Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Supporting Information

Selectively Forming Phthalimides from Amines, Aldehydes and CO by Pd-Catalyzed Oxidative C-H Aminocarbonylation

Renyi Shi, Fan Liao, Huiying Niu and Aiwen Lei

College of Chemistry and Molecular Sciences, the Institute for Advanced Studies (IAS), Wuhan University, Wuhan, Hubei 430072, P. R. China

National Research Center for Carbohydrate Synthesis, Jiangxi Normal University, Nanchang 330022, P. R. China

E-mail: aiwenlei@whu.edu.cn

Table of Contents

1. General

All reactions were carried out in oven-dried Schlenk tube under a mixed oxygen and carbon monoxide atmosphere with a balloon. Toluene and DMA were dried by molecular sieve. Unless otherwise noted, materials were obtained from commercial suppliers and used without further purification. Thin layer chromatography (TLC) employed glass 0.25 mm silica gel plates. GC yields were recorded with a SHIMADZU GC-2014 gas chromatograph instrument with a FID detector and biphenyl was added as an internal standard. ^{1}H and ^{13}C NMR data were recorded with Bruker Advanced II (400 MHz) spectrometers with tetramethylsilane as an internal standard. All chemical shifts (δ) are reported in ppm and coupling constants (*J*) in Hz. All chemical shifts are reported relative to tetramethylsilane or d^6-DMSO and d-solvent peaks (77.00 ppm, chloroform; 39.60 ppm, d^6-DMSO), respectively. High resolution massspectra (HRMS) were measured with a Bruker UltiMate3000 & Compact instrument and accurate masses were reported for the molecular ion $[M-H]^-$ or $[M+Na]^+$.

2. Pd-catalyzed aerobic imine-directed C-H bond carbonylation

General Procedure: In an oven-dried Schlenk tube equipped with a stir-bar, $PdCl_2$ (3.6 mg, 10 mol%), dppf (5.54 mg, 5 mol%), $Cu(OPiv)_2$ (10.6 mg, 20 mol%), amine (0.2 mmol, if it is solid.) were combined. A balloon filled with CO and oxygen (the ratio is 1:7 which beyond the explosion limit) was connected to the Schlenk tube via the side tube, then evacuated and refilled three times. All these operations involving CO must be in the fumehood with max air draft. Then DMSO (0.05 mL) and toluene (2.0 mL) were added to the tube via a syringe. At last, aldehyde (6 eq., If the amine is liquid, it will be injected together) was added to the tube. The Schlenk tube was heated at 110 °C for 24 hours and then cooled to room temperature. After the balloon gas was released carefully in the fumehood, the reaction was quenched by water and extracted with CH_2Cl_2 three times. The combined organic layers were dried over anhydrous Na_2SO_4 and evaporated in vacuum. The desired products were obtained in the corresponding yields after purification by flash chromatography on silica gel with hexane and ethyl acetate(10/1).

3. Preparation of *d*^{5}-benzoaldehyde

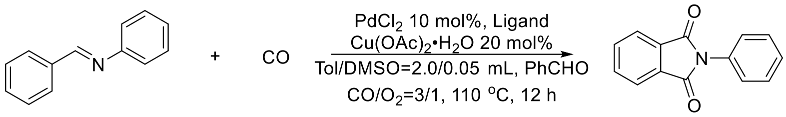
General Procedure[11]: Hexadeuterobenzene (0.84 g, 10 mmol) was added to the mixture of butyllithium (10 mmol) and potassium *tert*-butoxide (10 mmol) in hcxane (4 mL). After 1 h of vigorous stirring, DMF (10 mmol) was added and the products isolated by extraction and fractional distillation.

4. Preparation of 2-(*p*-tolyl)isoindolin-1-one

General Procedure[10]: *o*-Phthalaldehyde (10 mmol) and *p*-toluidine (10 mmol) were placed in a flame-dried round-bottomed flask, 15 mL of CH3CN/DMF (CH_3CN:DMF = 2:1) was used as reaction medium, 8 mmol TMSCl was added, and the mixture was stirred for 7-9 h at room temperature. The completion of reactions was determined by TLC. The mixture was extracted with (3*10 mL) acetate ester. The combined organic layer was evaporated in vacuum, and the residue was obtained as analytically pure product. An additional recrystallization with ethanol was carried out when necessary.

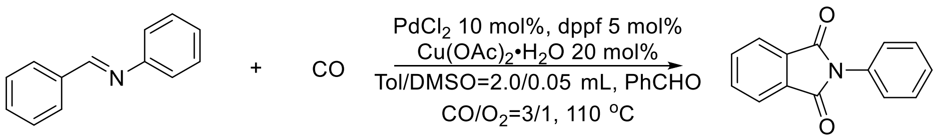
5. Optimization of reaction conditions

Optimization of ligands:



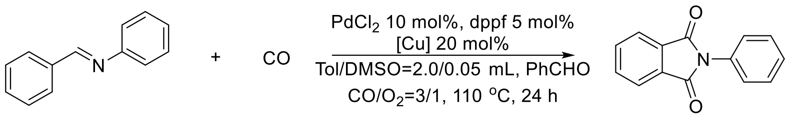
Entry	Ligand	Yield%	Con.%
1	none	43	100
2	dppm 5 mol%	43	93
3	dppe 5 mol%	23	53
4	dppp 5 mol%	29	77
5	dppb 5 mol%	34	78
6	dppf 5 mol%	50	97
7	Xantphos 5 mol%	35	79
8	DPEPhos 5 mol%	41	88
9	1,10-phenanthroline 5 mol%	0	0
10	PPh_3 10 mol%	32	85
11	tris(2,4-di-tert-butylphenyl)phosphite 5 mol%	7	27
12	di-tert-butylphenylphosphine 5 mol%	34	80
13	PCy_3 5 mol%	24	65
14	Ad_2PBu 5 mol%	24	64

Optimization of reaction time:



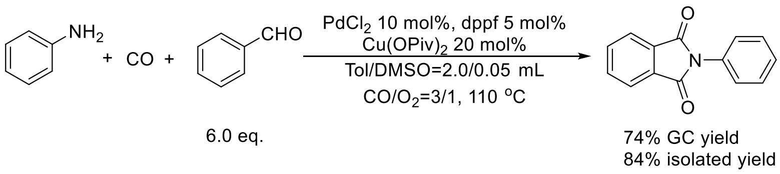
Entry	Reaction time	Yield%	Con.%
1	12 h	27	82
2	18 h	37	90
3	24 h	48	100

Optimization of coopper catalysts:



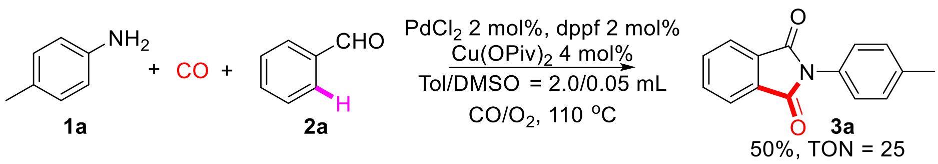
Entry	[Cu]	Yield%	Con.%
1	$Cu(OAc)_2$•H_2O	52	100
2	Cu(*i*-PrCOO)$_2$	52	100
3	$Cu(EtCOO)_2$	47	100
4	$Cu(C_5H_{11}COO)_2$	58	100
5	$Cu(C_{11}H_{23}COO)_2$	59	100
6	$Cu(OPiv)_2$	63	100
7	$Cu(PrCOO)_2$	51	100
8	$CuBr_2$	43	100
9	CuCl	28	65
10	CuBr	40	96
11	CuI	3	15

After small adjustment of solvent, we get 74% GC yield(84% isolated yield) with the combination of $PdCl_2$ (10 mol%), dppf (5 mol%) and $Cu(OPiv)_2$ (20 mol%) in a mixture of toluene and DMSO (2.0/0.05 mL) under the atmosphere of CO/O_2(the ratio is 1/7 which beyond the explosion limit, this operation must be fumehood) at 110 °C for 24 h.



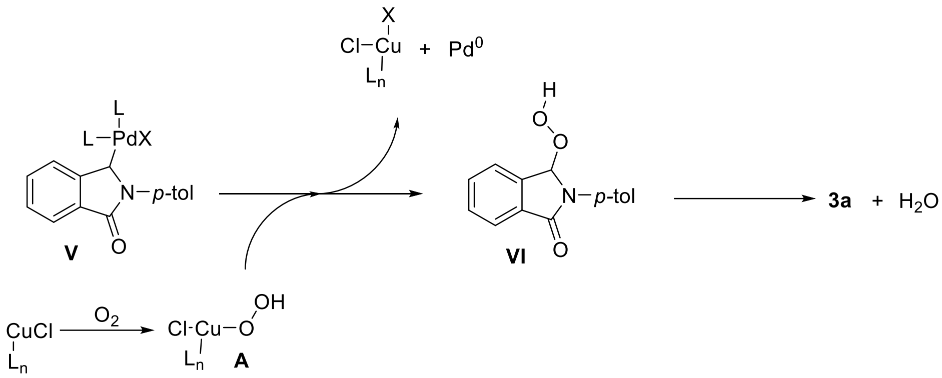
6. Scale-up experiment

General Procedure: In an oven-dried Schlenk tube equipped with a stir-bar, $PdCl_2$ (3.6 mg, 2 mol%), dppf (5.54 mg, 1 mol%), $Cu(OPiv)_2$ (10.6 mg, 4 mol%), p-toluidine (1 mmol) were combined. A balloon filled with CO and oxygen (the ratio is 1:7 which beyond the explosion limit) was connected to the Schlenk tube via the side tube, then evacuated and refilled three times. All these operations involving CO must be in the fumehood with max air draft. Then DMSO (0.05 mL) and toluene (2.0 mL) were added to the tube via a syringe. At last, aldehyde (6 eq.) was added to the tube. The Schlenk tube was heated at 110 °C for 24 hours and then cooled to room temperature. After the balloon gas was released carefully in the fumehood, the reaction was quenched by water and extracted with CH_2Cl_2 three times. The combined organic layers were dried over anhydrous Na_2SO_4 and evaporated in vacuum. The desired products were obtained in the corresponding yields after purification by flash chromatography on silica gel with hexane and ethyl acetate(10/1).

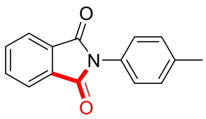


7. Proposed mechanism from V to 3a

Cu^I intermediate reacts with O_2 to generate **A** where Cl^- plays as a ligand.[13] Then **A** reacts with intermediate **V** to give **VI** and Pd^0. **VI** smoothly runs elimination to give desired product **3a**. On the other hand, Pd^0 would be oxidized by Cu^{II}/O_2 to obtain Pd^{II}.[14]



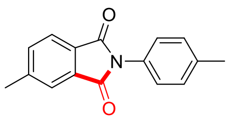
8. Analytical Data of Products



3a[1] **2-(*p*-tolyl)isoindoline-1, 3-dione**: white solid with 92% isolated yield(hexane/EA = 10/1).

^{1}H NMR (400 MHz, *d*^{6}-DMSO) δ 7.96-7.93 (m, 2H), 7.91-7.88 (m, 2H), 7.32 (t, *J* = 8.6 Hz, 4H), 2.37 (s, 3H).

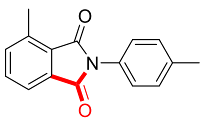
^{13}C NMR (101 MHz, *d*^{6}-DMSO) δ 167.27, 137.77, 134.81, 131.68, 129.48, 129.38, 127.38, 123.51, 20.90.



3b[8] **5-methyl-2-(*p*-tolyl)isoindoline-1,3-dione**: white solid with 70% isolated yield(hexane/EA = 10/1).

^{1}H NMR (400 MHz, *d*^{6}-DMSO) δ 7.83 (d, *J* = 7.6 Hz, 1H), 7.77 (s, 1H), 7.39 (d, *J* = 7.6 Hz, 1H), 7.33-7.28 (m, 4H), 2.51 (s, 3H), 2.37 (s, 3H).

^{13}C NMR (101 MHz, *d*^{6}-DMSO) δ 167.26, 167.17, 145.72, 137.61, 135.11, 131.98, 129.40(2C), 129.01, 127.26, 123.87, 123.40, 21.51, 20.85.

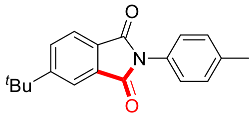


3c 4-methyl-2-(p-tolyl)isoindoline-1, 3-dione: white solid with 85% isolated yield(hexane/EA = 10/1).

^{1}H NMR (400 MHz, *d*^{6}-DMSO) δ 7.76-7.74 (m, 2H), 7.69-7.56 (m, 1H), 7.33-7.28 (m, 4H), 2.65 (s, 3H), 2.37 (s, 3H).

^{13}C NMR (101 MHz, *d*^{6}-DMSO) δ 167.89, 167.11, 137.67, 137.58, 136.82, 134.27, 132.05, 129.44, 129.37, 128.25, 127.43, 121.13, 20.89, 17.33.

HRMS (ESI) calcd for $C_{16}H_{13}NNaO_2$ $[M+Na]^+$: 274.0838; Found: 274.0832.

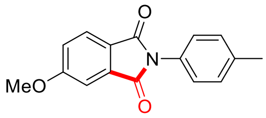


3e 5-(tert-butyl)-2-(*p*-tolyl)isoindoline-1, 3-dione: white solid with 67% isolated yield(hexane/EA = 10/1).

^{1}H NMR (400 MHz, d^6-DMSO) δ 7.93-7.91 (m, 2H), 7.87 (d, J = 8.4 Hz, 1H), 7.33-7.28 (m, 4H), 2.37 (s, 3H), 1.37 (s, 9H).

^{13}C NMR (101 MHz, d^6-DMSO) δ 167.36, 167.05, 158.49, 137.64, 131.93, 131.60, 129.45, 129.42, 129.14, 127.32, 123.40, 120.30, 35.63, 30.90, 20.86.

HRMS (ESI) calcd for $C_{19}H_{19}NNaO_2$ $[M+Na]^+$: 316.1308; Found: 316.1319.

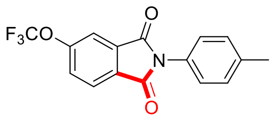


3f 5-methoxy-2-(*p*-tolyl)isoindoline-1, 3-dione: white solid with 58% isolated yield(hexane/EA = 10/1).

^{1}H NMR (400 MHz, d^6-DMSO) δ 7.86 (dd, J = 8.2 Hz, 1.0 Hz 1H), 7.45-7.44 (m, 1H), 7.37-7.35 (m, 1H), 7.33-7.28 (m, 4H), 3.94 (s, 3H), 2.36 (s, 3H).

^{13}C NMR (101 MHz, d^6-DMSO) δ 166.83(2C), 164.64, 137.58, 134.27, 129.45, 129.41, 127.22, 125.37, 123.38, 120.25, 108.45, 56.45, 40.23, 40.02, 20.86.

HRMS (ESI) calcd for $C_{16}H_{13}NNaO_3$ $[M+Na]^+$: 290.0788; Found: 290.0787.



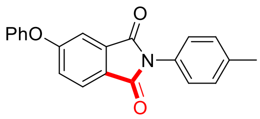
3h 2-(p-tolyl)-5-(trifluoromethoxy)isoindoline-1, 3-dione: white solid with 70% isolated yield(hexane/EA = 10/1).

^{1}H NMR (400 MHz, d^6-DMSO) δ 8.10 (d, J = 8.0 Hz, 1H), 7.95 (s, 1H), 7.88-7.87 (m, 1H), 7.35-7.30 (m, 4H), 2.37 (s, 3H).

^{13}C NMR (101 MHz, d^6-DMSO) δ 166.12, 165.84, 152.51, 137.98, 134.38, 130.55, 129.51, 129.21, 127.33, 127.09, 125.91, 119.99 (q, J = 259.27 Hz), 116.33, 20.90.

^{19}F NMR (377 MHz, DMSO) δ -56.89.

HRMS (ESI) calcd for $C_{16}H_9F_3NO_3$ $[M-H]^-$: 320.0540; Found: 320.0551.

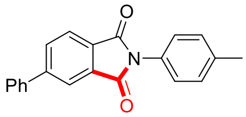


3i 5-phenoxy-2-(*p*-tolyl)isoindoline-1, 3-dione: white solid with 67% isolated yield(hexane/EA = 10/1).

^{1}H NMR (400 MHz, d^6-DMSO) δ 7.95 (d, J = 8.0 Hz, 1H), 7.52 (t, J = 8.0 Hz, 2H), 7.41 (dd, J = 8.2, 2.2 Hz, 1H), 7.33-7.27 (m, 6H), 7.20 (d, J = 7.6 Hz, 2H), 2.36 (s, 3H).

^{13}C NMR (101 MHz, d^6-DMSO) δ 166.61, 166.47, 162.89, 154.78, 137.76, 134.37, 130.75, 129.47, 129.34, 127.28, 125.96, 125.54, 125.47, 122.94, 120.41, 111.56, 20.89.

HRMS (ESI) calcd for $C_{21}H_{15}NNaO_3$ $[M+Na]^+$: 352.0944; Found: 352.0946.

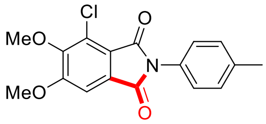


3j 5-phenyl-2-(*p*-tolyl)isoindoline-1, 3-dione: white solid with 74% isolated yield(hexane/EA = 10/1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.19 (d, *J* = 1.2 Hz, 1H), 8.05-8.00 (m, 2H), 7.71-7.69 (m, 2H), 7.57-7.48 (m, 3H), 7.38-7.34 (m, 4H), 2.45 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 167.37, 167.28, 147.72, 138.92, 138.15, 132.83, 132.61, 130.14, 129.75, 129.18, 128.94, 128.88, 127.31, 126.41, 124.11, 122.19, 21.20.

HRMS (ESI) calcd for $C_{21}H_{15}NNaO_2$ $[M+Na]^+$: 336.0995; Found: 336.1000.

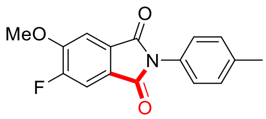


3k 4-chloro-5, 6-dimethoxy-2-(*p*-tolyl)isoindoline-1,3-dione: white solid with 48% isolated yield(hexane/EA = 10/1).

^{1}H NMR (400 MHz, d^6-DMSO) δ 7.60 (s, 1H), 7.33-7.27 (m, 4H), 4.04 (s, 3H), 3.86 (s, 3H), 2.37 (s, 3H).

^{13}C NMR (101 MHz, d^6-DMSO) δ 165.73, 164.78, 158.59, 149.65, 137.81, 129.47, 129.29, 128.80, 127.41, 124.43, 120.19, 106.85, 60.91, 57.42, 20.90.

HRMS (ESI) calcd for $C_{17}H_{14}ClNNaO_4$ $[M+Na]^+$: 354.0504; Found: 354.0508.



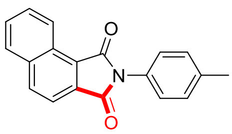
3l 5-fluoro-6-methoxy-2-(*p*-tolyl)isoindoline-1, 3-dione: white solid with 50% isolated yield(hexane/EA = 10/1).

^{1}H NMR (400 MHz, d^6-DMSO) δ 7.88 (d, *J* = 9.6 Hz, 1H), 7.74 (d, *J* = 7.2 Hz, 1H), 7.33-7.28 (m, 4H), 4.04 (s, 3H), 2.36 (s, 3H).

^{13}C NMR (101 MHz, d^6-DMSO) δ 166.45, 166.19, 155.14 (d, *J* = 254.82), 152.83 (d, *J* = 11.31 Hz), 137.72, 129.48, 129.37, 129.19 (d, *J* = 3.13 Hz), 127.24, 124.20 (d, *J* = 8.69 Hz), 111.65 (d, *J* = 21.92 Hz), 108.87 (d, *J* = 2.83 Hz), 57.32, 20.89.

^{19}F NMR (377 MHz, DMSO) δ -124.82.

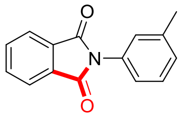
HRMS (ESI) calcd for $C_{16}H_{12}FNNaO_3$ $[M+Na]^+$: 308.0693; Found: 308.0693.



3m[12] 2-(p-tolyl)-1H-benzo[*e*]isoindole-1, 3(*2H*)-dione: white solid with 45% isolated yield(hexane/EA = 10/1).

^{1}H NMR (400 MHz, d^6-DMSO) δ 8.83 (d, *J* = 8.4 Hz, 1H), 8.44 (d, *J* = 8.0 Hz, 1H), 8.20 (d, *J* = 8.4 Hz, 1H), 7.85 (d, *J* = 7.6 Hz, 1H), 7.84 (t, *J* = 7.4 Hz, 1H), 7.77 (t, *J* = 7.6 Hz, 1H), 7.39-7.33 (m, 4H), 2.39 (s, 3H).

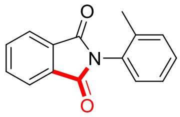
^{13}C NMR (101 MHz, d^6-DMSO) δ 168.43, 167.67, 137.64, 136.35, 135.66, 131.13, 130.03, 129.49, 129.36, 129.34, 129.01, 127.44, 127.41, 126.65, 124.11, 118.73, 20.92.



4a[2] **2-(*m*-tolyl)isoindoline-1, 3-dione**: white solid with 91% isolated yield(hexane/EA = 10/1).

^{1}H NMR (400 MHz, d^6-DMSO) δ 7.96-7.93 (m, 2H), 7.92-7.89 (m, 2H), 7.41 (t, J = 7.8 Hz, 1H), 7.25 (t, J = 9.2 Hz, 3H), 2.36 (s, 3H).

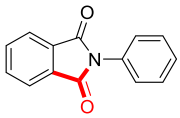
^{13}C NMR (101 MHz, d^6-DMSO) δ 167.17, 138.46, 134.83, 131.91, 131.63, 128.89, 128.81, 127.96, 124.65, 123.52, 20.99.



4b[1] **2-(*o*-tolyl)isoindoline-1, 3-dione**: white solid with 92% isolated yield(hexane/EA = 10/1).

^{1}H NMR (400 MHz, d^6-DMSO) δ 7.99-7.96 (m, 2H), 7.94-7.91 (m, 2H), 7.41 (dd, J = 4.4 Hz, 2.0 Hz, 2H), 7.38-7.33 (m, 2H), 2.12 (s, 3H).

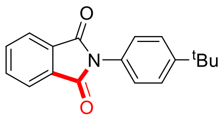
^{13}C NMR (101 MHz, d^6-DMSO) δ 167.15, 136.44, 134.90, 131.76, 131.07, 130.80, 129.36, 129.30, 126.79, 123.68, 17.56.



4c[1] **2-phenylisoindoline-1, 3-dione**: white solid with 86% isolated yield(hexane/EA = 10/1).

^{1}H NMR (400 MHz, d^6-DMSO) δ 7.98-7.95 (m, 2H), 7.92-7.89 (m, 2H),7.55-7.51 (m, 2H), 7.46-7.42 (m, 3H).

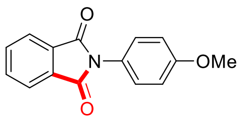
^{13}C NMR (101 MHz, d^6-DMSO) δ 167.15, 134.83, 132.01, 131.65, 128.98, 128.20, 127.53, 123.55.



4d[1] **2-(4-(*tert*-butyl)phenyl)isoindoline-1, 3-dione**: white solid with 99% isolated yield(hexane/EA = 10/1).

^{1}H NMR (400 MHz, d^6-DMSO) δ 7.97-7.94 (m, 2H), 7.92-7.89 (m, 2H), 7.54 (d, J = 8.4 Hz, 2.0 Hz, 2H), 7.36 (d, J = 8.8 Hz, 2.0 Hz, 2H), 1.33 (s, 9H).

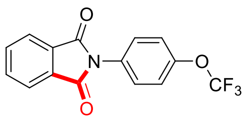
^{13}C NMR (101 MHz, d^6-DMSO) δ 167.20, 150.64, 134.75, 131.62, 129.33, 127.01, 125.74, 123.47, 34.54, 31.17.



4e[2] **2-(4-methoxyphenyl)isoindoline-1, 3-dione**: white solid with 70% isolated yield(hexane/EA = 10/1).

^{1}H NMR (400 MHz, d^6-DMSO) δ 7.96-7.93 (m, 2H), 7.91-7.88 (m, 2H), 7.35 (dt, J = 8.8 Hz, 2.6 Hz, 2H), 7.35 (dt, J = 12 Hz, 2.8 Hz, 2H), 3.81 (s, 3H).

^{13}C NMR (101 MHz, d^6-DMSO) δ 167.39, 158.97, 134.73, 131.69, 128.88, 124.51, 123.44, 114.23, 55.50.

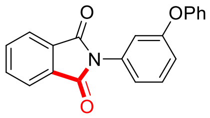


4f[4] **2-(4-(trifluoromethoxy)phenyl)isoindoline-1, 3-dione**: white solid with 82% isolated yield(hexane/EA = 10/1).

^{1}H NMR (400 MHz, d^{6}-DMSO) δ 8.00-7.97 (m, 2H), 7.94-7.90 (m, 2H), 7.62 (dt, J = 8.8 Hz, 2.4 Hz, 2H), 7.55 (d, J = 8.8 Hz, 2H).

^{13}C NMR (101 MHz, d^{6}-DMSO) δ 166.89, 147.57, 134.87, 131.62, 131.09, 129.37, 123.60, 121.69, 120.16 (t, J = 257.8 Hz).

^{19}F NMR (377 MHz, DMSO) δ -56.88.

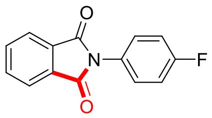


4g 2-(3-phenoxyphenyl)isoindoline-1,3-dione: white solid with 77% isolated yield(hexane/EA = 10/1).

^{1}H NMR (400 MHz, d^{6}-DMSO) δ 7.95-7.92 (m, 2H), 7.90-7.87 (m, 2H), 7.52 (t, J = 8.2 Hz, 1H), 7.42 (t, J = 8.0 Hz, 2H), 7.23 (dq, J = 8.0 Hz, 0.8 Hz, 1H), 7.19-7.14 (m, 2H), 7.10-7.05(m, 3H).

^{13}C NMR (101 MHz, d^{6}-DMSO) δ 166.93, 157.04, 156.10, 134.83, 133.41, 131.63, 130.31, 130.24, 124.08, 123.55, 122.26, 119.16, 117.86, 117.38.

HRMS (ESI) calcd for $C_{20}H_{13}NNaO_3$ [M+Na]$^{+}$: 338.0788; Found: 338.0788.

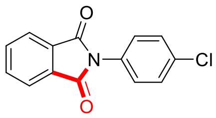


4h[2] **2-(4-fluorophenyl)isoindoline-1, 3-dione**: white solid with 91% isolated yield(hexane/EA = 10/1).

^{1}H NMR (400 MHz, d^{6}-DMSO) δ 7.98-7.95 (m, 2H), 7.92-7.89 (m, 2H), 7.53-7.48 (m, 2H), 7.41-7.35 (m, 2H).

^{13}C NMR (101 MHz, d^{6}-DMSO) δ 167.14, 161.50 (d, J = 245.94 Hz), 134.85, 131.68, 129.78 (d, J = 8.89 Hz), 128.27 (d, J = 2.93 Hz), 123.57, 115.92 (d, J = 22.93 Hz).

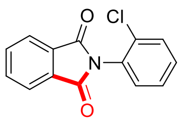
^{19}F NMR (377 MHz, DMSO) δ -113.51.



4i[2] **2-(4-chlorophenyl)isoindoline-1, 3-dione**: white solid with 77% isolated yield(hexane/EA = 10/1).

^{1}H NMR (400 MHz, d^{6}-DMSO) δ 7.99-7.96 (m, 2H), 7.93-7.90 (m, 2H), 7.61 (dt, J = 8.8 Hz, 2.4 Hz, 2H), 7.50 (dt, J = 8.4 Hz, 2.4 Hz, 2H).

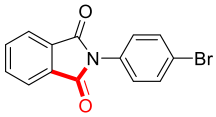
^{13}C NMR (101 MHz, d^{6}-DMSO) δ 166.93, 134.90, 132.66, 131.66, 130.93, 129.24, 129.04, 123.62.



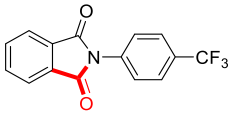
4j[3] **2-(2-chlorophenyl)isoindoline-1, 3-dione**: white solid with 79% isolated yield(hexane/EA = 10/1).

^{1}H NMR (400 MHz, d^{6}-DMSO) δ 8.04-7.99 (m, 2H), 7.97-7.93 (m, 2H), 7.72-7.70 (m, 1H), 7.65-7.63 (m, 1H), 7.59-7.51 (m, 2H).

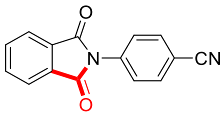
^{13}C NMR (101 MHz, d^{6}-DMSO) δ 166.43, 135.17, 132.32, 131.57, 131.47, 131.25, 129.96, 129.70, 128.27, 123.87.



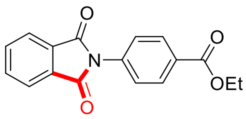
4k[2] **2-(4-bromophenyl)isoindoline-1, 3-dione**: white solid with 75% isolated yield(hexane/EA = 10/1).
^{1}H NMR (400 MHz, *d*^{6}-DMSO) δ 7.98-7.95 (m, 2H), 7.93-7.89 (m, 2H), 7.74 (dt, *J* = 8.8 Hz, 2.4 Hz, 2H), 7.43 (dt, *J* = 8.4 Hz, 2.2 Hz, 2H).
^{13}C NMR (101 MHz, *d*^{6}-DMSO) δ 166.86, 134.89, 131.98, 131.63, 131.36, 129.51, 123.61, 121.13.



