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Supporting Information

Palladium-catalyzed cross-dehydrogenative coupling of maleimides with simple arenes: A fast track to highly substituted maleimides

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1 General remarks: The following includes general experimental procedures, specific details for representative reactions, and isolation and spectroscopic information for the compounds. All reagents and metal catalysts were commercially available and used as received. The reactions were carried out in an oil bath using Microwave Vials (2-5 ml). ^{1}H and ^{13}C NMR spectra were recorded at room temperature on 400, 500 and 100, 125 MHz spectrometers respectively, using $CDCl_3$ as the NMR solvent. ^{1}H NMR spectra are referenced to tetramethylsilane (0.00 ppm) and ^{13}C NMR spectra are referenced from the solvent central peak (77.23 ppm). Chemical shifts are given in ppm.

2 Direct diarylation of maleimides

2-1 Screening of reaction conditions

Entry	[Pd]	Base	L	[O]	Solvent	T (°C)	t (h)	Yield (%)
1[b]	$Pd(OAc)_2$		Py	AgOAc	AcOH	100	24	0
2[b]	$Pd(OAc)_2$		Py	$K_2S_2O_8$	AcOH	100	24	0
3[b]	$Pd(OAc)_2$		Py	TBHP	AcOH	100	24	0
4[b]	$Pd(OAc)_2$		Py	$K_2S_2O_8$	AcOH	100	24	0
5[b]	$Pd(OAc)_2$		Bipy	$K_2S_2O_8$	AcOH	100	24	0
6[b]	$Pd(OAc)_2$		Phen	$K_2S_2O_8$	AcOH	100	24	0
7[c]	$Pd(OAc)_2$		Phen	$K_2S_2O_8$	AcOH	100	24	0
8[c]	$Pd(OAc)_2$			$K_2S_2O_8$	AcOH	100	24	0
9[d]	$Pd(OAc)_2$			$K_2S_2O_8$	TFA	100	24	10
10[e]	$Pd(OAc)_2$			AgOAc	TFA /PivOH	110	24	45
11[f]	$Pd(OAc)_2$			AgOAc	TFA /PivOH	110	24	30
12	**$Pd(OAc)_2$**			**AgOAc**	**TFA /PivOH**	**110**	**24**	**54**
13	$Pd(OAc)_2$			AgOAc	TFA /PivOH	130	24	51
14	$Pd(OAc)_2$			AgOAc	TFA /PivOH	110	36	54
15[g]	$Pd(OAc)_2$			AgOAc	TFA /PivOH	110	24	22
16	$Pd(OAc)_2$			AgOAc	TFA	110	24	35
17	$Pd(OAc)_2$			AgOAc	PivOH	110	24	trace
18	$Pd(OAc)_2$			$K_2S_2O_8$	TFA /PivOH	110	24	43
19	$Pd(OAc)_2$			$Cu(OAc)_2$	TFA /PivOH	110	24	38
20	$Pd(OAc)_2$			BQ	TFA /PivOH	110	24	0
21	$Pd(OAc)_2$			$AgNO_3$	TFA /PivOH	110	24	0
22	$Pd(OAc)_2$			Ag_2O	TFA /PivOH	110	24	52
23	$Pd(OAc)_2$			Ag_2CO_3	TFA /PivOH	110	24	50
24	$Pd(OAc)_2$			BP	TFA /PivOH	110	24	16
25	$Pd(OAc)_2$			TBP	TFA /PivOH	110	24	trace
26	$Pd(OAc)_2$			TBHP	TFA /PivOH	110	24	0
27	$Pd(OAc)_2$			$CuBr_2$	TFA /PivOH	110	24	0
28	$Pd(OAc)_2$			$CuCl_2$	TFA /PivOH	110	24	0
29[h]	$Pd(OAc)_2$		TFAA	AgOAc	TFA /PivOH	120	24	?
30[h]	$Pd(OAc)_2$		TFAA	$K_2S_2O_8$	TFA /PivOH	120	24	0
31[i]	$Pd(OAc)_2$			AgOAc	TFA /PivOH /ACN	110	24	0
32[i]	$Pd(OAc)_2$			AgOAc	TFA /PivOH/DCE	110	24	20
33[i]	$Pd(OAc)_2$			AgOAc	TFA /PivOH/dioxane	110	24	0
34	$Pd(TFA)_2$			AgOAc	TFA /PivOH	110	24	50
35	$PdCl_2$			AgOAc	TFA /PivOH	110	24	0
36	$Pd(dba)_2$			AgOAc	TFA /PivOH	110	24	0
37	$Pd(acac)_2$			AgOAc	TFA /PivOH	110	24	40
38[j]	$Pd(OAc)_2$		Phen.	AgOAc	TFA /PivOH	110	24	52
39[j]	$Pd(OAc)_2$		BiPy	AgOAc	TFA /PivOH	110	24	35
40[j]	$Pd(OAc)_2$		PPh_3	AgOAc	TFA /PivOH	110	24	50
41[j]	$Pd(OAc)_2$		DMEDA	AgOAc	TFA /PivOH	110	24	38
42[j]	$Pd(OAc)_2$		TBAI	AgOAc	TFA /PivOH	110	24	25
43[k]	$Pd(OAc)_2$		Phen.	$AgNO_3$	DMSO	110	22	0
44[l]	$Pd(OAc)_2$	K_2CO_3		AgOAc	TFA/PivOH	130	24	29

Table S1. Screening of the reaction conditions

[a]Reaction conditions: N-Methylmaleimide **1a** (0.1 mmol), toluene **2a** (4 mmol), catalyst (20 mol%), oxidant (0.3 mmol), TFA (1 mmol), PivOH (0.6 mmol), 24 h. [b]Catalyst (5 mol%), ligand (5 mol%), oxidant (0.1 mmol), solvent (1mL). [c]Likewise b but solvent (0.1 mL), [d]Catalyst (10 mol%), TFA (0.5 mmol). [e]Catalyst (5 mol%), [f]Catalyst (10

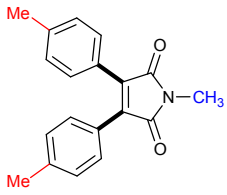
mol%). [g]Oxidant (1.5 mmol), [h]Catalyst (10 mol%), oxidant (0.1 mmol), TFAA (0.1 mmol), [i]TFA /PivOH /solvent (0.08, 0.06, 0.2 mL), [j]Ligand (20 mol%). [k]Catalyst (10 mol%), ligand (20 mol%), oxidant (0.2 mmol) , solvent (0.15 mL). [l]Catalyst (10 mol%), base (0.1 mmol), oxidant (0.4 mmol).

2-2 General experimental procedure

A vial equipped with a stir bar was charged with N-substituted maleimide (0.1 mmol, 1 equiv), arene (40 equiv), AgOAc (3 equiv), $Pd(OAc)_2$ (20 mol%), TFA (10 equiv), PivOH (6 equiv) and capped. The resulting mixture was heated in an oil bath at 110 °C for 24 h, cooled then filtered through a short plug of silica. Removal of the solvent gave a crude mixture which was purified by flash column chromatography (hexane/EtOAc gradient).

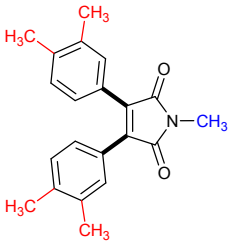
2-3 Compound characterization data

1-methyl-3,4-di-p-tolyl-1H-pyrrole-2,5-dione **(3a)**



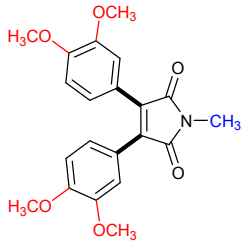
Yellow solid (54%); mp 178-180 °C; R_f = 0.45 on silica gel (hexane/EtOAc 9:1); ^{1}H NMR (250 MHz, $CDCl_3$) δ 2.37 (s, 6H), 3.15 (s, 3H), 7.17 (d, *J* = 8.2 Hz, 4H), 7.39 (d, *J* = 8.2 Hz, 4H); ^{13}C NMR (62.5 MHz, $CDCl_3$) δ 140.0, 129.8, 129.3, 126.0, 24.2, 21.5. MS *m/z* (%) 291 (M•+, 100), 276 (25), 233 (21), 219 (50), 206 (35), 191 (25). Anal. Calcd for $C_{19}H_{17}NO_2$: C, 78.33; H, 5.88; N, 4.81. Found: C, 78.61; H, 6.00; N, 5.02.

3,4-bis(3,4-dimethylphenyl)-1-methyl-1H-pyrrole-2,5-dione **(3b)**



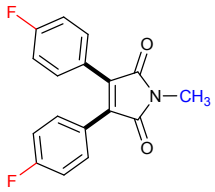
Yellow solid (63%); mp 149-150 °C; R_f = 0.46 on silica gel (hexane/EtOAc 9:1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.24 (s, 6H), 2.28 (s, 6H), 3.15 (s, 3H), 7.07-7.18 (m, 4H), 7.33 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.3, 138.6, 136.8, 135.7, 130.8, 129.7, 127.3, 126.4, 29.7, 24.2, 19.8. Anal. Calcd for $C_{21}H_{21}NO_2$: C, 78.97; H, 6.63; N, 4.39. Found: C, 79.28; H, 6.72; N, 4.54.

3,4-bis(3,4-dimethoxyphenyl)-1-methyl-1H-pyrrole-2,5-dione **(3c)**



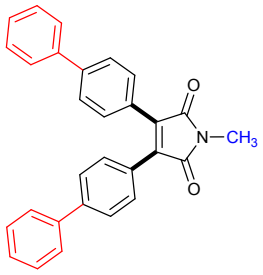
Orange solid (66%); mp 172-173 °C; R_f = 0.61 on silica gel (hexane/EtOAc 1:1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.14 (s, 3H), 3.72 (s, 6H), 3.91 (s, 6H), 6.87 (d, J = 8.2 Hz, 2H), 7.06 (d, J = 2.2 Hz, 2H), 7.21 (dd, J = 8.2, 2.2 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 23.1, 54.8, 54.9, 110.0, 111.6, 120.5, 122.5, 133.3, 147.7, 149.3, 170.3. Anal. Calcd for $C_{21}H_{21}NO_6$: C, 65.79; H, 5.52; N, 3.65. Found: C, 65.98; H, 5.62; N, 3.79.

3,4-bis(4-fluorophenyl)-1-methyl-1H-pyrrole-2,5-dione **(3d)**



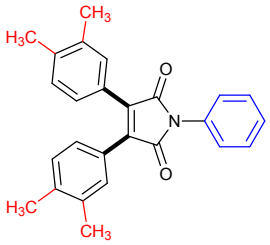
Yellow solid (21%); mp 176-178 °C; R_f = 0.33 on silica gel (hexane/EtOAc 9:1); ^{1}H NMR (300 MHz, $CDCl_3$) δ 3.17 (s, 3H), 7.05-7.11 (m, 4H), 7.46-7.51 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.6, 162.5 (d, $^1J_{CF}$ = 250.6 Hz), 134.1, 130.9 (d, $^3J_{CF}$ = 8.4 Hz), 126.5, 114.9 (d, $^2J_{CF}$ = 21.8 Hz), 23.3. Anal. Calcd for $C_{22}H_{15}NO_2$: C, 81.21; H, 4.65; N, 4.30. Found: C, 81.40; H, 4.74; N, 4.42.

3,4-di([1,1'-biphenyl]-4-yl)-1-methyl-1H-pyrrole-2,5-dione **(3e)**



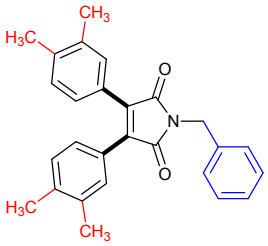
Yellow solid (51%); R_f = 0.60 on silica gel (hexane/EtOAc 8.5:1.5); ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.21 (s, 3H), 7.32-7.54 (m, 10H); 7.60-7.66 (m, 8H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 23.3, 126.1, 127.8, 127.9, 127.9, 129.3, 139.1, 139.1, 141.5, 141.6, 169.9. Anal. Calcd for $C_{22}H_{15}NO_2$: C, 81.21; H, 4.65; N, 4.30. Found: C, 81.40; H, 4.74; N, 4.43.

