

Electronic Supporting Information for

Photocatalytic C(sp²)-OMe Bond Cleavage and Amidation of Anisoles with *N*-Hydroxyphthalimides and Phthalimides

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Table of Contents

1) General information.....	S2
2) Experimental section.....	S2
3) Photochemical setup.....	S3
4) HRMS analysis	S3
5) Natural population analysis	S4-S7
6) Characterization data of products.....	S7-S20
7) References	S20-S21
8) NMR spectra of products	S22-S65

1. General information.

All visible light-induced reactions were conducted in borosilicate glass tubes with Teflon-coated magnetic stirring bars and placed 5 cm from two commercial blue LEDs (manufacturer: Qin Tao trading co., model: SSG11701, 18 W, $\lambda = 460\text{--}462$ nm, URL: <https://item.taobao.com/item.htm?spm=a230r.1.14.8.78777564LdOJDG&id=638346217581&ns=1&abbucket=13#detail>) with a fan placed upside for cooling (Figure S1). All starting materials and reagents were purchased from commercial sources and used as received unless otherwise noted. Reactions were monitored using thin-layer chromatography (TLC) on commercial silica gel plates. Visualization of the developed plates was performed under UV light (254 nm). NMR spectra data were obtained on Avance (III) HD 400 MHz instruments. ^1H NMR and ^{13}C NMR spectra were referenced to residual protic solvent peaks or TMS signal (0 ppm). ^{19}F NMR chemical shifts were externally referenced to CCl_3F (0 ppm). Data for ^1H NMR are recorded as follows: chemical shift (δ , ppm), multiplicity (s = singlet, d = doublet, t = triplet, m = multiplet or unresolved, br = broad singlet, coupling constant (s) in Hz, integration). Data for ^{13}C and ^{19}F NMR are reported in terms of chemical shift (δ , ppm). HRMS Spectra were obtained with Waters Q-TOF Premier (ESI, positive mode) spectrometers.

2. Experimental section

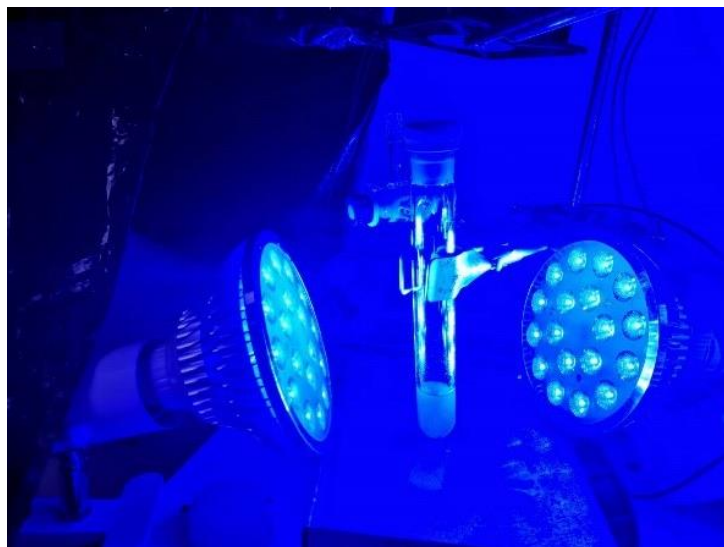
2.1 General experimental procedure for amidation of anisoles with *N*-hydroxyphthalimides.

To a 10 mL Schlenk tube equipped with a magnetic stir bar was added *N*-hydroxyphthalimides (**2**) (0.2 mmol), 4CzIPN (7.9 mg, 0.01 mmol), PPh_3 (78.6 mg, 0.3 mmol) and NaHCO_3 (25.2 mg, 0.3 mmol) and the tube was evacuated and backfilled with N_2 (three times). After addition of *tert*-butylmethylether (1.0 mL, 0.2 M) and anisole (**1**) (2 mmol) by syringe under N_2 , the mixture was placed on a stir-plate. Two 18 W 460–462 nm blue LEDs were placed on opposite sides of the vial at approximately 5 cm distance. The reaction mixture was stirred at rt for 22 h. After completion, the reaction mixture was concentrated in vacuo and the residue was purified by flash column chromatography to afford the products **3**.

2.2 General experimental procedure for amidation of anisoles with phthalimides.

To a 10 mL Schlenk tube equipped with a magnetic stir bar was added phthalimides (**4**) (0.2 mmol), 4CzIPN (7.9 mg, 0.01 mmol) and Cs_2CO_3 (97.8 mg, 0.3 mmol) and the tube was evacuated and backfilled with N_2 (three times). After addition of dichloromethane (1.0 mL, 0.2 M) and anisole (**1**) (2 mmol) by syringe under N_2 , the mixture was placed on a stir-plate. Two 18 W 460–462 nm blue LEDs were placed on opposite sides of the vial at approximately 5 cm distance. The reaction mixture was stirred at rt for 22 h. After completion, the reaction mixture was concentrated in vacuo and the residue was purified by flash column chromatography to afford the products **3**.

3. Photochemical setup



(A fan on the top of tube to make sure the reaction temperature is rt)

Figure S1 General reaction setup

4. HRMS analysis (radical trapping experiment with TEMPO)

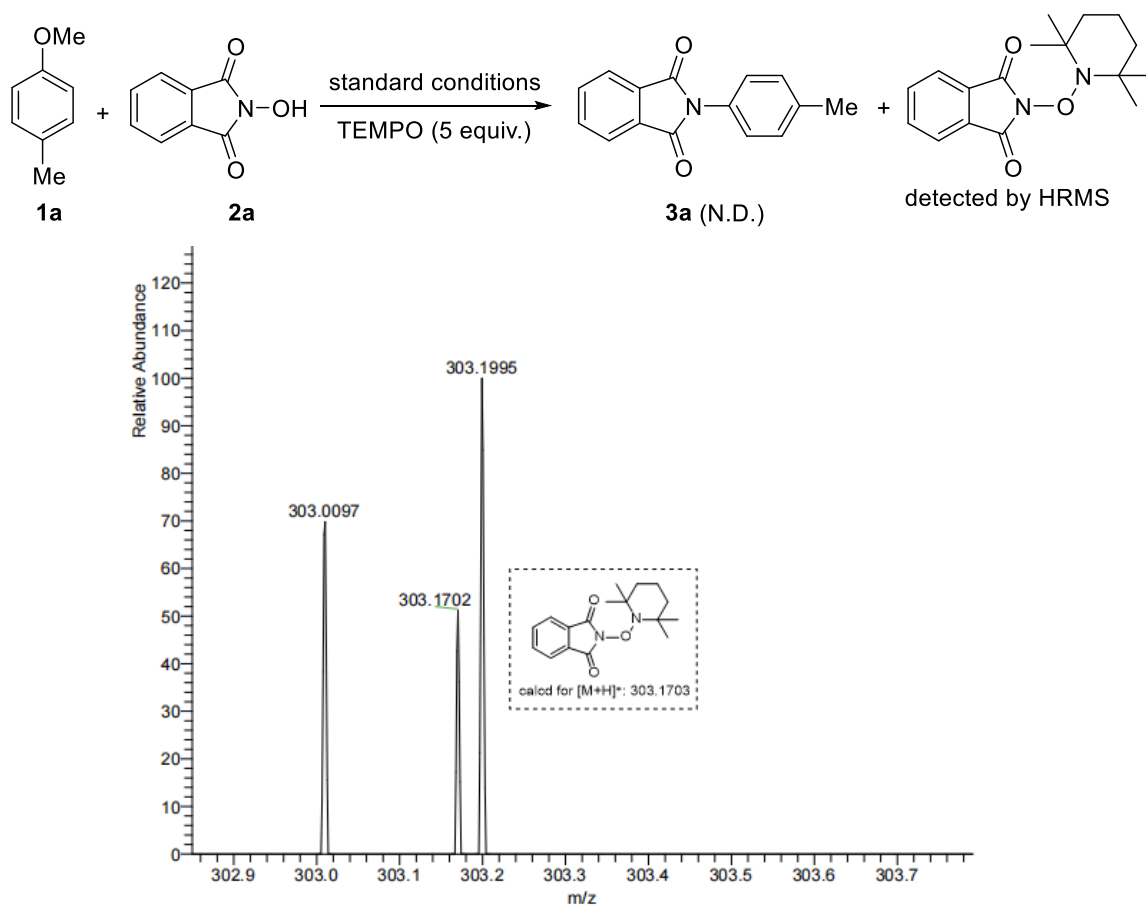
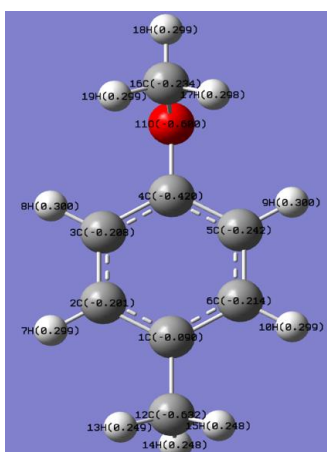


Figure S2 The result of HRMS analysis

5. Natural population analysis



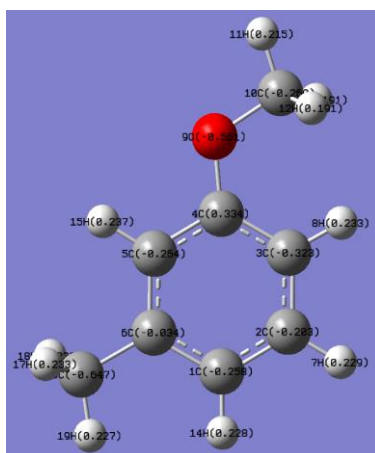
1a

Summary of Natural Population Analysis of **1a**:

Natural Population

Atom	No	Natural Charge	Core	Valence	Rydberg	Total
C	1	-0.08989	1.99897	4.03192	0.01557	6.04645
C	2	-0.20069	1.99891	4.20519	0.01557	6.21967
C	3	-0.20773	1.99887	4.23104	0.01782	6.24773
C	4	-0.42009	1.99859	3.68500	0.02626	5.70985
C	5	-0.24161	1.99887	4.23104	0.01782	6.24773
C	6	-0.21422	1.99891	4.20519	0.01557	6.21967
H	7	0.29901	0.00000	0.76934	0.00421	0.77355
H	8	0.29991	0.00000	0.76078	0.00539	0.76617
H	9	0.29971	0.00000	0.76078	0.00539	0.76617
H	10	0.29945	0.00000	0.76934	0.00421	0.77355
O	11	-0.59957	1.99976	6.57064	0.02917	8.59957
C	12	-0.63156	1.99933	4.62243	0.00979	6.63156
H	13	0.24891	0.00000	0.77420	0.00289	0.77709
H	14	0.24809	0.00000	0.76896	0.00265	0.77161

H	15	0.24814	0.00000	0.77420	0.00289	0.77709
C	16	-0.23388	1.99928	4.22159	0.01300	6.23388
H	17	0.29810	0.00000	0.81609	0.00312	0.81922
H	18	0.29902	0.00000	0.79825	0.00197	0.80022
H	19	0.29902	0.00000	0.81609	0.00312	0.81922



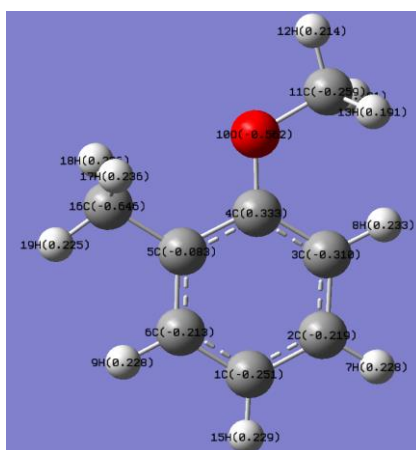
1n

Summary of Natural Population Analysis of **1n**:

Natural Population

Atom	No	Natural Charge	Core	Valence	Rydberg	Total
C	1	-0.25762	1.99889	4.24277	0.01597	6.25762
C	2	-0.20335	1.99902	4.18680	0.01754	6.20335
C	3	-0.32336	1.99888	4.30911	0.01537	6.32336
C	4	0.33403	1.99868	3.64246	0.02484	5.66597
C	5	-0.26421	1.99880	4.24741	0.01801	6.26421
C	6	-0.03356	1.99895	4.01899	0.01562	6.03356
H	7	0.22892	0.00000	0.76699	0.00409	0.77108
H	8	0.23305	0.00000	0.76176	0.00518	0.76695
O	9	-0.56114	1.99971	6.53314	0.02828	8.56114

C	10	-0.25959	1.99934	4.24743	0.01282	6.25959
H	11	0.21456	0.00000	0.78314	0.00230	0.78544
H	12	0.19066	0.00000	0.80583	0.00351	0.80934
H	13	0.19065	0.00000	0.80583	0.00351	0.80935
H	14	0.22756	0.00000	0.76812	0.00433	0.77244
H	15	0.23670	0.00000	0.75877	0.00453	0.76330
C	16	-0.64664	1.99931	4.63684	0.01049	6.64664
H	17	0.23318	0.00000	0.76405	0.00276	0.76682
H	18	0.23318	0.00000	0.76406	0.00276	0.76682
H	19	0.22699	0.00000	0.77014	0.00287	0.77301



1o

Summary of Natural Population Analysis of **1o**:

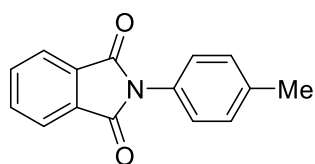
Natural Population

Atom	No	Natural Charge	Core	Valence	Rydberg	Total
C	1	-0.25144	1.99898	4.23533	0.01713	6.25144
C	2	-0.21857	1.99901	4.20251	0.01706	6.21857
C	3	-0.31044	1.99890	4.29571	0.01584	6.31044
C	4	0.33251	1.99859	3.64491	0.02399	5.66749

C	5	-0.08329	1.99886	4.06628	0.01816	6.08329
C	6	-0.21347	1.99891	4.19864	0.01592	6.21347
H	7	0.22848	0.00000	0.76738	0.00414	0.77152
H	8	0.23286	0.00000	0.76208	0.00506	0.76714
H	9	0.22789	0.00000	0.76792	0.00419	0.77211
O	10	-0.56158	1.99971	6.53510	0.02677	8.56158
C	11	-0.25925	1.99934	4.24693	0.01298	6.25925
H	12	0.21443	0.00000	0.78321	0.00236	0.78557
H	13	0.19106	0.00000	0.80538	0.00356	0.80894
H	14	0.19106	0.00000	0.80538	0.00356	0.80894
H	15	0.22908	0.00000	0.76655	0.00437	0.77092
C	16	-0.64557	1.99932	4.63509	0.01117	6.64557
H	17	0.23562	0.00000	0.76073	0.00365	0.76438
H	18	0.23561	0.00000	0.76074	0.00365	0.76439
H	19	0.22503	0.00000	0.77221	0.00276	0.77497

6. Characterization data of products

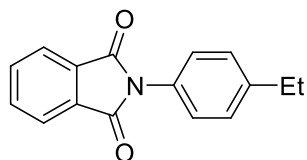
2-(*p*-Tolyl)isoindoline-1,3-dione (3a)



3a

EtOAc/*n*-Hexane = 1/8 (eluent); white solid, 77% yield, 36.5 mg; ¹H NMR (400 MHz, CDCl₃) δ 7.97 – 7.94 (m, 2H), 7.80 – 7.78 (m, 2H), 7.35 – 7.28 (m, 4H), 2.41 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 167.5, 138.3, 134.4, 131.8, 129.8, 128.9, 126.5, 123.8, 21.3. The spectral data are consistent with those reported in the literature.^[1]

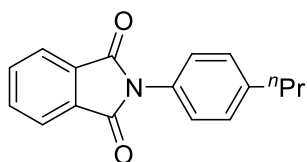
2-(4-Ethylphenyl)isoindoline-1,3-dione (3b)



3b

EtOAc/*n*-Hexane = 1/8 (eluent); white solid, 79% yield, 39.7 mg; ^1H NMR (400 MHz, CDCl_3) δ 7.97 – 7.95 (m, 2H), 7.83 – 7.75 (m, 2H), 7.34 (s, 4H), 2.71 (q, $J = 7.6$ Hz, 2H), 1.28 (t, $J = 7.6$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 167.5, 144.5, 134.4, 131.8, 129.1, 128.7, 126.5, 123.8, 28.6, 15.5. The spectral data are consistent with those reported in the literature.^[1]

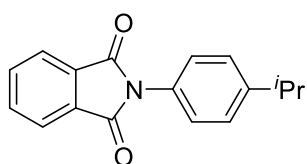
2-(4-Propylphenyl)isoindoline-1,3-dione (3c)



3c

EtOAc/*n*-Hexane = 1/8 (eluent); white solid, 74% yield, 39.2 mg; ^1H NMR (400 MHz, CDCl_3) δ 7.97 – 7.95 (m, 2H), 7.80 – 7.78 (m, 2H), 7.36 – 7.30 (m, 4H), 2.72 – 2.56 (m, 2H), 1.74 – 1.63 (m, 2H), 0.98 (t, $J = 7.3$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 167.5, 142.9, 134.4, 131.8, 129.2, 129.1, 126.4, 123.8, 37.8, 24.5, 13.9. The spectral data are consistent with those reported in the literature.^[2]

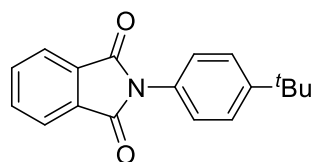
2-(4-Isopropylphenyl)isoindoline-1,3-dione (3d)



3d

EtOAc/*n*-Hexane = 1/8 (eluent); white solid, 69% yield, 36.6 mg; ^1H NMR (400 MHz, CDCl_3) δ 7.97 – 7.95 (m, 2H), 7.80 – 7.78 (m, 2H), 7.41 – 7.30 (m, 4H), 2.97 (dt, $J = 14.0, 6.8$ Hz, 1H), 1.29 (d, $J = 6.8$ Hz, 6H); ^{13}C NMR (100 MHz, CDCl_3) δ 167.5, 148.9, 134.4, 131.8, 129.2, 127.3, 126.5, 123.7, 33.9, 23.9. The spectral data are consistent with those reported in the literature.^[1]

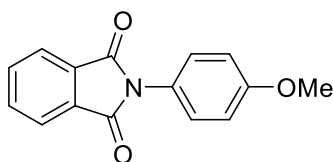
2-(4-*tert*-Butylphenyl)isoindoline-1,3-dione (3e)



3e

EtOAc/*n*-Hexane = 1/8 (eluent); white solid, 67% yield, 37.4 mg; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.97 – 7.95 (m, 2H), 7.81 – 7.79 (m, 2H), 7.53 (d, $J = 8.2$ Hz, 2H), 7.36 (d, $J = 8.2$ Hz, 2H), 1.36 (s, 9H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 167.5, 151.2, 134.4, 131.8, 128.9, 126.2, 126.1, 123.8, 34.8, 31.3. The spectral data are consistent with those reported in the literature.^[1]

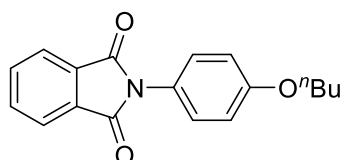
2-(4-Methoxyphenyl)isoindoline-1,3-dione (3f)



3f

EtOAc/*n*-Hexane = 1/5 (eluent); white solid, 84% yield, 42.5 mg; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.96 – 7.94 (m, 2H), 7.81 – 7.77 (m, 2H), 7.37 – 7.30 (m, 2H), 7.08 – 6.98 (m, 2H), 3.85 (s, 3H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 167.6, 159.3, 134.3, 131.8, 128.0, 124.3, 123.7, 114.5, 55.5. The spectral data are consistent with those reported in the literature.^[1]

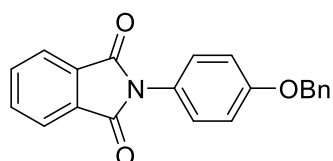
2-(4-Butoxyphenyl)isoindoline-1,3-dione (3g)



3g

EtOAc/*n*-Hexane = 1/5 (eluent); white solid, 84% yield, 42.5 mg; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.96 – 7.94 (m, 2H), 7.81 – 7.77 (m, 2H), 7.37 – 7.30 (m, 2H), 7.08 – 6.98 (m, 2H), 3.85 (s, 3H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 167.6, 158.8, 134.2, 131.8, 127.8, 123.9, 123.6, 114.9, 67.9, 31.2, 19.2, 13.8. The spectral data are consistent with those reported in the literature.^[3]

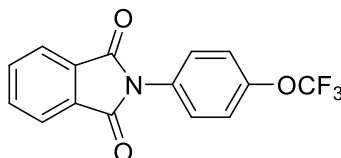
2-(4-(Benzyloxy)phenyl)isoindoline-1,3-dione (3h)



3h

EtOAc/*n*-Hexane = 1/5 (eluent); white solid, 84% yield, 42.5 mg; ^1H NMR (400 MHz, CDCl_3) δ 7.94 (m, 2H), 7.79 (m, 2H), 7.50 – 7.37 (m, 2H), 7.34 (d, $J = 8.4$ Hz, 4H), 7.09 (d, $J = 8.8$ Hz, 3H), 5.11 (s, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ 167.6, 158.5, 136.7, 134.3, 131.8, 128.7, 128.1, 128.0, 127.5, 124.5, 123.7, 115.4, 70.3. The spectral data are consistent with those reported in the literature.^[4]

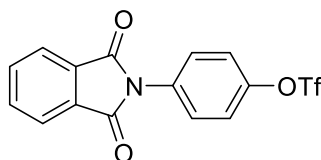
2-(4-(Trifluoromethoxy)phenyl)isoindoline-1,3-dione (3i)



3i

EtOAc/*n*-Hexane = 1/5 (eluent); white solid, 81% yield, 49.7 mg; ^1H NMR (400 MHz, CDCl_3) δ 8.00 – 7.95 (m, 2H), 7.84 – 7.80 (m, 2H), 7.55 – 7.47 (m, 2H), 7.36 (d, $J = 8.5$ Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ 167.0, 148.4, 134.7, 131.6, 130.2, 127.9, 123.9, 121.7, 120.4 (q, $J = 256$ Hz); ^{19}F NMR (376 MHz, CDCl_3) δ -57.85. The spectral data are consistent with those reported in the literature.^[1]

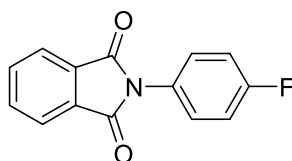
4-(1,3-Dioxoisoindolin-2-yl)phenyl trifluoromethanesulfonate (3j)



3j

EtOAc/*n*-Hexane = 1/5 (eluent); white solid, 81% yield, 49.7 mg; ^1H NMR (400 MHz, CDCl_3) δ 7.99 (m, 2H), 7.83 (m, 2H), 7.62 (d, $J = 8.8$ Hz, 2H), 7.43 (d, $J = 8.8$ Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ 166.8, 148.3, 134.8, 131.8, 131.5, 128.0, 124.0, 122.1, 119.1 (q, $J = 307$ Hz); ^{19}F NMR (376 MHz, CDCl_3) δ -72.67. The spectral data are consistent with those reported in the literature.^[5]

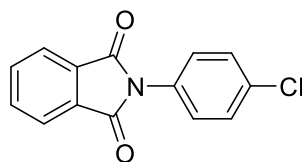
2-(4-Fluorophenyl)isoindoline-1,3-dione (3k)



3k

EtOAc/*n*-Hexane = 1/8 (eluent); white solid, 35% yield, 16.9 mg; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.97 – 7.95 (m, 2H), 7.82 – 7.80 (m, 2H), 7.49 – 7.37 (m, 2H), 7.23 – 7.18 (m, 2H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 167.2, 161.9 (d, $J = 246$ Hz), 134.5, 131.6, 128.4 (d, $J = 8$ Hz), 127.5 (d, $J = 3$ Hz), 123.8, 116.2 (d, $J = 23$ Hz); $^{19}\text{F NMR}$ (376 MHz, CDCl_3) δ -113.01. The spectral data are consistent with those reported in the literature.^[1]

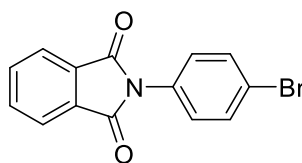
2-(4-Chlorophenyl)isoindoline-1,3-dione (3l)



3l

EtOAc/*n*-Hexane = 1/8 (eluent); white solid, 52% yield, 26.7 mg; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.98 – 7.94 (m, 2H), 7.83 – 7.79 (m, 2H), 7.51 – 7.45 (m, 2H), 7.44 – 7.37 (m, 2H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 166.9, 134.5, 133.7, 131.5, 130.1, 129.3, 127.6, 123.8. The spectral data are consistent with those reported in the literature.^[1]

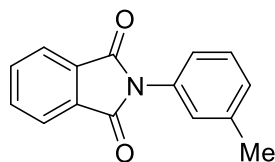
2-(4-Bromophenyl)isoindoline-1,3-dione (3m)



3m

EtOAc/*n*-Hexane = 1/8 (eluent); white solid, 58% yield, 34.9 mg; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.99 – 7.94 (m, 2H), 7.82 – 7.80 (m, 2H), 7.66 – 7.63 (m, 2H), 7.38 – 7.35 (m, 2H); $^{13}\text{C NMR}$ (100 MHz, DMSO) δ 166.9, 134.6, 132.3, 131.5, 130.6, 127.9, 123.9, 121.8. The spectral data are consistent with those reported in the literature.^[1]

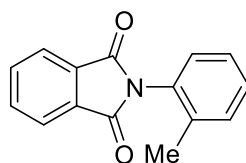
2-(*m*-Tolyl)isoindoline-1,3-dione (3n)



3n

EtOAc/*n*-Hexane = 1/8 (eluent); white solid, 27% yield, 12.8 mg; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.97 – 7.93 (m, 2H), 7.81 – 7.78 (m, 2H), 7.39 (t, $J = 7.8$ Hz, 1H), 7.23 (s, 2H), 7.21 (s, 1H), 2.42 (s, 3H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 167.4, 139.2, 134.4, 131.8, 131.5, 129.1, 129.0, 127.3, 123.8, 123.7, 21.4. The spectral data are consistent with those reported in the literature.^[1]

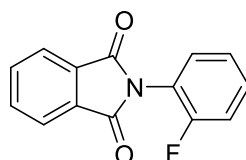
2-(*o*-Tolyl)isoindoline-1,3-dione (**3o**)



3o

EtOAc/*n*-Hexane = 1/8 (eluent); white solid, 16% yield, 7.6 mg; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.97 – 7.95 (m, 2H), 7.82 – 7.79 (m, 2H), 7.41 – 7.30 (m, 3H), 7.21 (d, $J = 7.6$ Hz, 1H), 2.21 (s, 3H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 167.3, 136.5, 134.3, 132.0, 131.1, 130.5, 129.4, 128.7, 126.9, 123.7, 18.0. The spectral data are consistent with those reported in the literature.^[1]

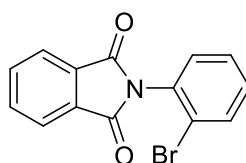
2-(2-Fluorophenyl)isoindoline-1,3-dione (**3p**)



3p

EtOAc/*n*-Hexane = 1/8 (eluent); white solid, 11% yield, 5.3 mg; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.98 – 7.95 (m, 2H), 7.82 – 7.78 (m, 2H), 7.49 – 7.41 (m, 1H), 7.37 (dd, $J = 10.4, 4.4$ Hz, 1H), 7.28 (dd, $J = 16.8, 8.4$ Hz, 2H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 166.5, 157.9 (d, $J = 251$ Hz), 134.4, 132.0, 130.8 (d, $J = 8$ Hz), 130.7, 129.8, 124.7 (d, $J = 4$ Hz), 123.9, 119.3 (d, $J = 13$ Hz), 116.7 (d, $J = 19$ Hz); $^{19}\text{F NMR}$ (376 MHz, CDCl_3) δ -118.70. The spectral data are consistent with those reported in the literature.^[6]

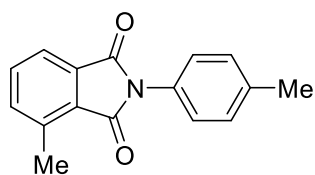
2-(2-Bromophenyl)isoindoline-1,3-dione (**3q**)



3q

EtOAc/*n*-Hexane = 1/8 (eluent); white solid, 18% yield, 10.8 mg; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 8.0 – 7.96 (m, 2H), 7.84 – 7.80 (m, 2H), 7.75 (d, $J = 8.0$ Hz, 1H), 7.48 (t, $J = 7.2$ Hz, 1H), 7.36 (t, $J = 8.0$ Hz, 2H). $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 166.6, 134.5, 133.6, 131.9, 131.4, 131.0, 130.9, 128.5, 124.0, 123.4. The spectral data are consistent with those reported in the literature.^[7]

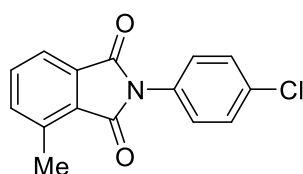
4-Methyl-2-(*p*-tolyl)isoindoline-1,3-dione (**3r**)



3r

EtOAc/*n*-Hexane = 1/8 (eluent); white solid, 64% yield, 32.1 mg; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.77 (d, $J = 7.2$ Hz, 1H), 7.63 (t, $J = 7.6$ Hz, 1H), 7.52 (d, $J = 7.6$ Hz, 1H), 7.30 (s, 4H), 2.74 (s, 3H), 2.41 (s, 3H). $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 168.2, 167.5, 138.4, 138.1, 136.7, 133.8, 132.3, 129.8, 129.1, 128.5, 126.6, 121.4, 21.3, 17.8. The spectral data are consistent with those reported in the literature.^[8]

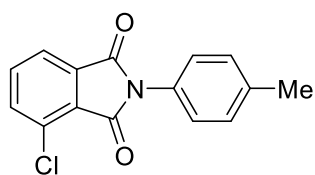
2-(4-Chlorophenyl)-4-methylisoindoline-1,3-dione (3s)



3s

EtOAc/*n*-Hexane = 1/8 (eluent); white solid, 50% yield, 27.1 mg; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.78 (d, $J = 7.2$ Hz, 1H), 7.65 (t, $J = 7.2$ Hz, 1H), 7.54 (d, $J = 7.6$ Hz, 1H), 7.47 (d, $J = 8.4$ Hz, 2H), 7.40 (d, $J = 8.4$ Hz, 2H), 2.74 (s, 3H). $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 167.7, 167.0, 138.6, 136.9, 134.0, 133.6, 132.0, 130.2, 129.2, 128.2, 127.7, 121.5, 17.7; HRMS ESI(m/z): $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{15}\text{H}_{11}\text{ClNO}_2$: 272.0473, found: 272.0470.

