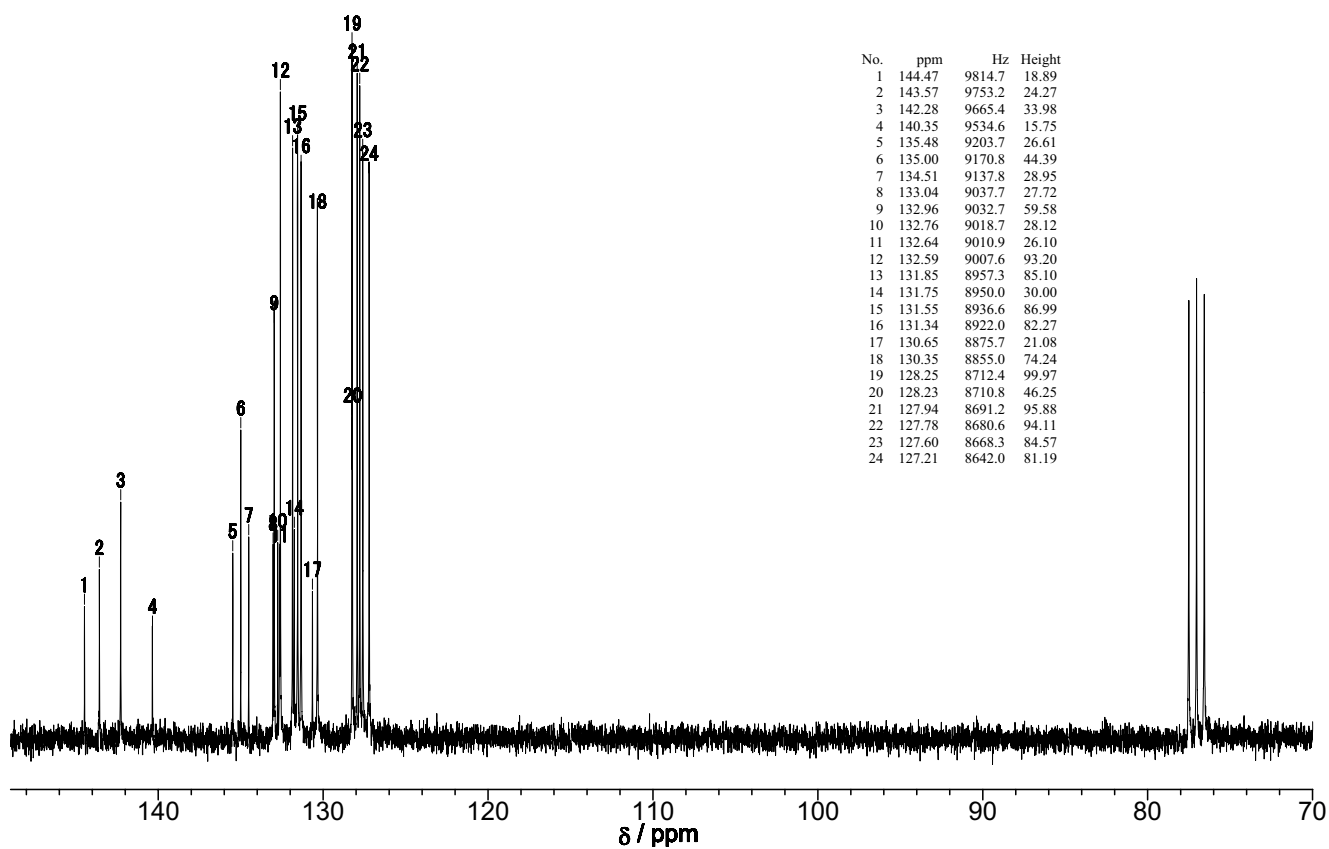
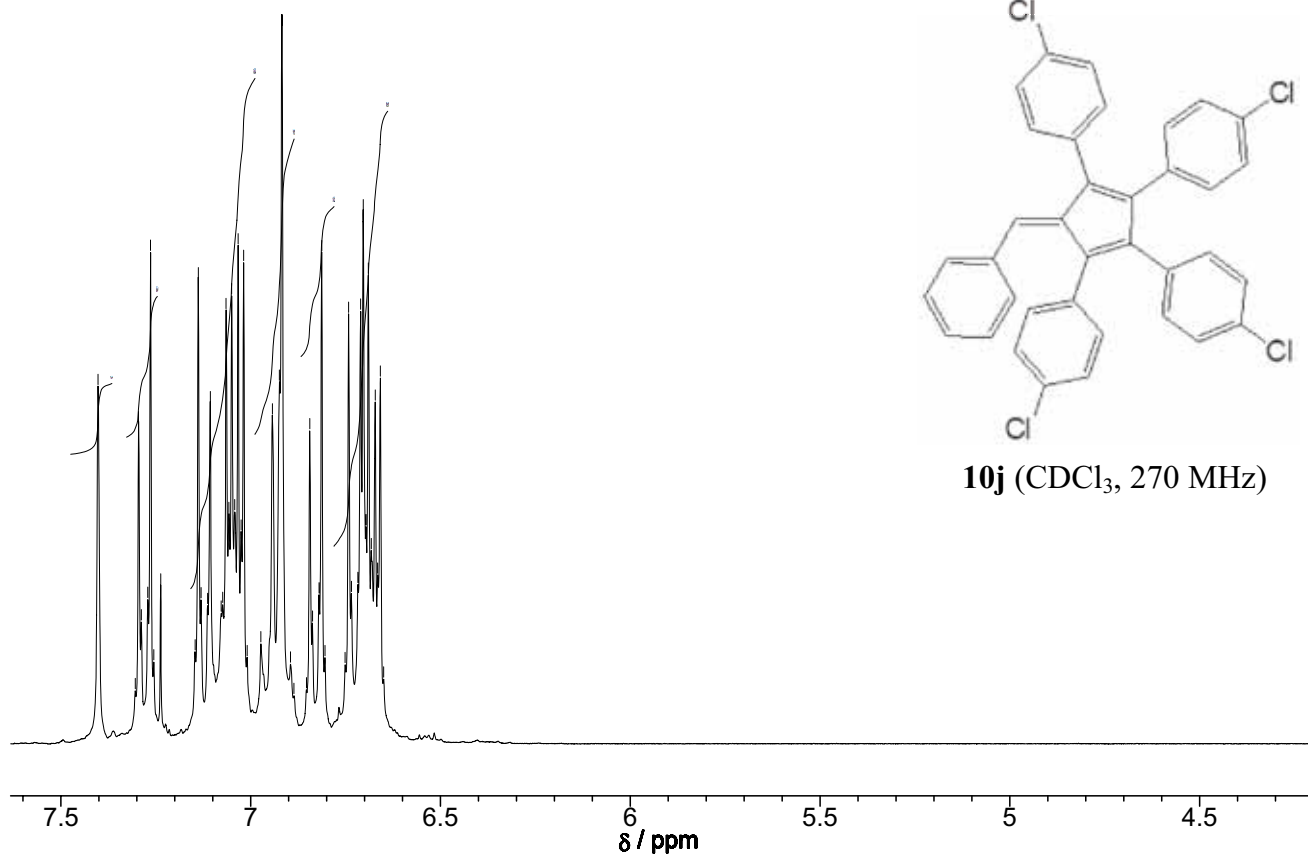
10h (CDCl₃, 270 MHz)

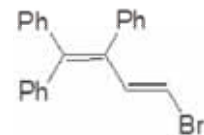




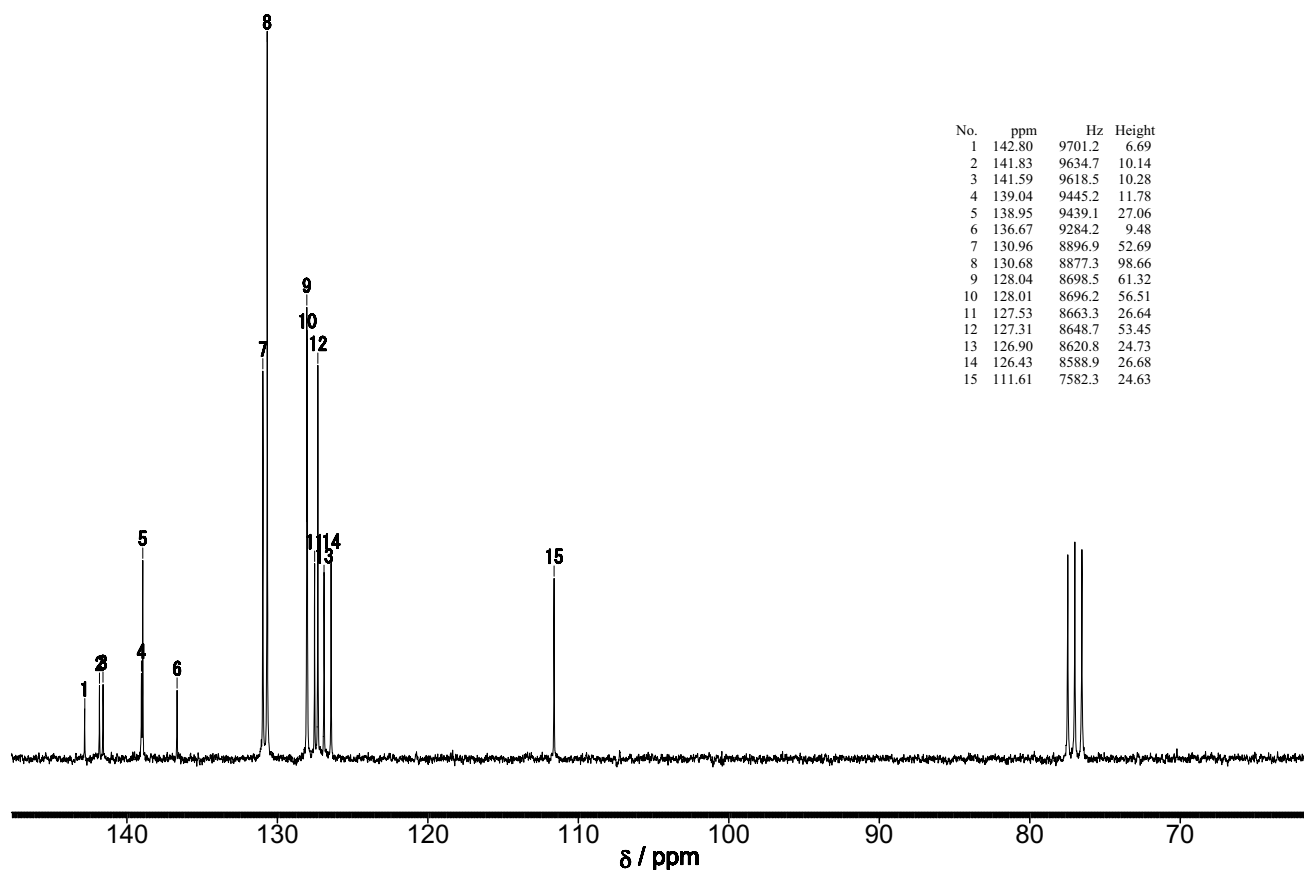
