Supporting Information for

Expedient access to *N*-alkylphthalimides *via* redox-neutral photocatalysed radical-polar crossover reactions

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1. General information

1.1 Solvents, reagents, and starting materials

All reactions were performed under nitrogen atmosphere. DMF and CH_2Cl_2 were dried from CaH. Acetone was dried from $MgSO_4$. The dehydrated solvents DMSO, DMA and acetonitrile were purchased from Energy Chemical Chemicals. Photocatalysts and alkyl silicates were reported in our previous work.[1] 4-Substituted-DHPs were synthesized with reported procedures.[2] *N*-vinylimides, including **1a-1c**,[3a] **1d**,[3d] **6a-6e**,[3e-3f] **6f**,[3g] and **10**[3h], were prepared according to literature procedures. All other chemicals were purchased from local vendors, and used as supplied unless otherwise stated. TLC analyses were performed on precoated GF_{254} silica gel plates and were visualized under UV254 nm light or by I_2 staining. Column chromatography was carried out using 300-400 mesh silica gel and eluted with petroleum/ethyl acetate unless otherwise noted.

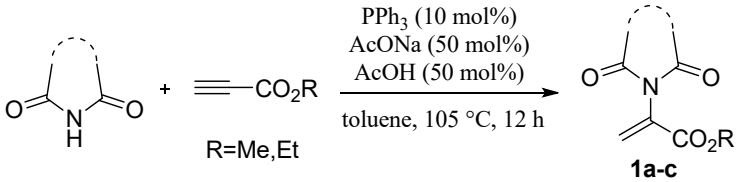
1.2 Instruments

NMR spectra were recorded on a Bruker Avance 500 spectrometer (500 MHz). Chemical shifts were reported in ppm downfield from tetramethylsilane, and calibrated using residue undeuterated solvent ($CHCl_3$ at 7.26 ppm ^{1}H NMR, 77.0 ppm ^{13}C NMR). Spectra were reported as follows: chemical shift (δ ppm), multiplicity (s = singlet, d = doublet, t = triplet, q =quartet, m = multiplet), coupling constants (Hz) and integration. High resolution mass spectra (HRMS) were recorded on an ESI-Q-TOF spectrometer Agilent 6210 ESI/TOF.

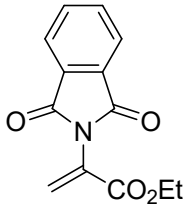
1.3 The photo-irradiation setup

2. Preparation of starting materials 1a-1d and 6a-6f

2.1 General procedure for the preparation of α-imidoacrylates 1a-1c

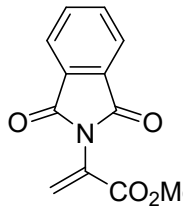


α-imidoacrylates **1a-1c** were synthesized with reported procedure.[3a] To a solution of phthalimide or succinimide (30 mmol), triphenylphosphine (786 mg, 3 mmol), and sodium acetate (1.23 g, 15 mmol) in toluene (60 mL) at 105 ºC were added sequentially acetic acid (0.9 g, 15 mmol) and propiolate (30 mmol). After the reaction was complete as monitored by TLC, the resulting mixture was partitioned between water and ethyl acetate, and the separated aqueous layer extracted with ethyl acetate. The combined organic layers were washed with brine, dried over anhydrous Na_2SO_4, filtered, and evaporated under reduced pressure. The residue was purified by flash column chromatography on silica gel to yield α-imidoacrylates **1a-1c**.



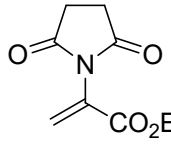
(**1a**)

Ethyl 2-(1,3-dioxoisoindolin-2-yl)acrylate (**1a**). Flash column chromatography to afford product as a pale yellow solid. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.99-7.85 (m, 2H), 7.85-7.66 (m, 2H), 6.67 (s, 1H), 5.98 (s, 1H), 4.28 (q, *J* = 7.1 Hz, 2H), 1.30 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 166.4, 162.2, 134.4, 131.8, 129.4, 127.7, 123.9, 62.0, 14.1. The spectral data match those previously reported.[3a]



(**1b**)

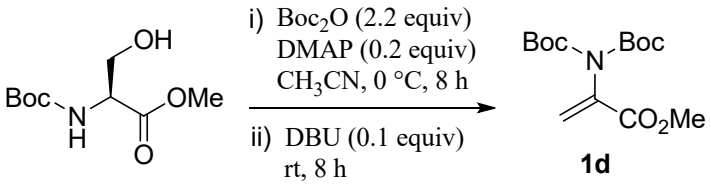
Methyl 2-(1,3-dioxoisoindolin-2-yl)acrylate (**1b**). Flash column chromatography to afford product as a pale yellow solid. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.95-7.89 (m, 2H), 7.81-7.75 (m, 2H), 6.69 (s, 1H), 5.99 (s, 1H), 3.82 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 166.4, 162.7, 134.5, 131.8, 129.1, 128.2, 123.9, 52.8. The spectral data match those previously reported.[3b]



(**1c**)

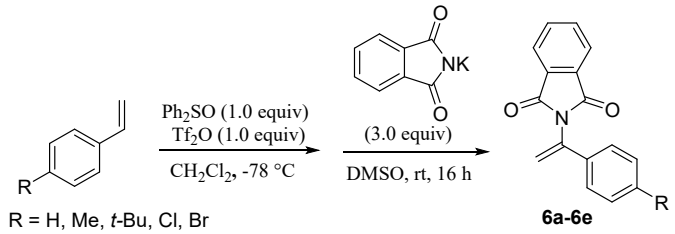
Ethyl 2-(2,5-dioxopyrrolidin-1-yl)acrylate (**1c**). Flash column chromatography to afford product as a colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$): δ 6.63 (s, 1H), 5.88 (s, 1H), 4.26 (q, J = 7.1 Hz, 2H), 2.84 (s, 4H), 1.30 (t, J = 7.1 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 175.2, 161.6, 129.8, 127.9, 62.1, 28.5, 14.0. This compound was reported in the literature.[3c]

2.2 Procedure for the preparation of compound 1d

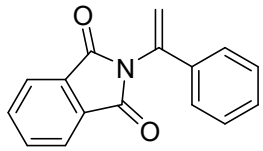


This compound **1d** was prepared according to the published literature.[3d] To a stirring solution of methyl (*tert*-butoxycarbonyl)-L-serinate (2.43 mL, 12 mmol, 1.0 equiv) in acetonitrile (20 mL) at 0 °C was added di-*tert*-butyl dicarbonate (6.5 mL, 28.1 mmol, 2.2 equiv) and 4-dimethylaminopyridine (0.312 g, 2.56 mmol, 0.2 equiv). The resulting solution was warmed to room temperature and stirred for 8 h. Then DBU (0.19 mL, 1.28 mmol, 0.1 equiv) was added, and the resulting mixture was stirred for an additional 8 h. The mixture was concentrated by rotary evaporation then diluted in ethyl acetate. The mixture was washed with 1M HCl and saturated aqueous $NaHCO_3$, dried over Na_2SO_4, filtered, and concentrated by rotary evaporation. The residue was purified by flash column chromatography on silica gel to afford the product **Methyl-2-(di(tert-butoxycarbonyl)amino)but-2-enoate** as a white solid. ^{1}H NMR (500 MHz, $CDCl_3$): δ 6.34 (s, 1H), 5.64 (s, 1H), 3.79 (s, 3H), 1.46 (s, 18H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 164.0, 150.6, 136.1, 124.7, 83.2, 52.4, 27.9. This compound was reported in the literature.[3d]

2.3 General procedure for the preparation of α-phthalimidostyrenes 6a-6e

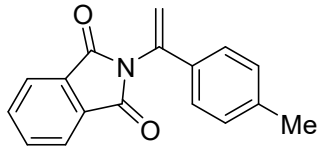


α-phthalimidostyrenes **6a-6e** was prepared according to the published procedure.[3e] To a stirred solution of diphenyl sulfoxide (3.03 g, 15 mmol) in dichloromethane (30 mL) was added triflic anhydride (2.5 mL, 15 mmol) under an argon atmosphere at -78 °C, followed by dropwise addition of styrene (15 mmol) in dichloromethane (30 mL) at the same temperature. After 30 min, the reaction mixture was warmed up to 0 °C, and the solvent was removed in vacuo. To the residue were added DMSO (30 mL) and potassium phthalimide (8.33 g, 45 mmol), and the mixture was stirred at room temperature for 16 h. The reaction mixture was quenched with cold water (100 mL), and the organic material was extracted with ethyl acetate. The combined organic layers were dried over anhydrous sodium sulfate, filtrated, and evaporated. The residue was purified by flash column chromatography to give the products α-phthalimidostyrenes **6a-6e**.



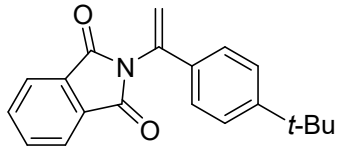
6a

2-(1-Phenylvinyl)isoindoline-1,3-dione (**6a**). Light yellow solid (2.43 g, 65% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.96-7.92 (m, 2H), 7.82-7.79 (m, 2H), 7.40-7.37 (m, 2H), 7.36-7.33 (m, 3H), 6.02 (d, J = 0.7 Hz, 1H), 5.45 (d, J = 0.6 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 167.1, 137.1, 135.3, 134.4, 131.8, 128.9, 128.6, 125.3, 123.8, 116.1. The spectral data match those previously reported.[3e]



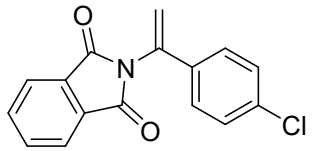
6b

2-(1-(*p*-Tolyl)vinyl)isoindoline-1,3-dione (**6b**). Light yellow solid (2.25 g, 57% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.95-7.93 (m, 2H), 7.80-7.79 (m, 2H), 7.31 (d, J = 8.2 Hz, 2H), 7.16 (d, J = 8.0 Hz, 2H), 5.99 (s, 1H), 5.42 (s, 1H), 2.36 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 167.1, 138.8, 137.1, 134.3, 132.5, 131.8, 129.3 125.2, 123.7, 115.0, 21.1. The spectral data match those previously reported.[3e]



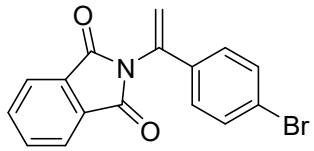
6c

2-(1-(4-(*tert*-Butyl)phenyl)vinyl)isoindoline-1,3-dione (**6c**). Light yellow solid (2.65 g, 58% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.94-7.92 (m, 2H), 7.79-7.77 (m, 2H), 7.36-7.31 (m, 4H), 6.00 (s, 1H), 5.40 (s, 1H), 1.30 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 167.1, 152.0, 137.0, 134.3, 132.3, 131.8, 125.6, 125.0, 123.7, 115.3, 34.6, 31.1. This compound was reported in the literature.[3f]



6d

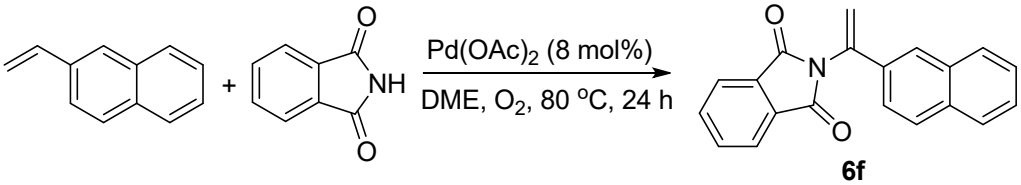
2-(1-(4-Chlorophenyl)vinyl)-3-methyleneisoindolin-1-one (**6d**). Light yellow solid (3.46 g, 82% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.94-7.92 (m, 2H), 7.82-7.79 (m, 2H), 7.33-7.29 (m, 4H), 5.97 (d, J = 0.9 Hz, 1H), 5.46 (d, J = 0.9 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 166.9, 136.1, 134.7, 134.4, 134.0, 131.6, 128.8, 126.7, 123.8, 116.4. The spectral data match those previously reported.[3e]



6e

2-(1-(4-Bromophenyl)vinyl)isoindoline-1,3-dione (**6e**). Yellow solid (1.76 g, 36% yield), m.p. 159.2-160.0 °C. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.94-7.93 (m, 2H), 7.81-7.79 (m, 2H), 7.46 (d, J = 8.3 Hz, 2H), 7.25 (d, J = 8.8 Hz, 2H), 5.98 (s, 1H), 5.47 (s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 166.9, 136.3, 134.54, 134.51, 131.8, 131.7, 127.1, 123.9, 123.1, 116.6; HRMS (ESI) calculated for $C_{16}H_{11}BrNO_2$ $[M+H]^+$ 327.9973, found 327.9975.

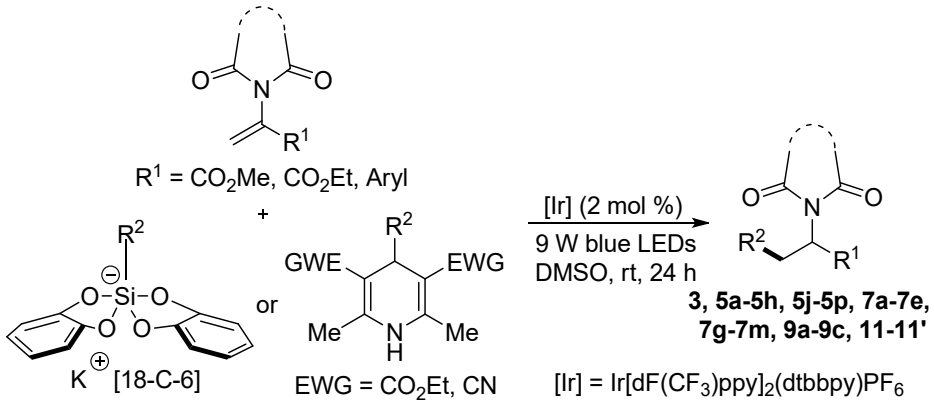
2.4 Procedure for the preparation of compound 6f



6f

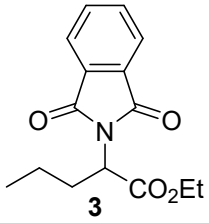
To a Schlenk tube equipped with stirring bar, $Pd(OAc)_2$ (18 mg, 0.08 mmol), 2-vinylnaphthalene (154 mg, 1 mmol) and phthalimide (294 mg, 2 mmol) was added. The tube was evacuated and filled with oxygen for 3 times. Then the reaction tube was charged with 1,2-dimethoxyethane (2 mL) and tightly sealed with a rubber cap. After stirring for 24 h in a 80 °C oil bath, the reaction mixture was allowed to cool and loaded directly onto a chromatography column to give the product **6f** as a white solid. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.97-7.96 (m, 2H), 7.83-7.76 (m, 6H), 7.56 (d, J = 8.5 Hz, 1H), 7.46-7.45 (m, 2H), 6.14 (s, 1H), 5.54 (s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 167.2, 137.3, 134.4, 133.5, 133.1, 132.8, 131.9, 128.5, 128.4, 127.6, 126.5, 126.4, 124.7, 123.9, 123.1, 116.6. This compound was reported in the literature.[3g]

3. General procedure for the synthesis of 3, 5a-5h, 5j-5p, 7a-7e, 7g-7m, 9a-9c and 11-11'



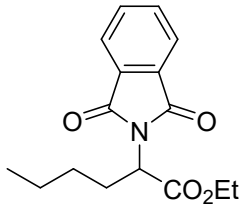
To an oven dried Schlenk tube equipped with stirring bar, $Ir[dF(CF_3)ppy)]_2(dtbbpy)PF_6$ (4.5 mg, 0.004 mmol, 0.02 equiv), potassium [18-crown-6] bis(catecholato) alkylsilicate or 4-substituted Hantzsch ester (0.4 mmol, 2.0 equiv), the *N*-vinylimides **1**, **6** or **10** (0.2 mmol, 1.0 equiv) were added. The tube was evacuated and filled with nitrogen for 3 times. Then the tube was

charged with degassed DMSO (6.0 mL), and irradiated with a 9 W blue LEDs strip spiraled within a bowel for 24 h (cooling with a fan). After the reaction was complete, the reaction solution was diluted with saturated Na_2CO_3 aqueous solution (10 mL), and organic material was extracted with EtOAc (3 x 10 mL). The organic layer was washed with brine, dried over anhydrous Na_2SO_4, filtered, and evaporated. The residue was purified by flash column chromatography to afford the product.



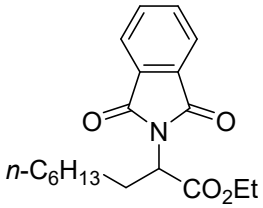
3

Ethyl 2-(1,3-dioxoisoindolin-2-yl)pentanoate (**3**). Light yellow oil (41.3 mg, 75% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.87-7.85 (m, 2H), 7.74-7.73 (m, 2H), 4.85 (dd, *J* = 11.1 Hz, 4.8 Hz, 1H), 4.23-4.16 (m, 2H), 2.31-2.23 (m, 1H), 2.20-2.13 (m, 1H), 1.36-1.27 (m, 2H), 1.22 (t, *J* = 7.1 Hz, 3H), 0.93 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 169.4, 167.7, 134.1, 131.8, 123.5, 61.7, 52.0, 30.6, 19.6, 14.1, 13.3. This compound was reported in the literature.[4]



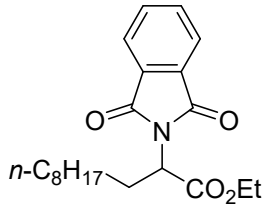
5a

Ethyl 2-(1,3-dioxoisoindolin-2-yl)hexanoate (**5a**). Light yellow oil (45.1 mg, 78% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.87-7.85 (m, 2H), 7.75-7.73 (m, 2H), 4.82 (dd, *J* = 10.7 Hz, 5.1 Hz, 1H), 4.24-4.14 (m, 2H), 2.30-2.17 (m, 2H), 1.40-1.23 (m, 4H), 1.22 (t, *J* = 7.1 Hz, 3H), 0.87-0.84 (m, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 169.4, 167.7, 134.1, 131.8, 123.5, 61.7, 52.3, 28.5, 28.3, 22.0, 14.1, 13.8. This compound was reported in the literature.[4]



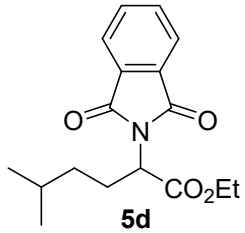
5b

Ethyl 2-(1,3-dioxoisoindolin-2-yl)nonanoate (**5b**). Light yellow oil (47.7 mg, 72% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.87-7.85 (m, 2H), 7.74-7.72 (m, 2H), 4.82 (dd, *J* = 10.8 Hz, 5.1 Hz, 1H), 4.23-4.14 (m, 2H), 2.29-2.15 (m, 2H), 1.36-1.17 (m, 13H), 0.84-0.81 (m, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 169.4, 167.7, 134.1, 131.8, 123.4, 61.7, 52.3, 31.6, 29.0, 28.8, 28.6, 26.3, 22.5, 14.04, 13.97; HRMS (ESI) calculated for $C_{19}H_{26}NO_4$ $[M+H]^+$ 332.1862, found 332.1865.



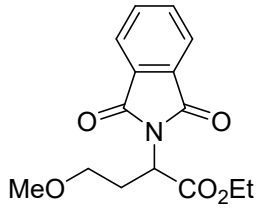
5c

Ethyl 2-(1,3-dioxoisoindolin-2-yl)undecanoate (**5c**). Light yellow oil (35.2 mg, 49%). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.88-7.86 (m, 2H), 7.75-7.74 (m, 2H), 4.82 (dd, *J* = 10.9 Hz, 4.9 Hz, 1H), 4.24-4.16 (m, 2H), 2.29-2.16 (m, 2H), 1.32-1.21 (m, 17H), 0.86-0.83 (m, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 169.5, 167.8, 134.1, 131.8, 123.5, 61.7, 52.3, 31.8, 29.4, 29.3, 29.2, 28.9, 28.6, 26.3, 22.6, 14.1; HRMS (ESI) calculated for $C_{21}H_{29}NO_4Na$ $[M+Na]^+$ 382.1994, found 382.1991.



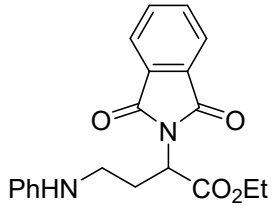
5d

Ethyl 2-(1,3-dioxoisoindolin-2-yl)-5-methylhexanoate (**5d**). Light yellow oil (45.5 mg, 75% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.88-7.85 (m, 2H), 7.75-7.73 (m, 2H), 4.80-4.77 (m, 1H), 4.24-4.14 (m, 2H), 2.26-2.21 (m, 2H), 1.61-1.53 (m, 1H), 1.27-1.21 (m, 4H), 1.14-1.06 (m, 1H), 0.87-0.84 (m, 6H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 169.4, 167.7, 134.1, 131.8, 123.5, 61.7, 52.6, 35.4, 27.6, 26.6, 22.6, 22.2, 14.1; HRMS (ESI) calculated for $C_{17}H_{21}NO_4Na$ $[M+Na]^+$ 326.1368, found 326.1372.



5e

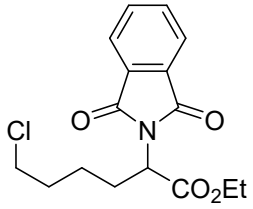
Ethyl 2-(1,3-dioxoisoindolin-2-yl)-4-methoxybutanoate (**5e**). Light yellow oil (36.1 mg, 62% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.88-7.86 (m, 2H), 7.75-7.73 (m, 2H), 5.06 (dd, *J* = 10.4 Hz, 4.5 Hz, 1H), 4.23-4.18 (m, 2H), 3.51-3.47 (m, 1H), 3.34-3.29 (m, 1H), 3.23 (s, 3H), 2.56-2.50 (m, 1H), 2.47-2.40 (m, 1H), 1.22 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 169.3, 167.7, 134.1, 131.9, 123.4, 69.0, 61.8, 58.6, 49.7, 28.9, 14.1; HRMS (ESI) calculated for $C_{15}H_{17}NO_5Na$ $[M+Na]^+$ 314.1004, found 314.1002.



5f

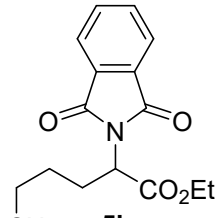
Ethyl 2-(1,3-dioxoisoindolin-2-yl)-4-(phenylamino)butanoate (**5f**). White solid (63.4 mg, 90% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.88-7.86 (m, 2H), 7.76-7.74 (m, 2H), 7.15-7.12 (m, 2H), 6.67 (t, *J* = 7.3 Hz, 1H), 6.59-6.57 (m, 2H), 5.01 (dd, *J* = 10.5 Hz, 4.7 Hz, 1H), 4.23-4.17 (m, 2H),

3.33-3.28 (m, 1H), 3.15-3.10 (m, 1H), 2.65-2.58 (m, 1H), 2.45-2.38 (m, 1H), 1.22 (t, J = 7.1 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 169.1, 167.7, 147.5, 134.3, 131.7, 129.2, 123.6, 117.5, 112.8, 62.0, 50.0, 40.5, 28.6, 14.1; HRMS (ESI) calculated for $C_{20}H_{21}N_2O_4$ $[M+H]^+$ 353.1501, found 353.1497.



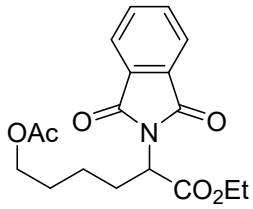
5g

Ethyl 6-chloro-2-(1,3-dioxoisoindolin-2-yl)hexanoate (**5g**). Light yellow oil (45.9 mg, 71% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.88-7.86 (m, 2H), 7.77-7.75 (m, 2H), 4.83 (dd, J = 10.4 Hz, 5.2 Hz, 1H), 4.23-4.16 (m, 2H), 3.51-3.48 (m, 2H), 2.32-2.21 (m, 2H), 1.88-1.74 (m, 2H), 1.51-1.41 (m, 2H), 1.22 (t, J = 7.2 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 169.1, 167.7, 134.2, 131.7, 123.6, 61.9, 52.0, 44.5, 31.8, 27.9, 23.6, 14.1; HRMS (ESI) calculated for $C_{16}H_{19}ClNO_4$ $[M+H]^+$ 324.1003, found 324.1007.



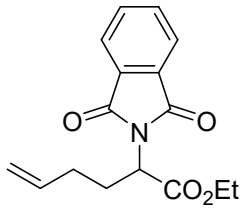
5h

Ethyl 5-cyano-2-(1,3-dioxoisoindolin-2-yl)pentanoate (**5h**). Yellow syrups (49.2 mg, 82% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.88-7.87 (m, 2H), 7.77-7.76 (m, 2H), 4.82 (dd, J = 10.0 Hz, 5.3 Hz, 1H), 4.24-4.18 (m, 2H), 2.42-2.31 (m, 4H), 1.77-1.67 (m, 2H), 1.23 (t, J = 7.1 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 168.5, 167.6, 134.4, 131.6, 123.7, 118.9, 62.1, 51.2, 28.0, 22.5, 16.6, 14.0; HRMS (ESI) calculated for $C_{16}H_{16}N_2O_4Na$ $[M+Na]^+$ 323.1008, found 323.1015.



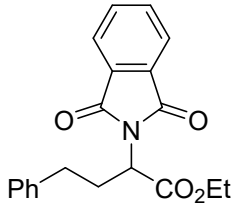
5j

Ethyl 6-acetoxy-2-(1,3-dioxoisoindolin-2-yl)hexanoate (**5j**). Light yellow syrups (52.1 mg, 75% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.88-7.85 (m, 2H), 7.76-7.74 (m, 2H), 4.82 (dd, J = 10.3 Hz, 5.4 Hz, 1H), 4.25-4.16 (m, 2H), 4.05-3.97 (m, 2H), 2.33-2.21 (m, 2H), 1.98 (s, 3H), 1.74-1.58 (m, 2H), 1.44-1.31 (m, 2H), 1.22 (t, J = 7.1 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 171.1, 169.1, 167.7, 134.2, 131.8, 123.5, 64.0, 61.8, 52.1, 28.3, 27.9, 22.9, 20.9, 14.1; HRMS (ESI) calculated for $C_{18}H_{21}NO_6Na$ $[M+Na]^+$ 370.1267, found 370.1267.



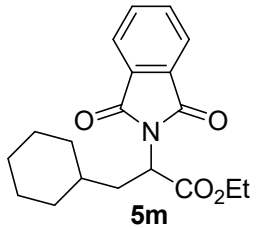
5k

Ethyl 2-(1,3-dioxoisoindolin-2-yl)hex-5-enoate (**5k**). Light yellow oil (12.1 mg, 21% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.88-7.85 (m, 2H), 7.76-7.73 (m, 2H), 5.81-5.73 (m, 1H), 5.03-4.95 (m, 2H), 4.86 (dd, J = 10.6 Hz, 4.9 Hz, 1H), 4.25-4.15 (m, 2H), 2.43-2.30 (m, 2H), 2.14-2.05 (m, 2H), 1.23 (t, J = 7.1 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 169.3, 167.7, 136.6, 134.2, 131.8, 123.5, 116.0, 61.8, 51.7, 30.5, 27.9, 14.1; HRMS (ESI) calculated for $C_{16}H_{18}NO_4$ $[M+H]^+$ 288.1236, found 288.1234.



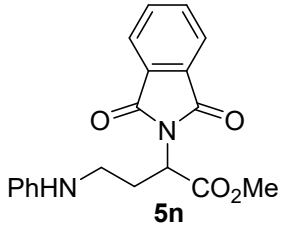
5l

Ethyl 2-(1,3-dioxoisoindolin-2-yl)-4-phenylbutanoate (**5l**). Light yellow oil (48.5 mg, 72% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.85-7.83 (m, 2H), 7.74-7.72 (m, 2H), 7.22-19 (m, 2H), 7.21-7.07 (m, 3H), 4.88-4.85 (m, 1H), 4.22-4.16 (m, 2H), 2.71-2.61 (m, 3H), 2.60-2.54 (m, 1H), 1.22 (t, J = 7.1 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 169.2, 167.7, 140.3, 134.1, 131.8, 128.36, 128.35, 126.0, 123.4, 61.8, 52.1, 32.7, 30.0, 14.1; HRMS (ESI) calculated for $C_{20}H_{19}NO_4Na$ $[M+Na]^+$ 360.1212, found 360.1211.



5m

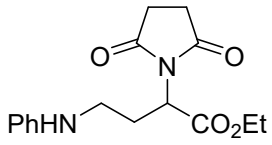
Ethyl 3-cyclohexyl-2-(1,3-dioxoisoindolin-2-yl)propanoate (**5m**). Light yellow syrups (54.6 mg, 83% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.87-7.84 (m, 2H), 7.75-7.72 (m, 2H), 4.95 (dd, J = 11.5 Hz, 4.4 Hz, 1H), 4.21-4.14 (m, 2H), 2.29-2.23 (m, 1H), 2.04-1.98 (m, 1H), 1.87 (d, J = 12.7 Hz, 1H), 1.71-1.60 (m, 4H), 1.20 (t, J = 7.1 Hz, 3H), 1.15-1.09 (m, 4H), 1.02-0.97 (m, 1H), 0.95-0.86 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 169.8, 167.7, 134.1, 131.8, 123.4, 61.7, 50.1, 35.9, 34.3, 33.7, 31.6, 26.3, 26.1, 25.8, 14.0. This compound was reported in the literature.[5]



5n

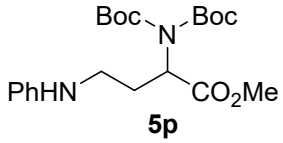
Methyl 2-(1,3-dioxoisoindolin-2-yl)-4-(phenylamino)butanoate (**5n**). Light yellow syrups (20.3 mg, 30% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.88-7.86 (m, 2H), 7.76-7.75 (m, 2H), 7.15-7.12

(m, 2H), 6.67 (t, *J* = 7.8 Hz, 1H), 6.58 (d, *J* = 8.0 Hz, 2H), 5.03 (dd, *J* = 10.5 Hz, 4.6 Hz, 1H), 3.73 (s, 3H), 3.33-3.28 (m, 1H), 3.15-3.09 (m, 1H), 2.65-2.58 (m, 1H), 2.45-2.38 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 169.6, 167.7, 147.5, 134.3, 131.7, 129.2, 123.6, 117.5, 112.8, 52.8, 49.8, 40.5, 28.5; HRMS (ESI) calculated for $C_{19}H_{19}N_2O_4$ $[M+H]^+$ 339.1345, found 339.1342.



