Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Supporting Information For

Samarium(II)-Electrocatalyzed Chemoselective Reductive Alkoxylation of Phthalimides

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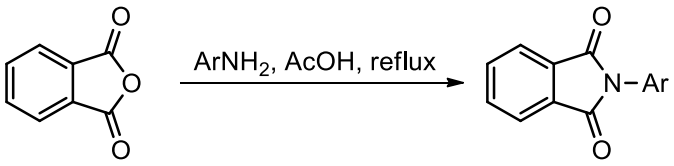
1. General Experimental Considerations.

THF was distilled from sodium metal/benzophenone before use. All commercially available chemicals were used without purification. ^{1}H NMR and ^{13}C NMR spectra were recorded on either a Bruker AM 360 (360 MHz), AM 300 (300 MHz), or AM 250 (250 MHz) instrument with samples dissolved in $CDCl_3$. ^{1}H NMR chemical shifts were referenced to the residual solvent signal; ^{13}C NMR chemical shifts were referenced to the deuterated solvent signal. Data are represented as follows: chemical shift δ (ppm), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad), coupling constant (*J*) in Hz and integration. Mass spectra were recorded on a micrOTOF-q Brucker Daltonics spectrometer. Flash chromatography (FC) was performed on 40-63 μm silica gel with mixtures of ethyl acetate (EA) and pentane. TLC plates were visualized by exposure to UV (254 nm) and/or $KMnO_4$ stain. The gas chromatography (GC) were performed on a spectrometer Varian GC-430 (injection: split/splitless, FID detector, column VF1-MS: 15m x 0.25 mm x 0.25 microns, program: 1 min 50 °C, 10 °C/min to 250 °C, 250 °C 2 min, 23 min total). Infrared spectra were recorded on a FTIR spectrometer (Perkin-Elmer spectrum one, NaCl pellets or Bruker Vertex 70 ATR Pike Germanium) and are reported in cm^{-1}. Melting points were determined using a Büchi melting point apparatus. Electrolysis were performed with a AUTOLAB potentiostat/galvanostat (model: PGSTAT302N), in an undivided three-electrodes cell containing samarium rod working electrode, a standard glassy carbon counter electrode and a saturated calomel electrode (SCE) as reference. The samarium electrode used is based on a samarium rod of 12.7 mm (0.5 in) diameter and 5 cm length, directly connected to a copper wire to ensure current conductivity. This self-made electrode is stored under inert atmosphere when it is not used. All the samarium rods are purchased from Alfa Aesar (99.9 % metals basis excluding Ta).

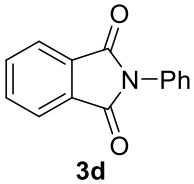
2. Preparation of the substrates

2.1. General procedure for 3d and 3e. [1]



A mixture of aniline (5.0 mmol) and Phthalic anhydride (740 mg, 5.0 mmol) in acetic acid (10 ml) was refluxed overnight with stirring. After cooling to the room temperature, diluted by water (20 ml) and extracted with ethyl acetate (2x10 ml). The organic phase was washed by saturated $NaHCO_3$ (5 ml) and brine (5 ml), dried over $MgSO_4$, concentrated and purified by flash chromatography on silica gel to afford the product.

2-phenylisoindoline-1,3-dione (3d)



3d

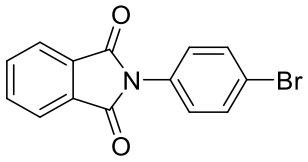
Following the general procedure, the product was obtained as a colorless solid (1.06 g, 95%) using ethyl acetate/pentane: 1:5 as eluent.

^{1}H NMR (360 MHz, $CDCl_3$)δ8.01 – 7.93 (m, 2H), 7.86 – 7.72 (m, 2H), 7.60 – 7.36 (m, 5H).

^{13}C NMR (90 MHz, $CDCl_3$)δ 167.3, 134.4, 131.8, 131.7, 129.2, 128.1, 126.6, 123.8.

HRMS (ESI) calculated for $C_{14}H_9NNaO_2^+$ [M+Na]: 246.0525. Found: 246.0534.

2-(4-bromophenyl)isoindoline-1,3-dione (3e)



3e

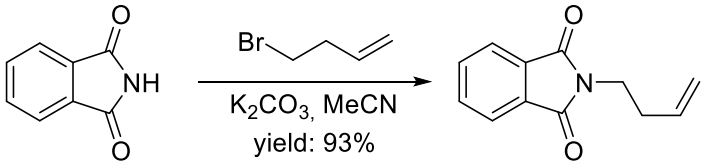
Following the general procedure, the product was obtained as a colorless solid (1.38 g, 92%) using ethyl acetate/pentane: 1:5 as eluent.

^{1}H NMR (360 MHz, $CDCl_3$) δ 7.97 (dd, *J* = 5.4, 3.1 Hz, 2H), 7.82 (dd, *J* = 5.4, 3.1 Hz, 2H), 7.65 (d, *J* = 8.7 Hz, 2H), 7.37 (d, *J* = 8.7 Hz, 2H).

^{13}C NMR (90 MHz, $CDCl_3$) δ 166.9, 134.6, 132.3, 131.6, 130.7, 128.0, 123.9, 121.8.

HRMS (ESI) calculated for $C_{14}H_8BrNNaO_2^+$ [M+Na]: 323.9631 and 325.9611. Found: 323.9621 and 325.9626.

2-(but-3-en-1-yl)isoindoline-1,3-dione (3g)[2]



3g

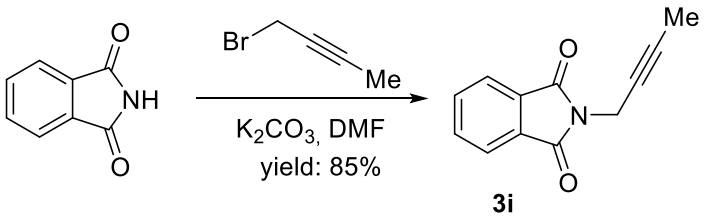
To a solution of K_2CO_3 (2.07 g, 15.0 mmol) and phthalimide (0.89 g, 6.0 mmol) in MeCN (15.0 ml), 4-bromobut-1-ene (0.61 g, 5.0 mmol, 0.43 ml) was added dropwise with stirring at room temperature. The resulting reaction was refluxed for 13 h. Then, cooled to room temperature and quenched by water (50 ml), extracted with ethyl acetate (2x15 ml). The organic phase was washed by 0.1 mol/L HCl (5 ml) and brine (5 ml), dried over $MgSO_4$, concentrated and purified by flash chromatography on silica gel (eluent: ethyl acetate/pentane: 1:5) to afford the product as a colorless solid (1.12g, 93%).

^{1}H NMR (360 MHz, $CDCl_3$) δ 7.82 (dd, *J* = 5.4, 3.1 Hz, 2H), 7.75 – 7.64 (m, 2H), 5.78 (ddt, *J* = 17.1, 10.1, 6.9 Hz, 1H), 5.04 (ddd, *J* = 14.8, 7.2, 5.9 Hz, 2H), 3.75 (t, *J* = 7.1 Hz, 2H), 2.43 (q, *J* = 7.0 Hz, 2H).

^{13}C NMR (90 MHz, $CDCl_3$) δ 168.4, 134.5, 134.0, 132.1, 123.3, 117.6, 37.4, 32.9.

HRMS (ESI) [$M+Na^+$] calculated for $C_{12}H_{11}NNaO_2^+$: 224.0682. Found: 224.0679.

2-(but-2-yn-1-yl)isoindoline-1,3-dione (3i)[3]



3i

To a solution of K_2CO_3 (1.73 g, 12.5 mmol) and phthalimide (0.74 g, 5.0 mmol) in DMF (5.0 ml), 1-bromobut-2-yne (1.06 g, 10.0 mmol, 0.70 ml) was added dropwise with stirring at room temperature overnight. Then, quenched by water (50 ml), extracted with ethyl acetate (2x15

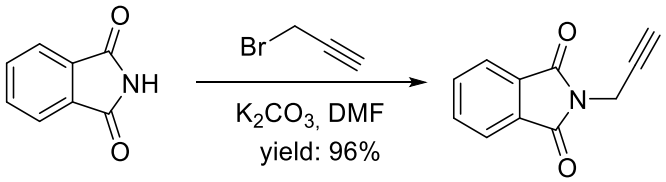
ml). The organic phase was washed by 0.1 mol/L HCl (5 ml) and brine (5 ml), dried over $MgSO_4$, concentrated and purified by flash chromatography on silica gel (eluent: ethyl acetate/pentane: 1:5) to afford the product as a yellow solid (0.85g, 85%).

^{1}H NMR (360 MHz, $CDCl_3$) δ 7.88 (dd, *J* = 5.3, 3.1 Hz, 2H), 7.74 (dd, *J* = 5.3, 3.0 Hz, 2H), 4.41 (d, *J* = 2.3 Hz, 2H), 1.78 (t, *J* = 2.1 Hz, 3iH).

^{13}C NMR (90 MHz, $CDCl_3$) δ 167.3, 134.1, 132.1, 123.5, 79.3, 72.6, 27.4, 3.5.

HRMS (ESI) [M+Na^+] calculated for $C_{12}H_9NNaO_2^+$: 222.0525. Found: 222.0532.

2-(prop-2-yn-1-yl)isoindoline-1,3-dione (3j)[4]



3j

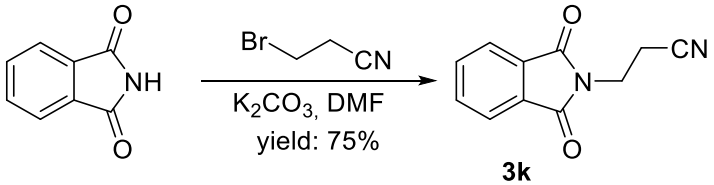
To a solution of K_2CO_3 (1.73 g, 12.5 mmol) and phthalimide (0.74 g, 5.0 mmol) in DMF (5.0 ml), 3-bromoprop-1-yne (1.19 g, 10.0 mmol, 0.86 ml) was added dropwise with stirring at room temperature overnight. Then, quenched by water (50 ml), extracted with ethyl acetate (2x15 ml). The organic phase was washed by 0.1 mol/L HCl (5 ml) and brine (5 ml), dried over $MgSO_4$, concentrated and purified by flash chromatography on silica gel (eluent: ethyl acetate/pentane: 1:5) to afford the product as a colorless solid (0.89g, 96%).

^{1}H NMR (360 MHz, $CDCl_3$) δ 7.87 (dd, *J* = 5.4, 2.9 Hz, 2H), 7.74 (dd, *J* = 5.4, 2.9 Hz, 2H), 4.45 (d, *J* = 2.3 Hz, 2H), 2.24 (d, *J* = 2.4 Hz, 1H).

^{13}C NMR (90 MHz, $CDCl_3$) δ 167.0, 134.2, 131.9, 123.6, 71.5, 27.0.

HRMS (ESI) [M+Na^+] calculated for $C_{11}H_7NNaO_2^+$: 208.0369. Found: 208.0371.

3-(1,3-dioxoisoindolin-2-yl)propanenitrile (3k)



3k

To a solution of K_2CO_3 (2.07 g, 15.0 mmol) and phthalimide (0.74 g, 5.0 mmol) in DMF (10.0 ml), 3-bromopropanenitrile (0.80 g, 6.0 mmol, 0.50 ml) was added dropwise with stirring at

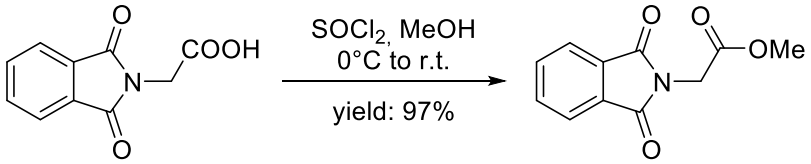
room temperature overnight. Then, quenched by water (50 ml), extracted with ethyl acetate (2x15 ml). The organic phase was washed by 0.1 mol/L HCl (5 ml) and brine (5 ml), dried over $MgSO_4$, concentrated and purified by flash chromatography on silica gel (eluent: ethyl acetate/pentane: 1:5) to afford the product as a yellow oil (0.75g, 75%).

^{1}H NMR (360 MHz, $CDCl_3$) δ 7.87 (dd, *J* = 5.4, 3.0 Hz, 1H), 7.76 (dd, *J* = 5.5, 3.1 Hz, 1H), 4.01 (t, *J* = 6.9 Hz, 1H), 2.81 (t, *J* = 6.9 Hz, 1H).

^{13}C NMR (90 MHz, $CDCl_3$) δ 167.6, 134.6, 131.7, 123.8, 117.0, 33.6, 17.3.

HRMS (ESI) [M+Na^+] calculated for $C_{11}H_8N_2NaO_2^+$: 223.0478. Found: 223.0465.

Methyl 2-(1,3-dioxoisoindolin-2-yl)acetate (3l)



3l

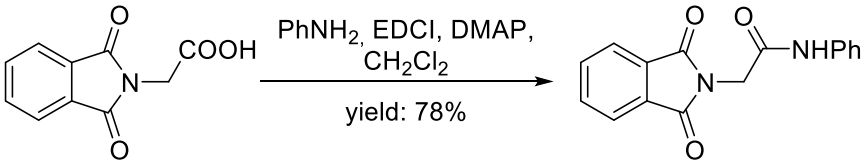
2-(1,3-dioxoisoindolin-2-yl)acetic acid(0.62 g, 3.0 mmol) was dissolved in MeOH (30 ml), then cooled to 0 ˚C. $SOCl_2$ (0.54 g, 4.5 mmol, 0.33 ml) was added into the mixture, warmed up to room temperature and stirred overnight. Quenched by saturated $NaHCO_3$ (10 ml), extracted with ethyl acetate (2x15 ml). The organic phase was washed by brine (5 ml), dried over $MgSO_4$, concentrated and purified by flash chromatography on silica gel (eluent: ethyl acetate/pentane: 1:2) to afford the product as a white solid (0.64 g, 97%).

^{1}H NMR (360 MHz, $CDCl_3$) δ 7.87 (dd, *J* = 5.1, 3.1 Hz, 2H), 7.74 (dd, *J* = 5.2, 3.1 Hz, 2H), 4.44 (s, 2H), 3.75 (s, 3H).

^{13}C NMR (90 MHz, $CDCl_3$) δ 167.7, 167.4, 134.3, 132.0, 123.6, 52.7, 38.7.

HRMS (ESI) [M+Na^+] calculated for $C_{11}H_9NNaO_4^+$: 242.0424. Found: 242.0421.

2-(1,3-dioxoisoindolin-2-yl)-N-phenylacetamide (3m)



3m

To a solution of 2-(1,3-dioxoisoindolin-2-yl)acetic acid (1.03 g, 5.0 mmol) and aniline (0.47 g, 5.0 mmol, 0.46 ml) in CH_2Cl_2 (100 ml), DMAP (61 mg, 0.5 mmol)and EDCI (1.15 g, 6.0 mmol) were added with stirring at room temperature. After 20 hours, concentrated and purified by

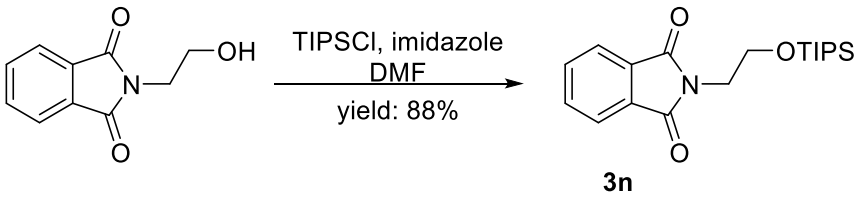
flash chromatography on silica gel (eluent: ethyl acetate/pentane: 1:1) to afford the product as a white solid (1.09 g, 78%).

^{1}H NMR (360 MHz, $CDCl_3$) δ 7.95 (dd, *J* = 5.2, 3.1 Hz, 2H), 7.80 (dd, *J* = 5.4, 3.0 Hz, 2H), 7.62 (s, 1H), 7.53 (d, *J* = 8.0 Hz, 2H), 7.34 (t, *J* = 7.7 Hz, 2H), 7.16 (d, *J* = 7.2 Hz, 1H), 4.55 (s, 2H).

^{13}C NMR (63 MHz, $CDCl_3$) δ 167.9, 138.9, 134.6, 131.8, 130.0, 129.7, 128.9, 128.8, 123.9, 61.7.

HRMS (ESI) [M+Na^+] calculated for $C_{16}H_{12}N_2NaO_3^+$: 303.0740. Found: 303.0732.

2-(2-((triisopropylsilyl)oxy)ethyl)isoindoline-1,3-dione (3n)



3n

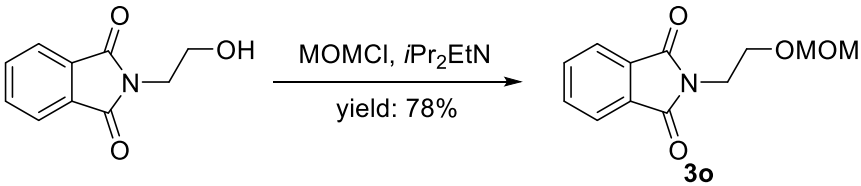
2-(2-hydroxyethyl)isoindoline-1,3-dione (0.96 g, 5.0 mmol) and imidazole (0.85g, 12.5 mmol) were added in DMF (10 ml), stirred at room temperature for 10 min, then TIPSCl (1.93 g, 10.0 mmol, 2.14 ml) was added dropwise into the mixture. After 15 h, the reaction was quenched by saturated NH_4Cl (20 ml), extracted with ethyl acetate (2x15 ml). The organic phase was washed by brine (5 ml), dried over $MgSO_4$, concentrated and purified by flash chromatography on silica gel (eluent: ethyl acetate/pentane: 1:20) to afford the product as a colorless solid (1.53 g, 88%).

^{1}H NMR (360 MHz, $CDCl_3$) δ 7.88 (dd, *J* = 5.3, 3.1 Hz, 2H), 7.74 (dd, *J* = 5.4, 3.0 Hz, 2H), 4.02 – 3.82 (m, 4H), 1.09 – 0.93 (m, 21H).

^{13}C NMR (90 MHz, $CDCl_3$) δ 168.4, 133.9, 132.2, 123.2, 60.3, 40.2, 17.8, 11.9.

HRMS (ESI) [M+Na^+] calculated for $C_{19}H_{29}NNaO_3Si^+$: 370.1809. Found: 370.1794.

2-(2-(methoxymethoxy)ethyl)isoindoline-1,3-dione (3o)



3o

2-(2-hydroxyethyl)isoindoline-1,3-dione (0.96 g, 5.0 mmol) and Chloromethyl methyl ether (0.81g, 10.0 mmol, 0.76 ml) were added in *i*Pr_2EtN (10 ml), stirred at room temperature overnight. Quenched by saturated $NaHCO_3$ (20 ml), extracted with ethyl acetate (2x15 ml). The organic phase was washed by brine (5 ml), dried over $MgSO_4$, concentrated and purified by

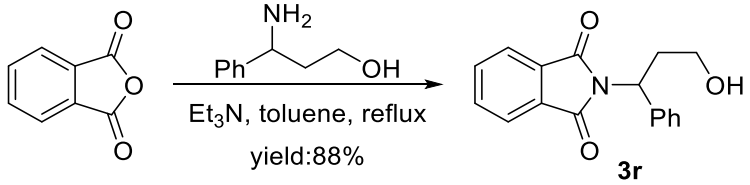
flash chromatography on silica gel (eluent: ethyl acetate/pentane: 1:5) to afford the product as a yellow solid (0.92 g, 78%).

^{1}H NMR (360 MHz, $CDCl_3$) δ 7.94 – 7.79 (m, 2H), 7.79 – 7.60 (m, 2H), 4.61 (d, *J* = 1.7 Hz, 2H), 3.93 (td, *J* = 5.8, 1.2 Hz, 2H), 3.85 – 3.67 (m, 2H), 3.30 (d, *J* = 1.9 Hz, 3H).

^{13}C NMR (90 MHz, $CDCl_3$) δ 168.3, 134.0, 132.1, 123.3, 96.2, 64.3, 55.3, 37.7.

HRMS (ESI) [M+Na^+] calculated for $C_{12}H_{13}NNaO_4^+$: 258.0737. Found: 258.0744.

2-(3-hydroxy-1-phenylpropyl)isoindoline-1,3-dione (3r)



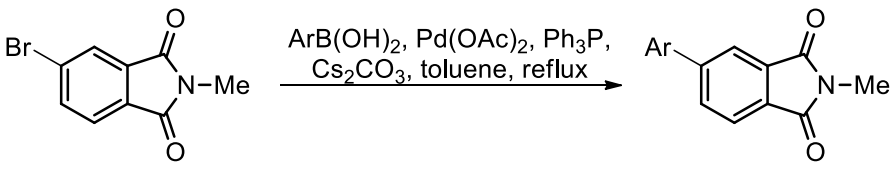
A mixture of 3-amino-3-phenylpropan-1-ol (0.76 g, 5.0 mmol), Phthalic anhydride (0.74 g, 5.0 mmol), trimethylamine (51 mg, 0.5 mmol, 0.06 ml) and toluene (7.5 ml) in a flask fitted with a Dean-Stark tube was heated under reflux for 3 h. Cooled to room temperature, removed the solvent by evaporator. The residue was dissolved with ethyl acetate (20 ml) and washed by 2N HCl (5 ml), sat. $NaHCO_3$ (5 ml) and brine (5 ml). The organic phase was dried over $MgSO_4$, concentrated and purified by flash chromatography on silica gel (eluent: ethyl acetate/pentane: 1:2) to afford the product as a yellow solid (1.24 g, 88%).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.79 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.73 – 7.62 (m, 2H), 7.62 – 7.51 (m, 2H), 7.37 – 7.21 (m, 3H), 5.62 (dd, *J* = 9.9, 6.2 Hz, 1H), 3.71 (dt, *J* = 7.4, 5.3 Hz, 2H), 2.95 – 2.72 (m, 1H), 2.63 – 2.46 (m, 1H), 2.25 (s, 1H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 168. 6, 139.2, 134.0, 131.8, 128.6, 128.2, 127.9, 123.3, 59.7, 51.5, 33.6.

HRMS (ESI) [M+Na^+] calculated for $C_{17}H_{15}NNaO_3^+$: 304.0944. Found: 304.0947.

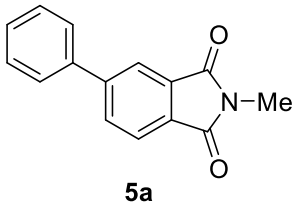
2.2. General procedure for 5a, 5b and 5c.



To a solution of 4-bromo-*N*-methylphthalimide(0.48 g, 2.0 mmol),organoborane (1.5 eq, 3.0 mmol), $Pd(OAc)_2$ (22.5 mg, 0.1 mmol), PPh_3 (105.2 mg, 0.4 mmol) and Cs_2CO_3 (1.3 g, 4.0

mmol) in toluene (10 mL), changed the air into argon, the mixture was refluxed overnight. When the reaction was complete, cooled to room temperature, diluted by distillated water (10 ml) and extracted with CH_2Cl_2 (3x 5 ml). The combined organic layers were washed by brine, dried over $MgSO_4$ and concentrated. The crude product was purified by silica gelcolumn chromatography to afford the corresponding ring-substituted*N*-methylphthalimide derivative.

2-methyl-5-phenylisoindoline-1,3-dione (5a)



5a

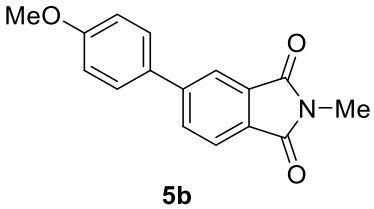
Following the general procedure, the product was obtained as a colorless solid (418 mg, 88%). (eluent: ethyl acetate/pentane: 1:3);

^{1}H NMR (250 MHz, $CDCl_3$) δ 8.06 (s, 1H), 7.91 (s, 2H), 7.71 – 7.57 (m, 2H), 7.49 (dd, *J* = 8.3, 6.9 Hz, 3H), 3.22 (s, 3H).

^{13}C NMR (63 MHz, $CDCl_3$) δ 168.4 (2xC), 147.3, 139.1, 133.1, 132.4, 130.7, 129.2, 128.8, 127.3, 123.6, 121.8, 24.0.

HRMS (ESI) [M+Na^+] calculated for $C_{15}H_{11}NNaO_2^+$: 260.0682. Found: 260.0677.

5-(4-methoxyphenyl)-2-methylisoindoline-1,3-dione (5b)



5b

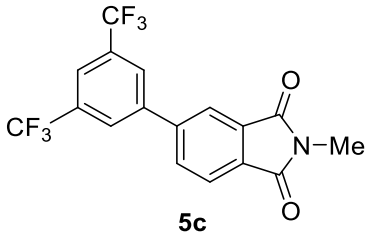
Following the general procedure, the product was obtained as a colorless solid (284 mg, 53%). (eluent: ethyl acetate/pentane: 1:3);

^{1}H NMR (250 MHz, $CDCl_3$) δ 7.98 (s, 1H), 7.84 (d, *J* = 1.0 Hz, 2H), 7.57 (d, *J* = 8.8 Hz, 2H), 7.01 (d, *J* = 8.8 Hz, 2H), 3.87 (s, 3H), 3.19 (s, 3H).

^{13}C NMR (63 MHz, $CDCl_3$) δ 168.5, 168.4, 160.3, 146.8, 133.1, 131.6, 131.3, 129.9, 128.4, 123.6, 121.1, 114.6, 55.4, 23.9.

HRMS (ESI) [M+Na^+] calculated for $C_{16}H_{13}NNaO_3^+$: 290.0788. Found: 290.0784.

5-(3,5-bis(trifluoromethyl)phenyl)-2-methylisoindoline-1,3-dione (5c)



5c

Following the general procedure, the product was obtained as a colorless solid (618 mg, 83%). (eluent: ethyl acetate/pentane: 1:5);

^{1}H NMR (360 MHz, $CDCl_3$) δ 8.12–8.06 (m, 3H), 8.02 – 7.94 (m, 3H), 3.25 (s, 3H).

^{13}C NMR (90 MHz, $CDCl_3$) δ 167.8 (2C), 144.0, 141.2, 133.5, 132.8, 132.7 (q, *J*= 33.3 Hz), 132.1, 127.5 (d, *J*= 2.7 Hz), 124.1, 122.5 (quint, *J*= 3.6 Hz), 121.9, 121.6, 24.2.

HRMS (ESI) [M+Na^+] calculated for $C_{17}H_9F_6NNaO_2^+$: 396.0430. Found: 396.0412.

3. Cyclic voltammetry Experiments

Cyclic voltammetry measurements were realized employing an AUTOLAB potentiostat/galvanostat (model: PGSTAT302N). The working electrode was a glassy carbon electrode (Ø = 2mm). The electrode was polished before use. The auxiliary electrode was a platinum wire, and the reference electrode was a saturated calomel electrode (SCE). The scan rate for all the experiments was 100 $mV.s^{-1}$.

3.1. Cyclic voltammetry Experiment of $SmCl_3$ in THF

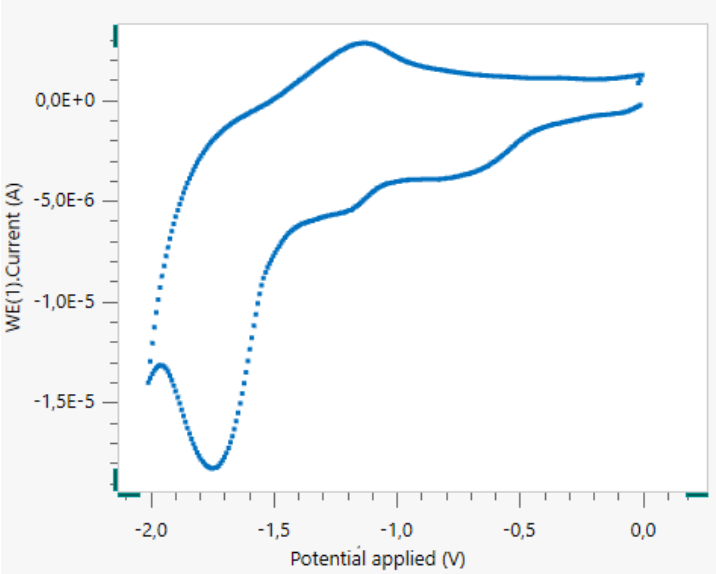


Figure S6: Cyclic voltammogram of $SmCl_3$ in THF solution with 0.1 M nBu$_4$NPF$_6$.

3.2. Cyclic voltammetry Experiment of *N*-methyl phtalimide in THF

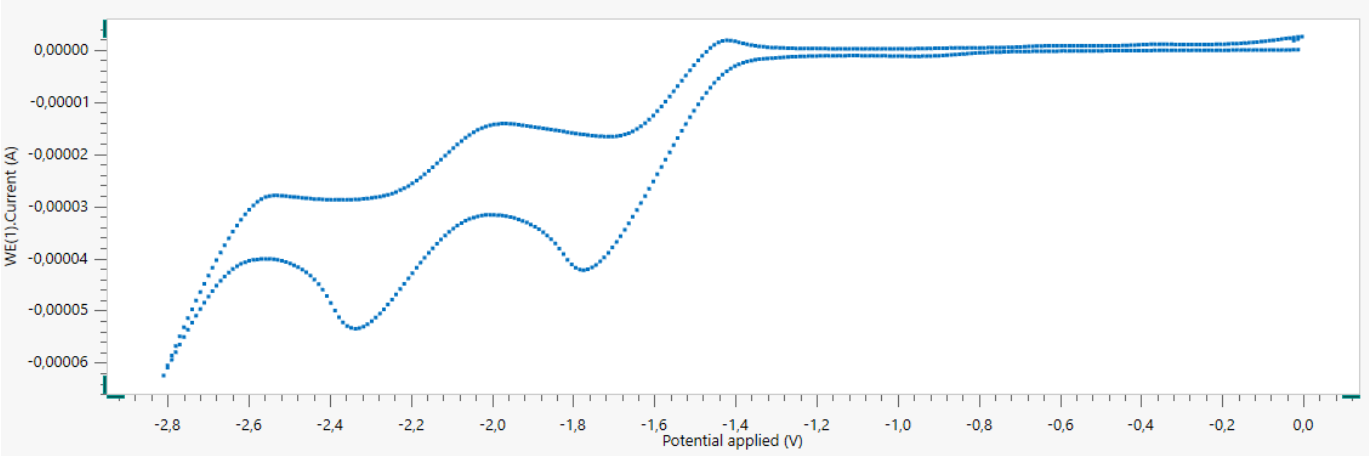


Figure S6: Cyclic voltammogram of *N*-methyl phtalimide in THF / 0.1 M nBu$_4$NPF$_6$. solution

3.3. Cyclic voltammetry Experiment to demonstrate the existence of catalytic current

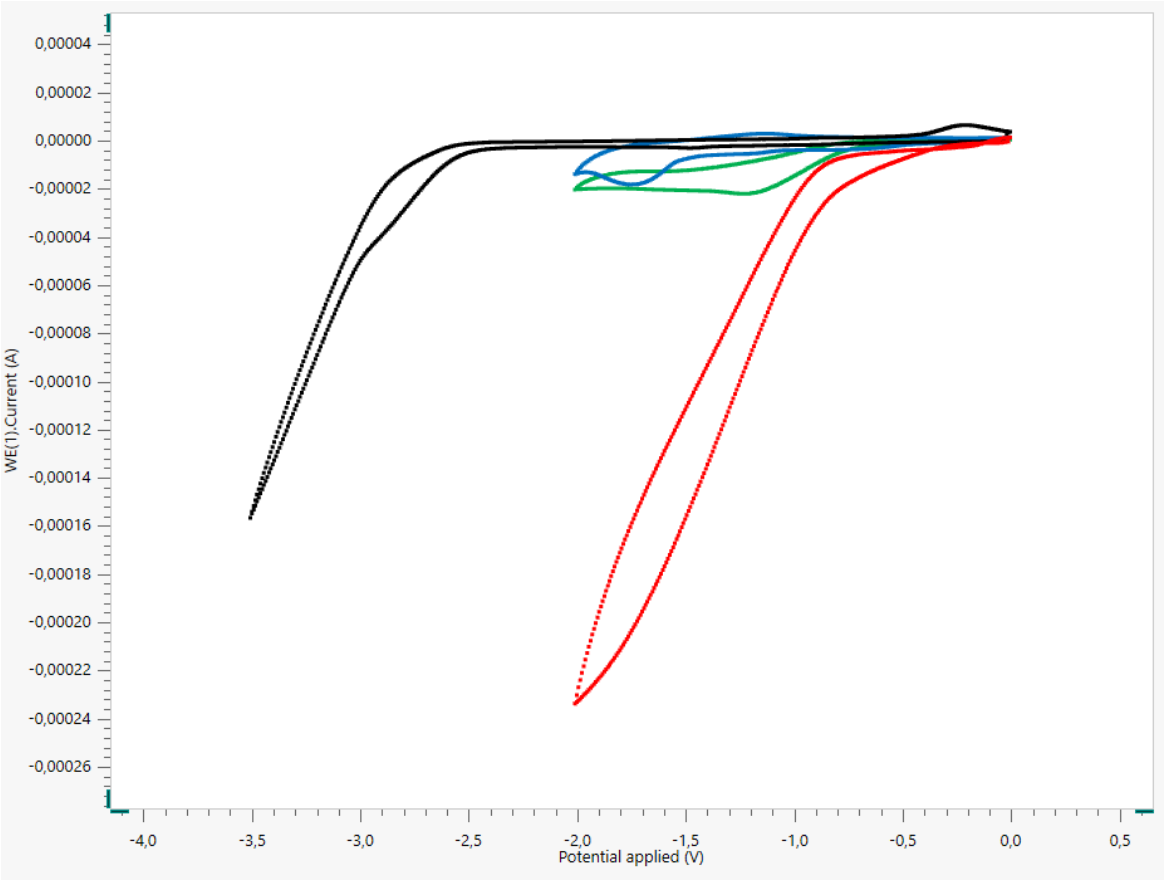


Figure S6: Electrochemical behavior of $SmCl_3$ in THF / 0.1 M nBu$_4$NPF$_6$ solution

Electrochemical behavior of $SmCl_3$ with *N*-methyl phtalimide **1.** Cyclic voltammetry performed using a GC electrode (20 mm^2) and a Pt wire as counter electrode with a scanning potential between 0 and - 2 V *vs*. SCE in THF with nBu$_4$NPF$_6$ [0.1 M]. Scan rate: 100 mV/s. Black curve: Blank realized with degassed THF / 0.1 M nBu$_4$NPF$_6$ solution. Blue curve: after introduction of 0.2 M $SmCl_3$. Green curve: after introduction of 1 mL of solution containing *N*-methyl phtalimide **1**/TMSCl/MeOH (10/8/1) in THF at 0.02 M concentration according the phtalimide. . Red curve: after introduction of 2 mL of *N*-methyl phtalimide **1**/TMSCl/MeOH (10/8/1)

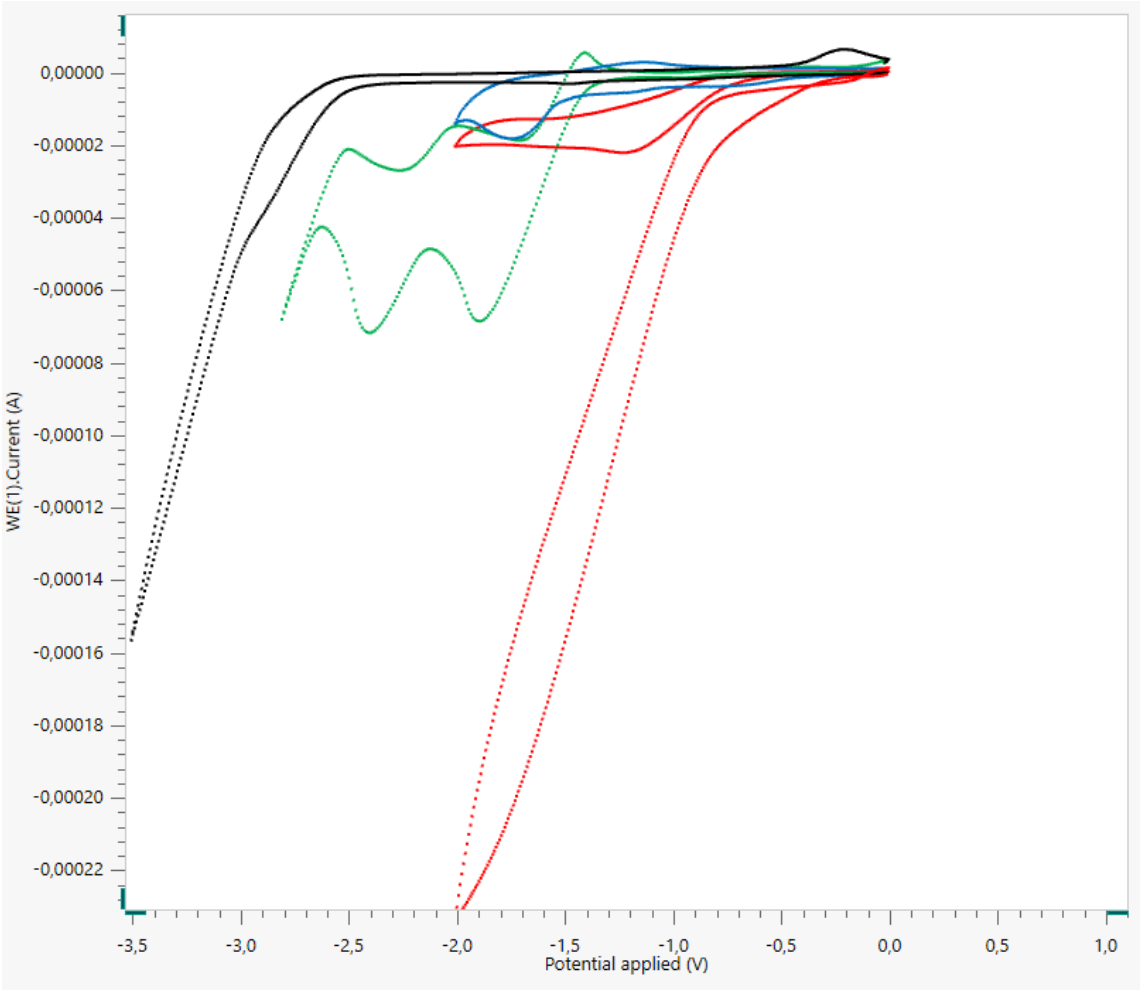
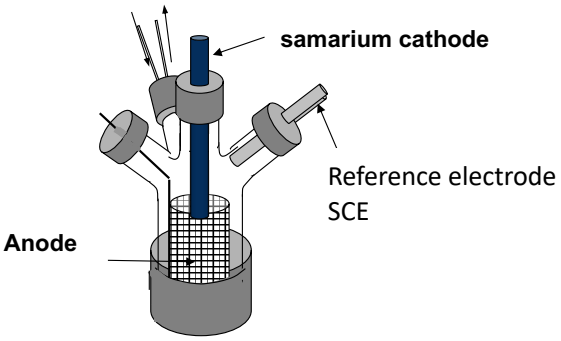


Figure S6: Electrochemical behavior of $SmCl_3$ in THF / 0.1 M nBu$_4$NPF$_6$ solution. The green curve corresponds to the reduction of phtalimide alone to compare.

3. Electrochemical set-up

The samarium electrode used is based on a samarium rod of 12.7mm (0.5in) diameter, directly connected to a copper wire to ensure current conductivity. This self-made electrode is stored under inert atmosphere when it is not used. All the samarium rods are purchased from Alfa Aesar (99.9% metals basis excluding Ta).



Samarium electrode

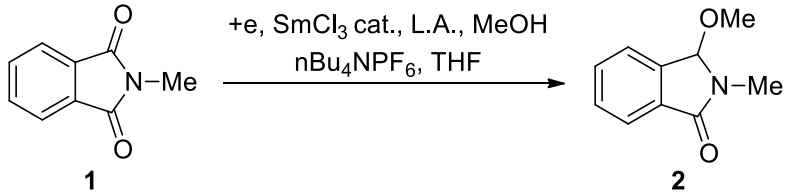
Scheme of the undivided electrochemical cell and picture of the samarium electrode used in this study.

Electrolysis was performed with a AUTOLAB potentiostat/galvanostat (model: PGSTAT302N), in an undivided three-electrodes cell containing samarium rod working electrode, a standard glassy carbon counter electrode and a saturated calomel electrode (SCE) as reference.

Electrolysis using Sm electrode as cathode and $SmCl_3$ to catalyze alkoxylation of phthalimide.

4. Characterization data of reductive alkoxylation of methyl phthalimide.

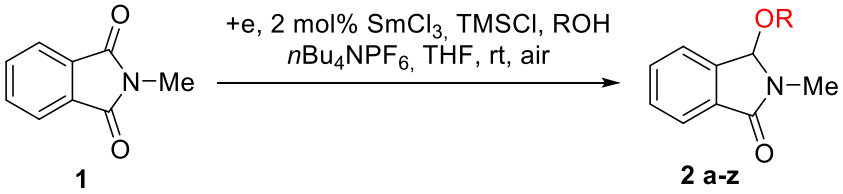
4.1. Optimization of reductive methoxylation of *N*-methyl phtalimide[a]



entry	$SmCl_3$ eq.	L. A. eq.	MeOH eq.	c mol/L	t h	conv. %	yield %[f]
1	10%	TMSCl 4.0	10	0.02[b]	2.0	66	60
2	10%	TMSOTf 4.0	10	0.02[b]	2.0	47	41
3	10%	$Et_2O{\cdot}BF_3$ 4.0	10	0.02[b]	2.0	<10	trace[e]
4	10%	$TiCl_4$ 4.0	10	0.02[b]	2.0	>99	0
5	10%	$SnCl_4$ 4.0	10	0.02[b]	2.0	>99	0
6	20%	TMSCl 4.0	10	0.02[b]	2.0	65	60
7	10%	TMSCl 8.0	10	0.02[b]	2.0	>99	89
8	10%	TMSCl 8.0	5	0.02[b]	2.0	45	41
9	10%	TMSCl 8.0	20	0.02[b]	2.0	>99	79
10	5%	TMSCl 8.0	10	0.02[b]	2.5	>99	96
11	2%	TMSCl 8.0	10	0.02[b]	2.5	>99	97
12	1%	TMSCl 8.0	10	0.02[b]	2.5	91	88
13	2%	TMSCl 1.0	10	0.1[c]	4.0	10	<5[e]
14	2%	TMSCl 2.0	10	0.1[c]	4.0	81	77
15	2%	TMSCl 3.0	10	0.1[c]	4.0	>99	97
16	2%	TMSCl 2.5	10	0.1[c]	4.0	>99	97
17	1%	TMSCl 2.5	10	0.1[c]	4.0	>99	93
18	no	TMSCl 2.5	10	0.1[c]	24	0	0
19	2%	no	10	0.1[c]	24	0	0
20[d]	2%	TMSCl 2.5	10	0.1[c]	24	0	0

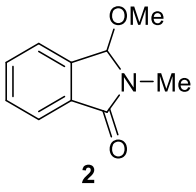
[a]standard conditions: 1 (1.0 mmol), $SmCl_3$ (0-0.1 equiv), MeOH (5-20 equiv), nBu_4NPF_6 (0.04 mol/L in THF) in THF, Sm as the cathode, carbon as the anode, I= 0.05 mA, r.t. in the air. [b]reaction in 50 ml THF. [c]reaction in 10 ml THF. [d]Without electricity. [e]Analyzed by GC. [f]Isolated yields.

4.2. General procedure for the reductive alkoxylation of methyl phthalimide.



Reactions were carried out in a three necked cell containing a magnetic stirring bar, samarium cathode (20 cm^2 area), glassy carbon anode (20 cm^2 area) and SCE as reference electrode. nBu_4NPF_6 (156 mg, 0.4 mmol, 0.04 mol/L in THF) as the electrolyte, methyl phthalimide (161 mg, 1.0 mmol), alcohol (10 equivalent, 10 mmol) and $SmCl_3$ (5.0 mg, 0.02 mmol)were added in anhydrous THF (10 ml), TMSCl (272 mg, 2.5 mmol, 0.32 ml) was added dropwise with stirring, then electrolysis was performed at i = 50 mA during 2.5 hours. After the reaction was complete, the mixture was quenched by 0.1 mol/L HCl (10 ml), extracted with ethyl acetate (2×20 mL). The combined organic layer was washed by 10% $Na_2S_2O_3$ (10 ml) and brine, dried over $MgSO_4$ and the solvent was removed under reduced pressure. The crude product was purified by flash chromatography on silica gel.

3-methoxy-2-methylisoindolin-1-one (2)



Following the general procedure, the product was obtained as a colorless oil (172 mg, 97%). (eluent: ethyl acetate/pentane: 1:3);

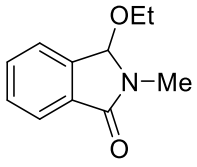
IR (film)ν_{max}: 3082, 3054, 2993, 2933, 2831, 1704, 1617, 1599, 1470, 1431, 1395, 1261, 1204, 1192, 1103, 1076, 1033, 746, 695 cm^{-1};

^{1}H NMR (300 MHz, $CDCl_3$)δ7.79 (dd, *J* = 6.3, 1.6 Hz, 1H), 7.60-7.43 (m, 3H), 5.73 (s, 1H), 3.05 (s, 3H,), 2.87 (s, 3H).

^{13}C NMR (75 MHz, $CDCl_3$)δ167.8 (C=O), 140.3 (C), 133.2 (C), 132.0 (CH), 130.00 (CH), 123.4 (2xCH), 88.0 (O-CH-N), 49.2 (O-CH_3), 26.5 (N-CH_3).

HRMS (ESI) [M+Na^+] calculated for $C_{10}H_{11}NNaO_2^+$: 200.0682. Found: 200.0689.

3-ethoxy-2-methylisoindolin-1-one (2a)



2a

Following the general procedure, the product was obtained as a colorless oil (180 mg, 94%). (eluent: ethyl acetate/pentane: 1:3);

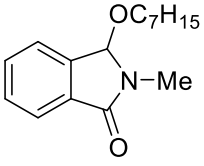
IR (film)ν_{max}: 2975, 2925, 2879, 1705, 1617, 1471, 1431, 1394, 1260, 1204, 1181, 1104, 1073, 1032 cm^{-1};

^{1}H NMR (360 MHz, $CDCl_3$)δ7.73 (d, *J* = 7.6 Hz, 1H), 7.55-7.39 (m, 3H), 5.68 (s, 1H), 3.02 (s, 3H,), 3.18-2.89 (m, 2H), 1.08 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (90 MHz, $CDCl_3$)δ167.5 (C=O), 141.0 (C), 133.0 (C), 131.9 (CH), 129.8 (CH), 123.2 (2xCH), 87.7 (O-CH-N), 57.8 (O-CH_2), 26.5 (N-CH_3), 15.1 (CH_3).

HRMS (ESI) [M+Na^+] calculated for $C_{11}H_{13}NNaO_2^+$: 214.0838. Found: 214.0843.

3-(heptyloxy)-2-methylisoindolin-1-one (2b)



2b

Following the general procedure, the product was obtained as a colorless oil (238 mg, 91%). (eluent: ethyl acetate/pentane: 1:3);

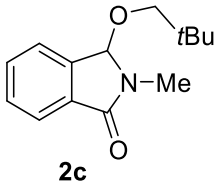
IR (film)ν_{max}: 2925, 2855, 1711, 1617, 1600, 1469, 1430, 1394, 1262, 1203, 1181, 1104, 1078, 1034, 745, 698 cm^{-1};

^{1}H NMR (360 MHz, $CDCl_3$)δ7.83 (d, *J* = 7.6 Hz, 1H), 7.62-7.49 (m, 3H), 5.78 (s, 1H), 3.16-3.06 (m, 1H), 3.11 (s, 3H,), 2.99-2.89 (m, 1H), 1.19-1.55 (m, 10H), 0.88 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (90 MHz, $CDCl_3$)δ167.6 (C=O), 141.0 (C), 133.1 (C), 131.9 (CH), 129.8 (CH), 123.3 (2xCH), 87.8 (O-CH-N), 62.2 (O-CH_2), 31.8 (CH_2), 29.6 (CH_2), 29.1 (CH_2), 26.6 (CH_3), 26.2 (CH_2), 22.6 (CH_2), 14.1(CH_3).

HRMS (ESI) [M+Na^+] calculated for $C_{16}H_{23}NNaO_2^+$: 284.1621. Found: 284.1616.

2-methyl-3-(neopentyloxy)isoindolin-1-one (2c)



2c

Following the general procedure, the product was obtained as a colorless oil (219 mg, 94%). (eluent: ethyl acetate/pentane: 1:3);

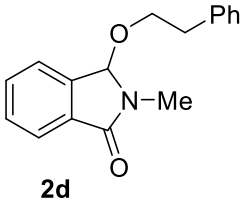
IR (film)ν_{max}: 2956, 2868, 1711, 1470, 1428, 1393, 1261, 1204, 1181, 1104, 1075, 1030, 746 cm^{-1};

^{1}H NMR (360 MHz, $CDCl_3$)δ7.78 (m, 1H), 7.57-7.42 (m, 3H), 5.75 (s, 1H), 3.05 (s, 3H), 2.71 (d, *J* = 10.1 Hz, 1H), 2.53 (d, *J* = 10.1 Hz, 1H), 0.85 (s, 9H).

^{13}C NMR (90 MHz, $CDCl_3$)δ167.7 (C=O), 141.1 (C), 133.1 (C), 131.9 (CH), 129.8 (CH), 123.4 (CH), 123.3 (CH), 87.8 (O-CH-N), 72.0 (O-CH_2), 31.5 (CH), 26.8 (3xCH_3), 26.7 (N-CH_3).

HRMS (ESI) [M+Na^+] calculated for $C_{14}H_{19}NNaO_2^+$: 256.1308. Found: 256.1307.

2-methyl-3-phenethoxyisoindolin-1-one (2d)



2d

Following the general procedure, the product was obtained as a colorless oil (259 mg, 97%). (eluent: ethyl acetate/pentane: 1:4);

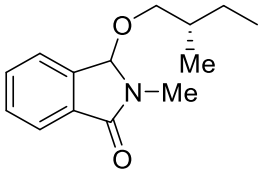
IR (film)ν_{max}: 3085, 3058, 3027, 2922, 2873, 1707, 1616, 1602, 1469, 1453, 1428, 1394, 1261, 1204, 1180, 1105, 1072, 1031, 746, 697 cm^{-1};

^{1}H NMR (360 MHz, $CDCl_3$)δ7.80 (dd, *J* = 6.3, 1.8 Hz, 1H), 7.55-7.45 (m, 2H), 7.35 (dd, *J* = 6.3, 1.8 Hz, 1H), 7.31-7.18 (m, 3H), 7.17-7.11 (m, 2H), 5.72 (s, 1H), 3.30 (dt, *J* = 9.4, 6.8 Hz, 1H), 3.17 (dt, *J* = 9.4, 6.8 Hz, 1H), 2.92 (s, 3H), 2.81 (t, *J* = 6.8 Hz, 2H).

^{13}C NMR (63 MHz, $CDCl_3$)δ167.6 (C=O), 140.7 (C), 138.7 (C), 133.0 (C), 131.9 (CH), 129.9 (CH), 129.0 (2xCH), 128.4 (2xCH), 126.4 (CH), 123.3 (2xCH), 87.8 (O-CH-N), 63.0 (O-CH_2), 36.2 (CH_2), 26.7 (N-CH_3).

HRMS (ESI) [M+Na^+] calculated for $C_{17}H_{17}NNaO_2^+$: 290.1151. Found: 290.1154.

2-methyl-3-((S)-2-methylbutoxy)isoindolin-1-one (2e)



2e

Following the general procedure, the product was obtained as a colorless oil (the mixture of two epimers: 228 mg, 98%). (eluent: ethyl acetate/pentane: 1:5);

IR (film)ν_{max}: 3055, 2960, 2925, 2873, 1702, 1617, 1600, 1466, 1430, 1395, 1262, 1203, 1182, 1105, 1070, 1034, 971, 798, 745, 699 cm^{-1};

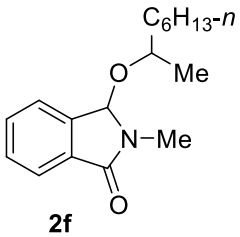
^{1}H NMR (360 MHz, $CDCl_3$) δ 7.79 (d, *J* = 7.6 Hz, 1H), 7.62 – 7.41 (m, 3H), 5.75 (s, 1H), 3.07 (s, 3H), 2.90 (ddd, *J* = 24.8, 8.8, 6.1 Hz, 1H), 2.72 (ddd, *J* = 21.4, 8.8, 6.2 Hz, 1H), 1.62 – 1.47 (m, 1H), 1.38 (dt, *J* = 12.9, 6.2 Hz, 1H), 1.15 – 0.98 (m, 1H), 0.94 – 0.74 (m, 6H).

^{13}C NMR (90 MHz, $CDCl_3$) δ 167.6 (C=O), 140.9 (C), 133.0 (C), 131.8 (CH), 129.7 (CH), 123.2 (2xCH), 87.7 (O-CH-N), 66.9 (O-CH_2), 66.8 (O-CH_2), 34.9 (CH_2), 34.8 (CH_2), 26.5 (N-CH_3), 26.1 (CH_2), 26.0 (CH_2), 16.6 (CH_2), 16.6 (CH_2), 11.3 (CH_3), 11.2 (CH_3).

HRMS (ESI) [M+Na^+] calculated for $C_{14}H_{19}NNaO_2^+$: 256.1308. Found: 256.1312.

d. r. = 1:1

2-methyl-3-(octan-2-yloxy)isoindolin-1-one (2f)



2f

Following the general procedure, the product was obtained as a colorless oil (the mixture of two epimers: 258 mg, 94%). (eluent: ethyl acetate/pentane: 1:3);

IR (film)ν_{max}: 2960, 2925, 2856, 1703, 1617, 1468, 1430, 1395, 1261, 1203, 1180, 1104, 1061, 1035, 746, 693 cm^{-1};

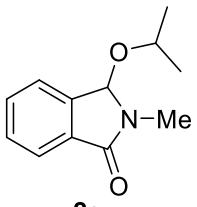
^{1}H NMR (360 MHz, $CDCl_3$) δ 7.79 (d, *J* = 7.4 Hz, 1H), 7.60 – 7.46 (m, 3H), 5.72 – 5.66 (m, 1H), 3.64 – 3.48 (m, 1H), 3.14 – 3.07 (m, 3H), 1.38 – 1.14 (m, 12H), 1.12 – 1.06 (m, 1H), 0.92 – 0.82 (m, 3H).

^{13}C NMR (90 MHz, $CDCl_3$) δ 167.6 (C=O), 167.4 (C=O), 142.4 (C), 141.9 (C), 132.6 (C), 132.5 (C), 131.7 (CH), 129.7 (CH), 123.4 (CH), 123.3 (CH), 123.2 (CH), 88.1 (O-CH-N), 87.9 (O-CH-N), 72.7 (O-CH_2), 72.6 (O-CH_2), 37.6 (CH_2), 31.8 (CH_2), 31.7 (CH_2), 29.4 (CH_2), 29.2 (CH_2), 27.0 (N-CH_3), 25.4 (CH_2), 25.2 (CH_2), 22.6 (CH_2), 21.5 (CH_3), 20.9 (CH_3), 14.1 (CH_3).

HRMS (ESI) [M+Na^+] calculated for $C_{17}H_{25}NNaO_2^+$: 298.1777. Found: 298.1773.

d. r. = 2:3

3-isopropoxy-2-methylisoindolin-1-one (2g)



2g

Following the general procedure, the product was obtained as a colorless oil (191 mg, 93%). (eluent: ethyl acetate/pentane: 1:3);

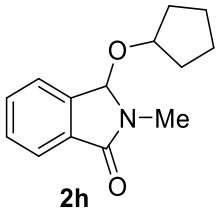
IR (film) ν_{max}: 2969, 2919, 2851, 1681, 1436, 1397, 1258, 1205, 1104, 1063 cm^{-1};

^{1}H NMR (360 MHz, $CDCl_3$)δ7.79 (d, *J* = 7.2 Hz, 1H), 7.60-7.45 (m, 3H), 5.69 (s, 1H), 3.65 (hept, *J* = 6.1 Hz, 1H), 3.11 (s, 3H,), 1.19 (d, *J* = 6.1 Hz, 3H), 1.09 (d, *J* = 6.1 Hz, 3H).

^{13}C NMR (90 MHz, $CDCl_3$)δ167.6 (C=O), 142.1 (C), 132.8 (C), 131.9 (CH), 129.9 (CH), 123.5 (CH), 123.4 (CH), 88.1 (O-CH-N), 68.3 (O-CH), 27.1 (N-CH_3), 23.9 (2xCH_3).

HRMS (ESI) [M+Na^+] calculated for $C_{12}H_{15}NNaO_2^+$: 228.0995. Found: 228.0994.

3-(cyclopentyloxy)-2-methylisoindolin-1-one (2h)



2h

Following the general procedure, under 3 hours electrolysis, the product was obtained as a colorless oil (168 mg, 73%). (eluent: ethyl acetate/pentane: 1:4);

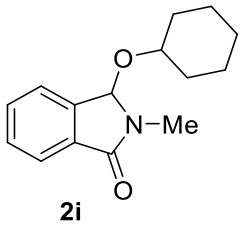
IR (film)ν_{max}: 2958, 2873, 1701, 1617, 1600, 1472, 1433, 1396, 1331, 1258, 1204, 1179, 1104, 1064, 1034, 746, 695 cm^{-1};

^{1}H NMR (360 MHz, $CDCl_3$)δ7.76 (d, *J* = 7.2 Hz, 1H), 7.60-7.43 (m, 3H), 5.67 (s, 1H), 3.78 (quint, *J* = 6.0 Hz, 1H), 3.06 (s, 3H), 1.76-1.56 (m, 4H), 1.55-1.30 (m, 4H).

^{13}C NMR (90 MHz, $CDCl_3$)δ167.7 (C=O), 142.1 (C), 132.8 (C), 131.8 (CH), 129.8 (CH), 123.6 (CH), 123.3 (CH), 88.0 (O-CH-N), 76.7 (O-CH_2), 33.7(2xCH_2), 26.9 (N-CH_3), 23.4 (CH_2), 23.2 (CH_2).

HRMS (ESI) [M+Na^+] calculated for $C_{14}H_{17}NNaO_2^+$: 254.1151. Found: 254.1154.

3-(cyclohexyloxy)-2-methylisoindolin-1-one (2i)



2i

Following the general procedure, under 3 hours electrolysis, the product was obtained as a colorless oil (178 mg, 73%). (eluent: ethyl acetate/pentane: 1:4);

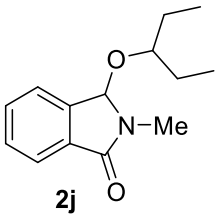
IR (film)ν_{max}: 2932, 2856, 1708, 1617, 1469, 1430, 1395, 1258, 1204, 1180, 1154, 1102, 1068, 1034, 748, 696 cm^{-1};

^{1}H NMR (360 MHz, $CDCl_3$)δ7.81 (d, *J* = 7.2 Hz, 1H), 7.60-7.43 (m, 3H), 5.73 (s, 1H), 3.32 (quint, *J* = 3.6 Hz, 1H), 3.12 (s, 3H), 1.90-1.58 (m, 4H), 1.54-1.04 (m, 6H).

^{13}C NMR (90 MHz, $CDCl_3$)δ167.7 (C=O), 142.4 (C), 132.8 (C), 131.9 (CH), 129.8 (CH), 123.5 (CH), 123.4 (CH), 88.1 (O-CH-N), 74.3 (O-CH_2), 34.0 (CH_2), 33.8 (CH_2), 27.1 (N-CH_3), 25.5 (CH_2), 24.3 (2xCH_2).

HRMS (ESI) [M+Na^+] calculated for $C_{15}H_{19}NNaO_2^+$: 268.1308. Found: 268.1310.

2-methyl-3-(pentan-3-yloxy)isoindolin-1-one (2j)



2j

Following the general procedure, under 3 hours electrolysis, the product was obtained as a colorless oil (142 mg, 61%). (eluent: ethyl acetate/pentane: 1:4);

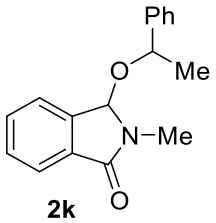
IR (film)ν_{max}: 2962, 2926, 2877, 2852, 1701, 1615, 1467, 1431, 1396, 1258, 1204, 1102, 1062, 1033, 747, 694 cm^{-1};

^{1}H NMR (360 MHz, $CDCl_3$) δ 7.81 (d, *J* = 7.3 Hz, 1H), 7.61 – 7.47 (m, 3H), 5.71 (s, 1H), 3.47 (p, *J* = 5.7 Hz, 1H), 3.14 (s, 3H), 1.72 – 1.59 (m, 4H), 0.95 – 0.89 (m, 6H).

^{13}C NMR (90 MHz, $CDCl_3$) δ 167.5 (C=O), 142.3 (C), 132.5 (C), 131.7 (CH), 129.6 (CH), 123.3 (2xCH), 88.2 (O-CH-N), 79.1 (O-CH), 27.2 (N-CH_3), 26.7 (CH_2), 26.1 (CH_2), 9.4 (CH_3), 9.0 (CH_3).

HRMS (ESI) [M+Na^+] calculated for $C_{14}H_{19}NNaO_2^+$: 256.1308. Found: 256.1306.

2-methyl-3-(1-phenylethoxy)isoindolin-1-one (2k)



2k

Following the general procedure, under 3 hours electrolysis, the product was obtained as a colorless oil (the mixture of two epimers: 216 mg, 81%). (eluent: ethyl acetate/pentane: 1:3);

IR (film)ν_{max}: 2977, 2925, 1694, 1647, 1472, 1430, 1395, 1052, 1034, 947, 797, 745, 701 cm^{-1};

Major product: **^{1}H NMR (360 MHz, $CDCl_3$)**δ 7.82 (dd, *J* = 6.3, 1.6 Hz, 1H), 7.54 (pd, *J* = 7.5, 1.3 Hz, 2H), 7.43 – 7.28 (m, 6H), 5.73 (s, 1H), 4.37 (q, *J* = 6.5 Hz, 1H), 2.72 (s, 3H), 1.43 (d, *J* = 6.5 Hz, 3H);

^{13}C NMR (90 MHz, $CDCl_3$)δ 167.5 (C=O), 143.4 (C), 141.5 (C), 132.7 (C), 131.9 (CH), 129.8 (CH), 128.6 (2xCH), 127.9 (CH), 126.4 (2xCH), 123.2 (2xCH), 88.2 (O-CH-N), 74.4 (O-CH), 27.0 (N-CH_3), 24.3(CH_3);

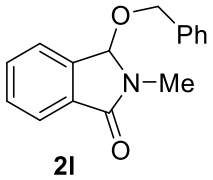
HRMS (ESI) [M+Na^+] calculated for $C_{17}H_{17}NNaO_2^+$: 290.1151. Found: 290.1151. Minor product: **^{1}H NMR (360 MHz, $CDCl_3$)**δ 7.77 (d, *J* = 7.6 Hz, 1H), 7.42 – 7.20 (m, 6H), 7.12 – 7.04 (m, 2H), 5.74 (s, 1H), 4.42 (q, *J* = 7.2 Hz, 1H), 3.10 (s, 3H), 1.47 (d, *J* = 6.5 Hz, 3H);

^{13}C NMR (90 MHz, $CDCl_3$) δ 167.4 (C=O), 143.8 (C), 141.4 (C), 132.3 (C), 132.0 (CH), 129.6 (CH), 128.5 (2xCH), 127.6 (CH), 126.1 (2xCH), 123.0 (2xCH), 87.5 (O-CH-N), 72.8 (O-CH), 26.9 (N-CH_3), 24.5 (CH_3);

HRMS (ESI) [M+Na^+] calculated for $C_{17}H_{17}NNaO_2^+$: 290.1151. Found: 290.1151.

d. r. = 1:3

3-(benzyloxy)-2-methylisoindolin-1-one (2l)



2l

Following the general procedure, the product was obtained as a colorless oil (238 mg, 94%). (eluent: ethyl acetate/pentane: 1:3);

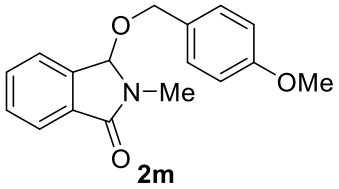
IR (film) ν_{max}: 3061, 3032, 2913, 1705, 1616, 1601, 1497, 1470, 1454, 1429, 1394, 1261, 1204, 1180, 1102, 1063, 1029, 749, 699 cm^{-1};

^{1}H NMR (360 MHz, $CDCl_3$)δ7.85 (d, *J* = 7.2 Hz, 1H), 7.60-7.49 (m, 3H), 7.35-7.21 (m, 5H), 5.87 (s, 1H), 4.12 (d, *J* = 10.8 Hz, 1H), 4.00 (d, *J* = 10.8 Hz, 1H), 3.10 (s, 3H).

^{13}C NMR (90 MHz, $CDCl_3$)δ167.6 (C=O), 140.6 (C), 137.2 (C), 133.0 (C), 132.0 (CH), 130.0 (CH), 128.5 (2xCH), 127.9 (CH), 127.8 (2xCH), 123.4 (2xCH), 87.8 (O-CH-N), 64.4 (O-CH_2), 26.7 (N-CH_3).

HRMS (ESI) [M+Na^+] calculated for $C_{16}H_{15}NNaO_2^+$: 276.0995. Found: 276.1002.

3-((4-methoxybenzyl)oxy)-2-methylisoindolin-1-one (2m)



2m

Following the general procedure, the product was obtained as a white solid (268 mg, 95%). (eluent: ethyl acetate/pentane: 1:4);

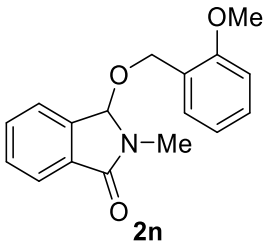
IR (film)ν_{max}: 3038, 3000, 2953, 2933, 2913, 2871, 2836, 1704, 1613, 1586, 1514, 1470, 1428, 1393, 1302, 1250, 1204, 1177, 1102, 1063, 1032, 821, 747, 695 cm^{-1};

^{1}H NMR (360 MHz, $CDCl_3$)δ7.82 (d, *J* = 7.2 Hz, 1H), 7.60-7.47 (m, 3H), 7.14 (d, *J* = 8.5 Hz, 2H), 6.83 (d, *J* = 8.5 Hz, 2H), 5.82 (s, 1H), 4.04 (d, *J* = 10.6 Hz, 1H), 3.93 (d, *J* = 10.6 Hz, 1H), 3.76 (s, 3H), 3.08 (s, 3H).

^{13}C NMR (63 MHz, $CDCl_3$)δ167.6 (C=O), 159.4(C), 140.8 (C), 133.0 (C), 132.0 (CH), 129.9 (CH), 129.5 (2xCH), 129.3 (C), 123.4 (CH), 123.4 (CH), 113.9 (2xCH), 87.8 (O-CH-N), 64.3 (O-CH_2), 55.3 (OCH_3), 26.7 (N-CH_3).

HRMS (ESI) [M+Na^+] calculated for $C_{17}H_{17}NNaO_3^+$: 306.1101. Found: 306.1108.

3-((2-methoxybenzyl)oxy)-2-methylisoindolin-1-one (2n)



2n

Following the general procedure, the product was obtained as a colorless oil (259 mg, 92%). (eluent: ethyl acetate/pentane: 1:4);

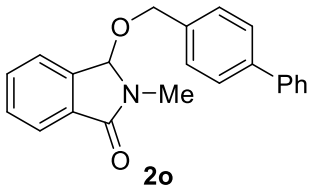
IR (KBr) ν_{max}: 3082, 3047, 3033, 3007, 2948, 2938, 2923, 2902, 2884, 2832, 2347, 1698, 1604, 1494, 1467, 1430, 1393, 1342, 1289, 1249, 1201, 1177, 1122, 1101, 1059, 1033, 753, 697 cm^{-1};

^{1}H NMR (360 MHz, $CDCl_3$)δ7.85 (d, *J* = 7.2 Hz, 1H), 7.59-7.48 (m, 3H), 7.33-7.22 (m, 2H), 6.96-6.89 (m, 1H), 6.83 (d, *J* = 8.3 Hz, 1H), 5.88 (s, 1H), 4.25 (d, *J* = 11.3 Hz, 1H), 4.05 (d, *J* = 11.3 Hz, 1H), 3.76 (s, 3H), 3.10 (s, 3H).

^{13}C NMR (90 MHz, $CDCl_3$)δ167.7 (C=O), 157.2(C), 140.9 (C), 133.1 (C), 131.9 (CH), 129.9 (CH), 129.6 (CH), 129.3 (CH), 125.6 (C), 123.5 (CH), 123.3 (CH), 120.5 (CH), 110.2 (CH), 88.0 (O-CH-N), 59.9 (O-CH_2), 55.2 (OCH_3), 26.6 (N-CH_3).

HRMS (ESI) [M+Na^+] calculated for $C_{17}H_{17}NNaO_3^+$: 306.1101. Found: 306.1105.

3-([1,1'-biphenyl]-4-ylmethoxy)-2-methylisoindolin-1-one (2o)



2o

Following the general procedure, the product was obtained as a white solid (303 mg, 96%). (eluent: ethyl acetate/pentane: 1:3);

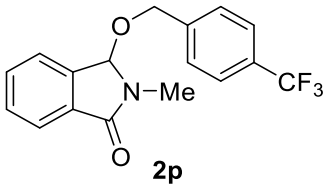
IR (KBr)ν_{max}: 3052, 3030, 2914, 2894, 2872, 2860, 1703, 1470, 1426, 1393, 1201, 1103, 1075, 1035, 825, 755, 694 cm^{-1};

^{1}H NMR (360 MHz, $CDCl_3$)δ7.91 (d, *J* = 7.2 Hz, 1H), 7.65-7.52 (m, 7H), 7.50-7.42 (m, 2H), 7.40-7.31 (m, 3H), 5.94 (s, 1H), 4.20 (d, *J* = 11.3 Hz, 1H), 4.07 (d, *J* = 11.3 Hz, 1H), 3.17 (s, 3H).

^{13}C NMR (90 MHz, $CDCl_3$)δ167.8 (C=O), 141.0 (C), 140.8 (C), 140.7 (C), 136.3 (C), 133.1 (C), 132.2 (CH), 130.2 (CH), 128.9 (2xCH), 128.4 (2xCH), 127.5 (CH), 127.3 (2xCH), 127.2 (2xCH), 123.5 (2xCH), 88.0 (O-CH-N), 64.2 (O-CH_2), 26.8 (N-CH_3).

HRMS (ESI) [M+Na^+] calculated for $C_{22}H_{19}NNaO_2^+$: 352.1308. Found: 352.1300.