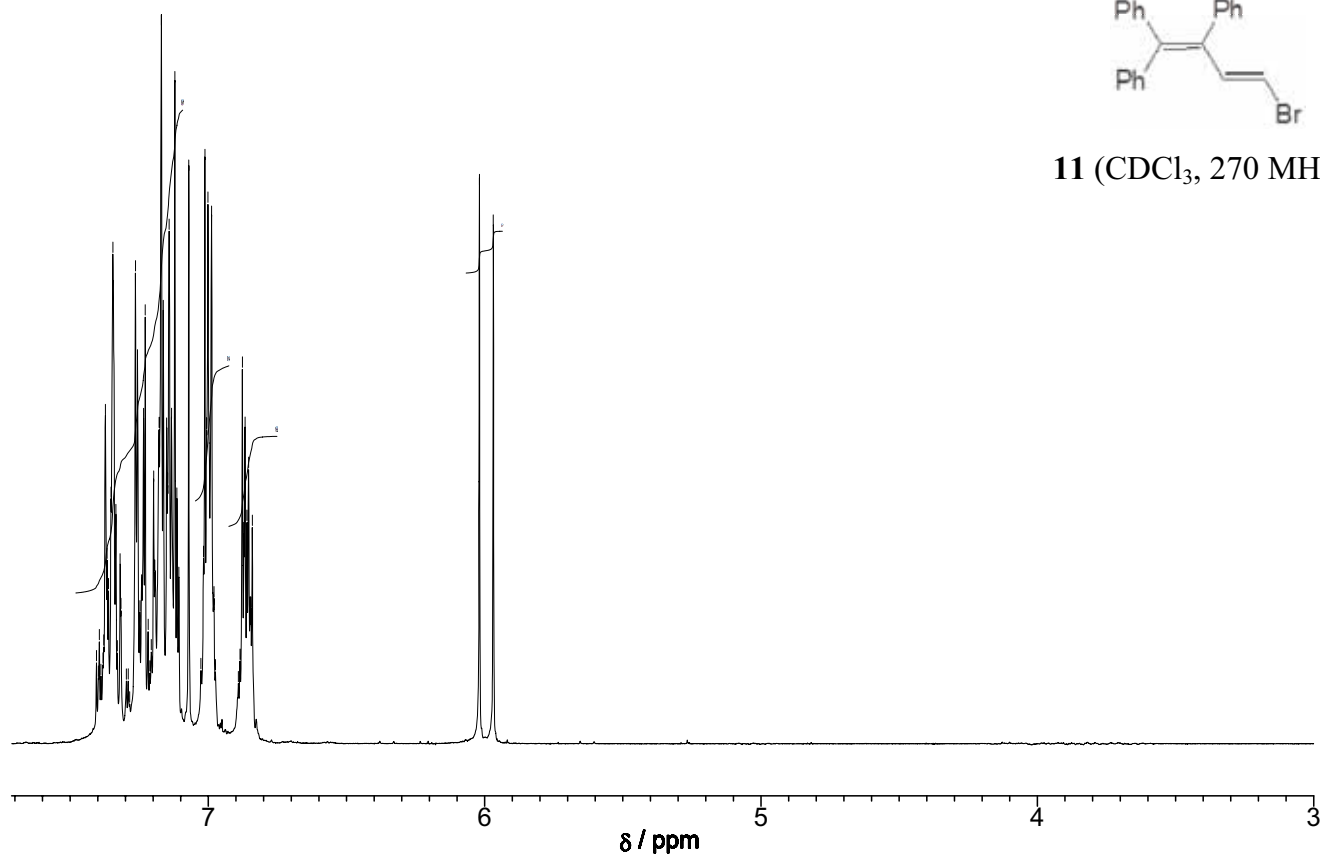


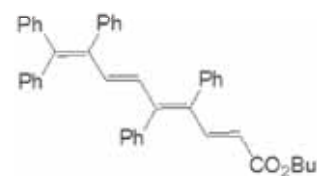
10j (CDCl₃, 270 MHz)



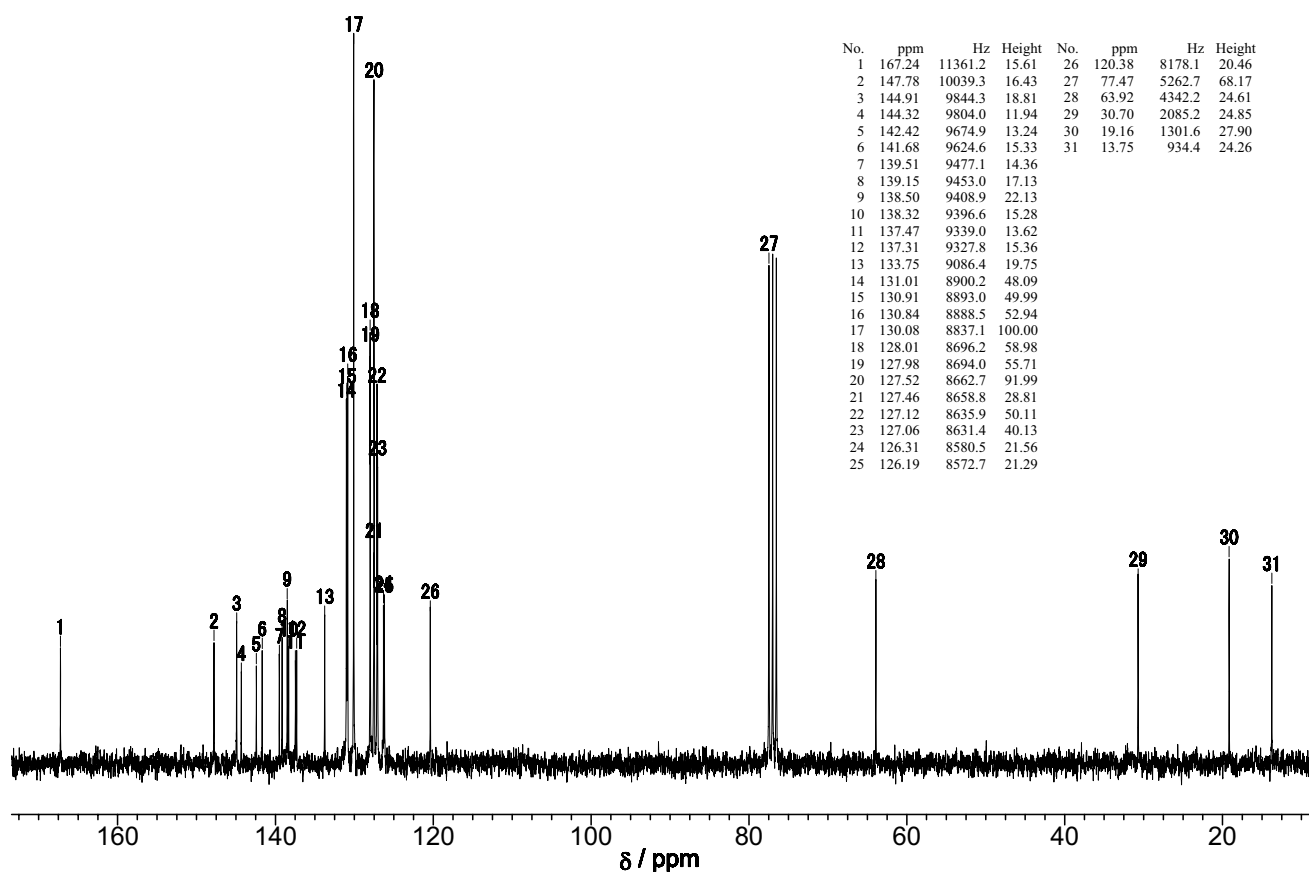
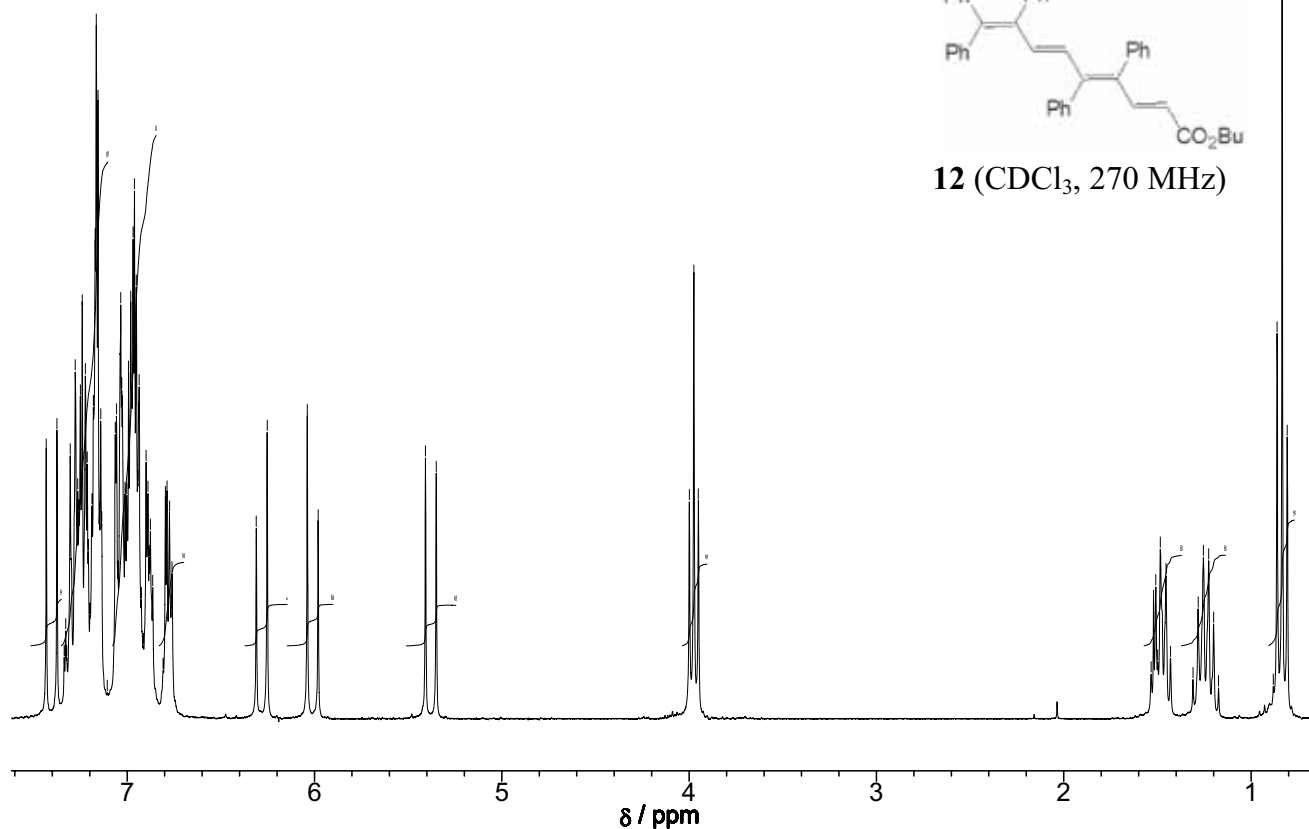