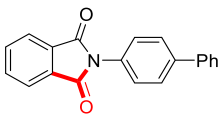
4l[2] **2-(4-(trifluoromethyl)phenyl)isoindoline-1, 3-dione**: white solid with 57% isolated yield(hexane/EA = 10/1).
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.00-7.97 (m, 2H), 7.84-7.82 (m, 2H), 7.78 (d, *J* = 8.4 Hz, 2H), 7.64 (d, *J* = 8.4 Hz, 2H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 166.79, 134.78, 130.17 (q, *J* = 266.54 Hz), 130.95, 129.66, 128.85, 126.46, 126.25 (q, *J* = 3.8 Hz), 124.04.
^{19}F NMR (377 MHz, $CDCl_3$) δ -62.60.



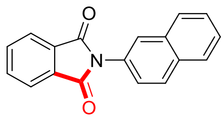
4m[5] **4-(1, 3-dioxoisoindolin-2-yl)benzonitrile**: white solid with 69% isolated yield(hexane/EA = 5/1).
^{1}H NMR (400 MHz, *d*^{6}-DMSO) δ 8.04-8.01 (m, 2H), 8.00-7.98 (m, 2H), 7.95-7.92 (m, 2H), 7.71 (d, *J* = 8.8 Hz, 2H).
^{13}C NMR (101 MHz, *d*^{6}-DMSO) δ 166.58, 136.32, 135.07, 133.11, 131.61, 127.92, 123.79, 118.63, 110.43.



4n[6] **ethyl 4-(1, 3-dioxoisoindolin-2-yl)benzoate**: white solid with 87% isolated yield(hexane/EA = 5/1).
^{1}H NMR (400 MHz, *d*^{6}-DMSO) δ 8.10 (d, *J* = 8.4 Hz, 2H), 8.00-7.96 (m, 2H), 7.93-7.90 (m, 2H), 7.64 (d, *J* = 8.4 Hz, 2H), 4.35 (q, *J* = 7.1 Hz, 2H), 1.34 (t, *J* = 7.2 Hz, 3H).
^{13}C NMR (101 MHz, *d*^{6}-DMSO) δ 166.77, 165.32, 136.27, 135.02, 131.61, 129.81, 129.12, 127.26, 123.71, 61.12, 14.31.



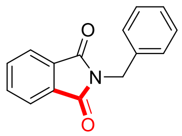
4o[5] **2-([1, 1'-biphenyl]-4-yl)isoindoline-1,3-dione**: white solid with 60% isolated yield(hexane/EA = 10/1).
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.00-7.96 (m, 2H), 7.83-7.80 (m, 2H), 7.73 (d, *J* = 8.4 Hz, 2H), 7.62 (d, *J* = 7.6 Hz, 2H), 7.53 (d, *J* = 8.4 Hz, 2H), 7.47 (t, *J* = 7.4 Hz, 2H), 7.38 (t, *J* = 7.6 Hz, 1H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 167.32, 141.09, 134.45, 133.54, 131.76, 130.74, 128.84, 127.88, 127.61, 127.22, 126.76, 123.80.



4p[7] **2-(naphthalen-2-yl)isoindoline-1,3-dione**: white solid with 70% isolated yield(hexane/EA = 10/1).

^{1}H NMR (400 MHz, d^{6}-DMSO) δ 8.08 (d, J = 8.4 Hz, 1H), 8.04-7.97 (m, 5H), 7.96-7.94 (m, 2H), 7.64-7.60 (m, 3H).

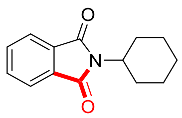
^{13}C NMR (101 MHz, d^{6}-DMSO) δ 167.29, 134.91, 132.78, 132.23, 131.71, 129.55, 128.60, 128.05, 127.82, 126.92, 126.83, 126.18, 125.44, 123.61.



4q[1] **2-benzylisoindoline-1, 3-dione**: white solid with 51% isolated yield(hexane/EA = 10/1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84 (dd, J = 5.2 Hz, 3.2 Hz, 2H), 7.69 (dd, J = 5.2 Hz, 3.2 Hz, 2H), 7.46 (d, J = 7.2 Hz, 2H), 7.36-7.26 (m, 3H), 4.87 (s, 2H).

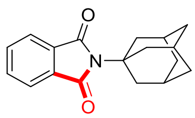
^{13}C NMR (101 MHz, $CDCl_3$) δ 167.84, 136.23, 133.81, 131.93, 128.51, 128.44, 127.66, 123.15, 41.44.



4r[2] **2-cyclohexylisoindoline-1, 3-dione**: white solid with 38% isolated yield(hexane/EA = 10/1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.81-7.78 (m, 2H), 7.71-7.68 (m, 2H), 4.10 (tt, J = 5.2 Hz, 4 Hz, 1H), 2.20 (dq, J = 12.4 Hz, 3.0 Hz, 2H), 1.88-1.84 (m, 2H), 1.74-1.67 (m, 3H), 1.42-1.24 (m, 3H).

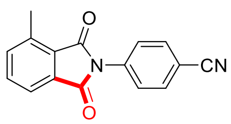
^{13}C NMR (101 MHz, $CDCl_3$) δ 168.34, 133.64, 131.92, 122.88, 50.73, 29.74, 25.90, 25.00.



4s[1] **2-((1S, 3s)-adamantan-1-yl)isoindoline-1,3-dione**: white solid with 59% isolated yield(hexane/EA = 10/1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.78-7.75 (m, 2H), 7.70-7.67 (m, 2H), 2.53 (d, J = 2.4 Hz, 6H), 2.18-2.07 (m, 3H), 1.83-1.71 (m, 6H).

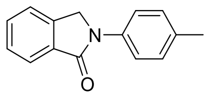
^{13}C NMR (101 MHz, $CDCl_3$) δ 169.79, 133.58, 131.91, 122.46, 60.34, 40.15, 36.14, 29.74.



4t[9] **4-(4-methyl-1, 3-dioxoisoindolin-2-yl)benzonitrile**: white solid with 80% isolated yield(hexane/EA = 10/1).

^{1}H NMR (400 MHz, d^{6}-DMSO) δ 8.02 (d, J = 8.4 Hz, 2H), 7.81-7.76 (m, 2H), 7.71-7.68 (m, 3H), 2.66 (s, 3H).

^{13}C NMR (101 MHz, d^{6}-DMSO) δ 167.16, 166.43, 137.89, 137.08, 136.31, 134.56, 133.05, 131.96, 128.20, 127.96, 121.44, 118.61, 110.38, 17.36.

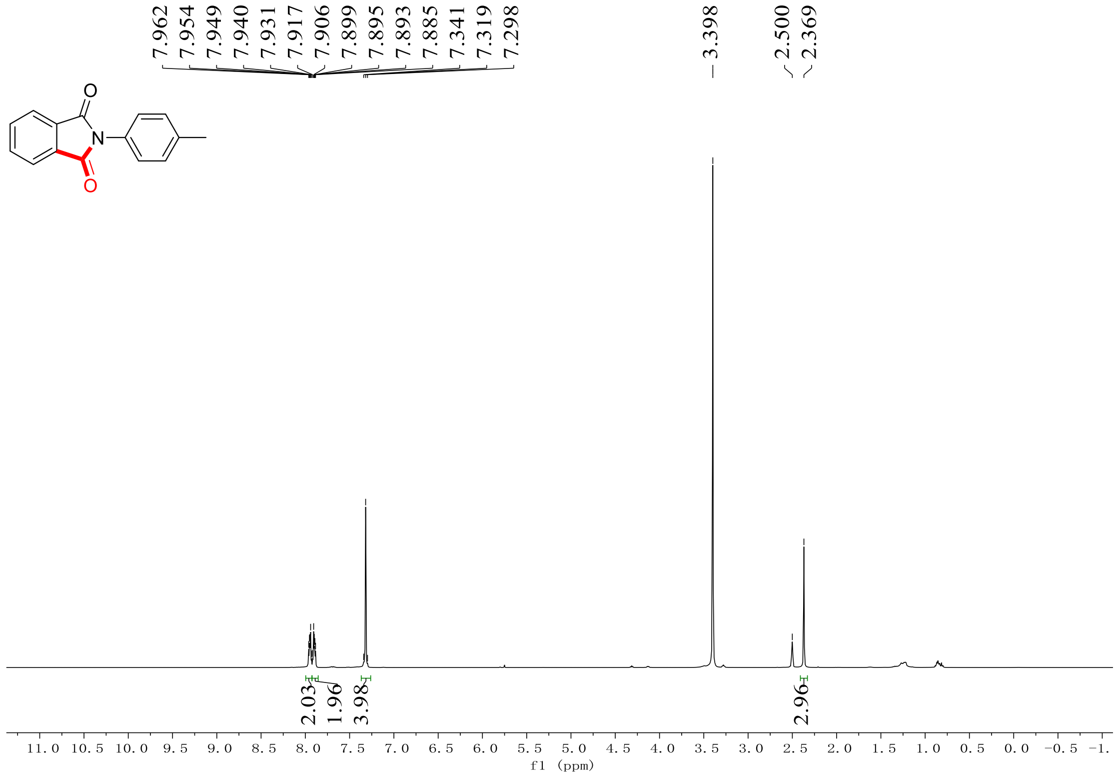


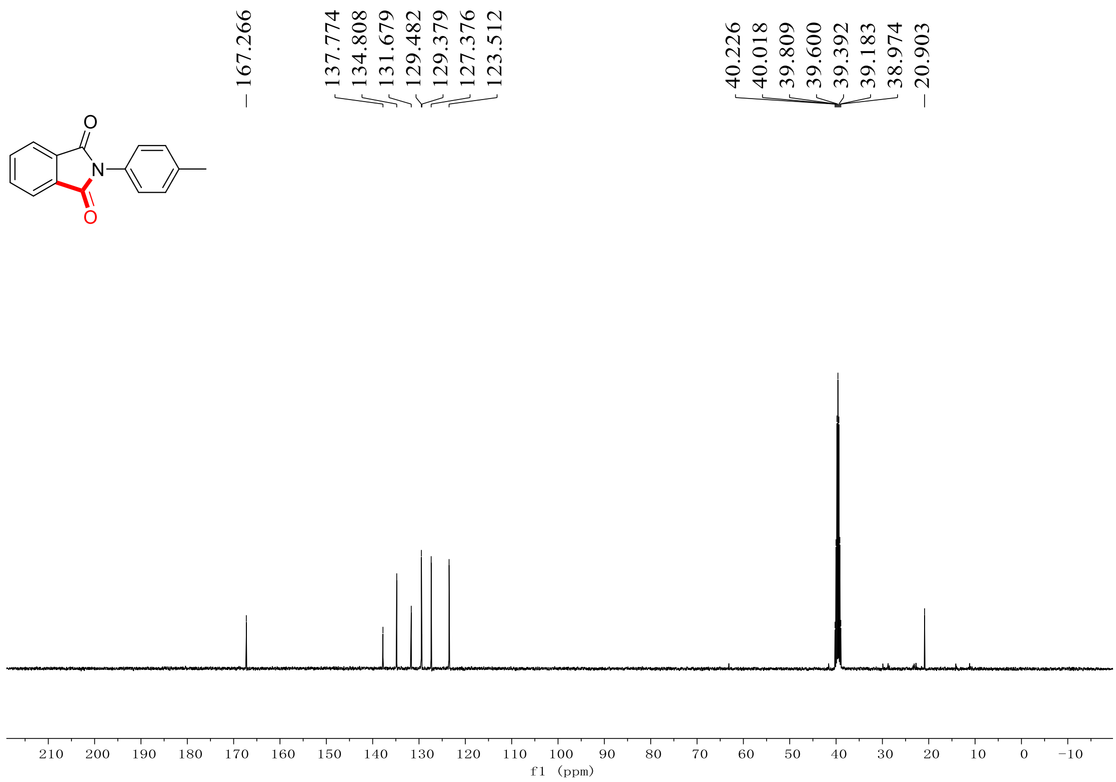
7[10] **2-(*p*-tolyl)isoindolin-1-one**

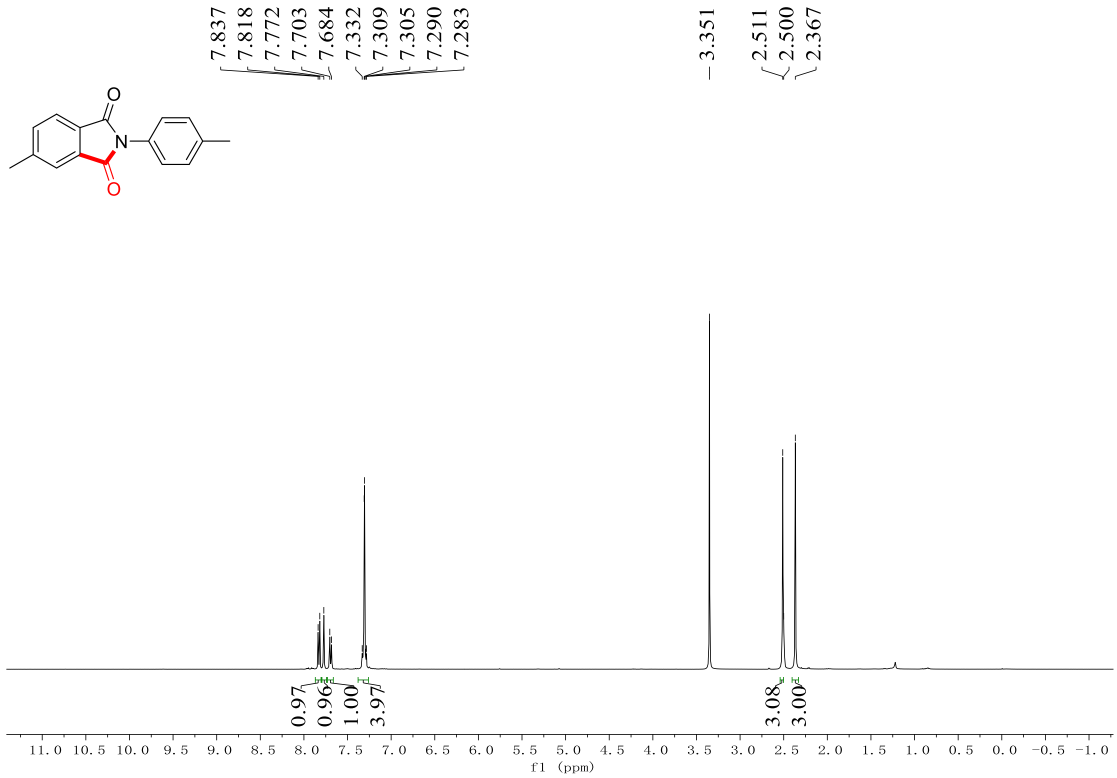
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.95 (d, *J* = 7.2 Hz, 1H), 7.77 (d, *J* = 8.4 Hz, 2H), 7.62 (d, *J* = 7.0 Hz, 1H), 7.53 (d, *J* = 7.2 Hz, 2H), 7.27 (d, *J* = 8.0 Hz, 1H), 4.86 (s, 2H), 2.39 (s, 3H).

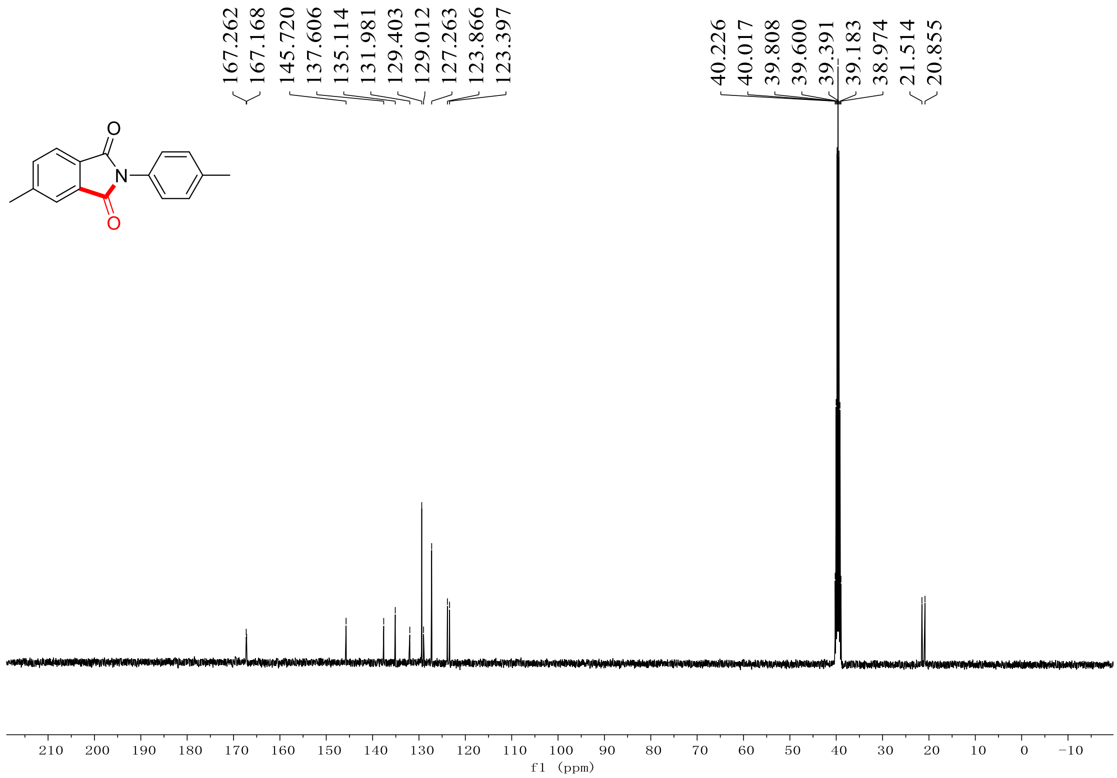
^{13}C NMR (101 MHz, $CDCl_3$) δ 167.36, 140.10, 136.91, 134.18, 133.29, 131.88, 129.65, 128.30, 124.05, 122.55, 119.59, 50.85, 20.85.

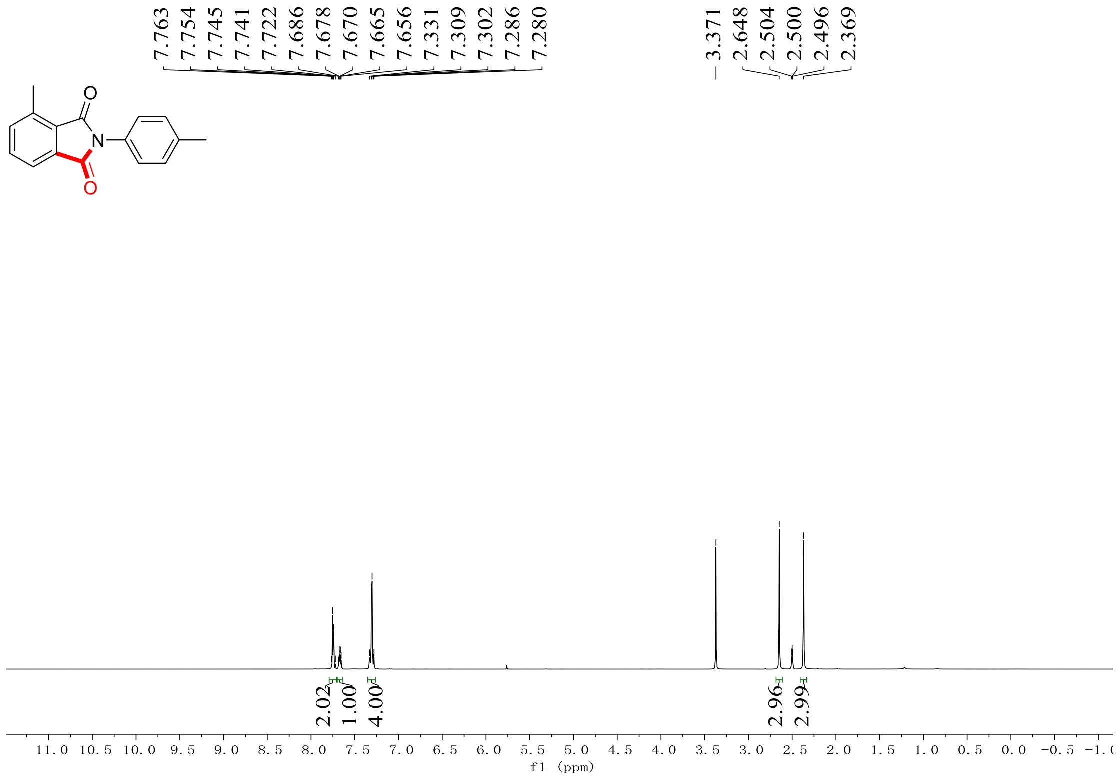
9. NMR Spectra of Substrates and Products

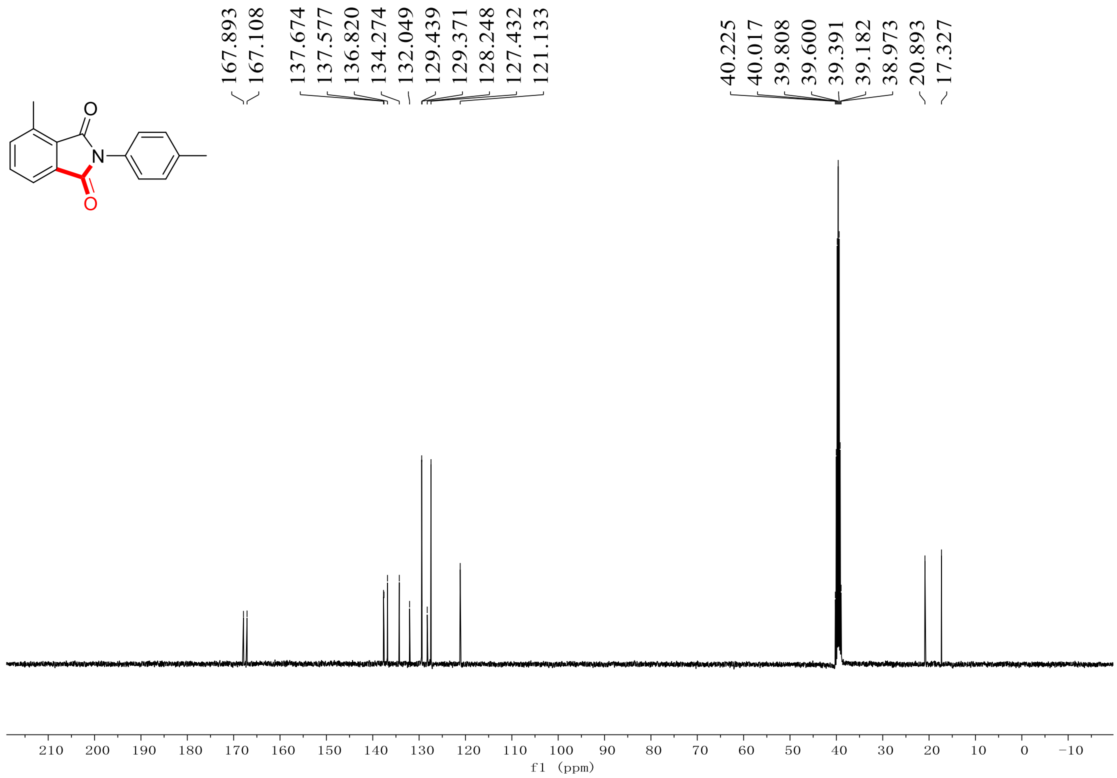


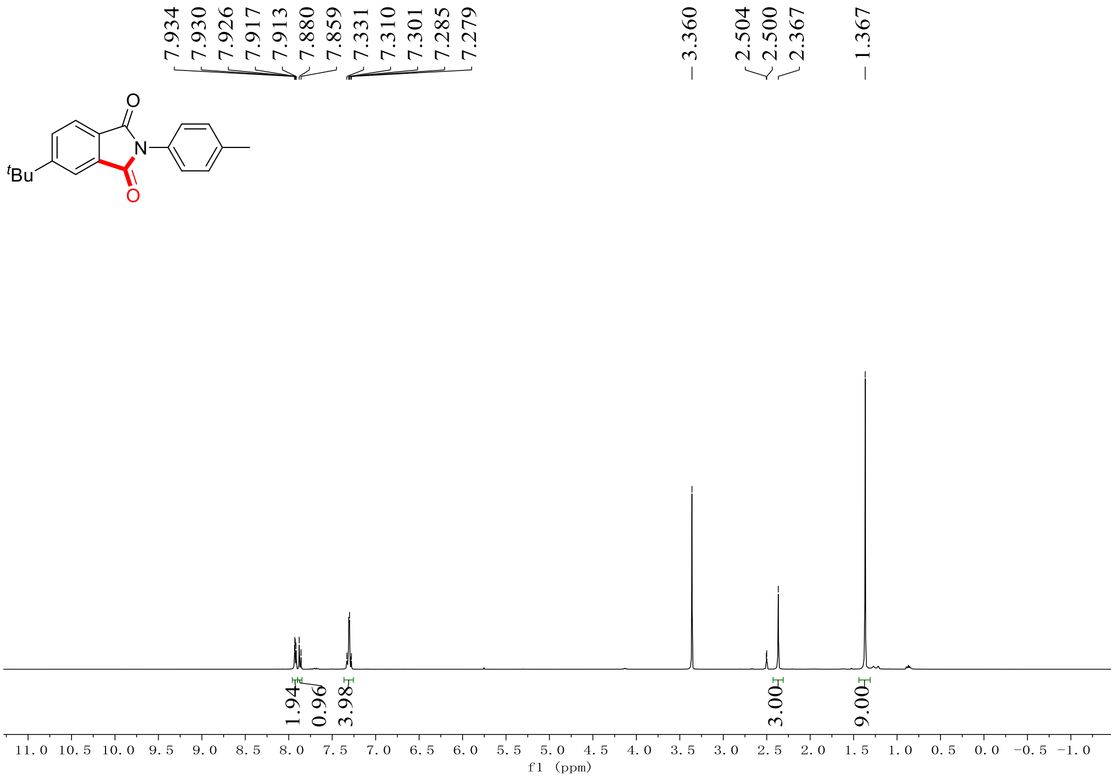


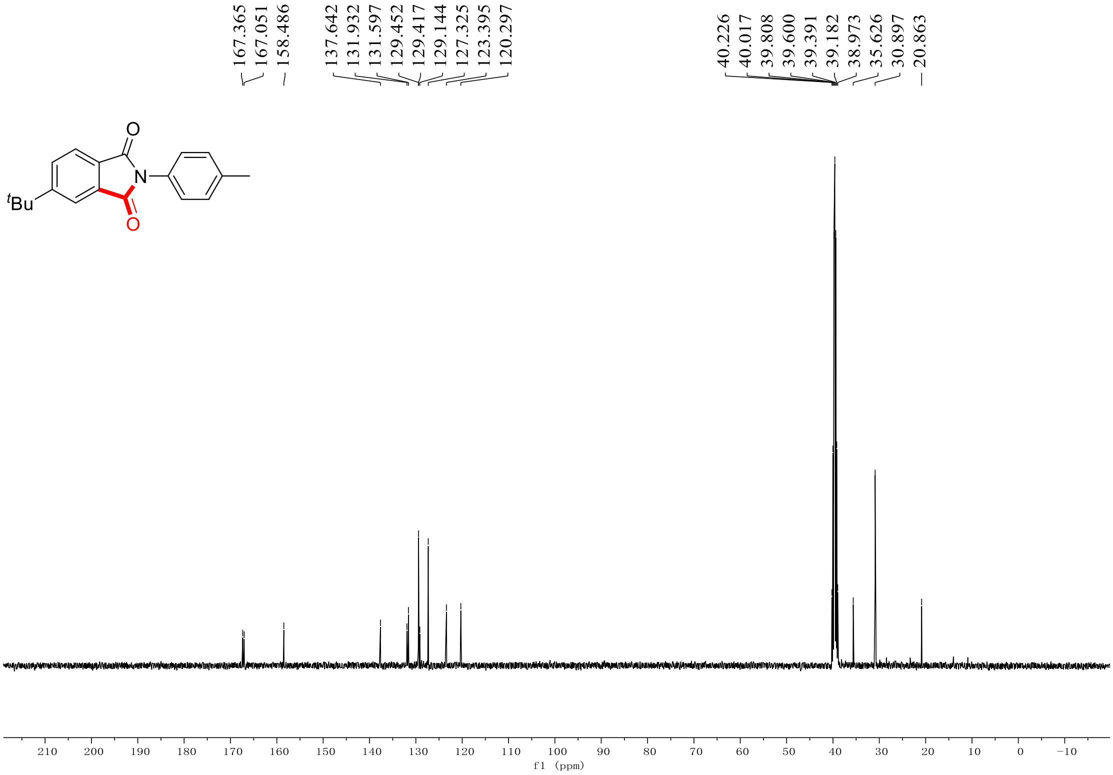

7.837
7.818
7.772
7.703
7.684
7.332
7.309
7.305
7.290
7.283
3.351
2.511
2.500
2.367
O
N
O
0.97
0.96
1.00
3.97
3.08
3.00
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

