Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Supporting Information

Direct Electrooxidation of Alkynes to Benzoin Bis-ethers

Zengzhuan Yang,[†, a] Fangling Lu,[†, b] Haiyan Li,[c] Yuying Zhang,[c] Wen Lin,[c] Peng Guo,[c] Juelin Wan,[c] Renyi Shi,*[, d] Tao Wang,*[, a, c] and Aiwen Lei*[, a, d]

[a]National Research Center for Carbohydrate Synthesis, Jiangxi Normal University, Nanchang 330022, Jiangxi, P. R. China.

[b]College of Chemistry and Chemical Engineering, Shaanxi Normal University, Xian, 710119, P.R.China.

[b]College of Chemistry and Chemical Engineering, Jiangxi Normal University, Nanchang 330022, P. R. China

[d]College of Chemistry and Molecular Sciences, the Institute for Advanced Studies (IAS), Wuhan University, Wuhan, Hubei 430072, P. R. China.

Table of Contents

General information

Unless otherwise noted, materials were obtained from commercial suppliers and used without further purification. The instrument for electrolysis is dual display potentiostat (DJS-292B) (made in China). The anodic electrode was graphite rod (ϕ 6 mm) and cathodic electrode was platinum plate (15 mm×15 mm×0.3 mm). Thin layer chromatography (TLC) employed glass 0.25 mm silica gel plates. Flash chromatography columns were packed with 300-400 mesh silica gel in petroleum (boiling point is between 60-90 ^{o}C). Gradient flash chromatography was conducted eluting with a continuous gradient from petroleum to the indicated solvent, and they are listed as volume/volume ratios. NMR spectra were recorded on a Bruker spectrometer at 400 MHz (^{1}H NMR), 100 MHz (^{13}C NMR), 376 MHz (^{19}F NMR). All chemical shifts were reported relative to tetramethylsilane (0 ppm for ^{1}H), $CDCl_3$ (77.0 ppm for ^{13}C), DMSO-d_6 (39.6 ppm for ^{13}C), respectively. And all ^{1}H, ^{13}C and ^{19}F NMR data spectra were reported in delta (δ) units, parts per million (ppm) downfield from the internal standard. Coupling constants are reported in Hertz (Hz). GC-MS spectra were recorded on a Shimadzu GC-MS QP2010 Ultra.

Experimental procedure

1. General procedure for the synthesis of 2,2-dimethoxy-1,2-diphenylethanone:

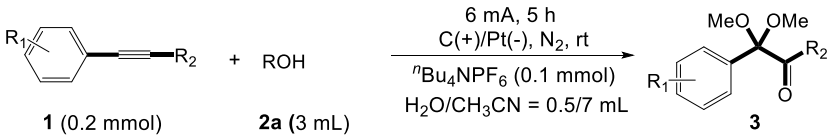
In an oven-dried undivided three-necked bottle (25 mL) equipped with a stir bar, **1a** (0.2 mmol, 35.6 mg), $^{n}Bu_4NPF_6$ (0.1 mmol, 38.7 mg) were added. The bottle was equipped with graphite rod (ϕ 6 mm, about 16 mm immersion depth in solution) as the anode and platinum plate (15 mm×15 mm×0.3 mm) as the cathode. Under the protection of N_2, MeOH (3 mL), H_2O (0.5 mL) and dry MeCN (7 mL) were injected respectively into the tubes via syringes. The reaction mixture was stirred and electrolyzed at a constant current of 6 mA at room temperature for 5 h. When the reaction was finished, concentrated under vacuum to give the crude product, which was purified by silica gel column with (petroleumether : ethyl acetate = 70:1) as the eluent to give the pure product.

2. Gram-scale synthesis of 2,2-dimethoxy-1,2-diphenylethanone:

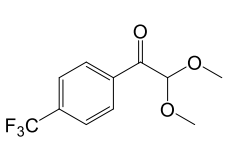
In an oven-dried undivided three-necked bottle (100 mL) equipped with a stir bar, **1a** (5 mmol, 891.1mg), $^{n}Bu_4NPF_6$ (0.5 mmol, 193.5 mg) were added. The bottle was equipped with graphite rod (ϕ 6 mm, about 16 mm immersion depth in solution) as the anode and platinum plate (15 mm×15 mm×0.3 mm) as the cathode. Under the protection of N_2, MeOH (15 mL), H_2O (2.5 mL) and dry MeCN (60 mL) were injected respectively into the tubes via syringes. The reaction mixture was stirred and electrolyzed at a constant current of 25 mA at room temperature for 37 h. When the reaction was finished, the reaction mixture was extracted with ethyl acetate, dried over Na_2SO_4 and concentrated in vacuo. which was purified by silica gel column with (petroleumether : ethyl acetate = 70:1) as the eluent to give the pure product (yield: 40%, 0.51g).

3. Unsuccessful substrates:

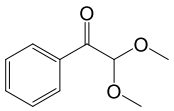
3.1 Direct electrooxidation of various alkynes with methyl alcohol to benzoin bis-ethers[a]:



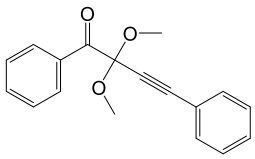
1 (0.2 mmol) **2a** (3 mL) **3**



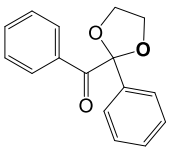
n.d.



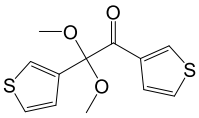
n.d.



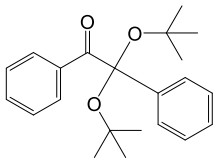
n.d.



n.d.[b]



n.d.

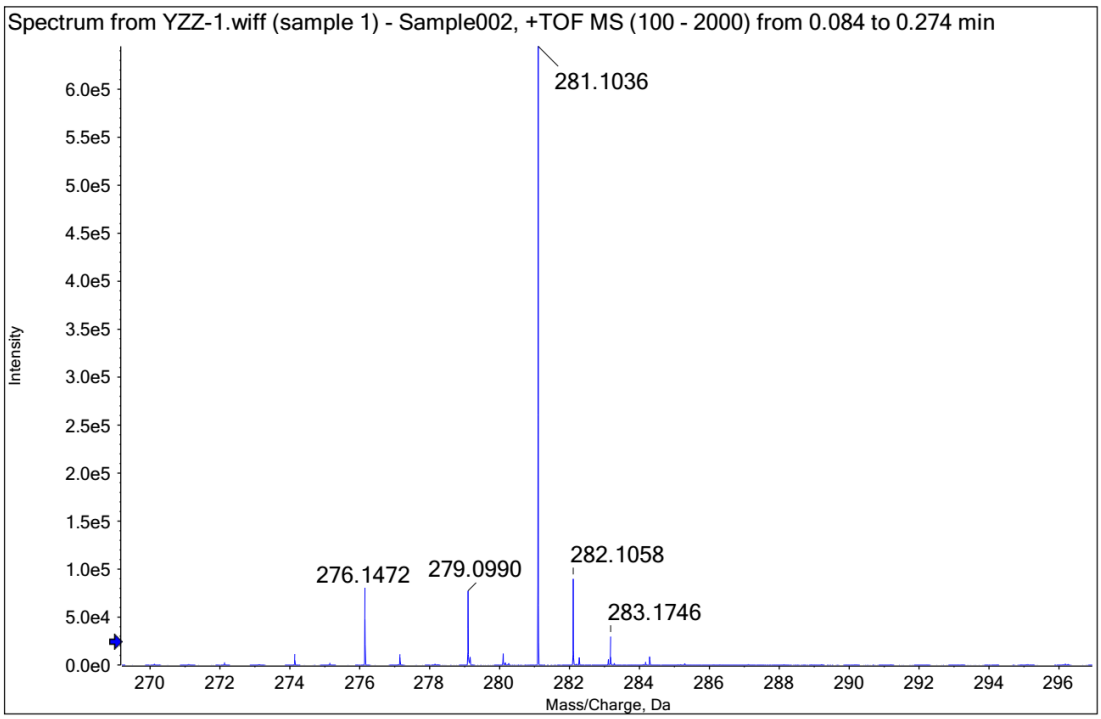


n.d.

[a] The reaction was carried out with 1 (0.2 mmol), 2a (3 mL), CH_3CN (7 mL), H_2O (0.5 mL), $^nBu_4NPF_6$ (0.1 mmol), N_2, 6 mA, 5 h, C(+)/Pt(-). [b] H_2O (20 eq).

4. Procedures for isotope labeling experiments ($H_2{}^{18}O$):

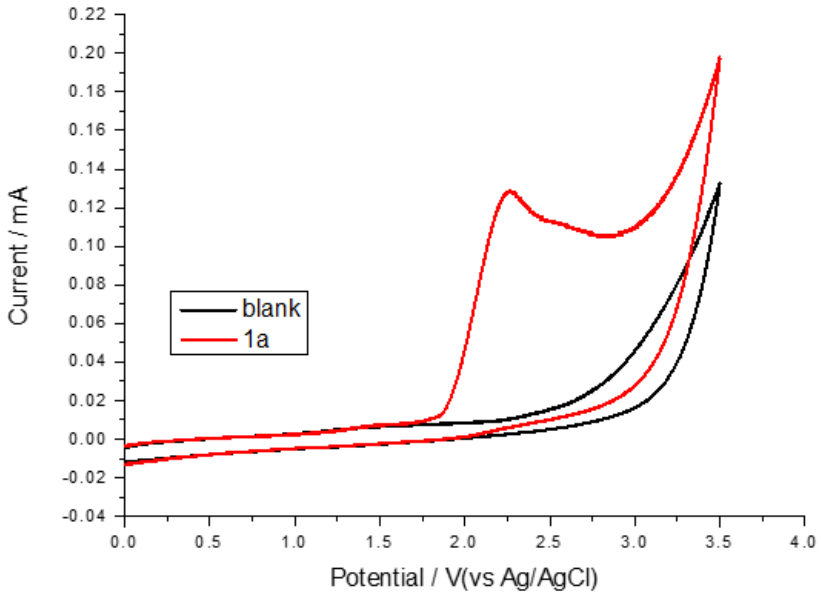
In an oven-dried undivided beaker (25 mL) equipped with a stir bar, **1a** (0.2 mmol, 35.6 mg), $^nBu_4NPF_6$ (0.1 mmol, 38.7 mg) were added. The bottle was equipped with graphite rod (ϕ 6 mm, about 16 mm immersion depth in solution) as the anode and platinum plate (15 mm×15 mm×0.3 mm) as the cathode. Under the protection of N_2, MeOH (3 mL), $H_2{}^{18}O$ (0.5 mL) and dry MeCN (7 mL) were injected respectively into the tubes via syringes. The reaction mixture was stirred and electrolyzed at a constant current of 6 mA at room temperature for 5 h. When the reaction was finished, concentrated under vacuum to give the crude product, the pure product was obtained by flash column chromatography on silica gel using petroleum ether and ethyl acetate as the eluent. The pure product was detected by HRMS, and the target product marked by isotope was detected. **HRMS (ESI)** Calcd. For $C_{16}H_{16}O_2{}^{18}O$**[M+Na$^+$]**: 281.1040. Found: m/z 281.1036. (See Supplementary Figure 1)



Supplementary Figure 1. Isotope labeling experiment of $H_2{}^{18}O$.

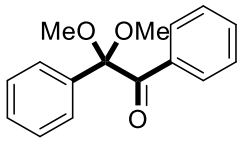
5. Procedure for cyclic voltammetry (CV):

Cyclic voltammetry was performed in a three-electrode cell connected to a Schlenk line under nitrogen at room temperature. The working electrode was a steady glassy carbon disk electrode while the counter electrode was a platinum wire. The reference was an Ag/AgCl electrode submerged in saturated aqueous KCl solution. 10.0 mL of acetonitrile containing $^{n}Bu_4NPF_6$ (0.1 mmol) was poured into the electrochemical cell in cyclic voltammetry experiments. The scan rate was 0.1 V/s, ranging from 0 V to 4.0 V.

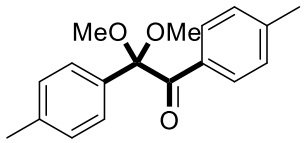


Supplementary Figure 2. Cyclic voltammogram: 1a, 0.1 mmol.

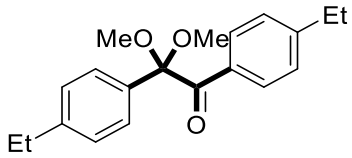
Detail descriptions for products



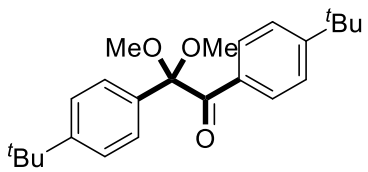
2, 2-dimethoxy-1, 2-diphenylethan-1-one (3a)[2]. Colorless oil (85%, 43.5 mg). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.05 (d, *J* = 7.6 Hz, 2H), 7.62 (d, *J* = 7.1 Hz, 2H), 7.42 (t, *J* = 7.3 Hz, 1H), 7.37 – 7.27 (m, 5H), 3.22 (s, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 195.16, 136.88, 134.32, 132.83, 129.98, 128.90, 128.51, 128.11, 126.94, 103.63, 50.05.



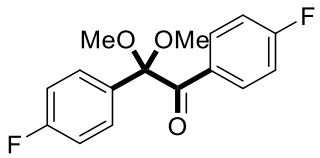
2, 2-dimethoxy-1, 2-di-p-tolylethan-1-one (3b)[2]. Colorless oil (40%, 22.7 mg). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.98 (d, *J* = 8.0 Hz, 2H), 7.48 (d, *J* = 7.8 Hz, 2H), 7.14 (d, *J* = 7.8 Hz, 2H), 7.09 (d, *J* = 8.0 Hz, 2H), 3.20 (s, 6H), 2.30 (s, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 194.73, 143.59, 138.65, 134.07, 131.71, 130.18, 129.19, 128.82, 126.82, 103.63, 49.95, 21.56, 21.15.



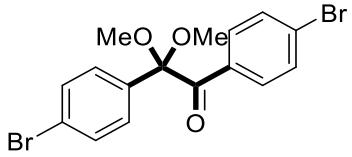
1, 2-bis(4-ethylphenyl)-2, 2-dimethoxyethan-1-one (3c)[3]. Colorless oil (46%, 28.7 mg). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.01 (d, *J* = 8.3 Hz, 2H), 7.52 (d, *J* = 8.2 Hz, 2H), 7.17 (d, *J* = 7.9 Hz, 2H), 7.12 (d, *J* = 8.1 Hz, 2H), 3.20 (s, 6H), 2.61 (q, *J* = 7.6 Hz, 4H), 1.22 – 1.17 (m, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 194.79, 149.65, 144.86, 134.25, 131.95, 130.30, 127.94, 127.60, 126.88, 103.73, 49.96, 28.84, 28.48, 15.13, 14.88.



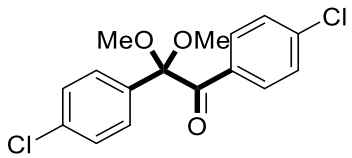
1, 2-bis(4-(tert-butyl)phenyl)-2, 2-dimethoxyethan-1-one (3d)[2]. Colorless oil (42%, 30.9 mg). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.03 (d, *J* = 6.6 Hz, 2H), 7.54 (d, *J* = 7.2 Hz, 2H), 7.34 (t, *J* = 10.5 Hz, 4H), 3.19 (s, 6H), 1.28 (s, 18H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 194.87, 156.38, 151.75, 133.87, 131.73, 130.09, 126.65, 125.37, 125.04, 103.85, 49.99, 34.99, 34.57, 31.26, 30.99.



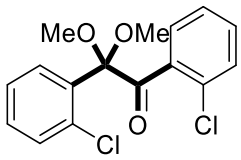
1, 2-bis(4-fluorophenyl)-2, 2-dimethoxyethan-1-one (3e). Colorless oil (55%, 32.1 mg). **^{1}H NMR** (400 MHz, DMSO-d_6) δ 8.11 – 8.07 (m, 2H), 7.57 – 7.54 (m, 2H), 7.29 – 7.23 (m, 4H), 3.14 (s, 6H). **^{13}C NMR** (100 MHz, DMSO-d_6) δ 193.29, 165.23 (d, J = 256.30 Hz), 162.75 (d, J = 249.20 Hz), 133.21 (d, J = 3.00 Hz), 130.97 (d, J = 3.40 Hz), 133.05 (d, J = 9.50 Hz), 129.41 (d, J = 9.00 Hz), 116.18 (d, J = 21.80 Hz), 116.04 (d, J = 21.80 Hz), 103.62, 50.19. **^{19}F NMR** (376 MHz, DMSO-d_6) δ -105.11, -112.57. **HRMS (ESI)** Calcd. for $C_{16}H_{14}F_2O_3$ **[M+H$^+$]**: 293.0989. Found: m/z 293.0984.



