

Supplementary Information

Rhodium-Catalyzed Aryl- and Alkylation–Oligomerization of Alkynoates with Organoboron Reagents Giving Salicylates

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General. All anaerobic and moisture-sensitive manipulations were carried out with standard Schlenk techniques under predried nitrogen or with glove box techniques under argon. NMR spectra were recorded on a JEOL JNM LA-500 spectrometer (500 MHz for ^1H , 125 MHz for ^{13}C). Chemical shifts are reported in δ ppm referenced to an internal SiMe_4 standard for ^1H NMR, chloroform-*d* (δ 77.16) for ^{13}C NMR unless otherwise noted: the following abbreviations are used; s: singlet, d: doublet, t: triplet, q: quartet, sext: sextet, sept: septet, m: multiplet, br: broad. Elemental analysis was performed at the Micro analytical center, Kyoto University. High-resolution mass spectra were obtained with a Bruker micrOTOF spectrometer. Flash column chromatography was performed with Silica Gel 60 N (spherical, neutral) (Cica-Reagent). Preparative thin-layer chromatography was performed with Silica Gel 60 PF₂₅₄ (Merck).

Materials. Et_2O was purified by passing through a neutral alumina column under nitrogen atmosphere. 1,4-Dioxane was distilled over benzophenone ketyl. Methanol was distilled over magnesium turnings. Triethylamine was distilled over KOH. 2-Pentynoic acid (Aldrich), *n*-BuLi (1.60 M solution in hexane, Kanto Chemicals), *n*-butylboronic acid (TCI), methyl 2-butynoate (TCI), ethyl 2-butynoate (TCI), methylboronic acid (Wako Chemicals), 4-phenyl-1-butyne (Wako Chemicals), 1-hexyne (Wako Chemicals), methyl chloroformate (Wako Chemicals), and ethyl chloroformate (Wako Chemicals) were used as received. $[\text{Rh}(\text{OH})(\text{cod})]_2$,¹ 4-(2-naphthyl)-1-butyne,² 5-(*tert*-butyldiphenylsiloxy)-1-pentyne,³ Methyl 2-bromo-3-methyl-2-butenolate⁴ were prepared according to literature procedures. All other chemicals were purchased from commercial suppliers and used as received.

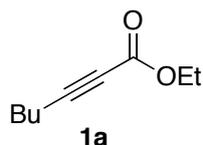
A General Procedure for Preparation of Alkynoates (1a, 1b, 1e, 1f, 1g). To a solution of the corresponding terminal alkyne (20 mmol) in Et_2O (20 mL) was slowly added *n*-BuLi (1.60 M in hexane, 22 mmol) at $-78\text{ }^\circ\text{C}$. After stirring at $-78\text{ }^\circ\text{C}$ for 30 min, the mixture was transferred to a solution of methyl chloroformate (1.7 mL, 22 mmol) or ethyl chloroformate (2.1 mL, 22 mmol) in Et_2O (20 mL) at $-78\text{ }^\circ\text{C}$ via a cannula, and the resulting solution was stirred for 30 min. The cooling bath was removed to warm up to room temperature and the mixture was stirred for additional 12 h. It was quenched with H_2O (20 mL) and extracted with Et_2O ($3 \times 20\text{ mL}$). The combined organic layers were washed with brine (20 mL), dried (MgSO_4), filtered, and concentrated on a rotary evaporator. The residue was purified by flash column chromatography on silica gel eluting with EtOAc /hexane.

(1) R. Uson, L. A. Oro and J. A. Cabeza, *Inorg. Synth.*, 1985, **23**, 129.

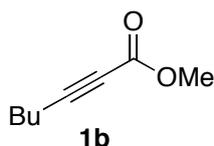
(2) L. Zhang and S. A. Kozmin, *J. Am. Chem. Soc.*, 2004, **126**, 10204.

(3) K. Muranaka, A. Sano, S. Ichikawa and A. Matsuda, *Bioorg. Med. Chem.*, 2008, **16**, 5862.

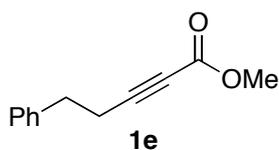
(4) M. Lloveras, I. Ramos, E. Molins and A. Messeguer, *Tetrahedron*, 2000, **56**, 3391.



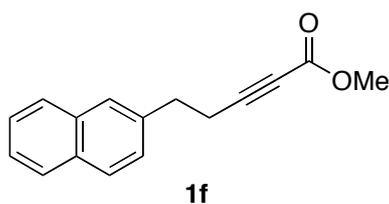
Compound **1a**: 92% yield. $^1\text{H NMR}$ (CDCl_3) δ 0.92 (t, $J = 7.3$ Hz, 3H), 1.31 (t, $J = 7.2$ Hz, 3H), 1.39–1.48 (m, 2H), 1.52–1.62 (m, 2H), 2.33 (t, $J = 7.1$ Hz, 2H), 4.22 (q, $J = 7.2$ Hz, 2H); $^{13}\text{C NMR}$ (CDCl_3) δ 13.6, 14.2, 18.5, 22.1, 29.7, 61.9, 73.3, 89.6, 154.0. HRMS (ESI) calcd for $\text{C}_9\text{H}_{14}\text{O}_2\text{Na}$ ($\text{M}+\text{Na}$) $^+$ 177.0886, found 177.0891.



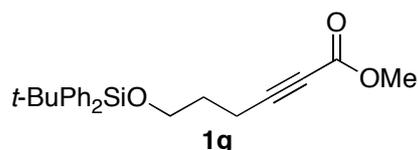
Compound **1b**: 35% yield. $^1\text{H NMR}$ (CDCl_3) δ 0.92 (t, $J = 7.3$ Hz, 3H), 1.39–1.48 (m, 2H), 1.52–1.61 (m, 2H), 2.34 (t, $J = 7.1$ Hz, 2H), 3.76 (s, 3H); $^{13}\text{C NMR}$ (CDCl_3) δ 13.6, 18.5, 22.1, 29.7, 52.7, 73.0, 90.1, 154.4. HRMS (ESI) calcd for $\text{C}_8\text{H}_{12}\text{O}_2\text{Na}$ ($\text{M}+\text{Na}$) $^+$ 163.0730, found 163.0725.



Compound **1e**: 70% yield. $^1\text{H NMR}$ (CDCl_3) δ 2.62 (t, $J = 7.6$ Hz, 2H), 2.90 (t, $J = 7.6$ Hz, 2H), 3.75 (s, 3H), 7.18–7.26 (m, 3H), 7.28–7.34 (m, 2H); $^{13}\text{C NMR}$ (CDCl_3) δ 21.0, 34.0, 52.7, 73.5, 88.9, 126.8, 128.5, 128.7, 139.7, 154.3. HRMS (ESI) calcd for $\text{C}_{12}\text{H}_{12}\text{O}_2\text{Na}$ ($\text{M}+\text{Na}$) $^+$ 211.0730, found 211.0732.

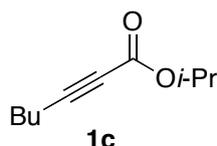


Compound **1f**: 64% yield. $^1\text{H NMR}$ (CDCl_3) δ 2.69 (t, $J = 7.6$ Hz, 2H), 3.03 (t, $J = 7.6$ Hz, 2H), 3.73 (s, 3H), 7.32 (dd, $J = 8.4, 1.7$ Hz, 1H), 7.38–7.49 (m, 2H), 7.64 (s, 1H), 7.73–7.83 (m, 3H); $^{13}\text{C NMR}$ (CDCl_3) δ 20.9, 34.1, 52.7, 73.6, 88.8, 125.7, 126.2, 126.8, 126.9, 127.7, 127.8, 128.3, 132.4, 133.6, 137.1, 154.2. HRMS (ESI) calcd for $\text{C}_{16}\text{H}_{14}\text{O}_2\text{Na}$ ($\text{M}+\text{Na}$) $^+$ 261.0886, found 261.0891.



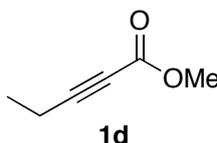
Compound **1g**: 93% yield. $^1\text{H NMR}$ (CDCl_3) δ 1.05 (s, 9H), 1.76–1.85 (m, 2H), 2.50 (t, $J = 7.2$ Hz, 2H), 3.73 (t, $J = 5.9$ Hz, 2H), 3.75 (s, 3H), 7.34–7.46 (m, 6H), 7.61–7.69 (m, 4H); $^{13}\text{C NMR}$ (CDCl_3) δ 15.4, 19.4, 27.0, 30.6, 52.7, 62.1, 73.1, 89.6, 127.8, 129.8, 133.7, 135.7, 154.3. HRMS (ESI) calcd for $\text{C}_{23}\text{H}_{28}\text{O}_3\text{SiNa}$ ($\text{M}+\text{Na}$) $^+$ 403.1700, found 403.1698.

Procedure for Preparation of 1c. To a solution of 2-heptynoic acid (1.6 g, 12.7 mmol) in *i*-PrOH (10 mL) was added *conc.* H_2SO_4 (0.20 mL), and the solution was refluxed for 18 h. After cooling to room temperature, the reaction mixture was quenched with H_2O (10 mL) and extracted with Et_2O (3×10 mL). The combined organic layers were washed with brine (10 mL), dried (MgSO_4), filtered, and concentrated on a rotary evaporator. The residue was purified by flash column chromatography on silica gel eluting with EtOAc/hexane (1/30) to give **1c** (1.4 g, 67% yield).



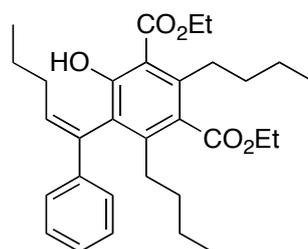
Compound **1c**: $^1\text{H NMR}$ (CDCl_3) δ 0.92 (t, $J = 7.3$ Hz, 3H), 1.29 (d, $J = 6.3$ Hz, 6H), 1.39–1.48 (m, 2H), 1.51–1.61 (m, 2H), 2.33 (t, $J = 7.1$ Hz, 2H), 5.08 (sept, $J = 6.3$ Hz, 1H); $^{13}\text{C NMR}$ (CDCl_3) δ 13.6, 18.5, 21.8, 22.1, 29.7, 69.7, 73.6, 89.2, 153.6. HRMS (ESI) calcd for $\text{C}_{10}\text{H}_{16}\text{O}_2\text{Na}$ ($\text{M}+\text{Na}$) $^+$ 191.1043, found 191.1043.

Procedure for Preparation of 1d. To a solution of 2-pentynoic acid (6.6 g, 67 mmol) in MeOH (14 mL) was added *conc.* H_2SO_4 (0.20 mL), and the solution was refluxed for 10 h. After cooling to room temperature, the reaction mixture was quenched with H_2O (20 mL) and extracted with pentane (3×20 mL). The combined organic layers were washed with brine (20 mL), dried (MgSO_4), filtered, and concentrated on a rotary evaporator. The residue was purified by Kugelrohr distillation (b.p. 78°C , 17 mmHg) to give **1d** (4.6 g, 61% yield).



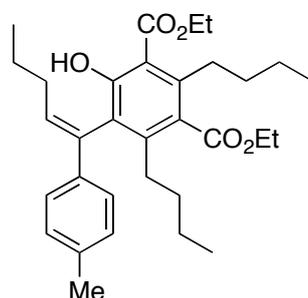
Compound **1d**: $^1\text{H NMR}$ (CDCl_3) δ 1.21 (t, $J = 7.5$ Hz, 3H), 2.35 (q, $J = 7.5$ Hz, 2H), 3.76 (s, 3H); $^{13}\text{C NMR}$ (CDCl_3) δ 12.5, 12.7, 52.7, 72.3, 91.0, 154.4. HRMS (ESI) calcd for $\text{C}_6\text{H}_8\text{O}_2\text{Na}$ ($\text{M}+\text{Na}$) $^+$ 135.0417, found 135.0418.

A Typical Procedure for Rhodium-Catalyzed Synthesis of Phenol Derivatives by Addition of Arylboroxines to Ethyl 2-Heptynoate (1a). To a solution of [Rh(OH)(cod)]₂ (3.3 mg, 0.014 mmol Rh) and phenylboroxine (93.2 mg, 0.299 mmol) in 1,4-dioxane (0.60 mL) was added ethyl 2-heptynoate (**1a**) (44.7 mg, 0.29 mmol). After stirring at 70 °C for 3 h, the mixture was diluted with Et₂O (3.0 mL), and filtered through a plug of silica gel. The plug was washed with Et₂O (20 mL) and the combined filtrates were concentrated on a rotary evaporator. The residue was purified by preparative TLC on silica gel eluting with EtOAc/hexane (1/12) to give a mixture of **3a-(E)** and **3a-(Z)** (38.3 mg, 0.077 mmol, 80% yield, *E/Z* = 10/90). The stereochemistry was determined by NOESY analysis. The results obtained for the addition of other arylboronic acids are summarized in Table 1.



3a-(Z)

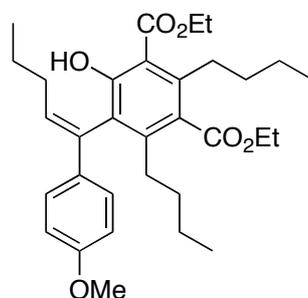
3a-(Z): ¹H NMR (CDCl₃) δ 0.73 (t, *J* = 7.2 Hz, 3H), 0.91 (t, *J* = 7.4 Hz, 3H), 0.94 (t, *J* = 7.4 Hz, 3H), 1.09–1.20 (m, 3H), 1.20–1.53 (m, 5H), 1.37 (t, *J* = 7.2 Hz, 3H), 1.44 (t, *J* = 7.2 Hz, 3H), 1.57–1.66 (m, 2H), 1.86 (ddt, *J* = 15.0, 8.1, 7.0 Hz, 1H), 1.96 (ddt, *J* = 15.0, 8.4, 7.0 Hz, 1H), 2.20–2.36 (m, 2H), 2.84 (dt, *J* = 13.1, 8.2 Hz, 1H), 2.88 (dt, *J* = 13.1, 8.3 Hz, 1H), 4.34 (q, *J* = 7.2 Hz, 2H), 4.46 (q, *J* = 7.2 Hz, 2H), 6.34 (t, *J* = 7.0 Hz, 1H), 7.14–7.20 (m, 1H), 7.20–7.25 (m, 4H), 11.13 (s, 1H); ¹³C NMR (CDCl₃) δ 13.7, 14.1, 14.2, 14.3, 22.4, 23.4, 23.5, 32.1, 32.2, 32.5, 33.4, 34.3, 61.2, 62.0, 110.7, 126.1, 126.4, 126.9, 128.4, 128.9, 132.6, 134.7, 141.0, 141.2, 144.9, 159.8, 170.3, 171.5. HRMS (ESI) calcd for C₃₁H₄₂O₅Na (M+Na)⁺ 517.2924, found 517.2936. **3a-(E):** Vinylic and phenolic protons are observed at δ 5.65 (t, *J* = 7.5 Hz, 1H) and 10.95 (s, 1H), respectively.



3b-(Z)

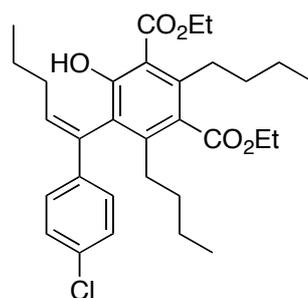
3b-(Z): ¹H NMR (CDCl₃) δ 0.74 (t, *J* = 7.1 Hz, 3H), 0.90 (t, *J* = 7.4 Hz, 3H), 0.94 (t, *J* = 7.4 Hz, 3H), 1.10–1.23 (m, 3H), 1.23–1.56 (m, 5H), 1.37 (t, *J* = 7.1 Hz, 3H), 1.43 (t, *J* = 7.1 Hz, 3H), 1.56–1.66 (m, 2H), 1.85 (ddt, *J* = 15.1, 8.0, 7.0 Hz, 1H), 1.94 (ddt, *J* = 15.1, 8.2, 7.0 Hz, 1H), 2.22–2.35 (m, 2H), 2.29 (s, 3H), 2.83 (dt, *J* = 13.8, 8.4 Hz, 1H), 2.87 (dt, *J* = 13.8, 8.4 Hz, 1H), 4.34 (q, *J* = 7.1 Hz, 2H), 4.45 (q, *J* = 7.1 Hz, 2H), 6.30 (t, *J* = 7.0 Hz, 1H), 7.04 (d, *J*

= 8.1 Hz, 2H), 7.12 (d, $J = 8.1$ Hz, 2H), 11.01 (s, 1H); ^{13}C NMR (CDCl_3) δ 13.8, 14.1, 14.22, 14.24, 14.3, 21.2, 22.5, 23.5, 23.6, 32.1, 32.2, 32.5, 33.4, 34.3, 61.2, 62.0, 110.8, 125.9, 126.5, 128.9, 129.1, 131.6, 134.4, 136.6, 138.1, 141.1, 144.9, 159.7, 170.4, 171.5. HRMS (ESI) calcd for $\text{C}_{32}\text{H}_{44}\text{O}_5\text{Na}$ ($\text{M}+\text{Na}$) $^+$ 531.3081, found 531.3088. **3b-(E)**: Vinylic and phenolic protons are observed at δ 5.61 (t, $J = 7.5$ Hz, 1H) and 10.84 (s, 1H), respectively.



3c-(Z)

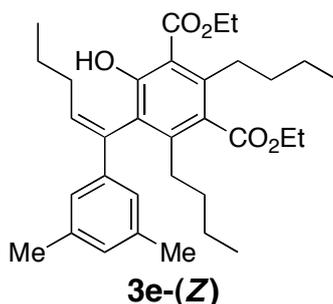
3c-(Z): ^1H NMR (CDCl_3) δ 0.74 (t, $J = 7.0$ Hz, 3H), 0.90 (t, $J = 7.5$ Hz, 3H), 0.94 (t, $J = 7.3$ Hz, 3H), 1.09–1.22 (m, 3H), 1.22–1.52 (m, 5H), 1.37 (t, $J = 7.2$ Hz, 3H), 1.43 (t, $J = 7.1$ Hz, 3H), 1.56–1.67 (m, 2H), 1.84 (ddt, $J = 15.1, 7.9, 7.1$ Hz, 1H), 1.93 (ddt, $J = 15.1, 8.2, 7.1$ Hz, 1H), 2.21–2.34 (m, 2H), 2.83 (dt, $J = 13.9, 8.3$ Hz, 1H), 2.88 (dt, $J = 13.9, 8.3$ Hz, 1H), 3.76 (s, 3H), 4.34 (q, $J = 7.2$ Hz, 2H), 4.45 (q, $J = 7.1$ Hz, 2H), 6.24 (t, $J = 7.1$ Hz, 1H), 6.78 (d, $J = 8.8$ Hz, 2H), 7.16 (d, $J = 8.8$ Hz, 2H), 11.00 (s, 1H); ^{13}C NMR (CDCl_3) δ 13.8, 14.1, 14.21, 14.23, 14.3, 22.5, 23.5, 23.6, 32.0, 32.2, 32.5, 33.3, 34.3, 55.4, 61.2, 62.0, 110.9, 113.9, 126.5, 127.1, 128.9, 130.7, 133.7, 134.0, 141.1, 144.9, 158.9, 159.6, 170.4, 171.5. HRMS (ESI) calcd for $\text{C}_{32}\text{H}_{44}\text{O}_6\text{Na}$ ($\text{M}+\text{Na}$) $^+$ 547.3030, found 547.3030. **3c-(E)**: Vinylic and phenolic protons are observed at δ 5.58 (t, $J = 7.3$ Hz, 1H) and 10.80 (s, 1H), respectively.



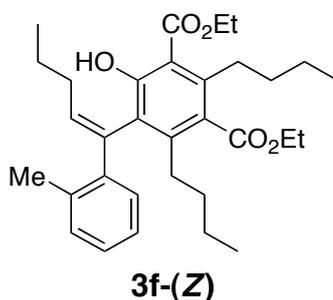
3d-(Z)

3d-(Z): ^1H NMR (CDCl_3) δ 0.75 (t, $J = 7.1$ Hz, 3H), 0.90 (t, $J = 7.4$ Hz, 3H), 0.95 (t, $J = 7.4$ Hz, 3H), 1.09–1.21 (m, 3H), 1.22–1.52 (m, 5H), 1.37 (t, $J = 7.2$ Hz, 3H), 1.44 (t, $J = 7.2$ Hz, 3H), 1.52–1.66 (m, 2H), 1.86 (ddt, $J = 15.2, 8.1, 7.1$ Hz, 1H), 1.94 (ddt, $J = 15.2, 8.2, 7.1$ Hz, 1H), 2.15–2.35 (m, 2H), 2.85 (dt, $J = 13.8, 8.2$ Hz, 1H), 2.89 (dt, $J = 13.8, 8.2$ Hz, 1H), 4.35 (q, $J = 7.2$ Hz, 2H), 4.46 (q, $J = 7.2$ Hz, 2H), 6.33 (t, $J = 7.1$ Hz, 1H), 7.17 (d, $J = 8.8$ Hz, 2H), 7.20 (d, $J = 8.8$ Hz, 2H), 11.31 (s, 1H); ^{13}C NMR (CDCl_3) δ 13.7, 14.1, 14.19, 14.21, 14.3, 22.4, 23.4, 23.6, 32.0, 32.2, 32.5, 33.4, 34.3, 61.3, 62.1, 110.6, 125.9, 127.3, 128.6, 129.0, 132.7, 133.1, 133.7, 139.6, 141.6, 145.0, 160.0, 170.2, 171.6. HRMS (ESI) calcd for $\text{C}_{31}\text{H}_{41}\text{O}_5\text{ClNa}$ ($\text{M}+\text{Na}$) $^+$ 551.2535, found 551.2539. **3d-(E)**: Vinylic and phenolic protons are observed at δ 5.58 (t, $J = 7.3$ Hz, 1H) and 10.80 (s, 1H), respectively.

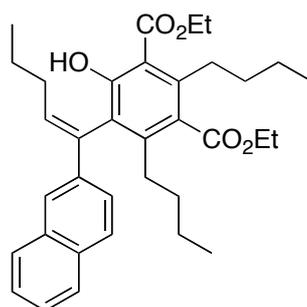
are observed at δ 5.65 (t, J = 7.4 Hz, 1H) and 11.19 (s, 1H), respectively.



3e-(Z): ¹H NMR (CDCl₃) δ 0.75 (t, J = 7.1 Hz, 3H), 0.90 (t, J = 7.4 Hz, 3H), 0.94 (t, J = 7.4 Hz, 3H), 1.10–1.23 (m, 3H), 1.23–1.53 (m, 5H), 1.37 (t, J = 7.1 Hz, 3H), 1.44 (t, J = 7.2 Hz, 3H), 1.57–1.67 (m, 2H), 1.83 (ddt, J = 15.1, 8.1, 7.0 Hz, 1H), 1.92 (ddt, J = 15.1, 8.2, 7.0 Hz, 1H), 2.24 (s, 6H), 2.23–2.35 (m, 2H), 2.78–2.92 (m, 2H), 4.34 (q, J = 7.1 Hz, 2H), 4.46 (q, J = 7.2 Hz, 2H), 6.27 (t, J = 7.0 Hz, 1H), 6.82 (s, 1H), 6.84 (s, 2H), 11.03 (s, 1H); ¹³C NMR (CDCl₃) δ 13.7, 14.1, 14.2, 14.3, 21.5, 22.5, 23.5, 23.6, 32.1, 32.2, 32.5, 33.4, 34.3, 61.2, 62.0, 110.8, 124.0, 126.6, 128.76, 128.81, 132.5, 134.9, 137.7, 141.0, 141.2, 144.9, 159.7, 170.4, 171.5. HRMS (ESI) calcd for C₃₃H₄₆O₅Na (M+Na)⁺ 545.3237, found 545.3230. **3e-(E):** Vinylic and phenolic protons are observed at δ 5.60 (t, J = 7.4 Hz, 1H) and 10.80 (s, 1H), respectively.



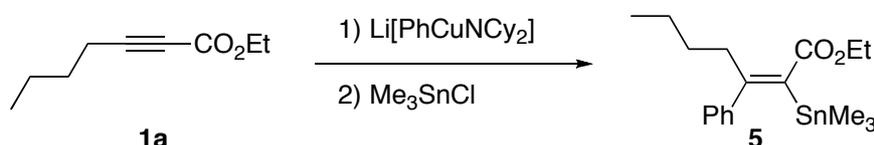
3f-(Z): ¹H NMR (CDCl₃) δ 0.76 (t, J = 7.4 Hz, 3H), 0.92 (t, J = 7.3 Hz, 3H), 0.91–1.06 (m, 1H), 0.93 (t, J = 7.3 Hz, 3H), 1.06–1.54 (m, 7H), 1.34 (t, J = 7.1 Hz, 3H), 1.44 (t, J = 7.1 Hz, 3H), 1.54–1.67 (m, 2H), 1.87 (ddt, J = 15.0, 8.0, 7.0 Hz, 1H), 1.99–2.12 (m, 1H), 2.21 (ddd, J = 13.1, 11.3, 5.1 Hz, 1H), 2.26 (ddd, J = 13.1, 11.2, 5.2 Hz, 1H), 2.39 (s, 3H), 2.79 (dt, J = 13.3, 8.3 Hz, 1H), 2.86 (dt, J = 13.3, 8.3 Hz, 1H), 4.31 (q, J = 7.1 Hz, 2H), 4.47 (q, J = 7.1 Hz, 2H), 5.96 (dd, J = 8.0, 6.3 Hz, 1H), 7.00–7.21 (m, 4H), 11.25 (s, 1H); ¹³C NMR (CDCl₃) δ 13.9, 14.1, 14.2, 14.25, 14.27, 20.9, 22.4, 23.5, 23.6, 32.30, 32.31, 32.5, 33.4, 34.3, 61.2, 62.0, 110.7, 125.8, 126.5, 127.5, 128.9, 129.4, 131.1, 133.2, 135.1, 137.5, 141.0, 141.7, 144.7, 159.7, 170.3, 171.6. HRMS (ESI) calcd for C₃₂H₄₄O₅Na (M+Na)⁺ 531.3081, found 531.3089. **3f-(E):** Vinylic and phenolic protons are observed at δ 5.71 (t, J = 7.4 Hz, 1H) and 10.94 (s, 1H), respectively.



3g-(Z)

3g-(Z): ¹H NMR (CDCl₃) δ 0.68 (t, *J* = 7.3 Hz, 3H), 0.94 (t, *J* = 7.5 Hz, 3H), 0.96 (t, *J* = 7.3 Hz, 3H), 1.05–1.24 (m, 3H), 1.24–1.45 (m, 3H), 1.36 (t, *J* = 7.0 Hz, 3H), 1.45 (t, *J* = 7.1 Hz, 3H), 1.45–1.57 (m, 2H), 1.60–1.69 (m, 2H), 1.92 (ddt, *J* = 15.2, 8.0, 7.1 Hz, 1H), 2.01 (ddt, *J* = 15.2, 8.2, 7.1 Hz, 1H), 2.29 (ddd, *J* = 12.9, 10.7, 5.9 Hz, 1H), 2.33 (ddd, *J* = 12.9, 11.8, 5.0 Hz, 1H), 2.84–2.94 (m, 2H), 4.34 (q, *J* = 7.0 Hz, 2H), 4.47 (q, *J* = 7.1 Hz, 2H), 6.49 (t, *J* = 7.1 Hz, 1H), 7.34–7.42 (m, 2H), 7.51 (s, 1H), 7.54 (dd, *J* = 8.7, 1.7 Hz, 1H), 7.67–7.79 (m, 3H), 11.18 (s, 1H); ¹³C NMR (CDCl₃) δ 13.7, 14.1, 14.2, 14.26, 14.31, 22.5, 23.4, 23.6, 32.1, 32.2, 32.6, 33.4, 34.3, 61.2, 62.1, 110.8, 124.5, 124.8, 125.6, 126.0, 126.3, 127.6, 128.0, 128.3, 129.0, 132.8, 133.3, 133.7, 134.7, 138.4, 141.4, 145.1, 159.9, 170.3, 171.6. HRMS (ESI) calcd for C₃₅H₄₄O₅Na (M+Na)⁺ 567.3081, found 567.3092. **3g-(E):** Vinylic and phenolic protons are observed at δ 5.73 (t, *J* = 7.6 Hz, 1H) and 11.03 (s, 1H), respectively.

Preparation of (Z)-Ethyl 3-Phenyl-2-(trimethylstannyl)hept-2-enoate **5**.⁵



To a solution of dicyclohexylamine (598 mg, 3.30 mmol) in Et₂O (18 mL) was added methyllithium (1.12 M in Et₂O, 2.9 mL, 3.3 mmol) at –30 °C, and the mixture was stirred at –30 °C for 30 min. To the mixture was added CuI (629 mg, 3.30 mmol), and the resulting white slurry was stirred at –30 °C for 30 min. PhLi (1.15 M in cyclohexane and Et₂O, 2.9 mL, 3.3 mmol) was added, and the mixture was cooled to –78 °C after being stirred at –30 °C for 30 min. Ethyl 2-heptynoate (**1a**) was added, and the mixture was stirred at –78 °C for 30 min. Me₃SnCl was added, and the mixture was stirred at –78 °C for 1 h. The mixture was allowed to warm to room temperature and it was further stirred at room temperature for 2 h. Aqueous NH₄Cl (2 mL) was added and the mixture was filtered through a pad of celite using Et₂O as an eluent. The filtrate was washed with H₂O, brine, dried (MgSO₄), filtered, and concentrated on a rotary evaporator. The residue containing **5-(E)** and **5-(Z)** (28/72) was subjected to flash column chromatography on silica gel eluting with EtOAc/hexane (1/100), and it was further purified by GPC to give **5-(Z)** (499 mg, 1.26 mmol, 42% yield). ¹H NMR (CDCl₃) δ –0.14 (s, satellite peaks: d, *J*_{119Sn-H} = 56.3 Hz; d, *J*_{117Sn-H} = 53.8 Hz, 9H), 0.81 (t, *J* =

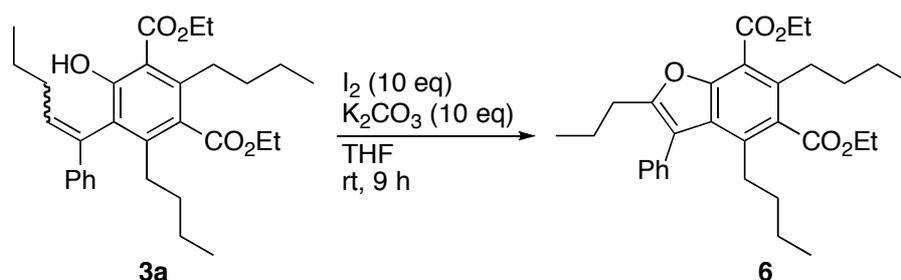
(5) T. Tsuda, T. Yoshida and T. Saegusa, *J. Org. Chem.*, 1988, **53**, 607.

7.1 Hz, 3H), 1.19–1.30 (m, 4H), 1.33 (t, $J = 7.1$ Hz, 3H), 2.60 (t, $J = 7.4$ Hz, 2H), 4.22 (q, $J = 7.1$ Hz, 2H), 7.14–7.19 (m, 2H), 7.26–7.35 (m, 3H). ^{13}C NMR (CDCl_3) δ -7.4, 14.0, 14.7, 22.6, 30.5, 37.1, 60.3, 127.6, 128.0, 128.3, 136.2, 144.2, 159.0, 172.7. HRMS (ESI) calcd for $\text{C}_{18}\text{H}_{28}\text{O}_2\text{SnNa}$ ($\text{M}+\text{Na}$) $^+$ 419.1006, found 419.1002.

Procedure for Rhodium-Catalyzed Addition of (Z)-Ethyl 3-Phenyl-2-(trimethylstannyl)hept-2-enoate (5) to Ethyl 2-Heptynoate (1a) (Scheme 5).

To a solution of **5** (132 mg, 0.33 mmol) in 1,4-dioxane (0.40 mL) and H_2O (40 μL) was added successively ethyl 2-heptynoate (**1a**) (26.2 mg, 0.17 mmol) and $[\text{Rh}(\text{OH})(\text{cod})]_2$ (1.9 mg, 0.0084 mmol Rh). After stirring at 100 $^\circ\text{C}$ for 3 h, the mixture was diluted with Et_2O (3.0 mL), and filtered through a plug of silica gel. The plug was washed with Et_2O (20 mL) and the combined filtrates were concentrated on a rotary evaporator. The residue was purified by preparative TLC on silica gel eluting with $\text{EtOAc}/\text{hexane}$ (1/10) to give a mixture of **3a-(E)** and **3a-(Z)** (19.9 mg, 0.040 mmol, 47% yield, $E/Z = 8/92$).

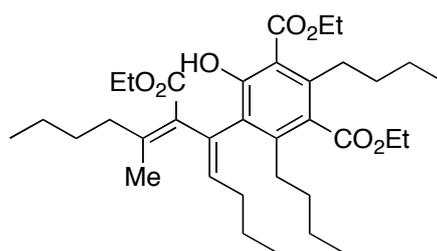
Transformation of 3a into 6.



To a solution of **3a** (50.6 mg, 0.102 mmol) in THF (1.0 mL) was added successively K_2CO_3 (138 mg, 1.0 mmol) and I_2 (127 mg, 1.0 mmol) at 0 $^\circ\text{C}$. The cooling bath was removed to warm up to room temperature and the mixture was stirred for additional 9 h. It was quenched with sat. $\text{Na}_2\text{S}_2\text{O}_3$ aq (2 mL) and extracted with Et_2O (3×2 mL). The combined organic layers were washed with brine (5 mL), dried (MgSO_4), filtered, and concentrated on a rotary evaporator. The residue was purified by preparative TLC on silica gel eluting with $\text{EtOAc}/\text{hexane}$ (1/10) to give **6** (46.9 mg, 0.0953 mmol, 93% yield). ^1H NMR (CDCl_3) δ 0.57 (t, $J = 7.3$ Hz, 3H), 0.73 (sext, $J = 7.3$ Hz, 2H), 0.89 (t, $J = 7.4$ Hz, 3H), 0.92 (t, $J = 7.4$ Hz, 3H), 1.09–1.23 (m, 2H), 1.35 (t, $J = 7.1$ Hz, 3H), 1.38 (sext, $J = 7.4$ Hz, 2H), 1.44 (t, $J = 7.1$ Hz, 3H), 1.53–1.67 (m, 2H), 1.67 (sext, $J = 7.4$ Hz, 2H), 2.28–2.42 (m, 2H), 2.55 (t, $J = 7.4$ Hz, 2H), 2.72–2.85 (m, 2H), 4.35 (q, $J = 7.1$ Hz, 2H), 4.49 (q, $J = 7.1$ Hz, 2H), 7.29–7.35 (m, 2H), 7.35–7.44 (m, 3H); ^{13}C NMR (CDCl_3) 13.6, 13.9, 14.0, 14.3, 14.5, 21.7, 22.9, 23.3, 28.4, 30.4, 31.4, 34.0, 34.4, 61.2, 61.4, 114.9, 117.6, 125.9, 127.8, 128.2, 130.6, 130.9, 133.7, 134.5, 135.9, 151.9, 156.9, 166.5, 170.3. HRMS (ESI) calcd for $\text{C}_{31}\text{H}_{40}\text{O}_5\text{Na}$ ($\text{M}+\text{Na}$) $^+$ 515.2768, found 515.2771.

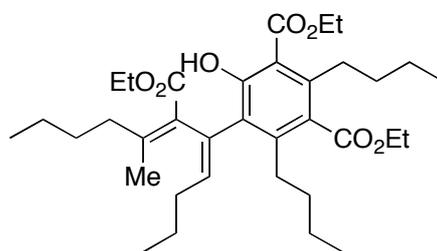
A Typical Procedure for Rhodium-Catalyzed Synthesis of Phenol Derivatives by Addition of Methylboronic Acid (7) to Alkynoates. To a solution of $[\text{Rh}(\text{OH})(\text{cod})]_2$ (5.6 mg, 0.025 mmol of Rh) and methylboronic acid (74.8 mg, 1.25 mmol) in 1,4-dioxane (1.0

mL) was added ethyl 2-heptynoate (**1a**) (77.1 mg, 0.50 mmol). After stirring at 70 °C for 12 h, the mixture was diluted with Et₂O (3.0 mL), and filtered through a plug of silica gel. The plug was washed with Et₂O (20 mL) and the combined filtrates were concentrated on a rotary evaporator. The residue was purified by preparative TLC on silica gel eluting with EtOAc/hexane (1/7) to give **8a-(E)** (46.4 mg, 0.079 mmol, 64% yield) and **8a-(Z)** (6.9 mg, 0.012 mmol, 10% yield). The yields were based on the alkynoate. The stereochemistry of **8a-(E)** was determined by NOESY analysis. The stereochemistry of **8a-(Z)** was determined by X-ray crystal analysis (Figure S1, vide infra) and NOESY analysis. The results obtained for the addition to other alkynoates are summarized in Table 2.



8a-(E)

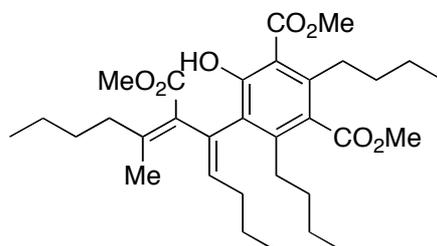
8a-(E): ¹H NMR (CDCl₃) δ 0.86 (t, *J* = 7.2 Hz, 3H), 0.87 (t, *J* = 7.4 Hz, 3H), 0.89 (t, *J* = 7.8 Hz, 3H), 0.91 (t, *J* = 7.3 Hz, 3H), 1.08 (t, *J* = 7.1 Hz, 3H), 1.23–1.62 (m, 14H), 1.37 (t, *J* = 7.2 Hz, 3H), 1.39 (t, *J* = 7.1 Hz, 3H), 1.78 (dtd, *J* = 15.0, 8.1, 6.8 Hz, 1H), 1.88 (s, 3H), 1.98 (ddt, *J* = 15.0, 8.7, 6.2 Hz, 1H), 2.12 (ddd, *J* = 12.6, 9.0, 6.1 Hz, 1H), 2.14 (ddd, *J* = 12.6, 9.2, 6.7 Hz, 1H), 2.30 (td, *J* = 12.6, 4.8 Hz, 1H), 2.51 (td, *J* = 12.6, 4.8 Hz, 1H), 2.65 (ddd, *J* = 13.3, 9.9, 6.6 Hz, 1H), 2.78 (ddd, *J* = 13.3, 10.1, 6.4 Hz, 1H), 3.99 (dq, *J* = 10.7, 7.1 Hz, 1H), 4.03 (dq, *J* = 10.7, 7.1 Hz, 1H), 4.34 (q, *J* = 7.1 Hz, 2H), 4.41 (q, *J* = 7.2 Hz, 2H), 5.88 (dd, *J* = 8.1, 6.2 Hz, 1H), 9.78 (s, 1H); ¹³C NMR (CDCl₃) δ 13.9, 14.0, 14.05, 14.07, 14.20, 14.22, 14.3, 19.9, 22.2, 22.9, 23.4, 23.6, 30.5, 31.7, 31.9, 32.81, 32.82, 34.1, 37.2, 60.6, 61.1, 61.7, 113.4, 124.5, 128.3, 129.2, 132.4, 139.6, 140.2, 141.4, 144.1, 157.4, 169.6, 170.3, 170.6. HRMS (ESI) calcd for C₃₅H₅₄O₇Na (M+Na)⁺ 609.3762, found 609.3764.



