Cu-catalyzed mild C(sp^2)-H functionalization assisted by carboxylic acids en route to hydroxylated arenes

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Supporting Information

General considerations. Unless otherwise stated, all reactions were carried out under an argon atmosphere in resealable screw-cap test tubes using standard Schlenk techniques for the manipulation of solvents and reagents. $Cu(OAc)_2$ was purchased from Aldrich, $[PhCO_2]_2$ (wet, 25% H_2O) from Alfa Aesar and HFIP from Fluorochem. All chemicals were used as received. All other reagents were purchased from commercial sources and used also as received. All benzoic acids were prepared via oxidation of the benzaldehydes,[1] hydrolysis of the corresponding ester derivative[2] and Suzuki-Miyaura coupling reactions[3] and purified by regular column chromatography.

Analytical methods. ^1H NMR and ^{13}C NMR spectra and melting points (where applicable) are included for all compounds. ^1H and ^{13}C NMR spectra were recorded on a Bruker 400 MHz and a Bruker 500 MHz at 20 ºC. All ^1H NMR spectra are reported in parts per million (ppm) downfield of TMS and were measured relative to the signals for $CHCl_3$ (7.27 ppm). All ^{13}C NMR spectra were reported in ppm relative to residual $CHCl_3$ (77 ppm) and were obtained with ^1H decoupling. Coupling constants, J, are reported in hertz. Melting points were measured using open glass capillaries in a Buchi B540 apparatus. Infrared spectra were recorded on a Bruker Tensor 27. Mass spectra were recorded on a Waters LCT Premier spectrometer. Gas chromatographic analyses were performed on Hewlett-Packard 6890 gas chromatography instrument with a FID detector using 25m x 0.20 mm capillary column with cross-linked methyl siloxane as the stationary phase. High Pressure Liquid Chromatographic (HPLC) analyses were performed on Agilent Technologies Model 1260 Infinity HPLC chromatography instrument equipped with Agilent Eclipse Plus C18 (3.5 um, 4.6 x 100 mm) column and UV/Vis detector. Flash chromatography was performed with EM Science silica gel 60 (230-400 mesh). The yields reported in Tables 2, Table 3 and Table 4 refer to isolated yields and represent an average of at least two independent runs. The procedures described in this section are representative. Thus, the

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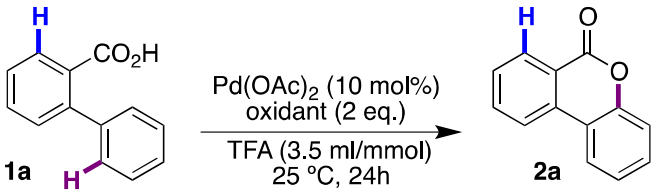
[1] Fleckenstein, C. A.; Plenio, H. *Chem. Eur. J.* **2007**, *13*, 2701
[2] Wang, C.; Rakshit, S.; Glorius, F. *J. Am. Chem. Soc.* **2010**, *132*, 14006
[3] Barder, T. E.; Walker, S. D.; Martinelli, J. R.; Buchwald, S. L. *J. Am. Chem. Soc.* **2005**, *127*, 4685.

yields may differ slightly from those given in Tables 2, 3, and 4.

Optimization Details

Using Pd as catalyst:

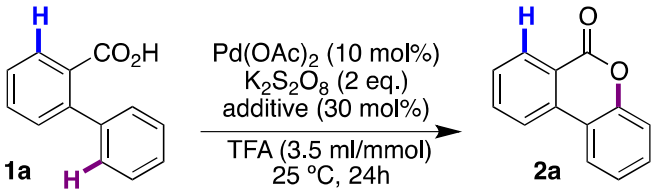
(1) Oxidant effect[a]



Entry	*Oxidant*	*GC yield*
1	Oxone	47
2	MnO_2	0
3	O_2	0
4	Selectfluor	43
5	$K_2S_2O_8$	60
6	NFSI	16

[a] **1a** (0.25 mmol), $Pd(OAc)_2$ (10 mol%), oxidant (2 equiv.), TFA (3.5 mL/mmol) at 25 ºC. [b] GC yield using dodecane as an internal standard.

(2) Additive effect[a]

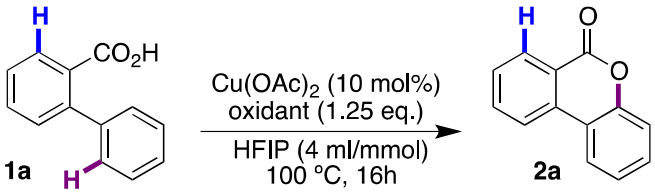


Entry	*Additive*	*Gc yield*
1	PivOH	58
2	$CH_3CH_3CO_2H$	43
3	$MesCO_2H$	62
4	$AdCO_2H$	58
5	*N*-Ac-Alanine	50
6	N-Ac-Isoleucine	62
7	N-Ac-Norleucine	52
8	**2-$FC_6H_4CO_2H$**	**70**
9	2-$NO_2C_6H_4CO_2H$	45
10	Phenenthroline	44
11	Neocuproine	10
12	Benzoquinone	38

[a] **1a** (0.25 mmol), $Pd(OAc)_2$ (10 mol%), $K_2S_2O_8$ (0.50 mmol), additive (30 mol%), TFA (3.5 mL/mmol) at 25 ºC. [b] GC yield using dodecane as an internal standard.

Using Cu as catalyst

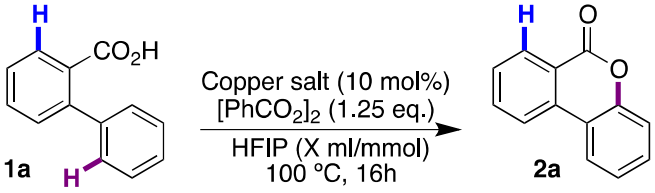
(1) Oxidants screened[a]



Entry	*Oxidant*	*Gc yield*
1	^{t}BuOOH (70% wt. H_2O)	15
2	H_2O_2 (35% wt. H_2O)	15
3	Cumene Hydroperoxide 80%	5
4	Urea·H_2O_2	7
5	$K_2S_2O_8$	3
7	O_2	0
8	*m*-CPBA	51
9	^{t}BuOOBz	3
10	**$[PhCO_2]_2$**	**80**

[a] **1a** (0.25 mmol), $Cu(OAc)_2$ (10 mol%), oxidant (1.25 equiv.), HFIP (4mL/mmol) at 100 ºC. [b] GC yield using decane as an internal standard.

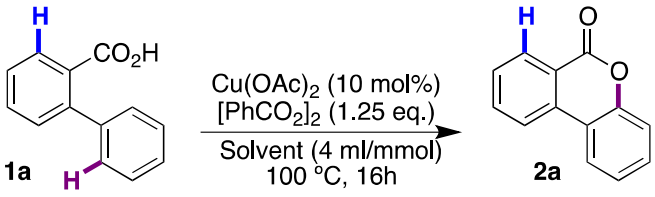
(2) Concentration effect[a]



Entry	*HFIP (X mL/mmol)*	*Gc yield*
1	2	73
2	3	73
3	4	80
4	6	88
5	**8**	**93**

[a] **1a** (0.25 mmol), $Cu(OAc)_2$ (10 mol%), $[PhCO_2]_2$ (1.25 equiv.), HFIP (X mL/mmol) at 100 ºC. [b] GC yield using decane as an internal standard.

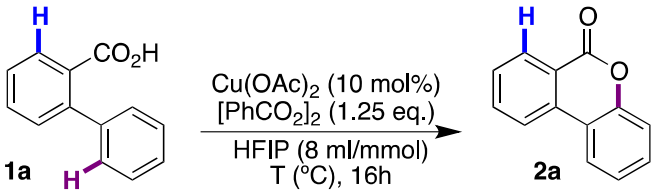
(3) Solvent effect[a]



Entry	*Solvent*	*Gc conv.*	*Gc yield*
1	DCE	49	15
2	1,4-Dioxane	61	21
3	Anisole	100	51
4	PhCl	55	47
5	$PhCF_3$	45	20
6	CF_3CH_2OH	49	30
7	**HFIP**	**100**	**80**
8	C_6F_6	100	67
9	PhF	100	18

[a] **1a** (0.25 mmol), $Cu(OAc)_2$ (10 mol%), $[PhCO_2]_2$ (1.25 equiv.), solvent (4mL/mmol) at 100 ºC. [b] GC yield using decane as an internal standard.

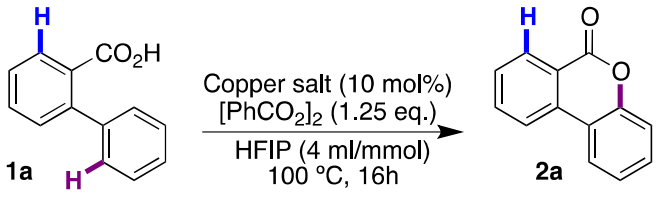
(4) Temperature effect[a]



Entry	*T(ºC)*	*Gc conv.*	*Gc yield*
1	25	100	73
2	50	100	73
3	**75**	**100**	**95**
4	110	100	88
5	125	100	93

[a] **1a** (0.25 mmol), $Cu(OAc)_2$ (10 mol%), $[PhCO_2]_2$ (1.25 equiv.), HFIP (8mL/mmol). [b] GC yield using decane as an internal standard.

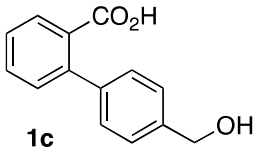
(5) Copper salt effect[a]



Entry	*Copper Salt*	*Gc conv.*	*Gc yield*
1	$Cu(OAc)_2$ (5 mol%)	100	97
2	$Cu(OTf)_2$	100	21
3	$CuBr_2$	100	79
4	CuOAc	100	88

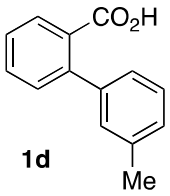
[a] **1a** (0.25 mmol), $Cu(OAc)_2$ (10 mol%), $[PhCO_2]_2$ (1.25 equiv.), HFIP (4mL/mmol) at 75 ºC. [b] GC yield using decane as an internal standard.

Starting Materials



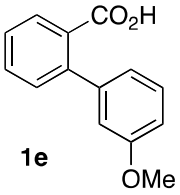
1c

4'-(hydroxymethyl)-[1,1'-biphenyl]-2-carboxylic acid (1c) White Solid. M.p. = 144-146 ºC. ^{1}H NMR (400 MHz, CD_3OD) δ 7.81 (dd, *J* = 7.7, 1.5 Hz, 1H), 7.58 (td, *J* = 7.7, 1.4 Hz, 1H), 7.45 (td, *J* = 7.6, 1.3 Hz, 1H), 7.44-7.38 (m, 3H), 7.38-7.29 (m, 2H), 4.67 (s, 2H) ppm. ^{13}C NMR (101 MHz, CD_3OD) δ 173.3, 144.1, 142.6, 142.6, 134.1, 133.0, 132.7, 131.4, 130.4, 129.0, 128.6, 65.9 ppm. IR (neat, cm^{-1}): 3355, 3009, 1687, 1682, 1670, 1406, 1135, 759. HRMS *calcd* for ($C_{14}H_{11}O_3$-H^+): 227.0708, found 227.0714.



1d

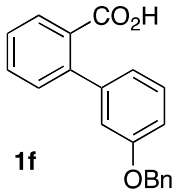
3'-methyl-[1,1'-biphenyl]-2-carboxylic acid (1d) White solid. M.p. = 97-98 ºC. ^{1}H NMR (400 MHz, CD_3OD) δ 7.79 (d, *J* = 7.8 Hz, 1H), 7.55 (m, 1H), 7.43 (m, 1H), 7.37 (dd, *J* = 7.7, 2.6 Hz, 1H), 7.27 (m, 1H), 7.18 (m, 1H), 7.15 (m, 1H), 7.13 (m, 1H), 2.39 (s, 3H) ppm. ^{13}C NMR (101 MHz, CD_3OD) δ 173.4, 144.4, 143.5, 139.6, 134.0, 132.9, 132.8, 132.6, 131.0, 129.8, 129.7, 128.9, 127.5, 22.4 ppm. The spectroscopic data correspond to those previously reported in the literature.[2]



1e

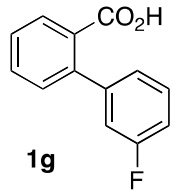
3'-methoxy-[1,1'-biphenyl]-2-carboxylic acid (1e) White solid. M.p. = 91-93 ºC. ^{1}H NMR (400 MHz, CD_3OD) δ 7.80 (ddd, *J* = 7.7, 1.5, 0.6 Hz, 1H), 7.56

(td, *J* = 7.6, 1.4 Hz, 1H), 7.44 (m, 2H), 7.40 (m, 1H), 6.93 (m, 3H), 3.82 (s, 3H) ppm. ^{13}C NMR (101 MHz, Methanol-d4) δ 173.3, 161.7, 144.8, 144.1, 134.2, 132.9, 132.5, 131.2, 131.0, 129.1, 122.8, 116.1, 114.7, 56.5 ppm. The spectroscopic data correspond to those previously reported in the literature.[4]



1f

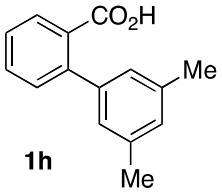
3'-(benzyloxy)-[1,1'-biphenyl]-2-carboxylic acid (1f) White Solid. M.p. = 105-106 ºC. ^{1}H NMR (500 MHz, CD_3OD) δ 7.79 (dd, *J* = 7.7, 1.4 Hz, 1H), 7.54 (td, *J* = 7.7, 1.4 Hz, 1H), 7.48-7.40 (m, 3H), 7.41-7.34 (m, 3H), 7.35-7.25 (m, 2H), 7.05-6.96 (m, 2H), 6.96-6.92 (m, 1H), 5.08 (s, 2H) ppm. ^{13}C NMR (126 MHz, CD_3OD) δ 173.3, 160.9, 144.9, 144.0, 139.5, 134.1, 132.9, 132.5, 131.2, 131.0, 130.3, 129.7, 129.5, 129.1, 123.2, 117.1, 115.6, 71.9 ppm. IR (neat, cm^{-1}): 2922, 2858, 2224, 2063, 1689, 1675, 1580, 1566, 1344, 1276. HRMS *calcd* for ($C_{20}H_{15}O_3$-H^+): 303,1021 found 303.1027.



1g

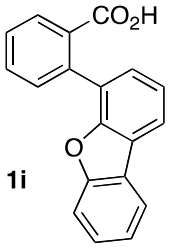
3'-fluoro-[1,1'-biphenyl]-2-carboxylic acid (1g) White Solid. M.p. = 128-130 ºC. ^{1}H NMR (500 MHz, CD_3OD) δ 7.86 (dd, *J* = 7.7, 1.4 Hz, 1H), 7.59 (d, *J* = 1.4 Hz, 1H), 7.48 (d, *J* = 1.3 Hz, 1H), 7.45-7.35 (m, 2H), 7.22-7.14 (m, 1H), 7.09 (dd, *J* = 9.4, 1.2 Hz, 2H) ppm. ^{13}C NMR (126 MHz, CD_3OD) δ 172.6, 165.7-163.8 (d, *J* = 246 Hz), 146.1, 146.0, 143.2, 143.2, 133.8, 133.2, 132.5, 131.6, 131.6, 129.6, 126.4, 126.4, 117.3, 117.2, 115.8, 115.6 ppm. The spectroscopic data correspond to those previously reported in the literature.[4]

[4] Hoovera, J. R. E.; Chow, A.W.; Stedman, R., J.; Hall, N. M.; Greenberg, H. M.; Dolan, M. M.; Ferlauto, R. J. *J. Med. Chem.* **1964**, *7*, 245.



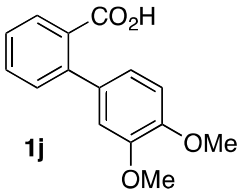
1h

3',5'-dimethyl-[1,1'-biphenyl]-2-carboxylic acid (1h) White solid. M.p. = 109-111 ºC. ^{1}H-NMR (400 MHz, CD_3OD) δ 7.77 (dd, *J* = 7.7 Hz, 1H), 7.54 (d, *J* = 1.5 Hz, 1H), 7.42 (d, *J* = 7.6 Hz, 1H), 7.36 (dd, *J* = 7.7, 1.3 Hz, 1H), 7.00 (s, 1H), 6.97 (s, 2H), 2.35 (s, 6H) ppm. ^{13}C NMR (101 MHz, CD_3OD) δ 173.5, 144.5, 143.4, 139.5, 134.1, 132.8, 132.5, 131.1, 130.6, 128.8, 128.2, 22.2 ppm. IR (neat, cm^{-1}): 3009, 2919, 2856, 2651, 1682, 1406, 1291, 1267. HRMS *calcd* for ($C_{15}H_{13}O_2$-H^+): 225,0916, found 225.0921.



1i

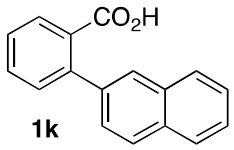
2-(dibenzo[b,d]furan-4-yl)benzoic acid (1i) Light yellow solid. M.p. = 179-180 ºC. ^{1}H NMR (500 MHz, CD_3OD) δ 8.04 (m, 2H), 8.02 (dd, *J* = 7.5, 1.4 Hz, 1H), 7.69 (td, J = 7.5, 1.4 Hz, 1H), 7.54 (m, 3H), 7.46 (m, 3H), 7.37 (td, *J* = 7.5, 1.2 Hz, 1H) ppm. ^{13}C NMR (126 MHz, CD_3OD) δ 172.4, 158.2, 155.8, 139.2, 134.2, 133.7, 133.4, 132, 129.8, 129.2, 128.3, 126.4, 125.9, 124.9, 124.8, 122.6, 121.7, 113.4 ppm. IR (neat, cm^{-1}): 3063, 3038, 2922, 2854, 1737, 1606, 1308, 1282. HRMS *calcd* for ($C_{19}H_{11}O_3$-H^+): 287.0708, found 287.0711.



1j

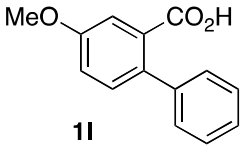
3',4'-dimethoxy-[1,1'-biphenyl]-2-carboxylic acid (1j) White solid. M.p. = 165-166 ºC. ^{1}H NMR δ (500 MHz, DMSO-d_6) δ 7.70 (dd, *J* = 7.0, 2.0 Hz, 1H), 7.57 (td, *J* = 7.0, 2.0 Hz, 1H), 7.47 (m, 2H), 7.03 (d, *J* = 2.0 Hz, 1H), 6.97 (d, *J*

= 2.5 Hz, 1H), 6.91 (dd, *J* = 10,0, 2.5 Hz, 1H), 4.62 (s, 3H), 4.61 (s, 3H) ppm. ^{13}C NMR (126 MHz, DMSO-d_6) δ 171.1, 149.2, 149.2, 141.3, 134.2, 133.6, 131.5, 131.2, 129.6, 127.7, 121.5, 113.2, 112.5, 56.5, 56.4 ppm. The spectroscopic data correspond to those previously reported in the literature.[5]



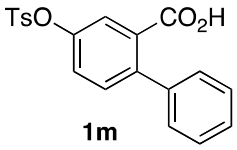
1k

2-(naphthalen-2-yl)benzoic acid (1k) White Solid. M.p. = 190-192 ºC. ^{1}H NMR (500 MHz, CD_3OD) δ 7.95-7.86 (m, 4H), 7.85-7.80 (m, 1H), 7.68-7.56 (m, 1H), 7.55-7.45 (m, 5H) ppm. ^{13}C NMR (101 MHz, CD_3OD) δ 173.1, 144.5, 141.2, 135.6, 134.9, 134.1, 133.2, 133.0, 131.6, 129.9, 129.5, 129.3, 129.2, 129.0, 128.9, 128.1, 127.9 ppm. The spectroscopic data correspond to those previously reported in the literature.[2]



1l

4-methoxy-[1,1'-biphenyl]-2-carboxylic acid (1l) White Solid. M.p. = 117-119 ºC. ^{1}H NMR (400 MHz, CD_3OD) δ 7.46-7.25 (m, 7H), 7.13 (dd, *J* = 8.5, 2.8 Hz, 1H), 3.88 (s, 3H) ppm. ^{13}C NMR (101 MHz, CD_3OD) δ 173.0, 160.9, 143.3, 136.7, 134.9, 133.9, 130.5, 129.8, 128.7, 118.6, 116.5, 56.8 ppm. The spectroscopic data correspond to those previously reported in the literature.[6]

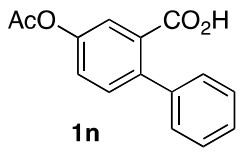


1m

4-(tosyloxy)-[1,1'-biphenyl]-2-carboxylic acid (1m) White Solid. M.p. = 140-142 ºC. ^{1}H NMR (300 MHz, CD_3OD) δ 7.79 (d, *J* = 8.2 Hz, 2H), 7.48 (d, *J* = 8.1 Hz, 2H), 7.43-7.27 (m, 7H), 7.20 (dd, *J* = 8.4, 2.6 Hz, 1H), 2.49 (s, 3H) ppm ^{13}C NMR (126 MHz, CD_3OD) δ 171.2, 150.6, 148.3, 143.4, 142.2, 135.3,

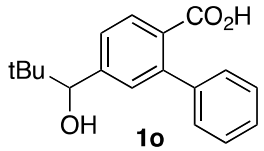
[5] Ciske, F.; Winton, J. *Synthesis*, **1995**, *8*, 1195.
[6] Wang, D.-H.; Mei, T.-S.; Yu, J.-Q. *J. Am. Chem. Soc.* **2008**, *130*, 17676.

134.2, 134.2, 132.0, 130.5, 130.4, 123.0, 129.4, 126.8, 125.2, 22.5 ppm. IR (neat, cm^{-1}): 3036, 1703, 1681, 1597, 1371, 785. HRMS *calcd* for ($C_{20}H_{15}O_5S$-H^+): 367,0640 found 367.0643.



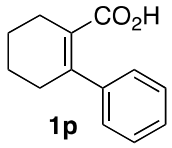
1n

4-acetoxy-[1,1'-biphenyl]-2-carboxylic acid (1n) White Solid. M.p. = 155-157 ºC. ^{1}H NMR (400 MHz, CD_3OD) δ 7.57 (dd, *J* = 2.5, 0.4 Hz, 1H), 7.45-7.34 (m, 7H), 2.34 (s, 3H) ppm. ^{13}C NMR (126 MHz, CD_3OD) δ 171.9, 171.8, 151.9, 142.7, 142.0, 134.9, 133.8, 130.5, 130.4, 129.9, 129.2, 126.3, 124.7, 21.7 ppm. IR (neat, cm^{-1}): 3068, 2924, 1757, 1689, 1482, 1194. HRMS *calcd* for ($C_{20}H_{15}O_5S$-H^+): 255.0657 found 255.060.



1o

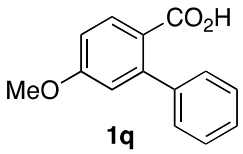
5-(1-hydroxy-2,2-dimethylpropyl)-[1,1'-biphenyl]-2-carboxylic acid (1o) White Solid. M.p. = 163-165 ºC. ^{1}H NMR (500 MHz, CD_3OD) δ 7.79 (d, *J* = 8.0 Hz, 1H), 7.44-7.37 (m, 3H), 7.38-7.34 (m, 4H), 4.45 (s, 1H), 0.97 (s, 9H) ppm. ^{13}C NMR (101 MHz, CD_3OD) δ 173.1, 148.4, 143.8, 143.6, 132.3, 132.2, 130.7, 130.4, 129.9, 128.9, 128.5, 83.3, 37.4, 27.4 ppm. IR (neat, cm^{-1}): 3410, 2954, 2908, 2867, 1688, 1297, 1266, 1232, 1053. HRMS *calcd* for ($C_{18}H_{19}O_3$-H^+): 283.1334 found 283.1337.



1p

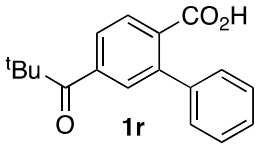
3,4,5,6-tetrahydro-[1,1'-biphenyl]-2-carboxylic acid (1p) White Solid. M.p. = 129-131 ºC. ^{1}H NMR (400 MHz, CD_3OD) δ 7.36-7.28 (m, 2H), 7.28-7.22 (m, 1H), 7.23-7.14 (m, 2H), 2.74-2.29 (m, 4H), 1.87-1.59 (m, 4H) ppm. ^{13}C NMR (126 MHz, CD_3OD) δ 174.5, 146.8, 145.5, 130.1, 129.8, 128.9, 128.8, 34.5,

28.8, 24.5, 24.0 ppm. The spectroscopic data correspond to those previously reported in the literature.[7]



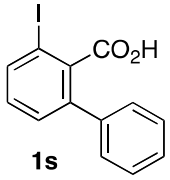
1q

5-methoxy-[1,1'-biphenyl]-2-carboxylic acid (1q) White Solid. M.p. = 171-173 ºC. ^{1}H NMR (500 MHz, CD_3OD) δ 7.89 (d, *J* = 8.7 Hz, 1H), 7.48-7.23 (m, 5H), 6.99 (dd, *J* = 8.7, 2.6 Hz, 1H), 6.85 (d, *J* = 2.6 Hz, 1H), 3.87 (s, 3H) ppm. ^{13}C NMR (126 MHz, CD_3OD) δ 172.3, 164.1, 147.5, 143.9, 134.3, 130.3, 129.7, 129.0, 125.3, 118.4, 114.1, 56.8 ppm. The spectroscopic data correspond to those previously reported in the literature.[4]



1r

5-pivaloyl-[1,1'-biphenyl]-2-carboxylic acid (1r) White Solid. M.p. = 160-161 ºC. ^{1}H NMR (400 MHz, CD_3OD) δ 7.87 (dd, *J* = 8.0, 0.5 Hz, 1H), 7.72 (dd, *J* = 8.0, 1.8 Hz, 1H), 7.59 (dd, *J* = 1.8, 0.5 Hz, 1H), 7.47-7.33 (m, 5H), 1.36 (s, 9H) ppm. ^{13}C NMR (126 MHz, CD_3OD) δ 211.7, 172.3, 144.1, 143.3, 142.7, 136.0, 131.5, 131.2, 130.3, 130.1, 129.5, 127.9, 46.2, 29.0 ppm. IR (neat, cm^{-1}): 2958, 2923, 2853, 2659, 1687, 1672, 1295, 1279. HRMS *calcd* for ($C_{18}H_{17}O_3$-H^+): 281.1178 found 281.1183.

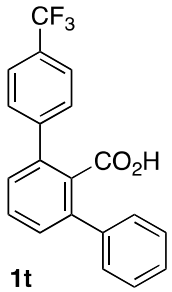


1s

3-iodo-[1,1'-biphenyl]-2-carboxylic acid (1s) White Solid. M.p. = 172-173 ºC. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.85 (dd, *J* = 8.0, 1.1 Hz, 1H), 7.38 (m, 5H), 7.35 (dd, *J* = 7.7, 1.1 Hz, 1H), 7.17 (t, *J* = 7.8 Hz, 1H) ppm. ^{13}C NMR (126 MHz, CD_3OD) δ 173.3, 142.9, 142.7, 141.9, 140.0, 132.3, 131.5, 130.4,

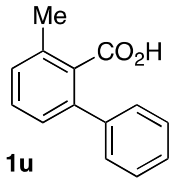
[7] Parham, W.E.; Moulton, W.E.; Zuckerbraun, A. *J. Org. Chem.* **1956**, *21*, 72.

130.3, 130.2, 129.8, 93.4 ppm. The spectroscopic data correspond to those previously reported in the literature.[8]



1t

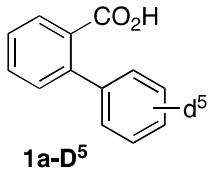
4-(trifluoromethyl)-[1,1':3',1''-terphenyl]-2'-carboxylic acid (1t) White solid. M.p. = 183-186 ºC. ^{1}H NMR (500 MHz, CD_3OD) δ 7.74 (d, *J* = 8.2 Hz, 1H), 7.66 (d, *J* = 8.0 Hz, 1H), 7.60 (t, *J* = 7.7 Hz, 7H), 7.52-7.36 (m, 3H). ^{13}C NMR (126 MHz, CD_3OD) δ 173.7, 146.9, 142.6, 142.2, 140.5, 136.0, 131.7 (d, J = 32.7 Hz), 131.6, 131.4 (d, J = 32.7 Hz), 131.3, 131.3, 130.6, 130.5, 130.2, 129.6, 127.7 (d, *J* = 272 Hz), 127.0 (d, *J* = 3.8 Hz), 127.0 (d, *J* = 3.8 Hz), 125.5 (d, *J* = 272 Hz). IR (neat, cm^{-1}): 2920, 2851, 2643, 1698, 1322, 1275, 1164, 1119, 1066, 775. HRMS *calcd* for ($C_{18}H_{17}O_3$-H^+): 341.0795 found 341.0797.



1u

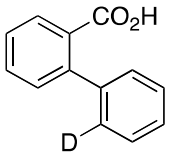
3-methyl-[1,1'-biphenyl]-2-carboxylic acid (1u) White Solid. M.p. = 138-139 ºC. ^{1}H NMR (500 MHz, CD_3OD) δ 7.47-7.42 (m, 2H), 7.42-7.33 (m, 4H), 7.26 (d, *J* = 7.7 Hz, 1H), 7.21 (d, *J* = 7.7 Hz, 1H), 2.44 (s, 3H). ^{13}C NMR (126 MHz, CD_3OD) δ 174.5, 143.0, 141.5, 136.5, 136.5, 131, 130.9, 130.4, 130.4, 130.1, 129.3, 129.2, 22.6 ppm. The spectroscopic data correspond to those previously reported in the literature.[8]

[8] Tilly, D.; Samanta, S. S.; Castanet, A.-S.; De, A.; Mortier, J. *Eur. J. Org. Chem.* **2006**, *2006*, 174.



1a-D^5

2-(D_5-phenyl)benzoic acid (1a-D^5) White Solid. M.p. = 108-109 ºC. ^{1}H NMR (400 MHz, CD_3OD) δ 7.82 (ddd, *J* = 7.7, 1.5, 0.5 Hz, 1H), 7.56 (td, *J* = 7.6, 1.4 Hz, 1H), 7.44 (td, *J* = 7.6, 1.3 Hz, 1H), 7.38 (ddd, *J* = 7.6, 1.3, 0.5 Hz, 1H) ppm. ^{13}C NMR (126 MHz, CD_3OD) δ 173.2, 144.3, 143.3, 134.0, 133.0, 132.6, 131.4, 130.2-129.2, 129.0, 128.8-128.3 ppm. The spectroscopic data correspond to those previously reported in the literature.[2]



1a-D^1

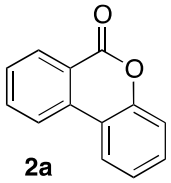
2-(D-phenyl)benzoic acid (1a-D^1) White Solid. M.p. = 110-111 ºC. ^{1}H NMR (500 MHz, CD_3OD) δ 7.82 (dd, *J* = 7.6, 1.5 Hz, 1H), 7.56 (td, *J* = 7.6, 1.5 Hz, 1H), 7.44 (td, *J* = 7.6, 1.3 Hz, 1H), 7.41-7.31 (m, 5H) ppm. ^{13}C NMR (126 MHz, CD_3OD) δ 173.2, 144.3, 143.4, 133.9, 133.0, 132.6, 131.4, 130.4, 130.2 (C-D),129.9, 129.8, 129.0, 129.0 ppm. The deuterium incorporation was found to be 87%. The spectroscopic data correspond to those previously reported in the literature.[9]

[9] Denney, D. B.; Klemchuk, P. *J. Am. Chem. Soc.*, **1958**, *80*, 3285.

Synthesis of benzo[c]chromenones (tables 2 and 3)

General procedure A for the synthesis of benzo[c]chromenones (Table 2 and 3). An oven-dried screw-cap test tube containing a stirring bar was charged with the benzoic acid derivative (0.50 mmol), $Cu(OAc)_2$ (4.5 mg, 5 mol%), benzoyl peroxide (202 mg, 0.625 mmol) and evacuated three times. Then, HFIP (4 mL) was added by syringe under a positive argon atmosphere. The mixture was stirred in a pre-heated oil bath (75 ºC) for 12 h. The mixture was then allowed to warm to room temperature and concentrated under vacuum. The resulting solid was washed with a saturated solution of $NaHCO_3$ and extracted with AcOEt (3 x 15 mL). The combined organic layers were dried over $MgSO_4$ and evaporated to yield the final benzolactone, which was purified by column chromatography on silica gel (eluting with hexanes/ethyl acetate mixtures).

General procedure B for the synthesis 2'-hydroxy-[1,1'-biphenyl]-2-carboxylic acid: following a modified procedure in the literature,[10] the crude lactone (0.25 mmol) and LiOH monohydrate (252 mg, 6 mmol, 24 equiv.) was charged to a 25 mL flask. To this mixture was then added MeOH (4 mL), THF (2 mL), and H_2O (1 mL). The reaction was then stirred for 24 h at room temperature and followed the course of the reaction by TLC until completion. The MeOH and THF was then removed in vacuo, and the resulting residue was diluted with H_2O (15mL), ice and EtOAc (20 mL). After acidification with 2 M HCl (pH 4-5), the solution was extracted with EtOAc for three times. The combined organic extract was washed with brine, dried over $MgSO_4$ and concentrated in vacuo. The crude was washed with AcOEt furnishing the final hydroxyacids without further purification.

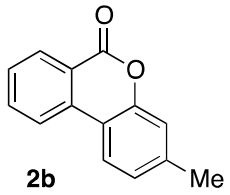


2a

6H-benzo[c]chromen-6-one (2a) Following general procedure A, [1,1'-biphenyl]-2 carboxylic acid (0.50 mmol) was used. Column chromatography: silica gel, hexanes/EtOAc 95:5. White solid; yield: 95 mg (97% yield). M.p. = 88-89 ºC. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.42 (ddd, *J* = 8.0, 1.4, 0.6 Hz, 1H), 8.14 (ddt, *J* = 8.1, 1.1, 0.5 Hz, 1H), 8.08 (dd, *J* = 8.0, 1.5 Hz, 1H), 7.84 (ddd, *J* = 8.0, 7.3, 1.4 Hz, 1H), 7.60 (ddd, *J* = 8.2, 7.3, 1.1 Hz, 1H), 7.49 (ddd, *J* = 8.5, 7.2, 1.5 Hz, 1H), 7.41-7.37 (m, 1H), 7.35 (ddd, *J* = 8.0, 7.2, 1.3 Hz, 1H) ppm. ^{13}C NMR (126 MHz, $CDCl_3$) δ 161.2, 151.3, 134.9, 134.8, 130.6, 130.5, 128.9, 124.6, 122.8, 121.7, 121.3, 118.1, 117.8 ppm. The spectroscopic data correspond to those previously reported in the literature.[11]

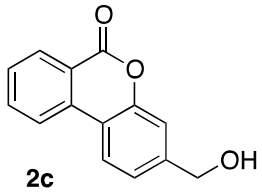
[10] Leow, D.; Li, G.; Mei, T.-S.; Yu, J.-Q. *Nature* **2012**, *486*, 518.

[11] Vishnumurthy, K.; Makriyannis, A. *J. Comb. Chem.* **2010**, *12*, 664.



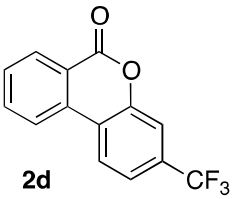
2b

3-methyl-6H-benzo[c]chromen-6-one (2b) Following general procedure A, 4'-methyl-[1,1'-biphenyl]-2 carboxylic acid (0.50 mmol) was used. Column chromatography: silica gel, hexanes/EtOAc 95:5. White solid; yield: 95 mg (90% yield). M.p. = 153-154 ºC. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.45-8.32 (m, 1H), 8.15-8.02 (m, 1H), 7.95 (d, *J* = 8.1 Hz, 1H), 7.81 (ddd, *J* = 8.1, 7.3, 1.4 Hz, 1H), 7.56 (ddd, *J* = 8.2, 7.3, 1.1 Hz, 1H), 7.21-7.12 (m, 2H), 2.46 (s, 3H) ppm. ^{13}C NMR (126 MHz, $CDCl_3$) δ 161.7, 151.6, 141.6, 135.3, 135.1, 130.8, 128.6, 126.0, 122.8, 121.7, 121.2, 118.2, 115.7, 21.8 ppm. The spectroscopic data correspond to those previously reported in the literature.[12]



2c

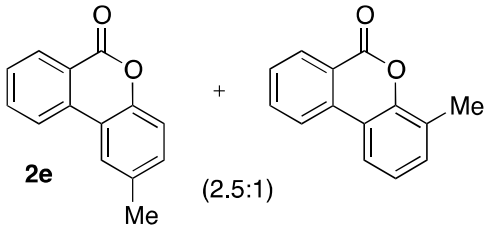
3-(hydroxymethyl)-6H-benzo[c]chromen-6-one (2c) Following general procedure A, 4'-(hydroxymethyl)-[1,1'-biphenyl]-2-carboxylic acid (0.50 mmol) was used. Column chromatography: silica gel, hexanes/EtOAc 95:5. White solid; yield: 66 mg (58% yield). M.p. = 176-177 ºC. ^{1}H NMR (500 MHz, CD_3OD) δ 8.47-8.28 (m, 2H), 8.23 (d, *J* = 8.1 Hz, 1H), 7.93 (ddd, *J* = 8.1, 7.3, 1.4 Hz, 1H), 7.73-7.56 (m, 1H), 7.48-7.30 (m, 2H), 4.74 (s, 2H) ppm. ^{13}C NMR (126 MHz, CD_3OD) δ 163.9, 153.5, 147.5, 137.3, 137.1, 132.0, 130.8, 125.2, 125.0, 124.1, 122.9, 119.0, 117.1, 65.1 ppm. IR (neat, cm^{-1}): 3294, 2920, 2850, 1732, 1608, 1307, 1267, 1049, 1030. HRMS *calcd* for ($C_{14}H_{10}O_3$+H^+): 228.0708, found 228.0732.



2d

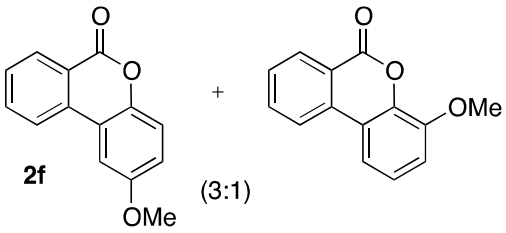
[12] Brown, B. P. M.; Russell, J.; Wylie, A. G. *J. Chem. Soc (C)* **1968**, 842.

3-(trifluoromethyl)-6H-benzo[c]chromen-6-one (2d) Following general procedure A, 4'-(trifluoromethyl)-[1,1'-biphenyl]-2-carboxylic acid (0.50 mmol) was used. Column chromatography: silica gel, hexanes/EtOAc 90:10. White solid; yield: 30 mg (22 % yield). M.p. = 123.1-124.1 ºC. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.43 (ddd, *J* = 7.9, 1.5, 0.6 Hz, 1H), 8.23-8.12 (m, 2H), 7.89 (ddd, *J* = 8.0, 7.3, 1.4 Hz, 1H), 7.68 (ddd, *J* = 8.2, 7.4, 1.1 Hz, 1H), 7.62 (d, *J* = 1.8 Hz, 1H), 7.59 (ddd, *J* = 8.4, 1.9, 0.9 Hz, 1H) ppm. ^{13}C NMR (126 MHz, $CDCl_3$) δ 160.6, 151.3, 135.5, 133.7, 132.7 (q, *J* = 32 Hz), 132.5, 131.2, 130.5, 124.7 (q, *J* = 275 Hz), 124.0, 122.5, 122.0, 121.5, 115.6 (q, *J* = 4 Hz) ppm. IR (neat, cm^{-1}): 1727, 1702, 1323, 1267, 1108. HRMS *calcd* for ($C_{14}H_{13}F_3O_2$+H^+): 264.0398, found 264.0399

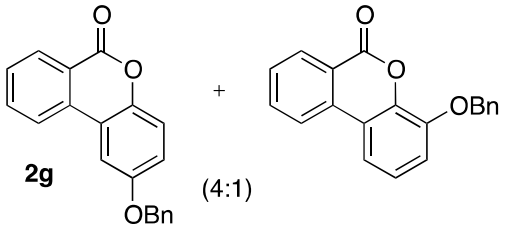


2-methyl-6H-benzo[c]chromen-6-one (2e) Following general procedure A, 3'-methyl-[1,1'-biphenyl]-2 carboxylic acid (0.50 mmol) was used. Column chromatography: silica gel, hexanes/EtOAc 95:5. White solid; yield: 84 mg (81 % yield; 2.5:1 regioisomeric mixture). ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.43 (dd, *J* = 7.9, 1.4 Hz, major + minor isomer), 8.15 (d, *J* = 8.1 Hz, major + minor), 8.01-7.90 (m, 1H), 7.90-7.72 (m, major + minor isomer), 7.59 (d, *J* = 1.1 Hz, major + minor isomer), 7.41-7.32 (m, 1H), 7.31-7.18 (m, major + minor isomer), 2.52 (s, major isomer, 3H), 2.48 (s, minor isomer, 1.18H) ppm. ^{13}C NMR (126 MHz, $CDCl_3$) δ 161.6, 161.5, 149.9, 149.6, 135.4, 135.1, 135.0, 135.0, 134.4, 132.0, 131.6, 130.8, 130.7, 128.9, 128.9, 127.3, 124.3, 123.0, 122.1, 121.9, 121.5, 121.3, 120.6, 117.9, 117.9, 117.7, 21.4, 16.3 ppm. The spectroscopic data of the major regioisomer corresponds to those previously reported in the literature.[13]

[13] Zhou, Q.; Worm, K.; Dolle, R. E., *J. Org. Chem.* **2004**, *69*, 5147.



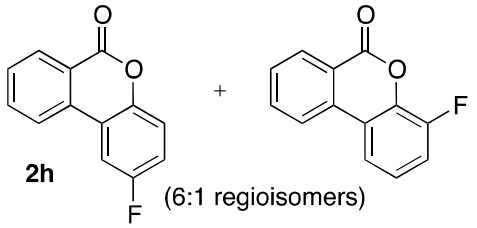
2-methoxy-6H-benzo[c]chromen-6-one (2f) Following general procedure A, 3',5'-dimethyl-[1,1'-biphenyl]-2-carboxylic acid (0.5 mmol) was used. Column chromatography: silica gel, hexanes/EtOAc 95:5. White solid; yield: 88 mg (78 % yield; 3:1 regioisomeric mixture). ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.43 (dd, *J* = 7.9, 1.6 Hz, 1H), 8.09 (d, *J* = 8.5 Hz, 1H), 7.85 (td, *J* = 7.6, 1.5 Hz, 1H), 7.61 (td, *J* = 7.7, 1.3 Hz, 1H), 7.51 (d, *J* = 2.9 Hz, 1H), 7.45-7.22 (m, 1H), 7.07 (dd, *J* = 9.0, 3.0 Hz, 1H), 3.93 (s, 3H) ppm. ^{13}C NMR (126 MHz, $CDCl_3$) δ 161.5, 160.8, 156.6, 148.3, 145.8, 141.3, 135.2, 135.0, 135.0, 134.9, 130.9, 130.8, 129.2, 129.2, 124.5, 122.4, 122.0, 121.6, 121.5, 119.0, 118.9, 118.8, 117.4, 114.4, 112.5, 106.6, 56.5, 56.1 ppm. The spectroscopic data of the major isomer corresponds to those previously reported in the literature.[14]



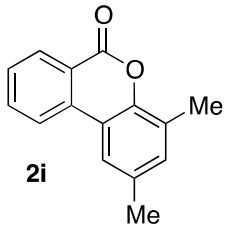
2-(benzyloxy)-6H-benzo[c]chromen-6-one (2g) Following general procedure A, 3'-(benzyloxy)-[1,1'-biphenyl]-2-carboxylic acid (0.50 mmol) was used. Column chromatography: silica gel, hexanes/EtOAc 95:5. White solid; yield: 148 mg (98 % yield; 4:1 regioisomeric mixture). ^{1}H NMR (500 MHz, CD_3OD) δ 8.40-8.30 (m, major + minor isomer), 8.11 (dd, major + minor isomer, *J* = 8.3, 1.4 Hz), 8.01-7.92 (m, 1H), 7.83-7.70 (m, major + minor isomer), 7.50-7.44 (m, 3H), 7.43-7.38 (m, 2H), 7.37-7.33 (m, 1H), 7.25 (d, *J* = 9.0 Hz, 1H), 7.08 (dd, *J* = 9.0, 2.9 Hz, 1H), 5.13 (s, 2H) ppm. ^{13}C NMR (126 MHz, $CDCl_3$) δ 172.2, 161.6, 161.0, 155.7, 147.2, 146.0, 141.9, 136.8, 136.8, 135.2, 135.0, 134.8, 134.0, 130.9, 130.8, 130.5, 129.6, 129.2, 129.2, 129.0, 129.0, 128.9, 128.8, 128.5, 128.3, 127.8, 127.6, 124.4, 122.4, 122.0, 121.6,

[14] Bowman, W. R.; Mann, E.; Parr, J. *J. Chem. Soc., Perkin Trans. 1*, **2000**, *17*, 2991

121.5, 119.3, 118.9, 118.9, 118.2, 114.9, 114.9, 108.0, 71.5, 71.0. IR (neat, cm^{-1}): 3063, 3028, 2928, 2874, 1728, 1684, 1450, 1417, 1318, 1292, 1069. HRMS *calcd* for ($C_{20}H_{14}O_3$+H^+): 303.1021, found 303.1022.