1, 2-bis(4-bromophenyl)-2, 2-dimethoxyethan-1-one (3f). Colorless oil (55%, 45.43 mg). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.93 – 7.91 (m, 2H), 7.49 – 7.45 (m, 6H), 3.21 (s, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 193.77, 135.74, 132.64, 131.93, 131.65, 131.51, 129.94, 128.62, 123.57, 103.25, 50.19. **HRMS (ESI)** Calcd. for $C_{16}H_{14}Br_2O_3$ **[M+Na$^+$]**: 436.9182. Found: m/z 436.9180.

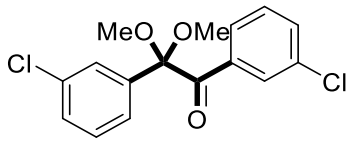


1, 2-bis(4-chlorophenyl)-2, 2-dimethoxyethan-1-one (3g)[4]. Colorless oil (65%, 42.1 mg). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.00 (d, J = 8.6 Hz, 2H), 7.53 (d, J = 8.2 Hz, 2H), 7.34 – 7.28 (m, 4H), 3.22 (s, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 193.60, 139.62, 135.23, 132.29, 131.48, 131.41, 128.93, 128.60, 128.32, 103.22, 50.15.

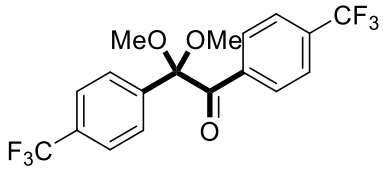


1, 2-bis(2-chlorophenyl)-2, 2-dimethoxyethan-1-one (3h). Colorless oil (40%, 25.9 mg). **^{1}H NMR** (400 MHz, DMSO-d_6) δ 7.95 (d, J = 7.5 Hz, 1H), 7.74 – 7.72 (m, 1H), 7.49 – 7.47 (m, 1H), 7.42 – 7.36 (m, 4H), 7.25 – 7.20 (m, 1H), 3.17 (s, 6H). **^{13}C NMR** (100 MHz, DMSO-d_6) δ 192.06, 134.62, 132.87, 132.32, 131.92, 131.56, 131.30, 130.81, 130.65, 130.45, 127.49, 126.70, 126.69, 101.09, 50.21.

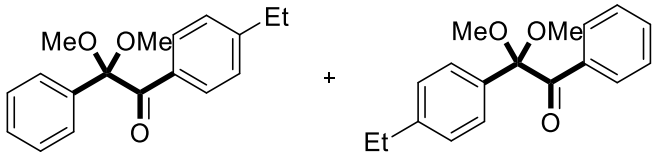
HRMS (ESI) Calcd. for $C_{16}H_{14}Cl_2O_3$ **[M+H$^+$]**: 325.0398. Found: m/z 325.0395.



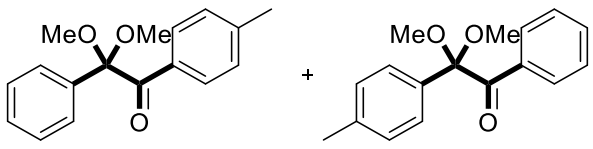
1,2-bis(3-chlorophenyl)-2,2-dimethoxyethan-1-one (3i). Colorless oil (44%, 28.5 mg). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.97 (t, *J* = 1.9 Hz, 1H), 7.86 (d, *J* = 7.9 Hz, 1H), 7.58 (s, 1H), 7.38 – 7.35 (m, 2H), 7.24 – 7.22 (m, 2H), 7.20 (d, *J* = 2.4 Hz, 1H), 3.16 (s, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 193.47, 138.65, 135.49, 134.90, 134.55, 133.11, 130.01, 129.92, 129.63, 129.43, 128.04, 127.09, 125.10, 103.11, 50.28. **HRMS (ESI)** Calcd. for $C_{16}H_{14}Cl_2O_3$ **[M+H$^+$]**: 325.0398. Found: m/z 325.0393.



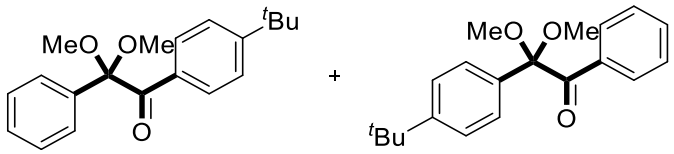
2, 2-dimethoxy-1, 2-bis(4-(trifluoromethyl)phenyl)ethan-1-one (3j). Colorless oil (42%, 32.9 mg). **^{1}H NMR** (400 MHz, DMSO-d_6) δ 8.16 (d, *J* = 8.2 Hz, 2H), 7.84 – 7.80 (m, 4H), 7.77 (d, *J* = 8.5 Hz, 2H), 3.21 (s, 6H). **^{13}C NMR** (100 MHz, DMSO-d_6) δ 194.33, 141.02, 137.60, 133.1 (q, *J* = 32.00 Hz), 130.60, 130.10 (q, *J* = 31.90 Hz), 128.90 (q, *J* = 186.30 Hz), 128.18, 126.30 (q, *J* = 7.30 Hz), 126.10 (q, *J* = 7.20 Hz), 124.31 (q, *J* = 270.70 Hz), 103.70, 50.50. **^{19}F NMR** (376 MHz, DMSO-d_6) δ -61.39, -61.96. **HRMS (ESI)** Calcd. for $C_{18}H_{14}F_6O_3$ **[M+Na$^+$]**: 415.0739. Found: m/z 415.0745.



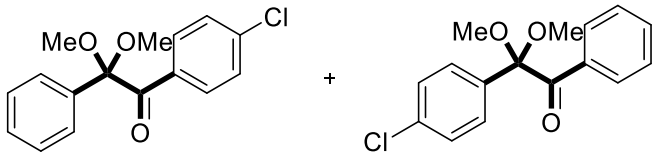
1-(4-ethylphenyl)-2, 2-dimethoxy-2-phenylethan-1-one (3k). Colorless oil (30%, 17.0 mg). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.07 (d, *J* = 8.0 Hz, 1H), 8.02 – 8.00 (m, 3H), 7.62 (d, *J* = 7.3 Hz, 3H), 7.52 (d, *J* = 7.2 Hz, 1H), 7.43 (t, *J* = 7.4 Hz, 1H), 7.37 – 7.29 (m, 5H), 7.18 (d, *J* = 7.6 Hz, 1H), 7.12 (d, *J* = 7.6 Hz, 3H), 3.21 (s, 12H), 2.62 – 2.60 (m, 4H), 1.23 – 1.17 (m, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 195.29, 194.64, 149.77, 144.98, 137.09, 132.76, 131.82, 130.28, 130.03, 128.81, 128.47, 128.09, 128.00, 127.64, 126.91, 103.56, 50.02, 28.85, 28.49, 15.16, 14.90. **HRMS (ESI)** Calcd. for $C_{18}H_{20}O_3$ **[M+H$^+$]**: 285.1491. Found: m/z 285.1485.



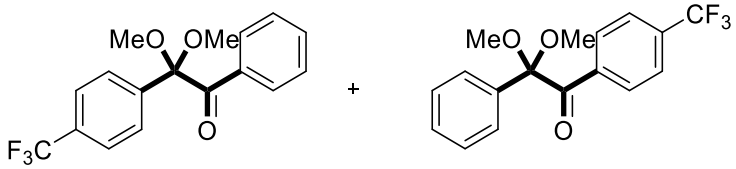
2, 2-dimethoxy-2-phenyl-1-(p-tolyl)ethan-1-one (3l)[2]. Colorless oil (35%, 18.9 mg). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.07 – 7.97 (m, 4H), 7.61 (d, *J* = 7.5 Hz, 2H), 7.49 (d, *J* = 8.0 Hz, 2H), 7.40 – 7.27 (m, 6H), 7.16 – 7.08 (m, 4H), 3.21 (d, *J* = 1.8 Hz, 12H), 2.30 (s, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 195.28, 194.63, 143.69, 138.76, 137.09, 133.86, 132.76, 130.17, 129.99, 129.25, 128.84, 128.46, 128.09, 126.90, 126.86, 103.70, 103.56, 50.01, 49.99, 21.56, 21.16.



1-(4-(tert-butyl)phenyl)-2,2-dimethoxy-2-phenylethan-1-one (3m). Colorless oil (45%, 28.1 mg). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.15 – 7.99 (m, 4H), 7.65 – 7.62 (m, 2H), 7.53 (d, *J* = 8.5 Hz, 2H), 7.46 – 7.40 (m, 1H), 7.37 – 7.28 (m, 9H), 3.21 (d, *J* = 2.2 Hz, 12H), 1.27 (d, *J* = 5.1 Hz, 18H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 195.34, 194.64, 156.52, 151.87, 137.07, 134.35, 133.63, 132.78, 131.46, 130.09, 130.03, 128.82, 128.49, 128.10, 126.94, 126.62, 125.43, 125.12, 103.81, 103.57, 50.04, 35.01, 34.59, 31.26, 30.97.

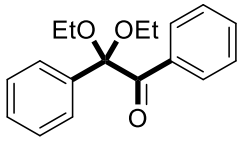


1-(4-chlorophenyl)-2,2-dimethoxy-2-phenylethan-1-one (3n). Colorless oil (36%, 20.9 mg). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.03 (t, *J* = 7.3 Hz, 4H), 7.57 (m, 4H), 7.45 (t, *J* = 7.3 Hz, 2H), 7.36 – 7.28 (m, 8H), 3.22 (s, 12H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 194.77, 193.99, 139.39, 136.57, 135.52, 135.01, 134.01, 133.10, 132.39, 131.50, 129.93, 129.09, 128.83, 128.65, 128.50, 128.41, 128.25, 126.84, 103.50, 103.22, 50.14, 50.11.

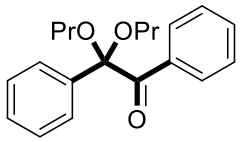


2, 2-dimethoxy-1-phenyl-2-(4-(trifluoromethyl)phenyl)ethan-1-one (3o). Colorless oil (62%, 40.2 mg). **^{1}H NMR** (400 MHz, DMSO-d_6) δ 8.15 (d, *J* = 8.2 Hz, 2H), 8.00 (d, *J* = 7.6 Hz, 2H), 7.81 – 7.77 (m, 6H), 7.60 – 7.51 (m, 3H), 7.46 – 7.35 (m, 5H), 3.17 (s, 7H), 3.16 (s, 5H). **^{13}C NMR** (100 MHz, DMSO-d_6) δ 194.61, 194.46, 141.70, 137.87, 136.45, 134.27, 133.92, 132.85 (q, *J* = 31.80 Hz), 130.56, 130.06, 129.73, 129.31, 129.08, 128.11, 127.13, 126.17 (q, *J* = 7.50 Hz), 125.60 (q, *J* = 7.40 Hz), 124.38

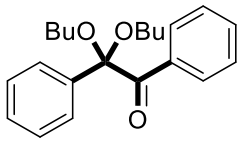
(q, *J* = 270.70 Hz), 124.04 (q, *J* = 271.00 Hz), 104.01, 103.59, 50.35, 50.27. **^{19}F NMR** (376 MHz, DMSO-d_6) δ -62.79, -63.36. **HRMS (ESI)** Calcd. for $C_{17}H_{15}F_3O_3$ **[M+H$^+$]**: 325.1052. Found: m/z 325.1046.



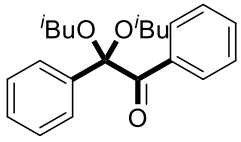
2, 2-diethoxy-1, 2-diphenylethan-1-one (3p)[2]. Colorless oil (62%, 35.2 mg). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.06 (d, *J* = 8.4 Hz, 2H), 7.65 (d, *J* = 7.5 Hz, 2H), 7.41 (t, *J* = 7.1 Hz, 1H), 7.36 – 7.26 (m, 5H), 3.47 – 3.38 (m, 4H), 1.22 – 1.18 (m, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 195.49, 137.88, 134.56, 132.64, 130.04, 128.70, 128.42, 127.98, 126.90, 103.14, 58.18, 15.05.



1, 2-diphenyl-2, 2-dipropoxyethan-1-one (3q)[2]. Colorless oil (60%, 37.4 mg). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.04 (d, *J* = 7.8 Hz, 2H), 7.64 (d, *J* = 7.4 Hz, 2H), 7.41 (t, *J* = 7.2 Hz, 1H), 7.36 – 7.26 (m, 5H), 3.36 – 3.26 (m, 4H), 1.66 – 1.57 (m, 4H), 0.93 – 0.89 (m, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 195.55, 138.04, 134.69, 132.60, 130.05, 128.67, 128.40, 127.96, 126.92, 102.80, 63.99, 22.83, 10.75.

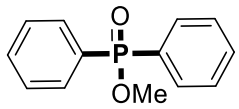


2, 2-dibutoxy-1, 2-diphenylethan-1-one (3r)[2]. Colorless oil (44%, 29.9 mg). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.03 (d, *J* = 7.5 Hz, 2H), 7.64 – 7.62 (m, 2H), 7.43 – 7.39 (m, 1H), 7.36 – 7.28 (m, 5H), 3.34 (q, *J* = 6.8 Hz, 4H), 1.60 – 1.53 (m, 4H), 1.36 (q, *J* = 7.5 Hz, 4H), 0.86 (t, *J* = 7.4 Hz, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 195.56, 138.03, 134.70, 132.56, 130.01, 128.65, 128.37, 127.94, 126.90, 102.86, 62.10, 31.68, 19.41, 13.85.



2, 2-diisobutoxy-1, 2-diphenylethan-1-one (3s)[2]. Colorless oil (53%, 36.0 mg). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.02 (d, *J* = 7.3 Hz, 2H), 7.62 (d, *J* = 7.1 Hz, 2H), 7.39 (t, *J* = 7.2 Hz, 1H), 7.35 – 7.27 (m, 5H), 3.12 (t, *J* = 9.5 Hz, 4H), 1.92 – 1.85 (m, 2H), 0.91 (d, *J* = 6.3 Hz, 12H). **^{13}C NMR** (100 MHz,

$CDCl_3$) δ 195.55, 138.13, 134.82, 132.54, 130.02, 128.63, 128.38, 127.92, 126.92, 102.56, 68.59, 28.47, 19.60, 19.55.



methyl diphenylphosphinate. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.86 – 7.76 (m, 4H), 7.57 – 7.40 (m, 6H), 3.77 (d, *J* = 11.1 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 132.26, 132.24, 131.73, 131.71, 131.63, 130.34, 128.66, 128.53, 51.60, 51.54.

References:

1. K. Sonogashira, *J. Organomet. Chem.*, **2002,** *653,* 46–49.

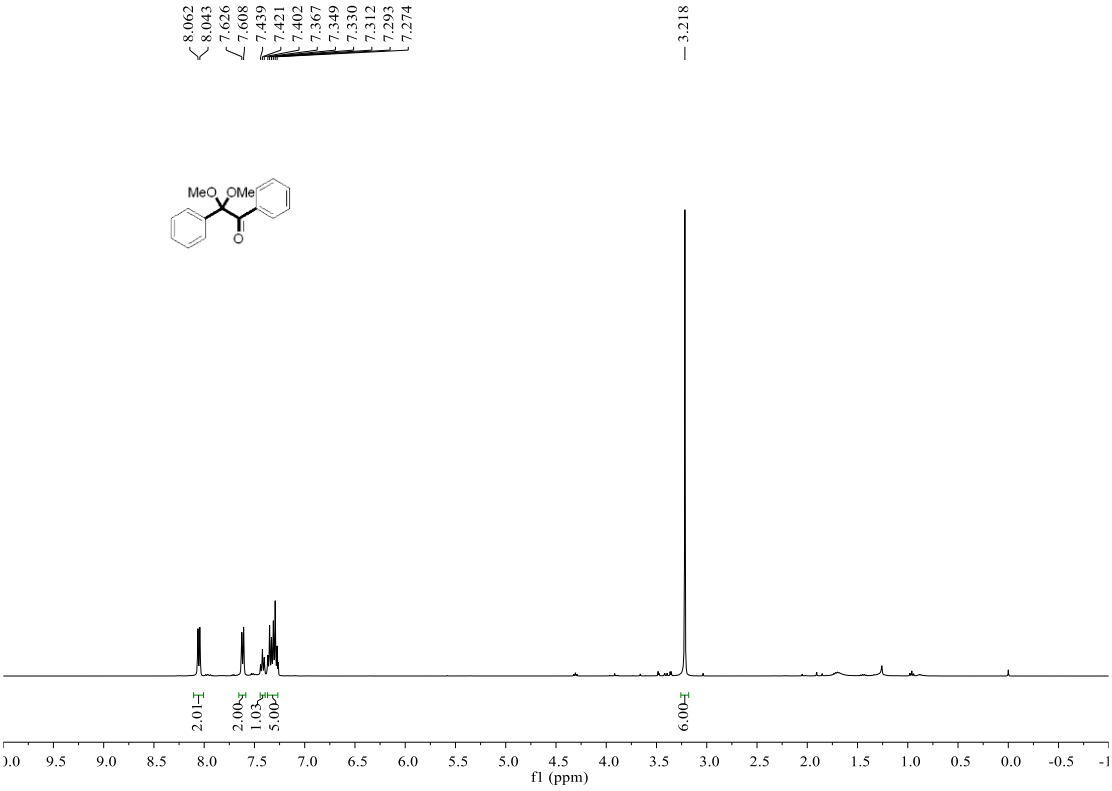
2. Qiang Liu, *Green Chem*., **2018,** *20,* 5479.

