Supporting Information

Palladium-Catalyzed Aerobic Dehydrogenation to α,β-unsaturated Carbonyl Compounds

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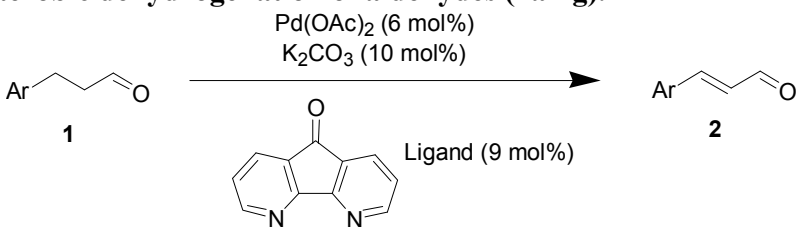
EXPERIMENTAL

General Methods. Commercial reagents were used as received, unless otherwise stated. Common solvents were purified before use. DMF and DMSO were purified by distillation from calcium hydride under reduced pressure. All reagents were reagent grade and purified when necessary. Reactions were monitored by thin layer chromatography (TLC) using Huanghai silica gel plate with HSGF 254. Qingdao Haiyang Chemical HG/T2354-92 silica gel was used for flash column chromatography. Gas chromatograph was recorded on a SHIMADZU GC-2014 spectrometer. Carbon and proton NMR spectra were recorded on a Bruker DRX-500 spectrometer at 298 K. ^{1}H NMR chemical shifts are reported as values (ppm) relative to internal tetramethylsilane and splitting patterns are designated as: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet. ^{13}C NMR chemical shifts are reported as values (ppm) relative to chloroform-d (77.0 ppm). High resolution mass spectra (HRMS) were recorded on a TurboIonspray QSTAR Elite Biosystem. The starting materials for the aerobic dehydrogenation, saturated aldehydes **1a-1g**, ketones **3a-3i**, and esters were synthesized according to literature reported procedures.[S1-S6]

General procedure for aerobic dehydrogenation of aldehydes (1a-1g).



$Pd(OAc)_2$ (13.4mg, 0.06mmol), K_2CO_3 (13.8 mg, 0.1mmol) and 4,5-diazafluorenone (16.4mg, 0.09mmol) were weighed in a 2-dram vial. Dry DMF (1 mL) was added, followed by the addition of the corresponding aldehyde (1 mmol). The reaction mixture was stirred at 30°C in an open flask manner for 36 h. The progress of the reaction was monitored by TLC and GC. Upon completion, the reaction mixture was diluted with EtOAc (1 mL)/water (4 mL), and separated. The aqueous layer was extracted twice with EtOAc (1 mL). The combined organic phase was washed with water, brine, dried over sodium sulfate, filtered and concentrated. The residue was purified by silica column chromatography (n-hexane/ethyl acetate) to give the desired unsaturated aldehyde **2a-2g**.

(*E*)-3-Phenyl-propenal 2a: Pale yellow oil. Yield: 86%. ^{1}H NMR (500 MHz, $CDCl_3$, 298 K)δ: 6.74 (dd, *J* = 7.8, 15.9 Hz, 1H), 7.43-7.45 (m, 3H), 7.49 (d, *J* = 15.9 Hz, 1H), 7.56-7.59 (m, 2H), 9.71 (d, *J* = 7.8 Hz, 1H) ppm. ^{13}C NMR (500MHz, $CDCl_3$, 298 K) δ: 128.5, 128.6, 129.1, 131.2, 134.0, 152.7, 193.7 ppm; HRMS $(M+H)^+$ calcd for C_9H_9O: 133.0635. Found: 133.0655.

(*E*)-4-(3-Oxo-propenyl)-benzoic acid ethyl ester 2b: Pale yellow oil. Yield: 84%. ^{1}H NMR (300 MHz, $CDCl_3$, 298 K)δ: 1.36 (t, *J* = 7.2 Hz, 3H), 4.34 (q, *J* = 7.2 Hz, 2H), 6.73 (dd, *J* = 7.5, 16.2 Hz, 1H), 7.46 (d, *J* = 16.2 Hz, 1H), 7.58 (d, *J* = 8.1 Hz, 2H), 8.04 (d, *J* = 8.4 Hz, 2H), 9.69 (d, *J* = 7.8 Hz, 1H) ppm. ^{13}C NMR (300MHz, $CDCl_3$, 298 K) δ: 14.3, 61.3, 128.3, 130.1, 130.2, 132.5, 137.9, 151.0, 165.7, 193.4 ppm; HRMS $(M+H)^+$ calcd for $C_{12}H_{13}O_3$: 205.0865. Found: 205.0857.

(*E*)-3-(4-Acetyl-phenyl)-propenal 2c: Pale yellow oil. Yield: 87%. ^{1}H NMR (300 MHz, $CDCl_3$, 298 K)δ: 2.64 (s, 3H), 6.79 (dd, *J* = 7.5, 16.2 Hz, 1H), 7.52 (d, *J* = 16.2 Hz, 1H), 7.66 (d, *J* = 8.4 Hz, 2H), 8.01 (d, *J* = 8.4 Hz, 2H), 9.75 (d, *J* = 7.5 Hz, 1H) ppm. ^{13}C NMR (300MHz, $CDCl_3$, 298 K) δ: 26.8, 128.6, 129.0, 130.5, 138.2, 138.7, 150.8, 193.4, 197.3 ppm; HRMS $(M+H)^+$ calcd for $C_{11}H_{11}O_2$: 175.0759. Found: 175.0753.

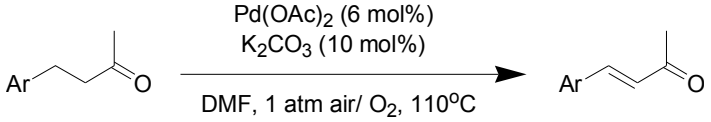
(*E*)-3-(4-Methoxy-phenyl)-propenal 2d: Pale yellow oil. Yield: 77%. ^{1}H NMR (300 MHz, $CDCl_3$, 298 K)δ: 3.87 (s, 3H), 6.61 (dd, *J* = 7.8, 16.2 Hz, 1H), 6.95 (d, *J* = 6.9 Hz, 2H), 7.43 (d, *J* = 16.2 Hz, 1H), 7.52 (d, *J* = 6.9 Hz, 2H), 9.65 (d, *J* = 7.8 Hz, 1H) ppm. ^{13}C NMR (300MHz, $CDCl_3$, 298 K) δ: 55.5, 114.6, 126.5, 126.8, 130.4, 152.8, 162.2, 193.8 ppm; HRMS $(M+H)^+$ calcd for $C_{10}H_{11}O_2$: 163.0759. Found: 163.0749.

(*E*)-3-*m*-Tolyl-propenal 2e: Pale yellow oil. Yield: 88%. ^{1}H NMR (500 MHz, $CDCl_3$, 298 K)δ: 2.37 (s, 3H), 6.61 (dd, *J* = 7.7, 15.9 Hz, 1H), 7.23-7.43 (m, *5*H), 9.67 (d, *J* = 7.7 Hz, 1H) ppm. ^{13}C NMR (500MHz, $CDCl_3$, 298 K) δ: 21.4, 125.8, 128.6, 129.1, 129.3, 132.2, 134.1, 138.9, 153.1, 193.8 ppm; HRMS $(M+H)^+$ calcd for $C_{10}H_{11}O$: 147.0810. Found: 147.0802.

(*E*)-3-(2,4-Dimethyl-phenyl)-propenal 2f: Pale yellow oil. Yield: 78%. ^{1}H NMR (500 MHz, $CDCl_3$, 298 K)δ: 2.34 (s, 3H), 2.43, (s, 3H), 6.61 (dd, *J* = 7.7, 15.8 Hz, 1H), 7.04 (d, *J* = 6.4 Hz, 2H), 7.47 (d, *J* = 8.5 Hz, 1H), 7.72 (d, *J* = 15.8 Hz, 1H), 9.69 (d, *J* = 7.7 Hz, 1H) ppm. ^{13}C NMR (500MHz, $CDCl_3$, 298 K) δ: 19.8, 21.5, 127.0, 127.6, 128.7, 130.2, 132.0, 138.1, 141.7, 150.4, 194.0 ppm; HRMS $(M+H)^+$ calcd for $C_{10}H_{13}O$: 161.0966. Found: 161.0960.

(*E*)-3-(2,4-Dichloro-phenyl)-propenal 2g: White solid. Yield: 66%; m. p. 106-108 °C (lit. m.p. 105-107 °C)[S7]; ^{1}H NMR (500 MHz, $CDCl_3$, 298 K)δ: 6.67 (dd, *J* = 7.6, 16.1 Hz, 1H), 7.32 (d, *J* = 8.5 Hz, 1H), 7.49 (s, 1H), 7.60 (d, *J* = 8.5 Hz, 1H), 7.86, (d, *J* = 16.1 Hz, 1H), 9.76 (d, *J* = 7.6 Hz, 1H) ppm. ^{13}C NMR (500MHz, $CDCl_3$, 298 K) δ: 127.9, 128.7, 130.4, 130.9, 131.0, 135.9, 137.6, 146.5, 193.2 ppm; HRMS $(M+H)^+$ calcd for $C_9H_7Cl_2O$: 200.9874. Found: 200.9875.

General procedure for aerobic dehydrogenation of ketones (3a-3i).



3 **4**

$Pd(OAc)_2$ (13.4mg, 0.06mmol), K_2CO_3 (13.8 mg, 0.1mmol) were weighed in a 2-dram vial. Dry DMF (1 mL) was added, followed by the addition of the corresponding ketone (1 mmol). The reaction mixture was stirred at 110°C open flask or under an air/O_2 balloon for 24 h. The progress of the reaction was monitored by TLC and GC. Upon completion, the reaction mixture was diluted with EtOAc (1 mL)/water (4 mL), and separated. The aqueous layer was extracted twice with EtOAc (1 mL). The combined organic phase was washed with water, brine, dried over sodium sulfate, filtered and concentrated. The residue was purified by silica column chromatography (n-hexane/ethyl acetate) to give the desired unsaturated ketone **4a-4i**.

(*E*)-4-Phenyl-but-3-en-2-one 4a: White solid. Yield: 92%; m. p. 36-38 °C (lit. m.p. 35-37 °C)[S8]; ^{1}H NMR (300 MHz, $CDCl_3$, 298 K) δ: 2.38 (s, 3H), 6.72 (d, *J* = 16.2 Hz, 1H), 7.38-7.42 (m, 3H), 7.50 (d, *J* = 16.2 Hz,

1H), 7.52-7.56 (m, 2H) ppm. ^{13}C NMR (500MHz, $CDCl_3$, 298 K) δ: 27.5, 127.2, 128.3, 128.9, 130.5, 134.4, 143.5, 198.5 ppm; HRMS $(M+H)^+$ calcd for $C_{10}H_{11}O$: 147.0810. Found: 147.0809.

(*E*)-4-(4-Bromo-phenyl)-but-3-en-2-one 4b: Pale yellow oil. Yield: 81%. ^{1}H NMR (500 MHz, $CDCl_3$, 298 K) δ: 2.38 (s, 3H), 6.70 (d, J = 16.2 Hz, 1H), 7.39-7.46 (m, 4H), 7.53 (d, J = 8.5 Hz, 1H) ppm. ^{13}C NMR (500MHz, $CDCl_3$, 298 K) δ: 27.8, 124.9, 127.8, 129.7, 132.4, 133.6, 141.9, 197.9 ppm; HRMS $(M+H)^+$ calcd for $C_{10}H_{10}BrO$: 224.9915. Found: 224.9905.

(*E*)-4-(4-Chloro-phenyl)-but-3-en-2-one 4c: Pale yellow oil. Yield: 82%. ^{1}H NMR (500 MHz, $CDCl_3$, 298 K) δ: 2.38 (s, 3H), 6.68 (d, J = 16.2 Hz, 1H), 7.37 (d, J = 8.5 Hz, 2H), 7.44-7.49 (m, 3H) ppm. ^{13}C NMR (500MHz, $CDCl_3$, 298 K) δ: 27.7, 127.7, 129.4, 129.5, 133.2, 136.6, 141.9, 197.9 ppm; HRMS $(M+H)^+$ calcd for $C_{10}H_{10}ClO$: 181.0420. Found: 181.0416.

(*E*)-4-(4-Methoxy-phenyl)-but-3-en-2-one 4d: Pale yellow oil. Yield: 67%. ^{1}H NMR (300 MHz, $CDCl_3$, 298 K) δ: 2.37 (s, 3H), 3.85 (s, 3H), 6.61 (d, J = 16.2 Hz, 1H), 6.92 (d, J = 9.0 Hz, 2H), 7.45-7.52 (m, 3H) ppm. ^{13}C NMR (300MHz, $CDCl_3$, 298 K) δ: 27.4, 55.7, 114.5, 125.1, 127.1, 130.0, 143.4, 161.6, 198.5 ppm; HRMS $(M+H)^+$ calcd for $C_{11}H_{13}O_2$: 177.0916. Found: 177.0908.

(*E*)-4-*o*-Tolyl-but-3-en-2-one 4e: Pale yellow oil. Yield: 71%. ^{1}H NMR (300 MHz, $CDCl_3$, 298 K) δ: 2.40 (s, 3H), 2.46 (s, 3H), 6.65 (d, J = 16.2 Hz, 1H), 7.20-7.30 (m, 3H), 7.57 (d, J = 7.5 Hz, 1H), 7.83 (d, J = 16.2 Hz, 1H) ppm. ^{13}C NMR (300MHz, $CDCl_3$, 298 K) δ: 19.8, 27.8, 126.5, 126.6, 128.3, 130.3, 130.9, 133.6, 137.9, 140.9, 198.3 ppm; HRMS $(M+H)^+$ calcd for $C_{11}H_{13}O$: 161.0966. Found: 161.0965.

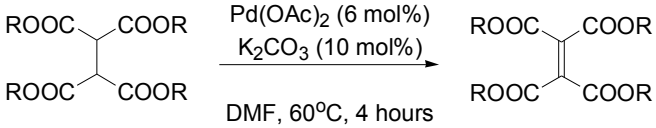
(*E*)-4-(2,4-Dimethyl-phenyl)-but-3-en-2-one 4f: Pale yellow oil. Yield: 62%. ^{1}H NMR (300 MHz, $CDCl_3$, 298 K) δ: 2.34 (s, 3H), 2.39 (s, 3H), 2.43 (s, 3H), 6.63 (d, J = 16.2 Hz, 1H), 7.04 (s, 2H), 7.49 (d, J = 8.4 Hz, 1H), 7.79 (d, J = 16.2 Hz, 1H) ppm. ^{13}C NMR (300MHz, $CDCl_3$, 298 K) δ: 19.7, 21.4, 27.8, 126.4, 127.1, 127.3, 130.1, 131.7, 137.9, 140.7, 140.9, 198.9 ppm; HRMS $(M+H)^+$ calcd for $C_{12}H_{15}O$: 175.1123. Found: 175.1115.

(*E*)-4-(2,4-Dichloro-phenyl)-but-3-en-2-one 4g: White solid. Yield: 86%; m. p. 81-83 ^{o}C (lit. m.p. 80-81 ^{o}C)[S9]; ^{1}H NMR (500 MHz, $CDCl_3$, 298 K) δ: 2.37 (s, 3H), 6.59 (d, J = 16.2 Hz, 1H), 7.22 (d, J = 8.7 Hz, 1H), 7.37 (s, 1H), 7.52 (d, J = 8.7 Hz, 1H), 7.78 (d, J = 16.2 Hz, 1H), ppm. ^{13}C NMR (500MHz, $CDCl_3$, 298 K) δ: 27.5, 127.8, 128.4, 129.9, 130.1, 131.4, 135.7, 136.6, 137.8, 197.9 ppm; HRMS $(M+H)^+$ calcd for $C_{10}H_9Cl_2O$: 215.0030. Found: 215.00225.

(*E*)-4-(2-Fluoro-phenyl)-but-3-en-2-one 4h: Pale yellow oil. Yield: 72%. ^{1}H NMR (300 MHz, $CDCl_3$, 298 K) δ: 2.35 (s, 3H), 6.74 (d, J = 16.5 Hz, 1H), 7.04-7.15 (m, 2H), 7.33 (m, 1H), 7.53 (t, J = 7.6 Hz, 1H), 7.63 (d, J = 16.5 Hz, 1H), ppm. ^{13}C NMR (300MHz, $CDCl_3$, 298 K) δ: 27.6, 116.2, 116.4, 122.58, 122.67, 124.66, 124.69, 128.81, 128.83, 129.3, 129.4, 132.0, 132.1, 135.71, 135.74, 160.5, 162.5, 198.4 ppm; HRMS $(M+H)^+$ calcd for $C_{10}H_{10}FO$: 165.0716. Found: 165.0715.

(*E*)-4-Naphthalen-2-yl-but-3-en-2-one 4i: White solid. Yield: 53%. m. p. 101-103 ^{o}C. ^{1}H NMR (300 MHz, $CDCl_3$, 298 K) δ: 2.43 (s, 3H), 6.83 (d, J = 16.2 Hz, 1H), 7.53 (m, 2H), 7.68 (m, 2H), 7.86 (m, 3H), 7.96 (s, 1H), ppm. ^{13}C NMR (300MHz, $CDCl_3$, 298 K) δ: 27.7, 123.7, 126.9, 127.5, 127.9, 128.7, 128.9, 130.4, 132.1, 133.5, 134.5, 143.6, 198.5 ppm; HRMS $(M+H)^+$ calcd for $C_{14}H_{13}O$: 197.0966. Found: 197.0960.

General procedure for aerobic dehydrogenation of esters.

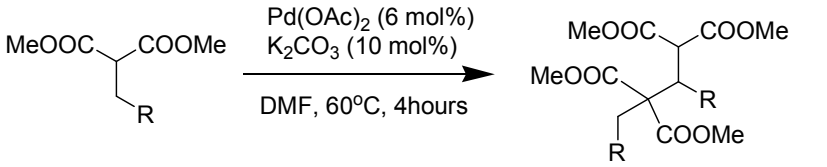


R=Me(**5a**), Et(**5b**)

$Pd(OAc)_2$ (13.4mg, 0.06mmol), K_2CO_3 (13.8 mg, 0.1mmol) were weighed in a 2-dram vial. Dry DMF (1 mL) was added, followed by the addition of the corresponding tetraesters (1 mmol). The reaction mixture was stirred at 60°C for 4 h. The progress of the reaction was monitored by TLC and GC. Upon completion, the reaction mixture was diluted with EtOAc (1 mL)/water (4 mL), and separated. The aqueous layer was extracted twice with EtOAc (1 mL). The combined organic phase was washed with water, brine, dried over sodium sulfate, filtered and concentrated. The residue was purified by silica column chromatography (n-hexane/ethyl acetate) to give the desired unsaturated ester **5a-5b.** Michael addition products **5c** and **5d** were isolated for triester substrates.

2,3-Bis-methoxycarbonyl-but-2-enedioic acid dimethyl ester 5a: White solid. Yield: 99%; m. p. 120-123 °C (lit. m.p. 119-120 °C);[S10] ^{1}H NMR (300 MHz, $CDCl_3$, 298 K) δ: 3.87 (s, 12H) ppm. ^{13}C NMR (300MHz, $CDCl_3$, 298 K) δ: 53.4, 135.5, 162.6 ppm; HRMS $(M+H)^+$ calcd for $C_{10}H_{13}O_8$: 261.0610. Found: 261.0599.

2,3-Bis-ethoxycarbonyl-but-2-enedioic acid diethyl ester 5b: White solid. Yield: 99%; m. p. 54-55 °C (lit. m.p. 53-55 °C);[S11] ^{1}H NMR (500 MHz, $CDCl_3$, 298 K) δ: 1.30 (t, J = 7.1 Hz, 12H), 4.29 (q, J = 7.1 Hz, 8H) ppm. ^{13}C NMR (500MHz, $CDCl_3$, 298 K) δ: 13.9, 62.6, 135.5, 162.5 ppm; HRMS $(M+H)^+$ calcd for $C_{10}H_{13}O_8$: 317.1236. Found: 317.1236.