8a-(Z)

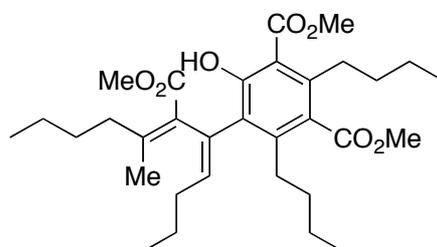
8a-(Z): ¹H NMR (CDCl₃) δ 0.87 (t, *J* = 7.3 Hz, 3H), 0.90 (t, *J* = 7.3 Hz, 3H), 0.91 (t, *J* = 7.3 Hz, 3H), 0.96 (t, *J* = 7.3 Hz, 3H), 1.11 (t, *J* = 7.1 Hz, 3H), 1.21–1.42 (m, 6H), 1.36 (t, *J* = 7.2 Hz, 3H), 1.38 (t, *J* = 7.2 Hz, 3H), 1.42–1.60 (m, 8H), 1.85 (s, 3H), 2.06 (br s, 2H), 2.25 (t, *J* = 7.9 Hz, 2H), 2.38 (br s, 1H), 2.55 (br s, 1H), 2.67 (t, *J* = 8.2 Hz, 2H), 4.02 (q, *J* = 7.2 Hz, 2H), 4.32 (q, *J* = 7.1 Hz, 2H), 4.39 (q, *J* = 7.2 Hz, 2H), 5.64 (t, *J* = 7.2 Hz, 1H), 9.67 (s, 1H); ¹³C NMR (CDCl₃) δ 13.95, 14.00, 14.06, 14.10, 14.26, 14.29, 21.0, 22.5, 23.0, 23.4, 23.7, 30.9,

31.7, 32.2, 32.7, 34.0, 34.1, 36.2, 60.7, 61.0, 61.6, 114.1, 127.7, 127.9, 128.1, 130.1, 138.6, 139.4, 143.5, 146.0, 157.8, 169.2, 170.37, 170.43. HRMS (ESI) calcd for $C_{35}H_{54}O_7Na$ ($M+Na$)⁺ 609.3762, found 609.3753.



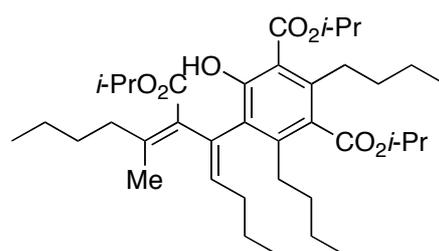
8b-(E)

8b-(E): ¹H NMR (CDCl₃) δ 0.866 (t, *J* = 7.1 Hz, 3H), 0.870 (t, *J* = 7.5 Hz, 3H), 0.89 (t, *J* = 7.3 Hz, 3H), 0.91 (t, *J* = 7.3 Hz, 3H), 1.22–1.57 (m, 14H), 1.77 (dtd, *J* = 15.0, 8.1, 6.8 Hz, 1H), 1.88 (s, 3H), 1.96 (ddt, *J* = 15.0, 8.5, 6.3 Hz, 1H), 2.11 (ddd, *J* = 13.0, 9.2, 6.1 Hz, 1H), 2.14 (ddd, *J* = 13.0, 9.4, 6.7 Hz, 1H), 2.29 (td, *J* = 12.6, 5.1 Hz, 1H), 2.48 (td, *J* = 12.6, 5.1 Hz, 1H), 2.64 (ddd, *J* = 13.5, 9.7, 6.6 Hz, 1H), 2.70 (ddd, *J* = 13.5, 9.4, 7.0 Hz, 1H), 3.56 (s, 3H), 3.85 (s, 3H), 3.93 (s, 3H), 5.87 (dd, *J* = 8.1, 6.3 Hz, 1H), 9.68 (s, 1H); ¹³C NMR (CDCl₃) δ 13.9, 14.0, 14.1, 14.2, 19.9, 22.2, 22.9, 23.4, 23.6, 30.5, 31.7, 31.9, 32.77, 32.83, 34.0, 37.2, 51.5, 51.9, 52.4, 113.4, 124.5, 128.1, 129.3, 132.1, 139.6, 140.5, 142.3, 144.2, 157.3, 169.9, 170.7, 170.9. HRMS (ESI) calcd for $C_{32}H_{48}O_7Na$ ($M+Na$)⁺ 567.3292, found 567.3273. Anal. Calcd for $C_{32}H_{48}O_7$: C, 70.56; H, 8.88. Found: C, 70.49; H, 8.77.



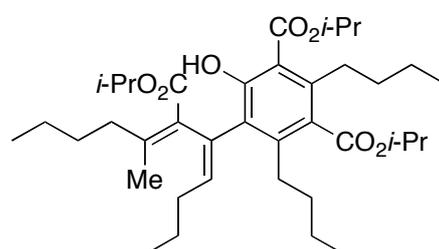
8b-(Z)

8b-(Z): ¹H NMR (CDCl₃) δ 0.88 (t, *J* = 7.3 Hz, 3H), 0.90 (t, *J* = 7.3 Hz, 3H), 0.91 (t, *J* = 7.2 Hz, 3H), 0.96 (t, *J* = 7.4 Hz, 3H), 1.20–1.54 (m, 14H), 1.85 (s, 3H), 2.06 (br d, *J* = 6.8 Hz, 2H), 2.25 (t, *J* = 7.8 Hz, 2H), 2.30–2.73 (m, 4H), 3.56 (s, 3H), 3.84 (s, 3H), 3.90 (s, 3H), 5.64 (t, *J* = 7.2 Hz, 1H), 9.57 (s, 1H); ¹³C NMR (CDCl₃) δ 13.9, 14.0, 14.1, 14.3, 21.1, 22.4, 23.0, 23.4, 23.6, 30.9, 31.5, 32.2, 32.7, 33.9, 34.0, 36.2, 51.6, 51.9, 52.3, 114.1, 127.5, 127.7, 127.9, 130.1, 138.8, 139.7, 143.8, 147.1, 157.8, 169.5, 170.78, 170.81. HRMS (ESI) calcd for $C_{32}H_{48}O_7Na$ ($M+Na$)⁺ 567.3292, found 567.3288.



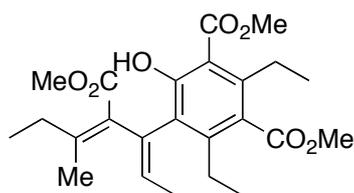
8c-(E)

8c-(E): ¹H NMR (CDCl₃) 0.86 (t, *J* = 7.4 Hz, 3H), 0.88 (t, *J* = 7.6 Hz, 3H), 0.89 (t, *J* = 7.3 Hz, 3H), 0.91 (t, *J* = 7.3 Hz, 3H), 1.01 (d, *J* = 6.3 Hz, 3H), 1.10 (d, *J* = 6.4 Hz, 3H), 1.16–1.59 (m, 14H), 1.35 (d, *J* = 6.3 Hz, 3H), 1.36 (d, *J* = 6.3 Hz, 3H), 1.37 (d, *J* = 6.3 Hz, 6H), 1.79 (dtd, *J* = 15.0, 8.2, 6.9 Hz, 1H), 1.87 (s, 3H), 1.99 (ddt, *J* = 15.0, 8.7, 6.1 Hz, 1H), 2.10 (ddd, *J* = 13.0, 9.5, 5.9 Hz, 1H), 2.13 (ddd, *J* = 13.0, 9.5, 6.4 Hz, 1H), 2.30 (td, *J* = 12.7, 4.7 Hz, 1H), 2.52 (td, *J* = 12.7, 4.7 Hz, 1H), 2.64 (ddd, *J* = 13.3, 10.2, 6.5 Hz, 1H), 2.80 (ddd, *J* = 13.3, 10.1, 6.5 Hz, 1H), 4.88 (sept, *J* = 6.3 Hz, 1H), 5.24 (sept, *J* = 6.3 Hz, 1H), 5.32 (sept, *J* = 6.3 Hz, 1H), 5.88 (dd, *J* = 8.2, 6.1 Hz, 1H), 9.69 (s, 1H); ¹³C NMR (CDCl₃) δ 19.0, 19.1, 24.6, 26.29, 26.34, 26.71, 26.74, 26.75, 26.80, 27.1, 27.9, 28.4, 28.6, 35.3, 36.6, 36.8, 37.6, 37.7, 39.0, 42.1, 72.9, 73.6, 74.3, 118.6, 129.2, 133.3, 133.9, 137.5, 144.3, 144.6, 145.2, 148.7, 162.1, 174.1, 174.6, 174.9. HRMS (ESI) calcd for C₃₈H₆₀O₇Na (M+Na)⁺ 651.4231, found 651.4231.



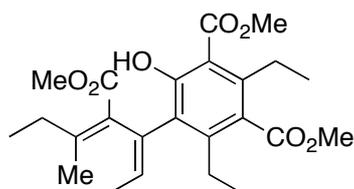
8c-(Z)

8c-(Z): ¹H NMR (CDCl₃) δ 0.87 (t, *J* = 7.2 Hz, 3H), 0.89 (t, *J* = 7.5 Hz, 3H), 0.91 (t, *J* = 7.5 Hz, 3H), 0.97 (t, *J* = 7.3 Hz, 3H), 1.08 (d, *J* = 6.3 Hz, 6H), 1.20–1.42 (m, 6H), 1.34 (d, *J* = 6.3 Hz, 6H), 1.36 (d, *J* = 6.3 Hz, 6H), 1.42–1.59 (m, 8H), 1.84 (s, 3H), 2.06 (br s, 2H), 2.23 (t, *J* = 7.8 Hz, 2H), 2.36 (br s, 1H), 2.57 (br s, 1H), 2.67 (br s, 2H), 4.88 (sept, *J* = 6.3 Hz, 1H), 5.22 (sept, *J* = 6.3 Hz, 1H), 5.30 (sept, *J* = 6.3 Hz, 1H), 5.64 (t, *J* = 7.3 Hz, 1H), 9.60 (s, 1H); ¹³C NMR (CDCl₃) δ 14.1, 14.3, 20.9, 21.6, 21.88, 21.93, 22.5, 23.1, 23.5, 23.7, 30.8, 31.8, 32.3, 32.6, 34.1, 34.2, 36.2, 68.3, 68.7, 69.3, 114.5, 127.5, 128.27, 128.29, 130.1, 138.5, 139.0, 143.2, 144.8, 157.6, 168.8, 169.87, 169.94. HRMS (ESI) calcd for C₃₈H₆₀O₇Na (M+Na)⁺ 651.4231, found 651.4221.



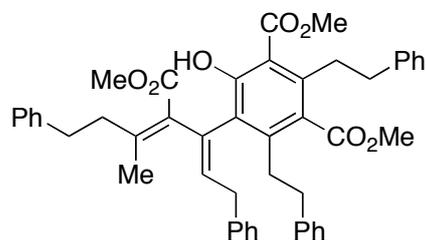
8d-(E)

8d-(E): ¹H NMR (CDCl₃) δ 1.04 (t, *J* = 7.5 Hz, 3H), 1.06 (t, *J* = 7.5 Hz, 3H), 1.17 (t, *J* = 7.5 Hz, 3H), 1.56 (d, *J* = 6.9 Hz, 3H), 1.87 (s, 3H), 2.13 (dq, *J* = 13.2, 7.5 Hz, 1H), 2.17 (dq, *J* = 13.2, 7.5 Hz, 1H), 2.33 (dq, *J* = 13.6, 7.5 Hz, 1H), 2.53 (dq, *J* = 13.6, 7.5 Hz, 1H), 2.70 (dq, *J* = 13.8, 7.5 Hz, 1H), 2.73 (dq, *J* = 13.8, 7.5 Hz, 1H), 3.57 (s, 3H), 3.87 (s, 3H), 3.94 (s, 3H), 5.97 (q, *J* = 6.9 Hz, 1H), 9.68 (s, 1H); ¹³C NMR (CDCl₃) δ 12.9, 14.9, 15.5, 15.9, 19.2, 24.8, 26.3, 30.5, 51.7, 52.0, 52.4, 113.5, 124.1, 127.9, 130.7, 131.5, 134.0, 141.6, 144.0, 145.4, 157.2, 169.9, 170.7, 170.9. HRMS (ESI) calcd for C₂₄H₃₂O₇Na (M+Na)⁺ 455.2040, found 455.2038.



8d-(Z)

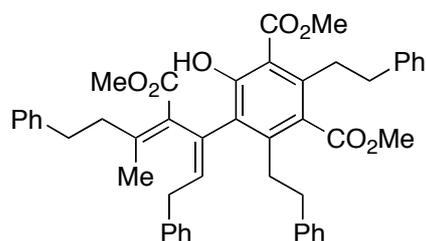
8d-(Z): ¹H NMR (CDCl₃) δ 1.07 (t, *J* = 7.5 Hz, 3H), 1.11 (t, *J* = 7.5 Hz, 3H), 1.16 (t, *J* = 7.5 Hz, 3H), 1.75 (d, *J* = 6.9 Hz, 3H), 1.87 (s, 3H), 2.29 (q, *J* = 7.5 Hz, 2H), 2.29–2.67 (m, 2H), 2.57–2.75 (m, 2H), 3.57 (s, 3H), 3.87 (s, 3H), 3.92 (s, 3H), 5.75 (q, *J* = 6.9 Hz, 1H), 9.47 (s, 1H); ¹³C NMR (CDCl₃) δ 13.3, 15.8, 15.9, 16.0, 20.5, 24.6, 26.1, 29.6, 51.7, 52.0, 52.4, 114.4, 126.5, 127.5, 127.6, 131.1, 133.4, 140.8, 144.9, 148.7, 157.5, 169.4, 170.6, 170.8. HRMS (ESI) calcd for C₂₄H₃₂O₇Na (M+Na)⁺ 455.2040, found 455.2046.



8e-(E)

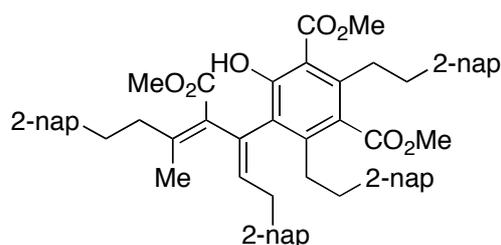
8e-(E): ¹H NMR (CDCl₃) δ 1.79 (s, 3H), 2.36 (ddd, *J* = 13.1, 10.6, 5.7 Hz, 1H), 2.45 (ddd, *J* = 13.1, 10.7, 5.8 Hz, 1H), 2.62–2.98 (m, 8H), 3.08 (ddd, *J* = 13.5, 10.0, 6.2 Hz, 1H), 3.11 (ddd, *J* = 13.5, 10.2, 7.2 Hz, 1H), 3.20 (dd, *J* = 16.3, 7.6 Hz, 1H), 3.40 (dd, *J* = 16.3, 6.7 Hz, 1H), 3.57 (s, 3H), 3.91 (s, 3H), 4.00 (s, 3H), 6.10 (dd, *J* = 7.6, 6.7 Hz, 1H), 7.09–7.34 (m, 20H), 10.68 (s, 1H); ¹³C NMR (CDCl₃) δ 20.1, 33.5, 34.8, 35.7, 36.3, 36.4, 38.1, 39.9, 51.7, 52.4, 52.7, 112.5, 125.1, 126.0, 126.1, 126.2, 128.2, 128.3, 128.4, 128.5, 128.6, 128.65, 128.70,

128.8, 130.1, 132.5, 137.5, 139.9, 140.2, 141.1, 141.7, 141.8, 142.0, 144.1, 159.0, 169.8, 170.5, 171.1. HRMS (ESI) calcd for $C_{48}H_{48}O_7Na$ ($M+Na$)⁺ 759.3292, found 759.3265.



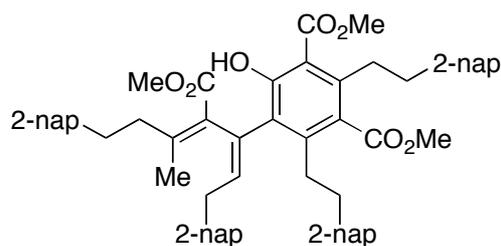
8e-(Z)

8e-(Z): ¹H NMR (CDCl₃) δ 1.94 (s, 3H), 2.46–3.17 (m, 10H), 2.84 (t, *J* = 8.0 Hz, 2H), 3.38 (d, *J* = 7.1 Hz, 2H), 3.55 (s, 3H), 3.88 (s, 3H), 3.97 (s, 3H), 5.86 (t, *J* = 7.1 Hz, 1H), 6.77–6.83 (m, 2H), 7.10–7.34 (m, 18H), 10.69 (s, 1H); ¹³C NMR (CDCl₃) δ 21.4, 33.4, 34.9, 35.6, 36.4, 37.7, 37.9, 38.2, 51.5, 52.2, 52.5, 112.0, 125.8, 125.9, 126.1, 126.2, 127.6, 127.9, 128.06, 128.14, 128.28, 128.35, 128.39, 128.5, 128.7, 128.8, 130.5, 136.0, 139.3, 139.8, 141.6, 142.0, 143.5, 146.4, 160.0, 169.0, 170.6, 171.1. HRMS (ESI) calcd for $C_{48}H_{48}O_7Na$ ($M+Na$)⁺ 759.3292, found 759.329



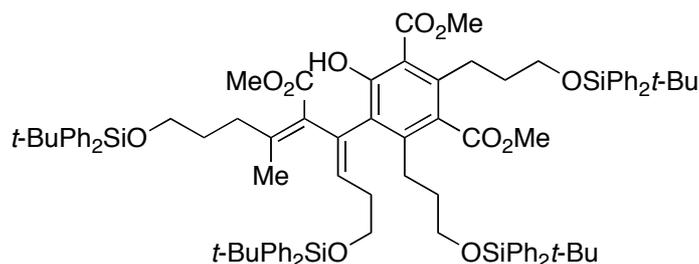
8f-(E)

8f-(E): ¹H NMR (CDCl₃) δ 1.81 (s, 3H), 2.47 (ddd, *J* = 13.2, 10.3, 5.8 Hz, 1H), 2.56 (ddd, *J* = 13.2, 10.3, 5.9 Hz, 1H), 2.74–3.05 (m, 6H), 3.05–3.15 (m, 2H), 3.15–3.27 (m, 2H), 3.40 (dd, *J* = 16.3, 7.8 Hz, 1H), 3.55 (s, 3H), 3.59 (dd, *J* = 16.3, 6.6 Hz, 1H), 3.90 (s, 3H), 4.02 (s, 3H), 6.22 (dd, *J* = 7.8, 6.6 Hz, 1H), 7.21 (dd, *J* = 8.4, 1.6 Hz, 1H), 7.23–7.27 (m, 1H), 7.30–7.48 (m, 11H), 7.53 (s, 1H), 7.60 (s, 1H), 7.62 (s, 1H), 7.64–7.84 (m, 12H), 10.60 (s, 1H); ¹³C NMR (CDCl₃) δ 20.2, 33.3, 34.9, 35.5, 36.50, 36.54, 38.2, 39.7, 51.8, 52.4, 52.8, 112.9, 125.2, 125.3, 125.4, 125.47, 125.48, 126.00, 126.04, 126.05, 126.12, 126.2, 126.4, 126.5, 126.9, 127.0, 127.2, 127.3, 127.48, 127.53, 127.59, 127.60, 127.69, 127.74, 127.8, 128.1, 128.2, 128.3, 128.9, 130.4, 132.17, 132.21, 132.28, 132.30, 132.7, 133.73, 133.74, 133.80, 133.84, 137.4, 137.5, 139.2, 139.3, 139.5, 140.3, 141.5, 144.1, 158.9, 169.8, 170.5, 171.1. HRMS (ESI) calcd for $C_{64}H_{56}O_7Na$ ($M+Na$)⁺ 959.3918, found 959.3885.



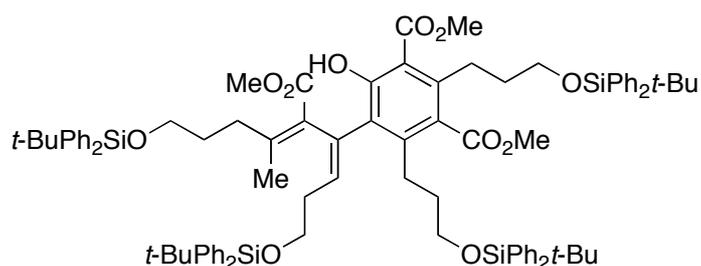
8f-(Z)

8f-(Z): ¹H NMR (CDCl₃) δ 2.01 (s, 3H), 2.60–3.30 (m, 12H), 3.49 (d, *J* = 7.2 Hz, 2H), 3.55 (s, 3H), 3.87 (s, 3H), 3.99 (s, 3H), 5.95 (t, *J* = 7.3 Hz, 1H), 6.68 (dd, *J* = 8.3, 1.6 Hz, 1H), 7.20–7.84 (m, 27H), 10.71 (s, 1H); ¹³C NMR (CDCl₃) δ 21.7, 33.3, 35.1, 35.6, 36.8, 38.0, 38.1, 38.2, 51.6, 52.4, 52.7, 112.2, 125.2, 125.3, 125.4, 125.8, 125.9, 126.0, 126.1, 126.2, 126.3, 126.7, 126.96, 126.98, 127.13, 127.39, 127.44, 127.57, 127.59, 127.61, 127.64, 127.70, 127.78, 127.79, 127.83, 127.9, 128.0, 128.06, 128.08, 128.2, 128.5, 128.7, 131.0, 132.1, 132.2, 132.3, 132.4, 133.6, 133.8, 133.86, 133.89, 136.0, 137.3, 139.15, 139.17, 139.60, 139.64, 143.8, 146.7, 160.3, 169.0, 170.8, 171.2. HRMS (ESI) calcd for C₆₄H₅₆O₇Na (M+Na)⁺ 959.3918, found 959.3931.



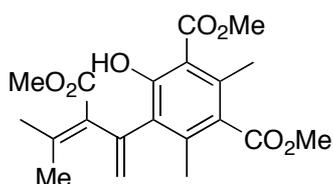
8g-(E)

8g-(E): ¹H NMR (CDCl₃) δ 1.01 (s, 9H), 1.03 (s, 9H), 1.04 (s, 9H), 1.07 (s, 9H), 1.56–1.82 (m, 4H), 1.72 (s, 3H), 1.82–1.92 (m, 2H), 2.00–2.16 (m, 2H), 2.16–2.28 (m, 2H), 2.45 (td, *J* = 12.7, 4.3 Hz, 1H), 2.56 (td, *J* = 12.7, 4.6 Hz, 1H), 2.69–2.87 (m, 2H), 3.43 (s, 3H), 3.53 (dd, *J* = 10.0, 6.3 Hz, 1H), 3.57 (dd, *J* = 10.0, 6.3 Hz, 1H), 3.60–3.72 (m, 6H), 3.70 (s, 3H), 3.78 (s, 3H), 5.96 (t, *J* = 6.9 Hz, 1H), 7.28–7.47 (m, 24H), 7.56–7.74 (m, 16H), 9.83 (s, 1H); ¹³C NMR (CDCl₃) δ 19.3, 19.36, 19.39, 19.44, 19.9, 26.96, 27.01, 27.03, 27.1, 28.7, 30.3, 31.4, 33.2, 33.5, 34.2, 34.7, 51.6, 52.1, 52.4, 63.1, 64.0, 64.3, 64.5, 113.7, 124.6, 127.75, 127.76, 127.78, 127.79, 128.5, 129.68, 129.73, 130.8, 132.2, 133.7, 133.8, 134.0, 134.07, 134.08, 134.09, 135.6, 135.66, 135.69, 135.70, 135.72, 136.0, 140.0, 141.8, 143.7, 157.3, 169.6, 170.3, 170.8. HRMS (ESI) calcd for C₉₂H₁₁₂O₁₁Si₄Na (M+Na)⁺ 1527.7174, found 1527.7173.



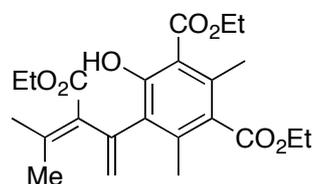
8g-(Z)

8g-(Z): ¹H NMR (CDCl₃) δ 1.01 (s, 9H), 1.04 (s, 9H), 1.05 (s, 9H), 1.07 (s, 9H), 1.59–1.94 (m, 6H), 1.68 (s, 3H), 2.03–2.87 (m, 8H), 3.40 (s, 3H), 3.56 (t, *J* = 5.9 Hz, 2H), 3.60–3.74 (m, 6H), 3.68 (s, 3H), 3.77 (s, 3H), 5.71 (t, *J* = 7.0 Hz, 1H), 7.26–7.46 (m, 24H), 7.56–7.74 (m, 16H), 9.24 (s, 1H); ¹³C NMR (CDCl₃) δ 19.3, 19.39, 19.43, 21.3, 26.98, 27.02, 27.04, 28.5, 29.9, 31.8, 33.1, 33.6, 34.5, 34.6, 51.5, 52.1, 52.3, 63.3, 64.0, 64.3, 64.5, 115.2, 127.3, 127.7, 127.8, 127.9, 129.7, 129.8, 131.8, 133.8, 134.0, 134.08, 134.11, 135.70, 135.73, 139.0, 142.8, 147.5, 157.0, 168.6, 170.3, 170.4. HRMS (ESI) calcd for C₉₂H₁₁₂O₁₁Si₄Na (M+Na)⁺ 1527.7174, found 1527.7173.



8h

8h: ¹H NMR (CDCl₃) δ 1.94 (s, 3H), 1.95 (s, 3H), 2.24 (s, 3H), 2.38 (s, 3H), 3.58 (s, 3H), 3.89 (s, 3H), 3.93 (s, 3H), 5.30 (d, *J* = 1.8 Hz, 1H), 5.50 (d, *J* = 1.8 Hz, 1H), 10.91 (s, 1H); ¹³C NMR (CDCl₃) δ 17.8, 20.1, 22.9, 23.6, 51.5, 52.3, 52.4, 111.5, 123.5, 127.8, 128.9, 130.3, 135.7, 139.2, 139.5, 143.0, 159.7, 169.3, 170.8, 171.6. HRMS (ESI) calcd for C₂₀H₂₄O₇Na (M+Na)⁺ 399.1414, found 399.1417.

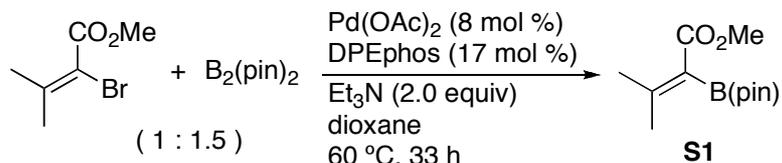


8i

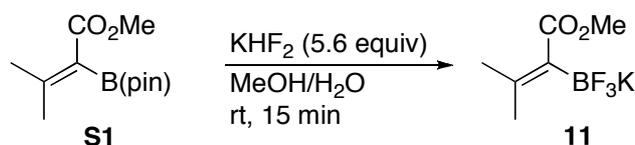
8i: ¹H NMR (CDCl₃) δ 1.12 (t, *J* = 7.1 Hz, 3H), 1.37 (t, *J* = 7.1 Hz, 3H), 1.40 (t, *J* = 7.1 Hz, 3H), 1.96 (s, 6H), 2.25 (s, 3H), 2.42 (s, 3H), 4.05 (q, *J* = 7.1 Hz, 2H), 4.37 (q, *J* = 7.1 Hz, 2H), 4.41 (q, *J* = 7.1 Hz, 2H), 5.31 (s, 1H), 5.51 (s, 1H), 10.94 (s, 1H); ¹³C NMR (CDCl₃) δ 13.9, 14.20, 14.22, 17.9, 19.9, 22.9, 23.4, 60.5, 61.3, 61.8, 111.6, 123.4, 127.7, 129.0, 130.6, 135.4, 139.07, 139.13, 142.2, 159.5, 168.8, 170.2, 171.1. HRMS (ESI) calcd for C₂₃H₃₀O₇Na (M+Na)⁺ 441.1884, found 441.1872.

171.2. HRMS (ESI) calcd for $C_{51}H_{54}O_7Na$ ($M+Na$)⁺ 801.3762, found 801.3762.

Preparation of Potassium Alkenyltrifluoroborate **11**.



Pd(OAc)_2 (90 mg, 0.40 mmol) and DPEphos (441 mg, 0.82 mmol) were dissolved in 1,4-dioxane (10 mL), and the mixture was stirred at room temperature for 10 min. To the mixture were added successively triethylamine (1.4 mL, 10 mmol), methyl 2-bromo-3-methyl-2-butenoate⁴ (0.95 g, 4.9 mmol), and bis(pinacolato)diboron (1.1 mL, 7.5 mmol), and the mixture was heated at 60 °C for 33 h. The mixture was filtered through a plug of silica gel. The plug was washed with Et_2O (50 mL) and the combined filtrates were concentrated on a rotary evaporator. The residue was purified by flash column chromatography on silica gel eluting with EtOAc /hexane (1/9) to give **S1** (0.31 g, 26% yield). ¹H NMR (CDCl_3) δ 1.30 (s, 12H), 1.99 (s, 3H), 2.06 (s, 3H), 3.70 (s, 3H); ¹³C NMR (CDCl_3) δ 23.4, 24.7, 25.8, 51.2, 83.8, 161.5, 169.9. HRMS (ESI) calcd for $C_{12}H_{21}BO_4Na$ ($M+Na$)⁺ 263.1427, found 263.1421.



To a solution of **S1** (0.30 g, 1.2 mmol) in methanol (2.0 mL) was added a solution of KHF_2 (0.52 g, 6.7 mmol) in H_2O (1.5 mL), and the mixture was stirred at room temperature for 15 min. The solvent was removed under reduced pressure, and the residue was extracted with hot acetone (3×3.0 mL). The combined extracts were concentrated on a rotary evaporator to give a white solids. The collected solids were washed with Et_2O (3×2.0 mL) to give **11** (179 mg, 73% yield). ¹H NMR (acetone-*d*₆, the chemical shifts are referenced to residual acetone (2.05 ppm)) δ 1.57 (s, 3H), 1.76 (s, 3H), 3.58 (s, 3H); ¹³C NMR (acetone-*d*₆, the chemical shifts are referenced to residual acetone (29.84 ppm)) δ 22.5, 23.6, 50.6, 138.8, 177.5. HRMS (ESI) calcd for $C_6H_9BF_3O_2Na_2$ ($M-K+2Na$)⁺ 227.0439, found 227.0439.

Procedure for Rhodium-Catalyzed Addition of Potassium Alkenyltrifluoroborate **11 to Methyl 2-Butynoate (**1h**) (Scheme 7).** To a solution of $[\text{Rh(OH)(cod)}]_2$ (1.7 mg, 0.0076 mmol Rh) and **11** (61.8 mg, 0.30 mmol) in 1,4-dioxane (0.30 mL) and H_2O (30 μL) was added methyl 2-butynoate (**1h**) (14.5 mg, 0.15 mmol). After stirring at 70 °C for 3 h, the mixture was diluted with Et_2O (3.0 mL), and filtered through a plug of silica gel. The plug was washed with Et_2O (20 mL) and the combined filtrates were concentrated on a rotary evaporator. The residue was purified by preparative TLC on silica gel eluting with EtOAc /hexane (1/7) to give **8h** (7.9 mg, 0.021 mmol, 43% yield).

Data for X-ray Crystal Structure of 8e-(Z). Colorless crystals of **8e-(Z)** suitable for X-ray crystallographic analysis were obtained by recrystallization from CH₂Cl₂/hexane. The ORTEP drawing of **8e-(Z)** is shown in Figure S1. The crystal structure has been deposited at the Cambridge Crystallographic Centre (deposition number: CCDC 746346). The data can be obtained free of charge via www.ccdc.cam.ac.uk/data_request/cif.

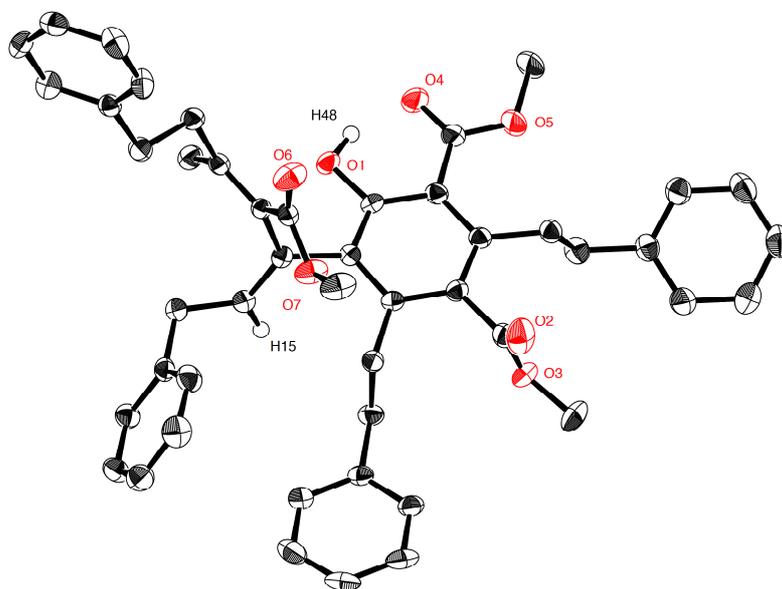


Figure S1. ORTEP illustration of **8e-(Z)** with thermal ellipsoids drawn at 50% probability level (Hydrogen atoms are omitted for clarity except for phenol and vinyl hydrogens).