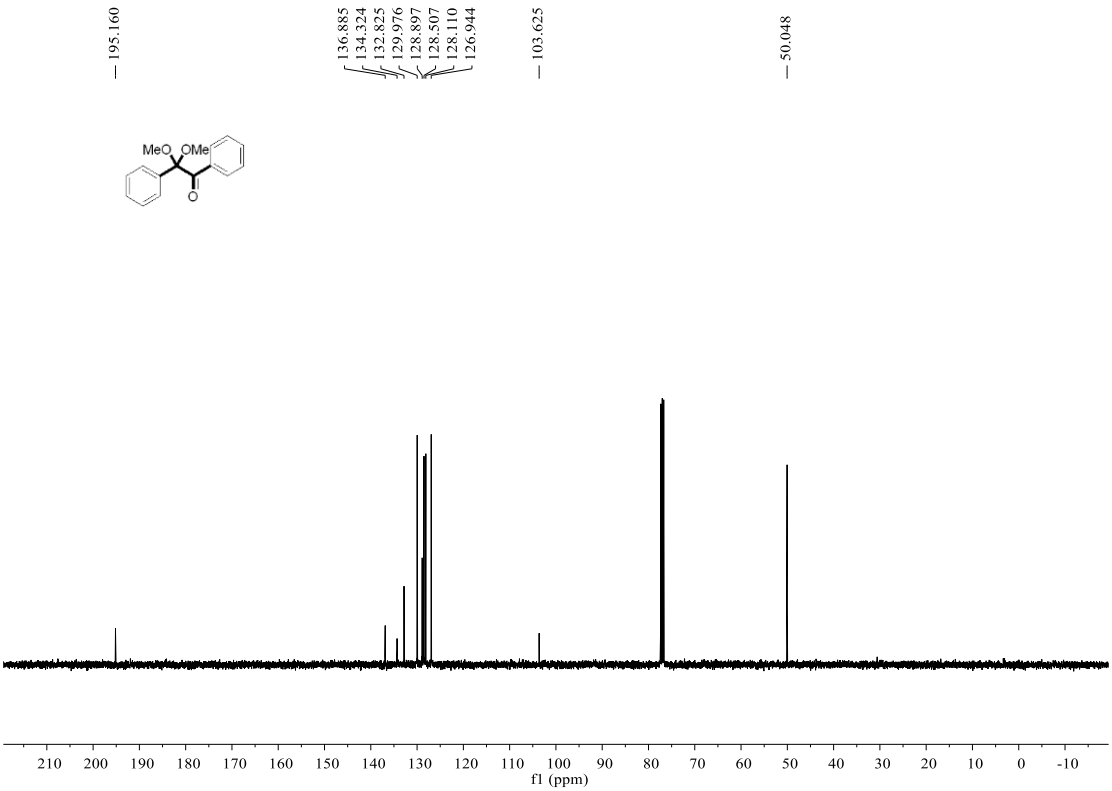
3. Aoki. Yoshio, α,α-Dialkoxyketone as photopolymerization initiator, and polymerizable liquid crystal composition, *JP* 2018052873, 2018.

4. T. Yukio, *Bull. Chem. Soc. Jpn.*, **2002,** *75*, 2059.

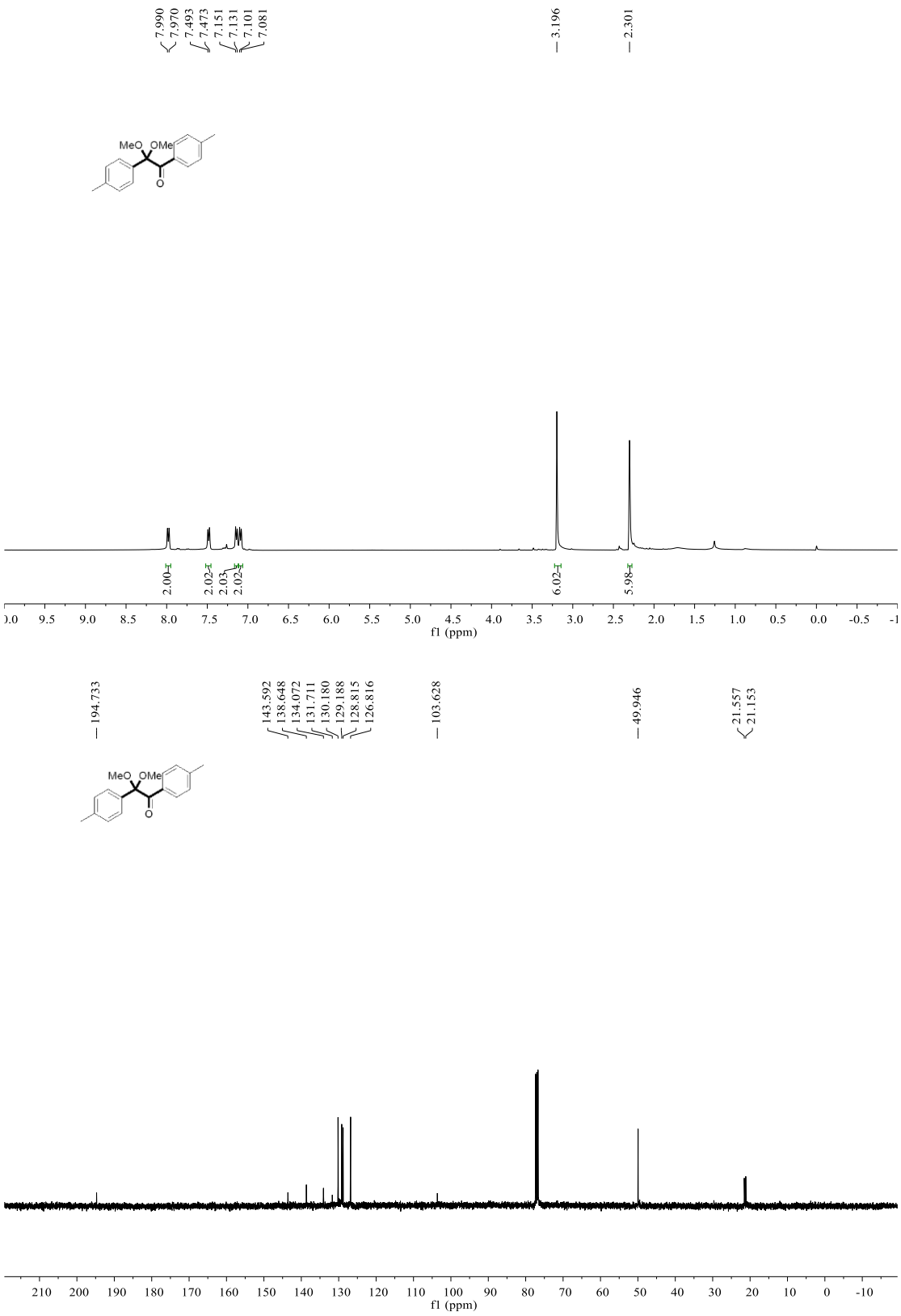
Copies of ^{1}H NMR, ^{13}C NMR and ^{19}F NMR spectra

2, 2-dimethoxy-1, 2-diphenylethan-1-one (3a)

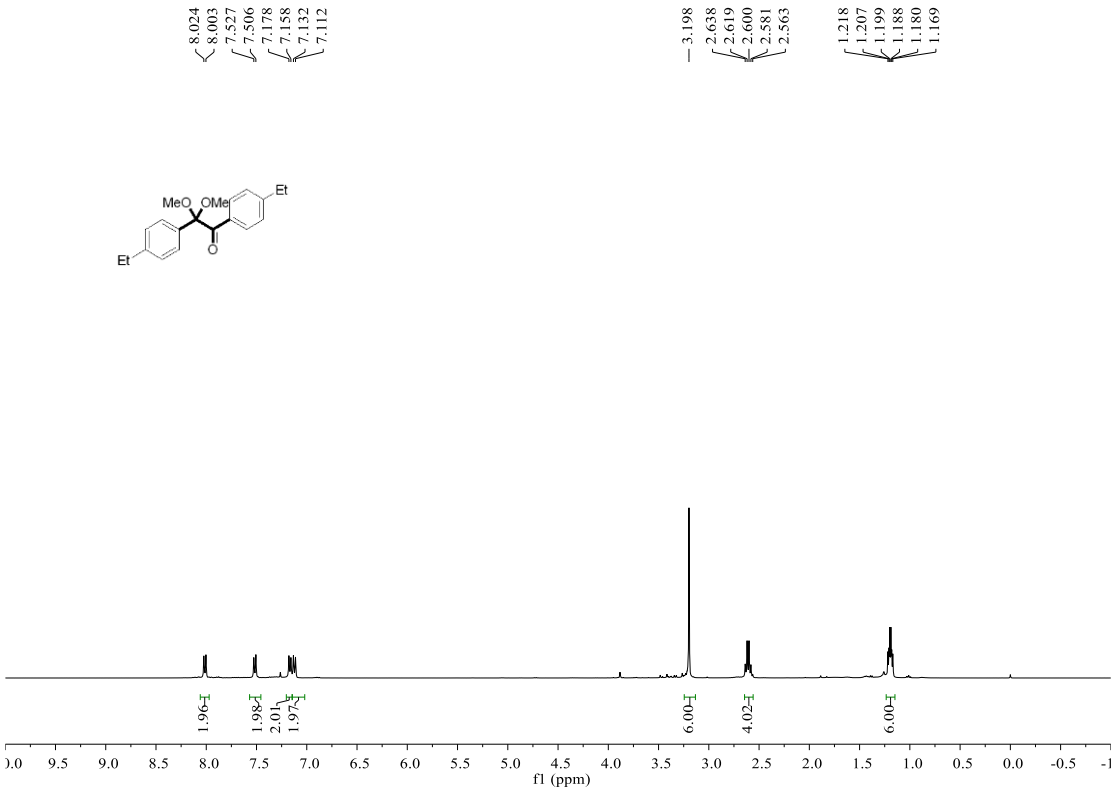


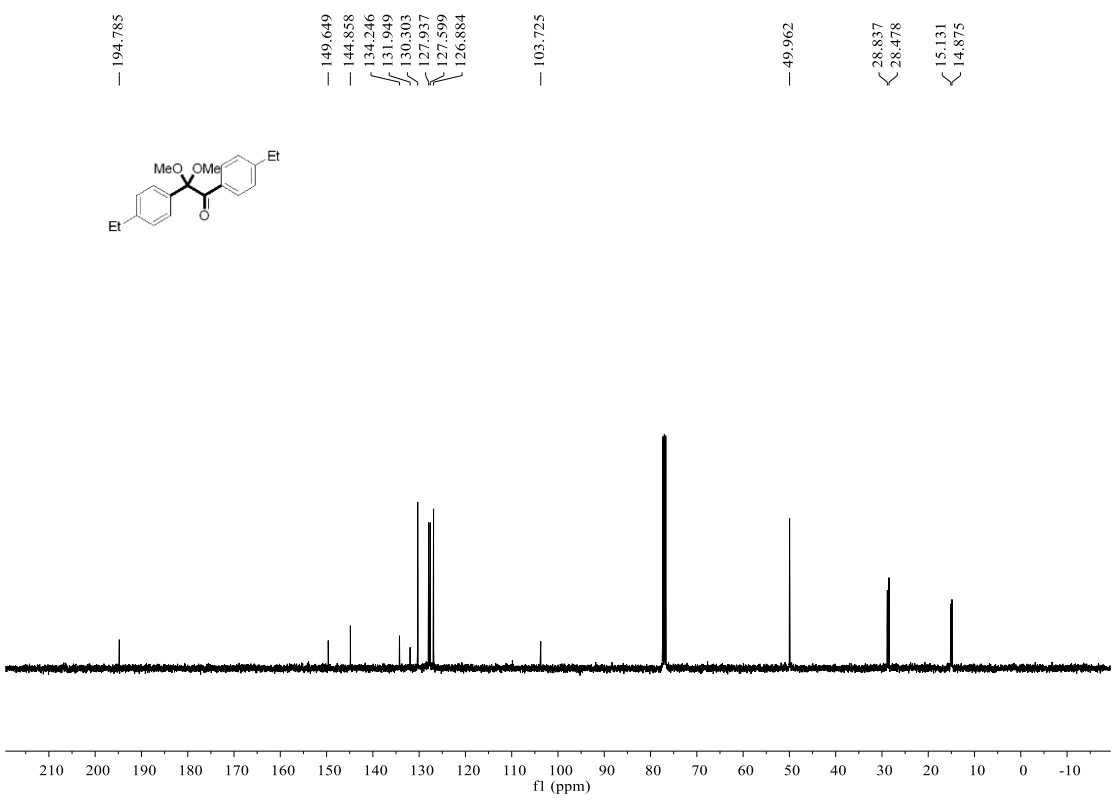


2, 2-dimethoxy-1, 2-di-p-tolylethan-1-one (3b)