2-methyl-3-((4-(trifluoromethyl)benzyl)oxy)isoindolin-1-one (2p)



2p

Following the general procedure, the product was obtained as a colorless oil (268 mg, 87%). (eluent: ethyl acetate/pentane: 1:4);

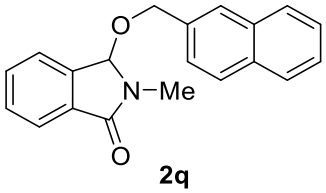
IR (film) ν_{max}: 3053, 2923, 2868, 1709, 1619, 1471, 1427, 1395, 1325, 1263, 1204, 1164, 1123, 1066, 1034, 1018, 824, 747, 698 cm^{-1};

^{1}H NMR (360 MHz, $CDCl_3$)δ7.89-7.84 (m, 1H), 7.62-7.51 (m, 3H), 7.57 (d, *J* = 7.6 Hz, 2H), 7.36 (d, *J* = 7.6 Hz, 2H), 5.93 (s, 1H), 4.19 (d, *J* = 12.1 Hz, 1H), 4.04 (d, *J* = 12.1 Hz, 1H), 3.11 (s, 3H).

^{13}C NMR (90 MHz, $CDCl_3$) δ 167.7 (C=O), 141.4 (C), 140.2(C), 132.9(C), 132.2 (CH), 130.2 (CH), 129.9 (q, *J*= 32.4 Hz) (C), 127.6 (2xCH), 125.3 (q, *J*= 4.5 Hz) (2xCH), 124.1 (q, *J*= 270.0 Hz) (CF_3), 123.4 (d, *J*= 9.0 Hz) (2xCH), 87.8(O-CH-N), 63.2 (O-CH_2), 26.7(N-CH_3).

HRMS (ESI) [M+Na^+] calculated for $C_{17}H_{14}F_3NNaO_2^+$: 344.0869. Found: 344.0868.

2-methyl-3-(naphthalen-2-ylmethoxy)isoindolin-1-one (2q)



2q

Following the general procedure, the product was obtained as a white solid (283 mg, 93%). (eluent: ethyl acetate/pentane: 1:4);

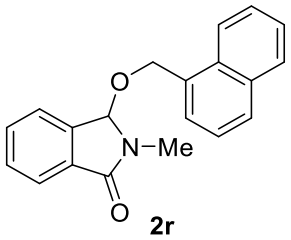
IR (film)ν_{max}: 3053, 2921, 2857, 1706, 1617, 1601, 1508, 1469, 1428, 1393, 1262, 1203, 1179, 1102, 1066, 1033, 894, 854, 819, 746, 694 cm^{-1};

^{1}H NMR (360 MHz, $CDCl_3$)δ7.94-7.88 (m, 1H), 7.88-7.79 (m, 3H), 7.72 (s, 1H), 7.64-7.45 (m, 5H), 7.42-7.35 (m, 1H), 5.96 (s, 1H), 4.32 (d, *J* = 11.3 Hz, 1H), 4.19 (d, *J* = 11.3 Hz, 1H), 3.17 (s, 3H).

^{13}C NMR (90 MHz, $CDCl_3$)δ167.9 (C=O), 140.8 (C), 134.8 (C), 133.4 (C), 133.2 (C), 133.1(C), 132.2 (CH), 130.2 (CH), 128.4 (CH), 128.0 (CH), 127.9 (CH), 126.7 (C), 126.4 (CH), 126.2 (CH), 125.8 (CH), 123.6 (2xCH), 88.1 (O-CH-N), 64.7 (O-CH_2), 26.9 (N-CH_3).

HRMS (ESI) [M+Na^+] calculated for $C_{20}H_{17}NNaO_2^+$: 326.1151. Found: 326.1161.

2-methyl-3-(naphthalen-1-ylmethoxy)isoindolin-1-one (2)



2r

Following the general procedure, the product was obtained as a white solid (278 mg, 92%). (eluent: ethyl acetate/pentane: 1:4);

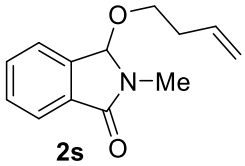
IR (film)ν_{max}: 3051, 2920, 2885, 1946, 1704, 1617, 1598, 1511, 1471, 1428, 1394, 1354, 1263, 1203, 1179, 1102, 1032, 800, 778, 747, 695 cm^{-1};

^{1}H NMR (360 MHz, $CDCl_3$)δ7.98 (d, *J* = 8.3 Hz, 1H), 7.96-7.77 (m, 3H), 7.66-7.47 (m, 5H), 7.46-7.32 (m, 2H), 5.93 (s, 1H), 4.60 (d, *J* = 11.2 Hz, 1H), 4.54 (d, *J* = 11.2 Hz, 1H), 3.10 (s, 3H).

^{13}C NMR (90 MHz, $CDCl_3$)δ167.8 (C=O), 140.8 (C), 133.8 (C), 133.1 (C), 133.0 (C), 132.1 (CH), 131.6 (C), 130.2 (CH), 128.9 (CH), 128.8 (CH), 126.6 (C), 126.5 (CH), 126.0 (CH), 125.4 (CH), 123.7 (CH), 123.6 (CH), 123.5 (CH), 88.0 (O-CH-N), 63.0 (O-CH_2), 26.9 (N-CH_3).

HRMS (ESI) [M+Na^+] calculated for $C_{20}H_{17}NNaO_2^+$: 326.1151. Found: 326.1152.

3-(but-3-en-1-yloxy)-2-methylisoindolin-1-one (2s)



2s

Following the general procedure, the product was obtained as a colorless oil (198 mg, 91%). (eluent: ethyl acetate/pentane: 1:4);

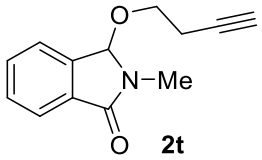
IR (film) ν_{max}: 3077, 2920, 2873, 1708, 1641, 1617, 1469, 1430, 1394, 1261, 1204, 1181, 1103, 1074, 1032, 746 cm^{-1};

^{1}H NMR (360 MHz, $CDCl_3$)δ7.82 (d, *J* = 7.2 Hz, 1H), 7.62-7.48 (m, 3H), 5.79 (s, 1H), 5.75 (ddt, *J* = 17.3, 10.4, 6.8 Hz, 1H), 5.07 (dd, *J* = 17.3, 1.4 Hz, 1H), 5.05 (dd, *J* = 10.4, 1.4 Hz, 1H), 3.15 (dt, *J* = 9.0, 6.7 Hz, 1H), 3.10 (s, 3H), 3.00 (dt, *J* = 9.0, 6.7 Hz, 1H), 2.27 (t, *J* = 6.8 Hz, 2H).

^{13}C NMR (90 MHz, $CDCl_3$)δ167.8 (C=O), 140.9 (C), 135.0 (=CH), 133.1 (C), 132.1 (CH), 130.1 (CH), 123.51 (CH), 123.48 (CH), 117.0 (=CH_2), 87.9 (O-CH-N), 61.4 (O-CH_2), 34.1 (CH_2), 26.7 (N-CH_3).

HRMS (ESI) [M+Na$^+$] calculated for $C_{13}H_{15}NNaO_2^+$: 240.0995. Found: 240.1005.

3-(but-3-yn-1-yloxy)-2-methylisoindolin-1-one (2t)



2t

Following the general procedure, the product was obtained as colorless oil (201 mg, 93%). (eluent: ethyl acetate/pentane: 1:3);

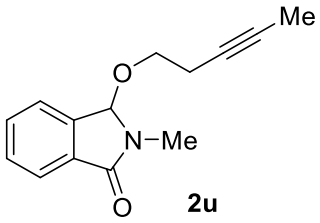
IR (film)ν_{max}: 3054, 2926, 2883, 2119, 1704, 1694, 1682, 1617, 1600, 1471, 1434, 1395, 1261, 1204, 1181, 1107, 1063, 1033, 1015, 746, 696 cm^{-1};

^{1}H NMR (360 MHz, $CDCl_3$)δ7.80 (d, *J* = 7.2 Hz, 1H), 7.60-7.46 (m, 3H), 5.80 (s, 1H), 3.23-3.00 (m, 2H), 3.10 (s, 3H), 2.42-2.34 (m, 2H), 2.01-1.97 (m, 1H).

^{13}C NMR (90 MHz, $CDCl_3$)δ167.6 (C=O), 140.4 (C), 133.0 (C), 132.1 (CH), 130.1 (CH), 123.4 (2xCH), 87.7 (O-CH-N), 81.1 (C≡), 69.7 (HC≡), 60.3 (O-CH_2), 26.7 (N-CH_3), 19.8 (CH_2).

HRMS (ESI) [M+Na$^+$] calculated for $C_{13}H_{13}NNaO_2^+$: 238.0838. Found: 238.0840.

2-methyl-3-(pent-3-yn-1-yloxy)isoindolin-1-one (2u)



2u

Following the general procedure, the product was obtained as a colorless oil (208 mg, 91%). (eluent: ethyl acetate/pentane: 1:5);

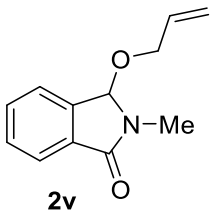
IR (film) ν_{max}: 2920, 2885, 2851, 1706, 1616, 1470, 1432, 1395, 1261, 1204, 1181, 1107, 1076, 1032, 845, 807, 746 cm^{-1};

^{1}H NMR (360 MHz, $CDCl_3$)δ7.80 (d, *J* = 7.2 Hz, 1H), 7.63-7.43 (m, 3H), 5.80 (s, 1H), 3.15 (dt, *J* = 8.6, 6.8 Hz, 1H), 3.10 (s, 3H), 3.01 (dt, *J* = 8.6, 6.8 Hz, 1H), 2.41-2.24 (m, 2H), 1.75 (t, *J* = 2.3 Hz, 1H).

^{13}C NMR (90 MHz, $CDCl_3$)δ167.7 (C=O), 140.7 (C), 133.1 (C), 132.1 (CH), 130.1 (CH), 123.5 (2xCH), 87.8 (O-CH-N), 77.1 (C≡), 75.7 (C≡), 61.0 (O-CH_2), 26.7 (N-CH_3), 20.2 (CH_2), 3.60 (CH_3).

HRMS (ESI) [M+Na$^+$] calculated for $C_{14}H_{15}NNaO_2^+$: 252.0995. Found: 252.1001.

3-(allyloxy)-2-methylisoindolin-1-one (2v)



2v

Following the general procedure, the product was obtained as a colorless oil (188 mg, 93%). (eluent: ethyl acetate/pentane: 1:3);

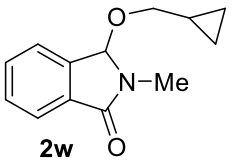
IR (film) ν_{max}: 3081, 3055, 3021, 2918, 2867, 1711, 1647, 1617, 1600, 1470, 1428, 1394, 1262, 1204, 1181, 1104, 1064, 1034, 1014, 747, 698 cm^{-1};

^{1}H NMR (360 MHz, $CDCl_3$)δ7.80 (d, *J* = 7.2 Hz, 1H), 7.60-7.46 (m, 3H), 5.88-5.72 (m, 1H), 5.80 (s, 1H), 5.21 (dd, *J* = 17.3, 1.4 Hz, 1H), 5.12 (dd, *J* = 10.4, 1.4 Hz, 1H), 3.61 (dd, *J* = 12.2, 5.4 Hz, 1H), 3.49 (d, *J* = 12.2, 5.4 Hz, 1H), 3.08 (s, 3H).

^{13}C NMR (90 MHz, $CDCl_3$)δ167.7 (C=O), 140.7 (C), 133.8 (=CH), 133.1 (C), 132.1 (CH), 130.1 (CH), 123.5 (CH), 123.4 (CH), 117.3 (=CH_2), 87.8 (O-CH-N), 63.4 (O-CH_2), 26.7 (N-CH_3).

HRMS (ESI) [M+Na^+] calculated for $C_{12}H_{13}NNaO_2^+$: 226.0838. Found: 226.0839.

3-(cyclopropylmethoxy)-2-methylisoindolin-1-one (2w)



2w

Following the general procedure, the product was obtained as a colorless oil (208 mg, 96%). (eluent: ethyl acetate/pentane: 1:3);

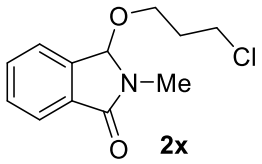
IR (film)ν_{max}: 2923, 1682, 1618, 1600, 1472, 1437, 1397, 1312, 1257, 1205, 1179, 1104, 1062, 1036, 746, 695 cm^{-1};

^{1}H NMR (300 MHz, $CDCl_3$)δ7.77 (d, *J* = 6.9 Hz, 1H), 7.57-7.43 (m, 3H), 5.76 (s, 1H), 3.07 (s, 3H), 2.88 (dd, *J* = 10.2, 7.2 Hz, 1H), 2.78 (dd, *J* = 10.2, 7.2 Hz, 1H), 1.02-0.87 (m, 1H), 0.54-0.41 (m, 1H), 0.11-0.00 (m, 1H).

^{13}C NMR (90 MHz, $CDCl_3$)δ167.6 (C=O), 141.0 (C), 133.0 (C), 132.0 (CH), 129.9 (CH), 123.4 (CH), 123.3 (CH), 87.7 (O-CH-N), 67.2 (O-CH_2), 26.6 (N-CH_3), 10.6 (CH), 3.3 (CH_2), 3.1 (CH_2).

HRMS (ESI) [M+Na^+] calculated for $C_{13}H_{15}NNaO_2^+$: 240.0995. Found: 240.0999.

3-(3-chloropropoxy)-2-methylisoindolin-1-one (2x)



2x

Following the general procedure, the product was obtained as a colorless oil (218 mg, 91%). (eluent: ethyl acetate/pentane: 1:3);

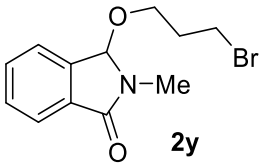
IR (film)ν_{max}: 3054, 2960, 2927, 2882, 1705, 1617, 1470, 1429, 1394, 1299, 1262, 1204, 1181, 1107, 1070, 1032, 745 cm^{-1};

^{1}H NMR (360 MHz, $CDCl_3$)δ7.80 (d, *J* = 7.2 Hz, 1H), 7.60-7.46 (m, 3H), 5.75 (s, 1H), 3.63 (t, *J* = 6.5 Hz, 1H), 3.26 (dt, *J* = 9.4, 5.8 Hz, 1H), 3.08 (s, 3H), 3.06 (dt, *J* = 9.4, 5.8 Hz, 1H), 1.94 (quint, *J* = 6.1 Hz, 2H).

^{13}C NMR (90 MHz, $CDCl_3$)δ167.7 (C=O), 140.6 (C), 133.0 (C), 132.1 (CH), 130.1 (CH), 123.5 (CH), 123.4 (CH), 87.8 (O-CH-N), 58.3 (O-CH_2), 41.6 (CH_2Cl), 32.3(CH_2), 26.7 (N-CH_3).

HRMS (ESI) [M+Na^+] calculated for $C_{12}H_{14}ClNNaO_2^+$: 262.0605. Found: 262.0605.

3-(3-bromopropoxy)-2-methylisoindolin-1-one (2y)



2y

Following the general procedure, the product was obtained as a colorless oil (248 mg, 88%). (eluent: ethyl acetate/pentane: 1:2);

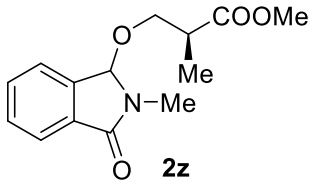
IR (film) ν_{max}: 3081, 3055, 3026, 2926, 2876, 1707, 1616, 1596, 1469, 1429, 1394, 1261, 1204, 1180, 1105, 1069, 1031, 743, 697 cm^{-1};

^{1}H NMR (360 MHz, $CDCl_3$)δ 7.82 (dd, *J* = 7.2, 1.0 Hz, 1H), 7.60 – 7.51 (m, 3H), 5.77 (s, 1H), 3.51 (td, *J* = 6.5, 1.3 Hz, 2H), 3.30 – 3.23 (m, 1H), 3.11 (s, 3H), 3.07 – 3.05 (m, 1H), 2.08 – 1.99 (m, 2H).

^{13}C NMR (90 MHz, $CDCl_3$) δ 167.6(C=O), 140.5 (C), 132.9 (C), 132.0(CH), 130.0 (CH), 123.4(CH), 123.3 (CH), 87.7(O-CH-N), 59.2(O-CH_2), 32.3 (Br-CH_2), 30.1(CH_2), 26.6(N-CH_3).

HRMS (ESI) [M+Na^+] calculated for $C_{12}H_{14}BrNNaO_2^+$: 306.0100 and 308.0081. Found: 306.0098 and 308.0078.

(2S)-methyl 2-methyl-3-((2-methyl-3-oxoisoindolin-1-yl)oxy)propanoate (2z)



2z

Following the general procedure, the product was obtained as a colorless oil (251 mg, 95%). (eluent: ethyl acetate/pentane: 1:2);

IR (film)ν_{max}: 2976, 2950, 2881, 1701, 1617, 1601, 1471, 1436, 1394, 1261, 1207, 1181, 1155, 1103, 1067, 1032, 749, 697 cm^{-1};

^{1}H NMR (360 MHz, $CDCl_3$) δ 7.80 – 7.73 (m, 1H), 7.58 – 7.44 (m, 3H), 5.73 (s, 1H), 3.66 (s, 3H), 3.29 – 2.88 (m, 2H), 3.04 (s, 3H). 2.72 – 2.57 (m, 1H), 1.07 (m, 3H).

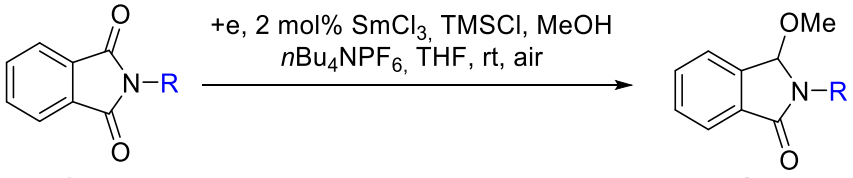
^{13}C NMR (90 MHz, $CDCl_3$)δ 174.8(C=O), 174.7(C=O), 167.6(C=O), 167.5(C=O), 140.3 (C), 132.9 (C), 132.8 (C), 132.0 (CH), 131.9 (CH), 123.0 (CH), 123.4 (CH), 123.3 (2xCH), 87.6 (O-CH-N), 87.5 (O-CH-N), 63.7 (O-CH_2), 63.6 (O-CH_2), 51.8 (O-CH_3), 39.7 (CH), 39.7 (CH), 26.5 (N-CH_3), 13.9 (CH_3).

HRMS (ESI) [M+Na^+] calculated for $C_{14}H_{17}NNaO_4^+$: 286.1050. Found:286.1051.

d. r. = 1:1

5. Characterization data of reductive methoxylation of *N*-substituted phthalimides

5.1. General procedure for the reductive methoxylation of *N*-substituted phthalimides:

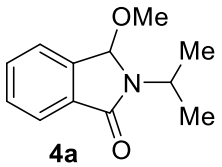


3a-p **4a-p**

Reactions were carried out in a three necked cell containing a magnetic stirring bar, samarium cathode (20 cm^2 area), glassy carbon anode (20 cm^2 area) and SCE as reference electrode. nBu_4NPF_6 (156 mg, 0.4 mmol, 0.04 mol/L in THF) as the electrolyte, N-substituted phthalimide (1.0 mmol), methanol (320 mg, 10 mmol, 0.4 ml) and $SmCl_3$ (5.0 mg, 0.02 mmol)were added in anhydrous THF (10 ml), TMSCl (272 mg, 2.5 mmol, 0.32 ml) was added dropwise with

stirring, then electrolysis was performed at i = 50 mA during 2 hours. After the reaction was complete, the mixture was quenched by 0.1 mol/L HCl (10 ml), extracted with ethyl acetate (2×20 mL). The combined organic layer was washed by 10% $Na_2S_2O_3$ (10 ml) and brine, dried over $MgSO_4$ and the solvent was removed under reduced pressure. The crude product was purified by flash chromatography on silica gel.

2-isopropyl-3-methoxyisoindolin-1-one (4a)



4a

Following the general procedure, the product was obtained as a colorless oil (199 mg, 97%). (eluent: ethyl acetate/pentane: 1:3);

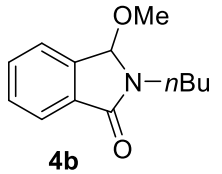
IR (film)ν_{max}: 2977, 2934, 2829, 1692, 1615, 1467, 1403, 1365, 1225, 1191, 1076, 750, 697 cm^{-1};

^{1}H NMR (360 MHz, $CDCl_3$)δ 7.79 (dd, *J* = 4.5, 3.7 Hz, 1H), 7.59 – 7.44 (m, 3H), 5.99 (s, 1H), 4.40 (hept, *J* = 6.9 Hz, 1H), 2.89 (s, 3H), 1.39 (dd, *J* = 9.0, 6.9 Hz, 6H).

^{13}C NMR (90 MHz, $CDCl_3$) δ 167.6(C=O), 140.4(C), 133.3 (C), 131.9 (CH), 129.8 (CH), 123.3(CH), 123.2 (CH), 85.6 (O-CH-N), 48.8 (O-CH_3), 43.8 (N-CH), 21.0 (CH_3), 20.1 (CH_3).

HRMS (ESI) [M+Na^+] calculated for $C_{12}H_{15}NNaO_2^+$: 228.0995. Found: 228.1002.

2-butyl-3-methoxyisoindolin-1-one (4b)



4b

Following the general procedure, the product was obtained as a colorless oil (198 mg, 90%). (eluent: ethyl acetate/pentane: 1:5);

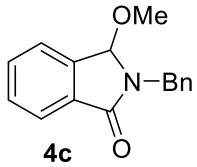
IR (film)ν_{max}: 3056, 2959, 2933, 2872, 2828, 1709, 1616, 1602, 1467, 1412, 1375, 1323, 1206, 1190, 1103, 1067, 937, 749, 696 cm^{-1};

^{1}H NMR (360 MHz, $CDCl_3$)δ 7.82 – 7.68 (m, 1H), 7.58 – 7.38 (m, 3H), 5.81 (s, 1H), 3.73 (dt, *J* = 13.8, 7.8 Hz, 1H), 3.15 (ddd, *J* = 13.9, 7.9, 6.3 Hz, 1H), 2.80 (s, 3H), 1.68 – 1.49 (m, 2H), 1.31 (dd, *J* = 14.9, 7.5 Hz, 2H), 0.88 (t, *J* = 7.3 Hz, 3H).

^{13}C NMR (90 MHz, $CDCl_3$)δ 167.5(C=O), 140.3 (C), 133.2 (C), 131.8(CH), 129.8(CH), 123.3 (2xCH), 86.1 (O-CH-N), 49.0 (O-CH_3), 39.1 (CH_2), 30.1 (CH_2), 20.2 (CH_2), 13.7 (CH_3).

HRMS (ESI) [M+Na^+] calculated for $C_{13}H_{17}NNaO_2^+$: 242.1151. Found: 242.1147.

2-benzyl-3-methoxyisoindolin-1-one (4c)



4c

Following the general procedure, the product was obtained as a colorless oil (245 mg, 97%). (eluent: ethyl acetate/pentane: 1:5);

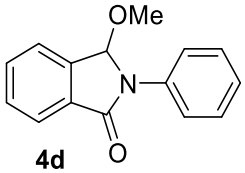
IR (KBr)ν_{max}: 2924, 2829, 1703, 1647, 1616, 1493, 1465, 1406, 1356, 1200, 1102, 1068 cm^{-1};

^{1}H NMR (360 MHz, $CDCl_3$)δ 7.89 (dd, *J* = 6.5, 1.1 Hz, 1H), 7.64 – 7.46 (m, 3H), 7.41 – 7.25 (m, 5H), 5.73 (s, 1H), 5.21 (d, *J* = 14.7 Hz, 1H), 4.22 (d, *J* = 14.7 Hz, 1H), 2.90 (s, 3H).

^{13}C NMR (90 MHz, $CDCl_3$) δ 167.5(C=O), 140.4 (C), 136.8 (C), 133.0 (C), 132.1(CH), 130.0 (CH), 128.7(2xCH), 128.6 (2xCH), 127.7 (CH), 123.7 (CH), 123.5(CH), 85.6 (O-CH-N), 49.3(O-CH_3), 43.1 (CH_2).

HRMS (ESI) [M+Na^+] calculated for $C_{16}H_{15}NNaO_2^+$: 276.0995. Found: 276.0996.

3-methoxy-2-phenylisoindolin-1-one (4d)



4d

Following the general procedure, the product was obtained as a colorless oil (203 mg, 85%). (eluent: ethyl acetate/pentane: 1:5);

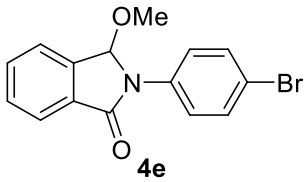
IR (KBr)ν_{max}: 3079, 3059, 3043, 3024, 2950, 2929, 1686, 1596, 1500, 1390, 1306, 1152, 751, 732 cm^{-1};

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.95 (d, *J* = 7.4 Hz, 1H), 7.88 – 7.76 (m, 2H), 7.72 – 7.57 (m, 3H), 7.47 (ddd, *J* = 10.1, 4.5, 2.0 Hz, 2H), 7.29 – 7.22 (m, 1H), 6.50 (s, 1H), 2.94 (s, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 166.7(C=O), 139.7 (C), 137.3(C), 132.9 (C), 132.8 (CH), 130.3 (CH), 129.1 (2xCH), 125.3 (CH), 123.9 (CH), 123.4 (CH), 121.7 (2xCH), 87.3(O-CH-N), 49.1(O-CH_3).

HRMS (ESI) [M+Na^+] calculated for $C_{15}H_{13}NNaO_2^+$: 262.0838. Found: 262.0847.

2-(4-bromophenyl)-3-methoxyisoindolin-1-one (4e)



4e

Following the general procedure, the product was obtained as a colorless oil (298 mg, 94%). (eluent: ethyl acetate/pentane: 1:5);

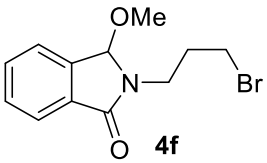
IR (KBr)ν_{max}: 3061, 2997, 2954, 2931, 2854, 2829, 1783, 1714, 1589, 1493, 1467, 1413, 1376, 1296, 1218, 1129, 1109, 1075, 1058 cm^{-1};

^{1}H NMR (360 MHz, $CDCl_3$) δ 7.94 (dd, *J* = 7.3, 1.2 Hz, 1H), 7.83 – 7.78 (m, 2H), 7.73 – 7.68 (m, 1H), 7.65 – 7.60 (m, 2H), 7.59 – 7.54 (m, 2H), 6.46 (s, 1H), 2.91 (s, 3H).

^{13}C NMR (63 MHz, $CDCl_3$) δ 166.6(C=O), 139.5(C), 136.6(C), 133.0(CH), 132.7 (C), 132.1(2xCH), 130.5 (CH), 124.0(CH), 123.5 (CH), 122.7(2xCH), 118.2 (C), 87.2 (O-CH-N), 49.0(O-CH_3).

HRMS (ESI) [M+Na^+] calculated for $C_{15}H_{12}BrNNaO_2^+$: 339.9944 and 341.9924. Found: 339.9941 and 341.9920.

2-(3-bromopropyl)-3-methoxyisoindolin-1-one (4f)



4f

Following the general procedure, the product was obtained as a colorless oil (249 mg, 88%). (eluent: ethyl acetate/pentane: 1:4);

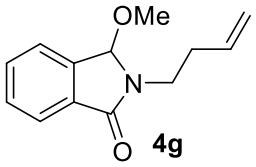
IR (film)ν_{max}: 2929, 2857, 2313, 1672, 1641, 1468, 1418, 1107, 1051 cm^{-1};

^{1}H NMR (360 MHz, $CDCl_3$) δ 7.89 – 7.77 (m, 1H), 7.65 – 7.47 (m, 3H), 5.90 (s, 1H), 3.94 – 3.73 (m, 1H), 3.58 – 3.37 (m, 3H), 2.92 (s, 3H), 2.43 – 2.13 (m, 2H).

^{13}C NMR (63 MHz, $CDCl_3$) δ 167.9(C=O), 140.4 (C), 132.8(C), 132.2 (CH), 130.0(CH), 123.5 (2xCH), 86.8 (O-CH-N), 49.5 (O-CH_3), 38.5 (N-CH_2), 31.3 (Br-CH_2), 30.4 (CH_2).

HRMS (ESI) [M+Na^+] calculated for $C_{12}H_{14}BrNNaO_2^+$: 306.0100 and 308.0081. Found: 306.0091 and 308.0074.

2-(but-3-en-1-yl)-3-methoxyisoindolin-1-one (4g)



4g

Following the general procedure, the product was obtained as a colorless oil (191 mg, 88%). (eluent: ethyl acetate/pentane: 1:2);

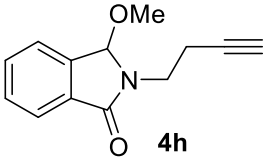
IR (film)ν_{max}: 3078, 2933, 2829, 1698, 1642, 1617, 1601, 1469, 1439, 1412, 1366, 1331, 1296, 1206, 1103, 1070, 918, 747, 697 cm^{-1};

^{1}H NMR (360 MHz, $CDCl_3$)δ 7.87 (dd, *J* = 8.1, 1.2 Hz, 1H), 7.59 (qdd, *J* = 8.4, 6.9, 1.2 Hz, 3H), 5.95 (s, 1H), 5.94 – 5.81 (m, 1H), 5.27 – 4.95 (m, 2H), 3.94 (dt, *J* = 14.1, 7.5 Hz, 1H), 3.34 (ddd, *J* = 13.8, 7.7, 6.3 Hz, 1H), 2.91 (s, 3H), 2.58 – 2.40 (m, 2H).

^{13}C NMR (90 MHz, $CDCl_3$) δ 167.7(C=O), 140.3 (C), 135.2 (=CH), 133.1 (C), 132.0 (CH), 129.9(CH), 123.4(CH), 123.4(CH), 117.1(=CH_2), 86.3 (O-CH-N), 49.1(O-CH_3), 38.7 (N-CH_2), 32.6 (CH_2).

HRMS (ESI) [M+Na^+] calculated for $C_{13}H_{15}NNaO_2^+$: 240.0995. Found: 240.0997.

2-(but-3-yn-1-yl)-3-methoxyisoindolin-1-one (4h)



4h

Following the general procedure, the product was obtained as a colorless oil (193 mg, 90%). (eluent: ethyl acetate/pentane: 1:3);

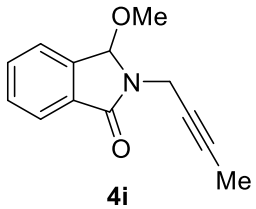
IR (film) ν_{max}: 3083, 3056, 2934, 2829, 2118, 1701, 1616, 1601, 1470, 1412, 1367, 1333, 1297, 1207, 1130, 1104, 1076, 1025, 962, 802, 745, 697 cm^{-1};

^{1}H NMR (360 MHz, $CDCl_3$) δ 7.88 – 7.74 (m, 1H), 7.64 – 7.42 (m, 3H), 6.06 (s, 1H), 3.92 (ddd, *J* = 13.4, 7.2, 5.9 Hz, 1H), 3.56 – 3.38 (m, 1H), 2.87 (s, 3H), 2.71 – 2.48 (m, 2H), 1.98 (s, 1H).

^{13}C NMR (90 MHz, $CDCl_3$) δ 167.7 (C=O), 140.4 (C), 132.8 (C), 132.1 (CH), 123.0 (CH), 123.5 (CH), 123.5 (CH), 86.8 (O-CH-N), 81.4 (C≡), 70.1 (HC≡), 49.3(O-CH_3), 38.2 (N-CH_2), 18.2 (CH_2).

HRMS (ESI) [M+Na^+] calculated for $C_{13}H_{13}NNaO_2^+$: 238.0838. Found: 238.0840.

2-(but-2-yn-1-yl)-3-methoxyisoindolin-1-one (4i)



4i

Following the general procedure, the product was obtained as a colorless oil (200 mg, 93%). (eluent: ethyl acetate/pentane: 1:5);

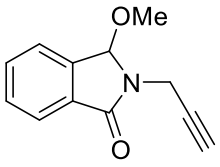
IR (film) ν_{max}: 3024, 2956, 2926, 2858, 2829, 1771, 1708, 1616, 1468, 1407, 1353, 1293, 1204, 1190, 1152, 1103, 1072, 945, 747 cm^{-1};

^{1}H NMR (400 MHz, $CDCl_3$)δ 7.88 – 7.82 (m, 1H), 7.65 – 7.51 (m, 3H), 6.06 (s, 1H), 4.69 (dd, *J* = 17.2, 2.5 Hz, 1H), 3.96 – 3.84 (m, 1H), 2.98 (s, 3H), 1.82 (dd, *J* = 3.1, 1.8 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 166.9(C=O), 140.5 (C), 132.8 (C), 132.2 (CH), 129.9 (CH), 123.7 (CH), 123.5 (CH), 85.9 (O-CH-N), 79.7(C≡), 73.2(C≡), 49.8 (O-CH_3), 29.4 (CH_2), 3.5 (CH_3).

HRMS (ESI) [M+Na^+] calculated for $C_{13}H_{15}NNaO_2^+$: 240.0995. Found: 240.0998.

3-methoxy-2-(prop-2-yn-1-yl)isoindolin-1-one (4j)



4j

Following the general procedure, the product was obtained as a colorless oil (177 mg, 88%). (eluent: ethyl acetate/pentane: 1:4);

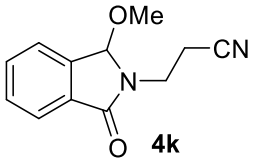
IR (film)ν_{max}: 2929, 2829, 2117, 1697, 1683, 1613, 1465, 1420, 1401, 1351, 1289, 1202, 1135, 1104, 1076, 1065, 934, 797, 752 cm^{-1};

^{1}H NMR (360 MHz, $CDCl_3$)δ 7.84 (d, *J* = 7.2 Hz, 1H), 7.57 (ddd, *J* = 10.5, 7.2, 5.3 Hz, 3H), 6.04 (s, 1H), 4.70 (dd, *J* = 17.5, 2.5 Hz, 1H), 3.97 (dd, *J* = 17.5, 2.4 Hz, 1H), 2.97 (s, 3H), 2.26 (s, 1H).

^{13}C NMR (90 MHz, $CDCl_3$)δ 166. 9(C=O), 140.5 (C), 132.4 (CH), 130.1 (CH), 123.8 (CH), 123.6 (CH), 86.0 (O-CH-N), 77.9(C≡), 72.0 (C≡), 49.9 (O-CH_3), 29.0 (CH_2).

HRMS (ESI) [M+Na^+] calculated for $C_{12}H_{11}NNaO_2^+$: 224.0682. Found: 224.0681.

3-(1-methoxy-3-oxoisoindolin-2-yl)propanenitrile (4k)



4k

Following the general procedure, the product was obtained as a colorless oil (197 mg, 91%). (eluent: ethyl acetate/pentane: 1:2);

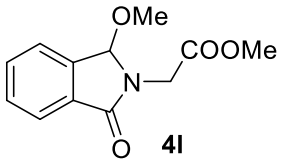
IR (film)ν_{max}: 2934, 2835, 2248, 1702, 1616, 1470, 1412, 1368, 1333, 1207, 1133, 1104, 1076, 749, 697 cm^{-1};

^{1}H NMR (360 MHz, $CDCl_3$)δ 7.92 – 7.82 (m, 1H), 7.71 – 7.52 (m, 3H), 6.07 (s, 1H), 3.98 (ddd, *J* = 13.9, 6.9, 5.5 Hz, 1H), 3.72 (ddd, *J* = 14.2, 8.0, 6.6 Hz, 1H), 2.96 (s, 3H), 2.94 – 2.84 (m, 1H), 2.76 (ddd, *J* = 16.8, 6.4, 5.5 Hz, 1H).

^{13}C NMR (90 MHz, $CDCl_3$) δ 168.0 (C=O), 140.3(C), 132.7 (CH), 132.2 (C), 130.3 (CH), 123.8 (CH), 123.7(CH), 117.9(CN), 87.0 (O-CH-N), 49.8(O-CH_3), 35.9 (N-CH_2), 17.1 (CH_2).

HRMS (ESI) [M+Na^+] calculated for $C_{12}H_{12}N_2NaO_2^+$: 239.0791. Found: 239.0790.

methyl 2-(1-methoxy-3-oxoisoindolin-2-yl)acetate (4l)



4l

Following the general procedure, the product was obtained as a yellow oil (202 mg, 86%). (eluent: ethyl acetate/pentane: 1:2);

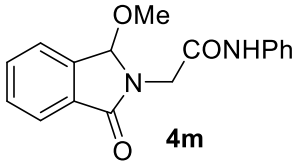
IR (film)ν_{max}: 2954, 2849, 1749, 1713, 1617, 1469, 1422, 1210, 1078, 946, 750 cm^{-1};

^{1}H NMR (360 MHz, $CDCl_3$)δ 7.85 (dd, *J* = 7.3, 1.0 Hz, 1H), 7.64 – 7.49 (m, 3H), 6.02 (s, 1H), 4.62 (d, *J* = 17.7 Hz, 1H), 3.95 (d, *J* = 17.7 Hz, 1H), 3.74 (s, 3H), 2.91 (s, 3H).

^{13}C NMR (90 MHz, $CDCl_3$)δ 169.3(C=O), 167.8 (C=O), 140.6(C), 132.4(CH), 132.3 (C), 130.0(CH), 123.8(CH), 123.6(CH), 86.7 (O-CH-N), 52.3 (O-CH_3), 49.6 (O-CH_3), 40.5 (CH_2).

HRMS (ESI) [M+Na^+] calculated for $C_{12}H_{13}NNaO_4^+$: 258.0737. Found: 258.0746.

2-(1-methoxy-3-oxoisoindolin-2-yl)-*N*-phenylacetamide (4m)



4m

Following the general procedure, the product was obtained as a colorless oil (246 mg, 83%). (eluent: ethyl acetate/pentane: 1:1);

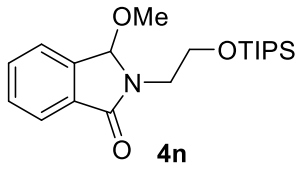
IR (film)ν_{max}: 2920, 2885, 2851, 2341, 1706, 1616, 1552, 1469, 1300, 1253, 1194, 946, 750 cm^{-1};

^{1}H NMR (250 MHz, $CDCl_3$)δ 8.89 (s, 1H), 7.94 – 7.76 (m, 1H), 7.58 (ddd, *J* = 17.3, 11.4, 6.7 Hz, 5H), 7.24 (t, *J* = 7.8 Hz, 2H), 7.05 (t, *J* = 7.4 Hz, 1H), 6.10 (s, 1H), 4.60 (d, *J* = 16.1 Hz, 1H), 4.20 (d, *J* = 16.1 Hz, 1H), 3.01 (s, 3H);

^{13}C NMR (90 MHz, $CDCl_3$)δ 168.7(C=O), 166.5(C), 141.0(C), 137.7 (C), 132.7 (CH), 131.9(C), 130.2 (CH), 128.9 (2xCH), 124.3(CH), 123.7(2xCH), 119.9(2xCH), 87.9(O-CH-N), 50.6 (O-CH_3), 44.5 (N-CH_2).

HRMS (ESI) [M+Na^+] calculated for $C_{17}H_{16}N_2NaO_3^+$: 319.1053. Found: 319.1053.

3-methoxy-2-(2-((triisopropylsilyl)oxy)ethyl)isoindolin-1-one (4n)



4n

Following the general procedure, the product was obtained as a colorless oil (338 mg, 93%). (eluent: ethyl acetate/pentane: 1:5);

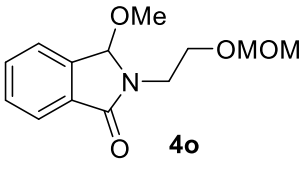
IR (film)ν_{max}: 2935, 2862, 2722, 1706, 1616, 1465, 1404, 1311, 1202, 1104, 1071, 995, 923, 881, 744 cm^{-1};

^{1}H NMR (360 MHz, $CDCl_3$) δ 7.87 – 7.76 (m, 1H), 7.61 – 7.43 (m, 3H), 6.08 (s, 1H), 3.99 (td, *J* = 8.6, 3.8 Hz, 1H), 3.95 – 3.85 (m, 2H), 3.46 – 3.29 (m, 1H), 2.87 (s, 3H), 1.15 – 0.92 (m, 21H).

^{13}C NMR (90 MHz, $CDCl_3$) δ 167.7(C=O), 140.8(C), 133.1 (C), 131.9(CH), 129.8 (CH), 123.3(2xCH), 87.6(O-CH-N), 61.7(SiO-CH_2), 49.2 (O-CH_3), 41.7 (N-CH_2), 17.9 (6xCH_3), 11.8 (3xCH).

HRMS (ESI) [M+Na^+] calculated for $C_{20}H_{33}NNaO_3Si^+$: 386.2122. Found: 386.2124.

3-methoxy-2-(2-(methoxymethoxy)ethyl)isoindolin-1-one (4o)



4o

Following the general procedure, the product was obtained as a colorless oil (228 mg, 91%). (eluent: ethyl acetate/pentane: 1:1);

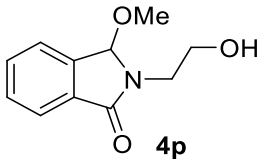
IR (film)ν_{max}: 2935, 2885, 2823, 2773, 1703, 1613, 1465, 1409, 1144, 1110, 1071, 917, 749 cm^{-1};

^{1}H NMR (360 MHz, $CDCl_3$)δ 7.80 (dd, *J* = 7.3, 0.9 Hz, 1H), 7.60 – 7.44 (m, 3H), 6.02 (s, 1H), 4.58 (s, 2H), 3.99 (dt, *J* = 14.3, 4.8 Hz, 1H), 3.76 (dd, *J* = 6.0, 5.0 Hz, 2H), 3.42 (dt, *J* = 14.3, 6.1 Hz, 1H), 3.27 (s, 3H), 2.86 (s, 3H).

^{13}C NMR (90 MHz, $CDCl_3$)δ 167.7(C=O), 140.6(C), 132.9(C), 132.0(CH), 129.9 (CH), 123.4(2xCH), 96.3 (OCH_2O), 87.1 (O-CH-N), 65.6 (O-CH_2), 55.3 (OMO-CH_3), 49.2 (O-CH_3), 39.1 (N-CH_2).

HRMS (ESI) [M+Na^+] calculated for $C_{13}H_{17}NNaO_4^+$: 274.1050. Found: 274.1051.

2-(2-hydroxyethyl)-3-methoxyisoindolin-1-one (4p)



4p

Following the general procedure, the product was obtained as a colorless oil (190 mg, 92%). (eluent: ethyl acetate/methanol: 5:1);

IR (film) ν_{max}: 3419, 2936, 2832, 2120, 1682, 1617, 1601, 1470, 1416, 1365, 1206, 1105, 1076, 748, 697 cm^{-1};

^{1}H NMR (360 MHz, $CDCl_3$)δ 7.88 – 7.78 (m, 1H), 7.65 – 7.48 (m, 3H), 5.95 (s, 1H), 3.90 (dd, *J* = 5.4, 4.0 Hz, 2H), 3.84 – 3.72 (m, 1H), 3.65 (ddd, *J* = 14.5, 5.8, 4.1 Hz, 1H), 3.41 (brs, 1H), 2.95 (s, 3H).

^{13}C NMR (90 MHz, $CDCl_3$) δ 169.0 (C=O), 140.4(C), 132.6(C), 132.3(CH), 130.1 (CH), 123.6(CH), 123.5 (CH), 87.7 (O-CH-N), 61.7(HO-CH_2), 49.6 (O-CH_3), 43.6 (N-CH_2).

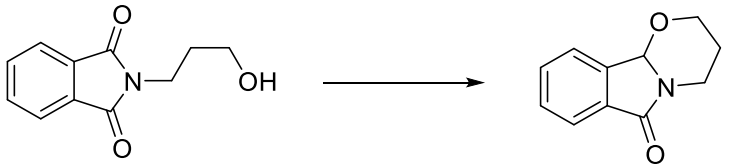
HRMS (ESI) [M+Na^+] calculated for $C_{11}H_{13}NNaO_3^+$: 230.0788. Found: 230.0790.

3,4-dihydro-2*H*-[1,3]oxazino[2,3-*a*]isoindol-6(10b*H*)-one (4q)

Reaction was carried out in a three necked cell containing a magnetic stirring bar, samarium cathode (20 cm^2 area), glassy carbon anode (20 cm^2 area) and SCE as reference electrode; *n*Bu_4NPF_6 (156 mg, 0.4 mmol, 0.04 mol/L in THF) as the electrolyte, 2-(3-hydroxypropyl)isoindoline-1,3-dione (205 mg, 1.0 mmol) and $SmCl_3$ (5.0 mg, 0.02 mmol)were

added in anhydrous THF (10 ml), TMSCl (272 mg, 2.5 mmol, 0.32 ml) was added dropwise with stirring, then electrolysis was performed at i = 50 mA during 3 hours.

After the reaction was complete, the mixture was quenched by 0.1 mol/L HCl (10 ml), extracted with ethyl acetate (2×20 mL). The combined organic layer was washed by 10% $Na_2S_2O_3$ (10 ml) and brine, dried over $MgSO_4$ and the solvent was removed under reduced pressure. The crude product was purified by flash chromatography on silica gel.



3q **4q** 97%

The product was obtained as a colorless oil (183 mg, 97%) Using eluent: ethyl acetate/pentane: 1:1.

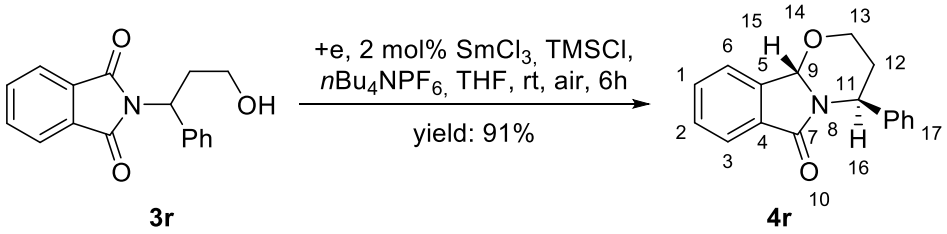
IR (film)ν_{max}: 2957, 2862, 1703, 1426, 1281, 1057 cm^{-1};

^{1}H NMR (360 MHz, $CDCl_3$)δ 7.84 (dd, *J* = 7.0, 1.1 Hz, 1H), 7.60 – 7.50 (m, 3H), 5.57 (s, 1H), 4.59 – 4.35 (m, 1H), 4.31 – 4.09 (m, 1H), 3.96 (td, *J* = 12.2, 2.1 Hz, 1H), 3.37 – 3.17 (m, 1H), 1.94 – 1.81 (m, 1H), 1.66 (dtd, *J* = 13.6, 3.6, 1.7 Hz, 1H).

^{13}C NMR (90 MHz, $CDCl_3$) δ 165.9(C=O), 141.2 (C), 132.8 (C), 131.9 (CH), 123.0 (CH), 123.6 (CH), 123.1 (CH), 85.1(O-CH-N), 67.3(O-CH_3), 37.8 (N-CH_2), 24.6 (CH_2).

HRMS (ESI) [M+Na^+] calculated for $C_{11}H_{11}NNaO_2^+$: 212.0682. Found: 212.0683.

4-phenyl-3,4-dihydro-2H-[1,3]oxazino[2,3-a]isoindol-6(10bH)-one (4r)



3r **4r**

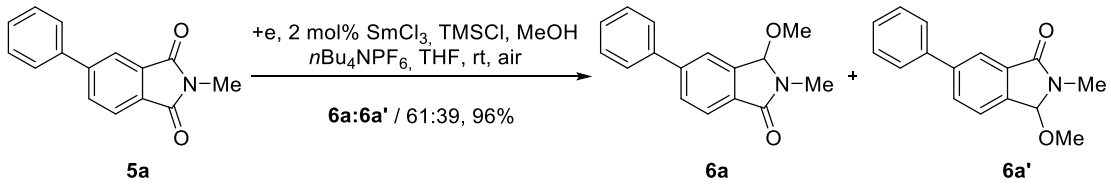
nBu_4NPF_6 (156 mg, 0.4 mmol, 0.04 mol/L in THF) as the electrolyte, 2-(3-hydroxy-1-phenylpropyl)isoindoline-1,3-dione (281 mg, 1.0 mmol) and $SmCl_3$ (5.0 mg, 0.02 mmol)were added in anhydrous THF (10 ml), TMSCl (272 mg, 2.5 mmol, 0.32 ml) was added dropwise with stirring, then electrolysis was performed at i = 50 mA during 3 hours. After the reaction was complete, the mixture was quenched by 0.1 mol/L HCl (10 ml), extracted with ethyl acetate (2×20 mL). The combined organic layer was washed by 10% $Na_2S_2O_3$ (10 ml) and brine, dried over $MgSO_4$ and the solvent was removed under reduced pressure. The crude product was

purified by flash chromatography on silica gel (eluent: ethyl acetate/pentane: 1:3) to afford the product as a colorless oil (241 mg, 91%). The relative stereochemistry of H15 and H16 was determined by NOESY experiment.

^{1}H NMR (360 MHz, $CDCl_3$) δ 7.93 (dd, *J* = 7.9, 1.6 Hz, 1H), 7.62 – 7.54 (m, 3H), 7.41 – 7.35 (m, 2H), 7.34 – 7.29 (m, 3H), 5.81 (d, *J* = 4.4 Hz, 1H), 5.72 (s, 1H), 4.13 (ddd, *J* = 6.6, 3.7, 2.3 Hz, 1H), 4.00 (ddd, *J* = 11.3, 7.0, 3.8 Hz, 1H), 2.38 – 2.26 (m, 2H).

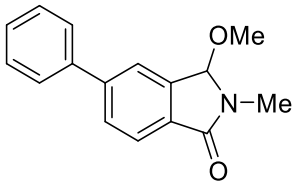
^{13}C NMR (90 MHz, $CDCl_3$) δ 166.9 (C=O), 141.5 (C), 138.9 (C), 132.3 (C), 132.2 (CH), 130.1 (CH), 128.9 (2xCH), 127.3 (CH), 126.7 (2xCH), 123.9 (CH), 123.4 (CH), 82.7(O-CH-N), 64.0 (O-CH_2), 48.6 (N-CH), 28.1 (CH_2).

6. Regioselectivity of reductive methoxylation of aryl-ring-substituted *N*-methylphtalimides



Following the general procedure, the mixture of 5.6a and 5.6a' was obtained in 96% yield (243 mg). (eluent: ethyl acetate/petane: 1:4).

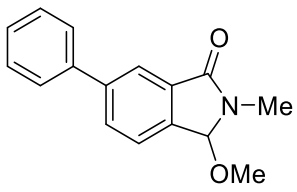
3-methoxy-2-methyl-5-phenylisoindolin-1-one (6a)



6a

^{1}H NMR (360 MHz, $CDCl_3$)δ7.87 (d, *J* = 8.2 Hz, 1H), 7.75-7.71 (m,1H), 7.65-7.55 (m, 3H), 7.51-7.35 (m, 3H), 5.80 (s, 1H),3.10 (s, 3H), 2.94 (s, 3H).

3-methoxy-2-methyl-6-phenylisoindolin-1-one (6a')

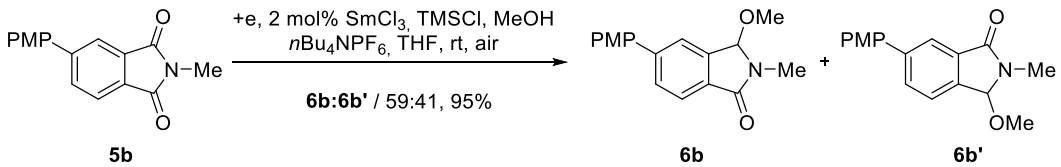


6a'

^{1}H NMR (360 MHz, $CDCl_3$)δ8.04 (d, *J* = 1.5 Hz, 1H), 7.80 (dd, *J* = 7.8, 1.6 Hz 1H), 7.65-7.55 (m,3H), 7.51-7.35 (m, 3H), 5.80 (s, 1H), 3.10 (s, 3H), 2.94 (s, 3H).

21a and 21a':**^{13}C NMR (90 MHz, $CDCl_3$)**δ167.8, 167.7, 145.4,143.4, 141.1, 140.1, 140.0, 139.1, 134.0, 132.1, 130.9, 129.1, 128.3, 128.1, 127.5, 127.3, 123.8, 122.0,121.9, 88.1, 88.0, 49.4, 26.6

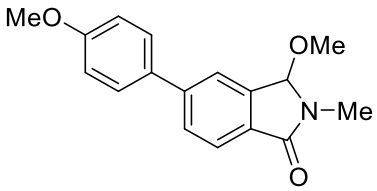
HRMS (ESI) [M+Na^+] calculated for $C_{16}H_{15}NNaO_2^+$: 276.0995. Found: 276.0997.



5b **6b** **6b'**

Following the general procedure, the mixture of 5.6b and 5.6b' was obtained in 95% yield (269 mg). (eluent: ethyl acetate/petane: 1:4).

3-methoxy-5-(4-methoxyphenyl)-2-methylisoindolin-1-one (6b)



6b

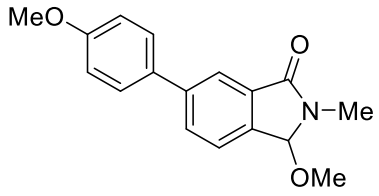
White solid.**IR (film)** ν_{max}: 3039, 2996, 2934, 2835, 2532, 2043, 1703, 1607, 1520, 1448, 1361, 1254, 1185, 1120, 1080, 1037, 960, 825, 779, cm^{-1};

^{1}H NMR (250 MHz, $CDCl_3$) δ 7.85 (d, *J* = 8.3 Hz, 1H), 7.77 – 7.64 (m, 2H), 7.57 (d, *J* = 8.7 Hz, 2H), 7.01 (d, *J* = 8.8 Hz, 2H), 5.80 (s, 1H), 3.87 (s, 3H), 3.10 (s, 3H), 2.94 (s, 3H).

^{13}C NMR (90 MHz, $CDCl_3$) δ 167.8(C=O), 159.9 (C), 144.9 (C), 141.1 (C), 132.4 (C), 131.4 (CH), 128.5 (2xCH), 123.7 (CH), 121.4(CH), 114.5 (2xCH), 88.0 (O-CH-N), 55.4 (ArO-CH_3), 49.2 (O-CH_3), 26.5 (N-CH_3).

HRMS (ESI) [M+Na^+] calculated for $C_{17}H_{17}NNaO_3^+$: 306.1101. Found: 306.1111.

3-methoxy-6-(4-methoxyphenyl)-2-methylisoindolin-1-one (6b')



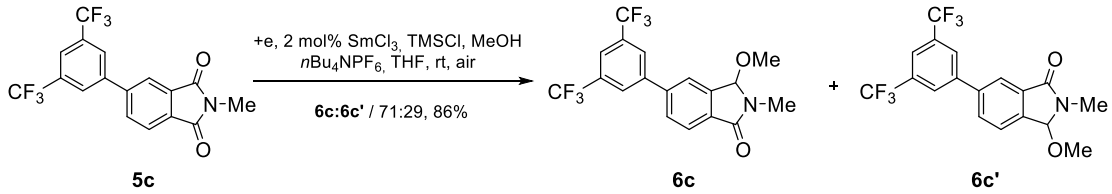
6b'

White solid. **IR (film)**ν_{max}: 3039, 2996, 2934, 2835, 2532, 2043, 1703, 1607, 1520, 1448, 1359, 1255, 1183, 1122, 1079, 1035, 959, 825, 779, 700 cm^{-1};

^{1}H NMR (360 MHz, $CDCl_3$) δ 8.02 (d, *J* = 1.5 Hz, 1H), 7.78 (dd, *J* = 7.8, 1.7 Hz, 1H), 7.63 – 7.54 (m, 3H), 7.07 – 6.98 (m, 2H), 5.82 (s, 1H), 3.88 (s, 3H), 3.12 (s, 3H), 2.96 (s, 3H).

^{13}C NMR (90 MHz, $CDCl_3$) δ 167.8 (C=O), 159.7 (C), 142.9(C), 138.3 (C), 133.9 (C), 132.3(C), 130.4 (CH), 128.3 (2xCH), 123.7 (CH), 121.3(CH), 114.4 (2xCH), 87.9(O-CH-N), 55.4 (ArO-CH_3), 49.2 (O-CH_3), 26.5(N-CH_3).

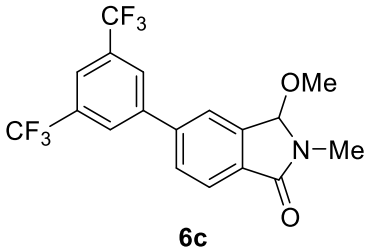
HRMS (ESI) [M+Na^+] calculated for $C_{17}H_{17}NNaO_3^+$: 306.1101. Found: 306.1111.



5c **6c** **6c'**

Following the general procedure, the mixture of 5.6c and 5.6c' was obtained in 86% yield (335 mg). (eluent: ethyl acetate/petane: 1:4).

5-(3,5-bis(trifluoromethyl)phenyl)-3-methoxy-2-methylisoindolin-1-one (6c)



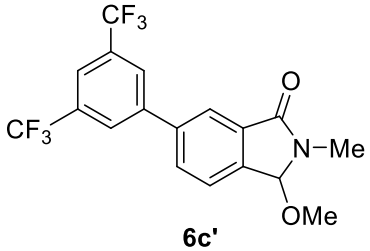
6c

^{1}H NMR (360 MHz, $CDCl_3$) δ 8.09 (s, 2H), 8.03 – 7.93 (m, 2H), 7.85 – 7.77 (m, 2H), 5.89 (s, 1H), 3.17 (s, 3H), 3.01 (s, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 167.0 (C=O), 142.2 (C), 142.1 (C), 141.6 (C), 133.5 (C), 132.5 (q, *J* = 33.0 Hz, C), 129.3 (CH), 127.5 (CH), 124.3 (CH), 123.2 (q, *J* = 270.8 Hz, C), 122.2 (CH), 121.8 (cinq, *J* = 3.8 Hz, CH), 87.9 (O-CH-N), 49.5 (O-CH_3), 26.6 (N-CH_3).

HRMS (ESI) [M+Na^+] calculated for $C_{18}H_{13}F_6NNaO_2^+$: 412.0743. Found: 412.0715.

6-(3,5-bis(trifluoromethyl)phenyl)-3-methoxy-2-methylisoindolin-1-one (6c')

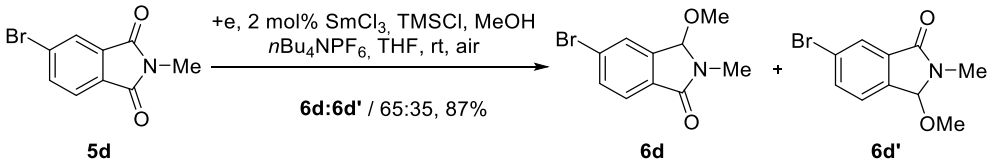


6c'

^{1}H NMR (250 MHz, $CDCl_3$) δ 8.10 (s, 3H), 7.94 (s, 1H), 7.87 (dd, *J* = 7.8, 1.7 Hz, 1H), 7.71 (d, *J* = 7.8 Hz, 1H), 5.88 (s, 1H), 3.16 (s, 3H), 2.99 (s, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 167.0 (C=O), 142.0 (C), 140.7 (C), 140.3 (C), 134.5 (C), 132.5 (q, *J* = 33.0 Hz, C) , 130.9 (CH), 127.4 (CH), 124.3 (CH), 123.2 (q, *J* = 270.8 Hz, C), 122.1 (CH), 121.7 (cinq, *J* = 3.8 Hz, CH), 87.9 (O-CH-N), 49.4(O-CH_3), 26.6 (N-CH_3).

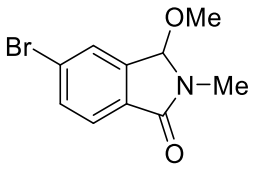
HRMS (ESI) [M+Na^+] calculated for $C_{18}H_{13}F_6NNaO_2^+$: 412.0743. Found: 412.0715.



5d **6d** **6d'**

Following the general procedure, the mixture of 5.6d and 5.6d' was obtained in 87% yield (222 mg). (eluent: ethyl acetate/petane: 1:4).

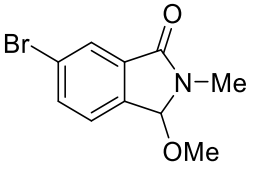
5-bromo-3-methoxy-2-methylisoindolin-1-one (6d)



6d

^{1}H NMR (360 MHz, $CDCl_3$)δ7.67-7.62 (m, 3H), 5.71 (s, 1H), 3.04 (s, 3H), 2.90 (s, 3H).

6-bromo-3-methoxy-2-methylisoindolin-1-one (6d')



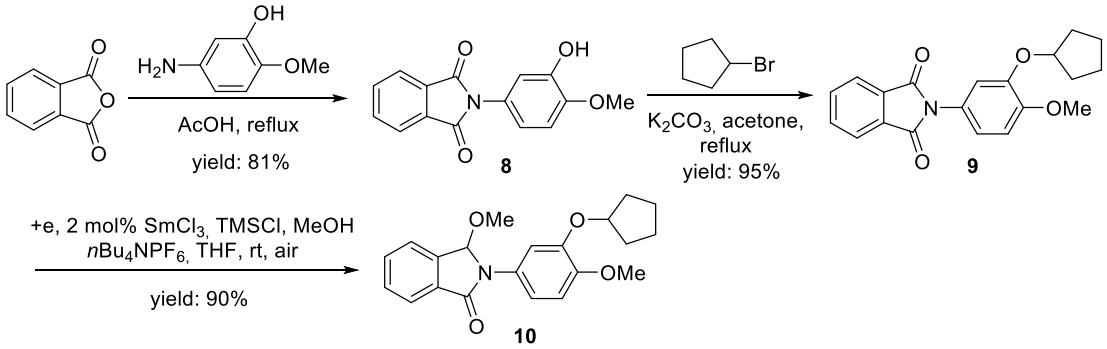
6d'

^{1}H NMR (360 MHz, $CDCl_3$)δ7.92 (d, *J* = 1.8, 1H), 7.70-7.66 (m, 1H), 7.38 (d, *J* = 8.0, 1H), 5.71 (s, 1H), 3.05 (s, 3H), 2.88 (s, 3H).

21d and 21d':**^{13}C NMR (90 MHz, $CDCl_3$)**δ166.8, 166.3, 142.3, 139.0, 135.2, 135.0, 133.4, 132.1, 126.9, 126.8, 126.7, 125.0, 124.9, 124.3, 87.7, 87.5, 49.53 (O-CH3, **2o'**), 49.3, 26.6, 26.6.

HRMS (ESI) [M+Na^+] calculated for $C_{10}H_{10}BrNNaO_2^+$: 277.9787. Found: 277.9781.

7. Synthesis of 3-methoxy-2-(3-cyclopentyloxy-4-methoxyphenyl)-1-isoindolinone (10)



2-(3-hydroxy-4-methoxyphenyl)isoindoline-1,3-dione (8)

a mixture of 5-amino-2-methoxyphenol (1.39 g, 10.0 mmol) and Phthalic anhydride (1.48 g, 10.0 mmol) in acetic acid (20 ml) was refluxed overnight with stirring. After cooling to the room temperature, diluted by water (20 ml) and extracted with ethyl acetate (2x10 ml). The organic phase was washed by saturated $NaHCO_3$ (5 ml) and brine (5 ml), dried over $MgSO_4$, concentrated and purified by flash chromatography on silica gel (eluent: ethyl acetate/pentane: 2:1) to afford the product as a white solid (2.18 g, 81%).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.96 (dd, *J* = 5.4, 3.1 Hz, 2H), 7.80 (dd, *J* = 5.5, 3.1 Hz, 2H), 6.96 (ddd, *J* = 10.8, 7.4, 2.3 Hz, 3H), 5.77 (s, 1H), 3.96 (s, 3H).

^{13}C NMR (90 MHz, $CDCl_3$) δ 167.5 (C=O), 146.5 (C), 146.0 (C), 134.3 (2xCH), 131.8 (CH), 124.8 (C), 123.7 (2xCH), 118.7 (CH), 113.5 (CH), 110.7 (CH), 56.1 (O-CH_3).

HRMS (ESI) [M+Na^+] calculated for $C_{15}H_{11}NNaO_4^+$: 292.0580. Found: 292.0579.

2-(3-(cyclopentyloxy)-4-methoxyphenyl)isoindoline-1,3-dione (9)

To a mixture of **5.8** (2.15 g, 8.0 mmol) and K_2CO_3 (5.52 g, 40 mmol) in acetone (40 ml),bromocyclopentane (5.92 g, 4.3 ml, 40 mmol) was added with stirring, then refluxed overnight. Cooled to room temperature, diluted by ethyl acetate (50 ml), filtered the turbid liquid to remove the salt, concentrated and purified by flash chromatography on silica gel (eluent: ethyl acetate/pentane: 1:1) to afford the product as a white solid (2.56 g, 95%).

^{1}H NMR (360 MHz, $CDCl_3$) δ 7.91 (s, 2H), 7.76 (s, 2H), 6.95 (s, 3H), 4.76 (s, 1H), 3.87 (s, 3H), 1.97 – 1.46 (m, 8H).

^{13}C NMR (90 MHz, $CDCl_3$) δ 167.6 (C=O), 149.7 (C), 147.9 (C), 134.3 (2xCH), 131.8 (CH), 124.2 (C), 123.6 (2xCH), 118.8 (CH), 113.3 (CH), 111.7 (CH), 80.6 (O-CH), 56.2 (O-CH_3), 32.7 (2xCH_2), 24.0 (2xCH_2).

HRMS (ESI) [M+H^+] calculated for $C_{20}H_{19}NNaO_4^+$: 360.1206. Found: 360.1199.

3-methoxy-2-(3-cyclopentyloxy-4-methoxyphenyl)-1-isoindolinone (10)

Reactions were carried out in a three necked cell containing a magnetic stirring bar, samarium cathode (20 cm^2 area), glassy carbon anode (20 cm^2 area) and SCE as reference electrode. nBu_4NPF_6 (312 mg, 0.8 mmol, 0.04 mol/L in THF) as the electrolyte, **24** (1.69 g, 5.0 mmol), methanol (1.60 g, 50 mmol, 2.0 ml) and $SmCl_3$ (25.0 mg, 0.1 mmol)were added in anhydrous THF (20 ml), TMSCl (1.36 g, 12.5 mmol, 1.6 ml) was added dropwise with stirring, then electrolysis was performed at i = 50 mA during 3 hours. After the reaction was complete, the mixture was quenched by 0.1 mol/L HCl (50 ml), extracted with ethyl acetate (3×20 mL). The combined organic layer was washed by 10% $Na_2S_2O_3$ (20 ml) and brine, dried over $MgSO_4$ and the solvent was removed under reduced pressure. The crude product was purified by flash chromatography on silica gel (eluent: ethyl acetate/pentane: 1:3) to afford the product as a colorless oil (1.59 g, 90%).

^{1}H NMR (360 MHz, $CDCl_3$) δ 7.91 – 7.84 (m, 1H), 7.58 (ddd, *J* = 21.1, 10.1, 4.2 Hz, 4H), 7.20 (dd, *J* = 8.7, 2.5 Hz, 1H), 6.90 (d, *J* = 8.8 Hz, 1H), 6.36 (s, 1H), 4.82 (dt, *J* = 9.6, 3.2 Hz, 1H), 3.85 (s, 3H), 2.93 (s, 3H), 1.99 – 1.80 (m, 6H), 1.64 – 1.54 (m, 2H).

^{13}C NMR (90 MHz, $CDCl_3$) δ 166.7 (C=O), 147.8 (C), 147.6 (C), 139.7 (C), 133.0 (C), 132.6 (CH), 130.5 (C), 130.3 (CH), 123.7 (CH), 123.4 (CH), 113.9 (CH), 112.0 (CH), 109.7 (CH), 87.8 (O-CH-N), 80.5 (O-CH), 56.2 (O-CH_3), 49.3 (O-CH_3), 32.8 (2xCH_2), 24.1 (2xCH_2).

HRMS (ESI) [M+H^+] calculated for $C_{20}H_{19}NNaO_4^+$: 360.1206. Found: 360.1199.

8. References

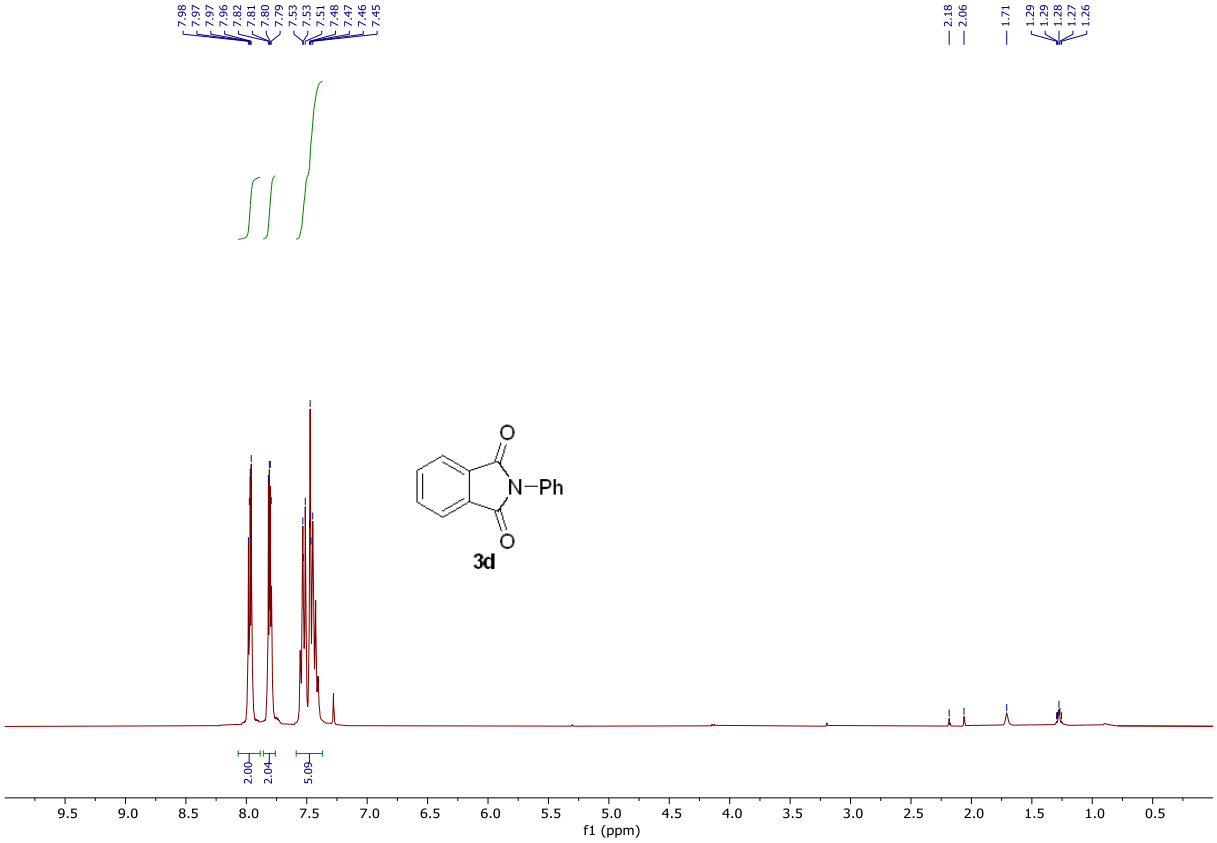
[1]- S. P. O. Assis, T. Gomes Araùjo, V. L. M. Sena, M. T. J. A. Catanho, M. N. Ramos, R. M. Srivastava, V. L. M. Lima, *Med Chem Res*, **2014**, *23*,708-716.