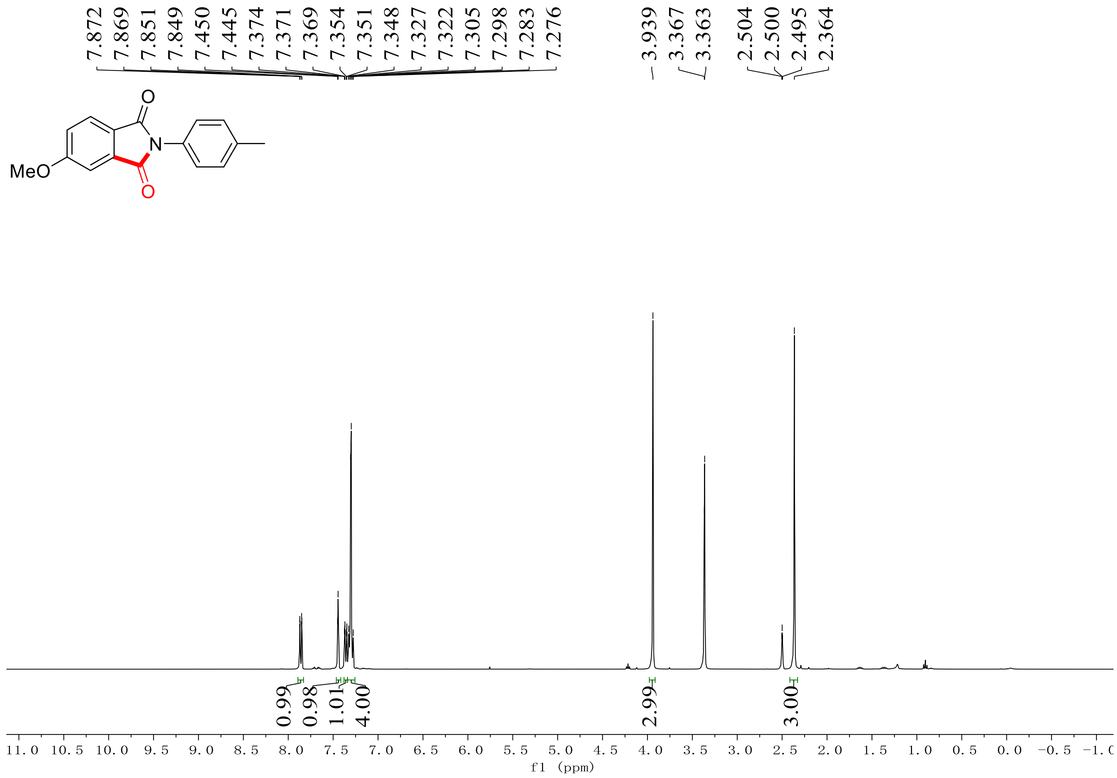

167.262
167.168
145.720
137.606
135.114
131.981
129.403
129.012
127.263
123.866
123.397
40.226
40.017
39.808
39.600
39.391
39.183
38.974
21.514
20.855
O
N
O
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

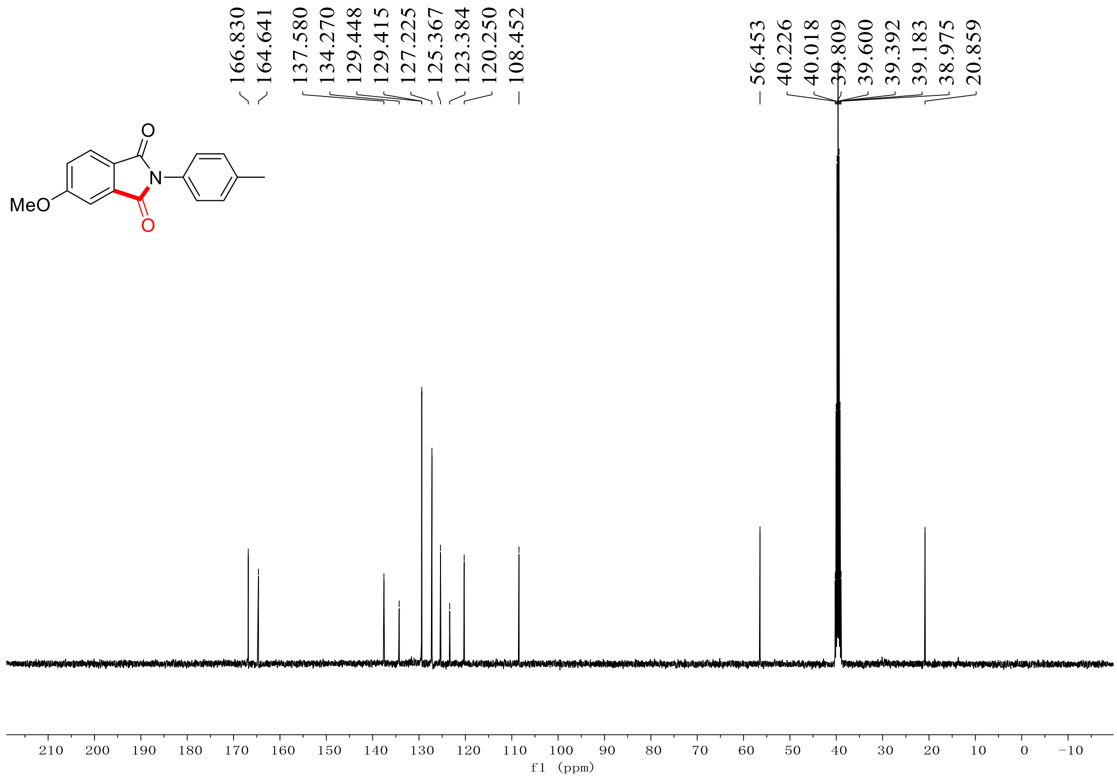

7.763
7.754
7.745
7.741
7.722
7.686
7.678
7.670
7.665
7.656
7.331
7.309
7.302
7.286
7.280
3.371
2.648
2.504
2.500
2.496
2.369
O
N
O
2.02
1.00
4.00
2.96
2.99
f1 (ppm)

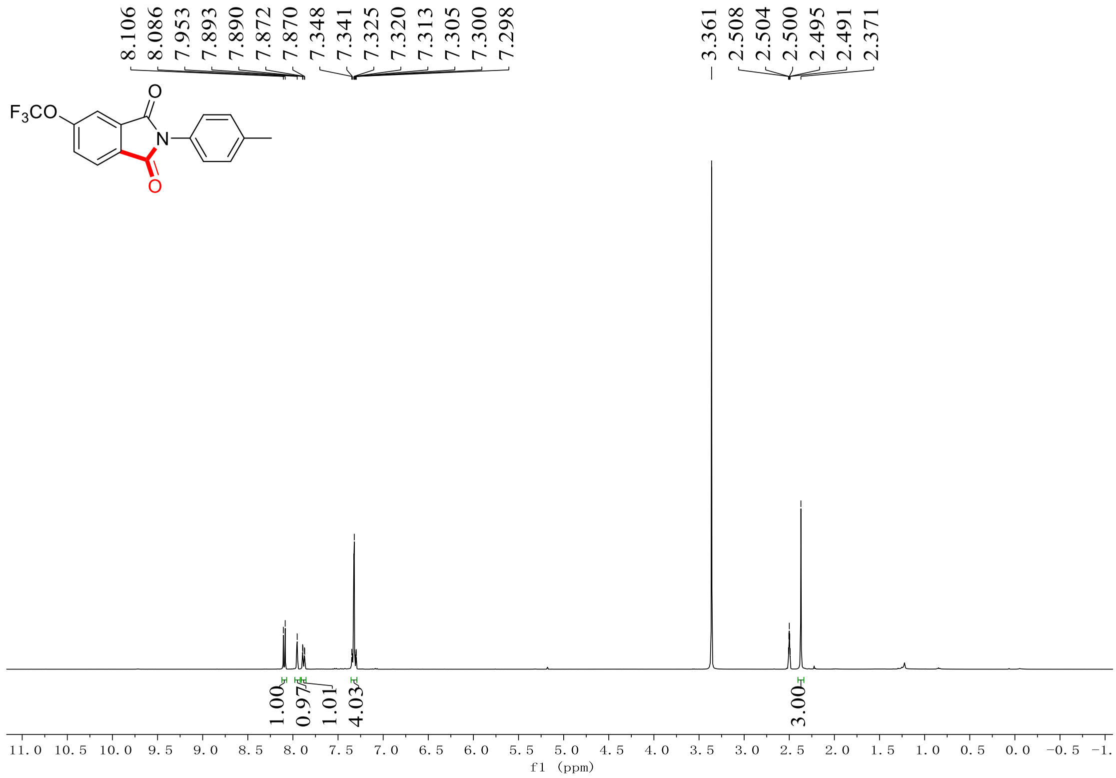

167.893
167.108
137.674
137.577
136.820
134.274
132.049
129.439
129.371
128.248
127.432
121.133
40.225
40.017
39.808
39.600
39.391
39.182
38.973
20.893
17.327
O
N
O
f1 (ppm)

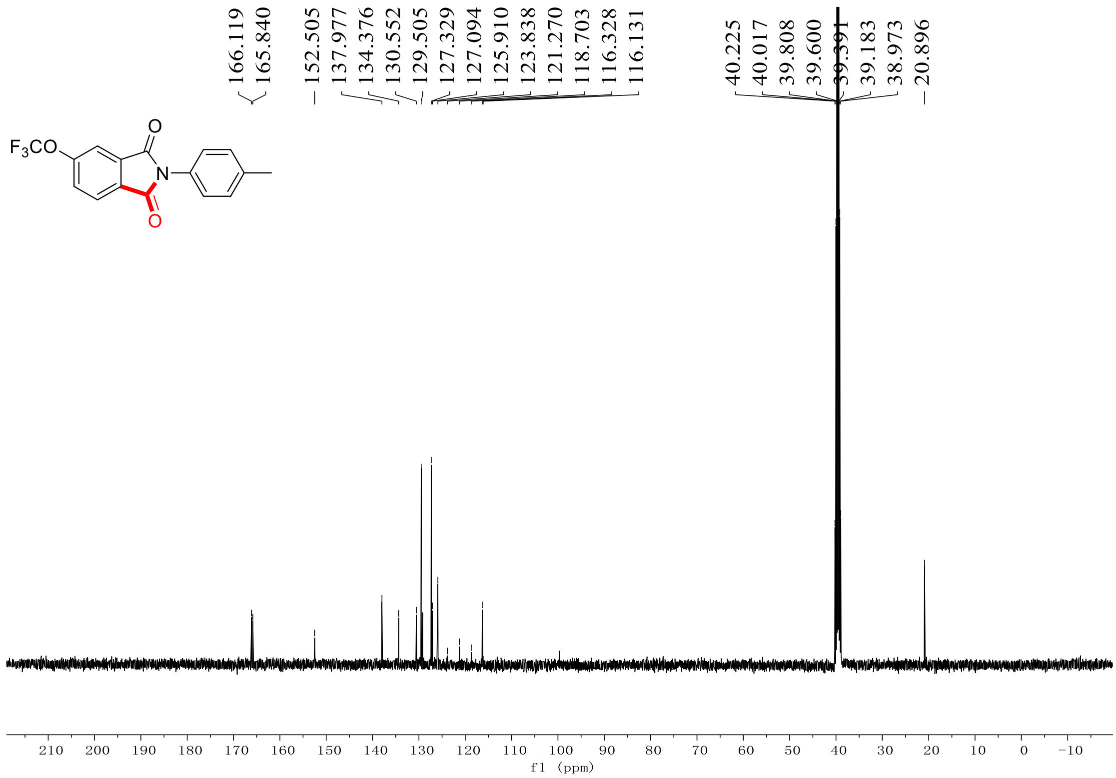

7.934
7.930
7.926
7.917
7.913
7.880
7.859
7.331
7.310
7.301
7.285
7.279
3.360
2.504
2.500
2.367
1.367
O
N
O
^{t}Bu
1.94
0.96
3.98
3.00
9.00
f1 (ppm)

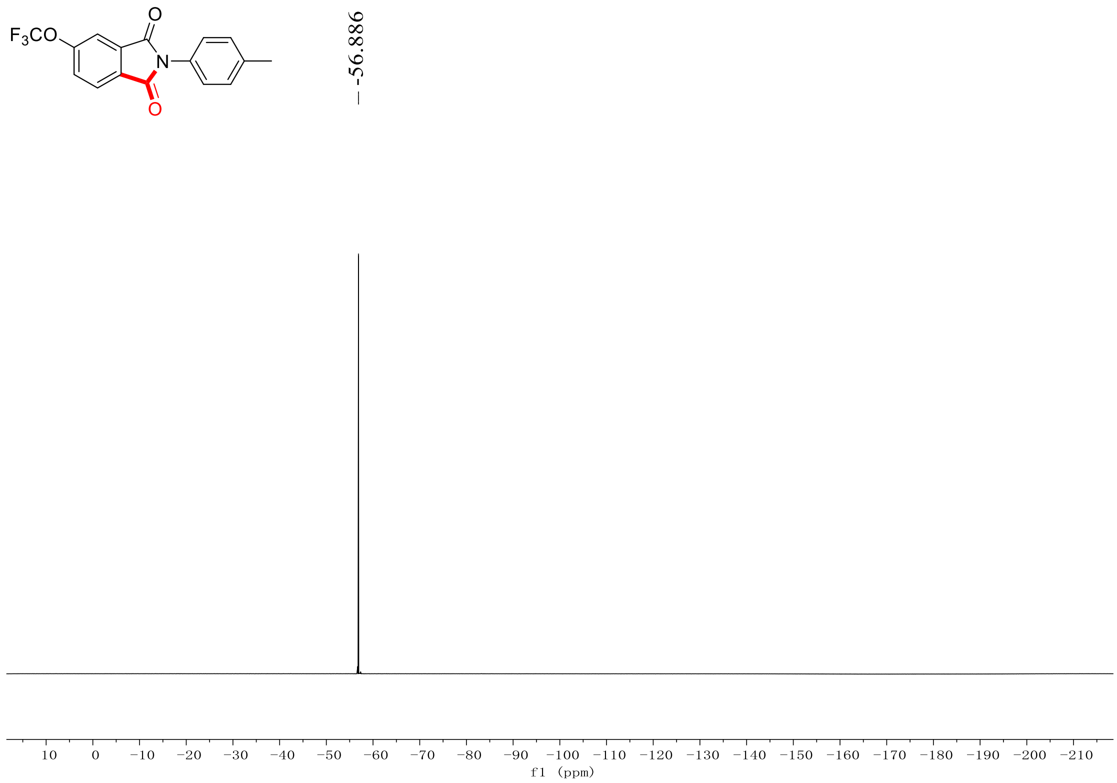

167.365
167.051
158.486
137.642
131.932
131.597
129.452
129.417
129.144
127.325
123.395
120.297
40.226
40.017
39.808
39.600
39.391
39.182
38.973
35.626
30.897
20.863
O
N
O
^{t}Bu
f1 (ppm)

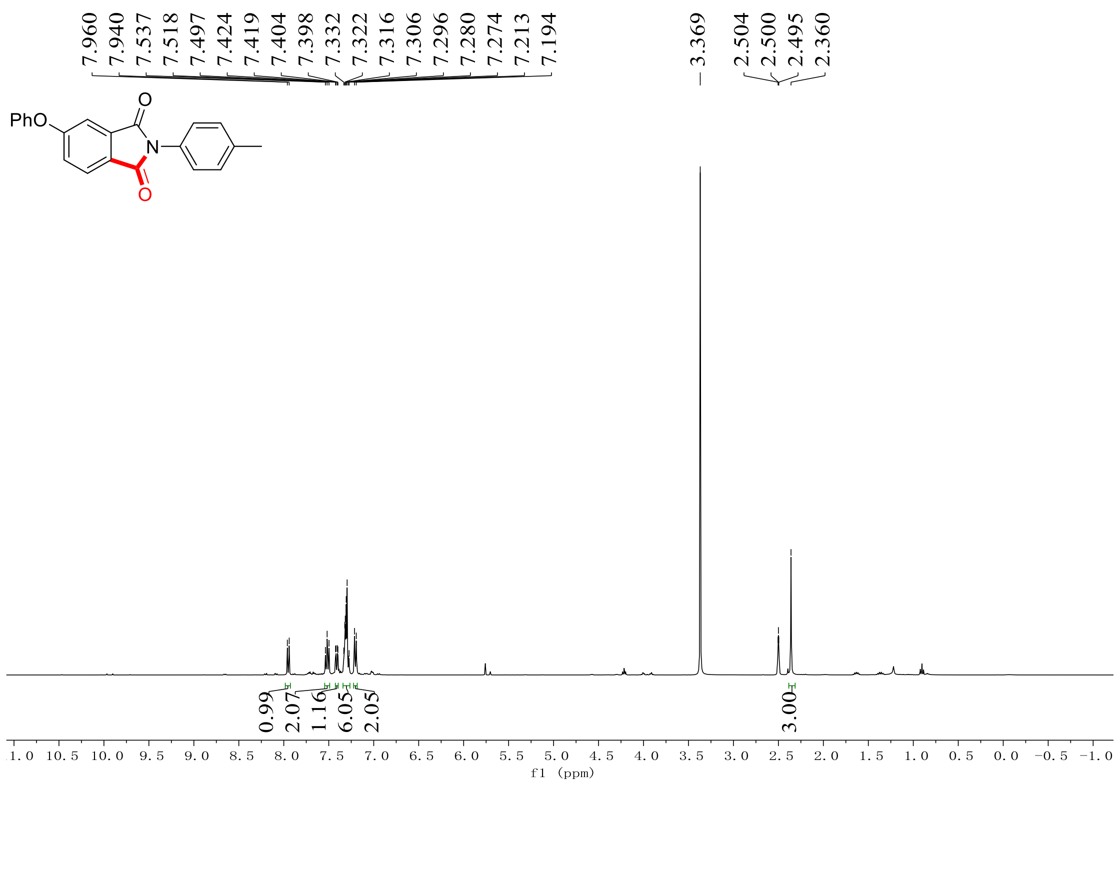

7.872
7.869
7.851
7.849
7.450
7.445
7.374
7.371
7.369
7.354
7.351
7.348
7.327
7.322
7.305
7.298
7.283
7.276
3.939
3.367
3.363
2.504
2.500
2.495
2.364
O
N
MeO
O
0.99
0.98
1.01
4.00
2.99
3.00
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5 −1.0
f1 (ppm)

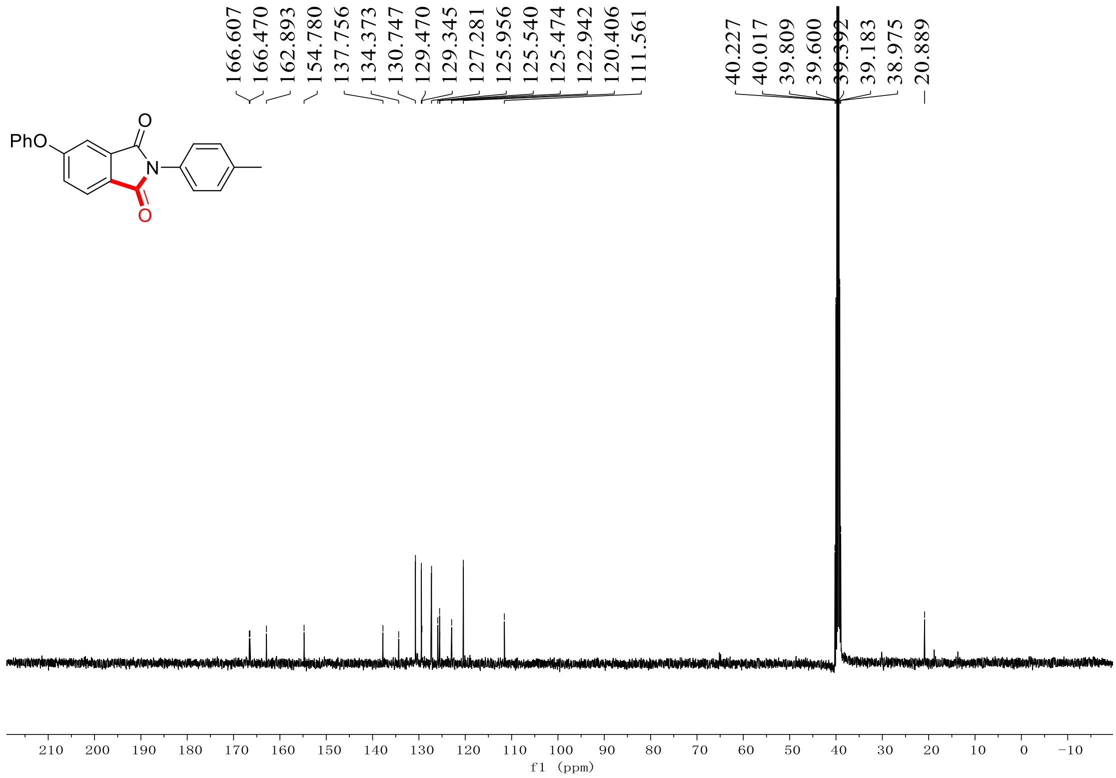

166.830
164.641
137.580
134.270
129.448
129.415
127.225
125.367
123.384
120.250
108.452
56.453
40.226
40.018
39.809
39.600
39.392
39.183
38.975
20.859
O
N
MeO
O
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)

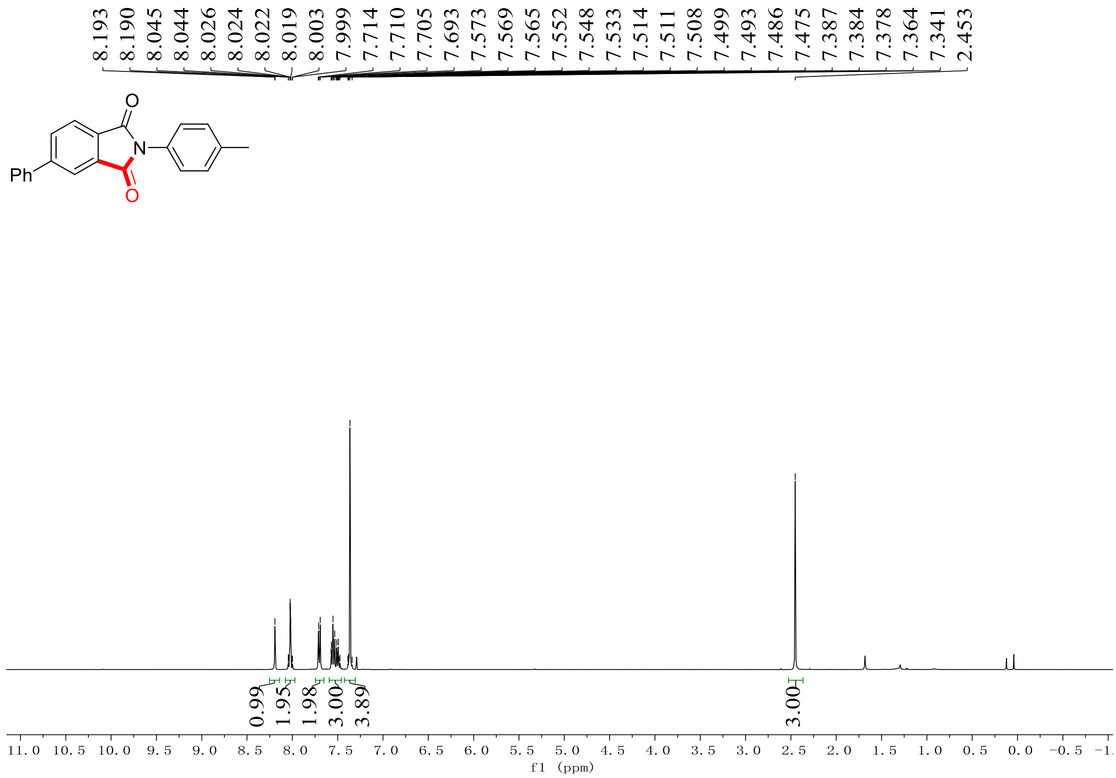


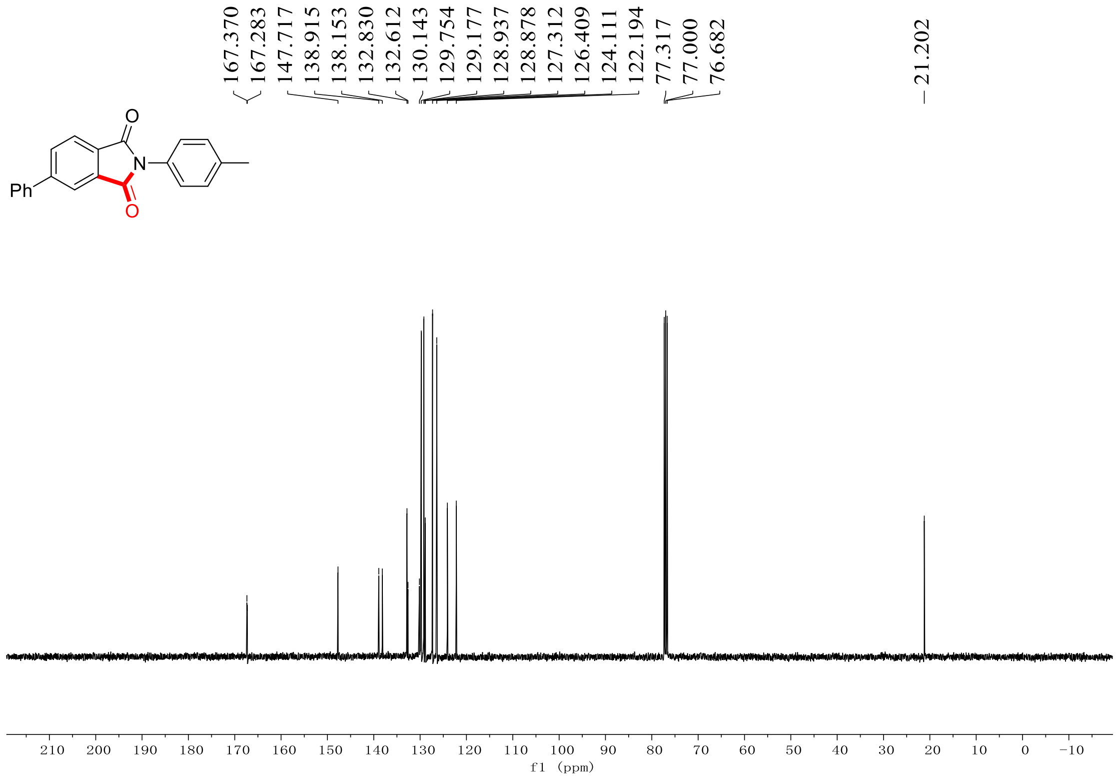


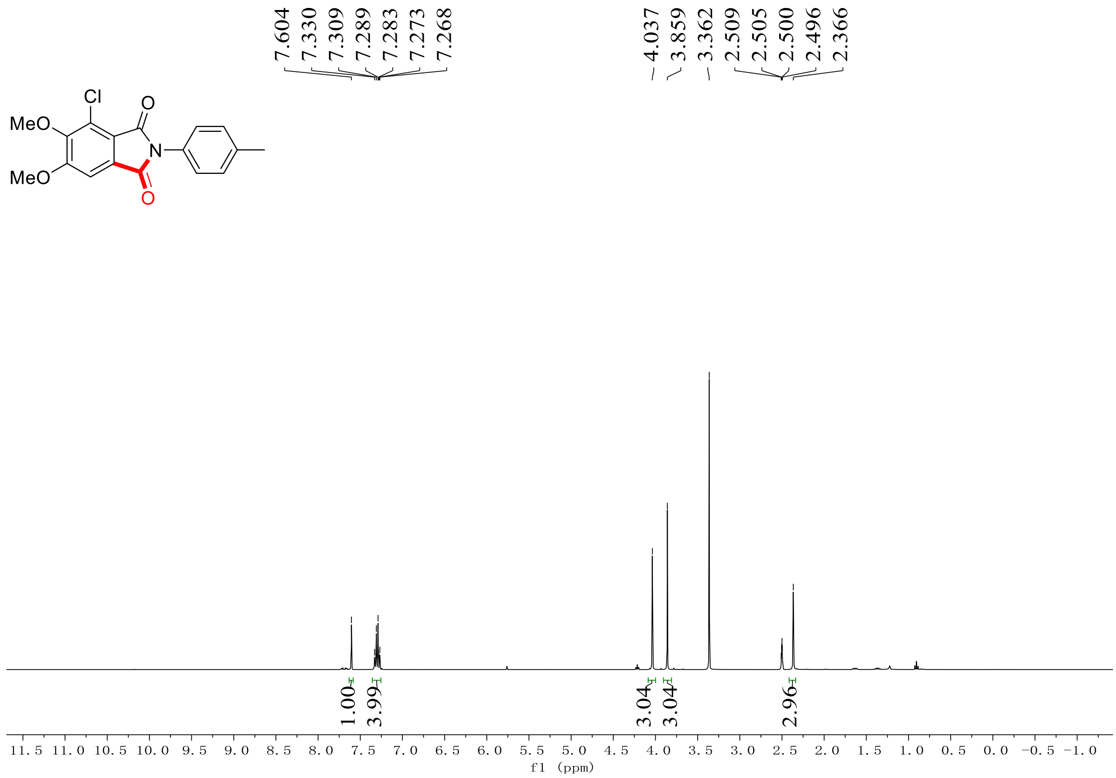


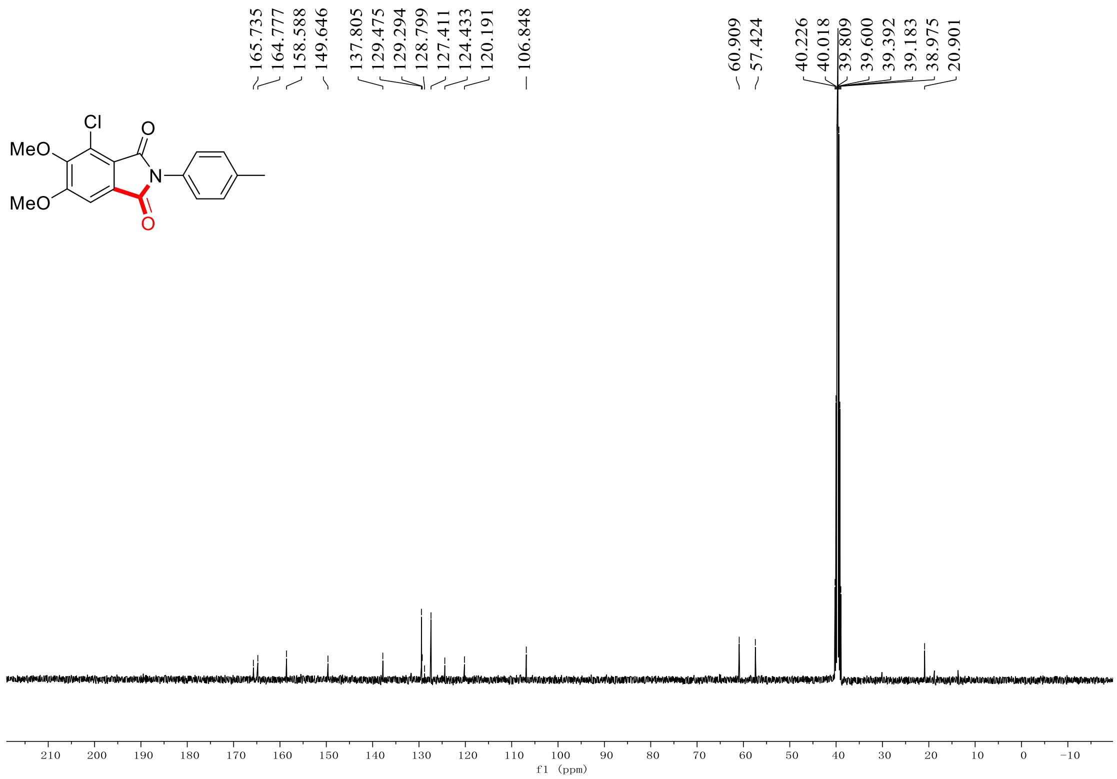


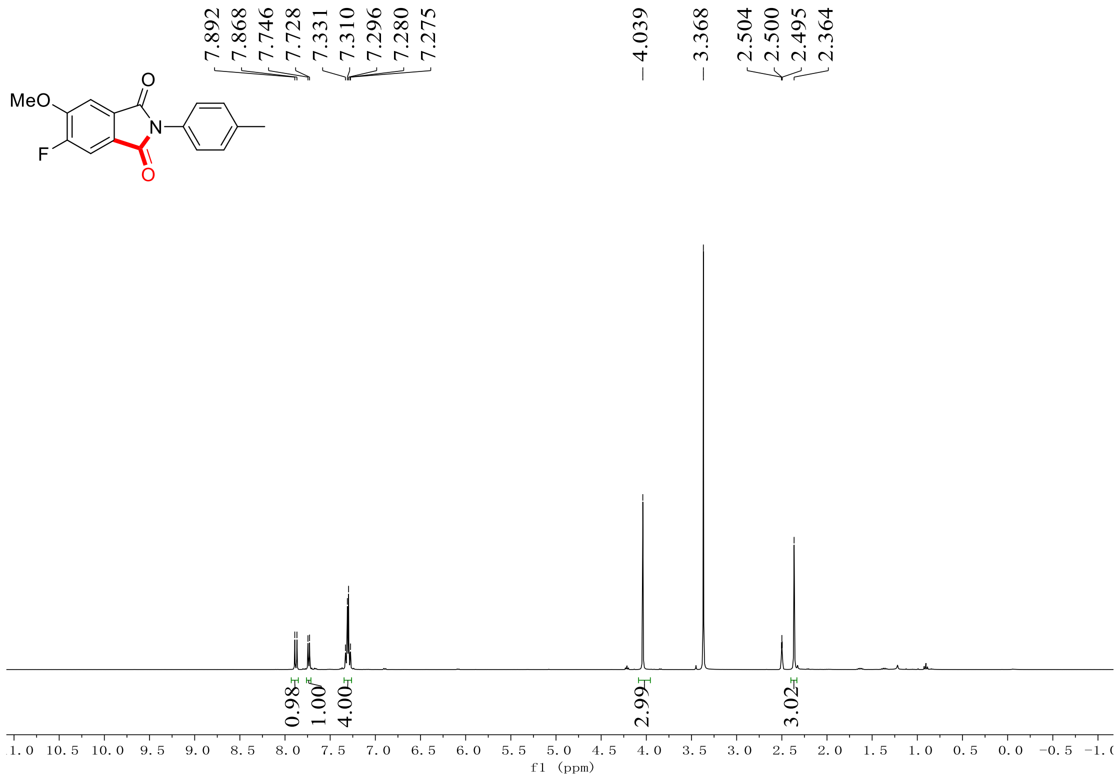

166.607
166.470
162.893
154.780
137.756
134.373
130.747
129.470
129.345
127.281
125.956
125.540
125.474
122.942
120.406
111.561
40.227
40.017
39.809
39.600
39.392
39.183
38.975
20.889
PhO
O
N
O
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