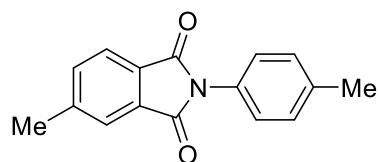
4-Chloro-2-(*p*-tolyl)isoindoline-1,3-dione (3t)



3t

EtOAc/*n*-Hexane = 1/8 (eluent); white solid, 78% yield, 42.3 mg; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.86 (p, $J = 4.4$ Hz, 1H), 7.70 (d, $J = 4.4$ Hz, 2H), 7.30 (s, 4H), 2.41 (s, 3H). $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 166.0, 165.0, 138.4, 136.1, 135.2, 133.9, 131.8, 129.8, 128.6, 127.5, 126.5, 122.2. The spectral data are consistent with those reported in the literature.^[9]

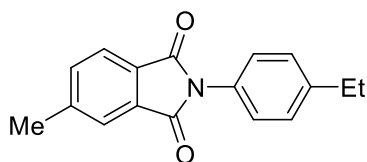
5-Methyl-2-(*p*-tolyl)isoindoline-1,3-dione (3u)



3u

EtOAc/*n*-Hexane = 1/8 (eluent); white solid, 67% yield, 33.6 mg; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.83 (d, $J = 7.4$ Hz, 1H), 7.75 (s, 1H), 7.57 (d, $J = 7.4$ Hz, 1H), 7.33 – 7.28 (, 4H), 2.55 (s, 3H), 2.41 (s, 3H). $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 167.7, 167.6, 145.7, 138.1, 134.9, 132.2, 129.8, 129.2, 129.1, 126.5, 124.2, 123.6, 22.1, 21.2. The spectral data are consistent with those reported in the literature.^[8]

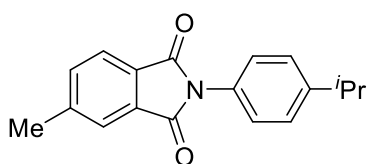
2-(4-Ethylphenyl)-5-methylisoindoline-1,3-dione (3v)



3v

EtOAc/*n*-Hexane = 1/8 (eluent); white solid, 65% yield, 34.5 mg; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.83 (d, $J = 7.6$ Hz, 1H), 7.75 (s, 1H), 7.58 (s, 1H), 7.33 (s, 4H), 2.70 (q, $J = 7.4$ Hz, 2H), 2.55 (s, 3H), 1.27 (t, $J = 7.6$ Hz, 3H). $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 167.6, 167.5, 145.6, 144.2, 134.8, 132.1, 129.2, 128.5, 126.4, 124.2, 123.6, 28.6, 22.0, 15.4. The spectral data are consistent with those reported in the literature.^[10]

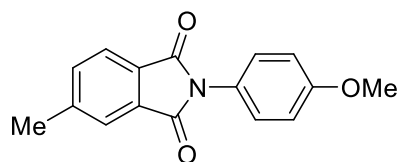
2-(4-Isopropylphenyl)-5-methylisoindoline-1,3-dione (3w)



3w

EtOAc/*n*-Hexane = 1/8 (eluent); white solid, 57% yield, 31.8 mg; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.82 (d, $J = 7.6$ Hz, 1H), 7.75 (s, 1H), 7.57 (d, $J = 7.6$ Hz, 1H), 7.39 – 7.31 (m, 4H), 2.96 (m, $J = 6.8$ Hz, 1H), 2.54 (s, 3H), 1.28 (d, $J = 6.8$ Hz, 6H). $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 167.6, 167.5, 148.7, 145.6, 134.9, 132.2, 129.2, 129.2, 127.1, 126.3, 124.2, 123.6, 33.9, 23.9, 22.0. The spectral data are consistent with those reported in the literature.^[11]

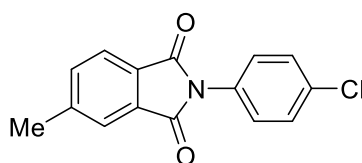
2-(4-Methoxyphenyl)-5-methylisoindoline-1,3-dione (3x)



3x

EtOAc/*n*-Hexane = 1/5 (eluent); white solid, 78% yield, 41.7 mg; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.82 (d, $J = 7.6$ Hz, 1H), 7.74 (s, 1H), 7.57 (d, $J = 7.6$ Hz, 1H), 7.33 (d, $J = 8.8$ Hz, 2H), 7.02 (d, $J = 8.8$ Hz, 2H), 3.85 (s, 3H), 2.54 (s, 3H). $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 167.8, 167.7, 159.2, 145.7, 134.9, 132.2, 129.2, 128.0, 124.4, 124.2, 123.6, 114.5, 55.5, 22.1. The spectral data are consistent with those reported in the literature.^[11]

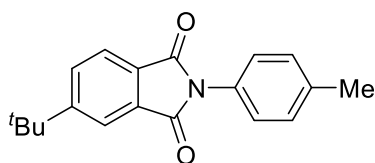
2-(4-Chlorophenyl)-5-methylisoindoline-1,3-dione (3y)



3y

EtOAc/*n*-Hexane = 1/8 (eluent); white solid, 54% yield, 29.3 mg; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.84 (d, $J = 7.6$ Hz, 1H), 7.76 (s, 1H), 7.59 (d, $J = 7.6$ Hz, 1H), 7.48 (d, $J = 8.8$ Hz, 2H), 7.41 (d, $J = 8.8$ Hz, 2H), 2.56 (s, 3H). $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 167.2, 167.1, 146.0, 135.2, 133.7, 132.0, 130.3, 129.3, 129.0, 127.7, 124.4, 123.8, 22.1. The spectral data are consistent with those reported in the literature.^[11]

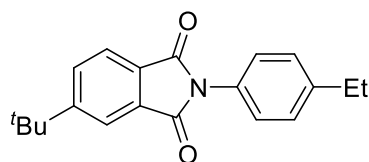
5-(*tert*-Butyl)-2-(*p*-tolyl)isoindoline-1,3-dione (3z)



3z

EtOAc/*n*-Hexane = 1/8 (eluent); white solid, 47% yield, 27.6 mg; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.98 (d, $J = 1.6$ Hz, 1H), 7.87 (d, $J = 8.0$ Hz, 1H), 7.80 (dd, $J = 8.0, 1.6$ Hz, 1H), 7.33 – 7.28 (m, 4H), 2.41 (s, 3H), 1.40 (s, 9H). $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 167.9, 167.5, 159.0, 138.1, 132.1, 131.4, 129.8, 129.2, 129.1, 126.5, 123.6, 120.9, 35.9, 31.2, 21.3. The spectral data are consistent with those reported in the literature.^[8]

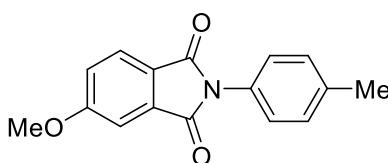
5-(*tert*-Butyl)-2-(4-ethylphenyl)isoindoline-1,3-dione (3a')



3a'

EtOAc/*n*-Hexane = 1/8 (eluent); white solid, 50% yield, 30.7 mg; ^1H NMR (400 MHz, CDCl_3) δ 7.99 (s, 1H), 7.87 (d, $J = 8.0$ Hz, 1H), 7.80 (d, $J = 8.0$ Hz, 1H), 7.33 (s, 4H), 2.71 (q, $J = 7.6$ Hz, 2H), 1.41 (s, 9H), 1.27 (t, $J = 7.6$ Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 167.9, 167.5, 159.0, 144.2, 132.0, 131.3, 129.2, 129.1, 128.6, 126.5, 123.5, 120.9, 35.8, 31.1, 28.6, 15.4.; HRMS ESI(m/z): $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{20}\text{H}_{22}\text{NO}_2$: 308.1645, found: 308.1643.

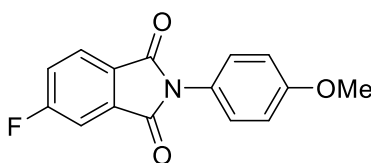
5-Methoxy-2-(*p*-tolyl)isoindoline-1,3-dione (3b')



3b'

EtOAc/*n*-Hexane = 1/5 (eluent); white solid, 45% yield, 24.0 mg; ^1H NMR (400 MHz, CDCl_3) δ 7.85 (d, $J = 8.4$ Hz, 1H), 7.42 (d, $J = 2.4$ Hz, 1H), 7.30 (s, 4H), 7.22 (dd, $J = 8.4, 2.4$ Hz, 1H), 3.95 (s, 3H), 2.41 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 167.3, 167.2, 164.9, 138.0, 134.4, 129.7, 129.0, 126.4, 125.4, 123.6, 120.3, 108.1, 56.1, 21.2. The spectral data are consistent with those reported in the literature.^[8]

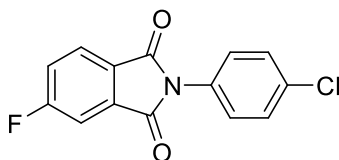
5-Fluoro-2-(4-methoxyphenyl)isoindoline-1,3-dione (3c')



3c'

EtOAc/*n*-Hexane = 1/5 (eluent); white solid, 81% yield, 43.9 mg; ^1H NMR (400 MHz, CDCl_3) δ 7.95 (dd, $J = 8.0, 4.4$ Hz, 1H), 7.61 (dd, $J = 7.2, 2.4$ Hz, 1H), 7.45 (td, $J = 8.4, 2.4$ Hz, 1H), 7.35 – 7.29 (m, 2H), 7.02 (dd, $J = 9.6, 2.4$ Hz, 2H), 3.85 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 166.6 (d, $J = 256$ Hz), 166.5, 166.1, 159.3, 134.7 (d, $J = 9$ Hz), 127.8, 127.6 (d, $J = 3$ Hz), 126.1 (d, $J = 9$ Hz), 124.0, 121.4 (d, $J = 24$ Hz), 114.5, 111.4 (d, $J = 25$ Hz), 55.5; ^{19}F NMR (376 MHz, CDCl_3) δ -101.23. The spectral data are consistent with those reported in the literature.^[12]

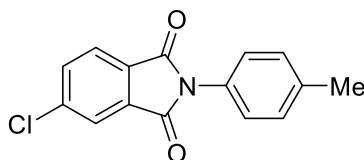
2-(4-Chlorophenyl)-5-fluoroisoindoline-1,3-dione (3d')



3d'

EtOAc/*n*-Hexane = 1/8 (eluent); white solid, 74% yield, 40.7 mg; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.97 (dd, $J = 8.0, 4.4$ Hz, 1H), 7.64 (dd, $J = 6.8, 1.6$ Hz, 1H), 7.49 (d, $J = 8.8$ Hz, 3H), 7.40 (d, $J = 8.8$ Hz, 2H). $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 168.0, 165.9, 165.7, 165.4, 134.5, 134.4, 134.0, 130.0, 129.4, 127.6, 127.4, 127.4, 126.4, 126.3, 121.8, 121.6, 111.8, 111.5. $^{19}\text{F NMR}$ (376 MHz, CDCl_3) δ -100.58. The spectral data are consistent with those reported in the literature.^[12]

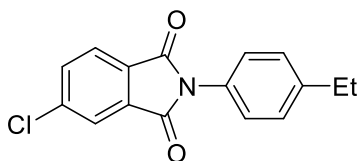
5-Chloro-2-(*p*-tolyl)isoindoline-1,3-dione (3e')



3e'

EtOAc/*n*-Hexane = 1/8 (eluent); white solid, 70% yield, 42.8 mg; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.94 – 7.85 (m, 2H), 7.78 – 7.71 (m, 1H), 7.29 (dd, $J = 13.2, 4.8$ Hz, 4H), 2.41 (s, 3H). $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 166.5, 166.2, 141.1, 138.5, 134.5, 133.5, 129.9, 129.2, 128.7, 126.4, 125.0, 124.2, 21.3. The spectral data are consistent with those reported in the literature; HRMS ESI(m/z): $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{15}\text{H}_{11}\text{ClNO}_2$: 242.0473, found: 242.0475.

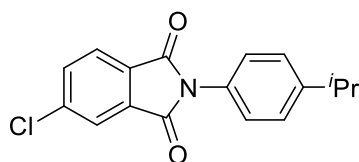
5-Chloro-2-(4-ethylphenyl)isoindoline-1,3-dione (3f')



3f'

EtOAc/*n*-Hexane = 1/8 (eluent); white solid, 78% yield, 44.5 mg; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.95 – 7.85 (m, 2H), 7.74 (dd, $J = 8.0, 1.6$ Hz, 1H), 7.39 – 7.30 (m, 4H), 2.71 (q, $J = 7.6$ Hz, 2H), 1.27 (t, $J = 7.6$ Hz, 3H). $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 166.5, 166.2, 144.7, 141.1, 134.4, 133.5, 129.9, 128.8, 128.7, 126.4, 125.0, 124.1, 28.6, 15.5; HRMS ESI(m/z): $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{16}\text{H}_{13}\text{ClNO}_2$: 286.0629, found: 286.0627.

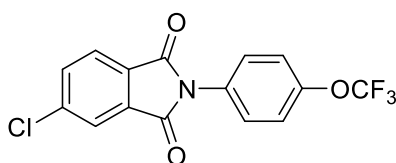
5-Chloro-2-(4-isopropylphenyl)isoindoline-1,3-dione (3g')



3g'

EtOAc/*n*-Hexane = 1/8 (eluent); white solid, 67% yield, 40.1 mg; ^1H NMR (400 MHz, CDCl_3) δ 7.93 – 7.85 (m, 2H), 7.74 (d, $J = 8.0$ Hz, 1H), 7.34 (q, $J = 8.4$ Hz, 4H), 2.97 (hept, $J = 6.8$ Hz, 1H), 1.28 (d, $J = 6.8$ Hz, 6H). ^{13}C NMR (100 MHz, CDCl_3) δ 166.5, 166.2, 149.2, 141.1, 134.4, 133.5, 129.9, 128.9, 127.3, 126.4, 125.0, 124.1, 34.0, 23.9; HRMS ESI(m/z): $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{17}\text{H}_{15}\text{ClNO}_2$: 300.0786, found: 300.0791.

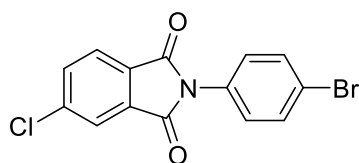
5-Chloro-2-(4-(trifluoromethoxy)phenyl)isoindoline-1,3-dione (3h')



3h'

EtOAc/*n*-Hexane = 1/5 (eluent); white solid, 79% yield, 53.9 mg; ^1H NMR (400 MHz, CDCl_3) δ 7.98 – 7.85 (m, 2H), 7.78 (d, $J = 8.0$ Hz, 1H), 7.50 (d, $J = 8.8$ Hz, 2H), 7.36 (d, $J = 8.8$ Hz, 2H). ^{13}C NMR (100 MHz, CDCl_3) δ 166.1, 165.8, 148.5, 141.4, 134.8, 133.2, 129.9, 129.6, 127.8, 125.2, 124.4, 121.7, 119.1. ^{19}F NMR (376 MHz, CDCl_3) δ -57.86; HRMS ESI(m/z): $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{15}\text{H}_8\text{ClF}_3\text{NO}_3$: 342.0139, found: 342.0135.

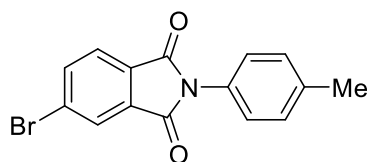
2-(4-Bromophenyl)-5-chloroisoindoline-1,3-dione (3i')



3i'

EtOAc/*n*-Hexane = 1/8 (eluent); white solid, 67% yield, 45.0 mg; ^1H NMR (400 MHz, CDCl_3) δ 7.90 (dd, $J = 11.6, 4.8$ Hz, 2H), 7.76 (dd, $J = 8.0, 1.6$ Hz, 1H), 7.67 – 7.60 (m, 2H), 7.37 – 7.30 (m, 2H). ^{13}C NMR (100 MHz, CDCl_3) δ 166.0, 165.7, 141.4, 134.7, 133.3, 132.4, 130.5, 129.6, 127.9, 125.1, 124.3, 122.1; HRMS ESI(m/z): $[\text{M}+\text{H}]^+$ calcd. for $\text{C}_{14}\text{H}_8\text{BrClNO}_2$: 335.9421, found: 335.9417.

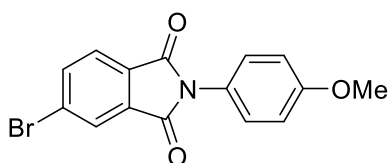
5-Bromo-2-(*p*-tolyl)isoindoline-1,3-dione (3j')



3j'

EtOAc/*n*-Hexane = 1/8 (eluent); white solid, 73% yield, 46.0 mg; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 8.08 (d, $J = 1.2$ Hz, 1H), 7.92 (dd, $J = 8.0, 1.6$ Hz, 1H), 7.81 (d, $J = 8.0$ Hz, 1H), 7.33 – 7.27 (m, 4H), 2.41 (s, 3H). $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 166.6, 166.1, 138.4, 137.3, 133.4, 130.3, 129.8, 129.2, 128.6, 127.0, 126.3, 125.0, 21.2. The spectral data are consistent with those reported in the literature.^[13]

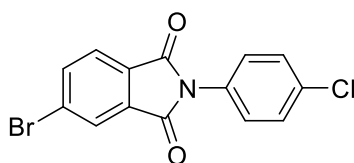
5-Bromo-2-(4-methoxyphenyl)isoindoline-1,3-dione (3k')



3k'

EtOAc/*n*-Hexane = 1/5 (eluent); white solid, 80% yield, 53.0 mg; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 8.07 (s, 1H), 7.95 – 7.88 (m, 1H), 7.80 (d, $J = 8.0$ Hz, 1H), 7.32 (d, $J = 8.8$ Hz, 2H), 7.02 (d, $J = 8.8$ Hz, 2H), 3.85 (s, 3H). $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 166.7, 166.2, 159.3, 137.3, 133.4, 130.3, 129.2, 127.8, 127.0, 125.0, 123.9, 114.5, 55.5. The spectral data are consistent with those reported in the literature.^[13]

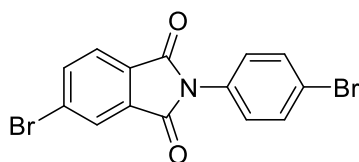
5-bromo-2-(4-chlorophenyl)isoindoline-1,3-dione (3l')



3l'

EtOAc/*n*-Hexane = 1/8 (eluent); white solid, 74% yield, 49.7 mg; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 8.09 (d, $J = 1.6$ Hz, 1H), 7.94 (dd, $J = 8.0, 1.6$ Hz, 1H), 7.82 (d, $J = 8.0$ Hz, 1H), 7.51 – 7.45 (m, 2H), 7.42 – 7.37 (m, 2H). $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 166.2, 165.7, 137.7, 134.1, 133.2, 130.1, 129.9, 129.6, 129.4, 127.6, 127.2, 125.2. The spectral data are consistent with those reported in the literature.^[9]

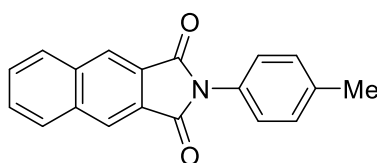
5-Bromo-2-(4-bromophenyl)isoindoline-1,3-dione (3m')



3m'

EtOAc/*n*-Hexane = 1/8 (eluent); white solid, 71% yield, 53.8 mg; ^1H NMR (400 MHz, CDCl_3) δ 8.09 (d, J = 1.6 Hz, 1H), 7.94 (dd, J = 8.0, 1.6 Hz, 1H), 7.82 (d, J = 8.0 Hz, 1H), 7.66 – 7.62 (m, 2H), 7.36 – 7.31 (m, 2H). ^{13}C NMR (100 MHz, CDCl_3) δ 166.1, 165.6, 137.7, 133.2, 132.4, 130.4, 130.1, 129.6, 127.9, 127.2, 125.3, 122.1. The spectral data are consistent with those reported in the literature.^[14]

2-(*p*-Tolyl)-1H-benzo[*f*]isoindole-1,3(2H)-dione (3n')



3n'

EtOAc/*n*-Hexane = 1/8 (eluent); white solid, 62% yield, 35.6 mg; ^1H NMR (400 MHz, CDCl_3) δ 8.45 (s, 2H), 8.14 – 8.05 (m, 2H), 7.78 – 7.69 (m, 2H), 7.36 (t, J = 6.4 Hz, 4H), 2.43 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 167.3, 138.4, 135.7, 130.4, 129.8, 129.4, 129.2, 127.5, 126.5, 125.3, 21.30. The spectral data are consistent with those reported in the literature.^[8]

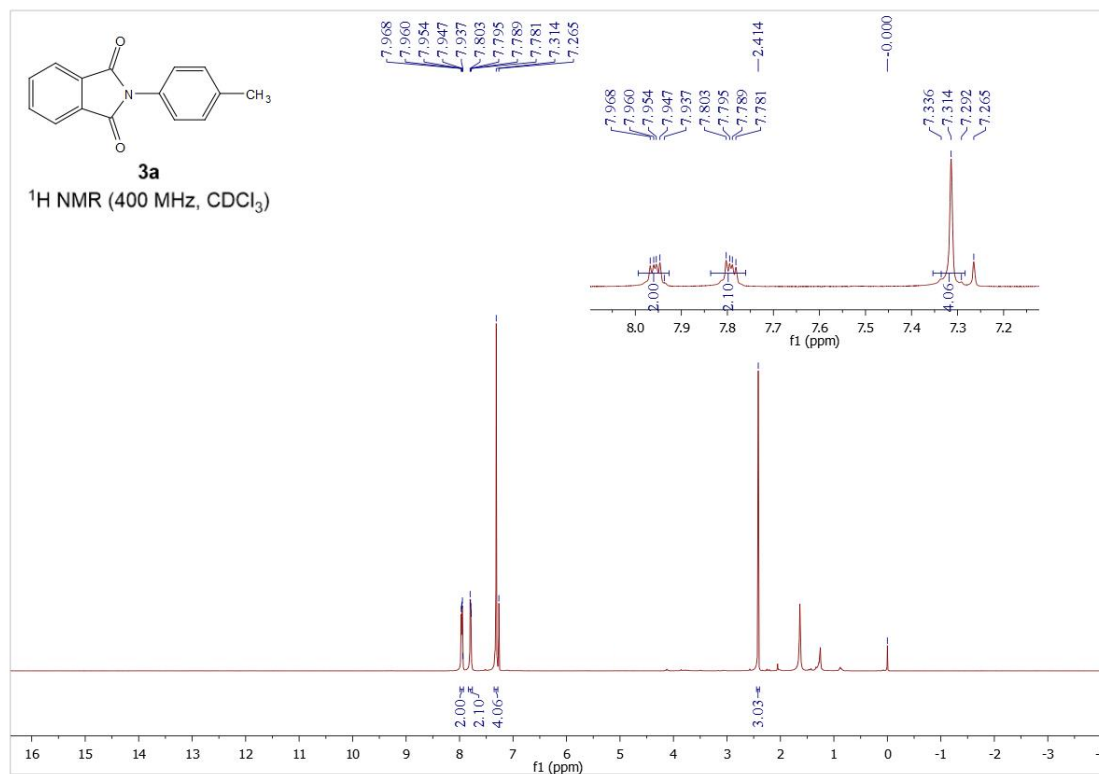
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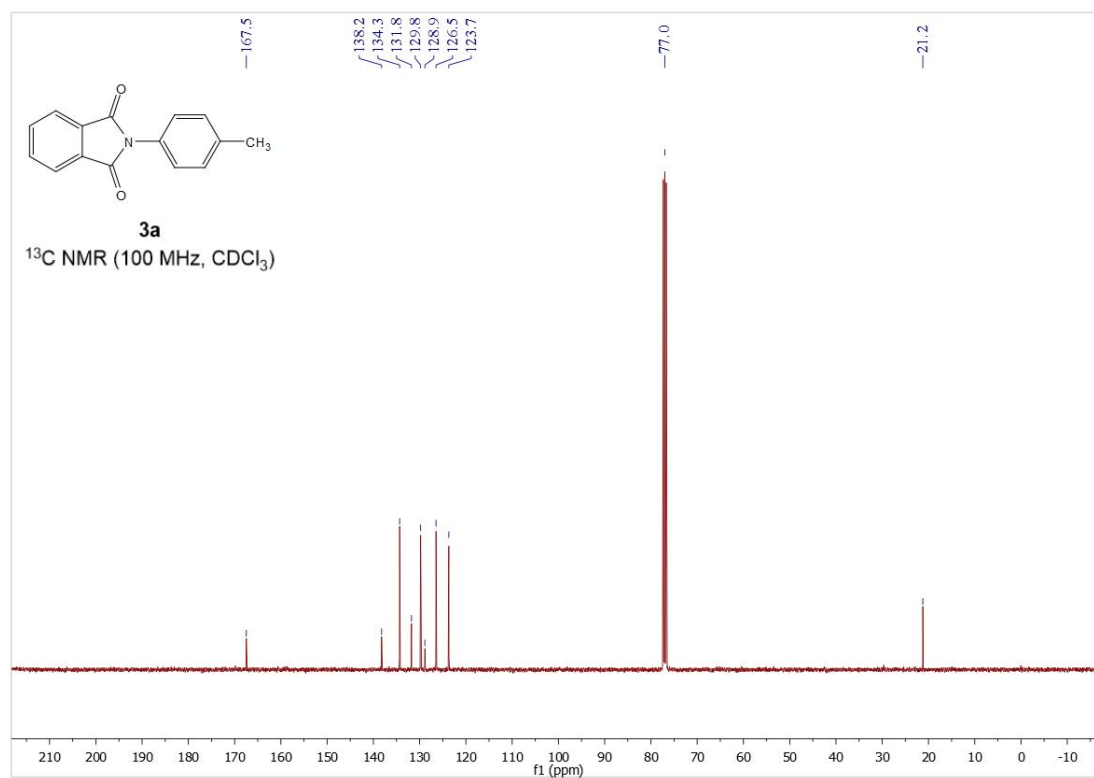
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8. NMR spectra of products

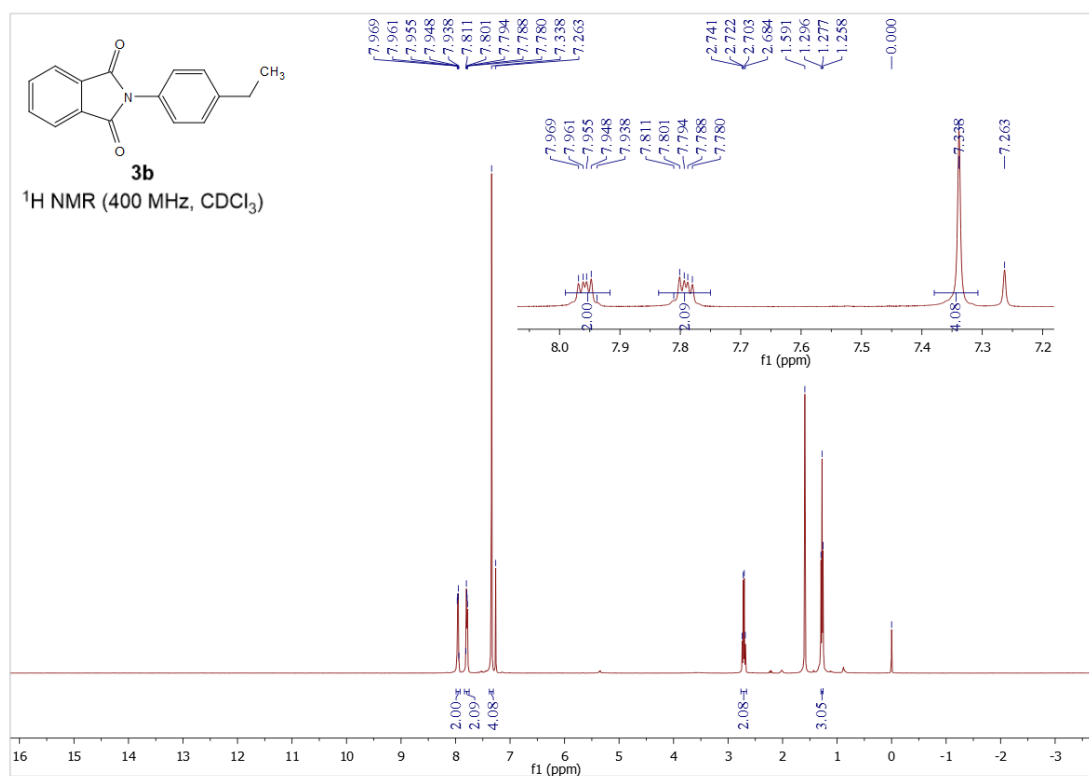
^1H NMR of Compound **3a** (400 MHz, CDCl_3)



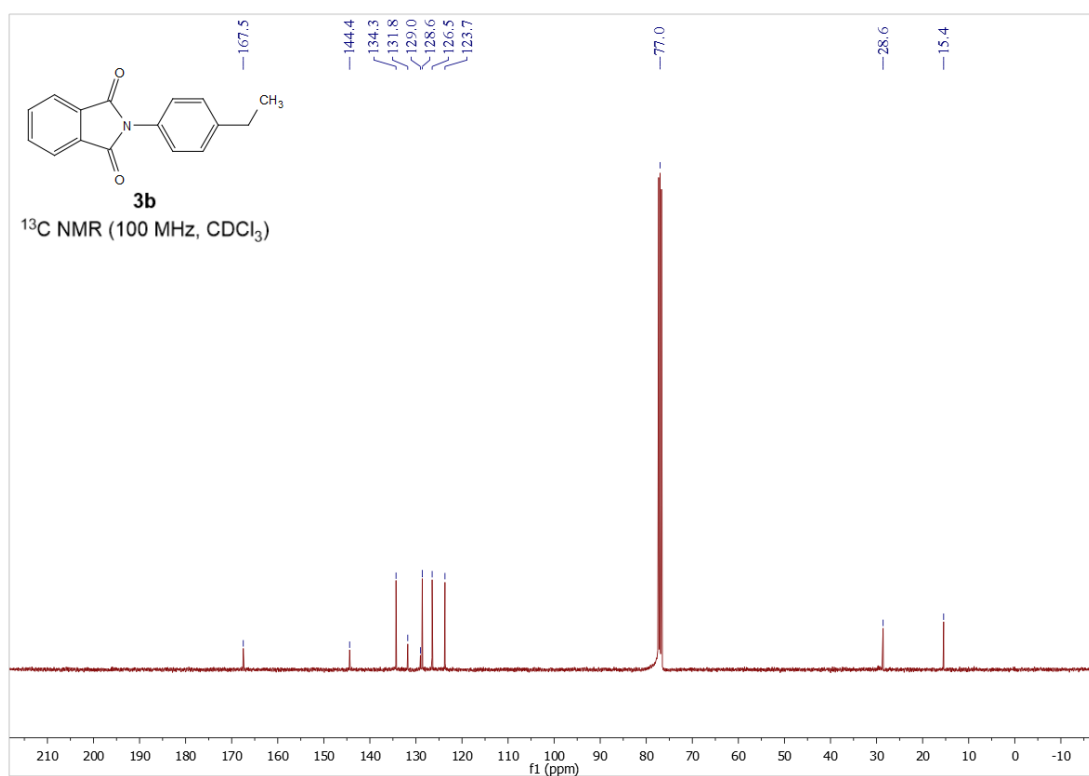
^{13}C NMR of Compound **3a** (100 MHz, CDCl_3)



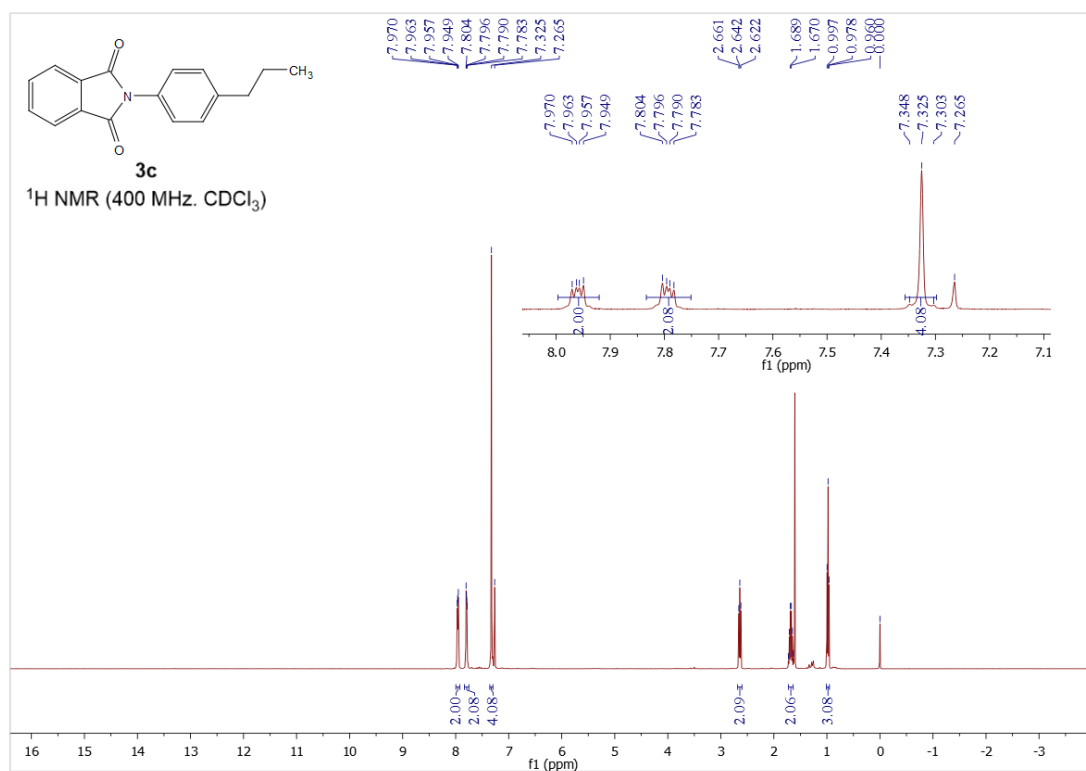
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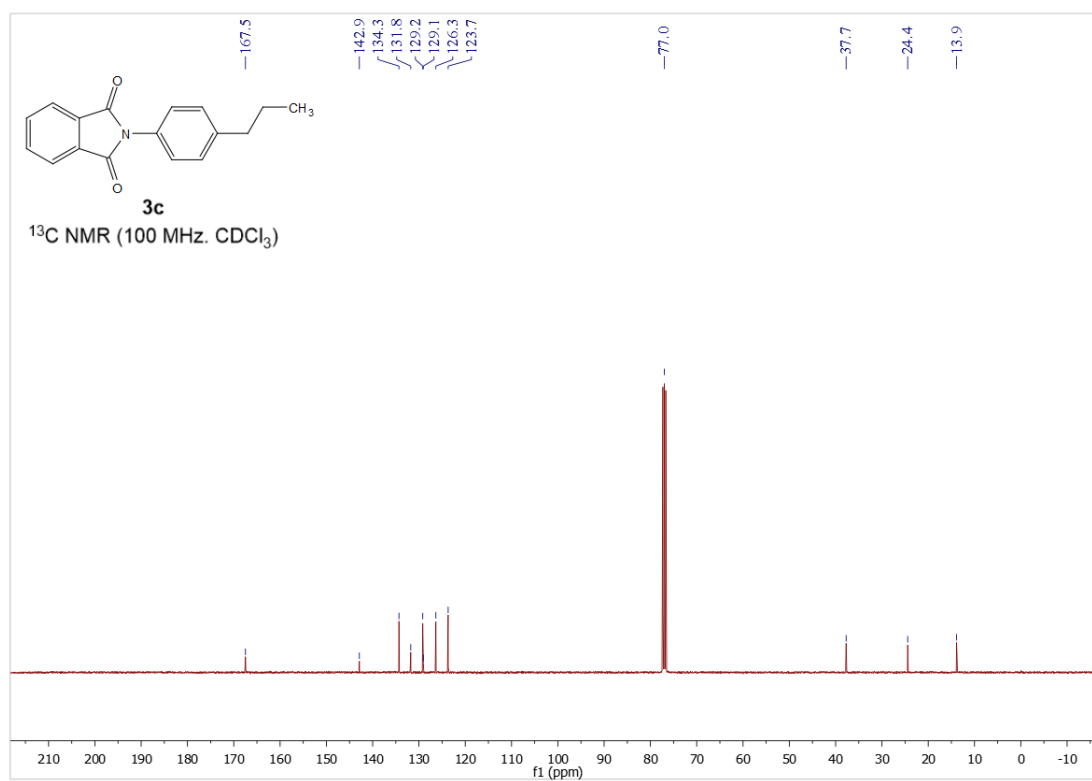
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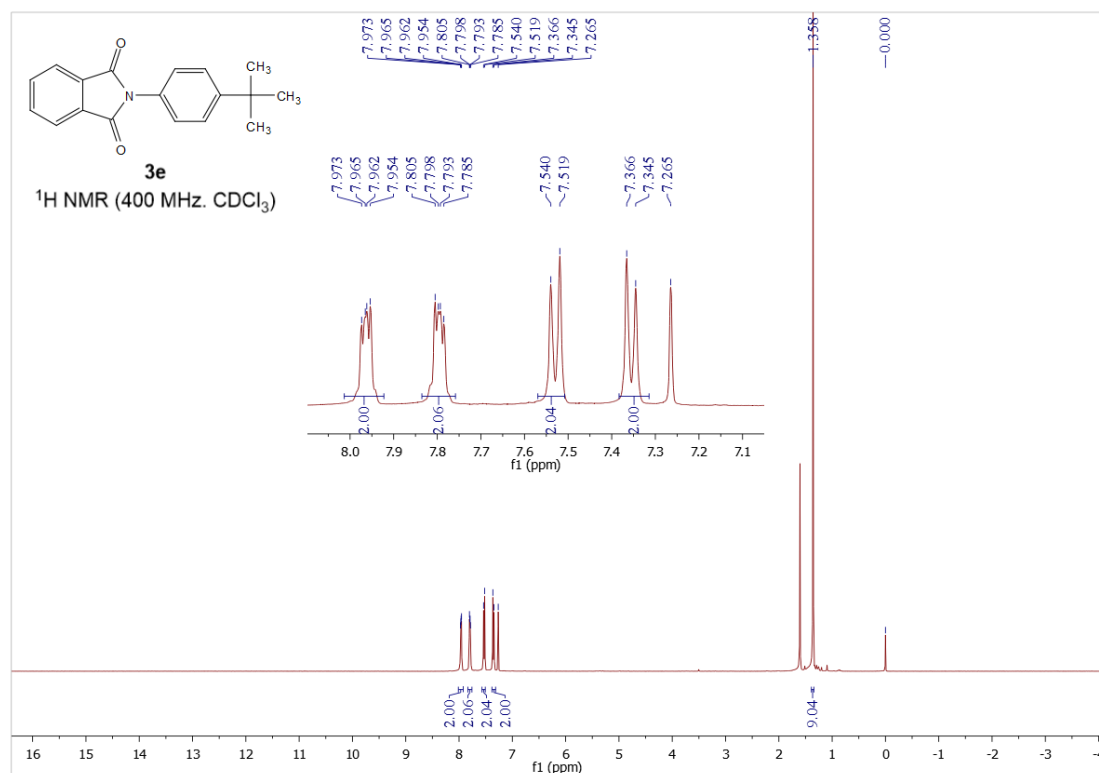
¹H NMR of Compound **3c** (400 MHz, CDCl₃)