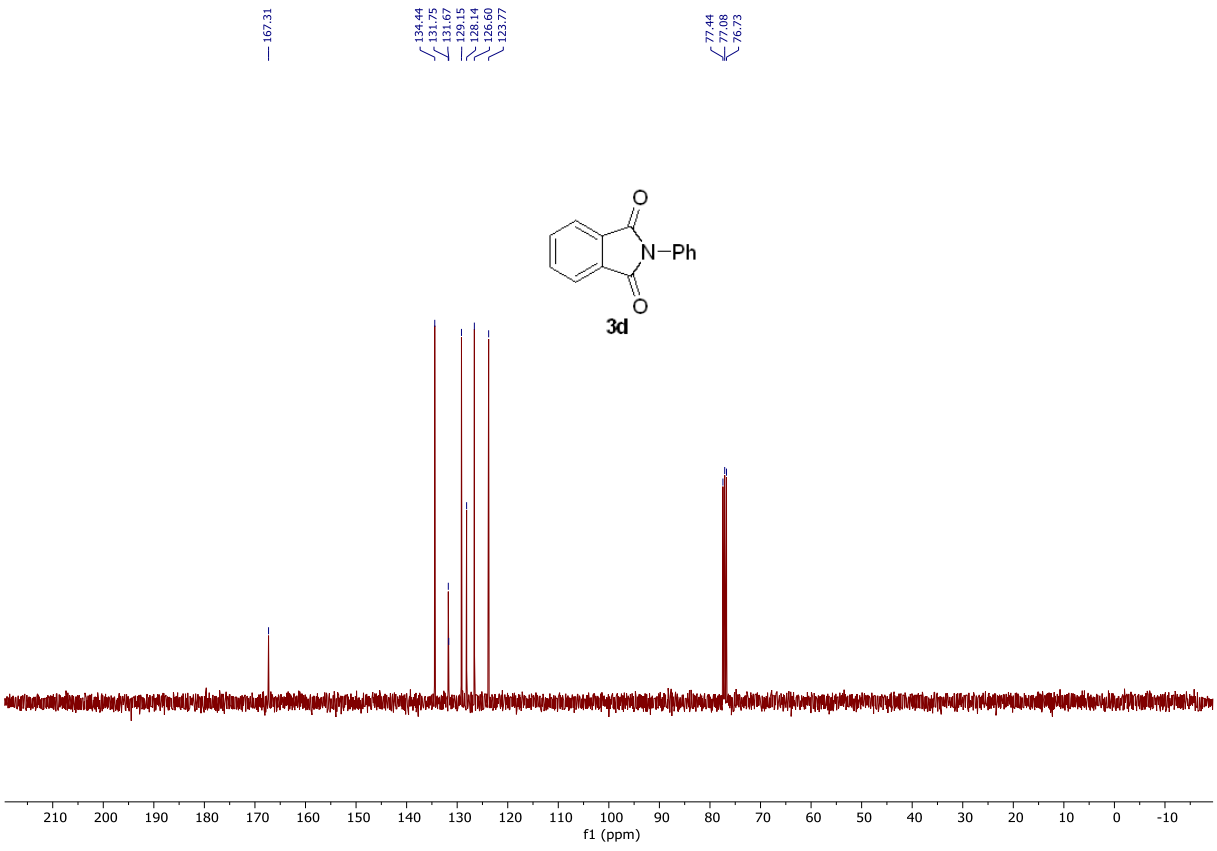
[2]- X.-M. Chen, X.-S. Ning, Y.-B. Kang, *Org. Lett.,* **2016**, *18*, 5368-5371.

[3]- M. M. Haugland, S. Borsley, D. F. Cairns-Gibson, A. Elmi, S. L. Cockroft, *ACS Nano*, **2019**, *13*, 4101-4110.

[4]- P. S. Phataka, R. D. Bakalea, S. T. Dhumalb, L. K Dahiwadec, P. B. Choudharic, V. S. Krishnad, D. Sriramd, K. P. Havala, *Synthetic Communications*, **2019**, *49*, 2017-2028.

9. NMR Spectra



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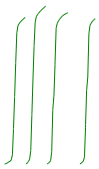
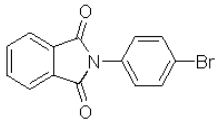
7.28

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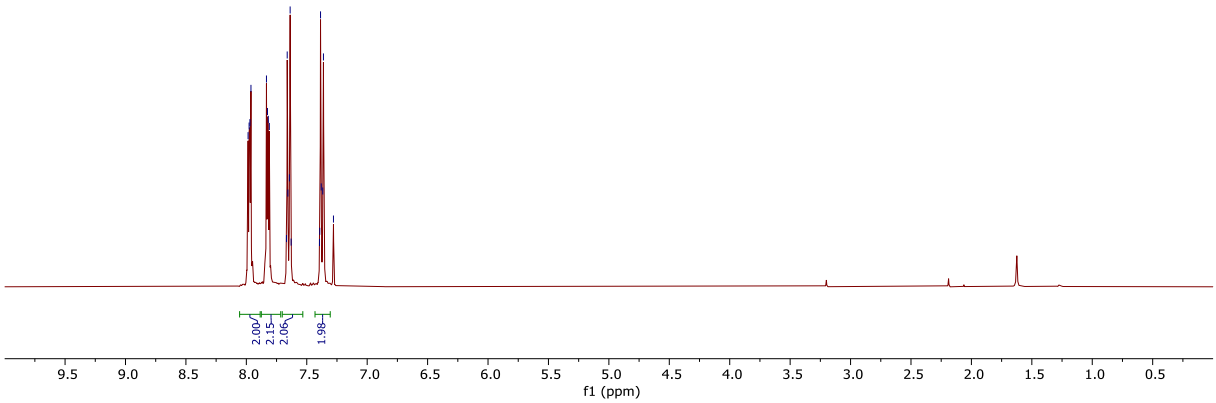
N
Br
O

3e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9.0
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8.0
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6.5
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5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)


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131.60
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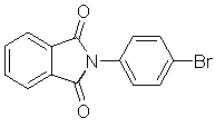
77.05
76.69


O
N
Br
O

3e

210
200
190
180
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150
140
130
120
110
100
90
80
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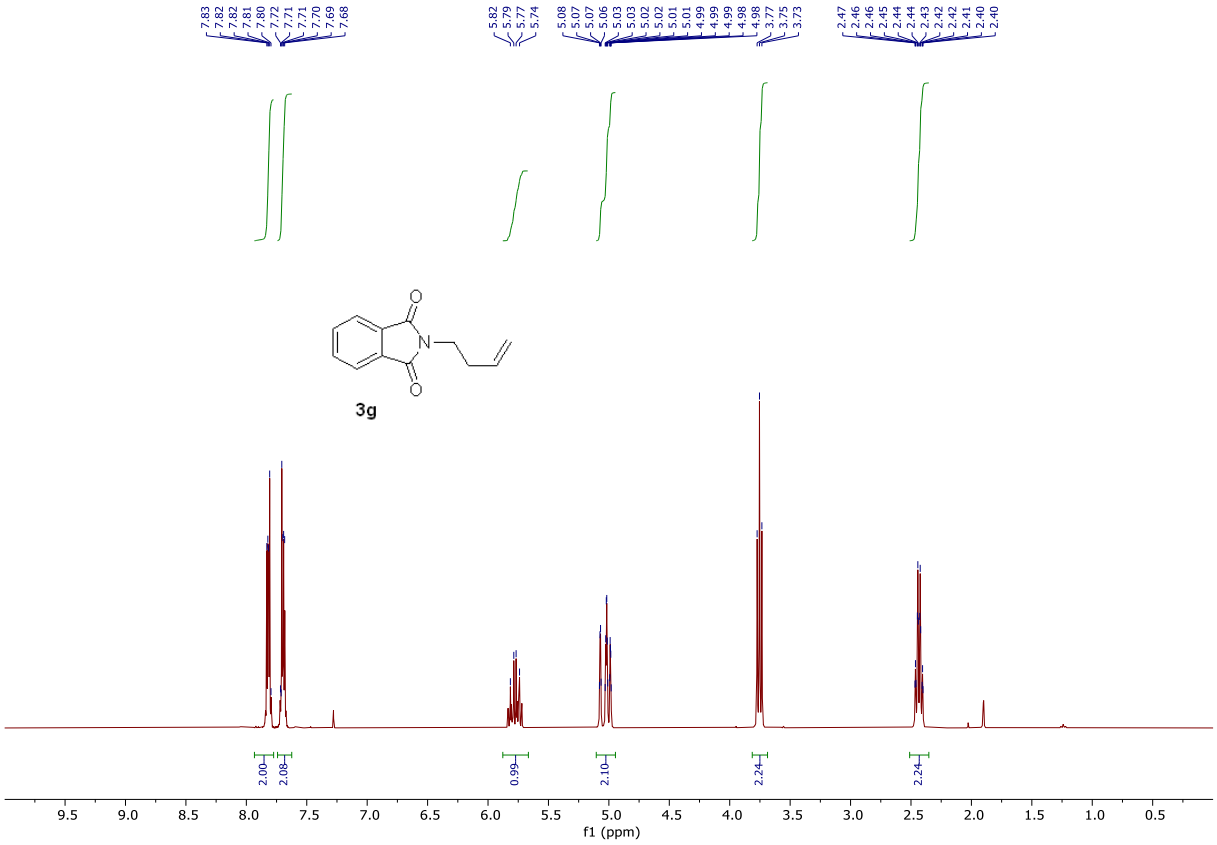
60
50
40
30
20
10
0
-10
f1 (ppm)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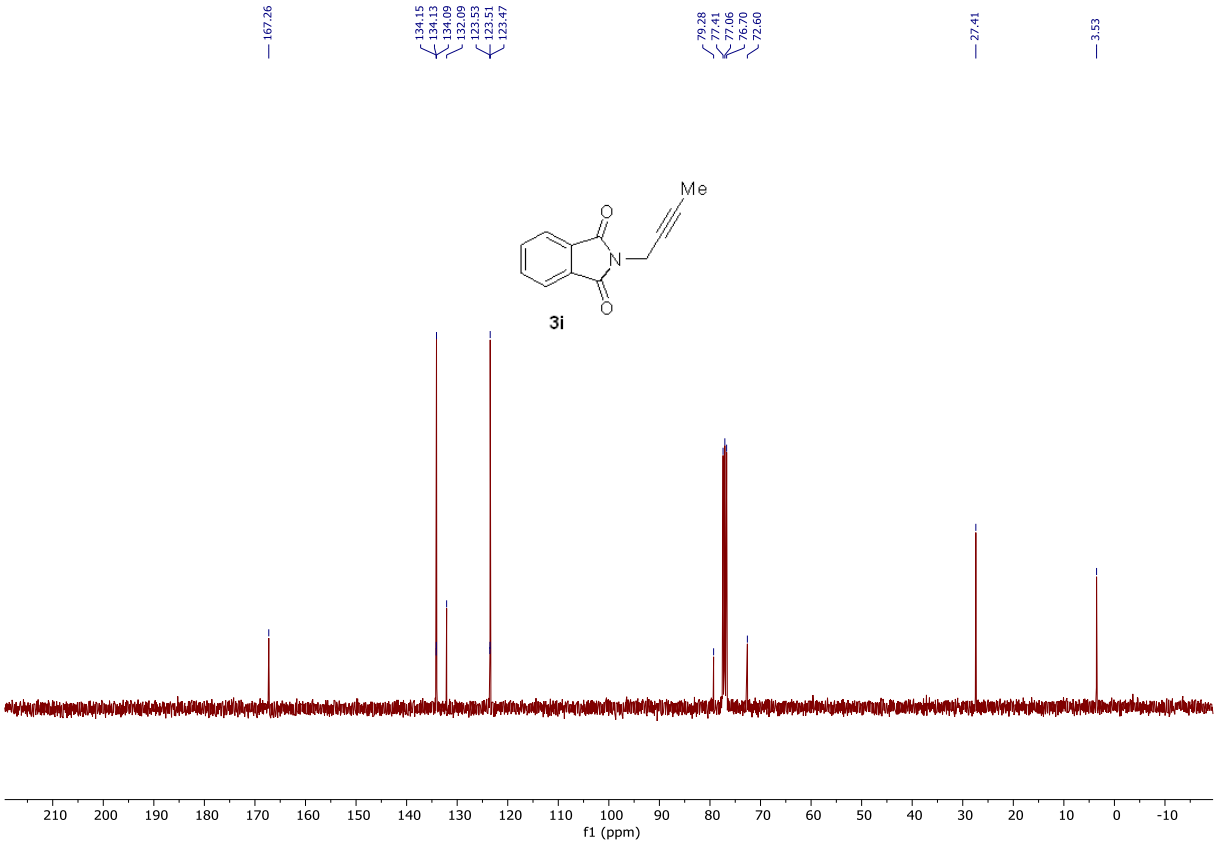
f1 (ppm)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27.41
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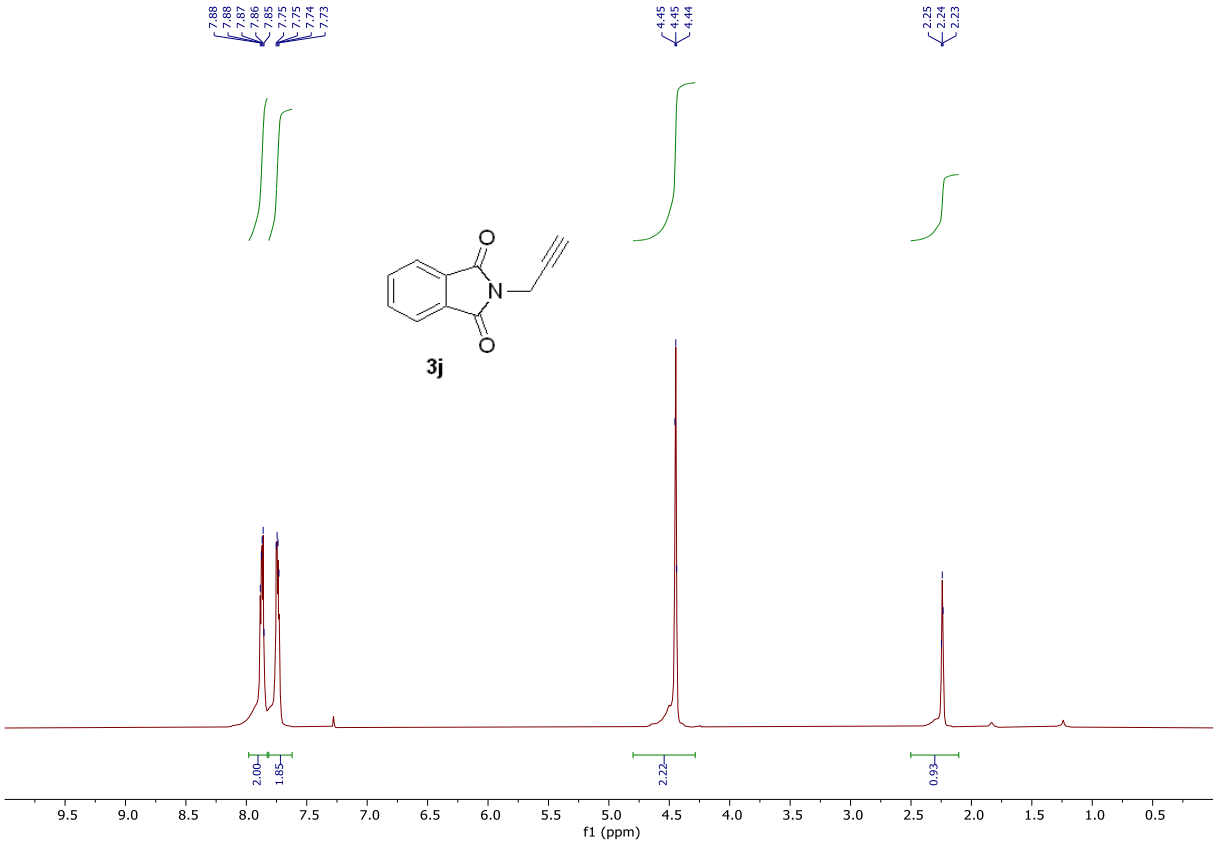
Me
3i
f1 (ppm)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O
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2.22
0.93
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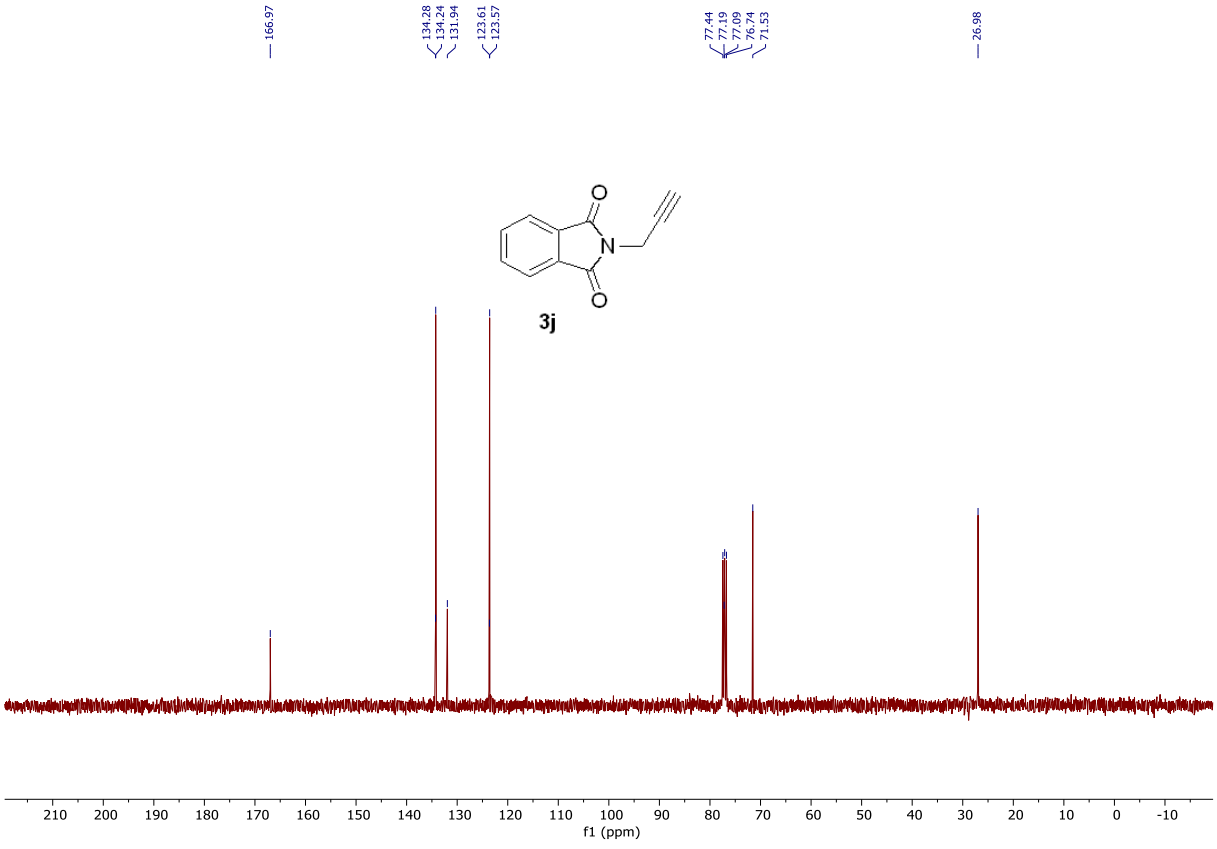
f1 (ppm)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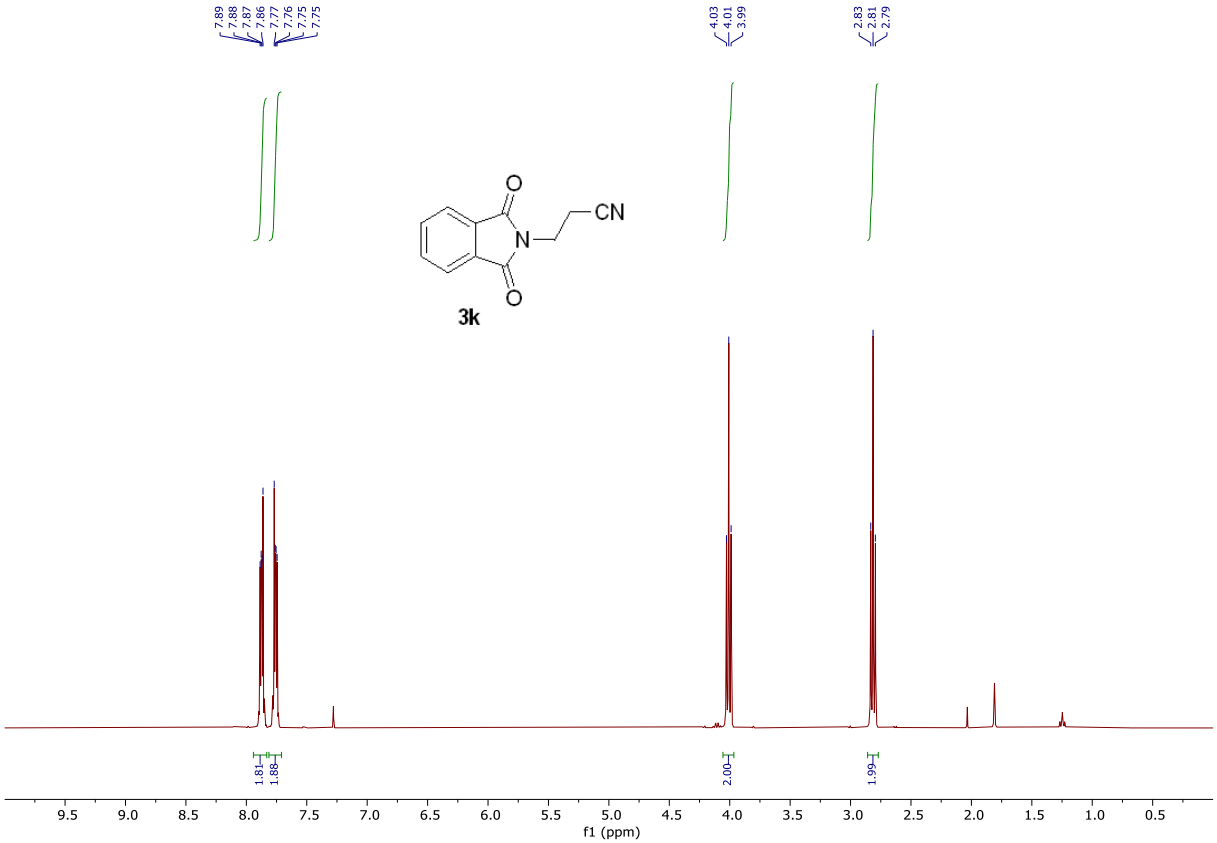
f1 (ppm)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2.00
1.99
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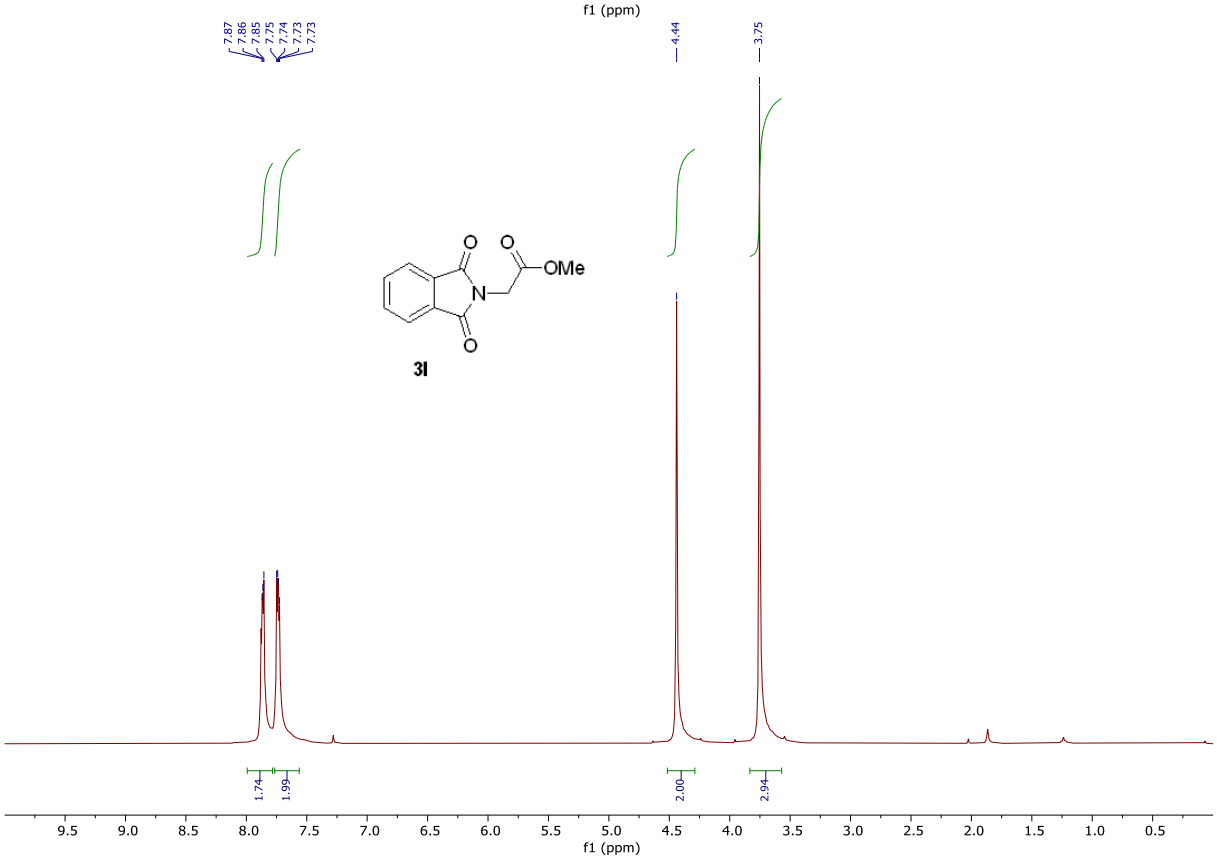
f1 (ppm)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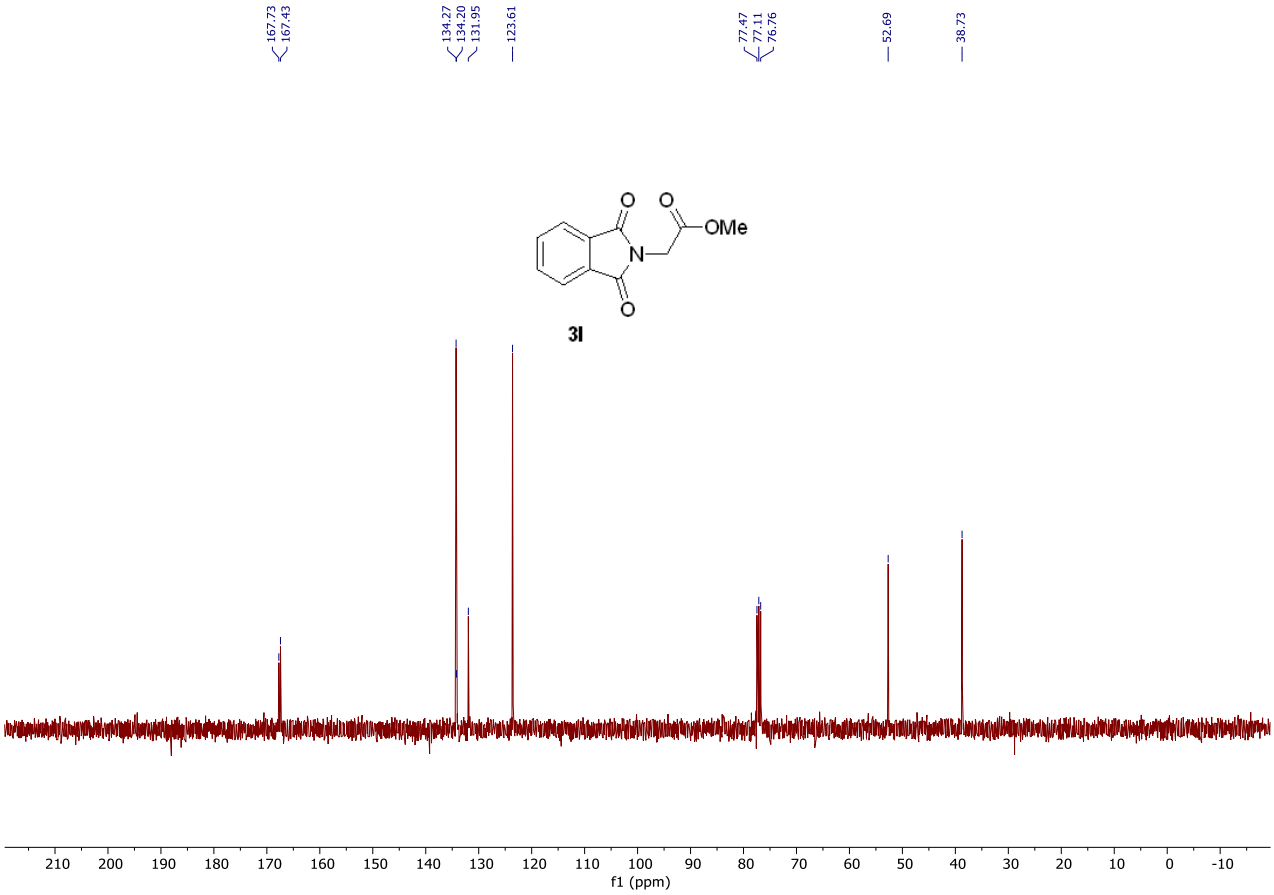
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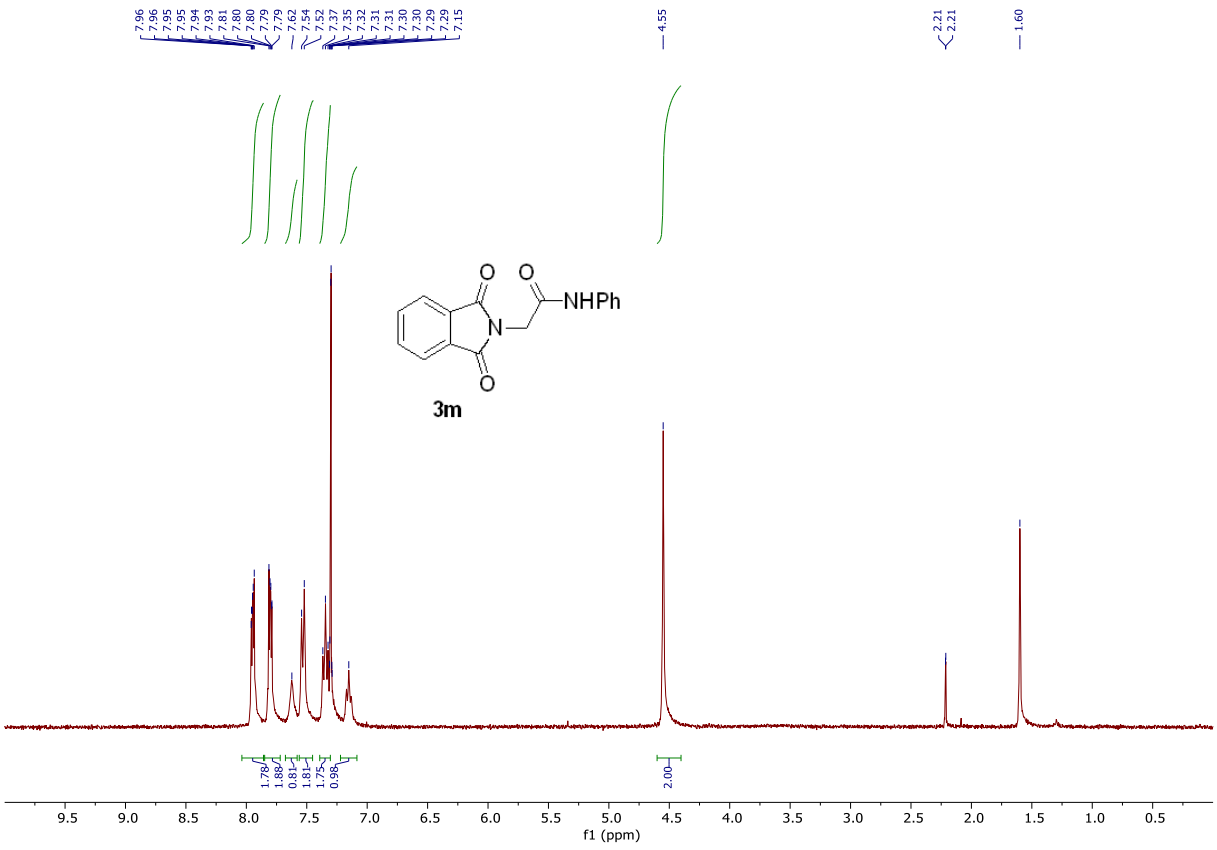
f1 (ppm)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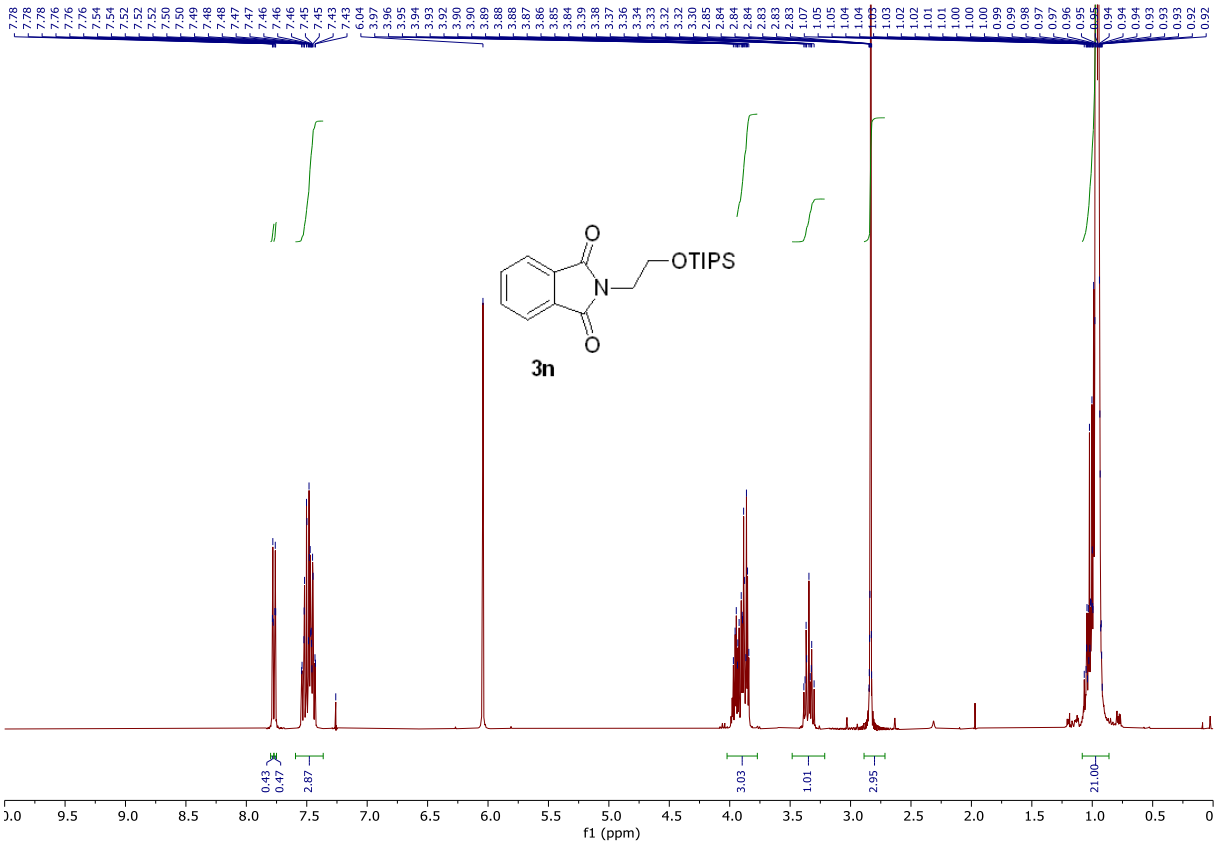
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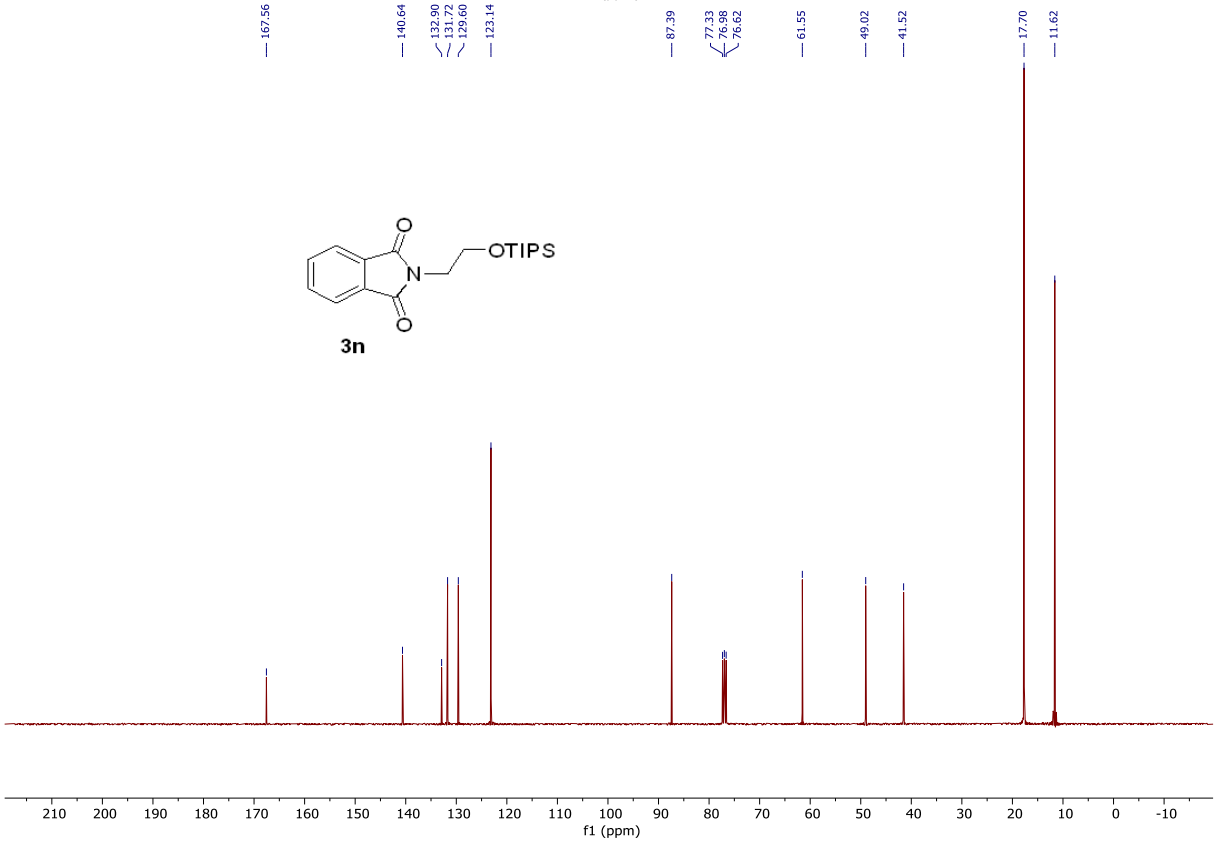
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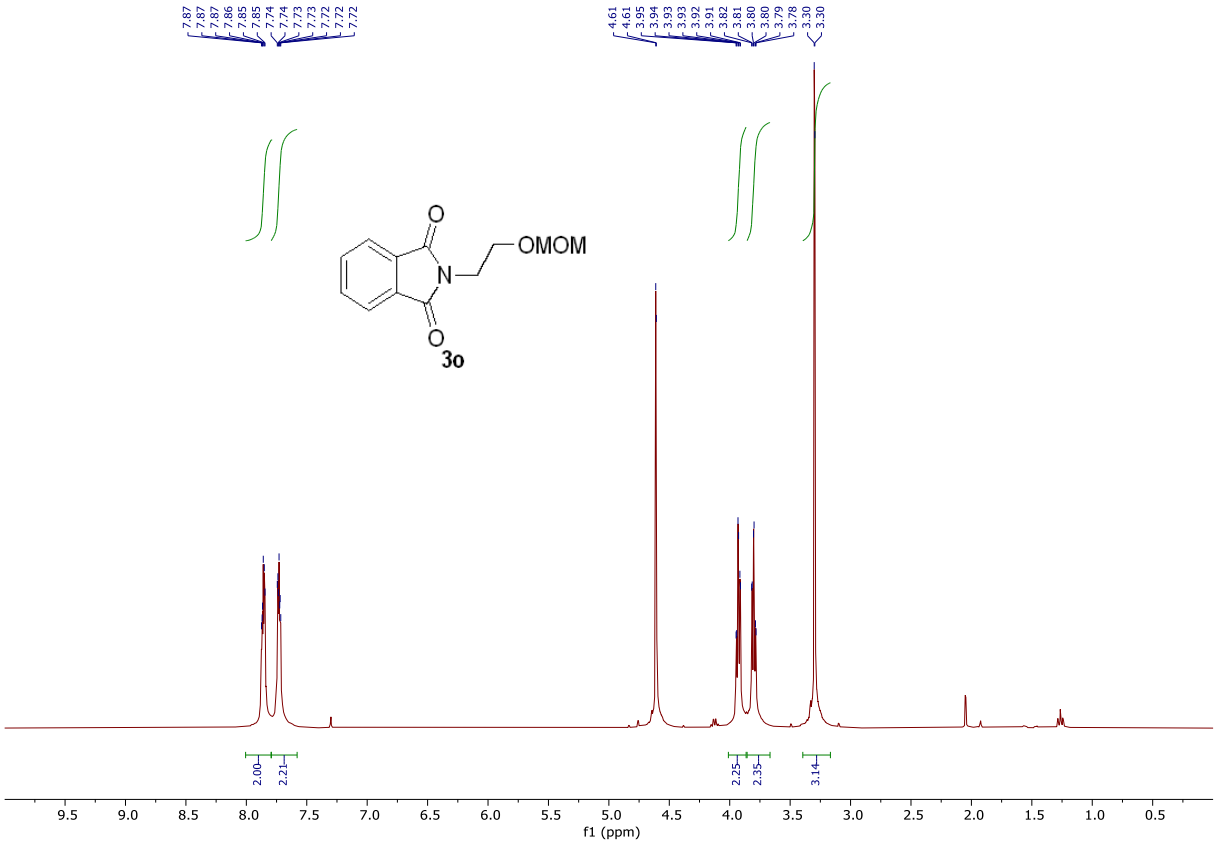
f1 (ppm)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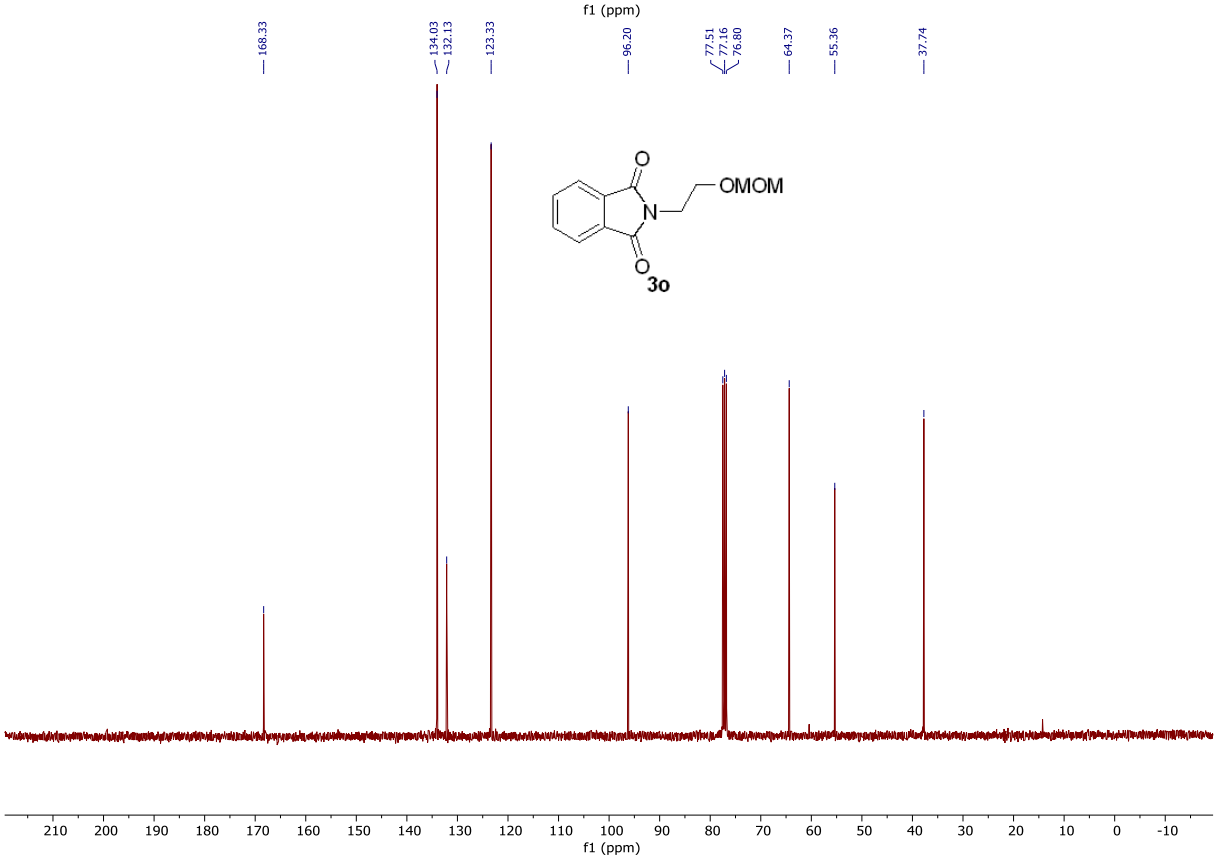
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120
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100
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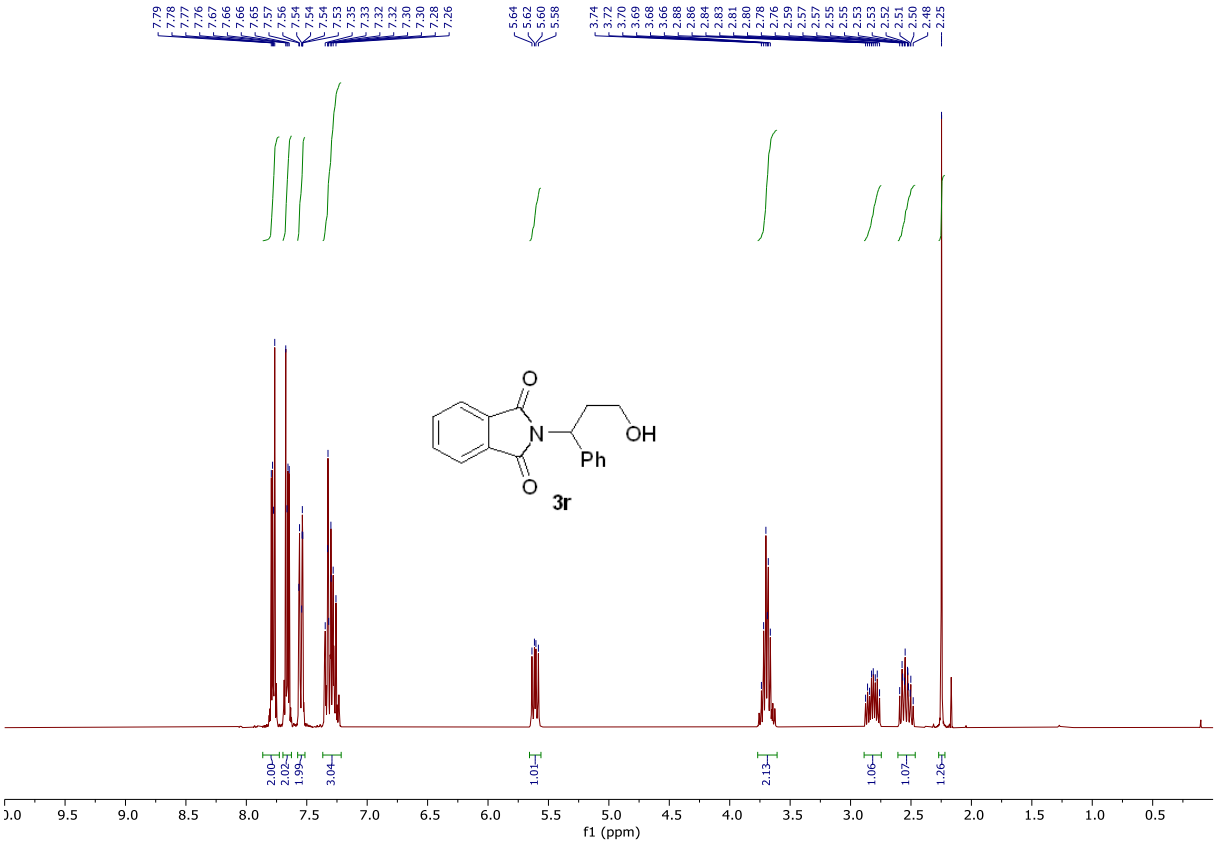
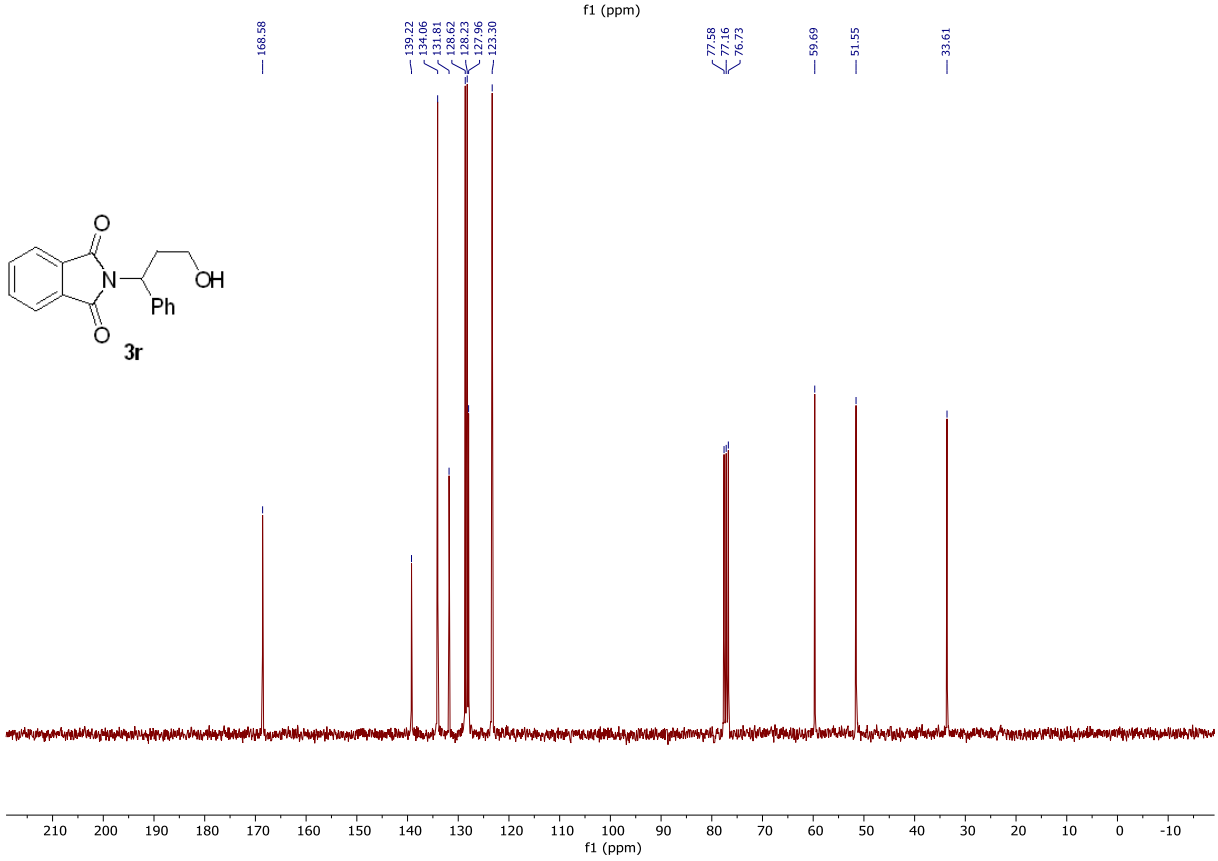
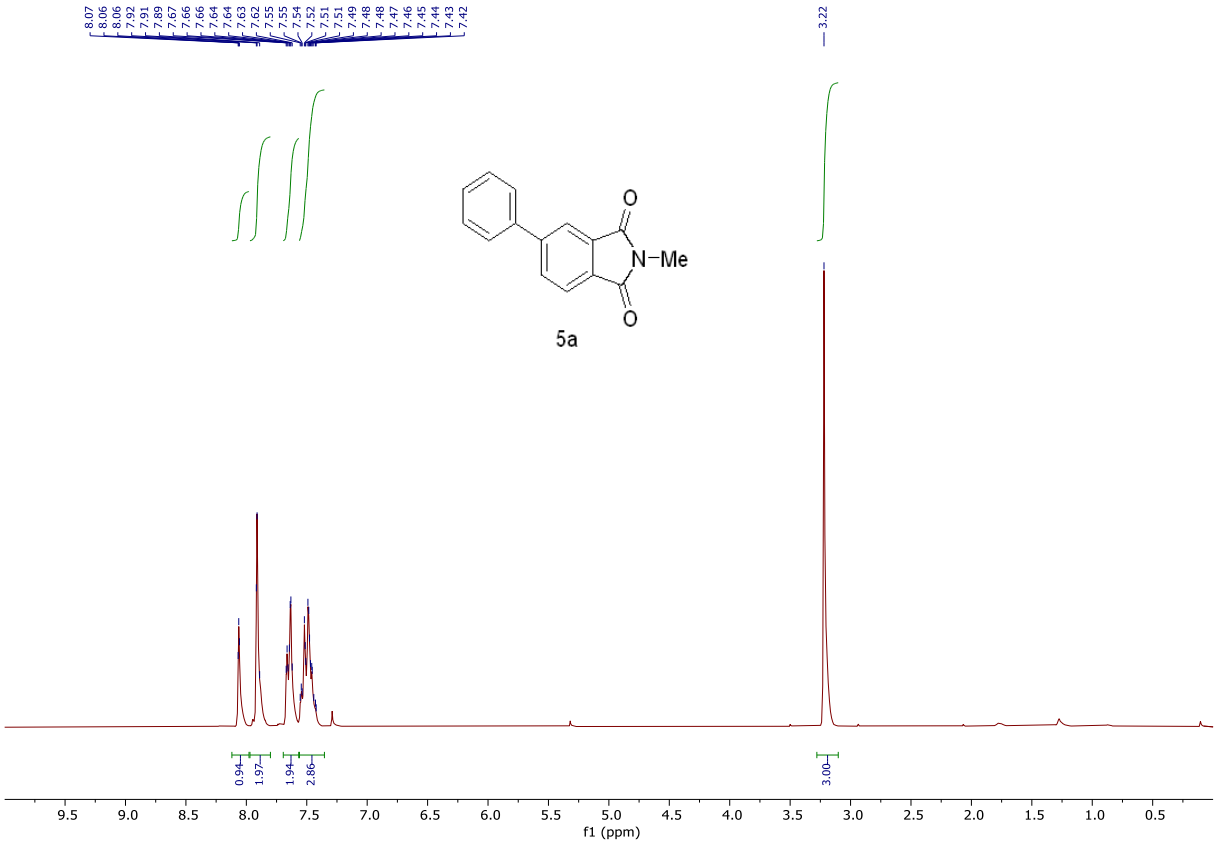
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


7.79 7.78 7.77 7.76 7.67 7.66 7.66 7.65 7.57 7.56 7.54 7.54 7.54 7.53 7.35 7.33 7.32 7.32 7.30 7.30 7.28 7.26
5.64 5.62 5.60 5.58
3.74 3.72 3.70 3.69 3.68 3.66 2.88 2.86 2.84 2.83 2.81 2.80 2.78 2.76 2.59 2.57 2.57 2.55 2.55 2.53 2.53 2.52 2.51 2.50 2.48 2.25
O
N
OH
Ph
O
3r
2.00 2.02 1.99 3.04 1.01 2.13 1.06 1.07 1.26
f1 (ppm)