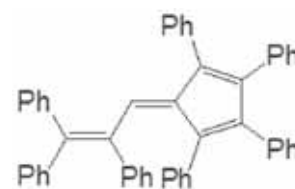
11 (CDCl₃, 270 MHz)



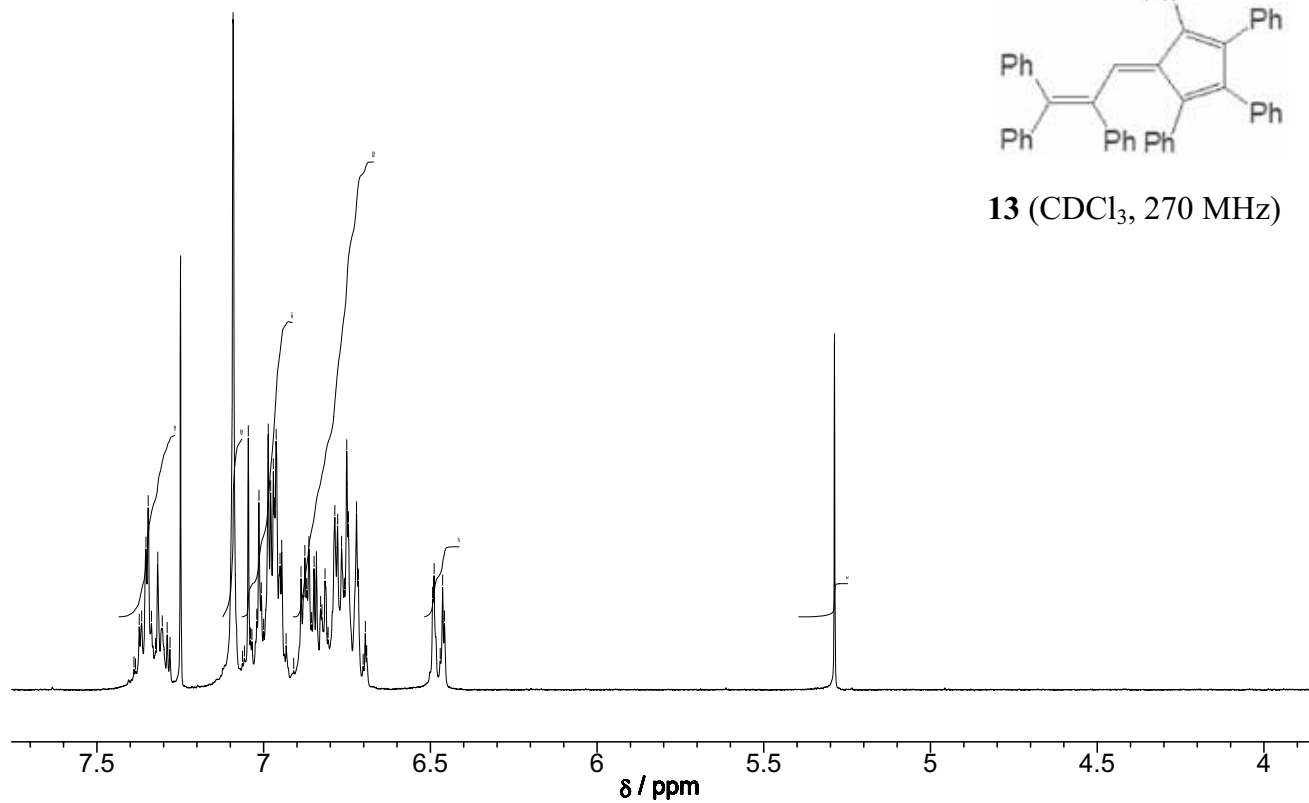


12 (CDCl₃, 270 MHz)





13 (CDCl₃, 270 MHz)



No.	ppm	Hz	Height
1	146.83	9974.5	4.07
2	145.47	9882.3	1.93
3	145.26	9868.3	4.70
4	144.24	9799.0	6.34
5	143.01	9715.2	6.48
6	142.19	9659.3	4.75
7	141.34	9601.7	6.66
8	139.25	9459.7	2.40
9	137.26	9324.5	3.25
10	136.82	9294.8	3.03
11	135.79	9225.0	3.18
12	135.57	9209.9	4.51
13	135.47	9203.2	3.24
14	135.33	9193.7	3.98
15	131.73	8948.9	1.55
16	131.37	8924.3	13.99
17	131.20	8912.5	16.75
18	131.13	8908.1	15.51
19	130.46	8862.8	14.12
20	130.24	8847.7	21.80
21	129.59	8803.5	13.36
22	127.99	8695.1	6.19
23	127.72	8676.1	15.01
24	127.48	8659.9	14.45
25	127.34	8651.0	14.85
26	127.27	8645.9	14.92
27	127.01	8628.0	17.00
28	126.97	8625.2	15.56
29	126.83	8615.7	15.19
30	126.70	8607.4	6.15
31	126.04	8562.6	10.84
32	125.93	8554.8	5.76
33	125.33	8514.0	5.35
34	125.03	8493.9	7.04