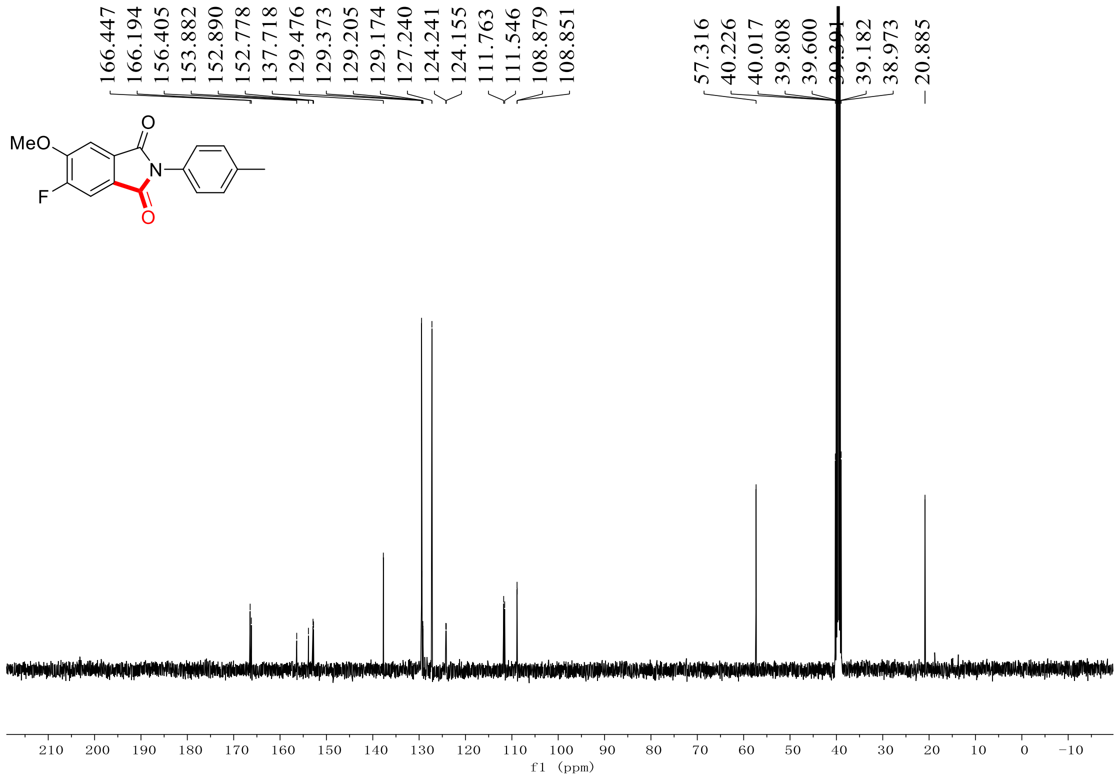

8.193
8.190
8.045
8.044
8.026
8.024
8.022
8.019
8.003
7.999
7.714
7.710
7.705
7.693
7.573
7.569
7.565
7.552
7.548
7.533
7.514
7.511
7.508
7.499
7.493
7.486
7.475
7.387
7.384
7.378
7.364
7.341
2.453
Ph
O
N
O
0.99
1.95
1.98
3.00
3.89
3.00
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.
f1 (ppm)

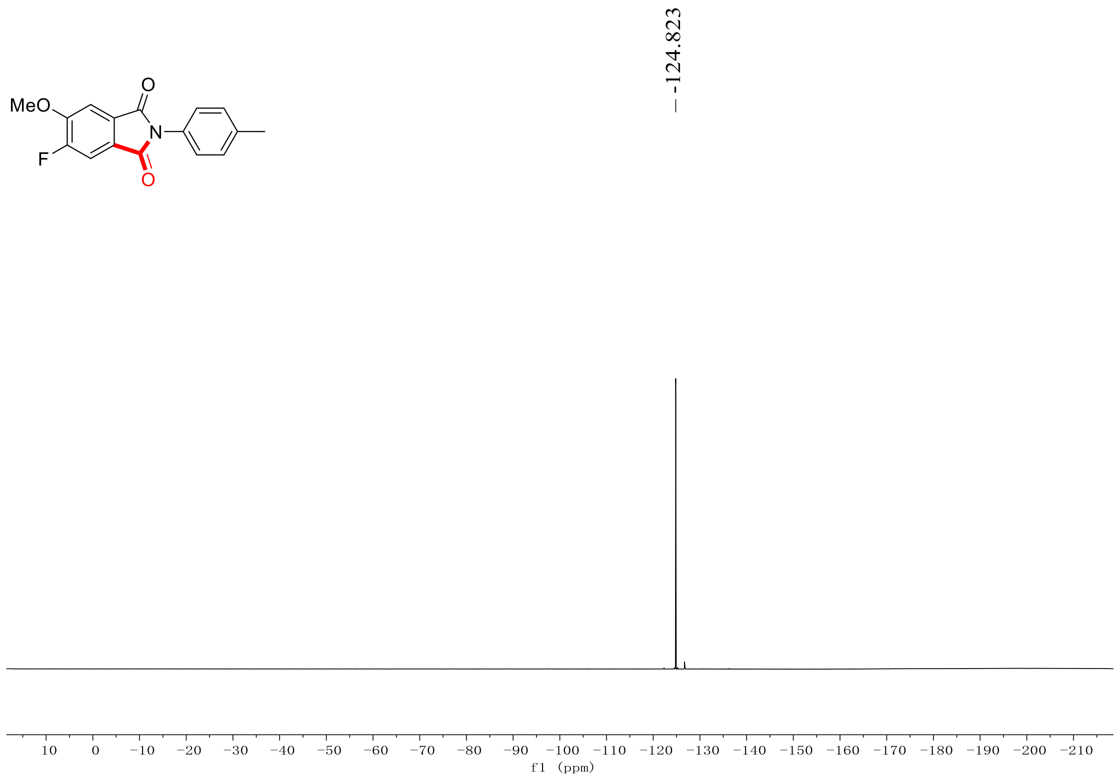

167.370
167.283
147.717
138.915
138.153
132.830
132.612
130.143
129.754
129.177
128.937
128.878
127.312
126.409
124.111
122.194
77.317
77.000
76.682
21.202
Ph
N
O
O
f1 (ppm)

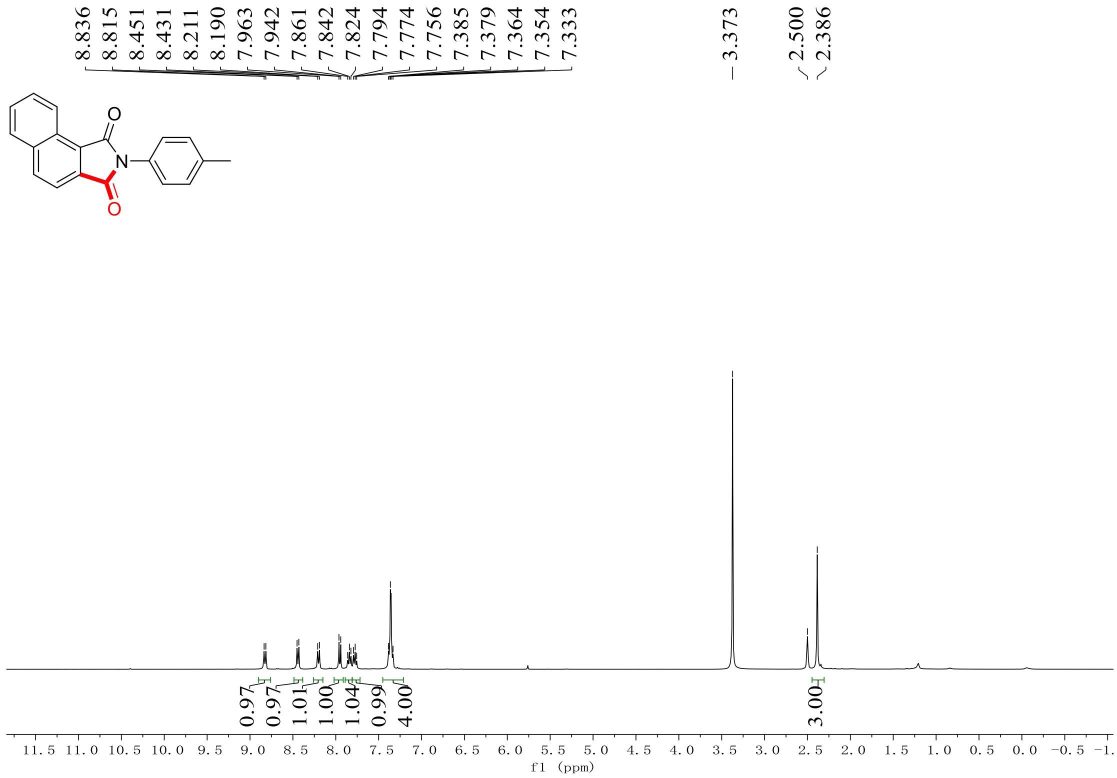

7.604
7.330
7.309
7.289
7.283
7.273
7.268
4.037
3.859
3.362
2.509
2.505
2.500
2.496
2.366
Cl
MeO
MeO
N
O
O
1.00
3.99
3.04
3.04
2.96
f1 (ppm)

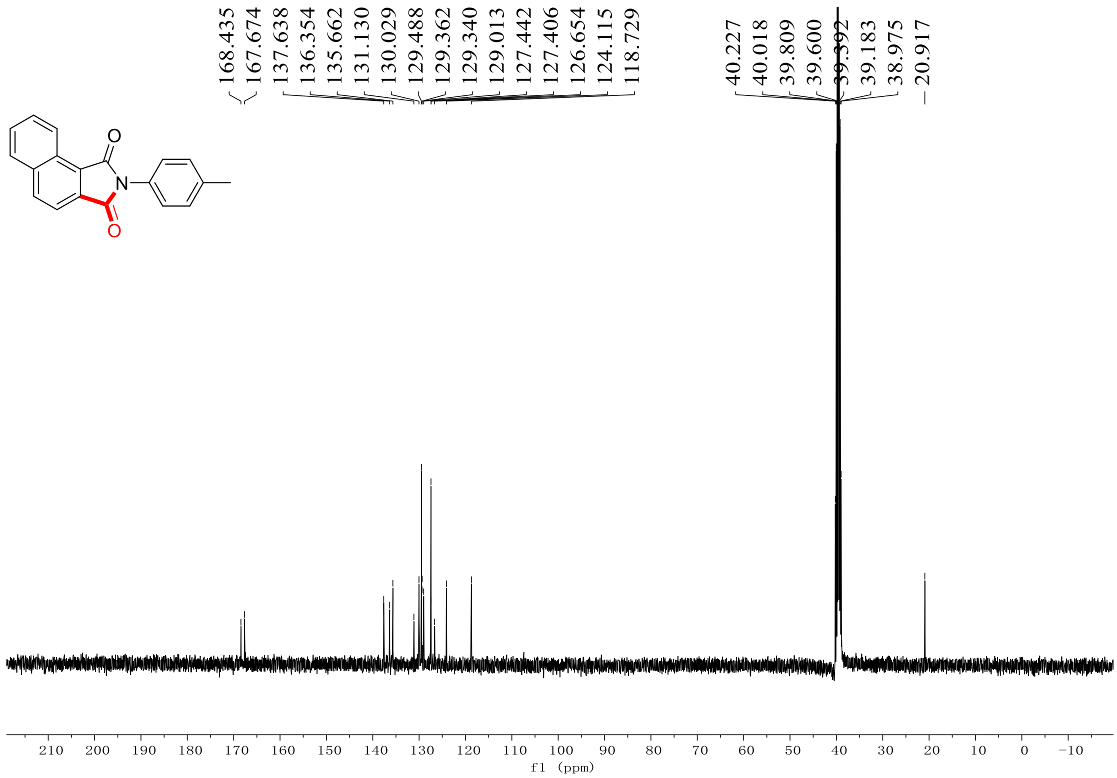

Cl
MeO
MeO
O
O
N
165.735
164.777
158.588
149.646
137.805
129.475
129.294
128.799
127.411
124.433
120.191
106.848
60.909
57.424
40.226
40.018
39.809
39.600
39.392
39.183
38.975
20.901
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

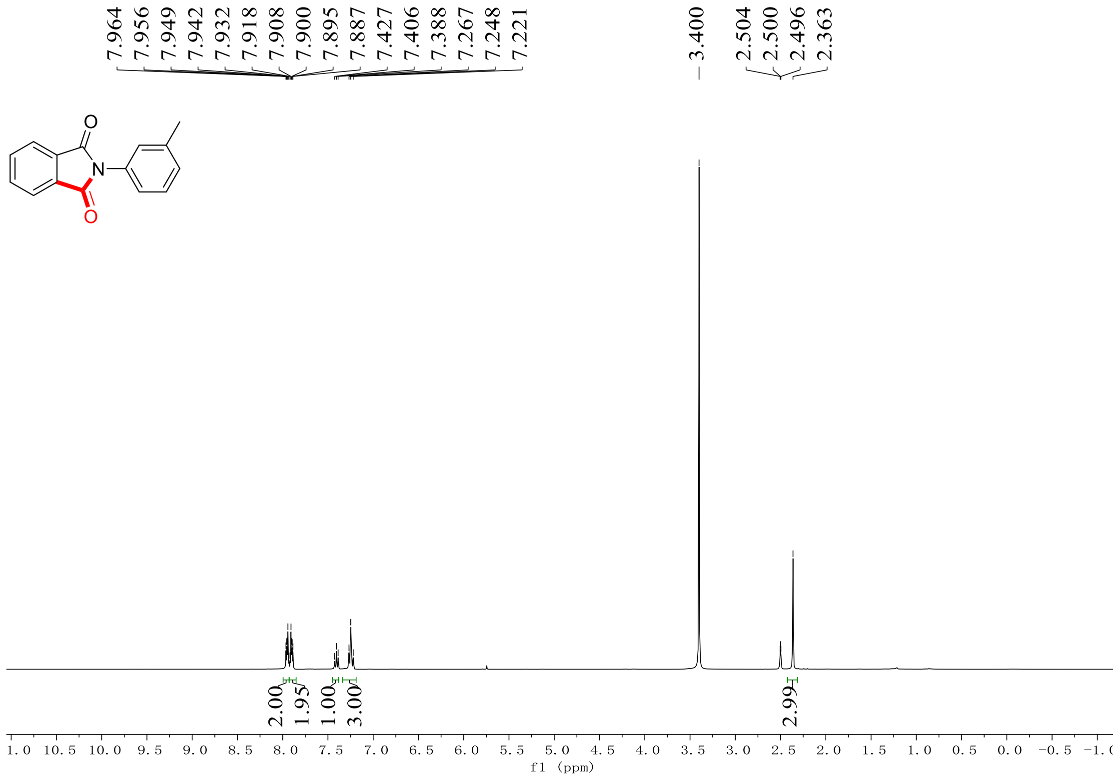

MeO
F
O
O
N
7.892
7.868
7.746
7.728
7.331
7.310
7.296
7.280
7.275
4.039
3.368
2.504
2.500
2.495
2.364
0.98
1.00
4.00
2.99
3.02
f1 (ppm)

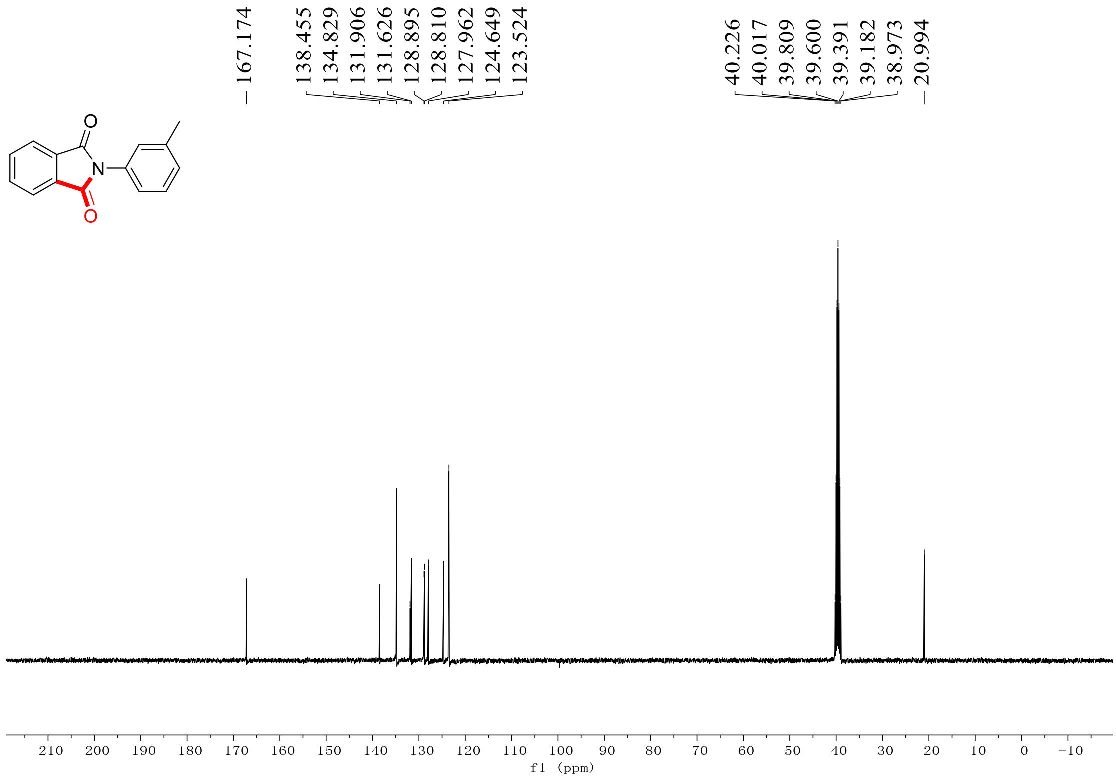

166.447
166.194
156.405
153.882
152.890
152.778
137.718
129.476
129.373
129.205
129.174
127.240
124.241
124.155
111.763
111.546
108.879
108.851
57.316
40.226
40.017
39.808
39.600
39.391
39.182
38.973
20.885
MeO
F
O
N
O
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)

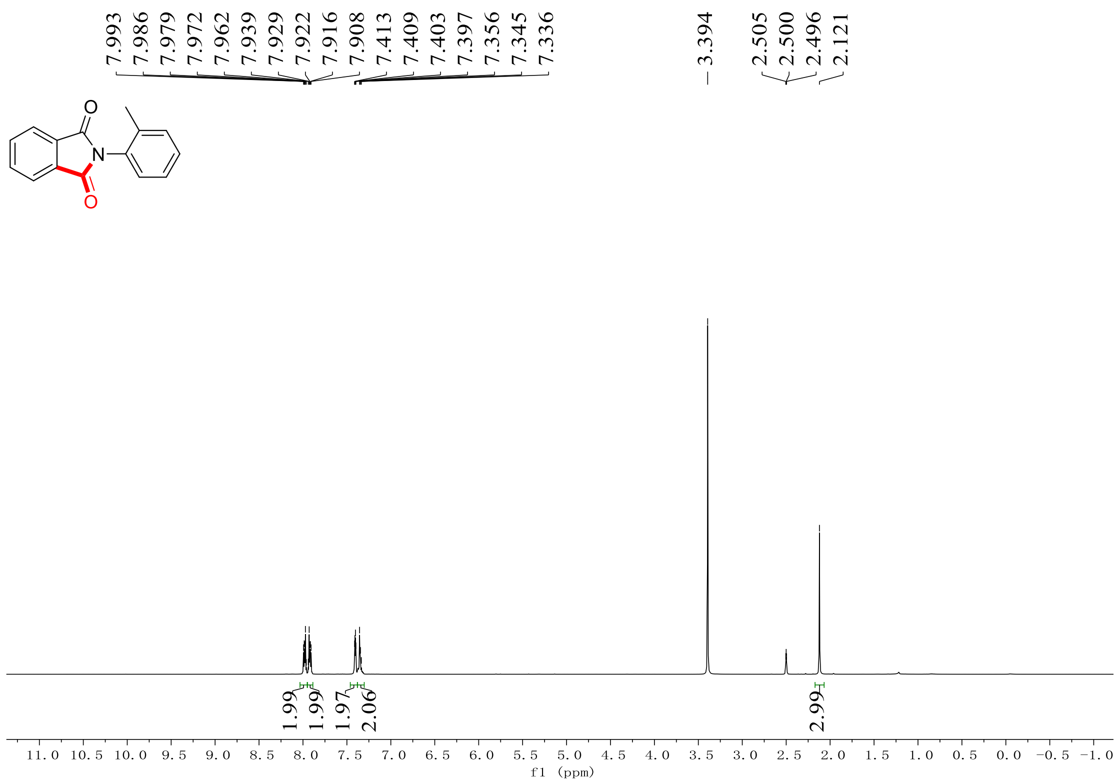

-124.823
MeO
F
O
N
O
10 0 −10 −20 −30 −40 −50 −60 −70 −80 −90 −100 −110 −120 −130 −140 −150 −160 −170 −180 −190 −200 −210
f1 (ppm)

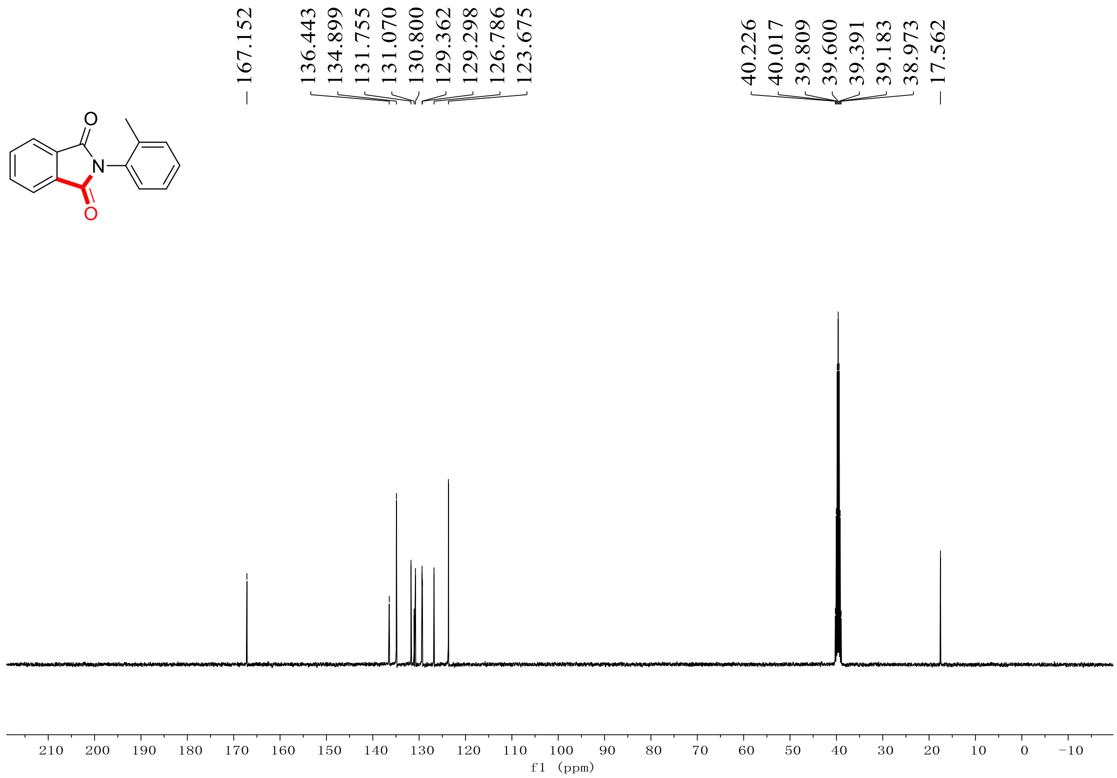

8.836
8.815
8.451
8.431
8.211
8.190
7.963
7.942
7.861
7.842
7.824
7.794
7.774
7.756
7.385
7.379
7.364
7.354
7.333
3.373
2.500
2.386
O
N
O
0.97
0.97
1.01
1.00
1.04
0.99
4.00
3.00
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5 −1.
f1 (ppm)

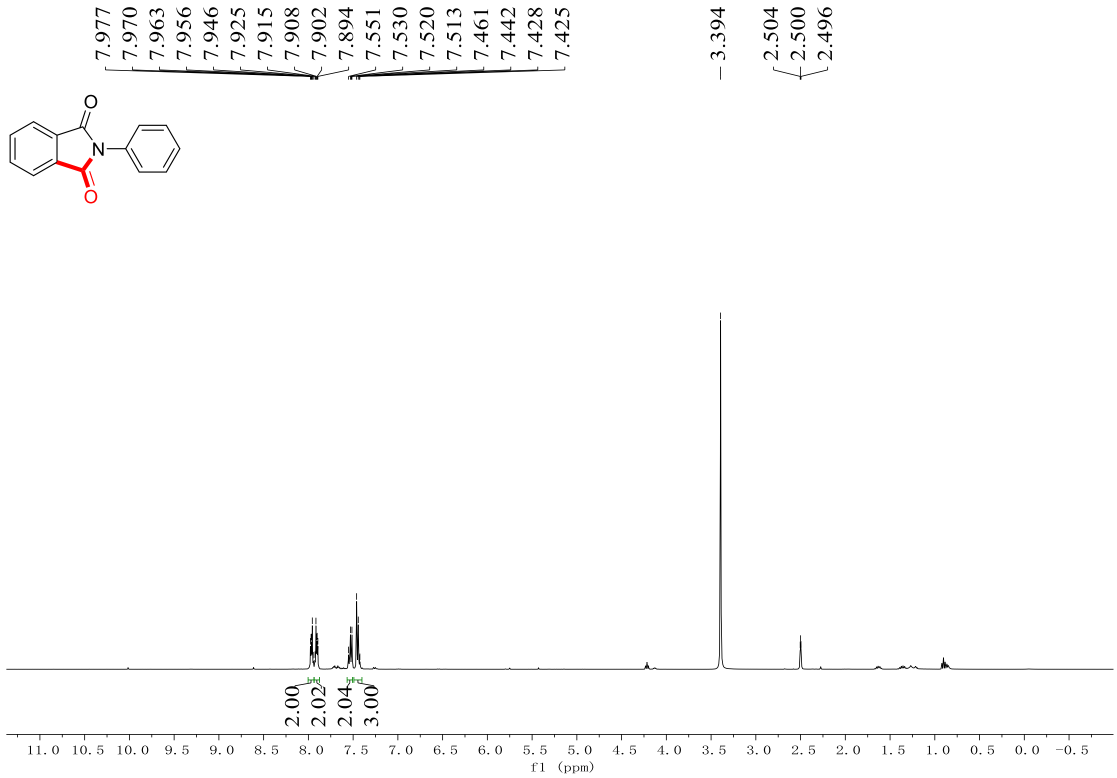

168.435
167.674
137.638
136.354
135.662
131.130
130.029
129.488
129.362
129.340
129.013
127.442
127.406
126.654
124.115
118.729
40.227
40.018
39.809
39.600
39.392
39.183
38.975
20.917
O
N
O
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)

