Electronic Supplementary Material (ESI) for Chemical Communications.

Supporting Information

Direct Synthesis of Phthalimides via Palladium-Catalyzed Double Carbonylation of *o*-Dihaloarenes with Nitroarenes

Chenwei Liu,[a] Tongshun An,[a] Weiheng Yuan,[a] Huiying Dai,[a] Xiaolan Liang,[a] Zhiping Yin,*[a]

[a] School of Pharmacy, Jiangsu University, Zhenjiang 212013, P. R. China,

E-mail: zhiping_yin@ujs.edu.cn

Contents

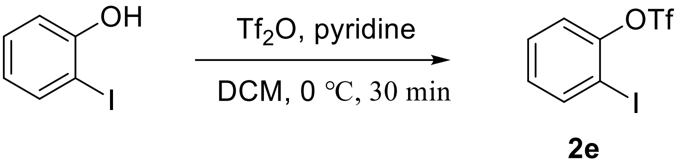
1. General Comments

Chemicals were purchased from Adamas, Bidepharm., TCI, Aladdin and used as such unless stated otherwise. All solvents like acetonitrile, tetrahydrofuran, *N*, *N*-dimethylmethanamide, 1,4-dioxane were purchased from Adamas (Water ≤ 30 ppm (by K.F.), 99.9%, SafeDry, with molecular sieves, Safeseal). NMR spectra were recorded on Bruker AV 400 or Bruker Fourier 300 spectrometer. Chemical shifts (ppm) are given relative to TMS (0.00 ppm) for ^{1}H and $CDCl_3$ (77.0 ppm) for ^{13}C solvent. Multiplets were assigned as s (singlet), d (doublet), t (triplet), q (quartet), p (pentet), dd (doublet of doublet), m (multiplet) and br.s (broad singlet). Infrared spectra were obtained using KBr plates on a Nicolet Avatar 370 DTGS. High-resolution mass spectra HRMS spectra were recorded on a Thermo Scientific Exactive Orbitrap Mass Spectrometer under Electron Spray Ionization conditions preparing sample solution in methanol. The data are given as mass units per charge (m/z). GC yields were calculated using hexadecane as an internal standard. Gas chromatography analysis was performed on an Agilent 6820 instrument with an FID detector and HP-5 capillary column (polydimethylsiloxane with 5% phenyl groups, 30 m, 0.32 mm i.d. 0.25 μm film thickness) using nitrogen as carrier gas. The products were isolated from the reaction mixture by column chromatography on silica gel., 54-74 μm, 200-300 mesh (Yucheng Chemical CO., LTD, Shanghai).

NOTE: a) As carbon monoxide will be released from $Mo(CO)_6$, the reactions should only be handled in a well-ventilated fume hood and the laboratory should be well-equipped with a CO detector and alarm system; b) The reaction was conducted under reflux conditions (temperature is higher than the boiling point of 1,4-dioxane).

2. Experimental Setup

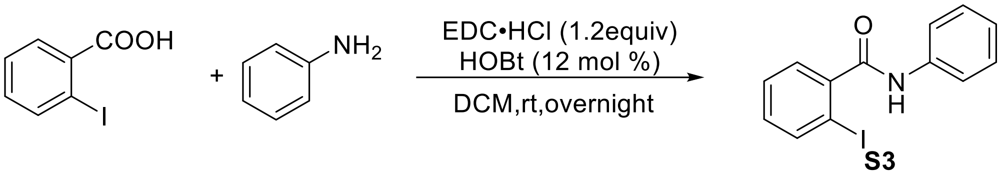
2.1.1 Preparation of 2-iodophenyl trifluoromethanesulfonate.[1]



2e

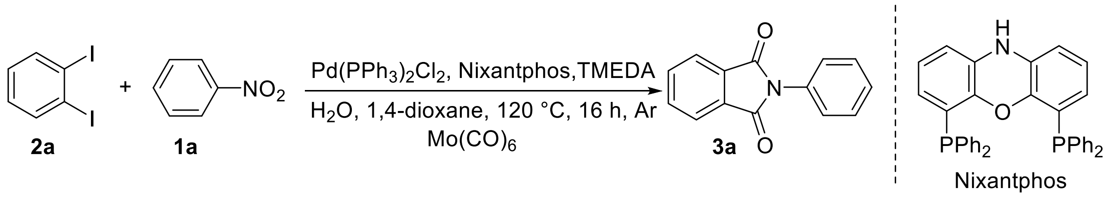
To the 2-iodophenol (1 mmol) in anhyd. CH_2Cl_2 was added pyridine (2 mmol) and the solution was cooled to 0 °C. Trifluoromethanesulfonic anhydride (1.2 mmol) was added dropwise and the mixture was warmed to r.t. The reaction was complete within 5 min as shown by TLC. The mixture was diluted with Et_2O, quenched with 10% aq HCl and washed successively with sat. $NaHCO_3$, and brine. After drying ($MgSO_4$) the solvent was evaporated and the residue was purified by column chromatography on silica gel using petroleum ether or mixtures of petroleum ether and Et_2O as eluent to give the triflates product **2e** as colorless to pale yellow clear liquids in 90% yield.

2.1.2 Preparation of 2-iodo-*N*-phenylbenzamide.[2]



Into an oven-dried 50 mL round-bottom flask equipped with a magnetic stirring bar and a rubber stopper were added EDC•HCl (1.40 g, 6.0 mmol, 1.2 equiv), HOBt (0.097 g, 0.72 mmol, 0.12 equiv), 2-iodobenzoic acid (6.0 mmol) and DCM (30 mL). Primary amine (7.2 mmol, 1.2 equiv) was then added into the flask, and the mixture was stirred at room temperature overnight. After the reaction finished, the mixture was diluted with DCM, washed successively with water, aqueous HCl solution (1.0 M), saturated aqueous $NaHCO_3$ solution and brine. The organic phases were dried over anhydrous $MgSO_4$, and then concentrated under reduced pressure on a rotary evaporator. The residual was purified by column chromatography on silica gel (eluent: PE and EA) to afford the pure product **S3**.

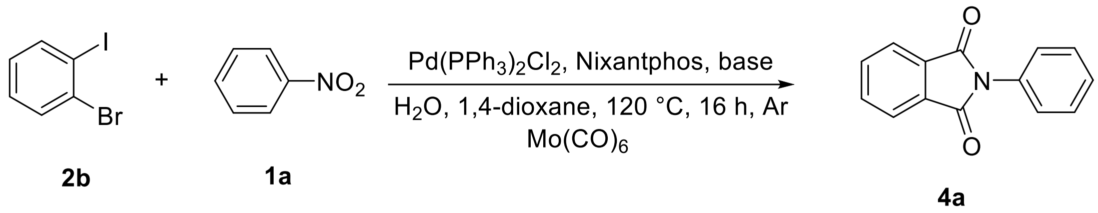
2.2.1 Solvents optimization for carbonylation of *o*-diiodobenzene with nitrobenzene



entry	solvent	yield (%)
1	CH_3CN	50
2	THF	trace
3	DCE	11
4	toluene	27
5	$PhCF_3$	31

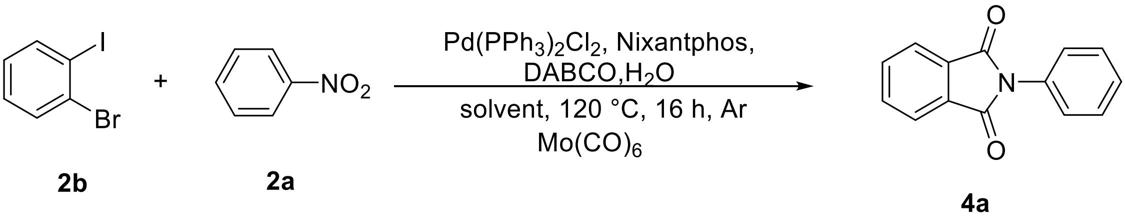
(Reaction conditions: **1a** (0.3 mmol, 1.5 equivalent), **2a** (0.2 mmol), $Pd(PPh_3)_2Cl_2$ (7.0 mg, 5 mol%), Nixantphos (11.0 mg, 10 mol%), $Mo(CO)_6$ (79.2 mg, 0.3 mmol, 1.5 equivalent), TMEDA (4.0 equivalent, 0.8 mmol, 120 µL), and H_2O (18 µL 1.0 mmol 5.0 equivalent), and solvent (2 mL), 120 °C, under Ar, 16 h, GC yields, using hexadecane as the internal standard.)

2.2.2 Bases and solvents optimization for carbonylation of 1-bromo-2-iodobenzene with nitrobenzene [a]



entry	base	yield (%)
1	TMEDA 4.0eq	Trace
2	K_2CO_3 4.0eq	Trace
3	DBU 4.0eq	31
4	DABCO 4.0eq	47

(Reaction conditions: **1a** (0.3 mmol, 1.5 equivalent), **2a** (0.2 mmol), $Pd(PPh_3)_2Cl_2$ (7.0 mg, 5 mol%), Nixantphos (11.0 mg, 10 mol%), $Mo(CO)_6$ (79.2 mg, 0.3 mmol, 1.5 equivalent), and H_2O (18 µL 1.0 mmol 5.0 equivalent),and dioxane (2 mL), 120 °C, under Ar, 16 h, GC yields, using hexadecane as the internal standard.)

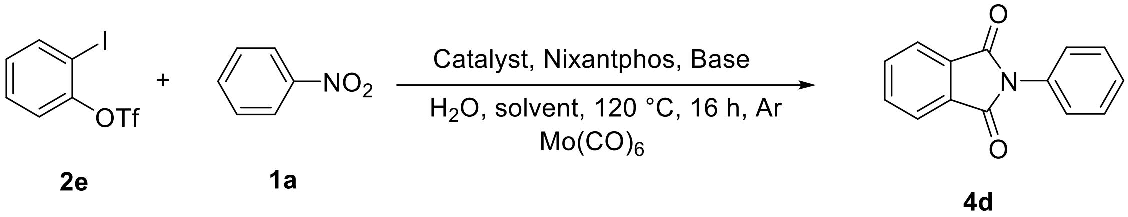


entry	solvent	yield (%)
1	1,4-dioxane	40
2	m-xylene	48
3	DMF	31

4	CH_3CN	55 (50)

([a] Reaction conditions: **1a** (0.3 mmol, 1.5 equivalent), **2a** (0.2 mmol), $Pd(PPh_3)_2Cl_2$ (7.0 mg, 5 mol%), Nixantphos (11.0 mg, 10 mol%), $Mo(CO)_6$ (79.2 mg, 0.3 mmol, 1.5 equivalent), DABCO (4.0 equivalent, 0.8 mmol, 120 µL), and H_2O (18 µL 1.0 mmol 5.0 equivalent), and solvent (2 mL), 120 °C, under Ar, 16 h, GC yield, using hexadecane as the internal standard. [b] isolated yield.)

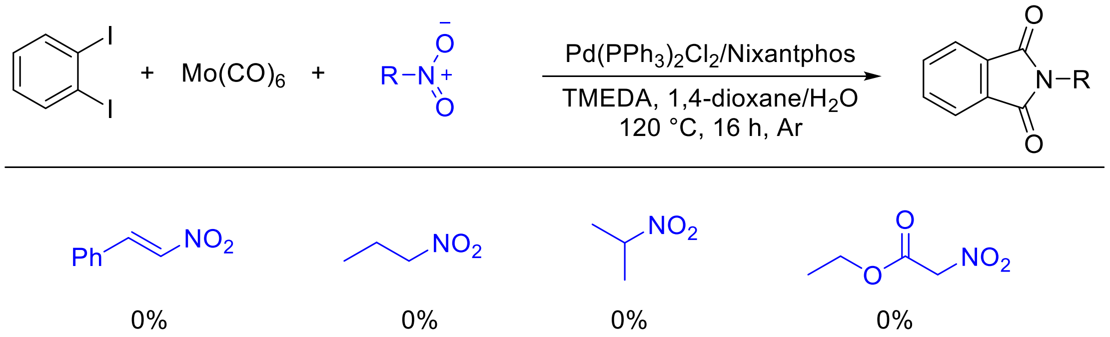
2.2.3 Reaction conditions optimization for carbonylation of 2-iodophenyl trifluoromethanesulfonate with nitrobenzene [a]



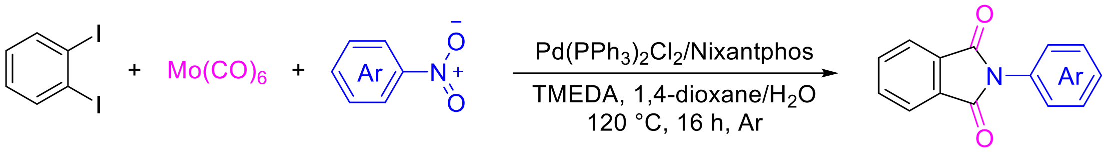
entry	catalyst	base	solvent	yield (%)
1	$Pd(PPh_3)_2Cl_2$ 5%	DABCO 4.0 eq	1,4-dioxane	Trace
2	$Pd(PPh_3)_2Cl_2$ 5%	Et_3N 4.0 eq	1,4-dioxane	Trace
3	$Pd(PPh_3)_2Cl_2$ 5%	TMEDA 4.0 eq	1,4-dioxane	Trace
4	$Pd(PPh_3)_2Cl_2$ 5%	DIPEA 4.0 eq	1,4-dioxane	18%
5	$Pd(PPh_3)_2Cl_2$ 5%	DIPEA 4.0eq	PhMe	Trace
6	$Pd(PPh_3)_2Cl_2$ 5%	DIPEA 4.0eq	DMF	Trace
7	$Pd(PPh_3)_2Cl_2$ 5%	DIPEA 4.0eq	CH_3CN	28
8	$Pd(TFA)_2$ 5%	DIPEA 4.0eq	CH_3CN	35
9	$Pd(PPh_3)_2Cl_2$ 5%	DIPEA 4.0eq	CH_3CN	50 (44[b])

([a] Reaction conditions: **1a** (0.3 mmol, 1.5 equivalent), **2a** (0.2 mmol), $Pd(PPh_3)_2Cl_2$ (7.0 mg, 5 mol%), Nixantphos (11.0 mg, 10 mol%), $Mo(CO)_6$ (79.2 mg, 0.3 mmol, 1.5 equivalent), and H_2O (18 µL 1.0 mmol 5.0 equivalent), and solvent (2 mL), 120 °C, under Ar, 16 h, GC yields, using hexadecane as the internal standard. [b] isolated yield)

2.3 Failed examples

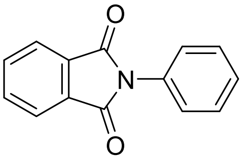


2.4 General Procedures for the palladium-catalyzed carbonylation of *o*-dihaloarenes with nitroarenes:



A flame-dried resealable Schlenk tube (10 mL) was added with aromatic nitro compounds **1a-1s** (0.2 mmol), $Mo(CO)_6$ (79.2 mg, 1.5 equivalent, 0.3 mmol), $Pd(PPh_3)_2Cl_2$ (7.0 mg, 5 mol%), Nixantphos (10 mol%, 11.0 mg). The Schlenk tube was capped with a rubber septum, evacuated, and backfilled with argon three times. The liquid 1,2-diiodobenzene (0.3 mmol,1.5 eq, 40 μL), TMEDA (4.0 equivalent, 0.8 mmol, 120 μL), H_2O (5.0 equivalent, 18 μL), and solvent 1,4-dioxane (2 mL) were added through the septum, then the septum was replaced with a Teflon screwcap quickly. The Schlenk tube was put into an aluminum heating block and stirred at 120 °C for 16 hours. After the reaction was completed, the reaction mixture was cooled to room temperature, diluted with ethyl acetate, and concentrated in vacuo. The crude material was purified by column chromatography on silica gel (eluent: PE and EA) to give the target product **3a-3s**.

3. Analytical Data

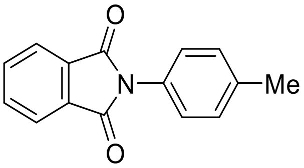


2-Phenylisoindoline-1,3-dione: (3a, 40.6 mg, white solid, yield: 91%**)**; mp = 209-210 °C; Rf =0.51 (petroleum ether / ethyl acetate = 5:1)

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.95 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.79 (dd, *J* = 5.5, 3.0 Hz, 2H), 7.63 – 7.34 (m, 5H).

^{13}C NMR (101 MHz, CDCl$_3$) δ 167.30, 134.42, 131.79, 131.71, 129.14, 128.13, 126.60, 123.77.

The analytical data are consistent with those reported in the literature.[3, 11]

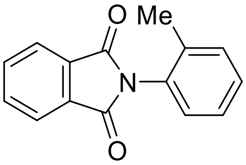


2-(*p*-Tolyl)isoindoline-1,3-dione: (3b, 42.3 mg, white solid, yield: 89%**)**; mp = 205-206 °C; Rf =0.51 (petroleum ether / ethyl acetate = 5:1)

^{1}H NMR (400 MHz, CDCl$_3$).δ 7.96 (dd, J = 5.5, 3.1 Hz, 2H), 7.80 (dd, J = 5.4, 3.0 Hz, 2H), 7.46 – 7.29 (m, 3H), 7.21 (d, J = 7.7 Hz, 1H), 2.22 (s, 3H).

^{13}C NMR (101 MHz, CDCl$_3$) δ 167.40, 138.14, 134.27, 131.77, 129.73, 128.91, 126.42, 123.64, 21.17.

The analytical data are consistent with those reported in the literature.[3,11]

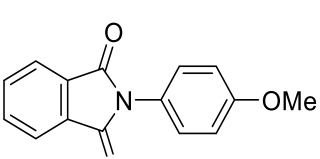


2-(*o*-Tolyl)isoindoline-1,3-dione (**3c**, 18 mg, white solid, yield: 38%); mp = 180-183 °C; Rf =0.51 (petroleum ether / ethyl acetate = 5:1)

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.88 (dd, J = 5.5, 3.1 Hz, 2H), 7.72 (dd, J = 5.4, 3.0 Hz, 2H), 7.34 – 7.10 (m, 4H), 2.14 (s, 3H).

^{13}C NMR (101 MHz, CDCl$_3$) δ 167.33, 136.51, 134.30, 131.99, 131.13, 130.54, 129.43, 128.69, 126.86, 123.74, 18.02.

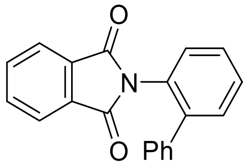
The analytical data are consistent with those reported in the literature.[3,12]



2-(4-Methoxyphenyl)isoindoline-1,3-dione (**3d,** 46.6 mg, white solid, yield: 92%**)**; mp = 160-161 °C; Rf =0.42 (petroleum ether / ethyl acetate = 3:1)

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.94 (dd, J = 5.4, 3.1 Hz, 2H), 7.78 (dd, J = 5.5, 3.0 Hz, 2H), 7.33 (d, J = 9.0 Hz, 2H), 7.02 (d, J = 9.0 Hz, 2H), 3.85 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 167.48, 159.15, 134.23, 131.71, 127.86, 124.17, 123.56, 114.37, 55.42. The analytical data are consistent with those reported in the literature.[3,11]

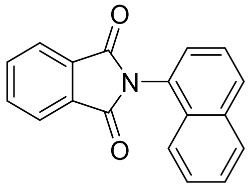


2-([1,1'-Biphenyl]-3-yl)isoindoline-1,3-dione (**3e,** 38.9 mg, white solid, yield: 65%); mp = 165-166 °C; Rf =0.32 (petroleum ether / ethyl acetate = 5:1)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.81 (dd, J = 5.5, 3.1 Hz, 2H), 7.70 (dd, J = 5.5, 3.1 Hz, 2H), 7.58 – 7.46 (m, 3H), 7.38 – 7.31 (m, 1H), 7.30 – 7.21 (m, 5H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 167.45, 141.64, 138.82, 134.12, 131.71, 130.92, 129.61, 129.34, 128.45, 128.32, 128.07, 127.43, 123.62.

The analytical data are consistent with those reported in the literature.[7]

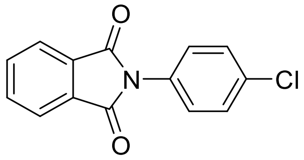


2-(Naphthalen-1-yl)isoindoline-1,3-dione (**3f,** 30 mg, white solid, yield: 40%**)**; mp = 182-185 °C; Rf = 0.28 (petroleum ether / ethyl acetate = 5:1)

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.06 – 7.97 (m, 3H), 7.95 (d, J = 7.7 Hz, 1H), 7.85 (dd, J = 5.4, 3.1 Hz, 2H), 7.66 – 7.58 (m, 2H), 7.57 – 7.44 (m, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 167.80, 134.48, 134.42, 131.98, 130.24, 129.95, 128.58, 128.12, 127.12, 126.97, 126.54, 125.40, 123.94, 122.40.

The analytical data are consistent with those reported in the literature.[6,13]

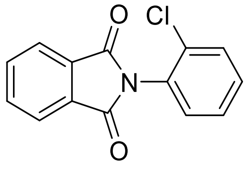


2-(4-Chlorophenyl)isoindoline-1,3-dione (**3g,** 48.5 mg, white solid, yield: 94%**)**; mp = 182-183 °C; Rf =0.51 (petroleum ether / ethyl acetate = 5:1)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96 (dd, J = 5.5, 3.1 Hz, 2H), 7.80 (dd, J = 5.5, 3.1 Hz, 2H), 7.48 (d, J = 8.9 Hz, 2H), 7.41 (d, J = 8.8 Hz, 2H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 166.92, 134.51, 133.72, 131.53, 130.15, 129.24, 127.61, 123.79.

The analytical data are consistent with those reported in the literature.[3,11]

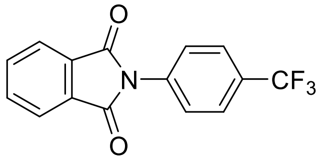


2-(2-Chlorophenyl)isoindoline-1,3-dione (**3h,** 25.2 mg, white solid, yield: 49%); mp = 143-145 °C; Rf =0.51 (petroleum ether / ethyl acetate = 5:1)

^{1}H NMR (400 MHz,CDCl$_3$) δ 7.97 (dd, J = 5.4, 3.1 Hz, 2H), 7.81 (dd, J = 5.5, 3.1 Hz, 2H), 7.64 – 7.52 (m, 1H), 7.46 – 7.40 (m, 2H), 7.39 – 7.32 (m, 1H).

