Pd(II)-Catalyzed Formal [4+1] Cycloadditions of Diazoacetates and Aryl Propargyl Alcohols to Form 2, 5-Dihydrofurans

Taoda Shi[#a], Xin Guo[#a], Wenhao Hu*[a,b]

[a] *Institute of Drug Discovery and Development, East China Normal University, Shanghai Engineering Research Center for Molecular Therapeutics and New Drug Development, Shanghai, 200062 (China)*

E-mail: whu@chem.ecnu.edu.cn

[b] *Department of Chemistry, East China Normal University, Shanghai 200062, P.R. (China.) Fax: (+86) 21-62233176*

[#] *The authors contributed equally.*

Supporting Information

General Information All reactions were carried out under nitrogen atmosphere with magnetic stirring. All ^{1}H NMR, and ^{13}C NMR spectra were recorded using a Brucker-400 MHz spectrometer in $CDCl_3$unless otherwise noted. Tetramethylsilane (TMS) served as an internal standard (δ = 0) for ^{1}H NMR. Chemical shifts are reported in parts per million as follows: chemical shift, multiplicity (s =singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br =broad). HRMS (ESI) Mass spectrawere recorded on Ion Spec FT-ICR mass spectrometer.

General Procedure for the synthesis of aryl propargyl alcohols

All the aryl propargyl alcohols are prepared as the reported procedures.[1] To a 50 mL three-necked flask equipped with a condenser and a nitrogen balloon, was added aryl bromide (5.0 mmol, 1.0 eq), $Pd(PPh_3)_2Cl_2$(0.05 mmol, 0.01 eq), CuI (0.5 mmol. 0.1 eq.), 25 mL Et_3N and propargyl alcohol (6.0 mmol, 1.2 eq.). Then the mixture was heated up to 80 ^{o}C overnight. After the reaction finished, the mixture was poured into cold ammonia solution, stirred for 5 mins, and extracted with ethyl acetate for 3 times (15 mL/per run). The combined organic phase was washed with NH_4Cl and brine, dried with anhydrous $MgSO_4$, filtered and evaporated. All the alcohols were purified on Silicon gel column chromatography with petrol ether and ethyl acetate as elution.

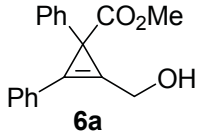
General Procedure for the synthesis of diazoacetates

All the diazoascetates were prepared as reported procedures.[2] Methyl arylacetates (10 mmol, 1.0 eq) and *p*-ABSA (11 mmol, 1.1eq.) was dissolved in a 100 mL flask with 40 mL CH_3CN. After the flask was fixed in ice bath for 10 min, 20 mL solution of DBU (1.1 mmol, 1.1 eq.) in CH_3CN was added drop wise. The starting material disappeared in 2 hours, monitored by TLC. Then the reaction mixture was dumped down to saturated aqueous NH_4Cl at 0 ^{o}C and stirred for 10 min. The resulting mixture was extracted with diethyl ether for 3 times (20 mL for each run). The combined organic phase was washed with 60 mL brine, dried with anhydrous Na_2SO_4, filtered and evaporated. The crude product, mixed with 5 g silica gel, was loaded on the chromatography column, using pure petrol ether as the elution, and the pure products were obtained.

Procedure for $Cu(OTf)_2$-catalyzed reaction of 1a and 2a

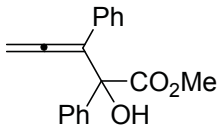
A 2-necked flask was dried with heat gun, and cooled down in the Nitrogen

atmosphere. To the flask, 200 mg 4 Å MS, $Cu(OTf)_2$ (0.04mmol, 0.1 eq.), **2a** (0.4mmol, 1.0 eq.) and 8 mL anhydrous dichloromethane was added, and then **1a**(0.6mmol, 1.5 eq.) dissolved in 4 mL anhydrous dichloromethane was added via a syringe pump over 1 hr at rt. To the crude product, 0.3g Silicon gel and 1 mL anhydrous dichloromethane was added, and then the solvent was vaporized via vacuum rotator. The pure products were obtained by column chromatography with petrol ether and ethyl acetate (v/v = 50 : 1-20 : 1) as eluent.



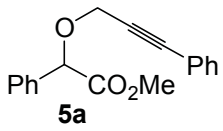
6a

Column chromatography afforded the desired product **6a** in 14% yield as colorless liquid (solidified when stored in freeze for several days): ^{1}HNMR(400 MHz, $CDCl_3$):δ7.36 (dd, J_1= 8.4 Hz, J_2= 1.6 Hz, 1H), 7.29 – 7.23 (m, 5H), 7.22-7.18 (m, 2H), 3.72 (s, 3H).^{13}CNMR (100 MHz, CDCl3): 175.2, 140.3, 129.8, 129.5, 129.0, 128.2, 128.1,126.6, 125.7, 113.5, 110.9, 56.7, 52.4, 36.7. HRMS: calcd for $C_{18}H_{16}O_3Na$ $[M+Na]^+$, 303.0997, found 303.1002.



4a

Column chromatography afforded the desired product **4a** in 28% yield as colorless liquid: ^{1}HNMR(400 MHz, $CDCl_3$):δ7.34 (dd, J_1= 16 Hz, 2H), 7.29 (d, J_1= 7.6 Hz, 2H), 7.28-7.13 (m, 6H), 5.12 (s, 2H), 4.23 (s, 1H), 3.70 (s, 3H).^{13}CNMR (100 MHz, CDCl3): 209.18, 174.42, 139.5, 133.3, 131.8, 128.1, 128.0, 127.9, 127.2, 126.9, 108.8, 81.3, 79.7, 53.3 HRMS: calcd for $C_{18}H_{16}O_3Na$ $[M+Na]^+$, 303.0997, found 303.0983.



5a

Column chromatography afforded the desired product **5a** in 5% yield as colorless liquid: ^{1}HNMR(400 MHz, $CDCl_3$):δ7.34 (dd, J_1= 16 Hz, 2H), 7.29 (d, J_1= 7.6 Hz, 2H), 7.28-7.13 (m, 6H), 5.12 (s, 2H), 4.23 (s, 1H), 3.70 (s, 3H).^{13}CNMR (100 MHz, CDCl3): 209.18, 174.42, 139.5, 133.3, 131.8, 128.1, 128.0, 127.9, 127.2, 126.9, 108.8, 81.3, 79.7, 53.3. HRMS: calcd for $C_{18}H_{16}O_3Na$ $[M+Na]^+$, 303.0997, found 303.0998.

Procedure for Complex of $[PdCl(\eta^3\text{-}C_3H_5)]_2$ and *t*-Bu-Box-catalyzed reaction of 1a and 2a.

A 2-necked flask was dried with heat gun, and cooled down in the Nitrogen atmosphere. To the flask, 200 mg 4 Å MS, $[PdCl(\eta^3\text{-}C_3H_5)]_2$(0.008mmol, 0.02 eq.), *t*-Bu-Box(0.016mmol, 0.04eq.) and 1 mL anhydrous dichloromethane was added was added and the reaction mixture was stirred at room temperature for 2 hours before

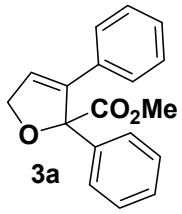
phenyl propargyl alcohol **2a** (0.4mmol, 1.0 eq.) and 8 mL anhydrous dichloromethane was added. Methyl phenyldiazoacetate **1a** (0.6mmol, 1.5 eq.) dissolved in 4 mL anhydrous dichloromethane was added via a syringe pump over 1 hour at rt. To the crude product, 0.3 g Silicon gel and 1 mL anhydrous dichloromethane was added, and the solvent was vaporized via a vacuum rotator. The pure products were obtained by column chromatography with petrol ether and ethyl acetate (v/v = 20 : 1) as eluent.

Procedure for $AgSbF_6$-activated $[Ir(COD)Cl]_2$ catalyzed cyclopropenation of 1a and 2a:

A 2-necked flask was dried with heat gun, and cooled down in the Nitrogen atmosphere. To the flask, 200 mg 4 Å MS, **$[Ir(COD)Cl]_2$**(0.01mmol, 0.05 equiv), $AgSbF_6$(0.02 mmol, 0.1 equiv), *R*-BINAP (0.01 mmol, 0.05equiv) and 1 mL anhydrous dichloromethane was added, and stirred at room temperature for 2 hours. **2a** (0.2 mmol, 1.0 eq.) and 8 mL anhydrous dichloromethane was added, and then **1a** (0.3mmol, 1.5 eq.) dissolved in 4 mL anhydrous dichloromethane was added via a syringe pump over 1 hour at rt. Then 4 Å MS was removed via flash column chromatography. To the crude product, 0.2 g Silicon gel and 1 mL anhydrous dichloromethane was added, and then the solvent was vaporized via vacuum rotator. The pure products were obtained by column chromatography with petrol ether and ethyl acetate (v/v = 3:1) as elution.

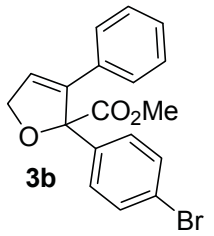
General Procedure for Pd(II)-catalyzed auto-tandem reaction of diazoesters and arylpropargyl alcohols

A 2-necked flask was dried with heat gun, and cooled down in the Nitrogen atmosphere. To the flask, 200 mg 4 Å MS, $[PdCl(\eta^3\text{-}C_3H_5)]_2$ (0.004mmol, 0.02equiv), aryl propargyl alcohol **2**(0.2 mmol, 1.0 eq.) and 8 mL anhydrous dichloromethane was added and stirred. Then diazoacetate **1**(0.3mmol, 1.5 eq.) dissolved in 4 mL anhydrous dichloromethane was added via a syringe pump over 1 hr at rt. To the crude product, 0.2 g Silicon gel and 1 mL anhydrous dichloromethane was added, and the solvent was vaporized via vacuum rotator. The pure products were obtained by column chromatography with petrol ether and ethyl acetate (v/v = 20:1) as elution.



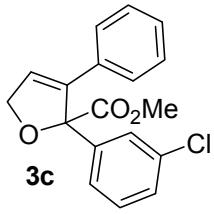
3a

Column chromatography afforded the desired product **3a** in 70% yield as colorless liquid: ^{1}HNMR(400 MHz, $CDCl_3$):δ7.35 – 7.20 (m, 10H), 6.47 (t, J = 2 Hz, 1H), 4.99 (dd, J_1= 5.2 Hz, J_2= 2 Hz, 1H), 4.93 (dd, J_1= 5.2 Hz, J_2= 2 Hz, 1H), 3.78 (s, 3H).^{13}CNMR(100 MHz, CDCl3): 172.0, 141.2, 139.4, 132.6, 128.3, 128.2, 128.0, 127.9, 127.4, 126.8, 94.8, 74.8, 52.5. HRMS: calcd for $C_{15}H_{13}N_3O_4Na$ $[M+Na]^+$, 322.0804, found 322.0813.



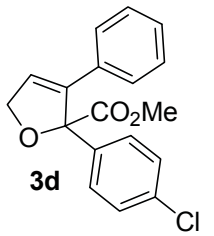
3b

Column chromatography afforded the desired product **3b** in 60% yield as colorless liquid: ^{1}HNMR(400 MHz, $CDCl_3$):δ7.44 (d, J = 8.8 Hz, 2H), 7.26 – 7.24 (m, 7H), 6.47 (t, J = 2 Hz, 1H), 4.96 (dd, J_1= 5.2 Hz, J_2= 2 Hz, 1H), 4.92 (dd, J_1= 5.2 Hz, J_2= 2 Hz, 1H), 3.78 (s, 3H).^{13}CNMR (100 MHz, CDCl3): 171.7, 141.0, 138.4, 132.3, 131.3, 129.2, 128.2, 128.1, 127.8, 127.1, 122.5, 94.1, 75.0, 52.7. HRMS: calcd for $C_{15}H_{13}N_3O_4Na$ $[M+Na]^+$, 322.0804，found 322.0813.



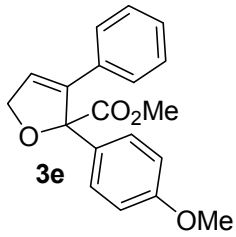
3c

Column chromatography afforded the desired product **3c** in 54%yield as colorless liquid: ^{1}HNMR(400 MHz, $CDCl_3$):δ7.39 (s, 1H), 7.29 – 7.22 (m, 8H), 6.45 (t, J = 2 Hz, 1H), 4.99 (dd, J_1= 13.6 Hz, J_2= 1.6 Hz, 1H), 4.93 (dd, J_1= 13.6 Hz, J_2= 1.6 Hz, 1H), 3.78 (s, 3H).^{13}CNMR (100 MHz, CDCl3): 171.5, 141.3, 141.0, 134.2, 132.3, 129.3, 128.4, 128.1, 128.0, 127.9, 127.6, 127.1, 125.6, 94.1, 75.0, 52.7. HRMS: calcd for $C_{18}H_{15}ClO_3Na$ $[M+Na]^+$, 337.0607，found 337.0616.



3d

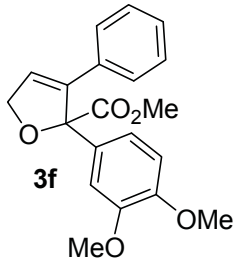
Column chromatography afforded the desired product **3d** in 69% yield as colorless liquid: ^{1}HNMR(400 MHz, CDCl3):δ7.30 – 7.21 (m, 9H), 6.48 (t, J = 2 Hz, 1H), 4.99 (dd, J_1= 12 Hz, J_2= 4 Hz, 1H), 4.93 (dd, J_1= 12 Hz, J_2= 4 Hz, 1H), 3.78 (s, 3H).^{13}CNMR (100 MHz, CDCl3): 171.8, 140.9, 137.8, 131.3, 128.9, 128.8, 128.1, 127.8, 127.0, 91.1, 75.0, 52.7. HRMS: calcd for $C_{18}H_{15}ClO_3Na$ $[M+Na]^+$, 337.0607，found 337.0619.



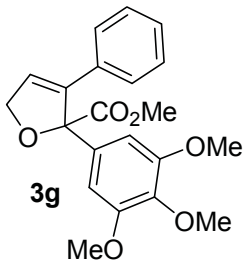
3e

Column chromatography afforded the desired product **3e** in 72% yield as colorless liquid: ^{1}HNMR(400 MHz, CDCl3):δ7.20 – 7.14 (m, 6H), 6.85-6.38 (m, 3H), 6.38 (t, J = 2 Hz, 1H), 4.91 (dd, J_1= 14 Hz, J_2= 2 Hz, 1H), 4.86 (dd, J_1= 14 Hz, J_2= 2 Hz, 1H), 3.70 (s, 3H), 3.66 (s, 3H).^{13}CNMR (100 MHz, $CDCl_3$): 171.9, 159.4, 141.3, 140.8, 132.7, 128.0, 127.9, 126.9, 119.7,

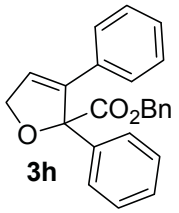
113.8, 113.2, 94.7, 74.8, 55.2, 52.6. HRMS: calcd for $C_{19}H_{18}O_4Na$ $[M+Na]^+$, 333.1103，found 333.1093



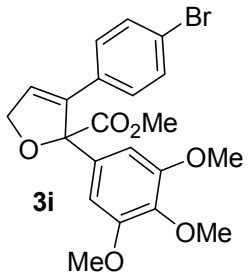
Column chromatography afforded the desired product **3f** in 74% yield as colorless liquid: ^{1}HNMR(400 MHz, CDCl3):δ7.24 – 6.79 (m, 8H),6.46 (t, J = 2 Hz, 1H), 4.98 (dd, J_1= 14 Hz, J_2= 2 Hz, 1H), 4.92 (dd, J_1= 14 Hz, J_2= 2 Hz, 1H), 3.86 (s, 3H), 3.78 (s, 3H), 3.74 (s, 3H).^{13}CNMR (100 MHz, $CDCl_3$): 172.2, 149.0, 148.6, 141.3, 132.8, 131.8, 128.0, 127.9, 127.8, 126.8, 119.9, 110.9, 110.6, 94.5, 74.6, 55.8, 55.7, 52.5. HRMS: calcd for $C_{20}H_{20}O_5Na$ $[M+Na]^+$, 363.1208，found 363.1217.



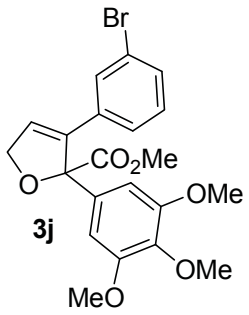
Column chromatography afforded the desired product **3g** in 78% yield as colorless liquid: ^{1}HNMR(400 MHz, CDCl3):δ7.28 – 7.25 (m, 5H), 6.57 (s, 2H), 6.41 (t, J = 1.6 Hz, 1H), 5.00 (dd, J_1= 13.8 Hz, J_2= 1.6 Hz, 1H), 4.92 (dd, J_1= 13.8 Hz, J_2= 1.6 Hz, 1H), 3.83 (s, 3H), 3.79 (s, 3H), 3.70 (s, 6H).^{13}CNMR (100 MHz, CDCl3): 172.0, 152.8, 141.4, 137.9, 134.5, 132.9, 128.2, 128.0, 127.9, 127.1, 104.7, 94.5, 74.9, 60.8, 56.0, 52.6. HRMS: calcd for $C_{21}H_{22}O_6Na$ $[M+Na]^+$, 393.1314，found 393.1328.



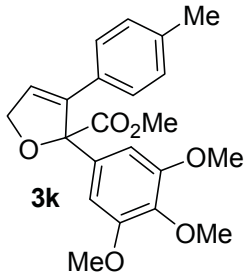
Column chromatography afforded the desired product **3h** in 59% yield as colorless liquid: ^{1}HNMR(400 MHz, $CDCl_3$):δ7.41 – 7.18 (m, 15H),6.53 (t, J = 1.6 Hz, 1H), 5.26 (s, 2H), 5.00 (dd, J_1= 14 Hz, J_2= 1.6 Hz, 1H), 4.92 (dd, J_1= 14 Hz, J_2= 1.6 Hz, 1H).^{13}CNMR (100 MHz, CDCl3): 171.3, 141.0, 139.3, 135.5, 132.7, 128.4, 128.3, 128.2,128.1 128.0, 127.9, 127.8,127.5, 127.1,94.8, 74.9, 67.1. HRMS: calcd for $C_{24}H_{20}O_3Na$ $[M+Na]^+$, 379.1310，found 379.1312.



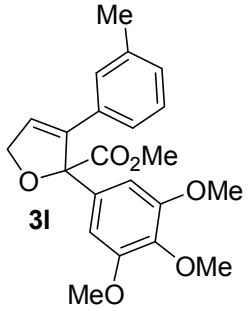
Column chromatography afforded the desired product **3i** in 58% yield as colorless liquid: ^{1}HNMR(400 MHz, $CDCl_3$):δ7.37 (d, J = 6.8 Hz, 2H),7.14 (d, J = 6.8 Hz, 2H),6.54(s, 2H), 6.44 (t, J = 2 Hz, 1H), 4.97 (dd, J_1= 14.2 Hz, J_2= 1.6 Hz, 1H), 4.92 (dd, J_1= 14.2 Hz, J_2= 1.6 Hz, 1H), 3.84 (s, 3H), 3.78 (s, 3H), 3.73 (s, 6H).^{13}CNMR (100 MHz, CDCl3): 171.8, 153.0 140.4, 138.2, 134.2, 131.7, 129.8, 127.6, 122.2, 104.6, 94.6, 74.7, 60.8, 56.1, 52.7. HRMS: calcd for $C_{21}H_{21}BrO_6Na$ $[M+Na]^+$, 471.0419，found 471.0424.



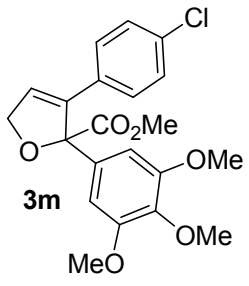
Column chromatography afforded the desired product **3j**in 54% yield as colorless liquid: ^{1}HNMR(400 MHz, $CDCl_3$):δ7.38 (d, J = 1.6 Hz, 1H),7.37(s, 1H), 7.15-7.07 (m, 2H),7.07(s, 1H), 6.39(t, J = 2 Hz, 1H), 4.98(dd, J_1= 14.2 Hz, J_2= 1.6 Hz, 1H), 4.94 (dd, J_1= 14.2 Hz, J_2= 1.6 Hz, 1H), 3.83 (s, 3H), 3.79 (s, 3H), 3.73 (s, 6H).^{13}CNMR (100 MHz, CDCl3): 171.7, 153.0 140.5, 138.2, 134.2, 131.2, 129.5, 128.2, 127.0, 122.2, 104.5, 94.5, 74.7, 60.8, 56.1, 52.7. HRMS: calcd for $C_{21}H_{21}BrO_6Na$ $[M+Na]^+$, 471.0419，found 471.0419.



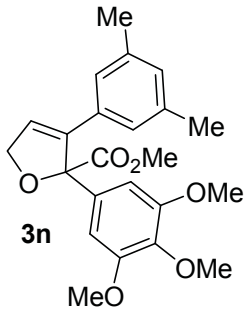
Column chromatography afforded the desired product **3k**in 62% yield as colorless liquid: ^{1}HNMR(400 MHz, CDCl3):δ7.18 (d, J = 8 Hz, 2H),7.05 (d, J = 8 Hz, 2H),6.58(s, 2H), 6.39 (t, J = 1.6 Hz, 1H), 4.98 (dd, J_1= 13.6 Hz, J_2= 1.6 Hz, 1H), 4.94 (dd, J_1= 13.6 Hz, J_2= 1.6 Hz, 1H), 3.83 (s, 3H), 3.78 (s, 3H), 3.72 (s, 6H), 2.31 (s, 3H).^{13}CNMR (100 MHz, CDCl3): 172.0, 152.8 141.2, 137.9, 134.6, 129.9, 128.8, 1287, 128.0, 126.2, 104.8, 94.5, 74.8, 60.8, 56.0, 52.6, 21.1. HRMS: calcd for $C_{22}H_{24}O_6Na$ $[M+Na]^+$, 407.1471, found 407.1487.



Column chromatography afforded the desired product **3l**in 63% yield as colorless liquid: ^{1}HNMR(400 MHz, CDCl3):δ7.13-7.02 (m, 4H),6.58(s, 2H), 6.36 (t, J = 1.6 Hz, 1H), 4.99 (dd, J_1= 13.8 Hz, J_2= 1.6 Hz, 1H), 4.94 (dd, J_1= 13.8 Hz, J_2= 1.6 Hz, 1H), 3.87 (s, 3H), 3.83 (s, 3H), 3.71 (s, 6H), 2.28 (s, 3H).^{13}CNMR (100 MHz, CDCl3): 172.0, 152.8 141.7, 137.9, 137.5, 134.6, 134.0,133.0, 128.8, 128.7, 127.9, 126.8, 125.3, 104.7, 104.1, 94.5, 74.8, 60.8, 56.2, 56.0, 52.6, 21.4. HRMS: calcd for $C_{22}H_{24}O_6Na$ $[M+Na]^+$, 407.1471, found 407.1478.