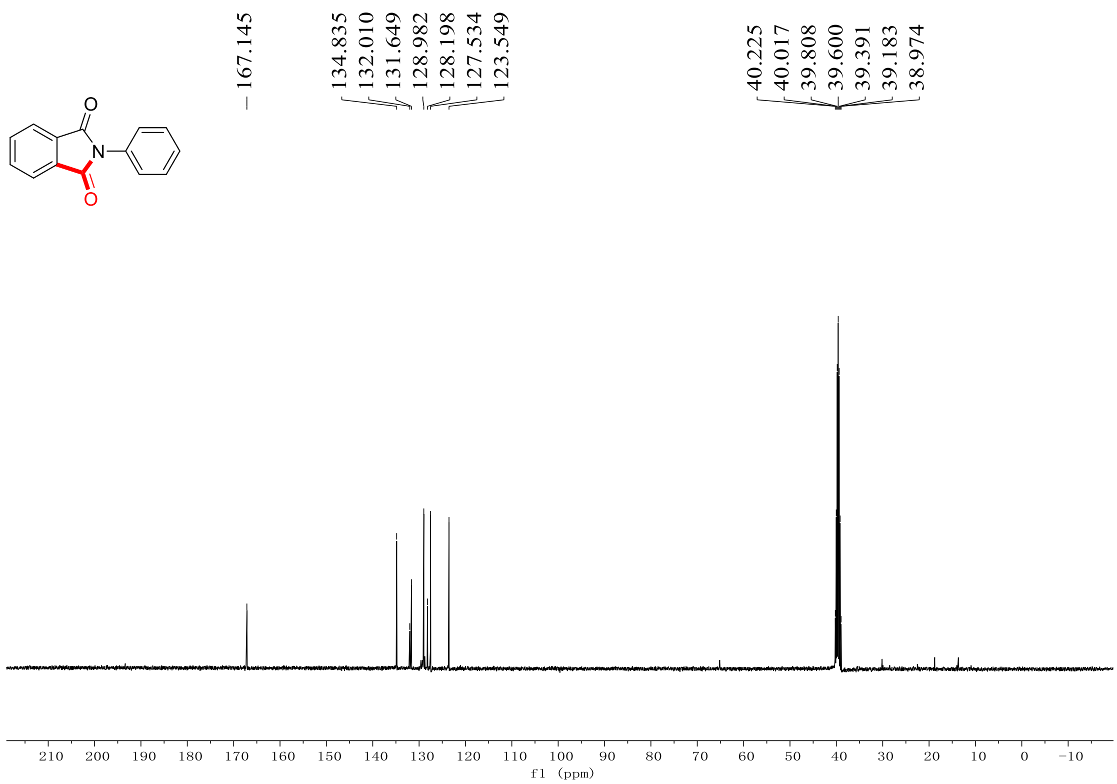

7.964
7.956
7.949
7.942
7.932
7.918
7.908
7.900
7.895
7.887
7.427
7.406
7.388
7.267
7.248
7.221
3.400
2.504
2.500
2.496
2.363
O
N
O
2.00
1.95
1.00
3.00
2.99
f1 (ppm)

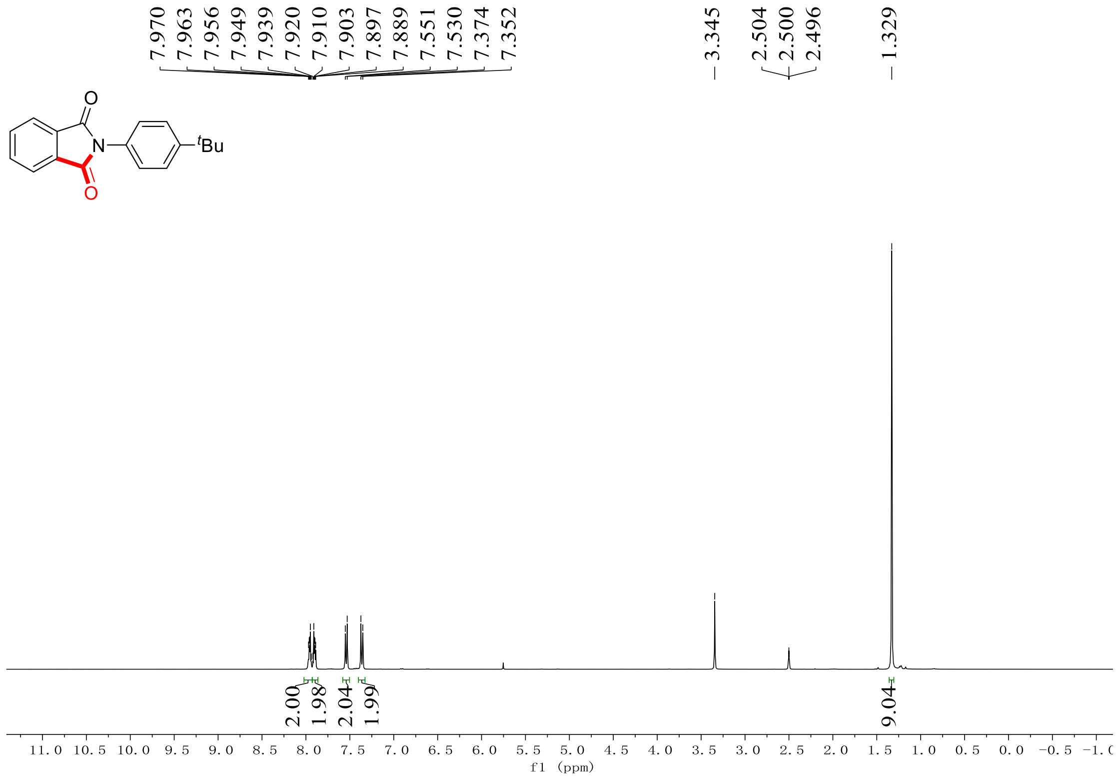

167.174
138.455
134.829
131.906
131.626
128.895
128.810
127.962
124.649
123.524
40.226
40.017
39.809
39.600
39.391
39.182
38.973
20.994
O
N
O
f1 (ppm)


7.993
7.986
7.979
7.972
7.962
7.939
7.929
7.922
7.916
7.908
7.413
7.409
7.403
7.397
7.356
7.345
7.336
3.394
2.505
2.500
2.496
2.121
O
N
O
1.99
1.99
1.97
2.06
2.99
11.0 10.5 10.0 9.5 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 −0.5 −1.0
f1 (ppm)

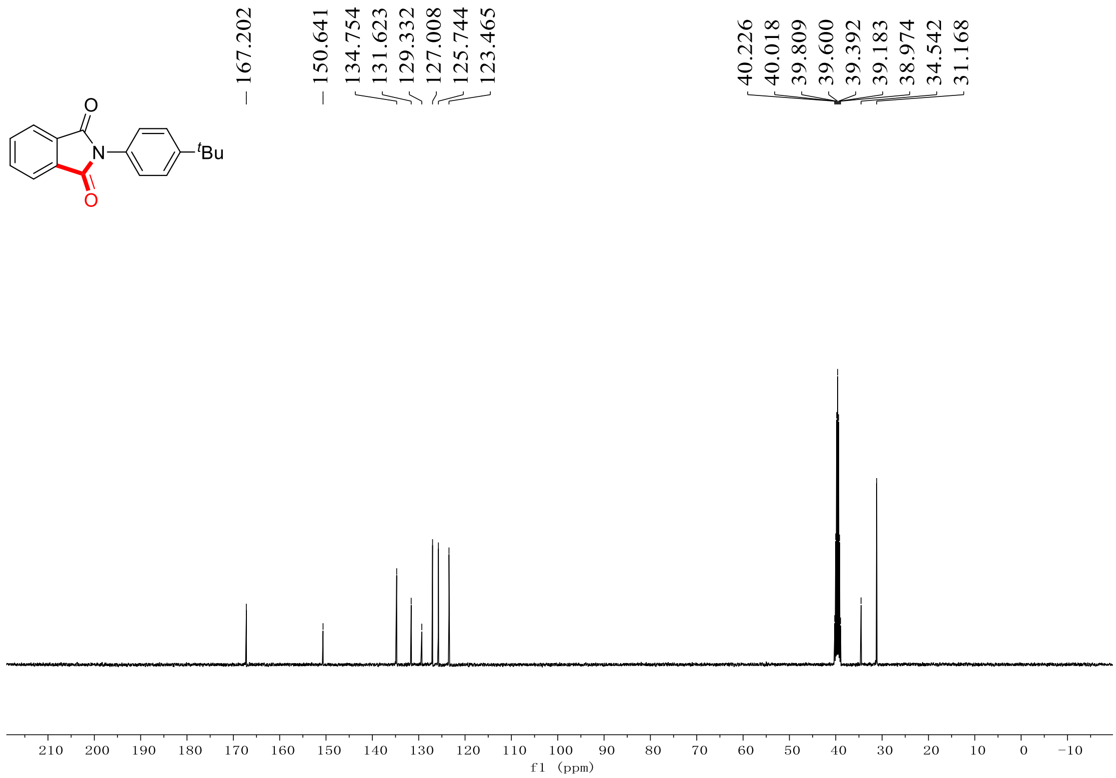

167.152
136.443
134.899
131.755
131.070
130.800
129.362
129.298
126.786
123.675
40.226
40.017
39.809
39.600
39.391
39.183
38.973
17.562
O
N
O
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)


7.977
7.970
7.963
7.956
7.946
7.925
7.915
7.908
7.902
7.894
7.551
7.530
7.520
7.513
7.461
7.442
7.428
7.425
3.394
2.504
2.500
2.496
O
N
O
2.00
2.02
2.04
3.00
11.0 10.5 10.0 9.5 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 -0.5
f1 (ppm)

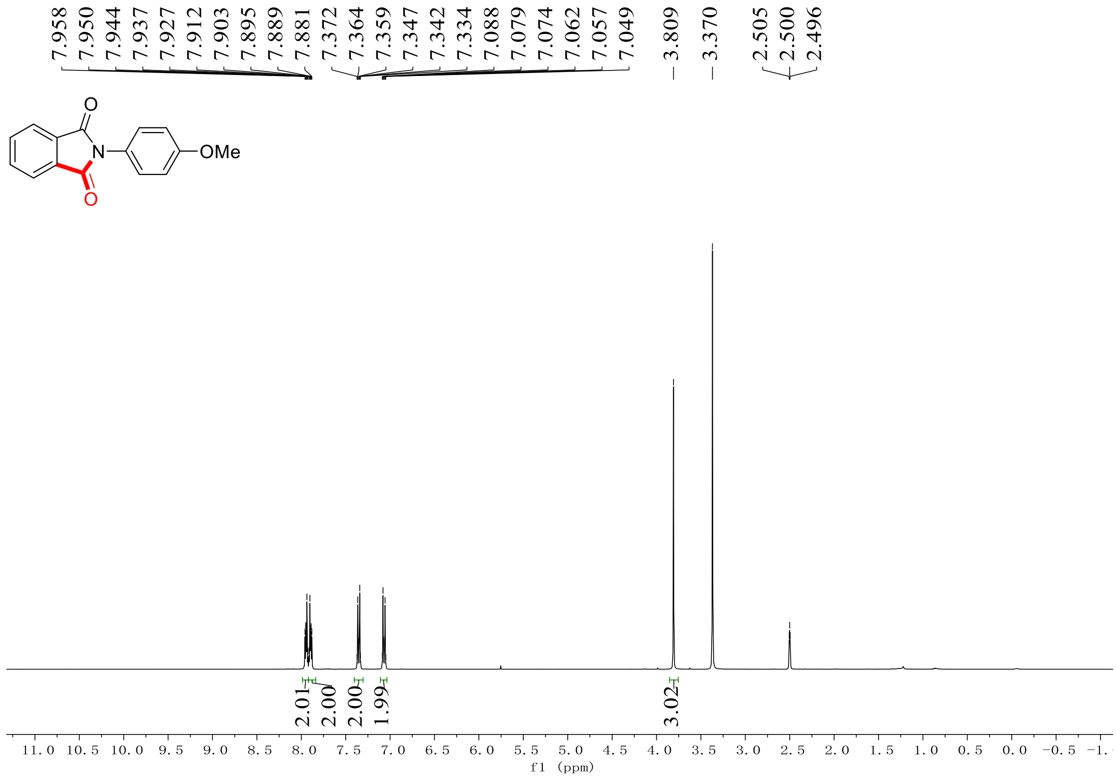

167.145
134.835
132.010
131.649
128.982
128.198
127.534
123.549
40.225
40.017
39.808
39.600
39.391
39.183
38.974
O
N
O
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)



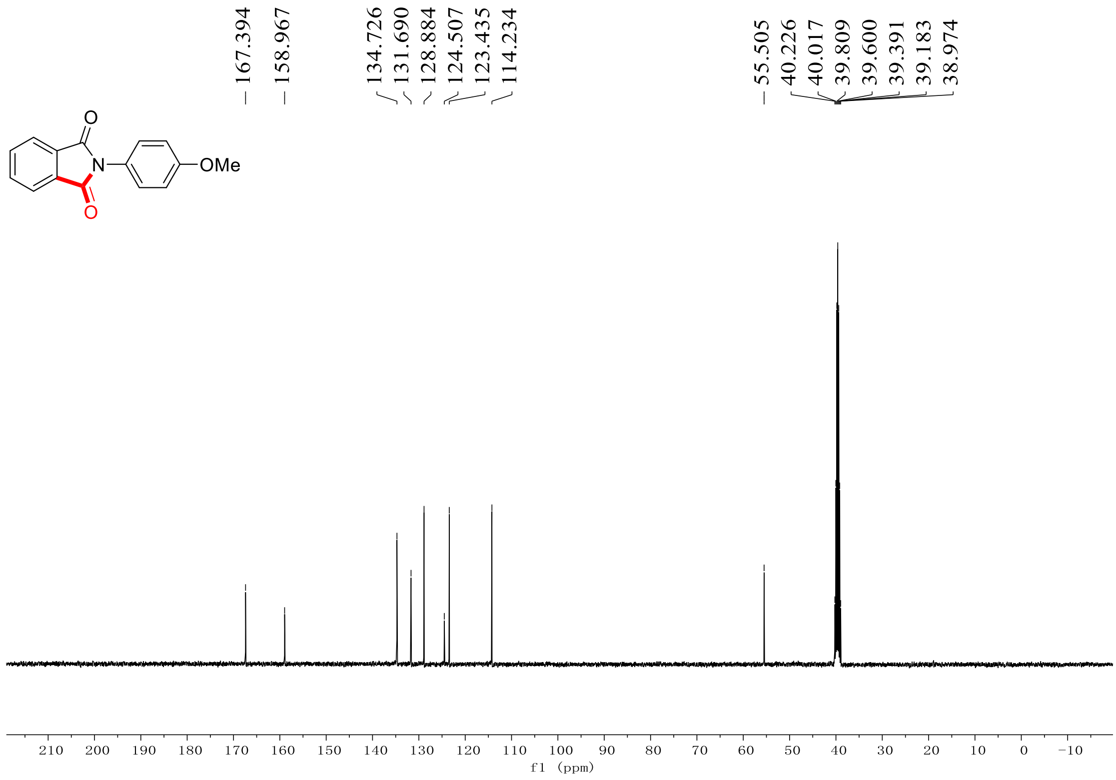
7.970 7.963 7.956 7.949 7.939 7.920 7.910 7.903 7.897 7.889 7.551 7.530 7.374 7.352
3.345
2.504 2.500 2.496
1.329
O
N
^{t}Bu
O
2.00 1.98 2.04 1.99
9.04
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5 −1.(
f1 (ppm)



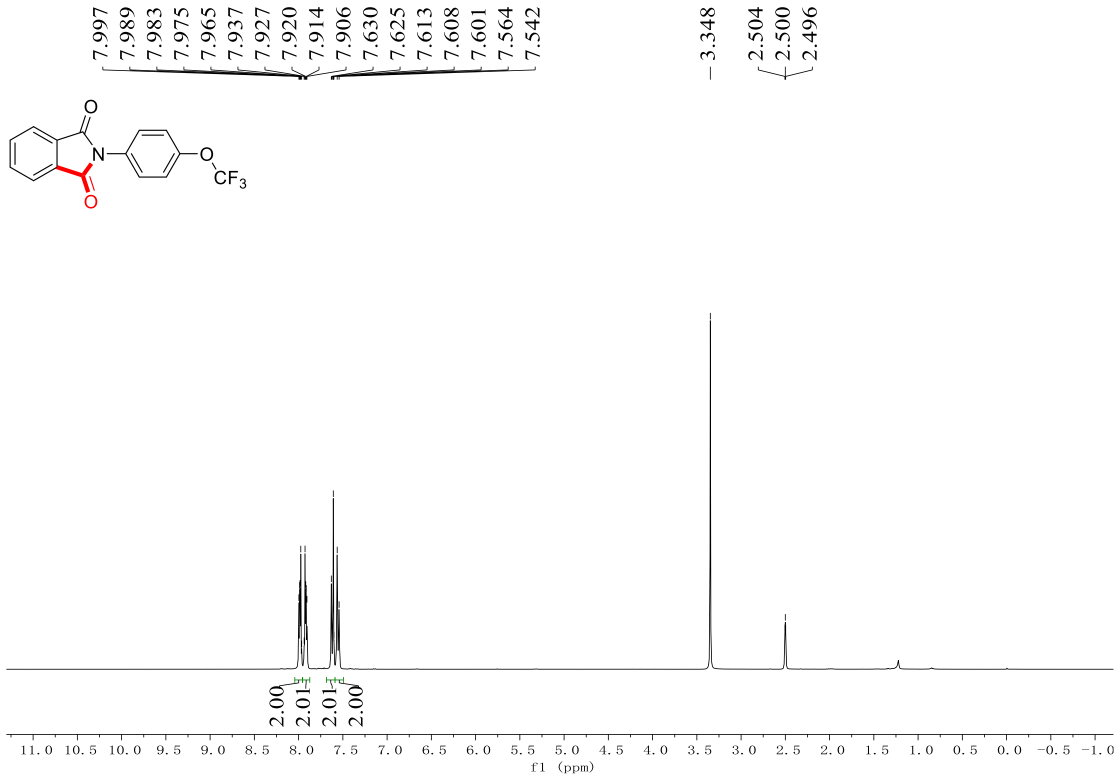
167.202
150.641 134.754 131.623 129.332 127.008 125.744 123.465
40.226 40.018 39.809 39.600 39.392 39.183 38.974 34.542 31.168
O
N
^{t}Bu
O
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)

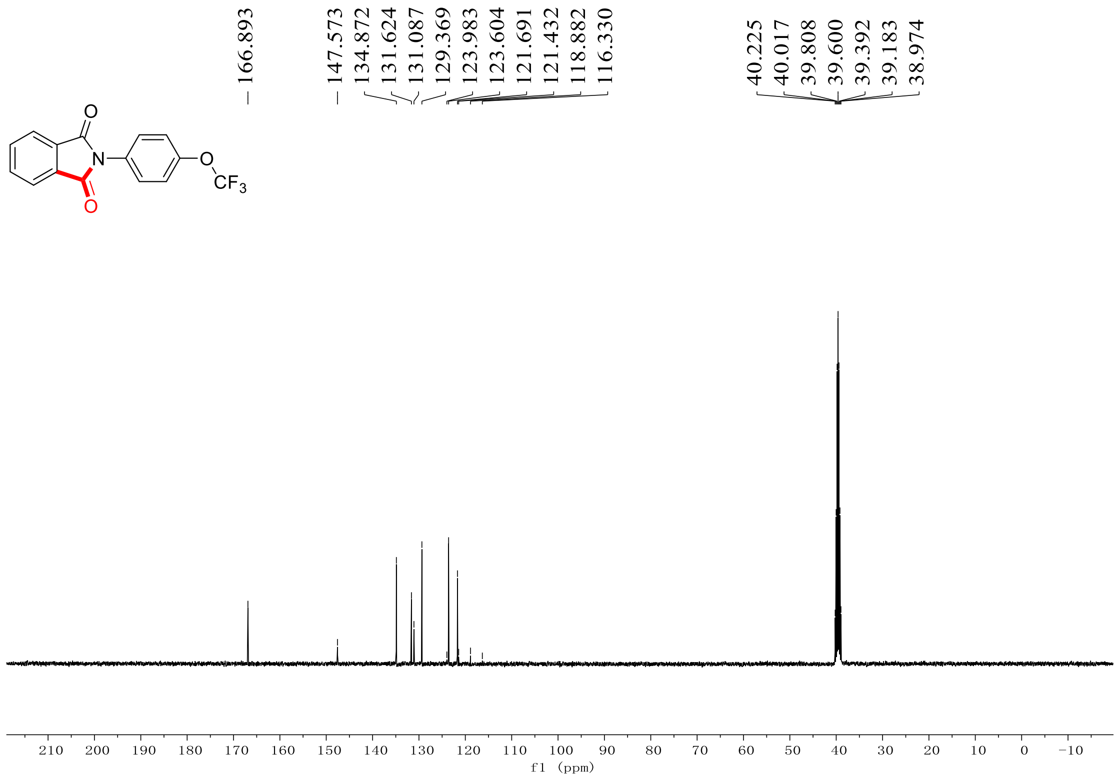


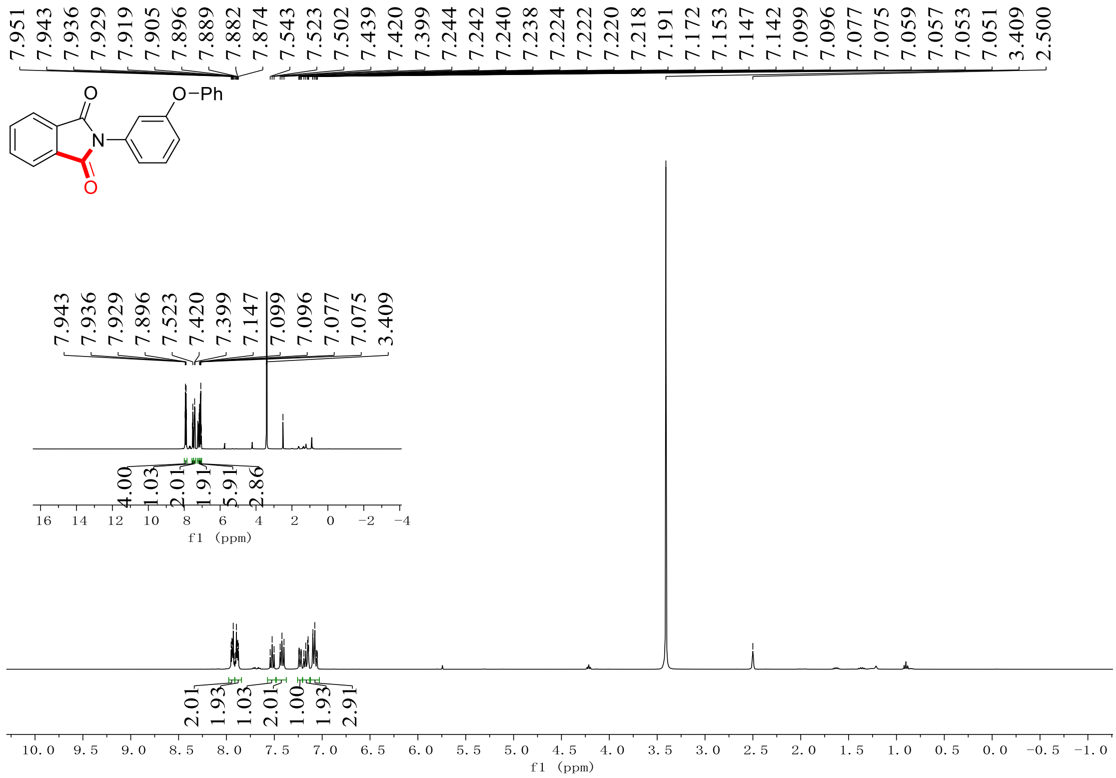
7.958
7.950
7.944
7.937
7.927
7.912
7.903
7.895
7.889
7.881
7.372
7.364
7.359
7.347
7.342
7.334
7.088
7.079
7.074
7.062
7.057
7.049
3.809
3.370
2.505
2.500
2.496
OMe
2.01
2.00
2.00
1.99
3.02
f1 (ppm)



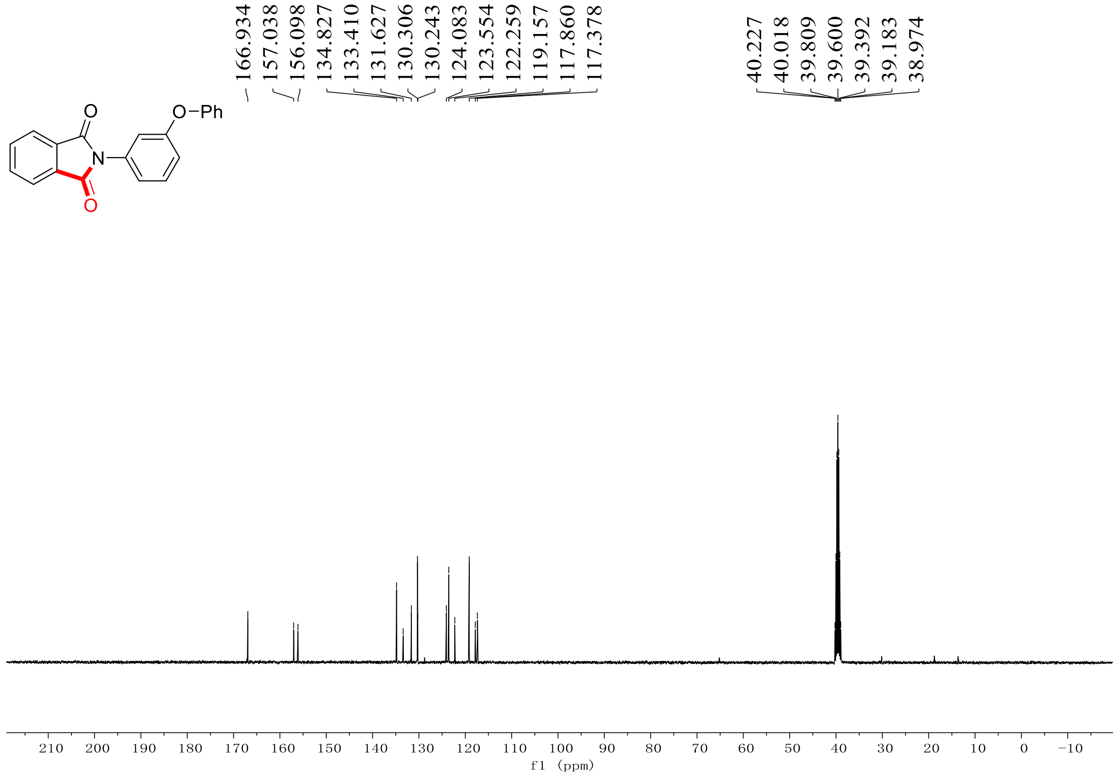
167.394
158.967
134.726
131.690
128.884
124.507
123.435
114.234
55.505
40.226
40.017
39.809
39.600
39.391
39.183
38.974
OMe
f1 (ppm)


7.997
7.989
7.983
7.975
7.965
7.937
7.927
7.920
7.914
7.906
7.630
7.625
7.613
7.608
7.601
7.564
7.542
3.348
2.504
2.500
2.496
2.00
2.01
2.01
2.00
f1 (ppm)

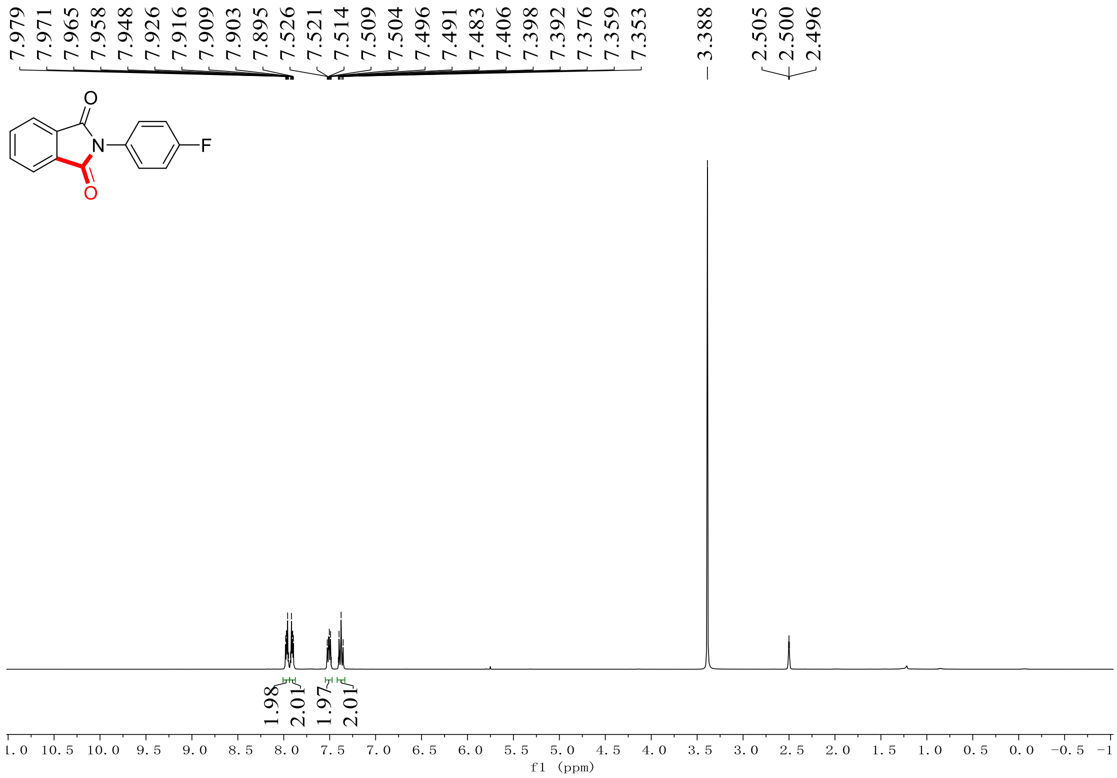

166.893
147.573
134.872
131.624
131.087
129.369
123.983
123.604
121.691
121.432
118.882
116.330
40.225
40.017
39.808
39.600
39.392
39.183
38.974
f1 (ppm)