Table S1. Crystal Data of **8e-(Z)**

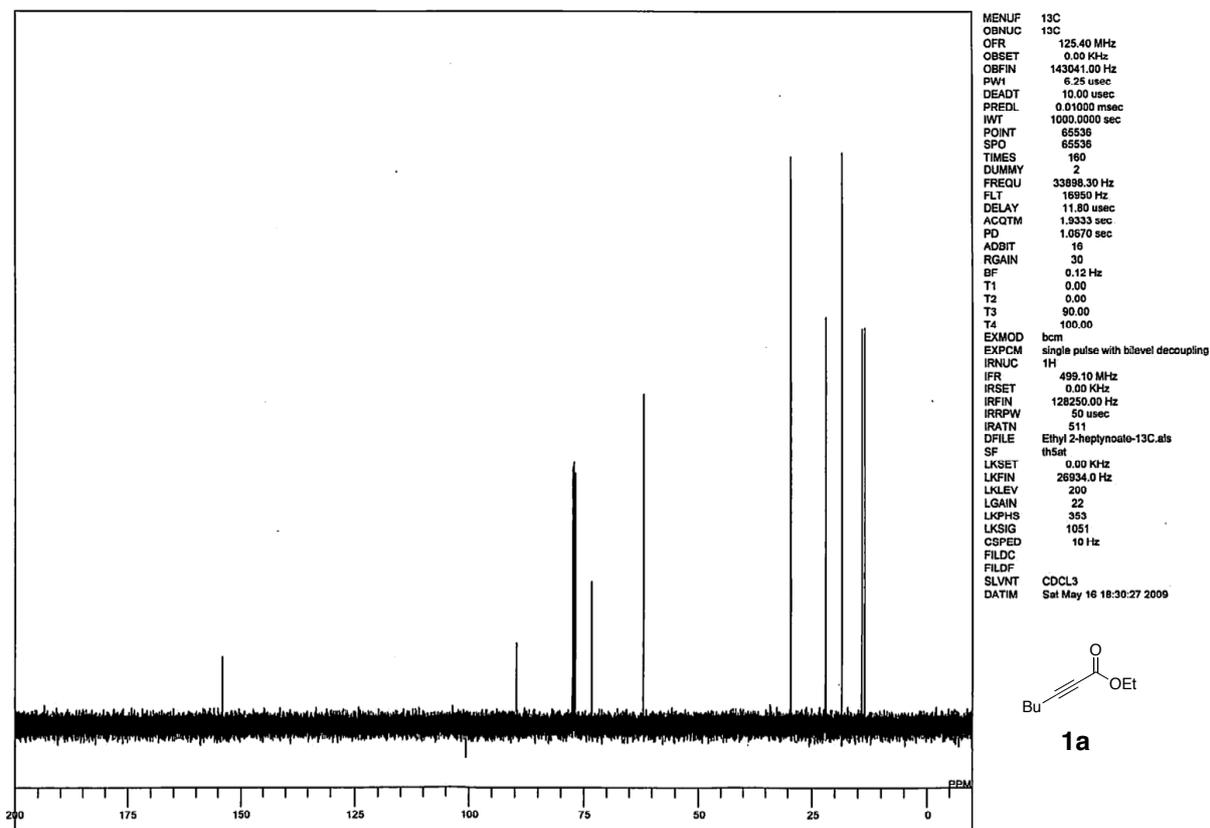
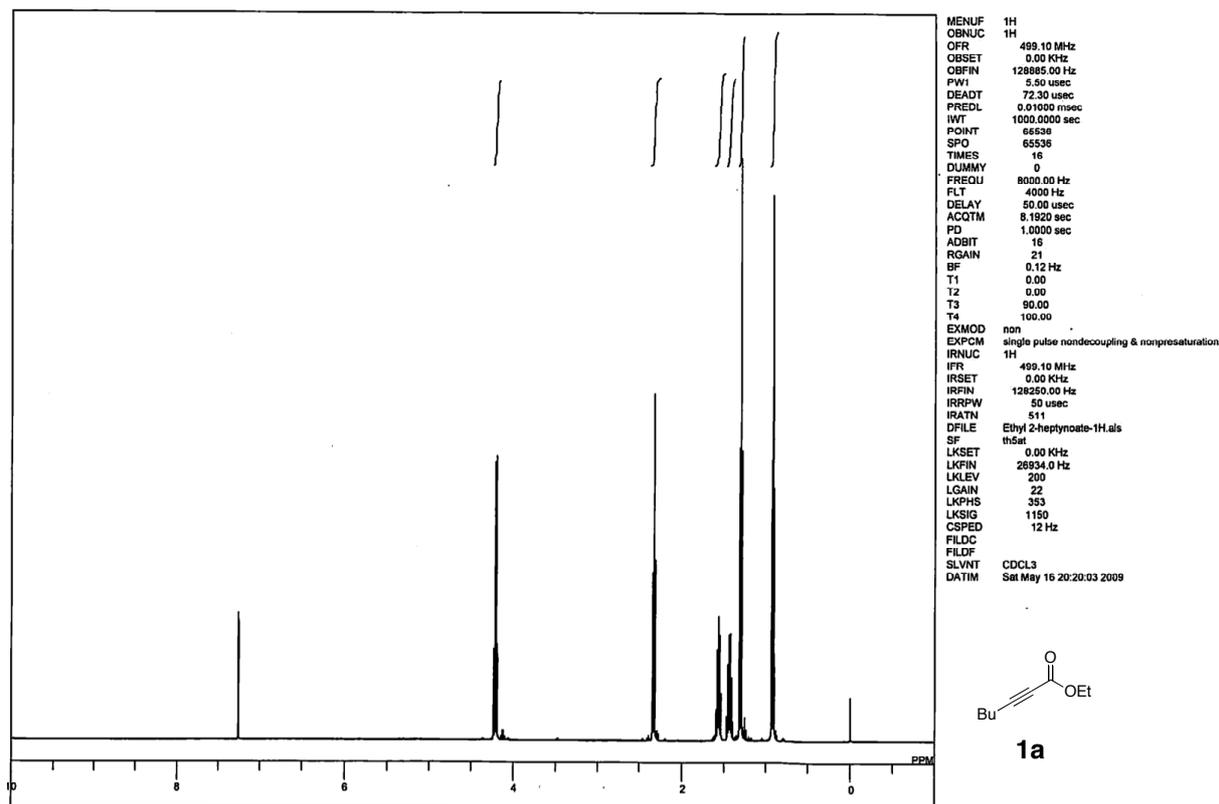
Empirical Formula	C ₄₈ H ₄₈ O ₇
Formula Weight	736.90
Crystal Color, Habit	colorless, prism
Crystal Dimensions	0.30 × 0.20 × 0.10 mm
Crystal System	monoclinic
Lattice Type	Primitive
Indexing Images	3 oscillations at 90.0 seconds
Detector Position	127.40 mm
Pixel Size	0.200 mm
Lattice Parameters	a = 20.104(5) Å b = 9.780(2) Å c = 20.524(4) Å β = 97.459(10)° V = 4001.5(15) Å ³
Space Group	P2 ₁ /c (#14)
Z value	4
D _{calc}	1.223 g/cm ³
F ₀₀₀	1568.00
μ(MoKα)	0.808 cm ⁻¹

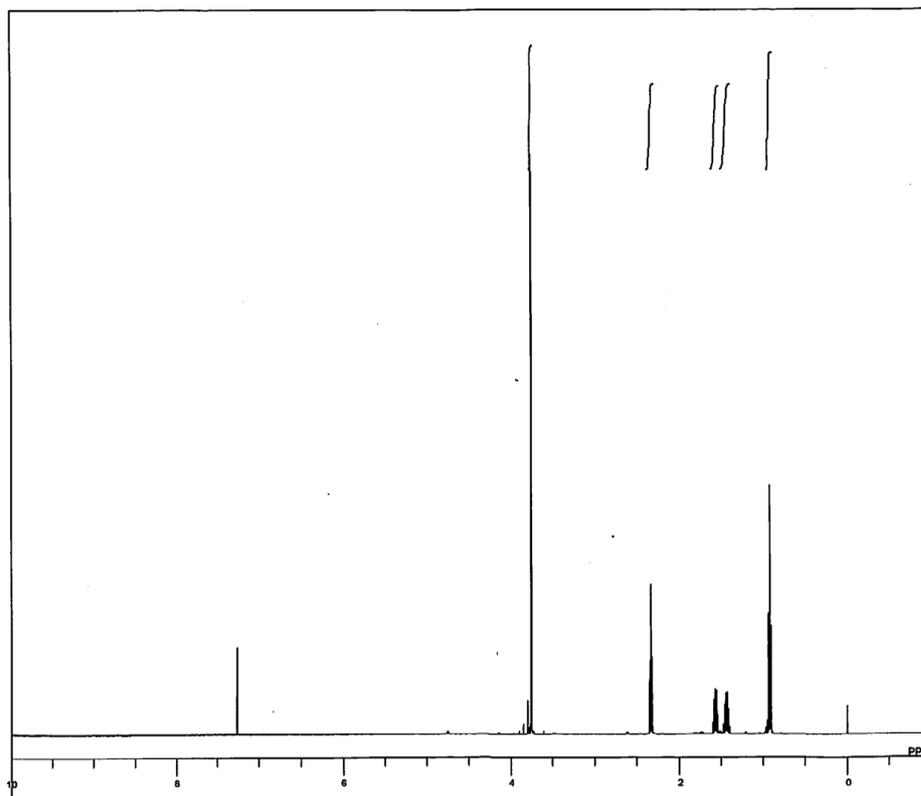
Table S2. Intensity Measurements

Diffractometer	Rigaku RAXIS-RAPID
Radiation	MoK α ($\lambda = 0.71075 \text{ \AA}$) graphite monochromated
Detector Aperture	280 nm \times 256 nm
Data Images	55 exposures
ω oscillation Range ($\chi = 45.0, \phi = 30.0$)	130.0 – 190.0°
Exposure Rate	200.0 sec./°
ω oscillation Range ($\chi = 45.0, \phi = 210.0$)	0.0 – 160.0°
Exposure Rate	200.0 sec./°
Detector Position	127.40 mm
Pixel Size	0.200 mm
$2\theta_{\max}$	55.0°
No. of Reflections Measured	Total: 36067 Unique: 9129 ($R_{\text{int}} = 0.087$)
Corrections	Lorentz-polarization

Table S3. Structure Solution and Refinement

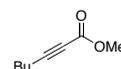
Structure Solution	Direct Methods (SIR92)
Refinement	Full-matrix least-squares on F^2
Function Minimized	$\Sigma w (F_o^2 - F_c^2)^2$
Least Squares Weights	$w = 1/[\sigma^2(F_o^2) + (0.0698 \cdot P)^2 + 2.0178 \cdot P]$ where $P = (\text{Max}(F_o^2, 0) + 2F_c^2)/3$
$2\theta_{\max}$ cutoff	55.0°
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (All reflections)	9129
No. Variables	501
Reflection/Parameter Ratio	18.22
Residuals: R1 ($I > 2.00\sigma(I)$)	0.0662
Residuals: R (All reflections)	0.1235
Residuals: wR2 (All reflections)	0.1736
Goodness of Fit Indicator	1.026
Max Shift/Error in Final Cycle	0.001
Maximum peak in Final Diff. Map	0.40 e/ \AA^3
Minimum peak in Final Diff. Map	-0.44 e/ \AA^3



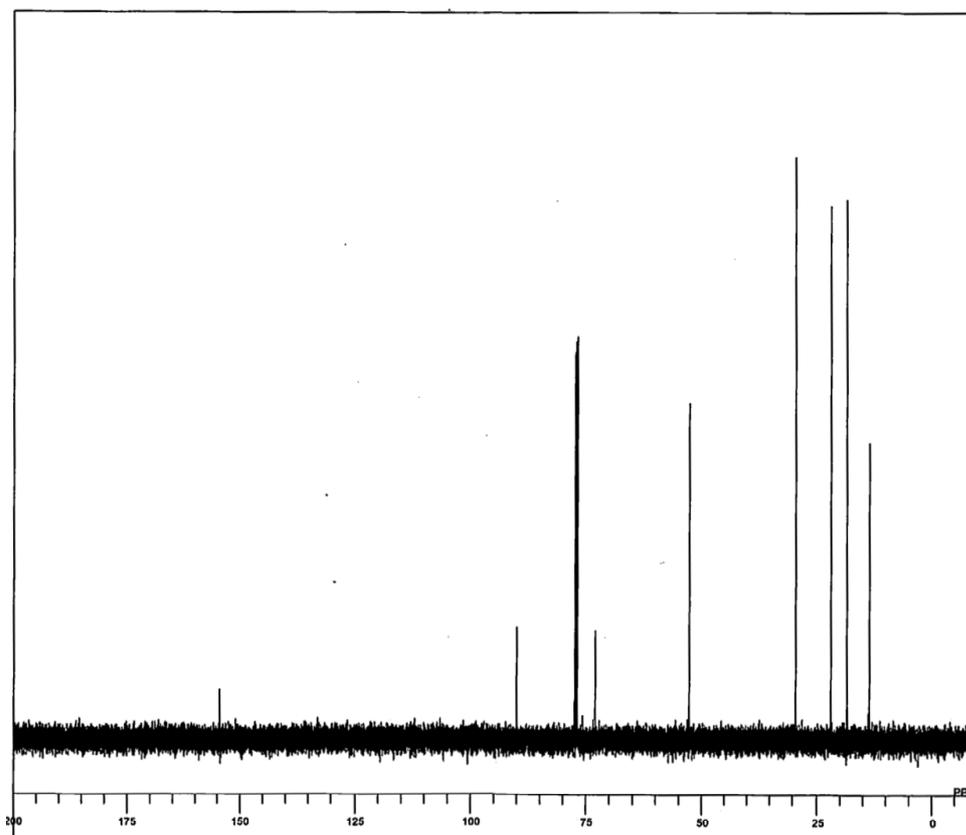


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OBSET 0.00 KHz
OBFIN 128885.00 Hz
PW1 5.50 usec
DEADT 72.30 usec
PREDL 0.01000 msec
IWT 1000.0000 sec
POINT 65536
SPO 65536
TIMES 16
DUMMY 0
FREQU 8000.00 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 8.1920 sec
PD 1.0000 sec
ADBIT -16
RGAIN 22
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD non
EXPCM single pulse nondecoupling & nonpresaturation
IRNUC 1H
IR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE Methyl 2-heptynoate-1H.als
SF thSet
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 19
LKPHS 353
LKSIG 595
CSPED 13 Hz
FILDC
FILDF
SLVNT CDCL3
DATIM Sat May 16 18:03:35 2009
    
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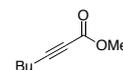


1b

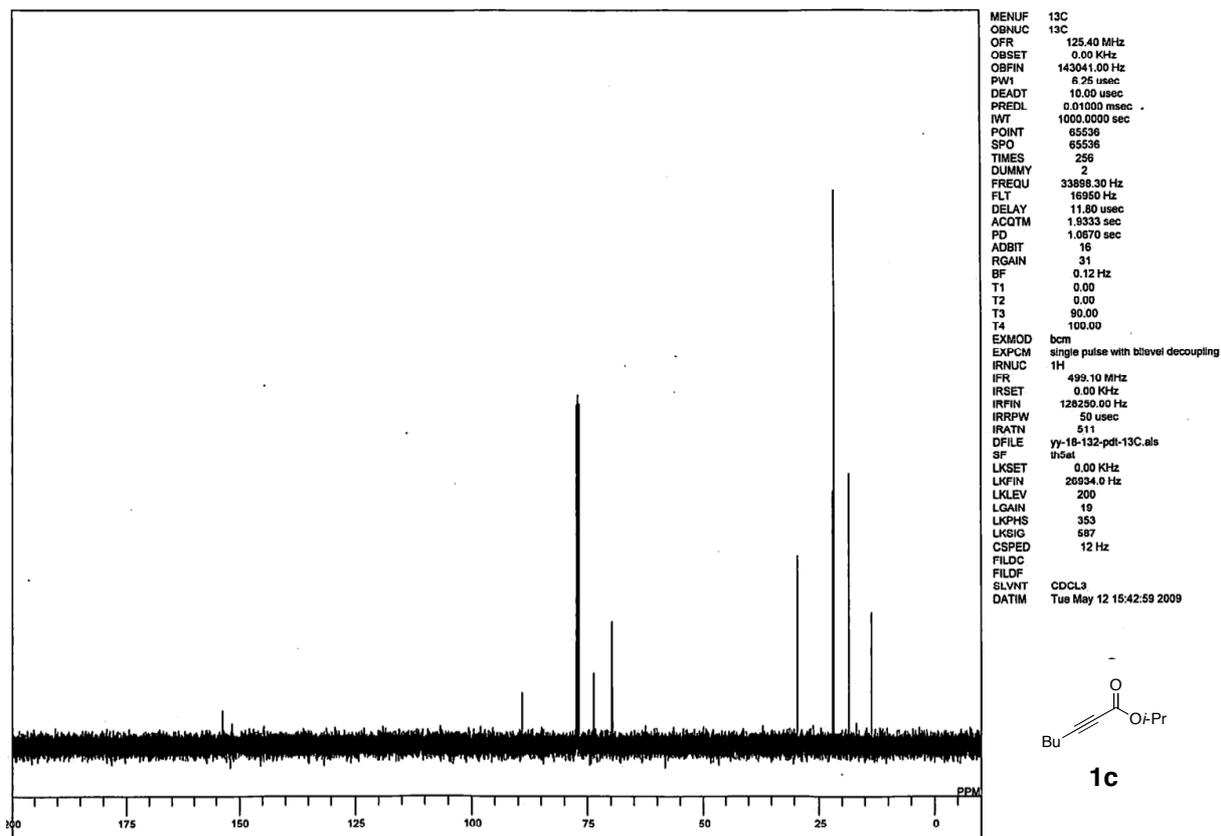
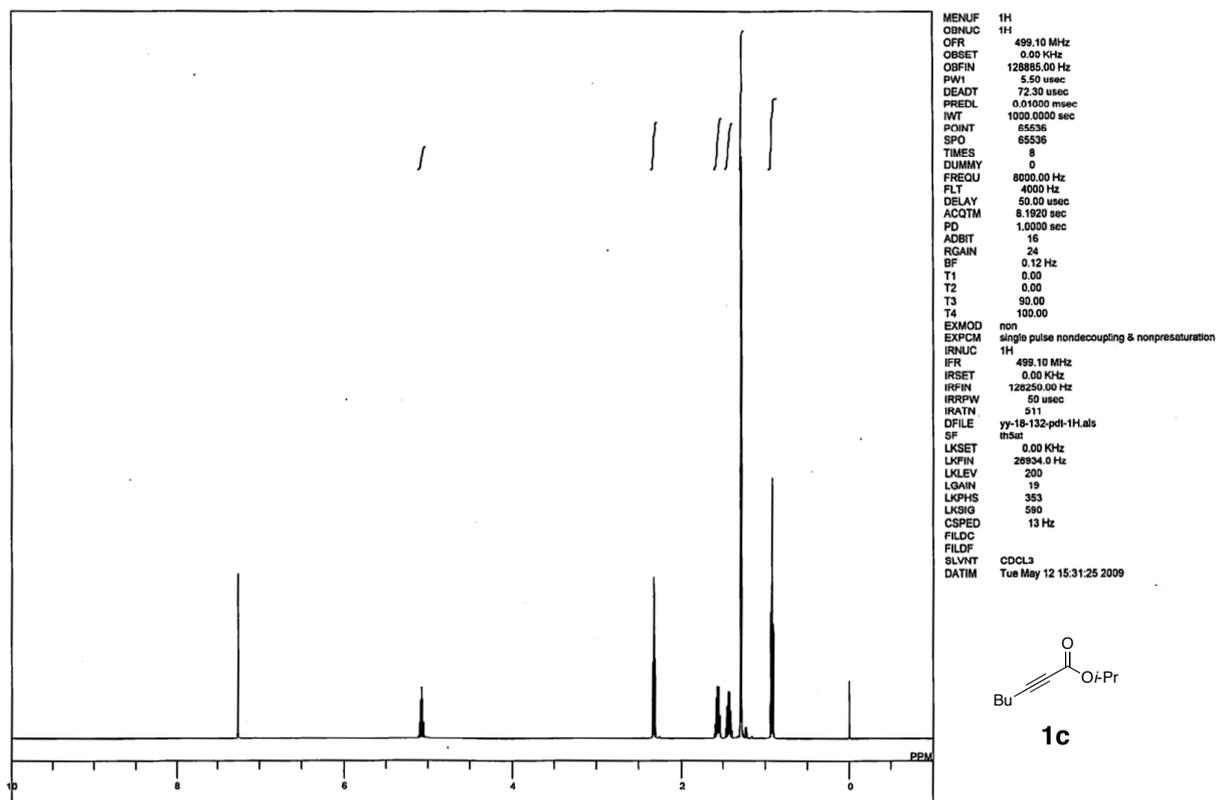


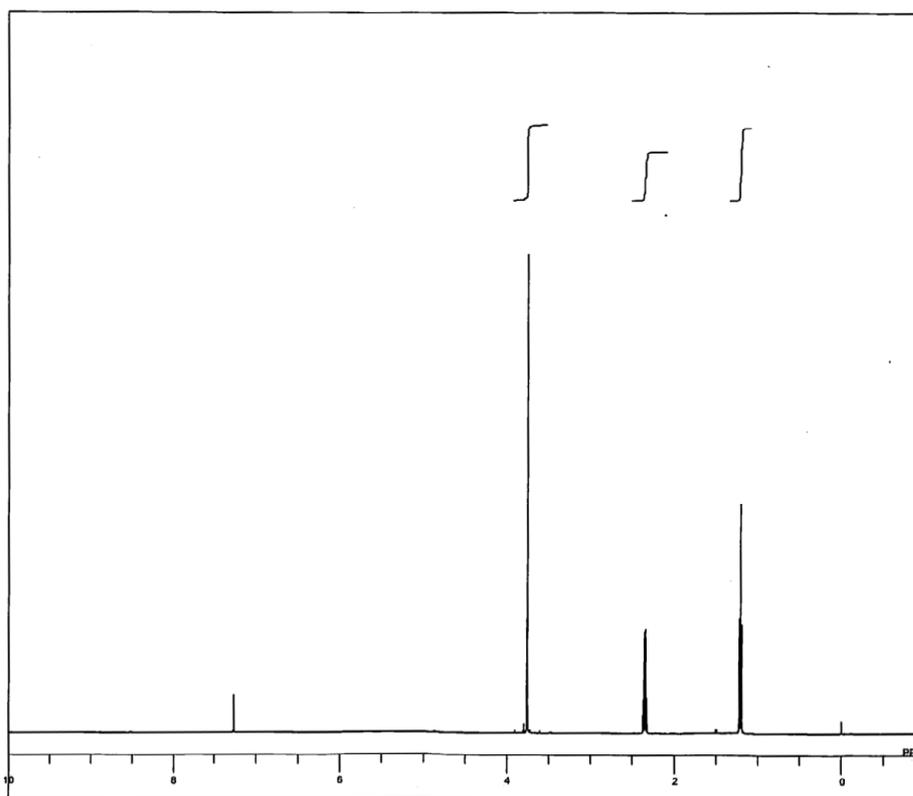
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MENUM 13C
OBNUC 13C
OFR 125.40 MHz
OBSET 0.00 KHz
OBFIN 143041.00 Hz
PW1 6.25 usec
DEADT 10.00 usec
PREDL 0.01000 msec
IWT 1000.0000 sec
POINT 65536
SPO 65536
TIMES 256
DUMMY 2
FREQU 33898.30 Hz
FLT 16950 Hz
DBLAY 11.80 usec
ACQTM 1.9333 sec
PD 1.0670 sec
ADBIT 16
RGAIN 31
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD bcm
EXPCM single pulse with bilevel decoupling
IRNUC 1H
IR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE Methyl 2-heptynoate-13C.als
SF thSet
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 19
LKPHS 353
LKSIG 589
CSPED 12 Hz
FILDC
FILDF
SLVNT CDCL3
DATIM Sat May 16 18:14:19 2009
    
```

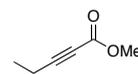


1b

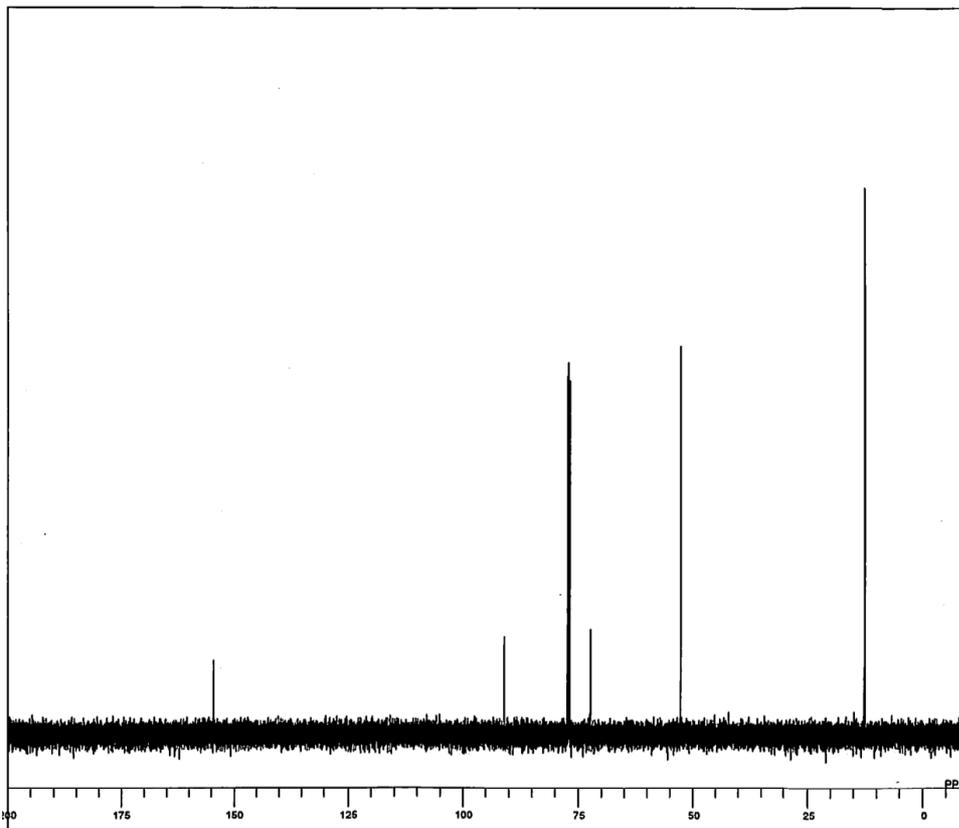




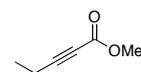
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MENUF 1H
OBNUC 1H
OFR 499.10 MHz
OBSET 0.00 KHz
OBFIN 128895.00 Hz
PW1 5.50 usec
DEADT 72.30 usec
PREDL 0.01000 msec
IWT 1000.0000 sec
POINT 65536
SPO 65536
TIMES 16
DUMMY 0
FREQU 8000.00 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 8.1920 sec
PD 1.0000 sec
ADBIT 16
RGAIN 21
SF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD non
EXPCM single pulse nondecoupling & nonpresaturation
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE Methyl 2-pentynoate-1H.tals
SF thsai
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 20
LKPHS 353
LKSIG 670
CSPED 14 Hz
FILDC
FILDF
SLVNT CDCL3
DATIM Sat May 16 20:27:28 2009
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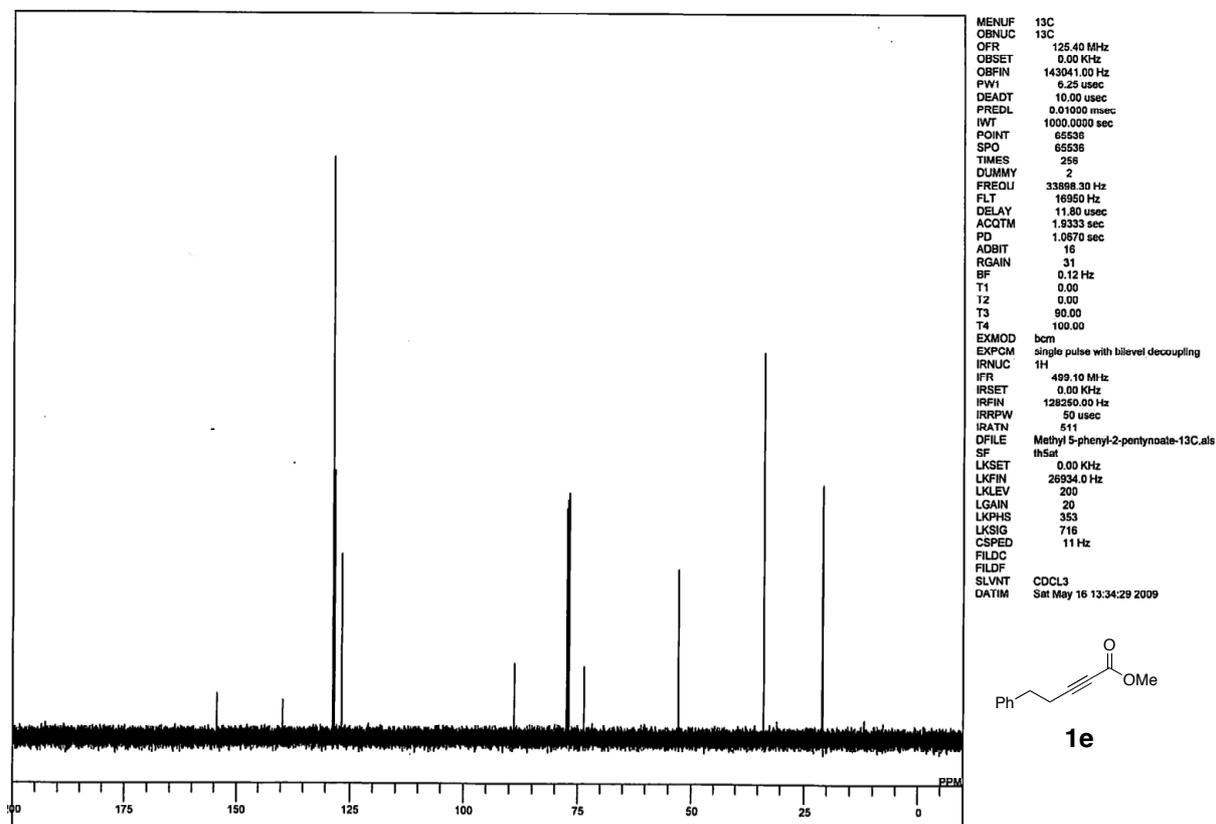
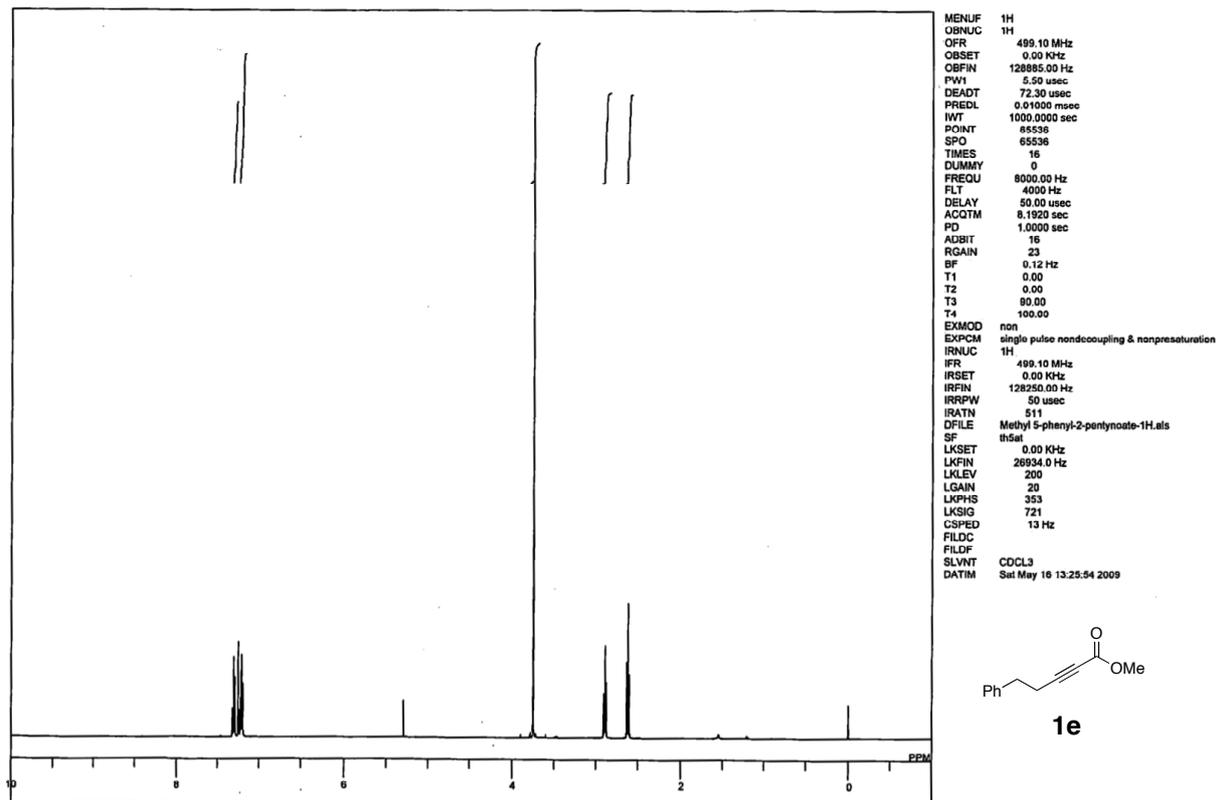
1d

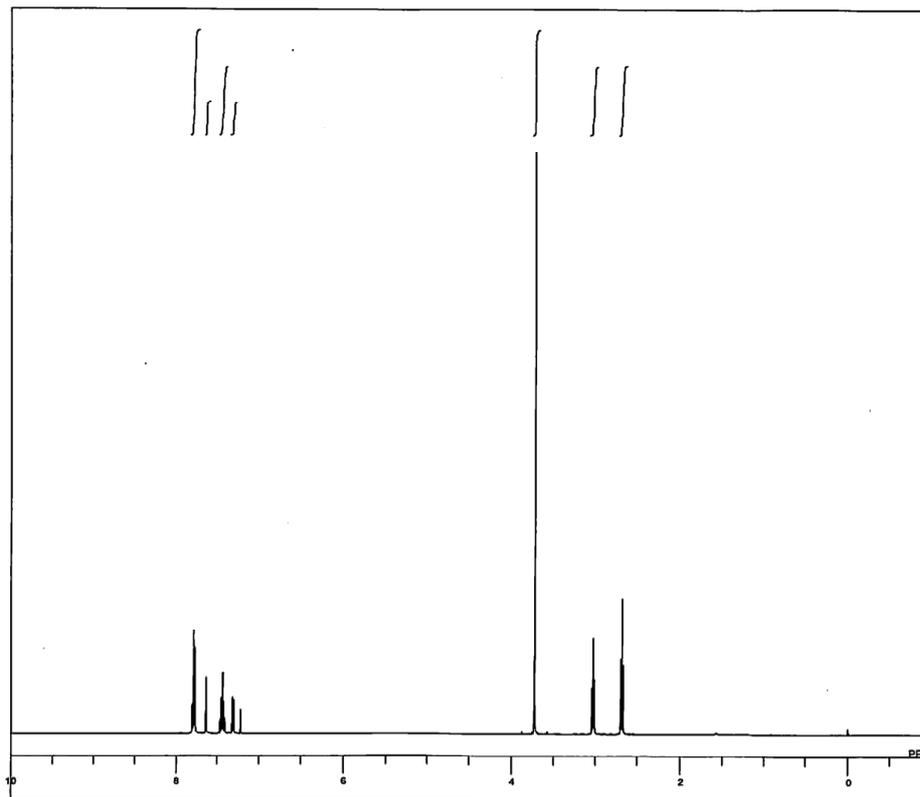


```
MENUF 13C
OBNUC 13C
OFR 125.40 MHz
OBSET 0.00 KHz
OBFIN 143041.00 Hz
PW1 6.25 usec
DEADT 10.00 usec
PREDL 0.01000 msec
IWT 1000.0000 sec
POINT 65536
SPO 65536
TIMES 256
DUMMY 2
FREQU 33898.30 Hz
FLT 16950 Hz
DELAY 11.80 usec
ACQTM 1.9333 sec
PD 1.0670 sec
ADBIT 16
RGAIN 29
SF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD bcm
EXPCM single pulse with bilevel decoupling
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE Methyl 2-pentynoate-13C.tals
SF thsai
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 20
LKPHS 353
LKSIG 669
CSPED 14 Hz
FILDC
FILDF
SLVNT CDCL3
DATIM Sat May 16 20:36:45 2009
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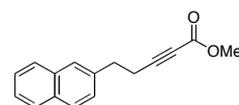
1d



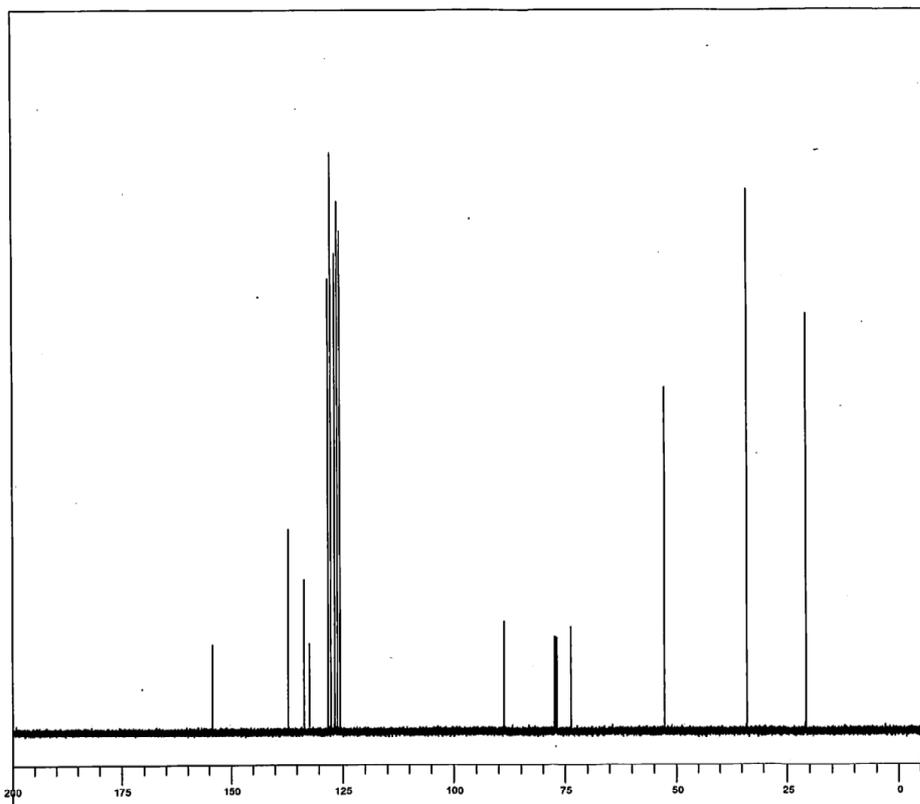


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MENUF 1H
OBNUC 1H
OFR 499.10 MHz
OBSET 0.00 KHz
OBFIN 128250.00 Hz
PW1 5.50 usec
DEADT 72.30 usec
FREDL 0.01000 msec
IWT 1000.0000 sec
POINT 65536
SPO 65536
TIMES 16
DUMMY 0
FREQU 8000.00 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 8.1920 sec
PD 1.0000 sec
ADBIT 16
RGAIN 17
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD non
EXPCM single pulse nondecoupling & nonpresaturation
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE Methyl 5-(2-naphthyl)-2-pentynoate-1H.als
SF thSat
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 20
LKPHS 353
LKSIG 664
CSPED 11 Hz
FILDC
FILDF
SLVNT CDCL3
DATIM Thu May 28 12:00:05 2009
    
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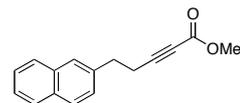


1f

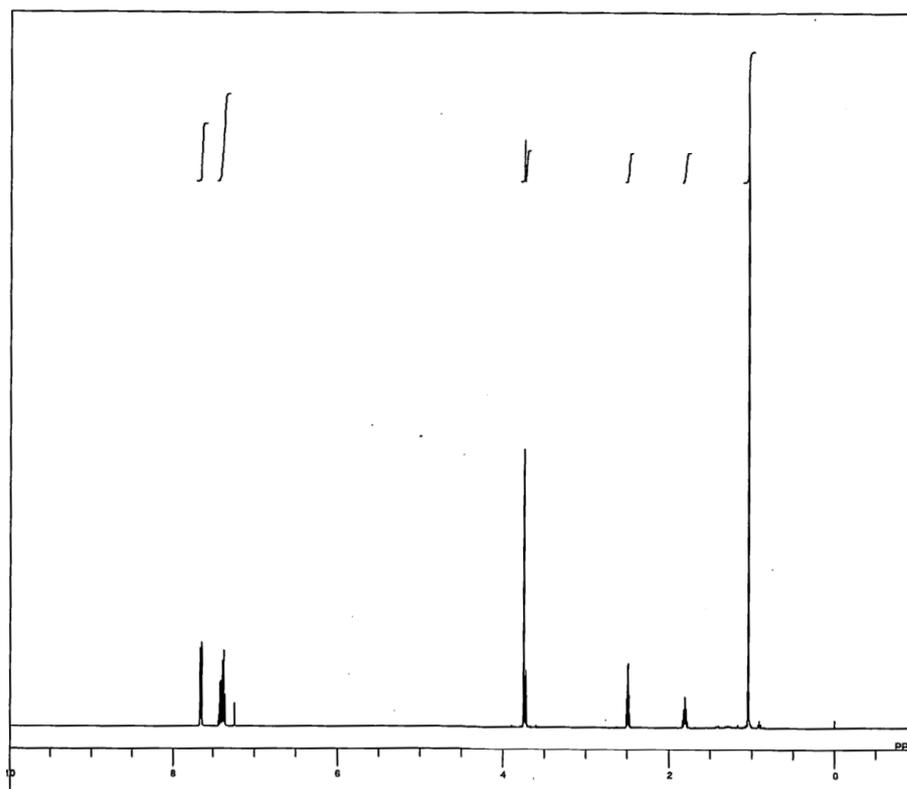


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MENUF 13C
OBNUC 13C
OFR 125.40 MHz
OBSET 0.00 KHz
OBFIN 143041.00 Hz
PW1 6.25 usec
DEADT 10.00 usec
FREDL 0.01000 msec
IWT 1000.0000 sec
POINT 65536
SPO 65536
TIMES 256
DUMMY 2
FREQU 33898.30 Hz
FLT 16950 Hz
DELAY 11.60 usec
ACQTM 1.9333 sec
PD 1.0670 sec
ADBIT 16
RGAIN 31
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD bcm
EXPCM single pulse with bilevel decoupling
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE Methyl 5-(2-naphthyl)-2-pentynoate-13C.als
SF thSat
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 20
LKPHS 353
LKSIG 663
CSPED 14 Hz
FILDC
FILDF
SLVNT CDCL3
DATIM Thu May 28 12:09:24 2009
    