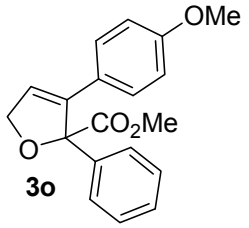


Column chromatography afforded the desired product **3m**in 58% yield as colorless liquid: ^{1}HNMR(400 MHz, CDCl3):δ7.21 (s, 4H),6.54(s, 2H), 6.43 (t, J = 1.6 Hz, 1H), 4.97 (dd, J_1= 14.0 Hz, J_2= 1.6 Hz, 1H), 4.93 (dd, J_1= 14.0 Hz, J_2= 1.6 Hz, 1H), 3.83 (s, 3H), 3.78 (s, 3H), 3.73 (s, 6H).^{13}CNMR (100 MHz, CDCl3): 171.8, 153.0 140.3, 138.2, 134.2, 134.0, 131.3, 129.5, 128.2, 127.6, 104.6, 94.6, 74.7, 60.8, 56.1, 52.7. HRMS: calcd for $C_{21}H_{21}ClO_6Na$ $[M+Na]^+$, 427.0924,found 427.0922.



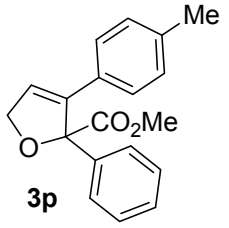
3n

Column chromatography afforded the desired product **3n**in 51% yield as colorless liquid: ^{1}HNMR(400 MHz, CDCl3):δ6.89(s, 1H), 6.85(s, 2H), 6.59(s, 2H), 6.31 (t, J = 1.6 Hz, 1H), 4.98 (dd, J_1= 13.7 Hz, J_2= 1.6 Hz, 1H), 4.91 (dd, J_1= 13.7Hz, J_2= 1.6 Hz, 1H), 3.82 (s, 3H), 3.79 (s, 3H), 3.71 (s, 3H), 2.23 (s, 6H).^{13}CNMR (100 MHz, CDCl3): 172.0, 152.6 141.8, 137.8, 137.3, 134.7, 133.0, 129.6, 126.6, 126.0, 104.6, 94.4, 74.8, 60.7, 55.9, 52.6, 21.2. HRMS: calcd for $C_{23}H_{26}O_6Na$ $[M+Na]^+$, 421.1627, found 421.1618.



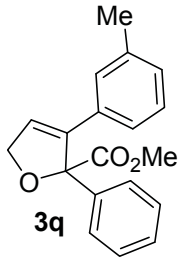
3o

Column chromatography afforded the desired product **3o** in 64% yield as colorless liquid: ^{1}HNMR(400 MHz, CDCl3):δ7.33 – 7.17 (m, 5H), 7.13 (d, J = 8 Hz, 2H), 6.66 (d, J = 8 Hz, 2H), 6.32 (t, J = 2 Hz, 1H), 4.88 (dd, J_1= 20 Hz, J_2= 4 Hz, 1H), 4.83 (dd, J_1= 20 Hz, J_2= 4 Hz, 1H), 3.70 (s, 3H), 3.67 (s, 3H).^{13}CNMR (100 MHz, CDCl3): 172.2, 159.3, 140.4, 139.5, 129.2, 128.7, 128.4, 128.3, 128.2, 127.5, 126.6, 125.0, 94.7, 74.8, 55.2, 52.6. HRMS: calcd for $C_{19}H_{18}O_4Na$ $[M+Na]^+$, 333.1103, found333.1114.



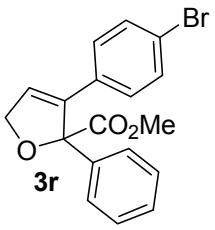
3p

Column chromatography afforded the desired product **3p** in 60% yield as colorless liquid: ^{1}HNMR(400 MHz, CDCl3):δ7.30 – 7.24 (m, 5H), 7.08 (d, J = 8 Hz, 2H), 6.94 (d, J = 8 Hz, 2H), 6.39 (t, J = 2 Hz, 1H), 4.90 (dd, J_1= 16 Hz, J_2= 4 Hz, 1H), 4.85 (dd, J_1= 16 Hz, J_2= 4 Hz, 1H), 3.71 (s, 3H), 2.22 (s, 3H).^{13}CNMR (100 MHz, CDCl3): 172.1, 140.9, 139.1, 137.8, 129.6, 128.7, 128.3, 128.2, 127.7, 127.4, 126.0, 94.7, 74.8, 52.5, 21.4. HRMS: calcd for $C_{19}H_{18}O_3Na$ $[M+Na]^+$, 317.1154, found 317.1138.

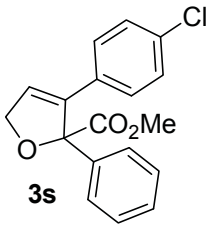


3q

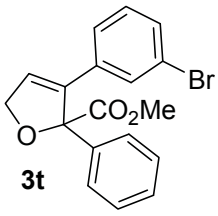
Column chromatography afforded the desired product **3q** in 61%yield as colorless liquid: ^{1}HNMR(400 MHz, CDCl3):δ7.28-7.24 (m, 5H), 7.18-6.99 (m, 4H), 6.38 (t, J = 4 Hz, 1H), 4.91 (dd, J_1= 12 Hz, J_2= 4 Hz, 1H), 4.85 (dd, J_1= 12 Hz, J_2= 4 Hz, 1H), 3.71 (s, 3H), 2.18 (s, 3H).^{13}CNMR (100 MHz, CDCl3): 172.1, 141.3, 139.4, 137.5, 132.6, 128.9, 128.7, 128.5, 128.3,128.2, 128.0, 127.9, 127.8, 127.4, 127.2, 126.7, 125.2, 125.1, 94.7, 74.8, 52.6, 21.1. HRMS: calcd for $C_{19}H_{18}O_3Na$ $[M+Na]^+$, 317.1154, found 317.1166.



Column chromatography afforded the desired product **3r** in 50%yield ascolorless liquid: ^{1}HNMR(400 MHz, CDCl3):δ7.27 – 7.24 (m, 6H), 7.19 (s, 1H), 7.04 (d, J = 8 Hz, 2H), 6.43 (t, J = 4 Hz, 1H), 4.90 (dd, J_1= 12 Hz, J_2= 4 Hz, 1H), 4.86 (dd, J_1= 12 Hz, J_2= 4 Hz, 1H), 3.72 (s, 3H).^{13}CNMR (100 MHz, CDCl3): 171.9, 140.3, 139.0, 131.2, 129.5, 128.6, 128.4, 127.2, 122.1, 94.7, 74.8, 52.6. HRMS: calcd for $C_{18}H_{15}O_3BrNa$ $[M+Na]^+$, 381.0102, found 381.0083.



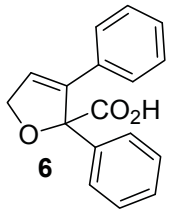
Column chromatography afforded the desired product **3s** in 55% yield as colorless liquid: ^{1}HNMR(400 MHz, CDCl3):δ7.26 (s, 5H), 7.10 (s, 4H), 6.11 (t, J = 4 Hz, 1H), 4.91 (dd, J_1= 12 Hz, J_2= 4 Hz, 1H), 4.85 (dd, J_1= 12 Hz, J_2= 4 Hz, 1H), 3.72 (s, 3H).^{13}CNMR (100 MHz, CDCl3): 170.9, 159.0, 158.1, 128.9, 128.7, 127.5, 124.6, 122.8, 121.2, 111.4, 106.7, 98.7, 79.7, 63.5,52.4.HRMS:calcd for $C_{18}H_{15}O_3ClNa$ $[M+Na]^+$, 337.0607, found 337.0623.



Column chromatography afforded the desired product **3t** in 52% yield as colorless liquid: ^{1}HNMR(400 MHz, CDCl3):δ7.36 – 7.19 (m, 7H), 7.03 – 6.69 (m, 2H), 6.41 (t, J = 4 Hz, 1H), 4.92 (dd, J_1= 12 Hz, J_2= 4 Hz, 1H), 4.87 (dd, J_1= 12 Hz, J_2= 4 Hz, 1H), 3.72 (s, 3H).^{13}CNMR (100 MHz, CDCl3): 171.8, 140.2, 139.0, 134.8, 130.9, 128.6, 128.4, 128.1,127.2, 126.6, 128.4, 128.1, 127.2, 126.6, 122.0, 94.7, 52.6. HRMS: calcd for $C_{18}H_{15}O_3BrNa$ $[M+Na]^+$, 381.0102, found 381.0121.

Procedure for hydrolysis of 2, 5-Dihydrofuran 3a

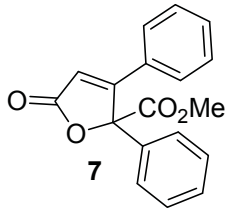
To a 20 mL flask, **3a** (0.2 mmol) and LiOH (1.0 mmol, 5.0 eq) dissolved in the mixture of water, THF and methanol was added at 0 ^{o}C. After refluxed for 4 h, the reaction was cooled down and quenched with 1 N HCl. The aqueous phase was extracted with ethyl acetate for 3 times. The combined organic phase was dried with anhydrous Na_2SO_4. The solvent was removed with vacuum rotator, affording the crude product. Then 0.2 g Silicon gel was added to make the uniformed sample for the chromatography. The pure products were obtained with dichloromethane and methanol (v/v = 50 : 1-10 : 1) as elution.



Column chromatography afforded the desired product **6** in 78% yield as colorless solid: ^{1}HNMR(400 MHz, d_6-acetone):δ13.00 (br, 1H), 7.36-7.31 (m, 10H), 6.81 (t, J = 2 Hz, 1H), 4.85 (dd, J_1= 16 Hz, J_2= 2 Hz, 1H), 4.80 (dd, J_1= 16 Hz, J_2= 2 Hz, 1H).^{13}CNMR (100 MHz, d_6-acetone): 172.5, 140.0, 139.9, 132.4, 127.9, 127.6, 127.5,127.4, 93.8,74.3. HRMS: calcd for $C_{17}H_{14}O_3Na$ $[M+Na]^+$, 289.0841, found 289.0834.

Procedure for PCC oxidation of 2, 5-Dihydrofuran 3a[3].

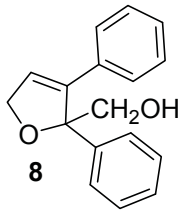
To a 20 mL flask, **3a** (0.2 mmol) was added and PCC (0.4 mmol, 2.0 eq) dissolved in $(CHCl)_2$ and KOAc (0.4 mmol, 2.0 eq) was added. After refluxed for 2 h, the reaction was cooled down and quenched with saturated NH_4Cl. The aqueous phase was extracted with ethyl acetate for 3 times. The combined organic phase was dried with anhydrous Na_2SO_4. The solvent was removed with vacuum rotator, affording the crude product. Then 0.2 g Silica gel was added to make the uniformed sample for the chromatography. The pure products were obtained with petrol ether and ethyl acetate (v/v = 10 : 1-5: 1) as elution.



Column chromatography afforded the desired product **7** in 89% yield as yellow solid: ^{1}HNMR(400 MHz, $CDCl_3$):δ7.42 – 7.27 (m, 10H), 6.55 (s, 1H), 3.72 (s, 3H).^{13}CNMR (100 MHz, CDCl3): 170.9, 168.0, 165.0, 134.7, 131.4, 129.7, 129.3,128.8, 128.0, 116.6, 91.0, 53.7. HRMS: calcd for $C_{18}H_{15}O_4Na$ $[M+Na]^+$, 295.0970, found 295.0982.

Procedure for reduction of 2, 5-Dihydrofuran 3a

A 2-necked flask was dried with heat gun, and cooled down in the Nitrogen atmosphere. To the flask, **3a** (0.2 mmol)and 5 mL THF was added and cooled down to 0 ^{o}C, then $LiAlH_4$ (0.4 mmol, 2.0 eq) was added in portions. After refluxed for 2 hrs, the reaction was cooled down and quenched with saturated NH_4Cl. The aqueous phase was extracted with ethyl acetate for 3 times. The combined organic phase was dried with anhydrous Na_2SO_4. The solvent was removed with vacuum rotator, and 0.2 g Silica gel was added to make the uniformed sample for the chromatography. The pure products were obtained with petrol ether and ethyl acetate (v/v = 8 : 1-5: 1) as elution.



8

Column chromatography afforded the desired product **8** in 54% yield as yellow solid: ^{1}HNMR(400 MHz, $CDCl_3$):δ7.21 – 7.08 (m, 10H), 6.11 (t, J = 1.6 Hz, 1H), 4.84 (dd, J_1= 13.4 Hz, J_2= 1.6 Hz, 1H), 4.79 (dd, J_1= 13.4 Hz, J_2= 1.6 Hz, 1H), 4.03 (br, 2 H), 2.20 (br, 1H).^{13}CNMR (100 MHz, CDCl3): 142.8, 141.4, 133.8, 128.3, 128.2, 127.9, 127.8,127.7, 126.0, 125.5, 94.4, 74.1, 65.9. HRMS: calcd for $C_{17}H_{16}O_2Na$ $[M+Na]^+$, 275.1048, found 275.1057.

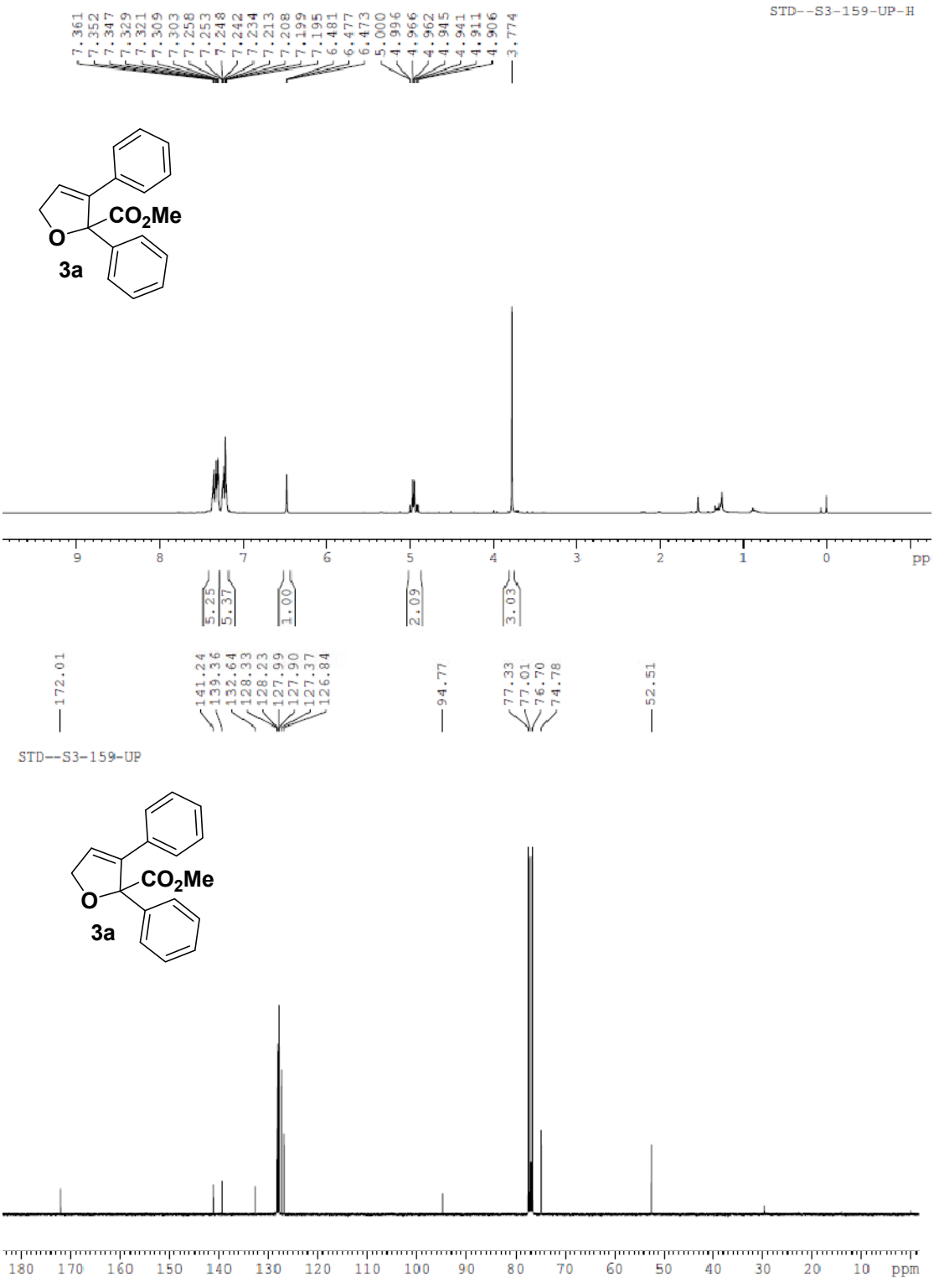
References

1. Y. Liang, Y.-X.Xie, and J.-H. Li, *J. Org. Chem.* 2006, **71**, 379.

2. (a) W. A. J.Starmans, L.Thijs and B.Zwanenburg, *Tetrahedron* 1998, **54**, 629; (b)M. K.-W.Choi, W.-Y. Yu, and C.-M.Che, *Org. Lett.* 2005, **7**, 1081; (c) W.-Y. Yu, Y.-T. Tsoi, Z. Y. Zhou, and A. S. C.Chan, *Org. Lett.* 2009, **11**, 469.

3. F. Bonadies and C. Bonini. *Syn. Comm.*, 1988, **18**, 1573.

NMR Spectra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7.234
7.213
7.208
7.199
7.195
6.481
6.477
6.473
5.000
4.996
4.966
4.962
4.945
4.941
4.911
4.906
3.774
CO2Me
O
3a
9
8
7
6
5
4
3
2
1
0
pp
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5.37
1.00
2.09
3.03
172.01
141.24
139.36
132.64
128.33
128.23
127.99
127.90
127.37
126.84
94.77
77.33
77.01
76.70
74.78
52.51
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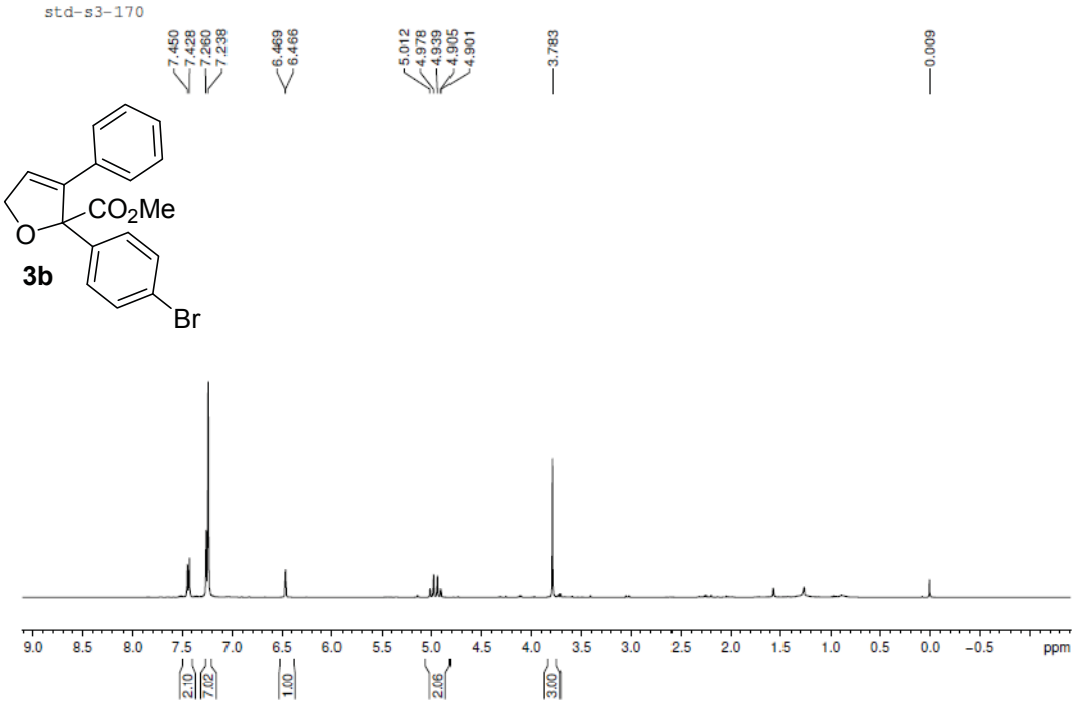
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3.783
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O
3b
Br
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8.0
7.5
7.0
6.5
6.0
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5.0
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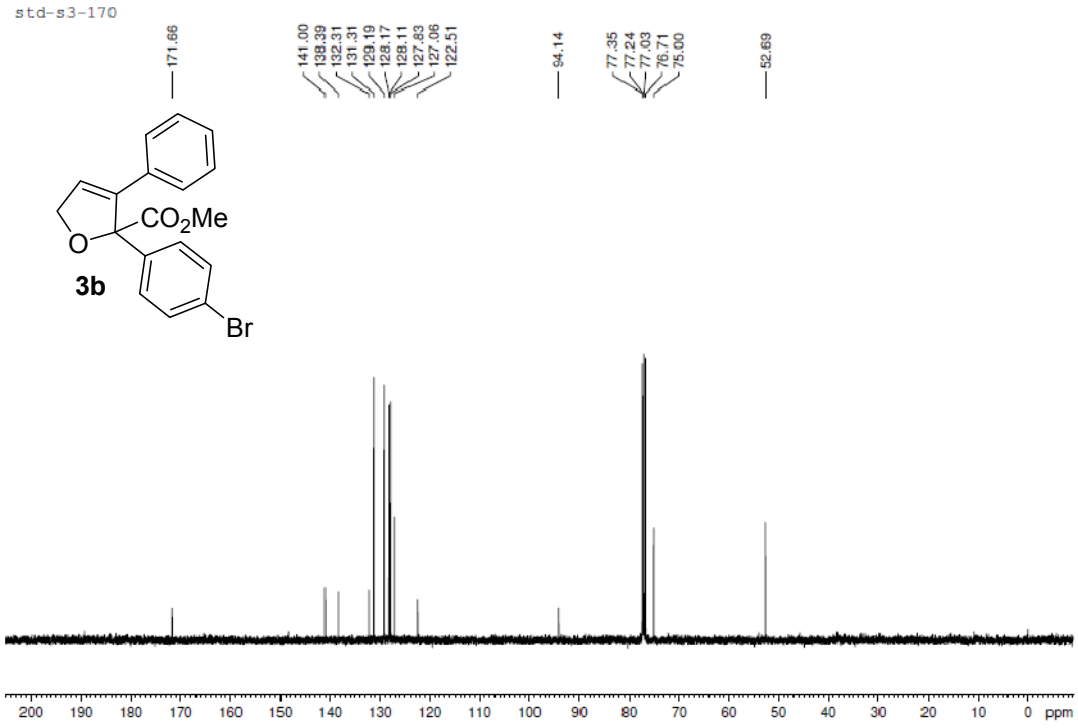
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76.71
75.00
52.69
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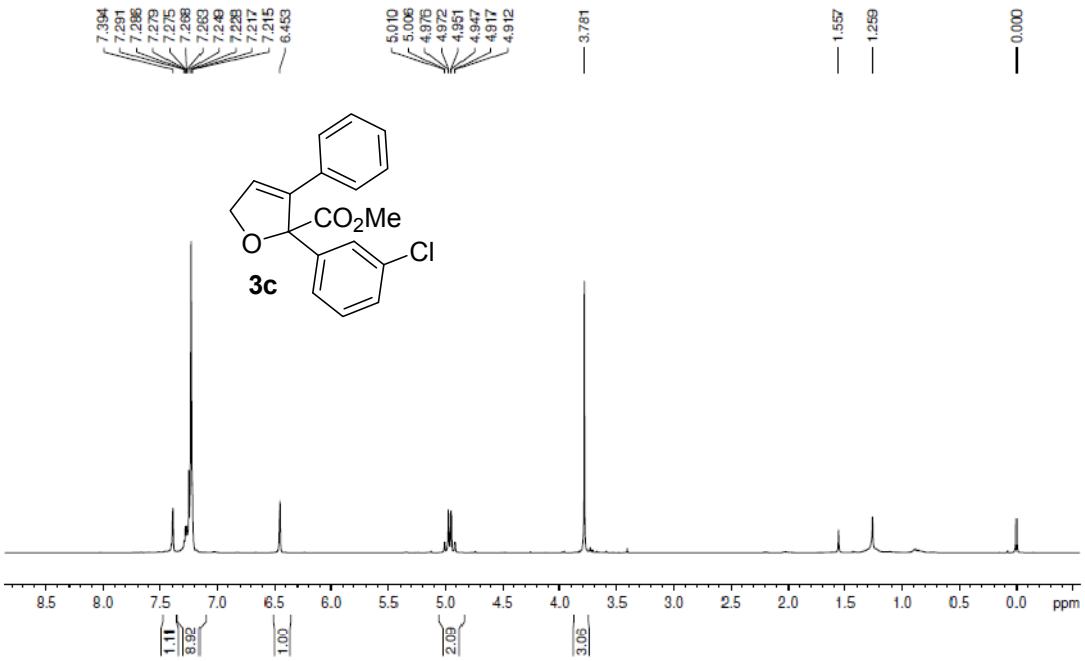