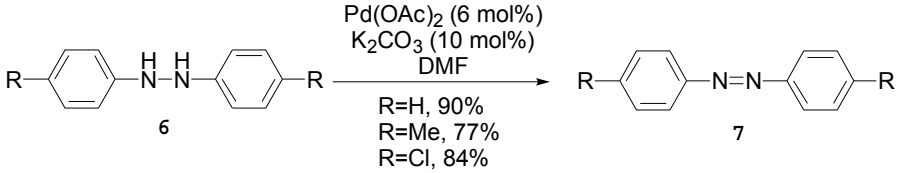


R=H(**5c**), COOMe (**5d**)

Tetramethyl butane-1,1,3,3-tetracarboxylate 5c: Pale yellow oil. Yield: 66%. ^{1}H NMR (300 MHz, $CDCl_3$, 298 K) δ: 1.44 (s, 3H), 2.55 (d, J= 6.3 Hz, 2H), 3.61 (t, J= 6.3 Hz, 1H), 3.71 (s, 6H), 3.74 (s, 6H), ppm. ^{13}C NMR (300MHz, $CDCl_3$, 298 K) δ: 20.7, 34.5, 48.4, 52.7, 52.8, 52.9, 169.7, 171.9 ppm; HRMS $(M+Na)^+$ calcd for $C_{12}H_{19}O_8Na$: 313.0899. Found: 313.0891.

Hexamethyl butane-1,1,2,3,3,4-hexacarboxylate 5d: Pale yellow oil. Yield: 65%. ^{1}H NMR (300 MHz, $CDCl_3$, 298 K) δ: 3.65 (s, 3H), 3.66 (s, 3H), 3.69 (s, 3H), 3.71 (s, 3H), 3.73 (s, 3H), 3.74-3.83 (m, 8H)ppm. ^{13}C NMR (300MHz, $CDCl_3$, 298 K) δ: 36.4, 39.7, 43.6, 47.9, 50.9, 51.9, 52.4, 56.3, 167.9, 168.8, 170.6, 171.6 ppm; HRMS $(M+H)^+$ calcd for $C_{16}H_{23}O_{12}$: 407.119. Found: 407.1208.

General procedure for aerobic dehydrogenation of hydrazines (6a-6c).



$Pd(OAc)_2$ (13.4mg, 0.06mmol), K_2CO_3 (13.8 mg, 0.1mmol) were weighed in a 2-dram vial. Dry DMF (1 mL) was added, followed by the addition of the corresponding hydrazine (1 mmol). The reaction mixture was

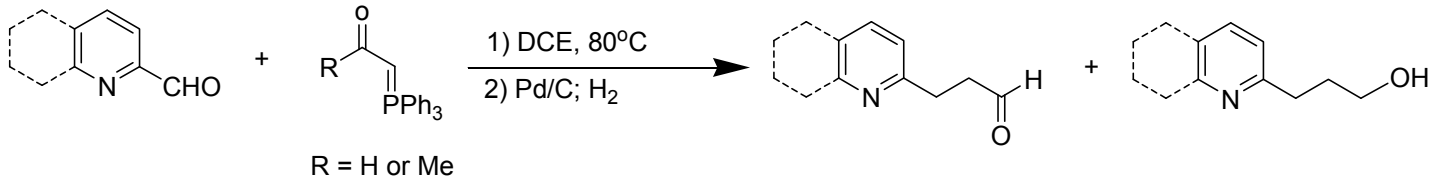
stirred at room temperature for 24 h. The progress of the reaction was monitored by TLC and GC. Upon completion, the reaction mixture was diluted with EtOAc (1 mL)/water (4 mL), and separated. The aqueous layer was extracted twice with EtOAc (1 mL). The combined organic phase was washed with water, brine, dried over sodium sulfate, filtered and concentrated. The residue was purified by silica column chromatography (n-hexane/ethyl acetate) to give the desired diazene **7a-7c**.

Diphenyldiazene 7a: Orange yellow solid. Yield: 90%. m. p. 65-67 ^{o}C (lit. m.p. 65-67 ^{o}C).[S12] ^{1}H NMR (300 MHz, $CDCl_3$, 298 K) δ: 7.57 (m, 3H), 8.05 (m, 2H) ppm. ^{13}C NMR (300MHz, $CDCl_3$, 298 K) δ: 123.0, 129.2, 131.1, 152.8 ppm. HRMS $(M+H)^+$ calcd for $C_{12}H_{11}N_2$: 183.0922. Found: 183.0914.

Di-*p*-tolyldiazene 7b: Orange yellow solid. Yield: 77%; m. p. 144-146 ^{o}C (lit. m.p. 144-145 ^{o}C).[S13] ^{1}H NMR (300 MHz, $CDCl_3$, 298 K) δ: 2.45 (s, 6H), 7.31 (d, J = 7.8 Hz, 4H), 7.83 (d, J = 8.1 Hz, 4H), ppm. ^{13}C NMR (300MHz, $CDCl_3$, 298 K) δ: 21.5, 122.7, 129.7, 141.2, 150.8 ppm. HRMS $(M+H)^+$ calcd for $C_{14}H_{15}N_2$: 211.1235. Found: 211.1229.

Bis-(4-chlorophenyl)diazene 7c: Orange yellow solid. Yield: 84%. m. p. 190-191 ^{o}C (lit. m.p. 188-189 ^{o}C).[S14] ^{1}H NMR (300 MHz, $CDCl_3$, 298 K) δ: 7.50 (d, J = 6.9 Hz, 4H), 7.87 (d, J = 6.9 Hz, 4H), ppm. ^{13}C NMR (300MHz, $CDCl_3$, 298 K) δ: 124.2, 129.4, 137.2, 150.8 ppm. HRMS $(M+H)^+$ calcd for $C_{12}H_9Cl_2N_2$: 251.0143. Found: 251.0140.

Procedure for preparation of heterocyclic substrates:



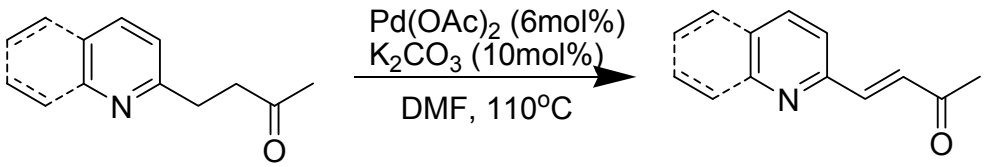
R = H or Me

Under an atmosphere of argon, to a schlenk tube were added phosphorane (3.0 mmol) and 5 mL of anhydrous CH_2ClCH_2Cl, followed by aldehyde (3.0 mmol). The resulting mixture was stirred at 80 ^{o}C for several hours till almost full conversion of the aldehyde by TLC analysis. Then the reaction was cooled to room temperature, the solvent was removed by vacuum, and 10% pd/C (0.03 mmol) and CH_3OH (10 mL) were added, the mixture was allowed to stir at room temperature in H_2 overnight. After filtration, concentrated, the desired product was obtained by column chromatography using petroleum/ethyl acetate (from 10:1 to 4:1) as eluent.

4-(pyridin-2-yl)butan-2-one: Pale yellow oil. Yield: 71%. ^{1}H NMR (500 MHz, $CDCl_3$, 298 K) δ: 2.16 (s, 3H), 2.93 (t, *J* = 7.1 Hz, 2H), 3.05 (t, *J* = 7.1 Hz, 2H), 7.07 (m, 1H), 7.16 (d, *J* = 7.7 Hz, 1H), 7.55 (m, 1H), 8.48 (d, *J* = 4.1 Hz, 1H) ppm. ^{13}C NMR (500MHz, $CDCl_3$, 298 K) δ: 30.1, 31.9, 42.6, 121.3, 123.3, 136.4, 149.3, 160.5, 208.1 ppm. HRMS $(M+H)^+$ calcd for $C_9H_{12}NO$: 150.0919. Found: 150.0931.

4-(quinolin-2-yl)butan-2-one: Pale yellow oil. Yield: 45%. ^{1}H NMR (500 MHz, $CDCl_3$, 298 K) δ: 2.17 (s, 3H), 2.98 (t, *J* = 7.2 Hz, 2H), 3.05 (t, *J* = 7.2 Hz, 2H), 7.21 (d, *J* = 8.4 Hz, 1H), 7.39 (m, 1H), 7.61 (m, Hz, 1H), 7.68 (d, *J* = 8.1 Hz, 1H), 8.48 (d, *J* = 4.1 Hz, 1H) ppm. ^{13}C NMR (500MHz, $CDCl_3$, 298 K) δ: 30.2, 32.5, 42.0, 121.8, 125.9, 126.9, 127.6, 128.8, 129.4, 136.2, 147.9, 160.9, 208.1 ppm. HRMS $(M+H)^+$ calcd for $C_{13}H_{14}NO$: 200.1075. Found: 200.1082.

General procedure for aerobic dehydrogenation of heterocyclic ketones.



$Pd(OAc)_2$ (13.4mg, 0.06mmol), K_2CO_3 (13.8 mg, 0.1mmol) were weighed in a 2-dram vial. Dry DMF (1 mL) was added, followed by the addition of the ketones (1 mmol). The reaction mixture was stirred at 110 °C for 24 h in oxygen balloon. The progress of the reaction was monitored by TLC and GC. Upon completion, the reaction mixture was diluted with EtOAc (1 mL)/water (4 mL), and separated. The aqueous layer was extracted twice with EtOAc (1 mL). The combined organic phase was washed with water, brine, dried over sodium sulfate, filtered and concentrated. The residue was purified by silica column chromatography (n-hexane/ethyl acetate) to give the desired products.

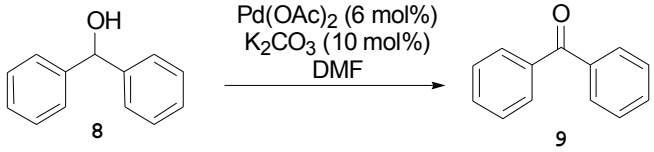
(E)-4-(pyridin-2-yl)but-3-en-2-one: Pale yellow oil. Yield: 61%. ^{1}H NMR (500 MHz, $CDCl_3$, 298 K) δ: 2.41 (s, 3H), 7.14 (d, *J* = 16.0 Hz, 1H), 7.29 (m, 1H), 7.49 (m, 1H), 7.72 (t, *J* = 1.6 Hz, 1H), 8.6 (d, *J* = 4.7 Hz, 1H) ppm. ^{13}C NMR (500MHz, $CDCl_3$, 298 K) δ: 28.1, 124.1, 124.3, 130.1, 136.8, 141.9, 150.1, 153.1, 198.5 ppm. HRMS $(M+H)^+$ calcd for $C_9H_{10}NO$: 148.0762. Found: 148.0762.

(E)-4-(quinolin-2-yl)but-3-en-2-one: White solid. Yield: 95%. m. p. 68-71 °C. ^{1}H NMR (500 MHz, $CDCl_3$, 298 K) δ: 2.37 (s, 3H), 7.05 (d, *J* = 16.4 Hz, 1H), 7.44-7.47 (m, 1H), 7.51 (d, *J* = 8.5 Hz, 1H), 7.62-7.69 (m, 2H), 7.71 (d, *J* = 8.2 Hz, 1H), 8.04(m, 2H) ppm. ^{13}C NMR (500MHz, $CDCl_3$, 298 K) δ: 27.6, 120.1, 127.5,

127.6, 128.1, 129.8, 130.2, 131.8, 136.8, 143.0, 148.3, 153.5, 198.6 ppm. HRMS $(M+H)^+$ calcd for $C_{13}H_{12}NO$: 198.0919. Found: 198.0923.

.

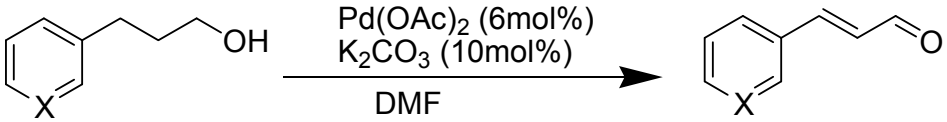
Procedure for aerobic dehydrogenation of diphenylmethanol (8).



$Pd(OAc)_2$ (13.4mg, 0.06mmol), K_2CO_3 (13.8 mg, 0.1mmol) were weighed in a 2-dram vial. Dry DMF (1 mL) was added, followed by the addition of diphenylmethanol **8** (1 mmol). The reaction mixture was stirred at room temperature for 24 h. The progress of the reaction was monitored by TLC and GC. Upon completion, the reaction mixture was diluted with EtOAc (1 mL)/water (4 mL), and separated. The aqueous layer was extracted twice with EtOAc (1 mL). The combined organic phase was washed with water, brine, dried over sodium sulfate, filtered and concentrated. The residue was purified by silica column chromatography (n-hexane/ethyl acetate) to give the desired ketone **9**.

Diphenylmethanone 9: White solid. Yield: 99%. m. p. 48-51 ^oC (lit. m.p. 48 ^oC).[S15] ^1H NMR (500 MHz, $CDCl_3$, 298 K) δ: 7.47 (t, J = 7.8 Hz, 2H), 7.57 (d, J = 7.4 Hz, 1H), 7.81 (m, 2H) ppm. ^{13}C NMR (500MHz, $CDCl_3$, 298 K) δ: 128.4, 130.2, 132.6, 137.8, 196.9 ppm.

Procedure for double aerobic dehydrogenation of phenylpropanol (10).



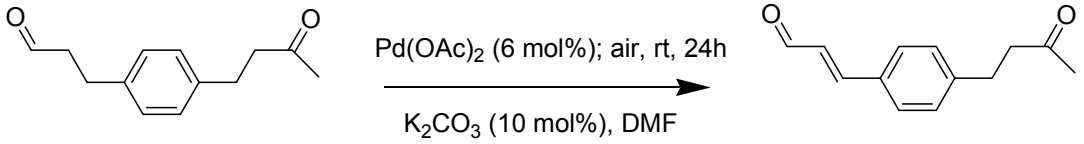
X= C, N **10**, **11** X= C, N **2a**, **12**

$Pd(OAc)_2$ (13.4mg, 0.06mmol), K_2CO_3 (13.8 mg, 0.1mmol) and 4,5-diazafluorenone (16.4mg, 0.09mmol) were weighed in a 2-dram vial. Dry DMF (1 mL) was added, followed by the addition of 3-phenyl-propan-1-ol **10** (1 mmol). The reaction mixture was stirred at room temperature for 24 h. The progress of the reaction was monitored by TLC and GC. Upon completion, the reaction mixture was diluted with EtOAc (1 mL)/water (4 mL), and separated. The aqueous layer was extracted twice with EtOAc (1 mL). The combined organic phase was washed with water, brine, dried over sodium sulfate, filtered and concentrated. The residue was purified by

silica column chromatography (n-hexane/ethyl acetate) to give the desired unsaturated aldehyde **2a**. The compound **11** was done as the same way but at a temperature of 80 °C to give product **12**.

(E)-3-(pyridin-3-yl)acrylaldehyde: White solid. Yield: 67%. m. p. 63-66 °C. ^{1}H NMR (500 MHz, $CDCl_3$, 298 K) δ: 6.80 (dd, *J* = 16.4, 7.6 Hz, 1H), 7.29 (m, 1H), 7.40 (t, *J* = 3.2 Hz, 1H), 7.51 (d, *J* = 16.4 Hz, 1H), 7.90 (t, *J* = 6.0 Hz, 1H), 8.67 (q, *J* = 1.6 Hz, 1H), 8.80 (d, *J* = 2.0 Hz, 1H), 9.75 (d, *J* = 7.6 Hz, 1H) ppm. ^{13}C NMR (500MHz, $CDCl_3$, 298 K) δ: 123.9, 129.8, 130.2, 134.4, 148.4, 150.0, 151.8, 192.9 ppm. HRMS $(M+H)^+$ calcd for C_8H_8NO: 134.0606. Found: 134.0605.

Procedure for selective aerobic dehydrogenation of 13.



13 **14**

$Pd(OAc)_2$ (13.4mg, 0.06mmol), K_2CO_3 (13.8 mg, 0.1mmol) and 4,5-diazafluorenone (16.4mg, 0.09mmol) were weighed in a 2-dram vial. Dry DMF (1 mL) was added, followed by the addition of 3-[4-(3-Oxo-butyl)-phenyl]-propionaldehyde **13** (1 mmol).[S16] The reaction mixture was stirred at room temperature for 24h. The progress of the reaction was monitored by TLC and GC. Upon completion, the reaction mixture was diluted with EtOAc (1 mL)/water (4 mL), and separated. The aqueous layer was extracted twice with EtOAc (1 mL). The combined organic phase was washed with water, brine, dried over sodium sulfate, filtered and concentrated. The residue was purified by silica column chromatography (n-hexane/ethyl acetate) to give the desired unsaturated aldehyde **14**.

(*E*)-3-(4-(3-oxobutyl)phenyl)acrylaldehyde 14: Pale yellow oil. Yield: 87%. ^{1}H NMR (500 MHz, $CDCl_3$, 298 K) δ: 2.16 (s, 3H), 2.78 (t, *J* = 7.6 Hz, 2H), 2.94 (t, *J* = 7.6 Hz, 2H), 6.69 (dd, *J* = 7.7, 15.9 Hz, 1H), 7.1 (s, 1H), 7.25 (s, 1H), 7.43-7.50 (m, 2H), 9.69 (d, *J* = 7.8 Hz, 1H) ppm. ^{13}C NMR (500MHz, $CDCl_3$, 298 K) δ: 29.7, 30.1, 44.7, 128,3, 128.8, 129.3, 132.3, 152.5, 193.7, 207.2 ppm. HRMS $(M+H)^+$ calcd for $C_{13}H_{15}O_2$: 203.1072. Found: 203.1069.