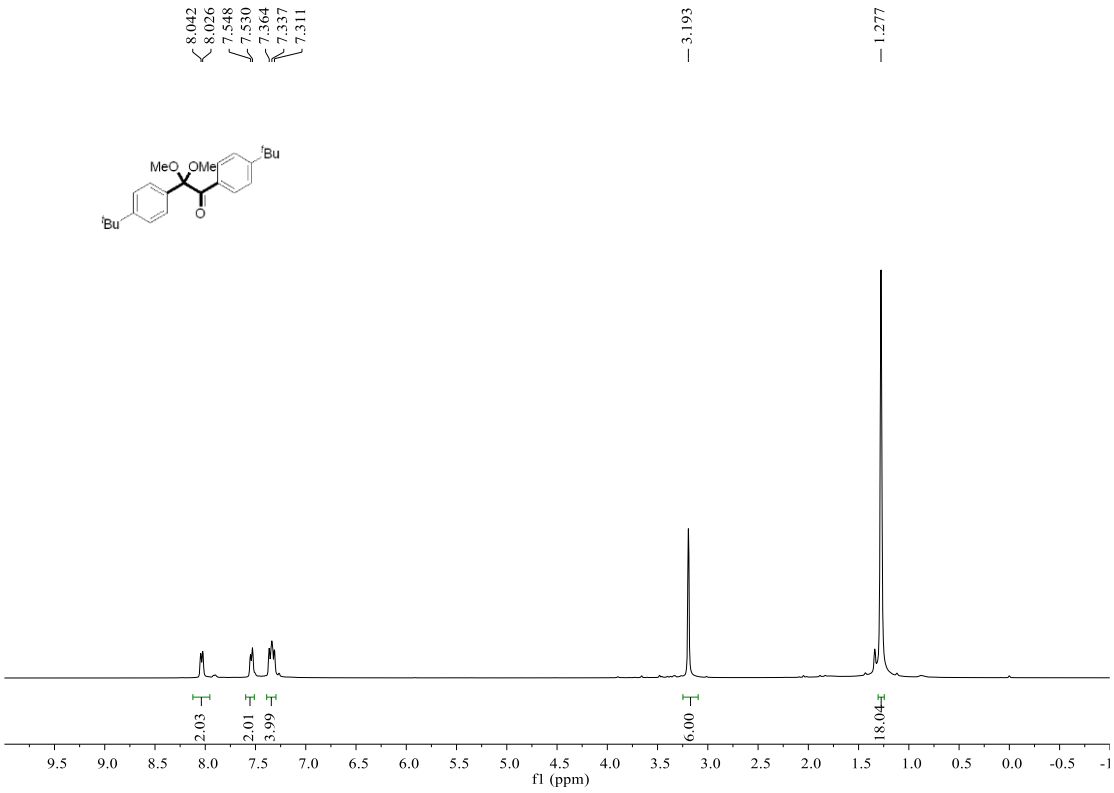


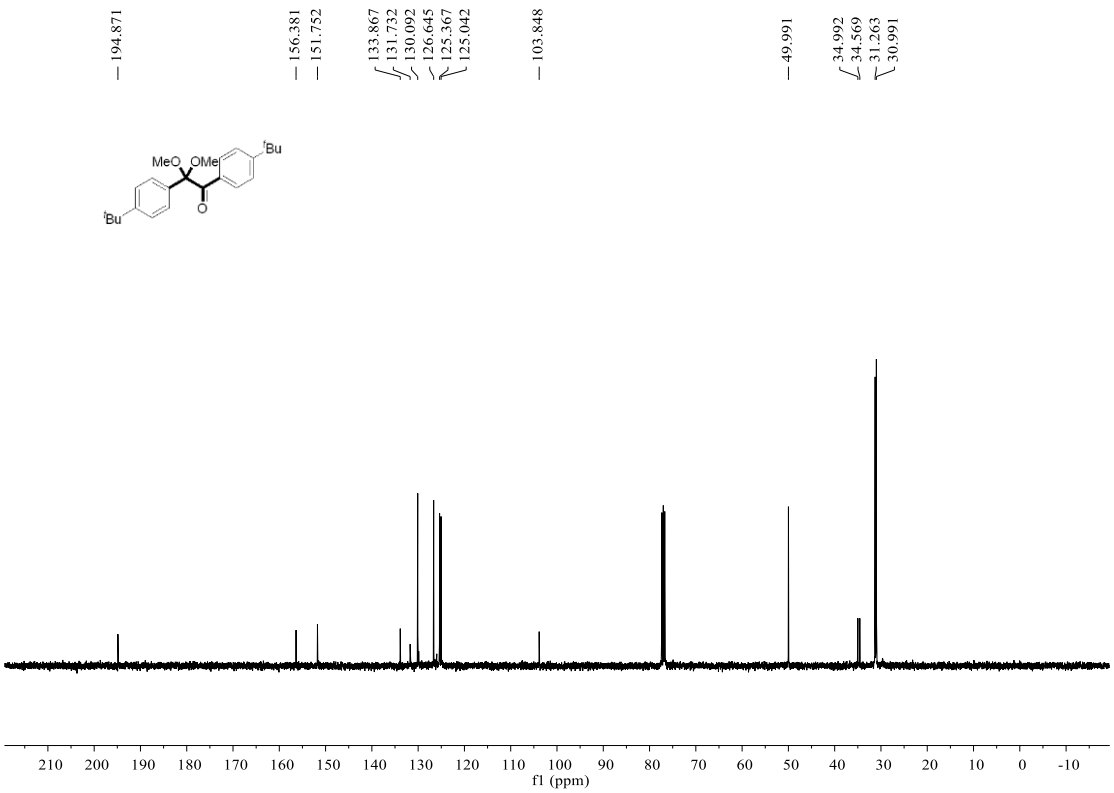
1, 2-bis(4-ethylphenyl)-2, 2-dimethoxyethan-1-one (3c)



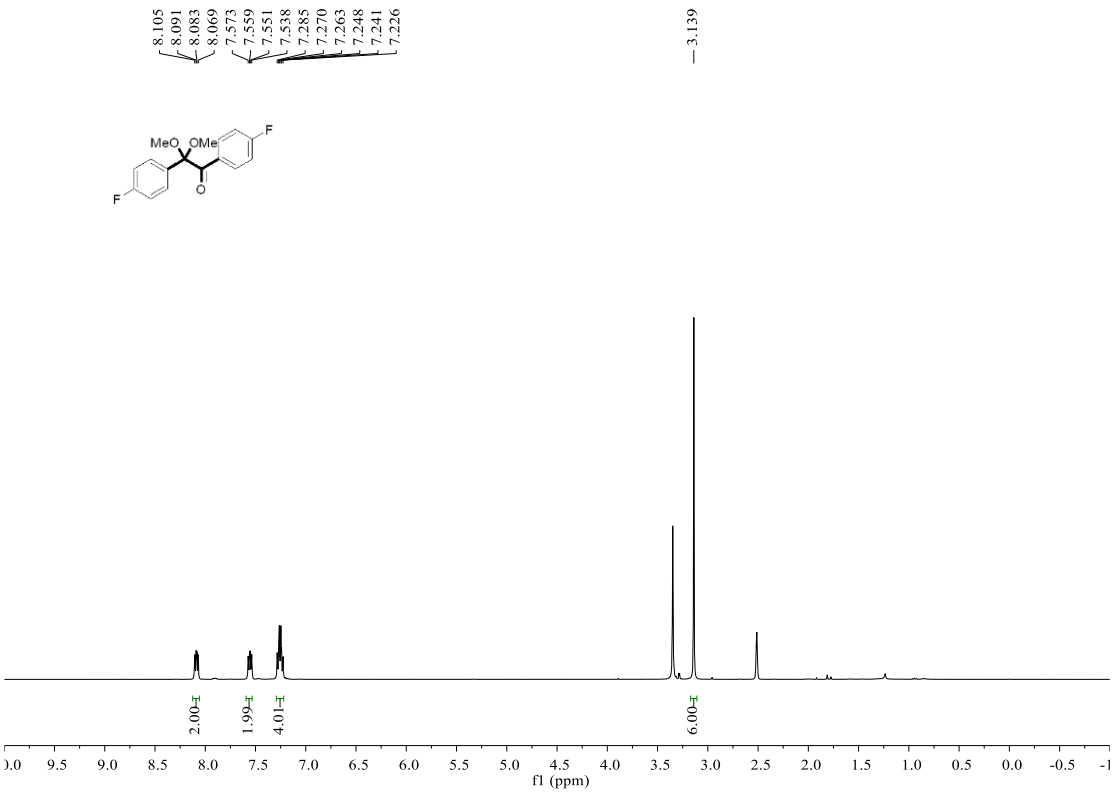


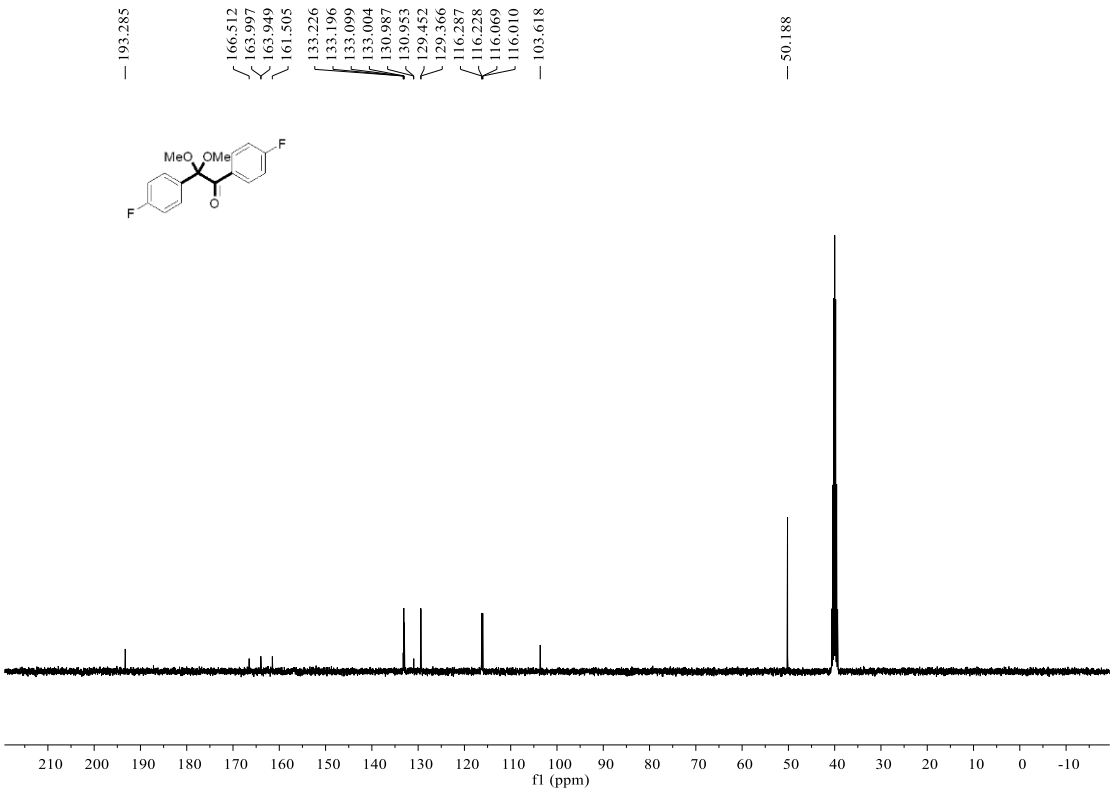
1, 2-bis(4-(tert-butyl)phenyl)-2, 2-dimethoxyethan-1-one (3d)

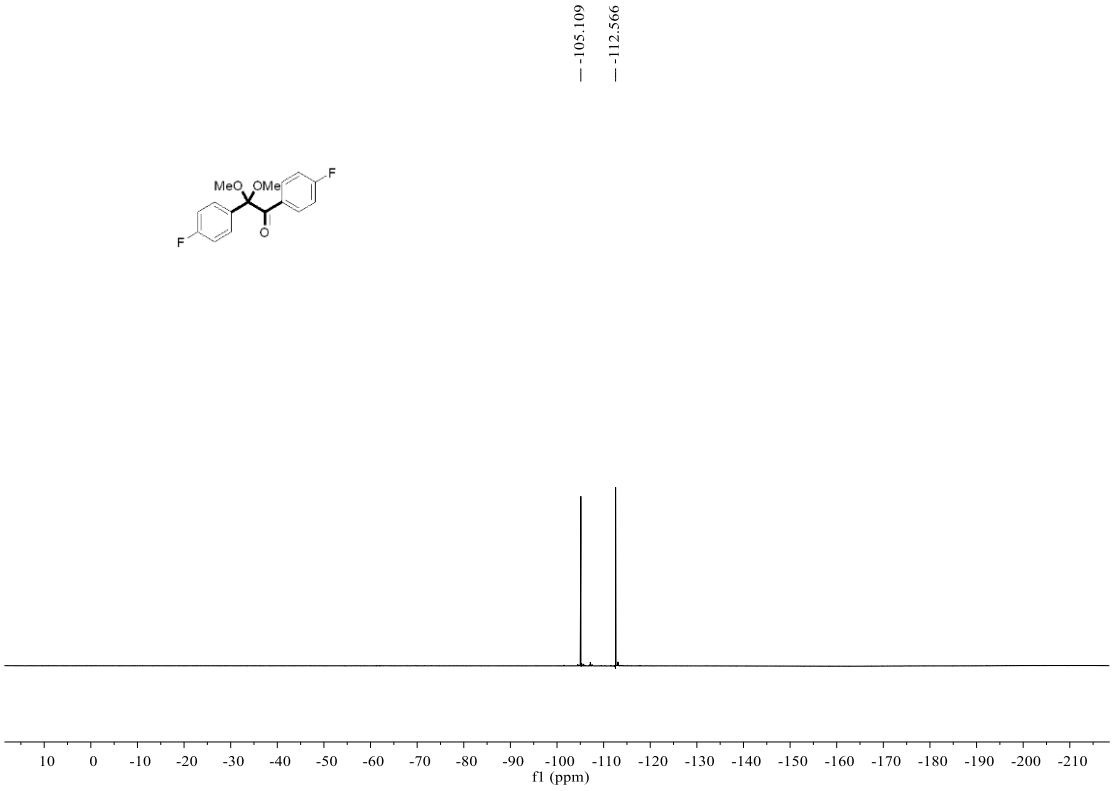




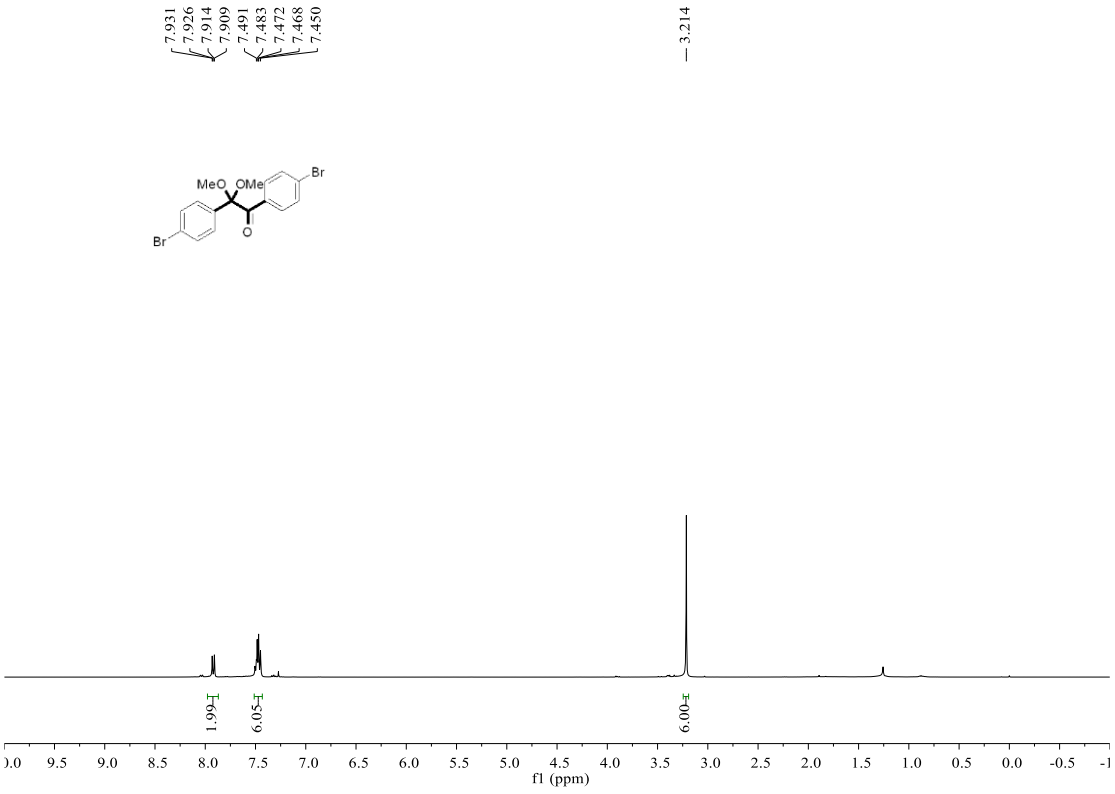
1, 2-bis(4-fluorophenyl)-2, 2-dimethoxyethan-1-one (3e)