^{13}C NMR (101 MHz, CDCl$_3$) δ 166.65, 134.49, 133.25, 131.93, 130.68, 130.45, 129.65, 127.74, 123.98.

The analytical data are consistent with those reported in the literature.[3,14]



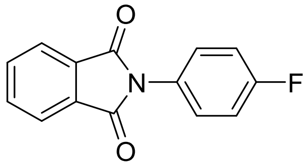
2-(4-(trifluoromethyl)phenyl)isoindoline-1,3-dione (**3i,** 44.2 mg, white solid, yield: 76%); mp = 257-259 °C; Rf =0.53 (petroleum ether / ethyl acetate = 5:1)

^{1}H NMR (400 MHz, Chloroform-d) δ 7.99 (dd, J = 5.5, 3.1 Hz, 2H), 7.84 (q, J = 3.2, 2.4 Hz, 2H), 7.78 (d, J = 8.4 Hz, 2H), 7.65 (d, J = 8.3 Hz, 2H).

^{13}C NMR (101 MHz, CDCl$_3$) δ 166.76, 134.75, 131.48, 129.96, 129.63, 127.82 (q, J_{C-F} = 272.7 Hz), 126.43, 126.21 (q, J_{C-F} = 4.04 Hz), 124.01.

^{19}F NMR (376 MHz, CDCl$_3$) δ -62.59.

The analytical data are consistent with those reported in the literature.[3,15]



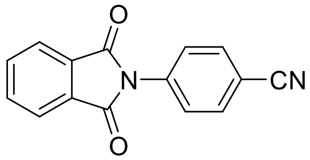
2-(4-Fluorophenyl)isoindoline-1,3-dione (**3j,** 34.7 mg, white solid, yield: 72%); mp = 180-191 °C; Rf =0.55 (petroleum ether / ethyl acetate = 5:1)

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.89 (dd, J = 5.7, 3.2 Hz, 2H), 7.83 – 7.69 (m, 2H), 7.52 – 7.27 (m, 2H), 7.25 – 7.03 (m, 2H).

^{13}C NMR (101 MHz, CDCl$_3$) δ 167.21, 161.92 (d, J_{C-F} = 249.47 Hz), 134.51, 131.59, 128.36 (d, J_{C-F} = 8.08 Hz), 127.51 (d, J_{C-F} = 3.03 Hz), 123.81, 116.14 (d, J_{C-F} =23.23 Hz).

^{19}F NMR (376 MHz, CDCl$_3$) δ -113.01.

The analytical data are consistent with those reported in the literature.[3,16]

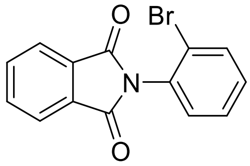


4-(1,3-Dioxoisoindolin-2-yl)benzonitrile (**3k,** 40.7 mg, white solid, yield: 82%); mp = 184-186 °C; Rf =0.31 (petroleum ether / ethyl acetate = 5:1)

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.99 (dd, J = 5.5, 3.0 Hz, 2H), 7.97 – 7.76 (m, 4H), 7.69 (dd, J = 8.7, 1.9 Hz, 2H).

^{13}C NMR (101 MHz, CDCl$_3$) δ 166.45, 134.92, 132.93, 131.34, 126.45, 124.10, 118.23, 111.30.

The analytical data are consistent with those reported in the literature.[3,17]

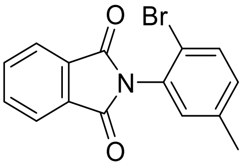


2-(2-Bromophenyl)isoindoline-1,3-dione (**3l,** 24.1 mg, white solid, yield: 40%); mp = 63-65 °C; Rf =0.51 (petroleum ether / ethyl acetate = 5:1)

^{1}H NMR (400 MHz,CDCl$_3$) δ 7.98 (td, J = 5.7, 2.7 Hz, 2H), 7.82 (dt, J = 5.4, 3.1 Hz, 2H), 7.79 – 7.71 (m, 1H), 7.47 (t, J = 7.4 Hz, 1H), 7.37 (dt, J = 9.1, 5.1 Hz, 2H).

^{13}C NMR (101 MHz, CDCl$_3$) δ 166.65, 134.52, 133.61, 131.91, 131.41, 130.97, 130.85, 128.47, 124.02, 123.37.

The analytical data are consistent with those reported in the literature.[4,18]



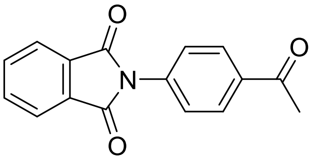
2-(2-Bromo-5-methylphenyl)isoindoline-1,3-dione (**3m,** 17 mg, white solid, yield: 27%); mp = 175-178 °C; Rf =0.51 (petroleum ether / ethyl acetate = 5:1)

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.97 (dd, J = 5.5, 3.1 Hz, 2H), 7.81 (dd, J = 5.5, 3.1 Hz, 2H), 7.65 – 7.57 (m, 1H), 7.21 – 7.11 (m, 2H), 2.37 (s, 3H).

^{13}C NMR (101 MHz, CDCl$_3$) δ 166.70, 138.80, 134.44, 133.12, 131.89, 131.85, 131.33, 130.96, 123.93, 119.74, 20.83.

IR (neat) cm^{-1} 2923, 1747, 1589, 1472, 1364, 1231, 1082, 1035, 872, 815, 717, 530.

HRMS: (ESI-TOF) Calc. for $C_{16}H_{11}Br^{79}NO_2$ [M+H]$^+$: 315.9968; found: 315.9962. $C_{16}H_{11}Br^{81}NO_2$ [M+H]$^+$: 317.9948; found: 317.9942.

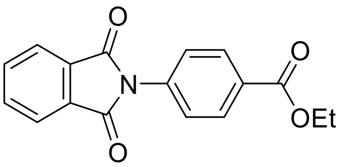


2-(4-Acetylphenyl)isoindoline-1,3-dione (**3n,** 31.3 mg, white solid, yield: 59%); mp = 245-247 °C; Rf =0.39 (petroleum ether / ethyl acetate = 5:1)

^{1}H NMR (400 MHz, CDCl$_3$) δ 8.17 – 8.06 (m, 2H), 7.98 (dd, J = 5.5, 3.1 Hz, 2H), 7.83 (dd, J = 5.5, 3.1 Hz, 2H), 7.67 – 7.56 (m, 2H), 2.65 (s, 3H).

^{13}C NMR (101 MHz, CDCl$_3$) δ 197.30, 166.78, 136.09, 135.99, 134.70, 131.61, 129.16, 126.09, 123.97, 26.65.

The analytical data are consistent with those reported in the literature.[9,18]

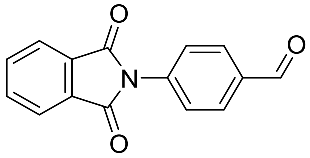


Ethyl 4-(1,3-dioxoisoindolin-2-yl)benzoate (**3o,** 39.5 mg, white solid, yield: 67%); mp = 130-132 °C; Rf = 0.8 (petroleum ether / ethyl acetate = 4:1)

^{1}H NMR (400 MHz, CDCl$_3$) δ 8.26 – 8.10 (m, 2H), 7.96 (dd, J = 5.5, 3.0 Hz, 2H), 7.80 (dd, J = 5.5, 3.1 Hz, 2H), 7.69 – 7.40 (m, 2H), 4.40 (q, J = 7.1 Hz, 2H), 1.40 (t, J = 7.1 Hz, 3H).

^{13}C NMR (101 MHz, CDCl$_3$) δ 166.76, 165.79, 135.70, 134.62, 131.53, 130.33, 129.62, 125.86, 123.87, 61.15, 14.28.

The analytical data are consistent with those reported in the literature.[3,19]

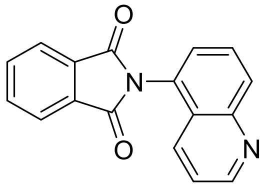


4-(1,3-dioxoisoindolin-2-yl)benzaldehyde (**3p,** 40.2 mg, white solid, yield: 80%); mp = 345-348 °C; Rf =0.31 (petroleum ether / ethyl acetate = 5:1)

^{1}H NMR (400 MHz, $(CD_3)_2SO$) δ 10.16 (s, 1H), 8.20 – 8.15 (m, 2H), 8.10 (dd, J = 5.5, 3.1 Hz, 2H), 8.03 (dd, J = 5.5, 3.1 Hz, 2H), 7.85 – 7.80 (m, 2H).

^{13}C NMR (101 MHz, $(CD_3)_2SO$) δ 192.90, 167.04, 137.60, 135.53, 135.37, 131.95, 130.42, 127.93, 124.07.

The analytical data are consistent with those reported in the literature.[10,20]

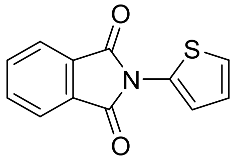


2-(Quinolin-5-yl)isoindoline-1,3-dione (**3q,** 32.9 mg, white solid, yield: 60%); mp = 206-208 °C; Rf =0.21 (petroleum ether / ethyl acetate = 5:1)

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.99 (s, 1H), 8.28 (d, J = 8.6 Hz, 1H), 8.09 – 7.95 (m, 3H), 7.94 – 7.81 (m, 3H), 7.55 (d, J = 7.4 Hz, 1H), 7.48 – 7.40 (m, 1H),.

^{13}C NMR (101 MHz, $CDCl_3$) δ 167.61, 150.89, 148.75, 134.78, 131.82, 131.35, 131.30, 129.00, 128.04, 127.50, 125.72, 124.14, 121.88.

The analytical data are consistent with those reported in the literature.[5]

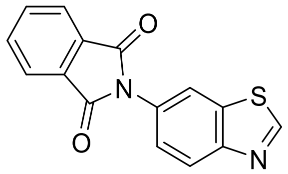


2-(Thiophen-2-yl)isoindoline-1,3-dione (**3r,** 20.6 mg, yellow solid, yield: 45%); mp = 196-198 °C; Rf =0.51 (petroleum ether / ethyl acetate = 5:1)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99 – 7.87 (m, 2H), 7.87 – 7.77 (m, 2H), 7.53 (dd, J = 3.9, 1.4 Hz, 1H), 7.22 (dd, J = 5.5, 1.3 Hz, 1H), 7.06 (dd, J = 5.5, 3.8 Hz, 1H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 13C NMR (101 MHz, CDCl3) δ 166.00, 134.66, 131.42, 125.28, 123.92, 123.72, 121.95, 120.44.

The analytical data are consistent with those reported in the literature.[4]

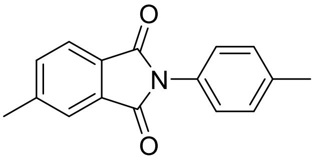


2-(Benzo[d]thiazol-6-yl)isoindoline-1,3-dione (**3s,** 44.6 mg, white solid, yield: 80%); mp = 227-228 °C; Rf =0.21 (petroleum ether / ethyl acetate = 5:1)

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.06 (s, 1H), 8.25 (d, J = 8.7 Hz, 1H), 8.07 (d, J = 2.0 Hz, 1H), 7.98 (dd, J = 5.4, 3.0 Hz, 2H), 7.81 (dd, J = 5.5, 3.1 Hz, 2H), 7.62 (dd, J = 8.7, 2.0 Hz, 1H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 167.19, 155.23, 152.52, 134.57, 134.20, 131.58, 129.15, 124.89, 123.92, 123.86, 120.15.

The analytical data are consistent with those reported in the literature.[6]

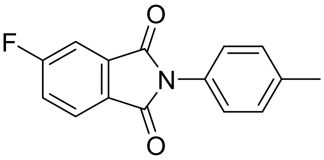


5-Methyl-2-(p-tolyl)isoindoline-1,3-dione (**4b,** 30 mg, white solid, yield: 60%**)**

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.78 – 7.71 (m, 1H), 7.65 (s, 1H), 7.48 (d, J = 7.2 Hz, 1H), 7.22 (d, J = 3.2 Hz, 4H), 2.45 (s, 3H), 2.32 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 167.55, 167.43, 145.59, 137.97, 134.82, 132.14, 129.67, 129.17, 129.05, 126.39, 124.11, 123.53, 21.99, 21.15.

The analytical data are consistent with those reported in the literature.[3]



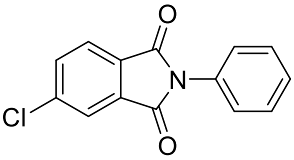
5-Fluoro-2-(p-tolyl)isoindoline-1,3-dione (**4f,** 44.9 mg, white solid, yield: 88%); mp = 180-182 °C; Rf =0.51 (petroleum ether / ethyl acetate = 5:1)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90 (dd, J = 8.2, 4.5 Hz, 1H), 7.63 – 7.52 (m, 1H), 7.44 – 7.36 (m, 1H), 7.31 – 7.21 (m, 4H), 2.37 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 166.50 (d, $J_{C\text{-}F}$ = 258.56 Hz), 166.31, 166.00 (d, $J_{C\text{-}F}$ = 2.02 Hz), 138.34, 134.60 (d, $J_{C\text{-}F}$ = 9.09 Hz), 129.78, 128.72, 127.52 (d, $J_{C\text{-}F}$ = 3.03 Hz), 126.30, 126.07 (d, $J_{C\text{-}F}$ = 9.09 Hz), 121.35 (d, $J_{C\text{-}F}$ = 23.23 Hz), 111.39 (d, $J_{C\text{-}F}$ = 25.25 Hz), 21.17.

^{19}F NMR (376 MHz, $CDCl_3$) δ -101.19.

The analytical data are consistent with those reported in the literature.[8]



5-Chloro-2-phenylisoindoline-1,3-dione (**4g,** 37 mg, white solid, yield: 72%); mp = 175-176 °C; Rf =0.72 (petroleum ether / ethyl acetate = 5:1)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99 – 7.85 (m, 2H), 7.75 (dd, J = 7.9, 1.9 Hz, 1H), 7.55 – 7.48 (m, 2H), 7.42 (d, J = 7.4 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 166.25, 165.94, 141.06, 134.43, 133.36, 131.35, 129.75, 129.13, 128.26, 126.41, 124.97, 124.11.

The analytical data are consistent with those reported in the literature.[4,16]

4. References

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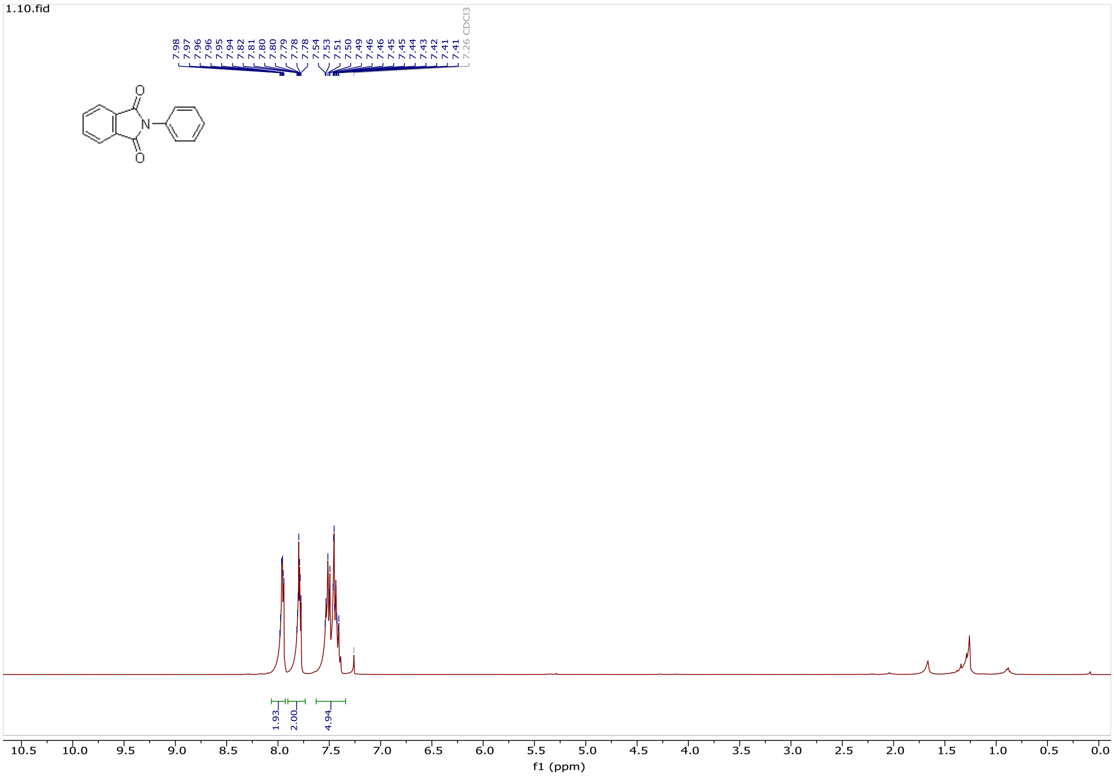
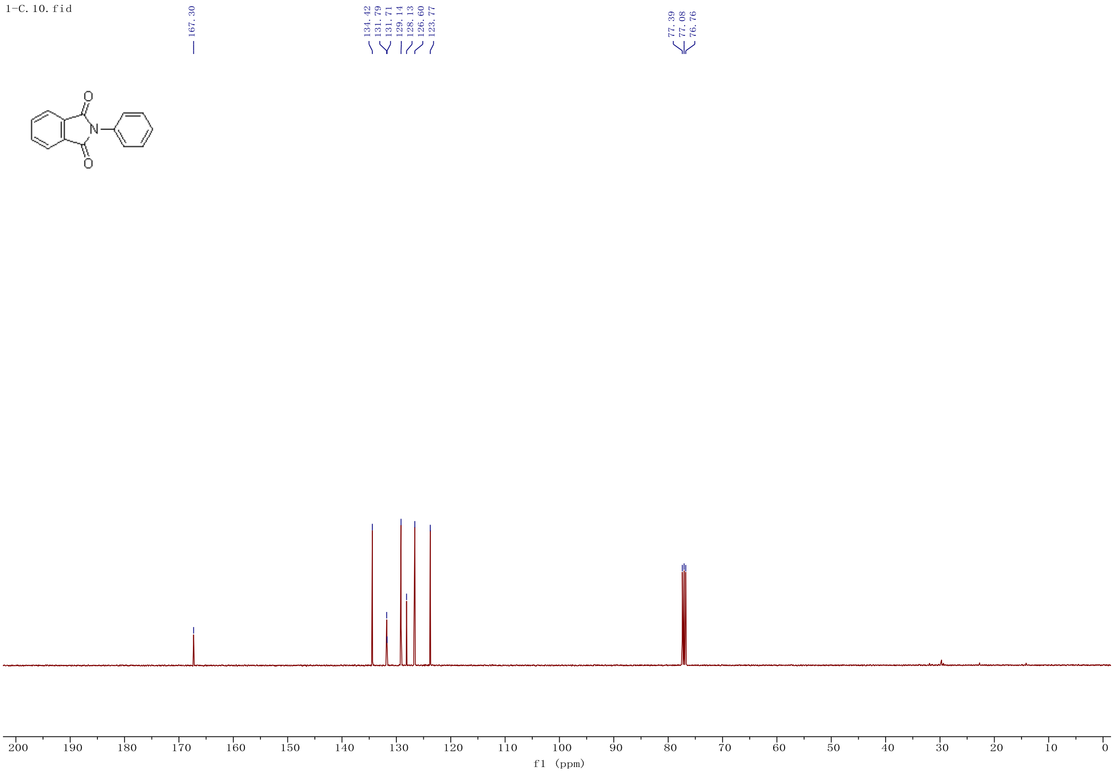
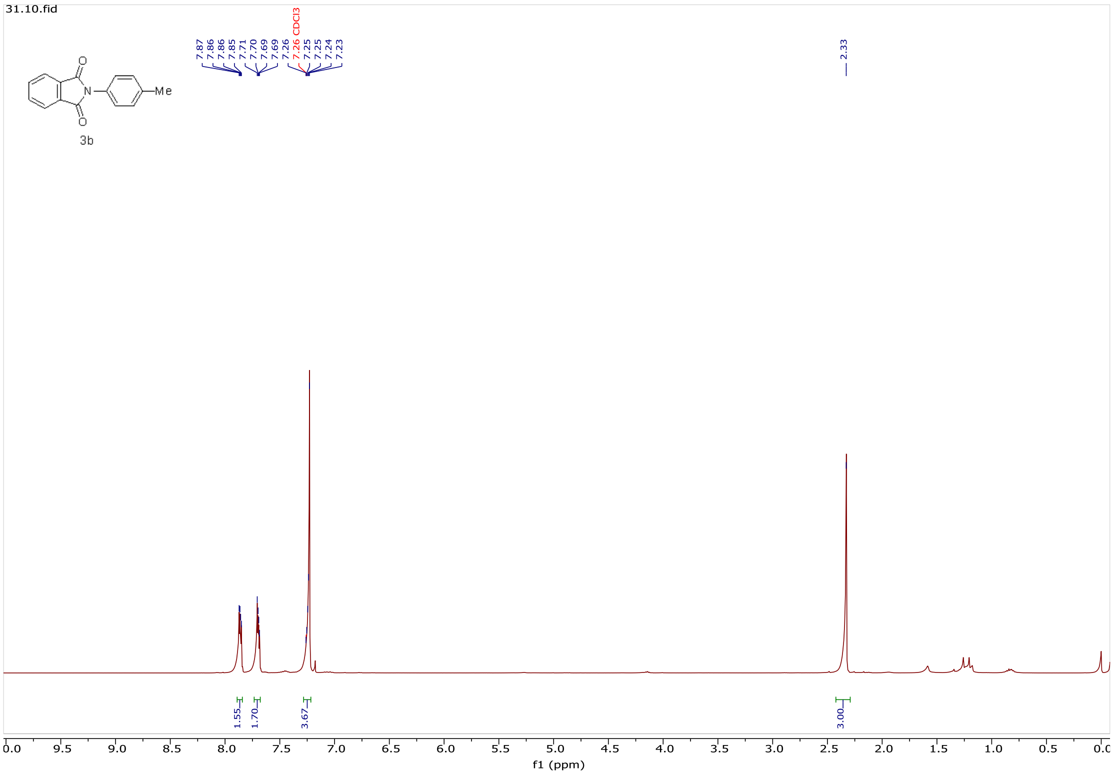
5. NMR Spectroscopic Data for Products



