Electronic Supplementary Material (ESI) for RSC Advances

Highly enantioselective aza-Michael addition reactions of 4-nitrophthalimide to α, β-unsaturated ketones

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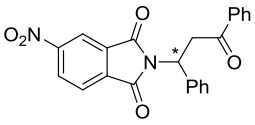
General Information: All the solvents were purified according to standard procedures. Catalysts **III**, **IV** and **V** were synthesized by literature procedure.[1] The ^{1}H NMR spectra were recorded at 400 MHz and ^{13}C NMR spectra were recorded at 100 MHz with Bruker AV400 spectrometer. ^{1}H and ^{13}C NMR chemical shifts were calibrated to tetramethylsilane as an external reference. Coupling constants are given in hertz. The following abbreviations are used to indicate the multiplicity: s, singlet; d, double; t, triplet; m, multiplet. HR-MS were recorded on an IonSpec FT-ICR mass spectrometer with ESI resource. All melting points were determined on a RY-I apparatus and are uncorrected. Optical rotations were measured with a Perkin–Elmer 341 polarimeter at 25 °C. HPLC analysis was performed on Shimadzu CTO-10AS by using a Chiralpak AD-H column purchased from Daicel Chemical Industries. The chemicals were purchased from commercial suppliers (Aldrich, USA and Shanghai Chemical Company, China), and were used without purification prior to use. All reactions unless otherwise noted were carried out directly under air.

Experimental Section:

General procedure for enantioselective aza-Michael addition of 4-nitrophthalimides 1b with α, β-unsaturated ketones 2:

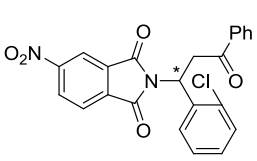
2-Cl-PhCOOH (6 mg, 40 mol%) and 4 Å MS (5 mg) were added to a stirred solution of catalyst **V** (7 mg, 20 mol%) in $CHCl_3$ (1 mL), and the solution was stirred for 5 minutes at room temperature. After addition of α, β-unsaturated ketones (0.1 mmol), the mixture was stirred for 10 minutes, then 4-nitrophthalimides (19 mg, 0.1 mmol) was added and stirring was continued for the indicated time. The crude reaction mixture was then loaded onto a silica gel column for purification (EtOAc/petroleum, 1:5) to afford the corresponding Michael adducts **3**.

Characterization of adducts 3:



5-nitro-2-(3-oxo-1,3-diphenylpropyl)isoindoline-1,3-dione (**3ba**).

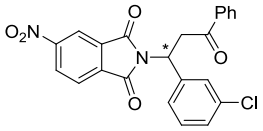
White solid, m.p. 63-65°C, 55% yield, $[\alpha]_D^{25}$ = +31.00 (*c* 0.2, CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.59 (s, 1H, O_2N-Ar*H*), 8.55 (d, *J* = 8.1 Hz, 1H, O_2N-Ar*H*), 7.97 (t, *J* = 7.5 Hz, 3H, Ar*H*), 7.67 – 7.53 (m, 3H, Ar*H*), 7.46 (t, *J* = 7.6 Hz, 2H, Ar*H*), 7.34 (dt, *J* = 23.6, 7.2 Hz, 3H, Ar*H*), 6.07 (dd, *J* = 10.3, 4.6 Hz, 1H, NC*H*), 4.69 (dd, *J* = 18.3, 10.3 Hz, 1H, C*H*HCO), 3.76 (dd, *J* = 18.3, 4.6 Hz, 1H, CH*H*CO). ^{13}C NMR (100 MHz, $CDCl_3$) δ 196.7, 166.2, 166.0, 151.7, 138.7, 136.3, 136.2, 133.7, 133.3, 129.2, 129.0, 128.8, 128.5, 128.1, 127.9, 124.5, 118.8, 51.3, 39.8. HRMS calcd for $C_{23}H_{16}N_2O_5Na$, $[M+Na]^+$ 423.0957, found 423.0950. The *ee* was determined by HPLC using a Chiralpak AD-H column [hexane/*i*-PrOH (80:20)]; flow rate 1.0 $mLmin^{-1}$; major = 30.2 min, minor = 37.3 min (>99% *ee*).



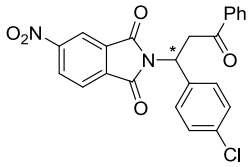
2-(1-(2-chlorophenyl)-3-oxo-3-phenylpropyl)-5-nitroisoindoline-1,3-dione (**3bb**).

White solid, m.p. 73-75°C, 61% yield, $[\alpha]_D^{25}$ = +34.46 (*c* 0.2, CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.65 (s, 1H, O_2N-Ar*H*), 8.60 (d, *J* = 8.1 Hz, 1H, O_2N-Ar*H*), 8.00 (dd, *J* = 26.9, 8.0 Hz, 3H, Ar*H*), 7.73 – 7.54 (m, 2H, Ar*H*), 7.53 – 7.39 (m, 3H, Ar*H*), 7.36 – 7.20 (m, 2H, Ar*H*), 6.48 (dd, *J* = 10.5, 4.3 Hz, 1H, NC*H*), 4.57 (dd, *J* = 18.1, 10.6 Hz, 1H, C*H*HCO), 3.74 (dd, *J* = 18.1, 4.3 Hz, 1H, CH*H*CO). ^{13}C NMR (100 MHz, $CDCl_3$) δ 196.5, 166.3, 166.1, 151.8, 136.2, 136.0, 133.7, 133.16, 133.0, 130.1, 129.5, 129.3, 128.9, 128.8, 128.1, 127.3, 124.7, 118.9, 48.8, 39.5. HRMS calcd for $C_{23}H_{15}ClN_2O_5Na$, $[M+Na]^+$ 457.0567, found 457.0557. The *ee* was determined by HPLC using a Chiralpak AD-H

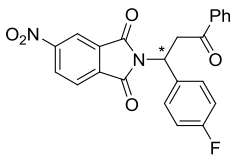
column [hexane/*i*-PrOH (80:20)]; flow rate 1.0 mLmin^{-1}; major = 26.6 min, minor = 28.6 min (95% *ee*).



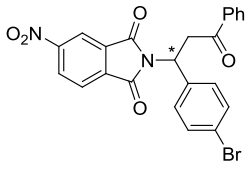
2-(1-(3-chlorophenyl)-3-oxo-3-phenylpropyl)-5-nitroisoindoline-1,3-dione (**3bc**). White solid, m.p. 66-68°C, 65% yield, $[\alpha]_D^{25}$ = +30.33 (*c* 0.2, CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.64 (d, *J* = 1.7 Hz, 1H, O_2N-Ar*H*), 8.59 (dd, *J* = 8.1, 1.9 Hz, 1H, O_2N-Ar*H*), 8.01 (dd, *J* = 16.4, 7.8 Hz, 3H, Ar*H*), 7.66 – 7.57 (m, 2H, Ar*H*), 7.49 (dd, *J* = 9.6, 5.9 Hz, 3H, Ar*H*), 7.36 – 7.27 (m, 2H, Ar*H*), 6.06 (dd, *J* = 10.0, 4.8 Hz, 1H, NC*H*), 4.66 (dd, *J* = 18.3, 10.1 Hz, 1H, C*H*HCO), 3.79 (dd, *J* = 18.3, 4.8 Hz, 1H, CH*H*CO). ^{13}C NMR (100 MHz, $CDCl_3$) δ 196.3, 166.1, 165.9, 151.8, 140.5, 136.1, 136.0, 134.8, 133.8, 133.1, 130.3, 129.3, 128.8, 128.7, 128.1, 126.1, 124.7, 118.9, 50.7, 39.6. HRMS calcd for $C_{23}H_{15}ClN_2O_5Na$, $[M+Na]^+$ 457.0567, found 457.0565. The *ee* was determined by HPLC using a Chiralpak AD-H column [hexane/*i*-PrOH (80:20)]; flow rate 1.0 mLmin^{-1}; major = 28.6 min, minor = 32.1 min (98% *ee*).



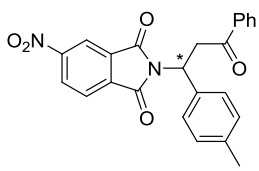
2-(1-(4-chlorophenyl)-3-oxo-3-phenylpropyl)-5-nitroisoindoline-1,3-dione (**3bd**). White solid, m.p. 73-75°C, 56% yield, $[\alpha]_D^{25}$ = +25.98 (*c* 0.2, CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.63 (s, 1H, O_2N-Ar*H*), 8.58 (d, *J* = 8.1 Hz, 1H, O_2N-Ar*H*), 8.00 (dd, *J* = 13.9, 8.0 Hz, 3H, Ar*H*), 7.59 (dd, *J* = 17.1, 7.8 Hz, 3H, Ar*H*), 7.49 (t, *J* = 7.5 Hz, 2H, Ar*H*), 7.36 (d, *J* = 8.2 Hz, 2H, Ar*H*), 6.07 (dd, *J* = 9.7, 4.9 Hz, 1H, NC*H*), 4.62 (dd, *J* = 18.2, 9.9 Hz, 1H, C*H*HCO), 3.81 (dd, *J* = 18.2, 4.9 Hz, 1H, CH*H*CO). ^{13}C NMR (100 MHz,) δ 196.3, 166.1, 165.9, 151.8, 137.1, 136.1, 136.1, 134.4, 133.8, 133.2, 129.4, 129.3, 129.2, 128.8, 128.1, 124.6, 118.8, 50.6, 39.7. HRMS calcd for $C_{23}H_{15}ClN_2O_5Na$, $[M+Na]^+$ 457.0567, found 457.0567. The *ee* was determined by HPLC using a Chiralpak AD-H column [hexane/*i*-PrOH (80:20)]; flow rate 1.0 mLmin^{-1}; major = 36.9 min, minor = 63.3 min (99% *ee*).



2-(1-(4-fluorophenyl)-3-oxo-3-phenylpropyl)-5-nitroisoindoline-1,3-dione (**3be**). White solid, m.p. 66-68°C, 60%yield, $[\alpha]_D^{25}$ = +38.00 (*c* 0.2, CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.63 (d, *J* = 1.6 Hz, 1H, O_2N-Ar*H*), 8.58 (dd, *J* = 8.1, 1.9 Hz, 1H, O_2N-Ar*H*), 8.00 (dd, *J* = 12.3, 7.8 Hz, 3H, Ar*H*), 7.62 (dt, *J* = 13.9, 6.8 Hz, 3H, Ar*H*), 7.49 (t, *J* = 7.7 Hz, 2H, Ar*H*), 7.08 (t, *J* = 8.6 Hz, 2H, Ar*H*), 6.08 (dd, *J* = 9.9, 4.9 Hz, 1H, NC*H*), 4.64 (dd, *J* = 18.2, 10.0 Hz, 1H, C*H*HCO), 3.79 (dt, *J* = 19.9, 9.9 Hz, 1H, CH*H*CO). ^{13}C NMR (100 MHz, $CDCl_3$) δ 196.4, 166.1, 165.9, 151.8, 136.2, 136.1, 134.5, 133.7, 133.2, 129.9, 129.8, 129.3, 128.8, 128.1, 124.6, 118.8, 116.0, 115.8, 50.5, 39.8. HRMS calcd for $C_{23}H_{15}FN_2O_5Na$, $[M+Na]^+$ 441.0863, found 441.0860. The *ee* was determined by HPLC using a Chiralpak AD-H column [hexane/*i*-PrOH (80:20)]; flow rate 1.0 mLmin^{-1}; major = 34.4 min, minor = 54.0 min (>99% *ee*).

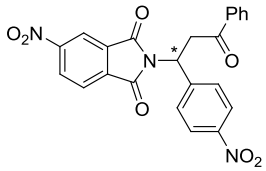


2-(1-(4-bromophenyl)-3-oxo-3-phenylpropyl)-5-nitroisoindoline-1,3-dione (**3bf**). White solid, m.p. 70-72°C, 62% yield, $[\alpha]_D^{25}$ = +23.44 (*c* 0.2, CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.62 (s, 1H, O_2N-Ar*H*), 8.58 (dd, *J* = 8.1, 1.6 Hz, 1H, O_2N-Ar*H*), 8.00 (dd, *J* = 14.2, 7.9 Hz, 3H, Ar*H*), 7.61 (t, *J* = 7.3 Hz, 1H, Ar*H*), 7.55 – 7.44 (m, 6H, Ar*H*), 6.06 (dd, *J* = 9.8, 5.0 Hz, 1H, NC*H*), 4.61 (dd, *J* = 18.2, 9.8 Hz, 1H, C*H*HCO), 3.81 (dd, *J* = 18.2, 5.0 Hz, 1H, CH*H*CO). ^{13}C NMR (100 MHz, $CDCl_3$) δ 196.3, 166.1, 165.9, 151.8, 137.6, 136.1, 136.1, 133.8, 133.1, 132.1, 129.7, 129.3, 128.8, 128.1, 124.6, 122.6, 118.8, 50.6, 39.6. HRMS calcd for $C_{23}H_{15}BrN_2O_5Na$, $[M+Na]^+$ 501.0062, found 501.0053. The *ee* was determined by HPLC using a Chiralpak AD-H column [hexane/*i*-PrOH (80:20)]; flow rate 1.0 mLmin^{-1}; major = 40.6 min, minor = 68.7 min (99% *ee*).

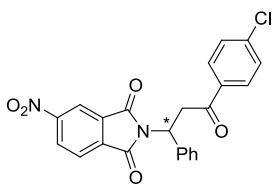


5-nitro-2-(3-oxo-3-phenyl-1-p-tolylpropyl)isoindoline-1,3-dione (**3bg**). White solid, m.p. 71-73°C, 69% yield, $[\alpha]_D^{25}$ = +28.81 (*c* 0.2, CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.59 (s, 1H, O_2N-Ar*H*), 8.54 (d, *J* = 8.2 Hz, 1H, O_2N-Ar*H*), 7.97 (dd, *J* = 8.0, 3.9 Hz, 3H, Ar*H*), 7.57 (t, *J* = 7.0 Hz, 1H, Ar*H*), 7.53 – 7.41 (m, 4H, Ar*H*), 7.18 (d, *J* = 7.8 Hz, 2H, Ar*H*), 6.04 (dd, *J* = 10.2, 4.6 Hz, 1H, NC*H*), 4.67 (dd, *J* = 18.2, 10.3 Hz, 1H, C*H*HCO),

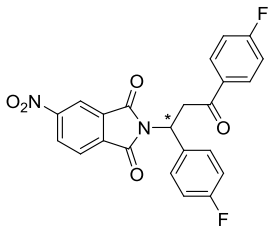
3.75 (dd, *J* = 18.2, 4.6 Hz, 1H, CH*H*CO), 2.33 (s, 3H, C*H*$_3$). ^{13}C NMR (100 MHz, $CDCl_3$) δ 196.7, 166.2, 166.0, 151.7, 138.4, 136.3, 136.2, 135.7, 133.6, 133.3, 129.6, 129.2, 128.8, 128.1, 127.8, 124.5, 118.7, 51.0, 39.8, 21.1. HRMS calcd for $C_{24}H_{18}N_2O_5Na$, $[M+Na]^+$ 437.1113, found 437.1104 The *ee* was determined by HPLC using a Chiralpak AD-H column [hexane/*i*-PrOH (80:20)]; flow rate 1.0 mLmin^{-1}; major = 32.3 min, minor = 48.5 min (99% *ee*).



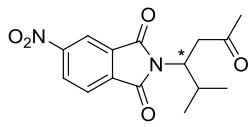
5-nitro-2-(1-(4-nitrophenyl)-3-oxo-3-phenylpropyl)isoindoline-1,3-dione (**3bh**). White solid, m.p. 93-95°C, 49% yield, $[\alpha]_D^{25}$ = +13.96 (*c* 0.2, CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.65 (s, 1H, Ar*H*), 8.64 – 8.57 (m, 1H, Ar*H*), 8.25 (d, *J* = 8.7 Hz, 2H, Ar*H*), 8.02 (dd, *J* = 25.8, 8.0 Hz, 3H, Ar*H*), 7.81 (d, *J* = 8.6 Hz, 2H, Ar*H*), 7.62 (t, *J* = 7.4 Hz, 1H, Ar*H*), 7.50 (t, *J* = 7.7 Hz, 2H, Ar*H*), 6.20 (dd, *J* = 9.2, 5.5 Hz, 1H, NC*H*), 4.58 (dd, *J* = 18.2, 9.3 Hz, 1H, C*H*HCO), 3.93 (dd, *J* = 18.2, 5.5 Hz, 1H, CH*H*CO). ^{13}C NMR (100 MHz, $CDCl_3$) δ 195.8, 166.0, 165.8, 151.9, 147.8, 145.4, 135.9, 135.8, 134.0, 133.0, 129.5, 129.0, 128.9, 128.1, 124.8, 124.2, 119.0, 50.5, 39.5. HRMS calcd for $C_{23}H_{15}N_3O_7Na$, $[M+Na]^+$ 468.0808, found 468.0800. The *ee* was determined by HPLC using a Chiralpak AD-H column [hexane/*i*-PrOH (70:30)]; flow rate 1.0 mLmin^{-1}; major = 70.6 min, minor = 84.4 min (>99% *ee*).



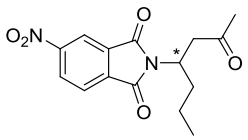
2-(3-(4-chlorophenyl)-3-oxo-1-phenylpropyl)-5-nitroisoindoline-1,3-dione (**3bi**). White solid, m.p. 178-179°C, 54% yield, $[\alpha]_D^{25}$ = +41.49 (*c* 0.2, CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.60 (s, 1H, O_2N-Ar*H*), 8.56 (d, *J* = 8.1 Hz, 1H, O_2N-Ar*H*), 7.99 (d, *J* = 8.1 Hz, 1H, Ar*H*), 7.91 (d, *J* = 8.4 Hz, 2H, Ar*H*), 7.59 (d, *J* = 7.4 Hz, 2H, Ar*H*), 7.43 (d, *J* = 8.4 Hz, 2H, Ar*H*), 7.35 (dt, *J* = 22.3, 7.1 Hz, 3H, Ar*H*), 6.05 (dd, *J* = 10.2, 4.5 Hz, 1H, NC*H*), 4.66 (dd, *J* = 18.3, 10.3 Hz, 1H, C*H*HCO), 3.72 (dd, *J* = 18.2, 4.6 Hz, 1H, CH*H*CO). ^{13}C NMR (100 MHz, $CDCl_3$) δ 195.5, 166.2, 166.0, 151.7, 140.2, 138.5, 136.2, 134.5, 133.2, 129.5, 129.3, 129.1, 129.0, 128.6, 127.8, 124.6, 118.8, 51.2, 39.8. HRMS calcd for $C_{23}H_{15}ClN_2O_5Na$, $[M+Na]^+$ 457.0567, found 457.0563. The *ee* was determined by HPLC using a Chiralpak AD-H column [hexane/*i*-PrOH (80:20)]; flow rate 1.0 mLmin^{-1}; major = 59.2 min, minor = 63.5 min (99% *ee*).