168.58
139.22 134.06 131.81 128.62 128.23 127.96 123.30
77.58 77.16 76.73
59.69
51.55
33.61
O
N
OH
Ph
O
3r
f1 (ppm)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8.06
8.06
8.06
7.92
7.91
7.89
7.67
7.66
7.66
7.64
7.64
7.63
7.62
7.55
7.55
7.54
7.52
7.51
7.51
7.49
7.48
7.48
7.47
7.46
7.45
7.44
7.43
7.42
3.22
O
N−Me
O
5a
0.94
1.97
1.94
2.86
3.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
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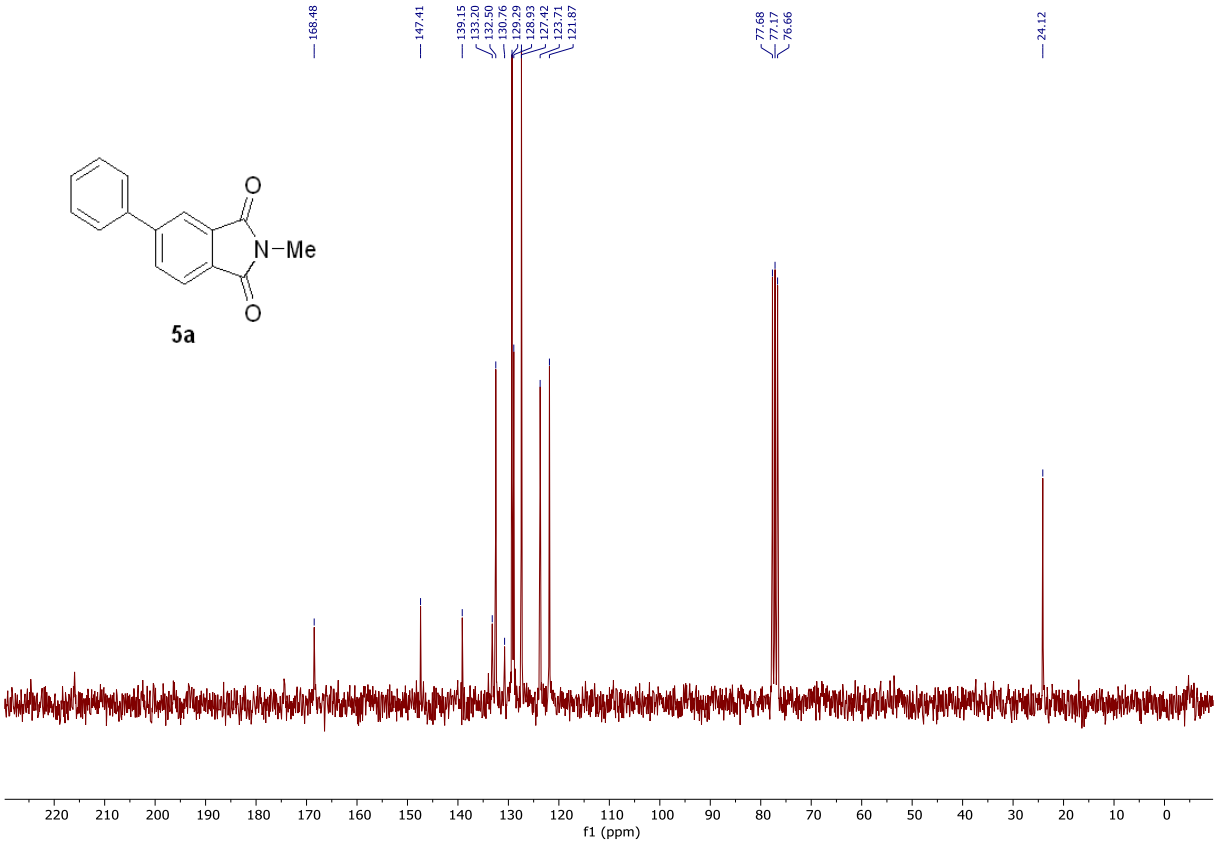
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)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147.41
139.15
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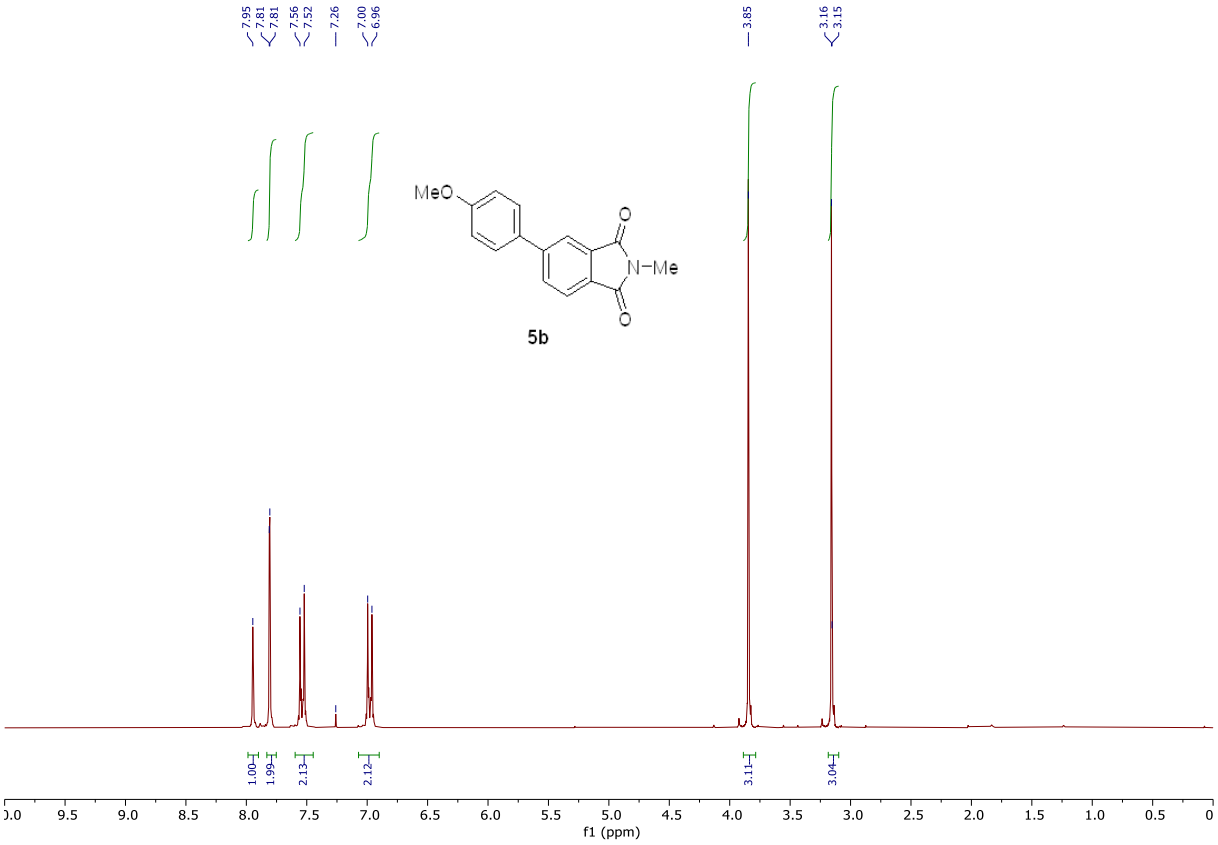
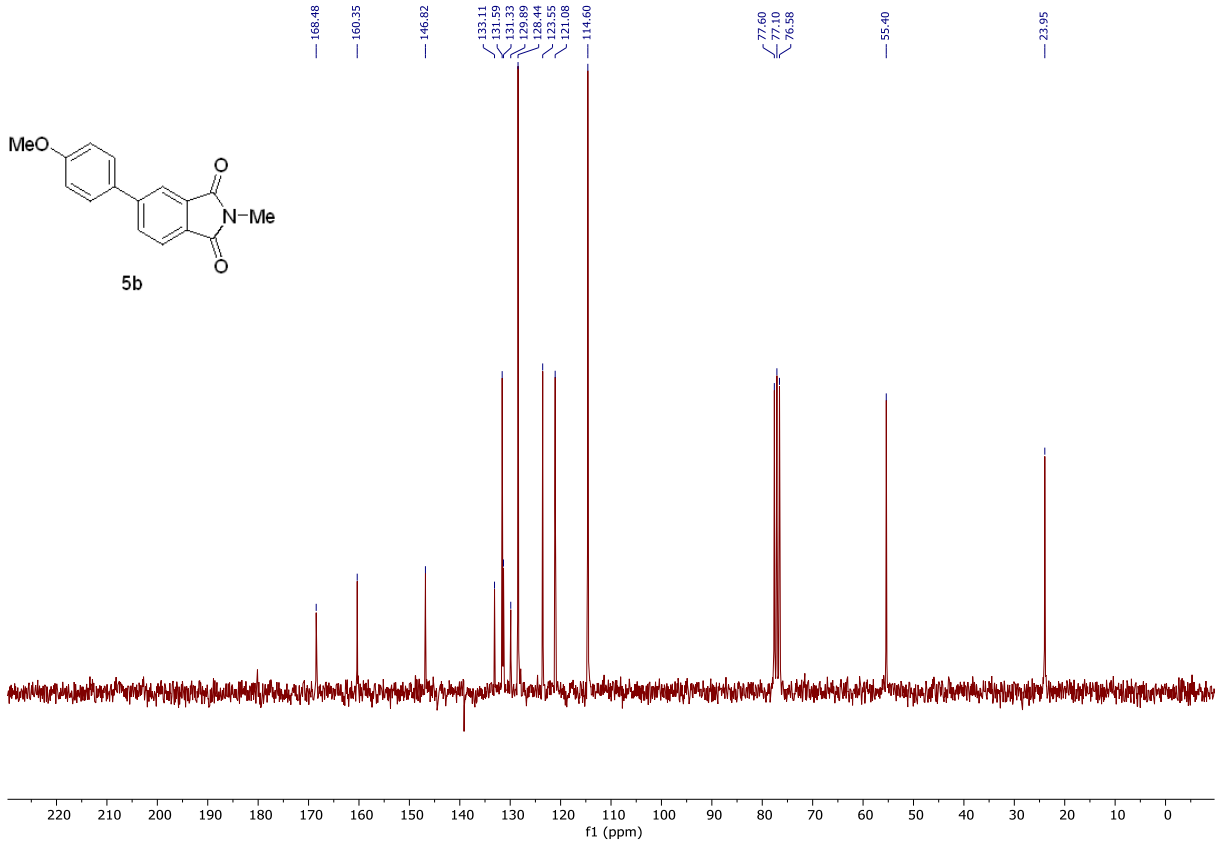
132.50
130.76
129.29
128.93
127.42
123.71
121.87
77.68
77.17
76.66
24.12
O
N−Me
O
5a
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.95
7.81
7.81
7.56
7.52
7.26
7.00
6.96
3.85
3.16
3.15
MeO
N–Me
O
O
5b
1.00
1.99
2.13
2.12
3.11
3.04
f1 (ppm)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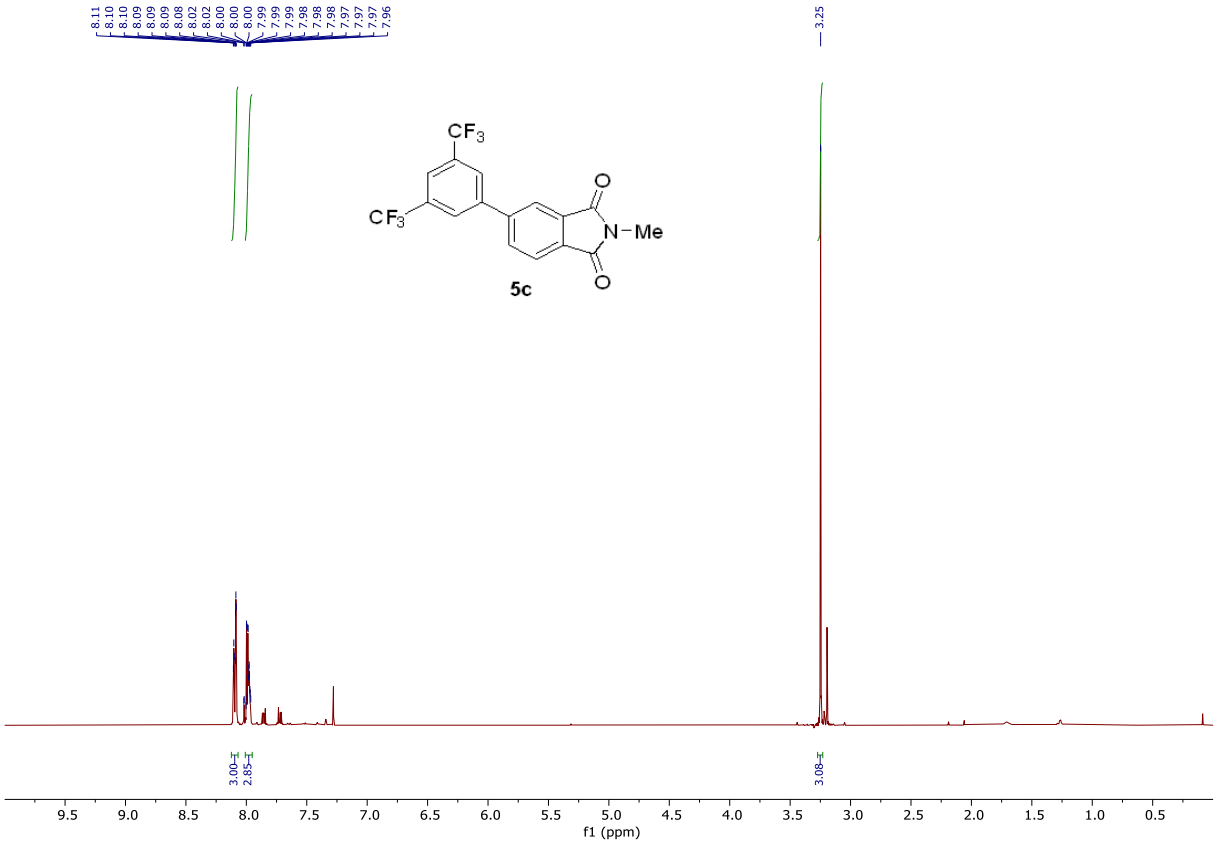
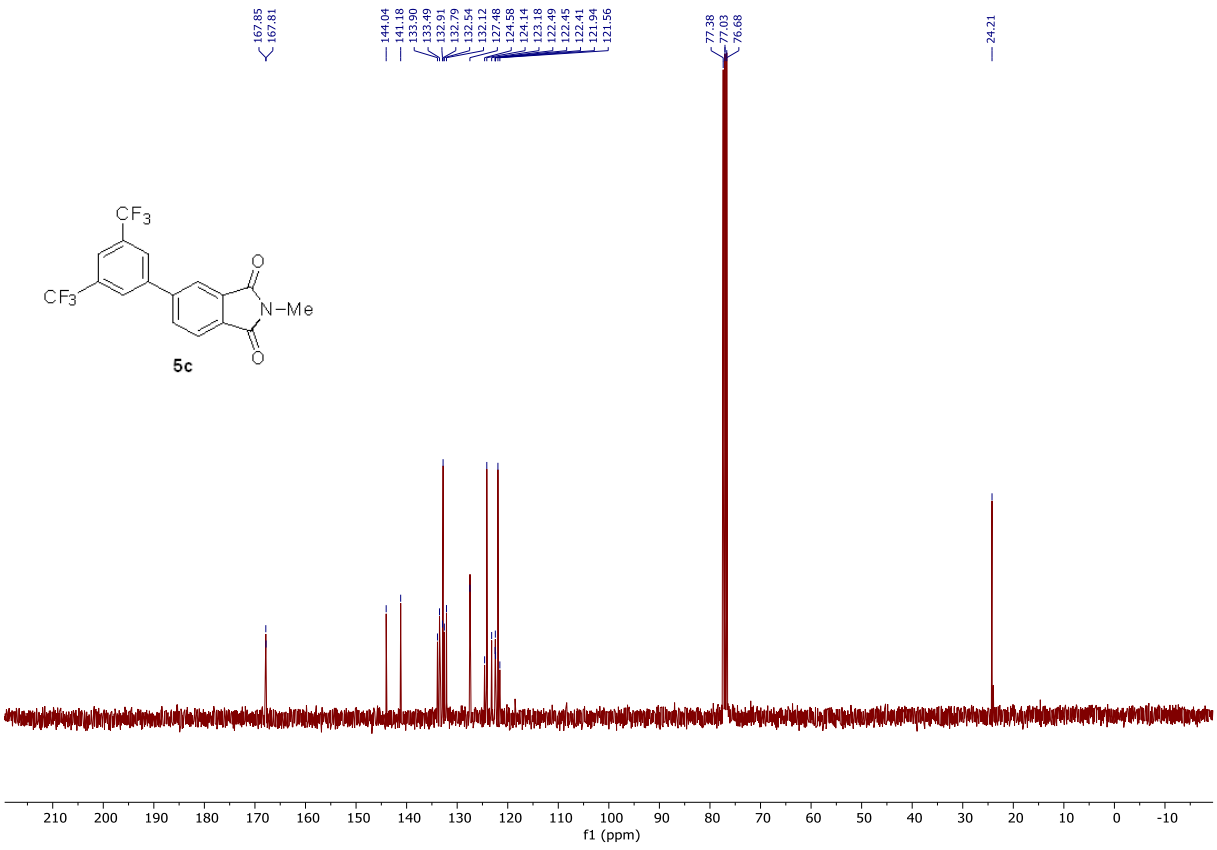
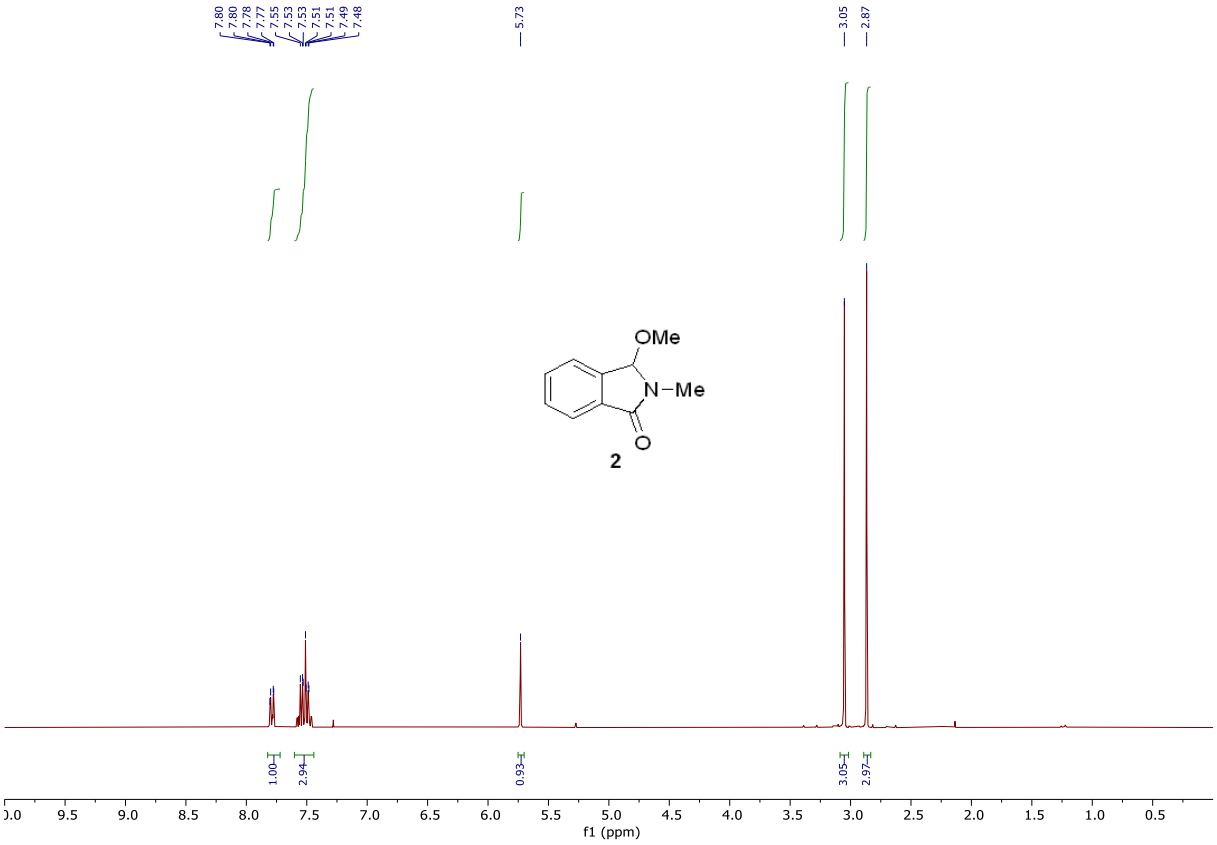
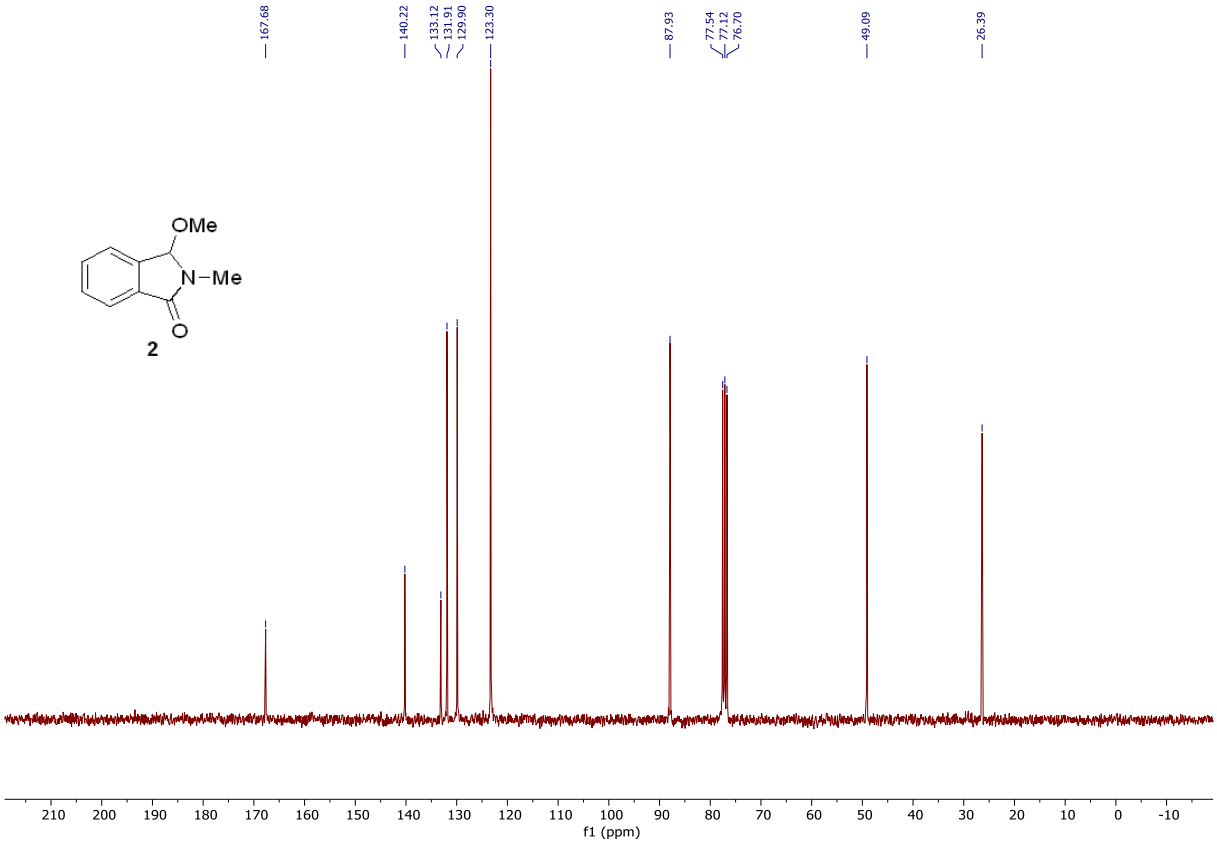
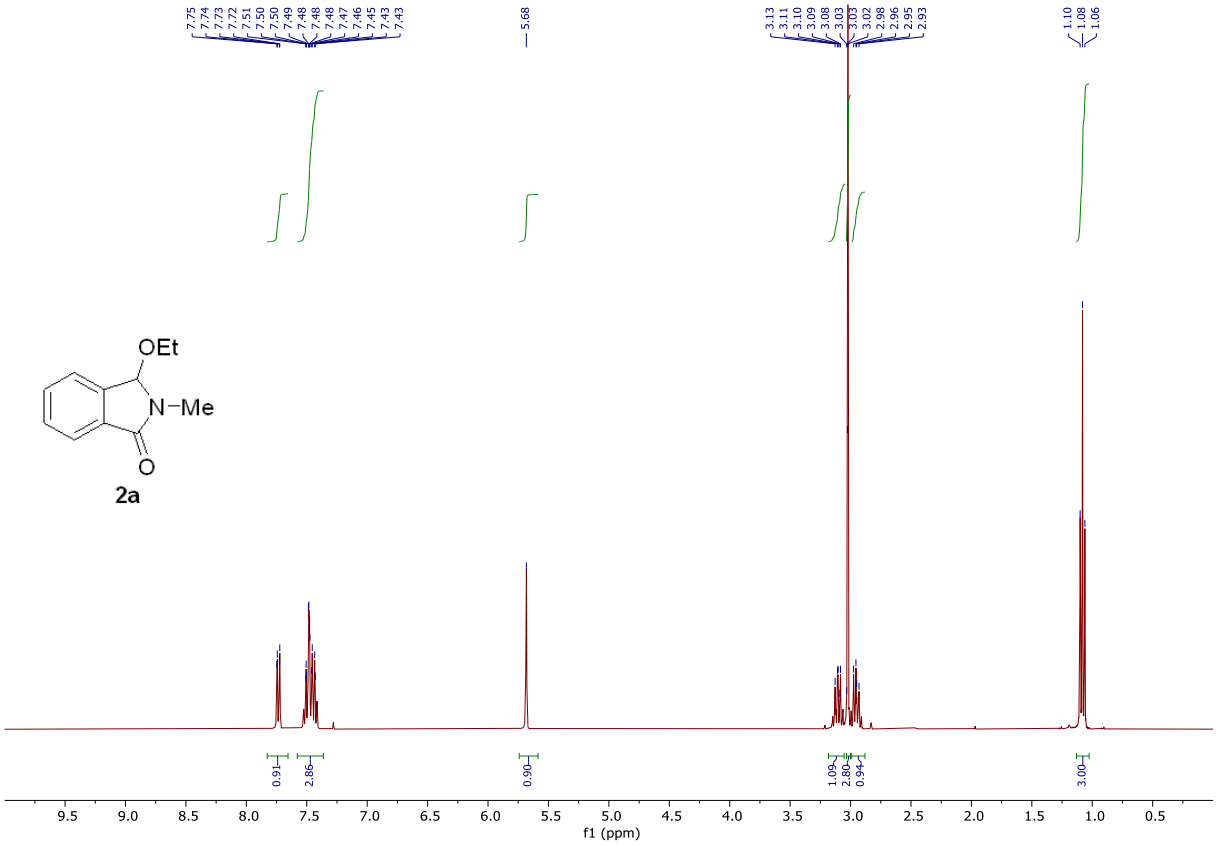
160.35
146.82
133.11
131.59
131.33
129.89
128.44
123.55
121.08
114.60
77.60
77.10
76.58
55.40
23.95
MeO
N–Me
O
O
5b
f1 (ppm)

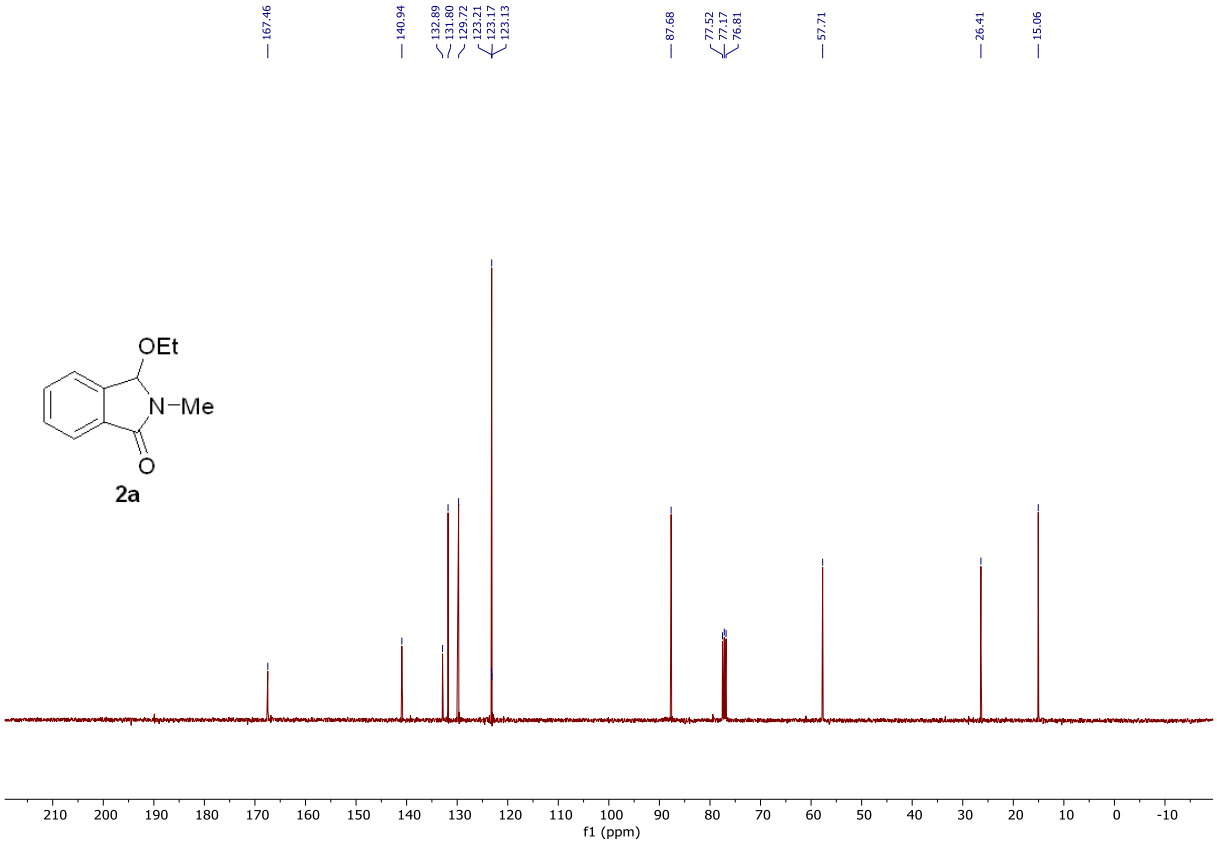

8.11
8.10
8.10
8.09
8.09
8.09
8.08
8.02
8.02
8.00
8.00
8.00
7.99
7.99
7.99
7.98
7.98
7.98
7.97
7.97
7.97
7.96
3.25
CF3
CF3
O
N−Me
O
5c
3.00
2.85
3.08
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)


167.85
167.81
144.04
141.18
133.90
133.49
132.91
132.79
132.54
132.12
127.48
124.58
124.14
123.18
122.49
122.45
122.41
121.94
121.56
77.38
77.03
76.68
24.21
CF3
CF3
O
N−Me
O
5c
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)


7.80
7.80
7.78
7.77
7.55
7.53
7.53
7.51
7.51
7.49
7.48
5.73
3.05
2.87
OMe
N−Me
O
2
1.00
2.94
0.93
3.05
2.97
f1 (ppm)


167.68
140.22
133.12
131.91
129.90
123.30
87.93
77.54
77.12
76.70
49.09
26.39
OMe
N−Me
O
2
f1 (ppm)


7.75
7.74
7.73
7.72
7.51
7.50
7.49
7.48
7.48
7.47
7.46
7.45
7.43
7.43
5.68
3.13
3.11
3.10
3.09
3.08
3.03
3.03
3.02
2.98
2.96
2.95
2.93
1.10
1.08
1.06
OEt
N–Me
O
2a
0.91
2.86
0.90
1.09
2.80
0.94
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
f1 (ppm)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140.94
132.89
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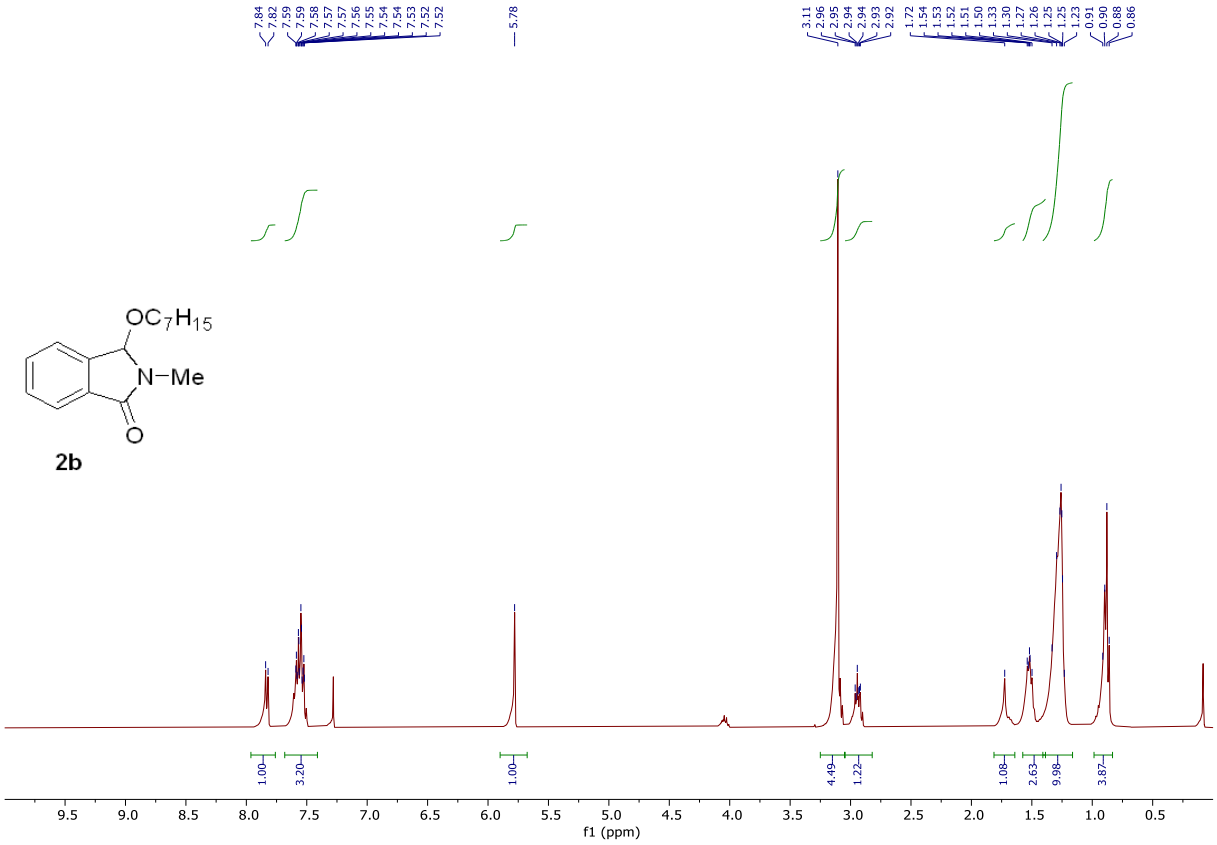
129.72
123.21
123.17
123.13
87.68
77.52
77.17
76.81
57.71
26.41
15.06
OEt
N–Me
O
2a
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)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7.82
7.59
7.58
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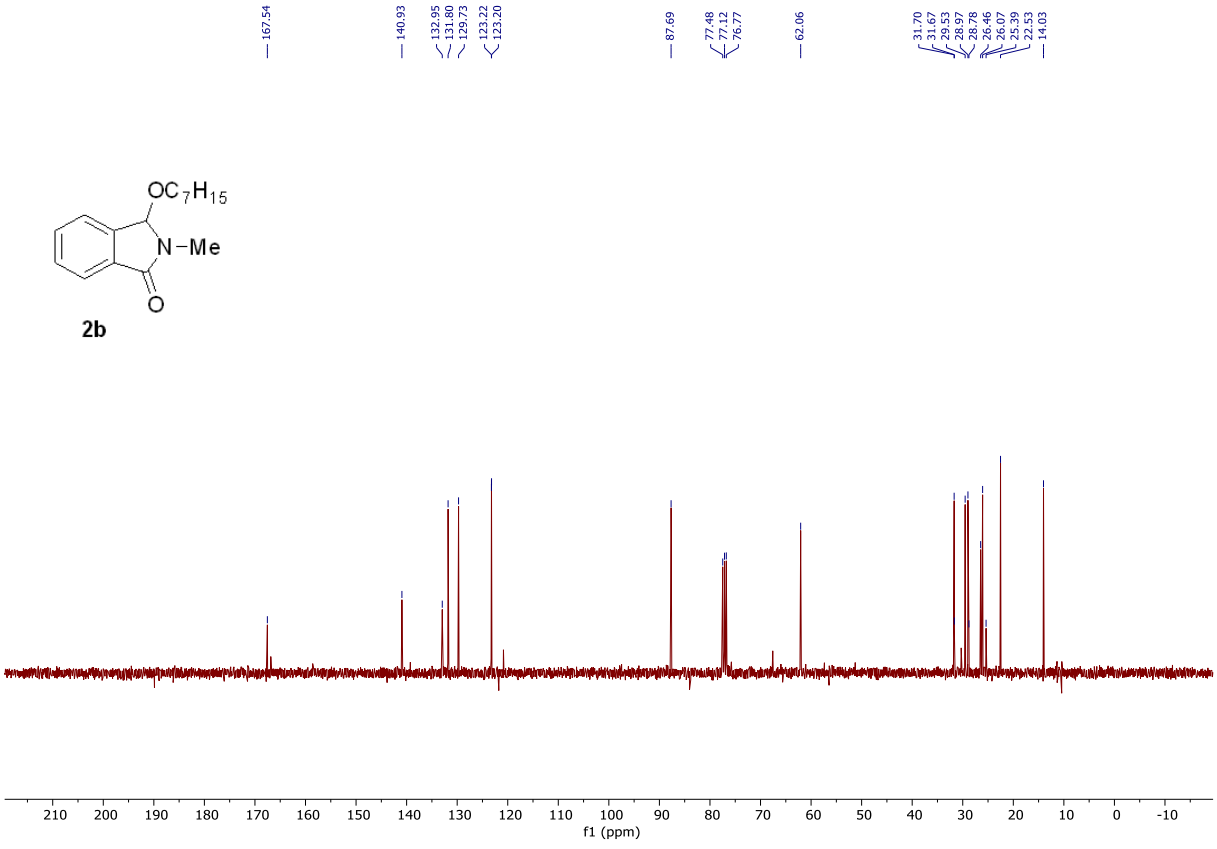
7.57
7.56
7.55
7.54
7.54
7.53
7.52
7.52
5.78
3.11
2.96
2.95
2.94
2.94
2.93
2.92
1.72
1.54
1.53
1.52
1.51
1.50
1.33
1.30
1.27
1.26
1.25
1.25
1.23
0.91
0.90
0.88
0.86
OC7H15
N−Me
O
2b
1.00
3.20
1.00
4.49
1.22
1.08
2.63
9.98
3.87
f1 (ppm)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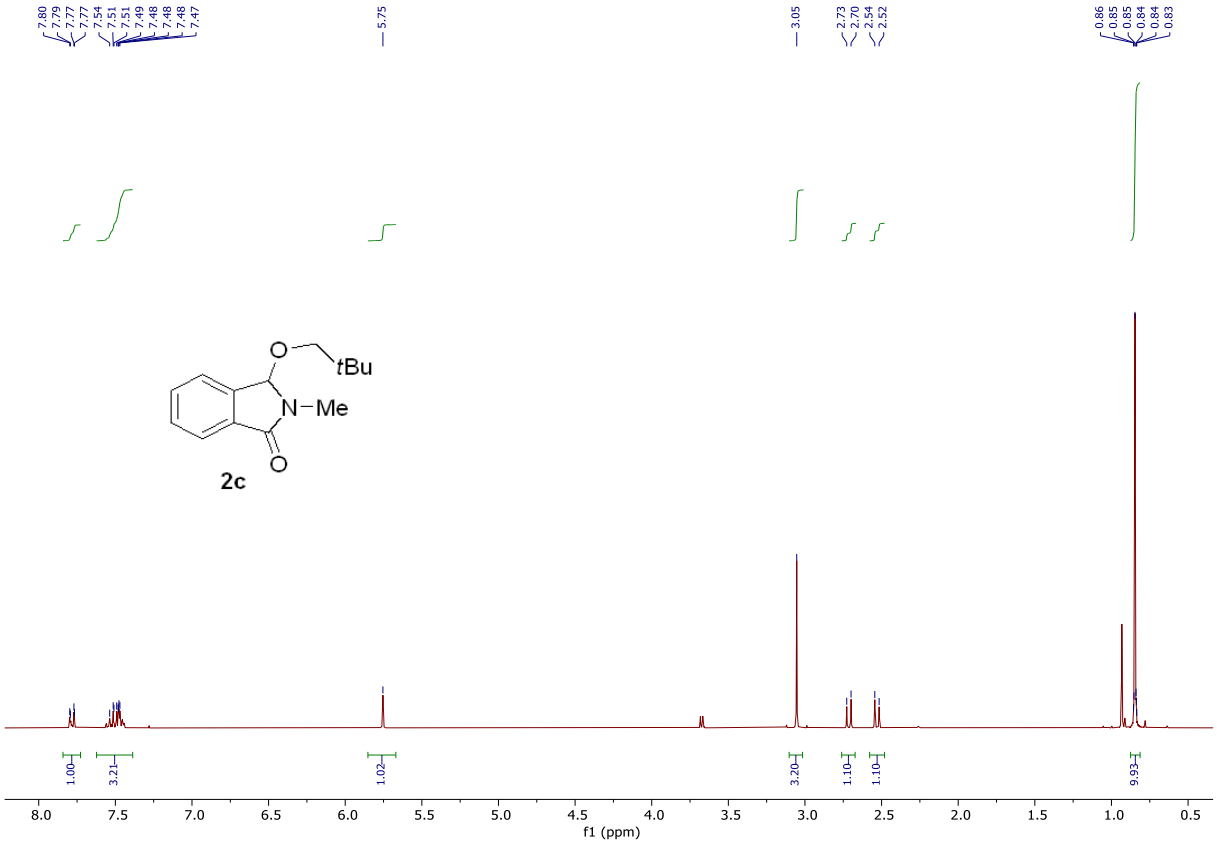
140.93
132.95
131.80
129.73
123.22
123.20
87.69
77.48
77.12
76.77
62.06
31.70
31.67
29.53
28.97
28.78
26.46
26.07
25.39
22.53
14.03
OC7H15
N−Me
O
2b
f1 (ppm)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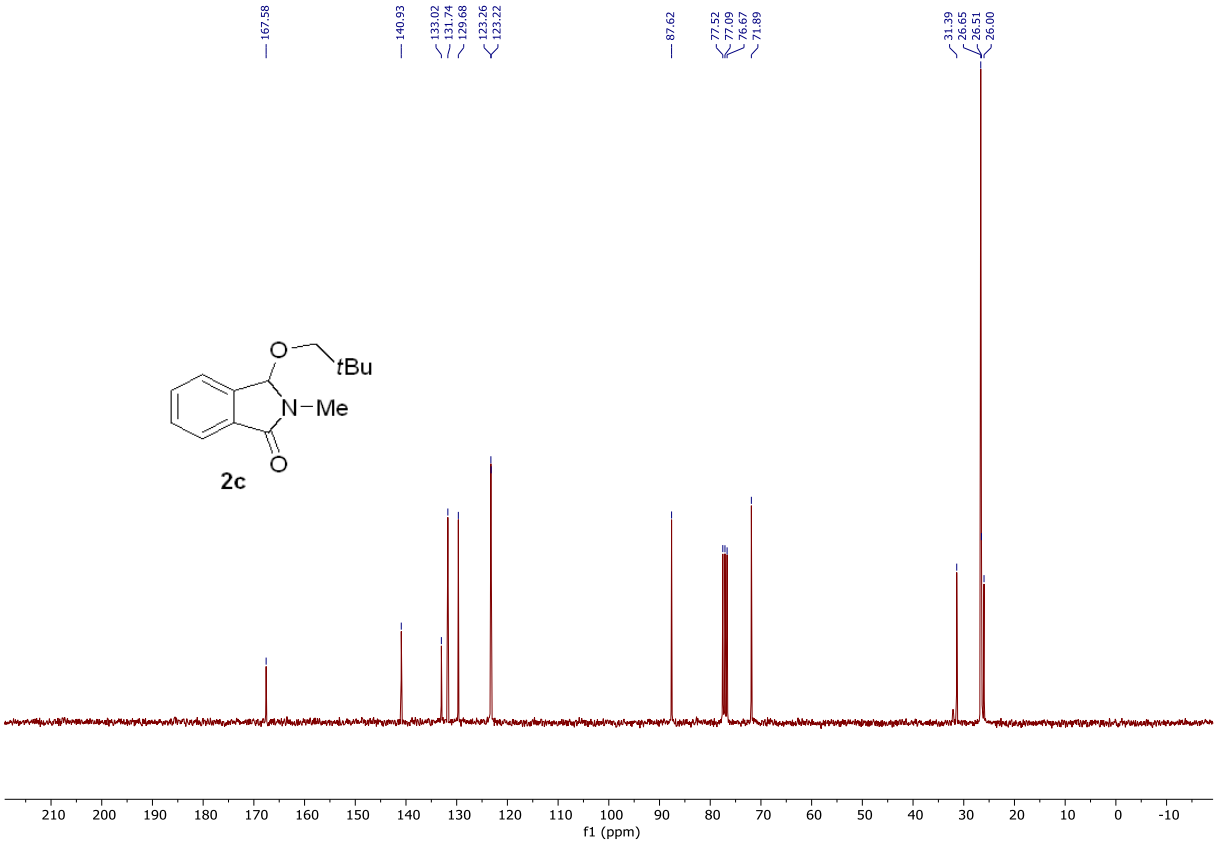
7.79
7.77
7.77
7.54
7.51
7.51
7.49
7.48
7.48
7.48
7.47
5.75
3.05
2.73
2.70
2.54
2.52
0.86
0.85
0.85
0.84
0.84
0.83
tBu
N−Me
O
O
2c
1.00
3.21
1.02
3.20
1.10
1.10
9.93
f1 (ppm)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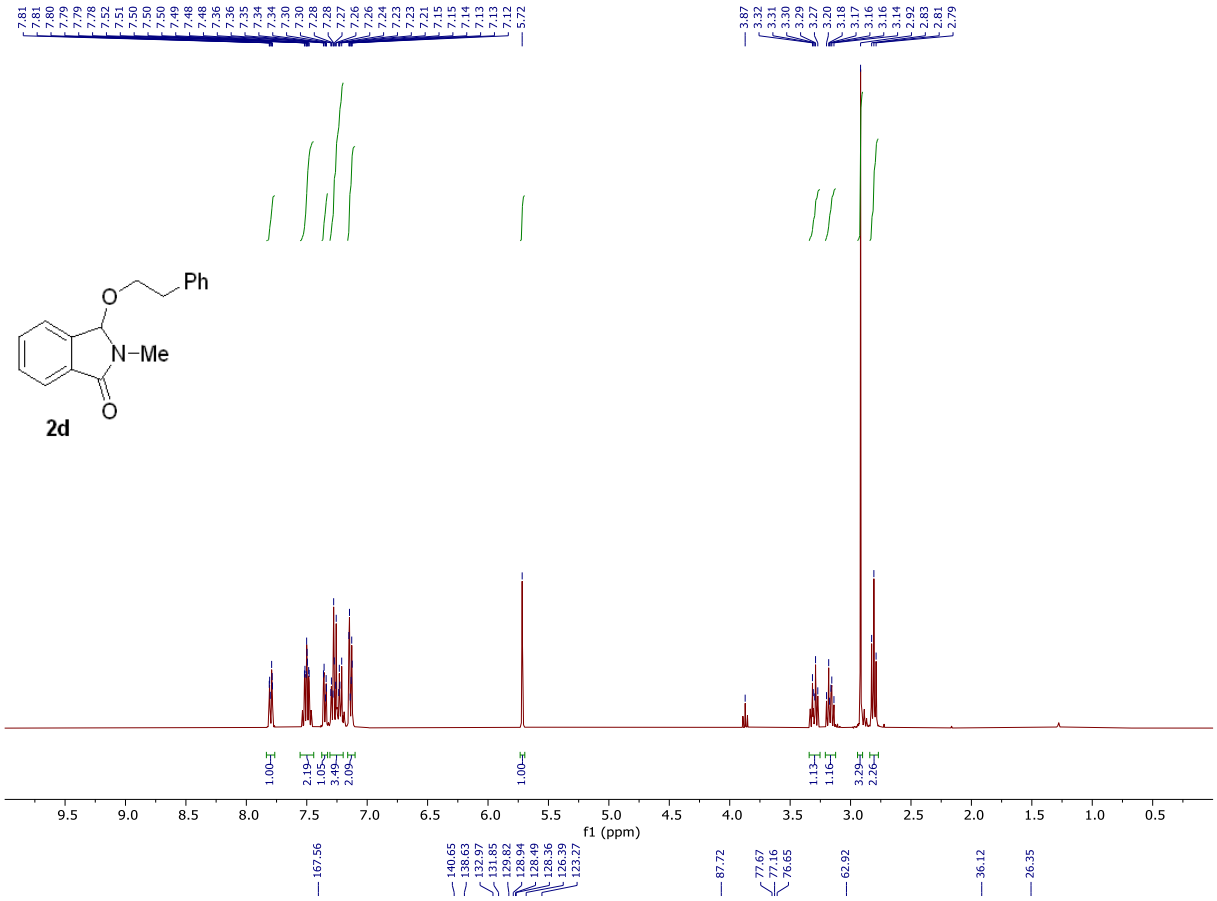
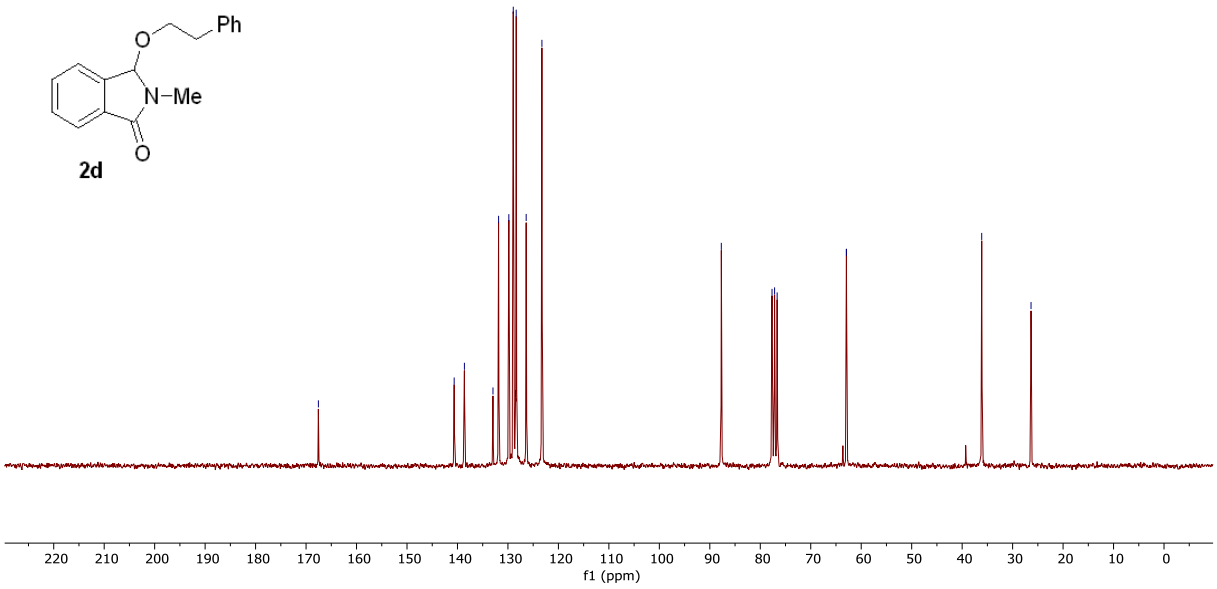
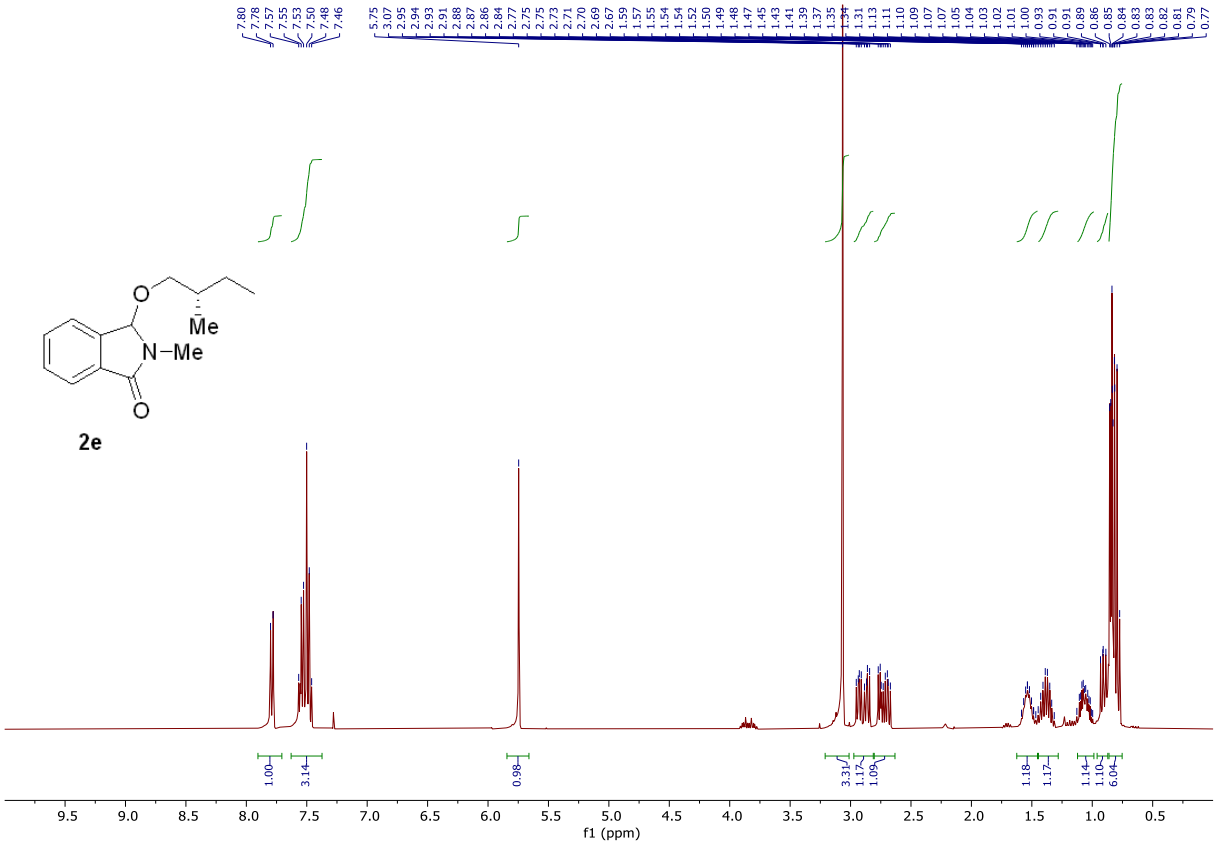
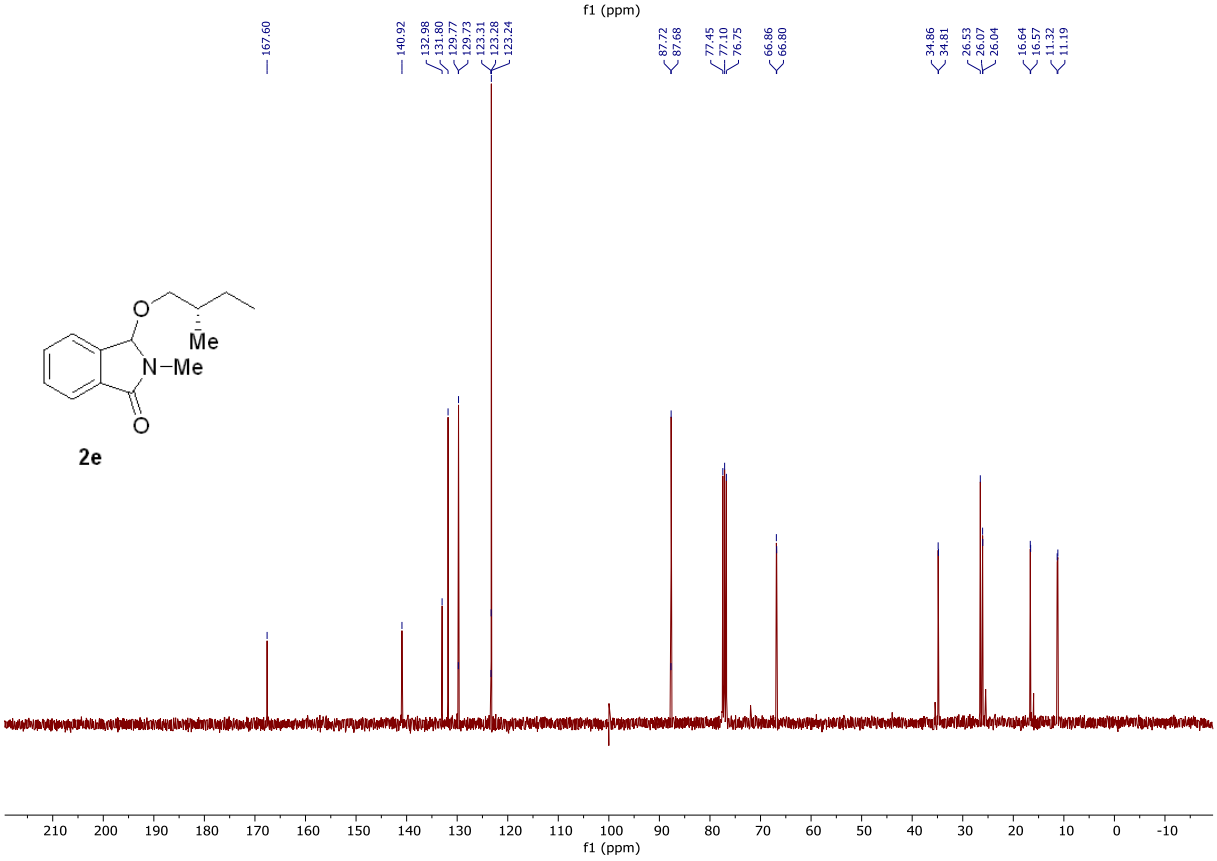
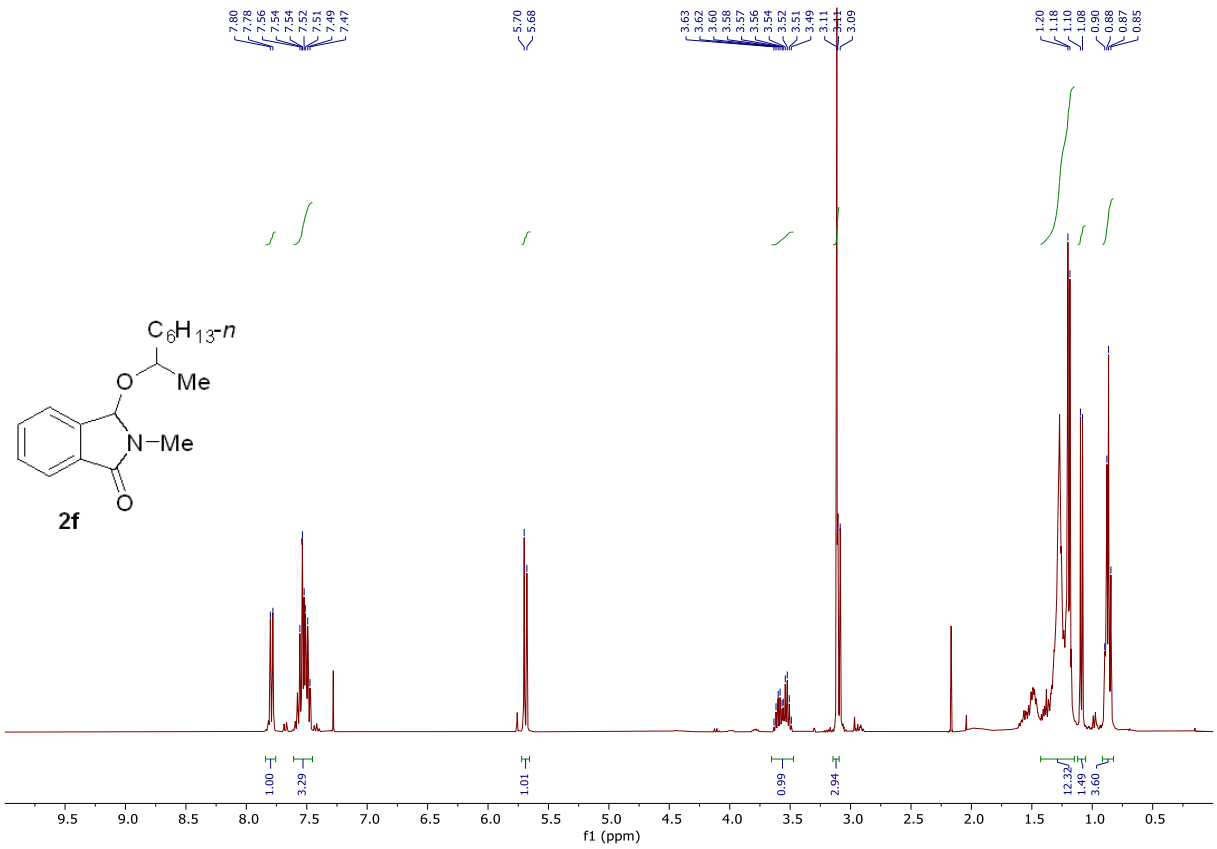
140.93
133.02
131.74
129.68
123.26
123.22
87.62
77.52
77.09
76.67
71.89
31.39
26.65
26.51
26.00
tBu
N−Me
O
O
2c
f1 (ppm)


7.81 7.81 7.80 7.79 7.78 7.52 7.51 7.50 7.50 7.50 7.49 7.48 7.48 7.36 7.36 7.35 7.34 7.34 7.30 7.30 7.28 7.28 7.27 7.26 7.26 7.24 7.23 7.23 7.21 7.15 7.15 7.14 7.13 7.13 7.12 5.72
3.87 3.32 3.31 3.30 3.29 3.27 3.20 3.18 3.17 3.16 3.16 3.14 2.92 2.83 2.81 2.79
Ph
O
N–Me
O
2d
1.00 2.19 1.05 3.49 2.09 1.00 1.13 1.16 3.29 2.26
f1 (ppm)
167.56
140.65 138.63 132.97 131.85 129.82 128.94 128.49 128.36 126.39 123.27
87.72
77.67 77.16 76.65
62.92
36.12
26.35


Ph
O
N–Me
O
2d
f1 (ppm)


7.80 7.78 7.57 7.55 7.53 7.50 7.48 7.46
5.75 3.07 2.95 2.94 2.93 2.91 2.88 2.87 2.86 2.84 2.77 2.75 2.73 2.71 2.70 2.69 2.67 2.59 1.57 1.55 1.54 1.54 1.52 1.50 1.49 1.48 1.47 1.45 1.43 1.41 1.39 1.37 1.35 1.34 1.31 1.13 1.11 1.10 1.09 1.07 1.07 1.05 1.04 1.03 1.02 1.01 1.00 0.93 0.91 0.91 0.89 0.86 0.85 0.84 0.83 0.83 0.82 0.81 0.79 0.77
O
Me
N–Me
O
2e
1.00 3.14 0.98 3.31 1.17 1.09 1.18 1.17 1.14 1.10 6.04
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)


167.60 140.92 132.98 131.80 129.77 129.73 123.31 123.28 123.24 87.72 87.68 77.45 77.10 76.75 66.86 66.80 34.86 34.81 26.53 26.07 26.04 16.64 16.57 11.32 11.19
O
Me
N–Me
O
2e
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)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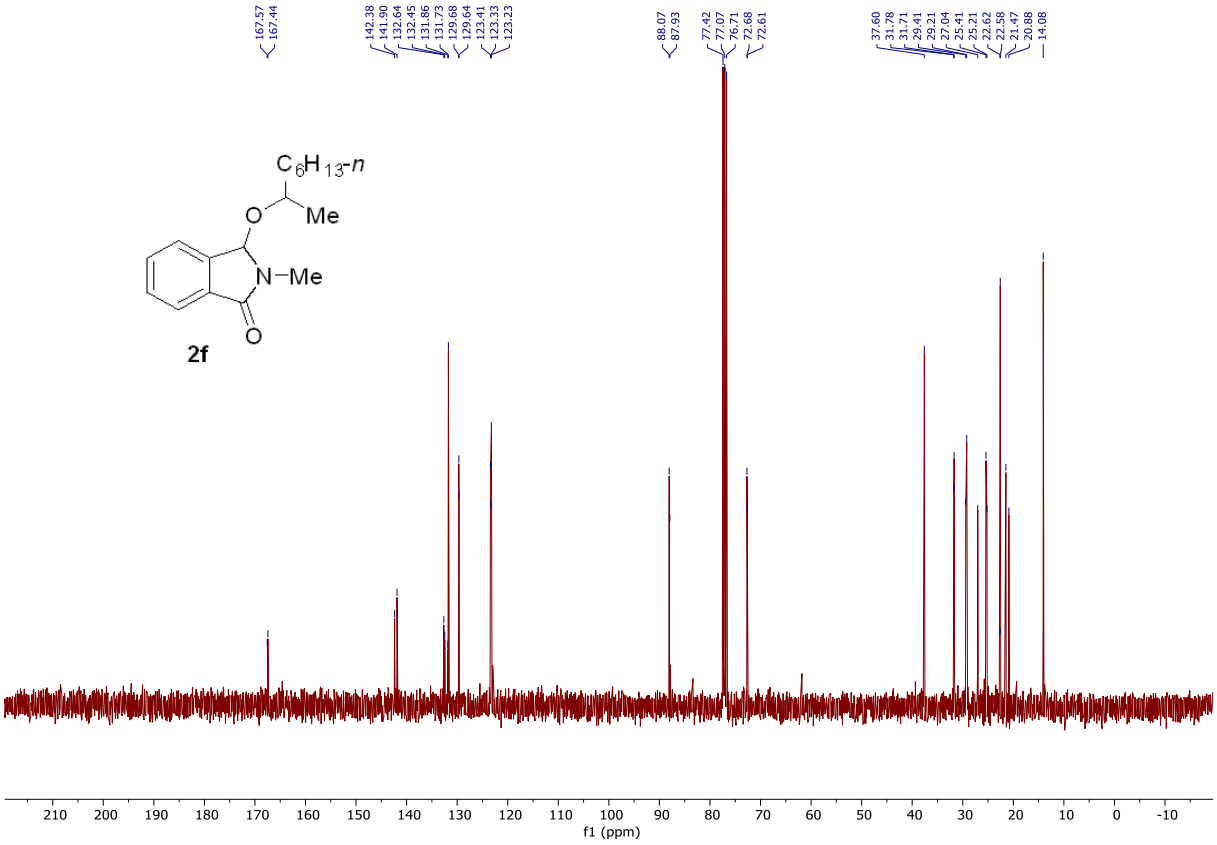
7.78
7.56
7.54
7.54
7.52
7.51
7.49
7.47
5.70
5.68
3.63
3.62
3.60
3.58
3.57
3.56
3.54
3.52
3.51
3.49
3.11
3.11
3.09
1.20
1.18
1.10
1.08
0.90
0.88
0.87
0.85
C6H13-n
O
Me
N−Me
O
2f
1.00
3.29
1.01
0.99
2.94
12.32
1.49
3.60
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)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142.38
141.90
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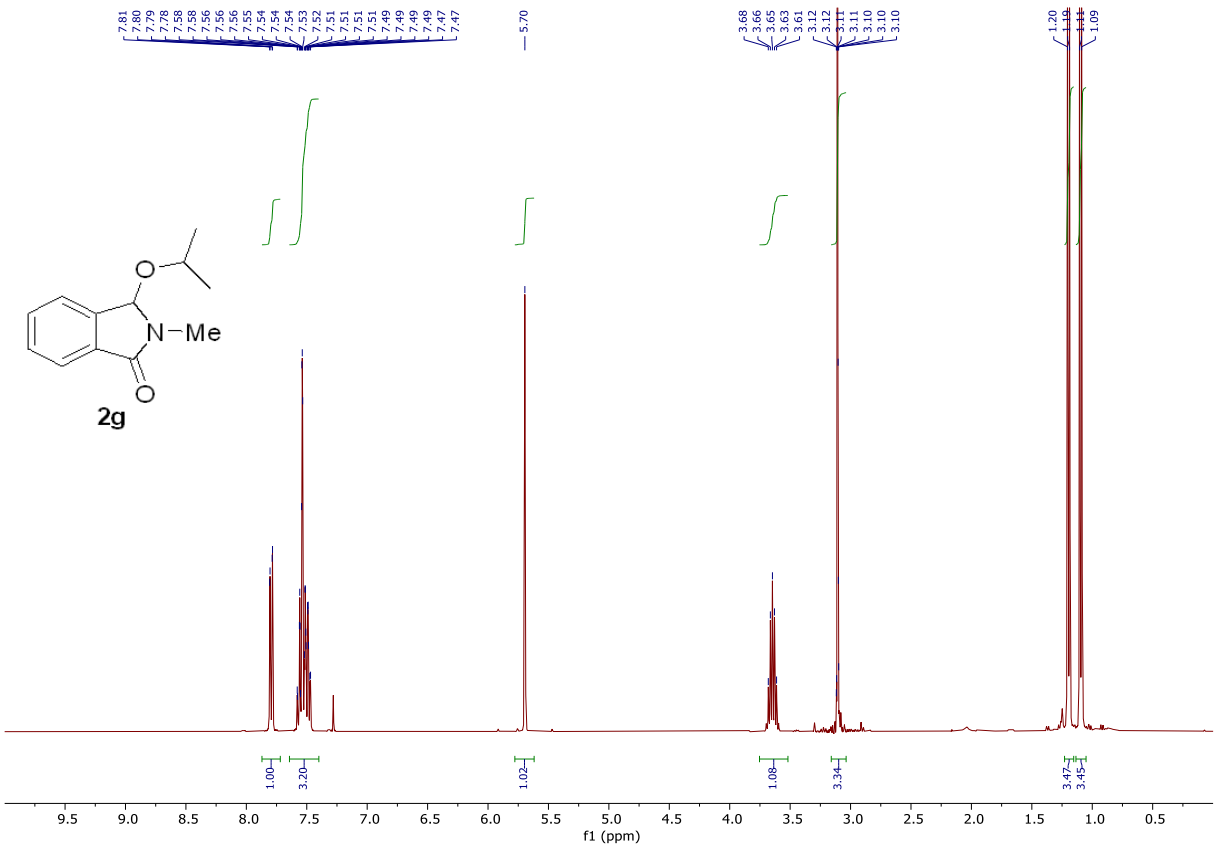
132.45
131.86
131.73
129.68
129.64
123.41
123.33
123.23
88.07
87.93
77.42
77.07
76.71
72.68
72.61
37.60
31.78
31.71
29.41
29.21
27.04
25.41
25.21
22.62
22.58
21.47
20.88
14.08
C6H13-n
O
Me
N−Me
O
2f
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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7.56
7.56
7.56
7.55
7.54
7.54
7.54
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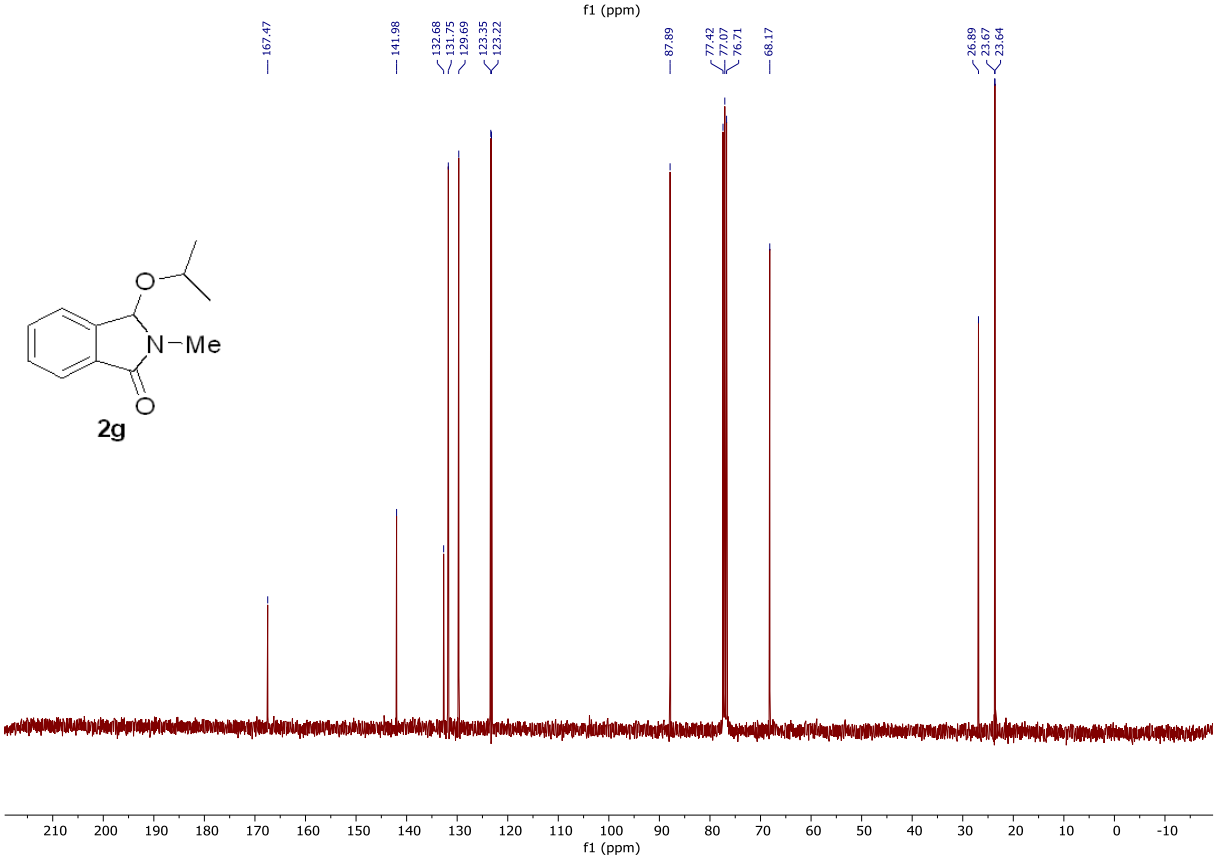
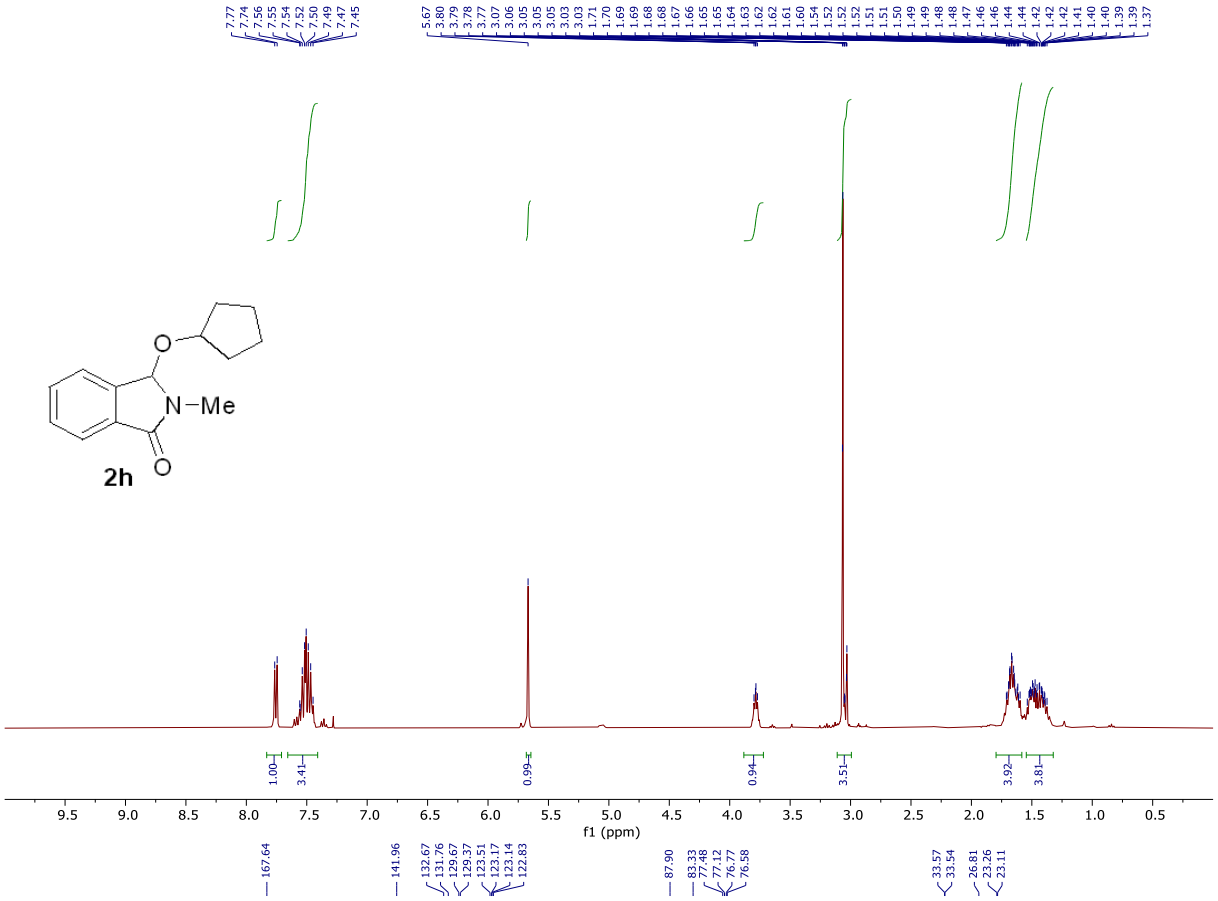
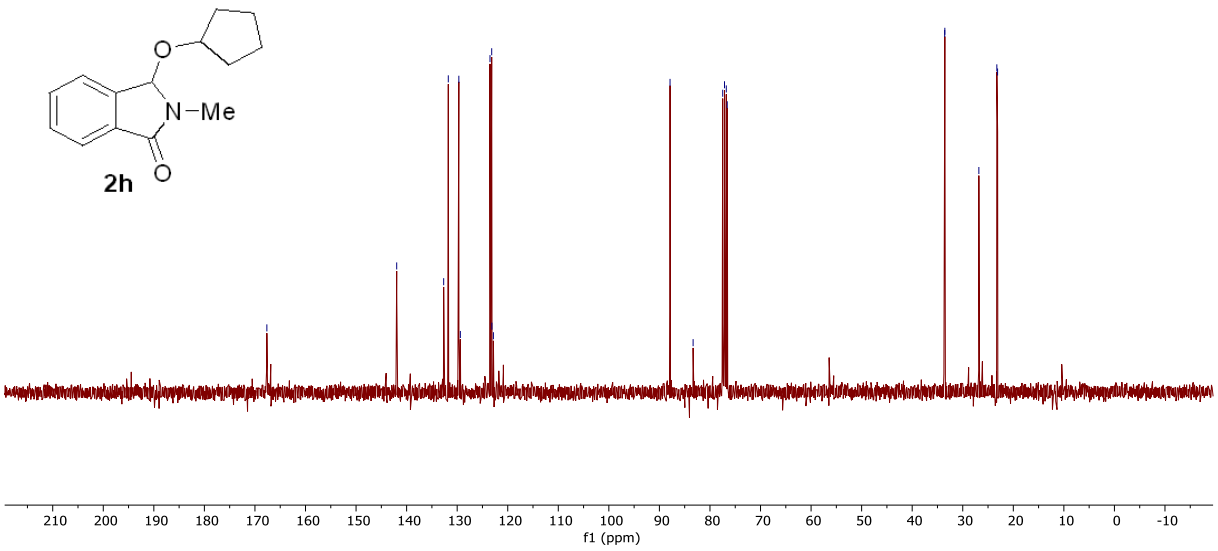
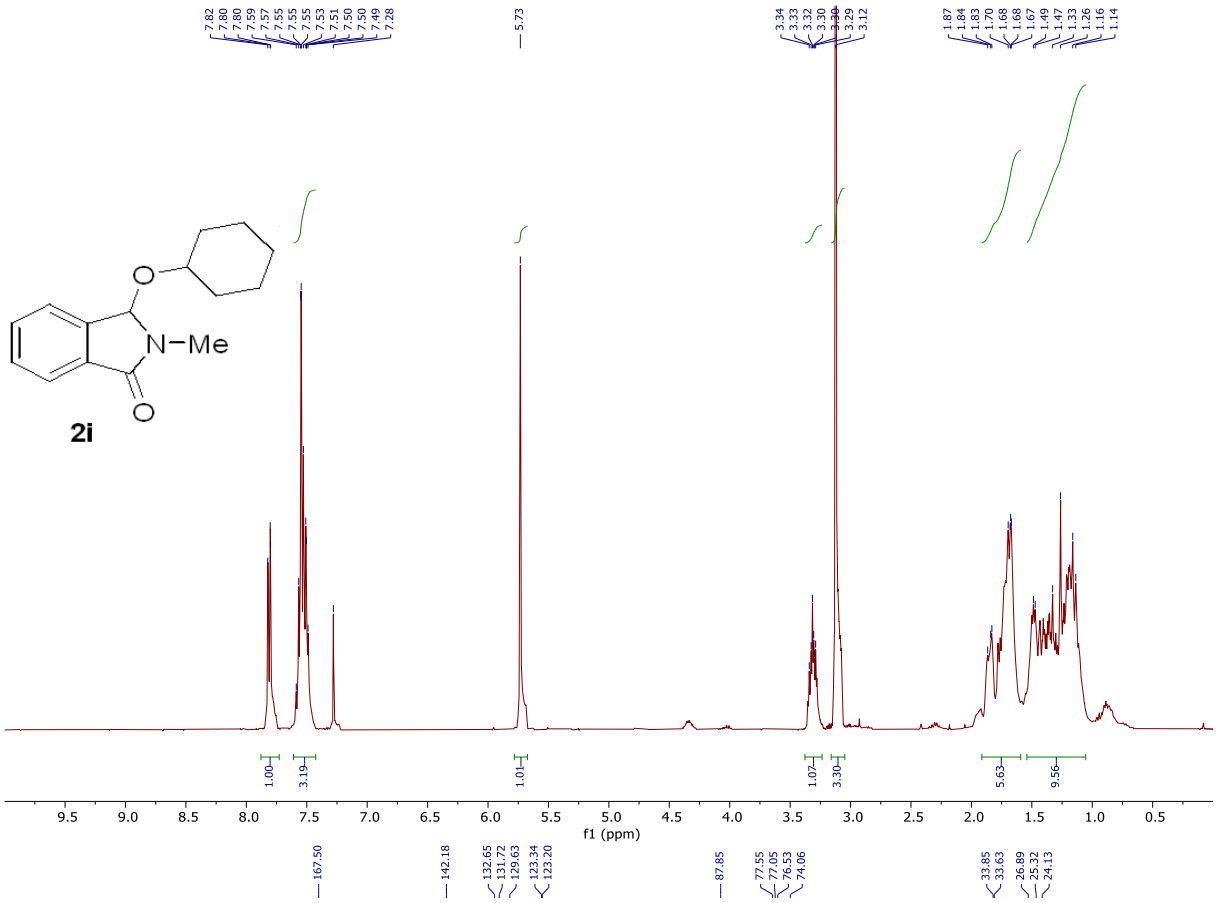
7.52
7.51
7.51
7.51
7.51
7.49
7.49
7.49
7.49
7.47
7.47
5.70
3.68
3.66
3.65
3.63
3.61
3.12
3.12
3.11
3.11
3.10
3.10
3.10
1.20
1.19
1.11
1.09
1.00
3.20
1.02
1.08
3.34
3.47
3.45
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)
O
N−Me
O
2g