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7.69
7.26
7.26 CDCl3
7.25
7.25
7.24
7.23
2.33
O
N
O
Me
3b
1.55
1.70
3.67
3.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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3.5
3.0
2.5
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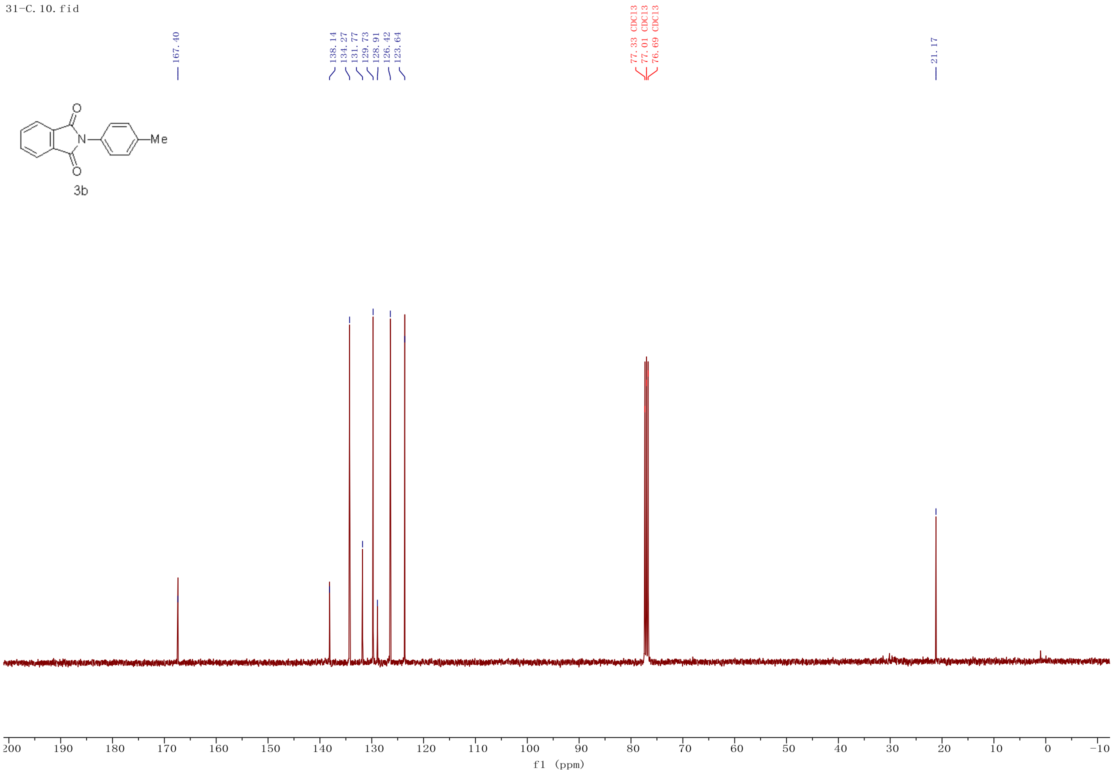
f1 (ppm)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77.01 CDCl3
76.69 CDCl3
21.17
O
N
O
Me
3b
200
190
180
170
160
150
140
130
120
110
100
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80
70
60
50
40
30
20
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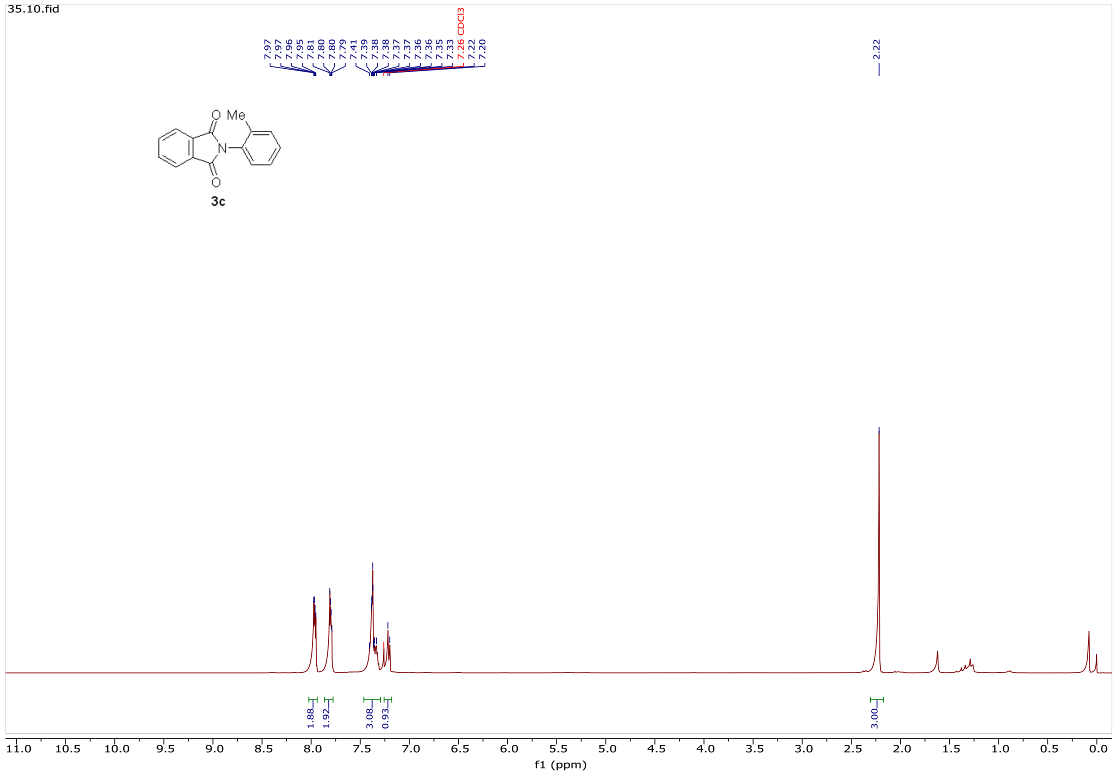
f1 (ppm)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7.37
7.36
7.36
7.35
7.33
7.26 CDCl3
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7.20
2.22
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Me
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O
3c
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7.5
7.0
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6.0
5.5
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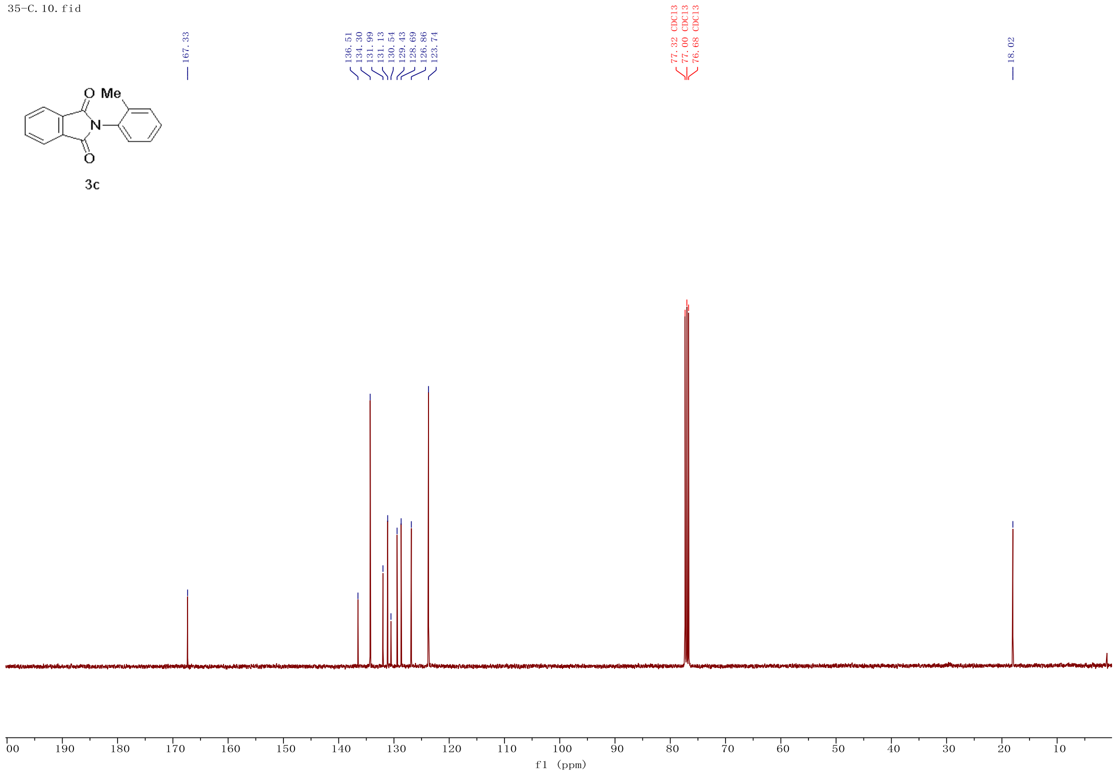
f1 (ppm)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O
3c
00
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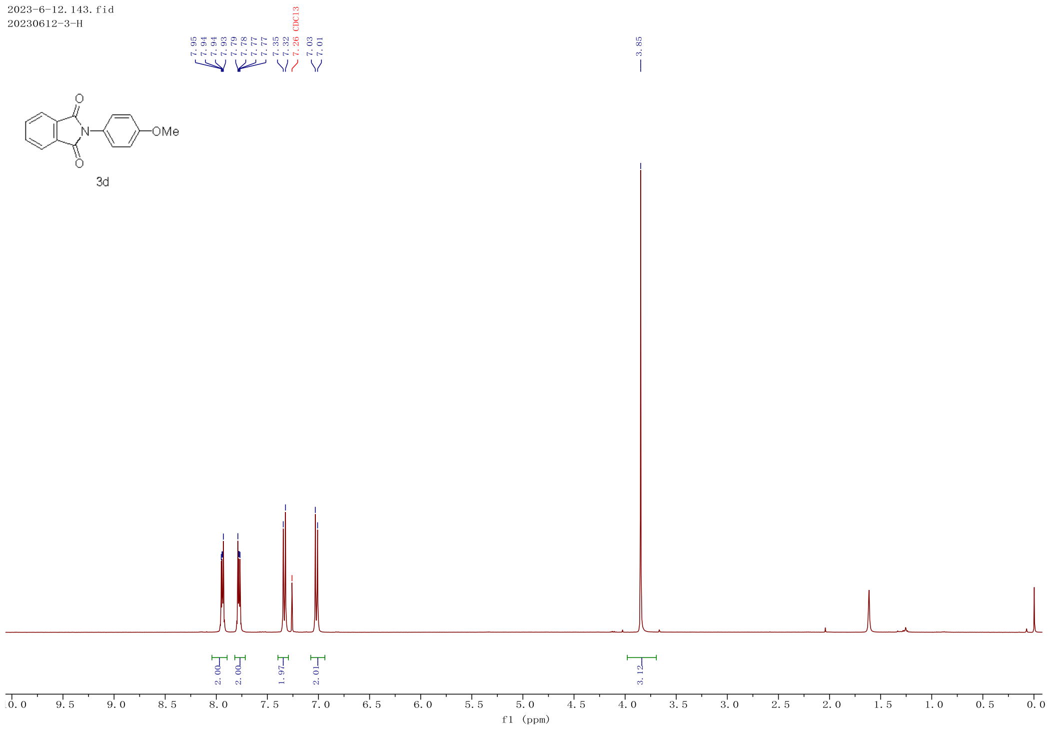
f1 (ppm)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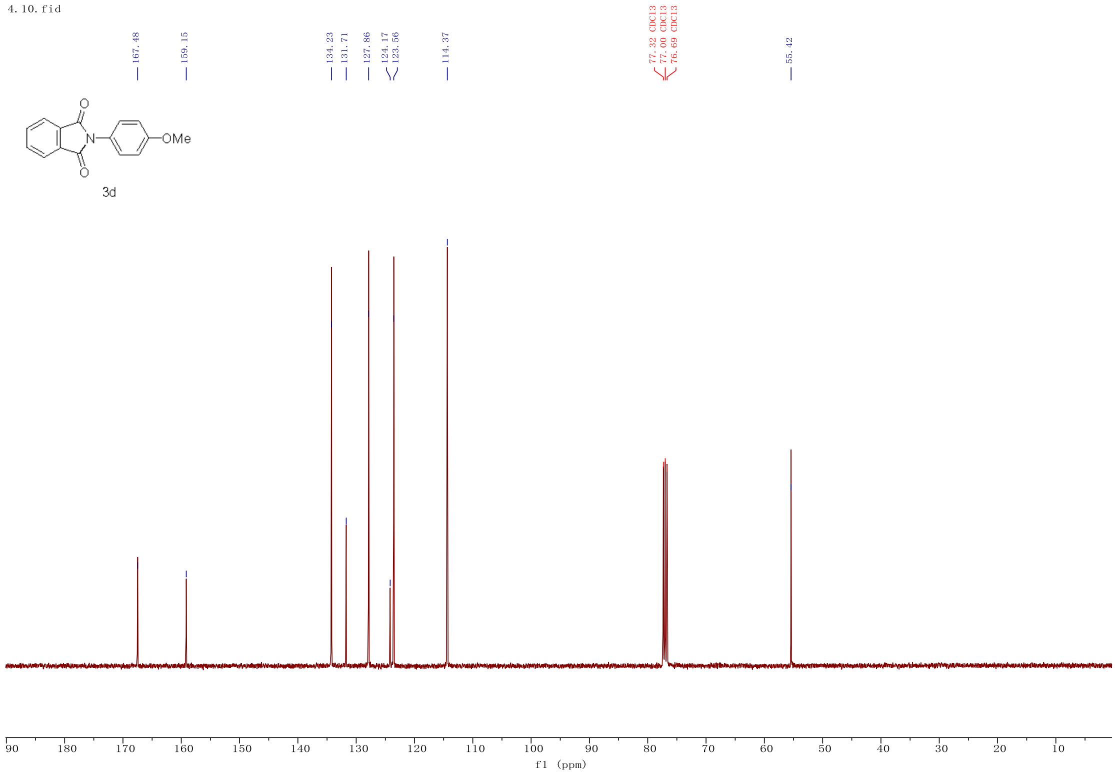
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OMe
O
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O
3d
190
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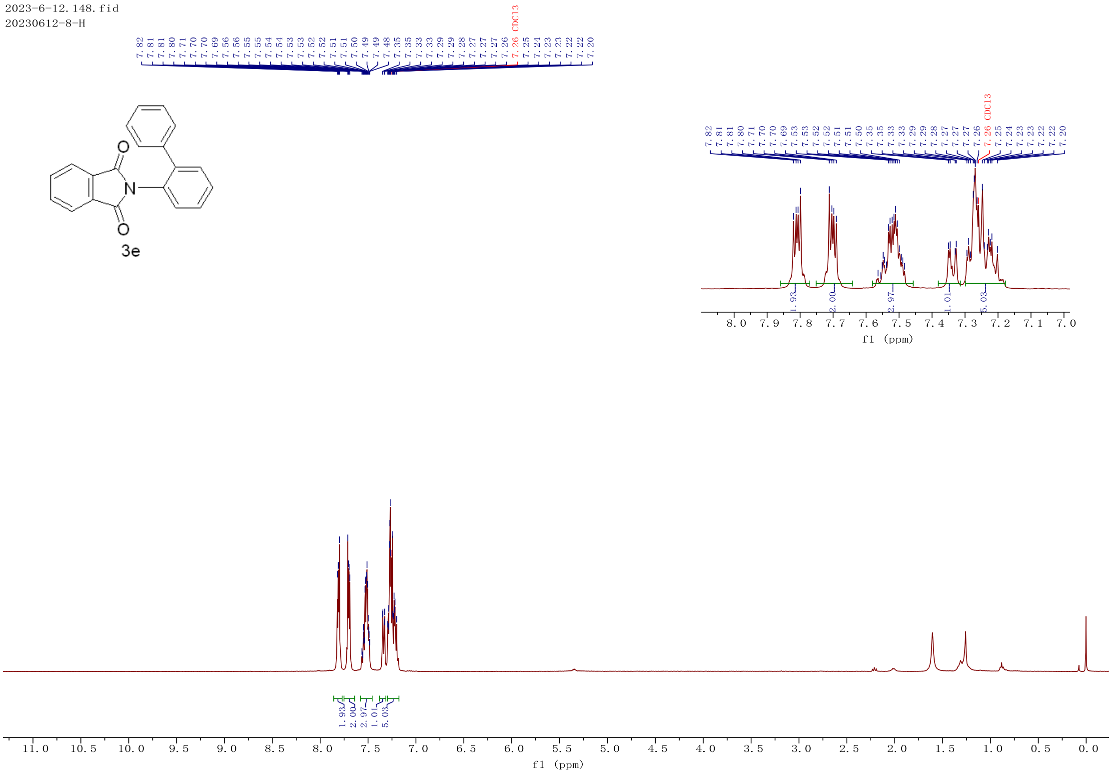
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O
N
O
3e
7.82 7.81 7.81 7.80 7.71 7.70 7.70 7.69 7.53 7.53 7.52 7.52 7.51 7.51 7.50 7.35 7.35 7.33 7.33 7.29 7.29 7.28 7.27 7.27 7.27 7.26 7.26 CDCl3 7.25 7.24 7.23 7.23 7.22 7.22 7.20
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2.00
2.97
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8.0 7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 7.0
f1 (ppm)
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2.00
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1.01
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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
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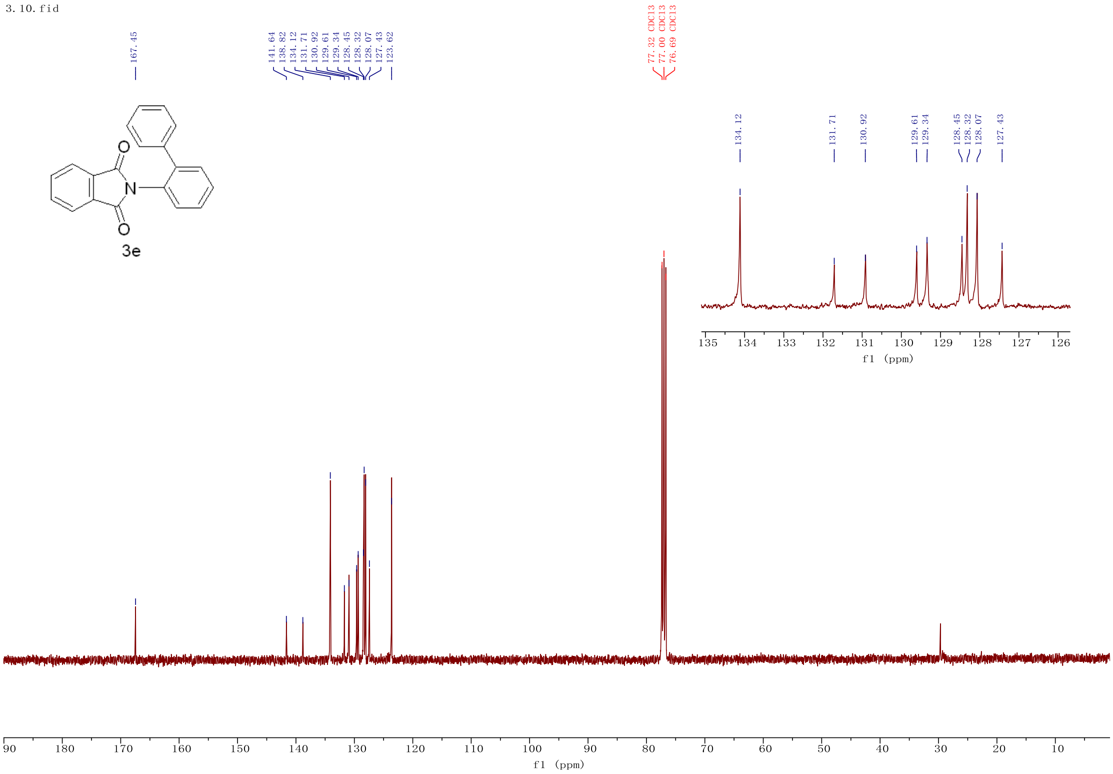
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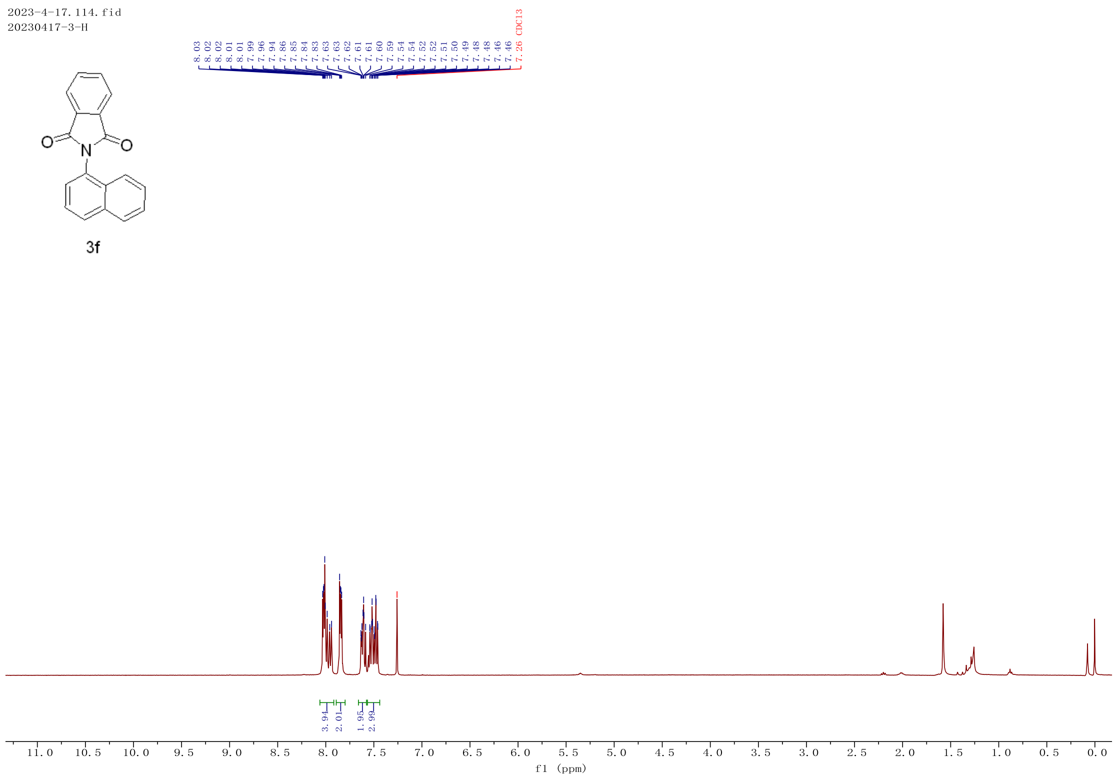
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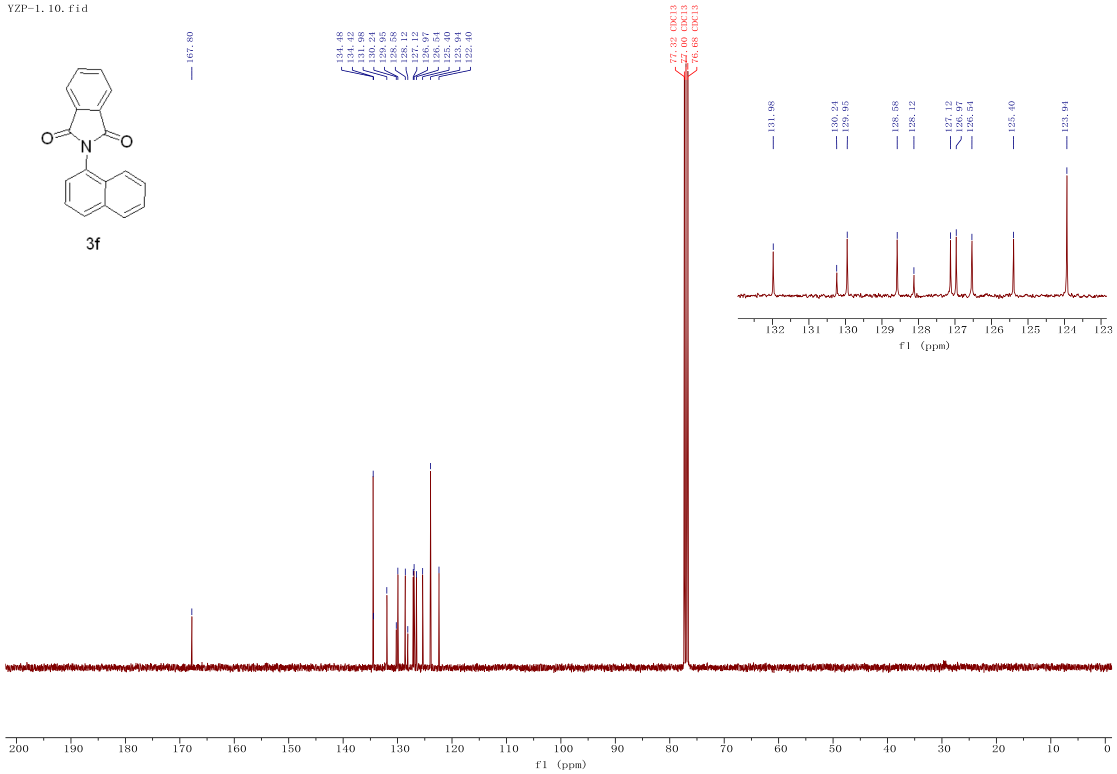
f1 (ppm)