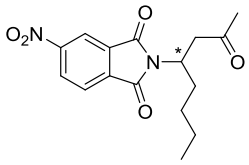
2-(1,3-bis(4-fluorophenyl)-3-oxopropyl)-5-nitroisoindoline-1,3-dione (**3bj**). White solid, m.p. 70-71°C, 71% yield, $[\alpha]_D^{25}$ = +16.52 (*c* 0.2, CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.63 (s, 1H, O_2N-Ar*H*), 8.59 (dd, *J* = 8.1, 1.7 Hz, 1H, O_2N-Ar*H*), 8.02 (dd, *J* = 8.2, 3.8 Hz, 3H, Ar*H*), 7.62 (dd, *J* = 8.5, 5.3 Hz, 2H, Ar*H*), 7.16 (t, *J* = 8.5 Hz, 2H, Ar*H*), 7.08 (t, *J* = 8.6 Hz, 2H, Ar*H*), 6.07 (dd, *J* = 9.9, 4.9 Hz, 1H, , NC*H*), 4.63 (dd, *J* = 18.2, 10.0 Hz, 1H, C*H*HCO), 3.77 (dd, *J* = 18.2, 5.0 Hz, 1H, CH*H*CO). ^{13}C NMR (100 MHz, $CDCl_3$) δ 194.8, 166.1, 165.9, 151.8, 136.1, 134.5, 134.4, 133.2, 132.6, 130.9, 130.8, 129.9, 129.8, 129.3, 124.63, 118.8, 116.1, 116.0, 115.9, 115.8, 50.5, 39.8. HRMS calcd for $C_{23}H_{14}F_2N_2O_5Na$, $[M+Na]^+$ 459.0768, found 459.0760. The *ee* was determined by HPLC using a Chiralpak AD-H column [hexane/*i*-PrOH (80:20)]; flow rate 1.0 mLmin^{-1}; major = 45.7 min, minor = 76.3 min (99% *ee*).



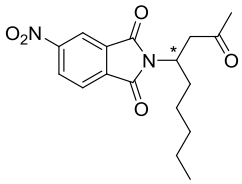
2-(2-methyl-5-oxohexan-3-yl)-5-nitroisoindoline-1,3-dione (**3bk**). Colorless oil; 75% yield, $[\alpha]_D^{25}$ = +33.05 (*c* 0.2, CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.64 (d, *J* = 1.6 Hz, 1H, Ar*H*), 8.60 (dd, *J* = 8.1, 1.9 Hz, 1H, Ar*H*), 8.03 (d, *J* = 8.1 Hz, 1H, Ar*H*), 4.45 – 4.35 (m, 1H, NC*H*), 3.58 – 3.45 (m, 1H, C*H*HCO), 2.94 (dd, *J* = 17.9, 3.9 Hz, 1H, CH*H*CO), 2.35 – 2.22 (m, 1H, CH_3CHCH_3), 2.14 (s, 3H, COC*H*$_3$), 1.04 (d, *J* = 6.7 Hz, 3H, C*H*$_3$CHCH$_3$), 0.90 (t, *J* = 7.5 Hz, 3H, CH_3CHCH_3). ^{13}C NMR (100 MHz, $CDCl_3$) δ 206.2, 166.5, 166.2, 151.7, 136.1, 133.1, 129.2, 124.5, 118.7, 53.7, 42.5, 30.7, 30.2, 19.9, 19.6. HRMS calcd for $C_{15}H_{16}N_2O_5Na$, $[M+Na]^+$ 327.0957, found 327.0954. The *ee* was determined by HPLC using a Chiralpak AD-H column [hexane/*i*-PrOH (80:20)]; flow rate 1.0 mLmin^{-1}; minor = 9.1 min, major = 9.6 min (97% *ee*).



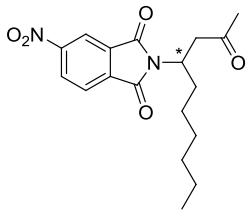
5-nitro-2-(2-oxoheptan-4-yl)isoindoline-1,3-dione (**3bl**). Colorless oil; 88% yield, $[\alpha]_D^{25}$ = +18.89 (*c* 0.2, CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.65 (d, *J* = 1.5 Hz, 1H, Ar*H*), 8.61 (dd, *J* = 8.1, 1.9 Hz, 1H, Ar*H*), 8.03 (d, *J* = 8.1 Hz, 1H, Ar*H*), 4.82 – 4.69 (m, 1H, NC*H*), 3.39 (dd, *J* = 17.9, 9.3 Hz, 1H, C*H*HCO), 2.97 (dd, *J* = 17.9, 5.2 Hz, 1H, CH*H*CO), 2.16 (s, 3H, COCH_3), 2.08 – 1.94 (m, 1H, NCHC*H*HCH$_2$), 1.75 – 1.61 (m, 1H, NCHCH*H*CH$_2$), 1.40 – 1.21 (m, 2H, CH_2CH$_3$), 0.93 (q, *J* = 7.7 Hz, 3H, CH$_2$CH_3). ^{13}C NMR (100 MHz, $CDCl_3$) δ 205.8, 166.3, 166.1, 151.7, 136.2, 133.2, 129.2, 124.4, 118.7, 47.5, 45.2, 34.5, 30.2, 19.5, 13.6. HRMS calcd for $C_{15}H_{16}N_2O_5Na$, $[M+Na]^+$ 327.0957, found 327.0948. The *ee* was determined by HPLC using a Chiralpak AD-H column [hexane/*i*-PrOH (80:20)]; flow rate 1.0 mLmin^{-1}; minor = 9.7 min, major = 10.4 min (96% *ee*).



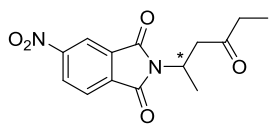
5-nitro-2-(2-oxooctan-4-yl)isoindoline-1,3-dione (**3bm**). Colorless oil, 89% yield, $[\alpha]_D^{25}$ = +22.69 (*c* 0.2, CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.63 (d, *J* = 1.5 Hz, 1H, Ar*H*), 8.59 (dd, *J* = 8.1, 2.0 Hz, 1H, Ar*H*), 8.02 (d, *J* = 8.2 Hz, 1H, Ar*H*), 4.78 – 4.64 (m, 1H, NC*H*), 3.37 (dd, *J* = 17.9, 9.3 Hz, 1H, C*H*HCO), 2.96 (dd, *J* = 17.9, 5.2 Hz, 1H, CH*H*CO), 2.14 (s, 3H, COCH_3), 2.05 – 1.90 (m, 1H, NCHC*H*HCH$_2$), 1.79 – 1.60 (m, 1H, NCHCH*H*CH$_2$), 1.43 – 1.12 (m, 4H, C$_2H_4$CH$_3$), 0.86 (t, *J* = 7.1 Hz, 3H, C$_2$H$_4$CH_3). ^{13}C NMR (100 MHz, $CDCl_3$) δ 205.8, 166.3, 166.1, 151.7, 136.2, 133.2, 129.2, 124.5, 118.7, 47.7, 45.2, 32.2, 30.2, 28.4, 22.2, 13.9. HRMS calcd for $C_{16}H_{18}N_2O_5Na$, $[M+Na]^+$ 341.1113, found 341.1107. The *ee* was determined by HPLC using a Chiralpak AD-H column [hexane/*i*-PrOH (80:20)]; flow rate 1.0 mLmin^{-1}; minor = 9.1 min, major = 9.6 min (95% *ee*).



5-nitro-2-(2-oxononan-4-yl)isoindoline-1,3-dione (**3bn**). Colorless oil, 98% yield, $[\alpha]_D^{25}$ = +24.04 (*c* 0.2, CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.63 (d, *J* = 1.4 Hz, 1H, Ar*H*), 8.59 (dd, *J* = 8.1, 1.8 Hz, 1H, Ar*H*), 8.02 (d, *J* = 8.1 Hz, 1H, Ar*H*), 4.85 – 4.57 (m, 1H, NC*H*), 3.53 – 3.25 (m, 1H, C*H*HCO), 2.95 (dd, *J* = 17.9, 5.2 Hz, 1H, CH*H*CO), 2.14 (s, 3H, COCH_3), 2.03 – 1.90 (m, 1H, NCHC*H*HCH$_2$), 1.70 (tt, *J* = 10.4, 5.1 Hz, 1H, NCHCH*H*CH$_2$), 1.29 (dd, *J* = 22.9, 4.0 Hz, 6H, C$_3H_6$CH$_3$), 0.84 (t, *J* = 6.7 Hz, 3H, C$_3$H$_6$CH_3). ^{13}C NMR (101 MHz, $CDCl_3$) δ 205.8, 166.3, 166.0, 151.7, 136.2, 133.2, 129.2, 124.5, 118.7, 47.7, 45.2, 32.4, 31.2, 30.2, 25.9, 22.4, 14.0. HRMS calcd for $C_{17}H_{20}N_2O_5Na$, $[M+Na]^+$ 355.1270, found 355.1269. The *ee* was determined by HPLC using a Chiralpak AD-H column [hexane/*i*-PrOH (80:20)]; flow rate 1.0 mLmin^{-1}; minor = 8.9 min, major = 9.3 min (95% *ee*).



5-nitro-2-(2-oxodecan-4-yl)isoindoline-1,3-dione (**3bo**). Colorless oil, 90% yield, $[\alpha]_D^{25}$ = +26.72 (*c* 0.2, CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.63 (d, *J* = 1.5 Hz, 1H, Ar*H*), 8.59 (dd, *J* = 8.1, 2.0 Hz, 1H, Ar*H*), 8.02 (d, *J* = 8.0 Hz, 1H, Ar*H*), 4.77 – 4.66 (m, 1H, NC*H*), 3.37 (dd, *J* = 17.9, 9.3 Hz, 1H, C*H*HCO), 2.95 (dd, *J* = 17.9, 5.2 Hz, 1H, CH*H*CO), 2.14 (s, 3H, COCH_3), 2.04 – 1.92 (m, 1H, NCHC*H*HCH$_2$), 1.70 (ddd, *J* = 17.7, 9.7, 4.6 Hz, 1H, NCHCH*H*CH$_2$), 1.36 – 1.14 (m, 8H, C$_4H_8$CH$_3$), 0.84 (t, *J* = 6.9 Hz, 3H, C$_4$H$_8$CH_3). ^{13}C NMR (101 MHz, $CDCl_3$) δ 205.8, 166.3, 166.1, 151.7, 136.2, 133.2, 129.2, 124.5, 118.7, 47.8, 45.2, 32.5, 31.6, 30.2, 28.8, 26.2, 22.5, 14.0. HRMS calcd for $C_{18}H_{22}N_2O_5Na$, $[M+Na]^+$ 369.1426, found 369.1415. The *ee* was determined by HPLC using a Chiralpak AD-H column [hexane/*i*-PrOH (80:20)]; flow rate 1.0 mLmin^{-1}; minor = 8.7 min, major = 9.3 min (96% *ee*).



5-nitro-2-(4-oxohexan-2-yl)isoindoline-1,3-dione (**3bp**). Colorless oil, 51% yield, $[\alpha]_D^{25}$ = +21.39 (*c* 0.2, CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.65 (d, *J* = 1.5 Hz, 1H, Ar*H*), 8.60 (dd, *J* = 8.1, 1.9 Hz, 1H, Ar*H*), 8.03 (d, *J* = 8.1 Hz, 1H, Ar*H*), 4.88 (tt, *J* = 13.8, 6.9 Hz, 1H, NC*H*), 3.37 (dd, *J* = 17.8, 8.9 Hz, 1H, C*H*HCO), 2.96 (dd, *J* = 17.8, 5.7 Hz, 1H, CH*H*CO), 2.53 – 2.37 (m, 2H, CH_2CH$_3$), 1.49 (d, *J* = 7.0 Hz, 3H, NCHCH_3), 1.07 – 0.98 (t, *J* = 7.3 Hz, 3H, CH$_2$CH_3). ^{13}C NMR (101 MHz, $CDCl_3$) δ 208.4, 166.1, 165.8, 151.7, 136.4, 133.4, 129.2, 124.4, 118.6, 44.9, 43.3, 36.1,

18.9, 7.6. HRMS calcd for $C_{14}H_{14}N_2O_5Na$, $[M+Na]^+$ 313.0800, found 313.0799. The *ee* was determined by HPLC using a Chiralpak AD-H column [hexane/*i*-PrOH (80:20)]; flow rate 1.0 $mLmin^{-1}$; minor = 12.7 min, major = 14.5 min (95% *ee*).

Crystallographic Structure Determination:

Recrystallization of 3bc: 2-(1-(3-chlorophenyl)-3-oxo-3-phenylpropyl)-5-nitroisoindoline-1,3-dione (**3bc**) was recrystallized from CH_2Cl_2/hexane solvent system at room temperature.

Data of X-ray diffraction were collected at 113 K on a Rigaku 007 Saturn 70 CCD diffractometer.

Crystal data and structure refinement for **3bc**.

Identification code	shelx
Empirical formula	C23 H15 Cl N2 O5
Formula weight	434.82
Temperature	113(2) K
Wavelength	0.71073 Å
Crystal system, space group	Monoclinic, P2(1)
Unit cell dimensions	a = 5.9139(18) Å alpha = 90 deg. b = 18.664(6) Å beta = 93.314(5) deg. c = 8.834(3) Å gamma = 90 deg.
Volume	973.4(5) A^3
Z, Calculated density	2, 1.483 Mg/m^3
Absorption coefficient	0.237 mm^-1
F(000)	448
Crystal size	0.20 x 0.18 x 0.12 mm
Theta range for data collection	2.18 to 27.85 deg.
Limiting indices	-7<=h<=7, -22<=k<=24, -11<=l<=11
Reflections collected / unique	11137 / 4549 [R(int) = 0.0391]
Completeness to theta = 27.85	100.0 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9721 and 0.9541
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	4549 / 1 / 280
Goodness-of-fit on F^2	1.017
Final R indices [I>2sigma(I)]	R1 = 0.0309, wR2 = 0.0531
R indices (all data)	R1 = 0.0434, wR2 = 0.0555
Absolute structure parameter	-0.06(4)
Largest diff. peak and hole	0.214 and -0.225 e.A^-3

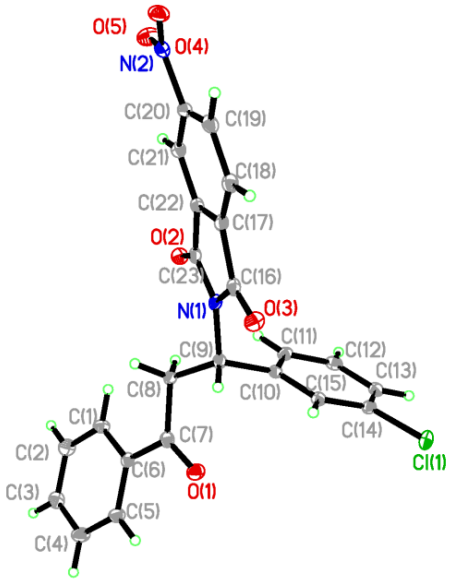
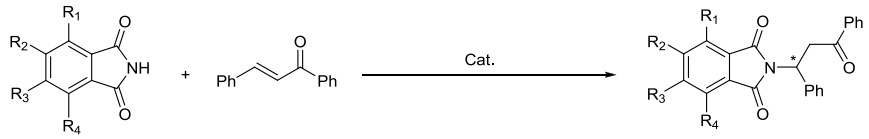


Fig. 1 Molecular structure of **3bc**

Calculation section:

Experimentally, we tried four nucleophiles (**1a** through **1d**) for the reaction as shown in Scheme 1, and the result showed that only **1b** took place nucleophilic addition with **2a** and no products were observed when the rest three nucleophiles **1a**, **1c** and **1d** were used. In order to explore the reason for this result, we performed computational study on the structures and the charge distributions of **1a**, **1b**, **1c** and **1d**. All geometry optimization, frequency and Merz–Kollman atomic charge[2] calculations were performed with B3LYP/6-31G(d) method in Gaussian 09.[3]



1 **2a** **3**

1a: $R_1=R_2=R_3=R_4=H$
1b: $R_1=R_3=R_4=H$, $R_2=NO_2$
1c: $R_1=NO_2$, $R_2=R_3=R_4=H$
1d: $R_1=R_2=R_3=R_4=Cl$

Scheme 1. Model reaction of nucleophilic addition between **1** and **2a**.

Our calculation results (Fig 2 and Table 1) showed that the N1 atom in **1b** has the most negative charge -0.633, compared with the N1 atoms in the rest three species. Moreover, the H2 atom in **1b** has the most positive charge 0.386, compared with the H2 atoms in the rest three species, which means the H2 atom in **1b** may be activated more easily. In addition, the N1-H2 bond length in **1b** is 0.00006 nm longer than that in **1a** and is the longest one among those of the four species. Therefore, it is easier for **1b** to take place nucleophilic addition than the rest three species.

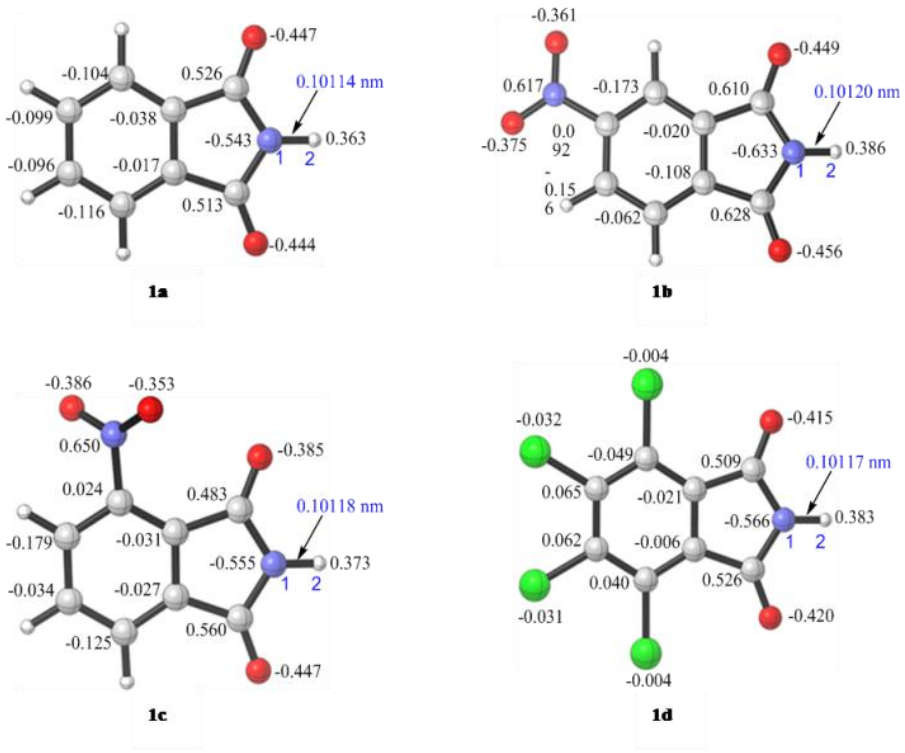


Fig 2. B3LYP/6-31G(d) calculated Merz–Kollman atomic charge and bond length in four species, **1a** through **1d**.