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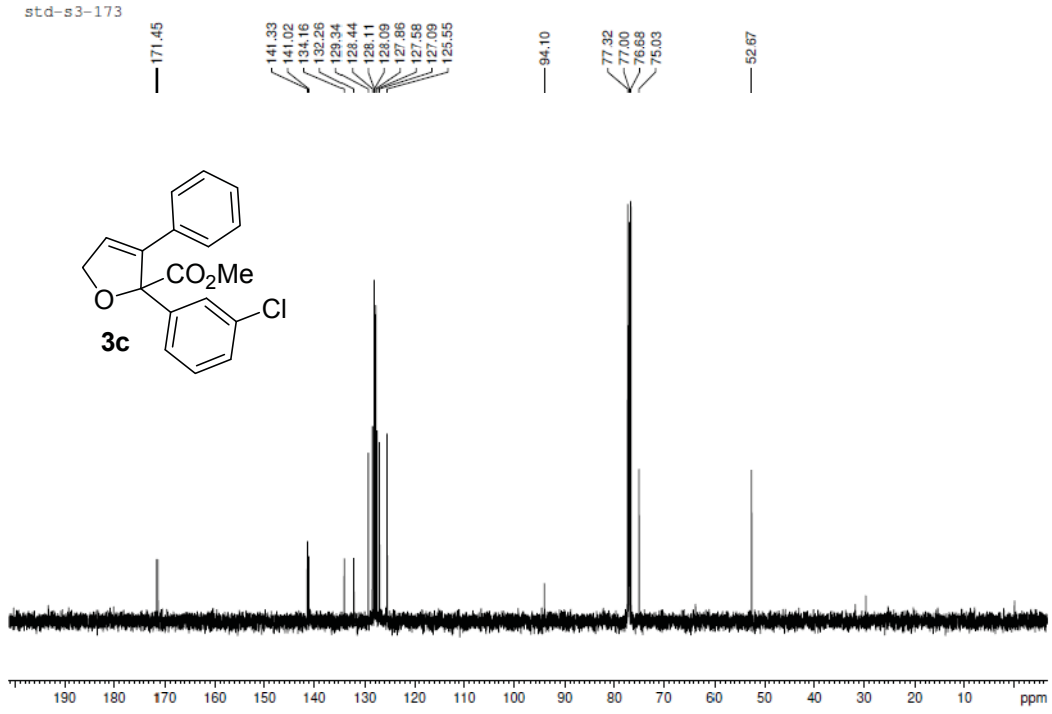

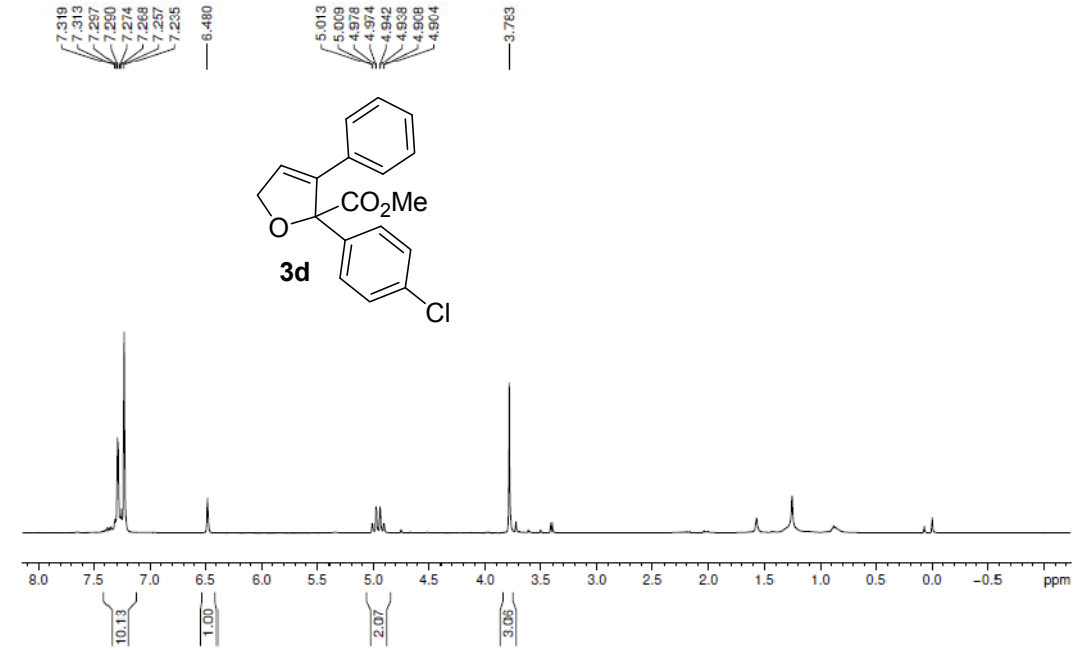
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4.942
4.938
4.908
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CO2Me
O
3d
Cl
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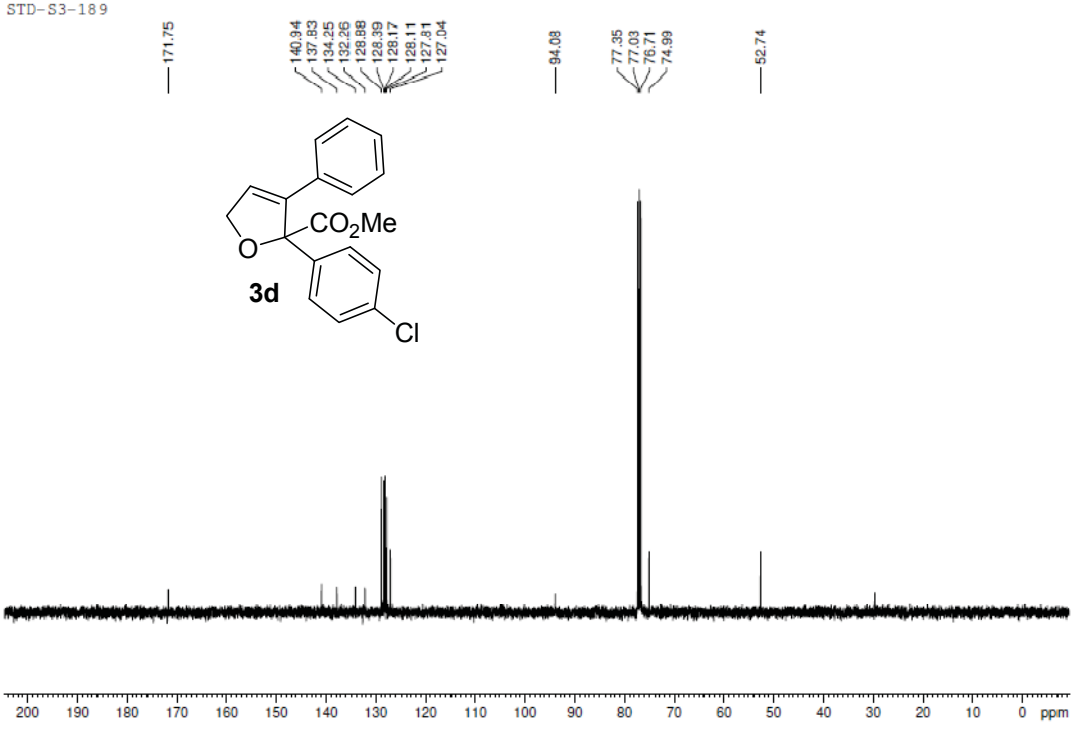
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77.03
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74.99
52.74
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O
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Cl
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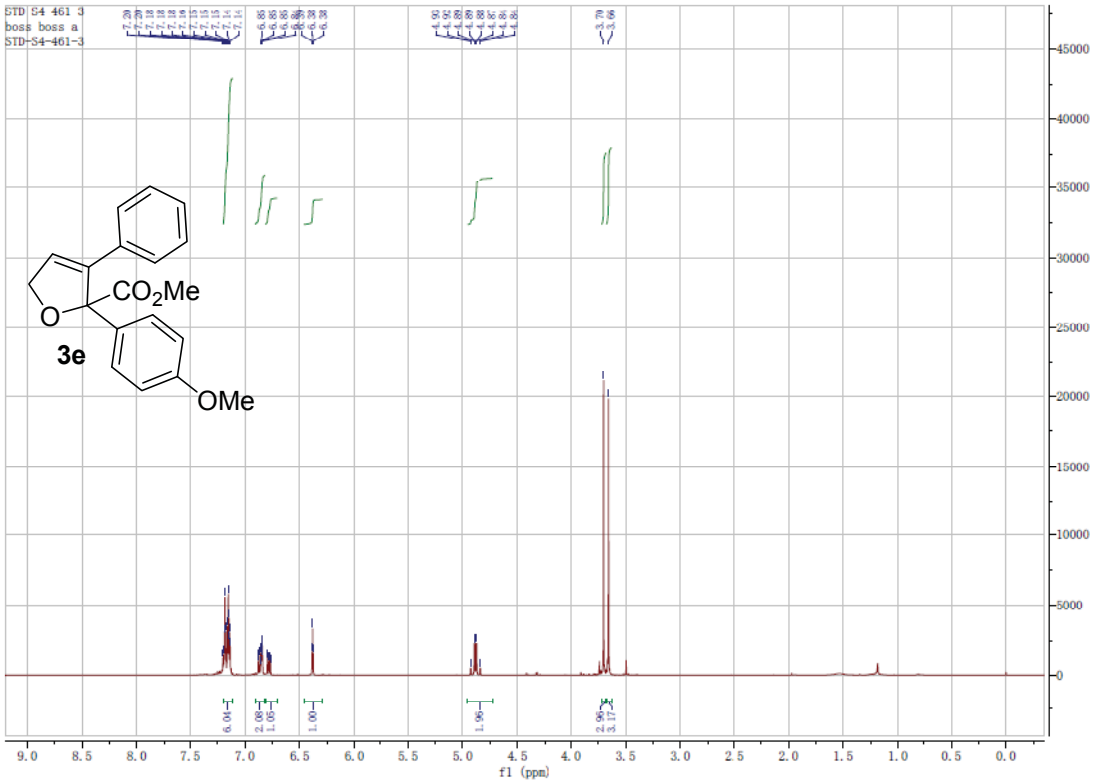
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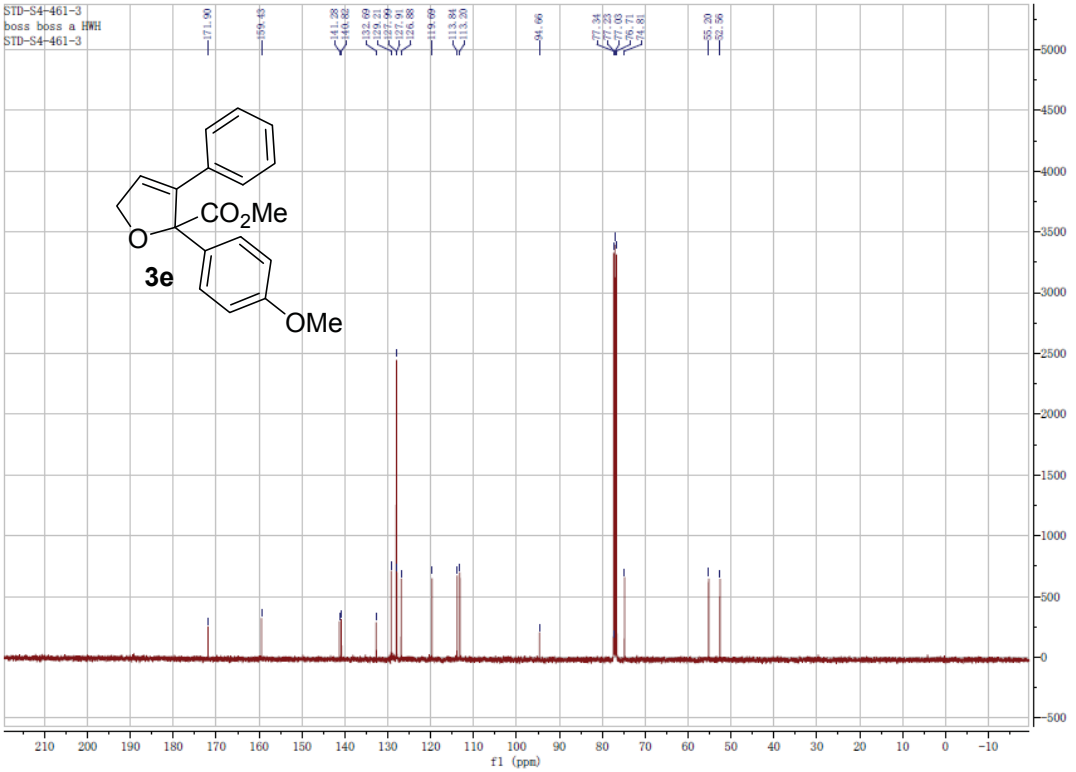
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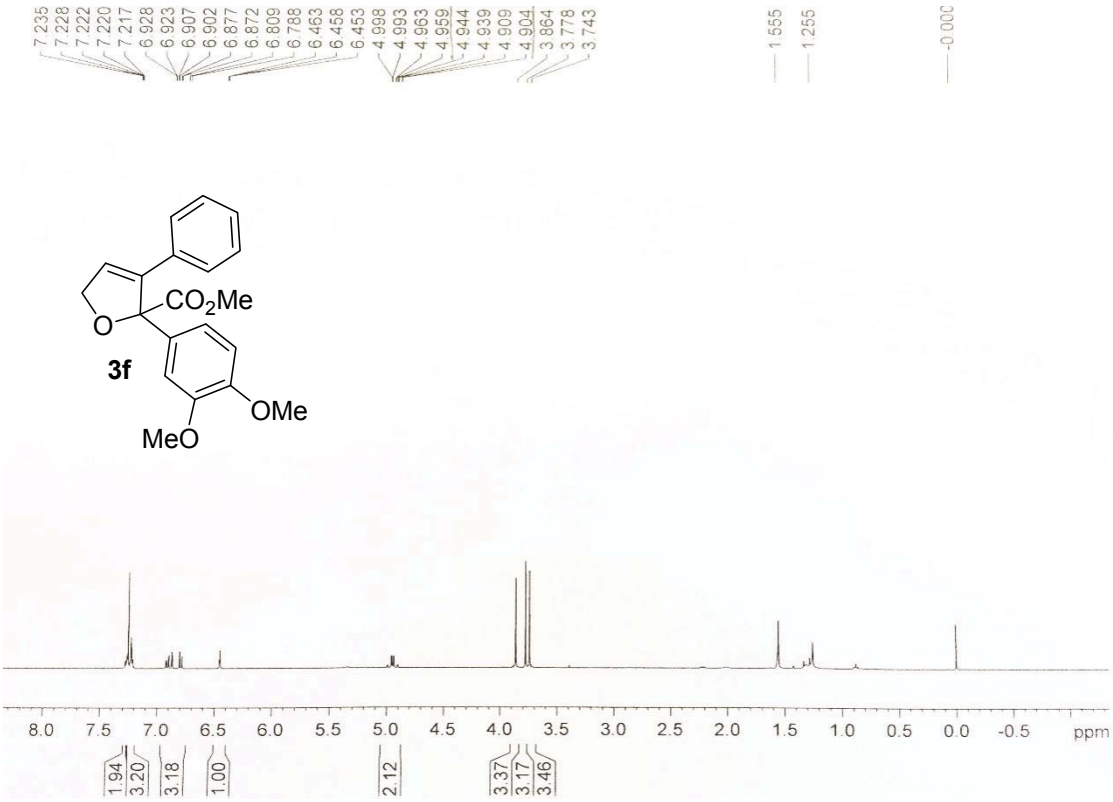
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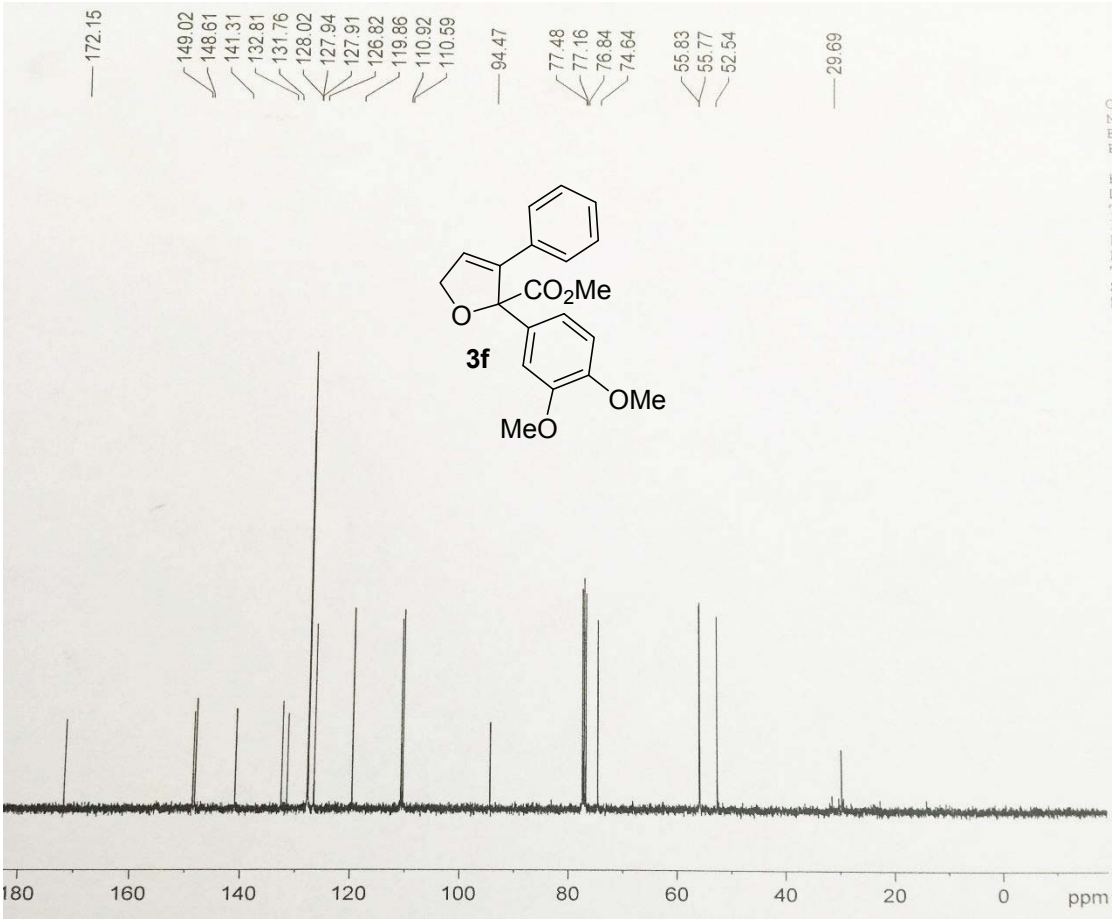
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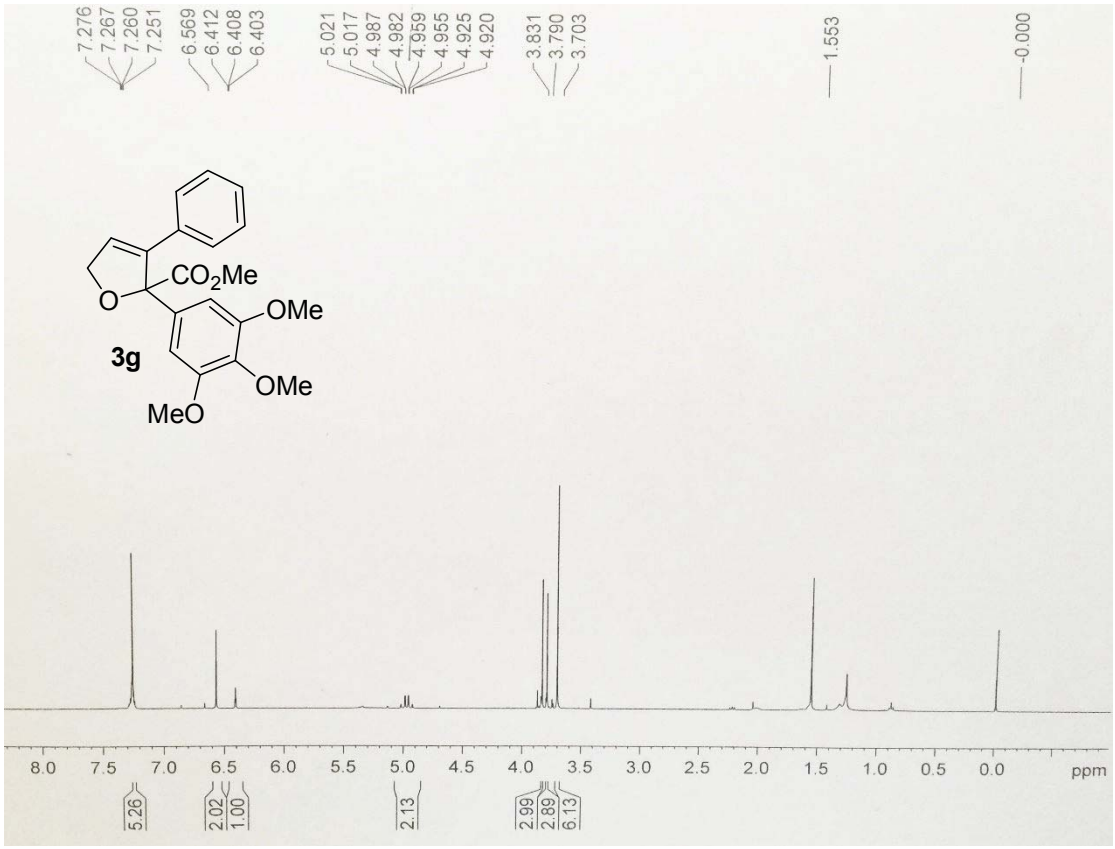
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OMe
OMe
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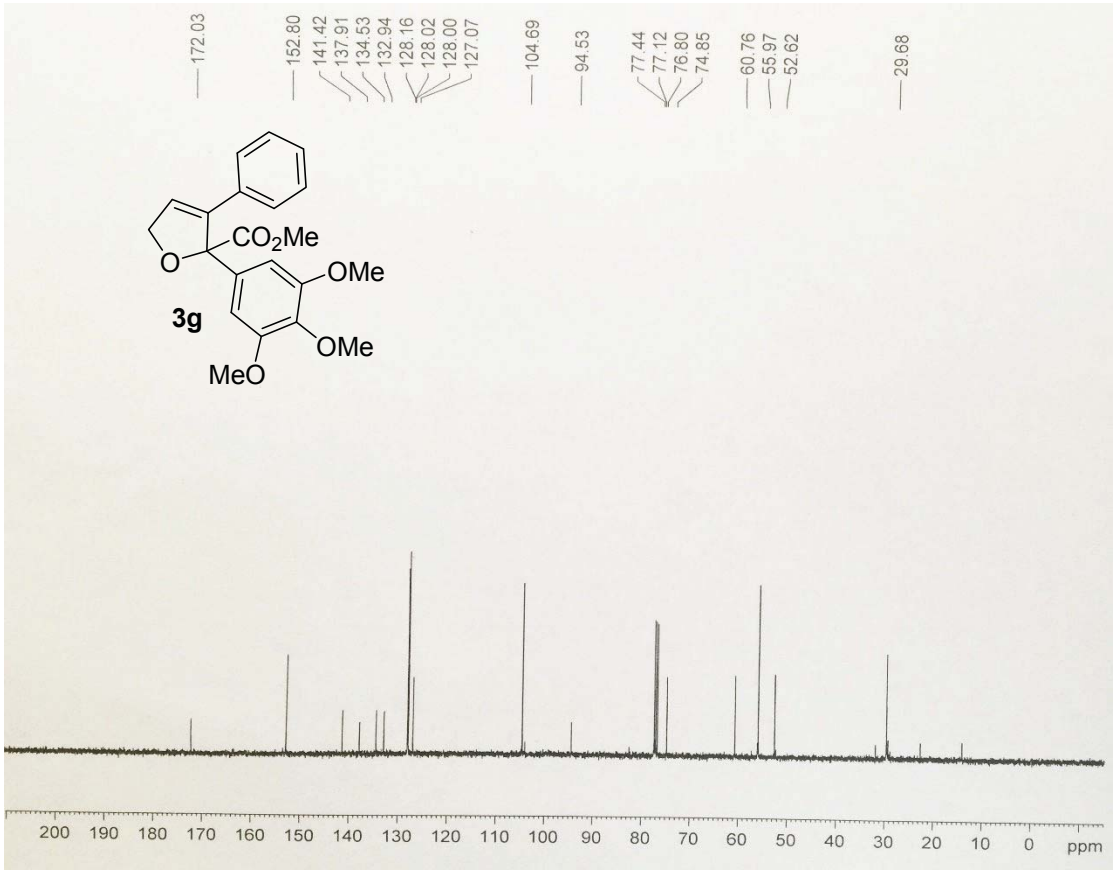
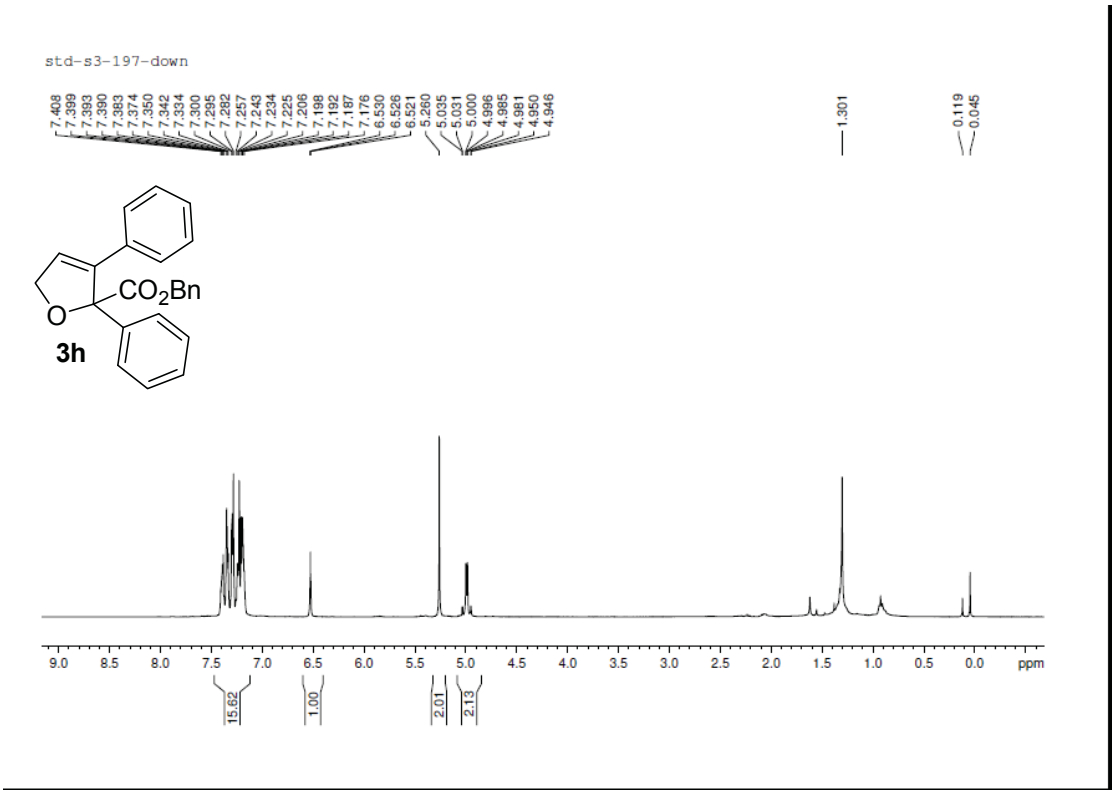
2.89
6.13


