Electronic Supplementary Material (ESI) for ChemComm.

Supporting Information

Selective organocatalytic oxidation of aldehydes and alcohols to unsaturated aldehydes: A homologation method to prepare polyenes

Xiaobei Chen, Yinan Zhang, Huixin Wan, Wei Wang, Shilei Zhang**

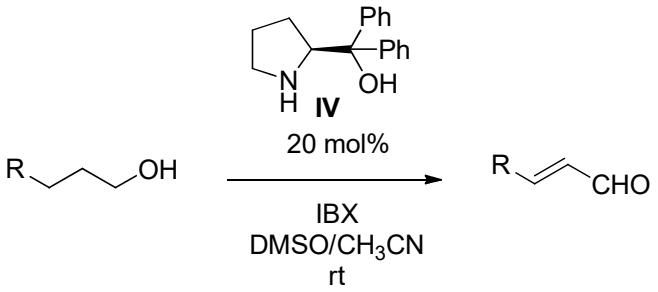
Jiangsu Key Laboratory of Translational Research and Therapy for Neuro-Psycho-Diseases & Department of Medicinal Chemistry, College of Pharmaceutical Sciences, Soochow University, 199 Ren'ai Road, Suzhou 215123, E-mail: zhangshilei@suda.edu.cn, China
Department of chemistry and chemical biology, University of New Mexico, Albuquerque, NM 87131-0001, E-mail: wwang@unm.edu, USA
School of Pharmacy, East China University of Science & Technology, 130 Mei-Long Road, Shanghai 200237, China
Central Research Institute of Shanghai Pharmaceuticals Holding Co., Ltd., Tower 4, No. 898 Ha Lei Road, Pudong New District, Shanghai, 201203, China

Contents

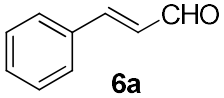
1. General Information:

Commercial reagents were used as received, unless otherwise stated. Merck 60 silica gel was used for chromatography, and Whatman silica gel plates with fluorescence F_{254} were used for thin-layer chromatography (TLC) analysis. ^{1}H and ^{13}C NMR spectra were recorded on Bruker Avance 500, 300, and Varian Unity Inova 400 instrument. Data for ^{1}H are reported as follows: chemical shift (ppm), and multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, dd = doublet doublet, dt = doublet triplet, m = multiplet). Data for ^{13}C NMR are reported as ppm. LR-ESI-MS were obtained using an Agilent 1100 MSD with methanol or acetonitrile as solvents in the positive ion mode; while HR-ESI-MS were carried out using AB SCIEX TripleTOF® 5600 System. Analytical high performance liquid chromatography (HPLC) was performed using a UV detector on a Agilent 1100 LC with HPLC-grade acetonitrile, deionized water, isopropanol and hexane as the eluting solvents. Semi-preparative HPLC separation was performed using a Varian Prostar 210 HPLC system equipped with a PDA detector 330 using a Supelco C18 column (25 × 21.2 mm, 10 μm; flow rate, 8 mL/min).

2. General procedures of organocatalytic oxidation



A solution of alcohol (0.15 mmol) in DMSO (0.3 mL) and CH_3CN (0.45 mL) was added IBX (105 mg, 0.375 mmol, 2.5 equiv.) and catalyst **IV** (7.6 mg, 0.03 mmol, 20 mol %). The resulting mixture was then stirred at room temp until the reaction was completed monitored with TLC (generally 4-24 h). After evaporating the volatiles, the crude products was first checked on ^{1}H-NMR for geometric E/Z ratio, and then direct column chromatography on silica gel using a mixture of hexane and ethyl acetate afforded the desired product.



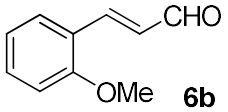
6a

Cinnamaldehyde (**6a, Figure 3**). The title compound was prepared according to general procedure of organocatalytic oxidation (reaction time: 3 h), as described above (17.6 mg, 89% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 9.71 (d, J = 8 Hz, 1H), 7.58-7.56 (m, 2H), 7.48 (d, J = 16 Hz, 1H), 7.44-7.41 (m, 3H), 6.72 (dd, J_1 = 16 Hz, J_2 = 7.5 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 193.6, 152.7, 134.0, 131.2, 129.1,

128.6, 128.4. LR-MS (ESI): m/z 133.1 $[M+H]^+$.

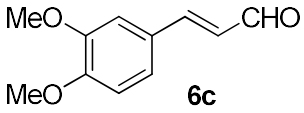
Scale-up:

A solution of 3-phenyl-1-propanol **5a** (3.0 mmol) in DMSO (6.0 mL) and CH_3CN (9.0 mL) was added IBX (2.1 g, 7.5 mmol, 2.5 equiv.) and catalyst **IV** (152 mg, 0.6 mmol, 20 mol %). The resulting mixture was stirred at room temp for 3 h. After evaporating the volatiles, the direct column chromatography on silica gel using a mixture of hexane and ethyl acetate afforded the desired product **6a** (337 mg, 85% yield).



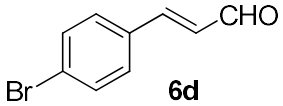
6b

***trans*-2-Methoxycinnamaldehyde** (**6b, Figure 3**). The title compound was prepared according to general procedure of organocatalytic oxidation (reaction time: 15 h), as described above (20.4 mg, 84% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 9.69 (d, J = 8.0 Hz, 1H), 7.84 (d, J = 16.0 Hz, 1H), 7.55 (d, J = 8.0 Hz, 1H), 7.43-7.39 (m, 1H), 7.00 (t, J = 7.0 Hz, 1H), 6.95 (d, J = 8.0 Hz, 1H), 6.64 (dd, J_1 = 16.0 Hz, J_2 = 8.0 Hz, 1H), 3.91 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 194.5, 158.3, 148.2, 132.7, 129.1, 128.9, 123.0, 120.9, 111.3, 55.5. LR-MS (ESI): m/z 163.1 $[M+H]^+$.



6c

***trans*-3,4-Dimethoxycinnamaldehyde** (**6c, Figure 3**). The title compound was prepared according to general procedure of organocatalytic oxidation (reaction time: 24 h), as described above (23.6 mg, 82% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 9.67 (d, J = 7.5 Hz, 1H), 7.42 (d, J = 16.0 Hz, 1H), 7.17 (dd, J_1 = 8.0 Hz, J_2 = 1.5 Hz, 1H), 7.08 (d, J = 1.5 Hz, 1H), 6.91 (d, J = 8 Hz, 1H), 6.62 (dd, J_1 = 16.0 Hz, J_2 = 8.0 Hz, 1H); 3.94 (s, 3H), 3.93 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 193.5, 152.8, 152.0, 149.4, 127.0, 126.7, 123.4, 111.1, 109.9, 56.0, 55.9. LR-MS (ESI): m/z 193.1 $[M+H]^+$.



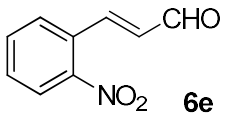
6d

***trans*-4-Bromocinnamaldehyde** (**6d, Figure 3**). The title compound was prepared according to general procedure of organocatalytic oxidation (reaction time: 9 h), as described above (28.7 mg, 91% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 9.70 (d, J = 7.5 Hz, 1H), 7.56 (d, J = 8.5 Hz, 2H), 7.43-7.39 (m, 3H), 6.69 (dd, J_1 = 16.0 Hz, J_2 = 7.5 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 193.2, 150.9, 132.9, 132.4, 129.7, 129.0, 125.6. LR-MS (ESI): m/z 211.0 $[M+H]^+$.

Scale-up:

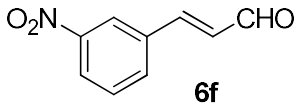
A solution of alcohol **5d** (3.0 mmol) in DMSO (6.0 mL) and CH_3CN (9.0 mL) was added IBX (2.1 g, 7.5 mmol, 2.5 equiv.) and catalyst **IV** (152 mg, 0.6 mmol, 20 mol %). The resulting mixture was stirred at room temp for 7 h. After evaporating the

volatiles, the direct column chromatography on silica gel using a mixture of hexane and ethyl acetate afforded the desired product **6d** (580 mg, 92% yield).



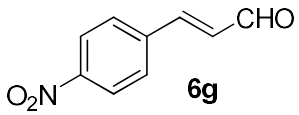
6e

***trans*-2-Nitrocinnamaldehyde** (**6e, Figure 3**). The title compound was prepared according to general procedure of organocatalytic oxidation (reaction time: 4 h), as described above (21.3 mg, 80% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 9.79 (d, J = 7.5 Hz, 1H), 8.11 (d, J = 8.0 Hz, 1H), 8.05 (d, J = 16.0 Hz, 1H), 7.74-7.69 (m, 2H), 7.64-7.61 (m, 1H), 6.64 (dd, J_1 = 16.0 Hz, J_2 = 7.5 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 193.0, 148.0, 147.2, 133.8, 132.6, 131.1, 130.0, 129.0, 125.1. LR-MS (ESI): m/z 178.1 $[M+H]^+$.



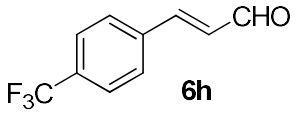
6f

(*E*)-3-(3-Nitrophenyl)acrylaldehyde (**6f, Figure 3**). The title compound was prepared according to general procedure of organocatalytic oxidation (reaction time: 8 h), as described above (23.6 mg, 89% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 9.77 (d, J = 7.5 Hz, 1H), 8.41 (s, 1H), 8.28 (d, J = 8.0 Hz, 1H), 7.89 (d, J = 7.5 Hz, 1H), 7.64 (t, J = 7.5 Hz, 1H), 7.53 (d, J = 16.5 Hz, 1H), 6.81 (dd, J_1 = 16.5 Hz, J_2 = 7.5 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 192.7, 148.9, 148.8, 135.7, 133.6, 130.9, 130.2, 125.3, 123.0. LR-MS (ESI): m/z 178.1 $[M+H]^+$.



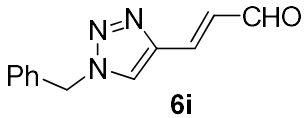
6g

***trans*-4-Nitrocinnamaldehyde** (**6g, Figure 3**). The title compound was prepared according to general procedure of organocatalytic oxidation (reaction time: 4 h), as described above (21.5 mg, 81% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 9.78 (d, J = 7.5 Hz, 1H), 8.29 (d, J = 8.5 Hz, 2H), 7.74 (d, J = 8.5 Hz, 2H), 7.54 (d, J = 16.0 Hz, 1H), 6.81 (dd, J_1 = 16.0 Hz, J_2 = 7.5 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 192.7, 149.0, 148.8, 139.9, 131.7, 129.0, 124.3. LR-MS (ESI): m/z 178.1 $[M+H]^+$.



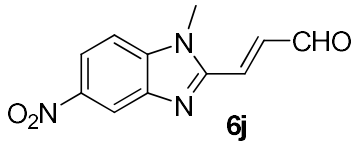
6h

***trans*-4-(Trifluoromethyl)cinnamaldehyde** (**6h, Figure 3**). The title compound was prepared according to general procedure of organocatalytic oxidation A (reaction time: 6 h), as described above (27.6 mg, 92% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 9.75 (d, J = 7.5 Hz, 1H), 7.68 (m, 4H), 7.50 (d, J = 16.0 Hz, 1H), 6.77 (dd, J_1 = 16.0 Hz, J_2 = 7.5 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 193.1, 150.2, 137.3, 130.5, 128.5, 126.0. LR-MS (ESI): m/z 201.0 $[M+H]^+$.



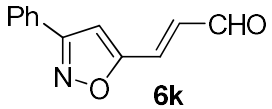
6i

(*E*)-3-(1-Benzyl-1H-1,2,3-triazol-4-yl)acrylaldehyde (**6i, Figure 3**). The title compound was prepared according to general procedure of organocatalytic oxidation (reaction time: 6 h), as described above (24.9 mg, 78% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 9.64 (d, J = 7.0 Hz, 1H), 7.72 (s, 1H), 7.48 (d, J = 16.0 Hz, 1H), 7.38 (m, 3H), 7.30 (m, 2H), 6.77 (dd, J_1 = 16.0 Hz, J_2 = 7.0 Hz, 1H), 5.56 (s, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 193.1, 143.7, 140.0, 133.8, 129.8, 129.3, 129.1, 128.1, 123.6, 54.4. LR-MS (ESI): m/z 214.1 $[M+H]^+$; HR-MS (ESI) m/z $[M + H]^+$ calcd for $C_{12}H_{12}N_3O$ 214.0980, found 214.0981.



6j

(*E*)-3-(1-Methyl-5-nitro-1H-benzo[d]imidazol-2-yl)acrylaldehyde (**6j, Figure 3**). The title compound was prepared according to general procedure of organocatalytic oxidation (reaction time: 10 h), as described above (29.8 mg, 86% yield). ^{1}H NMR (300 MHz, acetone-d^6): δ 9.82 (d, J = 7.8 Hz, 1H), 8.50 (s, 1H), 8.15 (dd, J = 9.0, 1.5 Hz, 1H), 8.01 (d, J = 15.6 Hz, 1H), 7.81 (d, J = 9.0 Hz, 1H), 7.08 (dd, J_1 = 15.6 Hz, J_2 = 7.8 Hz, 1H), 3.99 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 194.1, 152.1, 143.6, 141.6, 140.5, 136.6, 134.5, 118.9, 115.8, 111.8, 30.7. LR-MS (ESI): m/z 232.1 $[M+H]^+$; HR-MS (ESI) m/z $[M + H]^+$ calcd for $C_{11}H_{10}N_3O_3$ 232.0722, found 232.0711.

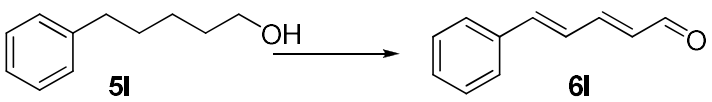


6k

(*E*)-3-(3-Phenylisoxazol-5-yl)acrylaldehyde (**6k, Figure 3**). The title compound was prepared according to general procedure of organocatalytic oxidation (reaction time: 8 h), as described above (23.9 mg, 80% yield). ^{1}H NMR (300 MHz, $CDCl_3$): δ 9.73 (d, J = 7.5 Hz, 1H), 7.81 (m, 2H), 7.47 (m, 3H), 7.34 (d, J = 16.2 Hz, 1H), 6.88 (s, 1H), 6.84 (dd, J_1 = 16.2 Hz, J_2 = 7.5 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 192.2, 165.8, 163.2, 133.3, 132.2, 130.5, 129.1, 128.1, 126.8, 105.1. LR-MS (ESI): m/z 200.1 $[M+H]^+$; HR-MS (ESI) m/z $[M + H]^+$ calcd for $C_{12}H_{10}NO_2$ 200.0712, found 200.0718.

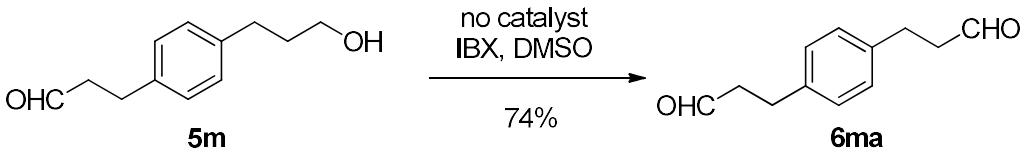
Scale-up:

A solution of alcohol **5k** (3.0 mmol) in DMSO (6.0 mL) and CH_3CN (9.0 mL) was added IBX (2.1 g, 7.5 mmol, 2.5 equiv.) and catalyst **IV** (152 mg, 0.6 mmol, 20 mol %). The resulting mixture was stirred at room temp for 8 h. After evaporating the volatiles, the direct column chromatography on silica gel using a mixture of hexane and ethyl acetate afforded the desired product **6k** (519 mg, 87% yield).



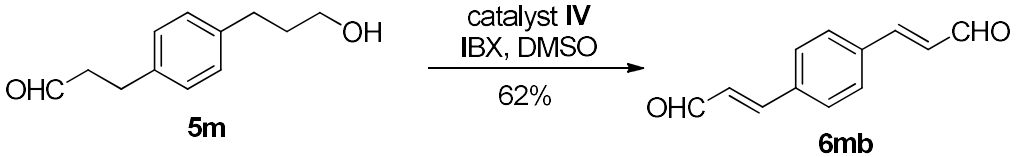
5l 6l

(2*E*,4*E*)-5-Phenylpenta-2,4-dienal (6l, Figure 3). The title compound was prepared according to general procedure of organocatalytic oxidation (4 eq. IBX was used, total reaction time: 21 h), as described above (5.7 mg, 24% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 9.61 (d, J = 8.0 Hz, 1H), 7.50 (m, 2H), 7.36 (m, 3H), 7.26 (m, 1H), 7.00 (m, 2H), 6.26 (dd, J_1 = 15.0 Hz, J_2 = 8.0 Hz, 1H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 193.4, 151.9, 142.3, 135.5, 131.5, 129.6, 128.8, 127.4, 126.1. LR-MS (ESI): m/z 159.1 $[M+H]^+$.



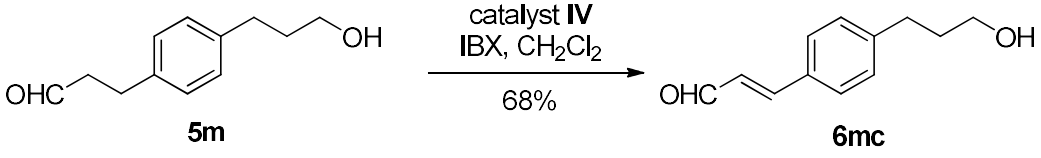
5m 6ma

3,3'-(1,4-phenylene)dipropanal (6ma, Scheme 1) Alcohol **5m** (19 mg, 0.1 mmol) was dissolved in DMSO (0.5 mL), followed by addition of IBX (34 mg, 0.12 mmol). The reaction mixture was stirred at rt for 5 h. The product was purified directly by column chromatography (eluted by EtOAc/hexane = 1/2) to afford 14.1 mg aldehyde **6ma**, yield: 74%. ^{1}H NMR (500 MHz, $CDCl_3$): δ 9.81 (s, 2H), 7.12 (s, 4H), 2.93 (t, J = 7.5 Hz, 4H), 2.76 (t, J = 7.5 Hz, 4H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 201.5, 138.3, 128.5, 45.2, 27.7. LR-MS (ESI): m/z 191.1 $[M+H]^+$; HR-MS (ESI) m/z $[M + H]^+$ calcd for $C_{12}H_{15}O_2$ 191.1072, found 191.1079.



5m 6mb

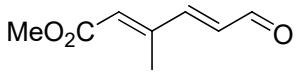
(2*E*,2'*E*)-3,3'-(1,4-phenylene)diacrylaldehyde (6mb, Scheme 1) Alcohol **5m** (19 mg, 0.1 mmol) was dissolved in DMSO (0.5 mL), followed by addition of IBX (92 mg, 0.33 mmol) and catalyst **IV** (3.3 mg, 0.01 mmol). The reaction mixture was stirred at rt for 4 h. The product was purified directly by column chromatography (eluted by EtOAc/hexane = 1/2) to afford 11.5 mg aldehyde **6mb**, yield: 62%. ^{1}H NMR (500 MHz, $CDCl_3$): δ 9.74 (d, J = 7.5 Hz, 2H), 7.63 (s, 4H), 7.48 (d, J = 16.0 Hz, 2H), 6.77 (dd, J = 16.0, 7.5 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 193.3, 150.8, 136.5, 129.8, 129.0. LR-MS (ESI): m/z 187.1 $[M+H]^+$; HR-MS (ESI) m/z $[M + H]^+$ calcd for $C_{12}H_{11}O_2$ 187.0759, found 187.0755.



5m 6mc

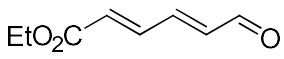
(*E*)-3-(4-(3-hydroxypropyl)phenyl)acrylaldehyde (6mc, Scheme 1) Alcohol **5m** (19

mg, 0.1 mmol) was dissolved in CH_2Cl_2 (0.5 mL), followed by addition of IBX (34 mg, 0.12 mmol) and catalyst **IV** (3.3 mg, 0.01 mmol). The reaction mixture was stirred at rt for 2 h. The product was purified directly by column chromatography (eluted by EtOAc/hexane = 1/1) to afford 13 mg aldehyde **6mc**, yield: 68%. ^{1}H NMR (500 MHz, $CDCl_3$): δ 9.68 (d, *J* = 8.0 Hz, 1H), 7.49 (d, *J* = 8.0 Hz, 2H), 7.45 (d, *J* = 16.0 Hz, 1H), 7.27 (d, *J* = 8.5 Hz, 2H), 6.68 (dd, *J* = 16.0, 7.5 Hz, 1H), 3.68 (t, *J* = 6.5 Hz, 2H), 2.76 (t, *J* = 7.5 Hz, 2H), 1.90 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 193.8, 152.8, 145.9, 131.7, 129.2, 128.6, 127.8, 61.9, 33.8, 32.0. LR-MS (ESI): m/z 191.1 $[M+H]^+$; HR-MS (ESI) m/z $[M + H]^+$ calcd for $C_{12}H_{15}O_2$ 191.1072, found 191.1085.



8a

(2*E*,4*E*)-Methyl 3-methyl-6-oxohexa-2,4-dienoate (**8a, Figure 4**). The title compound was prepared according to general procedure of organocatalytic oxidation (reaction time: 5 h), as described above (19.0 mg, 82% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 9.67 (d, *J* = 7.0 Hz, 1H), 7.10 (d, *J* = 15.5 Hz, 1H), 6.45 (dd, J_1 = 15.5 Hz, J_2 = 8.0 Hz, 1H), 6.13 (s, 1H), 3.77 (s, 3H), 2.32 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 193.3, 166.2, 154.0, 148.9, 132.8, 126.4, 51.6, 13.8. LR-MS (ESI): m/z 155.1 $[M+H]^+$; HR-MS (ESI) m/z $[M + H]^+$ calcd for $C_8H_{11}O_3$ 155.0708, found 155.0701.

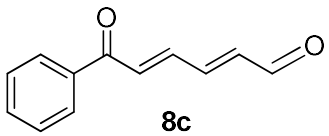


8b

(2*E*,4*E*)-Ethyl 6-oxohexa-2,4-dienoate (**8b, Figure 4**). The title compound was prepared according to general procedure of organocatalytic oxidation (reaction time: 4 h), as described above (21.0 mg, 91% yield). ^{1}H NMR (300 MHz, $CDCl_3$): δ 9.67 (d, *J* = 7.8 Hz, 1H), 7.41 (dd, J_1 = 15.3 Hz, J_2 = 11.1 Hz, 1H), 7.16 (dd, J_1 = 15.3 Hz, J_2 = 11.4 Hz, 1H), 6.41 (dd, J_1 = 15.3 Hz, J_2 = 7.8 Hz, 1H), 6.30 (d, *J* = 15.3 Hz, 1H), 4.25 (q, *J* = 7.2 Hz, 2H), 1.32 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 192.9, 165.4, 147.2, 140.3, 136.9, 129.9, 61.1, 14.2. LR-MS (ESI): m/z 155.1 $[M+H]^+$; HR-MS (ESI) m/z $[M + H]^+$ calcd for $C_8H_{11}O_3$ 155.0708, found 155.0710.

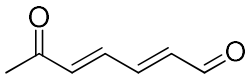
Scale-up:

A solution of alcohol **7b** (3.0 mmol) in DMSO (6.0 mL) and CH_3CN (9.0 mL) was added IBX (2.1 g, 7.5 mmol, 2.5 equiv.) and catalyst **IV** (152 mg, 0.6 mmol, 20 mol %). The resulting mixture was stirred at room temp for 4 h. After evaporating the volatiles, the direct column chromatography on silica gel using a mixture of hexane and ethyl acetate afforded the desired product **8b** (411 mg, 89% yield).



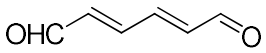
8c

(2*E*,4*E*)-6-Oxo-6-phenylhexa-2,4-dienal (**8c, Figure 4**). The title compound was prepared according to general procedure of organocatalytic oxidation (reaction time: 5 h), as described above (20.9 mg, 75% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 9.72 (d, J = 8.0 Hz, 1H), 7.98 (m, 2H), 7.62 (m, 1H), 7.54-7.51 (m, 3H), 7.39-7.26 (m, 2H), 6.51 (dd, J_1 = 15.5 Hz, J_2 = 8.0 Hz, 1H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 192.9, 189.4, 147.7, 139.9, 137.6, 137.1, 133.5, 133.0, 128.8, 128.5. LR-MS (ESI): m/z 187.0 $[M+H]^+$; HR-MS (ESI) m/z $[M + H]^+$ calcd for $C_{12}H_{11}O_2$ 187.0759, found 187.0771.



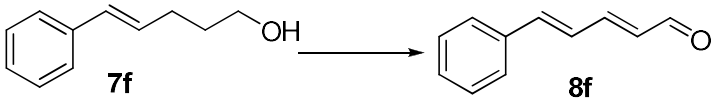
8d

(2*E*,4*E*)-6-Oxohepta-2,4-dienal (**8d, Figure 4**). The title compound was prepared according to general procedure of organocatalytic oxidation (reaction time: 5 h), as described above (14.3 mg, 77% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 9.67 (d, J = 7.5 Hz, 1H), 7.26-7.15 (m, 2H), 6.52-6.43 (m, 2H), 2.35 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 197.4, 192.7, 147.7, 138.6, 137.5, 137.3, 27.8. LR-MS (ESI): m/z 125.0 $[M+H]^+$, HR-MS (ESI) m/z $[M + H]^+$ calcd for $C_7H_9O_2$ 125.0603, found 125.0604.



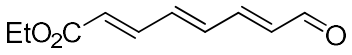
8e

(2*E*,4*E*)-Hexa-2,4-dienedial (**8e, Figure 4**). The title compound was prepared according to general procedure of organocatalytic oxidation (reaction time: 12 h), as described above (11.2 mg, 68% yield). ^{1}H NMR (300 MHz, $CDCl_3$): δ 9.72 (d, J = 7.5 Hz, 2H), 7.34-7.23 (m, 2H), 6.56-6.48 (m, 2H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 192.6, 146.4, 138.0. LR-MS (ESI): m/z 111.0 $[M+H]^+$; HR-MS (ESI) m/z $[M + H]^+$ calcd for $C_6H_7O_2$ 111.0446, found 111.0437.



7f **8f**

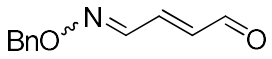
(2*E*,4*E*)-5-Phenylpenta-2,4-dienal (**8f/6l, Figure 4**). The title compound was prepared according to general procedure of organocatalytic oxidation (reaction time: 4 h), as described above (15.4 mg, 65% yield), same product as **6l**.



8g

(2*E*,4*E*,6*E*)-Ethyl 8-oxoocta-2,4,6-trienoate (**8g, Figure 4**). The title compound was prepared according to general procedure of organocatalytic oxidation (reaction time: 3.5 h), as described above (16.2 mg, 60% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 9.62 (d, J = 8.0 Hz, 1H), 7.34 (m, 1H), 7.15 (m, 1H), 6.74 (m, 2H), 6.27 (dd, J_1 = 15.5 Hz, J_2 = 8.0 Hz, 1H), 6.09 (d, J = 15.5 Hz, 1H), 4.22 (q, J = 7.0 Hz, 2H), 1.31 (t, J = 7.0 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 193.2, 166.2, 149.5, 142.1, 138.6, 136.5, 133.9, 125.9, 60.7, 14.2. LR-MS (ESI): m/z 181.1 $[M+H]^+$; HR-MS (ESI) m/z [M +

H]$^+$ calcd for $C_{10}H_{13}O_3$ 181.0865, found 181.0870.

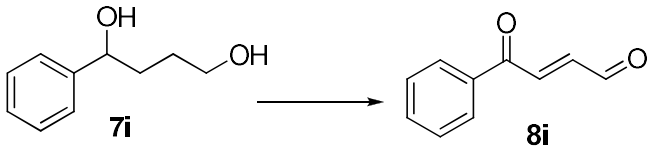


8h

(2*E*)-4-((Benzyloxy)imino)but-2-enal (**8h, Figure 4**). The title compound was prepared according to general procedure of organocatalytic oxidation (reaction time: 3 h), as described above (23.8 mg, 84% yield). major: ^{1}H NMR (500 MHz, $CDCl_3$): δ 9.66 (d, J = 7.5 Hz, 1H), 7.98 (d, J = 10.0 Hz, 1H), 7.37 (m, 5H), 7.16 (dd, J_1 = 16.0 Hz, J_2 = 10.0 Hz, 1H), 6.34 (dd, J_1 = 16.0 Hz, J_2 = 7.5 Hz, 1H), 5.24 (s, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 192.8, 148.3, 145.3, 143.9, 136.5, 135.5, 128.5, 128.4, 128.3, 77.4. LR-MS (ESI): m/z 190.1 [M+H]$^+$; HR-MS (ESI) m/z [M + H]$^+$ calcd for $C_{11}H_{12}NO_2$ 190.0868, found 190.0871.

Scale-up:

A solution of alcohol **7h** (3.0 mmol) in DMSO (6.0 mL) and CH_3CN (9.0 mL) was added IBX (2.1 g, 7.5 mmol, 2.5 equiv.) and catalyst **IV** (152 mg, 0.6 mmol, 20 mol %). The resulting mixture was stirred at room temp for 3 h. After evaporating the volatiles, the direct column chromatography on silica gel using a mixture of hexane and ethyl acetate afforded the desired product **8h** (448 mg, 79% yield).

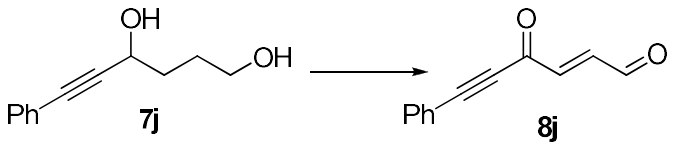


7i **8i**

(*E*)-4-Oxo-4-phenylbut-2-enal (**8i, Figure 4**). The title compound was prepared according to general procedure of organocatalytic oxidation (5 eq. IBX was used, total reaction time: 12 h), as described above (12.5 mg, 52% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 9.88 (d, J = 7.5 Hz, 1H), 7.98 (d, J = 8.0 Hz, 2H), 7.71 (d, J = 16.0 Hz, 1H), 7.64 (m, 1H), 7.53 (m, 2H), 6.98 (dd, J_1 = 16.0 Hz, J_2 = 7.5 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 192.7, 189.7, 142.0, 139.1, 136.2, 134.1, 129.0, 128.8. LR-MS (ESI): m/z 161.1 [M+H]$^+$; HR-MS (ESI) m/z [M + H]$^+$ calcd for $C_{10}H_9O_2$ 161.0603, found 161.0600.

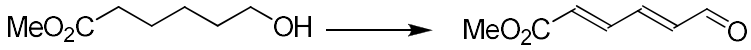
Scale-up:

A solution of alcohol **7i** (3.0 mmol) in DMSO (6.0 mL) and CH_3CN (9.0 mL) was added IBX (4.2 g, 15.0 mmol, 5.0 equiv.) and catalyst **IV** (152 mg, 0.6 mmol, 20 mol %). The resulting mixture was stirred at room temp for 12 h. After evaporating the volatiles, the direct column chromatography on silica gel using a mixture of hexane and ethyl acetate afforded the desired product **8i** (302 mg, 63% yield).



7j **8j**

(*E*)-4-Oxo-6-phenylhex-2-en-5-ynal (**8j, Figure 4**). The title compound was prepared according to general procedure of organocatalytic oxidation (5.0 eq. IBX was used, total reaction time: 10 h), as described above (9.7 mg, 35% yield). ^{1}H NMR (300 MHz, $CDCl_3$): δ 9.90 (d, J = 7.2 Hz, 1H), 7.64 (d, J = 6.9 Hz, 2H), 7.53 (m, 1H), 7.43 (m, 2H), 7.14 (dd, J_1 = 15.9 Hz, J_2 = 7.2 Hz, 1H), 6.98 (d, J = 16.2 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 192.8, 176.9, 145.9, 140.9, 133.4, 131.5, 128.8, 119.1, 94.4, 86.3. LR-MS (ESI): m/z 185.1 $[M+H]^+$; HR-MS (ESI) m/z $[M + H]^+$ calcd for $C_{12}H_9O_2$ 185.0603, found 185.0609.

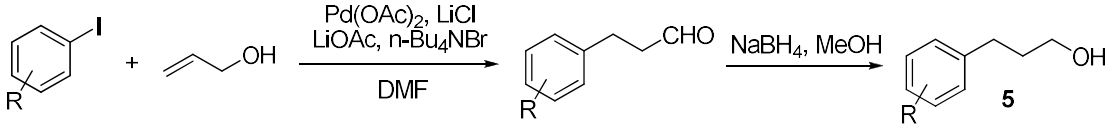


7k **8k**

(2*E*,4*E*)-Methyl 6-oxohexa-2,4-dienoate (**8k, Figure 4**). The title compound was prepared according to general procedure (total reaction time: 10 h), as described above (5.5 mg, 26% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 9.67 (d, J = 7.5 Hz, 1H), 7.42 (dd, J_1 = 15.5 Hz, J_2 = 11.5 Hz, 1H), 7.16 (dd, J_1 = 15.5 Hz, J_2 = 11.5 Hz, 1H), 6.42 (dd, J_1 = 15.5 Hz, J_2 = 7.5 Hz, 1H), 6.31 (d, J = 15.0 Hz, 1H), 3.80 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 192.9, 165.9, 147.1, 140.6, 137.1, 129.4, 52.1. LR-MS (ESI): m/z 141.0 $[M+H]^+$; HR-MS (ESI) m/z $[M + H]^+$ calcd for $C_7H_9O_3$ 141.0552, found 141.0543.

3. General procedures for preparation of substrates 5 and 7 unavailable from commercial sources.

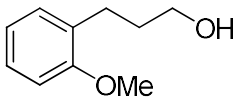
3.1 General procedures for preparation of 3-phenyl-1-propanols (5b-h)



A solution of iodobenzene (2 mmol) and allyl alcohol (151 mg, 177 μL, 2.6 mmol) in DMF (12 mL) were added $Pd(OAc)_2$ (14 mg, 0.06 mmol, 0.03 eq.), LiCl (85 mg, 2.0 mmol, 1.0 eq.), $LiOAc·2H_2O$ (612 mg, 6.0 mmol, 3 eq.) and n-Bu_4NBr (1.29 g, 4.0 mmol, 2.0 eq.). The reaction mixture was stirred at rt for 48 h. Water was added and extracted with EtOAc for 3 times. The combined organic layers were washed with water and brine and dried over sodium sulfate and concentrated under reduced pressure. The crude product was purified by column chromatography to give desired aldehyde.

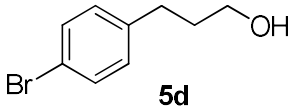
To a solution of the aldehyde intermediate (0.5 mmol) dissolved in MeOH (3 mL), $NaBH_4$ (19 mg, 0.5 mmol, 1 eq.) was added and the reaction was stirred at rt for 10 min. The reaction mixture was neutralized with the addition of water and extracted with EtOAc for 3 times. The combined organic layers were washed with brine and dried over sodium sulfate and concentrated under reduced pressure. The crude product

was purified by column chromatography to give desired alcohol **5**.



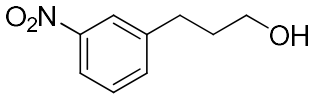
5b

3-(2-Methoxyphenyl)propan-1-ol (**5b, Figure 3**). The title compound was prepared according to general procedure, as described above (186 mg, 56% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.20 (t, *J* = 7.5 Hz, 1H), 7.16 (d, *J* = 7.0 Hz, 1H), 6.91 (t, *J* = 7.5 Hz, 1H), 6.87 (d, *J* = 8.0 Hz, 1H), 3.84 (s, 3H), 3.61 (t, *J* = 6.5 Hz, 2H), 2.74 (t, *J* = 7.0 Hz, 2H), 2.03 (br, 1H), 1.86 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 157.3, 130.0, 129.9, 127.1, 120.6, 110.2, 61.9, 55.3, 32.8, 25.9.



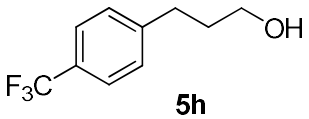
5d

3-(4-Bromophenyl)propan-1-ol (**5d, Figure 3**). The title compound was prepared according to general procedure, as described above (304 mg, 71% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.39 (d, *J* = 8.5 Hz, 2H), 7.06 (d, *J* = 8.0 Hz, 2H), 3.64 (t, *J* = 6.5 Hz, 2H), 2.65 (t, *J* = 7.5 Hz, 2H), 1.85 (m, 2H), 1.78 (br, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 140.7, 131.4, 130.1, 119.5, 61.9, 33.9, 31.4.



5f

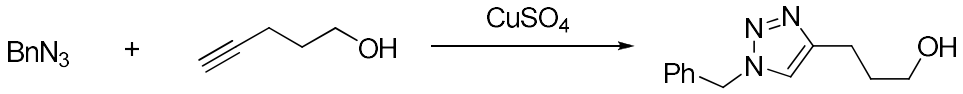
3-(3-Nitrophenyl)propan-1-ol (**5f, Figure 3**). The title compound was prepared according to general procedure, as described above (243 mg, 67% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.04 (s, 1H), 8.02 (d, *J* = 8.5 Hz, 1H), 7.52 (d, *J* = 7.5 Hz, 1H), 7.42 (t, *J* = 8.0 Hz, 1H), 3.67 (t, *J* = 6.5 Hz, 2H), 2.81 (t, *J* = 7.5 Hz, 2H), 2.01 (br, 1H), 1.91 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 148.3, 143.9, 134.7, 129.2, 123.1, 121.0, 61.5, 33.6, 31.6.



5h

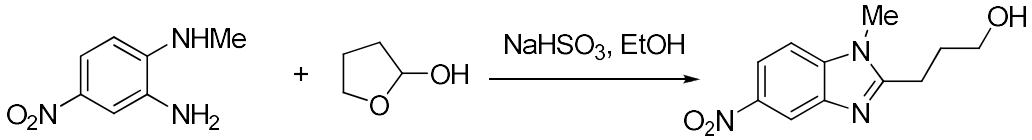
3-(4-(Trifluoromethyl)phenyl)propan-1-ol (**5h, Figure 3**). The title compound was prepared according to general procedure, as described above (253 mg, 62% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.53 (d, *J* = 8.0 Hz, 2H), 7.30 (d, *J* = 8.0 Hz, 2H), 3.67 (t, *J* = 6.5 Hz, 2H), 2.77 (t, *J* = 7.5 Hz, 2H), 1.89 (m, 2H), 1.85 (br, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 146.0, 128.7, 126.5, 125.3, 61.8, 33.8, 31.8.

3.2. Procedures for preparation of other 3-substituted aromatic propanols (5i-k)



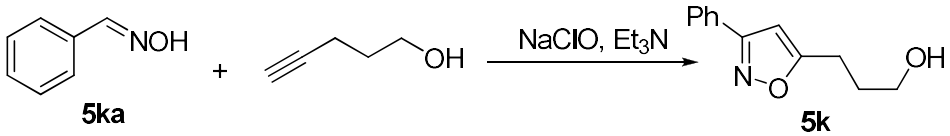
5i

3-(1-Benzyl-1H-1,2,3-triazol-4-yl)propan-1-ol (**5i, Figure 3**). BnN_3 (160 mg, 1.2 mmol) and pent-4-yn-1-ol **28** (84 mg, 1.0 mmol) were dissolved in *t*-BuOH (4 mL) and H_2O (4 mL), followed by addition of $CuSO_4 \cdot 5H_2O$ (25 mg, 0.1 mmol) and sodium ascorbate (99 mg, 0.5 mmol) in water (0.5 mL). The reaction was stirred at room temp for 2 h. Water was added and extracted with EtOAc for 3 times. The combined organic layers were washed with brine and dried over sodium sulfate and concentrated under reduced pressure. The crude product was purified by column chromatography to give 216 mg desired product **5i**, yield: 100%. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.33-7.21 (m, 5H), 5.45 (s, 2H), 3.64 (t, J = 6.5 Hz, 2H), 3.29 (br, 1H), 2.77 (t, J = 7.5 Hz, 2H), 1.87 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 134.7, 129.0, 128.6, 127.9, 61.4, 54.0, 31.9, 21.9. LR-MS (ESI): m/z 218.1 $[M+H]^+$.



5ja 5j

3-(1-Methyl-5-nitro-1H-benzo[*d*]imidazol-2-yl)propan-1-ol (**5j, Figure 3**). A solution of compound **5ja** (167 mg, 1.0 mmol) and tetrahydrofuran-2-ol[1] (88 mg, 1.0 mmol) in EtOH (6 mL) was added $NaHSO_3$ (104 mg, 1.0 mmol). The reaction was refluxed for 2 h. The solvent was removed under reduced pressure. The residue was added water and extracted with EtOAc for 3 times. The combined organic layers were washed with brine and dried over sodium sulfate and concentrated under reduced pressure. The crude product was purified by column chromatography to give 110 mg desired product **5j**, yield: 47%. ^{1}H NMR (300 MHz, $CDCl_3$): δ 8.39 (d, J = 2.1 Hz, 1H), 8.06 (dd, J_1 = 9.0 Hz, J_2 = 2.1 Hz, 1H), 7.29 (d, J = 9.0 Hz, 1H), 3.76 (s, 3H), 3.66 (t, J = 5.7 Hz, 2H), 2.98 (t, J = 7.2 Hz, 2H), 2.04 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 159.1, 143.2, 140.8, 139.5, 118.0, 114.8, 109.0, 60.7, 30.1, 29.3, 23.9. LR-MS (ESI): m/z 236.1 $[M+H]^+$.

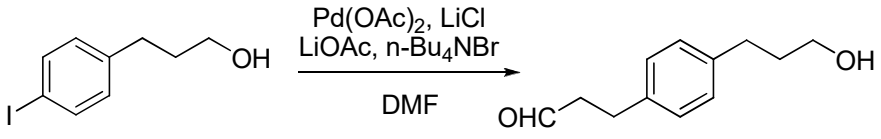


5ka 5k

3-(3-Phenylisoxazol-5-yl)propan-1-ol (5k, Figure 3). A solution of benzaldehyde oxime **5ka** (145 mg, 1.2 mmol), pent-4-yn-1-ol (84 mg, 1.0 mmol) and Et_3N (202 mg, 2.0 mmol) in CH_2Cl_2 (10 mL) was added aqueous NaClO (11-14% available chlorine, 10 mL). The mixture was stirred at rt for 10 h. Water was added and extracted with CH_2Cl_2 for 3 times. The combined organic layers were washed with brine and dried over sodium sulfate and concentrated under reduced pressure. The crude product was

purified by column chromatography to give 61 mg desired product, yield: 30%. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.60 (m, 2H), 7.41 (m, 3H), 6.30 (s, 1H), 3.70 (t, J = 6.0 Hz, 2H), 2.89 (t, J = 7.5 Hz, 2H), 2.50 (br, 1H), 1.97 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 173.6, 162.3, 129.8, 129.1, 128.8, 126.6, 99.1, 61.3, 30.2, 23.1. LR-MS (ESI): m/z 204.1 $[M+H]^+$.

3.3. Procedures for preparation of 3-substituted aromatic propanol 5m.

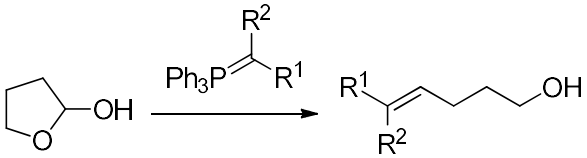


5ma **5m**

3-(4-(3-hydroxypropyl)phenyl)propanal (5m, Scheme 1). The title compound was prepared from **5ma**[3] (5 mmol) according to procedure described in section **3.1** in 88% yield (845 mg). ^{1}H NMR (500 MHz, $CDCl_3$): δ 9.81 (d, J = 1.0 Hz, 1H), 7.12 (m, 4H), 3.66 (t, J = 6.5 Hz, 2H), 2.93 (t, J = 7.5 Hz, 2H), 2.76 (t, J = 7.5 Hz, 2H), 2.67 (t, J = 7.5 Hz, 2H), 1.88 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 201.8, 139.8, 137.7, 128.6, 128.2, 62.1, 45.2, 34.2, 31.6, 27.6. LR-MS (ESI): m/z 193.1 $[M+H]^+$.

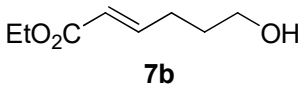
3.4. Procedures for preparation of substituted pent-4-en-1-ols 7.

3.4.1. General procedure for preparation of 7b-e from Wittig reactions between tetrahydrofuran-2-ol and Wittig reagents



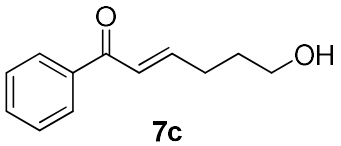
5jb **7b-e**

A solution of tetrahydrofuran-2-ol (176 mg, 2.0 mmol) and commercial available Wittig reagents (2.4 mmol) in CH_2Cl_2 (3 mL) was stirred at 45 °C until the reaction was completed (monitored with TLC). Direct column chromatography on silica gel gave the desired product.



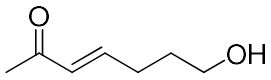
7b

(*E*)-Ethyl 6-hydroxyhex-2-enoate (**7b, Figure 4**). The title compound was prepared according to general procedure (reaction time: 6 h), as described above (149 mg, 47% yield). ^{1}H NMR (300 MHz, $CDCl_3$): δ 6.93 (m, 1H), 5.80 (d, J = 15.6 Hz, 1H), 4.13 (q, J = 7.2 Hz, 2H), 3.60 (t, J = 6.6 Hz, 2H), 2.38 (br, 1H), 2.26 (m, 2H), 1.67 (m, 2H), 1.24 (t, J = 7.2 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 166.8, 148.6, 121.6, 61.7, 60.2, 30.8, 28.5, 14.2. LR-MS (ESI): m/z 159.1 $[M+H]^+$.



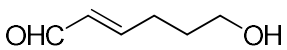
7c

(*E*)-6-Hydroxy-1-phenylhex-2-en-1-one (**7c, Figure 4**). The title compound was prepared according to general procedure (reaction time: 3 h), as described above (239 mg, 63% yield). ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.89 (d, *J* = 7.5 Hz, 2H), 7.51 (m, 1H), 7.43 (m, 2H), 7.05 (m, 1H), 6.89 (d, *J* = 15.3 Hz, 1H), 3.67 (t, *J* = 6.3 Hz, 2H), 2.54 (br, 1H), 2.40 (m, 2H), 1.77 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 191.1, 149.3, 137.8, 132.8, 128.5, 126.2, 61.8, 31.0, 29.2. LR-MS (ESI): m/z 191.1 $[M+H]^+$.



7d

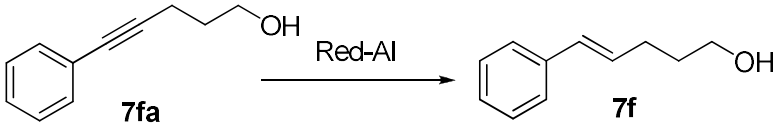
(*E*)-7-Hydroxyhept-3-en-2-one (**7d, Figure 4**). The title compound was prepared according to general procedure (reaction time: 6 h), as described above (200 mg, 78% yield) ^{1}H NMR (500 MHz, $CDCl_3$): δ 6.81 (m, 1H), 6.09 (d, *J* = 16.0 Hz, 1H), 3.67 (t, *J* = 6.0 Hz, 2H), 2.33 (m, 2H), 2.24 (s, 3H), 1.73 (m, 2H). LR-MS (ESI): m/z 129.1 $[M+H]^+$.