3,4-bis(3,4-dimethylphenyl)-1-phenyl-1H-pyrrole-2,5-dione **(3f)**



Yellow solid (78%); mp 181-182 °C; R_f = 0.52 on silica gel (hexane/EtOAc 9:1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.26 (s, 6H), 2.30 (s, 6H), 7.11 (d, 2H), 7.24 (dd, *J* = 8.0, 1.5 Hz 2H), 7.35-7.43 (m, 3H), 7.45-7.54 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 18.8, 28.7, 125.1, 125.2, 126.4, 126.6, 128.0, 128.8, 130.0, 131.0, 134.5, 135.8, 137.9, 169.0. Anal. Calcd for $C_{26}H_{23}NO_2$: C, 81.86; H, 6.08; N, 3.67. Found: C, 82.09; H, 6.18; N, 3.83.

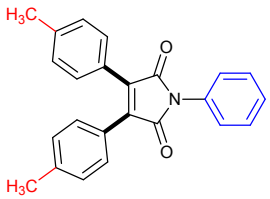
1-benzyl-3,4-bis(3,4-dimethylphenyl)-1H-pyrrole-2,5-dione **(3g)**



Yellow solid (56%); mp 109-110 °C; R_f = 0.55 on silica gel (hexane/EtOAc 9:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.24 (s, 6H), 2.28 (s, 6H), 4.81 (s, 2H), 7.05-7.11 (m, 2H), 7.18 (dd, *J* = 7.9, 1.4

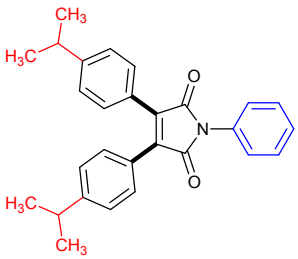
Hz, 2H), 7.29-7.38 (m, 5H), 7.46-7.50 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 19.7, 19.8, 41.8, 126.3, 127.3, 127.7, 128.6, 128.8, 129.7, 130.8, 135.4, 136.6, 136.7, 138.7, 170.8.

1-phenyl-3,4-di-p-tolyl-1H-pyrrole-2,5-dione **(3h)**



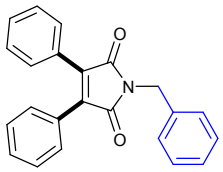
Orange solid (52%); mp 158-159°C; R_f = 0.50 on silica gel (hexane/EtOAc 9:1); ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.39 (s, 6H), 7.20 (d, *J* = 8.1 Hz, 4H), 7.45-7.56 (m, 9H); ^{13}C NMR (100 MHz, $CDCl_3$); δ 168.8, 139.2, 134.5, 130.9, 128.9, 128.7, 128.3, 128.0, 126.6, 125.1, 124.8, 20.5. Anal. Calcd for $C_{24}H_{19}NO_2$: C, 81.56; H, 5.42; N, 3.96. Found: C, 81.84; H, 5.56; N, 4.15.

3,4-bis(4-isopropylphenyl)-1-phenyl-1H-pyrrole-2,5-dione **(3i)**



Dark yellow solid (42%); R_f = 0.62 on silica gel (hexane/EtOAc 9:1); ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.17 (s, 6 H) 1.29 (s, 6 H) 2.82-2.99 (m, 2 H) 7.24 - 7.54 (m, 13 H). Anal. Calcd for $C_{28}H_{27}NO_2$: C, 82.12; H, 6.65; N, 3.42. Found: C, 82.34; H, 6.75; N, 3.58.

1-benzyl-3,4-diphenyl-1H-pyrrole-2,5-dione **(3j)**

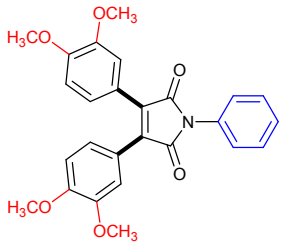


Green- yellow solid (40%); mp 159 -160 °C (Ref.[1] 132-134 °C); R_f = 0.50 on silica gel (hexane/EtOAc 9:1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.83 (s, 2H), 7.28-7.43 (m, 9H), 7.46-7.52 (m, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 42.1, 127.9, 128.6, 128.6, 128.7, 128.9, 129.9, 129.9, 136.2,

[1] H.-D. Xie, L. A. Ho, M. S. Truelove, B. Corry, S. G. Stewart, *J. Fluoresc.*, 2010, **20**, 1077.

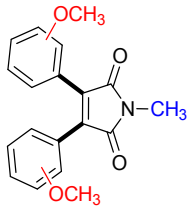
136.5, 170.5. Anal. Calcd for $C_{23}H_{17}NO_2$: C, 81.40; H, 5.05; N, 4.13. Found: C, 81.66; H, 5.19; N, 4.30.

3,4-bis(3,4-dimethoxyphenyl)-1-phenyl-1H-pyrrole-2,5-dione **(3k)**



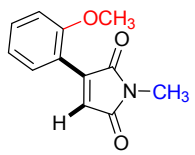
Yellow solid (72%); mp 187 °C; R_f = 0.65 on silica gel (hexane/EtOAc 1:1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.74 (s, 6H), 3.91 (s, 6H), 6.88 (d, *J* = 8.5 Hz, 2H), 7.14 (d, *J* = 1.7 Hz, 2H), 7.26 (dd, *J* = 8.2, 2.0 Hz, 2H), 7.34-7.41 (m, 1H), 7.43-7.50 (m, 4H); ^{13}C NMR (100MHz, $CDCl_3$) δ 54.8, 54.9, 109.9, 111.7, 120.2, 122.7, 125.2, 126.7, 128.1, 130.8, 133.1, 147.7, 149.5, 168.9. Anal. Calcd for $C_{26}H_{23}NO_6$: C, 70.10; H, 5.20; N, 3.14. Found: C, 70.44; H, 5.34; N, 3.33.

3,4-bis(2-methoxyphenyl)-1-methyl-1H-pyrrole-2,5-dione **(3m)**



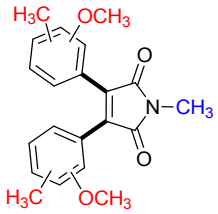
Orange solid (mixture of isomers). ^{1}H NMR (400MHz, $CDCl_3$) δ 3.15 (s, 3H), 3.44 (s, 1H), 3.55 (s, 2H), 3.81 (s, 2H), 3.84 (s, 2H), 6.82 (d, *J* = 8.78 Hz, 1H), 6.88 (d, *J* = 9.03 Hz, 1H), 6.93 (d, *J* = 8.28 Hz, 1H), 7.02 (t, *J* = 7.53 Hz, 1H), 7.37-7.44 (m, 1H), 7.46-7.52 (m, 2 H), 7.52-7.56 (m, 1H); ^{13}C NMR (100MHz, $CDCl_3$) δ 24.2, 55.0, 55.2, 55.3, 111.0, 111.5, 113.7, 114.1, 114.5, 120.3, 120.8, 120.9, 121.4, 130.4, 130.6, 130.9, 130.9, 131.1, 131.4, 137.1, 157.3, 160.7, 171.5.

3-(2-methoxyphenyl)-1-methyl-1H-pyrrole-2,5-dione **(3n)**



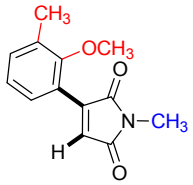
Yellow solid (33%); R_f = 0.43 on silica gel (hexane/EtOAc 8.5:1.5); ^{1}H NMR (400MHz, $CDCl_3$) δ 3.07 (s, 3H), 3.92 (s, 3H), 6.98 (d, *J* = 8.53 Hz, 1H), 7.05 (td, *J* = 7.53, 1.00 Hz, 1H), 7.10 (s, 1H), 7.42 (td, *J* = 7.91, 1.76 Hz, 1 H), 8.26 (dd, J = 7.78, 1.76 Hz, 1H); ^{13}C NMR (100MHz, $CDCl_3$) δ 171.7, 171.7, 159.4, 139.1, 131.9, 131.4, 127.5, 120.7, 117.9, 111.0, 55.5, 23.8. Anal. Calcd for $C_{12}H_{11}NO_3$: C, 66.35; H, 5.10; N, 6.45. Found: C, 66.72; H, 5.21; N, 6.66.

***3,4-bis(2-methoxy-3-methylphenyl)-1-methyl-1H-pyrrole-2,5-dione* (3o)**



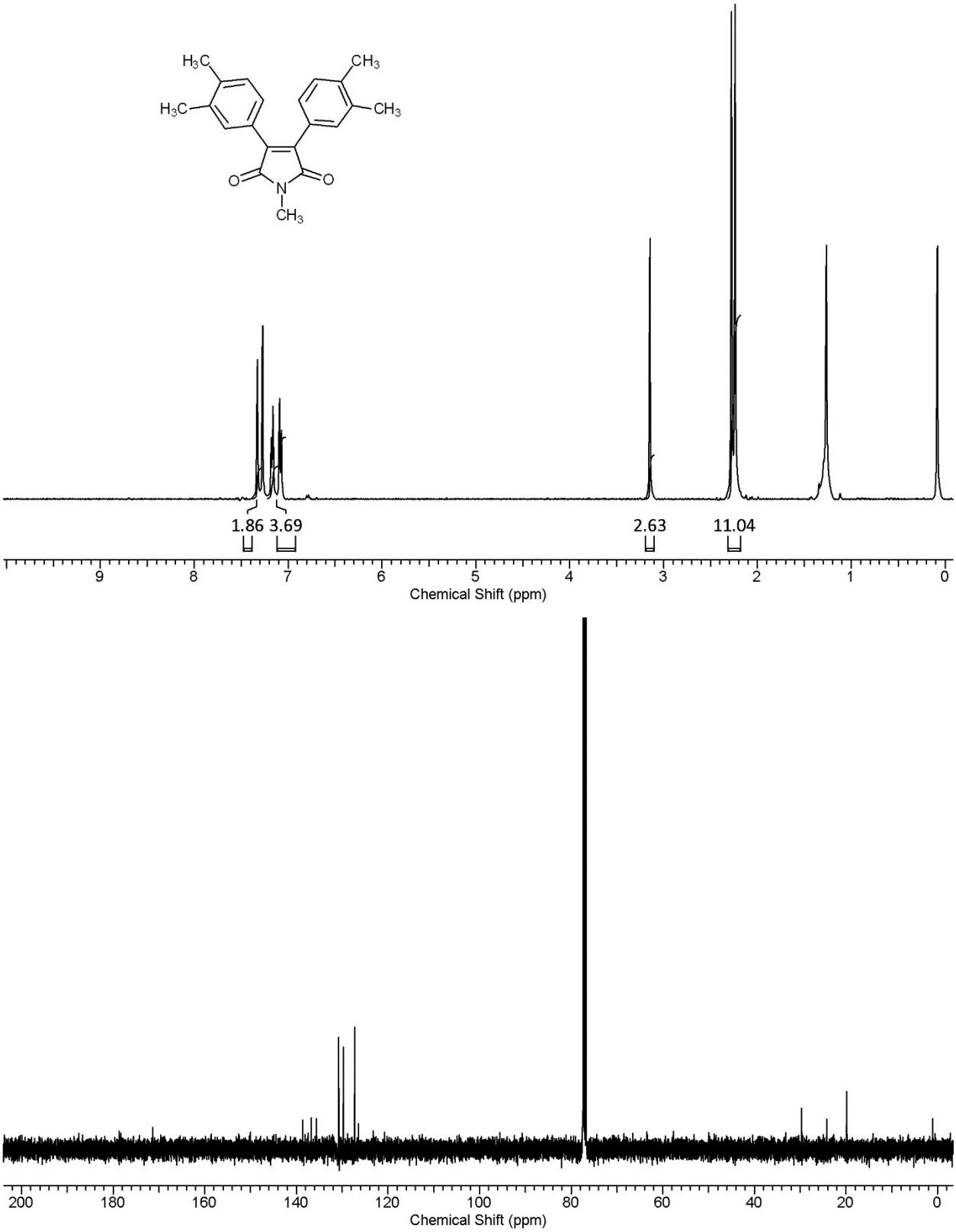
Yellow solid (41%); ^{1}H NMR (400MHz, $CDCl_3$) δ 2.17 (s, 6H), 3.13 (s, 3H), 3.85 (s, 6H), 6.79 (d, *J* = 8.28 Hz, 2H), 7.31-7.38 (m, 4H); ^{13}C NMR (100MHz, $CDCl_3$) δ 16.2, 24.1, 55.3, 109.8, 121.0, 126.8, 129.0, 131.9, 134.3, 158.9, 171.7.