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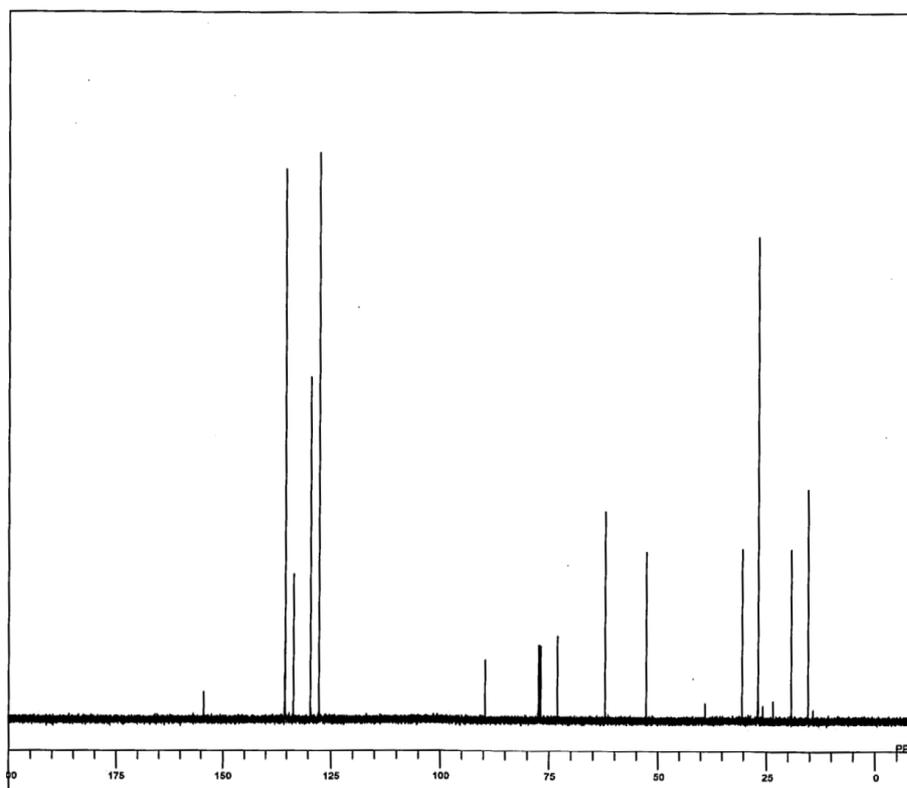
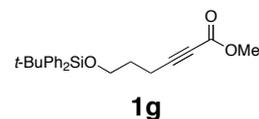


1f



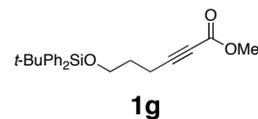
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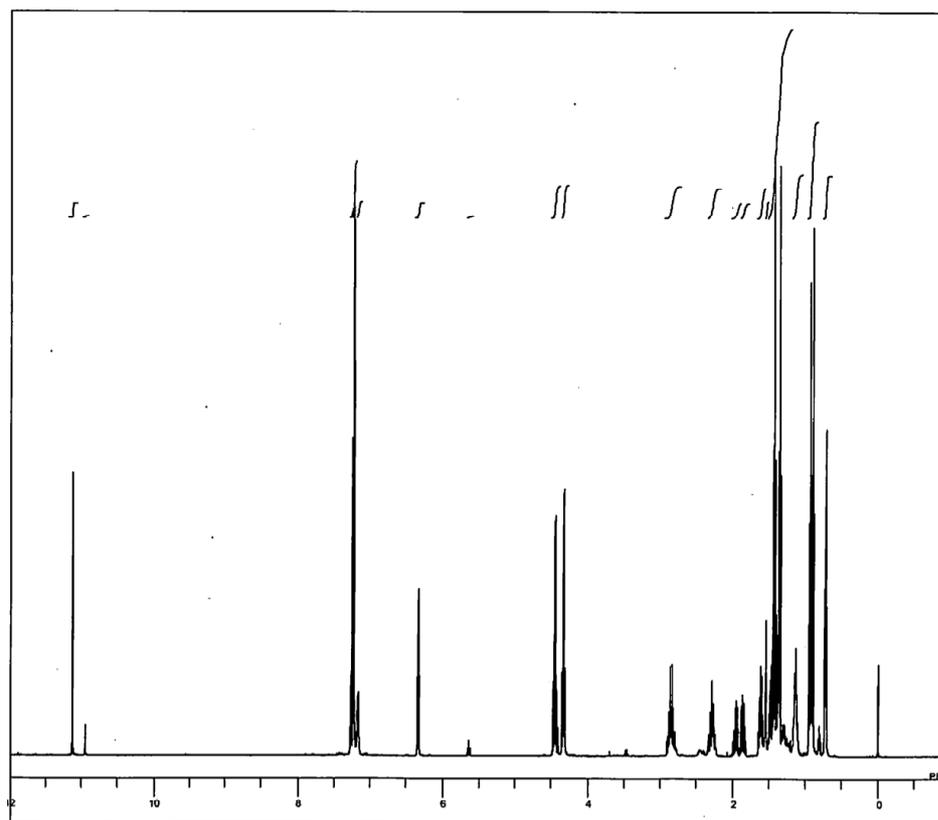
MENUMF 1H
OBNUC 1H
OFR 499.10 MHz
OBSET 0.00 KHz
OBFIN 12885.00 Hz
PWI 5.50 usec
DEADT 72.30 usec
PREDL 0.01000 msec
IWT 1000.0000 sec
POINT 65536
SPO 65536
TIMES 16
DUMMY 0
FREQU 8000.00 Hz
FLT 4000 Hz
DELAY 80.00 usec
ACQTM 8.1920 sec
PD 1.0000 sec
ADBIT 16
RGAIN 16
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD non
EXPCM single pulse nondecoupling & nonpresaturation
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE Methyl 6-(tertbutyl)diphenylsilyl-2-hexynoate-1H.tls
SF thSat
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 20
LKPHS 353
LKSIG 677
CSPED 13 Hz
FILDC
FILDF
SLVNT CDCL3
DATIM Thu May 28 12:18:10 2009
    
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```

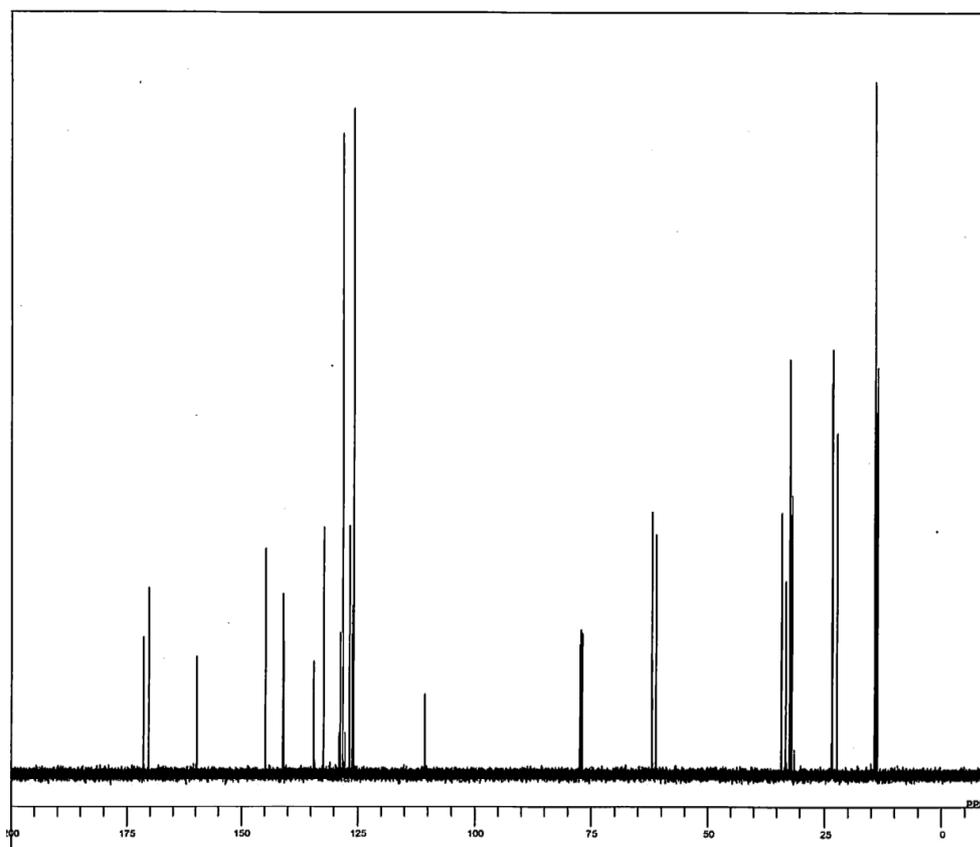
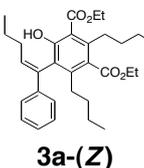
MENUMF 13C
OBNUC 13C
OFR 125.40 MHz
OBSET 0.00 KHz
OBFIN 143041.00 Hz
PWI 6.25 usec
DEADT 10.00 usec
PREDL 0.01000 msec
IWT 1000.0000 sec
POINT 65536
SPO 65536
TIMES 256
DUMMY 2
FREQU 33888.30 Hz
FLT 16950 Hz
DELAY 11.80 usec
ACQTM 1.9333 sec
PD 1.0670 sec
ADBIT 16
RGAIN 29
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD bcm
EXPCM single pulse with bilevel decoupling
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE Methyl 6-(tertbutyl)diphenylsilyl-2-hexynoate-13C.tls
SF thSat
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 20
LKPHS 353
LKSIG 670
CSPED 12 Hz
FILDC
FILDF
SLVNT CDCL3
DATIM Thu May 28 12:25:53 2009
    
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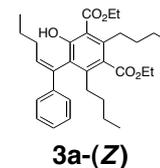
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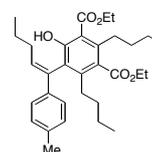
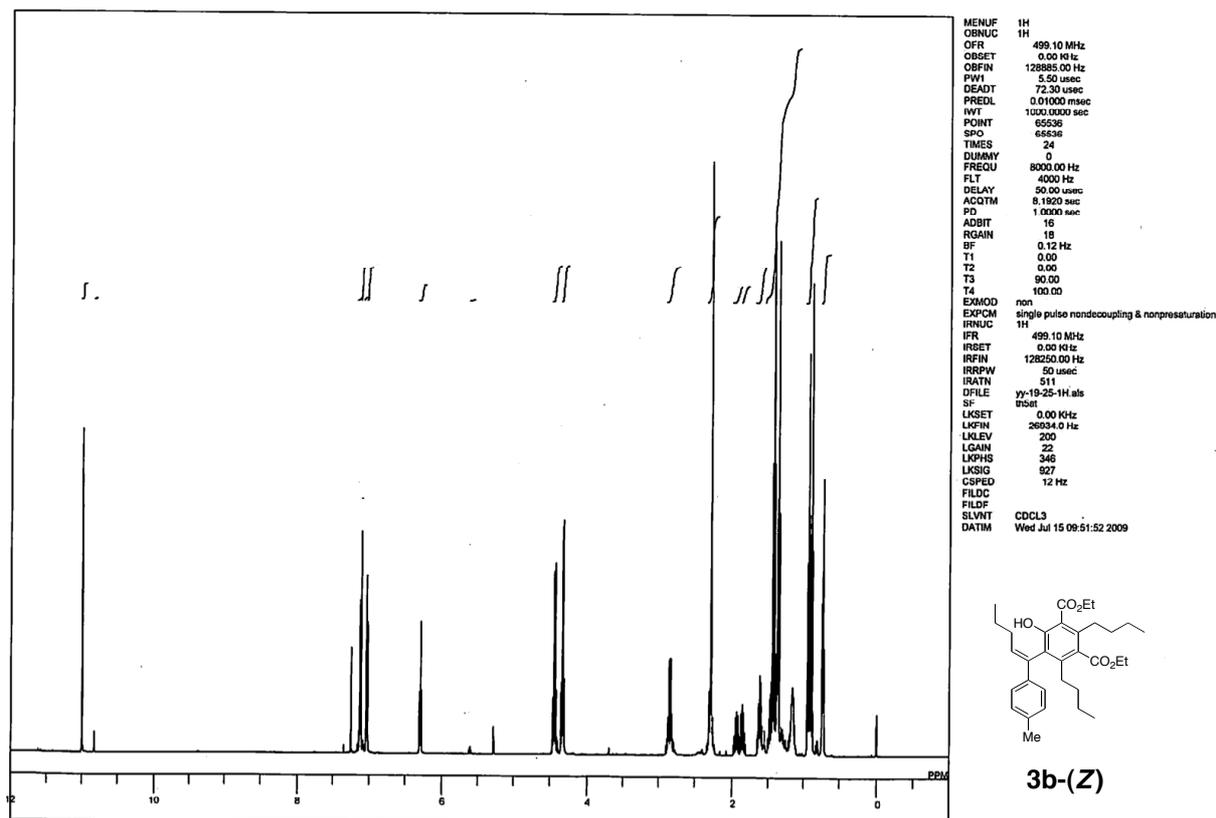
MENUF 1H
OBNUC 1H
OFR 499.10 MHz
OBSET 0.00 KHz
OBFIN 128250.00 Hz
PWI 5.50 usec
DEADT 72.30 usec
PREDL 0.01000 msec
IWT 1000.0000 sec
POINT 65536
SPO 24
TIMES 2
DUMMY 0
FREQU 8000.00 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 8.1920 sec
PD 1.0000 sec
ADBIT 16
RGAIN 21
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMDD non
EXPCM single pulse nondecoupling & nonpresaturation
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE yy-19-23-gpc-1H.als
SF 19561
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 19
LKPHS 346
LKSIG 530
CSPED 14 Hz
FILDC
FILDF
SLVNT CDCl3
DATM Thu Jul 09 16:56:44 2009
    
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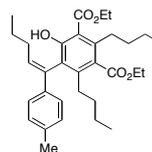
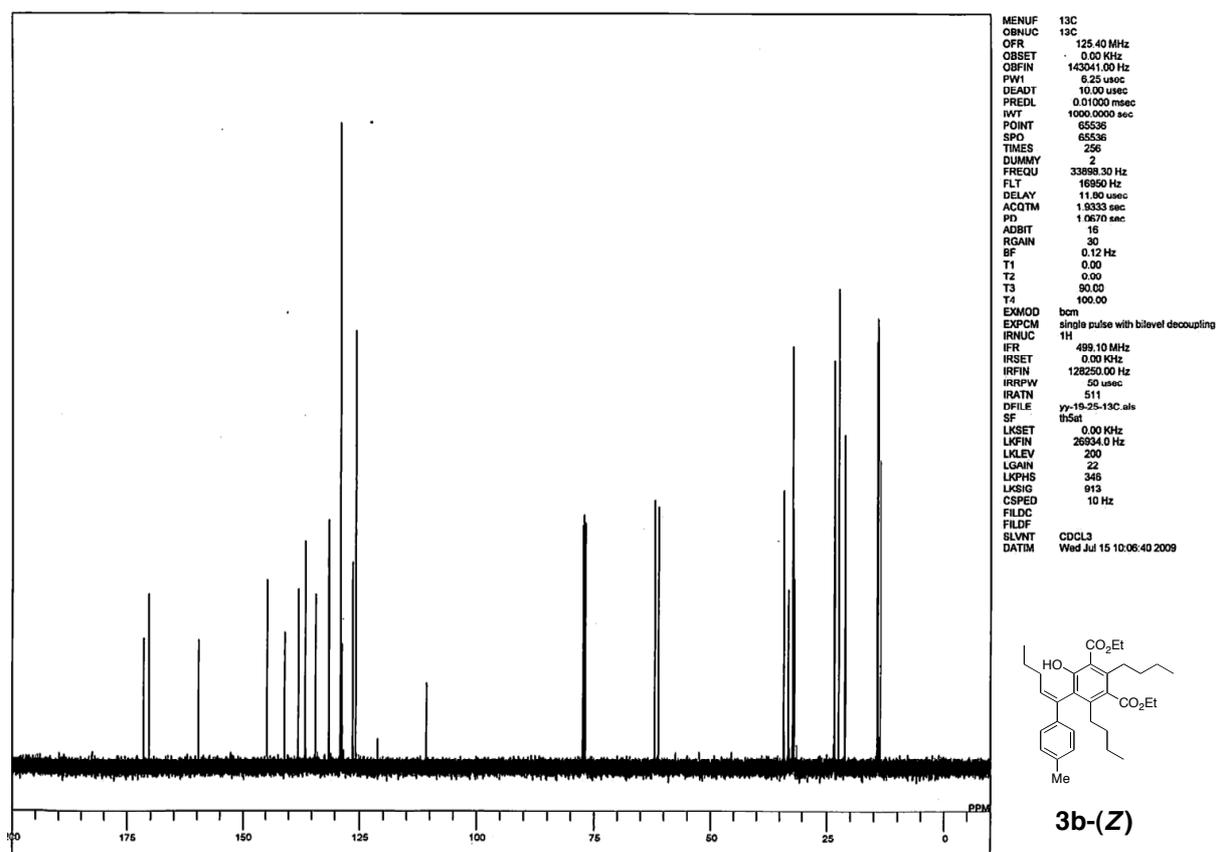
```

MENUF 13C
OBNUC 13C
OFR 125.40 MHz
OBSET 0.00 KHz
OBFIN 143041.00 Hz
PWI 6.25 usec
DEADT 10.00 usec
PREDL 0.01000 msec
IWT 1000.0000 sec
POINT 65536
SPO 360
TIMES 2
DUMMY 0
FREQU 33888.30 Hz
FLT 16950 Hz
DELAY 11.80 usec
ACQTM 1.8933 sec
PD 1.0670 sec
ADBIT 16
RGAIN 30
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMDD bcm
EXPCM single pulse with bilvel decoupling
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE yy-19-23-gpc-13C.als
SF 19561
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 22
LKPHS 346
LKSIG 824
CSPED 12 Hz
FILDC
FILDF
SLVNT CDCl3
DATM Thu Jul 09 22:46:10 2009
    
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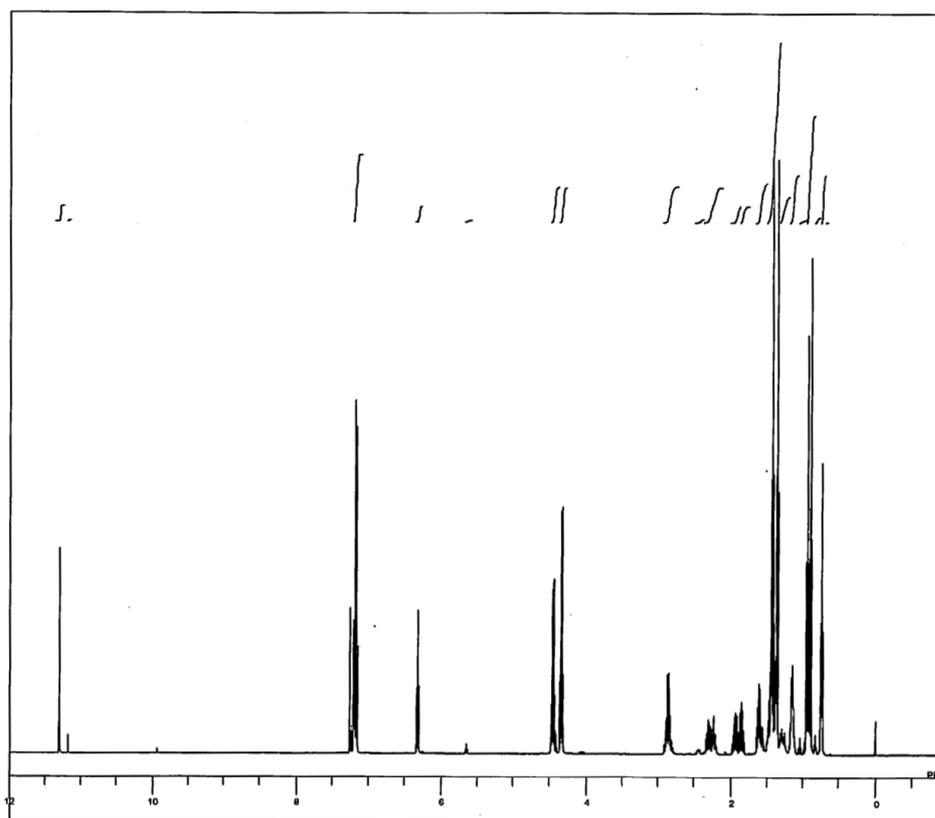




3b-(Z)

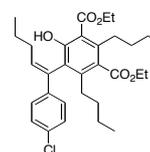


3b-(Z)

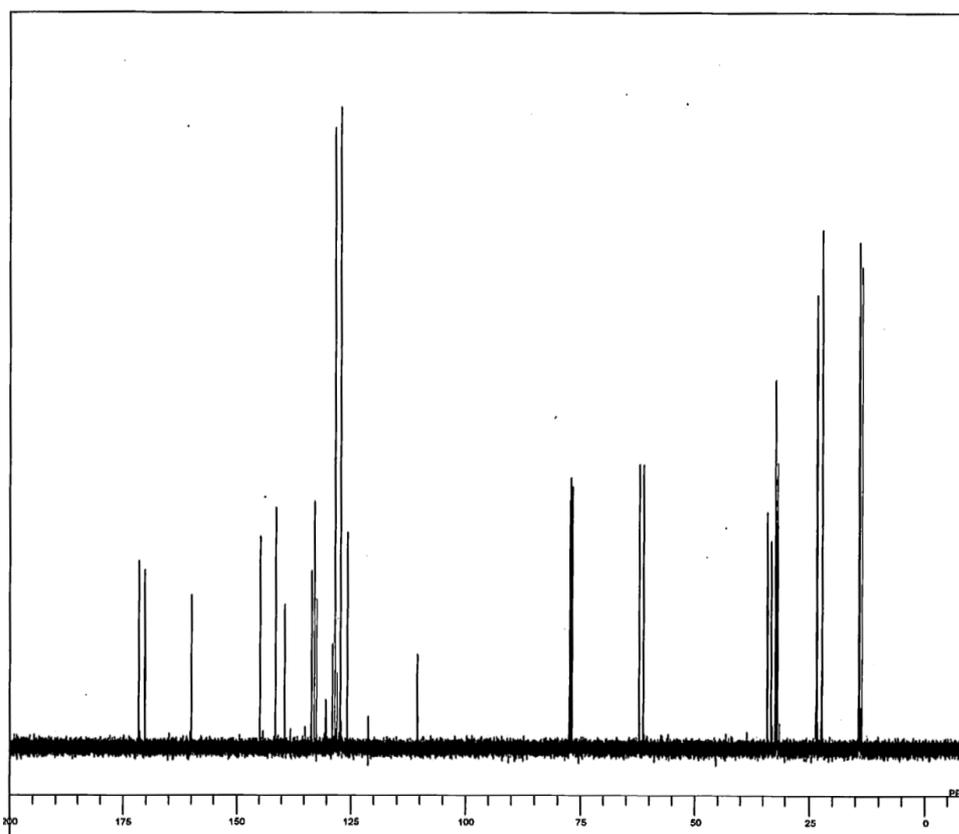


```

MENUF 1H
OBNUC 1H
OFR 499.10 MHz
OBSET 0.00 KHz
OBFIN 128955.00 Hz
PWI 5.50 usec
DEADT 72.30 usec
PREDL 0.01000 msec
WVT 1000.0000 sec
POINT 65536
SPO 65536
TIMES 24
DUMMY 0
FREQU 8000.00 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 0.1920 sec
PD 1.0500 sec
ADBIT 16
RGAIN 16
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD non
EXPCM single pulse nondecoupling & nonpresaturation
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE yy-19-24-1H.als
SF 1000
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 22
LKPHS 348
LKSIG 956
CSPED 12 Hz
FILDC
FILDF
SLWNT CDCL3
DATIM Mon Jul 13 15:07:04 2009
    
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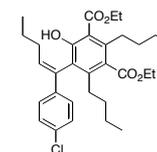


3d-(Z)

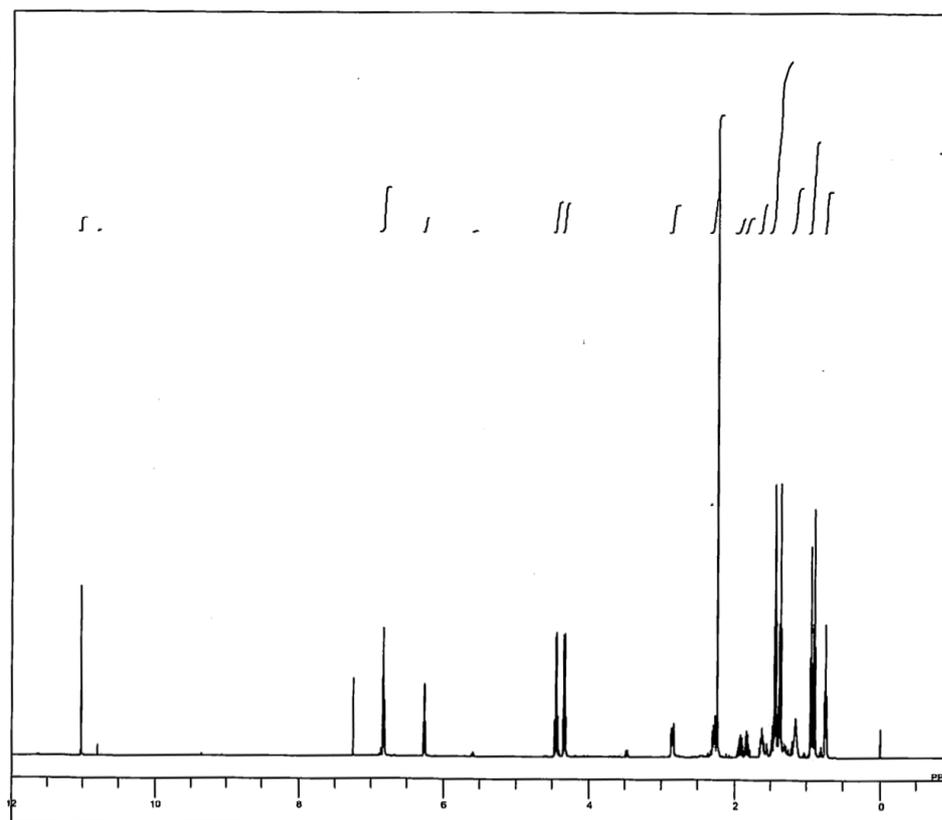


```

MENUF 13C
OBNUC 13C
OFR 125.40 MHz
OBSET 0.00 KHz
OBFIN 143041.00 Hz
PWI 0.25 usec
DEADT 10.00 usec
PREDL 0.01000 msec
WVT 1000.0000 sec
POINT 65536
SPO 65536
TIMES 256
DUMMY 2
FREQU 33896.30 Hz
FLT 16950 Hz
DELAY 11.60 usec
ACQTM 1.8333 sec
PD 1.0670 sec
ADBIT 16
RGAIN 30
BF 0.12 Hz
T1 0.00
T2 0.00
T3 80.00
T4 100.00
EXMOD bcm
EXPCM single pulse with bilateral decoupling
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE yy-19-24-13C.als
SF 1000
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 22
LKPHS 348
LKSIG 900
CSPED 11 Hz
FILDC
FILDF
SLWNT CDCL3
DATIM Mon Jul 13 15:21:11 2009
    
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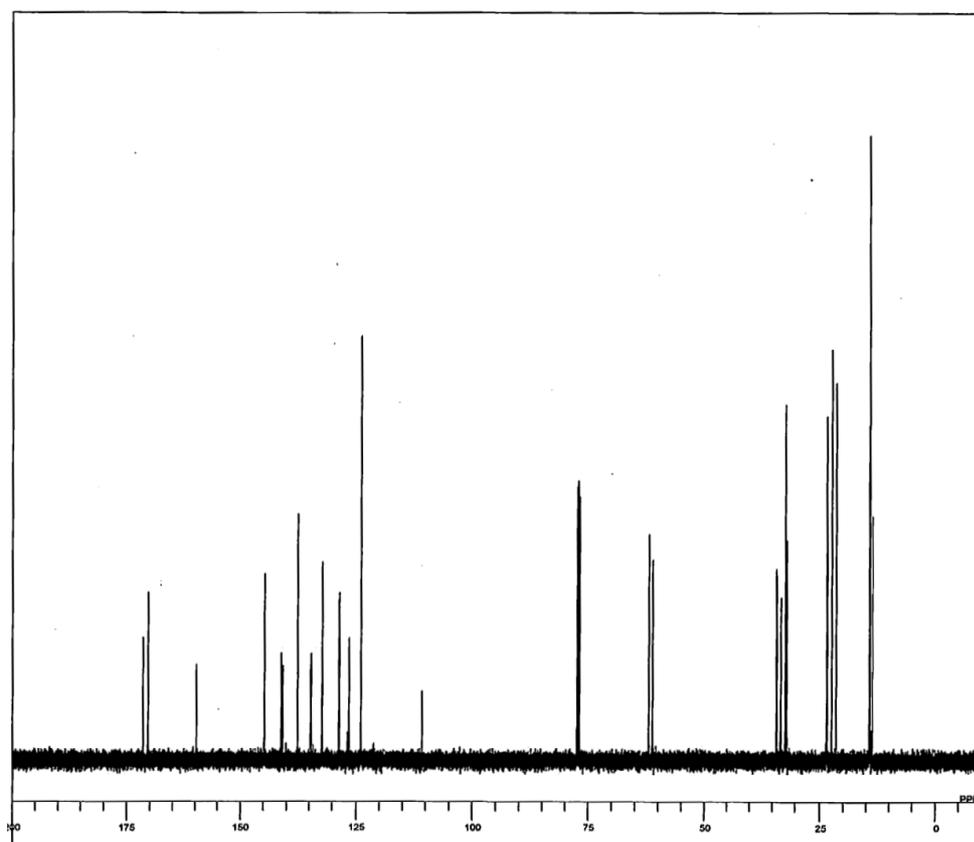
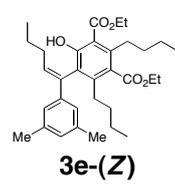


3d-(Z)



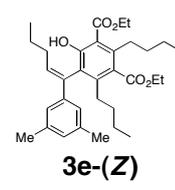
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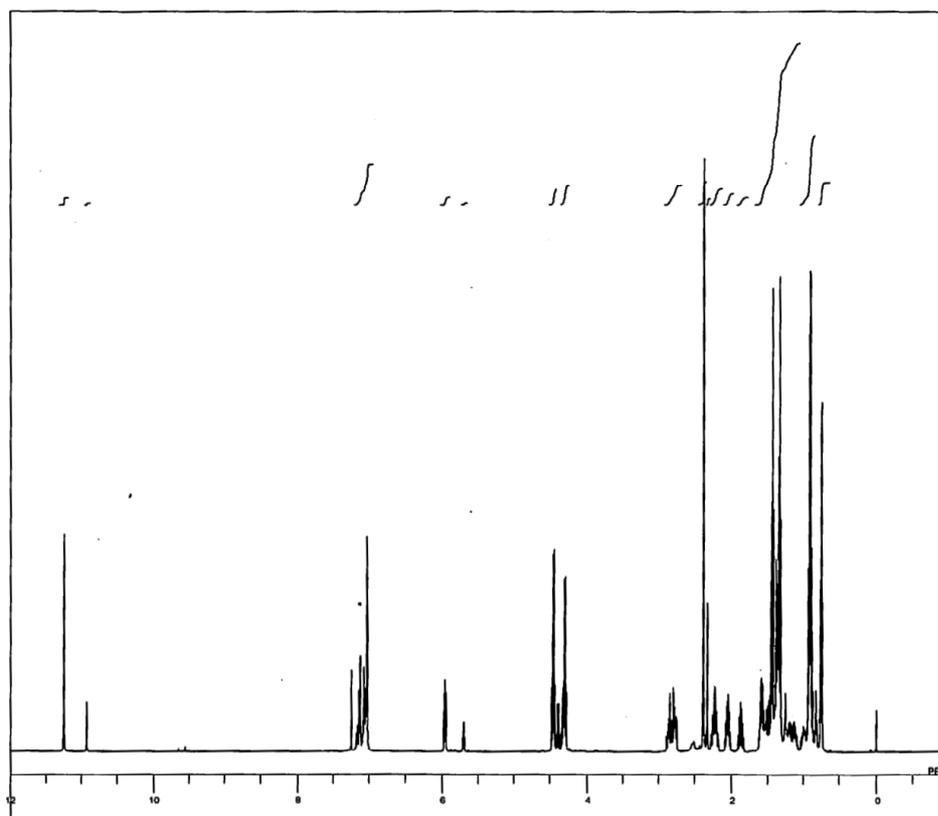
MENUF 1H
CNUC 1H
OFR 499.10 MHz
OBSET 0.00 KHz
OBFIN 128985.00 Hz
PWI 5.50 usec
DEADT 72.30 usec
PREDL 0.01000 msec
PWT 1000.0000 sec
POINT 65536
SPO 65536
TIMES 24
DUMMY 0
FREQU 8000.00 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 8.1920 sec
PD 1.0000 sec
ADBIT 16
RGAIN 18
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD non
EXPCM single pulse nondecoupling & nonpresaturation
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE yy-19-26-1H.q1s
SF 0.00 KHz
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 20
LKPHS 346
LKSIG 630
CSPED 10 Hz
FILDC
FILDF
SLWNT CDCL3
DATIM Thu Jul 16 00:15:13 2009
    
```



```

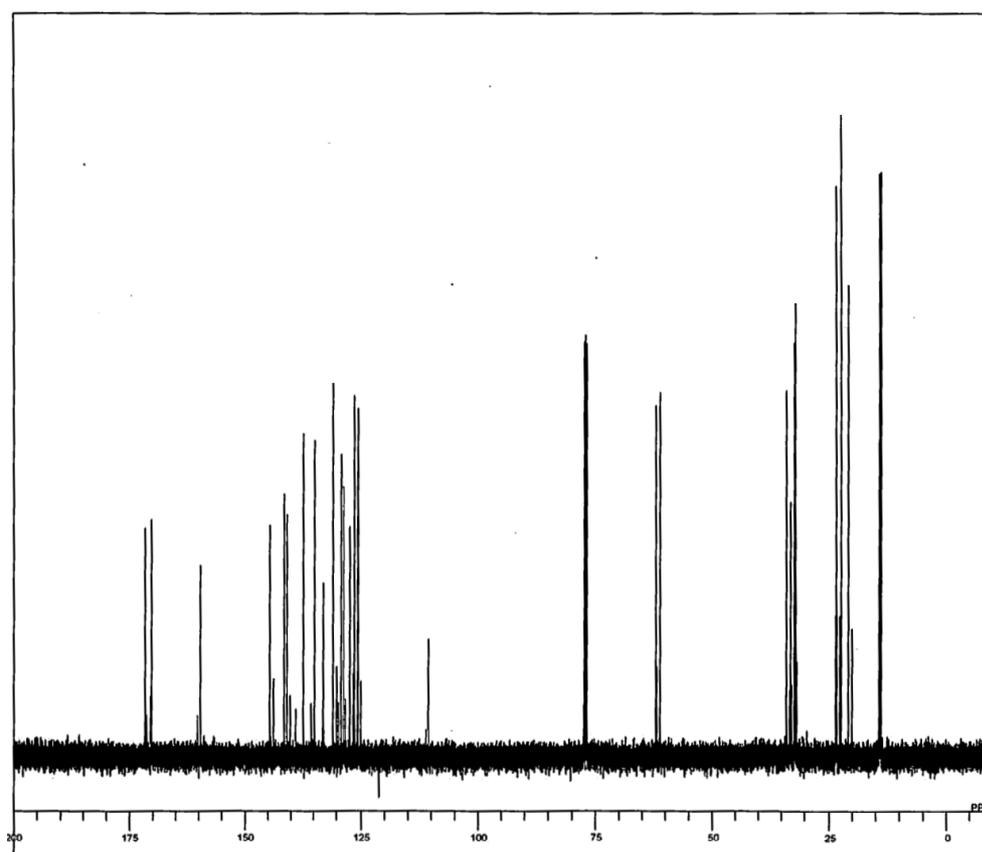
MENUF 13C
CNUC 13C
OFR 125.40 MHz
OBSET 0.00 KHz
OBFIN 143041.00 Hz
PWI 6.25 usec
DEADT 10.00 usec
PREDL 0.01000 msec
PWT 1000.0000 sec
POINT 65536
SPO 65536
TIMES 2
DUMMY 0
FREQU 33898.30 Hz
FLT 16950 Hz
DELAY 11.80 usec
ACQTM 1.8333 sec
PD 1.0870 sec
ADBIT 16
RGAIN 31
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD bcm
EXPCM single pulse with bilevel decoupling
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE yy-19-26-13C.q1s
SF 0.00 KHz
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 20
LKPHS 346
LKSIG 610
CSPED 12 Hz
FILDC
FILDF
SLWNT CDCL3
DATIM Thu Jul 16 00:29:08 2009
    
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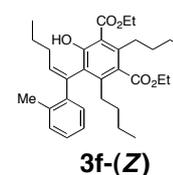
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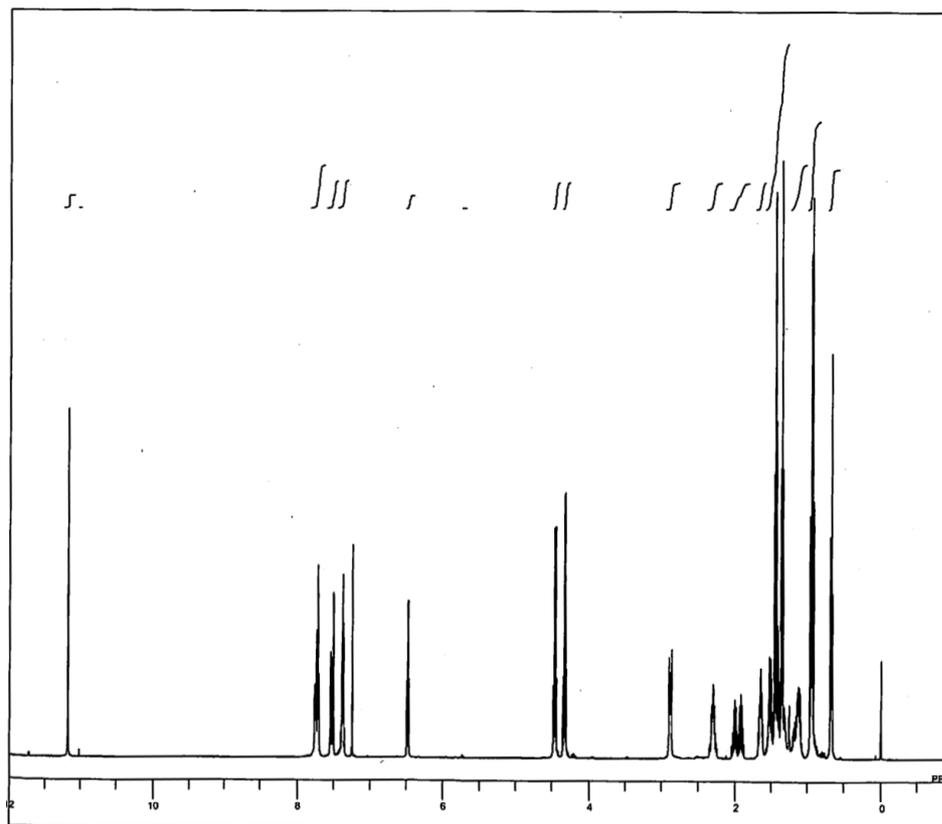
MENUF 1H
OBNUC 1H
OFR 499.10 MHz
OBSET 0.00 kHz
OBFIN 12885.00 Hz
PWI 5.50 usec
DEADT 72.30 usec
FREDL 0.0100 msec
IWT 1000.0000 sec
POINT 65536
SPD 65536
TIMES 32
DUMMY 0
FREQU 8000.00 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 6.1920 sec
PD 1.0020 sec
ADBIT 16
RGAIN 16
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD non
EXPCM single pulse nondecoupling & nonpresaturation
IRNUC 1H
IR 499.10 MHz
IRSET 0.00 kHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE yy-19-77-1H.als
SF 511.6184
LKSET 0.00 kHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 22
LKPHS 346
LKSIG 748
CSPED 12 Hz
FILDC
FILDF
SLVNT CDCL3
DATM Tue Aug 11 14:07:15 2009
    
```



```

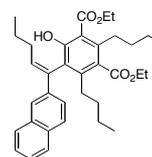
MENUF 13C
OBNUC 13C
OFR 125.40 MHz
OBSET 0.00 kHz
OBFIN 143041.00 Hz
PWI 6.25 usec
DEADT 10.00 usec
FREDL 0.0100 msec
IWT 1000.0000 sec
POINT 65536
SPD 65536
TIMES 256
DUMMY 2
FREQU 33898.30 Hz
FLT 16950 Hz
DELAY 11.80 usec
ACQTM 1.9333 sec
PD 1.0670 sec
ADBIT 16
RGAIN 30
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD bcm
EXPCM single pulse with bilevel decoupling
IRNUC 1H
IR 499.10 MHz
IRSET 0.00 kHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE yy-19-77-13C.als
SF 511.6184
LKSET 0.00 kHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 22
LKPHS 346
LKSIG 745
CSPED 13 Hz
FILDC
FILDF
SLVNT CDCL3
DATM Tue Aug 11 14:20:59 2009
    
```