Table 1. B3LYP/6-31G(d) calculated Merz–Kollman atomic charge and bond length in four species, **1a** through **1d**

species	Bond length of N1–H2 (nm)	Merz–Kollman atomic charge	
		N1	H2
1a	0.10114	-0.543	0.363
1b	0.10120	-0.633	0.386
1c	0.10118	-0.555	0.373
1d	0.10117	-0.566	0.383

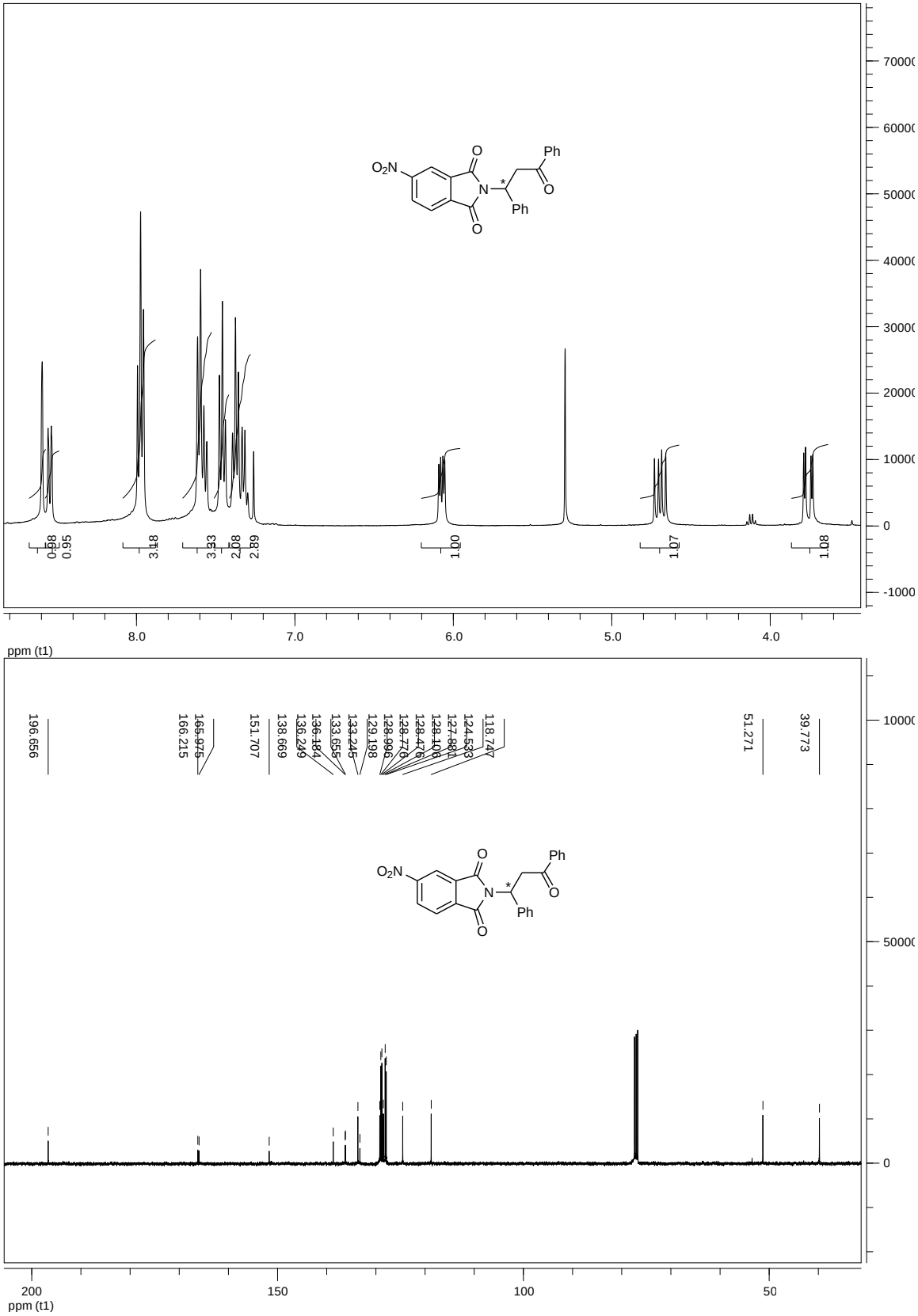
References:

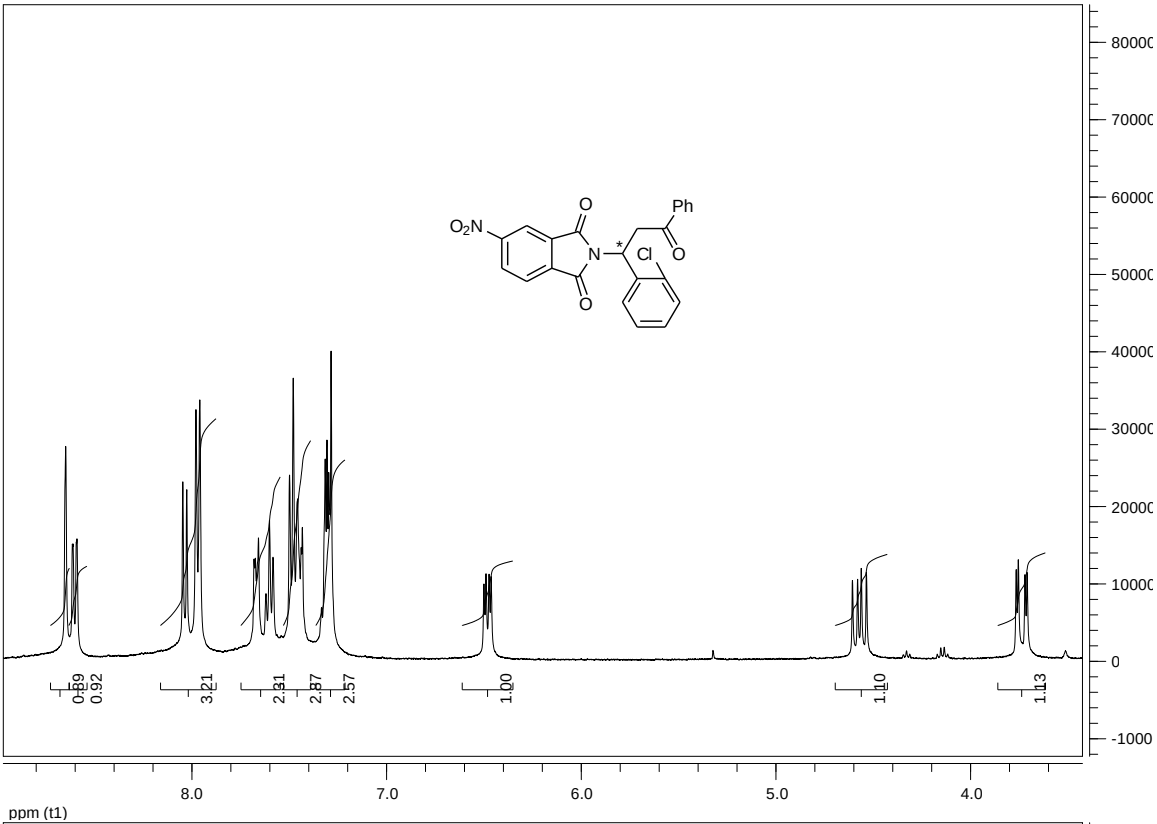
[1] a) A. Berkessel, B. Seelig, *Synthesis* **2009**, 2113 – 2115; b) B. Vakulya, S. Varga, A. Csámpai, T. Soós, *Org. Lett.* **2005**, 1967–1969.

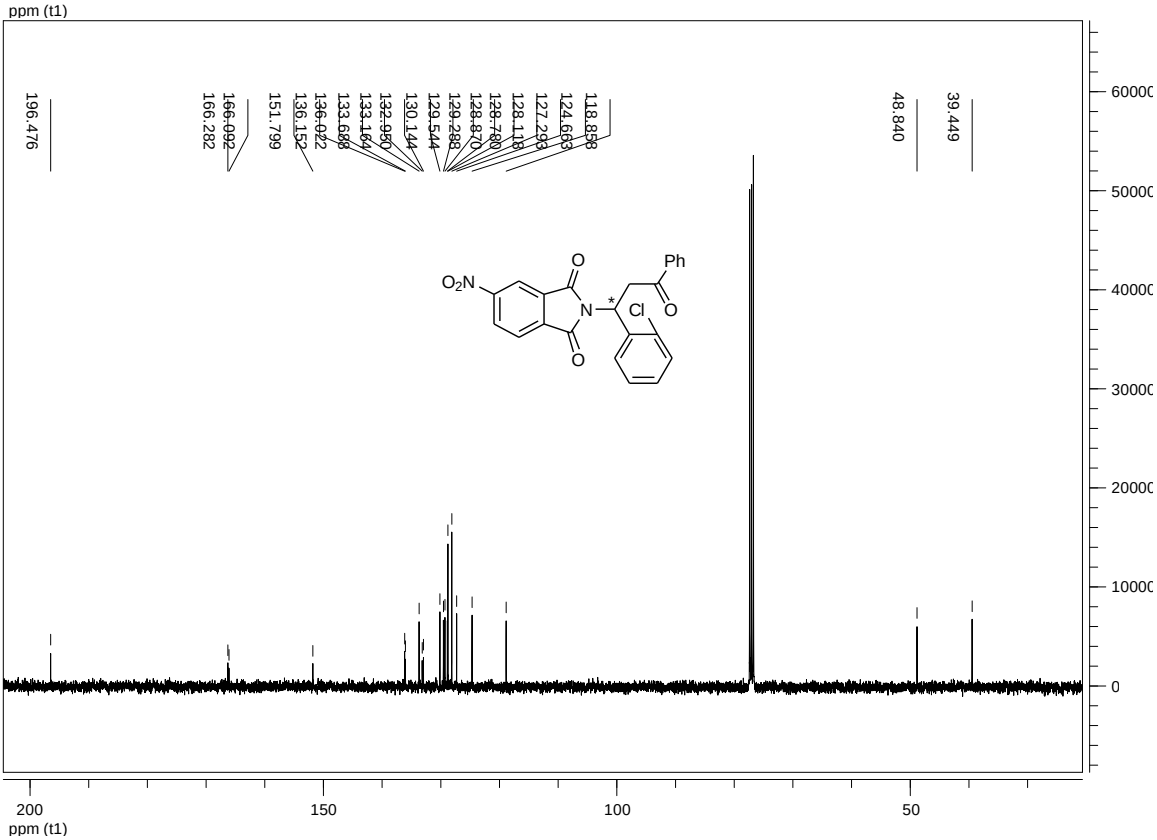
[2] Singh, U. C. and Kollman, P. A. *J. Comp. Chem.*, **5** (1984) 129-45.

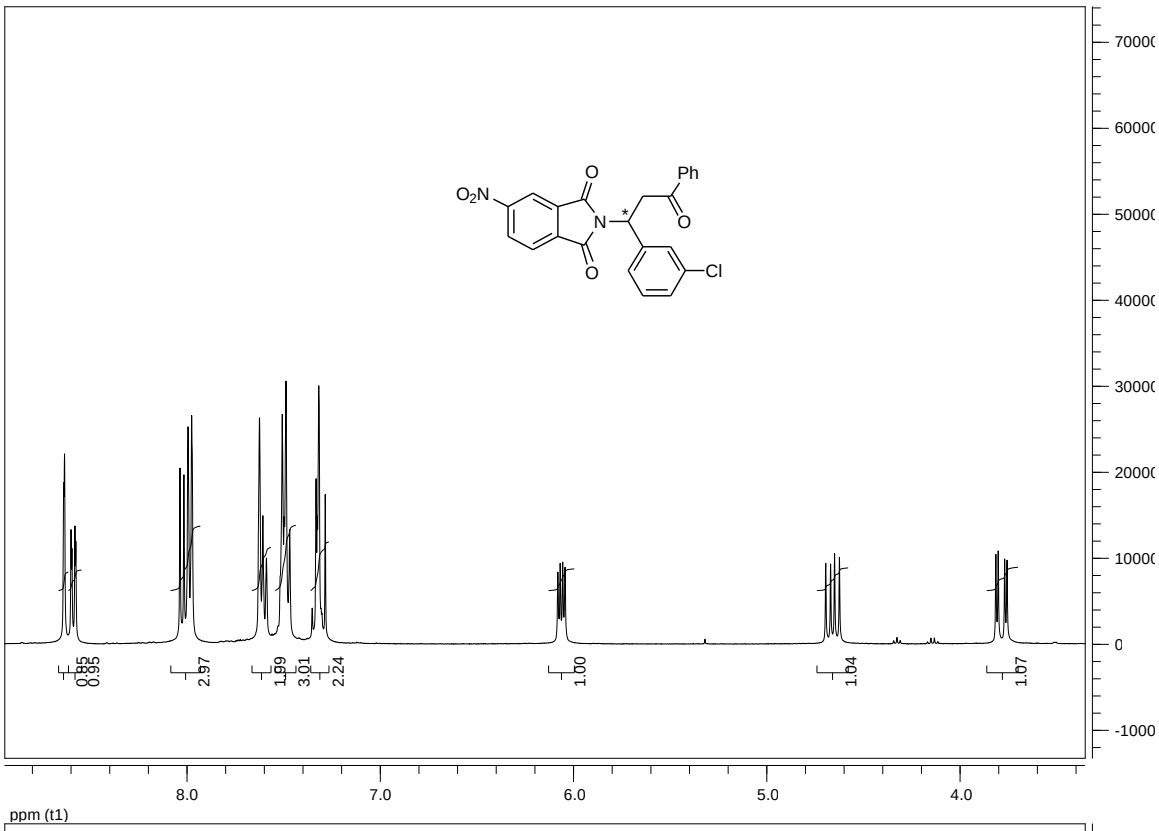
[3] Frisch, M. J.; et al. *Gaussian 09*, revision B.01; Gaussian, Inc.: Wallingford, CT, 2010.

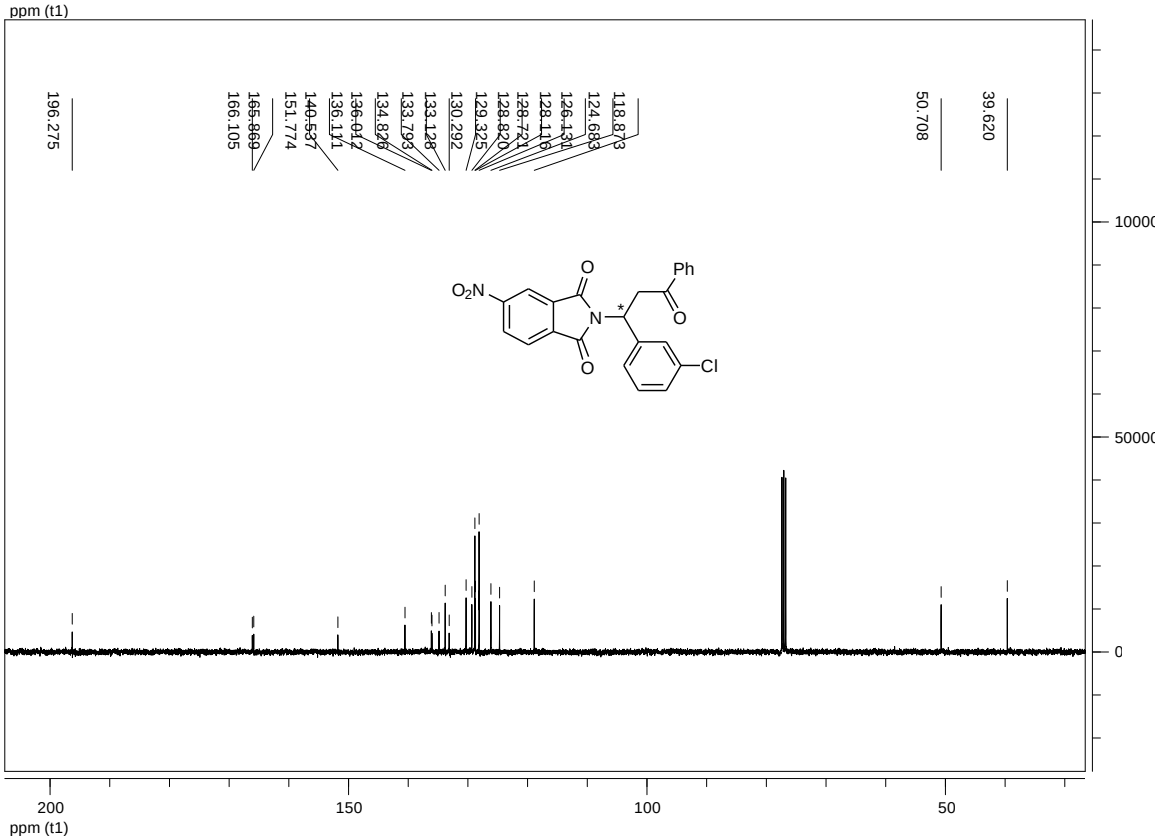
^{1}H and ^{13}C NMR spectrum of Michael adducts 3:

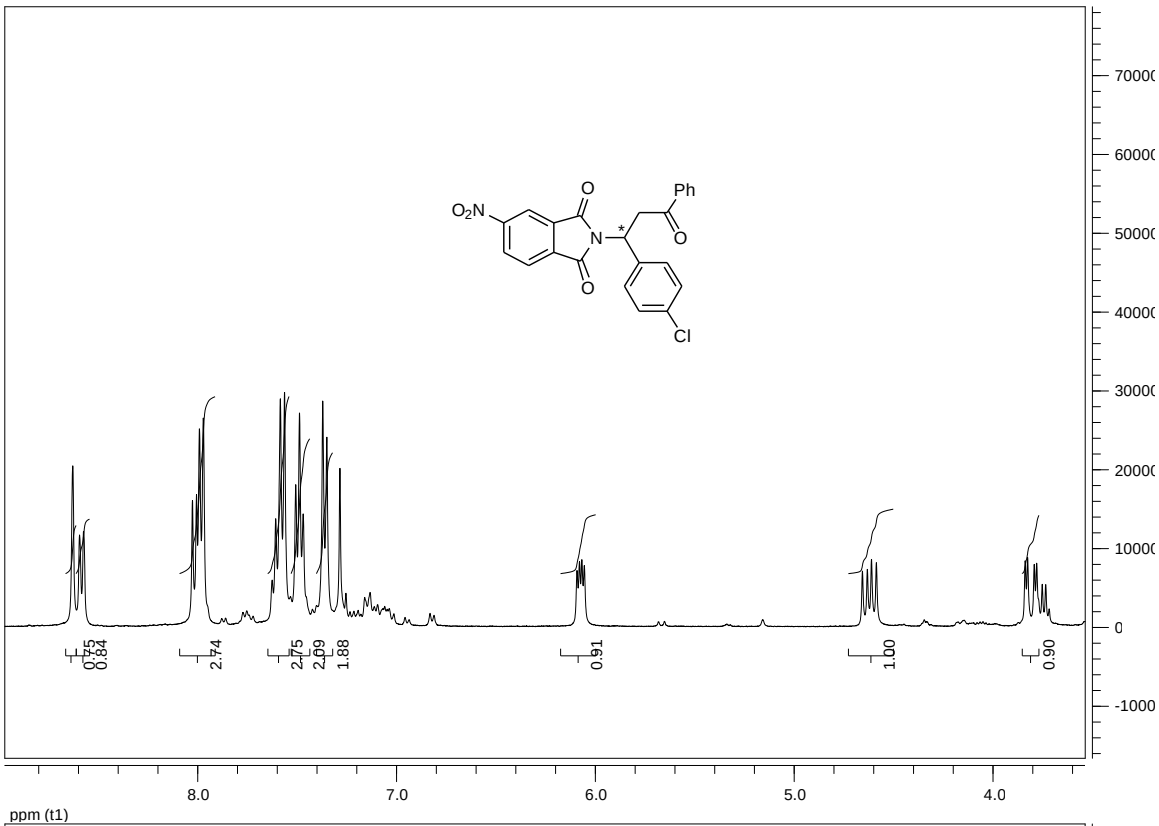


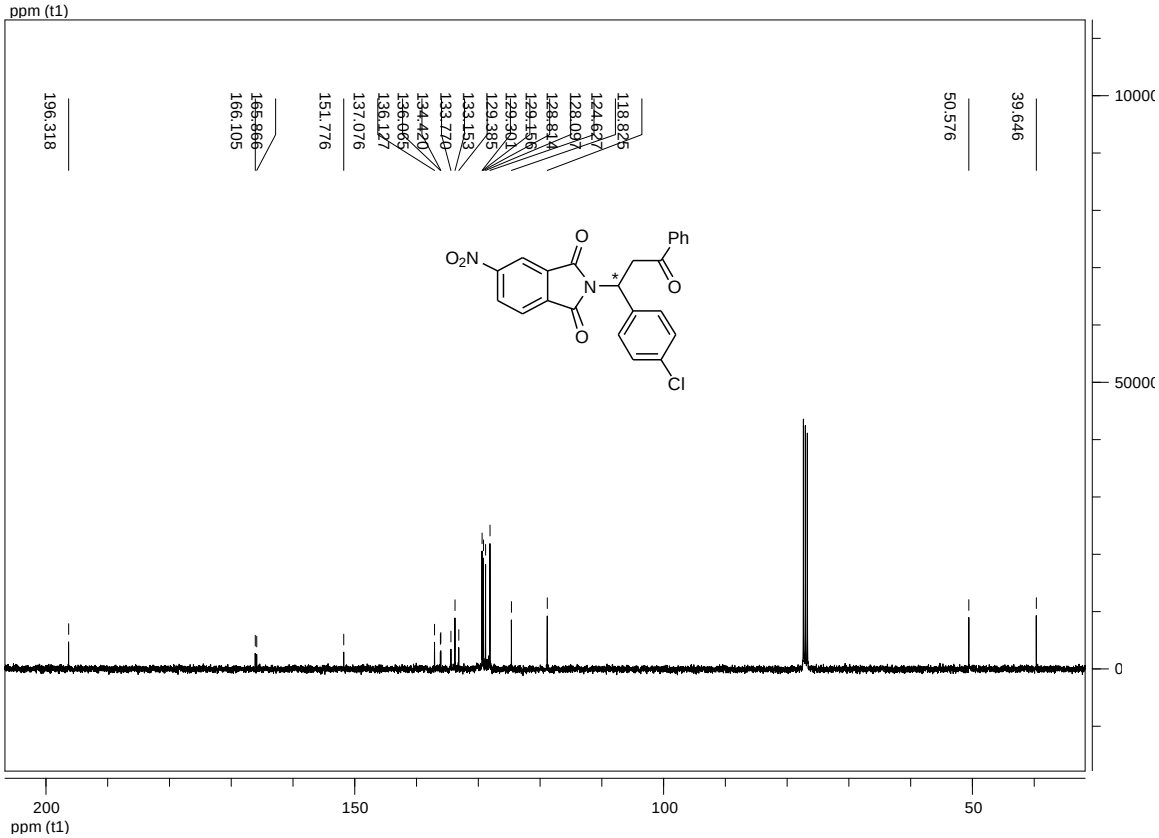


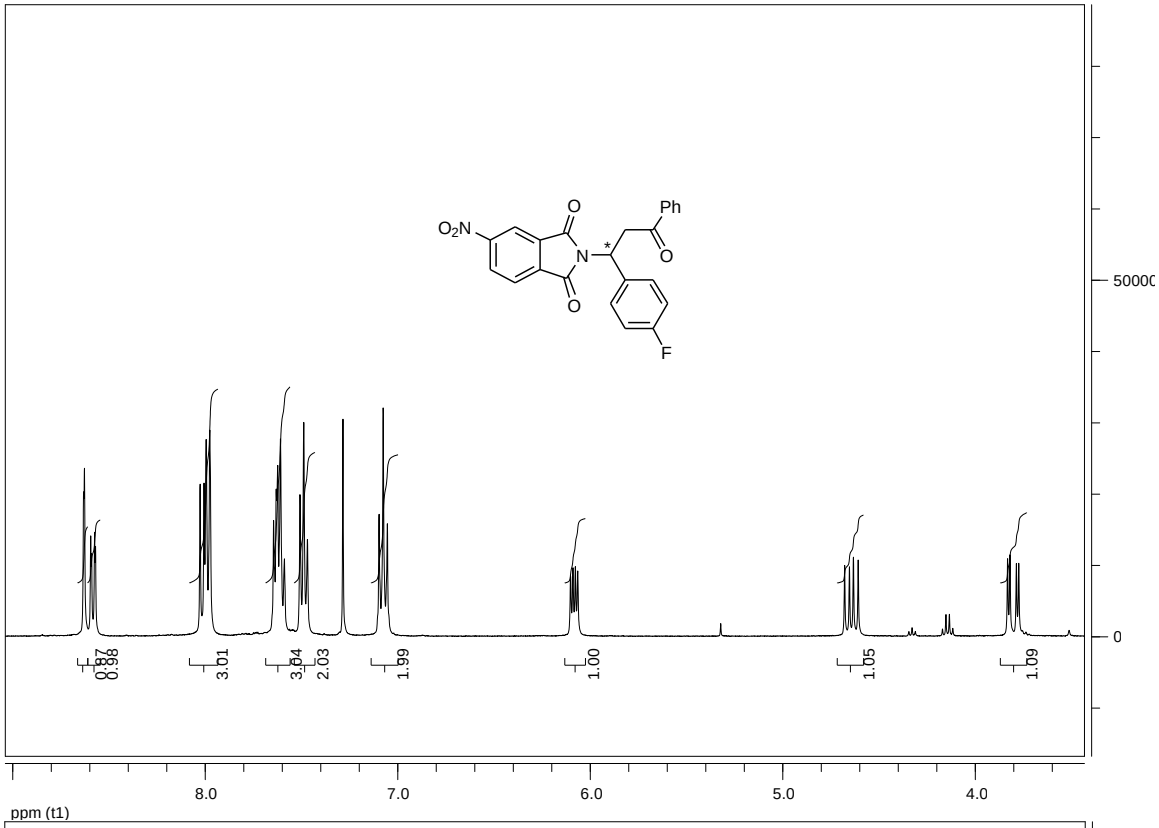


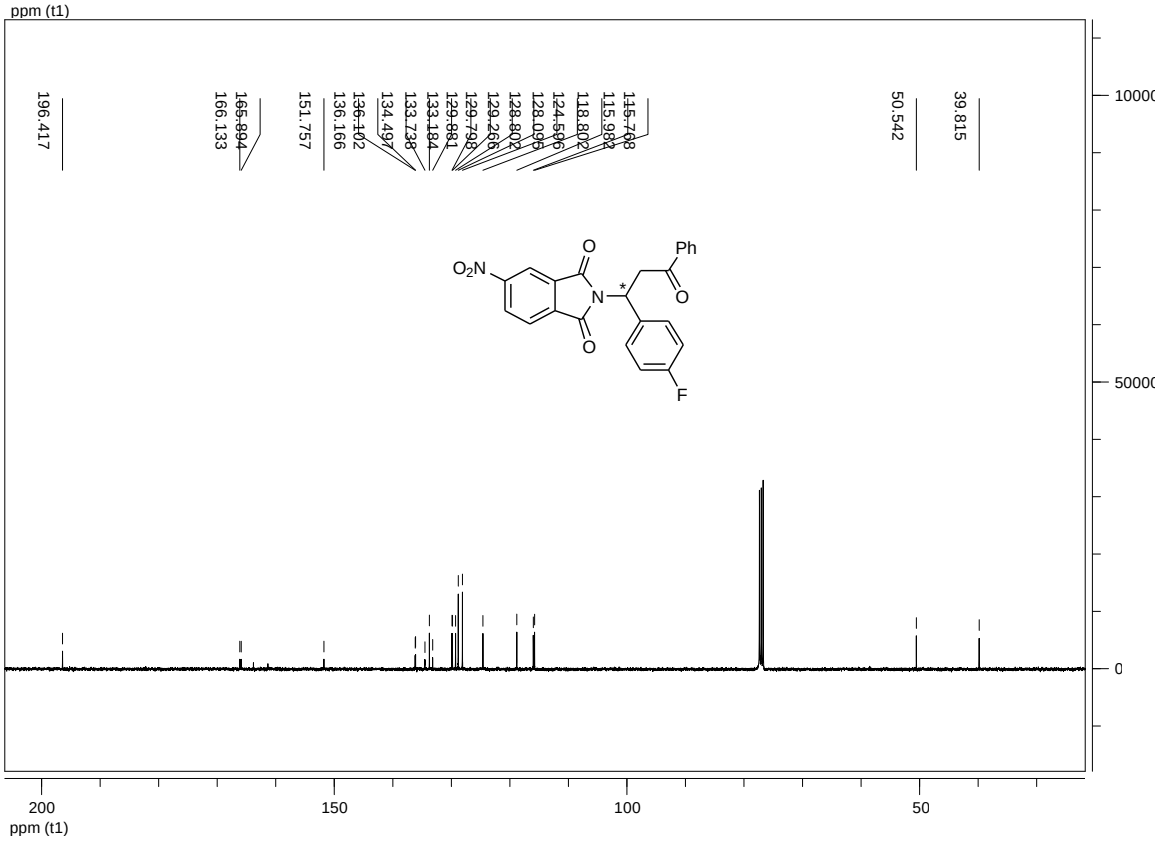


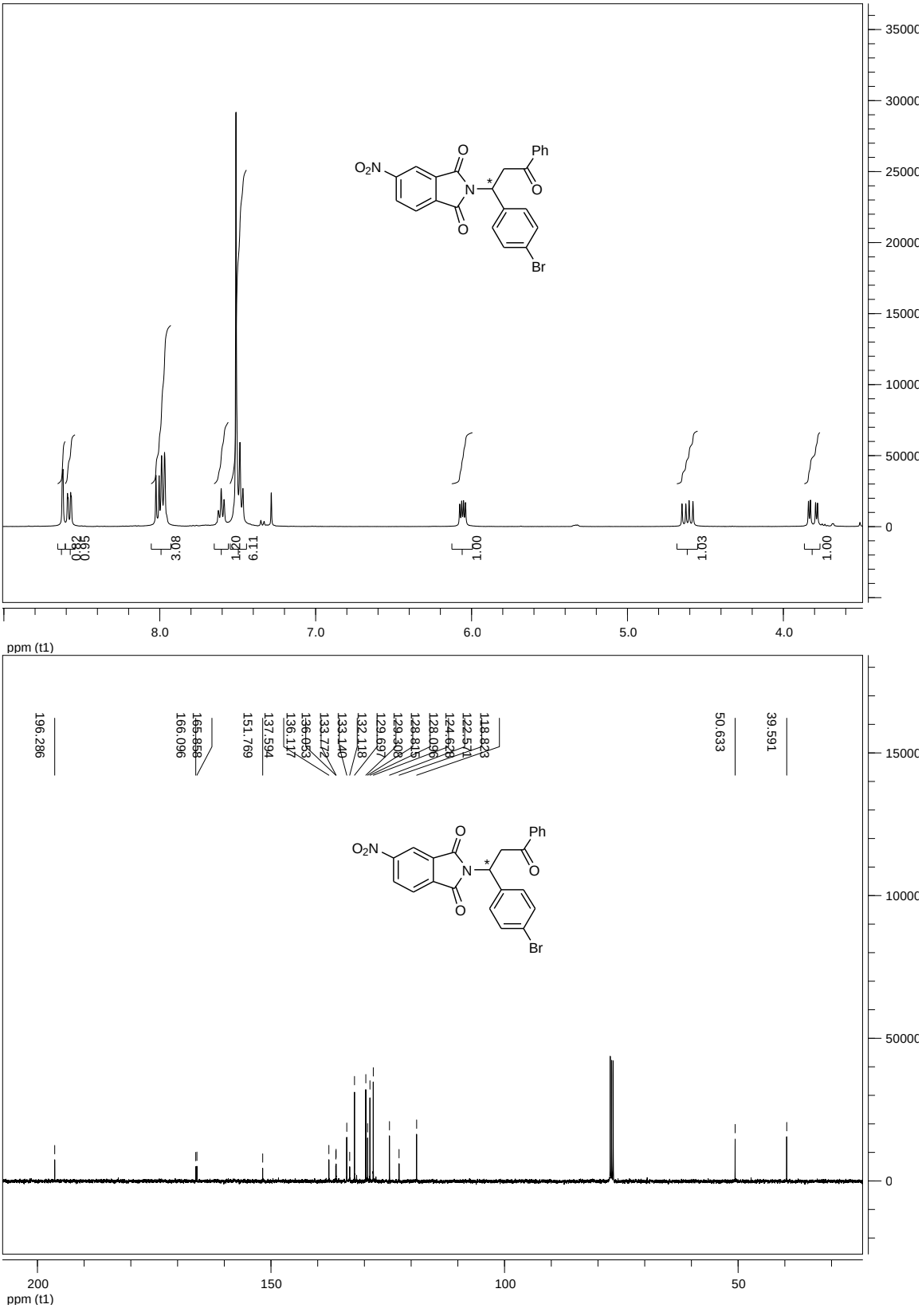


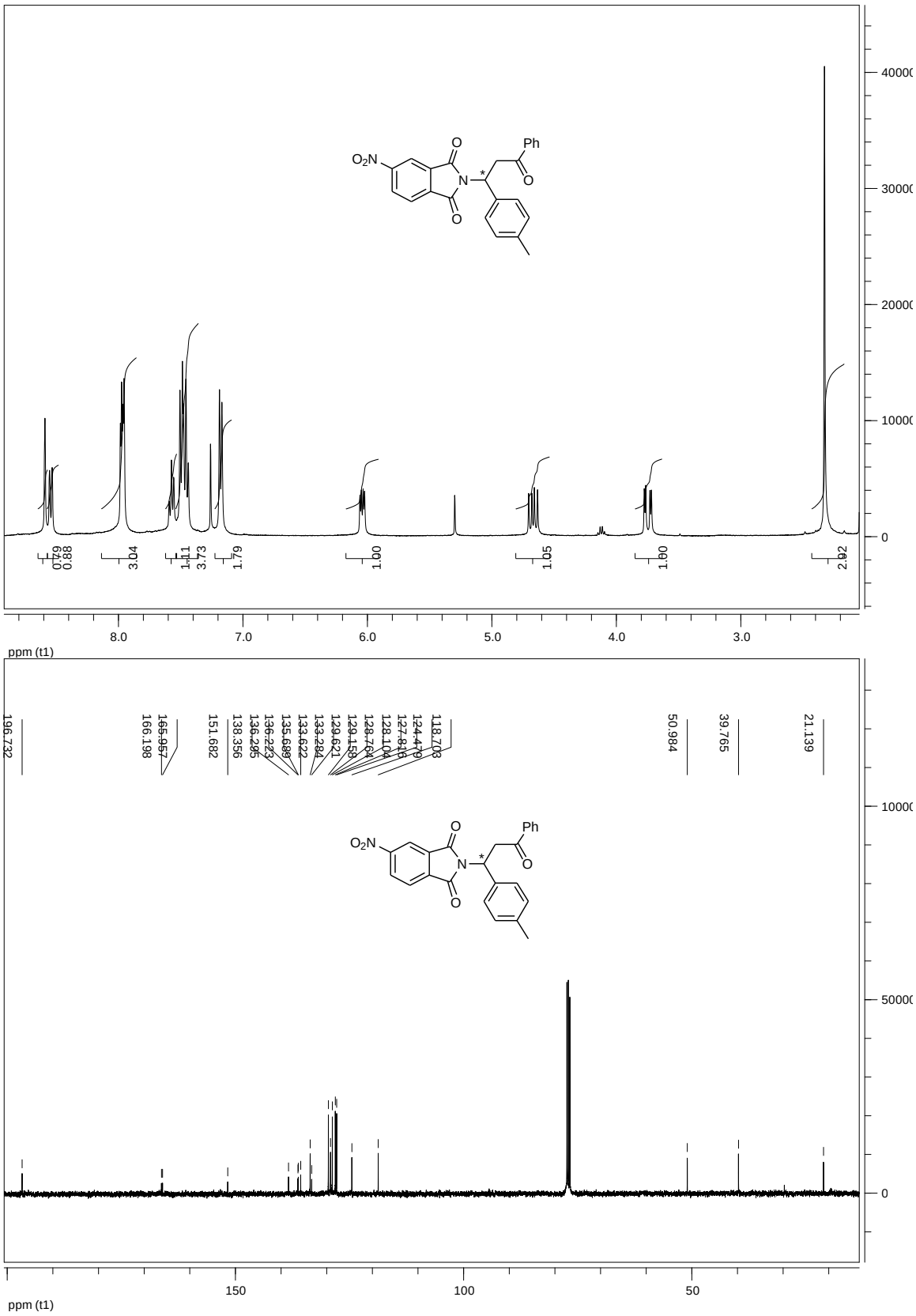


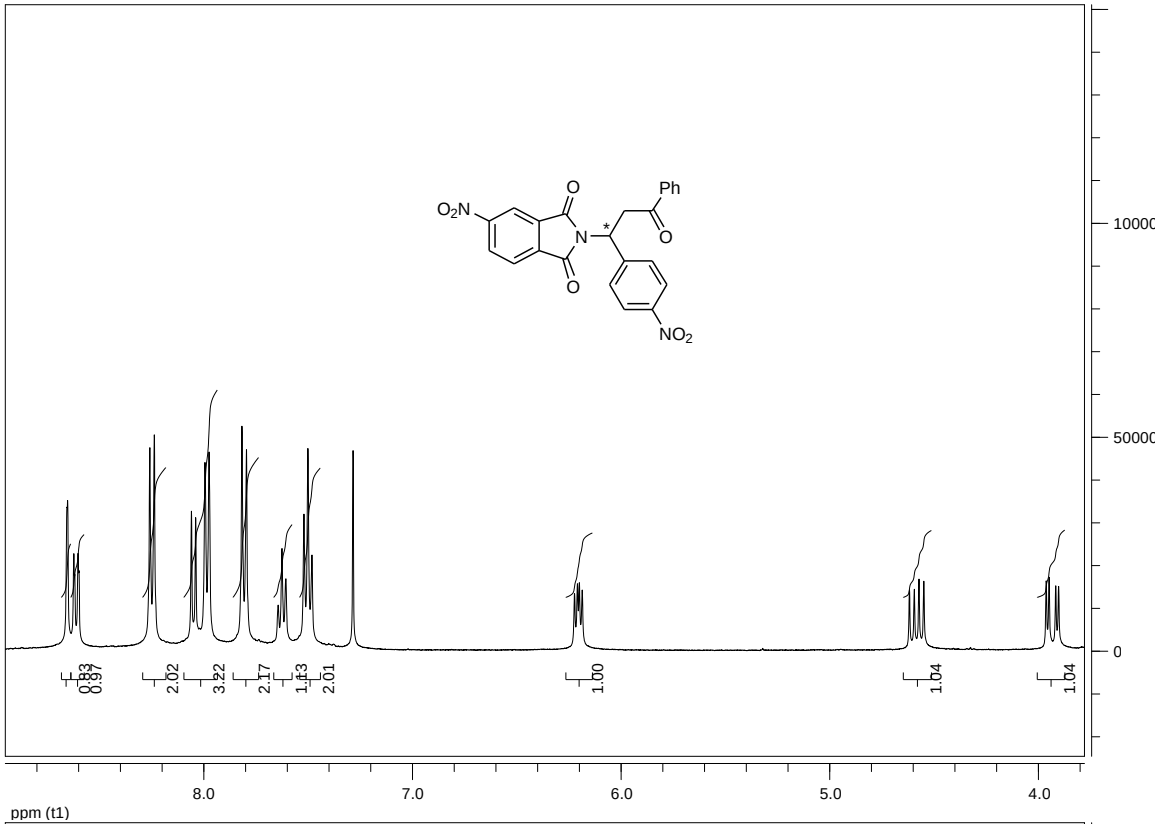