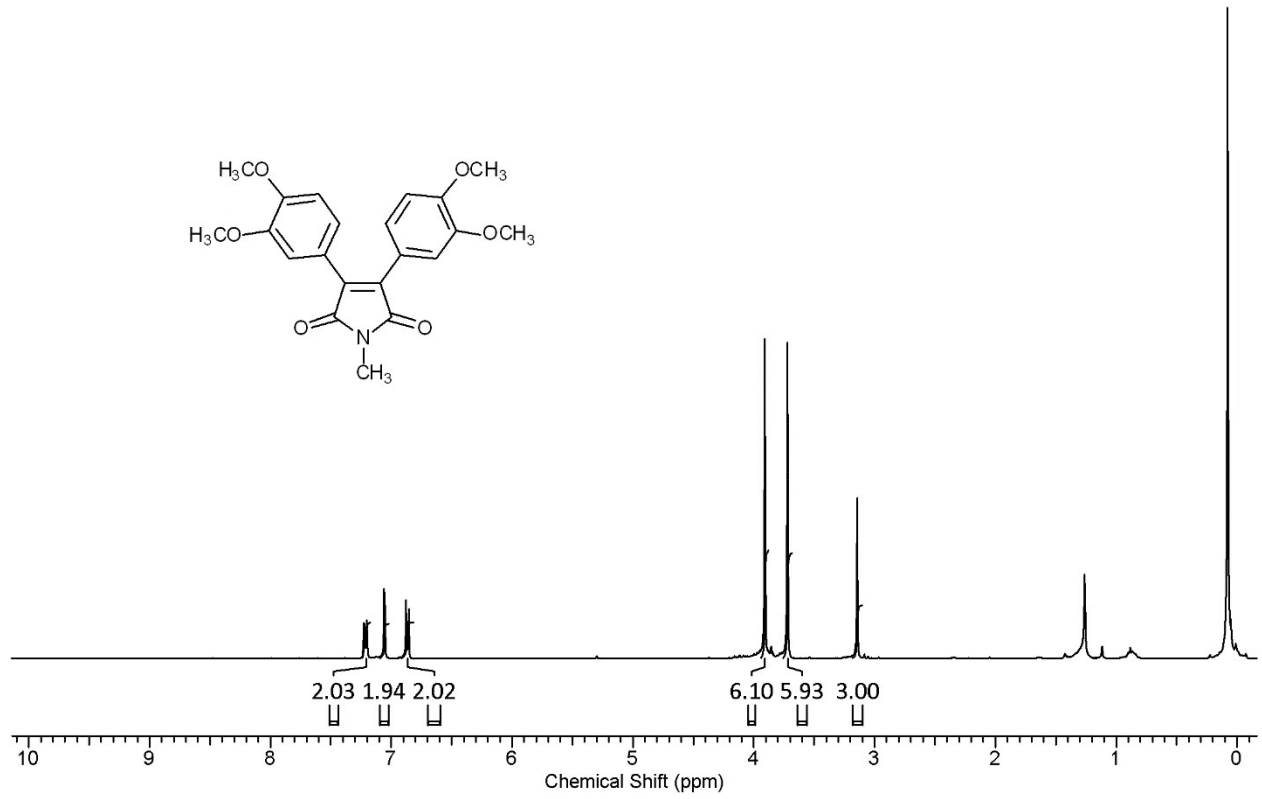
***3-(2-methoxy-3-methylphenyl)-1-methyl-1H-pyrrole-2,5-dione* (3p)**

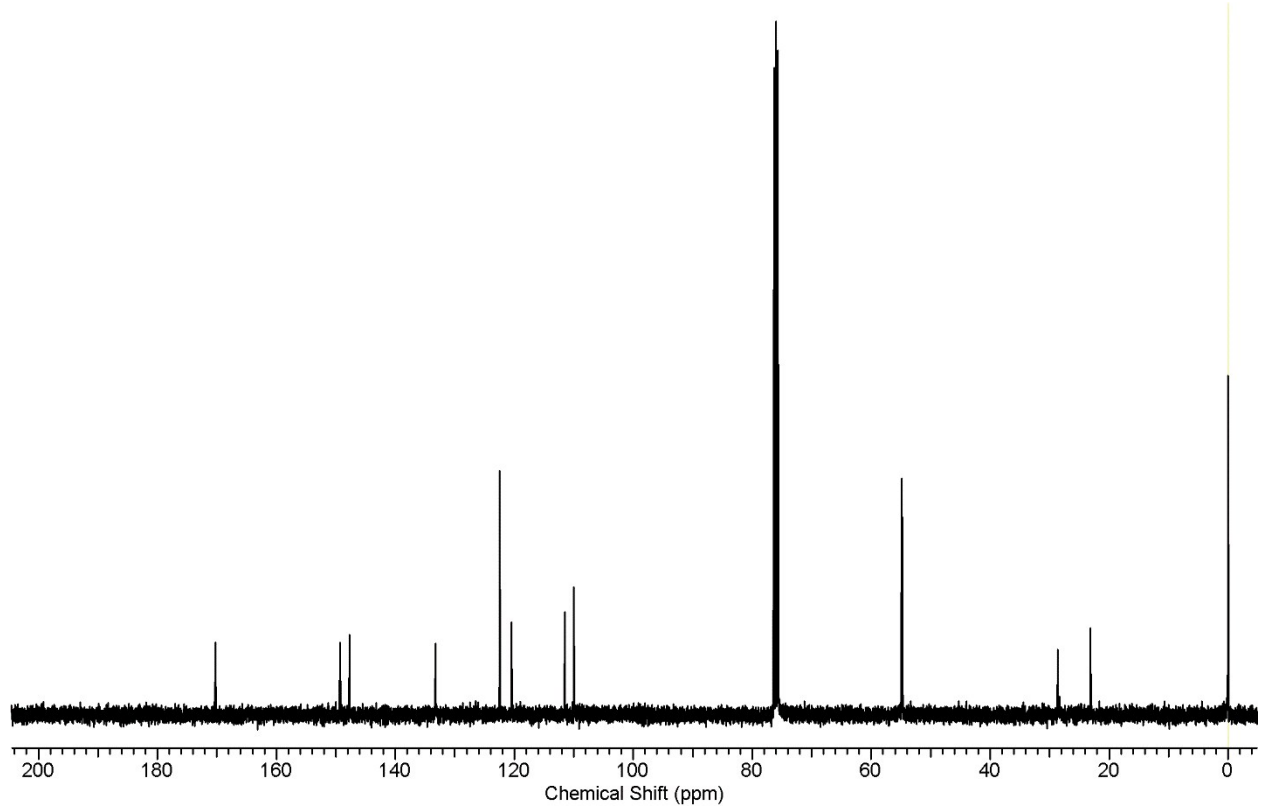


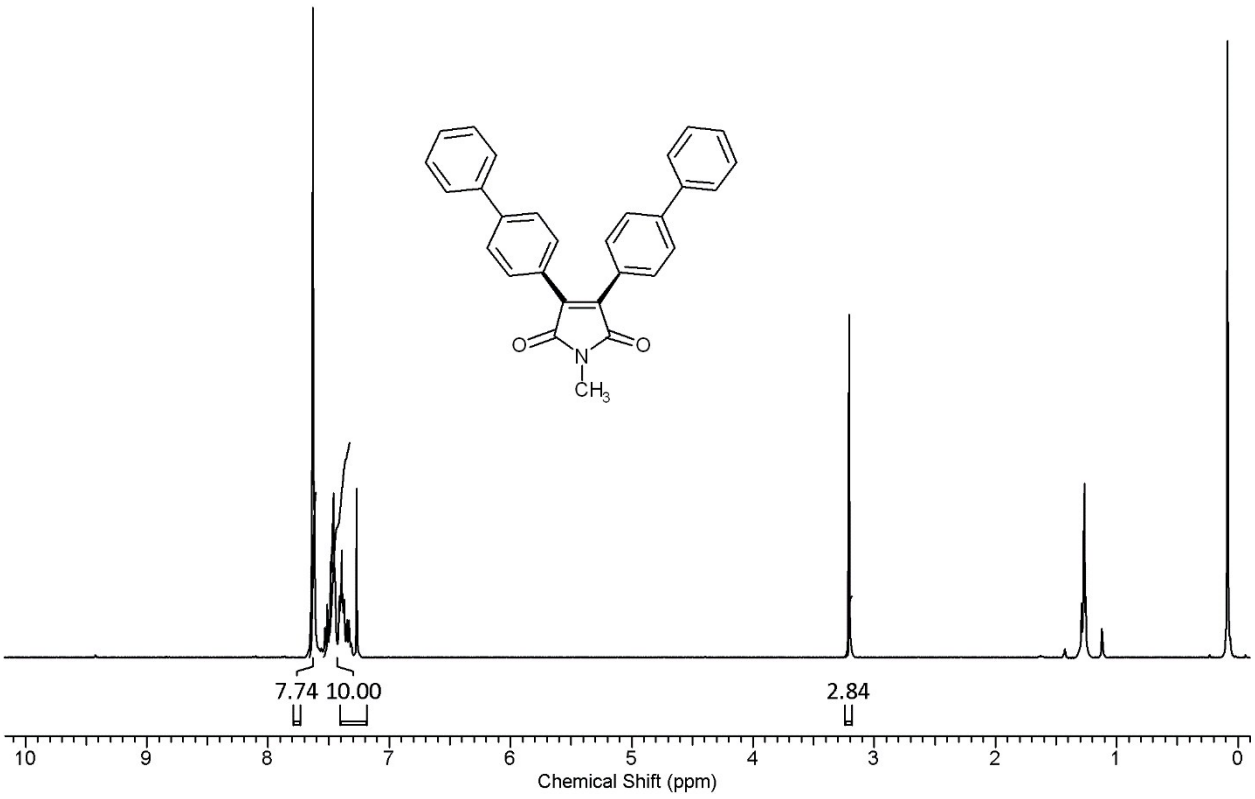
Yellow solid (39%,); mp 88-89 °C; R_f = 0.23 on silica gel (hexane/EtOAc 8.5:1.5); ^{1}H NMR (400MHz, $CDCl_3$) δ 2.26 (s, 3H), 3.07 (s, 3H), 3.89 (s, 3H), 6.58 (s, 1H), 6.88 (d, *J* = 8.5 Hz, 1H), 7.70 (d, *J* = 1.5 Hz, 1H), 7.90 (dd, *J* = 8.7, 2.13 Hz, 1H); ^{13}C NMR (100MHz, $CDCl_3$) δ 16.3, 23.7, 55.5, 110.0, 120.6, 121.0, 127.5, 128.3, 130.7, 143.7, 160.4, 170.9, 171.3. Anal. Calcd for $C_{13}H_{13}NO_3$: C, 67.52; H, 5.67; N, 6.06. Found: C, 67.85; H, 5.81; N, 6.26.