3f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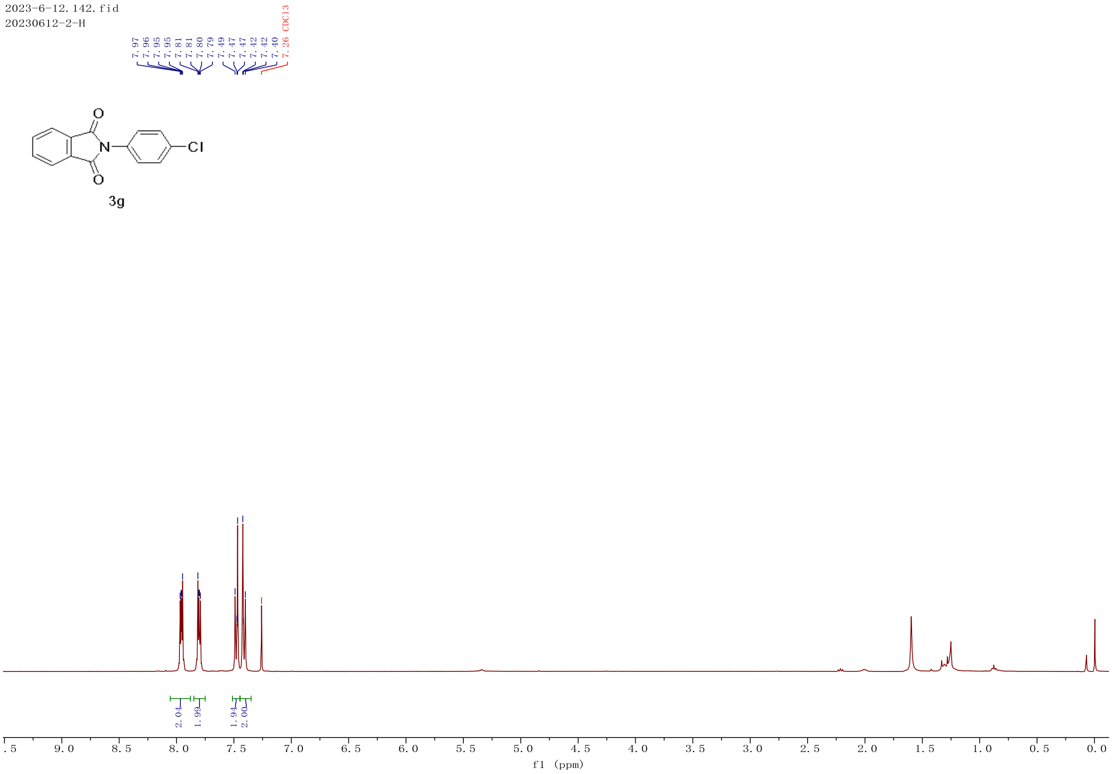
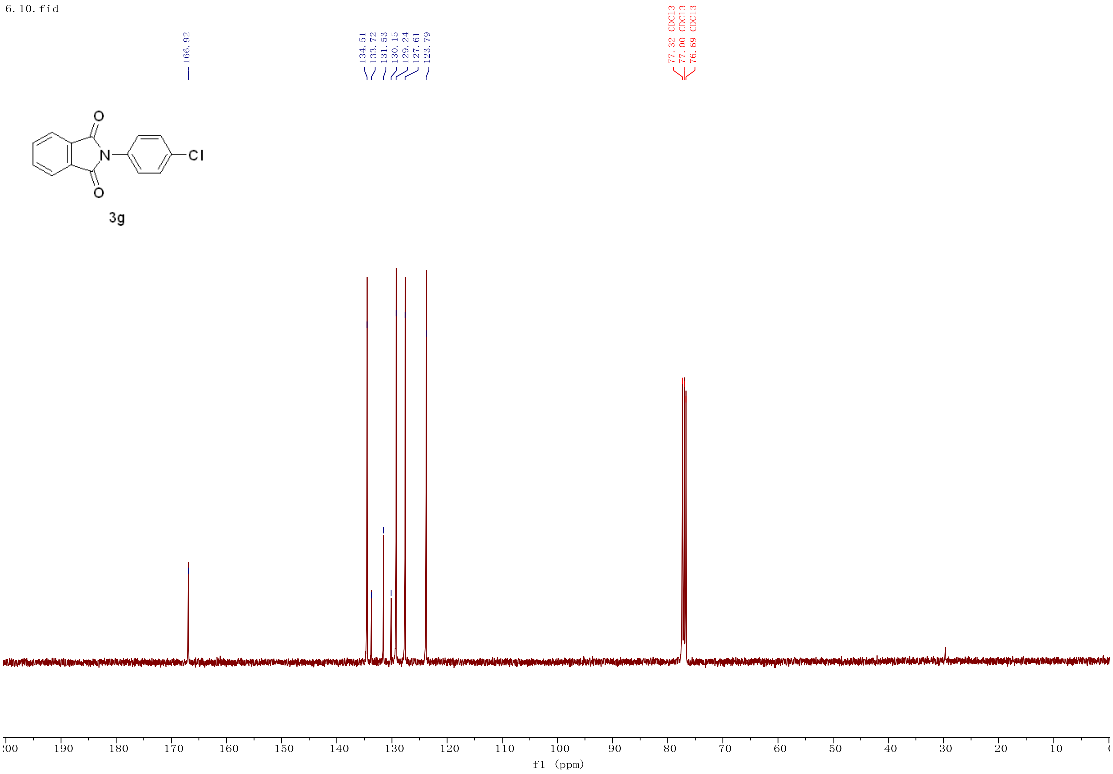
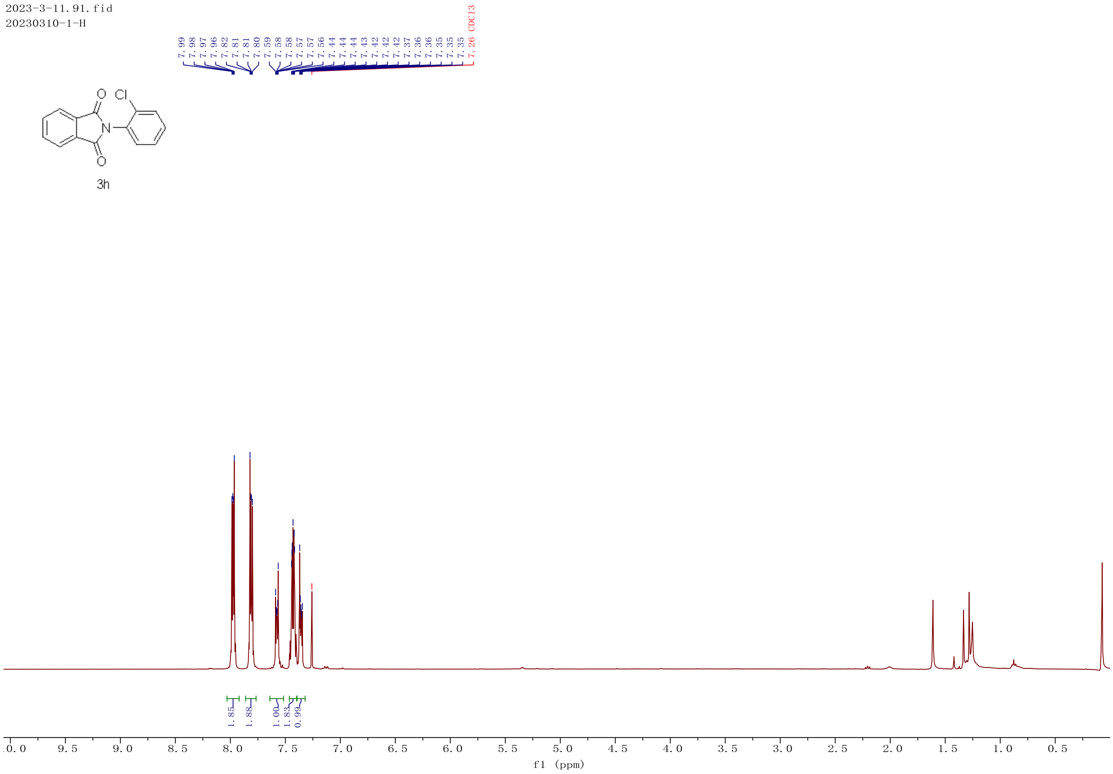
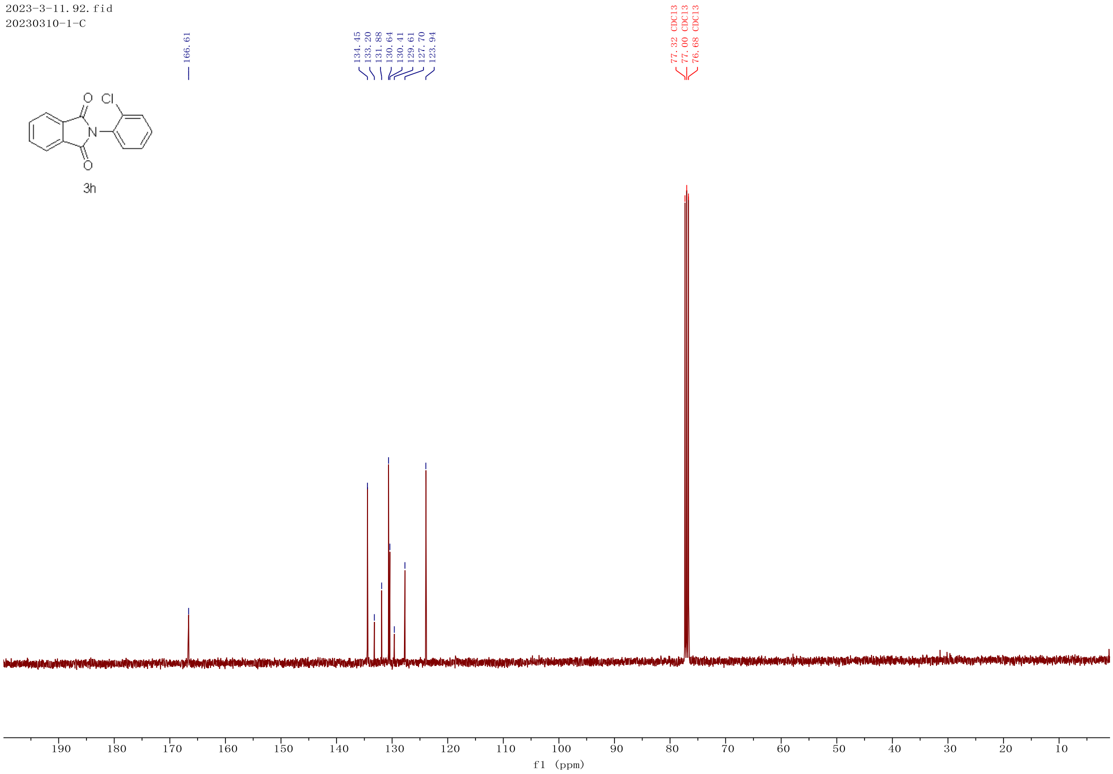
f1 (ppm)

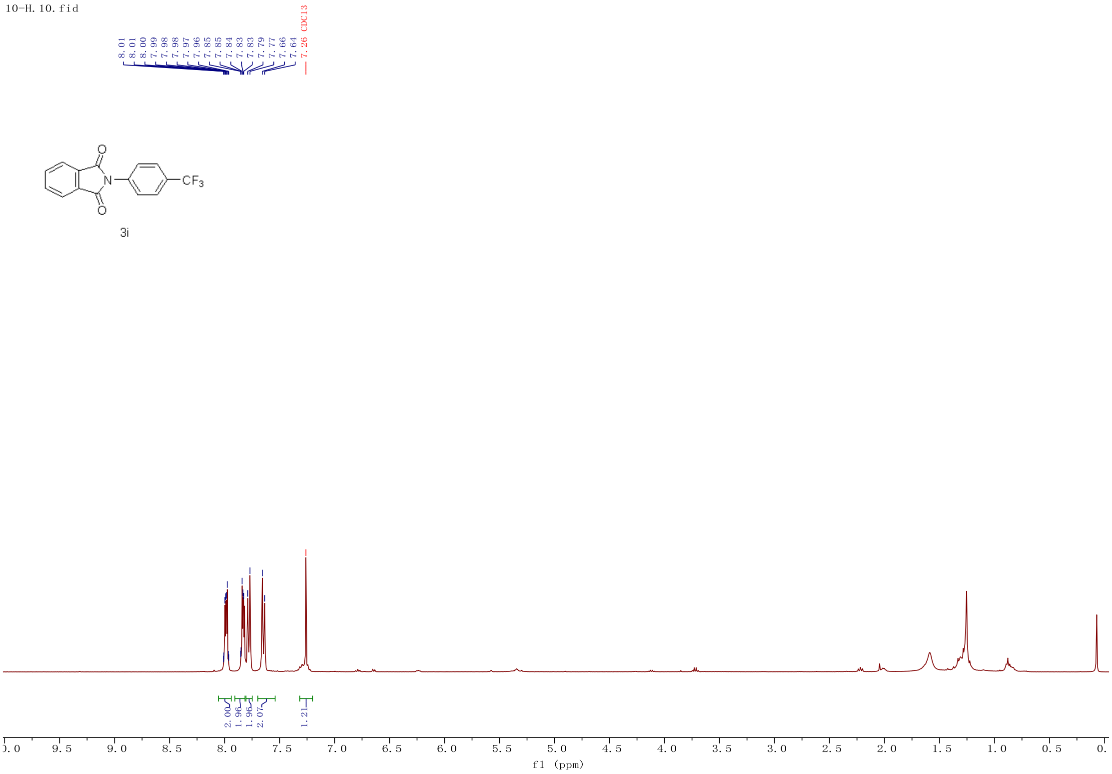
3f

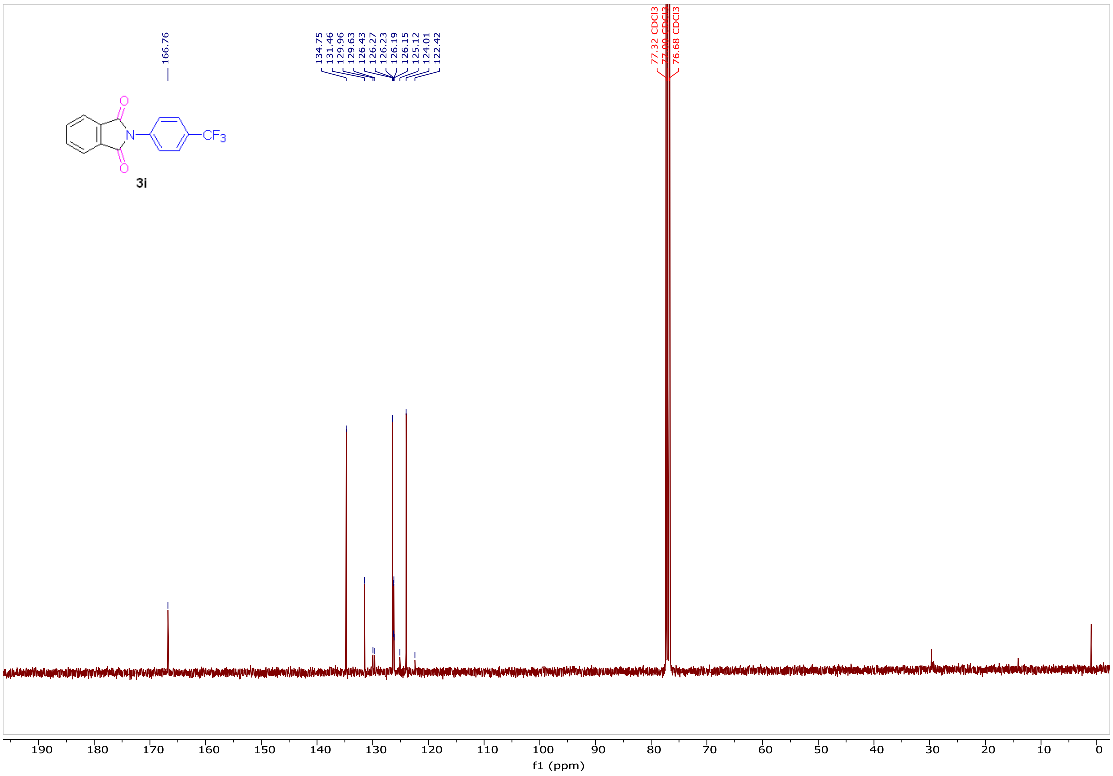

2023-6-12.142.fid
20230612-2-H
7.97
7.96
7.95
7.95
7.81
7.81
7.80
7.79
7.49
7.47
7.47
7.42
7.42
7.40
7.26 CDCl3
O
N
Cl
O
3g
2.04
1.99
1.94
2.00
.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

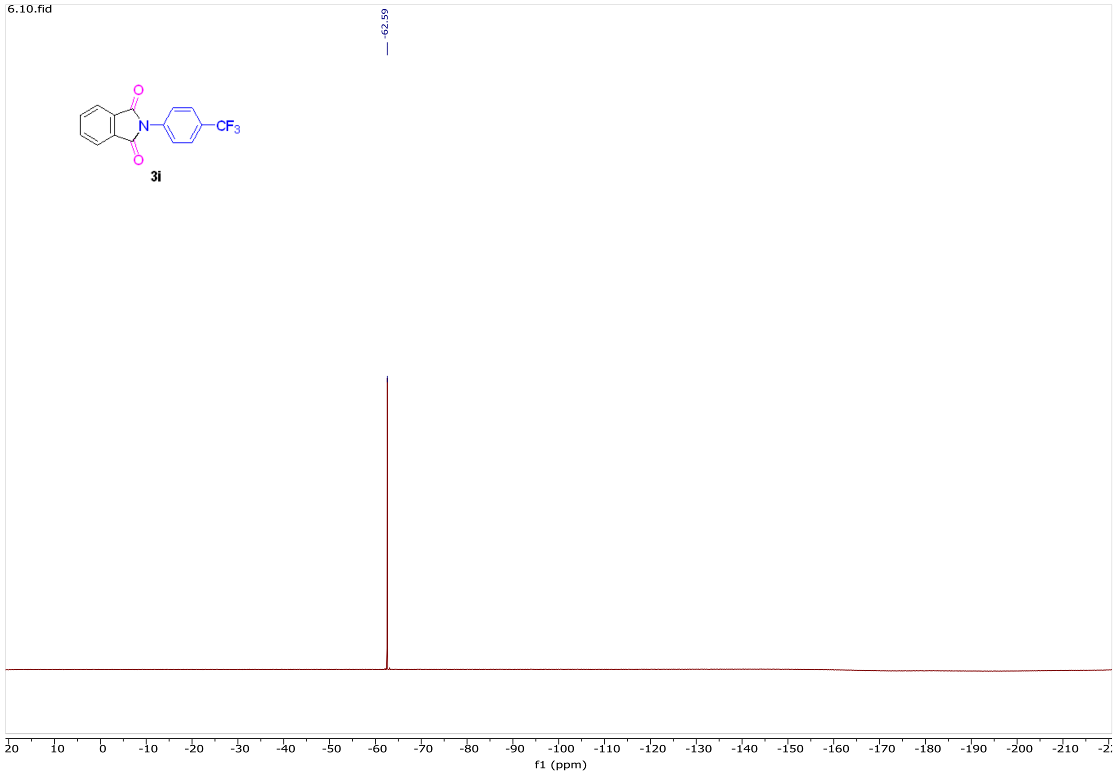

6.10.fid
166.92
134.51
133.72
131.53
130.15
129.24
127.61
123.79
77.32 CDCl3
77.00 CDCl3
76.69 CDCl3
O
N
Cl
O
3g
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