172.03
152.80
141.42
137.91
134.53
132.94
128.16
128.02
128.00
127.07
104.69
94.53
77.44
77.12
76.80
74.85
60.76
55.97
52.62
29.68
CO2Me
O
OMe
OMe
MeO
3g
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


std-s3-197-down
7.408
7.399
7.393
7.390
7.383
7.374
7.350
7.342
7.334
7.300
7.295
7.282
7.257
7.243
7.234
7.225
7.206
7.198
7.192
7.187
7.176
6.530
6.526
6.521
5.260
5.035
5.031
5.000
4.996
4.985
4.981
4.950
4.946
1.301
0.119
0.045
CO2Bn
O
3h
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
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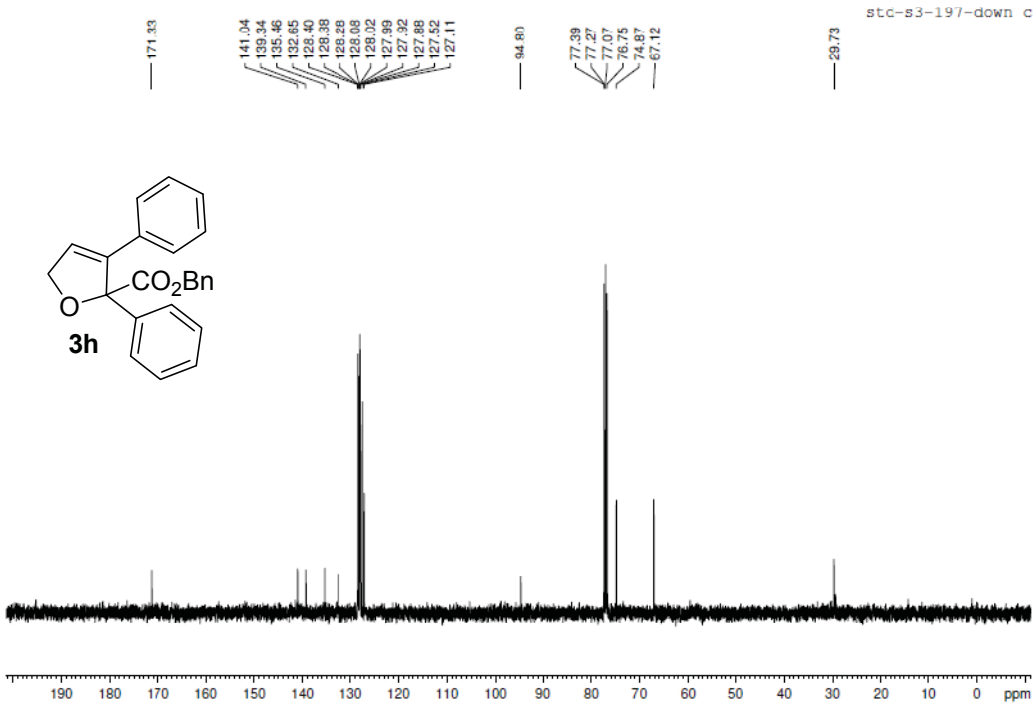
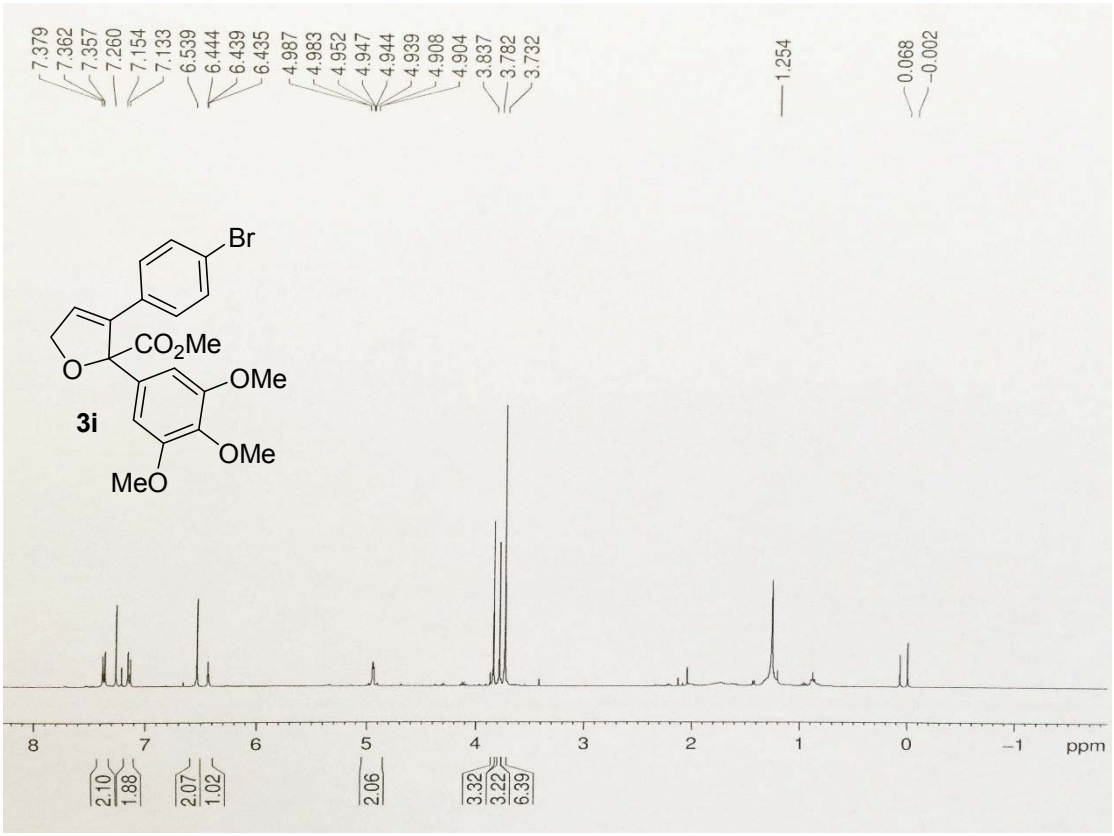
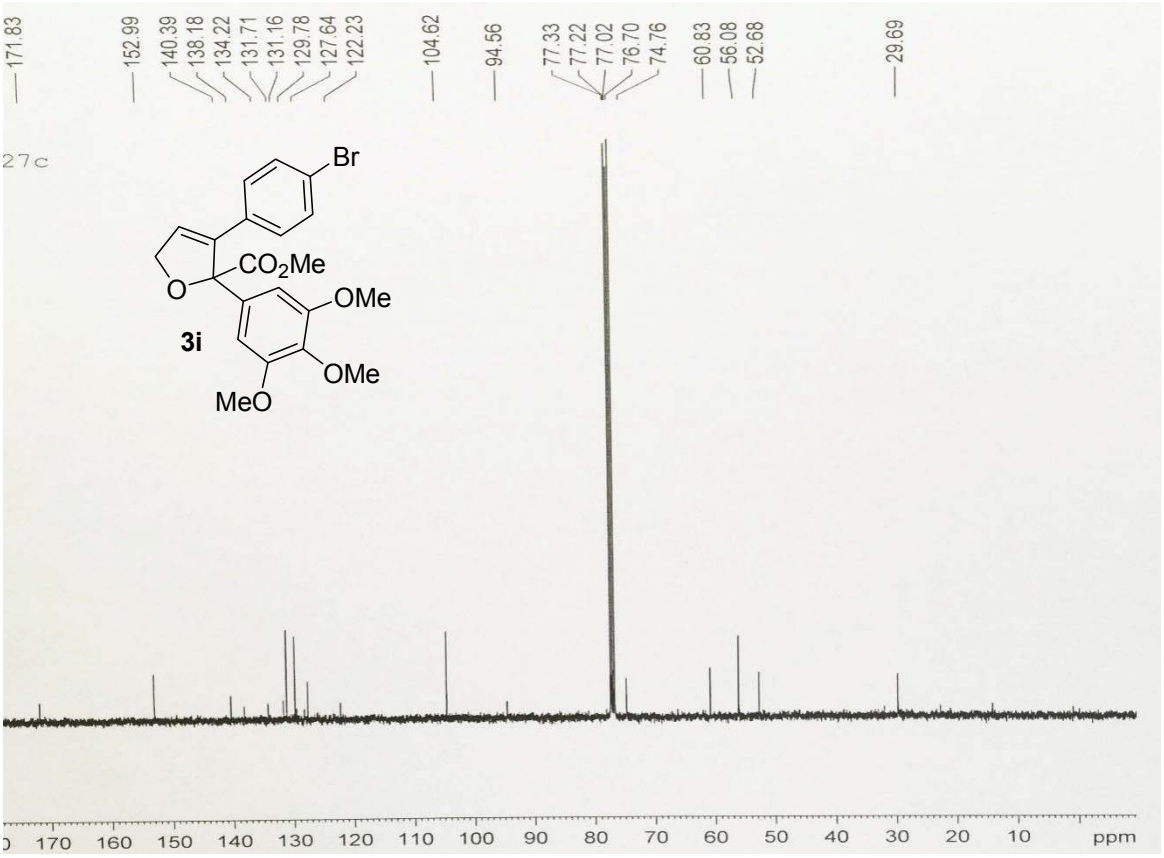
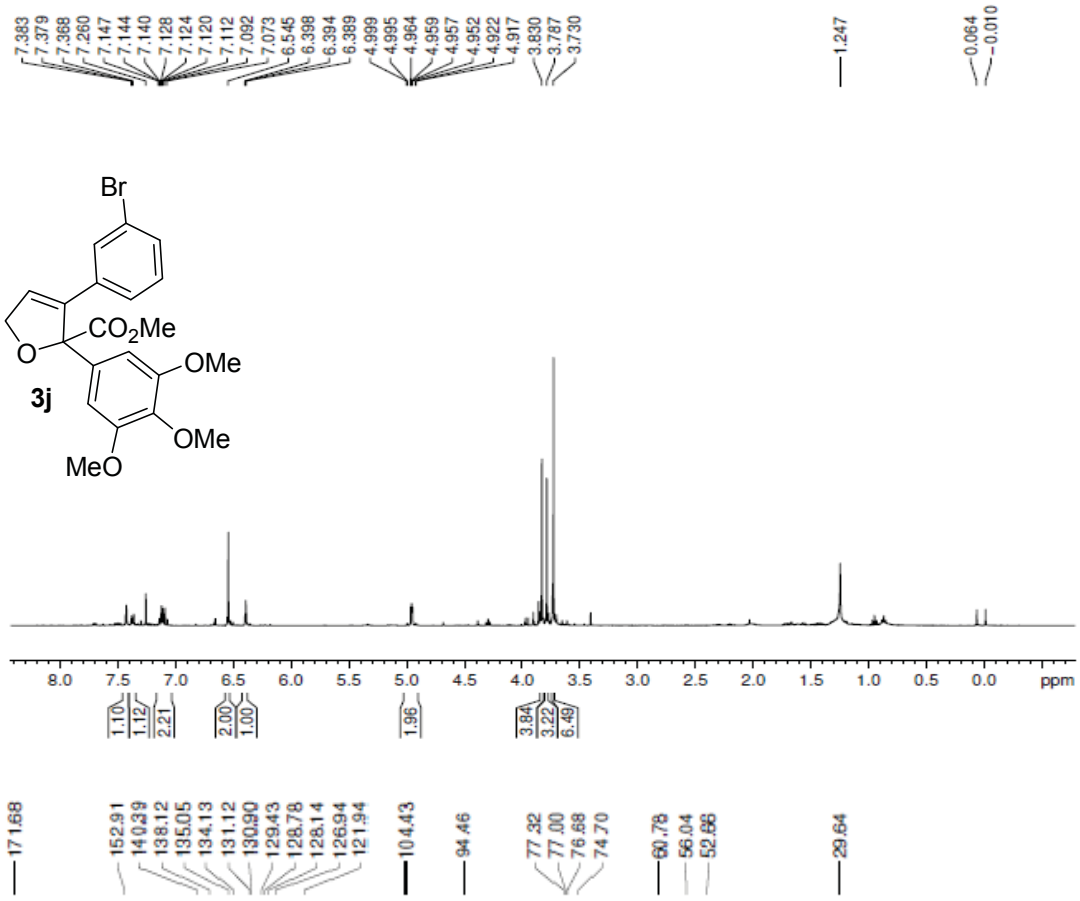
2.0
1.5
1.0
0.5
0.0
ppm
15.62
1.00
2.01
2.13

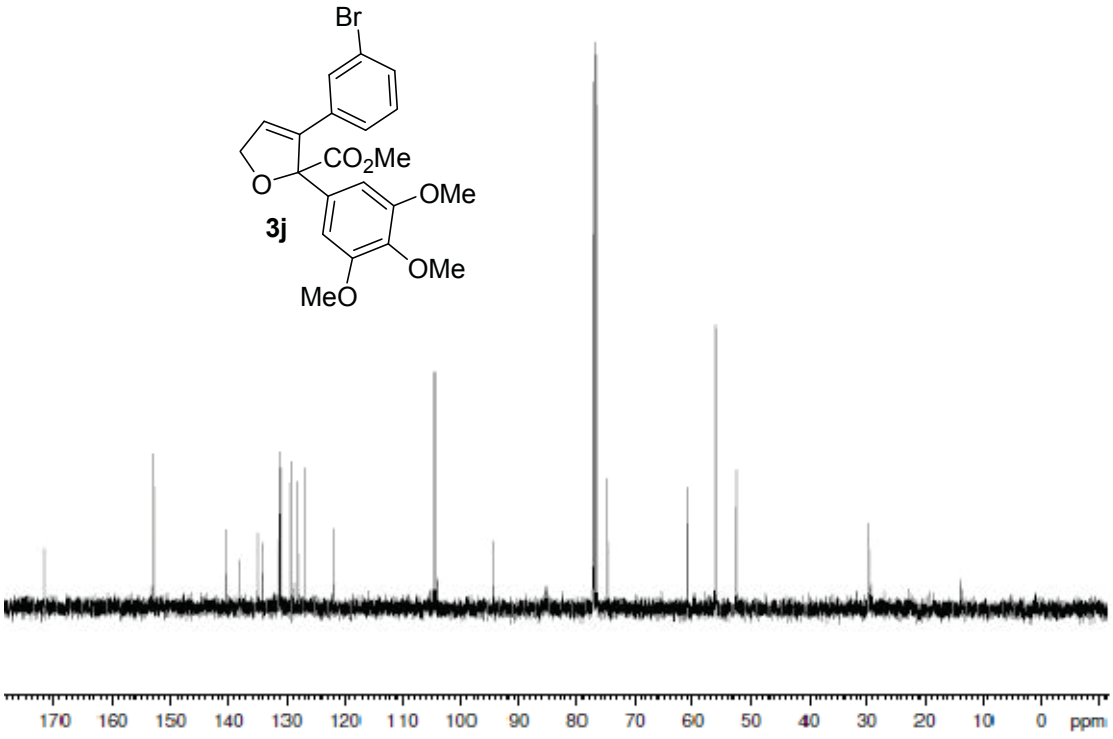

stc-s3-197-down c
171.33
141.04
139.34
135.46
132.65
128.40
128.38
128.28
128.08
128.02
127.99
127.92
127.88
127.52
127.11
94.80
77.39
77.27
77.07
76.75
74.87
67.12
29.73
CO2Bn
O
3h
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

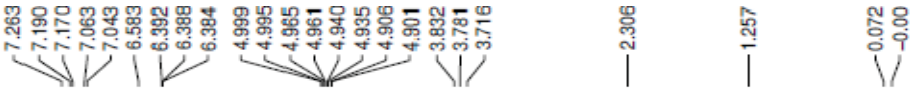

7.379
7.362
7.357
7.260
7.154
7.133
6.539
6.444
6.439
6.435
4.987
4.983
4.952
4.947
4.944
4.939
4.908
4.904
3.837
3.782
3.732
1.254
0.068
-0.002
Br
CO2Me
O
OMe
OMe
MeO
3i
8
7
6
5
4
3
2
1
0
−1
ppm
2.10
1.88
2.07
1.02
2.06
3.32
3.22
6.39

