Regiospecific formal [3+2] Annulation of *tert*-Propargyl alcohols with acyclic 1,3-diketones via the Cycloisomerization of homoallenyl ketones

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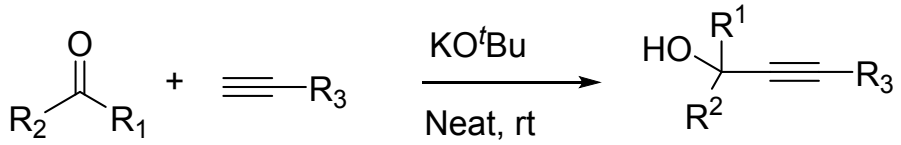
I. General Information: Unless otherwise noted, all reagents were used as received from commercial suppliers. Reactions were performed in flame-dried or oven-dried glassware with magnetic stirring. Reactions were monitored using thin-layer chromatography (TLC) with aluminium sheets silica gel 60 F_{254} from Merck.TLC plates were visualized with UV light (254 nm), iodine treatment or using *p*-anisaldehyde or $KMnO_4$ stain. Column chromatography was carried out using silica gel 60−120 mesh as the stationary phase. NMR spectra were recorded at 500 MHz and 400 MHz (^{1}H) and at 125 MHz and 100 MHz (^{13}C), respectively on Avance Bruker spectrometer. Chemical shifts (δ) are reported in ppm, using the residual solvent peak in $CDCl_3$ (^{1}H: δ= 7.26 and ^{13}C: δ = 77.16 ppm) as an internal standard, and coupling constants (*J*) are given in Hz. HRMS were recorded using ESI-TOF techniques. Melting points were measured with LABINDIA mepa melting apparatus.

II. General experimental procedures

II.1. General experimental procedure for the synthesis of tert-propargylic alcohol (1): A mixture of terminal alkyne (1.2 equiv), *t*-BuOK (1.5 equiv) and ketone (1 equiv) were placed into a reaction flask at room temperature under nitrogen atmosphere and the mixture was stirred for 2−6 h. After completion of the reaction (monitored by TLC), the resulting mixture was quenched with water and extracted into ethyl acetate (thrice). Combined organic layers were washed with brine solution and dried over anhydrous Na_2SO_4 and the solvent was evaporated to obtain the pure compounds in good yield.



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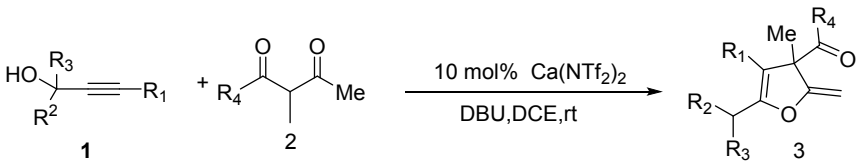
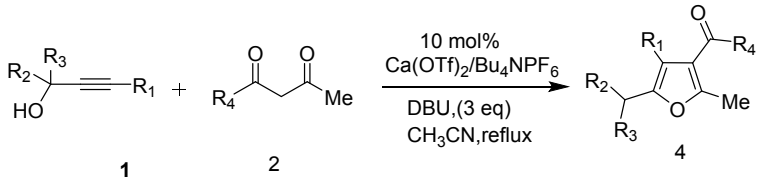
II.2. General experimental procedure for the synthesis of dihydrofuran 3: A mixture of aryl propargylic alcohol **1** (1 equiv) and respective 3-methylpentane-2,4-dione **2** (1.5 equiv) were stirred at rt in presence of $Ca(NTf_2)_2$ (10 mol%) for appropriate time till the formation of the allene (monitored by TLC). Then 3 equiv. of DBU was added to the reaction mixture and stirred till the completion of the reaction (monitored by TLC), the crude product was purified by using silica gel column chromatography (2-3 % EtOAc in pet ether) to obtain the desired product **3** in good yield.

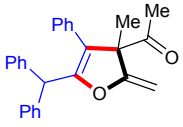


II.3. General experimental procedure for the synthesis of tetra substituted furan 4: A mixture of aryl propargylic alcohol **1** (1 equiv) and respective acetyl acetone **2** (1.5 equiv) were stirred at 90 ^oC in presence of $Ca(OTf)_2/Bu_4NPF_6$ (10/5 mol%) for appropriate time till the formation of the allene intermediate/consumption of **1** (monitored by TLC). Then 3 equiv. of DBU was added to the reaction mixture and stirred the tiil completion of the reaction (monitored by TLC), the crude product was purified by using silica gel column chromatography (3−5 % EtOAc in pet ether) to obtain the desired product **4** in moderate to good yield.

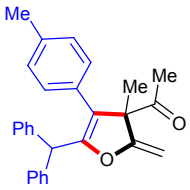


III. Spectral Data

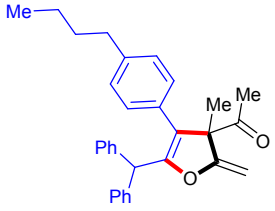
1-(5-benzhydryl-3-methyl-2-methylene-4-phenyl-2,3-dihydrofuran-3-yl)ethanone) (**3a**): Yield: 107 mg (80) as white solid; mp 137-138 ^oC; ^1H NMR ($CDCl_3$, 400 MHz): δ 7.37-7.32 (m, 5H), 7.30-7.24 (m, 5H), 7.21 (t, J = 1.6 Hz, 3H), 7.04-7.01 (m, 2H), 5.29 (s, 1H), 4.63 (d, J = 2.8 Hz, 1H), 4.14 (d, J = 2.8 Hz, 1H), 2.13 (s, 3H), 1.38 (s, 3H) ppm; ^{13}C NMR ($CDCl_3$, 125 MHz): δ 205.3, 164.7, 154.2, 140.3, 140.1, 131.8, 130.1, 128.9, 128.8, 128.7, 128.6, 128.4, 128.3, 127.6, 127.1, 126.9, 118.5, 85.5, 64.5, 48.4, 25.2, 21.4 ppm; HRMS (ESI-TOF): m/z calcd for $C_{27}H_{24}O_2$ [M+H]+ : 381.1856, found: 381.1855.



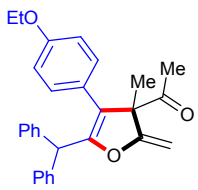
1-(5-benzhydryl-3-methyl-2-methylene-4-(p-tolyl)-2,3-dihydrofuran-3-yl)ethanone (**3b**): Yield: 104.5 mg (79%) as yellow liquid ; ^1H NMR ($CDCl_3$, 500 MHz): δ 7.36-7.22 (m, 9H), 7.13 (d, J = 8.5 Hz, 2H), 6.94 (d, J = 7.5 Hz, 3H), 5.31 (s, 1H), 4.64 (d, J = 2.5 Hz, 1H), 4.15 (d, J = 2.5 Hz, 1H), 2.35 (s, 3H), 2.15 (s, 3H), 1.40 (s, 3H) ppm; ^{13}C NMR ($CDCl_3$, 125 MHz) : δ 205.3, 164.8, 153.8, 140.4, 140.2, 137.4, 129.5, 128.9, 128.6, 128.6, 128.3, 128.2, 126.9, 118.4, 64.6, 48.4, 25.2, 21.4, 21.1 ppm; HRMS (ESI-TOF): m/z calcd for $C_{28}H_{26}O_2$ [M+H]+ : 395.2013, found: 395.2014.



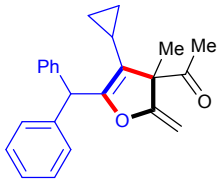
1-(5-benzhydryl-4-(4-butylphenyl)-3-methyl-2-methylene-2,3-dihydrofuran-3-yl)ethanone (**3c**): Yield: 100 mg (78%) as yellow liquid; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.41-7.30 (m, 5H), 7.28-7.23 (m, 5H), 7.14 (d, *J* = 8 Hz, 2H), 6.96 (d, *J* = 8 Hz, 2H), 5.33 (s, 1H), 4.64 (d, *J* = 2.4 Hz, 1H), 4.15 (d, *J* = 2.8 Hz, 1H), 2.61 (t, *J* = 7.6 Hz, 2H), 2.16 (s, 3H), 1.65-1.58 (m, 2H), 1.41 (s, 3H), 1.39-31 (m, 2H), 0.96 (t, *J* = 7.2 Hz, 3H) ppm; ^{13}C NMR ($CDCl_3$, 100 MHz) : δ 205.6, 164.9, 153.8, 142.5, 140.5, 140.4, 129.1, 128.9, 128.8, 128.7, 128.5, 127.1, 127.0, 118.6, 85.4, 64.7, 48.5, 35.4, 33.5, 25.4, 22.5, 21.6, 14.1 ppm; HRMS (ESI-TOF): m/z calcd for $C_{31}H_{32}O_2$ [M+H]+ : 437.2562, found: 437.2561.



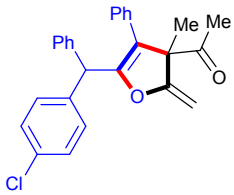
1-(5-benzhydryl-4-(4-ethoxyphenyl)-3-methyl-2-methylene-2,3-dihydrofuran-3-yl)ethanone (**3d**) Yield: 100.8 mg (79%) as yellow liquid; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.39 (q, *J* = 7.5 Hz, 2H), 7.35-7.31 (m, 5H), 7.30 (d, *J* = 1 Hz, 1H), 7.28-7.22 (m, 2H), 6.96 (q, *J* = 6.5 Hz, 2H), 6.84 (q, *J* = 7 Hz, 2H), 5.28 (s, 1H), 4.63 (d, *J* = 2.5 Hz, 1H), 4.14 (d, *J* = 2.5 Hz, 1H), 4.04 (q, *J* = 14 Hz, 2H), 2.14 (s, 3H), 1.43 (t, *J* = 7 Hz, 3H), 1.39 (s, 3H) ppm; ^{13}C NMR ($CDCl_3$, 100 MHz): δ 205.5, 165.1, 158.6, 153.6, 140.5, 140.4, 129.8, 129.1, 128.8, 128.7, 128.5, 127.1, 123.9, 118.3, 114.9, 85.3, 64.8, 63.5, 48.5, 25.3, 21.6, 14.9 ppm; HRMS (ESI-TOF): m/z calcd for $C_{29}H_{28}O_3$ [M+H]+ : 425.2118, found: 425.2119.



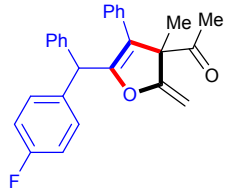
1-(5-benzhydryl-4-cyclopropyl-3-methyl-2-methylene-2,3-dihydrofuran-3-yl)ethanone (**3e**): Yield: 108 mg (78%) as yellow liquid; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.32 (d, *J* = 1.6 Hz, 1H), 7.31 (d, *J* = 2.8 Hz, 1H), 7.29-7.26 (m, 3H), 7.24-7.21 (m, 4H), 7.18 (d, *J* = 8.4 Hz, 1H), 5.41 (d, *J* = 2.8 Hz, 1H), 4.51 (d, *J* = 2.8 Hz, 1H), 4.01 (d, *J* = 2.8 Hz, 1H), 1.98 (s, 3H), 1.40 (s, 3H), 1.21-1.15 (m, 1H), 0.77-0.61 (m, 2H), 048-0.29 (m, 2H) ppm; ^{13}C NMR ($CDCl_3$, 100 MHz) : δ 205.5, 205.5, 164.8, 164.8, 153.8, 153.7, 139.8, 139.7, 138.8, 138.7, 132.8, 132.7, 130.2, 130.1, 128.7, 128.7, 128.6, 128.6, 128.5, 127.1, 127.1, 116.9, 116.9, 84.9, 64.5, 64.5, 47.1, 47.1, 25.1, 25.1, 21.8, 21.7, 5.2, 5.2, 5.1, 3.9, 3.9 ppm; HRMS (ESI-TOF): m/z calcd for $C_{24}H_{24}O_2$ [M+H]+ : 344.1776, found: 344.1775.



1-(5-((4-chlorophenyl)(phenyl)methyl)-3-methyl-2-methylene-4-phenyl-2,3-dihydrofuran-3-yl)ethanone (**3f**): Yield: 102.8 mg (79%) as yellow liquid; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.40 (d, *J* = 7 Hz, 1H), 7.37-7.32 (m, 6H), 7.30-7.28 (m, 3H), 7.21 (d, *J* = 7 Hz, 1H), 7.17 (d, *J* = 8.5 Hz, 1H), 7.03 (t, *J* = 6.5 Hz, 2H), 5.28 (d, *J* = 11.5 Hz, 1H), 4.67 (d, *J* = 3 Hz, 1H), 4.18 (d, *J* = 2.5 Hz, 1H), 2.16 (s, 3H), 1.42 (s, 3H) ppm; ^{13}C NMR ($CDCl_3$, 125 MHz) : δ 205.1, 164.6, 164.5, 153.6, 153.5, 139.8, 139.7, 138.8, 138.7, 132.9, 132.9, 131.6, 131.6, 130.3, 130.1, 128.8, 128.8, 128.8, 128.8, 128.6, 128.5, 128.5, 128.4, 128.3, 127.7, 127.7, 127.2, 118.8, 85.7, 64.6, 64.5, 47.8, 25.2, 25.2, 21.5, 21.4 ppm; HRMS (ESI-TOF): m/z calcd for $C_{27}H_{23}ClO_2$ [M+H]+ : 414.1387, found: 414.1388.

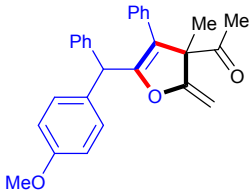


1-(5-((4-fluorophenyl)(phenyl)methyl)-3-methyl-2-methylene-4-phenyl-2,3-dihydrofuran-3-yl)ethanone (**3g**) :Yield: 102.8 mg (78%) as white solid; mp 108-109 ^{o}C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.41-7.28 (m, 8H), 7.28-7.18 (m, 2H), 7.09-6.99 (m, 4H), 5.29 (d, *J* = 11 Hz, 1H), 4.67 (d, *J* = 3 Hz, 1H), 4.18 (d, *J* = 5 Hz, 1H), 2.16 (s, 3H), 1.41 (s, 3H) ppm; ^{13}C NMR ($CDCl_3$, 125 MHz) : δ 205.1, 164.6, 164.5, 162.8, 162.8, 160.8,

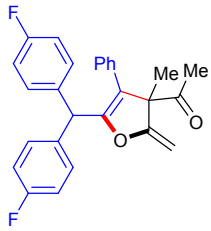


153.9, 153.8, 140.1, 140.1, 136.1, 135.8, 131.7, 131.6, 130.5, 130.4, 130.2, 130.2, 128.4, 128.3, 127.7, 127.6, 127.1, 127.1, 115.6, 115.4, 115.3, 115.2, 85.6, 64.6, 64.5, 47.7, 25.2, 21.5, 21.4 ppm; HRMS (ESI-TOF): m/z calcd for $C_{27}H_{23}FO_2$ [M+H]+ : 399.1762, found: 399.1766.

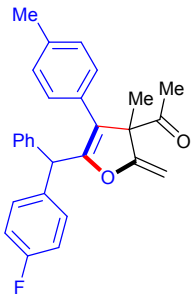
1-(5-((4-methoxyphenyl)(phenyl)methyl)-3-methyl-2-methylene-4-phenyl-2,3-dihydrofuran-3-yl)ethanone (**3h**): Yield: 104.5 mg (80%) as white solid; mp 140-141 ^{o}C; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.32-7.29 (m, 4H), 7.28-7.25 (m, 4H), 7.22 (t, *J* = 1.6 Hz, 2H), 7.06-7.04 (m, 2H), 6.94-6.92 (m, 2H), 5.27 (s, 1H), 4.65 (d, *J* = 2.8 Hz, 1H), 4.15 (d, *J* = 2.8 Hz, 1H), 3.85 (s, 3H), 2.16 (s, 3H), 1.40 (s, 3H) ppm; ^{13}C NMR ($CDCl_3$, 100 MHz) : δ 205.6, 164.7, 158.6, 154.5, 140.7, 132.2, 131.9, 129.8, 128.9, 128.5, 128.4, 127.6, 127.1, 118.3, 114.1, 85.6, 64.6, 55.4, 47.7, 25.4, 21.5 ppm; HRMS (ESI-TOF): m/z calcd for $C_{28}H_{26}O_3$ [M+H]+ : 410.1882; found: 410.1881.



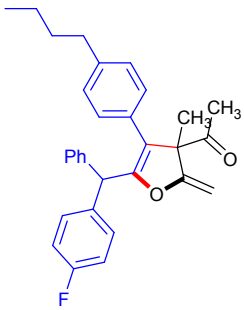
1-(5-(bis(4-fluorophenyl)methyl)-3-methyl-2-methylene-4-phenyl-2,3-dihydrofuran-3-yl)ethanone (**3i**): Yield: 102.7 mg (79%) as yellow liquid; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.32 (t, *J* = 7.5 Hz, 2H), 7.29 (t, *J* = 2 Hz, 3H), 7.19 (q, *J* = 5.5 Hz, 2H),7.08 (t, *J* = 8.5 Hz, 2H),7.0 2 (q, *J* = 8.5 Hz, 4H), 5.25 (s, 1H), 4.67 (d, *J* = 2.5 Hz, 1H), 4.19 (d, *J* = 2.5 Hz, 1H), 2.15 (s, 3H), 1.41 (s, 3H) ppm; ^{13}C NMR ($CDCl_3$, 100 MHz) : δ 205.1, 164.6, 163.1, 162.9, 161.1, 161.1, 153.7, 136.1, 135.9, 135.8, 135.8, 131.6, 130.5, 130.4, 130.2, 130.2, 129.1, 128.4, 127.9, 118.9, 115.8, 115.6, 115.6, 115.4, 85.9, 64.7, 47.1, 25.3, 21.6 ppm; HRMS (ESI-TOF): m/z calcd for $C_{27}H_{22}F_2O_2$ [M+H]+ : 416.1588, found: 416.1587.



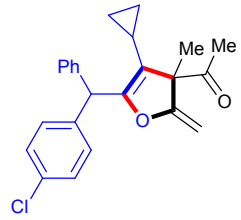
1-(5-((4-fluorophenyl)(phenyl)methyl)-3-methyl-2-methylene-4-(p-tolyl)-2,3-dihydrofuran-3-yl)ethanone (**3j**): Yield: 104.3 mg (80%) as yellow liquid; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.39 (d, *J* = 7.5 Hz, 1H), 7.34-7.28 (m, 4H), 7.22-7.19 (m, 2H), 7.15-7.00 (m, 4H), 6.94 (d, *J* = 2 Hz, 1H), 6.92 (d, *J* = 2 Hz, 1H), 5.27 (d, *J* = 10.5 Hz, 1H), 4.65 (d, *J* = 2.5 Hz, 1H), 4.16 (d, *J* = 2.5 Hz, 1H), 2.36 (s, 3H), 2.14 (s, 3H), 1.40 (s, 3H) ppm; ^{13}C NMR ($CDCl_3$, 100 MHz) : δ 205.2, 164.7, 164.6, 153.4, 140.2, 140.1, 137.5, 130.5, 130.4, 130.2, 130.2, 129.5, 128.7, 128.7, 128.6, 128.5, 128.4, 128.3, 128.2, 127.1, 118.6, 115.5, 115.3, 115.3, 115.1, 85.4, 64.6, 64.5, 47.6, 25.2, 21.5, 21.4, 21.1 ppm; HRMS (ESI-TOF): m/z calcd for $C_{28}H_{25}FO_2$ [M+H]+ : 413.1919, found: 413.1919.



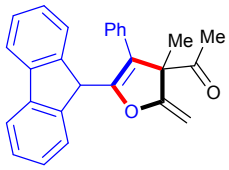
1-(5-benzhydryl-4-(4-butylphenyl)-3-methyl-2-methylene-2,3-dihydrofuran-3-yl)ethanone (**3k**): Yield: 98.9 mg (78%) as yellow liquid; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.87 (t, *J* = 3.5 Hz, 1H), 7.81-7.79 (m, 1H), 7.52 (d, *J* = 8 Hz, 1H), 7.34-7.28 (m, 2H), 7.21-2.00 (m, 7H), 6.95-6.93 (m, 2H), 5.30 (d, *J* = 10 Hz, 1H), 4.64 (d, *J* = 2.5Hz, 1H), 4.16 (d, *J* = 2.5Hz, 1H), 2.60 (t, *J* = 1.5 Hz, 2H), 2.15 (s, 3H), 1.63-1.41 (m, 2H), 1.40 (s, 3H), 1.39-0.95 (m, 2H), 0.94 (t, *J* = 1.0 Hz, 3H) ppm; ^{13}C NMR ($CDCl_3$, 125 MHz) : δ 205.4, 195.4, 164.7, 153.6, 153.5, 142.6, 140.3, 140.2, 137.6, 130.1, 128.9, 128.8, 128.4, 118.7, 115.6, 115.5, 115.5, 115.4, 115.2, 85.6, 64.7, 64.6, 47.7, 35.4, 33.5, 25.4, 22.5, 21.6, 21.5, `14.1 ppm; HRMS (ESI-TOF): m/z calcd for $C_{31}H_{32}O_2$ [M+H]+ : 436.2402, found: 436.2401.



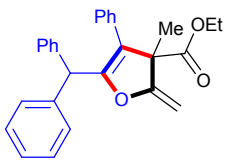
1-(5-((4-chlorophenyl)(phenyl)methyl)-4-cyclopropyl-3-methyl-2-methylene-2,3-dihydrofuran-3-yl)ethanone (**3l**): Yield: 104.5 mg (78%) as yellow liquid; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.62-7.34 (m, 5H), 7.33-7.27 (m, 4H), 5.48 (s, 1H), 4.54 (d, *J* = 2.4 Hz, 1H), 4.03 (d, *J* = 2.8 Hz, 1H), 2.01 (s, 3H), 1.43 (s, 3H), 1.25-1.21 (m, 1H), 0.77-0.74 (m, 2H), 0.68-0.66 (m, 1H), 0.50-0.36 (m, 1H) ppm; ^{13}C NMR ($CDCl_3$, 100 MHz) : δ 205.9, 165.1, 154.4, 140.3, 140.3, 132.5, 130.2, 128.9, 128.7, 128.6, 128.5, 128.4, 127.1, 126.9, 116.7, 84.8, 64.7, 47.8, 25.2, 21.8, 5.4, 5.2, 4.1 ppm; HRMS (ESI-TOF): m/z calcd for $C_{24}H_{23}ClO_2$ [M+H]+ : 378.1387, found: 378.1387.



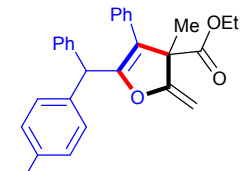
1-(5-(9H-fluoren-9-yl)-3-methyl-2-methylene-4-phenyl-2,3-dihydrofuran-3-yl)ethanone (**3m**): Yield:103.2 mg (77%) as yellow liquid; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 8.37 (d, *J* = 7.5 Hz, 1H), 7.80-7.43 (m, 2H), 7.42 (d, *J* = 7.5 Hz, 1H), 7.42-7.32 (m, 6H), 7.29-7.16 (m, 3H), 5.20 (d, *J* = 3 Hz, 1H), 5.12 (s, 1H), 4.52 (d, *J* = 3 Hz, 1H), 2.33 (s, 3H), 1.03 (s, 3H) ppm; ^{13}C NMR ($CDCl_3$, 125 MHz) : δ 205.1, 164.6, 164.5, 162.8, 162.8, 160.8, 153.9, 153.8, 140.1, 140.1, 136.1, 135.8, 135.8, 131.6, 131.6, 130.5, 130.4, 130.2, 130.2, 128.4, 128.3, 127.7, 127.6, 127.1, 127.1, 115.6, 115.4, 115.3, 115.2, 85.6, 64.6, 64.5, 47.7, 25.2, 25.1, 21.5, 21.4 ppm; HRMS (ESI-TOF): m/z calcd for $C_{27}H_{22}O_2$ [M+H]+ : 379.1700, found: 379.1700.



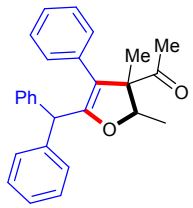
ethyl 5-benzhydryl-3-methyl-2-methylene-4-phenyl-2,3-dihydrofuran-3-carboxylate (**3n**): Yield: 114.10 mg (79%) as yellow liquid; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.39 (d, *J* = 1.5 Hz, 1H), 7.38-7.35 (m, 3H), 7.34-7.31 (m, 3H), 7.28 (t, *J* = 8.5 Hz, 4H), 7.24 (d, *J* = 7.5 Hz, 2H), 7.16-7.15 (m, 2H), 5.15 (s, 1H), 4.61 (d, *J* = 2.5 Hz, 1H), 4.29 (d, *J* = 3 Hz, 1H), 4.28-4.20 (m, 2H), 1.50 (s, 3H), 1.30 (t, *J* = 7 Hz, 3H) ppm; ^{13}C NMR ($CDCl_3$, 125 MHz) : δ 172.6, 165.1, 153.2, 140.8, 140.3,132.2, 129.3, 128.6, 128.5, 128.5, 128.1, 127.5, `126.1, 126.5,118.5, 84.4, 61.6, 58.1, 47.8, 22.9, 13.9 ppm; HRMS (ESI-TOF): m/z calcd for $C_{28}H_{26}O_3$ [M+H]+ : 411.1962, found: 411.1961.



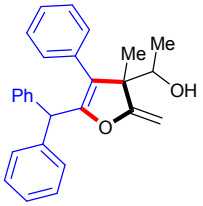
Ethyl5-((4-chlorophenyl)(phenyl)methyl)-3-methyl-2-methylene-4-phenyl-2,3-dihydrofuran-3-carboxylate (**3o**): Yield: 108.9 mg (78%) as yellow liquid; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.39 (d, *J* = 7 Hz, 1H), 7.35-7.32 (m, 6H), 7.31-7.28 (m, 2H), 7.24 (d, *J* = 9 Hz, 2H), 7.16 (d, *J* = 8.5 Hz, 1H), 7.14 (t, *J* = 1.5 Hz, 2H), 5.10 (s, 1H), 4.62 (d, *J* = 2.5 Hz, 1H), 4.31 (d, *J* = 3 Hz, 1H), 4.26 (q, *J* = 7 Hz, 2H), 1.50 (s, 3H), 1.31 (t, *J* = 7 Hz, 3H) ppm; ^{13}C NMR ($CDCl_3$, 125 MHz) : δ 172.4, 172.3, 164.9, 152.7, 140.3, 139.8, 139.3, 138.8, 132.8, 132.4, 132.1, 130.6, 130.1, 129.3, 129.2, 129.9, 128.7, 128.7, 128.6, 128.5, 128.5, 128.2, 127.7, 127.6, 127.1, 126.7, 118.8, 84.6, 61.7, 61.6, 58.2, 58.1, 47.3, 22.9, 14.1 ppm; HRMS (ESI-TOF): m/z calcd for $C_{28}H_{25}ClO_3$ [M+H]+ : 445.1572, found: 445.1573.



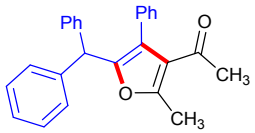
1-(5-benzhydryl-2,3-dimethyl-4-phenyl-2,3-dihydrofuran-3-yl)ethanone (**5**): Yield: 78.4 mg (78%) as yellow liquid; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.31 (t, *J* = 2 Hz, 4H), 7.22 (t, *J* = 7.2 Hz, 3H), 7.20-7.14 (m, 4H), 7.09 (t, *J* = 1.6 Hz, 2H), 6.92-6.89 (m, 2H), 5.27 (s, 1H), 4.43 (q, *J* = 6.8 Hz, 1H), 1.95 (s, 3H), 1.18 (d, *J* = 6.8 Hz, 3H), 1.09 (s, 3H) ppm; ^{13}C NMR ($CDCl_3$, 100 MHz) : δ 212.3, 156.5, 140.3, 133.7, 129.5, 128.8, 128.7, 128.6, 128.2, 127.5, 126.9, 126.8, 119.1, 86.7, 63.6, 49.1, 29.1, 19.4, 14.3 ppm; HRMS (ESI-TOF): m/z calcd for $C_{27}H_{26}O_2$ [M+H]+ : 382.1933, found: 382.1933.



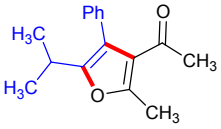
1-(5-benzhydryl-3-methyl-2-methylene-4-phenyl-2,3-dihydrofuran-3-yl)ethanol (**6**): Yield: 80.4 mg (80%) as yellow liquid; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.38 (t, J = 3.2 Hz, 3H), 7.37-7.32 (m, 4H), 7.30 (d, J = 2 Hz, 1H), 7.29-7.25 (m, 6H), 7.20 (t, J = 1.2 Hz, 2H), 5.01 (s, 1H), 4.60 (d, J = 2.5 Hz, 1H), 4.19 (d, J = 2.4 Hz, 1H), 3.65 (q, J = 6 Hz, 1H), 1.41 (s, 3H), 1.21 (d, J = 6.4 Hz, 3H) ppm; ^{13}C NMR ($CDCl_3$, 100 MHz) : δ 166.5, 152.4, 141.2, 140.7, 133.3, 130.4, 128.8, 128.6, 128.5, 128.3, 127.6, 126.7, 126.6, 118.5, 85.1, 73.1, 56.8, 47.9, 23.6, 17.1 ppm; HRMS (ESI-TOF): m/z calcd for $C_{27}H_{26}O_2$ [M+H]+ : 382.1933, found: 382.1933.



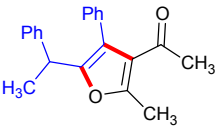
1-(5-benzhydryl-2-methyl-4-phenylfuran-3-yl)ethanone (**4a**): Yield: 103.1 mg 80%) as yellow liquid; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.44-7.40 (m, 5H), 7.33-7.28 (m, 5H), 7.27 (t, J = 7 Hz, 2H), 7.18 (d, J = 7.5 Hz, 3H), 5.23 (s, 1H), 2.58 (s, 3H), 1.94 (s, 3H) ppm; ^{13}C NMR ($CDCl_3$, 125 MHz) : δ 195.9, 157.7, 150.1, 141.6,133.3, 132.6, 130.1, 129.7, 128.7, 128.6, 128.4, 128.4, 128.3, 128.0, 127.8, 126.6, 124.0, 122.9, 122.5, 47.8, 30.7, 14.5 ppm; HRMS (ESI-TOF): m/z calcd for : $C_{26}H_{22}O_2$ [M+H]+ : 367.1700, found: 367.1701.



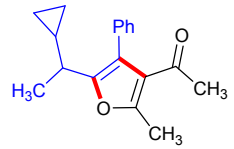
1-(5-isopropyl-2-methyl-4-phenylfuran-3-yl)ethanone (**4b**) :Yield: 119.5 mg (79%) as yellow liquid; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.50-7.47 (m, 2H), 7.27-7.18 (m, 1H), 7.09 (t, J = 7.6 Hz, 2H), 5.36 (s, 1H), 2.35 (s, 3H), 2.27 (s, 3H), 1.40 (s, 6H) ppm; ^{13}C NMR ($CDCl_3$, 100 MHz) : δ 193.3, 164.2, 163.1, 135.1, 128.3, 127.9, 125.9, 123.4, 99.4, 47.9, 30.7, 28.4, 15.7 ppm; HRMS (ESI-TOF): m/z calcd for $C_{16}H_{18}O_2$ [M+H]+ : 243.1387, found: 243.1389.



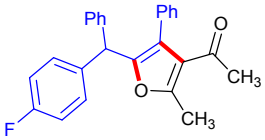
1-(2-methyl-4-phenyl-5-(1-phenylethyl)furan-3-yl)ethanone (**4c**): Yield: 108.1mg (79%) as yellow liquid; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.50 (dd, J_1 = 1.2 Hz, J_2 = 8.4 Hz, 2H), 7.38-7.34 (m, 3H), 7.32 (t, J = 6 Hz, 2H), 7.30-7.25 (m, 2H), 7.17 (d, J = 7.6 Hz, 1H), 5.19 (s, 1H), 2.59 (s, 3H), 1.96 (s, 3H), 1.93 (s, 3H) ppm; ^{13}C NMR ($CDCl_3$, 100 MHz) : δ 194.1, 164.8, 163.8, 144.8, 134.7, 128.5, 128.3, 128.1, 126.8, 126.4, 126.2, 123.2, 102.4, 54.2, 29.9, 26.6, 15.3 ppm; HRMS (ESI-TOF): m/z calcd for : $C_{21}H_{20}O_2$ [M+H]+ : 305.1543, found: 305.1543.



1-(5-(1-cyclopropylethyl)-2-methyl-4-phenylfuran-3-yl)ethanone (**4d**): Yield: 113.05 mg (78%) as yellow liquid; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.71 (d, J = 1.6 Hz, 1H) 7.69 (d, J = 3.2 Hz, 1H) 7.58-7.32 (m, 2H), 7.32-7.20 (m, 1H), 5.33 (s, 1H), 2.44 (s, 3H), 2.40 (s, 3H), 1.51 (s, 3H), 0.60-0.57 (m, 1H), 0.47-0.43 (m, 1H), 0.35-0.31 (m, 1H), 0.09-0.02 (m, 2H) ppm; ^{13}C NMR ($CDCl_3$, 100 MHz) : δ 193.6, 163.1, 159.3, 145.1, 134.7, 131.7, 128.3, 128.3, 128.2, 127.7, 126.1, 126.1, 126.1, 123.4, 102.4, 51.4, 30.9, 24.5, 19.7, 15.6, 1.4, 1.3 ppm; HRMS (ESI-TOF): m/z calcd for $C_{18}H_{20}O_2$ [M+Na]+ : 291.1360, found: 291.1354.