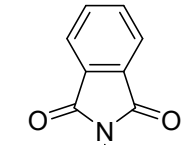
5o

Ethyl 2-(2,5-dioxopyrrolidin-1-yl)-4-(phenylamino)butanoate (**5o**). Yellow syrups (51.1 mg, 84% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.17-7.14 (m, 2H), 6.69 (t, *J* = 7.3 Hz, 1H), 6.57 (d, *J* = 7.8 Hz, 2H), 4.83 (dd, *J* = 10.2 Hz, 4.6 Hz, 1H), 4.22-4.16 (m, 2H), 3.82 (br, 1H), 3.27-3.21 (m, 1H), 3.14-3.08 (m, 1H), 2.66 (s, 4H), 2.53-2.46 (m, 1H), 2.36-2.29 (m, 1H), 1.24 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 176.6, 168.5, 147.5, 129.3, 117.5, 112.7, 62.0, 50.8, 40.6, 28.1, 27.3, 14.0; HRMS (ESI) calculated for $C_{16}H_{21}N_2O_4$ $[M+H]^+$ 305.1501, found 305.1501.



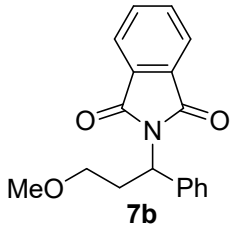
5p

Methyl 2-*N*,*N*-di(*tert*-butoxycarbonyl)-4-(phenylamino)butanoate (**5p**). White solid (75.9 mg, 93% yield), m.p. 81.0-81.7 °C. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.17-7.14 (m, 2H), 6.68 (t, *J* = 7.3 Hz, 1H), 6.61 (d, *J* = 8.1 Hz, 2H), 5.01 (dd, *J* = 9.0 Hz, 5.1 Hz, 1H), 3.96 (br, 1H), 3.72 (s, 3H), 3.32-3.27 (m, 1H), 3.19-3.14 (m, 1H), 2.53-2.46 (m, 1H), 2.14-2.07 (m, 1H), 1.49 (s, 18H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 171.2, 152.2, 147.9, 129.2, 117.2, 112.8, 83.4, 56.1, 52.2, 40.6, 29.8, 27.9; HRMS (ESI) calculated for $C_{21}H_{33}N_2O_6$ $[M+H]^+$ 409.2331, found 409.2339.



7a

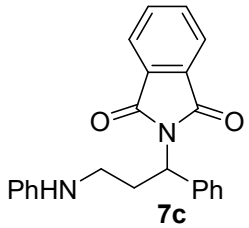
2-(1-Phenylbutyl)isoindoline-1,3-dione (**7a**). Light yellow syrups (37.4 mg, 67% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.71-7.70 (m, 2H), 7.59-7.58 (m, 2H), 7.46 (d, *J* = 7.8 Hz, 2H), 7.25-7.22 (m, 2H), 7.18-7.15 (m, 1H), 5.28-5.25 (m, 1H), 2.53-2.46 (m, 1H), 2.18-2.11 (m, 1H), 1.31-1.22 (m, 2H), 0.88 (t, *J* = 7.3 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 168.4, 139.8, 133.9, 131.8, 128.4, 128.1, 127.7, 123.1, 54.6, 32.9, 20.2, 13.7. The spectral data match those previously reported.[6]



7b

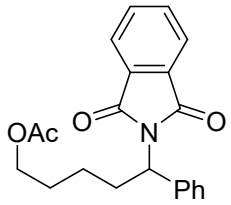
2-(3-Methoxy-1-phenylpropyl)isoindoline-1,3-dione (**7b**). Light yellow solid (49.0 mg, 83%

yield), m.p. 73.8-74.6 °C. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.81-7.80 (m, 2H), 7.69-7.68 (m, 2H), 7.55 (d, *J* = 7.5 Hz, 2H), 7.34-7.31 (m, 2H), 7.28-7.25 (m, 1H), 5.56 (dd, *J* = 9.5 Hz, 6.5 Hz, 1H), 3.42-3.39 (m, 2H), 3.26 (s, 3H), 2.89-2.82 (m, 1H), 2.58-2.52 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 168.4, 139.3, 133.9, 131.8, 128.5, 128.2, 127.8, 123.2, 69.6, 58.7, 51.9, 30.9; HRMS (ESI) calculated for $C_{18}H_{18}NO_3$ $[M+H]^+$ 296.1287, found 296.1283.



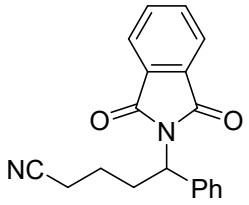
7c

2-(1-Phenyl-3-(phenylamino)propyl)isoindoline-1,3-dione (**7c**). Light yellow solid (37.0 mg, 52% yield), m.p. 79.2-79.8 °C. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.84-7.81 (m, 2H), 7.73-7.70 (m, 2H), 7.58 (d, *J* = 7.3 Hz, 2H), 7.37-7.34 (m, 2H), 7.31-7.28 (m, 1H), 7.17-7.14 (m, 2H), 6.71-6.68 (m, 1H), 6.60 (d, *J* = 7.8 Hz, 2H), 5.54 (dd, *J* = 9.6 Hz, 6.5 Hz, 1H), 3.81 (br, 1H), 3.31-3.26 (m, 1H), 3.22-3.17 (m, 1H), 2.92-2.85 (m, 1H), 2.72-2.65 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 168.3, 147.7, 139.1, 134.0, 131.8, 129.2, 128.6, 128.1, 128.0, 123.3, 117.5, 112.9, 52.7, 41.3, 30.6; HRMS (ESI) calculated for $C_{23}H_{21}N_2O_2$ $[M+H]^+$ 357.1603, found 357.1607.



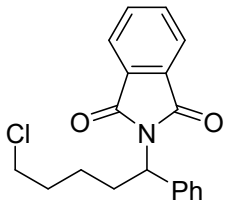
7d

5-(1,3-Dioxoisoindolin-2-yl)-5-phenylpentyl acetate (**7d**). Light yellow syrups (42.8 mg, 61% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.84-7.80 (m, 2H), 7.72-7.70 (m, 2H), 7.56 (d, *J* = 7.4 Hz, 2H), 7.36-7.27 (m, 3H), 5.35 (dd, *J* = 9.6 Hz, 6.7 Hz, 1H), 4.07-4.04 (m, 2H), 2.68-2.60 (m, 1H), 2.37-2.30 (m, 1H), 2.01 (s, 3H), 1.78-1.68 (m, 2H), 1.45-1.39 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 171.1, 168.3, 139.5, 133.9, 131.8, 128.5, 128.1, 127.9, 123.2, 64.1, 54.8, 30.6, 28.1, 23.4, 20.9; HRMS (ESI) calculated for $C_{21}H_{21}NO_4Na$ $[M+Na]^+$ 374.1368, found 374.1366.



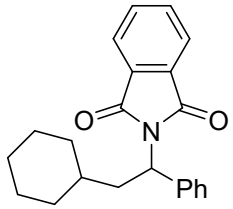
7e

5-(1,3-Dioxoisoindolin-2-yl)-5-phenylpentanenitrile (**7e**). Light yellow solid (41.3 mg, 68% yield), m.p. 83.0-83.7 °C. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.83-7.79 (m, 2H), 7.72-7.69 (m, 2H), 7.53 (d, *J* = 7.4 Hz, 2H), 7.35-7.26 (m, 3H), 5.33 (dd, *J* = 9.4 Hz, 6.9 Hz, 1H), 2.76-2.68 (m, 1H), 2.53-2.46 (m, 1H), 2.41 (t, *J* = 7.2 Hz, 2H), 1.76-1.66 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 168.2, 138.7, 134.1, 131.7, 128.7, 128.2, 128.0, 123.3, 119.1, 54.2, 30.1, 23.1, 16.8; HRMS (ESI) calculated for $C_{19}H_{17}N_2O_2$ $[M+H]^+$ 305.1290, found 305.1294.



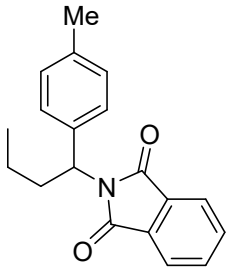
7g

2-(5-Chloro-1-phenylpentyl)isoindoline-1,3-dione (**7g**). Light yellow syrups (36.6 mg, 56% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.82-7.79 (m, 2H), 7.70-7.67 (m, 2H), 7.54 (d, *J* = 7.4 Hz, 2H), 7.34-7.31 (m, 2H), 7.28-7.25 (m, 1H), 5.33 (dd, *J* = 9.6 Hz, 6.7 Hz, 1H), 3.51 (t, *J* = 6.6 Hz, 2H), 2.66-2.58 (m, 1H), 2.35-2.28 (m, 1H), 1.90-1.79 (m, 2H), 1.52-1.46 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 168.3, 139.5, 133.9, 131.8, 128.6, 128.1, 127.9, 123.2, 54.8, 44.6, 32.1, 30.3, 24.3. The spectral data match those previously reported.[7]



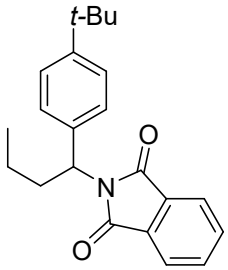
7h

2-(2-Cyclohexyl-1-phenylethyl)isoindoline-1,3-dione (**7h**). White solid (53.3 mg, 80% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.82-7.78 (m, 2H), 7.70-7.66 (m, 2H), 7.55 (d, *J* = 7.3 Hz, 2H), 7.33-7.30 (m, 2H), 7.27-7.24 (m, 1H), 5.49 (dd, *J* = 10.1 Hz, 6.3 Hz, 1H), 2.61-2.55 (m, 1H), 2.10-2.05 (m, 1H), 1.88 (d, *J* = 12.6 Hz, 1H), 1.76 (d, *J* = 12.8 Hz, 1H), 1.68-1.65 (m, 2H), 1.61 (s, 1H), 1.23-1.11 (m, 4H), 1.05-0.94 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 168.4, 140.1, 133.8, 131.9, 128.5, 128.2, 127.7, 123.2, 52.3, 38.4, 34.7, 33.5, 32.6, 26.5, 26.1, 26.0. This compound was reported in the literature.[8]



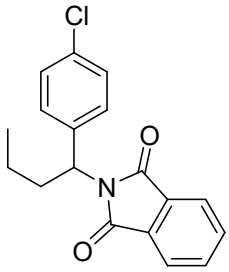
7i

2-(1-(*p*-Tolyl)butyl)isoindoline-1,3-dione (**7i**). Light yellow syrups (47.5 mg, 81% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.80-7.77 (m, 2H), 7.69-7.65 (m, 2H), 7.44 (d, *J* = 8.1 Hz, 2H), 7.13 (d, *J* = 8.0 Hz, 2H), 5.32 (dd, *J* = 9.6 Hz, 6.6 Hz, 1H), 2.59-2.52 (m, 1H), 2.31 (s, 3H), 2.27-2.19 (m, 1H), 1.39-1.31 (m, 2H), 0.97 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 168.4, 137.4, 136.8, 133.8, 131.9, 129.1, 128.1, 123.1, 54.4, 33.0, 21.0, 20.2, 13.6; HRMS (ESI) calculated for $C_{19}H_{19}NO_2Na$ $[M+Na]^+$ 316.1313, found 316.1318.



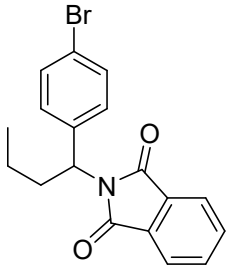
7j

2-(1-(4-(*tert*-Butyl)phenyl)butyl)isoindoline-1,3-dione (**7j**). Light yellow syrups (46.2 mg, 69% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.80-7.77 (m, 2H), 7.69-7.66 (m, 2H), 7.47 (d, *J* = 8.3 Hz, 2H), 7.33 (d, *J* = 8.3 Hz, 2H), 5.33 (dd, *J* = 9.8 Hz, 6.5 Hz, 1H), 2.63-2.55 (m, 1H), 2.24-2.17 (m, 1H), 1.38-1.33 (m, 2H), 1.28 (s, 9H), 0.96 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 168.4, 150.5, 136.8, 133.8, 131.9, 127.8, 125.3, 123.1, 54.4, 34.5, 33.0, 31.3, 20.2, 13.7; HRMS (ESI) calculated for $C_{22}H_{26}NO_2$ $[M+H]^+$ 336.1964, found 336.1963.



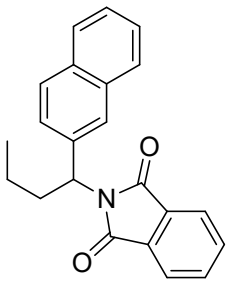
7k

2-(1-(4-Chlorophenyl)butyl)isoindoline-1,3-dione (**7k**). Light yellow syrups (48.2 mg, 77% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.81-7.79 (m, 2H), 7.70-7.68 (m, 2H), 7.48 (d, *J* = 8.5 Hz, 2H), 7.28 (d, *J* = 8.5 Hz, 2H), 5.31 (dd, *J* = 9.6 Hz, 6.7 Hz, 1H), 2.58-2.50 (m, 1H), 2.24-2.17 (m, 1H), 1.39-1.29 (m, 2H), 0.96 (t, *J* = 7.3 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 168.3, 138.2, 134.0, 133.6, 131.8, 129.6, 128.6, 123.2, 54.0, 32.9, 20.1, 13.6; HRMS (ESI) calculated for $C_{18}H_{17}ClNO_2$ $[M+H]^+$ 314.0948, found 314.0948.



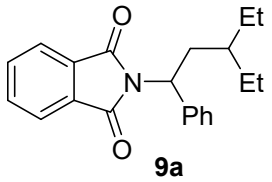
7l

2-(1-(4-Bromophenyl)butyl)isoindoline-1,3-dione (**7l**). Light yellow syrups (60.7 mg, 85% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.81-7.78 (m, 2H), 7.71-7.67 (m, 2H), 7.45-7.41 (m, 4H), 5.30 (dd, *J* = 9.7 Hz, 6.7 Hz, 1H), 2.57-2.49 (m, 1H), 2.24-2.17 (m, 1H), 1.38-1.30 (m, 2H), 0.96 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 168.3, 138.7, 134.0, 131.8, 131.6, 130.0, 123.2, 121.7, 54.0, 32.8, 20.1, 13.6; HRMS (ESI) calculated for $C_{18}H_{16}BrNO_2$ $[M+H]^+$ 358.0443, found 358.0440.



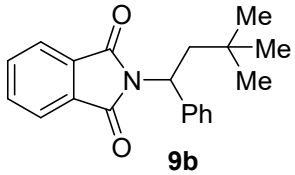
7m

2-(1-(naphthalen-2-yl)butyl)isoindoline-1,3-dione (**7m**). Light yellow syrups (44.8 mg, 68% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.99 (s, 1H), 7.85-7.79 (m, 5H), 7.71-7.66 (m, 3H), 7.46-7.44 (m, 2H), 5.53 (dd, *J* = 6.8 Hz, 9.5 Hz, 1H), 2.72-2.64 (m, 1H), 2.41-2.34 (m, 1H), 1.46-1.37 (m, 2H), 1.01 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 168.4, 137.2, 133.9, 133.2, 132.9, 131.9, 128.2, 128.1, 127.5, 127.2, 126.1, 126.0, 123.2, 54.8, 33.0, 20.2, 13.7; HRMS (ESI) calculated for $C_{22}H_{19}NO_2Na$ $[M+Na]^+$ 352.1313, found 352.1316.



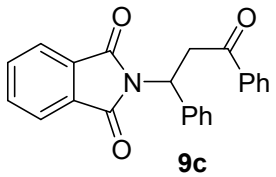
9a

2-(3-Ethyl-1-phenylpentyl)isoindoline-1,3-dione (**9a**). Light yellow syrups (39.8 mg, 62% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.83-7.82 (m, 2H), 7.72-7.69 (m, 2H), 7.59 (d, *J* = 7.7 Hz, 2H), 7.36-7.33 (m, 2H), 7.29-7.26 (m, 1H), 5.48 (dd, *J* = 10.0 Hz, 6.3 Hz, 1H), 2.67-2.61 (m, 1H), 2.18-2.12 (m, 1H), 1.52-1.45 (m, 1H), 1.43-1.33 (m, 3H), 1.24-1.17 (m, 1H), 0.87 (t, *J* = 7.4 Hz, 6H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 168.4, 140.1, 133.8, 131.9, 128.5, 128.2, 127.7, 123.1, 52.8, 37.3, 34.2, 25.2, 24.5, 10.7, 10.1; HRMS (ESI) calculated for $C_{21}H_{24}NO_2$ $[M+H]^+$ 322.1807, found 322.1807.



9b

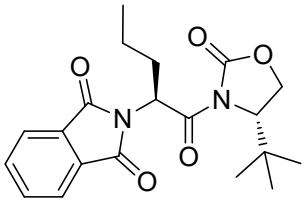
2-(3,3-Dimethyl-1-phenylbutyl)isoindoline-1,3-dione (**9b**). White solid (52.8 mg, 86% yield), m.p. 109.7-110.8 °C. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.82-7.80 (m, 2H), 7.70-7.68 (m, 2H), 7.60 (d, *J* = 7.3 Hz, 2H), 7.35-7.32 (m, 2H), 7.28-7.24 (m, 1H), 5.54 (dd, *J* = 9.2 Hz, 4.4 Hz, 1H), 2.80 (dd, *J* = 14.7 Hz, 9.2 Hz, 1H), 2.14 (dd, *J* = 14.7 Hz, 4.4 Hz, 1H), 0.96 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 168.3, 141.3, 133.8, 131.9, 128.5, 128.2, 127.6, 123.1, 52.0, 43.8, 30.8, 29.6; HRMS (ESI) calculated for $C_{20}H_{22}NO_2$ $[M+H]^+$ 308.1646, found 308.1651.



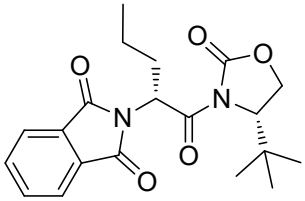
9c

2-(3-oxo-1,3-diphenylpropyl)isoindoline-1,3-dione (**9c**). White solid (38.3 mg, 54% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.98 (d, *J* = 7.5 Hz, 2H), 7.80-7.78 (m, 2H), 7.68-7.65 (m, 2H), 7.61

(d, J = 7.5 Hz, 2H), 7.57-7.54 (m, 1H), 7.46-7.43 (m, 2H), 7.37-7.34 (m, 2H), 7.30-7.27 (m, 1H), 6.08 (dd, J = 9.6 Hz, 5.2 Hz, 1H), 4.65-4.60 (m, 1H), 3.84-3.79 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 196.7, 168.3, 139.4, 136.4, 133.9, 133.4, 131.8, 128.8, 128.6, 128.1, 128.0, 127.8, 123.3, 50.4, 40.1. This compound was reported in the literature.[9]

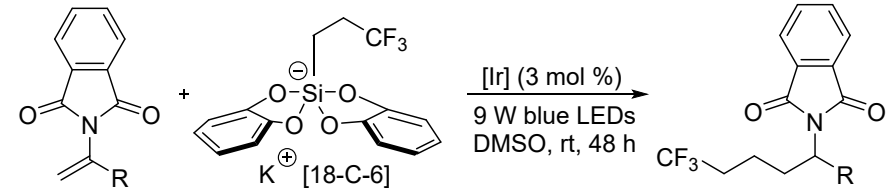


3-((S)-1-((S)-4-(tert-butyl)-2-oxooxazolidin-3-yl)-1-oxopentan-2-yl)isoindoline-1,3-dione (**11**). Light yellow syrups (41.7 mg, 56% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.87-7.86 (m, 2H), 7.73-7.72 (m, 2H), 5.72 (dd, J = 10.4 Hz, 4.4 Hz, 1H), 4.43-4.42 (m, 1H), 4.33-4.26 (m, 2H), 2.50-2.42 (m, 1H), 1.99-1.92 (m, 1H), 1.46-1.39 (m, 2H), 0.96-0.93 (m, 12H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 169.8, 167.9, 154.0, 134.1, 131.8, 123.4, 65.7, 62.2, 54.0, 35.9, 30.6, 25.7, 19.8, 13.4; HRMS (ESI) calculated for $C_{20}H_{24}N_2O_5Na$ $[M+Na]^+$ 395.1583, found 395.1586.



((R)-1-((S)-4-(tert-butyl)-2-oxooxazolidin-3-yl)-1-oxopentan-2-yl)isoindoline-1,3-dione (**11'**). Light yellow syrups (13.4 mg, 18% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.87-7.85 (m, 2H), 7.74-7.72 (m, 2H), 5.91 (dd, J = 11.6 Hz, 3.6 Hz, 1H), 4.47-4.45 (m, 1H), 4.33-4.32 (m, 2H), 2.78-2.70 (m, 1H), 2.20-2.14 (m, 1H), 1.49-1.42 (m, 2H), 0.99-0.94 (m, 12H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 170.1, 168.2, 153.8, 134.1, 131.8, 123.4, 65.7, 61.0, 54.8, 35.8, 30.4, 25.5, 20.1, 13.1; HRMS (ESI) calculated for $C_{20}H_{24}N_2O_5Na$ $[M+Na]^+$ 395.1583, found 395.1584.

4. Synthesis of 5i and 7f



R = CO_2Et or Ph

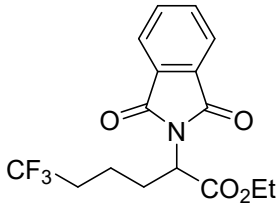
4i

5i: R = CO_2Et

7f: R = Ph

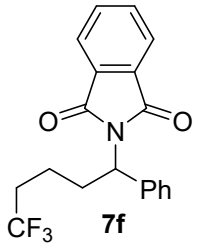
To an oven dried Schlenk tube equipped with stirring bar, Ir[dF(CF_3)ppy)]$_2$(dtbbpy)PF_6 (6.6 mg, 0.006 mmol, 0.03 equiv), 3,3,3-trifluoropropylsilicate **4i** (257.6 mg, 0.4 mmol, 2.0 equiv), the *N*-vinylimide **1a** or **6a** (0.2 mmol, 1.0 equiv) were added. The tube was evacuated and filled with nitrogen for 3 times. Then the tube was charged with degassed DMSO (6.0 mL), and irradiated with a 9 W blue LEDs strip spiraled within a bowel for 24 h. To the reaction mixture was added another portion alkyl silicate **4i** (128.8 mg, 0.2 mmol, 1.0 equiv) under nitrogen and stirred for an additional 24 h. After the reaction was complete, the reaction solution was diluted with saturated Na_2CO_3 aqueous solution (10 mL), and organic material was extracted with EtOAc (3 x 10 mL). The organic layer was washed with brine, dried over anhydrous Na_2SO_4, filtered, and evaporated.

The residue was purified by flash column chromatography to afford the product **5i** or **7f**.



5i

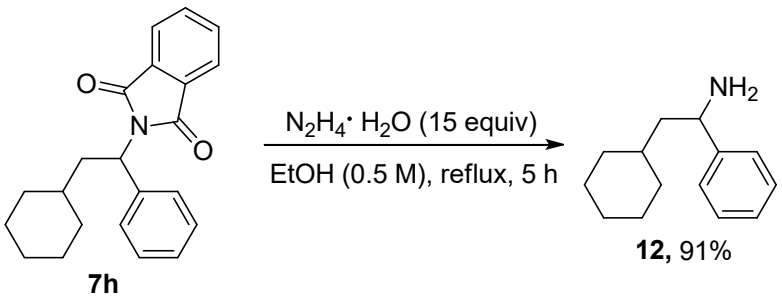
Ethyl 2-(1,3-dioxoisoindolin-2-yl)-6,6,6-trifluorohexanoate (**5i**). Light yellow oil (39.1 mg, 57% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.90-7.87 (m, 2H), 7.78-7.75 (m, 2H), 4.84-4.81 (m, 1H), 4.25-4.16 (m, 2H), 2.35-2.28 (m, 2H), 2.21-2.04 (m, 2H), 1.65-1.54 (m, 2H), 1.22 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 168.7, 167.7, 134.3, 131.6, 126.7 (d, *J* = 274.5 Hz), 123.6, 62.0, 51.6, 33.1 (q, *J* = 28.9 Hz), 27.8, 19.0 (d, *J* = 3.1 Hz), 14.0; ^{19}F NMR (470 MHz, $CDCl_3$): δ -66.3; HRMS (ESI) calculated for $C_{16}H_{17}F_3NO_4$ $[M+H]^+$ 344.1110, found 344.1113.



7f

2-(5,5,5-Trifluoro-1-phenylpentyl)isoindoline-1,3-dione (**7f**). White solid (27.1 mg, 39% yield), m.p. 83.5-84.2 °C. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.82-7.80 (m, 2H), 7.71-7.69 (m, 2H), 7.54 (d, *J* = 7.4 Hz, 2H), 7.35-7.26 (m, 3H), 5.33 (dd, *J* = 9.6 Hz, 6.8 Hz, 1H), 2.71-2.64 (m, 1H), 2.41-2.34 (m, 1H), 2.21-2.11 (m, 2H), 1.65-1.57 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 168.2, 139.0, 134.1, 131.7, 128.6, 128.0, 126.9 (d, *J* = 274.7 Hz) 123.2, 54.5, 33.2 (q, *J* = 28.7 Hz), 30.0, 19.6 (d, *J* = 3.0 Hz); ^{19}F NMR (470 MHz, $CDCl_3$): δ -66.3; HRMS (ESI) calculated for $C_{19}H_{17}F_3NO_2$ $[M+H]^+$ 348.1211, found 348.1212.

5. Procedure for the deprotection of 7h



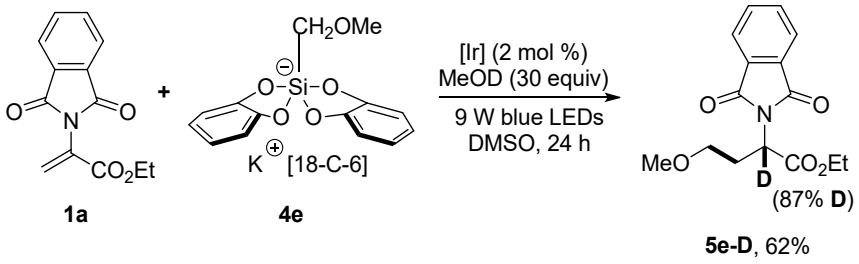
7h **12,** 91%

To a stirred solution of 2-(2-cyclohexyl-1-phenylethyl)isoindoline-1,3-dione **7h** (66.6 mg, 0.2 mmol) in EtOH (0.4 mL) was added hydrazine hydrate (w=80%, 0.18 mL, 15 equiv). The mixture was refluxed for 5 h. After complete, the solvent was evaporated in vacuo and the residue was purified by flash column chromatography over neutral Al_2O_3 to give the product 2-cyclohexyl-1-phenylethanamine **12** (36.9 mg, 91% yield) as a light yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.37-7.29 (m, 4H), 7.28-7.25 (m, 1H), 4.02 (t, *J* = 7.2 Hz, 1H), 1.78-1.53 (m, 8H), 1.23-1.14 (m, 3H), 1.00-0.90 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 147.0, 128.4, 126.8, 126.2, 53.3, 47.4,

34.5, 33.7, 33.1, 26.6, 26.2, 26.1. The spectral data match those previously reported.[10]

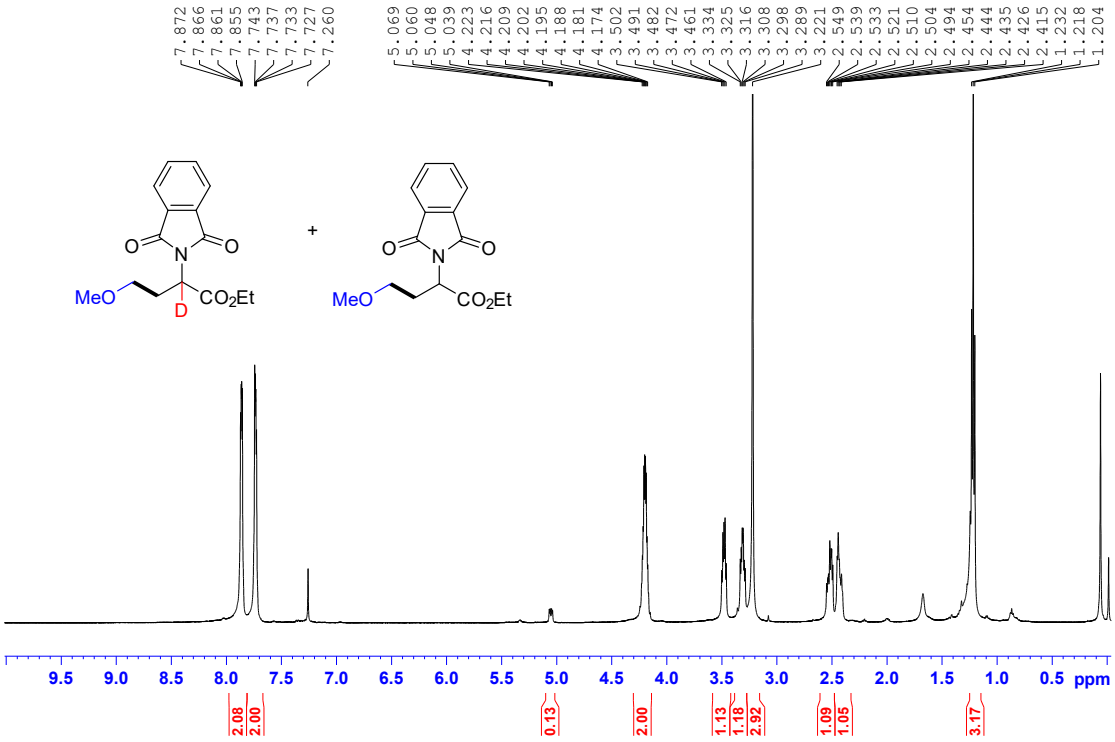
6. Deuteration experiments

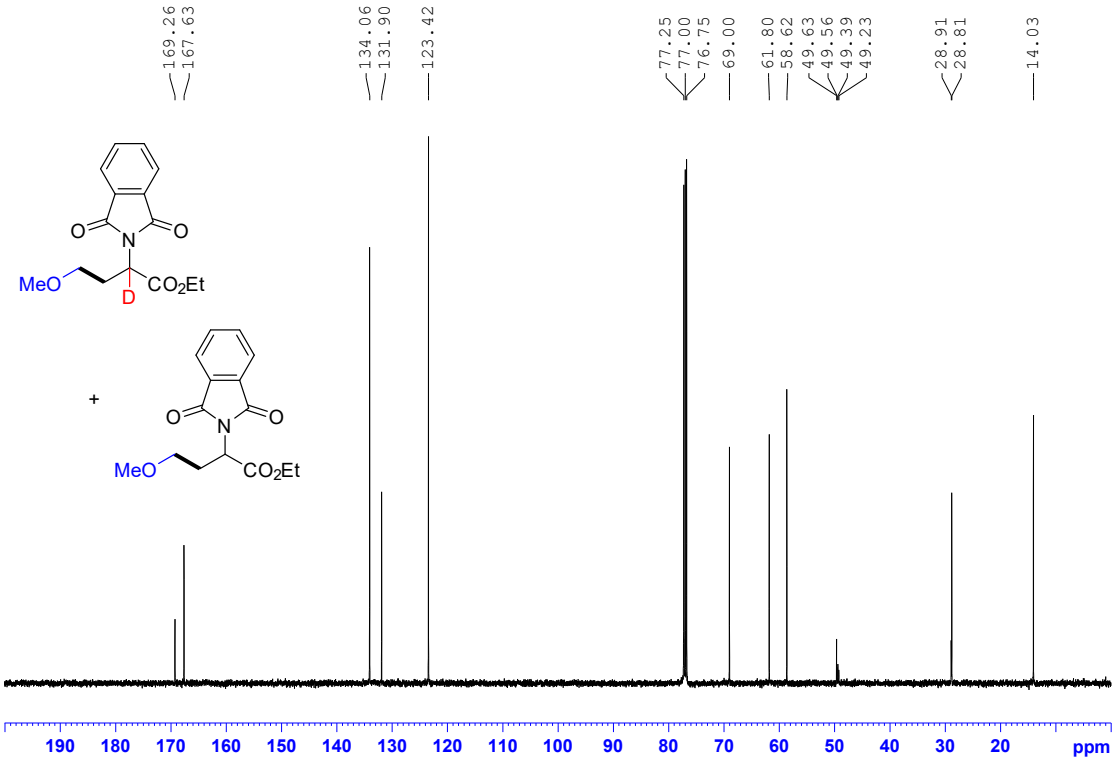
To an oven dried Schlenk tube equipped with stirring bar, $Ir[dF(CF_3)ppy)]_2(dtbbpy)PF_6$ (4.5 mg, 0.004 mmol), radical precursor **4e** or **8b** (0.4 mmol), the *N*-vinylimide **1a** or **6a** (0.2 mmol) were added. The tube was evacuated and filled with nitrogen for 3 times. Then the tube was charged with degassed DMSO (6.0 mL) and CD_3OD (0.18 mL, 6 mmol), and irradiated with a 9 W blue LEDs strip spiraled within a bowel for 24 h (cooling with a fan). After the reaction was complete, the reaction solution was diluted with saturated Na_2CO_3 aqueous solution (10 mL), and organic material was extracted with EtOAc (3 x 10 mL). The organic layer was washed with brine, dried over Na_2SO_4, filtered, and evaporated. Flash column chromatography on silica gel to give the mixture product.