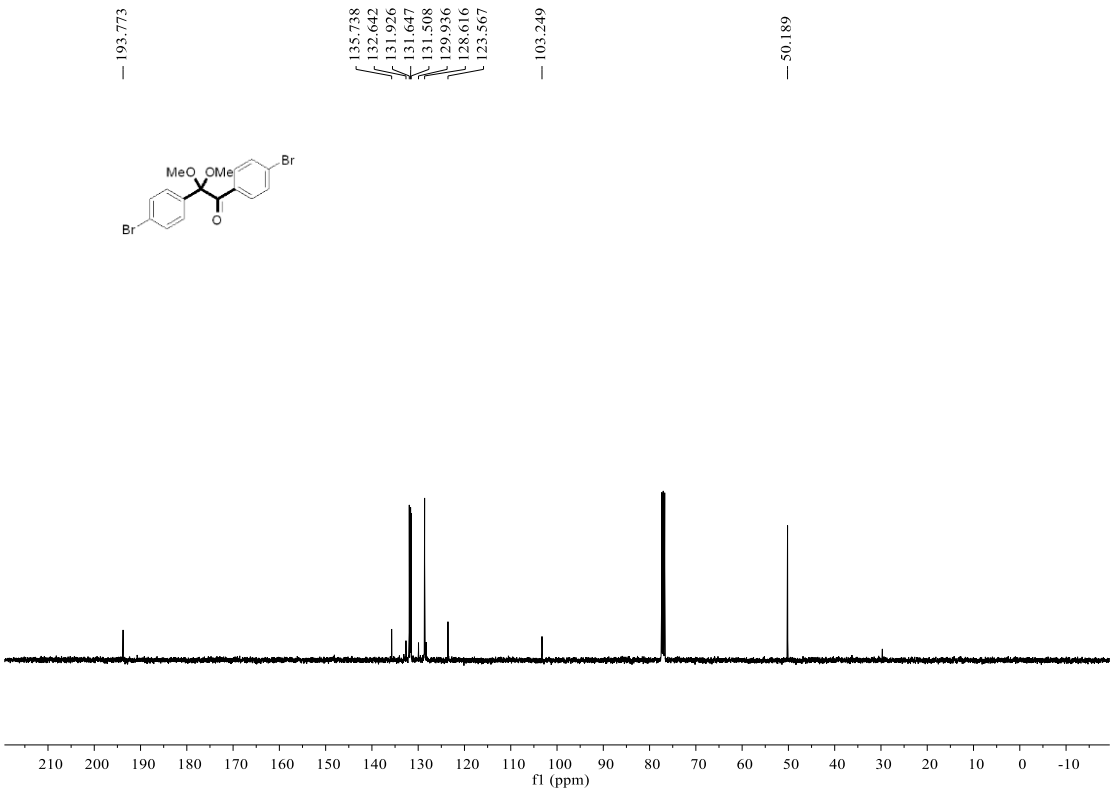




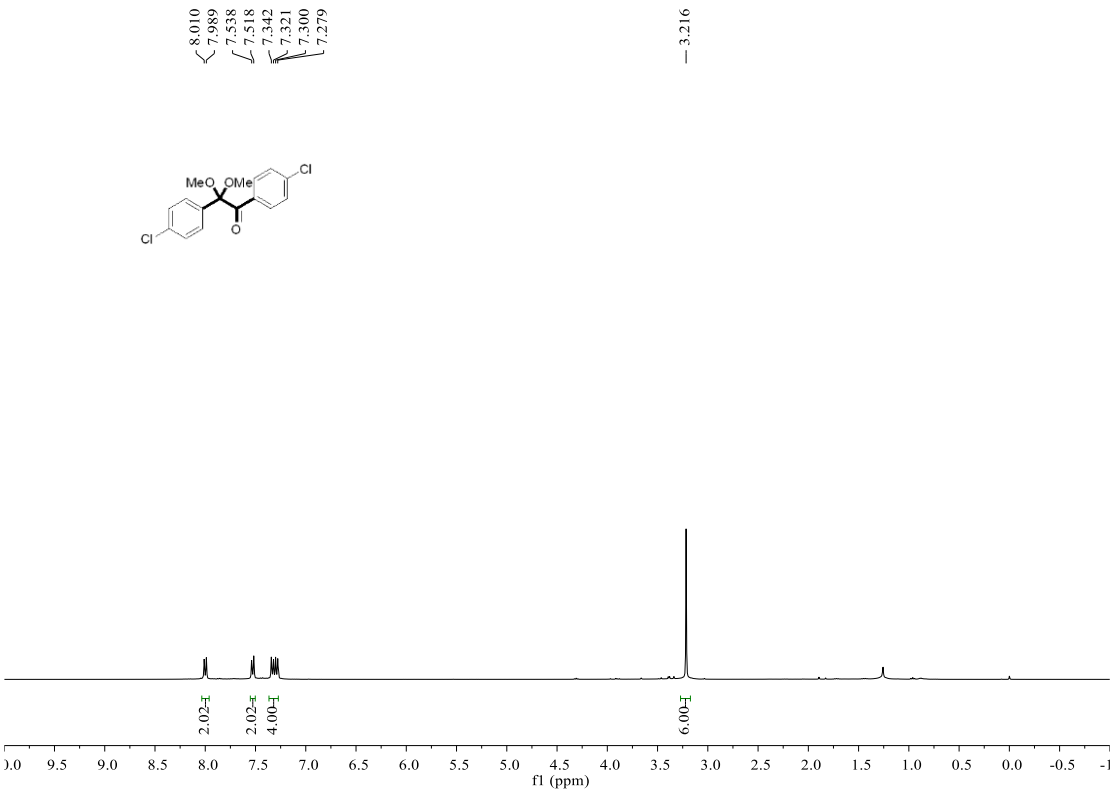

-105.109
-112.566
MeO
OMe
F
F
O
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

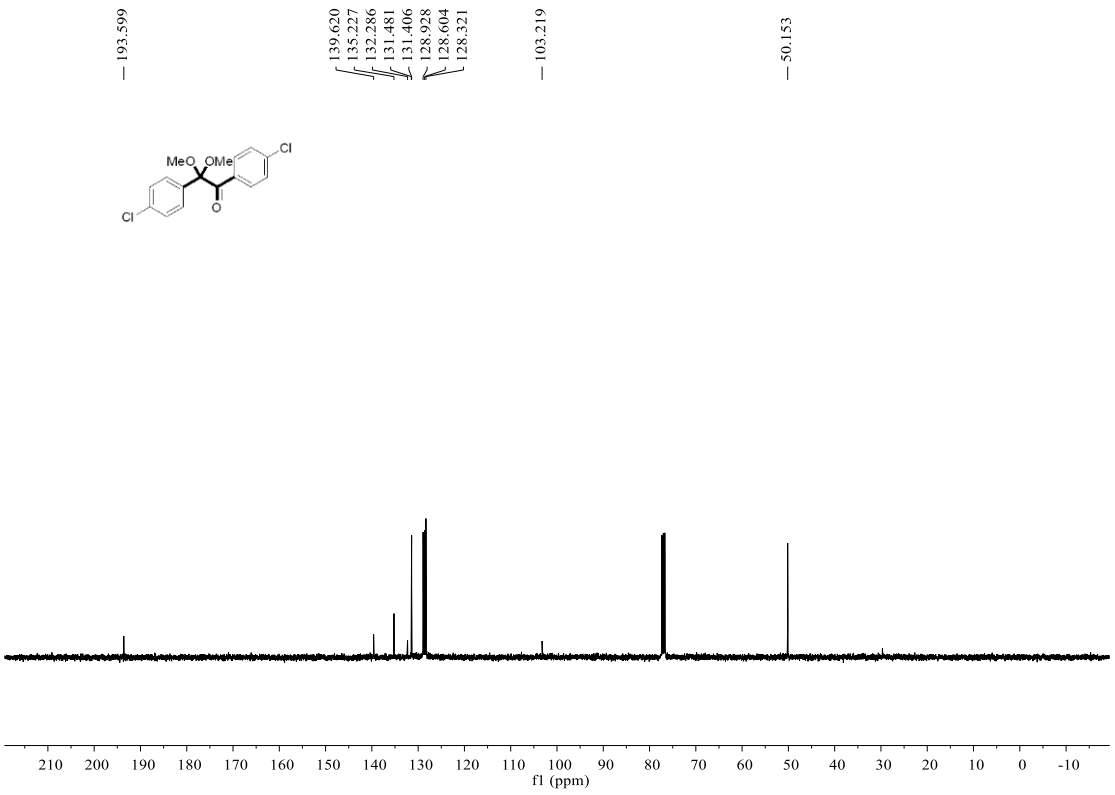
1, 2-bis(4-bromophenyl)-2, 2-dimethoxyethan-1-one (3f)



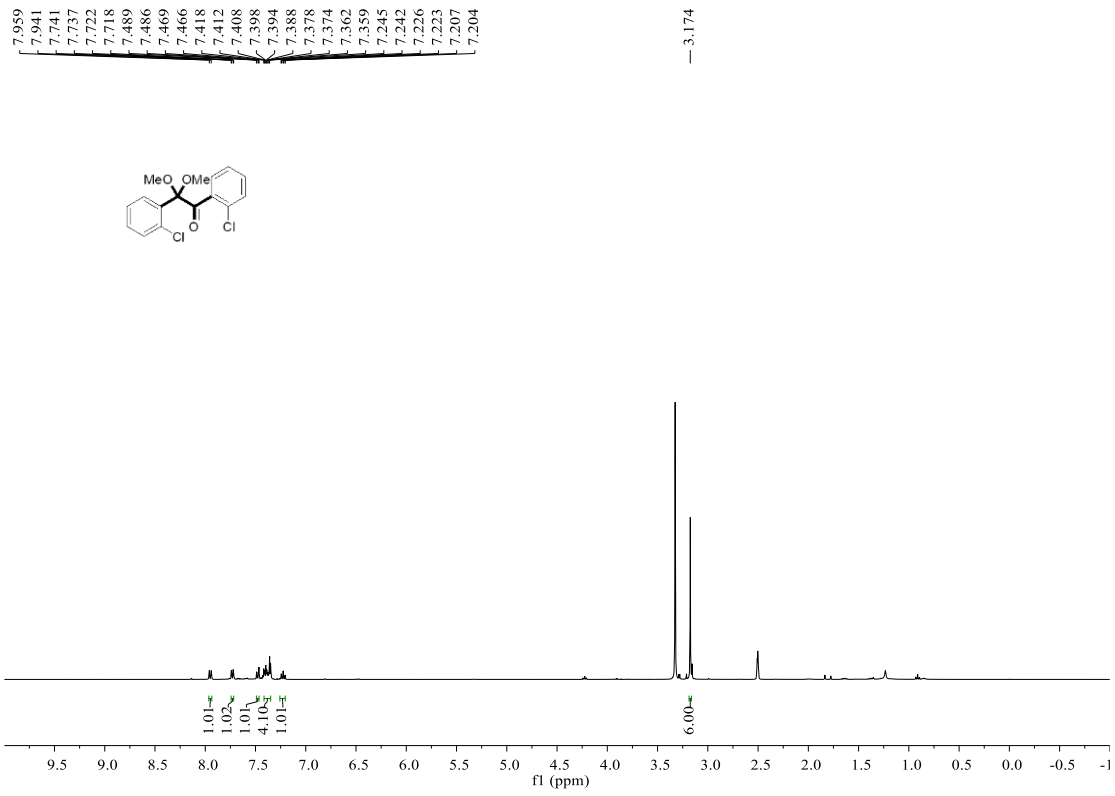


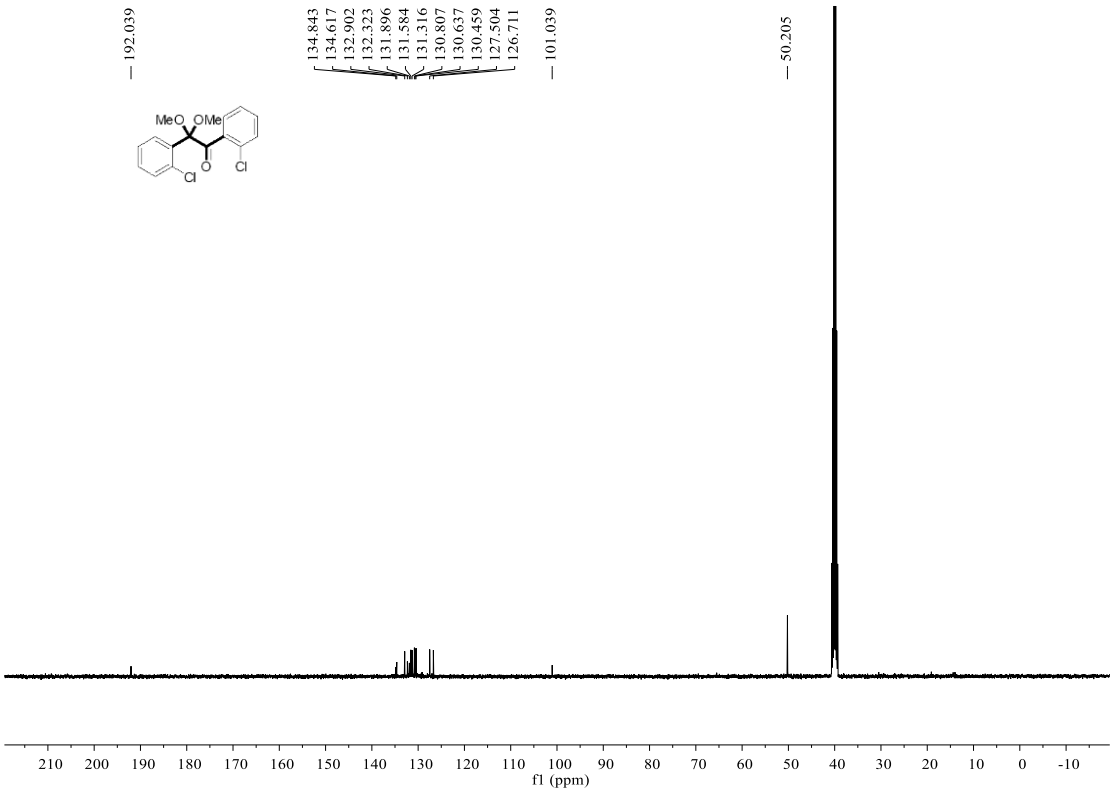
1, 2-bis(4-chlorophenyl)-2, 2-dimethoxyethan-1-one (3g)



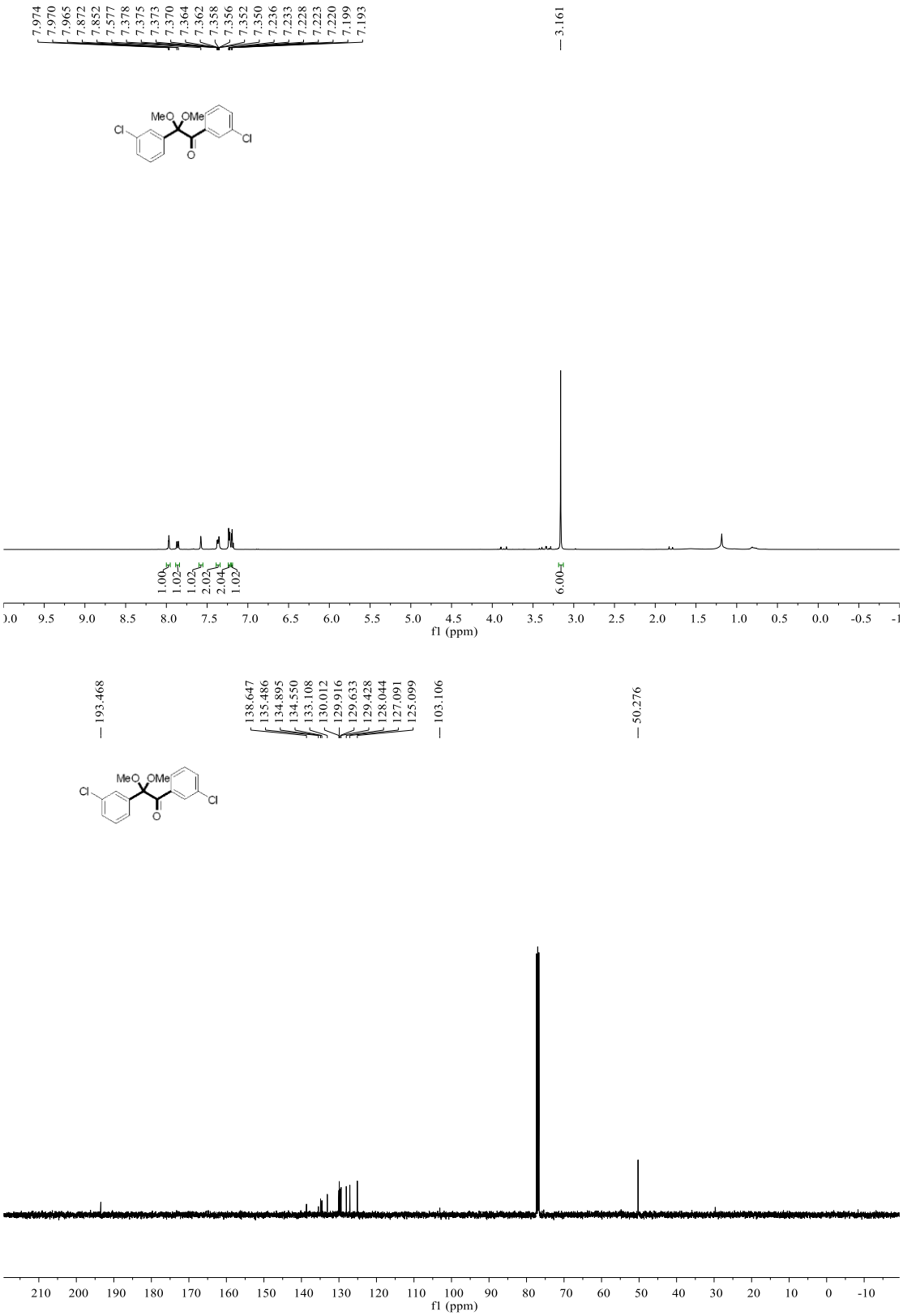


1, 2-bis(2-chlorophenyl)-2, 2-dimethoxyethan-1-one (3h)