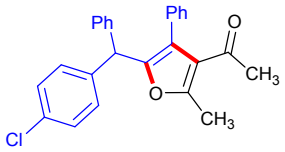


1-(5-((4-fluorophenyl)(phenyl)methyl)-2-methyl-4-phenylfuran-3-yl)ethanone (**4e**) : Yield: 99.21 mg (78%) as yellow liquid; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.43-7.40 (m, 3H), 7.32-7.30 (m, 2H), 7.27 (t, J = 2 Hz, 3H), 7.16-7.10 (m, 4H), 6.99 (t, J = 8.4 Hz, 2H), 5.20 (s, 1H), 2.58 (s, 3H), 1.93 (s, 3H) ppm; ^{13}C NMR ($CDCl_3$, 125 MHz) : δ 196.0, 162.7, 160.8, 157.9, 149.9, 141.5, 137.4, 137.4, 133.3, 130.4, 130.3, 130.1,

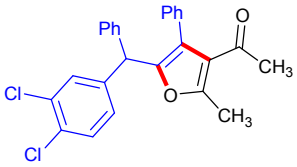


128.7, 128.6, 128.0, 126.9, 123.0, 122.7, 115.4, 115.2, 47.2, 30.8, 14.7 ppm; HRMS (ESI-TOF): m/z calcd for : $C_{26}H_{21}FO_2$ [M+H]+ : 385.1606, found: 385.1607.

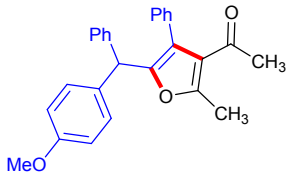
1-(5-((4-chlorophenyl)(phenyl)methyl)-2-methyl-4-phenylfuran-3-yl)ethanone (**4f**): Yield: 98.14 mg (78%) as yellow liquid; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.44 (d, *J* = 7.2 Hz, 3H), 7.33-7.27 (m, 7H), 7.16 (d, *J* = 7.2 Hz, 2H), 7.10 (d, *J* = 7.2 Hz, 2H), 5.20 (s, 1H), 2.59 (s, 3H), 1.95 (s, 3H) ppm; ^{13}C NMR ($CDCl_3$, 100 MHz) : δ 195.9, 157.9, 149.5, 141.1, 140.1, 133.1, 132.5, 130.1, 130.1, 128.6, 128.5, 127.9, 126.9, 122.9, 122.7, 47.1, 30.8, 14.6 ppm; HRMS (ESI-TOF): m/z calcd for $C_{26}H_{21}ClO_2$ [M+Na]+ : 400.1230, found: 400.1230.



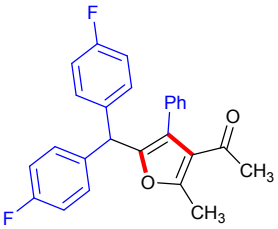
1-(5-((3,4-dichlorophenyl)(phenyl)methyl)-2-methyl-4-phenylfuran-3-yl)ethanone (**4g**): Yield: 95.69 mg (78%) as yellow liquid; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.43-7.41(m, 3H), 7.36-7.29 (m, 3H), 7.28-7.21 (m, 4H), 7.15-6.98 (m, 3H), 5.16 (s, 1H), 2.58 (s, 3H), 1.93 (s, 3H) ppm; ^{13}C NMR ($CDCl_3$, 100 MHz) : δ 195.7, 158.1, 148.8, 141.8, 140.4, 132.9, 132.4, 130.8, 130.6, 130.3, 129.9, 129.6, 128.7, 128.7, 128.6, 128.4, 128.4, 128.3, 128.1, 128.1, 127.1, 123.1, 122.9, 47.1, 30.7, 15.2, 14.6 ppm; HRMS (ESI-TOF): m/z calcd for $C_{26}H_{20}Cl_2O_2$ [M+Na]+ 435.0920, found: 435.0920.



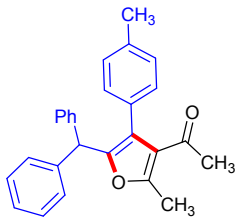
1-(5-((4-methoxyphenyl)(phenyl)methyl)-2-methyl-4-phenylfuran-3-yl)ethanone (**4h**): Yield: 98.1 mg (78%) as white solid; mp 135-136 °C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.39-7.34 (m, 3H), 7.25 (t, *J* = 7.5 Hz, 4H), 7.21-7.17 (m, 1H), 7.13 (d, *J* = 7.5 Hz, 2H), 7.06 (d, *J* = 8.5 Hz, 2H), 6.81 (d, *J* = 9 Hz, 2H), 5.15 (s, 1H), 3.75 (s, 3H), 2.54 (s, 3H), 1.90 (s, 3H) ppm; ^{13}C NMR ($CDCl_3$, 125 MHz) : δ 195.9, 158.3, 157.7, 150.4, 142.1, 133.7, 133.4, 130.1, 129.8, 128.7, 128.6, 128.4, 127.8, 126.6, 122.9, 122.3, 55.2, 47.1, 30.8, 12.6 ppm; HRMS (ESI-TOF): m/z calcd for $C_{27}H_{24}O_3$ [M+H]+ : 397.1805, found: 397.1805



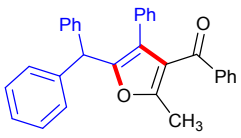
1-(5-(bis(4-fluorophenyl)methyl)-2-methyl-4-phenylfuran-3-yl)ethanone (**4i**): Yield: 99.27 mg (79%) as white solid; mp 116-117 °C;^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.42 (d, *J* = 7.5 Hz, 3H), 7.24 (d, *J* = 6 Hz, 2H), 7.09 (t, *J* = 7.5 Hz, 4H), 6.99 (t, *J* = 8.5 Hz, 4H), 5.17 (s, 1H), 2.57 (s, 3H), 1.92 (s, 3H) ppm; ^{13}C NMR ($CDCl_3$, 125 MHz) : δ 195.7, 162.6, 160.7, 157.8, 149.5, 137.1, 137.1, 133.1, 130.1, 130.1, 129.5, 128.6, 127.9, 122.9, 122.6, 115.4, 115.2, 64.3, 30.7, 15.6 ppm; HRMS (ESI-TOF): m/z calcd for : $C_{26}H_{20}F_2O_2$ [M+Na]+ : 403.1511, found: 403.1511.



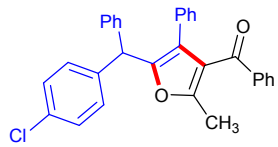
1-(5-benzhydryl-2-methyl-4-(p-tolyl)furan-3-yl)ethanone (**4j**): Yield: 99.50 mg (78%) as yellow liquid; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.32-7.28 (m, 6H), 7.26 (t, *J* = 0.8 Hz, 4H), 7.23-7.16 (m, 4H), 5.22 (s, 1H), 2.56 (s, 3H), 2.42 (s, 3H), 1.94 (s, 3H) ppm; ^{13}C NMR ($CDCl_3$, 100 MHz) : δ 196.2, 157.7, 155.1, 149.9, 141.7, 173.5, 130.2, 129.9, 129.3, 128.7, 128.4, 128.1, 126.6, 122.5, 47.6, 30.8, 21.3, 14.6 ppm; HRMS (ESI-TOF): m/z calcd for $C_{27}H_{24}O_2$ [M+H]+ : 381.1856, found: 381.1855.



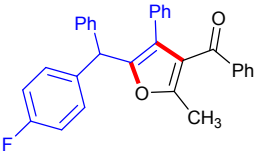
(5-benzhydryl-2-methyl-4-phenylfuran-3-yl)(phenyl)methanone (**4k**): Yield: 117.59 mg (78%) as yellow solid; mp 122-123 °C;^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.67 (d, *J* = 8.5 Hz, 1H), 7.39-7.28 (m, 8H), 7.28-7.25 (m, 4H), 7.23 (d, *J* = 8.0 Hz, 2H), 7.18 (d, *J* = 7.5 Hz, 2H), 7.16-7.12 (m, 3H), 5.49 (s, 1H), 2.40 (s, 3H) ppm; ^{13}C NMR ($CDCl_3$, 125 MHz) : δ 192.5, 156.3, 149.8, 141.8, 138.2, 132.4, 132.3, 129.6, 129.5, 129.5, 128.8, 128.8, 128.7, 128.5, 128.5, 128.4, 128.1, 127.9, 127.5, 126.9, 126.7, 123.6, 121.8, 47.8, 13.9 ppm; HRMS (ESI-TOF): m/z calcd for : $C_{31}H_{24}O_2$ [M+H]+ : 429.1856, found*:* 429.1857.



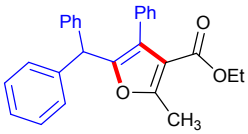
(5-((4-chlorophenyl)(phenyl)methyl)-2-methyl-4-phenylfuran-3-yl)(phenyl)methanone (**4l**): Yield: 114.80 mg (79%) as yellow liquid; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.66 (t, *J* = 7.2 Hz, 2H), 7.36 (t, *J* = 6.4 Hz, 3H), 7.35-7.30 (m, 5H), 7.26-7.23 (m, 3H), 7.19-7.16 (m, 5H), 7.12 (d, *J* = 1.6 Hz, 1H), 5.45 (s, 1H), 2.40 (s, 3H) ppm; ^{13}C NMR ($CDCl_3$, 100 MHz) : δ 192.4, 156.4, 149.3, 141.3, 140.3, 138.2, 132.6, 130.2, 129.4, 128.8, 128.6, 128.2, 128.1, 127.5, 127.1, 126.9, 123.8, 121.9, 47.3, 13.9 ppm; HRMS (ESI-TOF): m/z calcd for : $C_{31}H_{23}ClO_2$ [M+H]+ : 463.1467, found*:* 463.1467.

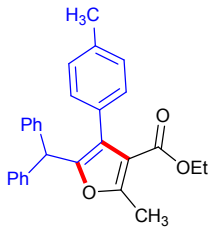


(5-((4-fluorophenyl)(phenyl)methyl)-2-methyl-4-phenylfuran-3-yl)(phenyl)methanone (**4m**): Yield: 116.66 mg (79%) as yellow liquid; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.67 (t, *J* = 1 Hz, 2H), 7.36 (t, *J* = 8.0 Hz, 3H), 7.28-7.22 (m, 4H), 7.21-7.17 (m, 6H), 7.11 (t, *J* = 1.5 Hz, 2H), 7.02 (t, *J* = 8.5 Hz, 2H), 5.45 (s, 1H), 2.39 (s, 3H) ppm; ^{13}C NMR ($CDCl_3$, 125 MHz) : δ 192.4, 162.6, 160.7, 156.3, 149.6, 141.6, 138.2, 137.5, 137.5, 132.4, 132.2, 130.3, 129.4, 128.7, 128.5, 128.1, 127.9, 126.8, 123.6, 121.9, 115.3, 115.2, 47.2, 13.8 ppm; HRMS (ESI-TOF): m/z calcd for : $C_{31}H_{23}FO_2$ [M+H]+ : 447.1762, found*:* 447.1768.



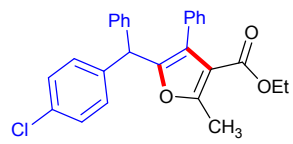
ethyl 5-benzhydryl-2-methyl-4-phenylfuran-3-carboxylate (**4n**): Yield: 110.20 mg (79%) as white solid; mp 120-121 °C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.90 (d, *J* = 8 Hz, 1H),7.39-7.32 (m, 5H) 7.28-7.21 (m, 5H), 7.18-7.10 (m, 4H), 5.26 (s, 1H), 4.08 (q, *J* = 7 Hz, 2H) 2.57 (s, 3H), 1.05 (t, *J* = 7.5 Hz, 3H) ppm; ^{13}C NMR ($CDCl_3$, 125 MHz) : δ 192.8, 164.3, 158.8, 154.7, 141.9, 141.5, 139.1, 138.4, 132.8, 130.2, 129.9, 129.4, 128.9, 128.8, 128.7, 128.5, 128.5, 128.4, 128.1, 127.8, 127.2, 126.7, 124.2, 59.9, 47.9, 14.4, 14.1 ppm; HRMS (ESI-TOF): m/z calcd for $C_{27}H_{24}O_3$ [M+H]+ : 397.1805; found397.1806.





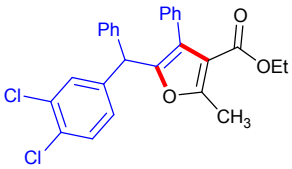
ethyl 5-benzhydryl-2-methyl-4-(p-tolyl)furan-3-carboxylate (**4o**): Yield: 107.36mg (78%) as yellow liquid; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.32-7.28 (m, 7H), 7.24-7.17 (m, 7H), 5.30 (s, 1H), 4.14 (q, *J* = 7.2 Hz, 2H) 2.60 (s, 3H), 2.40 (s, 3H), 1.13 (t, *J* = 7.2 Hz, 3H) ppm; ^{13}C NMR ($CDCl_3$, 100 MHz) : δ 164.6, 158.6, 150.1, 141.8, 136.8, 129.9, 129.7, 128.8, 128.5, 128.3, 126.5, 123.2, 59.8, 47.6, 21.3, 21.3, 14.4, 14.1 ppm; HRMS (ESI-TOF): m/z calcd for $C_{28}H_{26}O_3$ [M+H]+ : 411.1962, found: 411.1961.

ethyl 5-((4-chlorophenyl)(phenyl)methyl)-2-methyl-4-phenylfuran-3-carboxylate (**4p**): Yield: 105.50 mg (78%) as white solid; mp 98-99 °C;^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.38-7.32 (m, 4H), 7.30-7.24 (m, 5H), 7.17 (t, *J* = 1.6 Hz, 3H), 7.10 (d, *J* = 8.4 Hz, 2H), 5.26 (s, 1H), 4.13 (q, *J* = 7.2 Hz, 2H), 2,61 (s, 3H), 1.09 (t, *J* = 7.2 Hz, 3H) ppm; ^{13}C NMR ($CDCl_3$, 100 MHz) : δ 164.1, 158.9, 149.6, 141.2, 140.2,

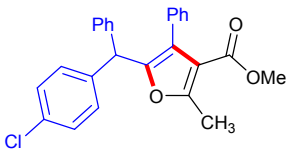


132.7, 132.5, 130.1, 130.1, 128.7, 128.5, 127.8, 127.3, 126.8, 123.5, 113.8, 59.8, 47.1, 14.3, 13.9 ppm; HRMS (ESI-TOF): m/z calcd for $C_{27}H_{23}ClO_3$ [M+H]+ : 431.1416, found: 431.1416.

ethyl 5-((3,4-dichlorophenyl)(phenyl)methyl)-2-methyl-4-phenylfuran-3-carboxylate (**4q**): Yield: 102.54 mg (78%) as yellow liquid; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.45 (t, *J* = 8 Hz, 1H), 7.42-7.36 (m, 5H), 7.35-7.28 (m, 2H), 7.24-7.22 (m, 1H), 7.16-7.15 (m, 2H), 7.00-6.97 (m, 2H), 5.23 (s, 1H), 4.13 (q, *J* = 5 Hz, 2H), 2.62 (s, 3H), 1.09 (t, *J* = 7.5 Hz, 3H) ppm; ^{13}C NMR ($CDCl_3$, 125 MHz) : δ 164.0, 159.0, 148.9, 142.0, 140.6, 132.4, 131.4, 130.6, 130.2, 130.0, 129.8, 129.0, 128.7, 128.6, 128.6, 128.6, 128.6, 128.4, 128.2, 127.8, 127.4, 127.1, 124.5, 113.9, 59.9, 47.0, 14.4, 13.9 ppm; HRMS (ESI-TOF): m/z calcd for $C_{27}H_{22}Cl_2O_3$ [M+H]+ : 465.1026, found: 465.1028.



methyl 5-((4-chlorophenyl)(phenyl)methyl)-2-methyl-4-phenylfuran-3-carboxylate (**4r**): Yield: 103.34 mg (79%) as white solid; mp 113-114 °C;^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.40-7.37 (m, 3H), 7.32 (d, *J* = 7.6 Hz, 2H), 7.29-7.26 (m, 5H), 7.16 (d, *J* = 7.6 Hz, 2H), 7.10 (d, *J* = 8.4 Hz, 2H), 5.27 (s, 1H), 3.68 (s, 3H), 2.62 (s, 3H) ppm; ^{13}C NMR ($CDCl_3$, 100 MHz) : δ 164.5, 159.1, 149.7, 141.2, 140.2, 132.5, 132.5, 130.1, 129.9, 128.7, 128.5, 127.9, 127.3, 126.8, 123.5, 113.5, 51.1, 47.1, 14.4 ppm; HRMS (ESI-TOF): m/z calcd for : $C_{26}H_{21}ClO_3$ [M+Na]+ : 417.1259, found: 417.1257.



IV. X-ray Data and Crystal Structures

X-ray data of Compound 3h:

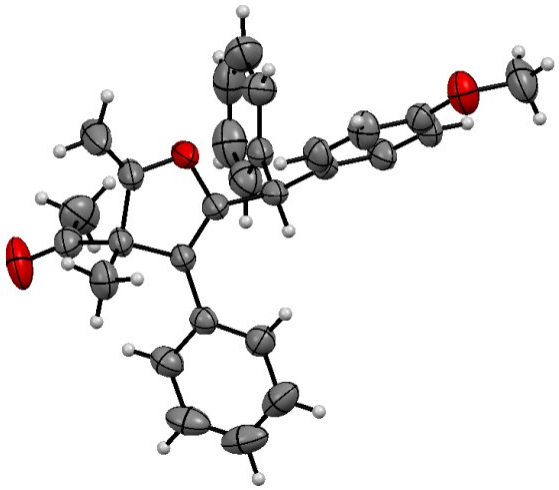


Figure S1. ORTEP representation of compound **3h** and thermal ellipsoids are drawn with 50% probability.

Crystal data and structure refinement for3h.

Identification code	shelx-97	
Empirical formula	$C_{28}H_{26}O_3$	
Formula weight	410.49	
Temperature	291 (2) K	
Wavelength	0.71073 Å	
Crystal system	'Triclinic'	
Space group	P-1	
Unit cell dimensions	a = 9.9166 (11) Å	α = 106.883° (5).
	b = 10.0554 (13) Å	β = 98.953 ° (5).
	c = 12.1593 (15) Å	γ = 94.007 ° (5).
Volume	1137.6(2)	
Z	2	
Density (calculated)	1.198 Mg/m^3	
Absorption coefficient	0.077 mm^{-1}	
F(000)	436.0	
Crystal size	0.4 x 0.32 x 0.28 mm^3	
Theta range for data collection	2.32 to 27.650 °.	
Index ranges	-12<=h<=12,-13<=k<=13,-15<=l<=15	
Reflections collected	5243	
Independent reflections	2905 [R(int) = 0.0607]	
Max. and min. transmission	0.971 and 0.973	
Refinement method	Full-matrix least-squares on F^2	
restraints / parameters	0 / 287	

Goodness-of-fit on F^2	0.956
Final R indices [I>2sigma(I)]	R1 = 0.0607, wR2 = 0.1611
R indices (all data)	R1 = 0.1298, wR2 = 0.1322

X-ray data of Compound 4h:

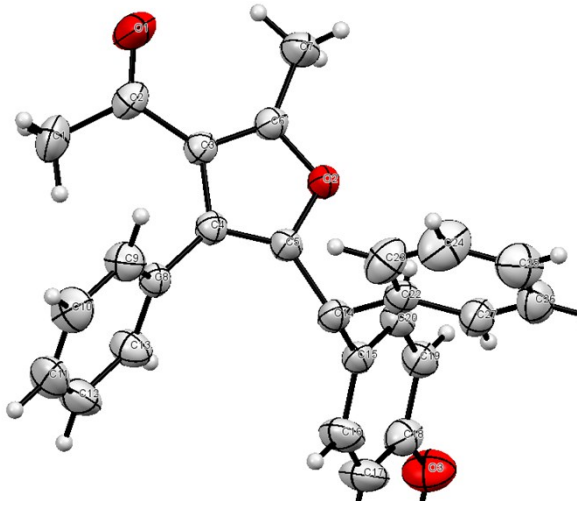


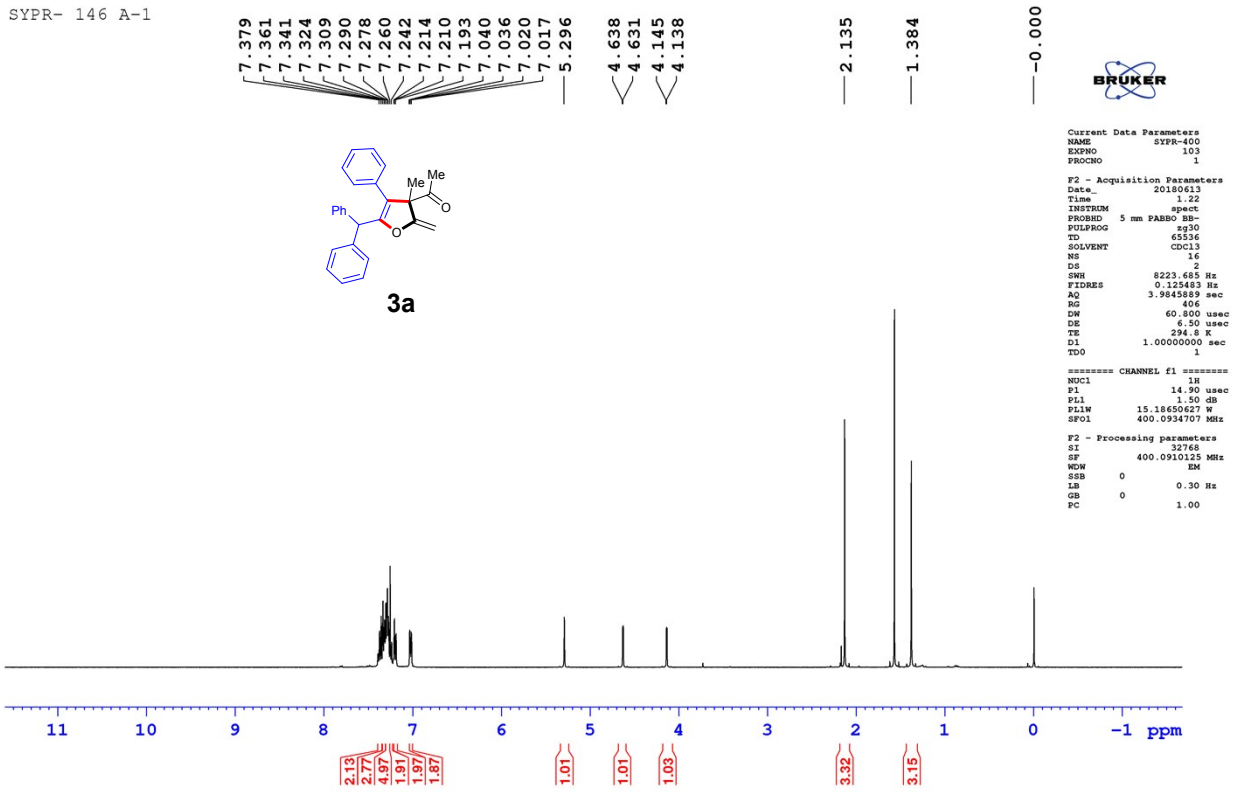
Figure S2. ORTEP representation of compound **4h** and thermal ellipsoids are drawn with 50% probability.

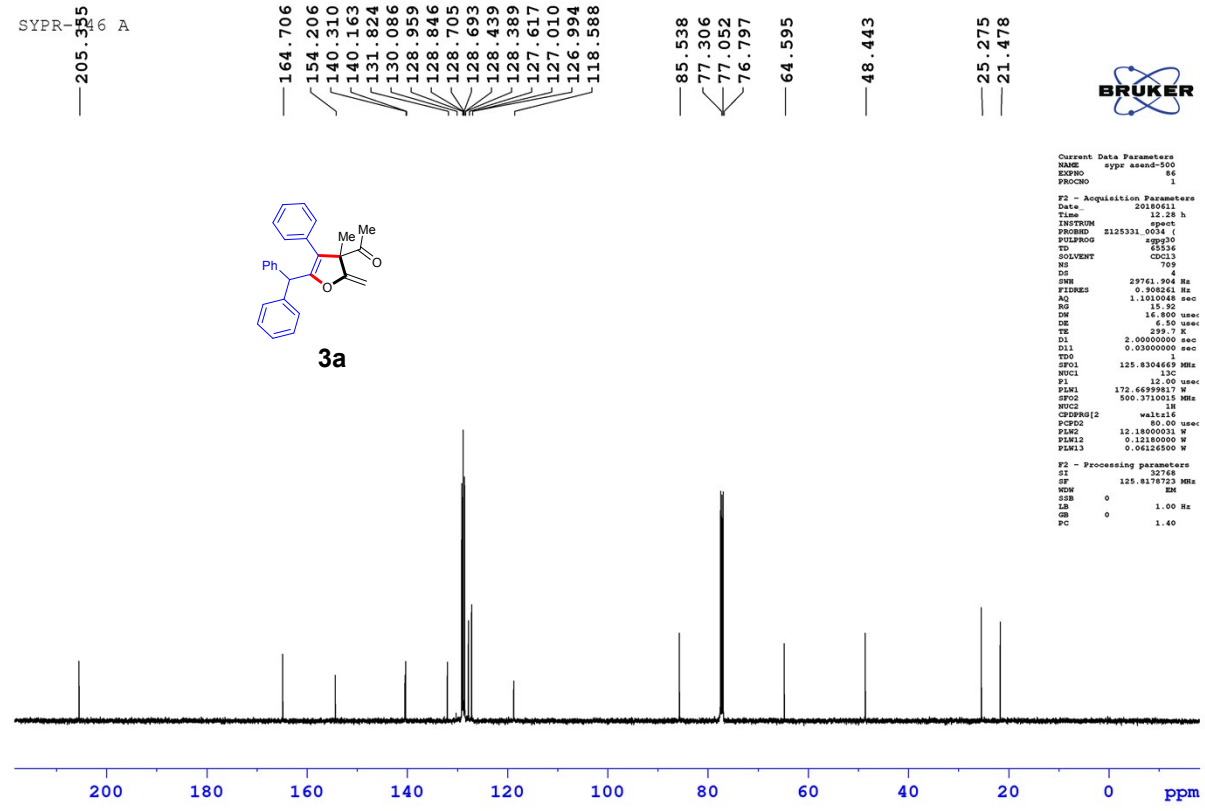
Crystal data and structure refinement for4h.

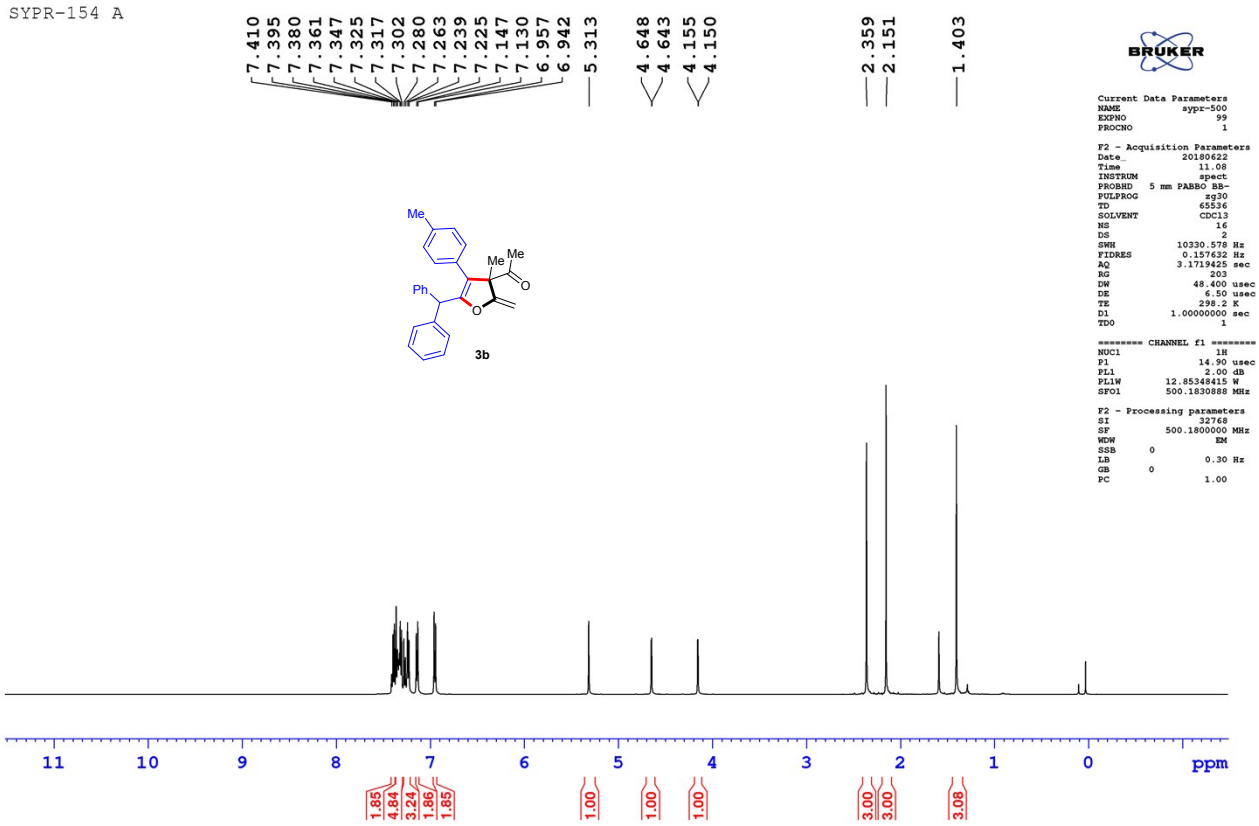
Identification code	'SHELXL-2014/7	
Empirical formula	$C_{27}H_{23}O_3$	
Formula weight	395.45	
Temperature	298 (2) K	
Wavelength	0.71073 Å	
Crystal system	'Triclinic'	
Space group	P-1	
Unit cell dimensions	a = 9.8049 (9) Å	α = 87.971° (4).
	b = 9.8109 (10) Å	β = 75.802 ° (4).
	c = 11.3980 (12) Å	γ =85.937 ° (4).