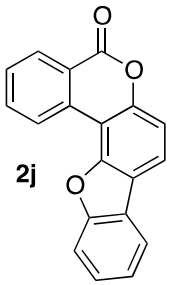


3-fluoro-6H-benzo[c]chromen-6-one (2h) Following general procedure A, 3',5'-dimethyl-[1,1'-biphenyl]-2-carboxylic acid (0.50 mmol) was used. Column chromatography: silica gel, hexanes/EtOAc 98:2. White solid; yield: 48 mg (46 % yield; 6:1 regioisomeric mixture). The spectroscopical data for the major isomer is the following: ^{1}H NMR (500 MHz, CD_3OD) δ 8.49-8.22 (m, 1H), 8.08-7.92 (m, 1H), 7.84 (ddd, *J* = 7.9, 7.3, 1.4 Hz, 1H), 7.69 (dd, *J* = 9.1, 2.9 Hz, 1H), 7.66-7.57 (m, 1H), 7.33 (dd, *J* = 9.0, 4.7 Hz, 1H), 7.18 (ddd, *J* = 9.0, 7.7, 2.9 Hz, 1H) ppm. ^{13}C NMR (126 MHz, $CDCl_3$) δ 161.1, 159.6 (d, *J* = 244.4 Hz), 147.69 (d, *J* = 2.1 Hz), 135.3, 134.8 (d, *J* = 2.8 Hz), 131.0, 129.9, 122.2, 121.6, 119.60 (d, *J* = 8.6 Hz), 119.5 (d, *J* = 8.8 Hz) 118.00 (d, *J* = 24.2 Hz), 109.11 (d, *J* = 24.9 Hz) ppm. The spectroscopic data of the major isomer corresponds to those previously reported in the literature.[12]



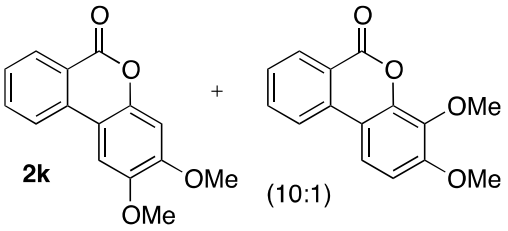
2,4-dimethyl-6H-benzo[c]chromen-6-one (2i) Following general procedure A, 3',5'-dimethyl-[1,1'-biphenyl]-2-carboxylic acid (0.50 mmol) was used. Column chromatography: silica gel, hexanes/EtOAc 95:5. White solid; yield: 83 mg (74 % yield). M.p. = 171-173 ºC. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.42 (dd, *J* = 8.1, 1.5 Hz, 1H), 8.13 (d, *J* = 8.1 Hz, 1H), 7.81 (ddd, *J* = 8.2, 7.3, 1.5 Hz, 1H), 7.76-7.68 (m, 1H), 7.65-7.46 (m, 1H), 7.21-7.04 (m, 1H), 2.47 (s, 3H), 2.43 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 161,7, 148.1, 135.5, 134.9, 133.7,

133.1, 130.8, 128.8, 127.0, 122.1, 121.4, 120.7, 117.6, 21.4, 16.2 ppm. IR (neat, cm^{-1}): 2919, 1714, 1600, 1279, 772. HRMS *calcd* for ($C_{15}H_{12}O_2+H^+$): 225.0916, found 225.0917.



2j

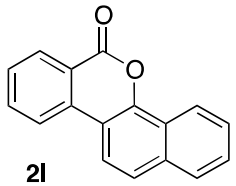
5H-benzo[c]benzofuro[2,3-f]chromen-5-one (2j) Following general procedure A, 2-(dibenzo[b,d]furan-4-yl)benzoic acid (0.50 mmol) was used. Column chromatography: silica gel, hexanes/EtOAc 9:1. Yellowish solid; yield: 102 mg (71 % yield). M.p. = 189-191 ºC. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.64 (dd, *J* = 7.9, 0.8 Hz, 1H), 8.18 (ddd, *J* = 7.9, 1.5, 0.6 Hz, 1H), 7.75-7.60 (m, 2H), 7.57 (d, *J* = 8.4 Hz, 1H), 7.46-7.36 (m, 2H), 7.31 (ddd, *J* = 8.3, 7.2, 1.4 Hz, 1H), 7.21 (td, *J* = 7.4, 1.0 Hz, 1H), 7.01 (d, *J* = 8.4 Hz, 1H) ppm. ^{13}C NMR (126 MHz, $CDCl_3$) δ 161.8, 156.5, 152.2, 150.7, 135.1, 132.8, 130.3, 128.9, 127.1, 126.5, 123.7, 123.2, 121.3, 121.0, 120.9, 120.4, 112.9, 111.9, 105.5 ppm. IR (neat, cm^{-1}): 2922, 2852, 1730, 1606, 1460, 1042. HRMS *calcd* for ($C_{19}H_{10}O_3+H^+$): 287.0708, found 287.07012.



2k (10:1)

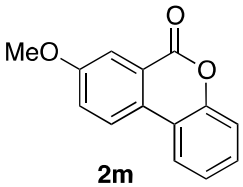
2,3-dimethoxy-6H-benzo[c]chromen-6-one (2k) Following general procedure A, 3',4'-dimethoxy-[1,1'-biphenyl]-2-carboxylic acid (0.5 mmol) was used. Column chromatography: silica gel, hexanes/EtOAc 95:5. White solid; yield: 76 mg (59 % yield); 10:1 regioisomeric mixture) ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.26 (ddd, *J* = 7.9, 1.5 Hz, 1H), 7.92-7.81 (m, 1H), 7.69 (ddd, *J* = 8.1, 7.2, 1.4 Hz, 1H), 7.41 (ddd, *J* = 8.2, 7.3, 1.1 Hz, 1H), 7.28 (s, 1H), 6.75 (s, 1H), 3.91 (s, 3H), 3.86 (s, 3H) ppm. ^{13}C NMR (126 MHz, $CDCl_3$) δ 161.9, 151.7, 146.8, 146.6, 135.4, 135.0, 130.9, 128.0, 121.3, 120.5, 110.2, 104.2,

101.1, 56.7, 56.5 ppm. The data of the major isomer corresponds to those previously reported in the literature.[15]



2l

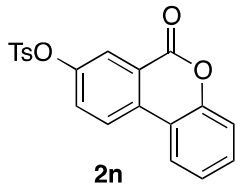
6H-dibenzo[c,h]chromen-6-one (2l) Following general procedure A, 2-(naphthalen-2-yl)benzoic acid (0.5 mmol) was used. Column chromatography: silica gel, hexanes/EtOAc 95:5. White solid; yield: 102 mg (83 % yield); M.p. = 181-182 ºC. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.60-8.46 (m, 1H), 8.46-8.34 (m, 1H), 8.11 (dd, *J* = 8.1, 1.0 Hz, 1H), 7.97 (d, *J* = 8.8 Hz, 1H), 7.87-7.77 (m, 2H), 7.69 (dd, *J* = 8.9, 0.8 Hz, 1H), 7.64-7.52 (m, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 161.5, 147.4, 135.6, 135.2, 134.5, 130.9, 128.9, 128.1, 127.9, 127.3, 124.7, 124.1, 122.6, 122.3, 121.4, 119.4, 113.2. The data correspond to those previously reported in the literature.[2]



2m

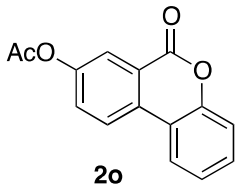
8-methoxy-6H-benzo[c]chromen-6-one (2m) Following general procedure A, 4-methoxy-[1,1'-biphenyl]-2-carboxylic acid (0.50 mmol) was used. Column chromatography: silica gel, hexanes/EtOAc 9:1. White solid; yield: 96 mg (86 % yield). M.p. = 149-151 ºC. ^{1}H NMR (500 MHz, CD_3OD) δ 8.05 (d, *J* = 8.8 Hz, 1H), 7.99 (dd, *J* = 7.9, 1.5 Hz, 1H), 7.82 (d, *J* = 2.8 Hz, 1H), 7.49-7.38 (m, 2H), 7.39-7.30 (m, 2H), 3.95 (s, 3H) ppm. ^{13}C NMR (126 MHz, $CDCl_3$) δ 161.7, 160.4, 150.8, 129.7, 128.5, 124.9, 124.6, 123.8, 122.8, 122.5, 118.5, 118.0, 111.6, 56.2 ppm. The data correspond to those previously reported in the literature.[10]

[15] Beugelmans, R.; Bois-Chonssy,M.; Chastanet, J.; Gleuher, M.; Zhu, J. P. *Heterocycles* **1993**, *36*, 2737.



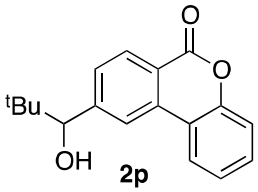
2n

6-oxo-6H-benzo[c]chromen-8-yl 4-methylbenzenesulfonate (2n) Following general procedure A, 4-(tosyloxy)-[1,1'-biphenyl]-2-carboxylic acid (0.50 mmol) was used. Column chromatography: silica gel, hexanes/EtOAc 9:1. White solid; yield: 98 mg (54 % yield). ^{1}H NMR (300 MHz, CD_3OD) δ 8.12 (d, *J* = 8.8 Hz, 1H), 8.07-7.95 (m, 1H), 7.85 (d, *J* = 2.6 Hz, 1H), 7.74 (d, *J* = 8.3 Hz, 2H), 7.64 (dd, *J* = 8.8, 2.6 Hz, 1H), 7.51 (s, 1H), 7.36 (t, *J* = 8.0 Hz, 4H), 2.46 (s, 3H) ppm. ^{13}C NMR (126 MHz, $CDCl_3$) δ 160.2, 151.4, 149.8, 146.4, 133.9, 132.1, 131.3, 130.4, 129.9, 128.8, 125.2, 124.1, 123.8, 123.2, 122.7, 118.1, 117.4, 22.1 ppm. HRMS *calcd* for ($C_{20}H_{14}O_5S$+Na): 389.0460, found 389.0465.



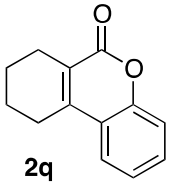
2o

6-oxo-6H-benzo[c]chromen-8-yl acetate (2o) Following general procedure A, 4-acetoxy-[1,1'-biphenyl]-2-carboxylic acid (0.50 mmol) was used. Column chromatography: silica gel, hexanes/EtOAc 9:1. White solid; yield: 66 mg (52 % yield). M.p. = 134-136 ºC. ^{1}H NMR (400 MHz, CD_3OD) δ 8.15 (d, *J* = 8.8 Hz, 1H), 8.12 (dd, *J* = 2.5, 0.5 Hz, 1H), 8.04 (dd, *J* = 8.0, 1.5 Hz, 1H), 7.59 (dd, *J* = 8.7, 2.5 Hz, 1H), 7.52-7.45 (m, 1H), 7.42-7.31 (m, 2H), 2.37 (s, 3H) ppm. IR (neat, cm^{-1}): 3071, 2923, 2852, 1742, 1685, 1608, 1221, 947. ^{13}C NMR (126 MHz, $CDCl_3$) δ 169.3, 160.7, 151.4, 151.1, 132.8, 130.8, 129.3, 125.1, 123.6, 123.2, 123.0, 122.8, 118.1, 117.8, 21.4 ppm. HRMS *calcd* for ($C_{15}H_{10}O_4$+H^+): 255.0651, found 255.0651.



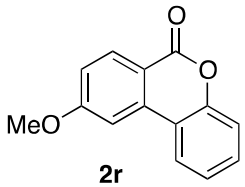
2p

9-(1-hydroxy-2,2-dimethylpropyl)-6H-benzo[c]chromen-6-one (2p) Following general procedure A, 5-(1-hydroxy-2,2-dimethylpropyl)-[1,1'-biphenyl]-2-carboxylic acid (0.50 mmol) was used. Column chromatography: silica gel, hexanes/EtOAc 4:1. White solid; yield: 106 mg (75 % yield). M.p. = 141-142 ºC. ^{1}H NMR (500 MHz, CD_3OD) δ 8.17 (d, *J* = 8.2 Hz, 1H), 8.03-7.97 (m, 1H), 7.98 (dd, *J* = 8.1, 1.5 Hz, 1H), 7.44 (ddd, *J* = 8.0, 7.4, 1.5 Hz, 2H), 7.33-7.25 (m, 2H), 4.58 (s, 1H), 0.99 (s, 9H) ppm. ^{13}C NMR (126 MHz, $CDCl_3$) δ 161.7, 151.5, 150.3, 134.3, 130.6, 129.9, 128.9, 124.9, 123.0, 120.7, 120.1, 118.3, 117.9, 82.2, 36.1, 26.2 ppm. IR (neat, cm^{-1}): 3478, 2952, 2869, 1705, 1613, 1282, 747. HRMS *calcd* for ($C_{18}H_{18}O_3$+Na): 305.1148, found 305.1155.



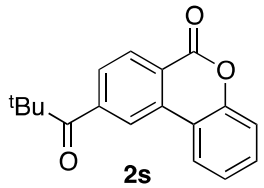
2q

7,8,9,10-tetrahydro-6H-benzo[c]chromen-6-one (2q) Following general procedure A, 3,4,5,6-tetrahydro-[1,1'-biphenyl]-2-carboxylic acid (0.50 mmol) was used. Column chromatography: silica gel, hexanes/EtOAc 95:5. White solid; yield: 65 mg (51 % yield). M.p. = 118-120 ºC. ^{1}H NMR (500 MHz, CD_3OD) δ 7.56 (dd, *J* = 7.9, 1.5 Hz, 1H), 7.45 (ddd, *J* = 8.5, 7.2, 1.5 Hz, 1H), 7.34-7.23 (m, 2H), 2.92- 2.74 (m, 2H), 2.70-2.51 (m, 2H), 1.95-1.71 (m, 4H) ppm. ^{13}C NMR (126 MHz, $CDCl_3$) δ 162.0, 152.3, 147.3, 130.5, 124.3, 124.1, 123.4, 120.5, 117.0, 25.5, 24.4, 21.9, 21.7 ppm. IR (neat, cm^{-1}): 2939, 2867, 1699, 1603, 1452, 1384, 1092, 1044, 1018. HRMS *calcd* for ($C_{13}H_{12}O_2$+Na): 223.0730, found 223.0737.



2r

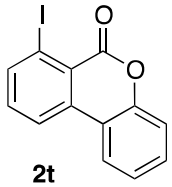
9-methoxy-6H-benzo[c]chromen-6-one (2r) Following general procedure A, 5-methoxy-[1,1'-biphenyl]-2-carboxylic acid (0.50 mmol) was used. Column chromatography: silica gel, hexanes/EtOAc 9:1. White solid; yield: 92 mg (78 % yield). M.p. = 117-119 ºC. ^{1}H NMR (500 MHz, CD_3OD) δ 8.36 (d, *J* = 8.8 Hz, 1H), 8.02 (dd, *J* = 7.9, 1.5 Hz, 1H), 7.51 (dd, *J* = 2.7, 1.9 Hz, 2H), 7.44-7.32 (m, 2H), 7.14 (dd, *J* = 8.8, 2.4 Hz, 1H), 4.02 (s, 3H) ppm. ^{13}C NMR (126 MHz, $CDCl_3$) δ 165.1, 161.3, 151.9, 137.2, 133.2, 130.8, 124.6, 123.0, 118.3, 118.1, 116.5, 114.6, 105.4, 56.0 ppm. The spectroscopic data corresponds to those previously reported in the literature.[16]



2s

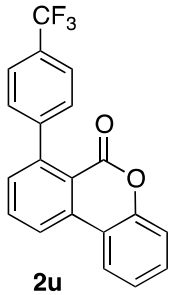
9-pivaloyl-6H-benzo[c]chromen-6-one (2s) Following general procedure A, 5-pivaloyl-[1,1'-biphenyl]-2-carboxylic acid (0.50 mmol) was used. Column chromatography: silica gel, hexanes/EtOAc 95:5. White solid; yield: 73 mg (52 % yield). M.p. = 85-86 ºC. ^{1}H NMR (500 MHz, CD_3OD) δ 8.39 (dd, *J* = 8.2, 0.6 Hz, 1H), 8.29-8.14 (m, 1H), 8.10-7.97 (m, 1H), 7.74 (dd, *J* = 8.2, 1.6 Hz, 1H), 7.48 (ddd, *J* = 8.5, 7.2, 1.5 Hz, 1H), 7.41-7.32 (m, 2H), 1.38 (s, 9H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$) δ 209.6, 160.7, 151.7, 145.3, 135.1, 131.3, 130.7, 127.2, 125.0, 123.2, 122.5, 121.1, 118.1, 117.8, 44.8, 27.9 ppm. IR (neat, cm^{-1}): 2958, 2923, 2853, 1687, 1672, 1295, 1280, 966. HRMS *calcd* for ($C_{18}H_{16}O_3$+H^+): 281.1172, found 281.1177.

[16] Cook, J. W.; Dickson, G.T.; Jack, J.; Loudon, J. D.; McKeown, J.; MacMillan, J.; Williamson, W.F. *J. Chem. Soc.* **1950**, *0*, 139.



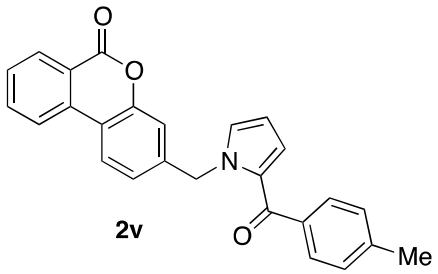
2t

7-iodo-6H-benzo[c]chromen-6-one (2t) Following general procedure A, 3-iodo-[1,1'-biphenyl]-2-carboxylic acid (0.50 mmol) was used. Column chromatography: silica gel, hexanes/EtOAc 95:5. White solid; yield: 116 mg (73 % yield). M.p. = 176-178 ºC. ^{1}H NMR (500 MHz, CD_3OD) δ 8.28 (dd, *J* = 7.7, 1.1 Hz, 1H), 8.15 (dd, *J* = 8.1, 1.1 Hz, 1H), 8.00 (dd, *J* = 8.4, 1.5 Hz, 1H), 7.50 (ddd, *J* = 8.6, 7.3, 1.5 Hz, 1H), 7.39 (t, *J* = 7.9 Hz, 1H), 7.36-7.31 (m, 2H) ppm. ^{13}C NMR (126 MHz, $CDCl_3$) δ 158.4, 151.2, 143.9, 137.3, 134.8, 131.3, 124.8, 123.3, 122.5, 121.9, 117.95, 117.8, 97.7 ppm. IR (neat, cm^{-1}): 2955, 2922, 2853, 1727, 1253, 1220, 1048, 749. HRMS *calcd* for ($C_{13}H_7IO_2$+Na): 344.9383, found 344.9398.



2u

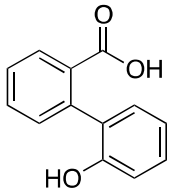
7-(4-(trifluoromethyl)phenyl)-6H-benzo[c]chromen-6-one (2u) Following general procedure A, 4-(trifluoromethyl)-[1,1':3',1''-terphenyl]-2'-carboxylic acid (0.5 mmol) was used. Column chromatography: silica gel, hexanes/EtOAc 95:5. White solid; yield: 72 mg (85 % yield; <20:1 regioisomeric mixture). White solid. M.p. = 145-147 ºC. 1H NMR (500 MHz, CDCl3) δ 8.24 (dd, *J* = 8.3, 1.1 Hz, 1H), 8.17-8.10 (m, 1H), 7.84 (dd, *J* = 8.1, 7.5 Hz, 1H), 7.74-7.67 (m, 2H), 7.51 (ddd, *J* = 8.1, 7.3, 1.5 Hz, 1H), 7.47-7.41 (m, 2H), 7.41-7.32 (m, 3H) ppm. 13C NMR (126 MHz, CDCl3) δ 159.6, 151.8, 146.0, 145.7, 136.7, 134.1, 132.4, 131.1, 129.8, 129.6, 129.0, 125.8 (d, *J* = 272 Hz), 125.2, 125.1 (d, *J* = 3.8 Hz), 125.1 (d, *J* = 3.8 Hz), 124.8, 123.6, 123.5, 122.2, 119.0, 118.2, 117.9 ppm. IR (neat, cm^{-1}): 2922, 2851, 1717, 1323, 1095, 813. HRMS *calcd* for ($C_{20}H_{11}F_3O_2$+Na): 363.0603, found 363.0607.



2v

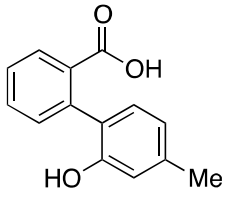
3-((2-(4-methylbenzoyl)-1H-pyrrol-1-yl)methyl)-6H-benzo[c]chromen-6-one (2v) Following general procedure A (0.17 mmol scale). Column chromatography: silica gel, hexanes/EtOAc 4:1. White solid; yield: 44 mg (64 % yield). M.p. = 168.5-169.1 ºC. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.37 (ddd, *J* = 7.9, 1.4, 0.6 Hz, 1H), 8.12-8.02 (m, 1H), 7.98 (d, *J* = 8.2 Hz, 1H), 7.79 (ddd, *J* = 8.1, 7.3, 1.4 Hz, 1H), 7.69 (d, *J* = 8.1 Hz, 2H), 7.56 (ddd, *J* = 8.2, 7.4, 1.1 Hz, 1H), 7.25-7.21 (m, 2H), 7.19 (dd, *J* = 8.2, 1.8 Hz, 1H), 7.07 (dd, *J* = 2.6, 1.7 Hz, 1H), 7.04-6.99 (m, 1H), 6.84 (dd, *J* = 4.0, 1.7 Hz, 1H), 6.28 (dd, *J* = 4.1, 2.6 Hz, 1H), 5.74 (s, 2H), 2.40 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 186.3, 161.5, 151.9, 142.5, 142.2, 137.2, 135.2, 135.0, 131.1, 130.9, 130.5, 129.8, 129.1, 129.1, 123.7, 123.4, 122.0, 121.4, 117.5, 116.0, 109.4, 52.3 ppm. IR (neat, cm^{-1}): 1721, 1616, 1602, 1406, 1391, 1259, 1101, 765. HRMS *calcd* for ($C_{26}H_{19}NO_3$+Na): 416.1263, found 416.1257.

Synthesis of 2'-hydroxy-[1,1'-biphenyl]-2-carboxylic acid (table 4)



3a

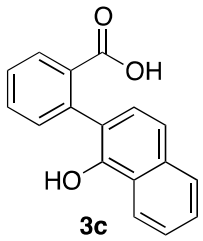
2'-hydroxy-[1,1'-biphenyl]-2-carboxylic acid (3a) Following general procedure B, 1,1'-biphenyl]-2 carboxylic acid (0.25 mmol) was used. White solid; yield: 50 mg (91 % yield) M.p. = 176-178 ºC. ^{1}H NMR (400 MHz, CD_3OD) δ 8.39-8.25 (m, 2H), 8.21 (dd, *J* = 8.0, 1.5 Hz, 1H), 7.90 (ddd, *J* = 8.1, 7.3, 1.4 Hz, 1H), 7.64 (ddd, *J* = 8.2, 7.3, 1.1 Hz, 1H), 7.53 (ddd, *J* = 8.2, 7.3, 1.5 Hz, 1H), 7.45-7.20 (m, 2H) ppm. ^{13}C NMR (126 MHz, CD_3OD) δ 163.5, 153.2, 137.2, 136.9, 132.5, 131.9, 130.9, 126.7, 125.1, 124.1, 122.9, 120.1, 119.2 ppm. The spectroscopical data corresponds to those previously reported in the literature. [17]



3b

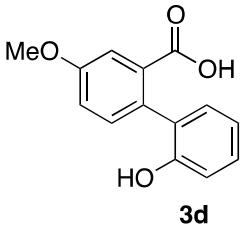
2'-hydroxy-4'-methyl-[1,1'-biphenyl]-2-carboxylic acid (3b) Following general procedure B, 4'-methyl-[1,1'-biphenyl]-2 carboxylic acid (0.25 mmol) was used. White solid; yield: 54 mg (84 % yield). M.p. = 131-132 ºC. ^{1}H NMR (400 MHz, CD_3OD) δ 8.30 (dd, *J* = 20.2, 8.0 Hz, 2H), 8.11 (d, *J* = 8.1 Hz, 1H), 8.02-7.70 (m, 1H), 7.62 (t, *J* = 7.6 Hz, 1H), 7.43-6.96 (m, 2H), 2.47 (s, 3H) ppm. ^{13}C NMR (126 MHz, DMSO-d_6) δ 161.3, 151.6, 142.1, 136.2, 135.4, 130.6, 129.7, 126.7, 124.3, 123.3, 121.1, 118.2, 116.0, 21.8.IR (neat, cm^{-1}): 3063, 3038, 2922, 2854, 1737, 1606, 1459, 1308. HRMS *calcd* for ($C_{14}H_{12}O_3$-OH): 226.0624, found 226.0631

[17] Hey, D. H.; Saunders, F. C.; Williams, G. H. *J. Chem. Soc.* **1961**, *0*, 554.



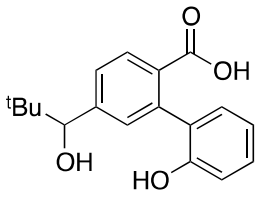
3c

2-(3-hydroxynaphthalen-2-yl)benzoic acid (3c) Following general procedure B, 2-(naphthalen-2-yl)benzoic acid (0.25 mmol) was used. White solid; yield: 51 mg (66 % yield). M.p. = 193-194 ºC. ^{1}H NMR (400 MHz, DMSO-d_6) δ 8.51 (dd, *J* = 8.0, 1.1 Hz, 1H), 8.42-8.36 (m, 2H), 8.34 (dd, *J* = 7.9, 1.4 Hz, 1H), 8.10-7.97 (m, 2H), 7.92 (dd, *J* = 8.8, 0.8 Hz, 1H), 7.79-7.67 (m, 3H) ppm. ^{13}C NMR (126 MHz, DMSO-d_6) δ 161.1, 147.3, 136.4, 135.7, 134.8, 130.7, 130.1, 128.8, 128.2, 125.3, 123.9, 123.8, 122.2, 121.5, 121.0, 113.9 ppm. IR (neat, cm^{-1}): 2951, 2904, 2880, 1736, 1719, 1609, 1101, 755. HRMS *calcd* for ($C_{17}H_{12}O_3$-OH): 247.0754, found 247.0762.



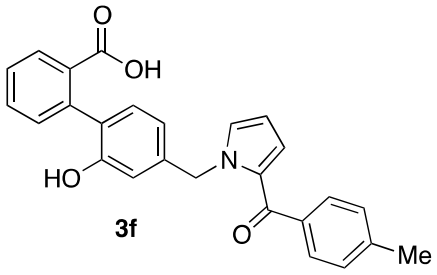
3d

2'-hydroxy-4-methoxy-[1,1'-biphenyl]-2-carboxylic acid (3d) Following general procedure B, 4-methoxy-[1,1'-biphenyl]-2-carboxylic acid (0.25 mmol) was used. White solid; yield: 45 mg (78 % yield). M.p. = 131-132 ºC. ^{1}H NMR (400 MHz, DMSO-d_6) δ 8.38 (d, *J* = 8.9 Hz, 1H), 8.30 (dd, *J* = 7.9, 1.5 Hz, 1H), 7.67 (d, *J* = 2.8 Hz, 1H), 7.62-7.49 (m, 2H), 7.47-7.37 (m, 1H), 3.94 (s, 3H) ppm. ^{13}C NMR (126 MHz, DMSO-d_6) δ 161.0, 160.6, 150.8, 130.5, 128.5, 125.7, 125.5, 124.6, 124.0, 122.8, 118.7, 118.0, 112.1, 56.6. IR (neat, cm^{-1}): 3006, 2922, 2845, 1710, 1612, 1481, 1460, 1298, 1279, 1242. HRMS *calcd* for ($C_{14}H_{12}O_4$-OH): 227.0703, found 227.0714.



3e

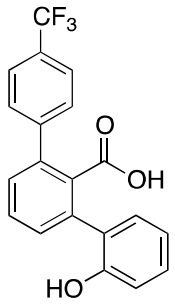
2'-hydroxy-5-(1-hydroxy-2,2-dimethylpropyl)-[1,1'-biphenyl]-2-carboxylic acid (3e) Following general procedure B, 5-(1-hydroxy-2,2-dimethylpropyl)-[1,1'-biphenyl]-2-carboxylic acid (0.25 mmol) was used. White solid; yield: 51 mg (63 % yield). M.p. = 143-145 ºC. ^{1}H NMR (400 MHz, DMSO-d_6) δ 8.39 (dd, *J* = 8.3, 1.6 Hz, 1H), 8.32 (d, *J* = 1.5 Hz, 1H), 8.24 (d, *J* = 8.2 Hz, 1H), 7.67 (dd, *J* = 8.2, 1.4 Hz, 1H), 7.64-7.57 (m, 1H), 7.51-7.40 (m, 2H), 5.60 (d, *J* = 4.4 Hz, 1H), 4.53 (d, *J* = 4.3 Hz, 1H), 0.93 (s, 9H) ppm. ^{13}C NMR (126 MHz, DMSO-d_6) δ 170.6, 162.1, 161.2, 143.6, 141.0, 139.3, 139.1, 135.2, 133.8, 131.7, 129.5, 128.3, 127.7, 90.0, 45.8, 36.3 ppm. IR (neat, cm^{-1}): 3481, 2953, 2868, 1705, 1613, 1420, 1283, 1235, 747. HRMS *calcd* for ($C_{18}H_{20}O_4$-OH): 283.1329, found 283.1337.



3f

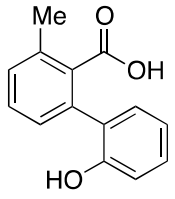
2'-hydroxy-4'-((2-(4-methylbenzoyl)-1H-pyrrol-1-yl)methyl)-[1,1'-biphenyl]-2-carboxylic acid (3f). Following general procedure B, 3-((2-(4-methylbenzoyl)-1H-pyrrol-1-yl)methyl)-6H-benzo[c]chromen-6-one was used. White solid; yield: 40 mg (95 % yield). M.p. = 169.8-171.2 ºC. ^{1}H NMR (500 MHz, DMSO-d_6) δ 8.39 (d, *J* = 8.1 Hz, 1H), 8.32 (d, *J* = 8.3 Hz, 1H), 8.25 (dd, *J* = 7.9, 1.4 Hz, 1H), 8.02-7.91 (m, 1H), 7.78-7.67 (m, 1H), 7.64 (d, *J* = 8.1 Hz, 2H), 7.59-7.50 (m, 1H), 7.33 (d, *J* = 7.8 Hz, 2H), 7.18 (dd, *J* = 8.2, 1.7 Hz, 1H), 7.11 (d, *J* = 1.7 Hz, 1H), 6.81 (dd, *J* = 4.1, 1.7 Hz, 1H), 6.35 (dd, *J* = 4.0, 2.5 Hz, 1H), 5.78 (s, 2H), 2.40 (s, 3H) ppm. ^{13}C NMR (126 MHz, $CDCl_3$) δ 185.8, 161.2, 151.7, 143.3, 142.9, 137.5, 136.4, 135.1, 133.1, 130.7, 130.2, 130.2, 130.0, 129.8, 124.9, 124.0, 123.9, 123.5, 121.4, 117.6, 116.0, 109.9,

51.9, 22.0 ppm. IR (neat, cm^{-1}): 3121, 1736, 1621, 1605, 1407, 1393, 1102, 744. HRMS *calcd* for ($C_{26}H_{20}NO_3$-OH): 394.1438, found 394.1438.



3g

2-hydroxy-4''-(trifluoromethyl)-[1,1':3',1''-terphenyl]-2'-carboxylic acid (3g) Following general procedure B, 4-(trifluoromethyl)-[1,1':3',1''-terphenyl]-2'-carboxylic acid (0.25 mmol) was used. White solid; yield: 85 mg (80 % yield). M.p. = 95-97 ºC. ^{1}H NMR (400 MHz, DMSO-d_6) δ 8.58 (dd, *J* = 8.2, 1.2 Hz, 1H), 8.46 (dd, *J* = 8.1, 1.5 Hz, 1H), 8.01 (t, *J* = 7.7 Hz, 1H), 7.79 (d, *J* = 8.0 Hz, 2H), 7.67-7.59 (m, 3H), 7.50 (dd, *J* = 7.4, 1.1 Hz, 1H), 7.48-7.40 (m, 2H) ppm. ^{13}C NMR (126 MHz, DMSO-d_6) δ 159.5, 151.7, 147.1, 144.9, 136.7, 135.3, 133.0, 131.9, 130.1, 128.4 (d, *J* = 31.8 Hz), 126.5 (d, *J* = 272 Hz), 125.6 , 125.3 (q, *J* = 3.8 Hz), 125.0 , 124.3 (d, *J* = 272 Hz), 123.7 , 119.0, 118.6 , 117.8 ppm. IR (neat, cm^{-1}): 3065, 2959, 2923, 1682, 1293, 1272.



3h

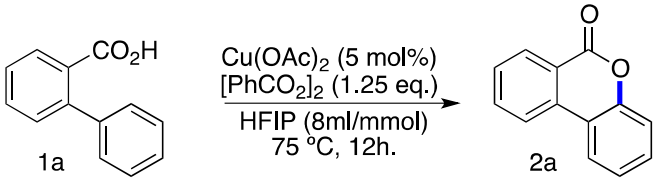
2'-hydroxy-3-methyl-[1,1'-biphenyl]-2-carboxylic acid (3h) Following general procedure B, 3-methyl-[1,1'-biphenyl]-2-carboxylic acid (0.25 mmol) was used. White solid; yield: 46 mg (81 % yield). M.p. = 100-101 ºC. ^{1}H NMR (400 MHz, DMSO-d_6) δ 8.22 (ddd, *J* = 16.2, 8.1, 1.3 Hz, 2H), 7.74 (t, *J* = 7.8 Hz, 1H), 7.52 (dd, *J* = 8.1, 1.5 Hz, 1H), 7.47-7.41 (m, 1H), 7.40-7.27 (m, 2H), 2.73 (s, 3H). ^{13}C NMR (126 MHz, DMSO-d_6) δ 160.4, 151.6, 135.3, 133.2, 131.5, 130.2, 129.5, 125.4, 124.7, 121.5, 119.8, 118.8, 117.7, 24.3 ppm. IR

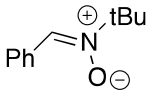
(neat, cm^{-1}): 3422, 3069, 3044, 2961, 2920, 2850, 1717, 1599, 1242, 1206. HRMS *calcd* for ($C_{14}H_{12}O_3$-OH): 211.0759, found 211.0763.

Mechanistic Considerations

Radical scavenger test

Following general procedure A, [1,1'-biphenyl]-2 carboxylic acid (0.5 mmol) and radical scavenger (0.625 mmol) were used.



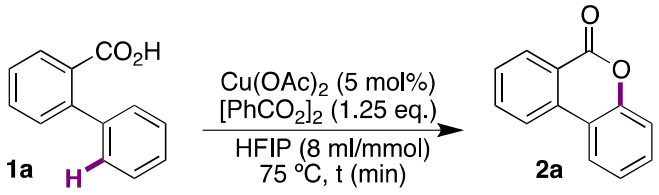
Entry	Radical scavenger	Yield[a]
1	TEMPO	0%
2		3%
3	BHT	17%
4		20%

[a] Yield determined by HPLC using naphtalene as an internal standard. TEMPO: 2,2,6,6-Tetramethyl-1-piperidinyloxy, BHT: 2,6-Di-tert-butyl-4-methylphenol

Kinetic isotope effect

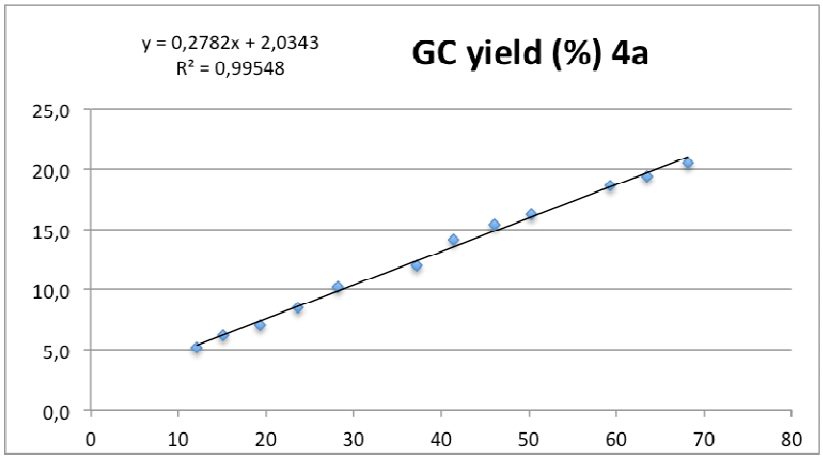
General Procedure. Kinetic experiments were run in a reactor tube filled with Ar. Reactions were run up to 50 % conversions. Only values up to 5-25%

were considered for measuring the kinetic isotope effect and the data (% product versus time) was analyzed using the initial rates method. Product yield from the corresponding reaction was monitored by GC analysis using decane as internal standard in the indicated interval based on the amount of 6H-benzo[c]chromen-6-one. The calculated KIE = 1.22.



t (min)	Area (decane)	Area (4a)	GC yield (%)
12,12	93,95	6,05	5,2
15,12	92,91	7,09	6,2
19,34	91,97	8,03	7,1
23,6	90,45	9,551	8,6
28,24	88,79	11,21	10,2
37,16	87,104	12,895	12,0
41,4	85,16	14,84	14,1
46,06	84,05	15,95	15,4
50,32	83,26	16,74	16,3
59,22	81,273	18,727	18,7
63,44	80,69	19,306	19,4
68,12	79,814	20,186	20,5

[a] **1a** (0.69 mmol), $Cu(OAc)_2$ (5 mol%), $[PhCO_2]_2$ (1.25 equiv.), HFIP (8 mL/mmol) at 75 ºC. [b] GC yield using decane as an internal standard.



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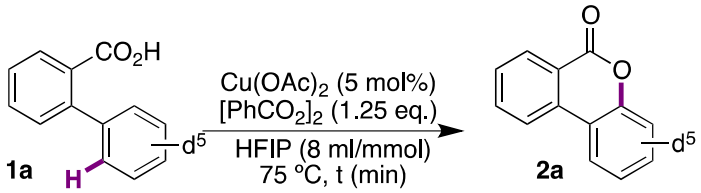
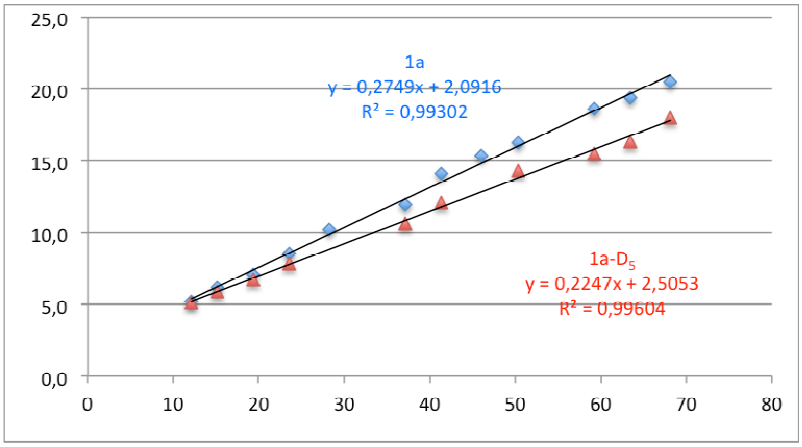


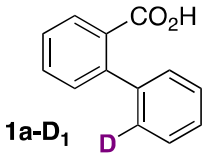
t (min)	Area (decane)	Area (4a)	GC yield (%)
12,12	93,98	6,018	5,2
15,12	93,22	6,7765	5,9
19,34	92,32	7,679	6,7
23,6	91,149	8,85	7,9
37,16	88,407	11,593	10,6
41,4	86,97	13,032	12,1
50,32	84,741	15,009	14,3
59,22	83,917	16,083	15,5
63,44	83,222	16,777	16,3
68,12	81,794	18,21	18,0

[a] **1a-D^5** (0.69 mmol), $Cu(OAc)_2$ (5 mol%), $[PhCO_2]_2$ (1.25 equiv.), HFIP (8 mL/mmol) at 75 ºC. [b] GC yield using decane as an internal standard.

KIE (1a vs 1a-D^5)



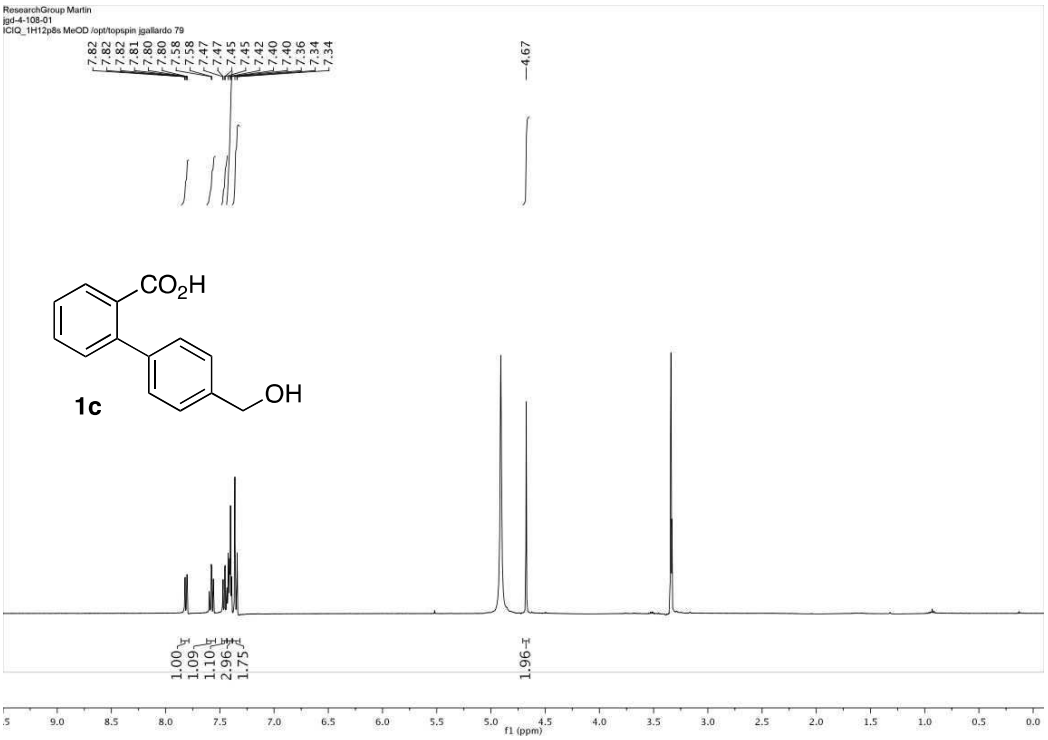
Intermolecular Isotope Effect

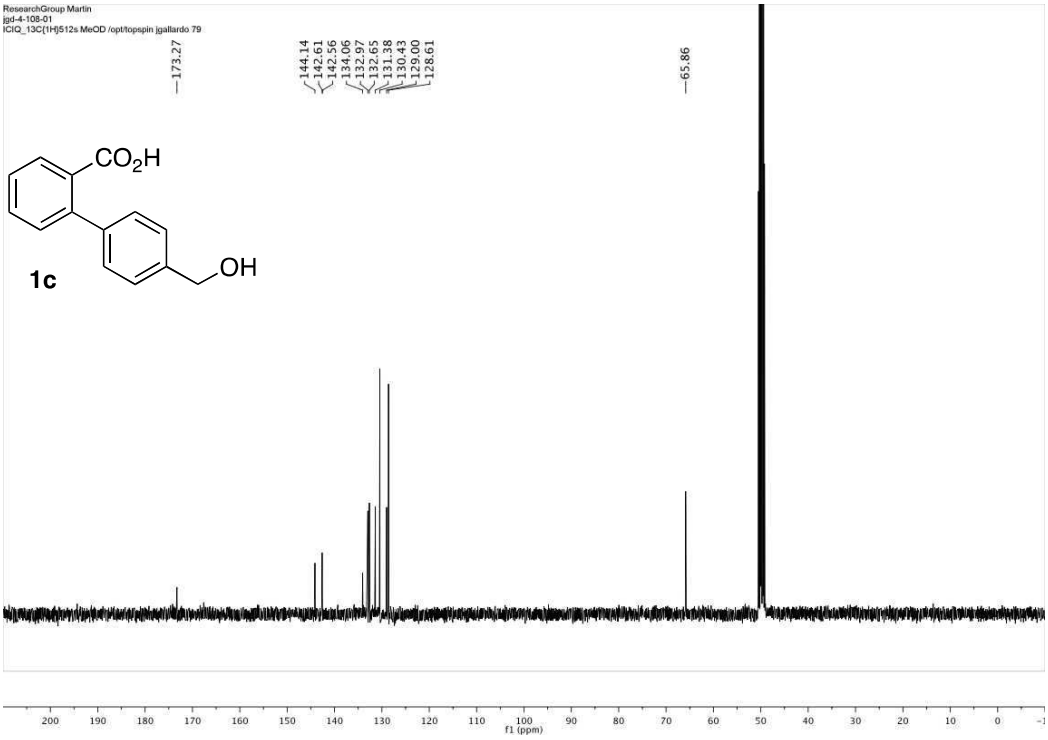


k_H/k_D =1.50

Following general procedure A, 5-methoxy-[1,1'-biphenyl]-2-carboxylic acid (0.50 mmol) was used. The reaction was stopped at 0.5h. Column chromatography: silica gel, hexanes/EtOAc 95:5. White solid; yield: 26 mg (26 % yield). The KIE observed was 1.50.