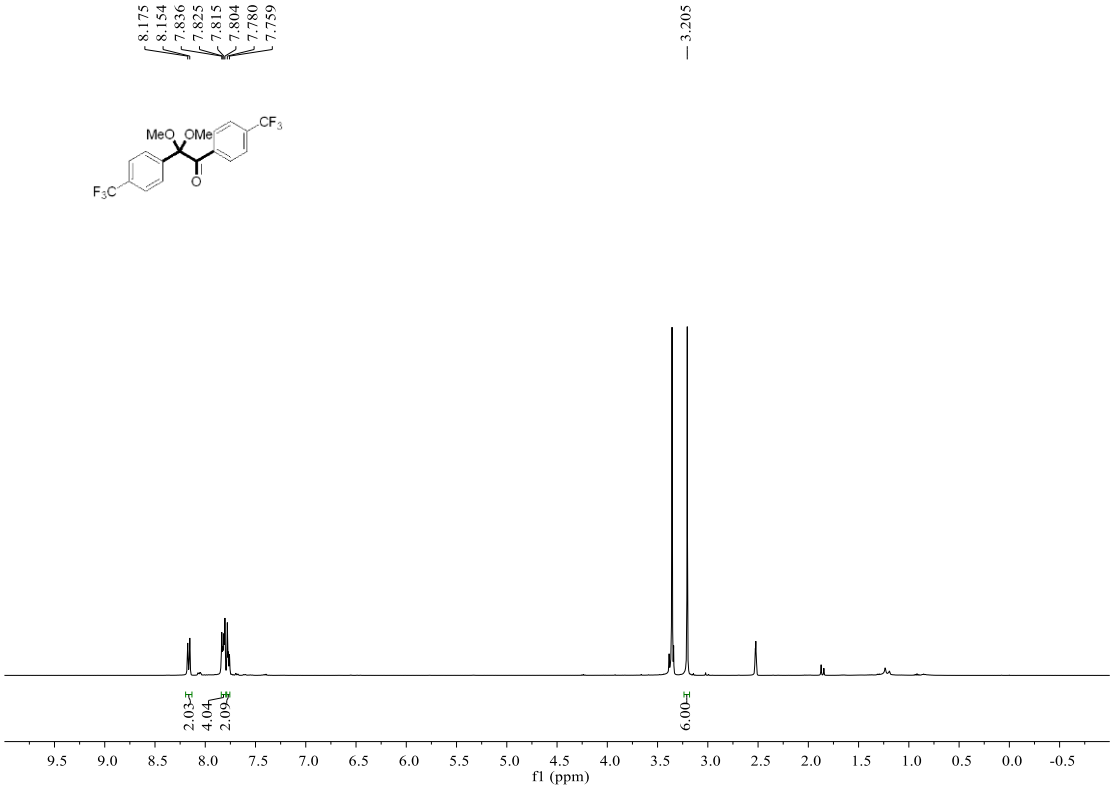


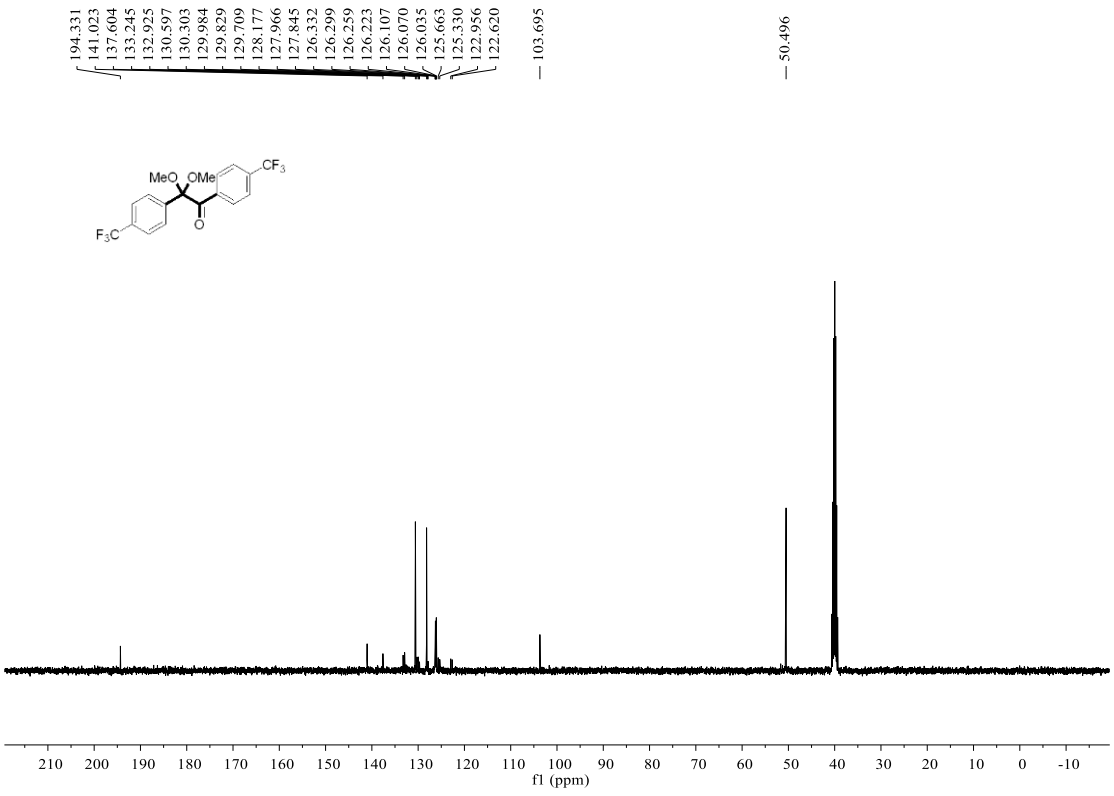


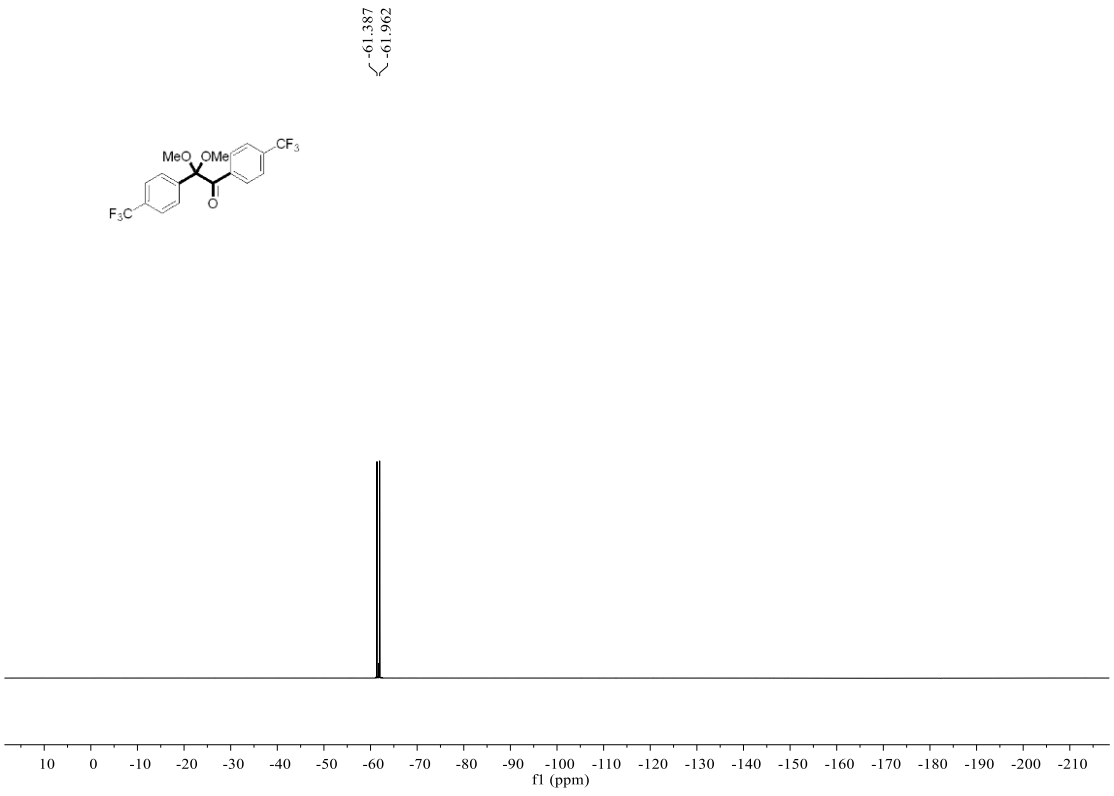
1, 2-bis(3-chlorophenyl)-2, 2-dimethoxyethan-1-one (3i)



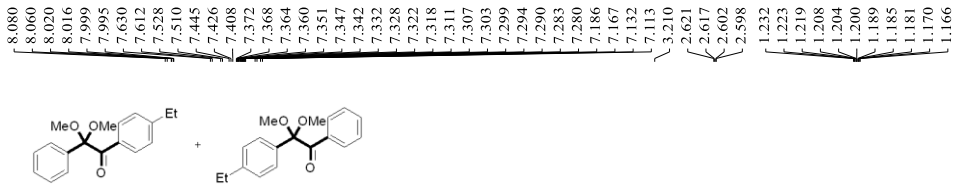
2, 2-dimethoxy-1, 2-bis(4-(trifluoromethyl)phenyl)ethan-1-one (3j)

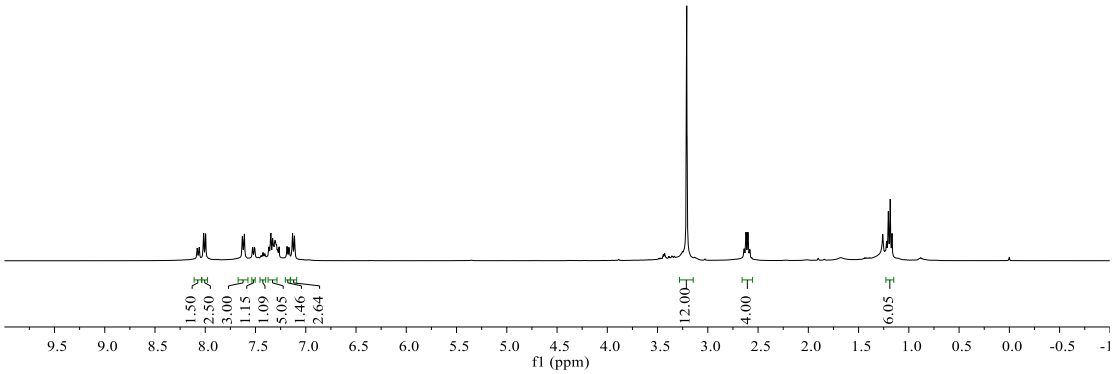


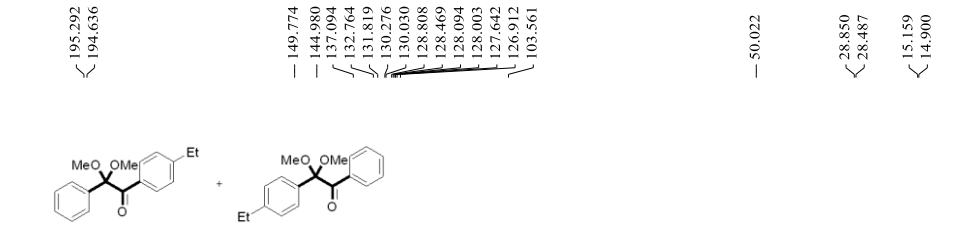


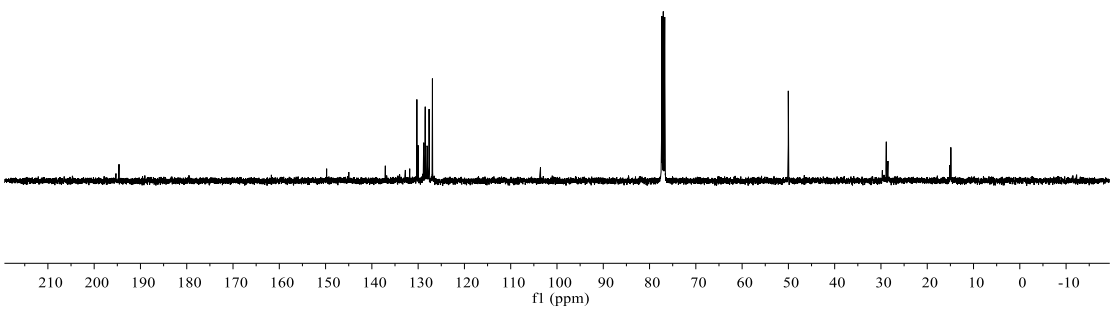

-61.387
-61.962
MeO
OMe
O
CF3
F3C
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)

1-(4-ethylphenyl)-2, 2-dimethoxy-2-phenylethan-1-one (3k)