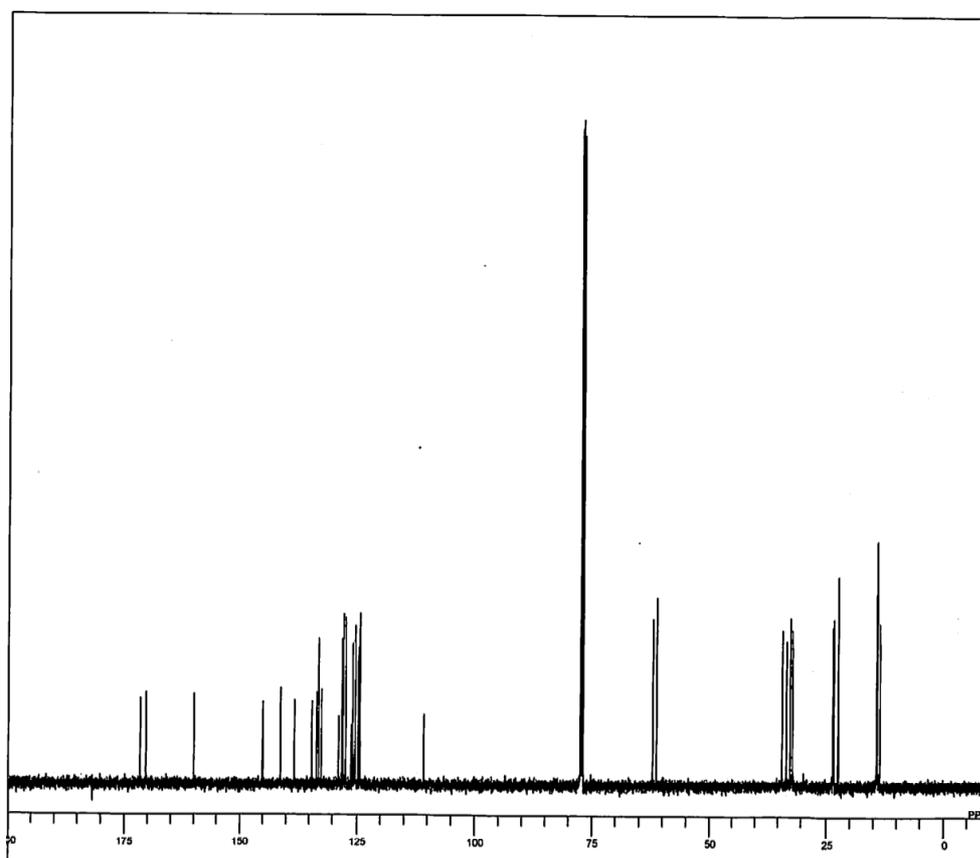


```

MENUF  non
OBNUC  1H
OFR    499.10 MHz
OBSET  0.00 KHz
OSFIN  128250.00 Hz
PW1    6.10 usec
DEADT  56.95 usec
PREDL  10.00000 msec
WT     0.5000 sec
POINT  32768
SPD    32768
TIMES  46
DUMMY  0
FREQU  9950.04 Hz
FLT    5000 Hz
DELAY  40.00 usec
ACQTM  3.2834 sec
PD     3.7165 sec
ADBIT  16
RGAIN  21
BF     0.12 Hz
T1    0.00
T2    0.00
T3    90.00
T4    100.00
EXMOD  non
EXPCM  single pulse nondecoupling & nonpresaturation
IRNUC  1H
IFR    499.10 MHz
IRSET  0.00 KHz
IRFIN  128250.00 Hz
IRRPW  50 usec
IRATN  511
DFILE  yy-19-78-rev-1H.als
SF     inSet
LKSET  0.00 KHz
LKFIN  26934.0 Hz
LKLEV  200
LGAIN  22
LKPHS  346
LKSIG  766
CSPED  12 Hz
FILDC  FILDF
SLVNT  CDCL3
DATM   Fri Aug 21 13:28:45 2009
    
```

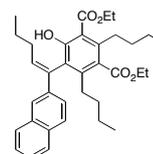


3g-(Z)

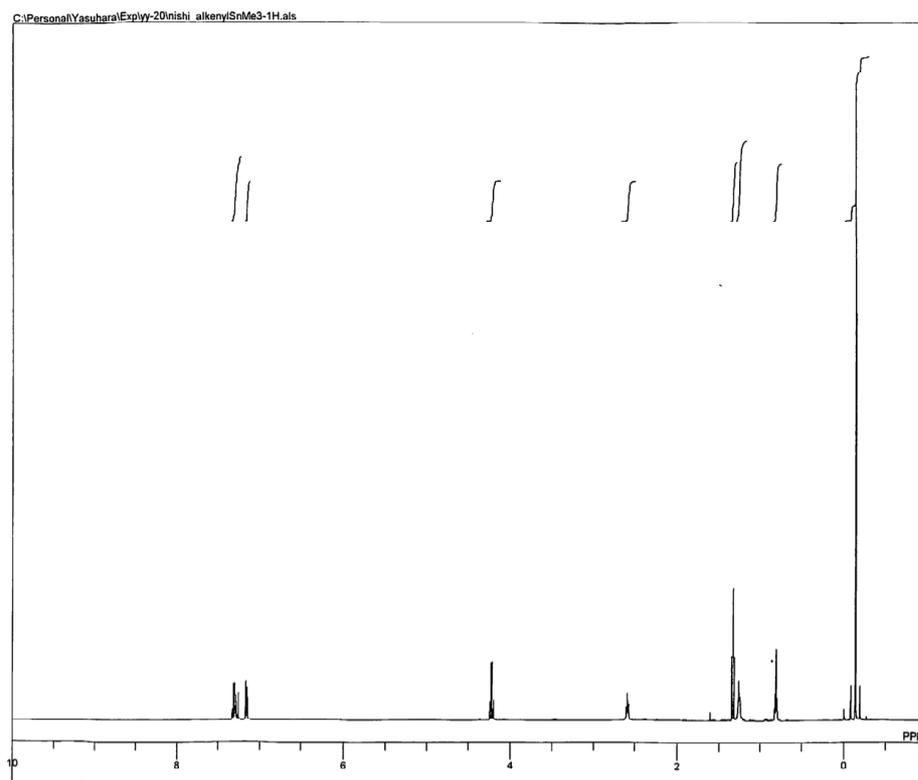


```

MENUF  bcm
OBNUC  13C
OFR    125.40 MHz
OBSET  0.00 KHz
OSFIN  143041.00 Hz
PW1    5.50 usec
DEADT  10.00 usec
PREDL  10.00000 msec
WT     10.0000 sec
POINT  32768
SPD    32768
TIMES  720
DUMMY  1
FREQU  33898.30 Hz
FLT    16950 Hz
DELAY  11.00 usec
ACQTM  0.9607 sec
PD     2.0333 sec
ADBIT  16
RGAIN  30
BF     1.20 Hz
T1    0.00
T2    0.00
T3    90.00
T4    100.00
EXMOD  bcm
EXPCM  single pulse with bilevel decoupling
IRNUC  13C
IFR    499.10 MHz
IRSET  0.00 KHz
IRFIN  128250.00 Hz
IRRPW  65 usec
IRATN  511
DFILE  yy-19-78-rev-13C.als
SF     inSet
LKSET  0.00 KHz
LKFIN  26934.0 Hz
LKLEV  200
LGAIN  22
LKPHS  346
LKSIG  762
CSPED  12 Hz
FILDC  FILDF
SLVNT  CDCL3
DATM   Fri Aug 21 14:05:02 2009
    
```



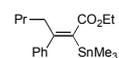
3g-(Z)



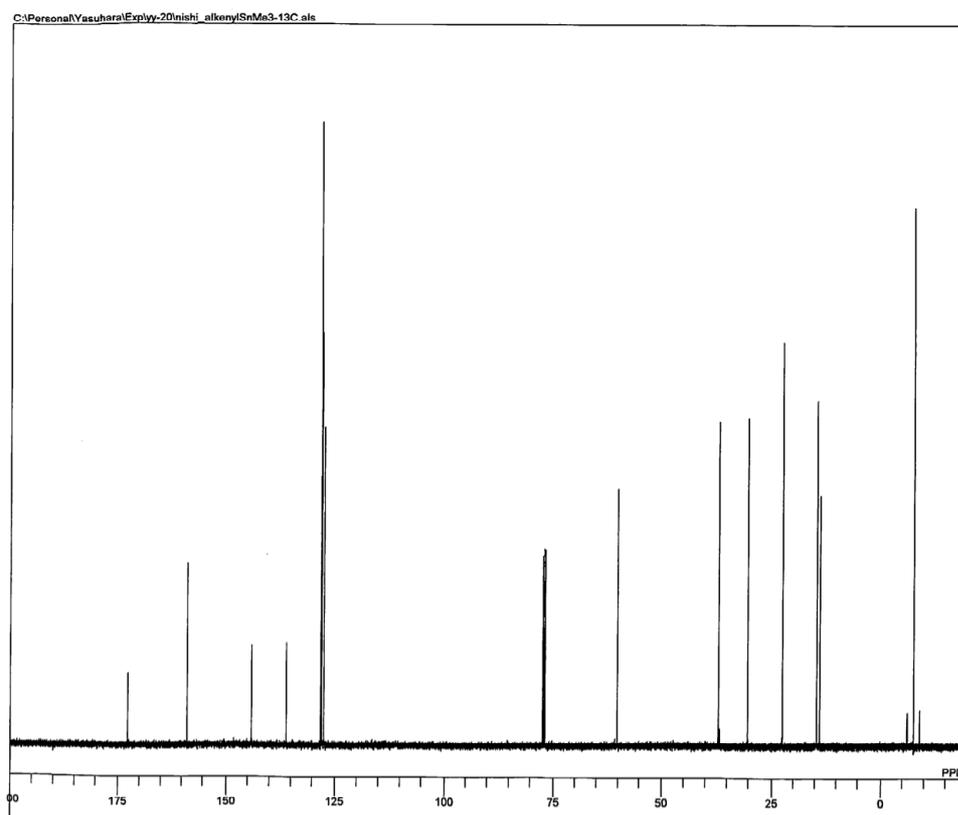
```

MENUMF 1H
OBNUC 1H
OFR 499.10 MHz
OBSET 0.00 KHz
OBFIN 128885.00 Hz
PW1 5.50 usec
DEADT 72.30 usec
PREDL 0.01000 msec
IWT 1000.0000 msec
POINT 65536
SPO 65536
TIMES 16
DUMMY 0
FREQU 8000.00 Hz
FLT 4000 Hz
DELAY 60.00 usec
ACQTM 8.1920 sec
PD 1.0000 sec
ADBIT 16
RGAIN 17
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD non
EXPCM single pulse nondecoupling & nonpresaturation
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE nishi_alkenylSnMe3-1H.als
SF th5at
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 20
LKPHS 346
LKSIG 605
CSPED 13 Hz
FILDC
FILDF
SLVNT CDCL3
DATIM Fri Dec 18 00:47:22 2009

```



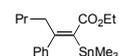
5



```

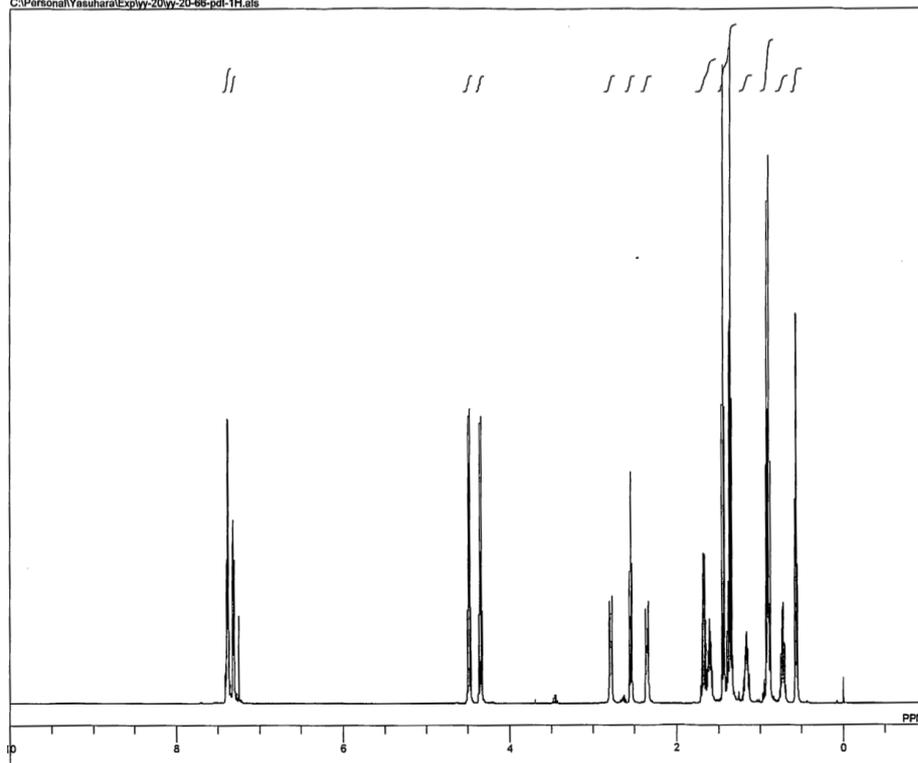
MENUMF 13C
OBNUC 13C
OFR 125.40 MHz
OBSET 0.00 KHz
OBFIN 143041.00 Hz
PW1 6.25 usec
DEADT 10.00 usec
PREDL 0.01000 msec
IWT 1000.0000 msec
POINT 65536
SPO 65536
TIMES 512
DUMMY 2
FREQU 33898.00 Hz
FLT 16950 Hz
DELAY 11.80 usec
ACQTM 1.9333 sec
PD 1.0670 sec
ADBIT 16
RGAIN 23
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD bcm
EXPCM single pulse with bilevel decoupling
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE nishi_alkenylSnMe3-13C.als
SF th5at
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 20
LKPHS 346
LKSIG 598
CSPED 13 Hz
FILDC
FILDF
SLVNT CDCL3
DATIM Fri Dec 18 01:08:26 2009

```



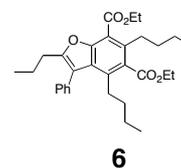
5

C:\Personal\Yasuhara\Explyy-20\yy-20-66-pdt-1H.als

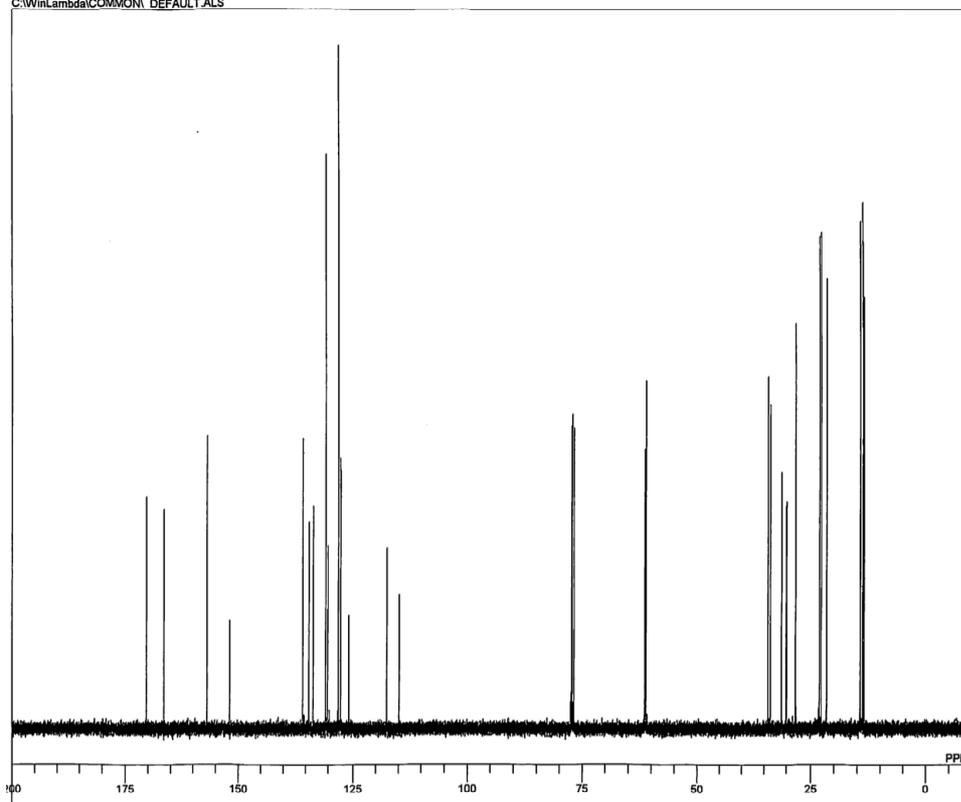


```

MENUF 1H
OBNUC 1H
OFR 499.10 MHz
OBSET 0.00 KHz
OBFIN 128885.00 Hz
PW1 5.50 usec
DEADT 72.30 usec
PREDL 0.01000 msec
IWT 1000.0000 msec
POINT 65536
SPO 65536
TIMES 24
DUMMY 0
FREQU 8000.00 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 8.1920 sec
PD 1.0000 sec
ADBIT 16
RGAIN 17
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD non
EXPCM single pulse nondecoupling & nonpresaturation
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE yy-20-66-pdt-1H.als
SF th5at
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 22
LKPHS 346
LKSG 828
CSPED 13 Hz
FILDC
FILDF
SLVNT CDCL3
DATIM Tue Dec 22 12:05:11 2009
    
```

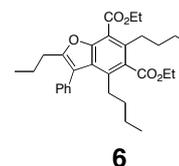


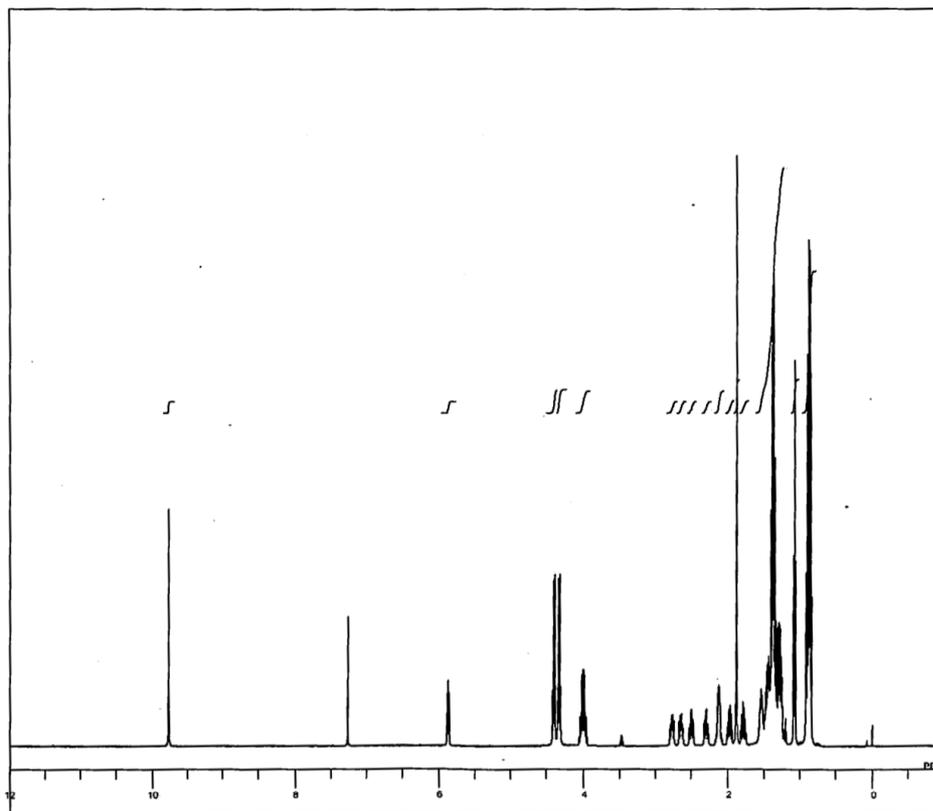
C:\WinLambda\COMMON_DEFAULTS



```

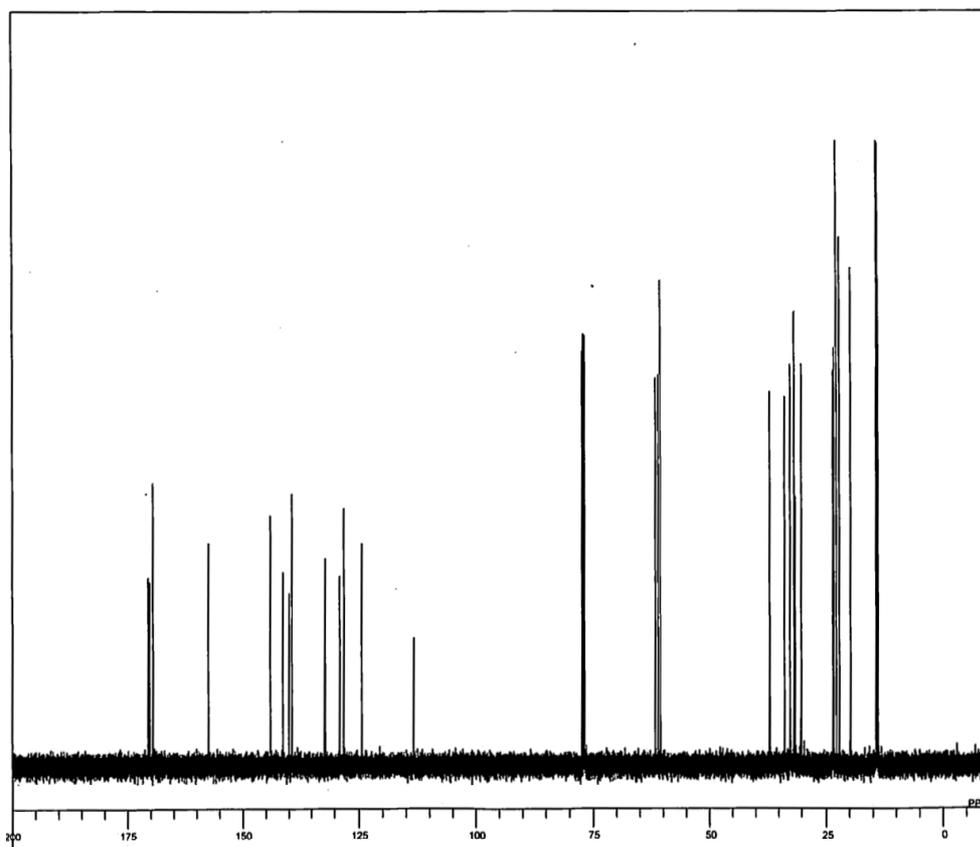
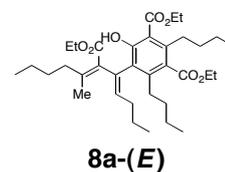
MENUF 13C
OBNUC 130C
OFR 125.40 MHz
OBSET 0.00 KHz
OBFIN 143041.00 Hz
PW1 6.25 usec
DEADT 10.00 usec
PREDL 0.01000 msec
IWT 1000.0000 msec
POINT 65536
SPO 65536
TIMES 592
DUMMY 2
FREQU 33898.30 Hz
FLT 16950 Hz
DELAY 11.80 usec
ACQTM 1.9333 sec
PD 1.0670 sec
ADBIT 16
RGAIN 24
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD bcm
EXPCM single pulse with bilevel decoupling
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE _DEFAULTS.ALS
SF th5at
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 22
LKPHS 346
LKSG 763
CSPED 13 Hz
FILDC
FILDF
SLVNT CDCL3
DATIM Tue Dec 22 12:36:48 2009
    
```





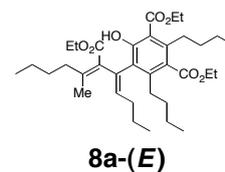
```

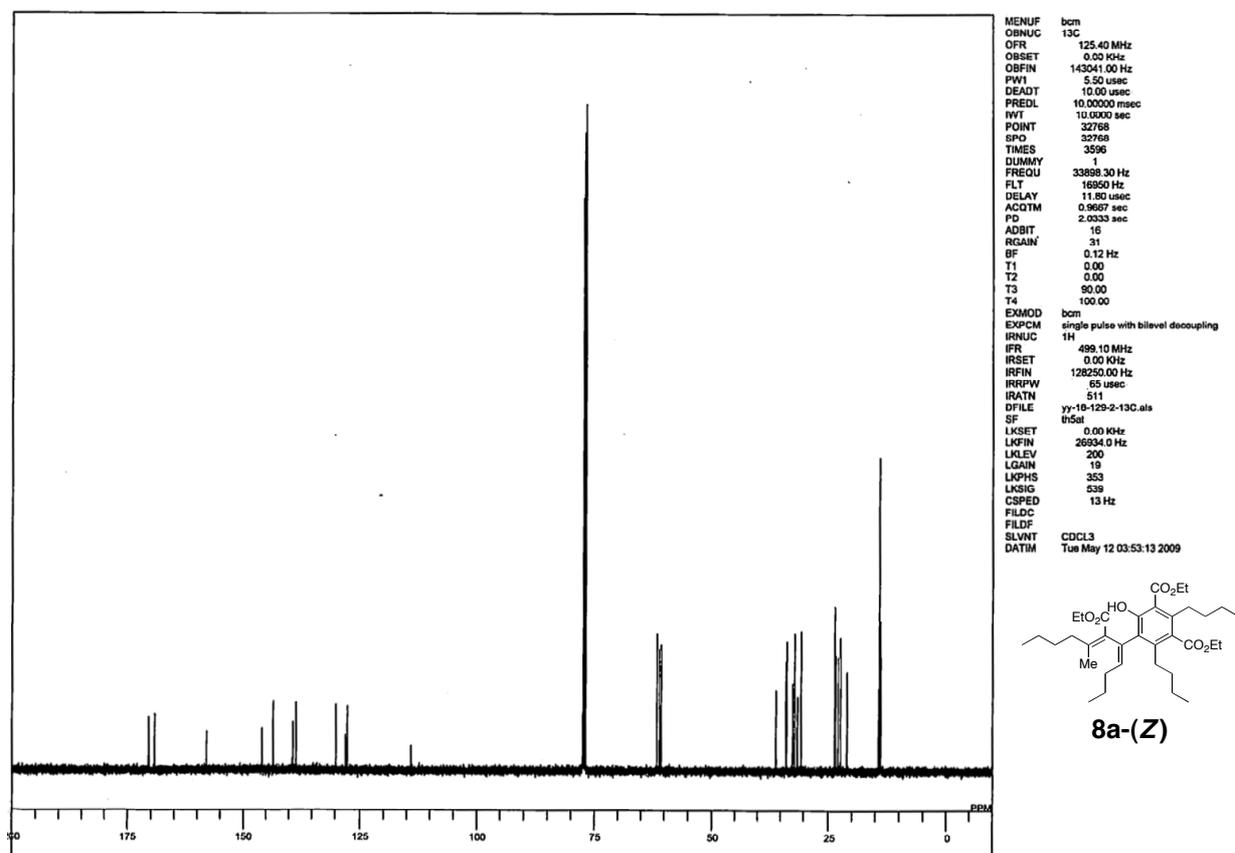
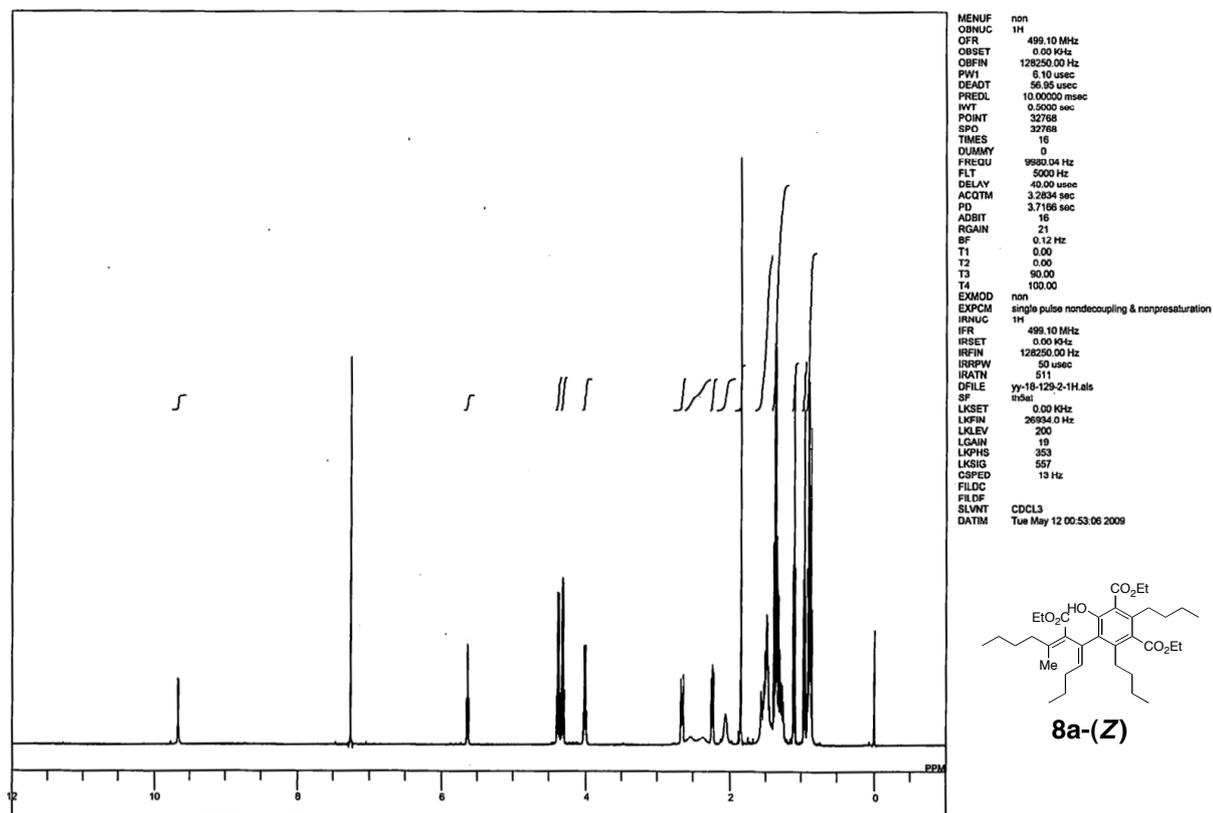
MENUF 1H
OBNUC 1H
OFR 499.10 MHz
OBSET 0.00 KHz
OBFIN 128885.00 Hz
PW1 5.50 usec
DEADT 72.30 usec
FREDL 0.01000 msec
IWT 1000.0000 sec
POINT 65536
SPO 16
TIMES 16
DUMMY 0
FREQU 8000.00 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 6.1920 sec
PD 1.0000 sec
ADBIT 10
RGAIN 16
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD non
EXPCM single pulse nondecoupling & nonpresaturation
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE yy-18-129-3(pure)-1H.als
SF 0.00 KHz
LKSET 28934.0 Hz
LKFIN 200
LKLEV 22
LKPHS 353
LKSIG 862
CSPED 12 Hz
FILDC
FILDF
SLVNT CDCL3
DATIM Wed May 13 18:36:22 2009
    
```

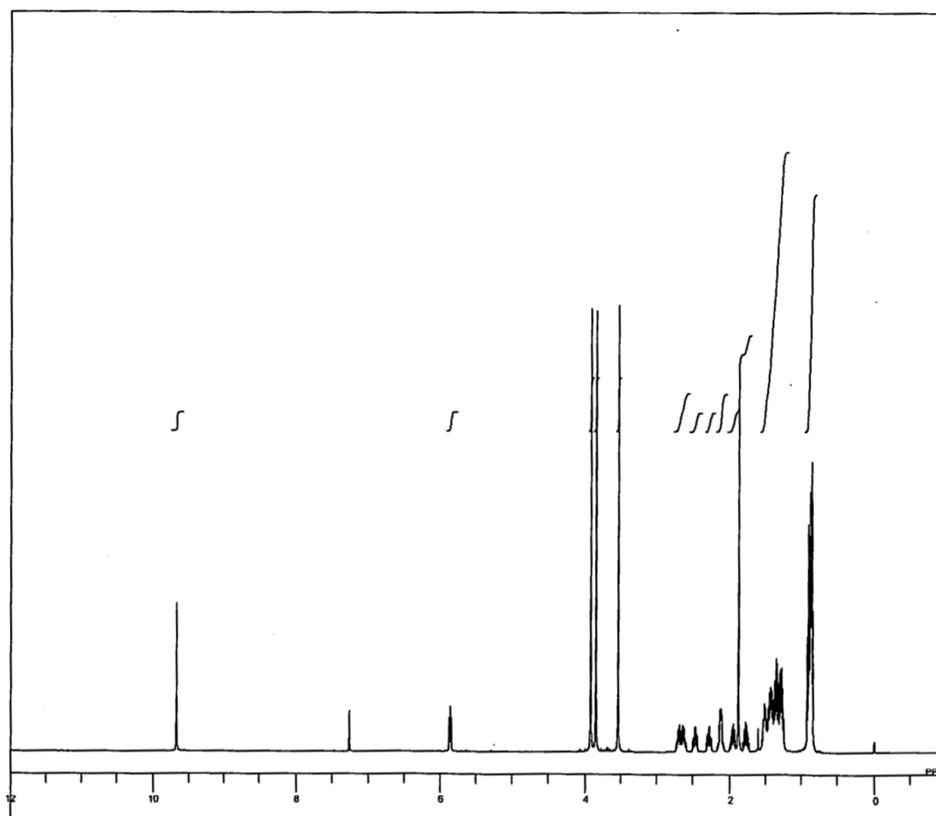


```

MENUF 13C
OBNUC 13C
OFR 125.40 MHz
OBSET 0.00 KHz
OBFIN 143041.00 Hz
PW1 6.25 usec
DEADT 10.00 usec
FREDL 0.01000 msec
IWT 1000.0000 sec
POINT 65536
SPO 400
TIMES 2
DUMMY 2
FREQU 33808.30 Hz
FLT 16950 Hz
DELAY 11.80 usec
ACQTM 1.8333 sec
PD 1.0670 sec
ADBIT 16
RGAIN 30
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD non
EXPCM single pulse with bilevel decoupling
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE yy-18-129-3(pure)-13C.als
SF 0.00 KHz
LKSET 26334.0 Hz
LKFIN 200
LKLEV 22
LKPHS 353
LKSIG 878
CSPED 12 Hz
FILDC
FILDF
SLVNT CDCL3
DATIM Wed May 13 18:57:30 2009
    
```