^{1}H NMR and ^{13}C NMR spectra


ResearchGroup Martin
jgd-4-108-01
ICIQ_1H12p8s MeOD /opt/topspin jgallardo 79
7.82
7.82
7.82
7.81
7.80
7.80
7.58
7.47
7.47
7.45
7.45
7.42
7.40
7.40
7.36
7.34
7.34
4.67
CO2H
OH
1c
1.00
1.09
1.10
2.96
1.75
1.96
f1 (ppm)


ResearchGroup Martin
jgd-4-108-01
ICIQ_13C{1H}512s MeOD /opt/topspin jgallardo 79
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142.56
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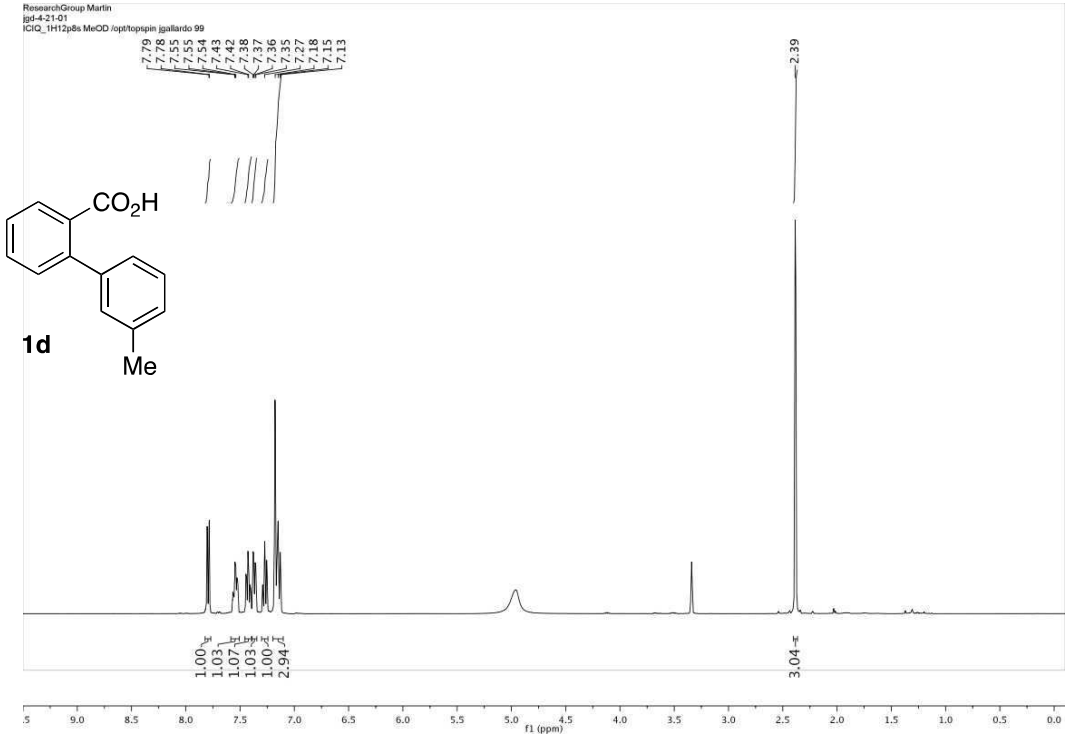
65.86
CO2H
OH
1c
f1 (ppm)


ResearchGroup Martin
jgd-4-21-01
ICIQ_1H12p8s MeOD /opt/topspin jgallardo 99
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7.55
7.55
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7.42
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7.37
7.36
7.35
7.27
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7.15
7.13
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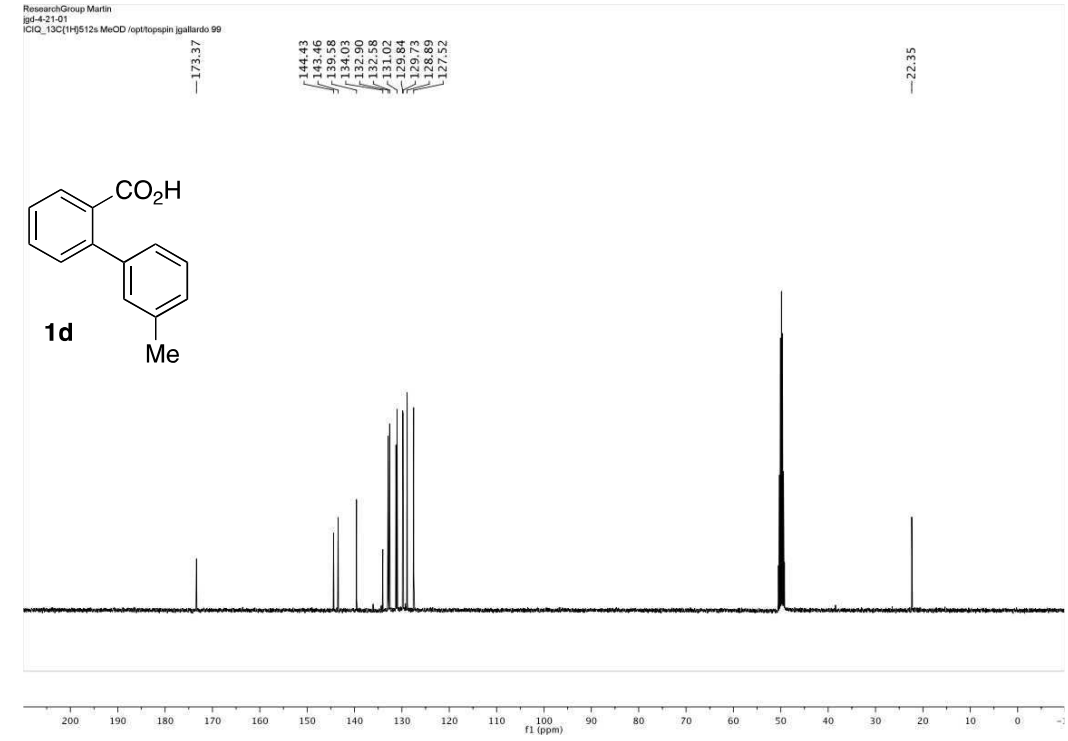
1.07
1.03
1.00
2.94
3.04
f1 (ppm)


ResearchGroup Martin
jgd-4-21-01
ICIQ_13C{1H}512s MeOD /opt/topspin jgallardo 99
173.37
144.43
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135.58
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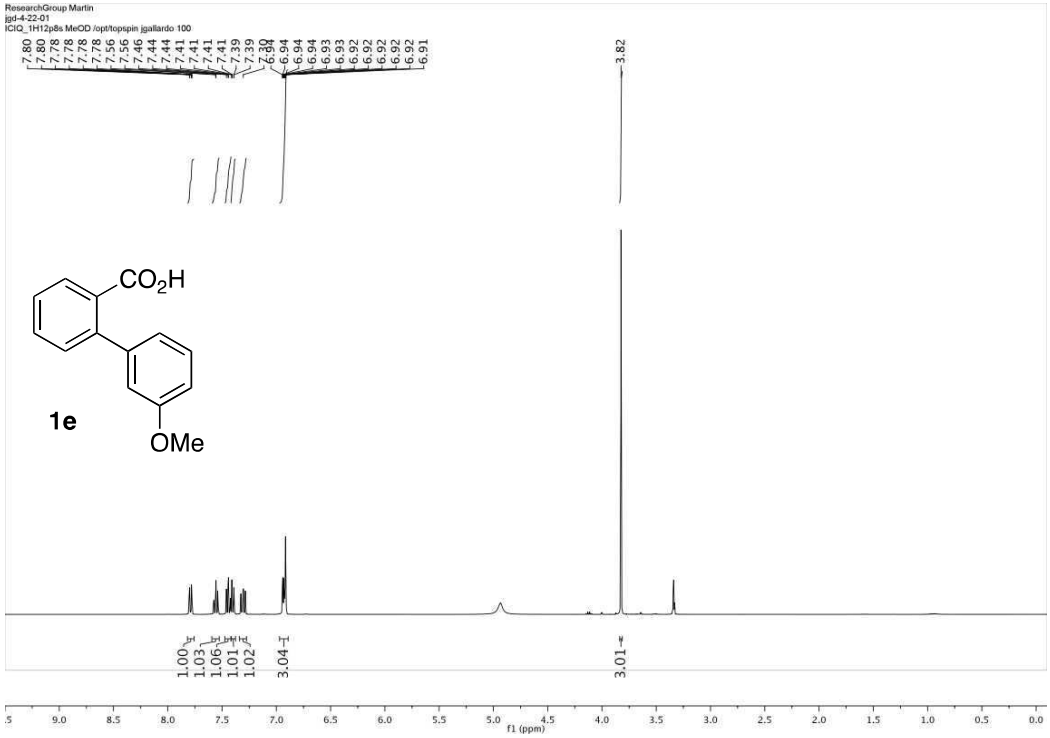
127.52
22.35
CO2H
1d
Me
f1 (ppm)


ResearchGroup Martin
jgd-4-22-01
ICIQ_1H12p8s MeOD /opt/topspin jgallardo 100
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7.80
7.78
7.78
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6.94
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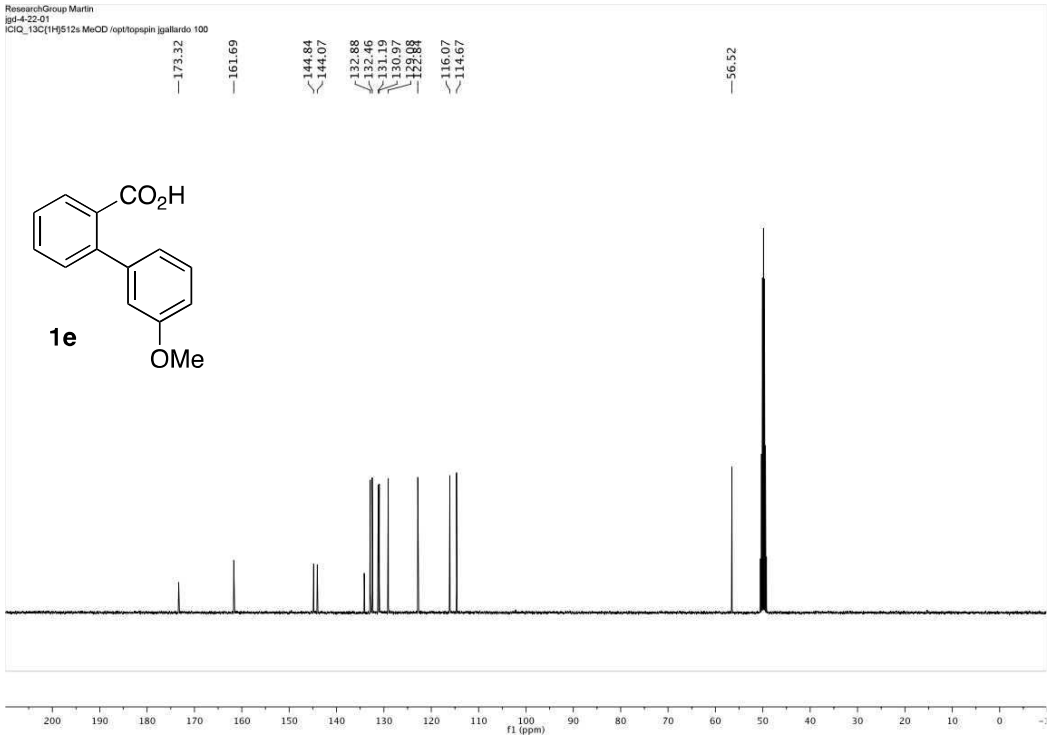
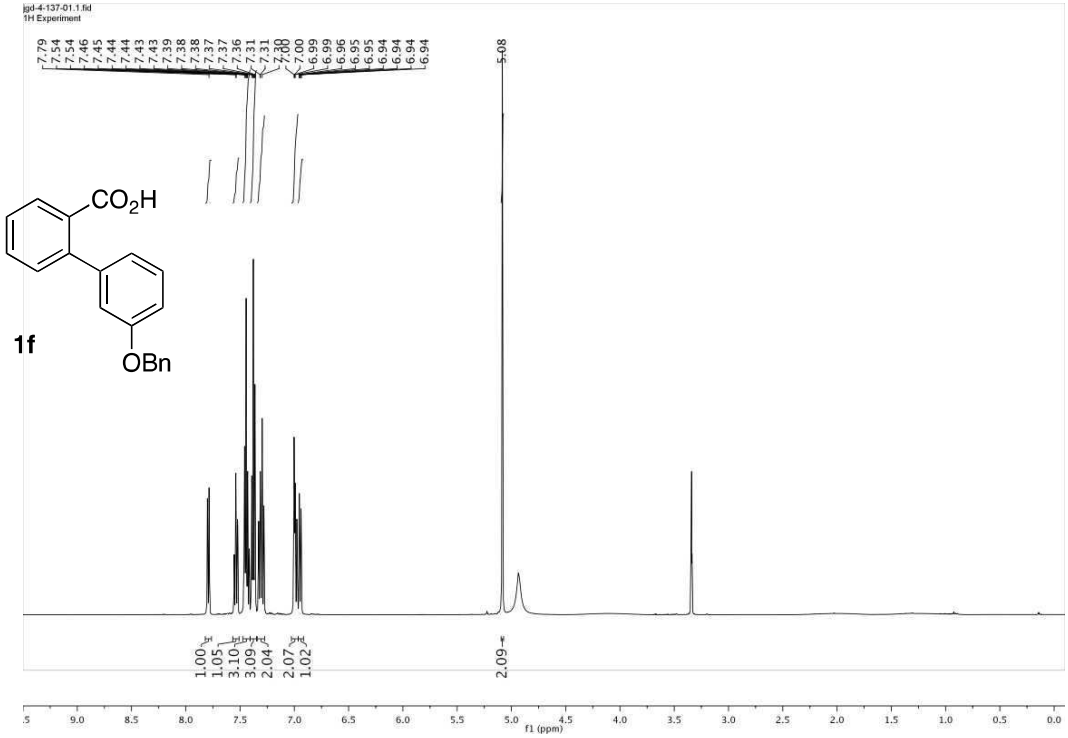
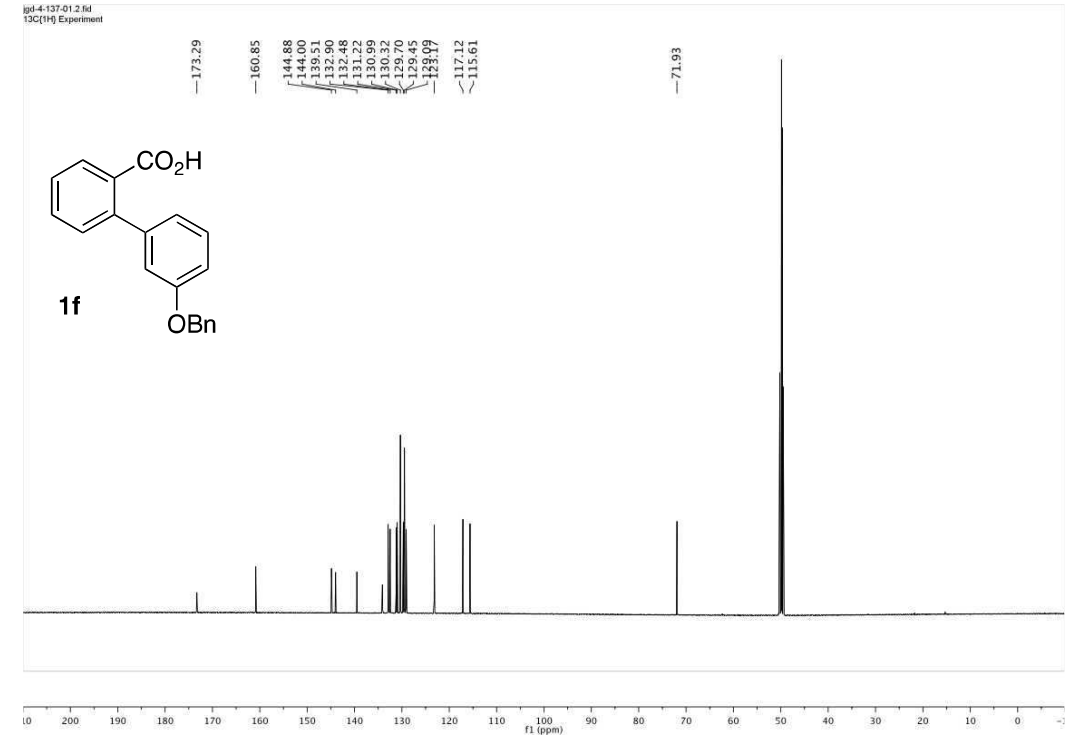
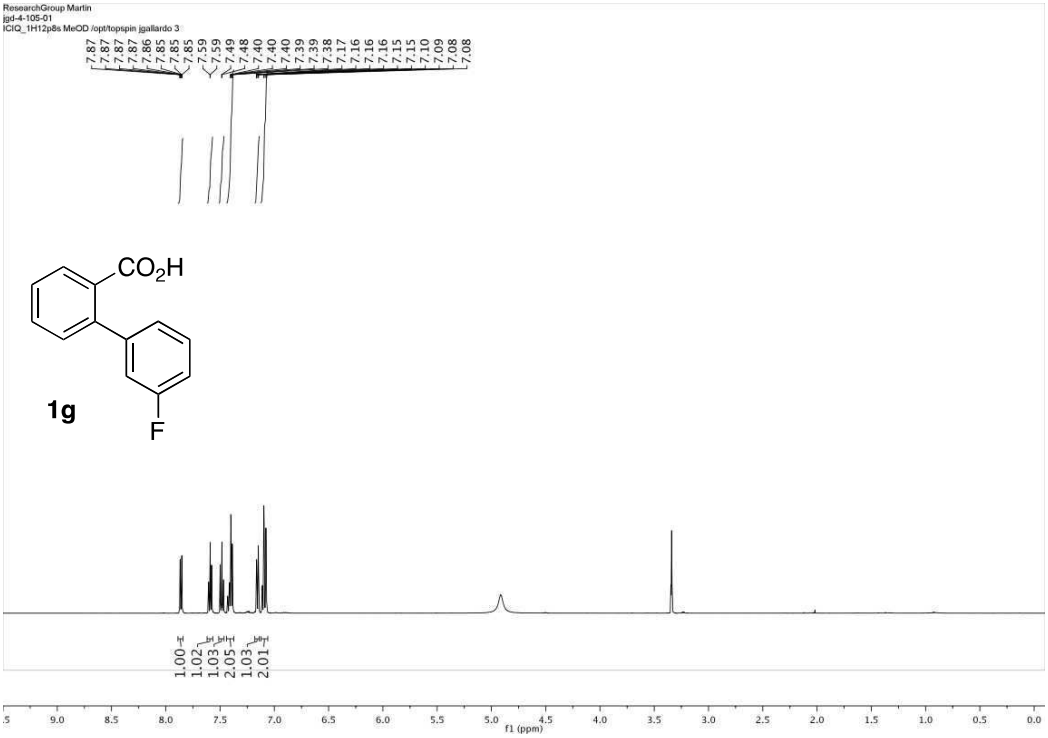
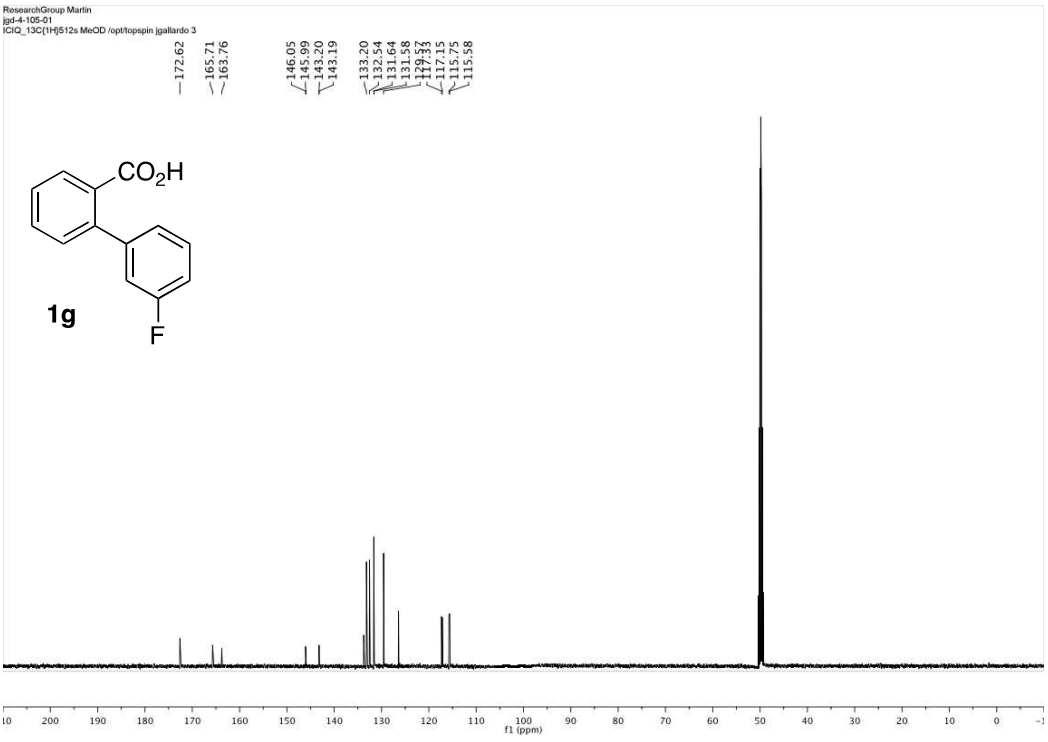
6.93
6.93
6.92
6.92
6.92
6.92
6.91
3.82
CO2H
1e
OMe
1.00
1.03
1.06
1.01
1.02
3.04
3.01
f1 (ppm)

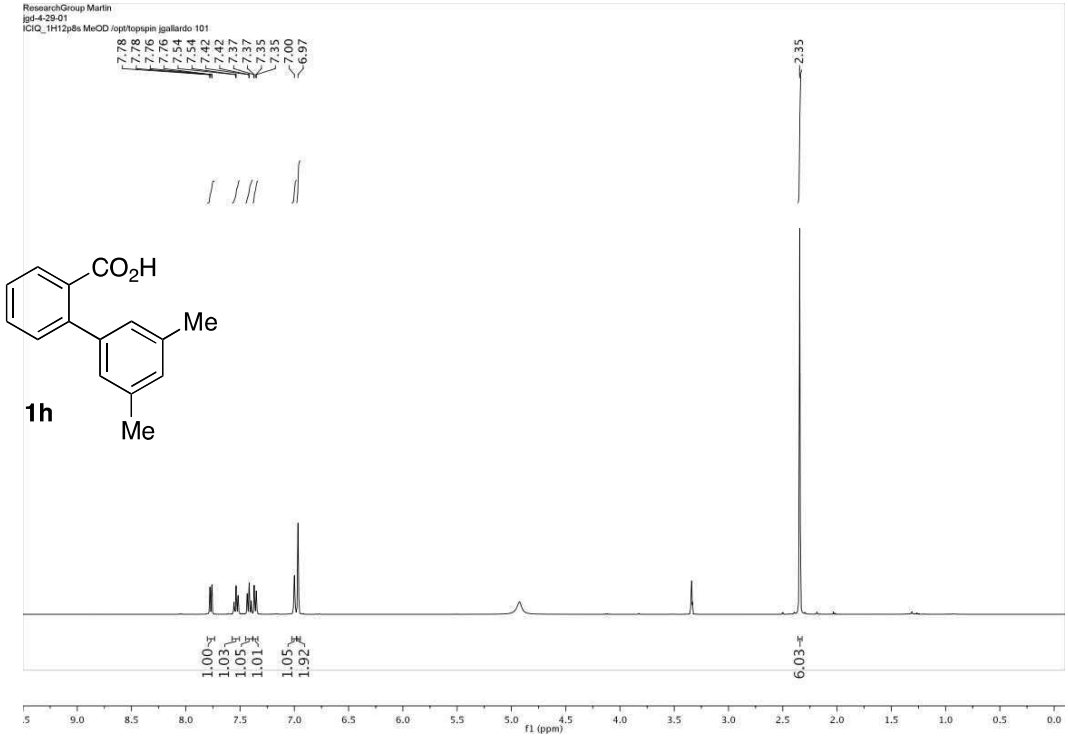

ResearchGroup Martin
jgd-4-22-01
ICIQ_13C{1H}512s MeOD /opt/topspin jgallardo 100
173.32
161.69
144.84
144.07
132.88
132.46
131.19
130.97
122.88
116.07
114.67
56.52
CO2H
1e
OMe
f1 (ppm)

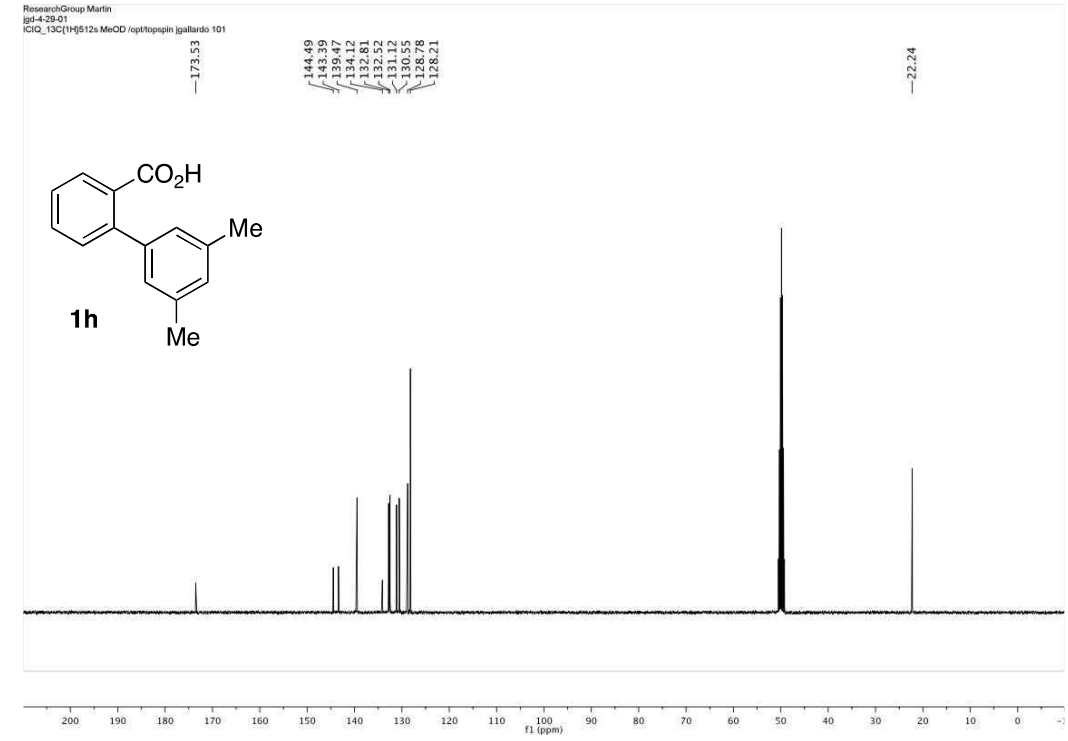

jgd-4-137-01.1.fid
1H Experiment
7.79
7.54
7.54
7.46
7.45
7.44
7.44
7.43
7.43
7.39
7.38
7.38
7.37
7.37
7.36
7.31
7.31
7.00
6.99
6.99
6.96
6.95
6.95
6.94
6.94
6.94
6.94
5.08
CO2H
1f
OBn
1.00
1.05
3.10
3.09
2.04
2.07
1.02
2.09
f1 (ppm)

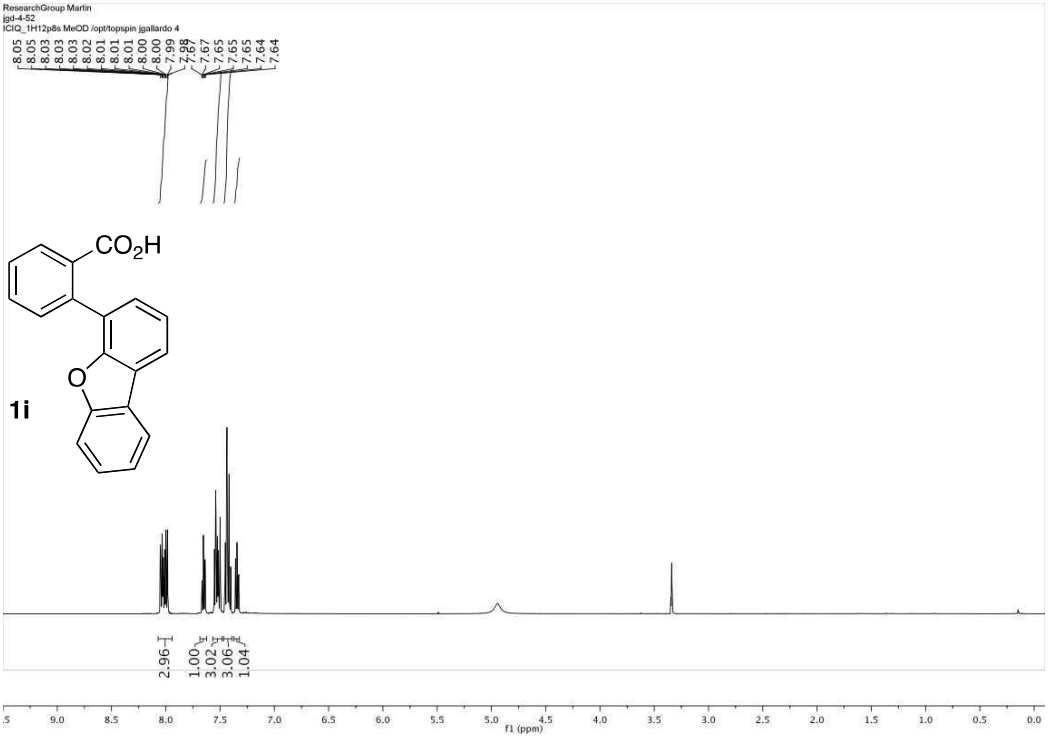

jgd-4-137-01.2.fid
13C{1H} Experiment
173.29
160.85
144.88
144.00
139.51
132.90
132.48
131.22
130.99
130.32
129.70
128.45
123.09
123.07
117.12
115.61
71.93
CO2H
1f
OBn
f1 (ppm)

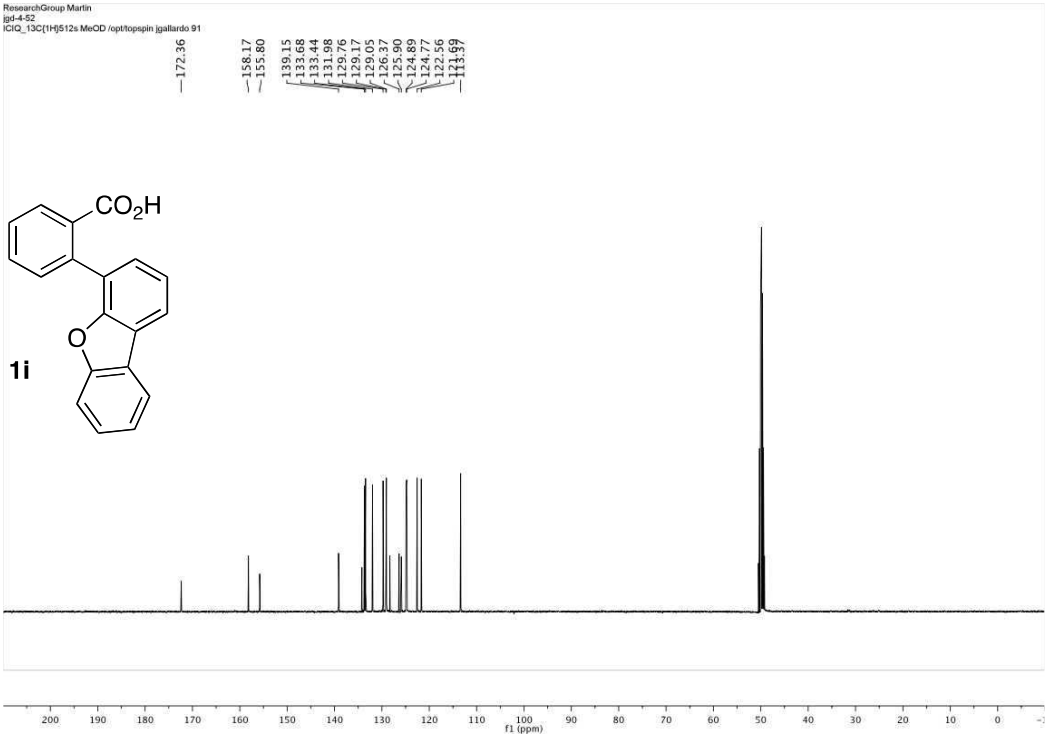

ResearchGroup Martin
jgd-4-105-01
ICIQ_1H12p8s MeOD /opt/topspin jgallardo 3
7.87
7.87
7.87
7.87
7.86
7.85
7.85
7.59
7.59
7.49
7.48
7.40
7.40
7.40
7.39
7.39
7.38
7.17
7.16
7.16
7.15
7.15
7.10
7.09
7.08
7.08
CO2H
1g
F
1.00
1.02
1.03
2.05
1.03
2.01
f1 (ppm)

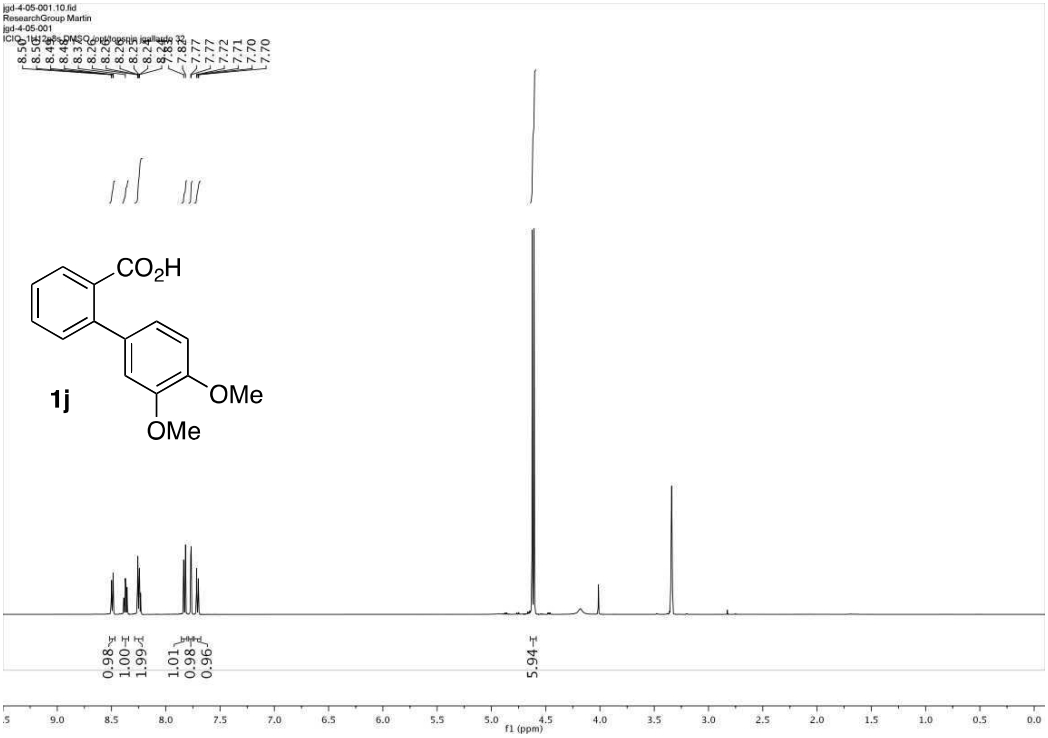

ResearchGroup Martin
jgd-4-105-01
ICIQ_13C{1H}512s MeOD /opt/topspin jgallardo 3
172.62
165.71
163.76
146.05
145.99
143.20
143.19
133.20
132.54
131.64
131.58
129.57
129.33
117.15
115.75
115.58
CO2H
1g
F
f1 (ppm)

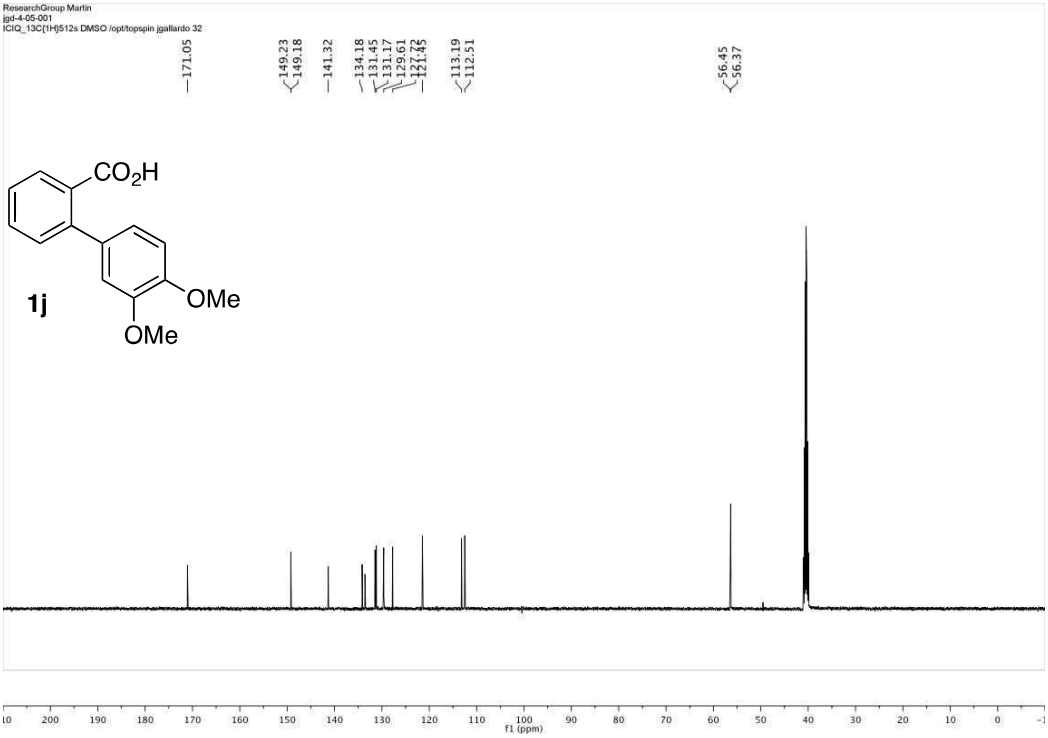

ResearchGroup Martin
jgd-4-29-01
ICIQ_1H12p8s MeOD /opt/topspin jgallardo 101
7.78
7.78
7.76
7.76
7.54
7.54
7.42
7.42
7.37
7.37
7.35
7.35
7.00
6.97
2.35
CO2H
Me
Me
1h
1.00
1.03
1.05
1.01
1.05
1.92
6.03
f1 (ppm)

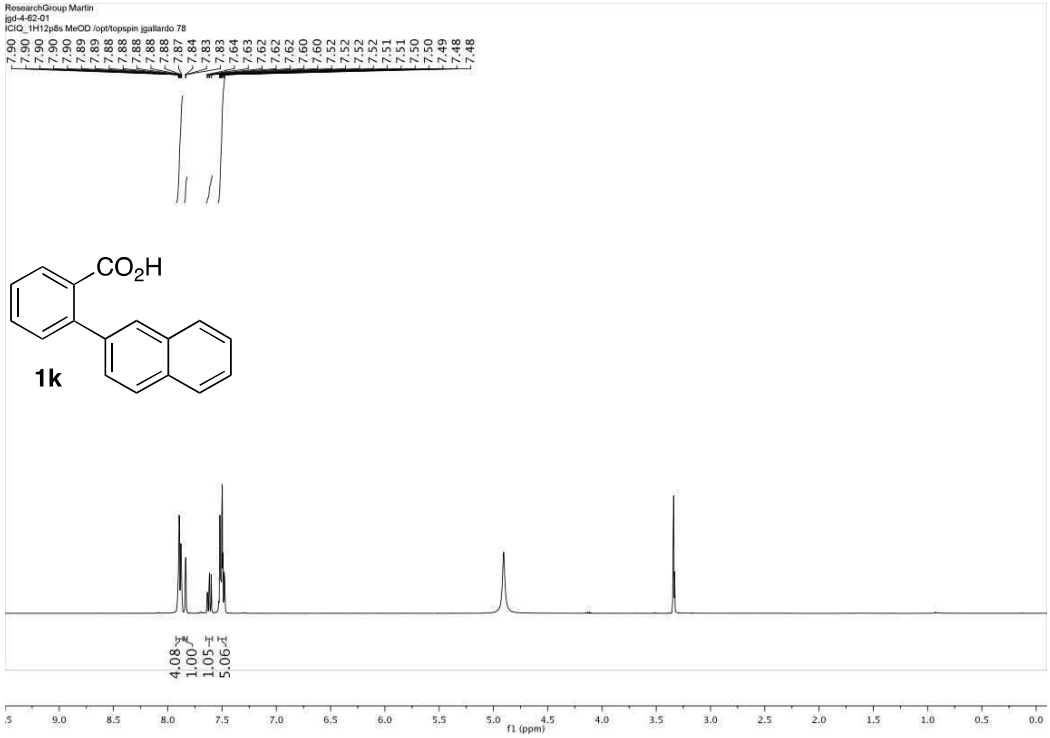

ResearchGroup Martin
jgd-4-29-01
ICIQ_13C{1H}512s MeOD /opt/topspin jgallardo 101
173.53
144.49
143.39
139.47
134.12
132.81
132.52
131.12
130.55
128.78
128.21
22.24
CO2H
Me
Me
1h
f1 (ppm)

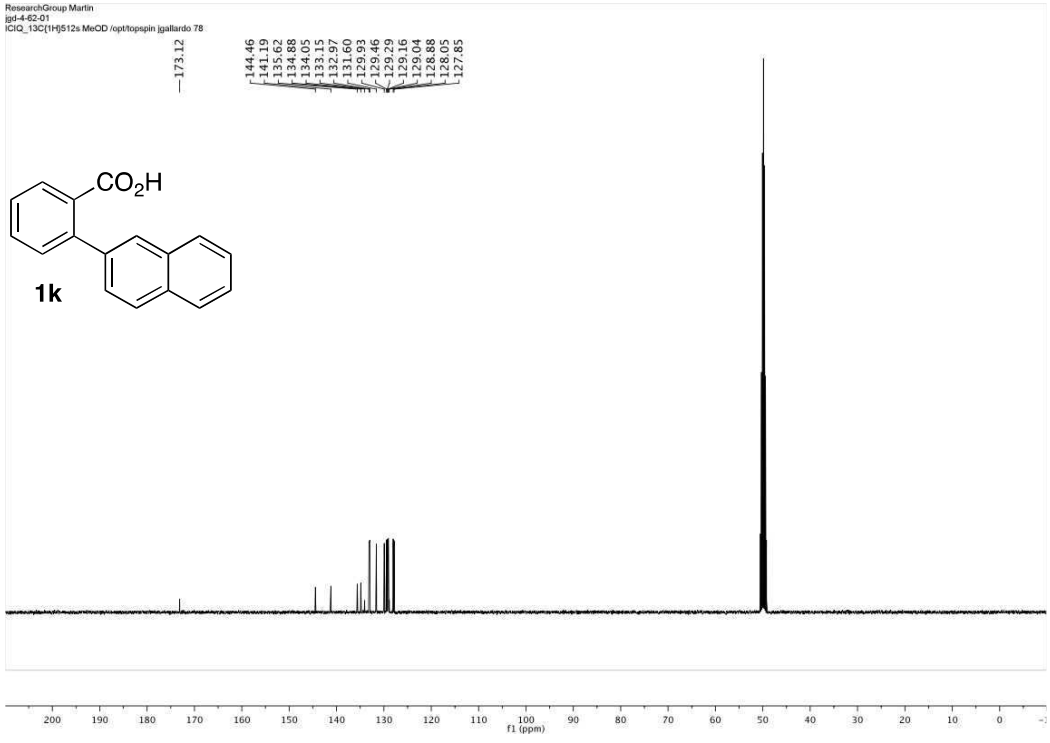

ResearchGroup Martin
jgd-4-52
ICIQ_1H12p8s MeOD /opt/topspin jgallardo 4
8.05
8.05
8.03
8.03
8.03
8.02
8.01
8.01
8.01
8.00
8.00
7.99
7.67
7.67
7.65
7.65
7.65
7.64
7.64
CO2H
O
1i
2.96
1.00
3.02
3.06
1.04
f1 (ppm)

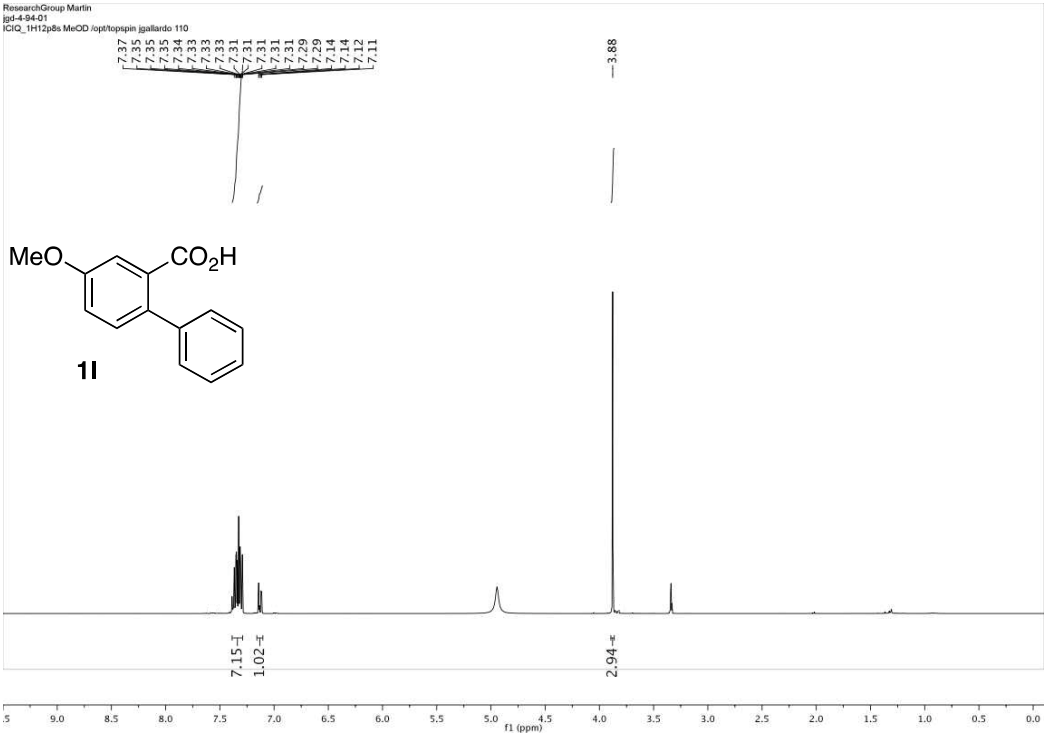

ResearchGroup Martin
jgd-4-52
ICIQ_13C{1H}512s MeOD /opt/topspin jgallardo 91
172.36
158.17
155.80
139.15
133.68
133.44
131.98
129.76
129.17
129.05
126.37
125.90
124.89
124.77
122.56
CO2H
O
1i
f1 (ppm)

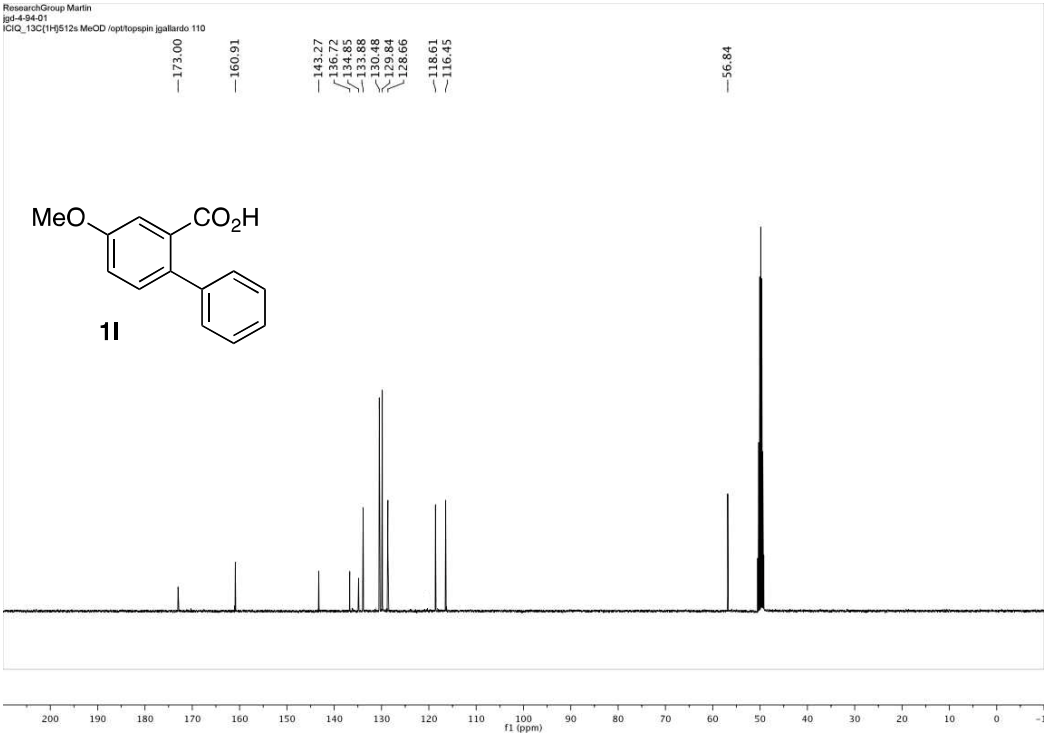

CO2H
1j
OMe
OMe
0.98
1.00
1.99
1.01
0.98
0.96
5.94
f1 (ppm)