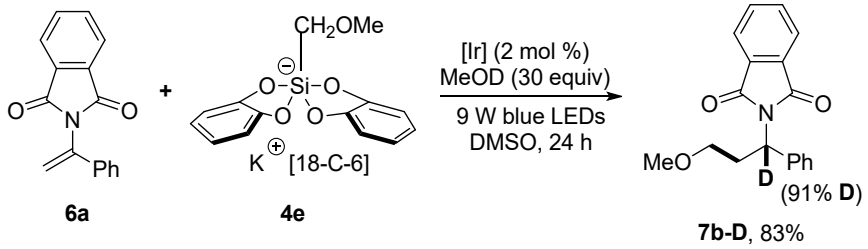


1a **4e** **5e-D**, 62%

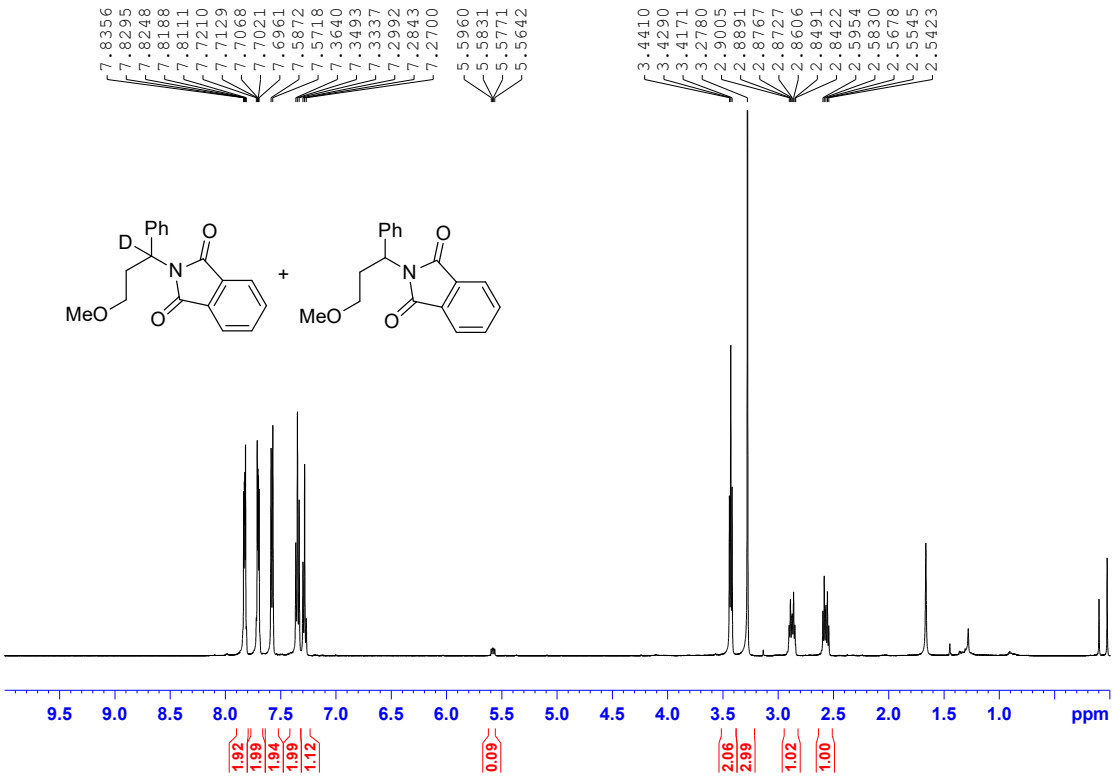
Compound **5e-D** (mixture, light yellow oil). ^1H NMR (500 MHz, $CDCl_3$): δ 7.87-7.86 (m, 2H), 7.74-7.73 (m, 2H), 4.22-4.17 (m, 2H), 3.50-3.46 (m, 1H), 3.33-3.29 (m, 1H), 3.22 (s, 3H), 2.55-2.49 (m, 1H), 2.45-2.41 (m, 1H), 1.22 (d, J = 7.1 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 169.3, 167.6, 134.1, 131.9, 123.4, 69.0, 61.8, 58.6, 49.4 (t, J = 20.6 Hz), 28.8, 14.0; HRMS (ESI) calculated for $C_{15}H_{16}DNO_5Na$ $[M+Na]^+$ 315.1062, found 315.1061.







Compound **7b-D** (mixture, light yellow solid). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.84-7.81 (m, 2H), 7.72-7.70 (m, 2H), 7.58 (d, J = 7.7 Hz, 2H), 7.36-7.33 (m, 2H), 7.30-7.27 (m, 1H), 3.44-3.42 (t, J = 6.0 Hz, 2H), 3.28 (s, 3H), 2.90-2.84 (m, 1H), 2.60-2.54 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 168.4, 139.4, 133.9, 131.9, 128.5, 128.2, 127.8, 123.2, 69.6, 58.7, 51.7 (m), 30.9; HRMS (ESI) calculated for $C_{18}H_{16}DNO_3$ $[M+Na]^+$ 319.1163, found 319.1171.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7.8248
7.8188
7.8111
7.7210
7.7129
7.7068
7.7021
7.6961
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7.5718
7.3640
7.3493
7.3337
7.2992
7.2843
7.2700
5.5960
5.5831
5.5771
5.5642
3.4410
3.4290
3.4171
3.2780
2.9005
2.8891
2.8767
2.8727
2.8606
2.8491
2.8422
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2.5545
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Ph
D
O
N
MeO
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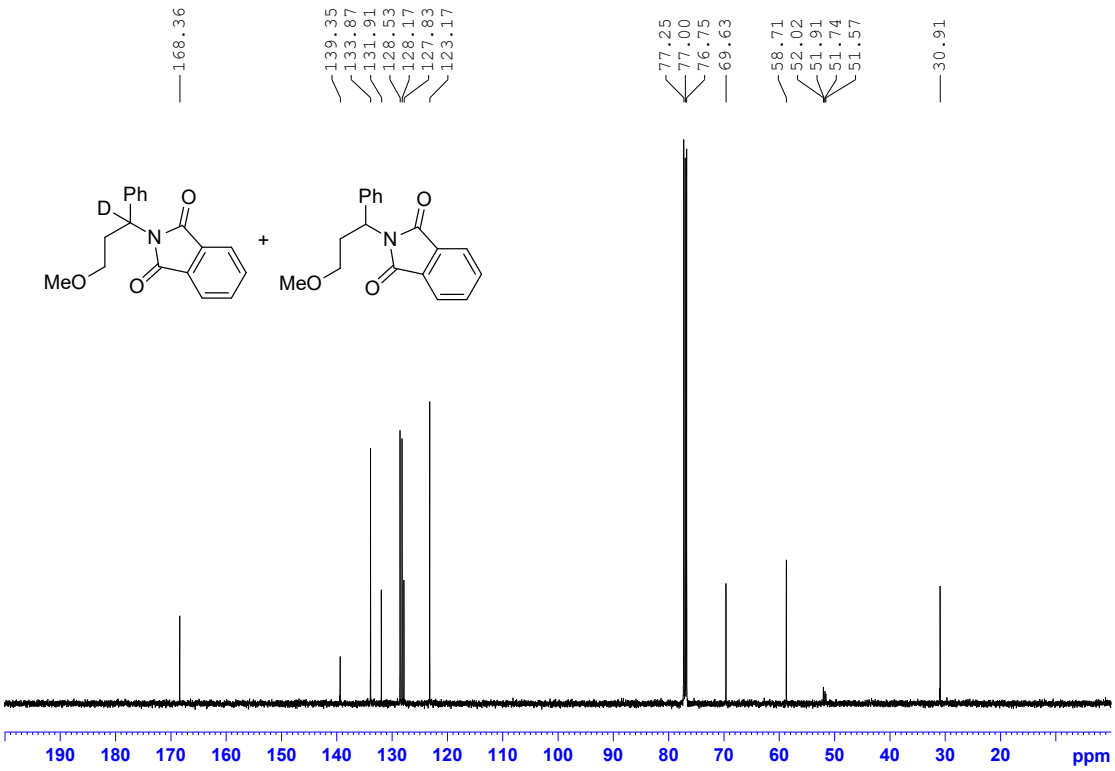
1.92
1.99
1.94
1.99
1.12
0.09
2.06
2.99
1.02
1.00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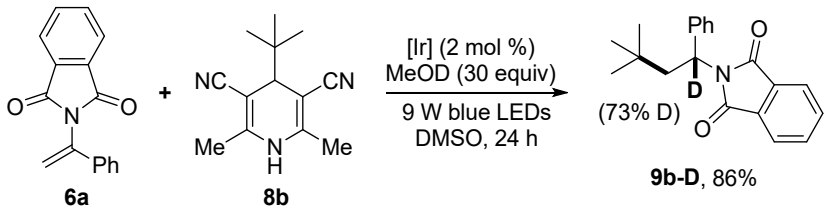
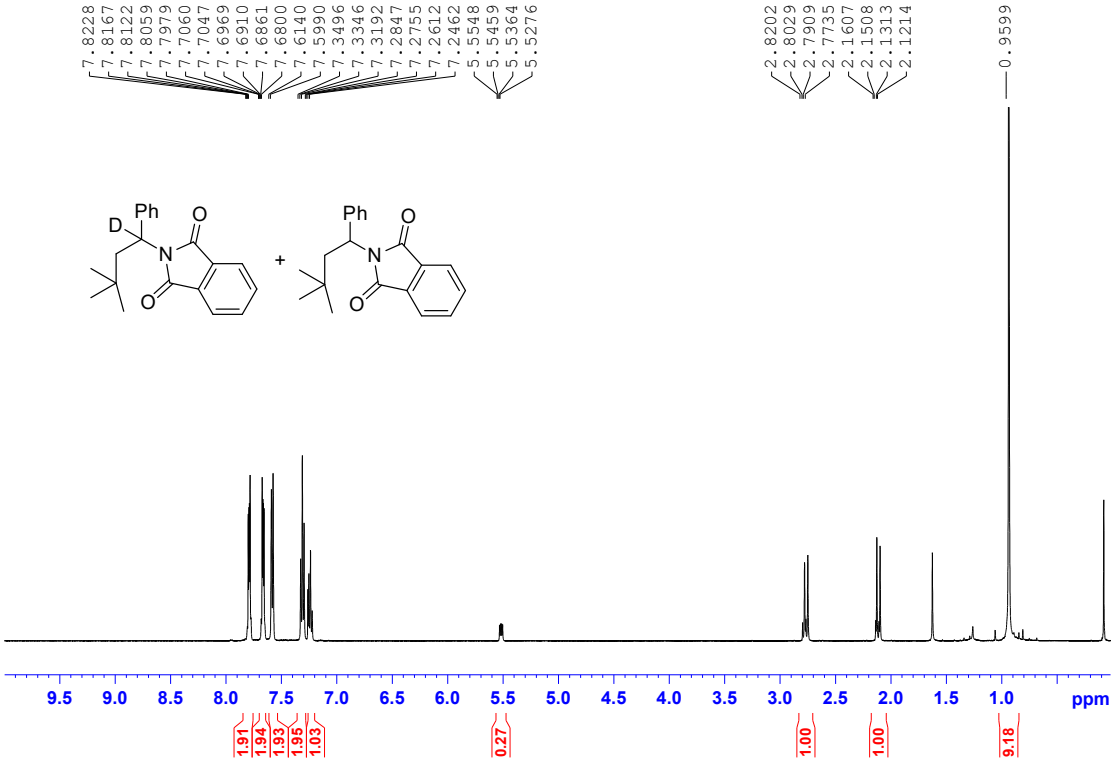
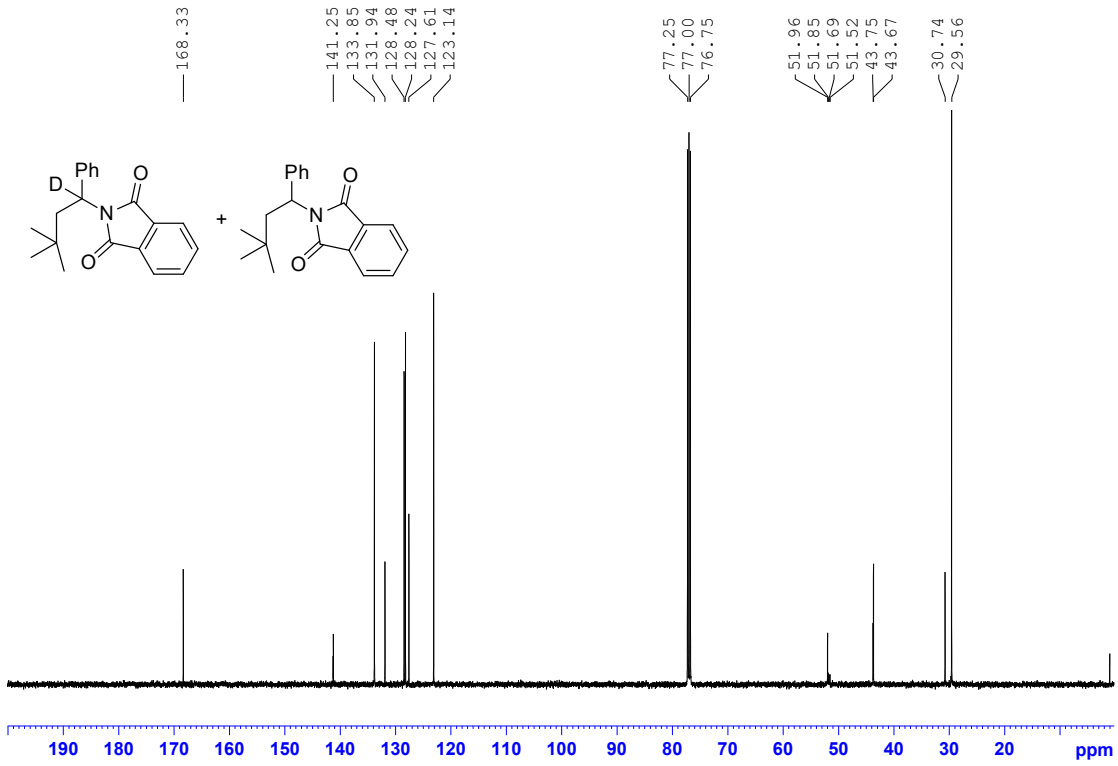
139.35
133.87
131.91
128.53
128.17
127.83
123.17
77.25
77.00
76.75
69.63
58.71
52.02
51.91
51.74
51.57
30.91
Ph
D
O
N
MeO
+
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm



Compound **9b-D** (mixture, white solid). ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.82-7.80 (m, 2H), 7.71-7.68 (m, 2H), 7.61 (d, *J* = 7.5 Hz, 2H), 7.35-7.32 (m, 2H), 7.25-7.28 (m, 1H), 2.82-2.77 (m, 1H), 2.16-2.12 (m, 1H), 0.96 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 168.3, 141.3, 133.8, 131.9, 128.5, 128.2, 127.6, 123.1, 51.7, 43.7, 30.7, 29.6; HRMS (ESI) calculated for $C_{20}H_{20}DNO_2$ $[M+Na]^+$ 331.1527, found 331.1535.

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141.25
133.85
131.94
128.48
128.24
127.61
123.14
77.25
77.00
76.75
51.96
51.85
51.69
51.52
43.75
43.67
30.74
29.56
D
Ph
O
N
O
+
Ph
O
N
O
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

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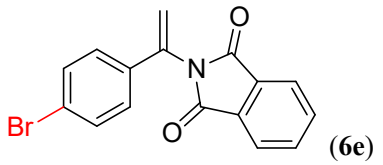
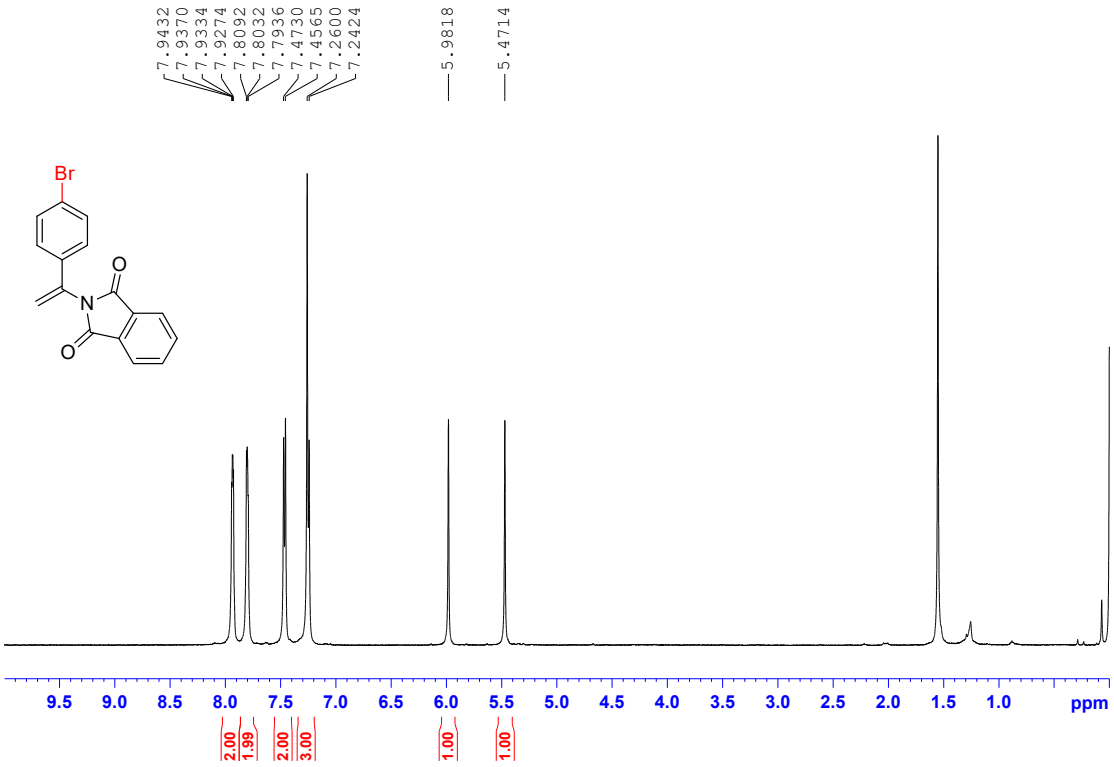
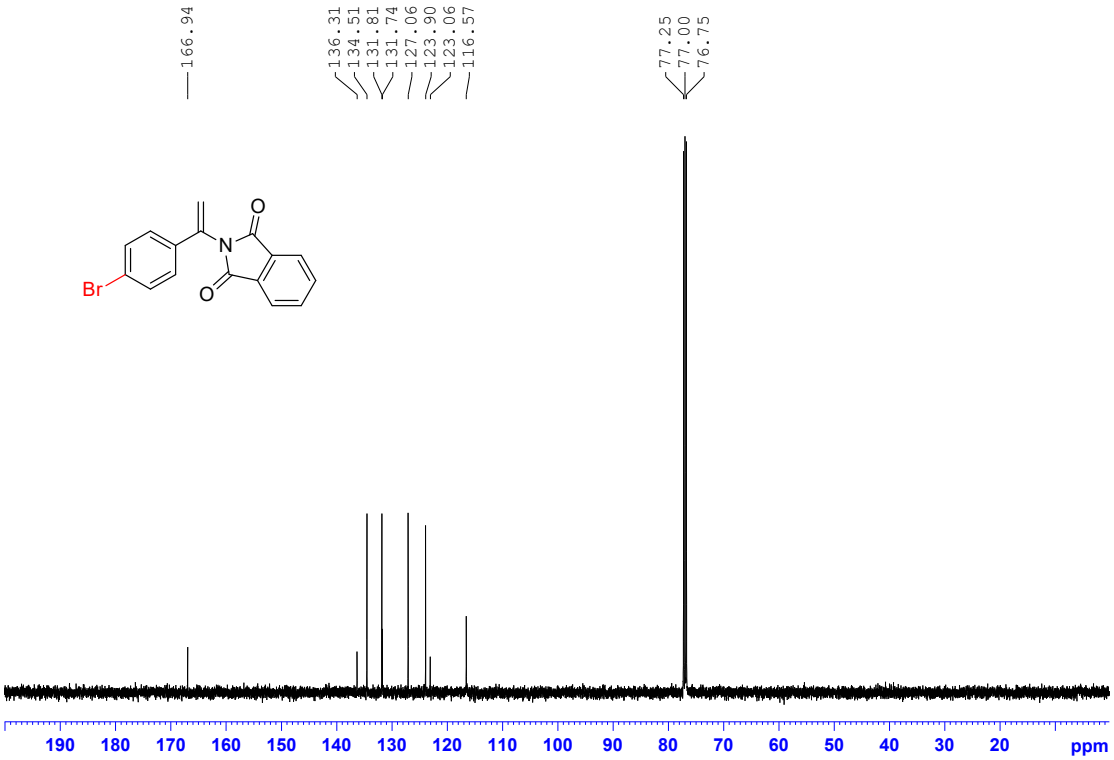
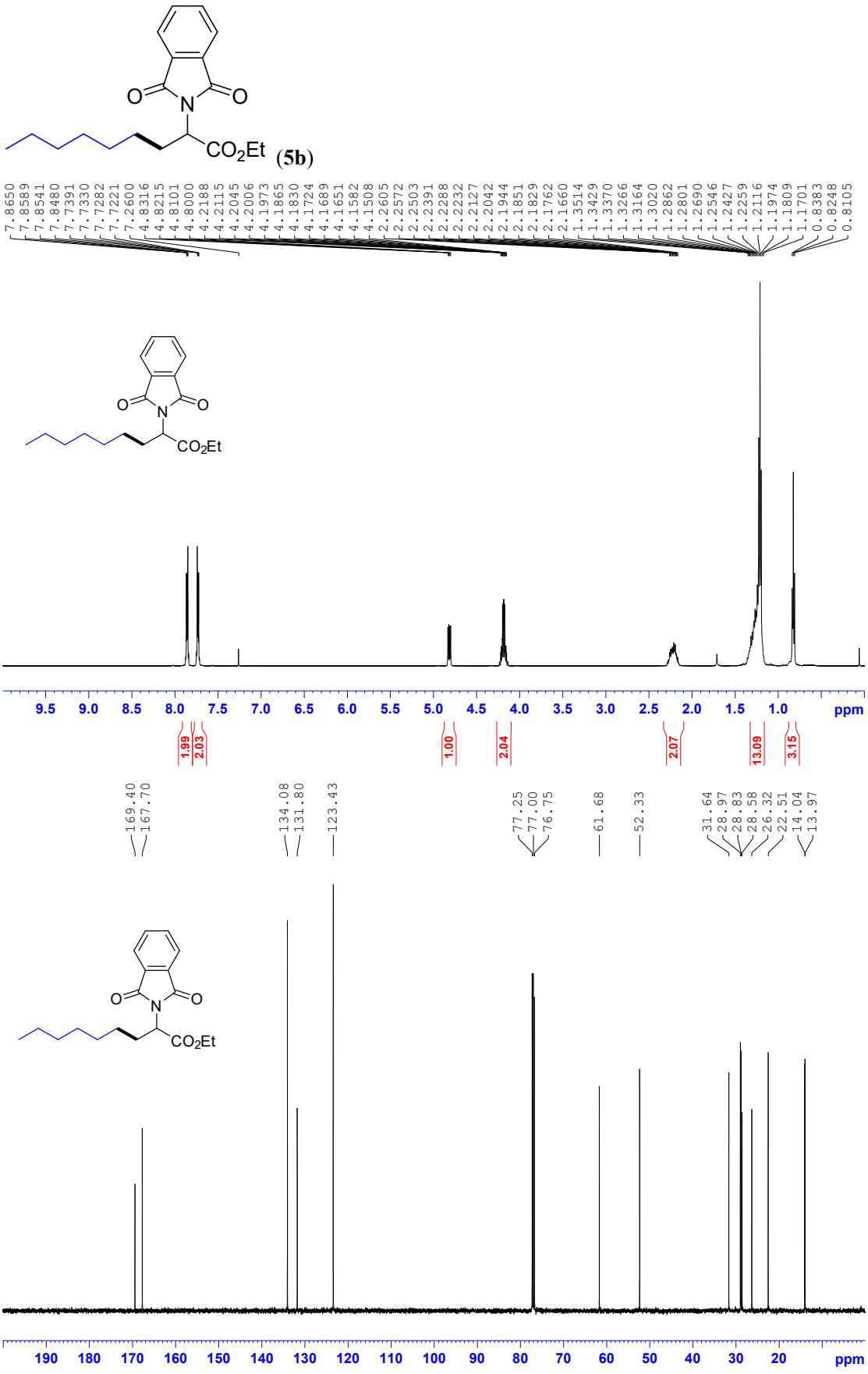
NMR spectra of new compounds



(6e)



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N
O
CO2Et
(5b)
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7.7221
7.2600
4.8316
4.8215
4.8101
4.8000
4.2188
4.2115
4.2045
4.2006
4.1973
4.1865
4.1830
4.1724
4.1689
4.1651
4.1582
4.1508
4.2605
2.2572
2.2503
2.2391
2.2288
2.2232
2.2127
2.2042
2.1944
2.1851
2.1829
2.1762
2.1660
1.3514
1.3429
1.3370
1.3266
1.3164
1.3020
1.2862
1.2801
1.2690
1.2546
1.2427
1.2259
1.2116
1.1974
1.1809
1.1701
0.8383
0.8248
0.8105
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 ppm
1.99
2.03
1.00
2.04
2.07
13.09
3.15
169.40
167.70
134.08
131.80
123.43
77.25
77.00
76.75
61.68
52.33
31.64
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28.58
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22.51
14.04
13.97
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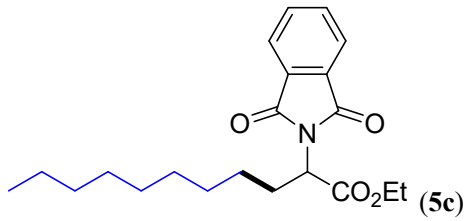
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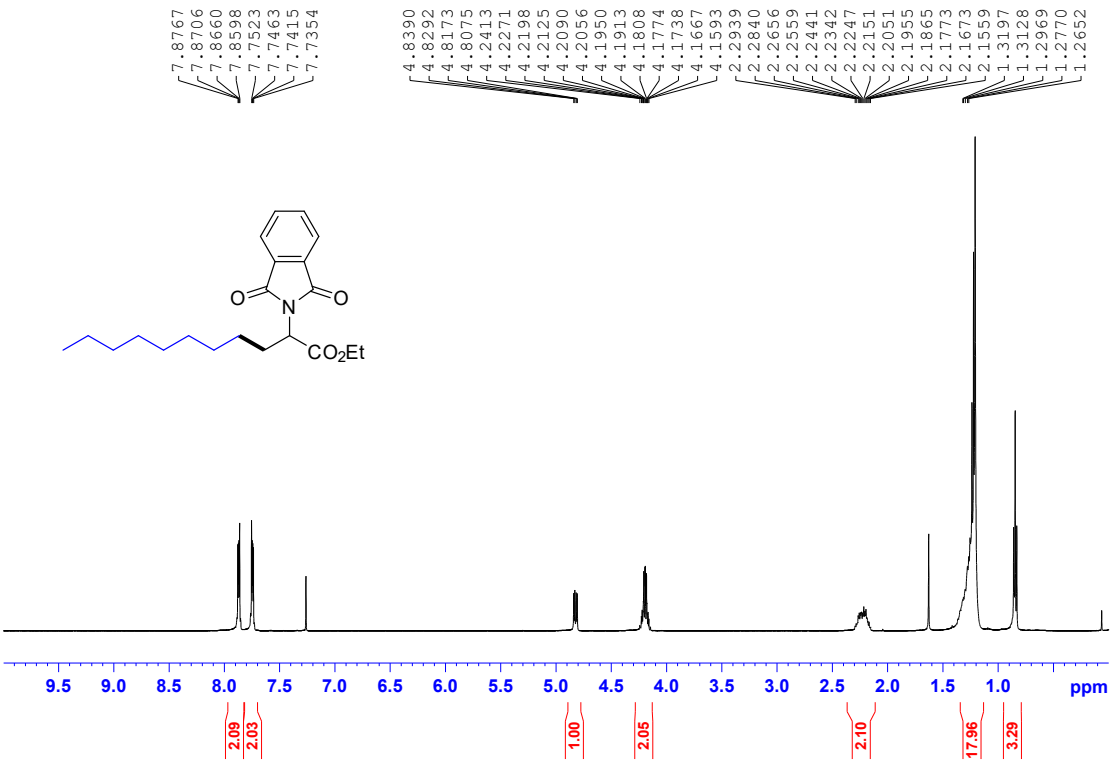
N
O
CO2Et