Volume	1060.08(18)
Z	2
Density (calculated)	1.239 Mg/m^3
Absorption coefficient	0.080 mm^{-1}
F(000)	418.0
Crystal size	0.42 x 0.32 x 0.22 mm^3
Theta range for data collection	2.466 to 27.973 °.
Index ranges	-12<=h<=12,-12<=k<=12,-15<=l<=15
Reflections collected	5052
Independent reflections	3935 [R(int) = 0.0603]
Max. and min. transmission	0.970 and 0.983
Refinement method	Full-matrix least-squares on F^2
restraints / parameters	0 / 271
Goodness-of-fit on F^2	1.065
Final R indices [I>2sigma(I)]	R1 = 0.0603, wR2 = 0.1658
R indices (all data)	R1 = 0.0779, wR2 = 0.1761

IV. Spectral Copies of Synthesized Compounds



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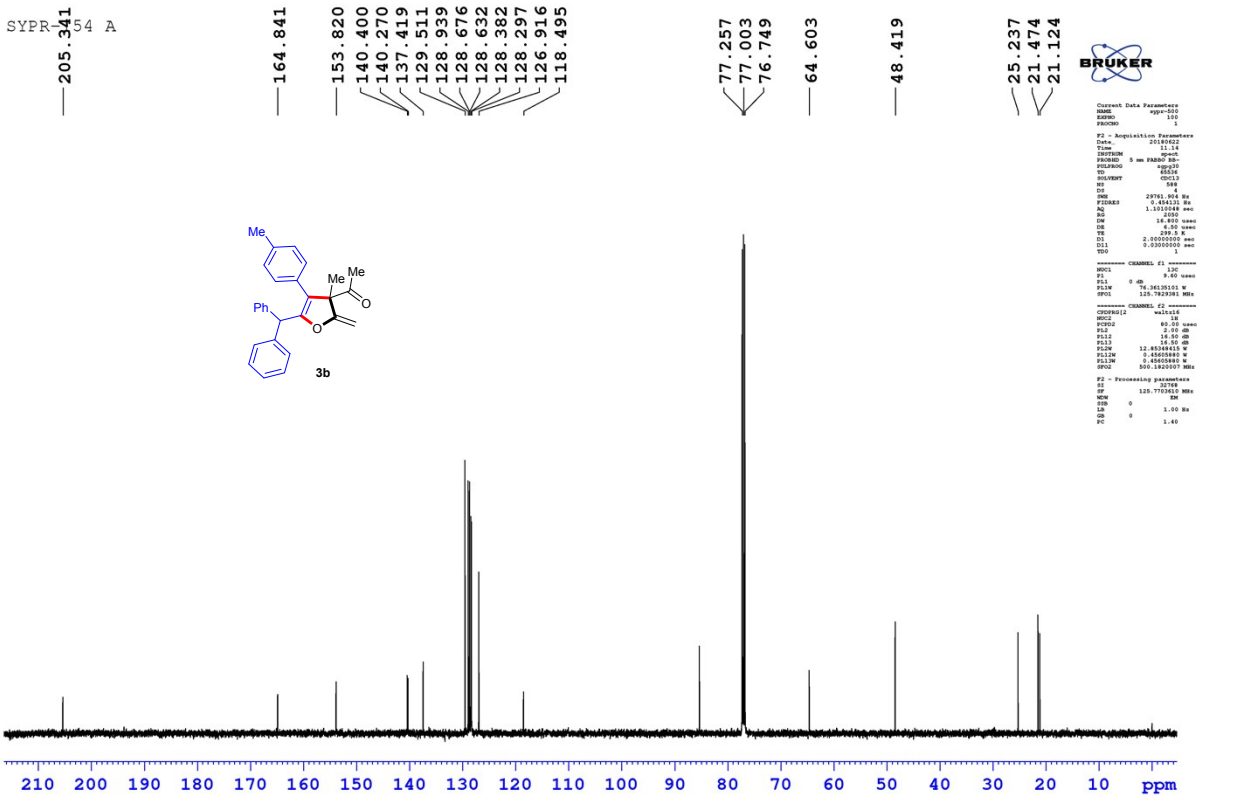
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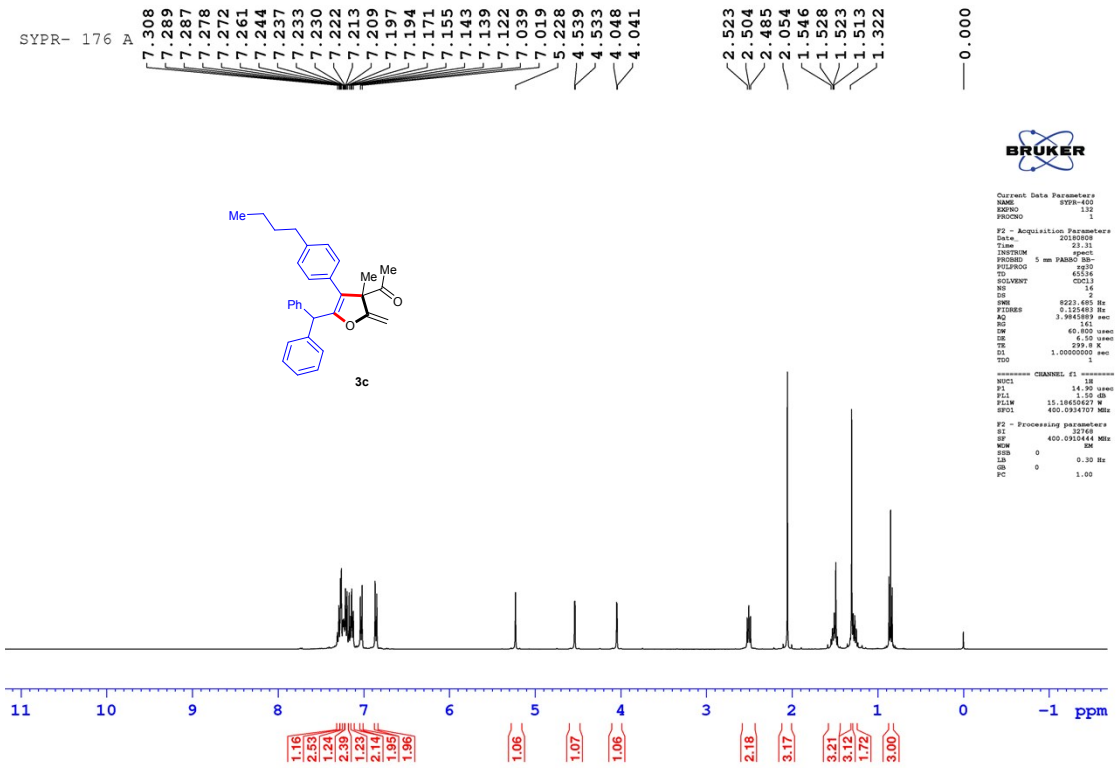
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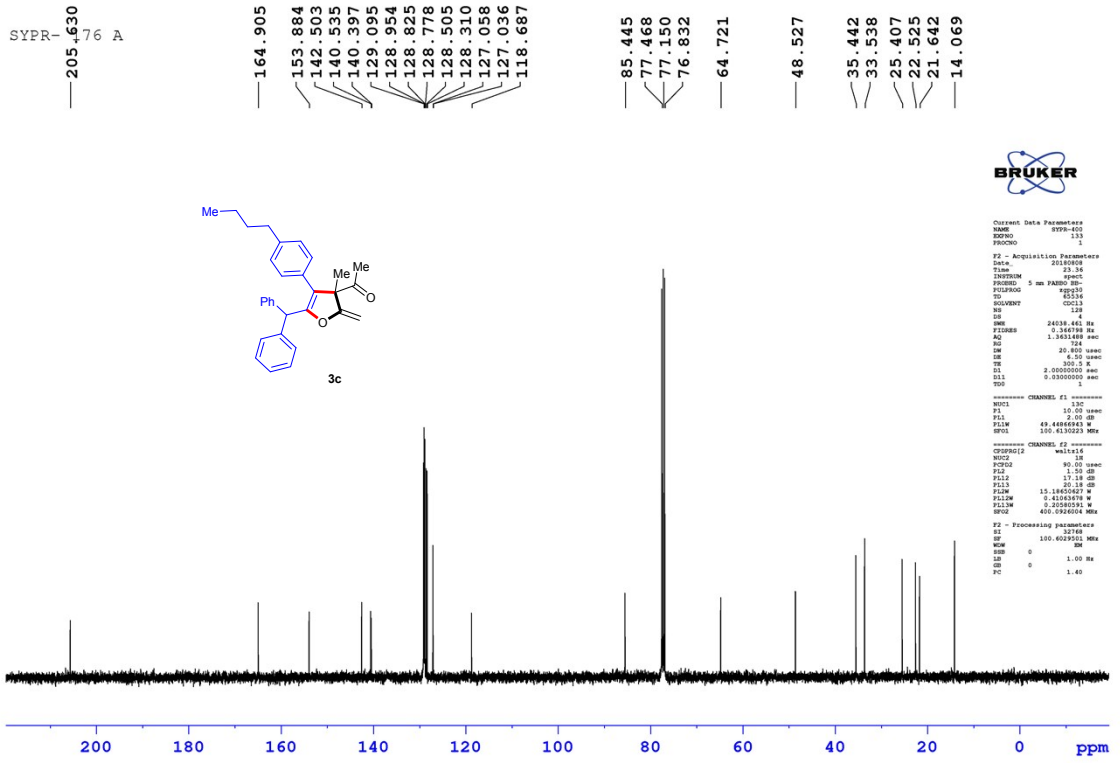
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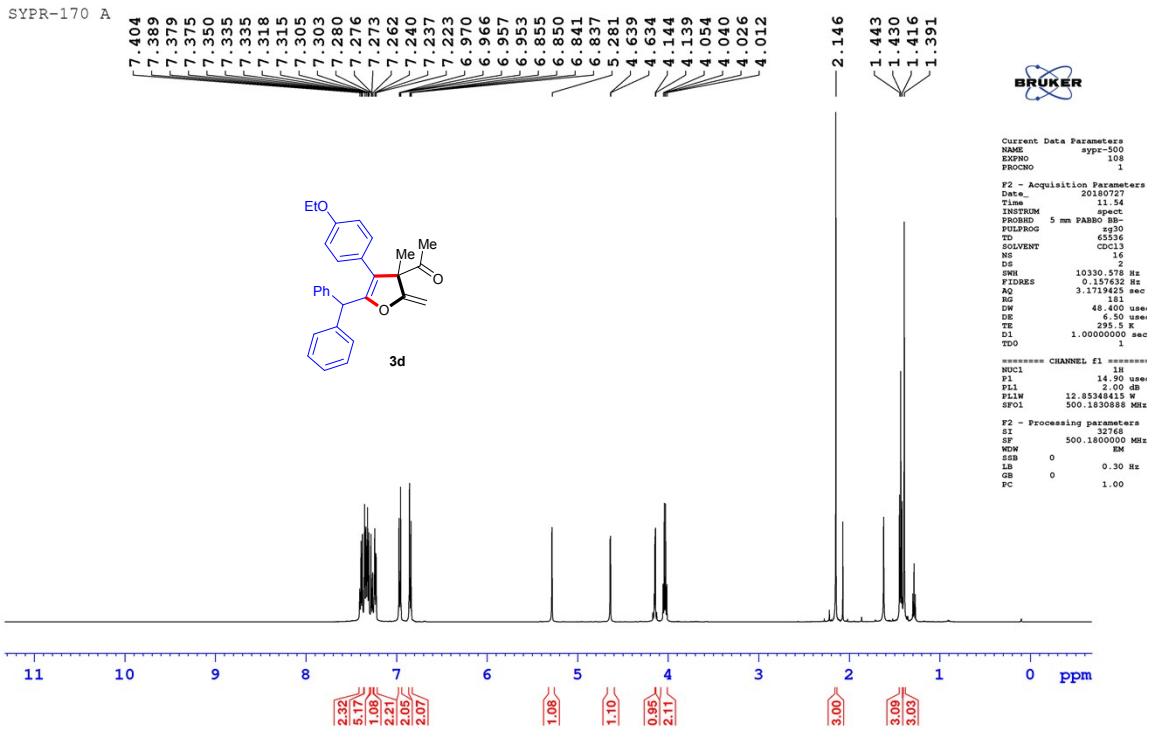
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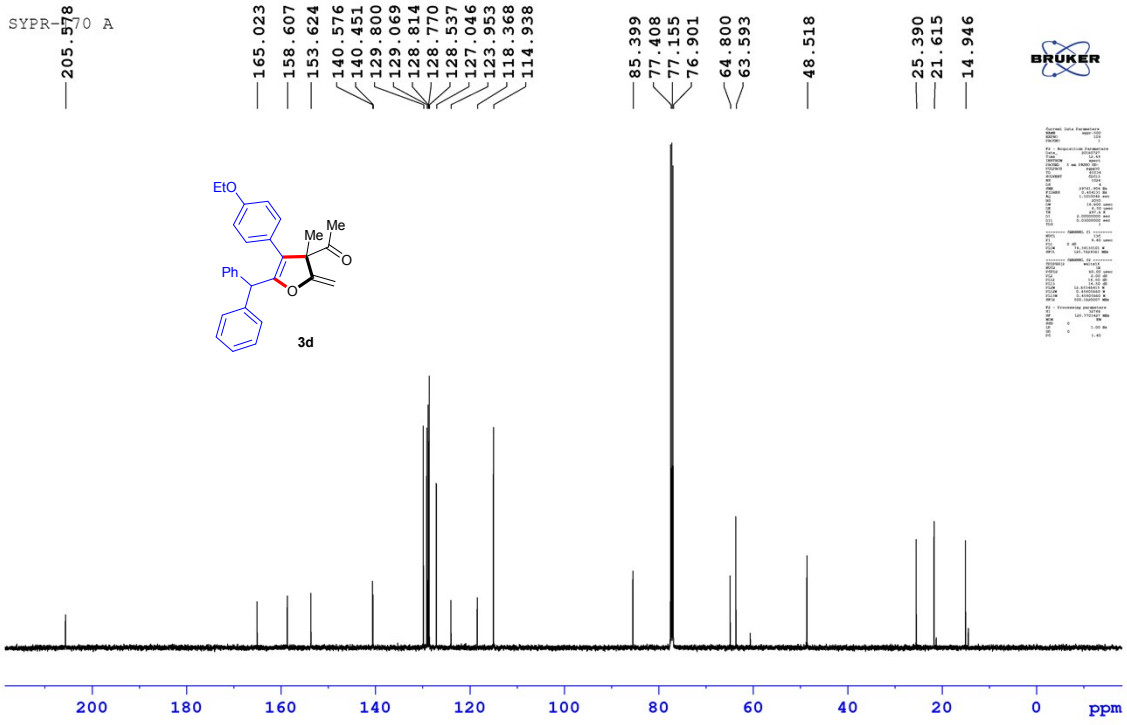
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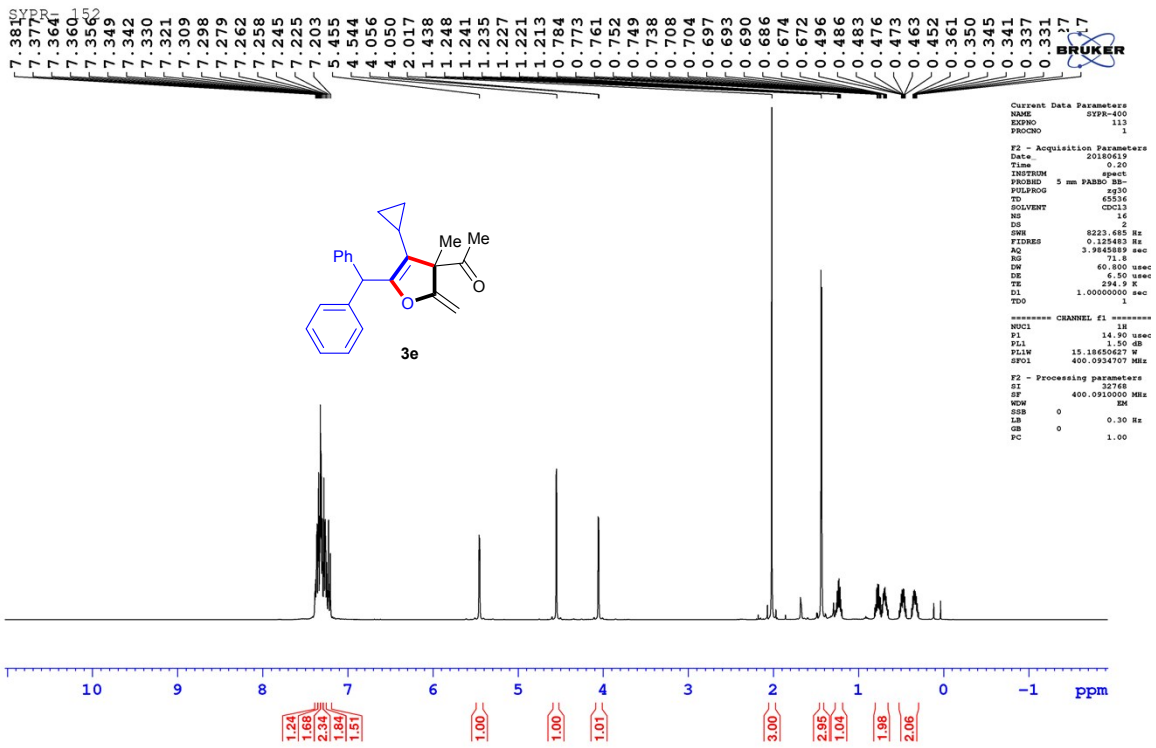
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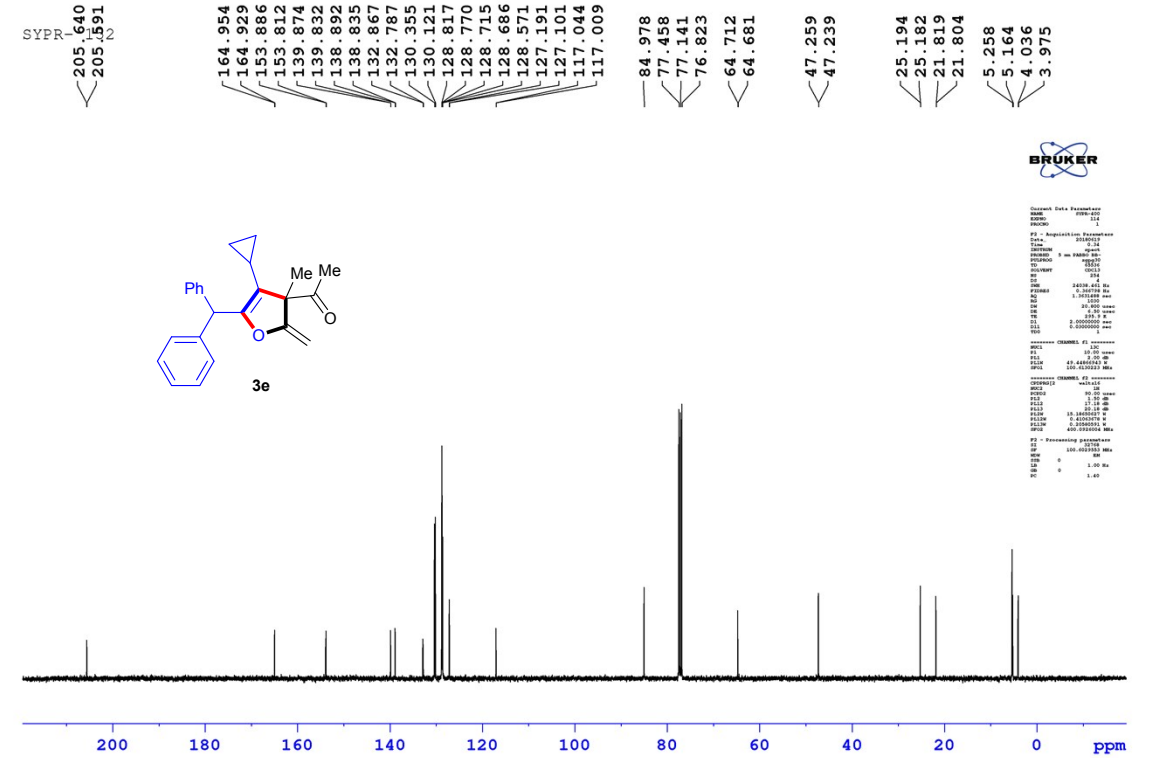
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BRUKER
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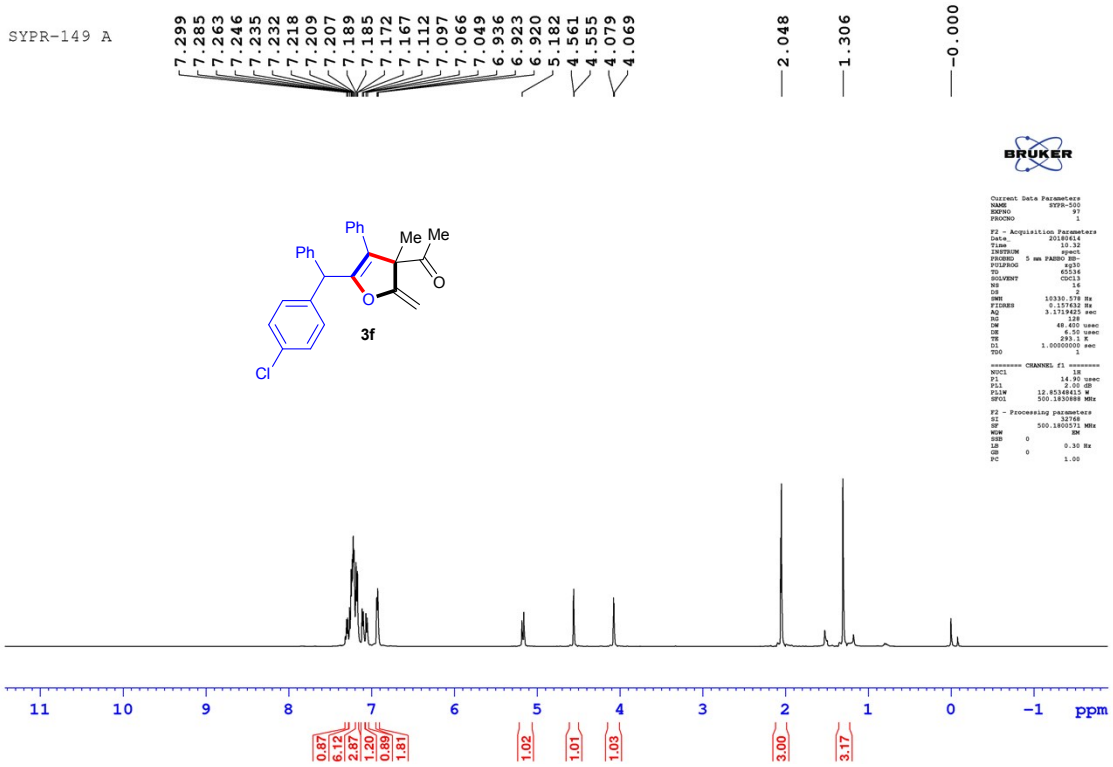
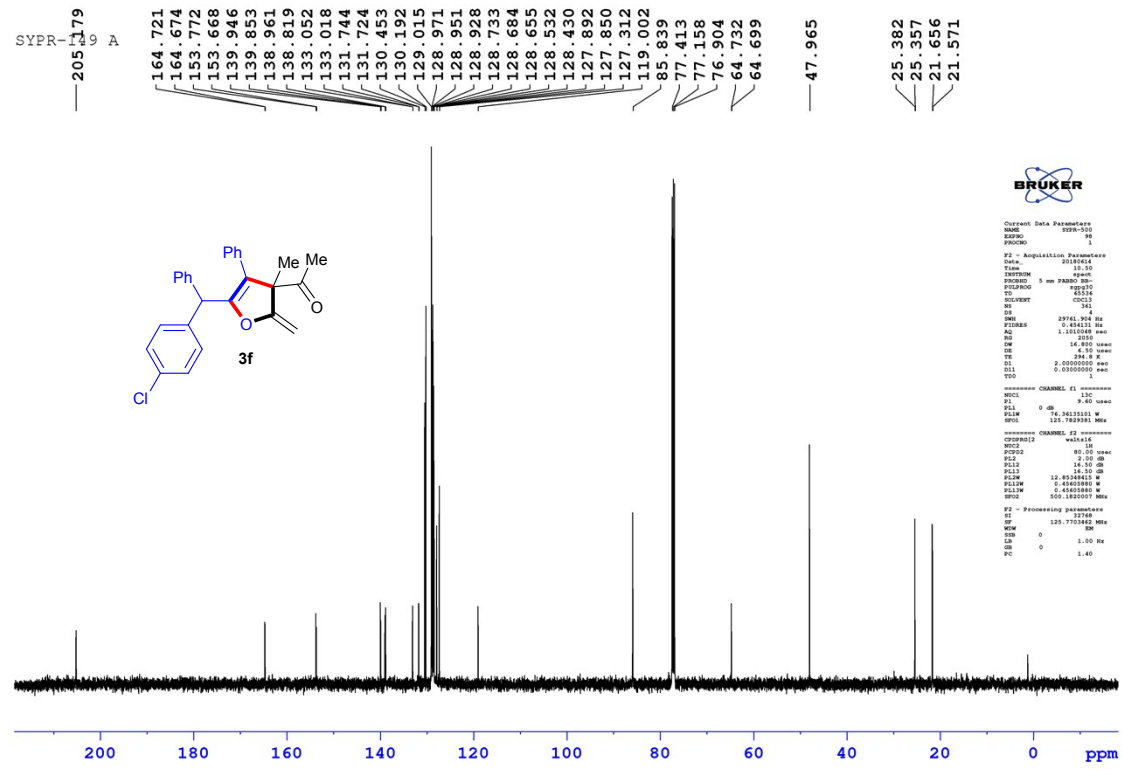
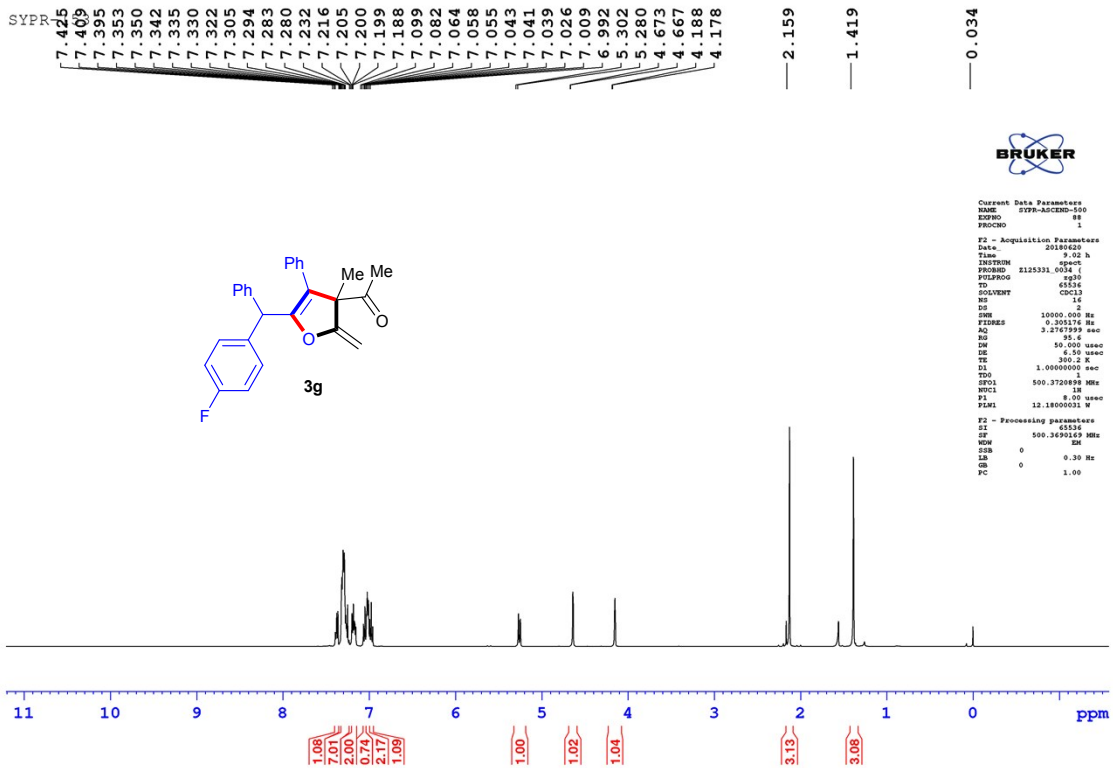
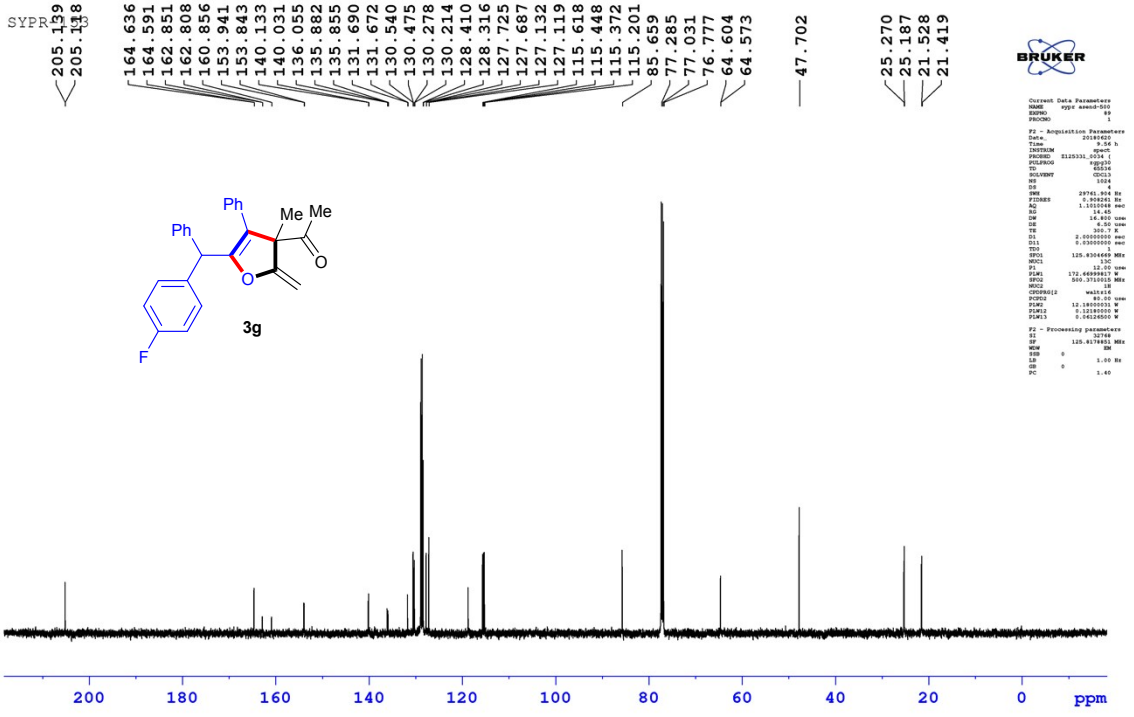
Me
Me
O
O
3e
200 180 160 140 120 100 80 60 40 20 0 ppm

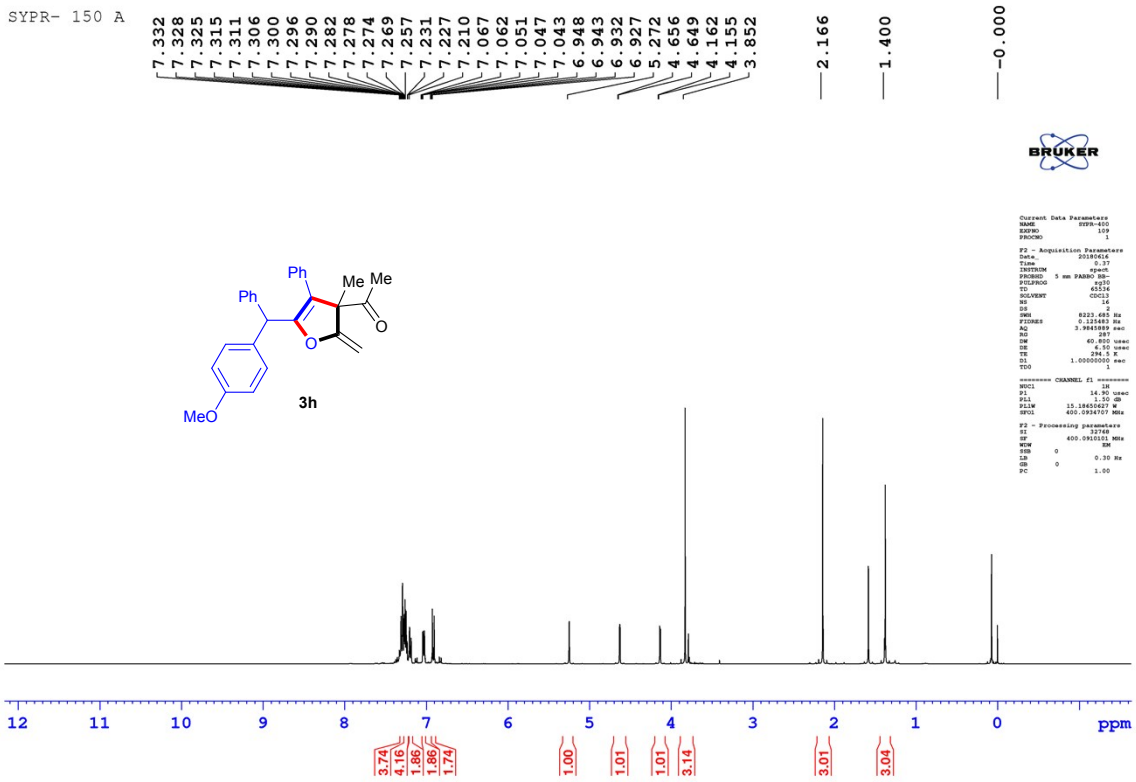

SYPR-149 A
7.299
7.285
7.263
7.246
7.235
7.232
7.218
7.209
7.207
7.189
7.185
7.172
7.167
7.112
7.097
7.066
7.049
6.936
6.923
6.920
5.182
4.561
4.555
4.079
4.069
2.048
1.306
0.000
Ph
Ph
Me
Me
O
O
Cl
3f
11
10
9
8
7
6
5
4
3
2
1
0
−1
ppm
0.87
6.12
2.87
1.20
0.89
1.81
1.02
1.01
1.03
3.00
3.17


SYPR-149 A
205.179
164.721
164.674
153.772
153.668
139.946
139.853
138.961
138.819
133.052
133.018
131.744
131.724
130.453
130.192
129.015
128.971
128.951
128.928
128.733
128.684
128.655
128.532
128.430
127.892
127.850
127.312
119.002
85.839
77.413
77.158
76.904
64.732
64.699
47.965
25.382
25.357
21.656
21.571
Ph
Ph
Me
Me
O
O
Cl
3f
200
180
160
140
120
100
80
60
40
20
0
ppm

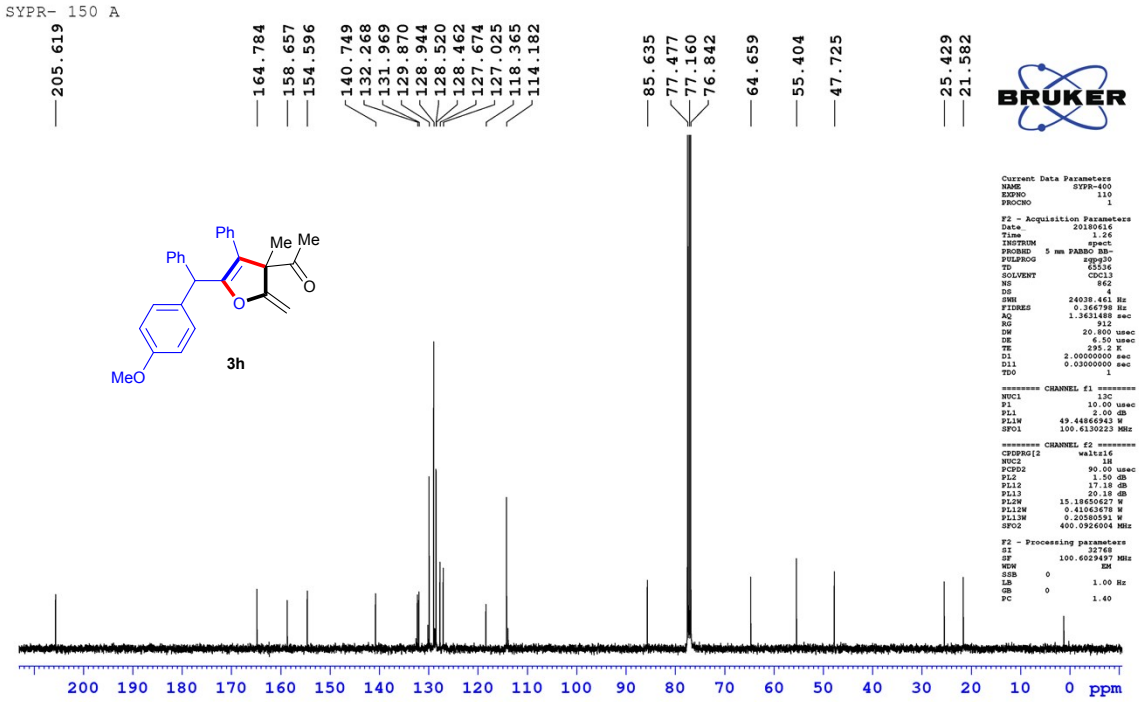

SYPR-
7.425
7.409
7.395
7.353
7.350
7.342
7.335
7.330
7.322
7.305
7.294
7.283
7.280
7.232
7.216
7.205
7.200
7.199
7.188
7.099
7.082
7.064
7.058
7.055
7.043
7.041
7.039
7.026
7.009
6.992
5.302
5.280
4.673
4.667
4.188
4.178
2.159
1.419
0.034
Ph
Ph
Me
Me
O
O
F
3g
11
10
9
8
7
6
5
4
3
2
1
0
ppm
1.08
7.01
2.00
0.74
2.17
1.09
1.00
1.02
1.04
3.13
3.08