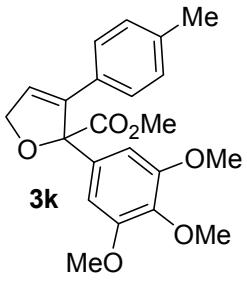

171.83
152.99
140.39
138.18
134.22
131.71
131.16
129.78
127.64
122.23
104.62
94.56
77.33
77.22
77.02
76.70
74.76
60.83
56.08
52.68
29.69
27c
Br
CO2Me
O
OMe
OMe
MeO
3i
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

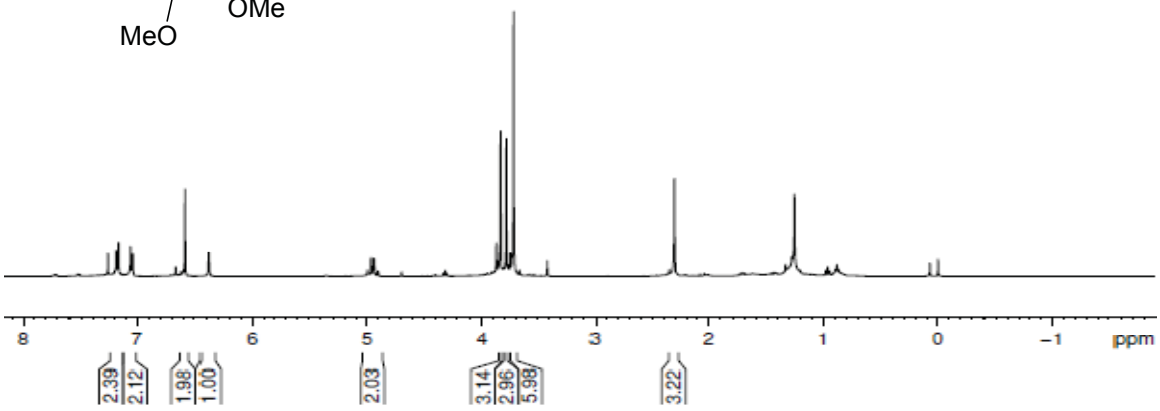

7.383
7.379
7.368
7.260
7.147
7.144
7.140
7.128
7.124
7.120
7.112
7.092
7.073
6.545
6.398
6.394
6.389
4.999
4.995
4.964
4.959
4.957
4.952
4.922
4.917
3.830
3.787
3.730
1.247
0.064
-0.010
Br
CO2Me
O
OMe
OMe
MeO
3j
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.10
1.12
2.21
2.00
1.00
1.96
3.84
3.22
6.49
171.68
152.91
140.39
138.12
135.05
134.13
131.12
130.90
129.43
128.78
128.14
126.94
121.94
104.43
94.46
77.32
77.00
76.68
74.70
60.78
56.04
52.66
29.64

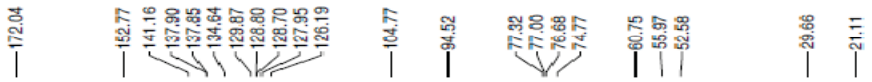
STD-S5-650 C


Br
CO2Me
O
OMe
OMe
MeO
3j
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

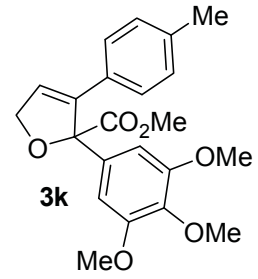


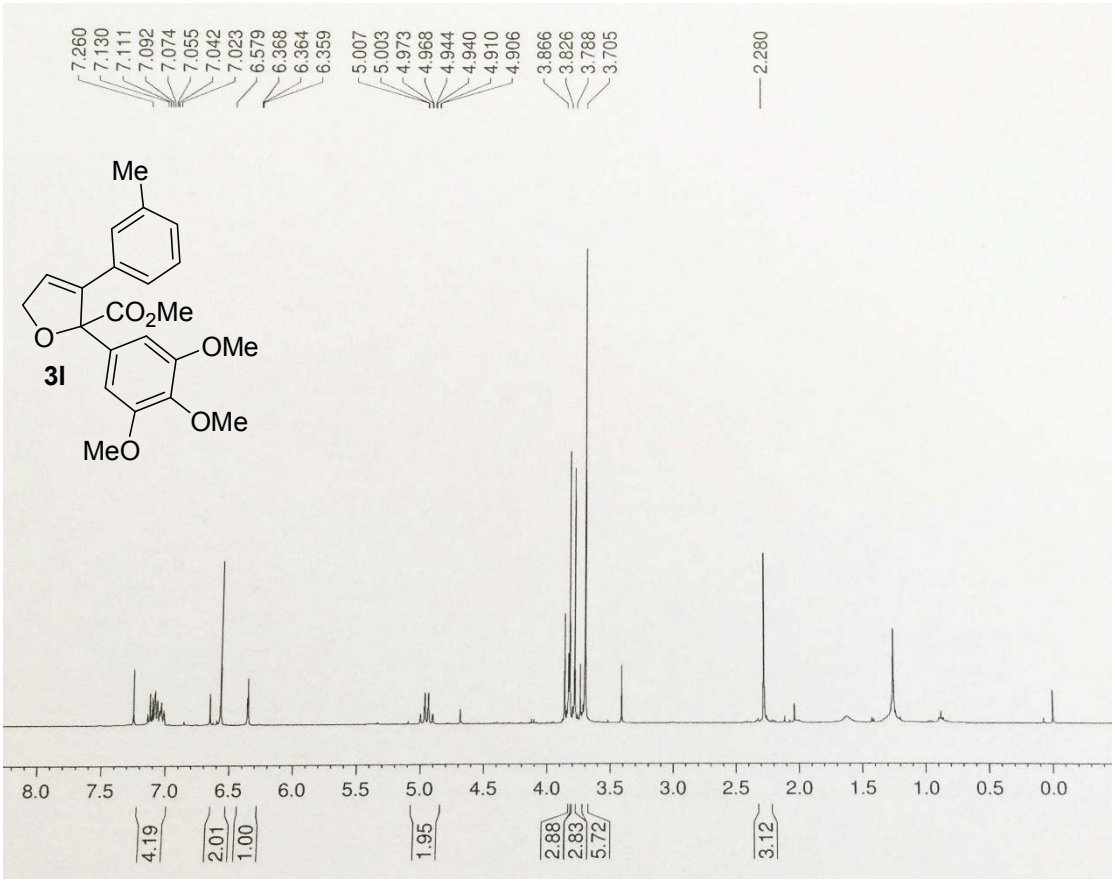






STD-S5-626-sec-C

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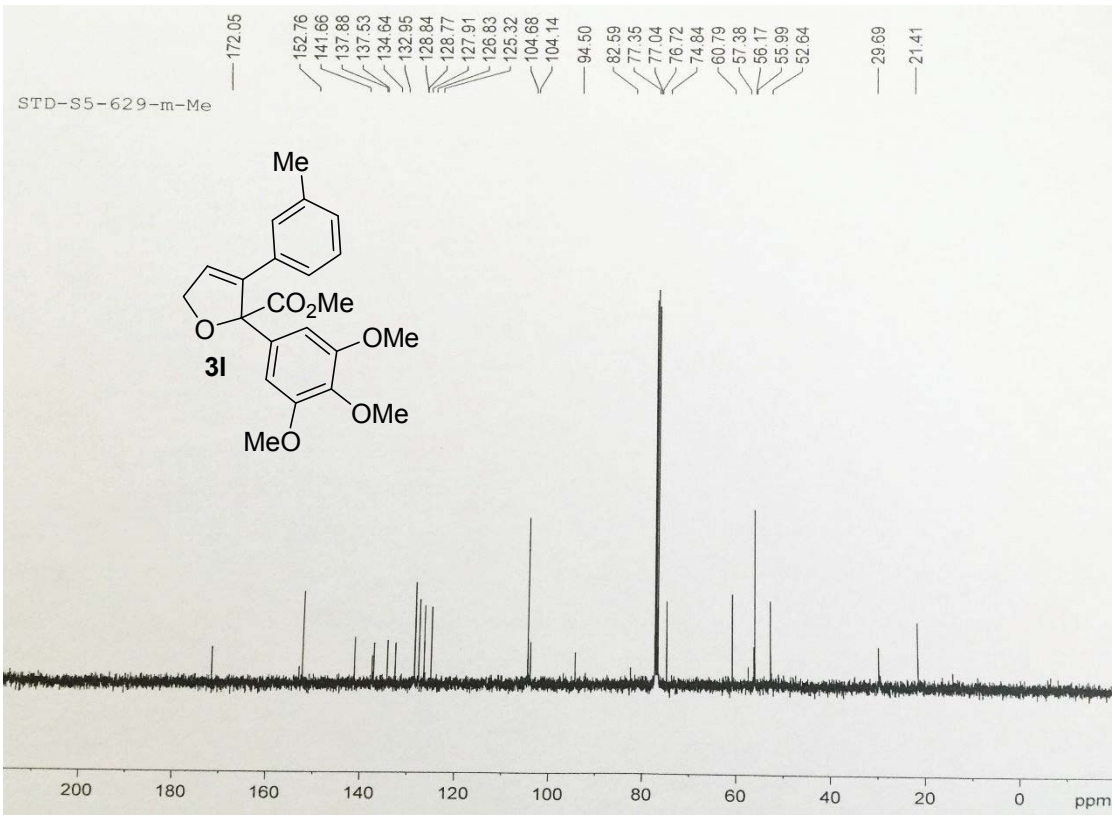
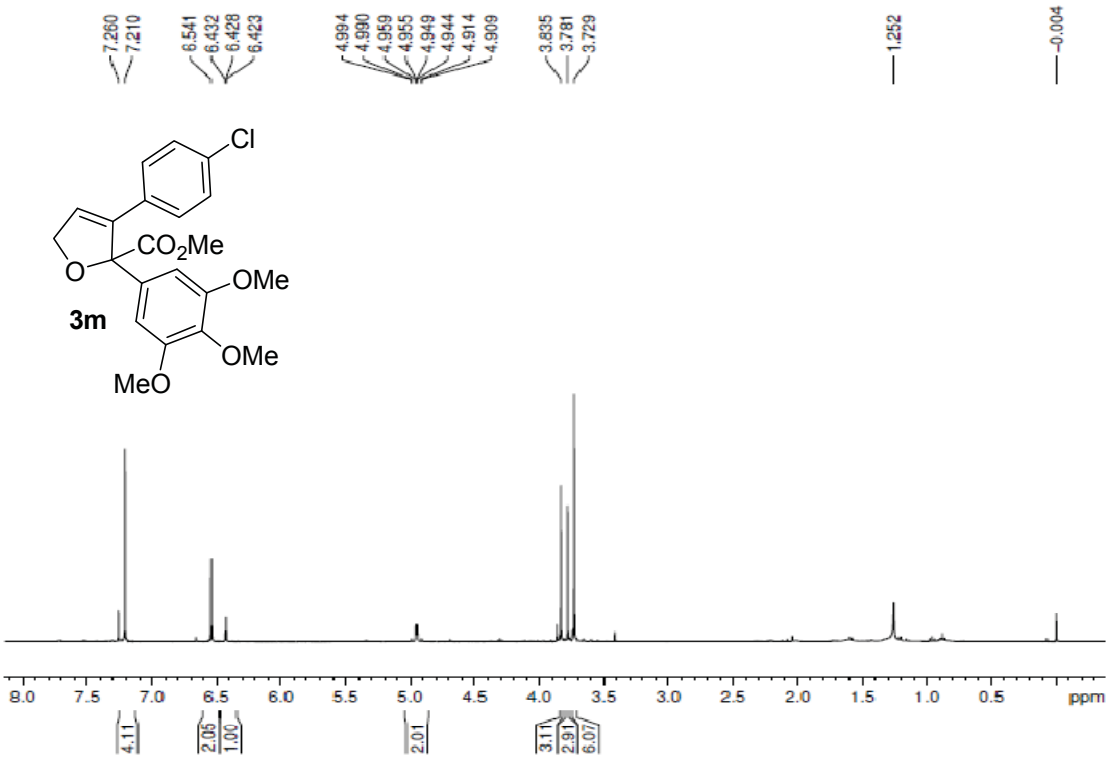
7.130
7.111
7.092
7.074
7.055
7.042
7.023
6.579
6.368
6.364
6.359
5.007
5.003
4.973
4.968
4.944
4.940
4.910
4.906
3.866
3.826
3.788
3.705
2.280
Me
CO2Me
O
OMe
OMe
MeO
3l
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
4.19
2.01
1.00
1.95
2.88
2.83
5.72
3.12


172.05
152.76
141.66
137.88
137.53
134.64
132.95
128.84
128.77
127.91
126.83
125.32
104.68
104.14
94.50
82.59
77.35
77.04
76.72
74.84
60.79
57.38
56.17
55.99
52.64
29.69
21.41
STD-S5-629-m-Me
Me
CO2Me
O
OMe
OMe
MeO
3l
200
180
160
140
120
100
80
60
40
20
0
ppm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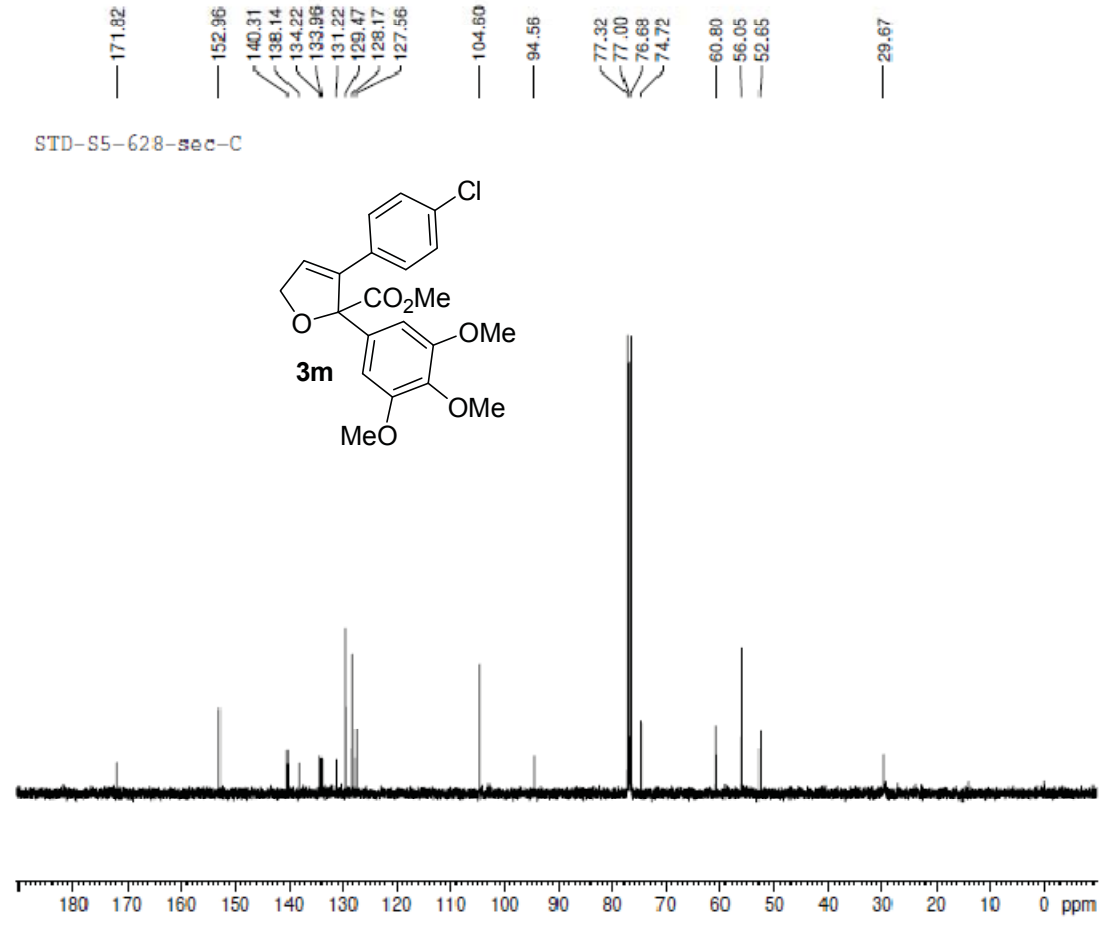
7.210
6.541
6.432
6.428
6.423
4.994
4.990
4.959
4.955
4.949
4.944
4.914
4.909
3.835
3.781
3.729
1.252
-0.004
Cl
CO2Me
O
OMe
OMe
MeO
3m
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
4.11
2.06
1.00
2.01
3.11
2.91
6.07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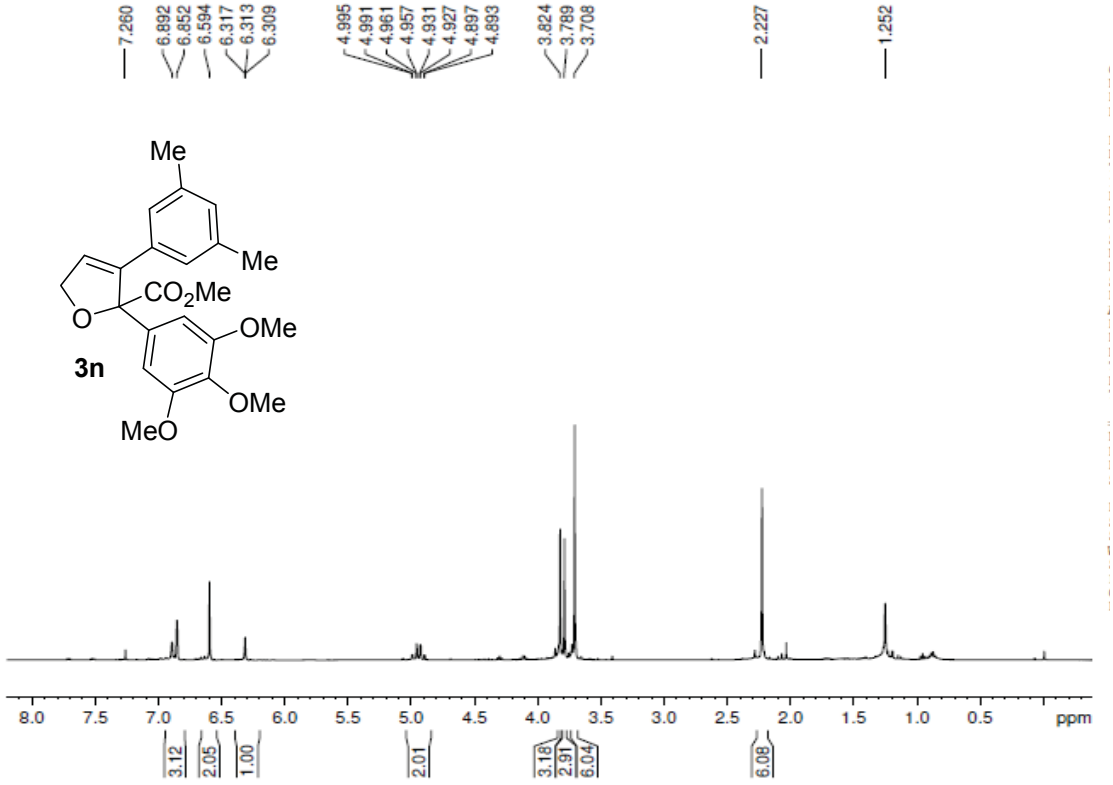
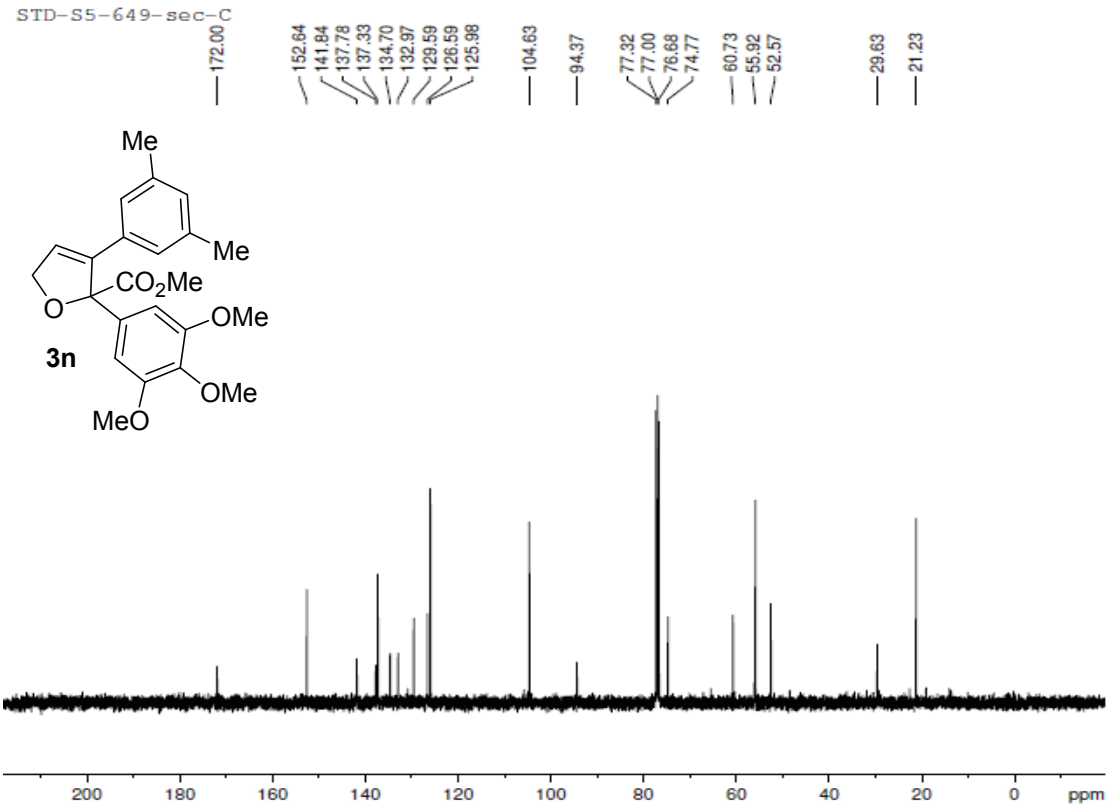
140.31
138.14
134.22
133.96
131.22
129.47
128.17
127.56
104.60
94.56
77.32
77.00
76.68
74.72
60.80
56.05
52.65
29.67
STD-S5-628-sec-C
Cl
CO2Me
O
OMe
OMe
MeO
3m
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


7.260
6.892
6.852
6.594
6.317
6.313
6.309
4.995
4.991
4.961
4.957
4.931
4.927
4.897
4.893
3.824
3.789
3.708
2.227
1.252
Me
Me
CO2Me
O
OMe
OMe
MeO
3n
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
3.12
2.05
1.00
2.01
3.18
2.91
6.04
6.08