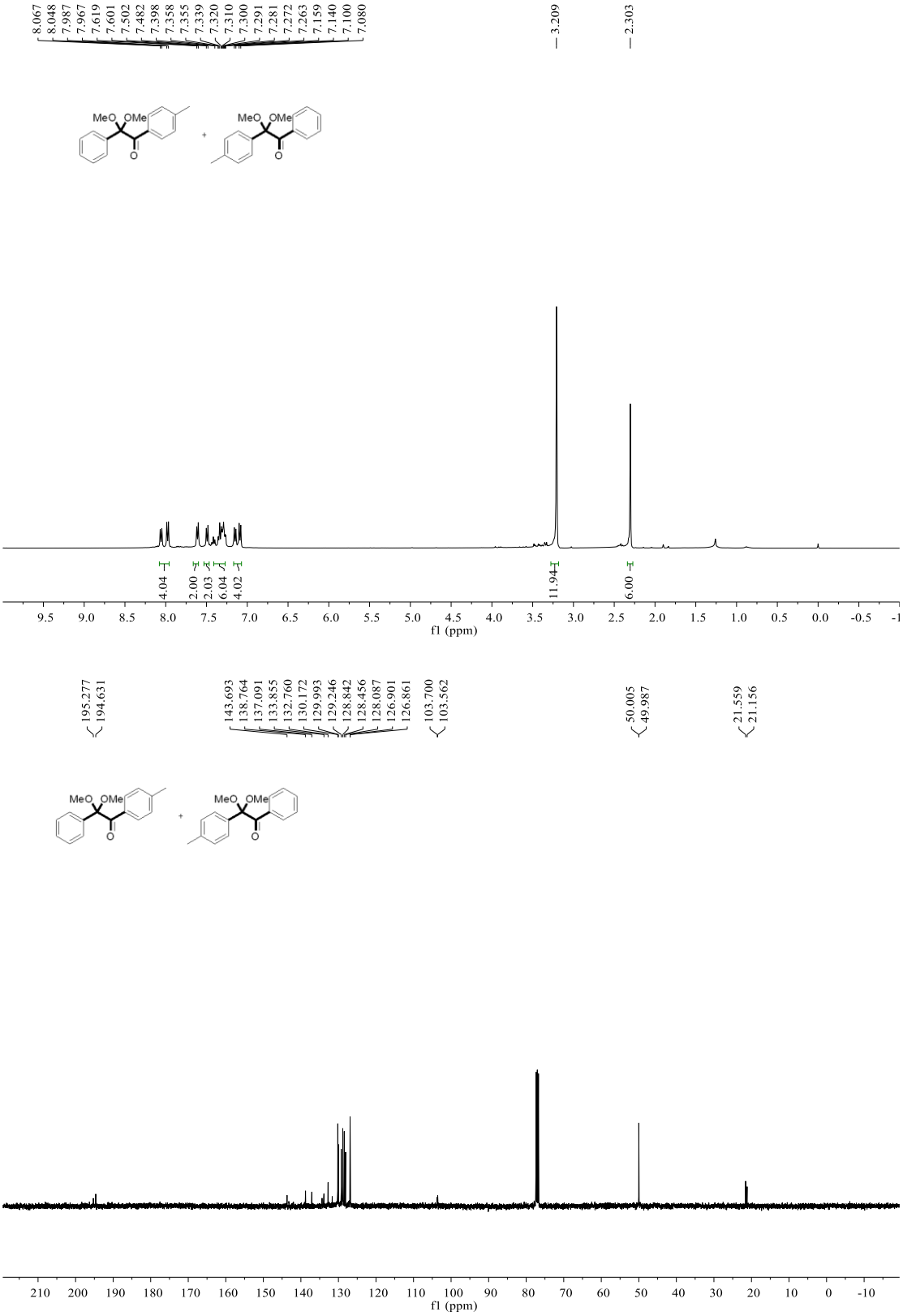




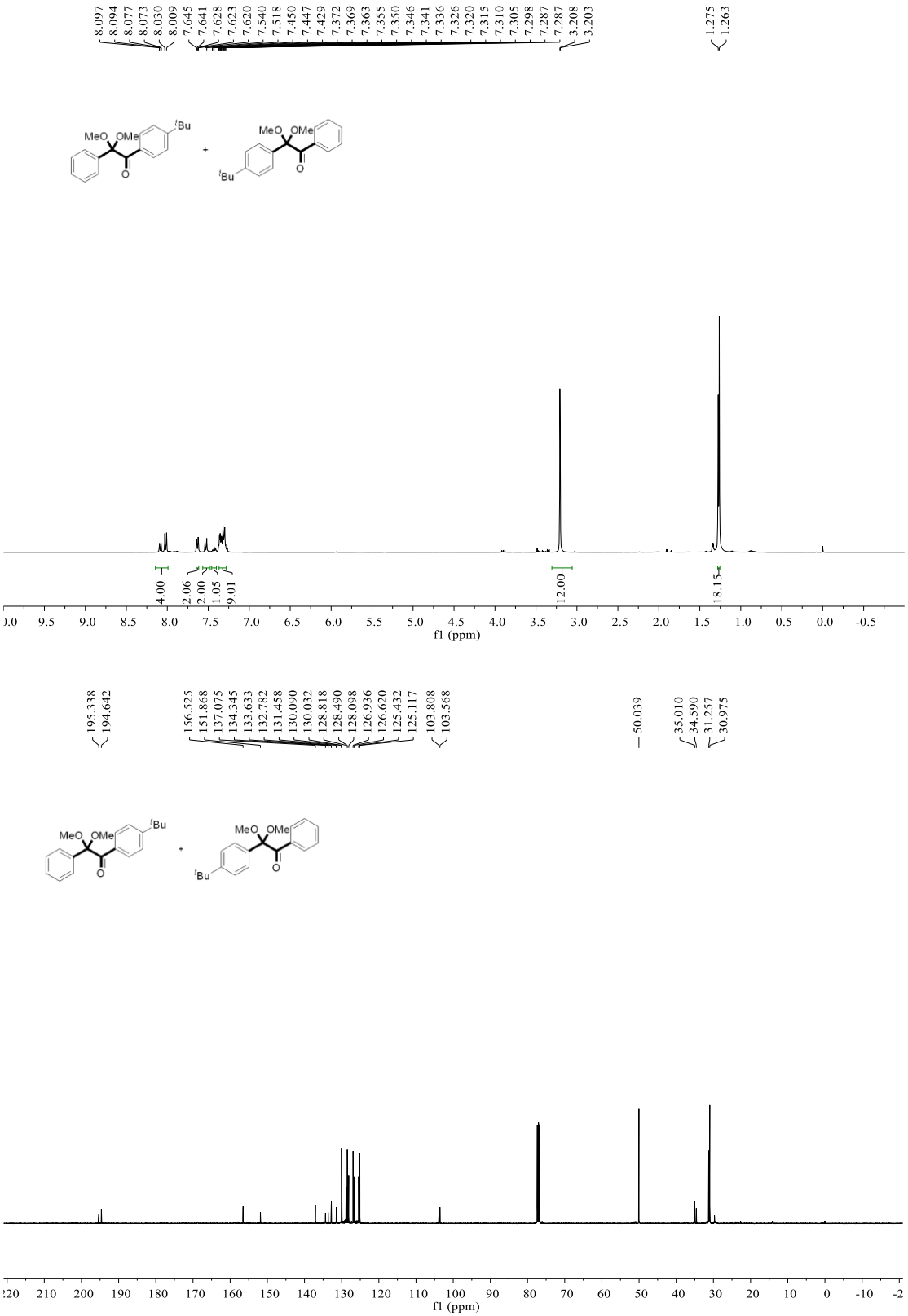




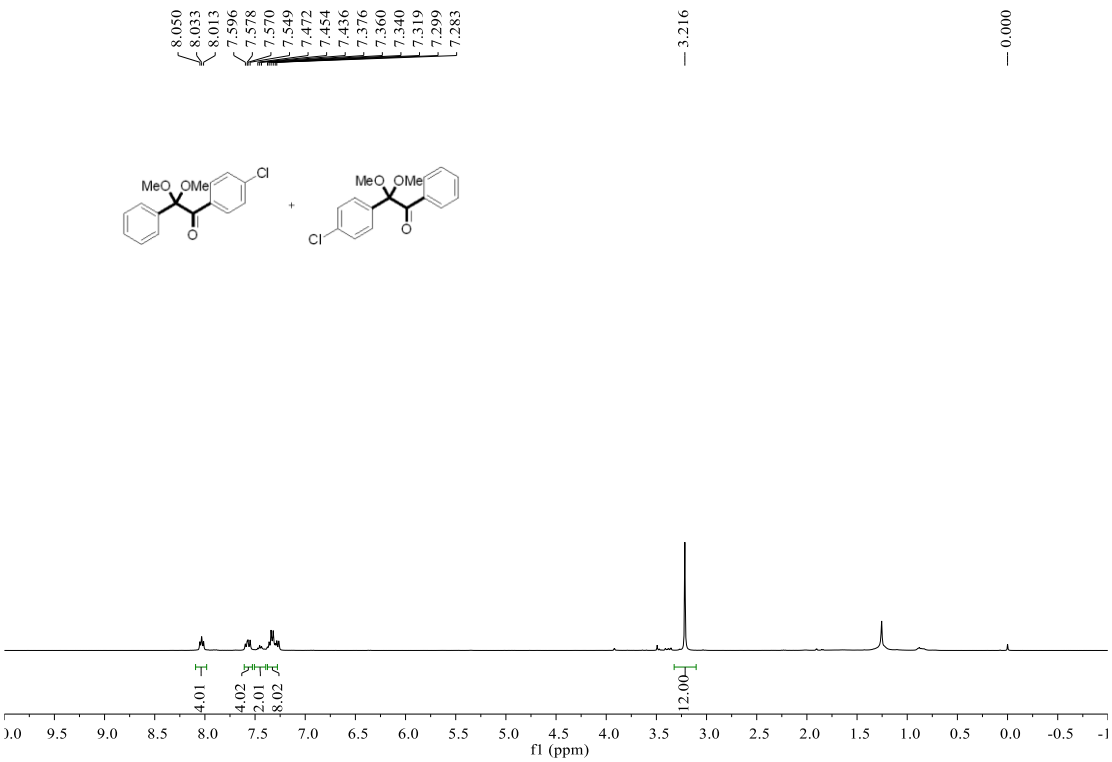
2, 2-dimethoxy-2-phenyl-1-(p-tolyl)ethan-1-one (3l)

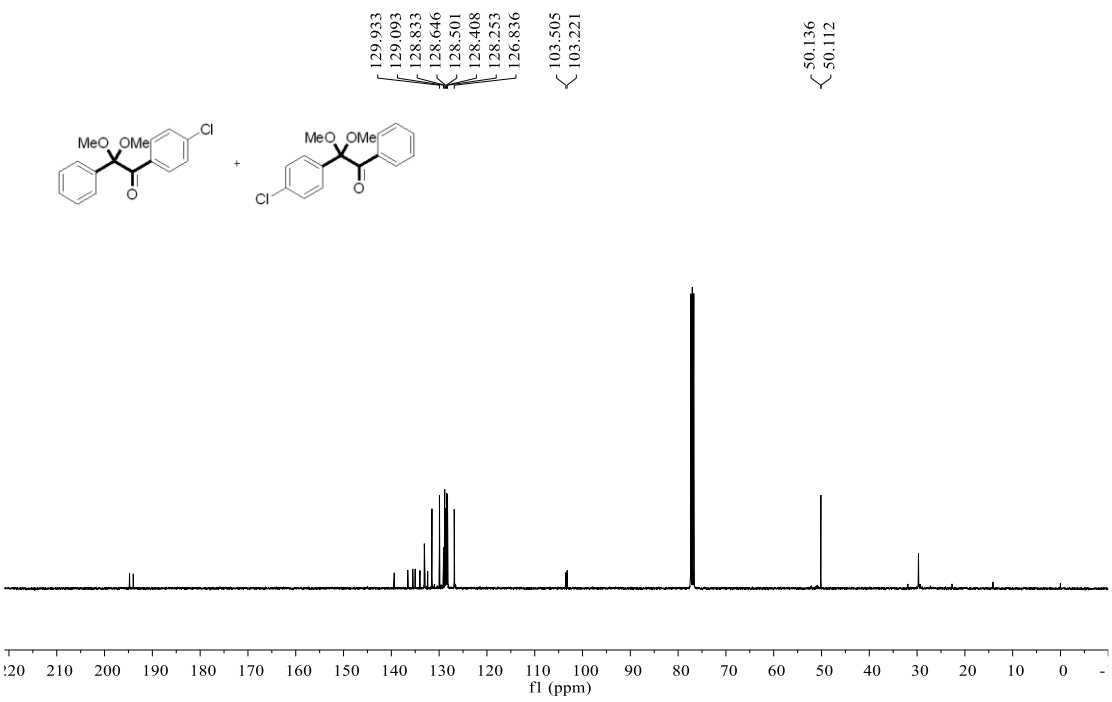


1-(4-(tert-butyl)phenyl)-2,2-dimethoxy-2-phenylethan-1-one (3m)

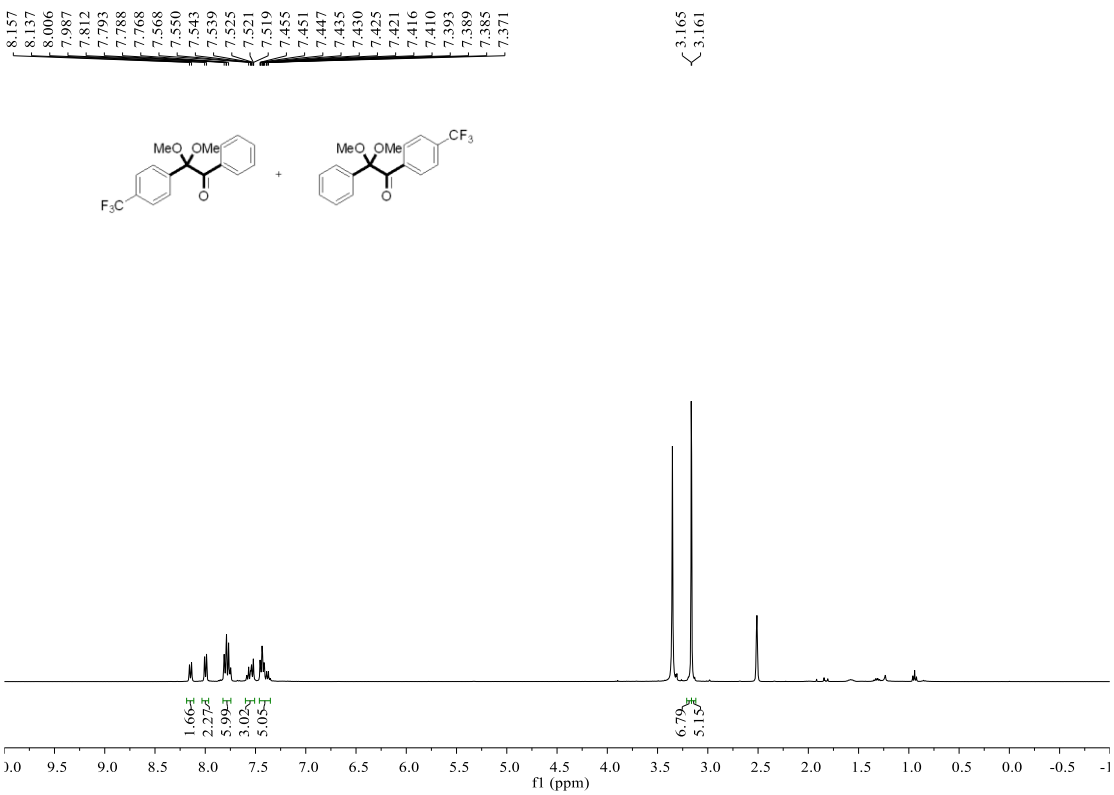


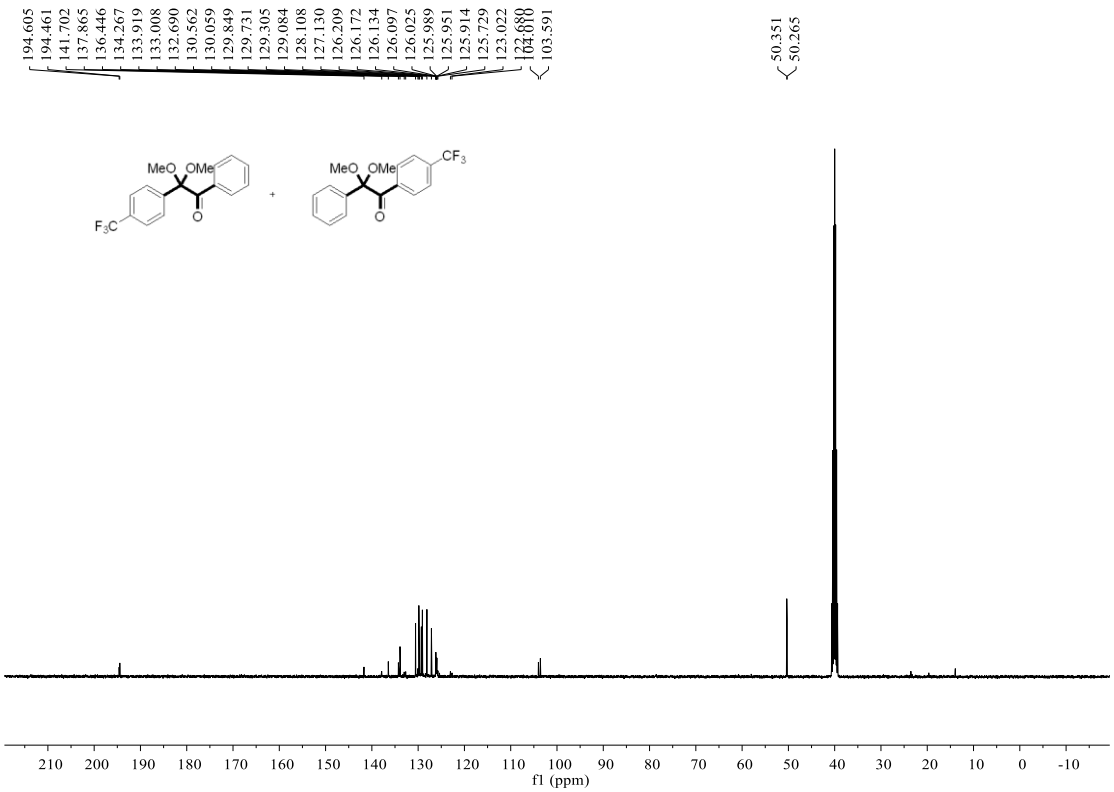
1-(4-chlorophenyl)-2,2-dimethoxy-2-phenylethan-1-one (3n)