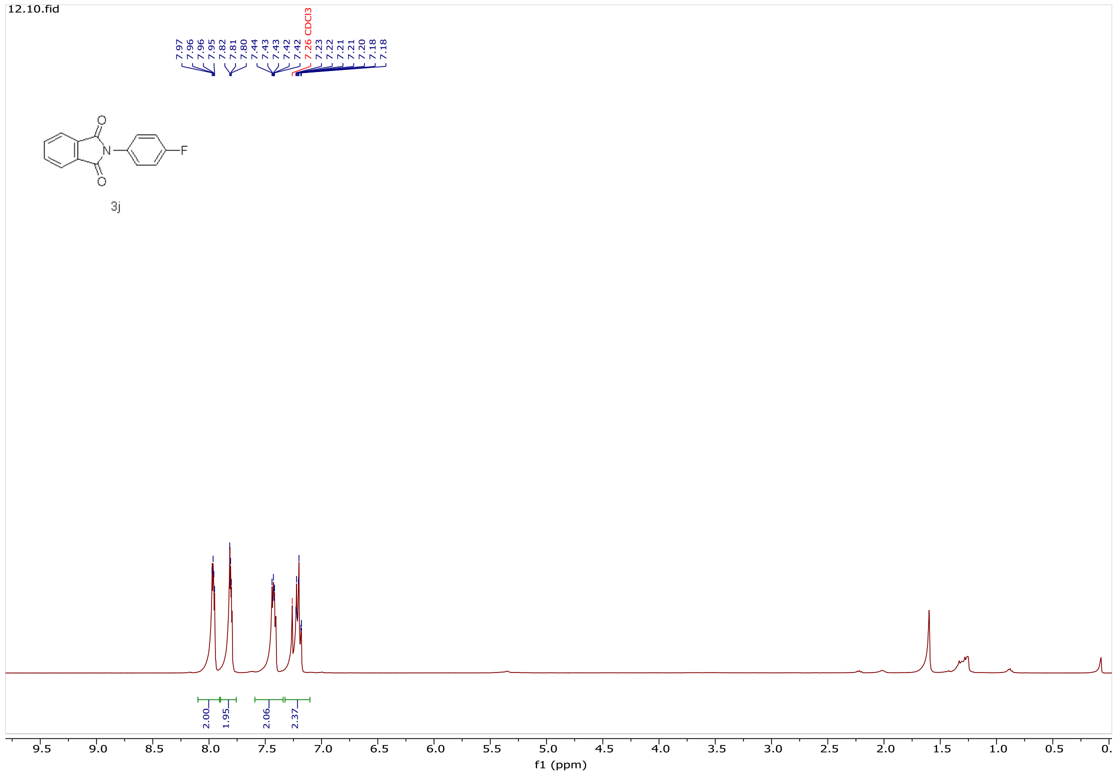

2023-3-11.91.fid
20230310-1-H
7.99
7.98
7.97
7.96
7.82
7.81
7.81
7.80
7.59
7.58
7.58
7.57
7.57
7.56
7.44
7.44
7.44
7.43
7.42
7.42
7.42
7.37
7.36
7.36
7.35
7.35
7.35
7.26 CDCl3
O
Cl
N
O
3h
1.85
1.88
1.00
1.83
0.99
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

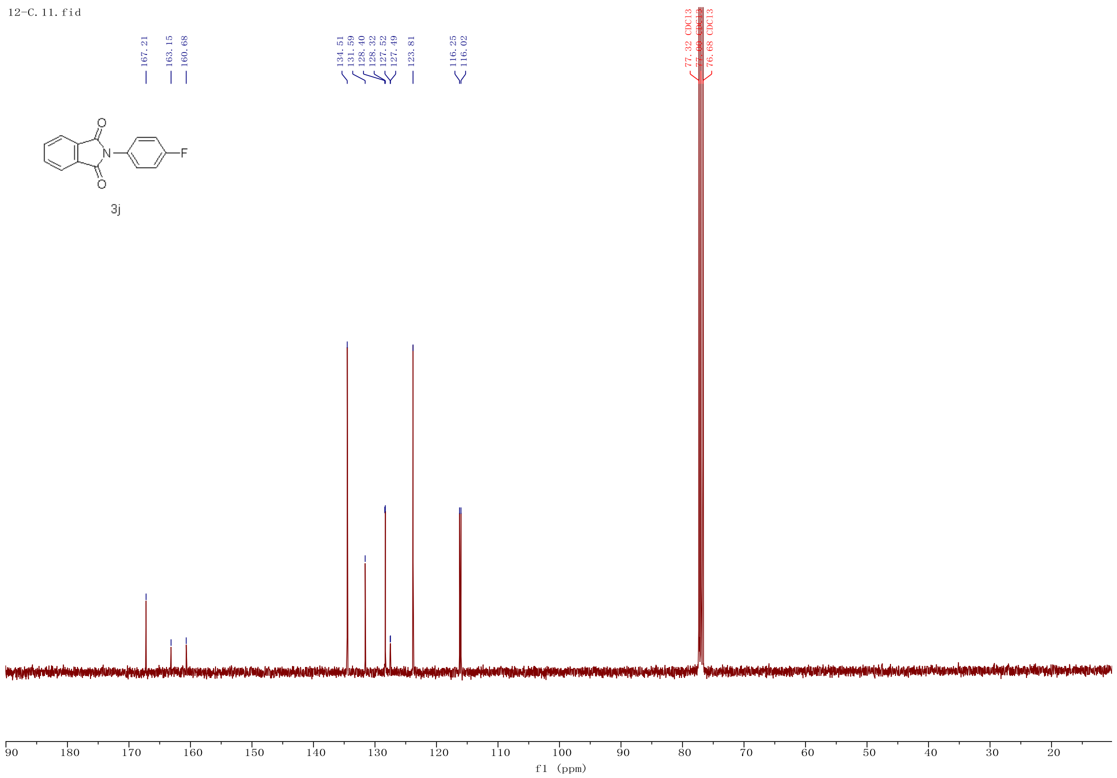

2023-3-11.92.fid
20230310-1-C
166.61
134.45
133.20
131.88
130.64
130.41
129.61
127.70
123.94
77.32 CDCl3
77.00 CDCl3
76.68 CDCl3
O
Cl
N
O
3h
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

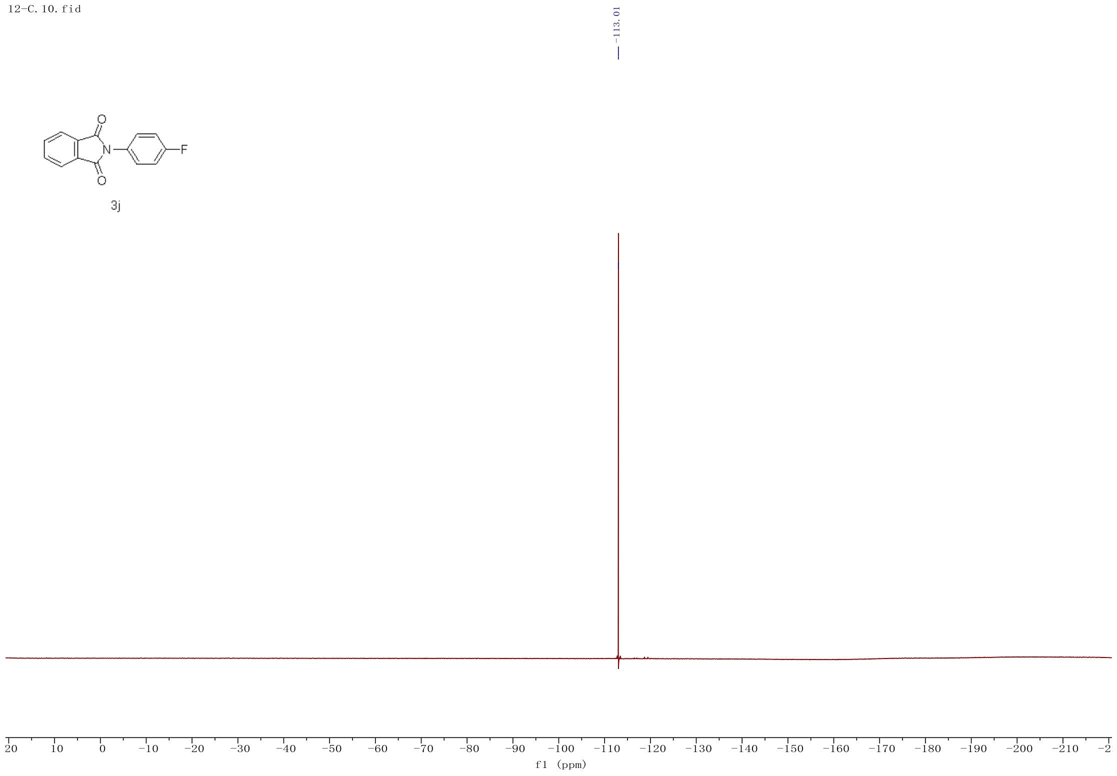

10-H.10.fid
8.01
8.01
8.00
7.99
7.98
7.98
7.97
7.96
7.85
7.85
7.84
7.83
7.83
7.79
7.77
7.66
7.64
7.26 CDCl3
O
N
CF3
O
3i
2.00
1.96
1.96
2.07
1.21
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

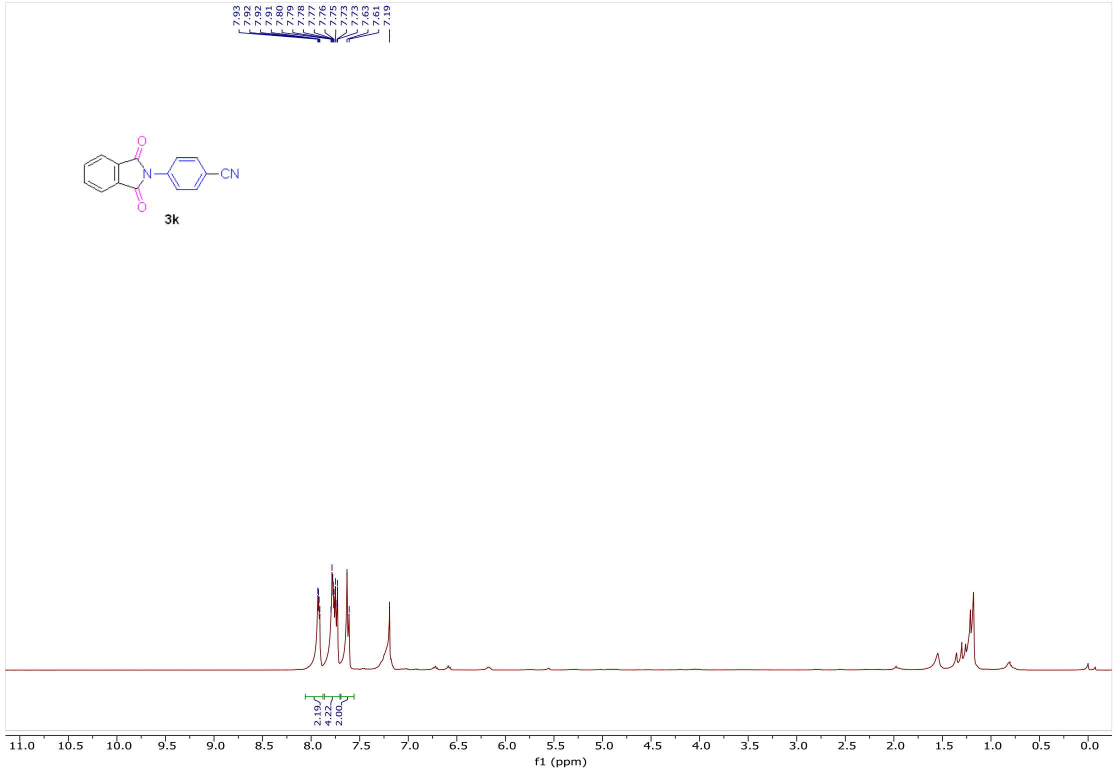

166.76
134.75
131.46
129.96
129.63
126.43
126.27
126.23
126.19
126.15
125.12
124.01
122.42
77.32 CDCl3
77.00 CDCl3
76.68 CDCl3
O
N
CF3
O
3i
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

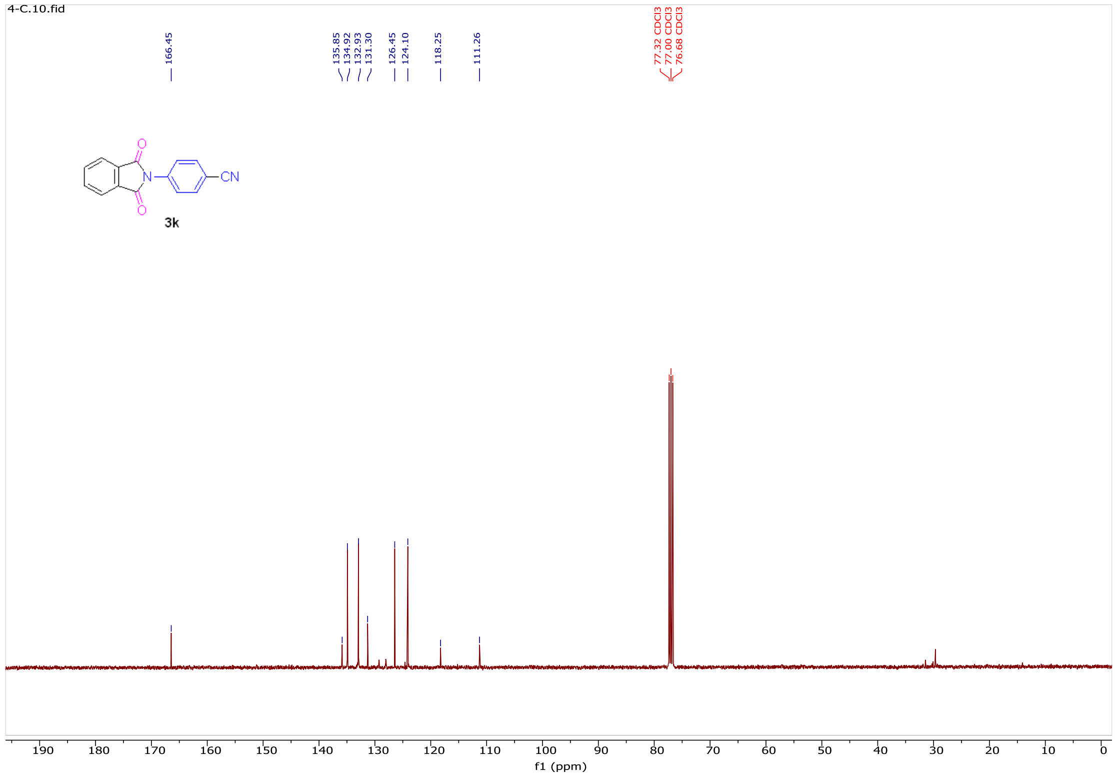

6.10.fid
-62.59
O
N
CF3
O
3i
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)

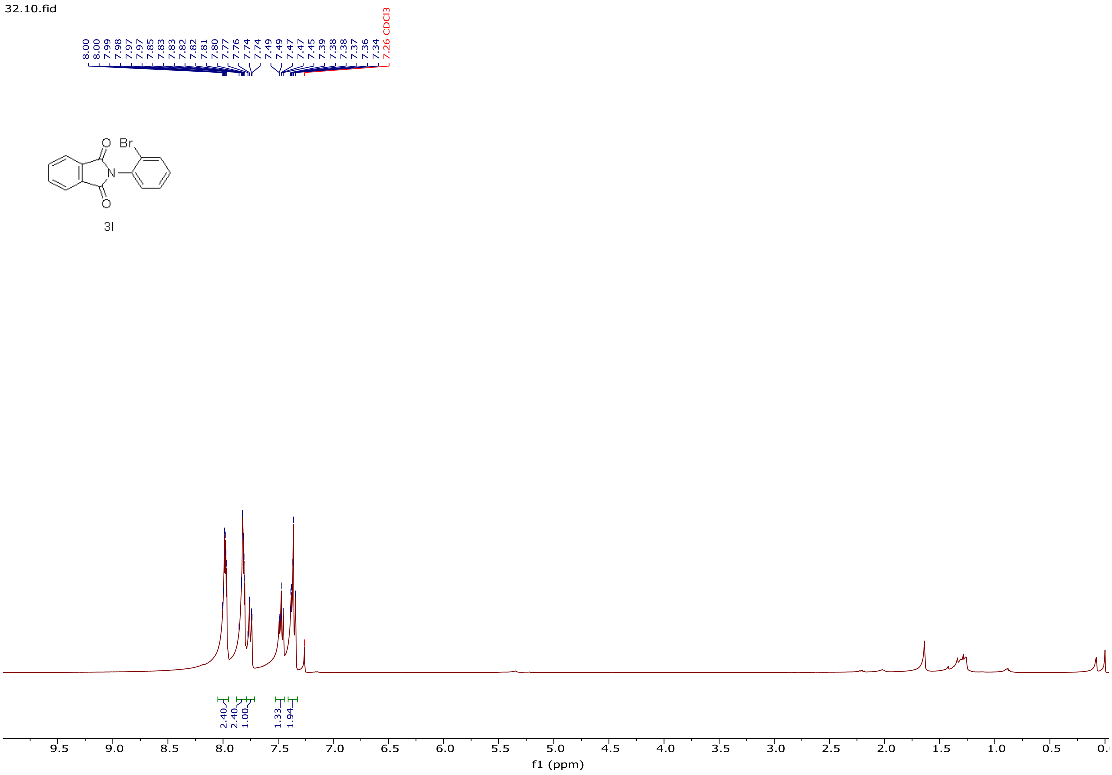

12.10.fid
7.97
7.96
7.96
7.95
7.82
7.81
7.80
7.44
7.43
7.43
7.42
7.42
7.26 CDCl3
7.23
7.22
7.21
7.21
7.20
7.18
7.18
O
N
F
O
3j
2.00
1.95
2.06
2.37
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

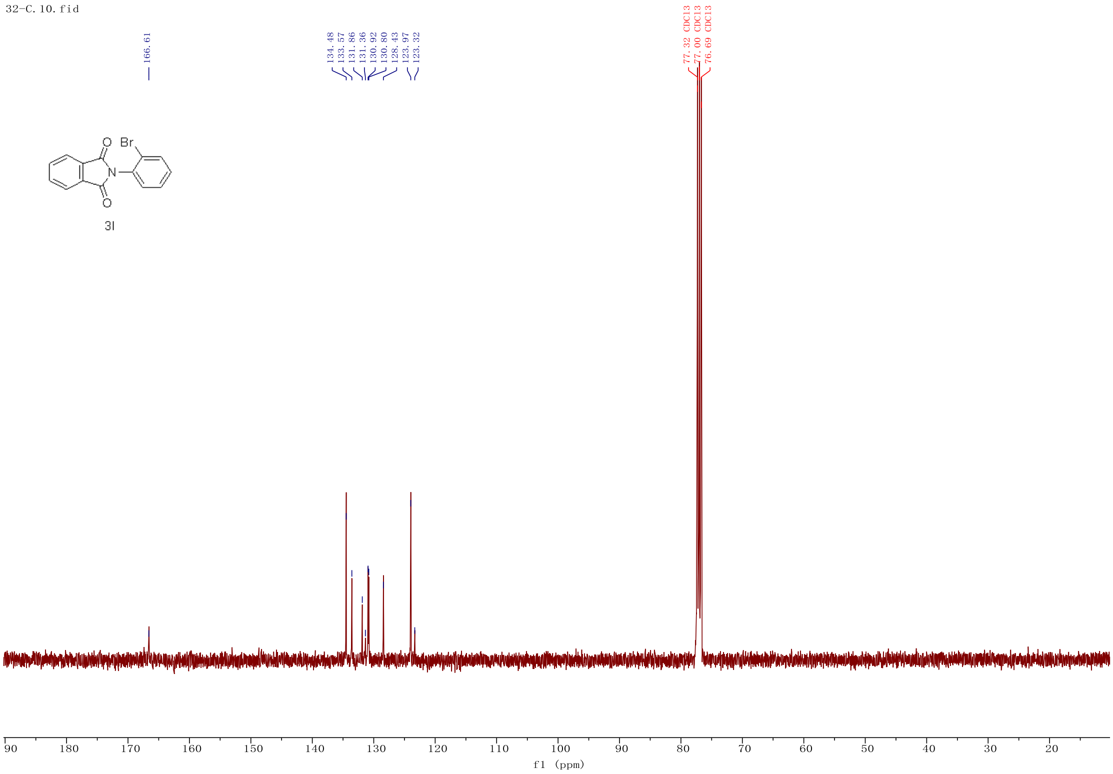

12-C.11.fid
167.21
163.15
160.68
134.51
131.59
128.40
128.32
127.52
127.49
123.81
116.25
116.02
77.32 CDCl3
77.00 CDCl3
76.68 CDCl3
O
N
F
O
3j
90
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
f1 (ppm)