STD-S5-649-sec-C
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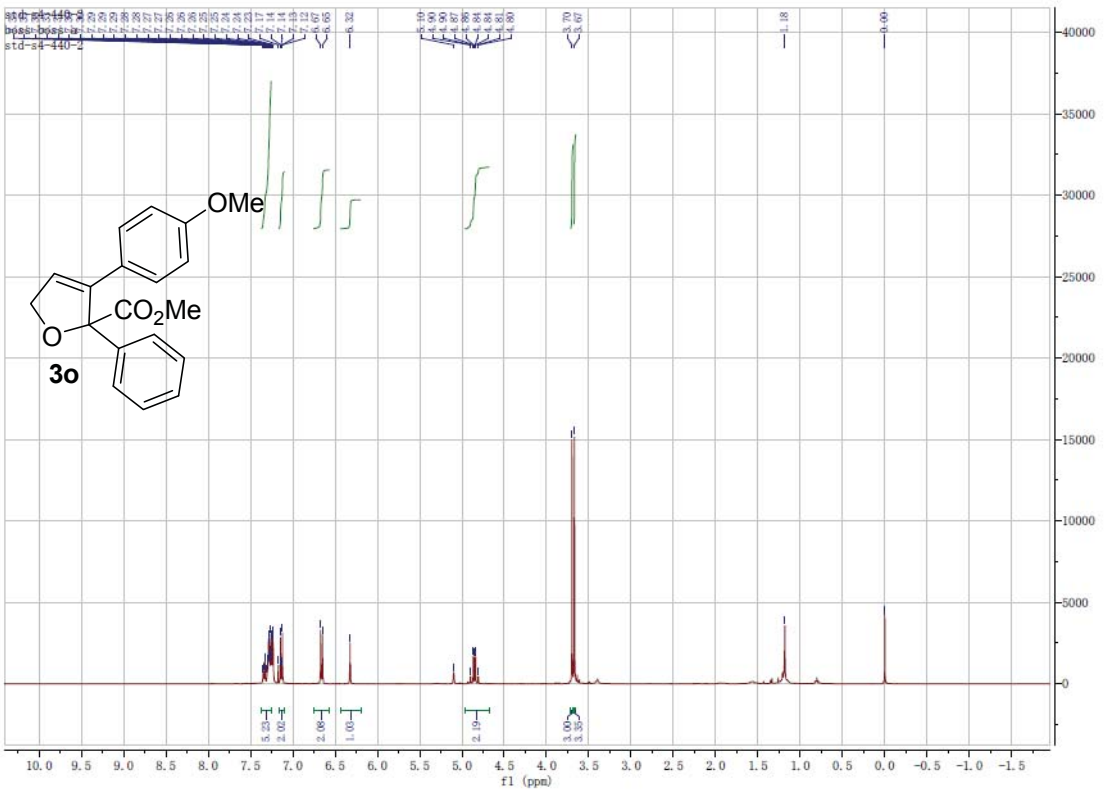
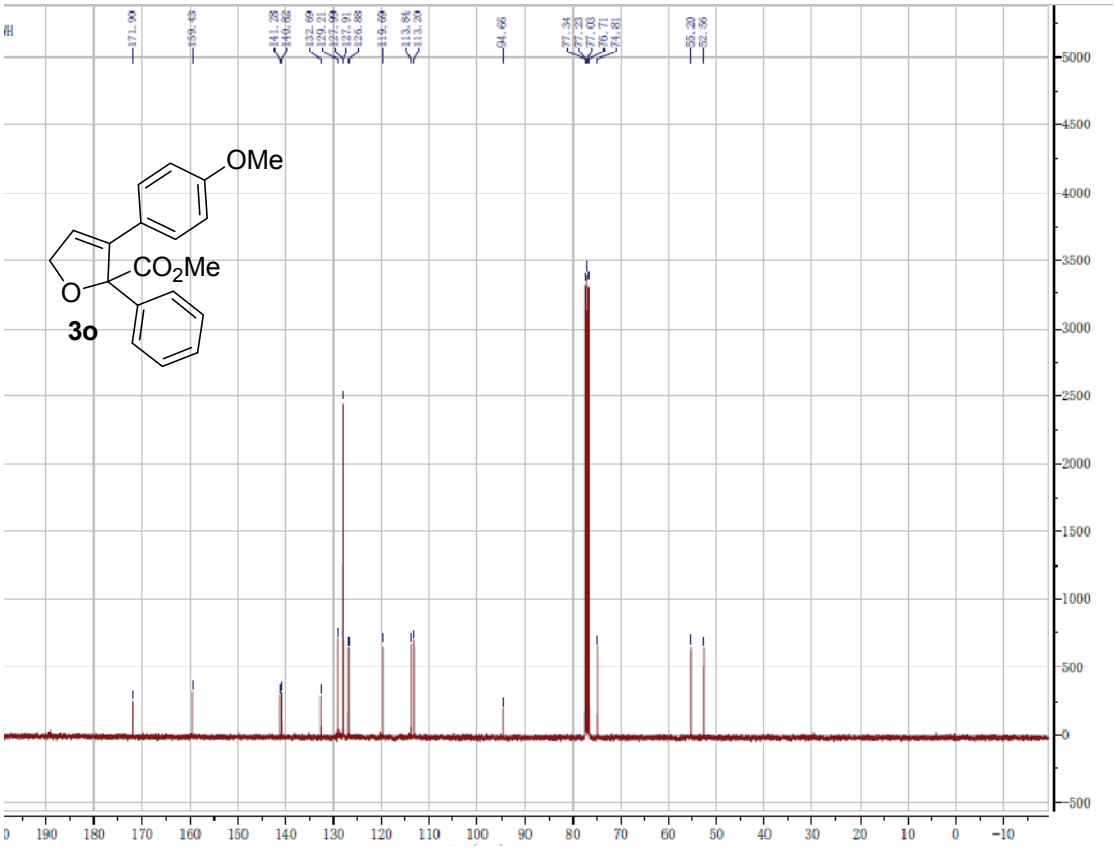
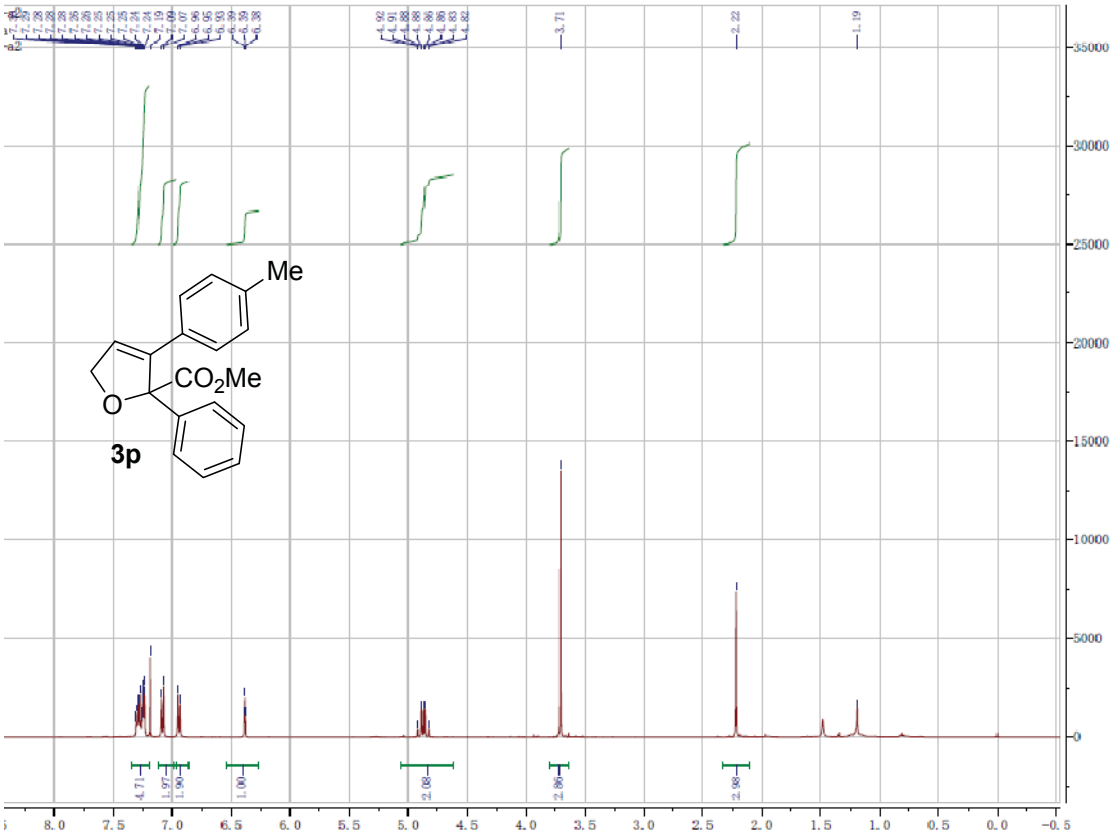
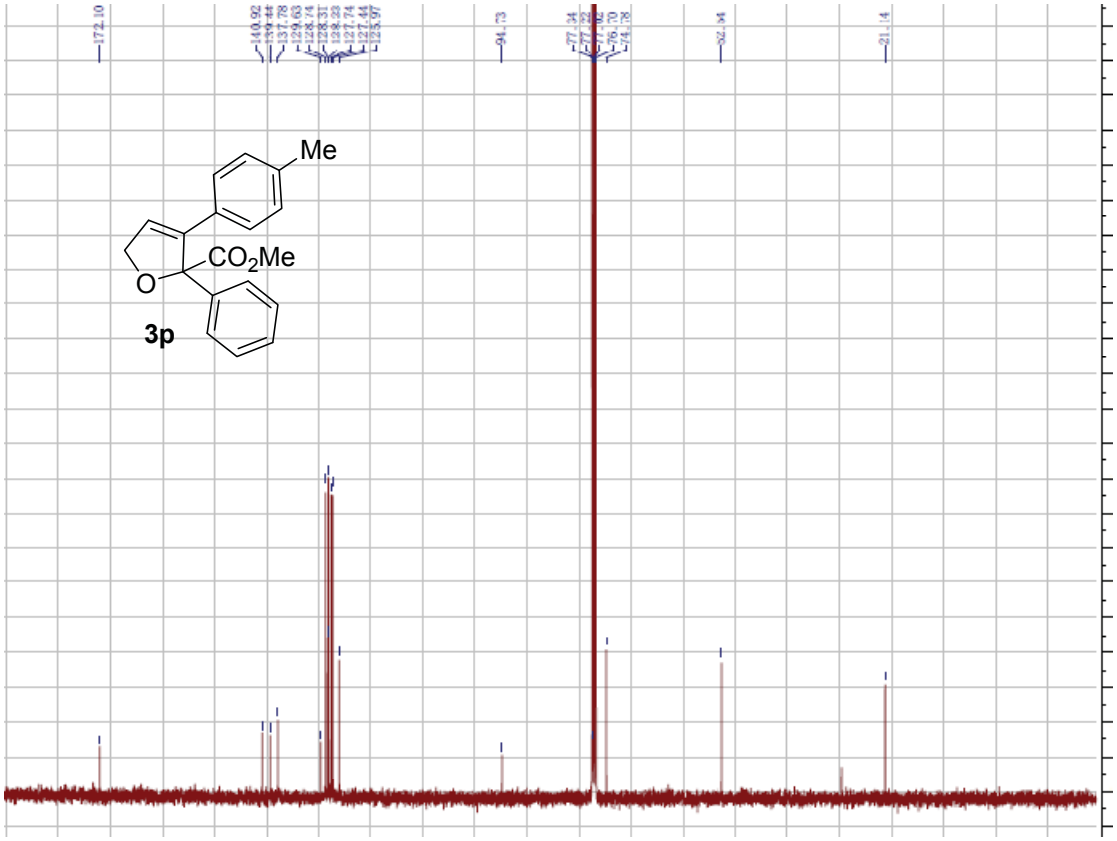
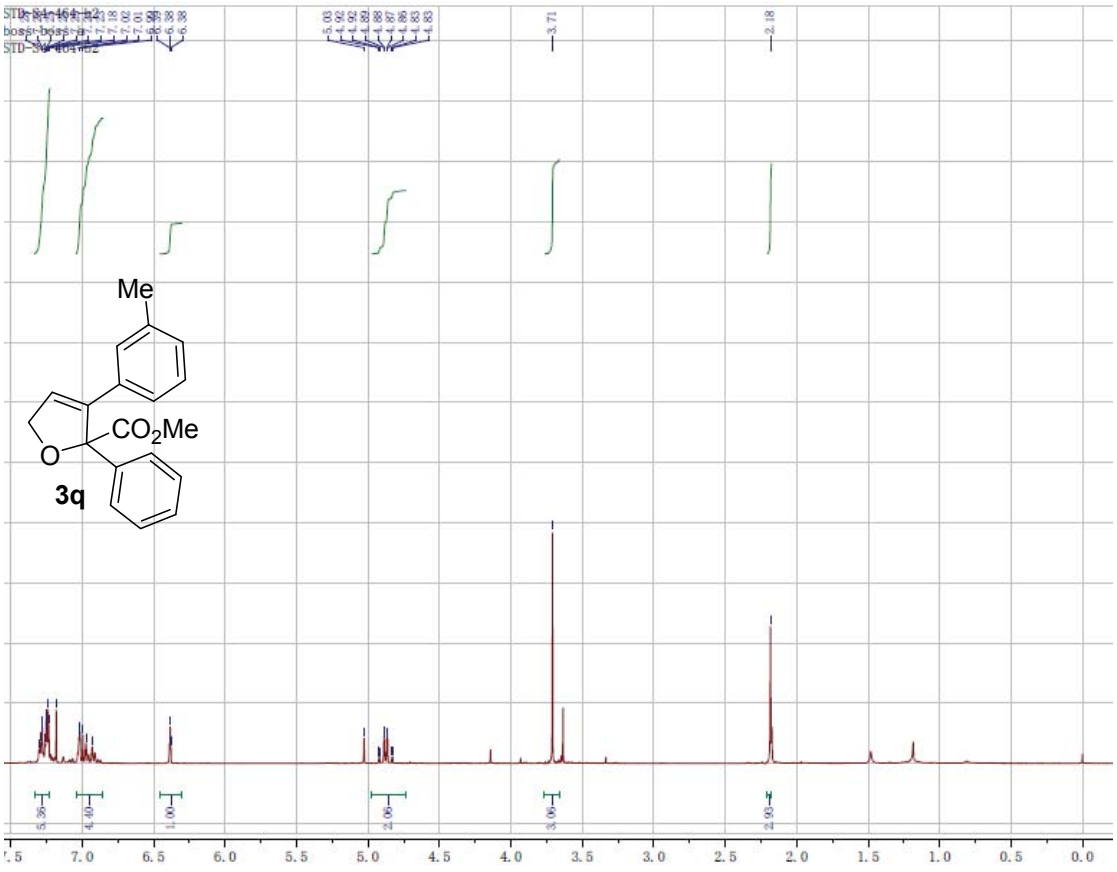
137.78
137.33
134.70
132.97
129.59
126.59
125.98
104.63
94.37
77.32
77.00
76.68
74.77
60.73
55.92
52.57
29.63
21.23
Me
Me
CO2Me
O
OMe
OMe
MeO
3n
200
180
160
140
120
100
80
60
40
20
0
ppm

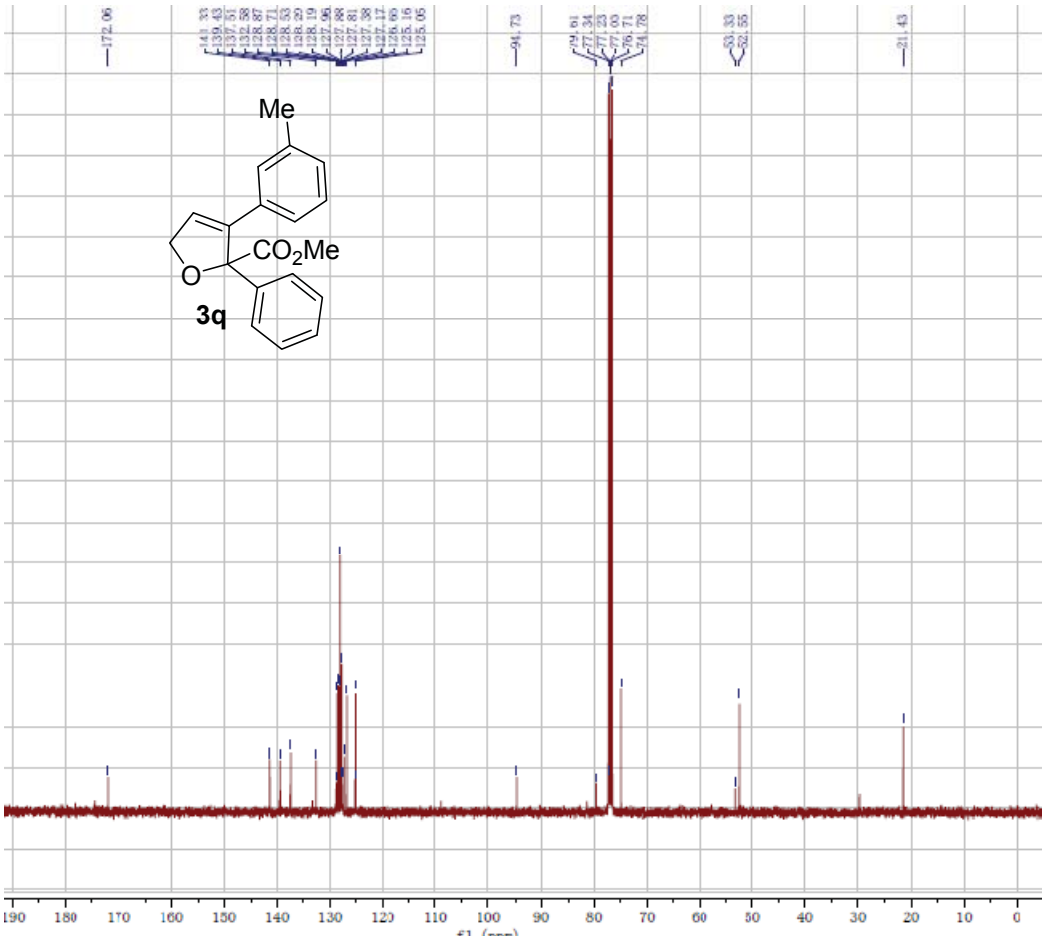

OMe
CO2Me
O
3o
f1 (ppm)