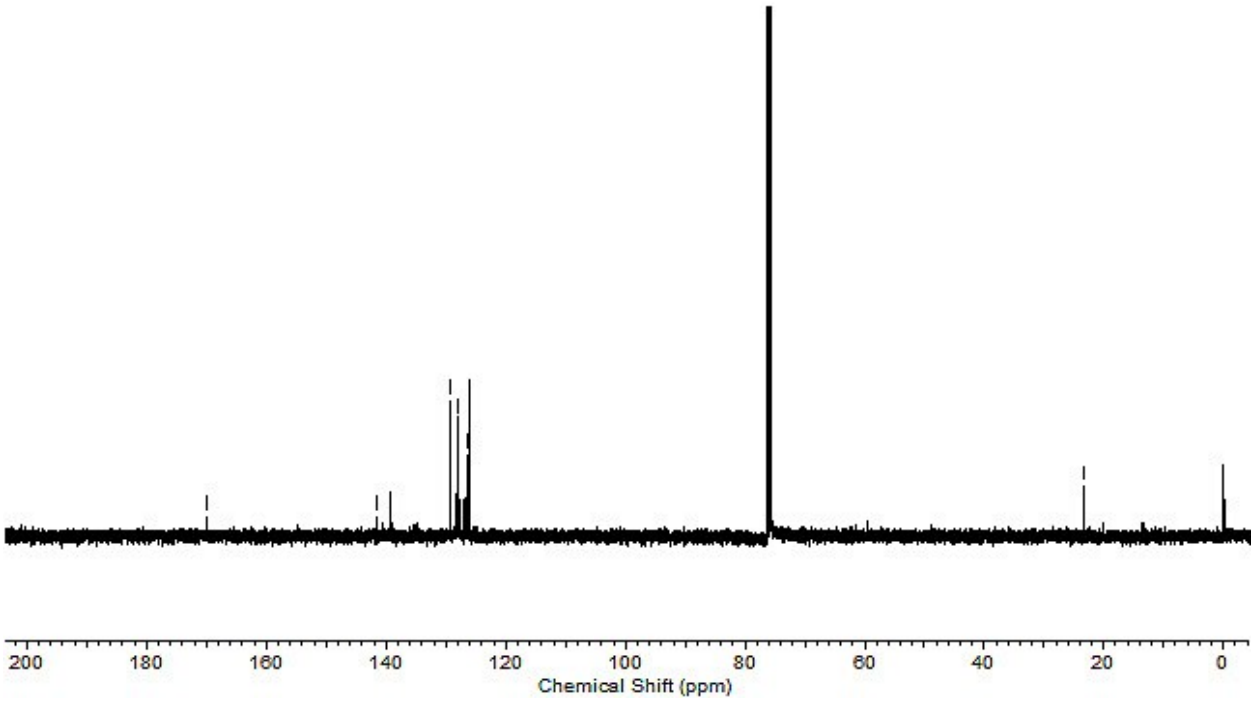
Copies of ^{1}H and ^{13}C NMR Spectra

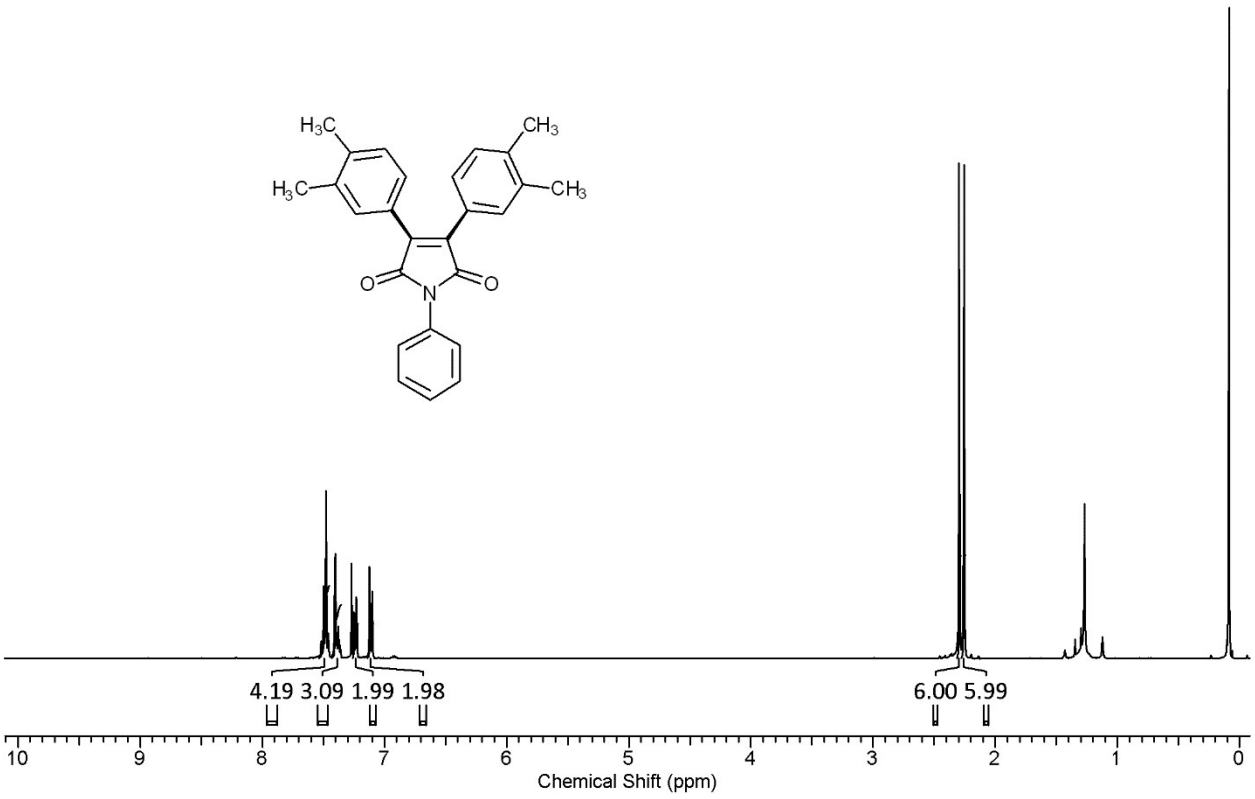


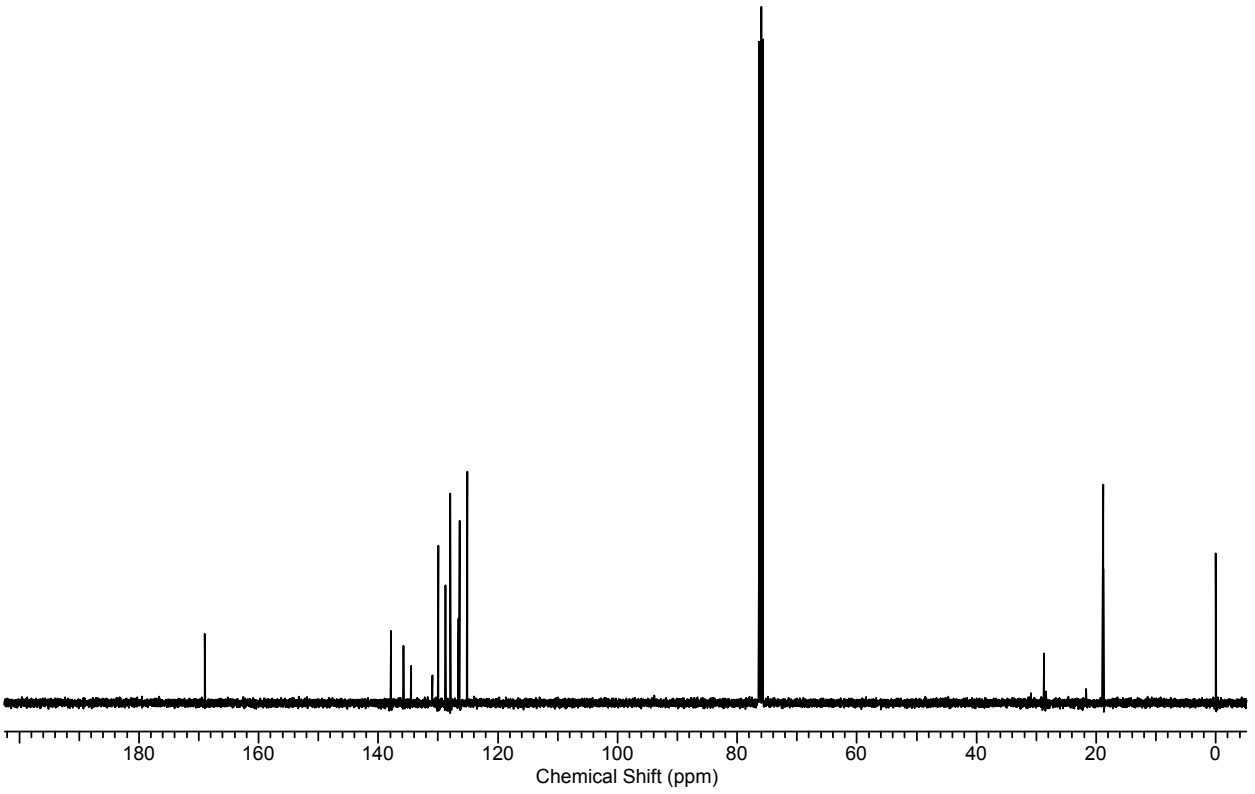

H3CO
OCH3
H3CO
OCH3
O
N
O
CH3
2.03 1.94 2.02
6.10 5.93 3.00
10
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