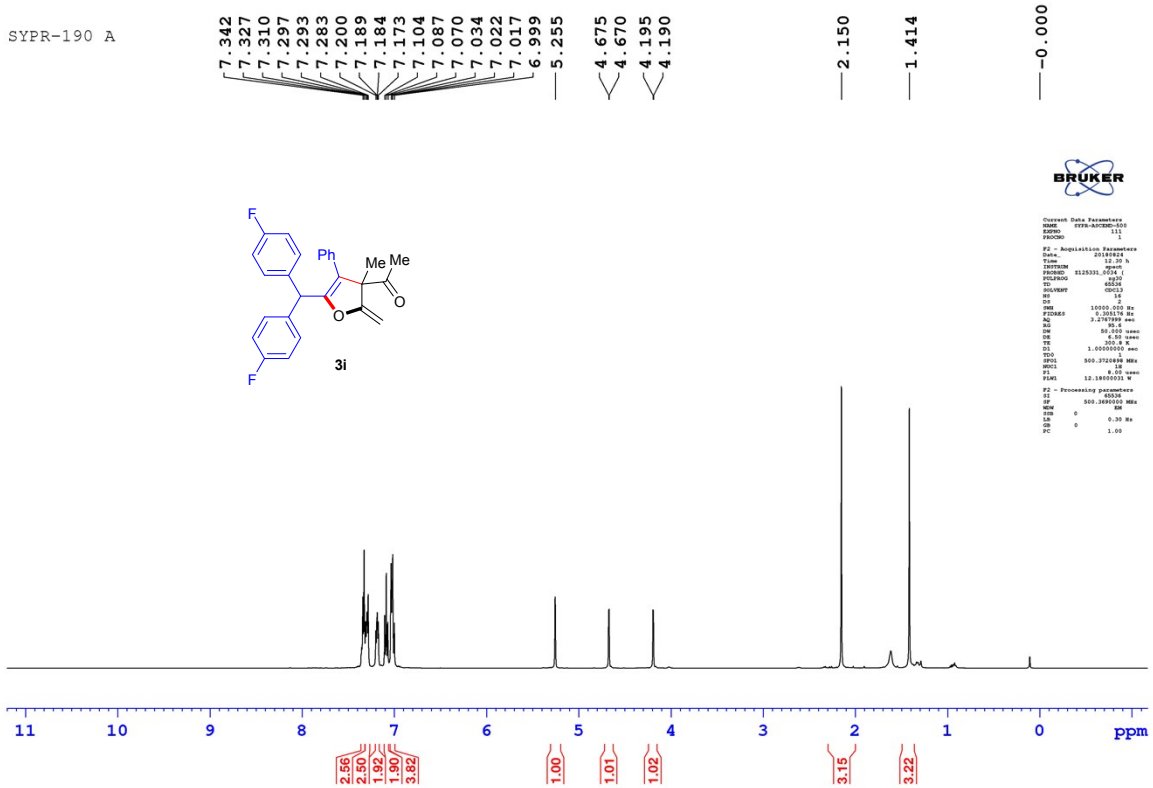

SYPR-
205.139
205.118
164.636
164.591
162.851
162.808
160.856
153.941
153.843
140.133
140.031
136.055
135.882
135.855
131.690
131.672
130.540
130.475
130.278
130.214
128.410
128.316
127.725
127.687
127.132
127.119
115.618
115.448
115.372
115.201
85.659
77.285
77.031
76.777
64.604
64.573
47.702
25.270
25.187
21.528
21.419
Ph
Ph
Me
Me
O
O
F
3g
200
180
160
140
120
100
80
60
40
20
0
ppm

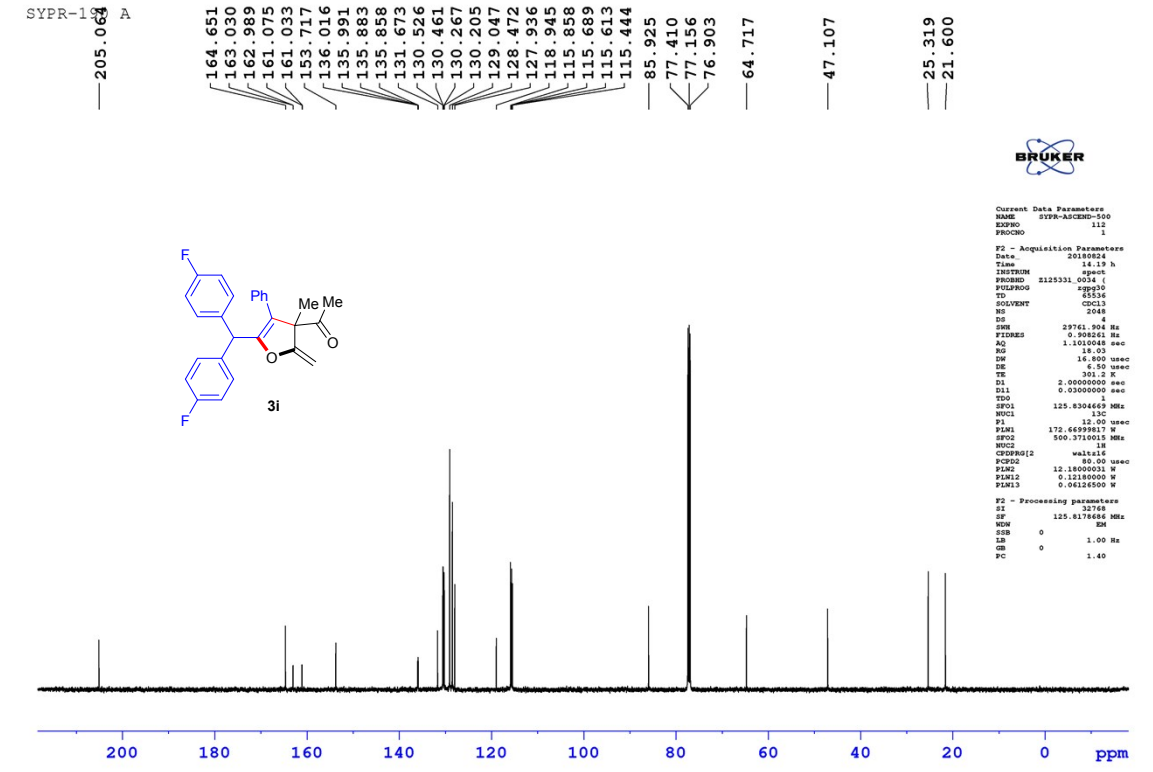
SYPR- 150 A

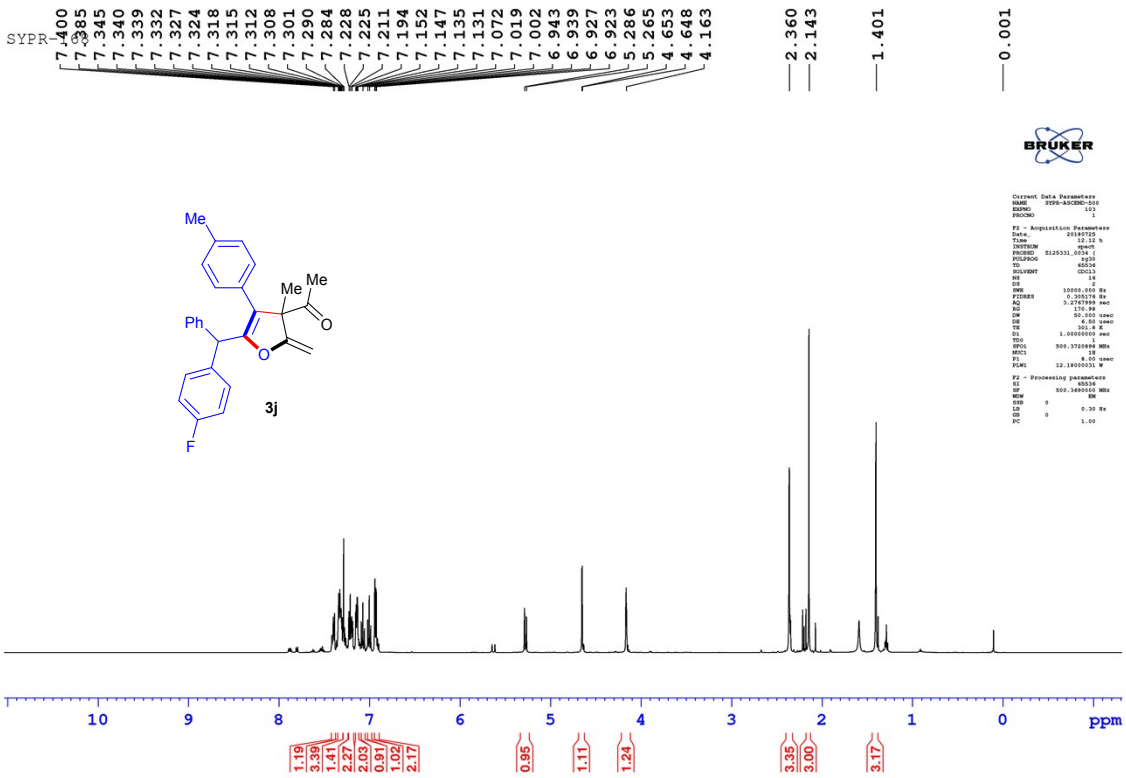


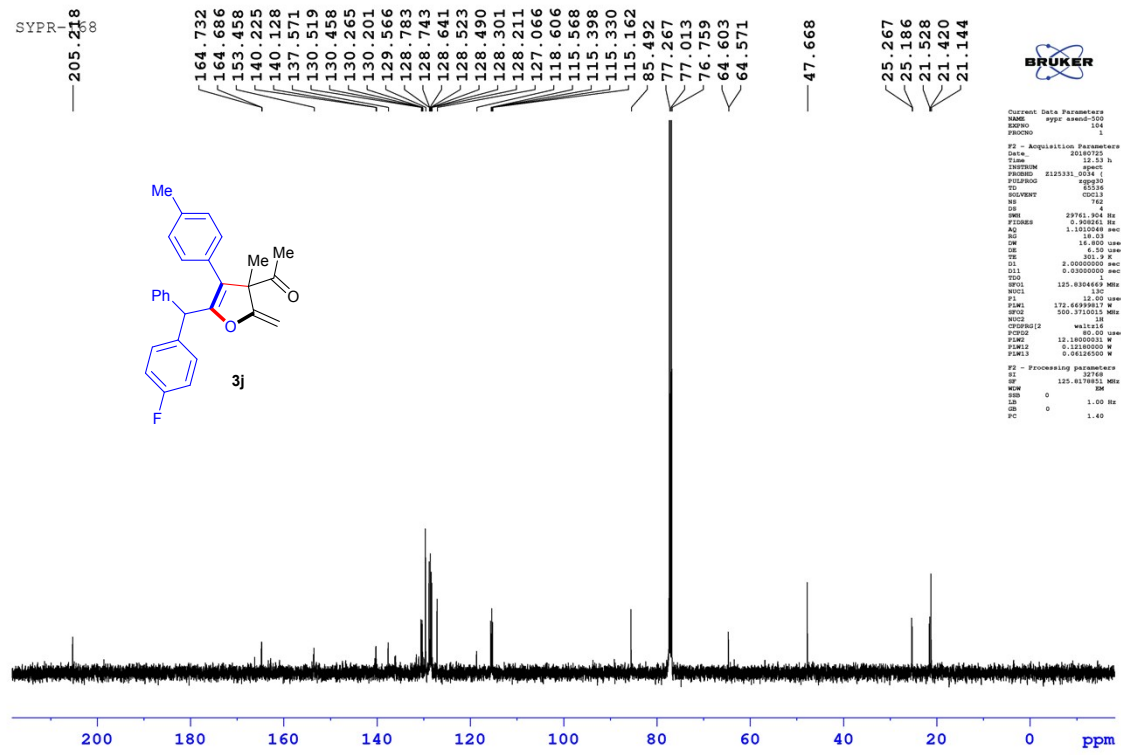
SYPR- 150 A

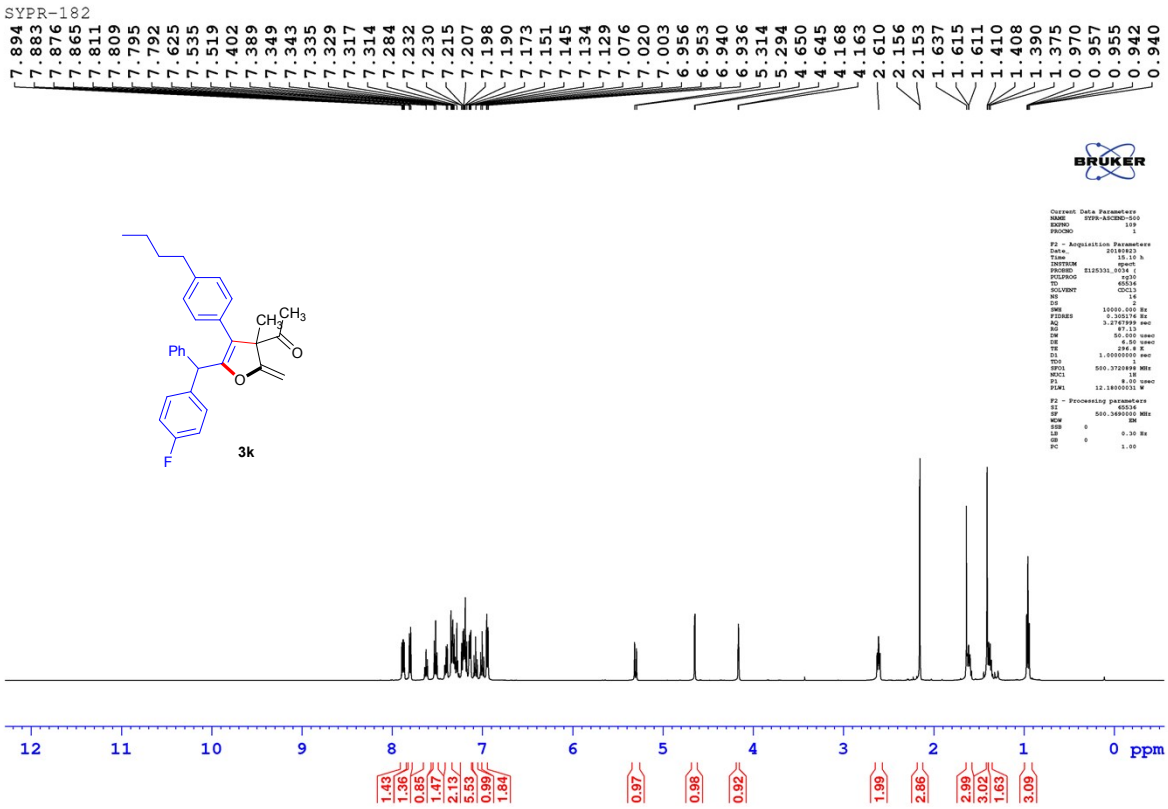


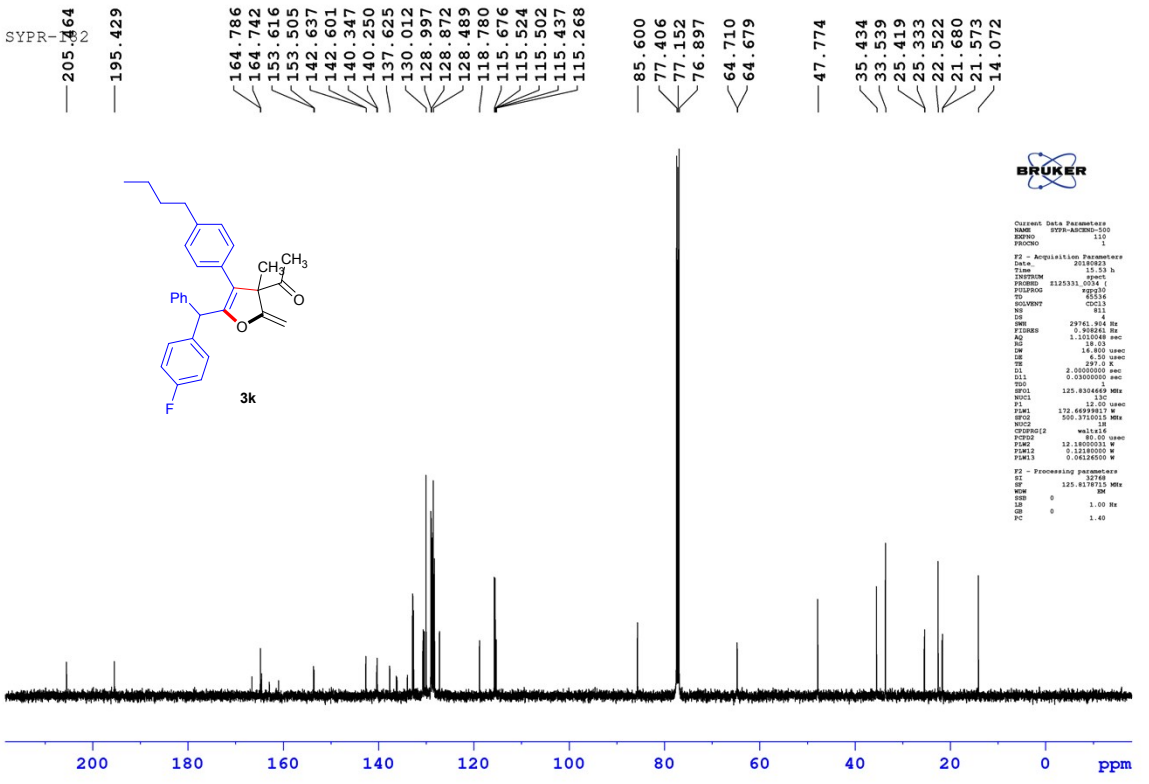

SYPR-190 A
7.342
7.327
7.310
7.297
7.293
7.283
7.200
7.189
7.184
7.173
7.104
7.087
7.070
7.034
7.022
7.017
6.999
5.255
4.675
4.670
4.195
4.190
2.150
1.414
0.000
BRUKER
F
Ph
Me Me
O
O
F
3i
11
10
9
8
7
6
5
4
3
2
1
0
ppm
2.56
2.50
1.92
1.90
3.82
1.00
1.01
1.02
3.15
3.22


SYPR-190 A
205.064
164.651
163.030
162.989
161.075
161.033
153.717
136.016
135.991
135.883
135.858
131.673
130.526
130.461
130.267
130.205
129.047
128.472
127.936
118.945
115.858
115.689
115.613
115.444
85.925
77.410
77.156
76.903
64.717
47.107
25.319
21.600
BRUKER
F
Ph
Me Me
O
O
F
3i
200
180
160
140
120
100
80
60
40
20
0
ppm

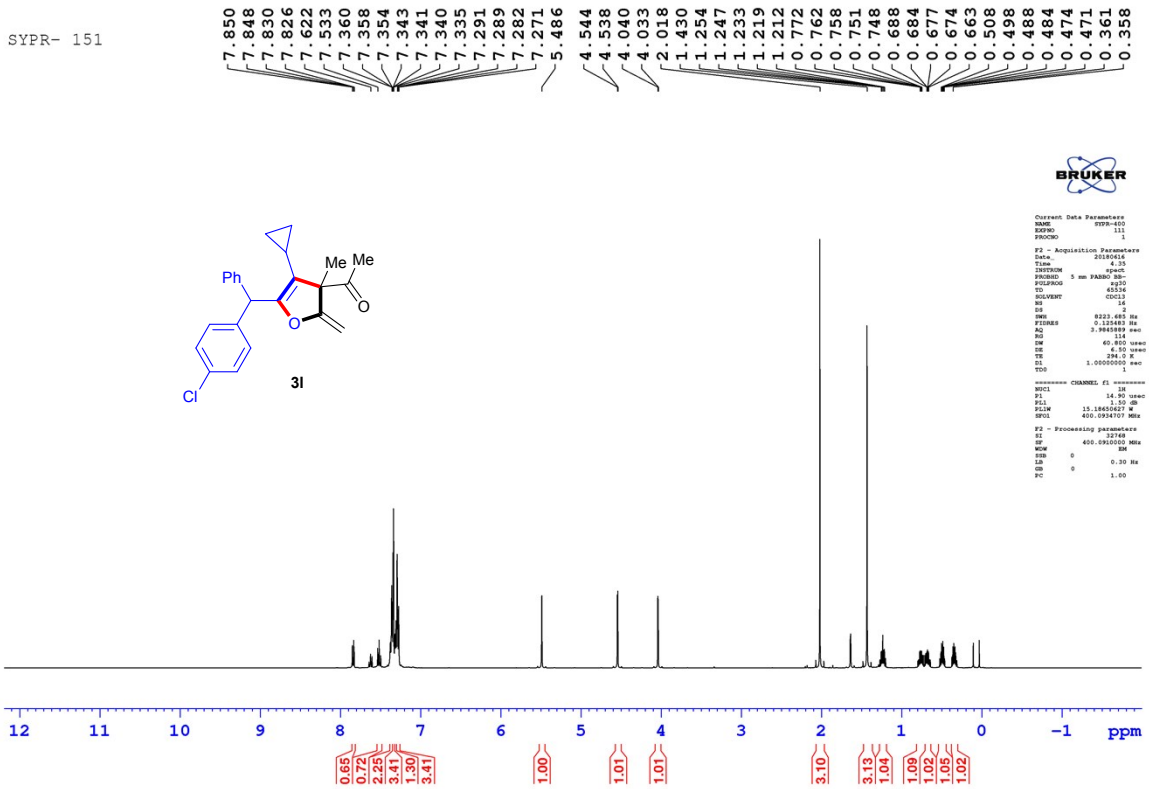

SYPR-168
7.400
7.385
7.345
7.340
7.339
7.332
7.327
7.324
7.318
7.315
7.312
7.308
7.301
7.290
7.284
7.228
7.225
7.211
7.194
7.152
7.147
7.135
7.131
7.072
7.019
7.002
6.943
6.939
6.927
6.923
5.286
5.265
4.653
4.648
4.163
2.360
2.143
1.401
0.001
Me
Me
Me
Ph
O
O
F
3j
BRUKER
10
9
8
7
6
5
4
3
2
1
0
ppm
1.19
3.39
1.41
2.27
2.03
0.91
1.02
2.17
0.95
1.11
1.24
3.35
3.00
3.17


SYPR-168
205.218
164.732
164.686
153.458
140.225
140.128
137.571
130.519
130.458
130.265
130.201
129.566
128.783
128.743
128.641
128.523
128.490
128.301
128.211
127.066
118.606
115.568
115.398
115.330
115.162
85.492
77.267
77.013
76.759
64.603
64.571
47.668
25.267
25.186
21.528
21.420
21.144
Me
Me
Me
Ph
O
O
F
3j
BRUKER
200
180
160
140
120
100
80
60
40
20
0
ppm

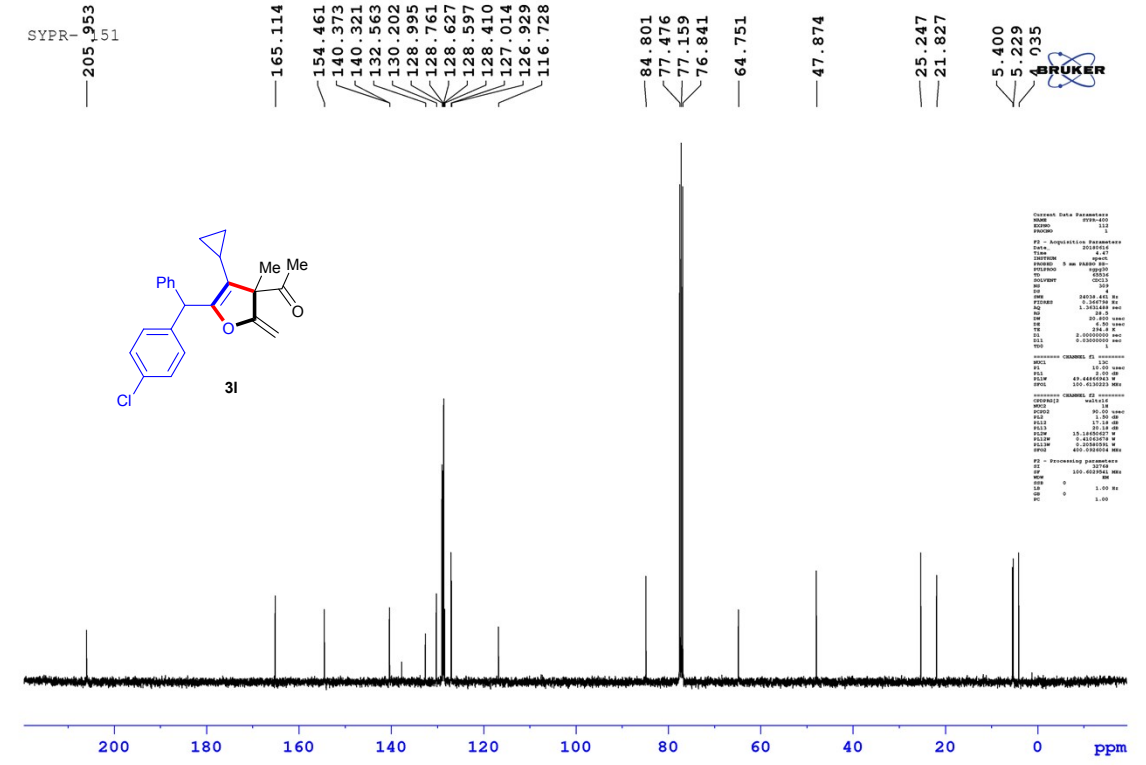

SYPR-182
7.894 7.883 7.876 7.865 7.811 7.809 7.795 7.792 7.625 7.535 7.519 7.402 7.389 7.349 7.343 7.335 7.329 7.317 7.314 7.284 7.232 7.230 7.215 7.207 7.198 7.190 7.173 7.151 7.145 7.134 7.129 7.076 7.020 7.003 6.956 6.953 6.940 6.936 5.314 5.294 4.650 4.645 4.168 4.163 2.610 2.156 2.153 1.637 1.615 1.611 1.410 1.408 1.390 1.375 0.970 0.957 0.955 0.942 0.940
BRUKER
Current Data Parameters
NAME SYPR-ASCEND-500
EXPNO 109
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180823
Time 15.10 h
INSTRUM spect
PROBHD Z125331_0034 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 4
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 97.13
DW 50.000 usec
DE 6.50 usec
TE 294.6 K
D1 1.00000000 sec
TD0 1
SFO1 500.3720898 MHz
NUC1 1H
P1 9.00 usec
PLW1 12.18000031 W
F2 - Processing parameters
SI 65536
SF 500.3690000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Ph
CH3
CH3
O
O
F
3k
12 11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.43 1.36 0.85 1.47 2.13 5.53 0.99 1.84 0.97 0.98 0.92 1.99 2.86 2.99 3.02 1.63 3.09

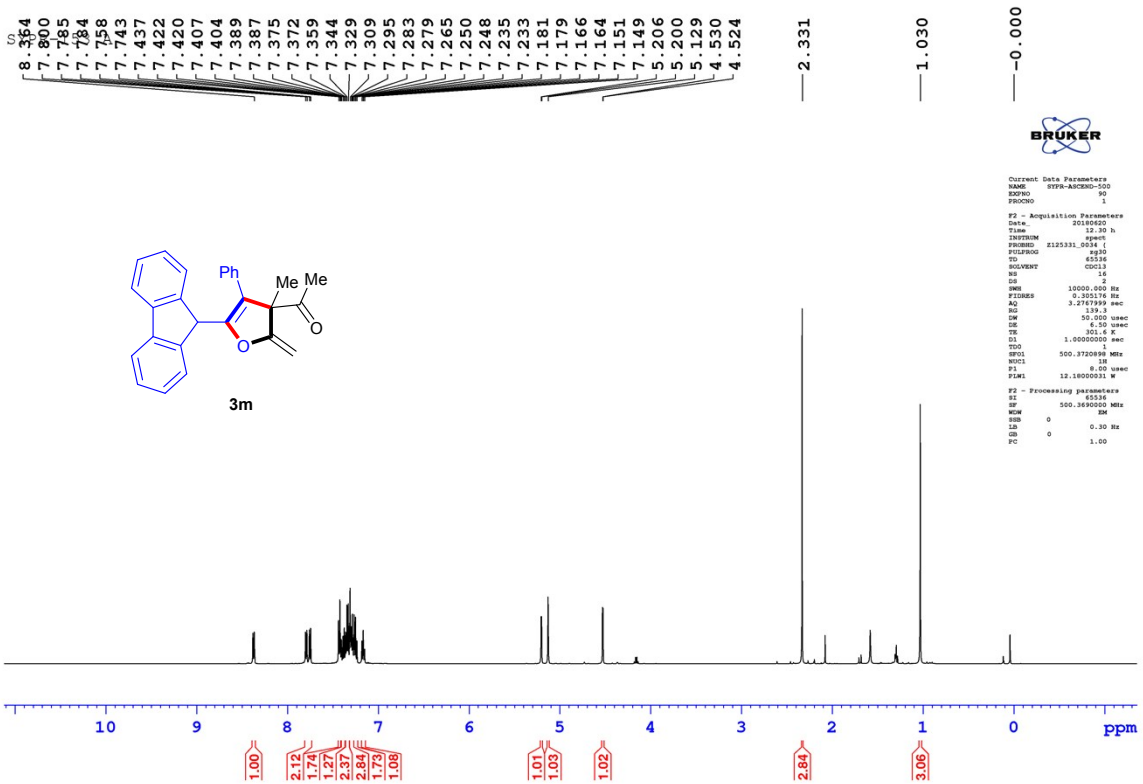

SYPR-182
205.464
195.429
164.786 164.742 153.616 153.505 142.637 142.601 140.347 140.250 137.625 130.012 128.997 128.872 128.489 118.780 115.676 115.524 115.502 115.437 115.268
85.600 77.406 77.152 76.897 64.710 64.679
47.774
35.434 33.539 25.419 25.333 22.522 21.680 21.573 14.072
BRUKER
Current Data Parameters
NAME SYPR-ASCEND-500
EXPNO 110
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180823
Time 15.53 h
INSTRUM spect
PROBHD Z125331_0034 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 811
DS 4
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 1.1010048 sec
RG 18.03
DW 16.800 usec
DE 6.50 usec
TE 297.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.8304669 MHz
NUC1 13C
P1 12.00 usec
PLW1 172.66999817 W
SFO2 500.3710015 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 12.18000031 W
PLW12 0.12180000 W
PLW13 0.06126500 W
F2 - Processing parameters
SI 32768
SF 125.8178715 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Ph
CH3
CH3
O
O
F
3k
200 180 160 140 120 100 80 60 40 20 0 ppm