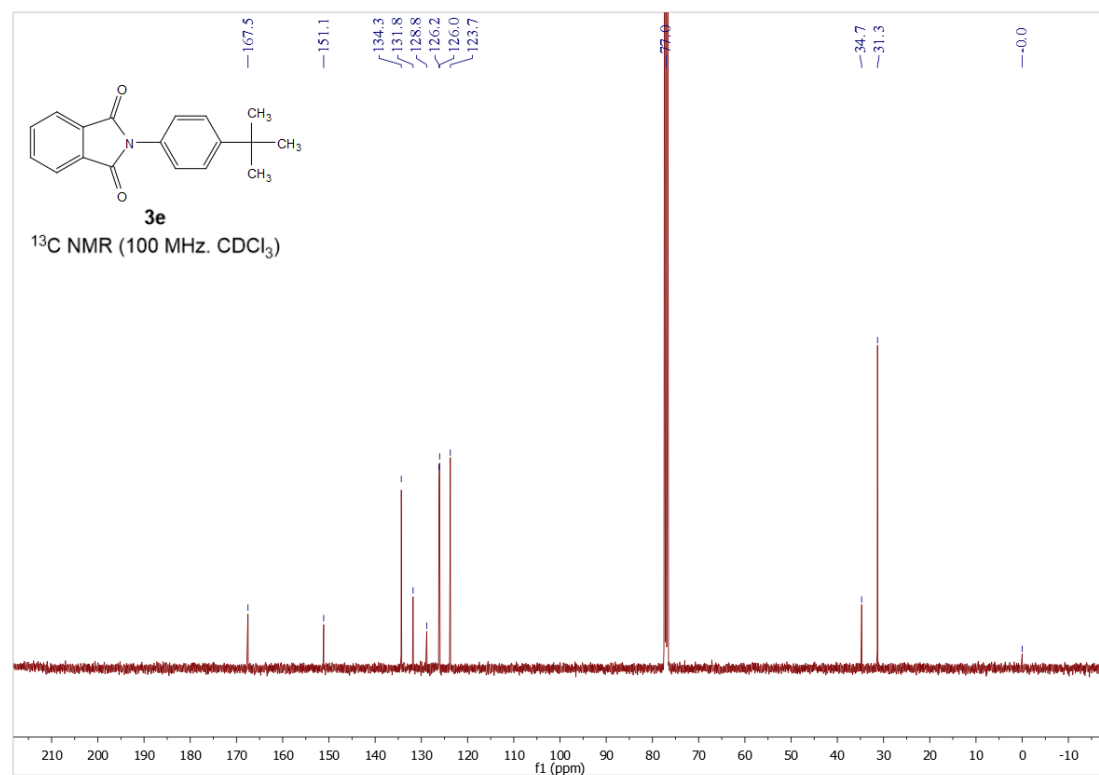
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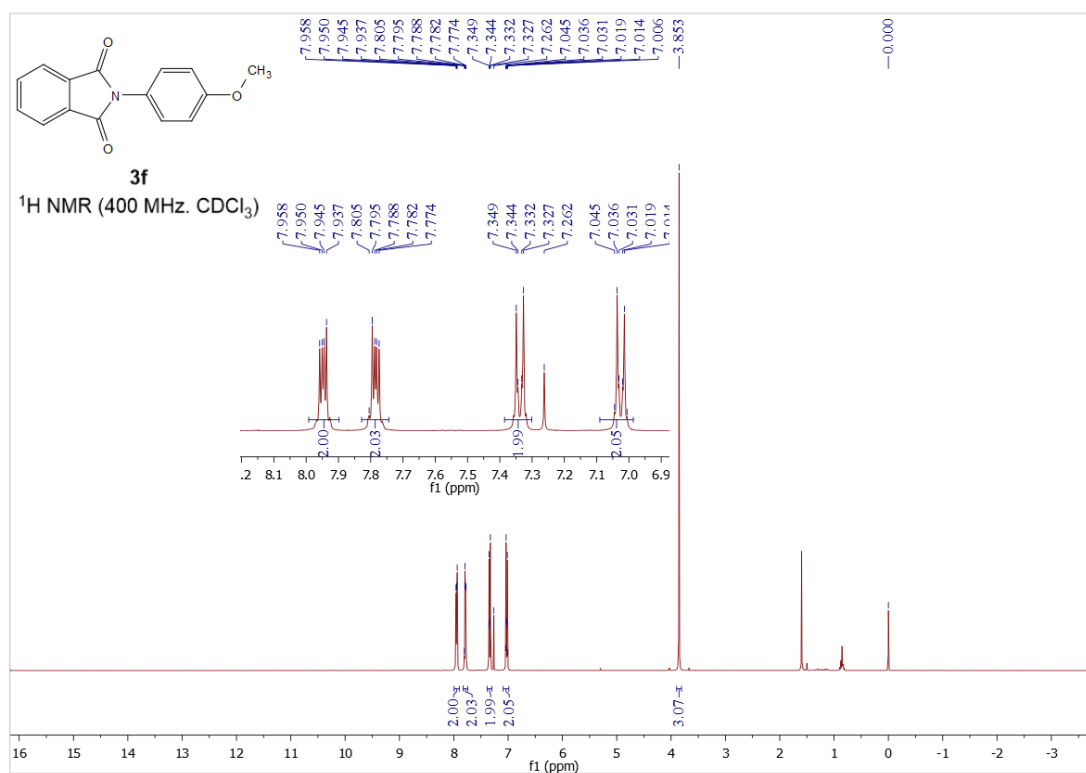
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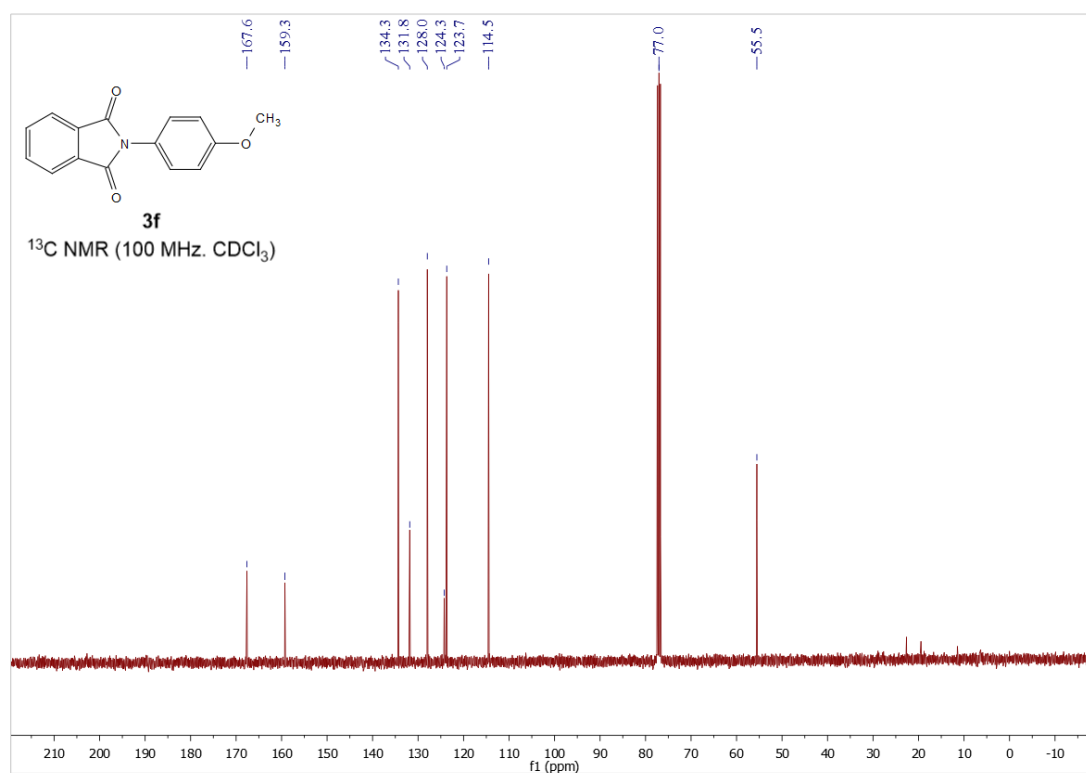
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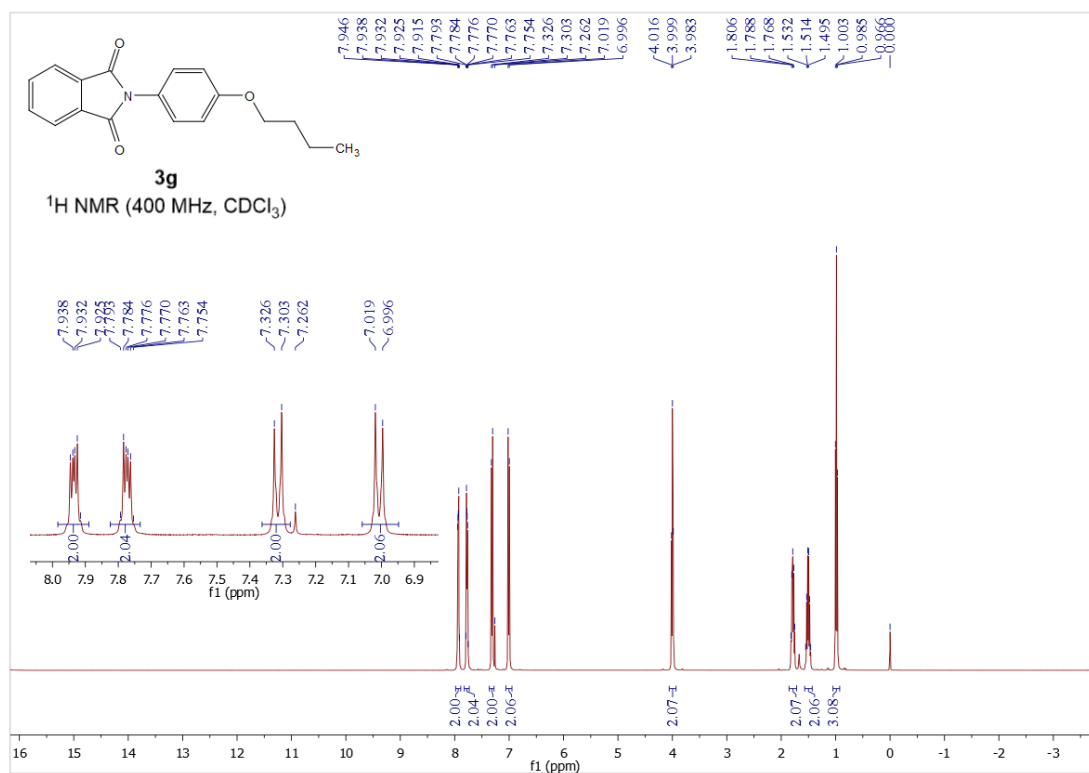
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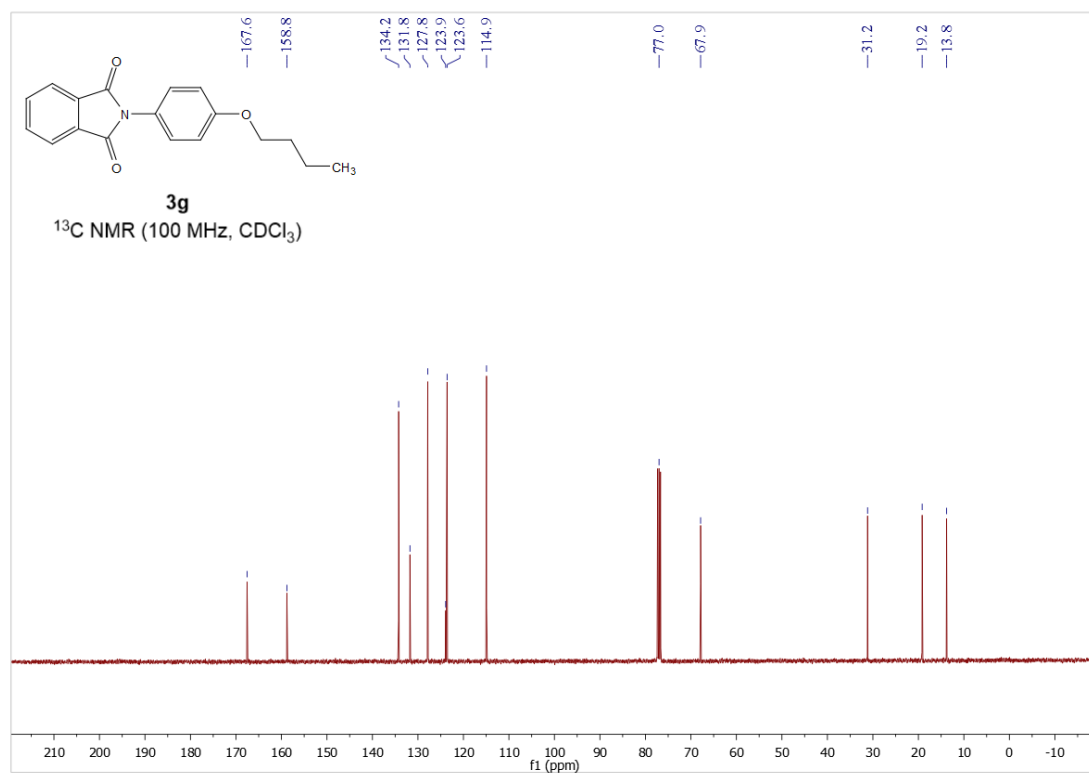
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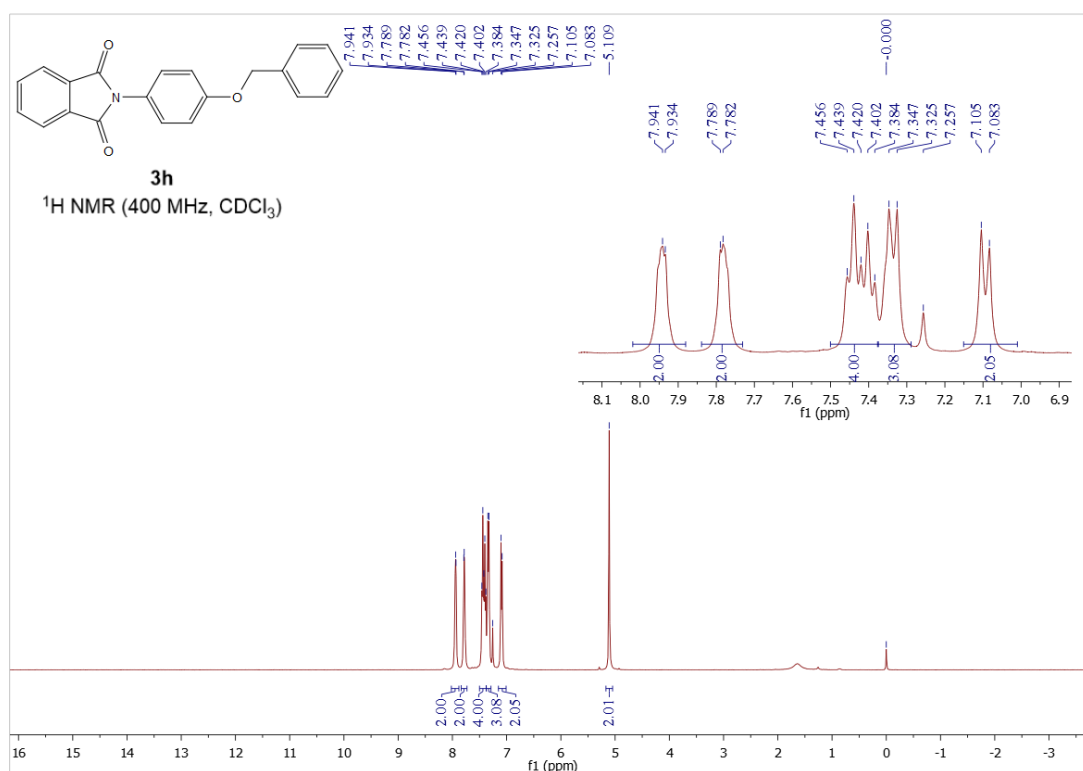
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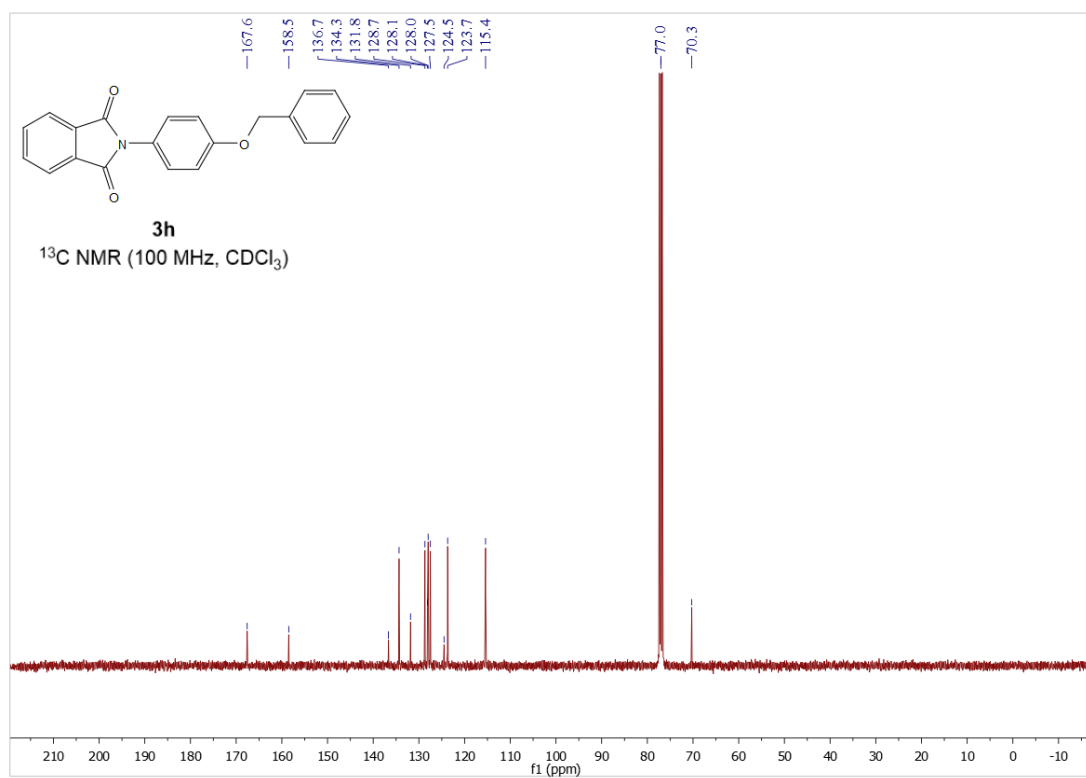
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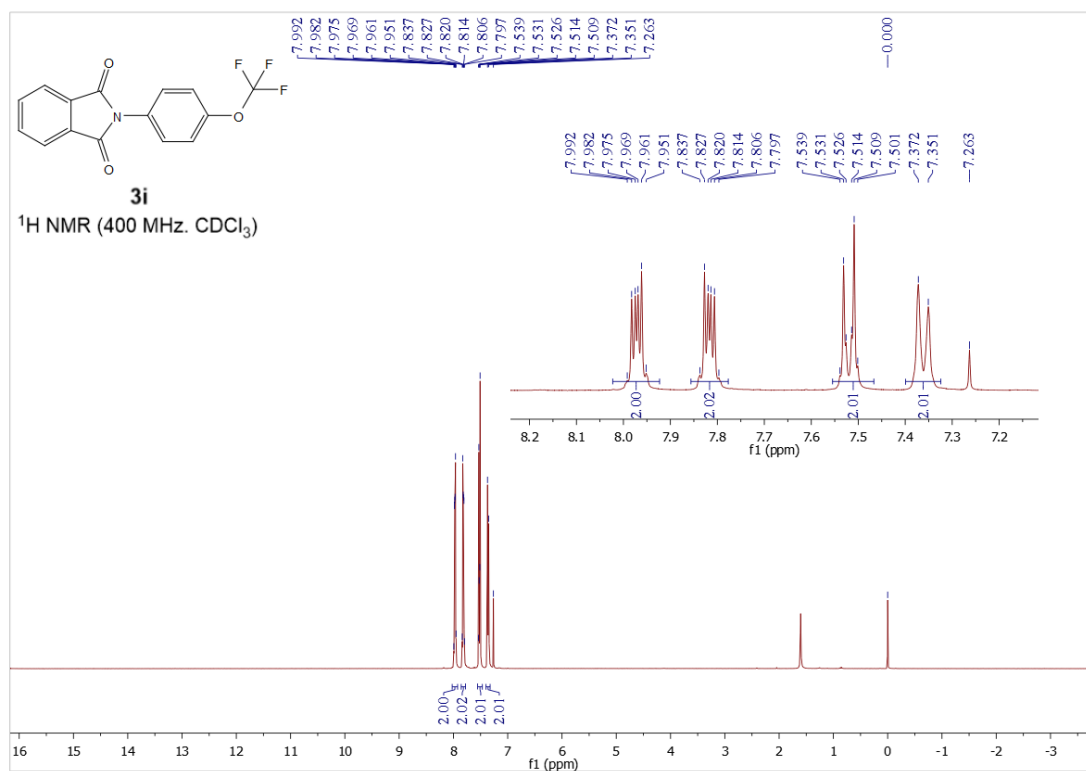
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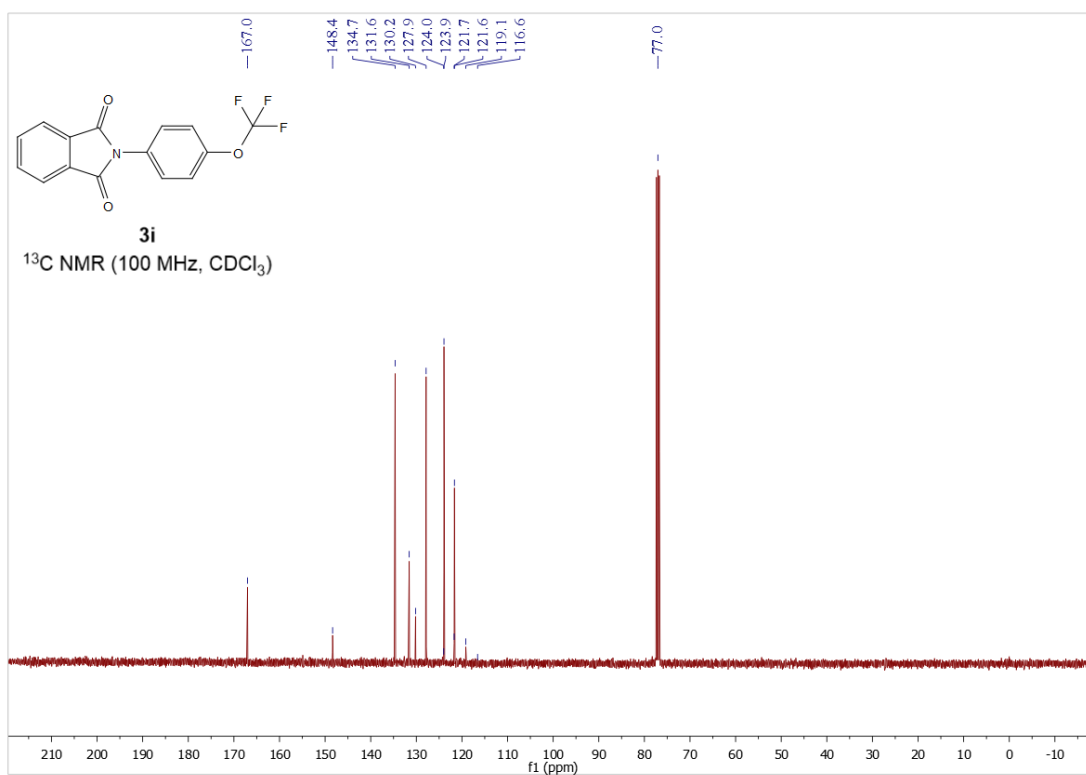
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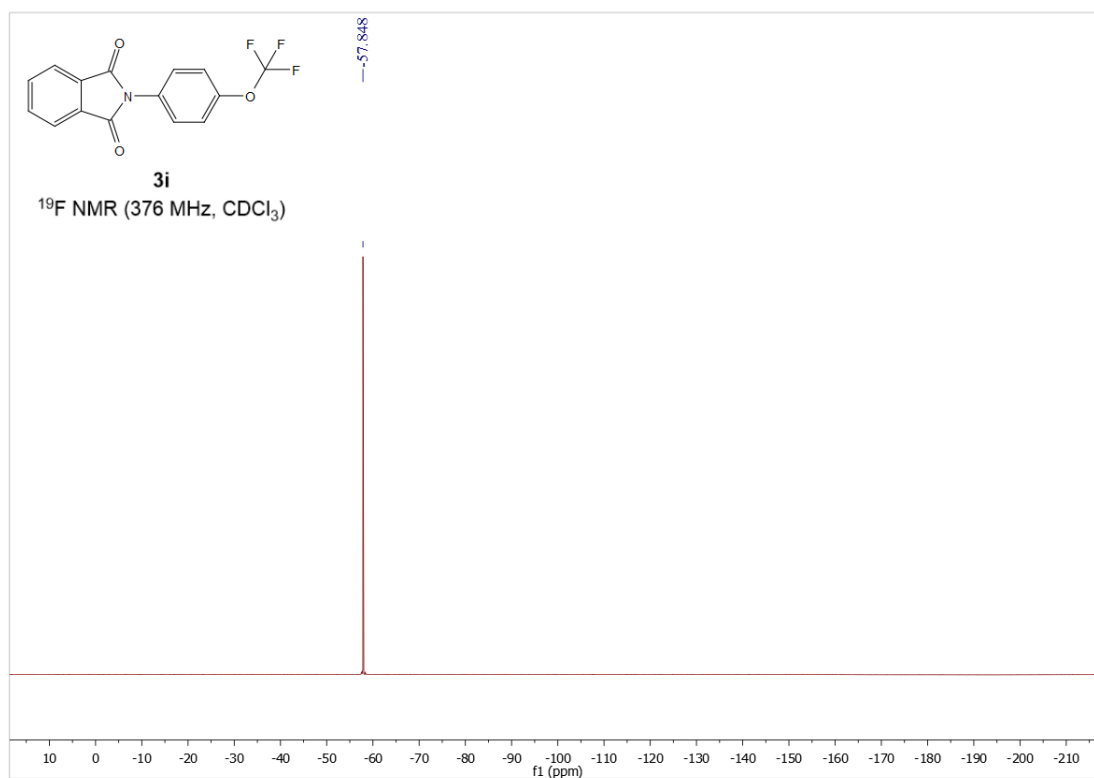
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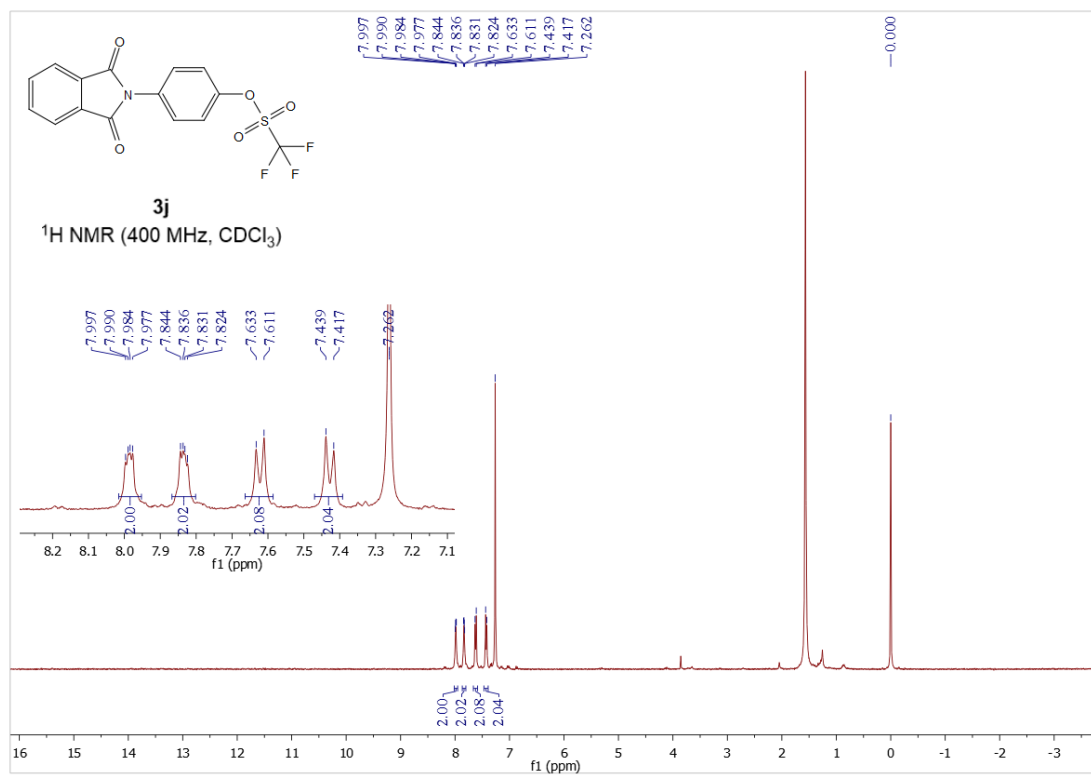
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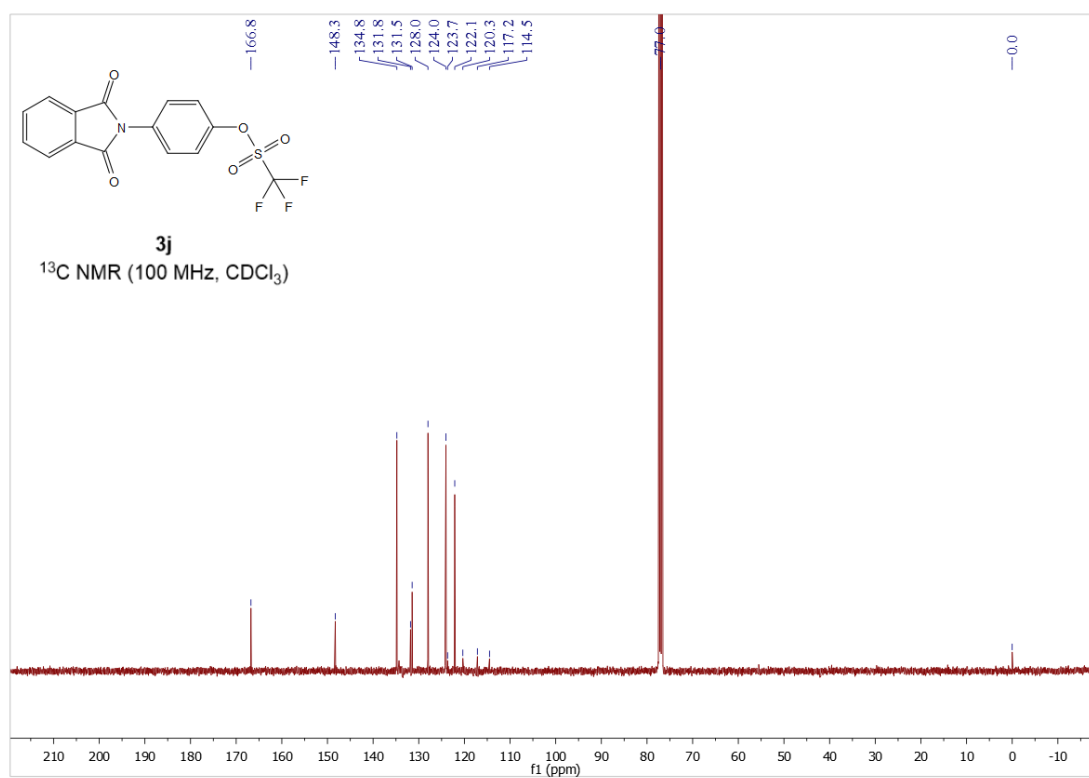
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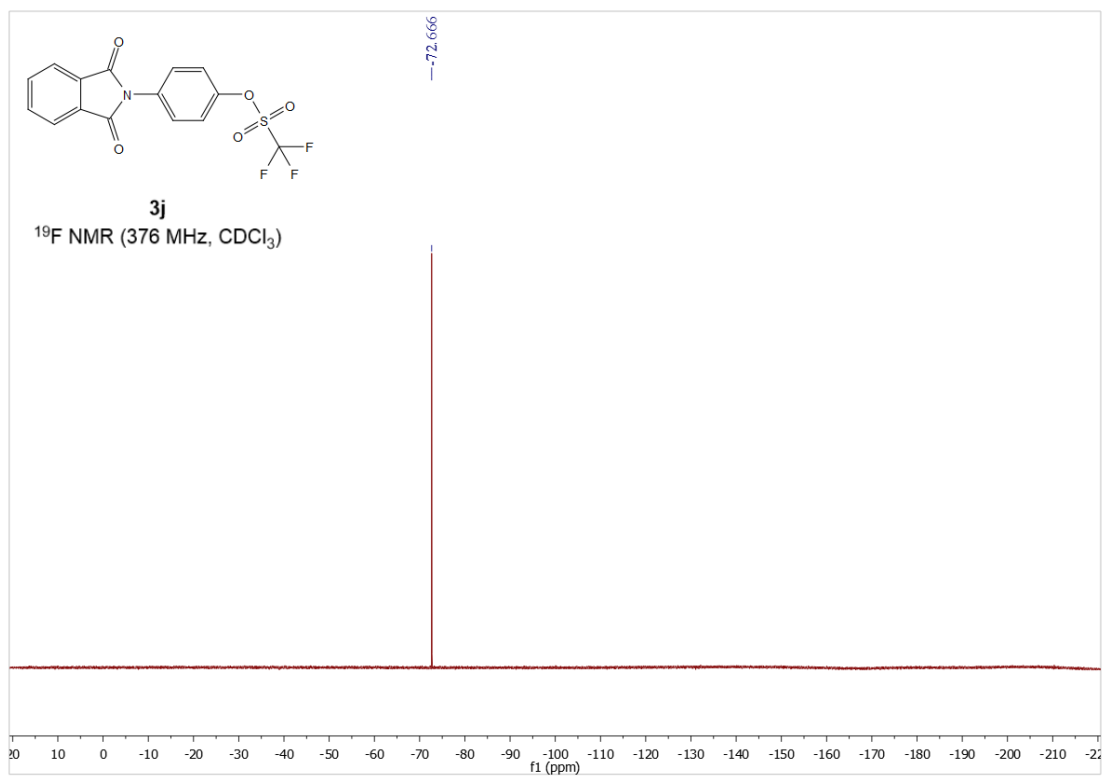
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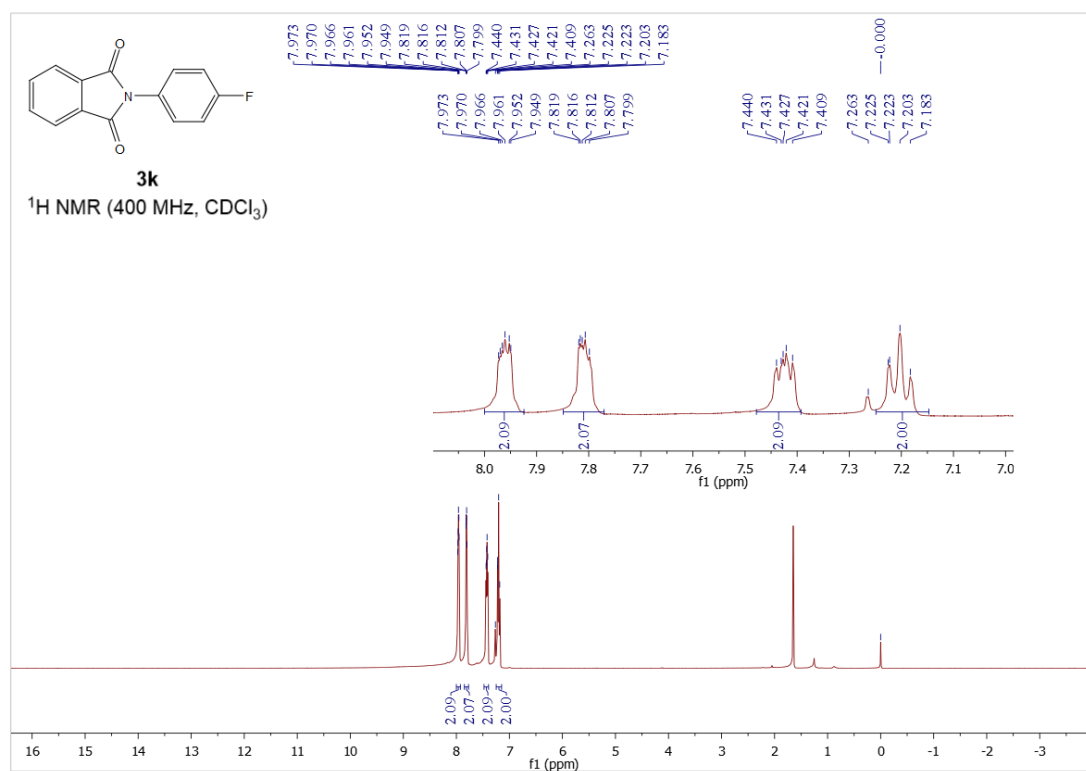
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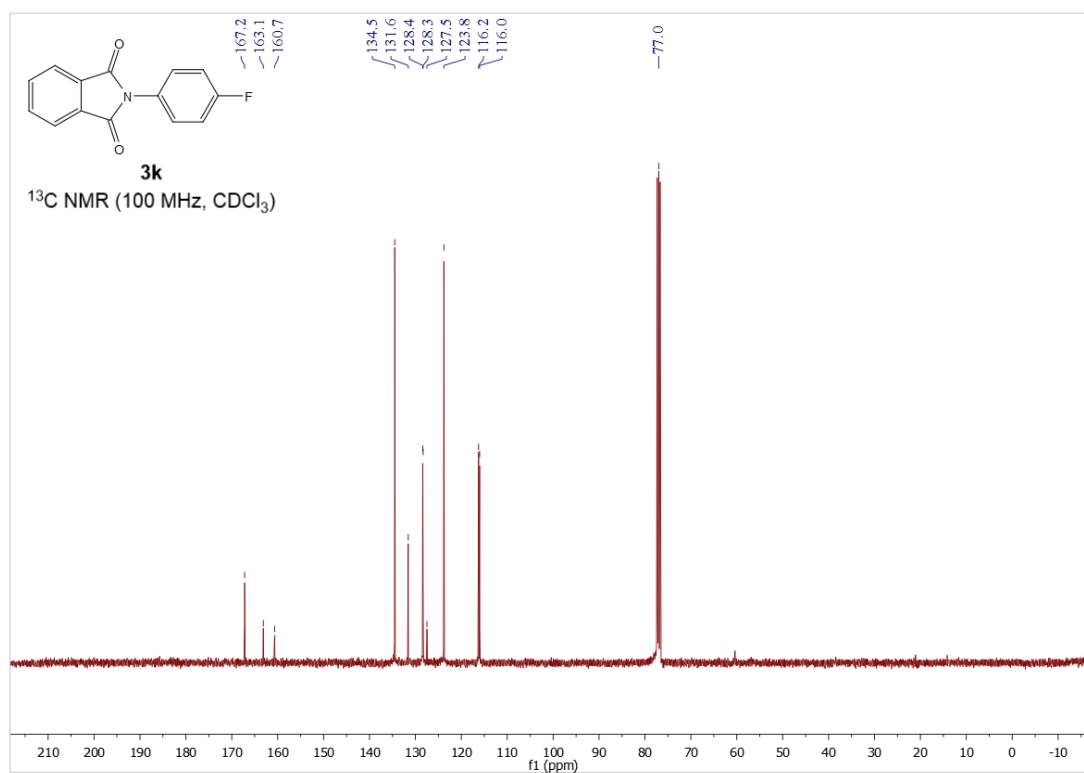
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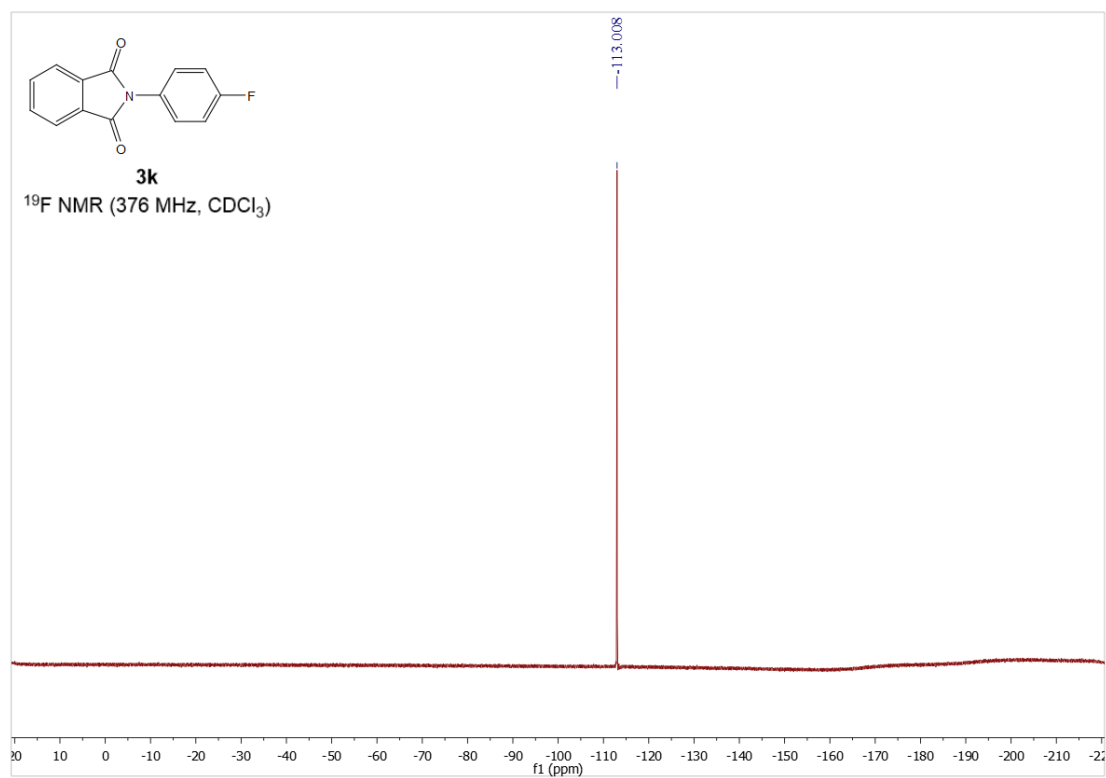
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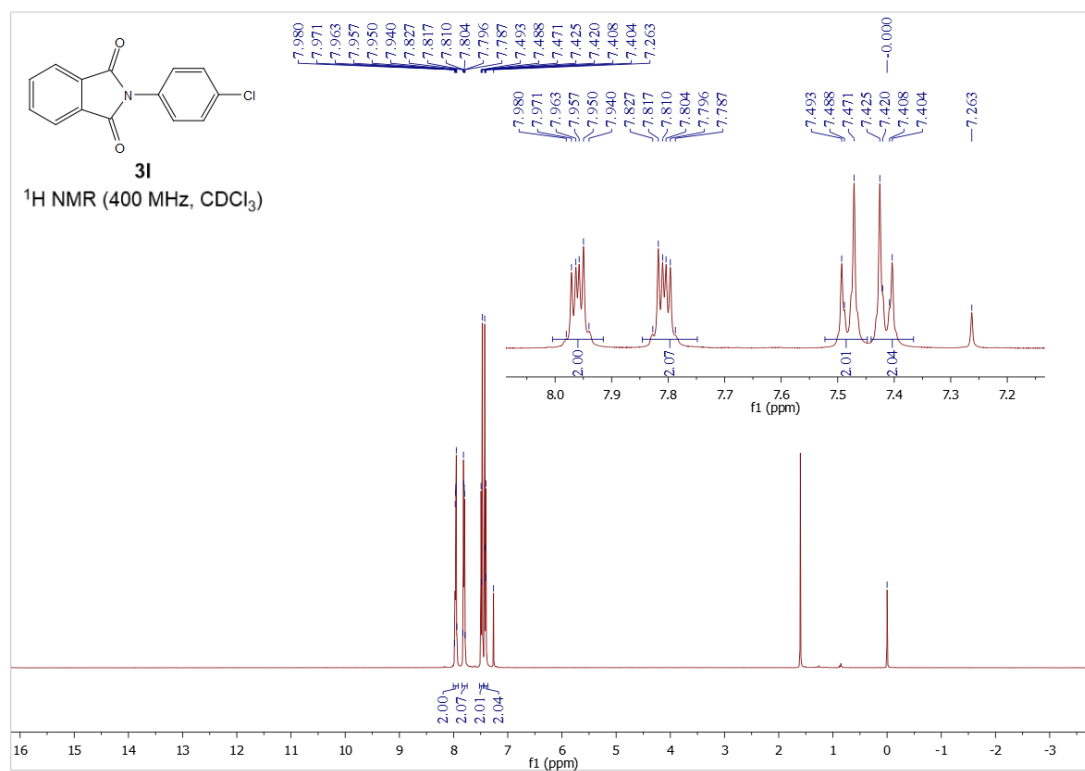
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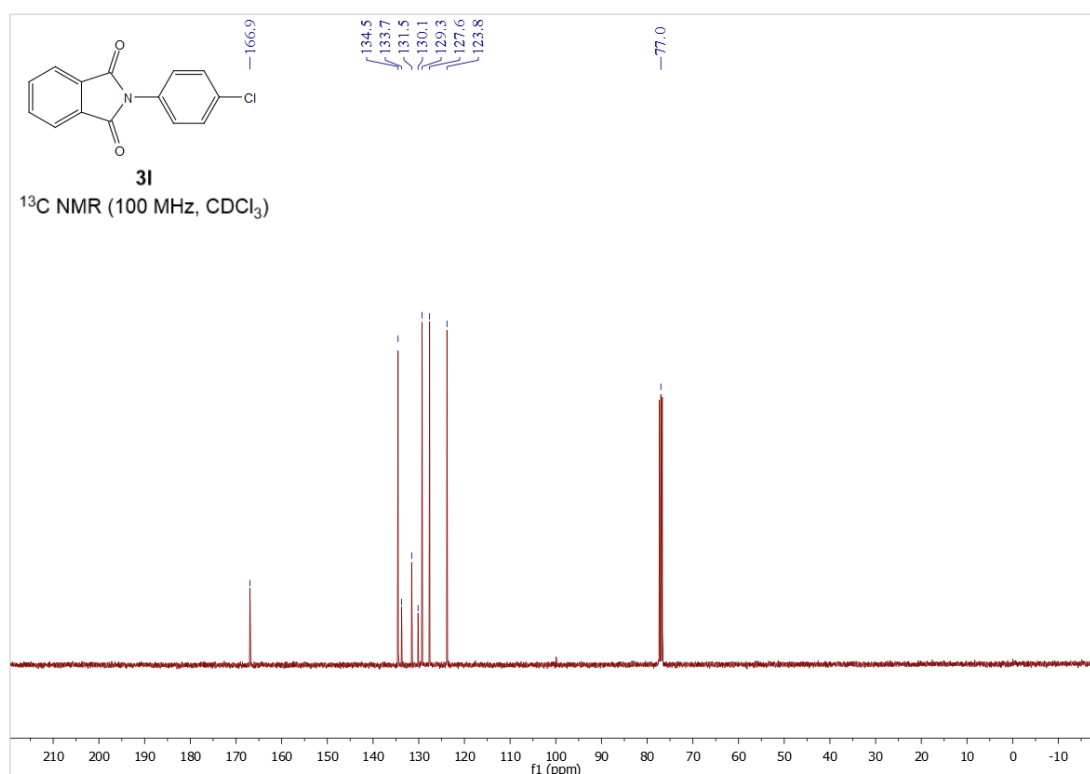
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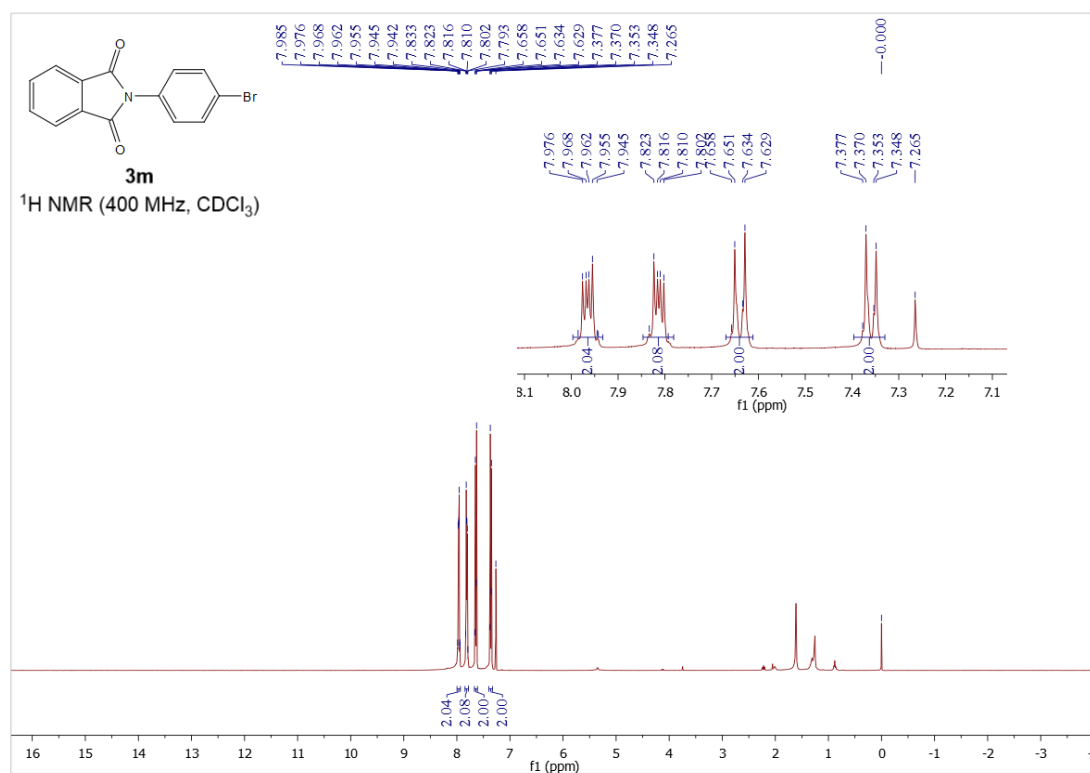
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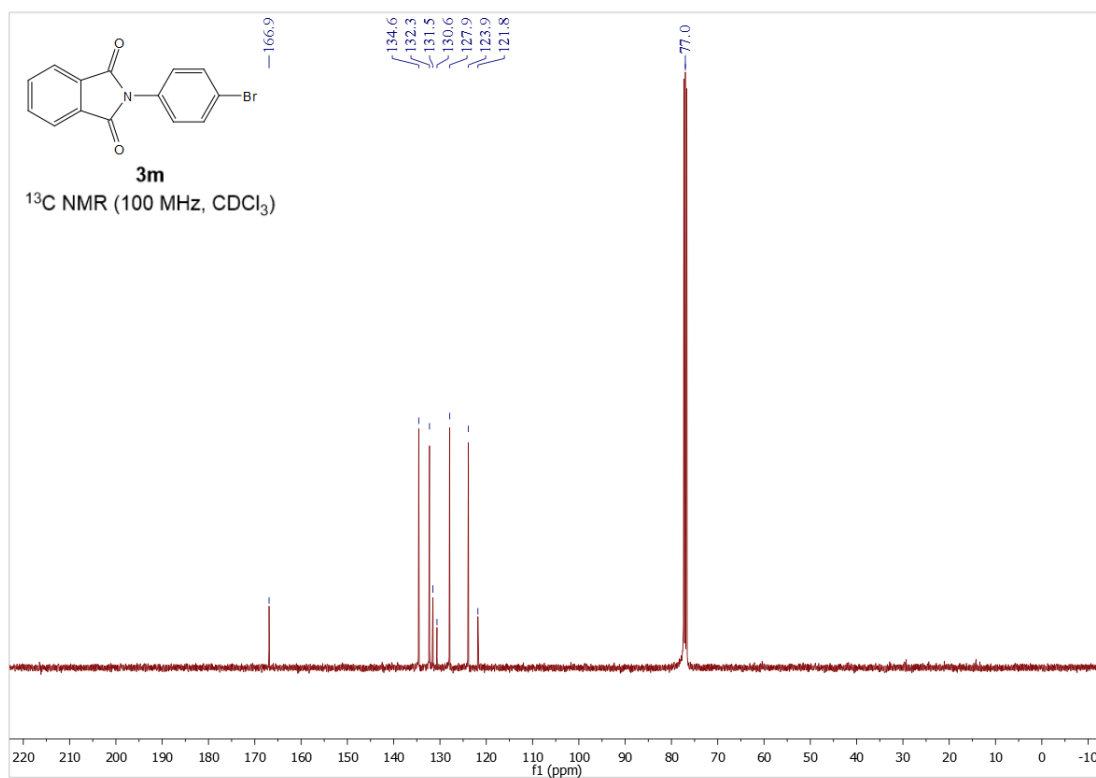
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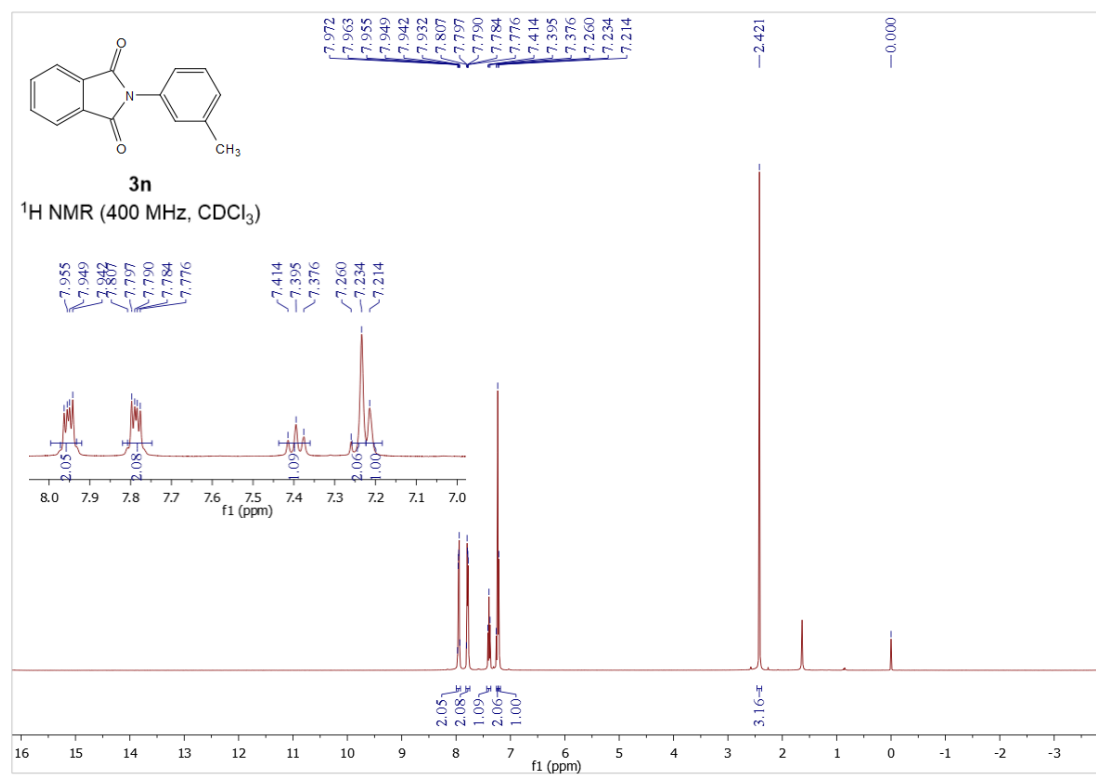
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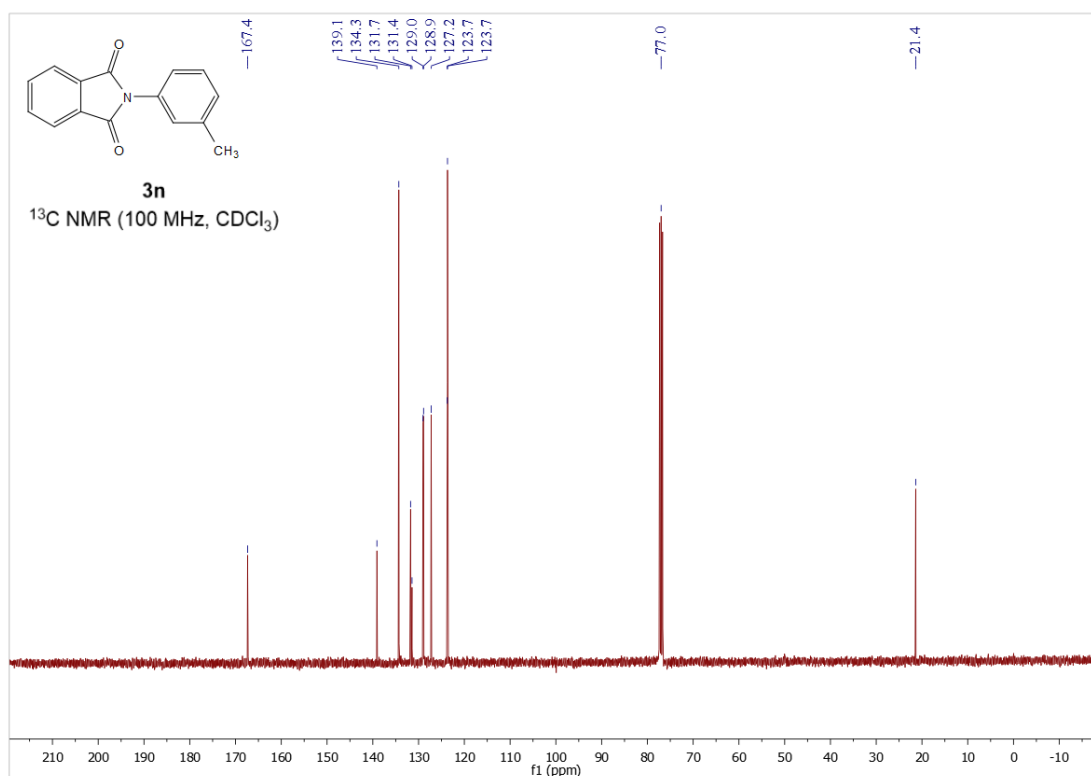
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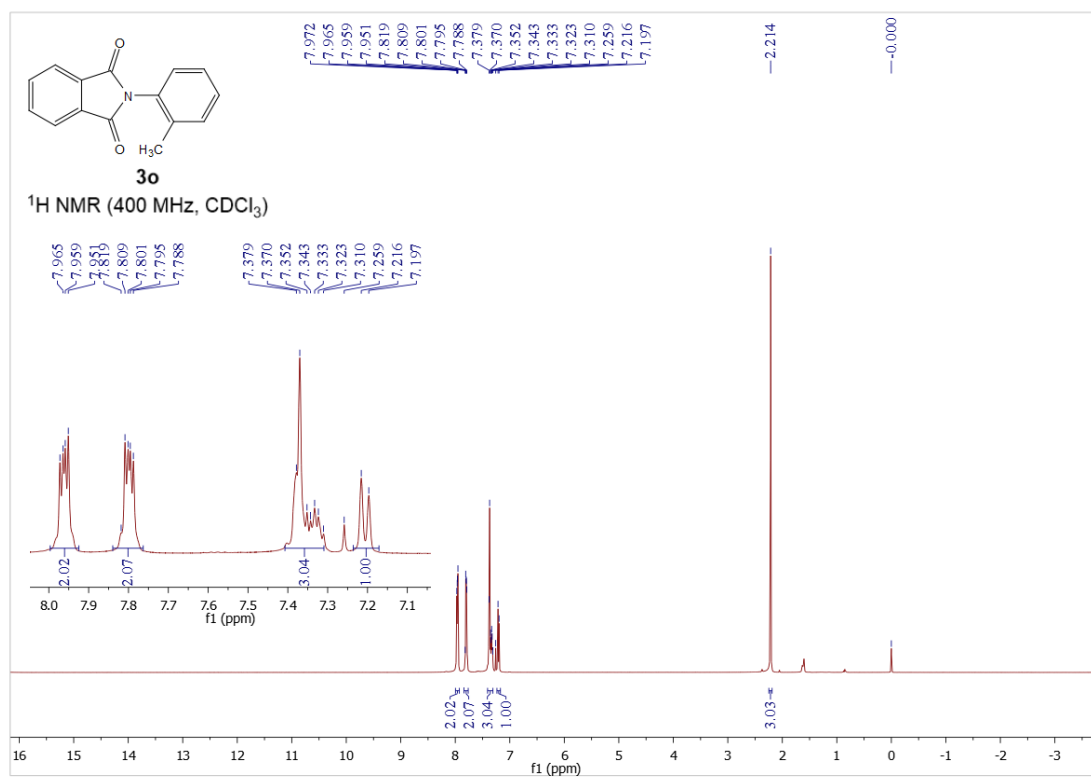
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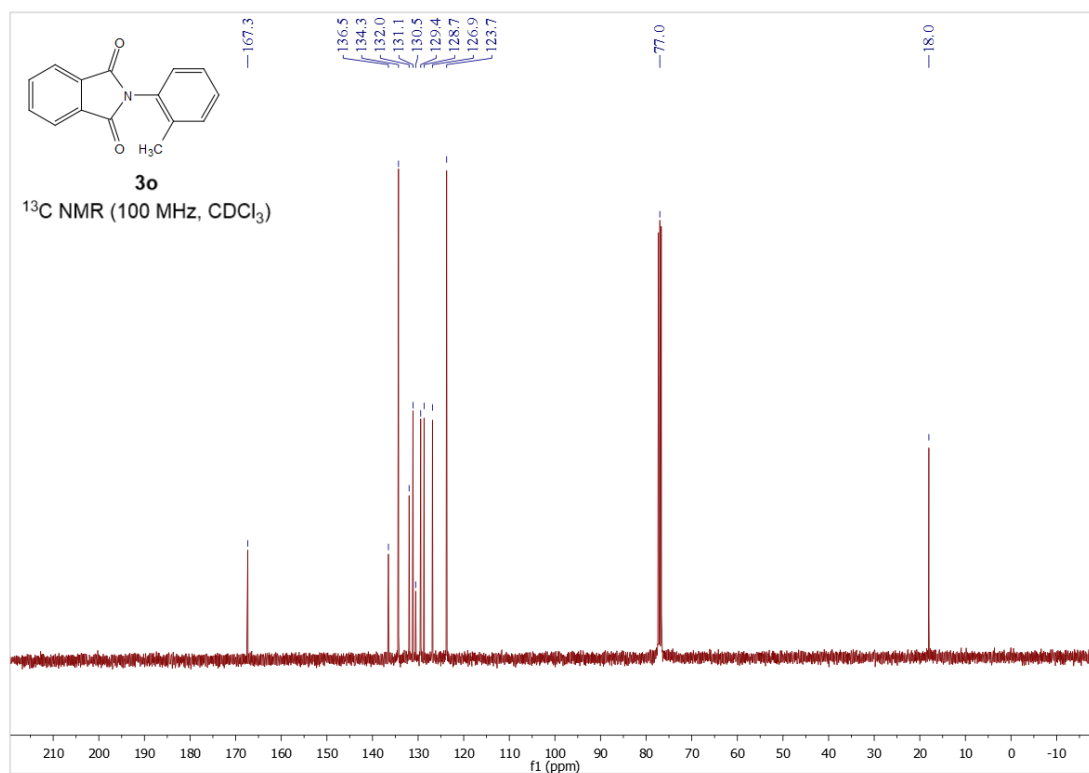
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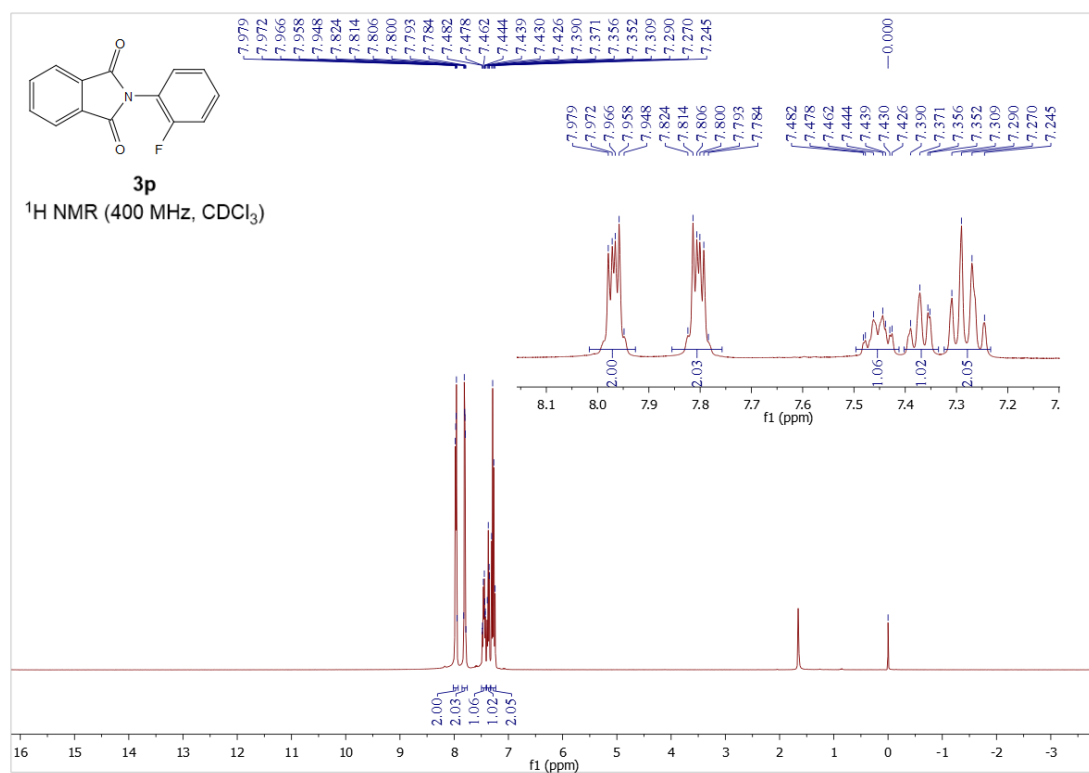
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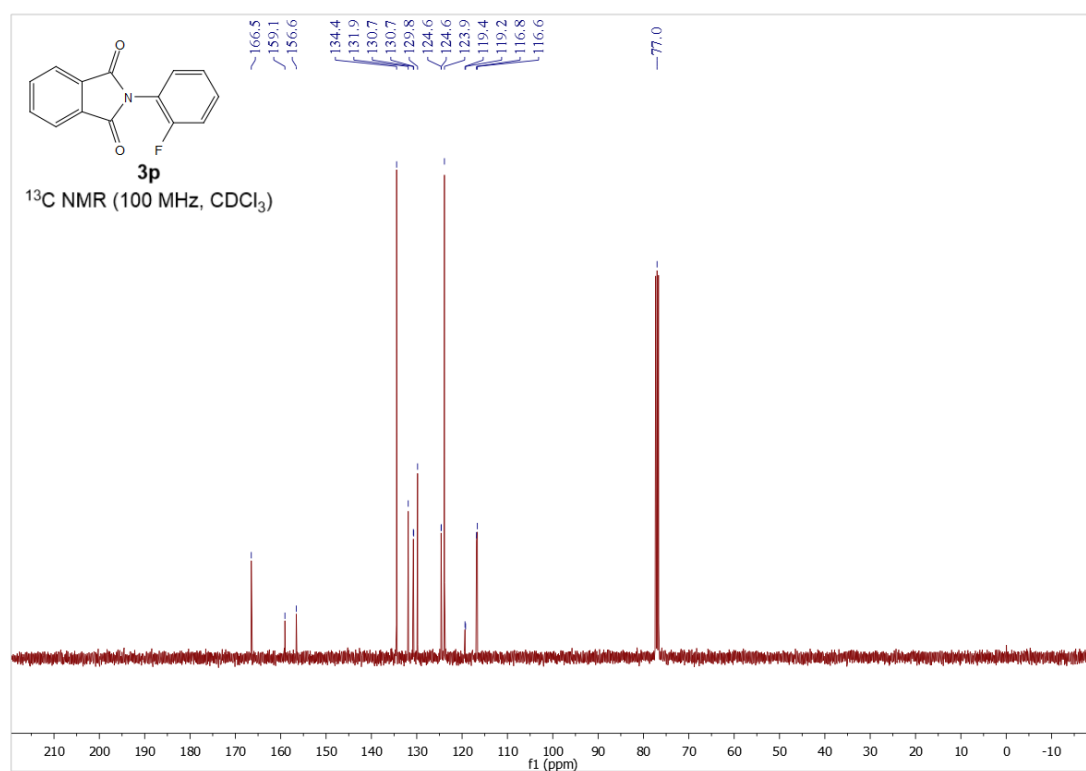
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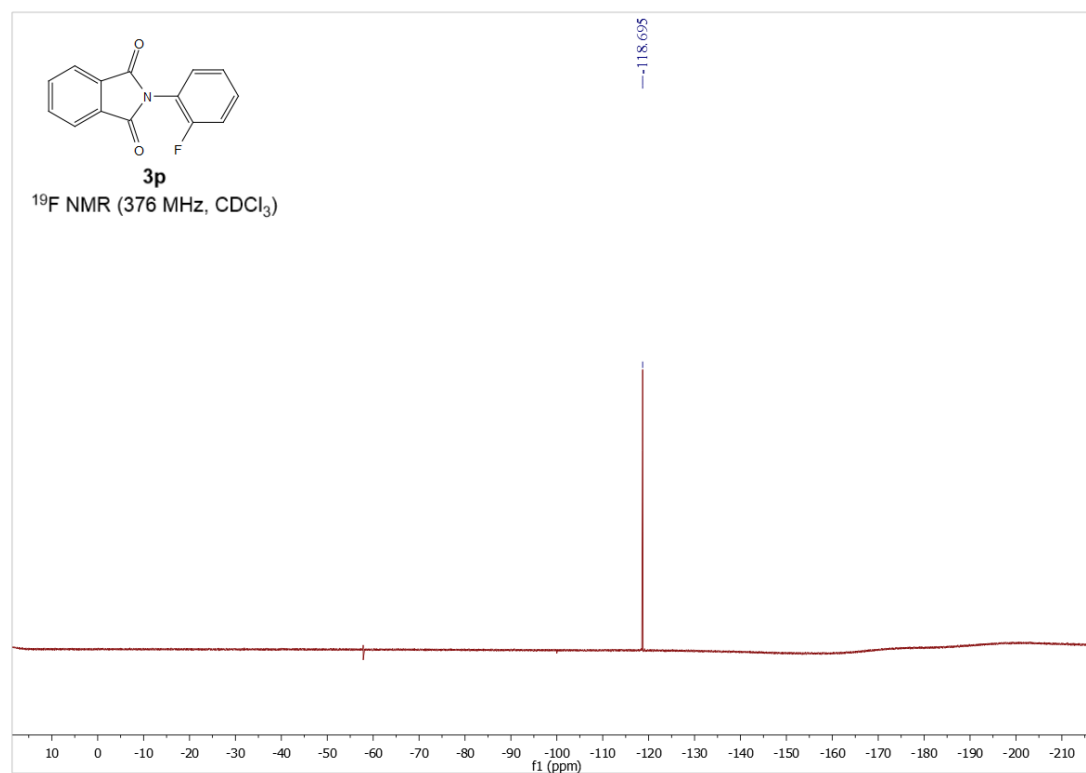
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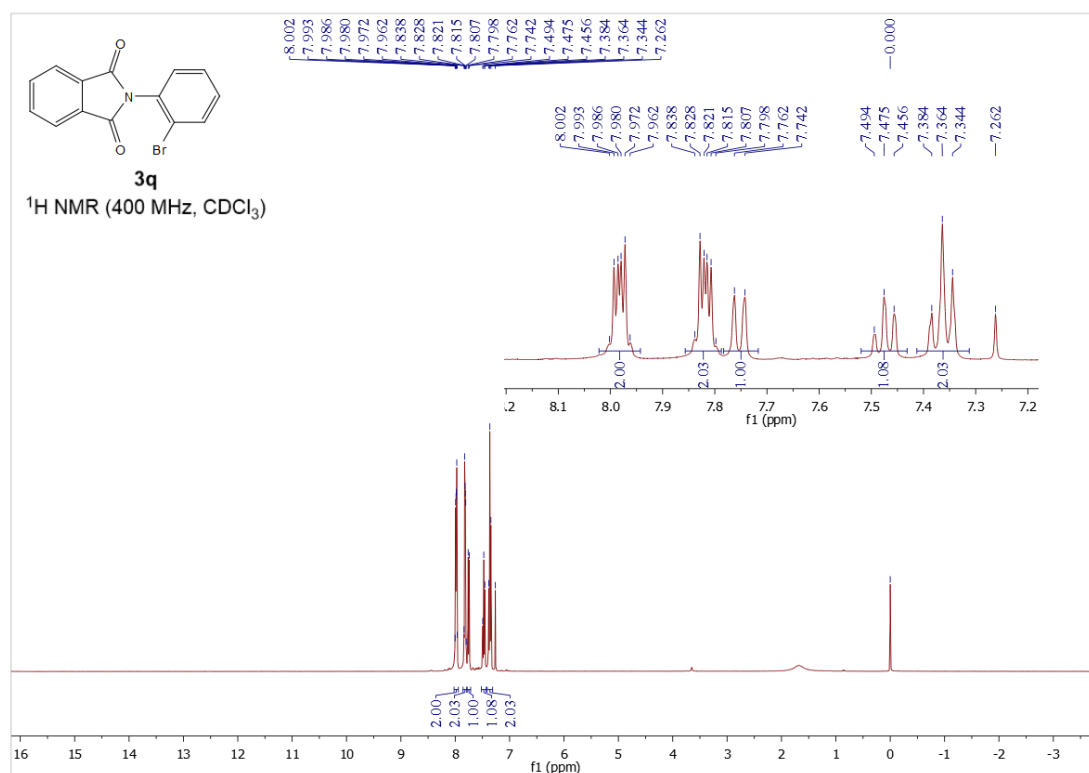
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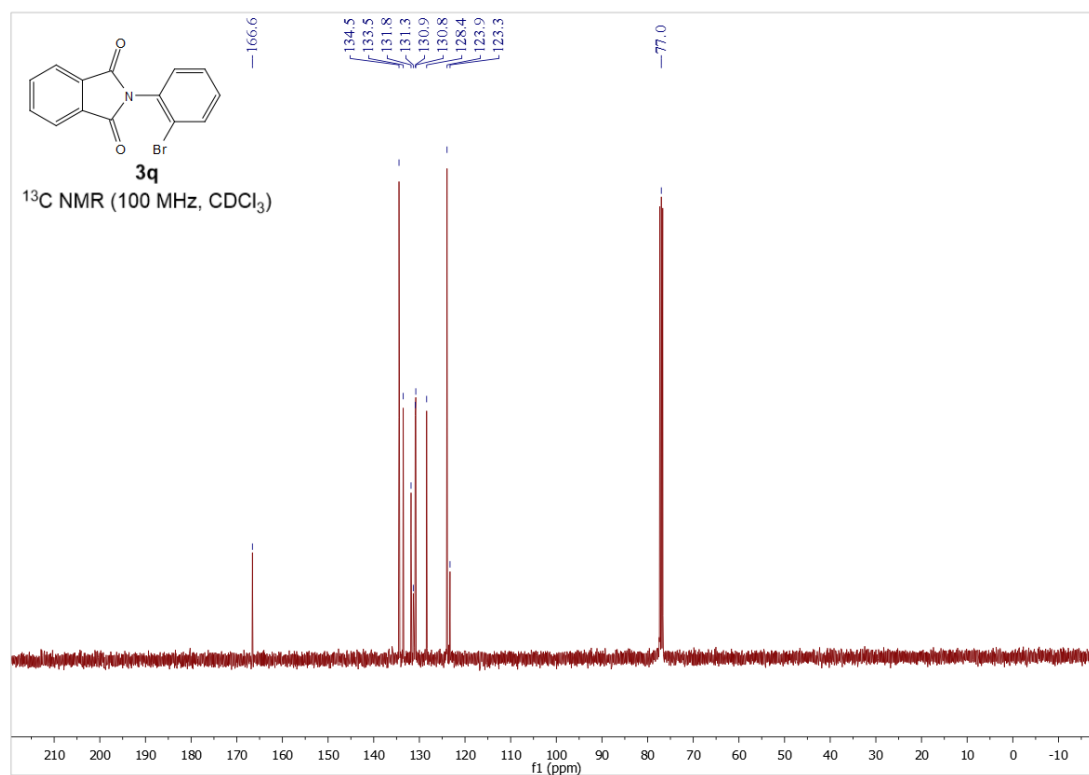
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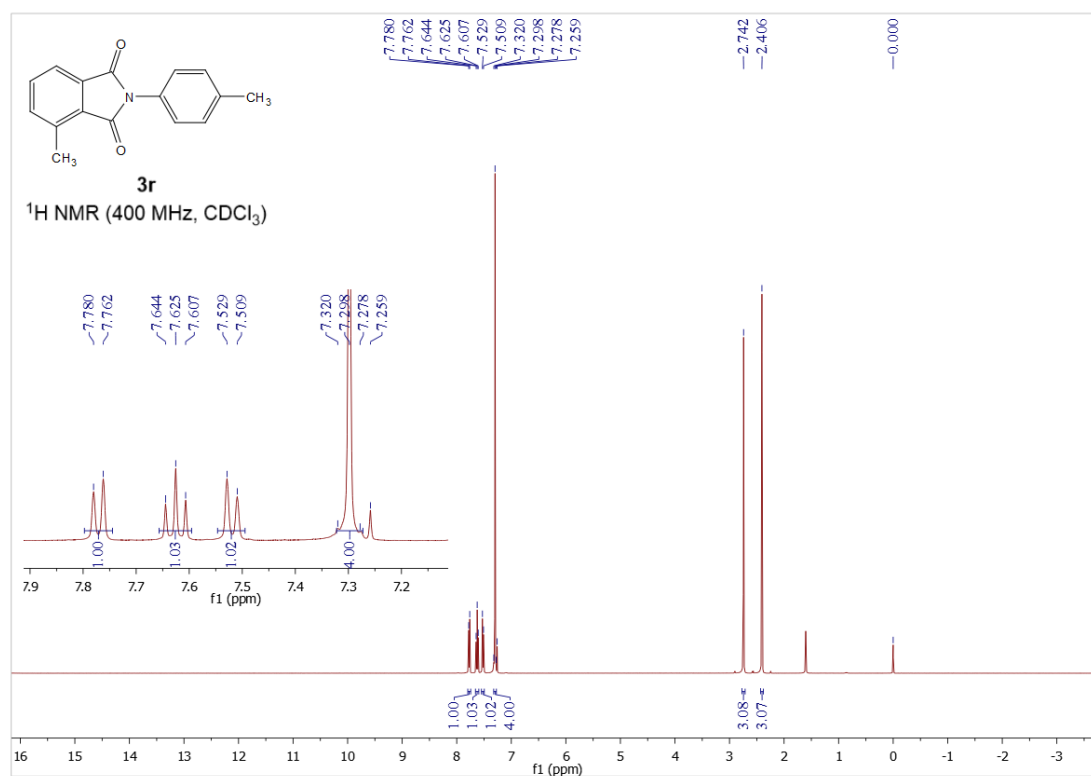
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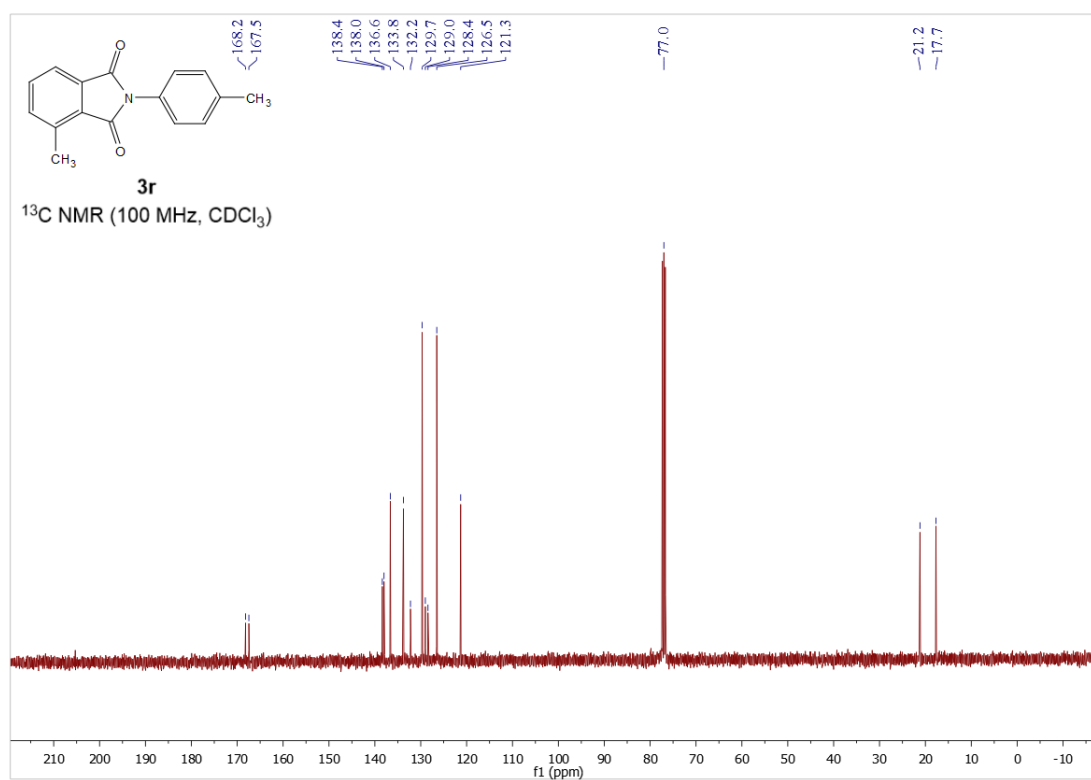
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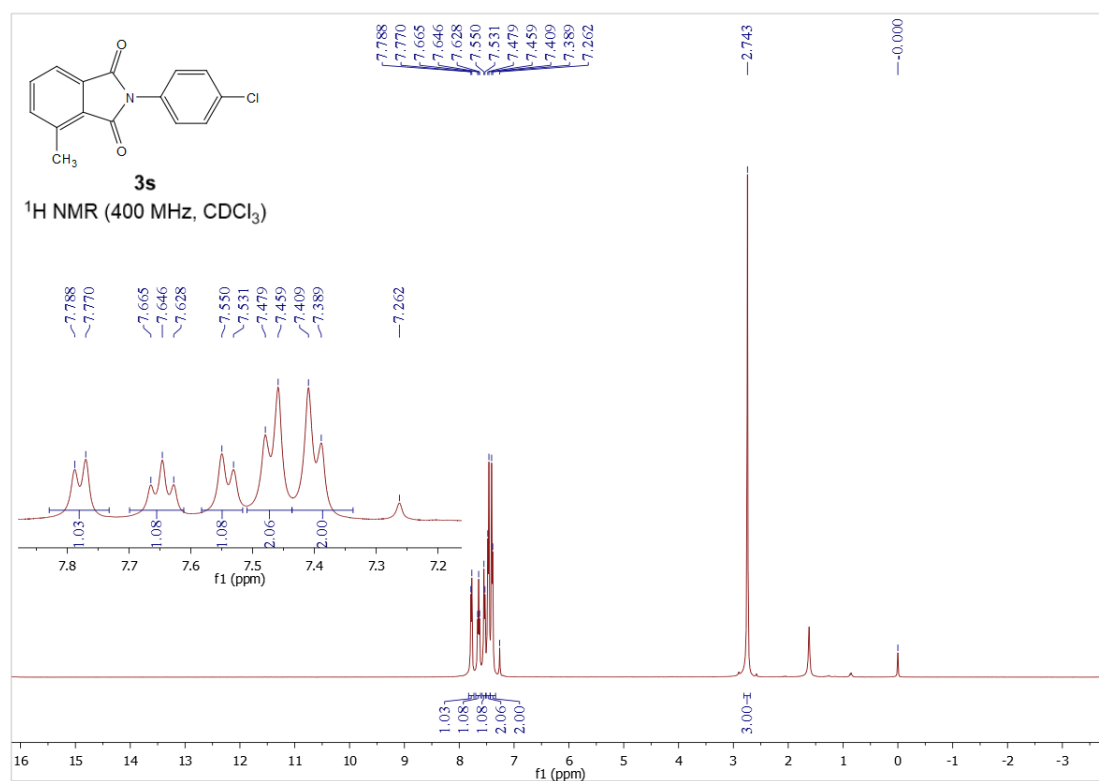
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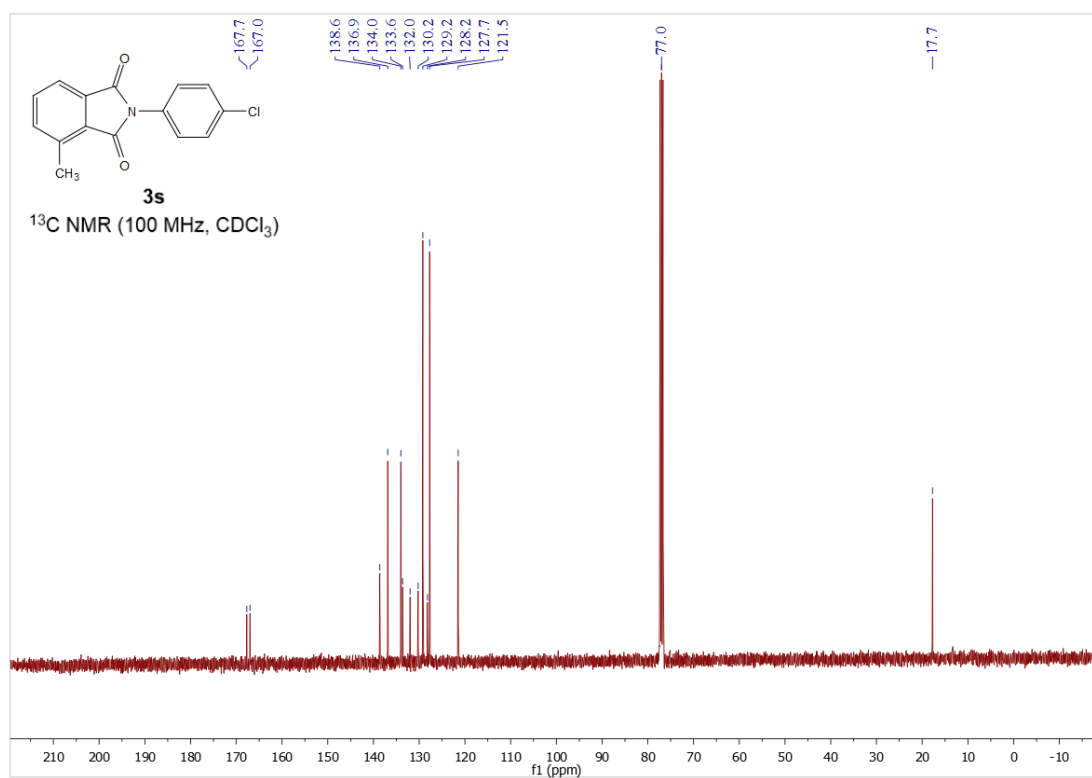
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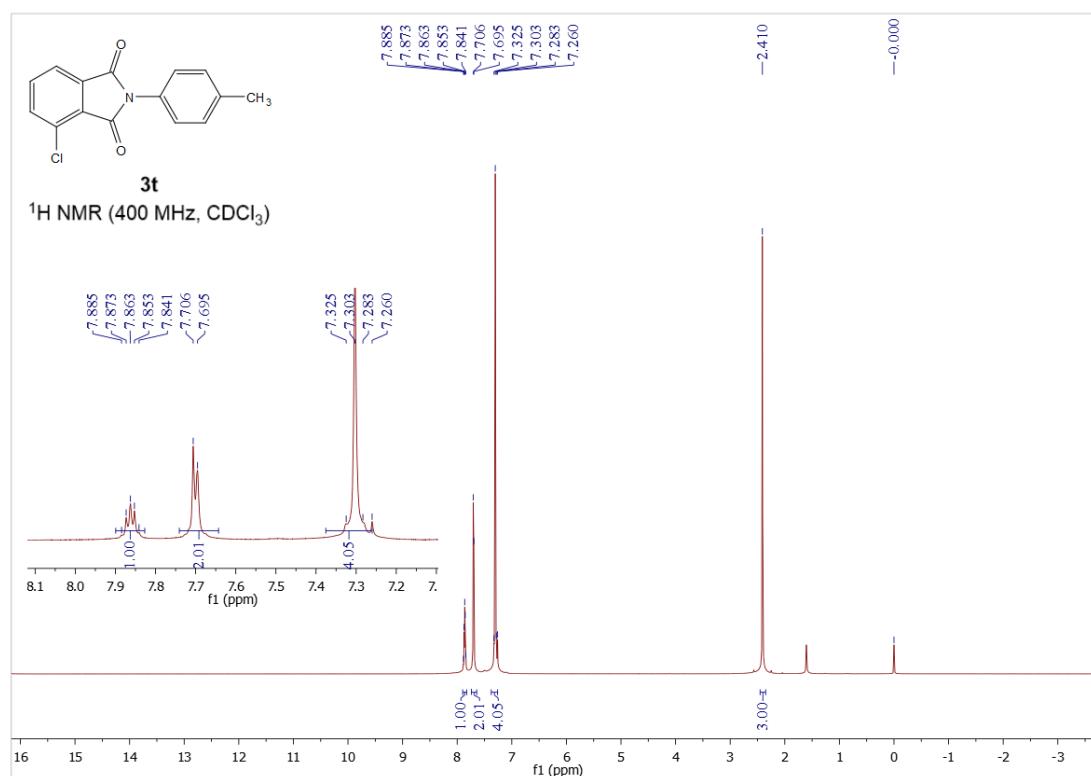
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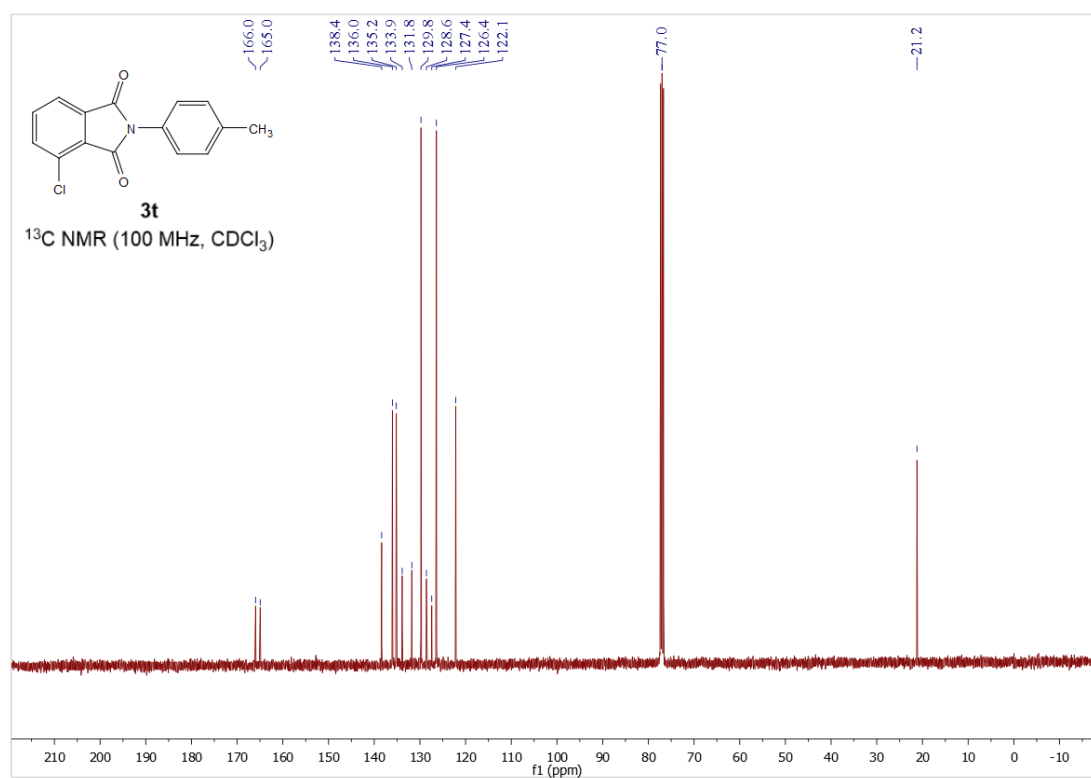
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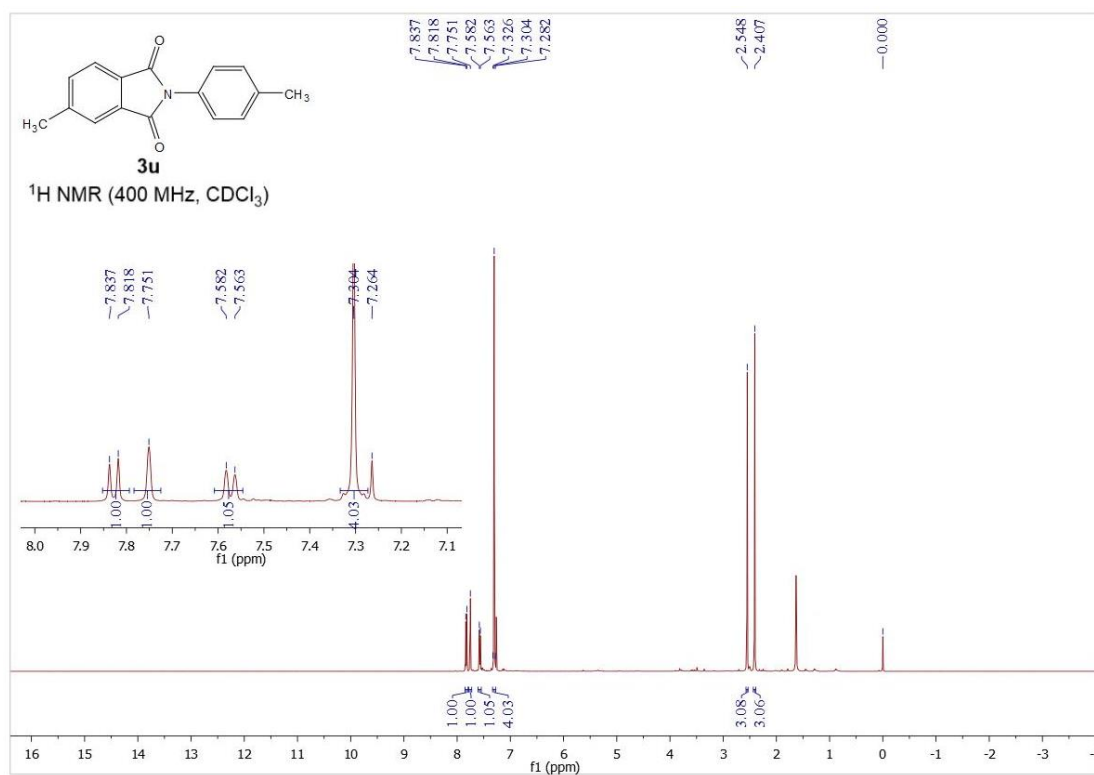
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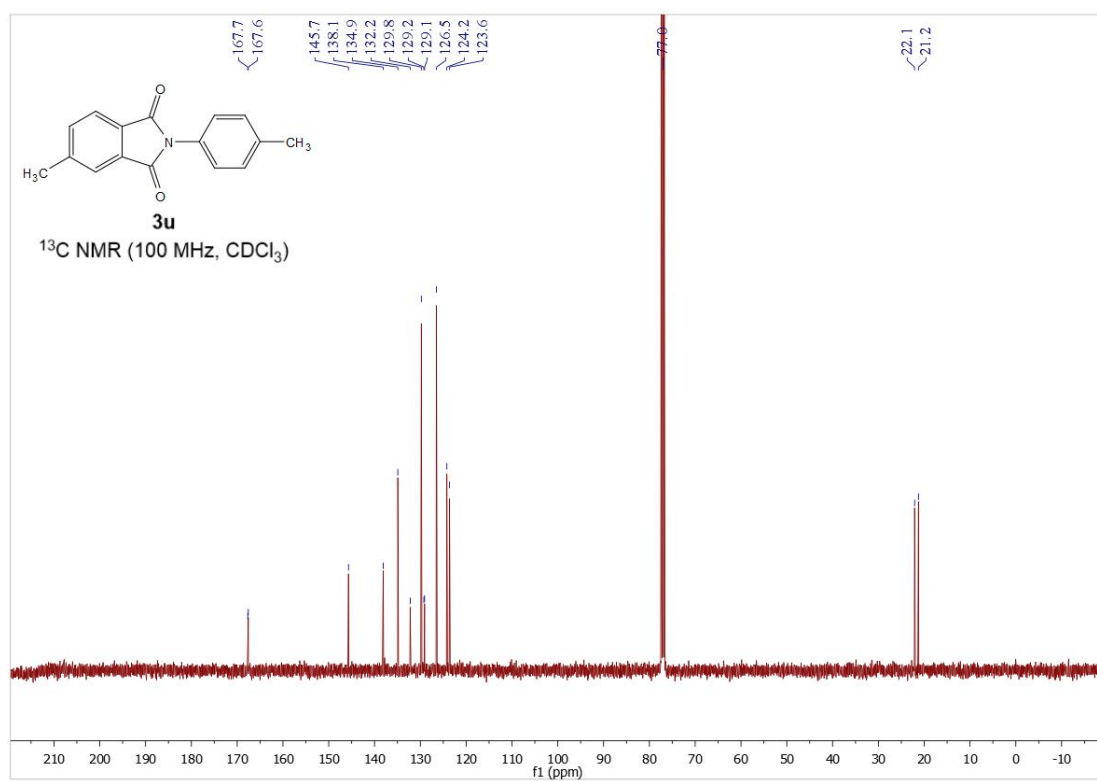
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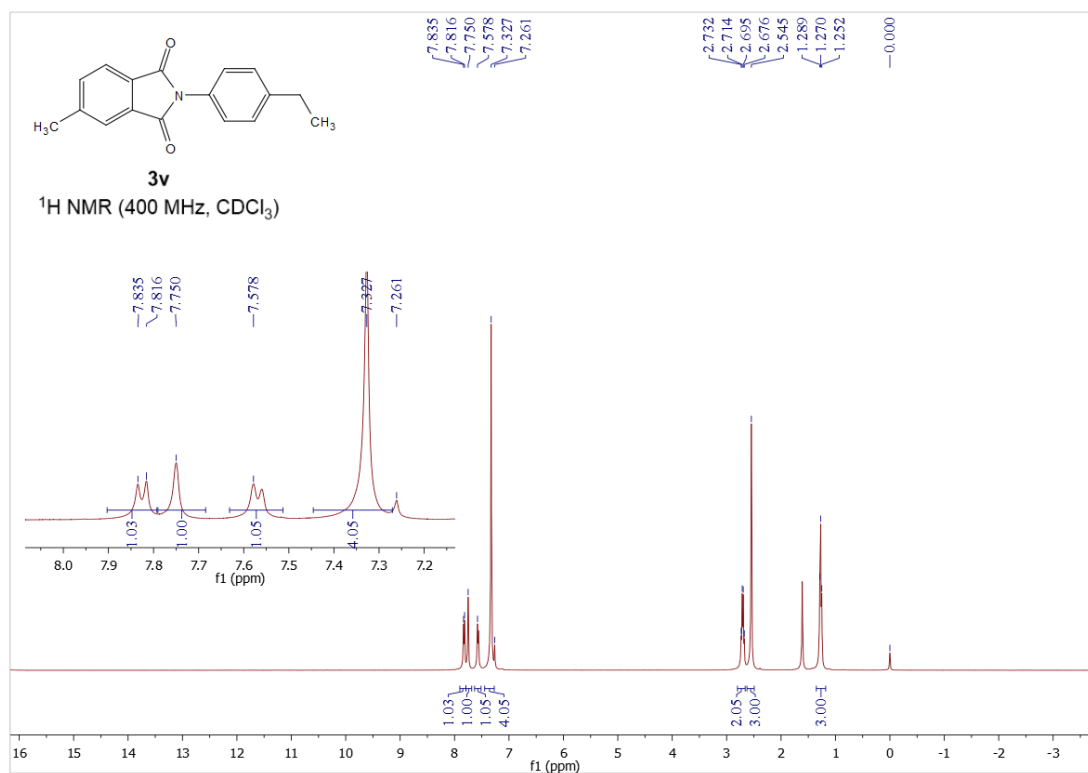
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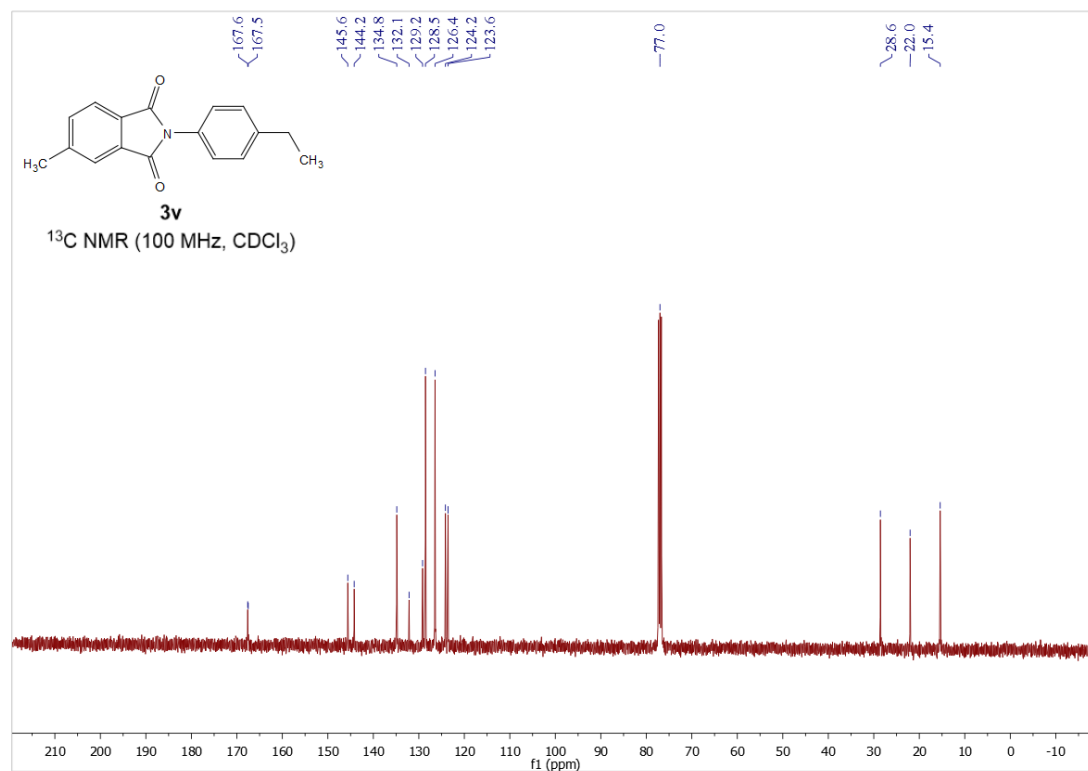
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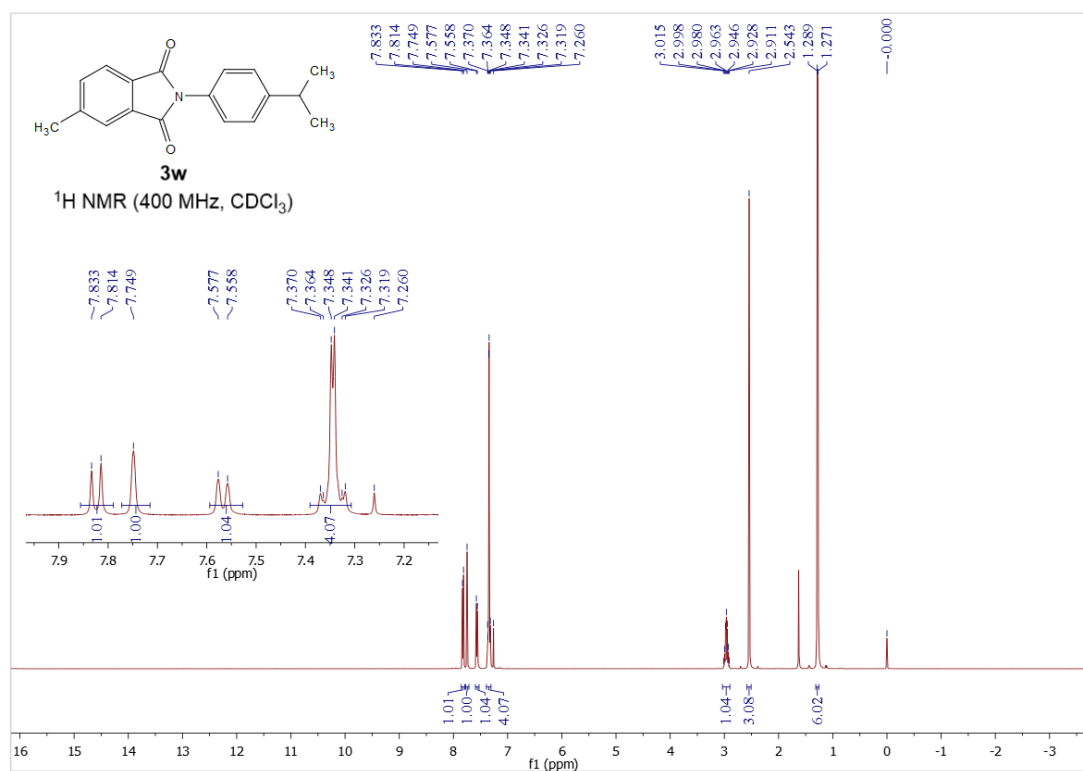
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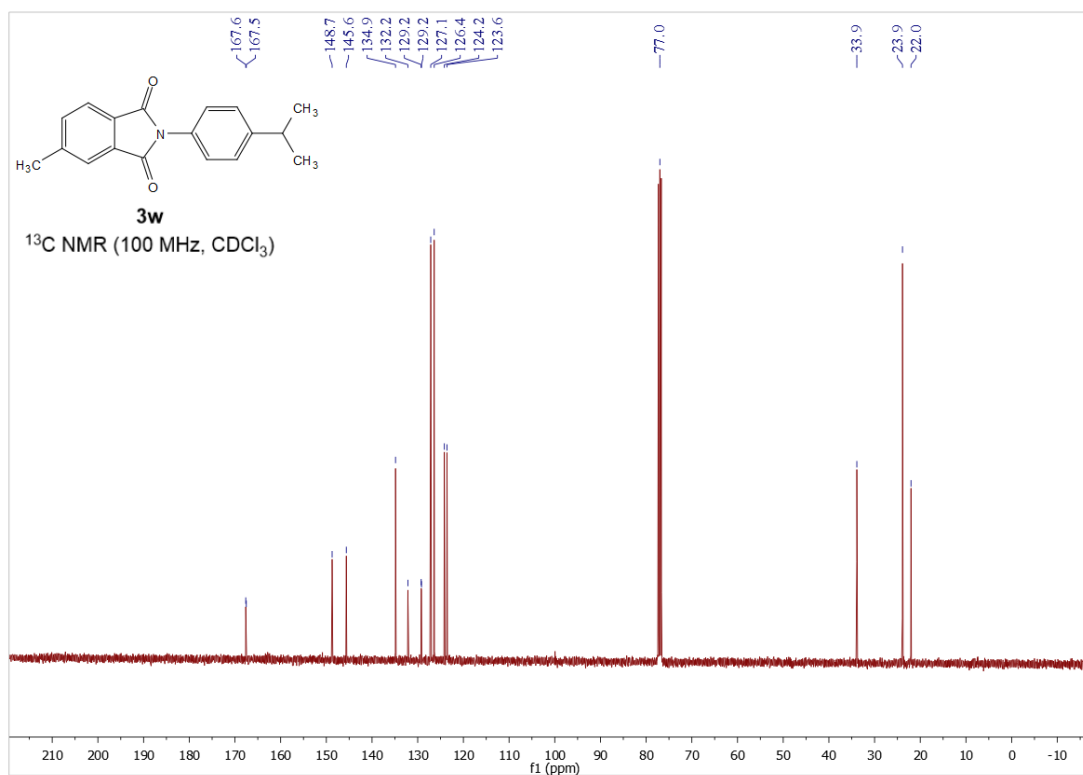
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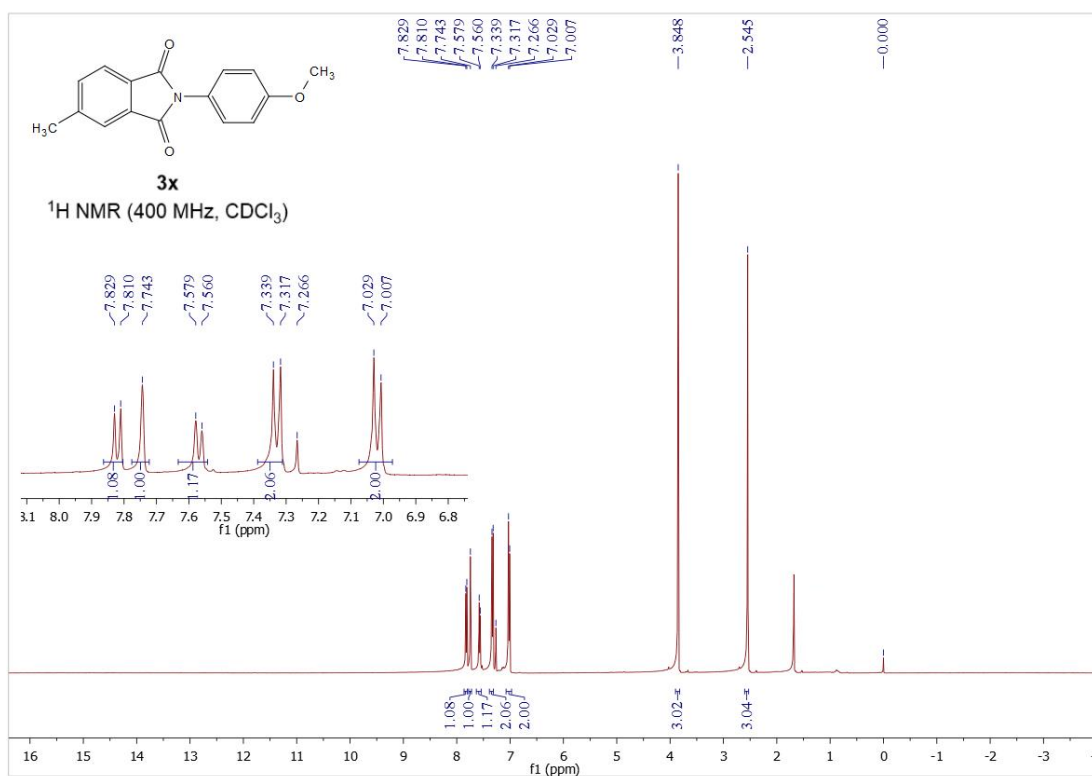
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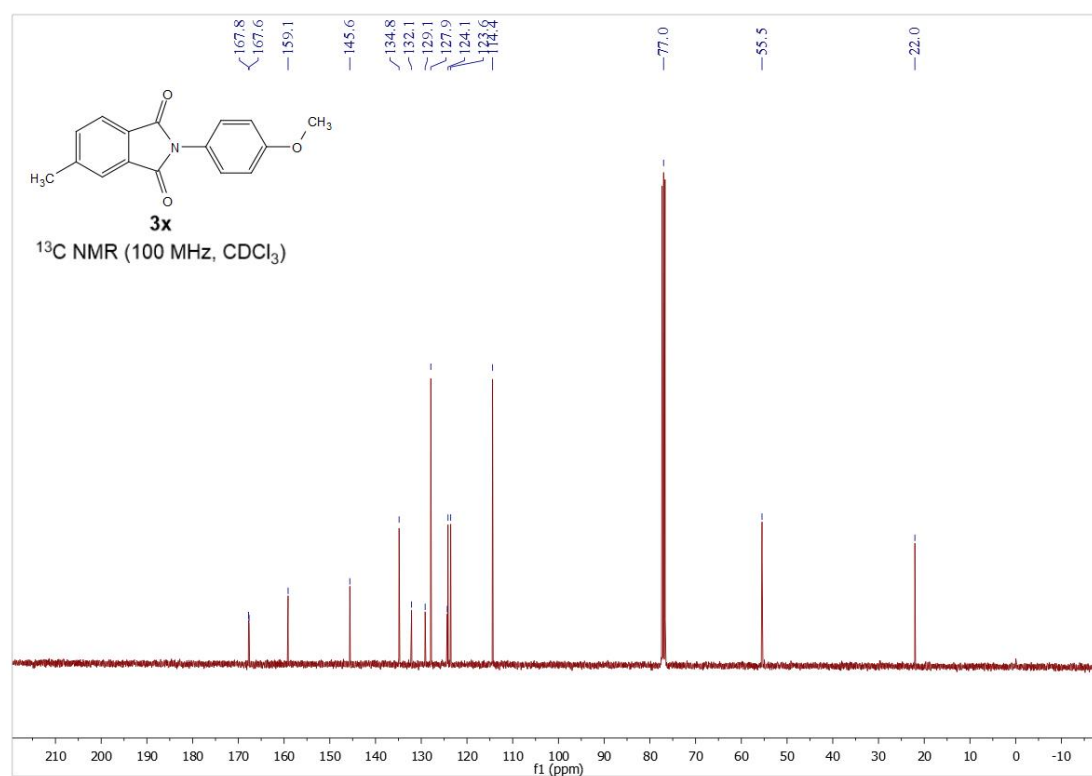
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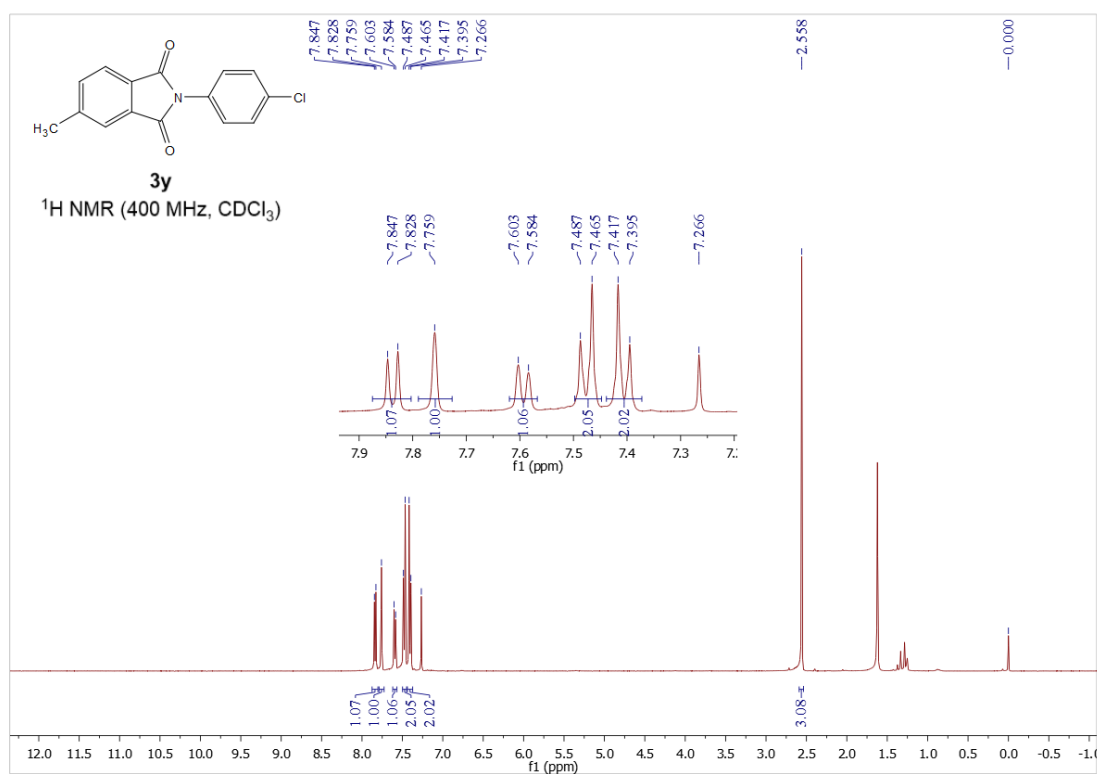
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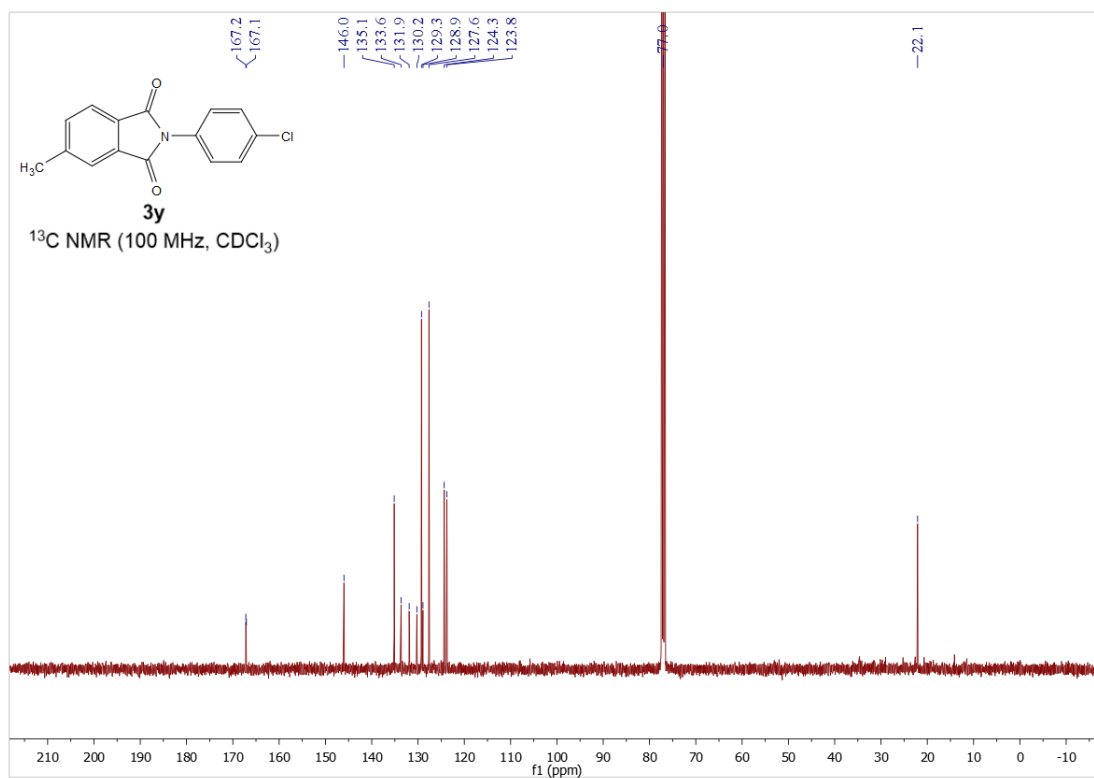
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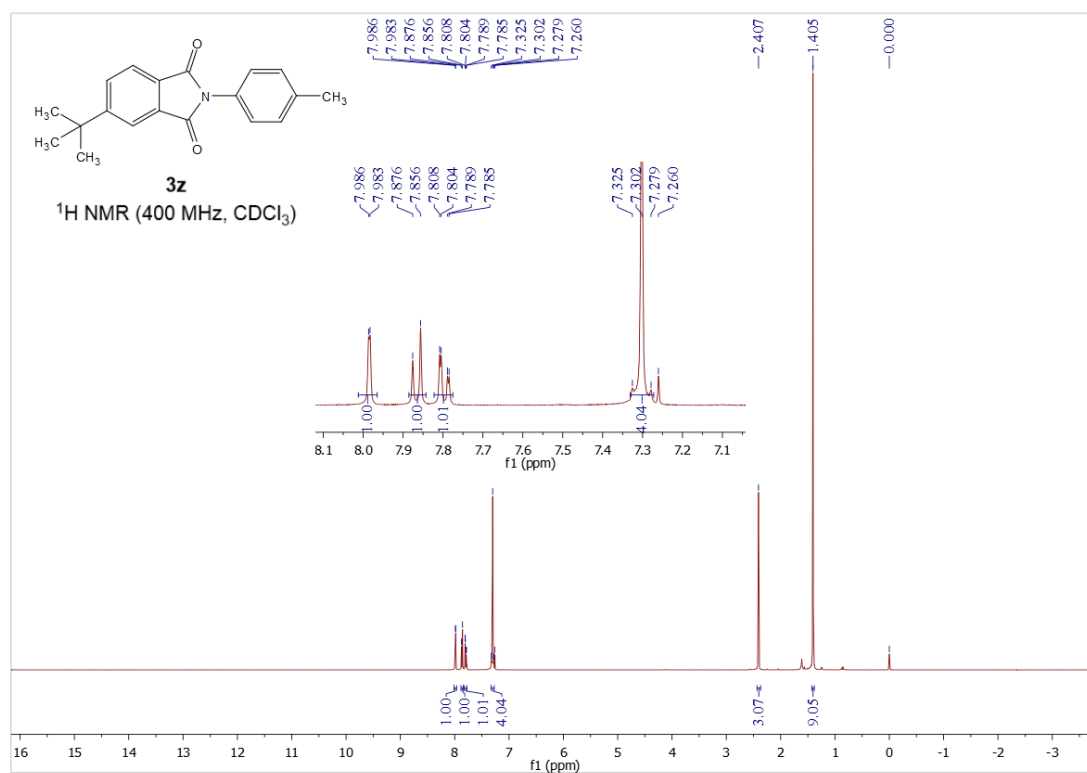
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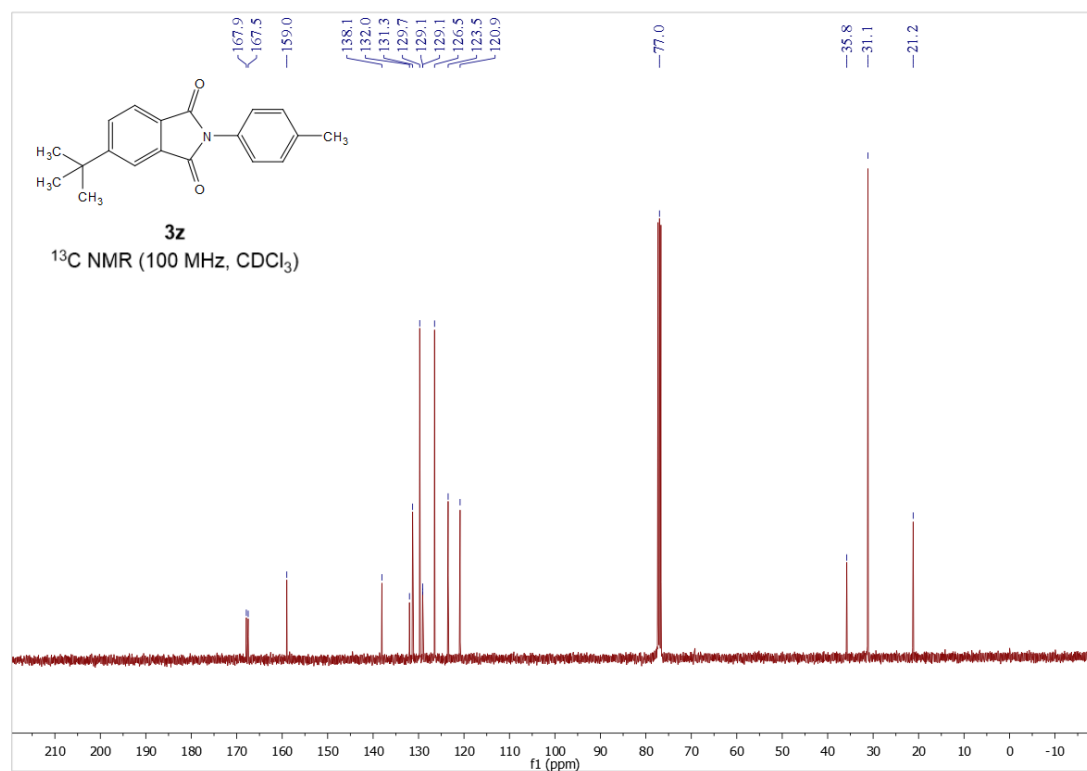
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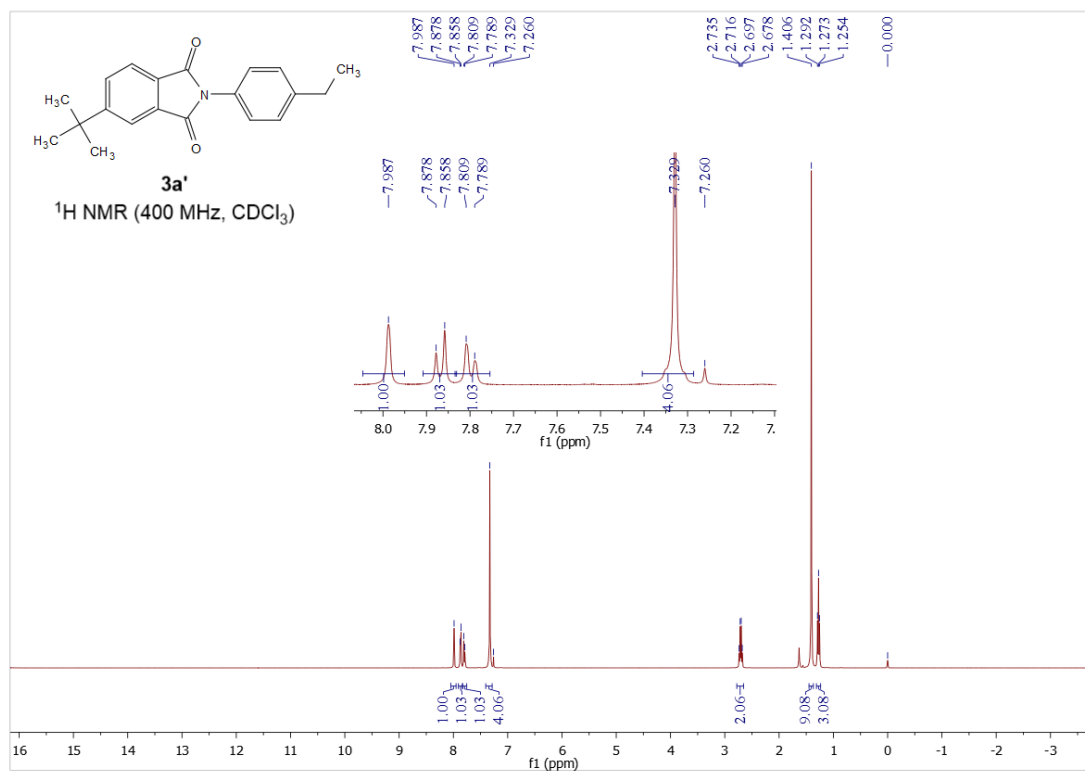
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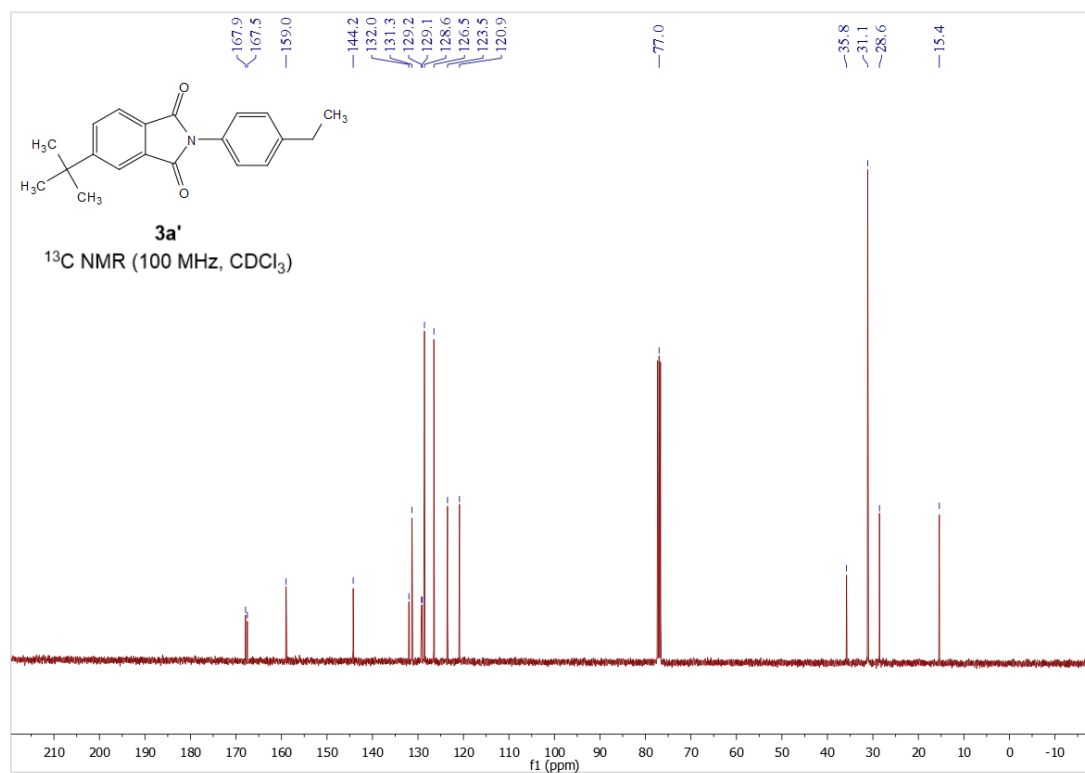
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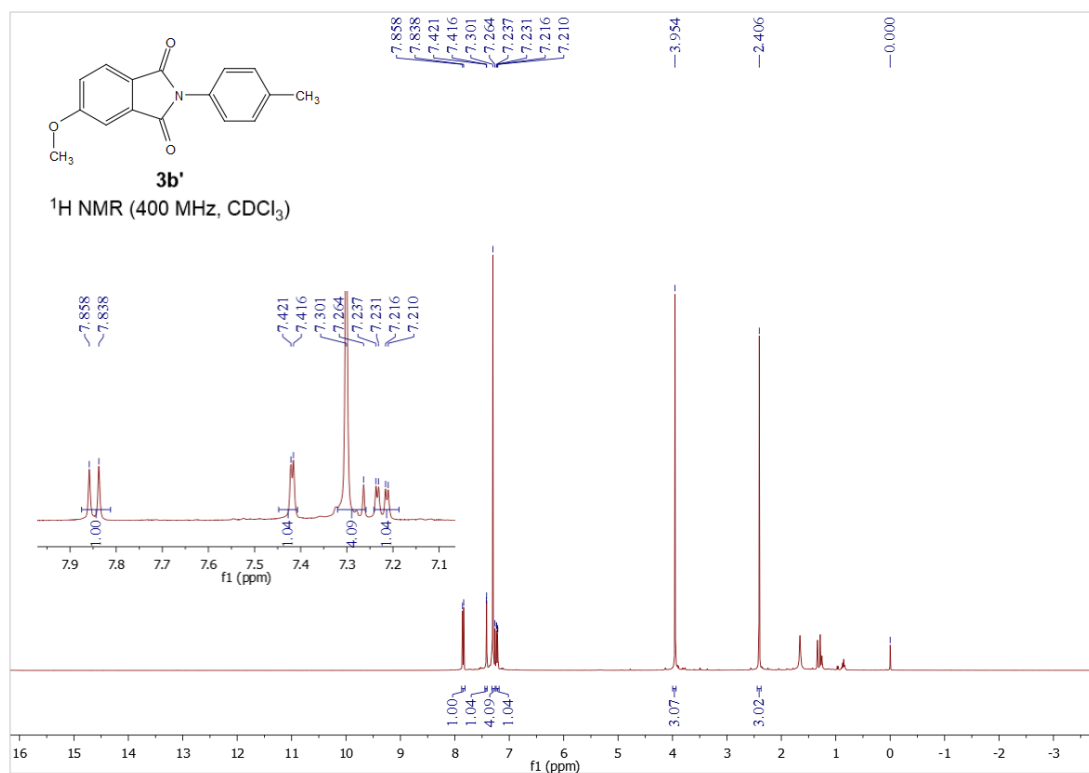
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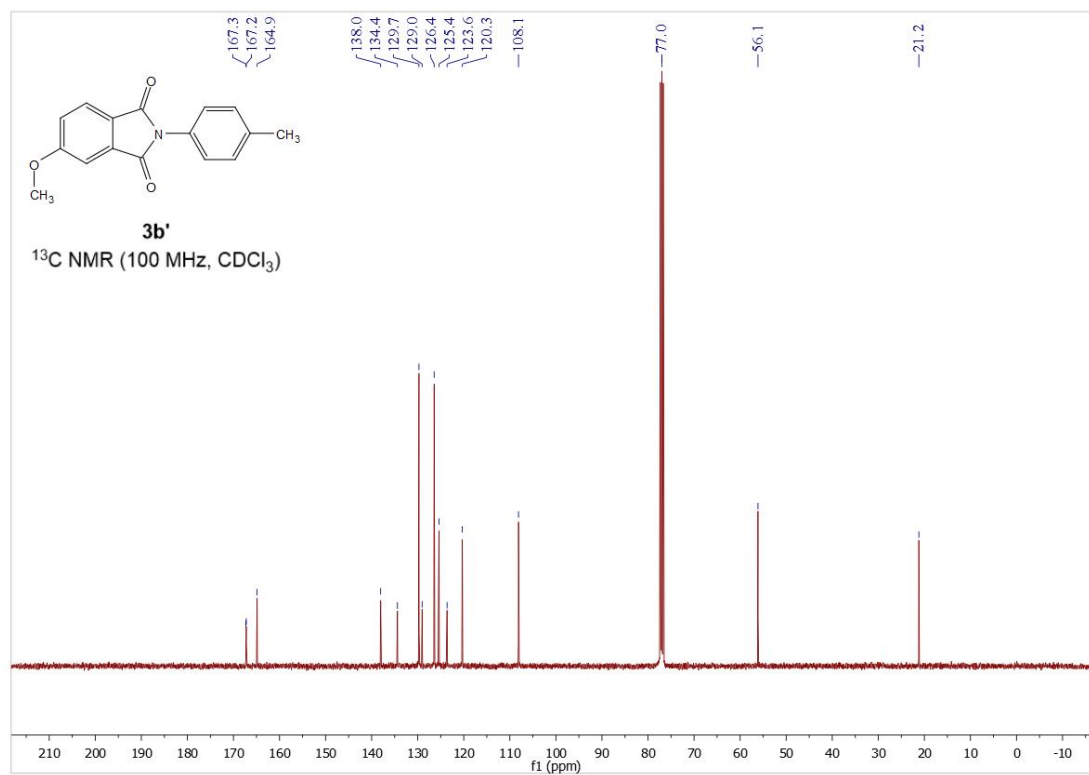
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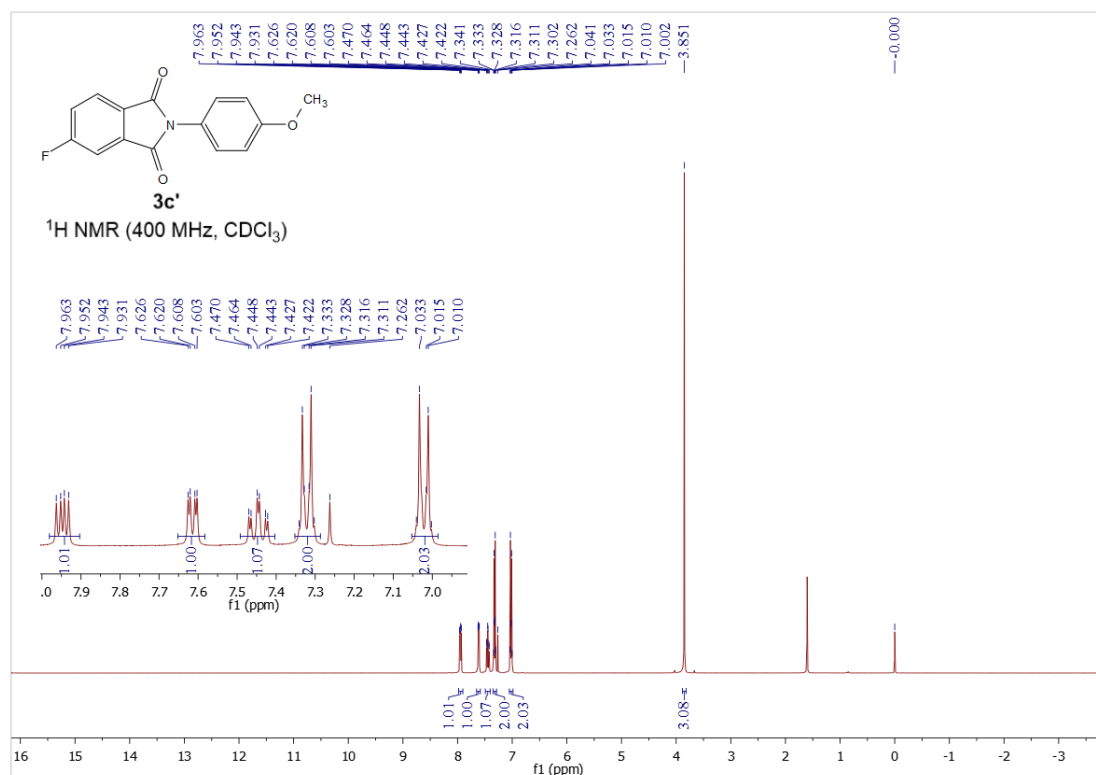
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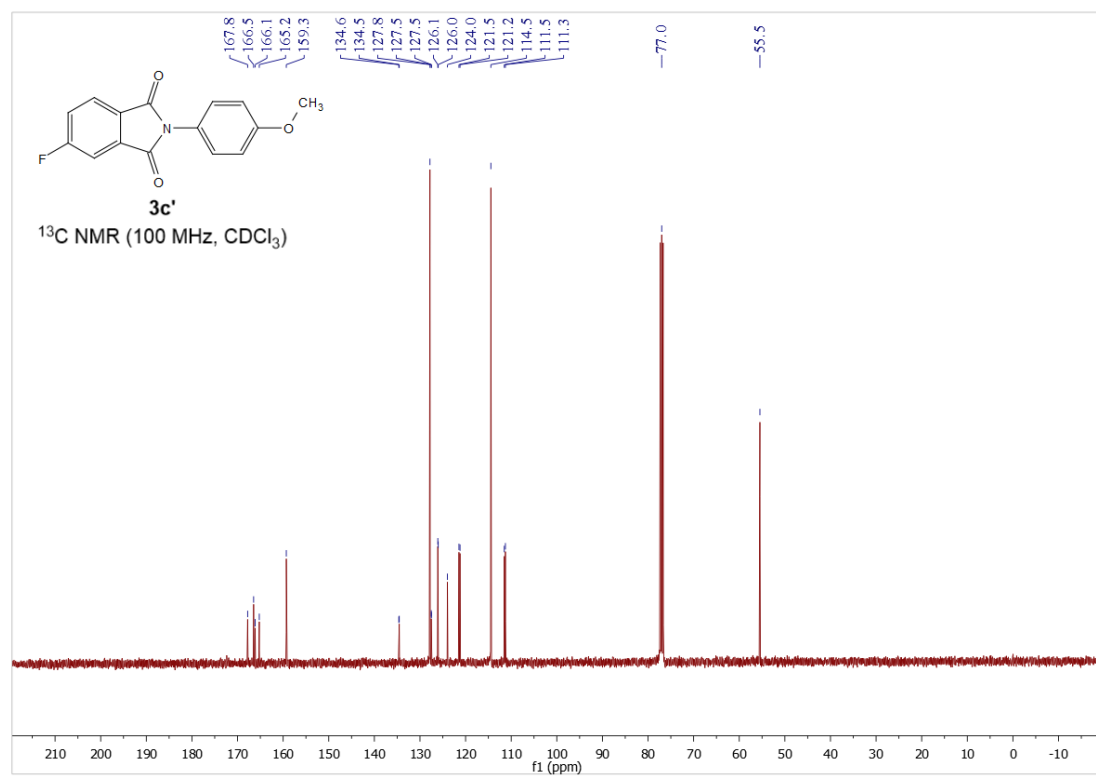
¹³C NMR of Compound **3b'** (100 MHz, CDCl₃)