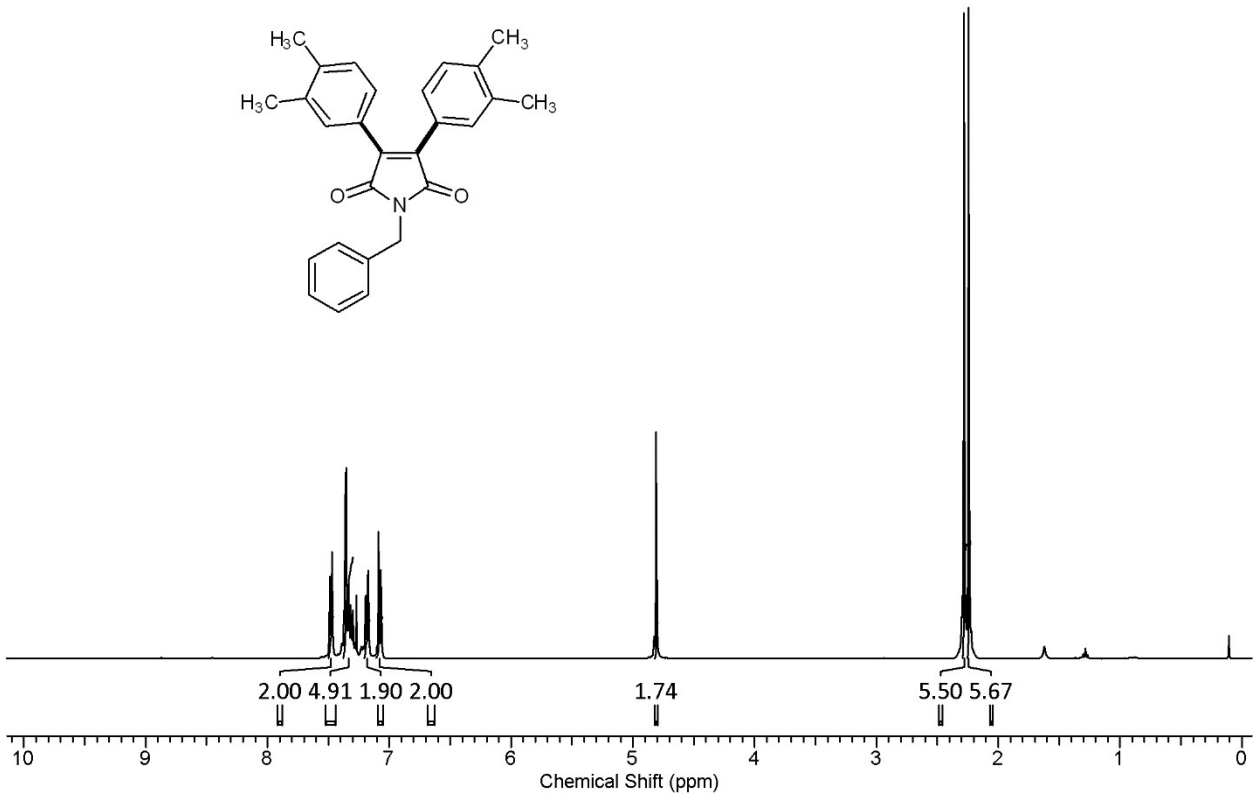

200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

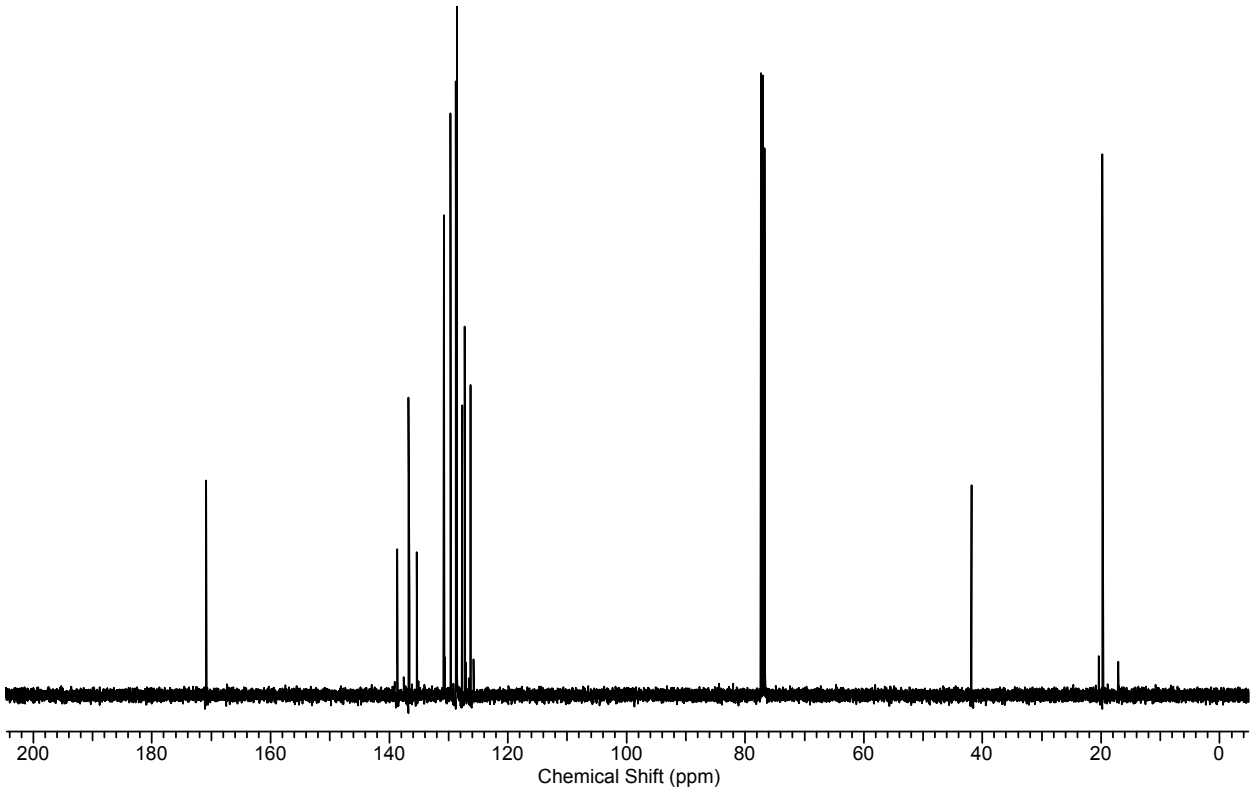

O
O
N
CH3
7.74
10.00
2.84
10
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

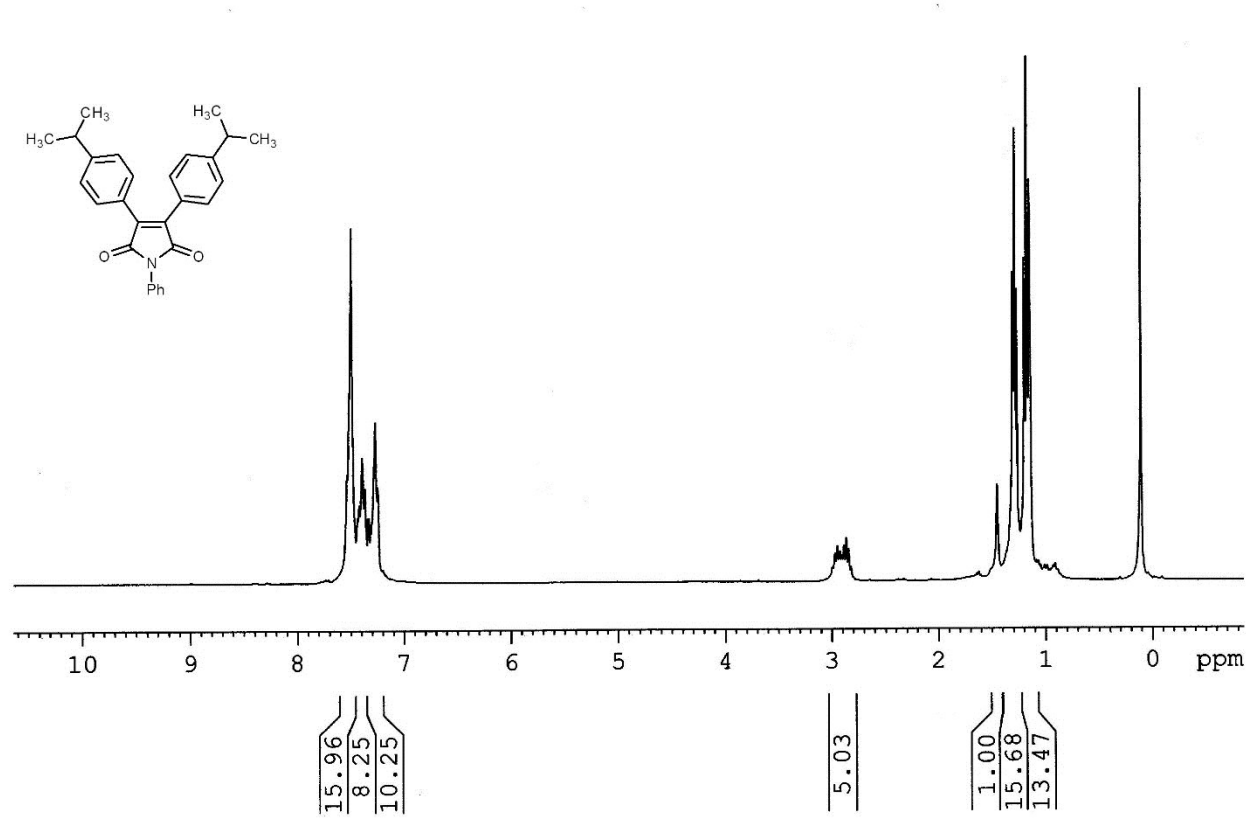

200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

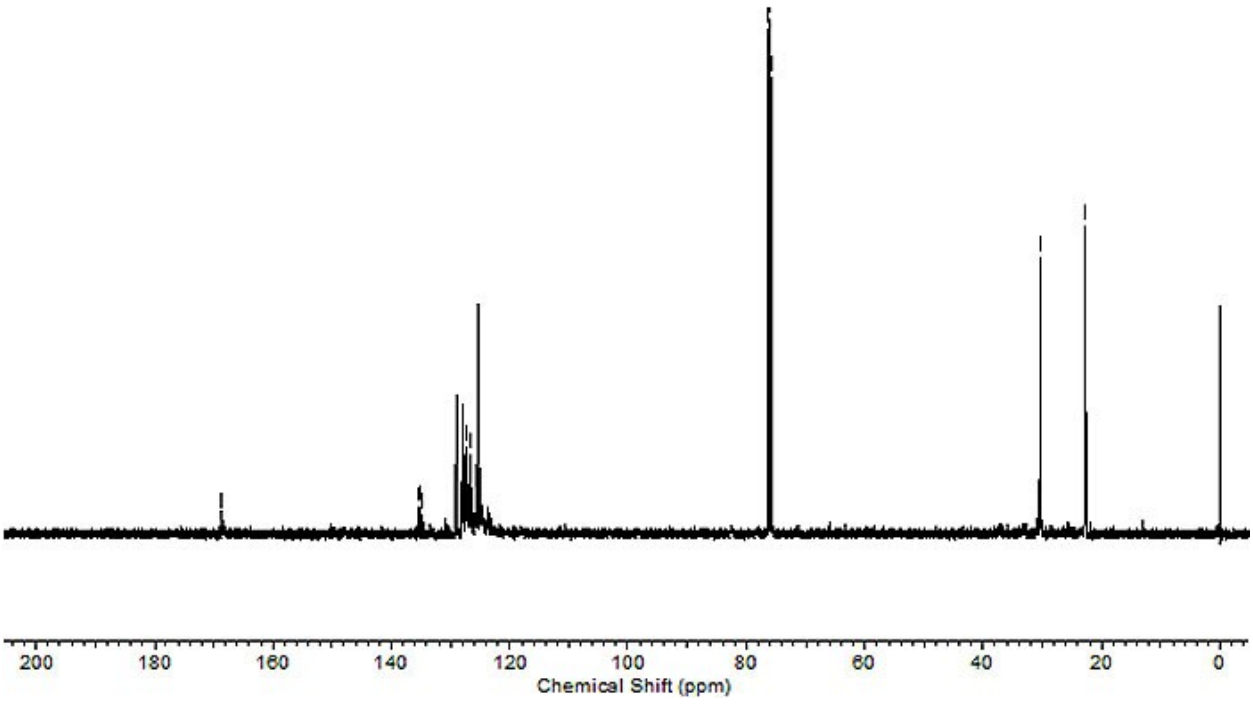

H3C
CH3
H3C
CH3
O
N
O
4.19 3.09 1.99 1.98
6.00 5.99
10 9 8 7 6 5 4 3 2 1 0
Chemical Shift (ppm)

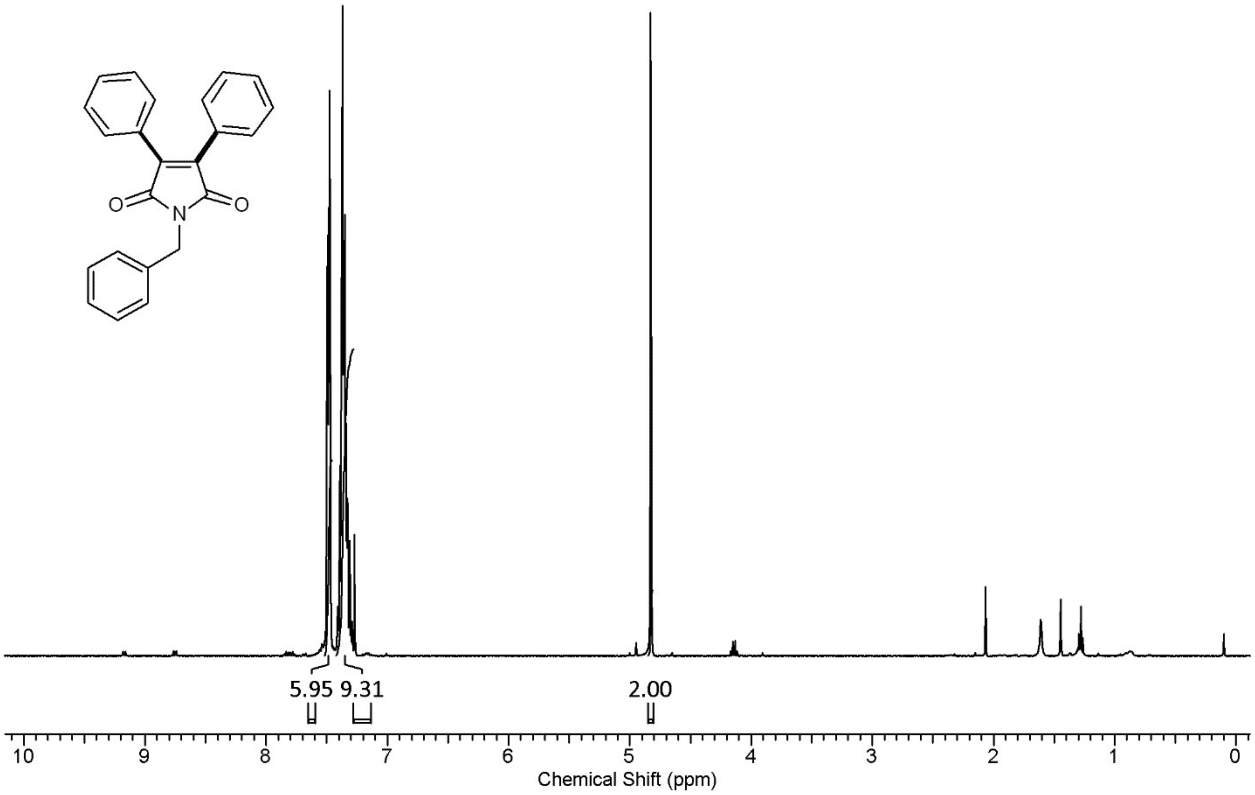

180 160 140 120 100 80 60 40 20 0
Chemical Shift (ppm)