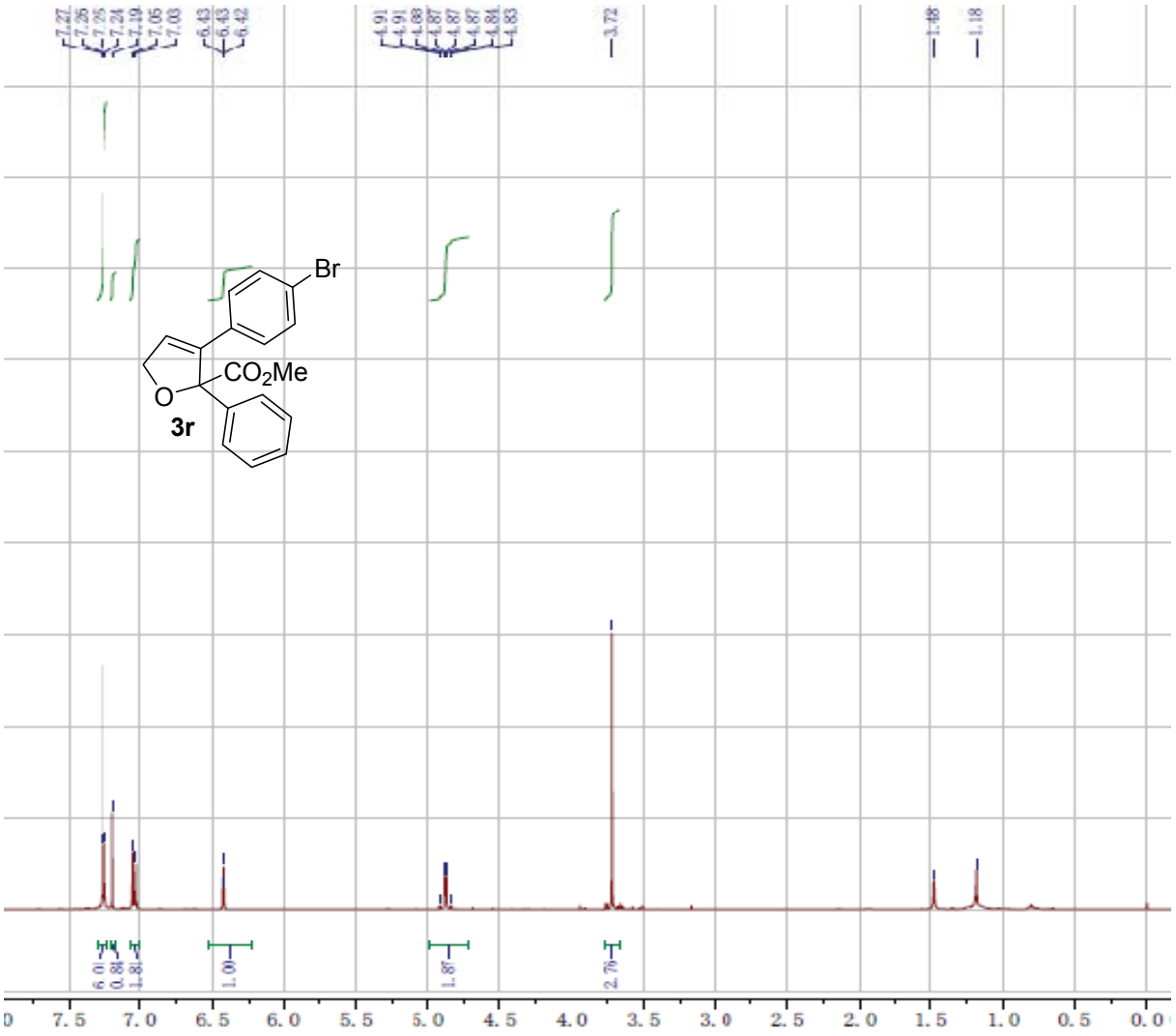

OMe
CO2Me
O
3o

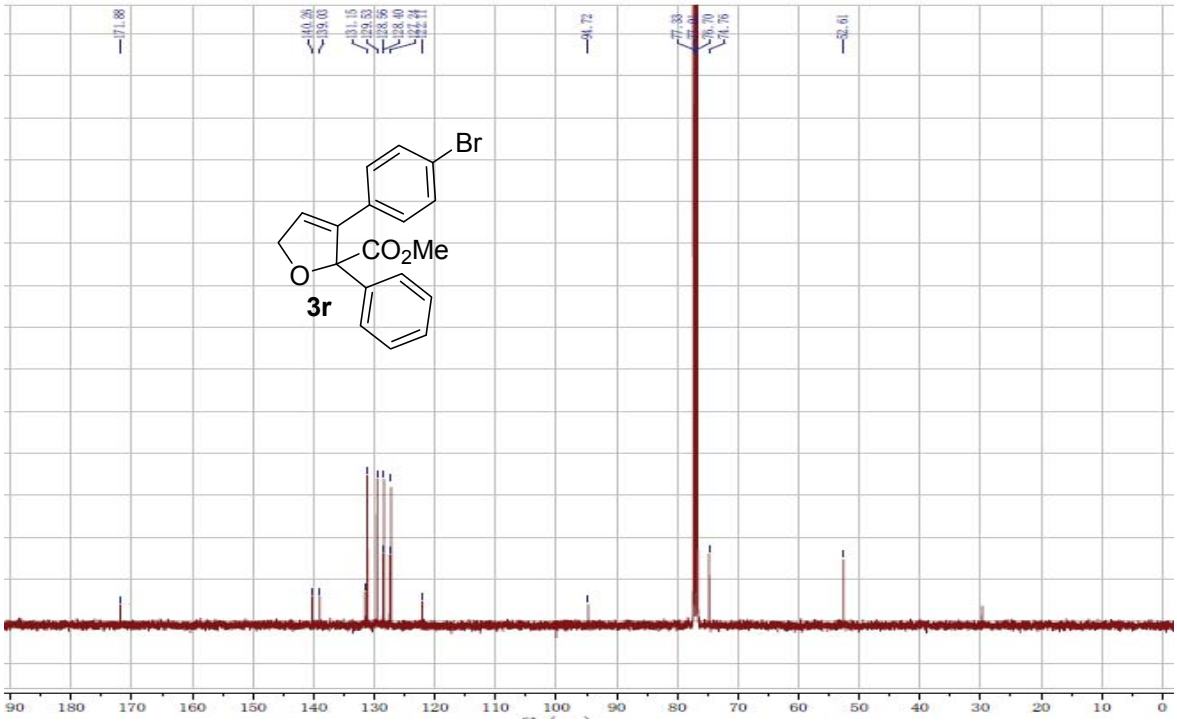

Me
CO2Me
O
3p

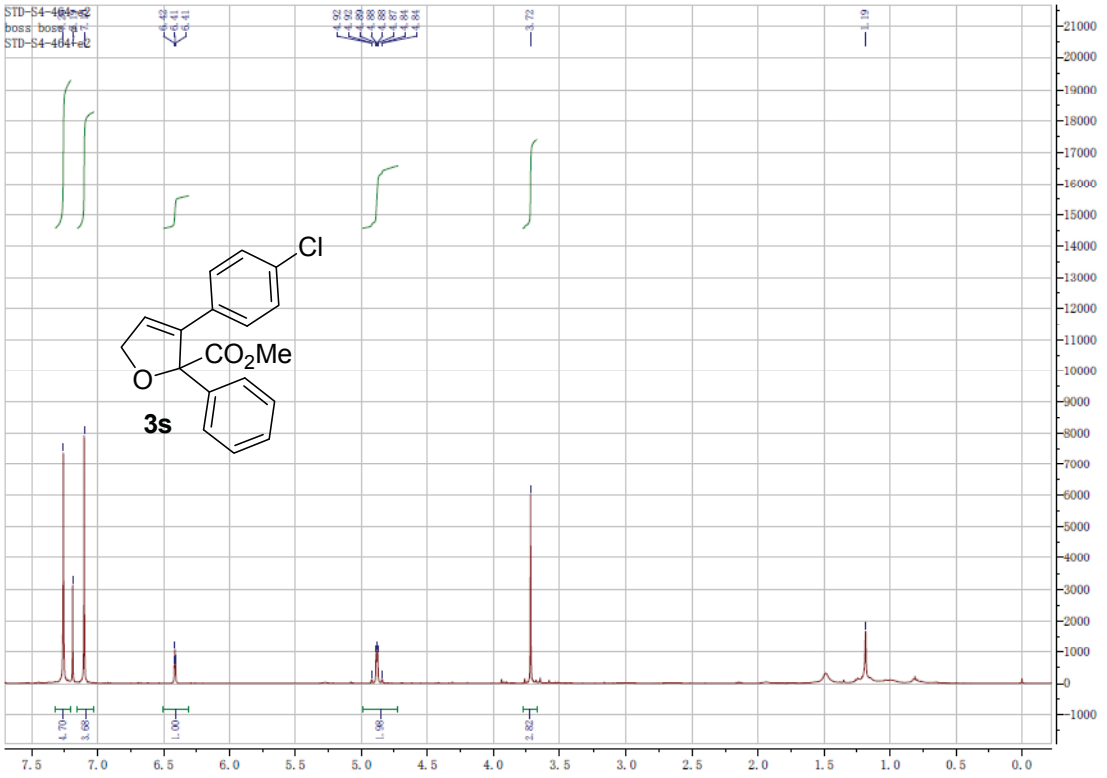

Me
CO2Me
O
3p

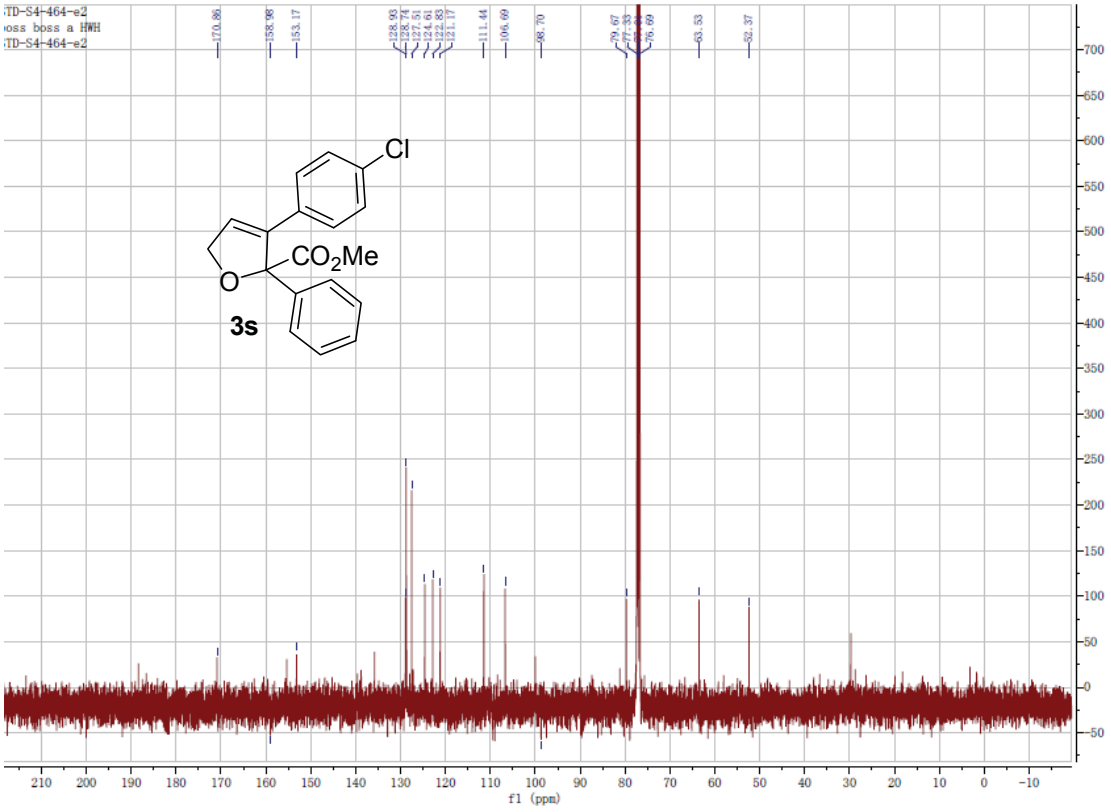

Me
CO2Me
O
3q

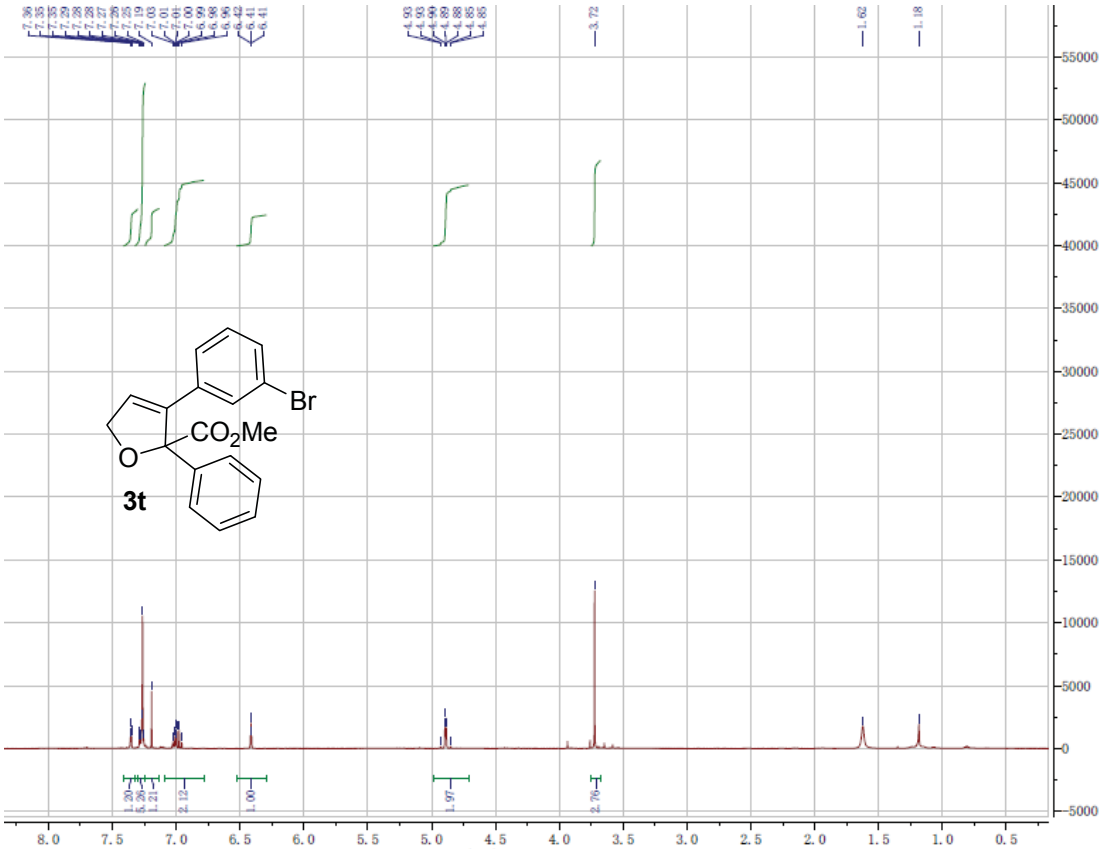

Me
CO2Me
O
3q

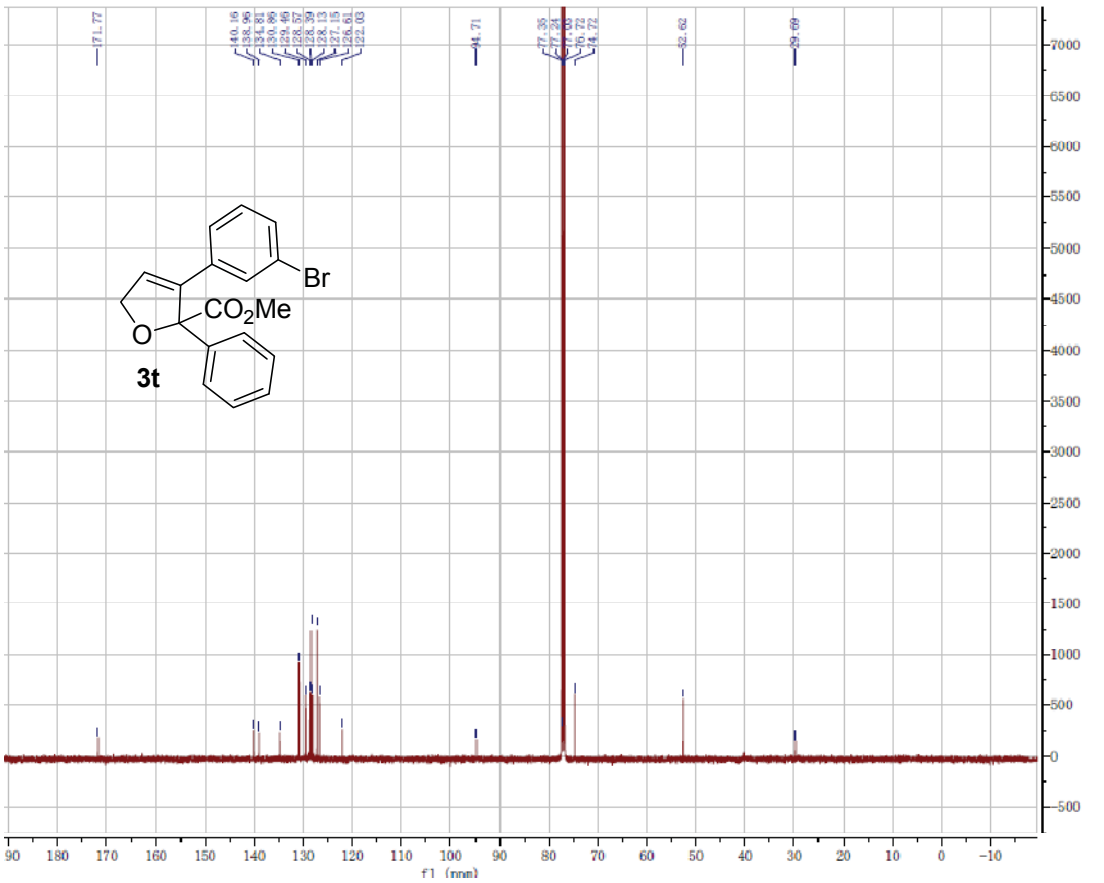

Br
CO2Me
O
3r

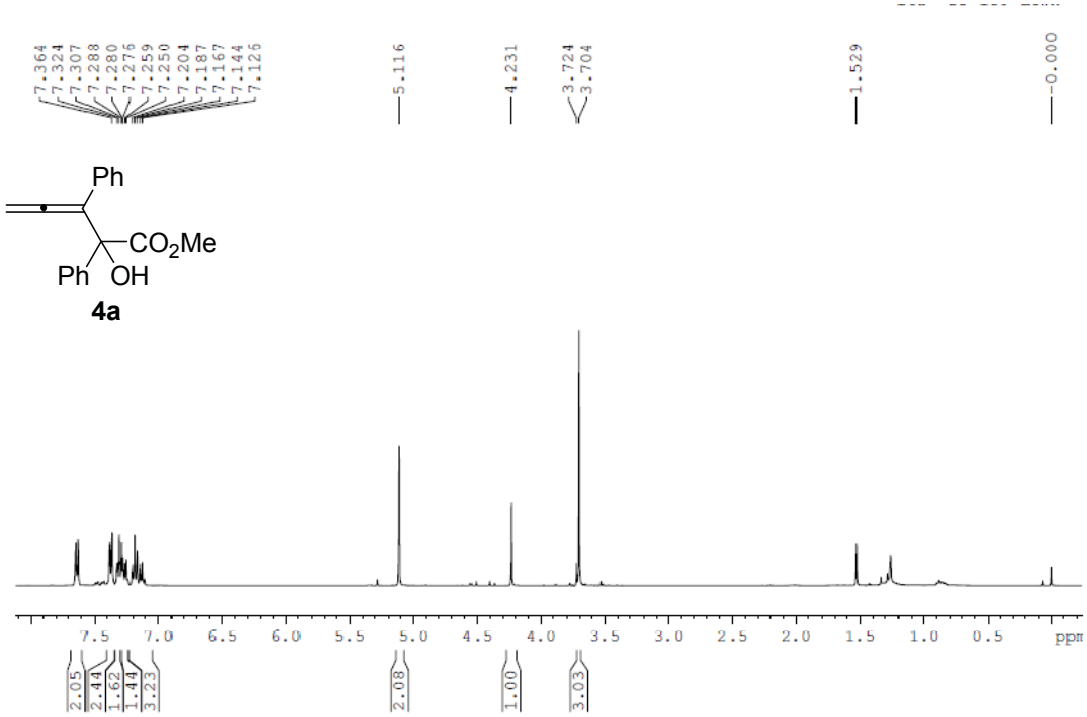

Br
CO2Me
O
3r

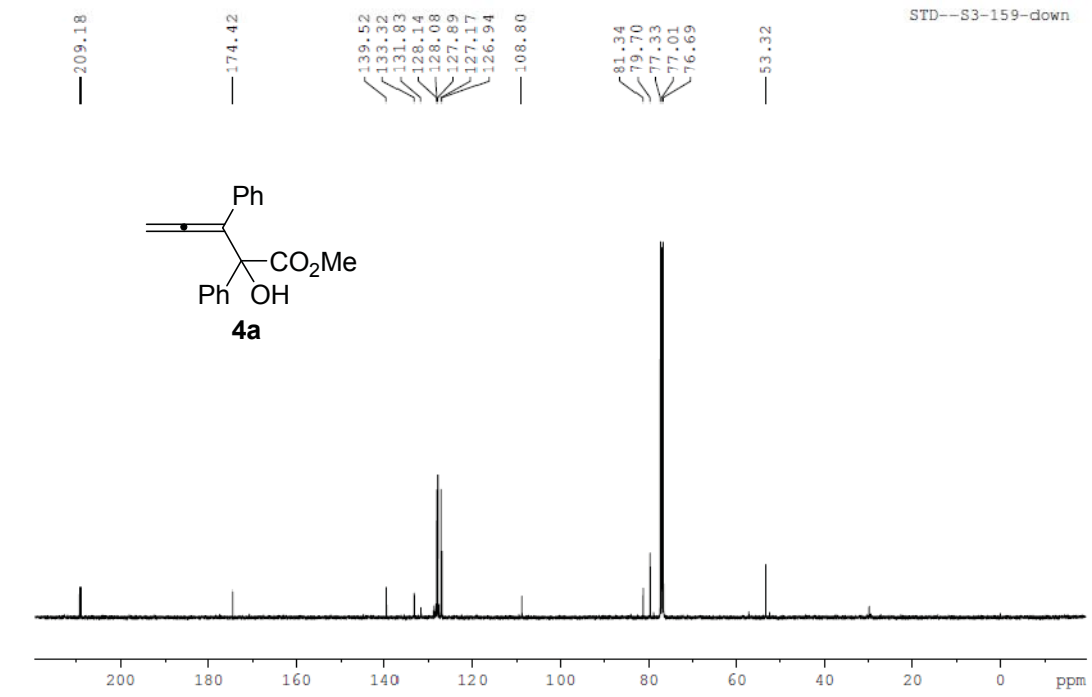

Cl
CO2Me
O
3s

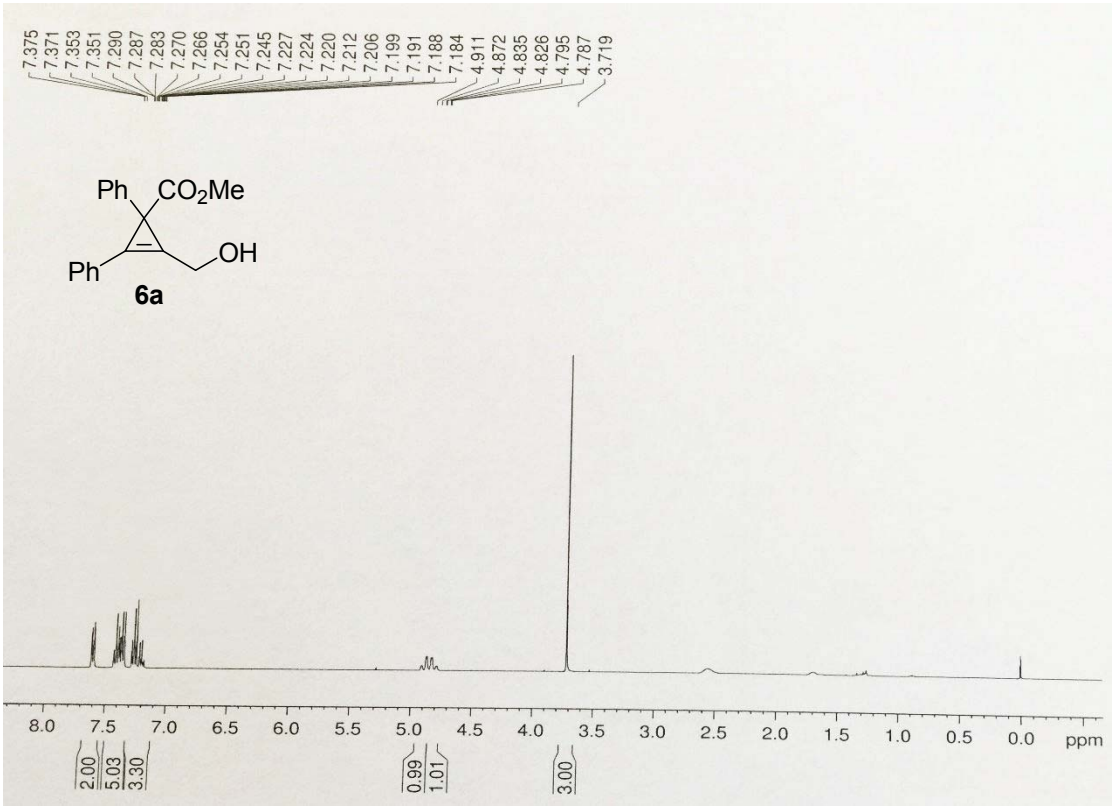

Cl
CO2Me
O
3s
f1 (ppm)


Br
CO2Me
O
3t


Br
CO2Me
O
3t


7.364
7.324
7.307
7.288
7.280
7.276
7.259
7.250
7.204
7.187
7.167
7.144
7.126
5.116
4.231
3.724
3.704
1.529
0.000
Ph
CO2Me
Ph OH
4a
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
2.05
2.44
1.62
1.44
3.23
2.08
1.00
3.03