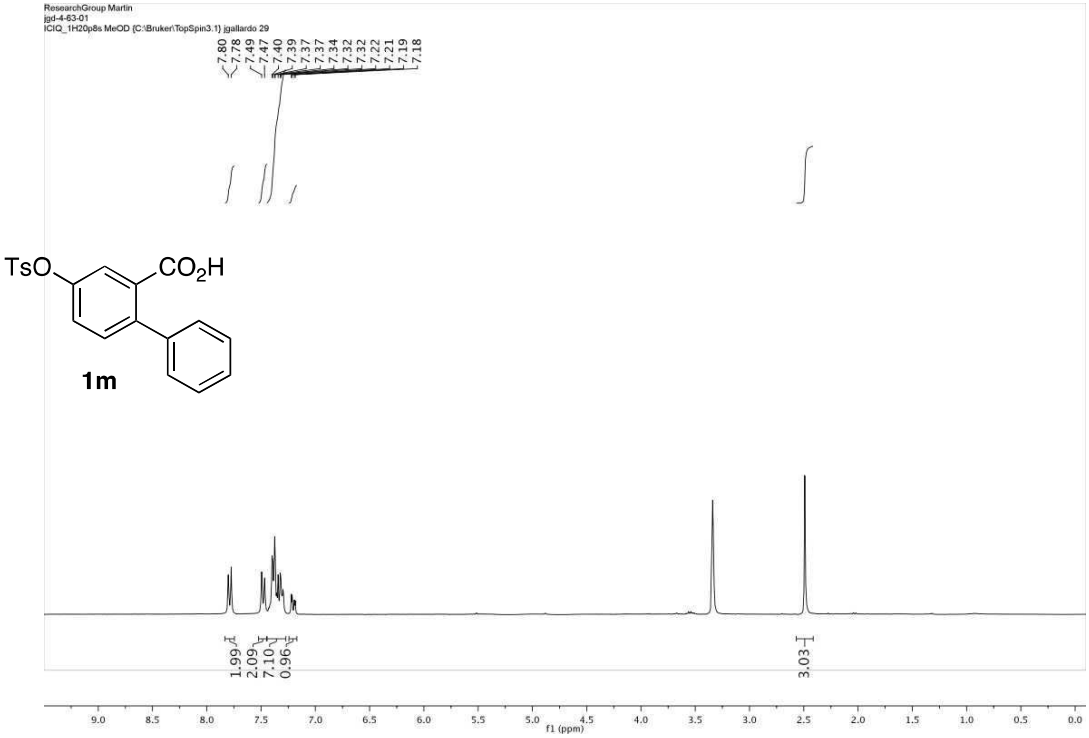

ResearchGroup Martin
171.05
149.23
149.18
141.32
134.18
131.45
131.17
129.61
113.19
112.51
56.45
56.37
CO2H
1j
OMe
OMe
f1 (ppm)

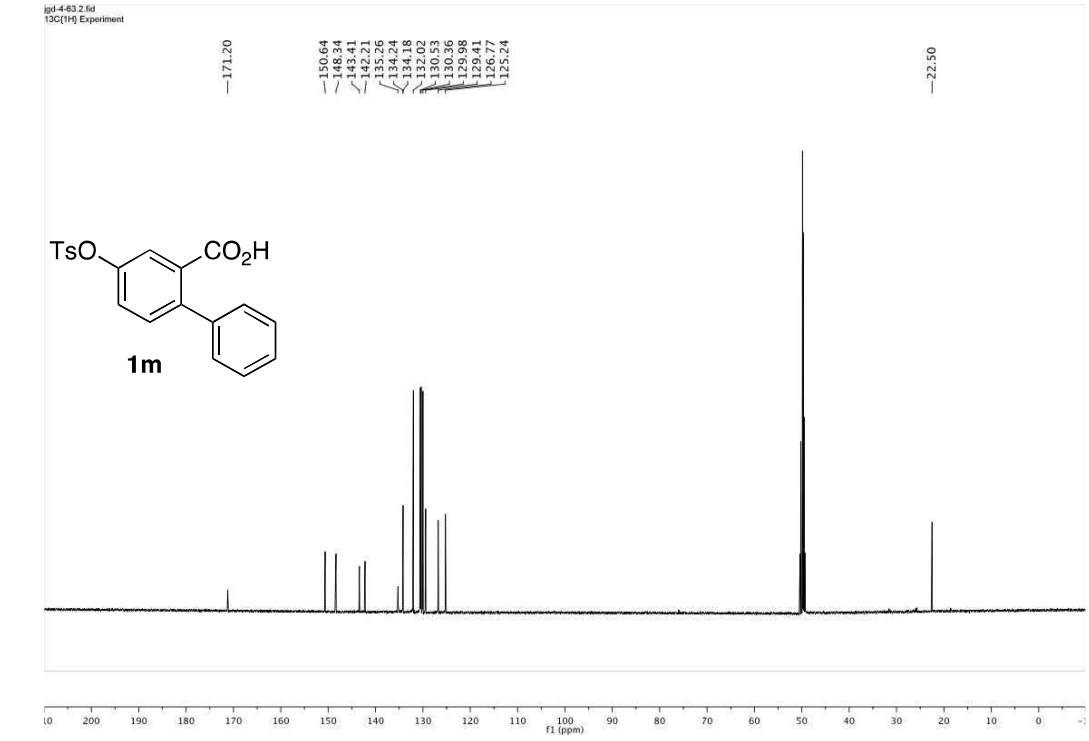

ResearchGroup Martin
jgd-4-62-01
ICIQ_1H12p8s MeOD /opt/topspin jgallardo 78
7.90 7.90 7.90 7.90 7.90 7.89 7.89 7.88 7.88 7.88 7.88 7.88 7.87 7.84 7.83 7.83 7.64 7.63 7.62 7.62 7.62 7.60 7.60 7.52 7.52 7.52 7.52 7.51 7.50 7.50 7.49 7.48 7.48
CO2H
1k
4.08
1.00
1.05
5.06
f1 (ppm)

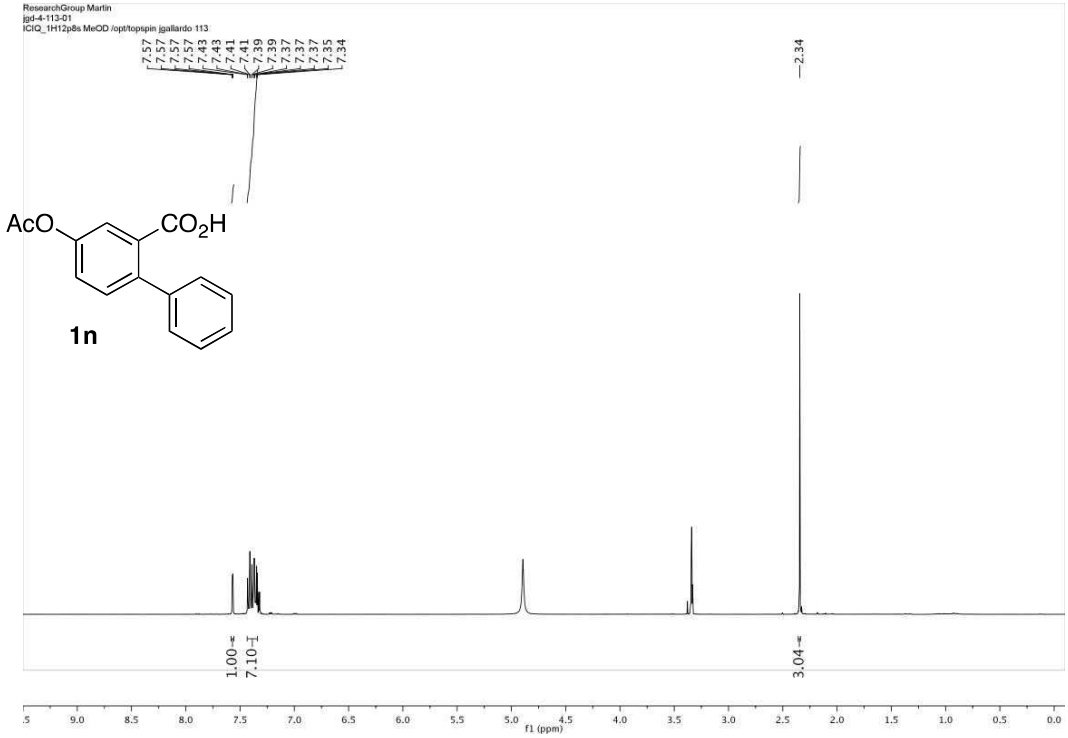

ResearchGroup Martin
jgd-4-62-01
ICIQ_13C{1H}512s MeOD /opt/topspin jgallardo 78
173.12
144.46 141.19 135.62 134.88 134.05 133.15 132.97 131.60 129.93 129.46 129.29 129.16 129.04 128.88 128.05 127.85
CO2H
1k
f1 (ppm)

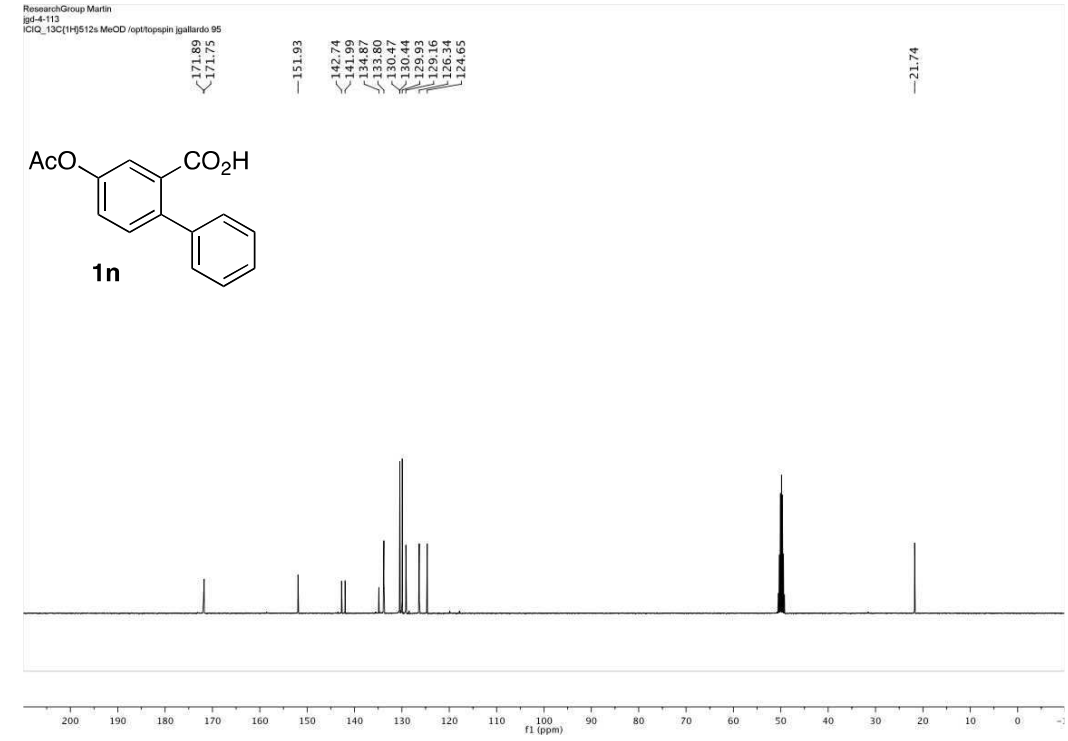

ResearchGroup Martin
jgd-4-94-01
ICIQ_1H12p8s MeOD /opt/topspin jgallardo 110
7.37
7.35
7.35
7.35
7.34
7.33
7.33
7.31
7.31
7.31
7.31
7.31
7.29
7.14
7.12
7.11
3.88
MeO
CO2H
1l
7.15
1.02
2.94
f1 (ppm)


ResearchGroup Martin
jgd-4-94-01
ICIQ_13C{1H}512s MeOD /opt/topspin jgallardo 110
173.00
160.91
143.27
136.72
134.85
133.88
130.48
129.84
128.66
118.61
116.45
56.84
MeO
CO2H
1l
f1 (ppm)

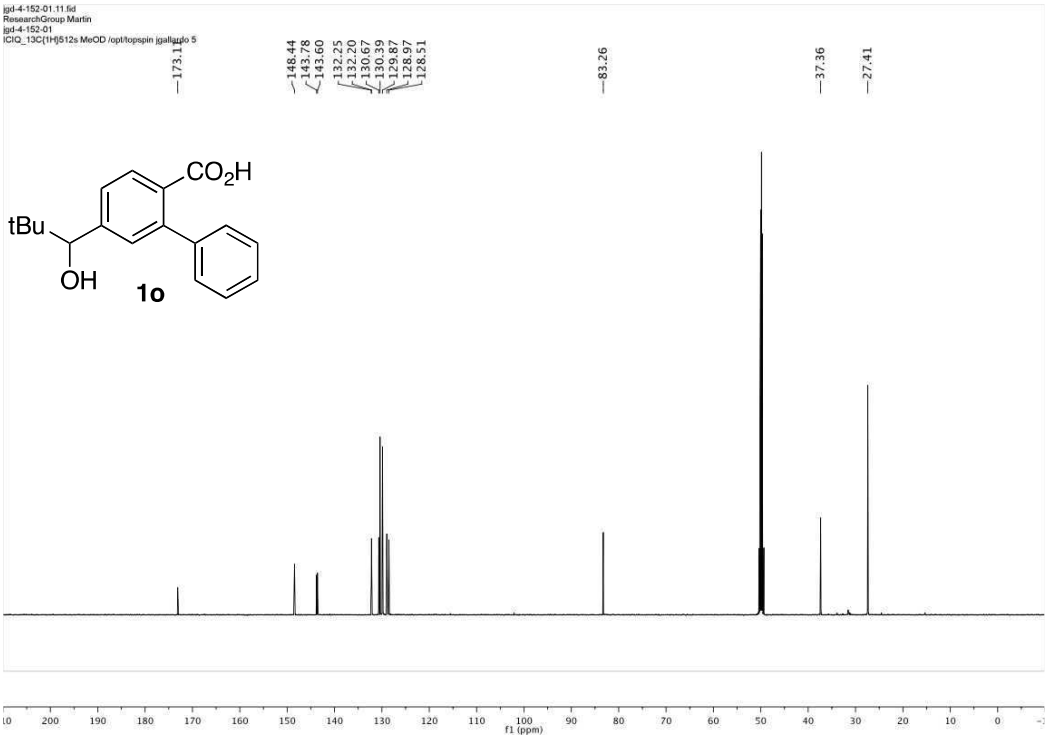

ResearchGroup Martin
jgd-4-63-01
ICIQ_1H20p8s MeOD (C:\Bruker\TopSpin3.1) jgallardo 29
7.80
7.78
7.49
7.47
7.40
7.39
7.37
7.37
7.34
7.32
7.22
7.21
7.19
7.18
TsO
CO2H
1m
1.99
2.09
7.10
0.96
3.03
f1 (ppm)

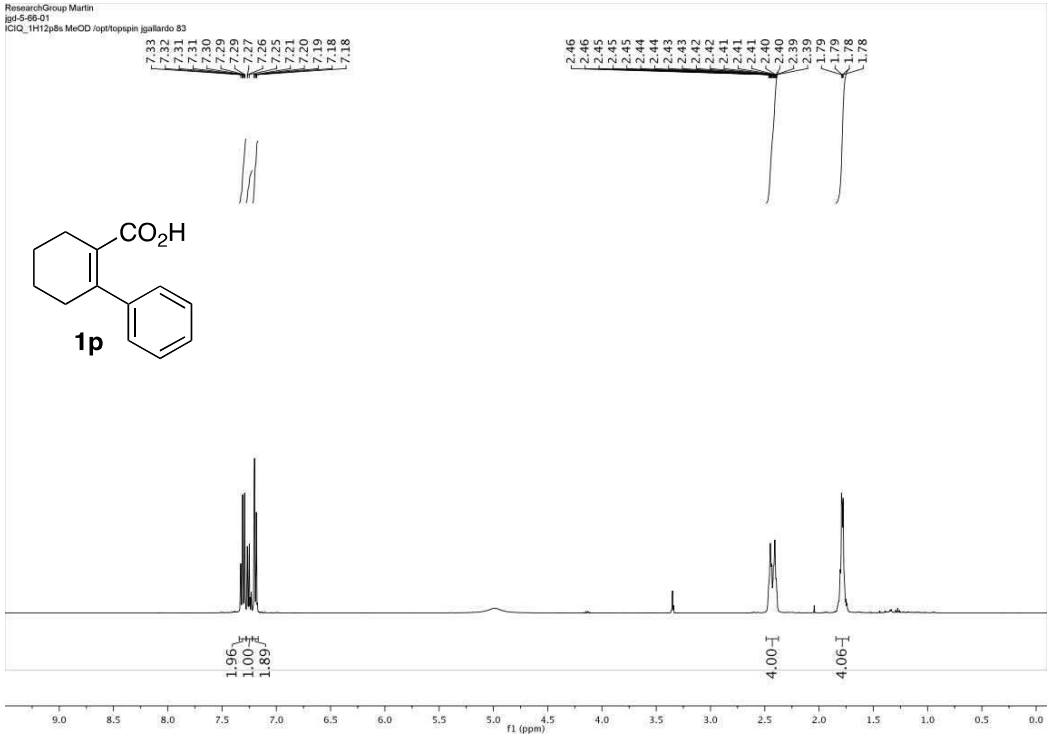

jgd-4-63.2.fid
13C{1H} Experiment
171.20
150.64
148.34
143.41
142.21
133.26
134.24
134.18
132.02
130.53
130.36
129.98
129.41
126.77
125.24
22.50
TsO
CO2H
1m
f1 (ppm)

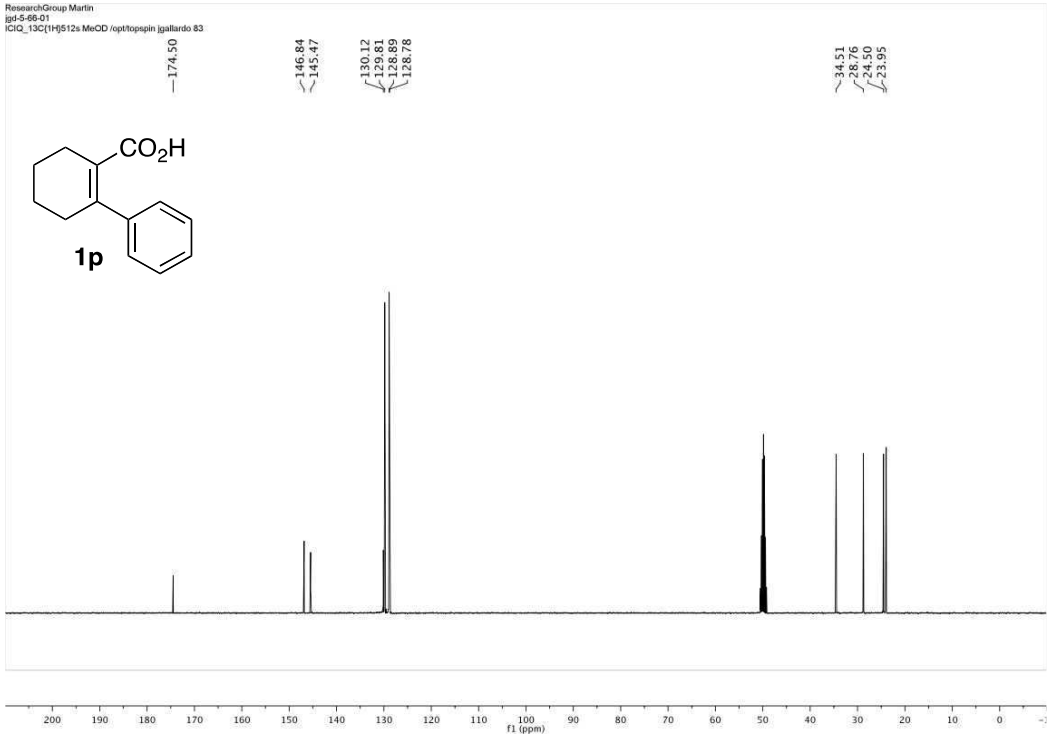

ResearchGroup Martin
jgd-4-113-01
ICIQ_1H12p8s MeOD /opt/topspin jgallardo 113
7.57
7.57
7.57
7.57
7.43
7.41
7.41
7.39
7.39
7.37
7.37
7.37
7.35
7.34
2.34
AcO
CO2H
1n
1.00
7.10
3.04
f1 (ppm)

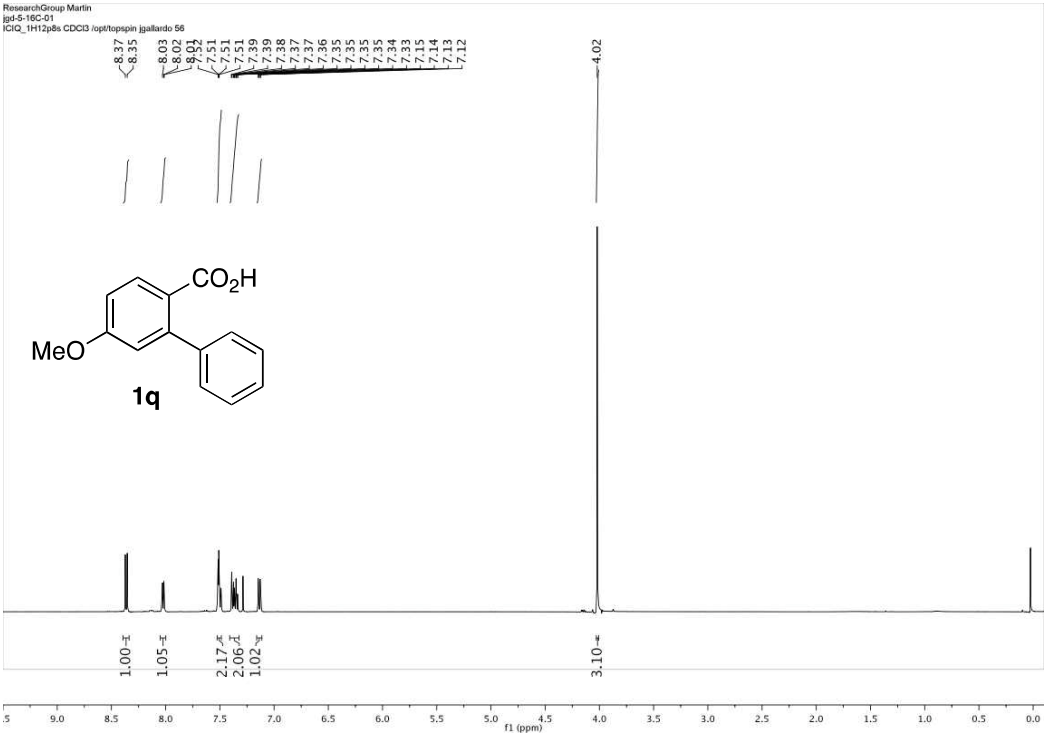

ResearchGroup Martin
jgd-4-113
ICIQ_13C{1H}512s MeOD /opt/topspin jgallardo 95
171.89
171.75
151.93
142.74
141.99
134.87
133.80
130.47
130.44
129.93
129.16
126.34
124.65
21.74
AcO
CO2H
1n
f1 (ppm)

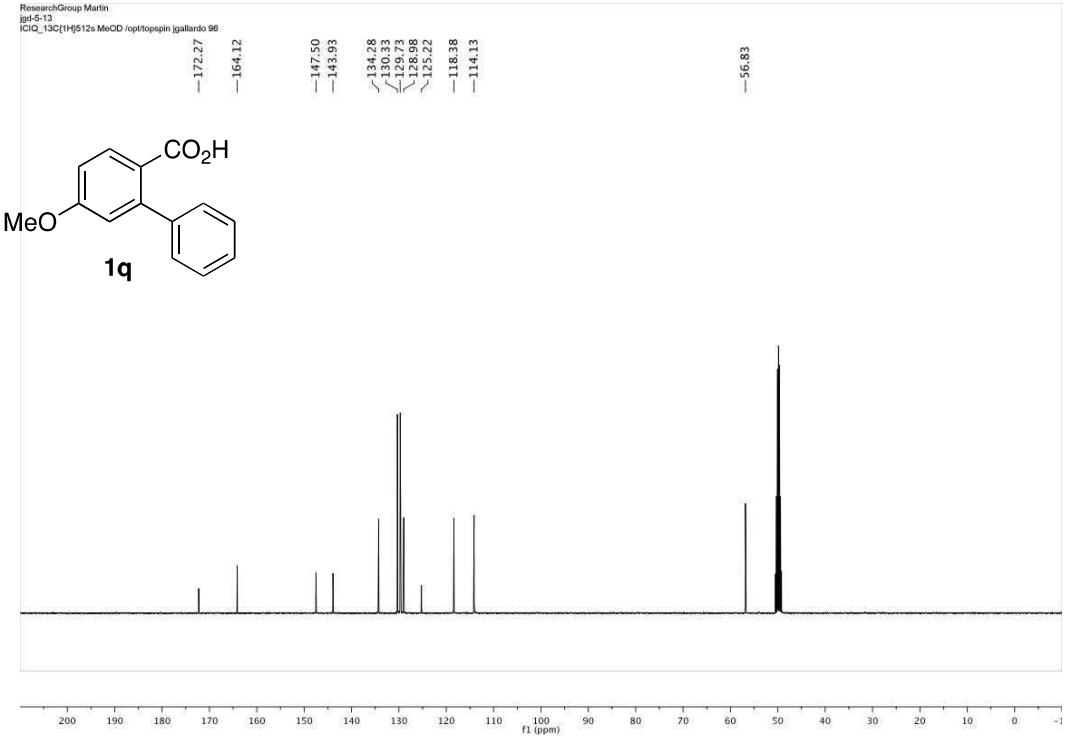

ResearchGroup Martin
jgd-4-152-01
ICIQ_1H12p8s MeOD /opt/topspin jgallardo 5
7.80
7.79
7.42
7.42
7.42
7.40
7.40
7.40
7.40
7.39
7.39
7.36
7.36
7.35
7.35
7.35
7.35
4.99
0.97
CO2H
tBu
OH
1o
1.00
3.04
3.95
1.02
8.93
f1 (ppm)

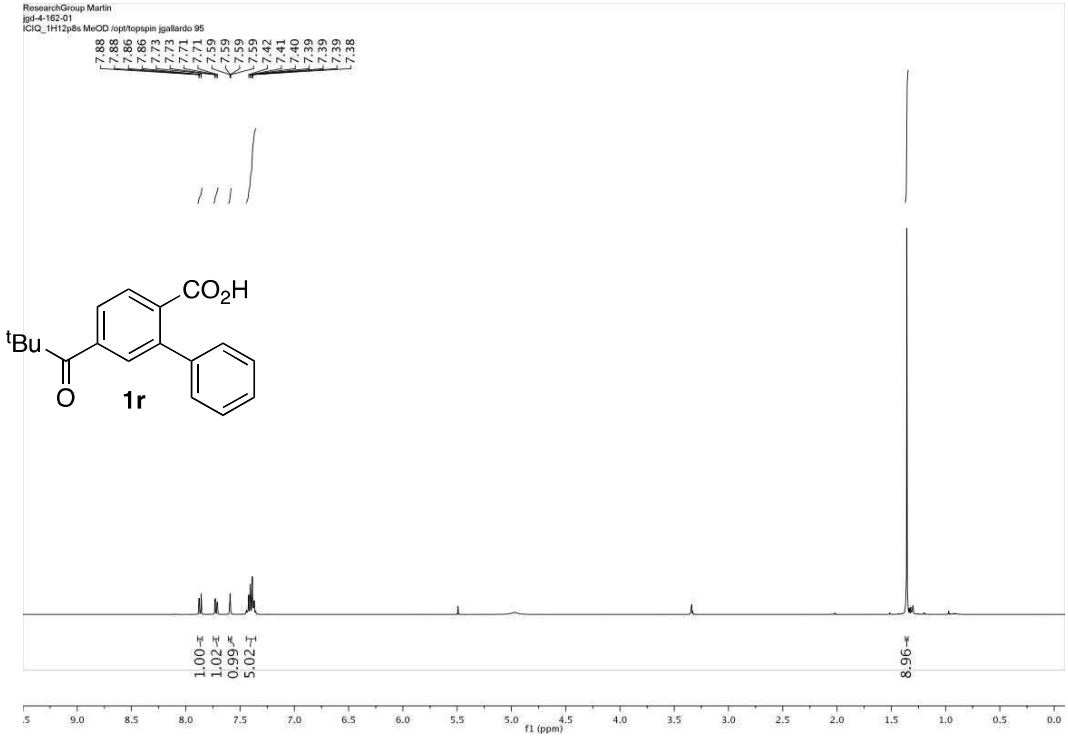

jgd-4-152-01.11.fid
ResearchGroup Martin
jgd-4-152-01
ICIQ_13C{1H}512s MeOD /opt/topspin jgallardo 5
173.11
148.44
143.78
143.60
132.25
132.20
130.67
130.39
129.87
128.97
128.51
83.26
37.36
27.41
CO2H
tBu
OH
1o
f1 (ppm)

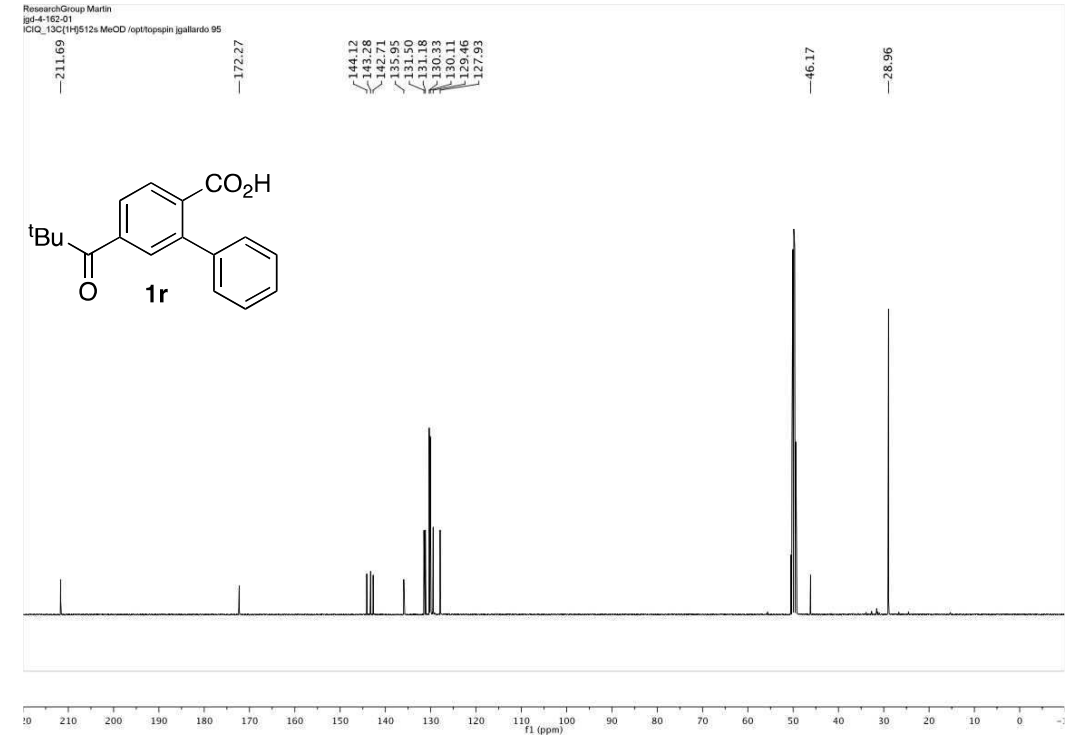

ResearchGroup Martin
jgd-5-66-01
ICIQ_1H12p8s MeOD /opt/topspin jgallardo 83
7.33
7.32
7.31
7.31
7.30
7.29
7.29
7.27
7.26
7.25
7.21
7.20
7.19
7.18
7.18
2.46
2.46
2.45
2.45
2.45
2.44
2.44
2.43
2.43
2.42
2.42
2.41
2.41
2.40
2.40
2.39
2.39
1.79
1.79
1.78
1.78
CO2H
1p
1.96
1.00
1.89
4.00
4.06
f1 (ppm)

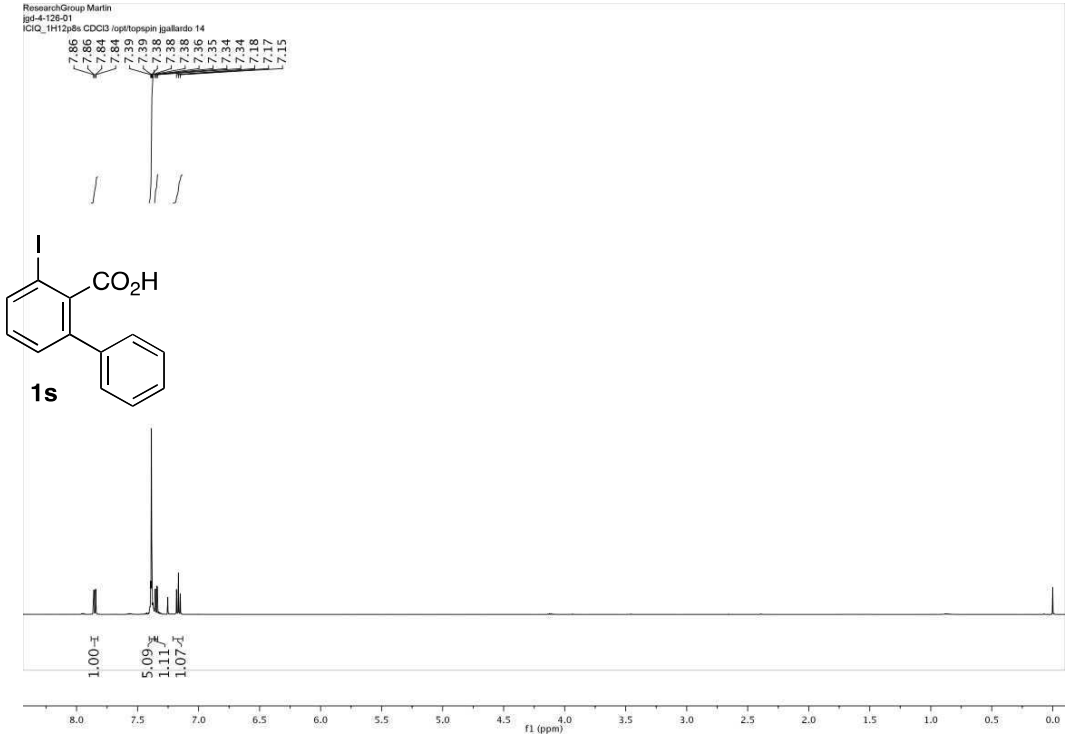

ResearchGroup Martin
jgd-5-66-01
ICIQ_13C{1H}512s MeOD /opt/topspin jgallardo 83
174.50
146.84
145.47
130.12
129.81
128.89
128.78
34.51
28.76
24.50
23.95
CO2H
1p
f1 (ppm)

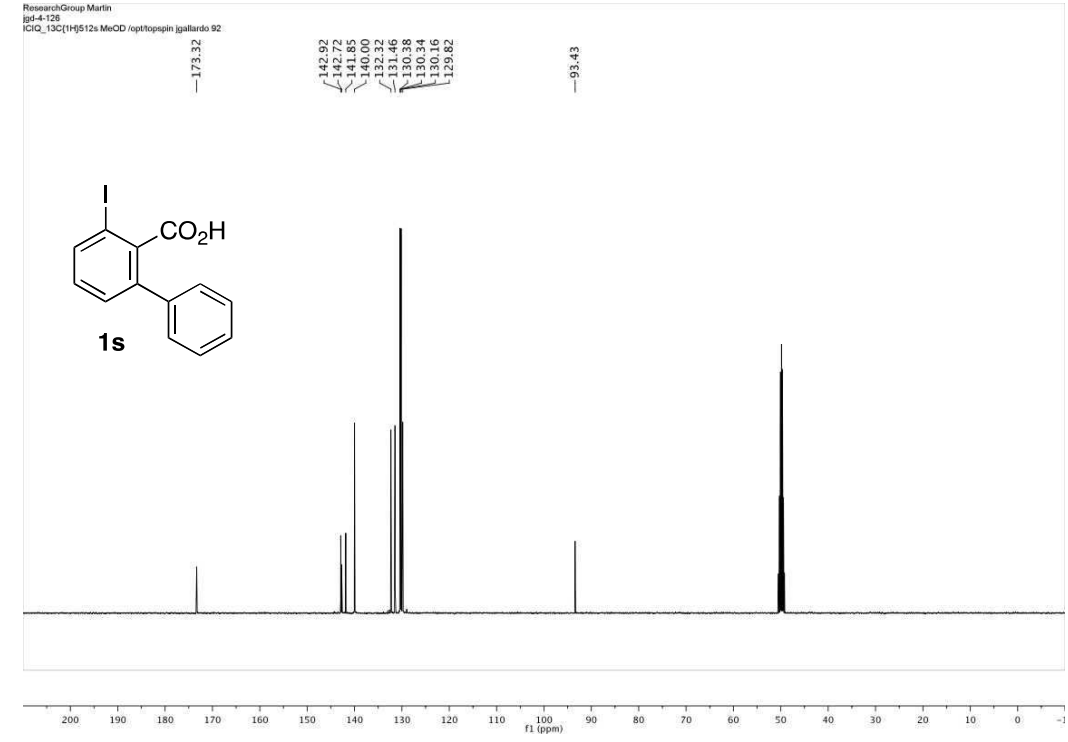

ResearchGroup Martin
jgd-5-16C-01
ICIQ_1H12p8s CDCl3 /opt/topspin jgallardo 56
8.37
8.35
8.03
8.02
8.01
7.52
7.51
7.51
7.51
7.39
7.39
7.38
7.37
7.36
7.35
7.35
7.35
7.34
7.33
7.15
7.14
7.13
7.12
4.02
CO2H
MeO
1q
1.00
1.05
2.17
2.06
1.02
3.10
f1 (ppm)

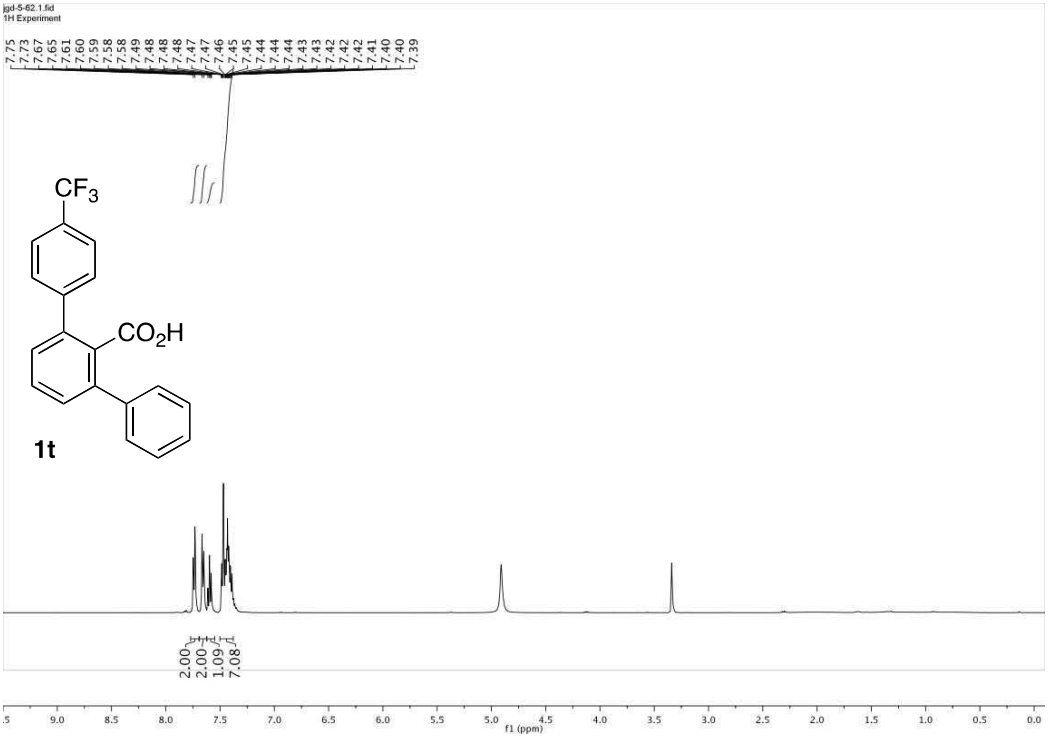

ResearchGroup Martin
jgd-5-13
ICIQ_13C{1H}512s MeOD /opt/topspin jgallardo 96
172.27
164.12
147.50
143.93
134.28
130.33
129.73
128.98
125.22
118.38
114.13
56.83
CO2H
MeO
1q
f1 (ppm)

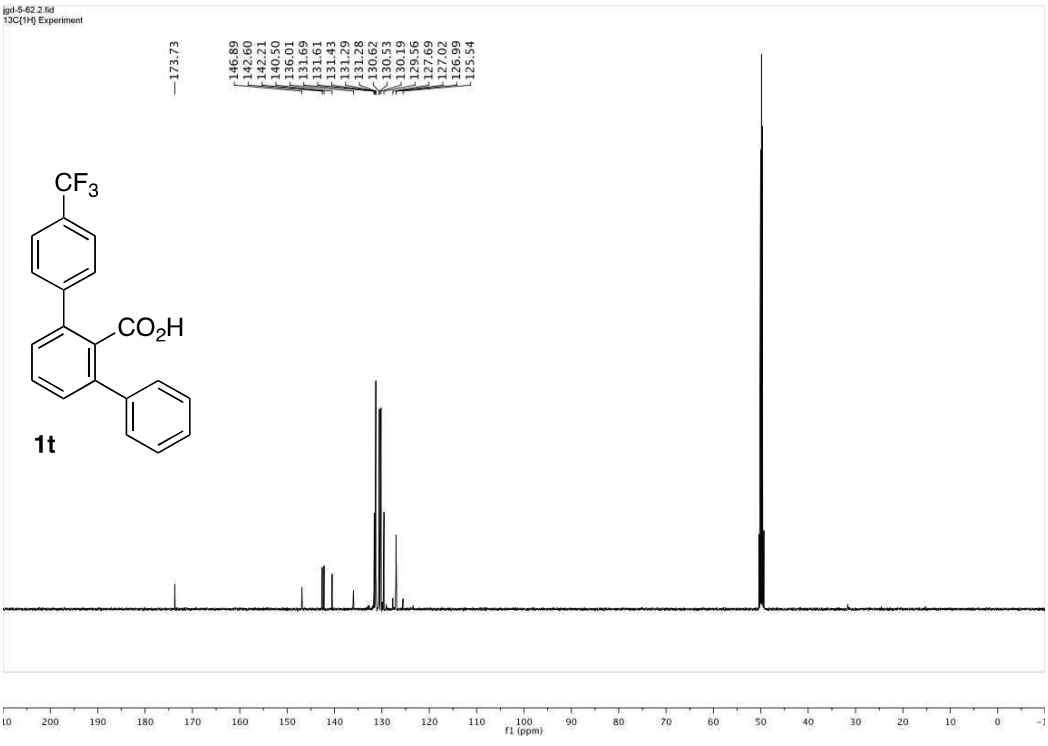

ResearchGroup Martin
jgd-4-162-01
ICIQ_1H12p8s MeOD /opt/topspin jgallardo 95
7.88
7.88
7.86
7.86
7.73
7.73
7.71
7.71
7.59
7.59
7.59
7.59
7.42
7.41
7.40
7.39
7.39
7.38
CO2H
tBu
O
1r
1.00
1.02
0.99
5.02
8.96
f1 (ppm)

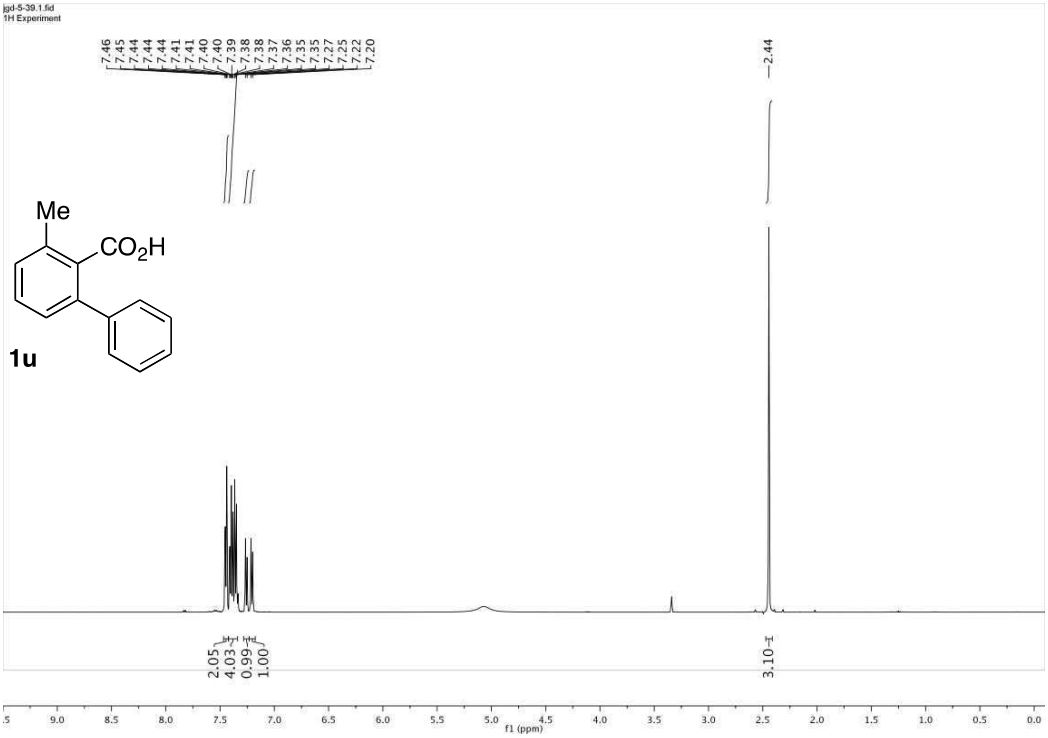

ResearchGroup Martin
jgd-4-162-01
ICIQ_13C{1H}512s MeOD /opt/topspin jgallardo 95
211.69
172.27
144.12
143.28
142.71
135.95
131.50
131.18
130.33
130.11
129.46
127.93
46.17
28.96
CO2H
tBu
O
1r
f1 (ppm)