167.47
141.98
132.68
131.75
129.69
123.35
123.22
87.89
77.42
77.07
76.71
68.17
26.89
23.67
23.64
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−Me
O
2g


7.77 7.74 7.56 7.55 7.54 7.52 7.50 7.49 7.47 7.45
5.67 3.80 3.79 3.78 3.77 3.07 3.06 3.05 3.05 3.05 3.03 3.03 1.71 1.70 1.69 1.69 1.68 1.68 1.67 1.66 1.65 1.65 1.64 1.63 1.62 1.62 1.61 1.60 1.54 1.52 1.52 1.52 1.51 1.51 1.50 1.49 1.49 1.48 1.48 1.47 1.46 1.46 1.44 1.44 1.42 1.42 1.42 1.41 1.40 1.40 1.39 1.39 1.37
N−Me
2h
1.00 3.41 0.99 0.94 3.51 3.92 3.81
f1 (ppm)


167.64 141.96 132.67 131.76 129.67 129.37 123.51 123.17 123.14 122.83 87.90 83.33 77.48 77.12 76.77 76.58 33.57 33.54 26.81 23.26 23.11
N−Me
2h
f1 (ppm)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7.55
7.55
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7.51
7.50
7.50
7.49
7.28
5.73
3.34
3.33
3.32
3.30
3.29
3.12
1.87
1.84
1.83
1.70
1.68
1.68
1.67
1.49
1.47
1.33
1.26
1.16
1.14
N−Me
O
O
2i
1.00
3.19
1.01
1.07
3.30
5.63
9.56
f1 (ppm)
167.50
142.18
132.65
131.72
129.63
123.34
123.20
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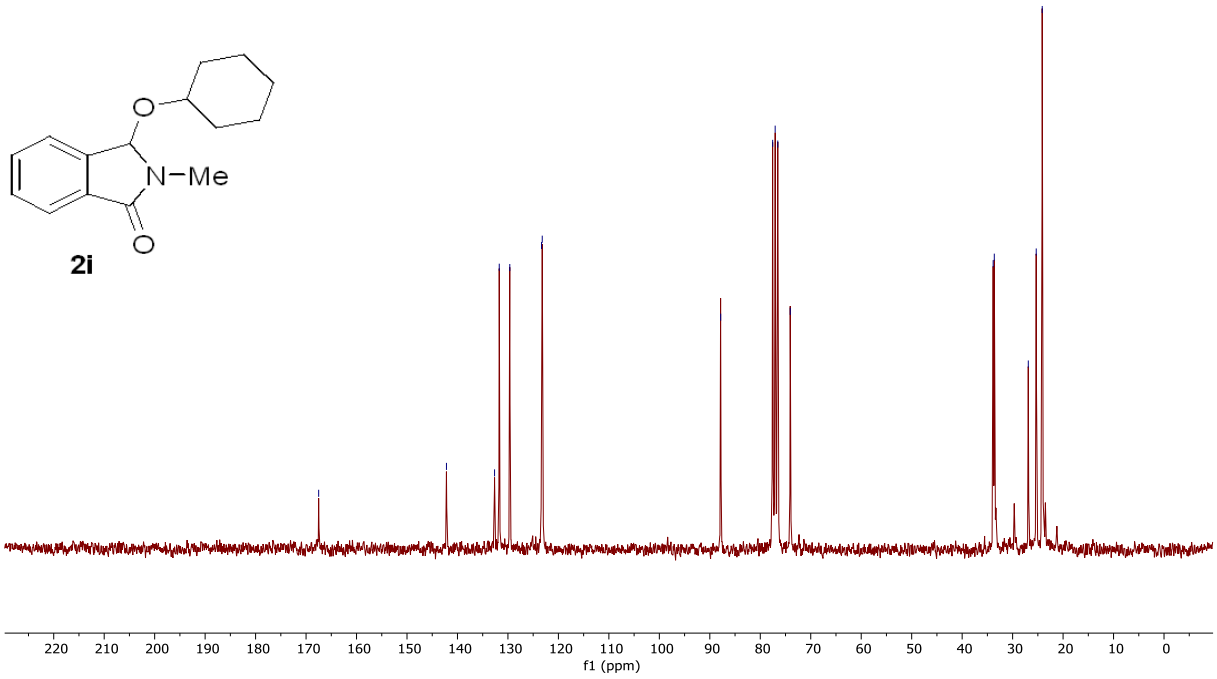
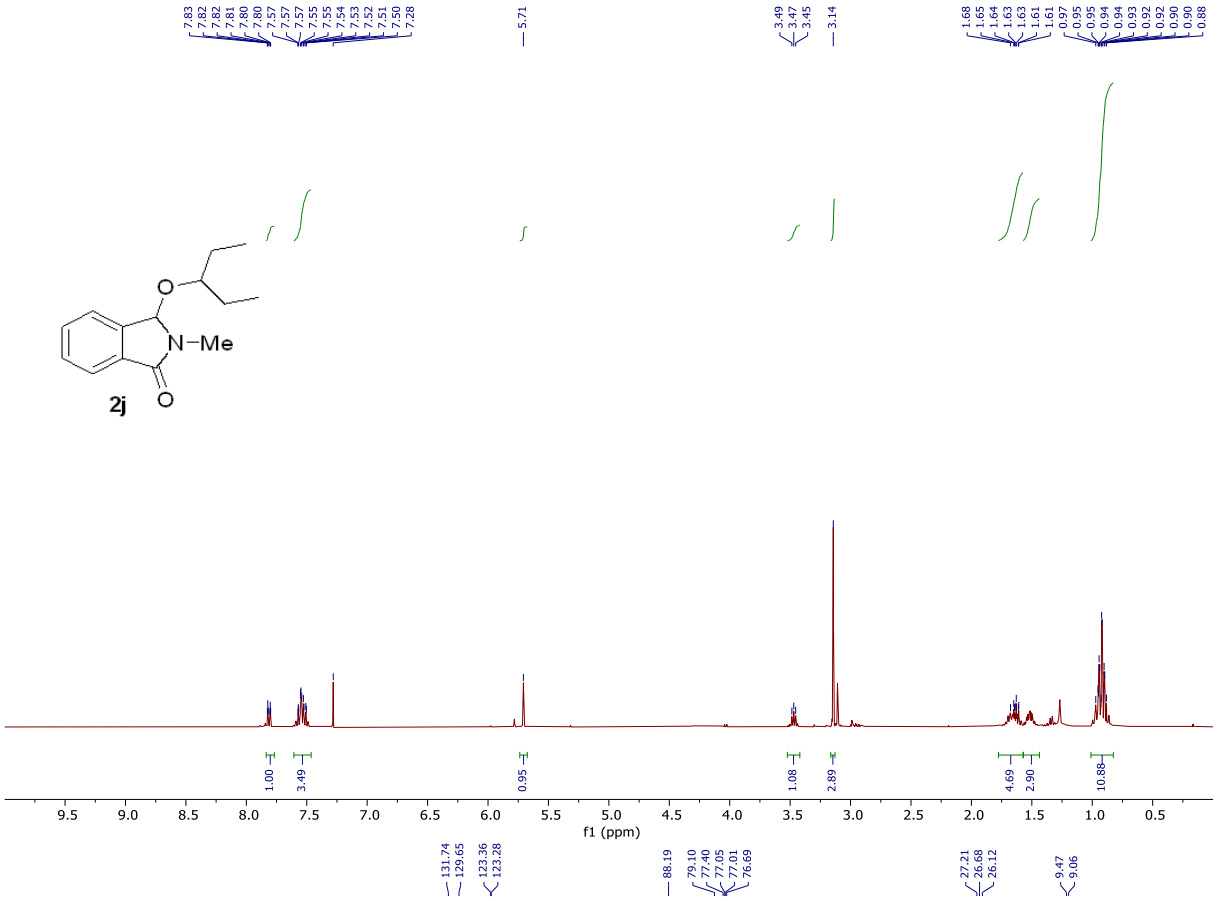
77.55
77.05
76.53
74.06
33.85
33.63
26.89
25.32
24.13


N−Me
O
O
2i
f1 (ppm)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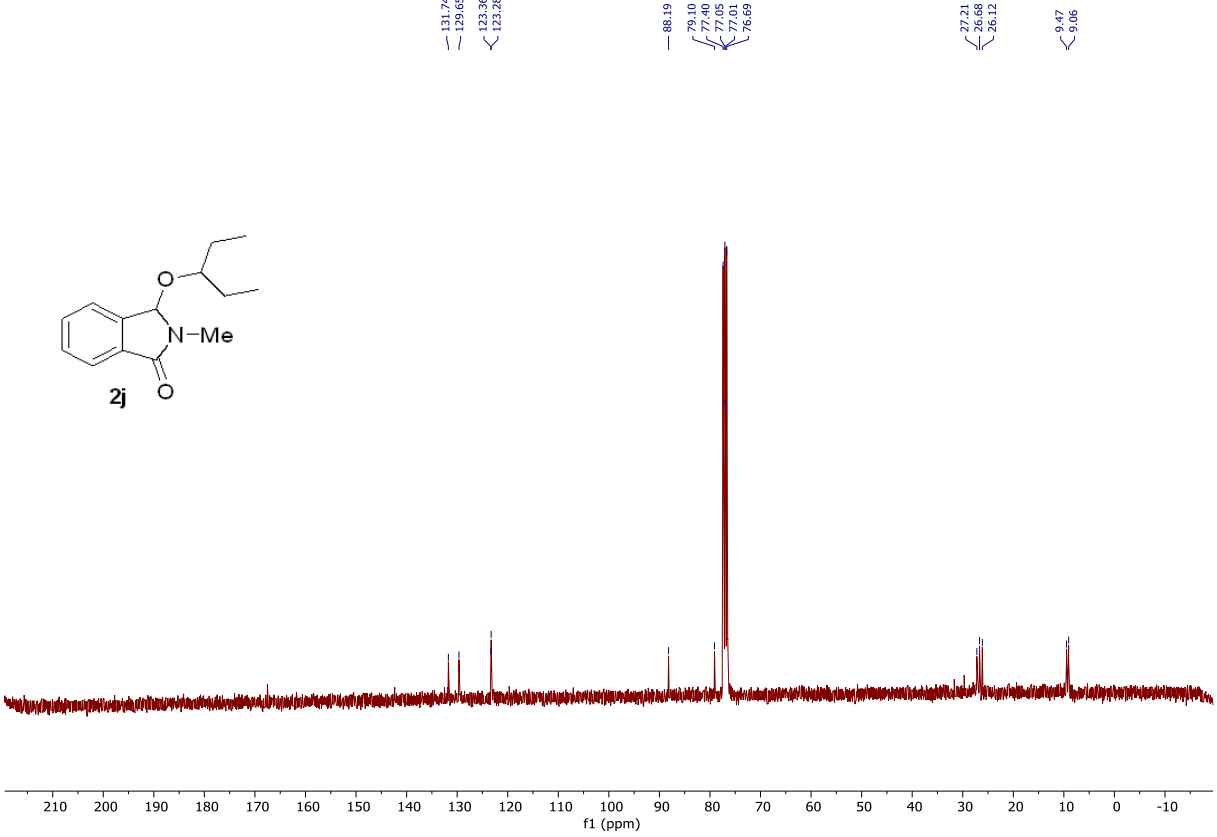
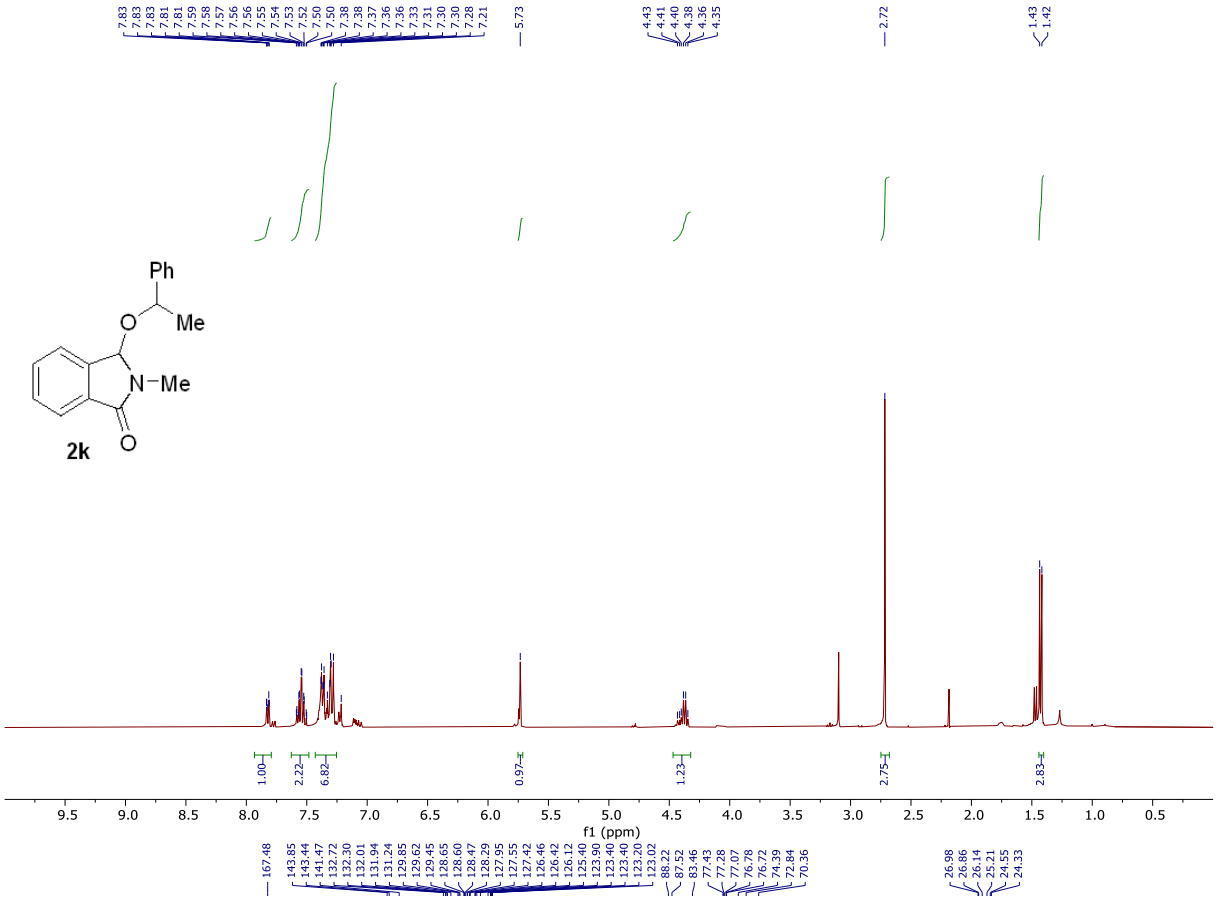
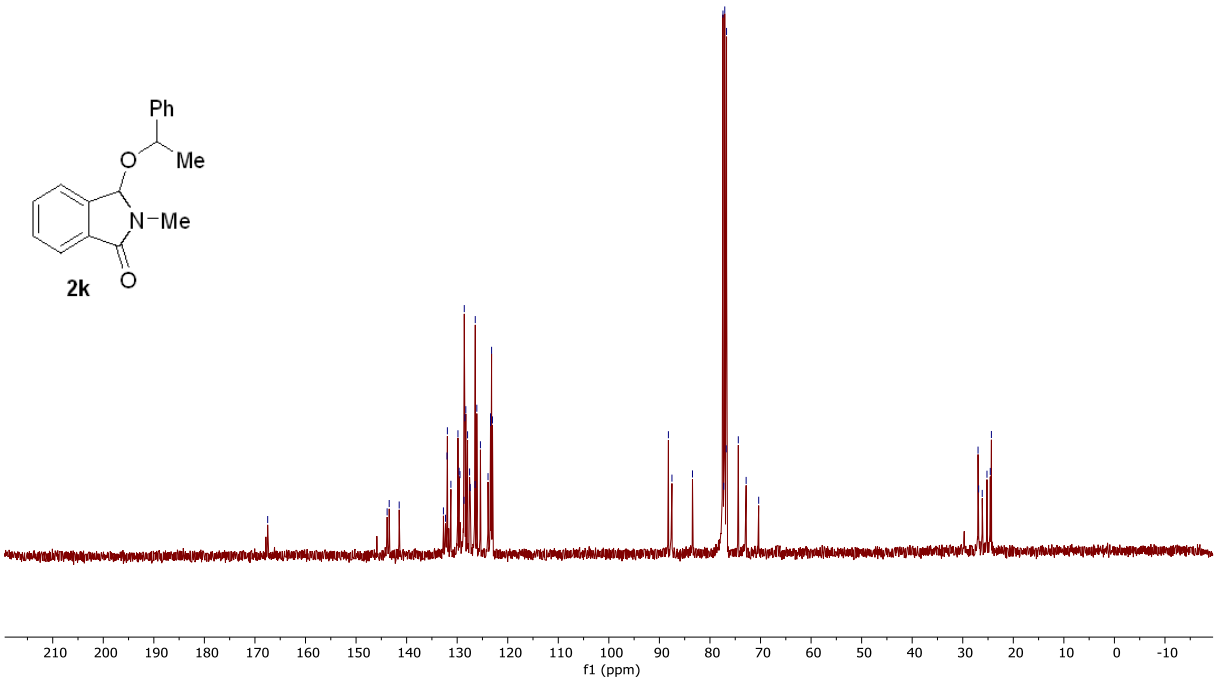
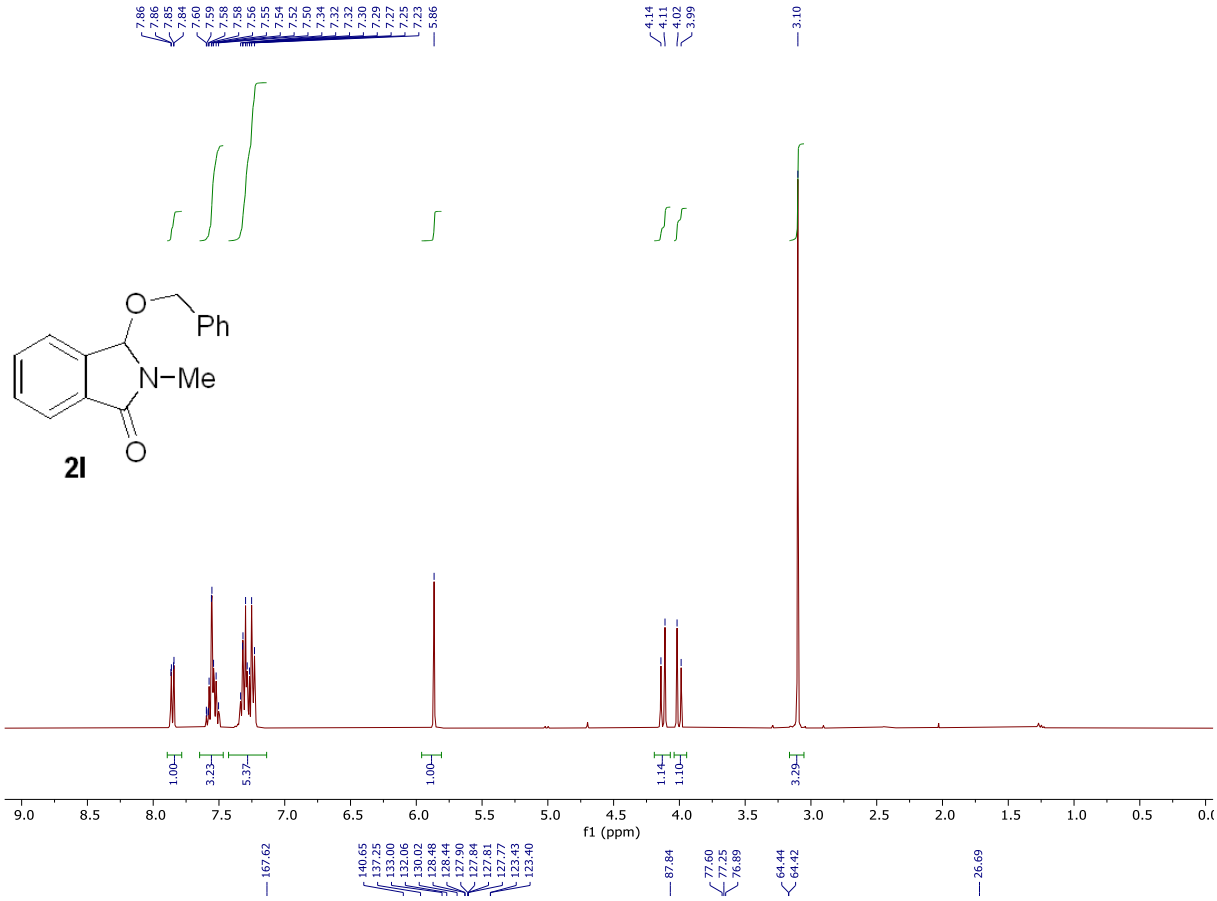
7.55
7.55
7.54
7.53
7.52
7.51
7.50
7.28
5.71
3.49
3.47
3.45
3.14
1.68
1.65
1.64
1.63
1.63
1.61
1.61
0.97
0.95
0.95
0.94
0.94
0.93
0.92
0.92
0.90
0.90
0.88
O
N–Me
O
2j
1.00
3.49
0.95
1.08
2.89
4.69
2.90
10.88
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)


131.74
129.65
123.36
123.28
88.19
79.10
77.40
77.05
77.01
76.69
27.21
26.68
26.12
9.47
9.06
O
N–Me
O
2j
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)


Ph
O
Me
N−Me
O
2k
f1 (ppm)


Ph
O
Me
N−Me
O
2k
f1 (ppm)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7.85
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7.58
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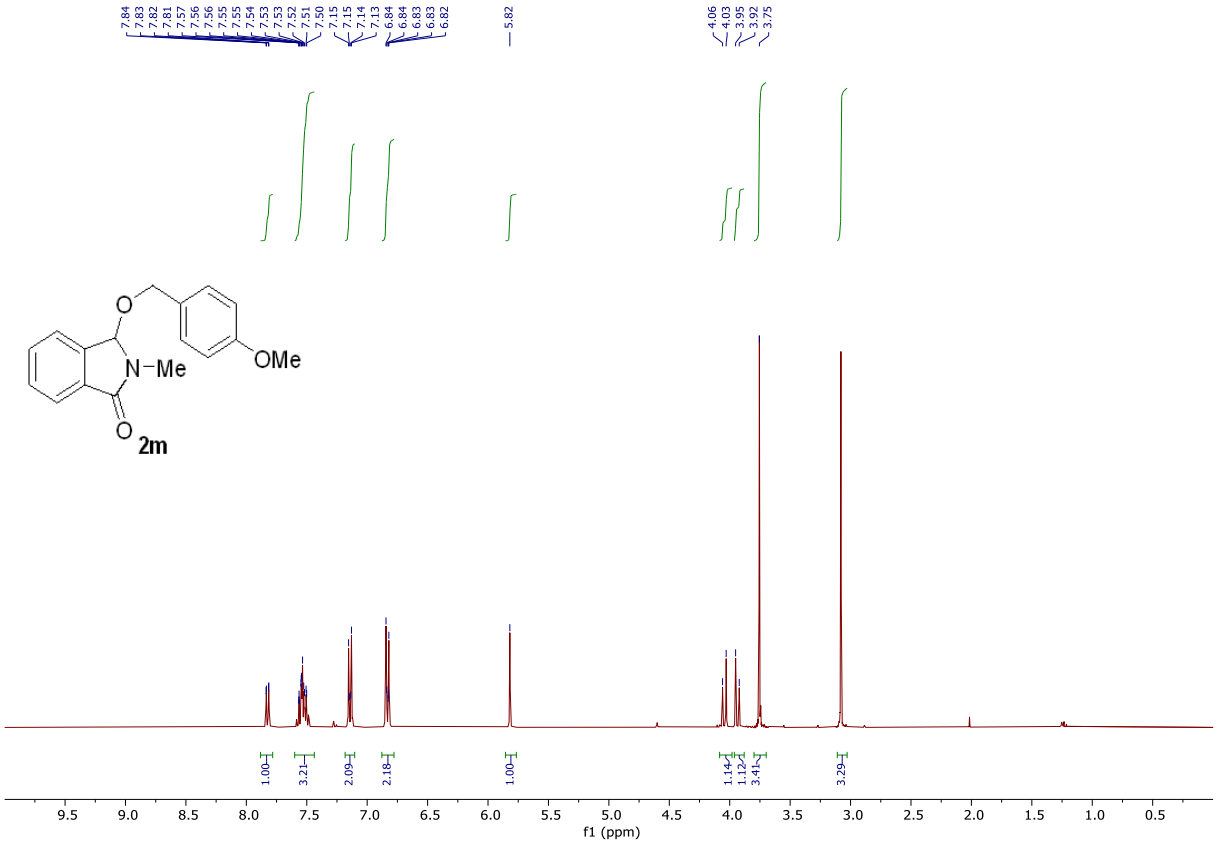
7.55
7.54
7.52
7.50
7.34
7.32
7.30
7.29
7.27
7.25
7.23
5.86
4.14
4.11
4.02
3.99
3.10
Ph
N−Me
O
O
2l
1.00
3.23
5.37
1.00
1.14
1.10
3.29
f1 (ppm)
167.62
140.65
137.25
133.00
132.06
130.02
128.48
128.44
127.90
127.84
127.81
127.77
123.43
123.40
87.84
77.60
77.25
76.89
64.44
64.42
26.69
f1 (ppm)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7.56
7.55
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7.54
7.53
7.53
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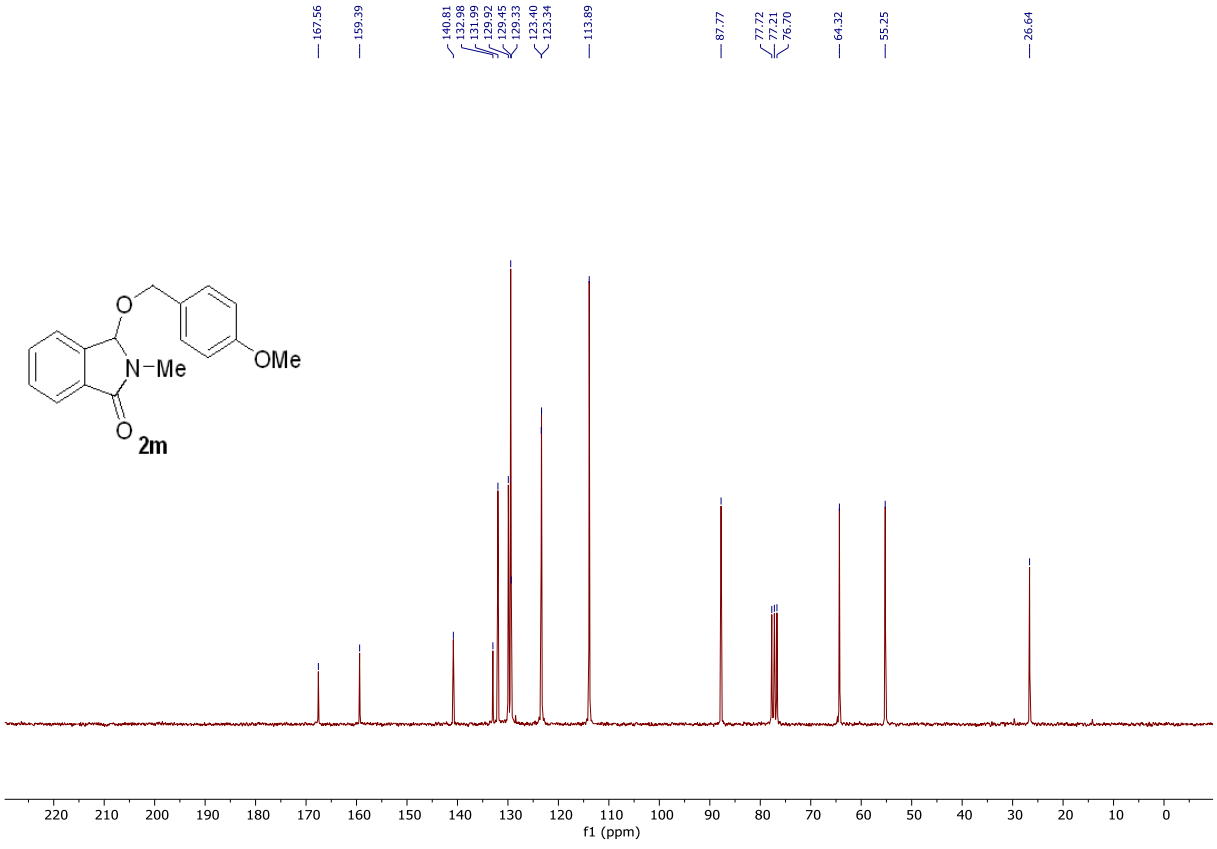
7.51
7.50
7.15
7.15
7.14
7.13
6.84
6.84
6.83
6.83
6.82
5.82
4.06
4.03
3.95
3.92
3.75
O
N−Me
OMe
O
2m
1.00
3.21
2.09
2.18
1.00
1.14
1.12
3.41
3.29
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)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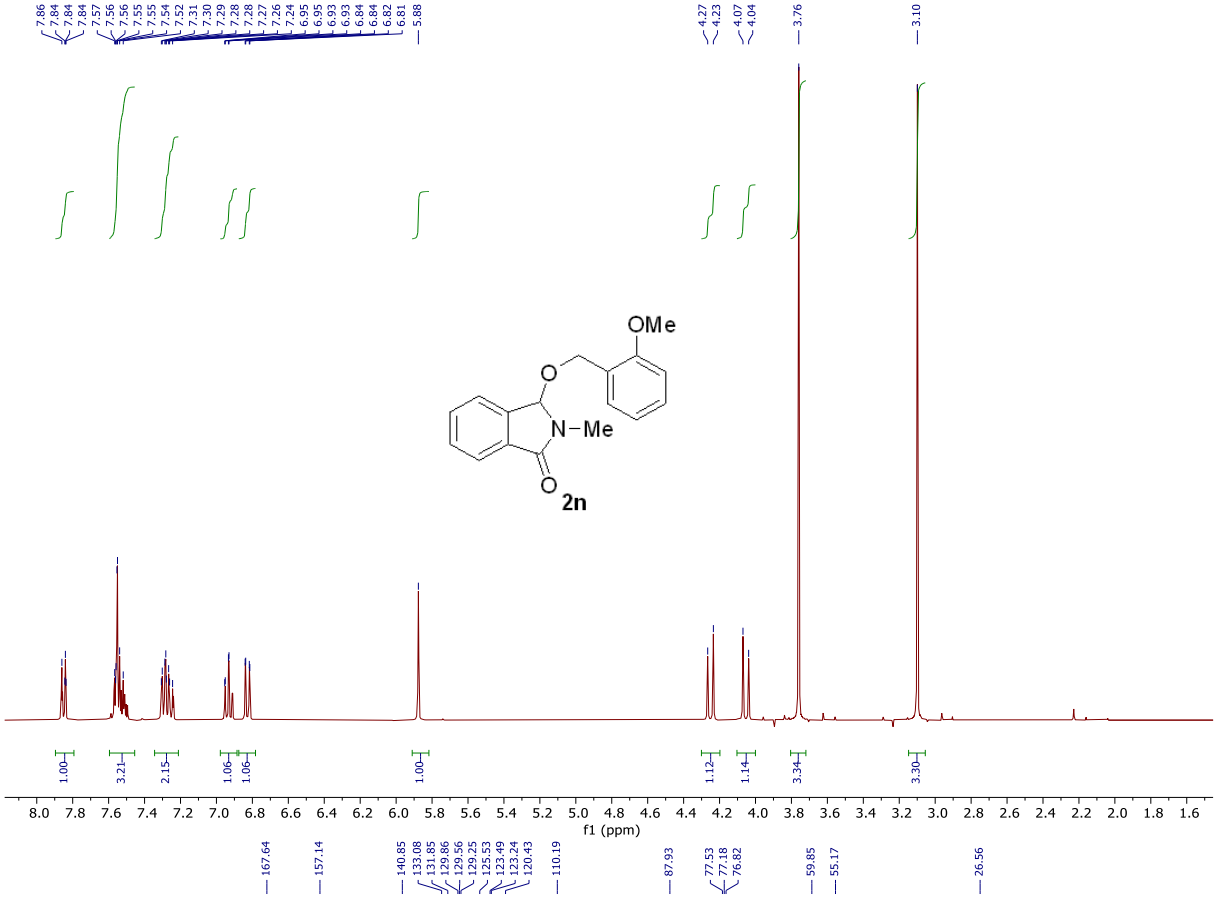
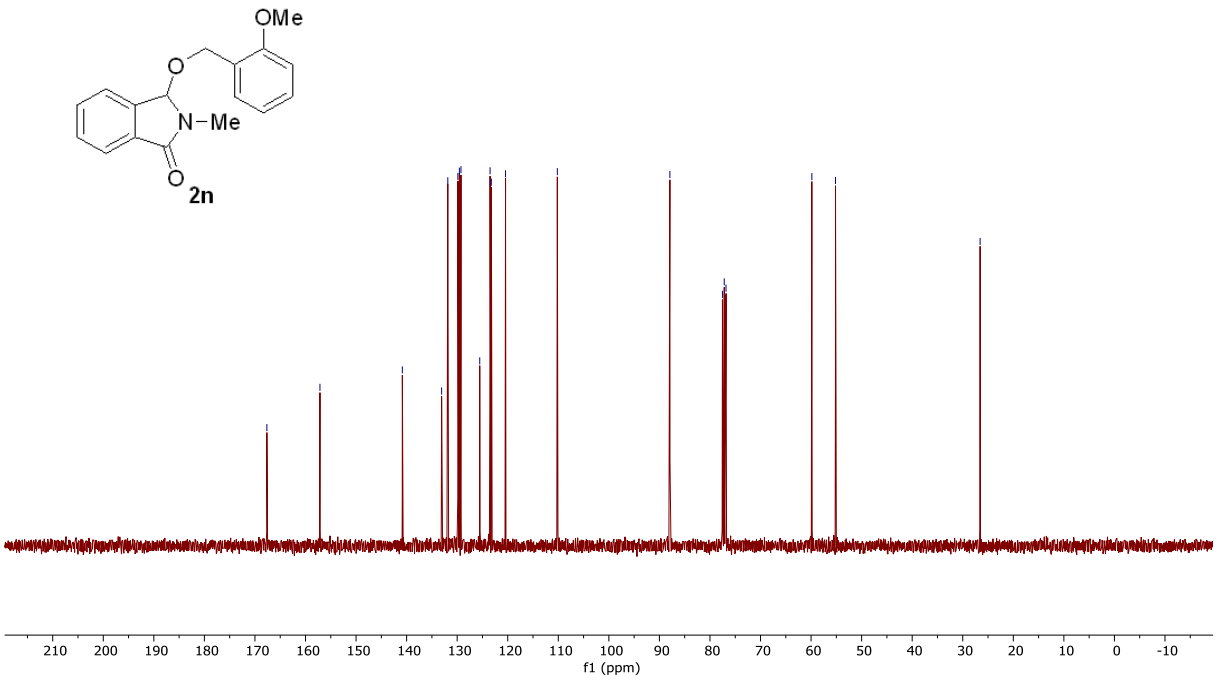
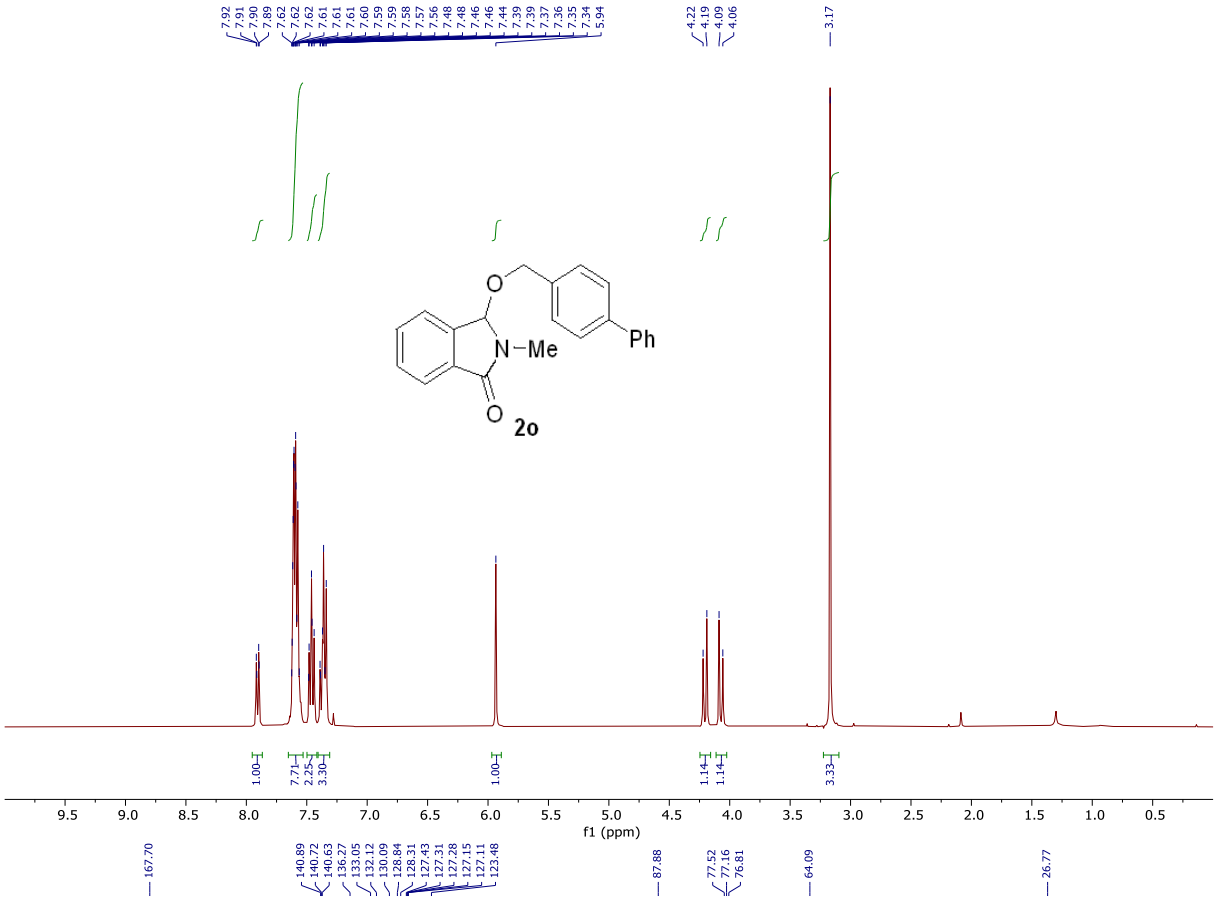
159.39
140.81
132.98
131.99
129.92
129.45
129.33
123.40
123.34
113.89
87.77
77.72
77.21
76.70
64.32
55.25
26.64
O
N−Me
OMe
O
2m
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


OMe
O
N−Me
O
2n
f1 (ppm)


OMe
O
N−Me
O
2n
f1 (ppm)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7.91
7.90
7.89
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7.62
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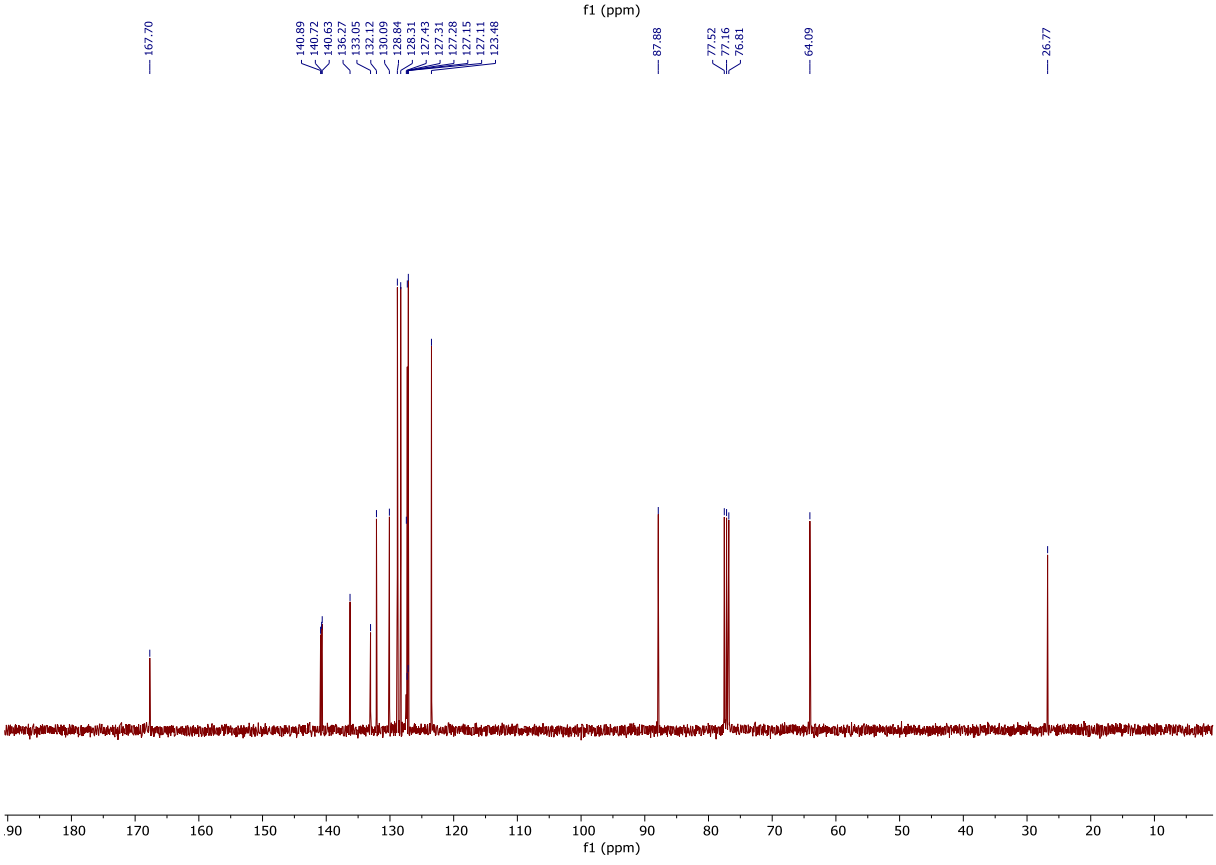
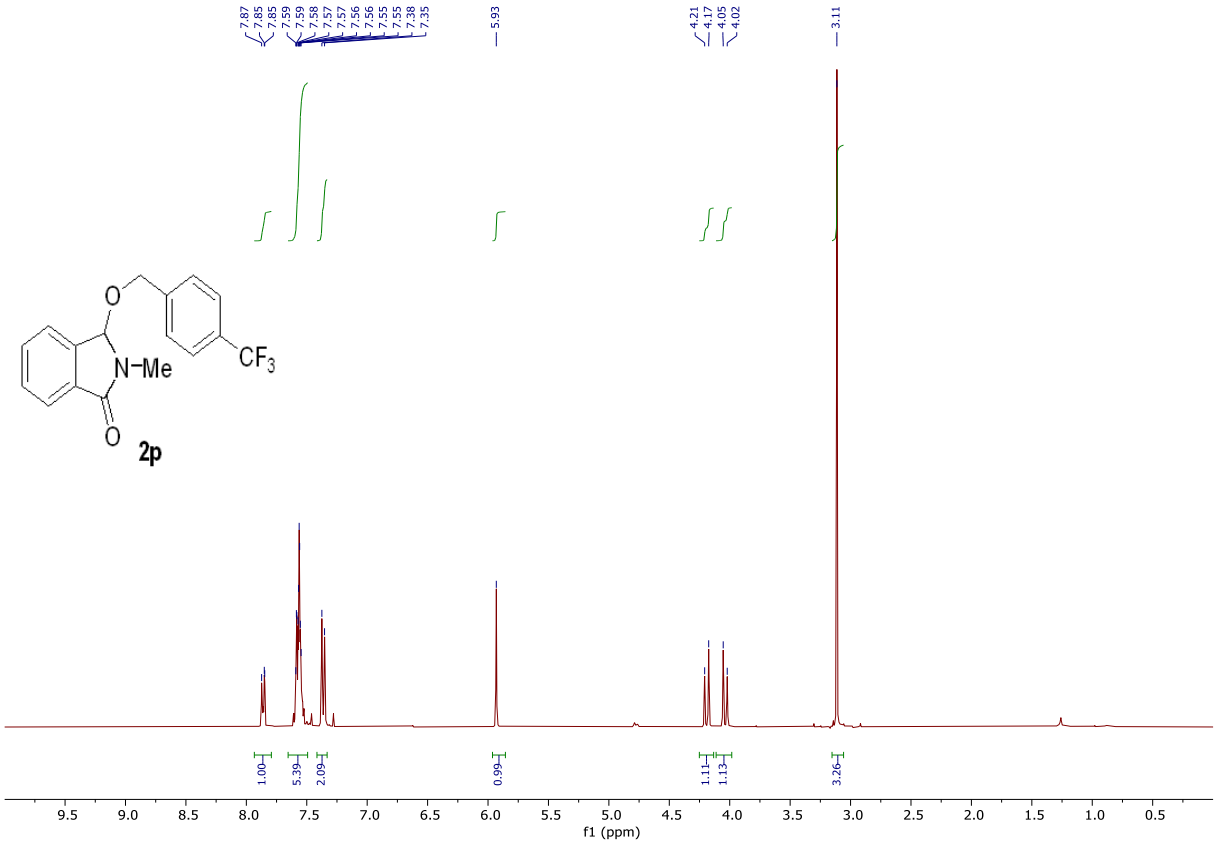
7.61
7.61
7.61
7.60
7.59
7.59
7.58
7.57
7.56
7.48
7.48
7.46
7.46
7.44
7.39
7.39
7.37
7.36
7.35
7.34
5.94
4.22
4.19
4.09
4.06
3.17
O
N−Me
Ph
O
2o
1.00
7.71
2.25
3.30
1.00
1.14
1.14
3.33
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)


167.70
140.89
140.72
140.63
136.27
133.05
132.12
130.09
128.84
128.31
127.43
127.31
127.28
127.15
127.11
123.48
87.88
77.52
77.16
76.81
64.09
26.77
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)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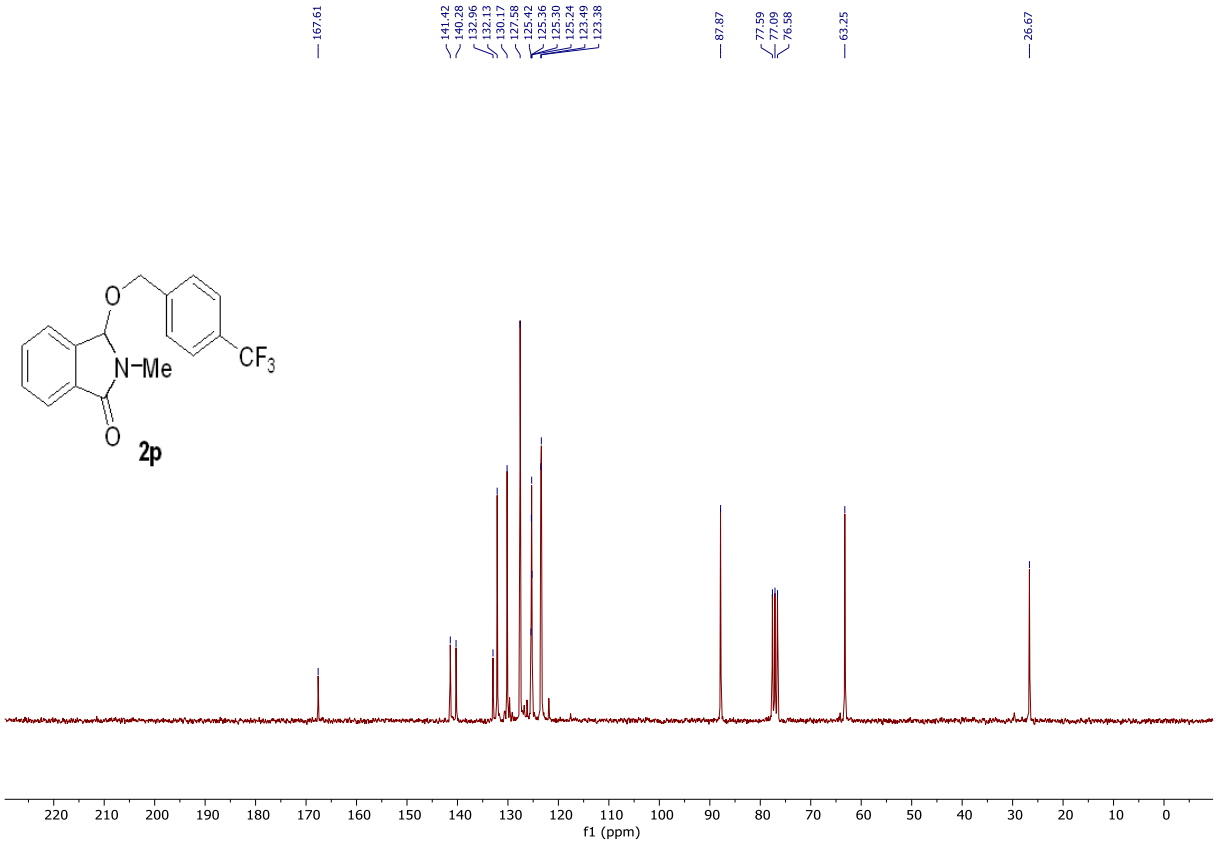
7.85
7.85
7.59
7.59
7.58
7.57
7.57
7.56
7.56
7.55
7.55
7.38
7.35
5.93
4.21
4.17
4.05
4.02
3.11
O
N-Me
CF3
O
2p
1.00
5.39
2.09
0.99
1.11
1.13
3.26
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)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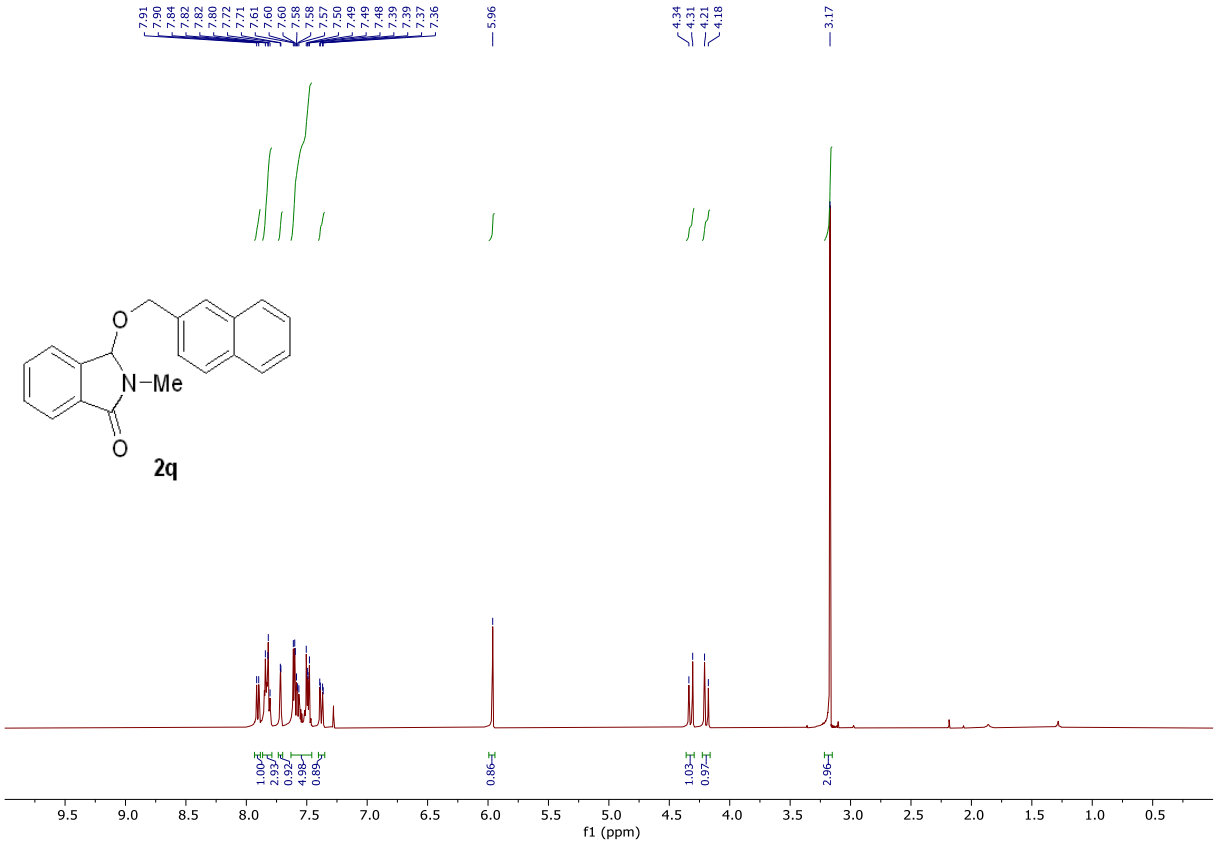
141.42
140.28
132.96
132.13
130.17
127.58
125.42
125.36
125.30
125.24
123.49
123.38
87.87
77.59
77.09
76.58
63.25
26.67
O
N-Me
CF3
O
2p
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.90
7.84
7.82
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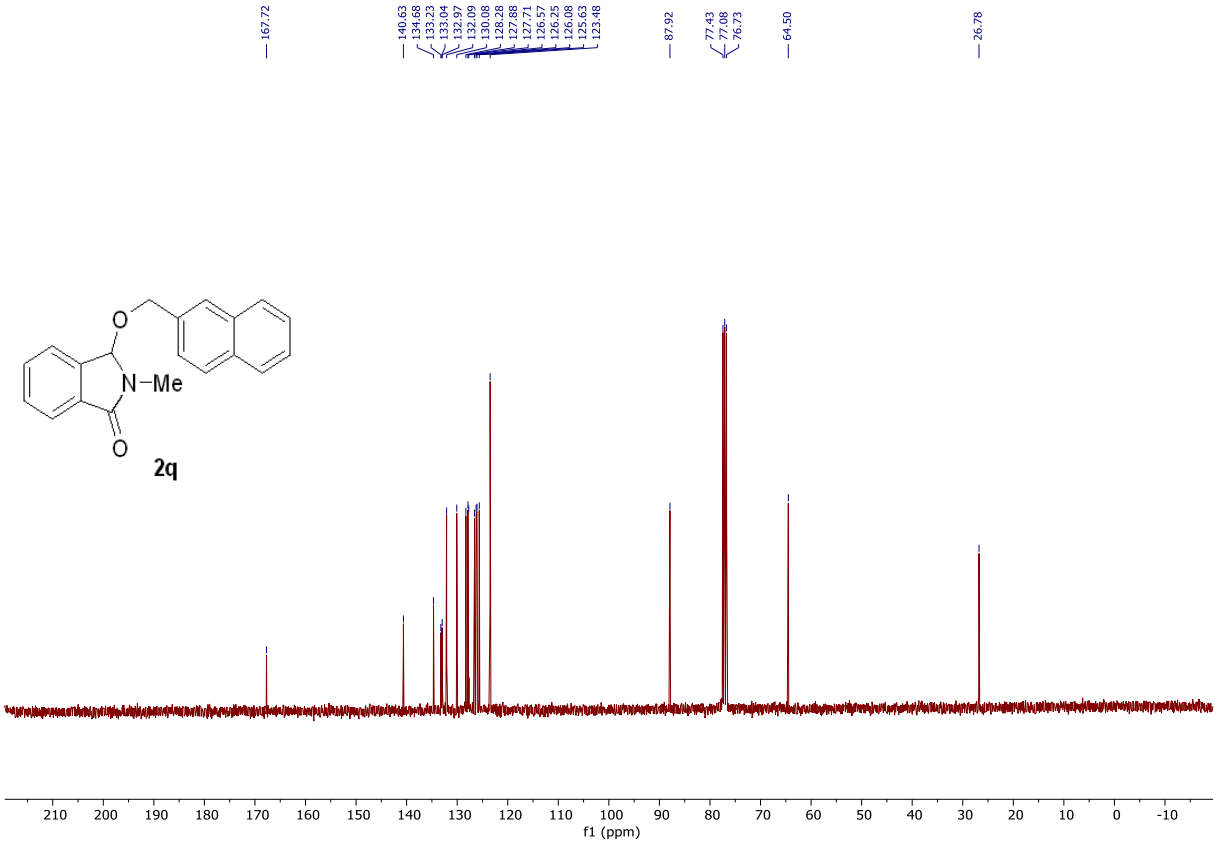
7.80
7.72
7.71
7.61
7.60
7.60
7.58
7.58
7.57
7.50
7.49
7.49
7.48
7.39
7.37
7.36
5.96
4.34
4.31
4.21
4.18
3.17
O
N–Me
O
2q
1.00
2.93
0.92
4.98
0.89
0.86
1.03
0.97
2.96
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)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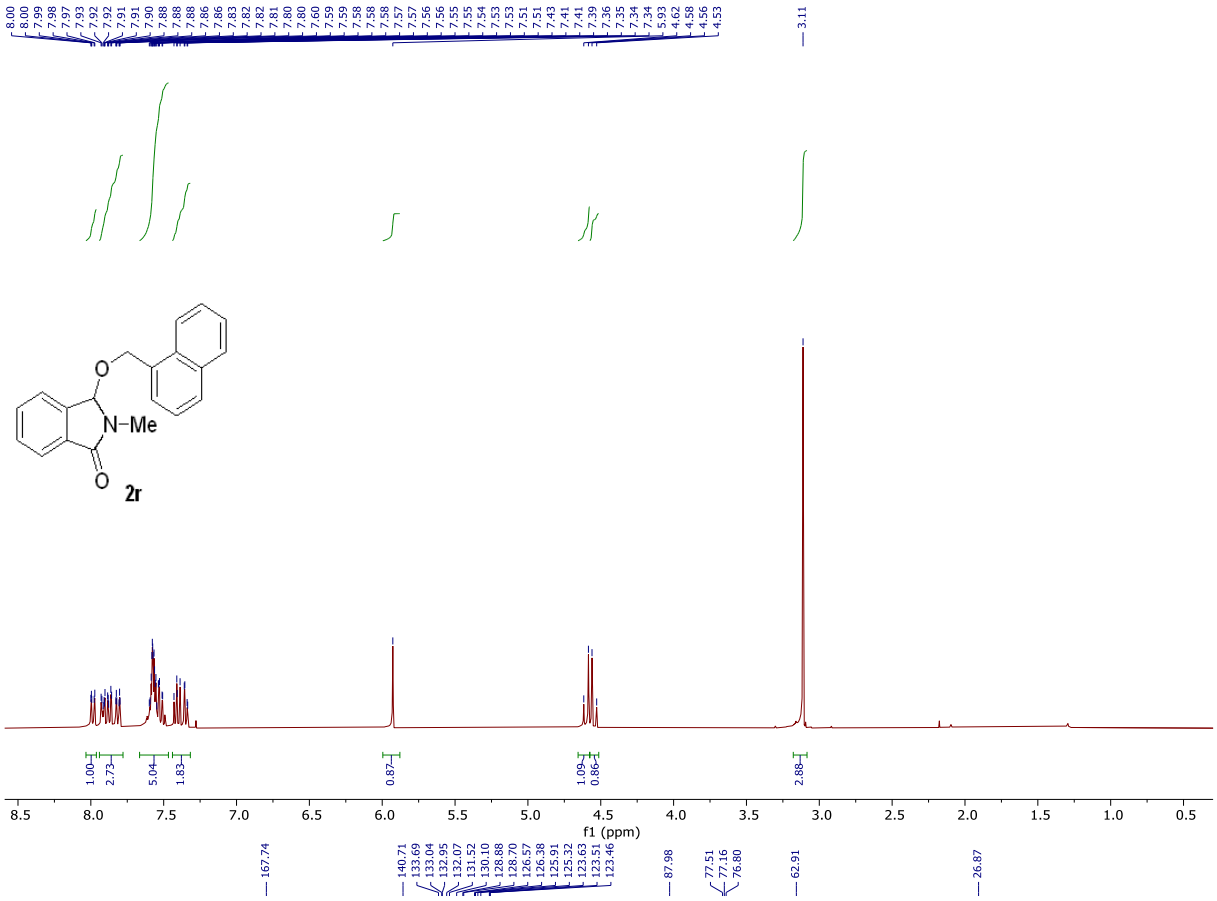
133.04
132.97
132.09
130.08
128.28
127.88
127.71
126.57
126.35
126.08
125.63
123.48
87.92
77.43
77.08
76.73
64.50
26.78
O
N–Me
O
2q
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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3.11
O
N–Me
O
2r
1.00 2.73 5.04 1.83 0.87 1.09 0.86 2.88
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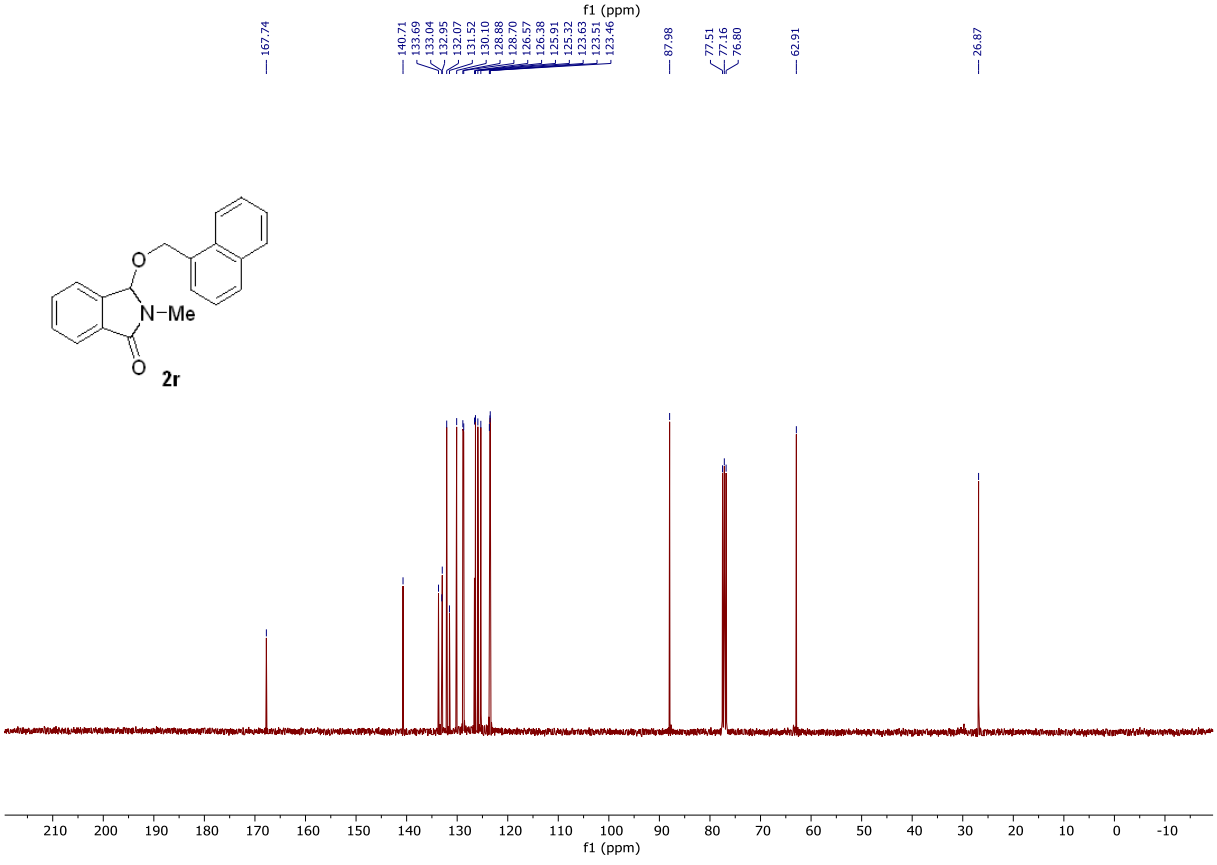
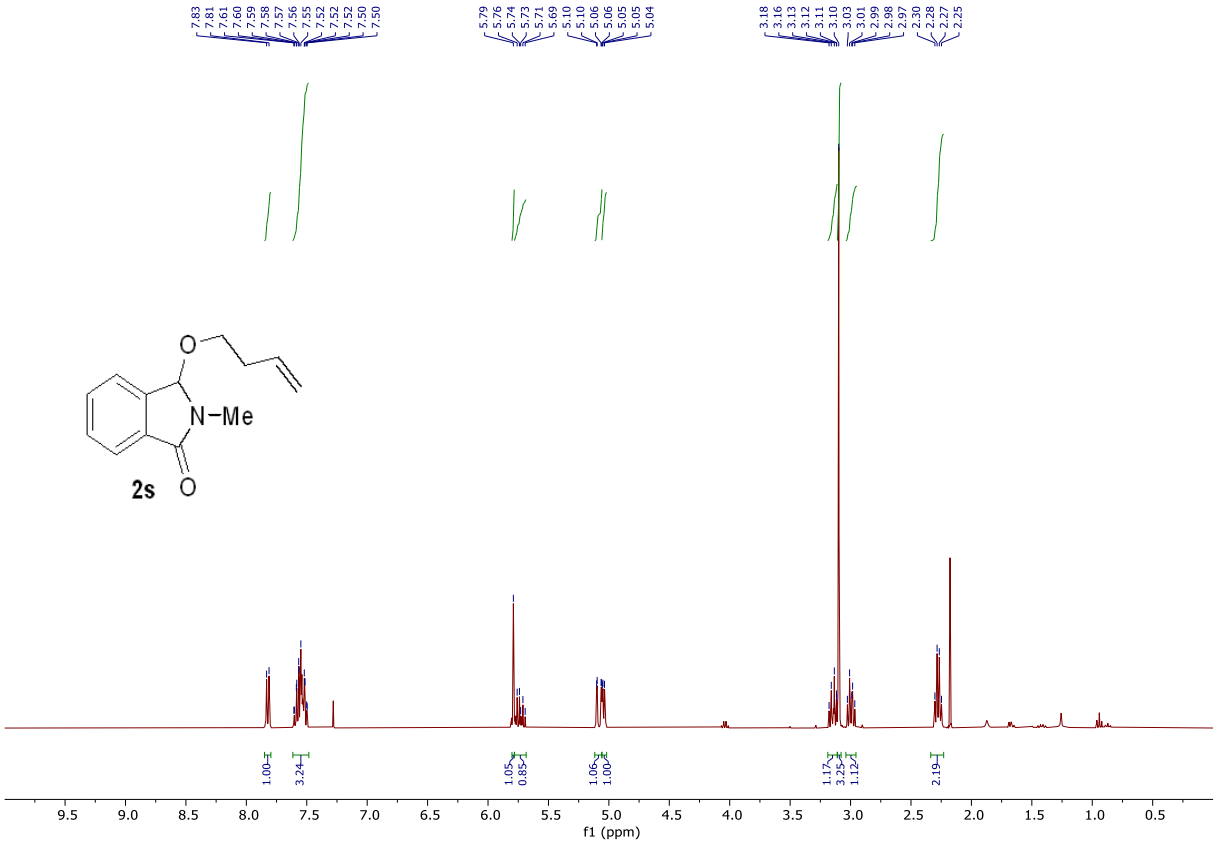
f1 (ppm)