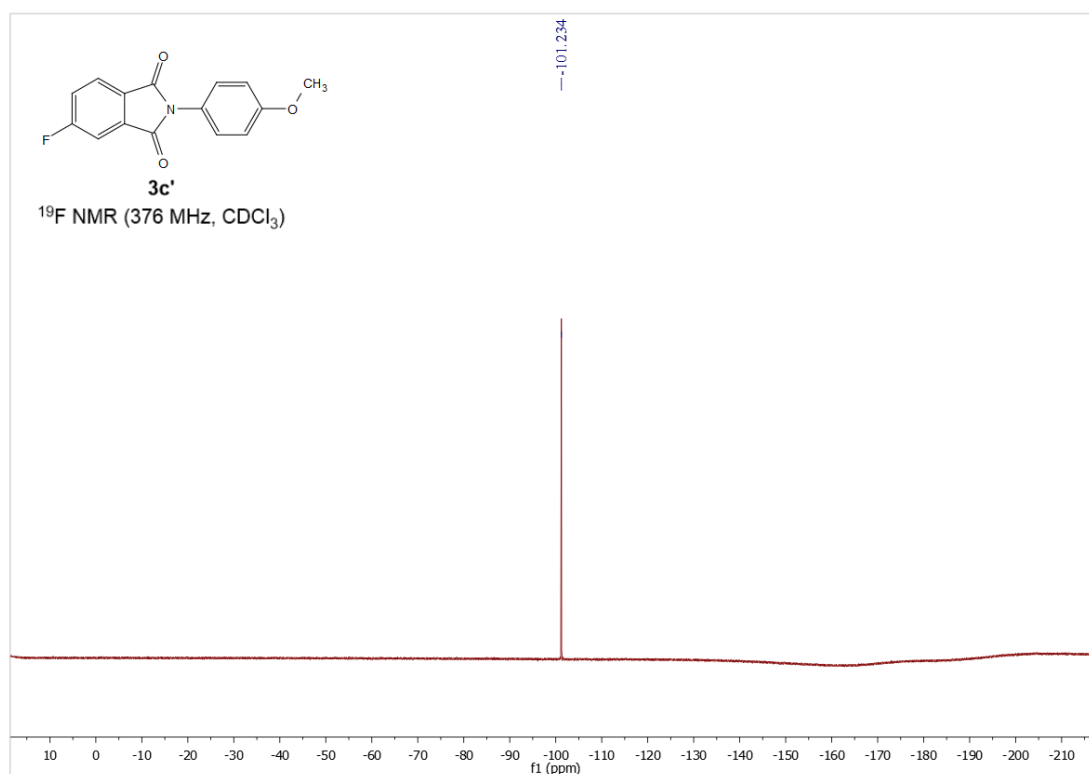
¹H NMR of Compound **3c'** (400 MHz, CDCl₃)