7e

(*E*)-6-Hydroxyhex-2-enal (**7e, Figure 4**). The title compound was prepared according to general procedure (reaction time: 5 h), as described above (80 mg, 35% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 9.51 (d, *J* = 7.5 Hz, 1H), 6.88 (m, 1H), 6.14 (dd, J_1 = 15.5 Hz, J_2 = 7.5 Hz, 1H), 3.71 (t, *J* = 6.0 Hz, 2H), 2.45 (m, 2H), 1.78 (m, 2H). LR-MS (ESI): m/z 115.0 $[M+H]^+$.

3.4.2. Preparation of 7f

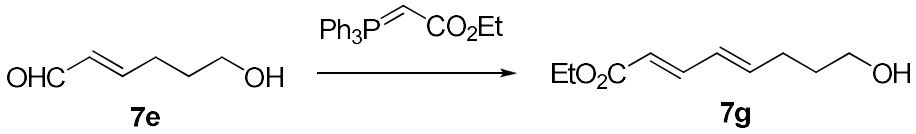


7fa **7f**

(*E*)-5-Phenylpent-4-en-1-ol (**7f, Figure 4**). A solution of compound **7fa** (80 mg, 0.5 mmol) in dry THF (5 mL) was added Red-Al (65% in toluene, 1 mL) at room temp. The mixture was stirred under Argon at 60 ^{o}C for 24 h. Water (20 mL) was added and extracted with EtOAc for 3 times. The combined organic layers were washed with brine and dried over sodium sulfate and concentrated under reduced pressure. The crude product was purified by column chromatography to give 65 mg desired product, yield: 82%. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.36-7.21 (m, 5H), 6.43 (d, *J* = 16.0 Hz, 1H), 6.24 (m, 1H), 3.71 (t, *J* = 6.3 Hz, 2H), 2.70 (br, 1H), 2.32 (m, 2H), 1.76 (m, 2H);

^{13}C NMR (125 MHz, $CDCl_3$): δ 137.5, 130.3, 130.0, 128.4, 126.9, 125.9, 62.2, 32.1, 29.2. LR-MS (ESI): m/z 163.1 $[M+H]^+$.

3.4.3. Preparation of 7g



7e 7g

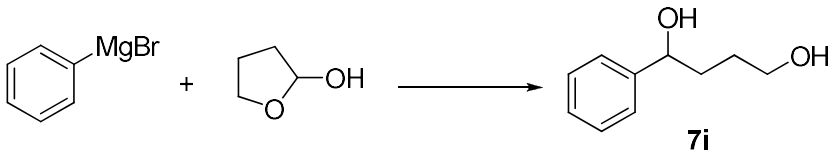
(2*E*,4*E*)-Ethyl 8-hydroxyocta-2,4-dienoate (**7g, Figure 4**). The title compound was prepared according to general procedure (reaction time: 15 h), as described above (302 mg, 82% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.20 (m, 1H), 6.12 (m, 2H), 5.74 (d, J = 16.0 Hz, 1H), 4.14 (q, J = 7.2 Hz, 2H), 3.60 (t, J = 6.0 Hz, 2H), 2.36 (br, 1H), 2.22 (m, 2H), 1.65 (m, 2H), 1.24 (t, J = 7.2 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 167.3, 144.8, 143.6, 128.6, 119.3, 61.8, 60.2, 31.4, 29.1, 14.1. LR-MS (ESI): m/z 185.1 $[M+H]^+$.

3.4.4. Preparation of 7h

7h

4-Hydroxybutanal O-benzyl oxime (**7h, Figure 4**). A solution of tetrahydrofuran-2-ol (106 mg, 1.2 mmol) in MeOH (1 mL) was added $BnONH_2$ (123 mg, 1.0 mmol). The reaction was stirred at rt for 5 h. Water was added and extracted with EtOAc for 3 times. The combined organic layers were washed with brine and dried over sodium sulfate and concentrated under reduced pressure. The crude product was purified by column chromatography to give 160 mg desired product, yield: 83%. isomer *E*:*Z*=2:1. ^{1}H NMR (500 MHz, $CDCl_3$) for *E* isomer: δ 7.50 (t, J = 5.5 Hz, 1H), 7.35 (m, 4H), 5.05 (s, 2H), 3.65 (t, J = 6.5 Hz, 2H), 2.31 (m, 2H), 1.75 (m, 2H); ^{1}H NMR (500 MHz, $CDCl_3$) for *Z* isomer: δ 7.35 (m, 4H), 6.73 (t, J = 5.5 Hz, 1H), 5.11 (s, 2H), 3.65 (t, J = 6.5 Hz, 2H), 2.47 (m, 2H), 1.75 (m, 2H). LR-MS (ESI): m/z 194.1 $[M+H]^+$.

3.4.5. Preparation of 7i

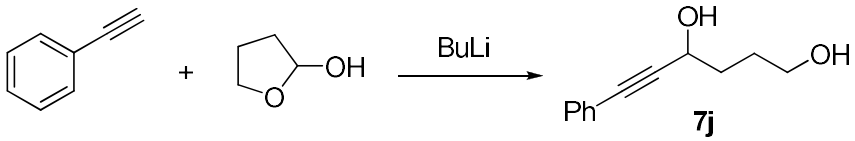


7i

1-Phenylbutane-1,4-diol (**7i, Figure 4**). A solution of tetrahydrofuran-2-ol (265 mg, 3.0 mmol) in dry THF (5 mL) was added benzene magnesium bromide (2 M in THF, 4.5 mL, 9.0 mmol) slowly at rt. After completion of addition, the reaction was stirred at rt for 20 min. Water was added and extracted with EtOAc for 3 times. The combined organic layers were washed with brine and dried over sodium sulfate and concentrated under reduced pressure. The crude product was purified by column chromatography to give 425 mg desired product, yield: 85%. ^{1}H NMR (500 MHz,

$CDCl_3$): δ 7.35 (m, 4H), 7.27 (m, 1H), 4.73 (t, *J* = 6.5 Hz, 1H), 3.67 (m, 2H), 2.6-2.1 (br, 2H), 1.86 (m, 2H), 1.68 (m, 2H). LR-MS (ESI): m/z 167.1 $[M+H]^+$.

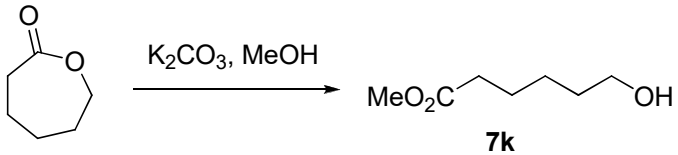
3.4.6. Preparation of 7j



7j

6-Phenylhex-5-yne-1,4-diol (**7j, Figure 4**). A solution of phenylacetylene (102 mg, 1.0 mmol) in dry THF (2 mL) was added BuLi (1.6 M in hexane, 0.63 mL, 1.0 mmol) slowly at 0 °C. Then tetrahydrofuran-2-ol (106 mg, 1.2 mmol) in dry THF (1 mL) was added within 10 min. After completion of addition, the reaction was stirred at 0 °C for 15 min. Water was added and extracted with EtOAc for 3 times. The combined organic layers were washed with brine and dried over sodium sulfate and concentrated under reduced pressure. The crude product was purified by column chromatography to give 38 mg desired product, yield: 20%. ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.41 (m, 2H), 7.28 (m, 3H), 4.66 (t, *J* = 5.7 Hz, 1H), 3.69 (m, 2H), 2.31 (br, 2H), 1.95-1.72 (m, 4H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 131.6, 128.3, 128.2, 122.6, 89.9, 84.8, 62.4, 34.8, 28.3. LR-MS (ESI): m/z 191.1 $[M+H]^+$.

3.4.7. Preparation of 7k

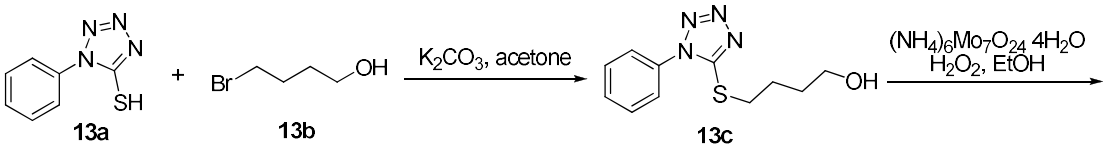


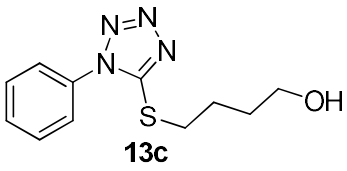
7k

Methyl 6-hydroxyhexanoate (**7k, Figure 4**). A solution of ε-caprolactone (1.14 g, 10 mmol) in MeOH (20 mL) was added K_2CO_3 (1.38 g, 10 mmol). The reaction was stirred at rt for 30 min. The solvent was removed under reduced pressure. The residue was added water and extracted with EtOAc for 3 times. The combined organic layers were washed with brine and dried over sodium sulfate and concentrated under reduced pressure. The crude product was purified by column chromatography to give 1.36 g desired product, yield: 93%. ^{1}H NMR (500 MHz, $CDCl_3$): δ 3.63 (s, 3H), 3.59 (t, *J* = 6.5 Hz, 2H), 2.29 (t, *J* = 7.5 Hz, 2H), 1.98 (br, 1H), 1.62 (m, 2H), 1.55 (m, 2H), 1.36 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 174.1, 62.4, 51.4, 33.9, 32.2, 25.2, 24.5. LR-MS (ESI): m/z 147.1 $[M+H]^+$.

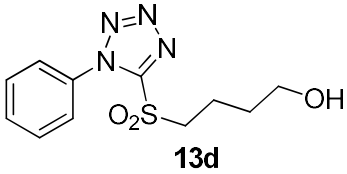
4. Homologous procedures to prepare polyene compounds 12 and 18

4.1. Preparation of Julia-Kocienski reagent 13.

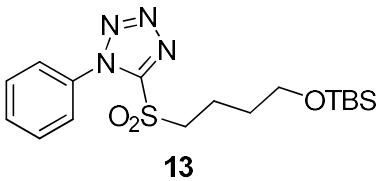




4-((1-Phenyl-1H-tetrazol-5-yl)thio)butan-1-ol (**13c**). A mixture of compound **13a** (5.34 g, 30 mmol), 4-bromobutanol **13b** (6.0 g, 39 mmol) and K_2CO_3 (8.3 g, 60 mmol) in acetone (100 mL) was stirred at 60 ^oC for 3.5 h. The reaction was cooled to room temp and filtered, washed with EtOAc. The combined organic layers were concentrated under reduced pressure. The crude product was purified by column chromatography to give 5.5 g desired product, yield: 71%. ^1H NMR (300 MHz, $CDCl_3$): δ 7.57 (m, 5H), 3.72 (t, J = 6.3 Hz, 2H), 3.43 (t, J = 7.5 Hz, 2H), 1.96 (m, 3H), 1.72 (m, 2H).



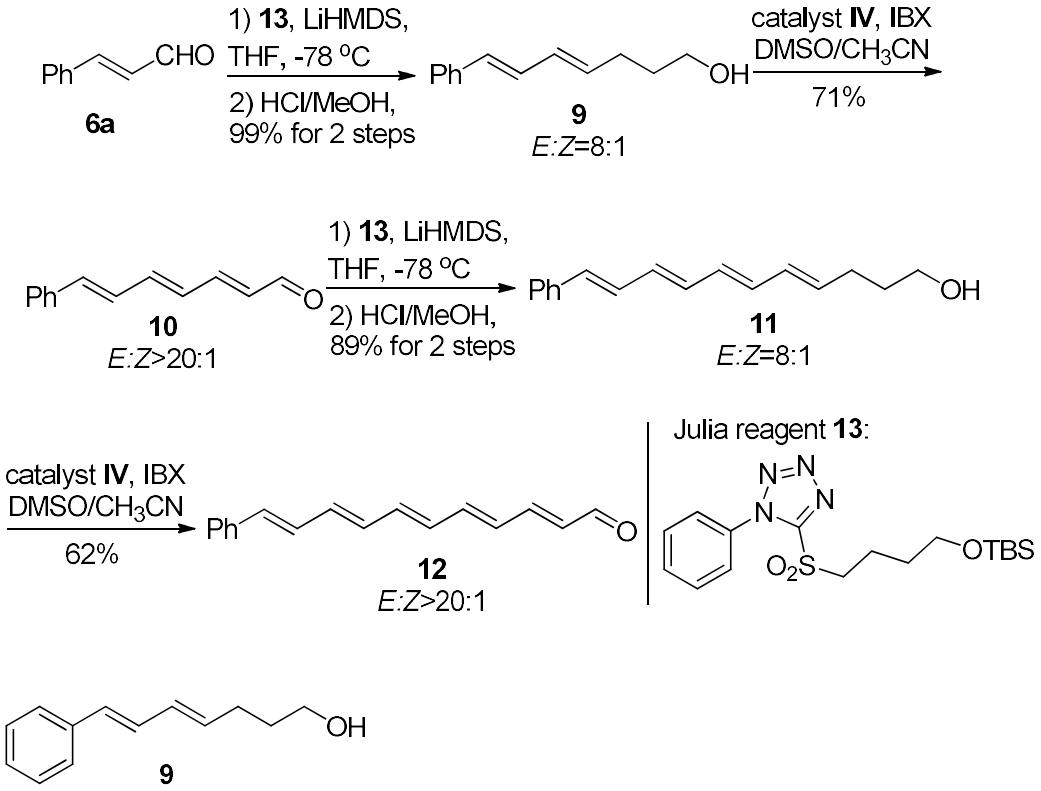
4-((1-Phenyl-1H-tetrazol-5-yl)sulfonyl)butan-1-ol (**13d**). A solution of compound **13c** (2.0 g, 8.0 mmol) in EtOH (32 mL) was added $(NH_4)_6Mo_7O_{24}·4H_2O$ (2.0 g, 1.6 mmol), followed by addition of H_2O_2 (35% aq., 16 g, 160 mmol) within 20 min. The reaction was stirred at room temp for 15 h. Water was added and extracted with EtOAc for 3 times. The combined organic layers were washed with aqueous $NaHSO_3$, water, brine and dried over sodium sulfate and concentrated under reduced pressure. The crude product was used directly in the next step without purification. ^1H NMR (300 MHz, $CDCl_3$): δ 7.64-7.52 (m, 5H), 3.75 (t, J = 7.8 Hz, 2H), 3.63 (t, J = 6.3 Hz, 2H), 2.68 (br, 1H), 2.01 (m, 2H), 1.69 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 153.4, 132.9, 131.5, 129.7, 125.2, 61.4, 55.7, 30.5, 19.0.



5-((4-((*tert*-Butyldimethylsilyl)oxy)butyl)sulfonyl)-1-phenyl-1H-tetrazole (**13**).

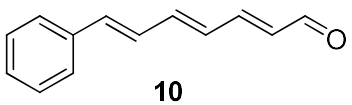
The above crude product **13d** was dissolved in DMF (6 mL). TBSCl (1.4 g, 9.3 mmol) and imidazole (800 mg, 11.7 mmol) were added. The mixture was stirred at rt for 30 min. Water was added and extracted with EtOAc for 3 times. The combined organic layers were washed with water and brine and dried over sodium sulfate and concentrated under reduced pressure. The crude product was purified by column chromatography to give 2.9 g desired product, yield: 91% for two steps. ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.67 (m, 2H), 7.59 (m, 3H), 3.80 (t, J = 7.8 Hz, 2H), 3.66 (t, J = 6.0 Hz, 2H), 2.05 (m, 2H), 1.71 (m, 2H), 0.87 (s, 9H), 0.04 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 153.4, 133.0, 131.4, 129.6, 125.0, 62.0, 55.8, 30.8, 25.8, 19.1, 18.2, -5.5. HR-MS (ESI) m/z $[M + H]^+$ calcd for $C_{17}H_{29}N_4O_3SSi$ 397.1730, found 397.1734.

4.2. Preparation of polyene 12



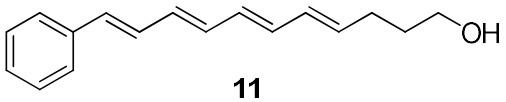
(4*E*,6*E*)-7-Phenylhepta-4,6-dien-1-ol (**9, Scheme 2**). A solution of compound **13** (1.8 g, 4.54 mmol) in dry THF (30 mL) was cooled to -78 °C under argon. LiHMDS (1.0 M in hexane, 5.3 mL) was added through syringe within 20 min. The reaction was stirred at -78 °C for 20 min before a solution of **6a** (900 mg, 6.8 mmol) in THF (15 mL) was added slowly. The reaction temperature was allowed to rise to room temp within three hours. Water was added to quench the reaction and THF was removed under reduced pressure. The residue was extracted with EtOAc for 3 times. The combined organic layers were washed with brine and dried over sodium sulfate and concentrated under reduced pressure. The crude product was dissolved in MeOH (20 mL), hydrochloric acid (12 N, 2 mL) was added slowly and stirred at rt for 10 min. Brine was added and extracted with EtOAc for 3 times. The combined organic layers

were washed with brine and dried over sodium sulfate and concentrated under reduced pressure. The crude product was purified by column chromatography to give 860 mg desired product **9** (*E*:*Z*=8:1), yield: 99% for two steps. ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.38 (m, 2H), 7.30 (m, 2H), 7.21 (m, 1H), 6.76 (dd, J_1 = 15.6 Hz, J_2 = 10.5 Hz, 1H), 6.46 (d, J = 15.9 Hz, 1H), 6.25 (dd, J_1 = 15.0 Hz, J_2 = 10.5 Hz, 1H), 5.84 (m, 1H), 3.68 (t, J = 6.6 Hz, 2H), 2.25 (m, 2H), 1.72 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 137.5, 134.6, 131.0, 130.4, 129.1, 128.5, 127.1, 126.1, 62.3, 32.1, 29.1.



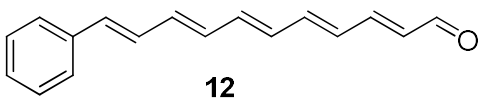
10

(2*E*,4*E*,6*E*)-7-Phenylhepta-2,4,6-trienal (**10, Scheme 2**). A solution of alcohol **9** (4.5 mmol) in DMSO (9.0 mL) and CH_3CN (13.5 mL) was added IBX (3.15 g, 11.25 mmol, 2.5 equiv.) and catalyst **IV** (228 mg, 0.9 mmol, 20 mol %). The resulting mixture was then stirred at room temp for 11 h. After evaporating the volatiles, the crude products was first checked on ^{1}H-NMR for geometric E/Z ratio, and then direct column chromatography on silica gel using a mixture of hexane and ethyl acetate gave 588 mg desired product **10** (*E*:*Z*>20:1), yield: 71%. ^{1}H NMR (500 MHz, $CDCl_3$): δ 9.58 (d, J = 7.5 Hz, 1H), 7.45 (d, J = 8.0 Hz, 2H), 7.35 (m, 2H), 7.29 (m, 1H), 7.17 (m, 1H), 6.93-6.79 (m, 3H), 6.57 (m, 1H), 6.19 (dd, J_1 = 15.0 Hz, J_2 = 8.0 Hz, 1H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 193.4, 151.6, 142.7, 138.3, 136.3, 131.2, 130.1, 128.8, 127.7, 127.0. HR-MS (ESI) m/z $[M + H]^+$ calcd for $C_{13}H_{13}O$ 185.0966, found 185.0957.



11

(4*E*,6*E*,8*E*,10*E*)-11-Phenylundeca-4,6,8,10-tetraen-1-ol (**11, Scheme 2**). The title compound was prepared according to the procedure described in preparation of compound **9** in 89% yield (427 mg, *E*:*Z*=8:1). ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.39 (m, 2H), 7.31 (m, 2H), 7.20 (m, 1H), 6.84 (m, 1H), 6.54 (d, J = 15.6 Hz, 1H), 6.43-6.11 (m, 5H), 5.76 (m, 1H), 3.67 (t, J = 6.3 Hz, 2H), 2.23 (m, 2H), 1.69 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 137.5, 134.8, 133.6, 133.4, 132.6, 132.1, 131.2, 129.3, 128.6, 127.4, 126.3, 62.4, 32.2, 29.2.

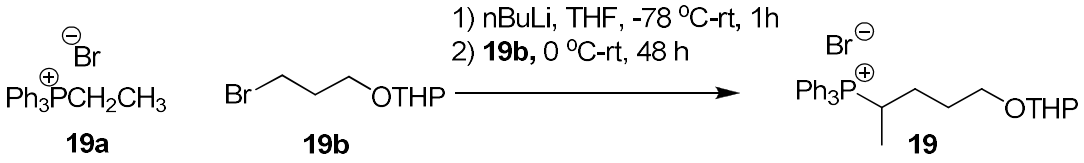


12

(2*E*,4*E*,6*E*,8*E*,10*E*)-11-Phenylundeca-2,4,6,8,10-pentaenal (**12, Scheme 2**). A solution of alcohol **11** (0.75 mmol) in DMSO (1.5 mL) and CH_3CN (2.25 mL) was added IBX (525 g, 1.88 mmol, 2.5 equiv.) and catalyst **IV** (38 mg, 0.15 mmol, 20 mol %). The resulting mixture was then stirred at room temp for 10 h. After evaporating the volatiles, the crude products was first checked on ^{1}H-NMR for geometric E/Z ratio, and then direct column chromatography on silica gel using a

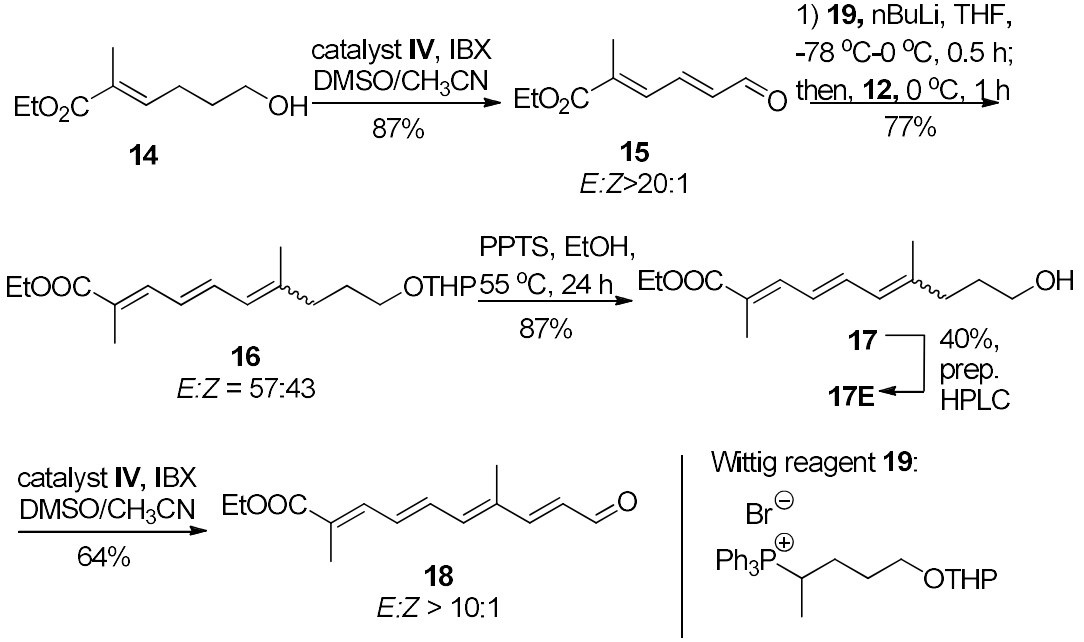
mixture of hexane and ethyl acetate gave 110 mg desired product **12** (*E*:*Z*>20:1), yield: 62%. ^{1}H NMR (300 MHz, $CDCl_3$): δ 9.57 (d, *J* = 7.5 Hz, 1H), 7.42 (d, *J* = 7.2 Hz, 2H), 7.34 (m, 2H), 7.24 (m, 1H), 7.15 (m, 1H), 6.88 (m, 1H), 6.78-6.35 (m, 7H), 6.16 (dd, J_1 = 15.3 Hz, J_2 = 8.1 Hz, 1H). ^{13}C NMR (75 MHz, $CDCl_3$:CD_3OD=10:1): δ 193.8, 152.2, 142.9, 139.0, 137.0, 136.8, 134.8, 132.6, 131.7, 130.7, 129.8, 128.7, 128.0, 126.6. HR-MS (ESI) m/z $[M + H]^+$ calcd for $C_{17}H_{17}O$ 237.1279, found 237.1288.

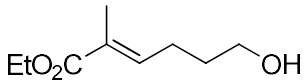
4.3. Preparation of Wittig reagent 19.[2]



A solution of ethyl triphenyl phosphonium bromide **19a** (1.5 g, 4 mmol) in dry THF (10 mL) was cooled to -78 °C under argon. nBuLi (2.75 mL, 4.4 mmol, 1.6 M in hexane) was added through syringe within 10 min. The reaction mixture was allowed to rise to room temp in 1 h and became dark red. The reaction was then cooled to 0 °C and **19b** (1.1 g, 5 mmol) in THF (2 mL) was added dropwise. The reaction was then warmed to room temp and stirred for 48 h. The resulting solution was recrystallized dichloromethane and hexane to give the product as colorless solid. In the event that no solid was formed, the upper solvent phase (a mixture of dichloromethane and hexane) was discarded and the lower oil-like phase was collected and concentrated to give colorless solid after vacuum overnight. (1.8 g, 90% yield). The NMR data is accordance with the reference.[2]

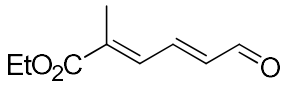
4.4. Preparation of polyene 12





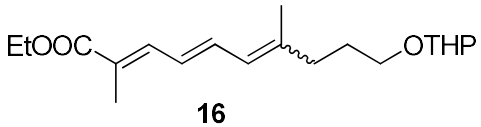
14

(*E*)-Ethyl 6-hydroxy-2-methylhex-2-enoate (**14, Scheme 3**). A solution of tetrahydrofuran-2-ol (880 mg, 10 mmol) and (Carbethoxyethylidene) triphenylphosphorane (4.34 g, 12 mmol) in CH_2Cl_2 (20 mL) was stirred at rt for 16 h until the reaction was completed monitored with TLC. After evaporating the volatiles, the residue was purified on silica gel to get the product as a colorless oil (1.41 g, 82%). ^{1}H NMR (300 MHz, $CDCl_3$): δ 6.71 (t, J = 7.5 Hz, 1H), 4.13 (q, J = 7.2 Hz, 2H), 3.60 (t, J = 6.6 Hz, 2H), 2.40 (br, 1H), 2.22 (m, 2H), 1.79 (s, 3H), 1.66 (m, 2H), 1.24 (t, J = 7.2 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 168.3, 141.5, 128.2, 62.0, 60.5, 31.4, 25.0, 14.2, 12.3. HR-MS (ESI) m/z $[M + H]^+$ calcd for $C_9H_{17}O_3$ 173.1178, found 173.1177.



15

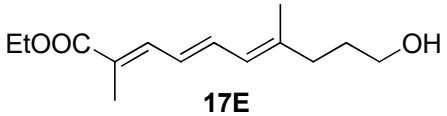
(2*E*,4*E*)-Ethyl 2-methyl-6-oxohexa-2,4-dienoate (**15, Scheme 3**). A solution of alcohol **14** (1.03 g, 6.0 mmol) in DMSO (12.0 mL) and CH_3CN (18.0 mL) was added IBX (4.20 g, 15.0 mmol, 2.5 equiv.) and catalyst **IV** (304 mg, 1.2 mmol, 20 mol %). The resulting mixture was then stirred at room temp for 5 h. After evaporating the volatiles, the crude products was first checked on ^{1}H-NMR for geometric E/Z ratio, and then direct column chromatography on silica gel using a mixture of hexane and ethyl acetate gave 877 mg desired product **15** (*E*:*Z*>20:1), yield: 87%. ^{1}H NMR (300 MHz, $CDCl_3$): δ 9.68 (d, J = 7.8 Hz, 1H), 7.46-7.26 (m, 2H), 6.37 (dd, J_1 = 15.0 Hz, J_2 = 7.8 Hz, 1H), 4.25 (q, J = 7.2 Hz, 2H), 2.10 (s, 3H), 1.32 (t, J = 7.2 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 193.2, 167.0, 145.0, 137.1, 135.9, 134.0, 61.3, 14.2, 13.5. HR-MS (ESI) m/z $[M + H]^+$ calcd for $C_9H_{13}O_3$ 169.0865, found 169.0860.



16

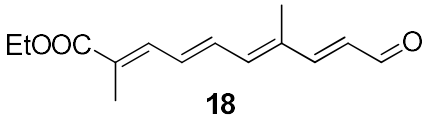
(4E/Z)-ethyl 2,7-dimethyl-10-(tetrahydro-2H-pyran-2-yloxy)deca-2,4,6-trienoate (**16, Scheme 3).** A solution of phosphonium bromide **19** (2.1 g, 4 mmol) in dry THF (10 mL) was cooled to -78 ^{o}C under argon. nBuLi (2.25 mL, 3.6 mmol, 1.6 M in hexane) was added through syringe within 10 min. The reaction mixture was brought to 0 ^{o}C and stirred for 30 min until the solution became dark red. A solution of **15** (340 mg, 2 mmol) in THF (5 mL) was added dropwise at 0 ^{o}C with stirring for 1 h. A saturated NH_4Cl aqueous solution was added to quench the reaction and the resulting mixture was extracted with EtOAc (2 x 20 mL). The combined organic layers were washed with brine, dried over sodium sulfate, and concentrated. The residue was purified on silica gel to get the product as pale yellow oil (500 mg, 77% yield, E/Z: 57/43). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.25-7.21 (m, 1H), 6.79-6.71 (m, 1H), 6.39-6.31 (m, 1H), 6.00 (d, J =11.2 Hz, 1H), 4.55-4.52 (m, 1H), 4.17 (dd, J_1 = 2.0, J_2

= 7.2 Hz, 2H), 3.86-3.81 (m, 1H), 3.70-3.68 (m, 1H), 3.49-3.46 (m, 1H), 3.37-3.30 (m, 1H), 2.36-2.16 (m, 2H), 1.91 (s, 3H), 1.83 (s, 1.63 H), 1.82 (s, 1.20 H), 1.76-1.51 (m, 6 H), 1.27 (dt, J_1 = 1.2, J_2 = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 168.64, 168.61, 144.04, 143.80, 138.95, 138.93, 136.03, 135.88, 126.47, 125.78, 125.72, 125.66, 125.63, 125.36, 99.06, 98.99, 67.12, 66.68, 62.46, 62.42, 60.51, 60.49, 30.86, 30.82, 29.17, 28.26, 27.94, 25.56, 24.17, 19.74, 19.72, 17.12, 14.43, 12.71. HR-MS (ESI) m/z $[M + H]^+$ calcd for $C_{19}H_{31}O_4$ 323.2222, found 323.2234.



17E

(2E,4E,6E)-ethyl 10-hydroxy-2,7-dimethyldeca-2,4,6-trienoate (17E, Scheme 3). A solution of ether **16** (483 mg, 1.5 mmol) and PPTS (75 mg, 0.3 mmol) in EtOH (15 mL) was heated at 55 °C for 24 h. The reaction was neutralized by the addition of saturated aqueous $NaHCO_3$ solution and extracted with EtOAc (2 x 20 mL). The combined organic layers were washed with brine, dried over sodium sulfate, and concentrated. The residue was purified on silica gel to get the mixture of E and Z products as colorless oil (300 mg, 87% yield). The product was further purified on Supelco C18 reverse HPLC (25 cm X 21.2 mm, 10 uM) to give the product **17E** as colorless oil (70 mg, 40% yield based on the E/Z ratio of crude product **17**). (10 mL/min, 40%-50% CH_3CN in water 20 min, 50% 5 min, 50-40% 2 min, t_{17E} = 20.38 min). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.26-7.23 (m, 1H), 6.74 (t, J = 12.0 Hz, 1H), 6.38 (t, J = 12.8 Hz, 1H), 6.00 (d, J =11.6 Hz, 1H), 4.20 (q, J = 6.8 Hz, 2H), 3.63 (t, J = 6.0 Hz, 2H), 2.19 (t, J = 7.6 Hz, 2H), 1.93 (s, 3H), 1.83 (s, 3H), 1.72-1.70 (m, 3H), 1.29 (t, J = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 168.7, 143.6, 138.9, 135.9, 126.0, 125.9, 125.5, 62.6, 60.6, 36.5, 30.8, 17.2, 14.5, 12.8. HR-MS (ESI) m/z $[M + H]^+$ calcd for $C_{14}H_{23}O_3$ 239.1647, found 239.1660.



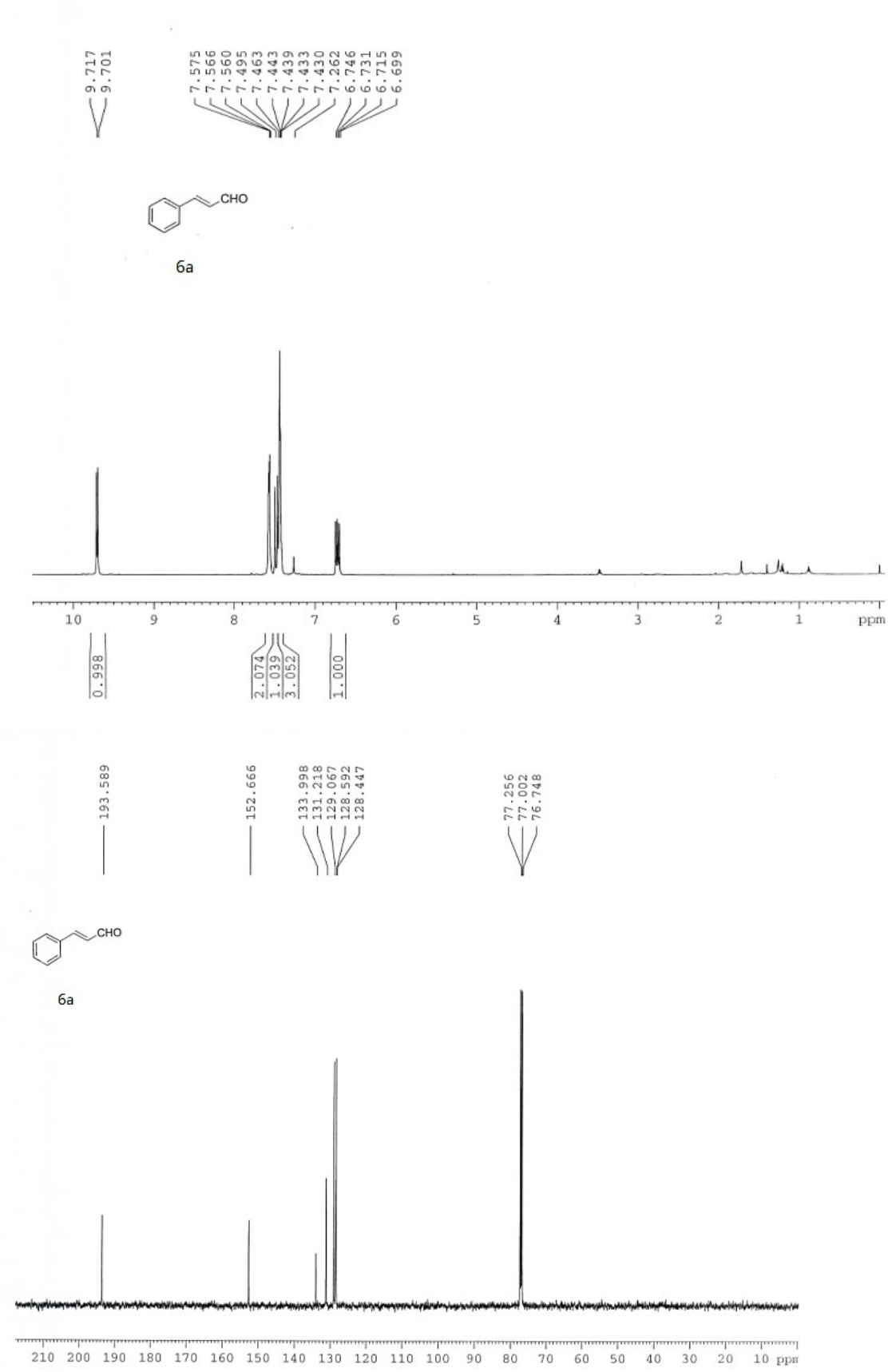
18

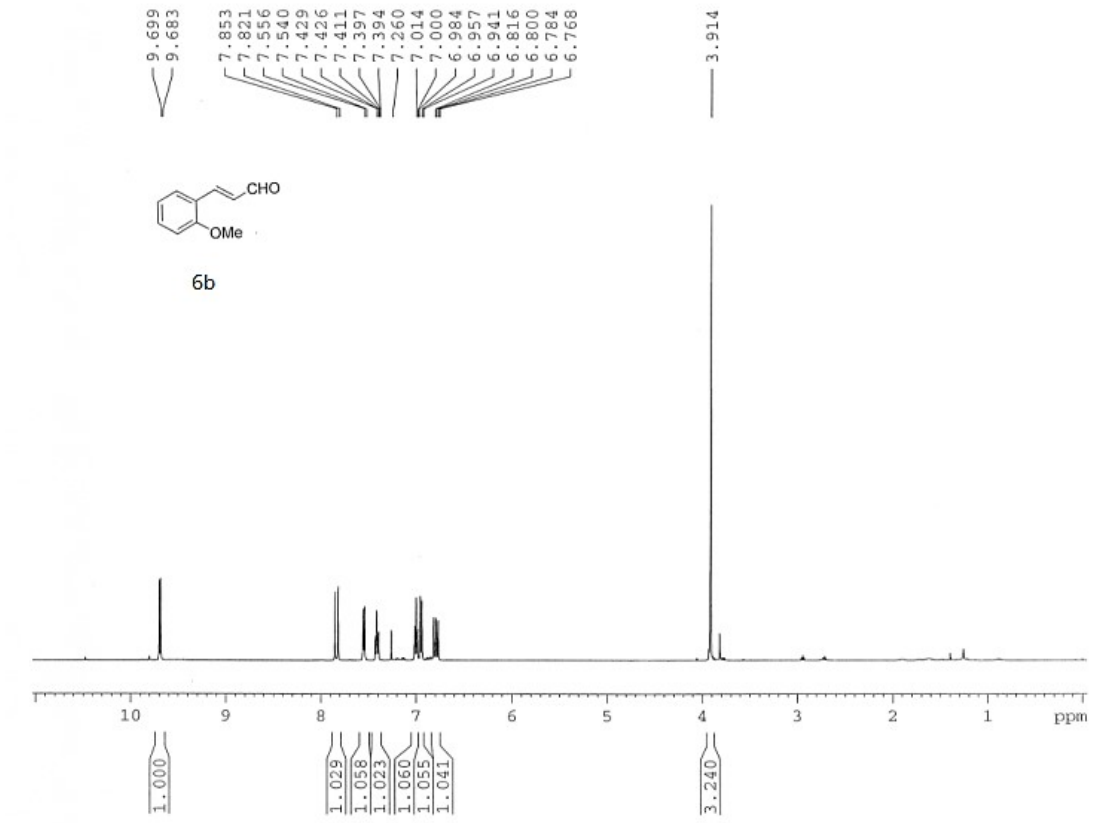
(4E,6E,8E)-ethyl 2,7-dimethyl-10-oxodeca-2,4,6,8-tetraenoate (18, Scheme 3). A solution of alcohol **17E** (60 mg, 0.25 mmol) in DMSO (0.5 mL) and CH_3CN (0.75 mL) was added IBX (175 mg, 0.625 mmol, 2.5 equiv.) and catalyst **IV** (12.7 mg, 0.05 mmol, 20 mol %). The resulting mixture was then stirred at room temp for 16 h. After evaporating the volatiles, the crude products was first checked on ^{1}H-NMR for geometric E/Z ratio, and then direct column chromatography on silica gel using a mixture of hexane and ethyl acetate gave 37.5 mg desired product **18** (*E*:*Z*>10:1), yield: 64%. ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.61 (d, J = 8.0 Hz, 1 H), 7.29 (d, J = 11.6 Hz, 1H), 7.16 (d, J = 15.6 Hz, 1H), 6.86 (dd, J_1 = 11.6, J_2 = 14.4 Hz, 1H), 6.75 (dd, J_1 = 11.6, J_2 = 14.4 Hz, 1H), 6.61 (d, J = 11.6 Hz, 1H), 6.25 (dd, J_1 = 8.0, J_2 = 15.6 Hz, 1H), 4.23 (q, J = 7.2 Hz, 2H), 2.02 (s, 3H), 2.00 (s, 3H), 1.32 (t, J = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 193.8, 168.1, 155.9, 139.5, 137.4, 136.9, 134.0, 133.1, 130.2, 128.8, 61.0, 14.5, 13.2, 13.0. HR-MS (ESI) m/z $[M + H]^+$ calcd for $C_{14}H_{19}O_3$ 235.1334, found 235.1339.

5. References

1. T. Saito, Y. Nishimoto, M. Yasuda, A. Baba, *J. Org. Chem.* **2006,** *71,* 8516.
2. A. Marinier, P. Deslongchamps, *Can. J. Chem.*, **1992**, *70*, 2350.
3. S-Y. Cheung, H-F. Chow, T. Ngai, X. Wei, *Chem. Eur. J.* **2009**, *15*, 2278-2288.