Ref:

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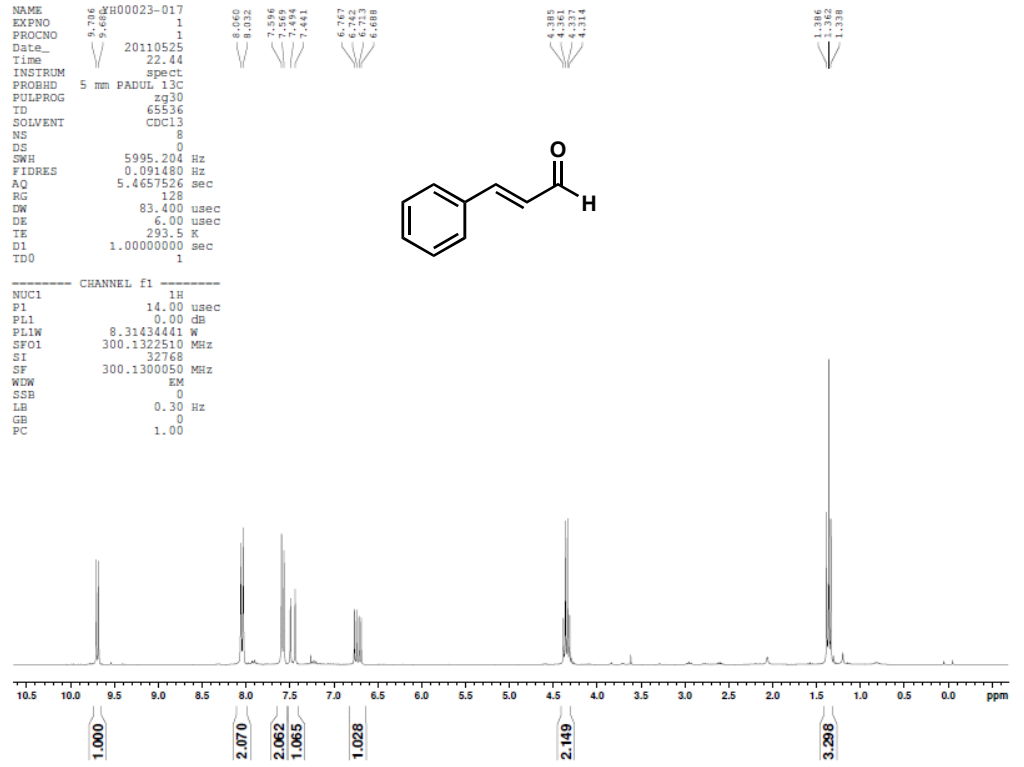
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TD 65536
SOLVENT CDCl3
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DS 0
SWH 5995.204 Hz
FIDRES 0.091480 Hz
AQ 5.4657526 sec
RG 128
DW 83.400 usec
DE 6.00 usec
TE 293.5 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 14.00 usec
PL1 0.00 dB
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SFO1 300.1322510 MHz
SI 32768
SF 300.1300050 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
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9.688
8.060
8.032
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7.568
7.494
7.441
6.767
6.742
6.723
6.688
4.385
4.361
4.337
4.314
1.386
1.362
1.338
O
H
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
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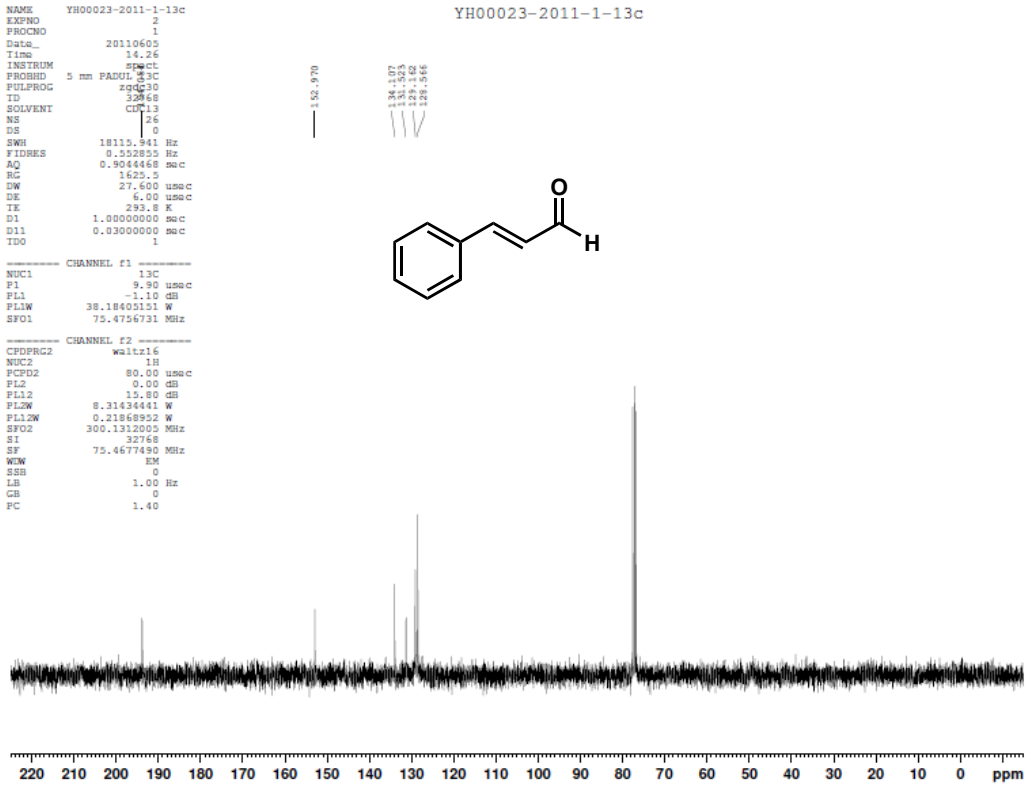
2.062
1.065
1.028
2.149
3.298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Time 14.26
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PULPROG zgpg30
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SOLVENT CDCl3
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DS 0
SWH 18115.941 Hz
FIDRES 0.552855 Hz
AQ 0.9044468 sec
RG 1625.5
DW 27.600 usec
DE 6.00 usec
TE 293.8 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 9.90 usec
PL1 -1.10 dB
PL1W 38.18405151 W
SFO1 75.4756731 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 15.80 dB
PL2W 8.31434441 W
PL12W 0.21868952 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
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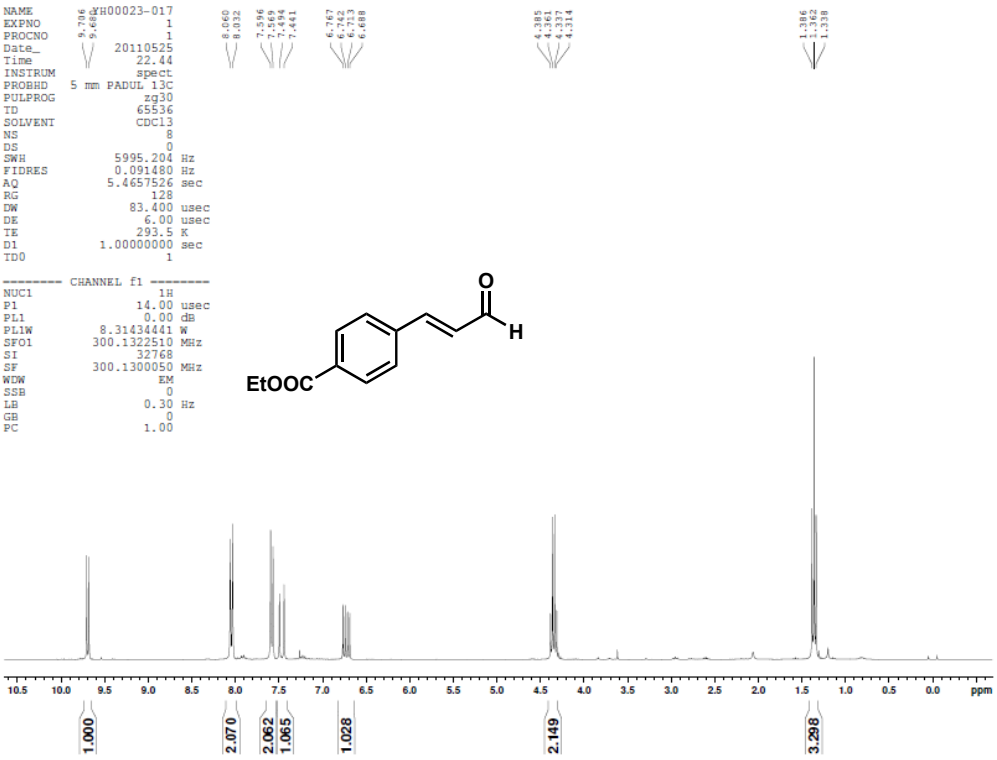
134.107
131.523
128.952
128.566
O
H
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

2b: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PROCNO 1
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PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 5995.204 Hz
FIDRES 0.091480 Hz
AQ 5.4657526 sec
RG 128
DW 83.400 usec
DE 6.00 usec
TE 293.5 K
D1 1.00000000 sec
TD0 1
NUC1 1H
P1 14.00 usec
PL1 0.00 dB
PL1W 8.31434441 W
SFO1 300.1322510 MHz
SI 32768
SF 300.1300050 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
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8.032
7.596
7.569
7.494
7.441
6.767
6.742
6.713
6.688
6.385
4.361
4.337
4.314
1.386
1.362
1.338
O
H
EtOOC
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
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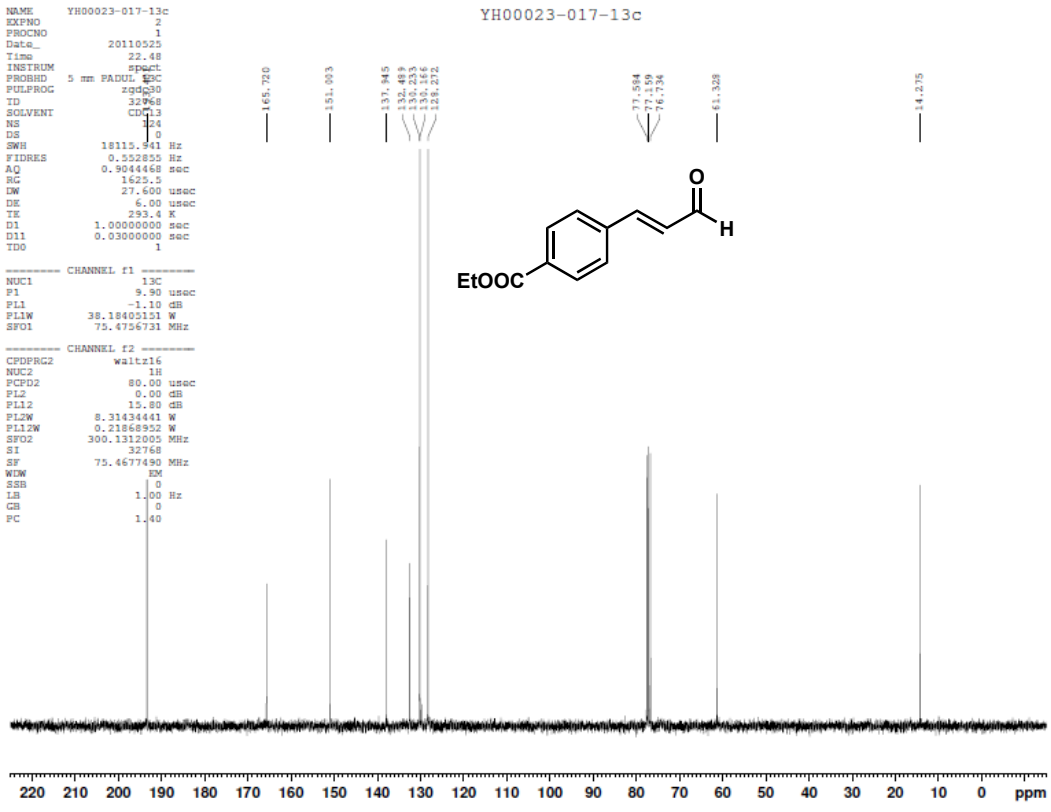
2.070
2.062
1.065
1.028
2.149
3.298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SOLVENT CDCl3
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DS 0
SWH 18115.941 Hz
FIDRES 0.552855 Hz
AQ 0.9044468 sec
RG 1625.5
DW 27.600 usec
DE 6.00 usec
TE 293.4 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1
NUC1 13C
P1 9.90 usec
PL1 -1.10 dB
PL1W 38.18405151 W
SFO1 75.4756731 MHz
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 15.80 dB
PL2W 8.31434441 W
PL12W 0.21868952 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
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151.003
137.945
132.483
130.253
130.166
128.272
77.584
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76.734
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14.275
O
H
EtOOC
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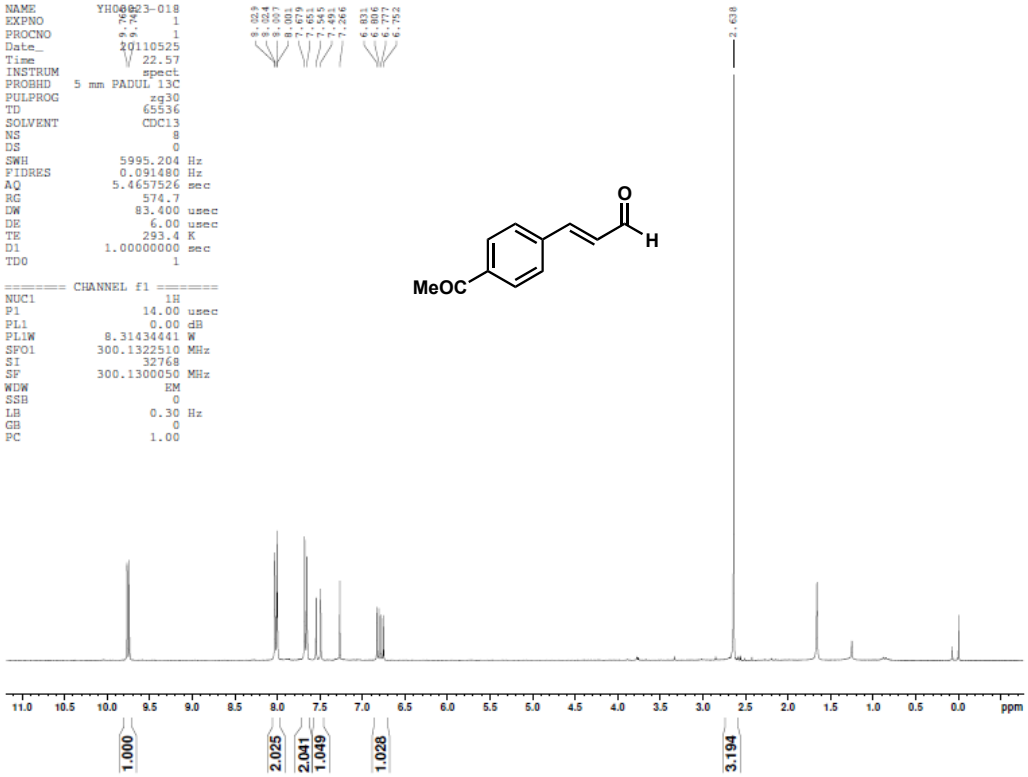
2c: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TD 65536
SOLVENT CDCl3
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FIDRES 0.091480 Hz
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RG 574.7
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DE 6.00 usec
TE 293.4 K
D1 1.00000000 sec
TD0 1
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PL1 0.00 dB
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WDW EM
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7.651
7.545
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7.486
6.831
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6.777
6.752
2.638
O
H
MeOC
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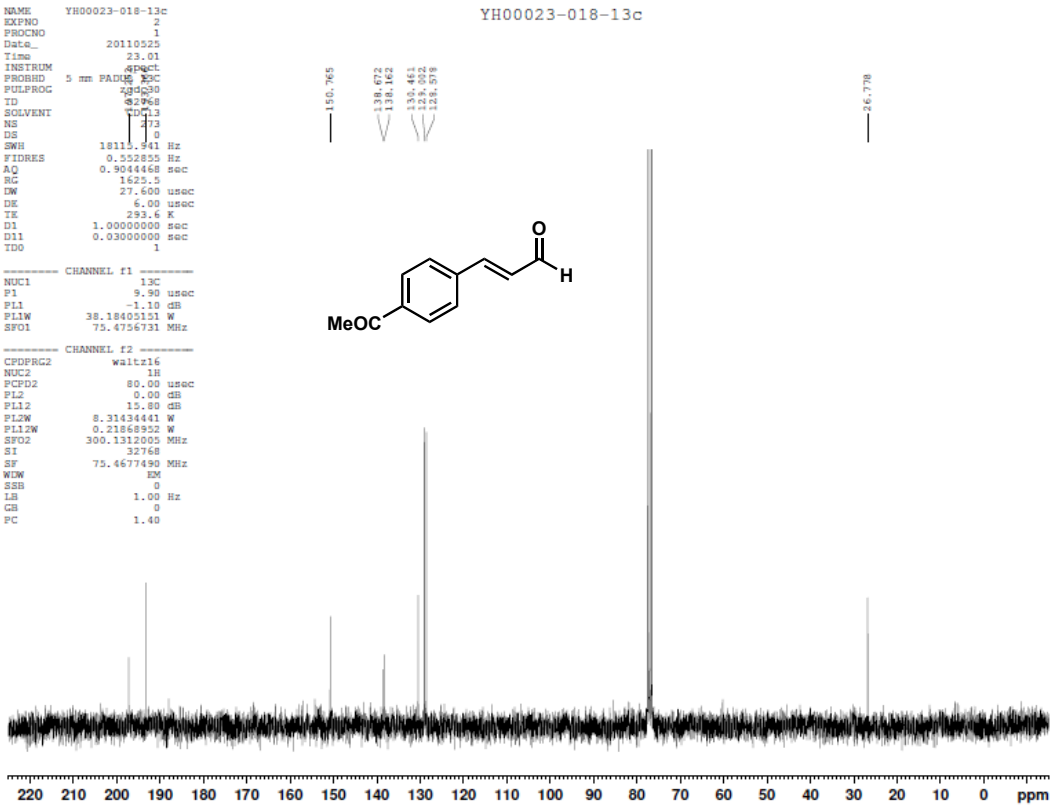
3.194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FIDRES 0.552855 Hz
AQ 0.9044468 sec
RG 1625.5
DW 27.600 usec
DE 6.00 usec
TE 293.6 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1
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P1 9.90 usec
PL1 -1.10 dB
PL1W 38.18405151 W
SFO1 75.4756731 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 15.80 dB
PL2W 8.31434441 W
PL12W 0.21868952 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
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138.672
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26.778
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H
MeOC
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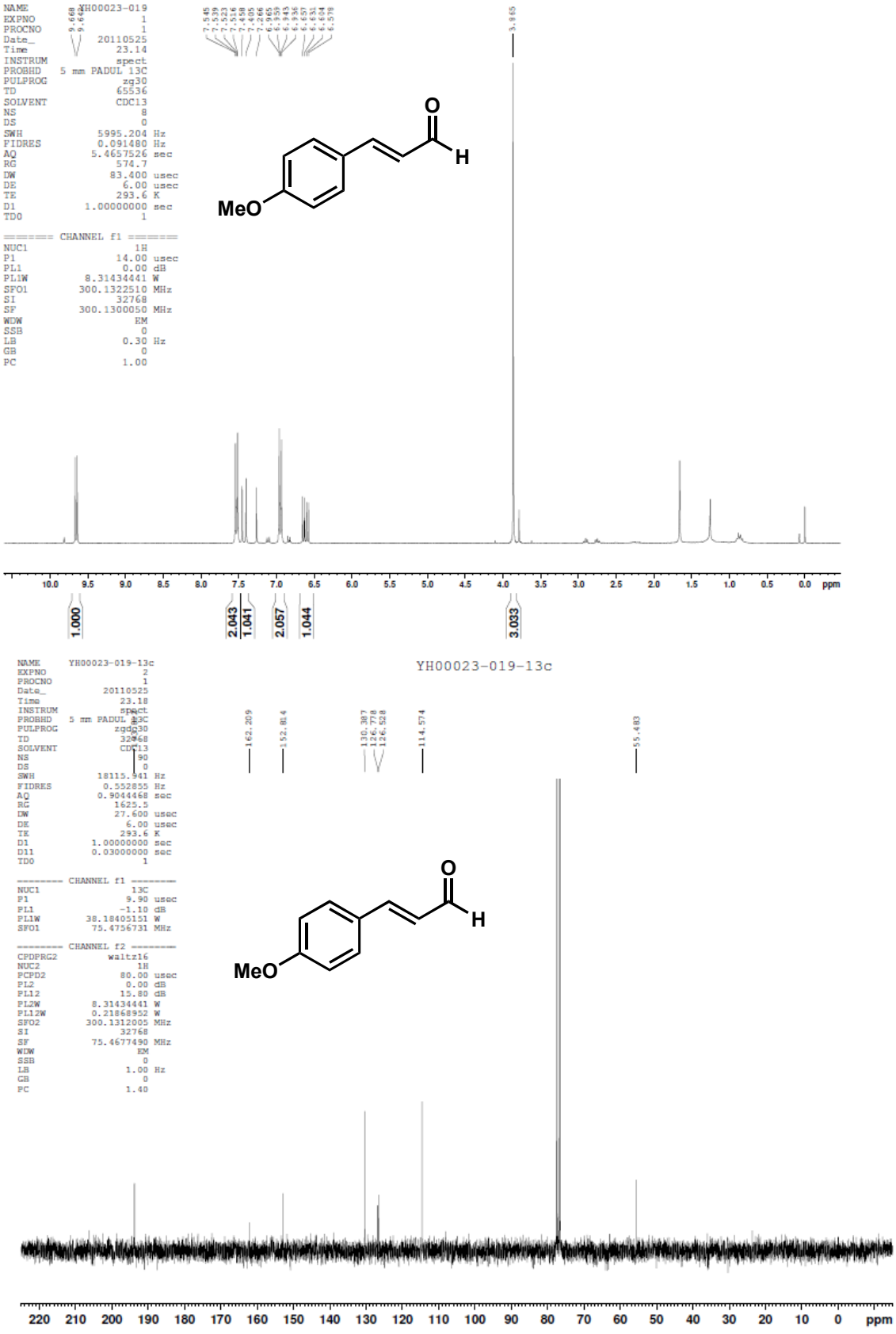
2d:



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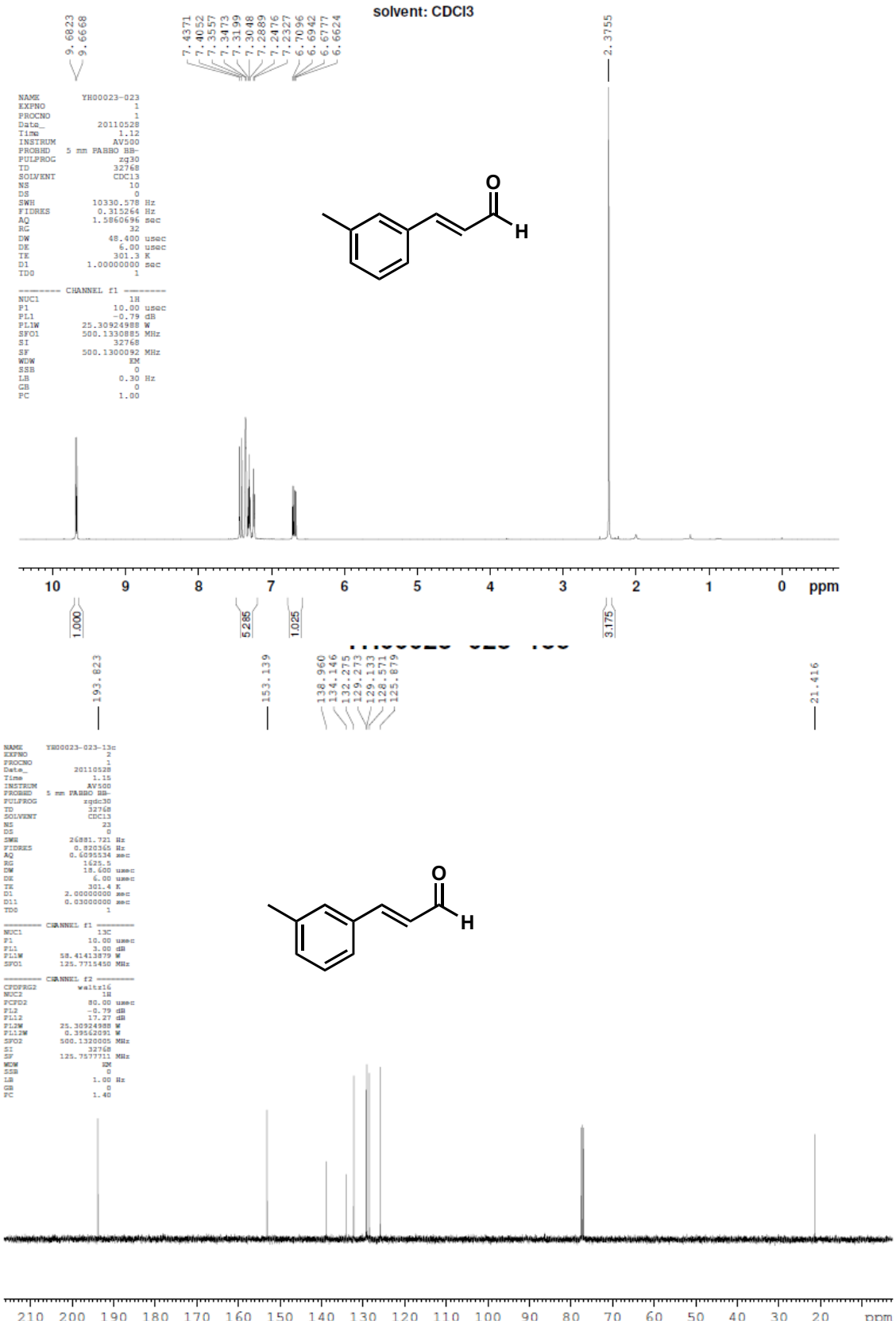
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7.4052
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7.3199
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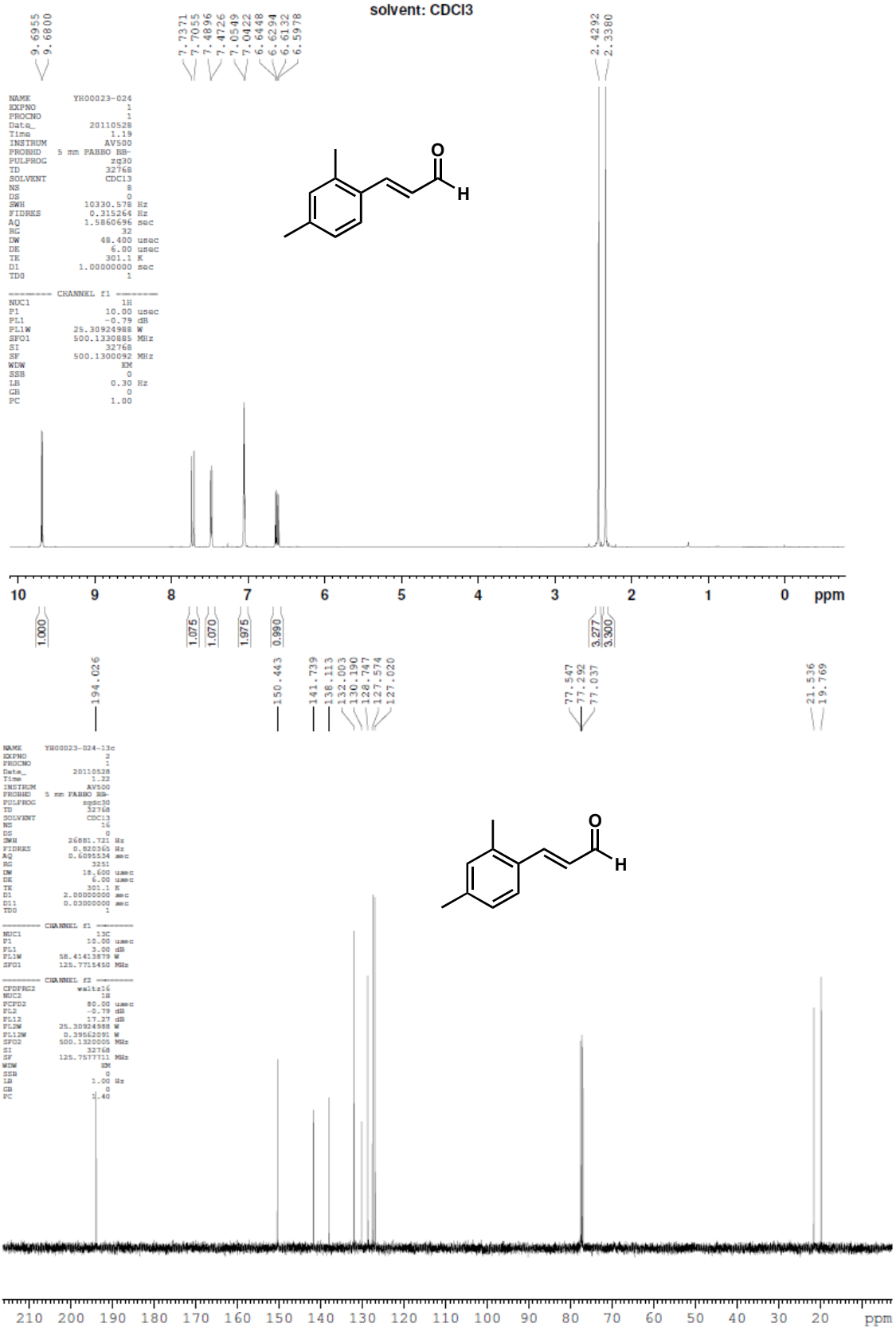
7.2327
6.7096
6.6942
6.6777
6.6624
2.3755
O
H
10
9
8
7
6
5
4
3
2
1
0
ppm
1.000
5.286
1.025
3.175
193.823
153.139
138.960
134.146
132.275
129.273
129.133
128.571
125.879
21.416
O
H
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

2f: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6.6132
6.5978
2.4292
2.3380
O
H
10
9
8
7
6
5
4
3
2
1
0
ppm
1.000
1.075
1.070
1.975
0.990
3.277
3.300
194.026
150.443
141.739
138.113
132.003
130.190
128.747
127.574
127.020
77.547
77.292
77.037
21.536
19.769
O
H
210
200
190
180
170
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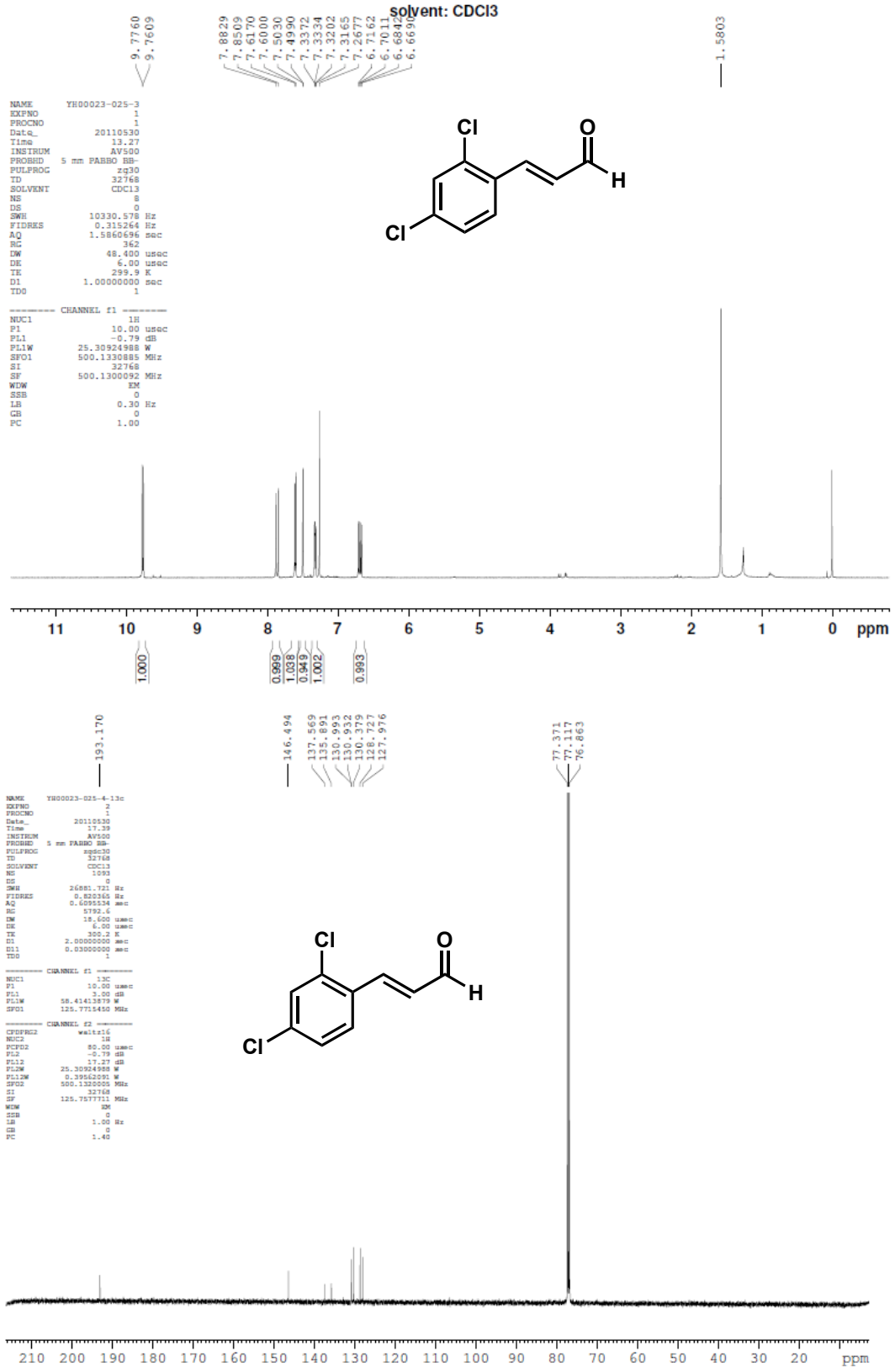
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

2g:



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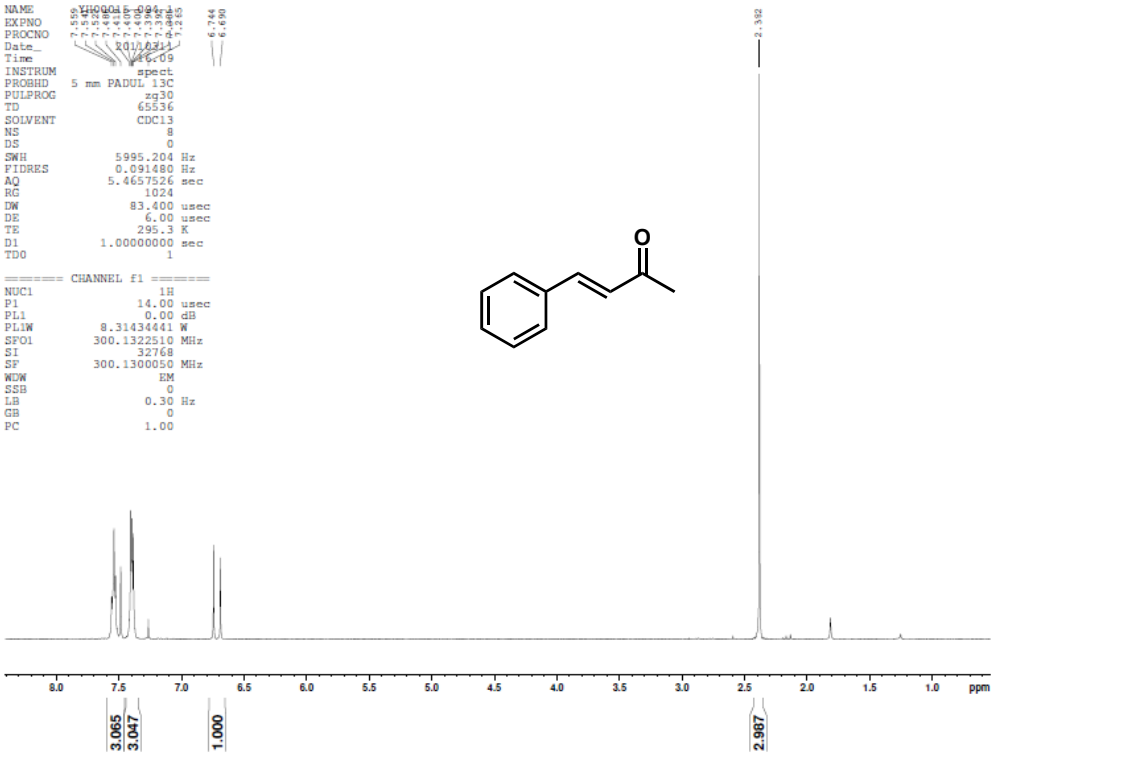
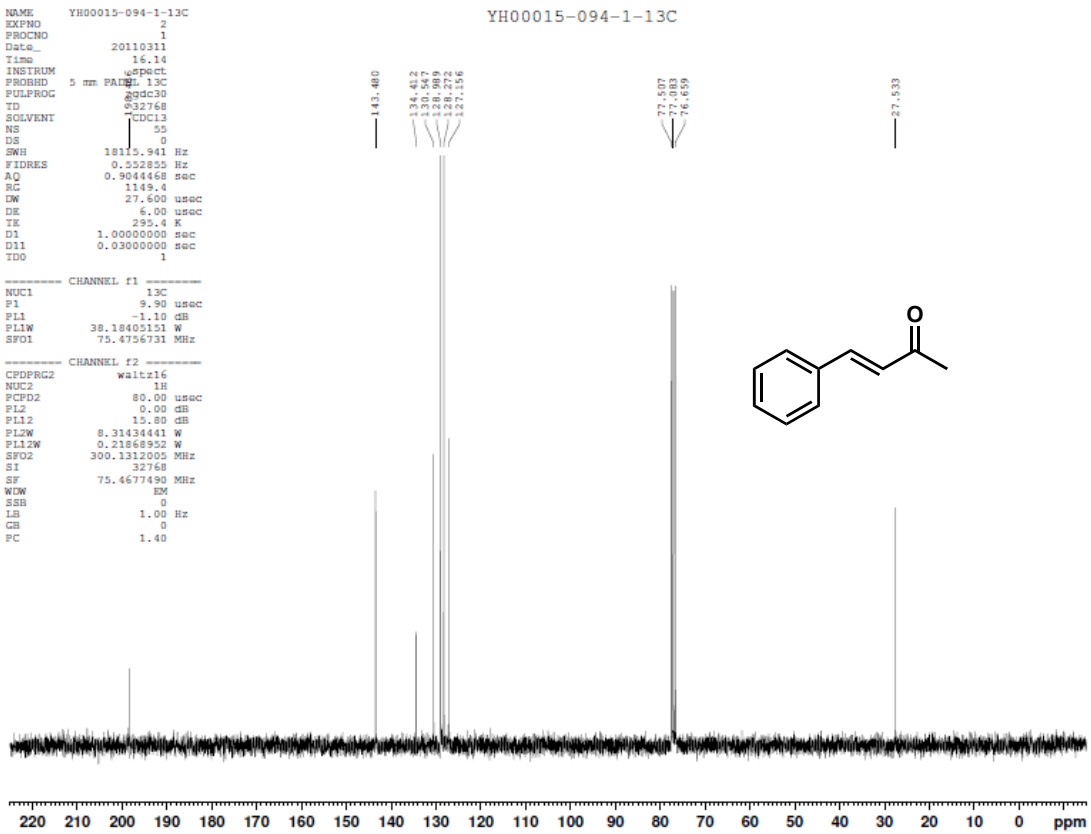
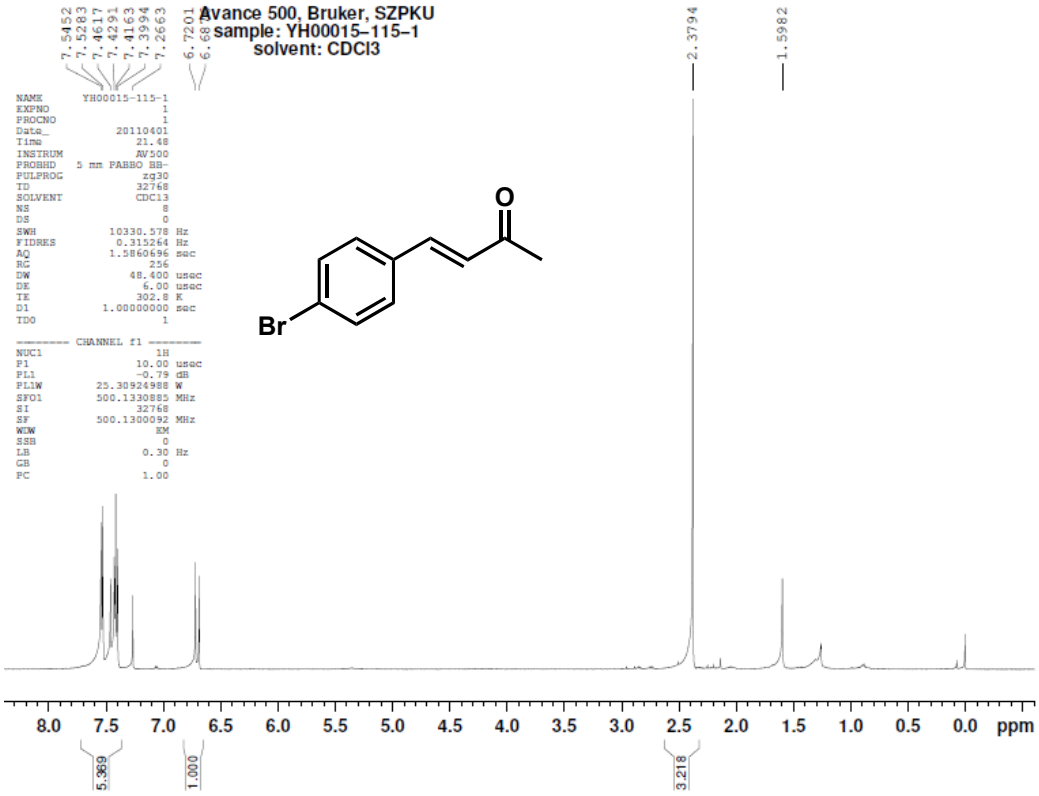
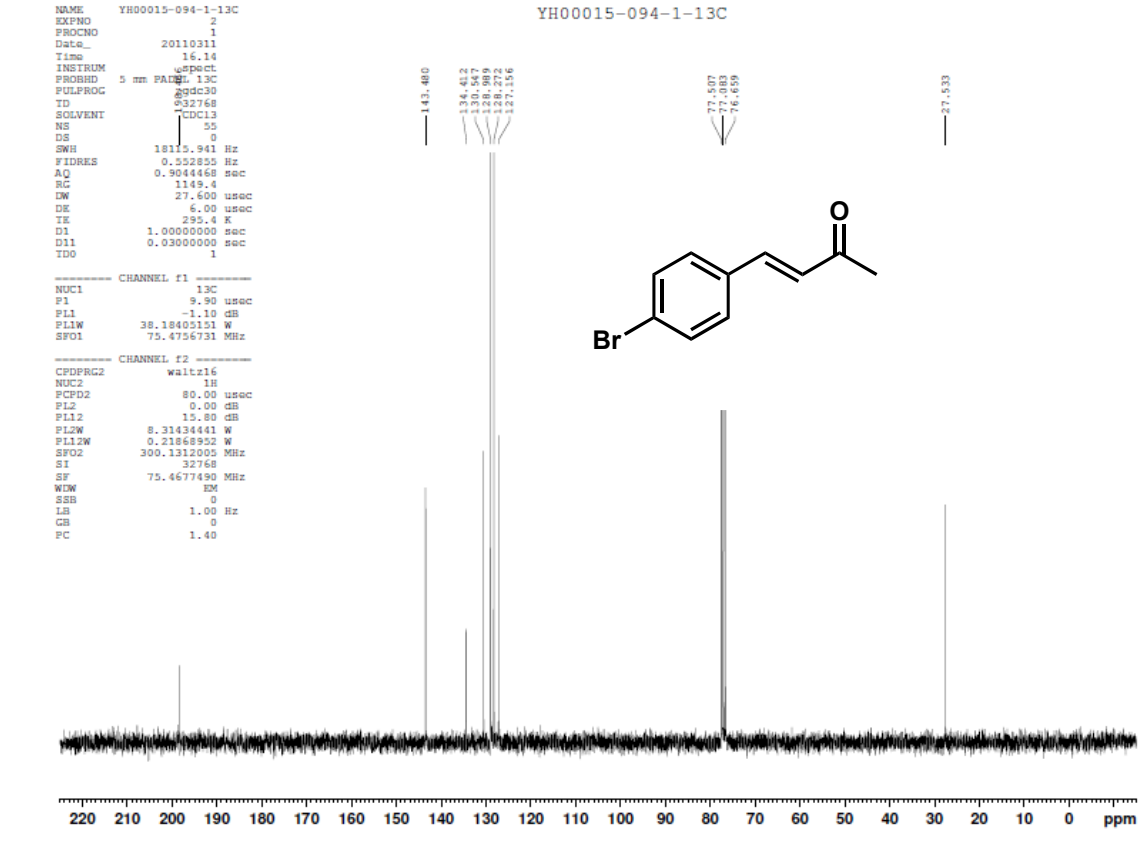