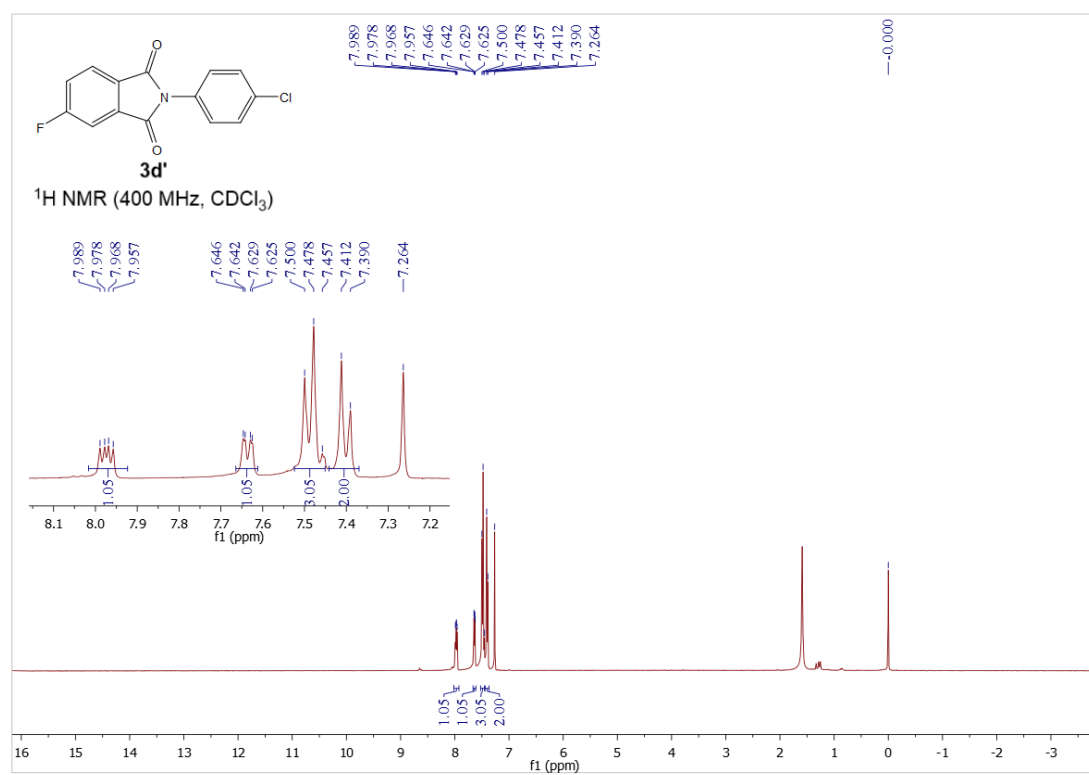
¹³C NMR of Compound **3c'** (100 MHz, CDCl₃)