(5c)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4.2413
4.2271
4.2198
4.2125
4.2090
4.2056
4.1950
4.1913
4.1808
4.1774
4.1738
4.1667
4.1593
2.2939
2.2840
2.2656
2.2559
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2.2051
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2.1865
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2.1673
2.1559
1.3197
1.3128
1.2969
1.2770
1.2652
O
N
O
CO2Et
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
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2.0
1.5
1.0
ppm
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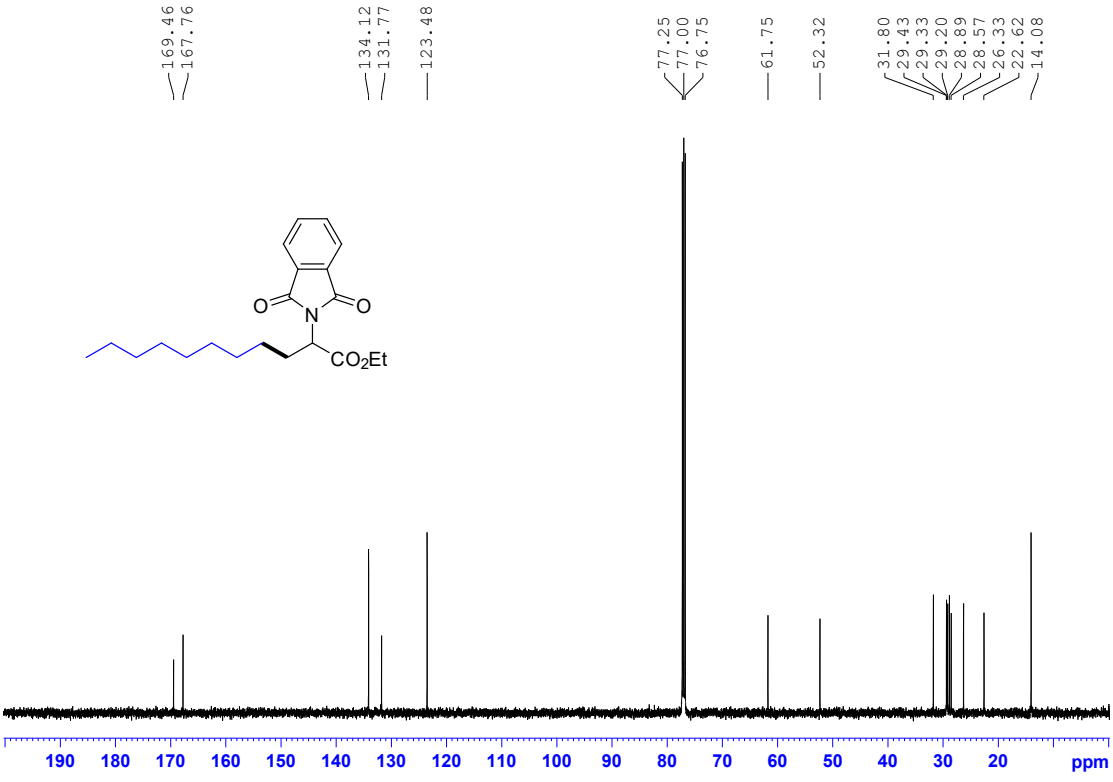
2.03
1.00
2.05
2.10
17.96
3.29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52.32
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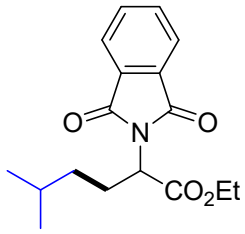
29.33
29.20
28.89
28.57
26.33
22.62
14.08
O
N
O
CO2Et
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm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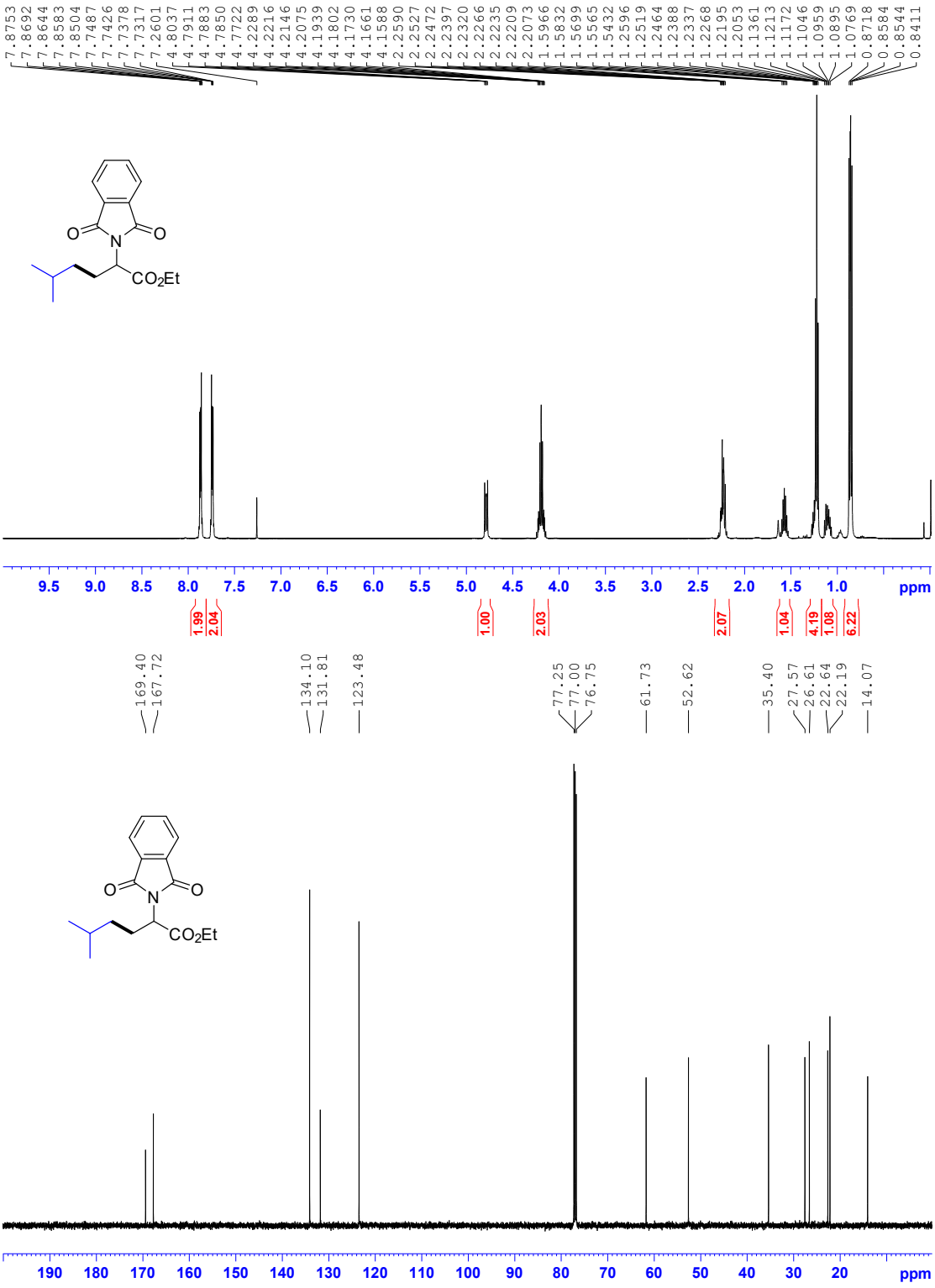
N
O
CO2Et

(5d)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4.1802
4.1730
4.1661
4.1588
2.2590
2.2527
2.2472
2.2397
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2.2266
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2.2073
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1.5832
1.5699
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1.5432
1.2596
1.2519
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1.2337
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1.2053
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1.1213
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1.1046
1.0959
1.0895
1.0769
0.8718
0.8584
0.8544
0.8411
O
N
O
CO2Et
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 ppm
1.99
2.04
1.00
2.03
2.07
1.04
4.19
1.08
6.22
169.40
167.72
134.10
131.81
123.48
77.25
77.00
76.75
61.73
52.62
35.40
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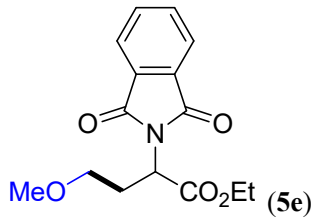
22.64
22.19
14.07
O
N
O
CO2Et
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm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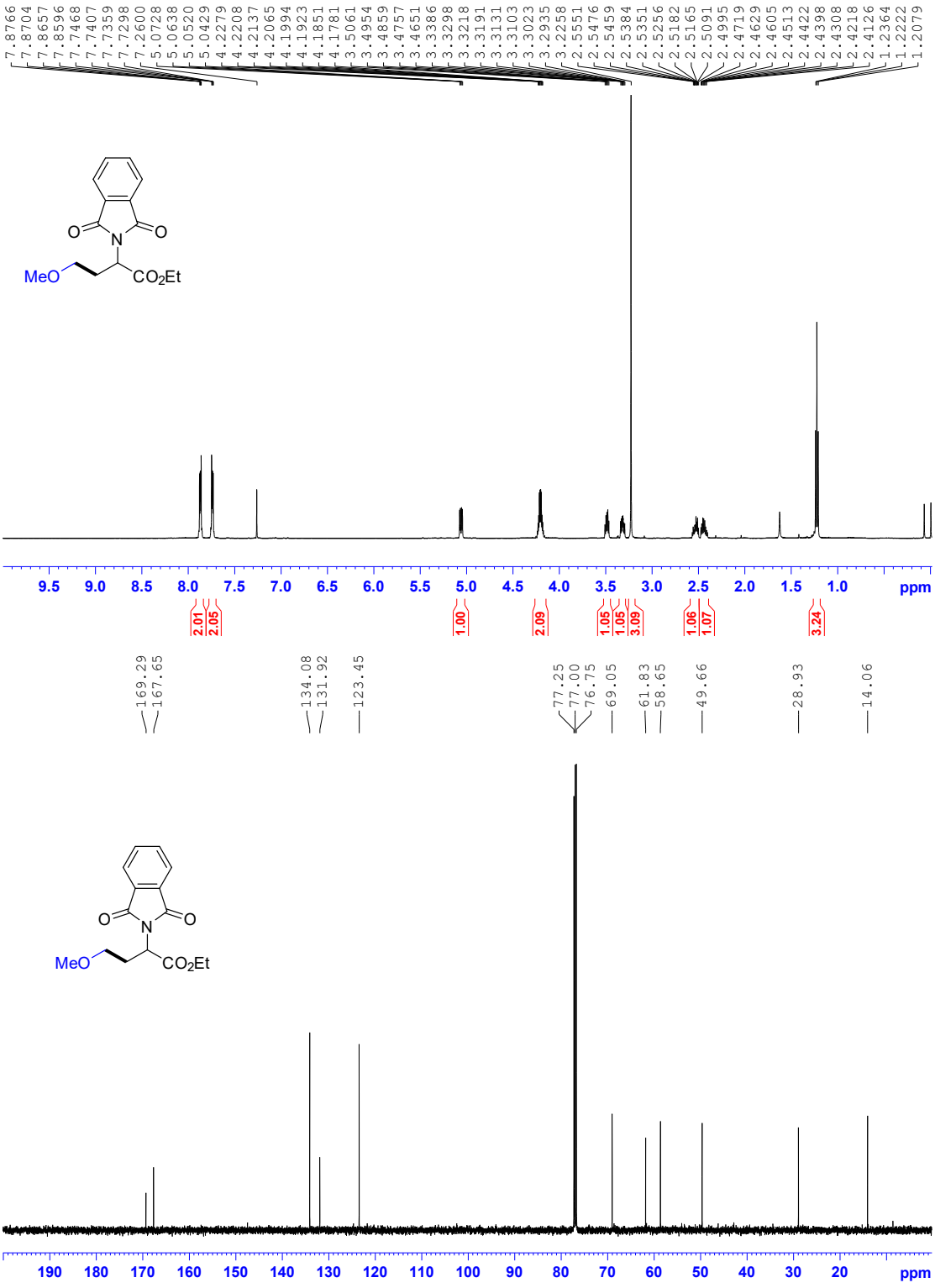
N
O
MeO
CO2Et

(5e)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1.2079
O
N
O
MeO
CO2Et
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 ppm
2.01
2.05
1.00
2.09
1.05
1.05
3.09
1.06
1.07
3.24
169.29
167.65
134.08
131.92
123.45
77.25
77.00
76.75
69.05
61.83
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28.93
14.06
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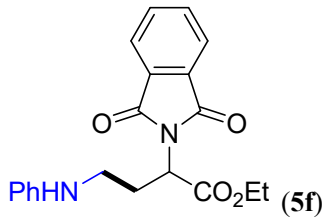
N
O
MeO
CO2Et
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm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O
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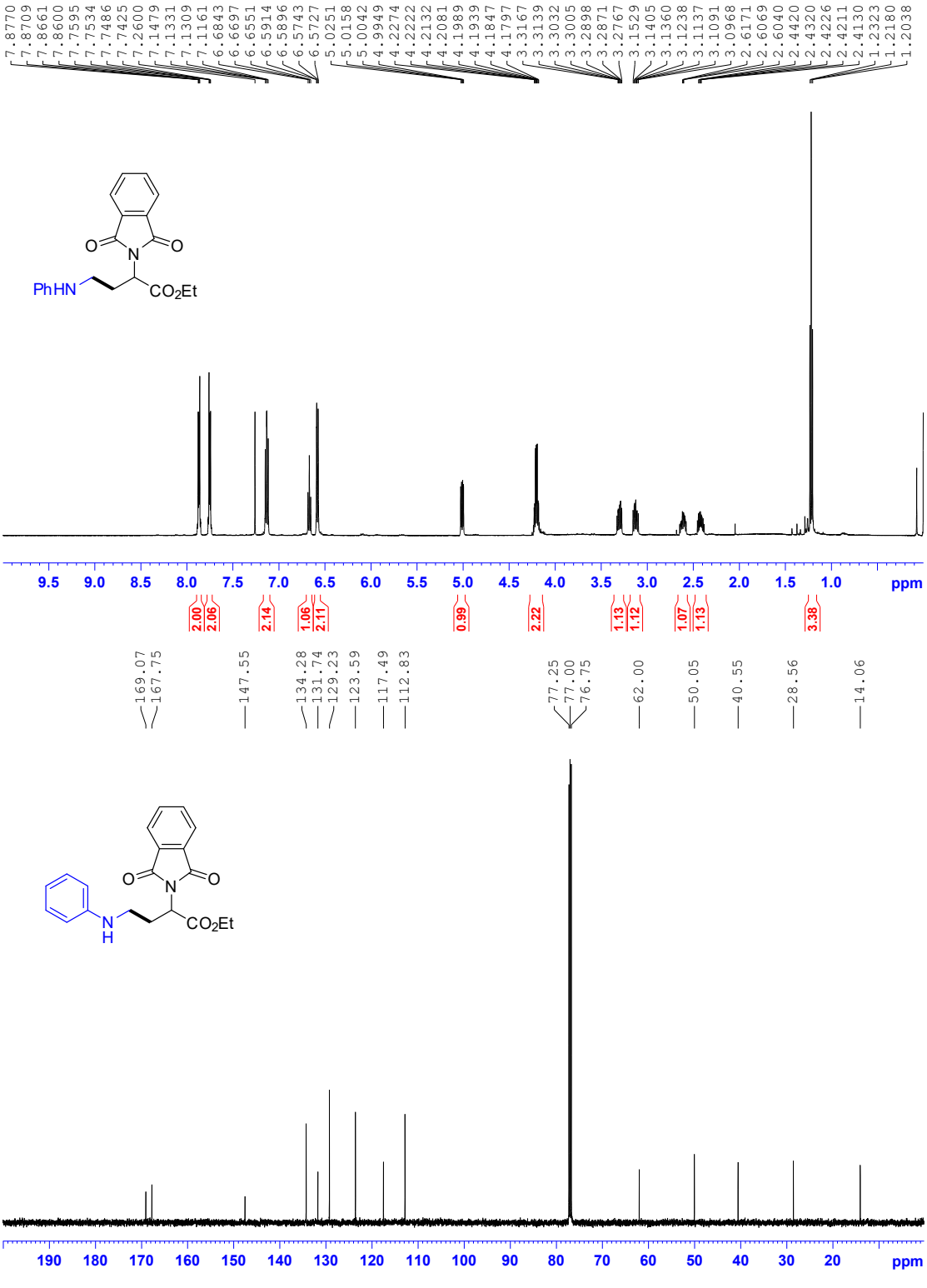
(5f)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PhHN
CO2Et
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2.00
2.06
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1.06
2.11
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1.13
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167.75
147.55
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117.49
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CO2Et
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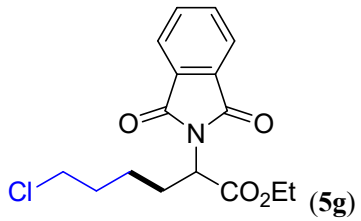
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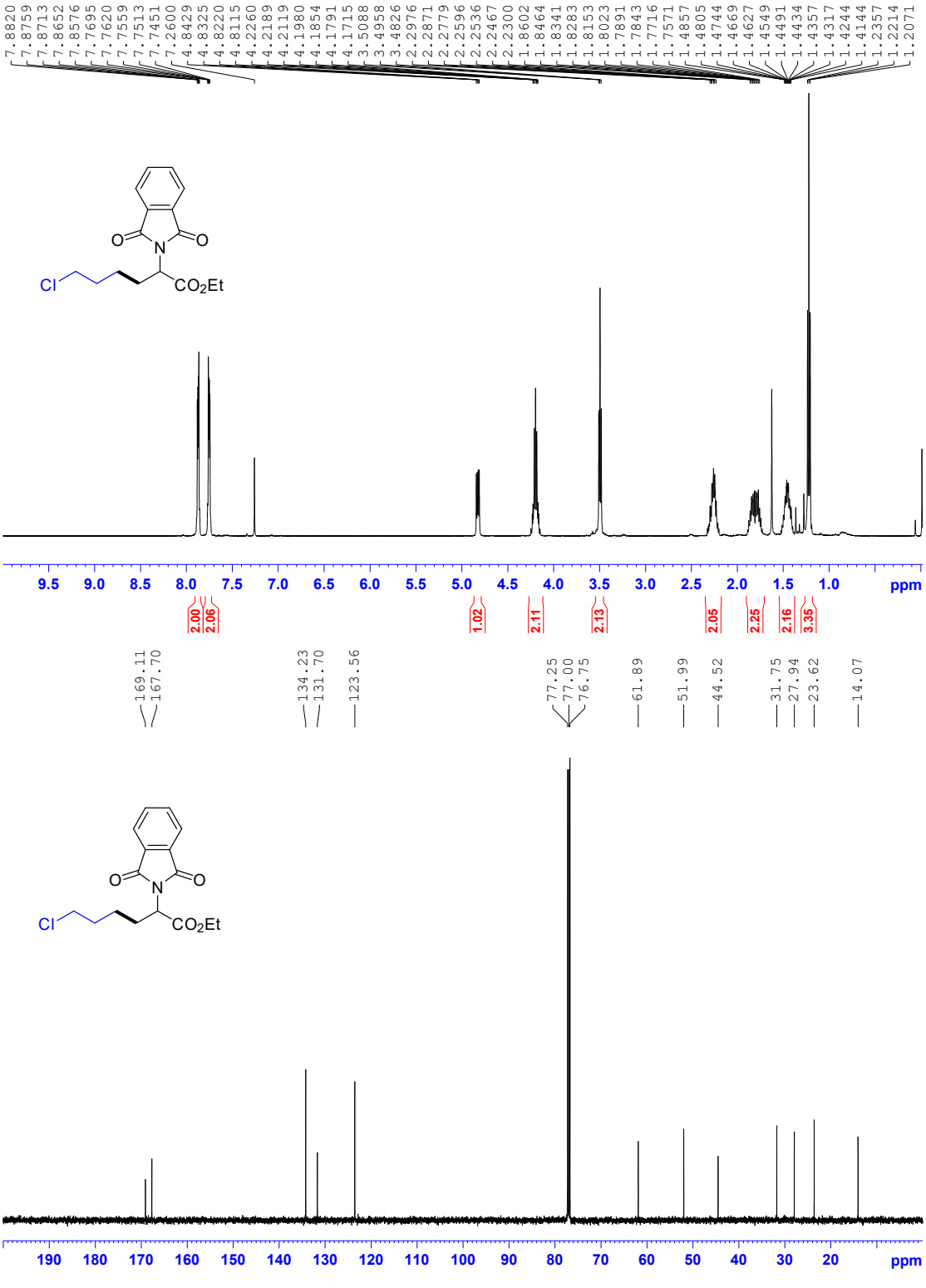
O
Cl
CO2Et

(5g)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7.2600
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1.4434
1.4357
1.4317
1.4244
1.4144
1.2357
1.2214
1.2071
O
N
O
Cl
CO2Et
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 ppm
2.00
2.06
1.02
2.11
2.13
2.05
2.25
2.16
3.35
169.11
167.70
134.23
131.70
123.56
77.25
77.00
76.75
61.89
51.99
44.52
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23.62
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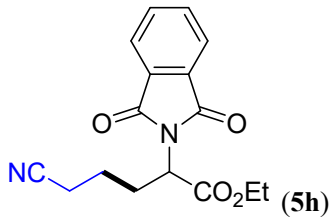
O
Cl
CO2Et
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm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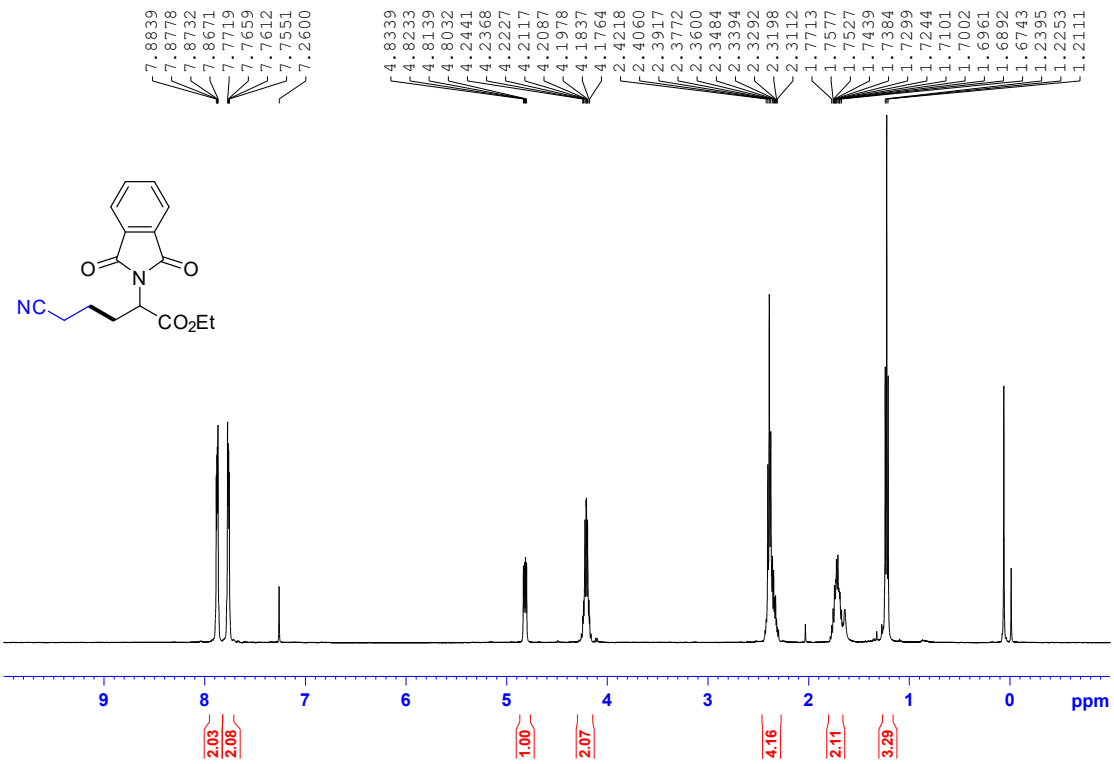
(5h)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4.1764
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2.4060
2.3917
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2.3600
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2.3292
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1.7244
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N
O
NC
CO2Et
9
8
7
6
5
4
3
2
1
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ppm
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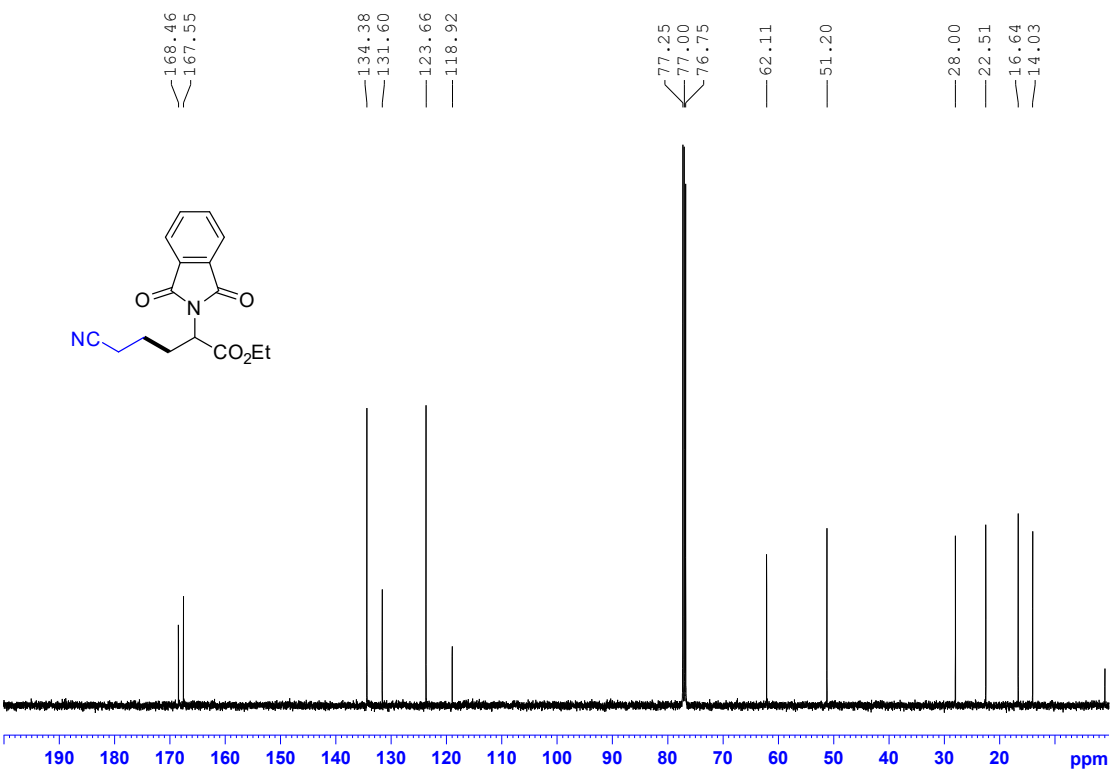
2.08
1.00
2.07
4.16
2.11
3.29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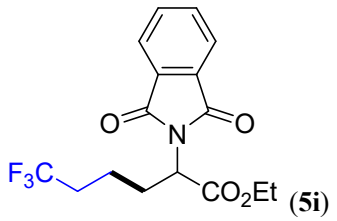
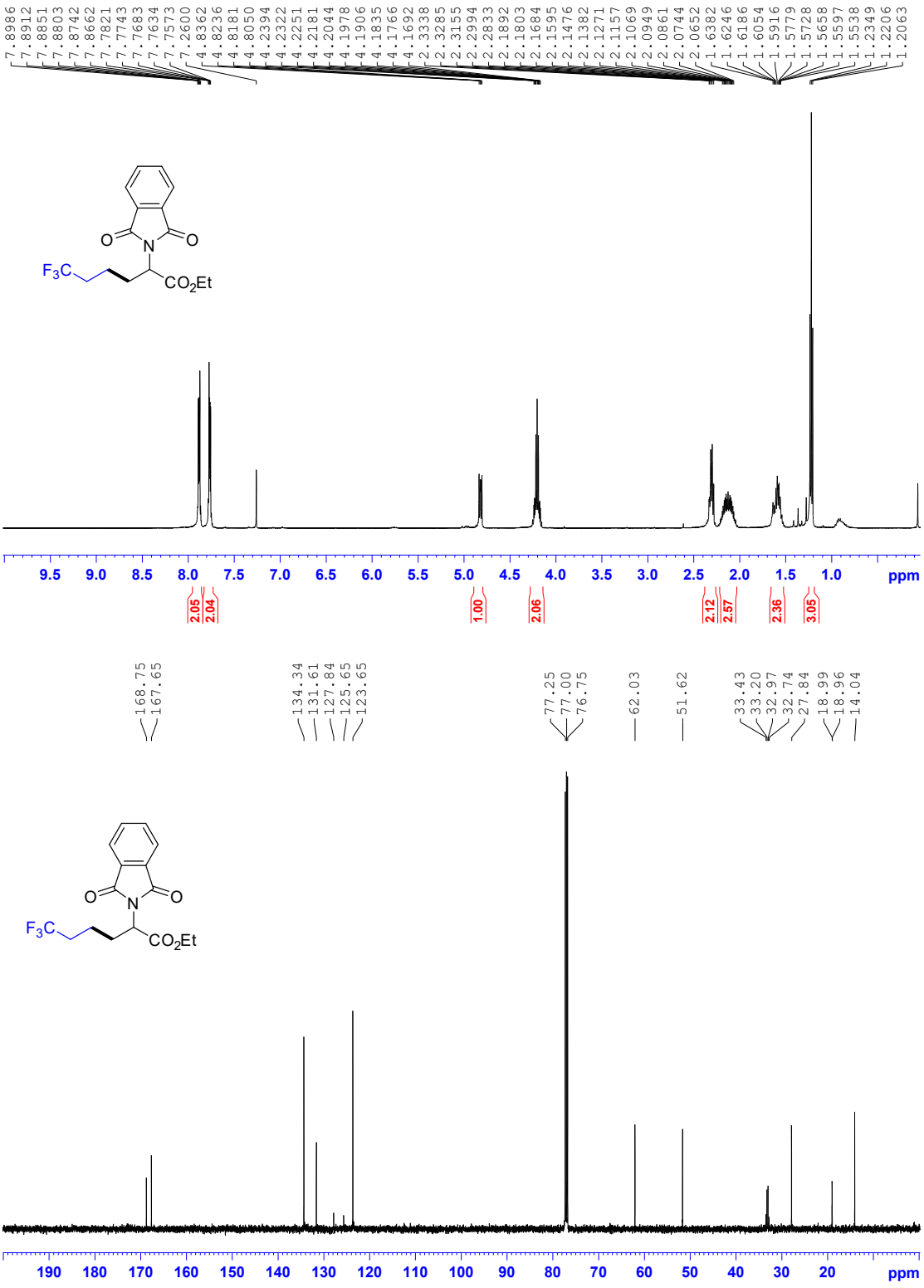
167.55
134.38
131.60
123.66
118.92
77.25
77.00
76.75
62.11
51.20
28.00
22.51
16.64
14.03
O
N
O
NC
CO2Et
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm


O
N
O
F3C
CO2Et

(5i)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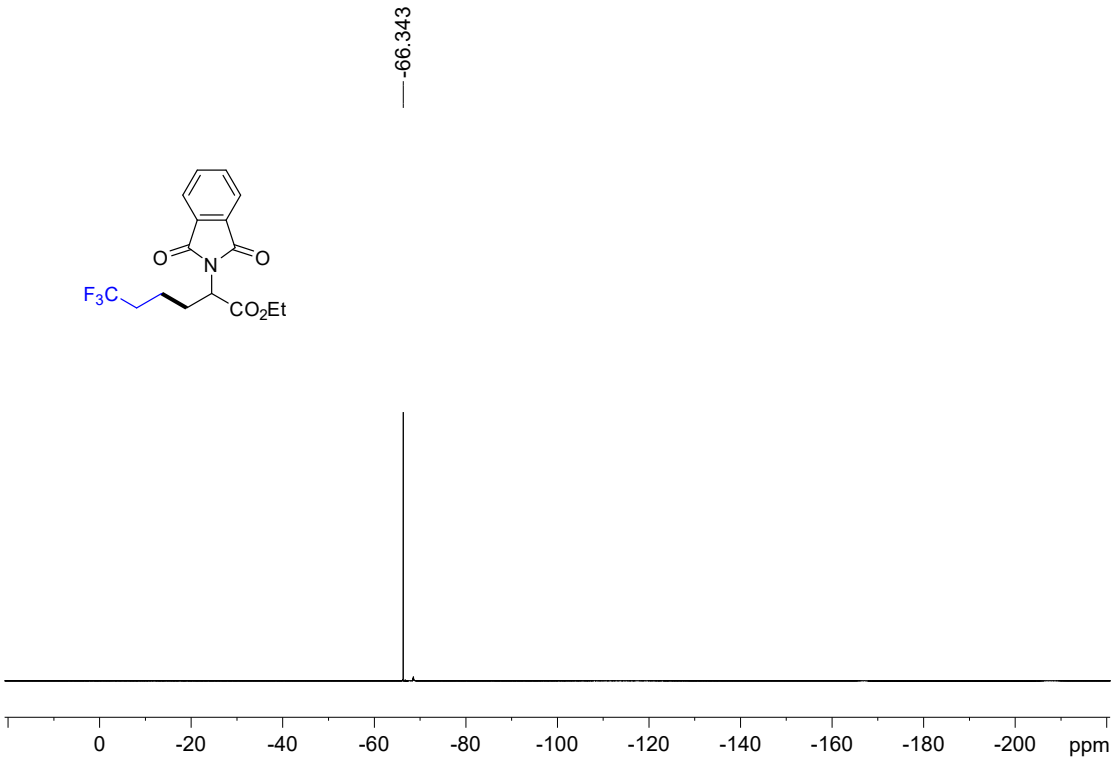
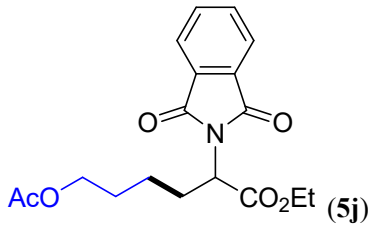
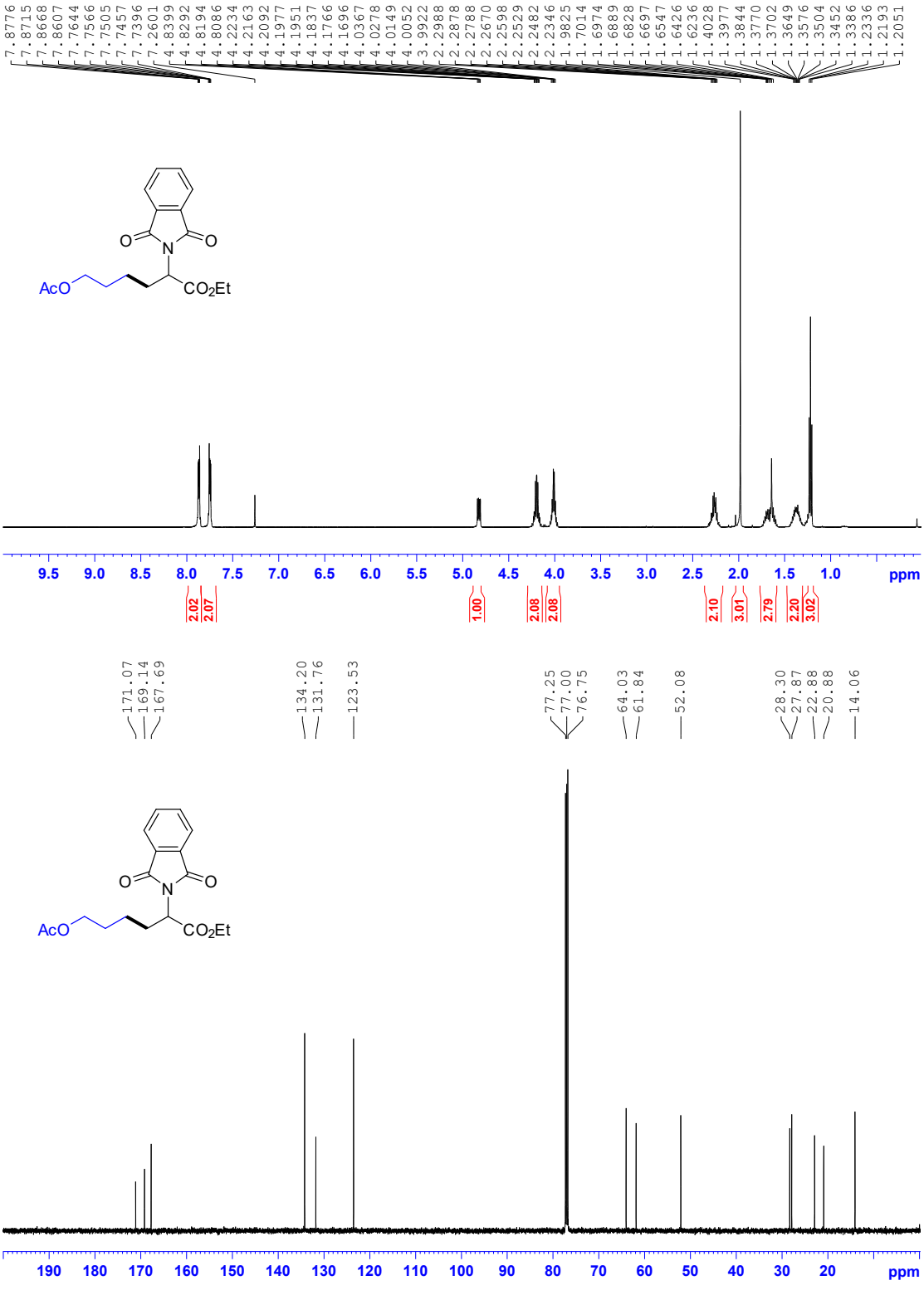
7.8662
7.7821
7.7743
7.7683
7.7634
7.7573
7.2600
4.8362
4.8236
4.8181
4.8050
4.2394
4.2322
4.2251
4.2181
4.2044
4.1978
4.1906
4.1835
4.1766
4.1692
2.3338
2.3285
2.3155
2.2994
2.2833
2.1892
2.1803
2.1684
2.1595
2.1476
2.1382
2.1271
2.1157
2.1069
2.0949
2.0861
2.0744
2.0652
1.6382
1.6246
1.6186
1.6054
1.5916
1.5779
1.5728
1.5658
1.5597
1.5538
1.2349
1.2206
1.2063
O
N
O
F3C
CO2Et
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
ppm
2.05
2.04
1.00
2.06
2.12
2.57
2.36
3.05
168.75
167.65
134.34
131.61
127.84
125.65
123.65
77.25
77.00
76.75
62.03
51.62
33.43
33.20
32.97
32.74
27.84
18.99
18.96
14.04
O
N
O
F3C
CO2Et
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm


-66.343
O
N
O
F3C
CO2Et
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
-200
ppm


O
N
O
AcO
CO2Et

(5j)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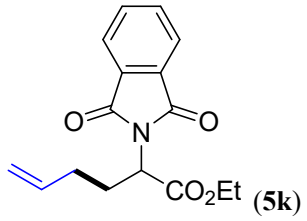
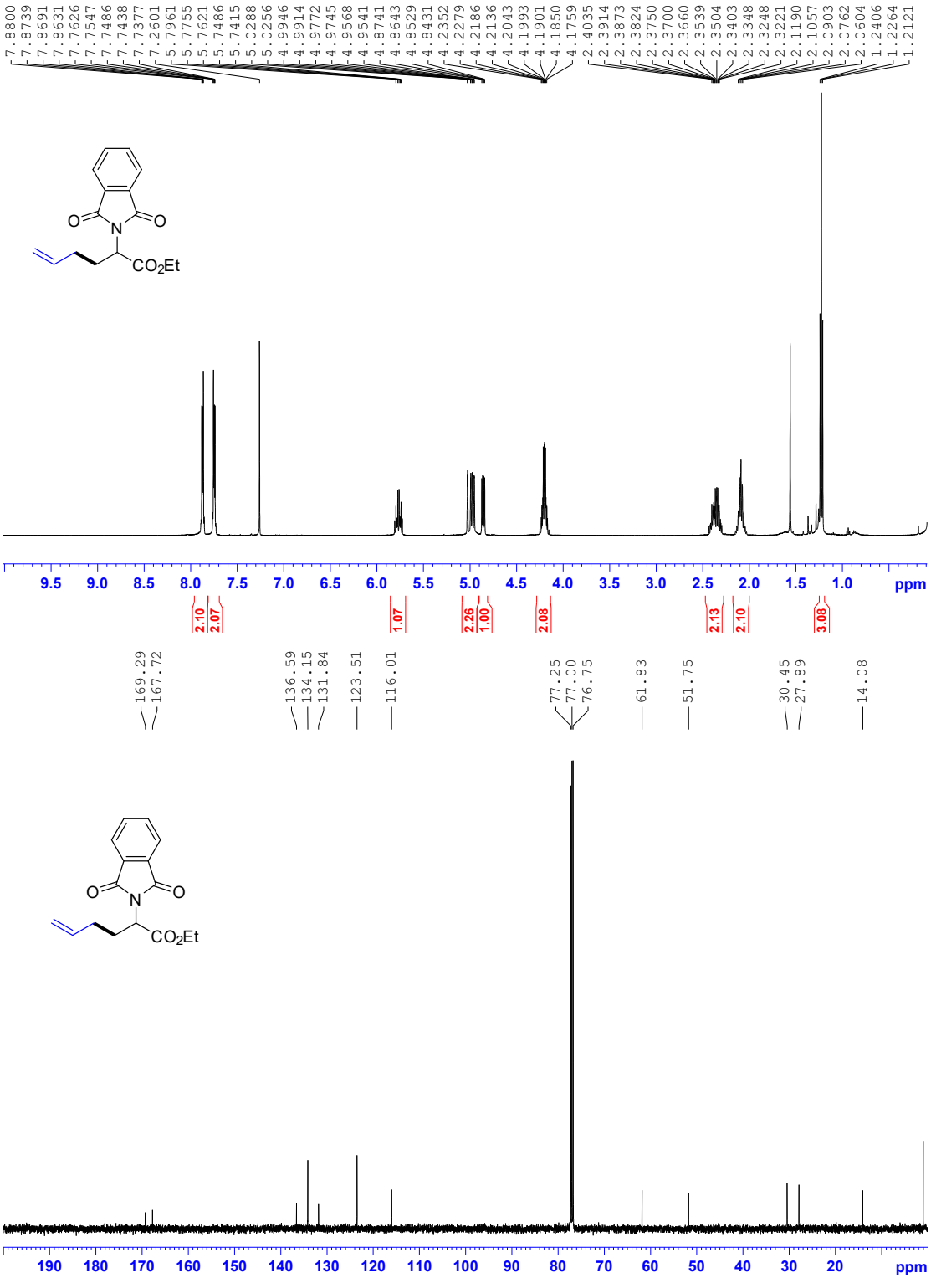
7.8668
7.8607
7.7644
7.7566
7.7505
7.7457
7.7396
7.2601
4.8399
4.8292
4.8194
4.8086
4.2234
4.2163
4.2092
4.1977
4.1951
4.1837
4.1766
4.1696
4.0367
4.0278
4.0149
4.0052
3.9922
2.2988
2.2878
2.2788
2.2670
2.2598
2.2529
2.2482
2.2346
1.9825
1.7014
1.6974
1.6889
1.6828
1.6697
1.6547
1.6426
1.6236
1.4028
1.3977
1.3844
1.3770
1.3702
1.3649
1.3576
1.3504
1.3452
1.3386
1.2336
1.2193
1.2051
AcO
CO2Et
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 ppm
2.02 2.07 1.00 2.08 2.08 2.10 3.01 2.79 2.20 3.02
171.07
169.14
167.69
134.20
131.76
123.53
77.25
77.00
76.75
64.03
61.84
52.08
28.30
27.87
22.88
20.88
14.06
AcO
CO2Et
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm


O
N
O
CO2Et

(5k)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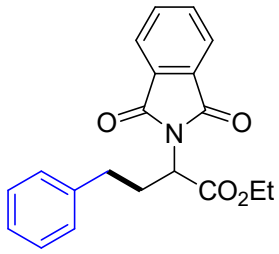
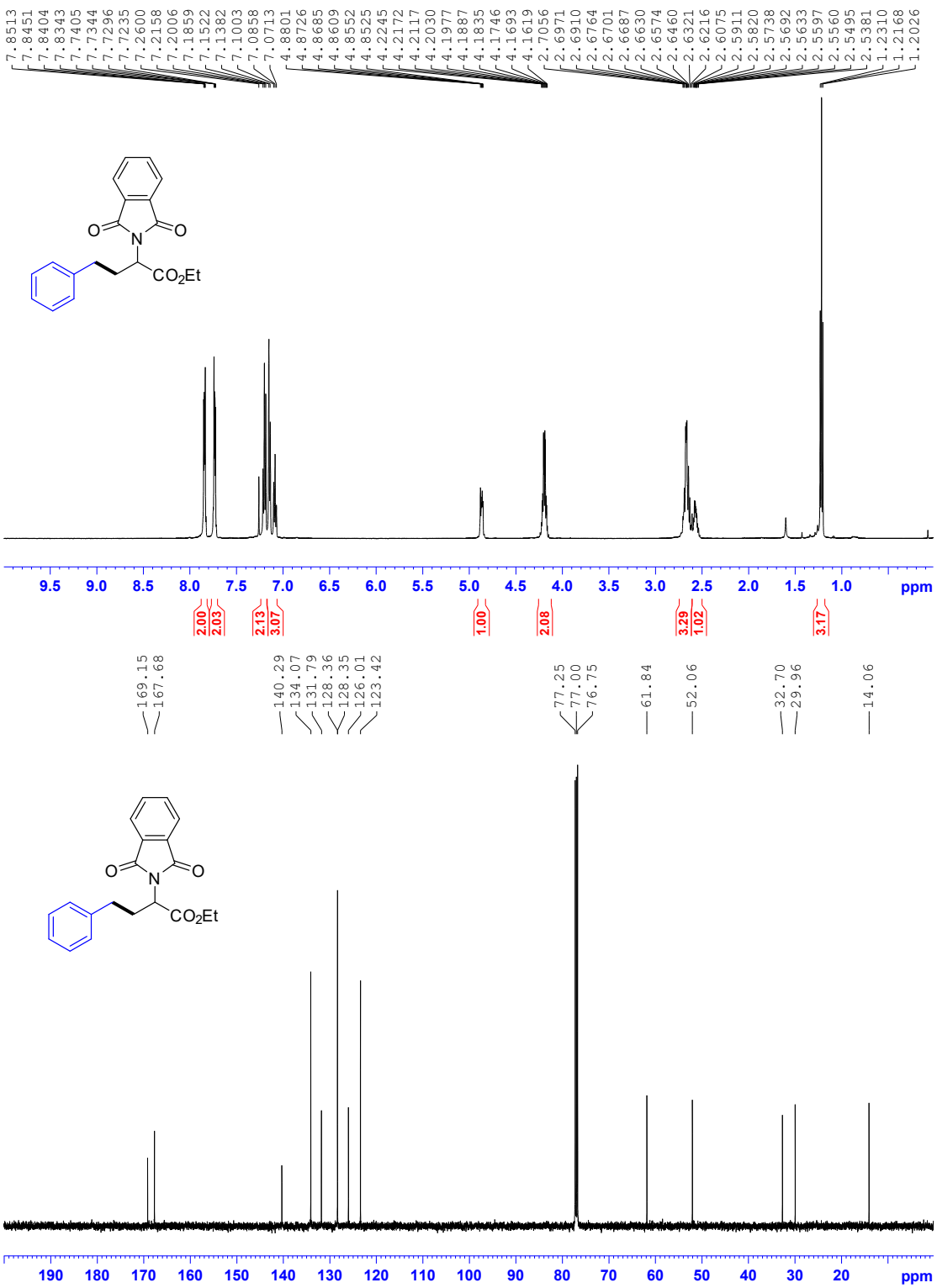
7.8739
7.8691
7.8631
7.7626
7.7547
7.7486
7.7438
7.7377
7.2601
5.7961
5.7755
5.7621
5.7486
5.7415
5.0288
5.0256
4.9946
4.9914
4.9772
4.9745
4.9568
4.9541
4.8741
4.8643
4.8529
4.8431
4.352
4.2279
4.2186
4.2136
4.2043
4.1993
4.1901
4.1850
4.1759
2.4035
2.3914
2.3873
2.3824
2.3750
2.3700
2.3660
2.3539
2.3504
2.3403
2.3348
2.3221
2.1190
2.1057
2.0903
2.0762
2.0604
1.2406
1.2264
1.2121
O
N
O
CO2Et
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 ppm
2.10
2.07
1.07
2.26
1.00
2.08
2.13
2.10
3.08
169.29
167.72
136.59
134.15
131.84
123.51
116.01
77.25
77.00
76.75
61.83
51.75
30.45
27.89
14.08
O
N
O
CO2Et
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm


O
N
O
CO2Et

(5l)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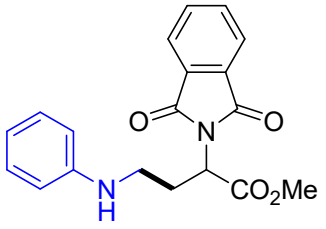
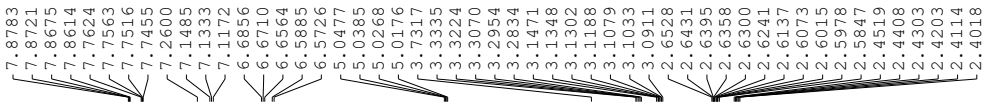
7.8343
7.7405
7.7344
7.7296
7.7235
7.2600
7.2158
7.2006
7.1859
7.1522
7.1382
7.1003
7.0858
7.0713
4.8801
4.8726
4.8685
4.8609
4.8552
4.8525
4.2245
4.2172
4.2117
4.2030
4.1977
4.1887
4.1835
4.1746
4.1693
4.1619
2.7056
2.6971
2.6910
2.6764
2.6701
2.6687
2.6630
2.6574
2.6460
2.6321
2.6216
2.6075
2.5911
2.5820
2.5738
2.5692
2.5633
2.5597
2.5560
2.5495
2.5381
1.2310
1.2168
1.2026
O
N
O
CO2Et
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
ppm
2.00
2.03
2.13
3.07
1.00
2.08
3.29
1.02
3.17
169.15
167.68
140.29
134.07
131.79
128.36
128.35
126.01
123.42
77.25
77.00
76.75
61.84
52.06
32.70
29.96
14.06
O
N
O
CO2Et
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm


O
N
O
N
H
CO2Me

(5n)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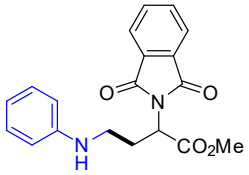
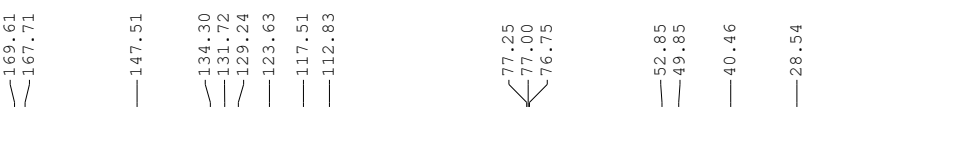
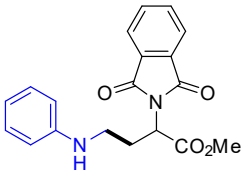
7.8614
7.7624
7.7563
7.7516
7.7455
7.2600
7.1485
7.1333
7.1172
6.6856
6.6710
6.6564
6.5885
6.5726
5.0477
5.0385
5.0268
5.0176
3.7317
3.3335
3.3224
3.3070
3.2954
3.2834
3.1471
3.1348
3.1302
3.1188
3.1079
3.1033
3.0911
2.6528
2.6431
2.6395
2.6358
2.6300
2.6241
2.6137
2.6073
2.6015
2.5978
2.5847
2.4519
2.4408
2.4303
2.4203
2.4114
2.4018

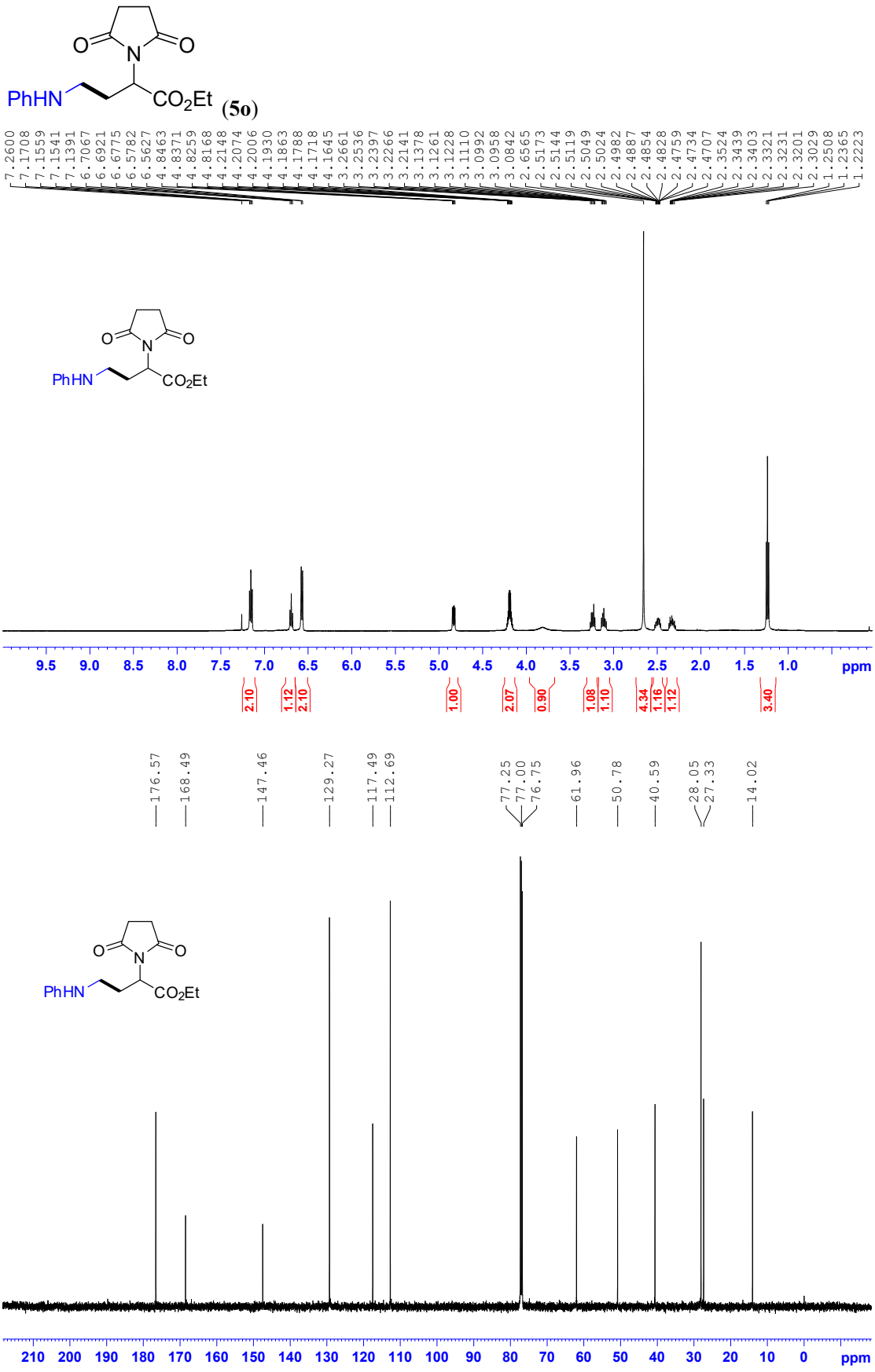

O
N
O
N
H
CO2Me

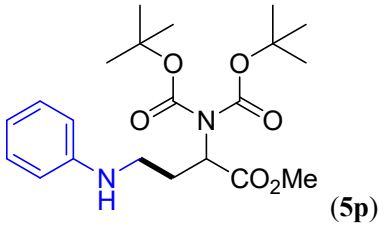
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
2.03
2.04
2.34
1.31
2.31
1.00
3.22
1.17
1.15
1.09
1.13


169.61
167.71
147.51
134.30
131.72
129.24
123.63
117.51
112.83
77.25
77.00
76.75
52.85
49.85
40.46
28.54

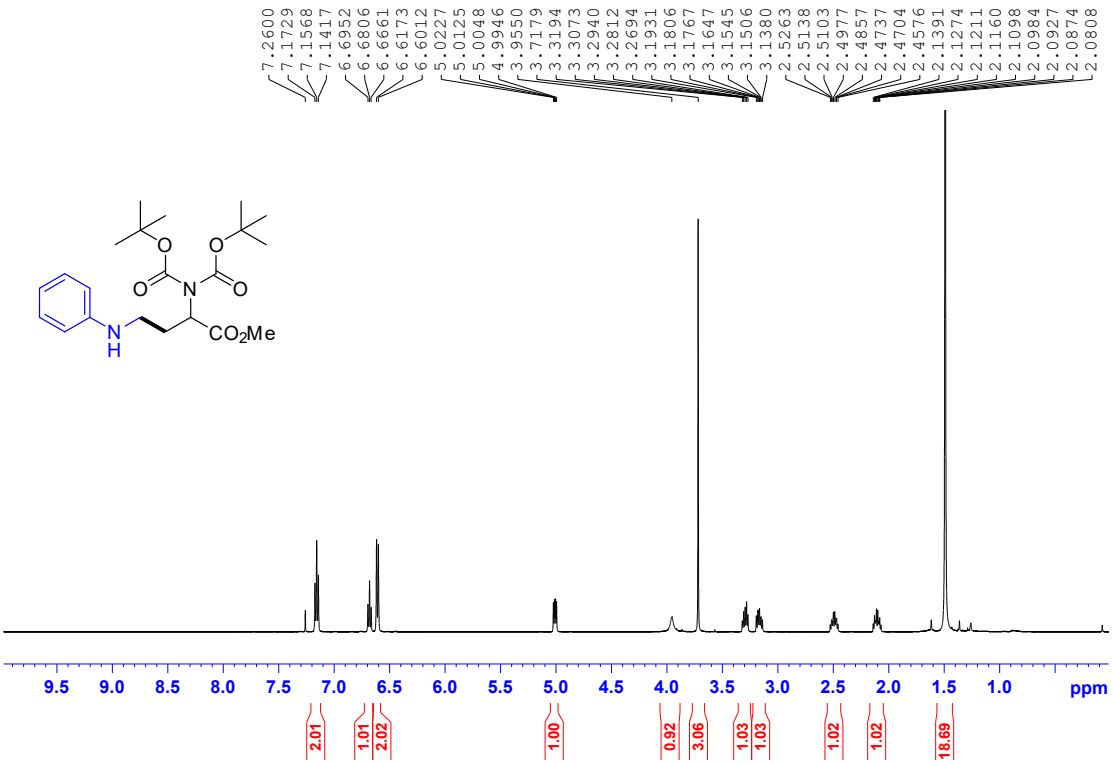

O
N
O
N
H
CO2Me

190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm


PhHN
CO2Et
(5o)
7.2600
7.1708
7.1559
7.1541
7.1391
6.7067
6.6921
6.6775
6.5782
6.5627
4.8463
4.8371
4.8259
4.8168
4.2148
4.2074
4.2006
4.1930
4.1863
4.1788
4.1718
4.1645
3.2661
3.2536
3.2397
3.2266
3.2141
3.1378
3.1261
3.1228
3.1110
3.0992
3.0958
3.0842
2.6565
2.5173
2.5144
2.5119
2.5049
2.5024
2.4982
2.4887
2.4854
2.4828
2.4759
2.4734
2.4707
2.3524
2.3439
2.3403
2.3321
2.3231
2.3201
2.3029
2.2508
1.2365
1.2223
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 ppm
2.10
1.12
2.10
1.00
2.07
0.90
1.08
1.10
4.34
1.16
1.12
3.40
176.57
168.49
147.46
129.27
117.49
112.69
77.25
77.00
76.75
61.96
50.78
40.59
28.05
27.33
14.02
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm



(5p)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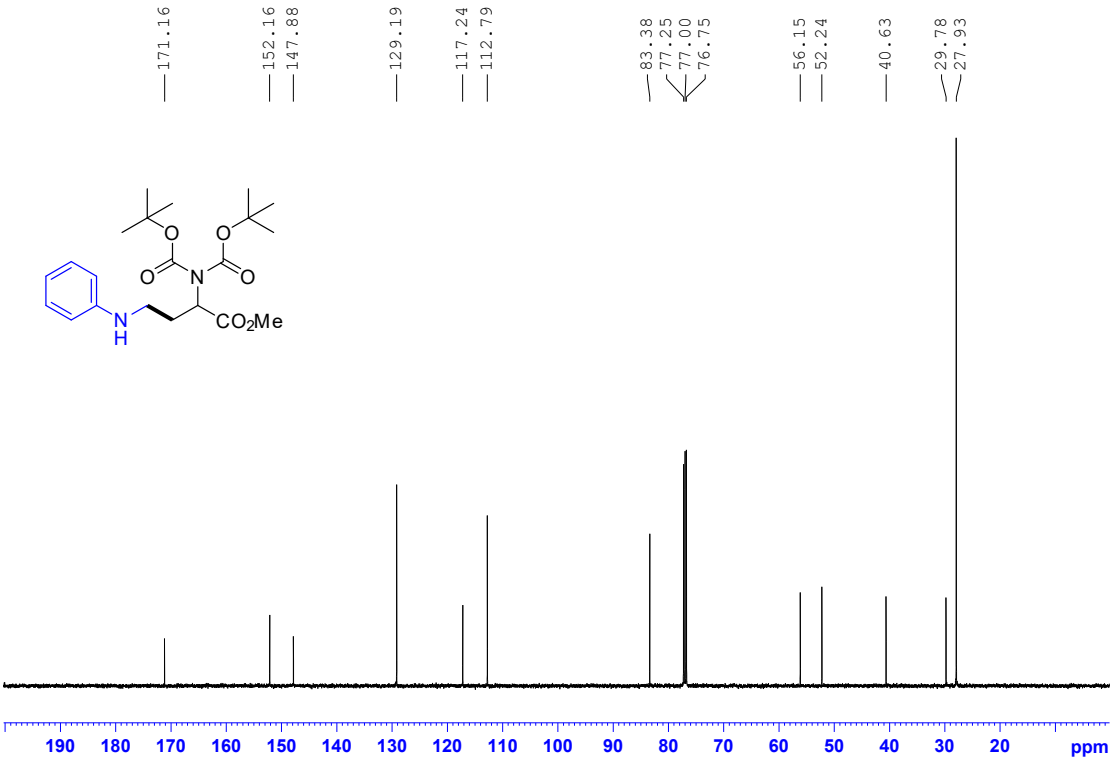
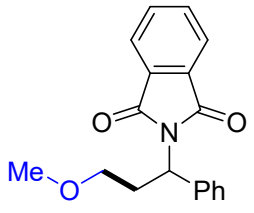
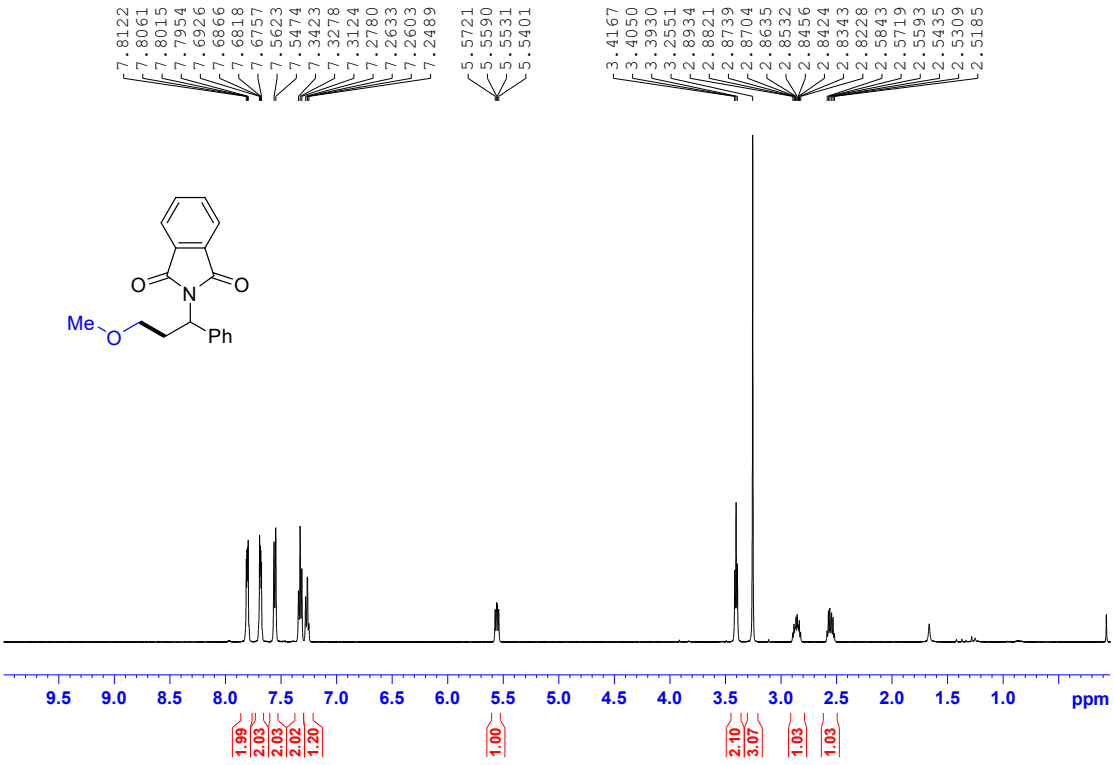
7.1729
7.1568
7.1417
6.6952
6.6806
6.6661
6.6173
6.6012
5.0227
5.0125
5.0048
4.9946
3.9550
3.7179
3.3194
3.3073
3.2940
3.2812
3.2694
3.1931
3.1806
3.1767
3.1647
3.1545
3.1506
3.1380
2.5263
2.5138
2.5103
2.4977
2.4857
2.4737
2.4704
2.4576
2.1391
2.1274
2.1211
2.1160
2.1098
2.0984
2.0927
2.0874
2.0808
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 ppm
2.01
1.01
2.02
1.00
0.92
3.06
1.03
1.03
1.02
1.02
18.69


171.16
152.16
147.88
129.19
117.24
112.79
83.38
77.25
77.00
76.75
56.15
52.24
40.63
29.78
27.93
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm


O
N
O
Me
O
Ph

(7b)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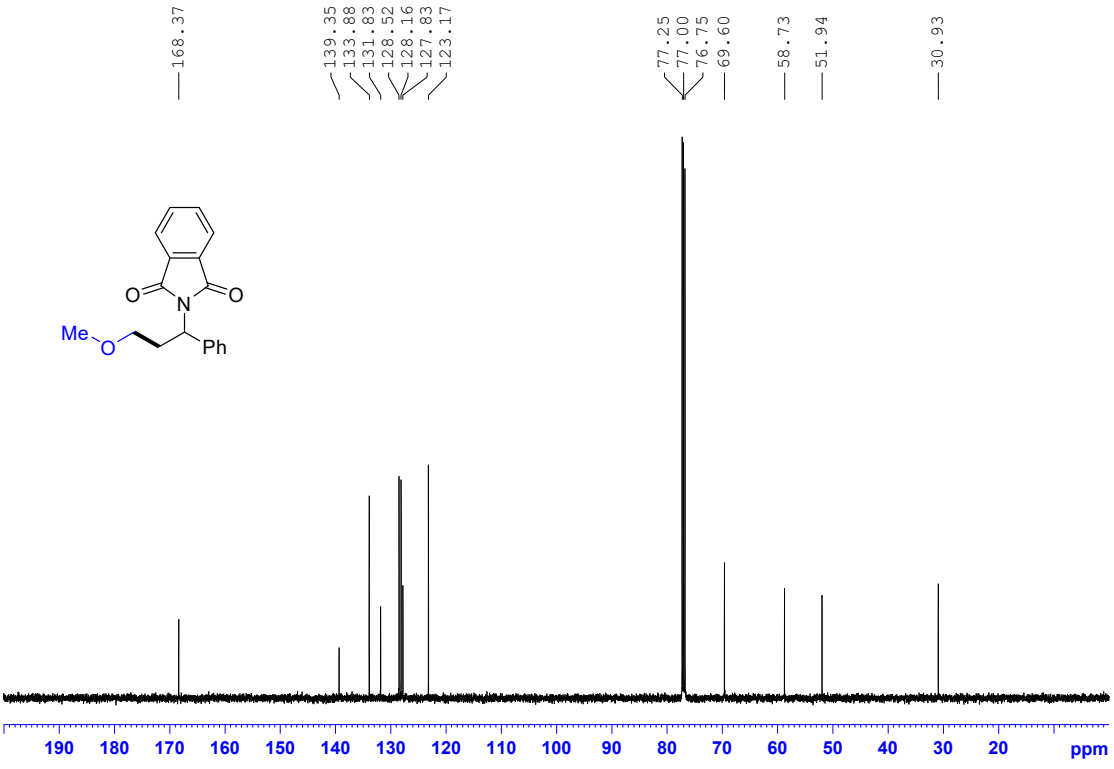
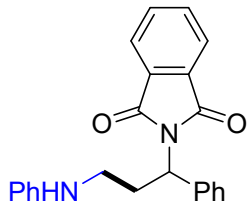
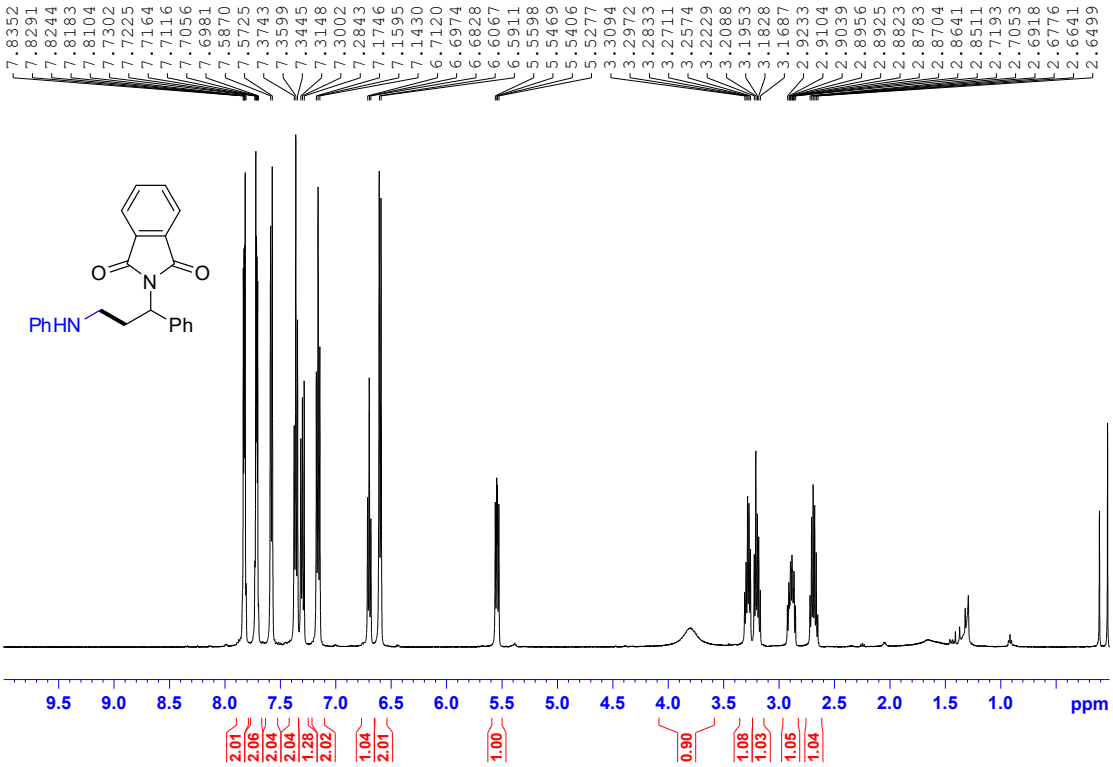
7.8015
7.7954
7.6926
7.6866
7.6818
7.6757
7.5623
7.5474
7.3423
7.3278
7.3124
7.2780
7.2633
7.2603
7.2489
5.5721
5.5590
5.5531
5.5401
3.4167
3.4050
3.3930
3.2551
2.8934
2.8821
2.8739
2.8704
2.8635
2.8532
2.8456
2.8424
2.8343
2.8228
2.5843
2.5719
2.5593
2.5435
2.5309
2.5185
O
N
O
Me
O
Ph
9.5
9.0
8.5
8.0
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
ppm
1.99
2.03
2.03
2.02
1.20
1.00
2.10
3.07
1.03
1.03


168.37
139.35
133.88
131.83
128.52
128.16
127.83
123.17
77.25
77.00
76.75
69.60
58.73
51.94
30.93
O
N
O
Me
O
Ph
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm


O
N
O
PhHN
Ph

(7c)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7.8104
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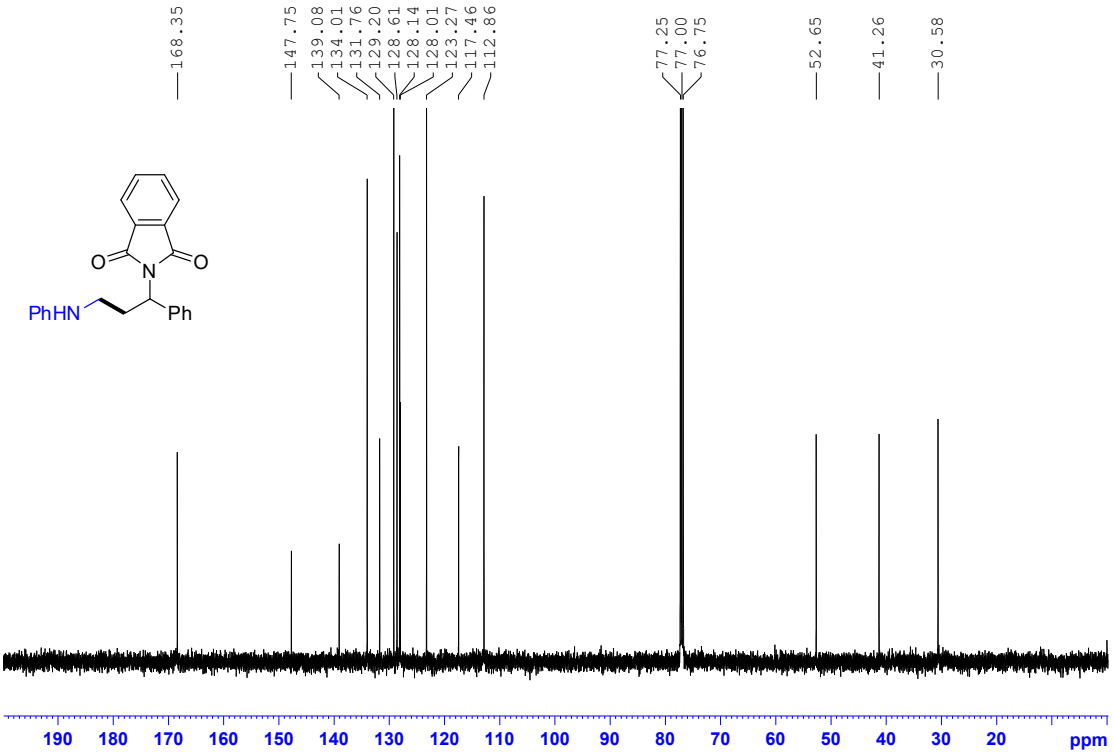
7.7225
7.7164
7.7116
7.7056
7.6981
7.5870
7.5725
7.3743
7.3599
7.3445
7.3148
7.3002
7.2843
7.1746
7.1595
7.1430
7.7120
6.6974
6.6828
6.6067
6.5911
5.5598
5.5469
5.5406
5.5277
3.3094
3.2972
3.2833
3.2711
3.2574
3.2229
3.2088
3.1953
3.1828
3.1687
2.9233
2.9104
2.9039
2.8956
2.8925
2.8823
2.8783
2.8704
2.8641
2.8511
2.7193
2.7053
2.6918
2.6776
2.6641
2.6499
O
N
O
PhHN
Ph
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
ppm
2.01
2.06
2.04
2.04
1.28
2.02
1.04
2.01
1.00
0.90
1.08
1.03
1.05
1.04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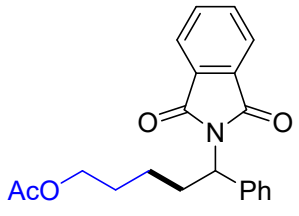
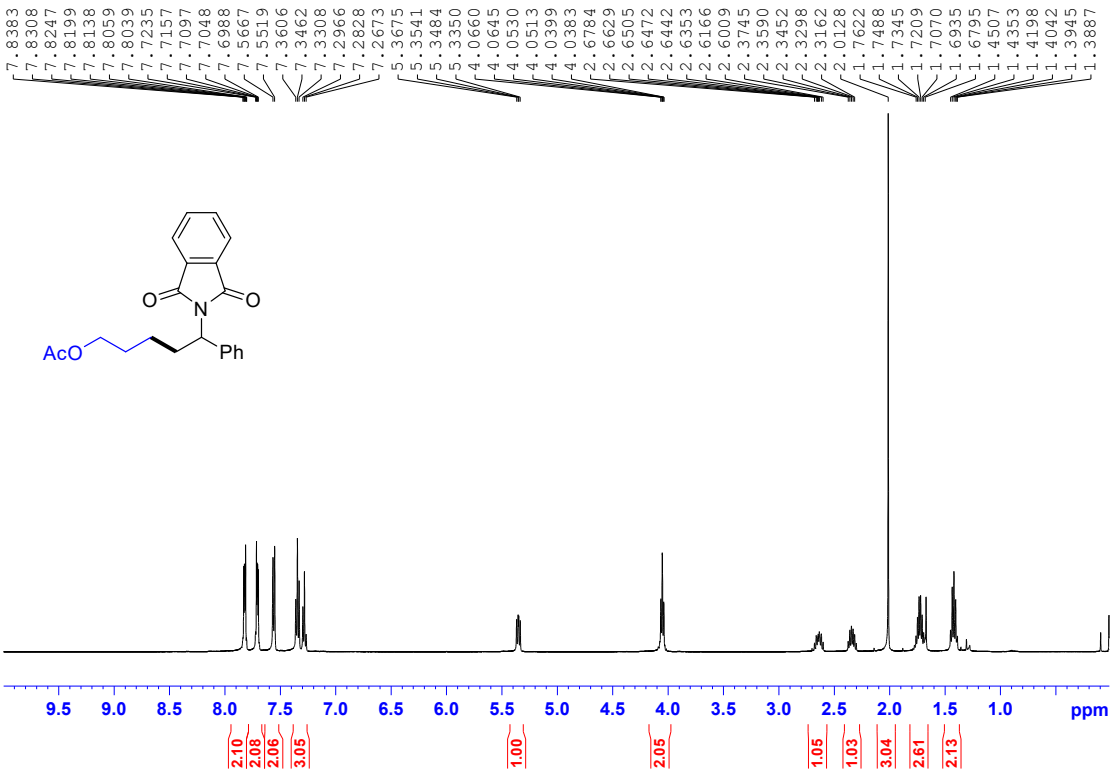
147.75
139.08
134.01
131.76
129.20
128.61
128.14
128.01
123.27
117.46
112.86
77.25
77.00
76.75
52.65
41.26
30.58
O
N
O
PhHN
Ph
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm


O
N
O
AcO
Ph

(7d)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7.8247
7.8199
7.8138
7.8059
7.8039
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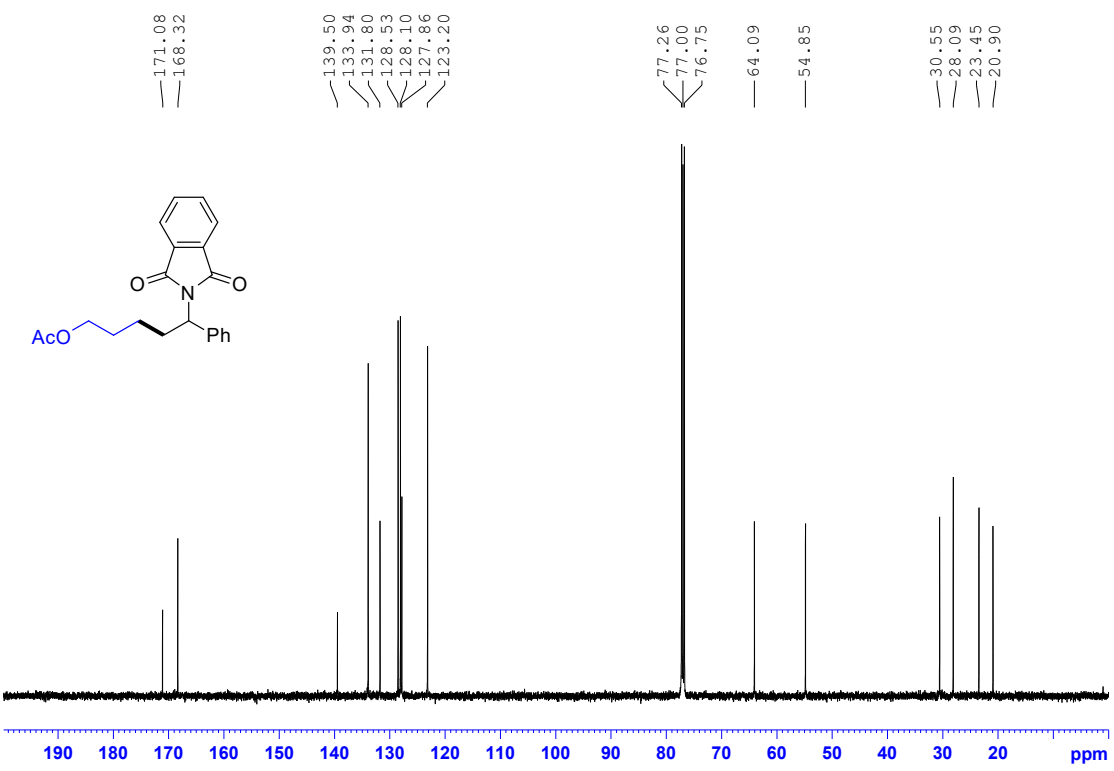
7.7157
7.7097
7.7048
7.6988
7.5667
7.5519
7.3606
7.3462
7.3308
7.2966
7.2828
7.2673
5.3675
5.3541
5.3484
5.3350
4.0660
4.0645
4.0530
4.0513
4.0399
4.0383
2.6784
2.6629
2.6505
2.6472
2.6442
2.6353
2.6166
2.6009
2.3745
2.3590
2.3452
2.3298
2.3162
2.0128
1.7622
1.7488
1.7345
1.7209
1.7070
1.6935
1.6795
1.4507
1.4353
1.4198
1.4042
1.3945
1.3887
O
N
O
AcO
Ph
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
ppm
2.10
2.08
2.06
3.05
1.00
2.05
1.05
1.03
3.04
2.61
2.13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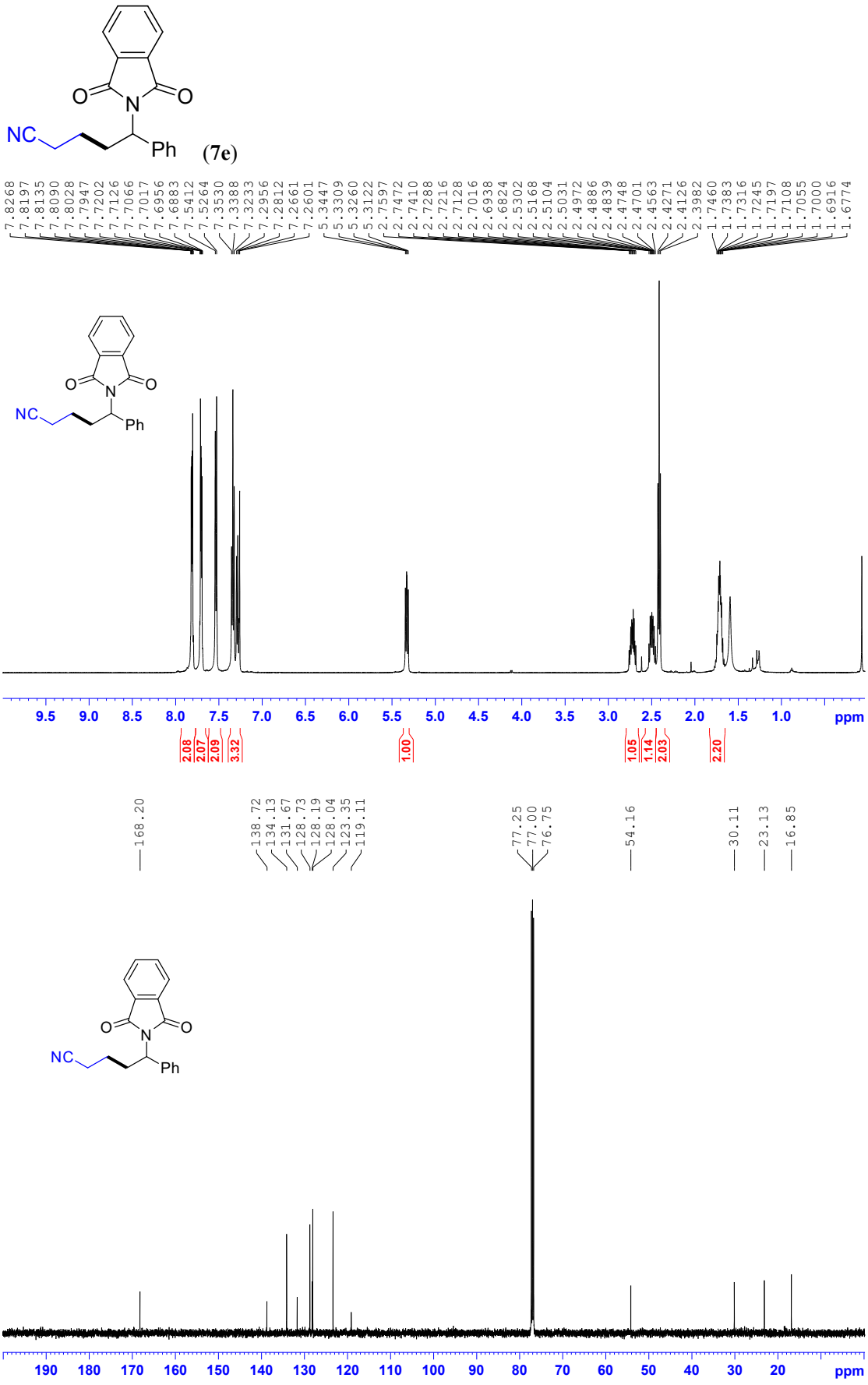
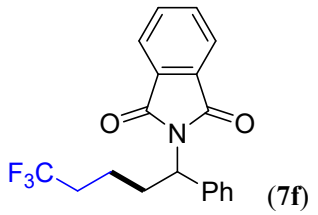
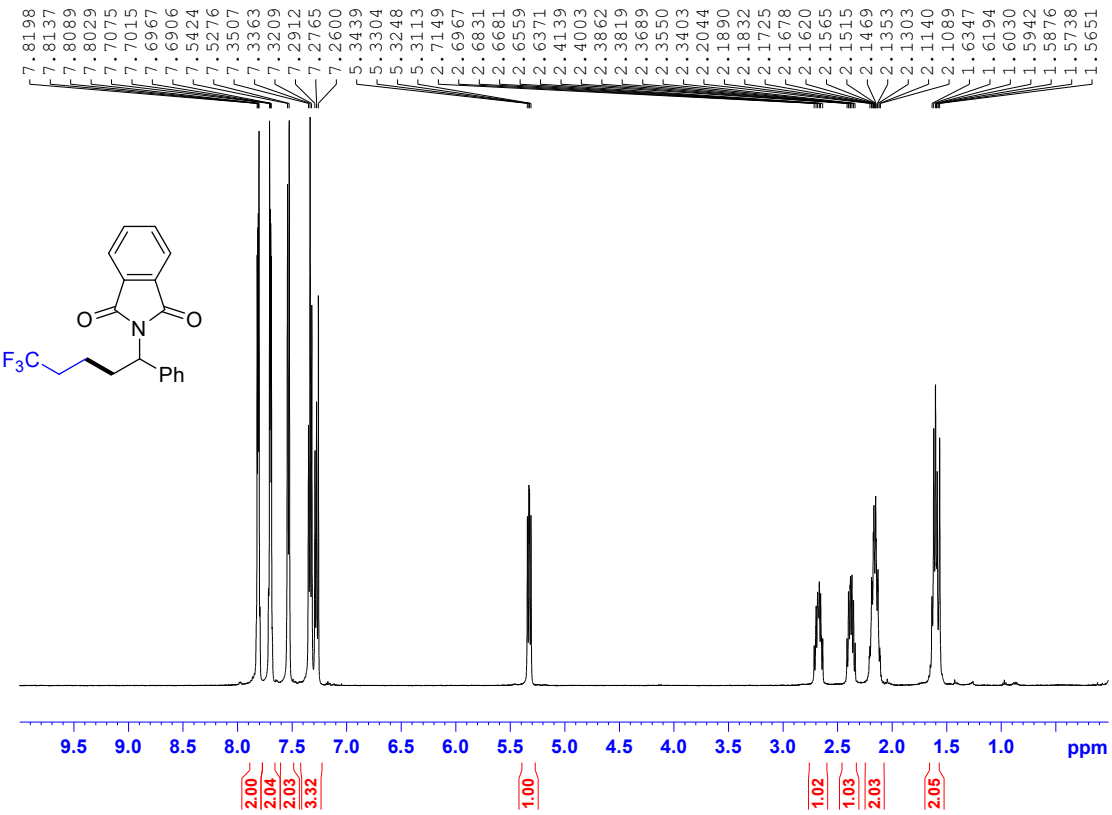
168.32
139.50
133.94
131.80
128.53
128.10
127.86
123.20
77.26
77.00
76.75
64.09
54.85
30.55
28.09
23.45
20.90
O
N
O
AcO
Ph
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm


NC
Ph
(7e)
7.8268
7.8197
7.8135
7.8090
7.8028
7.7947
7.7202
7.7126
7.7066
7.7017
7.6956
7.6883
7.5412
7.5264
7.3530
7.3388
7.3233
7.2956
7.2812
7.2661
7.2601
5.3447
5.3309
5.3260
5.3122
2.7597
2.7472
2.7410
2.7288
2.7216
2.7128
2.7016
2.6938
2.6824
2.5302
2.5168
2.5104
2.5031
2.4972
2.4886
2.4839
2.4748
2.4701
2.4563
2.4271
2.4126
2.3982
1.7460
1.7383
1.7316
1.7245
1.7197
1.7108
1.7055
1.7000
1.6916
1.6774
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 ppm
2.08
2.07
2.09
3.32
1.00
1.05
1.14
2.03
2.20
168.20
138.72
134.13
131.67
128.73
128.19
128.04
123.35
119.11
77.25
77.00
76.75
54.16
30.11
23.13
16.85
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm


O
N
O
F3C
Ph

(7f)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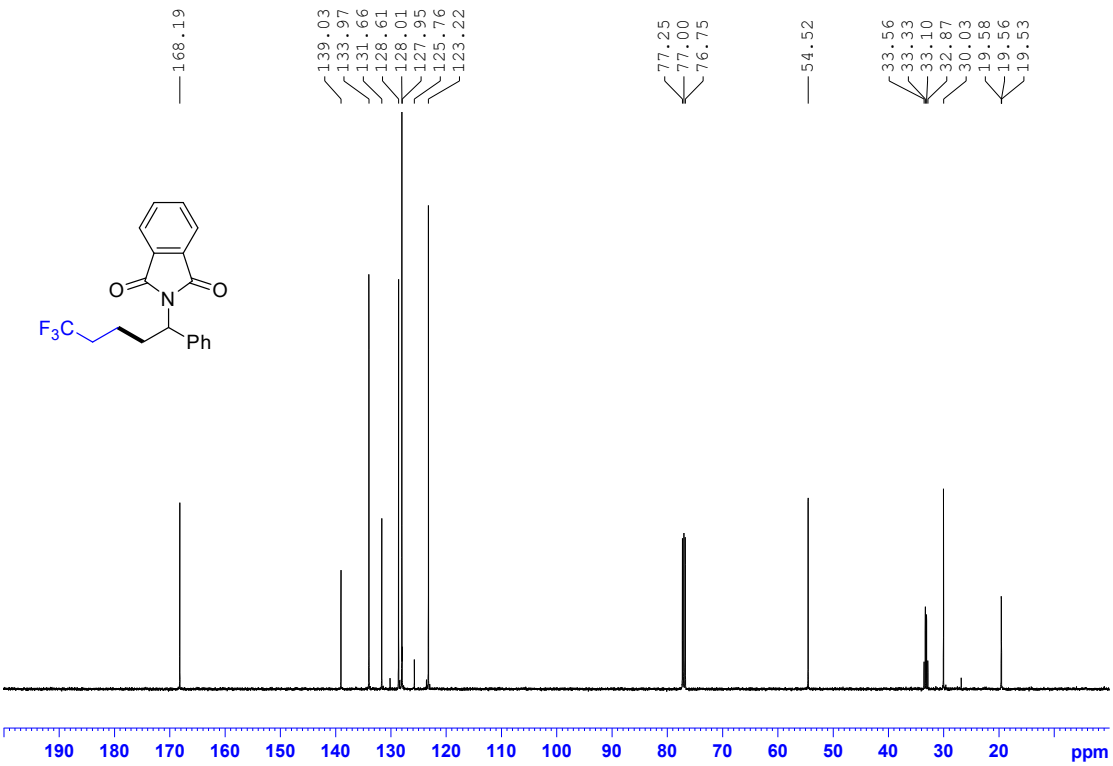
7.8029
7.7075
7.7015
7.6967
7.6906
7.5424
7.5276
7.3507
7.3363
7.3209
7.2912
7.2765
7.2600
5.3439
5.3304
5.3248
5.3113
2.7149
2.6967
2.6831
2.6681
2.6559
2.6371
2.4139
2.4003
2.3862
2.3819
2.3689
2.3550
2.3403
2.2044
2.1890
2.1832
2.1725
2.1678
2.1620
2.1565
2.1515
2.1469
2.1353
2.1303
2.1140
2.1089
1.6347
1.6194
1.6030
1.5942
1.5876
1.5738
1.5651
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 ppm
2.00
2.04
2.03
3.32
1.00
1.02
1.03
2.03
2.05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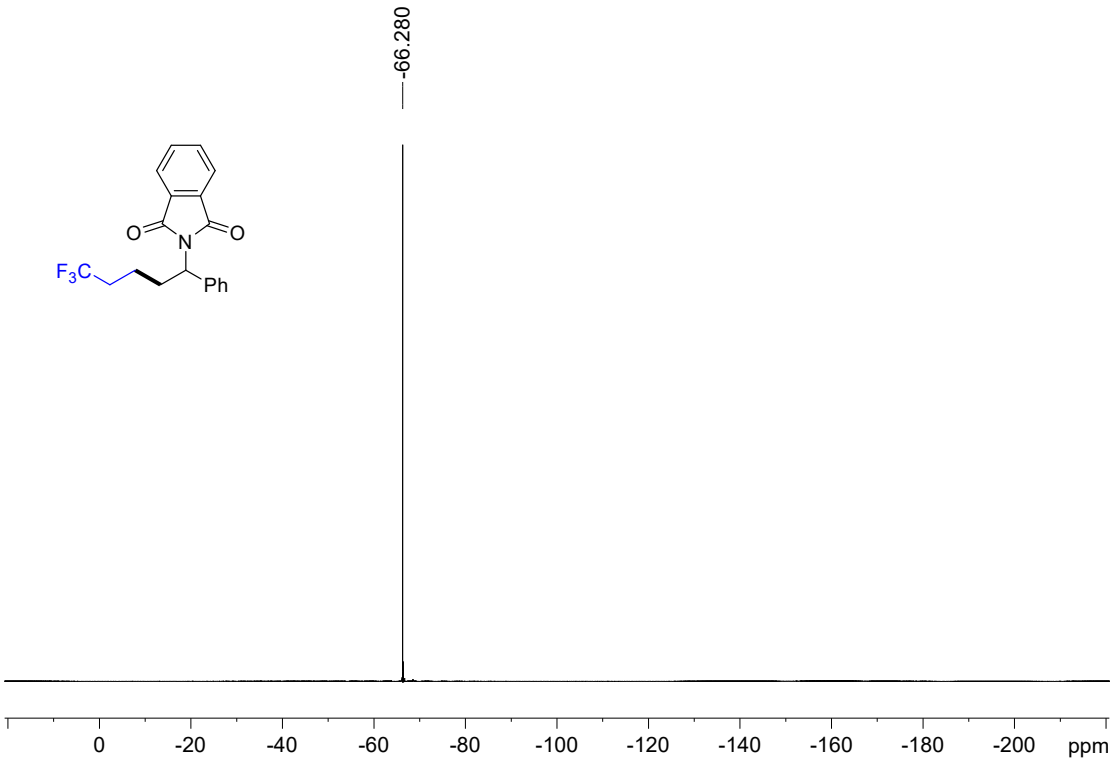
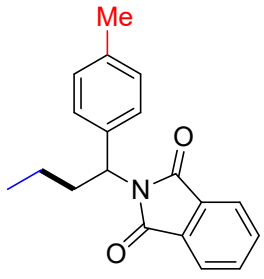
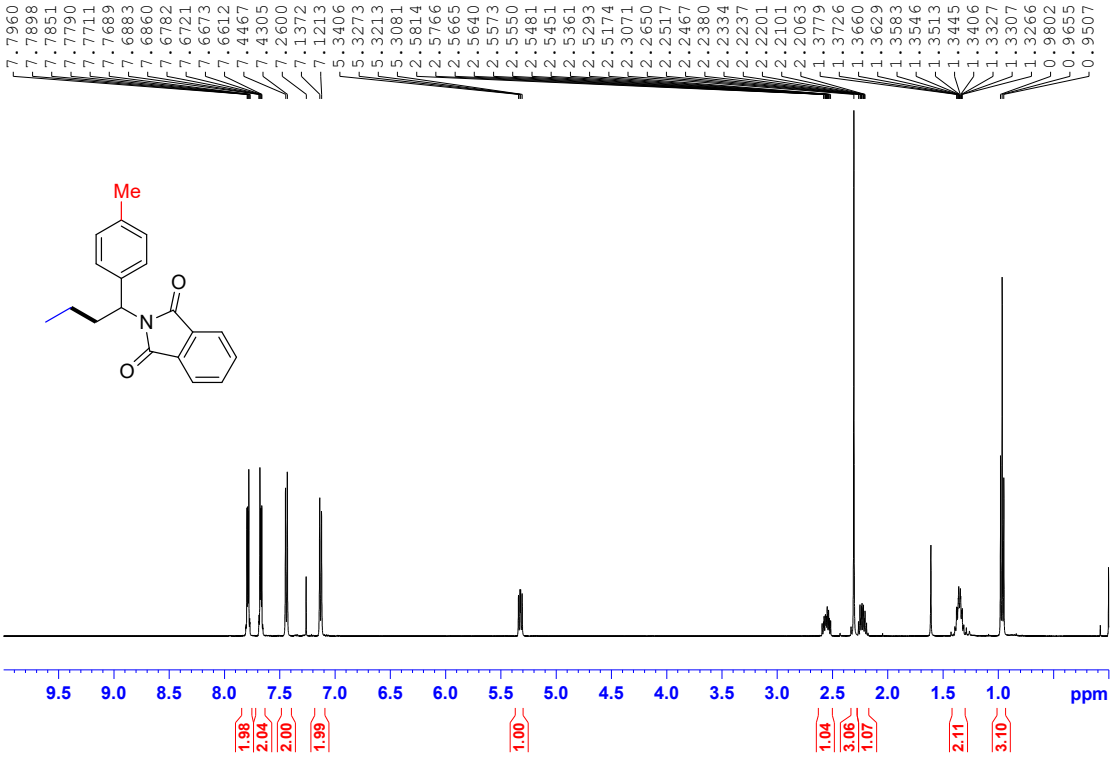
139.03
133.97
131.66
128.61
128.01
127.95
125.76
123.22
77.25
77.00
76.75
54.52
33.56
33.33
33.10
32.87
30.03
19.58
19.56
19.53
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm


-66.280
O
N
O
F3C
Ph
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
-200
ppm


Me
O
N
O

(7i)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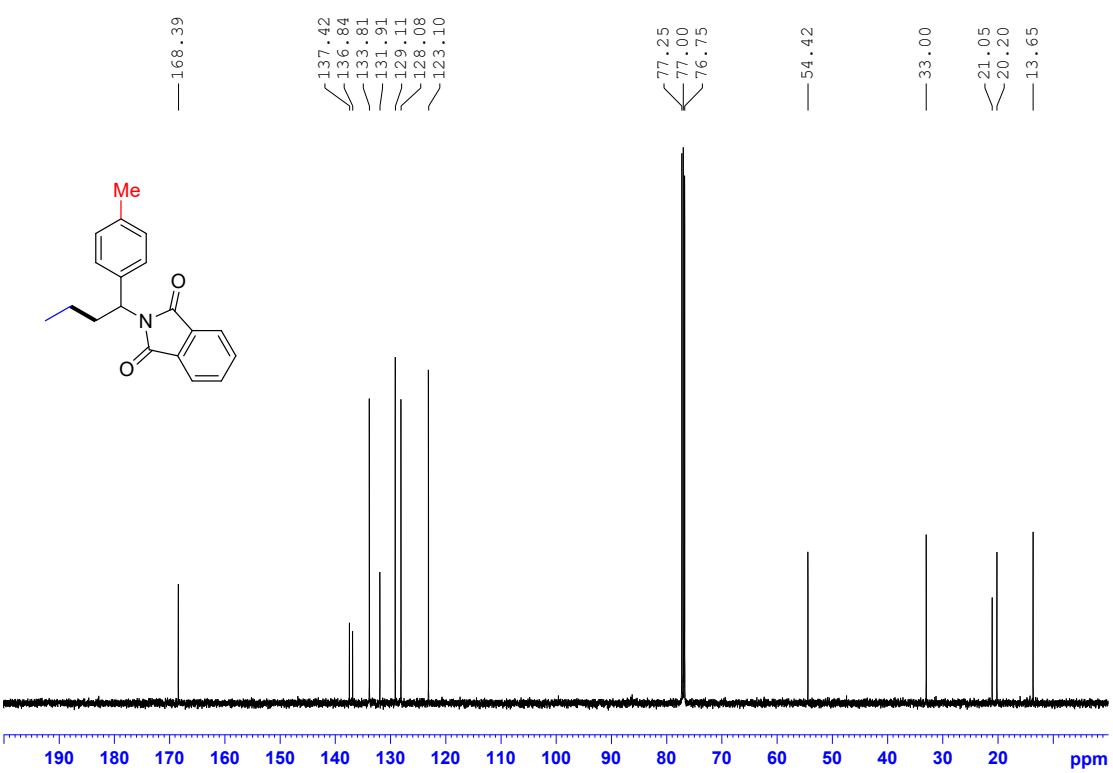
7.7790
7.7711
7.7689
7.6883
7.6860
7.6782
7.6721
7.6673
7.6612
7.4467
7.4305
7.2600
7.1372
7.1213
5.3406
5.3273
5.3213
5.3081
2.5814
2.5766
2.5665
2.5640
2.5573
2.5550
2.5481
2.5451
2.5361
2.5293
2.5174
2.3071
2.2650
2.2517
2.2467
2.2380
2.2334
2.2237
2.2201
2.2101
2.2063
1.3779
1.3726
1.3660
1.3629
1.3583
1.3546
1.3513
1.3445
1.3406
1.3327
1.3307
1.3266
0.9802
0.9655
0.9507
Me
O
N
O
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
ppm
1.98
2.04
2.00
1.99
1.00
1.04
3.06
1.07
2.11
3.10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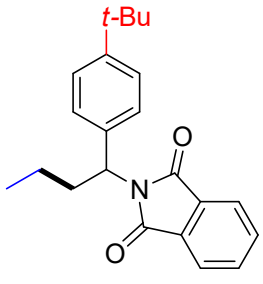
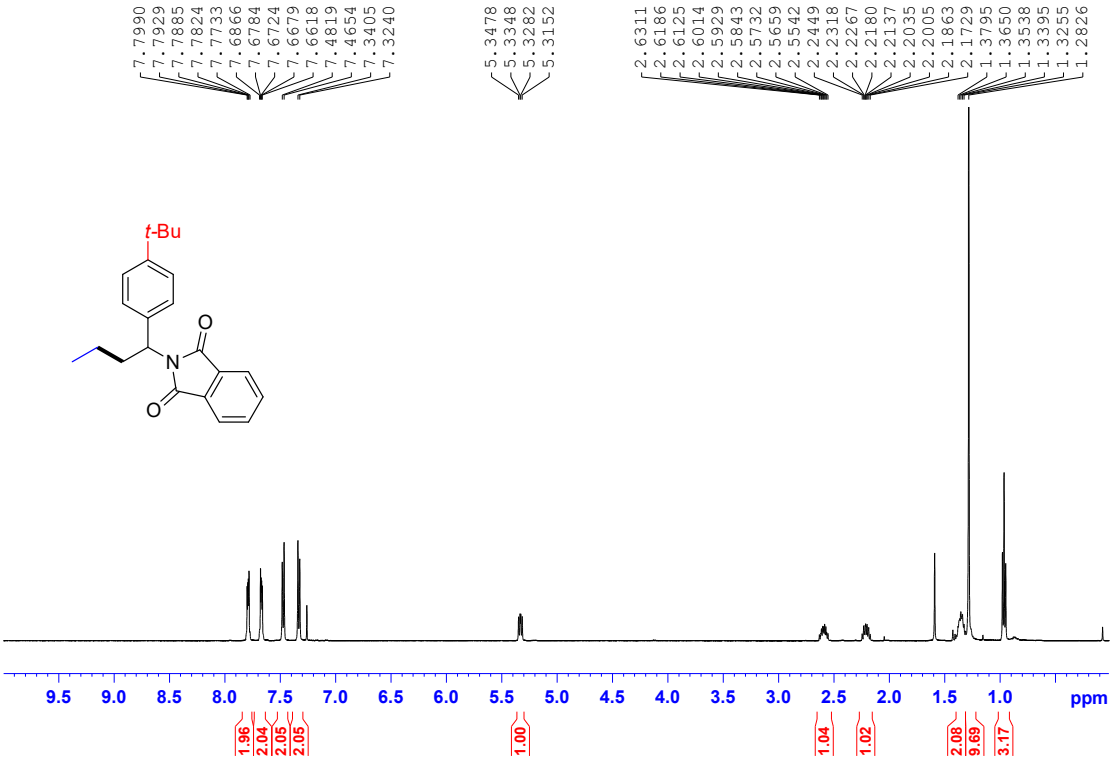
137.42
136.84
133.81
131.91
129.11
128.08
123.10
77.25
77.00
76.75
54.42
33.00
21.05
20.20
13.65
Me
O
N
O
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm


t-Bu
O
N
O

(7j)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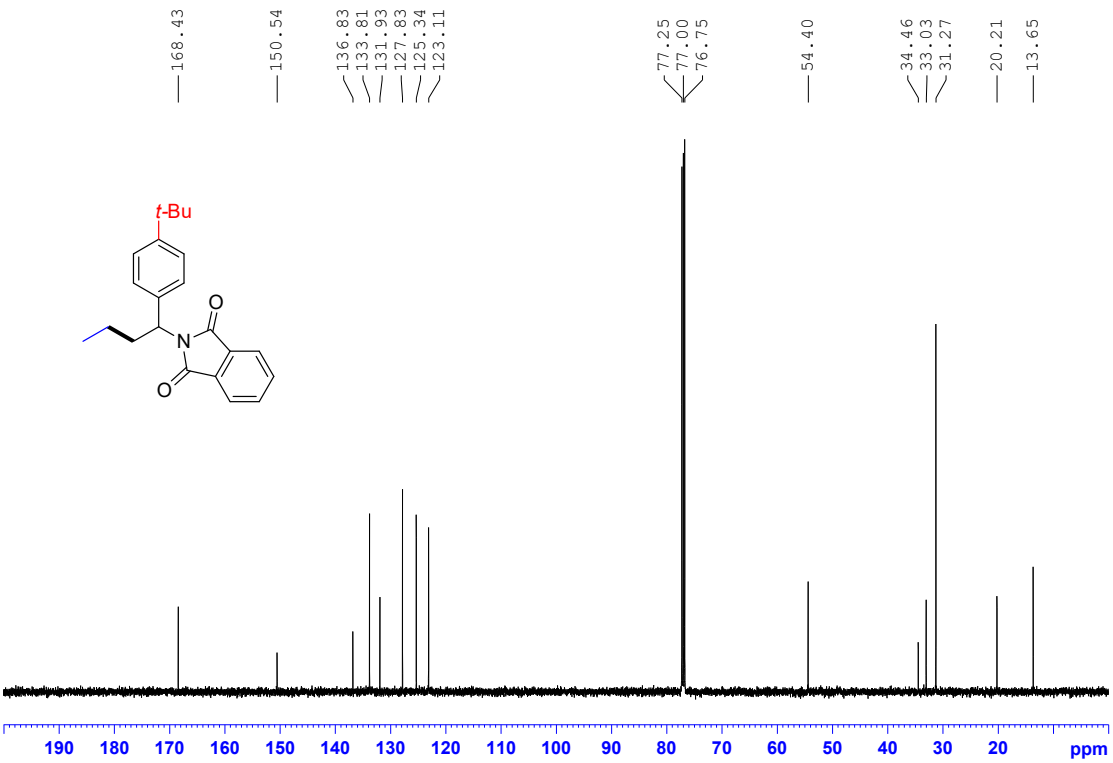
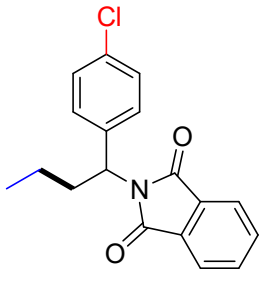
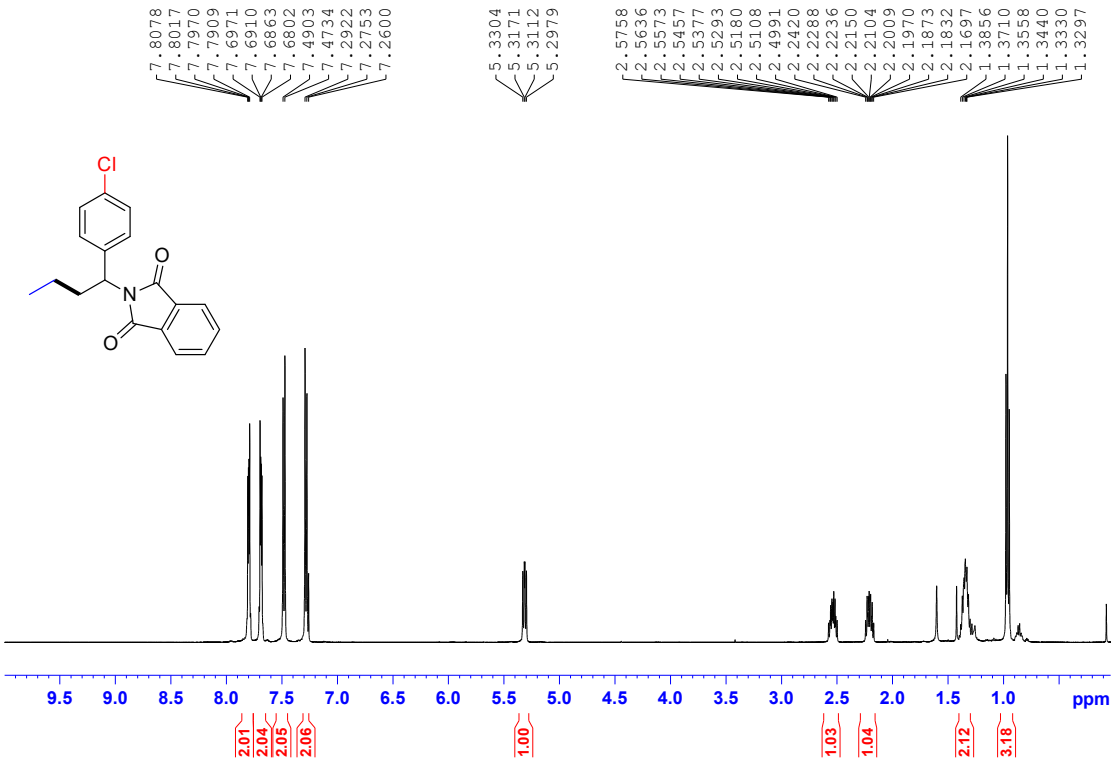
7.7824
7.7733
7.6866
7.6784
7.6724
7.6679
7.6618
7.4819
7.4654
7.3405
7.3240
5.3478
5.3348
5.3282
5.3152
2.6311
2.6186
2.6125
2.6014
2.5929
2.5843
2.5732
2.5659
2.5542
2.2449
2.2318
2.2267
2.2180
2.2137
2.2035
2.2005
2.1863
2.1729
1.3795
1.3650
1.3538
1.3395
1.3255
1.2826
t-Bu
O
N
O
9.5 9.0 8.5 8.0 7.5 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
ppm
1.96
2.04
2.05
2.05
1.00
1.04
1.02
2.08
9.69
3.17


168.43
150.54
136.83
133.81
131.93
127.83
125.34
123.11
77.25
77.00
76.75
54.40
34.46
33.03
31.27
20.21
13.65
t-Bu
O
N
O
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20
ppm


Cl
O
N
O

(7k)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7.7970
7.7909
7.6971
7.6910
7.6863
7.6802
7.4903
7.4734
7.2922
7.2753
7.2600
5.3304
5.3171
5.3112
5.2979
2.5758
2.5636
2.5573
2.5457
2.5377
2.5293
2.5180
2.5108
2.4991
2.2420
2.2288
2.2236
2.2150
2.2104
2.2009
2.1970
2.1873
2.1832
2.1697
1.3856
1.3710
1.3558
1.3440
1.3330
1.3297
Cl
O
N
O
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
ppm
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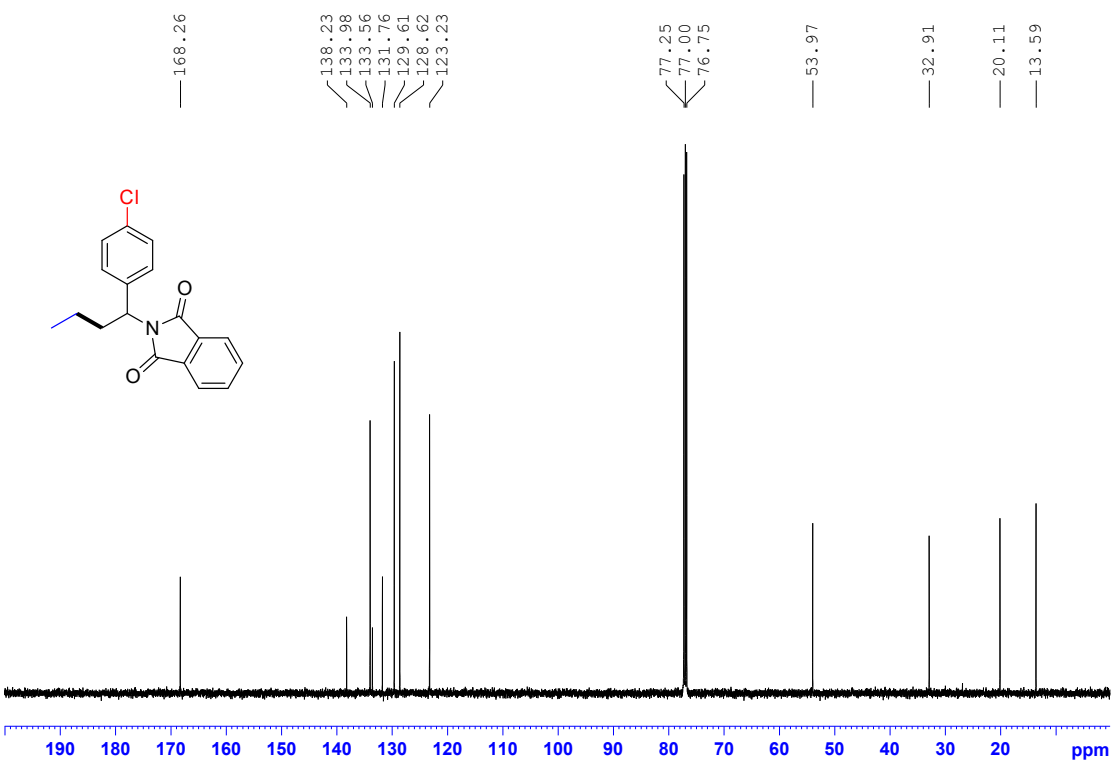
2.04
2.05
2.06
1.00
1.03
1.04
2.12
3.18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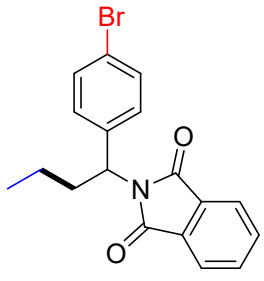
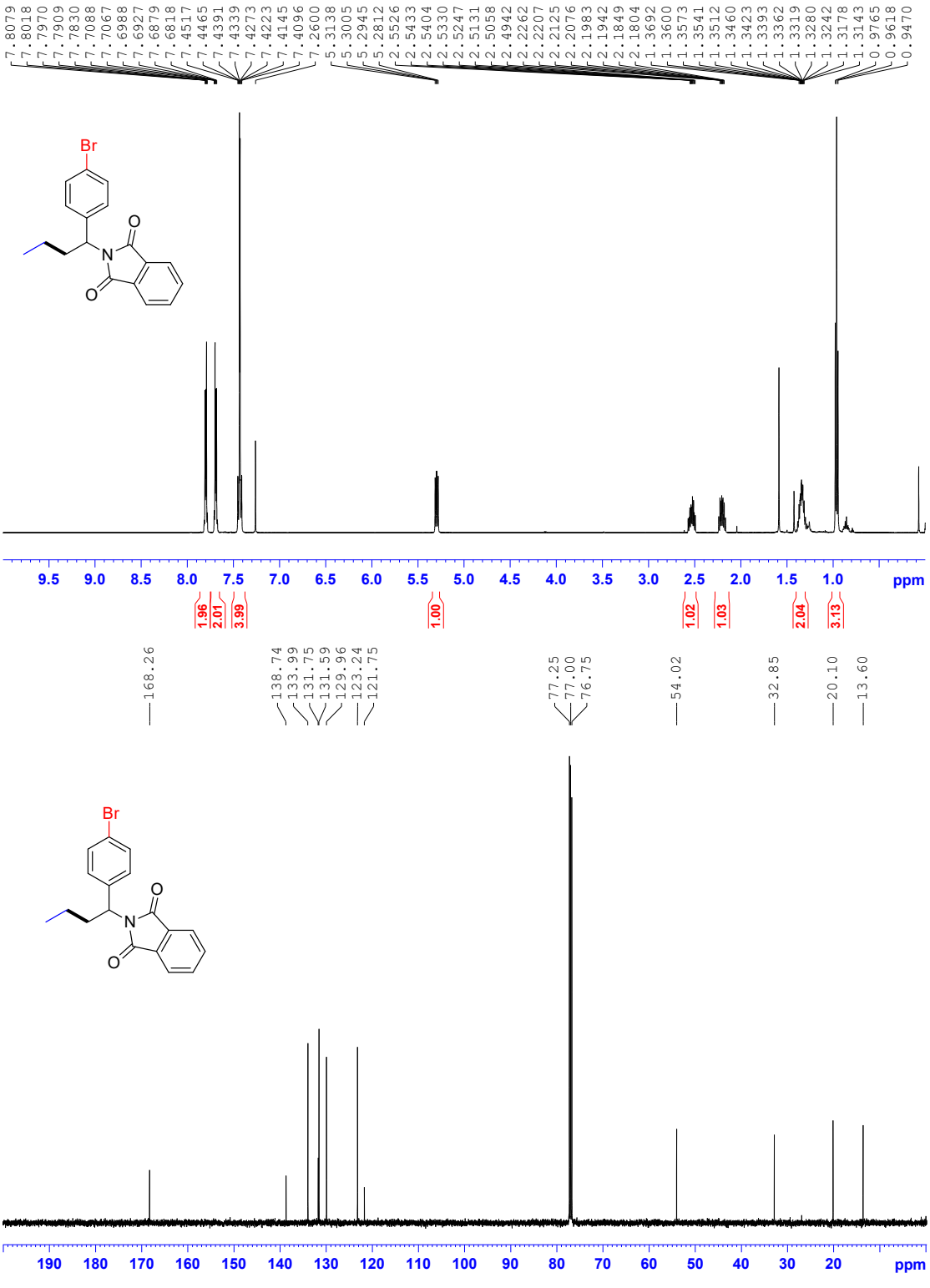
138.23
133.98
133.56
131.76
129.61
128.62
123.23
77.25
77.00
76.75
53.97
32.91
20.11
13.59
Cl
O
N
O
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20
ppm