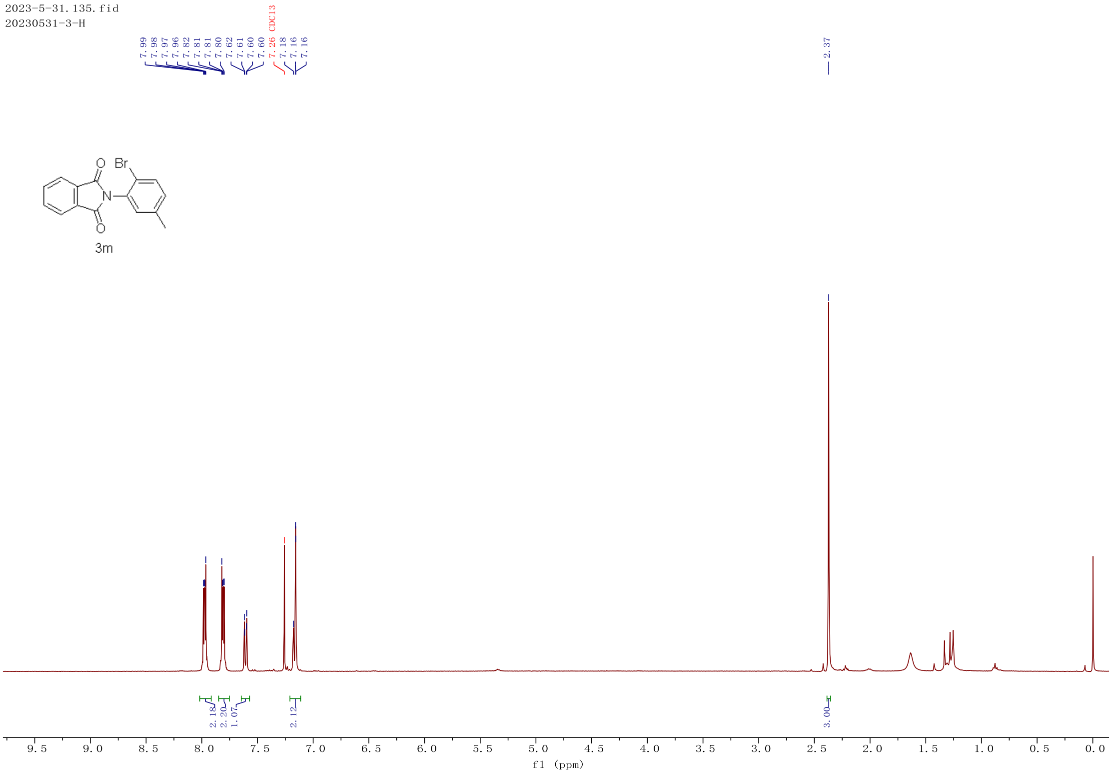

12-C.10.fid
-113.01
O
N
F
O
3j
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
-2
f1 (ppm)

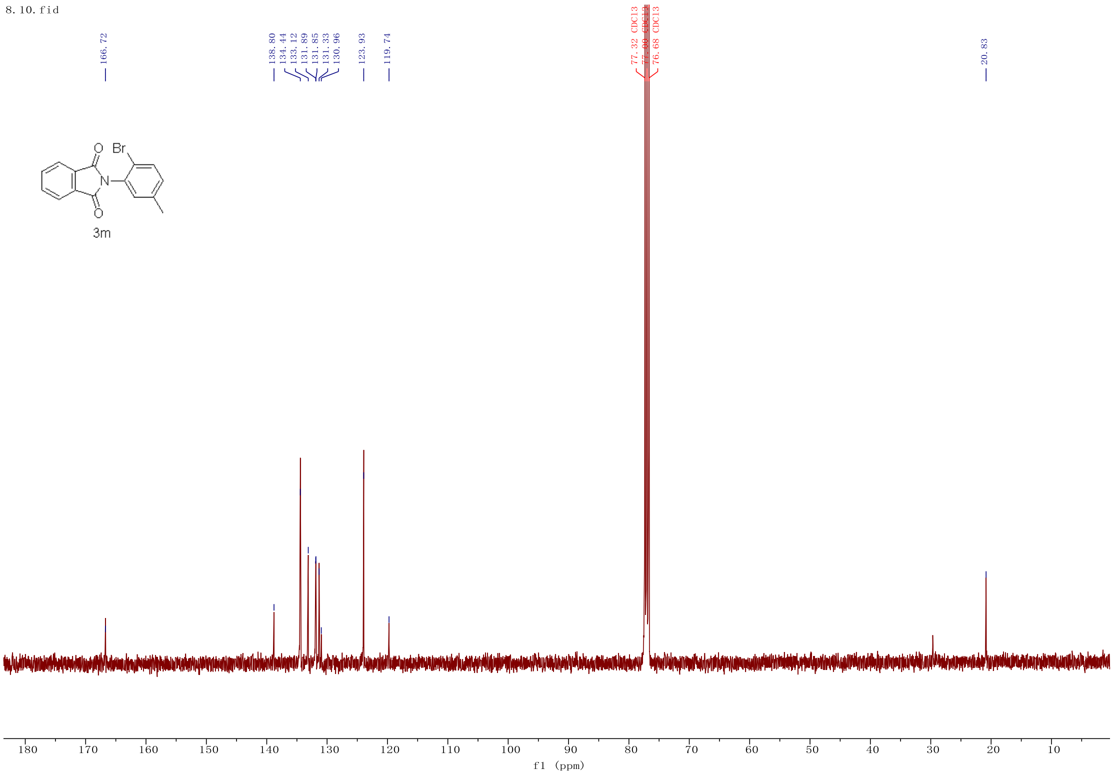

7.93
7.92
7.92
7.91
7.80
7.79
7.78
7.77
7.76
7.75
7.73
7.73
7.63
7.61
7.19
O
N
CN
O
3k
2.19
4.22
2.00
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

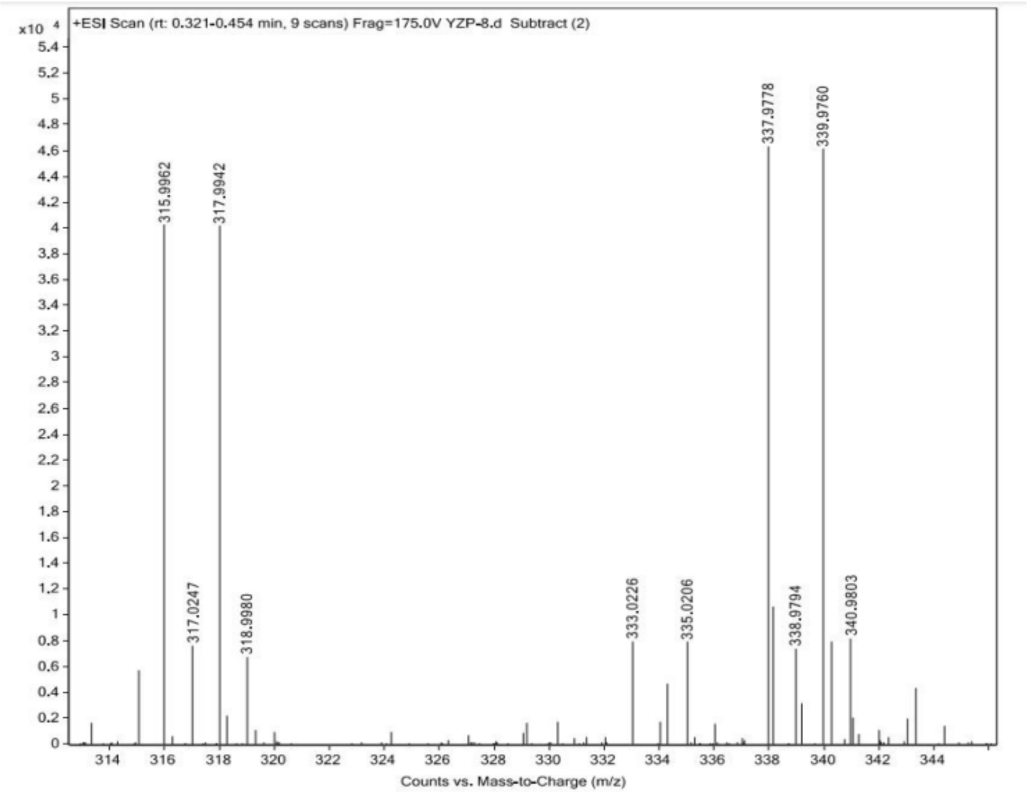

4-C.10.fid
166.45
135.85
134.92
132.93
131.30
126.45
124.10
118.25
111.26
77.32 CDCl3
77.00 CDCl3
76.68 CDCl3
O
N
CN
O
3k
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

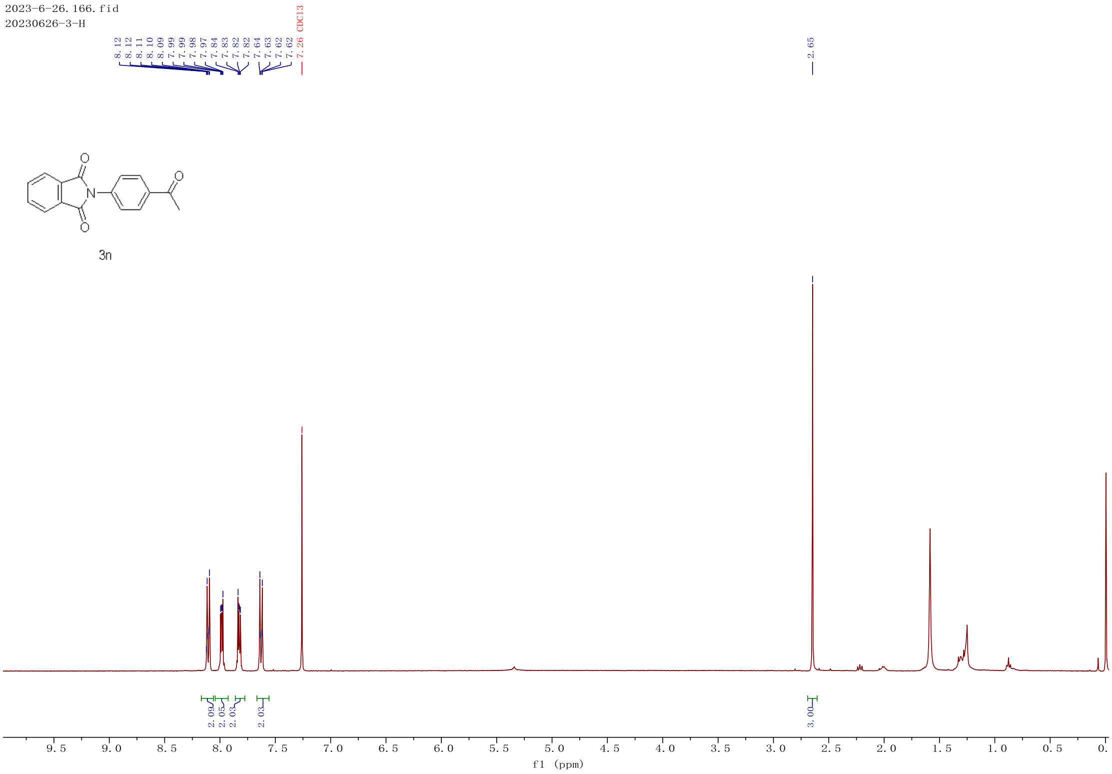

32.10.fid
8.00
8.00
7.99
7.98
7.97
7.97
7.85
7.83
7.83
7.82
7.82
7.81
7.80
7.77
7.76
7.74
7.74
7.49
7.49
7.47
7.47
7.45
7.39
7.38
7.38
7.37
7.36
7.34
7.26 CDCl3
O
Br
N
O
3l
2.40
2.40
1.00
1.33
1.94
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.
f1 (ppm)

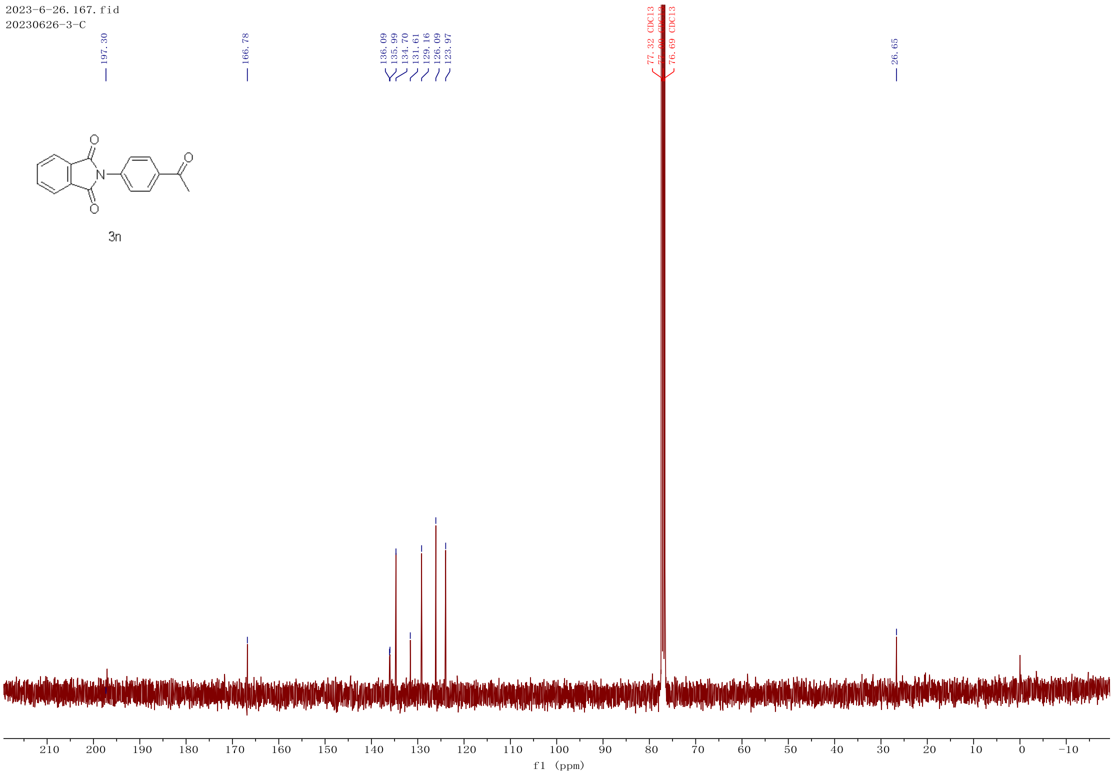

32-C.10.fid
166.61
134.48
133.57
131.86
131.36
130.92
130.80
128.43
123.97
123.32
77.32 CDCl3
77.00 CDCl3
76.69 CDCl3
O
Br
N
O
3l
90
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
f1 (ppm)


2023-5-31.135.fid
20230531-3-H
7.99
7.98
7.97
7.96
7.82
7.81
7.81
7.80
7.62
7.61
7.60
7.60
7.26 CDCl3
7.18
7.16
7.16
2.37
O
Br
N
O
3m
2.18
2.20
1.07
2.12
3.00
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
f1 (ppm)

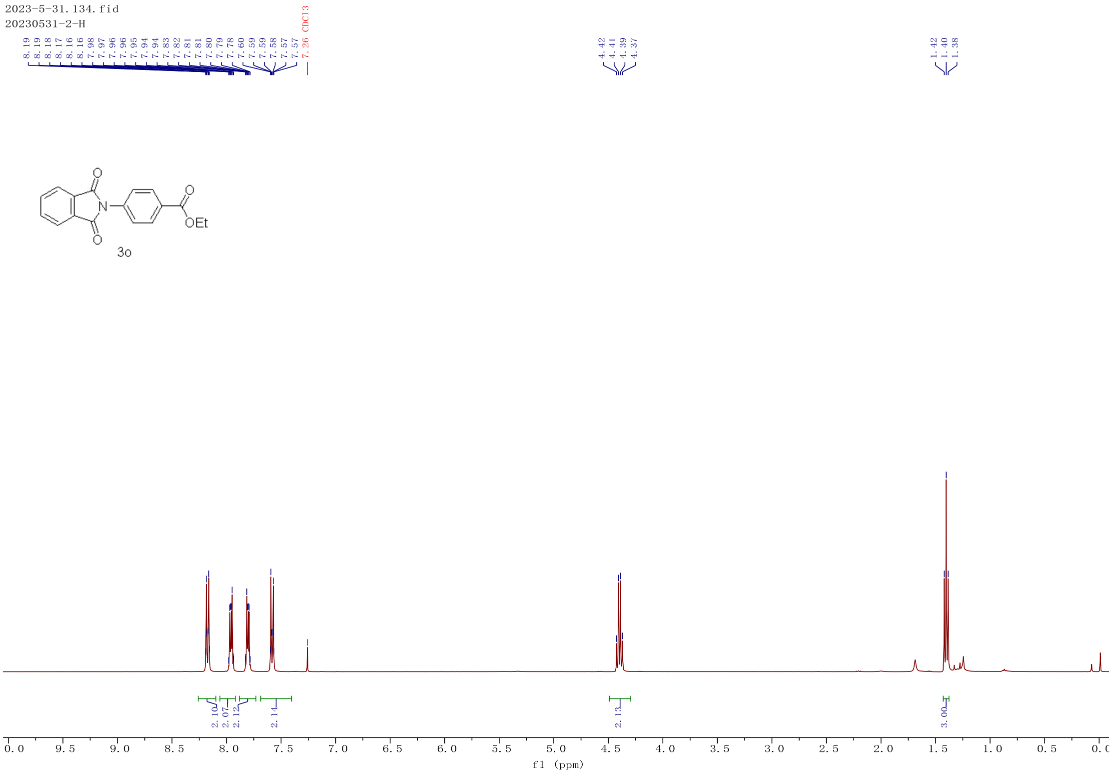

8.10.fid
166.72
138.80
134.44
133.12
131.89
131.85
131.33
130.96
123.93
119.74
77.32 CDCl3
77.00 CDCl3
76.68 CDCl3
20.83
O
Br
N
O
3m
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)


+ESI Scan (rt: 0.321-0.454 min, 9 scans) Frag=175.0V YZP-8.d Subtract (2)
x10 4
5.4
5.2
5
4.8
4.6
4.4
4.2
4
3.8
3.6
3.4
3.2
3
2.8
2.6
2.4
2.2
2
1.8
1.6
1.4
1.2
1
0.8
0.6
0.4
0.2
0
315.9962
317.9942
317.0247
318.9980
333.0226
335.0206
337.9778
339.9760
338.9794
340.9803
314
316
318
320
322
324
326
328
330
332
334
336
338
340
342
344
Counts vs. Mass-to-Charge (m/z)

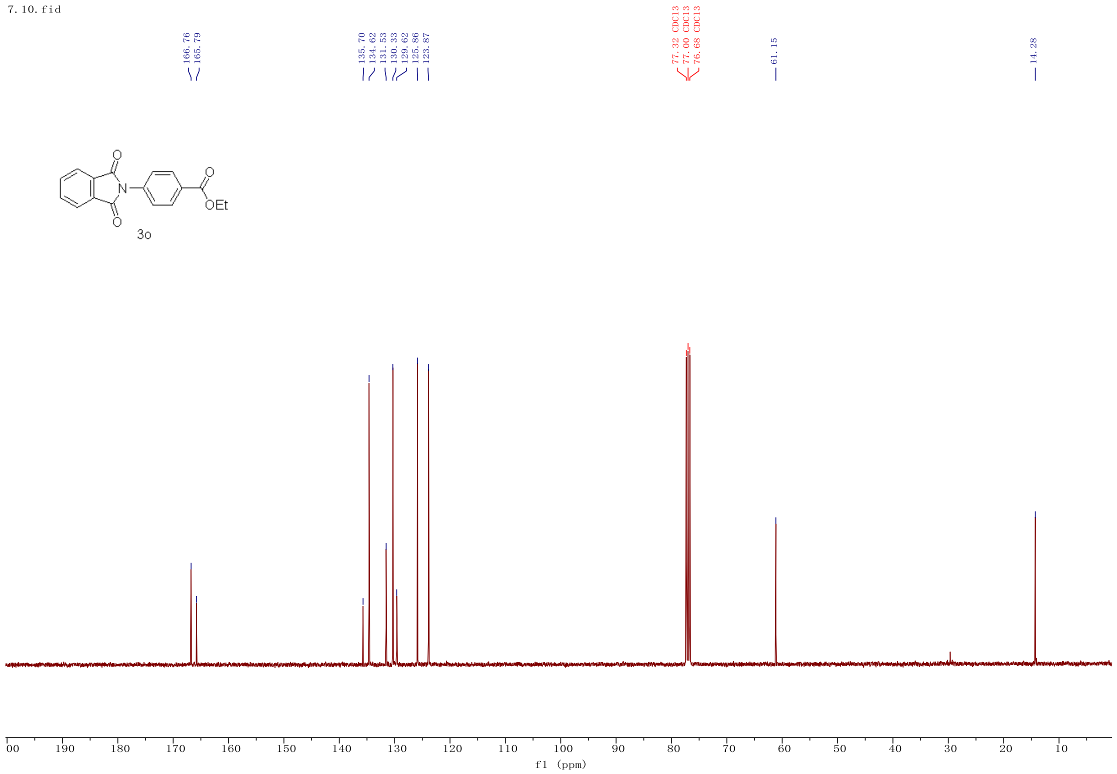

2023-6-26.166.fid
20230626-3-H
8.12
8.12
8.11
8.10
8.09
7.99
7.99
7.98
7.97
7.84
7.83
7.82
7.82
7.64
7.63
7.62
7.62
7.26 CDCl3
2.65
O
N
O
O
3n
2.09
2.05
2.03
2.03
3.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.
f1 (ppm)

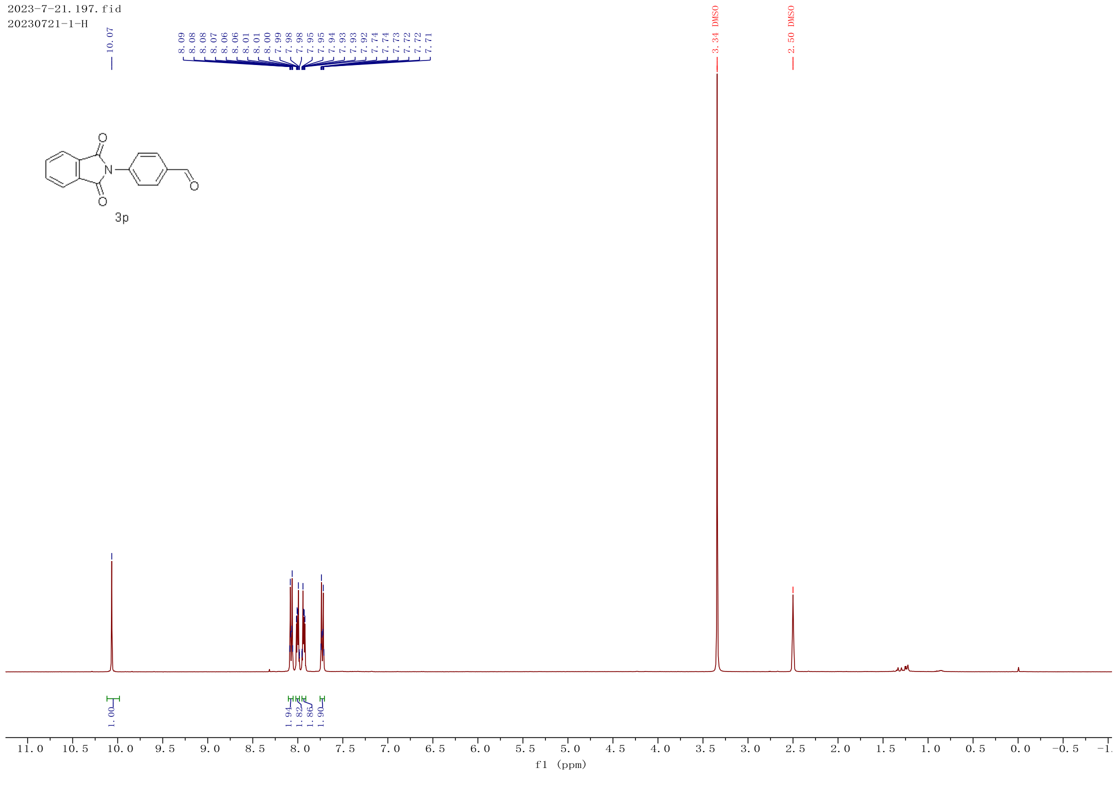

2023-6-26.167.fid
20230626-3-C
197.30
166.78
136.09
135.99
134.70
131.61
129.16
126.09
123.97
77.32 CDCl3
77.00 CDCl3
76.69 CDCl3
26.65
O
N
O
O
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