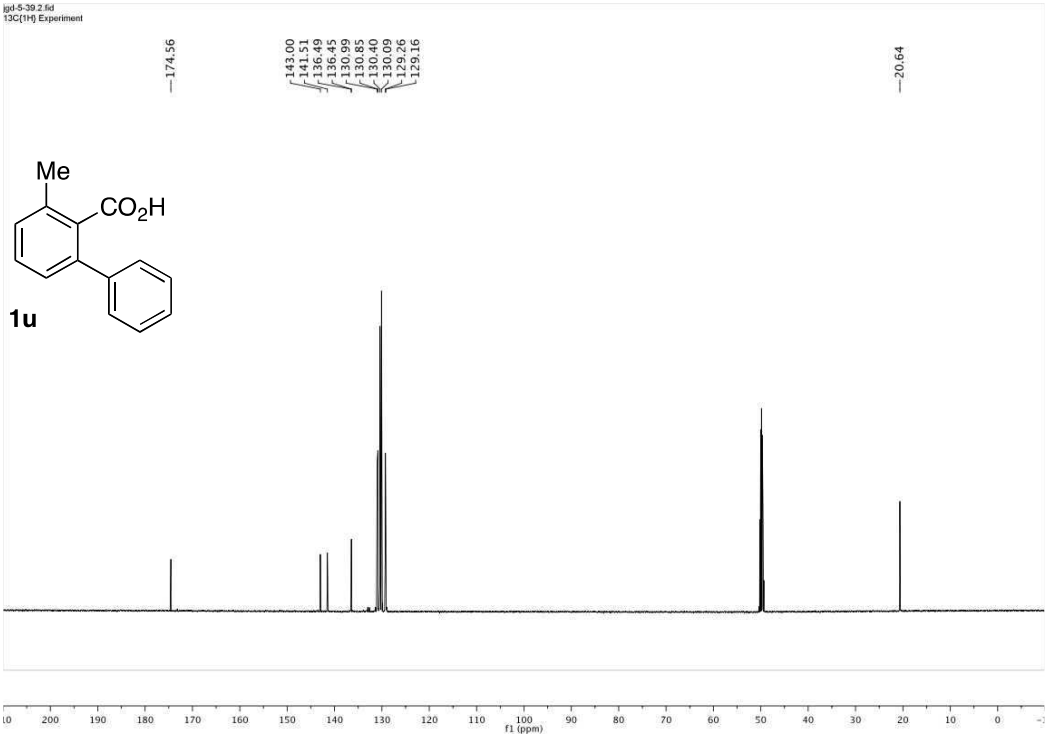

ResearchGroup Martin
jgd-4-126-01
ICIQ_1H12p8s CDCl3 /opt/topspin jgallardo 14
7.86
7.86
7.84
7.84
7.39
7.39
7.38
7.38
7.38
7.36
7.35
7.34
7.34
7.18
7.17
7.15
I
CO2H
1s
1.00
5.09
1.11
1.07
f1 (ppm)

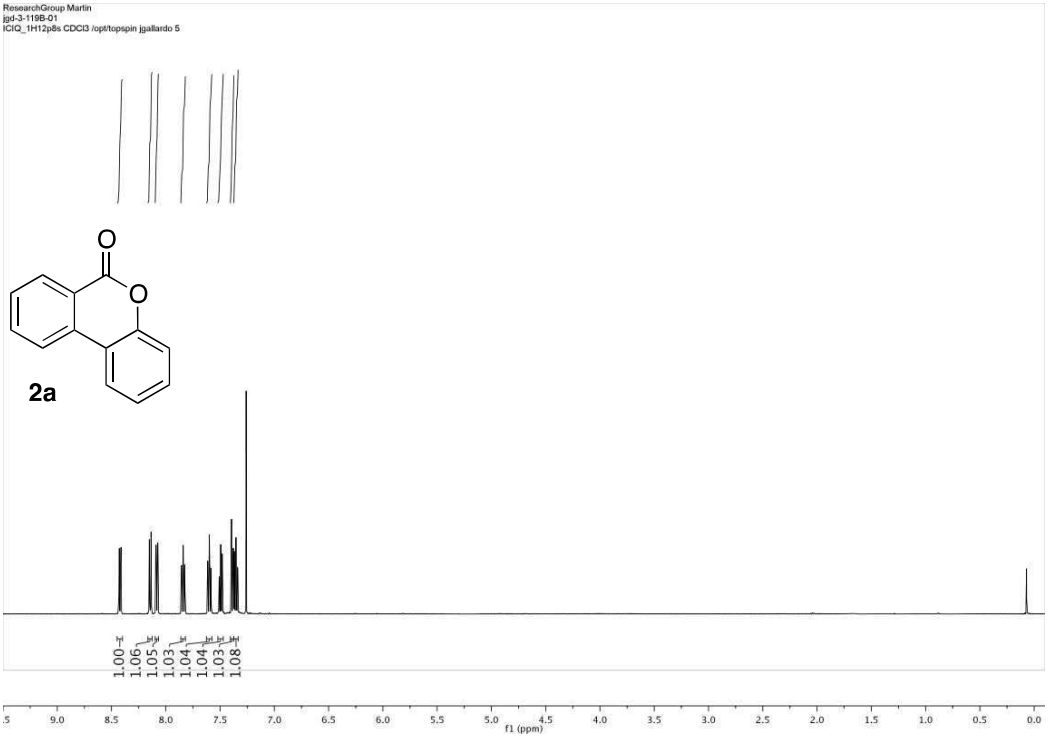

ResearchGroup Martin
jgd-4-126
ICIQ_13C{1H}512s MeOD /opt/topspin jgallardo 92
173.32
142.92
142.72
141.85
140.00
132.32
131.46
130.38
130.34
130.16
129.82
93.43
I
CO2H
1s
f1 (ppm)

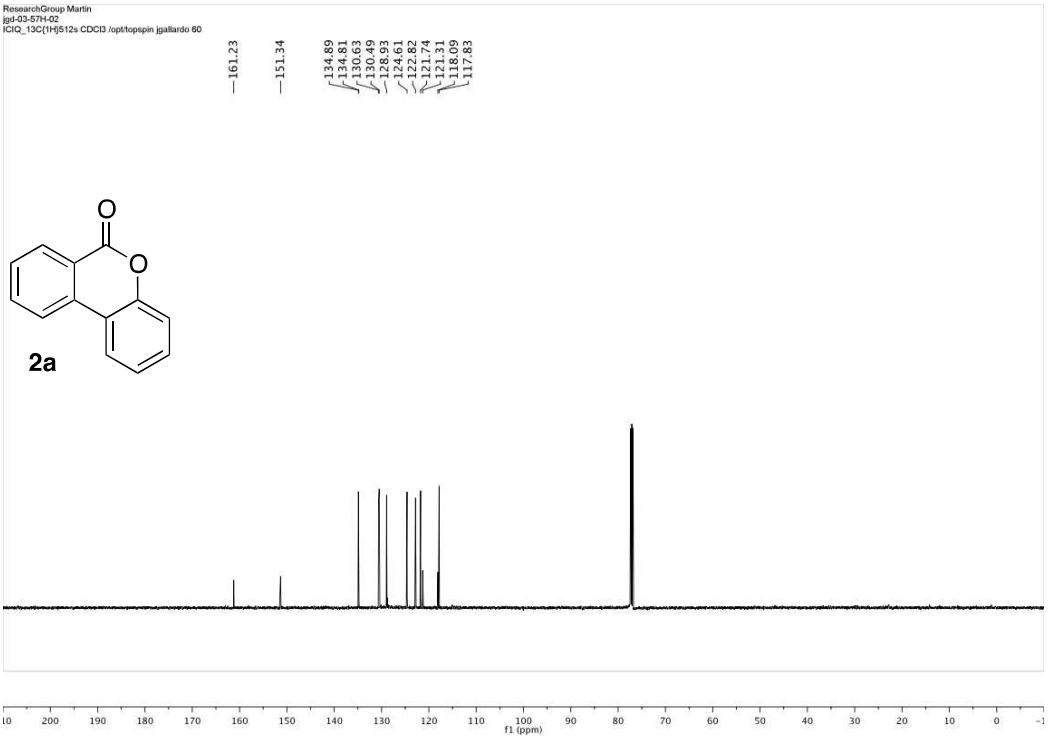

jgd-5-62.1.fid
1H Experiment
7.75
7.73
7.67
7.65
7.61
7.60
7.59
7.58
7.58
7.49
7.48
7.48
7.48
7.47
7.47
7.46
7.45
7.45
7.44
7.44
7.44
7.43
7.43
7.42
7.42
7.42
7.41
7.40
7.40
7.39
CF3
CO2H
1t
2.00
2.00
1.09
7.08
f1 (ppm)

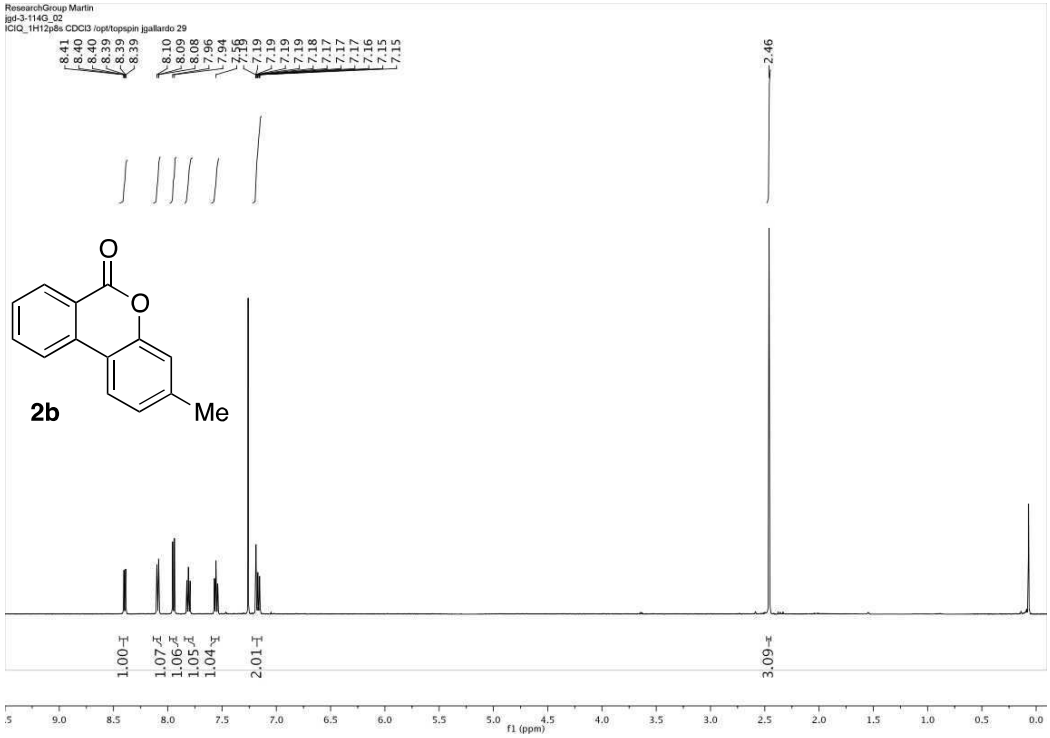

jgd-5-62.2.fid
13C{1H} Experiment
173.73
146.89
142.60
142.21
140.50
136.01
131.69
131.61
131.43
131.29
131.28
130.62
130.53
130.19
129.56
127.69
127.02
126.99
125.54
CF3
CO2H
1t
f1 (ppm)

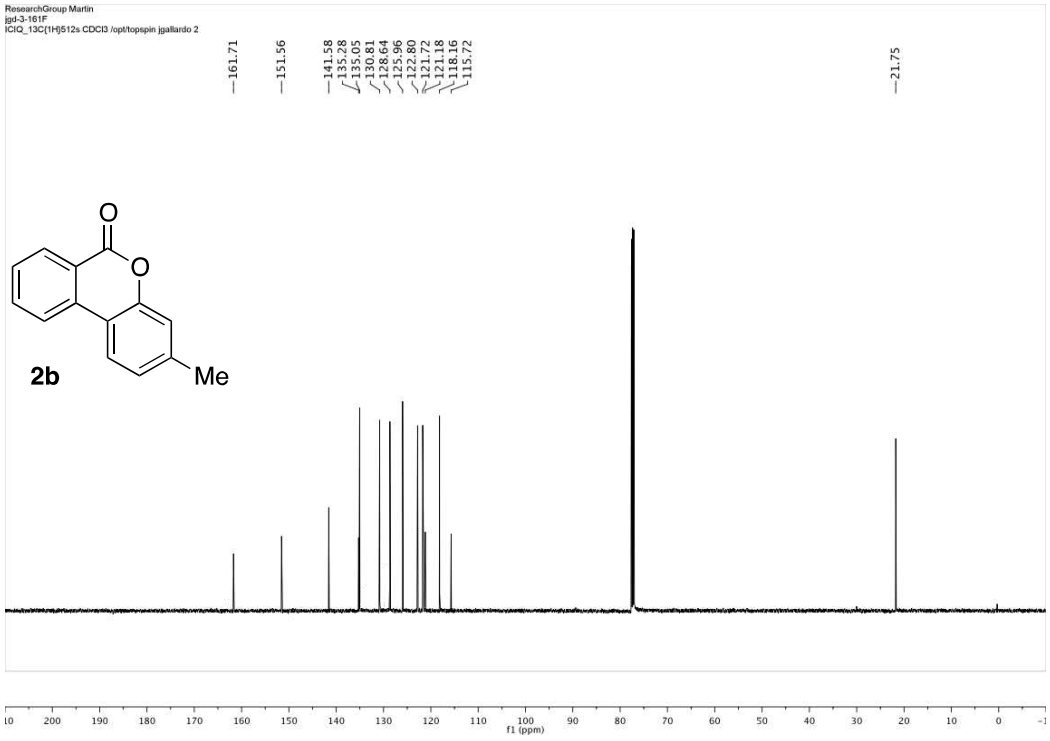

jgd-5-39.1.fid
1H Experiment
7.46
7.45
7.44
7.44
7.44
7.41
7.41
7.40
7.40
7.39
7.38
7.38
7.37
7.36
7.35
7.35
7.27
7.25
7.22
7.20
2.44
Me
CO2H
1u
2.05
4.03
0.99
1.00
3.10
f1 (ppm)

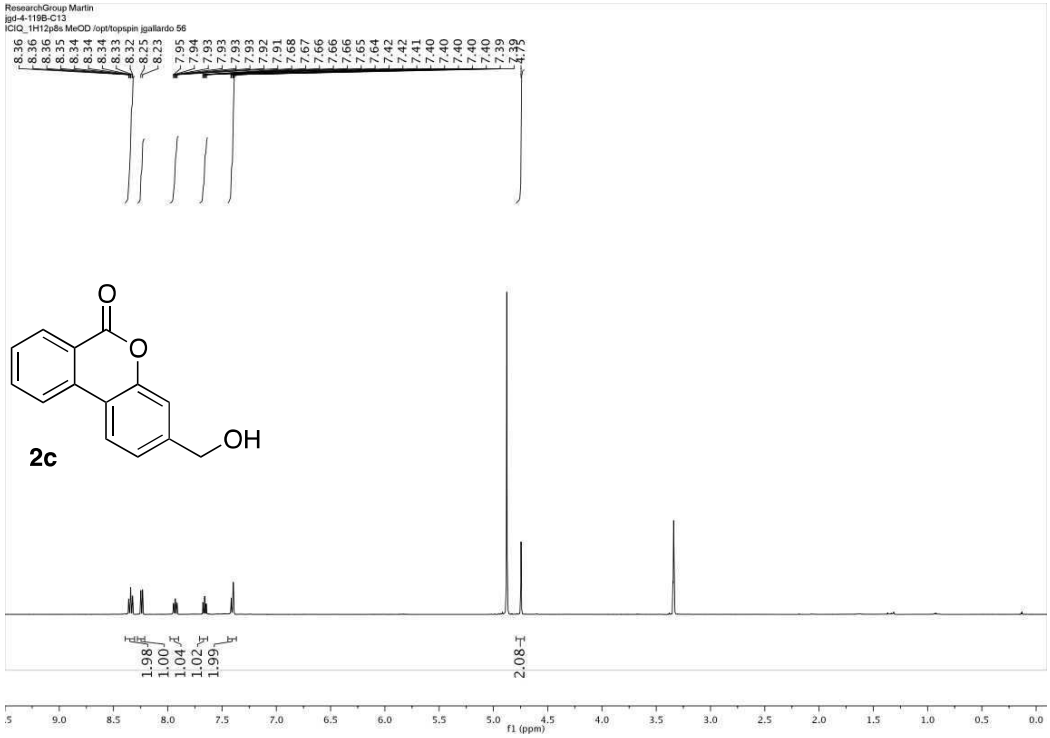

jgd-5-39.2.fid
13C{1H} Experiment
174.56
143.00
141.51
136.49
136.45
130.99
130.85
130.40
130.09
129.26
129.16
20.64
Me
CO2H
1u
f1 (ppm)

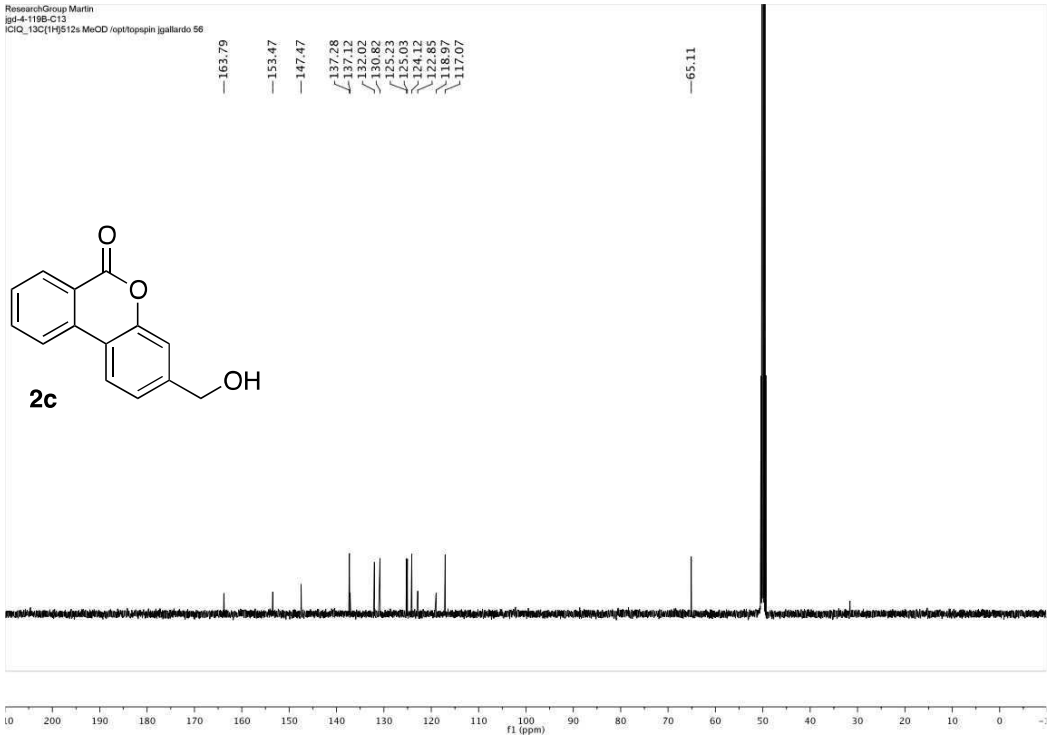

ResearchGroup Martin
jgd-3-119B-01
ICIQ_1H12p8s CDCl3 /opt/topspin jgallardo 5
2a
1.00
1.06
1.05
1.03
1.04
1.04
1.03
1.08
f1 (ppm)

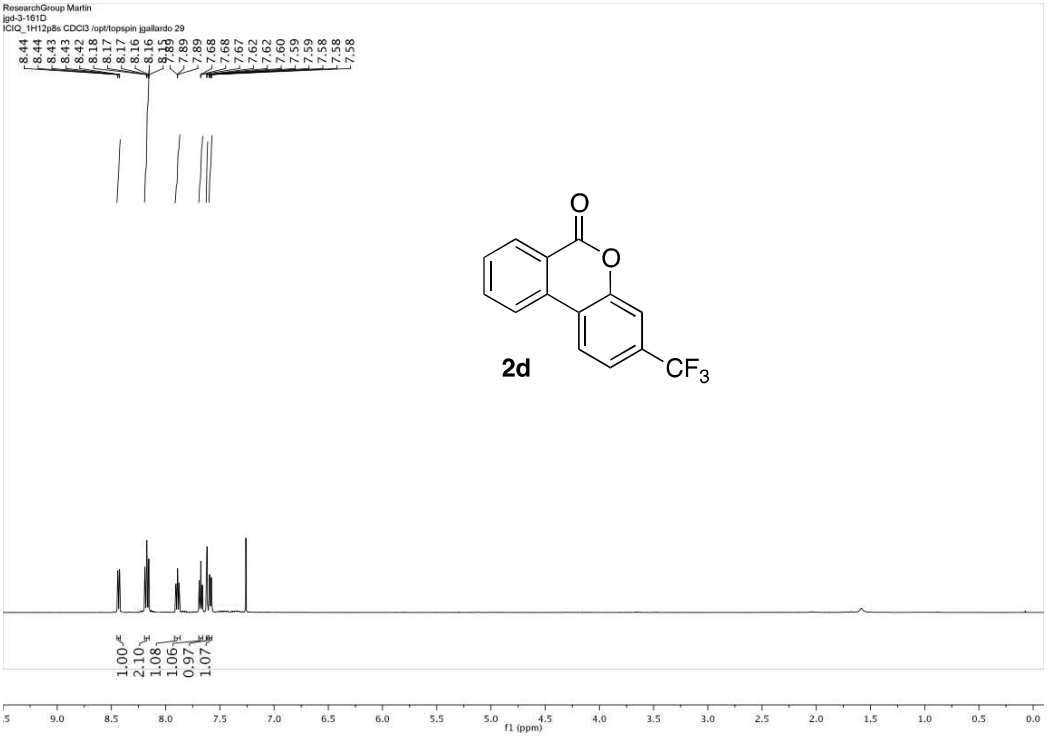

ResearchGroup Martin
jgd-02-57H-02
ICIQ_13C{1H}512s CDCl3 /opt/topspin jgallardo 60
161.23
151.34
134.89
134.81
130.63
130.49
128.93
124.61
122.82
121.74
121.31
118.09
117.83
2a
f1 (ppm)

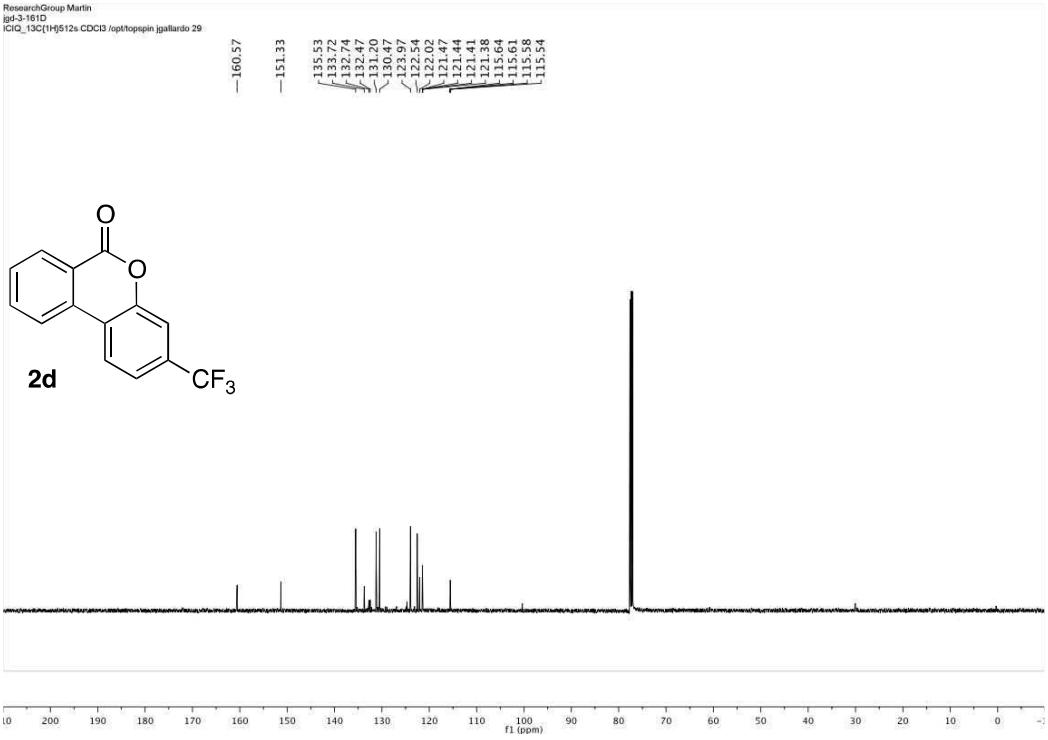

ResearchGroup Martin
jgd-3-114Q_02
ICIQ_1H12p8s CDCl3 /opt/topspin jgallardo 29
8.41
8.40
8.40
8.39
8.39
8.39
8.10
8.09
8.08
7.96
7.94
7.56
7.19
7.19
7.19
7.19
7.18
7.17
7.17
7.17
7.16
7.15
7.15
2.46
O
O
2b
Me
1.00
1.07
1.06
1.05
1.04
2.01
3.09
f1 (ppm)

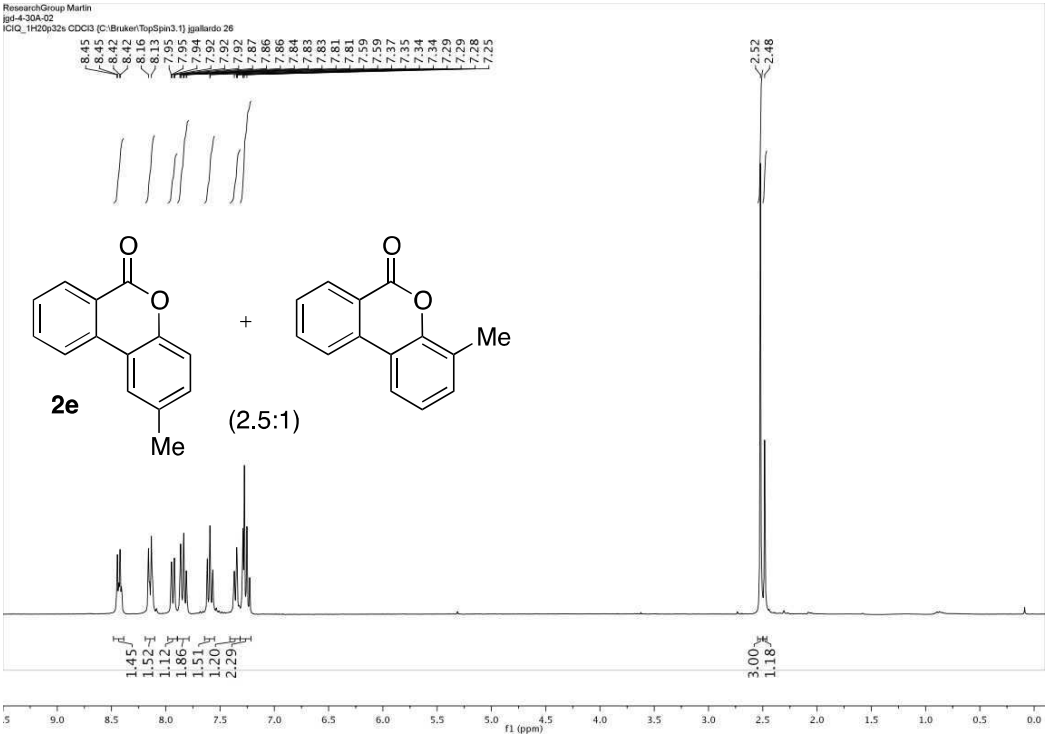

ResearchGroup Martin
jgd-3-161F
ICIQ_13C{1H}512s CDCl3 /opt/topspin jgallardo 2
161.71
151.56
141.58
135.28
135.05
130.81
128.64
125.96
122.80
121.72
121.18
118.16
115.72
21.75
O
O
2b
Me
f1 (ppm)

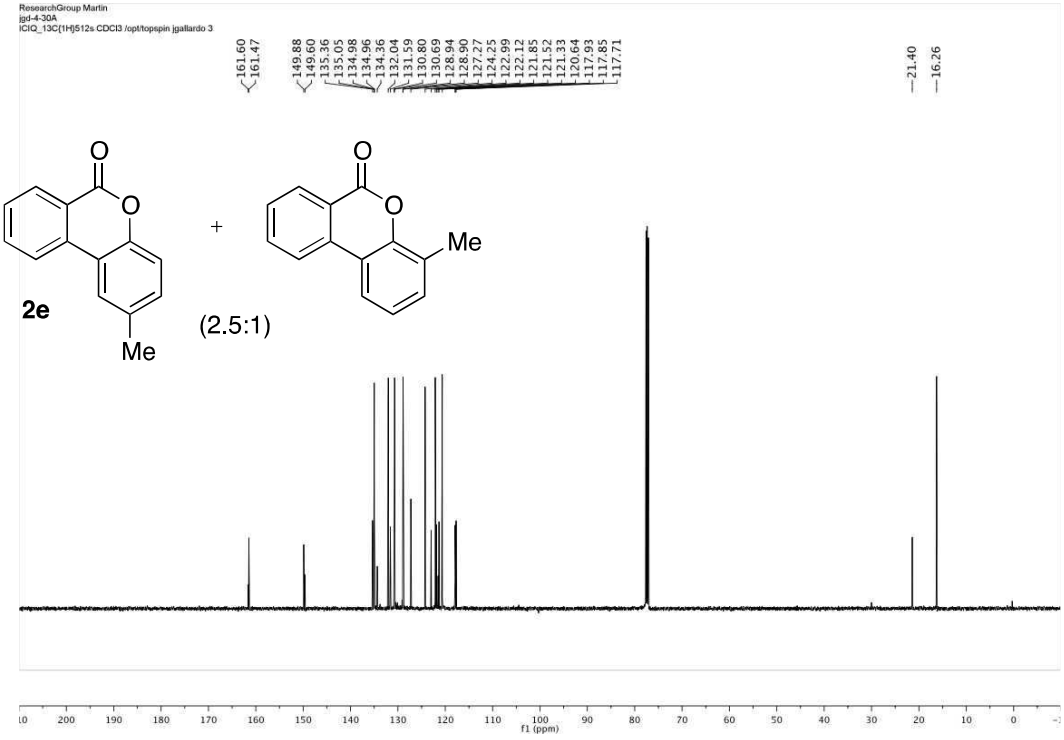

ResearchGroup Martin
jgd-4-119B-C13
ICIQ_1H12p8s MeOD /opt/topspin jgallardo 56
8.36
8.36
8.36
8.35
8.34
8.34
8.34
8.33
8.32
8.15
8.23
7.95
7.94
7.93
7.93
7.93
7.92
7.91
7.68
7.67
7.66
7.66
7.66
7.65
7.64
7.42
7.42
7.41
7.40
7.40
7.40
7.40
7.39
7.39
7.39
O
O
OH
2c
1.98
1.00
1.04
1.02
1.99
2.08
f1 (ppm)

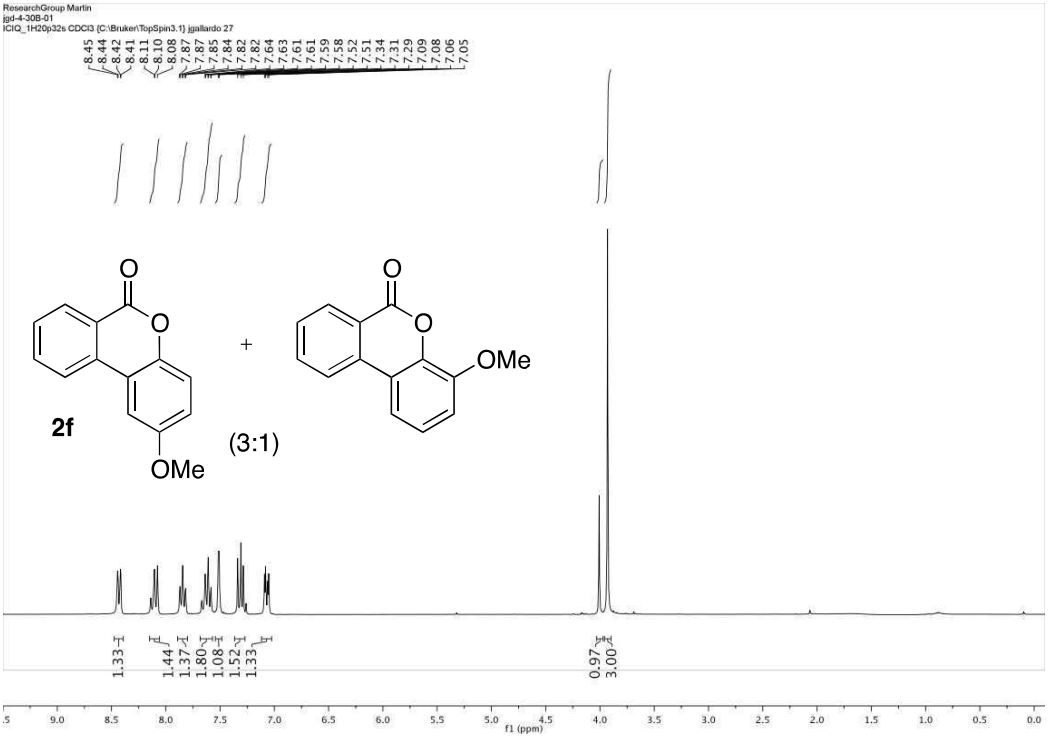

ResearchGroup Martin
jgd-4-119B-C13
ICIQ_13C{1H}512s MeOD /opt/topspin jgallardo 56
163.79
153.47
147.47
137.28
137.12
132.02
130.82
125.23
125.03
124.12
122.85
118.97
117.07
65.11
O
O
OH
2c
f1 (ppm)

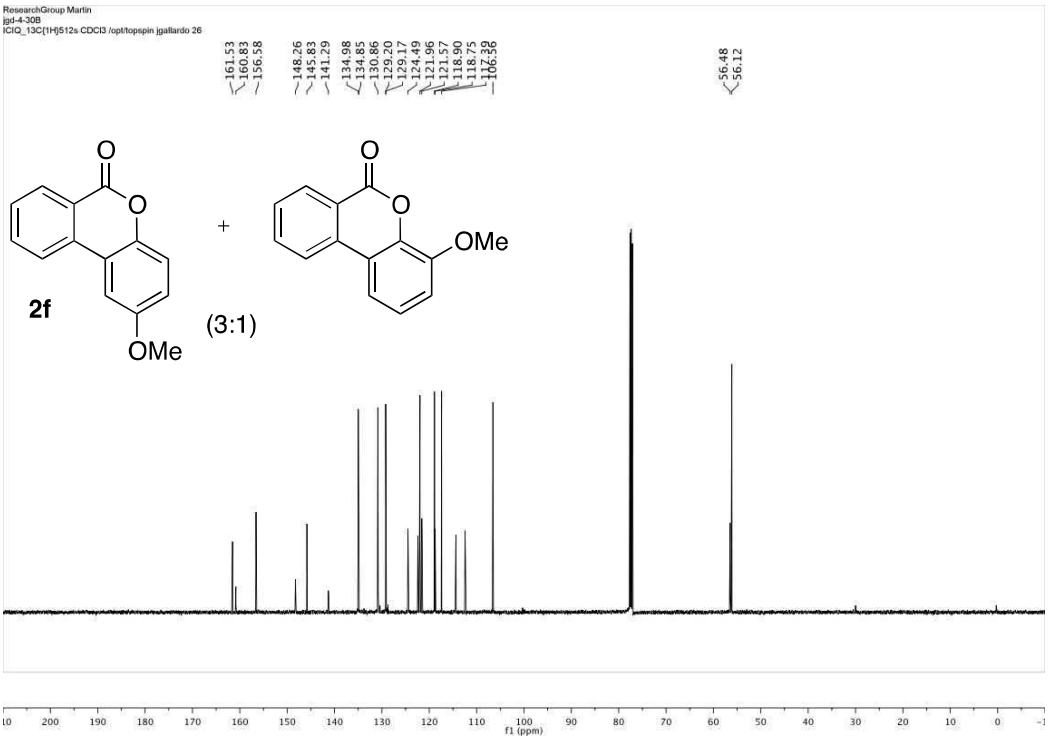

ResearchGroup Martin
jgd-3-161D
ICIQ_1H12p8s CDCl3 /opt/topspin jgallardo 29
8.44
8.44
8.43
8.43
8.42
8.18
8.17
8.17
8.16
8.16
7.89
7.89
7.68
7.68
7.67
7.62
7.62
7.60
7.59
7.58
7.58
7.58
O
O
2d
CF3
1.00
2.10
1.08
1.06
0.97
1.07
f1 (ppm)

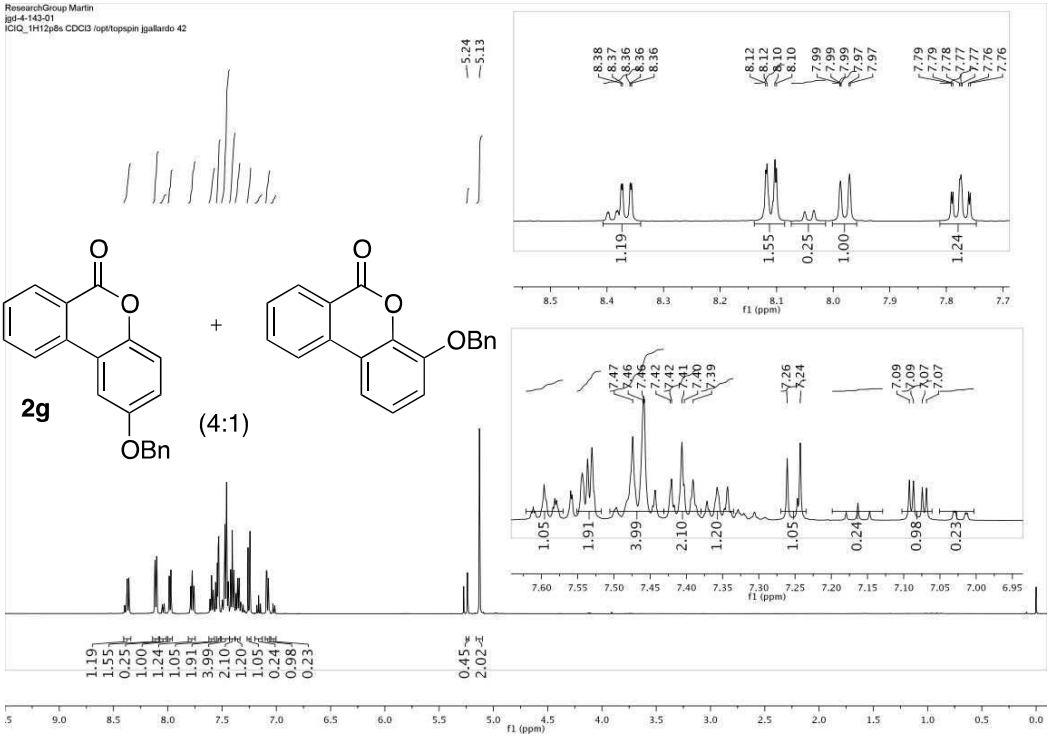

ResearchGroup Martin
jgd-3-161D
ICIQ_13C{1H}512s CDCl3 /opt/topspin jgallardo 29
160.57
151.33
135.53
133.72
132.74
132.47
131.20
130.47
123.97
122.54
122.02
121.47
121.44
121.41
121.38
115.64
115.61
115.58
115.54
O
O
2d
CF3
f1 (ppm)

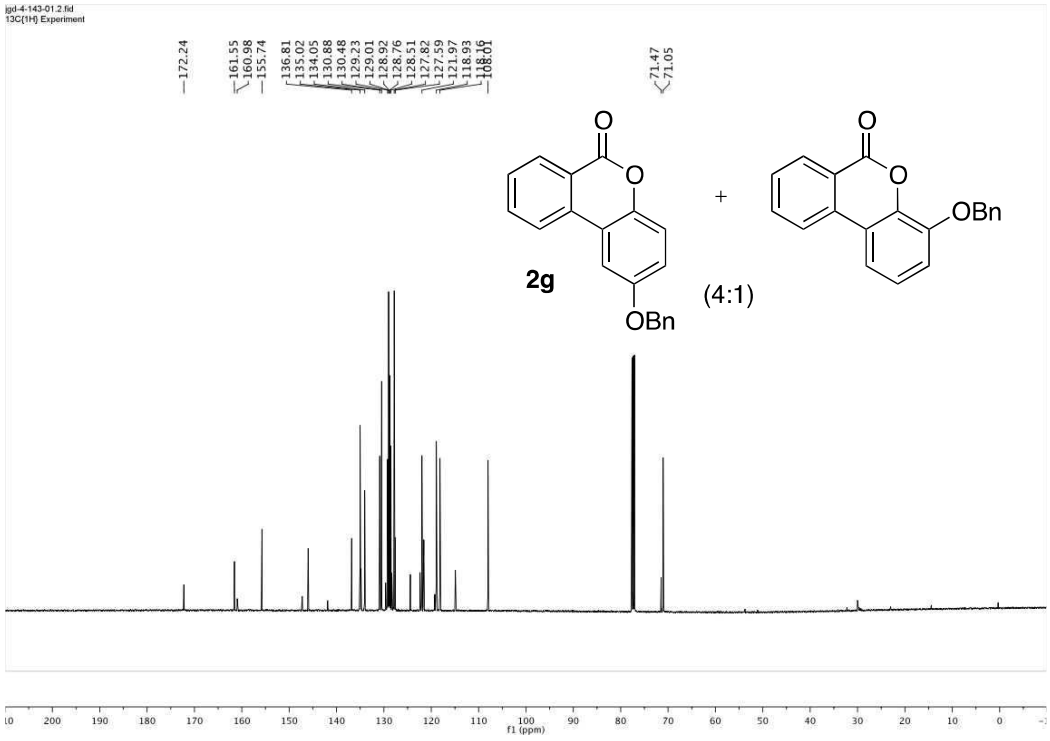

ResearchGroup Martin
jgd-4-30A-02
ICIQ_1H20p32s CDCl3 (C:\Bruker\TopSpin3.1) jgallardo 26
2e
Me
(2.5:1)
Me
f1 (ppm)

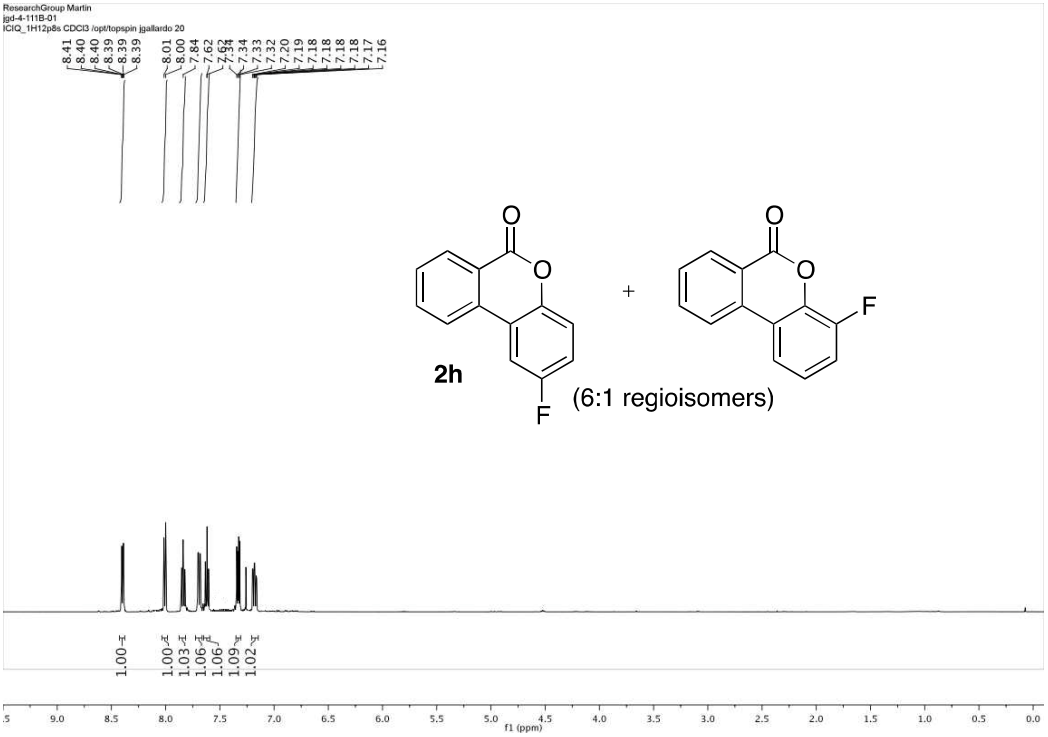

ResearchGroup Martin
jgd-4-30A
ICIQ_13C{1H}512s CDCl3 /opt/topspin jgallardo 3
2e
Me
(2.5:1)
Me
f1 (ppm)