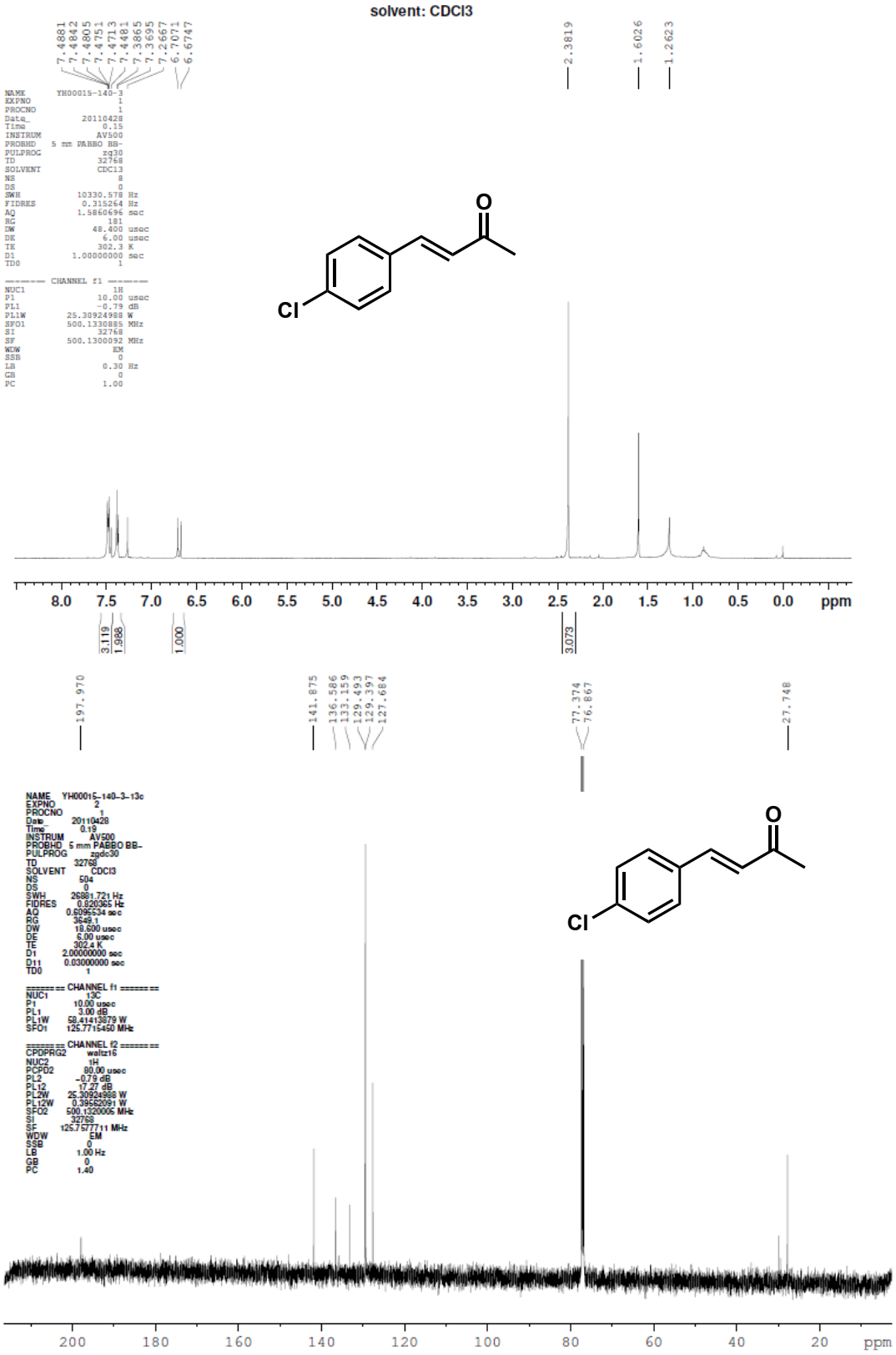


4b:

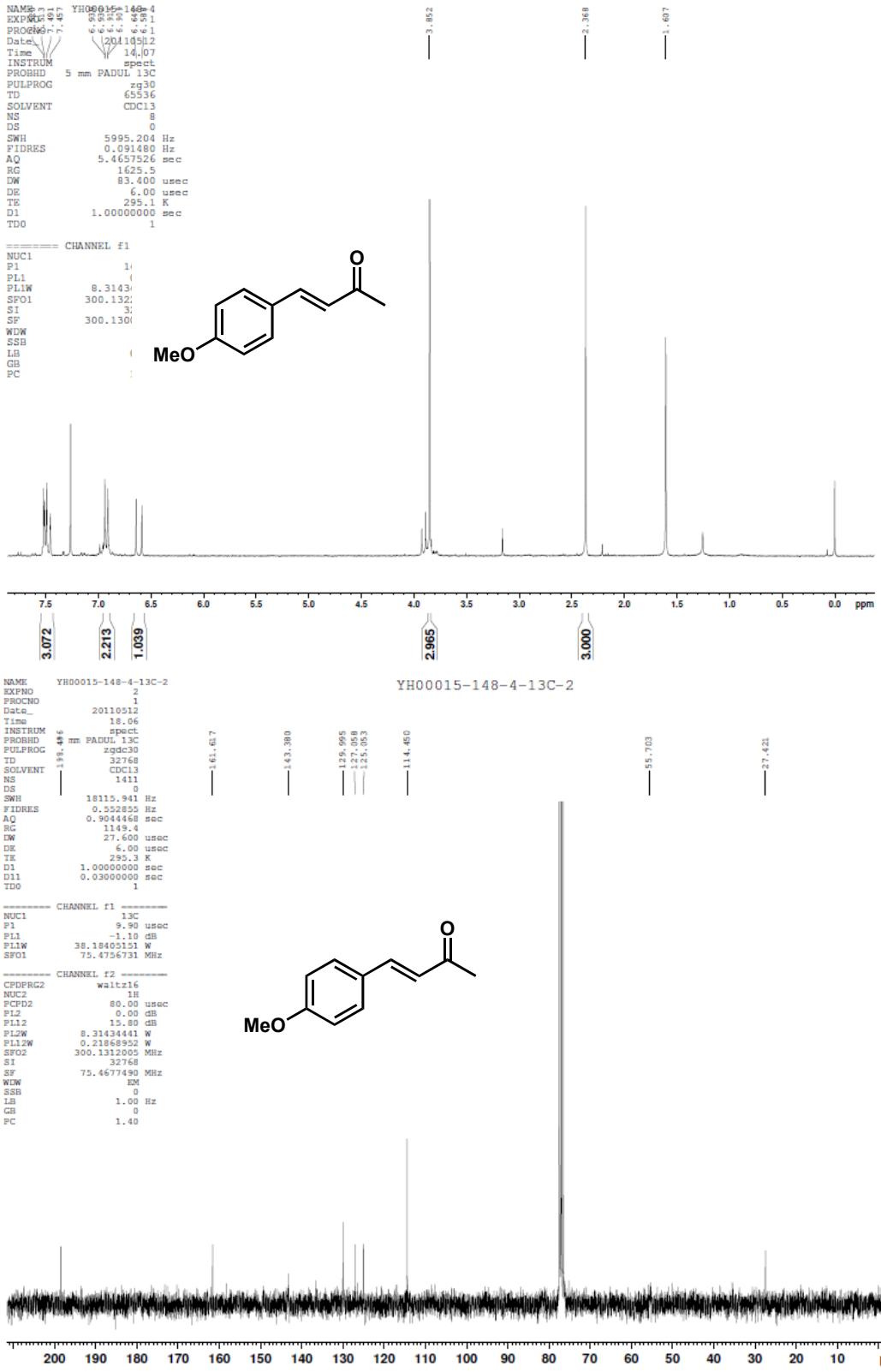




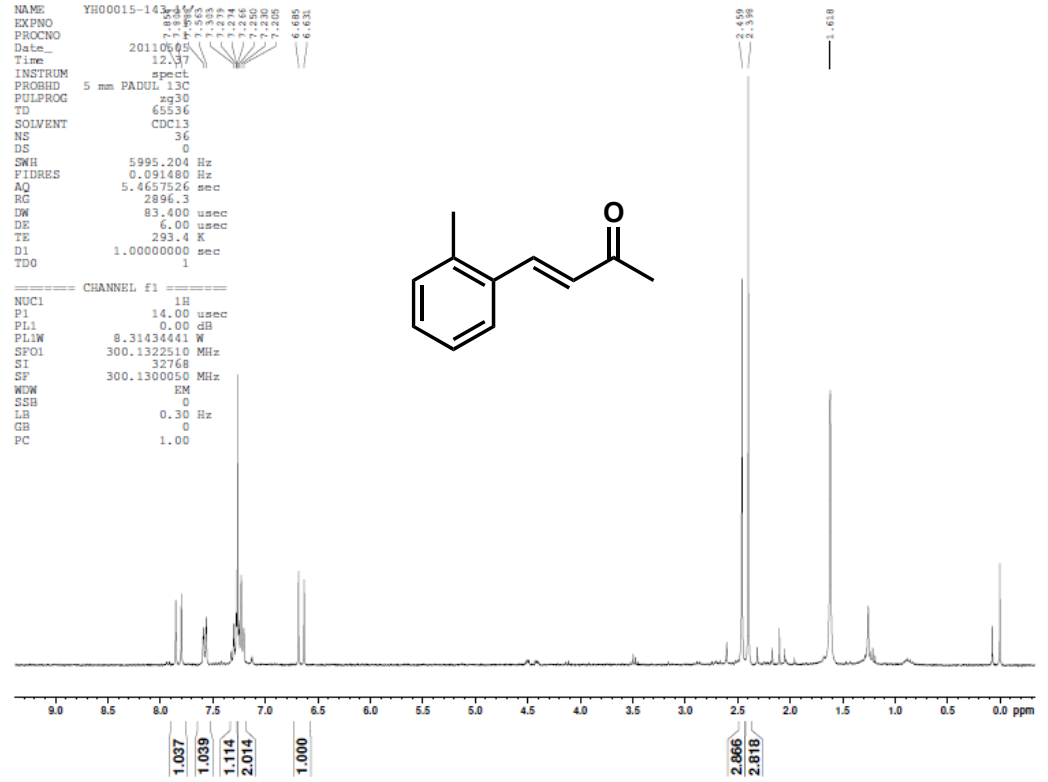
4c:

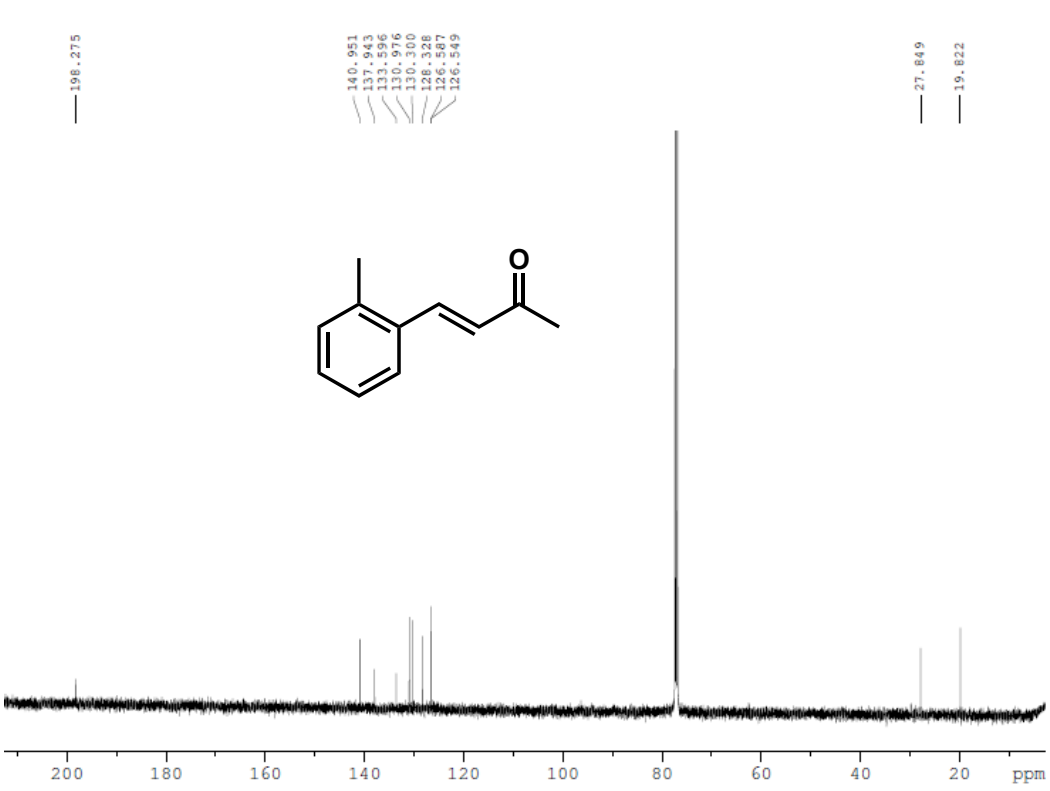


4d:


MeO
O
3.852
2.368
1.697
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
3.072
2.213
1.039
2.965
3.000
YH00015-148-4-13C-2
MeO
O
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

4e:

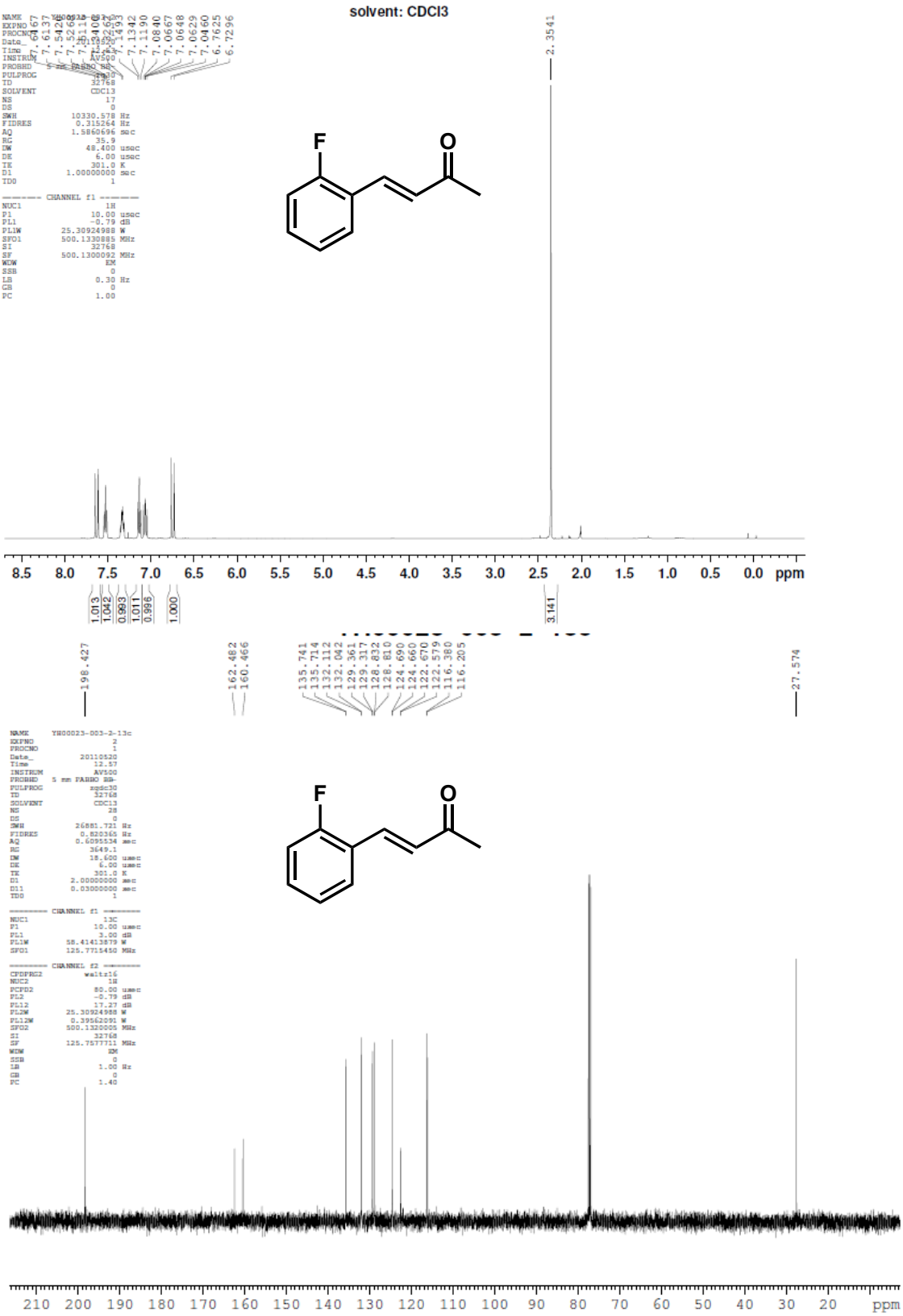




4f:

4g:

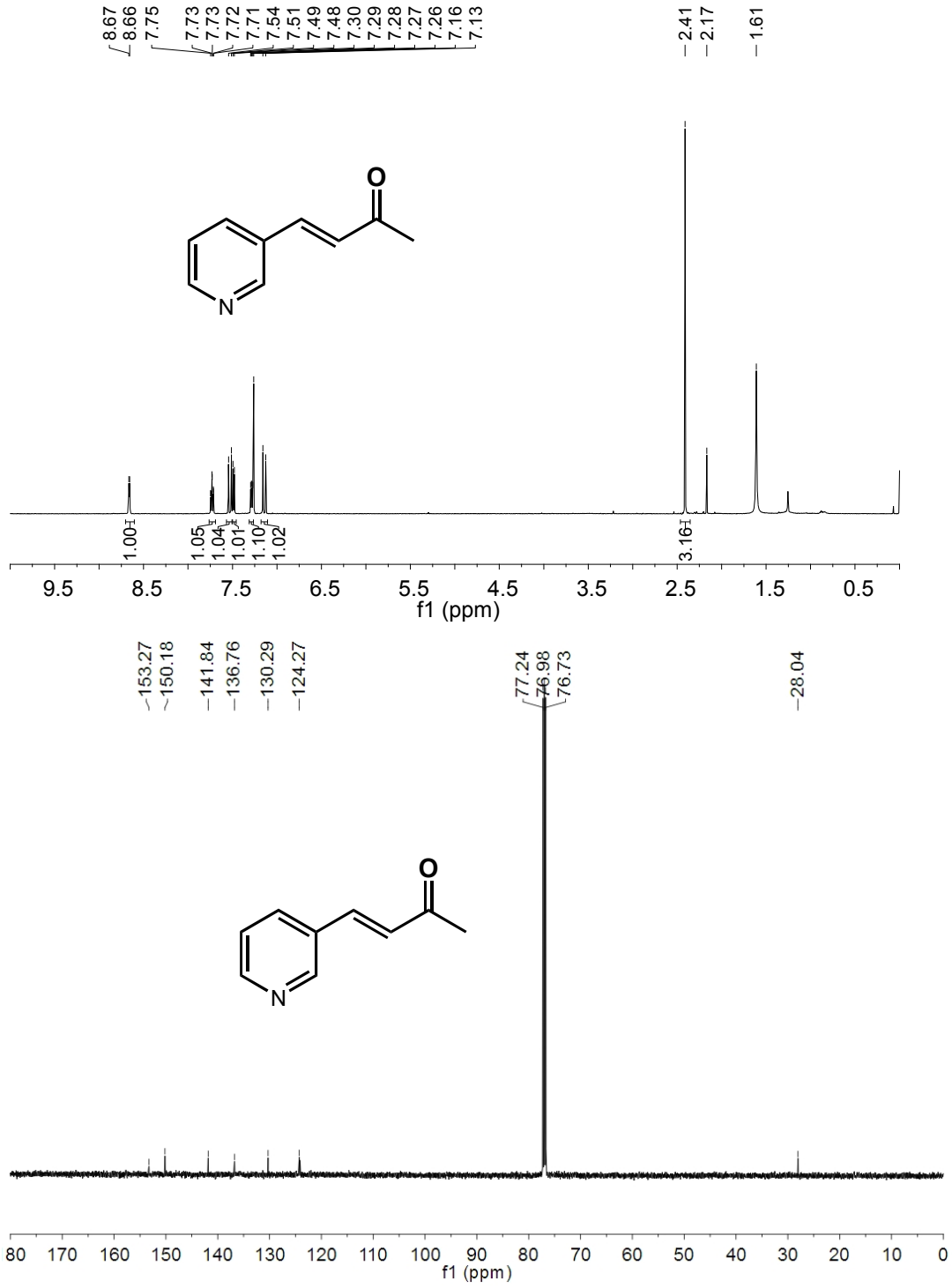
4h:


solvent: CDCl3
F
O
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
198.427
162.482
160.466
135.741
135.714
132.112
132.042
129.361
129.317
128.832
128.810
124.690
124.660
122.670
122.579
116.380
116.205
27.574
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

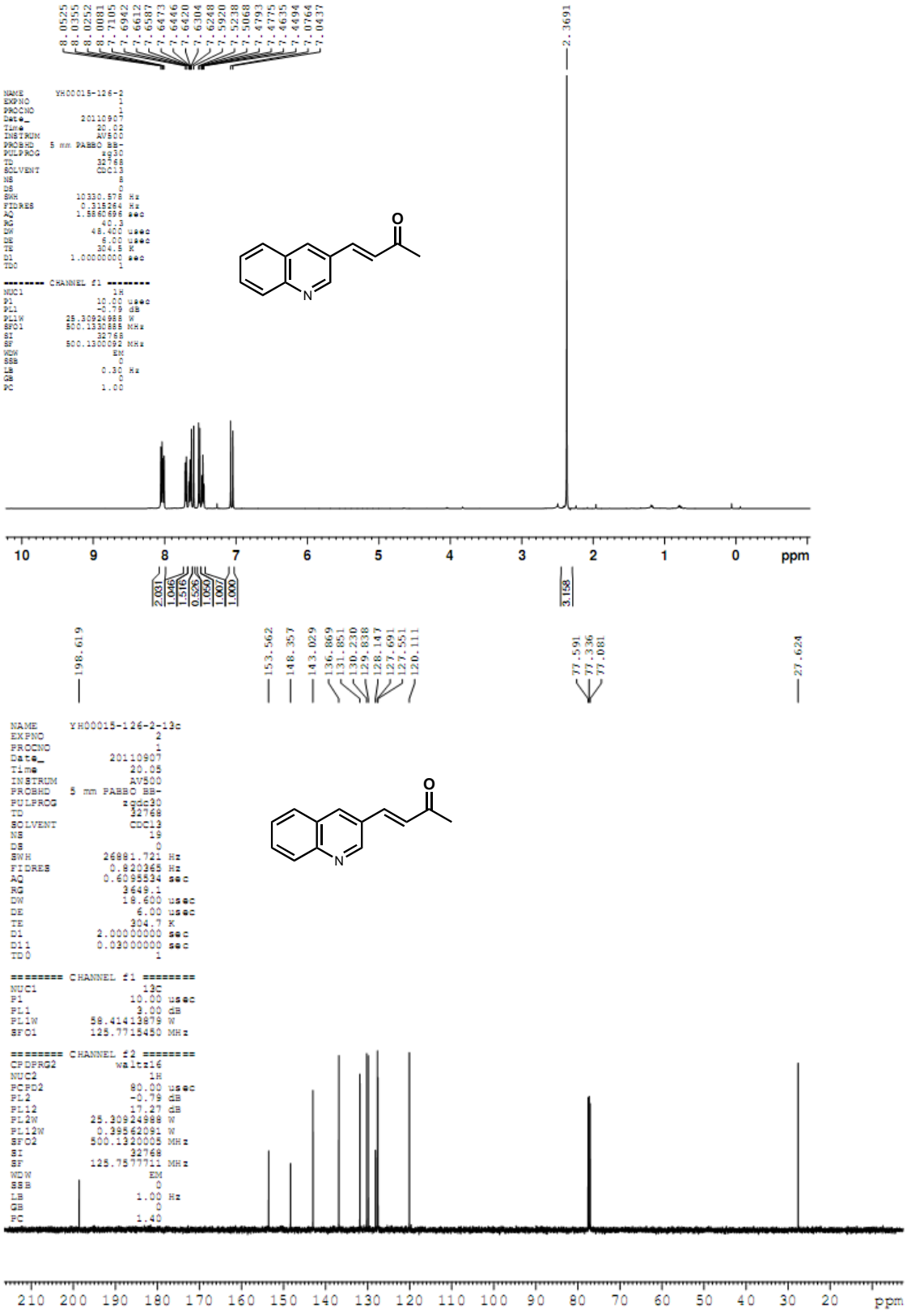
4i:


solvent: CDCl3
2.4322
1.6398
ppm
198.473
143.559
134.516
133.485
132.147
130.369
128.933
128.704
127.952
127.503
127.467
126.922
123.704
27.719
ppm

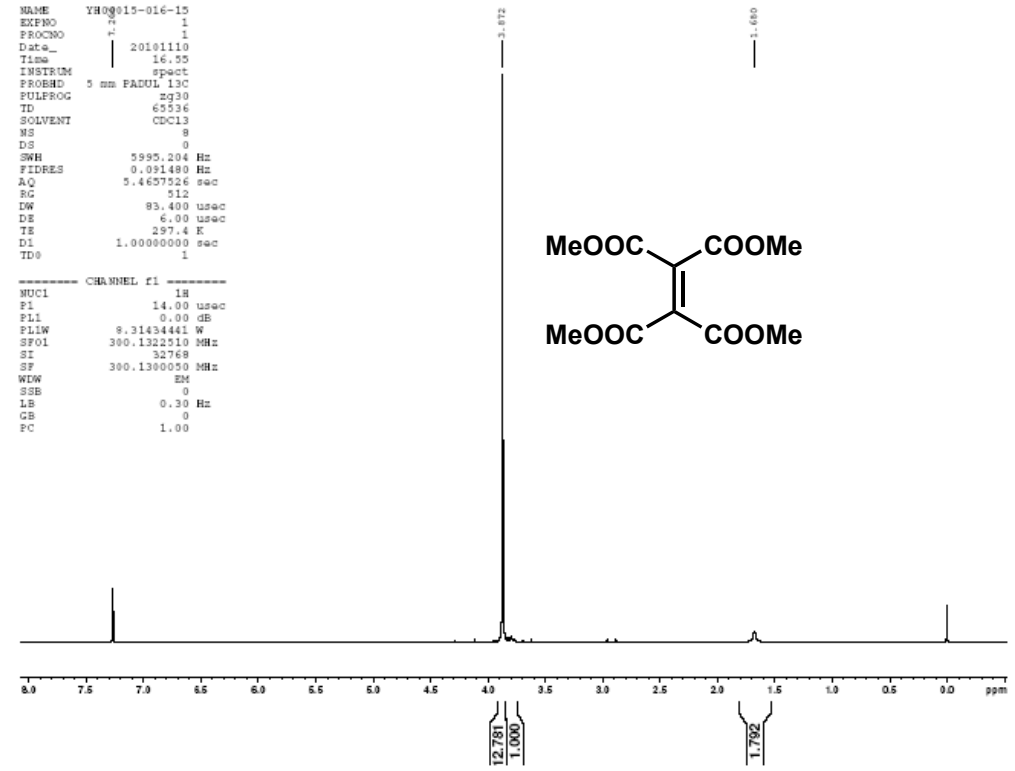
4j:

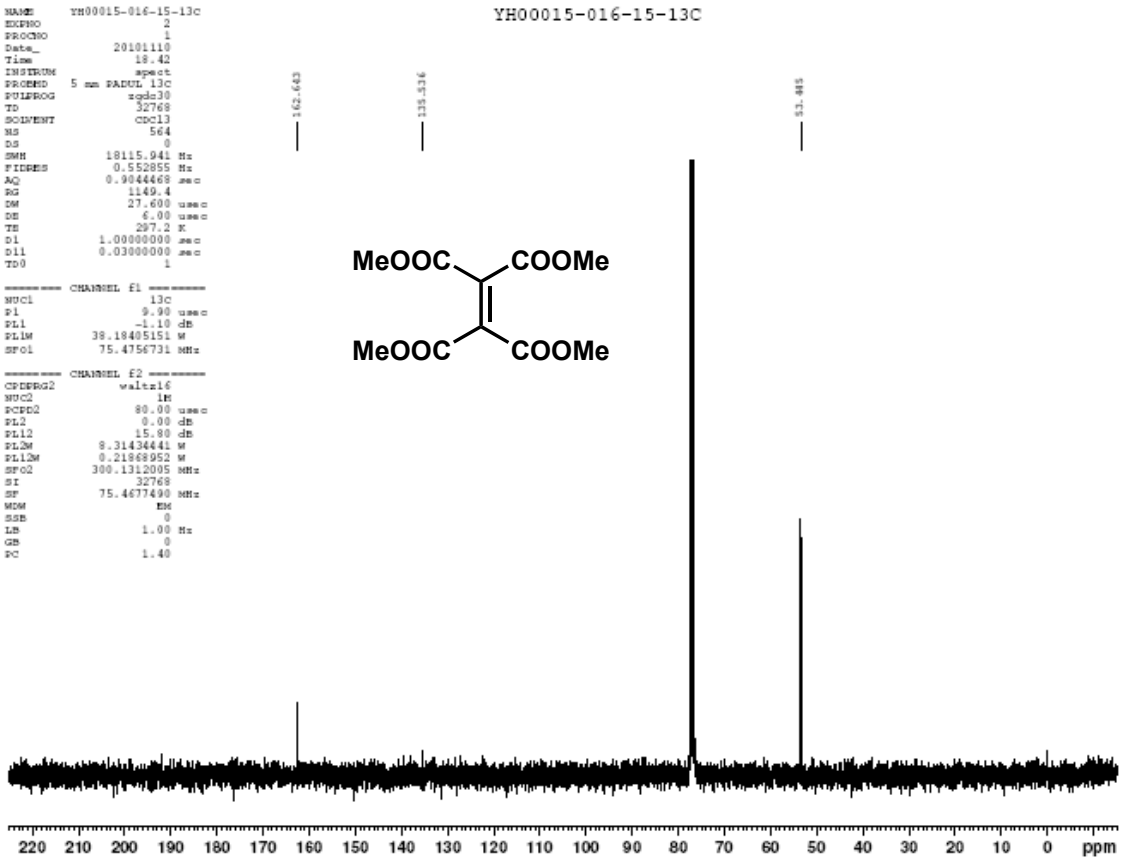


4k:

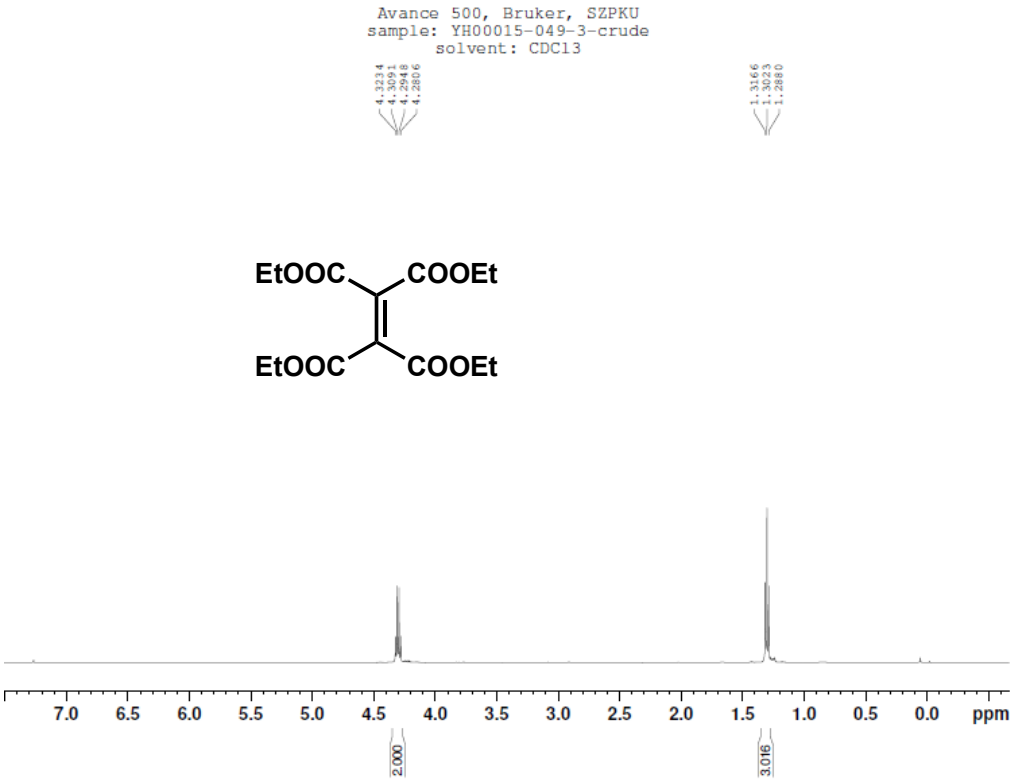


5a:

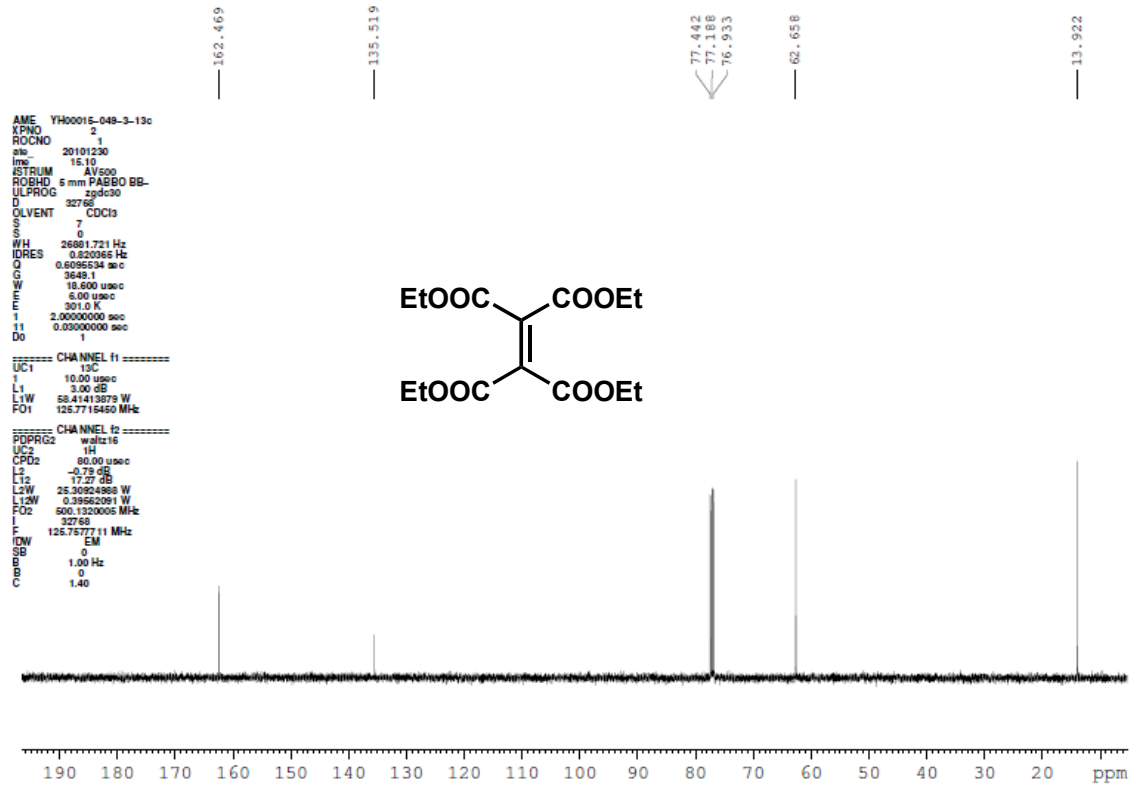

MeOOC
COOMe
MeOOC
COOMe
3.872
1.660
12.781
1.000
1.792
ppm


YH00015-016-15-13C
MeOOC
COOMe
MeOOC
COOMe
162.643
135.536
53.485
ppm

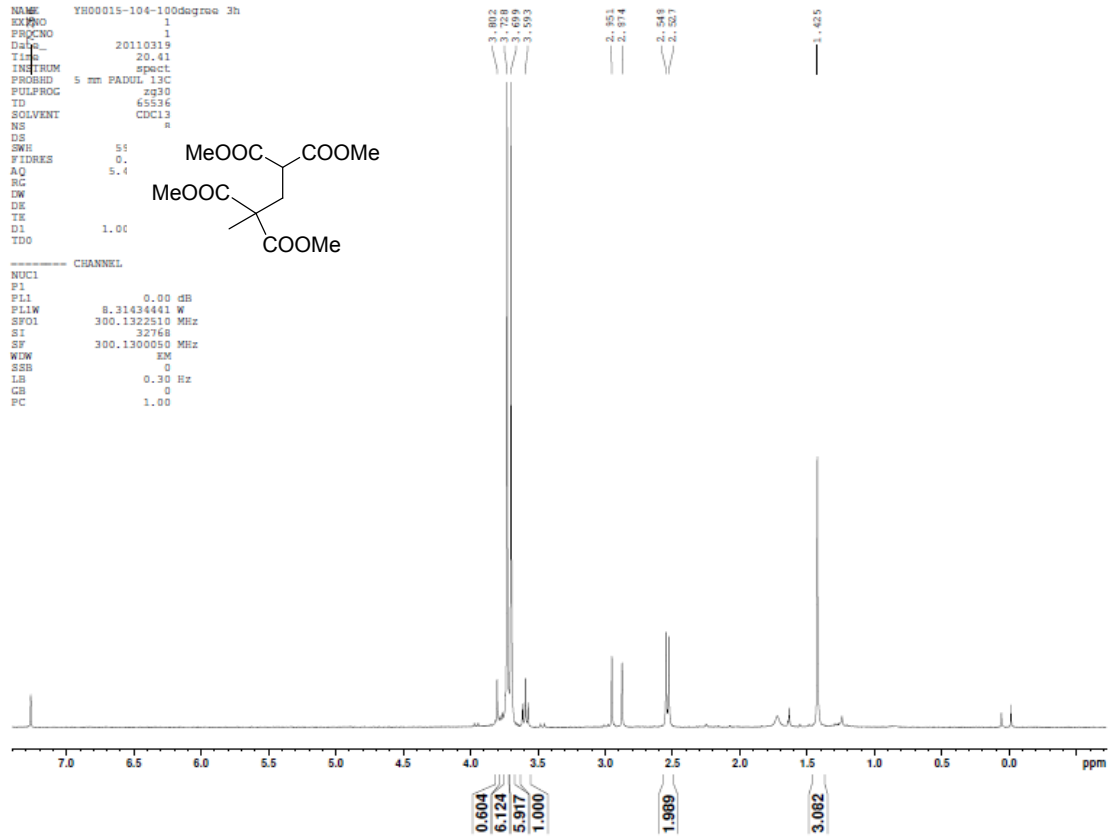
5b:

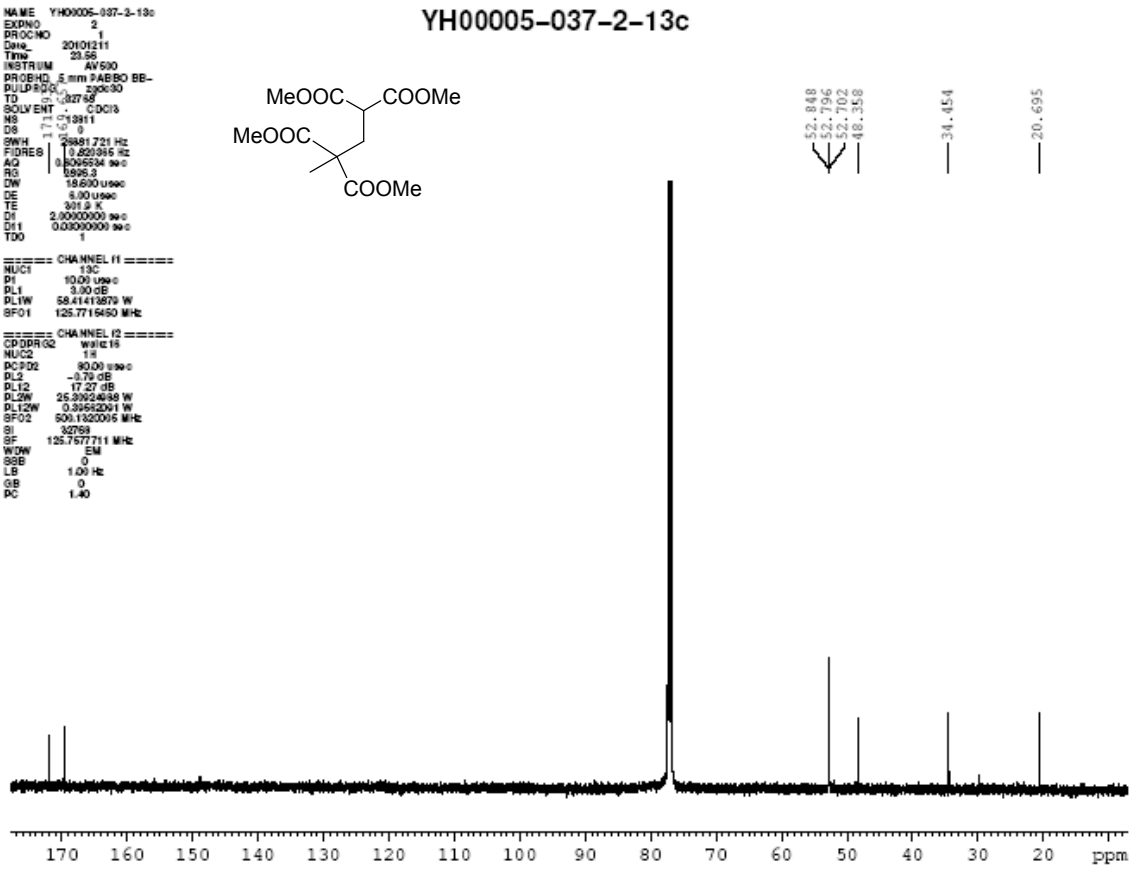


YH00015–049–3–13c

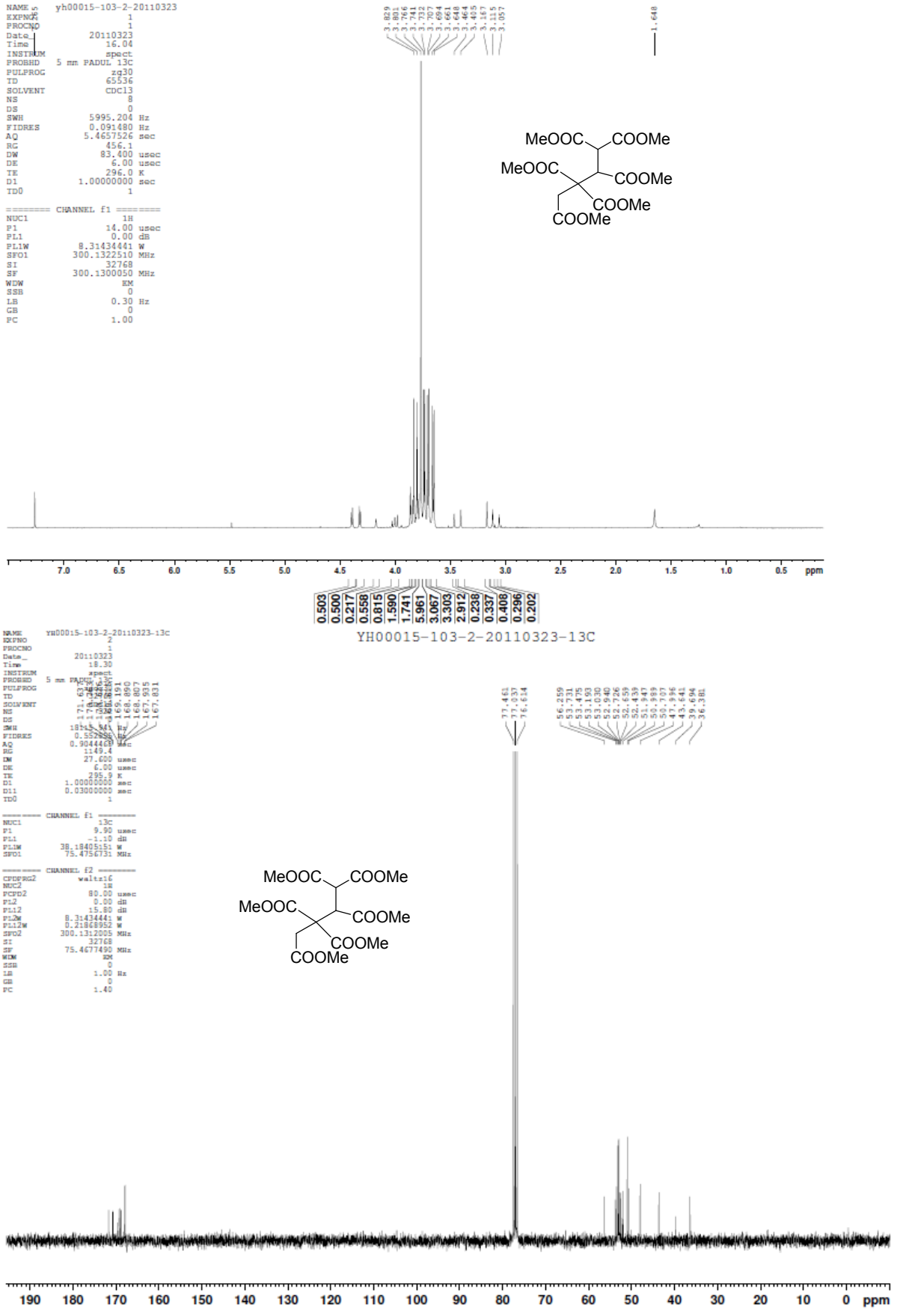


5c:

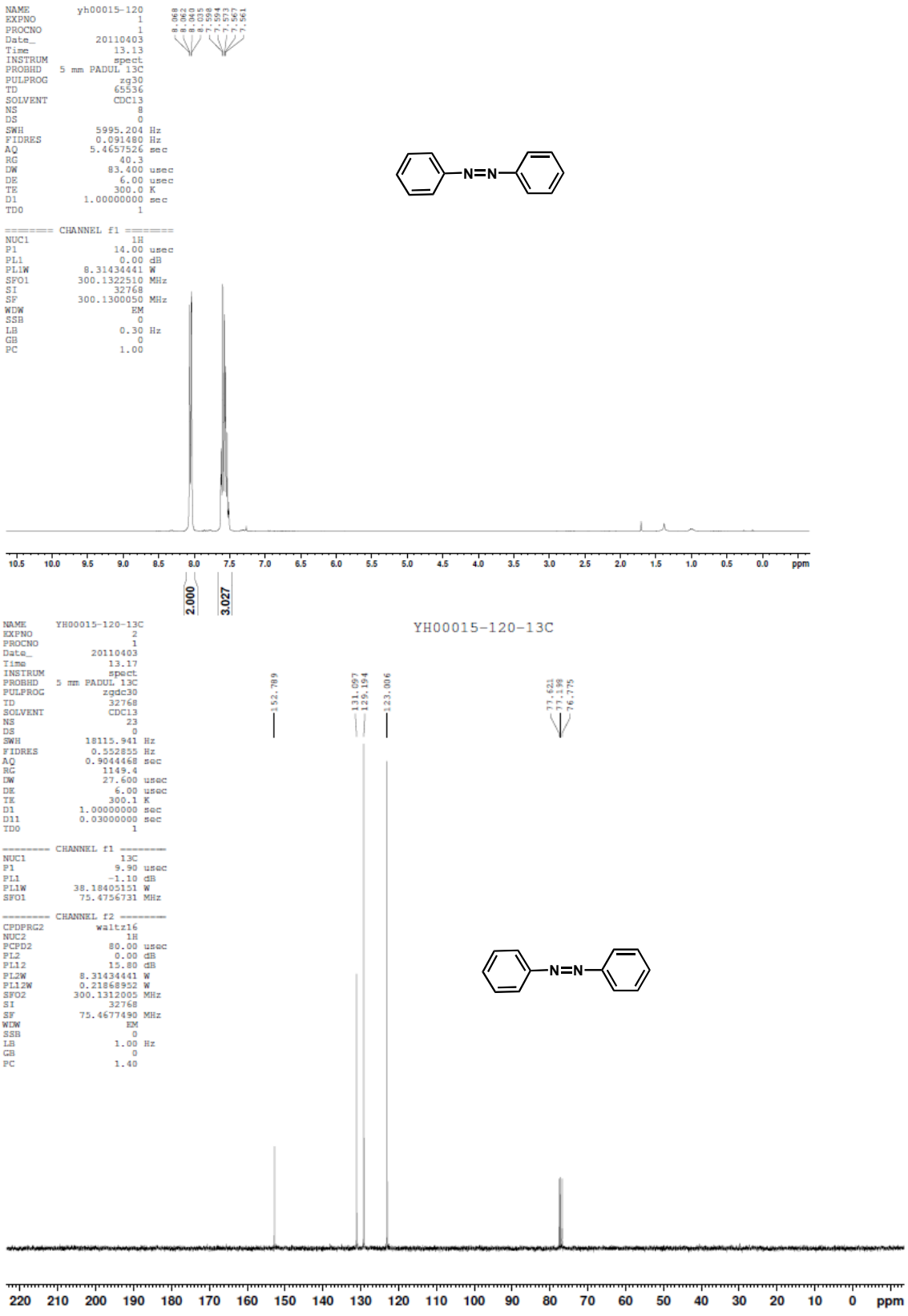

MeOOC
COOMe
MeOOC
COOMe
3.802
3.728
3.699
3.593
2.951
2.874
2.548
2.527
1.425
0.604
6.124
5.917
1.000
1.989
3.082
ppm


YH00005-037-2-13c
MeOOC
COOMe
MeOOC
COOMe
52.848
52.796
52.702
48.358
34.454
20.695
ppm

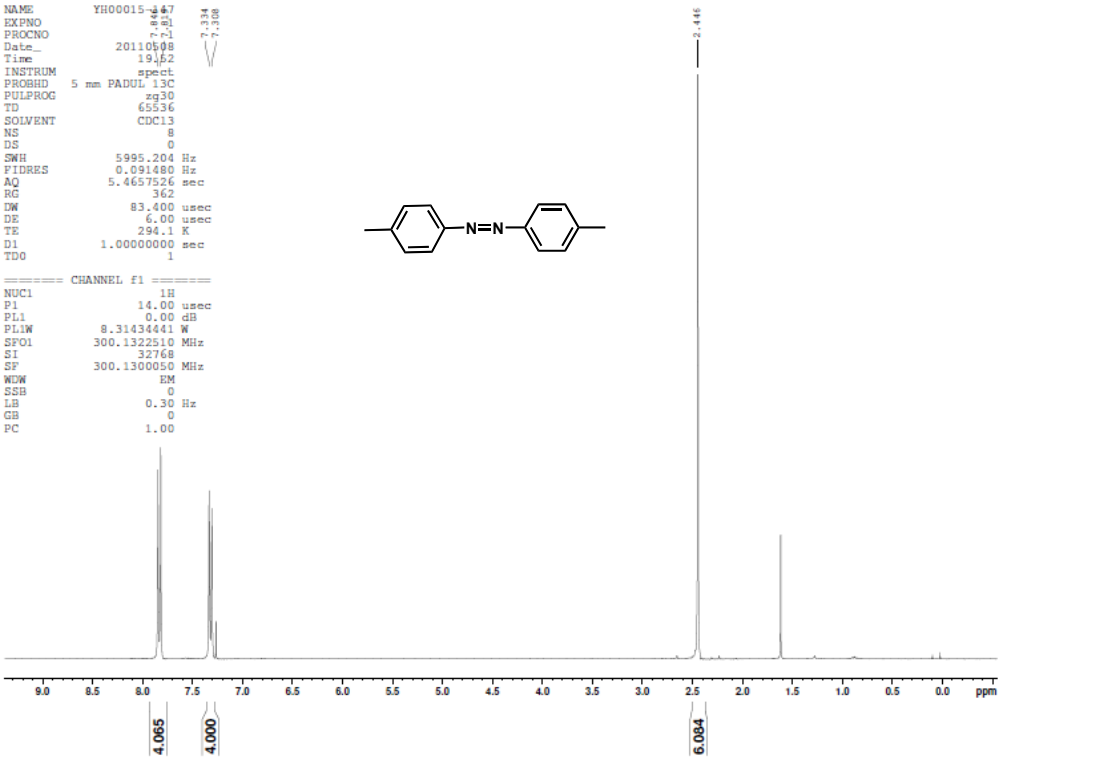
5d:

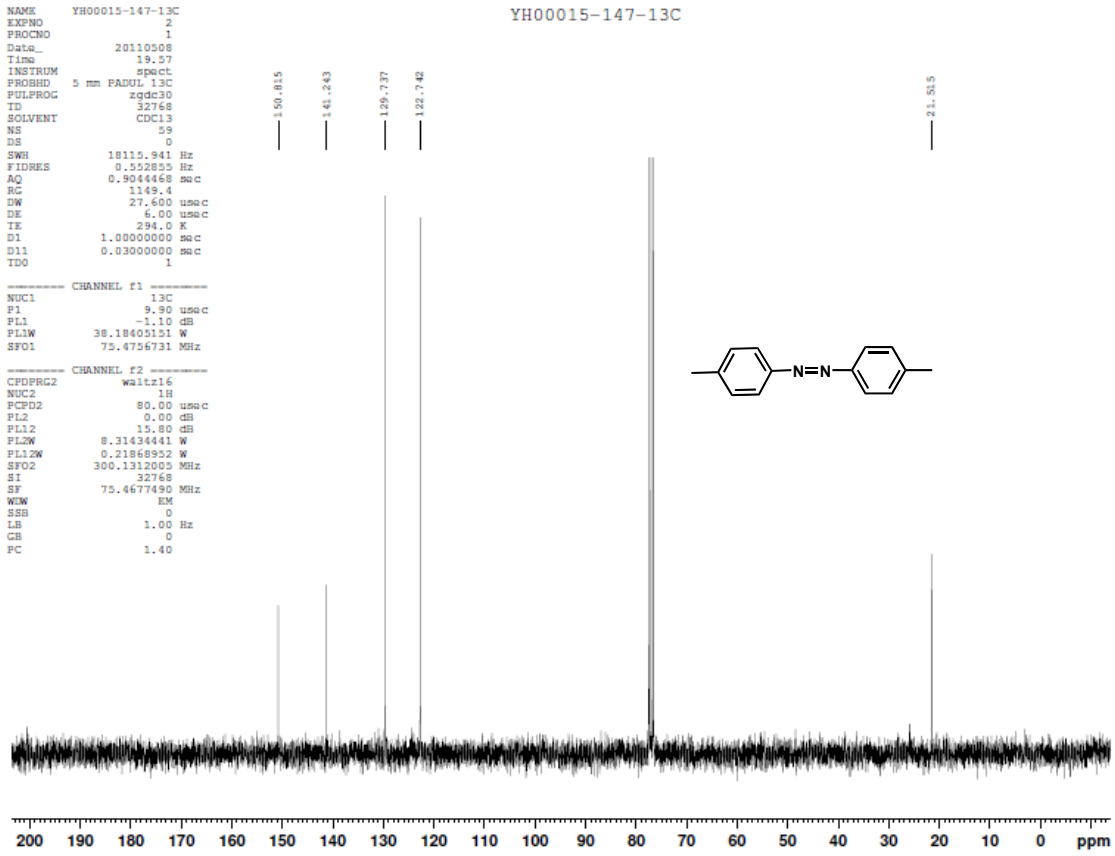


7a:

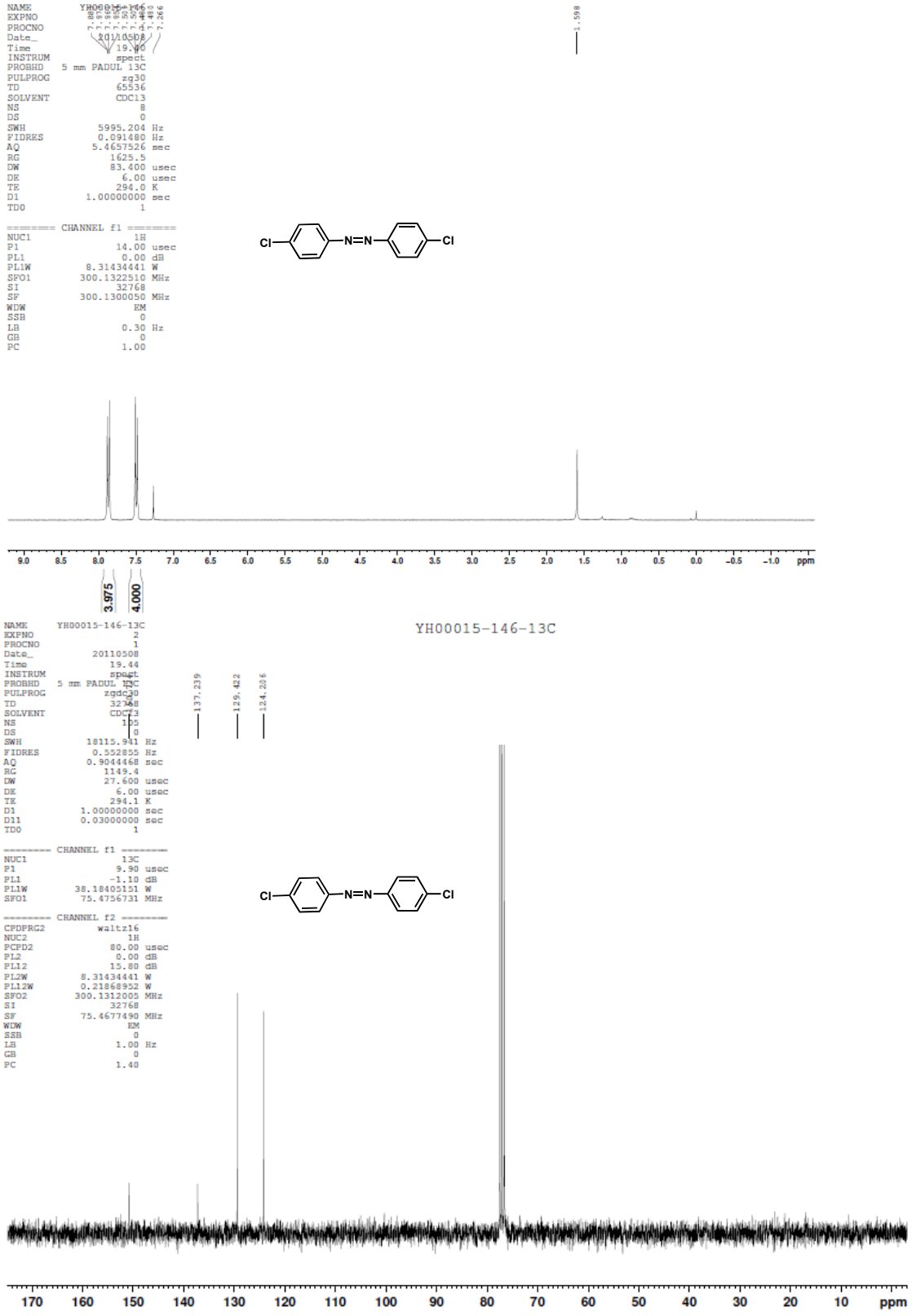

NAME yh00015-120
EXPNO 1
PROCNO 1
Date_ 20110403
Time 13.13
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 8
DS 0
SWH 5995.204 Hz
FIDRES 0.091480 Hz
AQ 5.4657526 sec
RG 40.3
DW 83.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 0.00 dB
PL1W 8.31434441 W
SFO1 300.1322510 MHz
SI 32768
SF 300.1300050 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.068
8.062
8.040
8.035
7.598
7.594
7.573
7.567
7.561
N=N
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
2.000
3.027
NAME YH00015-120-13C
EXPNO 2
PROCNO 1
Date_ 20110403
Time 13.17
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zgdc30
TD 32768
SOLVENT CDC13
NS 23
DS 0
SWH 18115.941 Hz
FIDRES 0.552855 Hz
AQ 0.9044468 sec
RG 1149.4
DW 27.600 usec
DE 6.00 usec
TE 300.1 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.90 usec
PL1 -1.10 dB
PL1W 38.18405151 W
SFO1 75.4756731 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 15.80 dB
PL2W 8.31434441 W
PL12W 0.21868952 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
YH00015-120-13C
152.789
131.097
129.194
123.006
77.621
77.198
76.775
N=N
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

7b:

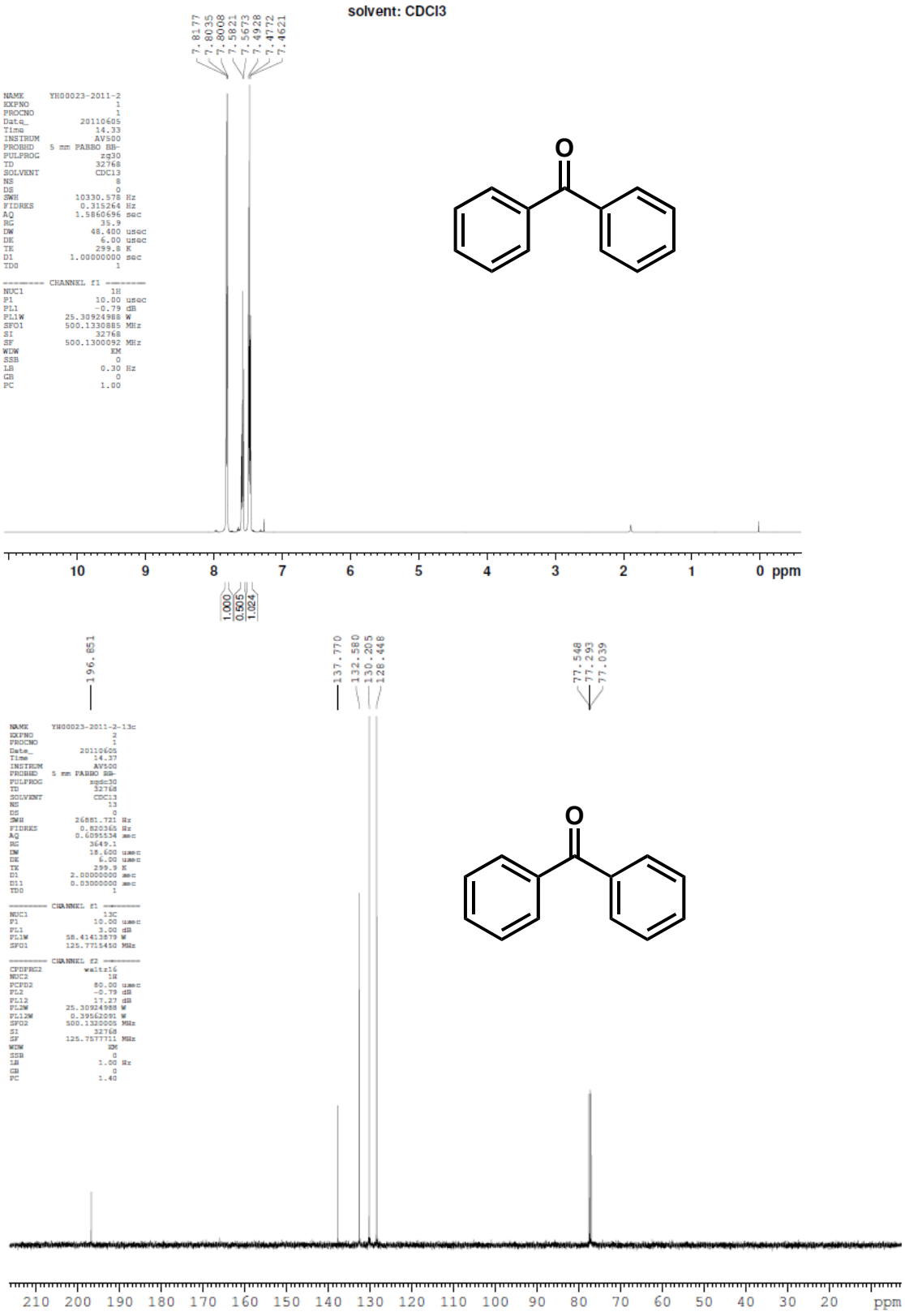




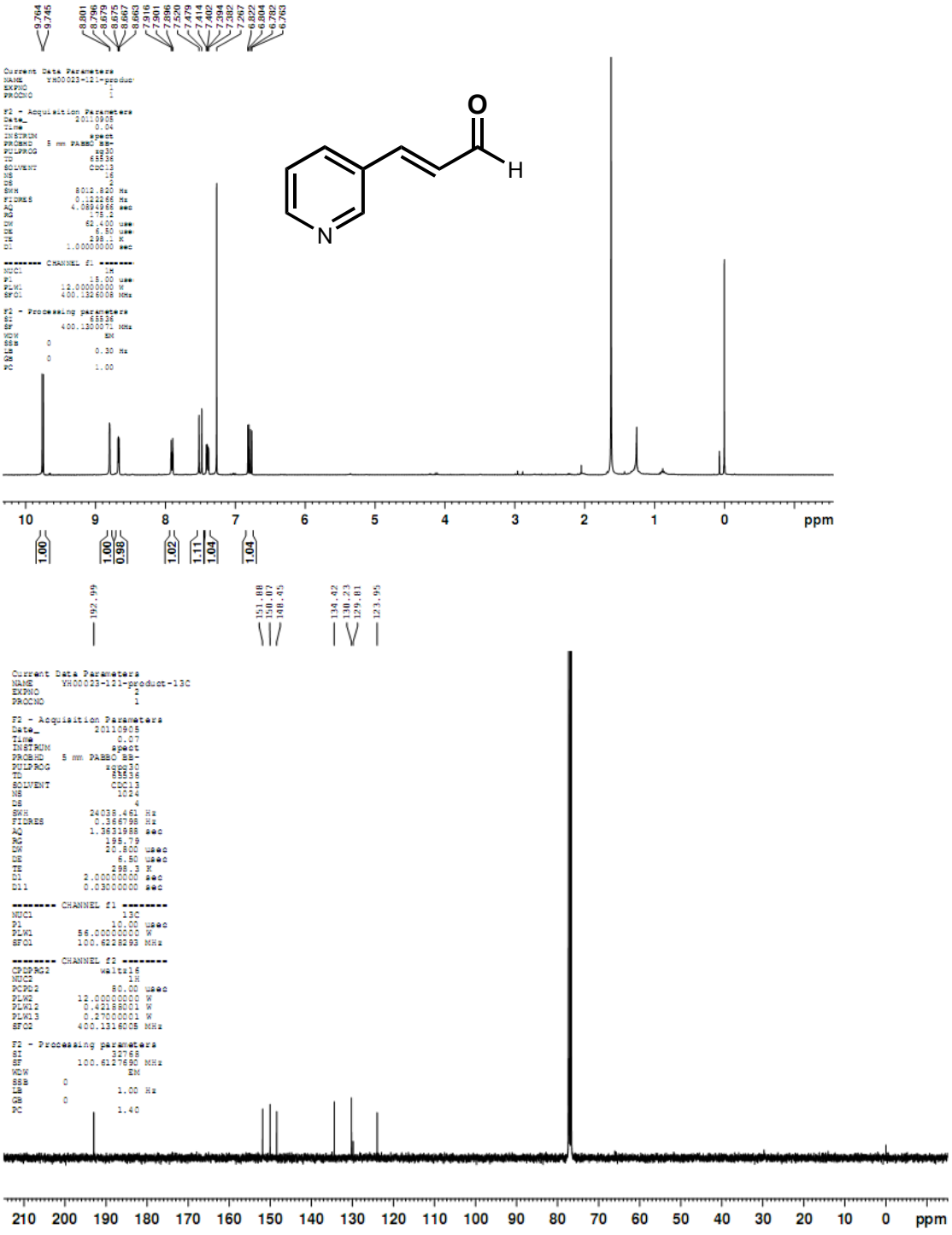
7c:



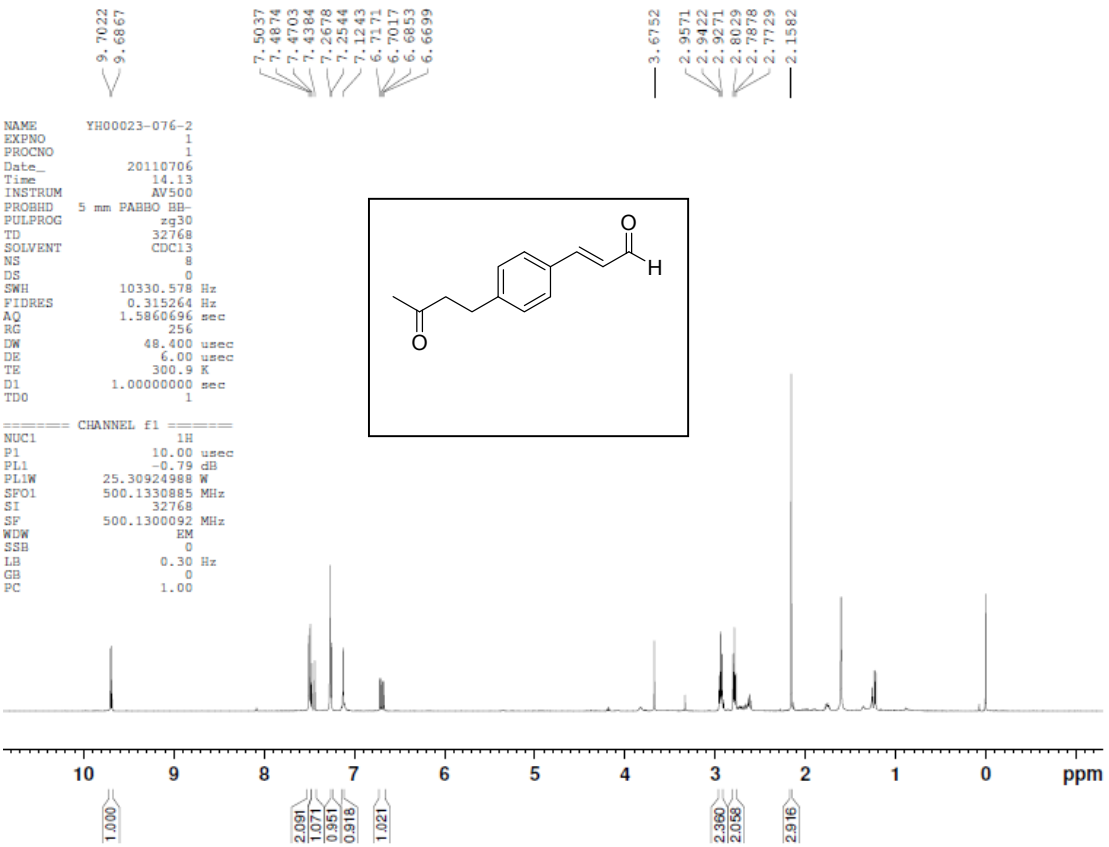
9:

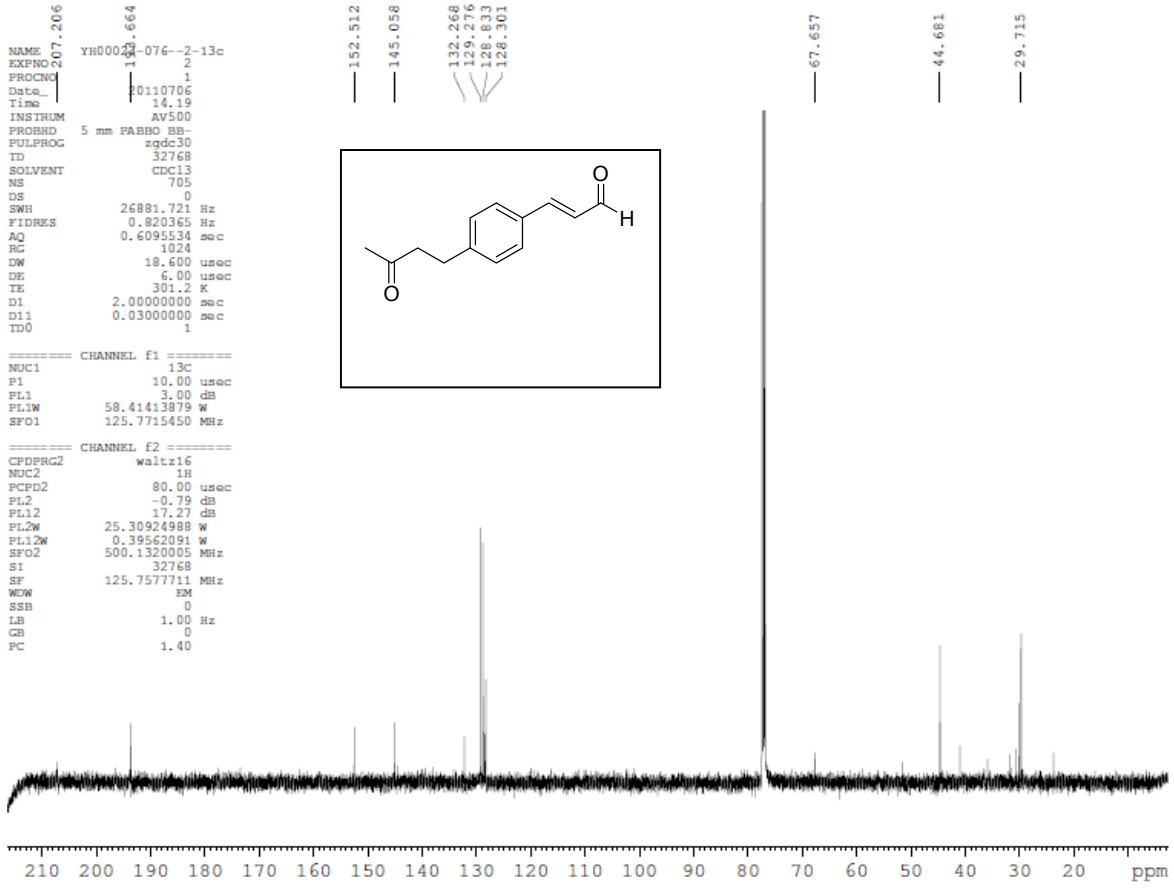

solvent: CDCl3
7.8177
7.8035
7.8008
7.5821
7.5673
7.4928
7.4772
7.4621
O
10
9
8
7
6
5
4
3
2
1
0 ppm
1.000
0.505
1.024
196.851
137.770
132.580
130.205
128.448
77.548
77.293
77.039
O
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

12:


9.764
9.745
8.801
8.796
8.679
8.675
8.667
8.663
7.916
7.901
7.896
7.520
7.479
7.414
7.402
7.394
7.382
7.367
6.822
6.804
6.782
6.763
O
H
N
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
1.00
0.98
1.02
1.11
1.04
1.04
192.99
151.88
150.07
148.45
134.42
130.23
129.81
123.95
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

14:





Mass spectra