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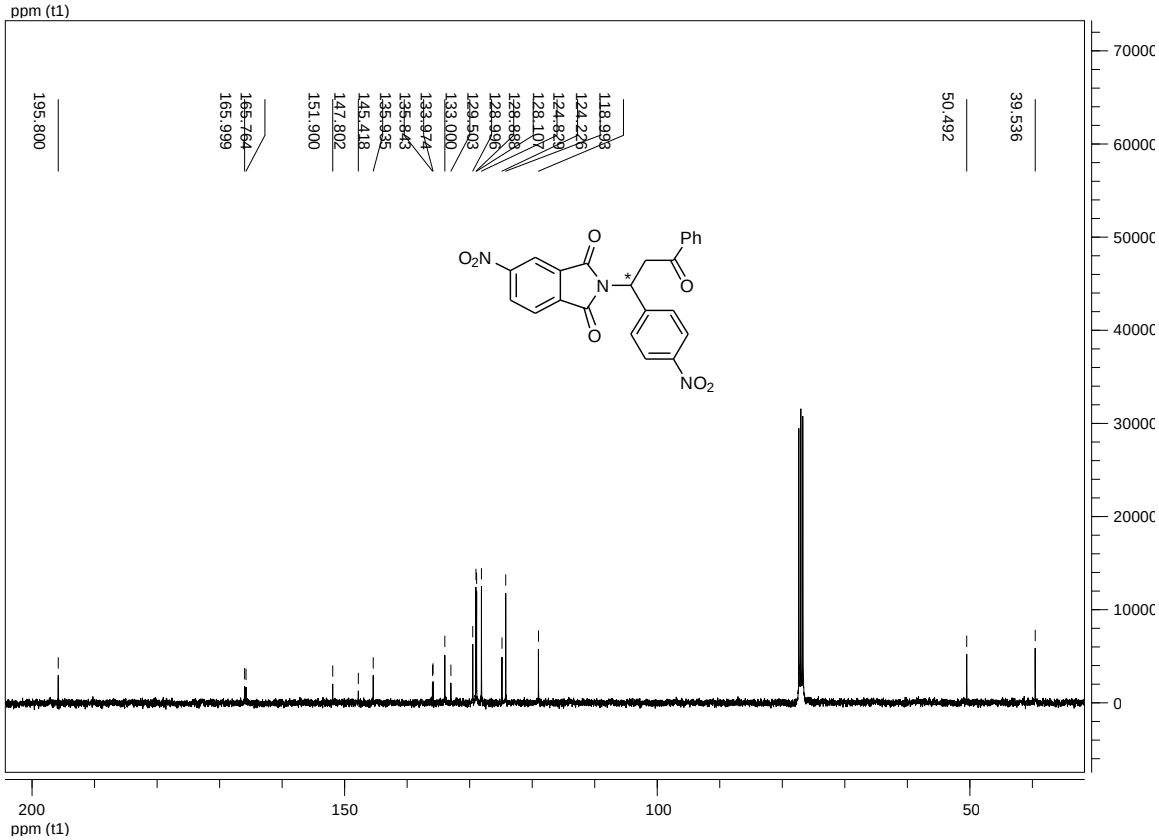
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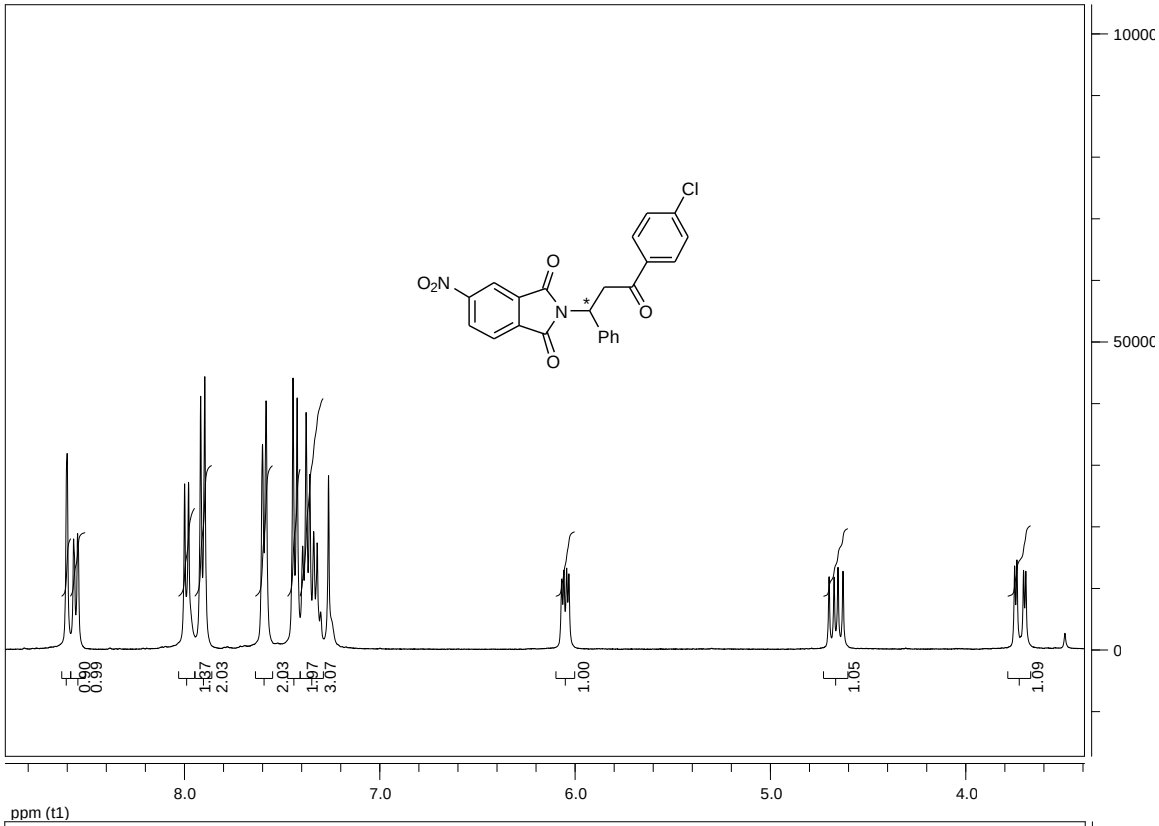
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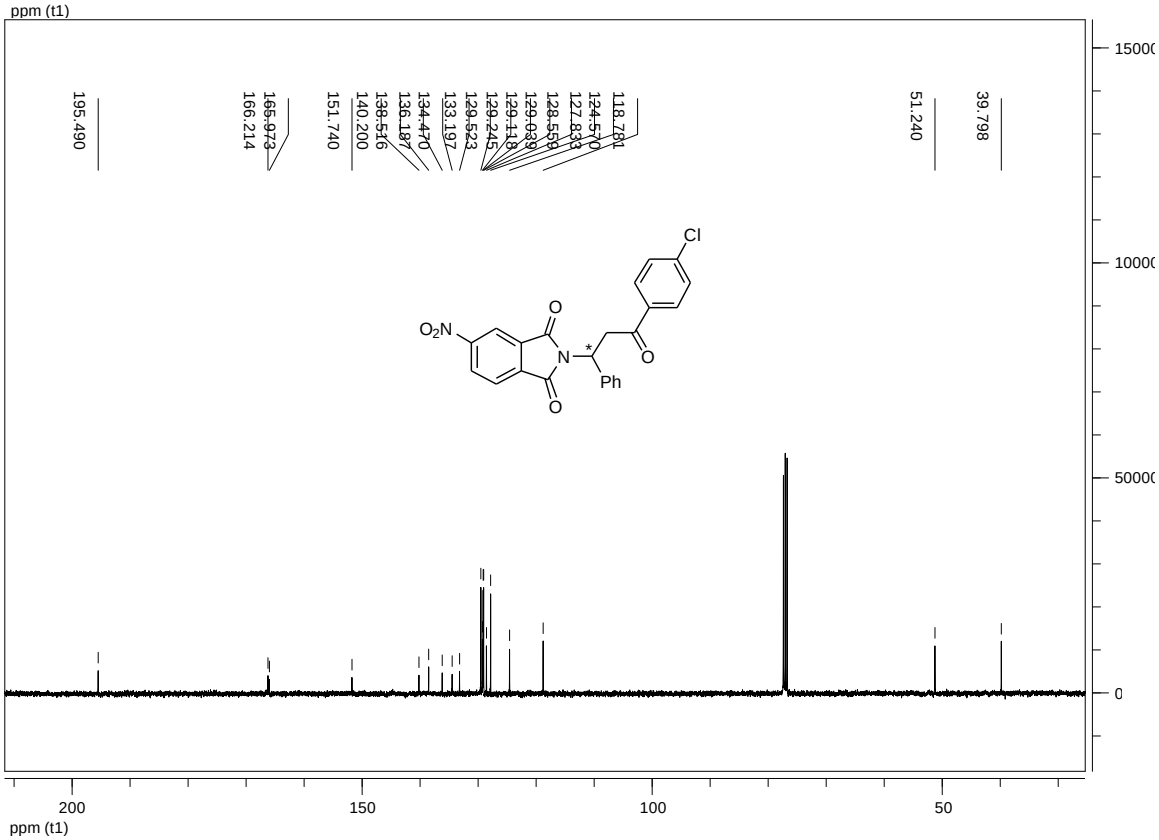