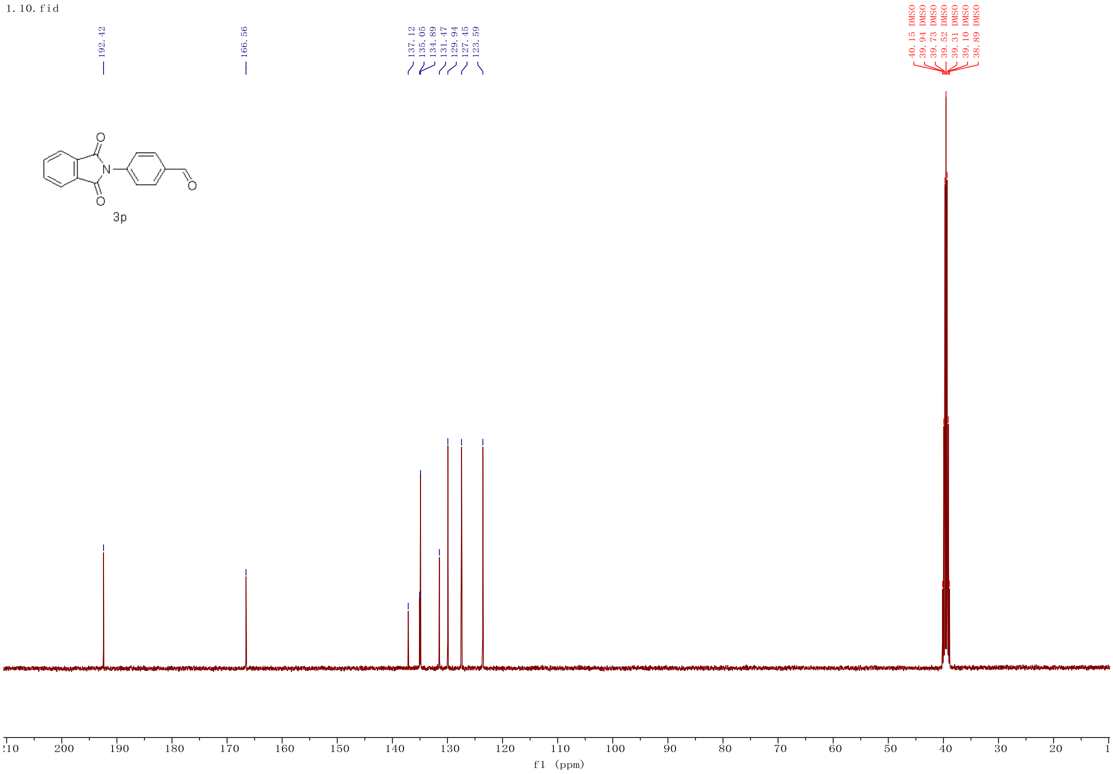
3n

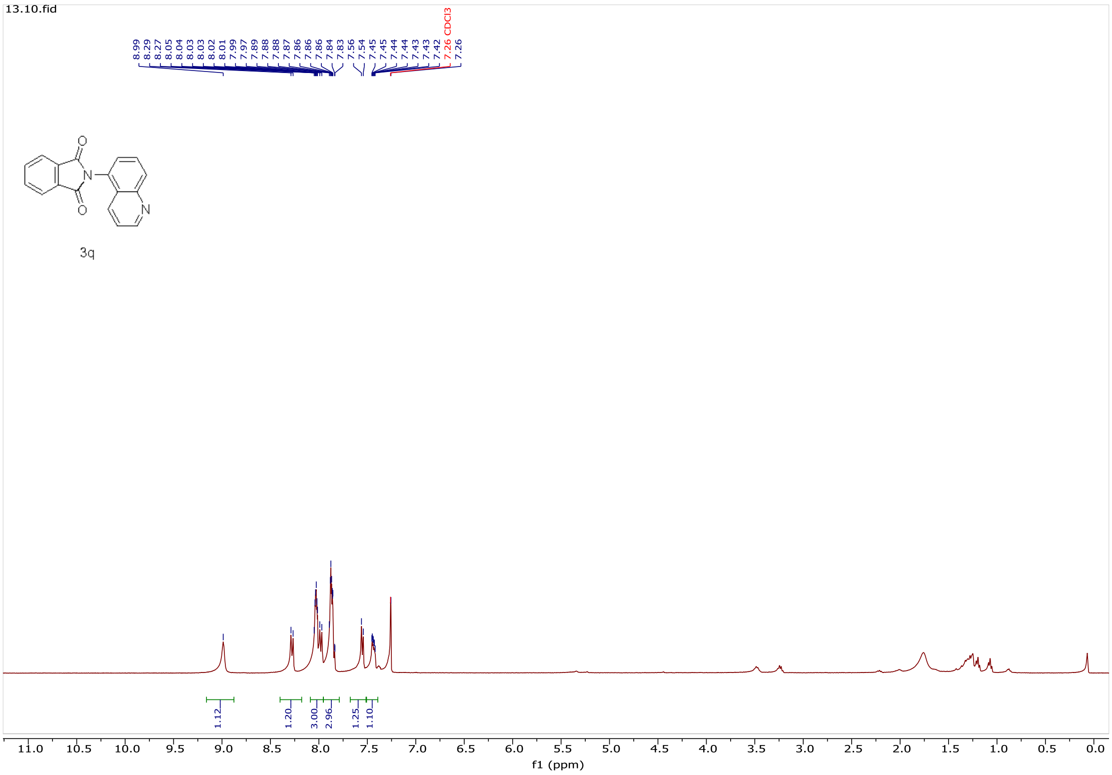

2023-5-31.134.fid
20230531-2-H
8.19
8.19
8.18
8.17
8.16
8.16
7.98
7.97
7.96
7.96
7.95
7.94
7.94
7.83
7.82
7.81
7.81
7.80
7.79
7.78
7.60
7.59
7.59
7.58
7.57
7.57
7.26 CDCl3
4.42
4.41
4.39
4.37
1.42
1.40
1.38
O
N
O
O
OEt
2.10
2.07
2.12
2.14
2.13
3.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

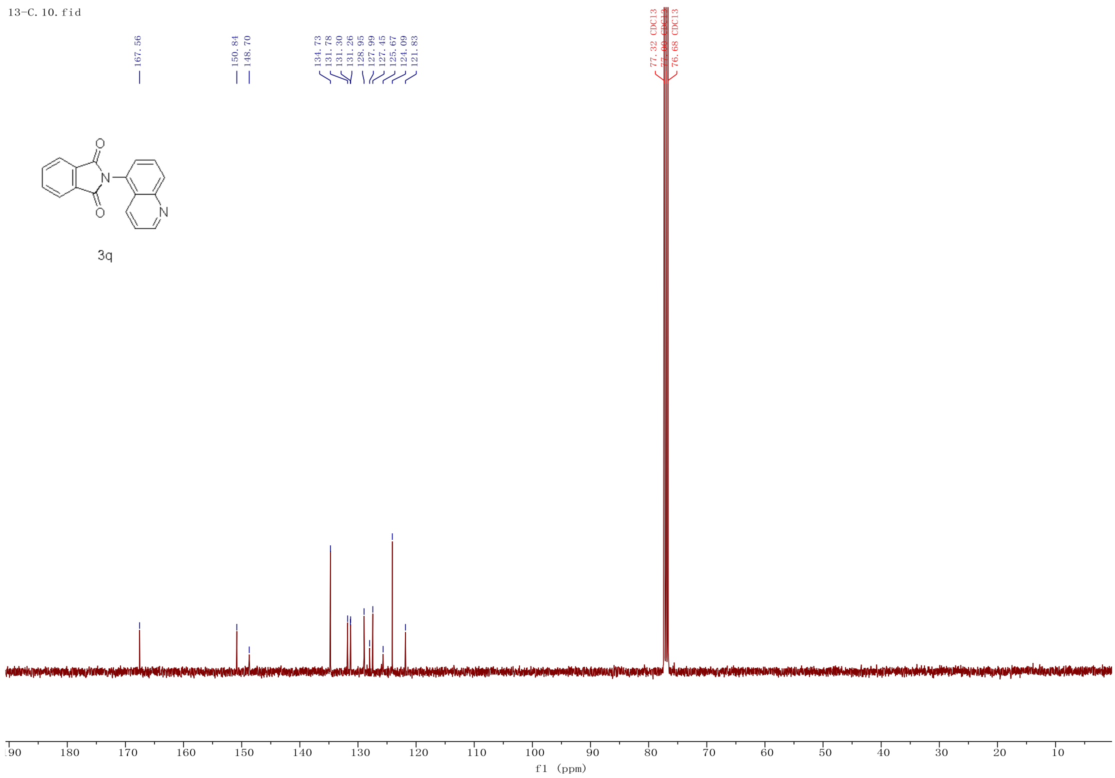
3o

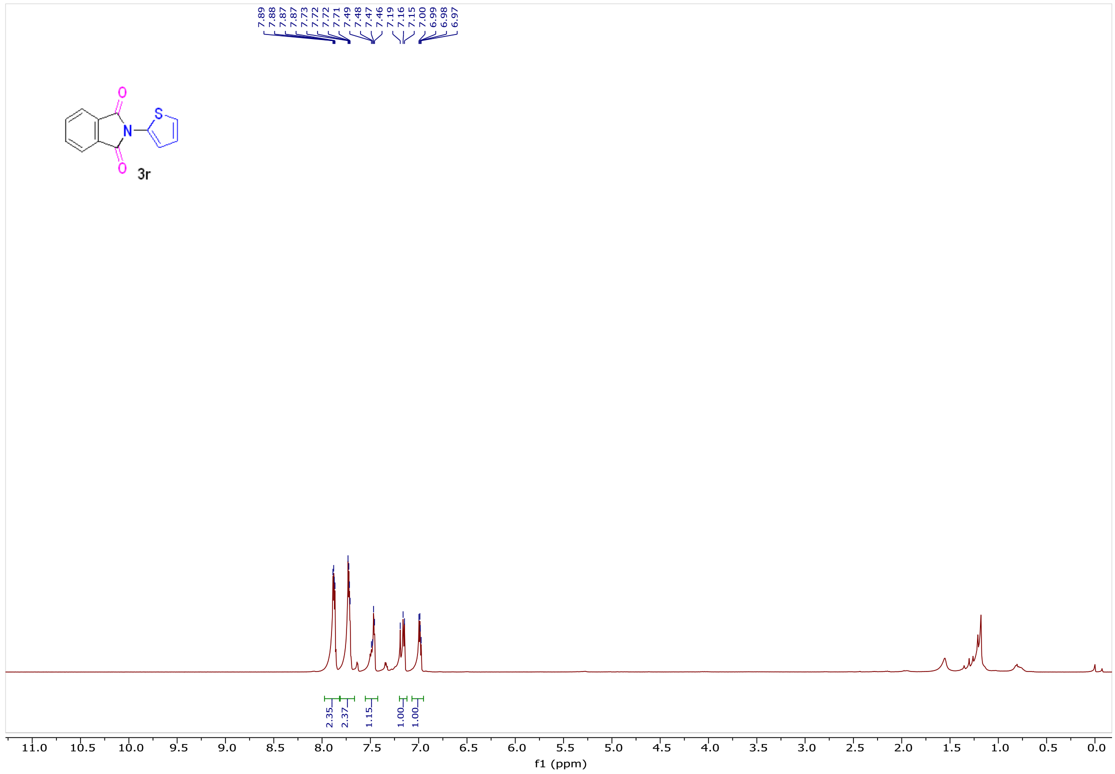

7.10.fid
166.76
165.79
135.70
134.62
131.53
130.33
129.62
125.86
123.87
77.32 CDCl3
77.00 CDCl3
76.68 CDCl3
61.15
14.28
O
N
O
O
OEt
3o
00
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

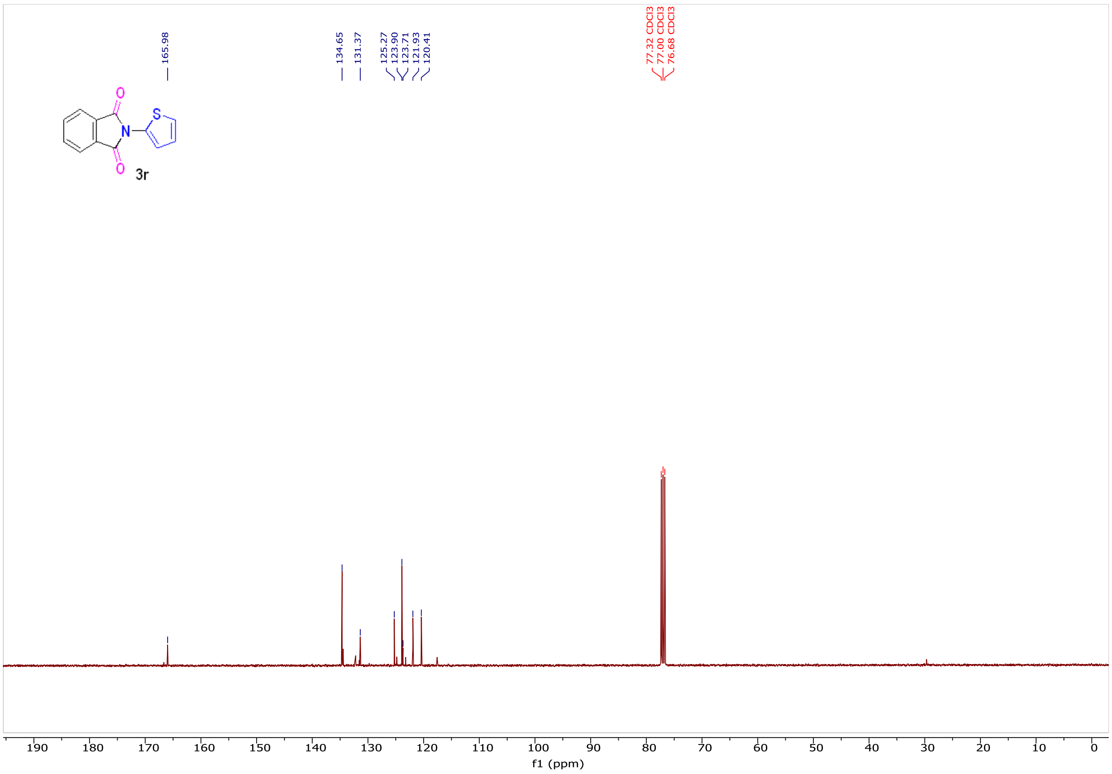

2023-7-21.197.fid
20230721-1-H
10.07
8.09
8.08
8.08
8.07
8.06
8.06
8.01
8.01
8.00
7.99
7.98
7.98
7.95
7.95
7.94
7.93
7.93
7.92
7.74
7.74
7.73
7.72
7.72
7.71
3.34 DMSO
2.50 DMSO
O
N
O
O
3p
1.00
1.94
1.82
1.86
1.90
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1
f1 (ppm)

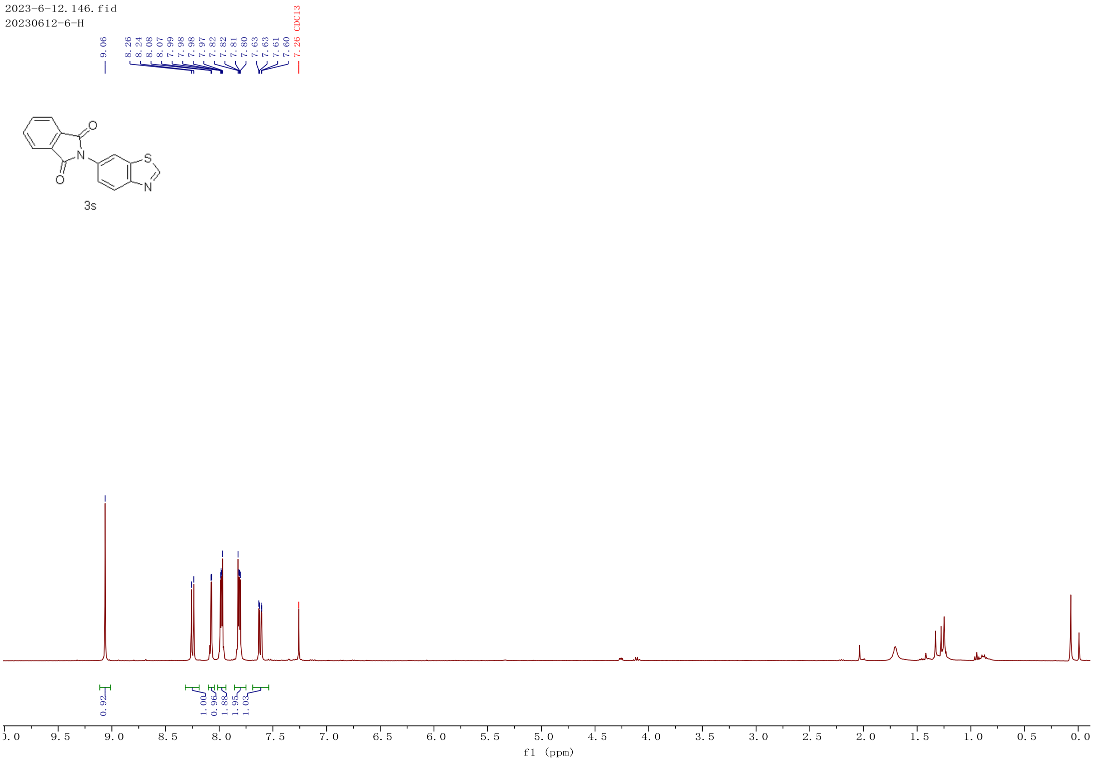

1.10.fid
192.42
166.56
137.12
135.05
134.89
131.47
129.94
127.45
123.59
40.15 DMSO
39.94 DMSO
39.73 DMSO
39.52 DMSO
39.31 DMSO
39.10 DMSO
38.89 DMSO
O
N
O
O
3p
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
1
f1 (ppm)

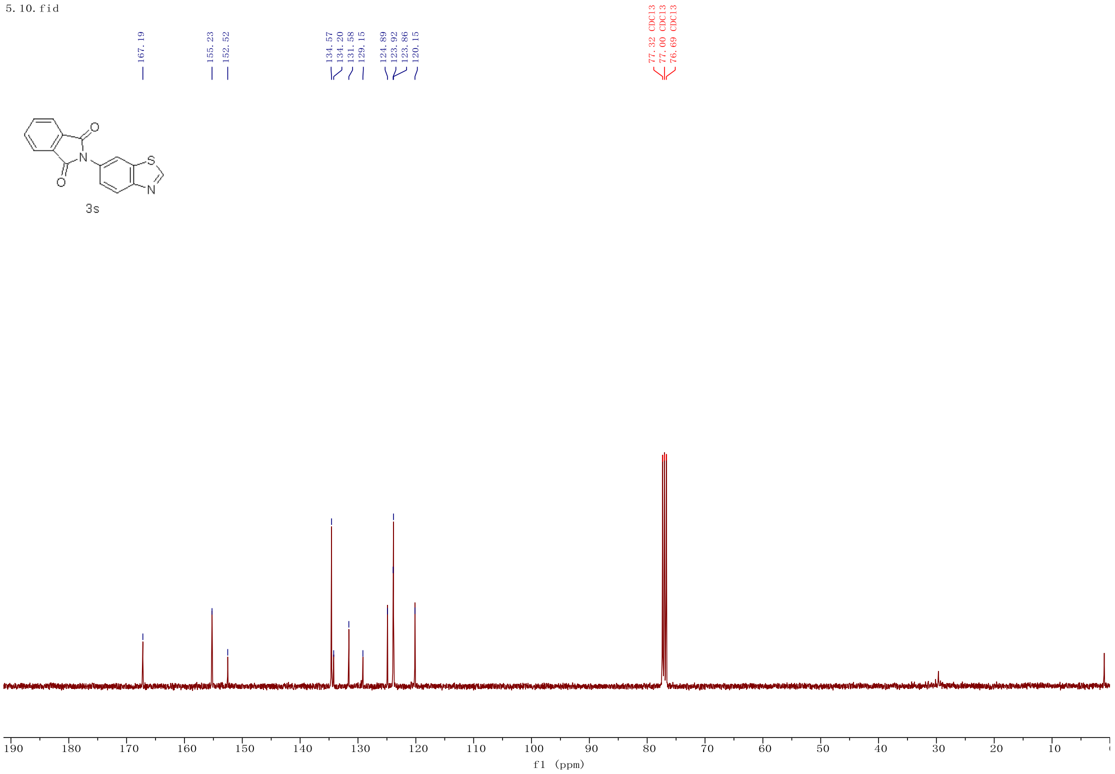

13.10.fid
8.99
8.29
8.27
8.05
8.04
8.03
8.03
8.02
8.01
7.99
7.97
7.89
7.88
7.88
7.87
7.86
7.86
7.86
7.84
7.83
7.56
7.54
7.45
7.45
7.44
7.44
7.43
7.43
7.42
7.26 CDCl3
7.26
O
N
O
N
3q
1.12
1.20
3.00
2.96
1.25
1.10
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