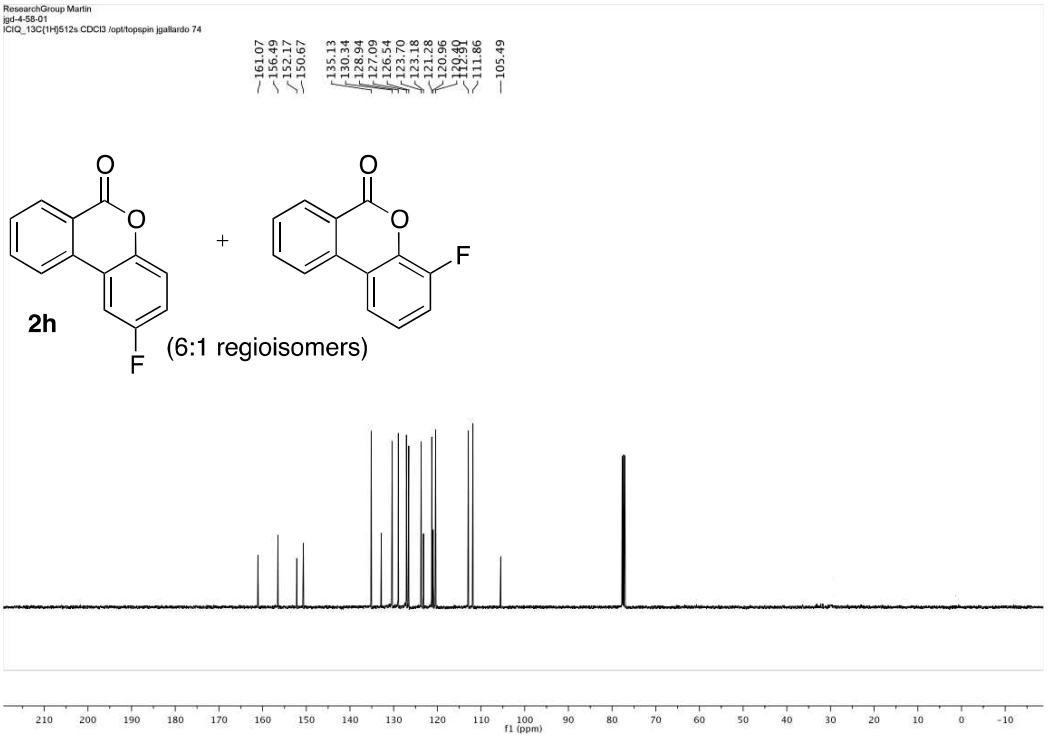

ResearchGroup Martin
jgd-4-30B-01
ICIQ_1H20p32s CDCl3 (C:\Bruker\TopSpin3.1) jgallardo 27
2f
(3:1)
OMe
f1 (ppm)

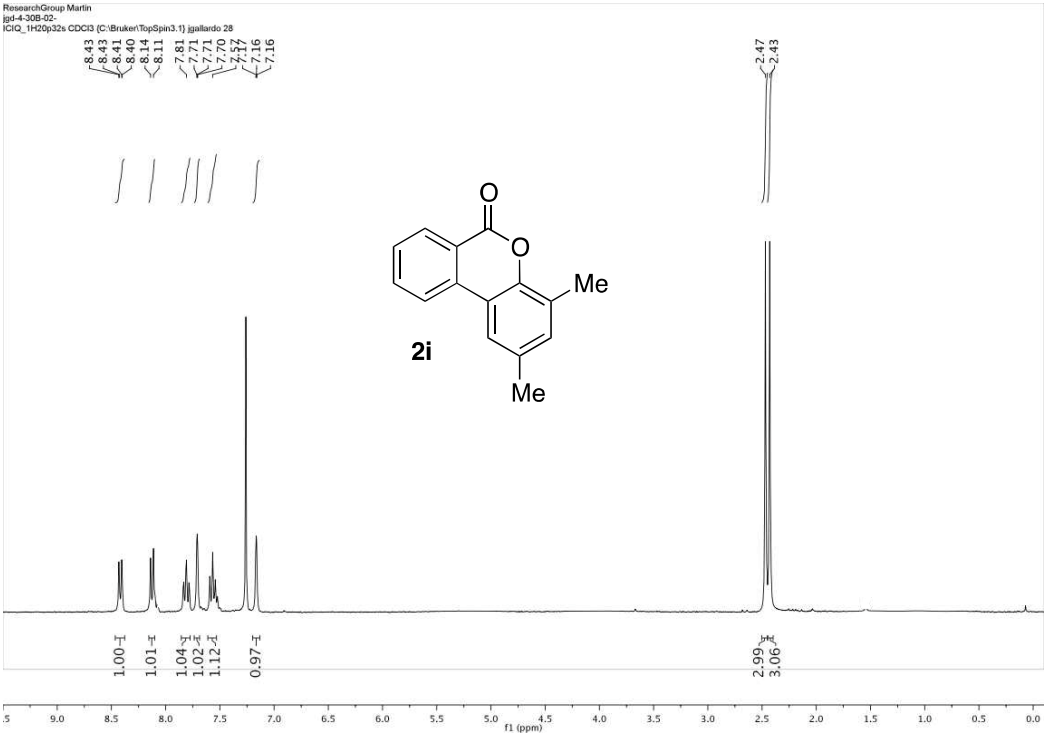

ResearchGroup Martin
jgd-4-30B
ICIQ_13C{1H}512s CDCl3 /opt/topspin jgallardo 26
2f
(3:1)
OMe
f1 (ppm)

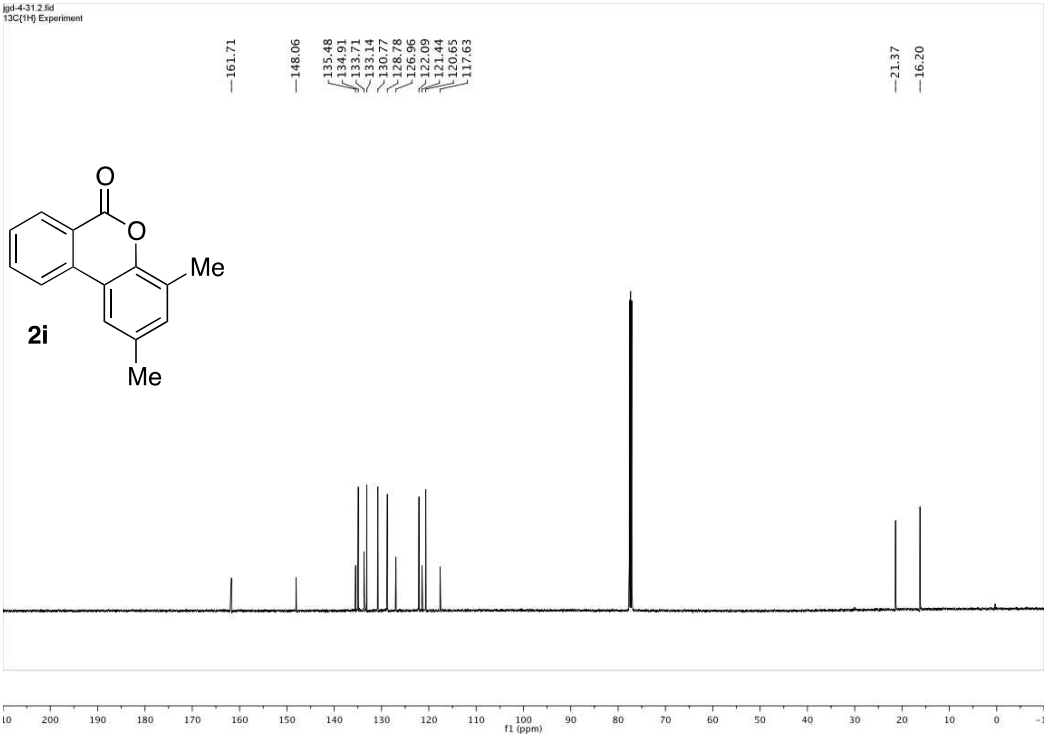

ResearchGroup Martin
jgd-4-143-01
ICIQ_1H12p8s CDCl3 /opt/topspin jgallardo 42
2g
(4:1)
OBn
f1 (ppm)

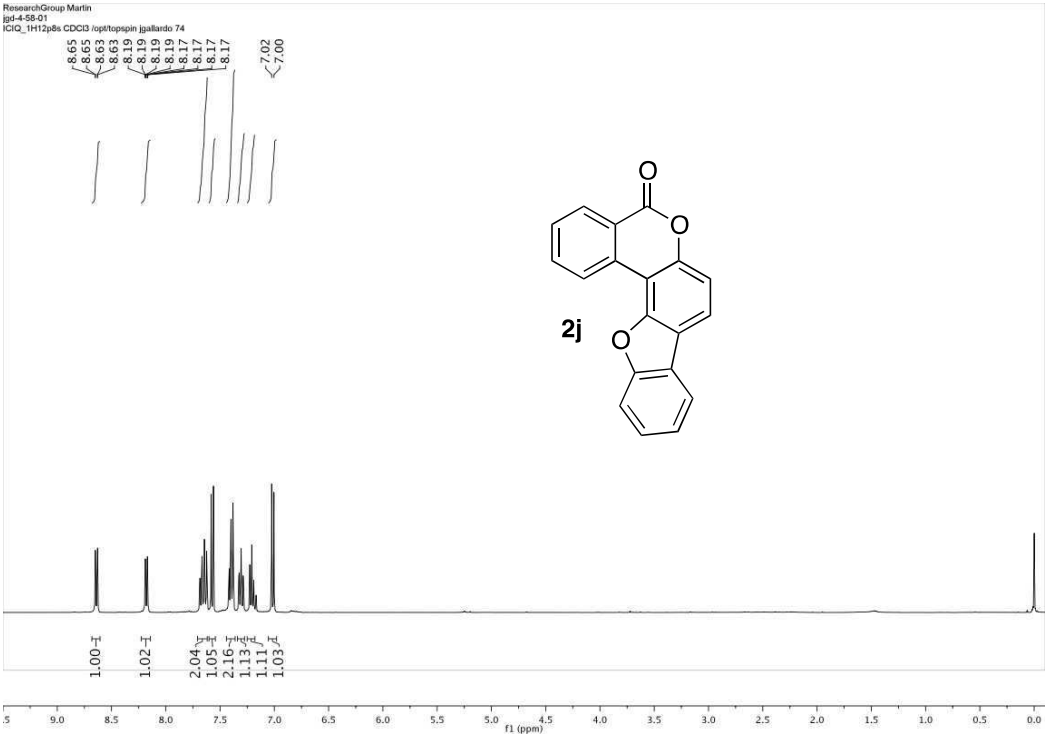

jgd-4-143-01.2.fid
13C{1H} Experiment
2g
(4:1)
OBn
f1 (ppm)

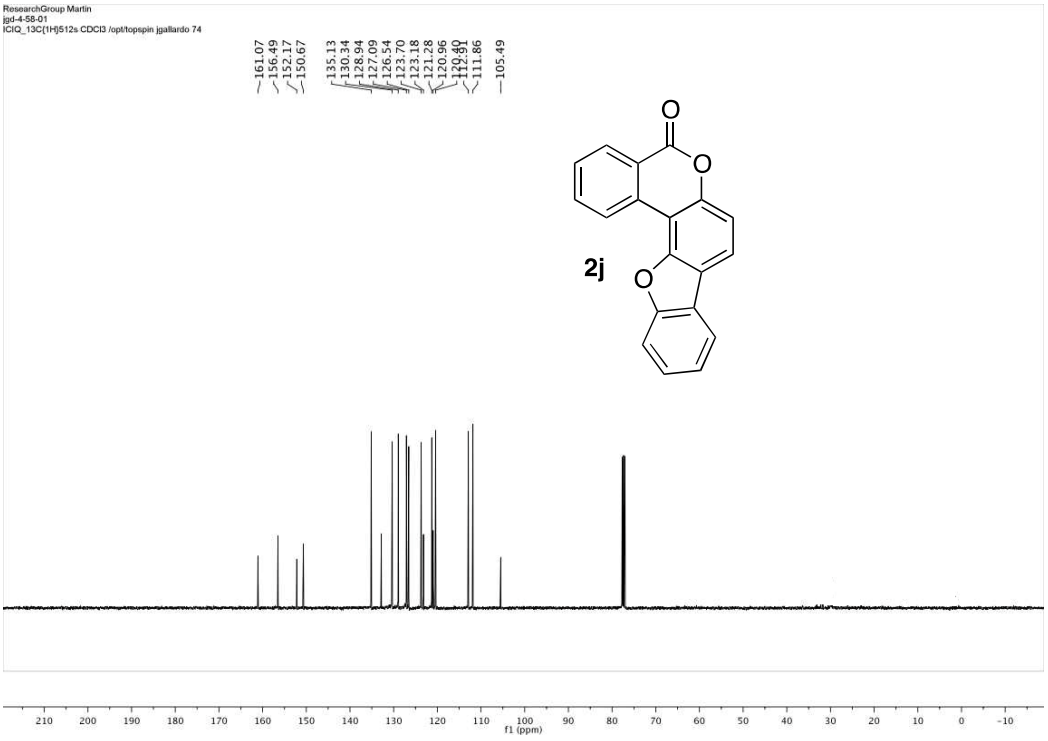

ResearchGroup Martin
jgd-4-111B-01
ICIQ_1H12p8s CDCl3 /opt/topspin jgallardo 20
8.41
8.40
8.40
8.39
8.39
8.39
8.01
8.00
7.84
7.62
7.62
7.34
7.34
7.33
7.32
7.20
7.19
7.18
7.18
7.18
7.18
7.17
7.16
O
O
+
O
O
F
2h
F
(6:1 regioisomers)
1.00
1.00
1.03
1.06
1.06
1.09
1.02
f1 (ppm)

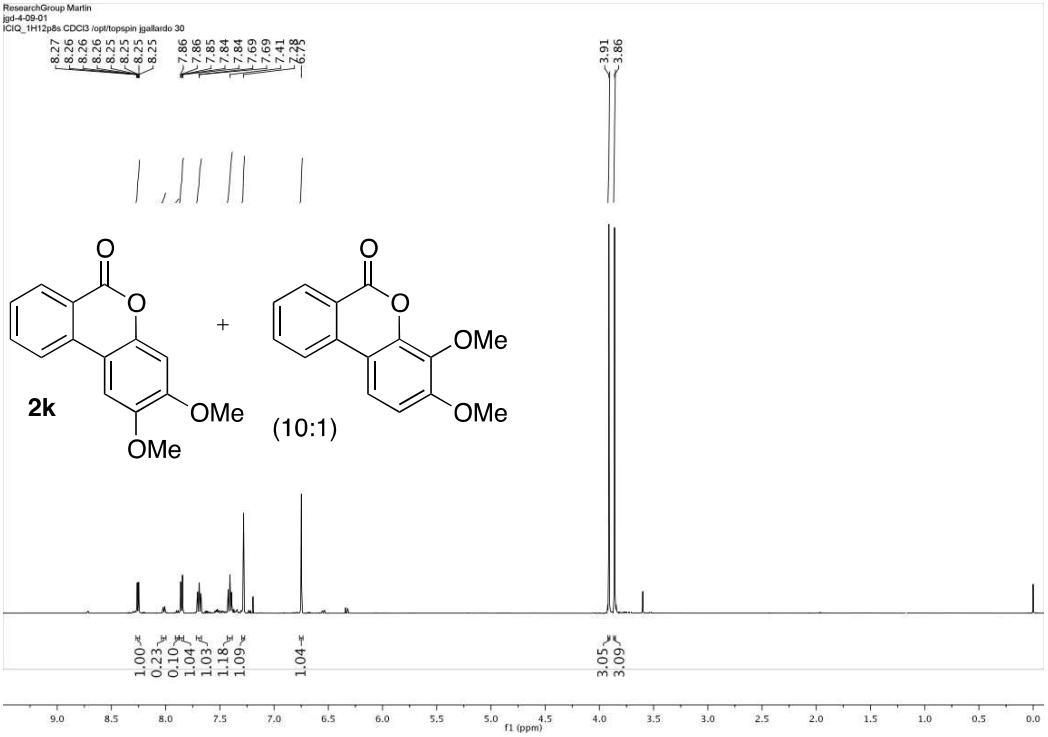

ResearchGroup Martin
jgd-4-58-01
ICIQ_13C{1H}512s CDCl3 /opt/topspin jgallardo 74
161.07
156.49
152.17
150.67
135.13
130.34
128.94
127.09
126.54
123.70
123.18
121.28
120.96
111.86
105.49
O
O
+
O
O
F
2h
F
(6:1 regioisomers)
f1 (ppm)

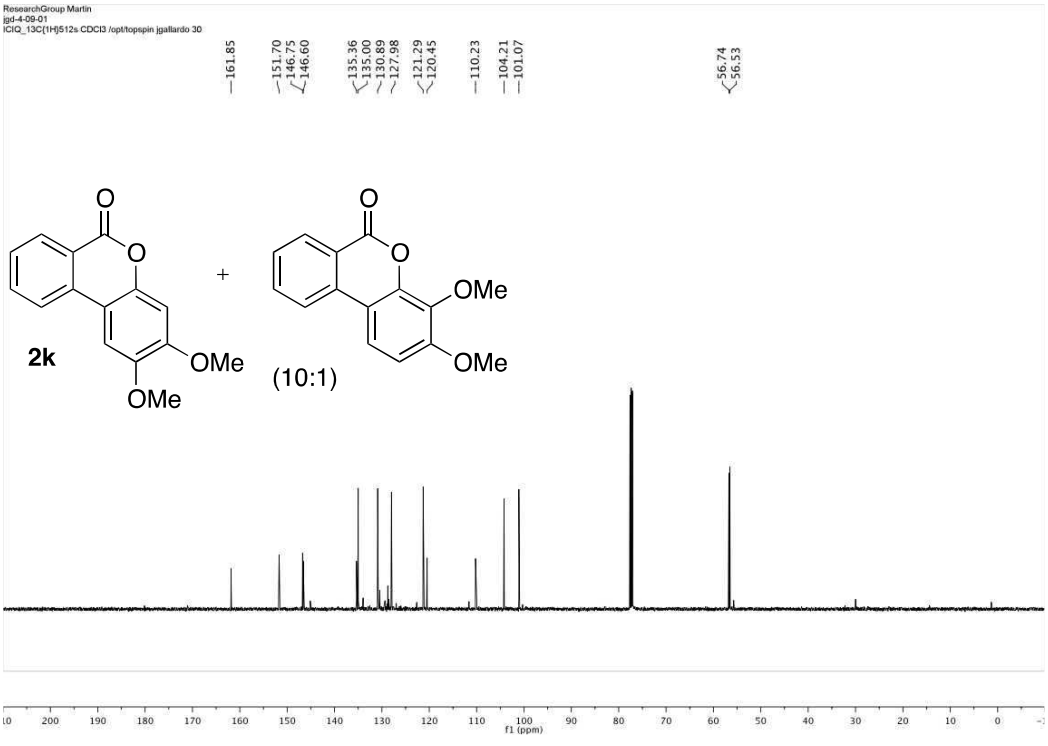

ResearchGroup Martin
jgd-4-30B-02-
ICIQ_1H20p32s CDCl3 (C:\Bruker\TopSpin3.1) jgallardo 28
8.43
8.43
8.41
8.40
8.14
8.11
7.81
7.71
7.71
7.70
7.57
7.17
7.16
7.16
2.47
2.43
O
O
Me
Me
2i
1.00
1.01
1.04
1.02
1.12
0.97
2.99
3.06
f1 (ppm)

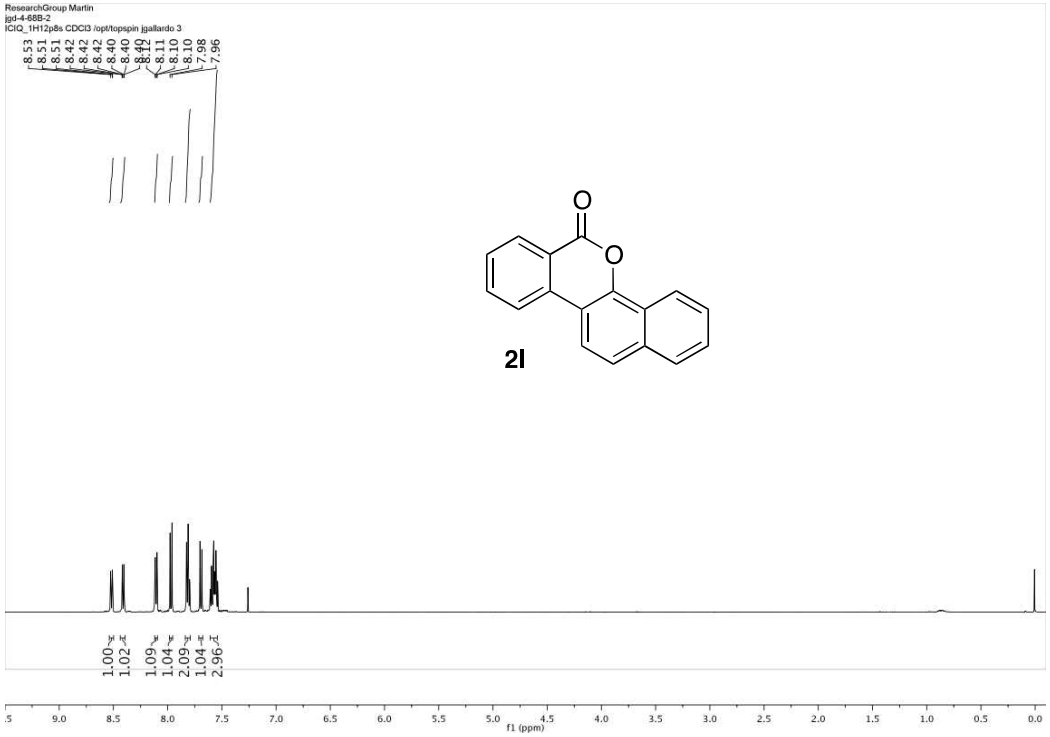

jgd-4-31.2.fid
13C{1H} Experiment
161.71
148.06
135.48
134.91
133.71
133.14
130.77
128.78
126.96
122.09
121.44
120.65
117.63
21.37
16.20
O
O
Me
Me
2i
f1 (ppm)

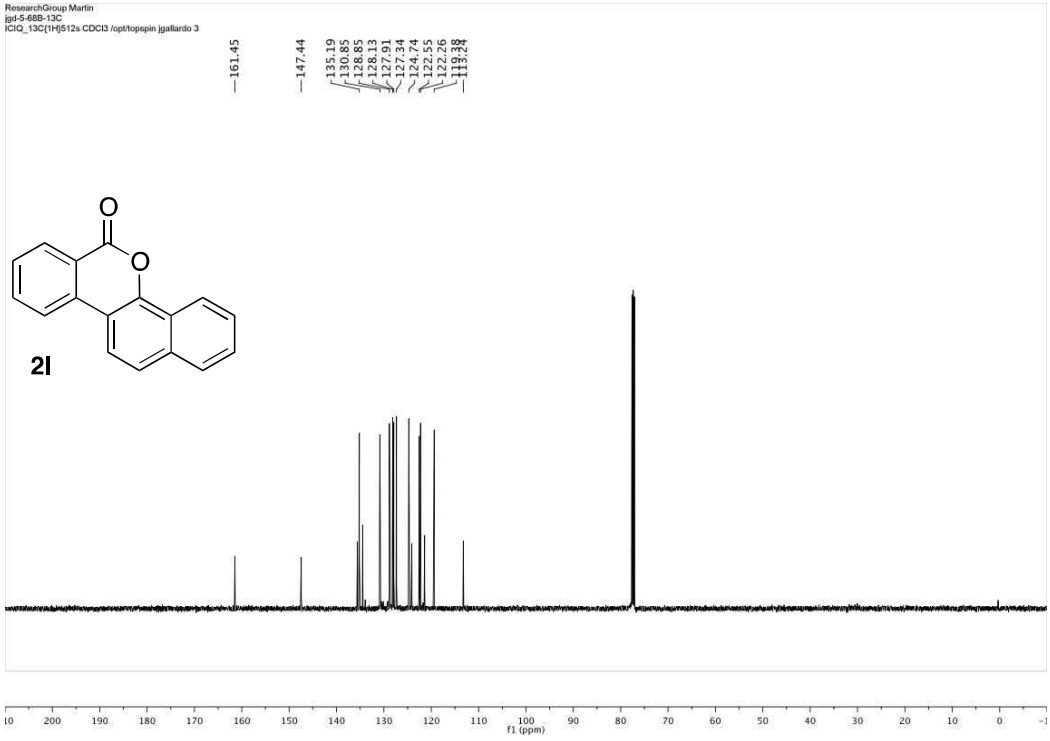

ResearchGroup Martin
jgd-4-58-01
ICIQ_1H12p8s CDCl3 /opt/topspin jgallardo 74
8.65
8.65
8.63
8.63
8.19
8.19
8.19
8.17
8.17
8.17
8.17
7.02
7.00
2j
1.00
1.02
2.04
1.05
2.16
1.13
1.11
1.03
f1 (ppm)

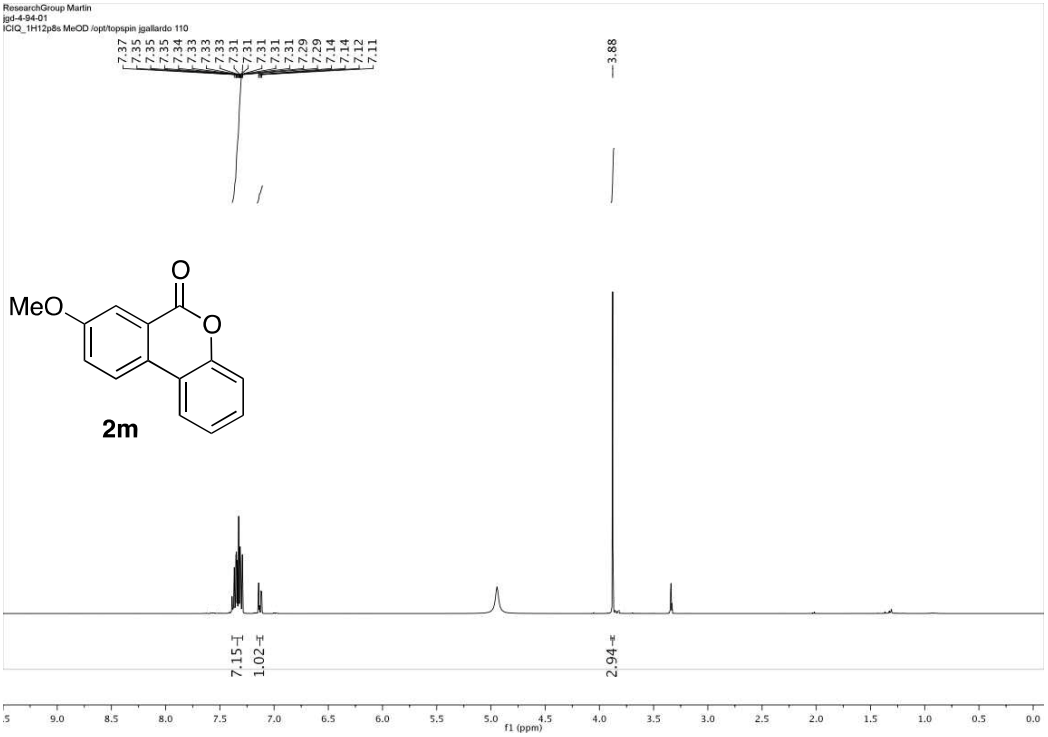

ResearchGroup Martin
jgd-4-58-01
ICIQ_13C{1H}512s CDCl3 /opt/topspin jgallardo 74
161.07
156.49
152.17
150.67
135.13
130.34
128.94
127.09
126.54
123.70
123.18
121.28
120.96
111.86
105.49
2j
f1 (ppm)

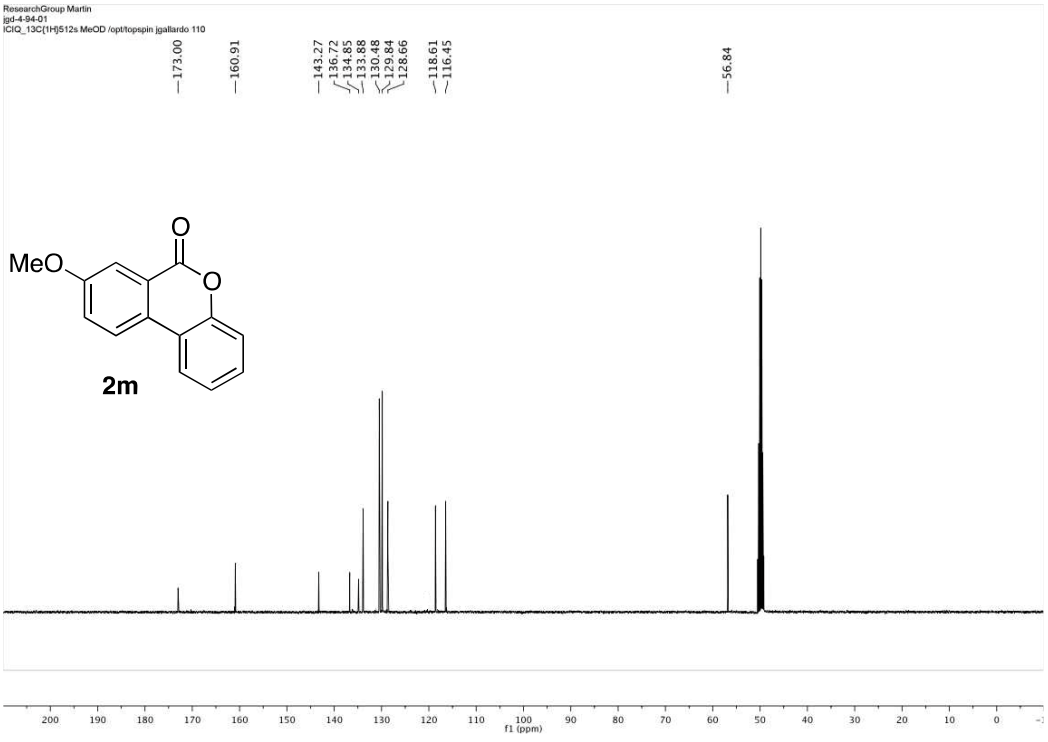

ResearchGroup Martin
jgd-4-09-01
ICIQ_1H12p8s CDCl3 /opt/topspin jgallardo 30
8.27
8.26
8.26
8.26
8.26
8.25
8.25
8.25
8.25
7.86
7.86
7.85
7.84
7.84
7.69
7.69
7.41
7.28
6.75
3.91
3.86
O
O
OMe
OMe
2k
+
O
O
OMe
OMe
(10:1)
1.00
0.23
0.10
1.04
1.03
1.18
1.09
1.04
3.05
3.09
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

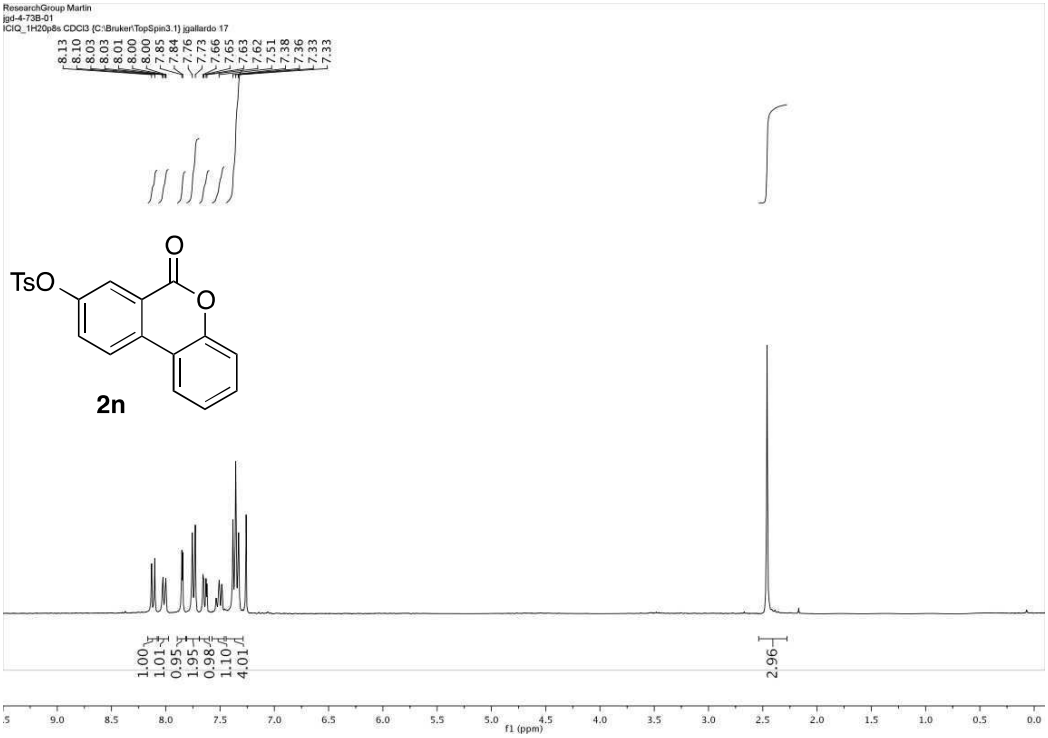

ResearchGroup Martin
jgd-4-09-01
ICIQ_13C{1H}512s CDCl3 /opt/topspin jgallardo 30
161.85
151.70
146.75
146.60
135.36
135.00
130.89
127.98
121.29
120.45
110.23
104.21
101.07
56.74
56.53
O
O
OMe
OMe
2k
+
O
O
OMe
OMe
(10:1)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

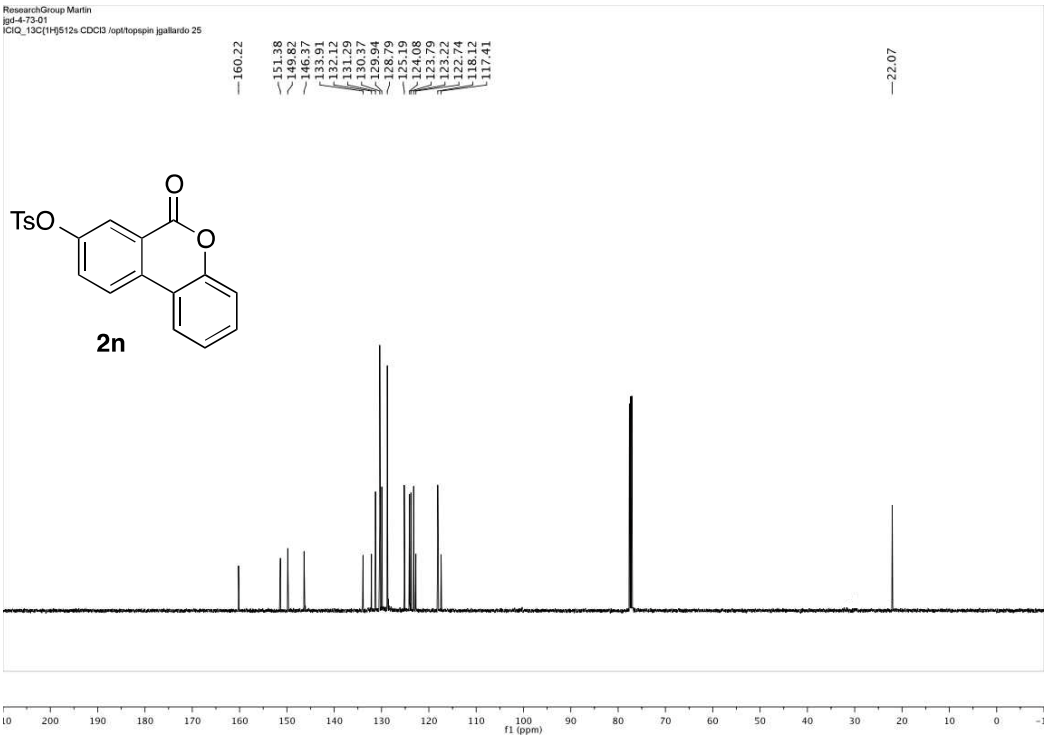

ResearchGroup Martin
jgd-4-68B-2
ICIQ_1H12p8s CDCl3 /opt/topspin jgallardo 3
8.53
8.51
8.51
8.42
8.42
8.42
8.40
8.40
8.40
8.12
8.11
8.10
8.10
7.98
7.96
O
O
2l
1.00
1.02
1.09
1.04
2.09
1.04
2.96
f1 (ppm)

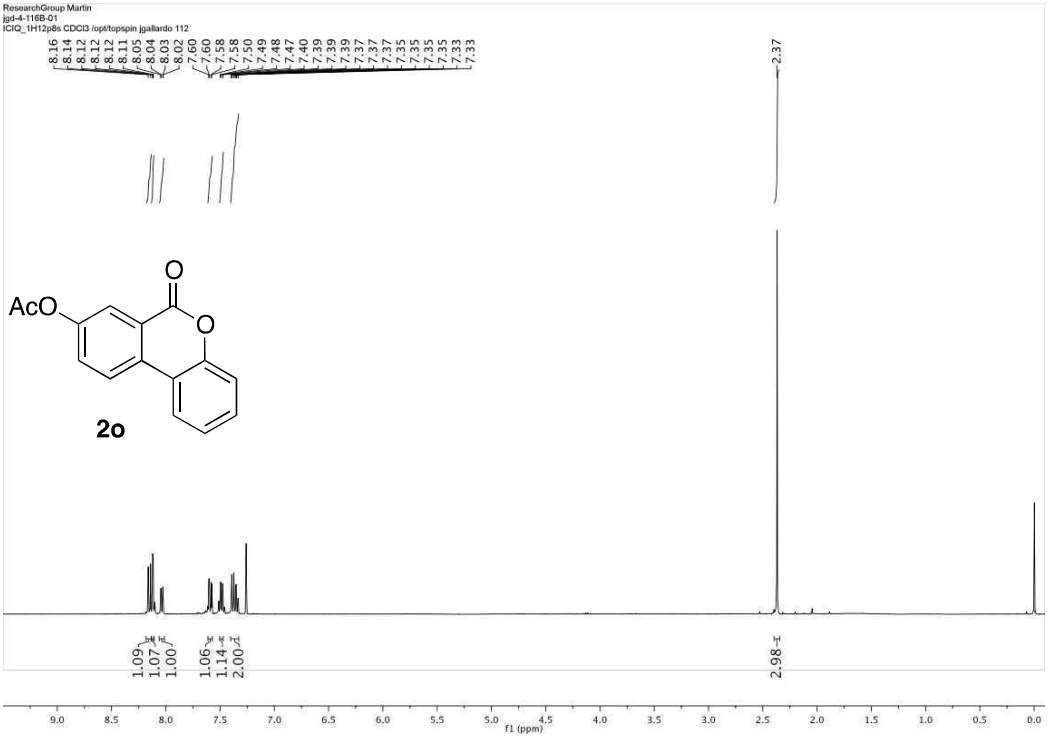

ResearchGroup Martin
jgd-5-68B-13C
ICIQ_13C{1H}512s CDCl3 /opt/topspin jgallardo 3
161.45
147.44
135.19
130.85
128.85
128.13
127.91
127.34
124.74
122.55
122.26
119.38
119.24
O
O
2l
f1 (ppm)

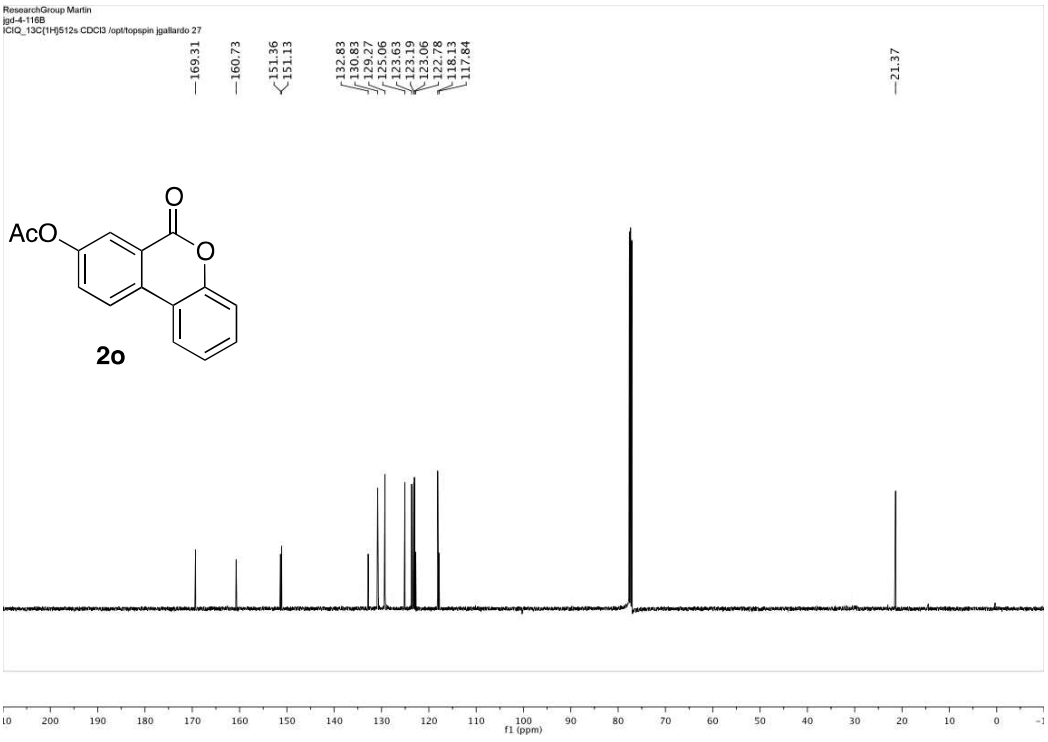

ResearchGroup Martin
jgd-4-94-01
ICIQ_1H12p8s MeOD /opt/topspin jgallardo 110
7.37
7.35
7.35
7.35
7.34
7.33
7.33
7.31
7.31
7.31
7.31
7.31
7.29
7.14
7.12
7.11
3.88
MeO
O
O
2m
7.15
1.02
2.94
f1 (ppm)

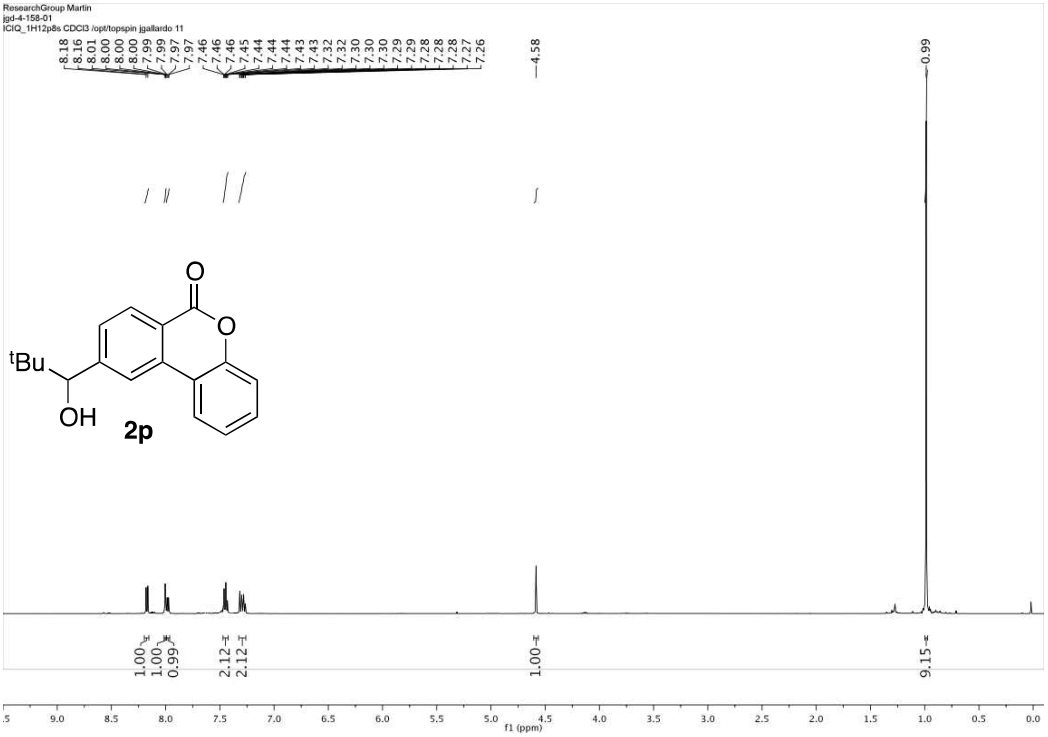

ResearchGroup Martin
jgd-4-94-01
ICIQ_13C{1H}512s MeOD /opt/topspin jgallardo 110
173.00
160.91
143.27
136.72
134.85
133.88
130.48
129.84
128.66
118.61
116.45
56.84
MeO
O
O
2m
f1 (ppm)

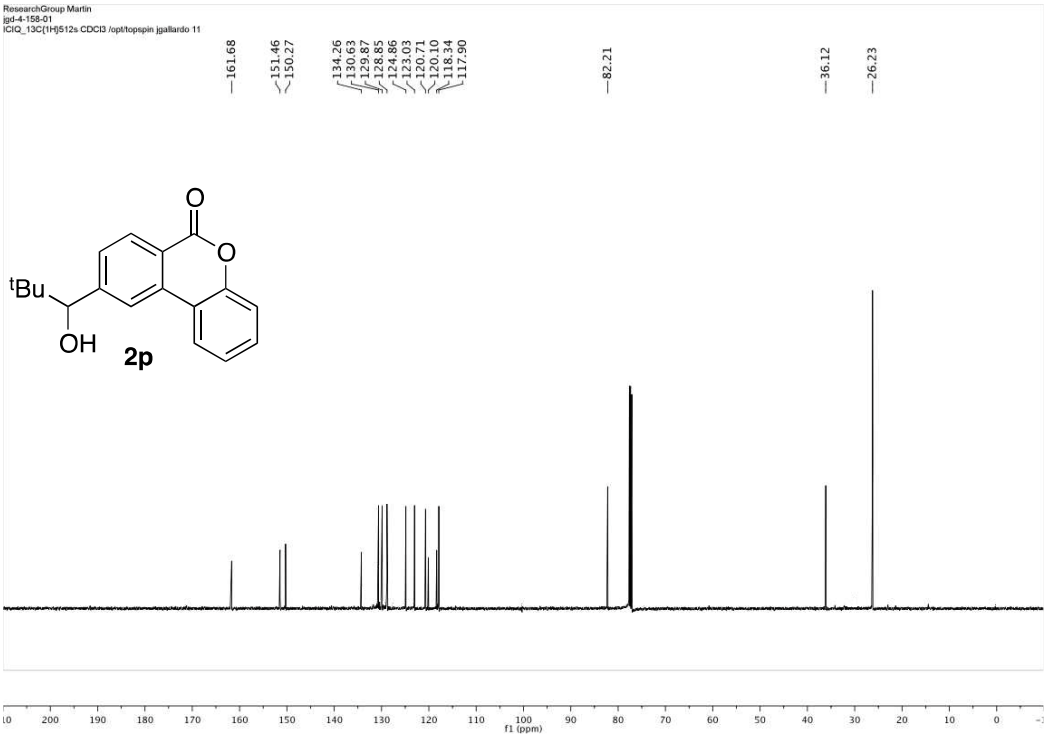

ResearchGroup Martin
jgd-4-73B-01
ICIQ_1H20p8s CDCl3 {C:\Bruker\TopSpin3.1} jgallardo 17
8.13
8.10
8.03
8.03
8.01
8.00
8.00
7.85
7.84
7.76
7.73
7.66
7.65
7.63
7.62
7.51
7.38
7.36
7.33
7.33
TsO
O
O
2n
1.00
1.01
0.95
1.95
0.98
1.10
4.01
2.96
f1 (ppm)