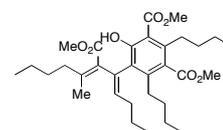




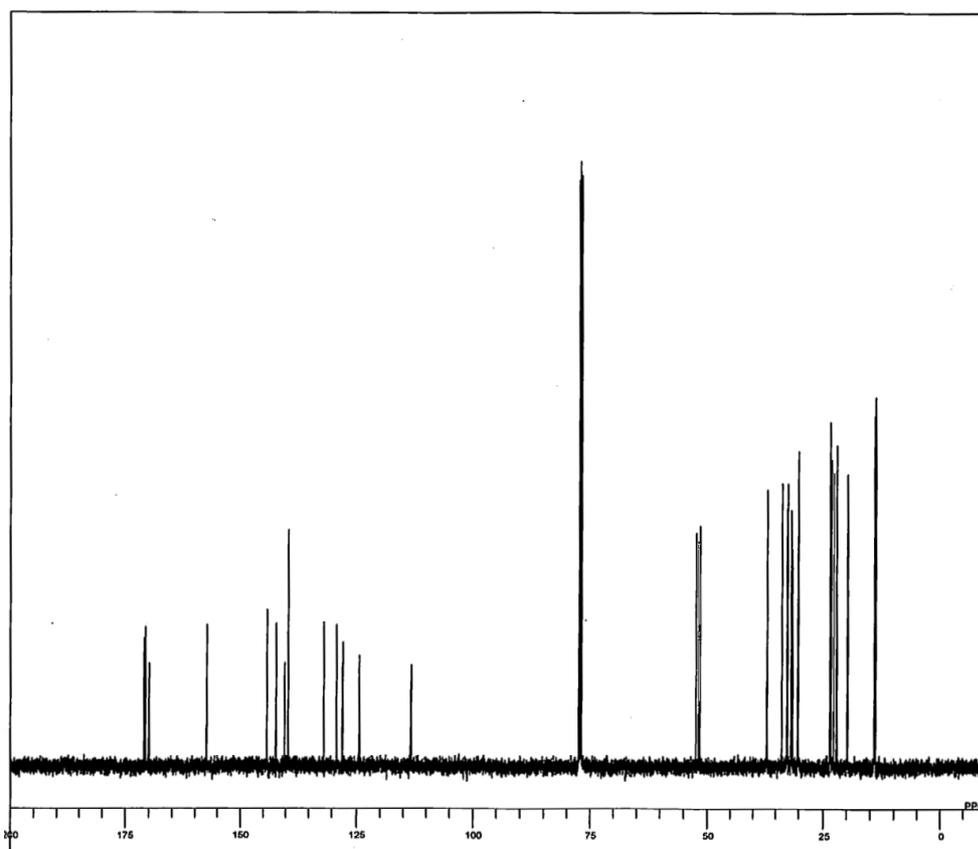


```

MENUF non
OBNUC 1H
OFR 499.10 MHz
OBSET 0.00 kHz
OBFIN 128250.00 Hz
PWI 6.10 usec
DEADT 56.95 usec
PREDL 10.00000 msec
RWT 0.5000 sec
POINT 32768
SPO 32768
TIMES 16
DUMMY 0
FREQU 9960.04 Hz
FLT 5000 Hz
DELAY 40.00 usec
ACQTM 3.2834 sec
PD 3.7196 sec
ADBIT 16
RGAIN 18
BF 1.20 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD non
EXPCM single pulse nondecoupling & nonpresaturation
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 kHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE yy-17-110-1H.als
SF 500.135
LKSET 0.00 kHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 19
LKPHS 353
LKSIG 519
OSPED 13 Hz
FILDC
FILDF
SLVNT CDCL3
DATM Fri Nov 14 12:20:31 2008
    
```

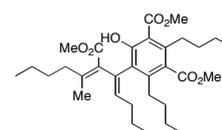


8b-(E)

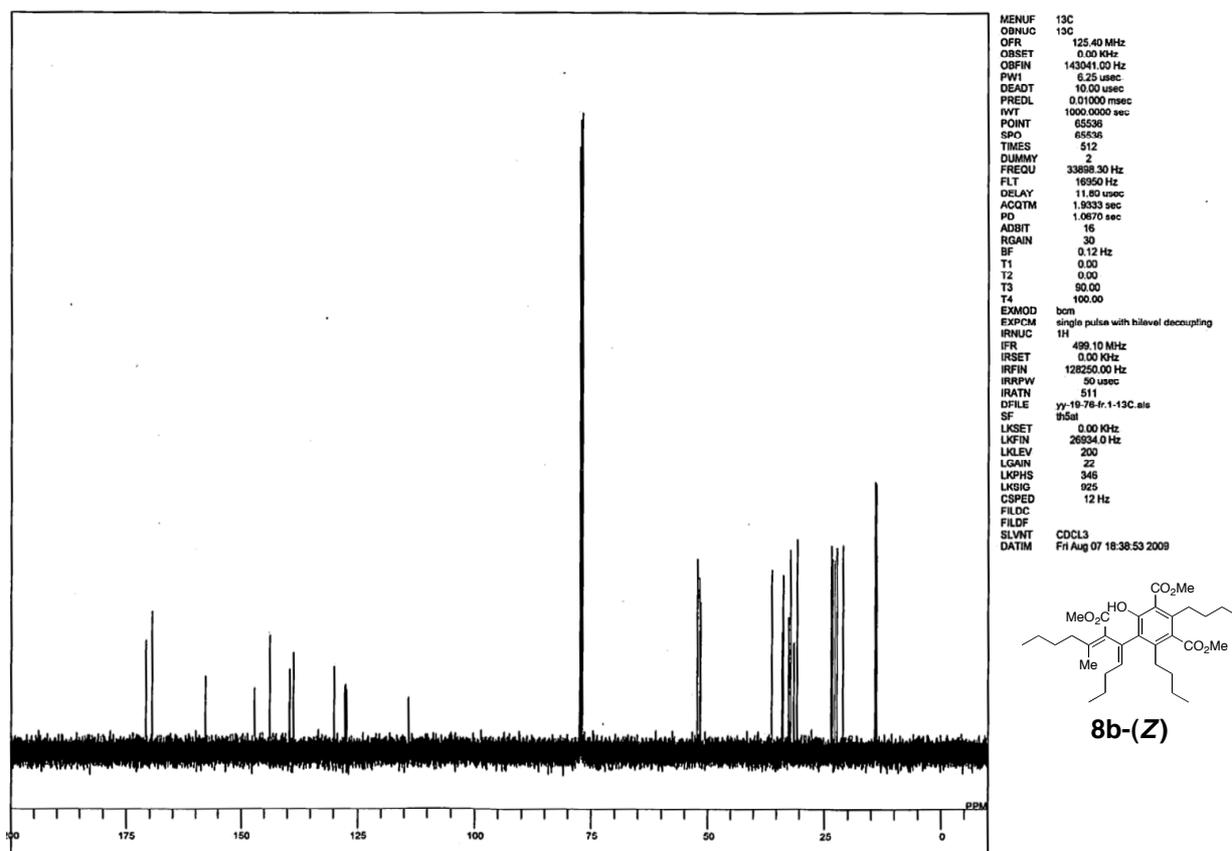
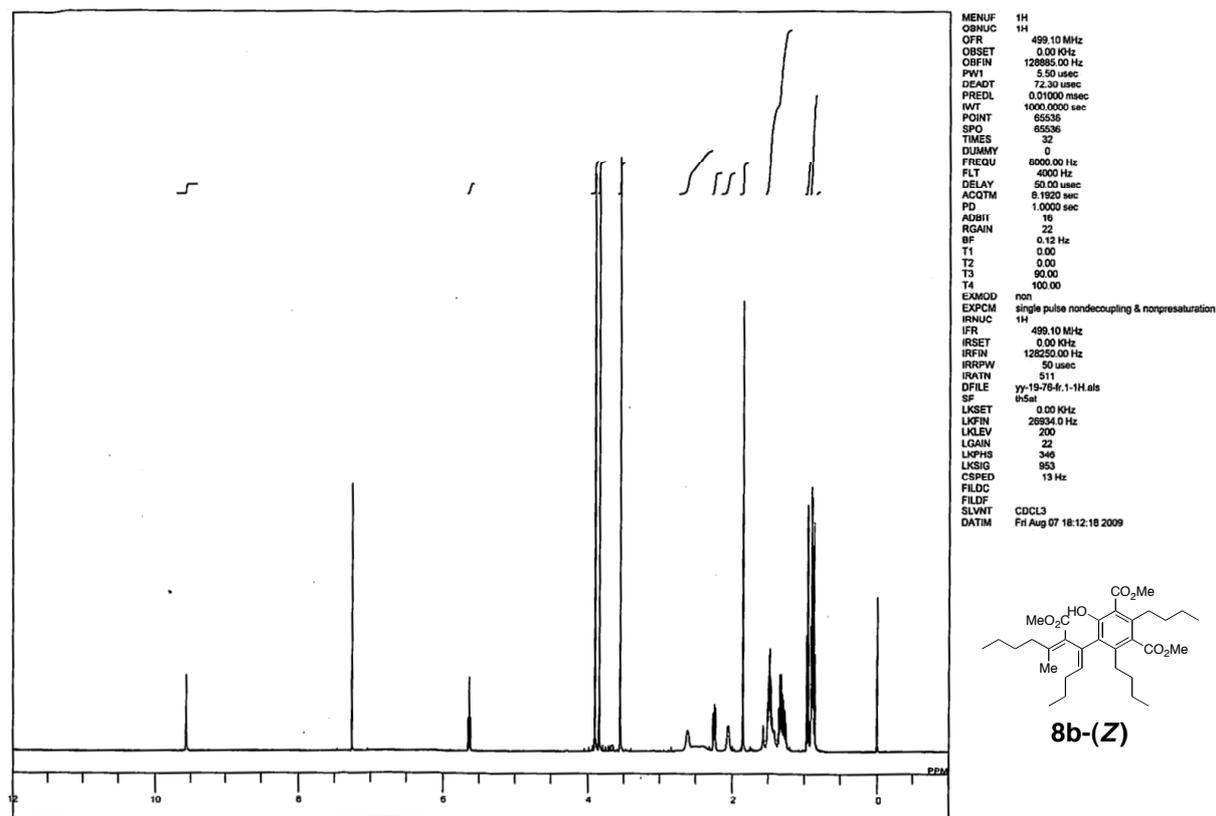


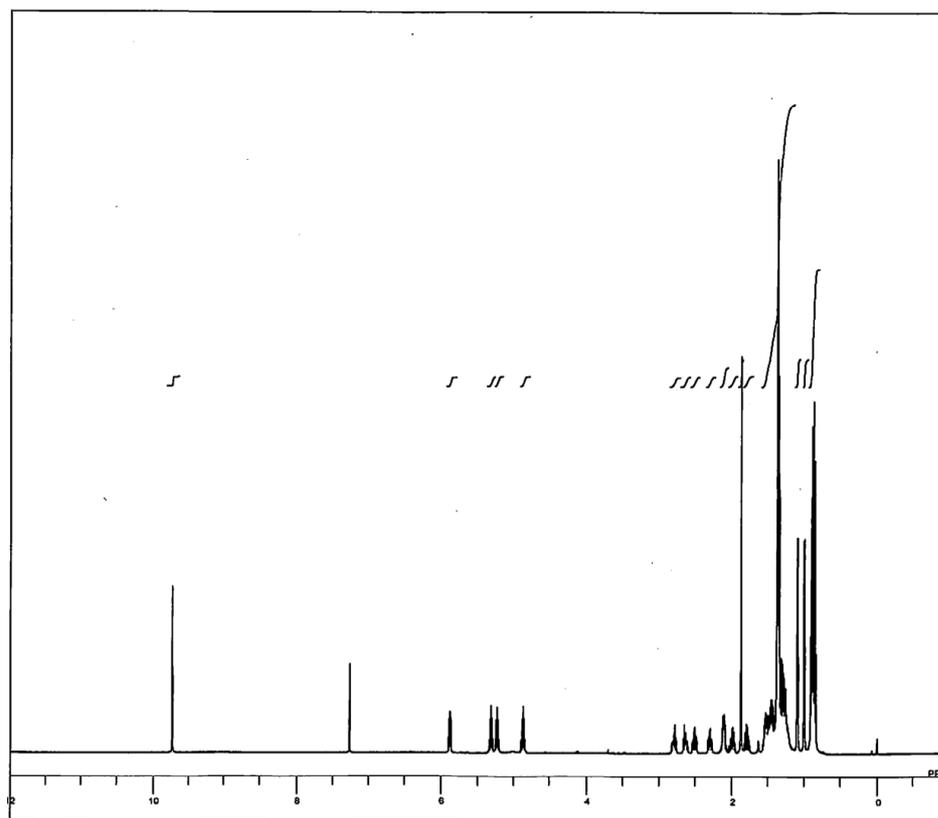
```

MENUF bcm
OBNUC 13C
OFR 125.40 MHz
OBSET 0.00 kHz
OBFIN 143041.00 Hz
PWI 5.50 usec
DEADT 10.00 usec
PREDL 10.00000 msec
RWT 10.00000 sec
POINT 32768
SPO 32768
TIMES 360
DUMMY 1
FREQU 33898.30 Hz
FLT 16950 Hz
DELAY 11.60 usec
ACQTM 0.9667 sec
PD 2.0333 sec
ADBIT 16
RGAIN 30
BF 1.20 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD bcm
EXPCM single pulse with bilvel decoupling
IRNUC 13C
IFR 499.10 MHz
IRSET 0.00 kHz
IRFIN 128250.00 Hz
IRRPW 60 usec
IRATN 511
DFILE yy-17-110-13C.als
SF 125.760
LKSET 0.00 kHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 19
LKPHS 353
LKSIG 519
OSPED 14 Hz
FILDC
FILDF
SLVNT CDCL3
DATM Fri Nov 14 12:38:49 2008
    
```



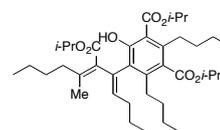
8b-(E)



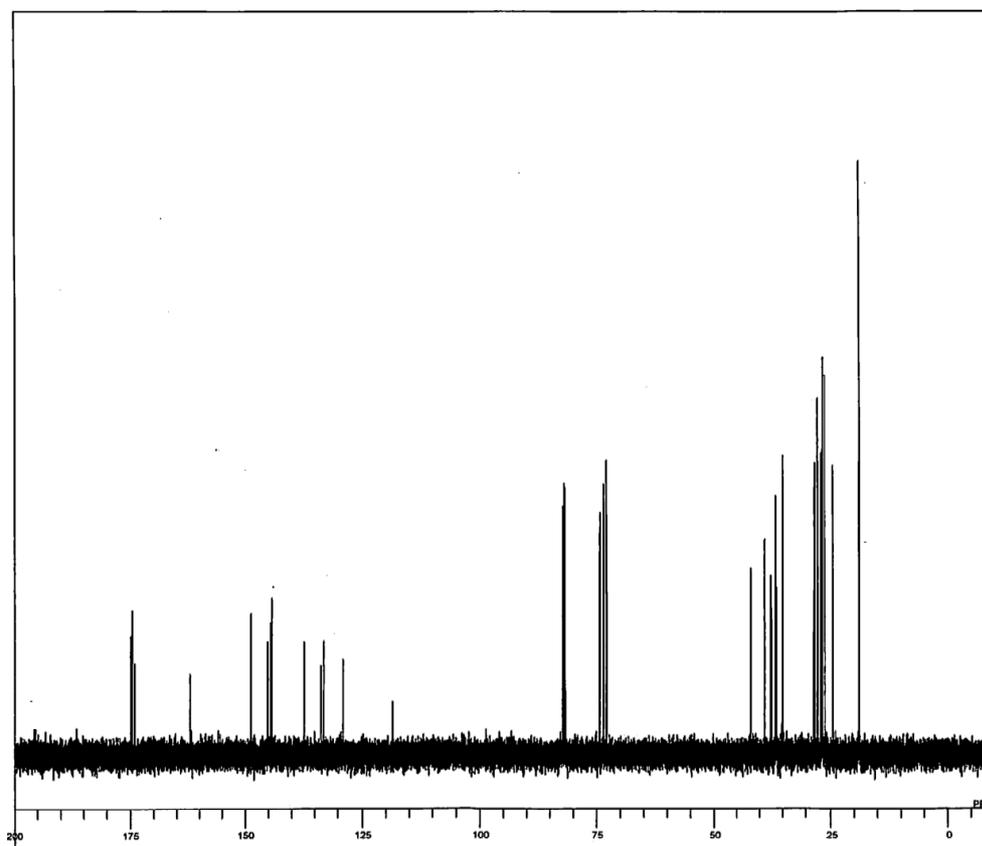


```

MENUF 1H
OBNUC 1H
OFR 499.10 MHz
OBSFET 0.00 kHz
OBFIN 128250.00 Hz
PW1 5.50 usec
DEADT 72.30 usec
PREDL 0.01000 msec
IWT 1000.0000 sec
POINT 65536
SPD 65536
TIMES 8
DUMMY 0
FREQU 8000.00 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 8.1920 sec
PD 1.0000 sec
ADBIT 16
RGAIN 17
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD non
EXPCM single pulse nondecoupling & nonpre-saturation
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 kHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE yy-18-135-4-1H.als
SF th5at
LKSET 0.00 kHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 22
LKPHS 353
LKSIG 869
CSPED 14 Hz
FILDC
FILDF
SLVNT CDCL3
DATIM Thu May 14 09:50:11 2009
    
```

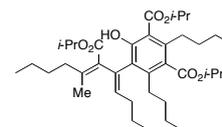


8c-(E)

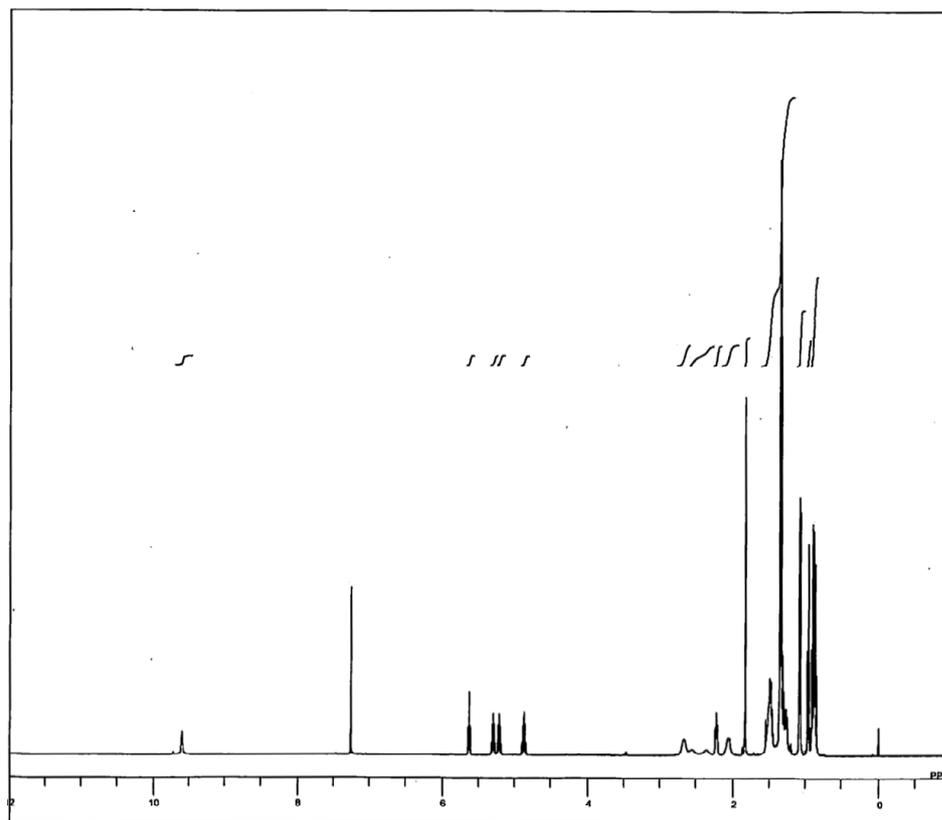


```

MENUF 13C
OBNUC 13C
OFR 125.40 MHz
OBSFET 0.00 kHz
OBFIN 143041.00 Hz
PW1 6.25 usec
DEADT 10.00 usec
PREDL 0.01000 msec
IWT 1000.0000 sec
POINT 65536
SPD 65536
TIMES 2
DUMMY 2
FREQU 33899.30 Hz
FLT 16950 Hz
DELAY 11.80 usec
ACQTM 1.9333 sec
PD 1.0670 sec
ADBIT 16
RGAIN 30
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD bcm
EXPCM single pulse with bilevel decoupling
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 kHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE yy-18-135-4-13C.als
SF th5at
LKSET 0.00 kHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 22
LKPHS 353
LKSIG 869
CSPED 11 Hz
FILDC
FILDF
SLVNT CDCL3
DATIM Thu May 14 09:57:27 2009
    
```

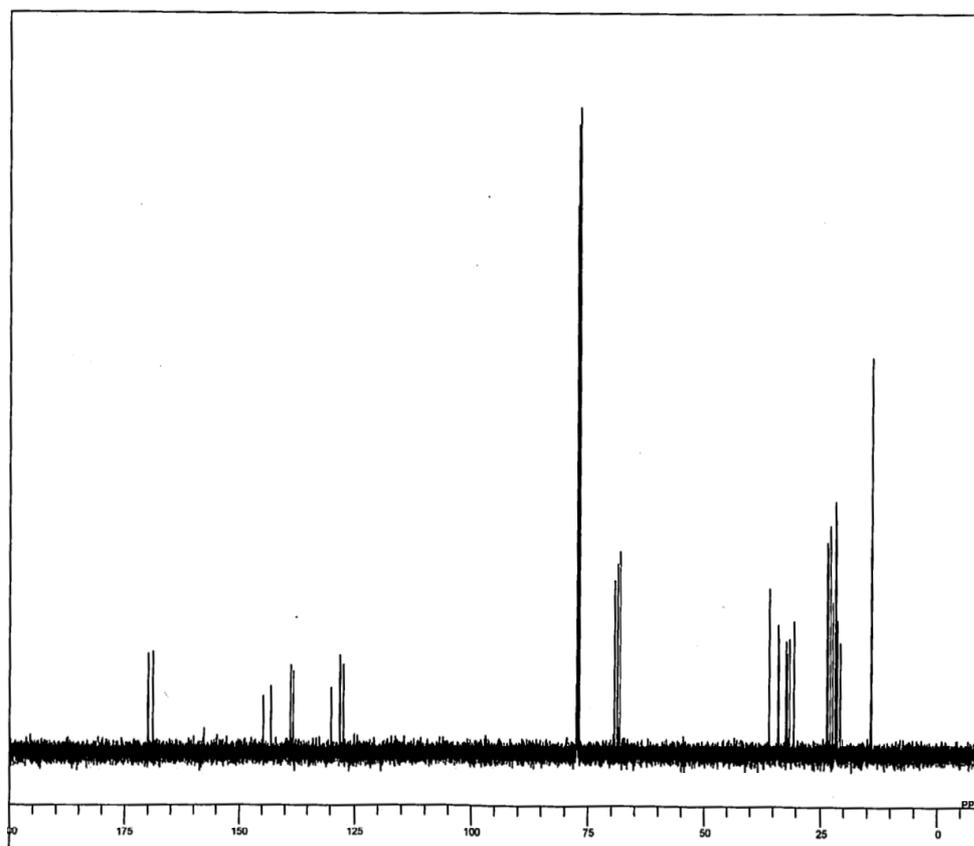
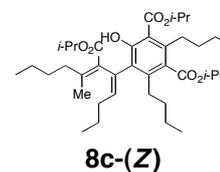


8c-(E)



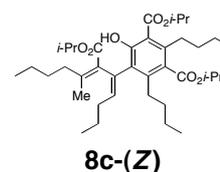
```

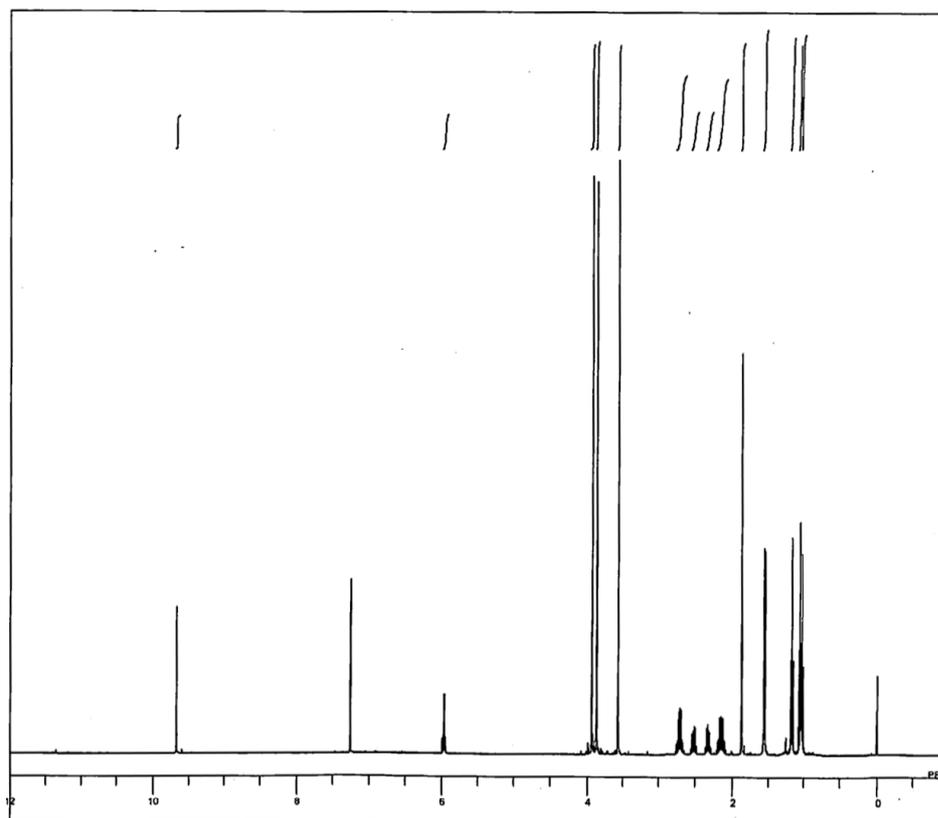
MENUF  non
OBNUC  1H
OFR    499.10 MHz
OBSET  0.00 KHz
OBFIN  128250.00 Hz
PW1    6.10 usec
DEADT  56.95 usec
PREDL  10.0000 msec
IWT    0.5000 sec
POINT  32768
SPO    32768
TIMES  32
DUMMY  0
FREQU  9990.04 Hz
FLT    5000 Hz
DELAY  40.00 usec
ACQTM  3.2834 sec
PD     3.7165 sec
ADBIT  16
RGAIN  21
BF     0.12 Hz
T1    0.00
T2    0.00
T3    90.00
T4    100.00
EXMOD  non
EXPCM  single pulse nondecoupling & nonpresaturation
IRNUC  1H
IFR    499.10 MHz
IRSET  0.00 KHz
IRFIN  128250.00 Hz
IRRPW  50 usec
IRATN  511
DFILE  yy-18-135-3-1H.afs
SF     inst:
LKSET  0.00 KHz
LKFIN  26934.0 Hz
LKLEV  200
LGAIN  19
LKPHS  353
LKSIG  502
CSFED  11 Hz
FILDC  FILDC
FILDF  CDCL3
SLWNT  Fri May 15 01:42:51 2009
DATIM
    
```



```

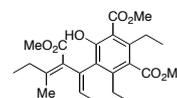
MENUF  bcm
OBNUC  13C
OFR    125.40 MHz
OBSET  0.00 KHz
OBFIN  143041.00 Hz
PW1    5.50 usec
DEADT  10.00 usec
PREDL  10.0000 msec
IWT    10.0000 sec
POINT  32768
SPO    32768
TIMES  1024
DUMMY  1
FREQU  33898.30 Hz
FLT    16950 Hz
DELAY  11.90 usec
ACQTM  0.9667 sec
PD     2.0333 sec
ADBIT  16
RGAIN  31
BF     0.12 Hz
T1    0.00
T2    0.00
T3    90.00
T4    100.00
EXMOD  bcm
EXPCM  single pulse with bilevel decoupling
IRNUC  1H
IFR    499.10 MHz
IRSET  0.00 KHz
IRFIN  128250.00 Hz
IRRPW  65 usec
IRATN  511
DFILE  yy-18-135-3-13C.afs
SF     inst:
LKSET  0.00 KHz
LKFIN  26934.0 Hz
LKLEV  200
LGAIN  19
LKPHS  353
LKSIG  496
CSFED  13 Hz
FILDC  FILDC
FILDF  CDCL3
SLWNT  Fri May 15 02:34:24 2009
DATIM
    
```



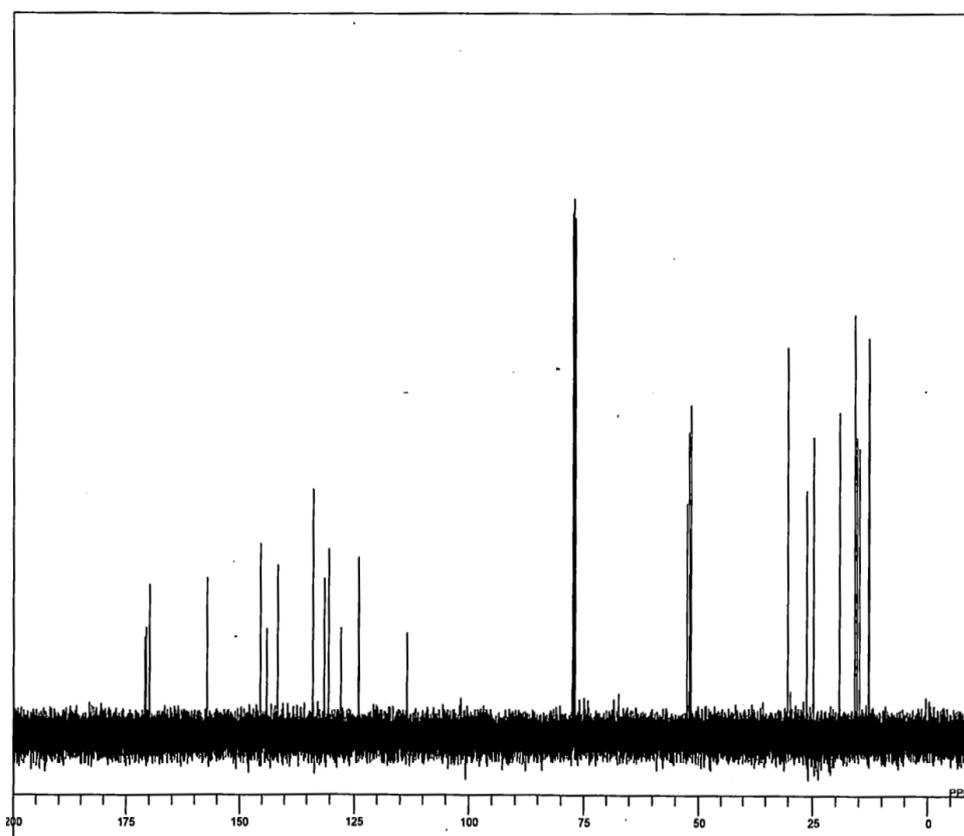


```

MENUF 1H
OBNUC 1H
OPR 499.10 MHz
OBSET 0.00 KHz
OBFIN 128250.00 Hz
PW1 5.50 usec
DEADT 72.30 usec
FREDL 0.01000 msec
IWT 1000.0000 sec
POINT 65536
SPO 40
TIMES 2
DUMMY 0
FREQU 9000.00 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 8.1920 sec
PD 1.0000 sec
ADBIT 16
RGAIN 22
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD non
EXPCM single pulse nondecoupling & nonpresaturation
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE yy-18-133-fr-2-1H.als
SF thSet
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 20
LKPHS 346
LKSIG 575
CSPED 15 Hz
FILDC
FILDF
SLVNT CDCL3
DATIM Tue Aug 18 20:20:32 2009
    
```

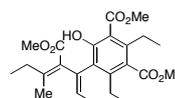


8d-(E)

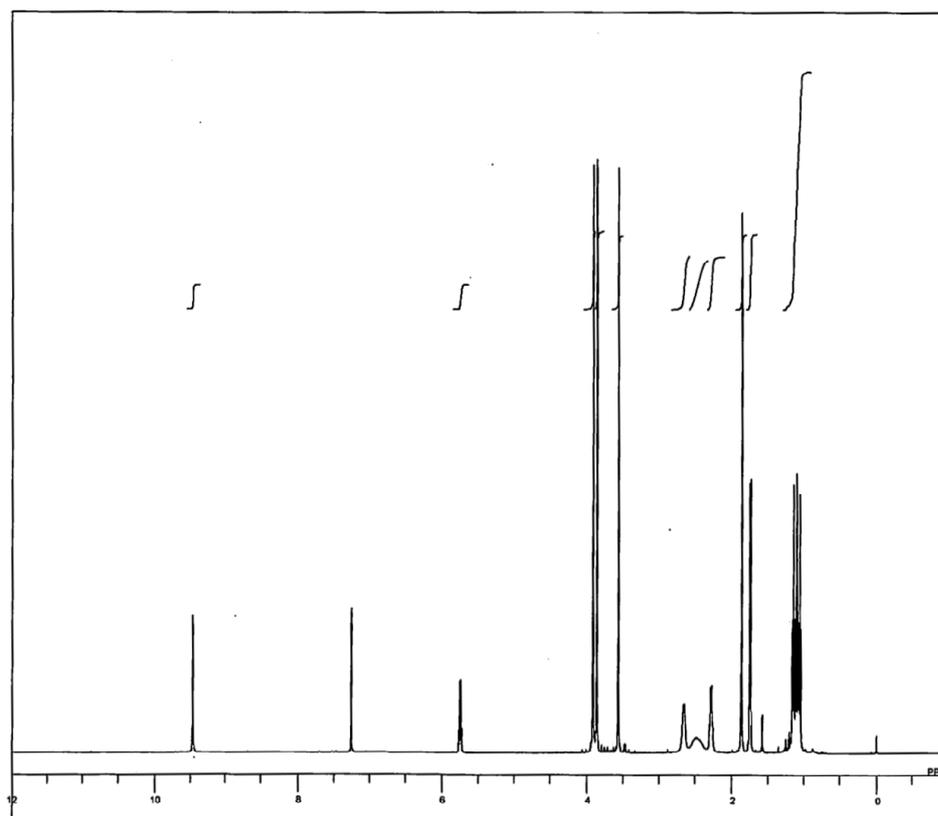


```

MENUF 13C
OBNUC 13C
OPR 125.40 MHz
OBSET 0.00 KHz
OBFIN 143041.00 Hz
PW1 6.25 usec
DEADT 10.00 usec
FREDL 0.01000 msec
IWT 1000.0000 sec
POINT 65536
SPO 40
TIMES 2
DUMMY 2
FREQU 33898.30 Hz
FLT 16950 Hz
DELAY 11.80 usec
ACQTM 1.9333 sec
PD 1.0670 sec
ADBIT 16
RGAIN 31
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD bcm
EXPCM single pulse with bilevel decoupling
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE yy-18-133-2-13C.als
SF thSet
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 20
LKPHS 353
LKSIG 689
CSPED 15 Hz
FILDC
FILDF
SLVNT CDCL3
DATIM Mon May 18 22:28:34 2009
    
```

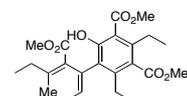


8d-(E)

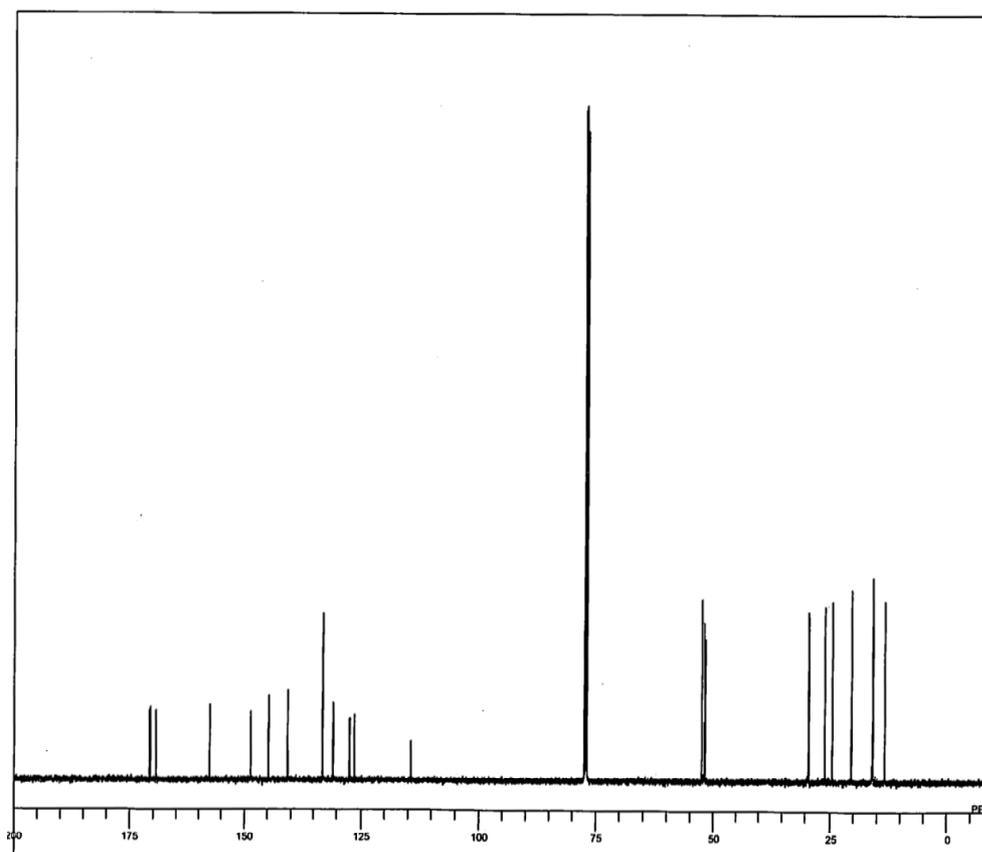


```

MENUF  non
OBNUC  1H
OFR    499.10 MHz
OBSET  0.00 KHz
OBFIN  128250.00 Hz
PW1    8.10 usec
DEADT  56.95 usec
FREDL  10.0000 msec
WVT    0.6000 sec
POINT  32768
SPD    32768
TIMES  32
DUMMY  0
FREQU  9980.04 Hz
FLT    5000 Hz
DELAY  40.00 usec
ACQTM  3.2834 sec
PD     16
ADBIT  16
RGAIN  20
BF     1.20 Hz
T1     0.00
T2     0.00
T3     90.00
T4     100.00
EXMOD  non
EXPCM  single pulse nondecoupling & nonpresaturation
IRNUC  1H
IFR    499.10 MHz
IRSET  0.00 KHz
IRFIN  128250.00 Hz
IRRPW  80 usec
IRATN  511
DFILE  yy-18-133-1-1H.als
SF     0.00 KHz
LKSET  26934.0 Hz
LKFIN  200
LKLEV  19
LGAIN  353
LKPHS  578
LKSIG  14 Hz
OSPED  14 Hz
FILDC  CDCL3
FILDF  Wed May 20 01:19:44 2009
    
```

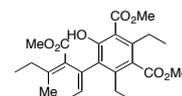


8d-(Z)