SYPR- 151



SYPR-151

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7.437
7.422
7.420
7.407
7.404
7.389
7.387
7.375
7.372
7.359
7.344
7.329
7.309
7.295
7.283
7.279
7.265
7.250
7.248
7.235
7.233
7.181
7.179
7.166
7.164
7.151
7.149
5.206
5.200
5.129
4.530
4.524
2.331
1.030
0.000
Ph
Me
Me
O
O
3m
BRUKER
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
2.12
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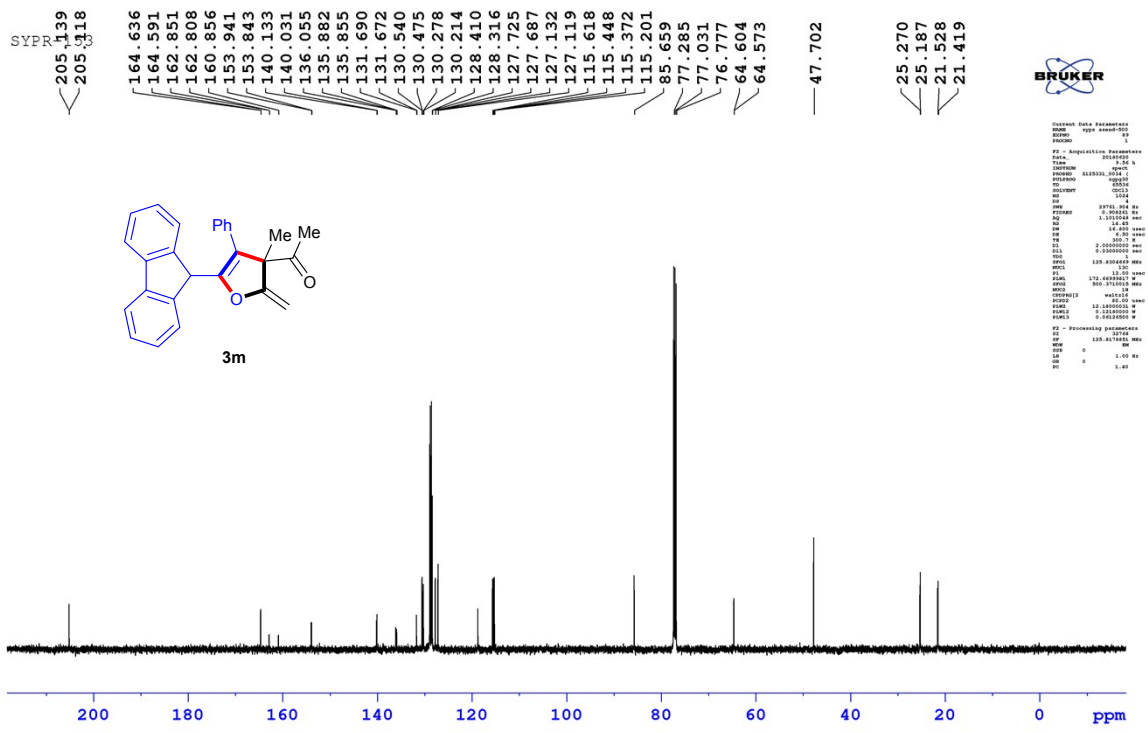
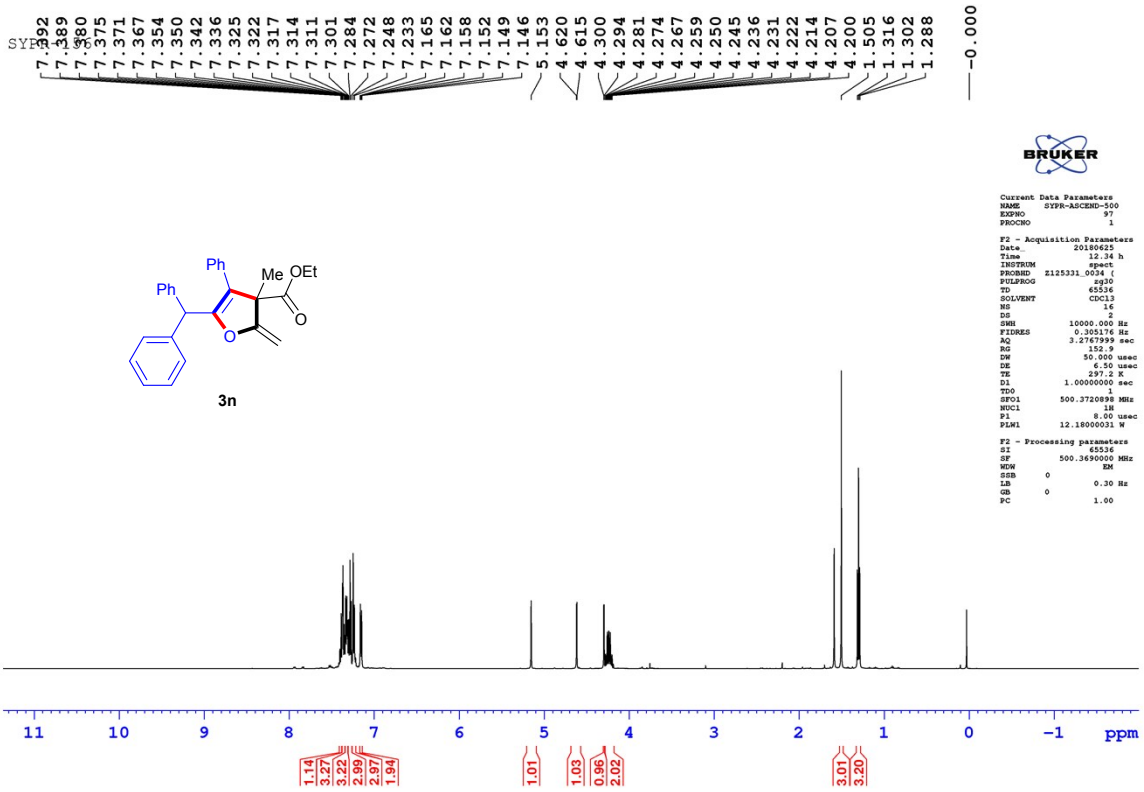
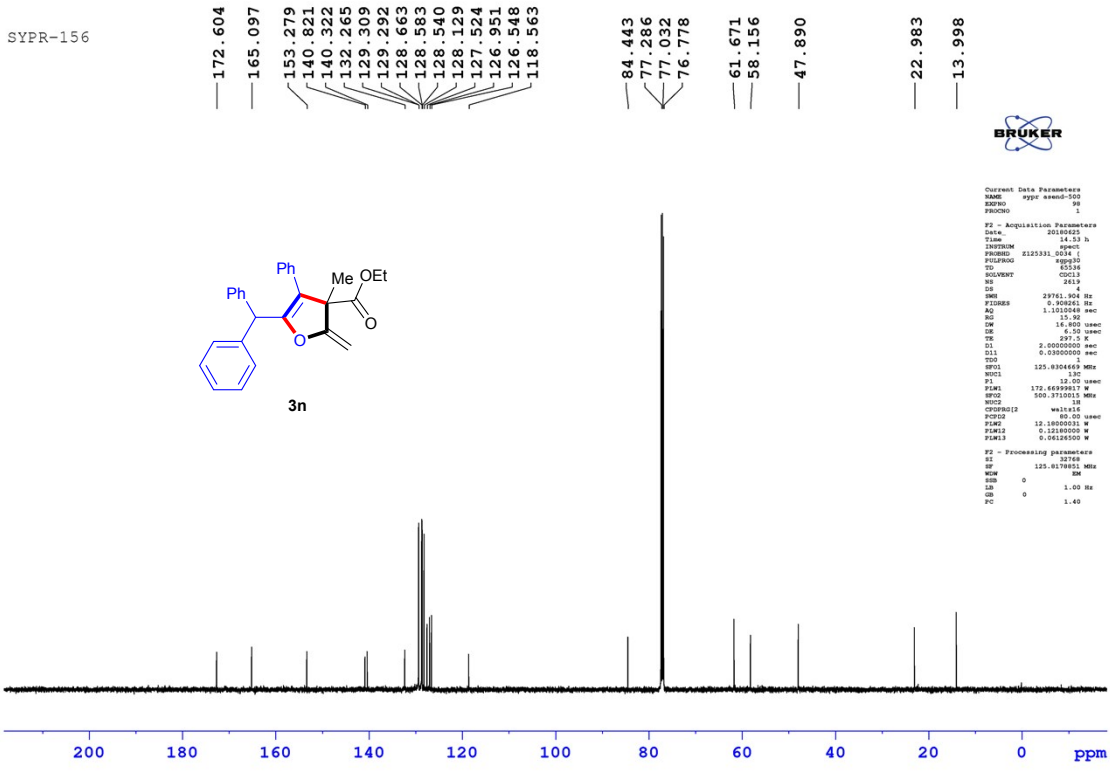
1.27
2.37
2.84
1.73
1.08
1.01
1.03
1.02
2.84
3.06


SYPR-1-13
205.139
205.118
164.636
164.591
162.851
162.808
160.856
153.941
153.843
140.133
140.031
136.055
135.882
135.855
131.690
131.672
130.540
130.475
130.278
130.214
128.410
128.316
127.725
127.687
127.132
127.119
115.618
115.448
115.372
115.201
85.659
77.285
77.031
76.777
64.604
64.573
47.702
25.270
25.187
21.528
21.419
Ph
Me
Me
O
O
3m
BRUKER
200
180
160
140
120
100
80
60
40
20
0
ppm


SYPR-156
7.392
7.389
7.380
7.375
7.371
7.367
7.354
7.350
7.342
7.336
7.325
7.322
7.317
7.314
7.311
7.301
7.284
7.272
7.248
7.233
7.165
7.162
7.158
7.152
7.149
7.146
5.153
4.620
4.615
4.300
4.294
4.281
4.274
4.267
4.259
4.250
4.245
4.236
4.231
4.222
4.214
4.207
4.200
1.505
1.316
1.302
1.288
0.000
Ph
Ph
Me
OEt
O
O
3n
BRUKER
Current Data Parameters
NAME SYPR-ASCEND-500
EXPNO 97
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180625
Time 12.34 h
INSTRUM spect
PROBHD Z125331_0034 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 152.9
DW 50.000 usec
DE 6.50 usec
TE 297.2 K
D1 1.00000000 sec
TD0 1
SFO1 500.3720898 MHz
NUC1 1H
P1 8.00 usec
PLW1 12.18000031 W
F2 - Processing parameters
SI 65536
SF 500.3690000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
11
10
9
8
7
6
5
4
3
2
1
0
−1
ppm
1.14
3.27
3.22
2.99
2.97
1.94
1.01
1.03
0.96
2.02
3.01
3.20


SYPR-156
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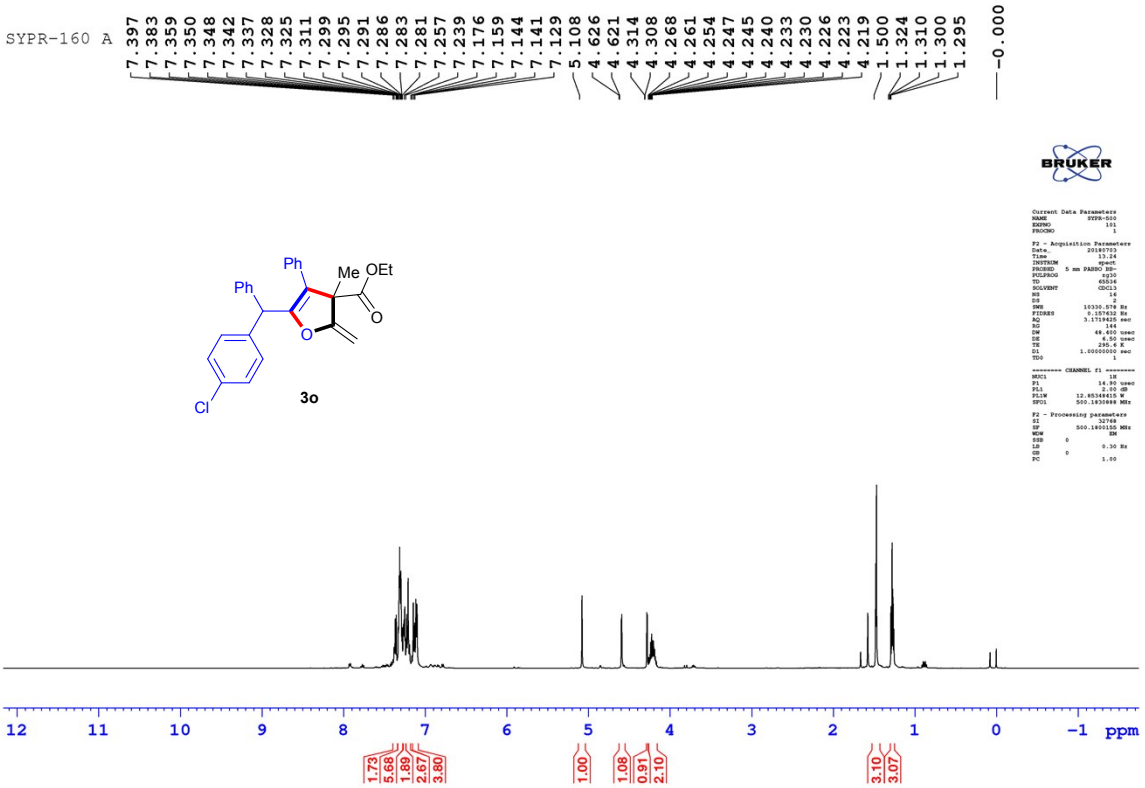
153.279
140.821
140.322
132.265
129.309
129.292
128.663
128.583
128.540
128.129
127.524
126.951
126.548
118.563
84.443
77.286
77.032
76.778
61.671
58.156
47.890
22.983
13.998
Ph
Ph
Me
OEt
O
O
3n
BRUKER
Current Data Parameters
NAME sypr asend-500
EXPNO 90
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180625
Time 14.53 h
INSTRUM spect
PROBHD Z125331_0034 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2619
DS 4
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 1.1010048 sec
RG 15.92
DW 16.800 usec
DE 6.50 usec
TE 297.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.0304669 MHz
NUC1 13C
P1 12.00 usec
PLW1 172.66999817 W
SFO2 500.3710015 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 12.18000031 W
PLW12 0.12160000 W
PLW13 0.06126500 W
F2 - Processing parameters
SI 32768
SF 125.0178851 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm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7.383
7.359
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7.342
7.337
7.328
7.325
7.311
7.299
7.295
7.291
7.286
7.283
7.281
7.257
7.239
7.176
7.159
7.144
7.141
7.129
5.108
4.626
4.621
4.314
4.308
4.268
4.261
4.254
4.247
4.245
4.240
4.233
4.230
4.226
4.223
4.219
1.500
1.324
1.310
1.300
1.295
0.000
Ph
Ph
Me
OEt
O
O
Cl
3o
BRUKER
12
11
10
9
8
7
6
5
4
3
2
1
0
−1
ppm
1.73
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1.89
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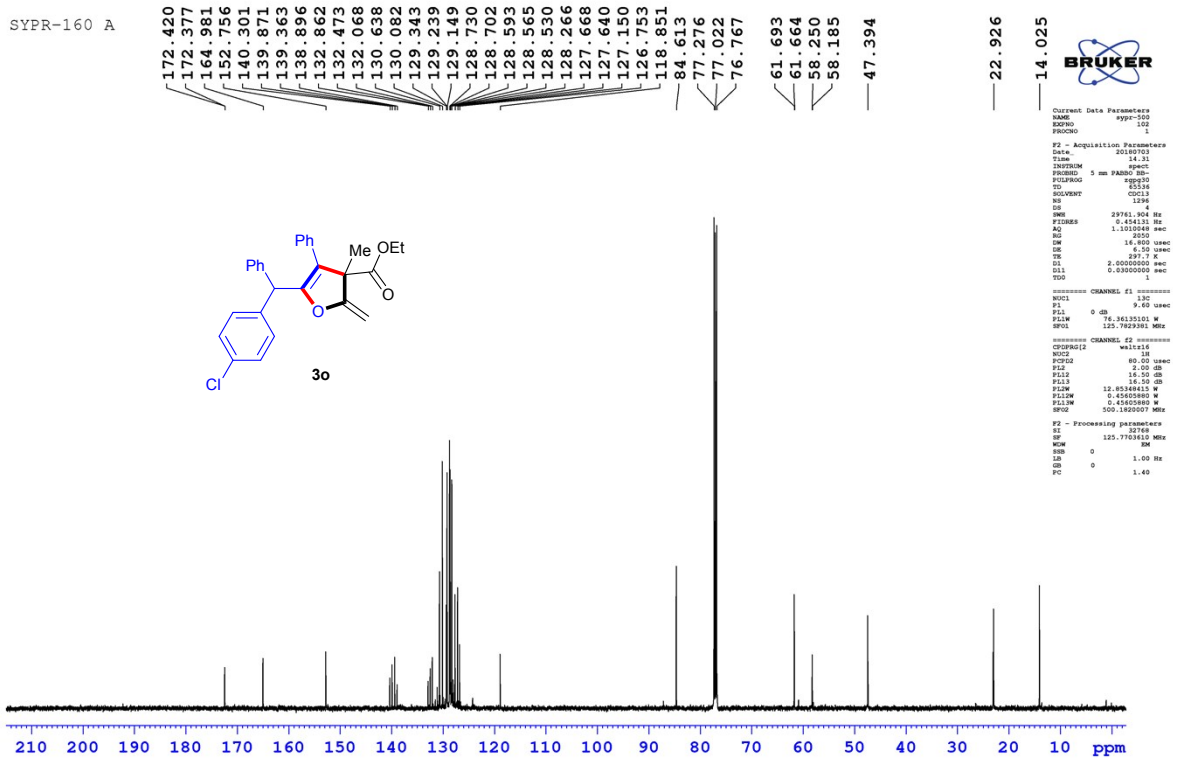
3.80
1.00
1.08
0.91
2.10
3.10
3.07


SYPR-160 A
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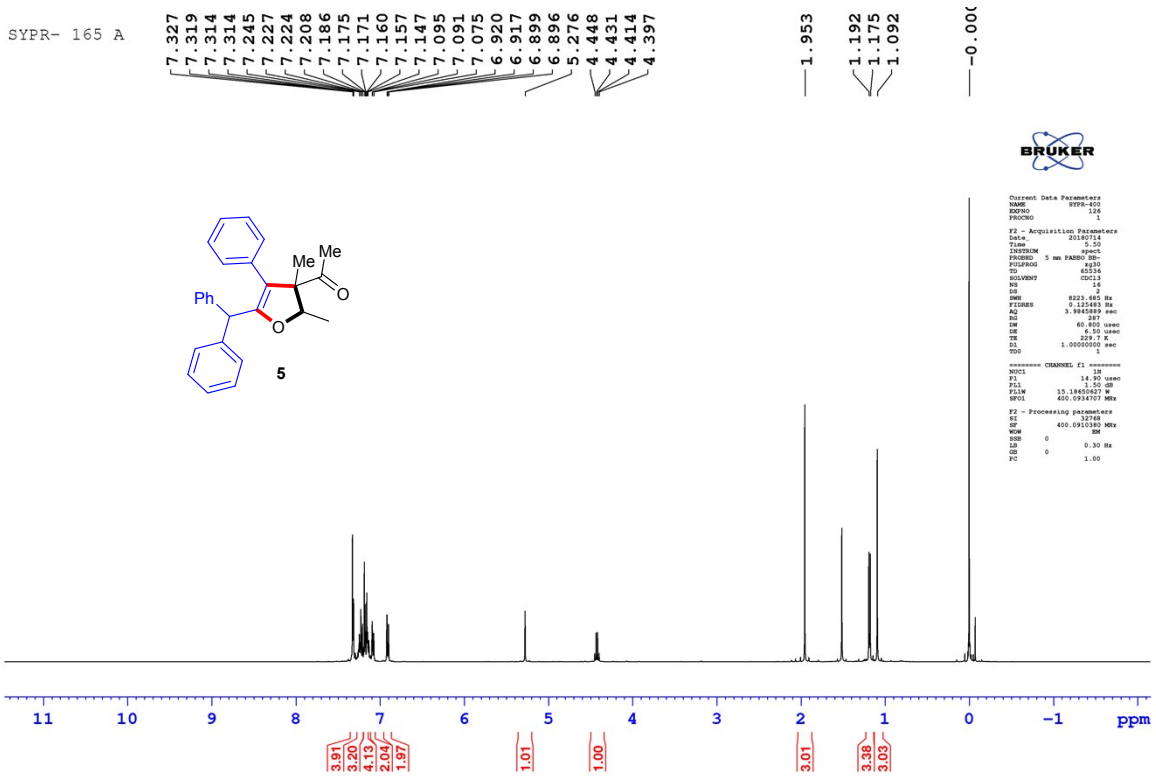
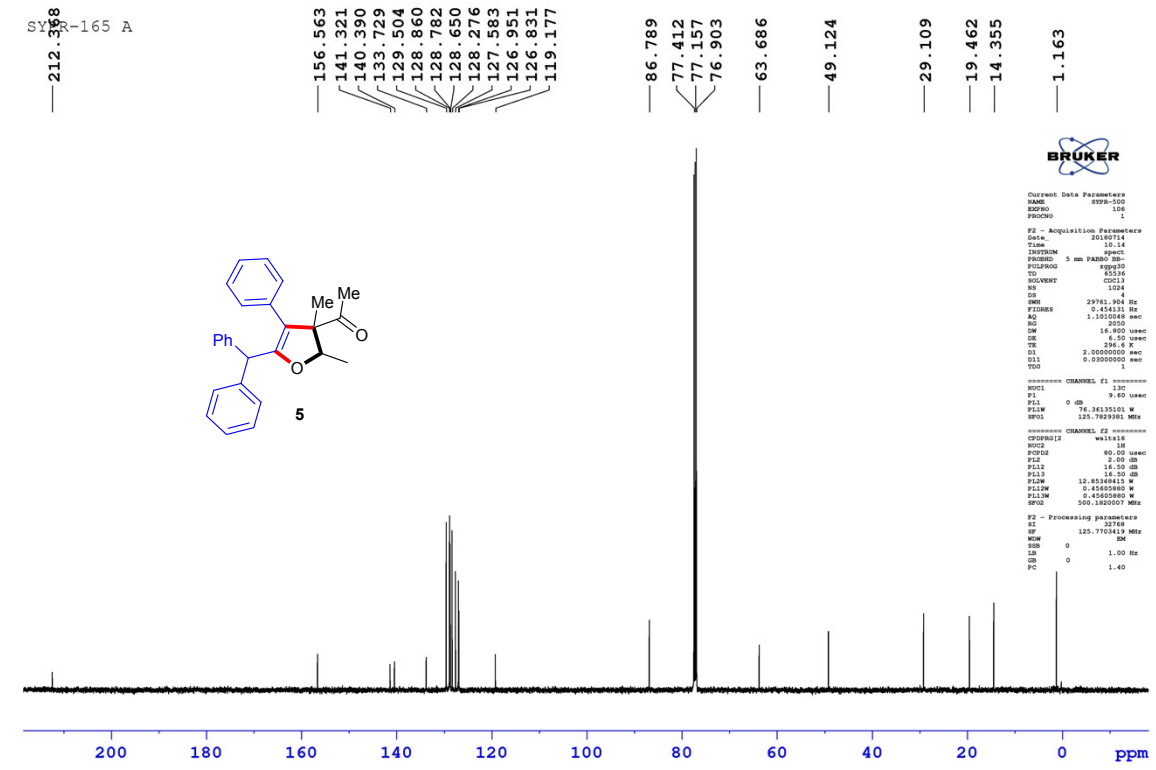
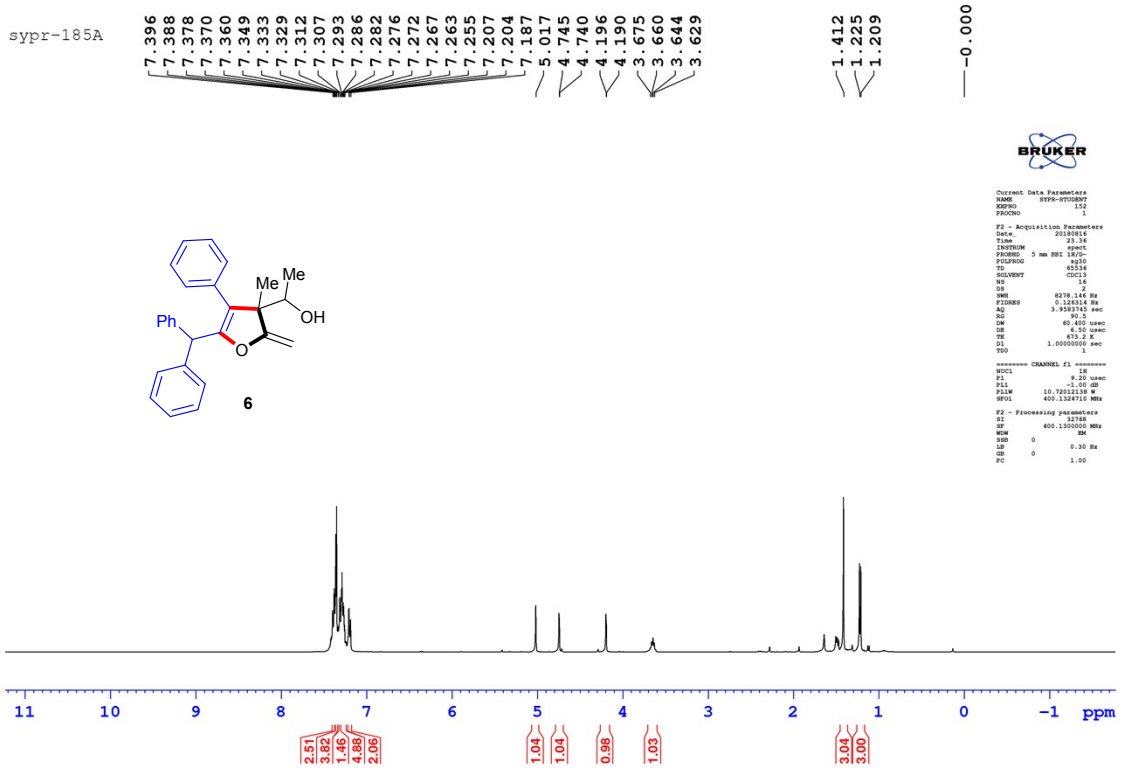
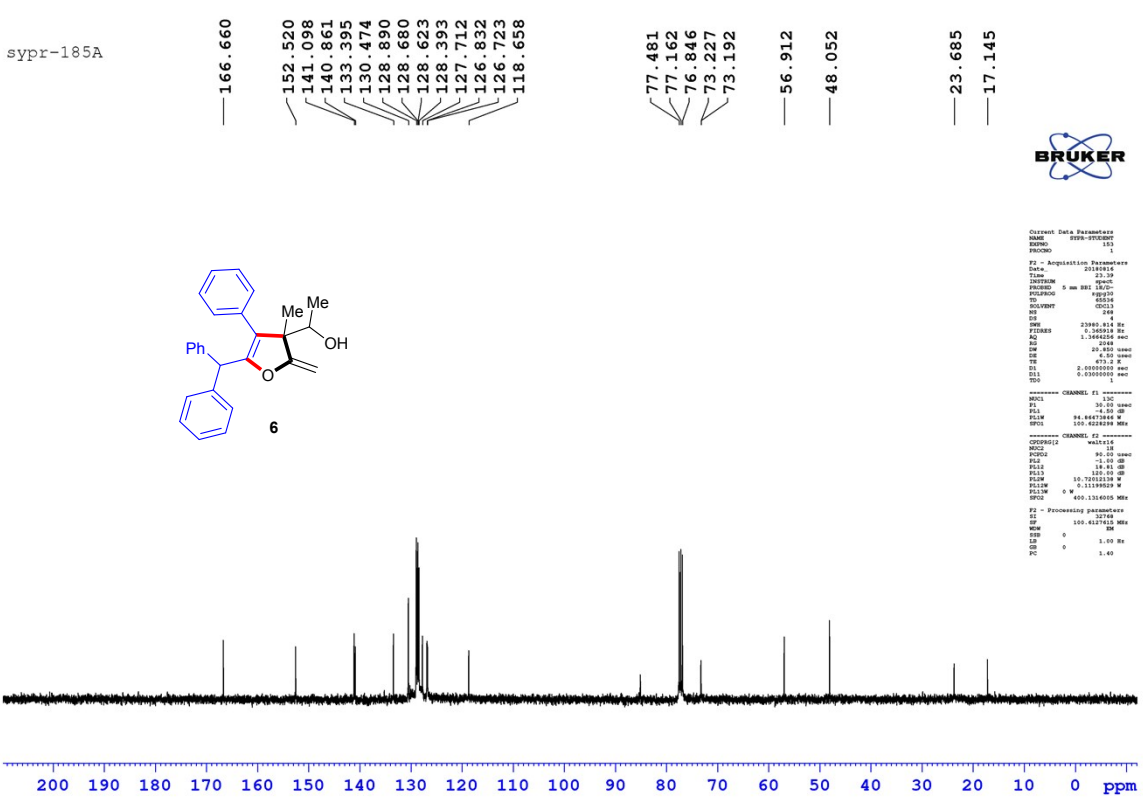
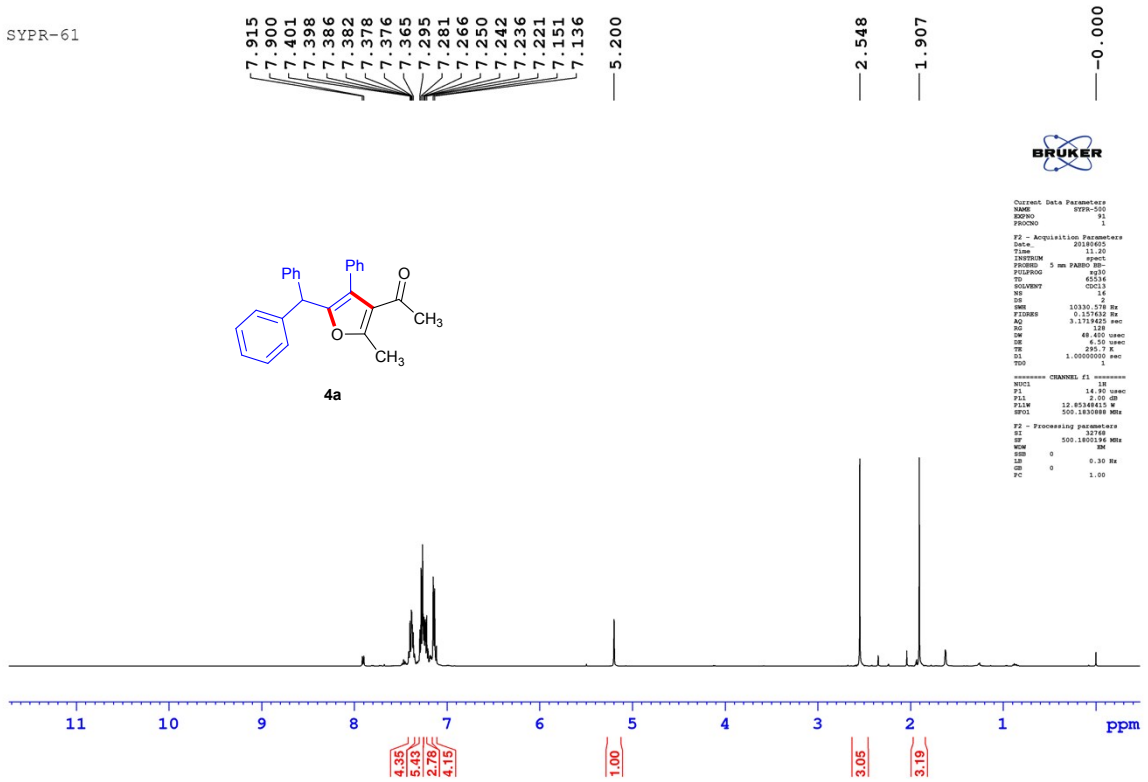
172.377
164.981
152.756
140.301
139.871
139.363
138.896
132.862
132.473
132.068
130.638
130.082
129.343
129.239
129.149
128.730
128.702
128.593
128.565
128.530
128.266
127.668
127.640
127.150
126.753
118.851
84.613
77.276
77.022
76.767
61.693
61.664
58.250
58.185
47.394
22.926
14.025
Ph
Ph
Me
OEt
O
O
Cl
3o
BRUKER
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

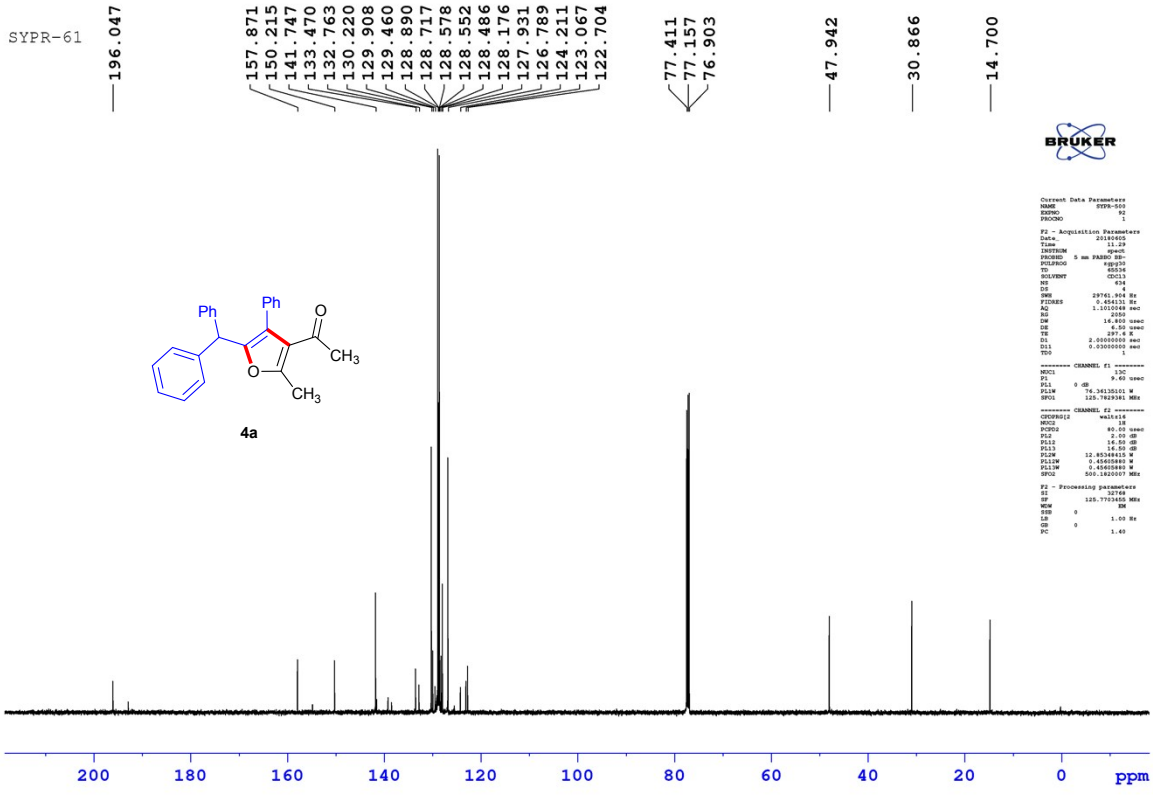

SYPR- 165 A
7.327
7.319
7.314
7.314
7.245
7.227
7.224
7.208
7.186
7.175
7.171
7.160
7.157
7.147
7.095
7.091
7.075
6.920
6.917
6.899
6.896
5.276
4.448
4.431
4.414
4.397
1.953
1.192
1.175
1.092
-0.00
BRUKER
Me
Me
Ph
O
O
5
11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
3.91
3.20
4.13
2.04
1.97
1.01
1.00
3.01
3.38
3.03