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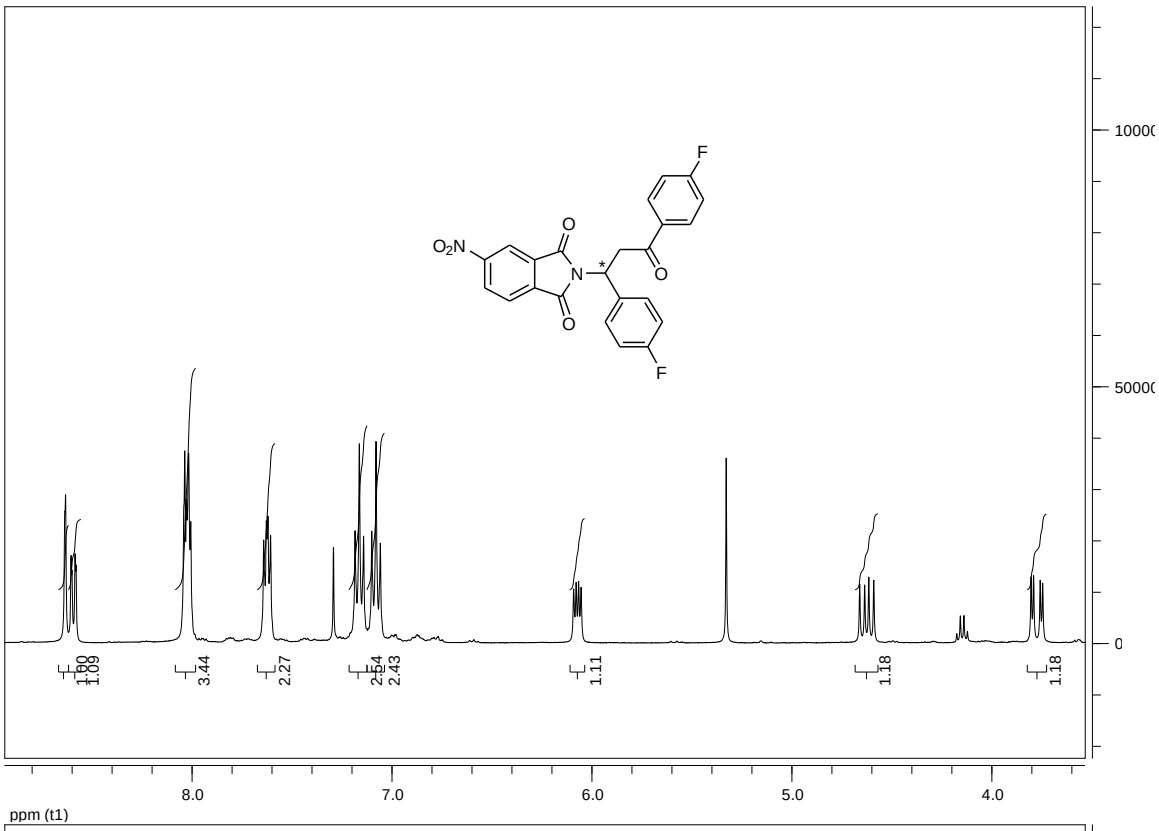
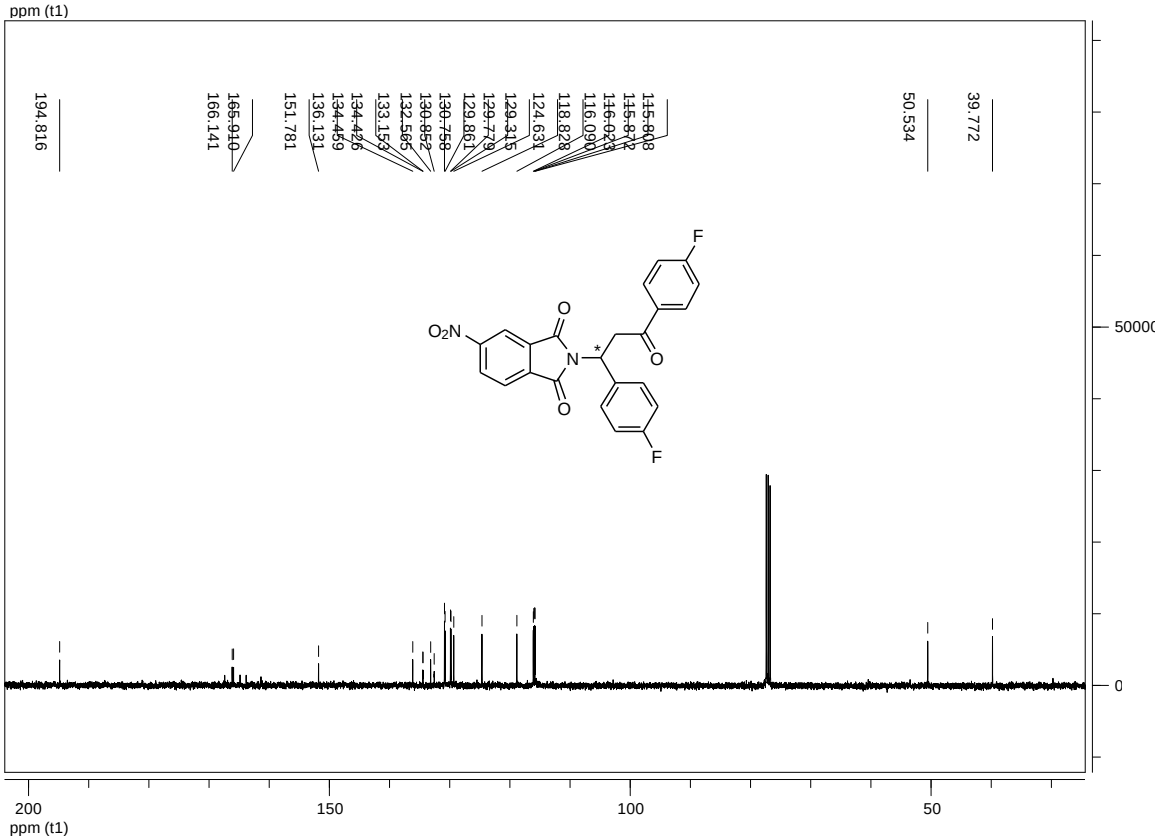
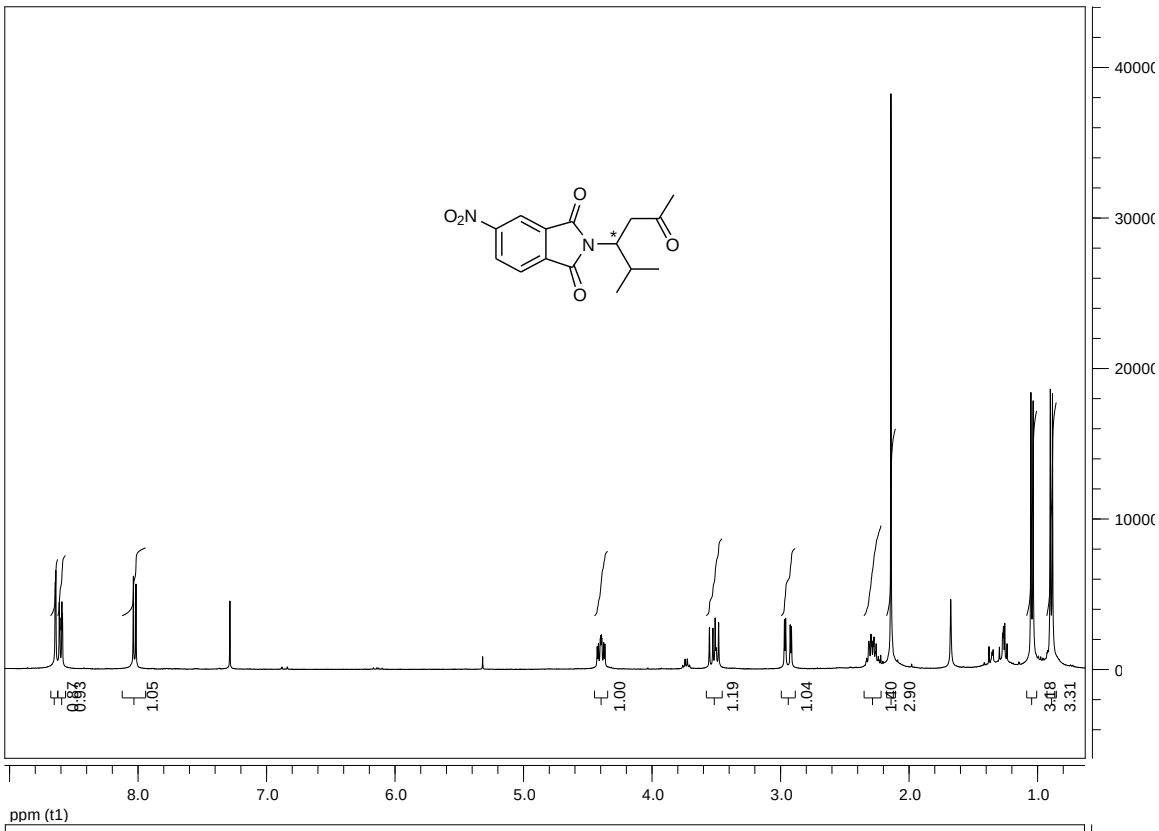
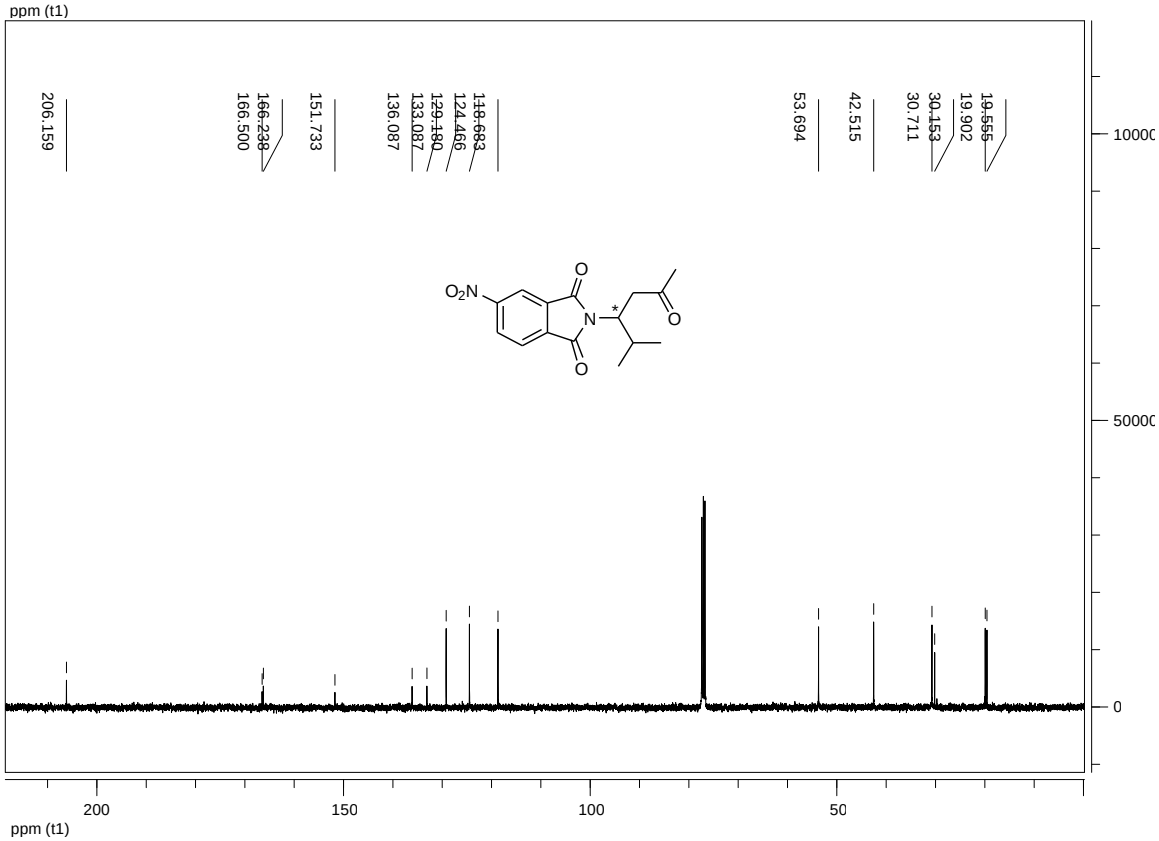
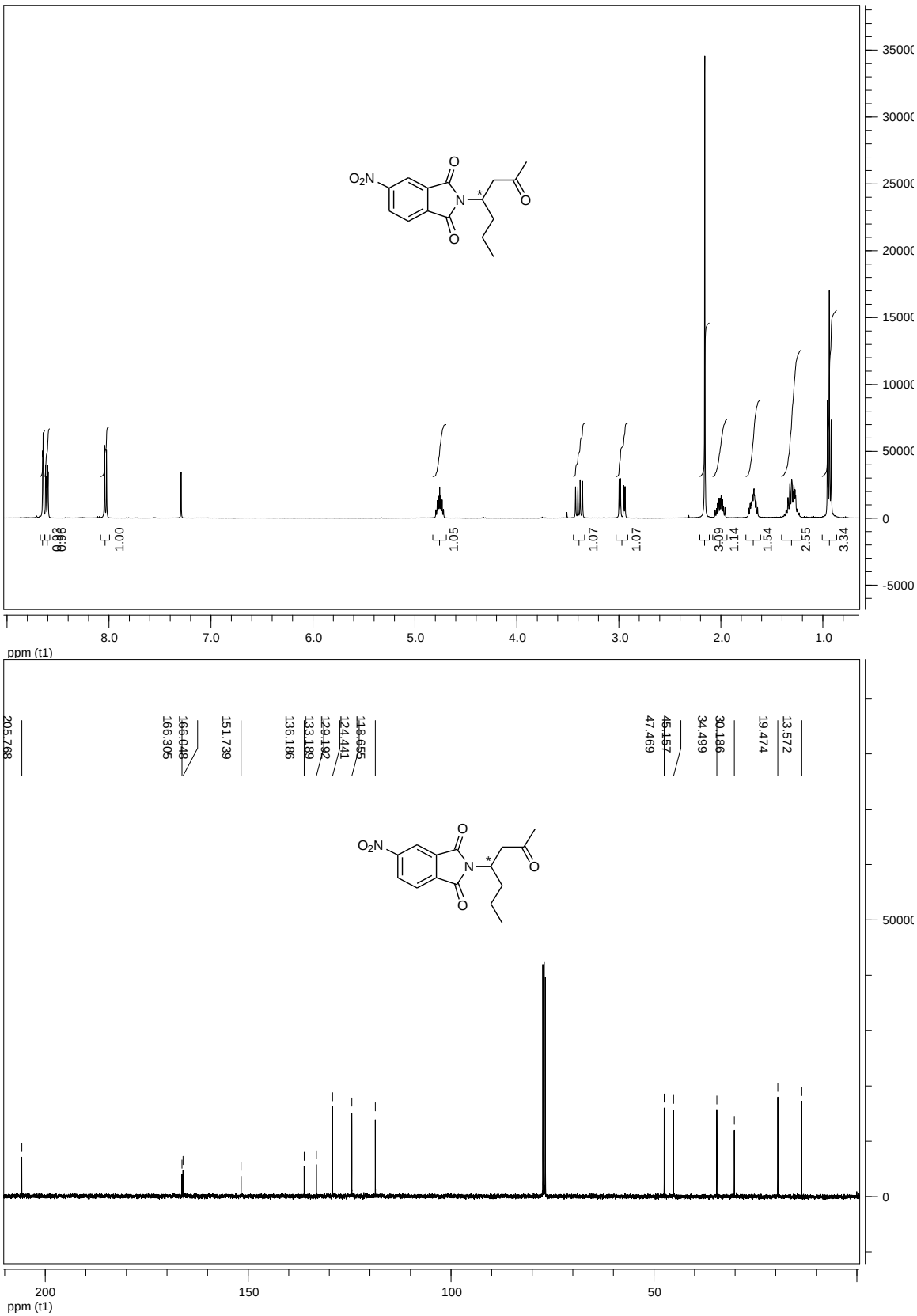
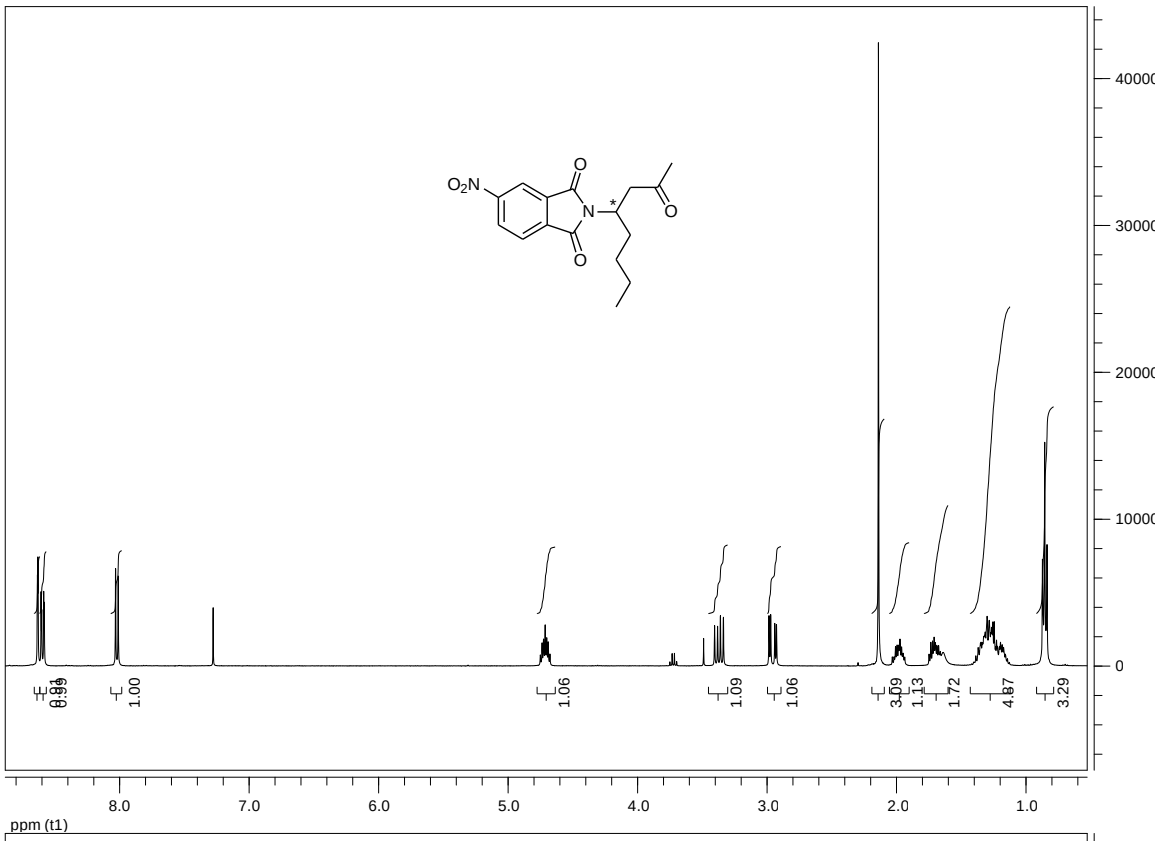