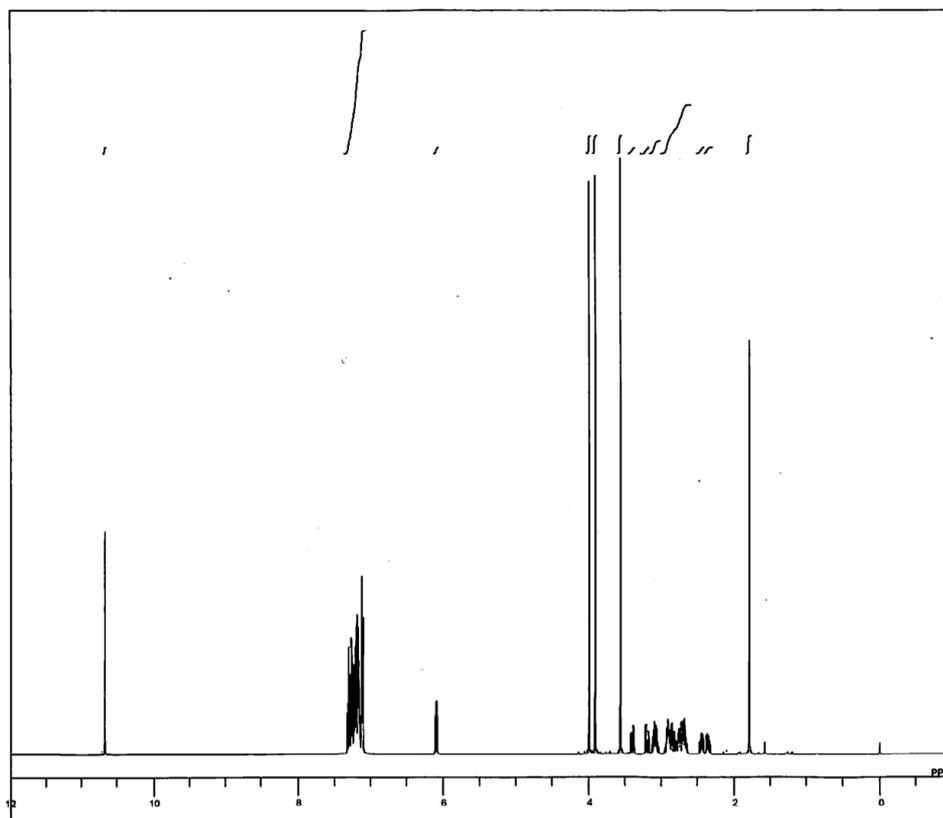


```

MENUF  bcm
OBNUC  13C
OFR    125.40 MHz
OBSET  0.00 KHz
OBFIN  143041.00 Hz
PW1    5.50 usec
DEADT  10.00 usec
FREDL  10.0000 msec
WVT    10.0000 sec
POINT  32768
SPD    32768
TIMES  1600
DUMMY  1
FREQU  33898.30 Hz
FLT    16950 Hz
DELAY  11.50 usec
ACQTM  0.9667 sec
PD     16
ADBIT  16
RGAIN  31
BF     1.20 Hz
T1     0.00
T2     0.00
T3     90.00
T4     100.00
EXMOD  bcm
EXPCM  single pulse with bilvel decoupling
IRNUC  1H
IFR    499.10 MHz
IRSET  0.00 KHz
IRFIN  128250.00 Hz
IRRPW  65 usec
IRATN  511
DFILE  yy-18-133-1-13C.als
SF     0.00 KHz
LKSET  26934.0 Hz
LKFIN  200
LKLEV  19
LGAIN  353
LKPHS  562
LKSIG  12 Hz
OSPED  12 Hz
FILDC  CDCL3
FILDF  Wed May 20 02:40:04 2009
    
```

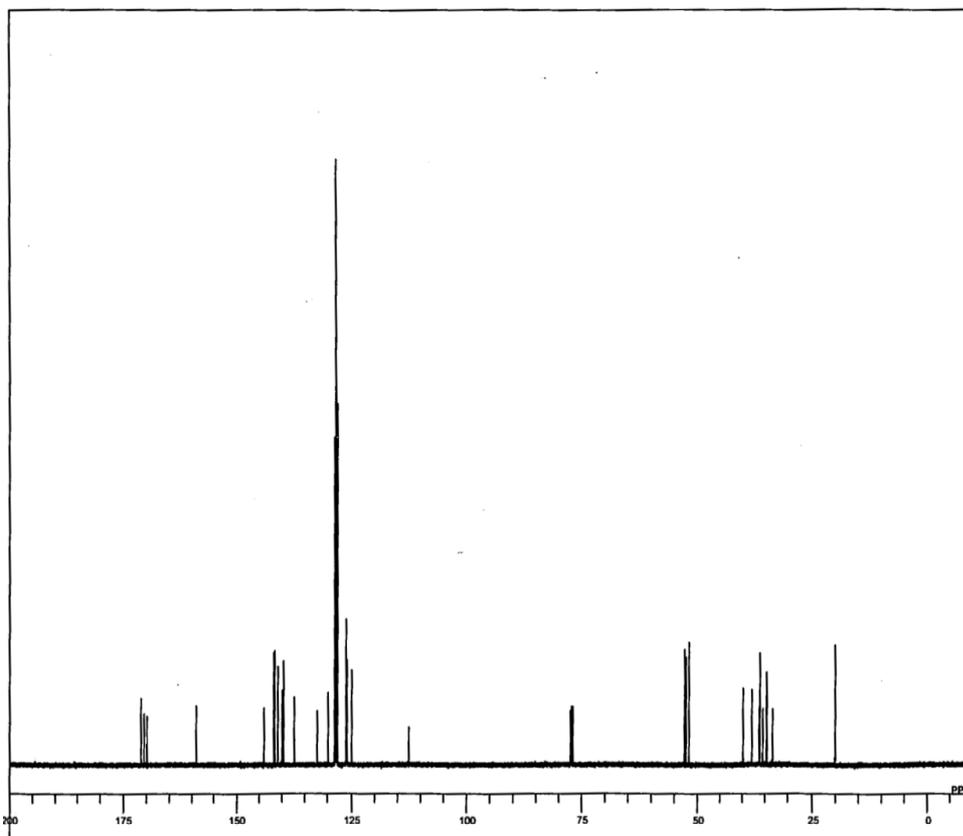
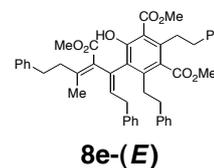


8d-(Z)



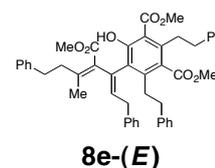
```

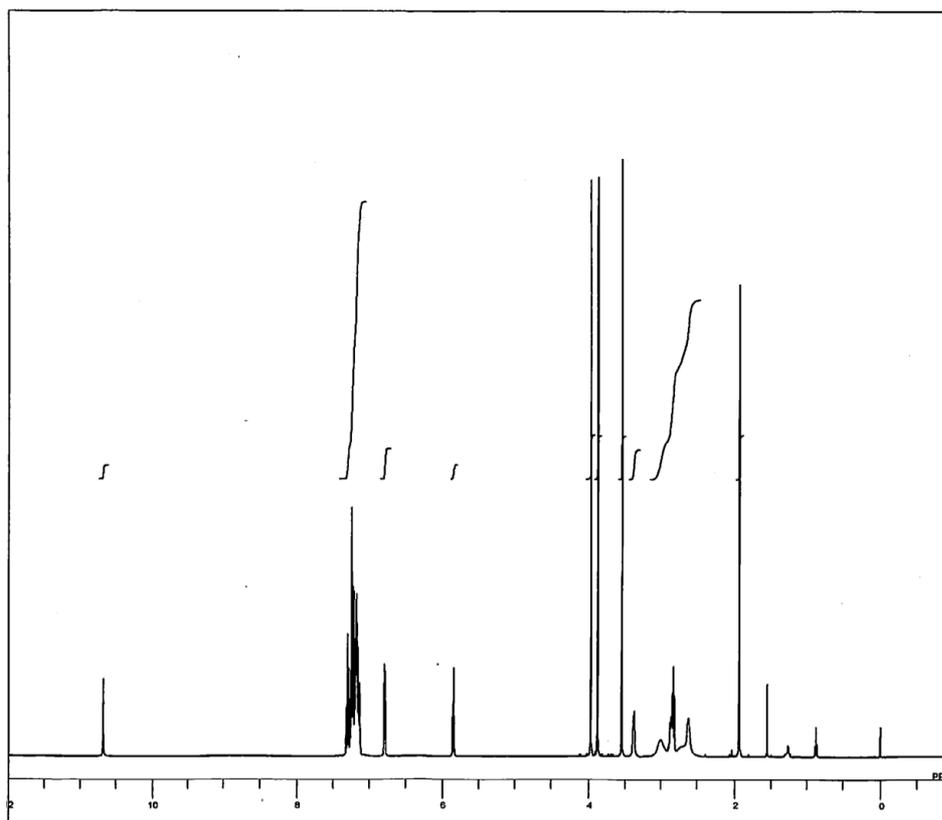
MENUMF 1H
OBNUC 1H
OFR 499.10 MHz
OSSET 0.00 KHz
UBFIN 128885.00 Hz
PW1 5.50 usec
DEADT 72.30 usec
PREDL 0.01000 msec
IWT 1000.0000 sec
POINT 65536
SFO 65536
TIMES 16
DUMMY 0
FREQU 80000.00 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 8.1920 sec
PD 1.0000 sec
ADBIT 16
RGAIN 15
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD non
EXPCM single pulse nondecoupling & nonpresaturation
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE 131-after GPC.als
SF thSat
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 22
LKPHS 353
KPSIG 661
CSPED 11 Hz
FILDC
FILDF
SLVNT CDCl3
DATIM Thu Dec 04 10:37:12 2008
    
```



```

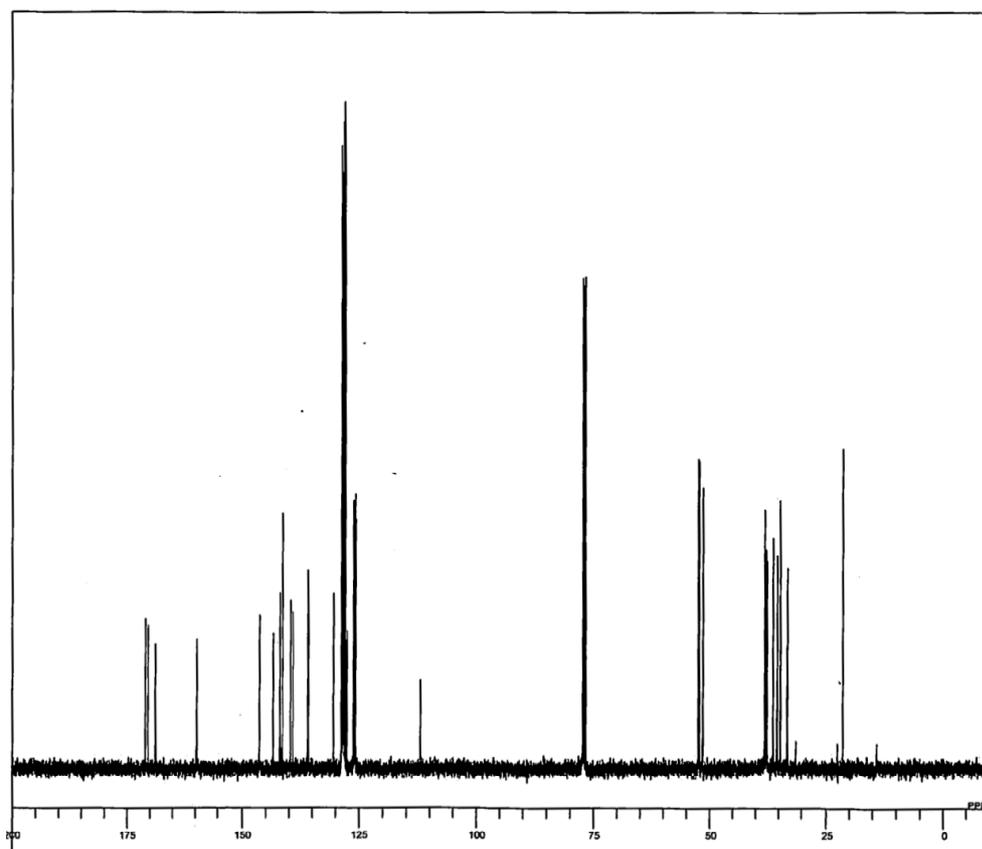
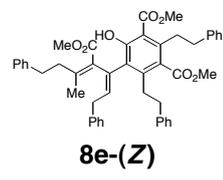
MENUMF 13C
OBNUC 13C
OFR 125.40 MHz
OSSET 0.00 KHz
UBFIN 143041.00 Hz
PW1 6.25 usec
DEADT 10.00 usec
PREDL 0.01000 msec
IWT 1000.0000 sec
POINT 65536
SFO 65536
TIMES 256
DUMMY 2
FREQU 39998.30 Hz
FLT 16950 Hz
DELAY 11.80 usec
ACQTM 1.9333 sec
PD 1.0670 sec
ADBIT 16
RGAIN 30
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD bom
EXPCM single pulse with bilevel decoupling
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE 131-after GPC-13C.als
SF thSat
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 22
LKPHS 353
KPSIG 669
CSPED 11 Hz
FILDC
FILDF
SLVNT CDCl3
DATIM Thu Dec 04 10:50:46 2008
    
```





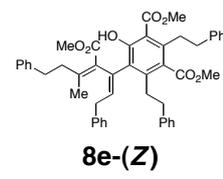
```

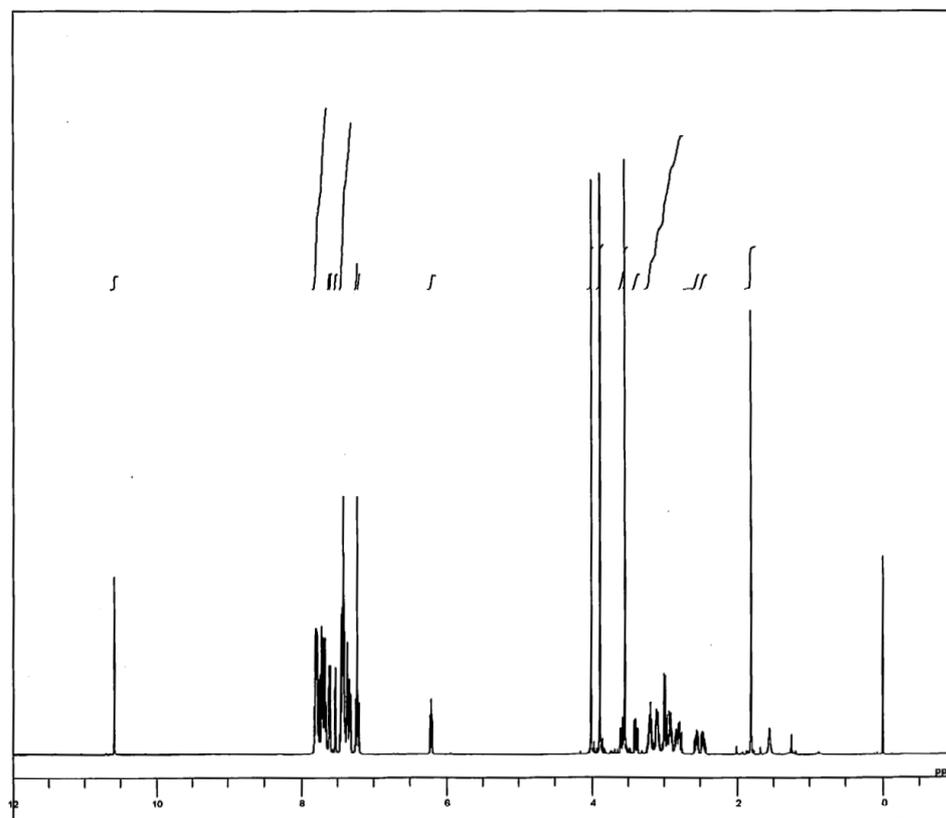
MENUF 1H
OBNUC 1H
OFR 499.10 MHz
OBSET 0.00 KHz
OSFIN 12885.00 Hz
PW1 5.50 usec
DEADT 72.30 usec
FREDL 0.01000 msec
IWT 1000.0000 sec
POINT 65536
SFC1 65536
TIMES 16
DUMMY 0
FREQU 8000.00 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 8.1920 sec
PD 1.0000 sec
ADBIT 16
RGAIN 19
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD non
EXPCM single pulse nondecoupling & nonpresaturation
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE 131-fr-1-H.als
SF 131.41
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 19
LKPHS 353
LKSIG 569
CSPED 12 Hz
FILDC
FILDF
SLVNT CDCl3
DATIM Wed Dec 10 12:11:16 2008
    
```



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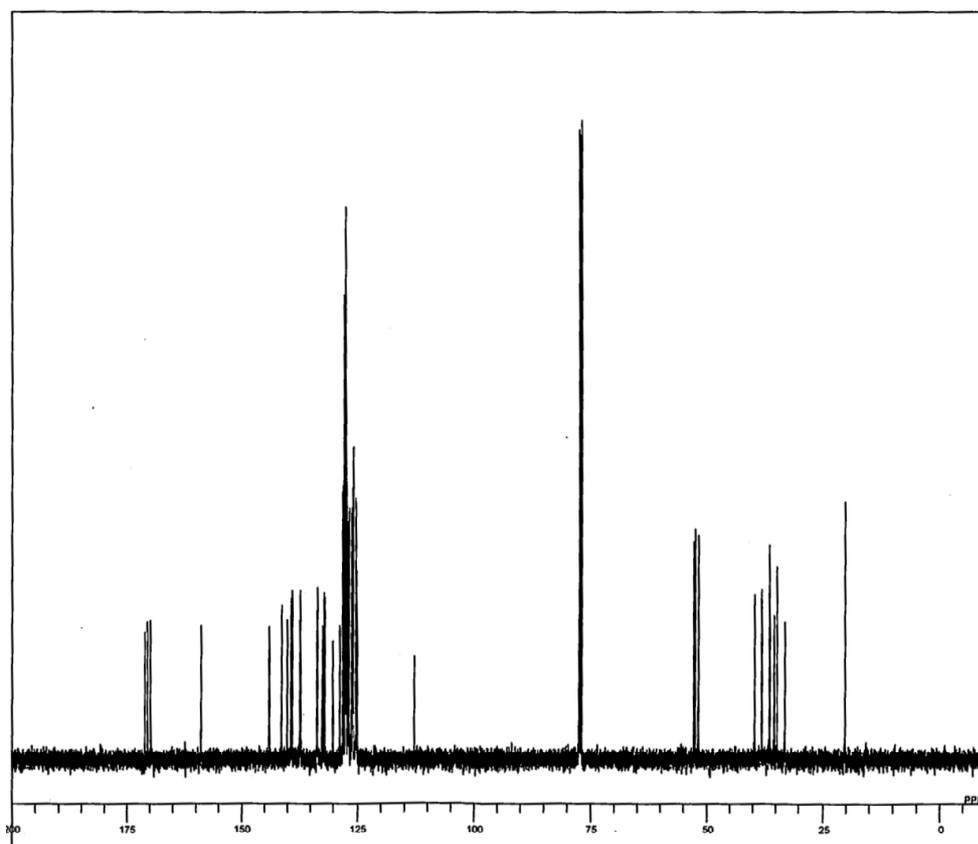
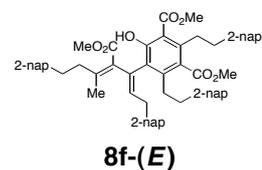
MENUF bcm
OBNUC 13C
OFR 125.40 MHz
OBSET 0.00 KHz
OSFIN 143941.00 Hz
PW1 5.50 usec
DEADT 10.00 usec
FREDL 10.00000 msec
IWT 10.0000 sec
POINT 32768
SFC1 32768
TIMES 512
DUMMY 1
FREQU 33999.30 Hz
FLT 16950 Hz
DELAY 11.80 usec
ACQTM 0.9667 sec
PD 2.0333 sec
ADBIT 16
RGAIN 20
BF 1.20 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD bcm
EXPCM single pulse with bitlevel decoupling
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 65 usec
IRATN 511
DFILE 17-131-fr-13bcm_E1_FT.als
SF 131.41
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 19
LKPHS 353
LKSIG 522
CSPED 12 Hz
FILDC
FILDF
SLVNT CDCl3
DATIM Thu Dec 18 05:09:38 2008
    
```





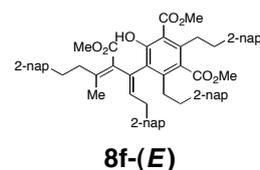
```

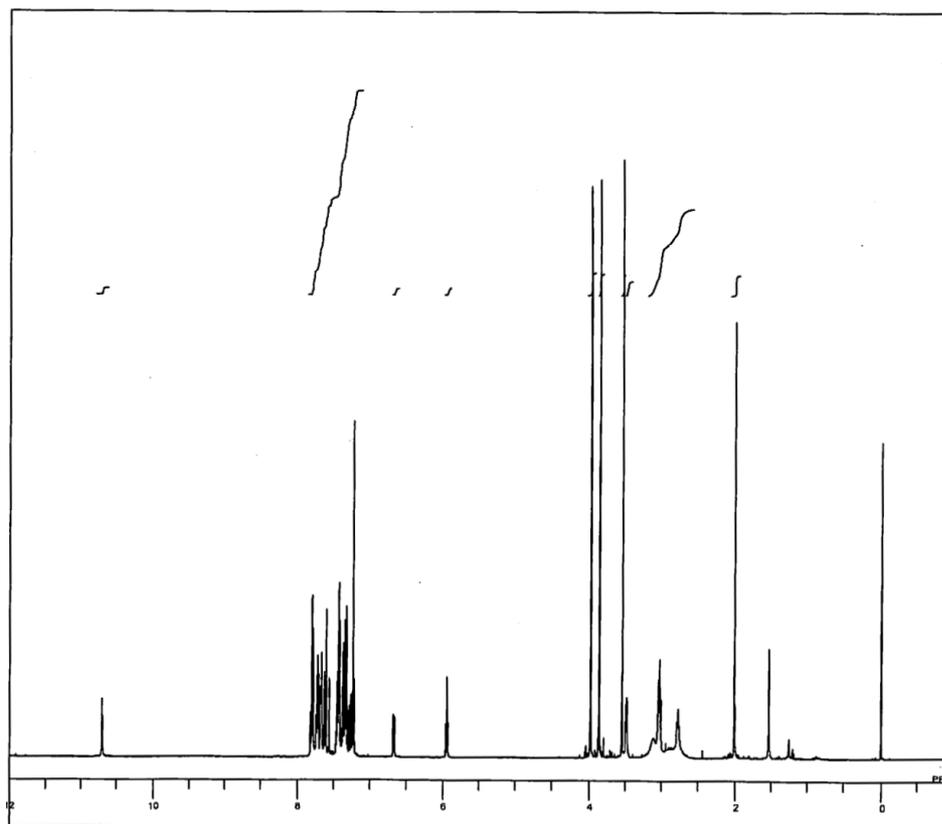
MENUF 1H
OBNUC 1H
OFR 499.10 MHz
OBSET 0.00 KHz
CSFIN 128250.00 Hz
PW1 5.50 usec
DEADT 72.30 usec
PREDL 0.01000 msec
IWT 1000.0000 sec
POINT 65536
SPO 65536
TIMES 32
DUMMY 0
FREQU 6000.00 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 8.1020 sec
PD 1.0000 sec
ADBIT 16
RGAIN 21
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD non
EXPCM single pulse nondecoupling & nonpresaturation
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE yy-18-138-fr-2-1H.als
SF th5at
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 20
LKPHS 346
LKSIG 557
CSPED 11 Hz
FILDC
FILDF
SLVNT CDCL3
DATIM Tue Aug 11 14:38:12 2009
    
```



```

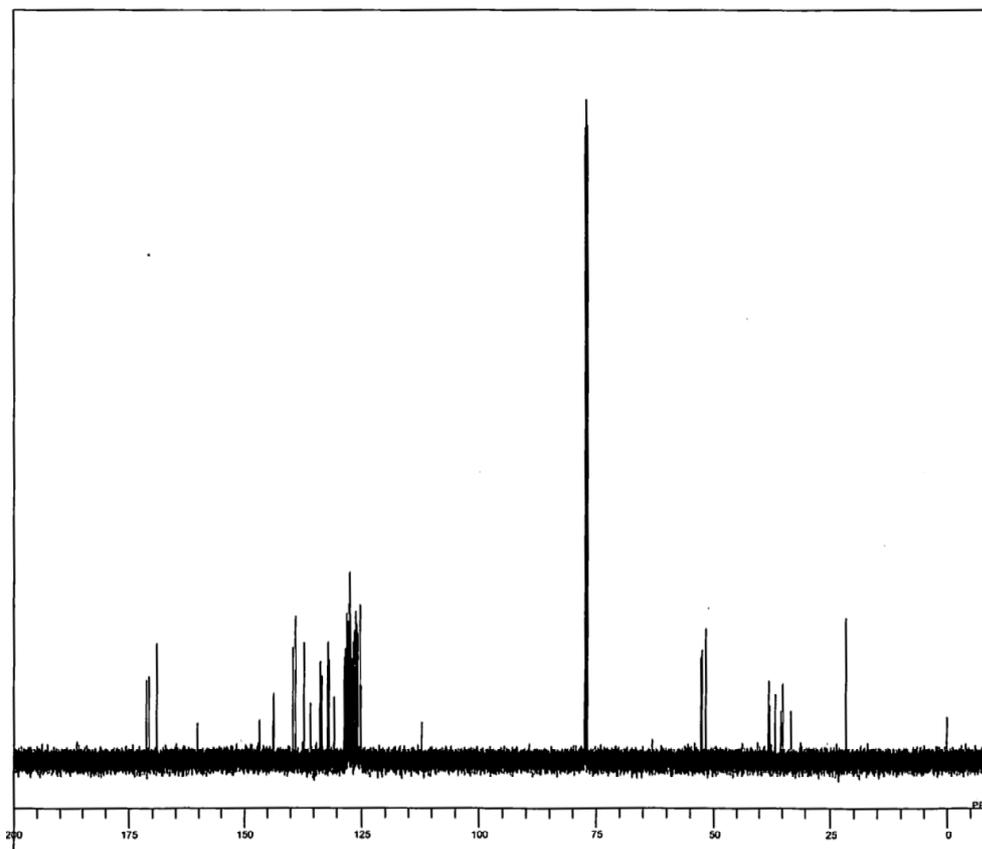
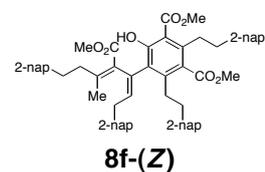
MENUF 13C
OBNUC 13C
OFR 125.40 MHz
OBSET 0.00 KHz
CSFIN 142041.00 Hz
PW1 6.25 usec
DEADT 10.00 usec
PREDL 0.01000 msec
IWT 1000.0000 sec
POINT 65536
SPO 65536
TIMES 256
DUMMY 2
FREQU 33698.30 Hz
FLT 16950 Hz
DELAY 11.80 usec
ACQTM 1.9333 sec
PD 1.0670 sec
ADBIT 16
RGAIN 31
BF 1.20 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD beam
EXPCM single pulse with bilvel decoupling
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE yy-18-138-fr-2-13C.als
SF th5at
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 19
LKPHS 346
LKSIG 454
CSPED 13 Hz
FILDC
FILDF
SLVNT CDCL3
DATIM Tue Aug 11 21:48:41 2009
    
```





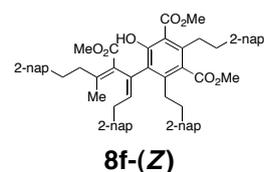
```

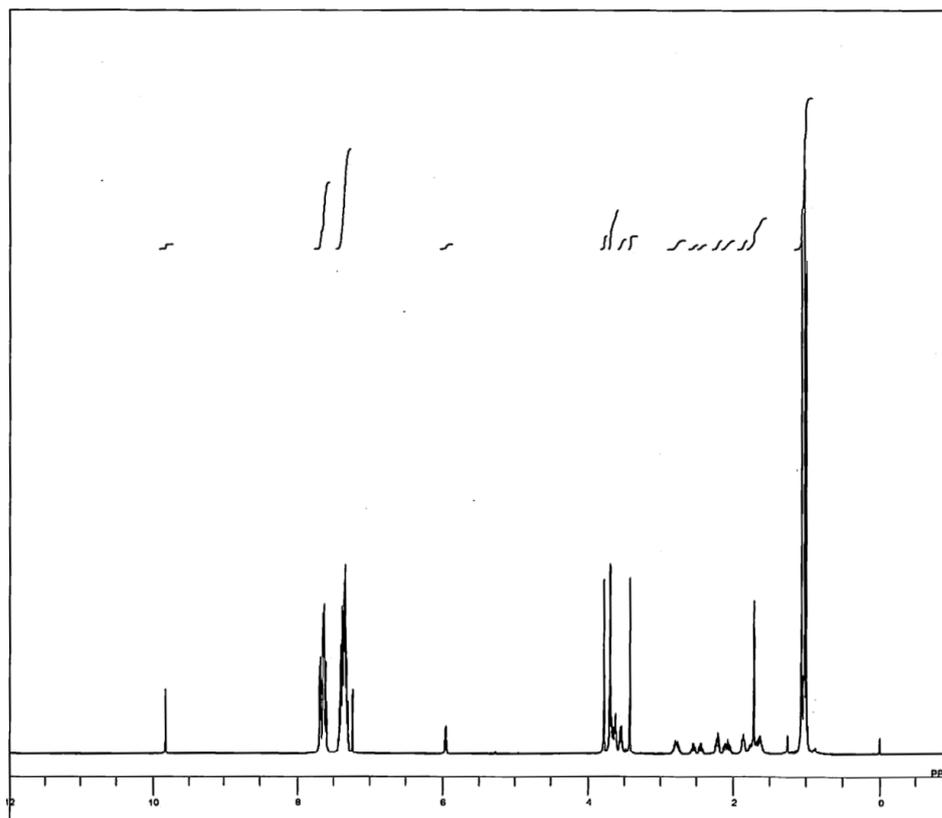
MENUF 1H
OBNUC 1H
OFR 499.10 MHz
OBSET 0.00 KHz
OBFIN 128250.00 Hz
PW1 5.50 usec
DEADT 72.30 usec
PREDL 0.01000 msec
IWT 1000.0000 sec
POINT 65536
SFO 65536
TIMES 32
DUMMY 0
FREQU 4000.00 Hz
FLT 4300 Hz
DELAY 50.00 usec
ACQTM 8.1920 sec
PD 1.0000 sec
ADBIT 16
RGAIN 22
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD non
EXPCM single pulse nondecoupling & nonpresaturation
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE yy-18-138-fr-1-1H.als
SF 65536
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LQAIN 22
LKPHS 346
LKSIG 941
CSPED 14 Hz
FILDC
FILDF
SLVNT CDCL3
DATM Tue Aug 11 19:03:51 2009
    
```



```

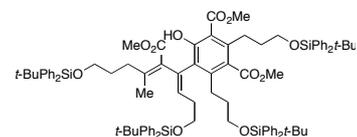
MENUF 13C
OBNUC 13C
OFR 125.40 MHz
OBSET 0.00 KHz
OBFIN 143041.00 Hz
PW1 6.25 usec
DEADT 10.00 usec
PREDL 0.01000 msec
IWT 1000.0000 sec
POINT 65536
SFO 65536
TIMES 512
DUMMY 2
FREQU 33898.30 Hz
FLT 16950 Hz
DELAY 11.80 usec
ACQTM 1.9333 sec
PD 1.0670 sec
ADBIT 16
RGAIN 31
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD bon
EXPCM single pulse with bilevel decoupling
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE yy-18-138-fr-1-13C.als
SF 65536
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LQAIN 22
LKPHS 346
LKSIG 924
CSPED 13 Hz
FILDC
FILDF
SLVNT CDCL3
DATM Tue Aug 11 19:30:32 2009
    
```



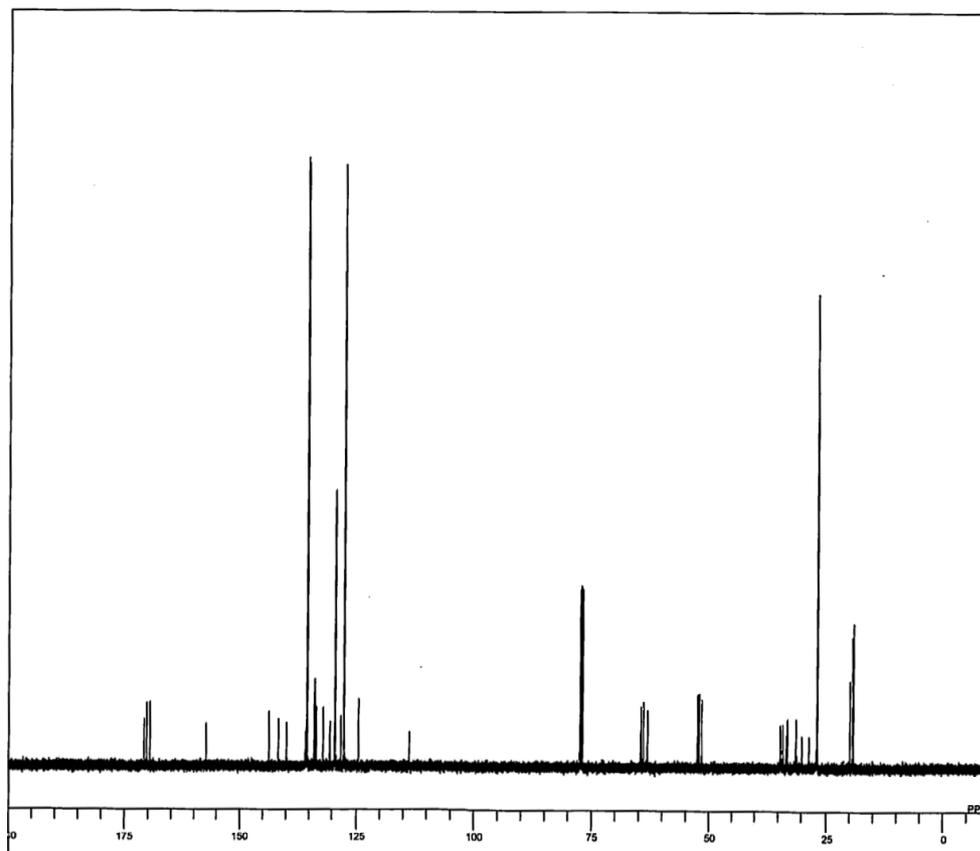


```

MENUF 1H
OBNUC 1H
OFR 499.10 MHz
OBSET 0.00 KHz
OSPIN 12885.00 Hz
PW1 5.50 usec
DEADT 72.30 usec
PREDL 0.01000 msec
IWT 1000.0000 sec
POINT 65536
SPD 65536
TIMES 16
DUMMY 0
FREQU 8000.00 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 8.1920 sec
PD 1.0000 sec
ADBIT 16
RGAIN 18
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD non
EXPCM single pulse nondecoupling & nonpresaturation
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE yy-18-130-3-1H.als
SF 1000.0000
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 22
LKPHS 353
LKSIG 859
CSFED 12 Hz
FILDC
FILDF
SLVNT CDCL3
DATM Wed May 13 21:50:24 2009
    
```

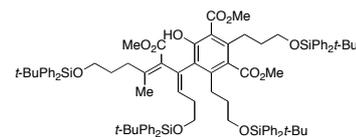


8g-(E)

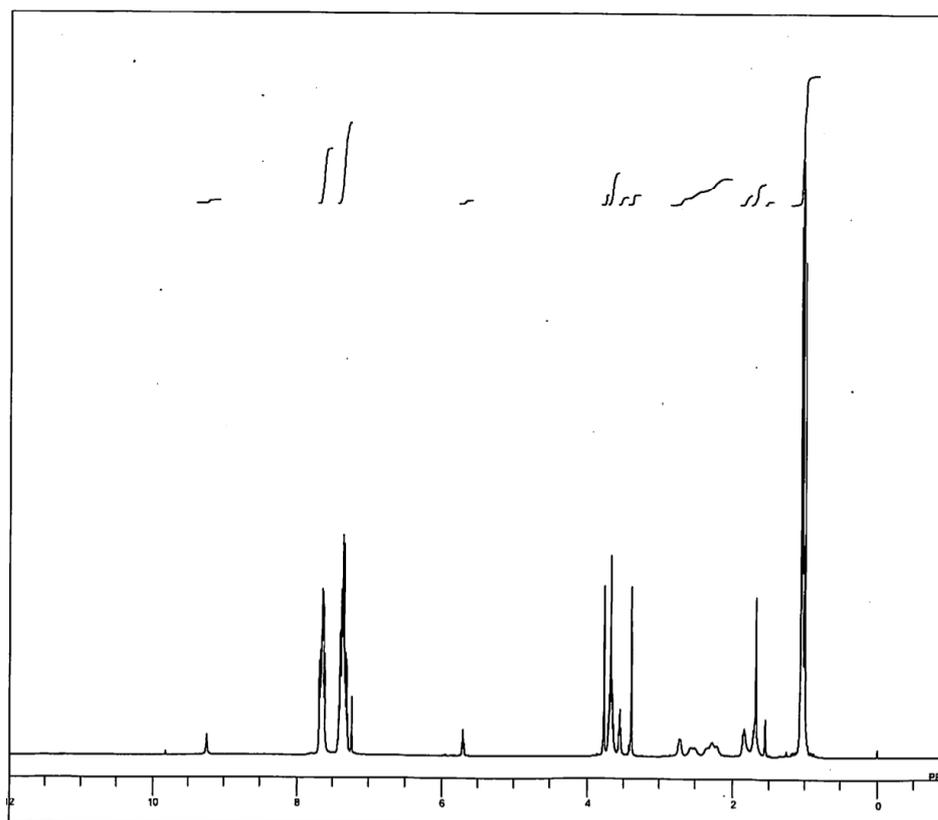


```

MENUF 13C
OBNUC 13C
OFR 125.40 MHz
OBSET 0.00 KHz
OSPIN 143041.00 Hz
PW1 6.25 usec
DEADT 10.00 usec
PREDL 0.01000 msec
IWT 1000.0000 sec
POINT 65536
SPD 65536
TIMES 350
DUMMY 2
FREQU 33898.30 Hz
FLT 16950 Hz
DELAY 11.00 usec
ACQTM 1.9333 sec
PD 1.0670 sec
ADBIT 16
RGAIN 28
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD bcm
EXPCM single pulse with bilevel decoupling
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE yy-18-130-3-13C.als
SF 1000.0000
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 20
LKPHS 353
LKSIG 571
CSFED 11 Hz
FILDC
FILDF
SLVNT CDCL3
DATM Wed May 13 12:28:24 2009
    