6. ^{1}H and ^{13}C NMR spectra of compounds 5-18

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6.957
6.941
6.816
6.800
6.784
6.768
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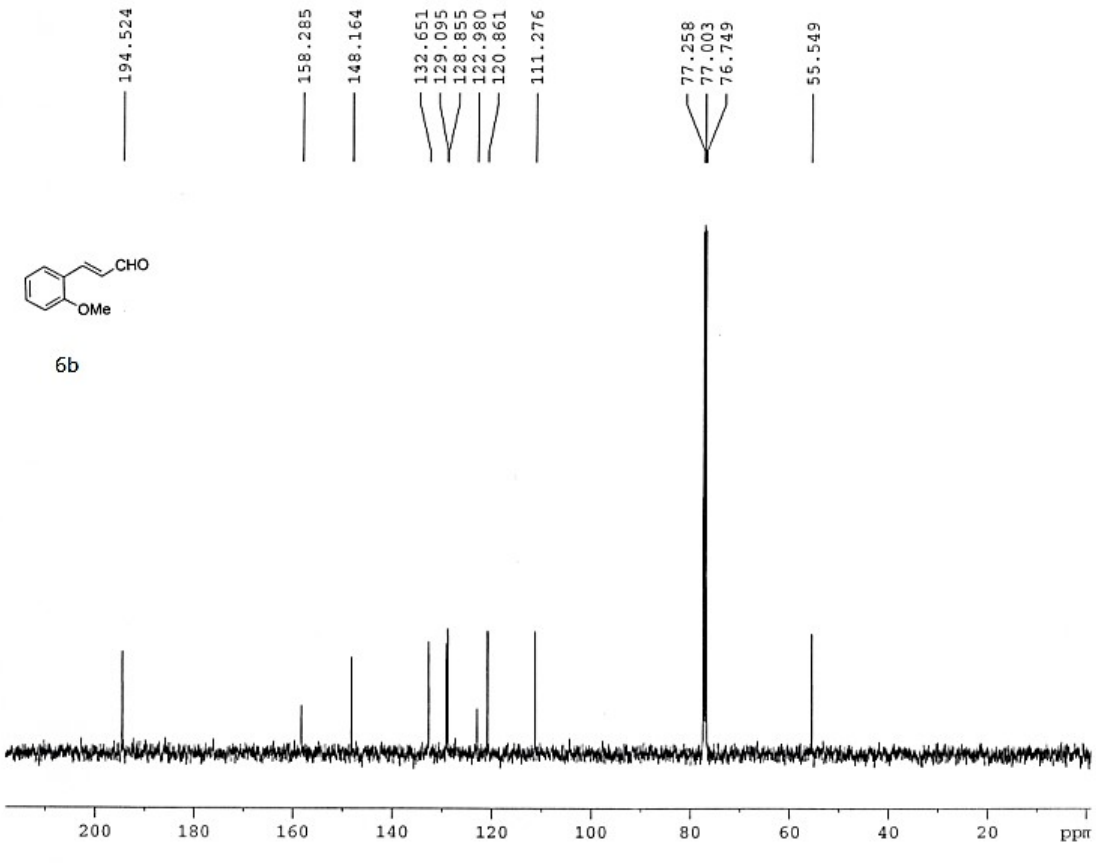
3.240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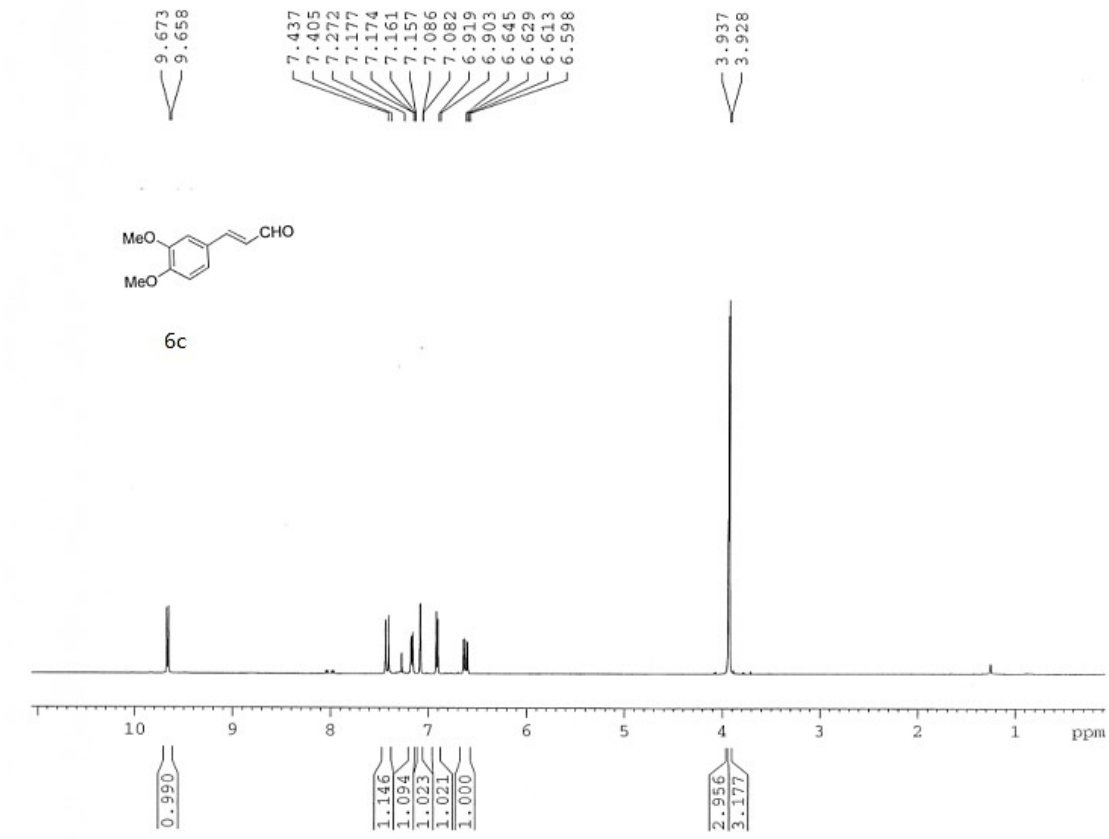
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6.598
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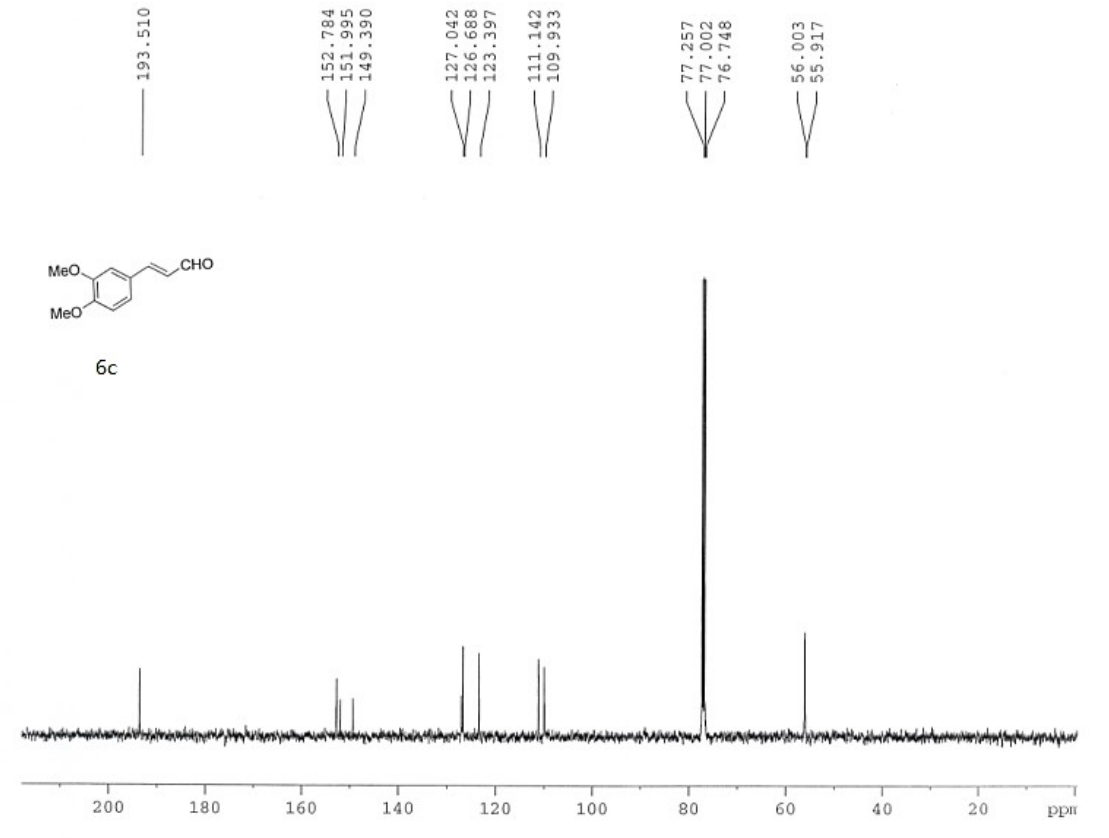
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6c


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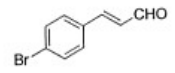
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Br

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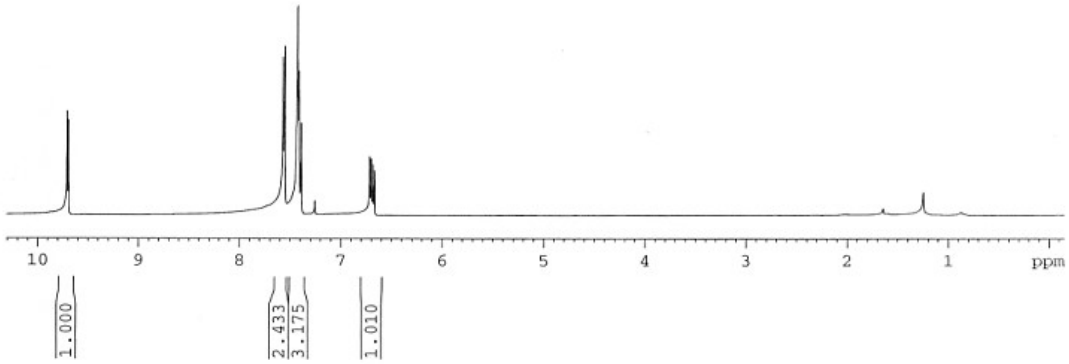
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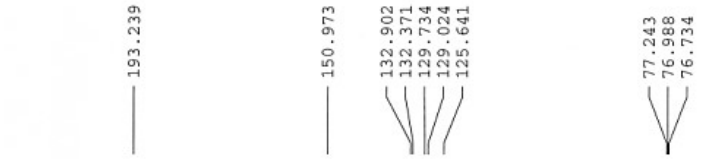
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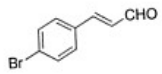
76.734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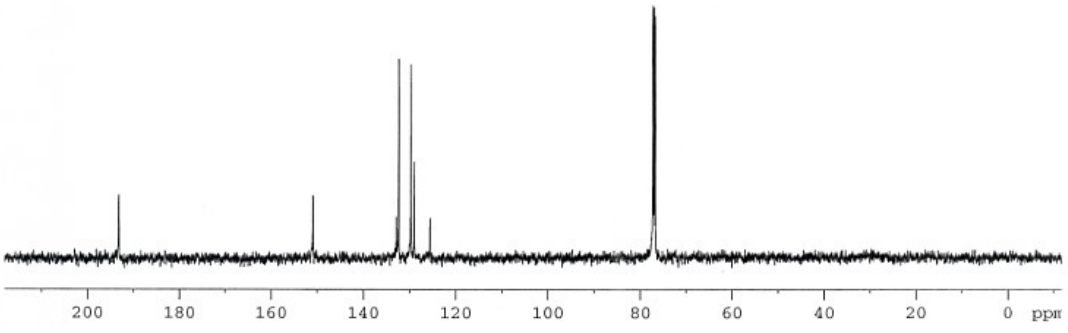
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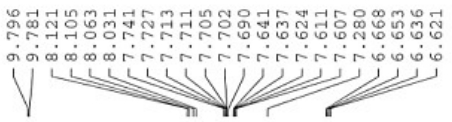
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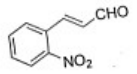
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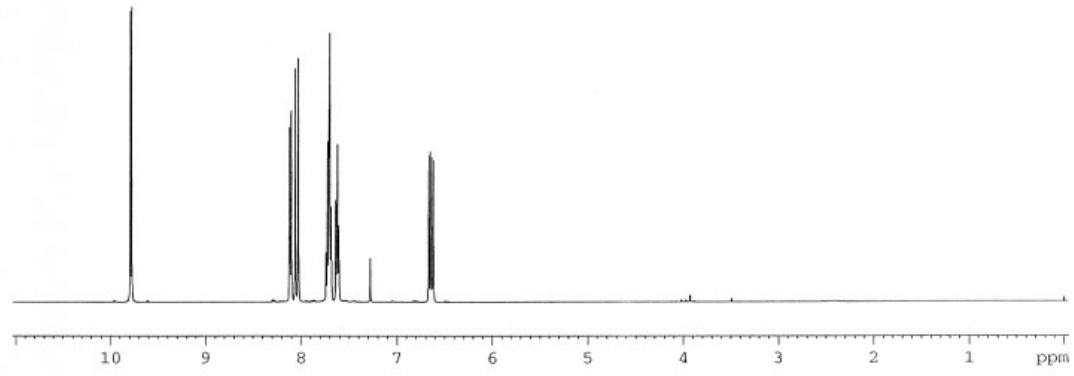
6e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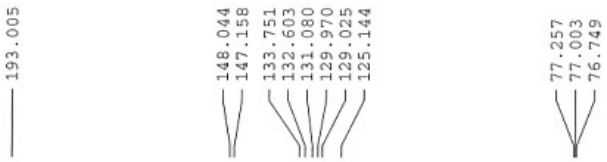
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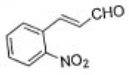
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6e

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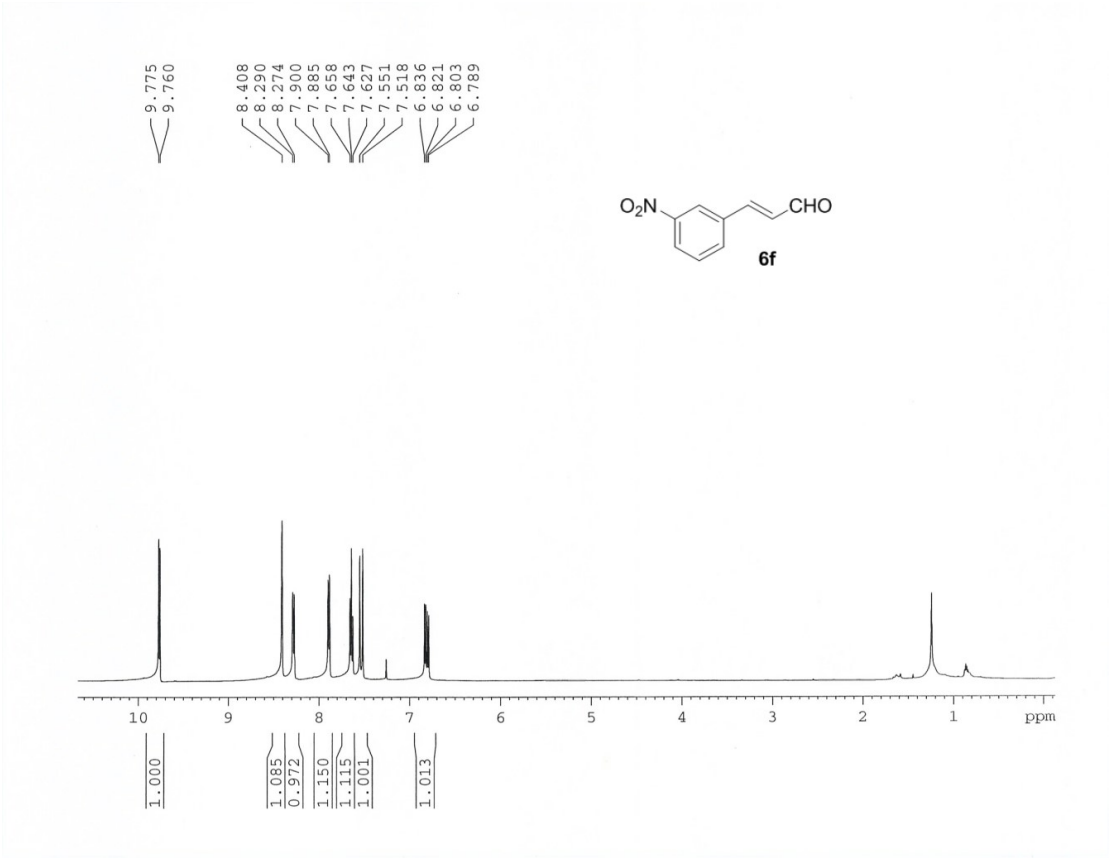
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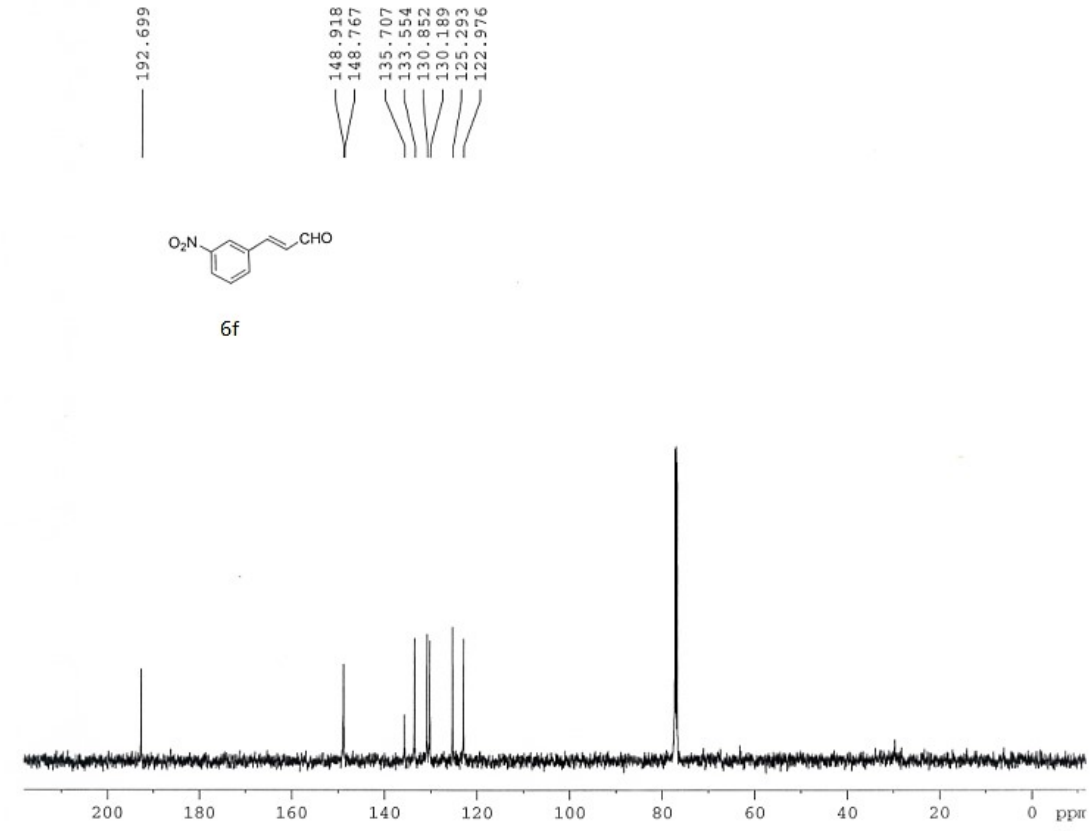
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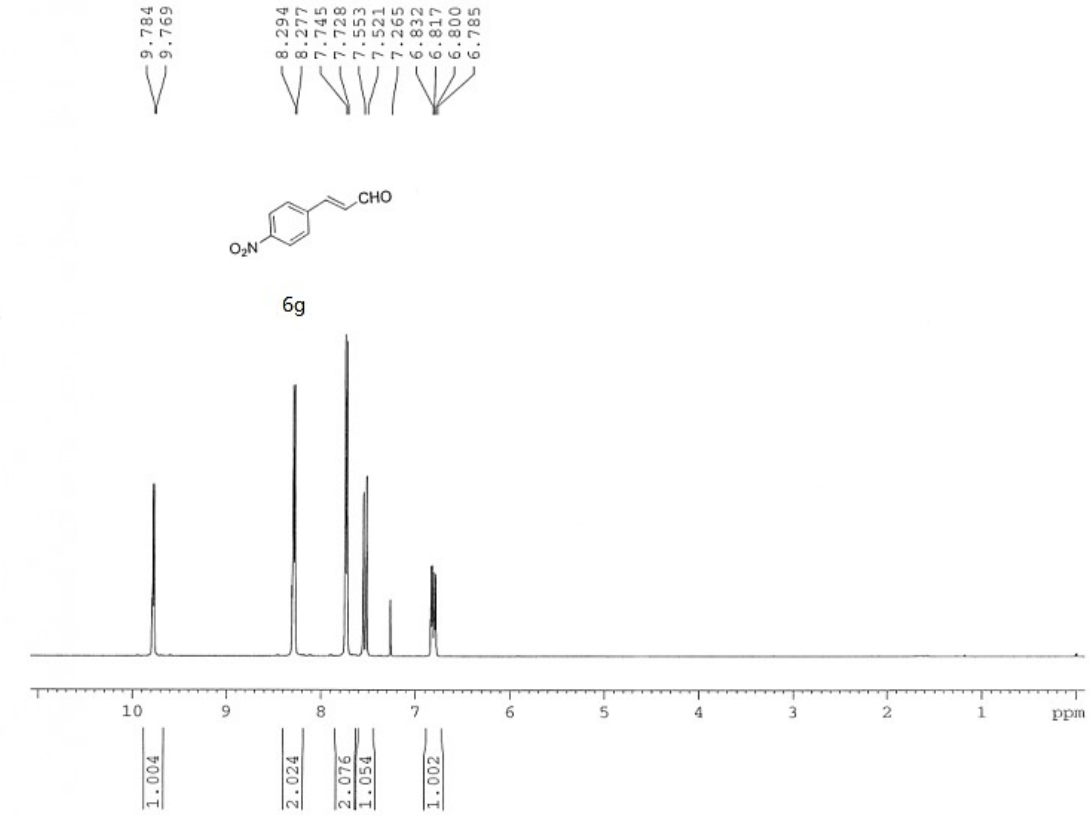
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4
3
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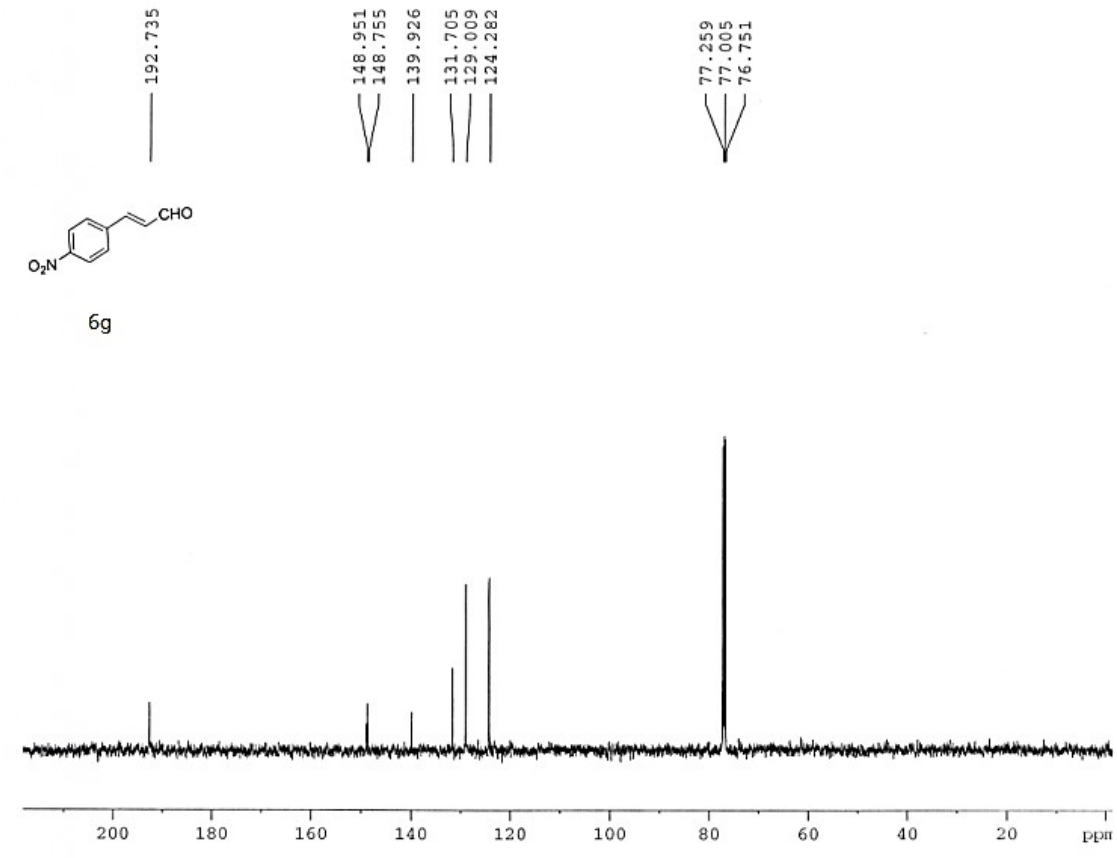
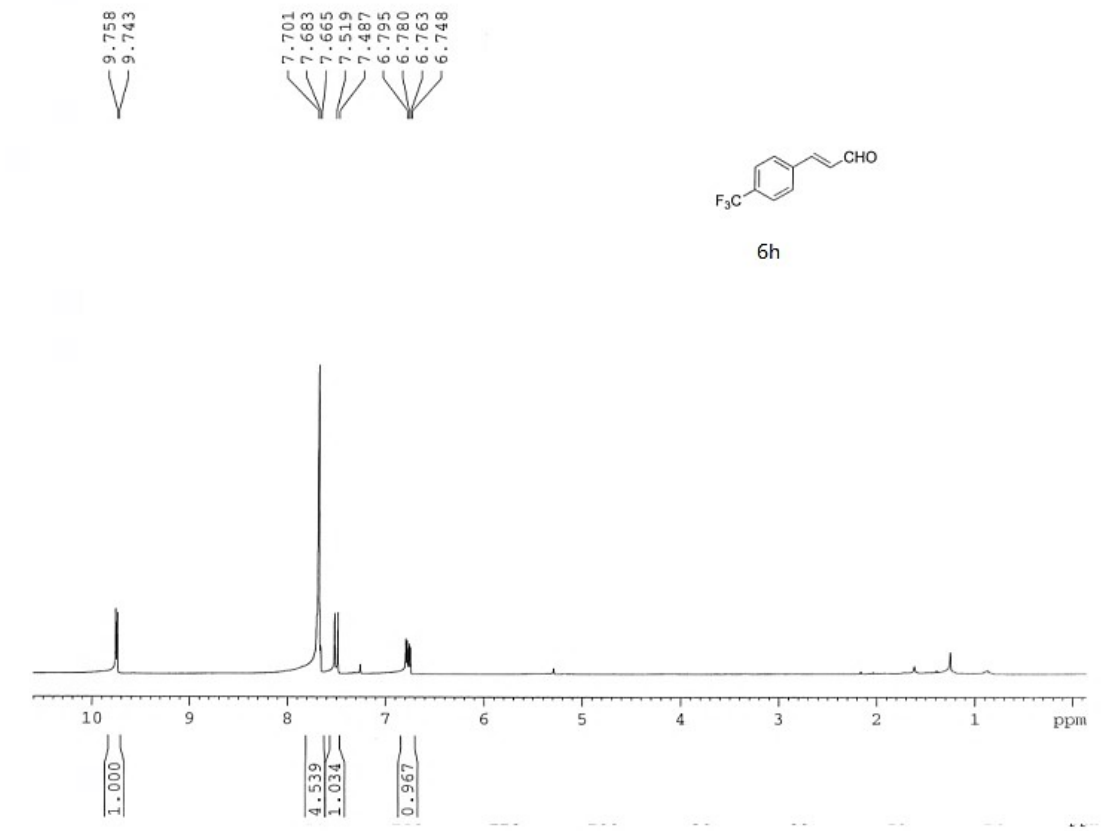
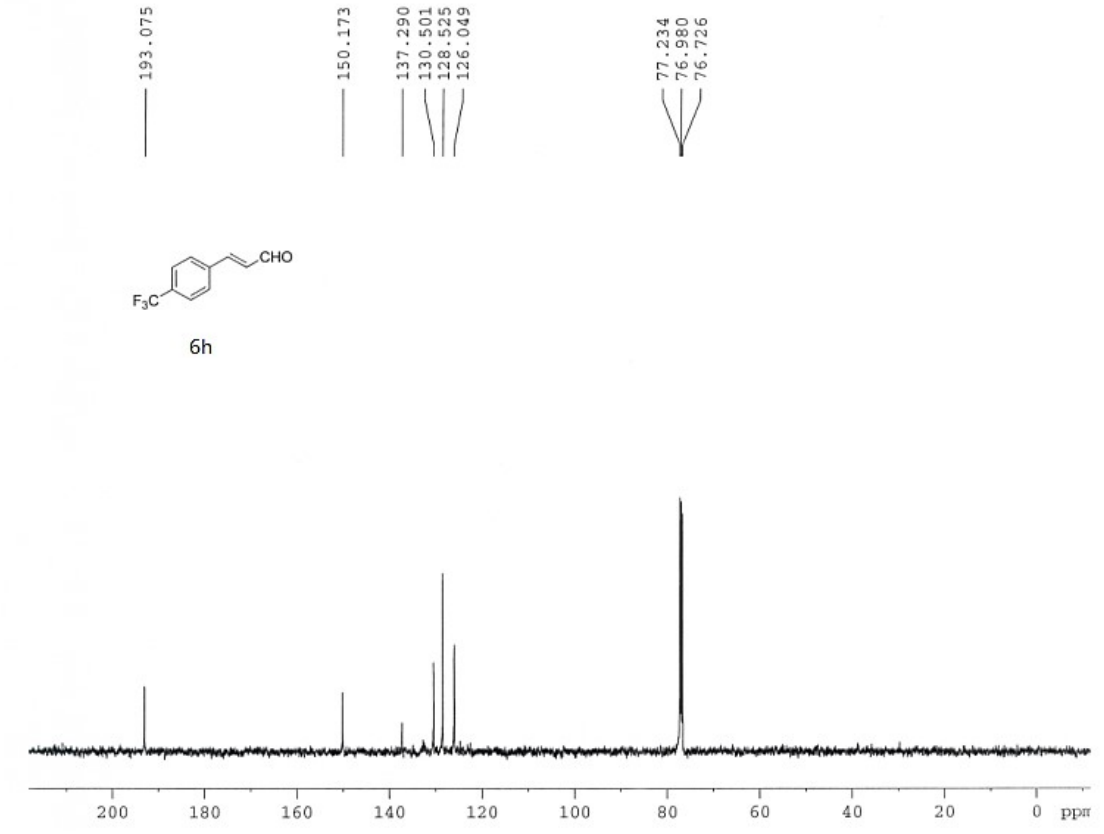
ppm
1.004
2.024
2.076
1.054
1.002

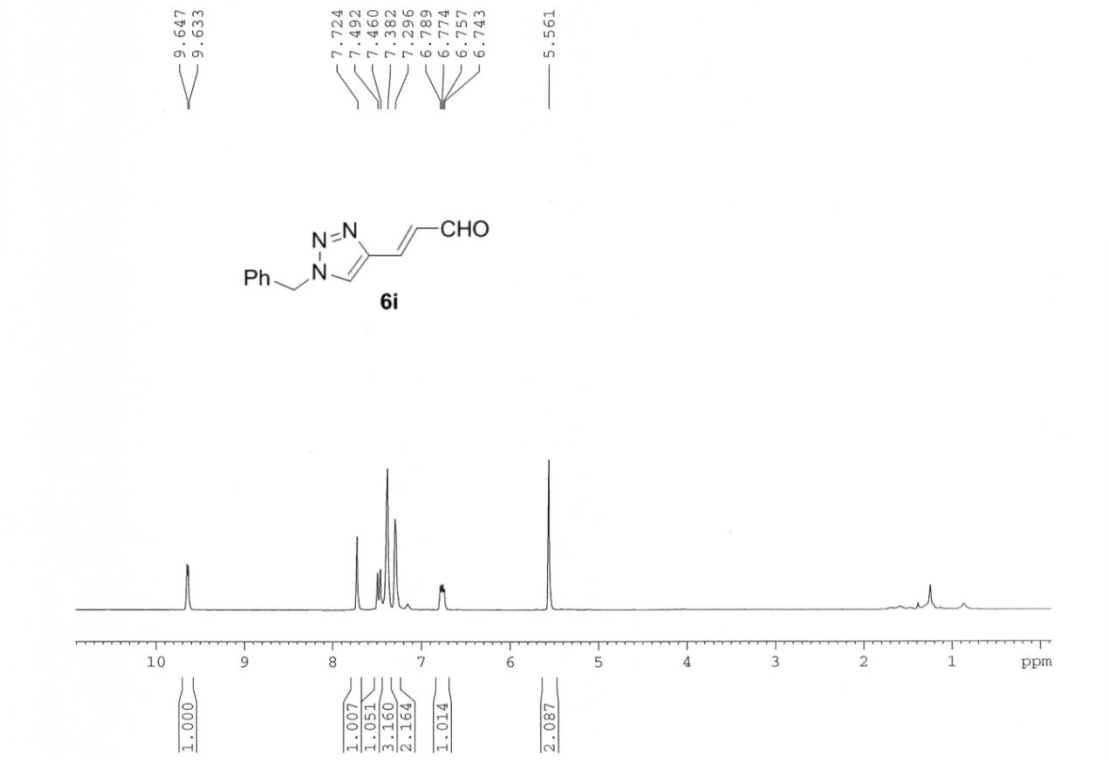
6g

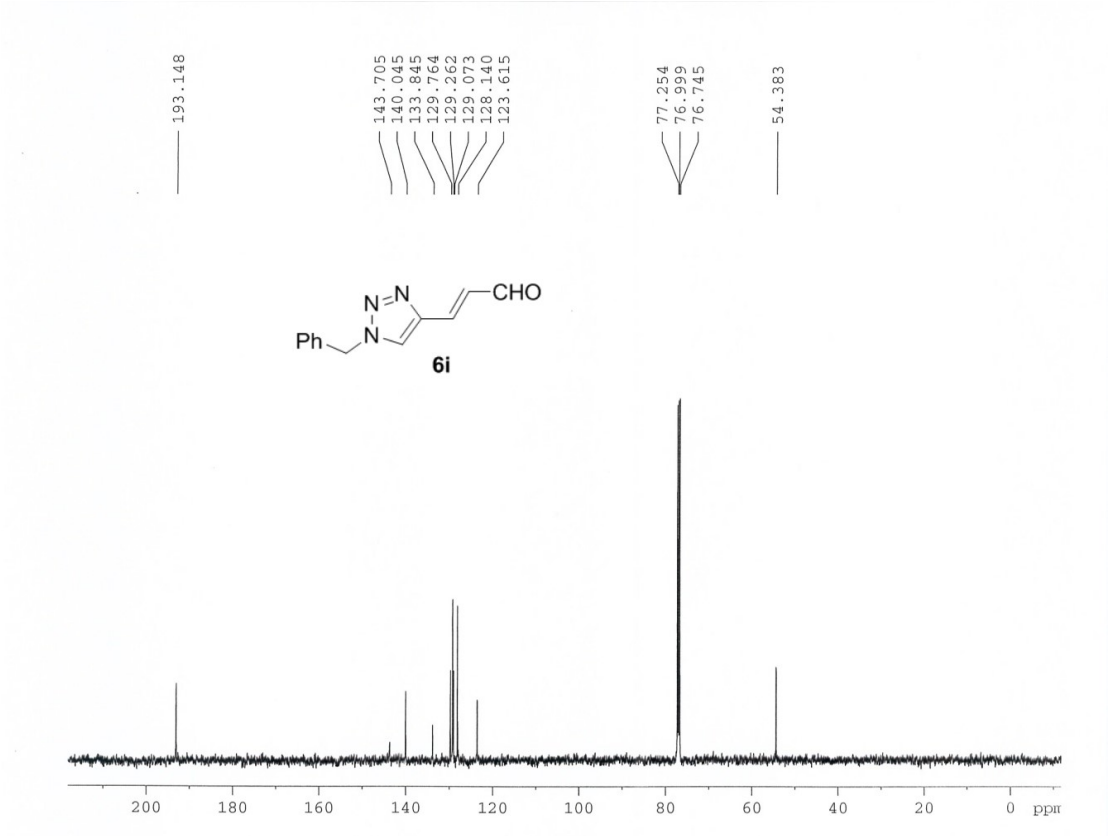

192.735
148.951
148.755
139.926
131.705
129.009
124.282
77.259
77.005
76.751
CHO
O2N
200
180
160
140
120
100
80
60
40
20
ppm

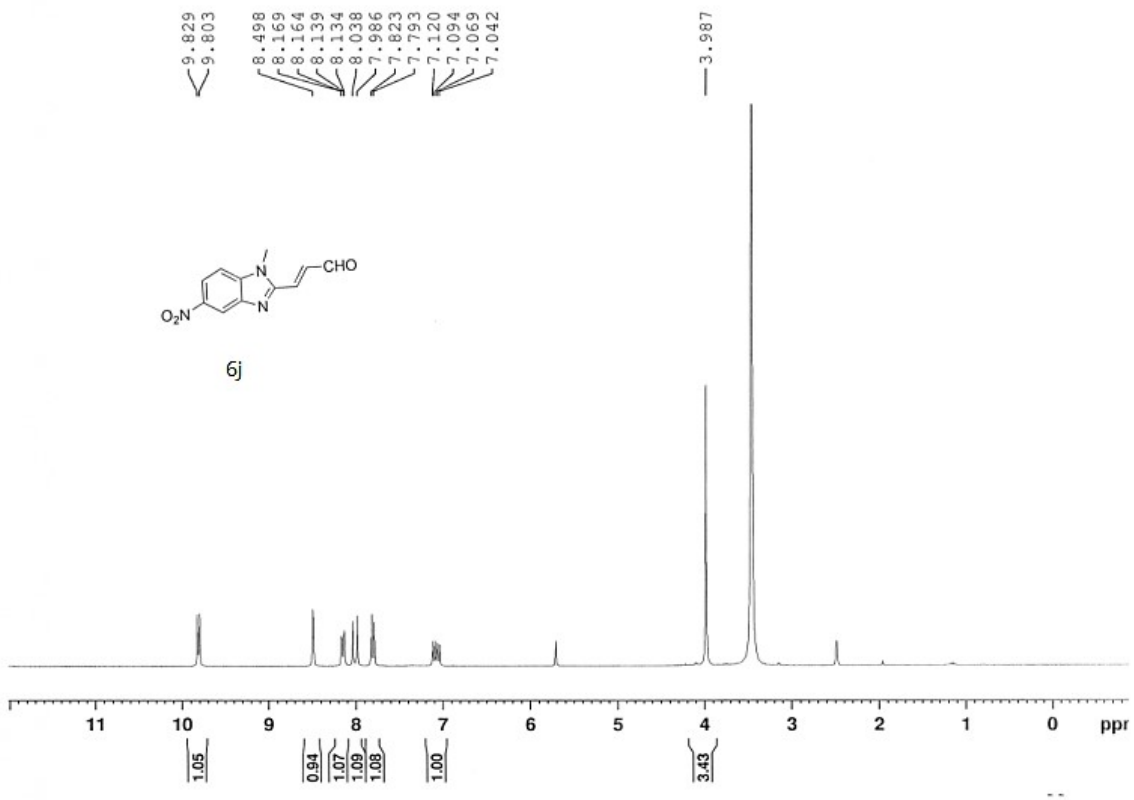
6g


9.758
9.743
7.701
7.683
7.665
7.519
7.487
6.795
6.780
6.763
6.748
CHO
F3C
6h
10
9
8
7
6
5
4
3
2
1
ppm
1.000
4.539
1.034
0.967

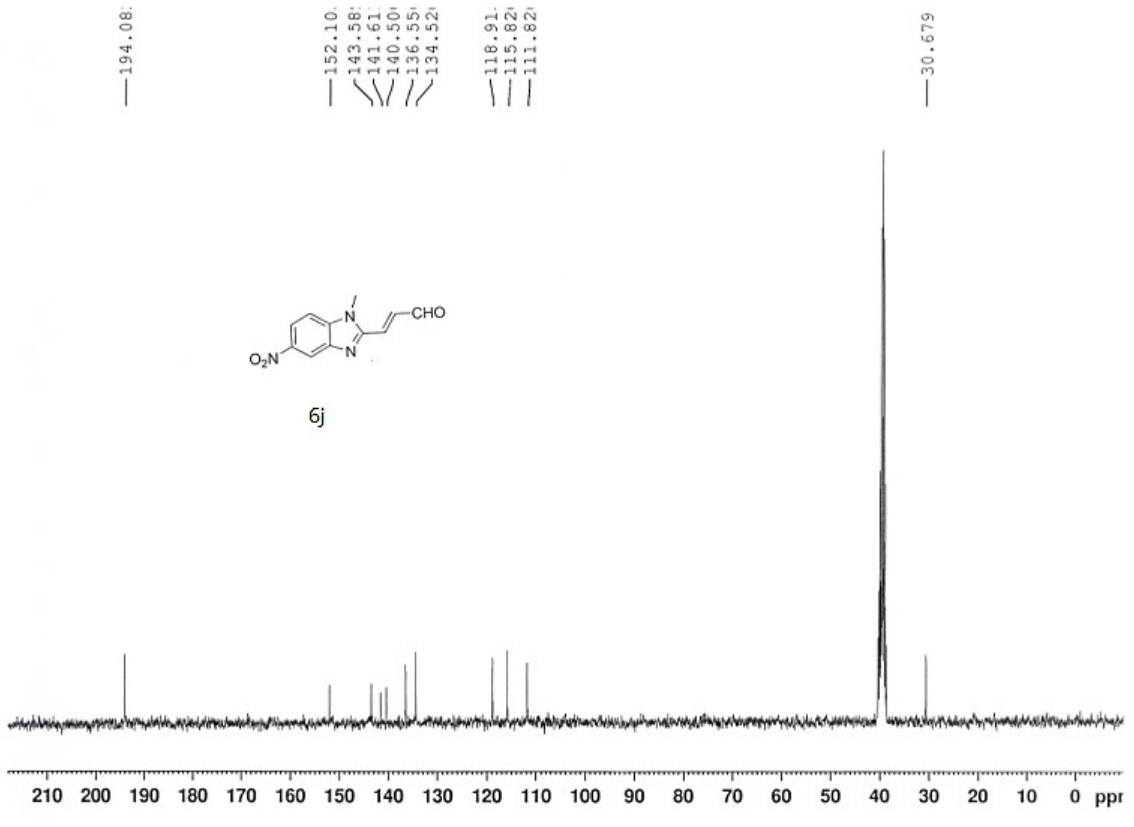

193.075
150.173
137.290
130.501
128.525
126.049
77.234
76.980
76.726
CHO
F3C
6h
200
180
160
140
120
100
80
60
40
20
0
ppm


9.647
9.633
7.724
7.492
7.460
7.382
7.296
6.789
6.774
6.757
6.743
5.561
CHO
N
N
Ph
6i
10
9
8
7
6
5
4
3
2
1
ppm
1.000
1.007
1.051
3.160
2.164
1.014
2.087

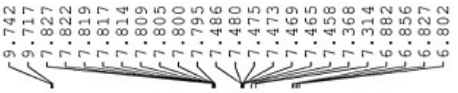

193.148
143.705
140.045
133.845
129.764
129.262
129.073
128.140
123.615
77.254
76.999
76.745
54.383
CHO
N
N
Ph
6i
200
180
160
140
120
100
80
60
40
20
0
ppm

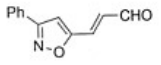

9.829
9.803
8.498
8.169
8.164
8.139
8.134
8.038
7.986
7.823
7.793
7.120
7.094
7.069
7.042
3.987
CHO
O2N
N
N
11
10
9
8
7
6
5
4
3
2
1
0
ppm
1.05
0.94
1.07
1.09
1.08
1.00
3.43

6j

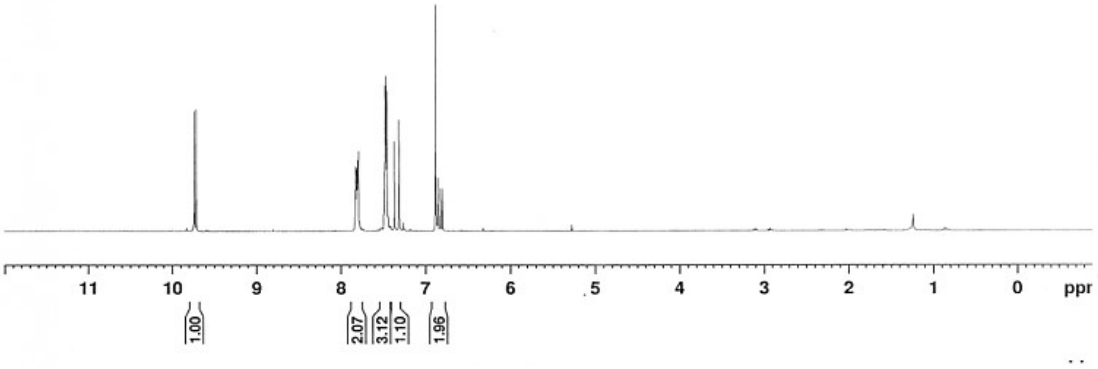

194.08
152.10
143.58
141.61
140.50
136.55
134.52
118.91
115.82
111.82
30.679
CHO
O2N
N
N
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

6j

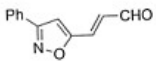

9.742
9.717
7.827
7.822
7.819
7.817
7.814
7.809
7.805
7.800
7.795
7.486
7.480
7.475
7.473
7.469
7.465
7.458
7.368
7.314
6.882
6.856
6.827
6.802


Ph
N
O
CHO

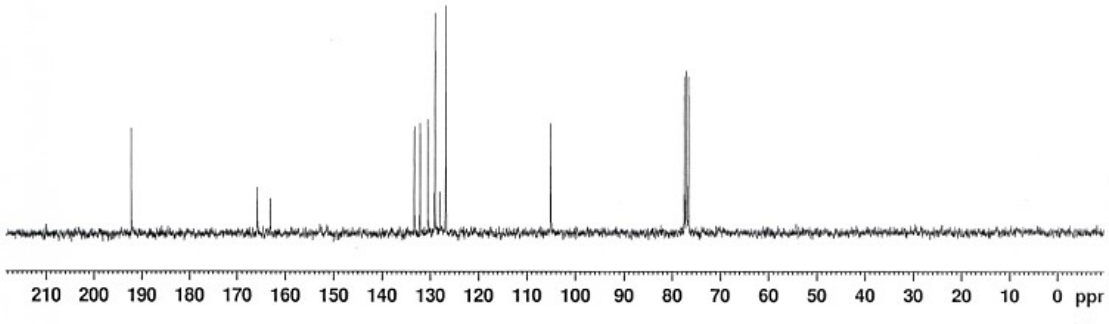
6k

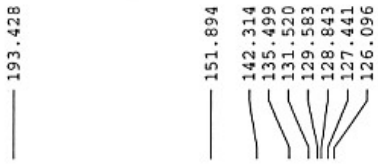

11
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
2.07
3.12
1.10
1.96

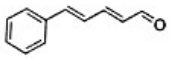

192.17
165.77
163.17
133.32
132.16
130.53
129.10
128.11
126.80
105.08


Ph
N
O
CHO

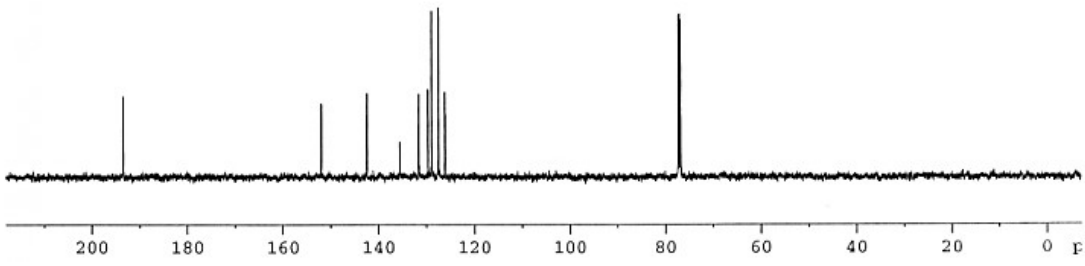
6k


210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

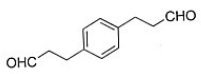

193.428
151.894
142.314
135.499
131.520
129.583
128.843
127.441
126.096


O

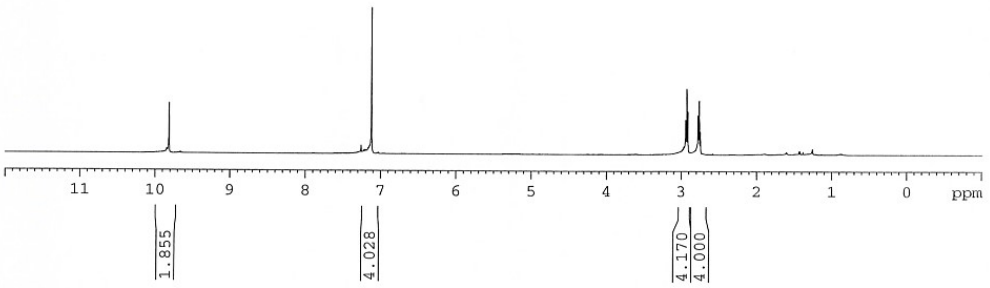
6l

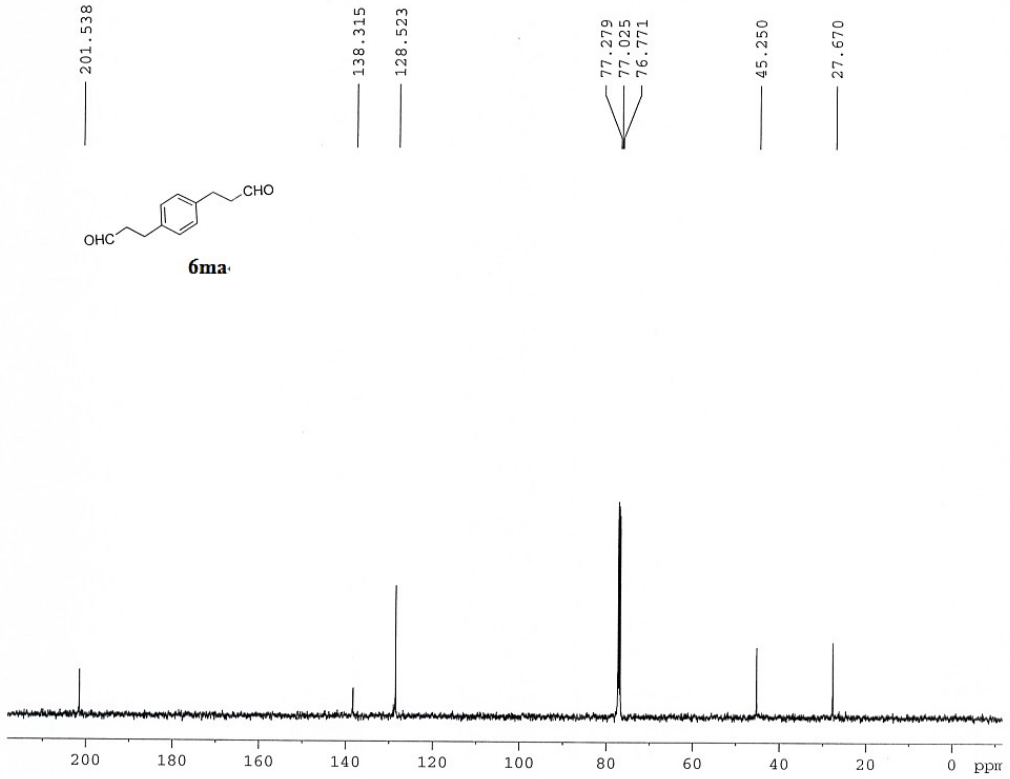

200
180
160
140
120
100
80
60
40
20
0


9.813
7.121
2.943
2.928
2.913
2.777
2.762
2.747

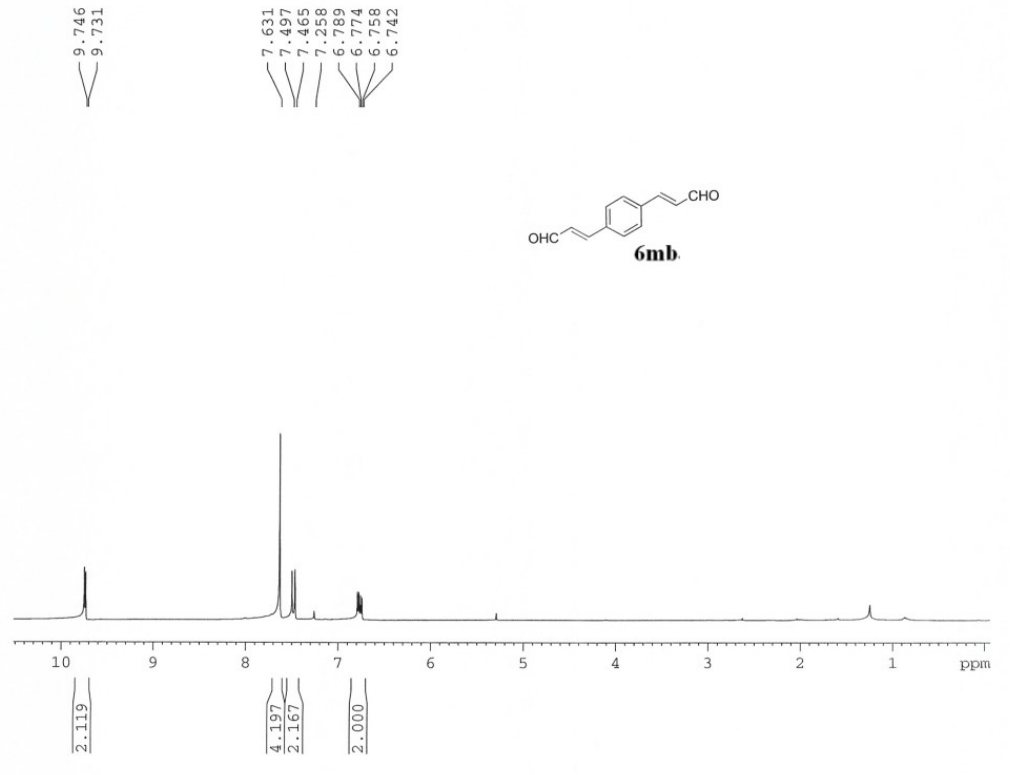

CHO
OHC

6ma

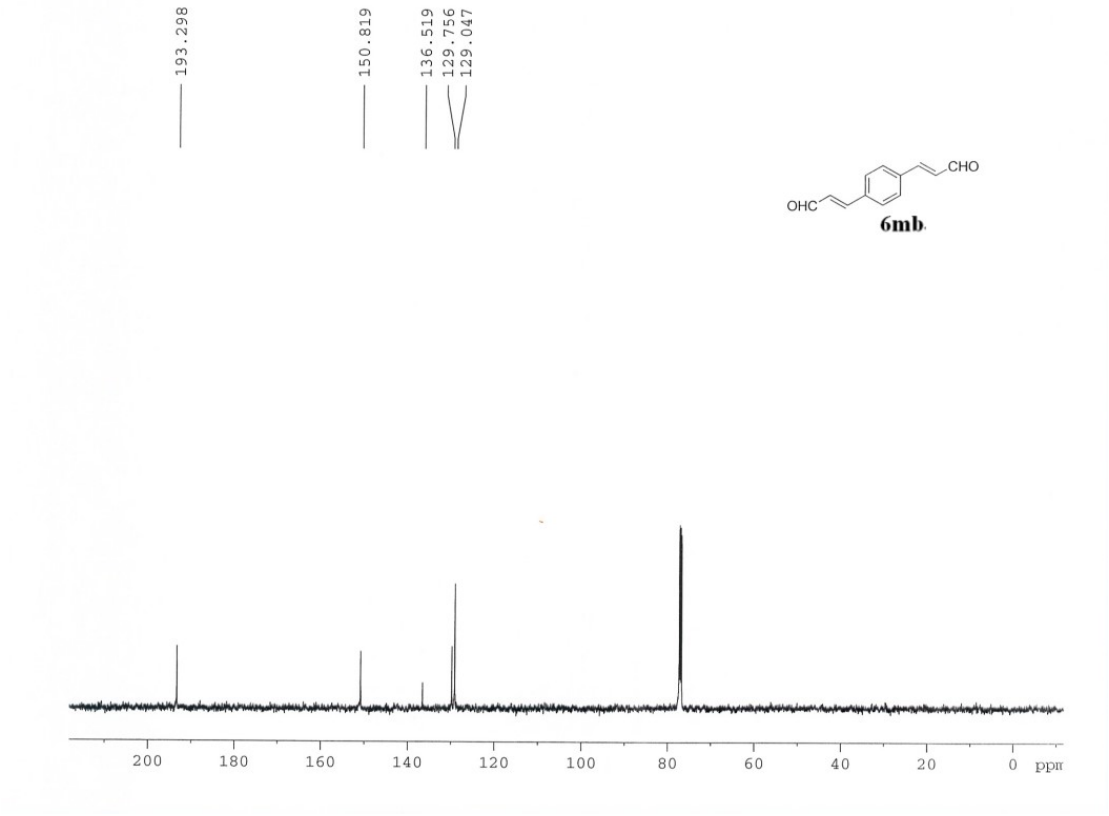

11
10
9
8
7
6
5
4
3
2
1
0
ppm
1.855
4.028
4.170
4.000

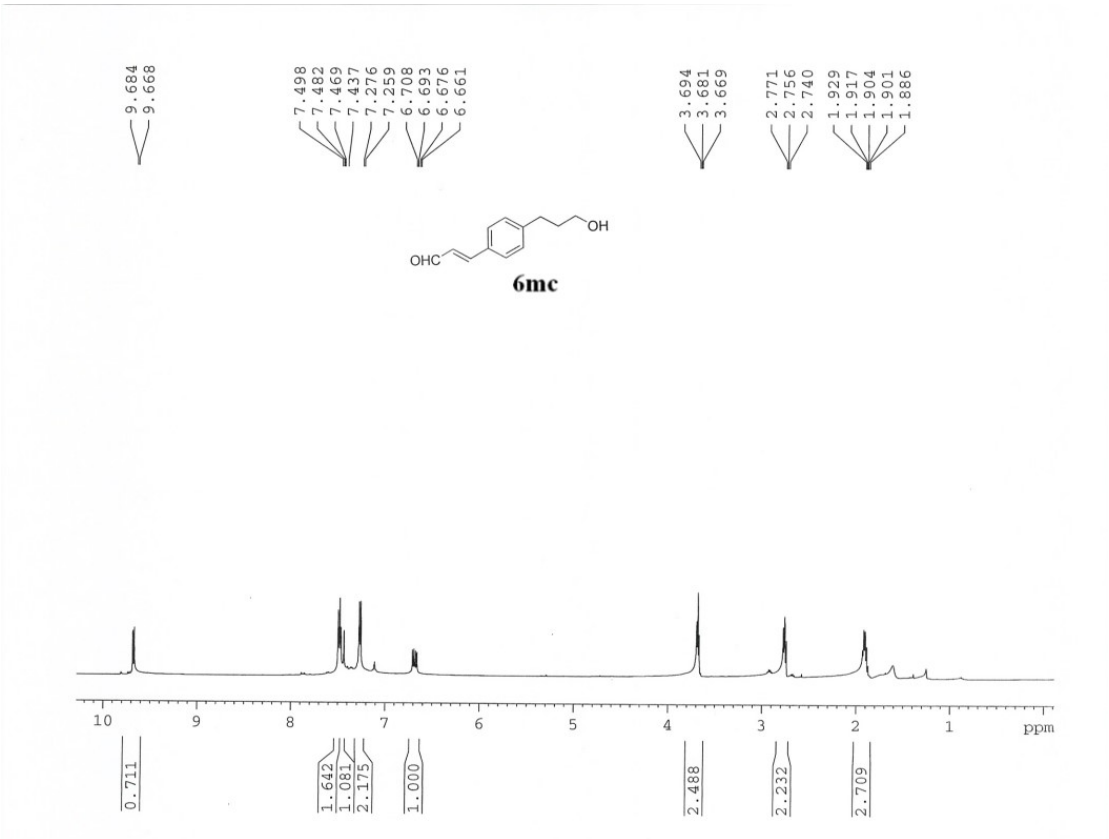

201.538
138.315
128.523
77.279
77.025
76.771
45.250
27.670
CHO
OHC
200
180
160
140
120
100
80
60
40
20
0
ppm