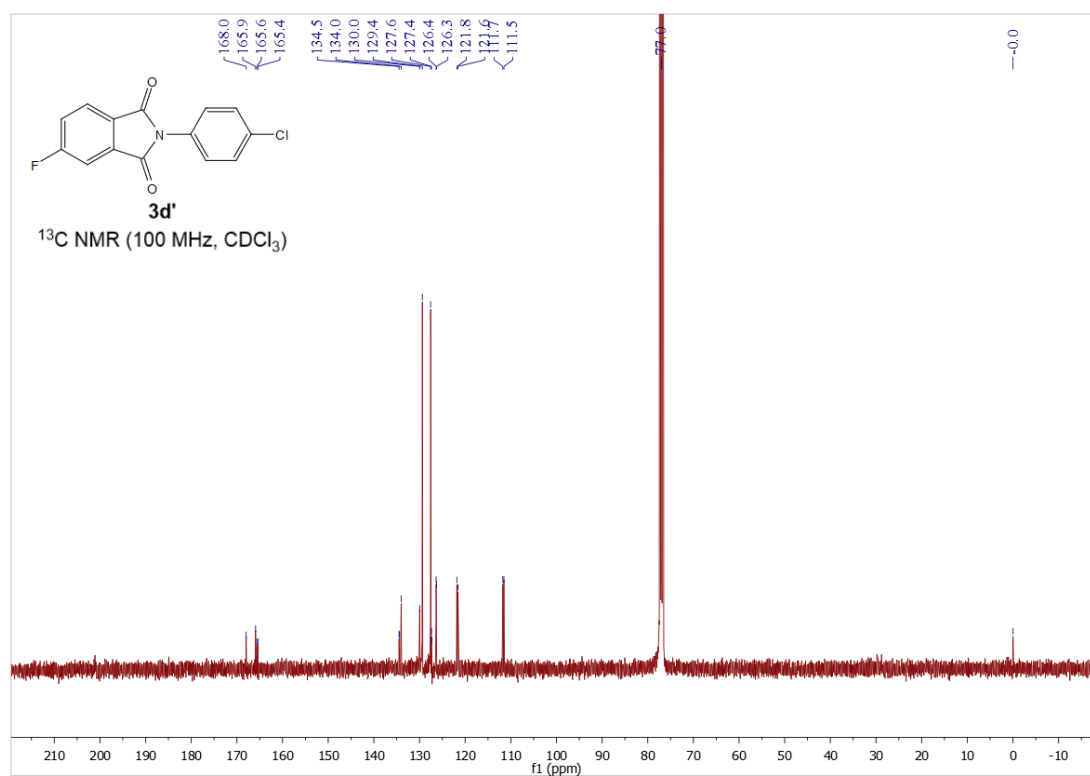
^{19}F NMR of Compound **3c'** (376 MHz, CDCl_3)