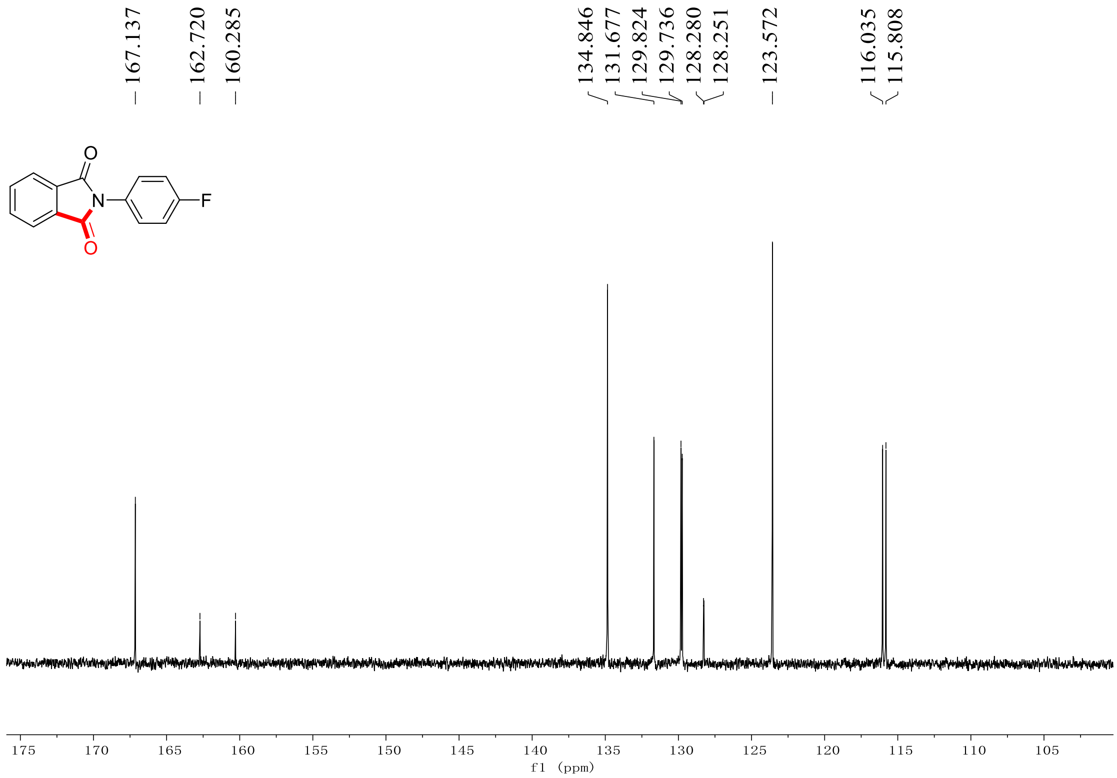


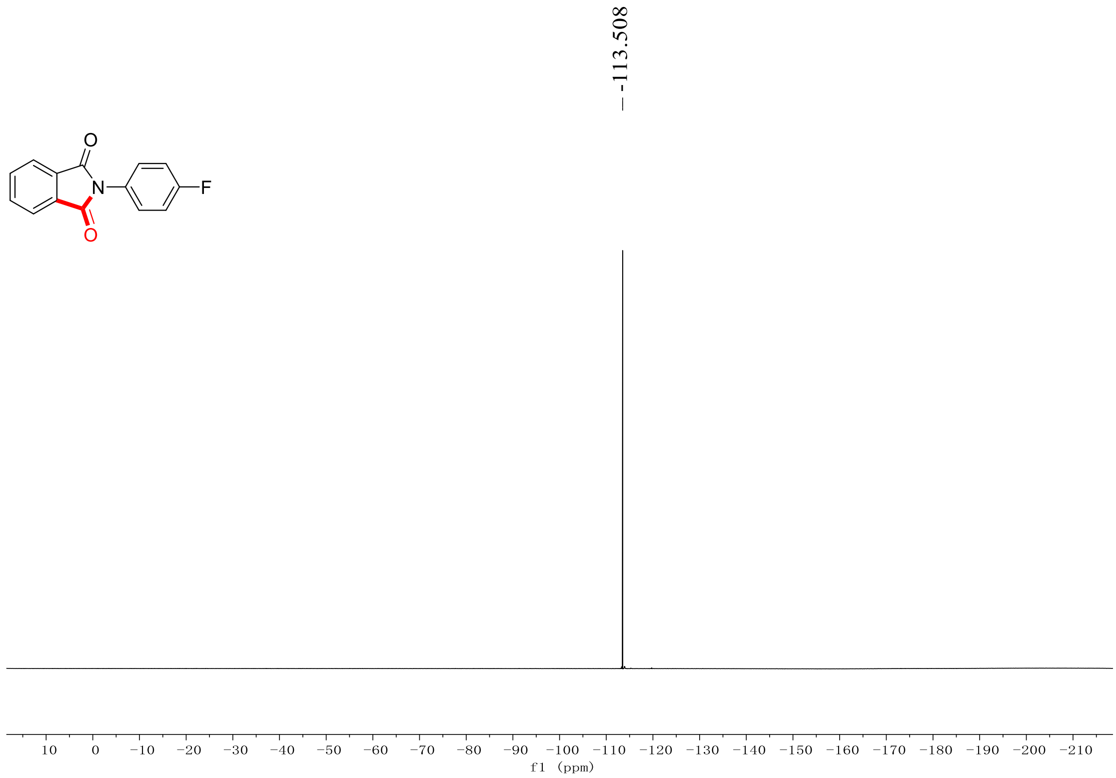
7.951 7.943 7.936 7.929 7.919 7.905 7.896 7.889 7.882 7.874 7.543 7.523 7.502 7.439 7.420 7.399 7.244 7.242 7.240 7.238 7.224 7.222 7.220 7.218 7.191 7.172 7.153 7.147 7.142 7.099 7.096 7.077 7.075 7.059 7.057 7.053 7.051 3.409 2.500
O−Ph
N
O
O
7.943 7.936 7.929 7.896 7.523 7.420 7.399 7.147 7.099 7.096 7.077 7.075 3.409
4.00 1.03 2.01 1.91 5.91 2.86
16 14 12 10 8 6 4 2 0 −2 −4
f1 (ppm)
2.01 1.93 1.03 2.01 1.00 1.93 2.91
10.0 9.5 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 −0.5 −1.0
f1 (ppm)



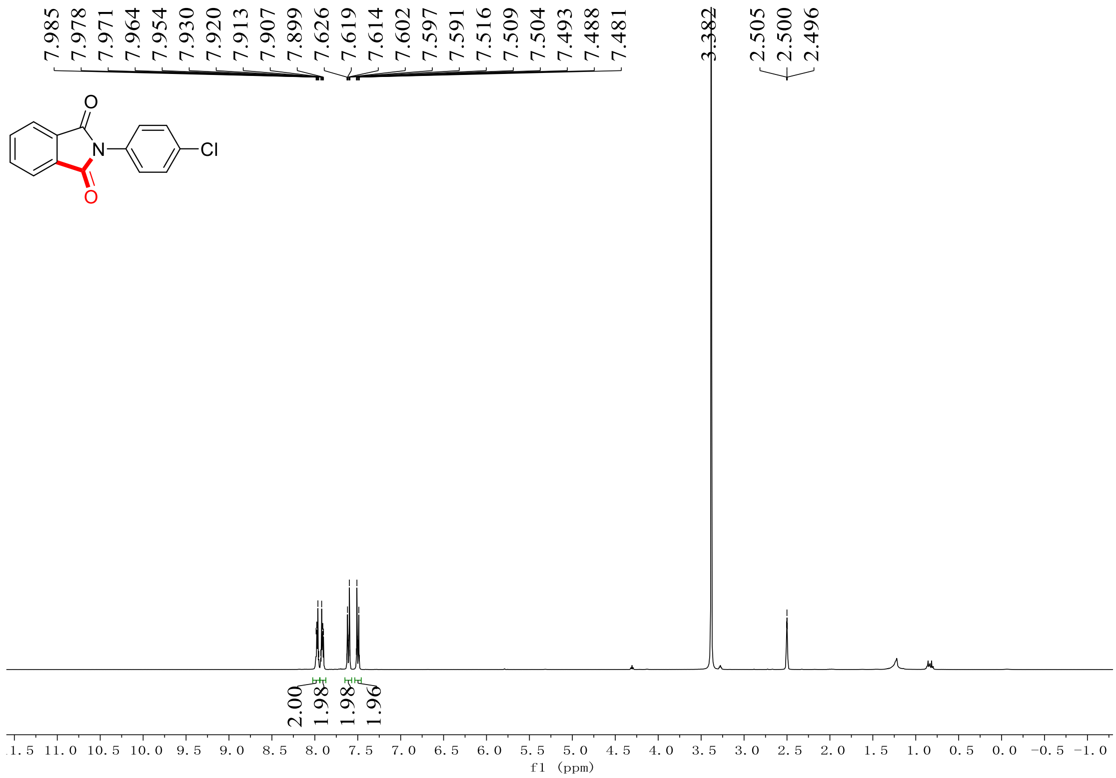
166.934 157.038 156.098 134.827 133.410 131.627 130.306 130.243 124.083 123.554 122.259 119.157 117.860 117.378
40.227 40.018 39.809 39.600 39.392 39.183 38.974
O−Ph
N
O
O
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)



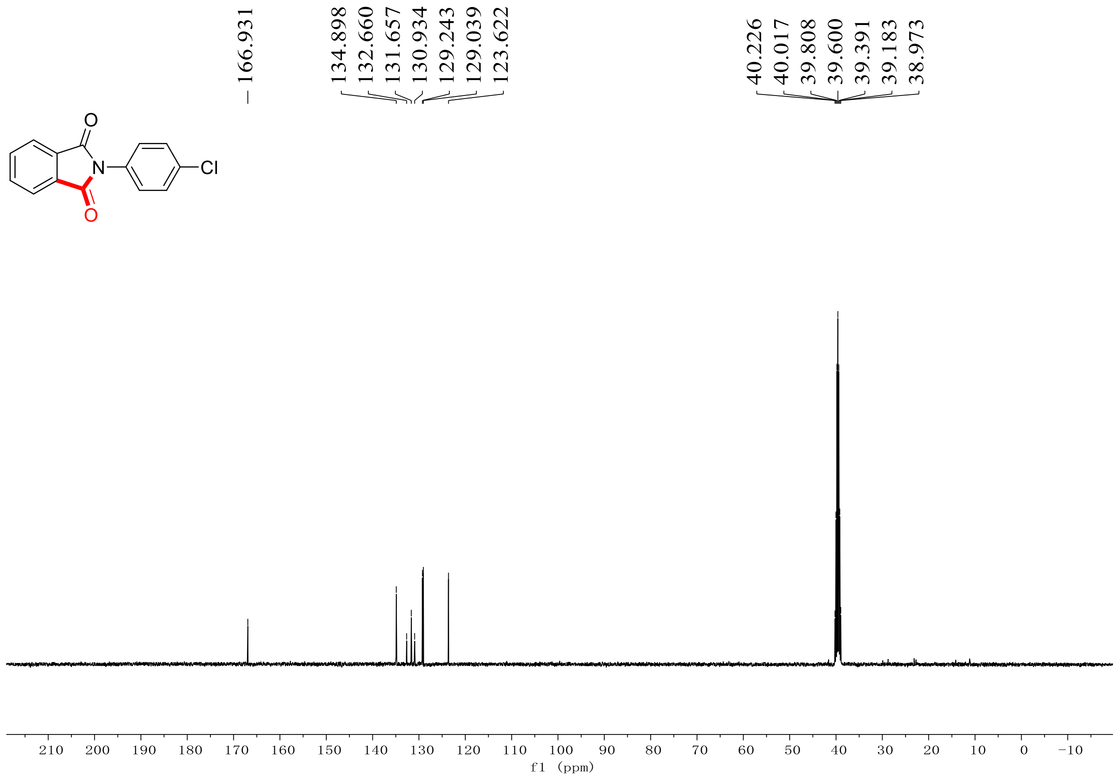




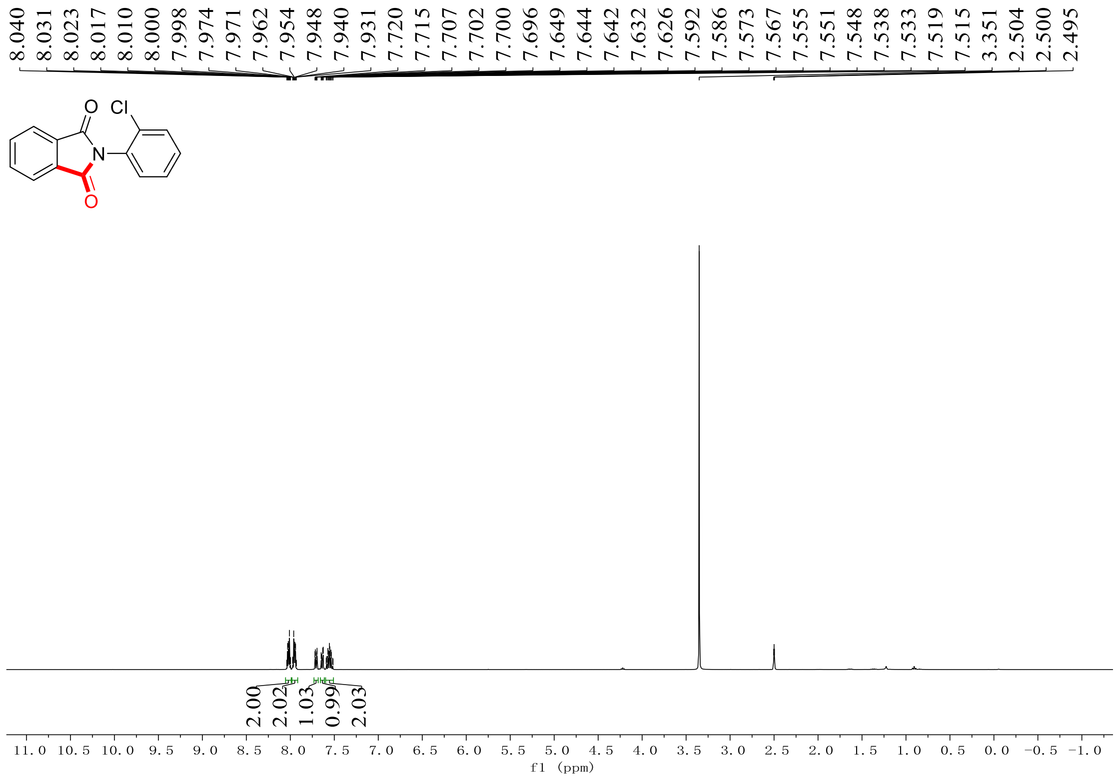
-113.508
O
N
F
O
10 0 −10 −20 −30 −40 −50 −60 −70 −80 −90 −100 −110 −120 −130 −140 −150 −160 −170 −180 −190 −200 −210
f1 (ppm)



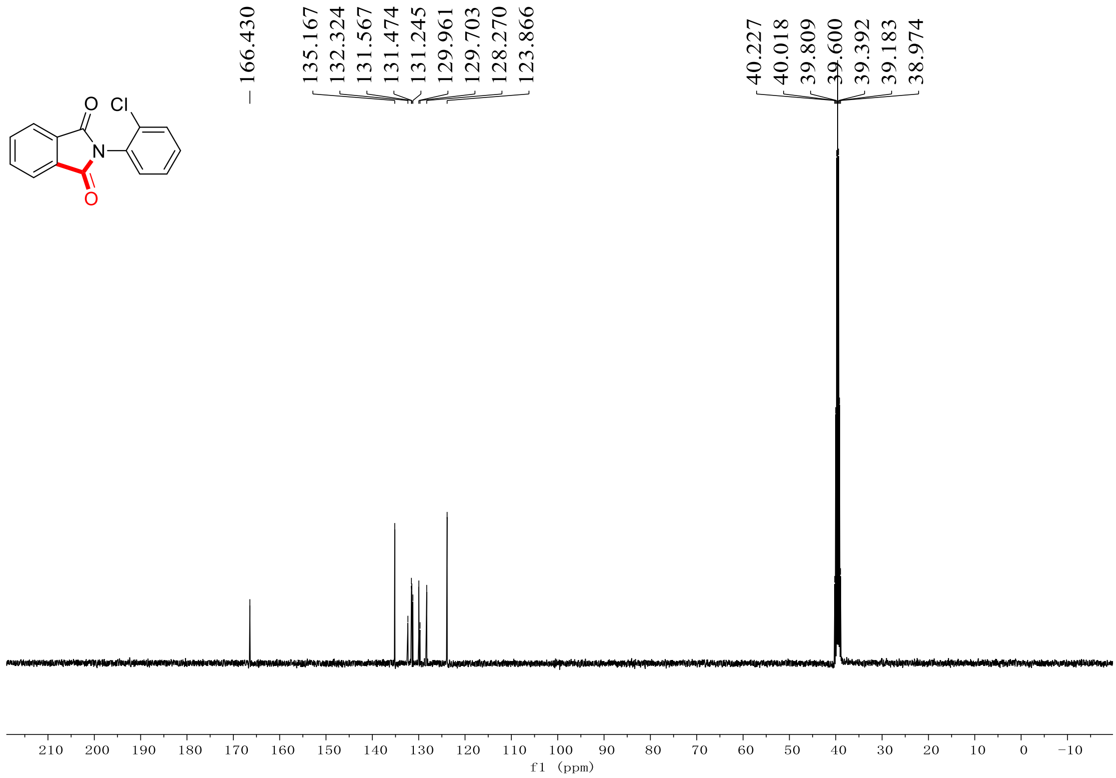
7.985
7.978
7.971
7.964
7.954
7.930
7.920
7.913
7.907
7.899
7.626
7.619
7.614
7.602
7.597
7.591
7.516
7.509
7.504
7.493
7.488
7.481
3.382
2.505
2.500
2.496
O
N
Cl
O
2.00
1.98
1.98
1.96
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5 −1.0
f1 (ppm)

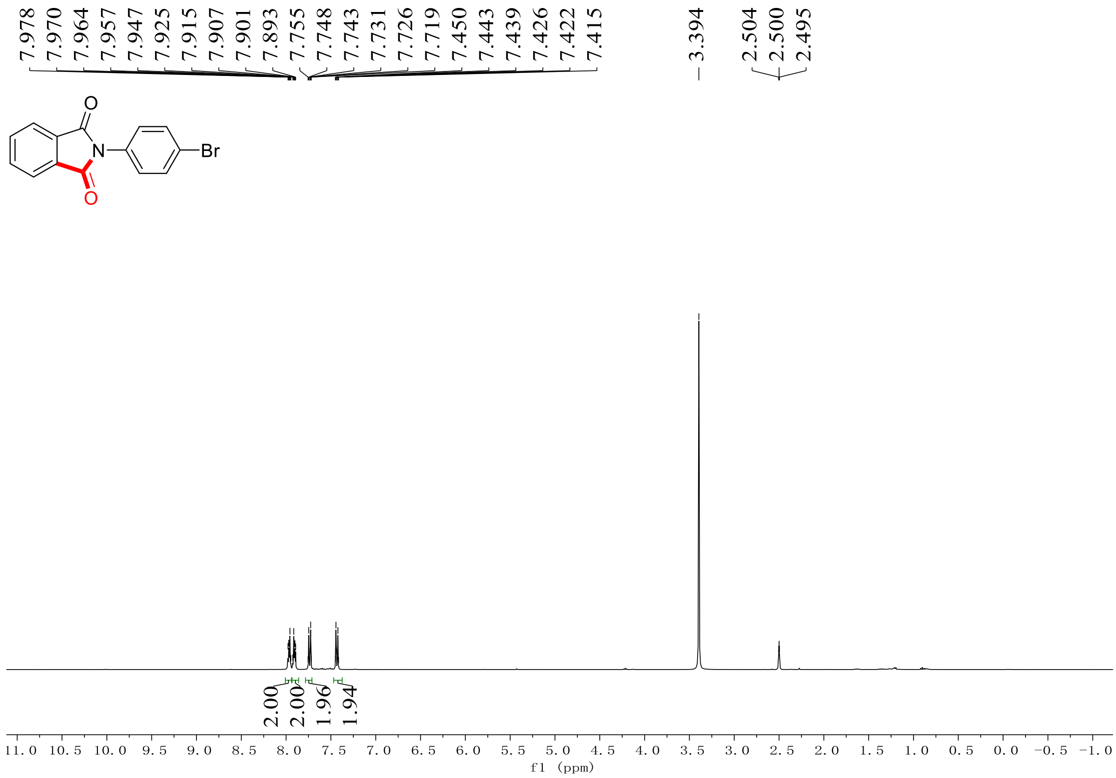


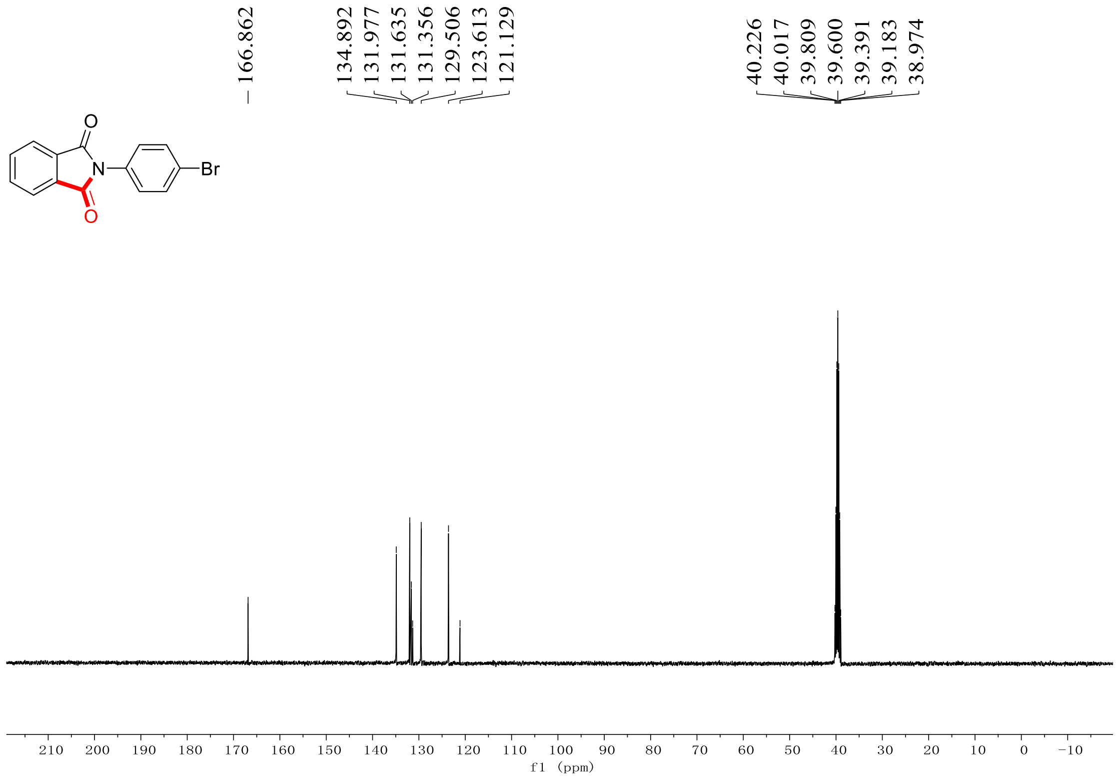
166.931
134.898
132.660
131.657
130.934
129.243
129.039
123.622
40.226
40.017
39.808
39.600
39.391
39.183
38.973
O
N
Cl
O
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

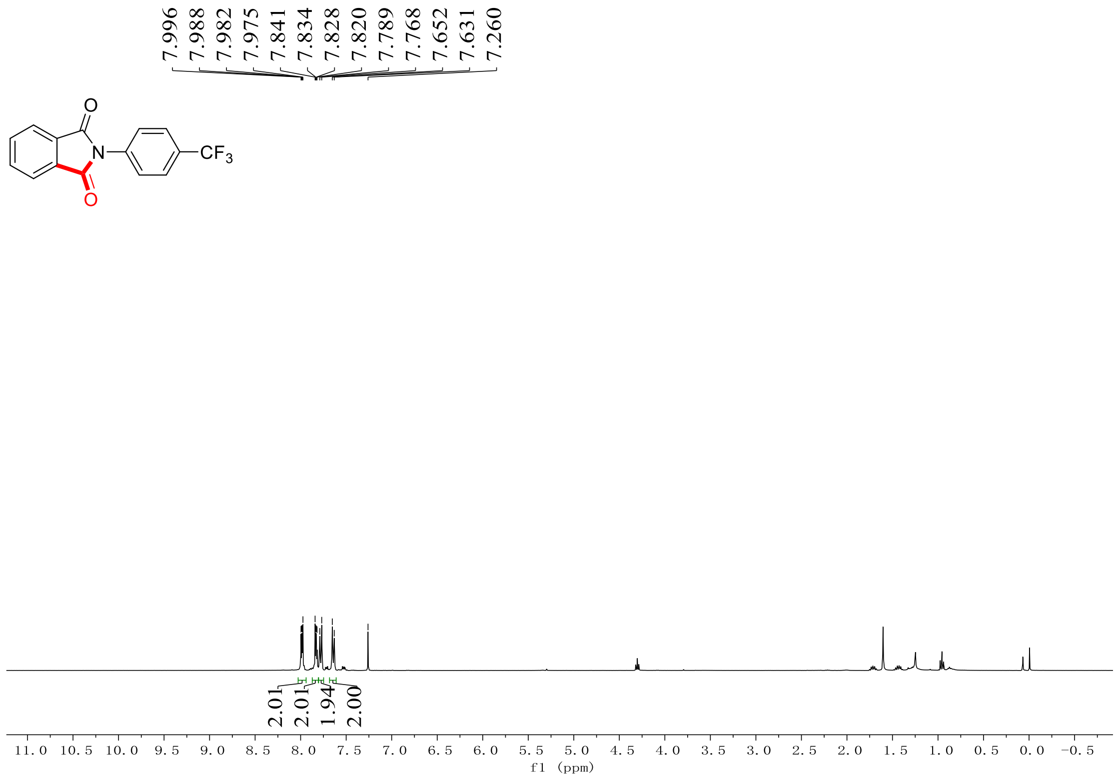


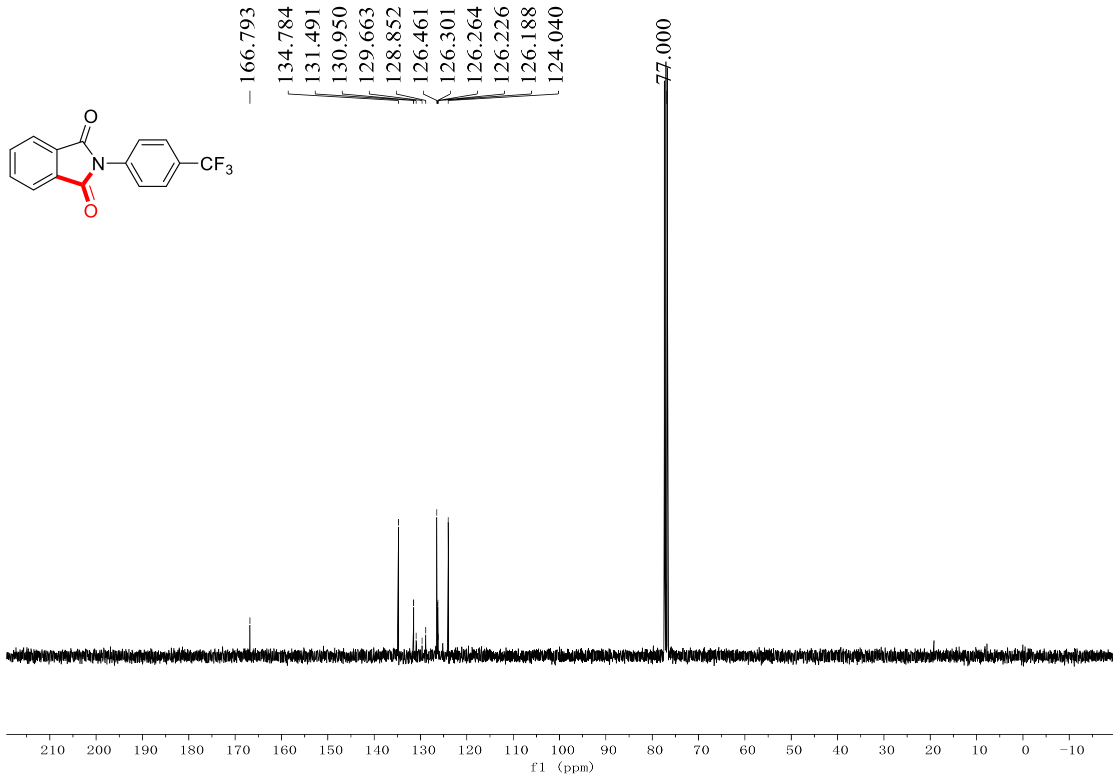
8.040
8.031
8.023
8.017
8.010
8.000
7.998
7.974
7.971
7.962
7.954
7.948
7.940
7.931
7.720
7.715
7.707
7.702
7.700
7.696
7.649
7.644
7.642
7.632
7.626
7.592
7.586
7.573
7.567
7.555
7.551
7.548
7.538
7.533
7.519
7.515
3.351
2.504
2.500
2.495
O
Cl
N
O
2.00
2.02
1.03
0.99
2.03
11.0 10.5 10.0 9.5 9.0 8.5 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 -0.5 -1.0
f1 (ppm)