STD--S3-159-down
209.18
174.42
139.52
133.32
131.83
128.14
128.08
127.89
127.17
126.94
108.80
81.34
79.70
77.33
77.01
76.69
53.32
Ph
CO2Me
Ph OH
4a
200 180 160 140 120 100 80 60 40 20 0 ppm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7.351
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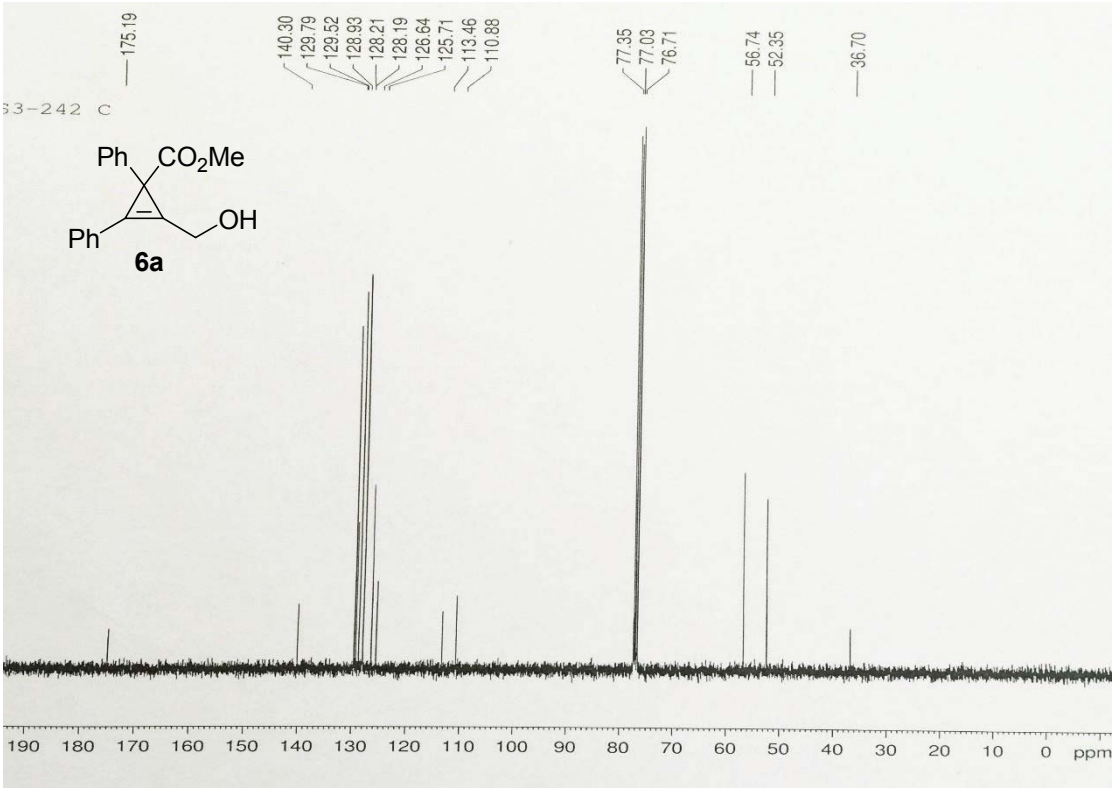
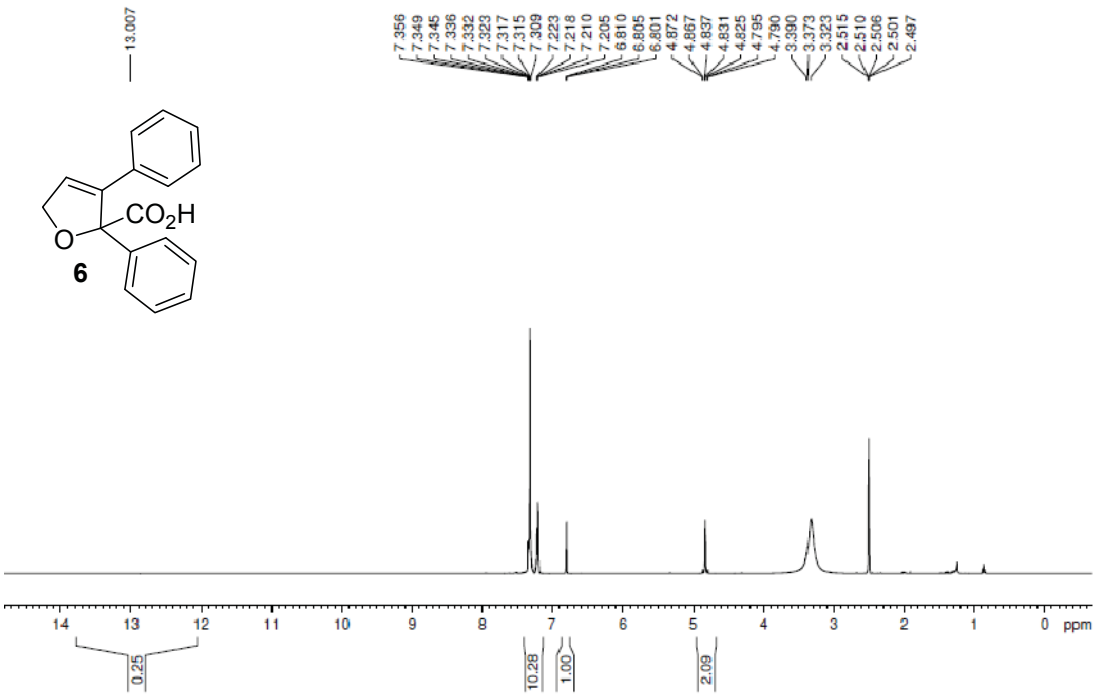
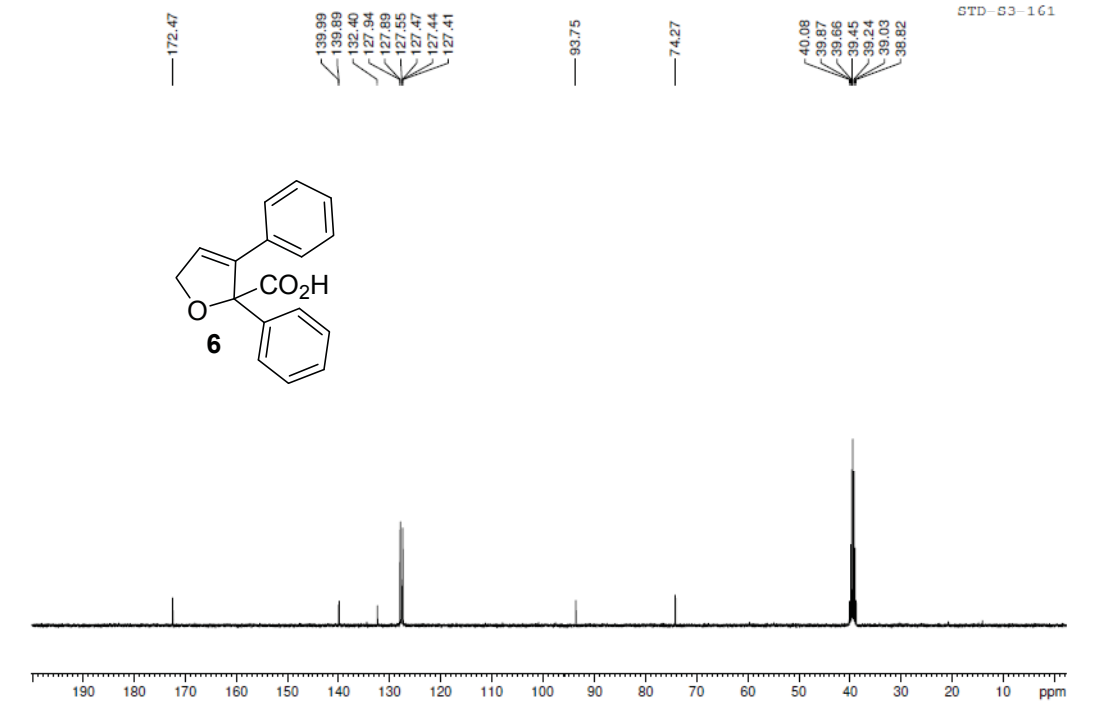
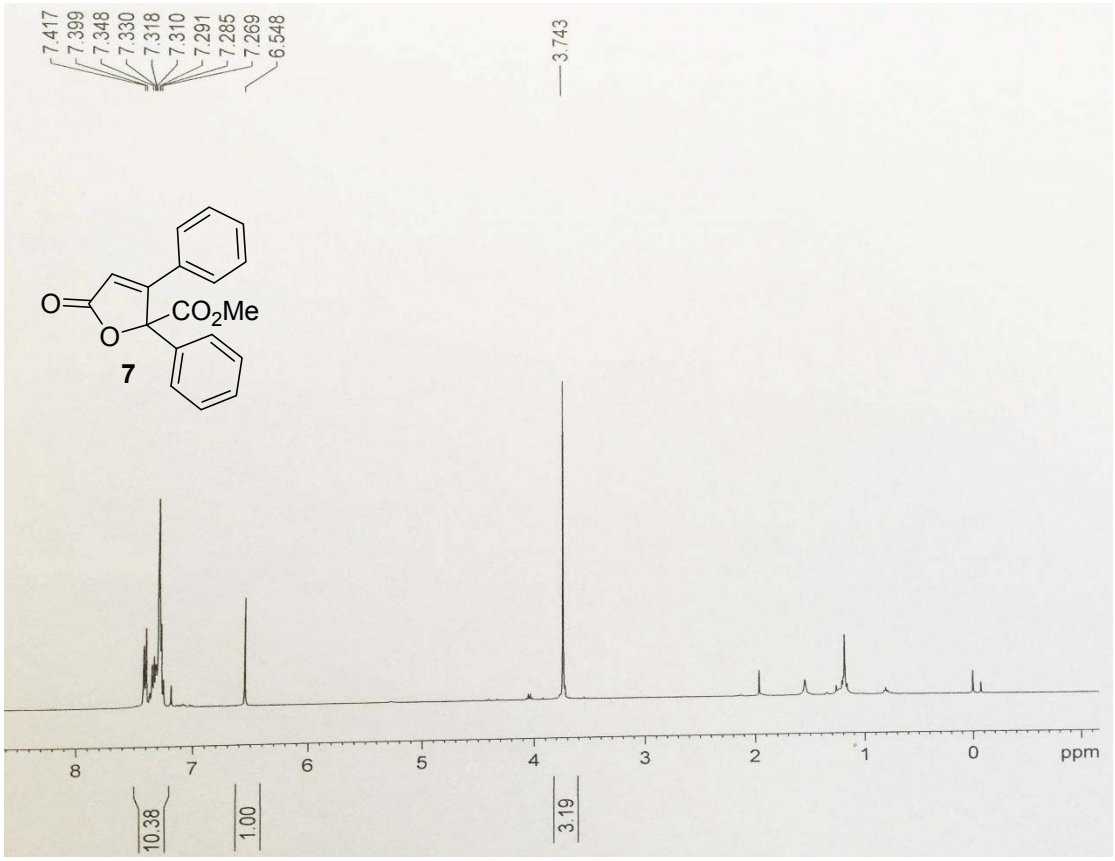
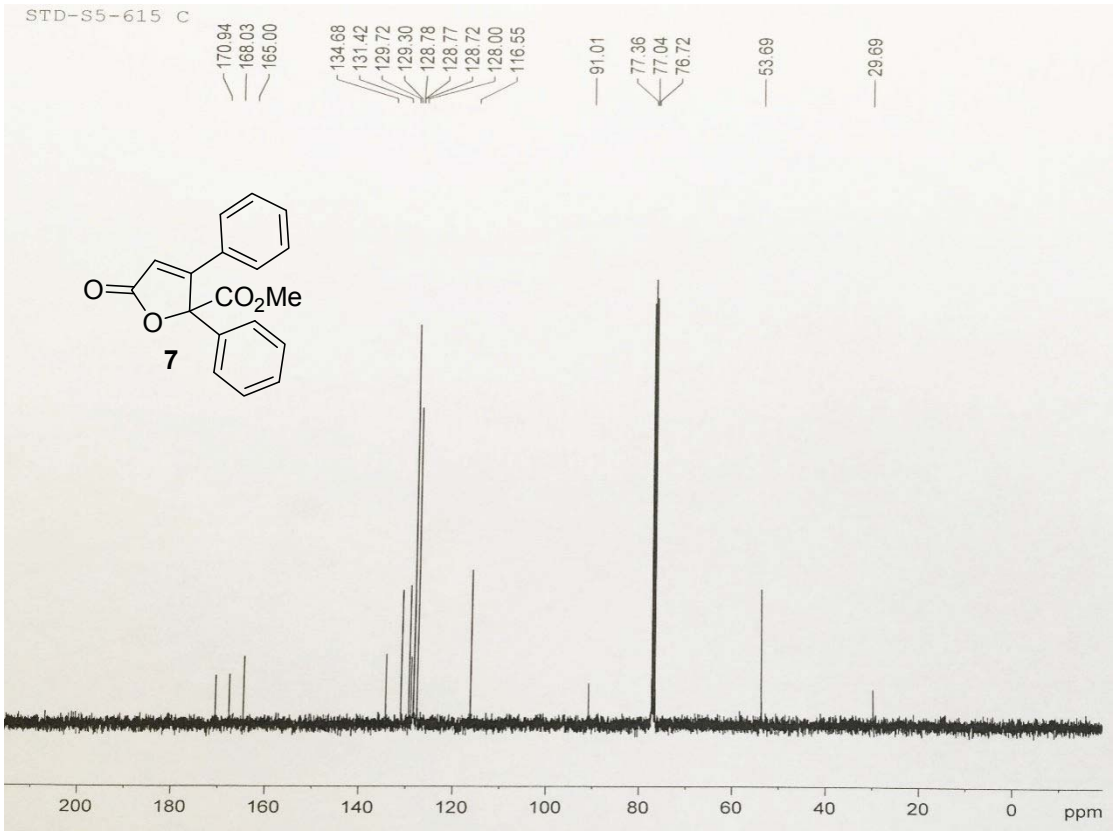
7.287
7.283
7.270
7.266
7.254
7.251
7.245
7.227
7.224
7.220
7.212
7.206
7.199
7.191
7.188
7.184
4.911
4.872
4.835
4.826
4.795
4.787
3.719
Ph
CO2Me
OH
Ph
6a
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
2.00
5.03
3.30
0.99
1.01
3.00

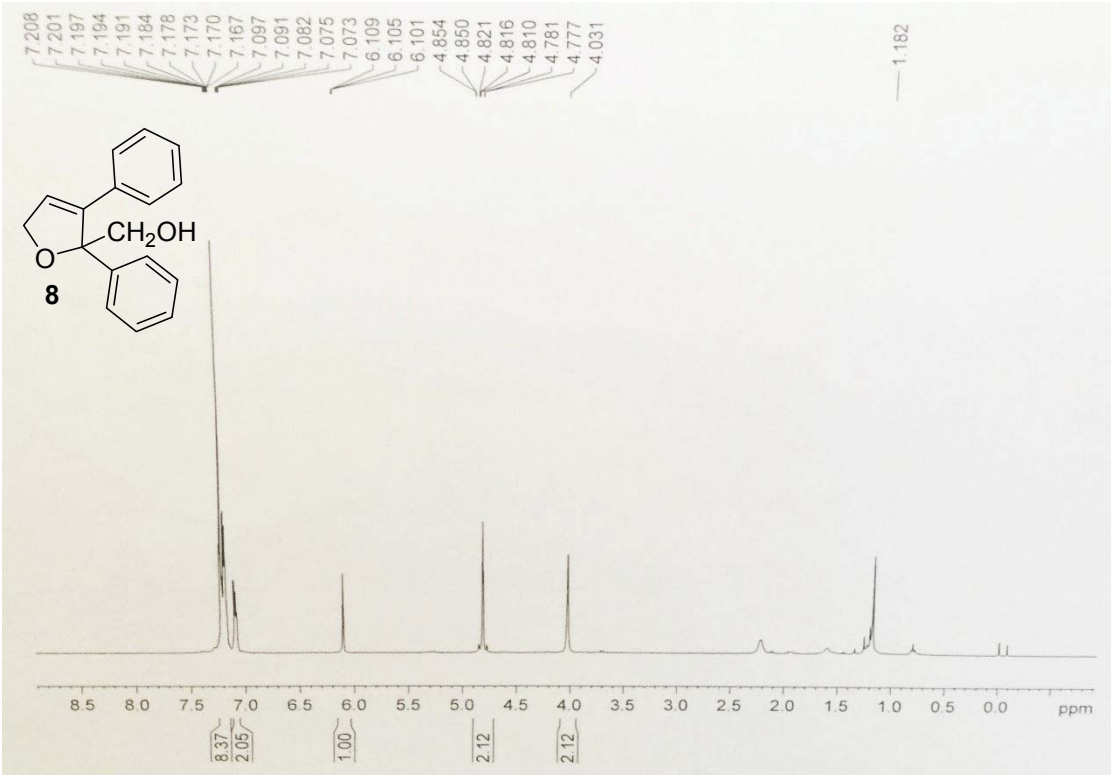

175.19
140.30
129.79
129.52
128.93
128.21
128.19
126.64
125.71
113.46
110.88
77.35
77.03
76.71
56.74
52.35
36.70
Ph
CO2Me
OH
Ph
6a
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

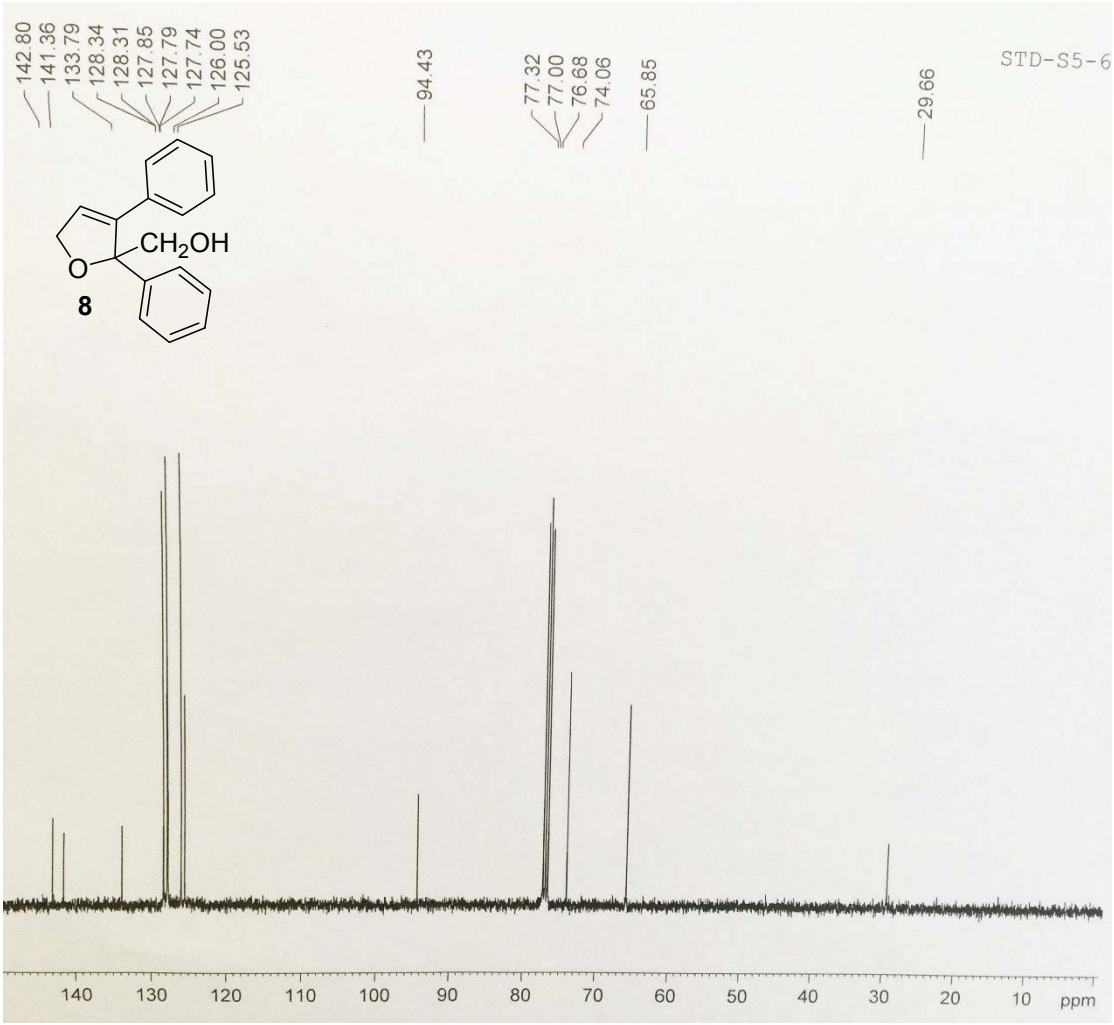

13.007
7.356
7.349
7.345
7.336
7.332
7.323
7.317
7.315
7.309
7.223
7.218
7.210
7.205
6.810
6.805
6.801
4.872
4.867
4.837
4.831
4.825
4.795
4.790
3.390
3.373
3.323
2.515
2.510
2.506
2.501
2.497
CO2H
O
6
14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 ppm
0.25
10.28
1.00
2.09


STD-S3-161
172.47
139.99
139.89
132.40
127.94
127.89
127.55
127.47
127.44
127.41
93.75
74.27
40.08
39.87
39.66
39.45
39.24
39.03
38.82
CO2H
O
6
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


7.417
7.399
7.348
7.330
7.318
7.310
7.291
7.285
7.269
6.548
3.743
O
O
CO2Me
7
8
7
6
5
4
3
2
1
0
ppm
10.38
1.00
3.19

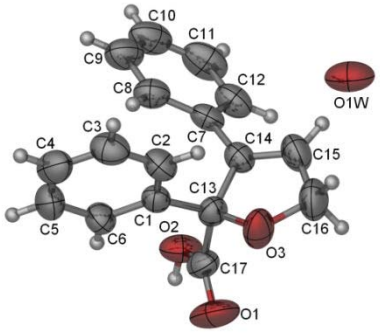

STD-S5-615 C
170.94
168.03
165.00
134.68
131.42
129.72
129.30
128.78
128.77
128.72
128.00
116.55
91.01
77.36
77.04
76.72
53.69
29.69
O
O
CO2Me
7
200
180
160
140
120
100
80
60
40
20
0
ppm


7.208
7.201
7.197
7.194
7.191
7.184
7.178
7.173
7.170
7.167
7.097
7.091
7.082
7.075
7.073
6.109
6.105
6.101
4.854
4.850
4.821
4.816
4.810
4.781
4.777
4.031
1.182
CH2OH
O
8
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
8.37
2.05
1.00
2.12
2.12


142.80
141.36
133.79
128.34
128.31
127.85
127.79
127.74
126.00
125.53
94.43
77.32
77.00
76.68
74.06
65.85
29.66
STD-S5-6
CH2OH
O
8
140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

X-ray Crystal Structure Data

CCDC 1035999contains the supplementary crystallographic data for **8a**. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.



checkCIF/PLATON report (basic structural check)

No syntax errors found. CIF dictionary
Please wait while processing .... Interpreting this report

Datablock: z

Bond precision: C-C = 0.0036 A Wavelength=0.71073

Cell: a=6.7978(7) b=7.3605(7) c=29.386(3)
alpha=90 beta=90 gamma=90

Temperature: 296 K

	Calculated	Reported
Volume	1470.3(3)	1470.3(3)
Space group	P 21 21 21	P2(1)2(1)2(
Hall group	P 2ac 2ab	?
Moiety formula	C17 H14 O3, O	?
Sum formula	C17 H14 O4	C17 H14 O4
Mr	202.20	202.20
Dx,g cm-3	1.275	1.275
Z	4	4
Mu (mm-1)	0.091	0.091
F000	592.0	592.0
F000'	592.33	
h,k,lmax	9,9,39	9,9,39
Nref	2212[3789]	3711
Tmin,Tmax	0.972,0.980	0.966,0.980
Tmin'	0.966	

Correction method= MULTI-SCAN

Data completeness= 1.68/0.98 Theta(max)= 28.710

R(reflections)= 0.0541(2749) wR2(reflections)= 0.1607(3711)

S = 1.044 Npar= 190