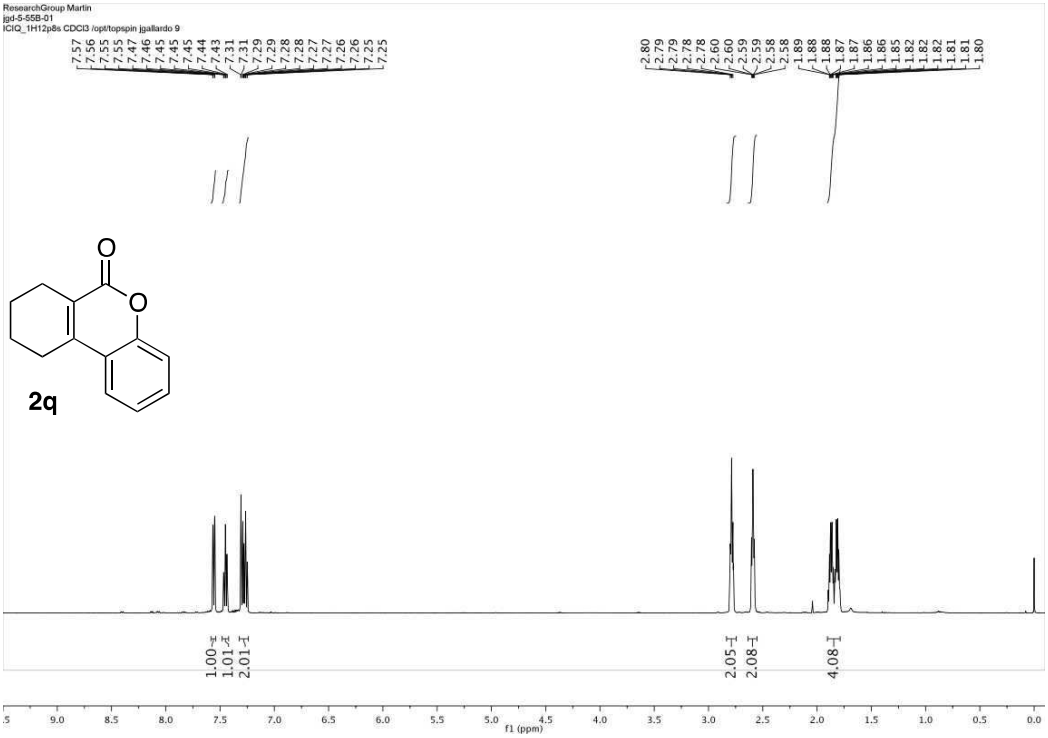

ResearchGroup Martin
jgd-4-73-01
ICIQ_13C{1H}512s CDCl3 /opt/topspin jgallardo 25
160.22
151.38
149.82
146.37
133.91
132.12
131.29
130.37
129.94
128.79
125.19
124.08
123.79
123.22
122.74
118.12
117.41
22.07
TsO
O
O
2n
f1 (ppm)

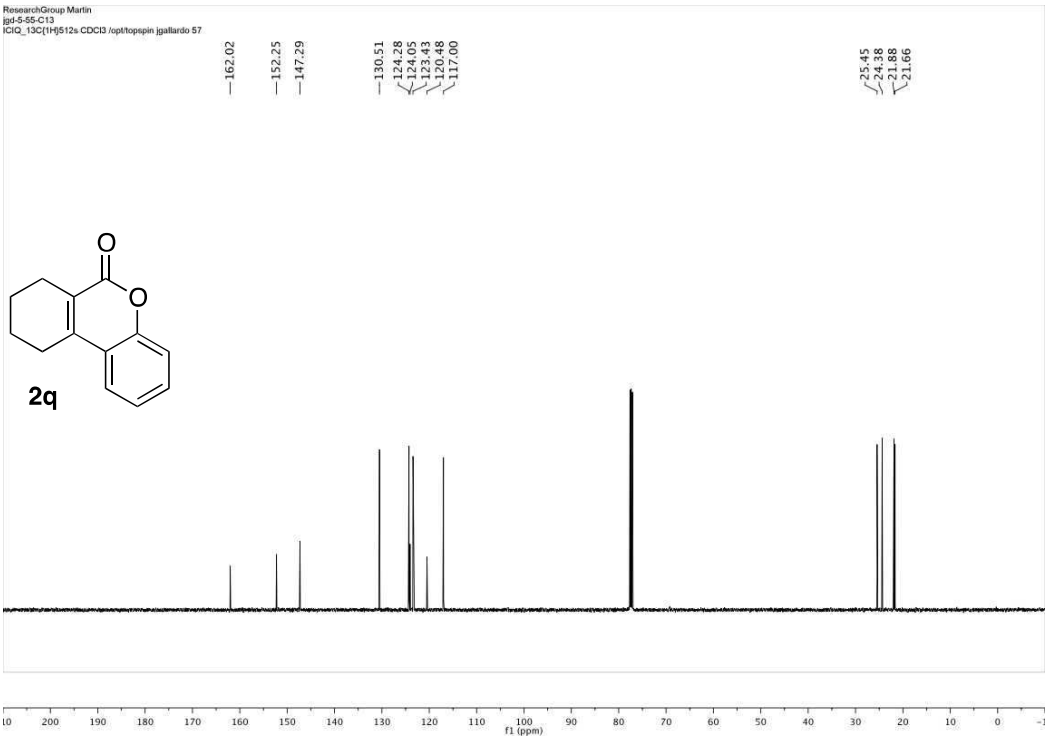

ResearchGroup Martin
jgd-4-116B-01
ICIQ_1H12p8s CDCl3 /opt/topspin jgallardo 112
8.16
8.14
8.12
8.12
8.12
8.12
8.11
8.05
8.04
8.03
8.02
7.60
7.60
7.58
7.58
7.50
7.49
7.48
7.47
7.40
7.39
7.39
7.39
7.37
7.37
7.37
7.35
7.35
7.35
7.35
7.33
7.33
2.37
AcO
O
O
2o
1.09
1.07
1.00
1.06
1.14
2.00
2.98
f1 (ppm)

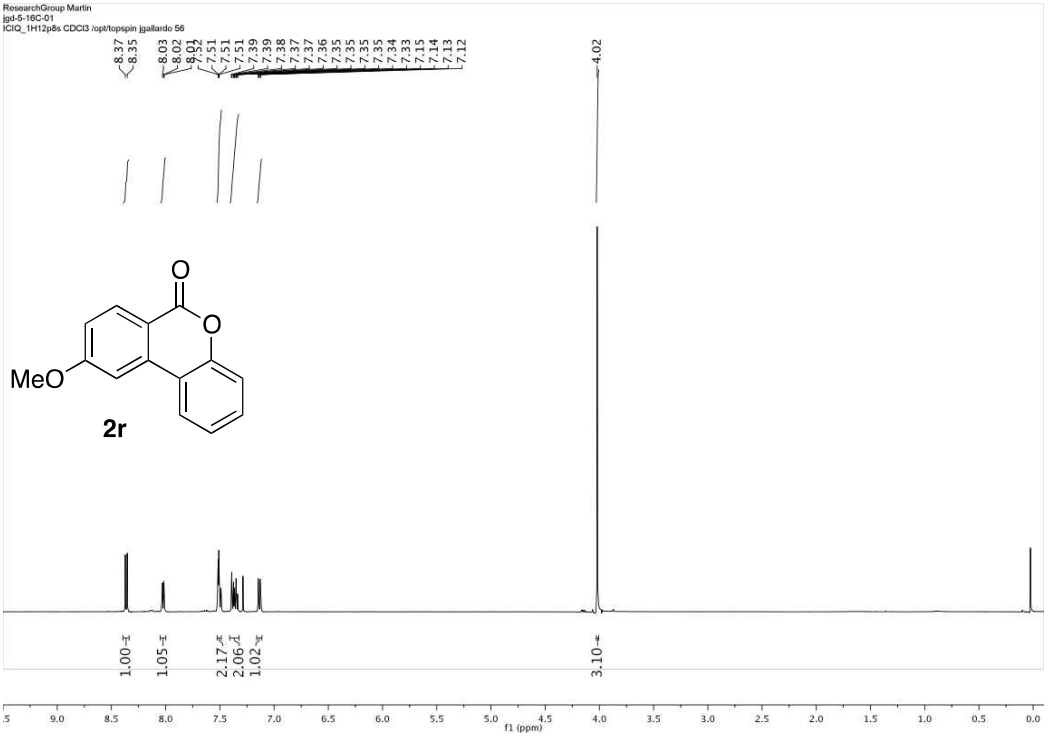

ResearchGroup Martin
jgd-4-116B
ICIQ_13C{1H}512s CDCl3 /opt/topspin jgallardo 27
169.31
160.73
151.36
151.13
132.83
130.83
129.27
125.06
123.63
123.19
123.06
122.78
118.13
117.84
21.37
AcO
O
O
2o
f1 (ppm)

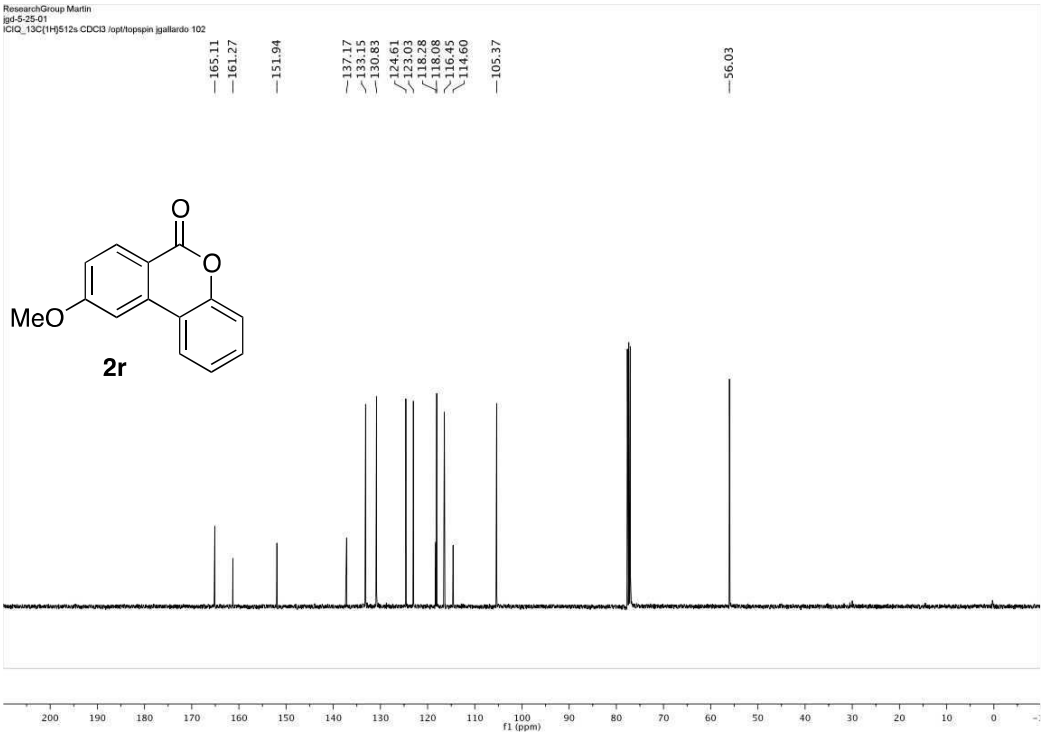

ResearchGroup Martin
jgd-4-158-01
ICIQ_1H12p8s CDCl3 /opt/topspin jgallardo 11
8.18
8.16
8.01
8.00
8.00
8.00
7.99
7.99
7.97
7.97
7.46
7.46
7.46
7.45
7.44
7.44
7.44
7.43
7.43
7.32
7.32
7.30
7.30
7.29
7.29
7.28
7.28
7.28
7.27
7.26
4.58
0.99
1.00
1.00
0.99
2.12
2.12
1.00
9.15
f1 (ppm)
tBu
OH
2p

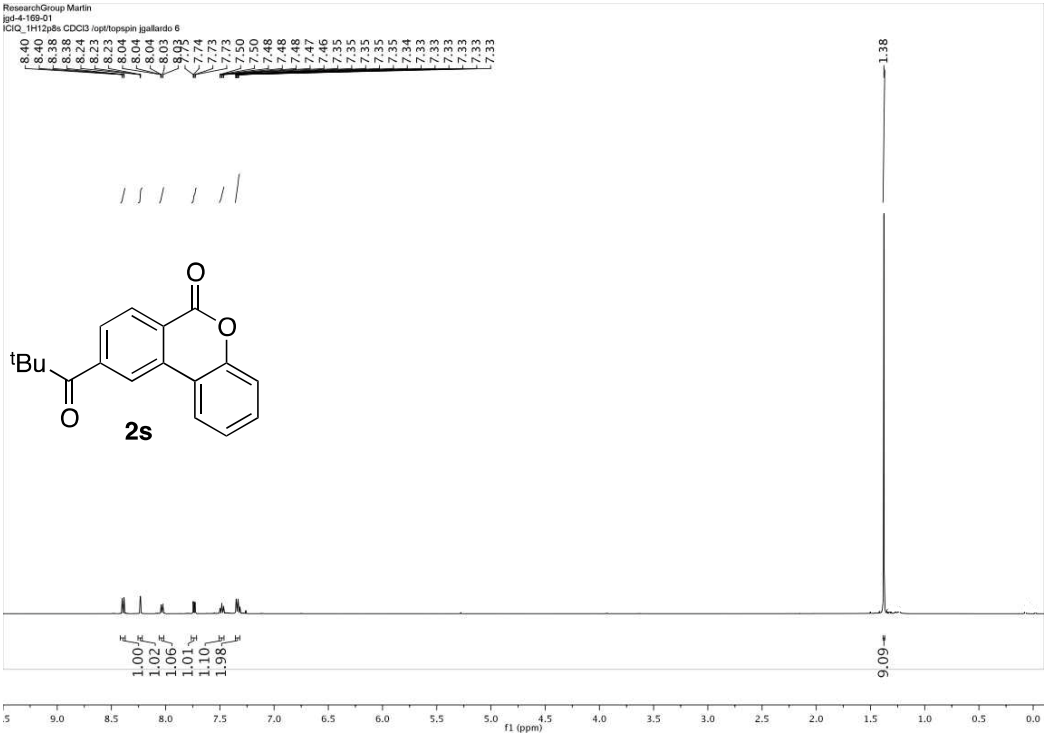

ResearchGroup Martin
jgd-4-158-01
ICIQ_13C{1H}512s CDCl3 /opt/topspin jgallardo 11
161.68
151.46
150.27
134.26
130.63
129.87
128.85
124.86
123.03
120.71
120.10
118.34
117.90
82.21
36.12
26.23
f1 (ppm)
tBu
OH
2p

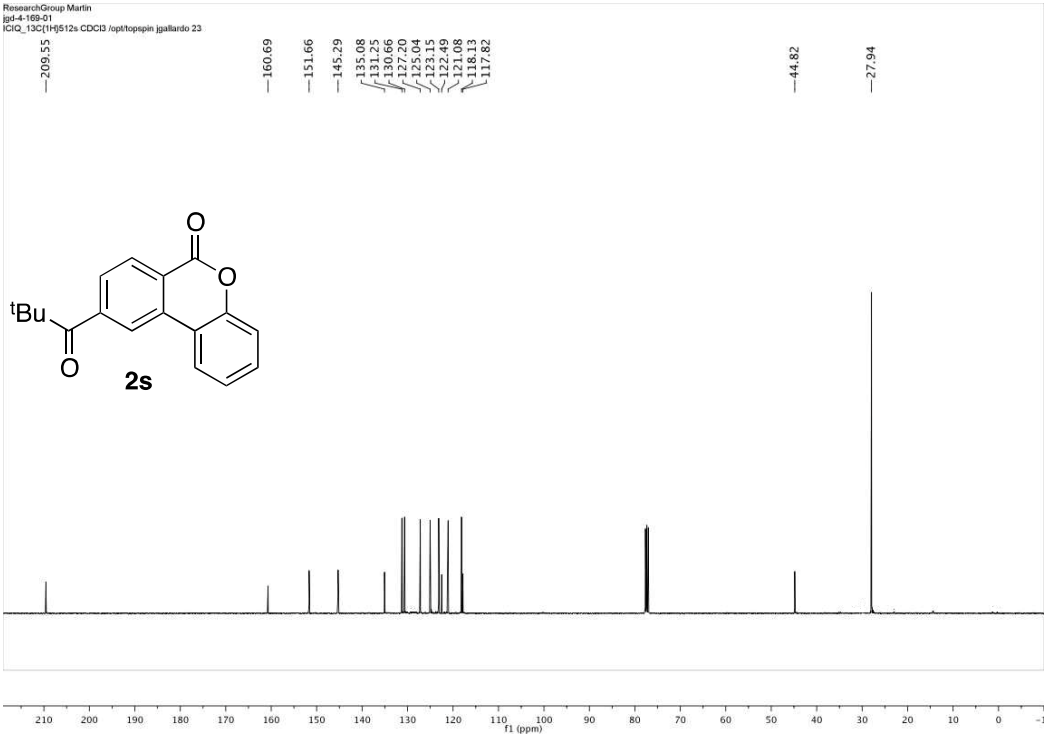

ResearchGroup Martin
jgd-5-55B-01
ICIQ_1H12p8s CDCl3 /opt/topspin jgallardo 9
7.57 7.56 7.55 7.55 7.47 7.46 7.45 7.45 7.45 7.44 7.43 7.31 7.29 7.29 7.28 7.28 7.27 7.27 7.26 7.26 7.25 7.25
2.80 2.79 2.79 2.78 2.78 2.60 2.60 2.59 2.59 2.58 2.58 1.89 1.88 1.88 1.87 1.87 1.86 1.86 1.85 1.82 1.82 1.82 1.81 1.81 1.80
O
O
2q
1.00
1.01
2.01
2.05
2.08
4.08
f1 (ppm)

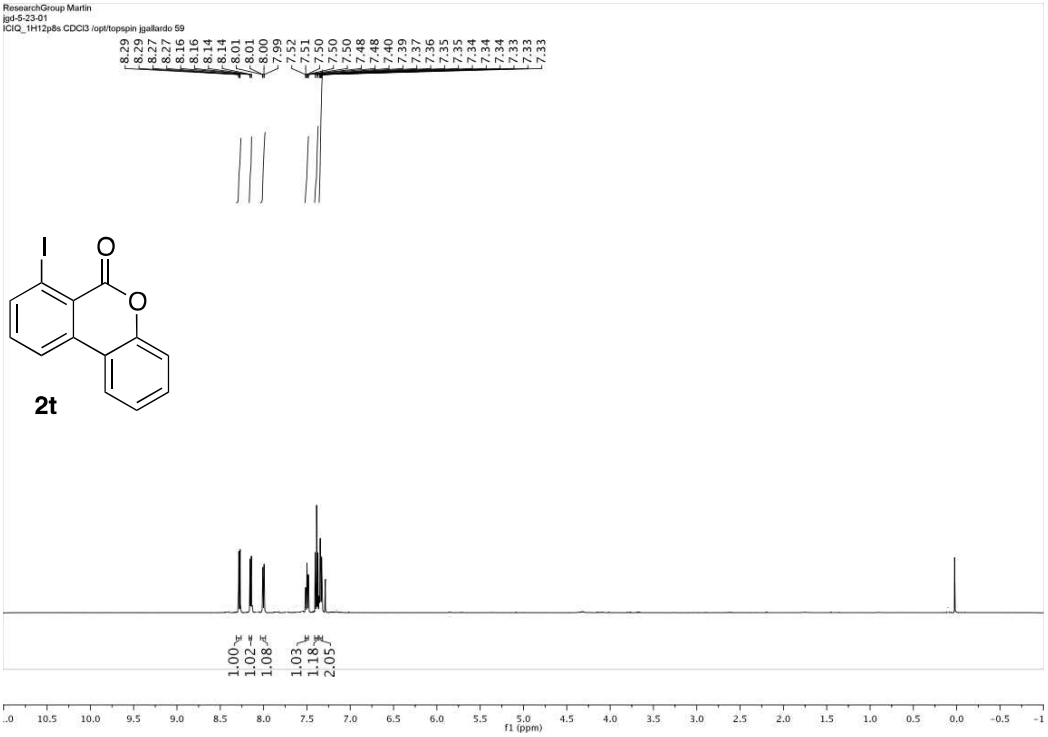

ResearchGroup Martin
jgd-5-55-C13
ICIQ_13C{1H}512s CDCl3 /opt/topspin jgallardo 57
162.02
152.25
147.29
130.51
124.28
124.05
123.43
120.48
117.00
25.45
24.38
21.88
21.66
O
O
2q
f1 (ppm)

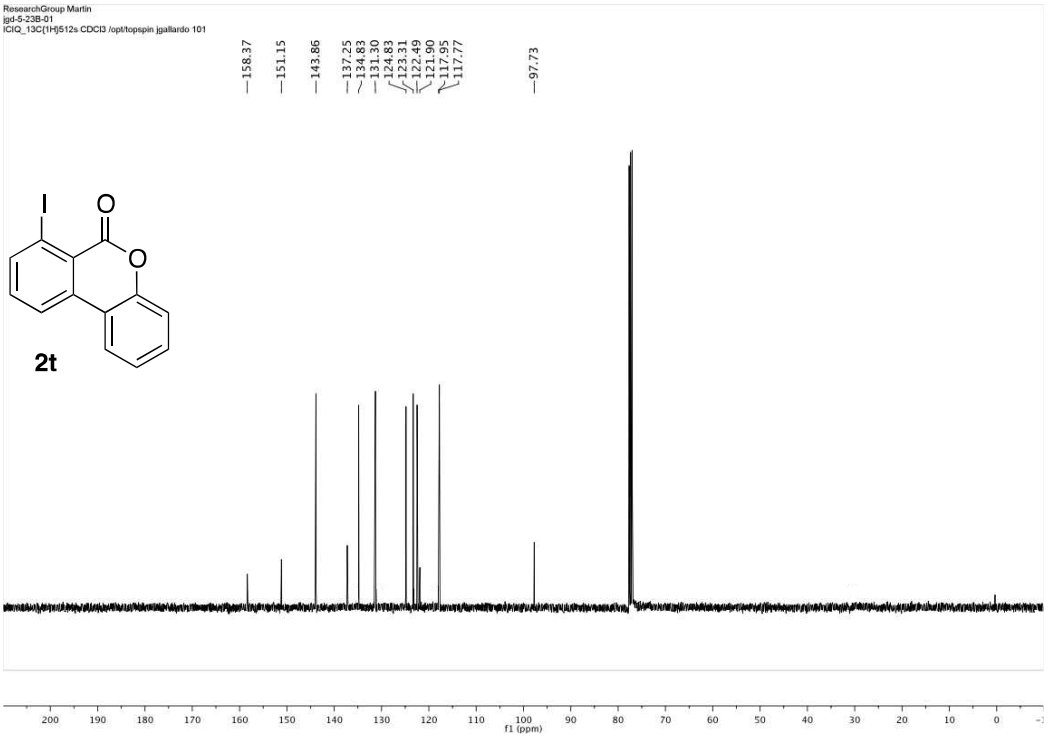

ResearchGroup Martin
jgd-5-16C-01
ICIQ_1H12p8s CDCl3 /opt/topspin jgallardo 56
8.37
8.35
8.03
8.02
8.01
7.52
7.51
7.51
7.39
7.39
7.38
7.37
7.36
7.35
7.35
7.35
7.34
7.33
7.15
7.14
7.13
7.12
4.02
O
O
MeO
2r
1.00
1.05
2.17
2.06
1.02
3.10
f1 (ppm)

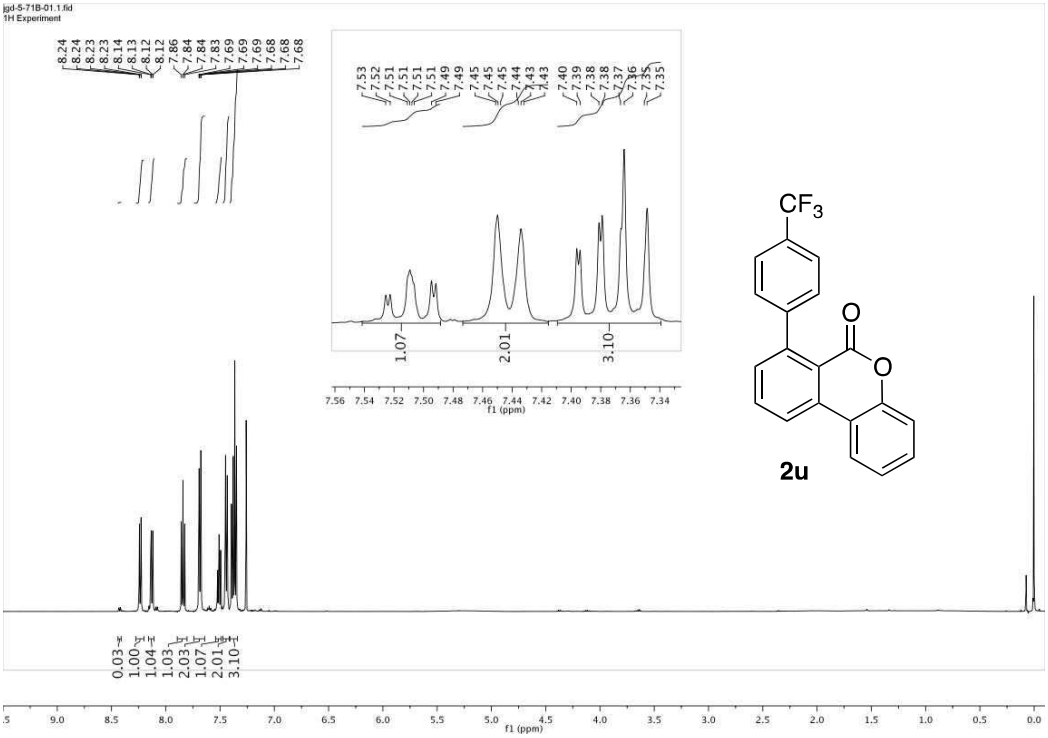

ResearchGroup Martin
jgd-5-25-01
ICIQ_13C{1H}512s CDCl3 /opt/topspin jgallardo 102
165.11
161.27
151.94
137.17
133.15
130.83
124.61
123.93
118.28
118.08
116.45
114.60
105.37
56.03
O
O
MeO
2r
f1 (ppm)

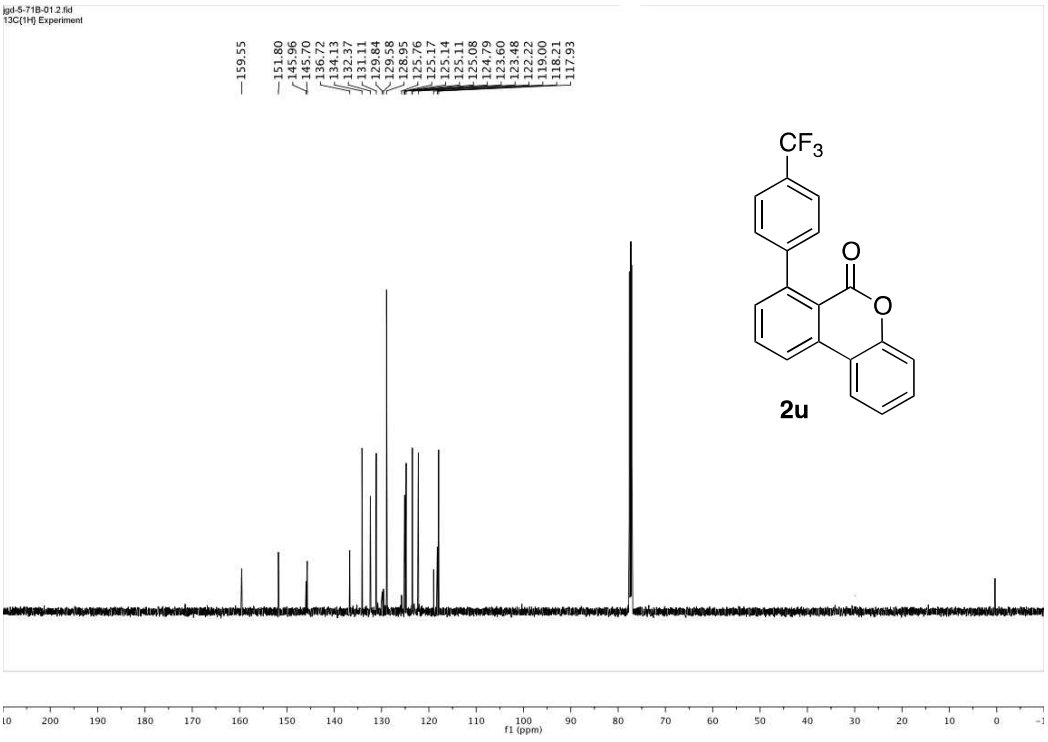

ResearchGroup Martin
jgd-4-169-01
ICIQ_1H12p8s CDCl3 /opt/topspin jgallardo 6
8.40
8.40
8.38
8.38
8.24
8.23
8.23
8.04
8.04
8.04
8.03
8.03
7.75
7.74
7.73
7.73
7.50
7.50
7.48
7.48
7.47
7.46
7.35
7.35
7.35
7.35
7.34
7.33
7.33
7.33
7.33
7.33
1.38
tBu
O
O
O
2s
1.00
1.02
1.06
1.01
1.10
1.98
9.09
f1 (ppm)

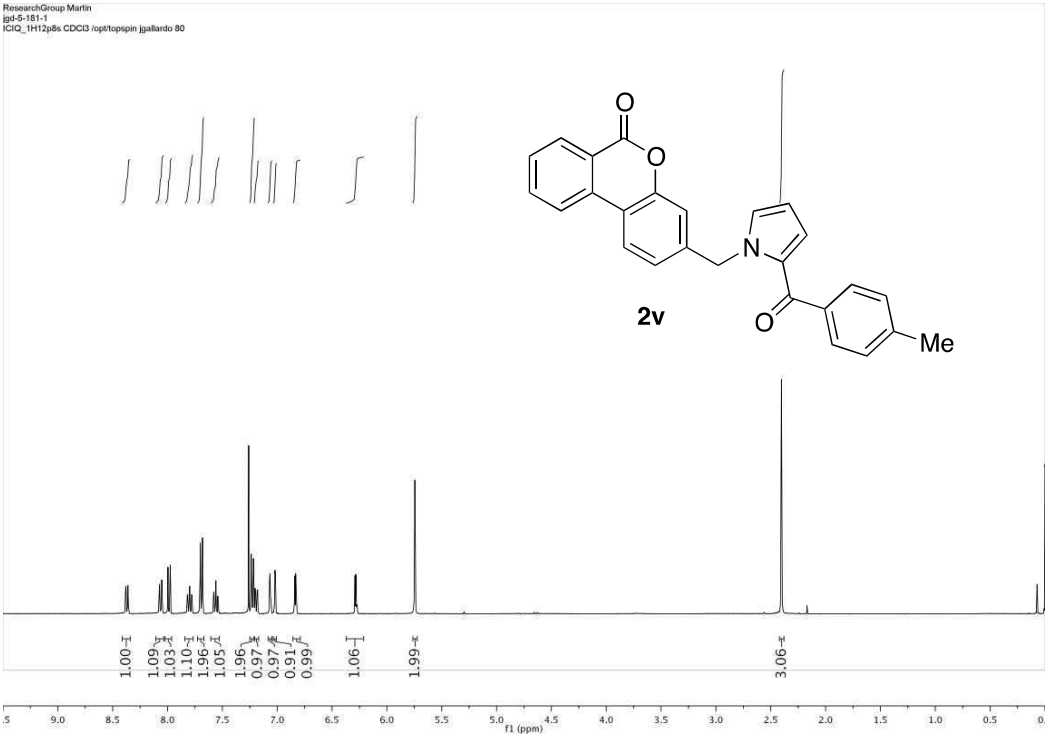

ResearchGroup Martin
jgd-4-169-01
ICIQ_13C{1H}512s CDCl3 /opt/topspin jgallardo 23
209.55
160.69
151.66
145.29
135.08
131.25
130.66
127.20
125.04
123.15
122.49
121.08
118.13
117.82
44.82
27.94
tBu
O
O
O
2s
f1 (ppm)

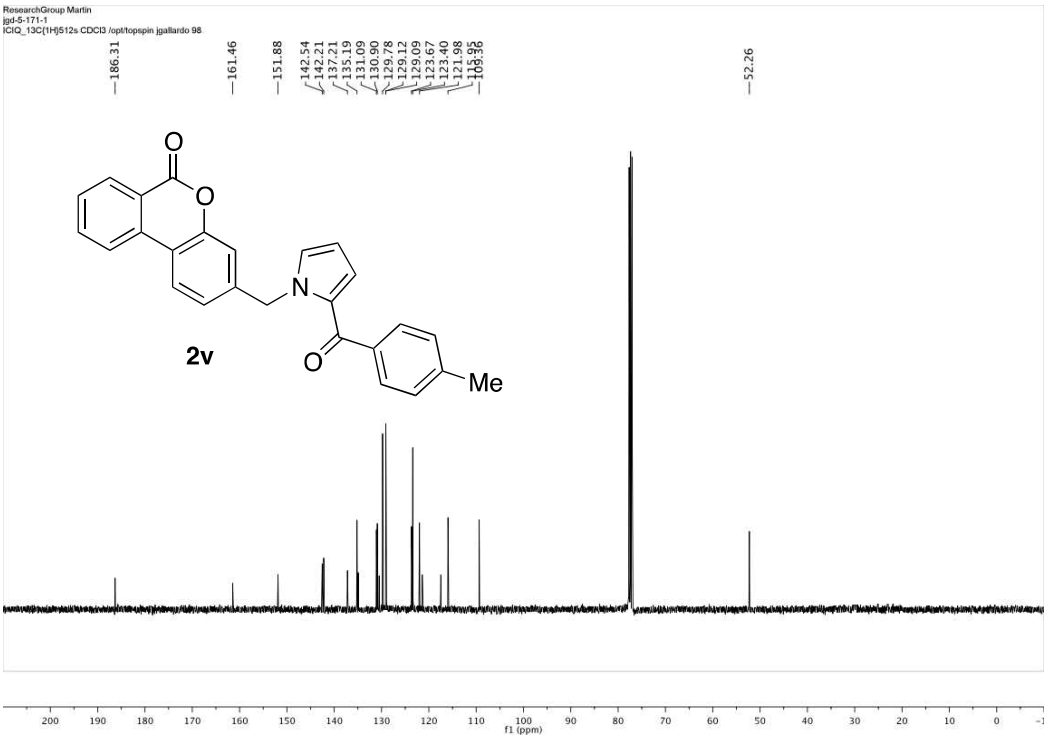

ResearchGroup Martin
jgd-5-23-01
ICIQ_1H12p8s CDCl3 /opt/topspin jgallardo 59
8.29
8.29
8.27
8.27
8.16
8.16
8.14
8.14
8.01
8.01
8.00
7.99
7.52
7.51
7.50
7.50
7.50
7.48
7.48
7.40
7.39
7.37
7.36
7.35
7.35
7.34
7.34
7.33
7.33
7.33
I
O
O
2t
1.00
1.02
1.08
1.03
1.18
2.05
f1 (ppm)

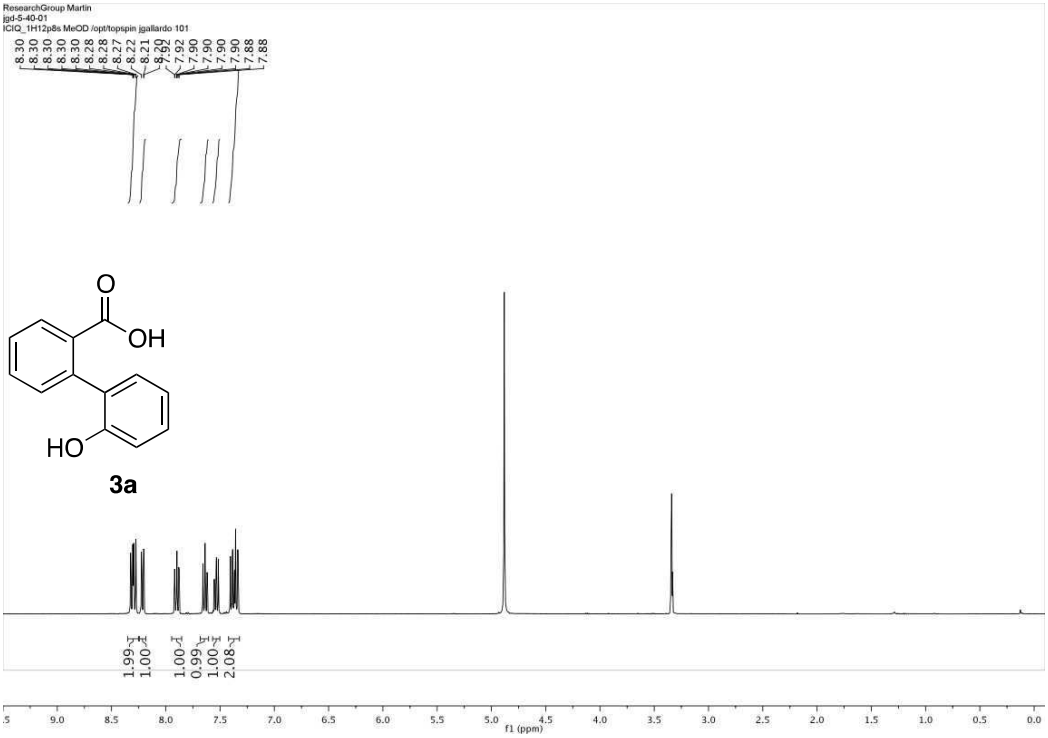

ResearchGroup Martin
jgd-5-23B-01
ICIQ_13C{1H}512s CDCl3 /opt/topspin jgallardo 101
158.37
151.15
143.86
137.25
134.83
131.30
124.83
123.31
122.49
121.90
117.95
117.77
97.73
I
O
O
2t
f1 (ppm)

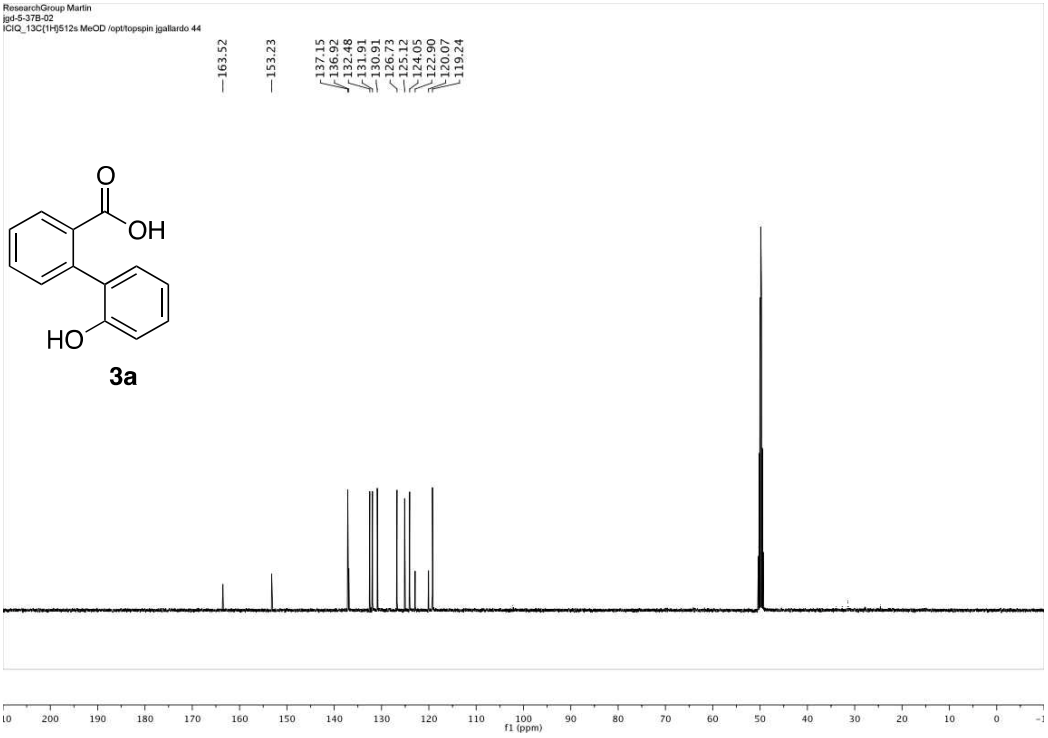

jgd-5-71B-01.1.fid
1H Experiment
8.24
8.24
8.23
8.23
8.14
8.13
8.12
8.12
7.86
7.84
7.84
7.83
7.69
7.69
7.69
7.68
7.68
7.68
7.53
7.52
7.51
7.51
7.51
7.51
7.49
7.49
7.45
7.45
7.45
7.44
7.43
7.43
7.40
7.39
7.38
7.38
7.37
7.36
7.35
7.35
1.07
2.01
3.10
7.56 7.54 7.52 7.50 7.48 7.46 7.44 7.42 7.40 7.38 7.36 7.34
f1 (ppm)
CF3
O
O
2u
0.03
1.00
1.04
1.03
2.03
1.07
2.01
3.10
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

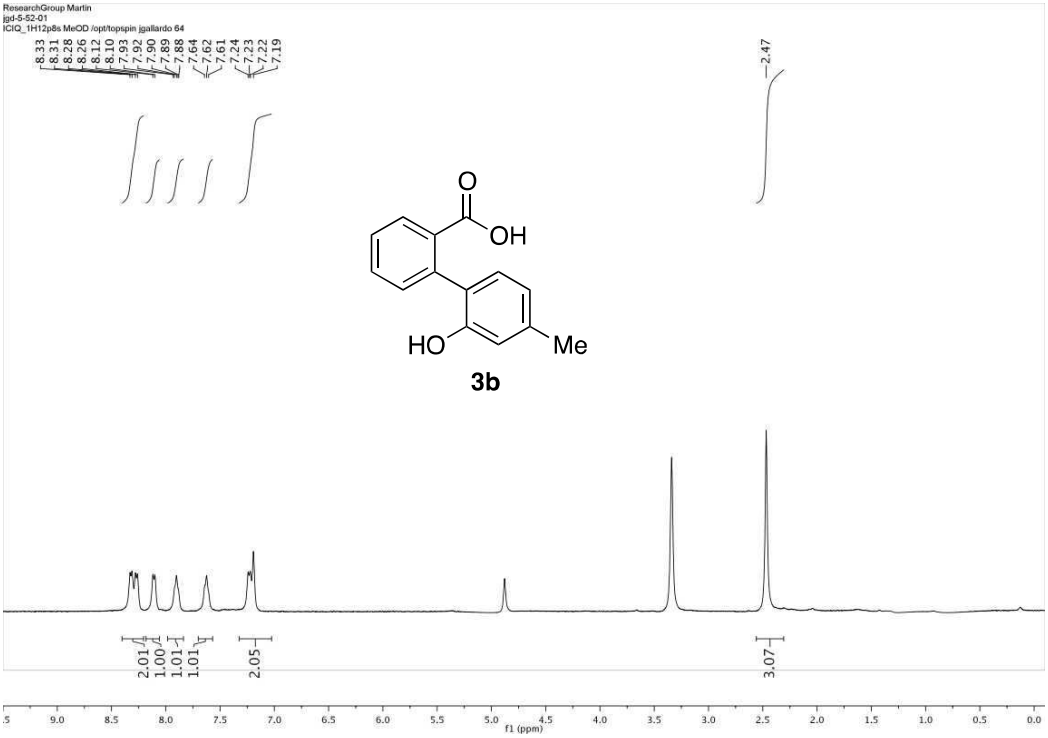

jgd-5-71B-01.2.fid
13C{1H} Experiment
159.55
151.80
145.96
145.70
136.72
134.13
132.37
131.11
129.84
129.58
128.95
125.76
125.17
125.14
125.11
125.08
124.79
123.60
123.48
122.22
119.00
118.21
117.93
CF3
O
O
2u
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

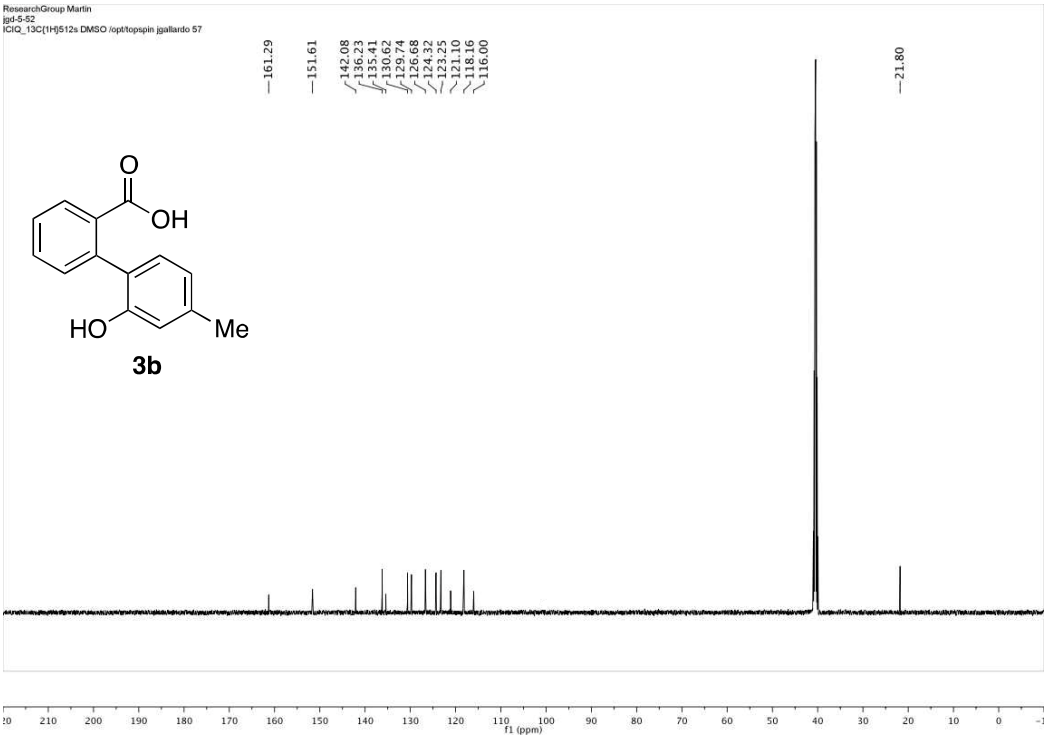

ResearchGroup Martin
jgd-5-181-1
ICIQ_1H12p8s CDCl3 /opt/topspin jgallardo 80
2v
Me
1.00
1.09
1.03
1.10
1.96
1.05
1.96
0.97
0.97
0.91
0.99
1.06
1.99
3.06
f1 (ppm)