O
Cl
N
O
166.430
135.167
132.324
131.567
131.474
131.245
129.961
129.703
128.270
123.866
40.227
40.018
39.809
39.600
39.392
39.183
38.974
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)

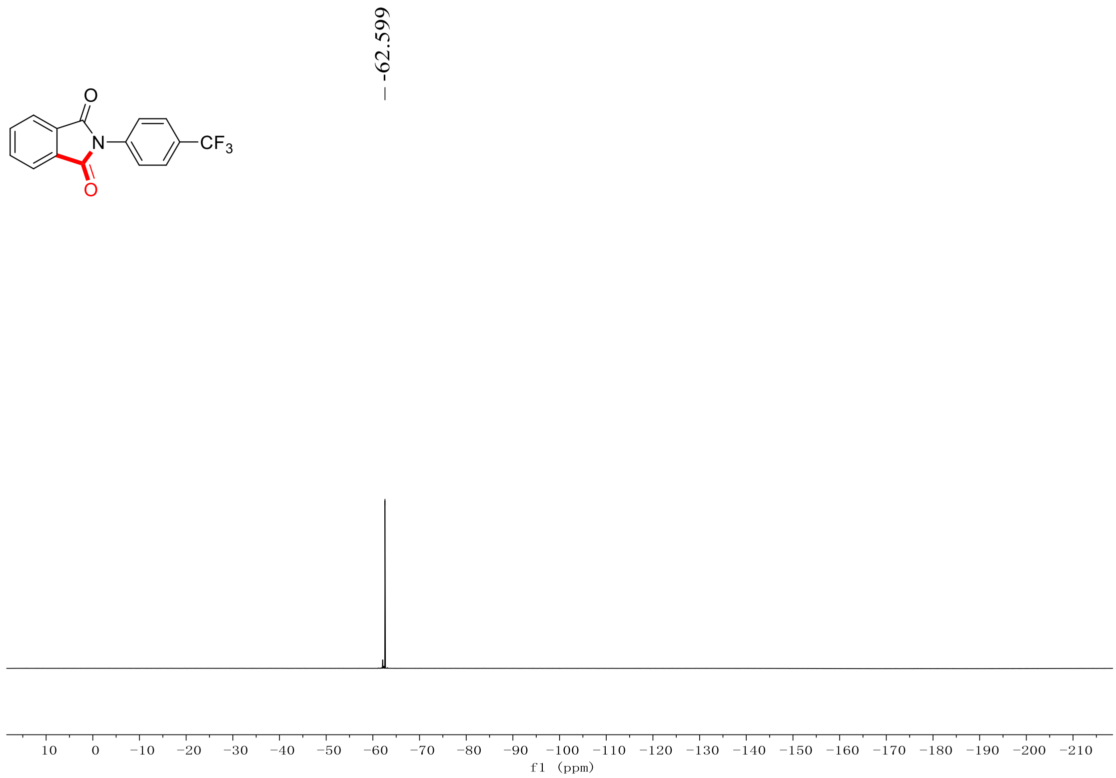

O
N
Br
O
7.978
7.970
7.964
7.957
7.947
7.925
7.915
7.907
7.901
7.893
7.755
7.748
7.743
7.731
7.726
7.719
7.450
7.443
7.439
7.426
7.422
7.415
3.394
2.504
2.500
2.495
2.00
2.00
1.96
1.94
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5 −1.0
f1 (ppm)


166.862
134.892
131.977
131.635
131.356
129.506
123.613
121.129
40.226
40.017
39.809
39.600
39.391
39.183
38.974
O
N
Br
O
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)

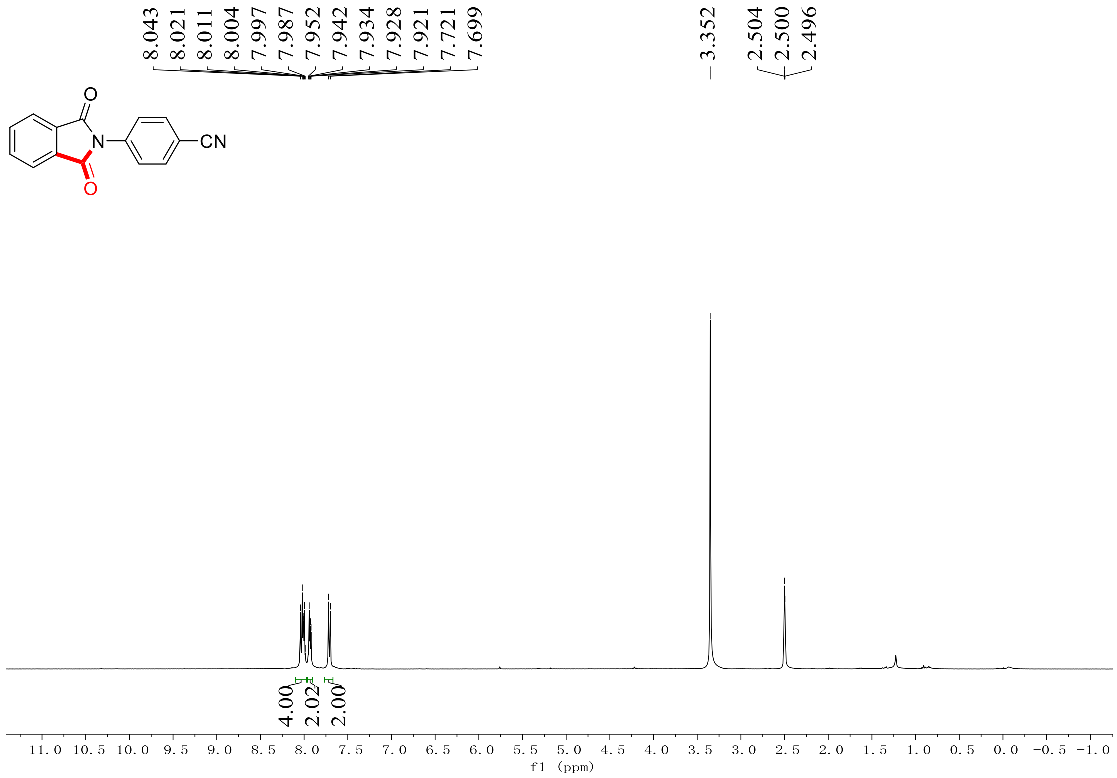

7.996
7.988
7.982
7.975
7.841
7.834
7.828
7.820
7.789
7.768
7.652
7.631
7.260
O
N
CF3
O
2.01
2.01
1.94
2.00
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5
f1 (ppm)

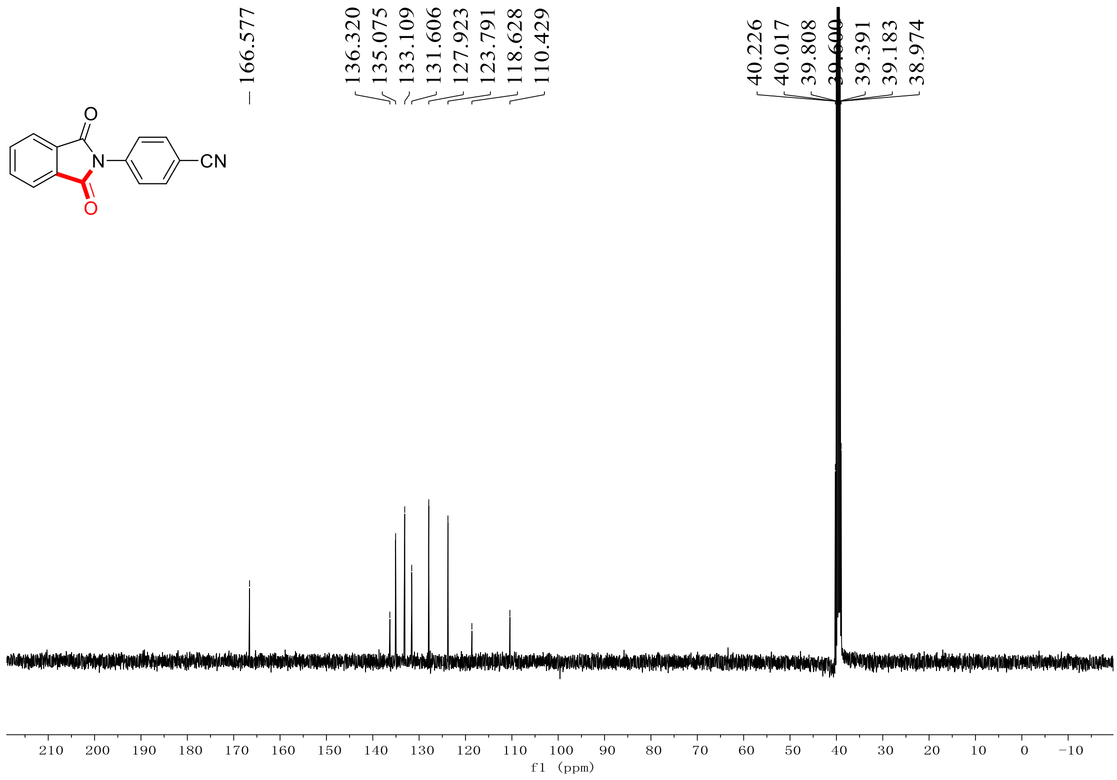


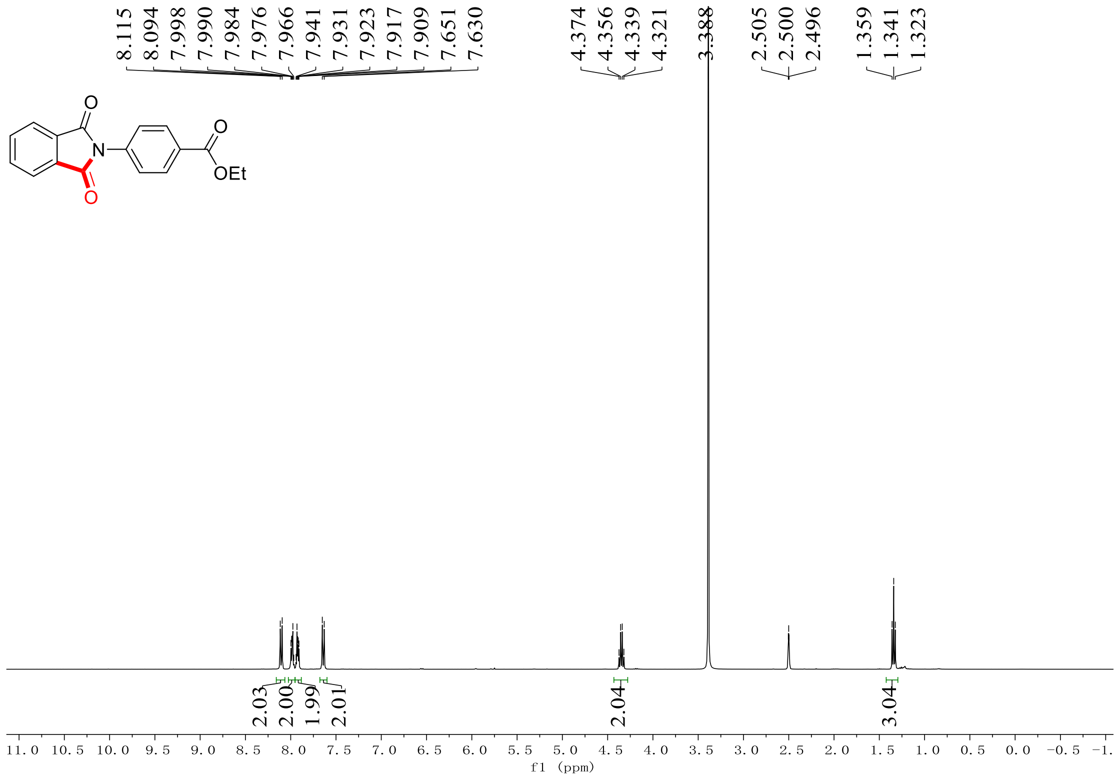
166.793
134.784
131.491
130.950
129.663
128.852
126.461
126.301
126.264
126.226
126.188
124.040
77.000
O
N
CF3
O
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)

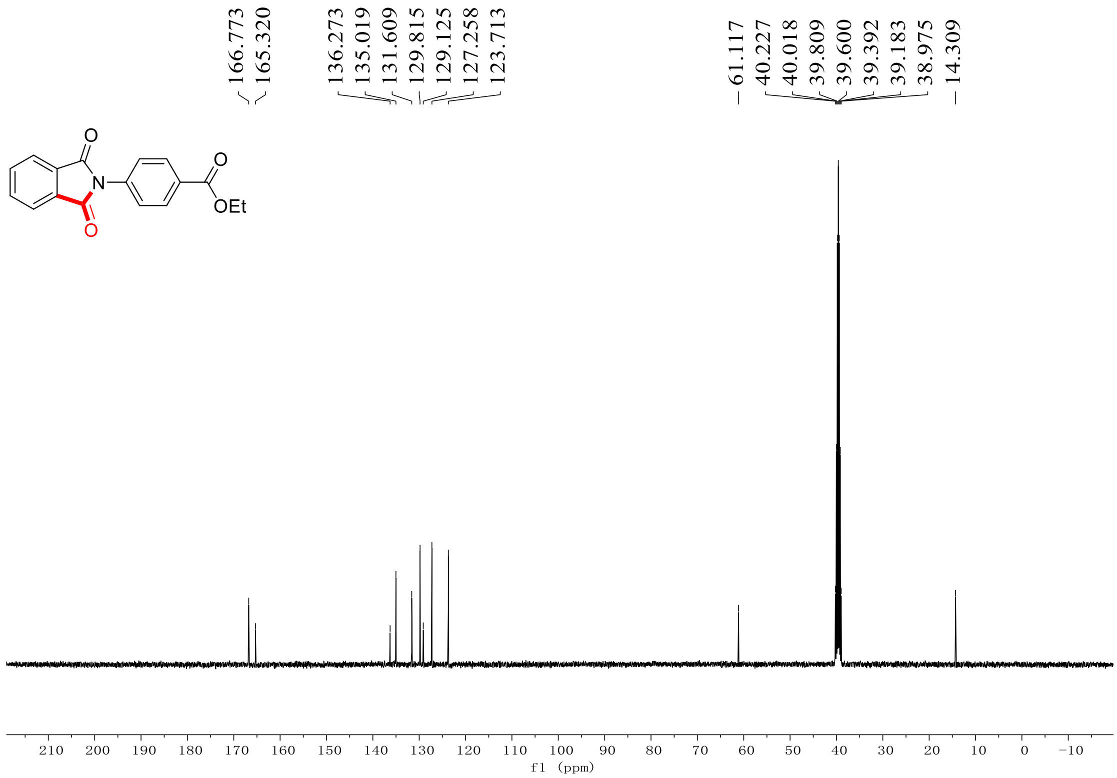


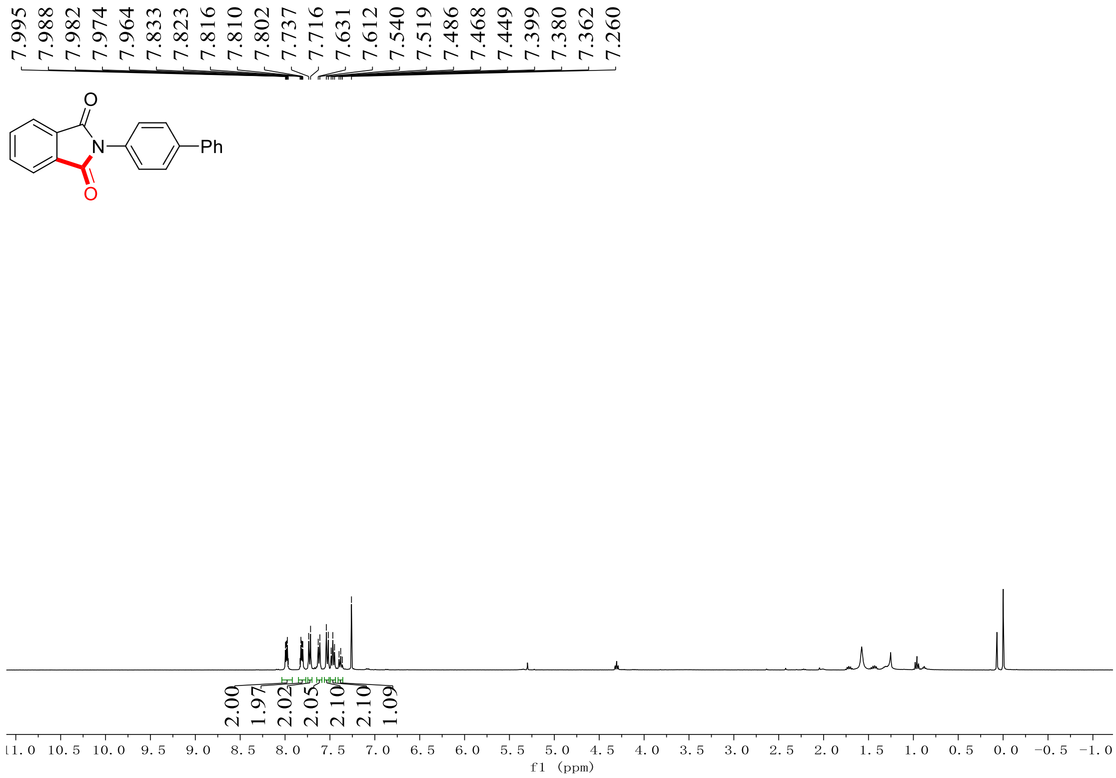
-62.599
O
N
CF3
O
10 0 −10 −20 −30 −40 −50 −60 −70 −80 −90 −100 −110 −120 −130 −140 −150 −160 −170 −180 −190 −200 −210
f1 (ppm)

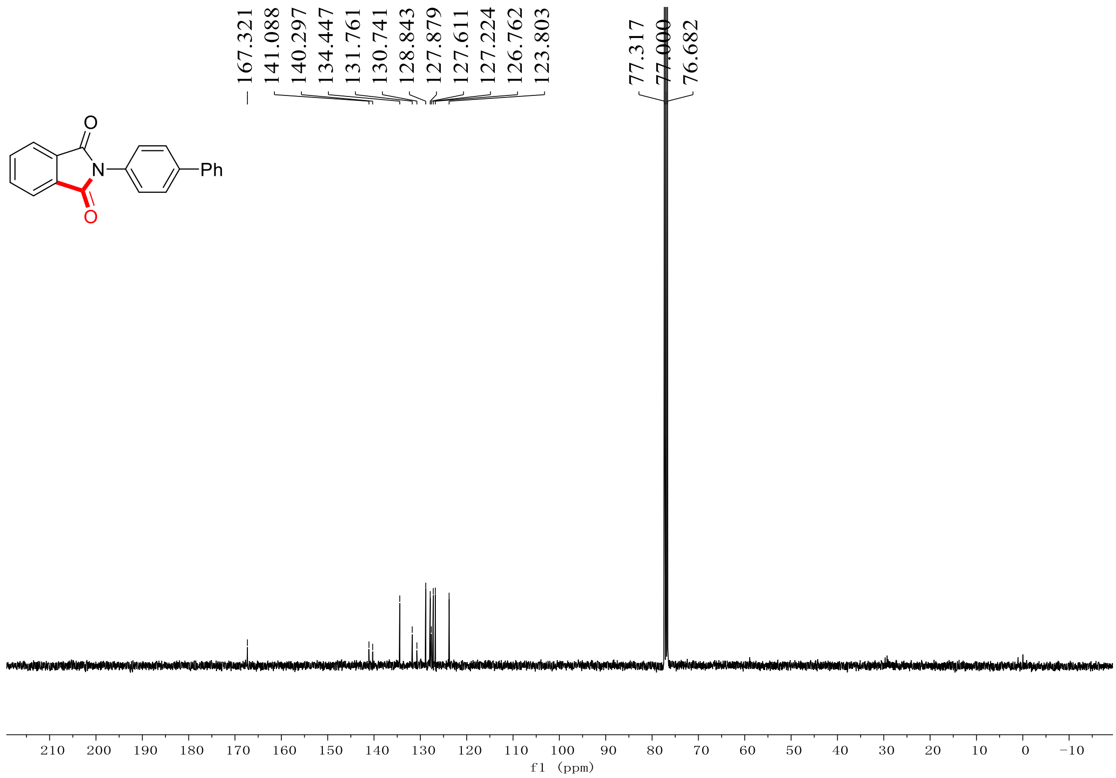

8.043
8.021
8.011
8.004
7.997
7.987
7.952
7.942
7.934
7.928
7.921
7.721
7.699
3.352
2.504
2.500
2.496
O
N
CN
O
4.00
2.02
2.00
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5 −1.0
f1 (ppm)

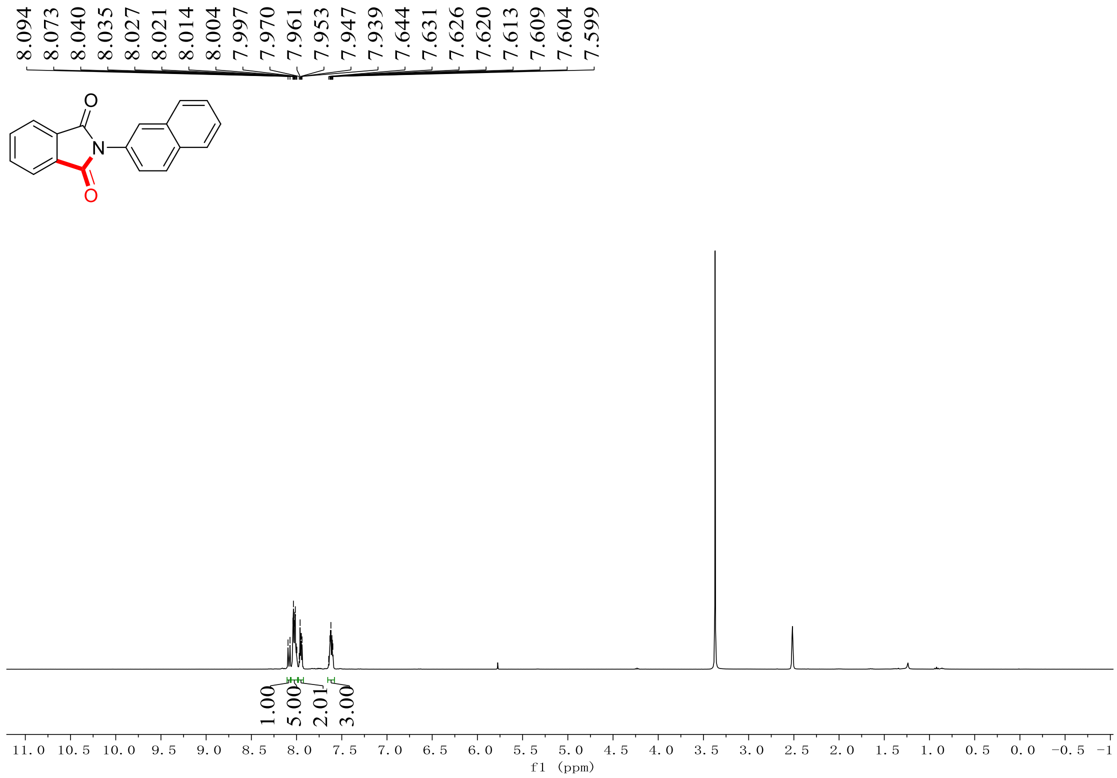

166.577
136.320
135.075
133.109
131.606
127.923
123.791
118.628
110.429
40.226
40.017
39.808
39.600
39.391
39.183
38.974
O
N
CN
O
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)

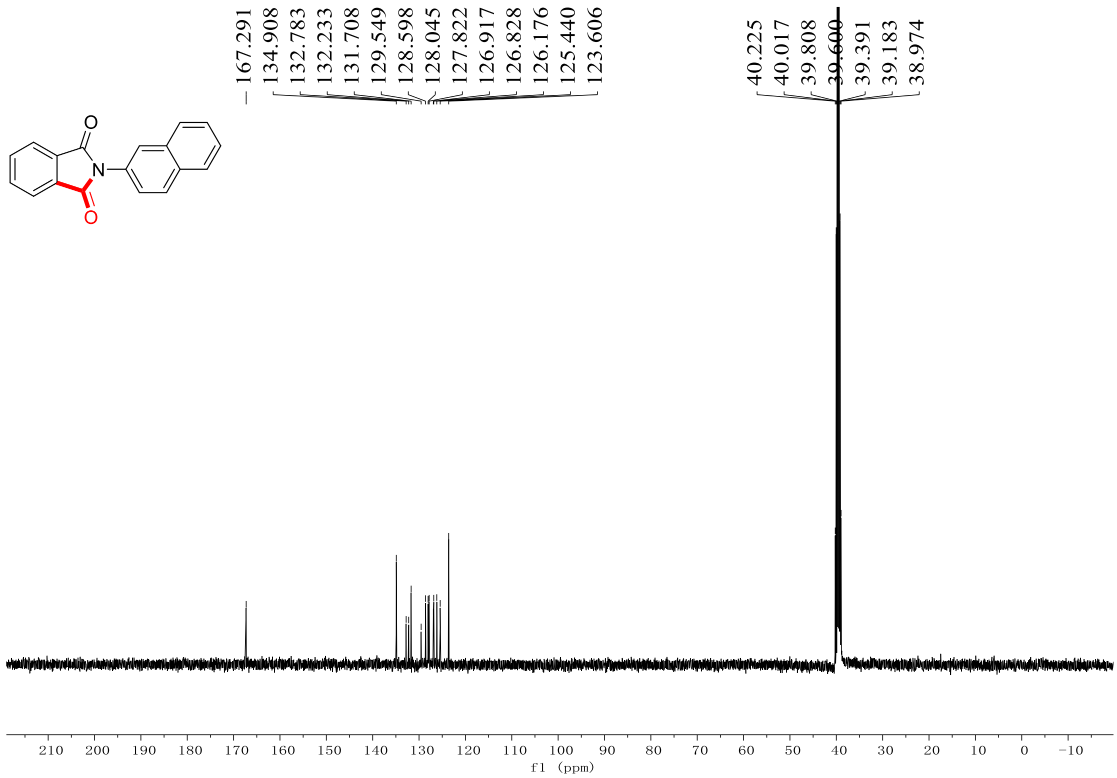

8.115
8.094
7.998
7.990
7.984
7.976
7.966
7.941
7.931
7.923
7.917
7.909
7.651
7.630
4.374
4.356
4.339
4.321
3.388
2.505
2.500
2.496
1.359
1.341
1.323
O
N
O
OEt
O
2.03
2.00
1.99
2.01
2.04
3.04
f1 (ppm)

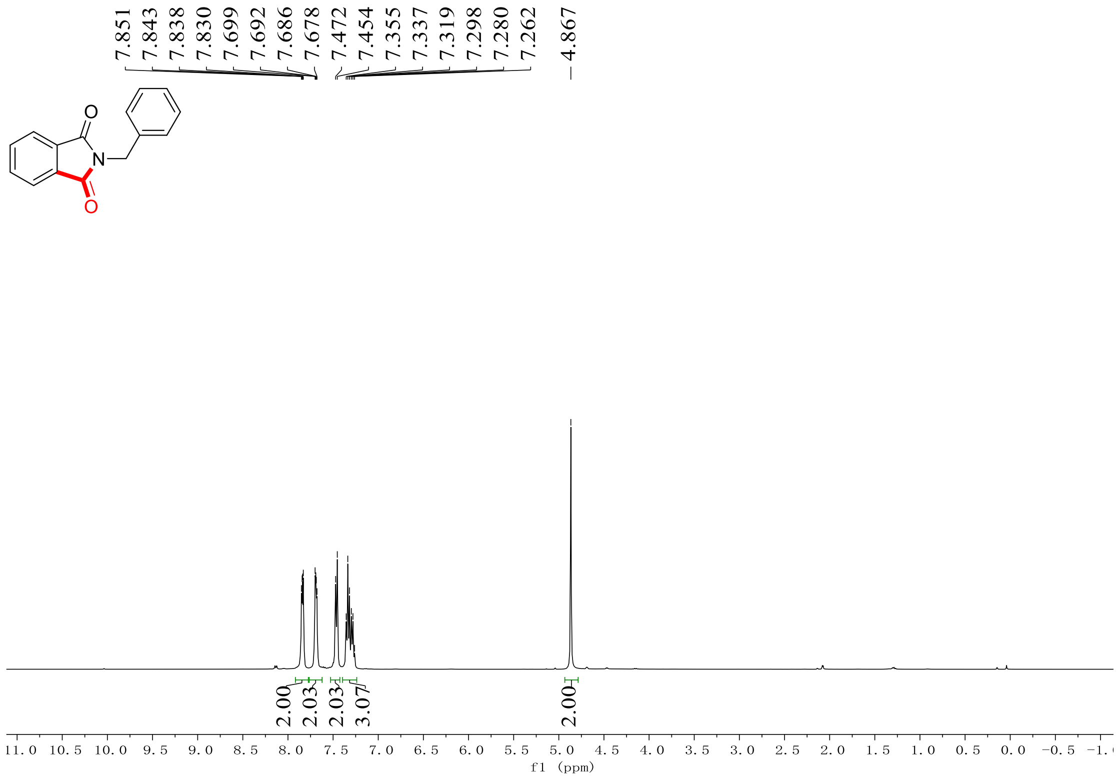

166.773
165.320
136.273
135.019
131.609
129.815
129.125
127.258
123.713
61.117
40.227
40.018
39.809
39.600
39.392
39.183
38.975
14.309
O
N
O
OEt
O
f1 (ppm)