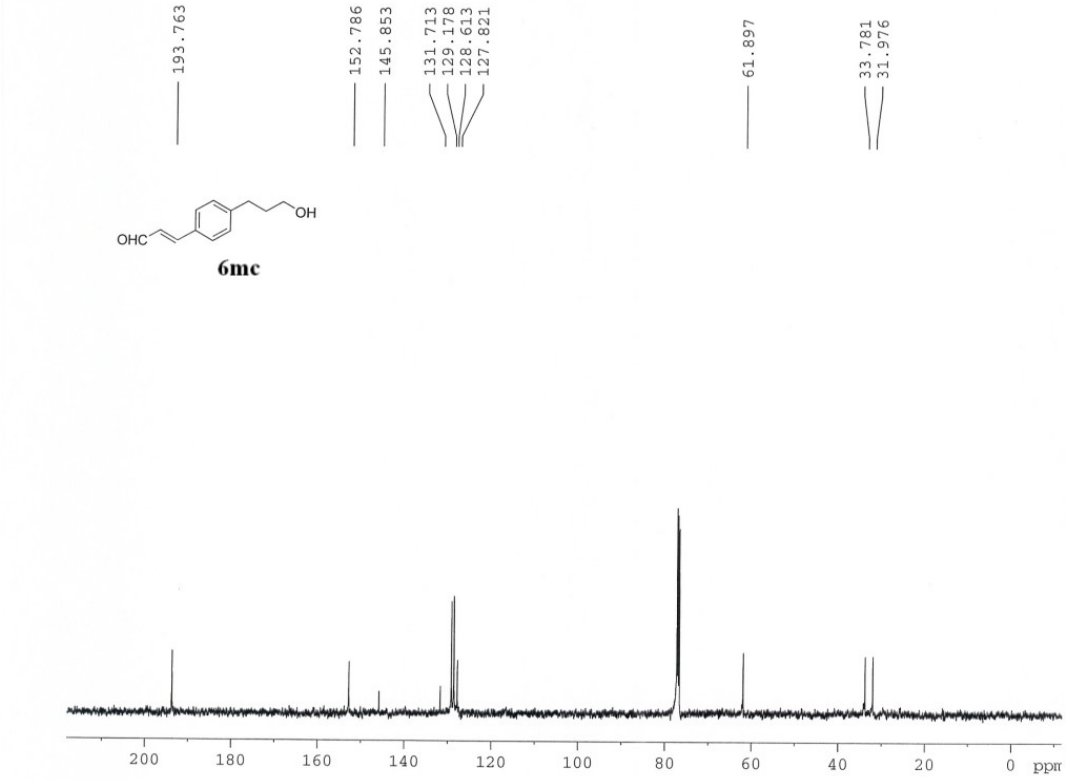
6ma


9.746
9.731
7.631
7.497
7.465
7.258
6.789
6.774
6.758
6.742
CHO
OHC
10
9
8
7
6
5
4
3
2
1
ppm
2.119
4.197
2.167
2.000

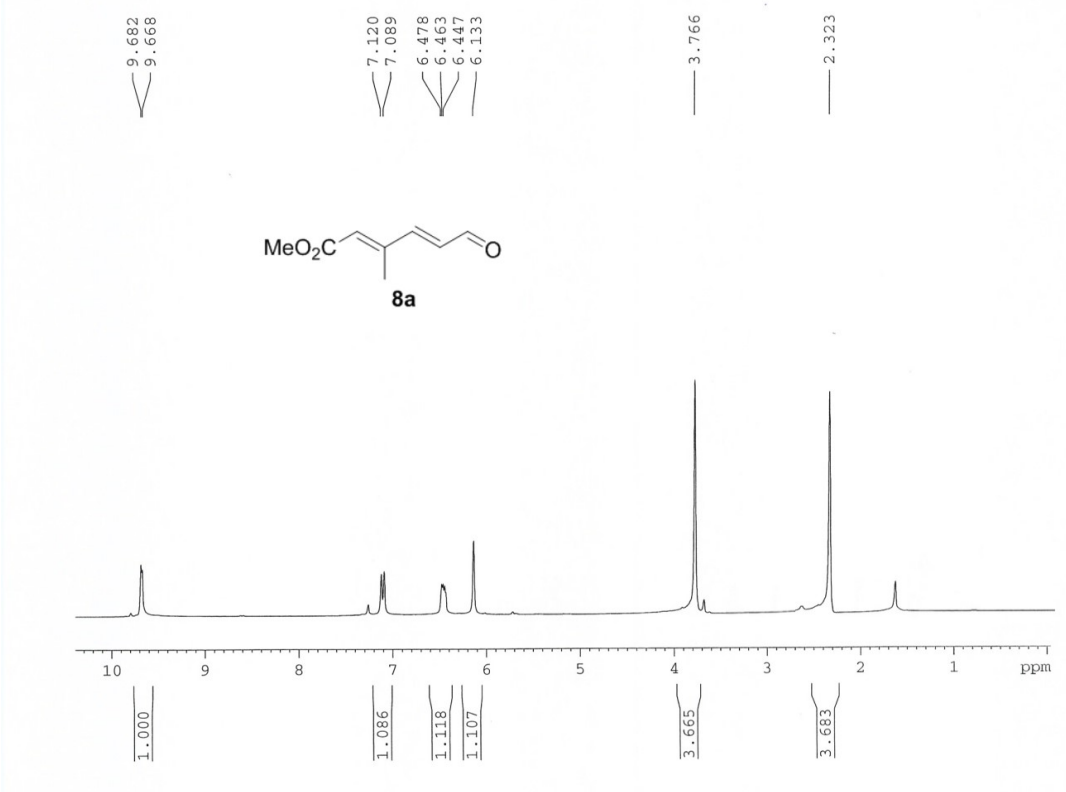
6mb


193.298
150.819
136.519
129.756
129.047
CHO
OHC
6mb
200
180
160
140
120
100
80
60
40
20
0
ppm

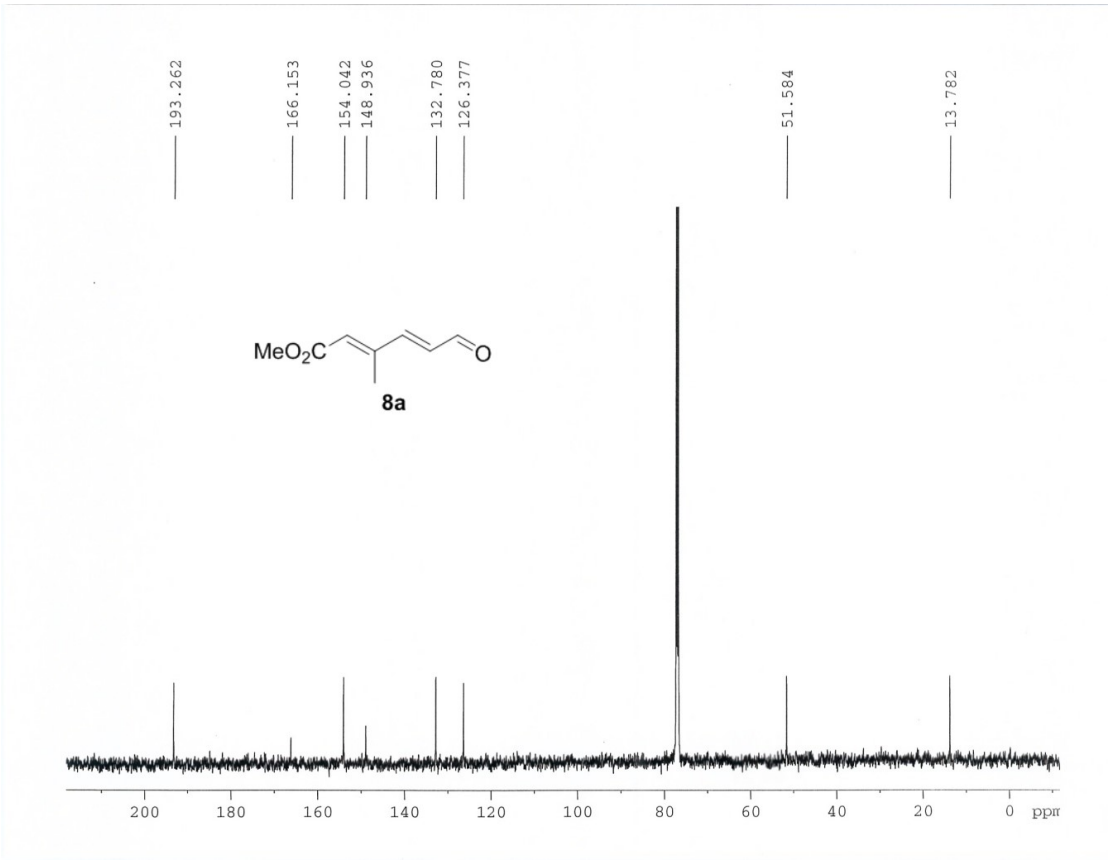

9.684
9.668
7.498
7.482
7.469
7.437
7.276
7.259
6.708
6.693
6.676
6.661
3.694
3.681
3.669
2.771
2.756
2.740
1.929
1.917
1.904
1.901
1.886
OH
OHC
6mc
10
9
8
7
6
5
4
3
2
1
ppm
0.711
1.642
1.081
2.175
1.000
2.488
2.232
2.709


193.763
152.786
145.853
131.713
129.178
128.613
127.821
61.897
33.781
31.976
OHC
OH
200
180
160
140
120
100
80
60
40
20
0 ppm

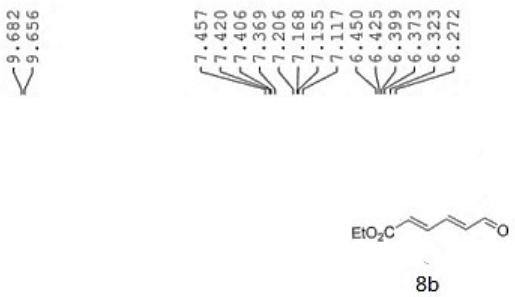
6mc


9.682
9.668
7.120
7.089
6.478
6.463
6.447
6.133
3.766
2.323
MeO2C
O
10
9
8
7
6
5
4
3
2
1
ppm
1.000
1.086
1.118
1.107
3.665
3.683

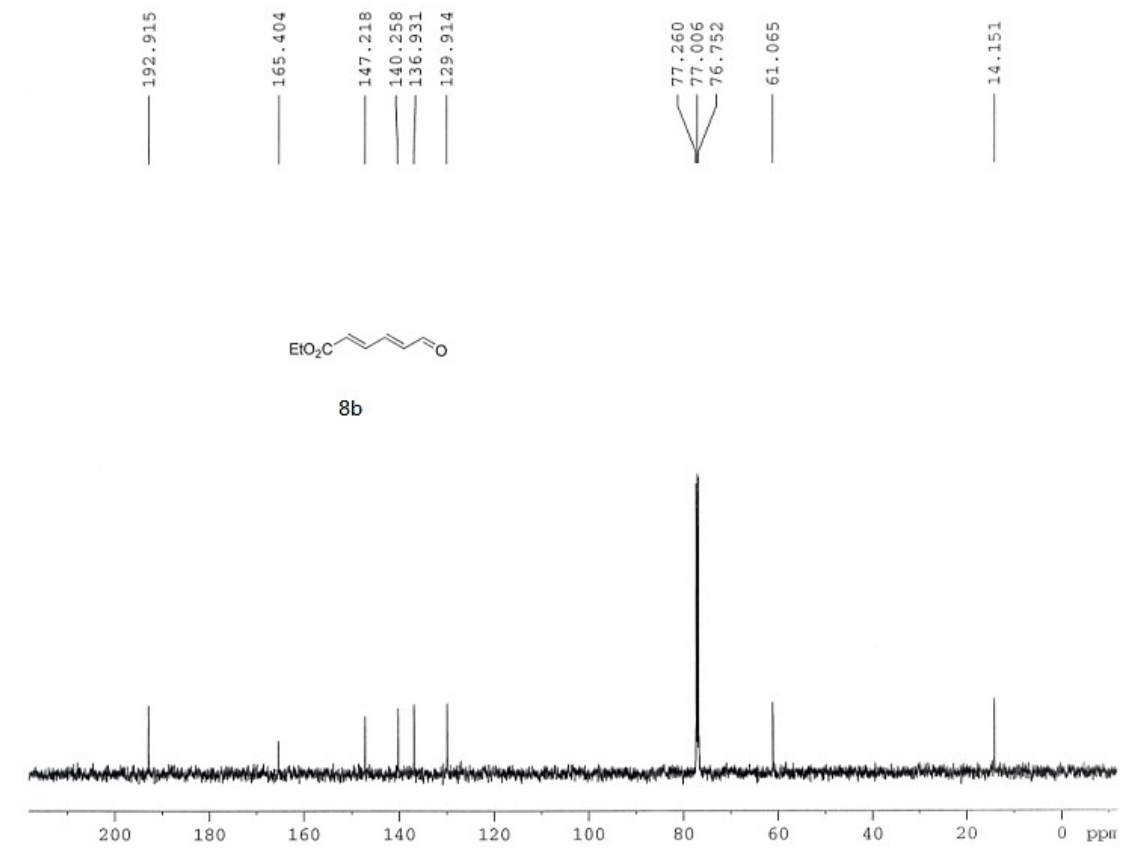
8a


193.262
166.153
154.042
148.936
132.780
126.377
51.584
13.782
MeO2C
O
200
180
160
140
120
100
80
60
40
20
0 ppm

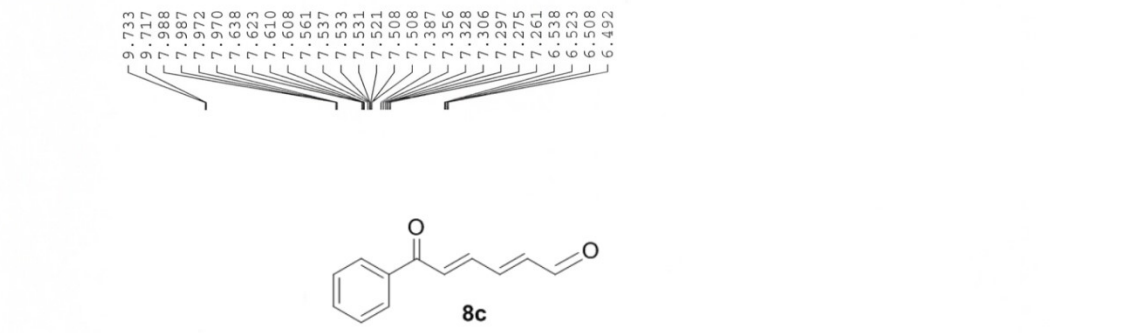
8a


9.682
9.656
7.457
7.420
7.406
7.369
7.206
7.168
7.155
7.117
6.450
6.425
6.399
6.373
6.323
6.272
4.289
4.265
4.241
4.217
1.343
1.319
1.296
EtO2C
O
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
ppm
1.00
1.01
1.06
0.99
0.99
2.08
3.20

8b

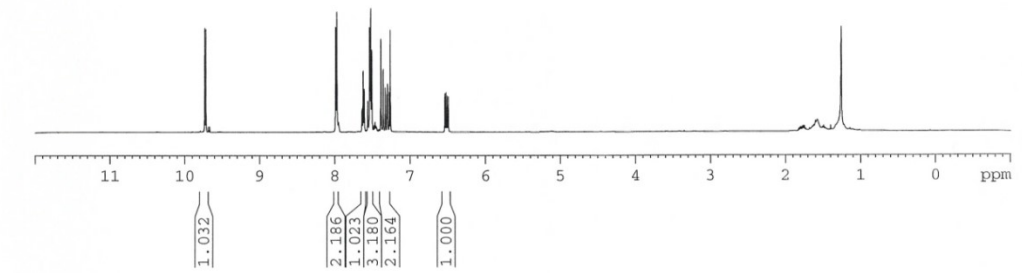

192.915
165.404
147.218
140.258
136.931
129.914
77.260
77.006
76.752
61.065
14.151
EtO2C
O
200
180
160
140
120
100
80
60
40
20
0
ppm

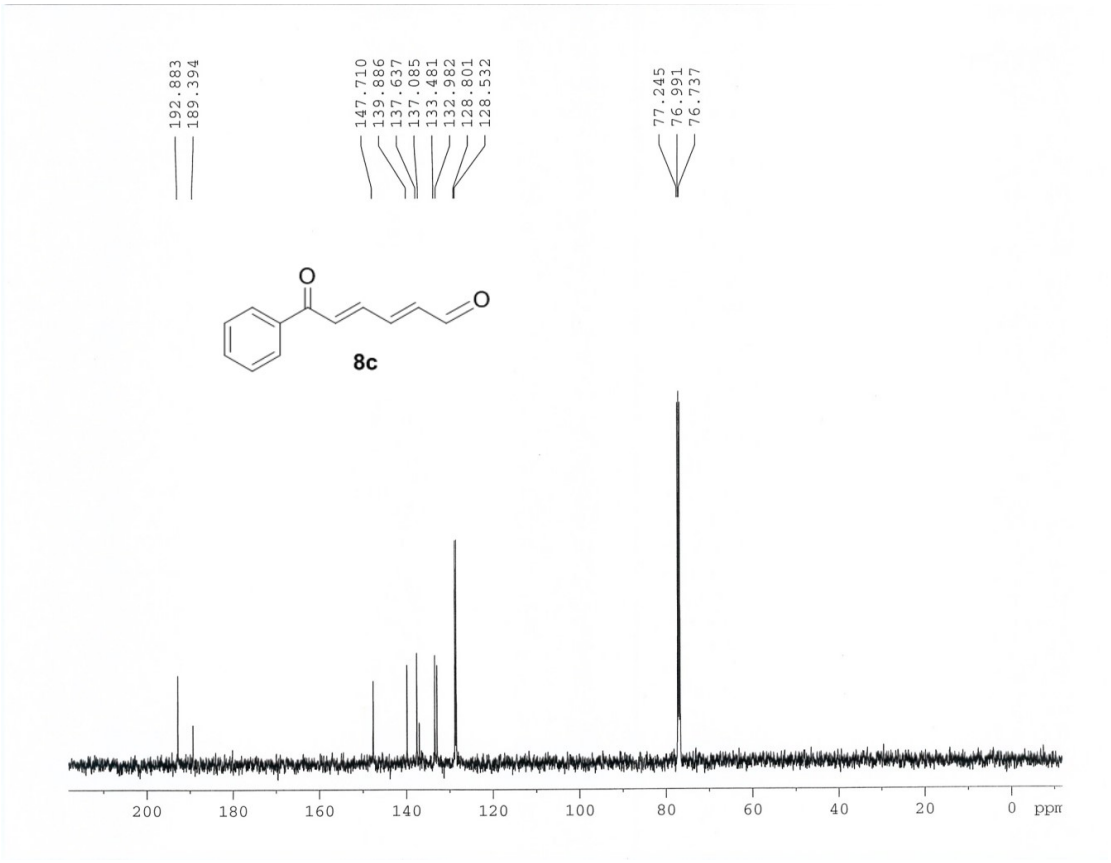
8b

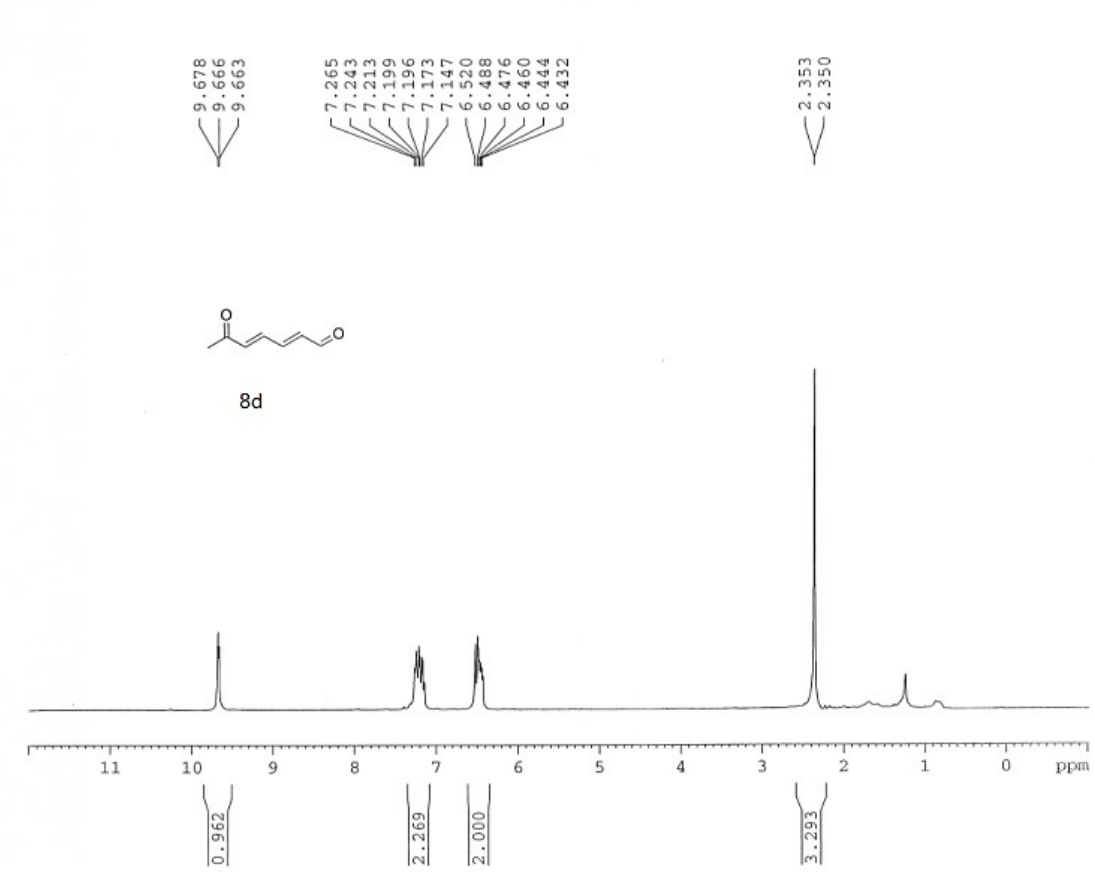

9.733
9.717
7.988
7.987
7.972
7.970
7.638
7.623
7.610
7.608
7.561
7.537
7.533
7.531
7.521
7.508
7.508
7.387
7.356
7.328
7.306
7.297
7.275
7.261
6.538
6.523
6.508
6.492

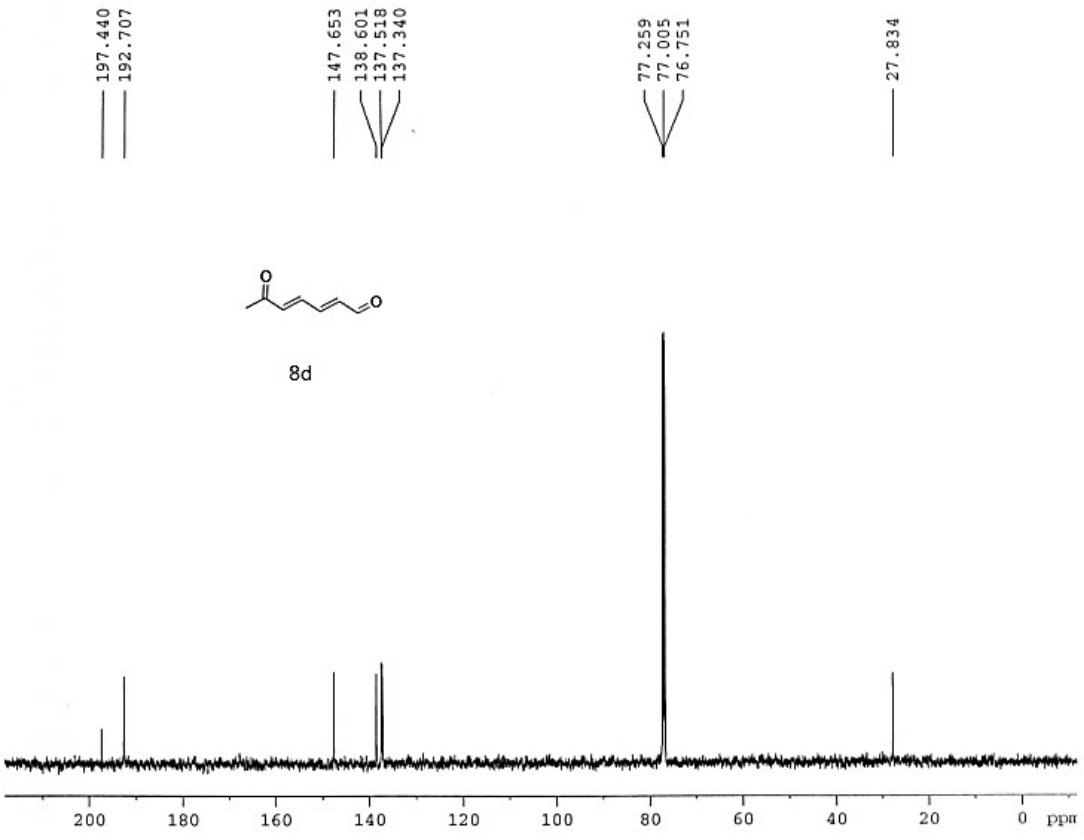
O
O

8c

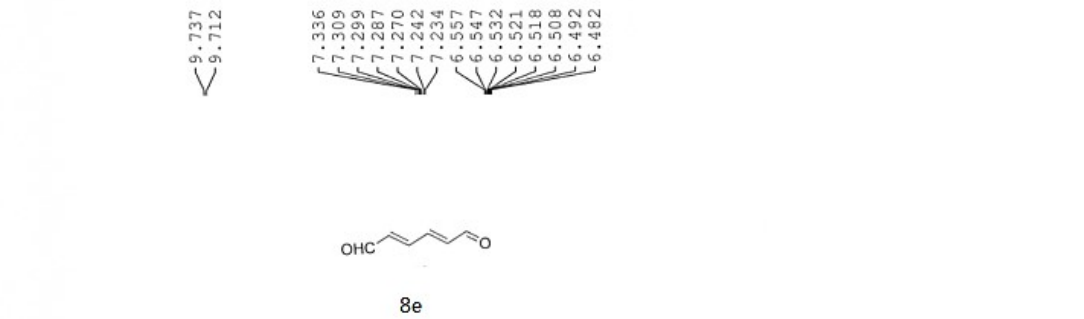

11
10
9
8
7
6
5
4
3
2
1
0
ppm
1.032
2.186
1.023
3.180
2.164
1.000


192.883
189.394
147.710
139.886
137.637
137.085
133.481
132.982
128.801
128.532
77.245
76.991
76.737
O
O
8c
200
180
160
140
120
100
80
60
40
20
0
ppm

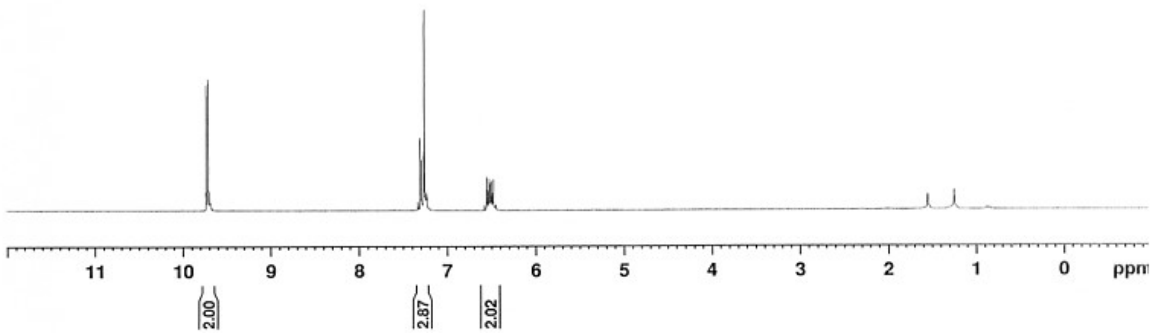

9.678
9.666
9.663
7.265
7.243
7.213
7.199
7.196
7.173
7.147
6.520
6.488
6.476
6.460
6.444
6.432
2.353
2.350
O
O
8d
11
10
9
8
7
6
5
4
3
2
1
0
ppm
0.962
2.269
2.000
3.293

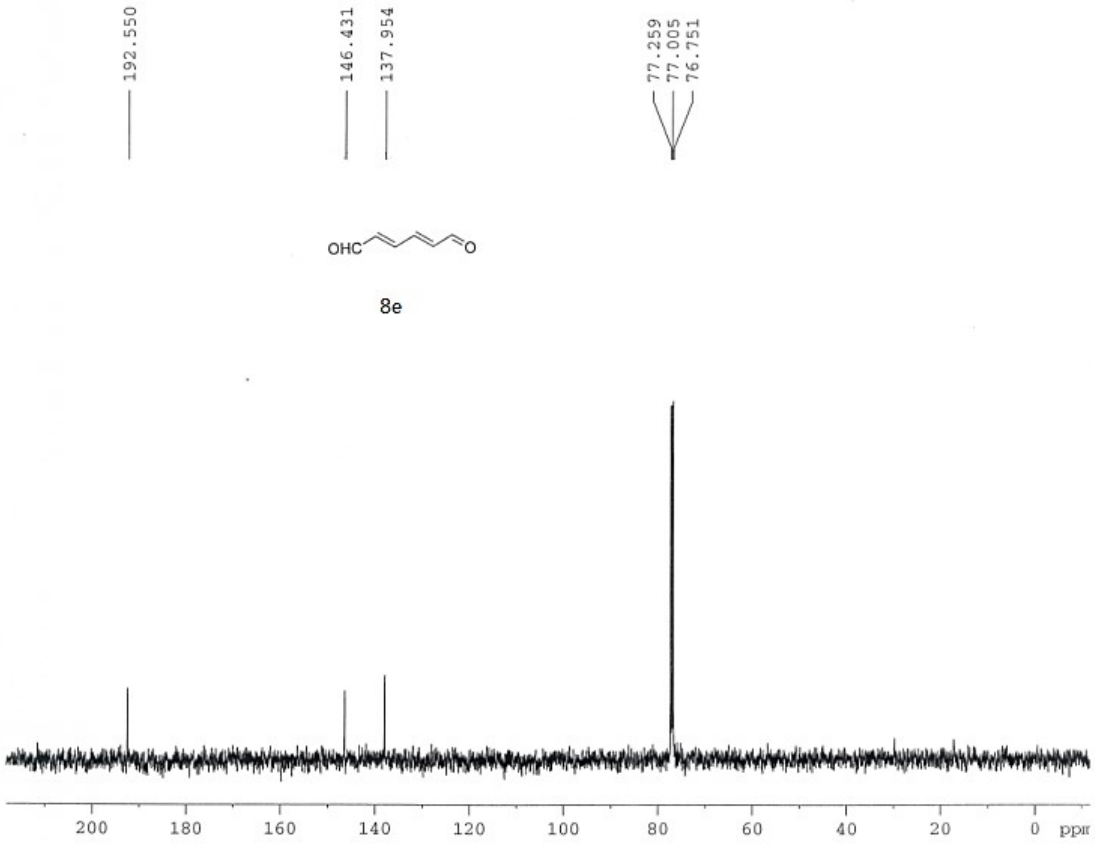

197.440
192.707
147.653
138.601
137.518
137.340
77.259
77.005
76.751
27.834
O
O
200
180
160
140
120
100
80
60
40
20
0
ppm

8d

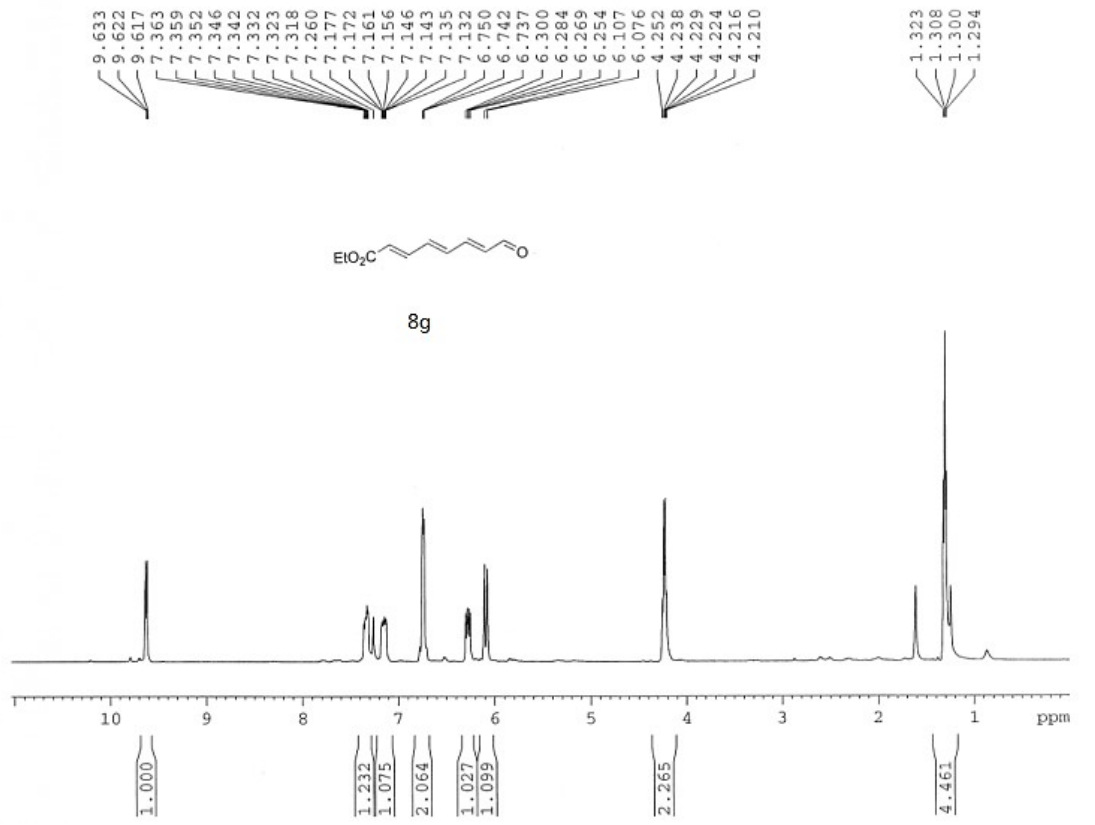

9.737
9.712
7.336
7.309
7.299
7.287
7.270
7.242
7.234
6.557
6.547
6.532
6.521
6.518
6.508
6.492
6.482
OHC
O

8e

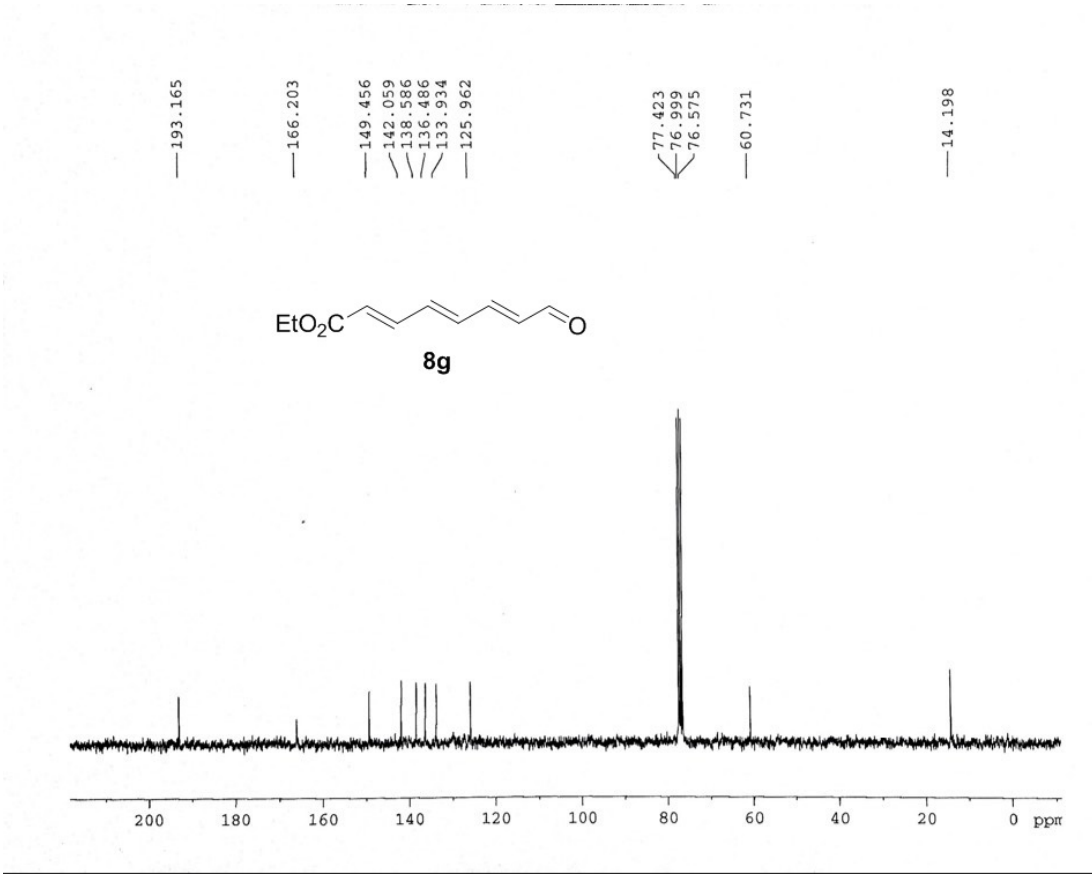

11
10
9
8
7
6
5
4
3
2
1
0
ppm
2.00
2.87
2.02

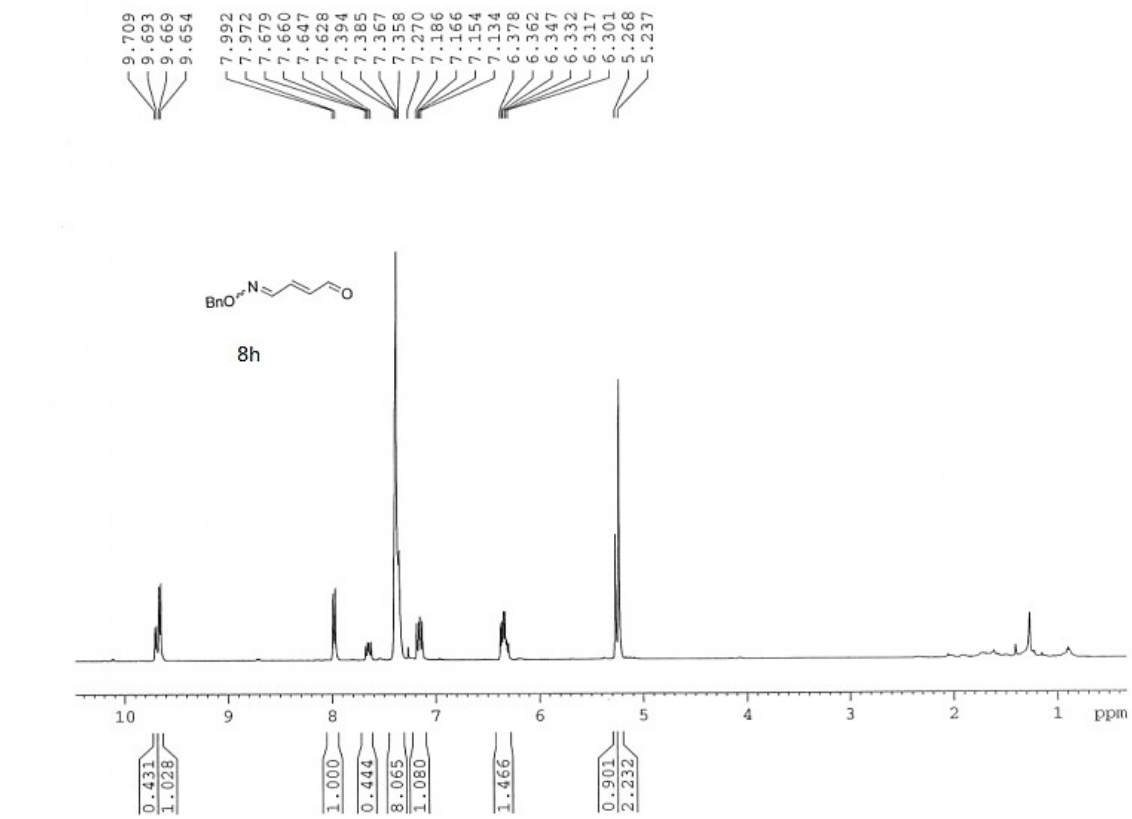

192.550
146.431
137.954
77.259
77.005
76.751
OHC
O
200
180
160
140
120
100
80
60
40
20
0
ppm