```

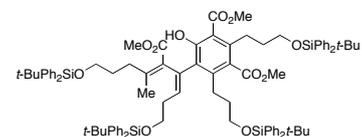


8g-(E)

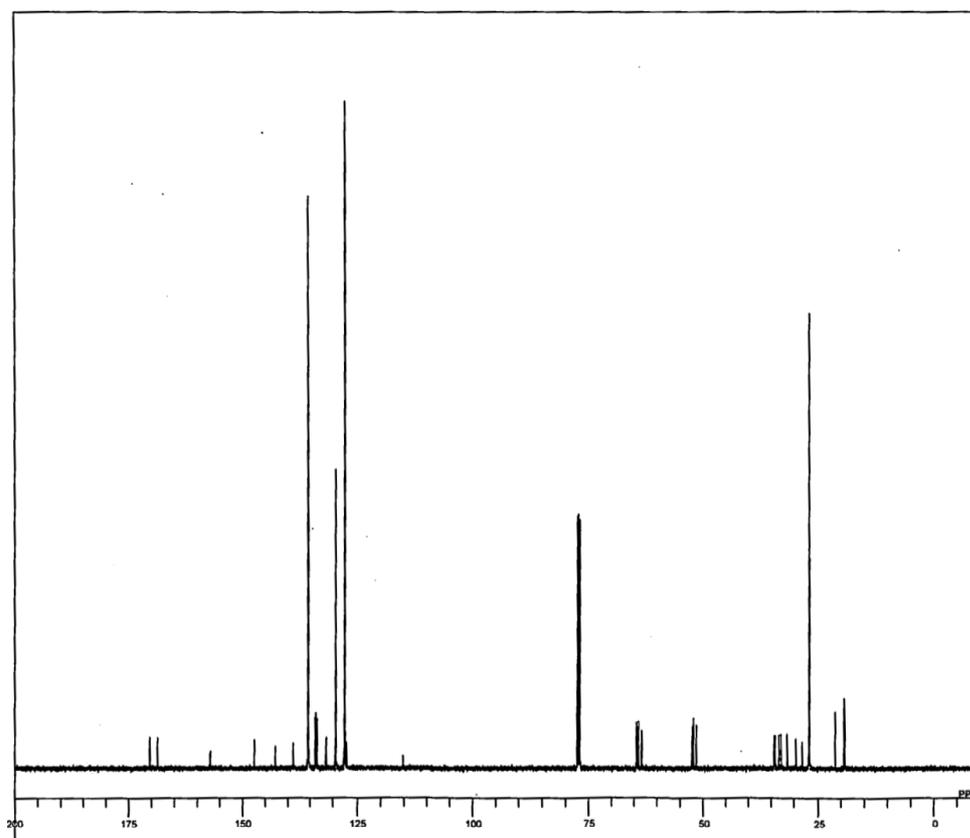


```

MENUF  non
OBNUC  1H
OFR    499.10 MHz
OBSET  0.00 KHz
OBFIN  128250.00 Hz
PWI    5.10 usec
DEADT  56.95 usec
PREDL  10.00000 msec
WT     0.50000 sec
POINT  32768
SPD    32768
TIMES  16
DUMMY  0
FREQU  9980.04 Hz
FLT    5000 Hz
DELAY  40.00 usec
ACQTM  3.2834 sec
PD     3.7166 sec
ADBIT  16
RGAIN  16
BF     1.20 Hz
T1     0.00
T2     0.00
T3     90.00
T4     100.00
EXMOD  non
EXPCM  single pulse nondecoupling & nonpresaturation
IRNUC  1H
IFR    499.10 MHz
IRSET  0.00 KHz
IRFIN  128250.00 Hz
IRRPW  50 usec
IRATN  511
DFILE  yy-18-130-2-1H.als
SF     0.00 KHz
LKSET  0.00 KHz
LKFIN  26934.0 Hz
LKLEV  200
LQAIN  20
LKPHS  353
LKSIG  575
12 Hz
CSPED  12 Hz
FILDG  CDCL3
FILDG  CDCL3
SLWNT  Wed May 13 00:18:03 2009
DATIM  Wed May 13 00:18:03 2009
    
```

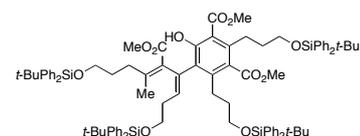


8g-(Z)

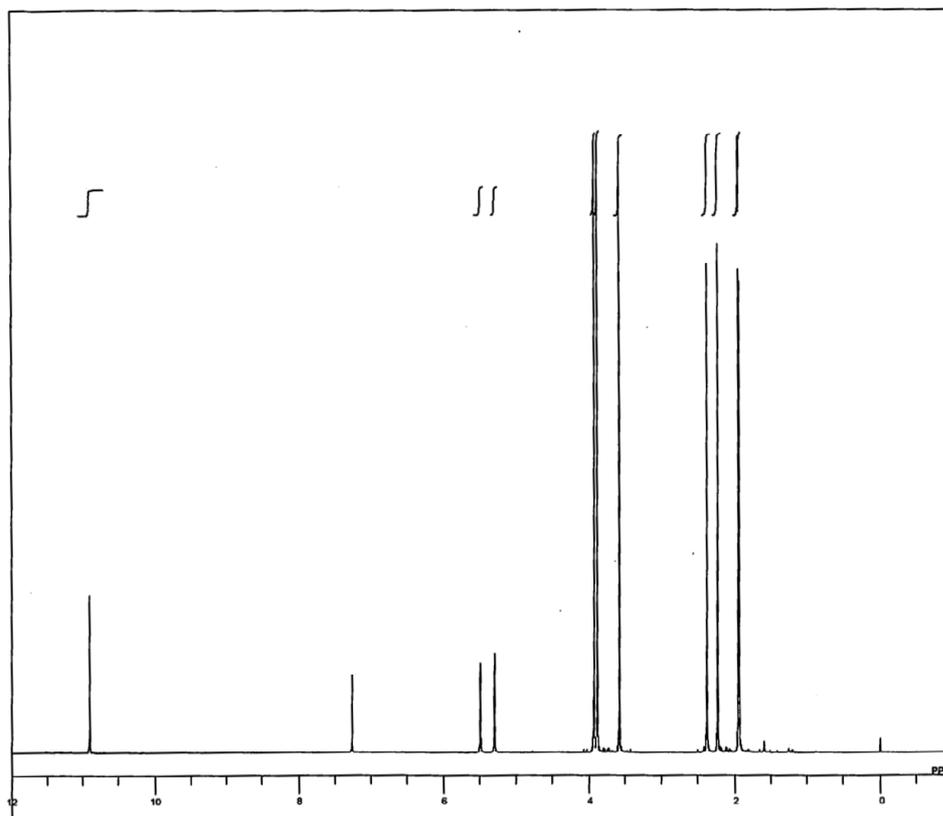


```

MENUF  bcm
OBNUC  13C
OFR    125.40 MHz
OBSET  0.00 KHz
OBFIN  143041.00 Hz
PWI    5.50 usec
DEADT  10.00 usec
PREDL  10.00000 msec
WT     10.00000 sec
POINT  32768
SPD    32768
TIMES  1012
DUMMY  1
FREQU  33898.30 Hz
FLT    16950 Hz
DELAY  11.80 usec
ACQTM  0.9687 sec
PD     2.0333 sec
ADBIT  16
RGAIN  29
BF     1.20 Hz
T1     0.00
T2     0.00
T3     90.00
T4     100.00
EXMOD  bcm
EXPCM  single pulse with bilevel decoupling
IRNUC  13C
IFR    499.10 MHz
IRSET  0.00 KHz
IRFIN  128250.00 Hz
IRRPW  65 usec
IRATN  511
DFILE  yy-18-130-2-13C.als
SF     0.00 KHz
LKSET  0.00 KHz
LKFIN  26934.0 Hz
LKLEV  200
LQAIN  20
LKPHS  353
LKSIG  559
12 Hz
CSPED  12 Hz
FILDG  CDCL3
FILDG  CDCL3
SLWNT  Wed May 13 01:08:56 2009
DATIM  Wed May 13 01:08:56 2009
    
```

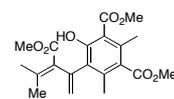


8g-(Z)

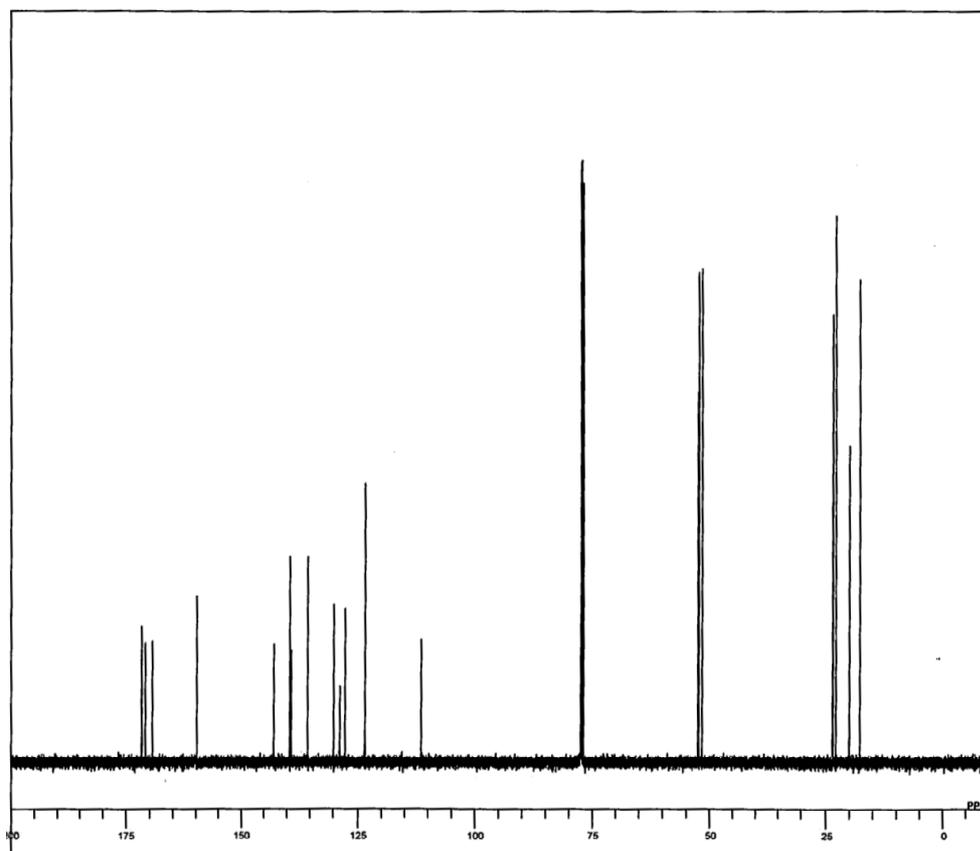


```

MENUF non
OBNUC 1H
OFR 499.10 MHz
OBSET 0.00 KHz
OSPIN 128250.00 Hz
PW1 6.10 usec
DEADT 56.95 usec
FREDL 10.0000 msec
IWT 0.5000 sec
POINT 32768
SFO 32768
TIMES 16
DUMMY 0
FREQU 9960.04 Hz
FLT 5000 Hz
DELAY 40.00 usec
ACQTM 3.2634 sec
PD 3.7166 sec
ADBIT 16
RGAIN 20
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD non
EXPCM single pulse nondecoupling & nonpresaturation
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE yy-18-131-1H.als
SF 0.00 KHz
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 22
LKPHS 353
LKSIG 922
CSPED 12 Hz
FILDC
FILDF
SLVNT CDCL3
DATIM Sat May 16 02:18:12 2009
    
```

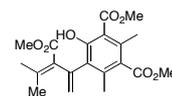


8h

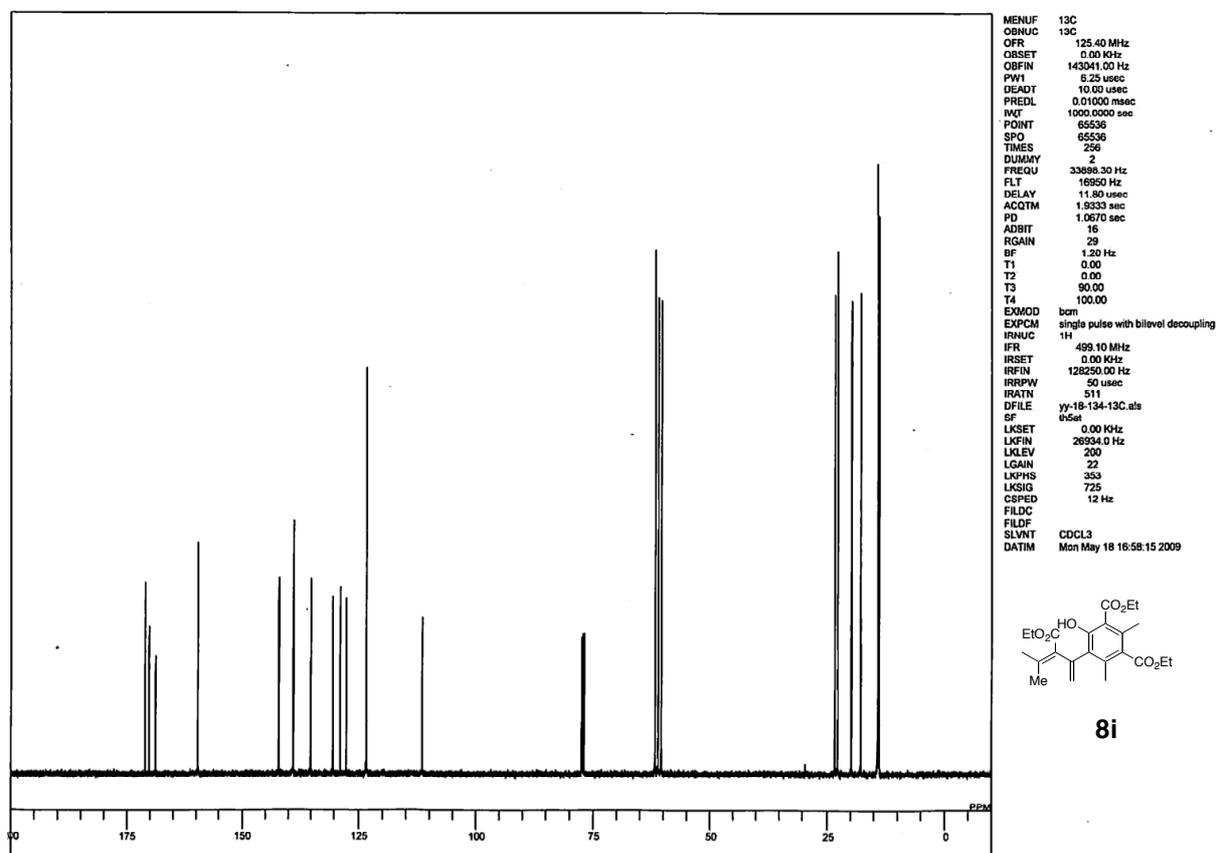
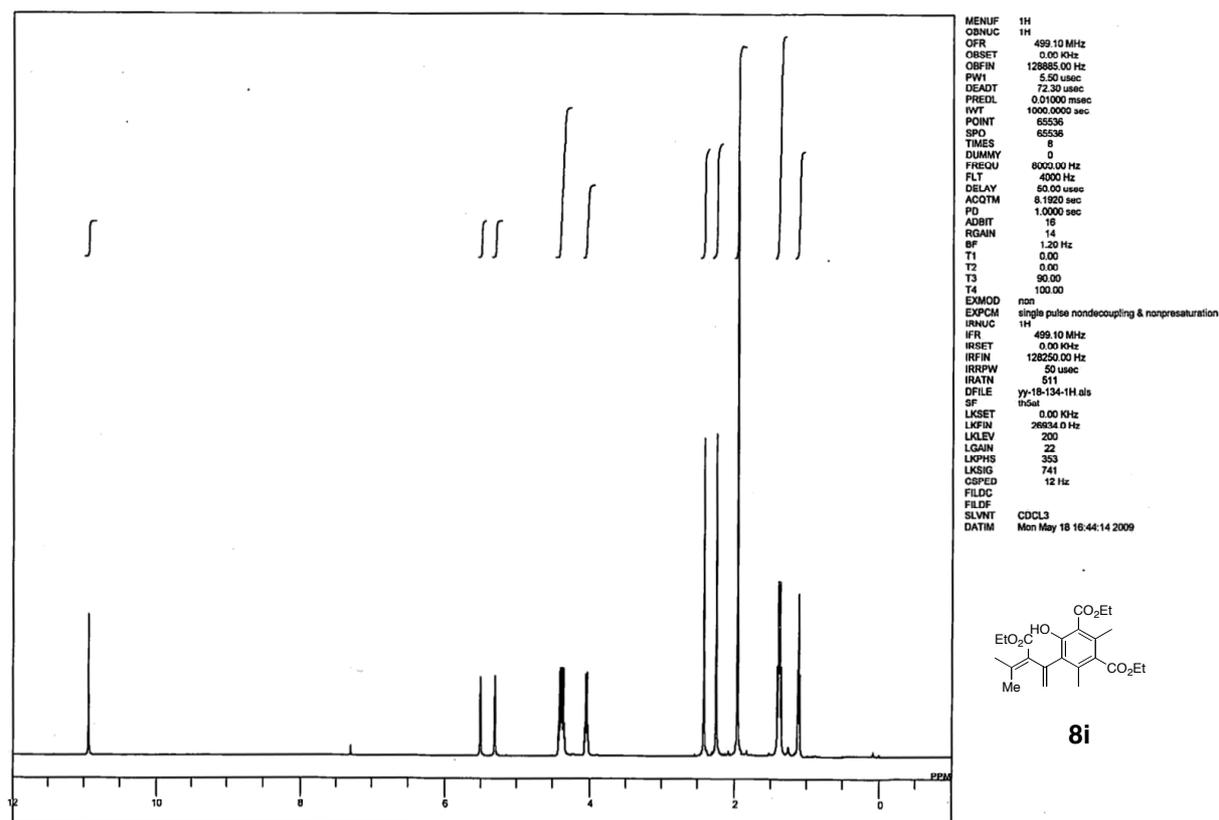


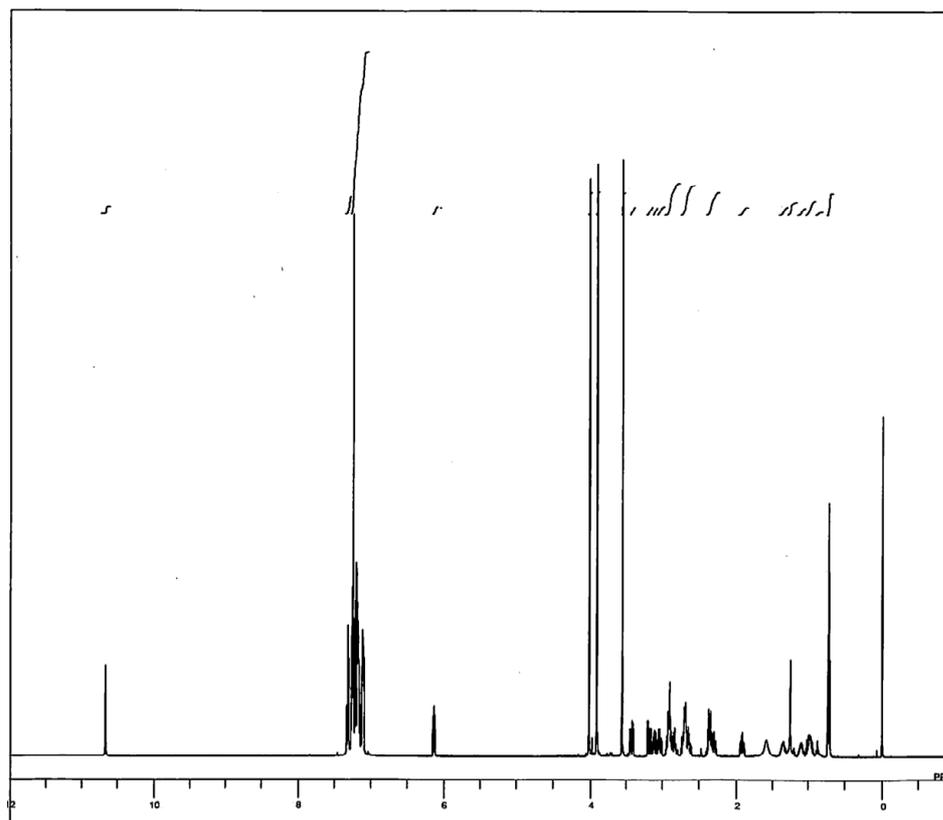
```

MENUF bcm
OBNUC 13C
OFR 125.40 MHz
OBSET 0.00 KHz
OBFIN 143041.00 Hz
PW1 5.50 usec
DEADT 10.00 usec
FREDL 10.0000 msec
IWT 10.0000 sec
POINT 32768
SFO 32768
TIMES 1536
DUMMY 1
FREQU 33696.30 Hz
FLT 16950 Hz
DELAY 11.60 usec
ACQTM 0.9607 sec
PD 2.0333 sec
ADBIT 16
RGAIN 31
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD bcm
EXPCM single pulse with bilevel decoupling
IRNUC 13C
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 65 usec
IRATN 511
DFILE yy-18-131-13C.als
SF 0.00 KHz
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 22
LKPHS 353
LKSIG 923
CSPED 12 Hz
FILDC
FILDF
SLVNT CDCL3
DATIM Sat May 16 03:36:21 2009
    
```



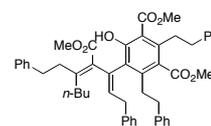
8h



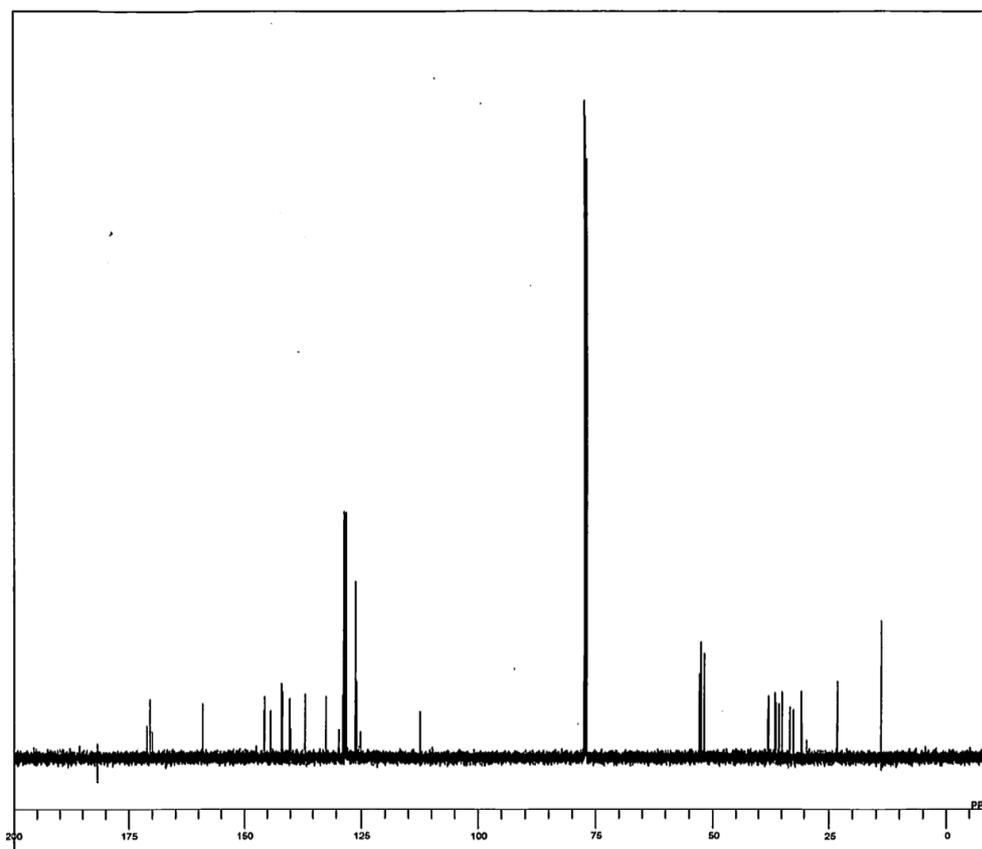


```

MENUF 1H
OBNUC 1H
OFR 499.10 MHz
OBSET 0.00 KHz
OBFIN 128250.00 Hz
PW1 5.50 usec
DEADT 72.30 usec
PREDL 0.01000 msec
IWT 1000.0000 sec
POINT 65538
SPD 65538
TIMES 40
DUMMY 0
FREQU 8000.00 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 8.1920 sec
PD 1.0000 sec
ADBIT 16
RGAIN 24
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD ncn
EXPCM single pulse nondecoupling & nonpresaturation
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE yy-18-136-rev-fr-2-1H.als
SF 0.00 KHz
LKSET 26934.0 Hz
LKFIN 26934.0 Hz
LKLEV 20
LGAIN 20
LKPHS 348
LKSIG 680
CSPED 13 Hz
FILDC
FILDF
SLVNT CDCL3
DATIM Thu Aug 20 11:11:28 2009
    
```

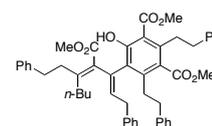


10-(E)

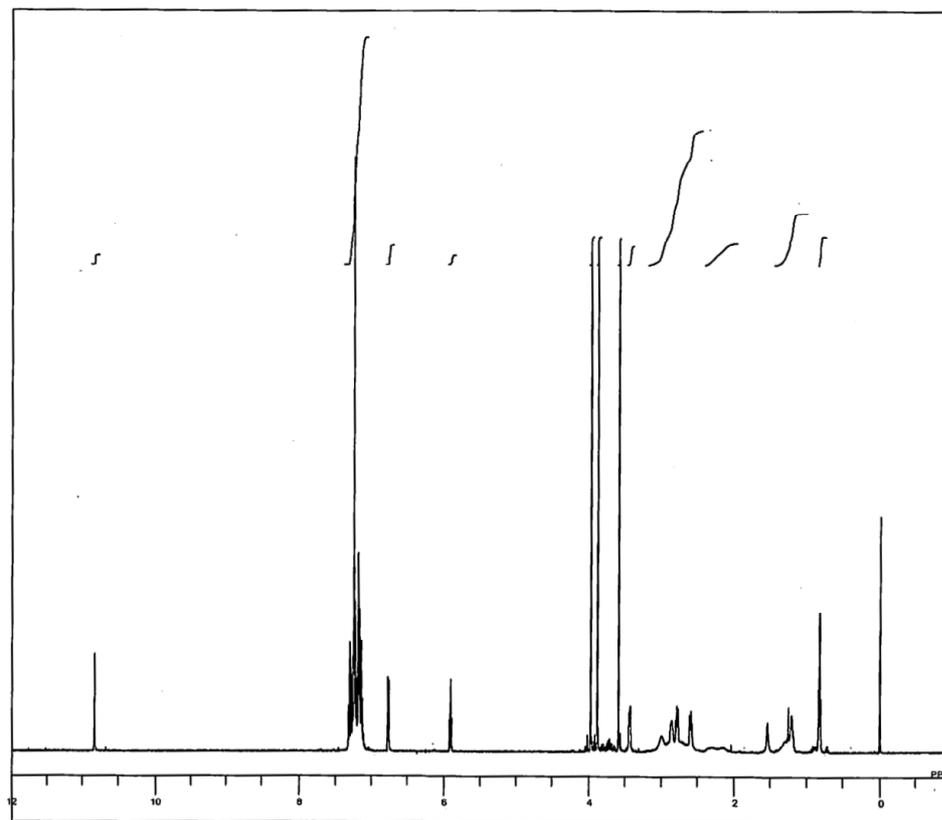


```

MENUF bcm
OBNUC 13C
OFR 125.40 MHz
OBSET 0.00 KHz
OBFIN 143041.00 Hz
PW1 5.50 usec
DEADT 10.00 usec
PREDL 10.00000 msec
IWT 10.0000 sec
POINT 32768
SPD 32768
TIMES 1024
DUMMY 1
FREQU 33898.30 Hz
FLT 16550 Hz
DELAY 11.80 usec
ACQTM 0.8667 sec
PD 2.0533 sec
ADBIT 16
RGAIN 30
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD bcm
EXPCM single pulse with bilevel decoupling
IRNUC 13C
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 65 usec
IRATN 511
DFILE yy-18-136-rev-fr-2-13C.als
SF 0.00 KHz
LKSET 26934.0 Hz
LKFIN 26934.0 Hz
LKLEV 20
LGAIN 20
LKPHS 348
LKSIC 545
CSPED 10 Hz
FILDC
FILDF
SLVNT CDCL3
DATIM Thu Aug 20 12:57:14 2009
    
```

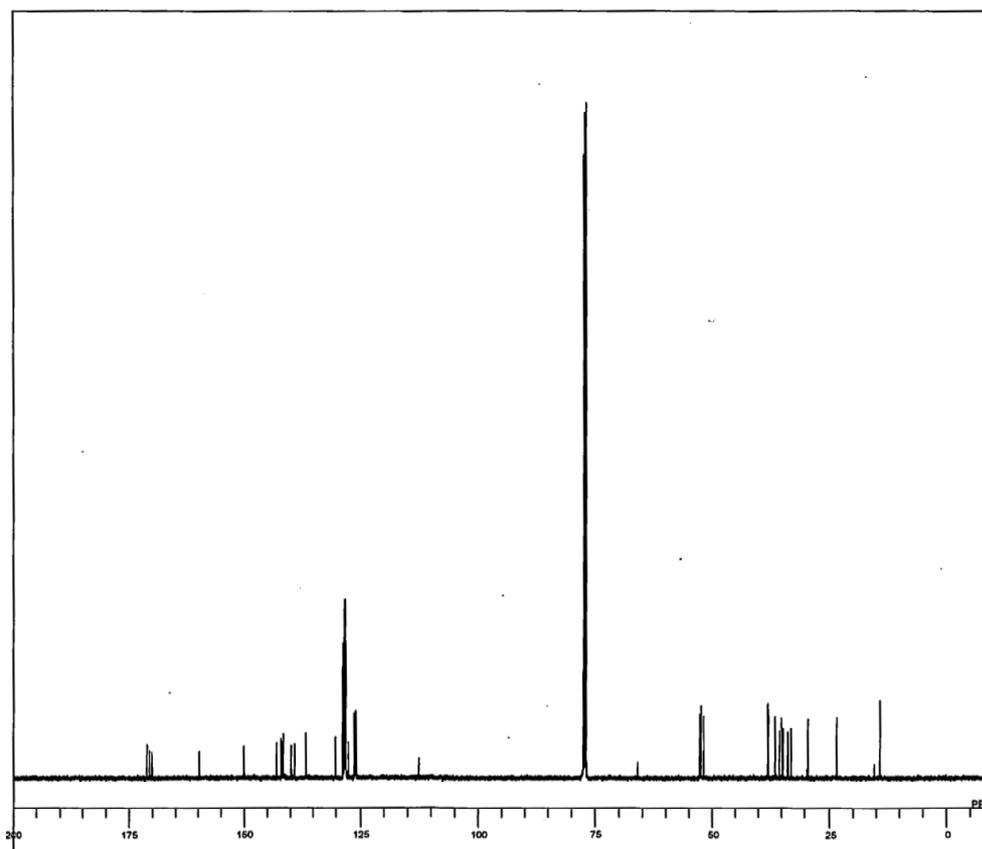
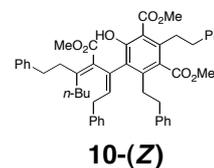


10-(E)



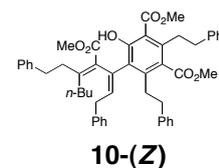
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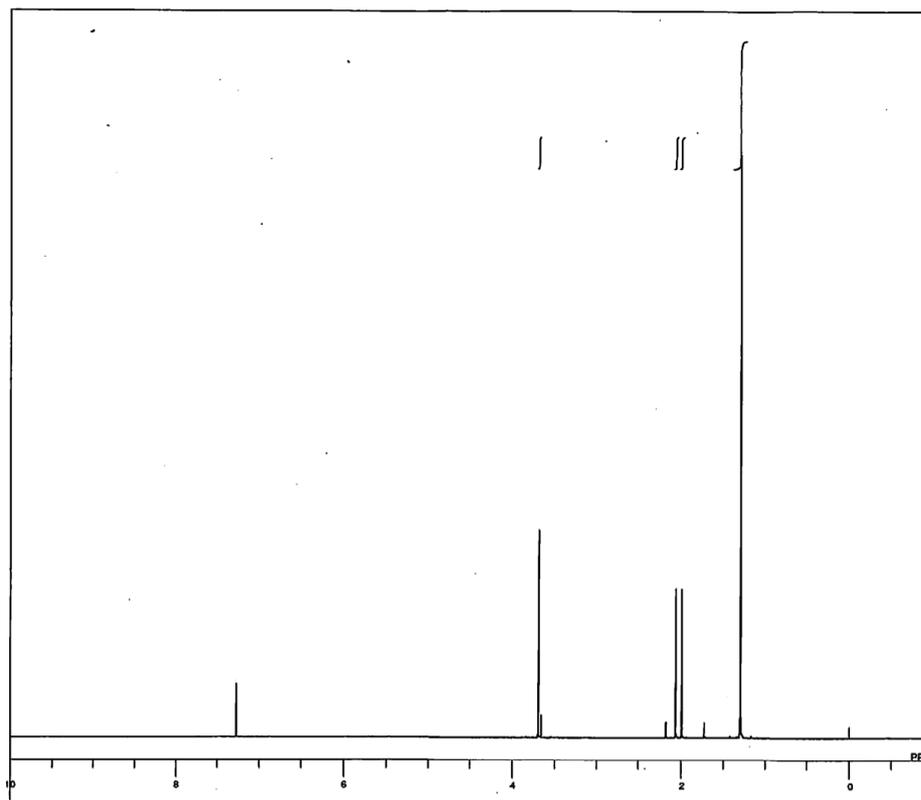
MENUF 1H
OBNUC 1H
OFR 499.10 MHz
OBSET 0.00 KHz
OBFIN 128250.00 Hz
PW1 5.50 usec
DEADT 72.30 usec
PREDL 0.01000 msec
IWT 1000.0000 sec
POINT 65536
SPO 65536
TIMES 8
DUMMY 0
FREQU 8000.00 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 8.1620 sec
PD 1.0000 sec
ADBIT 16
RGAIN 24
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD non
EXPCM single pulse nondecoupling & nonpresaturation
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE yy-18-136-rev-fr-1-1H.als
SF t5sat
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 22
LKPHS 346
LKSIG 962
CSPED 13 Hz
FILDC
FILDF
SLVNT CDCl3
DATM Wed Aug 19 20:02:44 2009
    
```



```

MENUF bcm
OBNUC 13C
OFR 125.40 MHz
OBSET 0.00 KHz
OBFIN 143041.00 Hz
PW1 5.50 usec
DEADT 10.00 usec
PREDL 10.00000 msec
IWT 10.0000 sec
POINT 32768
SPO 32768
TIMES 5000
DUMMY 1
FREQU 33898.30 Hz
FLT 16950 Hz
DELAY 11.80 usec
ACQTM 0.9667 sec
PD 2.0333 sec
ADBIT 16
RGAIN 30
BF 1.20 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD bcm
EXPCM single pulse with blevel decoupling
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 65 usec
IRATN 511
DFILE yy-18-136-13C.als
SF t5sat
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 22
LKPHS 353
LKSIG 1093
CSPED 14 Hz
FILDC
FILDF
SLVNT CDCl3
DATM Thu May 21 08:03:06 2009
    
```



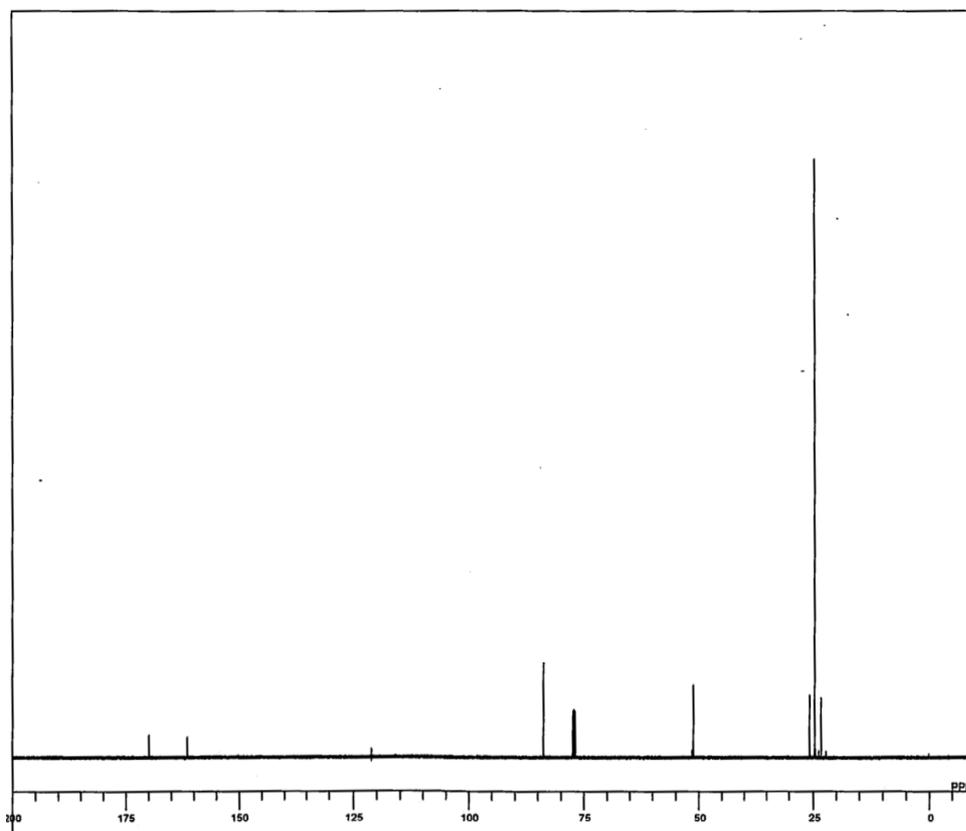


```

MENUF 1H
OBNUC 1H
OFR 499.10 MHz
OBSET 0.00 KHz
OSFIN 12685.00 Hz
PWI 5.50 usec
DEADT 72.30 usec
PREDL 0.01000 msec
IWT 1000.0000 sec
POINT 65536
SPO 65536
TIMES 8
DUMMY 0
FREQU 8000.00 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 8.1920 sec
PD 1.0200 sec
ADBIT 16
RGAIN 17
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD non
EXPCM single pulse nondecoupling & nonpresaturation
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 126250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE yy-19-70-1H.als
SF thset
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 22
LKPHS 346
LKSIG 879
CSPED 12 Hz
FILDC
FILDF
SLVNT CDCL3
DATIM Sat Aug 08 15:34:11 2009
    
```



S1



```

MENUF 13C
OBNUC 13C
OFR 125.40 MHz
OBSET 0.00 KHz
OSFIN 143041.00 Hz
PWI 8.25 usec
DEADT 10.00 usec
PREDL 0.01000 msec
IWT 1000.0000 sec
POINT 65536
SPO 65536
TIMES 360
DUMMY 2
FREQU 33898.30 Hz
FLT 16850 Hz
DELAY 11.80 usec
ACQTM 1.9333 sec
PD 1.0670 sec
ADBIT 16
RGAIN 30
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD bcm
EXPCM single pulse with bitelev decoupling
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 126250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE yy-19-70-13C.als
SF thset
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 22
LKPHS 346
LKSIG 879
CSPED 13 Hz
FILDC
FILDF
SLVNT CDCL3
DATIM Sat Aug 08 15:48:03 2009
    
```



S1