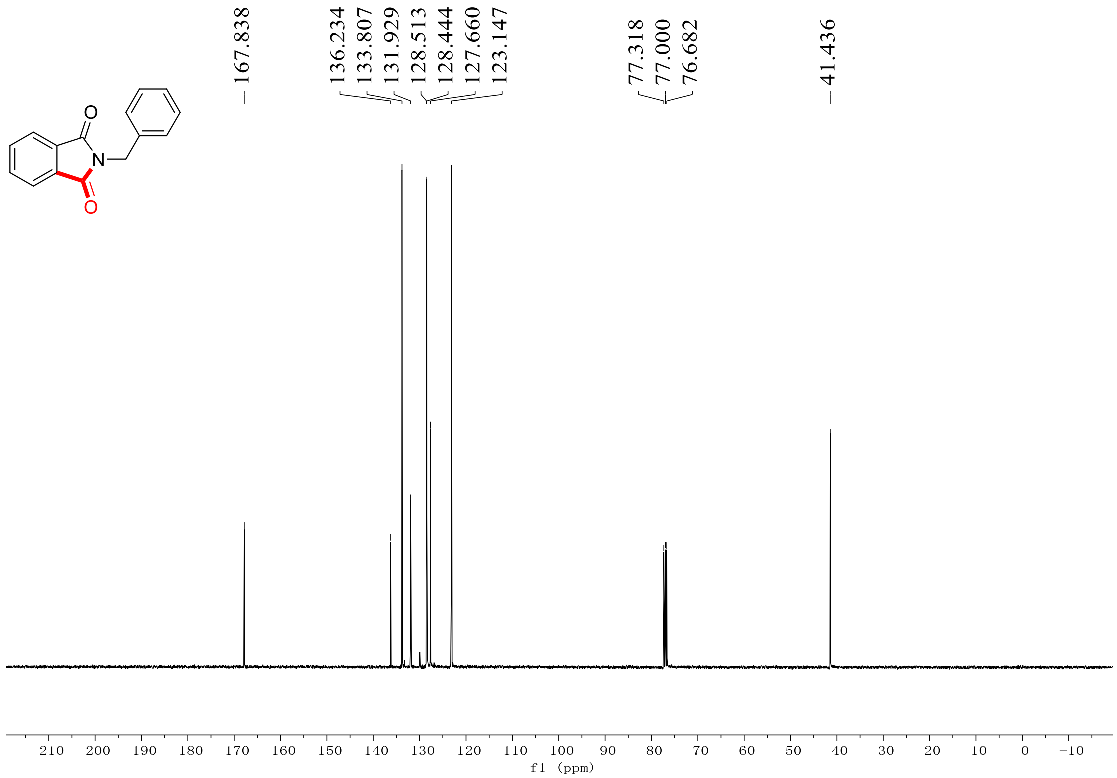

7.995
7.988
7.982
7.974
7.964
7.833
7.823
7.816
7.810
7.802
7.737
7.716
7.631
7.612
7.540
7.519
7.486
7.468
7.449
7.399
7.380
7.362
7.260
O
N
Ph
O
2.00
1.97
2.02
2.05
2.10
2.10
1.09
f1 (ppm)

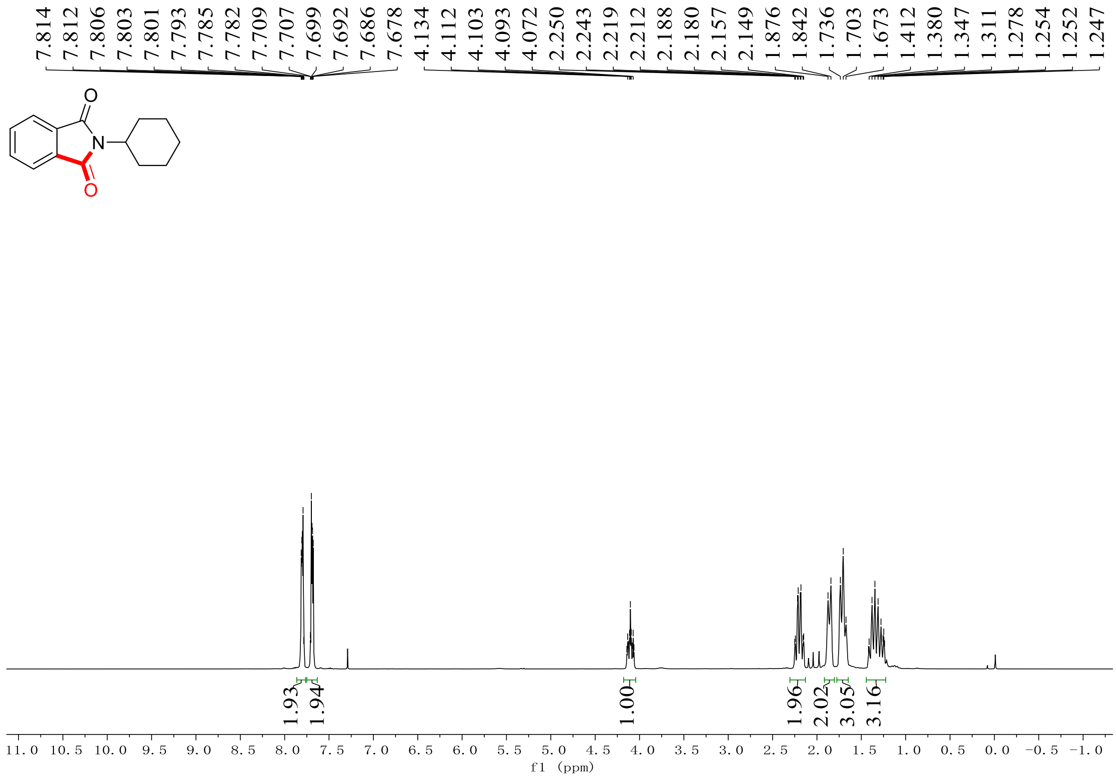

167.321
141.088
140.297
134.447
131.761
130.741
128.843
127.879
127.611
127.224
126.762
123.803
77.317
77.000
76.682
O
N
Ph
O
f1 (ppm)

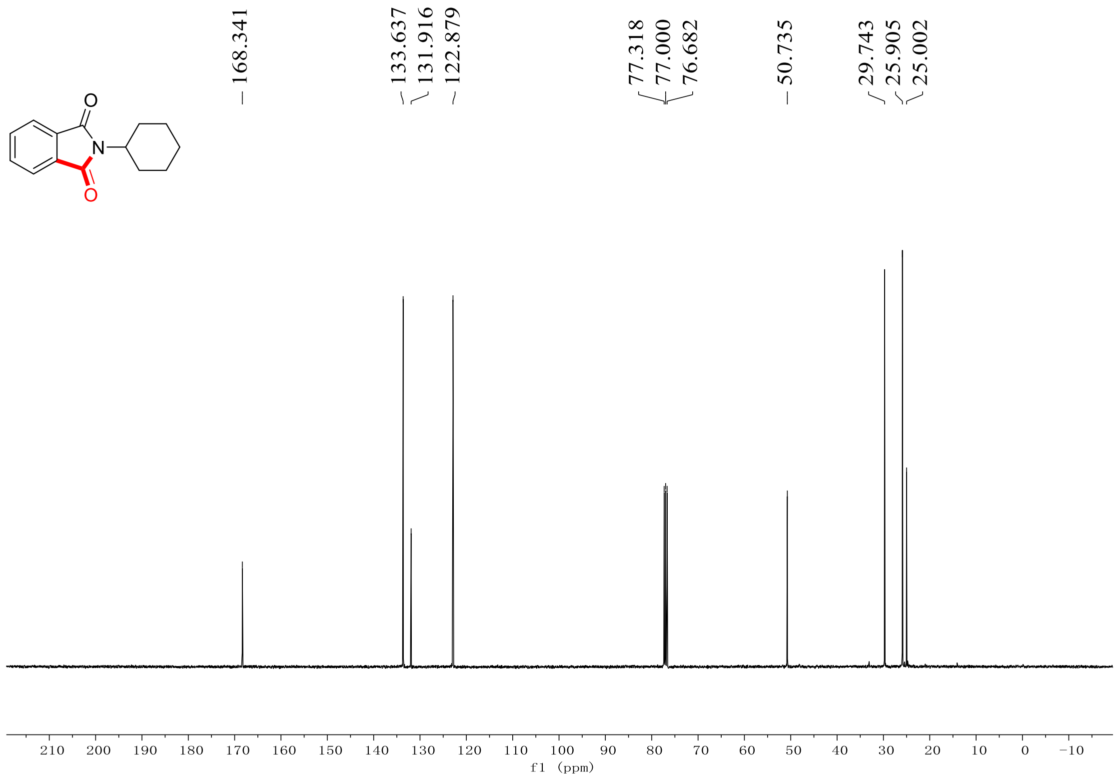

8.094
8.073
8.040
8.035
8.027
8.021
8.014
8.004
7.997
7.970
7.961
7.953
7.947
7.939
7.644
7.631
7.626
7.620
7.613
7.609
7.604
7.599
O
N
O
1.00
5.00
2.01
3.00
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1
f1 (ppm)

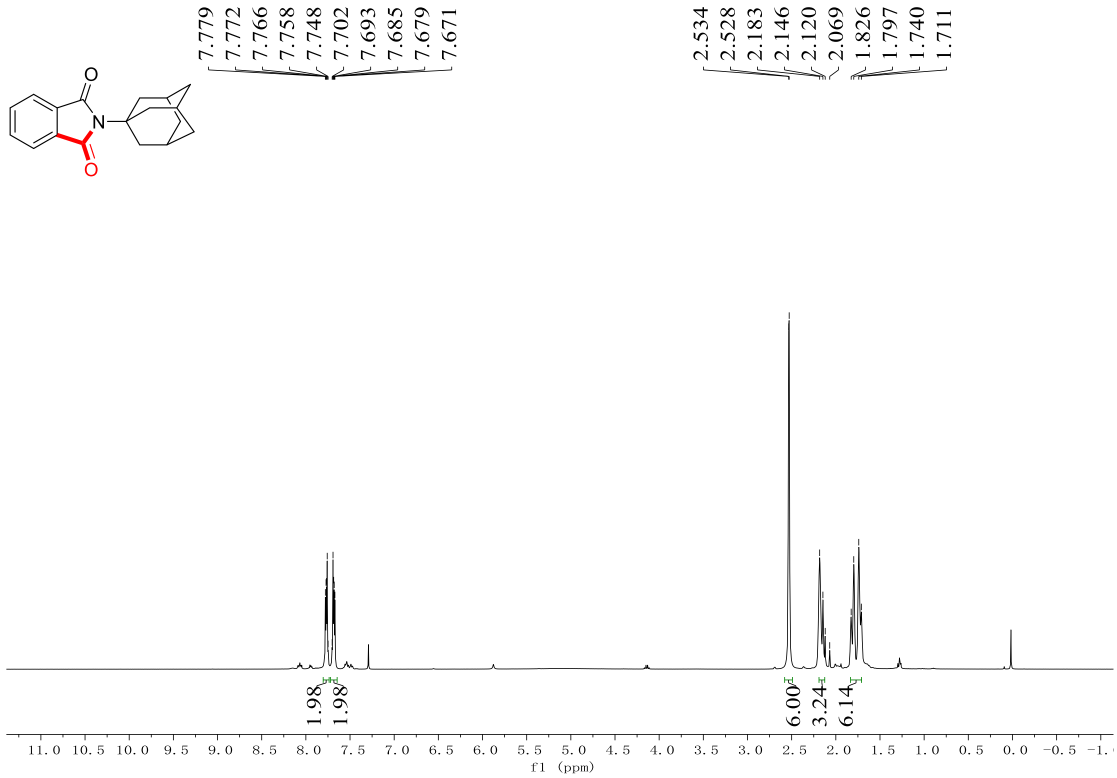

167.291
134.908
132.783
132.233
131.708
129.549
128.598
128.045
127.822
126.917
126.828
126.176
125.440
123.606
40.225
40.017
39.808
39.600
39.391
39.183
38.974
O
N
O
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

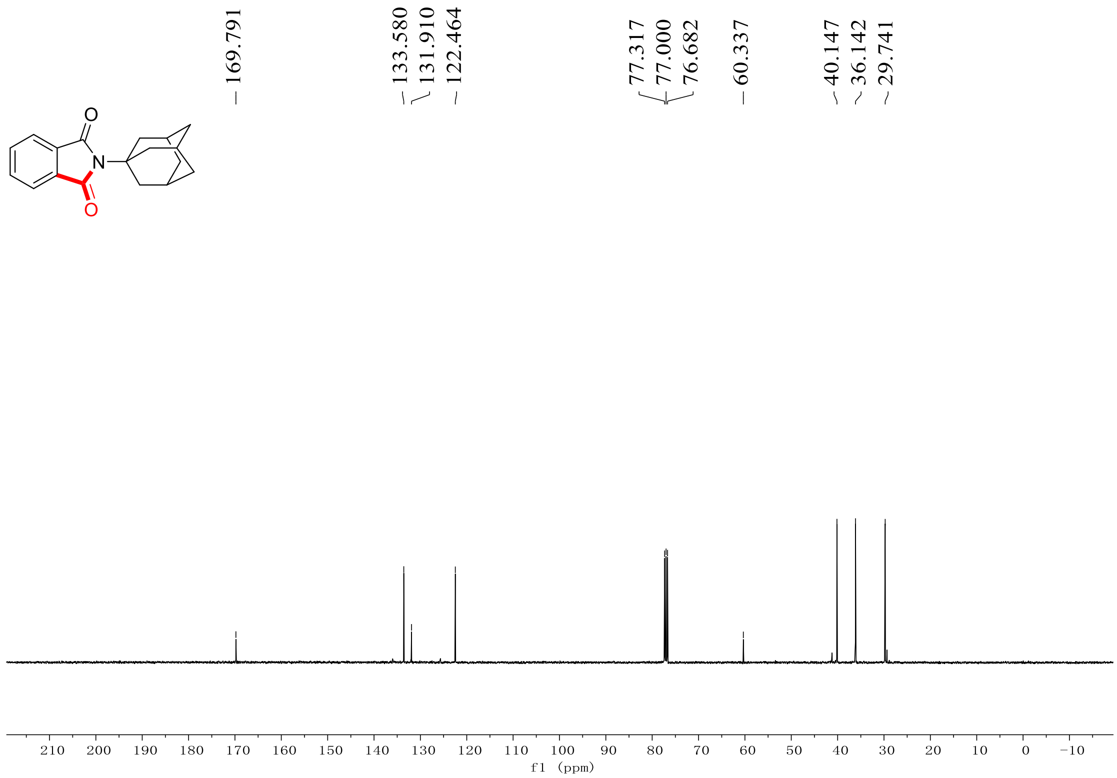


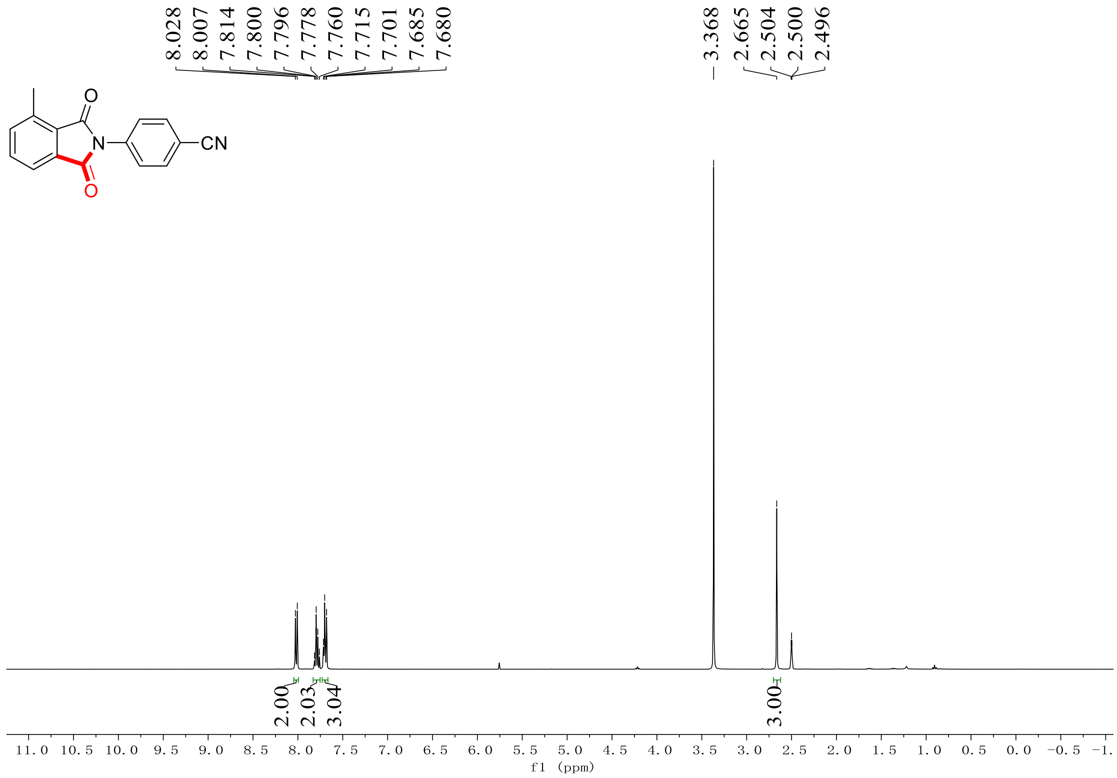


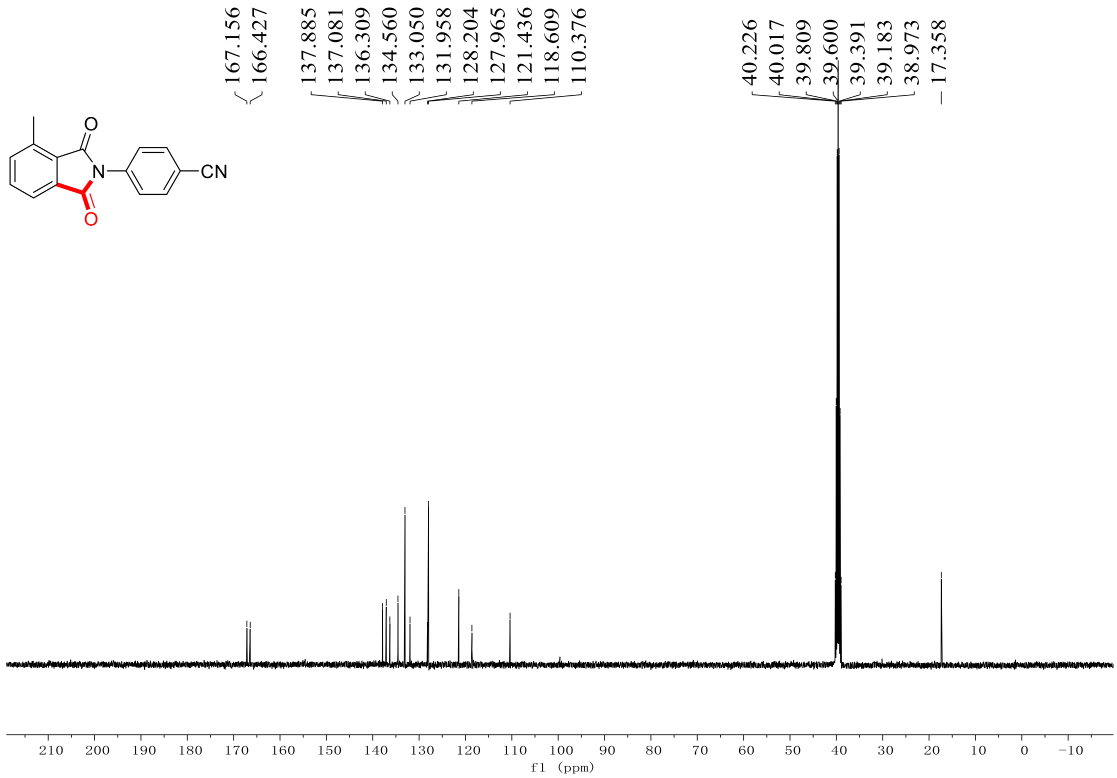


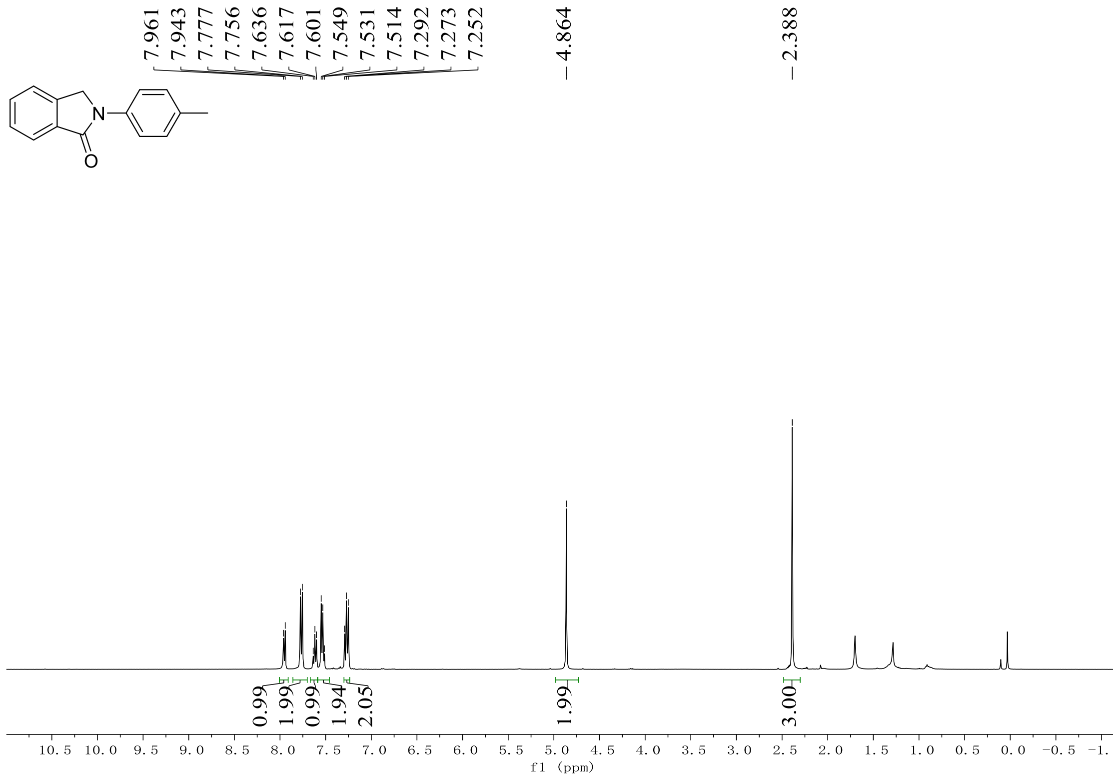


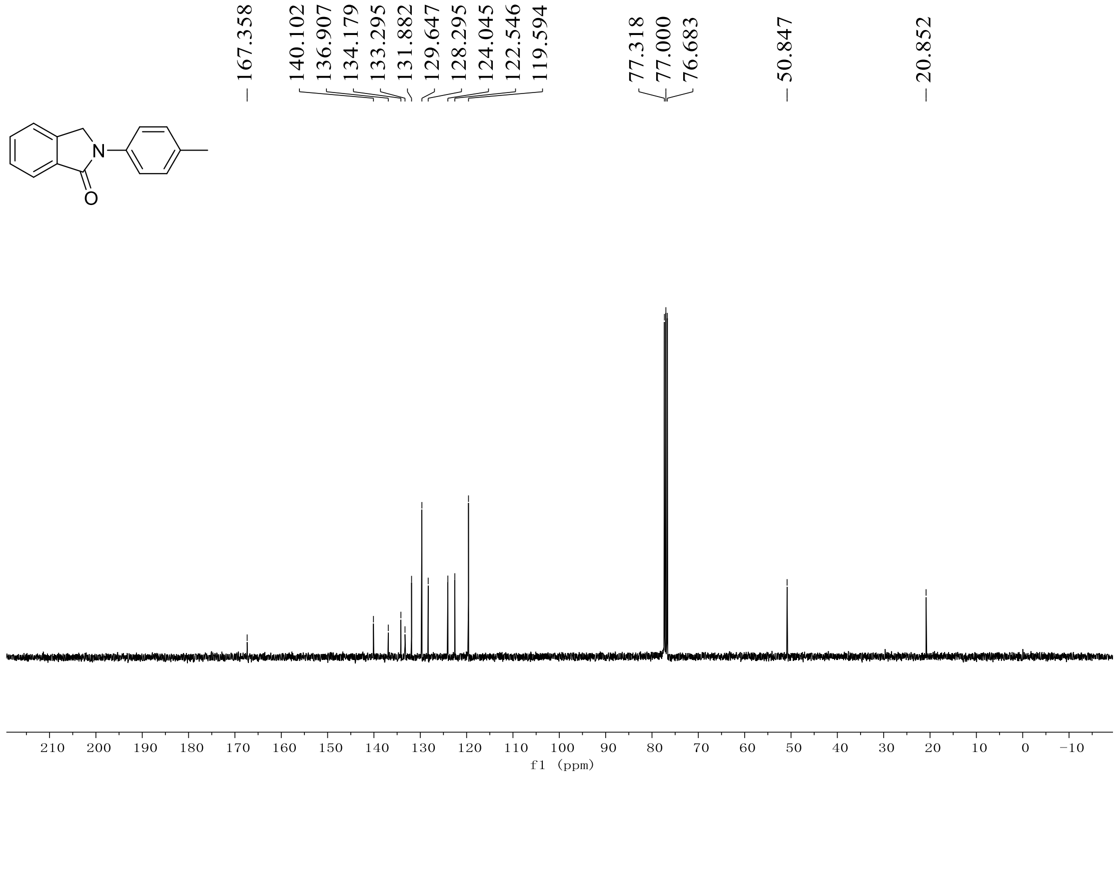

O
N
O
7.779
7.772
7.766
7.758
7.748
7.702
7.693
7.685
7.679
7.671
2.534
2.528
2.183
2.146
2.120
2.069
1.826
1.797
1.740
1.711
1.98
1.98
6.00
3.24
6.14
f1 (ppm)


O
N
O
169.791
133.580
131.910
122.464
77.317
77.000
76.682
60.337
40.147
36.142
29.741
f1 (ppm)


8.028
8.007
7.814
7.800
7.796
7.778
7.760
7.715
7.701
7.685
7.680
3.368
2.665
2.504
2.500
2.496
CN
N
O
O
2.00
2.03
3.04
3.00
11.0 10.5 10.0 9.5 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 −0.5 −1.
f1 (ppm)


167.156
166.427
137.885
137.081
136.309
134.560
133.050
131.958
128.204
127.965
121.436
118.609
110.376
40.226
40.017
39.809
39.600
39.391
39.183
38.973
17.358
CN
N
O
O
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)


7.961
7.943
7.777
7.756
7.636
7.617
7.601
7.549
7.531
7.514
7.292
7.273
7.252
4.864
2.388
N
O
0.99
1.99
0.99
1.94
2.05
1.99
3.00
f1 (ppm)


167.358
140.102
136.907
134.179
133.295
131.882
129.647
128.295
124.045
122.546
119.594
77.318
77.000
76.683
50.847
20.852
N
O
f1 (ppm)

10. Reference

[1] M. V. Khedkar, A. R. Shinde, T. Sasaki, B. M. Bhanage, *J. Mol. Catal. A: Chem.* **2014**, *385*, 91-97.

[2] S. Liu, Q. Deng, W. Fang, J.-F. Gong, M.-P. Song, M. Xu, T. Tu, *Org. Chem. Front.* **2014**, *1*, 1261-1265.

[3] H. Kim, T. Kim, D. G. Lee, S. W. Roh, C. Lee, *Chem. Commun.* **2014**, *50*, 9273-9276.

[4] M. Srinivas, A. D. Hudwekar, V. Venkateswarlu, G. L. Reddy, K. A. A. Kumar, R. A. Vishwakarma, S. D. Sawant, Tetrahedron Lett. 2015, 56, 4775-4779.

[5] R. J. Perry, S. R. Turner, *J. Org. Chem.* **1991**, *56*, 6573-6579.

[6] G. Gardner, J. E. Semple, *J. Plant Growth Regul.* **1990**, *9*, 161.

[7] Y. Fang, S.-Y. Wang, S.-J. Ji, *Tetrahedron* **2015**, *71*, 2768-2771.

[8] L. Wan, X. Sun, S. Shi, J. Zhang, X. Li, Z. Li, K. Guo, *Catal. Commun.* **2017**, *88*, 30-34.

[9] S. Mangani, L. Cancian, R. Leone, C. Pozzi, S. Lazzari, R. Luciani, S. Ferrari, M. P. Costi, *J. Med. Chem.* **2011**, *54*, 5454-5467.

[10] J. Wan, B. Wu, Y. Pan *Tetrahedron* **2007**, *63*, 9338–9344.

[11] M. Schlosser, H. C. Jung, S. Takagishi, *Tetrahedron* **1990**, *46*, 5633-5648.

[12] A. Demeter, T. Berces, L. Biczok, V. Wintgens, P. Valat, J. Kossanyi, *J. Chem. Soc., Faraday Trans.* **1994**, *90*, 2635-2641.

[13] Elwell, C. E.; Gagnon, N. L.; Neisen, B. D.; Dhar, D.; Spaeth, A. D.; Yee, G. M.; Tolman, W. B. *Chem. Rev.* **2017**, *117*, 2059.

[14] Lu, X. In *Handbook of Organopalladium Chemistry for Organic Synthesis*; John Wiley & Sons, Inc.: 2003, p 2267.