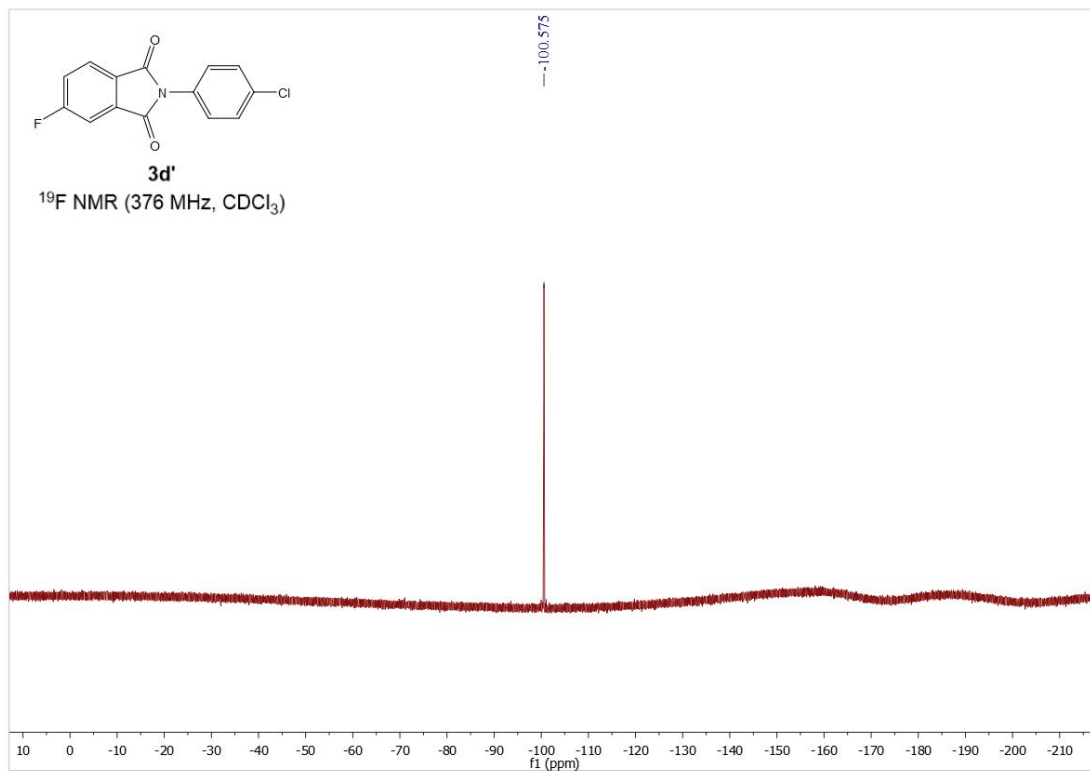
^1H NMR of Compound **3d'** (400 MHz, CDCl_3)



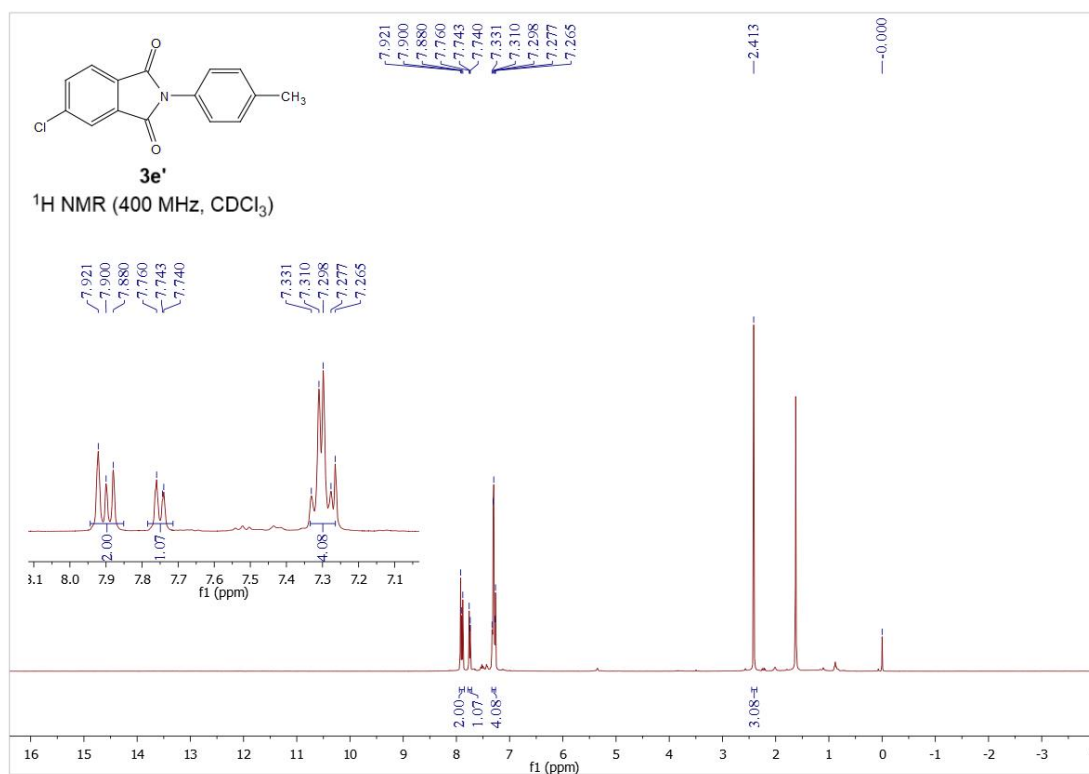
^{13}C NMR of Compound **3d'** (100 MHz, CDCl_3)



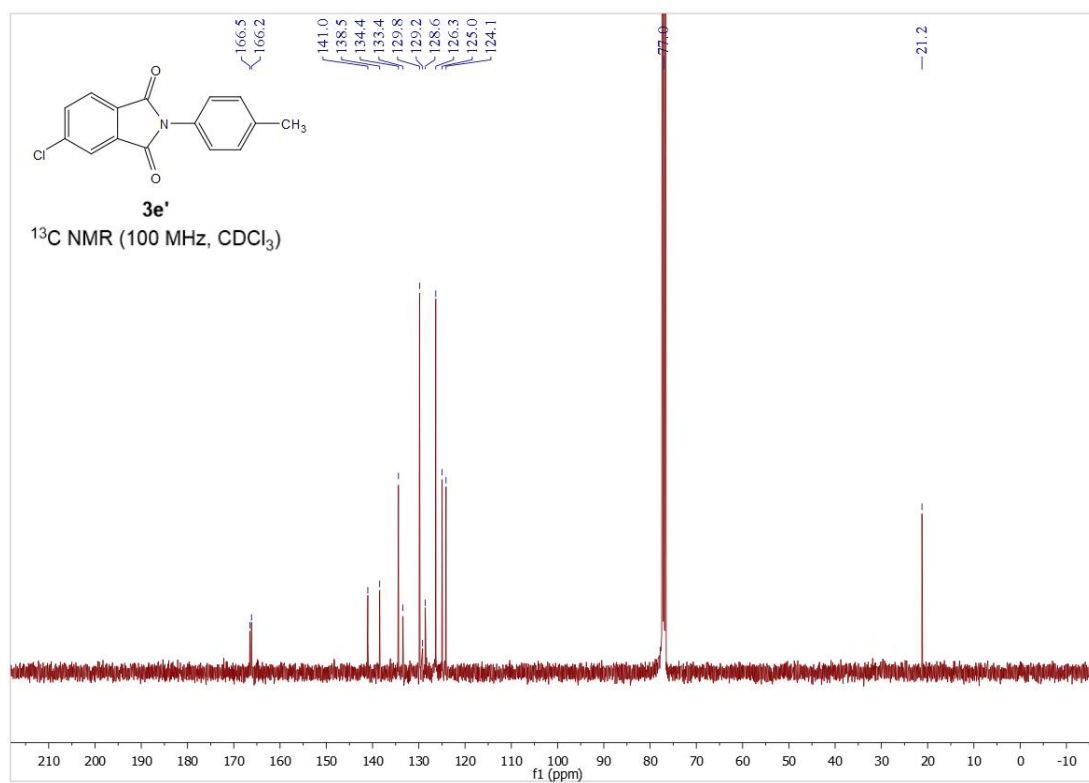
^{19}F NMR of Compound **3d'** (376 MHz, CDCl_3)



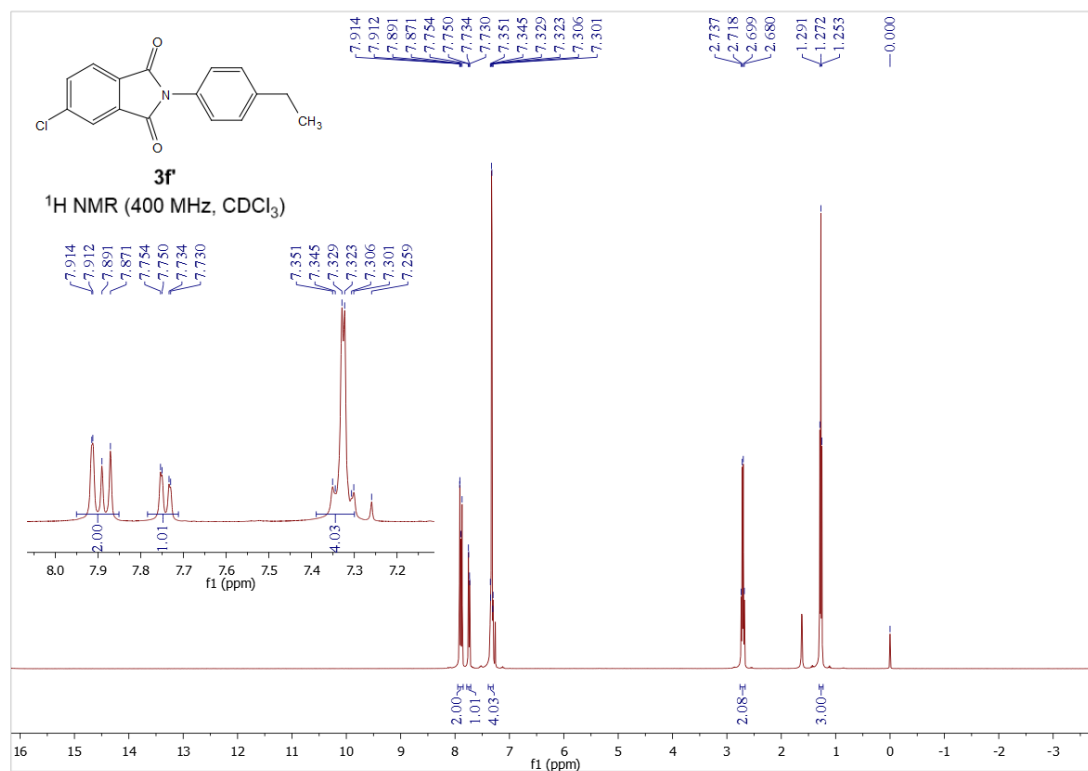
¹H NMR of Compound **3e'** (400 MHz, CDCl₃)



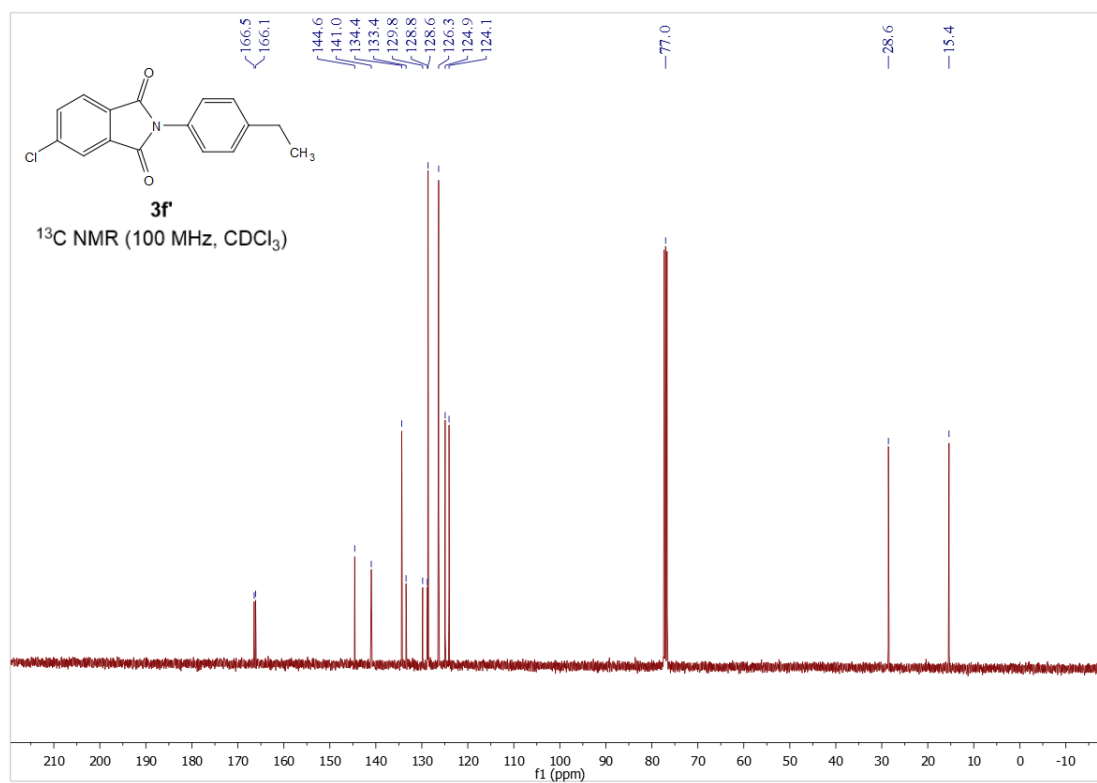
¹³C NMR of Compound **3e'** (100 MHz, CDCl₃)



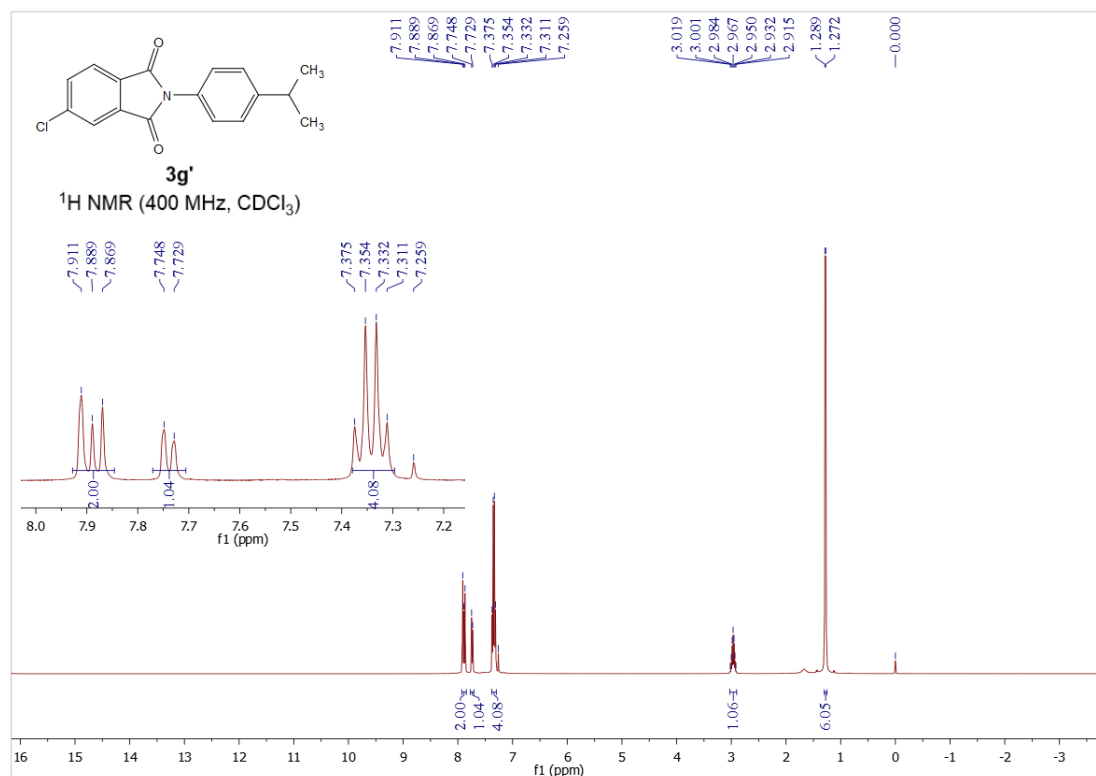
¹H NMR of Compound **3f'** (400 MHz, CDCl₃)



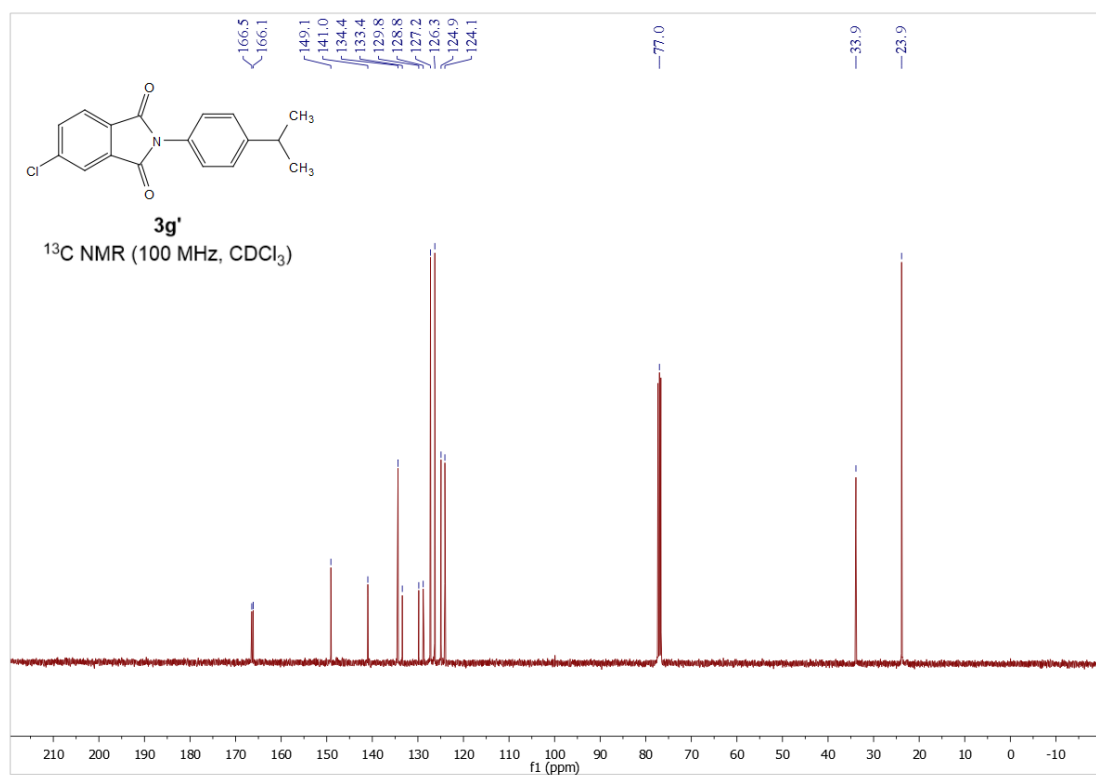
¹³C NMR of Compound **3f'** (100 MHz, CDCl₃)