SYPR-165 A
212.368
156.563
141.321
140.390
133.729
129.504
128.860
128.782
128.650
128.276
127.583
126.951
126.831
119.177
86.789
77.412
77.157
76.903
63.686
49.124
29.109
19.462
14.355
1.163
BRUKER
Me
Me
Ph
O
O
5
200
180
160
140
120
100
80
60
40
20
0
ppm


sypr-185A
7.396
7.388
7.378
7.370
7.360
7.349
7.333
7.329
7.312
7.307
7.293
7.286
7.282
7.276
7.272
7.267
7.263
7.255
7.207
7.204
7.187
5.017
4.745
4.740
4.196
4.190
3.675
3.660
3.644
3.629
1.412
1.225
1.209
0.000
Me
Me
OH
Ph
O
6
11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
2.51
3.82
1.46
4.88
2.06
1.04
1.04
0.98
1.03
3.04
3.00

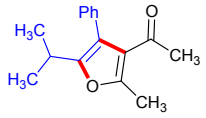

sypr-185A
166.660
152.520
141.098
140.861
133.395
130.474
128.890
128.680
128.623
128.393
127.712
126.832
126.723
118.658
77.481
77.162
76.846
73.227
73.192
56.912
48.052
23.685
17.145
Me
Me
OH
Ph
O
6
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


SYPR-61
7.915
7.900
7.401
7.398
7.386
7.382
7.378
7.376
7.365
7.295
7.281
7.266
7.250
7.242
7.236
7.221
7.151
7.136
5.200
2.548
1.907
0.000
BRUKER
Ph
Ph
O
CH3
O
CH3
4a
11
10
9
8
7
6
5
4
3
2
1
ppm
4.35
5.43
2.78
4.15
1.00
3.05
3.19

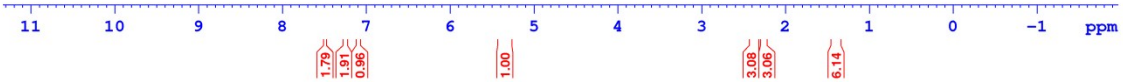

SYPR-61
196.047
157.871
150.215
141.747
133.470
132.763
130.220
129.908
129.460
128.890
128.717
128.578
128.552
128.486
128.176
127.931
126.789
124.211
123.067
122.704
77.411
77.157
76.903
47.942
30.866
14.700
BRUKER
Ph
Ph
O
CH3
O
CH3
4a
200
180
160
140
120
100
80
60
40
20
0
ppm

SYPR- 135 A

7.503 7.500 7.496 7.482 7.480 7.271 7.252 7.236 7.232 7.184 7.116 7.097 7.079 5.366 2.357 2.274 1.409 0.000



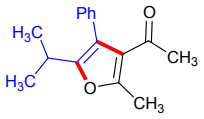
4b



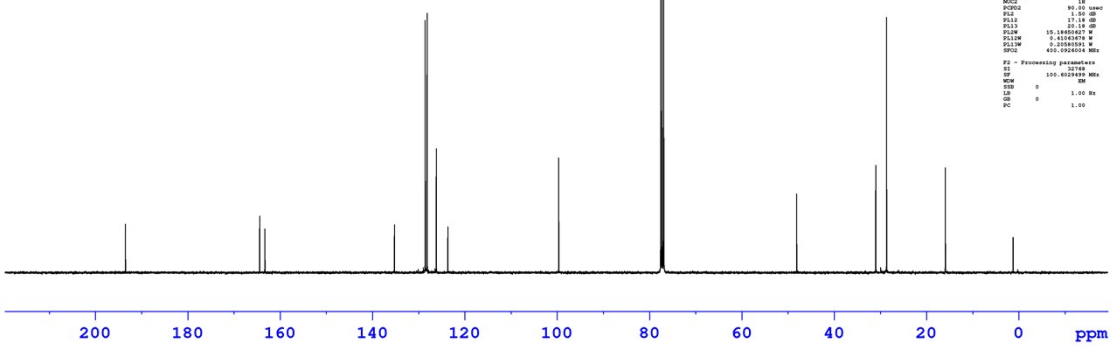
1.79 1.91 0.96 1.00 3.08 3.06 6.14

SYPR- 135 A

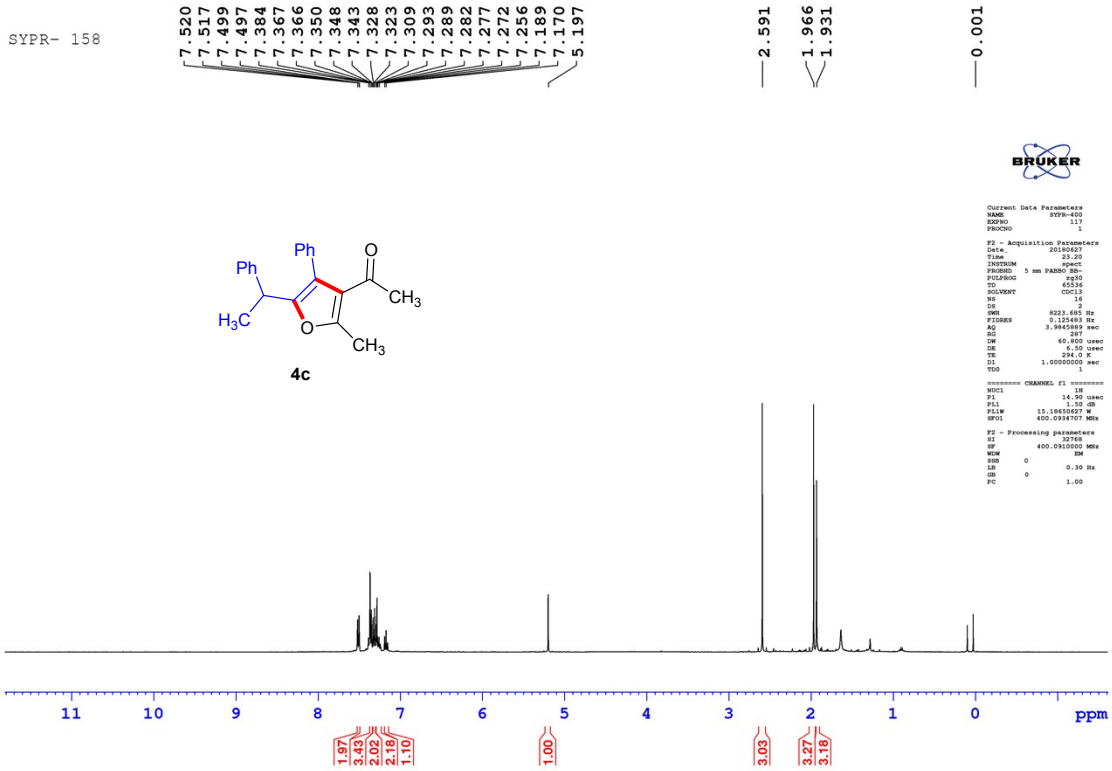
193.452 164.350 163.242 135.163 128.509 128.085 126.102 123.586 99.580 77.471 77.153 76.836 48.028 30.892 28.557 15.840 1.150



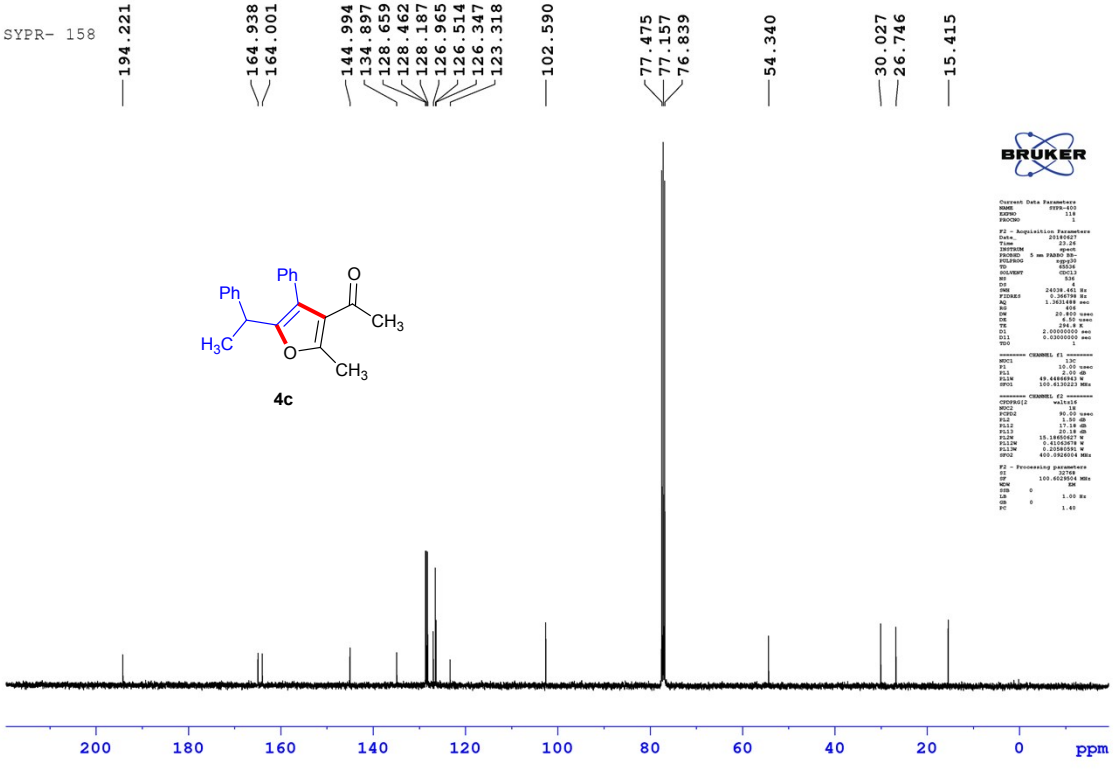
4b

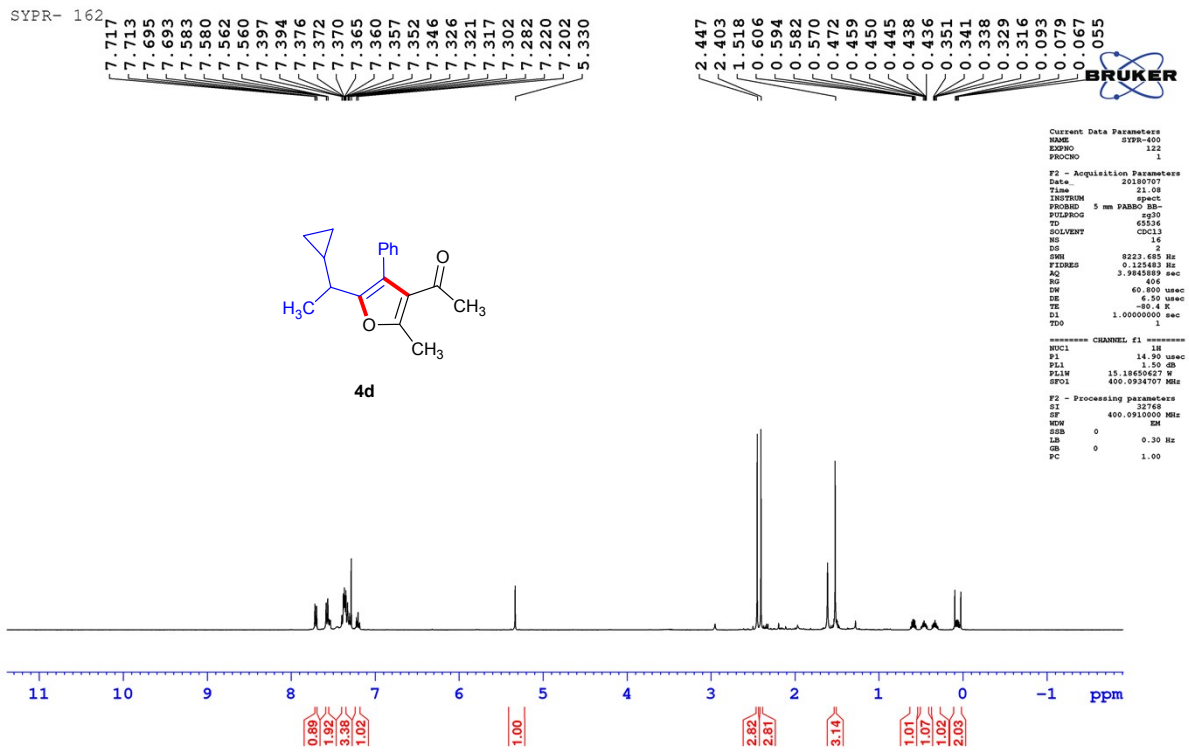


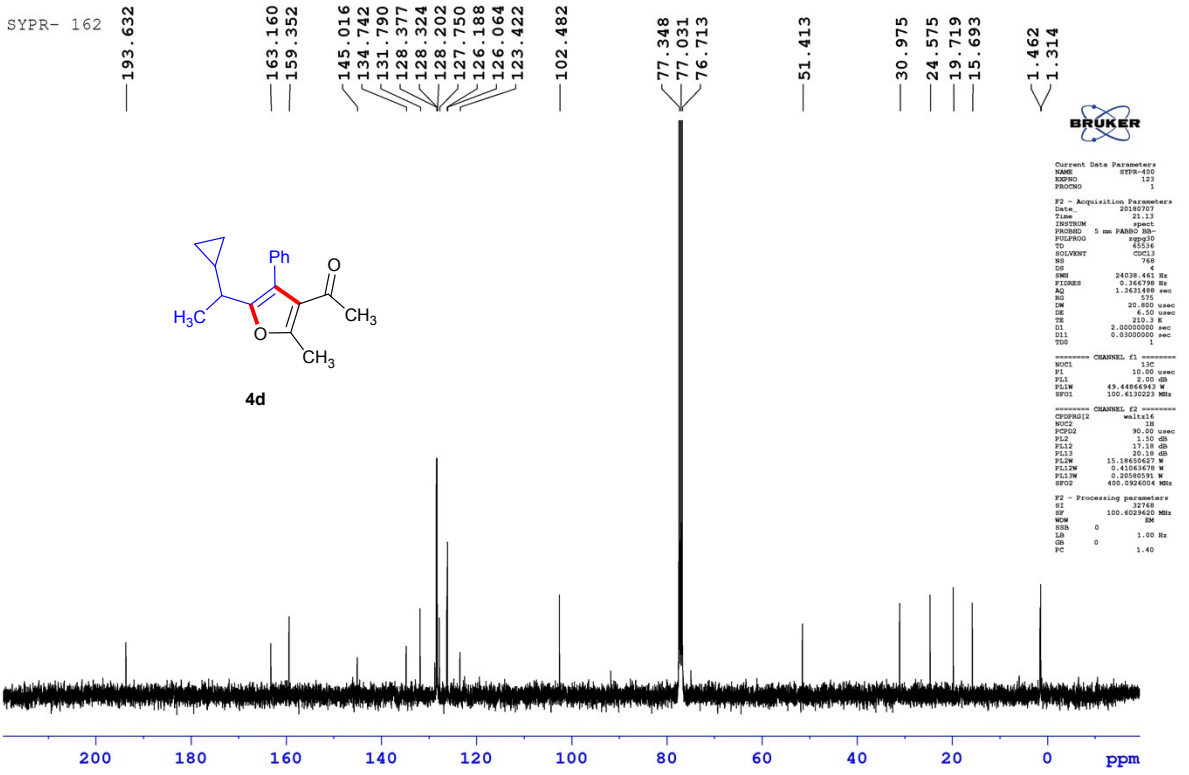
SYPR- 158

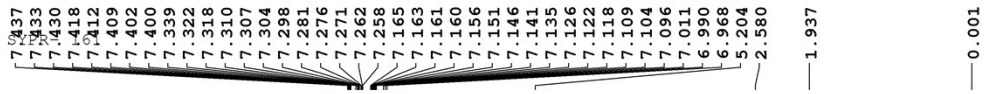


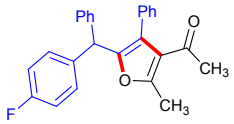
SYPR- 158



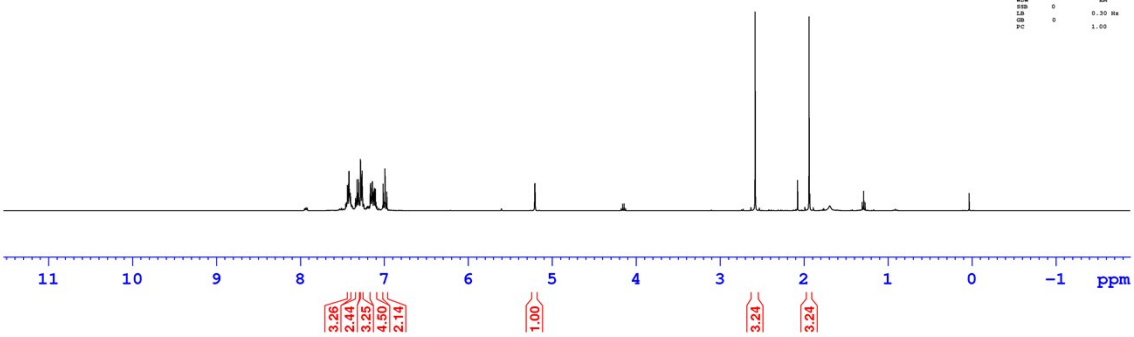

SYPR- 162
7.717
7.713
7.695
7.693
7.583
7.580
7.562
7.560
7.397
7.394
7.376
7.372
7.370
7.365
7.360
7.357
7.352
7.346
7.326
7.321
7.317
7.302
7.282
7.220
7.202
5.330
2.447
2.403
1.518
0.606
0.594
0.582
0.570
0.472
0.459
0.450
0.445
0.438
0.436
0.351
0.341
0.338
0.329
0.316
0.093
0.079
0.067
0.055
BRUKER
Current Data Parameters
NAME SYPR-400
EXPNO 122
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180707
Time 21.08
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9845889 sec
RG 406
DW 60.800 usec
DE 6.50 usec
TE ~80.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.90 usec
PL1 1.50 dB
PL1W 15.18650627 W
SFO1 400.0934707 MHz
F2 - Processing parameters
SI 32768
SF 400.0910000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Ph
O
H3C
CH3
O
CH3
4d
11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
0.89
1.92
3.38
1.02
1.00
2.82
2.81
3.14
1.01
1.07
1.02
2.03


SYPR- 162
193.632
163.160
159.352
145.016
134.742
131.790
128.377
128.324
128.202
127.750
126.188
126.064
123.422
102.482
77.348
77.031
76.713
51.413
30.975
24.575
19.719
15.693
1.462
1.314
BRUKER
Current Data Parameters
NAME SYPR-400
EXPNO 123
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180707
Time 21.13
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 768
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 575
DW 20.800 usec
DE 6.50 usec
TE 210.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 2.00 dB
PL1W 49.44866943 W
SFO1 100.6130223 MHz
======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 1.50 dB
PL12 17.18 dB
PL13 20.18 dB
PL2W 15.18650627 W
PL12W 0.41063678 W
PL13W 0.20580591 W
SFO2 400.0926004 MHz
F2 - Processing parameters
SI 32768
SF 100.6029620 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Ph
O
H3C
CH3
O
CH3
4d
200
180
160
140
120
100
80
60
40
20
0
ppm

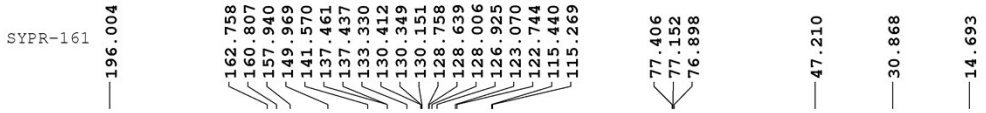


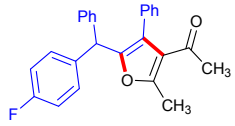


4e

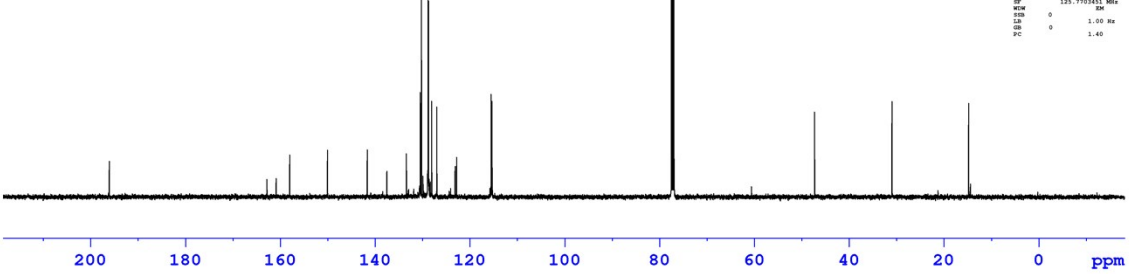


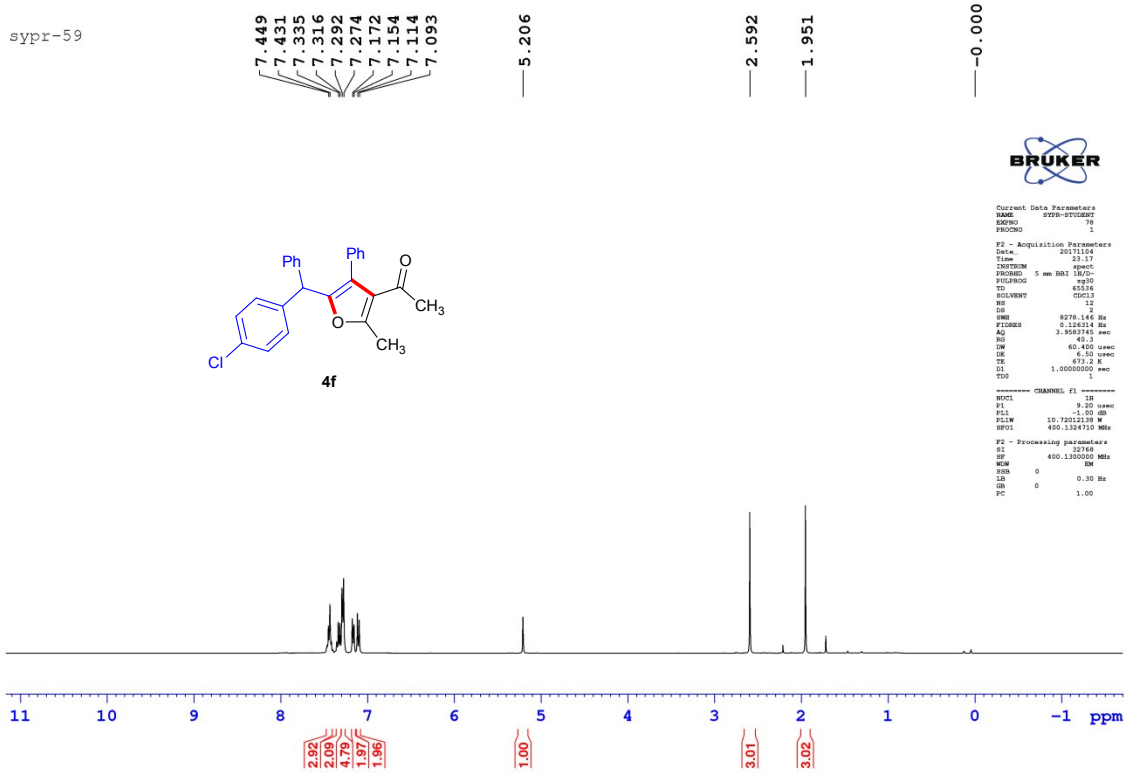
SYPR-161

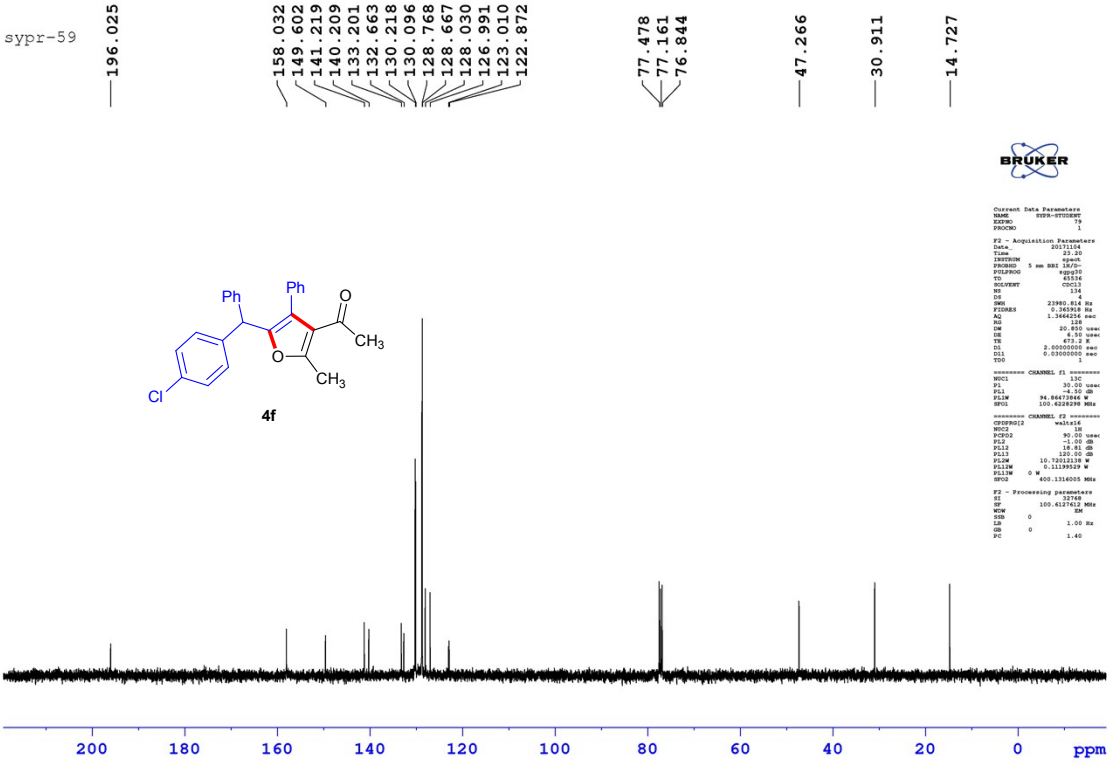


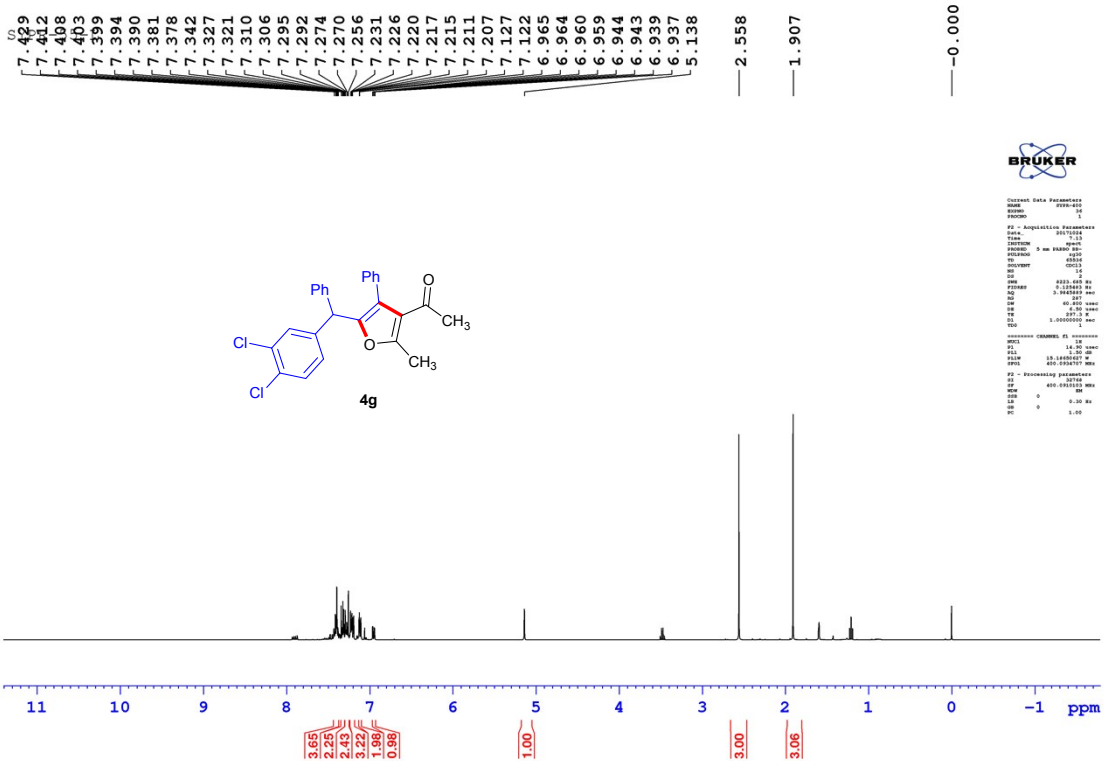


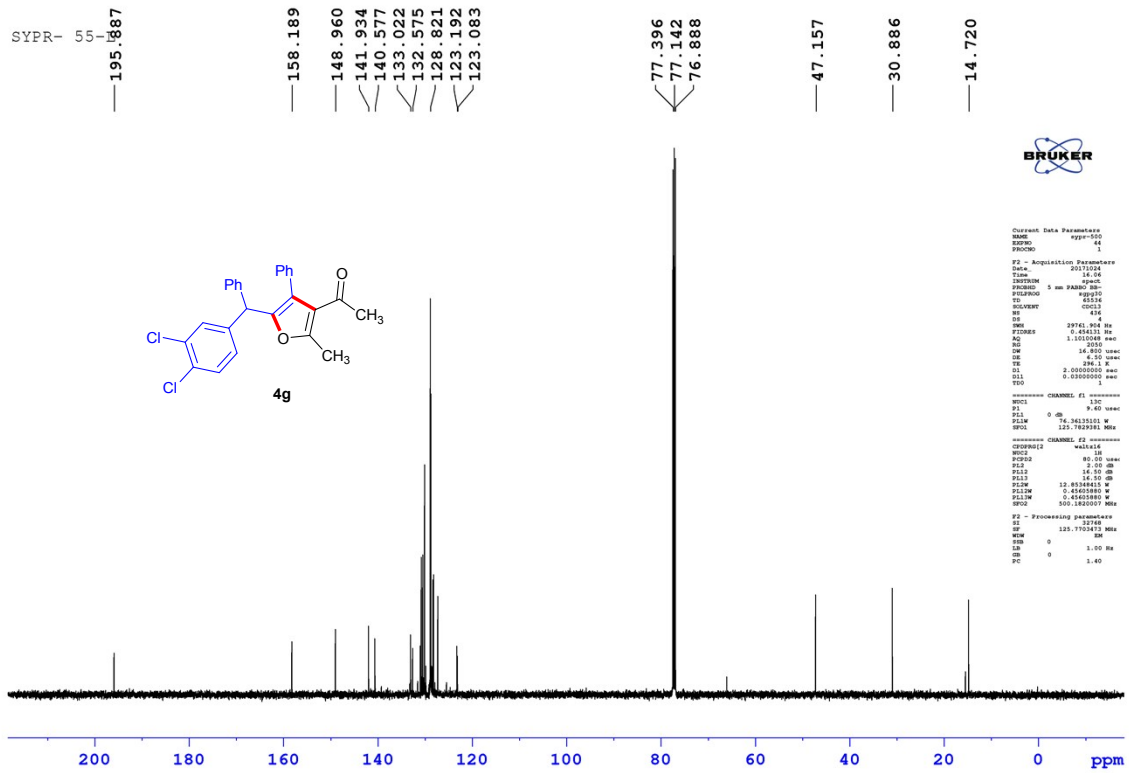
4e

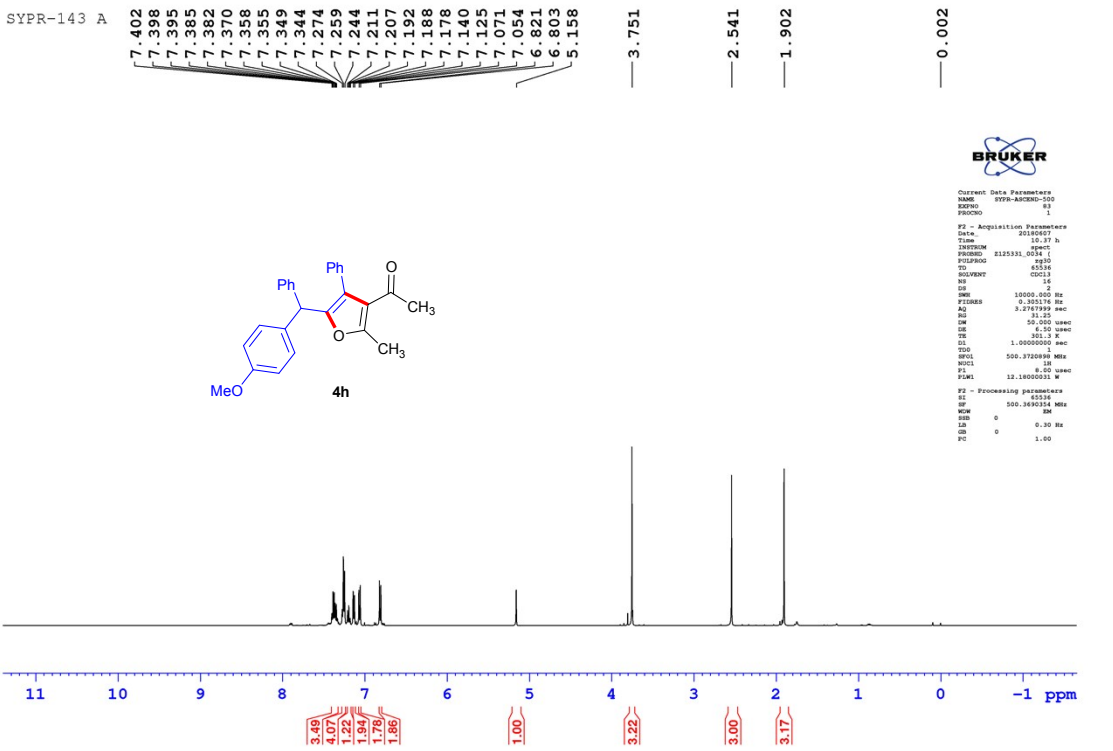


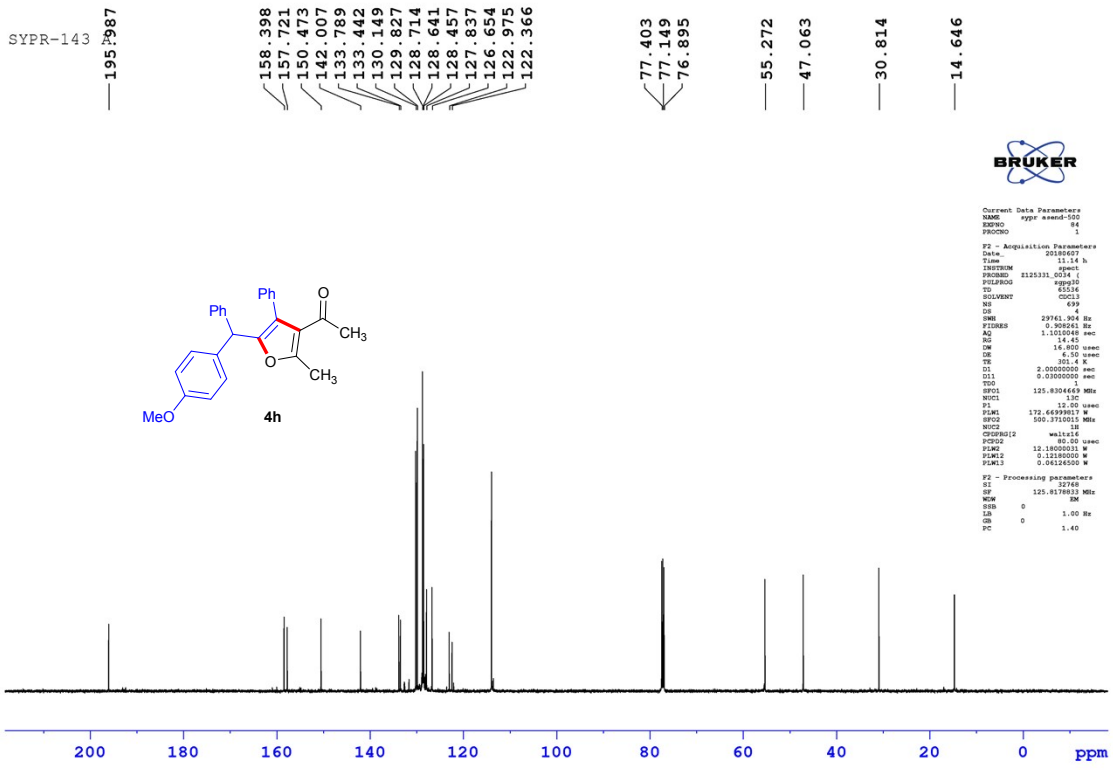

sypr-59
7.449
7.431
7.335
7.316
7.292
7.274
7.172
7.154
7.114
7.093
5.206
2.592
1.951
0.000
BRUKER
Ph
Ph
O
CH3
O
CH3
Cl
4f
11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
2.92
2.09
4.79
1.97
1.96
1.00
3.01
3.02