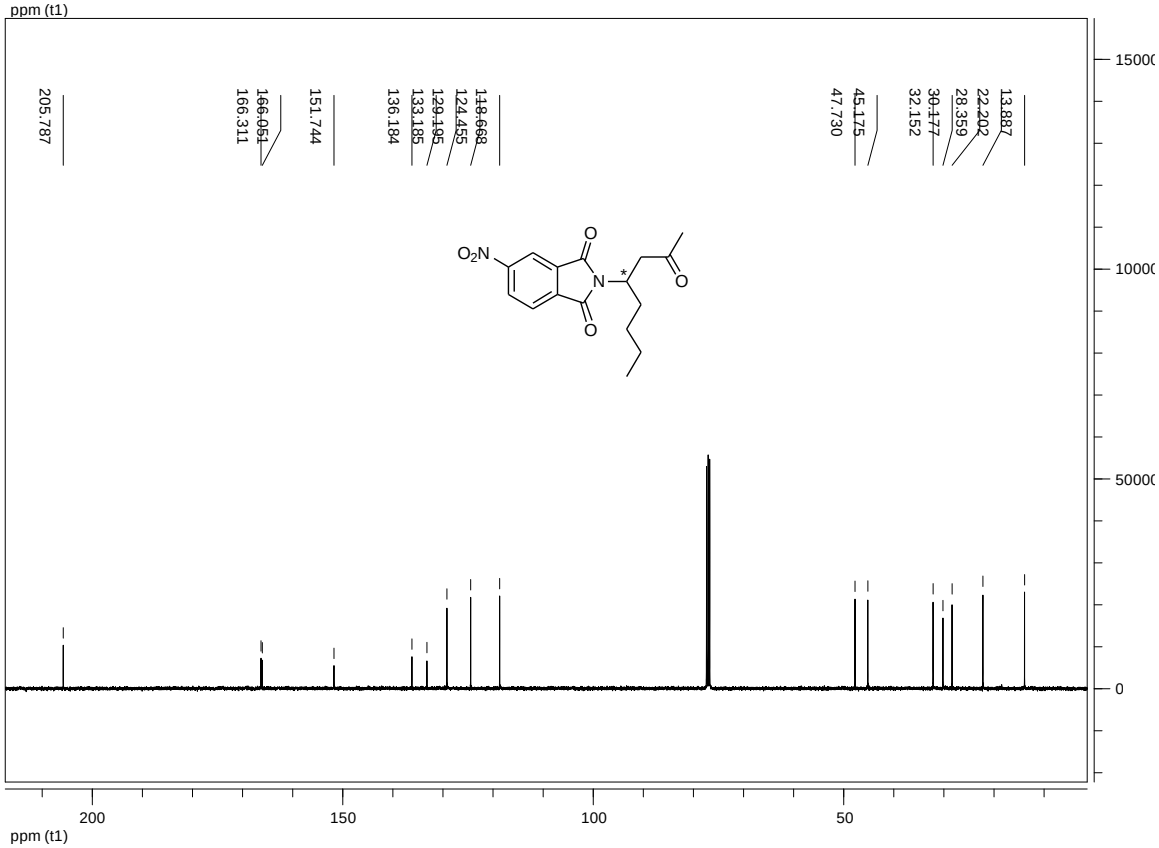


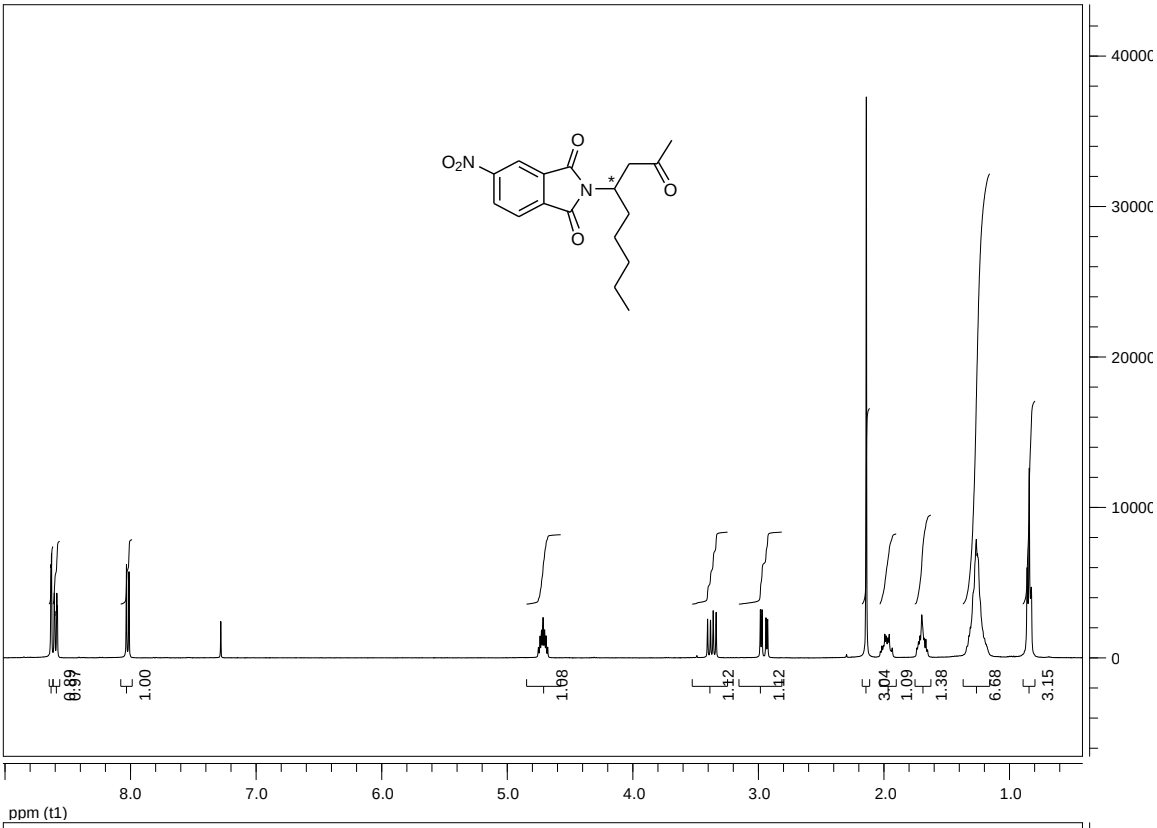


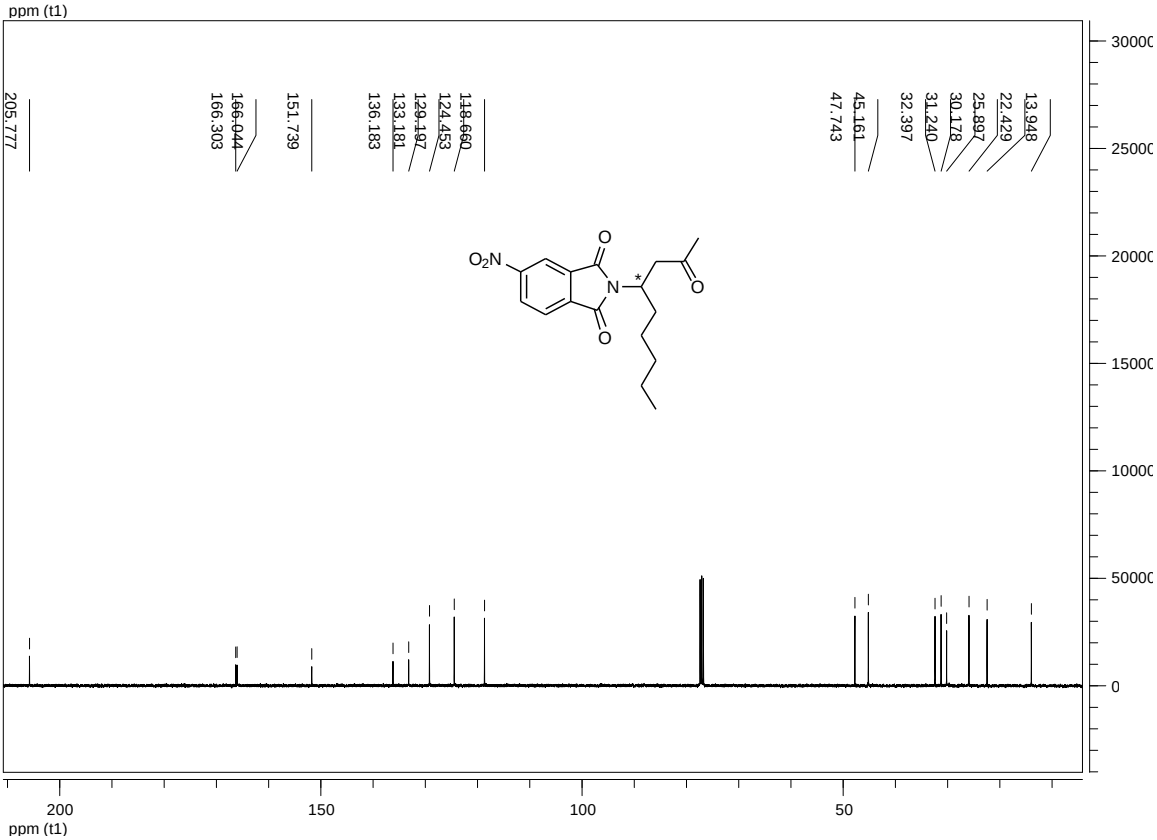


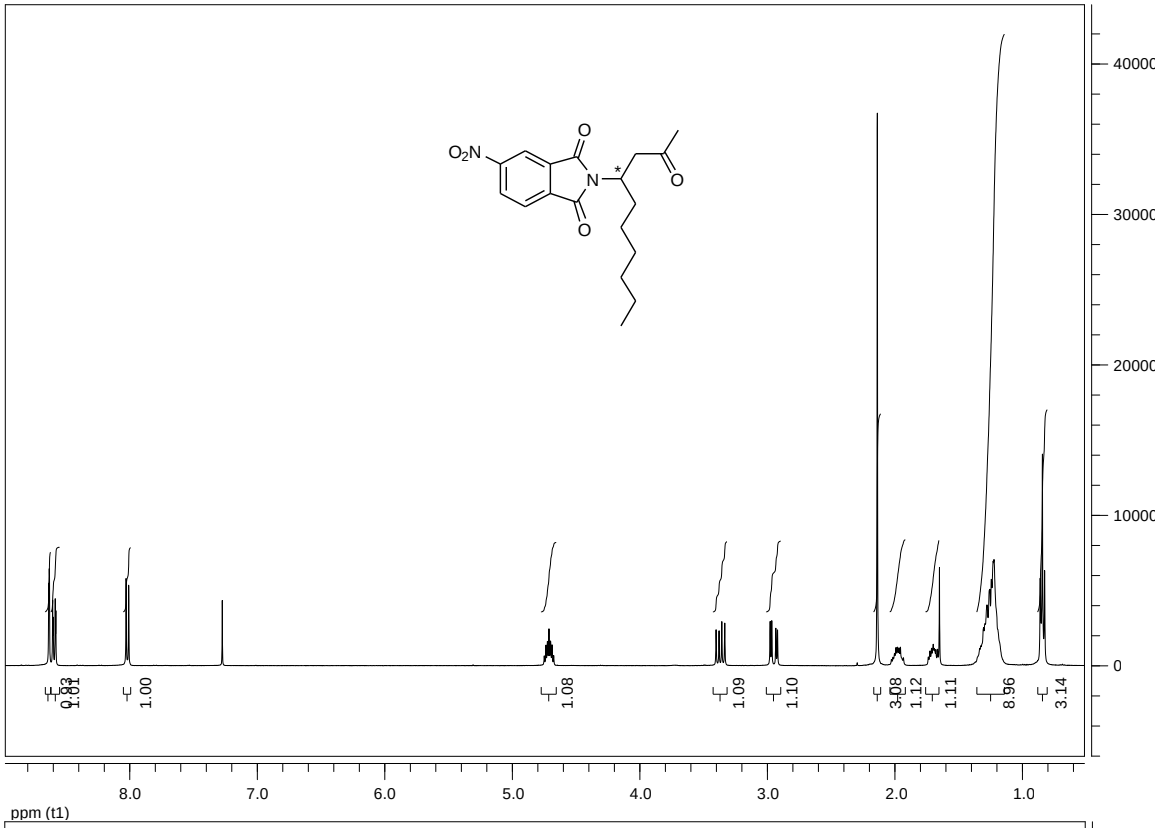


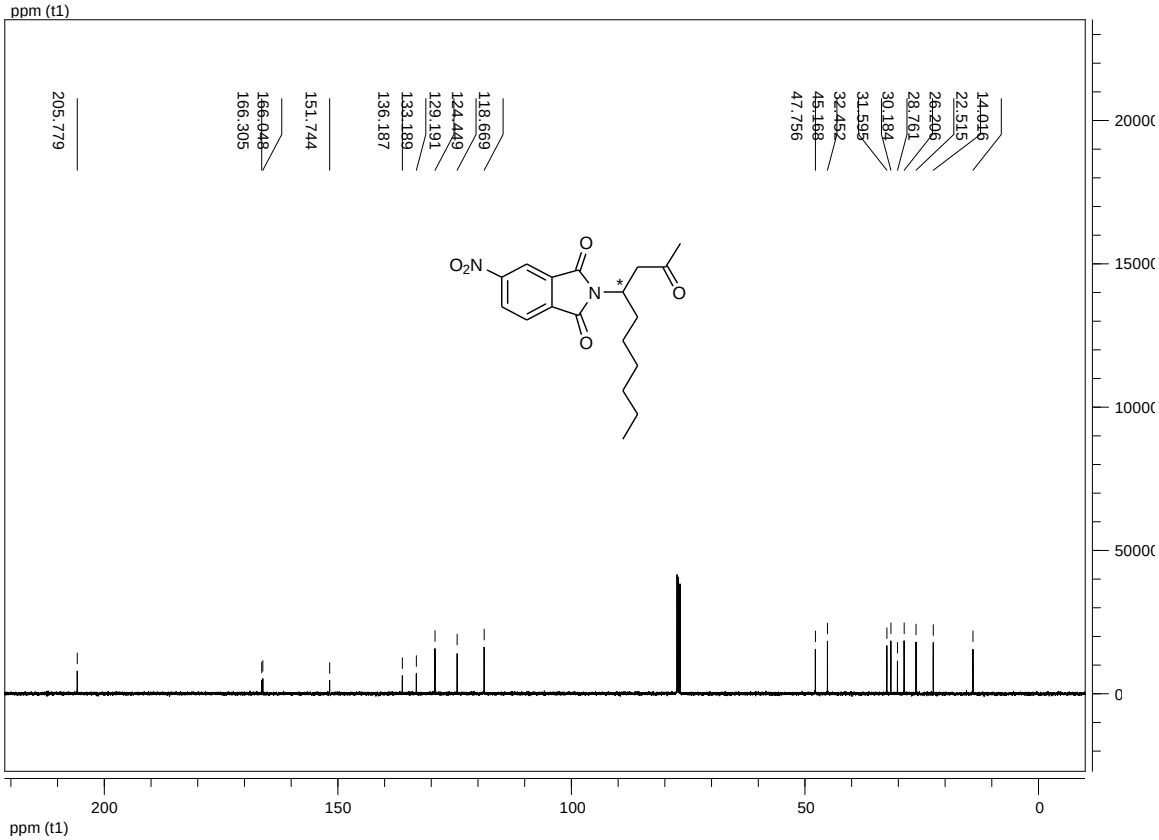


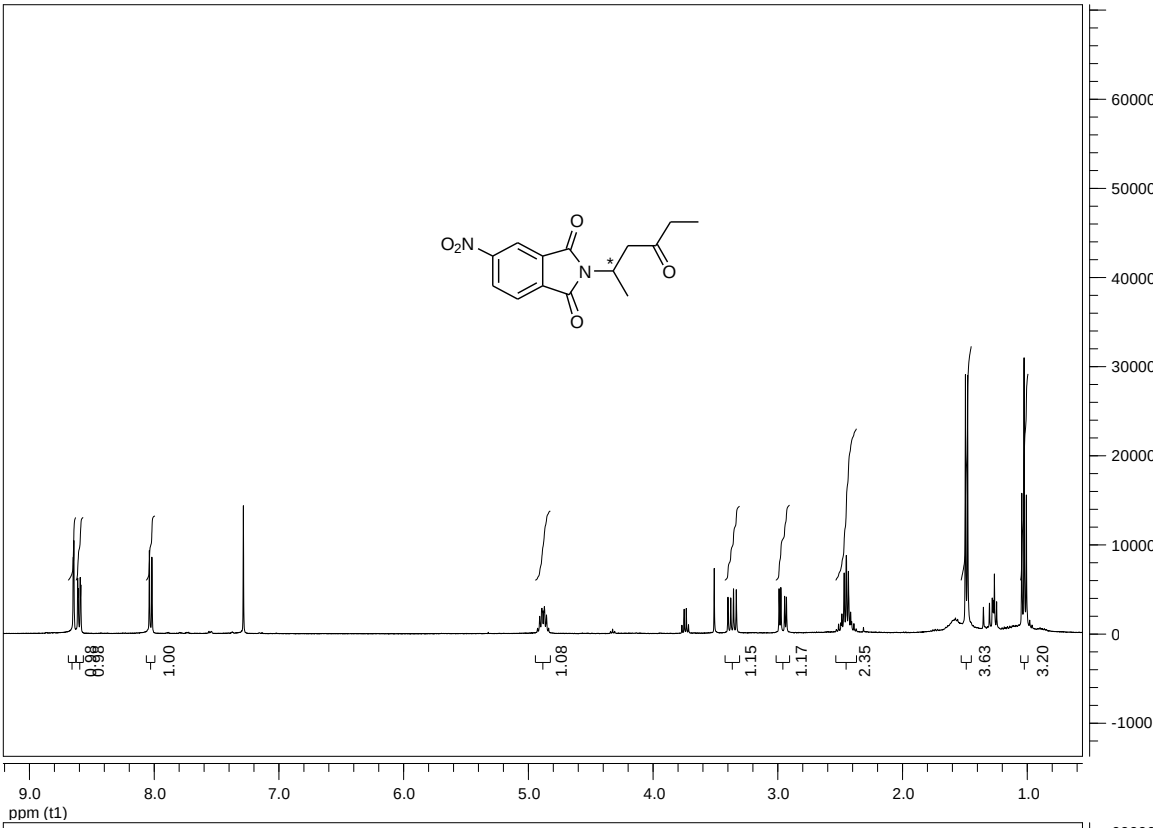


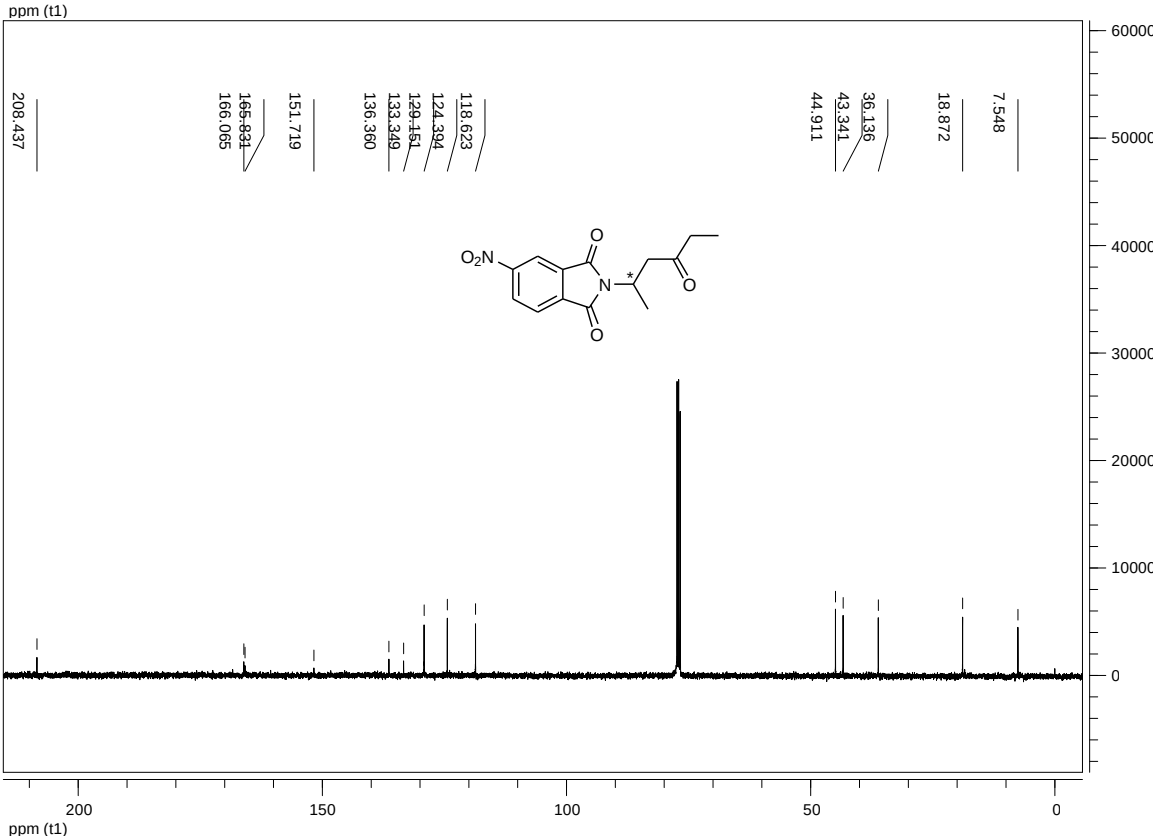

O2N
O
N
*
O
O
4000
3000
2000
1000
0
0.99
1.00
1.08
1.12
1.12
3.04
1.09
1.38
9.98
3.15
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (t1)


205.777
166.303
166.044
151.739
136.183
133.181
129.197
124.453
118.660
47.743
45.161
32.397
31.240
30.178
25.897
22.429
13.948
O2N
O
N
*
O
O
3000
2500
2000
1500
1000
5000
0
200
150
100
50
ppm (t1)



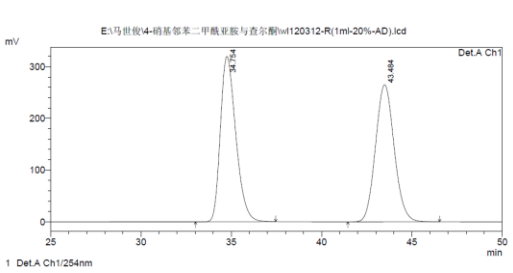






HPLC spectra of Michael adducts 3:

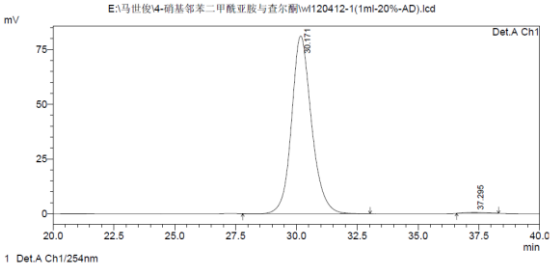
5-nitro-2-(3-oxo-1,3-diphenylpropyl)isoindoline-1,3-dione (3ba)



PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	34.754	18979841	319788	49.880	54.670
2	43.484	19070804	265149	50.120	45.330
Total		38050645	584937	100.000	100.000

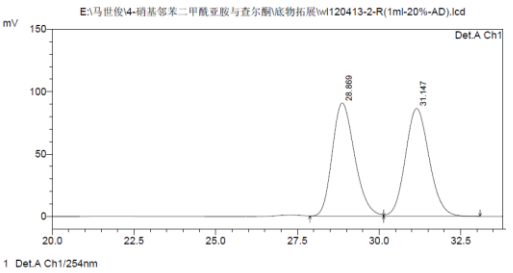


PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	30.171	4646409	81303	99.522	99.508
2	37.295	22314	402	0.478	0.492
Total		4668723	81705	100.000	100.000

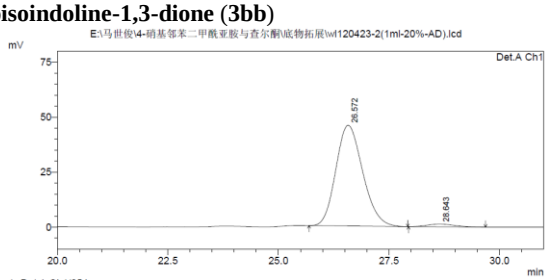
2-(1-(2-chlorophenyl)-3-oxo-3-phenylpropyl)-5-nitroisoindoline-1,3-dione (3bb)



PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	28.869	4323334	90593	49.855	51.248
2	31.147	4348528	86181	50.145	48.752
Total		8671862	176774	100.000	100.000

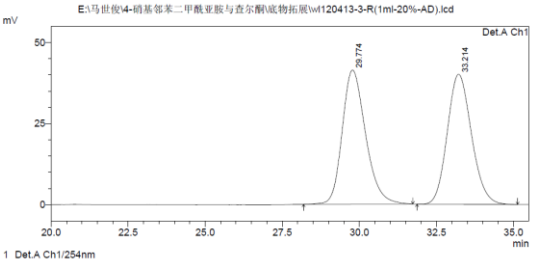


PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	26.572	1935464	45807	97.366	97.359
2	28.643	52356	1243	2.634	2.641
Total		1987820	47050	100.000	100.000

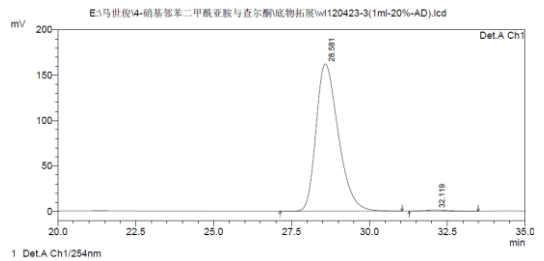
2-(1-(3-chlorophenyl)-3-oxo-3-phenylpropyl)-5-nitroisoindoline-1,3-dione (3bc)



PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	29.774	2182128	41344	49.905	50.777
2	33.214	2190435	40078	50.095	49.223
Total		4372563	81422	100.000	100.000

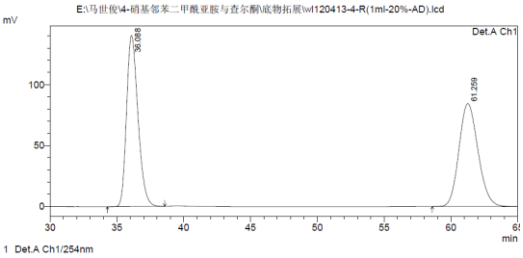


PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	28.581	8060237	162148	99.208	99.211
2	32.119	64513	1290	0.792	0.789
Total		8124550	163438	100.000	100.000

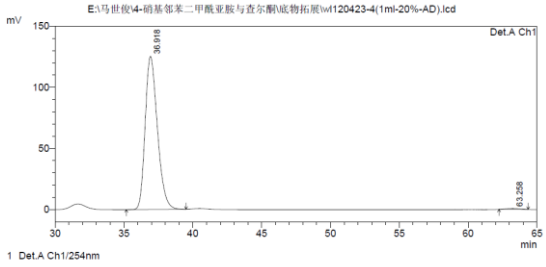
2-(1-(4-chlorophenyl)-3-oxo-3-phenylpropyl)-5-nitroisoindoline-1,3-dione (3bd).



PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	36.088	8361135	140433	49.951	62.428
2	61.259	8377632	84517	50.049	37.572
Total		16738767	224951	100.000	100.000

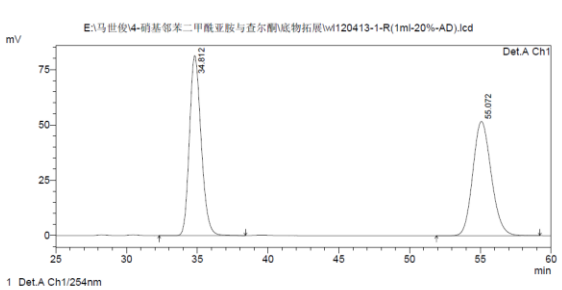


PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	36.918	7722030	125141	99.444	99.535
2	63.258	43143	585	0.556	0.465
Total		7765174	125726	100.000	100.000

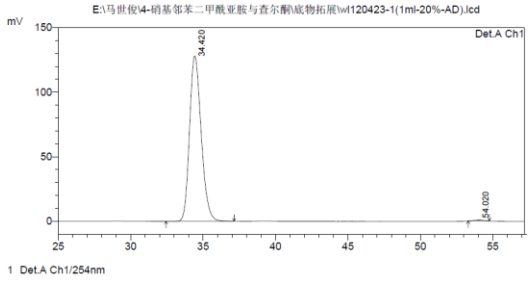
2-(1-(4-fluorophenyl)-3-oxo-3-phenylpropyl)-5-nitroisoindoline-1,3-dione (3be).



PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	34.812	4674552	81328	49.982	61.169
2	55.072	4677928	51628	50.018	38.831
Total		9352480	132956	100.000	100.000

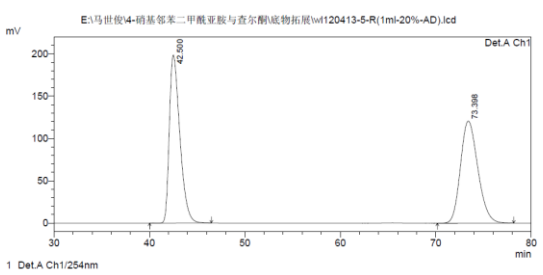


PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	34.420	7157848	128042	99.608	99.584
2	54.020	28143	535	0.392	0.416
Total		7185991	128578	100.000	100.000

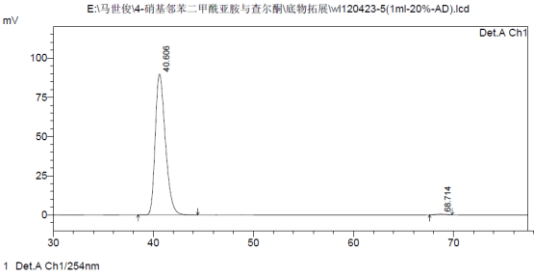
2-(1-(4-bromophenyl)-3-oxo-3-phenylpropyl)-5-nitroisoindoline-1,3-dione (3bf)



PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	42.500	15211776	198589	49.900	62.185
2	73.398	15272608	120763	50.100	37.815
Total		30484383	319353	100.000	100.000

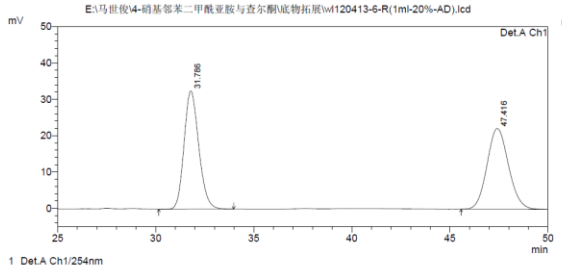


PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	40.606	6305492	89702	99.453	99.512
2	68.714	34667	439	0.547	0.488
Total		6340159	90141	100.000	100.000

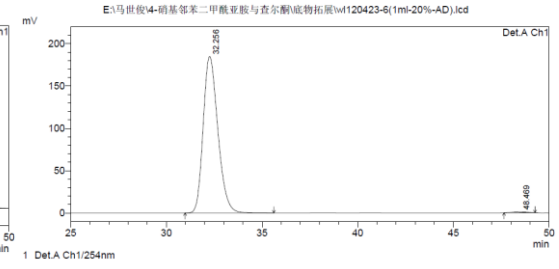
5-nitro-2-(3-oxo-3-phenyl-1-p-tolylpropyl)isoindoline-1,3-dione (3bg)



PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	31.786	1699783	32562	50.081	59.400
2	47.416	1694295	22257	49.919	40.600
Total		3394078	54819	100.000	100.000

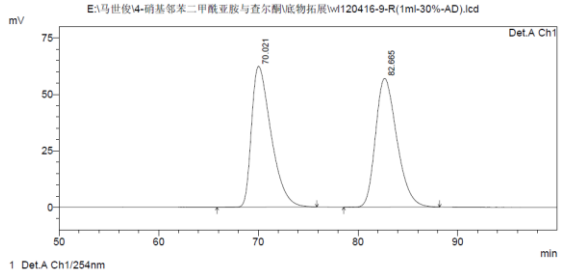


PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	32.256	9862350	184931	99.517	99.543
2	48.469	47913	849	0.483	0.457
Total		9910264	185781	100.000	100.000

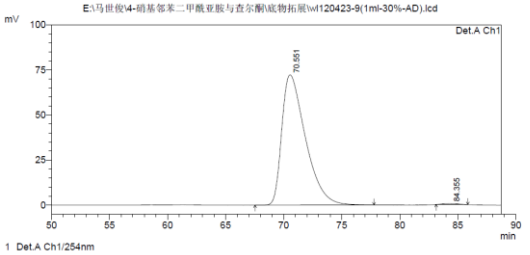
5-nitro-2-(1-(4-nitrophenyl)-3-oxo-3-phenylpropyl)isoindoline-1,3-dione (3bh)



PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	70.021	8517373	62401	50.011	52.236
2	82.665	8513756	57058	49.989	47.764
Total		17031129	119458	100.000	100.000

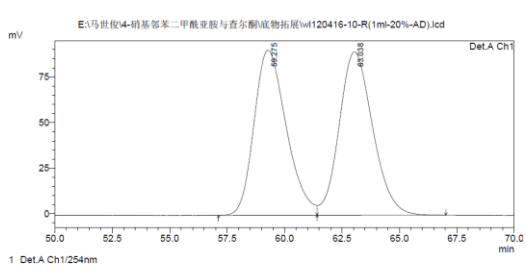


PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	70.651	10232722	72298	99.587	99.395
2	84.355	42423	440	0.413	0.605
Total		10275144	72739	100.000	100.000

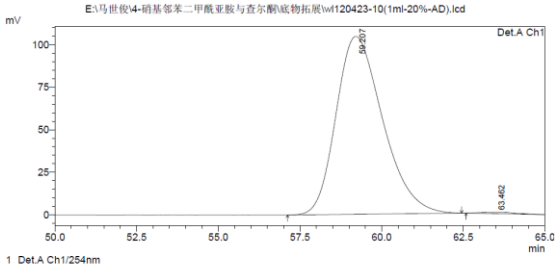
2-(3-(4-chlorophenyl)-3-oxo-1-phenylpropyl)-5-nitroisoindoline-1,3-dione (3bi)



PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	59.275	8818529	90455	49.282	50.268
2	65.038	9075385	89491	50.718	49.732
Total		17893913	179946	100.000	100.000

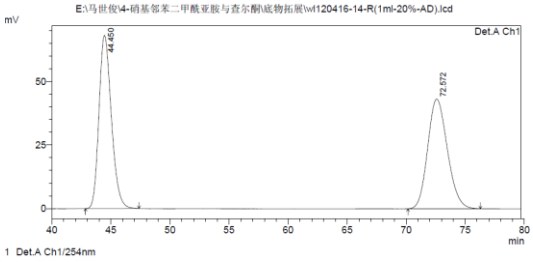


PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	59.207	10379441	104545	99.406	99.233
2	63.462	61993	808	0.594	0.767
Total		10441434	105353	100.000	100.000

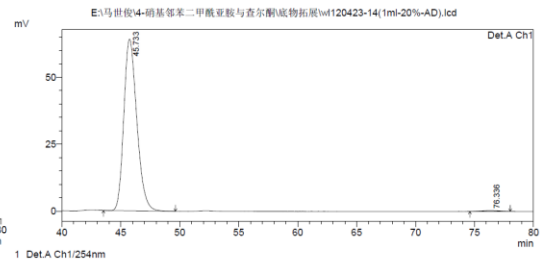
2-(1,3-bis(4-fluorophenyl)-3-oxopropyl)-5-nitroisoindoline-1,3-dione (3bj)



PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	44.450	4968649	68032	49.871	61.217
2	72.572	4994374	43100	50.129	38.783
Total		9963023	111132	100.000	100.000

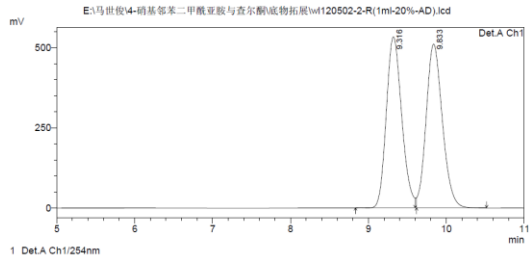


PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	45.733	4911579	64077	99.368	99.526
2	76.336	31253	305	0.632	0.474
Total		4942832	64383	100.000	100.000

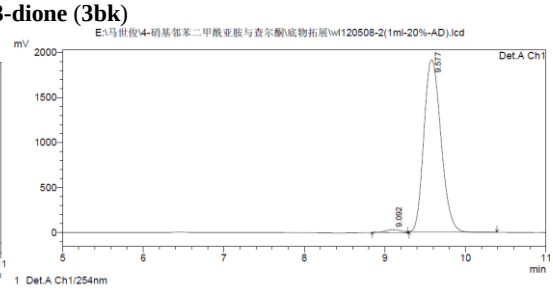
2-(2-methyl-5-oxohexan-3-yl)-5-nitroisoindoline-1,3-dione (3bk)



PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	9.316	7286136	532538	49.734	51.093
2	9.833	7364037	509758	50.266	48.907
Total		14650173	1042295	100.000	100.000

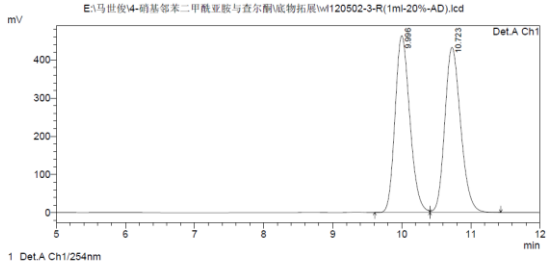


PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	9.092	434926	33944	1.523	1.742
2	9.577	28119452	1915120	98.477	98.258
Total		28554377	1949064	100.000	100.000

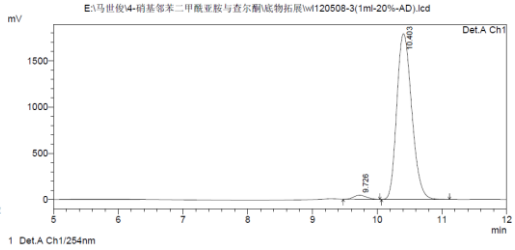
5-nitro-2-(2-oxoheptan-4-yl)isoindoline-1,3-dione (3bl)



PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	9.996	6850012	462984	49.906	51.719
2	10.723	6875905	432212	50.094	48.281
Total		13725916	895196	100.000	100.000

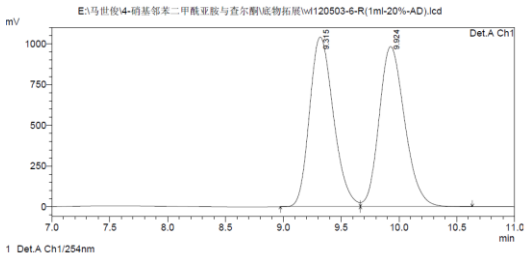


PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	9.726	614657	45022	2.091	2.451
2	10.403	28786342	1791994	97.909	97.549
Total		29400999	1837016	100.000	100.000

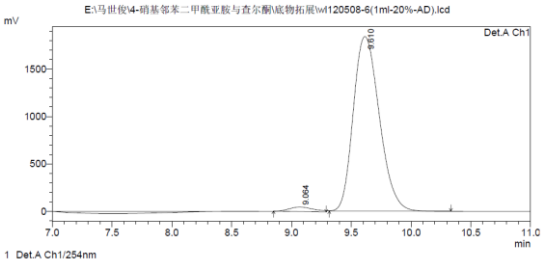
5-nitro-2-(2-oxooctan-4-yl)isoindoline-1,3-dione (3bm)



PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	9.315	14925441	1039963	49.803	51.449
2	9.924	15043525	981396	50.197	48.551
Total		29968966	2021359	100.000	100.000

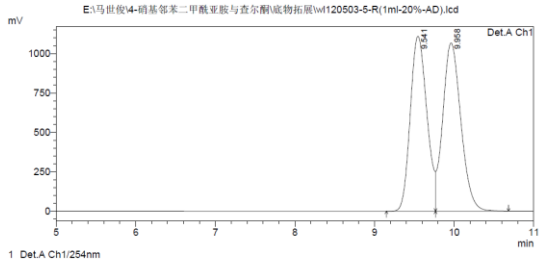


PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	9.064	673208	47868	2.363	2.537
2	9.610	27822017	1839124	97.637	97.463
Total		28495226	1886993	100.000	100.000

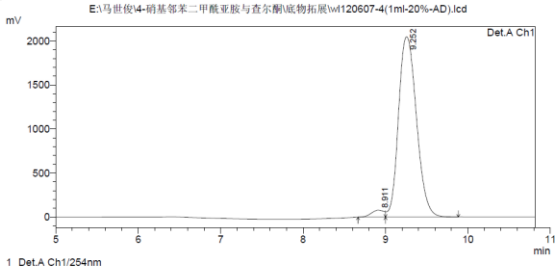
5-nitro-2-(2-oxononan-4-yl)isoindoline-1,3-dione (3bn)



PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	9.541	16128316	1110383	48.802	50.956
2	9.958	16920369	1068740	51.198	49.044
Total		33048686	2179123	100.000	100.000

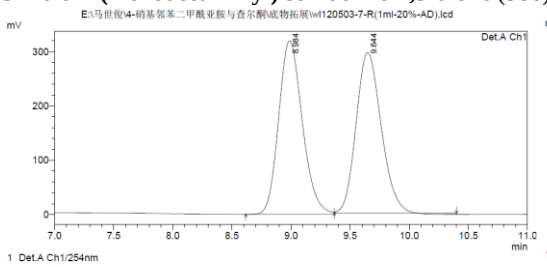


PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	8.911	842055	79041	2.642	3.715
2	9.252	31031479	2048603	97.358	96.285
Total		31873535	2127644	100.000	100.000

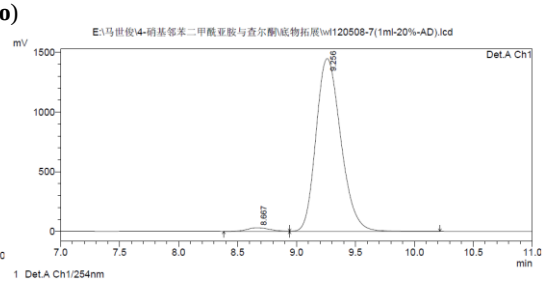
5-nitro-2-(2-oxodecan-4-yl)isoindoline-1,3-dione (3bo)



PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	8.984	4438252	319268	50.550	51.869
2	9.644	4341698	296255	49.450	48.131
Total		8779951	615523	100.000	100.000

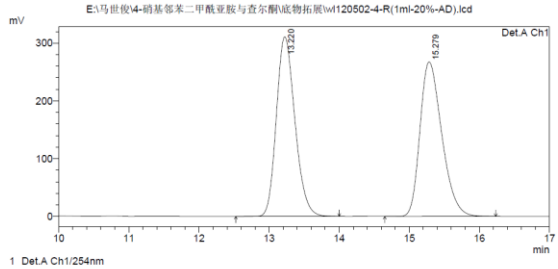


PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	8.667	401428	30500	1.859	2.061
2	9.256	21198051	1449225	98.141	97.939
Total		21599479	1479726	100.000	100.000

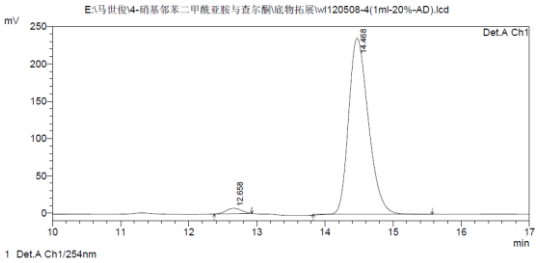
5-nitro-2-(4-oxohexan-2-yl)isoindoline-1,3-dione (3bp)



PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	13.220	5725554	311059	49.749	53.755
2	15.279	5783335	267597	50.251	46.245
Total		11508888	578655	100.000	100.000



PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	12.658	114007	7347	2.341	3.019
2	14.468	4754993	236003	97.659	96.981
Total		4869000	243350	100.000	100.000