167.74
140.71 133.69 133.04 132.95 132.07 131.52 130.10 128.88 128.70 126.57 126.38 125.91 125.32 123.63 123.51 123.46
87.98
77.51 77.16 76.80
62.91
26.87
O
N–Me
O
2r
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)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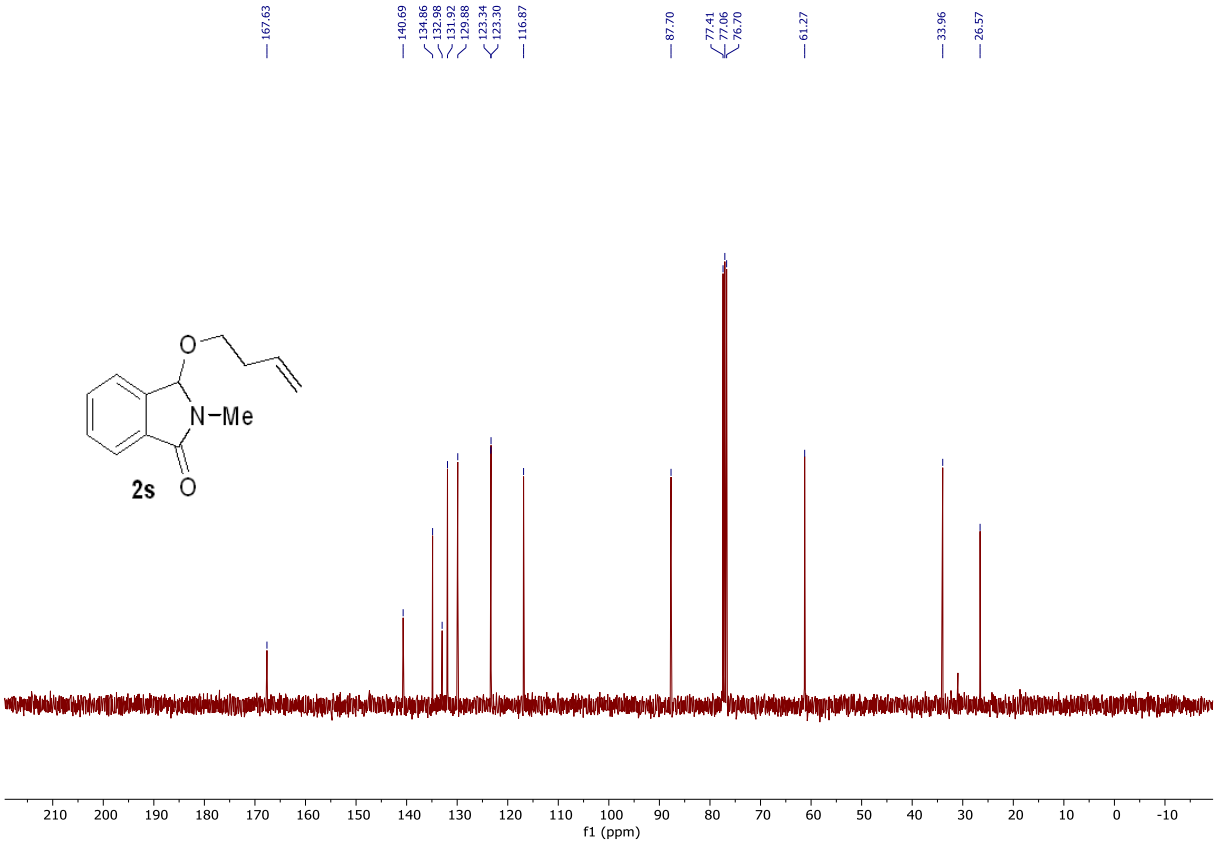
7.52
7.50
7.50
5.79
5.76
5.74
5.73
5.71
5.69
5.10
5.10
5.06
5.06
5.05
5.05
5.04
3.18
3.16
3.13
3.12
3.11
3.10
3.03
3.01
2.99
2.98
2.97
2.30
2.28
2.27
2.25
O
N–Me
O
2s
1.00
3.24
1.05
0.85
1.06
1.00
1.17
3.25
1.12
2.19
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)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129.88
123.34
123.30
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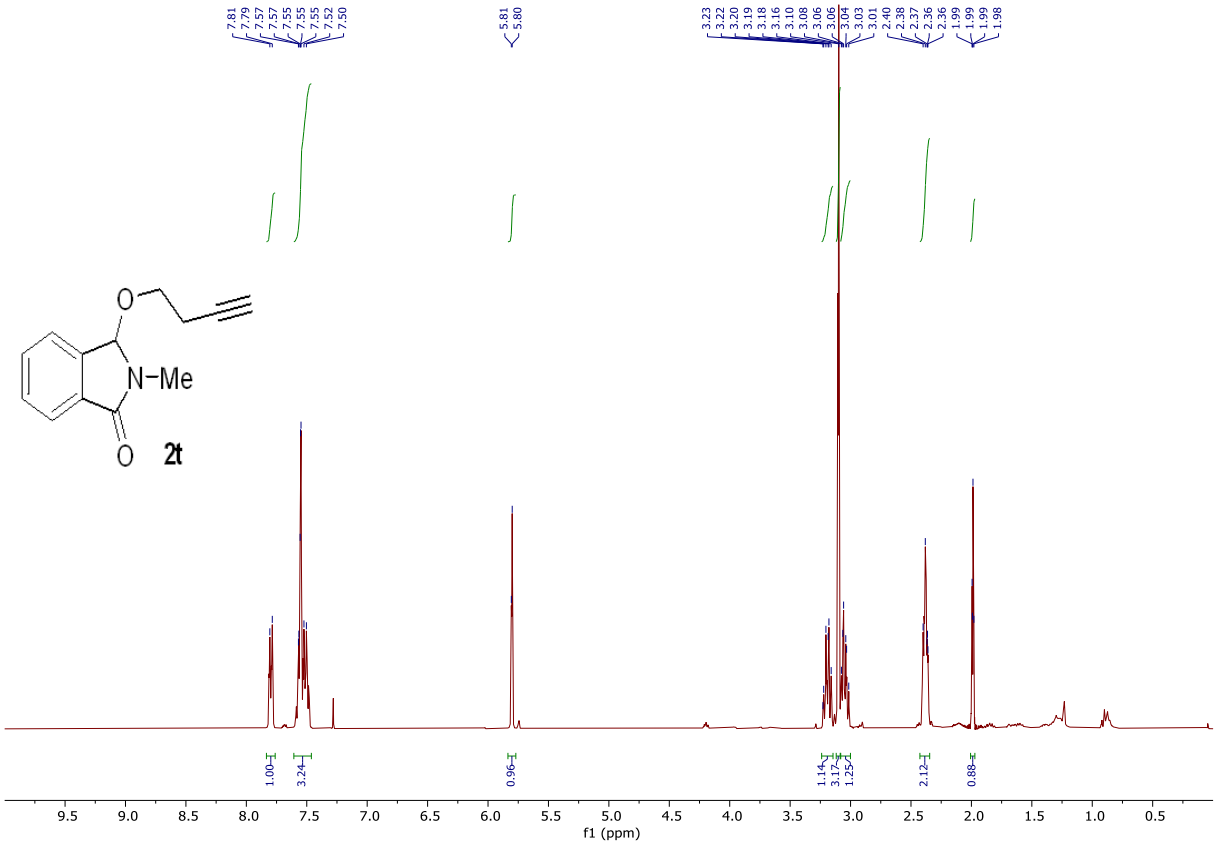
87.70
77.41
77.06
76.70
61.27
33.96
26.57
O
N–Me
O
2s
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)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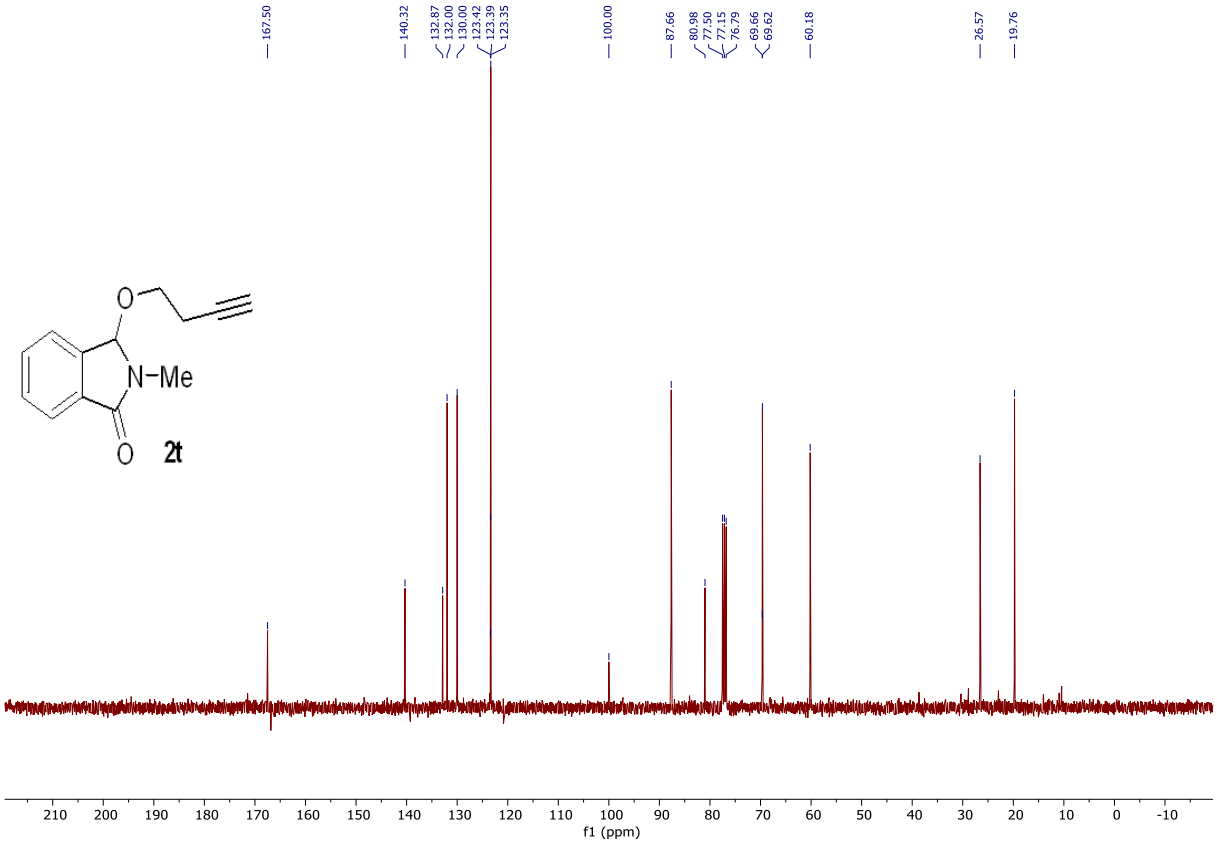
7.57
7.55
7.55
7.55
7.52
7.50
5.81
5.80
3.23
3.22
3.20
3.19
3.18
3.16
3.10
3.08
3.06
3.06
3.04
3.03
3.01
2.40
2.38
2.37
2.36
2.36
1.99
1.99
1.99
1.98
O
N−Me
O
2t
1.00
3.24
0.96
1.14
3.17
1.25
2.12
0.88
f1 (ppm)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123.39
123.35
100.00
87.66
80.98
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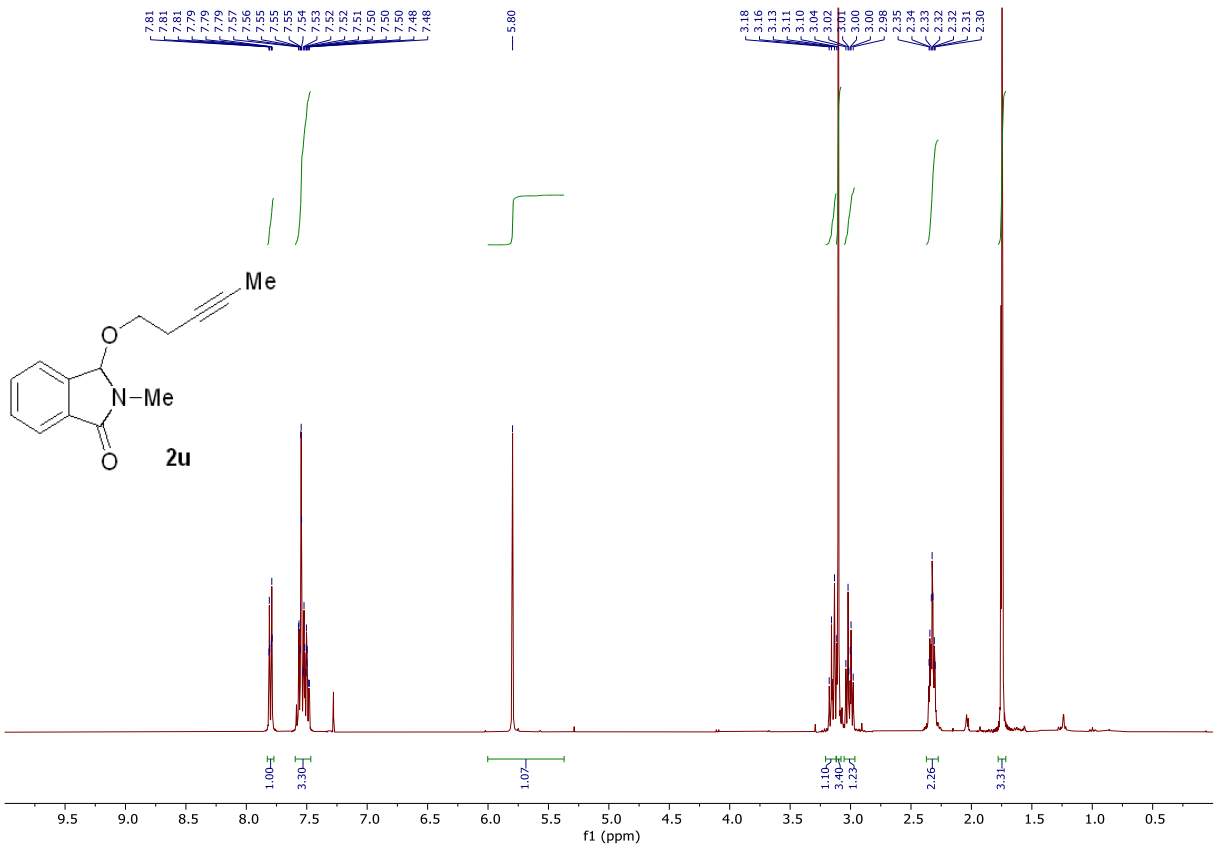
77.15
76.79
69.66
69.62
60.18
26.57
19.76
O
N−Me
O
2t
f1 (ppm)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7.81
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7.55
7.55
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7.52
7.52
7.51
7.50
7.50
7.50
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7.48
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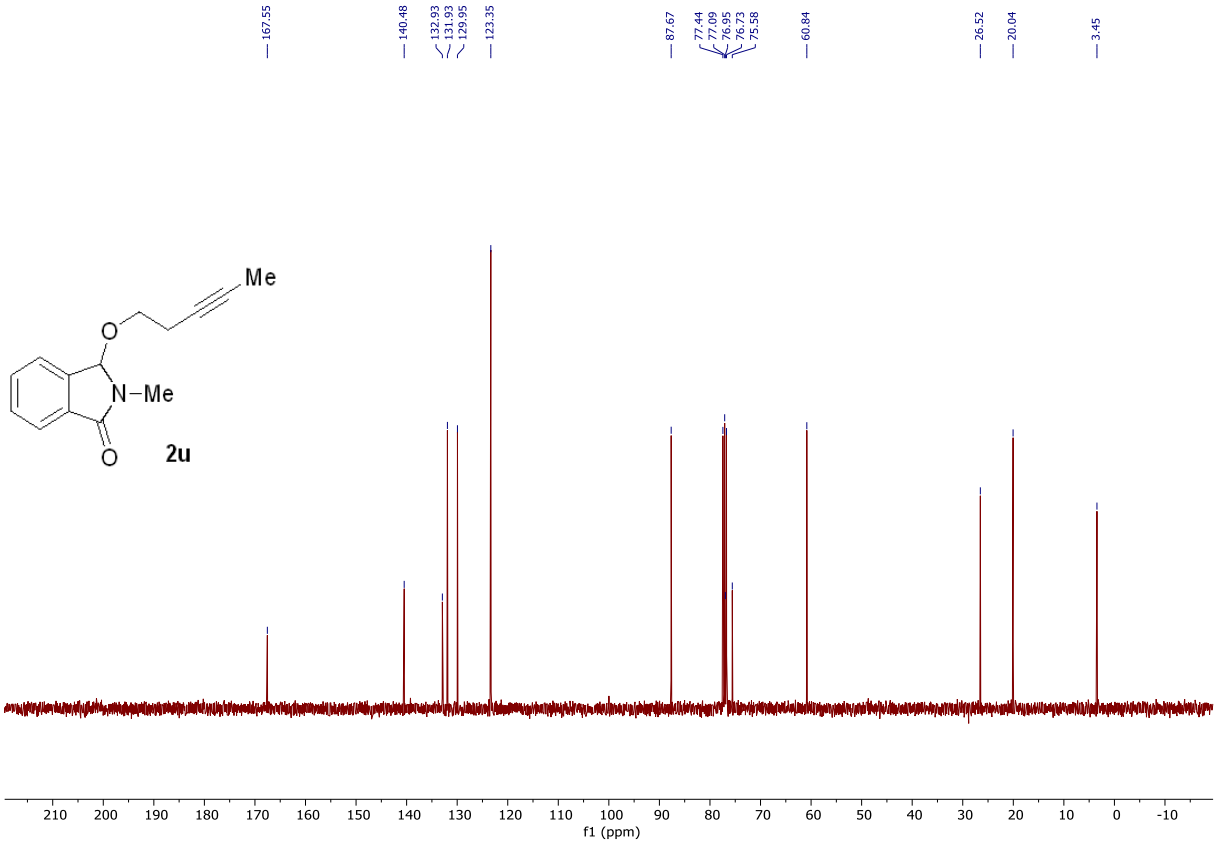
3.16
3.13
3.11
3.10
3.04
3.02
3.01
3.00
3.00
2.98
2.35
2.34
2.33
2.32
2.32
2.31
2.30
Me
O
N–Me
O
2u
1.00
3.30
1.07
1.10
3.40
1.23
2.26
3.31
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)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123.35
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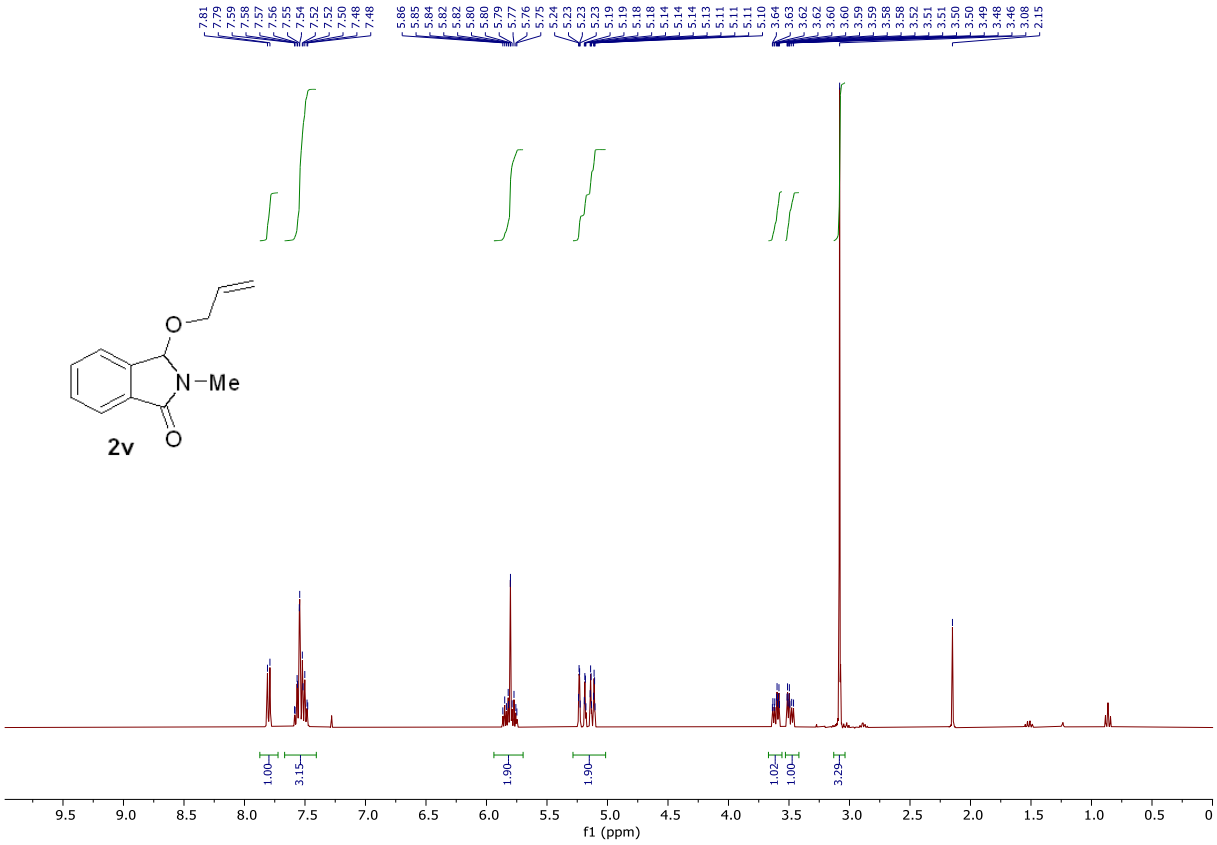
76.95
76.73
75.58
60.84
26.52
20.04
3.45
Me
O
N–Me
O
2u
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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7.54
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7.50
7.48
7.48
5.86
5.85
5.84
5.82
5.80
5.80
5.79
5.77
5.76
5.75
5.24
5.23
5.23
5.23
5.19
5.19
5.18
5.18
5.14
5.14
5.13
5.11
5.11
5.11
5.10
3.64
3.63
3.62
3.62
3.60
3.60
3.59
3.59
3.58
3.58
3.52
3.51
3.51
3.50
3.50
3.49
3.48
3.46
3.08
2.15
O
N−Me
O
2v
1.00
3.15
1.90
1.90
1.02
1.00
3.29
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
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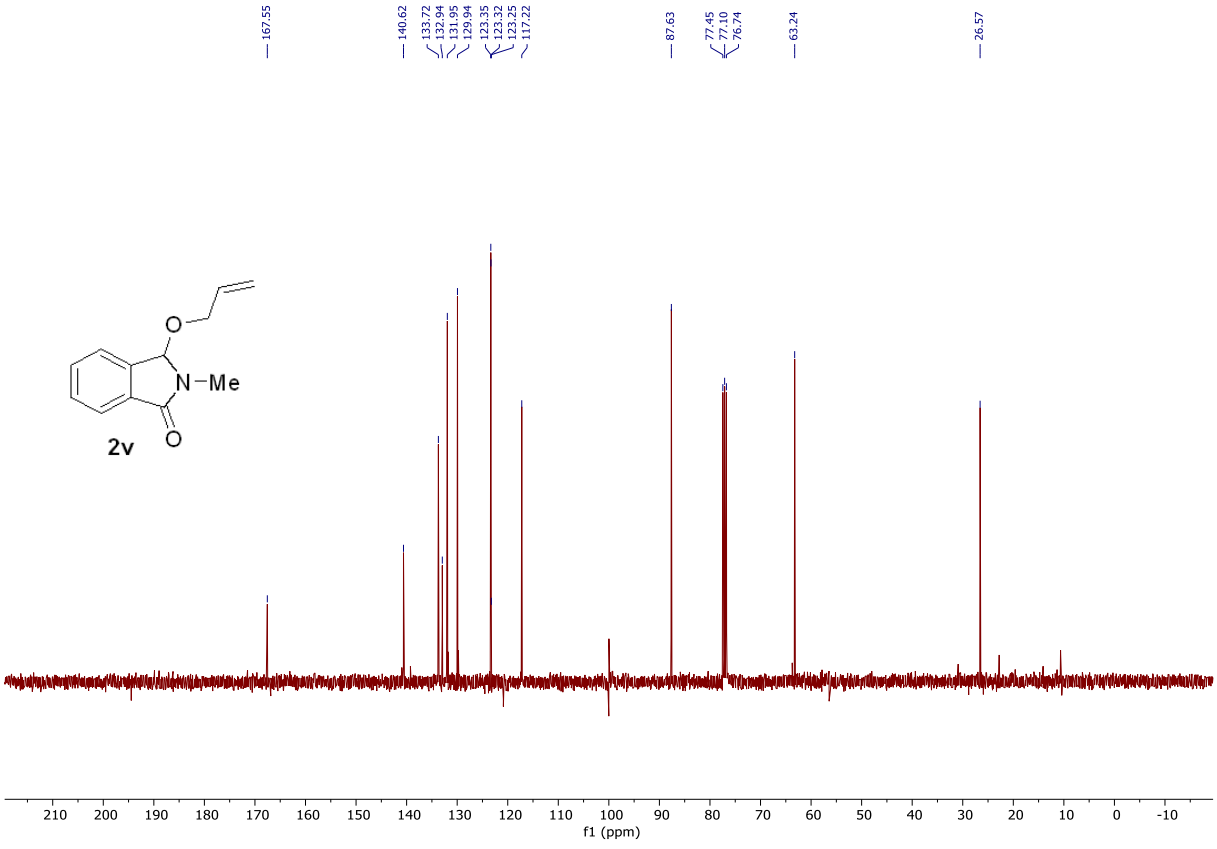
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0
f1 (ppm)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132.94
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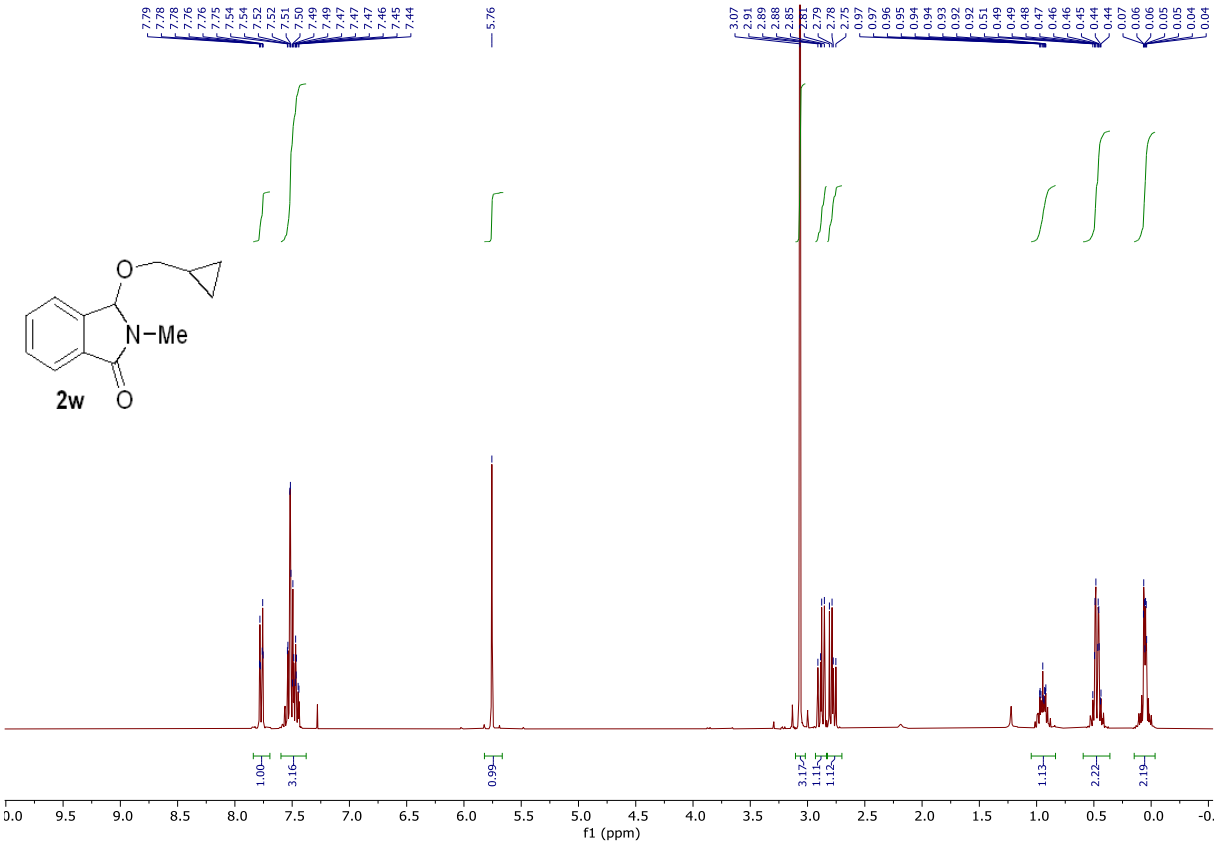
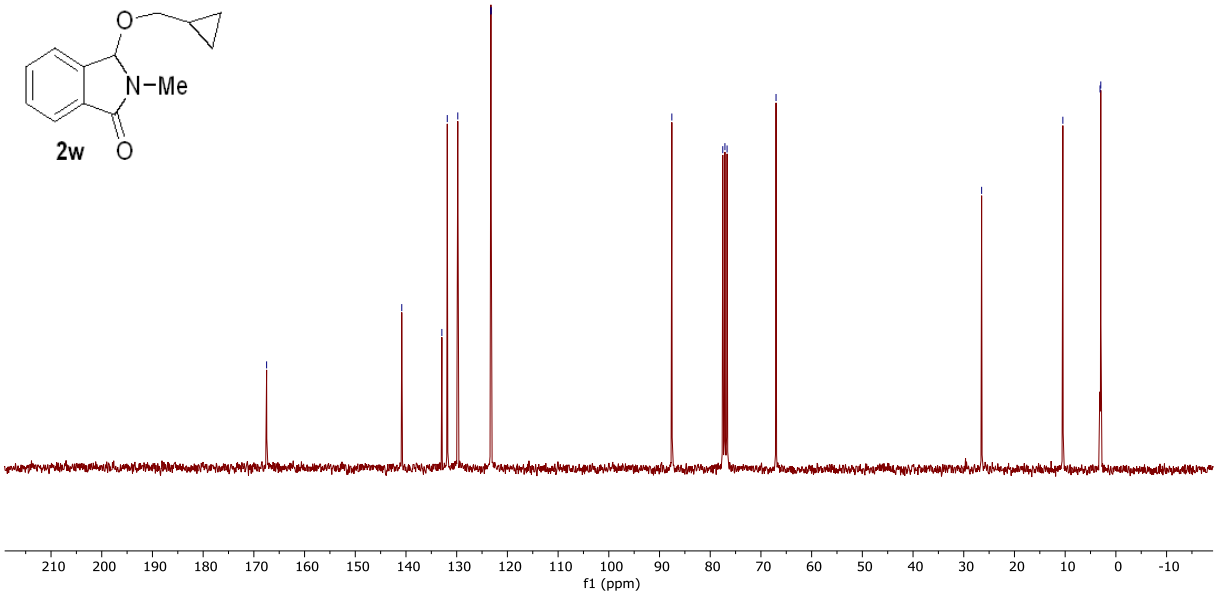
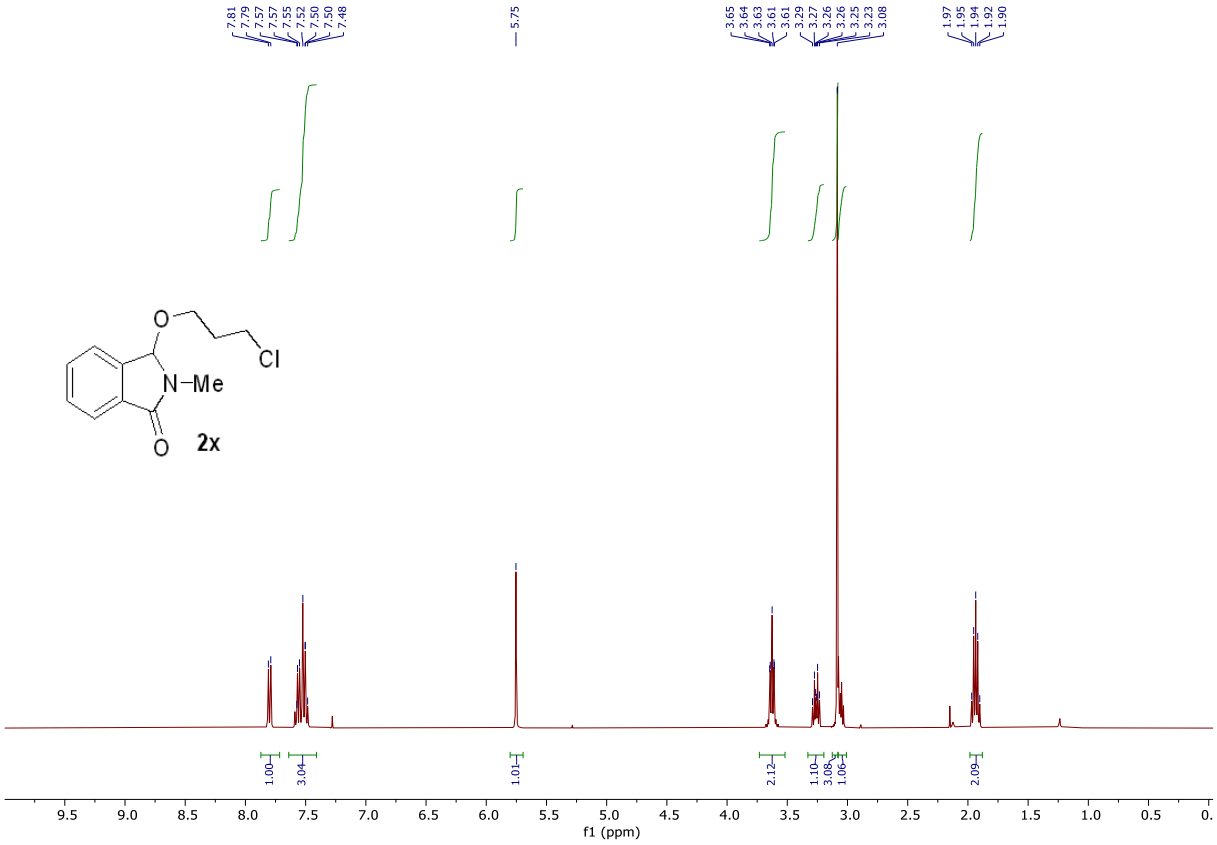
129.94
123.35
123.32
123.25
117.22
87.63
77.45
77.10
76.74
63.24
26.57
O
N−Me
O
2v
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


N–Me
O
2w
f1 (ppm)


N–Me
O
2w
f1 (ppm)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7.79
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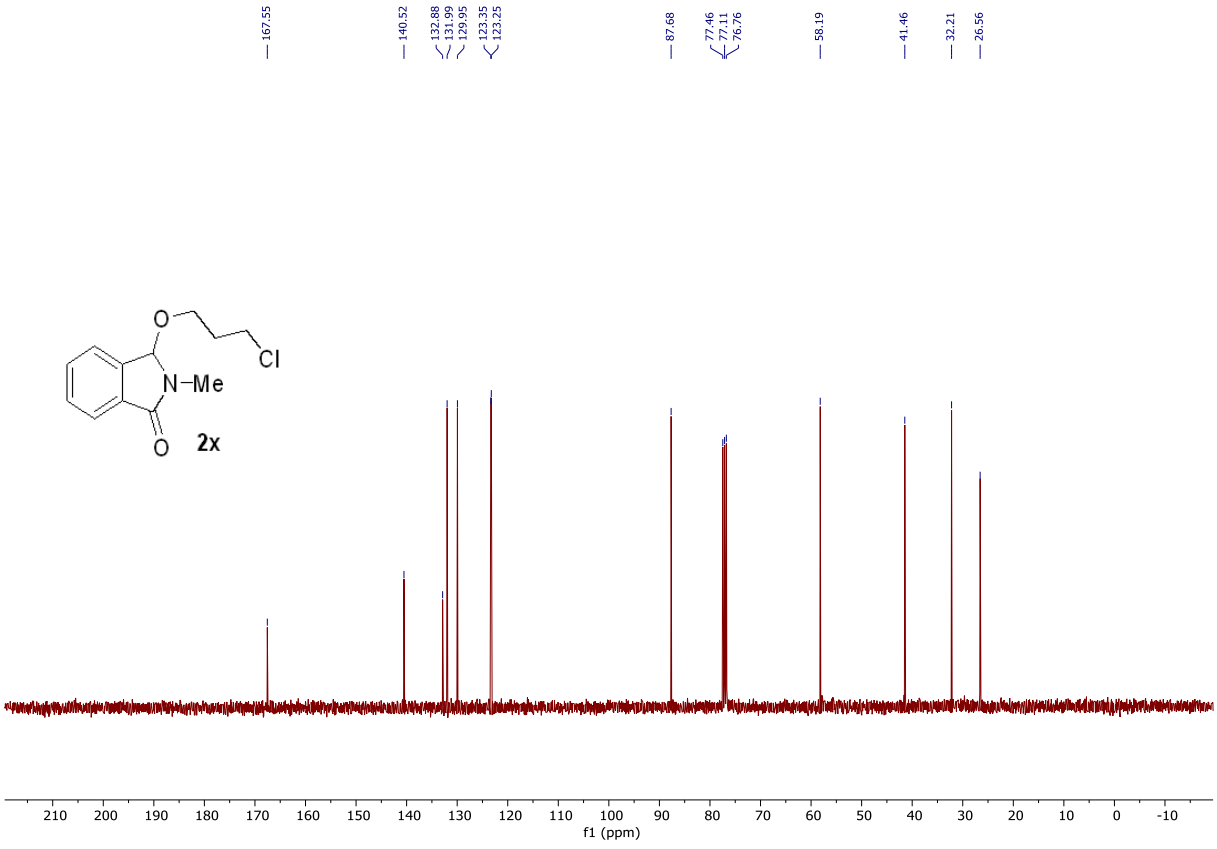
7.48
5.75
3.65
3.64
3.63
3.61
3.61
3.29
3.27
3.26
3.26
3.25
3.23
3.08
1.97
1.95
1.94
1.92
1.90
O
Cl
N–Me
O
2x
1.00
3.04
1.01
2.12
1.10
3.08
1.06
2.09
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.
f1 (ppm)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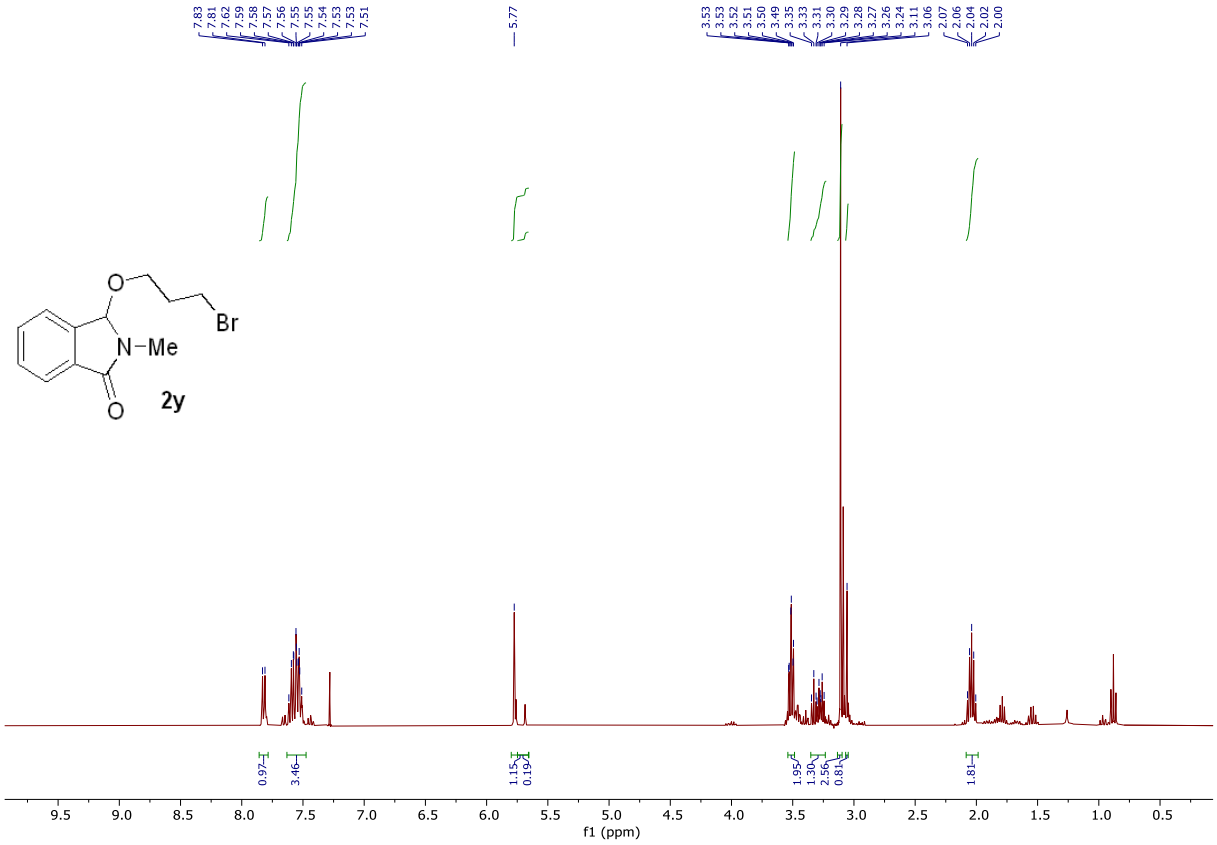
129.95
123.35
123.25
87.68
77.46
77.11
76.76
58.19
41.46
32.21
26.56
O
Cl
N–Me
O
2x
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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7.56
7.55
7.55
7.54
7.53
7.53
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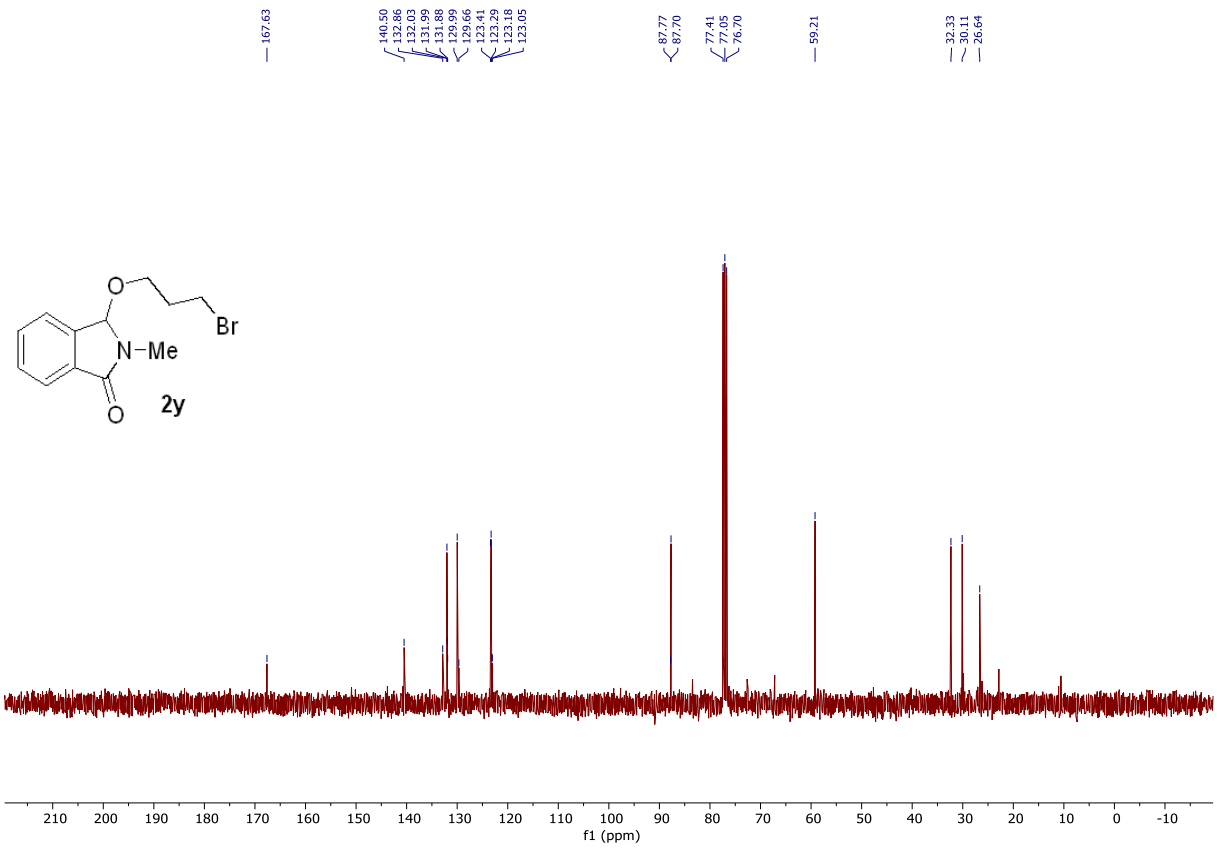
5.77
3.53
3.53
3.52
3.51
3.50
3.49
3.35
3.33
3.31
3.30
3.29
3.28
3.27
3.26
3.24
3.11
3.06
2.07
2.06
2.04
2.02
2.00
O
Br
N–Me
O
2y
0.97
3.46
1.15
0.19
1.95
1.30
2.56
0.81
1.81
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)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140.50
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132.03
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131.88
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129.66
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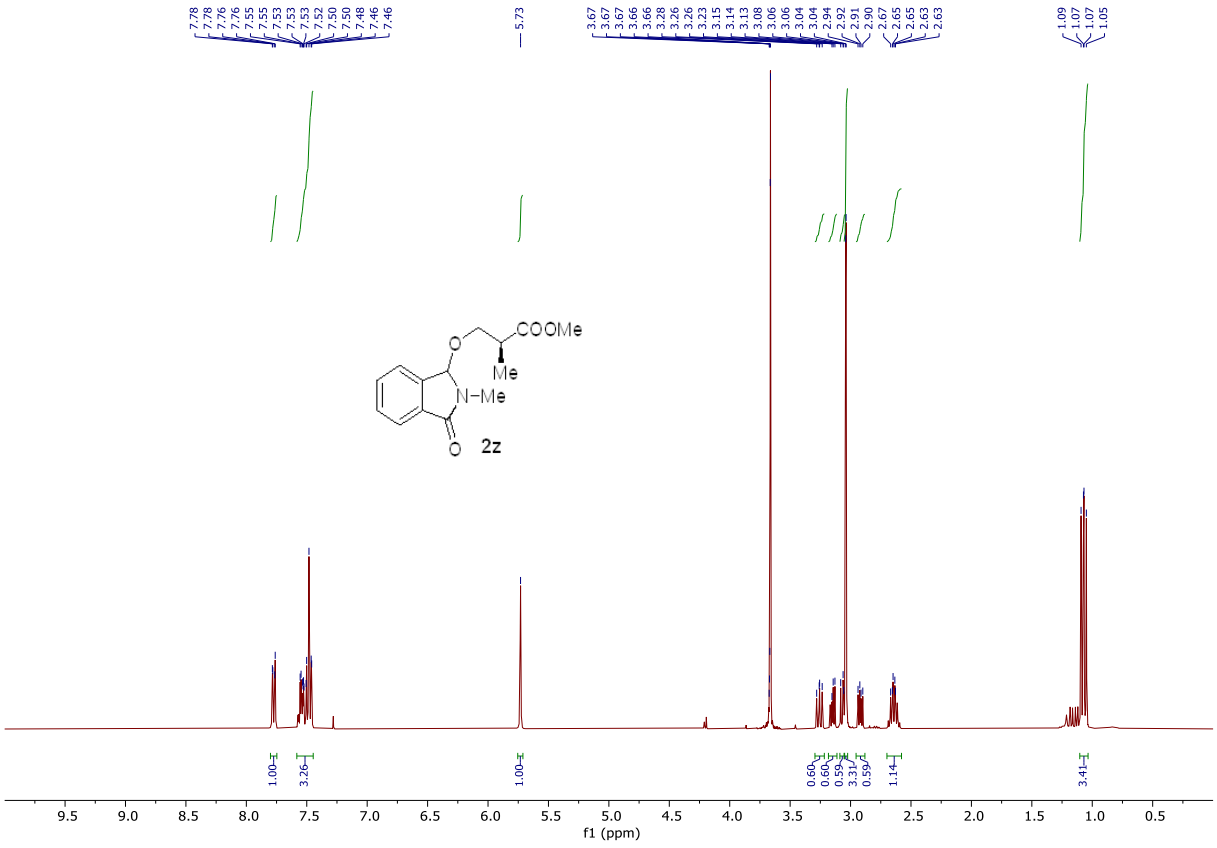
123.29
123.18
123.05
87.77
87.70
77.41
77.05
76.70
59.21
32.33
30.11
26.64
O
Br
N–Me
O
2y
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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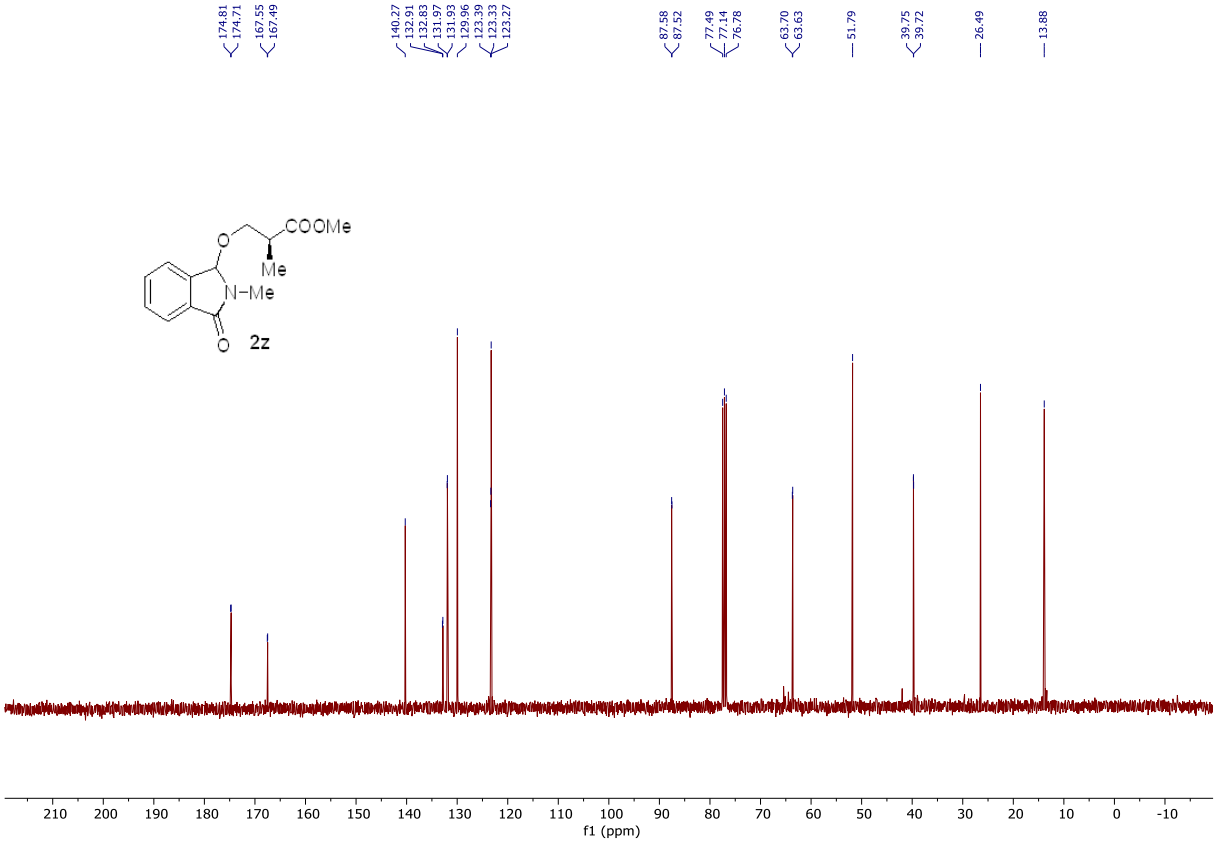
7.78
7.76
7.76
7.55
7.55
7.53
7.53
7.53
7.52
7.50
7.50
7.48
7.46
7.46
5.73
3.67
3.67
3.67
3.66
3.66
3.28
3.26
3.26
3.23
3.15
3.14
3.13
3.08
3.06
3.06
3.04
3.04
2.94
2.92
2.91
2.90
2.67
2.65
2.65
2.63
2.63
1.09
1.07
1.07
1.07
1.05
COOMe
Me
N–Me
O
2z
1.00
3.26
1.00
0.60
0.60
0.59
3.31
0.59
1.14
3.41
9.5
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)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174.71
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167.49
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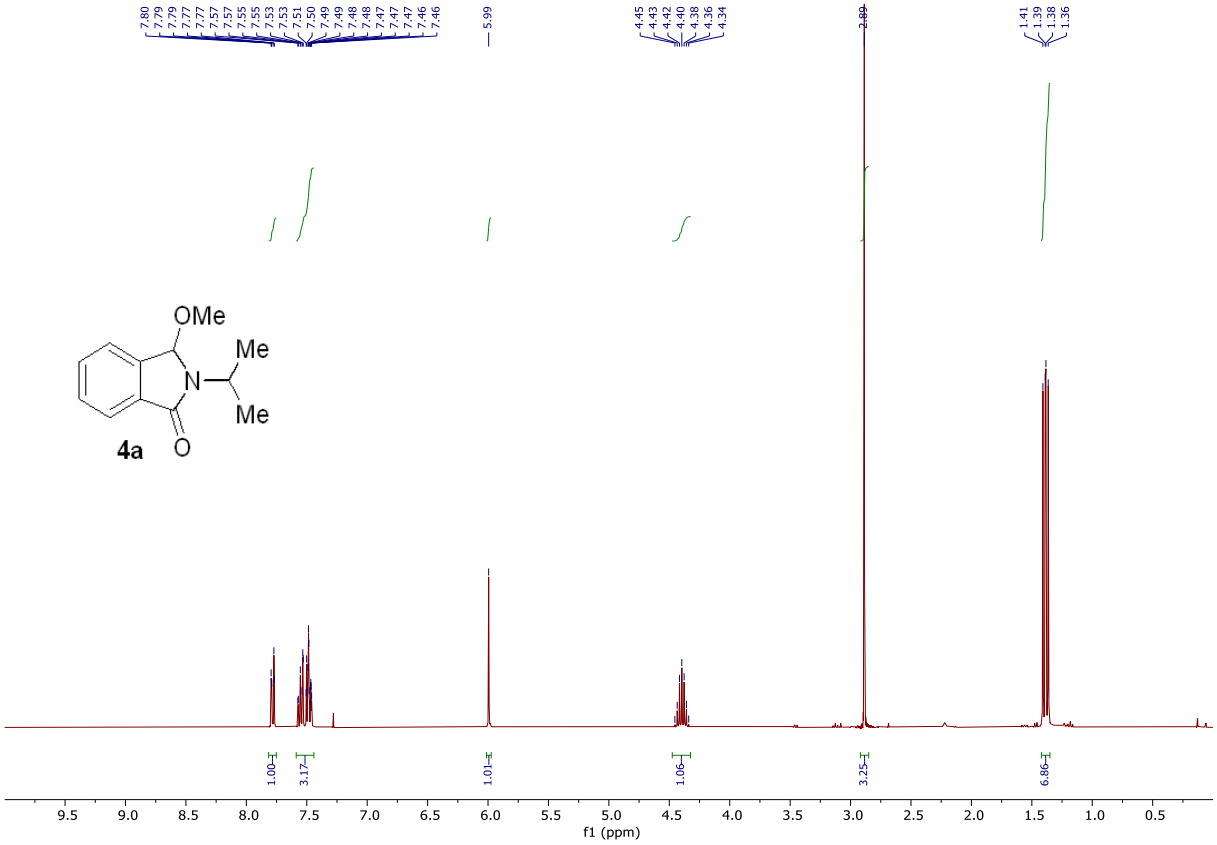
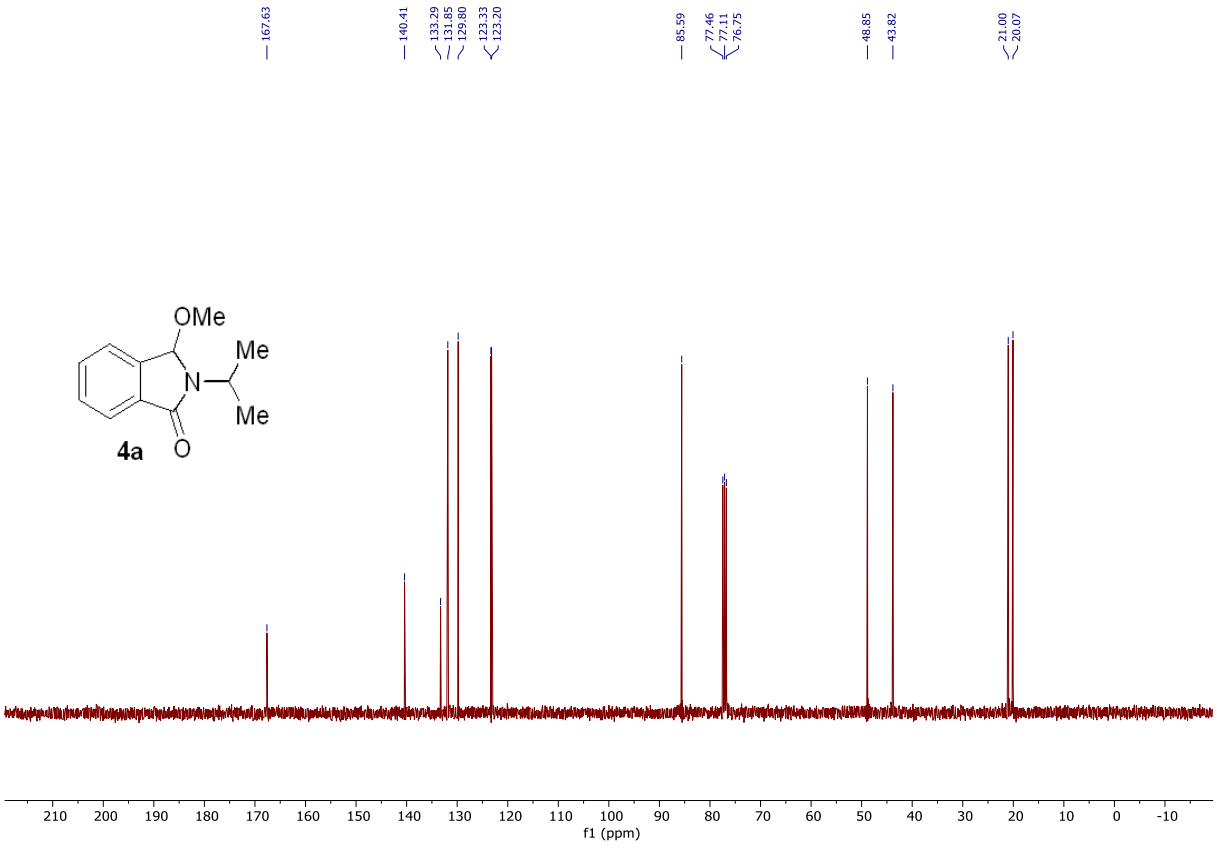
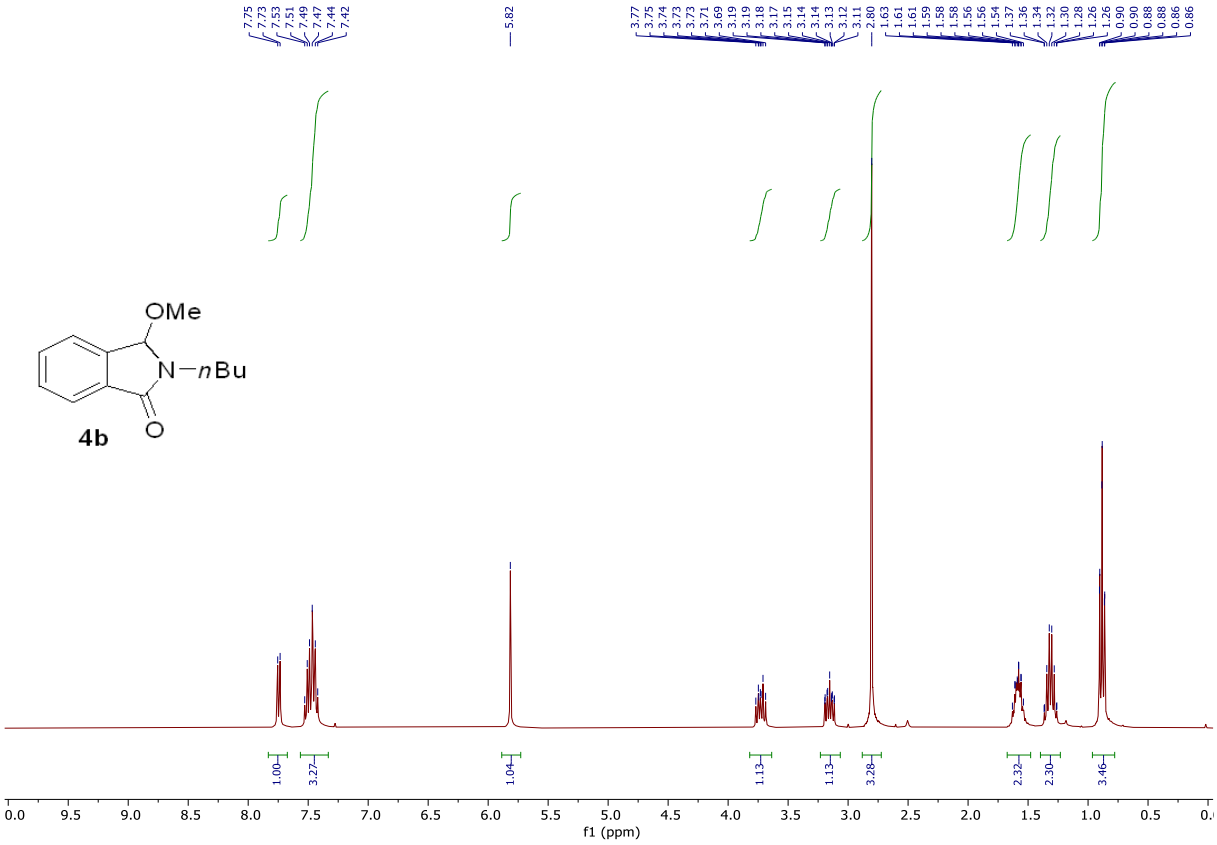
132.91
132.83
131.97
131.93
129.96
123.39
123.33
123.27
87.58
87.52
77.49
77.14
76.78
63.70
63.63
51.79
39.75
39.72
26.49
13.88
COOMe
Me
N–Me
O
2z
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

Characterization data of reductive methoxylation of *N*-substituted phthalimides



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7.47
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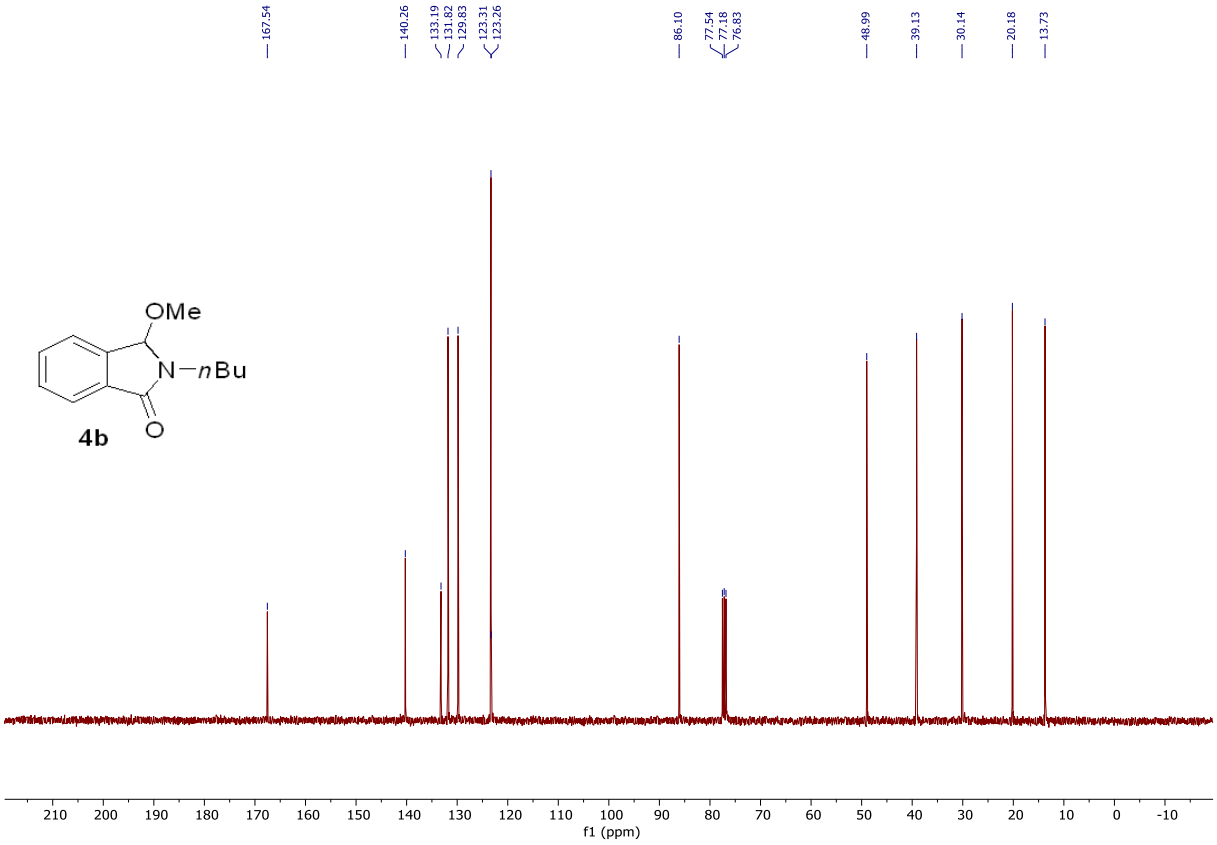
7.42
5.82
3.77
3.75
3.74
3.73
3.73
3.71
3.69
3.19
3.19
3.18
3.17
3.15
3.14
3.14
3.13
3.12
3.11
2.80
1.63
1.61
1.61
1.59
1.58
1.58
1.56
1.56
1.54
1.37
1.36
1.34
1.32
1.30
1.28
1.26
1.26
0.90
0.90
0.88
0.88
0.86
0.86
OMe
N—nBu
O
4b
1.00
3.27
1.04
1.13
1.13
3.28
2.32
2.30
3.46
f1 (ppm)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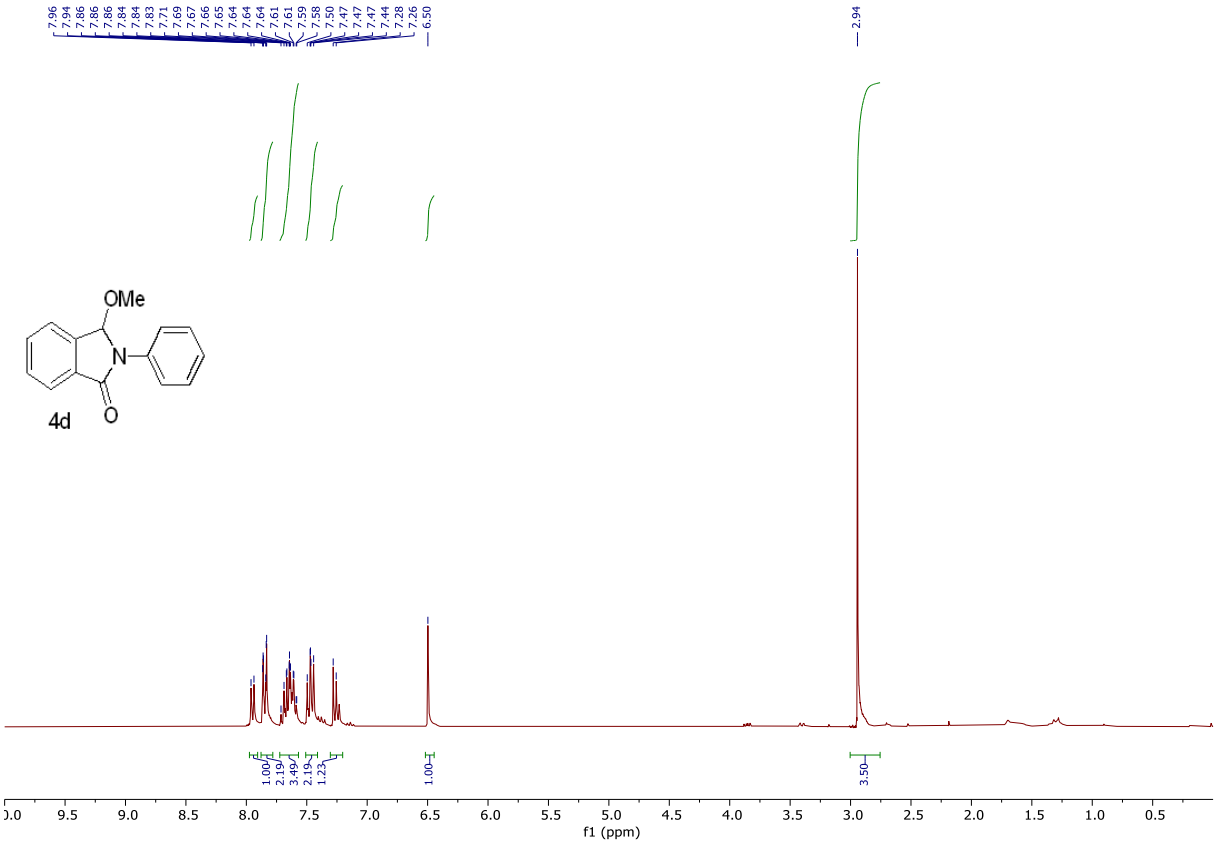
131.82
129.83
123.31
123.26
86.10
77.54
77.18
76.83
48.99
39.13
30.14
20.18
13.73
OMe
N—nBu
O
4b
f1 (ppm)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7.86
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7.84
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7.71
7.69
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7.66
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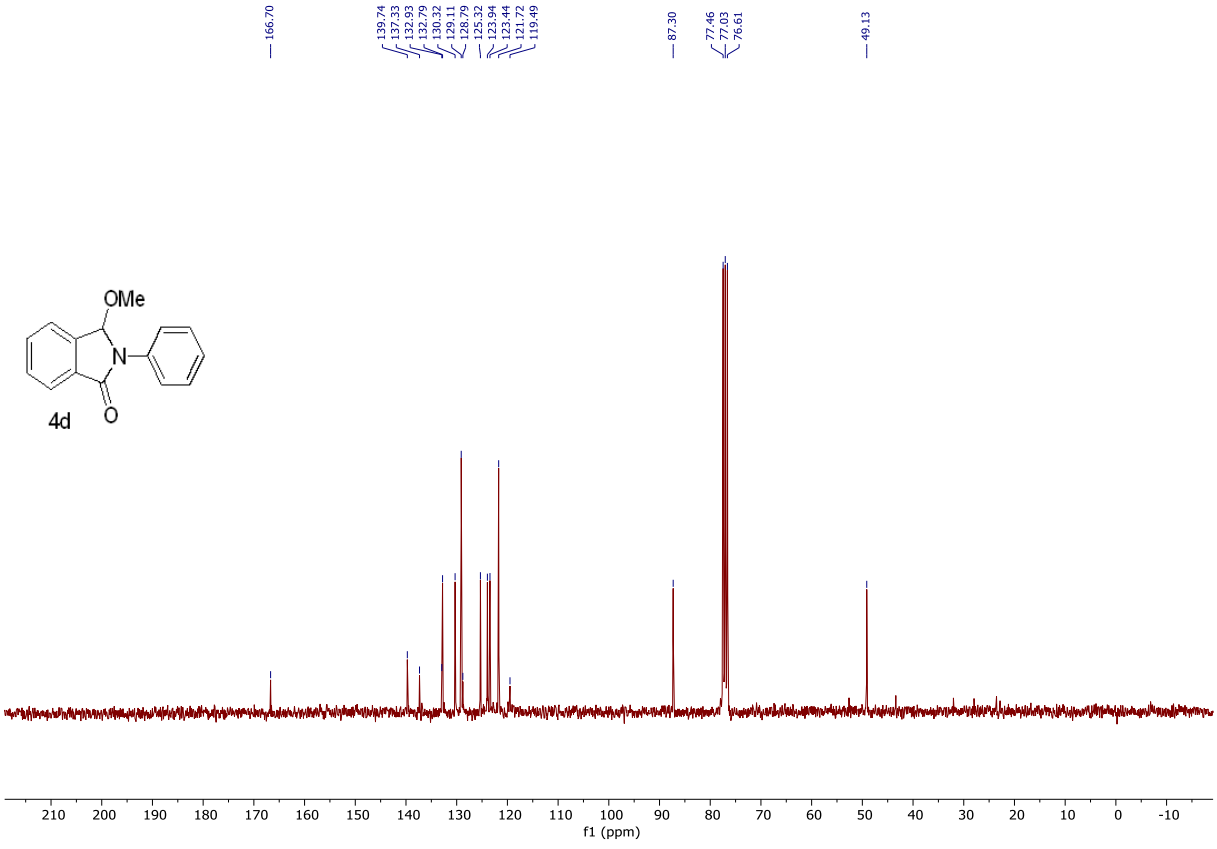
7.64
7.64
7.64
7.61
7.61
7.59
7.58
7.50
7.47
7.47
7.47
7.44
7.28
7.26
6.50
2.94
OMe
N
O
4d
1.00
2.19
3.49
2.19
1.23
1.00
3.50
f1 (ppm)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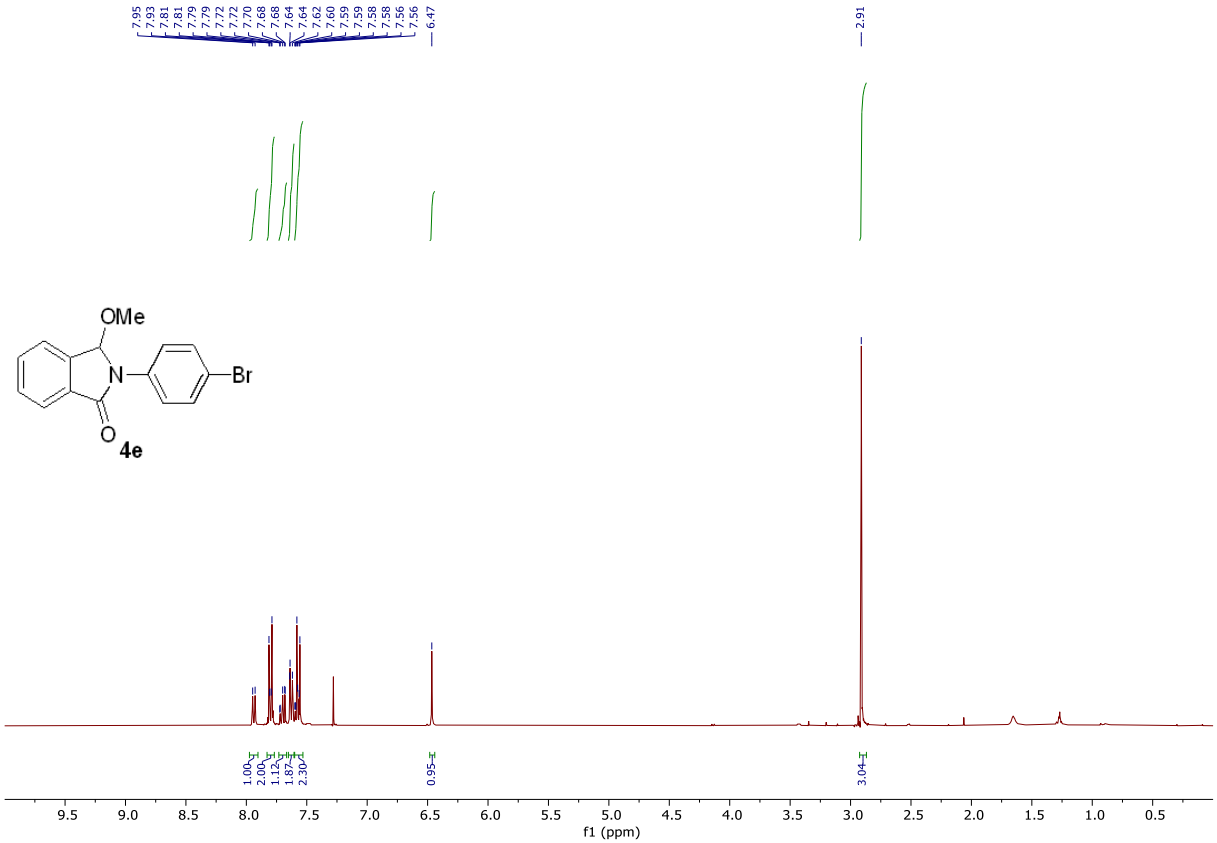
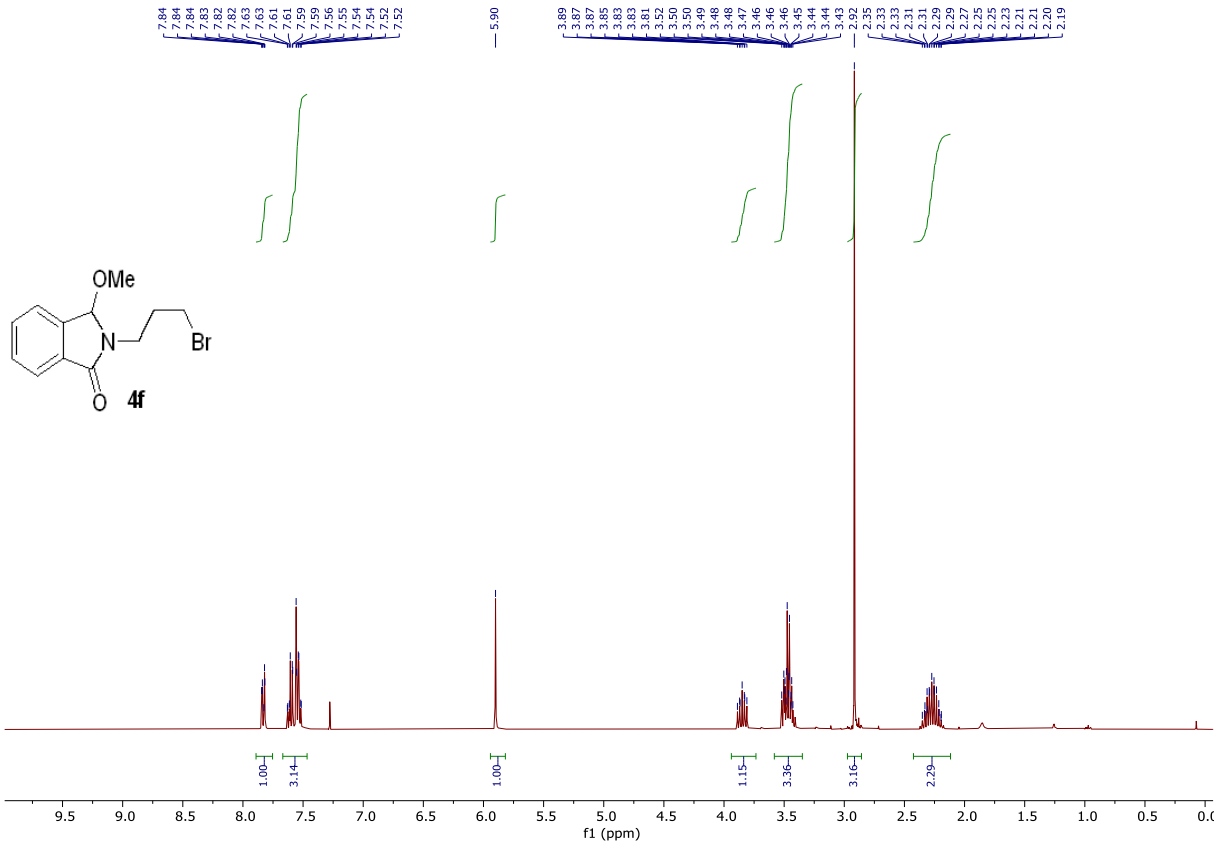
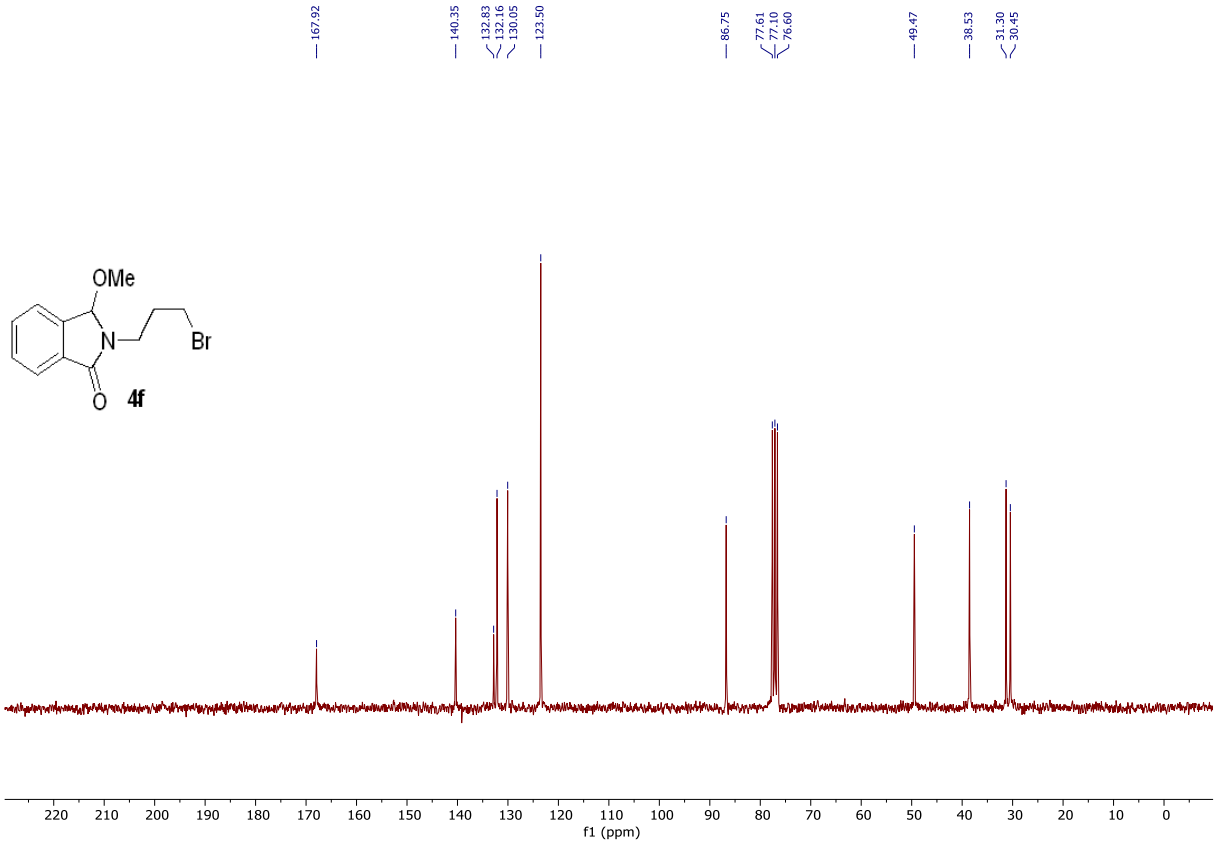
128.79
125.32
123.94
123.44
121.72
119.49
87.30
77.46
77.03
76.61
49.13
OMe
N
O
4d
f1 (ppm)