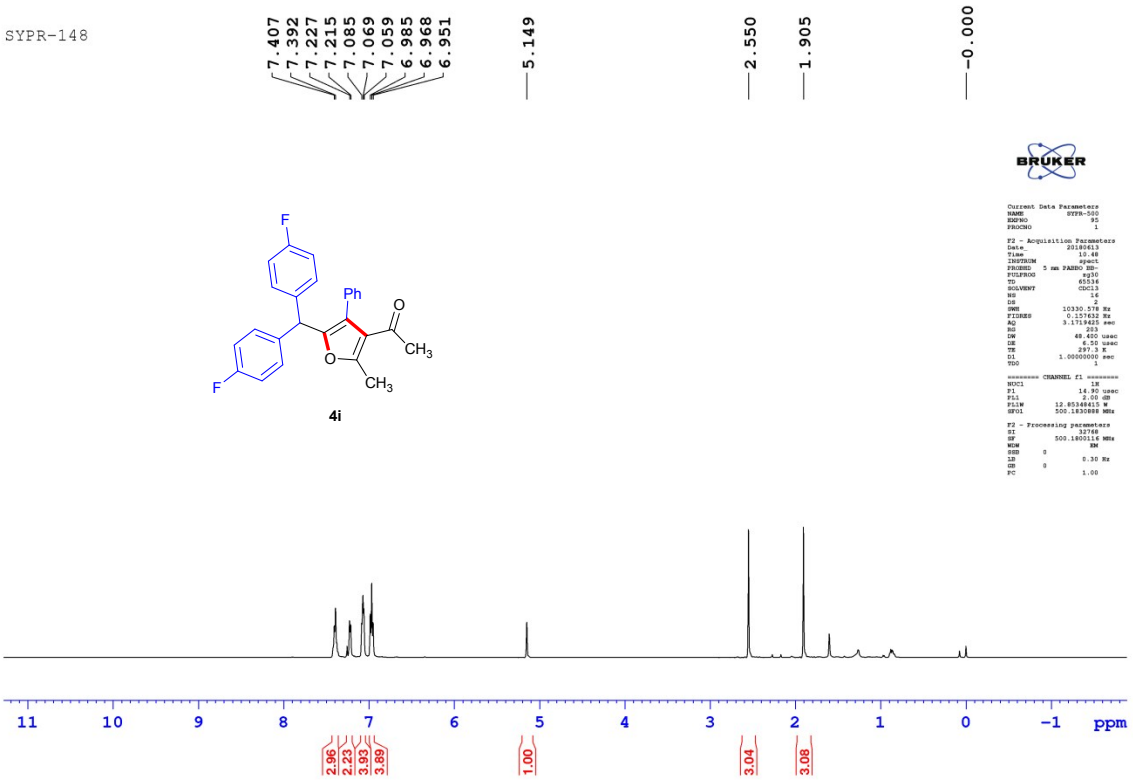

sypr-59
196.025
158.032
149.602
141.219
140.209
133.201
132.663
130.218
130.096
128.768
128.667
128.030
126.991
123.010
122.872
77.478
77.161
76.844
47.266
30.911
14.727
BRUKER
Ph
Ph
O
CH3
O
CH3
Cl
4f
200
180
160
140
120
100
80
60
40
20
0
ppm

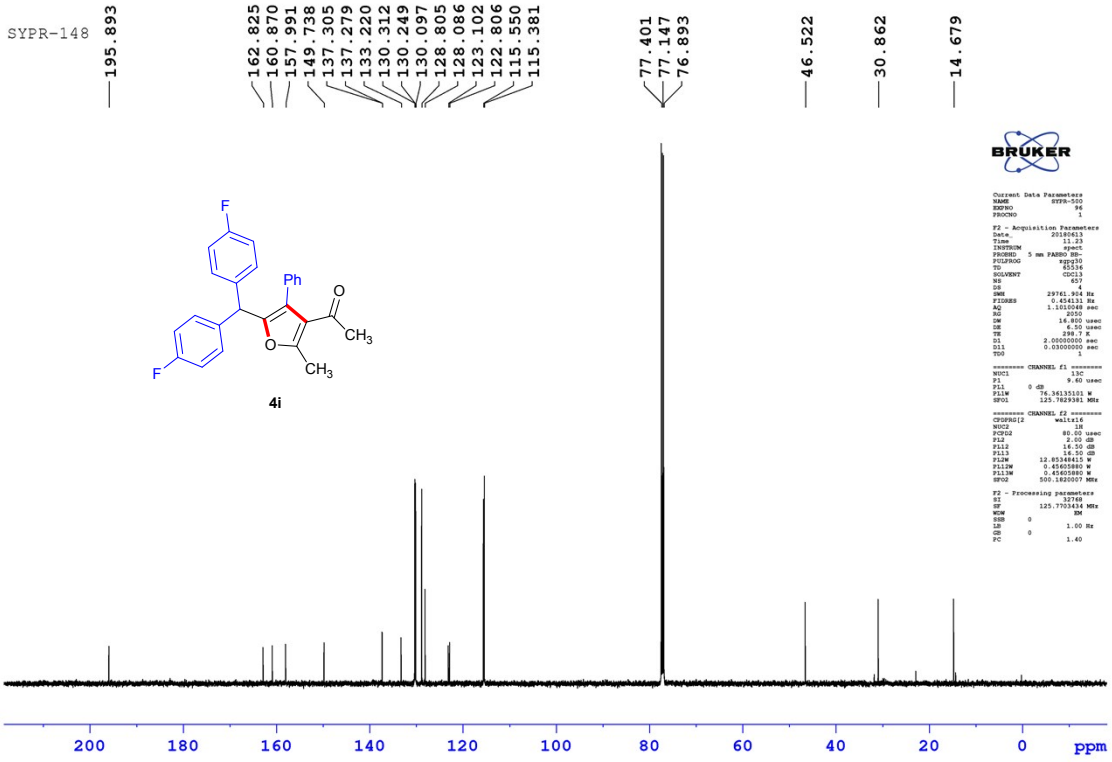

7.429
7.422
7.408
7.403
7.399
7.394
7.390
7.381
7.378
7.342
7.327
7.321
7.310
7.306
7.295
7.292
7.274
7.270
7.256
7.231
7.226
7.220
7.217
7.215
7.211
7.207
7.127
7.122
6.965
6.964
6.960
6.959
6.944
6.943
6.939
6.937
5.138
2.558
1.907
0.000
Ph
Ph
O
CH3
CH3
Cl
Cl
O
4g
11 10 9 8 7 6 5 4 3 2 1 0 -1 ppm
3.65
2.25
2.43
2.22
1.90
0.98
1.00
3.00
3.06

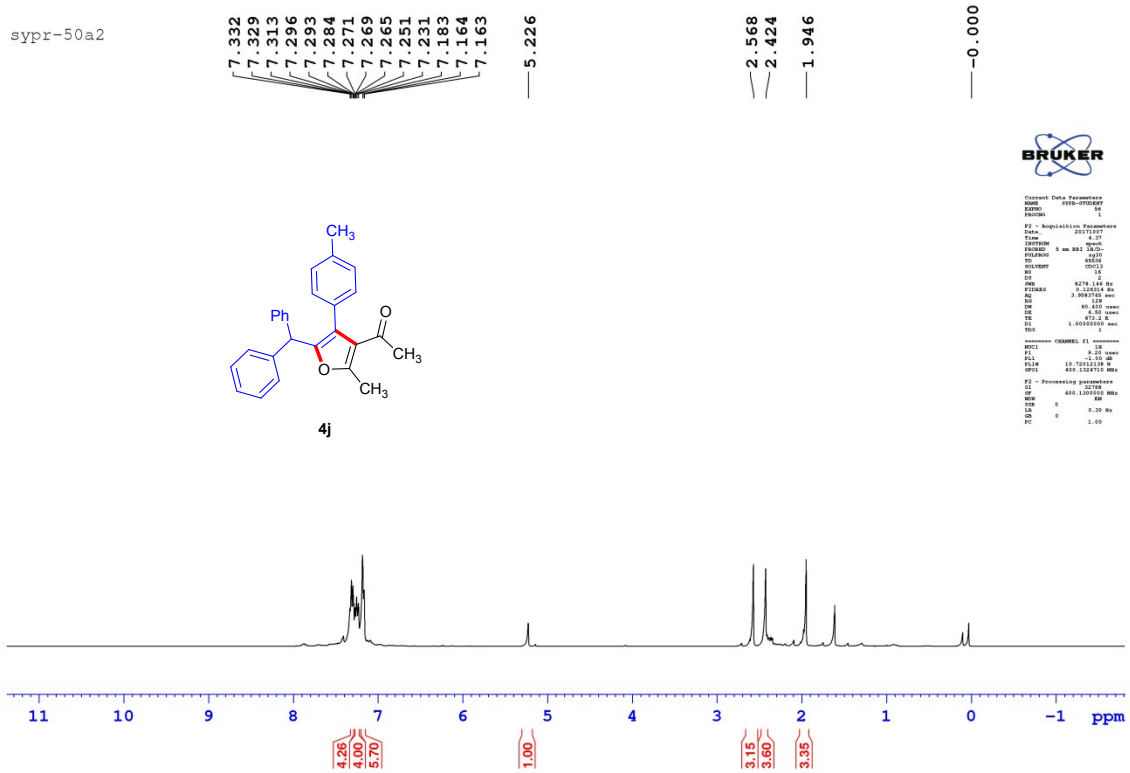

SYPR- 55-1
195.887
158.189
148.960
141.934
140.577
133.022
132.575
128.821
123.192
123.083
77.396
77.142
76.888
47.157
30.886
14.720
Ph
Ph
O
CH3
CH3
Cl
Cl
O
4g
200 180 160 140 120 100 80 60 40 20 0 ppm

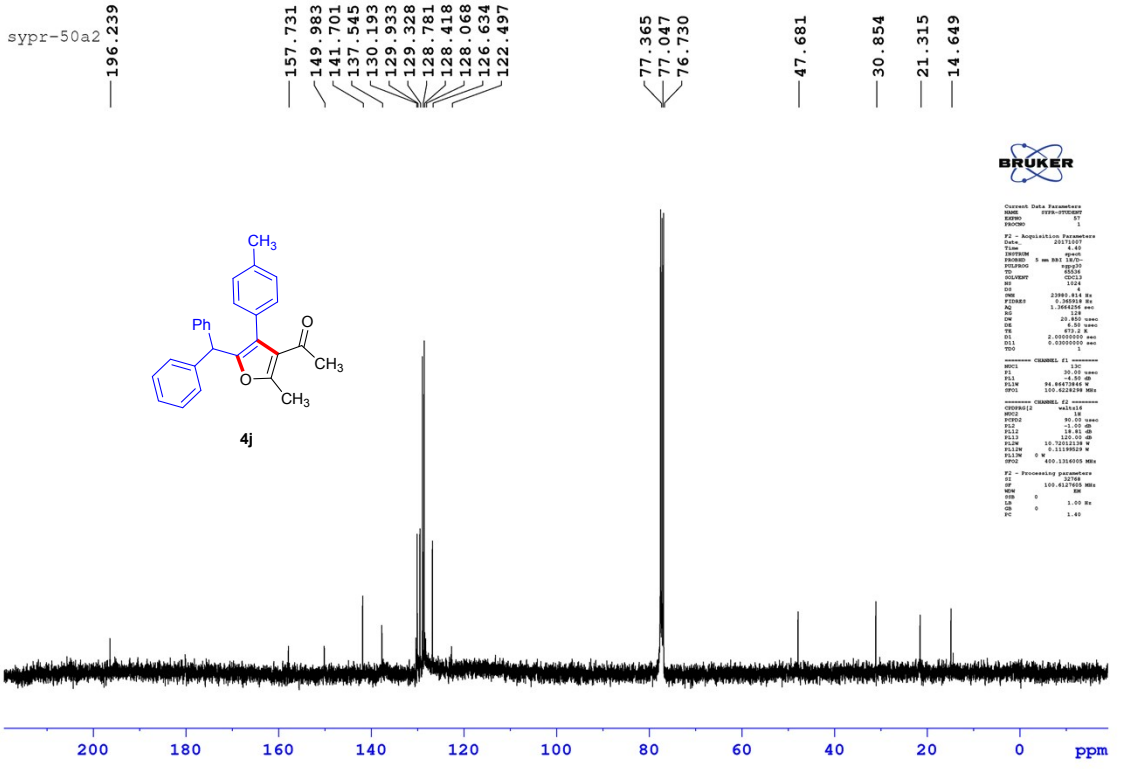

SYPR-143 A
7.402
7.398
7.395
7.385
7.382
7.370
7.358
7.355
7.349
7.344
7.274
7.259
7.244
7.211
7.207
7.192
7.188
7.178
7.140
7.125
7.071
7.054
6.821
6.803
5.158
3.751
2.541
1.902
0.002
Ph
Ph
O
CH3
O
CH3
MeO
4h
BRUKER
Current Data Parameters
NAME SYPR-ASCEND-500
EXPNO 83
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180607
Time 10.37 h
INSTRUM spect
PROBHD Z125331_0034 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 91.23
DW 50.000 usec
DE 6.50 usec
TE 301.3 K
D1 1.00000000 sec
TD0 1
SFO1 500.3720899 MHz
NUC1 1H
P1 8.00 usec
PLW1 12.18000031 W
F2 - Processing parameters
SI 65536
SF 500.3690354 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
11
10
9
8
7
6
5
4
3
2
1
0
-1 ppm
3.49
4.07
1.22
1.94
1.78
1.86
1.00
3.22
3.00
3.17

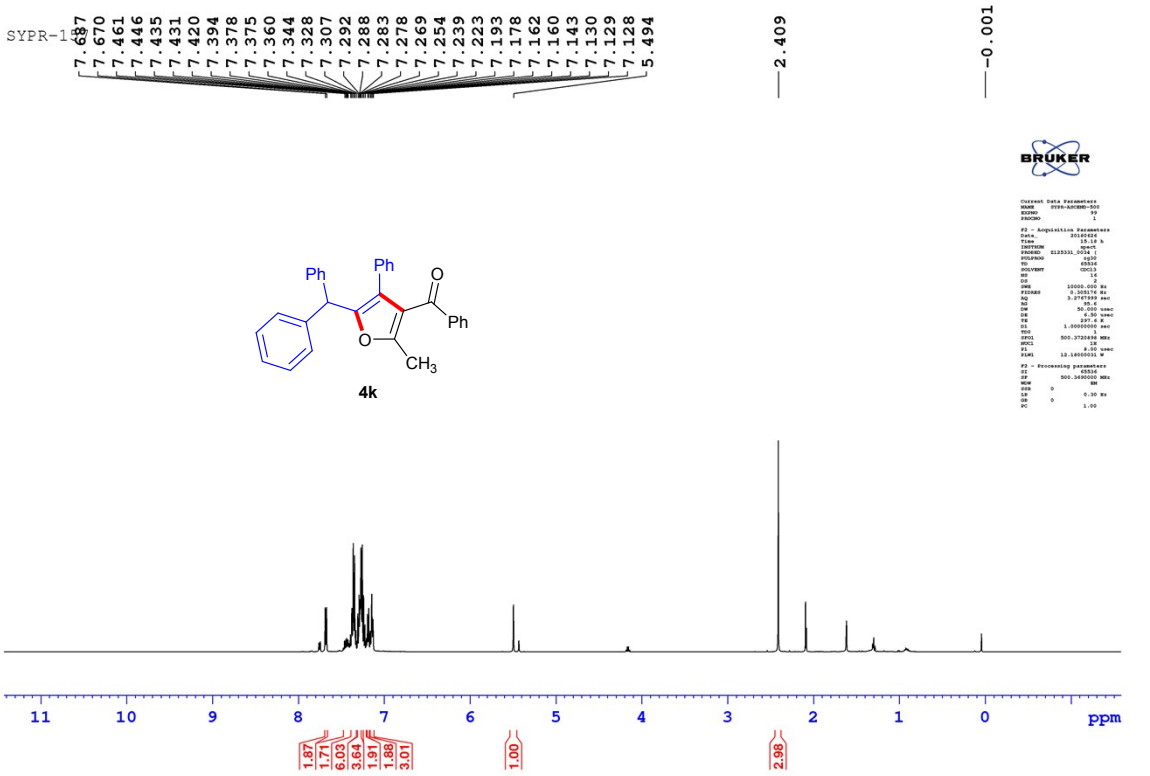

SYPR-143 A
195.987
158.398
157.721
150.473
142.007
133.789
133.442
130.149
129.827
128.714
128.641
128.457
127.837
126.654
122.975
122.366
77.403
77.149
76.895
55.272
47.063
30.814
14.646
Ph
Ph
O
CH3
O
CH3
MeO
4h
BRUKER
Current Data Parameters
NAME sypr sscnd-500
EXPNO 84
PROCNO 1
F2 - Acquisition Parameters
Date_ 20180607
Time 11.14 h
INSTRUM spect
PROBHD Z125331_0034 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 699
DS 4
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 1.1010048 sec
RG 14.45
DW 16.800 usec
DE 6.50 usec
TE 301.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.8304669 MHz
NUC1 13C
P1 12.00 usec
PLW1 172.66999817 W
SFO2 500.3710015 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 12.18000031 W
PLW12 0.12180000 W
PLW13 0.06126500 W
F2 - Processing parameters
SI 32768
SF 125.8178833 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

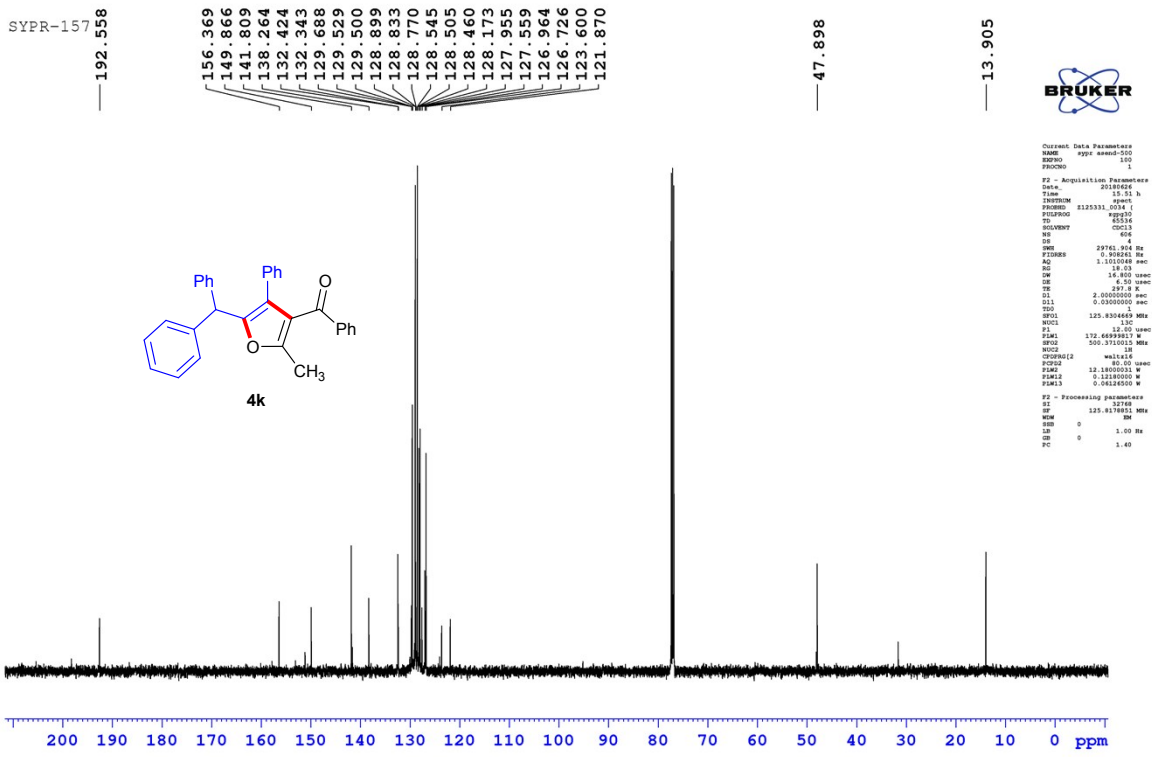

SYPR-148
7.407
7.392
7.227
7.215
7.085
7.069
7.059
6.985
6.968
6.951
5.149
2.550
1.905
0.000
F
Ph
O
CH3
O
CH3
F
4i
BRUKER
11
10
9
8
7
6
5
4
3
2
1
0
−1
ppm
2.96
2.23
3.93
3.89
1.00
3.04
3.08

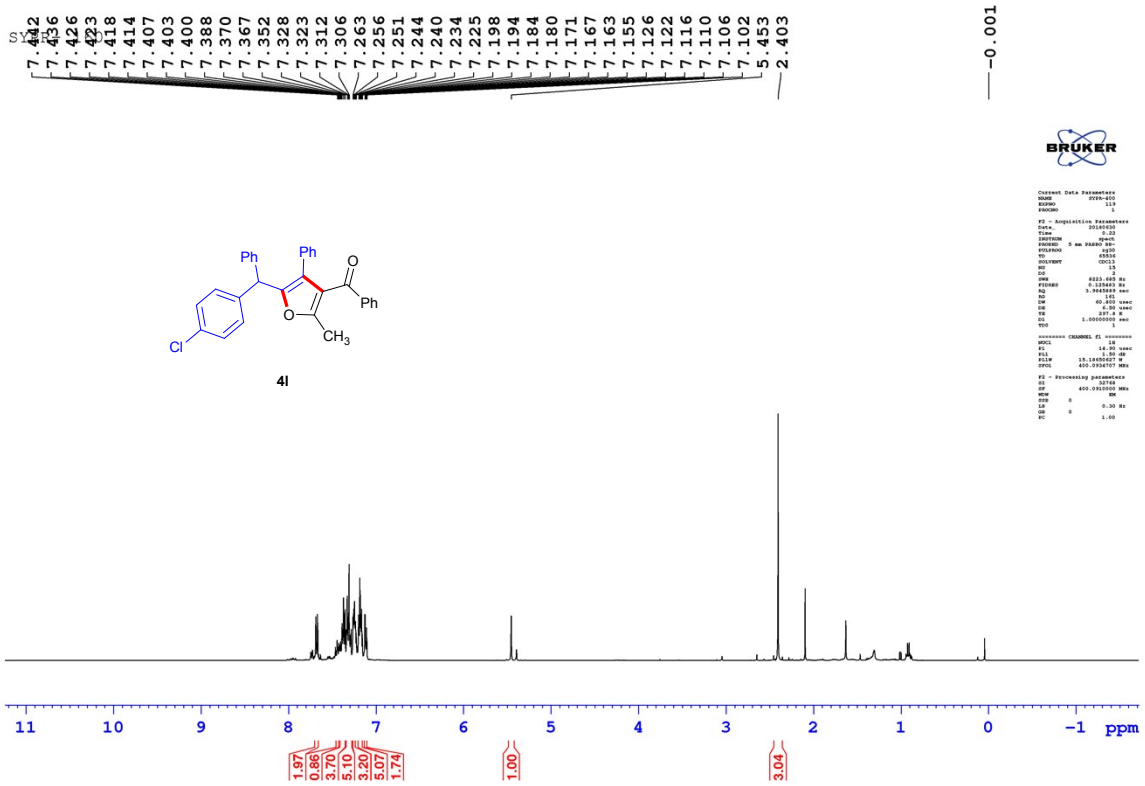

SYPR-148
195.893
162.825
160.870
157.991
149.738
137.305
137.279
133.220
130.312
130.249
130.097
128.805
128.086
123.102
122.806
115.550
115.381
77.401
77.147
76.893
46.522
30.862
14.679
F
Ph
O
CH3
O
CH3
F
4i
BRUKER
200
180
160
140
120
100
80
60
40
20
0
ppm

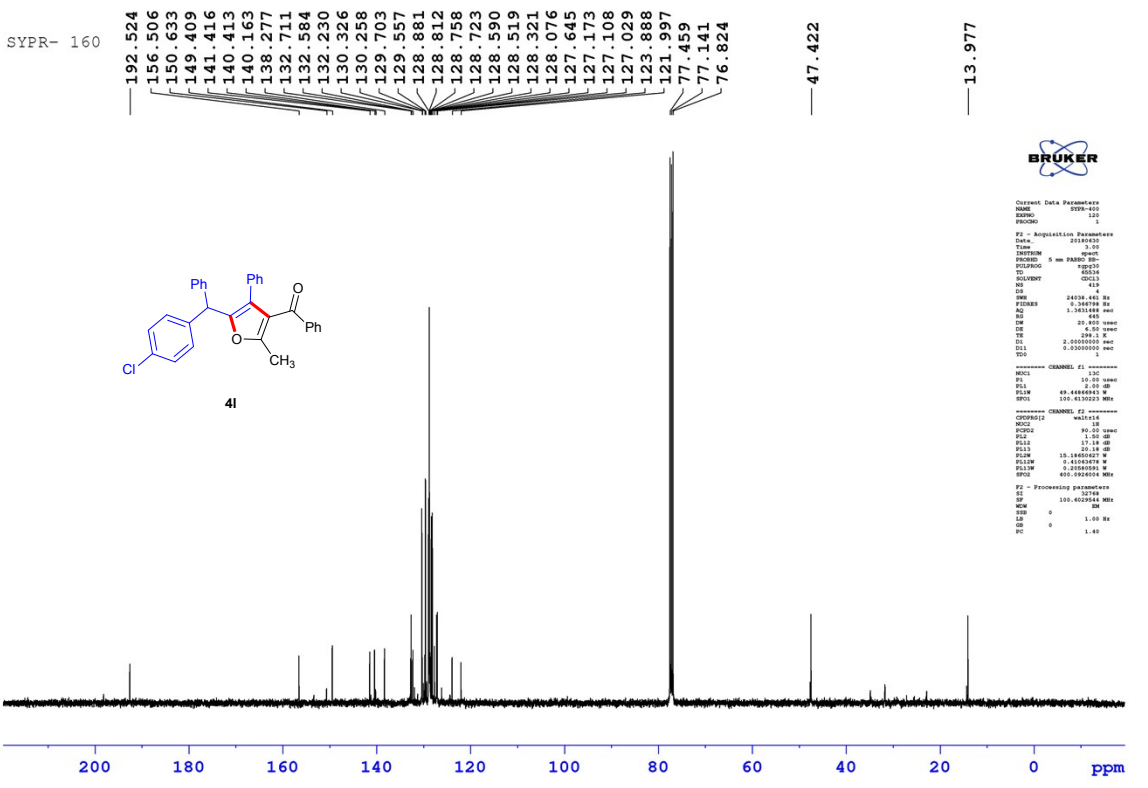

sypr-50a2
7.332
7.329
7.313
7.296
7.293
7.284
7.271
7.269
7.265
7.251
7.231
7.183
7.164
7.163
5.226
2.568
2.424
1.946
0.000
BRUKER
CH3
Ph
O
CH3
O
CH3
4j
ppm
4.26
4.00
5.70
1.00
3.15
3.60
3.35

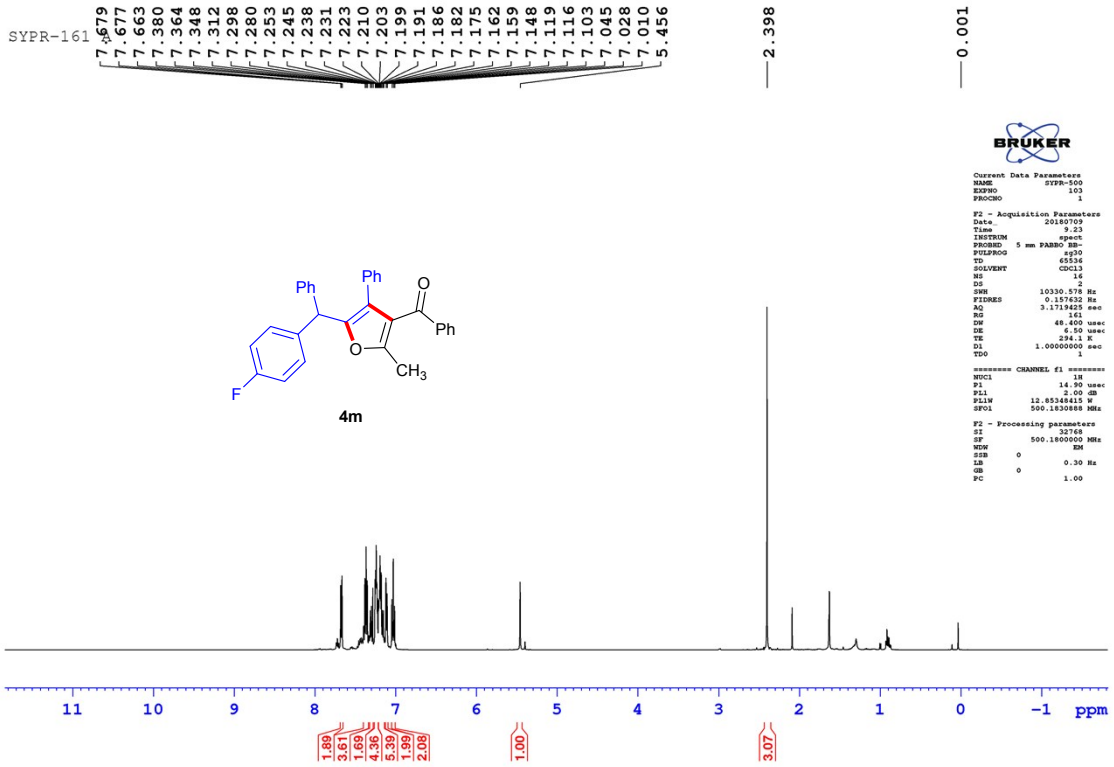

sypr-50a2
196.239
157.731
149.983
141.701
137.545
130.193
129.933
129.328
128.781
128.418
128.068
126.634
122.497
77.365
77.047
76.730
47.681
30.854
21.315
14.649
BRUKER
CH3
Ph
O
CH3
O
CH3
4j
ppm