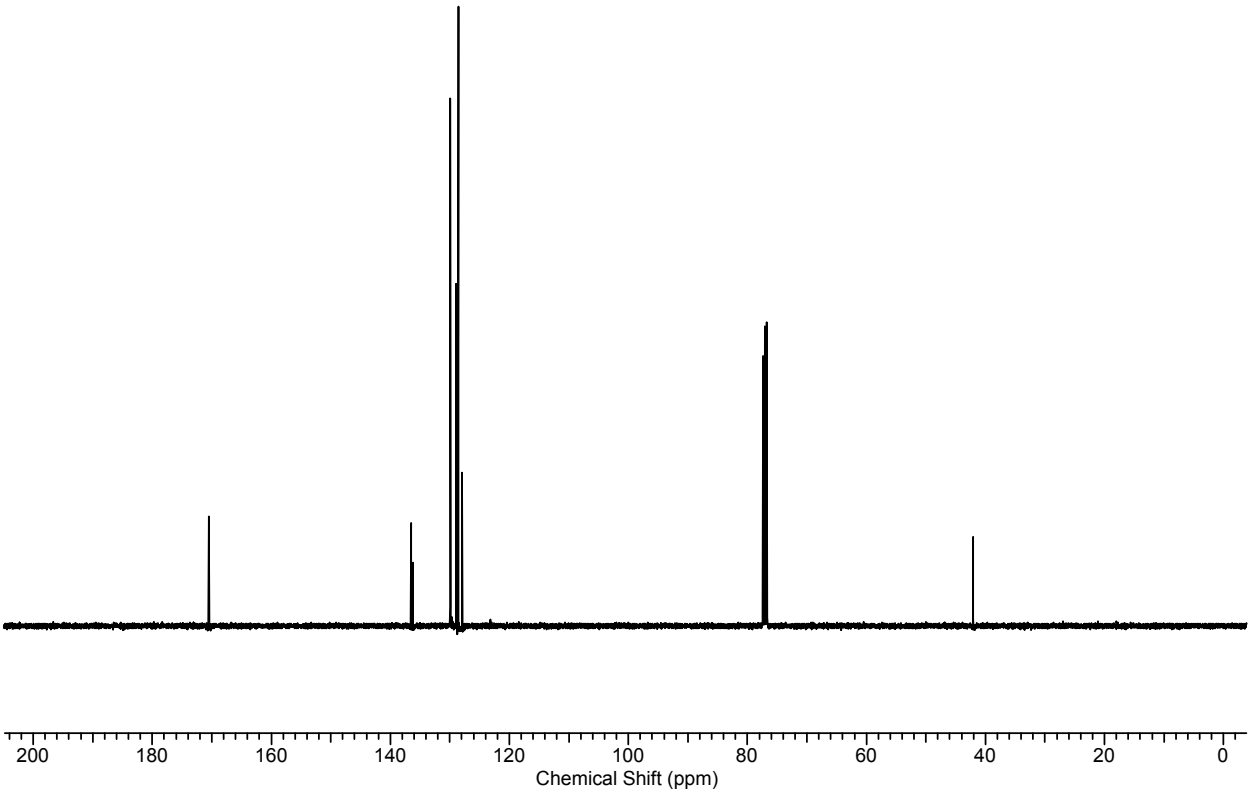

H3C
CH3
H3C
CH3
O
N
O
2.00 4.91 1.90 2.00
1.74
5.50 5.67
10 9 8 7 6 5 4 3 2 1 0
Chemical Shift (ppm)

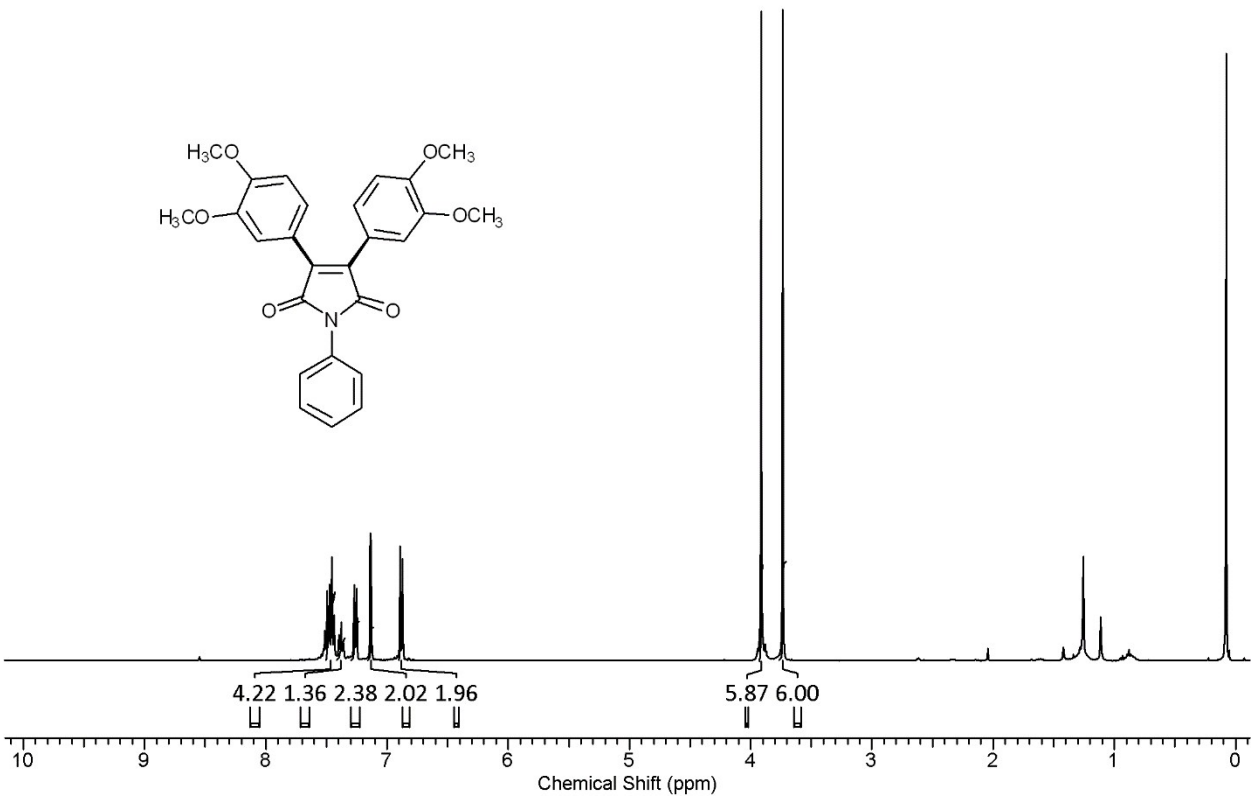

200 180 160 140 120 100 80 60 40 20 0
Chemical Shift (ppm)

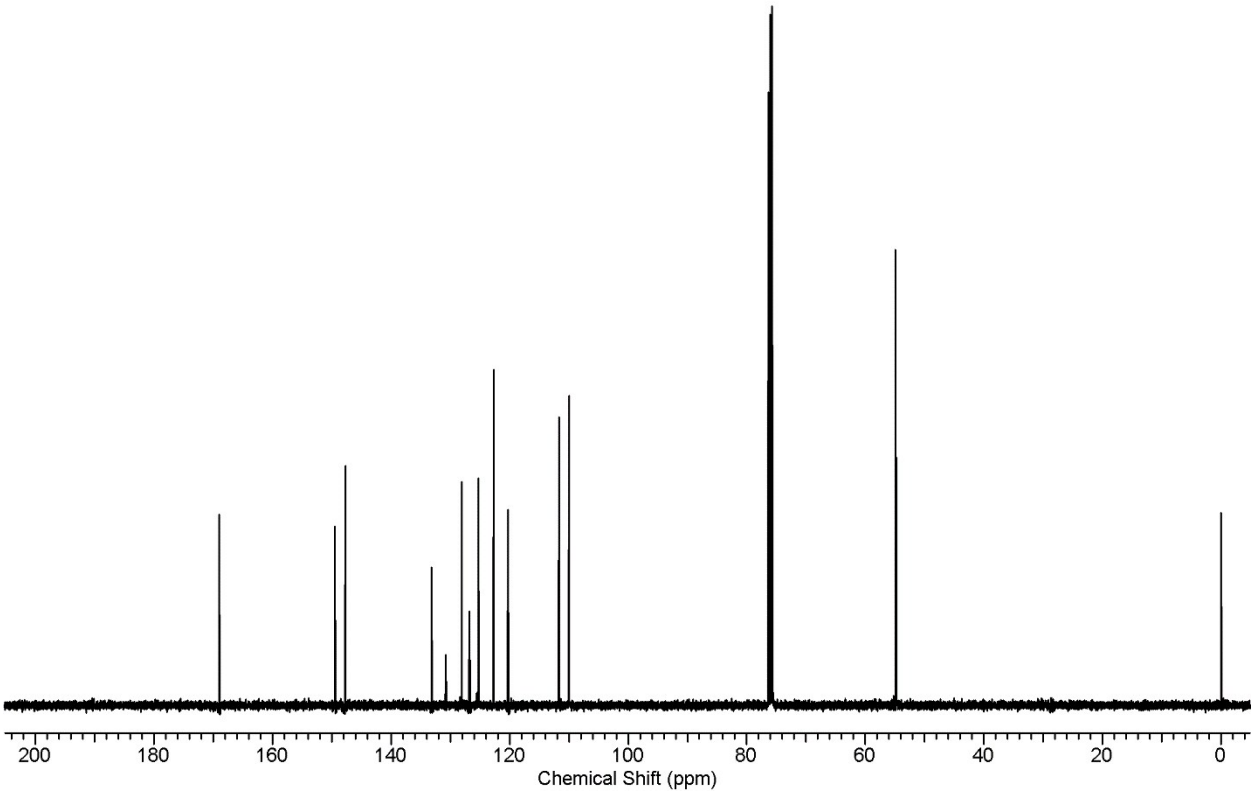

CH3
H3C
H3C
CH3
O
N
O
Ph
10
9
8
7
6
5
4
3
2
1
0
ppm
15.96
8.25
10.25
5.03
1.00
15.68
13.47