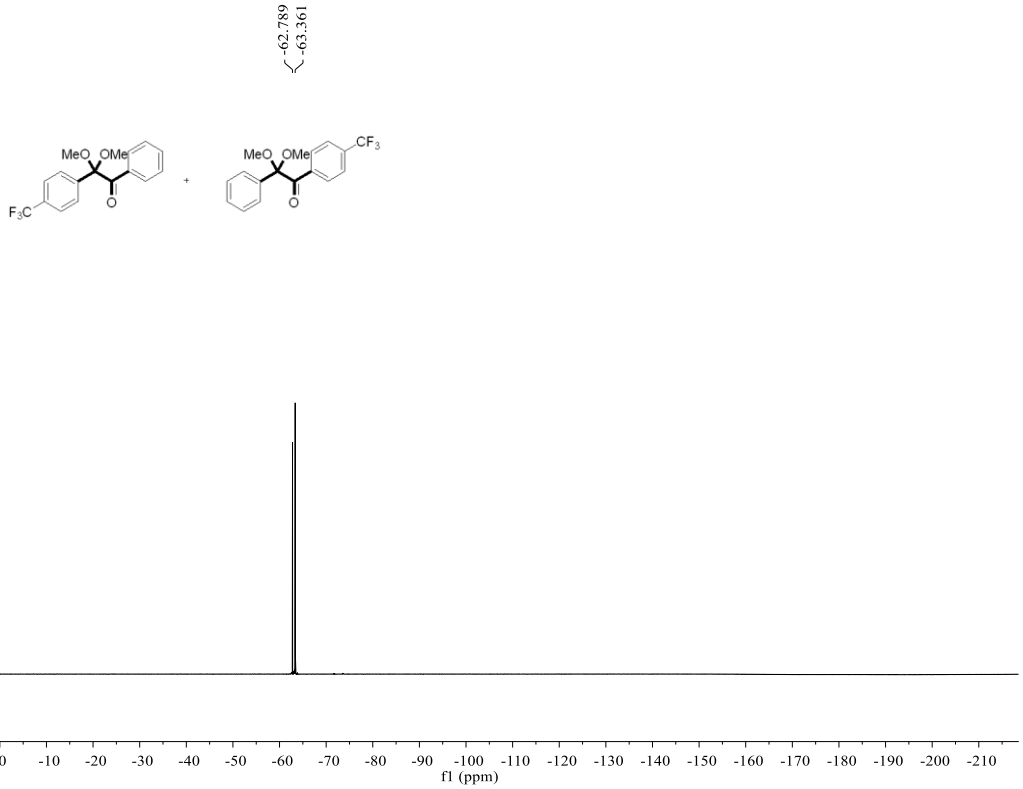




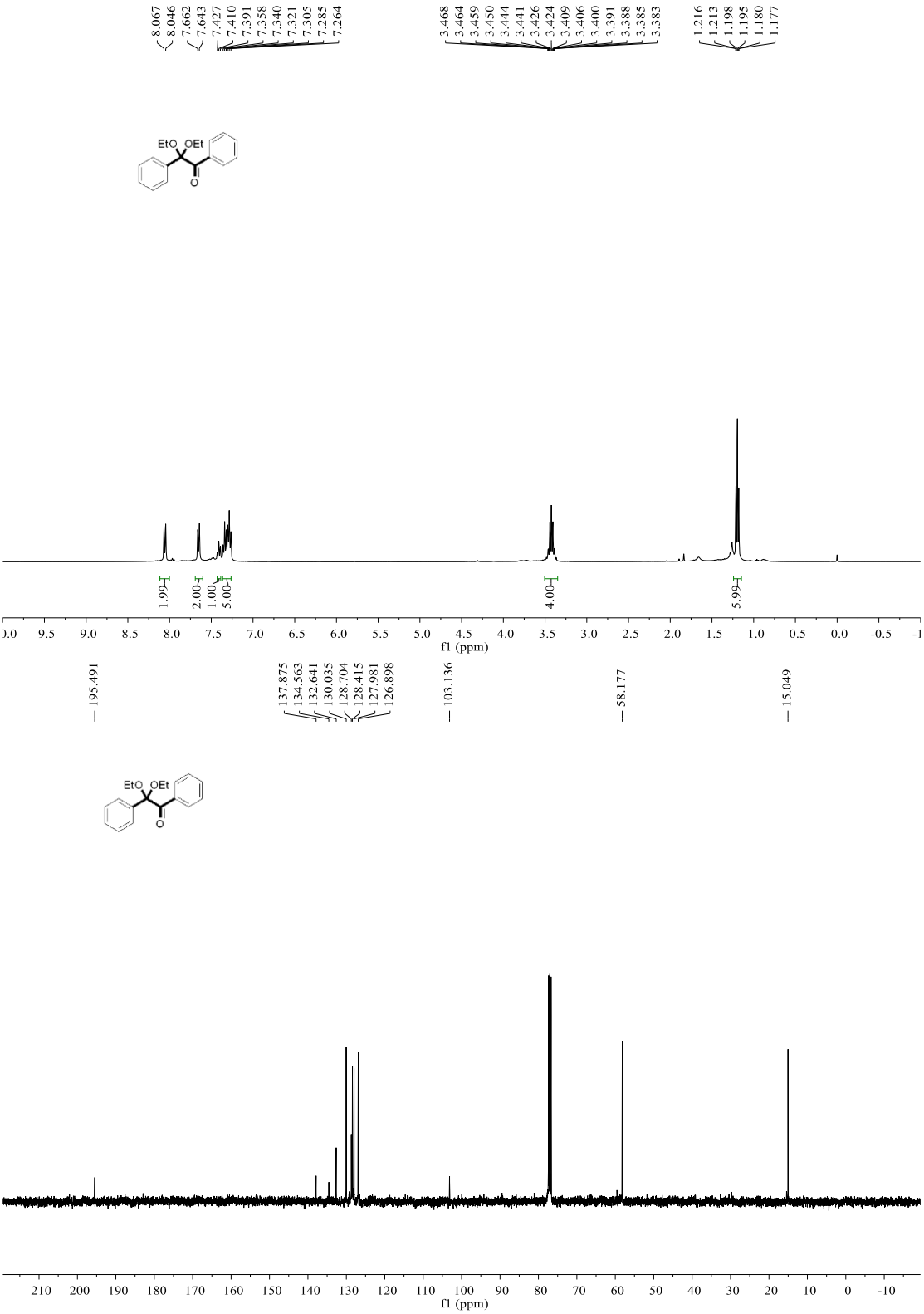
2, 2-dimethoxy-1-phenyl-2-(4-(trifluoromethyl)phenyl)ethan-1-one (3o)



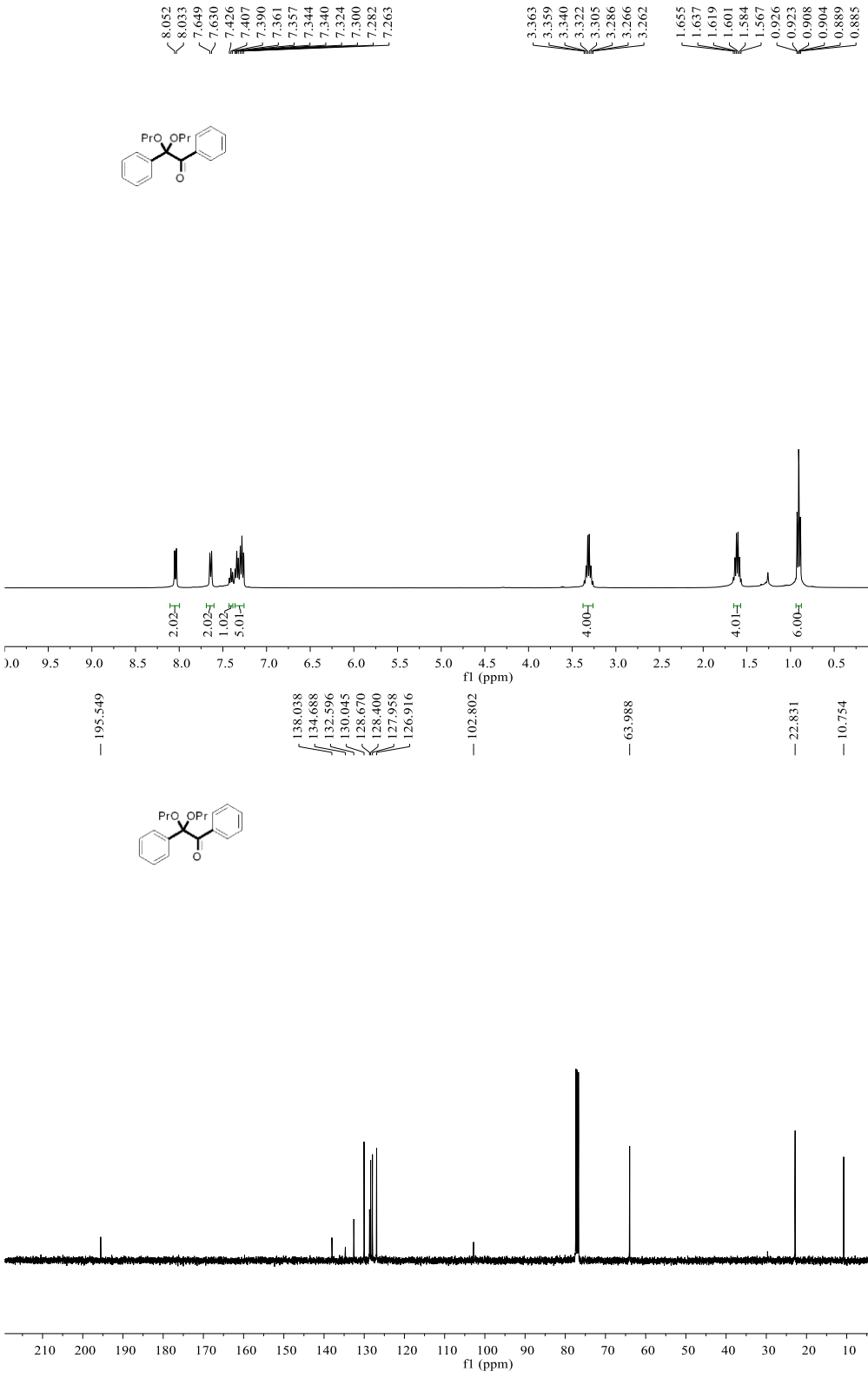



-62.789
-63.361
MeO
OMe
O
F3C
CF3
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)

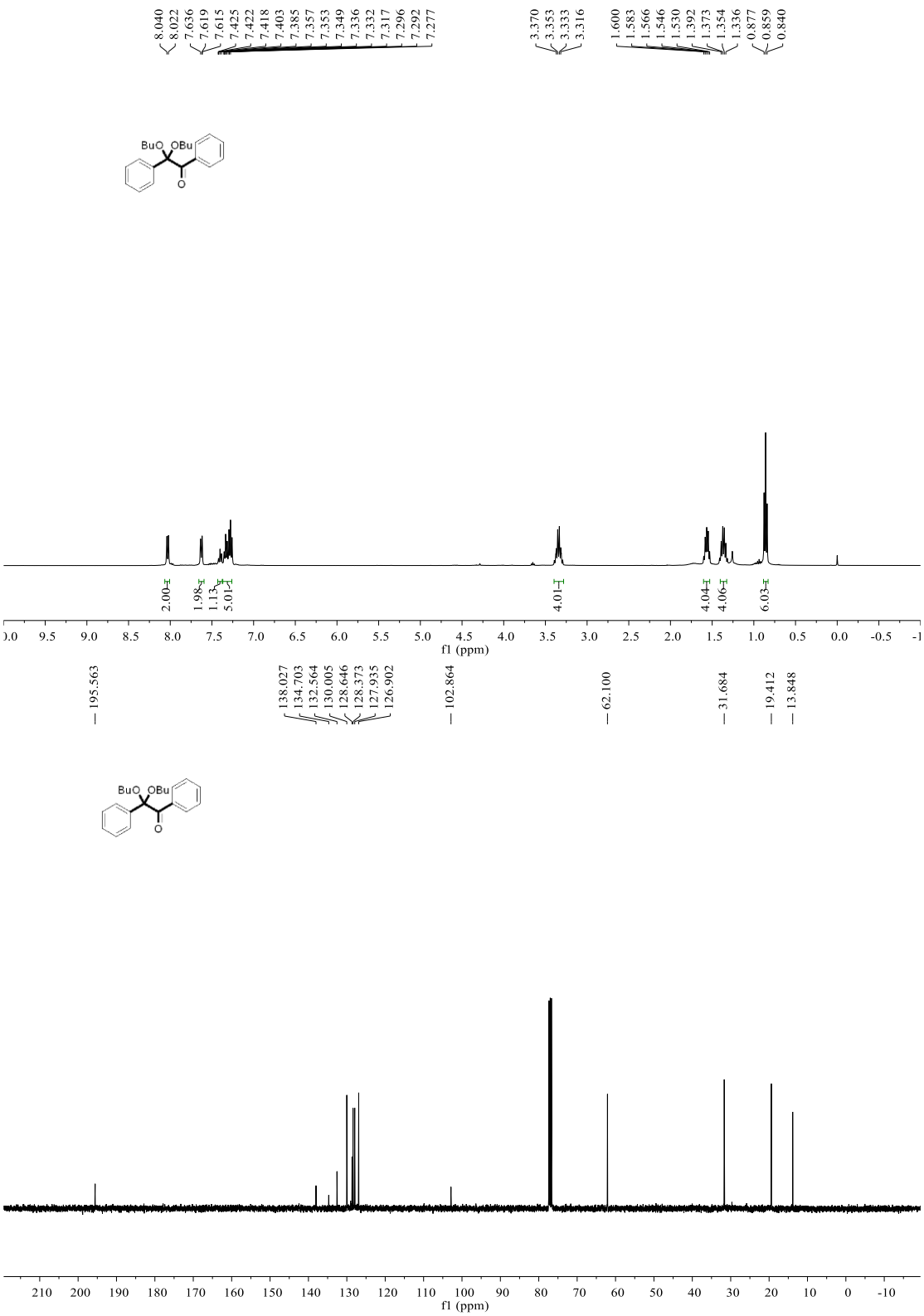
2, 2-diethoxy-1, 2-diphenylethan-1-one (3p)



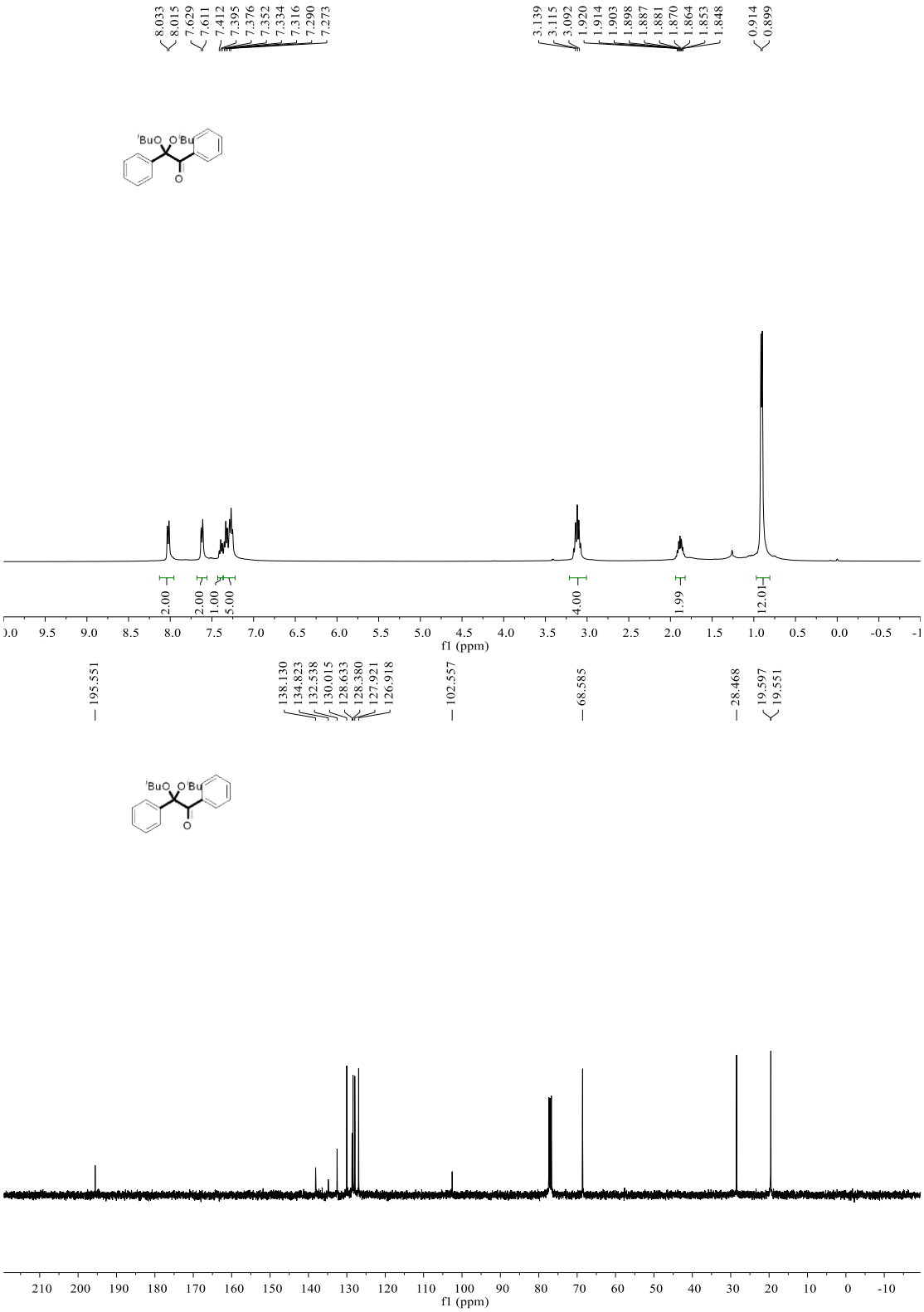
1, 2-diphenyl-2, 2-dipropoxyethan-1-one (3q)



2, 2-dibutoxy-1, 2-diphenylethan-1-one (3r)



2, 2-diisobutoxy-1, 2-diphenylethan-1-one (3s)



methyl diphenylphosphinate