OMe
N
Br
O
4e
f1 (ppm)


OMe
N
Br
O
4f
f1 (ppm)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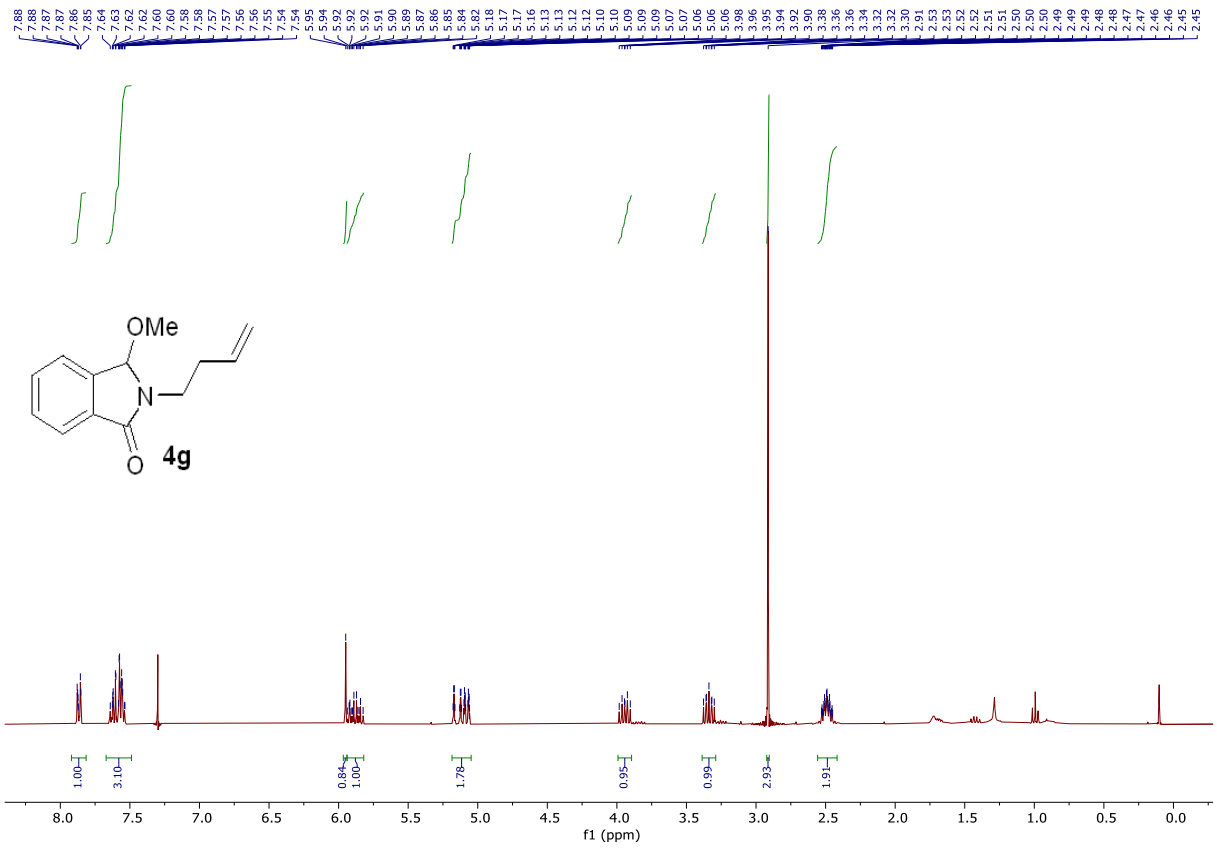
76.60
49.47
38.53
31.30
30.45
OMe
N
Br
O
4f
f1 (ppm)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N
O
4g
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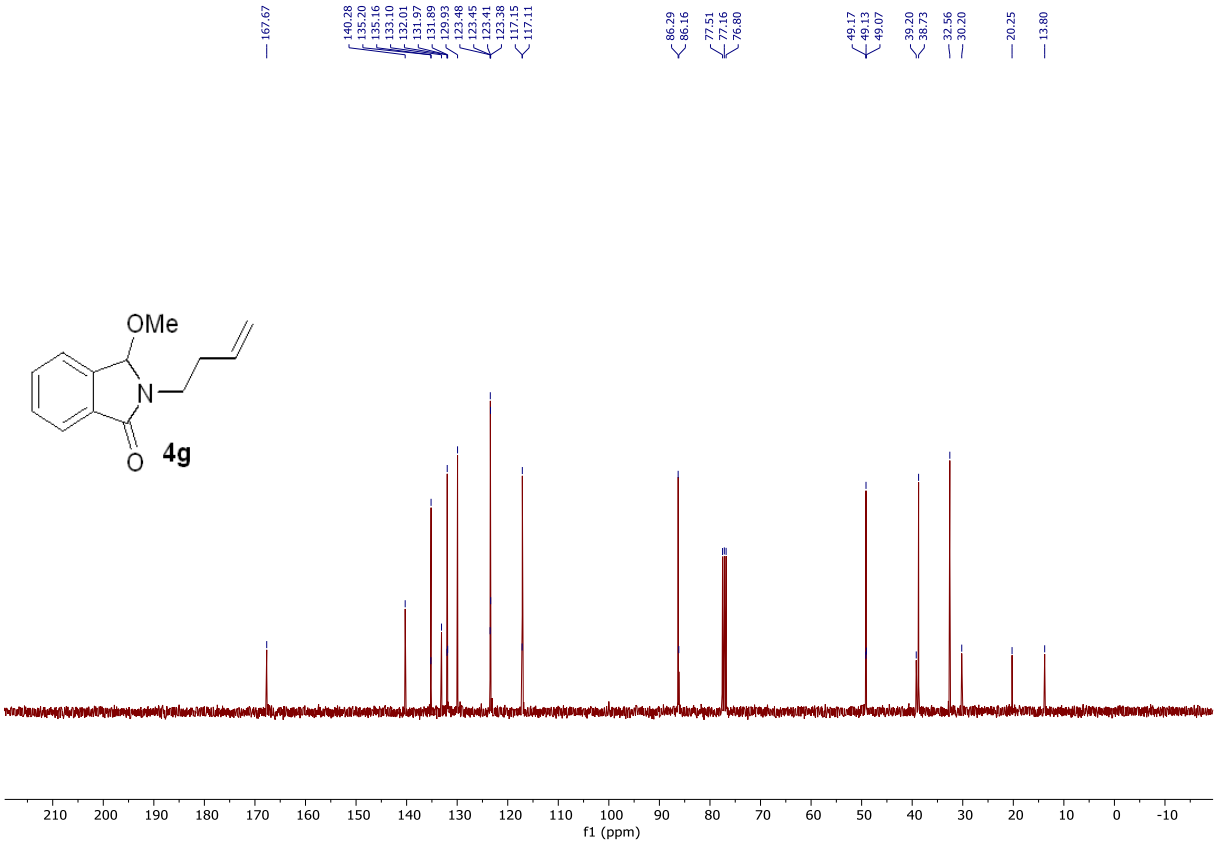
0.84
1.00
1.78
0.95
0.99
2.93
1.91
f1 (ppm)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123.45
123.41
123.38
117.15
117.11
86.29
86.16
77.51
77.16
76.80
49.17
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38.73
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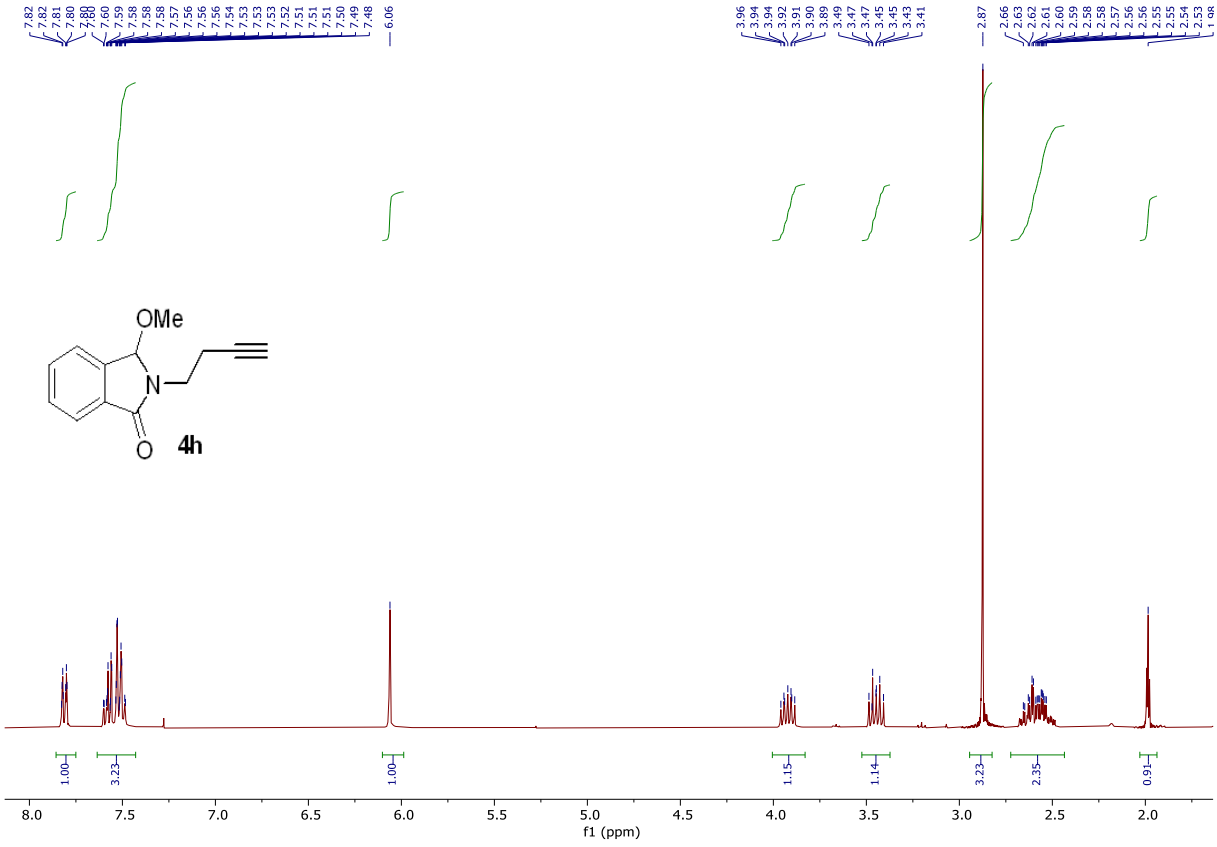
13.80
OMe
N
O
4g
f1 (ppm)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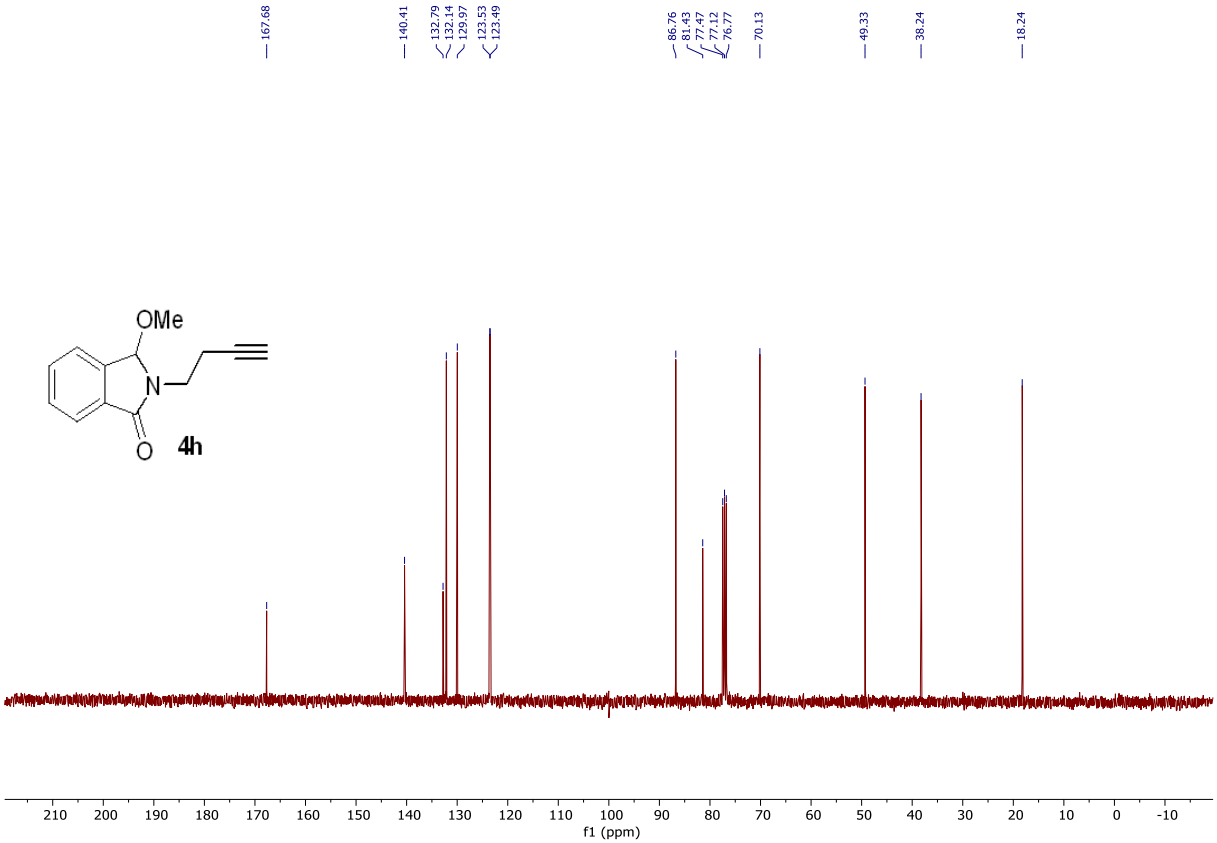
7.50
7.49
7.48
6.06
3.96
3.94
3.92
3.91
3.90
3.89
3.49
3.47
3.45
3.43
3.41
2.87
2.66
2.63
2.62
2.61
2.60
2.59
2.58
2.57
2.56
2.55
2.54
2.53
OMe
N
O
4h
1.00
3.23
1.00
1.15
1.14
3.23
2.35
0.91
f1 (ppm)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132.14
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123.49
86.76
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77.47
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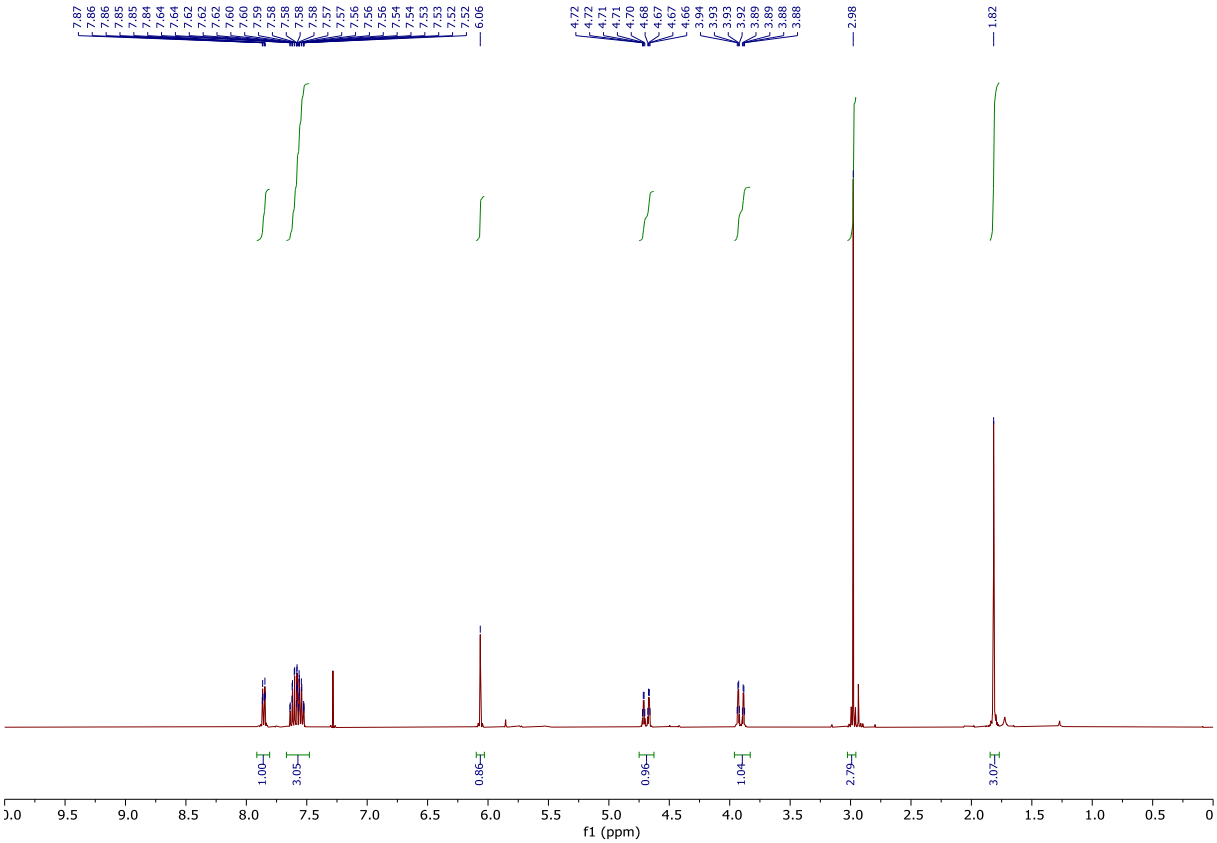
76.77
70.13
49.33
38.24
18.24
OMe
N
O
4h
f1 (ppm)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7.64
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7.62
7.60
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7.59
7.58
7.58
7.58
7.58
7.57
7.57
7.56
7.56
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7.54
7.54
7.53
7.52
7.52
6.06
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4.72
4.71
4.71
4.70
4.68
4.67
4.67
4.66
3.94
3.93
3.93
3.92
3.89
3.89
3.88
3.88
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1.82
1.00
3.05
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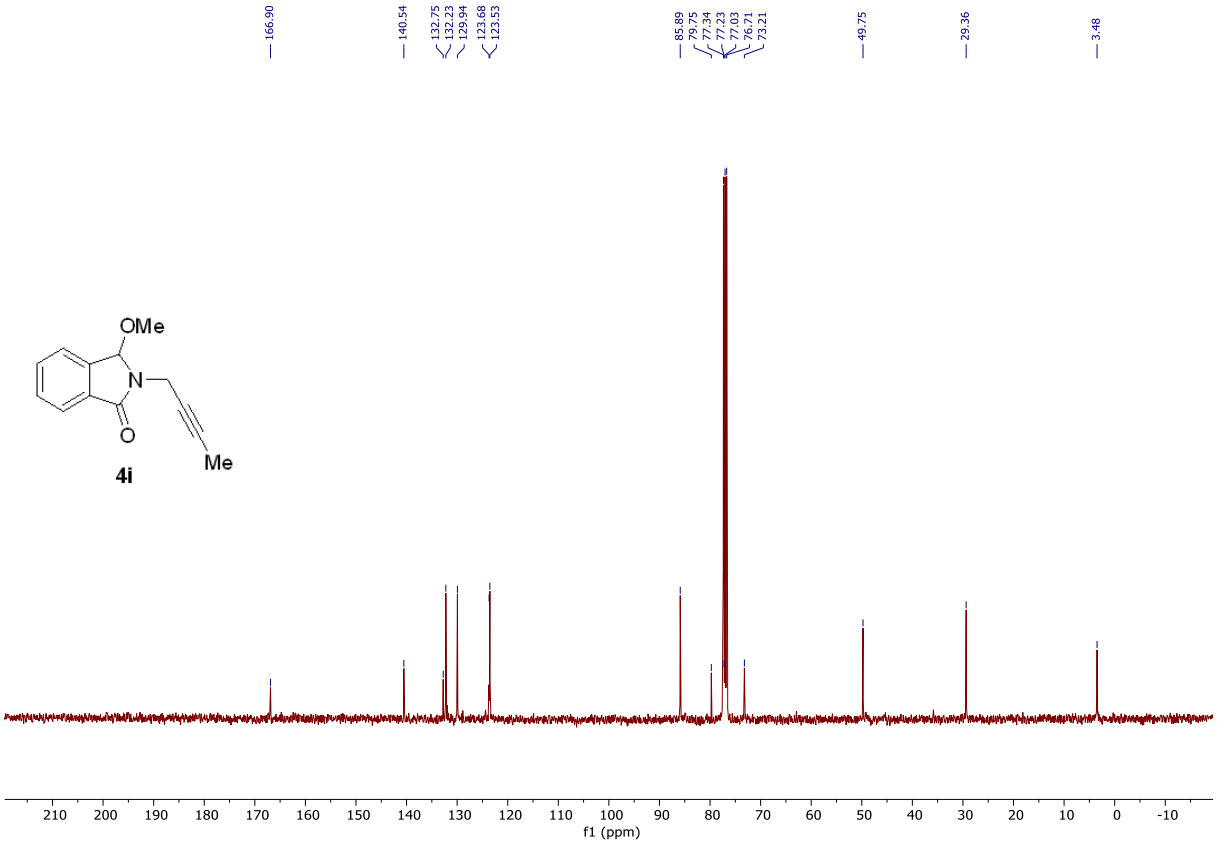
0.96
1.04
2.79
3.07
f1 (ppm)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85.89
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77.34
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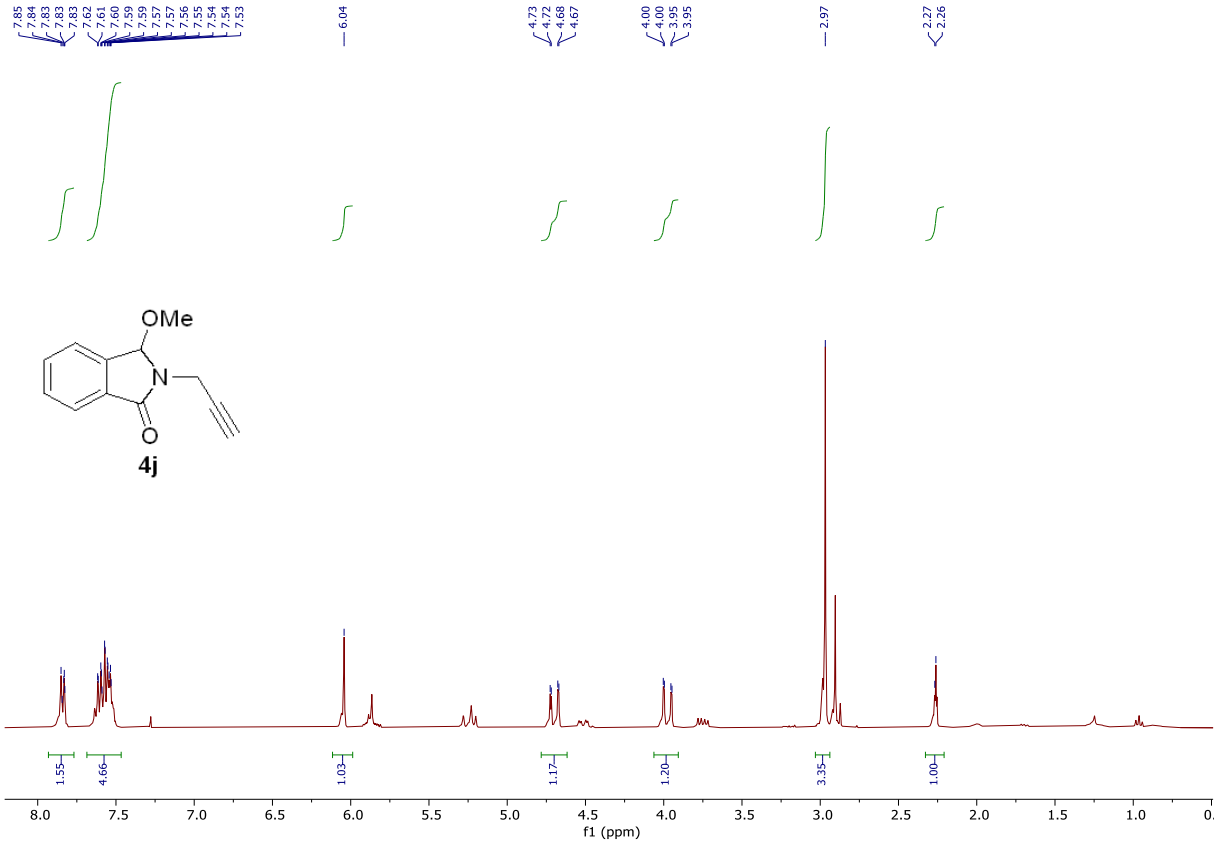
76.71
73.21
49.75
29.36
3.48
OMe
N
O
Me
4i
f1 (ppm)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7.83
7.83
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7.60
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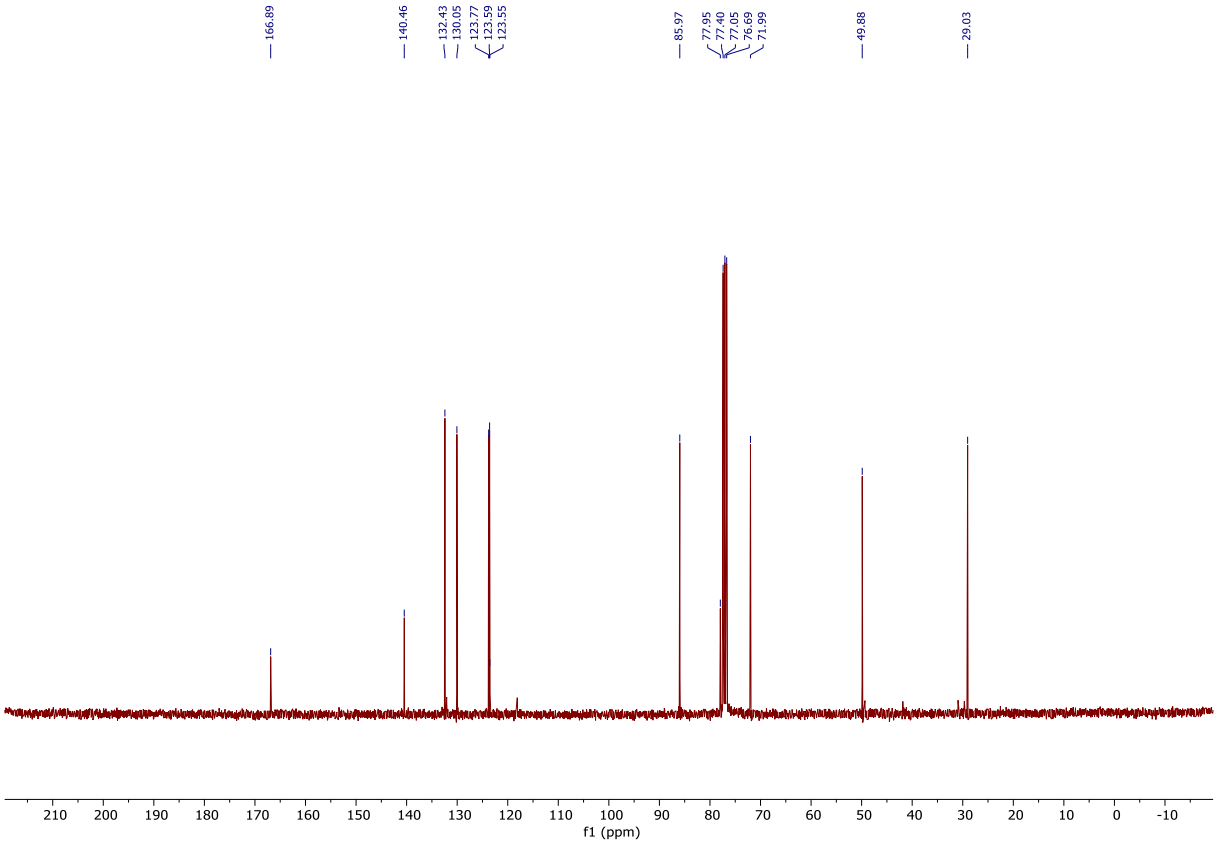
7.59
7.57
7.57
7.56
7.55
7.54
7.54
7.53
6.04
4.73
4.72
4.68
4.67
4.00
4.00
3.95
3.95
2.97
2.27
2.26
OMe
N
O
4j
1.55
4.66
1.03
1.17
1.20
3.35
1.00
f1 (ppm)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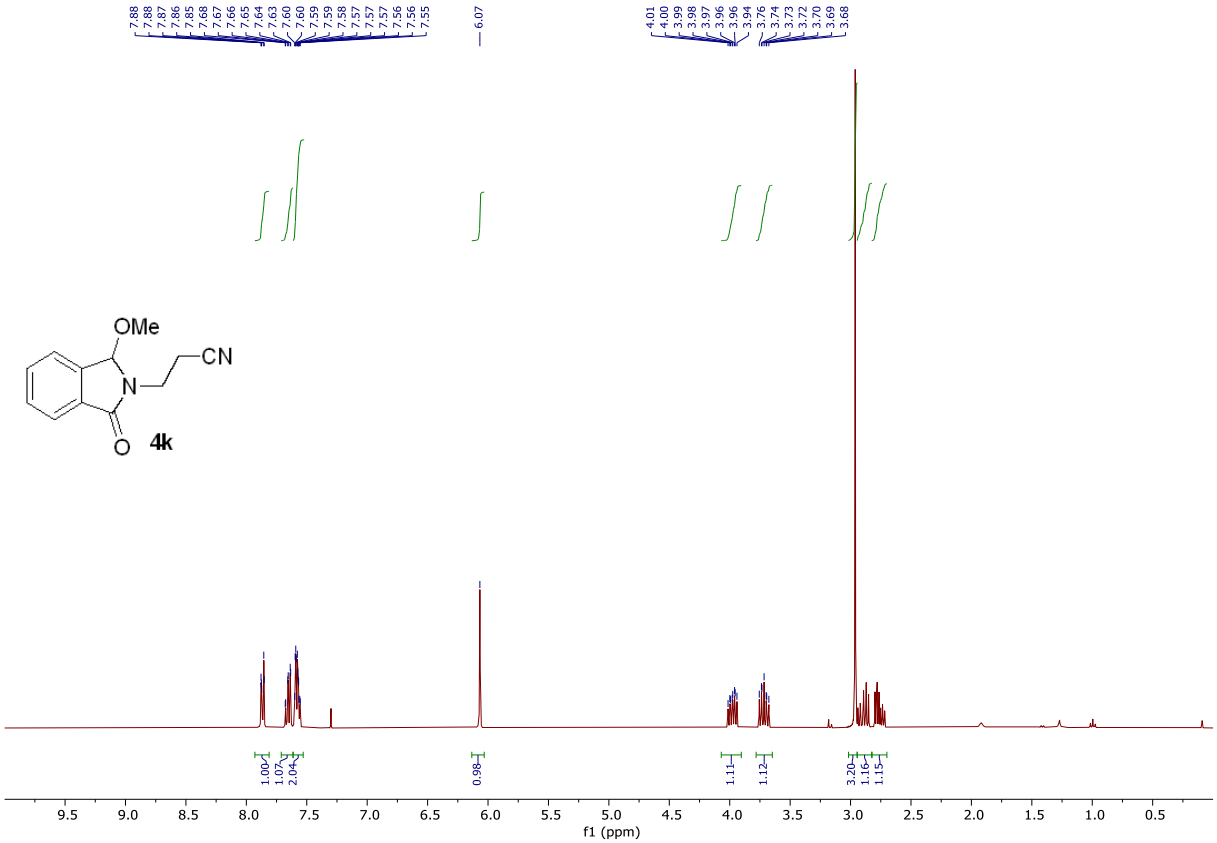
77.05
76.69
71.99
49.88
29.03
f1 (ppm)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7.87
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7.56
7.55
6.07
4.01
4.00
3.99
3.98
3.97
3.96
3.96
3.94
3.76
3.74
3.73
3.72
3.70
3.69
3.68
OMe
CN
N
O
4k
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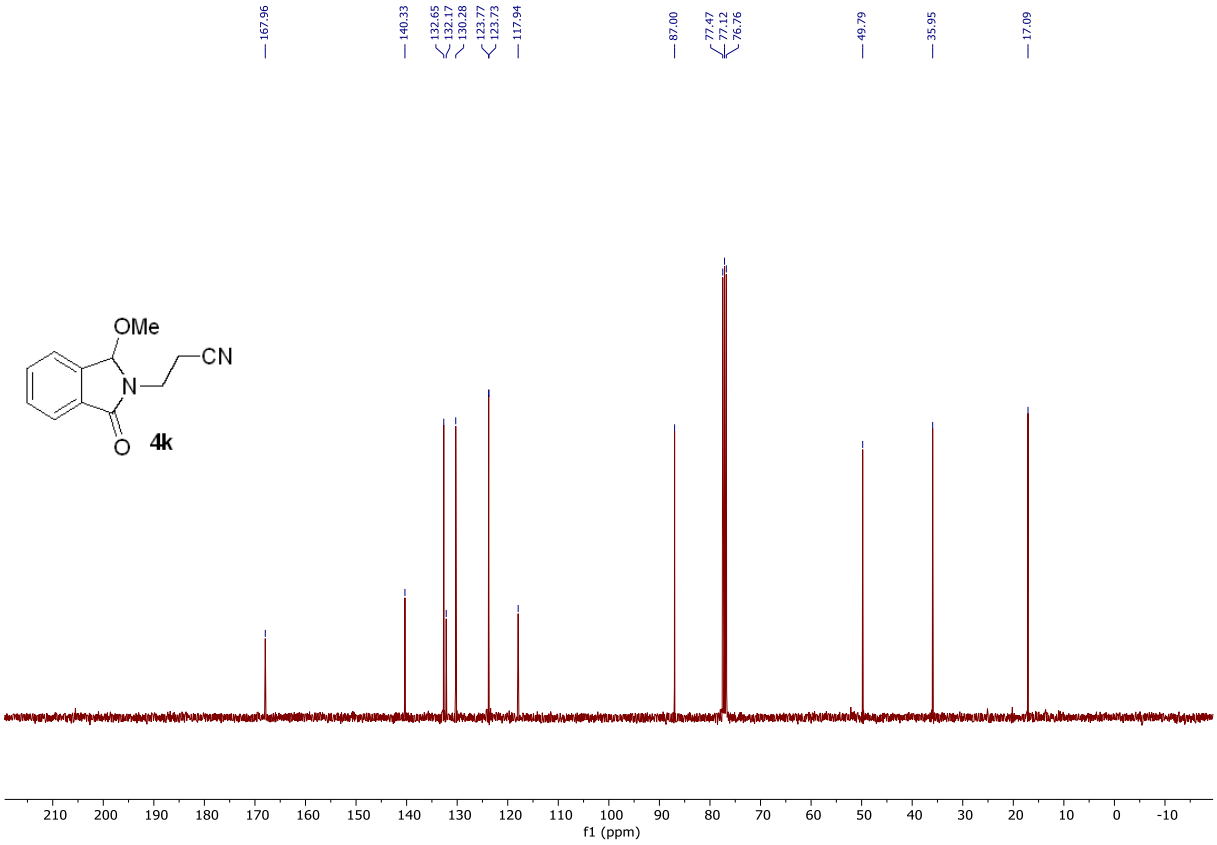
1.07
2.04
0.98
1.11
1.12
3.20
1.16
1.15
f1 (ppm)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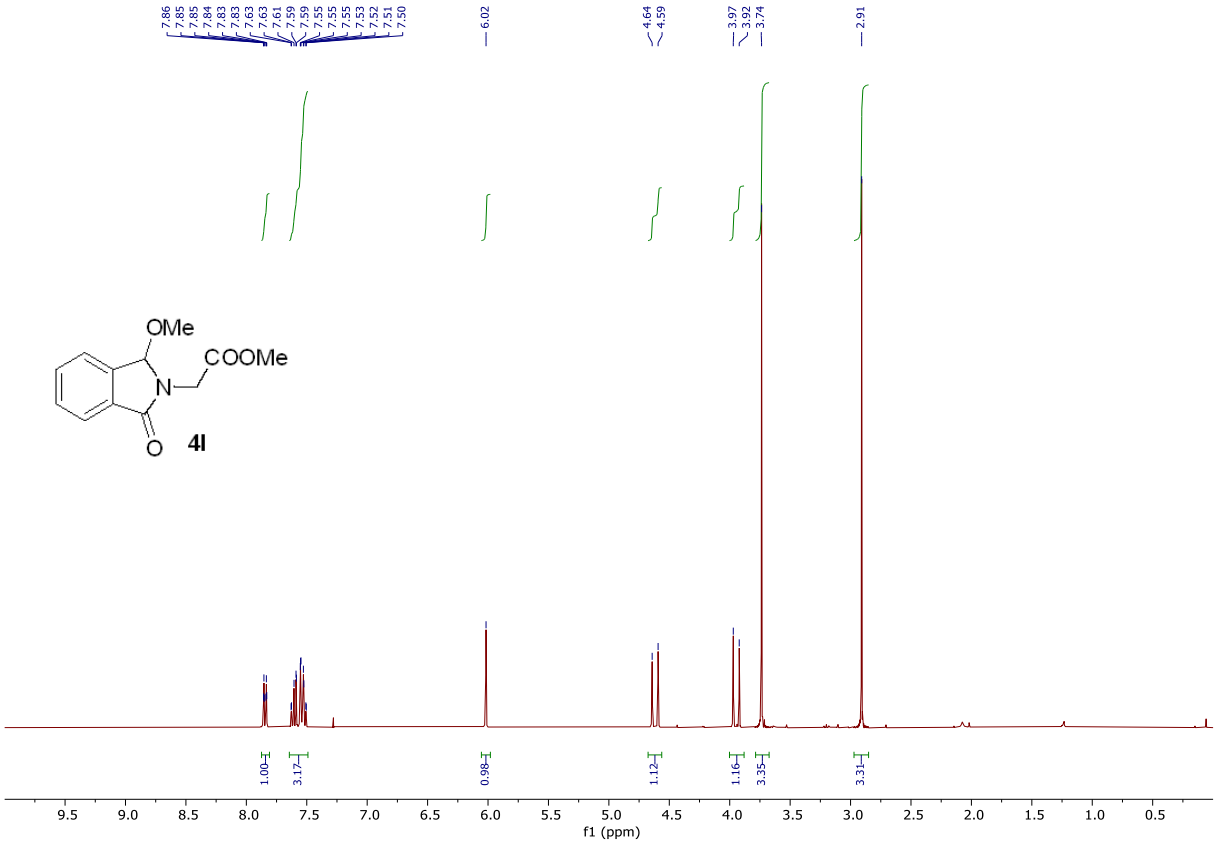
140.33
132.65
132.17
130.28
123.77
123.73
117.94
87.00
77.47
77.12
76.76
49.79
35.95
17.09
OMe
CN
N
O
4k
f1 (ppm)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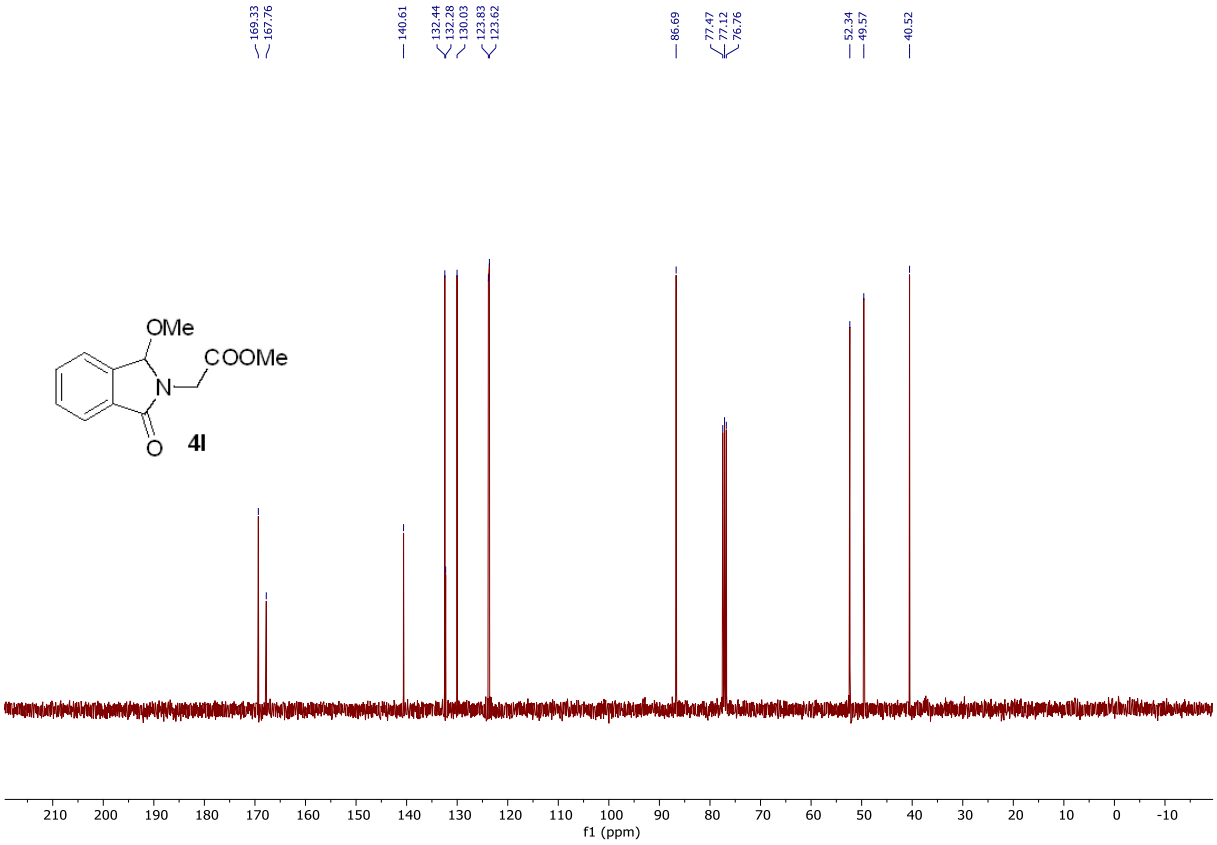
7.85
7.84
7.83
7.83
7.63
7.63
7.61
7.59
7.59
7.55
7.55
7.55
7.53
7.52
7.51
7.50
6.02
4.64
4.59
3.97
3.92
3.74
2.91
OMe
COOMe
N
O
4l
1.00
3.17
0.98
1.12
1.16
3.35
3.31
f1 (ppm)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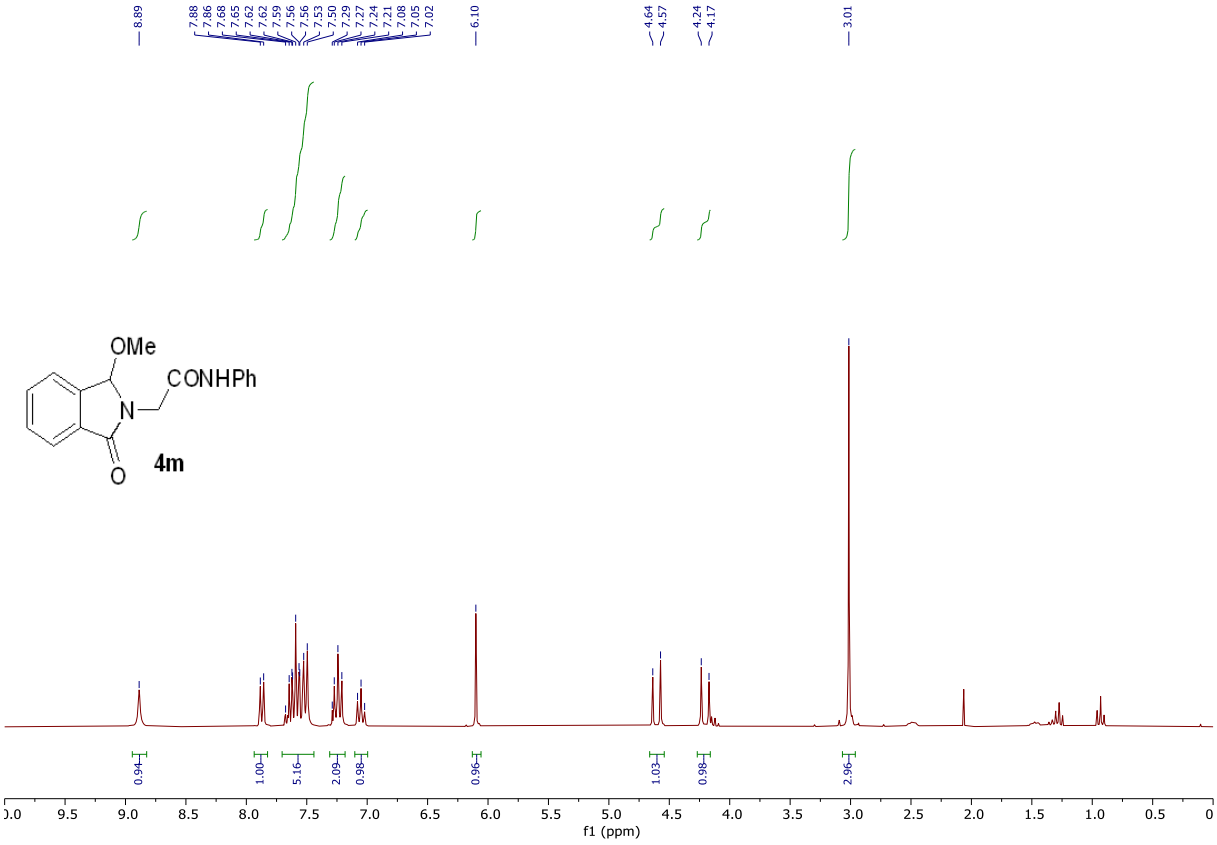
167.76
140.61
132.44
132.28
130.03
123.83
123.62
86.69
77.47
77.12
76.76
52.34
49.57
40.52
OMe
COOMe
N
O
4l
f1 (ppm)


8.89
7.88
7.86
7.68
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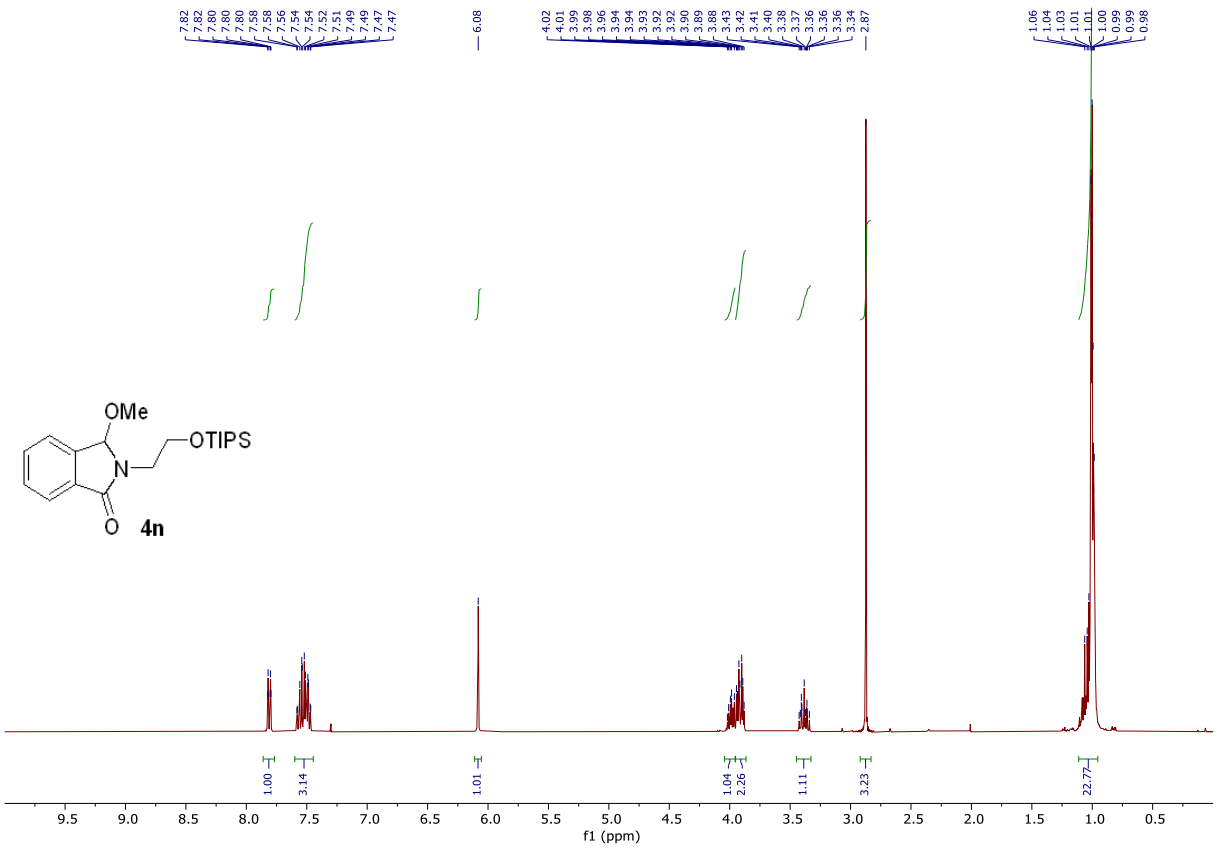
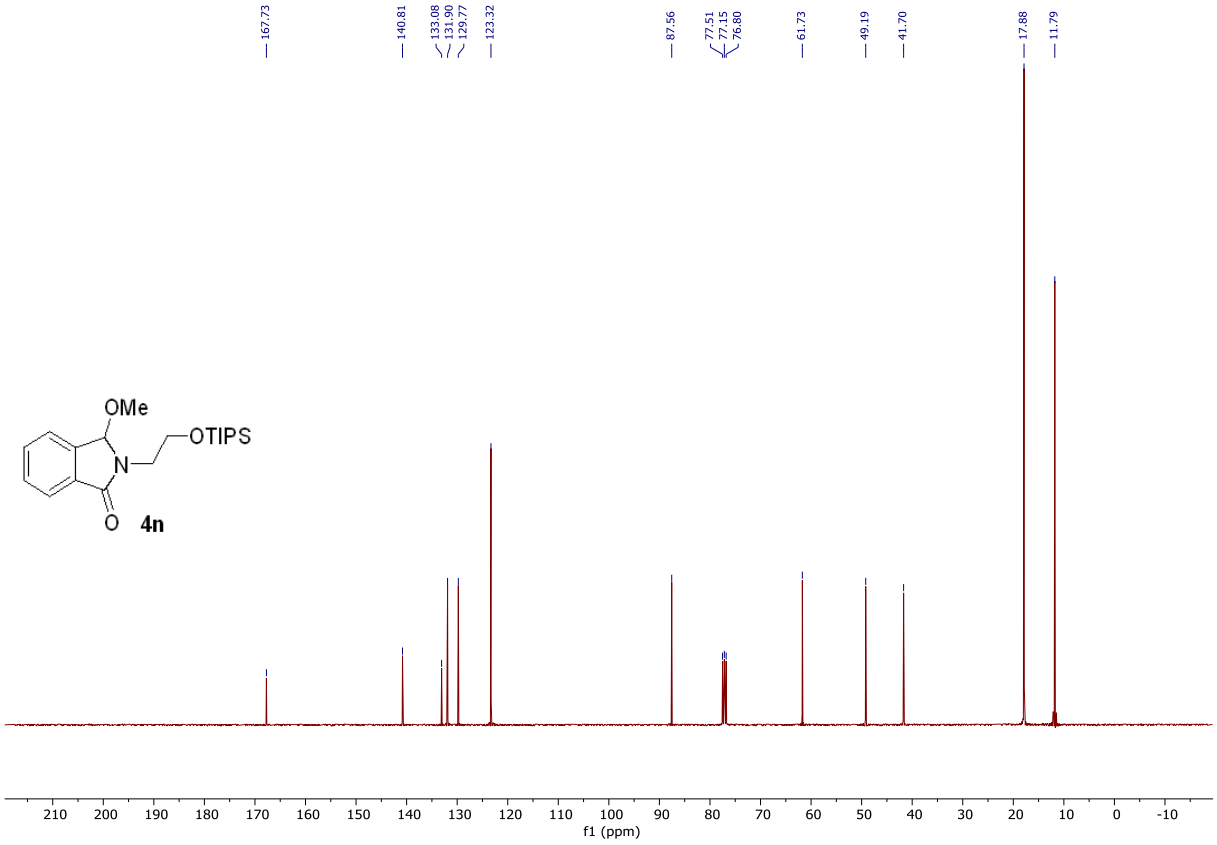
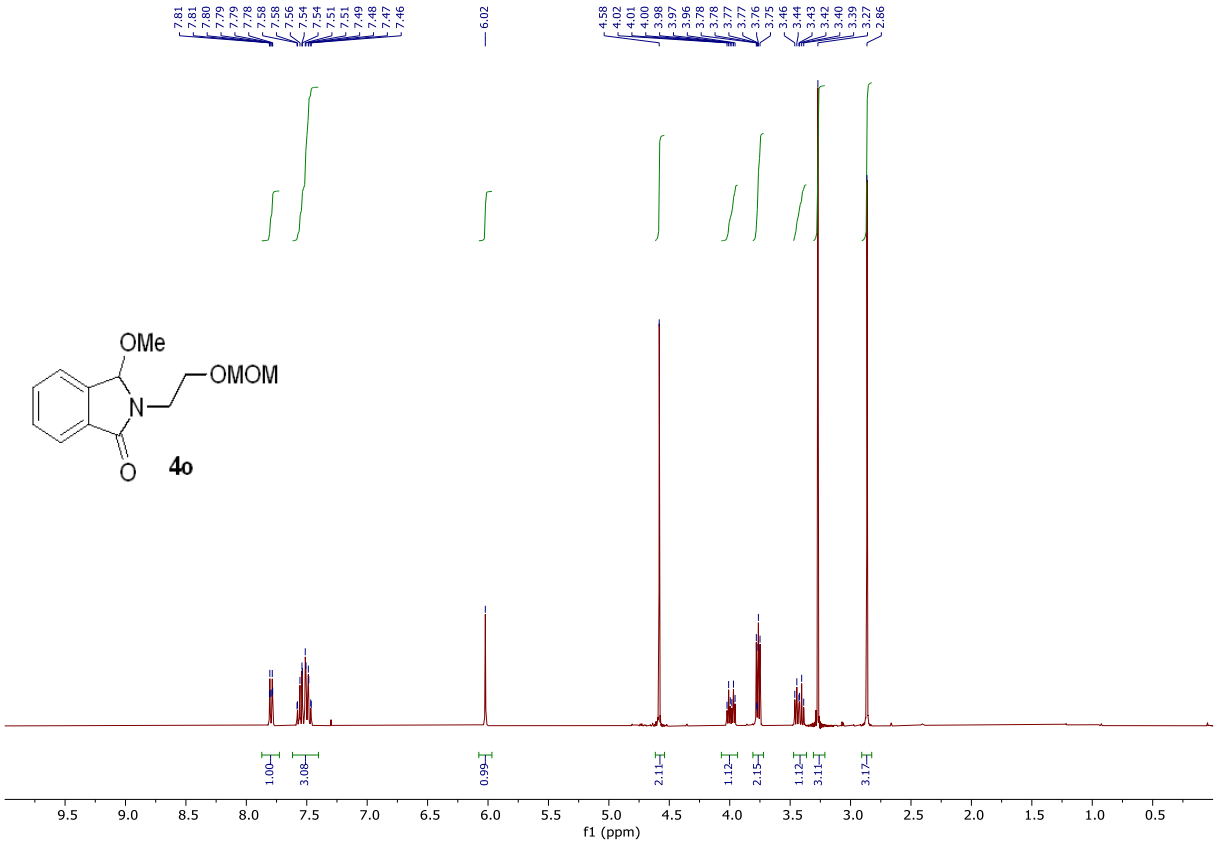
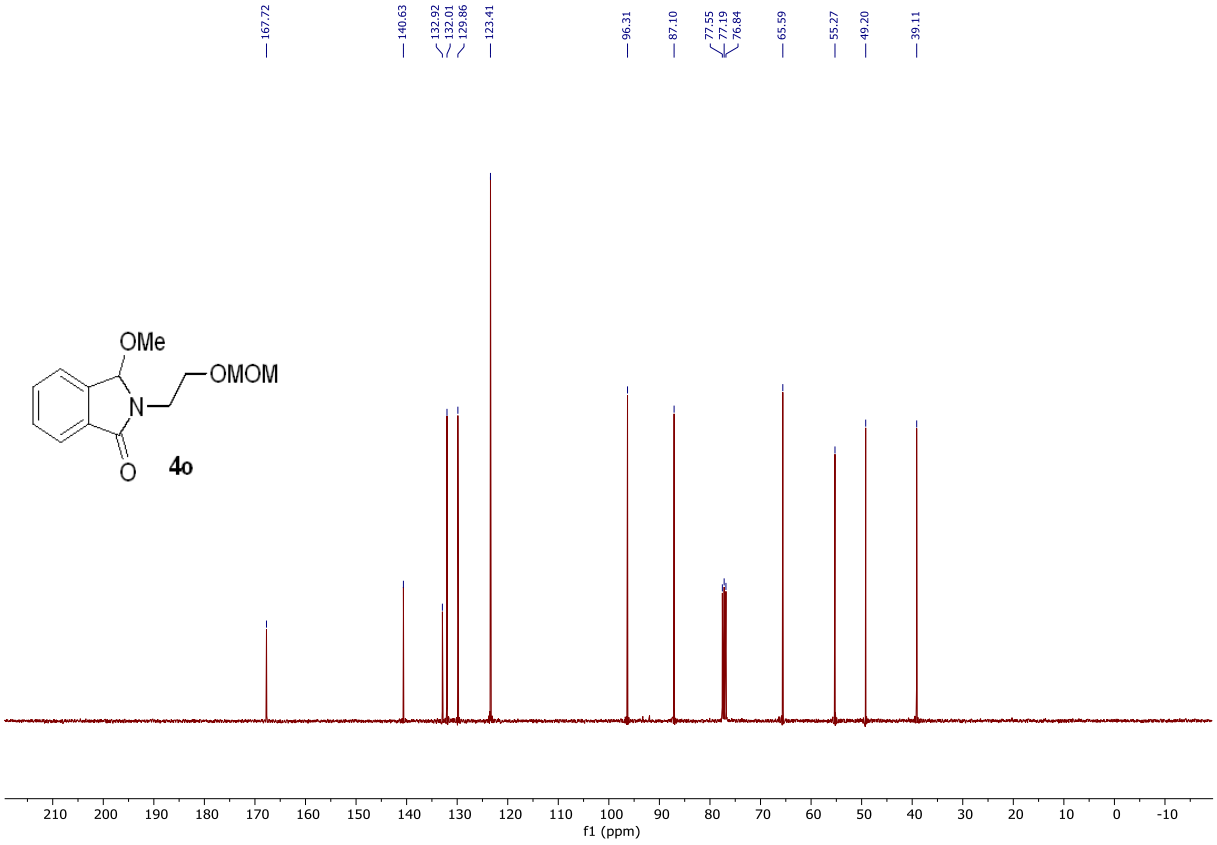
7.62
7.62
7.59
7.56
7.56
7.53
7.50
7.29
7.27
7.24
7.21
7.08
7.05
7.02
6.10
4.64
4.57
4.24
4.17
3.01
OMe
CONHPh
N
O
4m
0.94
1.00
5.16
2.09
0.98
0.96
1.03
0.98
2.96
f1 (ppm)


OMe
OTIPS
N
O
4n
f1 (ppm)


OMe
OTIPS
N
O
4n
f1 (ppm)