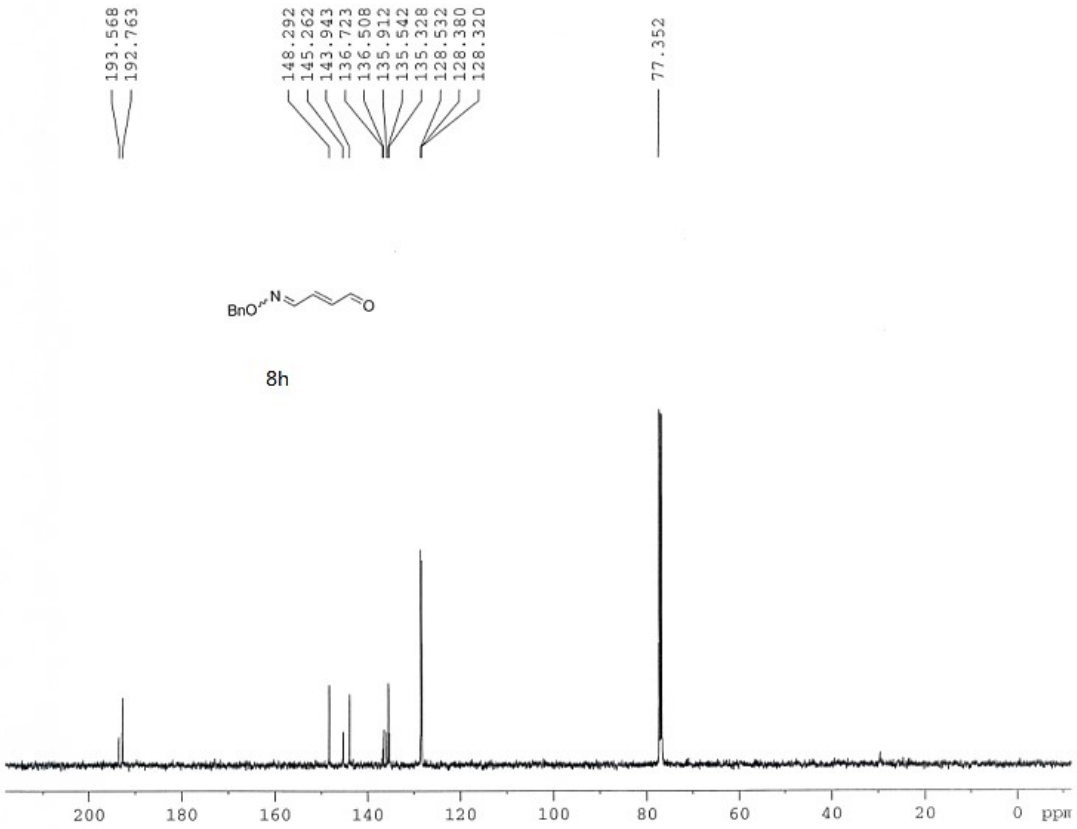
8e


9.633
9.622
9.617
7.363
7.359
7.352
7.346
7.342
7.332
7.323
7.318
7.260
7.177
7.172
7.161
7.156
7.146
7.143
7.135
7.132
6.750
6.742
6.737
6.300
6.284
6.269
6.254
6.107
6.076
4.252
4.238
4.229
4.224
4.216
4.210
1.323
1.308
1.300
1.294
EtO2C
O
10
9
8
7
6
5
4
3
2
1
ppm
1.000
1.232
1.075
2.064
1.027
1.099
2.265
4.461

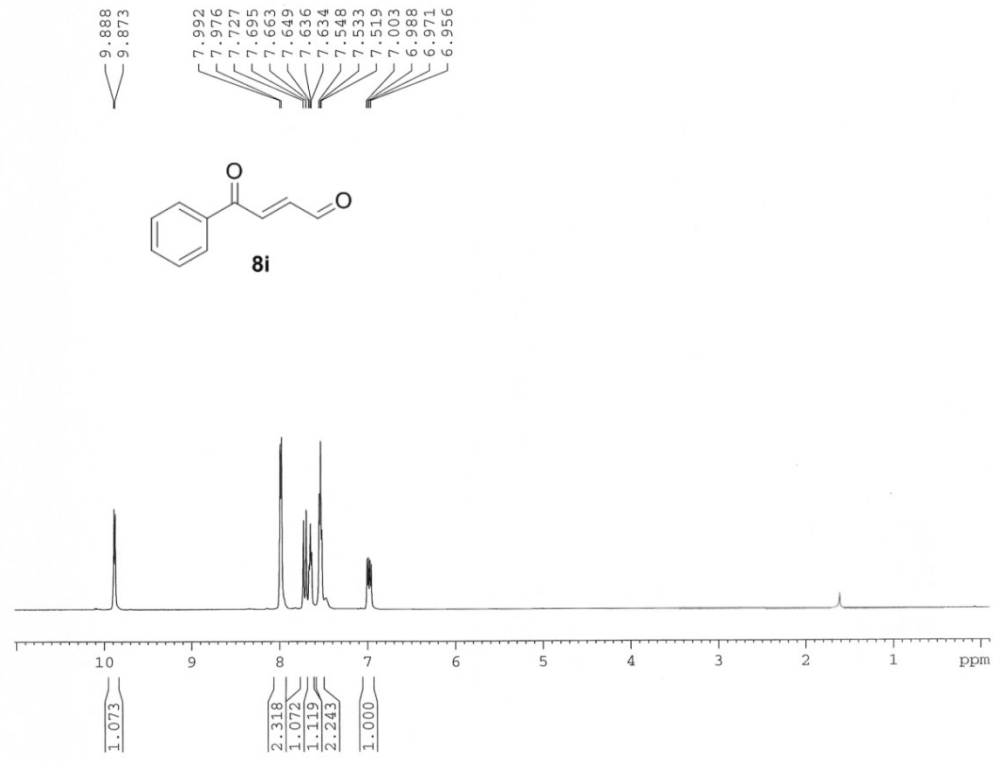
8g


193.165
166.203
149.456
142.059
138.586
136.486
133.934
125.962
77.423
76.999
76.575
60.731
14.198
EtO2C
O
8g
200
180
160
140
120
100
80
60
40
20
0 ppm

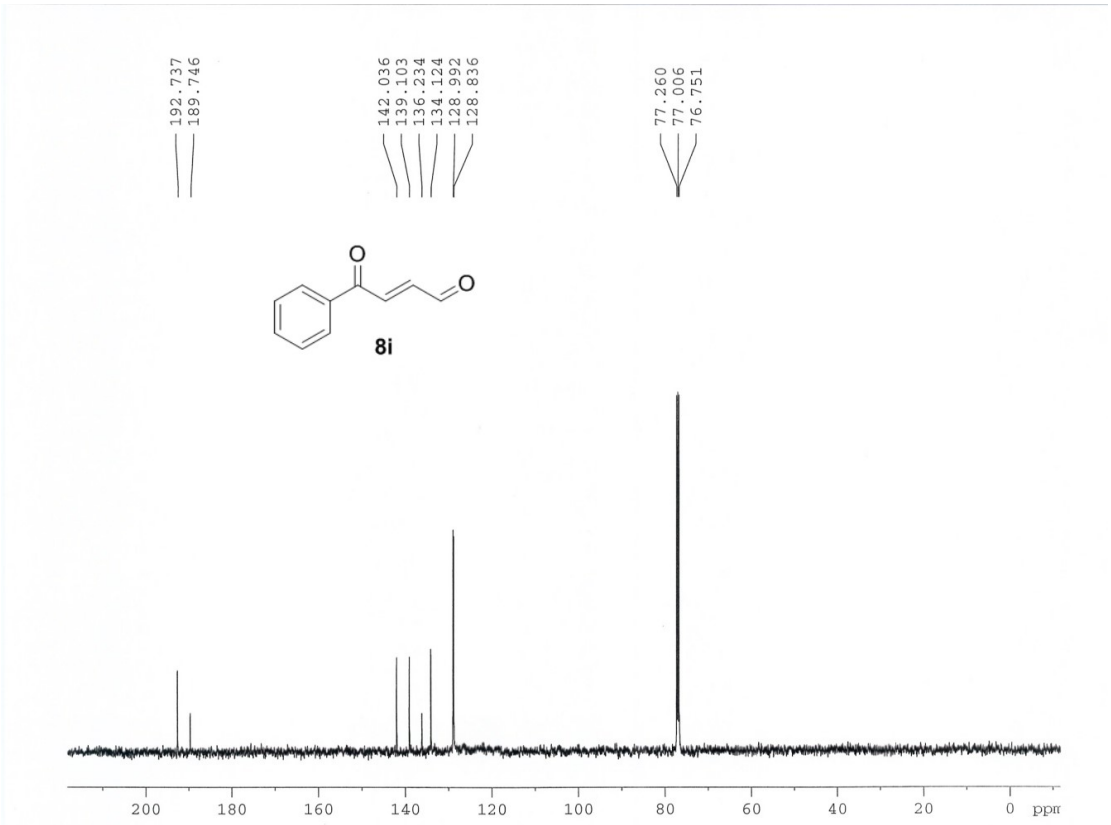

9.709
9.693
9.669
9.654
7.992
7.972
7.679
7.660
7.647
7.628
7.394
7.385
7.367
7.358
7.270
7.186
7.166
7.154
7.134
6.378
6.362
6.347
6.332
6.317
6.301
5.268
5.237
BnO
N
O
8h
10
9
8
7
6
5
4
3
2
1
ppm
0.431
1.028
1.000
0.444
8.065
1.080
1.466
0.901
2.232

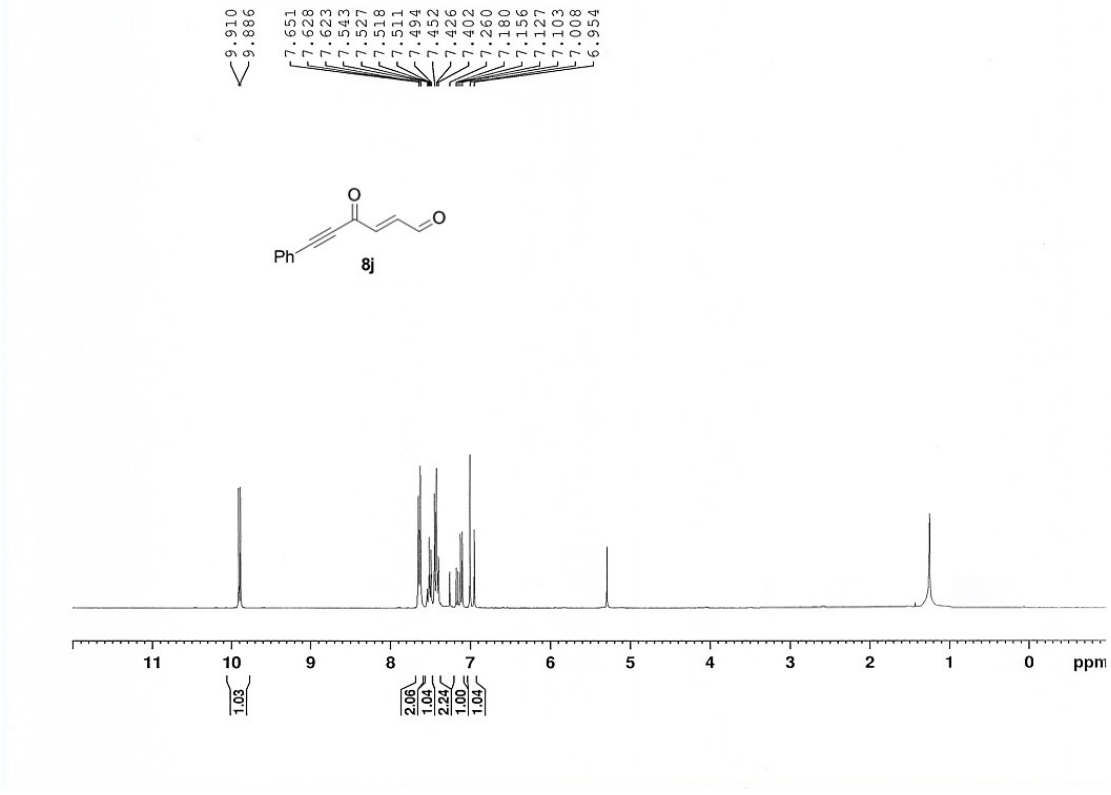

193.568
192.763
148.292
145.262
143.943
136.723
136.508
135.912
135.542
135.328
128.532
128.380
128.320
77.352
BnO
N
O
200
180
160
140
120
100
80
60
40
20
0
ppm

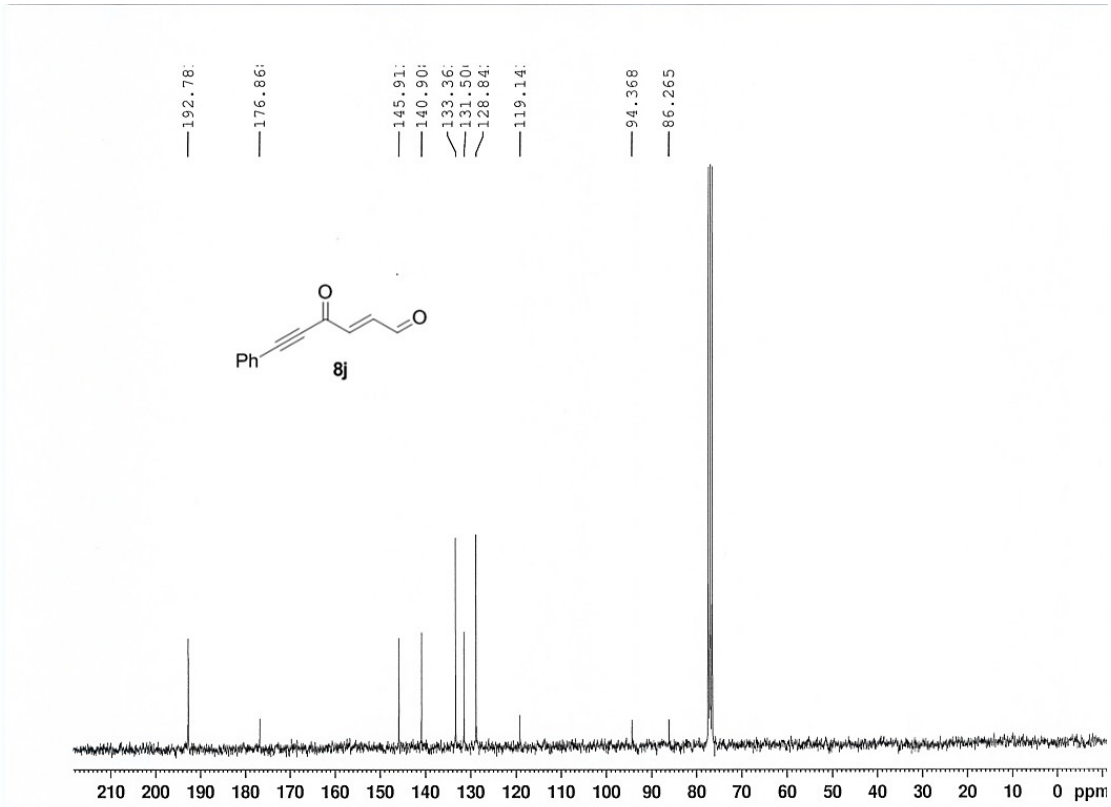
8h

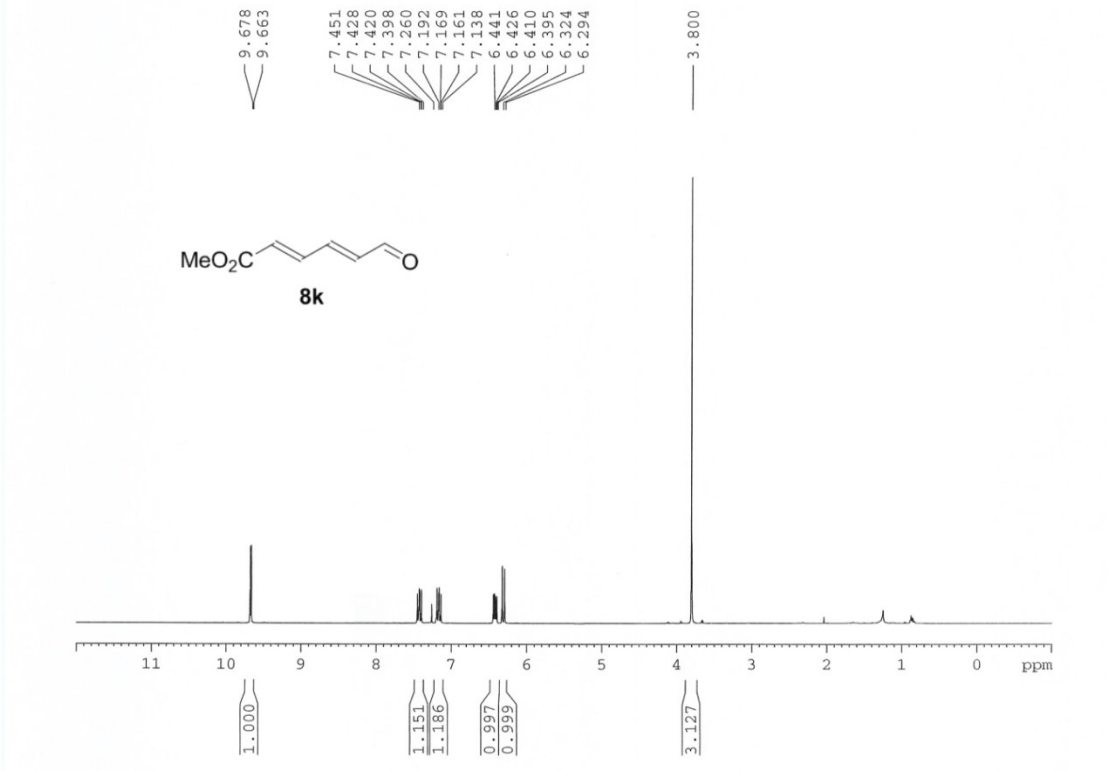

9.888
9.873
7.992
7.976
7.727
7.695
7.663
7.649
7.636
7.634
7.548
7.533
7.519
7.003
6.988
6.971
6.956
O
O
10
9
8
7
6
5
4
3
2
1
ppm
1.073
2.318
1.072
1.119
2.243
1.000

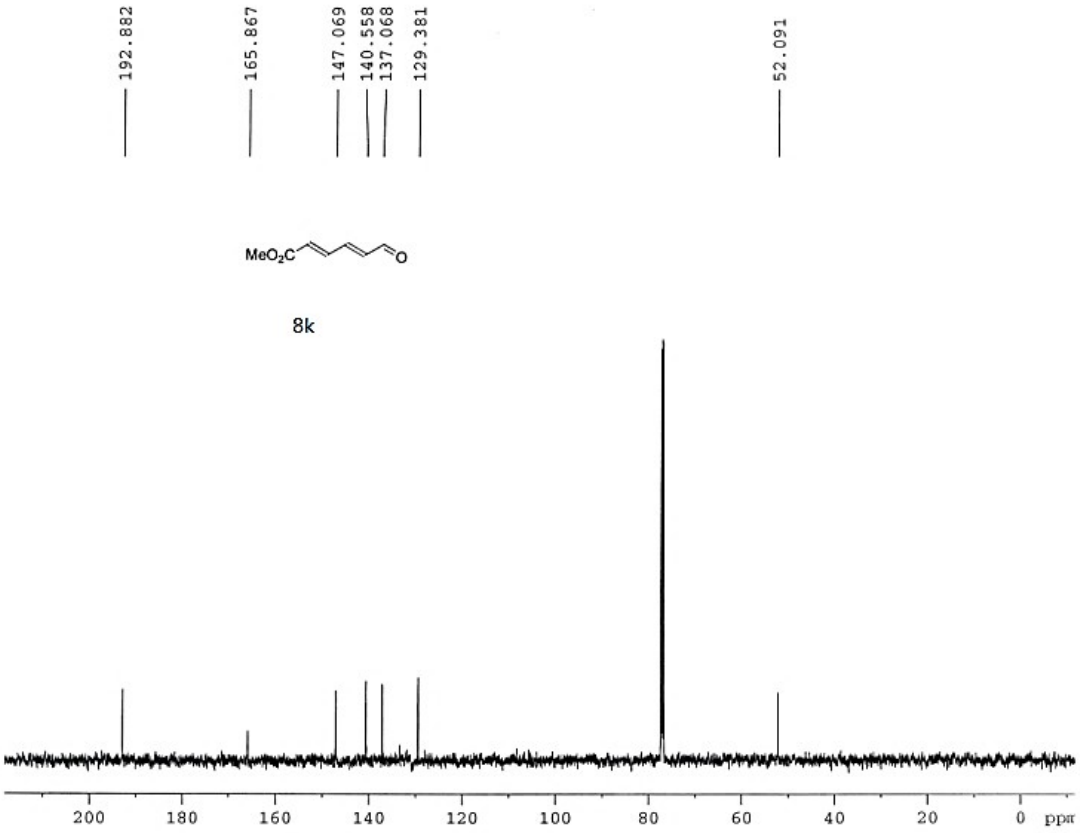
8i


192.737
189.746
142.036
139.103
136.234
134.124
128.992
128.836
77.260
77.006
76.751
O
O
8i
200
180
160
140
120
100
80
60
40
20
0
ppm

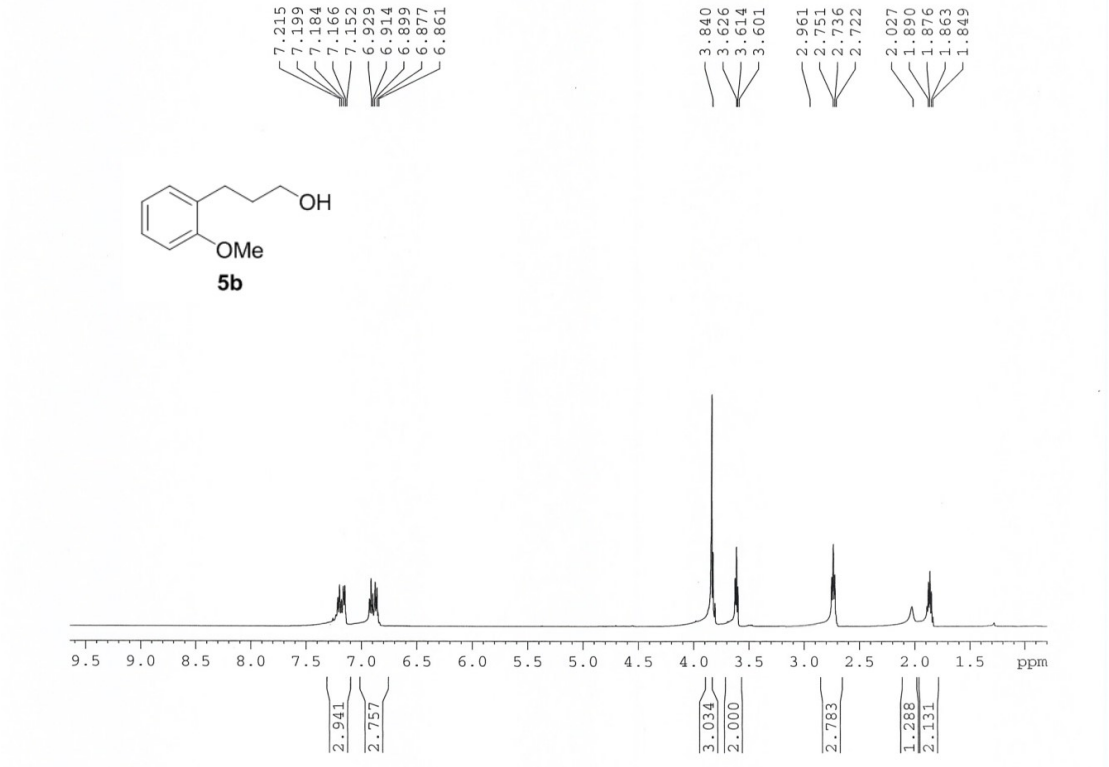

9.910
9.886
7.651
7.628
7.623
7.543
7.527
7.518
7.511
7.494
7.452
7.426
7.402
7.260
7.180
7.156
7.127
7.103
7.008
6.954
O
O
Ph
8j
11
10
9
8
7
6
5
4
3
2
1
0
ppm
1.03
2.06
1.04
2.24
1.00
1.04


192.78
176.86
145.91
140.90
133.36
131.50
128.84
119.14
94.368
86.265
O
O
Ph
8j
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

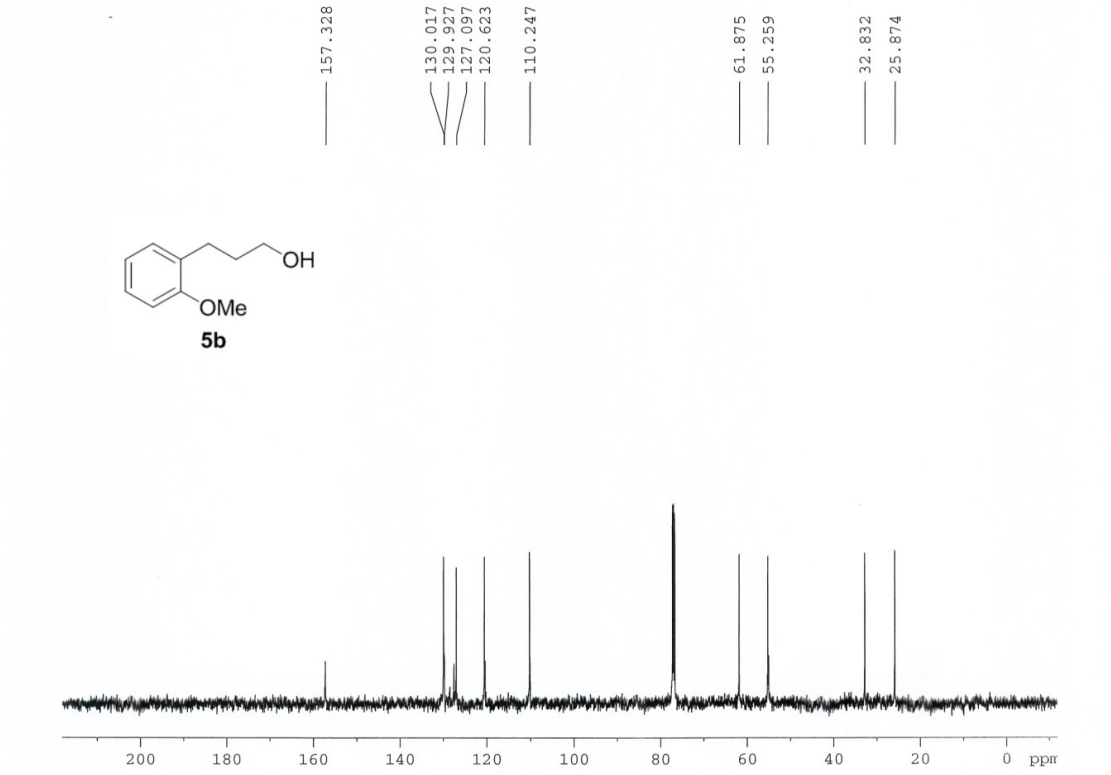

9.678
9.663
7.451
7.428
7.420
7.398
7.260
7.192
7.169
7.161
7.138
6.441
6.426
6.410
6.395
6.324
6.294
3.800
MeO2C
O
8k
11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.000
1.151
1.186
0.997
0.999
3.127

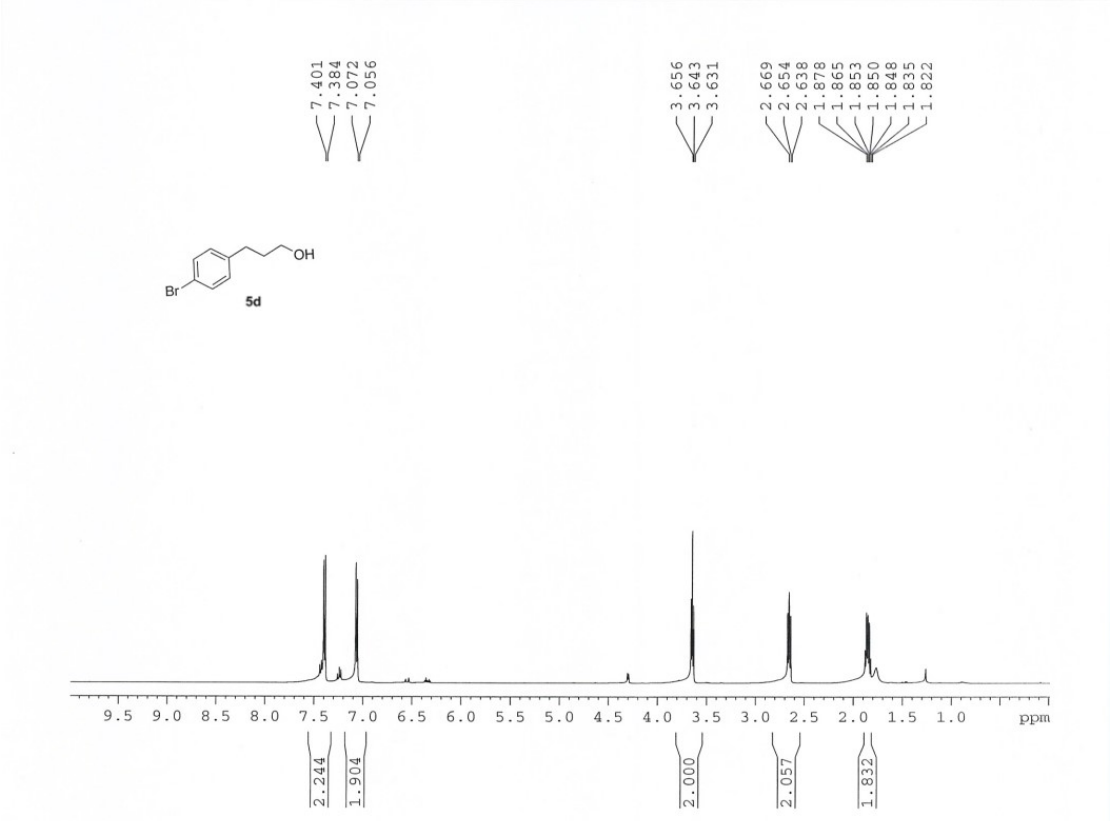

192.882
165.867
147.069
140.558
137.068
129.381
52.091
MeO2C
O
200
180
160
140
120
100
80
60
40
20
0
ppm

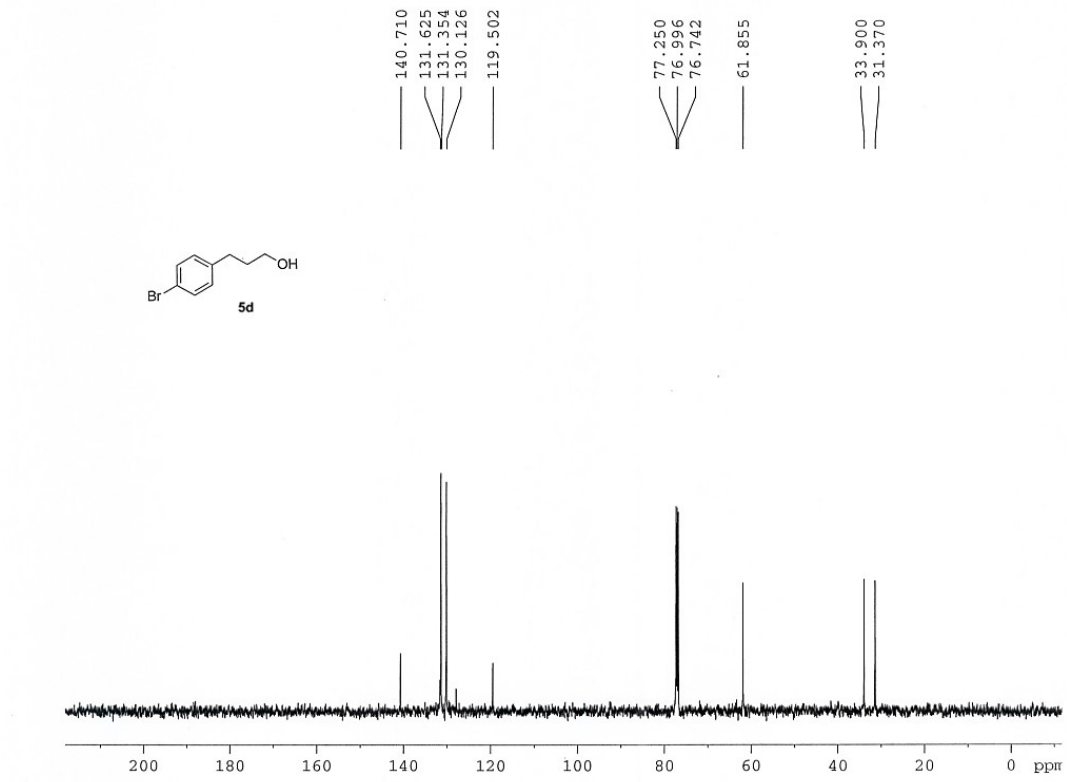
8k

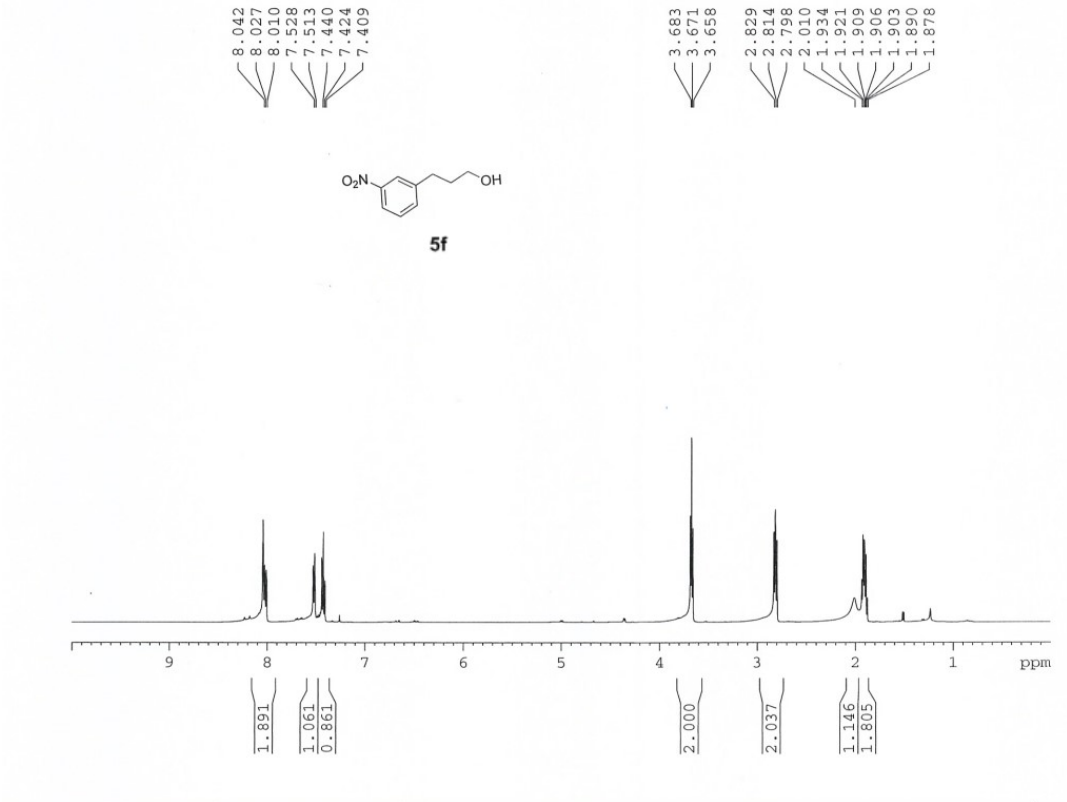

7.215
7.199
7.184
7.166
7.152
6.929
6.914
6.899
6.877
6.861
3.840
3.626
3.614
3.601
2.961
2.751
2.736
2.722
2.027
1.890
1.876
1.863
1.849
OH
OMe
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
ppm
2.941
2.757
3.034
2.000
2.783
1.288
2.131

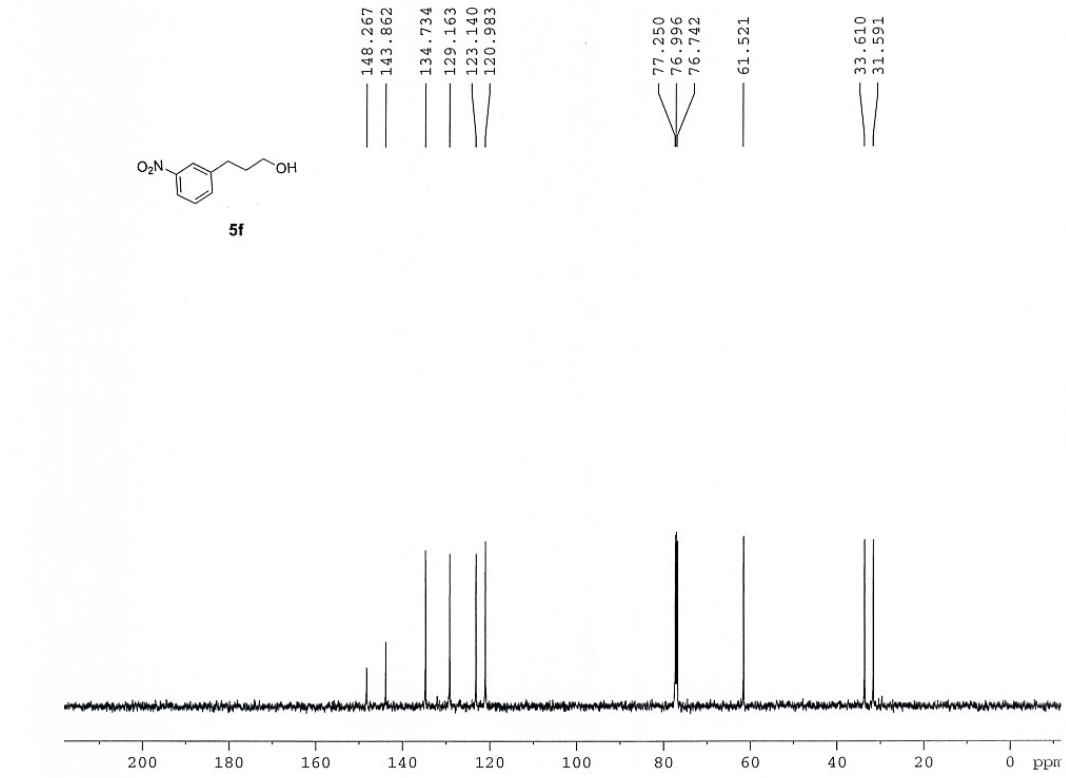
5b

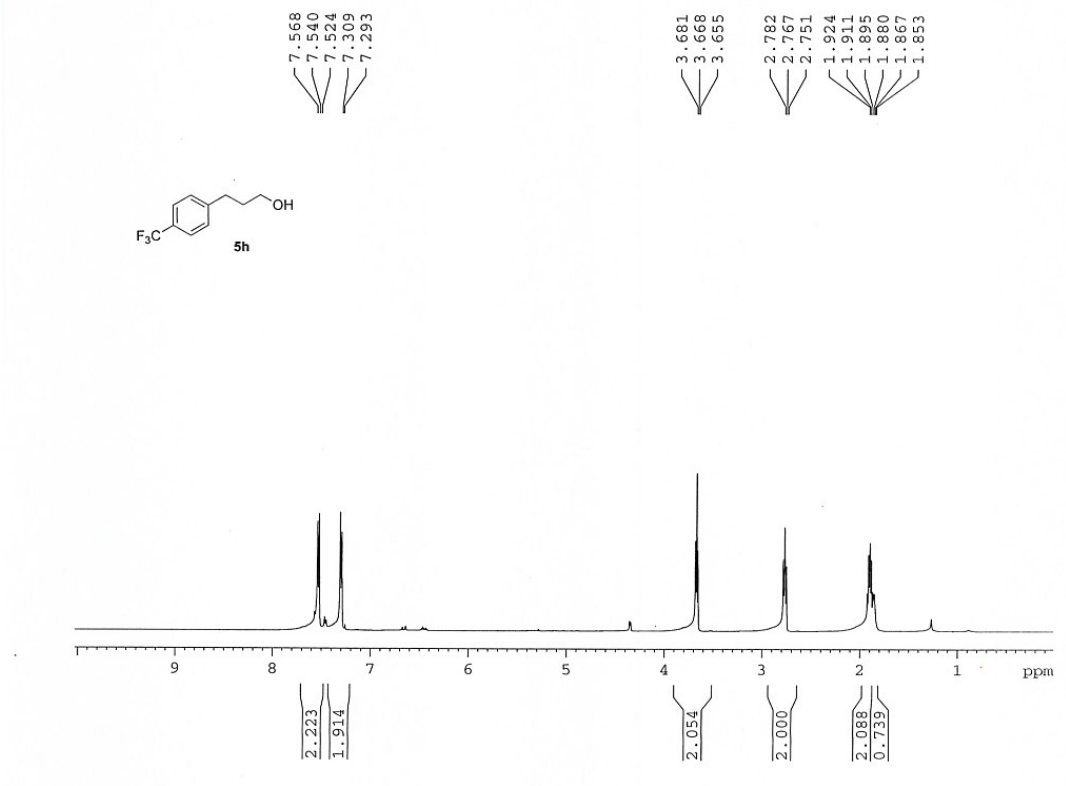

157.328
130.017
129.927
127.097
120.623
110.247
61.875
55.259
32.832
25.874
OH
OMe
5b
200
180
160
140
120
100
80
60
40
20
0
ppm

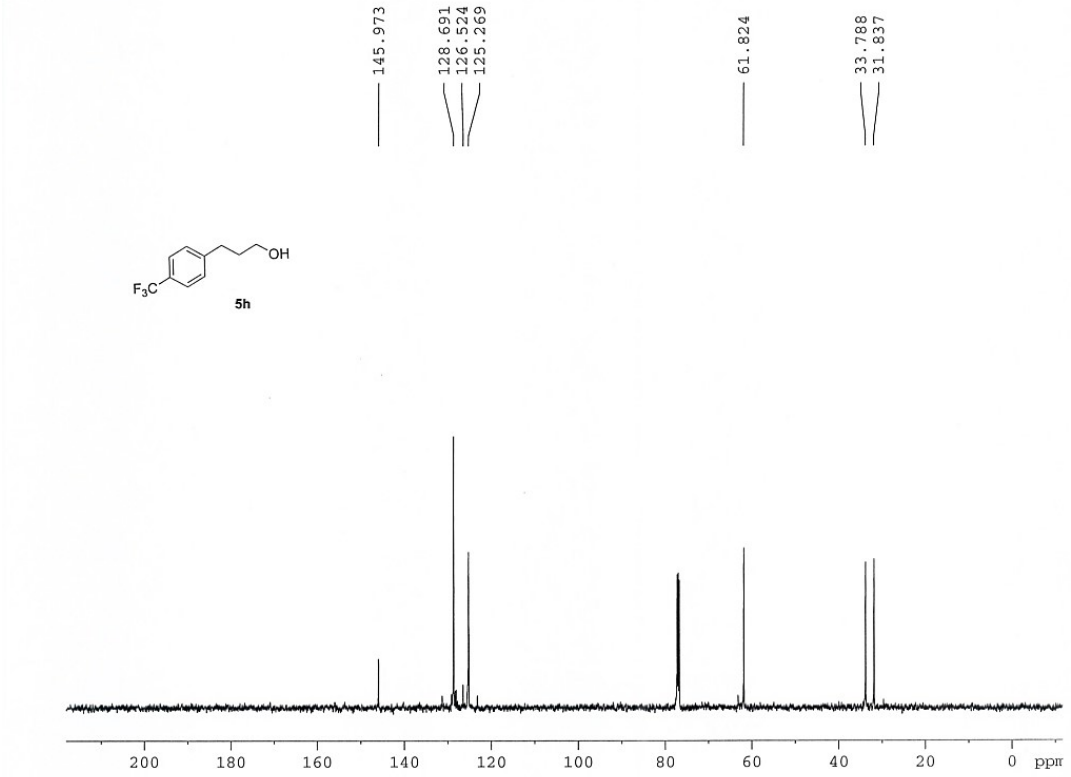

7.401
7.384
7.072
7.056
3.656
3.643
3.631
2.669
2.654
2.638
1.878
1.865
1.853
1.850
1.848
1.835
1.822
OH
Br
5d
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
2.244
1.904
2.000
2.057
1.832

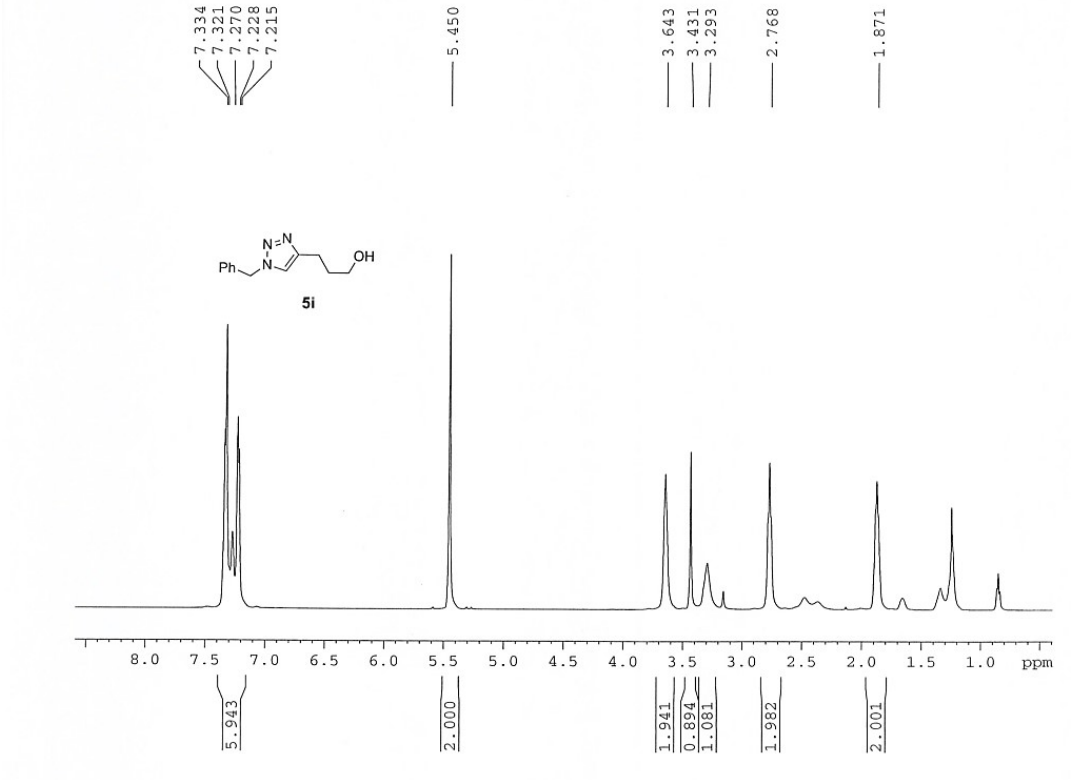

140.710
131.625
131.354
130.126
119.502
77.250
76.996
76.742
61.855
33.900
31.370
OH
Br
5d
200
180
160
140
120
100
80
60
40
20
0
ppm

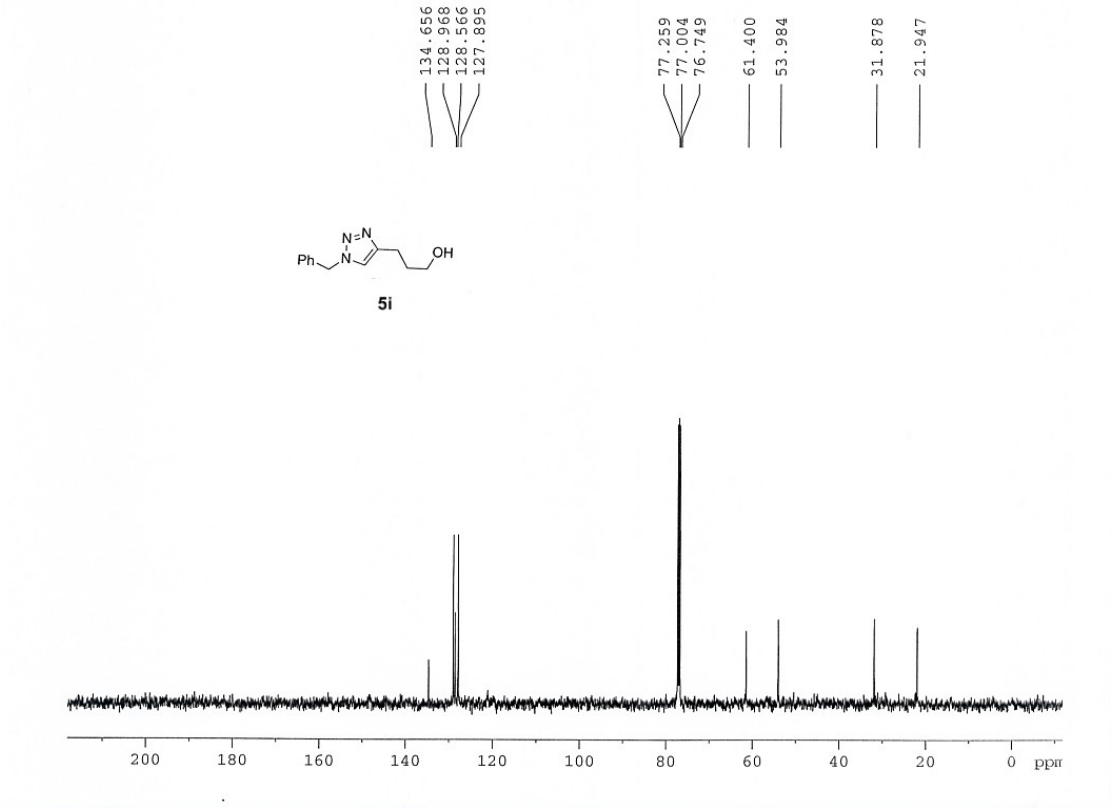

8.042
8.027
8.010
7.528
7.513
7.440
7.424
7.409
3.683
3.671
3.658
2.829
2.814
2.798
2.010
1.934
1.921
1.909
1.906
1.903
1.890
1.878
O2N
OH
5f
9
8
7
6
5
4
3
2
1
ppm
1.891
1.061
0.861
2.000
2.037
1.146
1.805


148.267
143.862
134.734
129.163
123.140
120.983
77.250
76.996
76.742
61.521
33.610
31.591
O2N
OH
5f
200
180
160
140
120
100
80
60
40
20
0
ppm

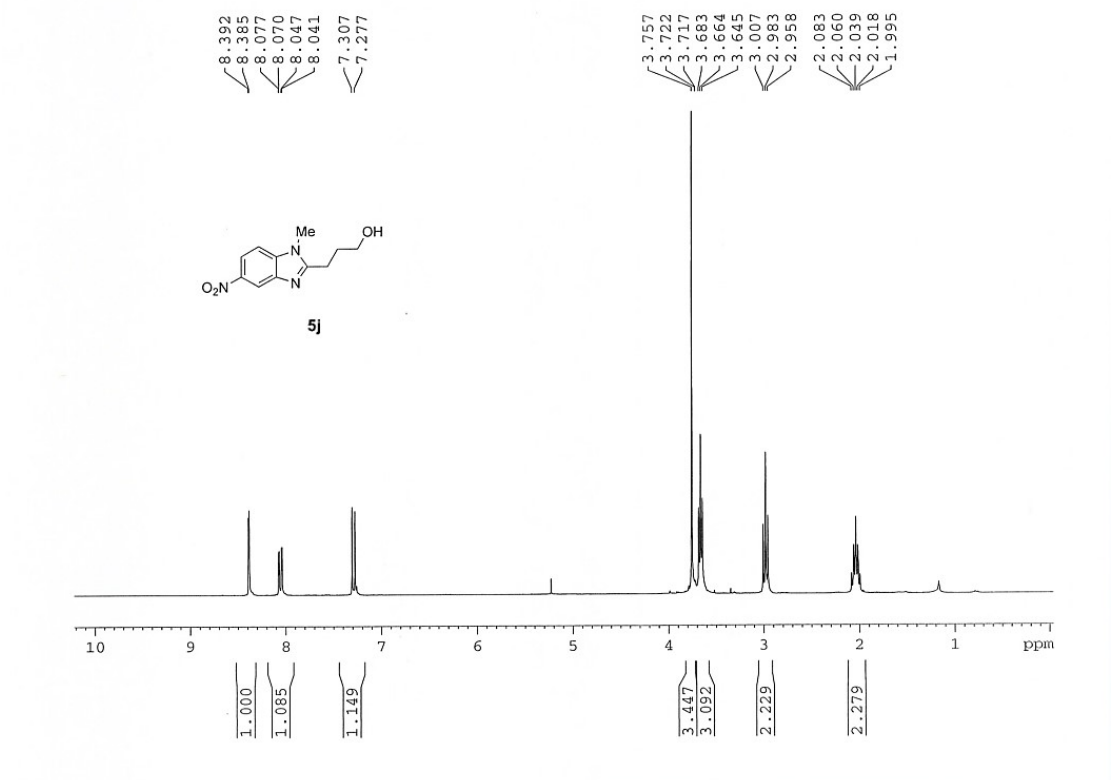

7.568
7.540
7.524
7.309
7.293
3.681
3.668
3.655
2.782
2.767
2.751
1.924
1.911
1.895
1.880
1.867
1.853
OH
F3C
5h
9
8
7
6
5
4
3
2
1
ppm
2.223
1.914
2.054
2.000
2.088
0.739


145.973
128.691
126.524
125.269
61.824
33.788
31.837
OH
F3C
5h
200
180
160
140
120
100
80
60
40
20
0
ppm

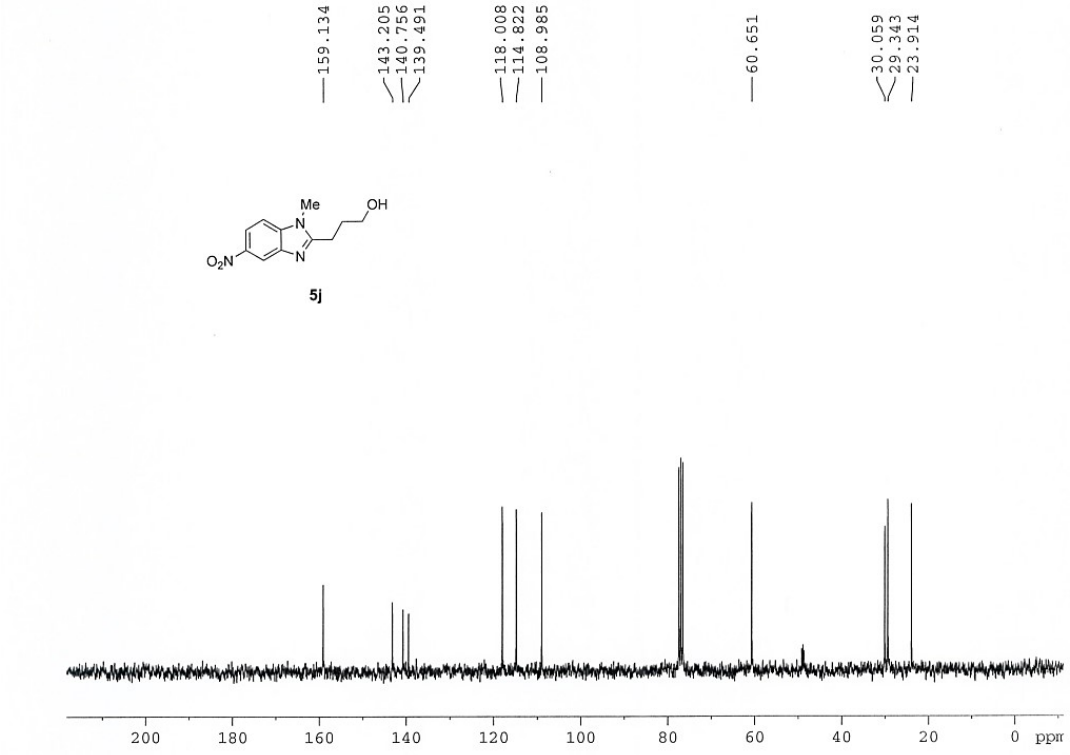

7.334
7.321
7.270
7.228
7.215
5.450
3.643
3.431
3.293
2.768
1.871
N
N
N
Ph
OH
5i
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
5.943
2.000
1.941
0.894
1.081
1.982
2.001


134.656
128.968
128.566
127.895
77.259
77.004
76.749
61.400
53.984
31.878
21.947
N=N
N
Ph
OH
200
180
160
140
120
100
80
60
40
20
0
ppm

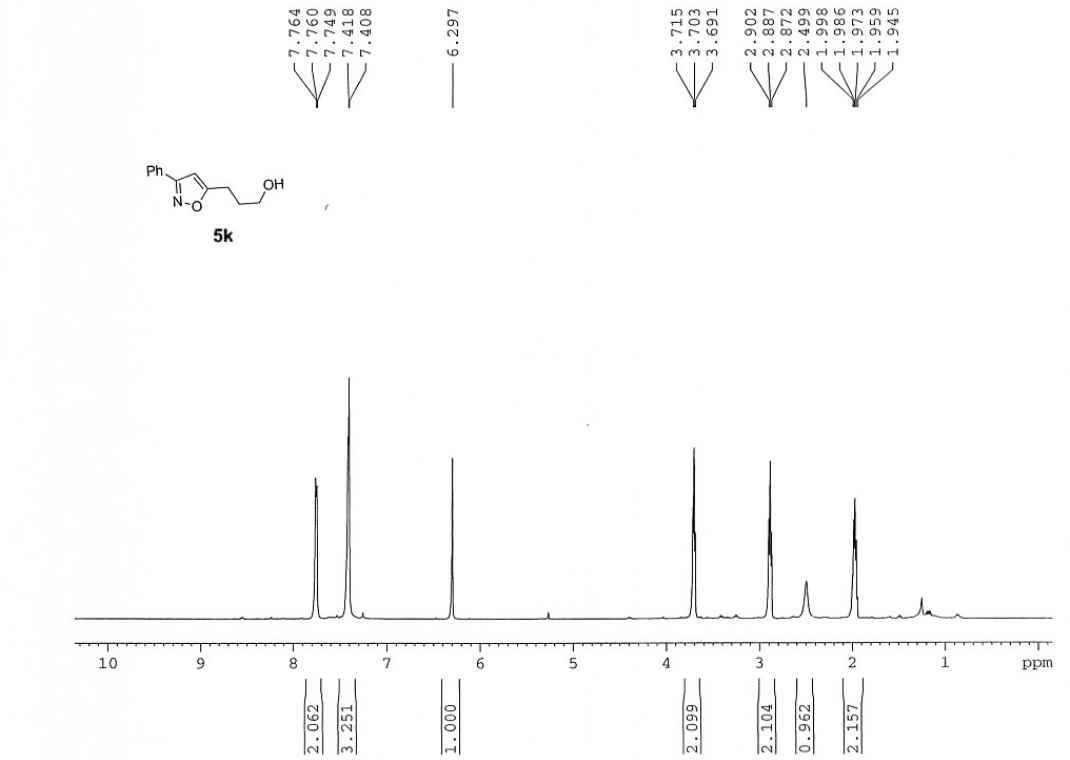
5i


8.392
8.385
8.077
8.070
8.047
8.041
7.307
7.277
3.757
3.722
3.717
3.683
3.664
3.645
3.007
2.983
2.958
2.083
2.060
2.039
2.018
1.995
Me
N
OH
O2N
10
9
8
7
6
5
4
3
2
1
ppm
1.000
1.085
1.149
3.447
3.092
2.229
2.279

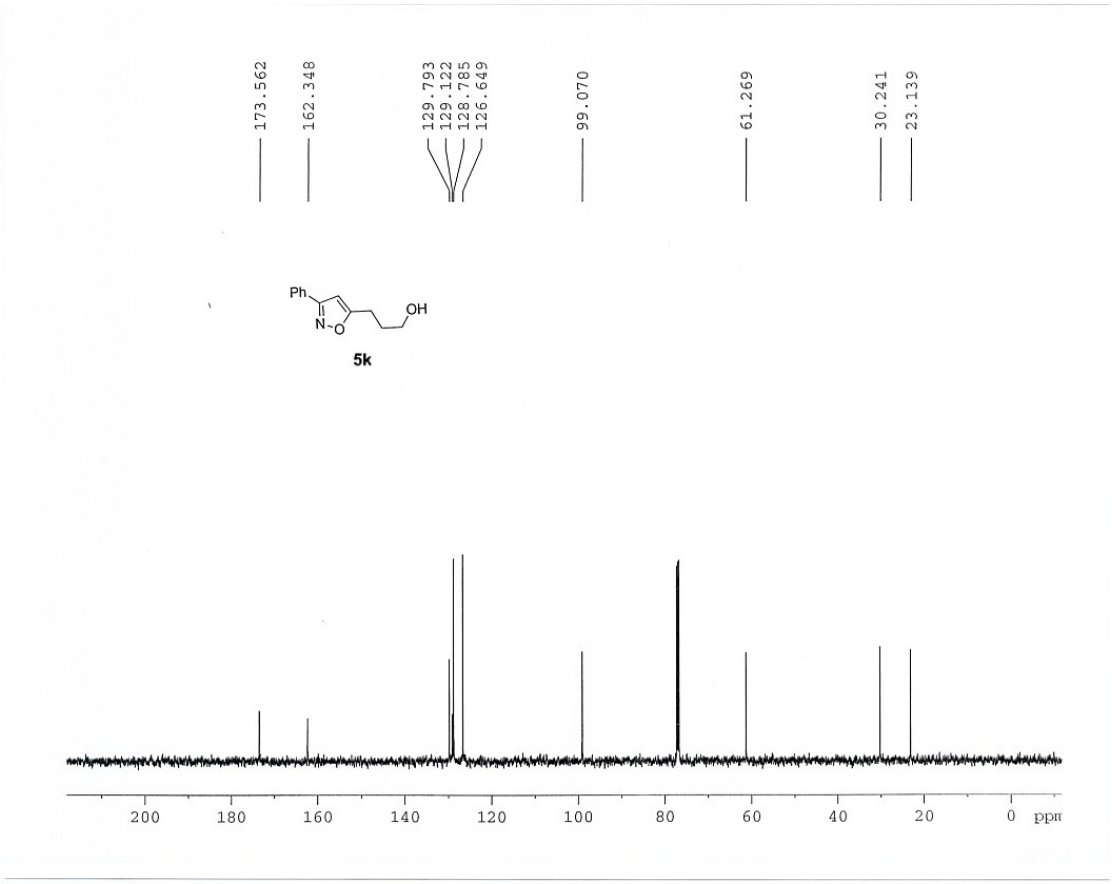
5j

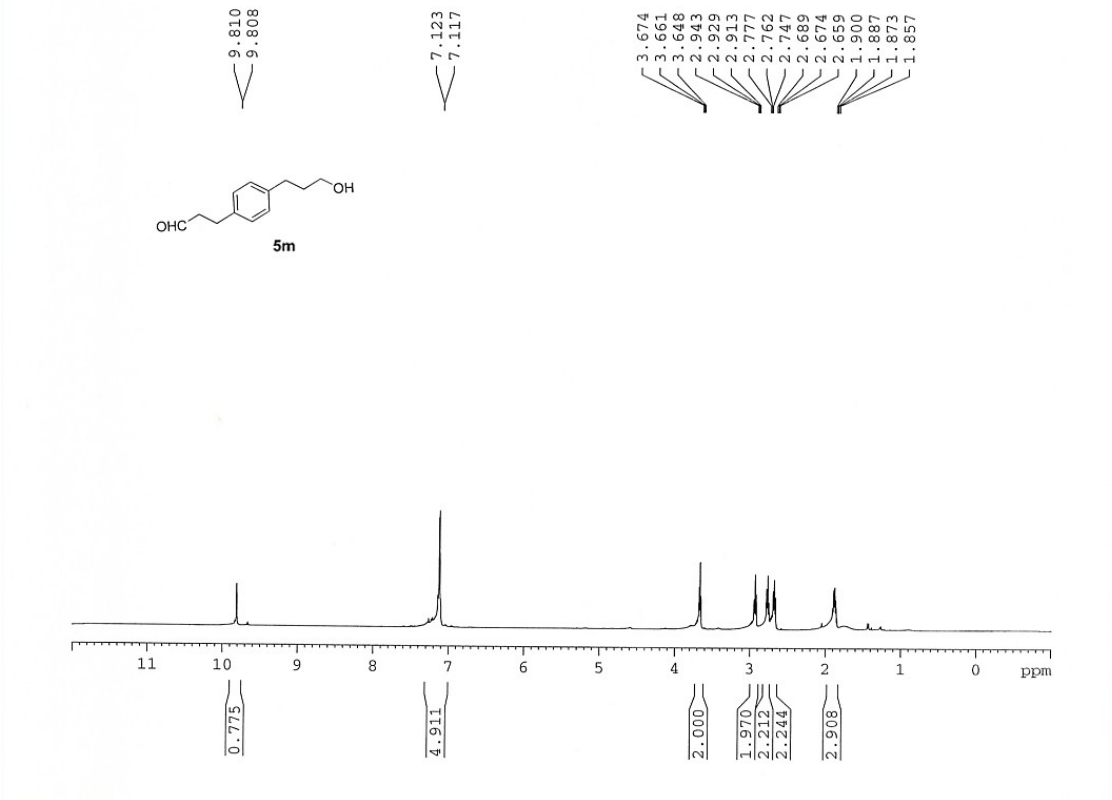

159.134
143.205
140.756
139.491
118.008
114.822
108.985
60.651
30.059
29.343
23.914
Me
OH
N
N
O2N
200
180
160
140
120
100
80
60
40
20
0
ppm

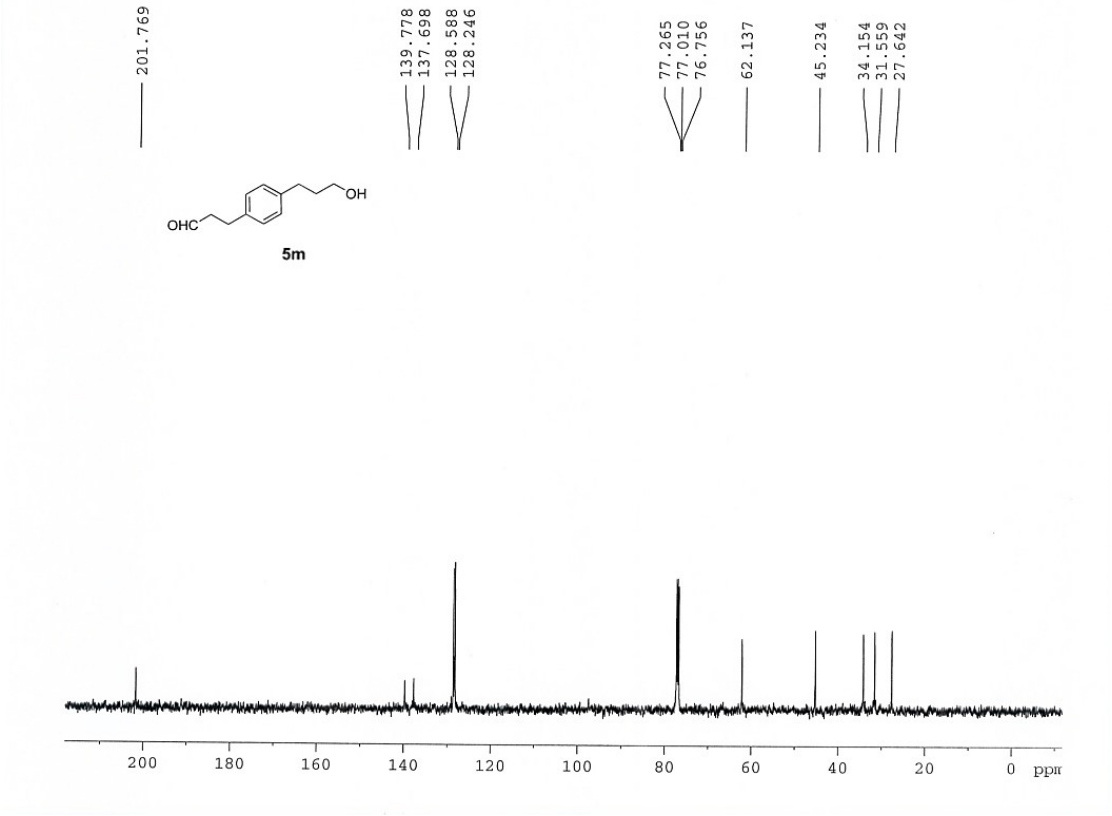
5j

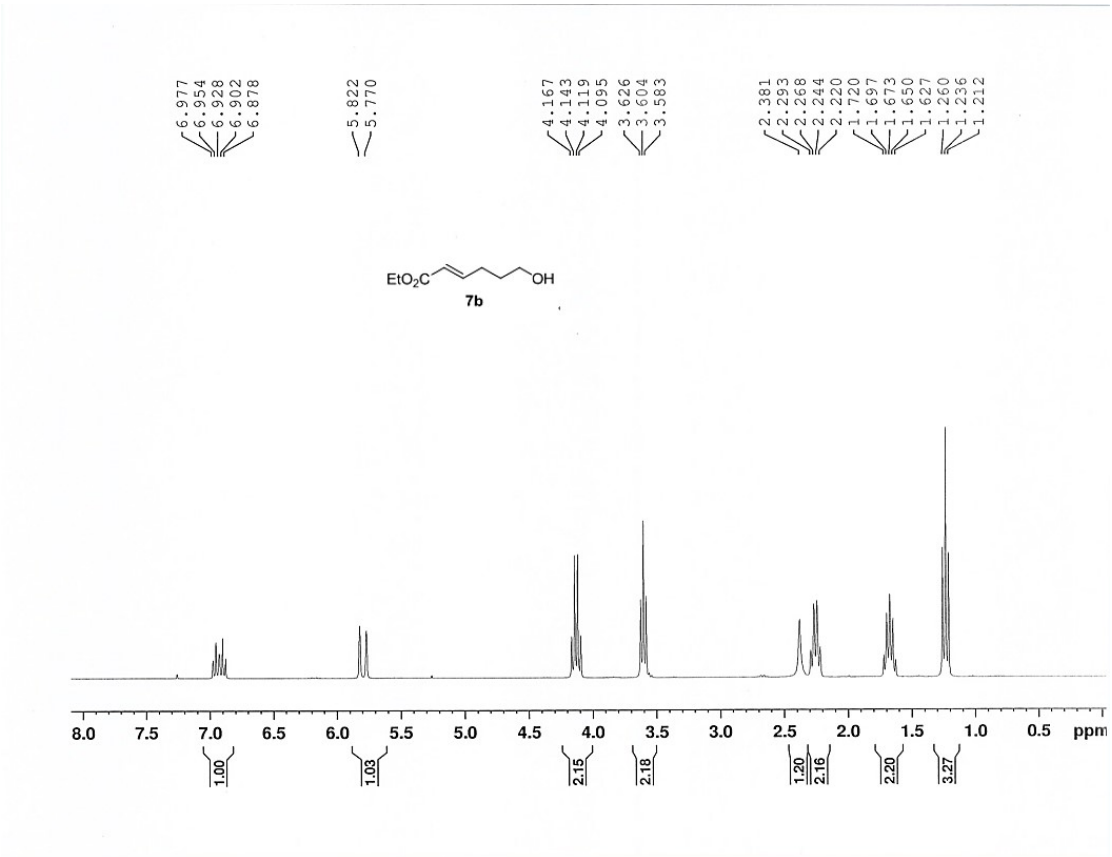

7.764
7.760
7.749
7.418
7.408
6.297
3.715
3.703
3.691
2.902
2.887
2.872
2.499
1.998
1.986
1.973
1.959
1.945
Ph
N
O
OH
10
9
8
7
6
5
4
3
2
1
ppm
2.062
3.251
1.000
2.099
2.104
0.962
2.157

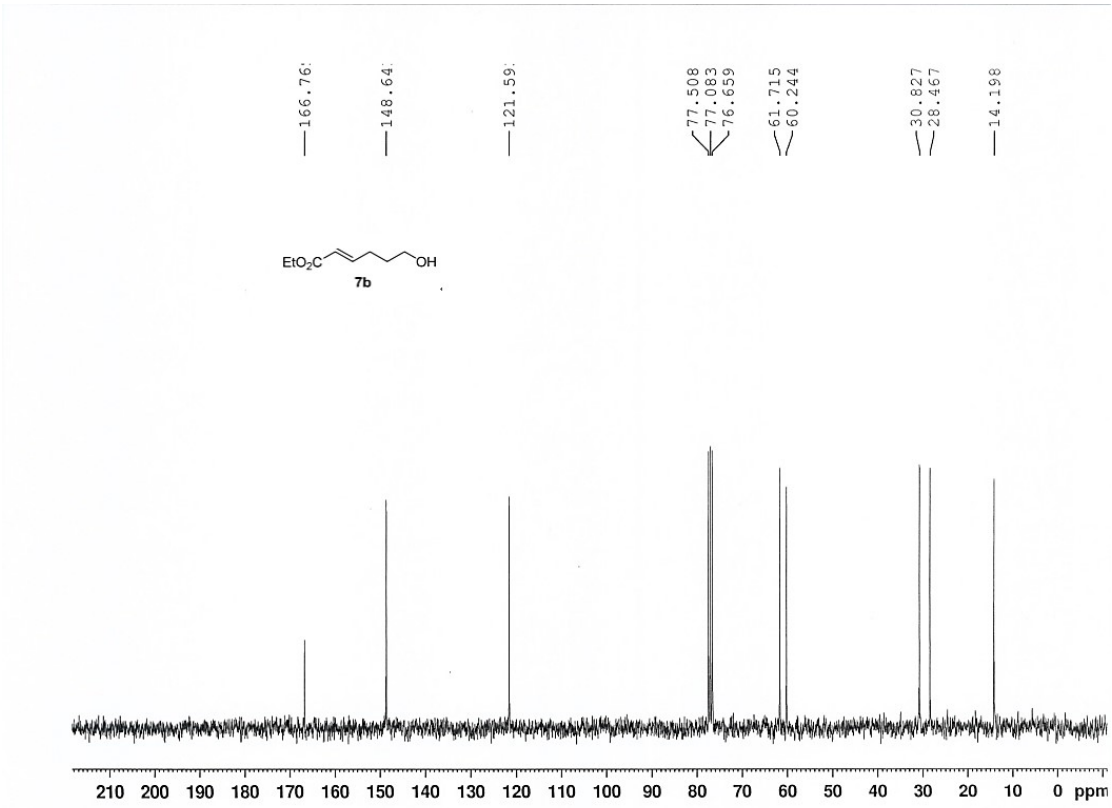
5k

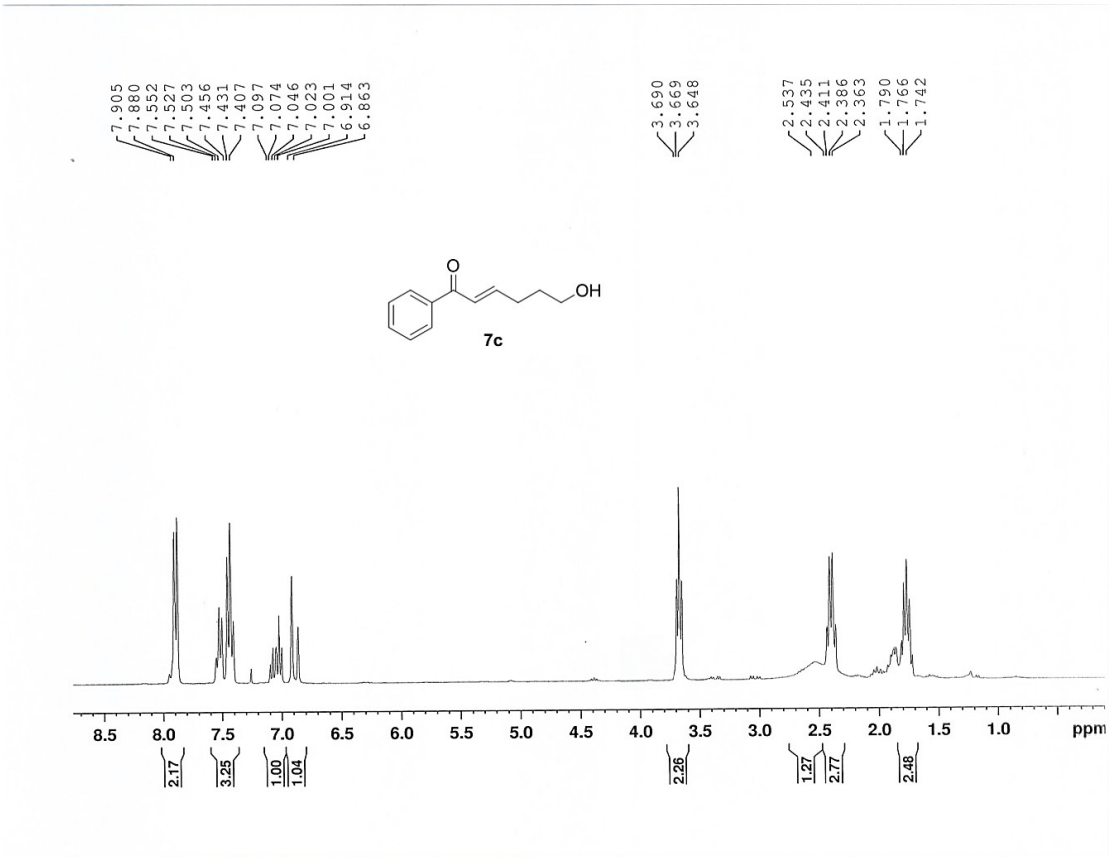

173.562
162.348
129.793
129.122
128.785
126.649
99.070
61.269
30.241
23.139
Ph
N
O
OH
5k
200
180
160
140
120
100
80
60
40
20
0
ppm

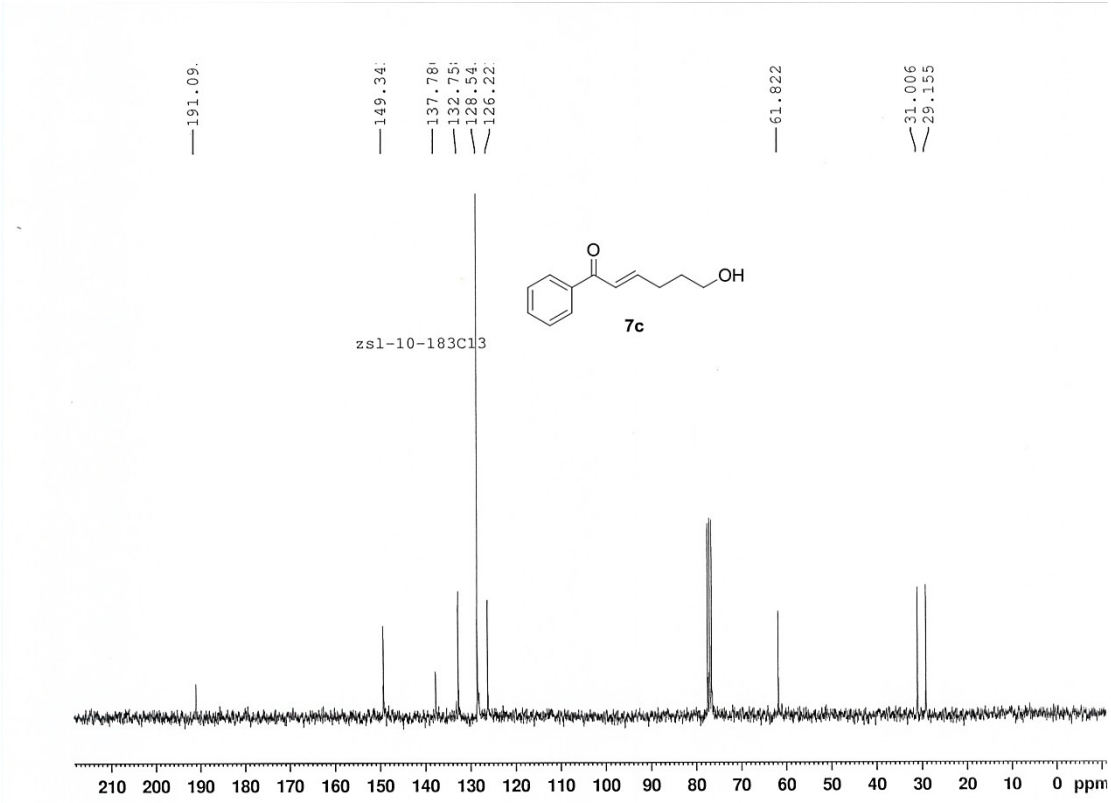

9.810
9.808
7.123
7.117
3.674
3.661
3.648
2.943
2.929
2.913
2.777
2.762
2.747
2.689
2.674
2.659
1.900
1.887
1.873
1.857
OHC
OH
5m
11
10
9
8
7
6
5
4
3
2
1
0
ppm
0.775
4.911
2.000
1.970
2.212
2.244
2.908

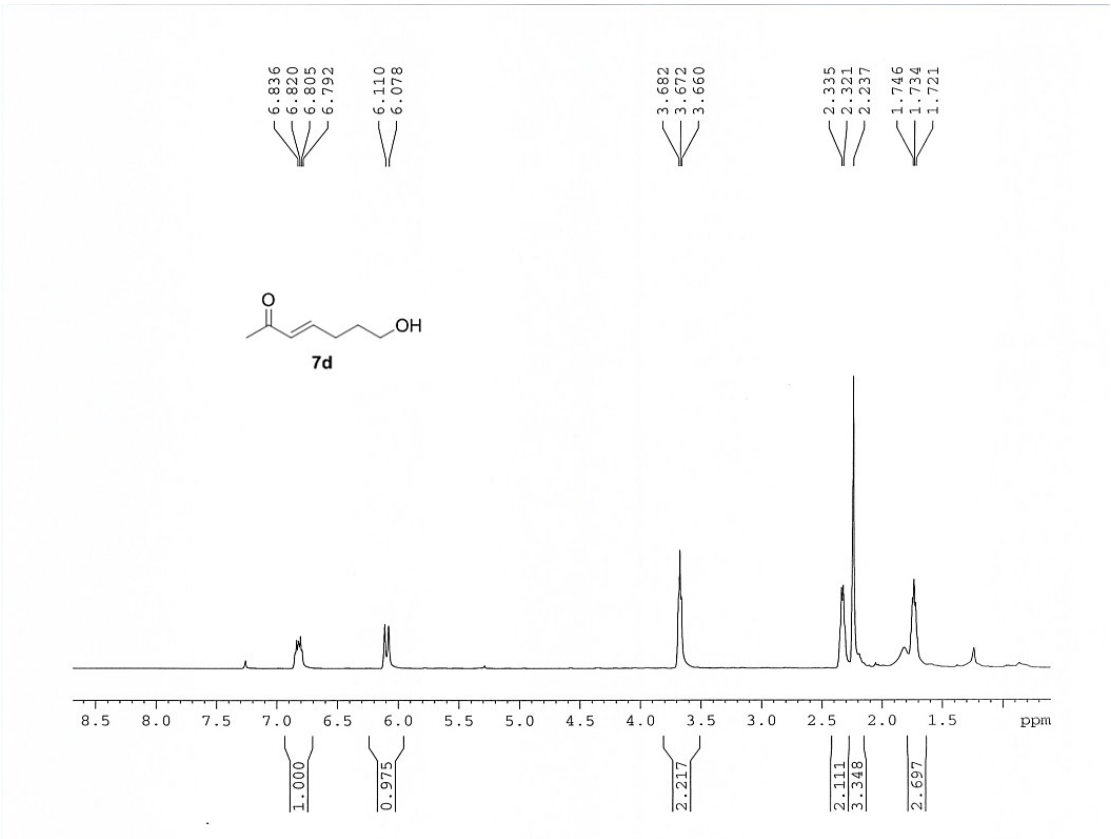

201.769
139.778
137.698
128.588
128.246
77.265
77.010
76.756
62.137
45.234
34.154
31.559
27.642
OH
OHC
5m
200
180
160
140
120
100
80
60
40
20
0
ppm

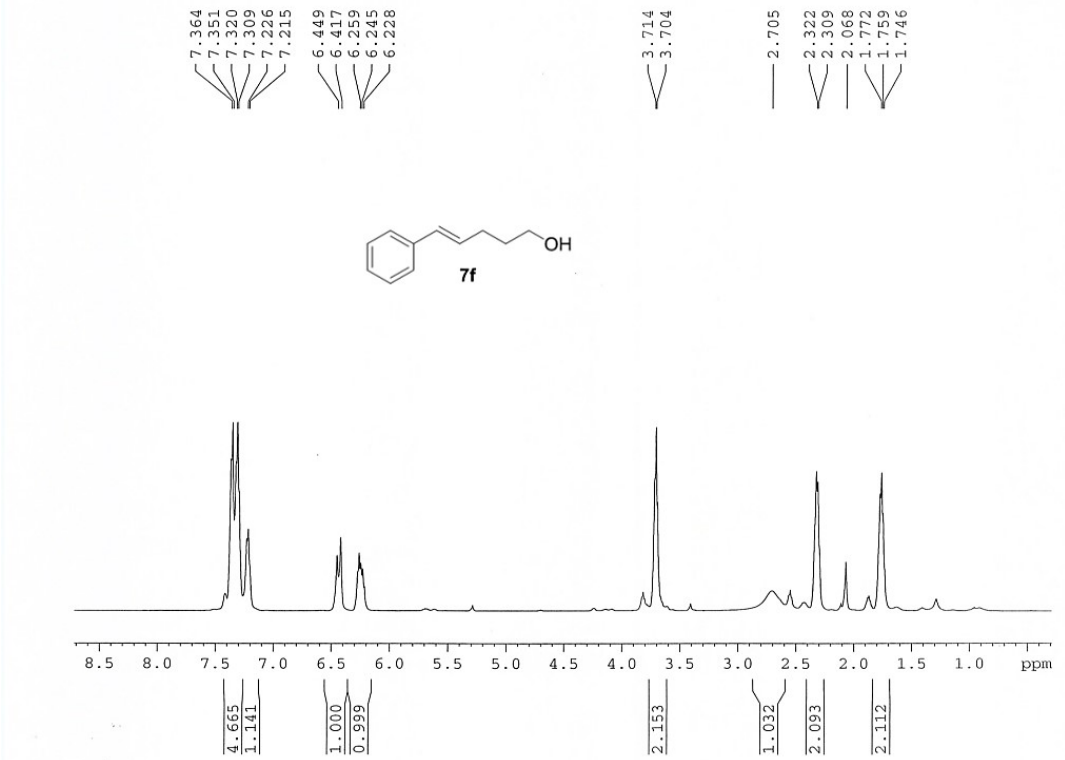

6.977
6.954
6.928
6.902
6.878
5.822
5.770
4.167
4.143
4.119
4.095
3.626
3.604
3.583
2.381
2.293
2.268
2.244
2.220
1.720
1.697
1.673
1.650
1.627
1.260
1.236
1.212
EtO2C
OH
7b
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
1.00
1.03
2.15
2.18
1.20
2.16
2.20
3.27

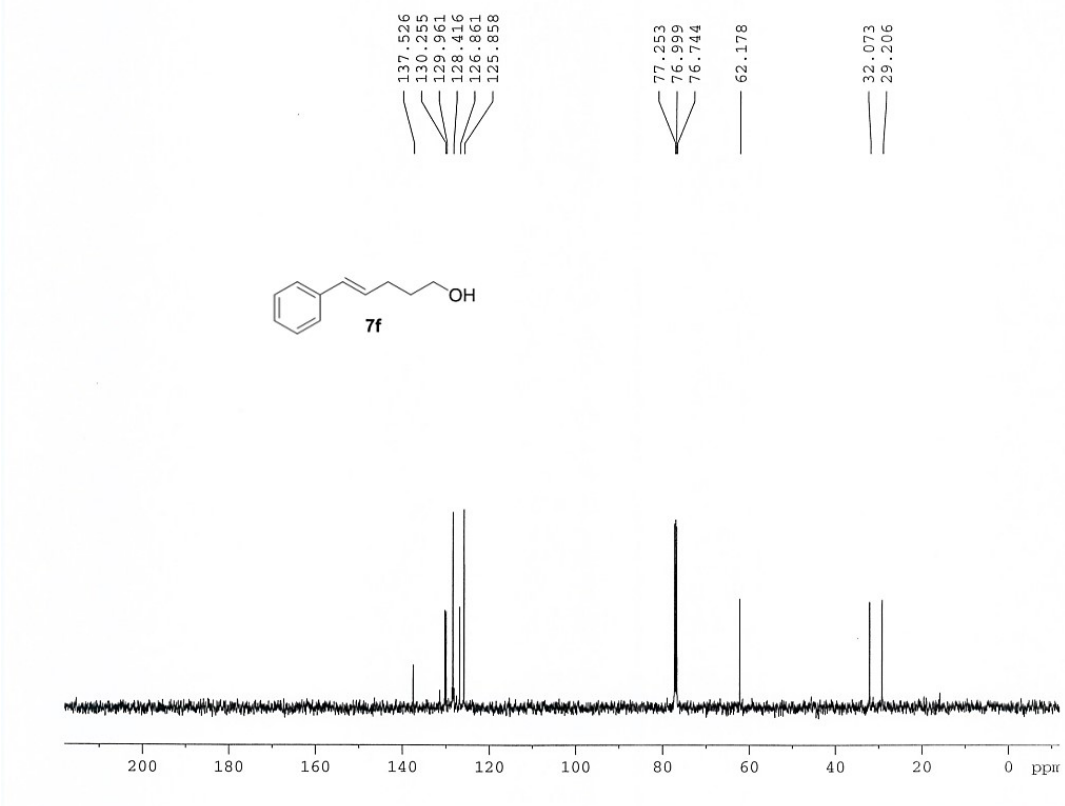

166.76
148.64
121.59
77.508
77.083
76.659
61.715
60.244
30.827
28.467
14.198
EtO2C
OH
7b
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

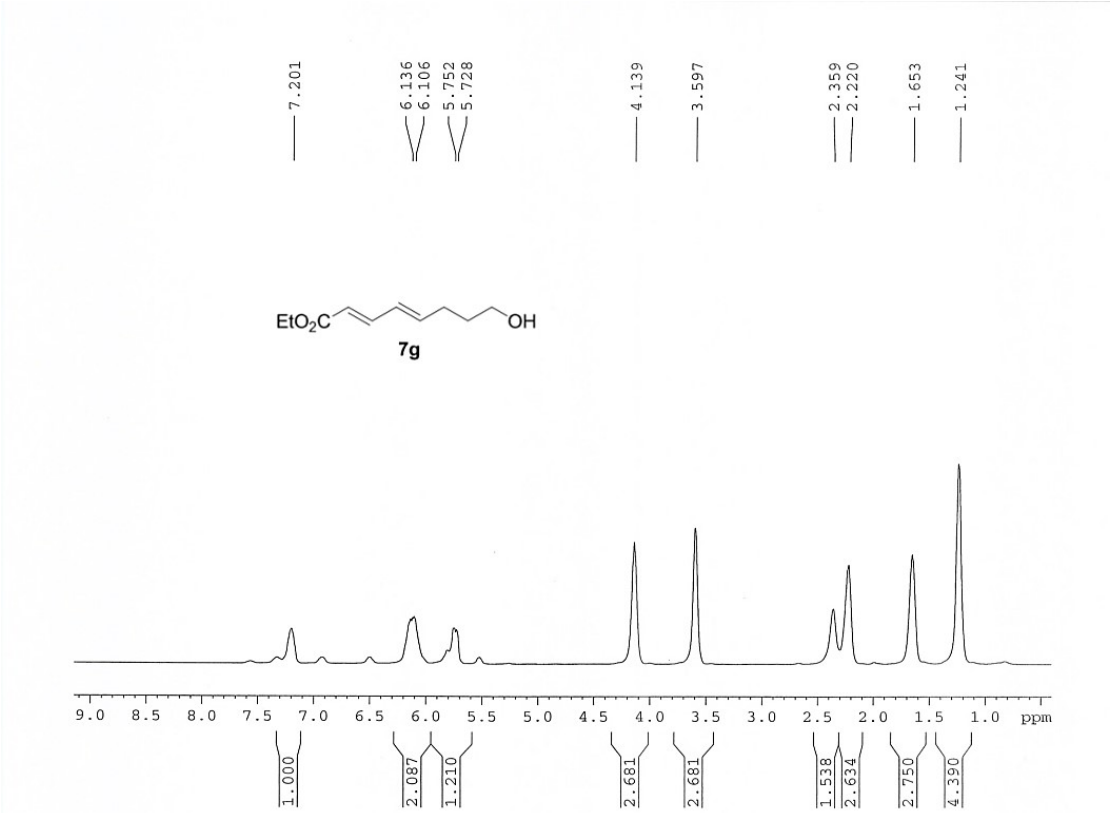

7.905
7.880
7.552
7.527
7.503
7.456
7.431
7.407
7.097
7.074
7.046
7.023
7.001
6.914
6.863
3.690
3.669
3.648
2.537
2.435
2.411
2.386
2.363
1.790
1.766
1.742
O
OH
7c
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
2.17
3.25
1.00
1.04
2.26
1.27
2.77
2.48

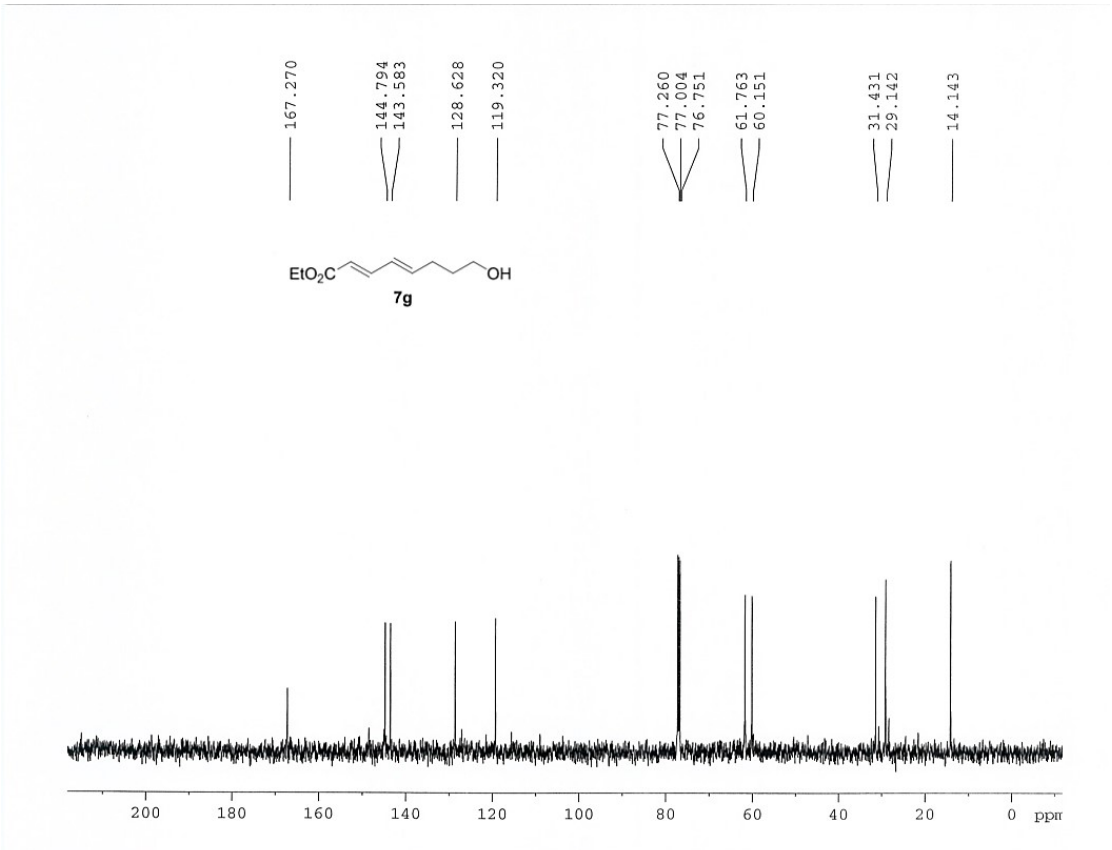

191.09
149.34
137.78
132.75
128.54
126.22
61.822
31.006
29.155
zsl-10-183C13
O
OH
7c
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

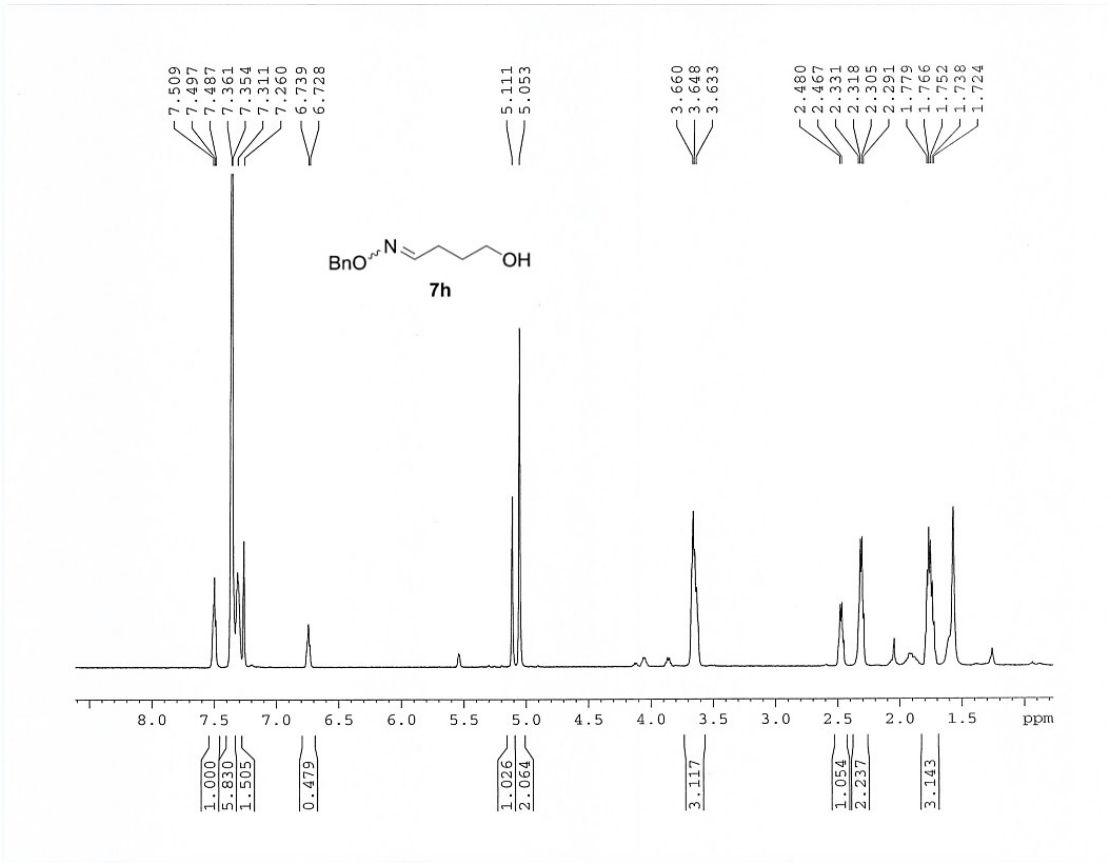

6.836
6.820
6.805
6.792
6.110
6.078
3.682
3.672
3.660
2.335
2.321
2.237
1.746
1.734
1.721
O
OH
7d
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 ppm
1.000
0.975
2.217
2.111
3.348
2.697

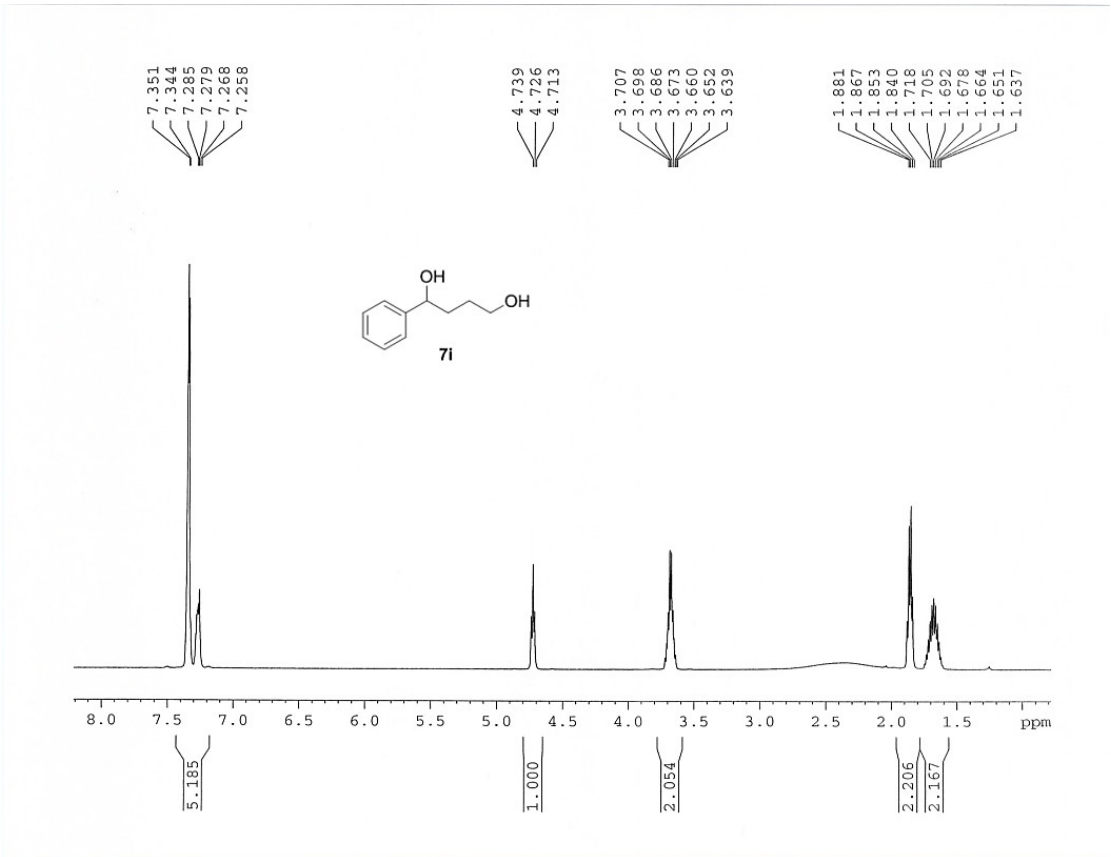

7.364
7.351
7.320
7.309
7.226
7.215
6.449
6.417
6.259
6.245
6.228
3.714
3.704
2.705
2.322
2.309
2.068
1.772
1.759
1.746
OH
7f
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
4.665
1.141
1.000
0.999
2.153
1.032
2.093
2.112

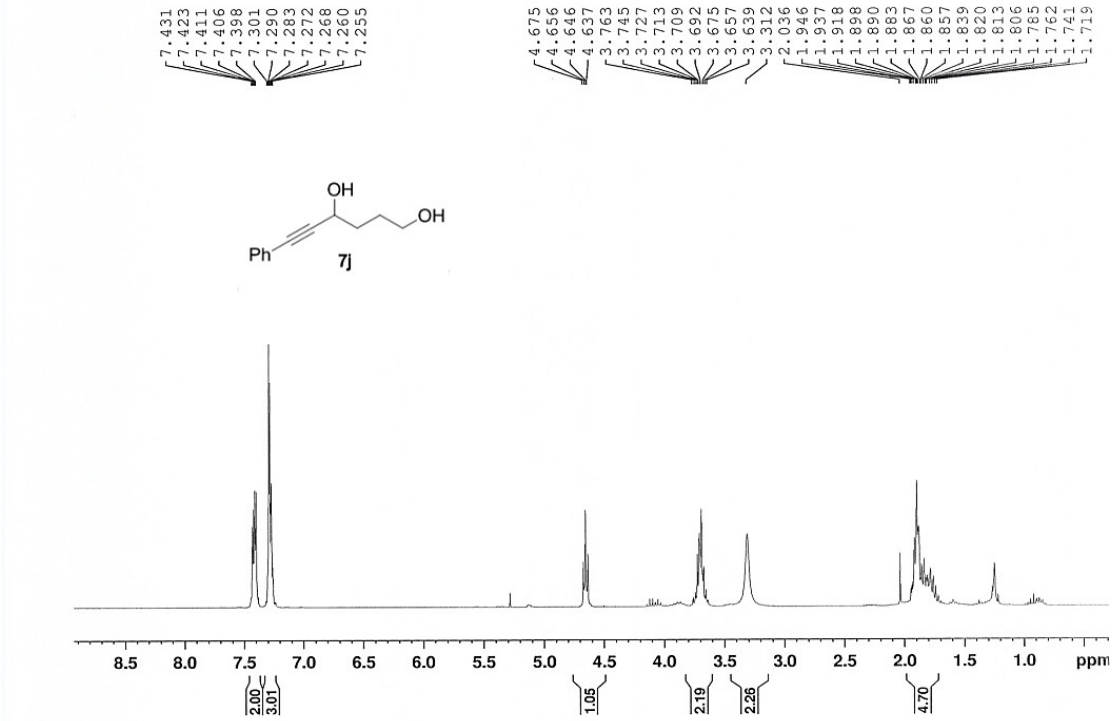

137.526
130.255
129.961
128.416
126.861
125.858
77.253
76.999
76.744
62.178
32.073
29.206
OH
7f
200 180 160 140 120 100 80 60 40 20 0 ppm

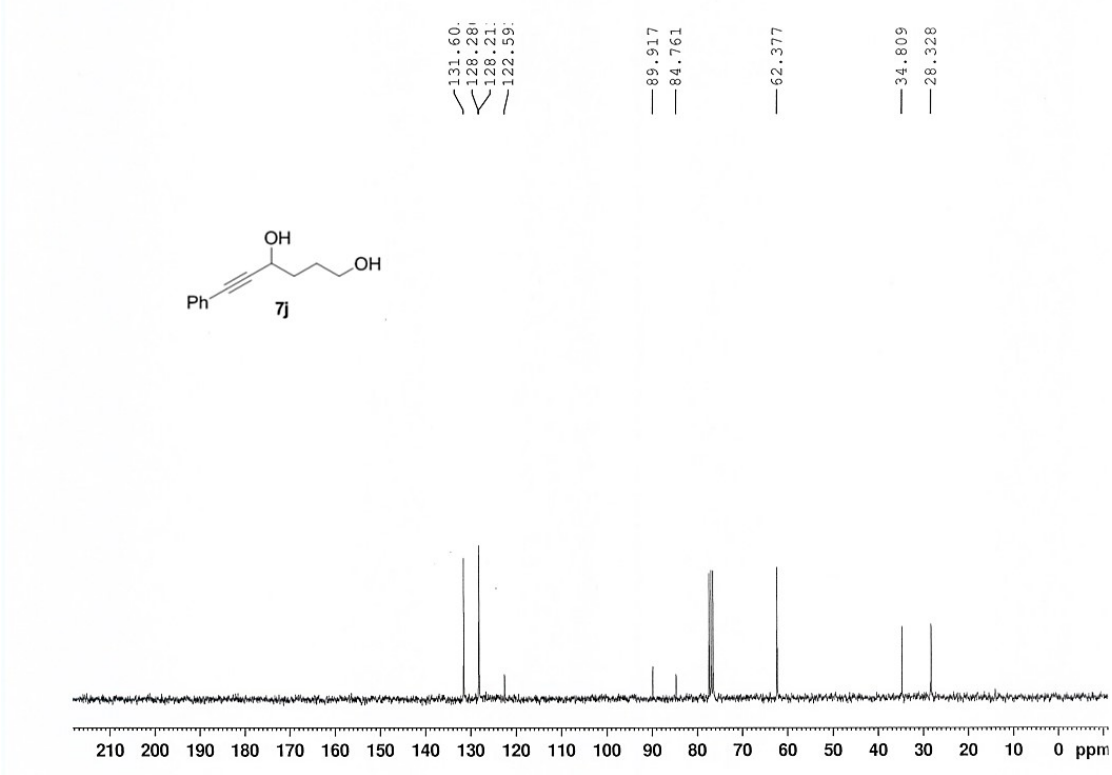

7.201
6.136
6.106
5.752
5.728
4.139
3.597
2.359
2.220
1.653
1.241
EtO2C
OH
7g
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
1.000
2.087
1.210
2.681
2.681
1.538
2.634
2.750
4.390

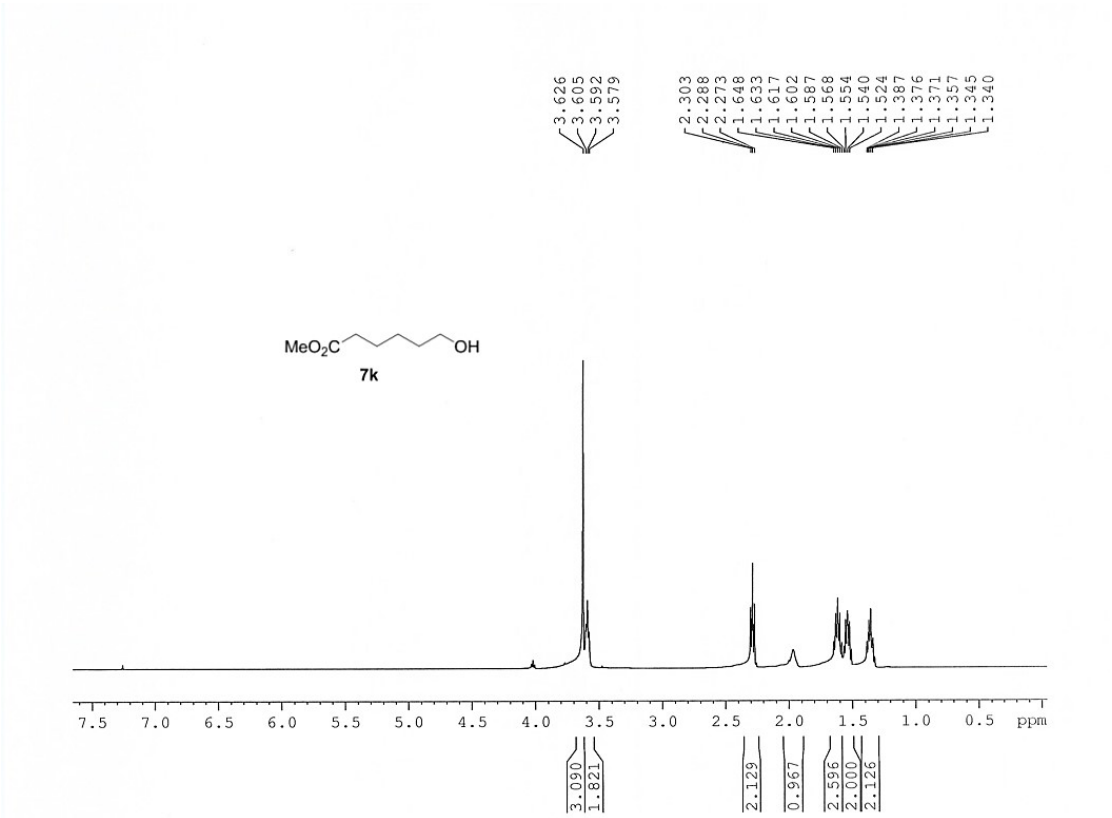

167.270
144.794
143.583
128.628
119.320
77.260
77.004
76.751
61.763
60.151
31.431
29.142
14.143
EtO2C
OH
7g
200 180 160 140 120 100 80 60 40 20 0 ppm

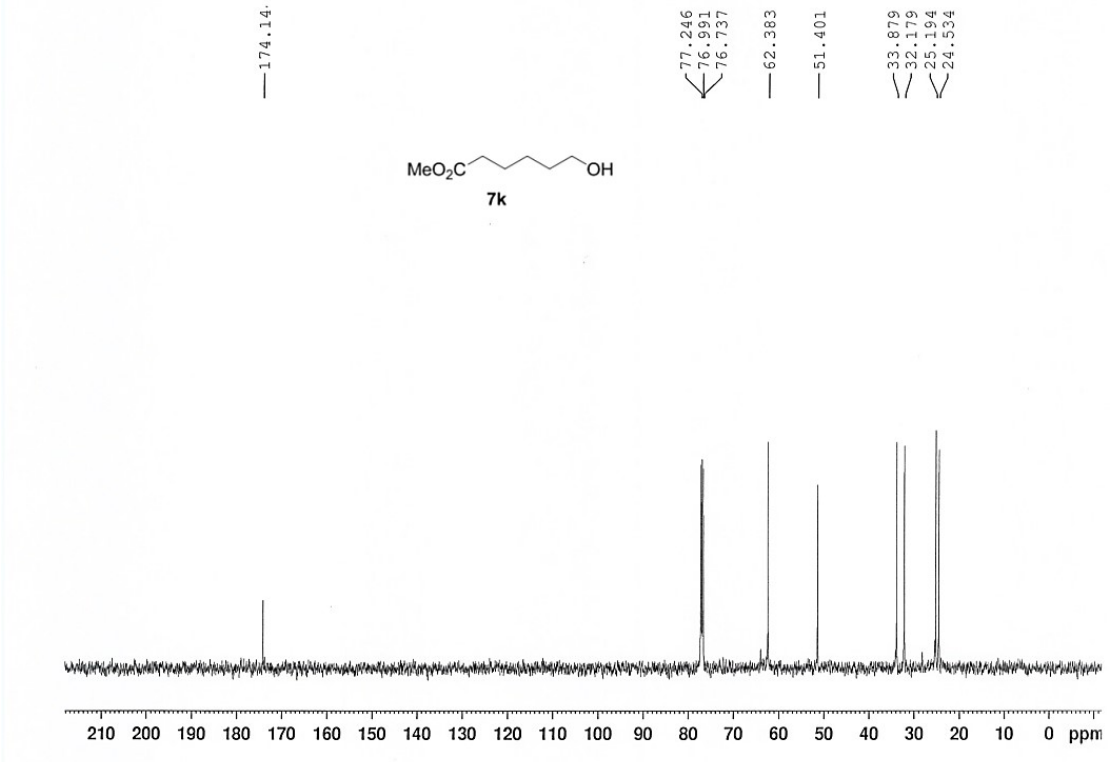

7.509
7.497
7.487
7.361
7.354
7.311
7.260
6.739
6.728
5.111
5.053
3.660
3.648
3.633
2.480
2.467
2.331
2.318
2.305
2.291
1.779
1.766
1.752
1.738
1.724
BnO
N
OH
7h
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
ppm
1.000
5.830
1.505
0.479
1.026
2.064
3.117
1.054
2.237
3.143

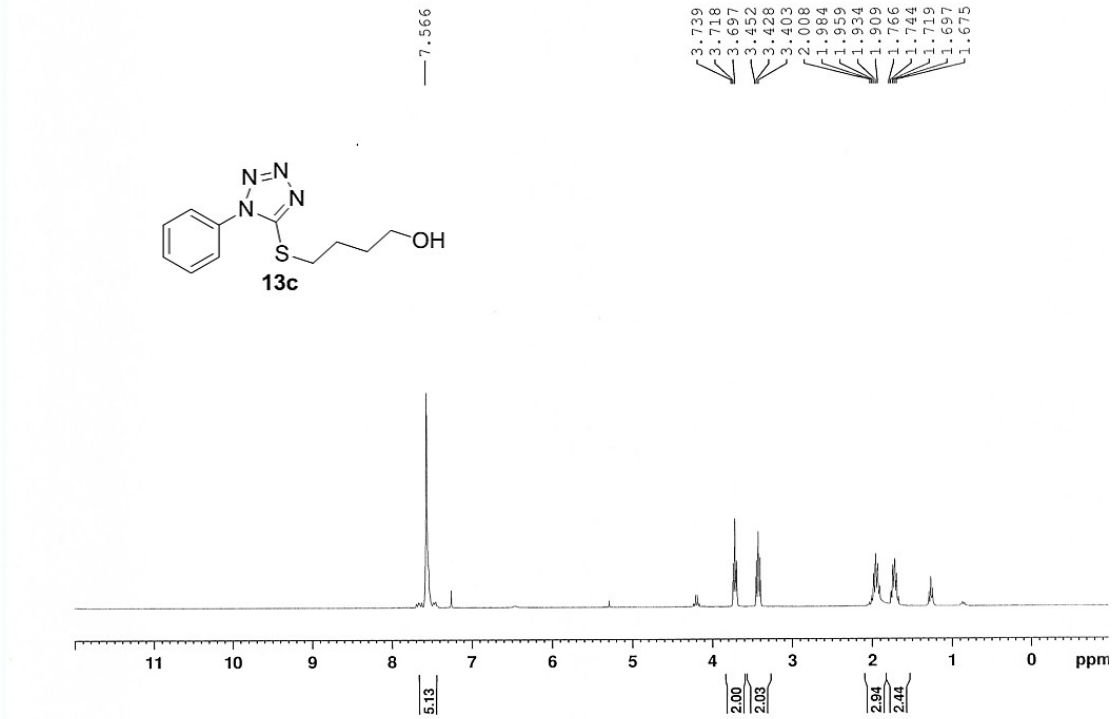

7.351
7.344
7.285
7.279
7.268
7.258
4.739
4.726
4.713
3.707
3.698
3.686
3.673
3.660
3.652
3.639
1.881
1.867
1.853
1.840
1.718
1.705
1.692
1.678
1.664
1.651
1.637
OH
OH
7i
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
ppm
5.185
1.000
2.054
2.206
2.167

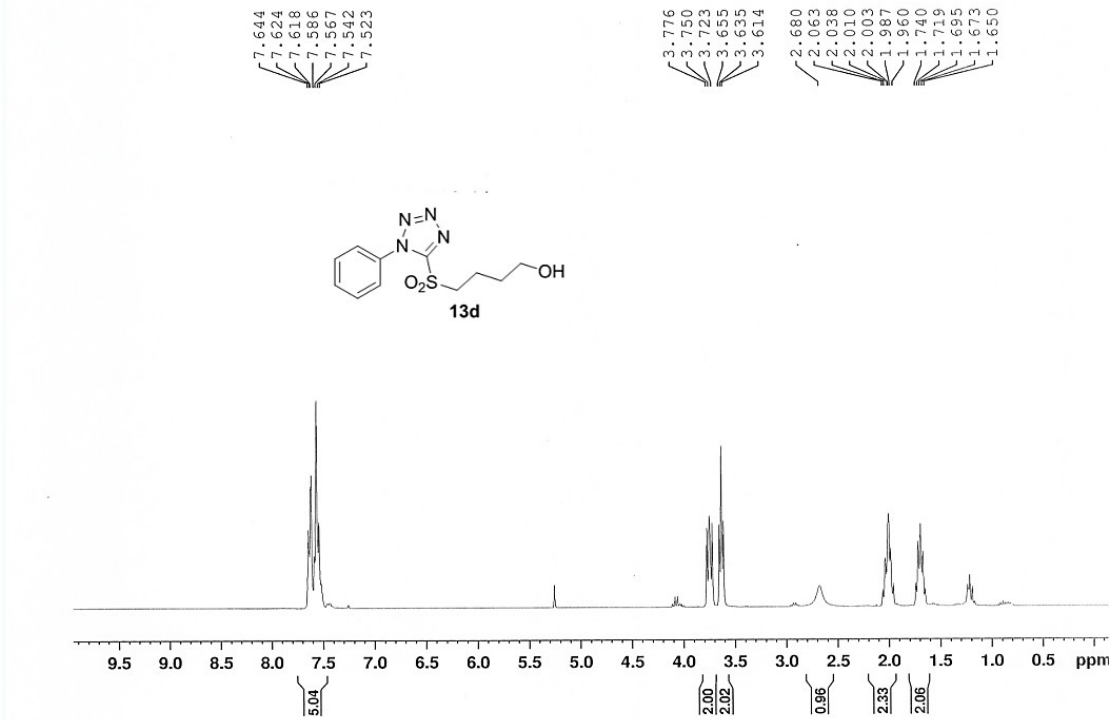

7.431
7.423
7.411
7.406
7.398
7.301
7.290
7.283
7.272
7.268
7.260
7.255
4.675
4.656
4.646
4.637
3.763
3.745
3.727
3.713
3.709
3.692
3.675
3.657
3.639
3.312
2.036
1.946
1.937
1.918
1.898
1.890
1.883
1.867
1.860
1.857
1.839
1.820
1.813
1.806
1.785
1.762
1.741
1.719
OH
OH
Ph
7j
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
2.00
3.01
1.05
2.19
2.26
4.70

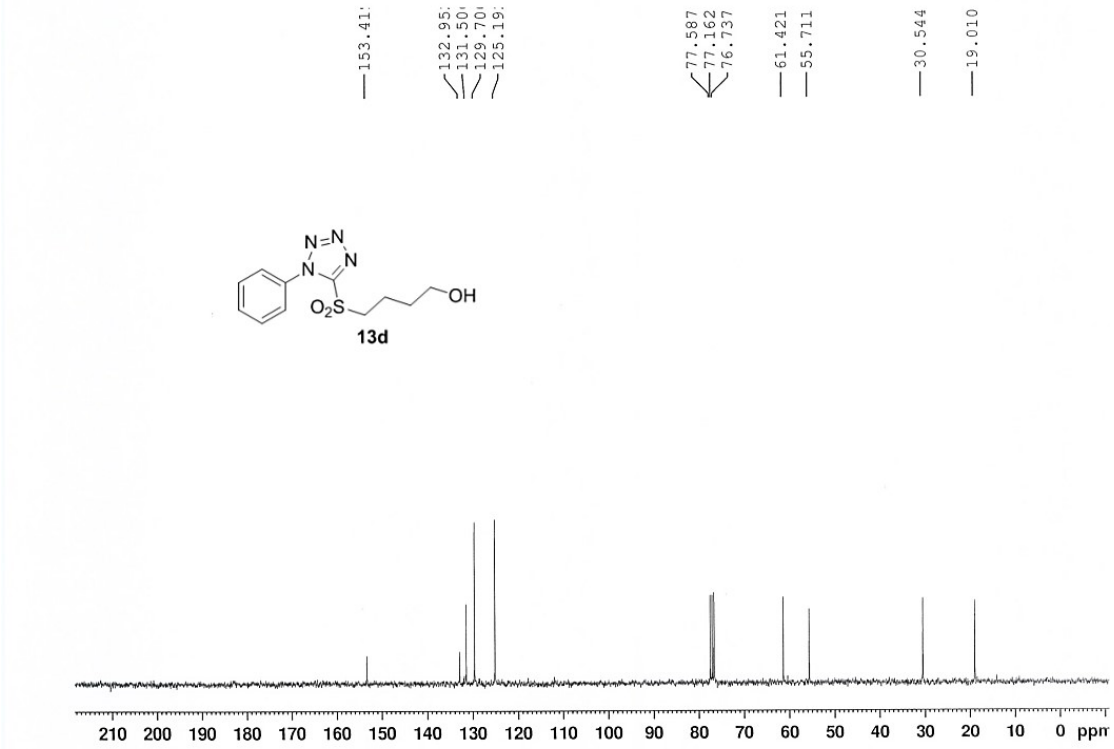

131.60.
128.28
128.21.
122.59
89.917
84.761
62.377
34.809
28.328
OH
OH
Ph
7j
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

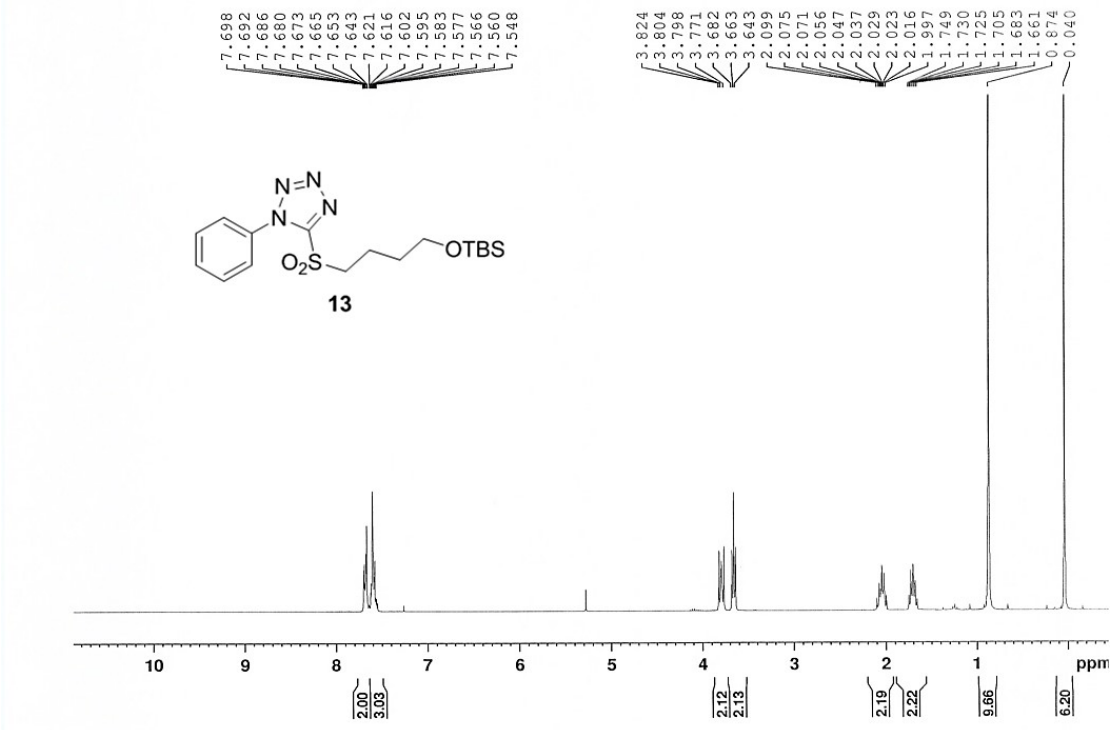

3.626
3.605
3.592
3.579
2.303
2.288
2.273
1.648
1.633
1.617
1.602
1.587
1.568
1.554
1.540
1.524
1.387
1.376
1.371
1.357
1.345
1.340
MeO2C
OH
7k
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
3.090
1.821
2.129
0.967
2.596
2.000
2.126

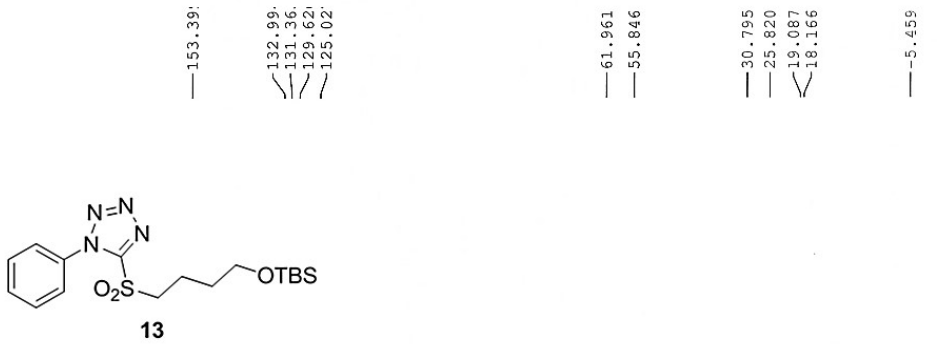

174.14
77.246
76.991
76.737
62.383
51.401
33.879
32.179
25.194
24.534
MeO2C
OH
7k
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


7.566
3.739
3.718
3.697
3.452
3.428
3.403
2.008
1.984
1.959
1.934
1.909
1.766
1.744
1.719
1.697
1.675
OH
13c
11
10
9
8
7
6
5
4
3
2
1
0
ppm
5.13
2.00
2.03
2.94
2.44


7.644
7.624
7.618
7.586
7.567
7.542
7.523
3.776
3.750
3.723
3.655
3.635
3.614
2.680
2.063
2.038
2.010
2.003
1.987
1.960
1.740
1.719
1.695
1.673
1.650
OH
13d
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
ppm
5.04
2.00
2.02
0.96
2.33
2.06

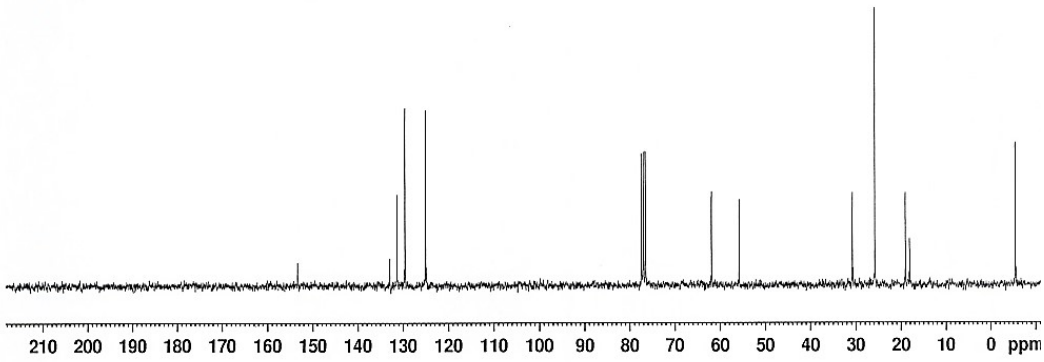

153.41
132.95
131.50
129.70
125.19
77.587
77.162
76.737
61.421
55.711
30.544
19.010
OH
O2S
13d
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

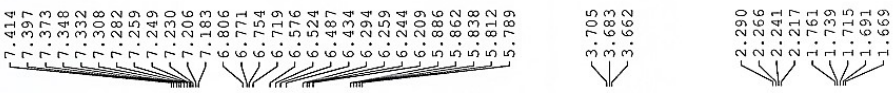

7.698
7.692
7.686
7.680
7.673
7.665
7.653
7.643
7.621
7.616
7.602
7.595
7.583
7.577
7.566
7.560
7.548
3.824
3.804
3.798
3.771
3.682
3.663
3.643
2.099
2.075
2.071
2.056
2.047
2.037
2.029
2.023
2.016
1.997
1.749
1.730
1.725
1.705
1.683
1.661
0.874
0.040
OTBS
O2S
13
10 9 8 7 6 5 4 3 2 1 ppm
2.00
3.03
2.12
2.13
2.19
2.22
9.66
6.20

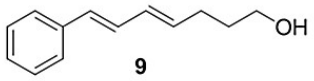

153.39
132.99
131.36
129.62
125.02
61.961
55.846
30.795
25.820
19.087
18.166
-5.459

N=N
N
N
O2S
OTBS

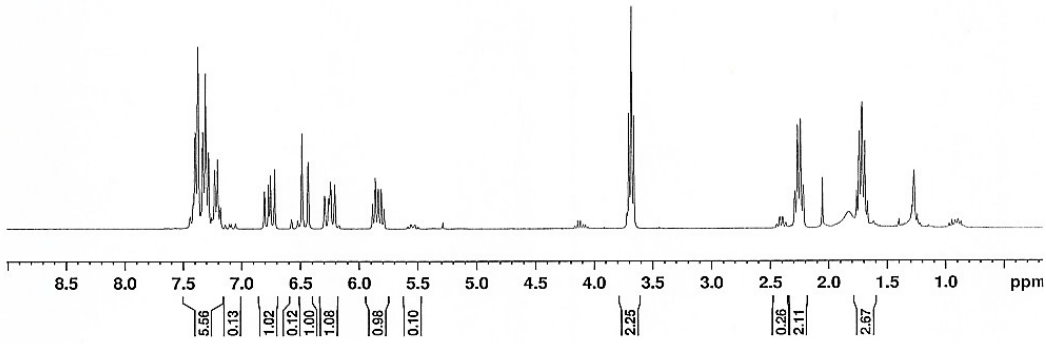
13

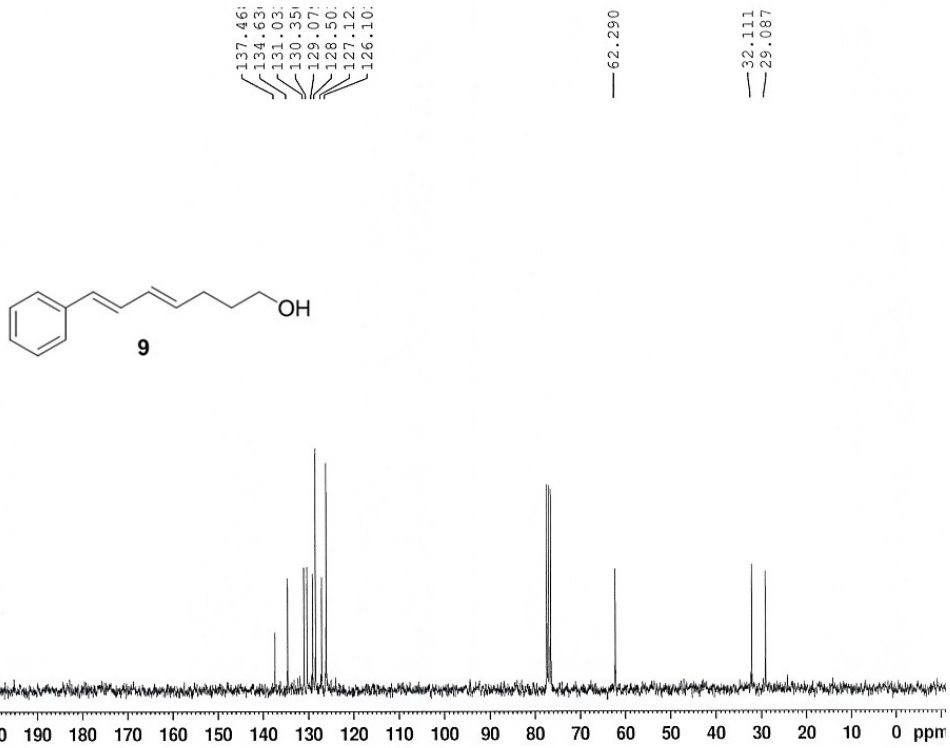

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

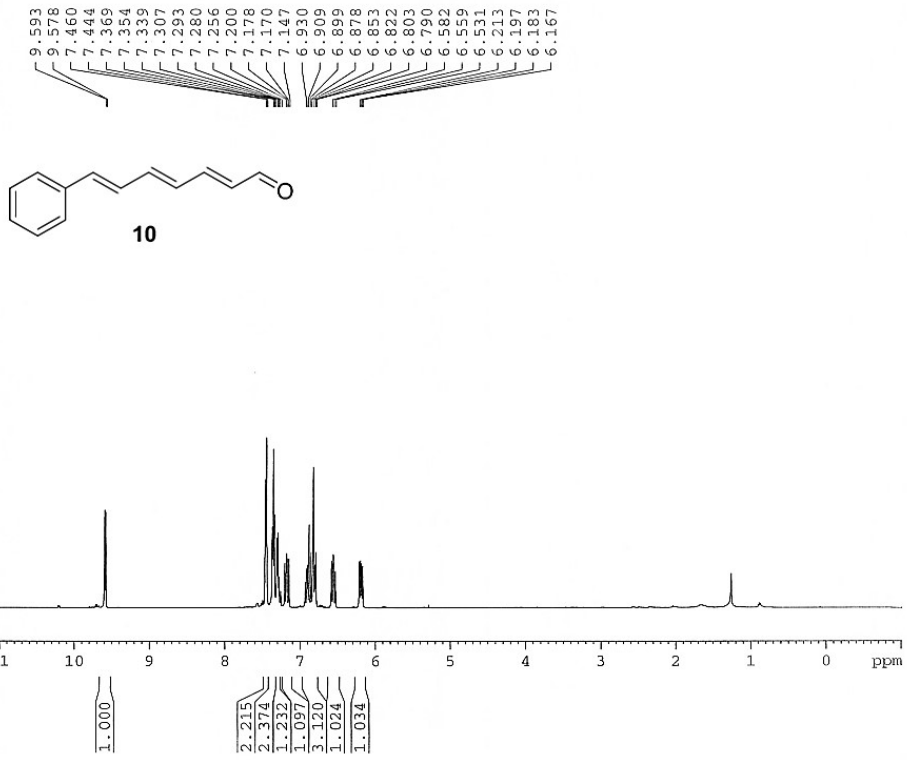

7.414
7.397
7.373
7.348
7.332
7.308
7.282
7.259
7.249
7.230
7.206
7.183
6.806
6.771
6.754
6.719
6.576
6.524
6.487
6.434
6.294
6.259
6.244
6.209
5.886
5.862
5.838
5.812
5.789
3.705
3.683
3.662
2.290
2.266
2.241
2.217
1.761
1.739
1.715
1.691
1.669

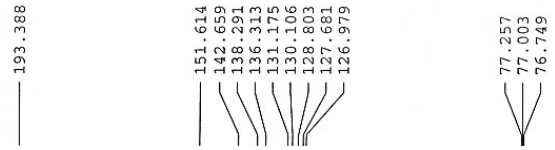

OH

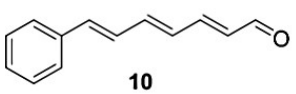
9


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 ppm
5.56
0.13
1.02
0.12
1.00
1.08
0.98
0.10
2.25
0.26
2.11
2.67

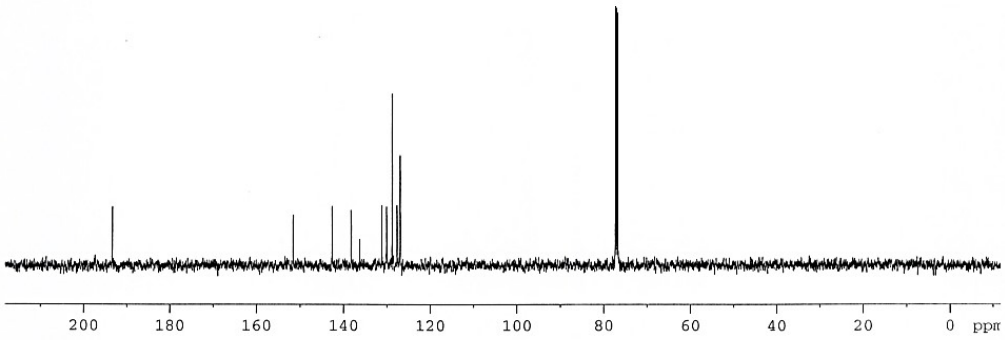

137.46
134.63
131.03
130.35
129.07
128.50
127.12
126.10
62.290
32.111
29.087
OH
9
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

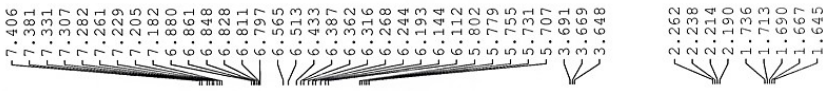

9.593
9.578
7.460
7.444
7.369
7.354
7.339
7.307
7.293
7.280
7.256
7.200
7.178
7.170
7.147
6.930
6.909
6.899
6.878
6.853
6.822
6.803
6.790
6.582
6.559
6.531
6.213
6.197
6.183
6.167
O
10
11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.000
2.215
2.374
1.232
1.097
3.120
1.024
1.034

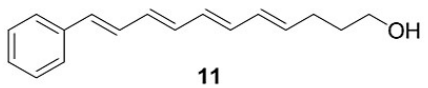

193.388
151.614
142.659
138.291
136.313
131.175
130.106
128.803
127.681
126.979
77.257
77.003
76.749


O

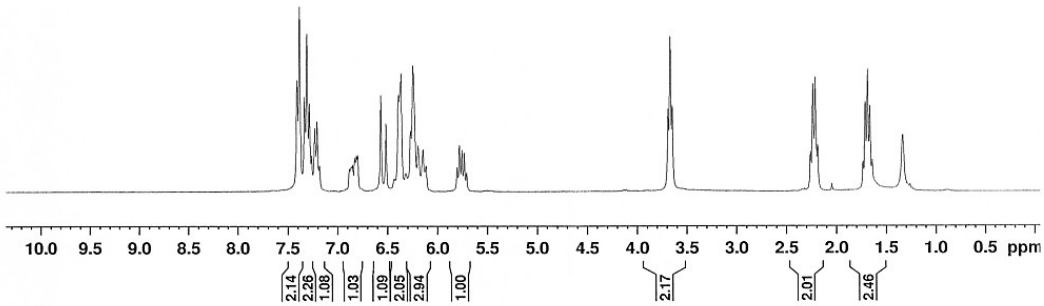
10

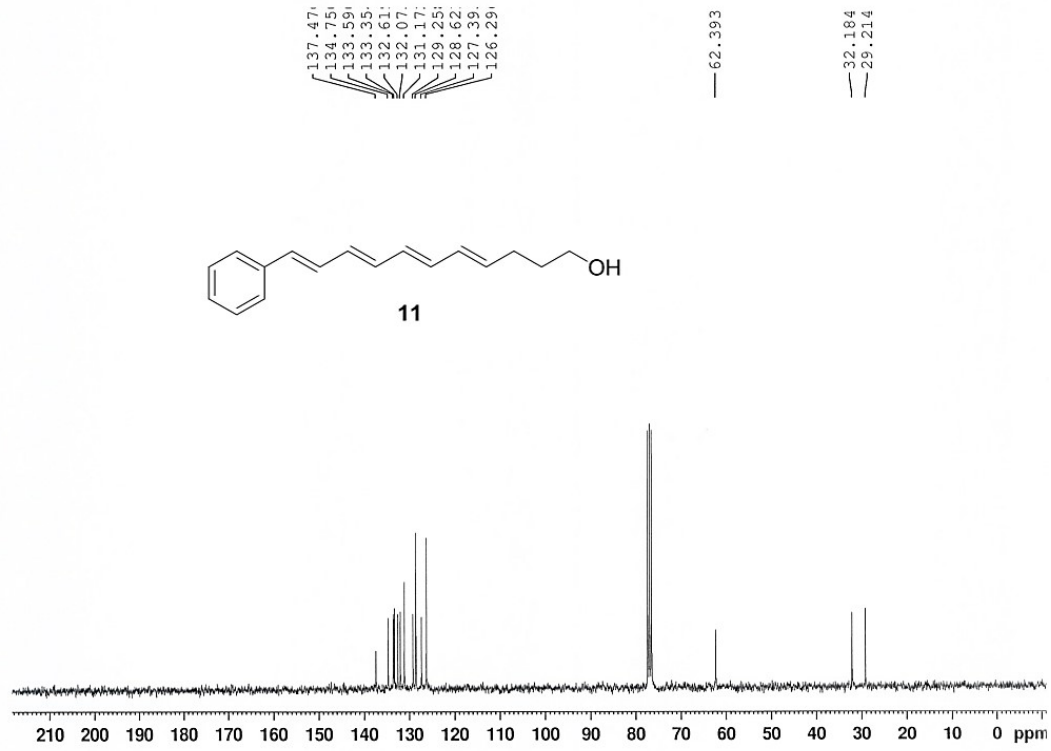

200
180
160
140
120
100
80
60
40
20
0
ppm

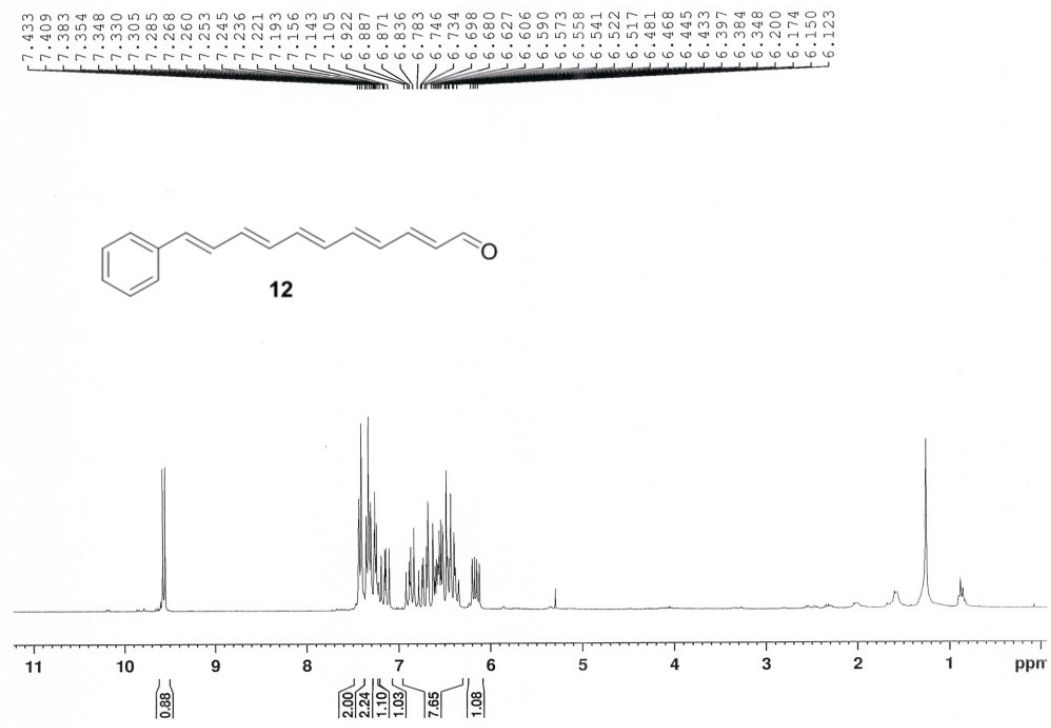

7.406
7.381
7.331
7.307
7.282
7.261
7.229
7.205
7.182
6.880
6.861
6.848
6.828
6.811
6.797
6.565
6.513
6.433
6.387
6.362
6.316
6.268
6.244
6.193
6.144
6.112
5.802
5.779
5.755
5.731
5.707
3.691
3.669
3.648
2.262
2.238
2.214
2.190
1.736
1.713
1.690
1.667
1.645