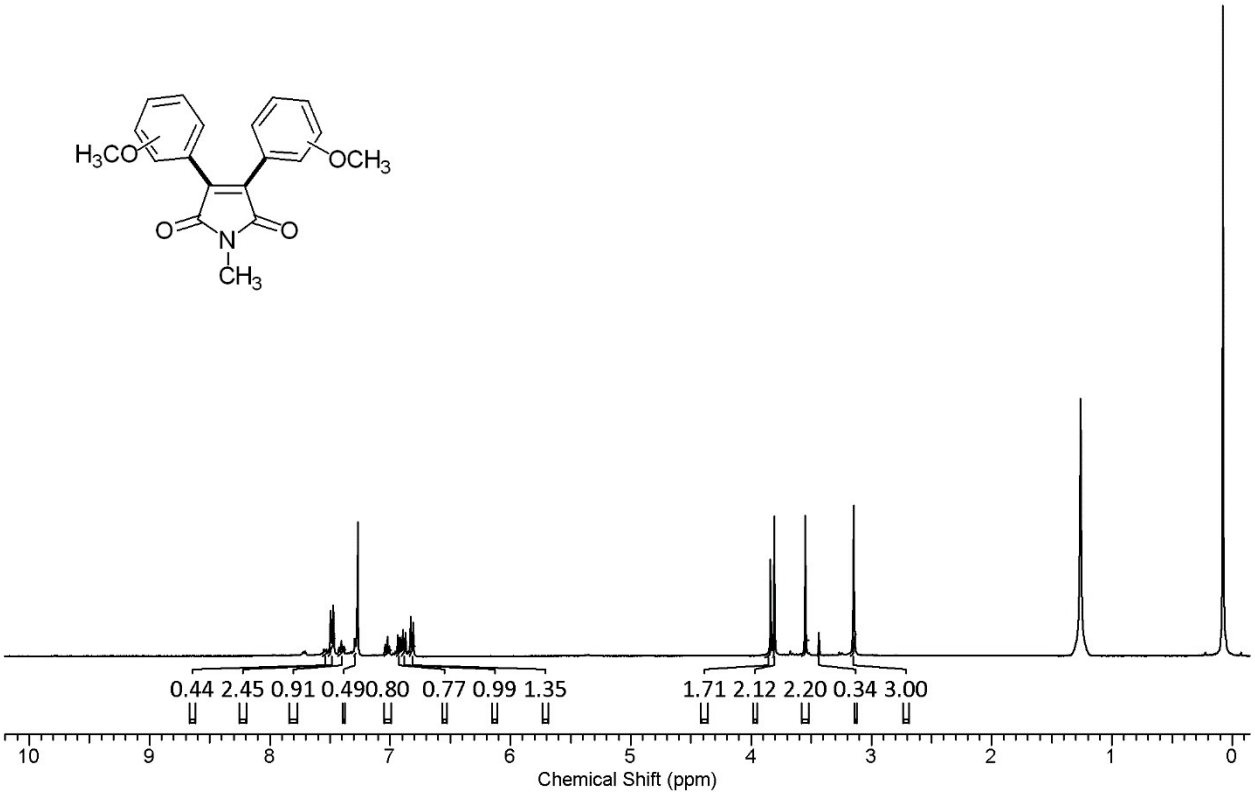

200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

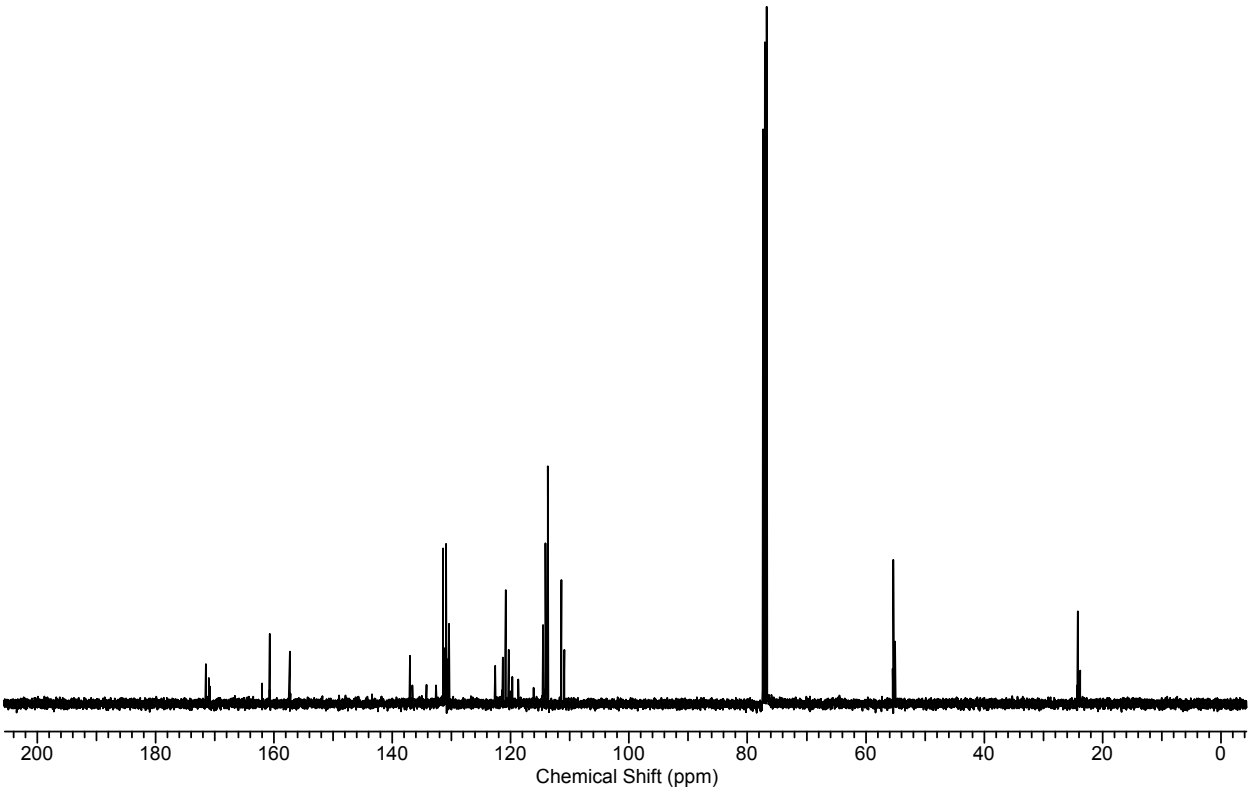

O
N
O
5.95
9.31
2.00
10
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

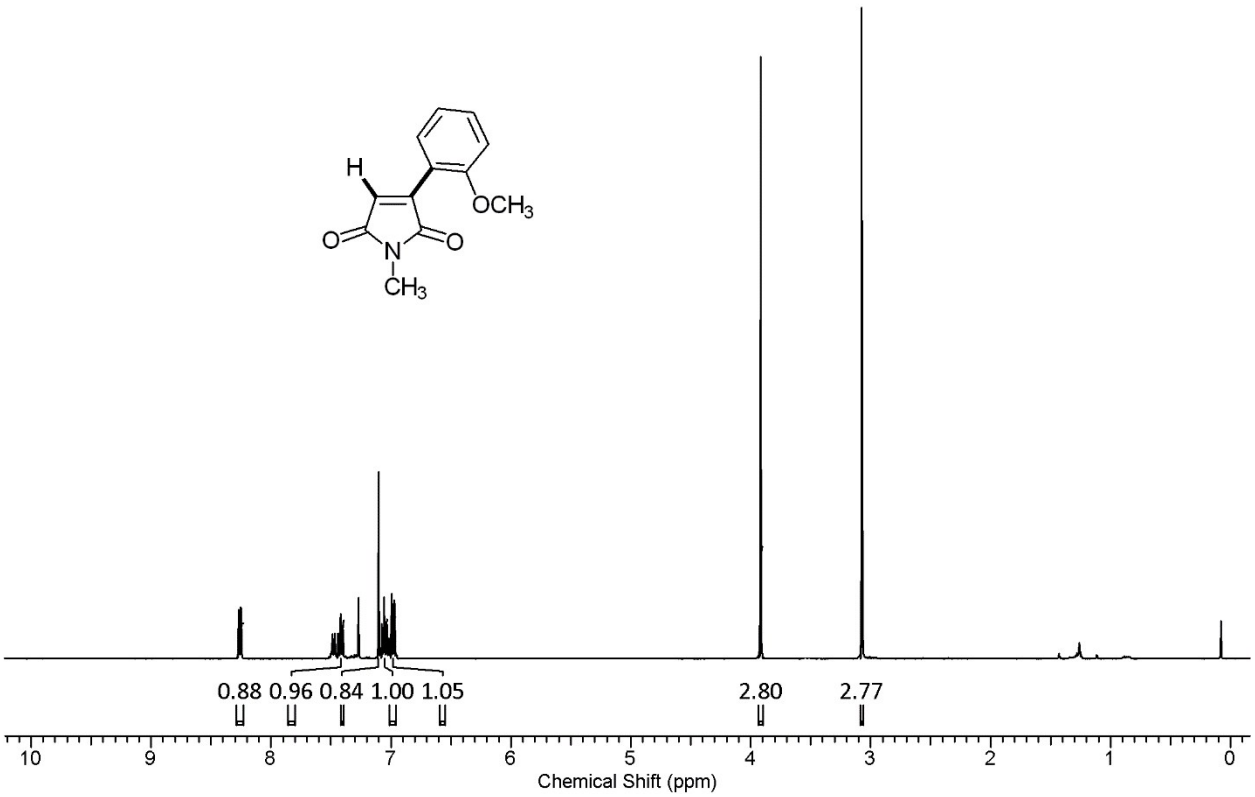

200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

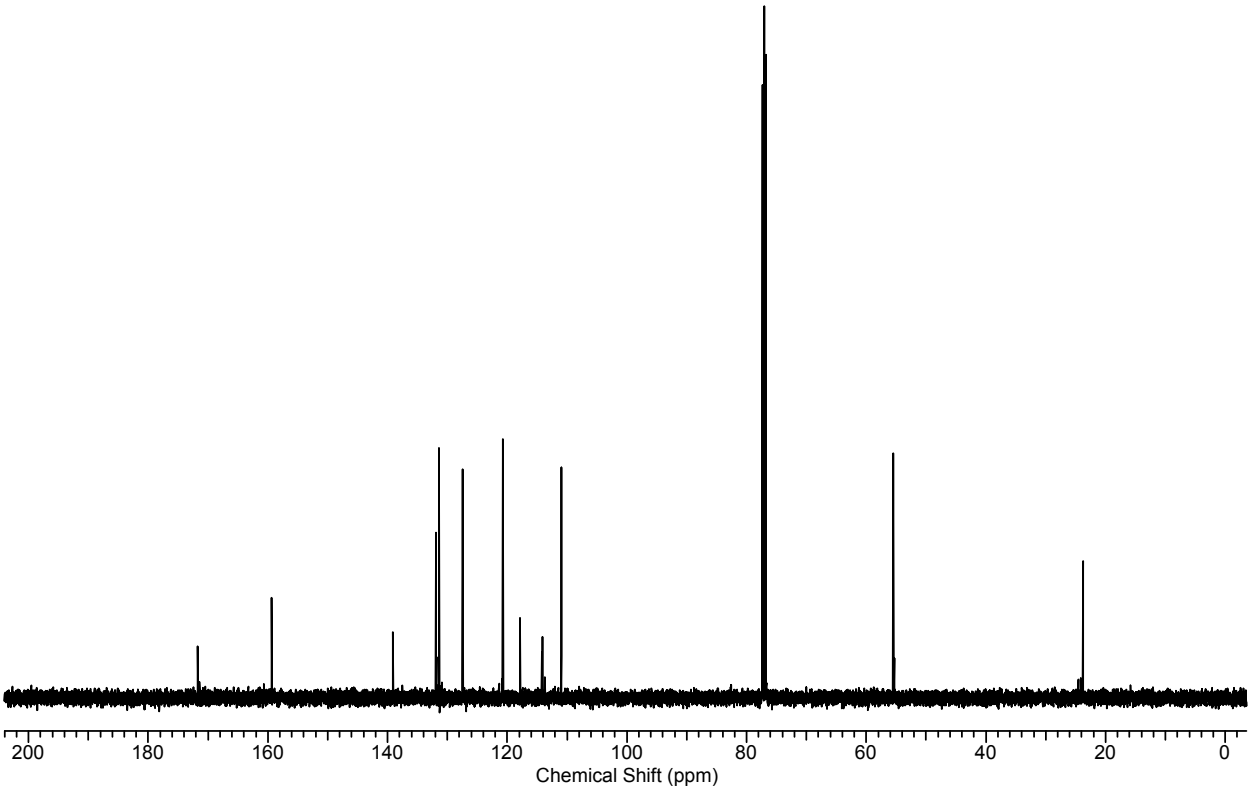

H3CO
OCH3
H3CO
OCH3
O
N
O
4.22 1.36 2.38 2.02 1.96
5.87 6.00
10 9 8 7 6 5 4 3 2 1 0
Chemical Shift (ppm)