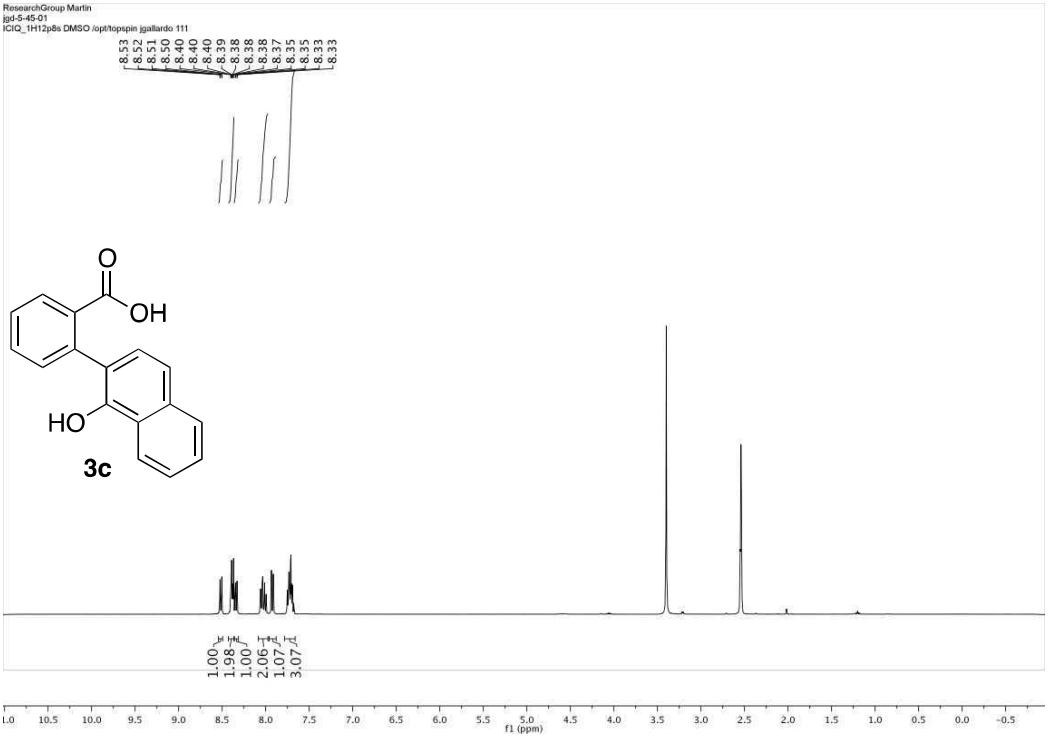

ResearchGroup Martin
jgd-5-171-1
ICIQ_13C{1H}512s CDCl3 /opt/topspin jgallardo 98
186.31
161.46
151.88
142.54
142.21
137.21
135.19
131.09
130.90
129.78
129.12
129.09
123.67
123.40
121.98
115.95
109.36
52.26
2v
Me
f1 (ppm)

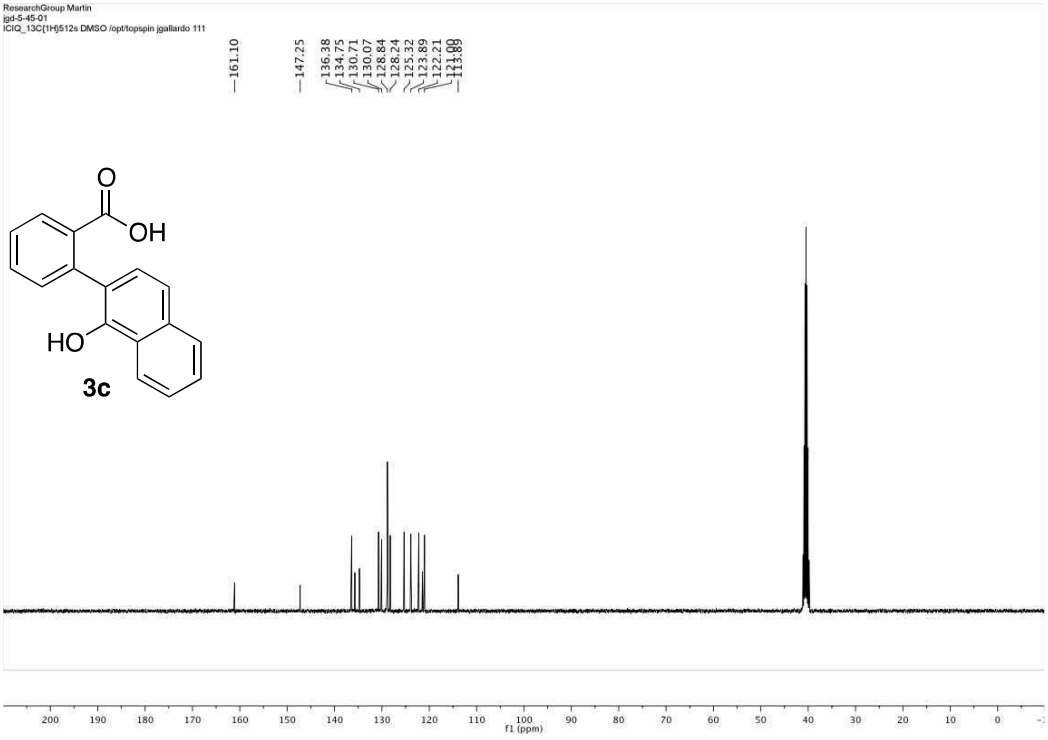

ResearchGroup Martin
jgd-5-40-01
ICIQ_1H12p8s MeOD /opt/topspin jgallardo 101
8.30
8.30
8.30
8.30
8.30
8.28
8.28
8.27
8.22
8.21
7.92
7.90
7.90
7.90
7.90
7.88
7.88
O
OH
HO
3a
1.99
1.00
1.00
0.99
1.00
2.08
f1 (ppm)

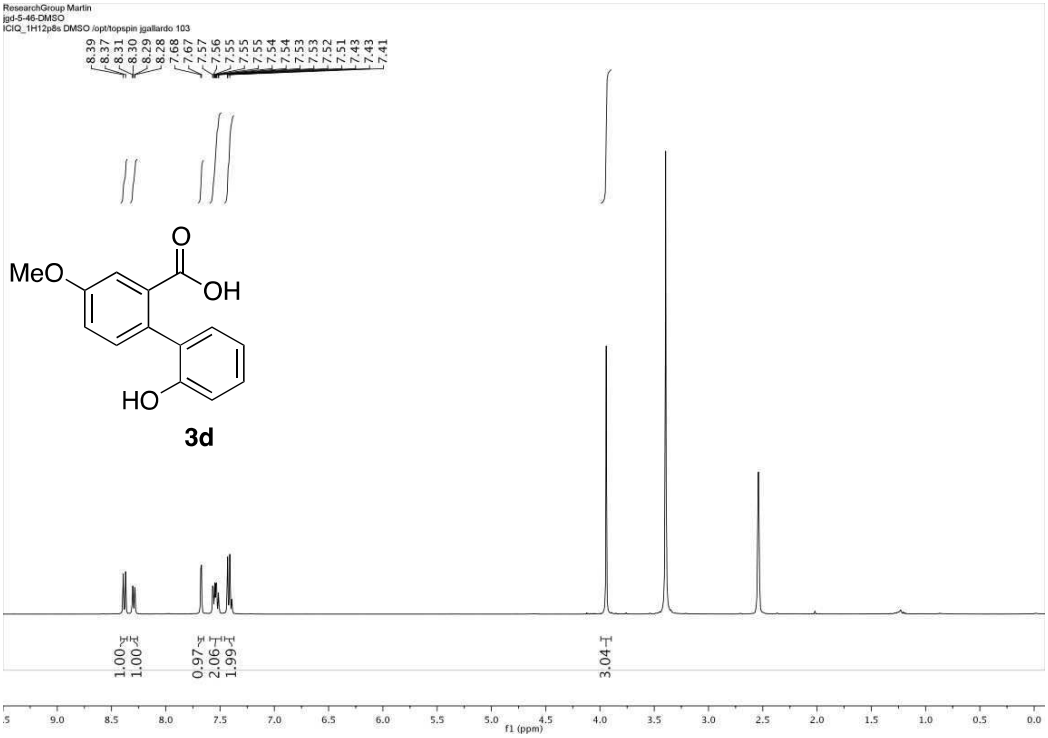

ResearchGroup Martin
jgd-5-37B-02
ICIQ_13C{1H}512s MeOD /opt/topspin jgallardo 44
163.52
153.23
137.15
136.92
132.48
131.91
130.91
126.73
125.12
124.05
122.90
120.07
119.24
O
OH
HO
3a
f1 (ppm)

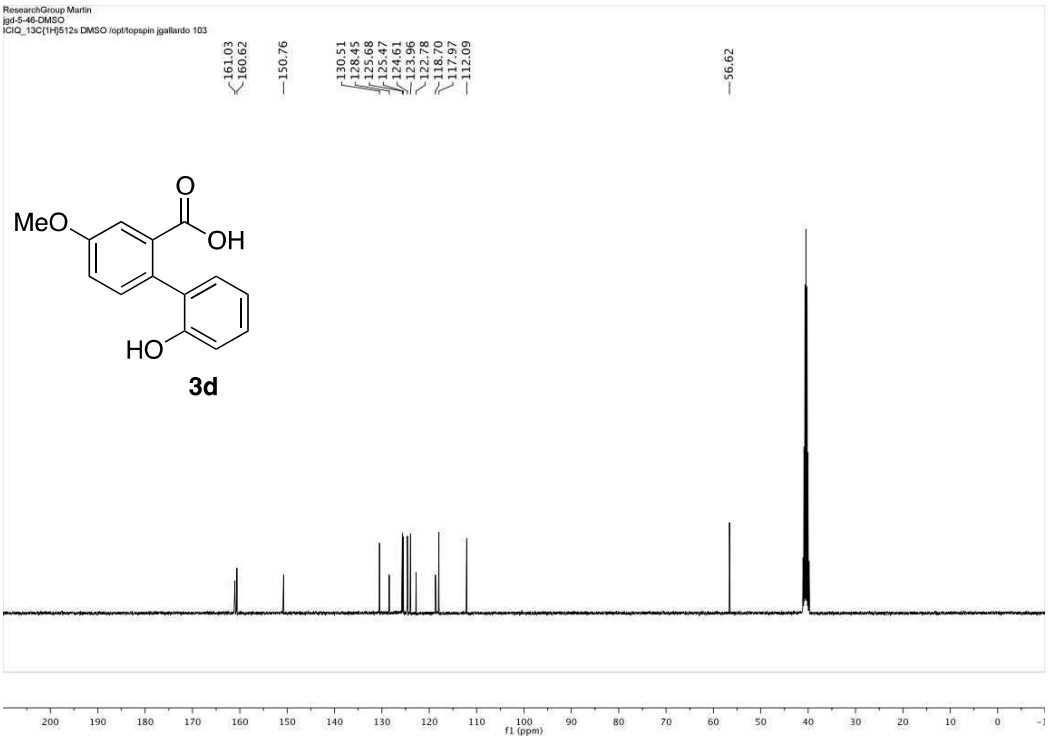

ResearchGroup Martin
jgd-5-52-01
ICIQ_1H12p8s MeOD /opt/topspin jgallardo 64
8.33
8.31
8.28
8.26
8.12
8.10
7.93
7.92
7.90
7.89
7.88
7.64
7.62
7.61
7.24
7.23
7.22
7.19
2.47
O
OH
HO
Me
3b
2.01
1.00
1.01
1.01
2.05
3.07
f1 (ppm)

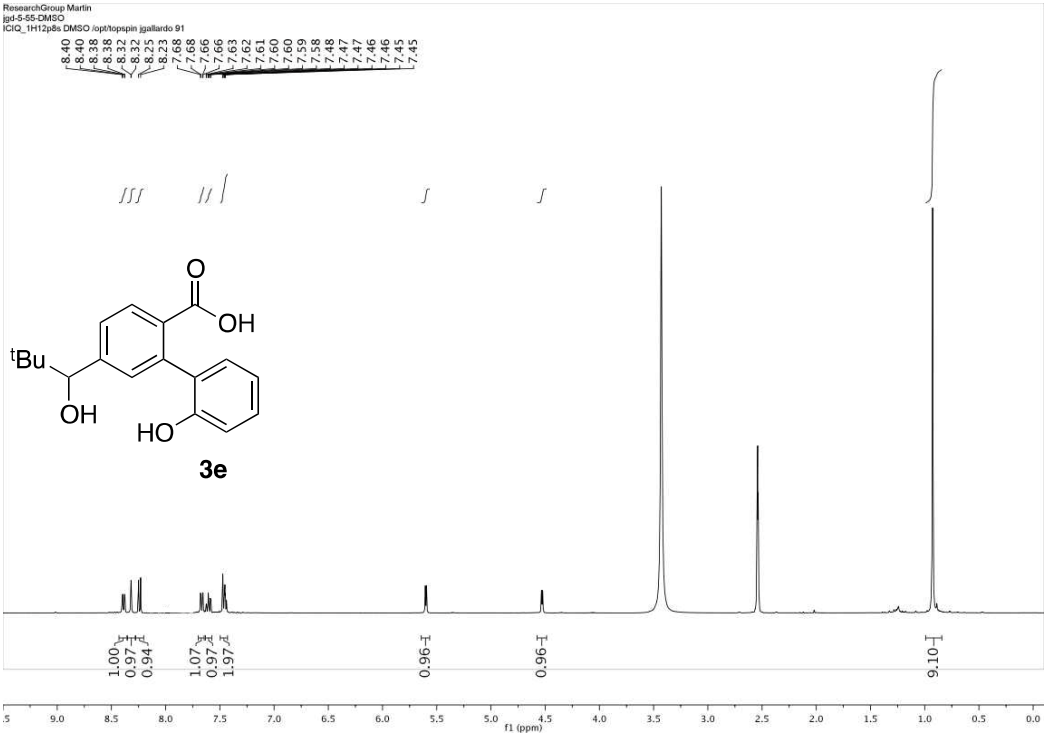

ResearchGroup Martin
jgd-5-52
ICIQ_13C{1H}512s DMSO /opt/topspin jgallardo 57
161.29
151.61
142.08
136.23
135.41
130.62
129.74
126.68
124.32
123.25
121.10
118.16
116.00
21.80
O
OH
HO
Me
3b
f1 (ppm)

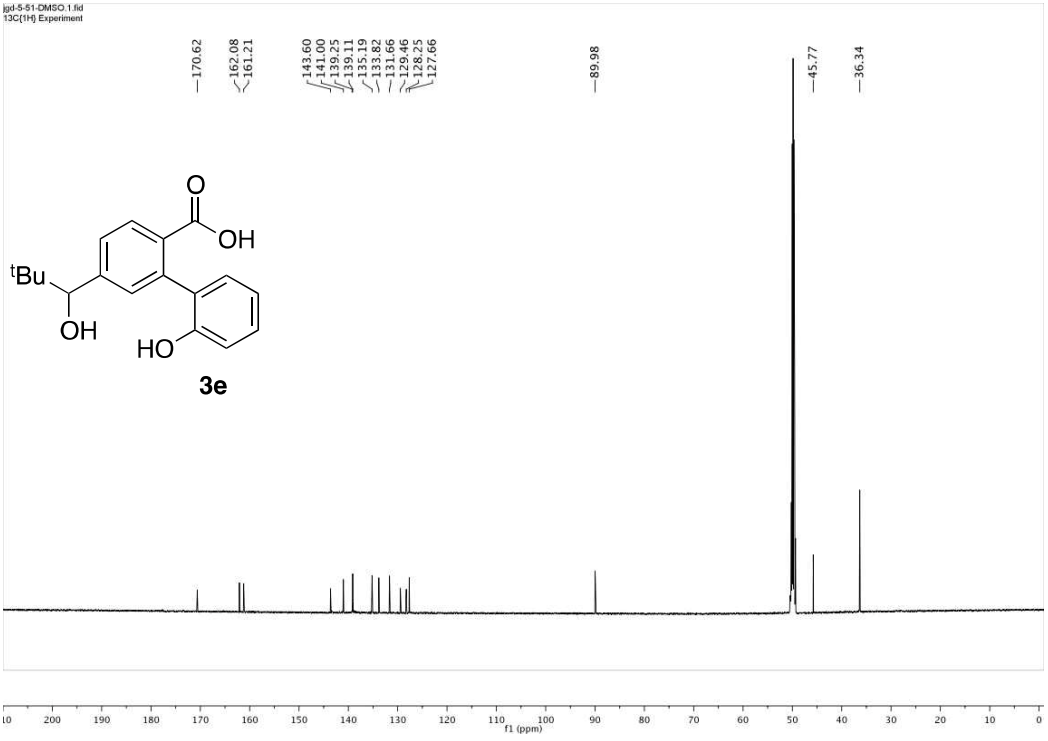

ResearchGroup Martin
jgd-5-45-01
ICIQ_1H12p8s DMSO /opt/topspin jgallardo 111
8.53
8.52
8.51
8.50
8.40
8.40
8.40
8.39
8.38
8.38
8.38
8.37
8.35
8.35
8.33
8.33
O
OH
HO
3c
1.00
1.98
1.00
2.06
1.07
3.07
f1 (ppm)

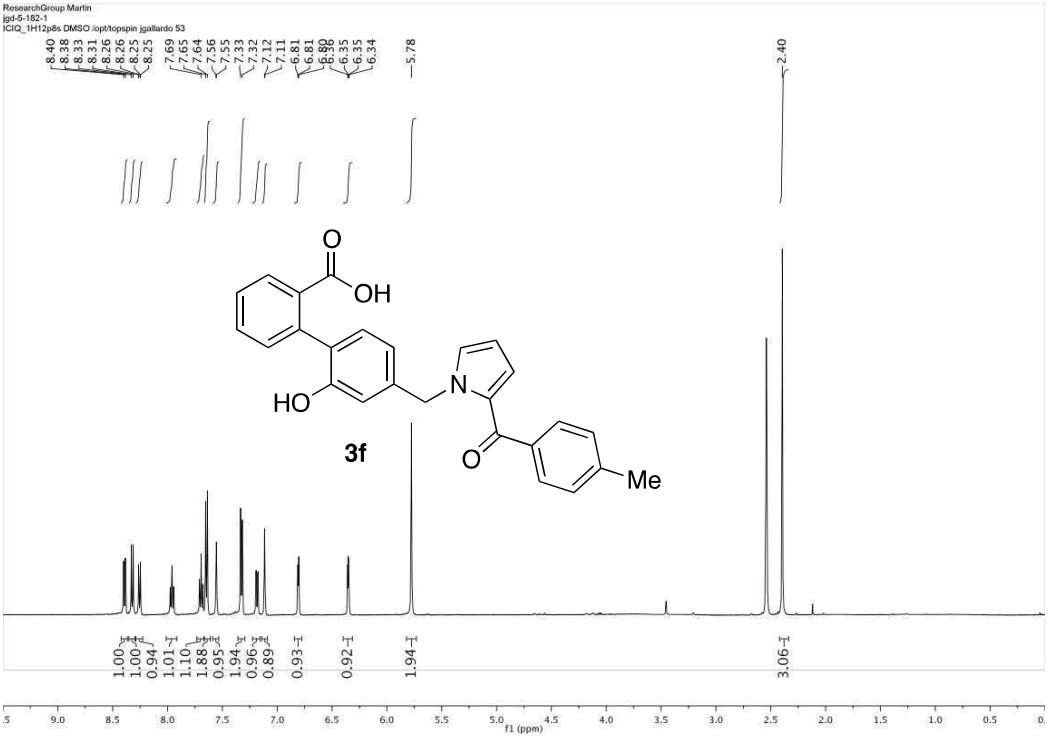

ResearchGroup Martin
jgd-5-45-01
ICIQ_13C{1H}512s DMSO /opt/topspin jgallardo 111
161.10
147.25
136.38
134.75
130.71
130.07
128.84
128.24
125.32
123.89
122.21
O
OH
HO
3c
f1 (ppm)

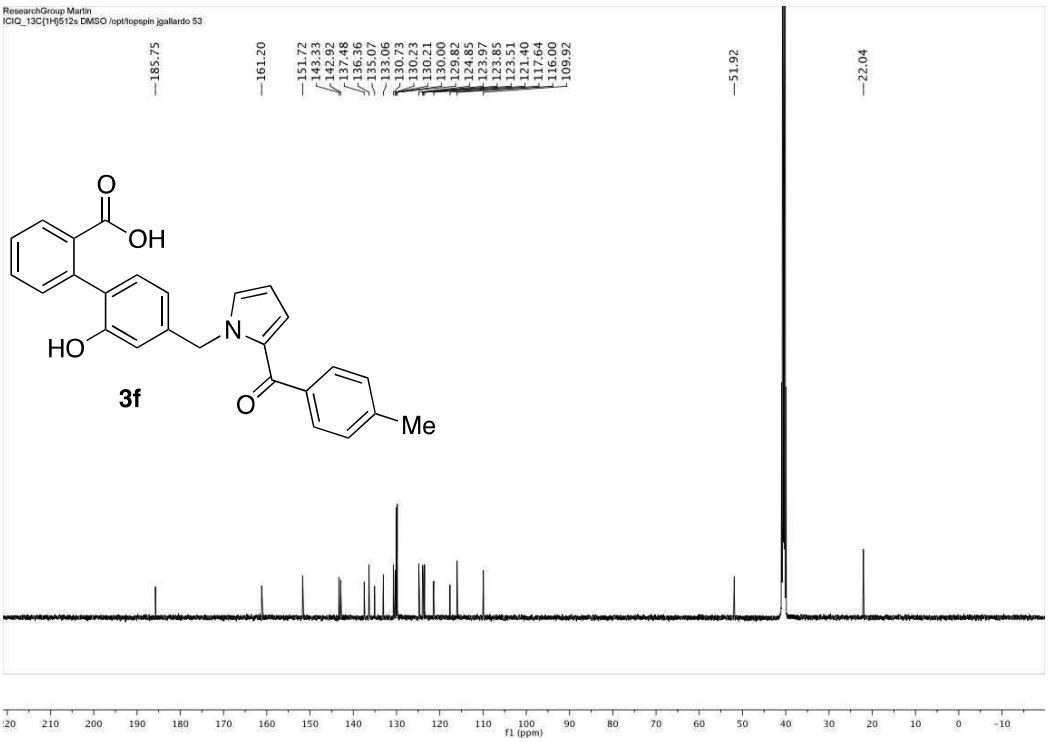

ResearchGroup Martin
jgd-5-46-DMSO
ICIQ_1H12p8s DMSO /opt/topspin jgallardo 103
8.39
8.37
8.31
8.30
8.29
8.28
7.68
7.67
7.57
7.56
7.55
7.55
7.55
7.54
7.54
7.53
7.53
7.52
7.51
7.43
7.43
7.41
MeO
O
OH
HO
3d
1.00
1.00
0.97
2.06
1.99
3.04
f1 (ppm)

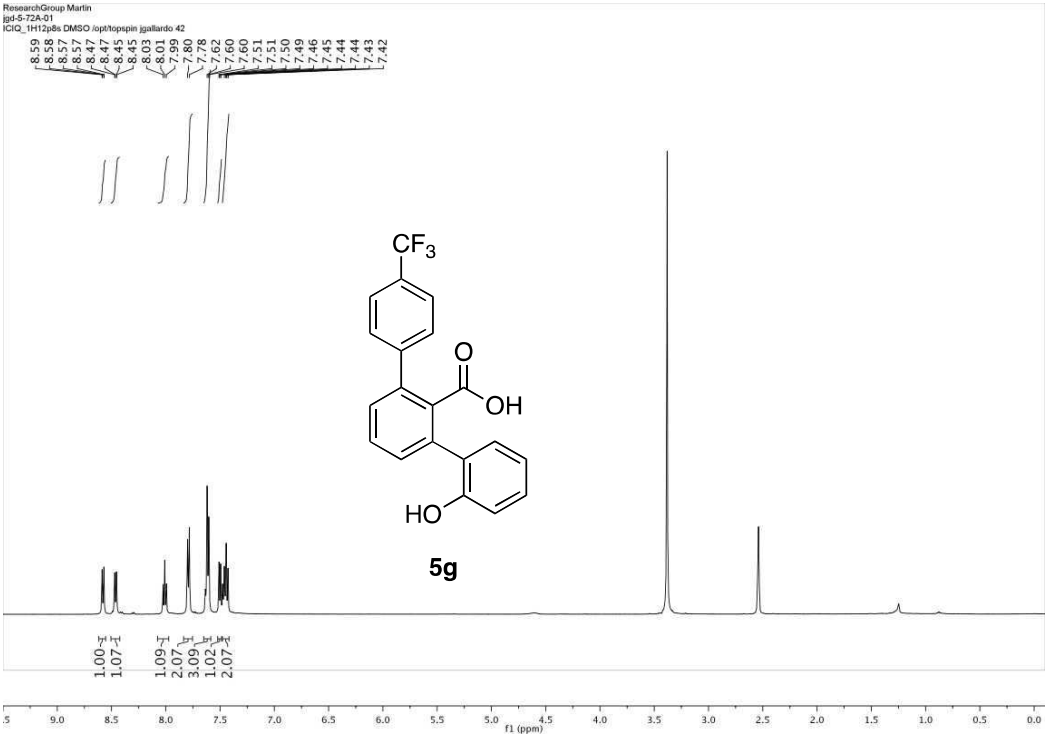

ResearchGroup Martin
jgd-5-46-DMSO
ICIQ_13C{1H}512s DMSO /opt/topspin jgallardo 103
161.03
160.62
150.76
130.51
128.45
125.68
125.47
124.61
123.96
122.78
118.70
117.97
112.09
56.62
MeO
O
OH
HO
3d
f1 (ppm)

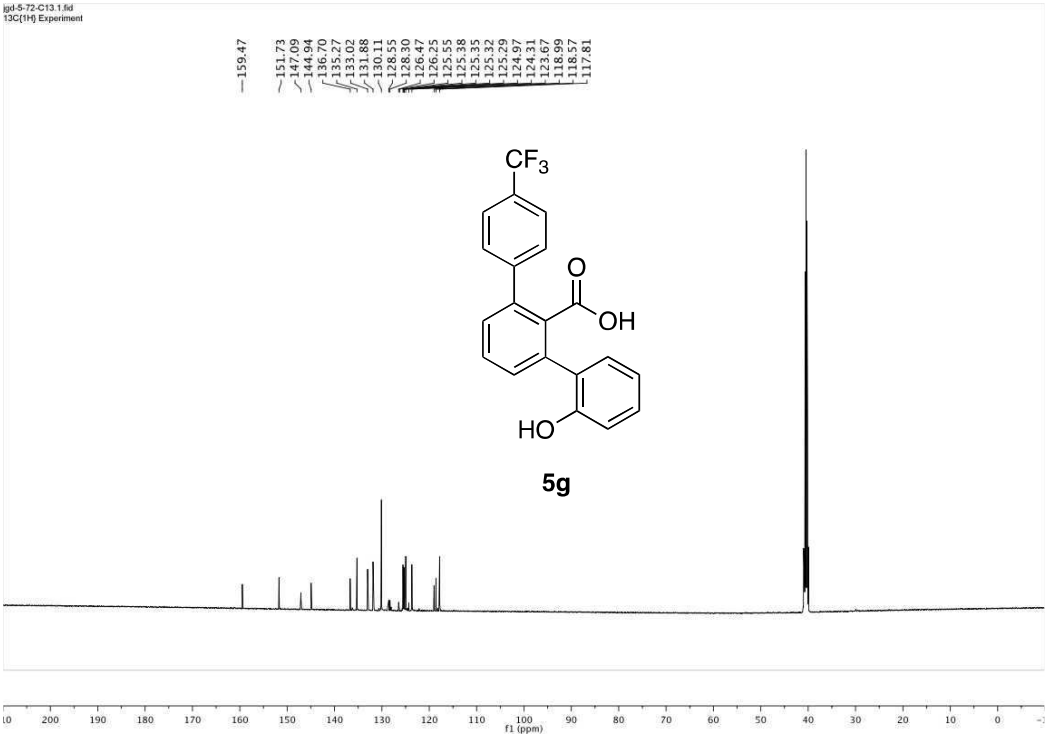

ResearchGroup Martin
jgd-5-55-DMSO
ICIQ_1H12p8s DMSO /opt/topspin jgallardo 91
8.40
8.40
8.38
8.38
8.32
8.32
8.25
8.23
7.68
7.68
7.66
7.66
7.63
7.62
7.61
7.60
7.60
7.59
7.58
7.48
7.47
7.47
7.46
7.46
7.45
7.45
O
OH
tBu
OH
HO
3e
1.00
0.97
0.94
1.07
0.97
1.97
0.96
0.96
9.10
f1 (ppm)

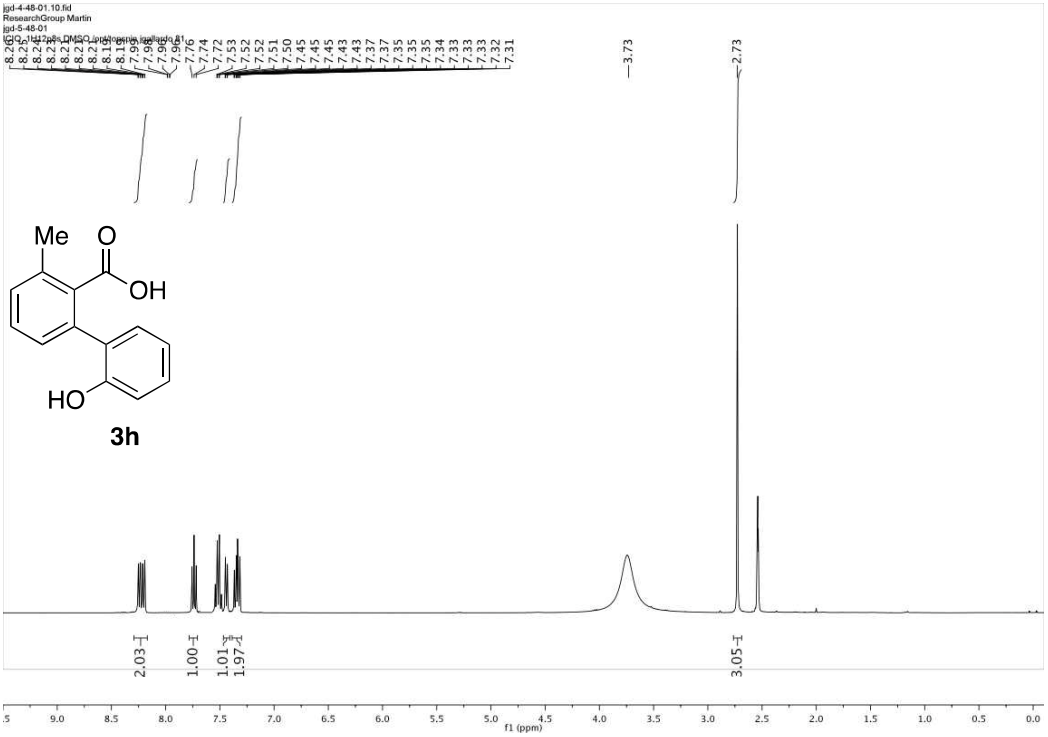

jgd-5-51-DMSO.1.fid
13C{1H} Experiment
170.62
162.08
161.21
143.60
141.00
139.25
139.11
135.19
133.82
131.66
129.46
128.25
127.66
89.98
45.77
36.34
O
OH
tBu
OH
HO
3e
f1 (ppm)

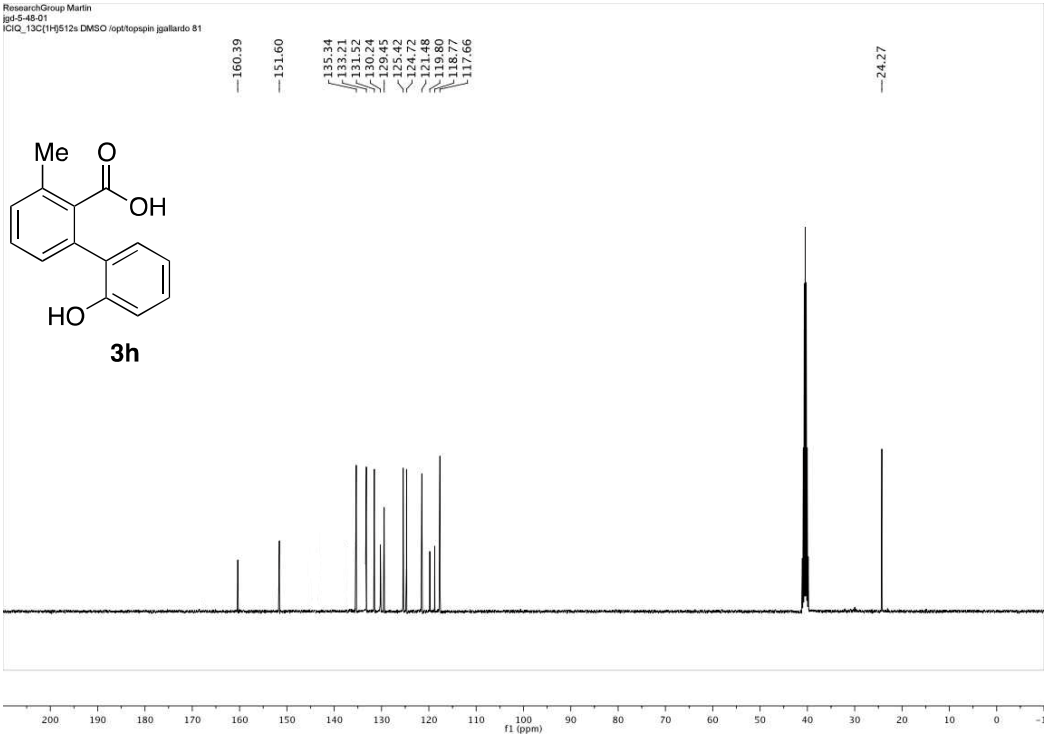

ResearchGroup Martin
jgd-5-182-1
ICIQ_1H12p8s DMSO /opt/topspin jgallardo 53
8.40
8.38
8.33
8.31
8.26
8.26
8.25
8.25
7.69
7.65
7.64
7.56
7.55
7.33
7.32
7.12
7.11
6.81
6.81
6.80
6.35
6.35
6.34
5.78
2.40
O
OH
HO
N
O
Me
3f
1.00
1.00
0.94
1.01
1.10
1.88
0.95
1.94
0.96
0.89
0.93
0.92
1.94
3.06
f1 (ppm)

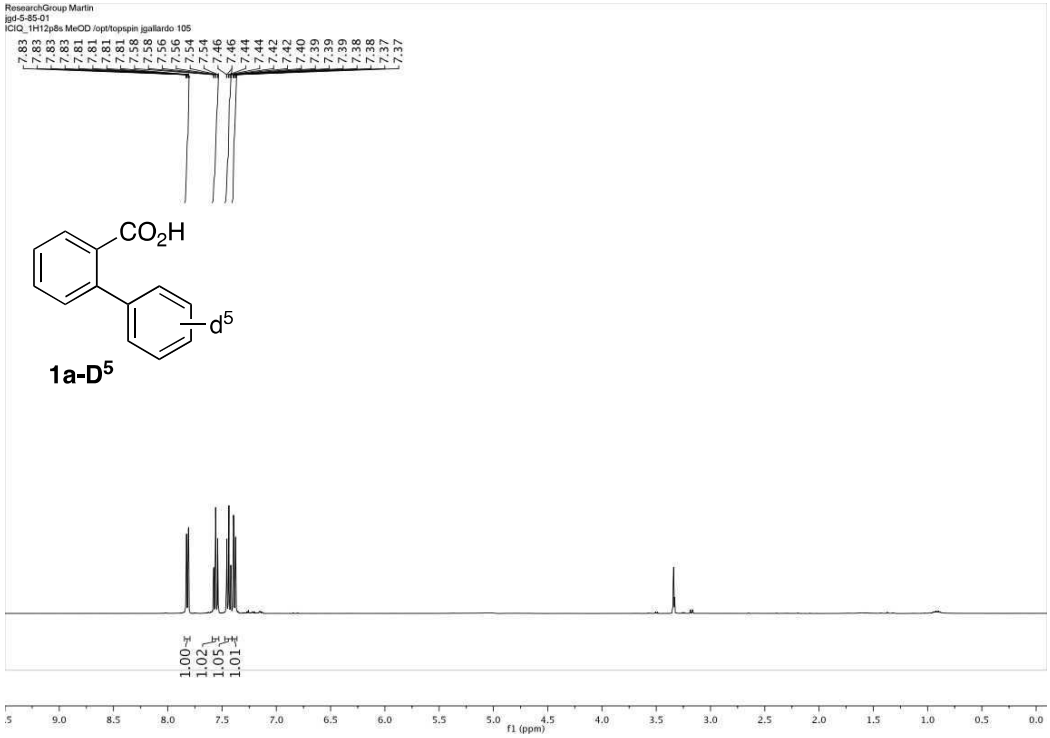

ResearchGroup Martin
ICIQ_13C{1H}512s DMSO /opt/topspin jgallardo 53
185.75
161.20
151.72
143.33
142.92
137.48
136.36
135.07
133.06
130.73
130.23
130.21
130.00
129.82
124.85
123.97
123.85
123.51
121.40
117.64
116.00
109.92
51.92
22.04
O
OH
HO
N
O
Me
3f
f1 (ppm)

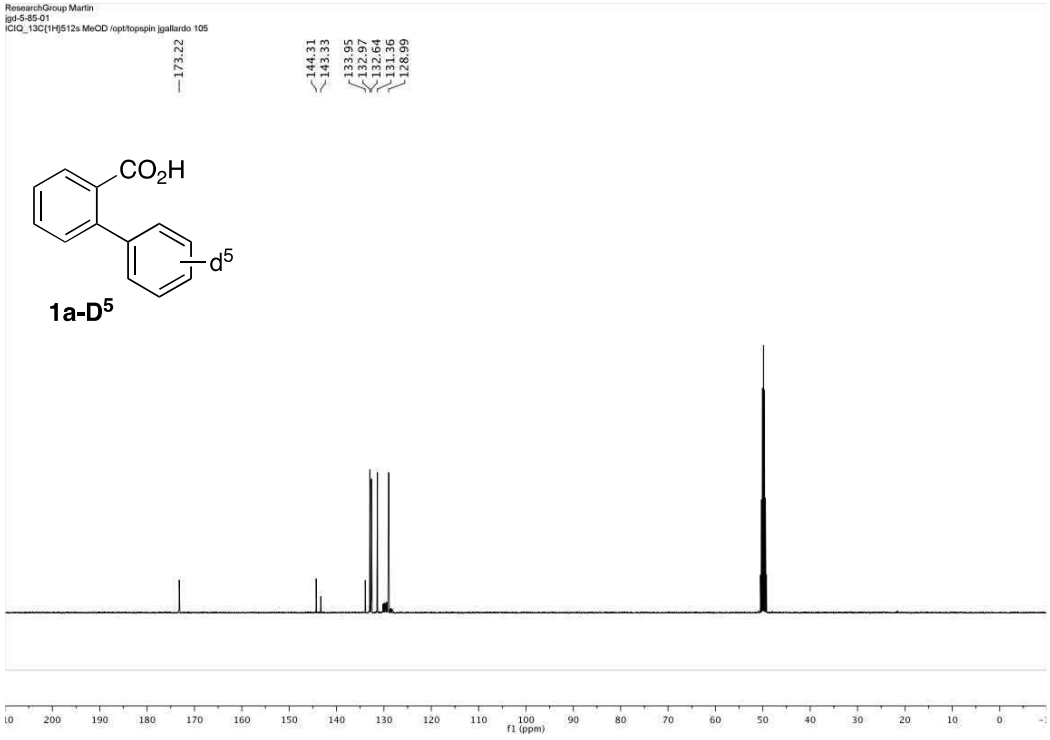

ResearchGroup Martin
jgd-5-72A-01
ICIQ_1H12p8s DMSO /opt/topspin jgallardo 42
8.59
8.58
8.57
8.57
8.47
8.47
8.45
8.45
8.03
8.01
7.99
7.80
7.78
7.62
7.60
7.60
7.51
7.51
7.50
7.49
7.46
7.45
7.44
7.44
7.43
7.42
CF3
O
OH
HO
5g
1.00
1.07
1.09
2.07
3.09
1.02
2.07
f1 (ppm)

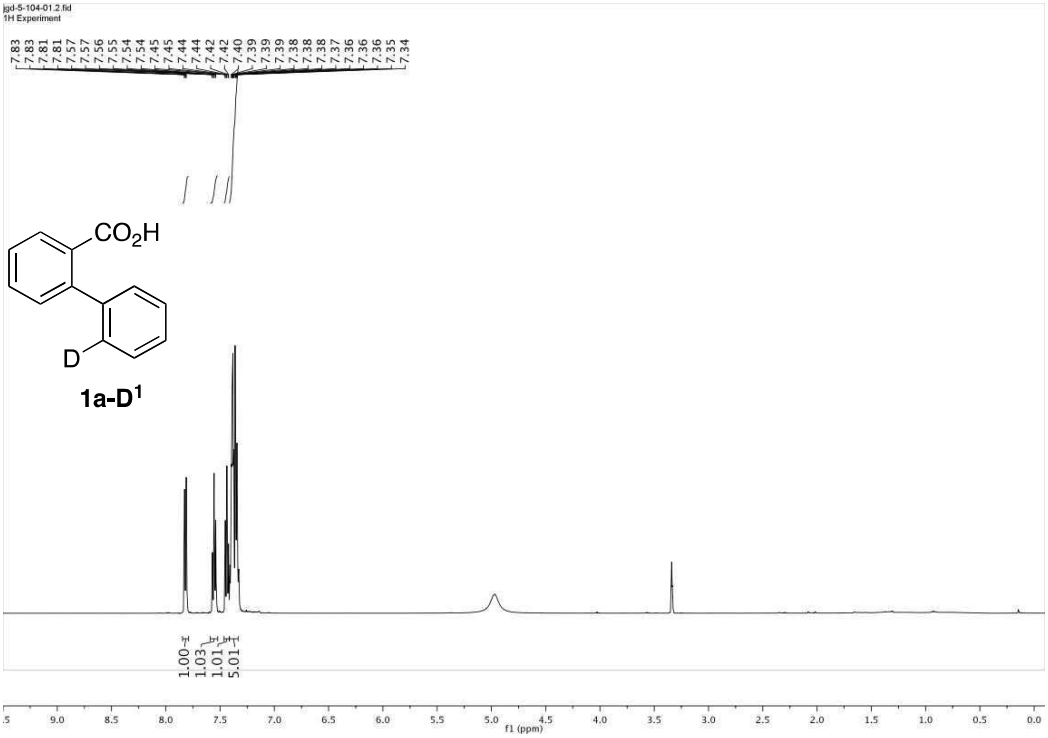

jgd-5-72-C13.1.fid
13C{1H} Experiment
159.47
151.73
147.09
144.94
136.70
135.27
133.02
131.88
130.11
128.55
128.30
126.47
126.25
125.55
125.38
125.35
125.32
125.29
124.97
124.31
123.67
118.99
118.57
117.81
CF3
O
OH
HO
5g
f1 (ppm)

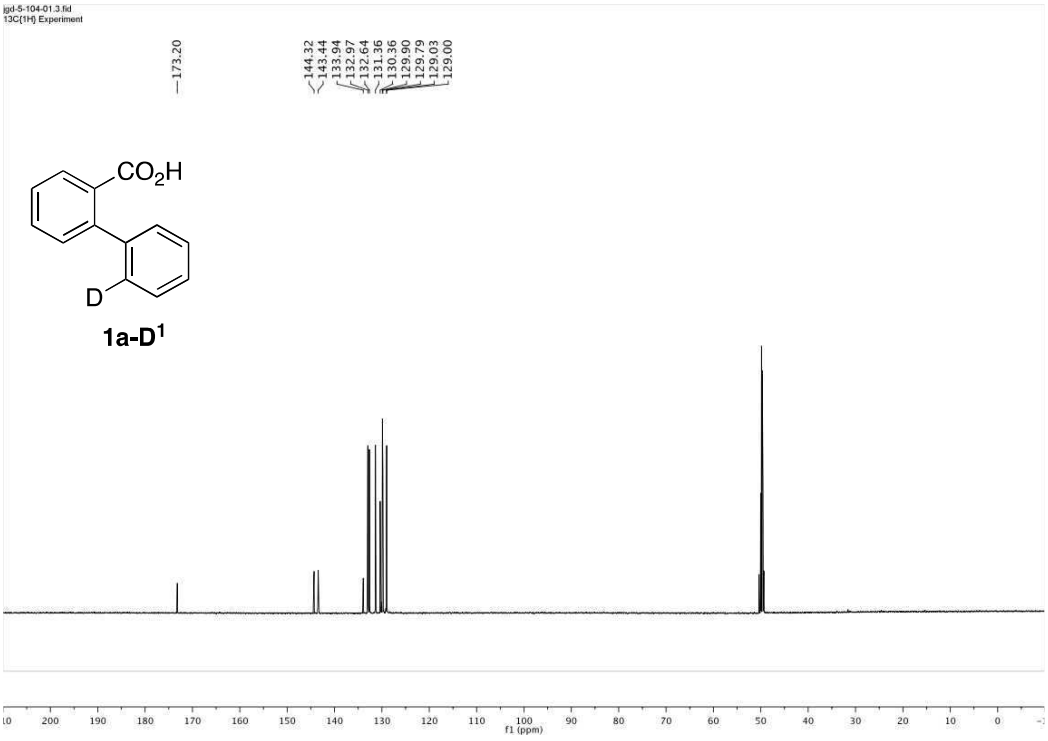

Me
O
OH
HO
3h
3.73
2.73
2.03
1.00
1.01
1.97
3.05
f1 (ppm)


Me
O
OH
HO
3h
160.39
151.60
135.34
133.21
131.52
130.24
129.45
125.42
124.72
121.48
119.80
118.77
117.66
24.27
f1 (ppm)


ResearchGroup Martin
jgd-5-85-01
ICIQ_1H12p8s MeOD /opt/topspin jgallardo 105
7.83
7.83
7.83
7.83
7.81
7.81
7.81
7.81
7.58
7.58
7.56
7.56
7.54
7.54
7.46
7.46
7.44
7.44
7.42
7.42
7.40
7.39
7.39
7.39
7.38
7.38
7.37
7.37
CO2H
d5
1a-D5
1.00
1.02
1.05
1.01
f1 (ppm)


ResearchGroup Martin
jgd-5-85-01
ICIQ_13C{1H}512s MeOD /opt/topspin jgallardo 105
173.22
144.31
143.33
133.95
132.97
132.64
131.36
128.99
CO2H
d5
1a-D5
f1 (ppm)


gd-5-104-01.2.fid
1H Experiment
7.83
7.83
7.81
7.81
7.57
7.56
7.55
7.54
7.54
7.45
7.44
7.42
7.42
7.40
7.39
7.39
7.38
7.38
7.38
7.37
7.36
7.36
7.35
7.34
CO2H
D
1a-D1
1.00
1.03
1.01
5.01
f1 (ppm)


gd-5-104-01.3.fid
13C{1H} Experiment
173.20
144.32
143.44
133.94
132.97
132.64
131.36
130.36
129.90
129.79
129.03
129.00
CO2H
D
1a-D1
f1 (ppm)