7.81
7.81
7.80
7.79
7.79
7.78
7.58
7.56
7.54
7.54
7.51
7.51
7.49
7.48
7.47
7.46
6.02
4.58
4.02
4.01
4.00
3.98
3.97
3.96
3.78
3.78
3.77
3.77
3.76
3.75
3.46
3.44
3.43
3.42
3.40
3.39
3.27
2.86
OMe
OMOM
N
O
4o
1.00
3.08
0.99
2.11
1.12
2.15
1.12
3.11
3.17
f1 (ppm)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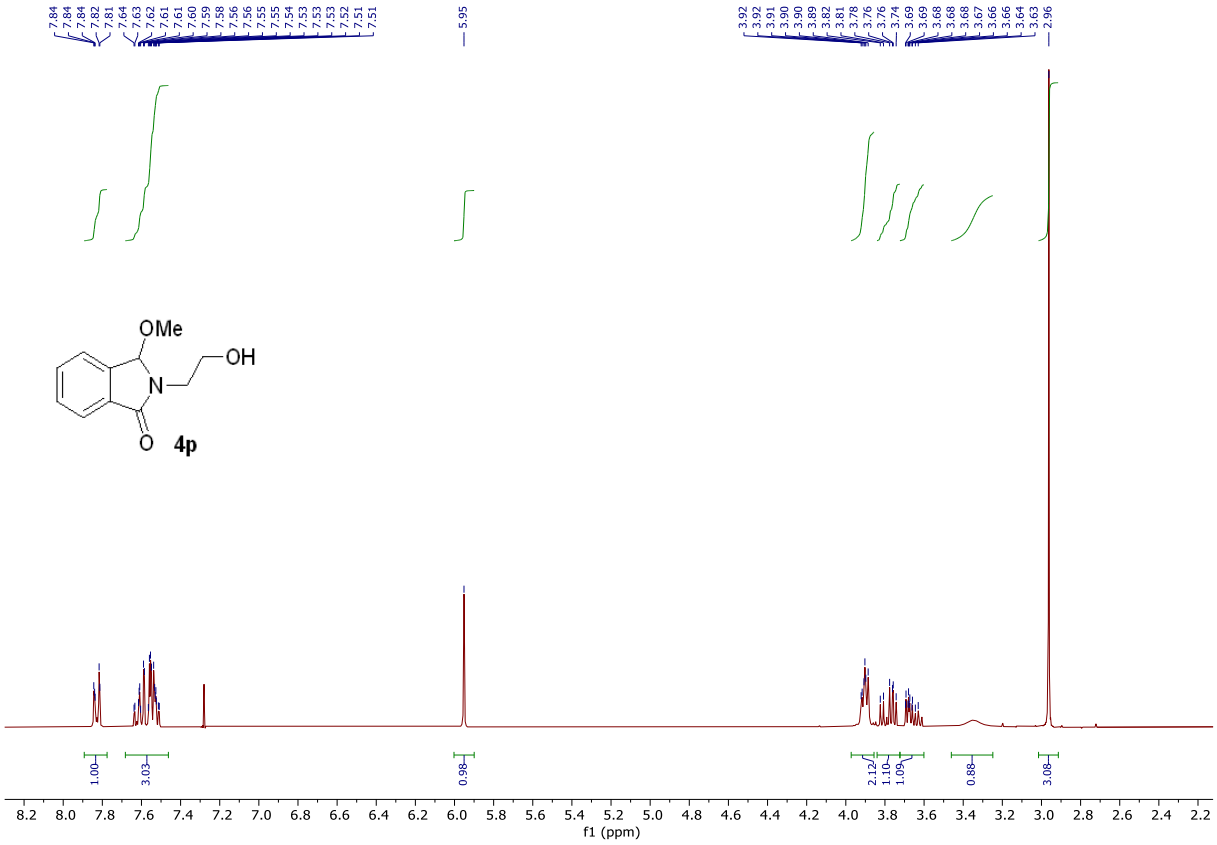
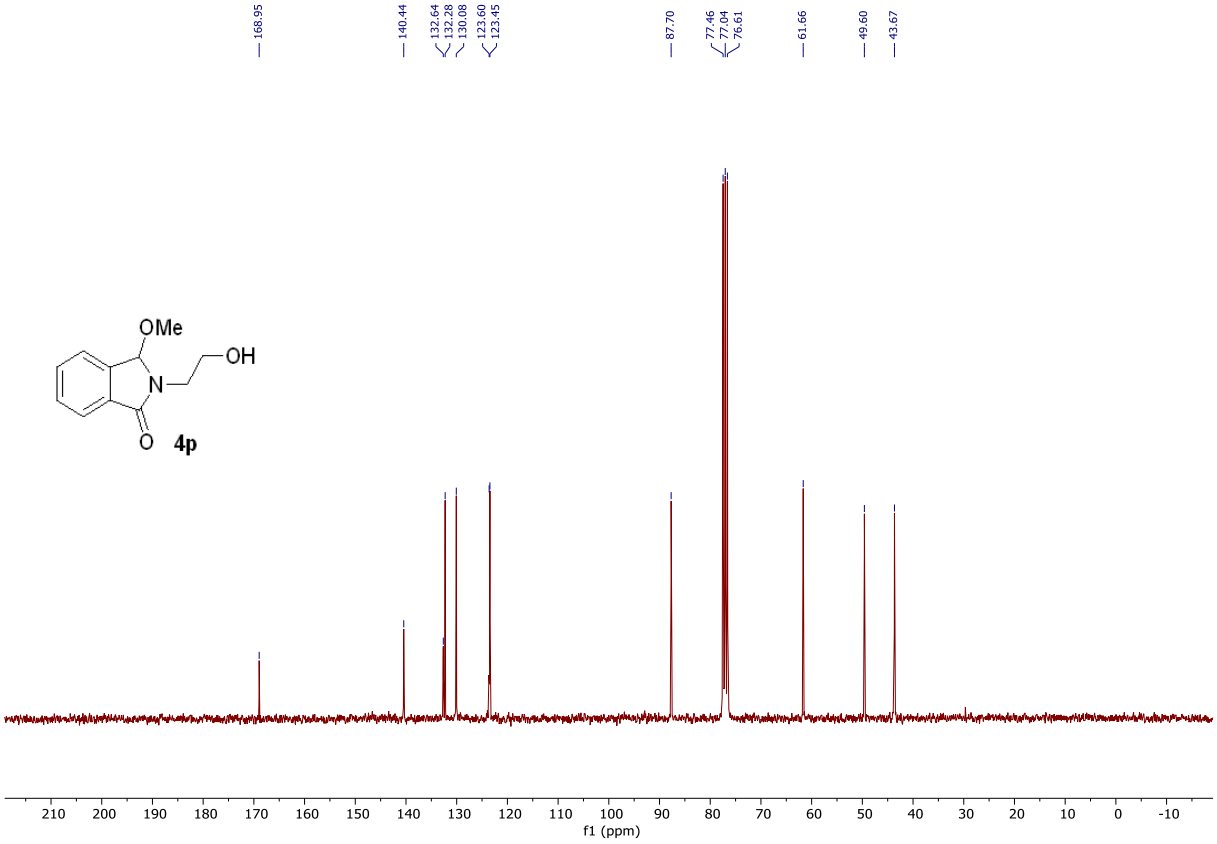
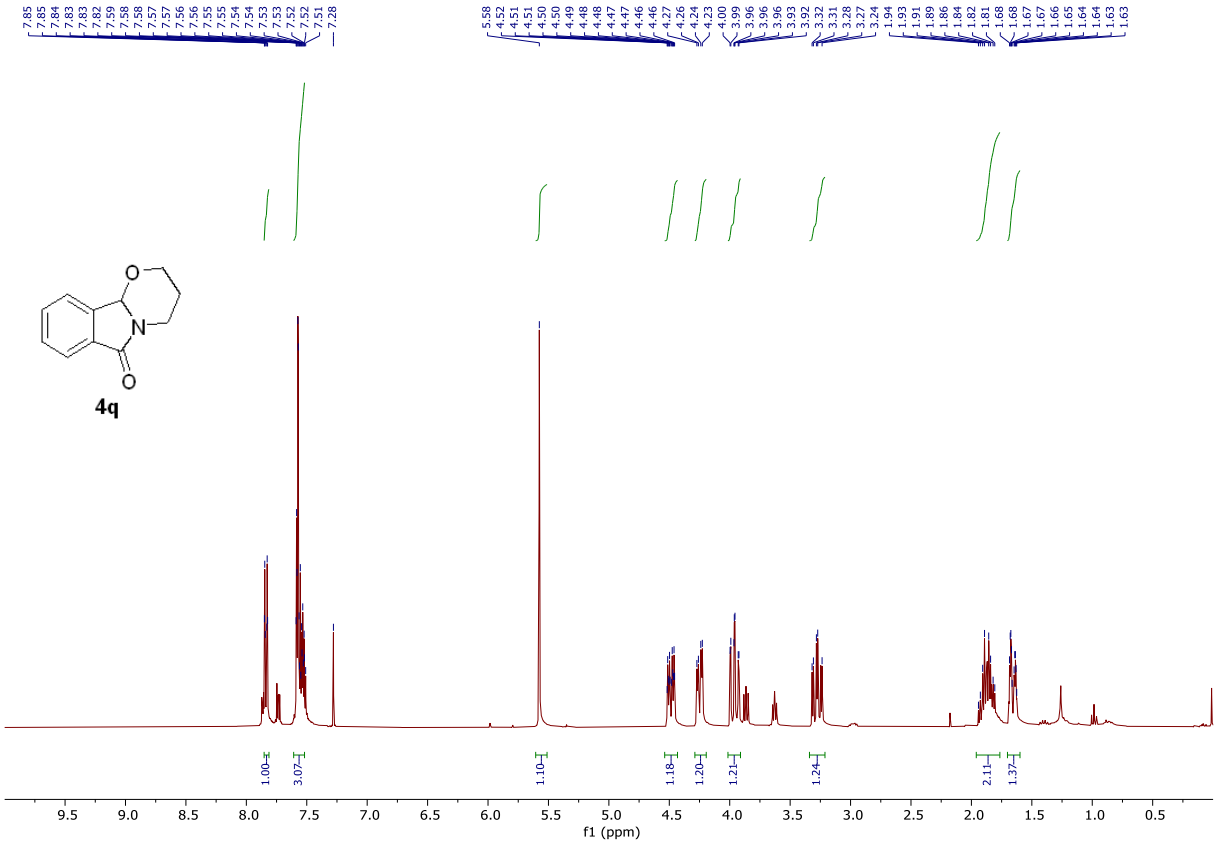
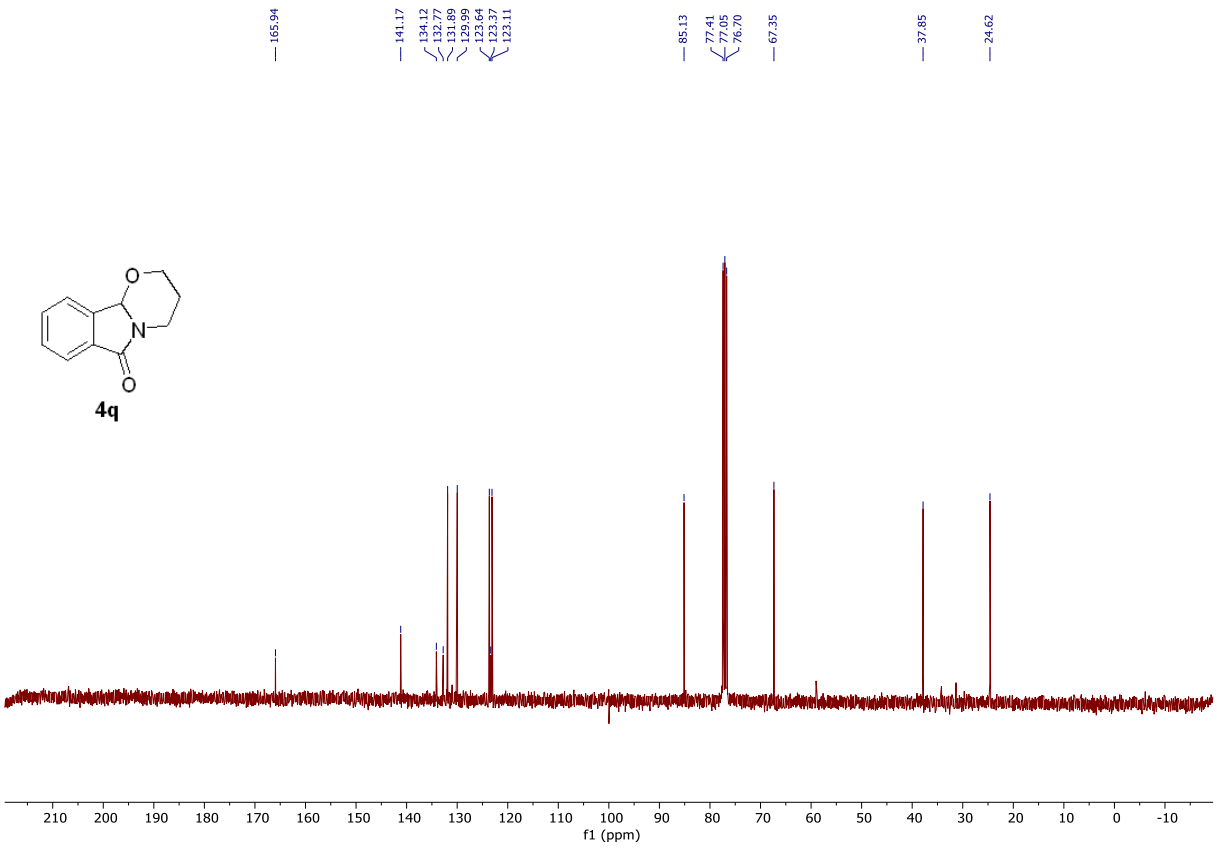
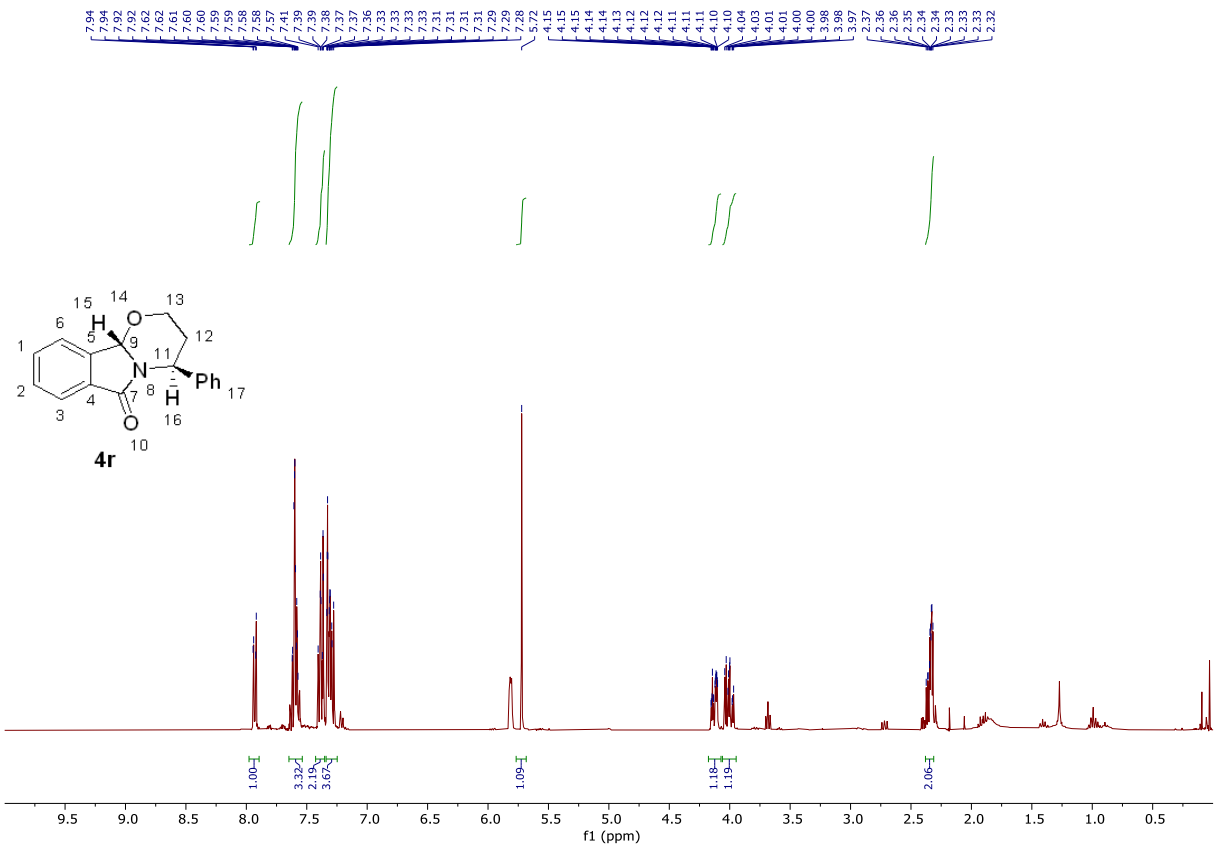
140.63
132.92
132.01
129.86
123.41
96.31
87.10
77.55
77.19
76.84
65.59
55.27
49.20
39.11
OMe
OMOM
N
O
4o
f1 (ppm)

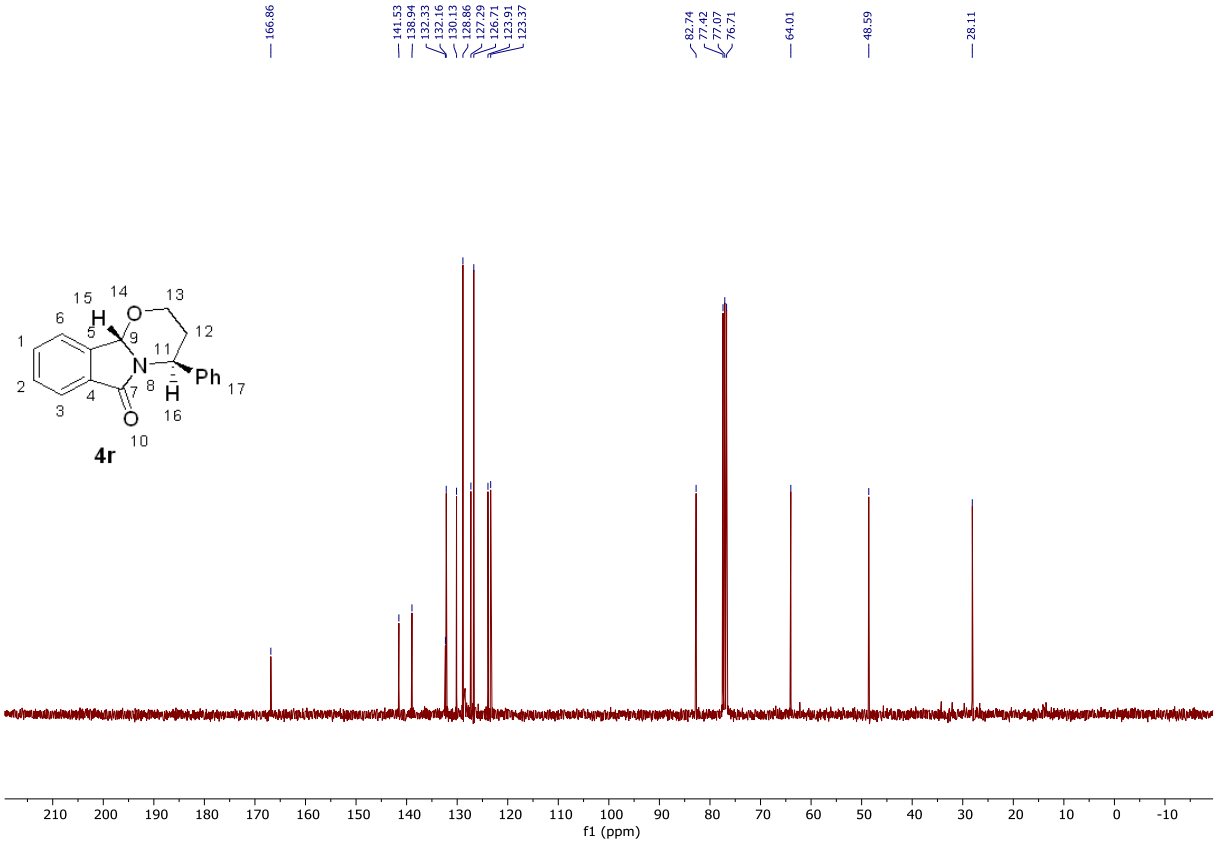

OMe
OH
N
O
4p
f1 (ppm)

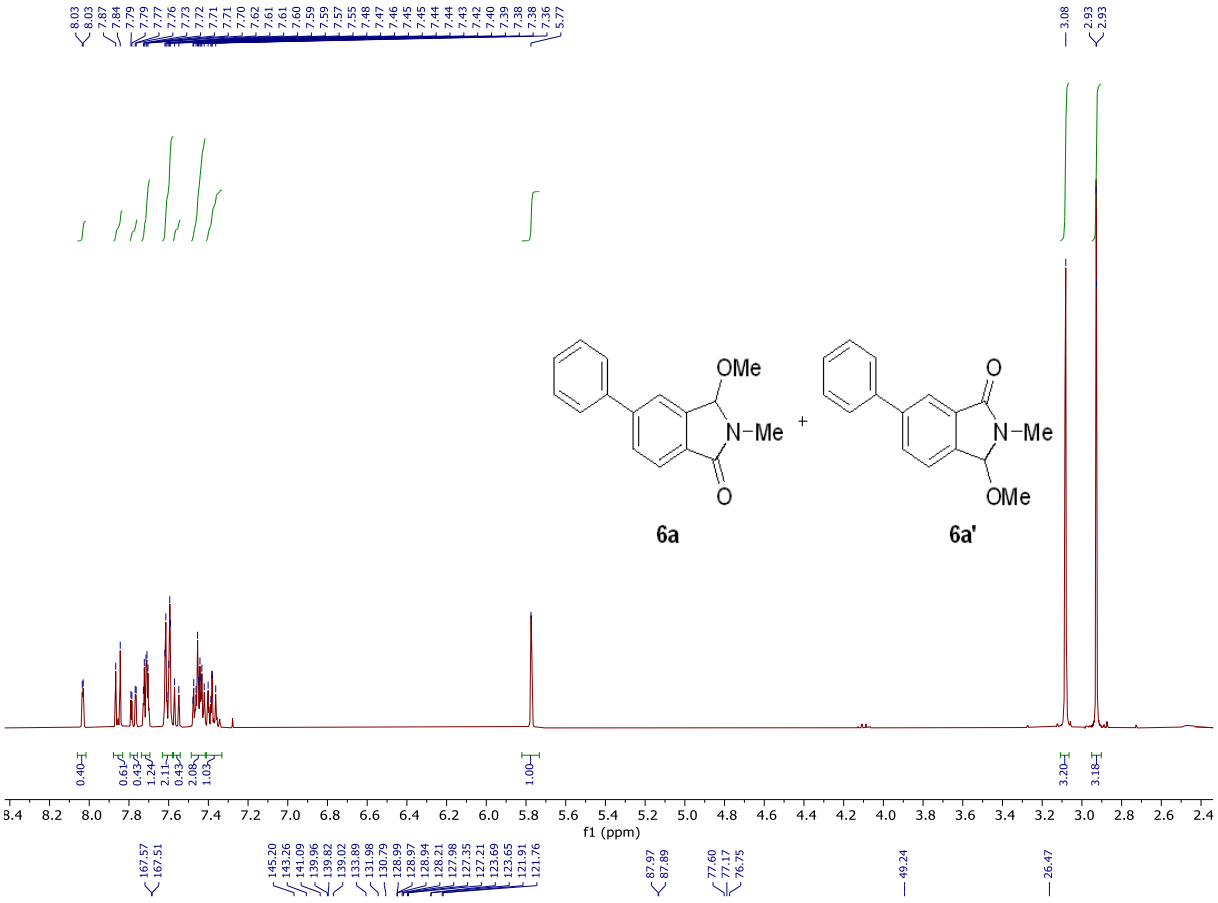

OMe
OH
N
O
4p
f1 (ppm)

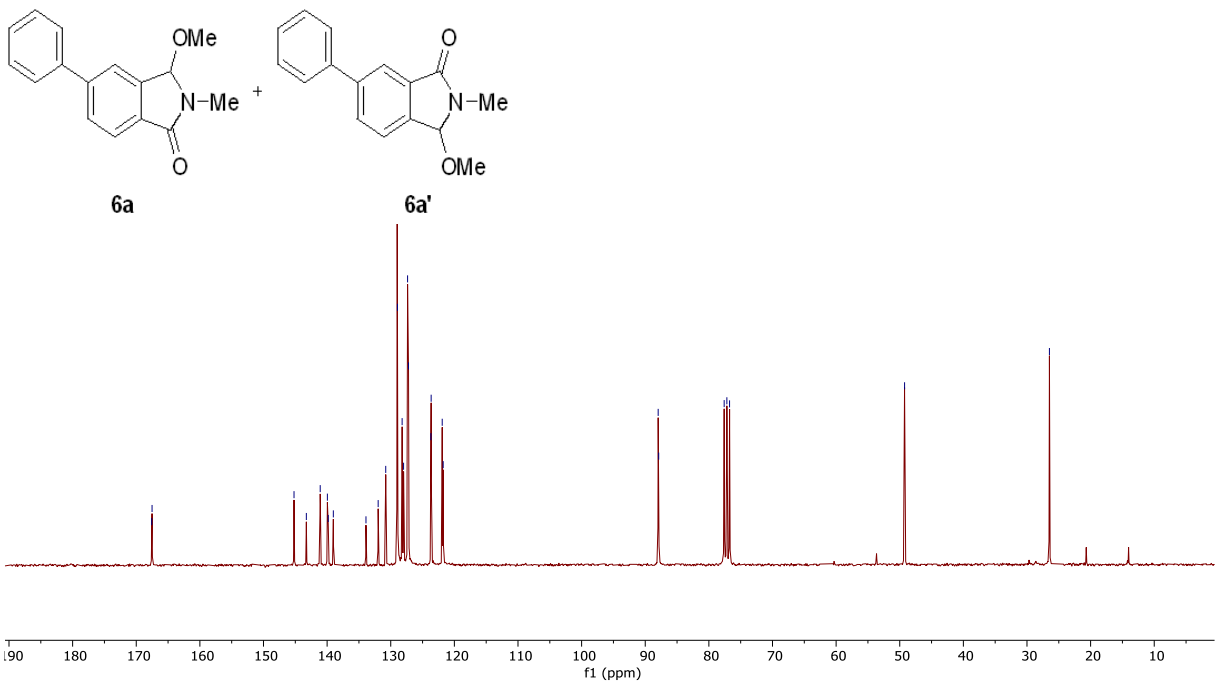

7.85
7.85
7.84
7.83
7.83
7.82
7.59
7.58
7.58
7.57
7.57
7.56
7.56
7.55
7.55
7.54
7.54
7.53
7.53
7.52
7.52
7.51
7.28
5.58
4.52
4.51
4.51
4.50
4.50
4.49
4.48
4.48
4.47
4.47
4.46
4.46
4.27
4.26
4.24
4.23
4.00
3.99
3.96
3.96
3.96
3.93
3.92
3.32
3.31
3.28
3.27
3.24
1.94
1.93
1.91
1.89
1.86
1.84
1.82
1.81
1.68
1.68
1.67
1.67
1.66
1.65
1.64
1.64
1.63
1.63
O
N
O
4q
1.00
3.07
1.10
1.18
1.20
1.21
1.24
2.11
1.37
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)

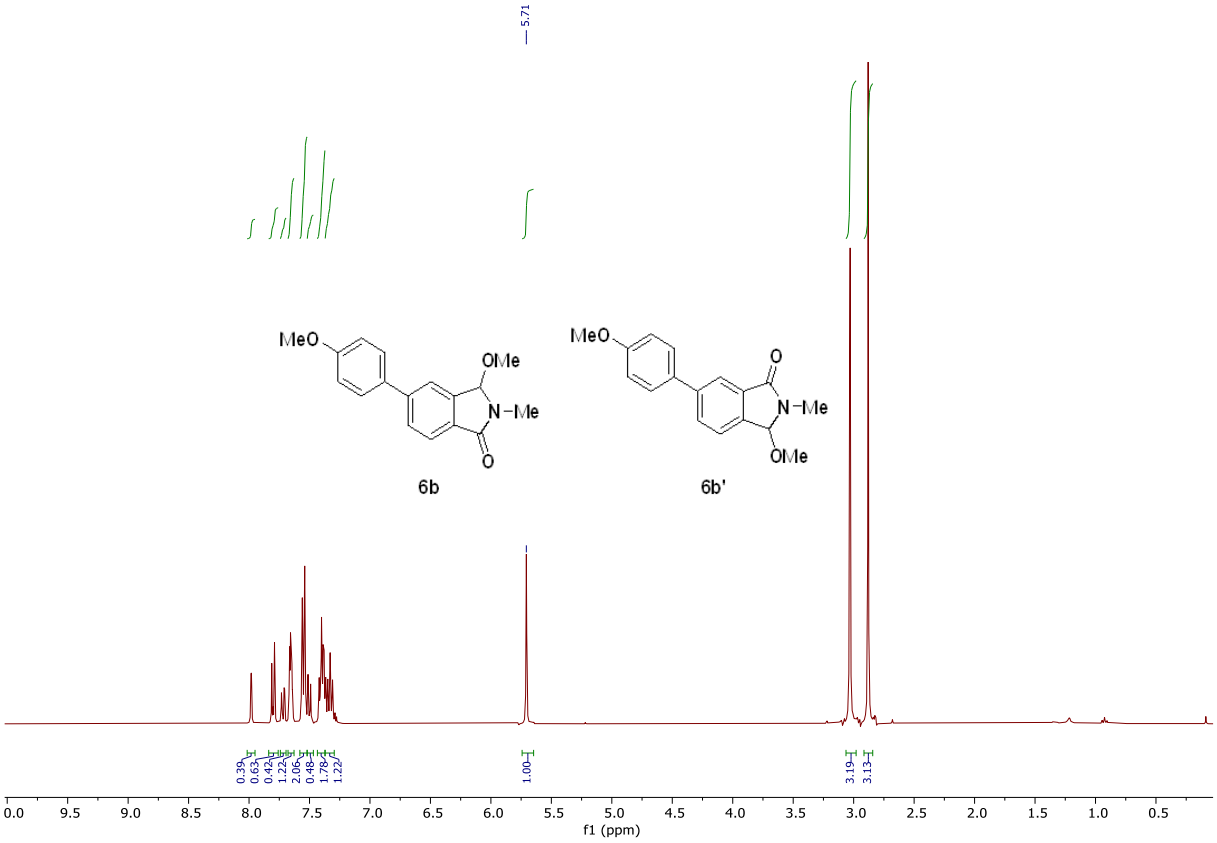

165.94
141.17
134.12
132.77
131.89
129.99
123.64
123.37
123.11
85.13
77.41
77.05
76.70
67.35
37.85
24.62
O
N
O
4q
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

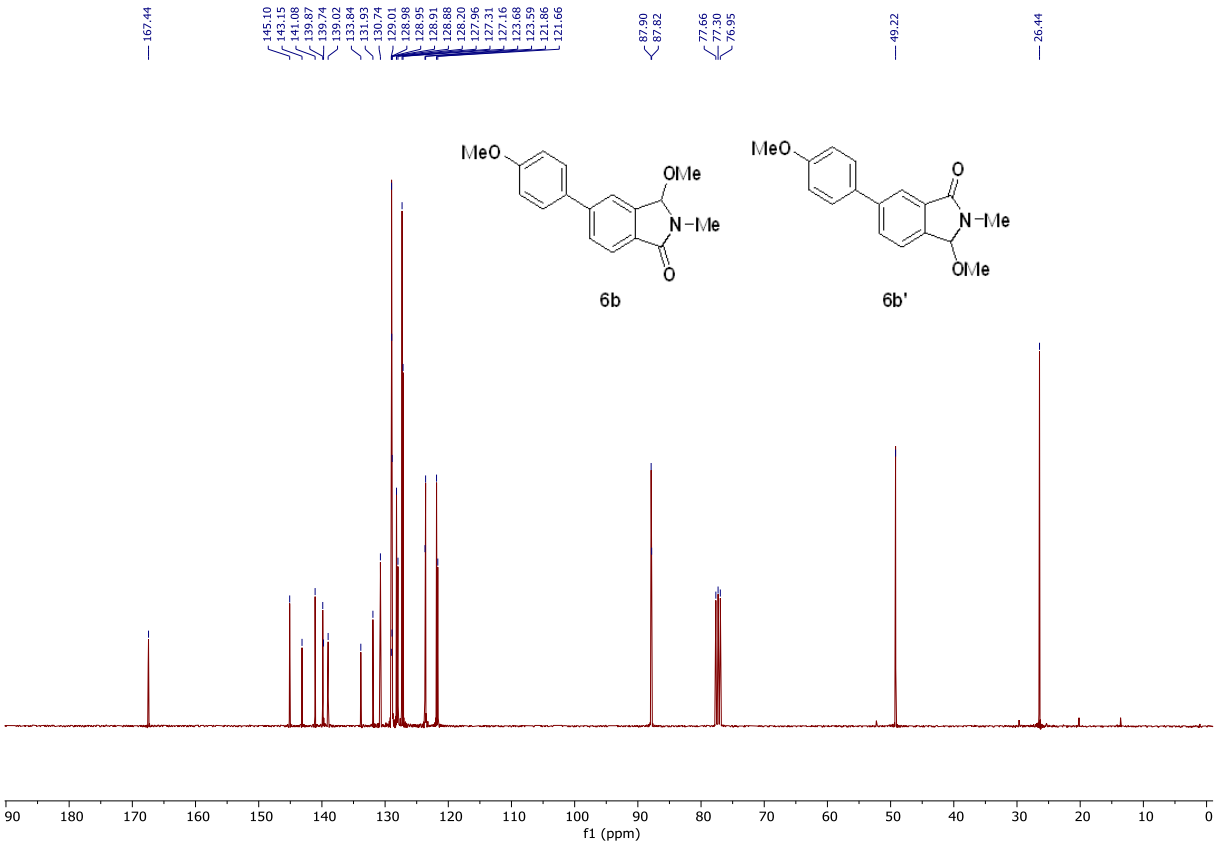

4r
f1 (ppm)

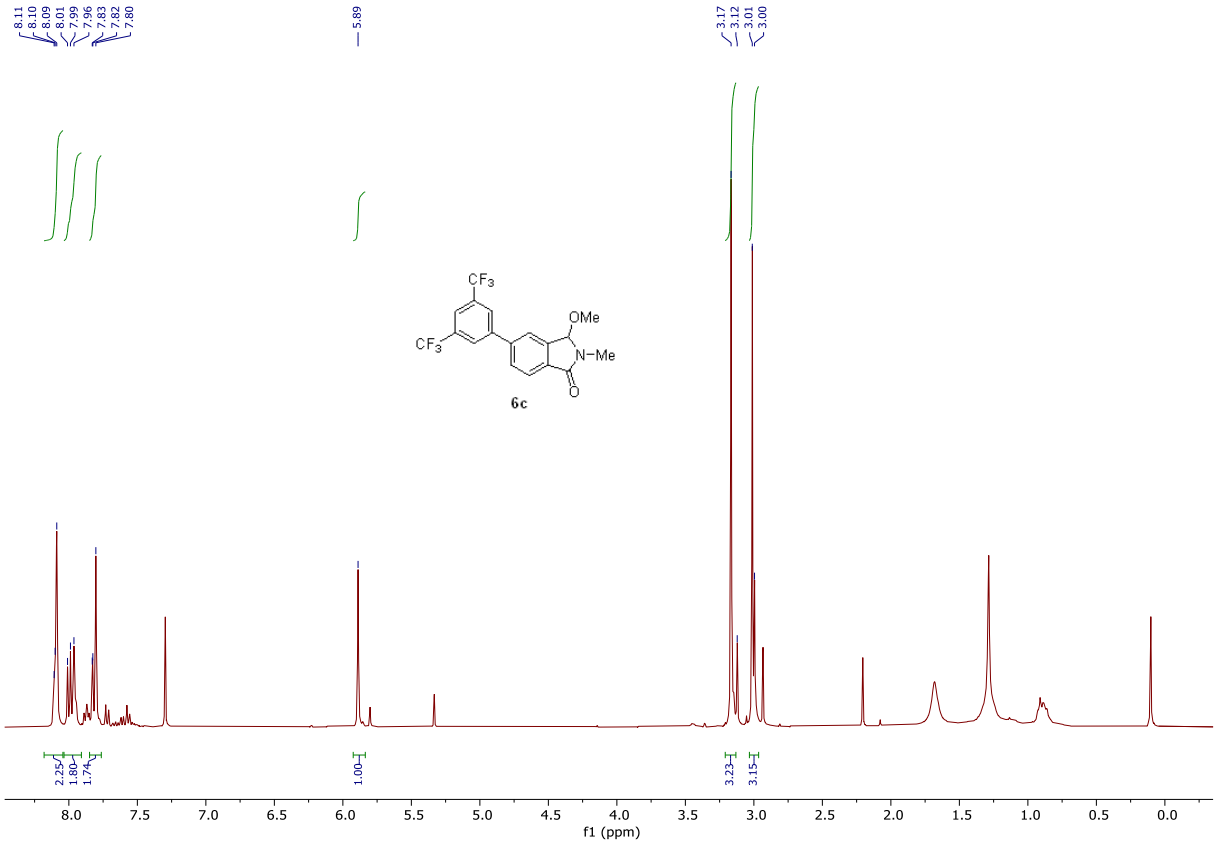

4r
f1 (ppm)

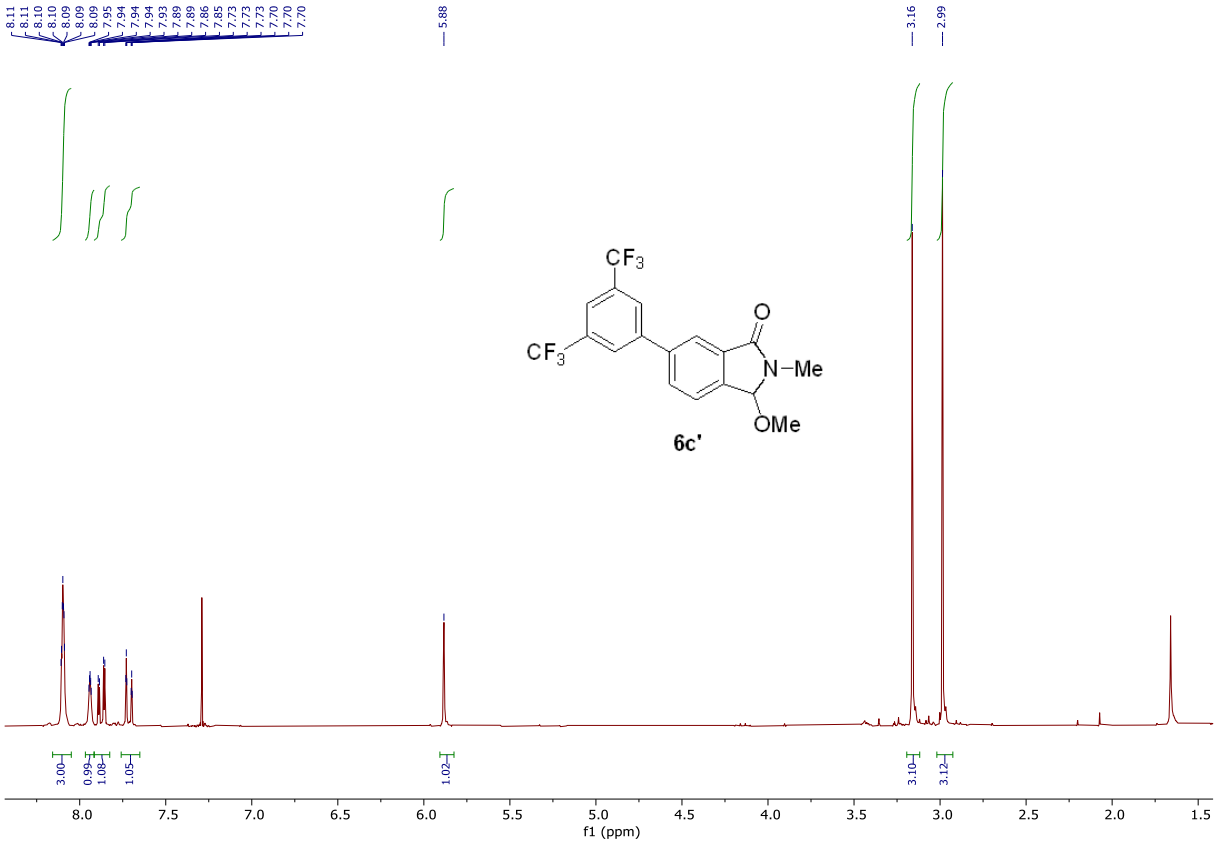

OMe
N–Me
O
6a
O
N–Me
OMe
6a'
f1 (ppm)

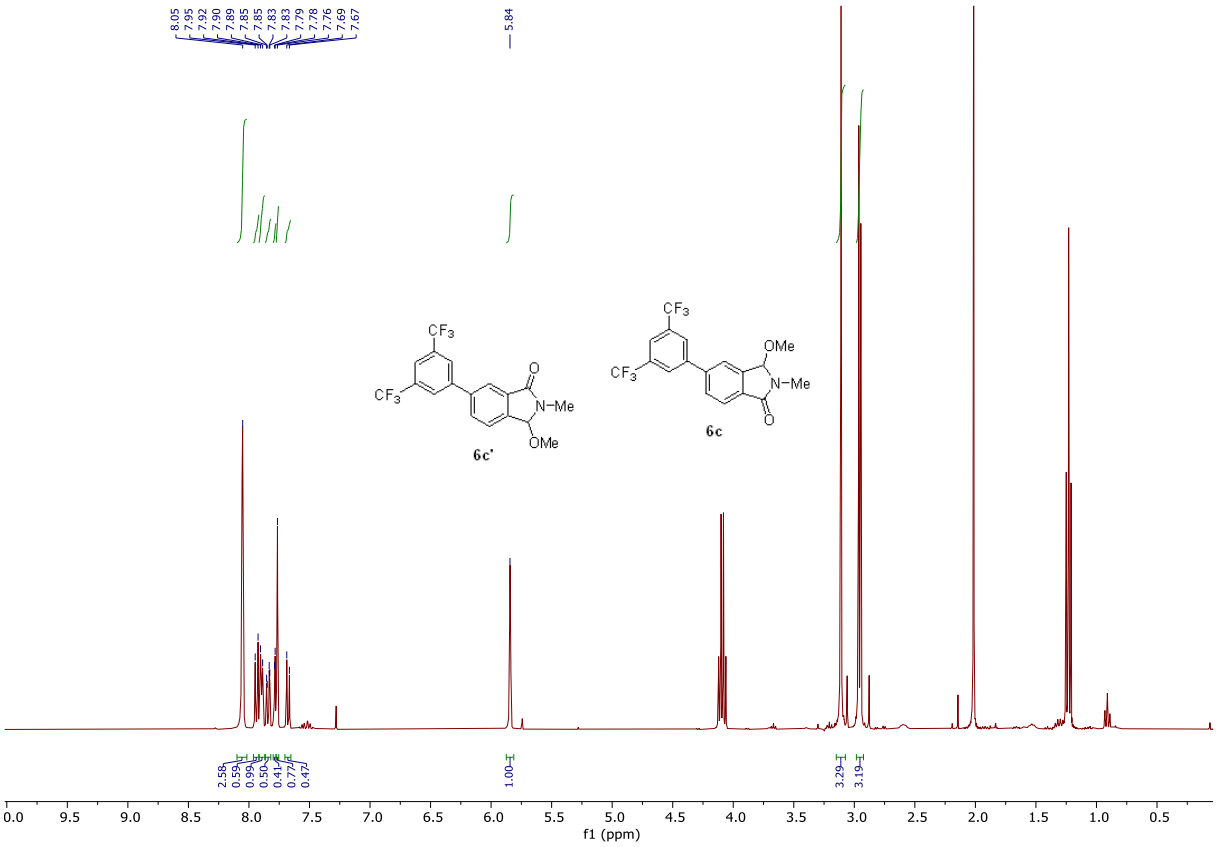

OMe
N–Me
O
6a
O
N–Me
OMe
6a'
f1 (ppm)

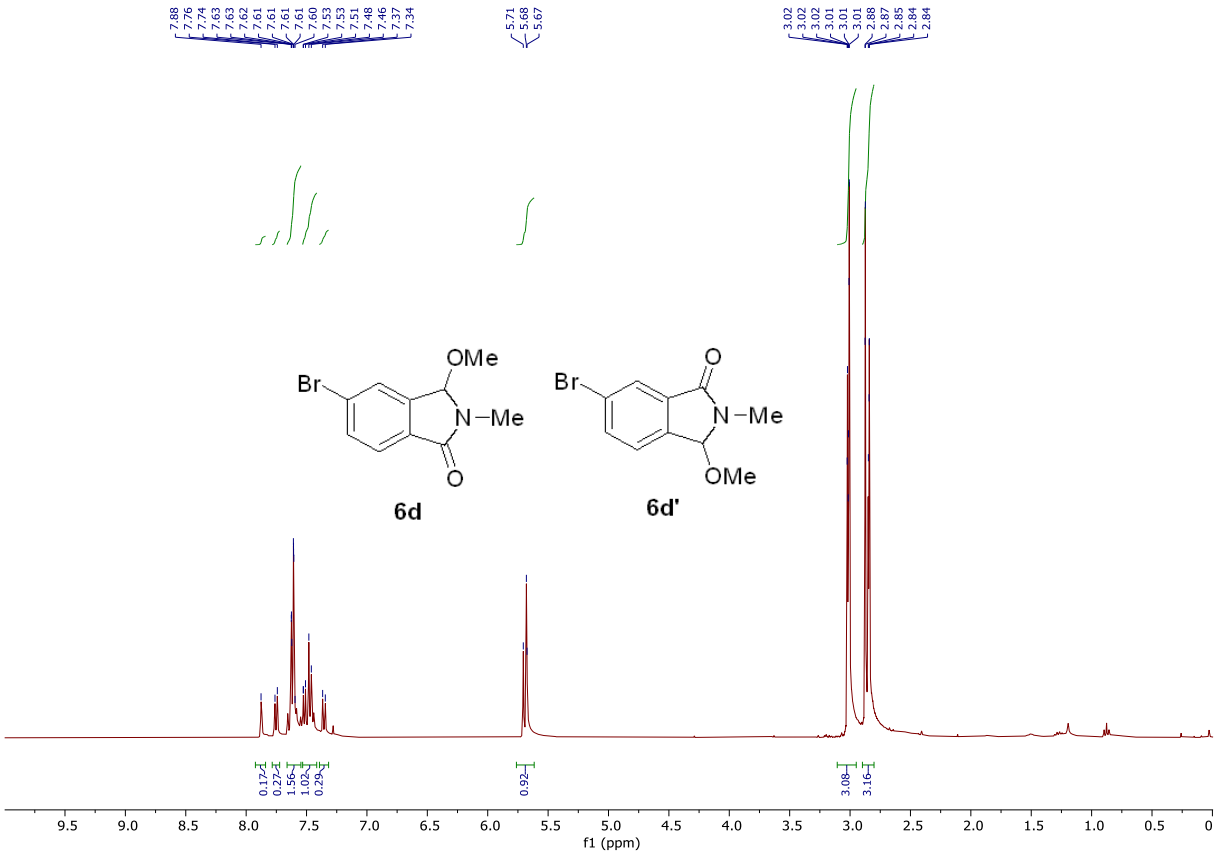

5.71
MeO
OMe
N–Me
O
6b
MeO
O
N–Me
OMe
6b'
0.39
0.63
0.42
1.22
2.06
0.48
1.78
1.22
1.00
3.19
3.13
0.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)

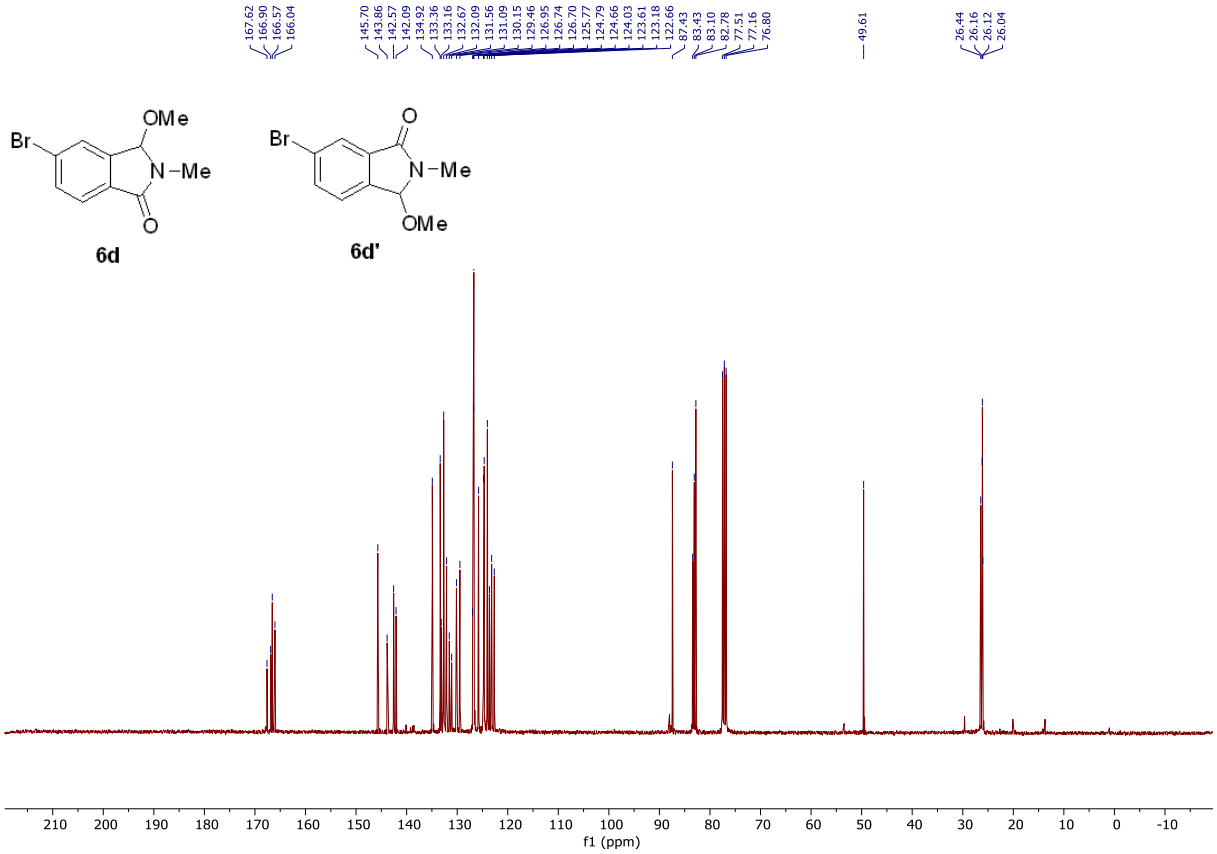

167.44
145.10
143.15
141.08
139.87
139.74
139.02
133.84
131.93
130.74
129.01
128.98
128.95
128.91
128.88
128.20
127.96
127.31
127.16
123.68
123.59
121.86
121.66
87.90
87.82
77.66
77.30
76.95
49.22
26.44
MeO
OMe
N–Me
O
6b
MeO
O
N–Me
OMe
6b'
90 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

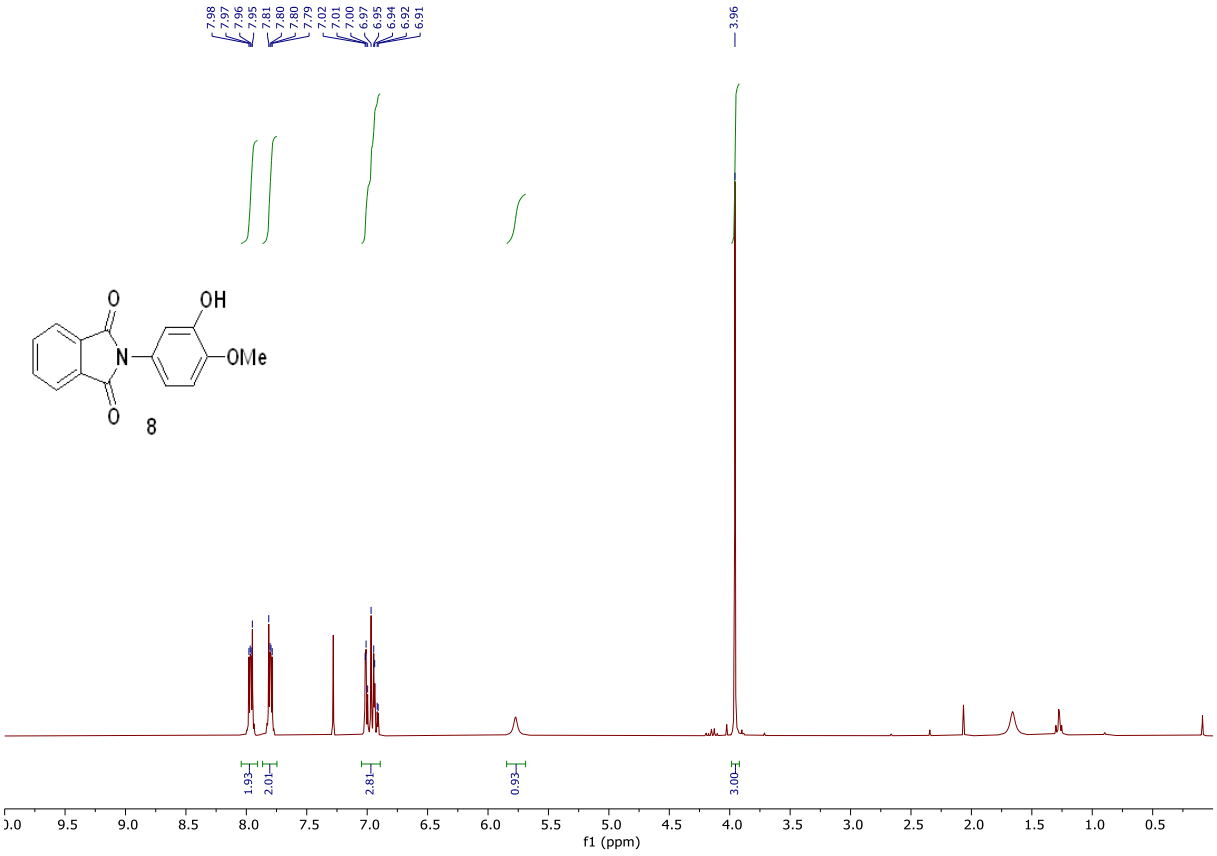

8.11
8.10
8.09
8.01
7.99
7.96
7.83
7.82
7.80
5.89
3.17
3.12
3.01
3.00
CF3
CF3
OMe
N–Me
O
6c
2.25
1.80
1.74
1.00
3.23
3.15
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


8.11
8.11
8.10
8.10
8.09
8.09
8.09
7.95
7.94
7.94
7.93
7.89
7.89
7.86
7.85
7.73
7.73
7.73
7.70
7.70
7.70
5.88
3.16
2.99
CF3
CF3
O
N–Me
OMe
6c'
3.00
0.99
1.08
1.05
1.02
3.10
3.12
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
f1 (ppm)


8.05
7.95
7.92
7.90
7.89
7.85
7.85
7.83
7.83
7.79
7.78
7.76
7.69
7.67
5.84
CF3
CF3
O
N–Me
OMe
6c'
CF3
CF3
OMe
N–Me
O
6c
2.58
0.59
0.99
0.50
0.41
0.77
0.47
1.00
3.29
3.19
0.0 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)


7.88
7.76
7.74
7.63
7.63
7.62
7.61
7.61
7.61
7.61
7.60
7.53
7.53
7.51
7.48
7.46
7.37
7.34
5.71
5.68
5.67
3.02
3.02
3.02
3.01
3.01
3.01
2.88
2.87
2.85
2.84
2.84
OMe
Br
N–Me
O
6d
O
Br
N–Me
OMe
6d'
0.17
0.27
1.56
1.02
0.29
0.92
3.08
3.16
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)


167.62
166.90
166.57
166.04
145.70
143.86
142.57
142.09
134.92
133.36
133.16
132.67
132.09
131.56
131.09
130.15
129.46
126.95
126.74
126.70
125.77
124.79
124.66
124.03
123.61
123.18
122.66
87.43
83.43
83.10
82.78
77.51
77.16
76.80
49.61
26.44
26.16
26.12
26.04
Br
OMe
N–Me
O
6d
Br
O
N–Me
OMe
6d'
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)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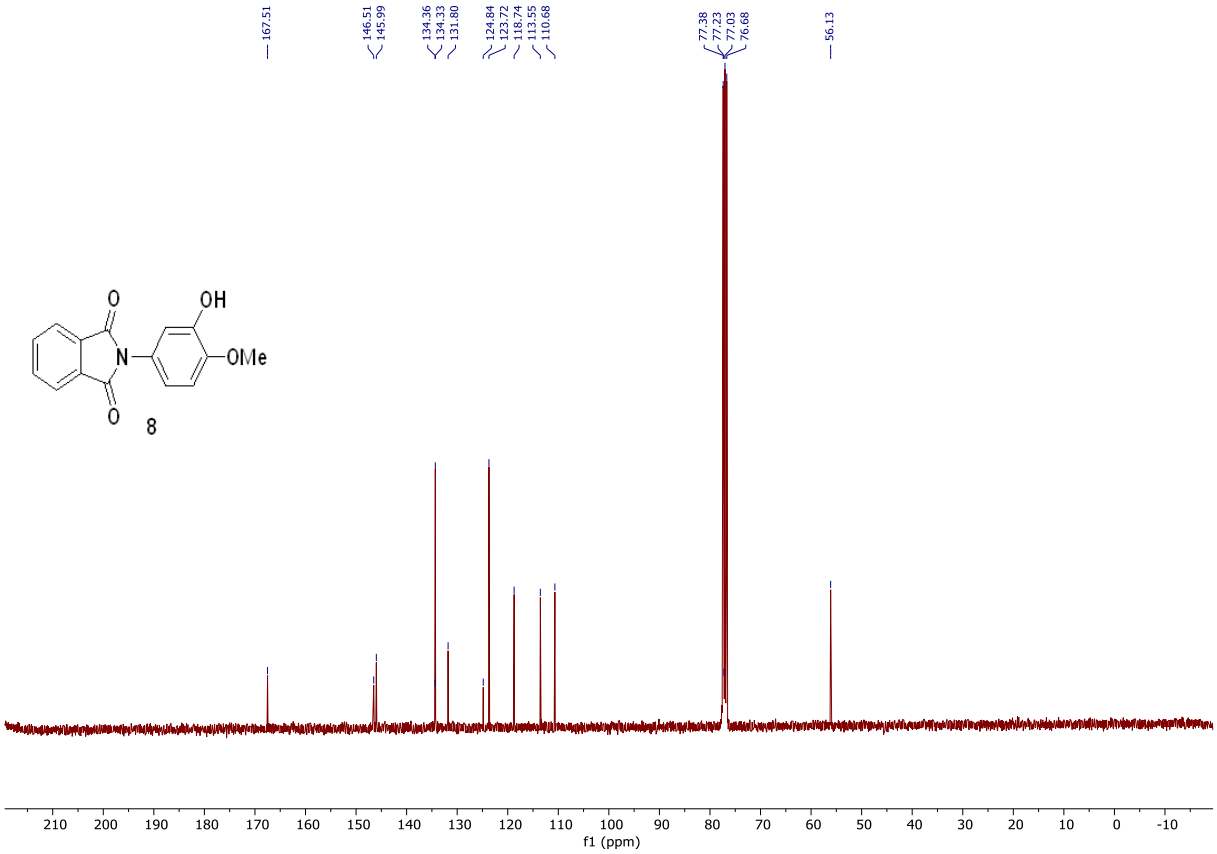
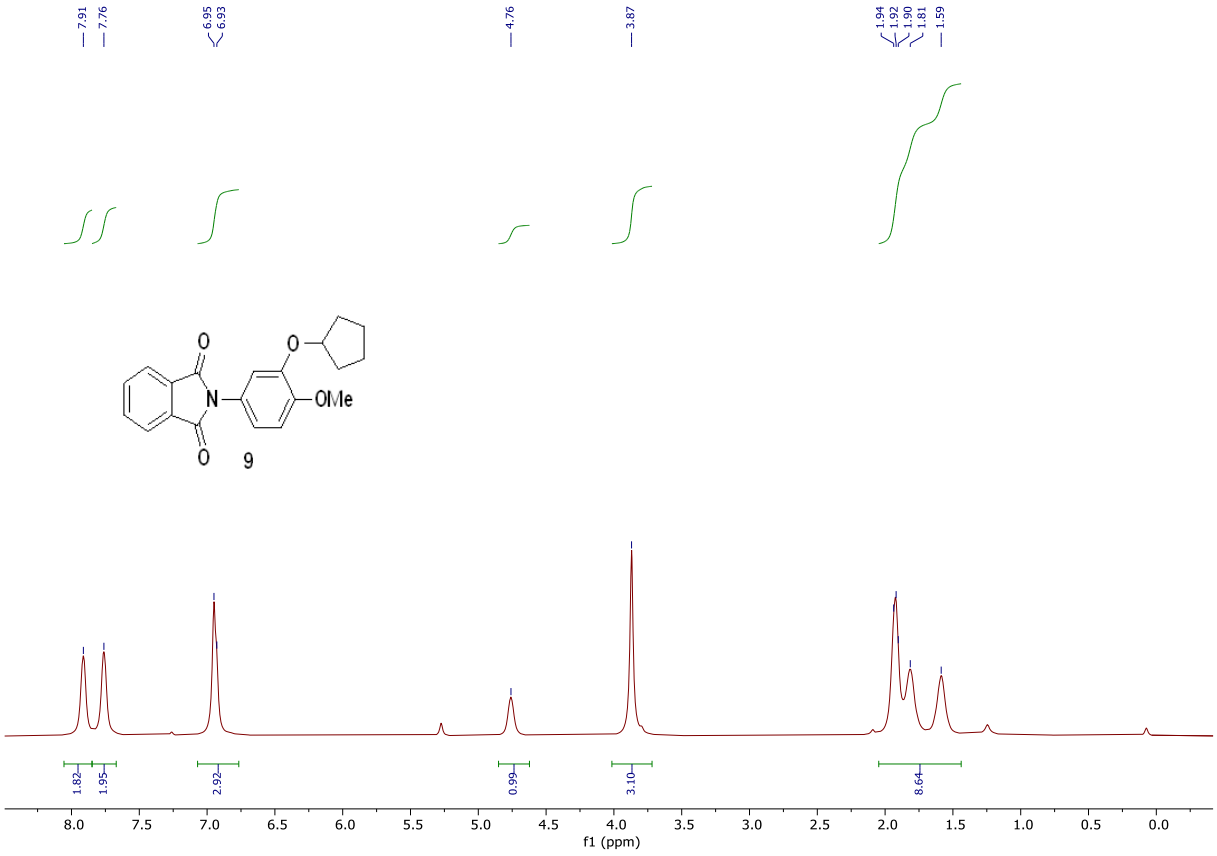
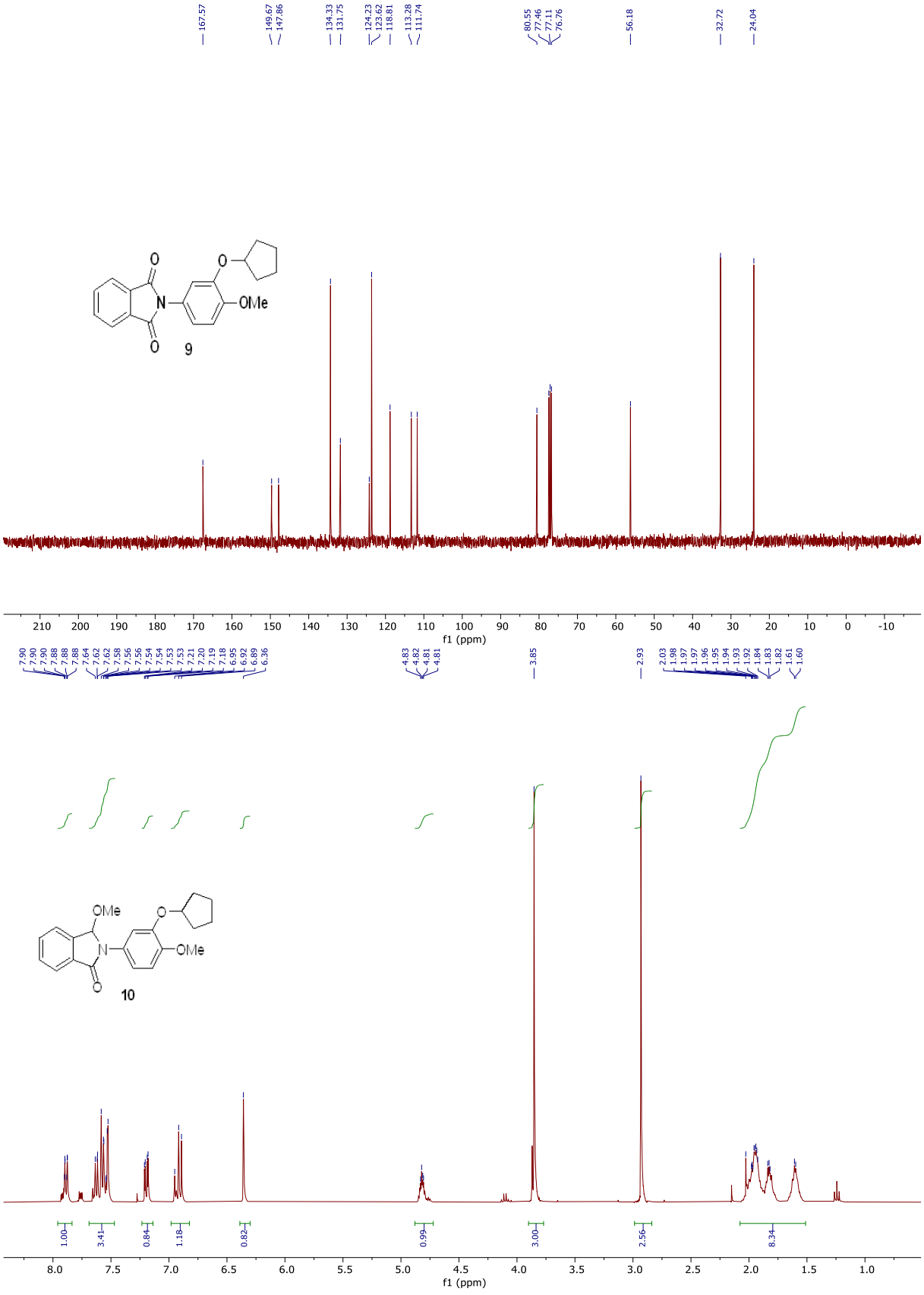
7.97
7.96
7.95
7.81
7.80
7.80
7.79
7.02
7.01
7.00
6.97
6.95
6.94
6.92
6.91
3.96
O
OH
N
OMe
O
8
1.93
2.01
2.81
0.93
3.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)


167.51
146.51
145.99
134.36
134.33
131.80
124.84
123.72
118.74
113.55
110.68
77.38
77.23
77.03
76.68
56.13
O
OH
N
OMe
O
8
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)


7.91
7.76
6.95
6.93
4.76
3.87
1.94
1.92
1.90
1.81
1.59
O
O
N
OMe
O
9
1.82
1.95
2.92
0.99
3.10
8.64
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)


167.57
149.67
147.86
134.33
131.75
124.23
123.62
118.81
113.28
111.74
80.55
77.46
77.11
76.76
56.18
32.72
24.04
O
O
N
O
OMe
O
9
f1 (ppm)
7.90
7.90
7.90
7.88
7.88
7.88
7.64
7.62
7.62
7.58
7.56
7.56
7.54
7.54
7.53
7.53
7.21
7.20
7.19
7.18
6.95
6.92
6.89
6.36
4.83
4.82
4.81
4.81
4.81
3.85
2.93
2.03
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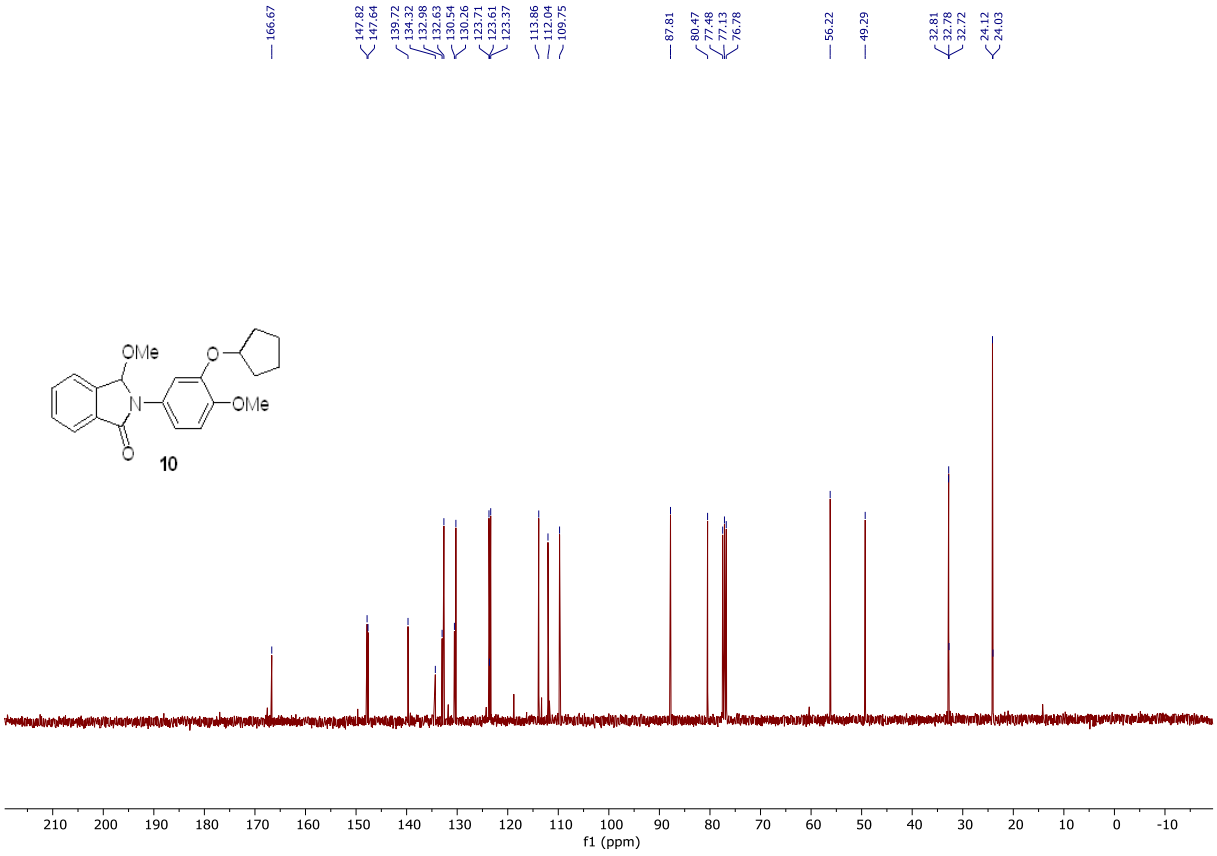
1.97
1.97
1.96
1.95
1.94
1.93
1.92
1.84
1.83
1.82
1.61
1.60
OMe
O
N
OMe
O
10
1.00
3.41
0.84
1.18
0.82
0.99
3.00
2.56
8.34
f1 (ppm)


166.67
147.82
147.64
139.72
134.32
132.98
132.63
130.54
130.26
123.71
123.61
123.37
113.86
112.04
109.75
87.81
80.47
77.48
77.13
76.78
56.22
49.29
32.81
32.78
32.72
24.12
24.03
OMe
O
N
OMe
O
10
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)