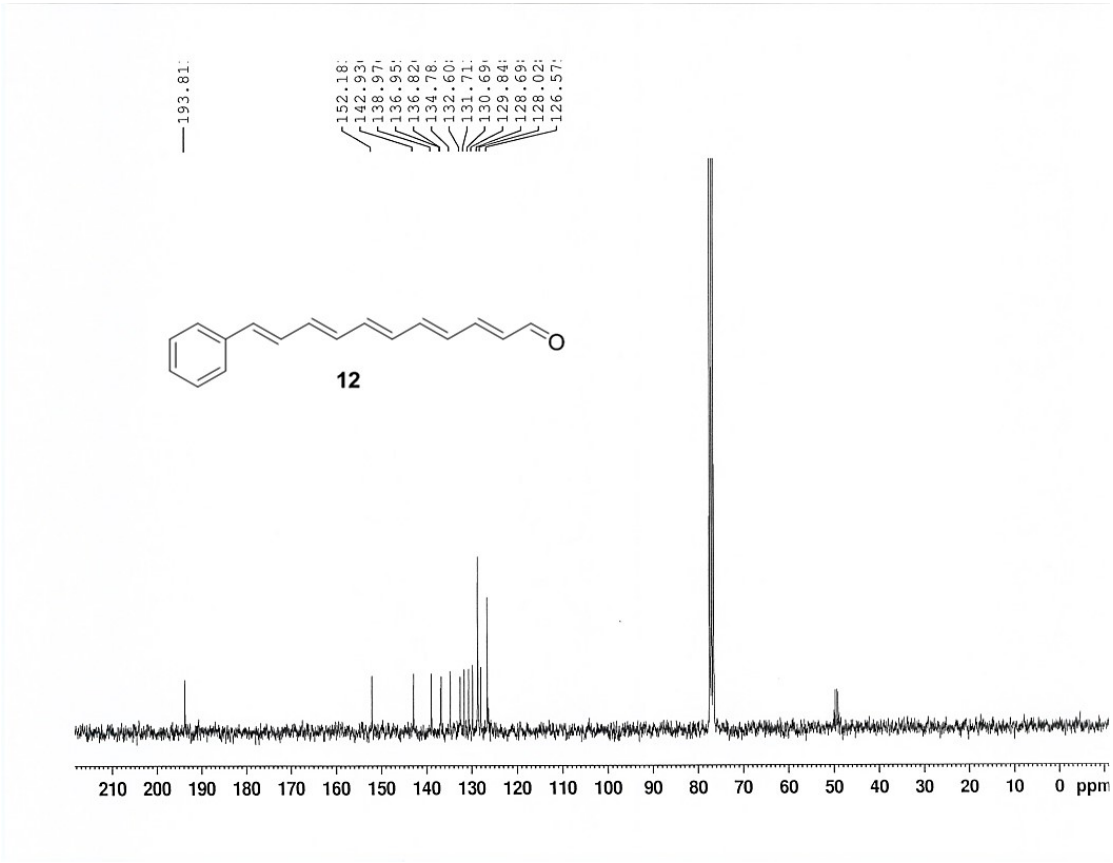

OH

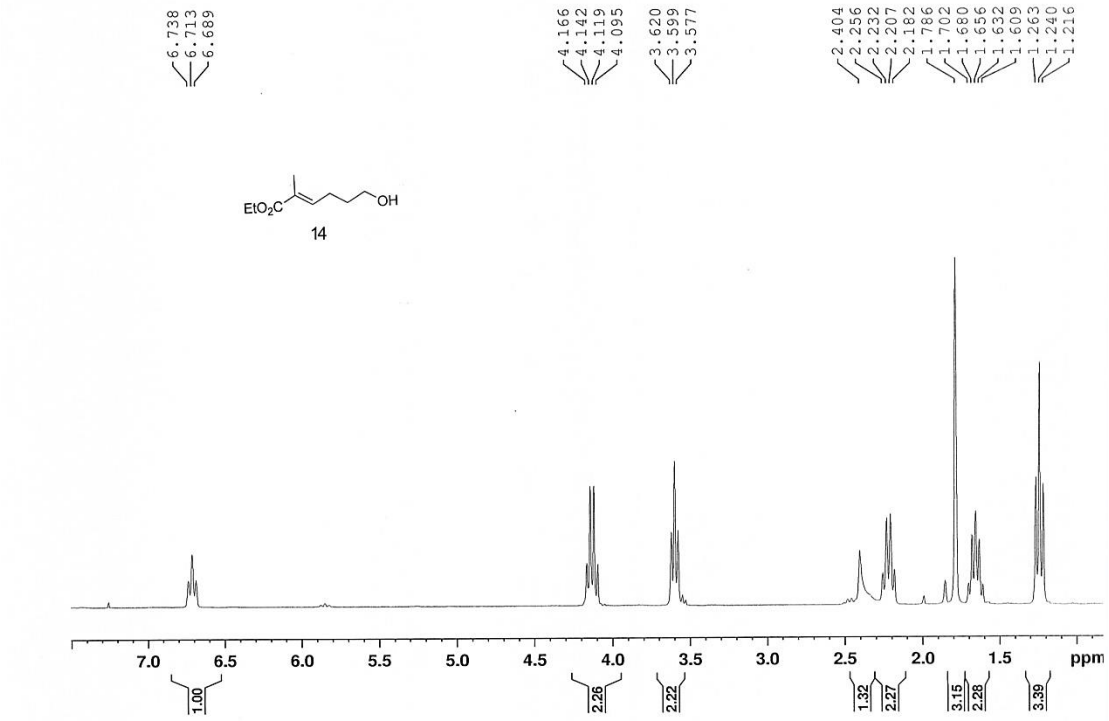
11

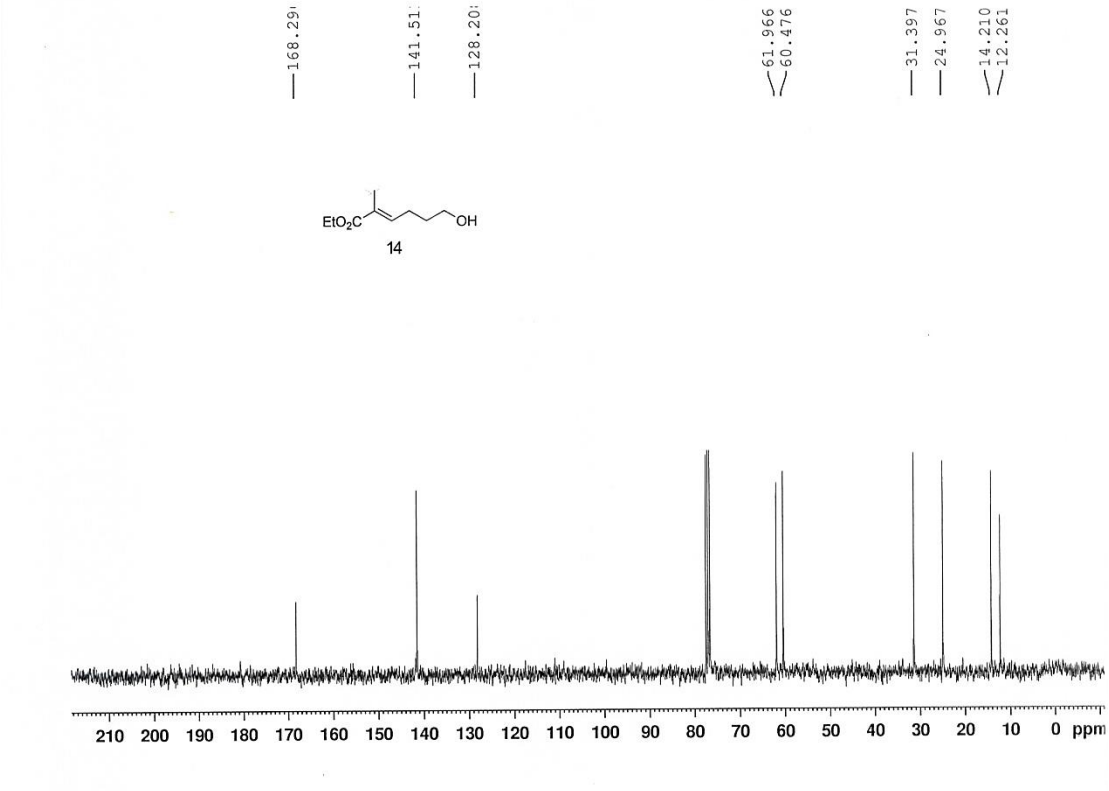

10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
2.14
2.26
1.08
1.03
1.09
2.05
2.94
1.00
2.17
2.01
2.46

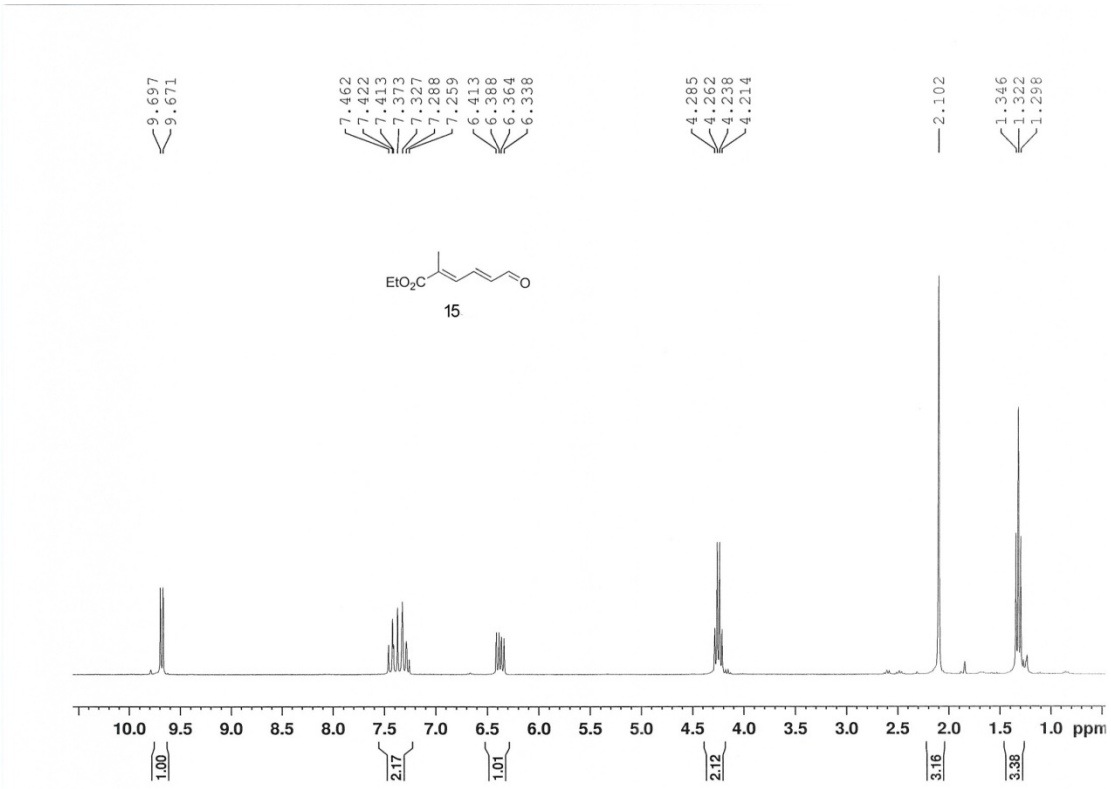

137.47
134.75
133.59
133.35
132.61
132.07
131.17
129.25
128.62
127.39
126.29
62.393
32.184
29.214
OH
11
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

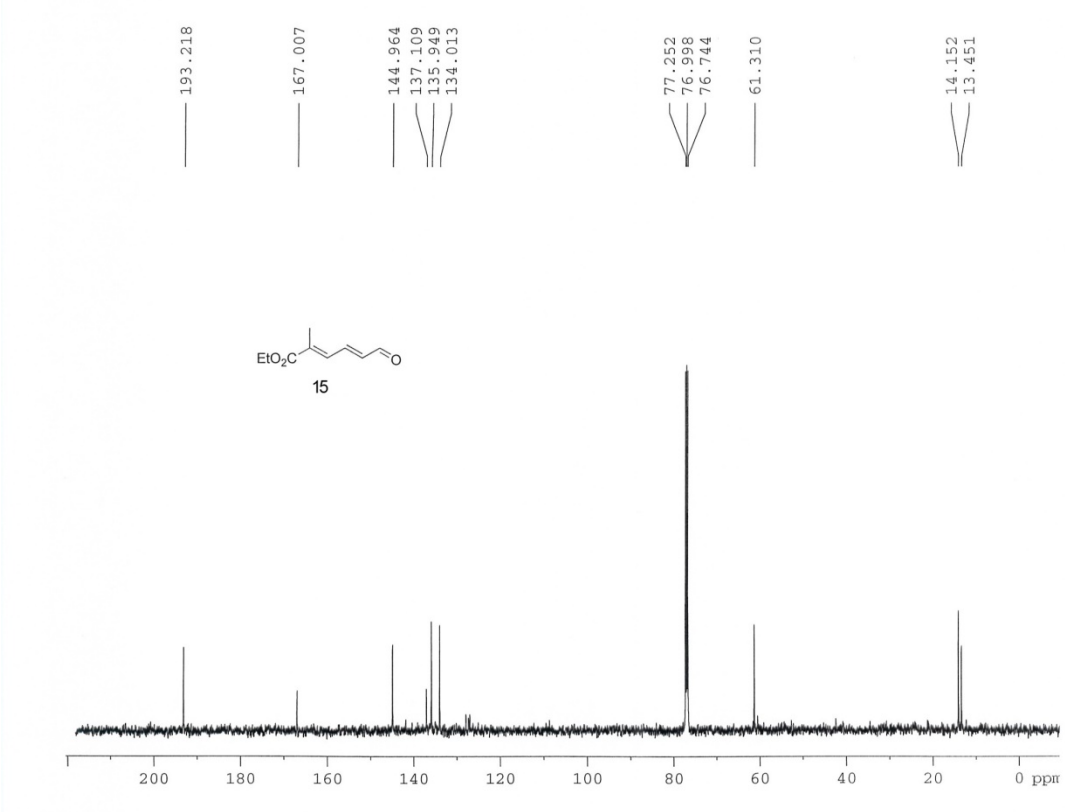

7.433
7.409
7.383
7.354
7.348
7.330
7.305
7.285
7.268
7.260
7.253
7.245
7.236
7.221
7.193
7.156
7.143
7.105
6.922
6.887
6.871
6.836
6.783
6.746
6.734
6.698
6.680
6.627
6.606
6.590
6.573
6.558
6.541
6.522
6.517
6.481
6.468
6.445
6.433
6.397
6.384
6.348
6.200
6.174
6.150
6.123
O
12
11 10 9 8 7 6 5 4 3 2 1 ppm
0.88
2.00
2.24
1.10
1.03
7.65
1.08

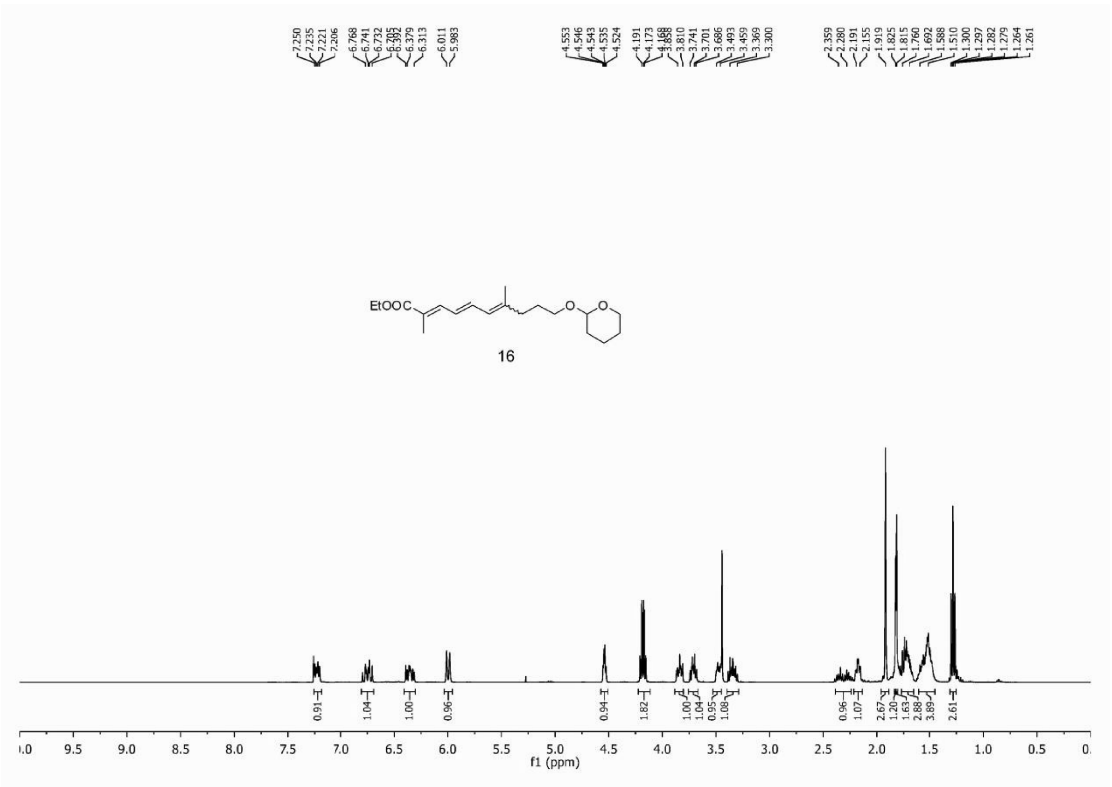

193.81
152.18
142.93
138.97
136.95
136.82
134.78
132.60
131.71
130.69
129.84
128.69
128.02
126.57
O
12
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

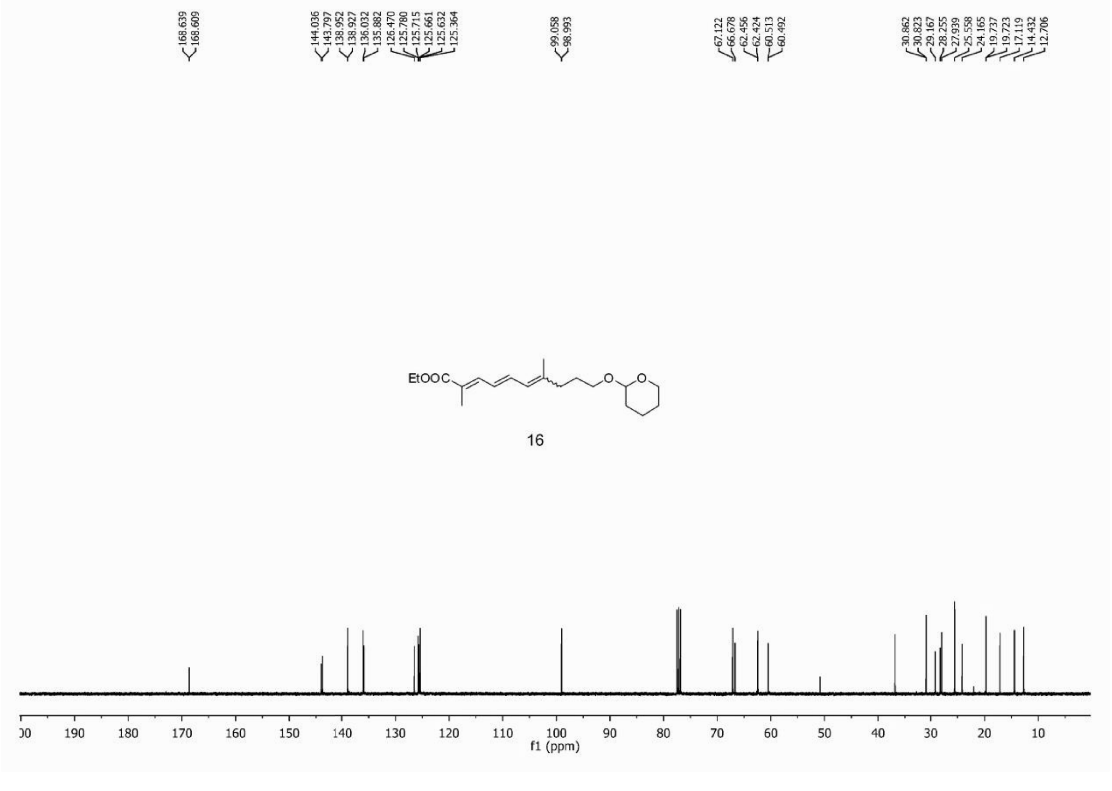

6.738
6.713
6.689
4.166
4.142
4.119
4.095
3.620
3.599
3.577
2.404
2.256
2.232
2.207
2.182
1.786
1.702
1.680
1.656
1.632
1.609
1.263
1.240
1.216
EtO2C
OH
14
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
ppm
1.00
2.26
2.22
1.32
2.27
3.15
2.28
3.39

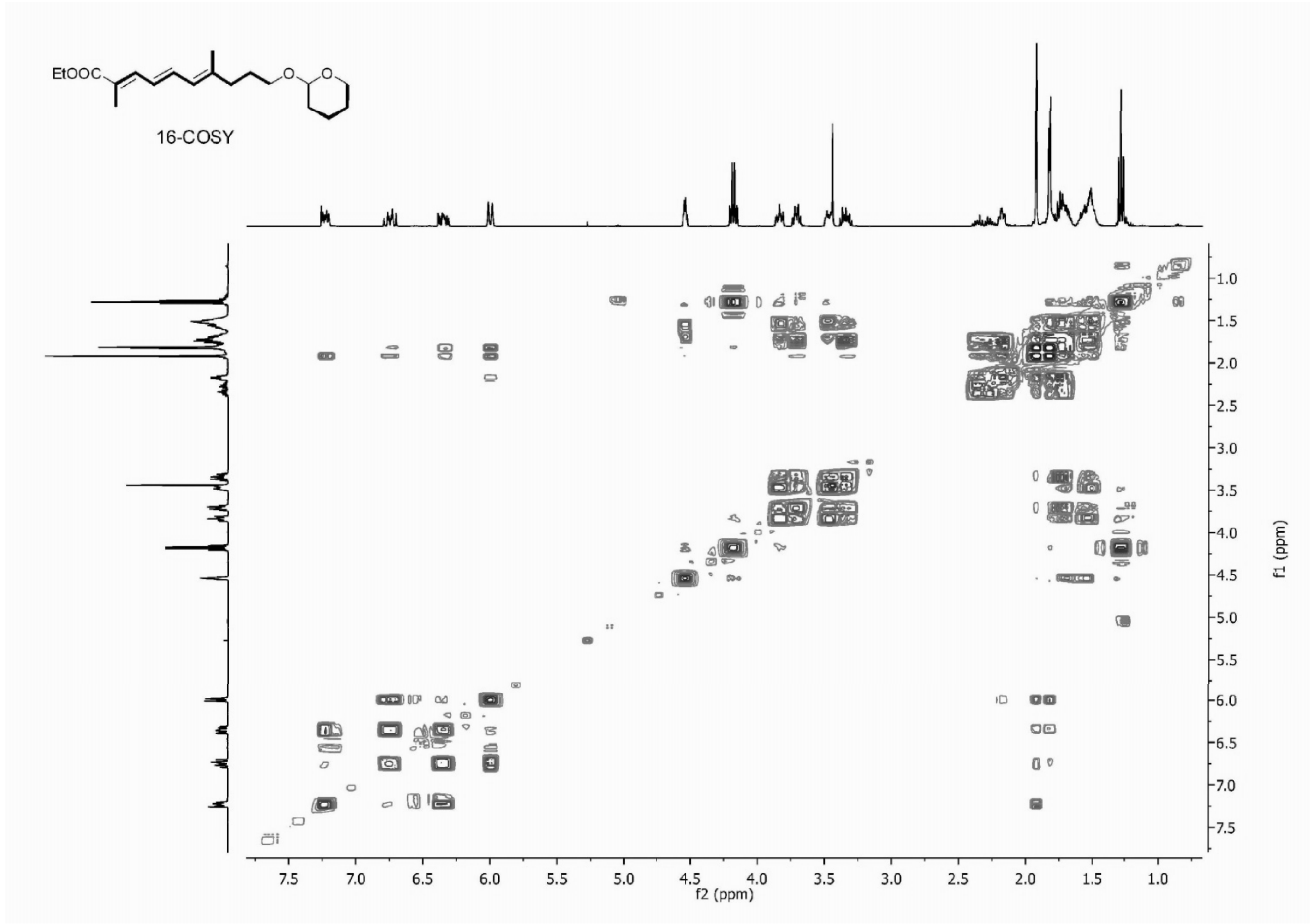

168.29
141.51
128.20
61.966
60.476
31.397
24.967
14.210
12.261
EtO2C
OH
14
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

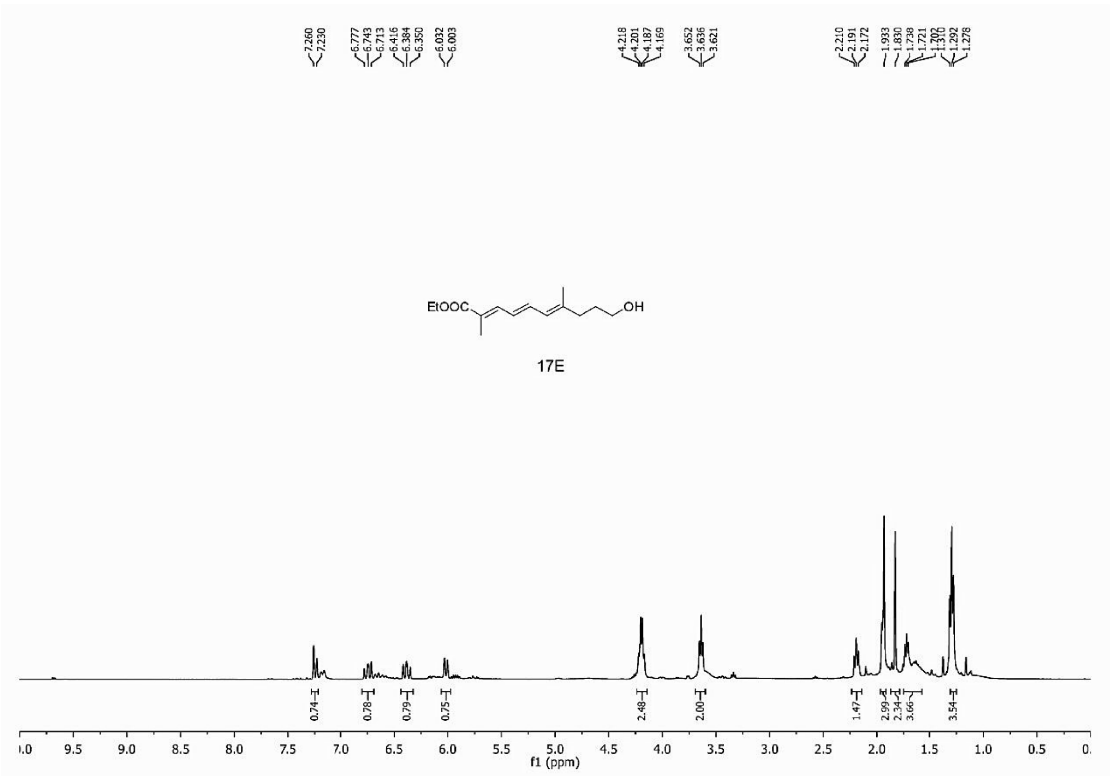

9.697
9.671
7.462
7.422
7.413
7.373
7.327
7.288
7.259
6.413
6.388
6.364
6.338
4.285
4.262
4.238
4.214
2.102
1.346
1.322
1.298
EtO2C
O
15
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
1.00
2.17
1.01
2.12
3.16
3.38

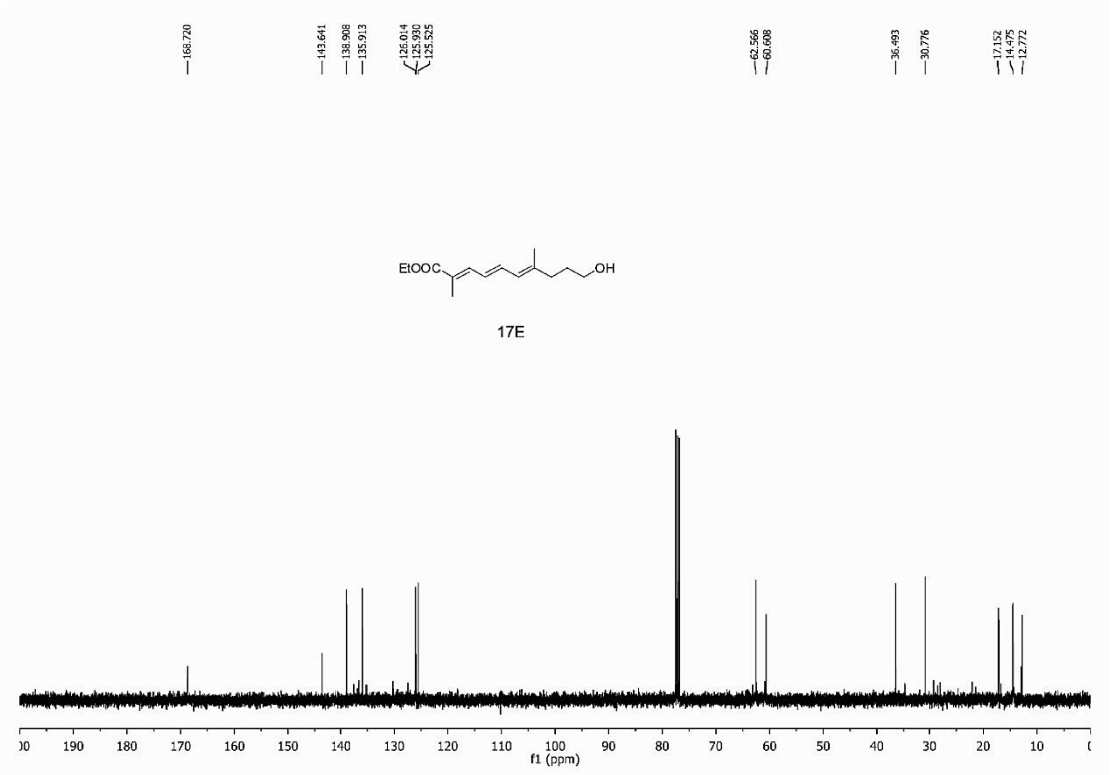

193.218
167.007
144.964
137.109
135.949
134.013
77.252
76.998
76.744
61.310
14.152
13.451
EtO2C
O
15
200 180 160 140 120 100 80 60 40 20 0 ppm

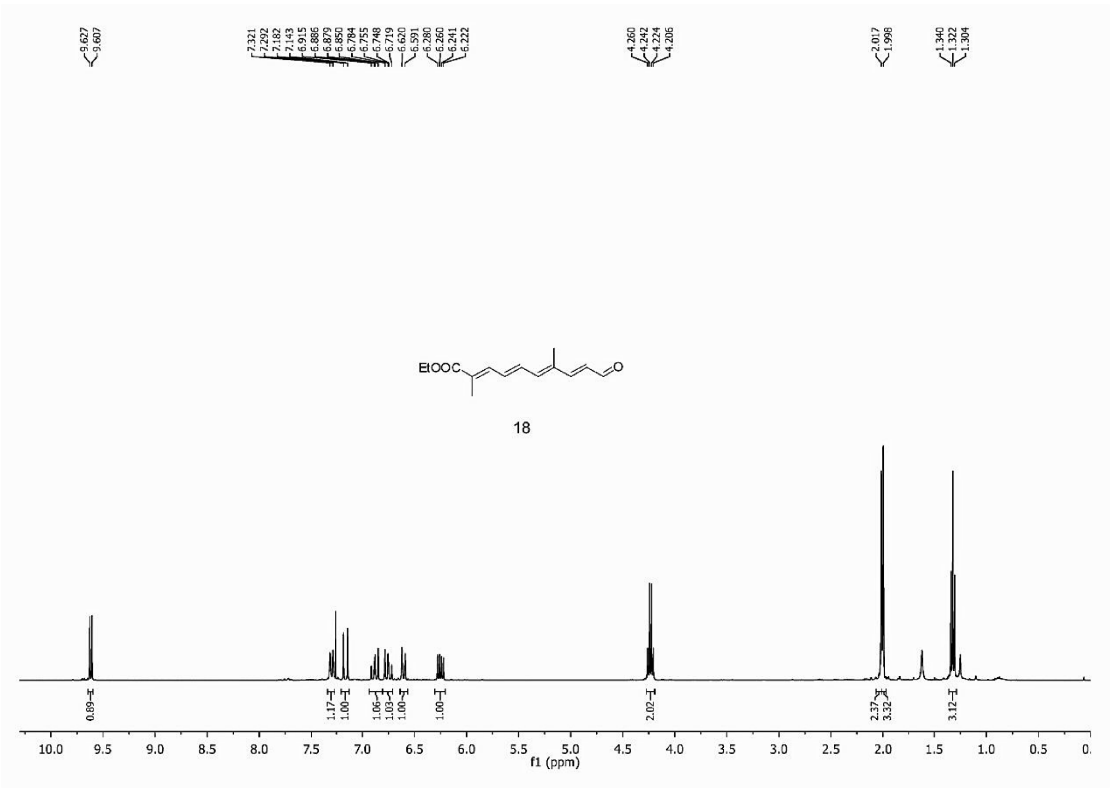

7.290
7.235
7.221
7.206
6.768
6.741
6.732
6.705
6.392
6.379
6.313
6.011
5.983
4.553
4.546
4.543
4.535
4.524
4.191
4.173
4.168
4.155
3.810
3.741
3.701
3.686
3.493
3.459
3.369
3.300
2.359
2.280
2.191
2.155
1.919
1.825
1.815
1.760
1.692
1.588
1.510
1.300
1.282
1.279
1.264
1.261
EtOOC
O
O
16
0.91
1.04
1.00
0.96
0.94
1.82
1.00
1.04
0.95
1.08
0.96
1.07
2.67
1.20
1.63
2.88
3.89
2.61
f1 (ppm)

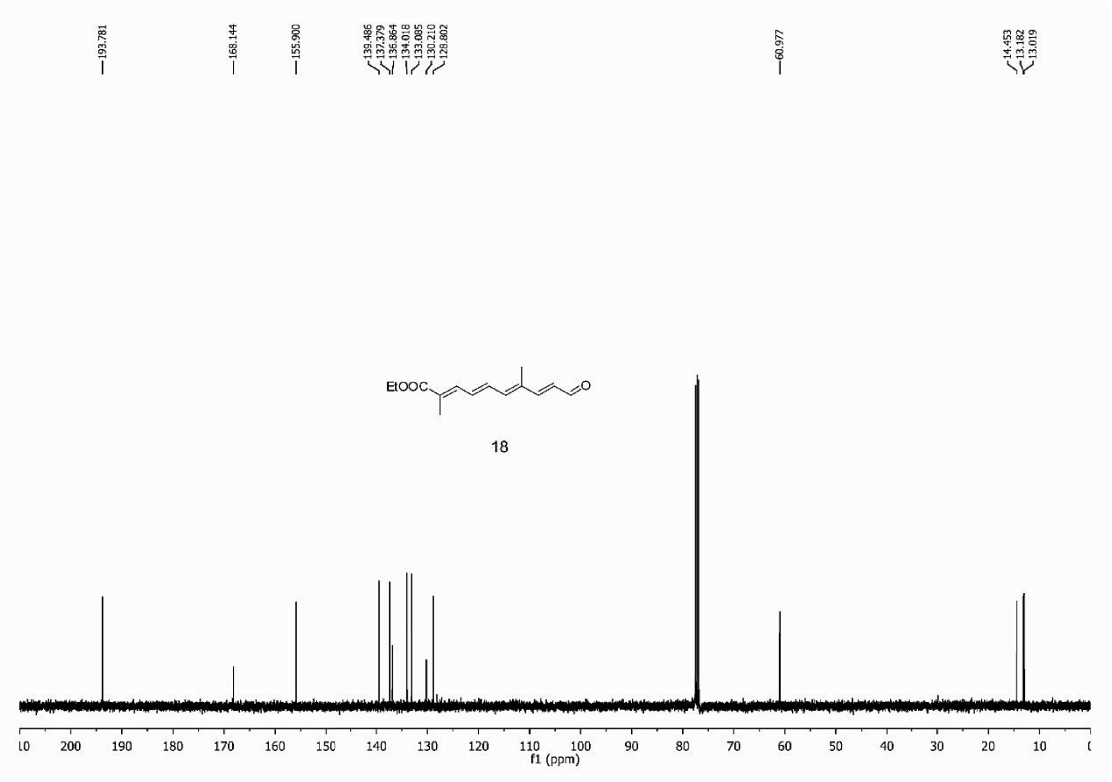

168.639
168.089
144.036
143.797
138.952
138.927
136.032
135.588
126.470
125.780
125.715
125.661
125.632
125.364
99.058
98.993
67.122
66.678
62.456
62.434
60.513
60.492
30.862
30.823
29.167
28.255
27.939
25.558
24.165
19.777
19.723
17.119
14.432
12.706
EtOOC
O
O
16
f1 (ppm)

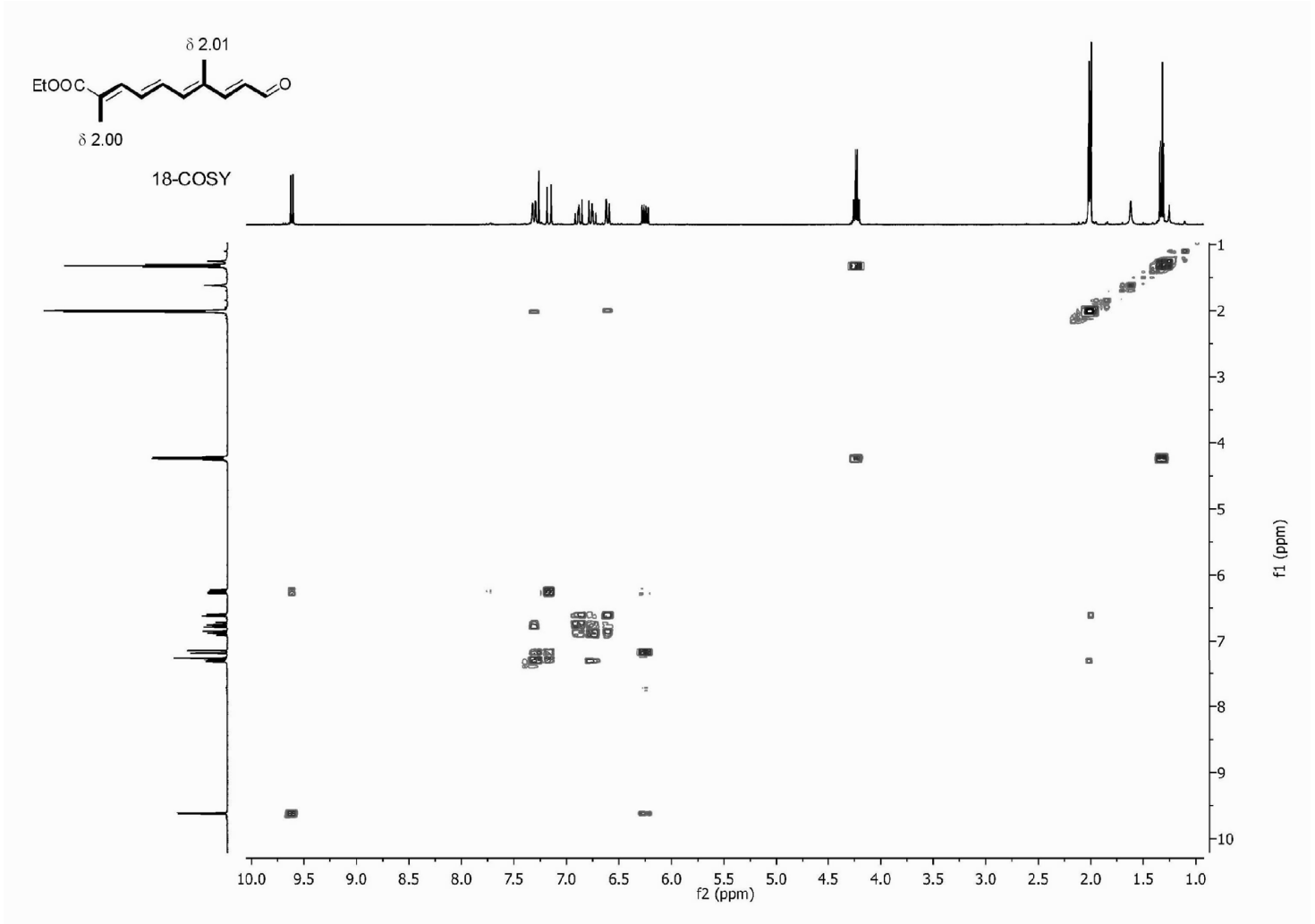

EtOOC
O
O
16-COSY
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
f2 (ppm)
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
7.0
7.5
f1 (ppm)

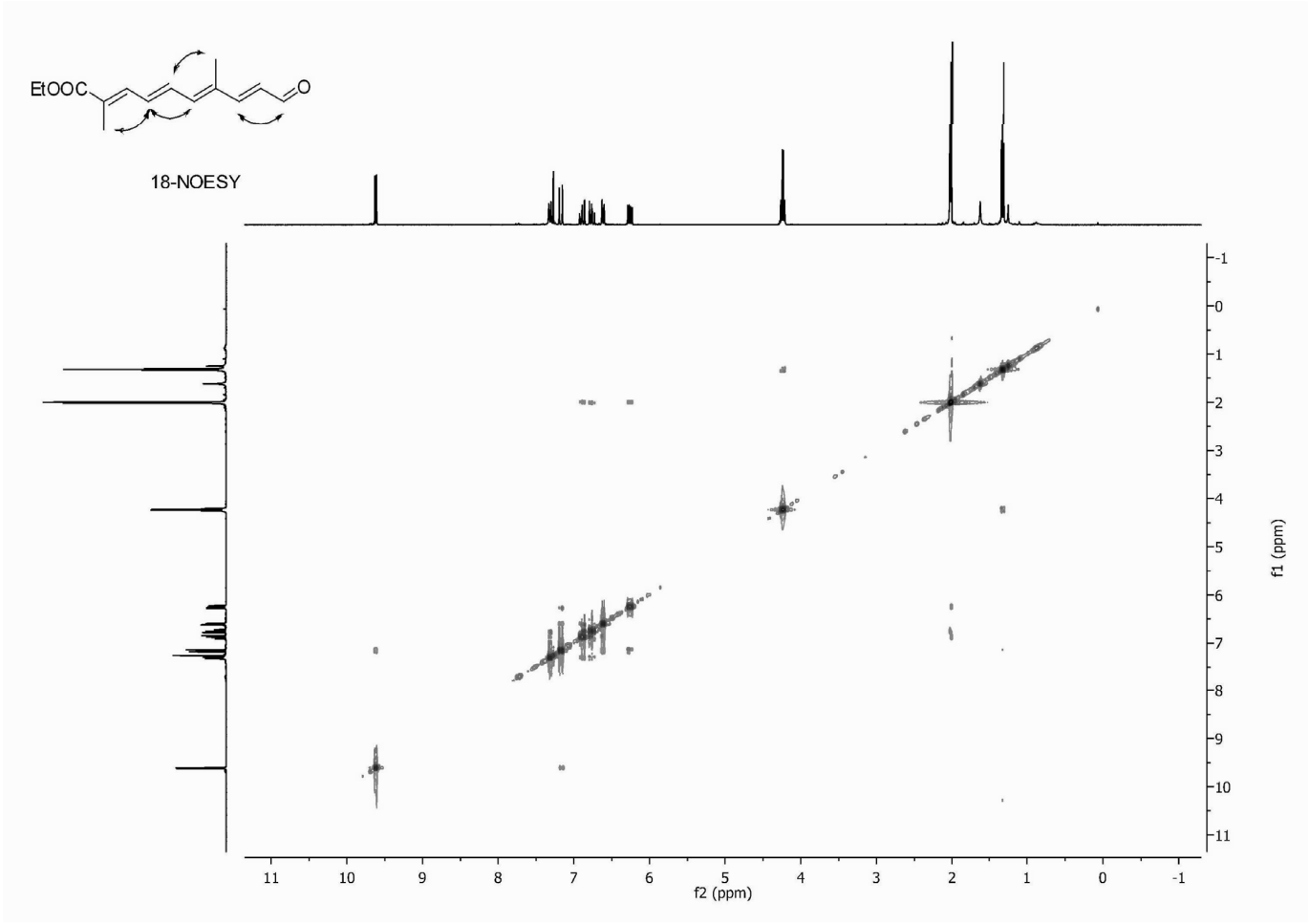

7.260
7.230
6.777
6.743
6.713
6.416
6.384
6.350
6.032
6.003
4.218
4.201
4.187
4.169
3.652
3.636
3.621
2.210
2.191
2.172
1.933
1.830
1.728
1.721
1.701
1.310
1.292
1.278
EtOOC
OH
17E
0.74
0.78
0.79
0.75
2.48
2.00
1.47
2.99
2.34
3.66
3.54
f1 (ppm)


168.720
143.641
138.908
135.913
126.014
125.930
125.525
62.566
60.608
36.493
30.776
17.152
14.475
12.772
EtOOC
OH
17E
f1 (ppm)


9.627
9.607
7.321
7.292
7.182
7.143
6.915
6.886
6.879
6.850
6.784
6.755
6.748
6.719
6.620
6.591
6.280
6.260
6.241
6.222
4.260
4.242
4.224
4.206
2.017
1.998
1.340
1.322
1.304
EtOOC
O
18
0.89
1.17
1.00
1.06
1.03
1.00
1.00
2.02
2.37
3.32
3.12
f1 (ppm)


193.781
168.144
155.900
139.486
137.379
135.864
134.418
133.695
130.210
128.802
60.977
14.453
13.182
13.019
EtOOC
O
18
f1 (ppm)


δ 2.01
EtOOC
O
δ 2.00
18-COSY
f2 (ppm)
f1 (ppm)


EtOOC
O
18-NOESY
f2 (ppm)
f1 (ppm)