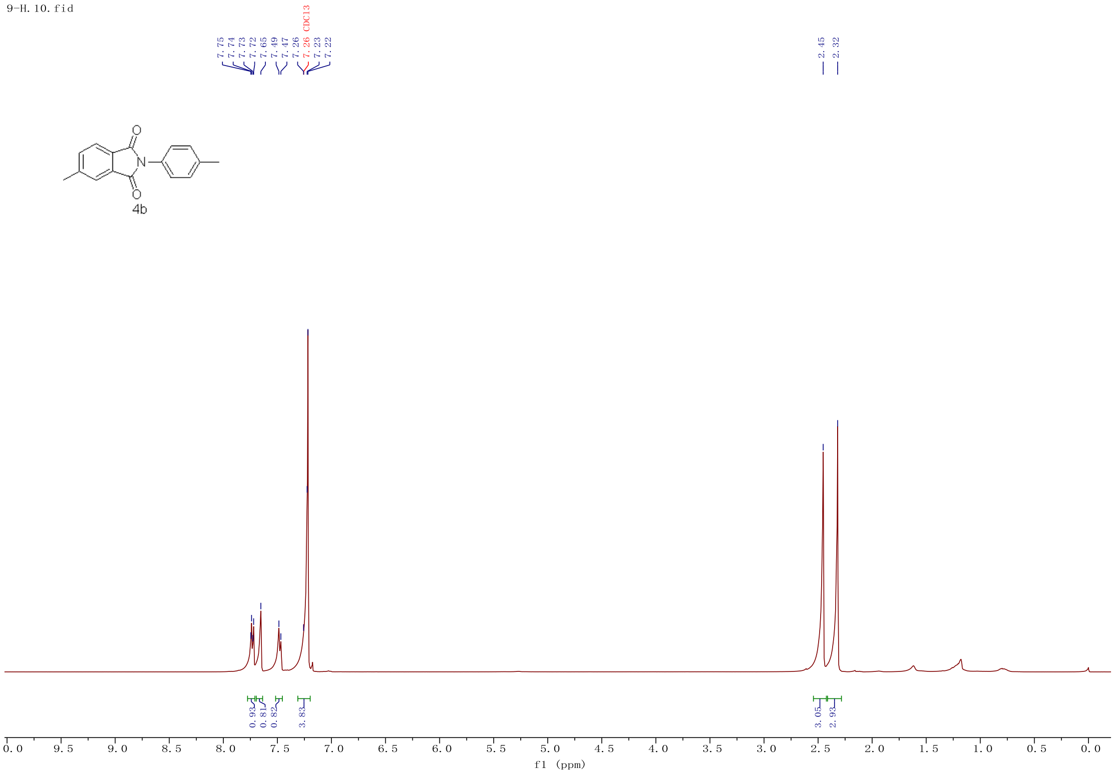

13-C.10.fid
167.56
150.84
148.70
134.73
131.78
131.30
131.26
128.95
127.99
127.45
125.67
124.09
121.83
77.32 CDCl3
77.00 CDCl3
76.68 CDCl3
O
N
O
N
3q
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

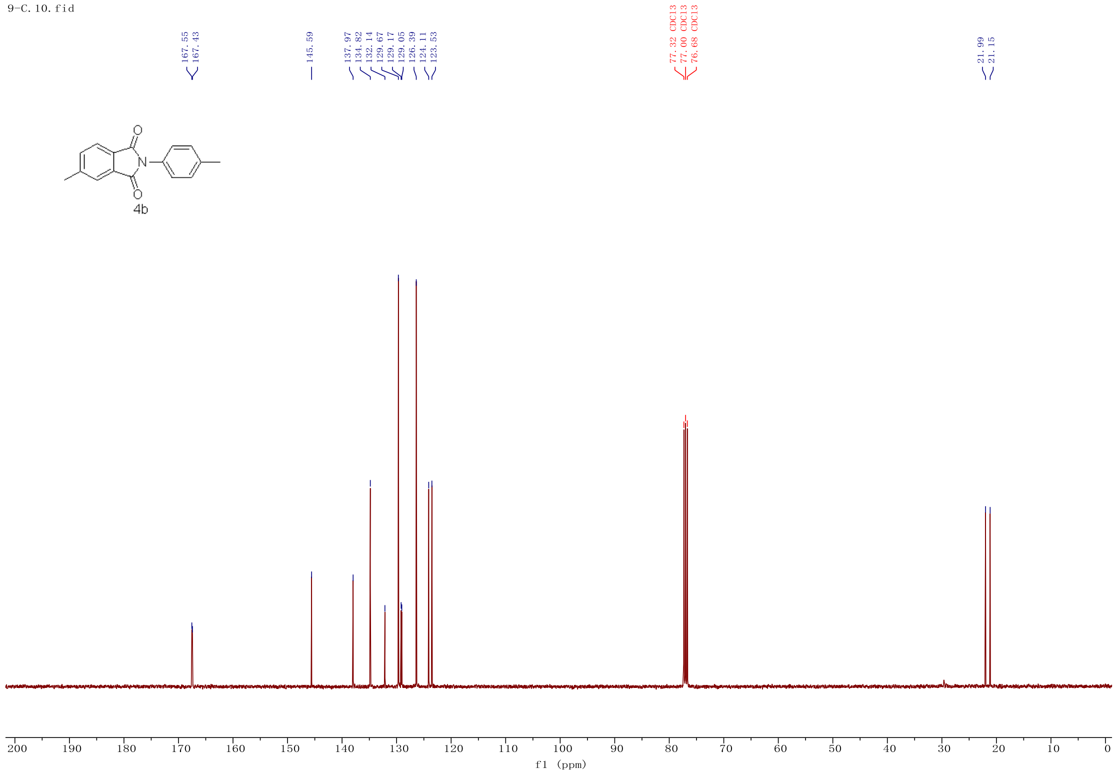

7.89
7.88
7.87
7.87
7.73
7.72
7.72
7.71
7.49
7.48
7.47
7.46
7.19
7.16
7.15
7.00
6.99
6.98
6.97
O
N
S
O
3r
2.35
2.37
1.15
1.00
1.00
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

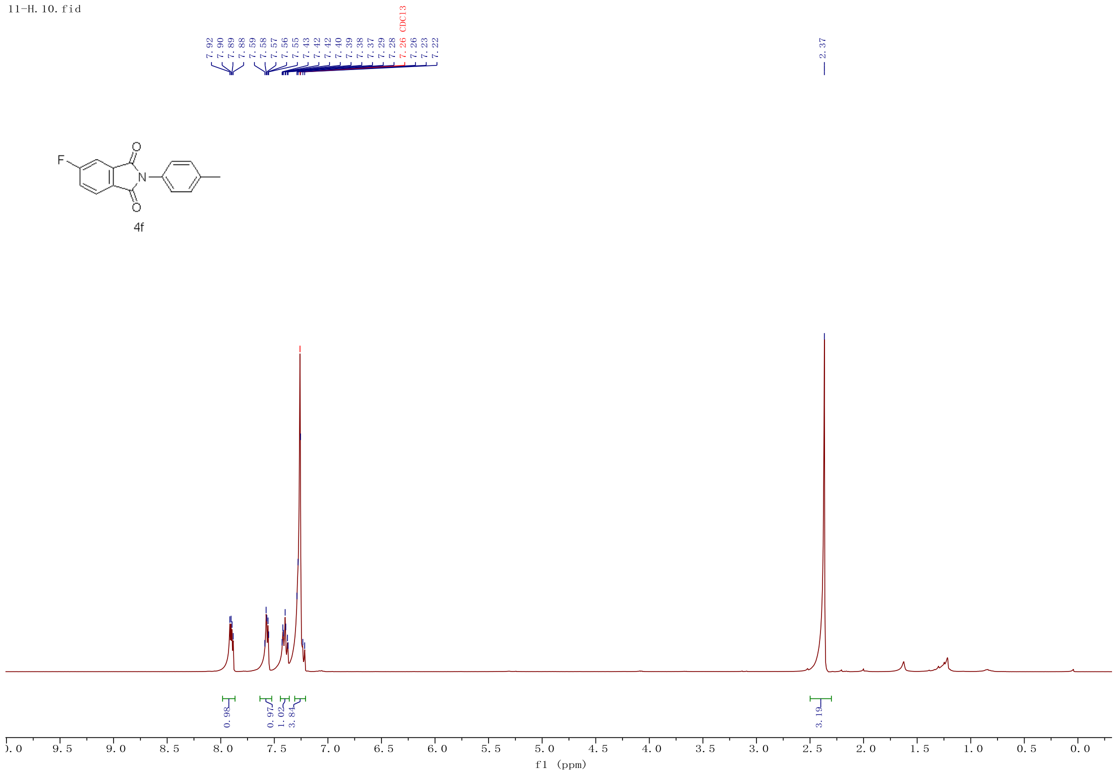

3r
165.98
134.65
131.37
125.27
123.90
123.71
121.93
120.41
77.32 CDCl3
77.00 CDCl3
76.68 CDCl3
O
N
S
O
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


3s
2023-6-12.146.fid
20230612-6-H
9.06
8.26
8.24
8.08
8.07
7.99
7.98
7.98
7.97
7.82
7.82
7.81
7.80
7.63
7.63
7.61
7.60
7.26 CDCl3
O
N
O
S
N
0.92
1.00
0.96
1.88
1.95
1.03
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
f1 (ppm)


5.10.fid
167.19
155.23
152.52
134.57
134.20
131.58
129.15
124.89
123.92
123.86
120.15
77.32 CDCl3
77.00 CDCl3
76.69 CDCl3
O
N
O
S
N
3s
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)


9-H.10.fid
7.75
7.74
7.73
7.72
7.65
7.49
7.47
7.26 CDCl3
7.23
7.22
2.45
2.32
O
N
O
4b
0.93
0.81
0.82
3.83
3.05
2.93
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
f1 (ppm)


9-C.10.fid
167.55
167.43
145.59
137.97
134.82
132.14
129.67
129.17
129.05
126.39
124.11
123.53
77.32 CDCl3
77.00 CDCl3
76.68 CDCl3
21.99
21.15
O
N
O
4b
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


11-H.10.fid
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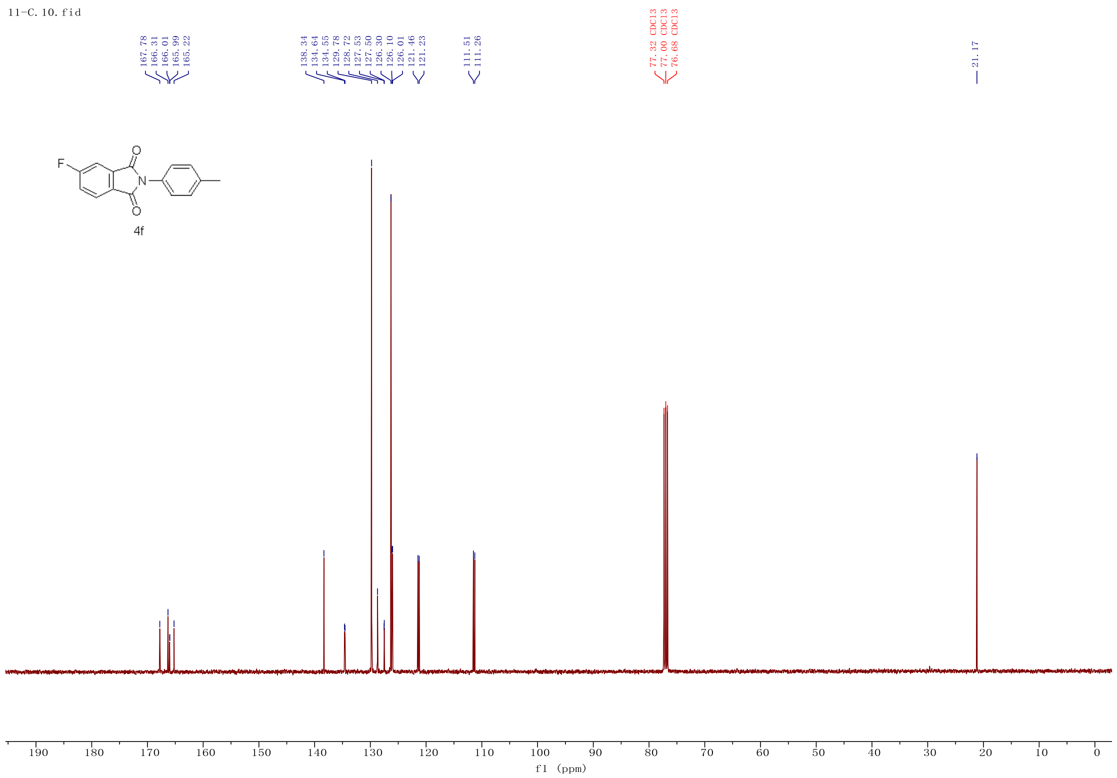
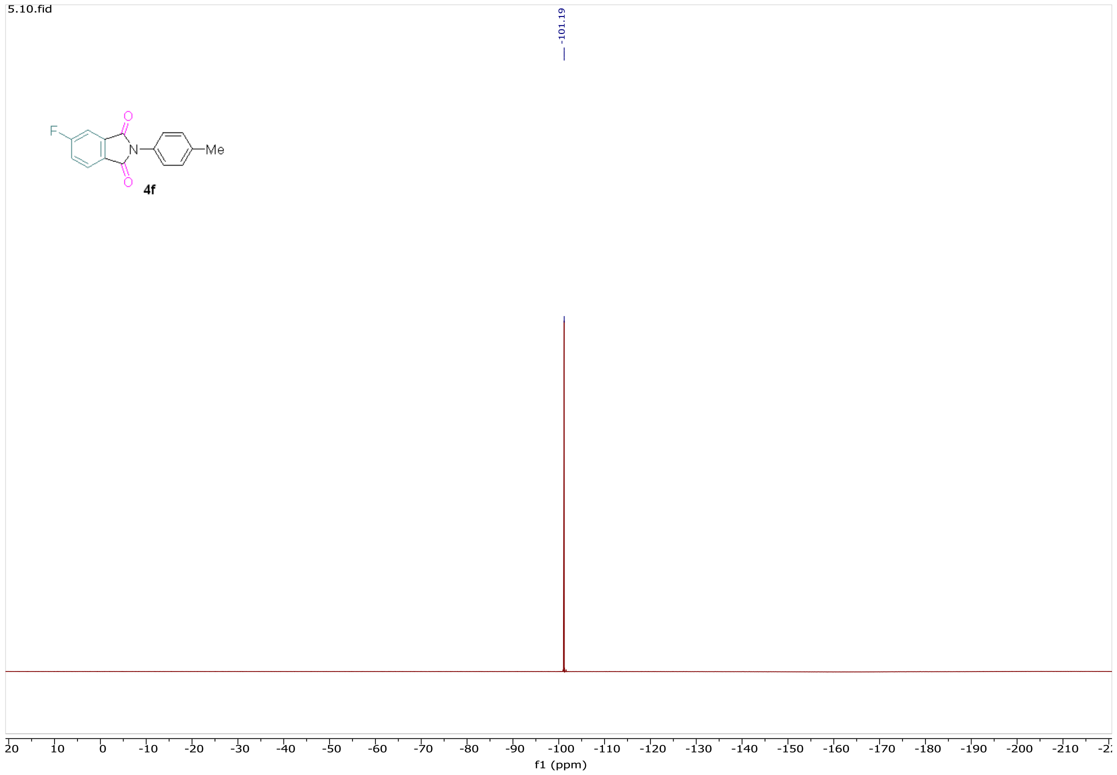
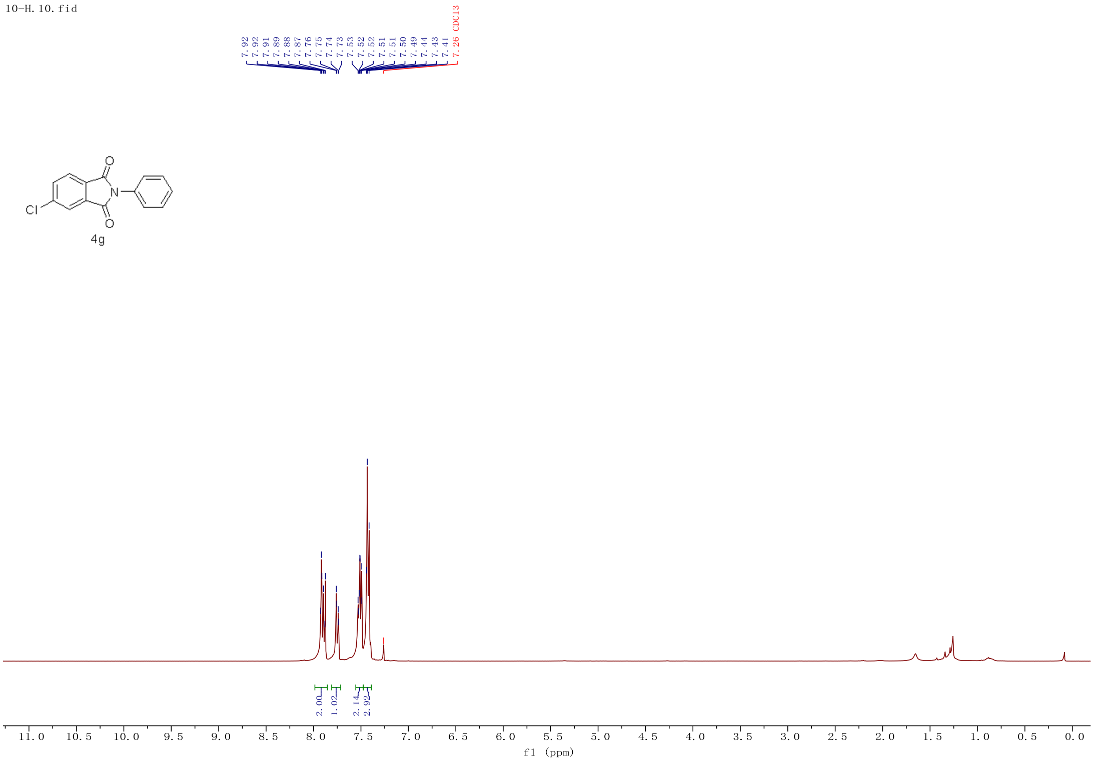
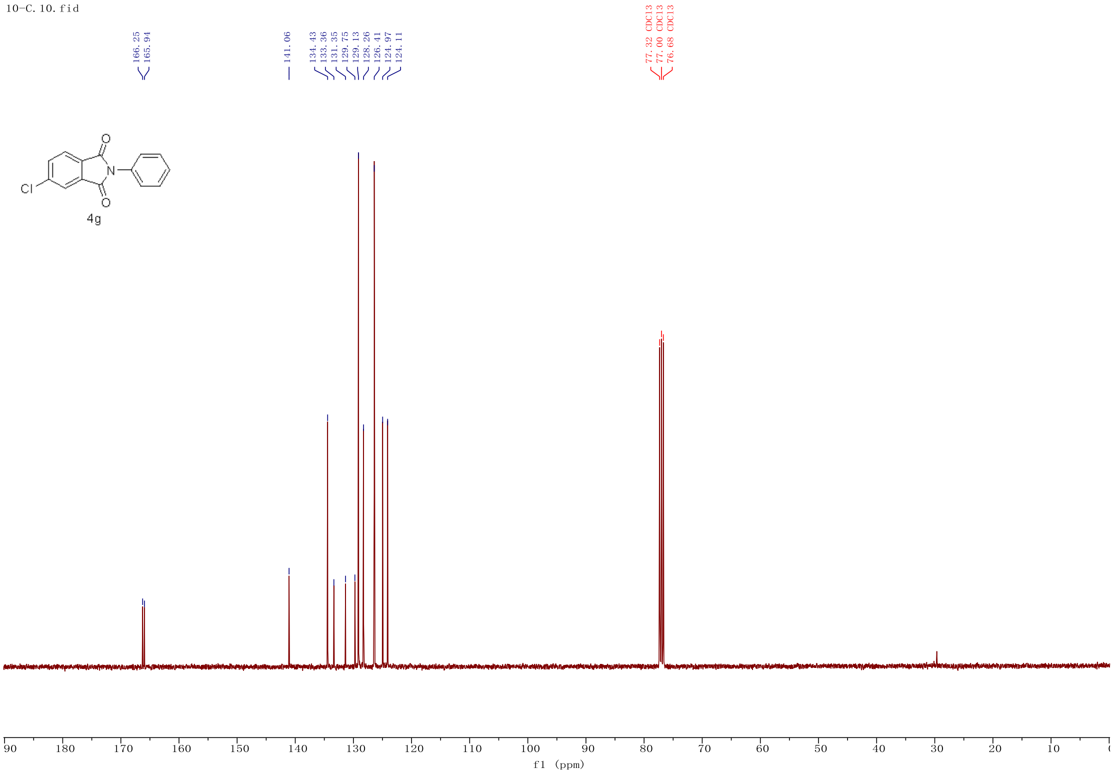
7.90
7.89
7.88
7.59
7.58
7.57
7.56
7.55
7.43
7.42
7.40
7.39
7.38
7.37
7.29
7.28
7.26 CDCl3
7.26
7.23
7.22
2.37
F
O
N
O
4f
0.98
0.97
1.02
3.84
3.19
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


11-C.10.fid
167.78
166.31
166.01
165.99
165.22
138.34
134.64
134.55
129.78
128.72
127.53
127.50
126.30
126.10
126.01
121.46
121.23
111.51
111.26
77.32 CDCl3
77.00 CDCl3
76.68 CDCl3
21.17
F
O
N
O
4f
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


5.10.fid
-101.19
F
O
N
Me
O
4f
20
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)


10-H.10.fid
7.92
7.92
7.91
7.89
7.88
7.87
7.76
7.75
7.74
7.73
7.53
7.52
7.52
7.51
7.51
7.50
7.49
7.44
7.43
7.41
7.26 CDCl3
Cl
O
N
O
4g
2.00
1.02
2.14
2.92
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
f1 (ppm)


10-C.10.fid
166.25
165.94
141.06
134.43
133.36
131.35
129.75
129.13
128.26
126.41
124.97
124.11
77.32 CDCl3
77.00 CDCl3
76.68 CDCl3
Cl
O
N
O
4g
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)