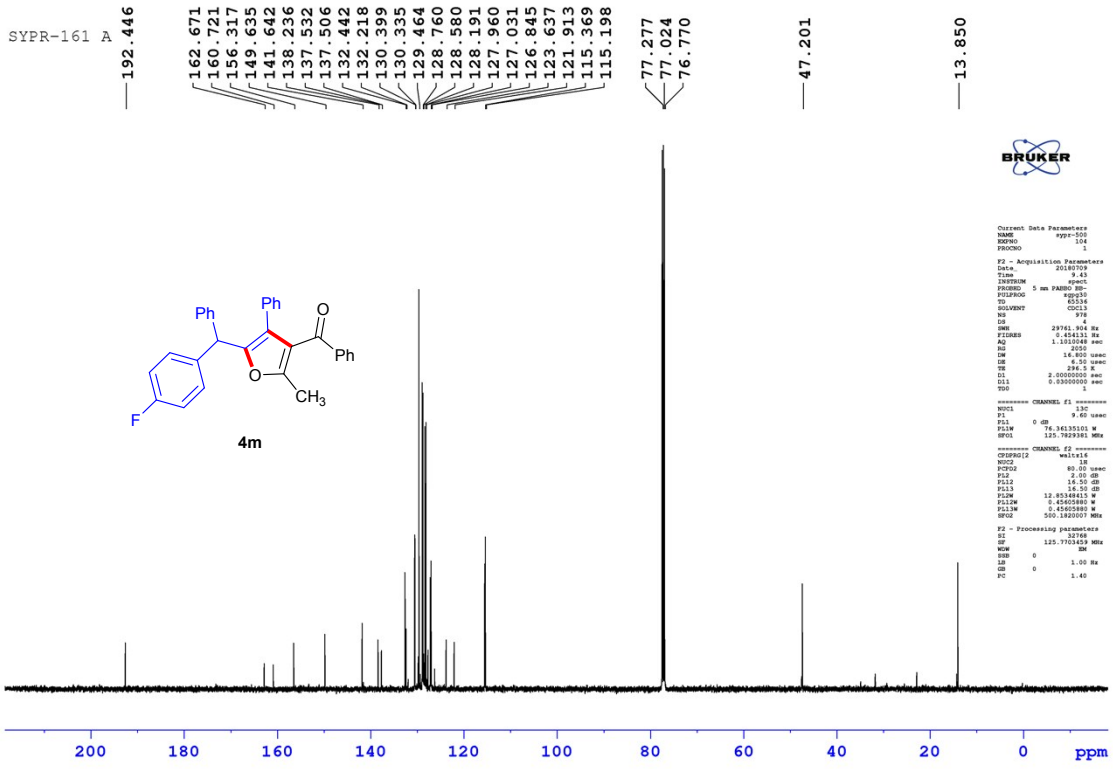

SYPR-157
7.687
7.670
7.461
7.446
7.435
7.431
7.420
7.394
7.378
7.375
7.360
7.344
7.328
7.307
7.292
7.288
7.283
7.278
7.269
7.254
7.239
7.223
7.193
7.178
7.162
7.160
7.143
7.130
7.129
7.128
5.494
2.409
0.001
BRUKER
Ph
Ph
O
Ph
O
CH3
4k
11
10
9
8
7
6
5
4
3
2
1
0
ppm
1.87
1.71
6.03
3.64
1.91
1.88
3.01
1.00
2.98

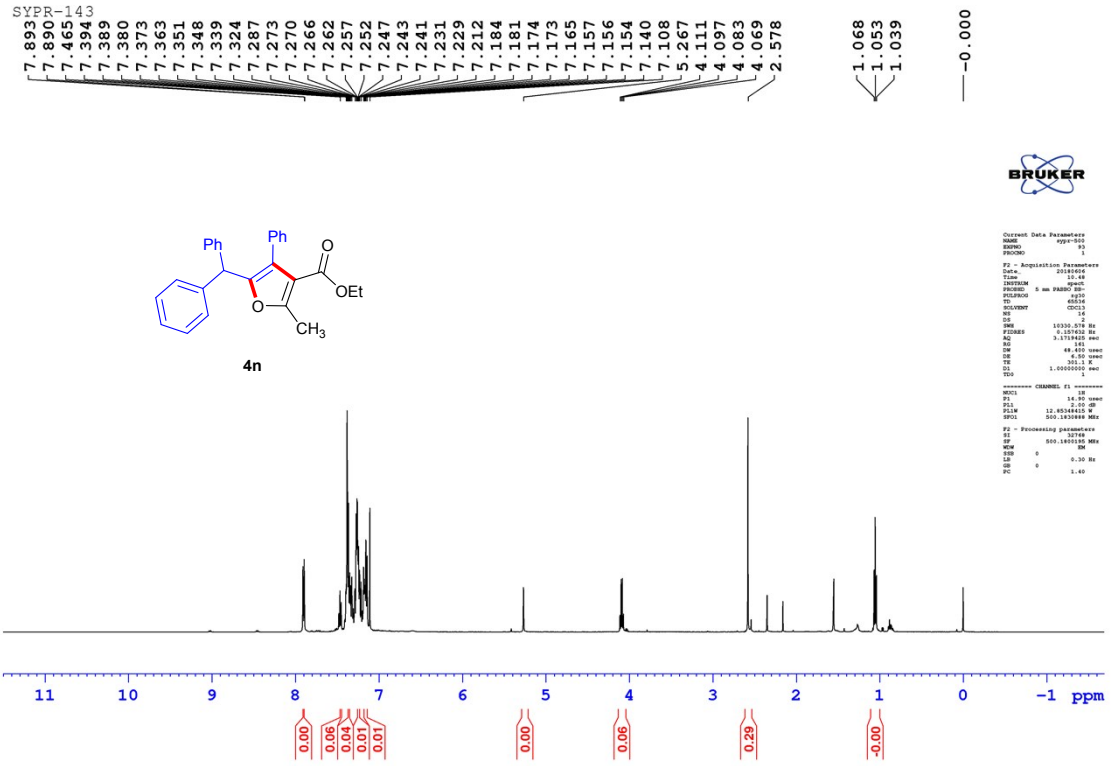

SYPR-157
192.558
156.369
149.866
141.809
138.264
132.424
132.343
129.688
129.529
129.500
128.899
128.833
128.770
128.545
128.505
128.460
128.173
127.955
127.559
126.964
126.726
123.600
121.870
47.898
13.905
BRUKER
Ph
Ph
O
Ph
O
CH3
4k
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

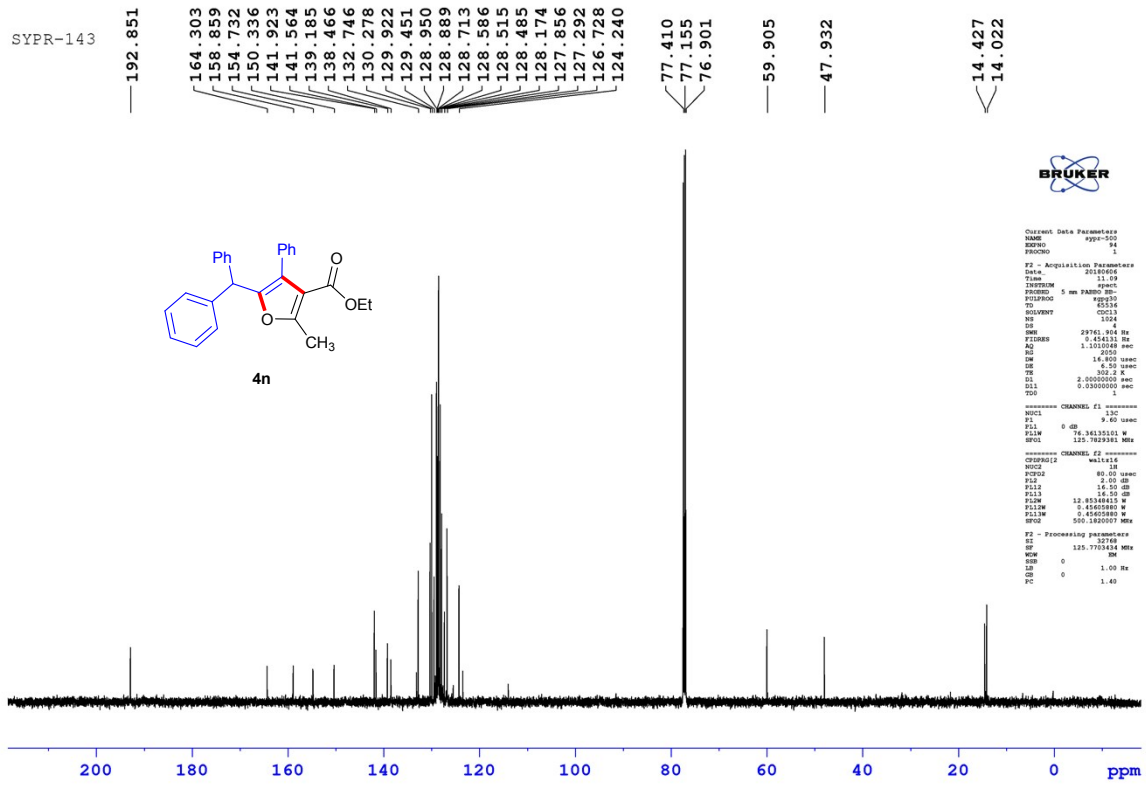

SYPR-160
7.442
7.436
7.426
7.423
7.418
7.414
7.407
7.403
7.400
7.388
7.370
7.367
7.352
7.328
7.323
7.312
7.306
7.263
7.256
7.251
7.244
7.240
7.234
7.225
7.198
7.194
7.184
7.180
7.171
7.167
7.163
7.155
7.126
7.122
7.116
7.110
7.106
7.102
5.453
2.403
0.001
BRUKER
Ph
Ph
O
Ph
O
Cl
CH3
4l
11
10
9
8
7
6
5
4
3
2
1
0
−1
ppm
1.97
0.86
3.70
5.10
3.20
5.07
1.74
1.00
3.04


SYPR- 160
192.524
156.506
150.633
149.409
141.416
140.413
140.163
138.277
132.711
132.584
132.230
130.326
130.258
129.703
129.557
128.881
128.812
128.758
128.723
128.590
128.519
128.321
128.076
127.645
127.173
127.108
127.029
123.888
121.997
77.459
77.141
76.824
47.422
13.977
BRUKER
Ph
Ph
O
Ph
O
Cl
CH3
4l
200
180
160
140
120
100
80
60
40
20
0
ppm

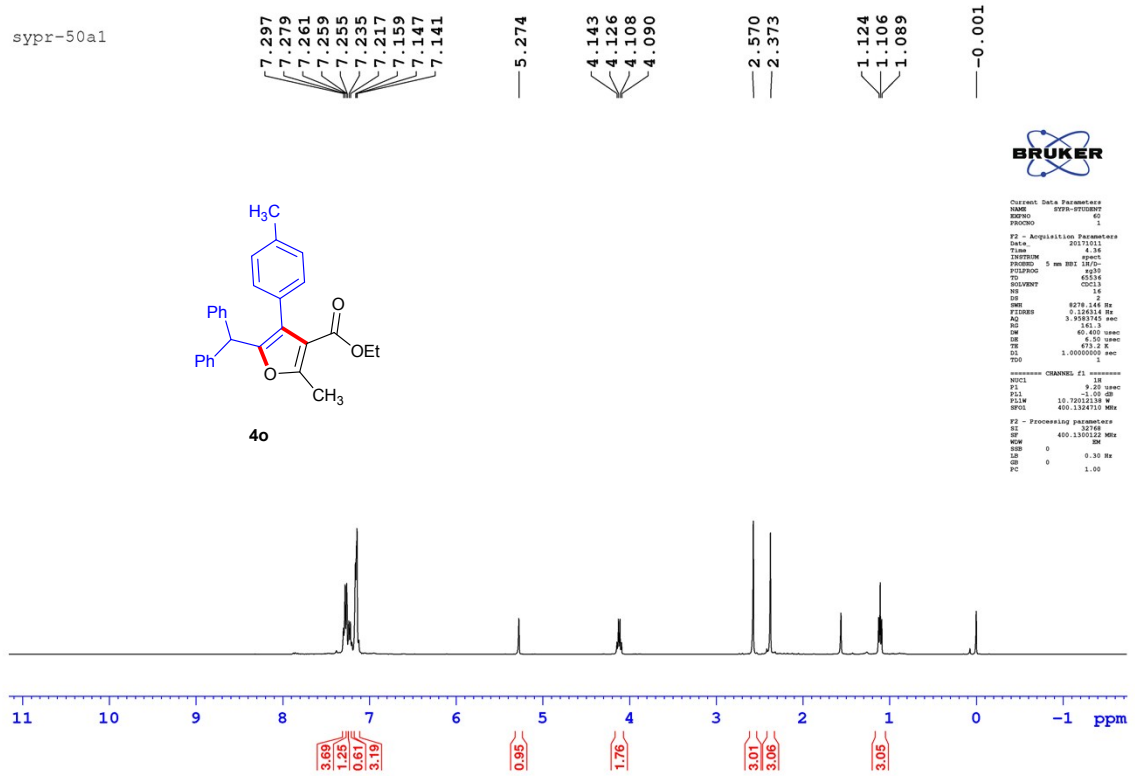

SYPR-161
7.679
7.677
7.663
7.380
7.364
7.348
7.312
7.298
7.280
7.253
7.245
7.238
7.231
7.223
7.210
7.203
7.199
7.191
7.186
7.182
7.175
7.162
7.159
7.148
7.119
7.116
7.103
7.045
7.028
7.010
5.456
2.398
0.001
BRUKER
Ph
Ph
O
Ph
O
CH3
F
4m
11
10
9
8
7
6
5
4
3
2
1
0
−1
ppm
1.89
3.61
1.69
4.36
5.39
1.99
2.08
1.00
3.07


SYPR-161 A
192.446
162.671
160.721
156.317
149.635
141.642
138.236
137.532
137.506
132.442
132.218
130.399
130.335
129.464
128.760
128.580
128.191
127.960
127.031
126.845
123.637
121.913
115.369
115.198
77.277
77.024
76.770
47.201
13.850
BRUKER
Ph
Ph
O
Ph
O
CH3
F
4m
200
180
160
140
120
100
80
60
40
20
0
ppm


SYPR-143
7.893
7.890
7.465
7.394
7.389
7.380
7.373
7.363
7.351
7.348
7.339
7.324
7.287
7.273
7.270
7.266
7.262
7.257
7.252
7.247
7.243
7.241
7.231
7.229
7.212
7.184
7.181
7.174
7.173
7.165
7.157
7.156
7.154
7.140
7.108
5.267
4.111
4.097
4.083
4.069
2.578
1.068
1.053
1.039
-0.000
Ph
Ph
O
OEt
O
CH3
4n
11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
0.00
0.06
0.04
0.01
0.01
0.00
0.06
0.29
-0.00

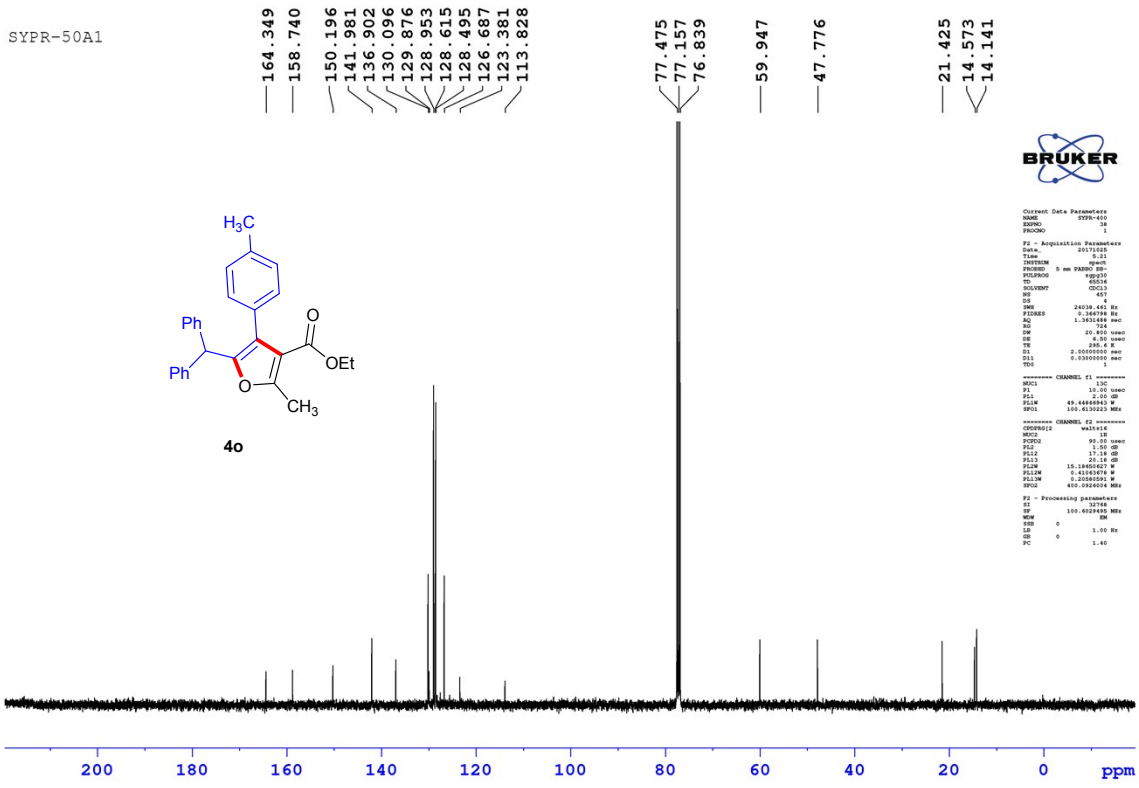

SYPR-143
192.851
164.303
158.859
154.732
150.336
141.923
141.564
139.185
138.466
132.746
130.278
129.922
129.451
128.950
128.889
128.713
128.586
128.515
128.485
128.174
127.856
127.292
126.728
124.240
77.410
77.155
76.901
59.905
47.932
14.427
14.022
Ph
Ph
O
OEt
O
CH3
4n
200
180
160
140
120
100
80
60
40
20
0
ppm

sypr-50a1



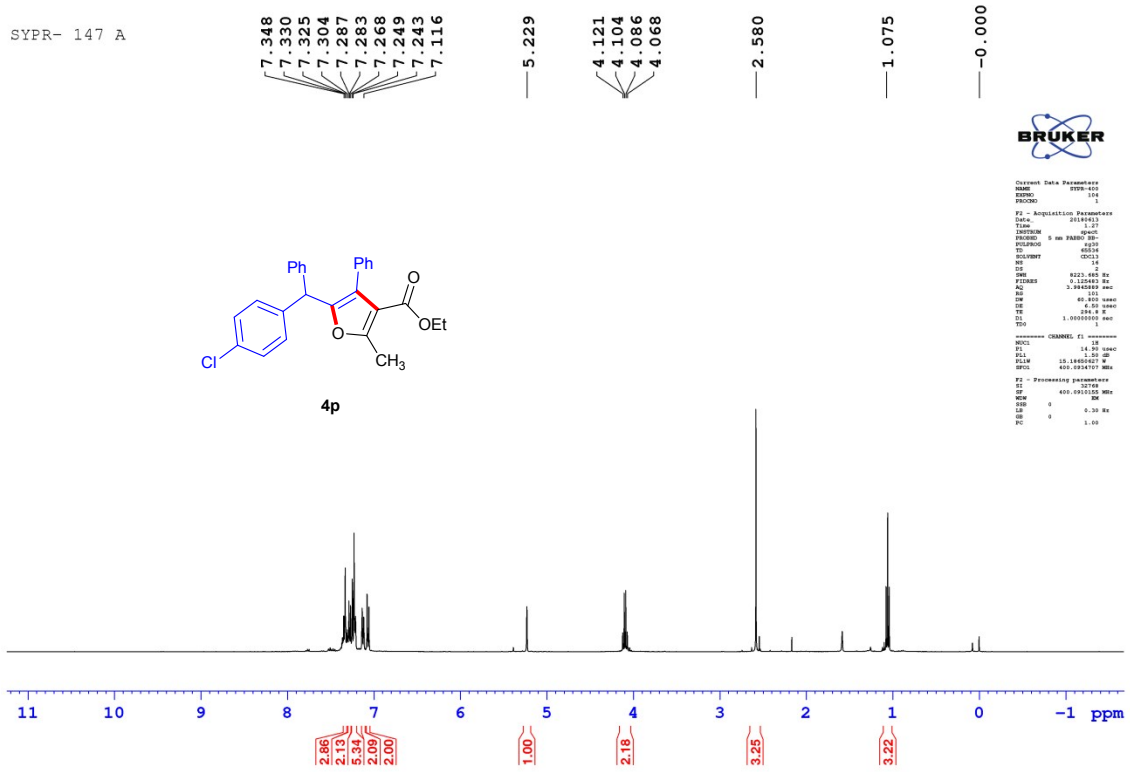
4o

SYPR-50A1

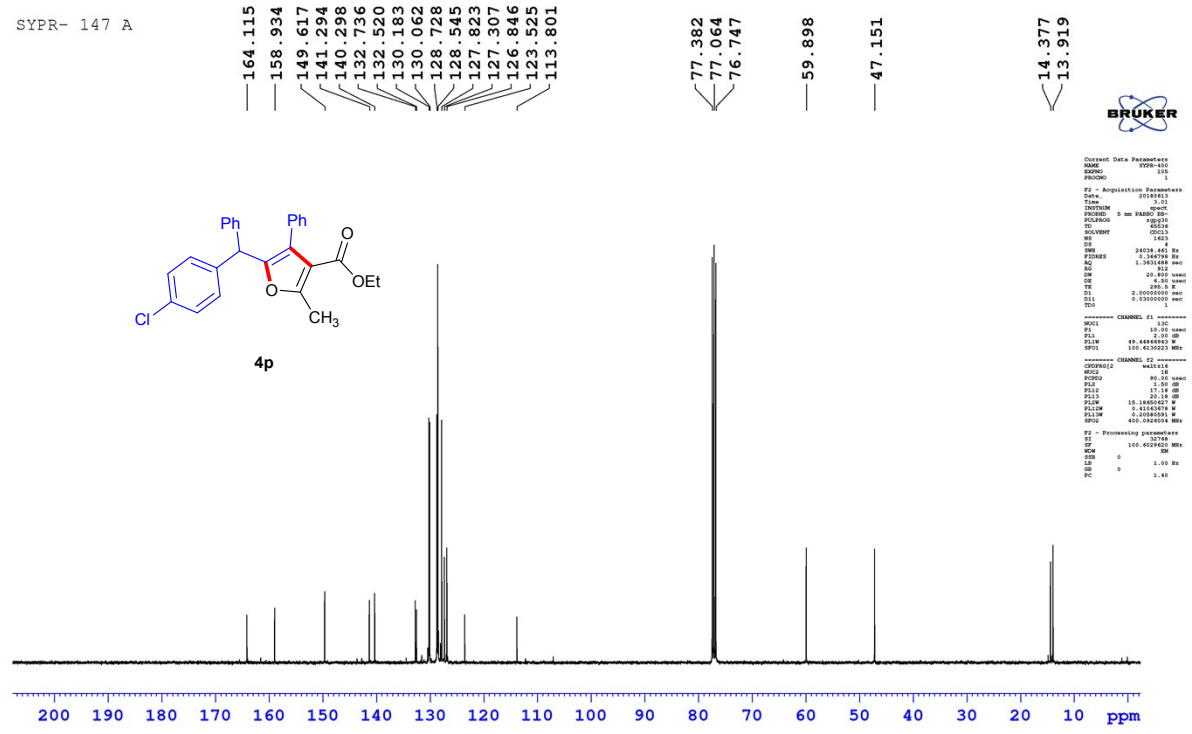


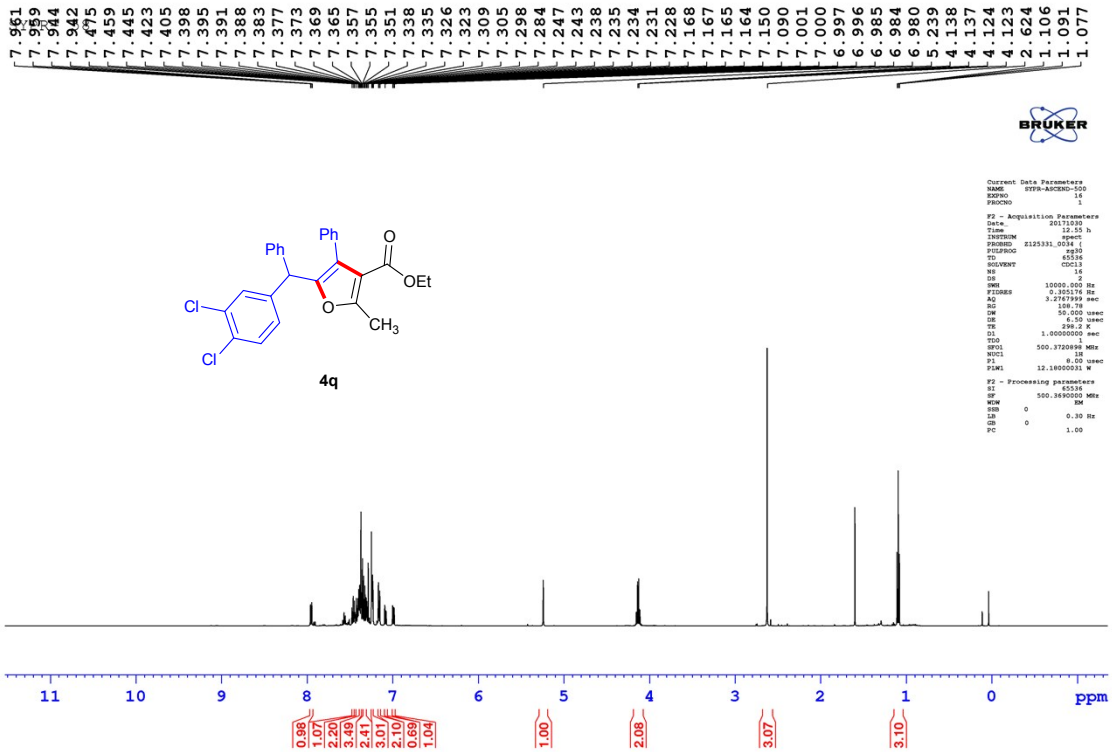
4o

SYPR- 147 A



SYPR- 147 A

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7.944
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7.459
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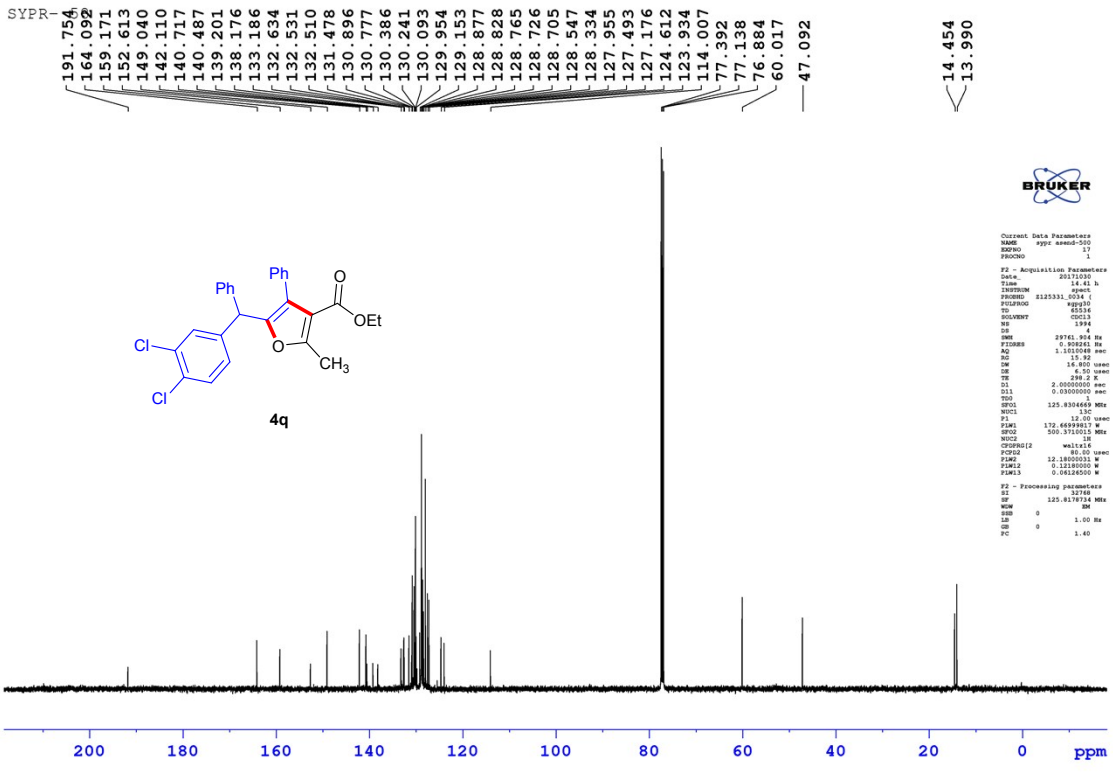
7.423
7.405
7.398
7.395
7.391
7.388
7.383
7.377
7.373
7.369
7.365
7.357
7.355
7.351
7.338
7.335
7.326
7.323
7.309
7.305
7.298
7.284
7.247
7.243
7.238
7.235
7.234
7.231
7.228
7.168
7.167
7.165
7.164
7.150
7.090
7.001
7.000
6.997
6.996
6.985
6.984
6.980
5.239
4.138
4.137
4.124
4.123
2.624
1.106
1.091
1.077
BRUKER
Ph
Ph
O
OEt
Cl
Cl
O
CH3
4q
Current Data Parameters
NAME SYPR-ASCEND-500
EXPNO 16
PROCNO 1
F2 - Acquisition Parameters
Date_ 20171030
Time 12.55 h
INSTRUM spect
PROBHD Z125331_0034 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 4
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 189.79
DW 50.000 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
SFO1 500.3720898 MHz
NUC1 1H
P1 0.00 usec
PLW1 12.18000031 W
F2 - Processing parameters
SI 65536
SF 500.3690000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
11
10
9
8
7
6
5
4
3
2
1
0
ppm
0.98
1.07
2.20
3.49
2.41
3.01
2.10
0.69
1.04
1.00
2.08
3.07
3.10


SYPR-ASCEND-500
191.756
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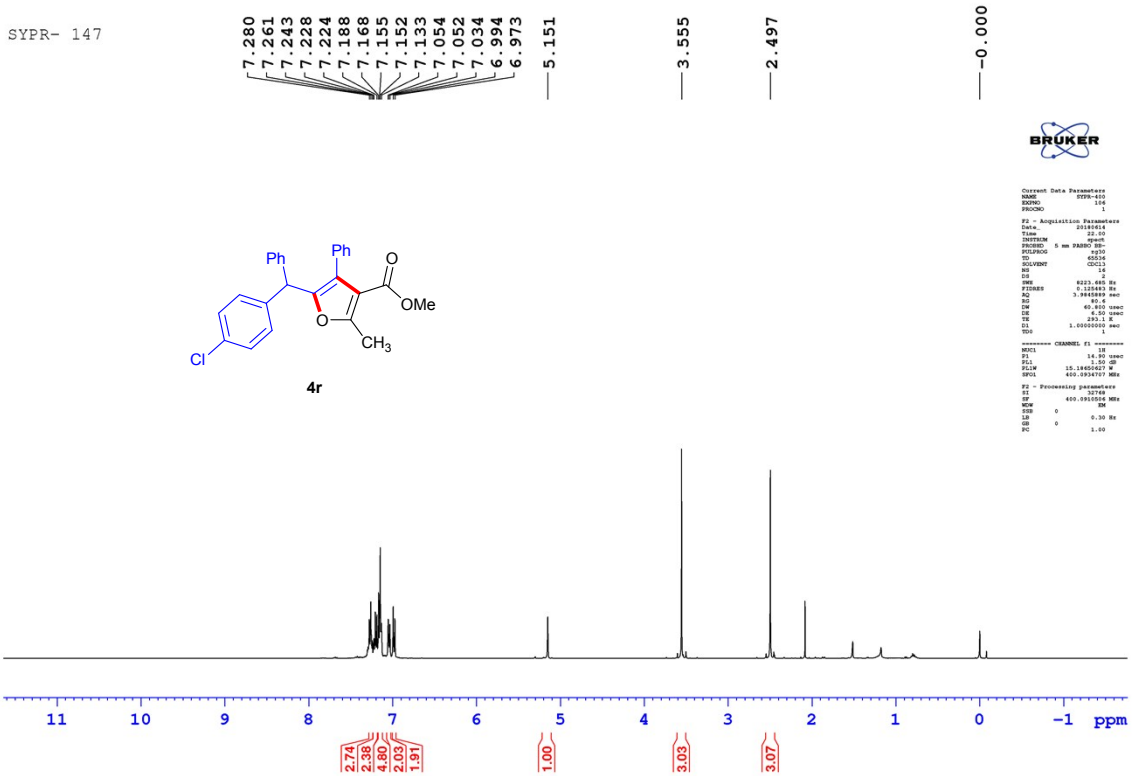
159.171
152.613
149.040
142.110
140.717
140.487
139.201
138.176
133.186
132.634
132.531
132.510
131.478
130.896
130.777
130.386
130.241
130.093
129.954
129.153
128.877
128.828
128.765
128.726
128.705
128.547
128.334
127.955
127.493
127.176
124.612
123.934
114.007
77.392
77.138
76.884
60.017
47.092
14.454
13.990
BRUKER
Ph
Ph
O
OEt
Cl
Cl
O
CH3
4q
Current Data Parameters
NAME sypr ascend-500
EXPNO 17
PROCNO 1
F2 - Acquisition Parameters
Date_ 20171030
Time 14.41 h
INSTRUM spect
PROBHD Z125331_0034 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1994
DS 4
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 1.1010048 sec
RG 15.92
DW 16.800 usec
DE 6.50 usec
TE 298.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 125.8304669 MHz
NUC1 13C
P1 12.00 usec
PLW1 172.66999817 W
SFO2 500.3710015 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 12.18000031 W
PLW12 0.12180000 W
PLW13 0.06126500 W
F2 - Processing parameters
SI 32768
SF 125.8178734 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

SYPR- 147



SYPR- 147