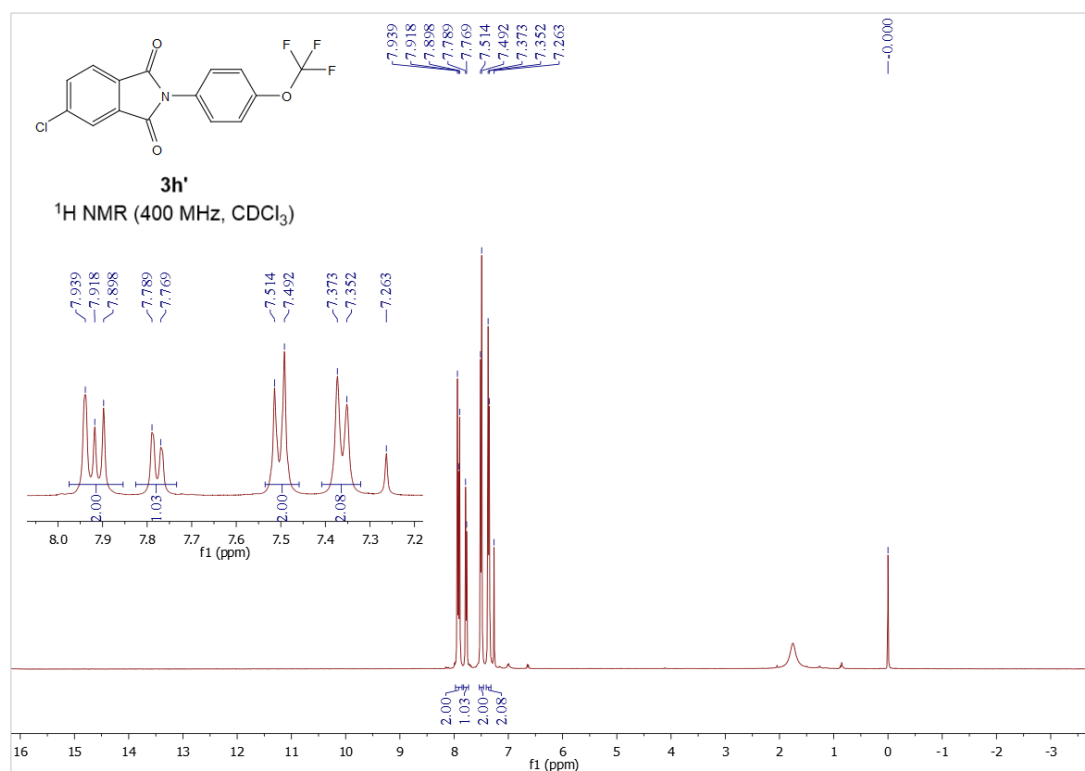
¹H NMR of Compound **3g'** (400 MHz, CDCl₃)



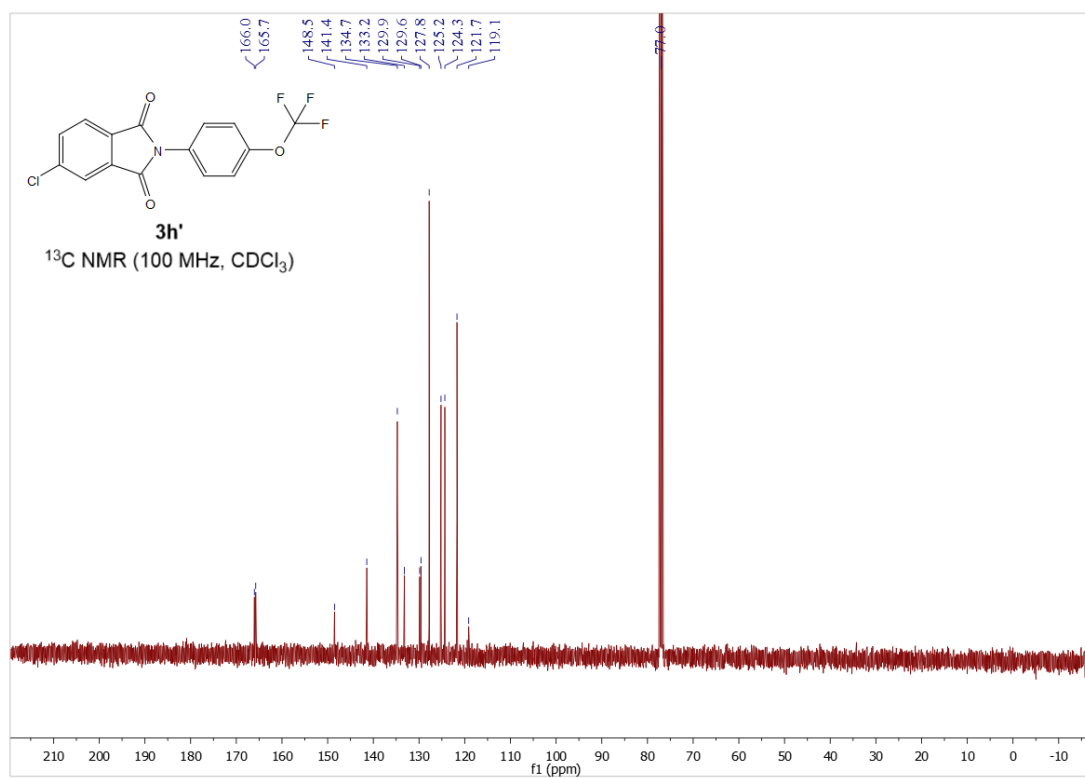
¹³C NMR of Compound **3g'** (100 MHz, CDCl₃)



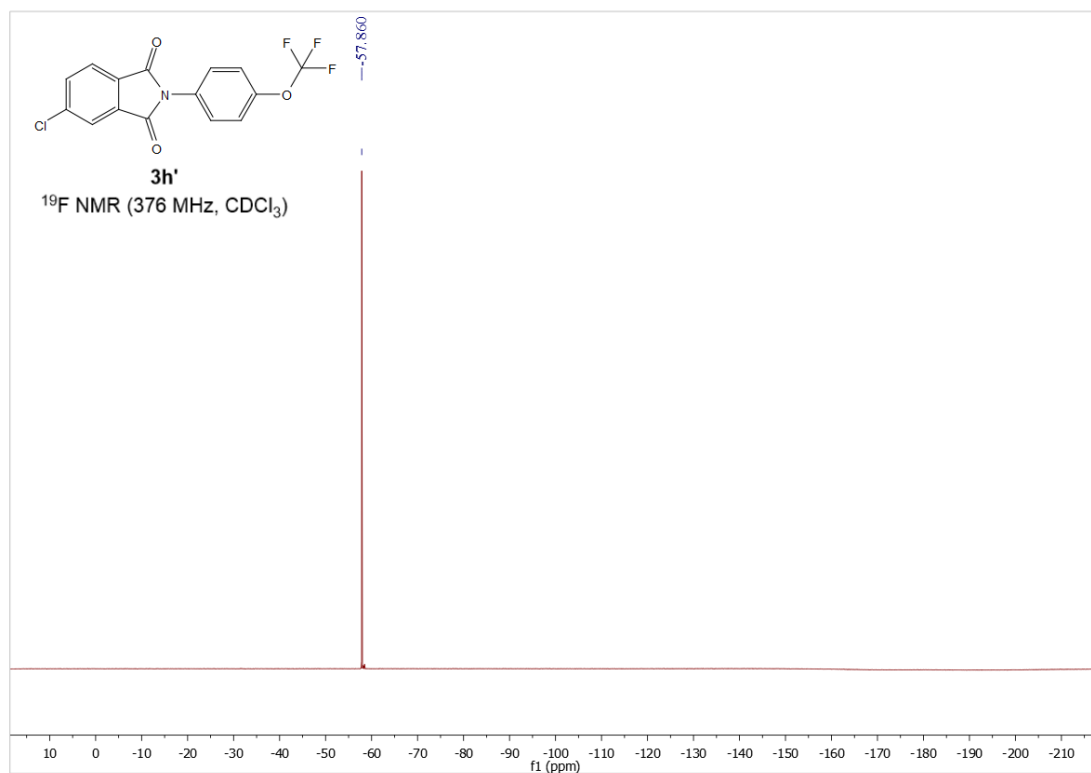
¹H NMR of Compound **3h'** (400 MHz, CDCl₃)



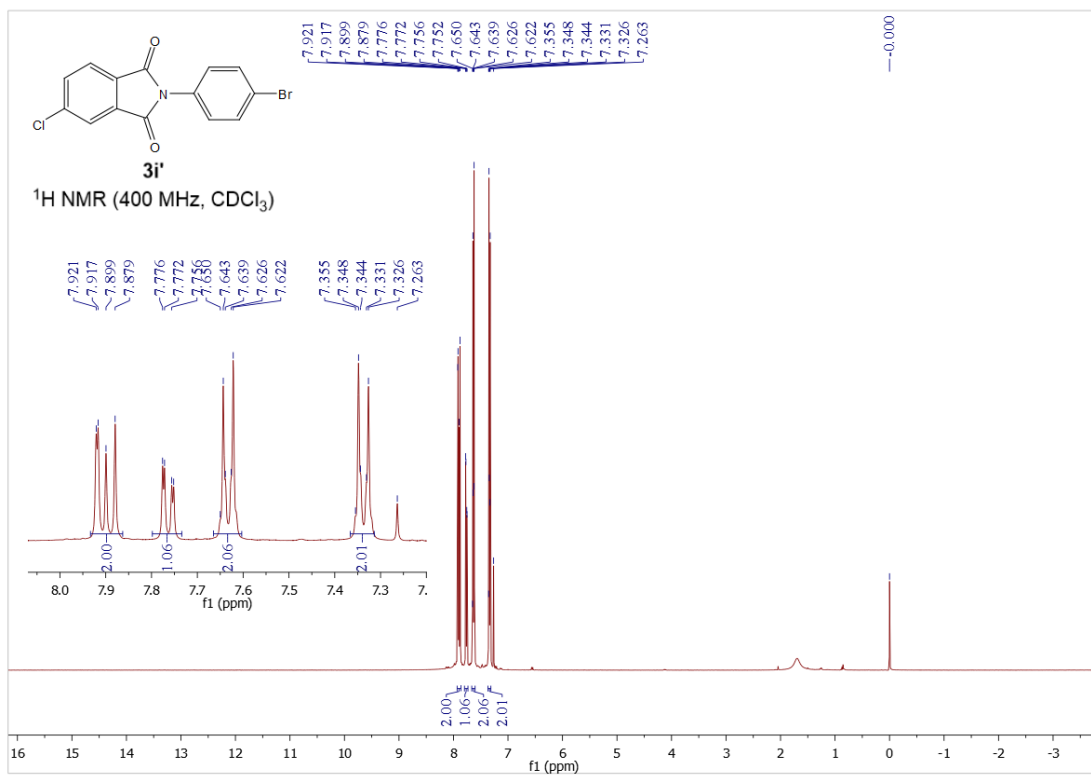
¹³C NMR of Compound **3h'** (100 MHz, CDCl₃)



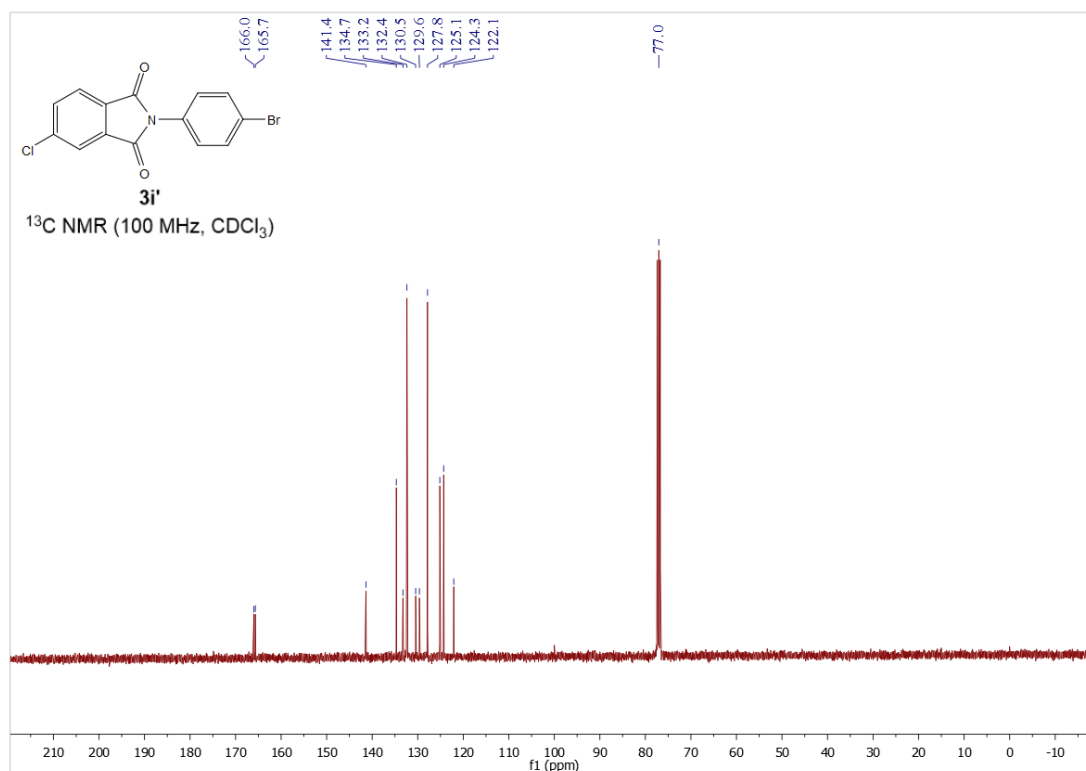
^{19}F NMR of Compound **3h'** (376 MHz, CDCl_3)



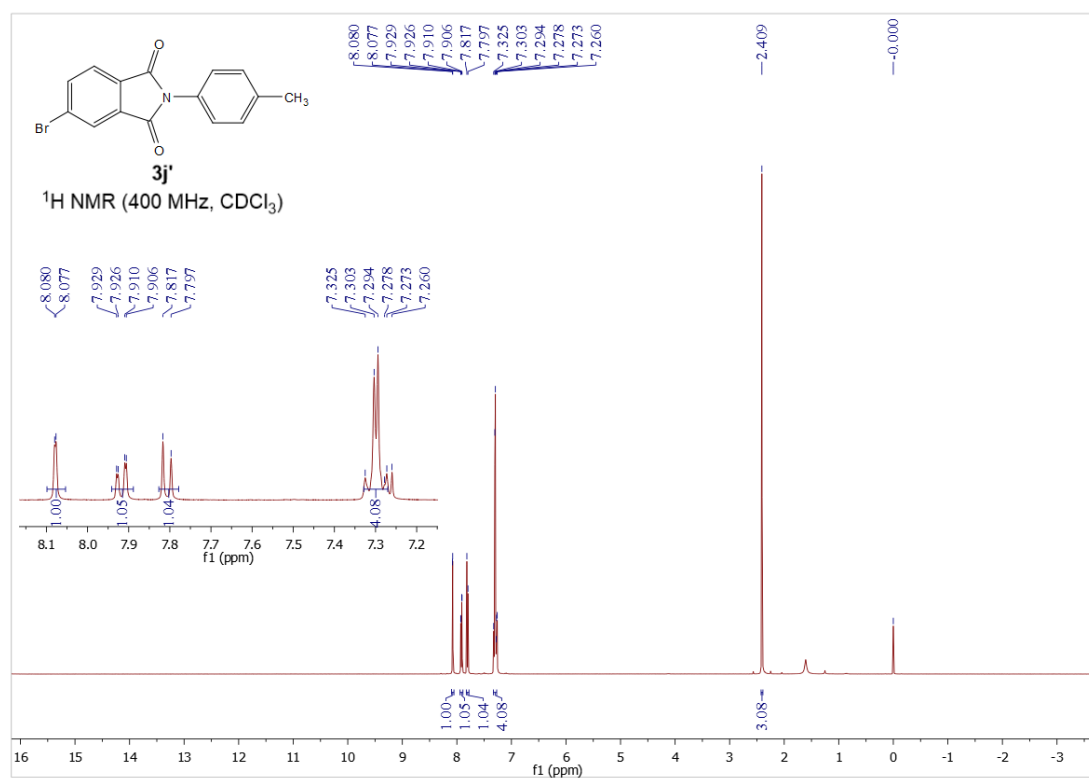
^1H NMR of Compound **3i'** (400 MHz, CDCl_3)



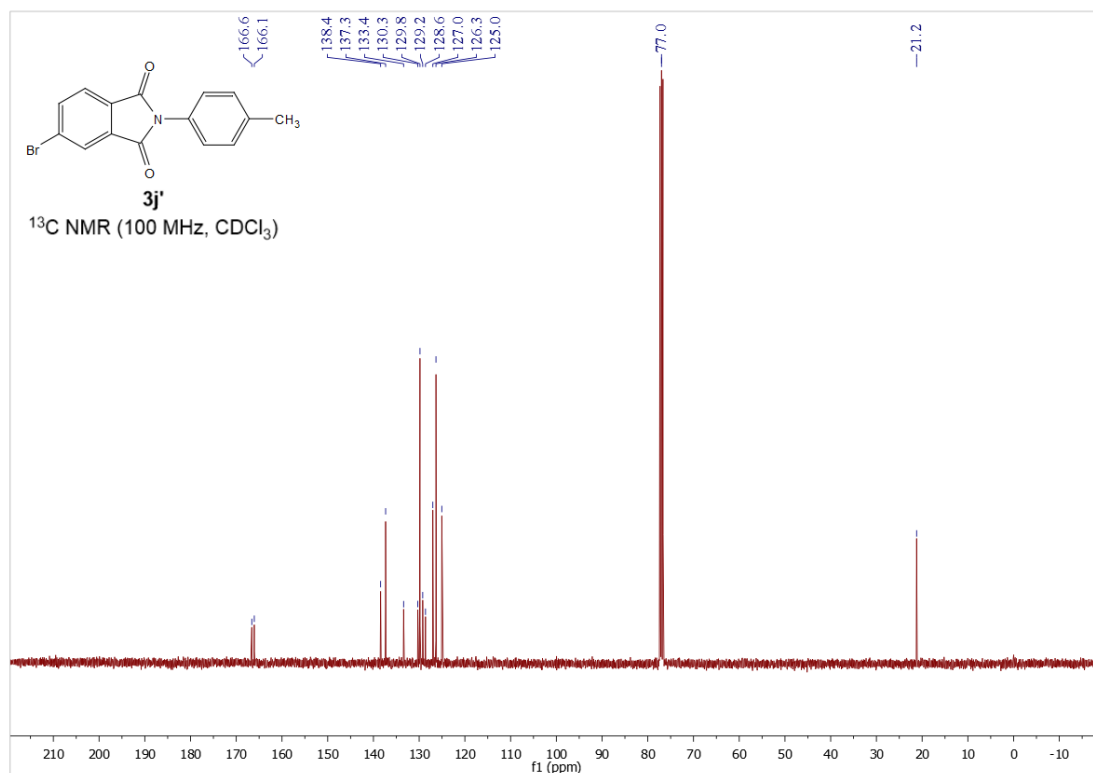
^{13}C NMR of Compound **3i'** (100 MHz, CDCl_3)



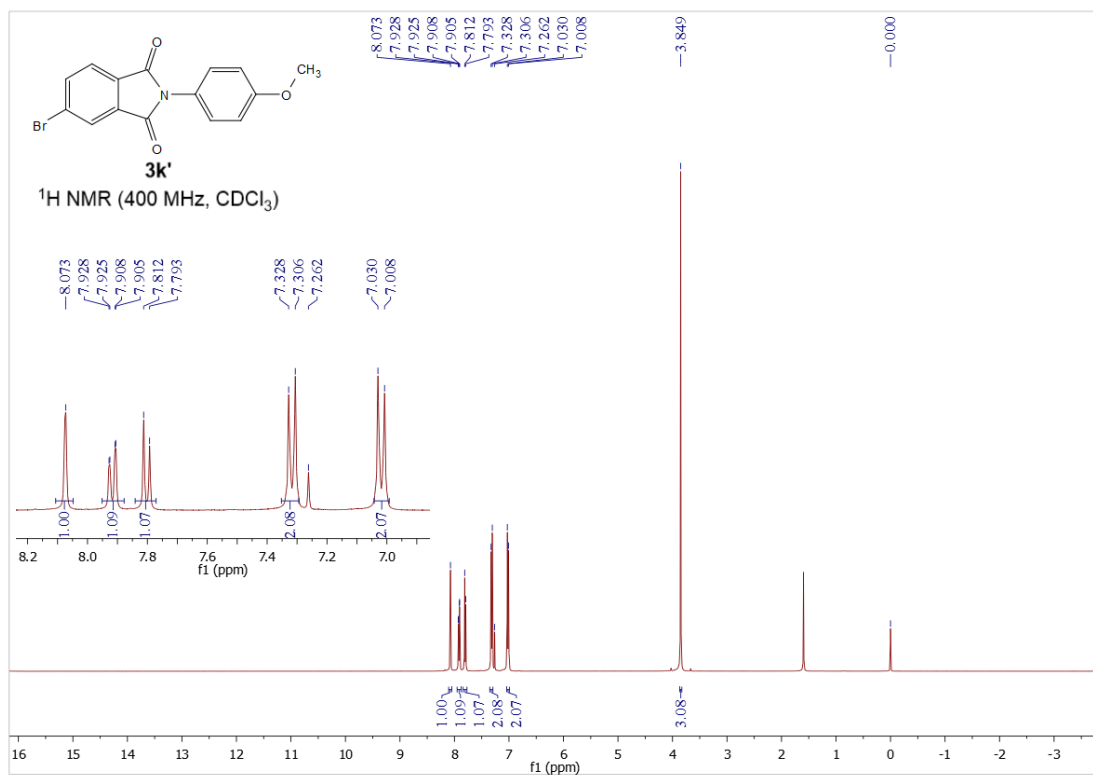
^1H NMR of Compound **3j'** (400 MHz, CDCl_3)



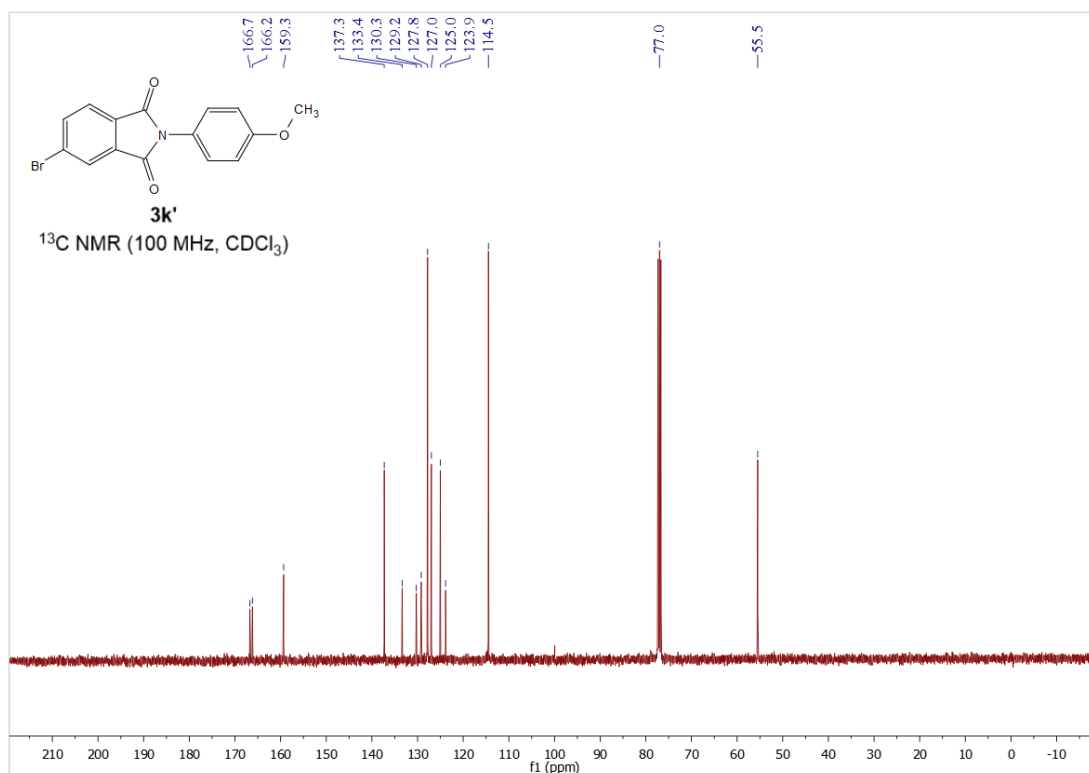
^{13}C NMR of Compound **3j'** (100 MHz, CDCl_3)



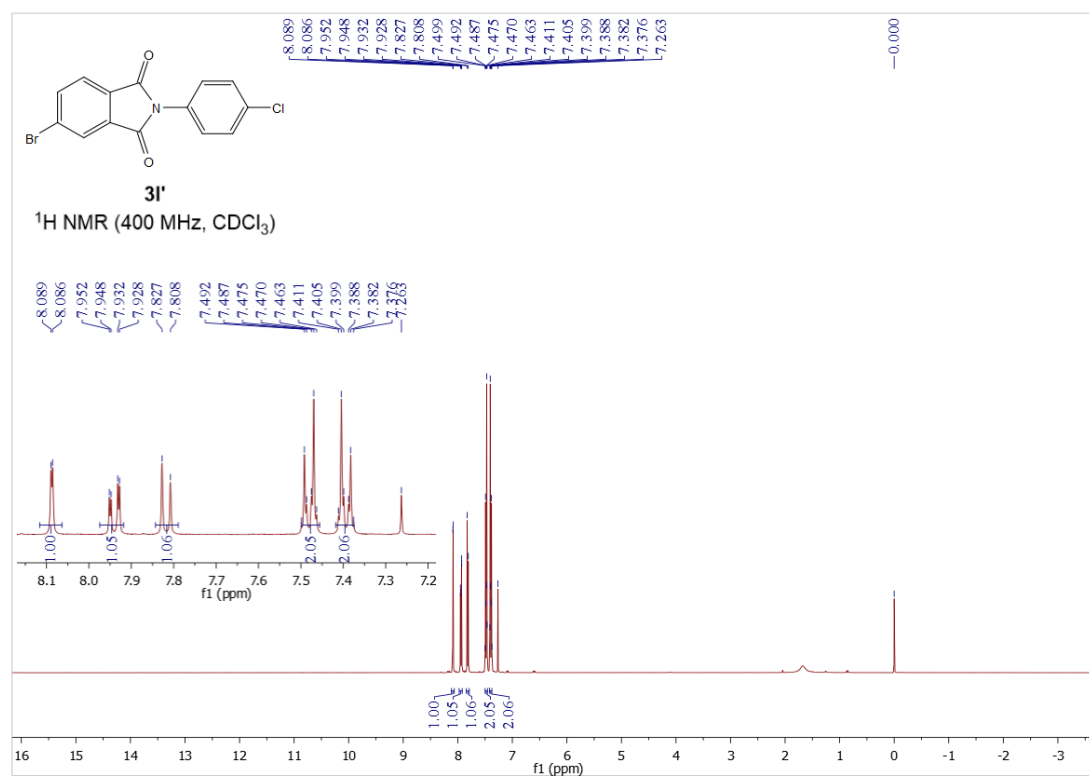
^1H NMR of Compound **3k'** (400 MHz, CDCl_3)



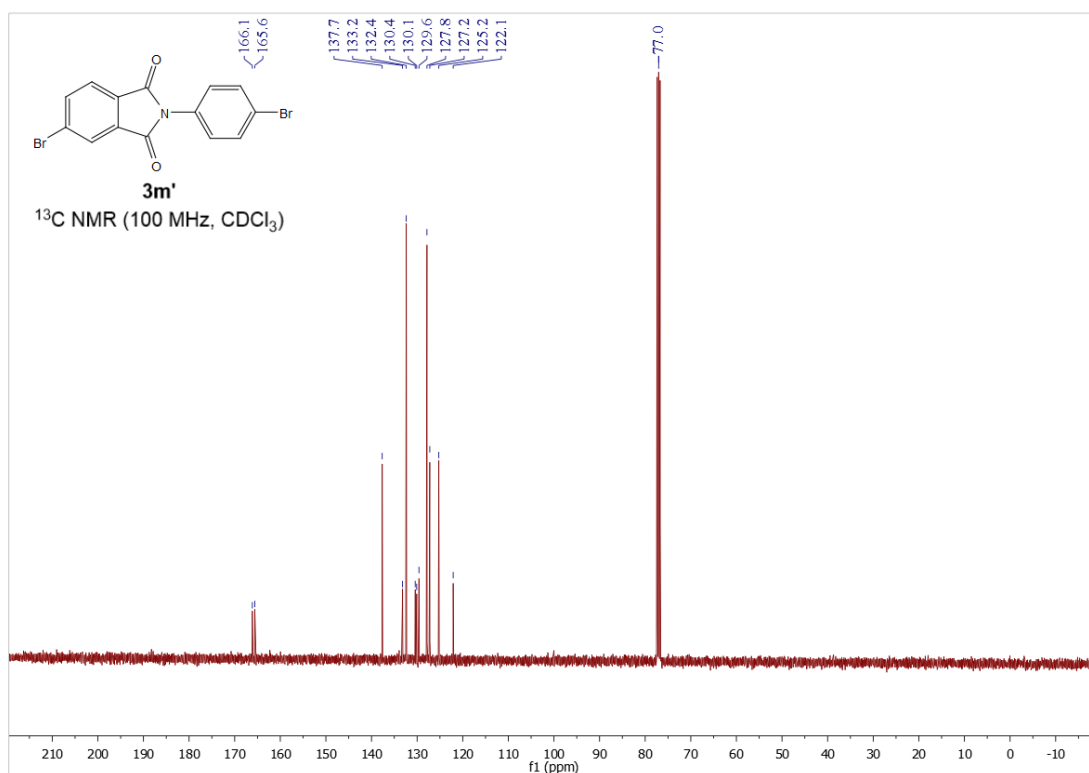
¹³C NMR of Compound **3k'** (100 MHz, CDCl₃)



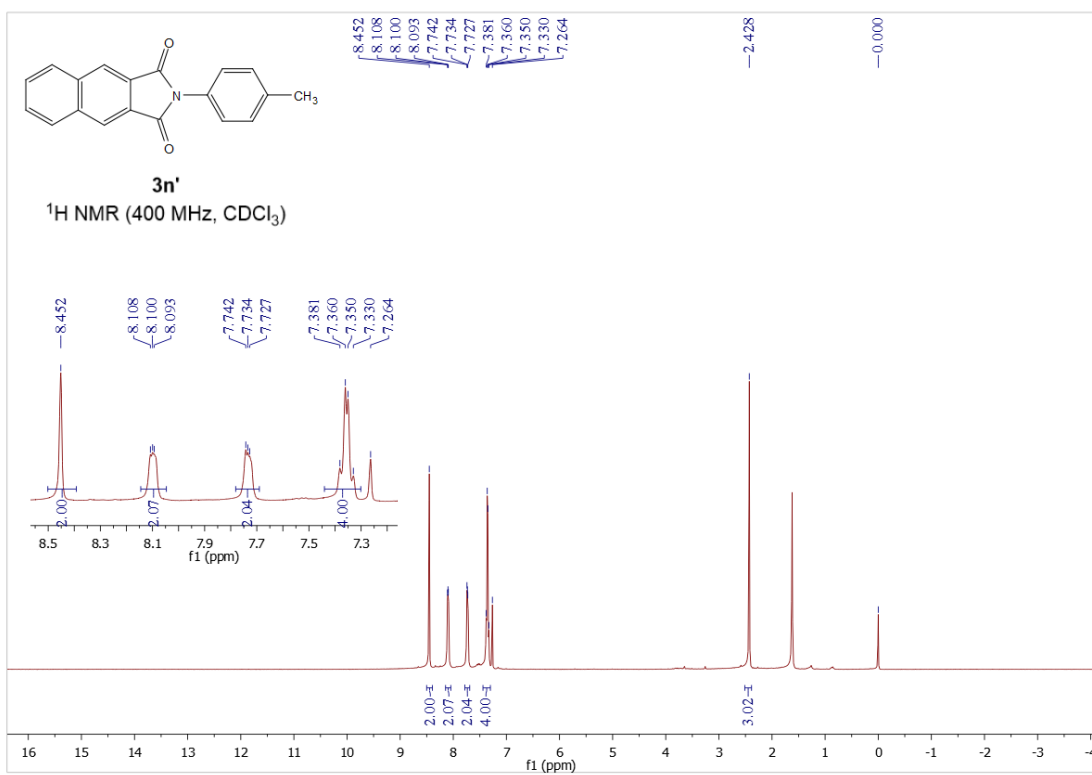
¹H NMR of Compound **3l'** (400 MHz, CDCl₃)



^{13}C NMR of Compound **3m'** (100 MHz, CDCl_3)



^1H NMR of Compound **3n'** (400 MHz, CDCl_3)



^{13}C NMR of Compound **3n'** (100 MHz, CDCl_3)