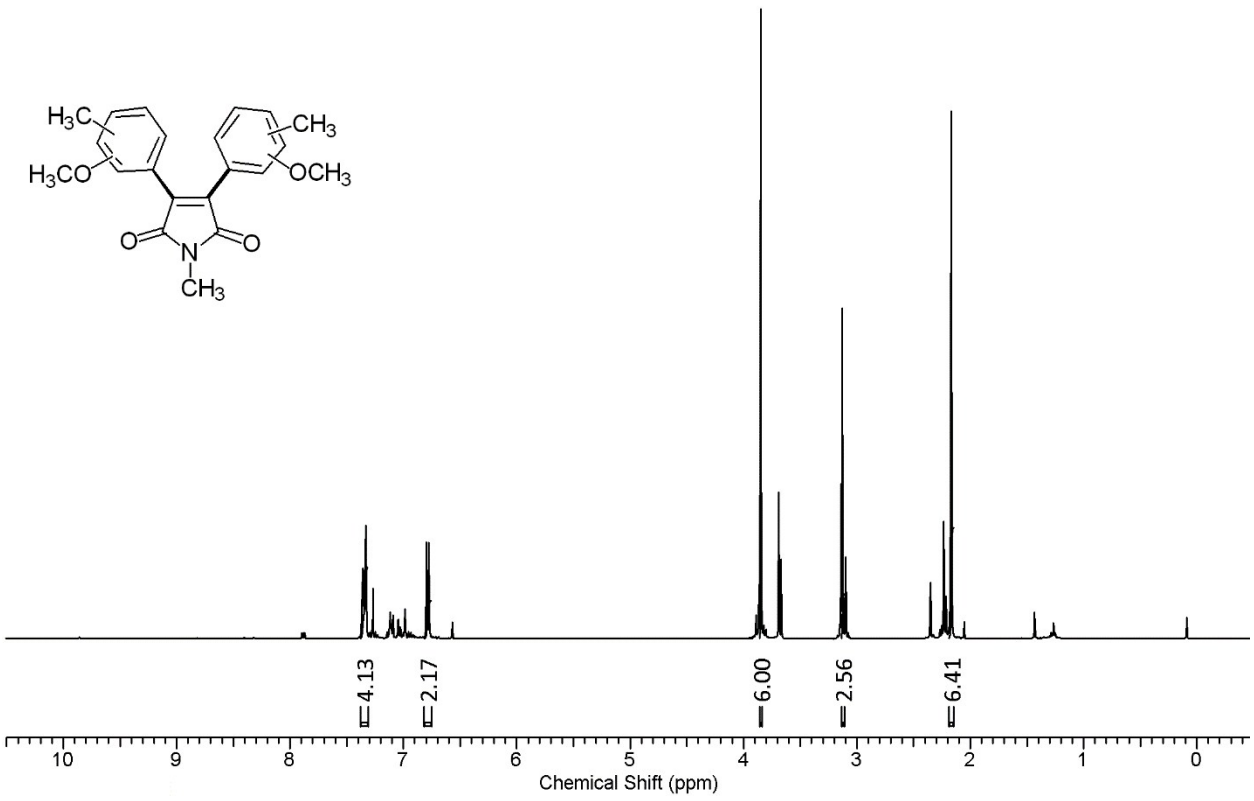

200 180 160 140 120 100 80 60 40 20 0
Chemical Shift (ppm)

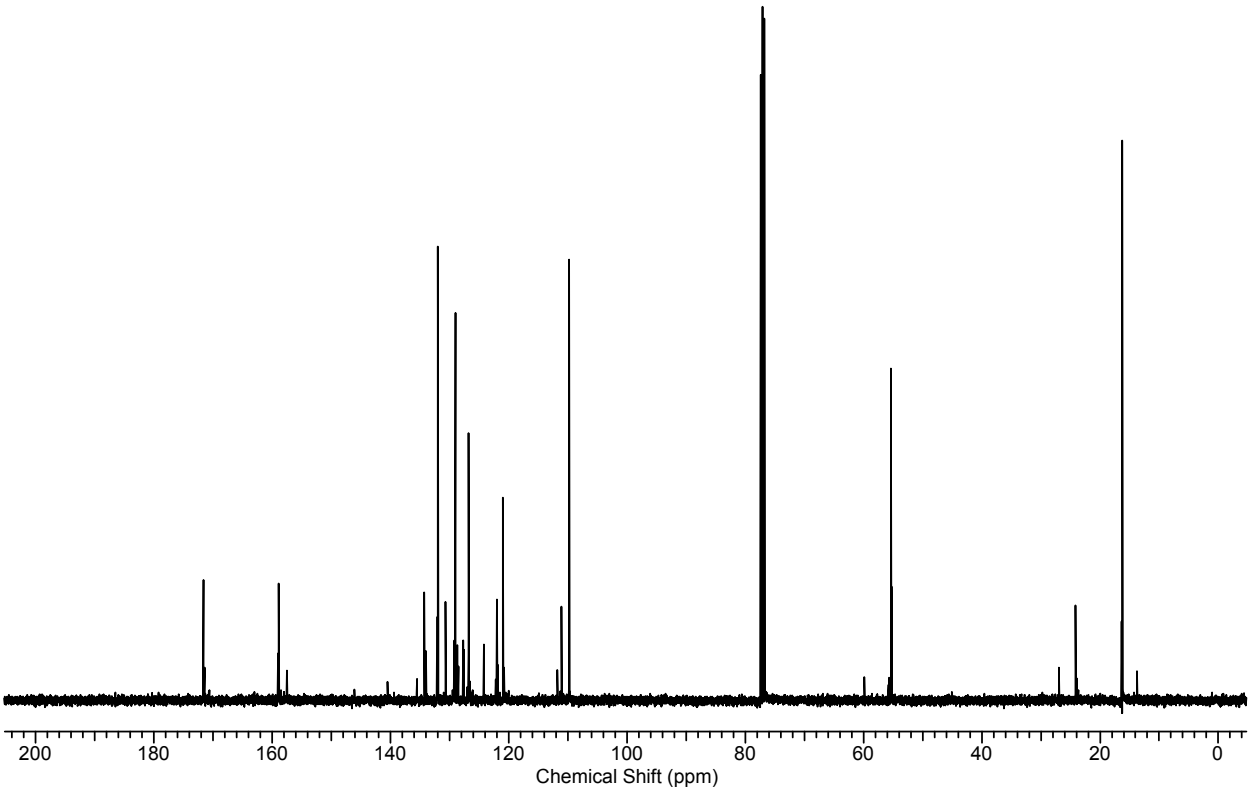

H3CO
OCH3
O
N
O
CH3
0.44 2.45 0.91 0.49 0.80 0.77 0.99 1.35
1.71 2.12 2.20 0.34 3.00
10 9 8 7 6 5 4 3 2 1 0
Chemical Shift (ppm)

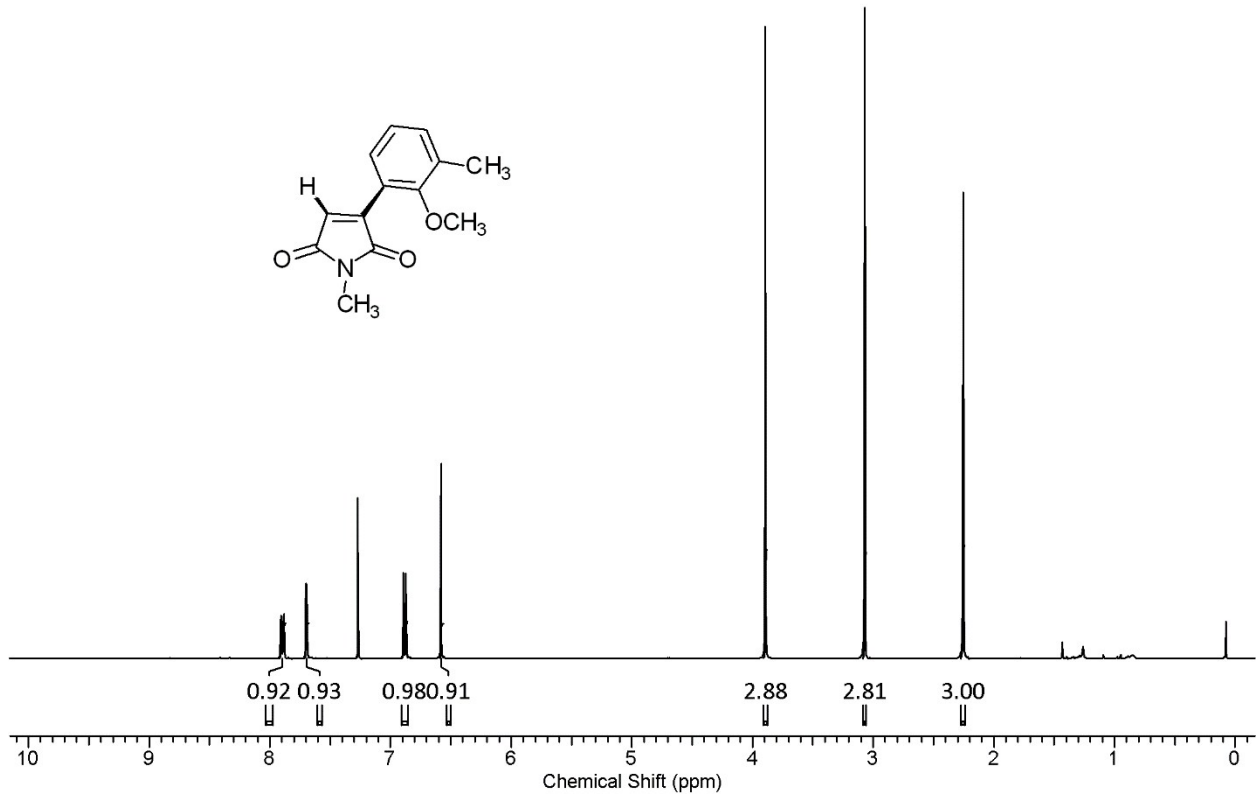

200 180 160 140 120 100 80 60 40 20 0
Chemical Shift (ppm)

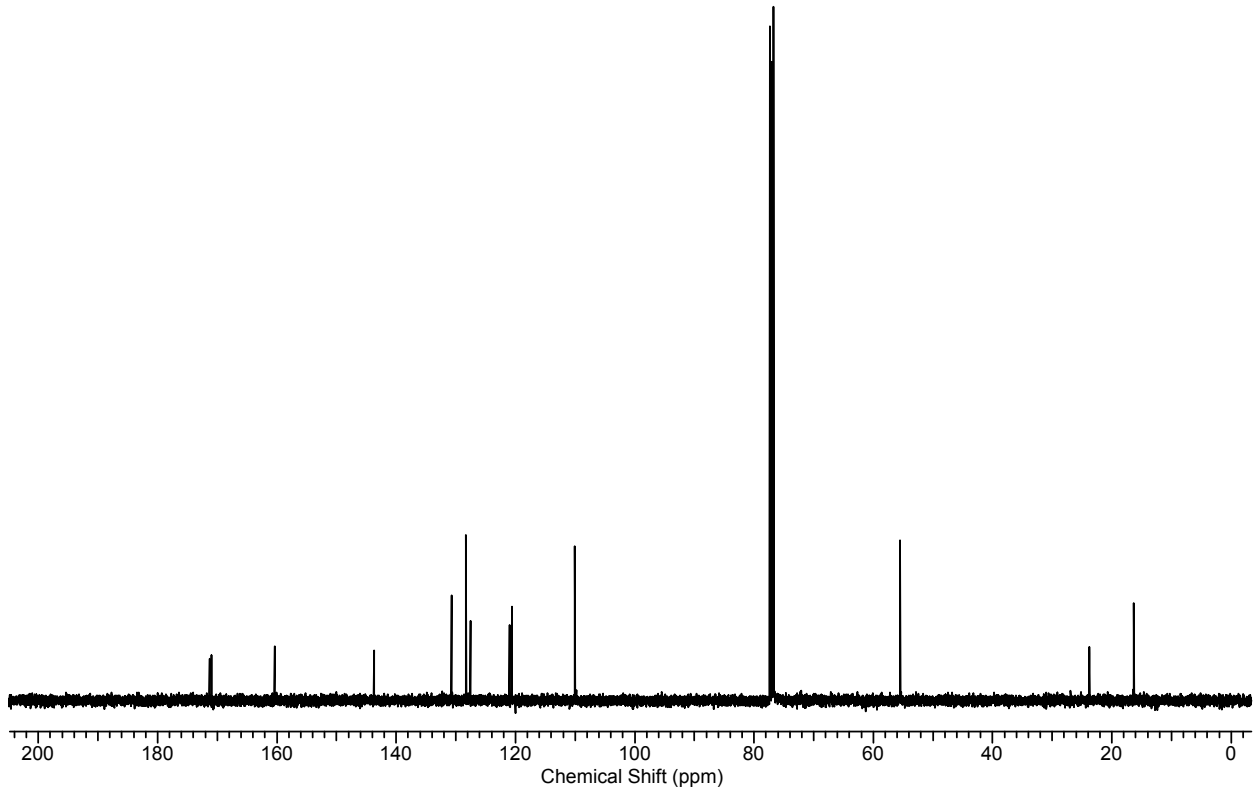

H
OCH3
O
N
O
CH3
0.88 0.96 0.84 1.00 1.05
2.80
2.77
10
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)


200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)


H3C
CH3
H3CO
OCH3
O
N
O
CH3
4.13
2.17
6.00
2.56
6.41
10
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)


200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)


H
CH3
OCH3
O
N
O
CH3
0.92 0.93 0.98 0.91
2.88
2.81
3.00
10 9 8 7 6 5 4 3 2 1 0
Chemical Shift (ppm)


200 180 160 140 120 100 80 60 40 20 0
Chemical Shift (ppm)