Br
O
N
O

(7l)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7.7909
7.7830
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7.7067
7.6988
7.6927
7.6879
7.6818
7.4517
7.4465
7.4391
7.4339
7.4273
7.4223
7.4145
7.4096
7.2600
5.3138
5.3005
5.2945
5.2812
2.5526
2.5433
2.5404
2.5330
2.5247
2.5131
2.5058
2.4942
2.2262
2.2207
2.2125
2.2076
2.1983
2.1942
2.1849
2.1804
1.3692
1.3600
1.3573
1.3541
1.3512
1.3460
1.3423
1.3393
1.3362
1.3319
1.3280
1.3242
1.3178
1.3143
0.9765
0.9618
0.9470
Br
O
N
O
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 ppm
1.96
2.01
3.99
1.00
1.02
1.03
2.04
3.13
168.26
138.74
133.99
131.75
131.59
129.96
123.24
121.75
77.25
77.00
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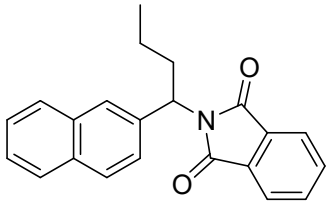
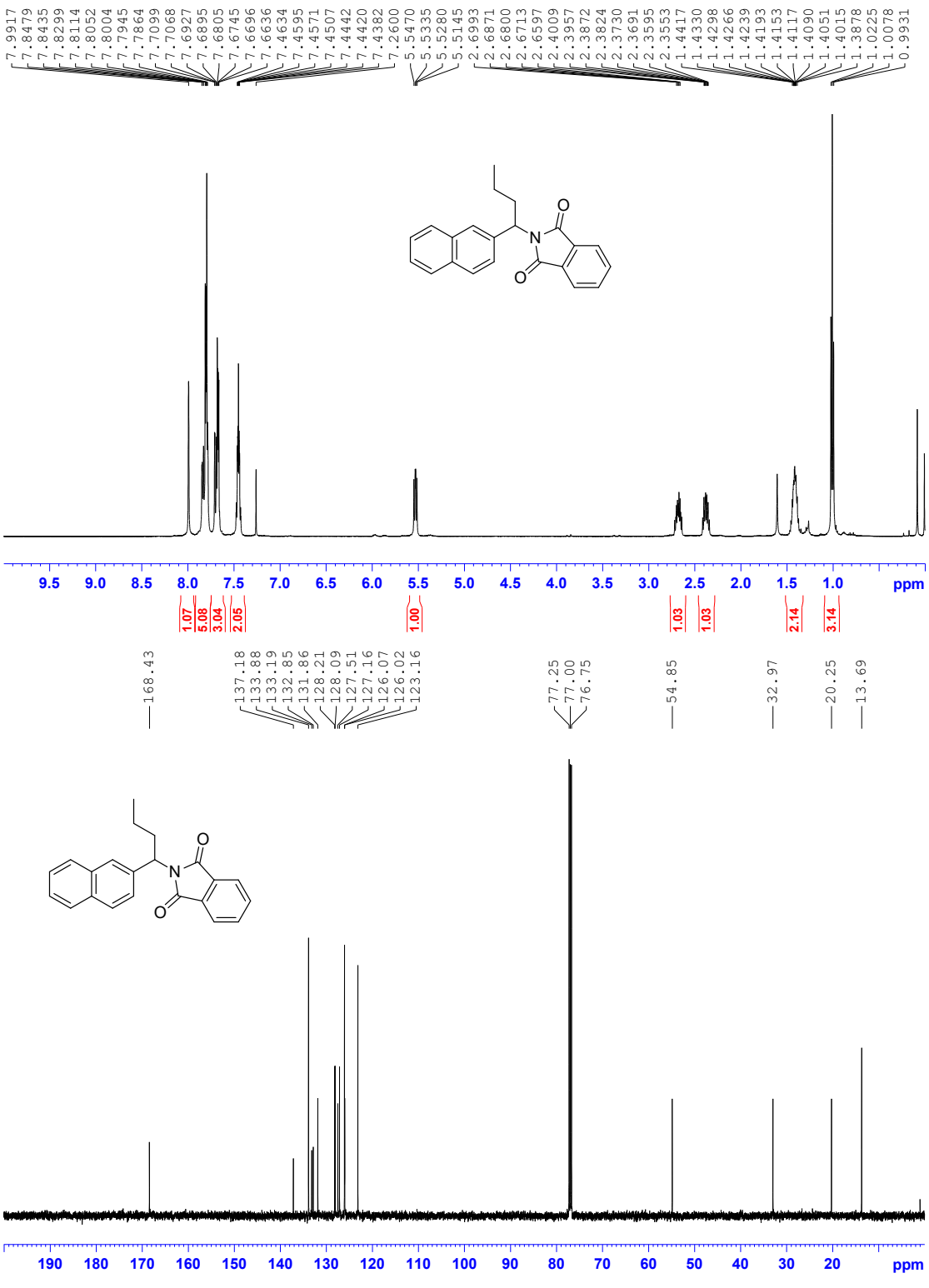
32.85
20.10
13.60
Br
O
N
O
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm



(7m)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7.8114
7.8052
7.8004
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7.7864
7.7099
7.7068
7.6927
7.6895
7.6805
7.6745
7.6696
7.6636
7.4634
7.4595
7.4571
7.4507
7.4442
7.4420
7.4382
7.2600
5.5470
5.5335
5.5280
5.5145
2.6993
2.6871
2.6800
2.6713
2.6597
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2.3957
2.3872
2.3824
2.3730
2.3691
2.3595
2.3553
1.4417
1.4330
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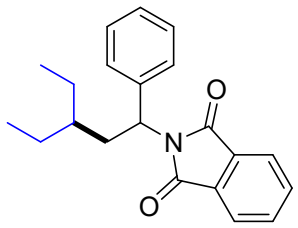
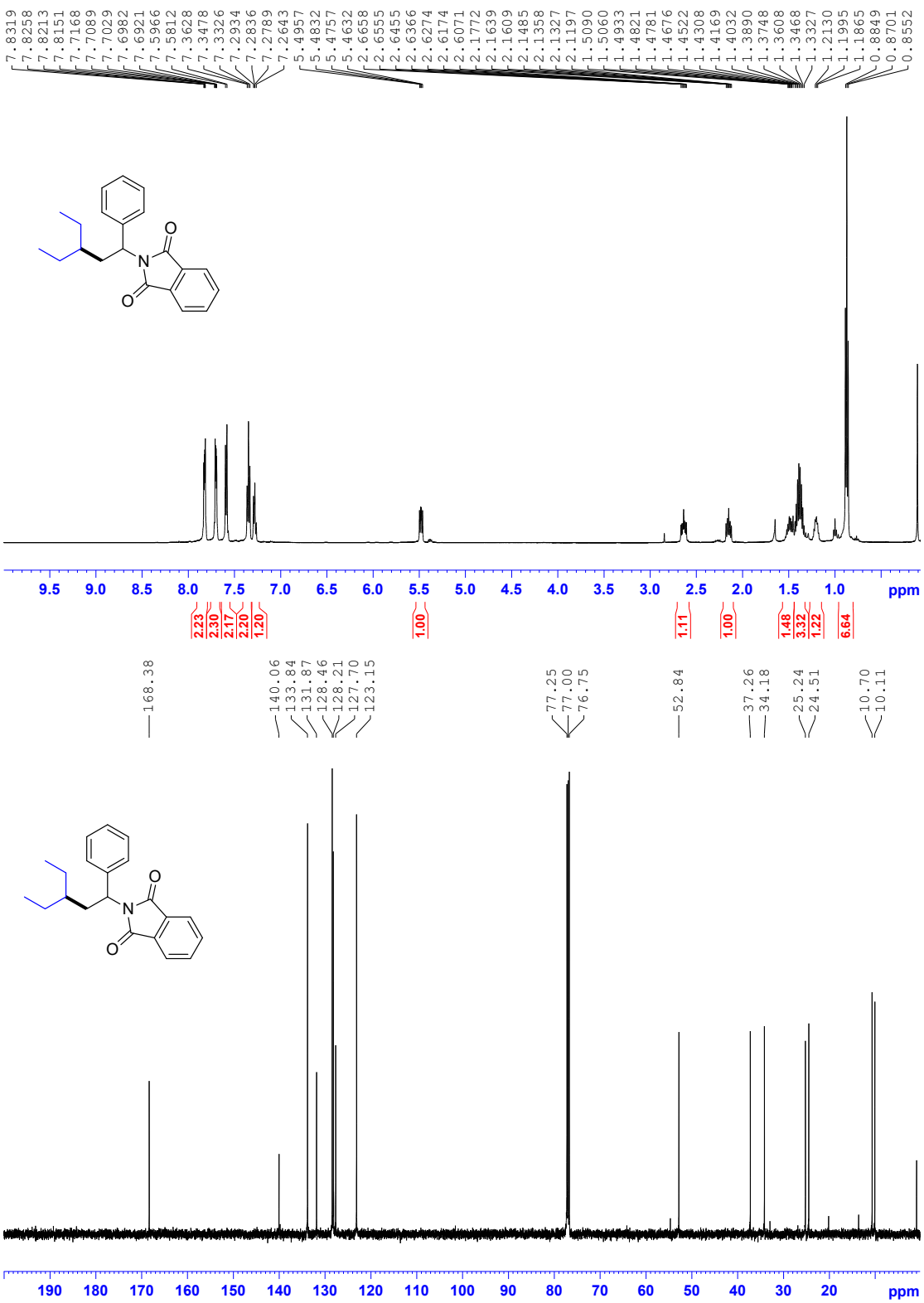
1.4015
1.3878
1.0225
1.0078
0.9931
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
1.07
5.08
3.04
2.05
1.00
1.03
1.03
2.14
3.14
168.43
137.18
133.88
133.19
132.85
131.86
128.21
128.09
127.51
127.16
126.07
126.02
123.16
77.25
77.00
76.75
54.85
32.97
20.25
13.69
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm



(9a)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7.8258
7.8213
7.8151
7.7168
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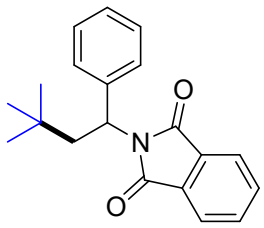
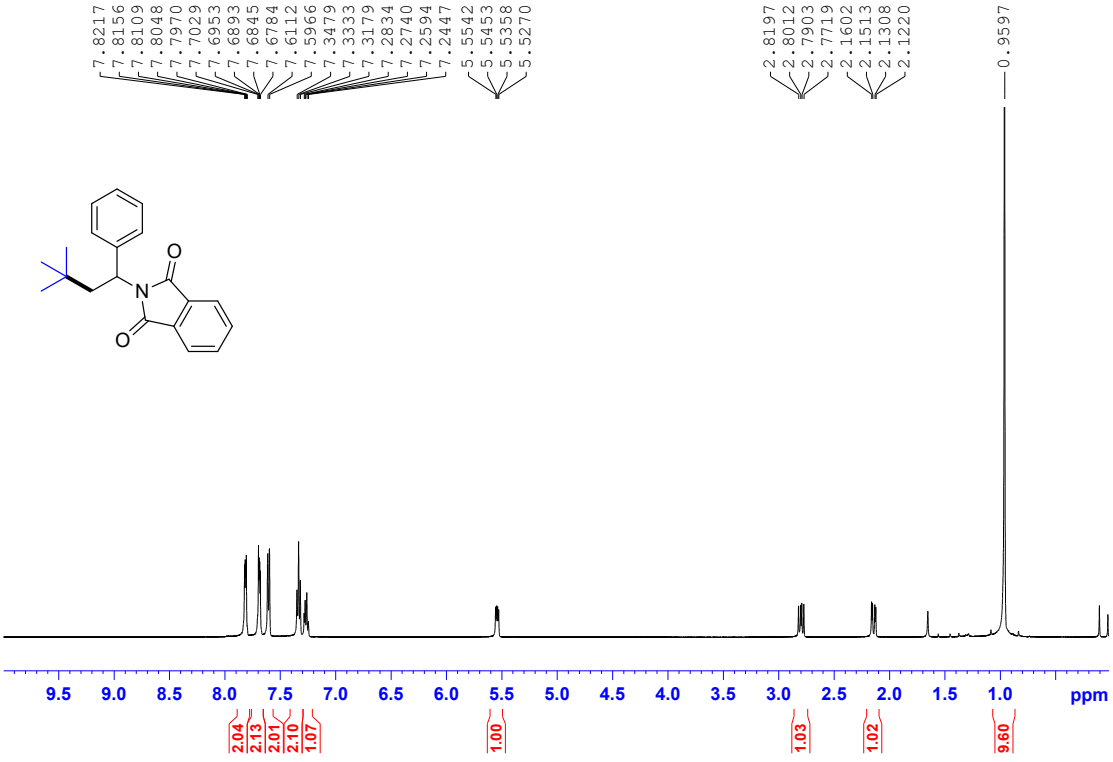
7.7029
7.6982
7.6921
7.5966
7.5812
7.3628
7.3478
7.3326
7.2934
7.2836
7.2789
7.2643
5.4957
5.4832
5.4757
5.4632
2.6658
2.6555
2.6455
2.6366
2.6274
2.6174
2.6071
2.1772
2.1639
2.1609
2.1485
2.1358
2.1327
2.1197
1.5090
1.5060
1.4933
1.4821
1.4781
1.4676
1.4522
1.4308
1.4169
1.4032
1.3890
1.3748
1.3608
1.3468
1.3327
1.2130
1.1995
1.1865
0.8849
0.8701
0.8552
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 ppm
2.23
2.30
2.17
2.20
1.20
1.00
1.11
1.00
1.48
3.32
1.22
6.64
168.38
140.06
133.84
131.87
128.46
128.21
127.70
123.15
77.25
77.00
76.75
52.84
37.26
34.18
25.24
24.51
10.70
10.11
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm



(9b)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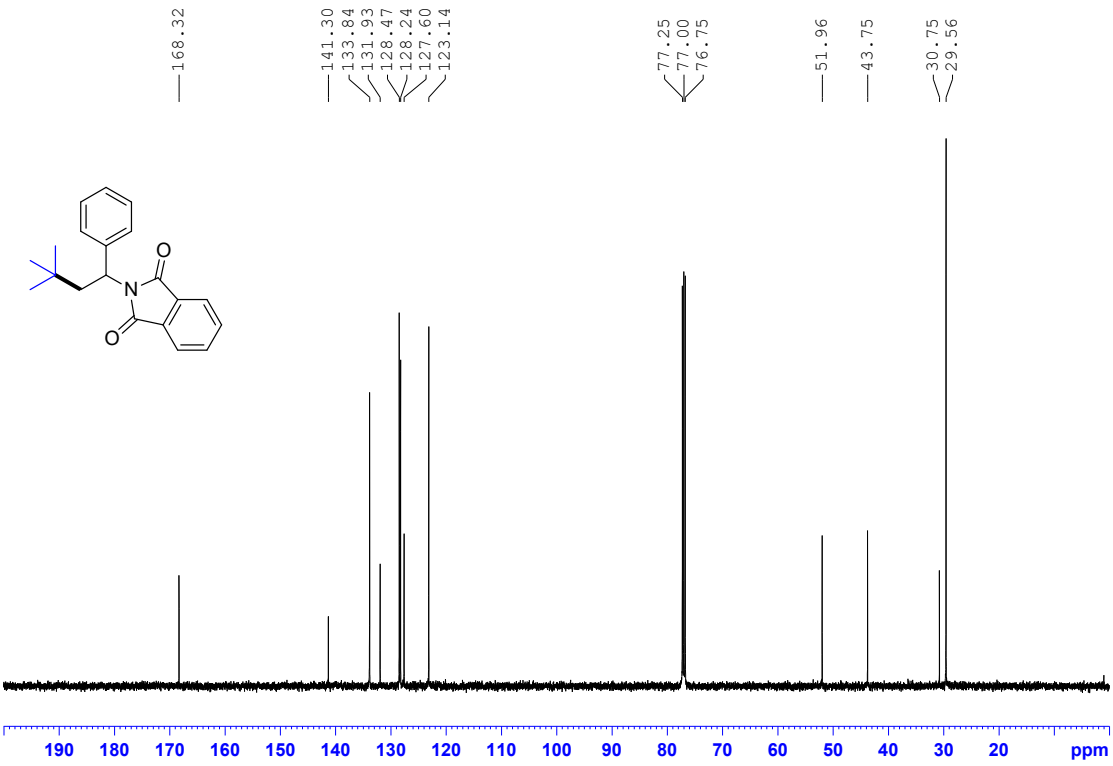
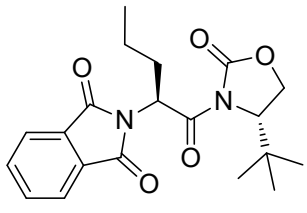
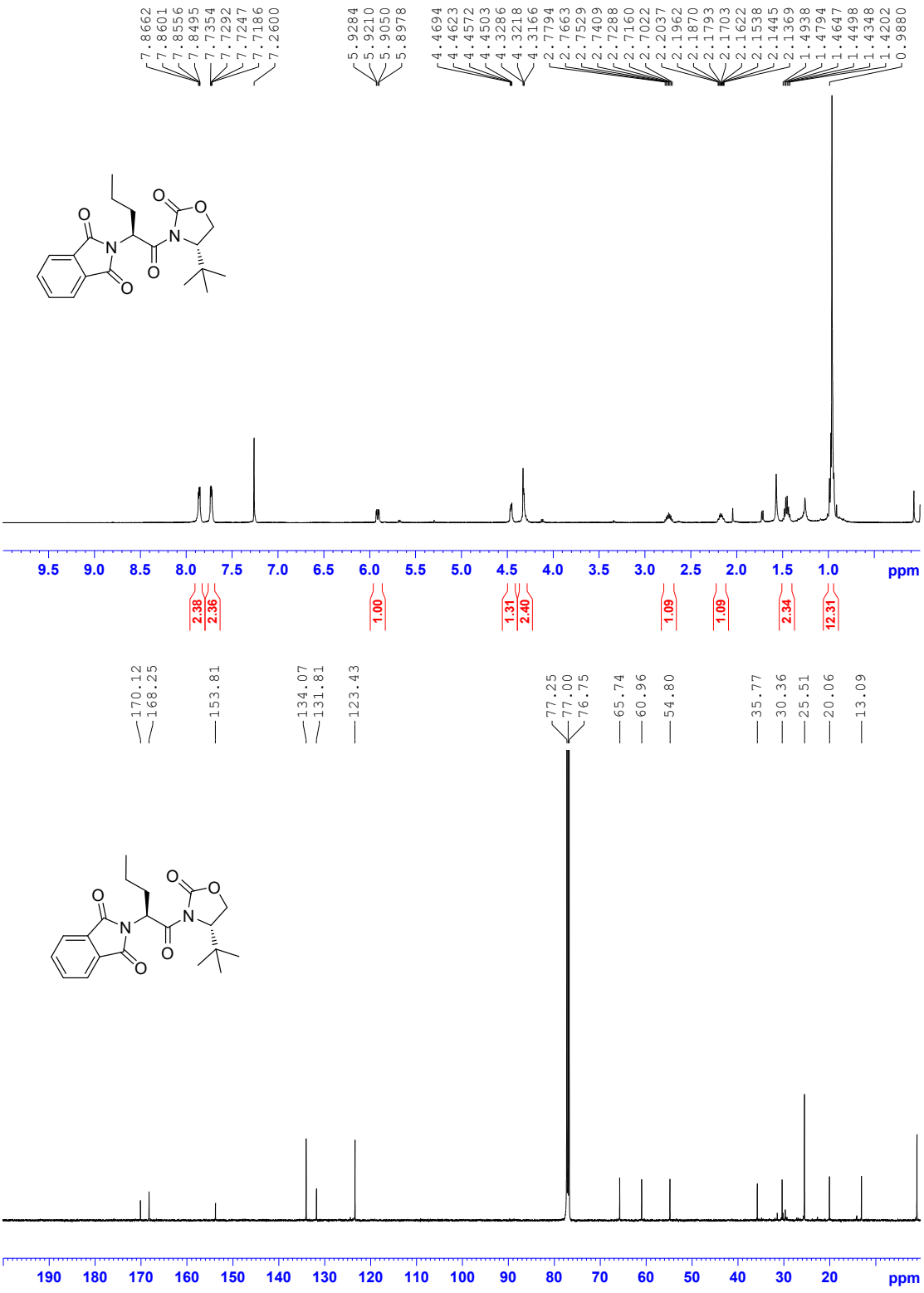
7.8156
7.8109
7.8048
7.7970
7.7029
7.6953
7.6893
7.6845
7.6784
7.6112
7.5966
7.3479
7.3333
7.3179
7.2834
7.2740
7.2594
7.2447
5.5542
5.5453
5.5358
5.5270
2.8197
2.8012
2.7903
2.7719
2.1602
2.1513
2.1308
2.1220
0.9597
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
2.04
2.13
2.01
2.10
1.07
1.00
1.03
1.02
9.60


168.32
141.30
133.84
131.93
128.47
128.24
127.60
123.14
77.25
77.00
76.75
51.96
43.75
30.75
29.56
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm



(11)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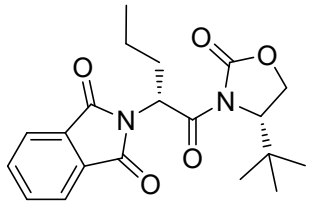
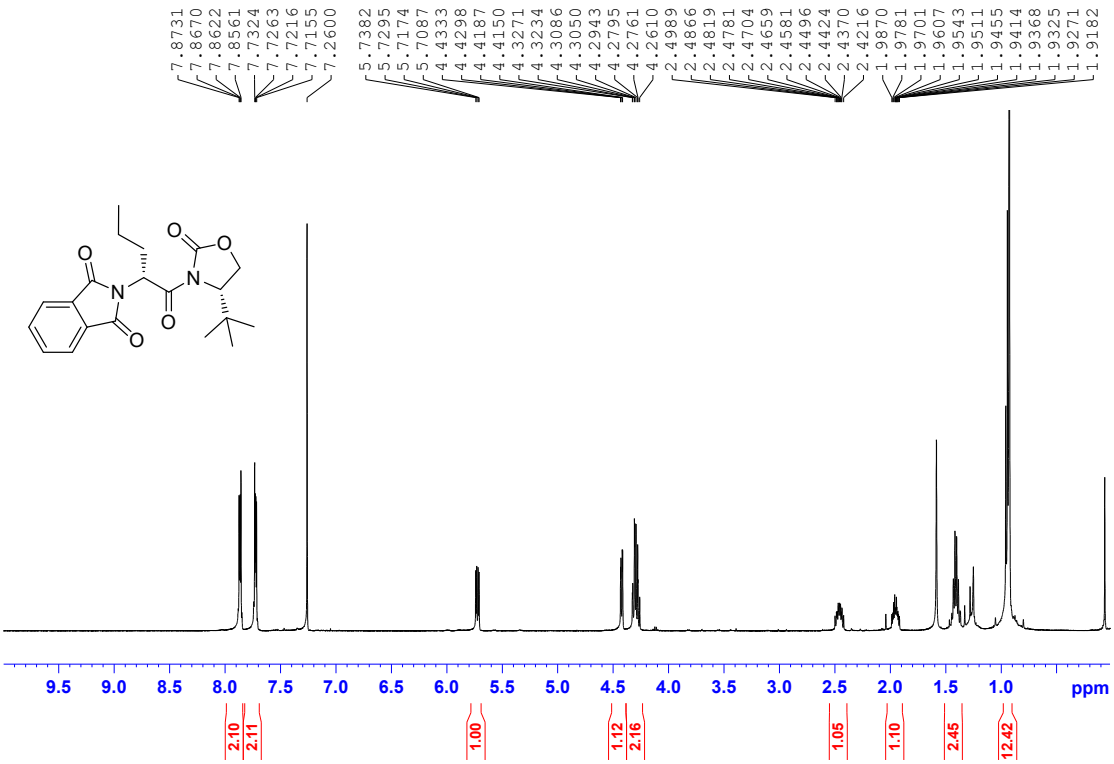
7.8556
7.8495
7.7354
7.7292
7.7247
7.7186
7.2600
5.9284
5.9210
5.9050
5.8978
4.4694
4.4623
4.4572
4.4503
4.3286
4.3218
4.3166
2.7794
2.7663
2.7529
2.7409
2.7288
2.7160
2.7022
2.2037
2.1962
2.1870
2.1793
2.1703
2.1622
2.1538
2.1445
2.1369
1.4938
1.4794
1.4647
1.4498
1.4348
1.4202
0.9880
9.5 9.0 8.5 8.0 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 ppm
2.38
2.36
1.00
1.31
2.40
1.09
1.09
2.34
12.31
170.12
168.25
153.81
134.07
131.81
123.43
77.25
77.00
76.75
65.74
60.96
54.80
35.77
30.36
25.51
20.06
13.09
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm



(**11'**)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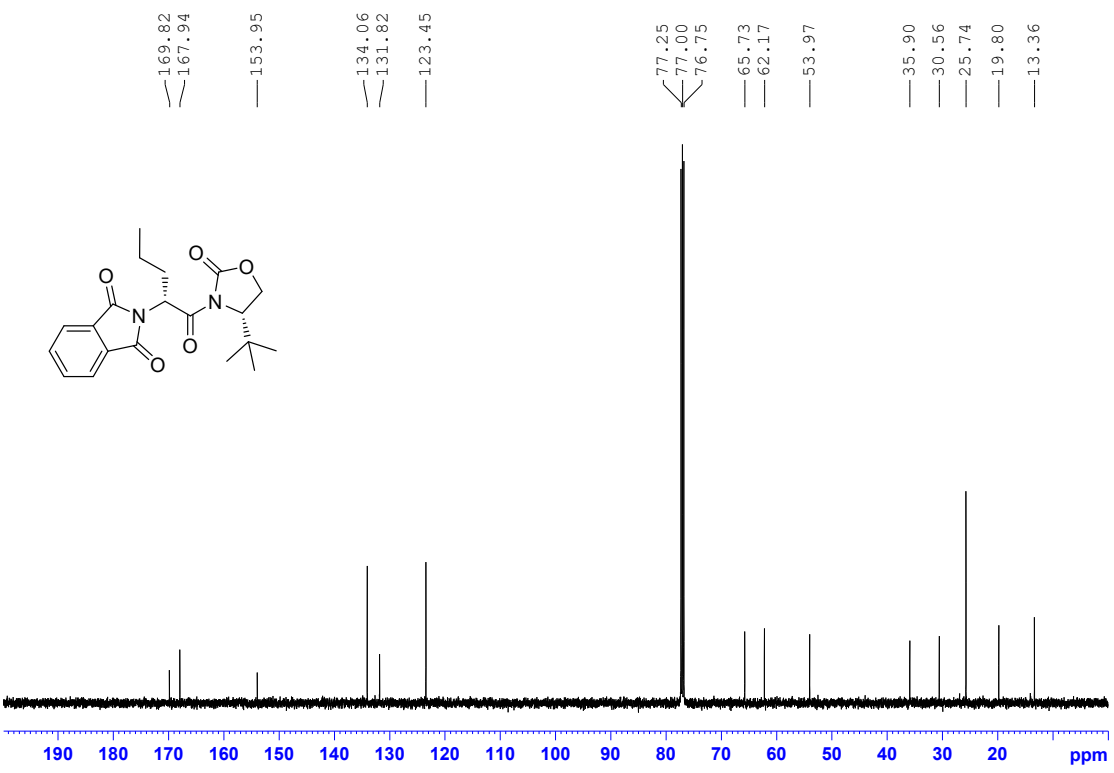
7.8670
7.8622
7.8561
7.7324
7.7263
7.7216
7.7155
7.2600
5.7382
5.7295
5.7174
5.7087
4.4333
4.4298
4.4187
4.4150
4.3271
4.3234
4.3086
4.3050
4.2943
4.2795
4.2761
4.2610
2.4989
2.4866
2.4819
2.4781
2.4704
2.4659
2.4581
2.4496
2.4424
2.4370
2.4216
1.9870
1.9781
1.9701
1.9607
1.9543
1.9511
1.9455
1.9414
1.9368
1.9325
1.9271
1.9182
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 ppm
2.10
2.11
1.00
1.12
2.16
1.05
1.10
2.45
12.42


169.82
167.94
153.95
134.06
131.82
123.45
77.25
77.00
76.75
65.73
62.17
53.97
35.90
30.56
25.74
19.80
13.36
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm