Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Supporting Information

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1.General information

General procedures and methods

Experiments involving moisture and/or air sensitive components were performed under a positive pressure of nitrogen in oven-dried glassware equipped with a rubber septum inlet. Dried solvents and liquid reagents were transferred by oven-dried syringes or hypodermic syringe cooled to ambient temperature in a desiccator. Reactions mixtures were stirred in 10 mL sample vial with Teflon-coated magnetic stirring bars unless otherwise stated. Moisture in non-volatile reagents/compounds was removed in high *vacuo* by means of an oil pump and subsequent purging with nitrogen. Solvents were removed in vacuo under ~30 mmHg and heated with a water bath at 30−35 °C using Changcheng rotary evaporator with Changcheng aspirator. The condenser was cooled with running water at 0 °C.

All experiments were monitored by analytical thin layer chromatography (TLC). TLC was performed on pre-coated plates. After elution, plate was visualized under UV illumination at 254 nm for UV active material. Further visualization was achieved by staining $KMnO_4$, ceric molybdate, or anisaldehyde solution. For those using the aqueous stains, the TLC plates were heated on a hot plate.

Columns for flash chromatography (FC) contained silica gel 200−300 mesh. Columns were packed as slurry of silica gel in petroleum ether and equilibrated solution using the appropriate solvent system. The elution was assisted by applying pressure of about 2 atm with an air pump.

Instrumentations

Proton nuclear magnetic resonance (^{1}H NMR) and carbon NMR (^{13}C NMR) were recorded in $CDCl_3$ otherwise stated. ^{1}H (300 MHz) and ^{13}C (75 MHz) were performed on Bruker AVANCE-III (300 MHz) spectrometers. Chemical shifts are reported in parts per million (ppm), using the residual solvent signal as an internal standard: $CDCl_3$ (^{1}H NMR: δ 7.26, singlet; ^{13}C NMR: δ 77.0, triplet). Multiplicities were given as: *s* (singlet), *d* (doublet), *t* (triplet), *q* (quartet), *quintet*, *m* (multiplets), *dd* (doublet of doublets), *dt* (doublet of triplets), and *br* (broad). Coupling constants (*J*) were recorded in Hertz (Hz). The number of proton atoms (*n*) for a given resonance was indicated by *n*H. The number of carbon atoms (*n*) for a given resonance was indicated by *n*C. HRMS was reported in units of mass of charge ratio

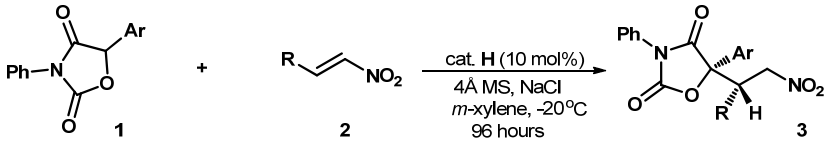
(m/z). Optical rotations were recorded on a polarimeter with a sodium lamp of wavelength 589 nm and reported as follows; $[\alpha]_{\lambda}^{T^{\circ}C}$ (c = g/100 mL, solvent). Melting points were determined on a melting point apparatus.

Enantiomeric excesses were determined by chiral High Performance Liquid Chromatography (HPLC) analysis. UV detection was monitored at 254 nm. HPLC samples were dissolved in HPLC grade isopropanol (IPA) unless otherwise stated.

Materials

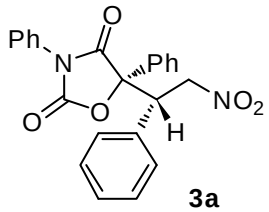
All commercial reagents were purchased with the highest purity grade. They were used without further purification unless specified. All solvents used, mainly petroleum ether (PE) and ethyl acetate (EtOAc) were distilled. Anhydrous DCM and MeCN were freshly distilled from CaH_2 and stored under N_2 atmosphere. THF, Et_2O, *m-xylene* and toluene were freshly distilled from sodium/benzophenone before use. Anhydrous methanol and etanol were distilled from Mg. All compounds synthesized were stored in a −20 °C freezer and light-sensitive compounds were protected with aluminium foil.

2. **General procedure for the asymmetric addition of 1 to 2 catalyzed by H**



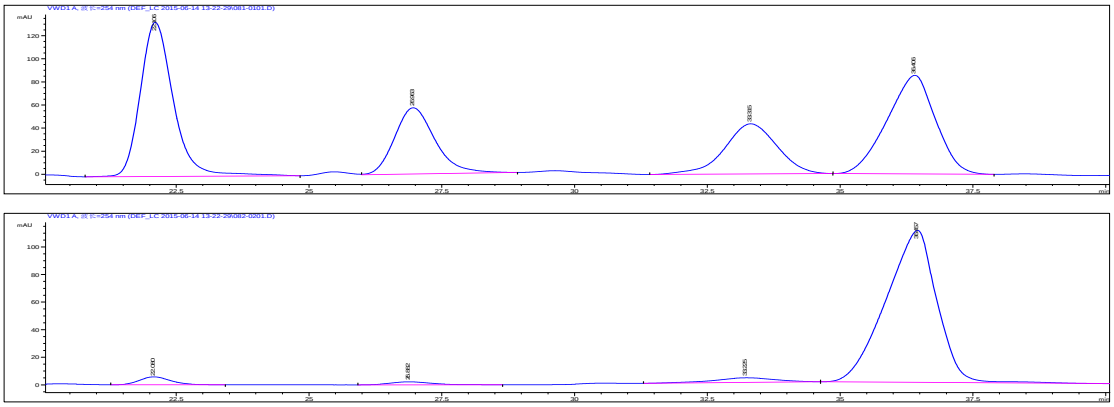
Diphenyloxazolidine-2,4-dione **1** (0.1 mmol, 1.0 equiv), nitroolefin **2** (0.12 mmol, 1.2 equiv), **H** (0.01 mmol, 0.1 equiv), NaCl (0.1 equiv) and 4Å molecular sieves (50 mg) were dissolved in *m*-xylene (1.0 mL). The reaction mixture was stirred at −20 °C for 72−96 hours and monitored by TLC. Upon complete consumption of **1**, the reaction mixture was concentrated under reduced pressure, The recovered crude material was subsequently purified by flash column chromatography on *silica gel* with PE/EtOAc mixture (20:1−5:1 ratio, the crude material was completely dissolved in CH_2Cl_2/PE before loaded on *silica gel*). After removing the solvent in *vacuo*, adducts **3** were obtained.

3. Characterization of adducts

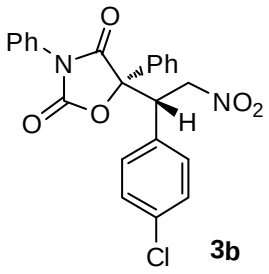


3a

White solid; Mp 152.3–153.1 °C; 94% ee; dr = 19:1; 98% yield; $[\alpha]_D^{22}$ +22.8 (*c* 1.00, $CHCl_3$); ^1H NMR (300 MHz, $CDCl_3$) δ 7.78 (d, *J* = 6.8, 2H), 7.52–7.33 (m, 8H), 7.27–7.12 (m, 3H), 6.59–6.44 (m, 2H), 4.91 (dd, *J* = 13.4, 11.4, 1H), 4.49 (dd, *J* = 11.4, 4.0, 1H), 4.35 (dd, *J* = 13.5, 4.0, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.3, 152.5, 132.3, 131.6, 130.1, 129.8, 129.6 (two peaks), 129.4 (three peaks), 129.1, 125.7, 124.9, 87.9, 74.1, 51.3; HRMS (ESI) m/z 403.1297 ($M+H^+$), Calcd for $C_{23}H_{19}N_2O_5$ 403.1294; The ee was determined by HPLC analysis. CHIRALPAK IA (4.6 mm i.d. x 250 mm) + CHIRALPAK IA (4.6 mm i.d. x 250 mm); Hexane/2-propanol = 90/10; flow rate 1.0 mL/min; 25 °C; 254 nm; t_R = 22.1 min (minor peak), 26.9 min, 33.2 min, 36.4 min (major peak).



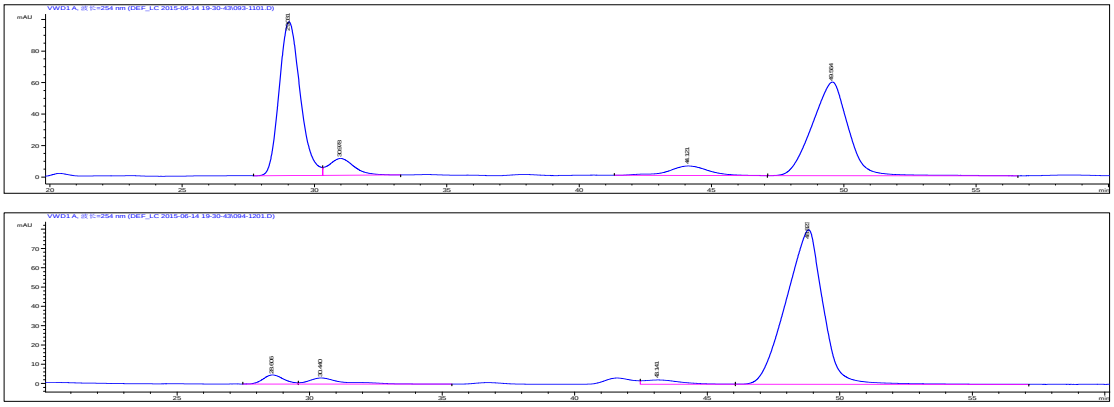
Entry	Retention Time	Area	Height	%Area
1	22.08	231	5.7	2.893
2	26.882	111.9	2.1	1.401
3	33.225	270.4	3.3	3.387
4	36.457	7371.9	110.3	92.319



3b

White solid; Mp 123.2–124.1 °C; 94% ee; dr = 19:1; 92% yield; $[\alpha]_D^{22}$ +20.5 (*c* 1.00, $CHCl_3$); ^1H NMR (300 MHz, $CDCl_3$) δ 7.85 (dd, *J* = 8.0, 1.5, 2H), 7.61–7.50 (m, 3H), 7.50–7.39 (m, 4H), 7.36–7.30 (m, 3H), 6.77–6.45 (m, 2H), 4.94 (dd, *J* = 13.5, 11.5, 1H), 4.56 (dd, *J* = 11.4, 4.0, 1H), 4.42 (dd, *J* = 13.6, 4.0, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.2, 152.4, 135.9, 132.1, 130.7, 130.3,130.2, 129.7 (two peaks), 129.6, 129.4, 125.5, 124.8, 87.7, 74.0, 50.8; HRMS (ESI) m/z 437.0902 ($M+H^+$), Calcd for $C_{23}H_{18}ClN_2O_5$ 437.0904; The ee was determined by HPLC analysis. CHIRALPAK IA (4.6 mm i.d. x 250

mm) + CHIRALPAK IA (4.6 mm i.d. x 250 mm); Hexane/2-propanol = 90/10; flow rate 1.0 mL/min; 25 °C; 254 nm; t_R = 28.6 min (minor peak), 30.4 min, 43.1 min, 48.8 min (major peak).



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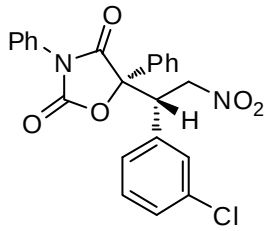
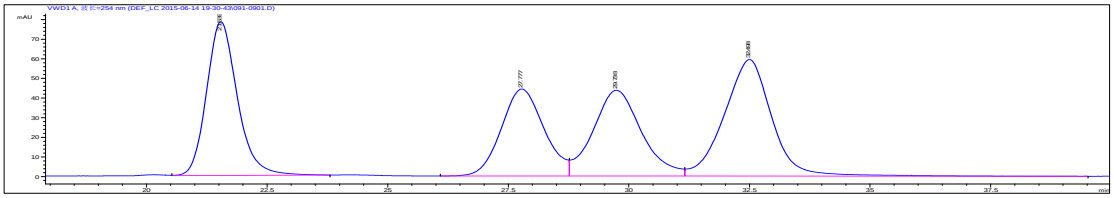
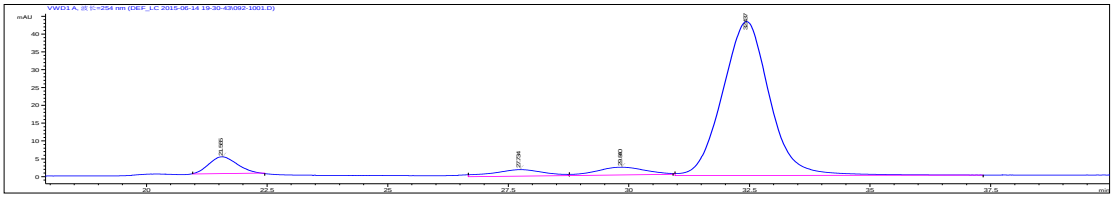
1	28.605	247.4	4.6	2.946
2	30.44	266.7	3.1	3.177
3	43.141	206.7	2.2	2.462
4	48.823	7676.2	80	91.415



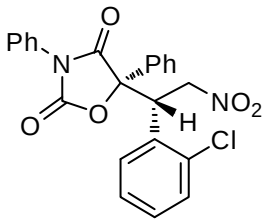
3c

White solid; Mp 135.8–136.5 °C; 95% ee; dr = 10:1; 96% yield; $[\alpha]_D^{22}$ +21.3 (*c* 1.00, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.85 (dd, *J* = 7.9, 1.4, 2H), 7.65–7.31 (m, 10H), 6.73 (dd, *J* = 6.4, 3.2, 2H), 4.94 (dd, *J* = 13.6, 11.4, 1H), 4.65–4.31 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.1, 152.3, 135.4, 133.8, 132.0, 129.9, 129.8, 129.7, 129.5 (two peaks), 129.4, 127.1, 125.5, 124.9, 87.6, 73.9, 50.9; HRMS (ESI) m/z 437.0901 ($M+H^+$), Calcd for $C_{23}H_{18}ClN_2O_5$ 437.0904; The ee was determined by HPLC analysis. CHIRALPAK IA (4.6 mm i.d. x 250 mm) + CHIRALPAK IA (4.6 mm i.d. x 250 mm); Hexane/2-propanol = 90/10; flow rate 1.0 mL/min; 25 °C; 254 nm; t_R = 21.3 min (minor peak), 27.4 min, 29.2 min, 32.4 min (major peak).



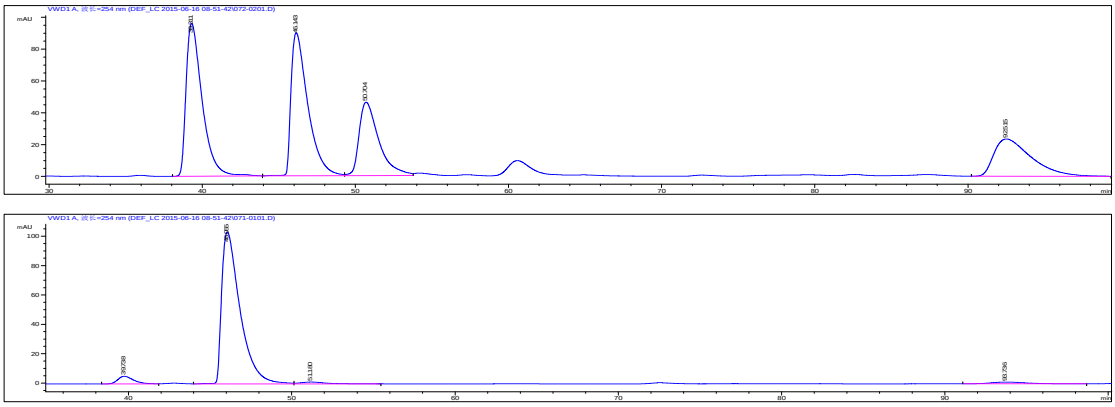


Entry	Retention Time	Area	Height	%Area
1	21.323	214	5.8	2.621
2	27.414	90.4	1.9	1.107
3	29.199	167	3.1	2.045
4	32.402	7694.8	133.8	94.227

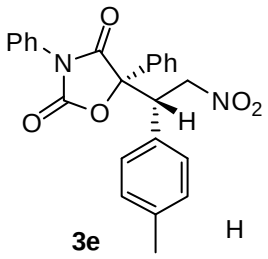


3d

White solid; Mp 106.7–107.2 °C; 92% ee; dr = >19:1; 92% yield; $[\alpha]_D^{22}$ +26.3 (*c* 1.00, $CHCl_3$); ^1H NMR (300 MHz, $CDCl_3$) δ 7.81 (d, *J* = 7.0, 2H), 7.62–7.18 (m, 10H), 6.62 (dd, *J* = 6.5, 2.6, 2H), 5.35 (dd, *J* = 11.3, 3.9, 1H), 4.85 (dd, *J* = 13.6, 11.5, 1H), 4.45 (dd, *J* = 13.8, 3.9, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 169.4, 152.6, 136.4, 132.6, 131.2, 130.6, 130.3, 130.0 (two peaks), 129.3 (two peaks), 128.0 (two peaks), 125.6, 124.9, 87.8, 74.5, 46.1; HRMS (ESI) m/z 437.0897 ($M+H^+$), Calcd for $C_{23}H_{18}ClN_2O_5$ 437.0904; The ee was determined by HPLC analysis. CHIRALPAK IA (4.6 mm i.d. x 250 mm) + CHIRALPAK ID-3 (4.6 mm i.d. x 250 mm);Hexane/2-propanol = 95/05; flow rate 1.0 mL/min; 25 °C; 254 nm; t_R = 39.7 min (minor peak), 46.1 min (major peak), 51.2 min, 93.7 min.

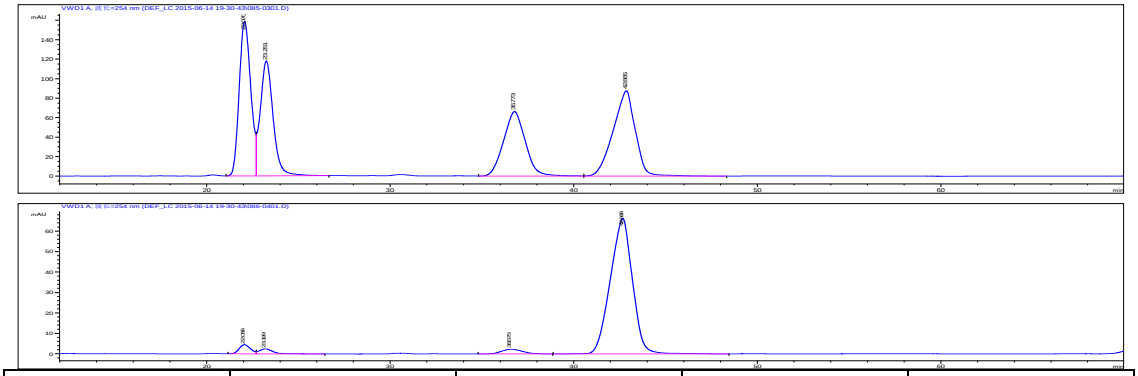


Entry	Retention Time	Area	Height	%Area
1	39.738	347.3	5.2	3.977
2	46.053	8076	103.5	92.481
3	51.174	131.1	1.2	1.502
4	93.74	178.3	1.2	2.041

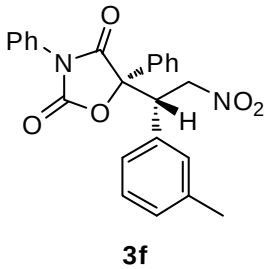


3e

White solid; Mp 146.5–147.7 °C; 94% ee; dr = 18:1; 88% yield; $[\alpha]_D^{22}$ +28.4 (*c* 1.00, $CHCl_3$); ^1H NMR (300 MHz, $CDCl_3$) δ 7.74 (dd, *J* = 17.9, 11.2, 2H), 7.54–7.35 (m, 3H), 7.32–7.08 (m, 7H), 6.67–6.39 (m, 2H), 4.86 (dd, *J*=13.3, 11.4, 1H), 4.53–4.21 (m, 2H), 2.24 (d, *J* = 6.1, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.4, 152.6, 139.6, 132.4, 130.1, 130.0 (two peaks), 129.6. 129.3, 129.2 (two peaks), 128.5, 125.7, 124.9, 88.1, 74.2, 51.1, 21.1; HRMS (ESI) m/z 417.1446 ($M+H^+$), Calcd for $C_{24}H_{21}N_2O_5$ 417.1450; The ee was determined by HPLC analysis. CHIRALPAK IA (4.6 mm i.d. x 250 mm) + CHIRALPAK IA (4.6 mm i.d. x 250 mm); Hexane/2-propanol = 90/10; flow rate 1.0 mL/min; 25 °C; 254 nm; t_R = 22.1 min (minor peak), 23.2min, 36.5 min, 42.7 min (major peak).



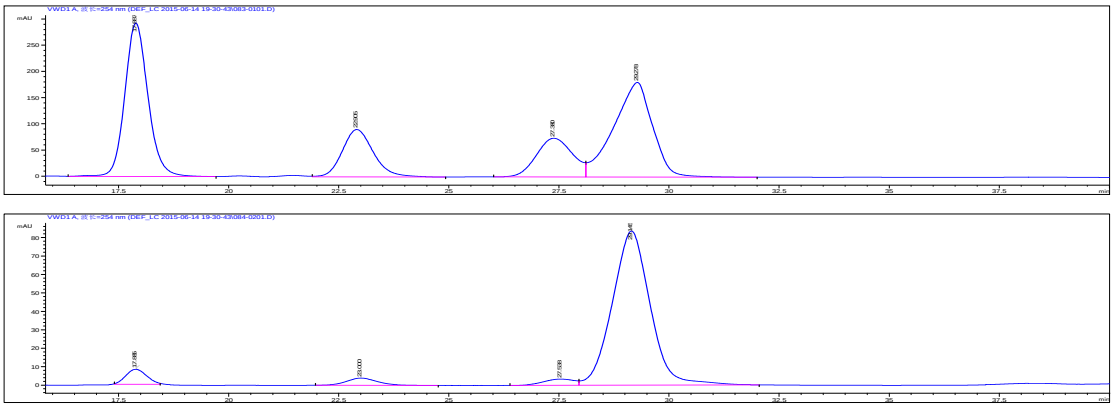
Entry	Retention Time	Area	Height	%Area
1	22.055	195	4.5	3.212
2	23.199	128	2.5	2.109
3	36.575	182.4	2.3	3.005
4	42.668	5565.6	66.2	91.674



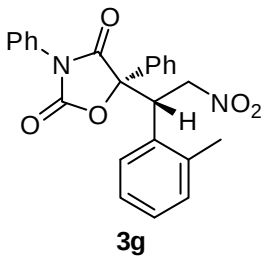
3f

White solid; Mp 139.8–140.2 °C; 90% ee; dr = 19:1; 96% yield; $[\alpha]_D^{22}$ +18.4 (*c* 1.00, $CHCl_3$); ^1H NMR (300 MHz, $CDCl_3$) δ 7.98–7.68 (m, 1H), 7.65–7.39 (m, 1H), 7.37–7.13 (m, 2H), 6.69–6.46 (m, 1H), 4.98 (dd, *J* = 13.3, 11.3, 0H), 4.65–4.33 (m, 1H), 2.35 (d, *J* = 10.2, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.3, 152.6, 139.2, 132.4, 131.6, 130.3, 130.1, 130.0, 129.6, 129.5, 129.4 (two peaks), 126.3, 125.7, 125.6, 125.0, 87.8, 74.1, 51.6, 21.4; HRMS (ESI) m/z 439.1266 ($M+Na^+$), Calcd for $C_{24}H_{20}N_2O_5Na$ 439.1270; The ee was determined by HPLC analysis. CHIRALPAK IA (4.6 mm i.d. x 250 mm) + CHIRALPAK IA (4.6 mm i.d. x 250 mm); Hexane/2-propanol = 90/10; flow rate 1.0 mL/min; 25 °C; 3 nm; t_R = 17.9 min (minor

peak), 23.0 min, 27.5 min, 29.1 min (minor peak).

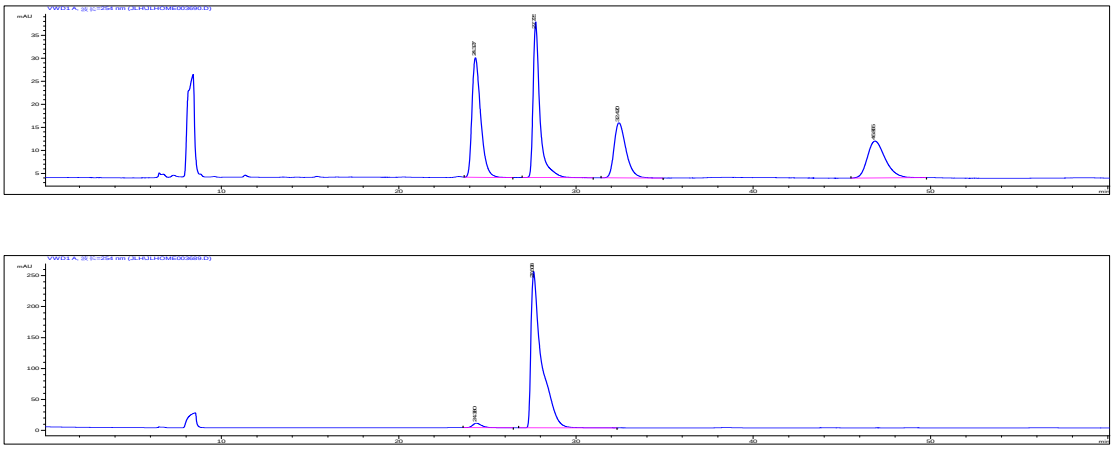


Entry	Retention Time	Area	Height	%Area
1	17.885	268.8	8.2	4.753
2	23.001	197	3.9	3.484
3	27.534	168.4	3.5	2.978
4	29.145	5020.1	83.7	88.785

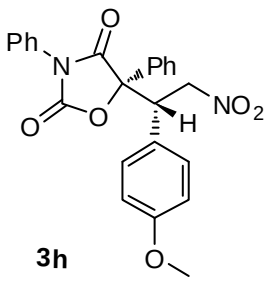


3g

White solid; Mp 205.9–206.1 °C; 96% ee; dr = >19:1; 95% yield; $[\alpha]_D^{22}$ +25.6 (*c* 1.00, $CHCl_3$); ^1H NMR (300 MHz, $CDCl_3$) δ 7.96–7.84 (m, 2H), 7.65–7.48 (m, 5H), 7.38–7.26 (m, 5H), 6.66–6.55 (m, 2H), 5.04–4.88 (m, 2H), 4.48 (q, *J* = 10.3, 1H), 2.59 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.4, 152.7, 138.9, 132.9. 131.9, 131.0, 130.1, 129.9, 129.6, 129.4, 129.3, 127.0, 126.4, 125.9, 125.6, 124.9, 88.3, 75.0, 46.3, 20.0; HRMS (ESI) m/z 417.1447 ($M+H^+$), Calcd for $C_{24}H_{21}N_2O_5$ 417.1450; The ee was determined by HPLC analysis. CHIRALPAK IA (4.6 mm i.d. x 250 mm) + CHIRALPAK ID-3 (4.6 mm i.d. x 250 mm); Hexane/2-propanol = 95/05; flow rate 1.0 mL/min; 25 °C; 254 nm; t_R = 24.3 min (minor peak), 27.6 min (major peak).

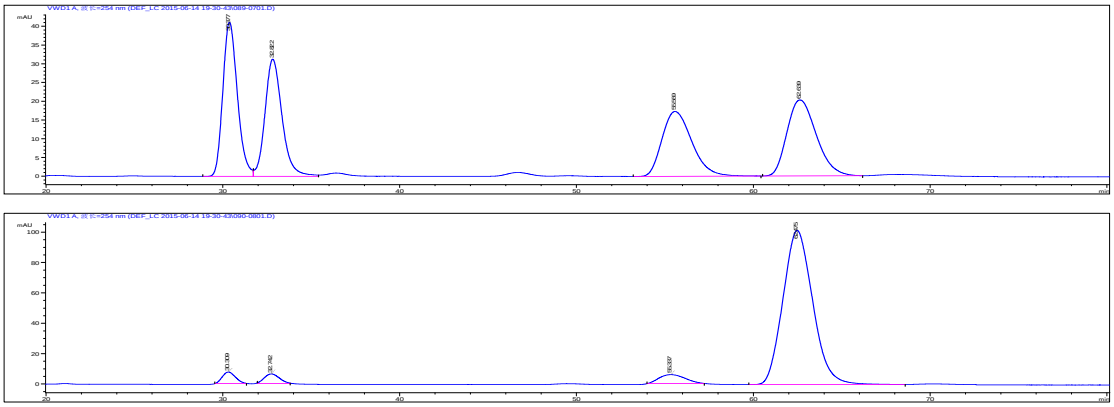


Entry	Retention Time	Area	Height	%Area
1	24.38	254.2	7.1	2.366
2	27.608	10489.2	252.2	97.634

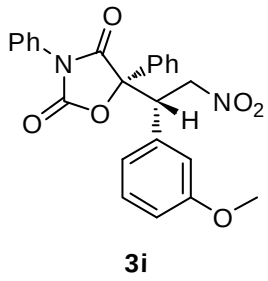


3h

White solid; Mp 102.3–103.7 °C; 91% ee; dr = 16:1; 96% yield; $[\alpha]_D^{22}$ +21.2 (*c* 1.00, $CHCl_3$); ^1H NMR (300 MHz, $CDCl_3$) δ 7.95–7.82 (m, 2H), 7.63–7.27 (m, 8H), 7.08–6.84 (m, 2H), 6.69 (dd, *J* = 6.7, 2.9, 2H), 4.93 (dd, *J* = 13.1, 11.6, 1H), 4.61–4.33 (m, 2H), 3.89–3.74 (m, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.5, 160.5, 152.7, 132.4, 130.5, 130.1, 130.0, 129.6, 129.4, 129.3, 125.7, 124.9, 123.3, 114.8, 88.2, 74.4, 55.4, 50.8; HRMS (ESI) m/z 433.1404 ($M+H^+$), Calcd for $C_{24}H_{21}N_2O_6$ 433.1400; The ee was determined by HPLC analysis. CHIRALPAK IA (4.6 mm i.d. x 250 mm) + CHIRALPAK IA (4.6 mm i.d. x 250 mm); Hexane/2-propanol = 90/10; flow rate 1.0 mL/min; 25 °C; 254 nm; t_R = 30.3 min (minor peak), 32.7 min, 55.3 min, 62.4 min (major peak).



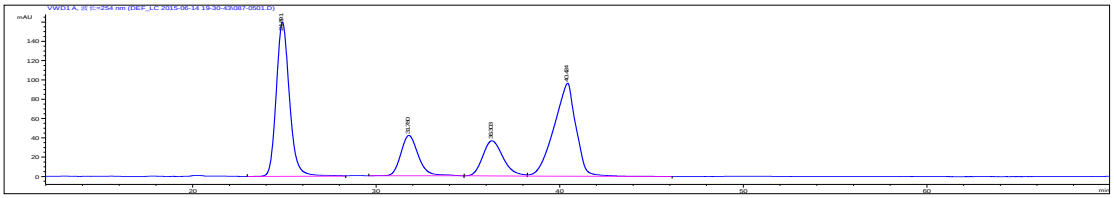
Entry	Retention Time	Area	Height	%Area
1	30.31	378.4	7.5	2.867
2	32.74	367.1	6.3	2.782
3	55.338	583.9	5.8	4.425
4	62.475	11867.2	101.7	89.926

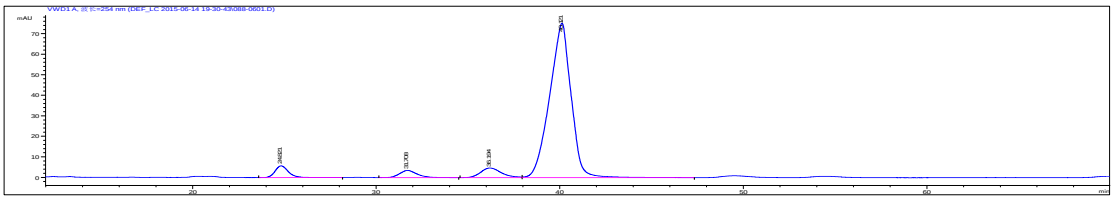


3i

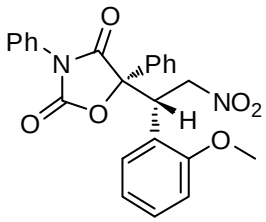
White solid; Mp 106.6–108.1 °C; 91% ee; dr = 14:1; 98% yield; $[\alpha]_D^{22}$ +27.4 (*c* 1.00, $CHCl_3$); ^1H NMR (300 MHz, $CDCl_3$) δ 7.86 (dd, *J* = 8.1, 1.4, 2H), 7.62–7.17 (m, 7H), 7.14–6.82 (m, 3H), 6.67 (dt, *J* = 8.7, 3.6, 2H), 4.97 (dd, *J*=13.4, 11.3, 1H), 4.68–4.28 (m, 2H), 3.78 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.3, 160.1, 152.6, 133.1, 132.3, 130.4, 130.2, 130.0, 129.6, 129.3, 125.7, 124.9, 121.4, 115.6, 115.0, 114.4, , 129.7, 87.9, 74.2,

55.4, 51.4; HRMS (ESI) m/z 433.1411 ($M+H^+$), Calcd for $C_{24}H_{21}N_2O_6$ 433.1400; The ee was determined by HPLC analysis. CHIRALPAK IA (4.6 mm i.d. x 250 mm) + CHIRALPAK IA (4.6 mm i.d. x 250 mm); Hexane/2-propanol = 90/10; flow rate 1.0 mL/min; 25 °C; 254 nm; t_R = 24.9 min (minor peak), 31.8min, 36.3 min, 40.4 min (major peak).



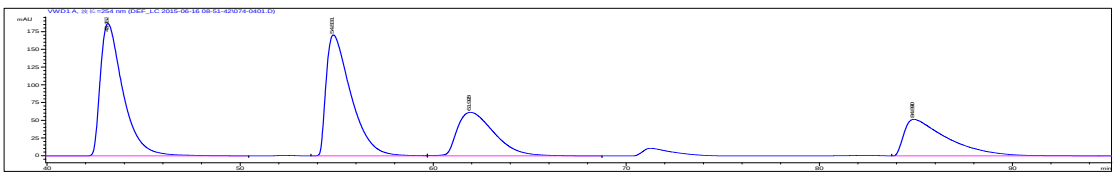


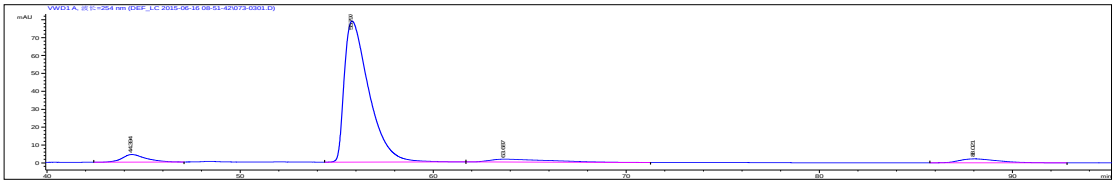
Entry	Retention Time	Area	Height	%Area
1	24.822	280.2	5.7	4.032
2	31.708	223.8	3.4	3.221
3	36.194	356.8	4.6	5.135
4	40.122	6088.6	75.2	87.612



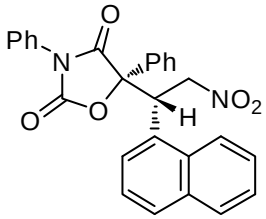
3j

White solid; Mp 117.5–119.2 °C; 91% ee; dr = 8:1; 95% yield; $[\alpha]_D^{22}$ +31.7 (*c* 1.00, $CHCl_3$); ^1H NMR (300 MHz, $CDCl_3$) δ 7.89 (dd, *J* = 8.1, 1.3, 2H), 7.60–7.27 (m, 8H), 6.96 (m, 2H), 6.65 (dd, *J* = 6.7, 2.9, 2H), 5.39–4.93 (m, 2H), 4.50 (dd, *J* = 13.5, 4.3, 1H), 3.84 (d, *J* = 23.4, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 169.9, 158.1, 152.9, 132.9, 130.7, 130.2, 130.0, 129.4, 129.3, 129.2, 128.9, 125.9, 125.1, 121.2, 120.8, 112.1, 74.3, 56.2; HRMS (ESI) m/z 433.1403 ($M+H^+$), Calcd for $C_{24}H_{21}N_2O_6$ 433.1400; The ee was determined by HPLC analysis. CHIRALPAK IA (4.6 mm i.d. x 250 mm) + CHIRALPAK ID-3 (4.6 mm i.d. x 250 mm); Hexane/2-propanol = 95/05; flow rate 1.0 mL/min; 25 °C; 254 nm; t_R = 44.4 min (minor peak), 55.8 min (major peak), 63.7 min, 88.0 min.



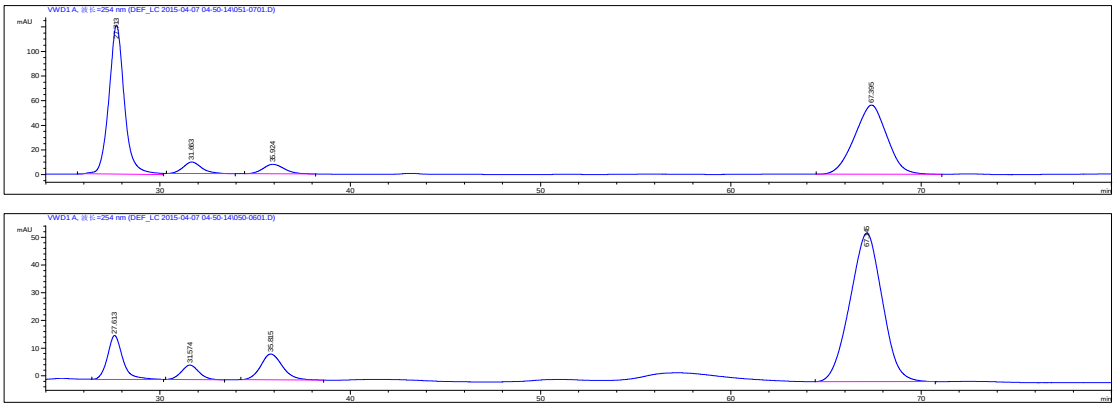


Entry	Retention Time	Area	Height	%Area
1	44.398	353.8	4.2	4.458
2	55.795	6949.3	78.8	87.552
3	63.684	335	1.6	4.221
4	88.011	299.2	2.2	3.769

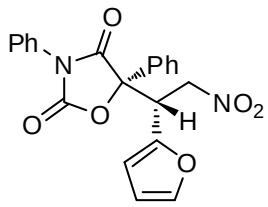


3k

White solid; Mp 130.8–132.3 °C; 86% ee; dr = 7:1; 95% yield; $[\alpha]_D^{22}$ +21.1 (*c* 1.00, $CHCl_3$); ^1H NMR (300 MHz, $CDCl_3$) δ 8.04–7.70 (m, 6H), 7.65–6.95 (m, 9H), 6.35 (d, *J* = 7.8, 2H), 5.27–4.96 (m, 1H), 4.86–4.38 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.4, 152.6, 133.5, 133.2, 132.4, 130.2, 129.7, 129.4, 129.3, 129.1, 129.0, 128.3, 127.6, 127.2, 127.0, 125.7, 125.5, 125.0, 88.2, 74.3, 51.6; HRMS (ESI) m/z 453.1441 ($M+H^+$), Calcd for $C_{27}H_{21}N_2O_5$; The ee was determined by HPLC analysis. CHIRALPAK IA (4.6 mm i.d. x 250 mm) + CHIRALPAK IA (4.6 mm i.d. x 250 mm); Hexane/2-propanol = 90/10; flow rate 1.0 mL/min; 25 °C; 254 nm; t_R = 30.4 min (minor peak), 37.8 min, 41.6 min, 69.3 min (major peak).

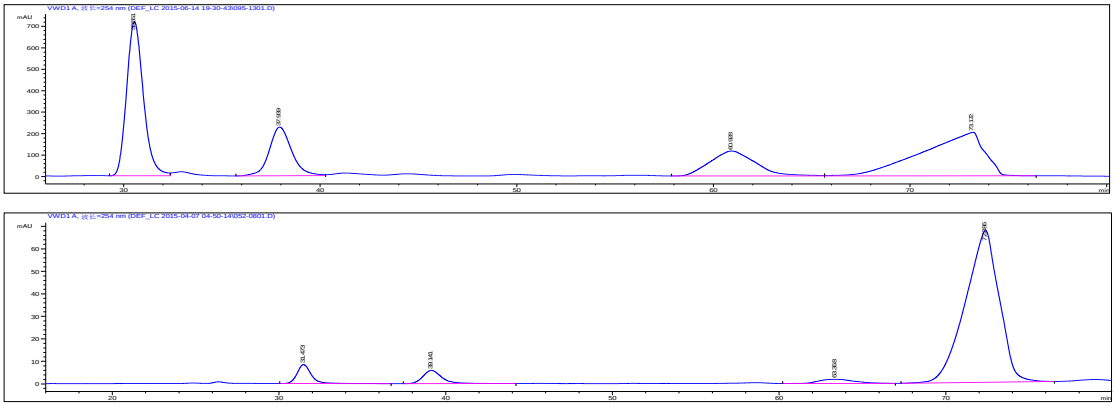


Entry	Retention Time	Area	Height	%Area
1	30.466	534.3	8.9	6.126
2	37.881	628	8.2	7.200
3	41.688	472.9	6.8	5.422
4	69.346	7087.1	46.3	81.252

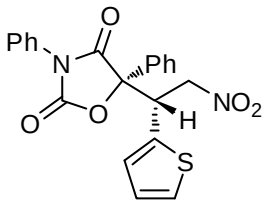


3l

White solid; Mp 152.9–154.1 °C; 94% ee; dr = 16:1; 98% yield; $[\alpha]_D^{22}$ +25.8 (*c* 1.00, $CHCl_3$); ^1H NMR (300 MHz, $CDCl_3$) δ 7.89 (m, 7H), 7.71–7.44 (m, 7H), 7.21–6.99 (m, 3H), 6.35 (d, *J* = 7.8, 2H), 5.12 (dd, *J* = 13.5, 11.5, 1H), 4.86–4.38 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.5, 152.7, 133.6, 133.2, 132.5, 130.3, 129.8, 129.2, 128.4, 127.7, 127.3, 127.1, 125.6, 125.1, 88.3, 74.4, 51.7;HRMS (ESI) m/z 415.0916 ($M+Na^+$), Calcd for $C_{21}H_{16}N_2O_6Na$ 415.0906; The ee was determined by HPLC analysis. CHIRALPAK IA (4.6 mm i.d. x 250 mm) + CHIRALPAK IA (4.6 mm i.d. x 250 mm); Hexane/2-propanol = 90/10; flow rate 1.0 mL/min; 25 °C; 254 nm; t_R = 31.9 min (minor peak), 39.1 min, 63.4 min, 72.4 min (major peak).



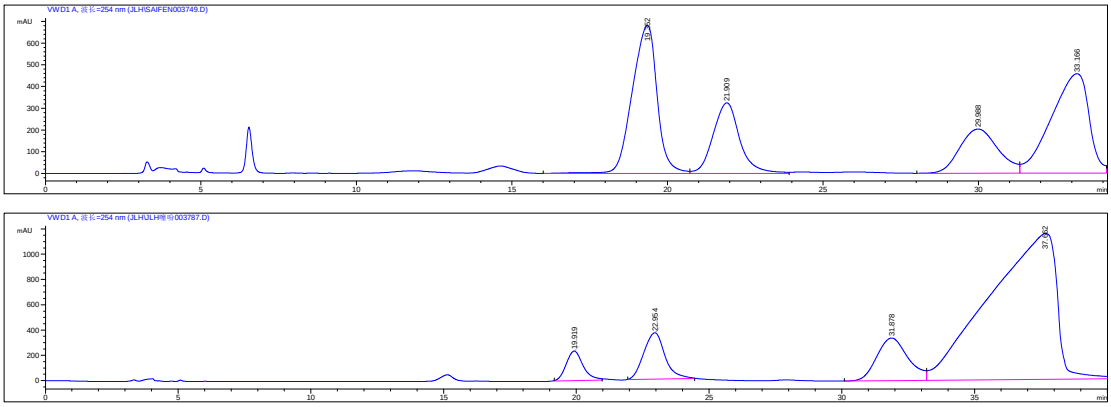
Entry	Retention Time	Area	Height	%Area
1	31.474	495.9	8.4	4.607
2	39.141	479.3	5.9	4.453
3	63.368	276.7	1.9	2.571
4	72.386	9511.3	67.4	88.369



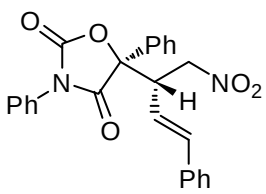
3m

White solid; Mp 114.5–116.2 °C; 91% ee; dr = 8:1; 98% yield; $[\alpha]_D^{22}$ +18.2 (*c* 1.00, $CHCl_3$); ^1H NMR (300 MHz, $CDCl_3$) δ 7.85 (d, *J* = 6.7, 2H), 7.63–7.47 (m, 3H), 7.44–7.32 (m, 4H), 7.24 (d, *J* = 3.5, 1H), 7.16–6.95 (m, 1H), 6.81 (dd, *J* = 6.1, 3.2, 2H), 4.95–4.75 (m, 2H), 4.44 (dd, *J* = 12.2, 2.3, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.2, 152.7, 133.2, 132.0, 130.3, 130.0, 129.7, 129.6, 129.4, 127.6, 127.3, 125.8, 124.9, 87.8, 75.6, 47.1; HRMS (ESI) m/z 431.0671 ($M+Na^+$), Calcd for $C_{21}H_{16}N_2O_5SNa$ 431.0678; The ee was determined by HPLC analysis. CHIRALPAK IA (4.6 mm i.d. x 250 mm); Hexane/2-propanol = 95/05; flow rate 1.0 mL/min; 25 °C; 254 nm; t_R = 19.9 min (minor peak), 22.9 min, 31.9

min, 37.7 min (major peak).

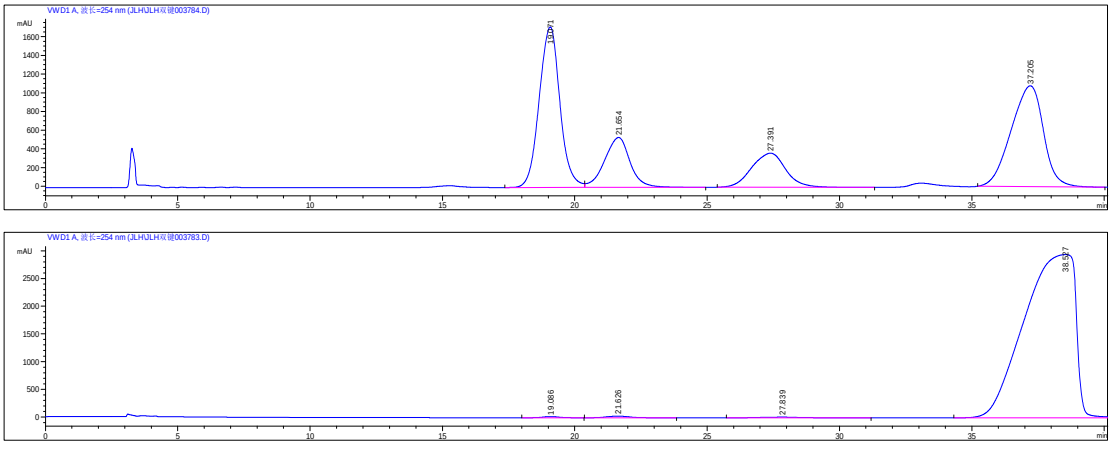


Entry	Retention Time	Area	Height	%Area
1	19.919	10332.5	233.3	3.942
2	22.954	20514.5	367.9	7.826
3	31.878	27664	337.3	10.553
4	37.662	203626.3	1155.5	77.679

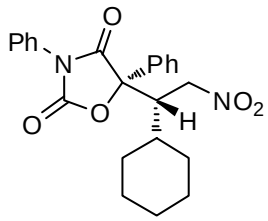


3n

White solid; Mp 170.9–172.1 °C; 99% ee; dr = 19:1; 90% yield; $[\alpha]_D^{22}$ +20.6 (*c* 1.00, $CHCl_3$); ^1H NMR (300 MHz, $CDCl_3$) δ 7.91–7.71 (m, 2H), 7.64–7.45 (m, 3H), 7.39–7.29 (m, 8H), 7.07 (dd, *J* = 7.5, 2.1, 2H), 6.88 (d, *J* = 15.9, 1H), 6.12 (dd, *J* = 15.9, 9.1, 1H), 4.74–4.43 (m, 1H), 4.33–4.06 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.7, 152.9, 139.8, 135.1, 132.1, 130.2, 129.7, 129.4, 129.0, 128.8, 126.8, 126.1, 125.9, 124.7, 118.4, 88.2, 74.8, 49.4; HRMS (ESI) m/z 451.1282 ($M+Na^+$), Calcd for $C_{25}H_{20}N_2O_5Na$ 451.1270; The ee was determined by HPLC analysis. CHIRALPAK IA (4.6 mm i.d. x 250 mm); Hexane/2-propanol = 95/05; flow rate 1.0 mL/min; 25 °C; 254 nm; t_R = 19.1 min (minor peak), 21.6 min, 27.8 min, 38.5 min (major peak).

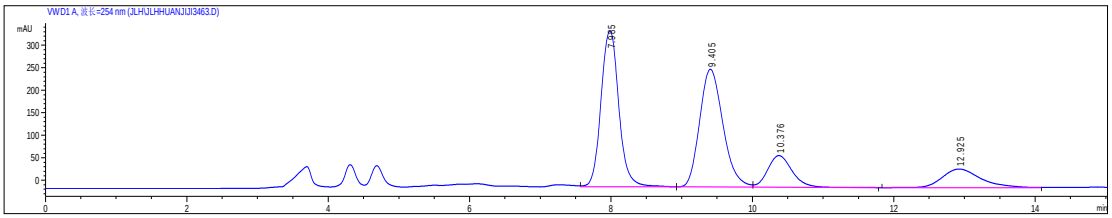


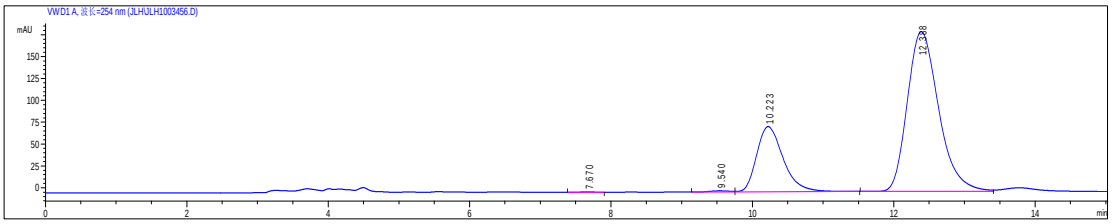
Entry	Retention Time	Area	Height	%Area
1	19.086	835	19	0.208
2	21.626	1663.8	28.3	0.415
3	27.839	1208.5	11.8	0.301
4	38.527	397356	2945.3	99.076



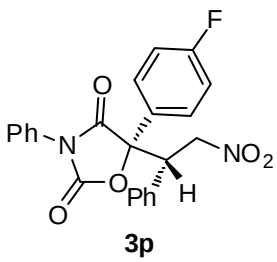
3o

White solid; Mp 130.5–131.7 °C; 50% ee; dr = 15:1; 59% yield; $[\alpha]_D^{22}$ +23.3 (*c* 1.00, $CHCl_3$); ^1H NMR (300 MHz, $CDCl_3$) δ 7.68 (ddd, *J* = 7.5, 4.0, 2.3, 2H), 7.58–7.30 (m, 8H), 4.52 (dd, *J* = 14.2, 6.8, 1H), 4.31 (dd, *J* = 14.2, 4.8, 1H), 3.47 (t, *J* = 5.1, 1H), 1.94–1.60 (m, 6H), 1.15 (m, 4H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 171.5, 152.8, 133.6, 130.4, 130.0, 129.5, 129.3, 125.6, 124.9, 89.9, 72.0, 49.6, 39.5, 33.0, 28.9, 26.9, 26.7, 25.8; HRMS (ESI) m/z 409.1759 ($M+H^+$), Calcd for $C_{23}H_{25}N_2O_5$ 409.1763; The ee was determined by HPLC analysis. CHIRALPAK IA (4.6 mm i.d. x 250 mm); Hexane/2-propanol = 90/10; flow rate 1.0 mL/min; 25 °C; 254 nm; t_R = 7.6 min, 9.5 min, 10.2 min (minor peak), 12.4 min (major peak).





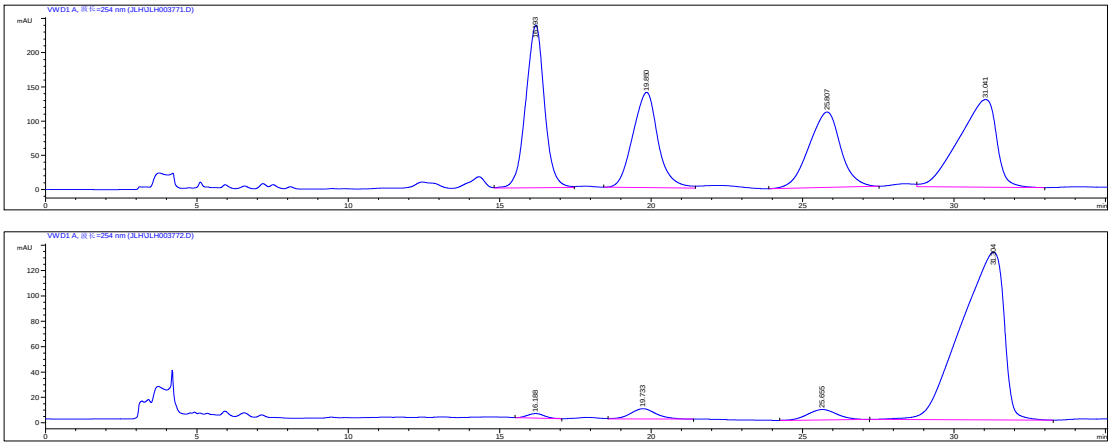
Entry	Retention Time	Area	Height	%Area
1	7.67	4.8	3.2E-1	0.063
2	9.54	24.8	1.3	0.327
3	10.223	1940.5	74.5	25.567
4	12.388	5619.8	182.6	74.043



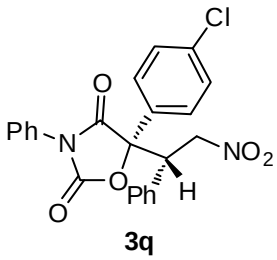
3p

White solid; Mp 162.3–163.1 °C; 98% ee; dr = 14:1; 90% yield; $[\alpha]_D^{22}$ +20.5 (*c* 1.00, $CHCl_3$); ^1H NMR (300 MHz, $CDCl_3$) δ 7.89 (dd, *J* = 8.4, 5.1, 2H), 7.58–7.16 (m, 10H), 6.63 (d, *J* = 5.4, 2H),

5.13–4.91 (m, 1H), 4.64–4.32 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.3, 165.3, 162.0, 152.4, 131.5, 129.7, 129.5, 129.3, 128.1, 127.2, 127.0, 125.7, 116.9, 116.6, 87.6, 74.1, 51.4; HRMS (ESI) m/z 421.1195 ($M+H^+$), Calcd for $C_{23}H_{18}FN_2O_5$ 421.1200; The ee was determined by HPLC analysis. CHIRALPAK IA (4.6 mm i.d. x 250 mm); Hexane/2-propanol = 95/05; flow rate 1.0 mL/min; 25 °C; 254 nm; t_R = 16.2 min (minor peak), 19.7 min, 25.6 min, 31.3 min (major peak).

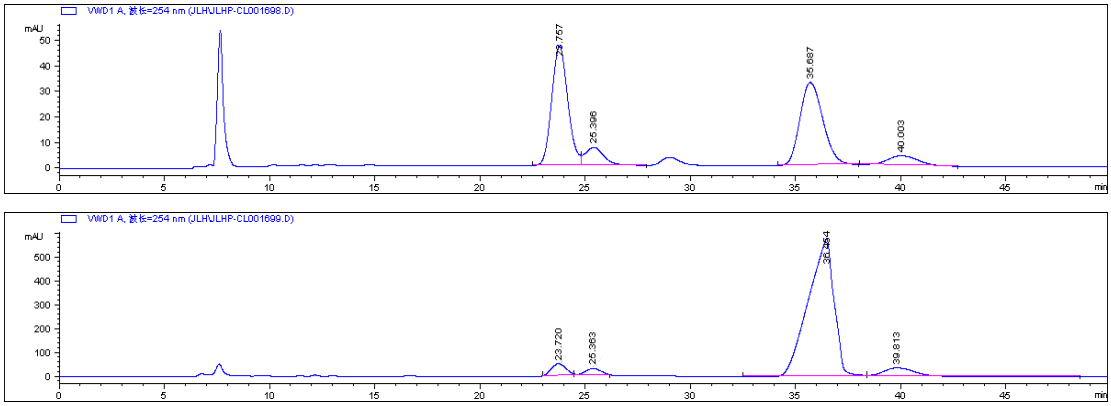


Entry	Retention Time	Area	Height	%Area
1	16.188	136.4	3.6	0.963
2	19.733	477.1	8.1	3.368
3	25.655	545.4	8.2	3.850
4	31.304	13007.1	132.1	91.819

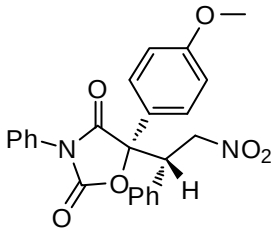


3q

White solid; Mp 151.8–153.1 °C; 91% ee; dr = 12:1; 89% yield; $[\alpha]_D^{22}$ +21.3 (*c* 1.00, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.74 (d, *J* = 8.4, 2H), 7.52–7.13 (m, 13H), 6.52 (d, *J* = 7.9, 2H), 4.96–4.84 (m, 1H), 4.45 (dd, *J* = 11.1, 3.8, 1H), 4.36 (dd, *J* = 13.4, 3.9, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.1, 152.3, 136.6, 131.4, 130.8, 130.1, 129.8, 129.7, 129.5, 129.4, 129.3, 126.4, 125.8, 87.6, 74.1, 51.4; HRMS (ESI) m/z 437.0901 ($M+H^+$), Calcd for $C_{23}H_{18}ClN_2O_5$ 437.0904;The ee was determined by HPLC analysis. CHIRALPAK IA (4.6 mm i.d. x 250 mm) + CHIRALPAK ID-3 (4.6 mm i.d. x 250 mm); Hexane/2-propanol = 90/10; flow rate 1.0 mL/min; 25 °C; 254 nm; t_R = 23.7 min (minor peak), 25.3 min, 36.4 min (major peak), 39.8 min.

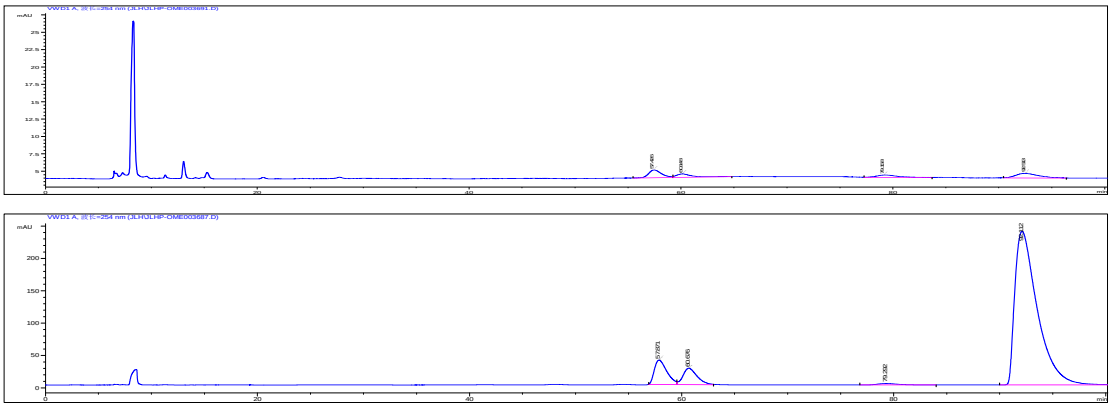


Entry	Retention Time	Area	Height	%Area
1	23.72	2445.5	50.1	4.288
2	25.363	1472.7	27.5	2.582
3	36.454	49165.5	577.9	86.198
4	39.813	3954.4	36.2	6.933



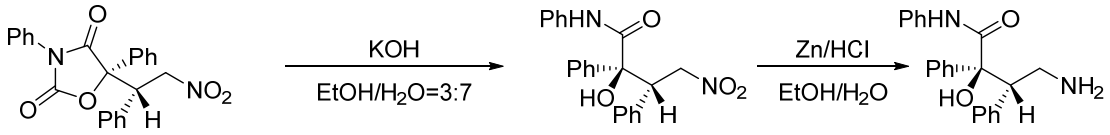
3r

White solid; Mp 156.7–158.2 °C; 85% ee; dr = 11:1; 91% yield; $[\alpha]_D^{22}$ +25.3 (*c* 1.00, $CHCl_3$); ^1H NMR (300 MHz, $CDCl_3$) δ 7.76 (d, *J* = 8.9, 2H), 7.42 (ddd, *J* = 11.0, 9.7, 4.7, 7H), 7.33–7.28 (m, 2H), 7.09–6.95 (m, 2H), 6.65–6.54 (m, 2H), 4.97 (td, *J* = 14.5, 4.7, 1H), 4.67–4.31 (m, 2H), 3.86 (d, *J* = 4.3, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.6, 160.9, 152.7, 131.8, 129.9, 129.5, 129.4, 129.3, 129.1, 127.1, 126.3, 125.7, 124.0, 114.9, 87.9, 74.3, 55.5, 51.4; HRMS (ESI) m/z 455.1225 ($M+Na^+$), Calcd for $C_{24}H_{20}N_2O_6Na$ 455.1219; The ee was determined by HPLC analysis. CHIRALPAK IA (4.6 mm i.d. x 250 mm) + CHIRALPAK ID-3 (4.6 mm i.d. x 250 mm); Hexane/2-propanol = 95/5; flow rate 1.0 mL/min; 25 °C; 254 nm; t_R = 57.8 min (minor peak), 60.6 min, 79.3 min, 92.1 min (major peak).



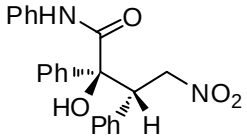
Entry	Retention Time	Area	Height	%Area
1	57.871	3100.7	37.8	7.590
2	60.676	2340	25.4	5.728
3	79.292	312.7	2.4	0.765
4	92.112	35097.4	237.6	85.916

4. Synthesis and characterization of 4



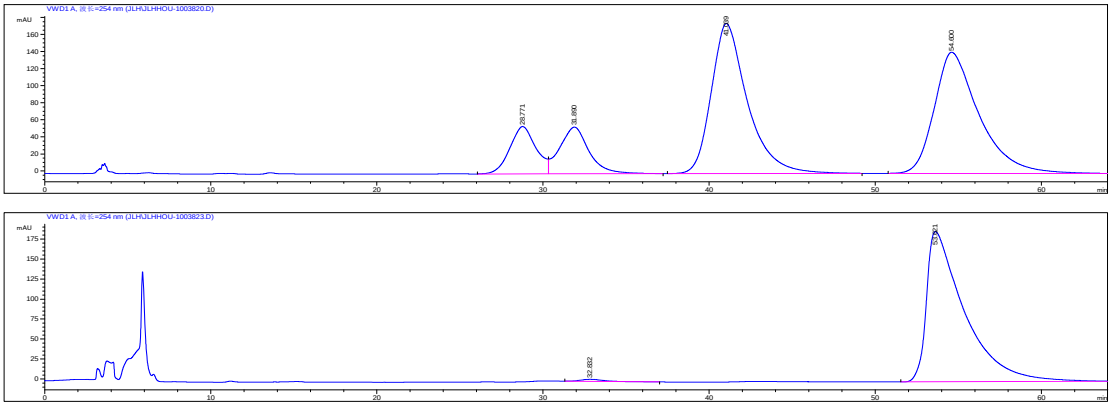
3a **4** **5**

A mixture of **3a** (0.169 g, 0.42 mmol) and KOH (0.094 g, 1.68 mmol) in a mixture of EtOH/H_2O (3:7) (10 mL) was stirred at room temperature for 1 h. The solvent was removed under vacuum, and the residue was dissolved in CH_2Cl_2 (10 mL) and washed with a 5% aqueous solution of HCl until neutral. The organic layer was dried (Na_2SO_4), and the solvent removed under vacuum. The residue was purified by column chromatography *silica gel* to give **4** as a white solid.



4

White solid; Mp 152.8–154.1 °C; >99% ee; dr > 19:1; 85% yield; $[\alpha]_D^{22}$ +28.8 (*c* 1.00, $CHCl_3$); ^{1}H NMR (300 MHz, Acetone) δ 8.97 (s, 1H), 7.97 (dd, *J* = 10.2, 8.8, 2H), 7.76–6.44 (m, 13H), 5.70 (s, 1H), 5.12–4.80 (m, 2H), 4.57 (dd, *J* = 12.4, 2.9, 1H); ^{13}C NMR (75 MHz, Acetone) δ 169.8, 140.3, 137.9, 135.7, 130.1, 128.6, 128.5, 128.2, 128.0, 127.8, 125.8, 124.0, 120.0, 80.6, 76.7, 50.5; HRMS (ESI) m/z 377.1502 ($M+H^+$), Calcd for $C_{22}H_{21}N_2O_4$ 377.1501; The ee was determined by HPLC analysis. CHIRALPAK IA (4.6 mm i.d. x 250 mm); Hexane/2-propanol = 90/10; flow rate 1.0 mL/min; 25 °C; 254 nm; t_R = 28.8 min, 31.9 min, 41.0 min (minor peak), 54.6 min (major peak).



Entry	Retention Time	Area	Height	%Area
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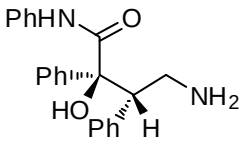
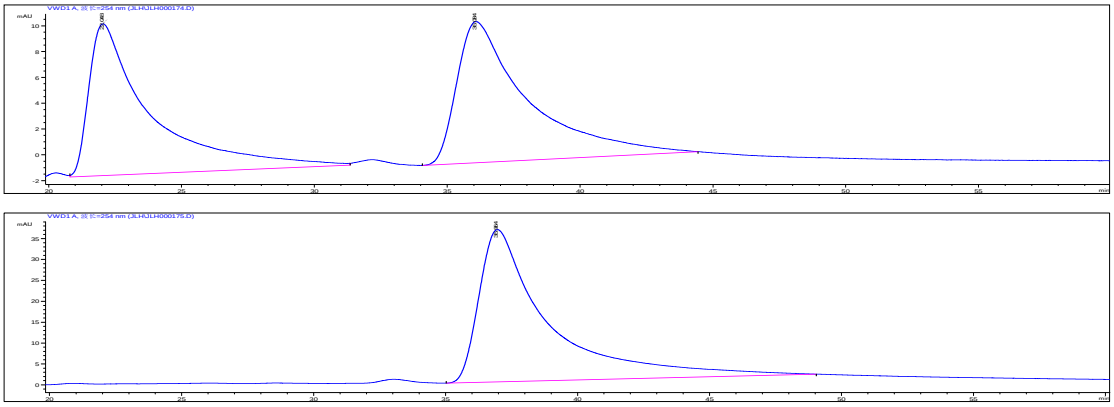
2	53.621	29879.6	187.7	99.195

To a solution of **3a** (68 mg, 0.18 mmol) in EtOH/H_2O (1.7 and 0.2 mL) was added Zn dust (130 mg, 2.02 mmol) and *conc.* HCl (0.34 mL). The reaction mixture was refluxed for 4 hours. Reaction quenched with sat. NH_4Cl (aq) (10 mL) and extracted with CH_2Cl_2 (3 x 10 mL), dried (magnesium sulfate), filtered and concentrated *in vacuo* to afford the compound **5** as a colorless oil.



5

Colorless oil; >99% ee; dr > 19:1; 78% yield; $[\alpha]_D^{22}$ +30.2 (*c* 1.00, $CHCl_3$); ^{1}H NMR (300 MHz, DMSO) δ 9.37 (d, *J* = 42.8, 1H), 8.12–6.80 (m, 15H), 4.04 (d, *J* = 9.3, 2H), 3.22–2.95 (m, 2H), 2.70 (d, *J* = 12.8, 1H); ^{13}C NMR (75 MHz, DMSO) δ 171.3, 142.9, 138.5, 130.8, 128.8, 128.5, 128.3, 127.9, 127.4, 126.5, 124.0, 120.3, 82.0, 50.8, 41.4; HRMS (ESI) m/z 347.1712 ($M+H^+$), Calcd for $C_{22}H_{21}N_2O_4$ 347.1715; The ee was determined by HPLC analysis. CHIRALPAK IA (4.6 mm i.d. x 250 mm); Hexane/2-propanol = 80/10; flow rate 1.0 mL/min; 25 °C; 254 nm; t_R = 22.1 min (minor peak), 36.1 min (major peak).



Entry	Retention Time	Area	Height	%Area
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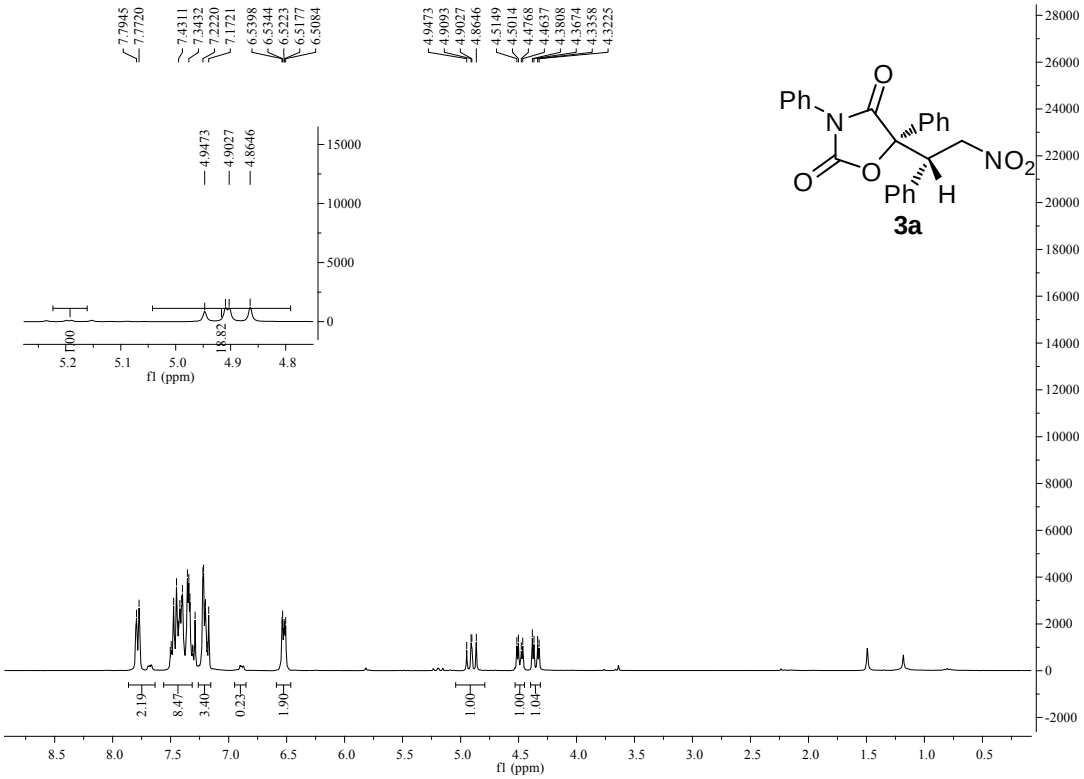
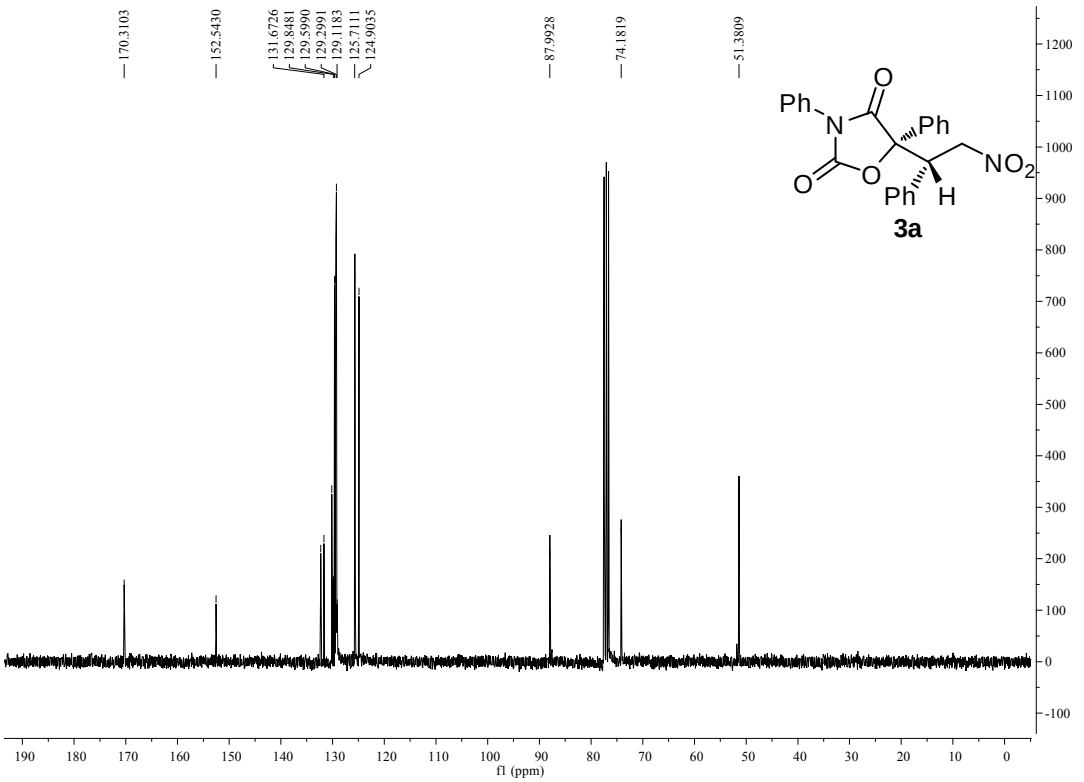
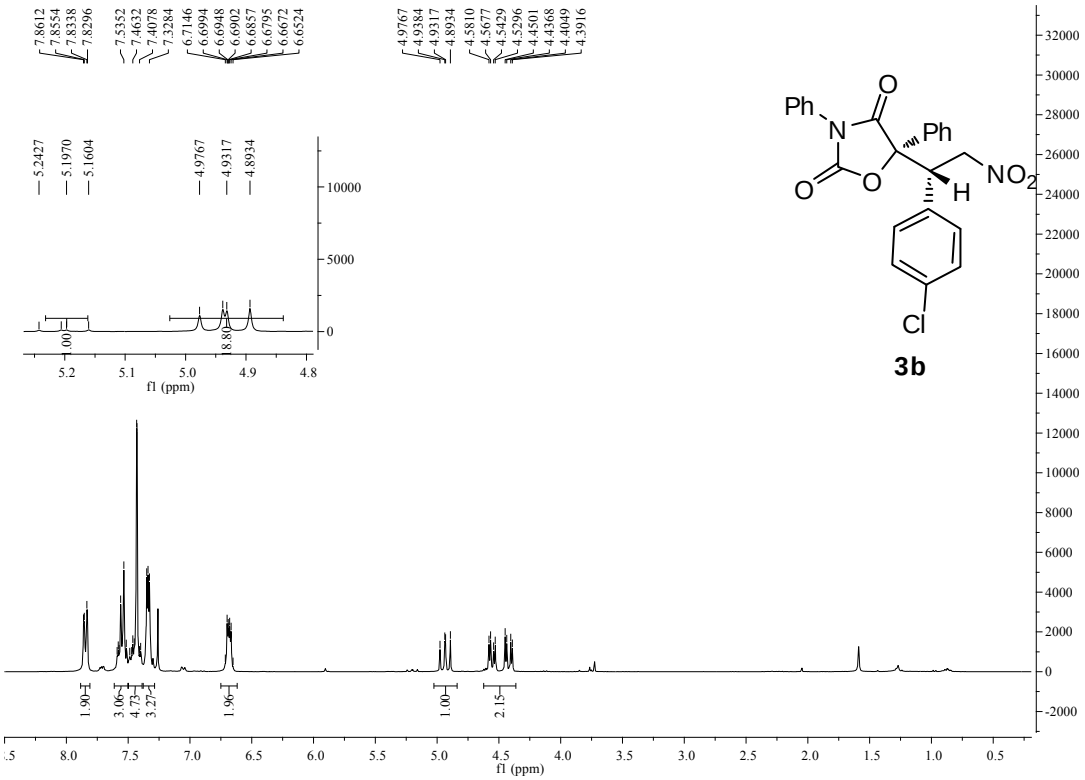
5. Copies of NMR Spectra



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6.6857
6.6795
6.6672
6.6524
4.9767
4.9384
4.9317
4.8934
4.5810
4.5677
4.5429
4.5296
4.4501
4.4368
4.4049
4.3916
5.2427
5.1970
5.1604
4.9767
4.9317
4.8934
1.00
18.80
f1 (ppm)
Ph
N
O
O
O
Ph
NO2
H
Cl
3b
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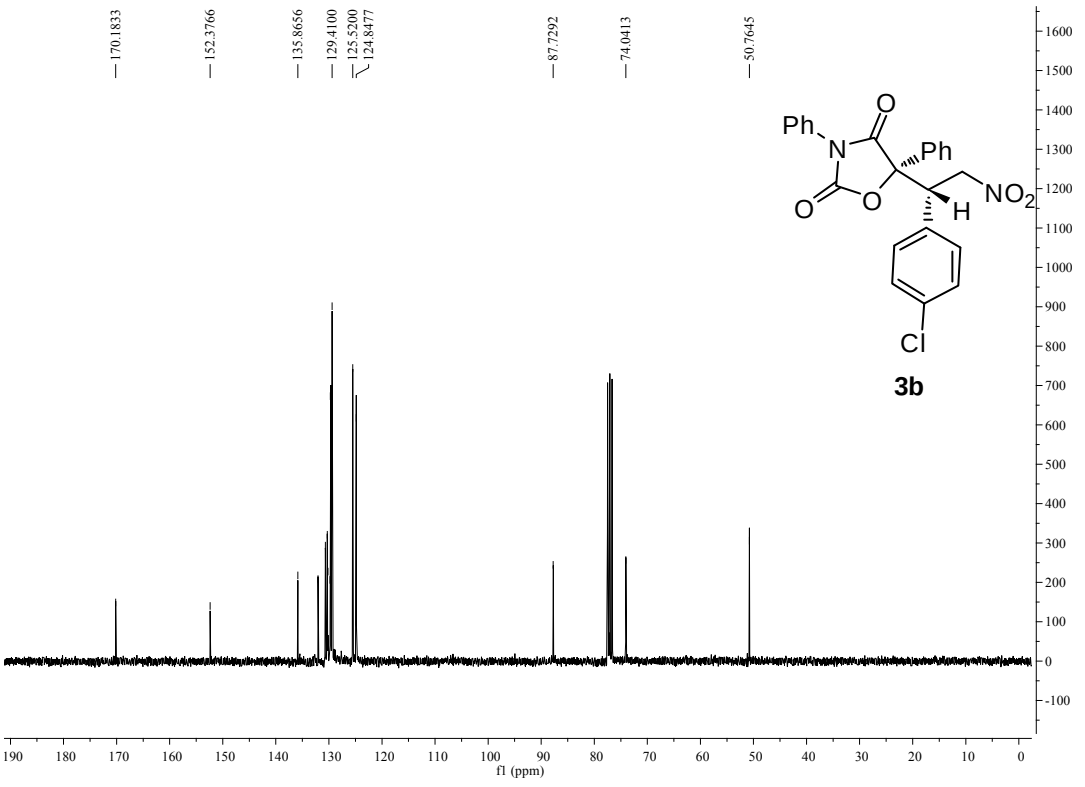
3.06
4.73
3.27
1.96
1.00
2.15
f1 (ppm)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135.8656
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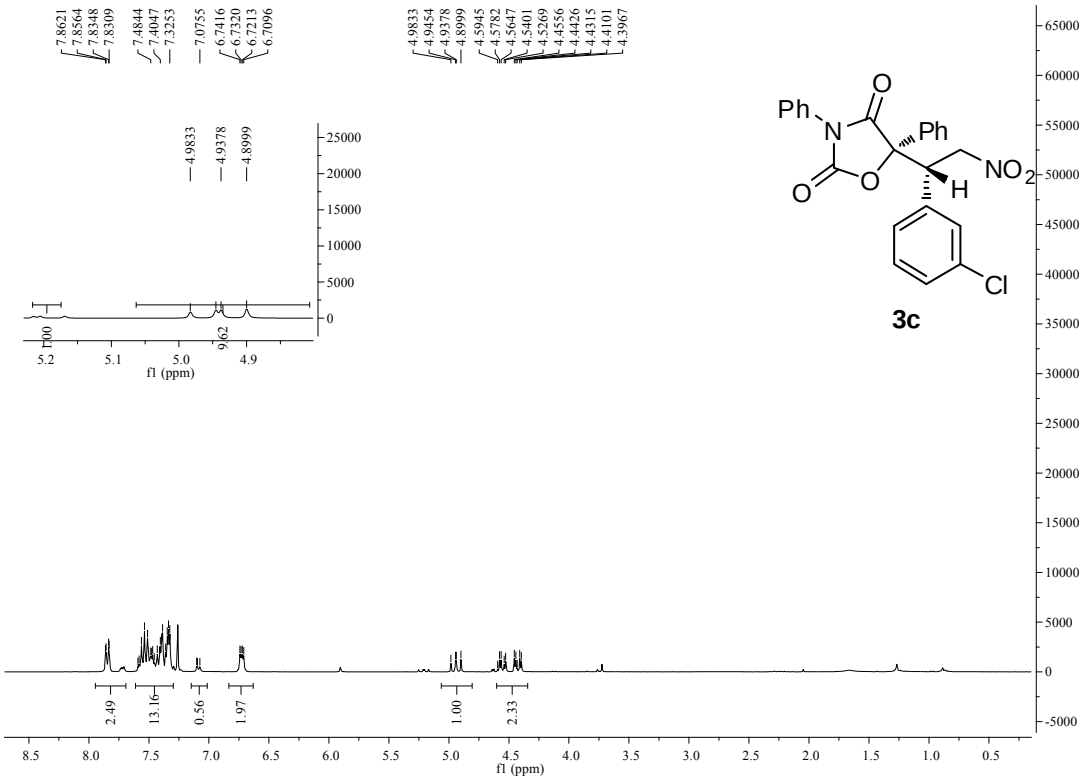
O
O
Ph
NO2
H
Cl
3b
f1 (ppm)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6.7213
6.7096
4.9833
4.9454
4.9378
4.8999
4.5945
4.5782
4.5647
4.5401
4.5269
4.4556
4.4426
4.4315
4.4101
4.3967
Ph
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O
O
Ph
NO2
H
Cl
3c
f1 (ppm)
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13.16
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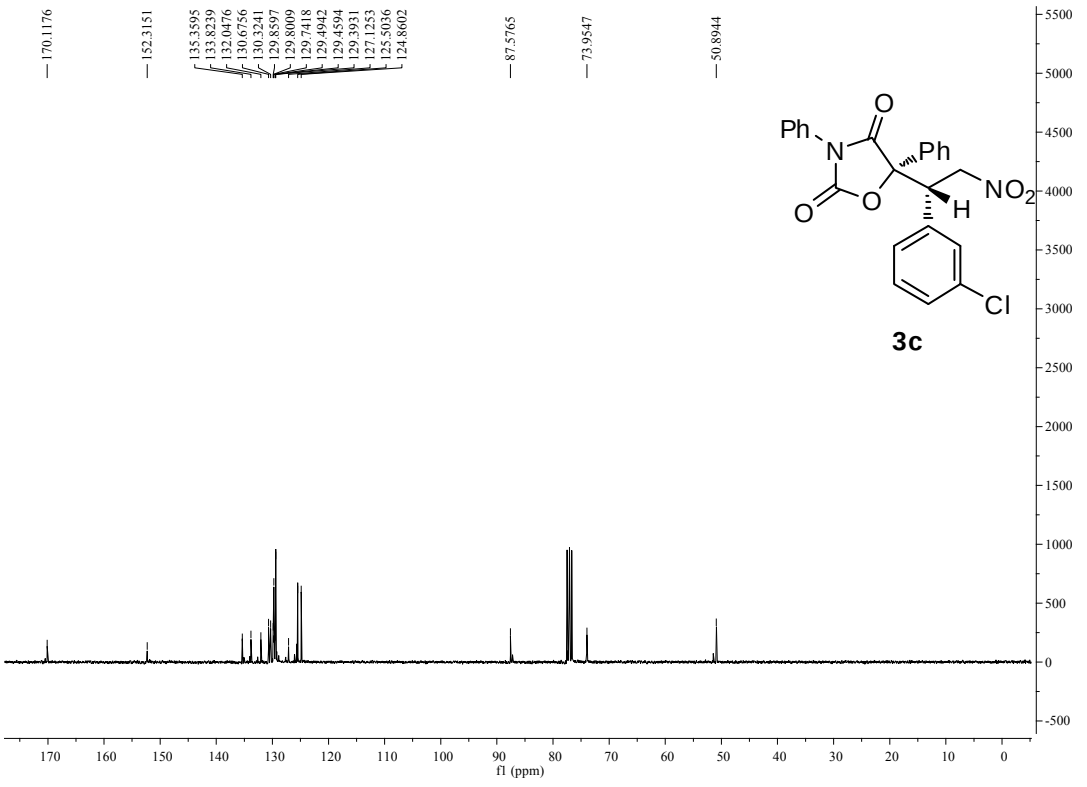
1.00
2.33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129.4942
129.4594
129.3931
127.1253
125.5036
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73.9547
50.8944
Ph
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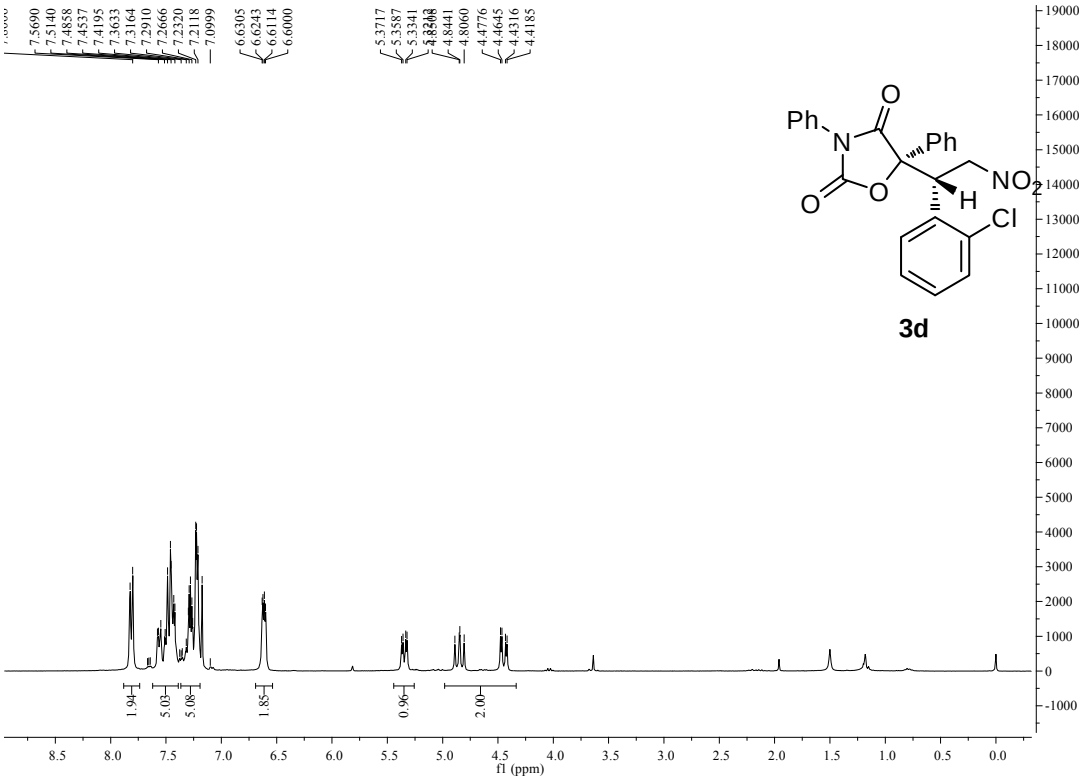
O
O
Ph
NO2
H
Cl
3c
f1 (ppm)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7.0999
6.6305
6.6243
6.6114
6.6000
5.3717
5.3587
5.3341
4.8441
4.8060
4.4776
4.4645
4.4316
4.4185
1.94
5.03
5.08
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0.96
2.00
f1 (ppm)

Ph
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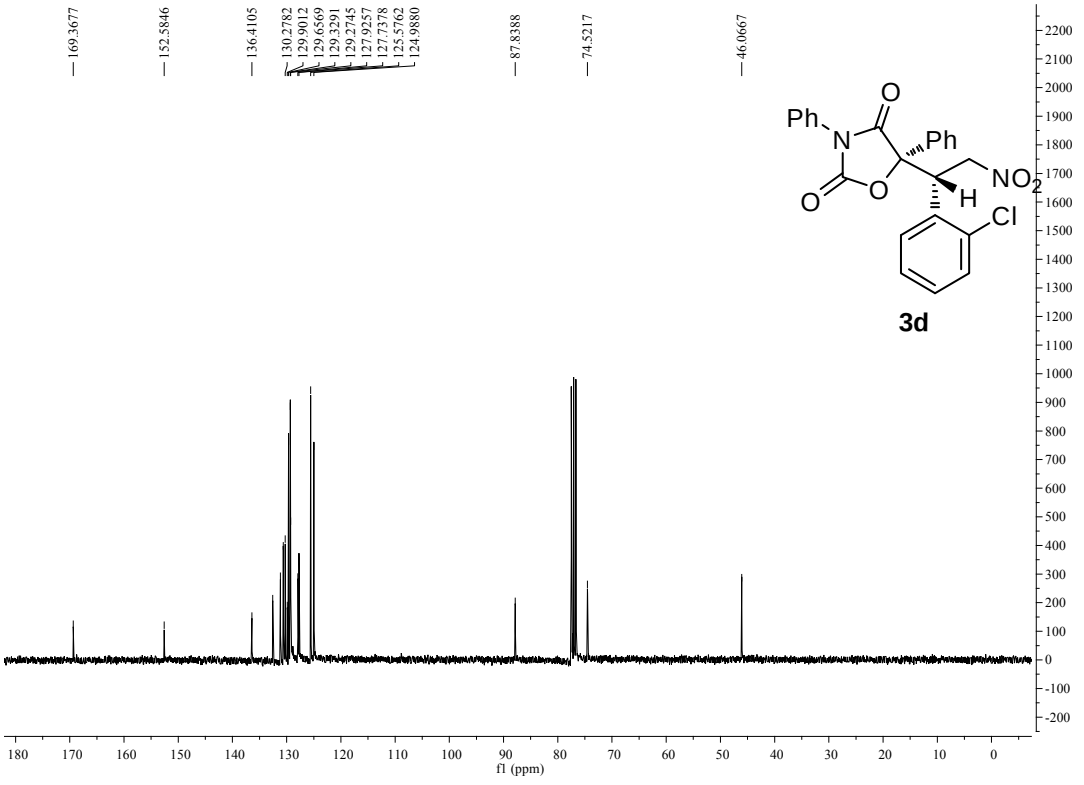
O
O
O
Ph
NO2
H
Cl

3d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125.5762
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f1 (ppm)

Ph
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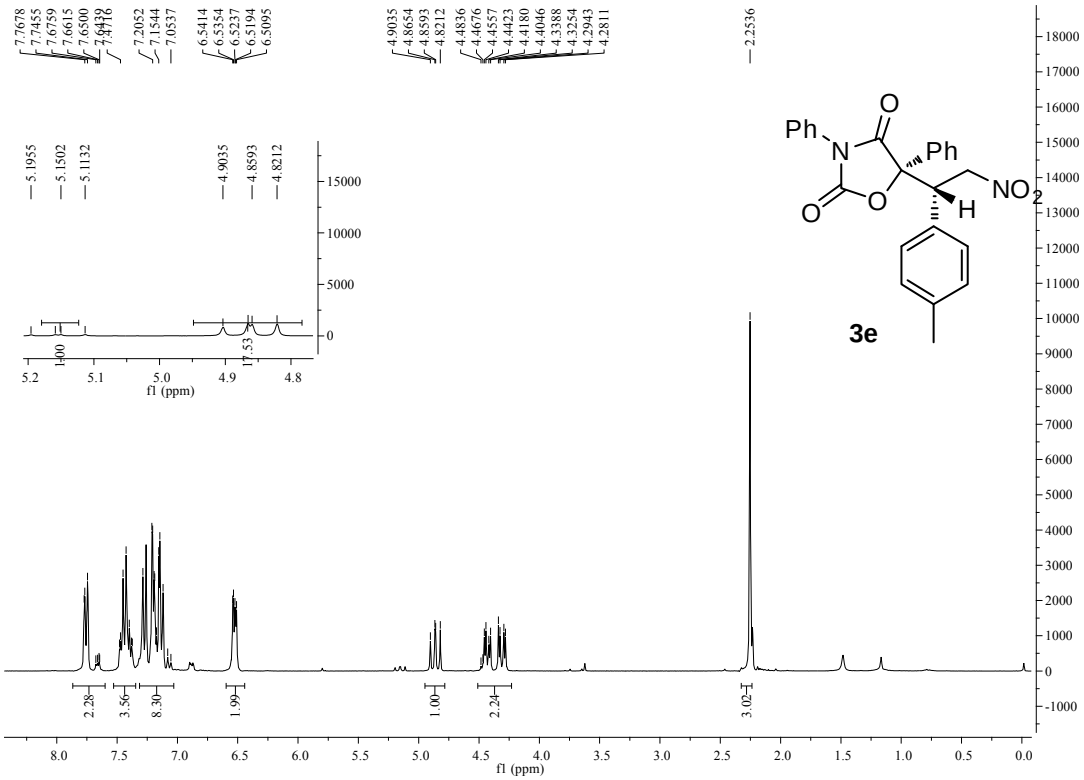
O
O
O
Ph
NO2
H
Cl

3d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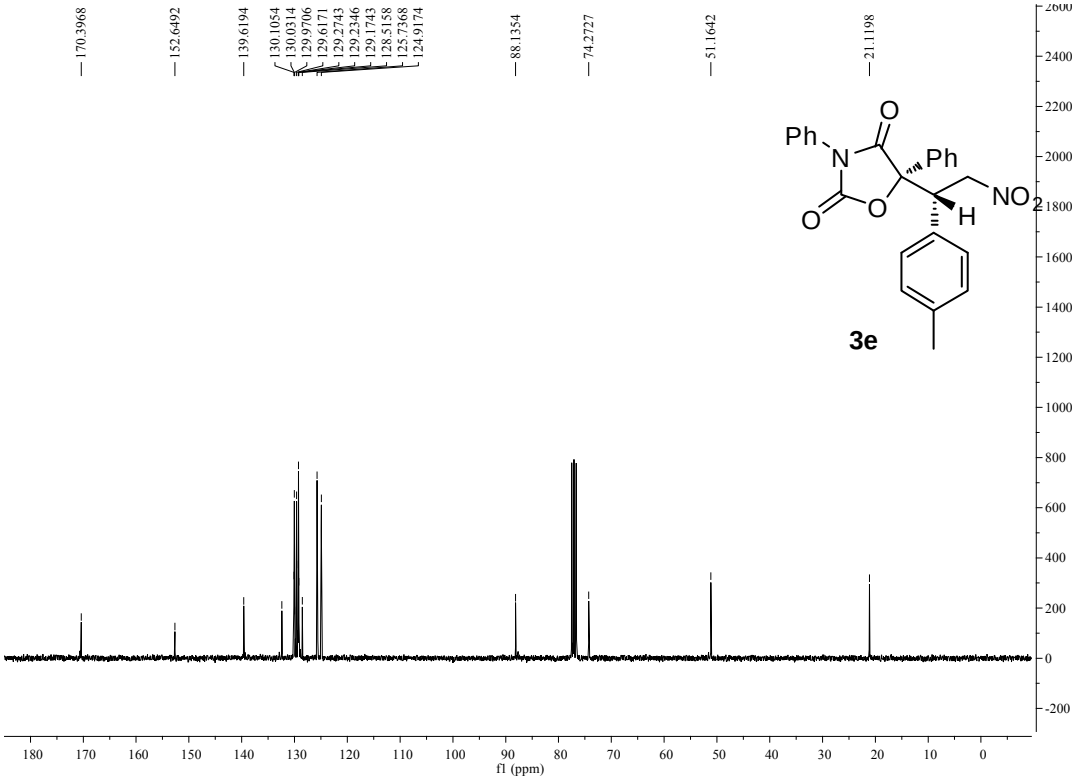
f1 (ppm)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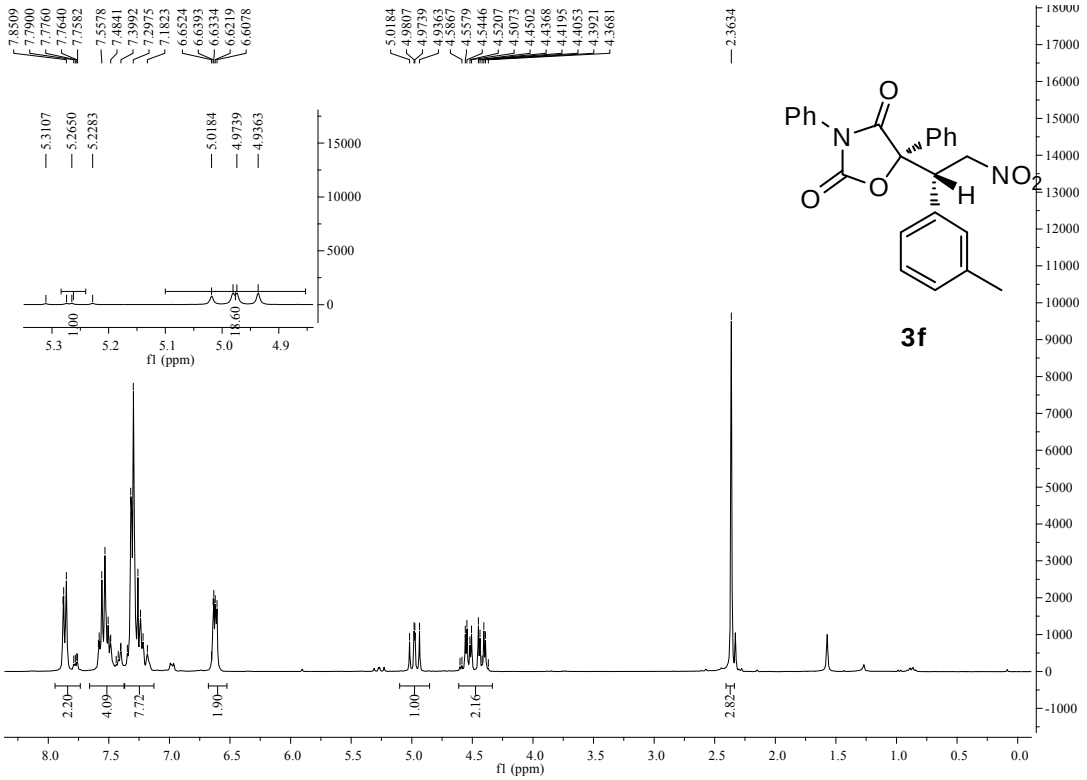
f1 (ppm)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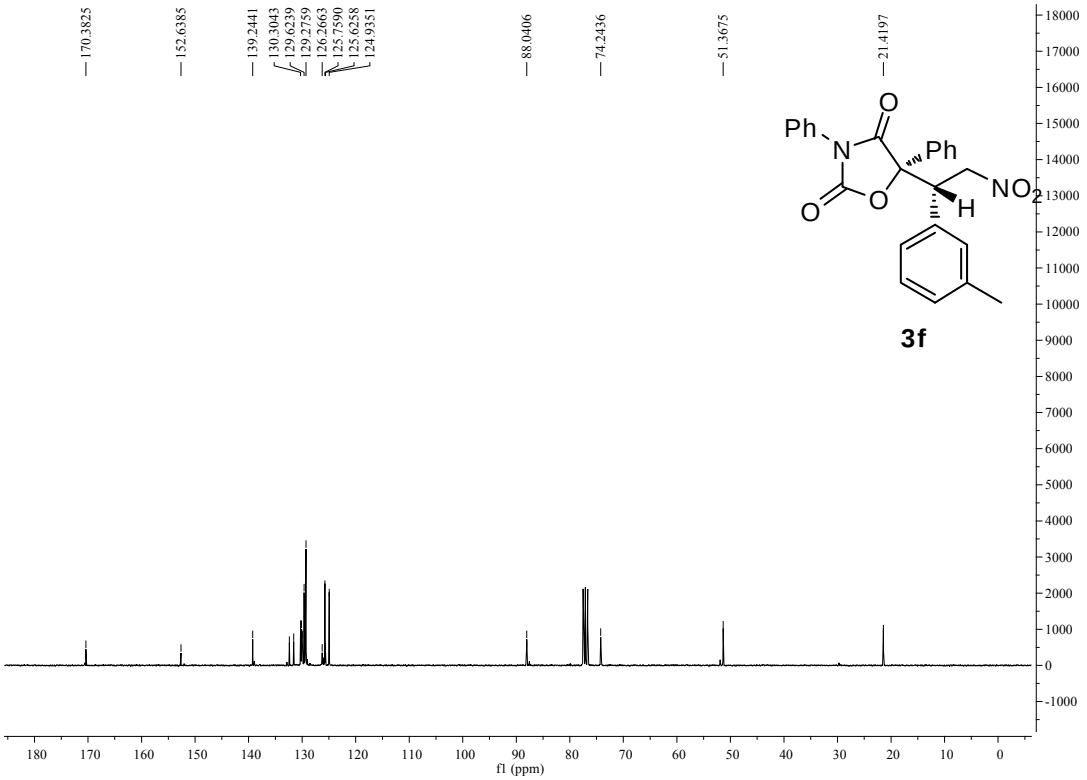
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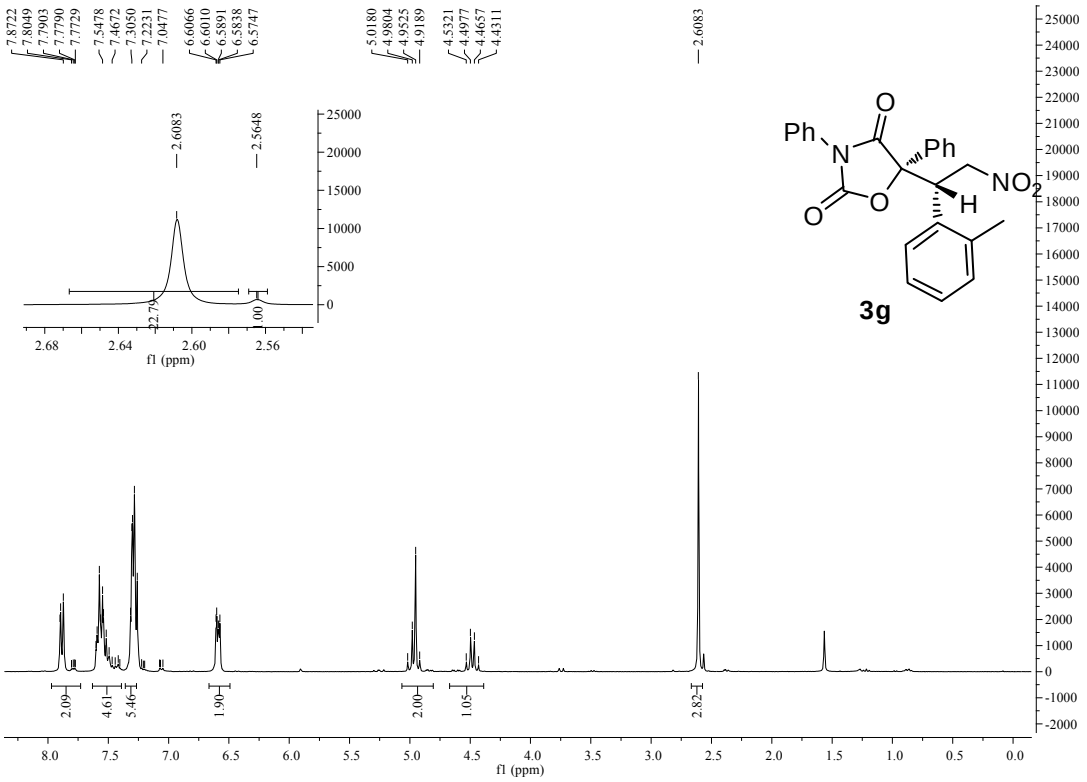
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6.6010
6.5891
6.5838
6.5747
5.0180
4.9804
4.9525
4.9189
4.5321
4.4977
4.4657
4.4311
2.6083
2.5648
22.79
1.00
f1 (ppm)
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O
O
Ph
NO2
H
3g
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5.46
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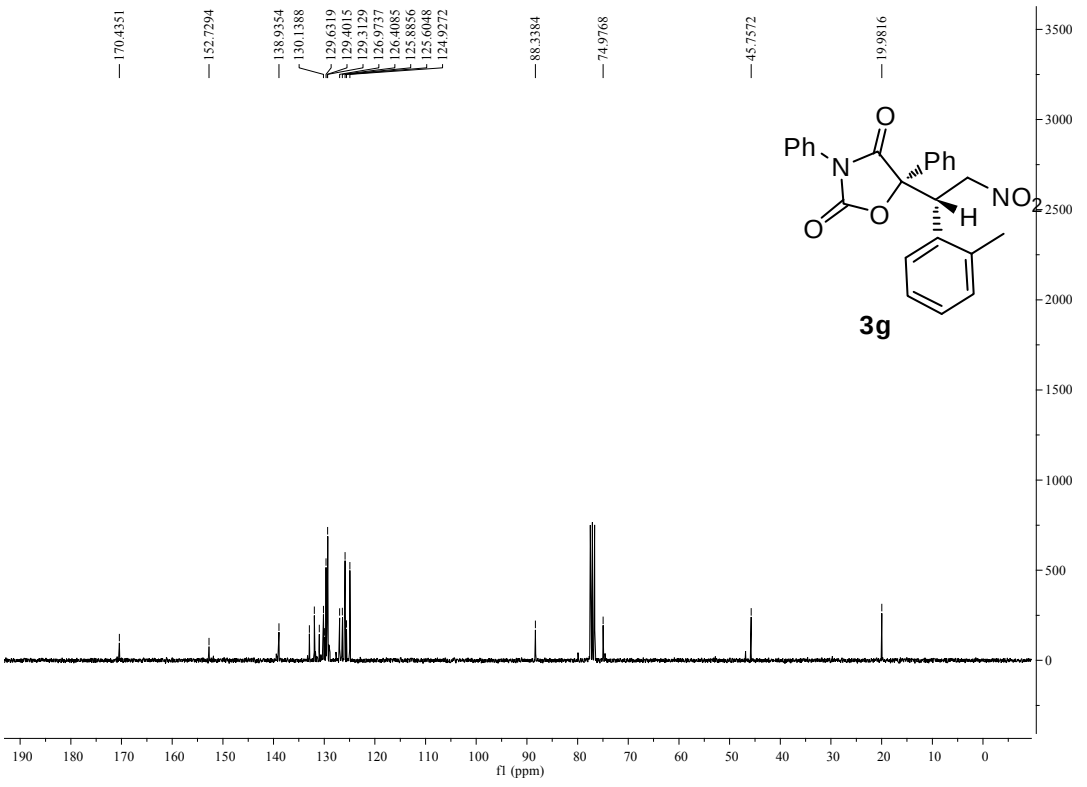
2.00
1.05
2.82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125.6048
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19.9816
Ph
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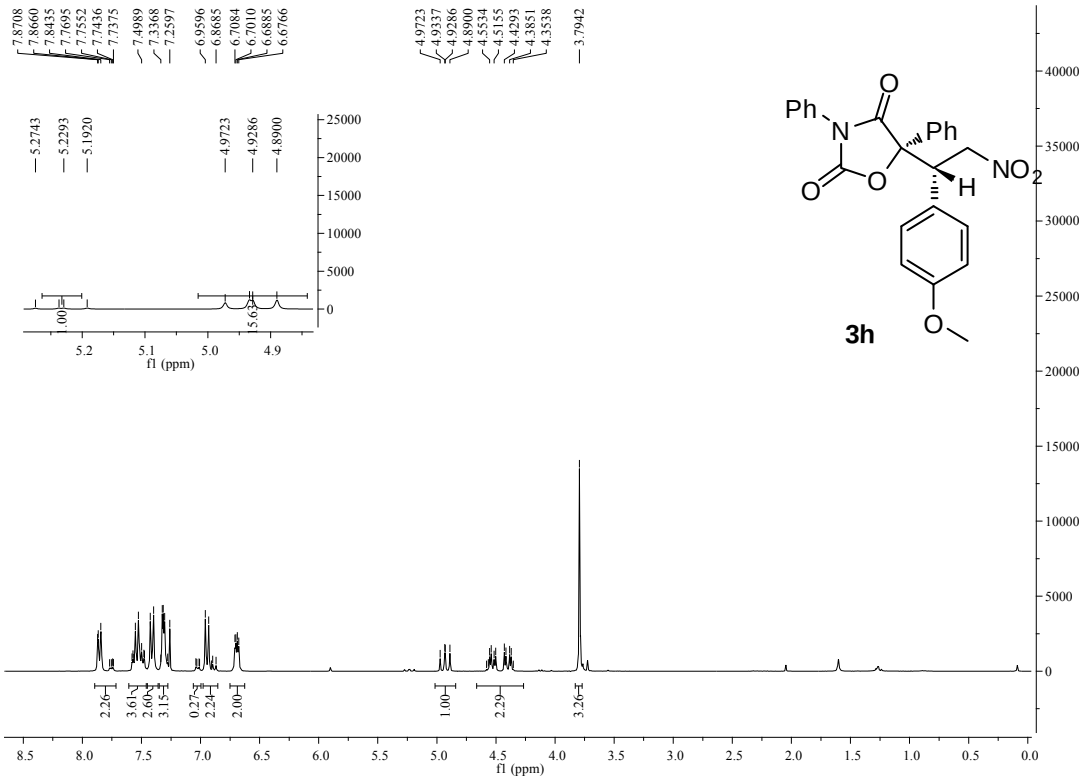
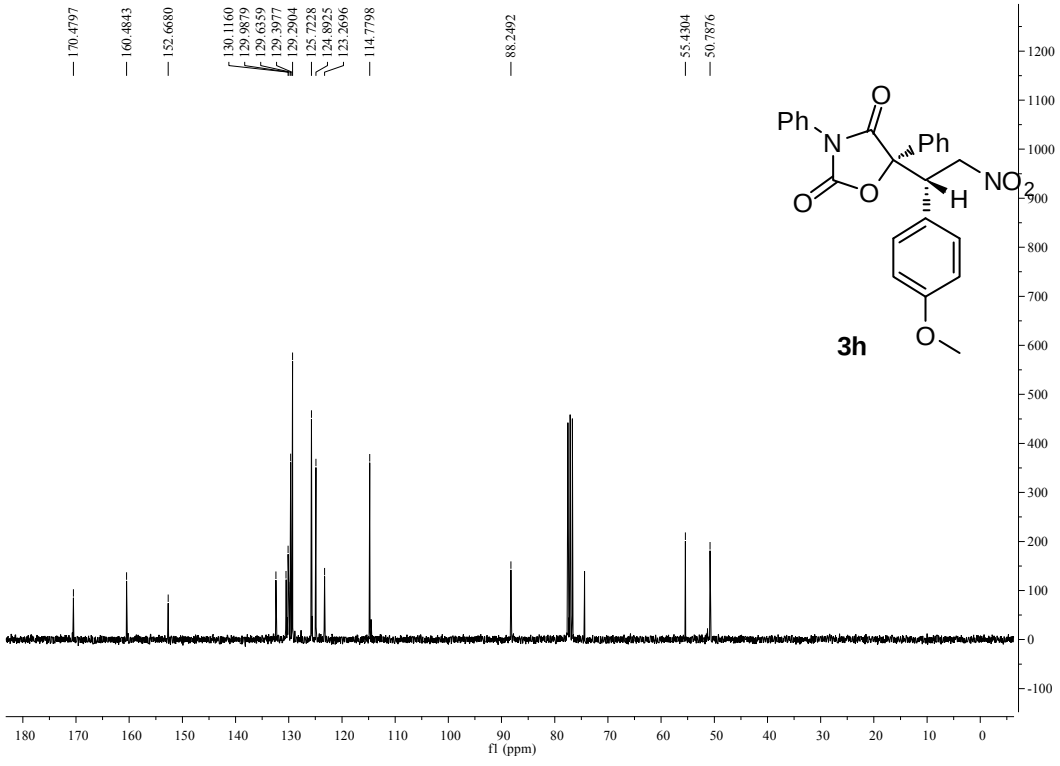
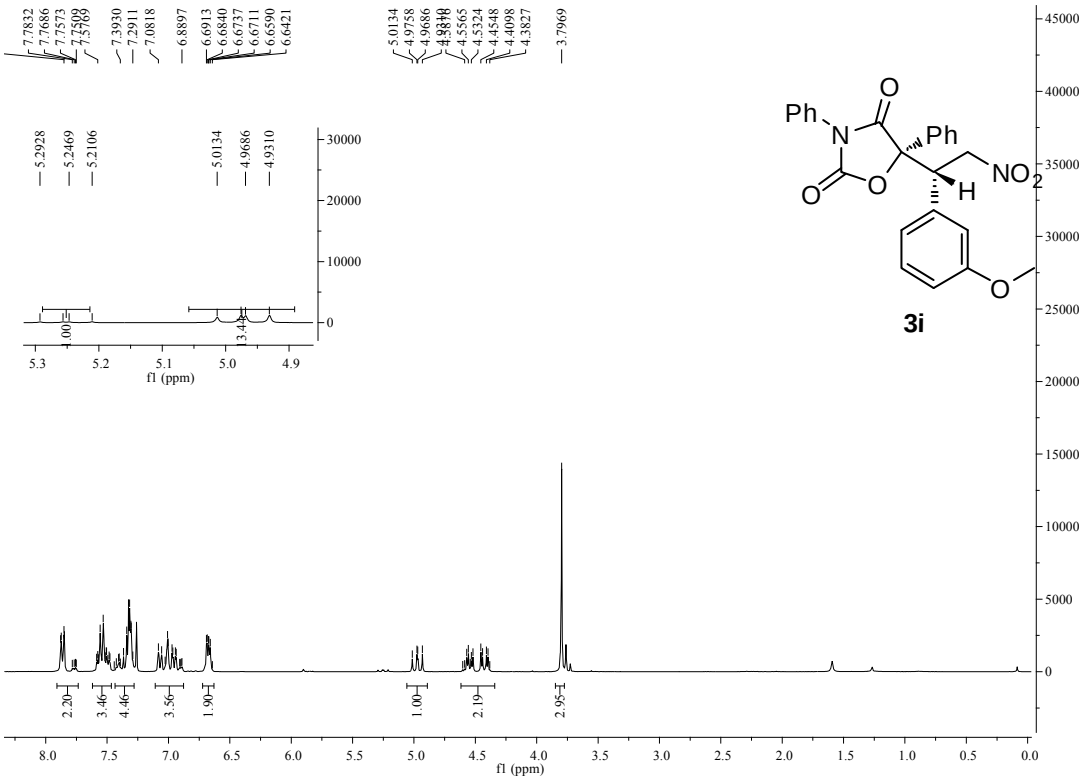
O
O
Ph
NO2
H
3g
f1 (ppm)


3h
f1 (ppm)


3h
f1 (ppm)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6.6913
6.6840
6.6737
6.6711
6.6590
6.6421
5.0134
4.9758
4.9686
4.5565
4.5324
4.4548
4.4098
4.3827
3.7969
5.2928
5.2469
5.2106
5.0134
4.9686
4.9310
1.00
3.44
f1 (ppm)
Ph
N
O
O
O
Ph
NO2
H
O
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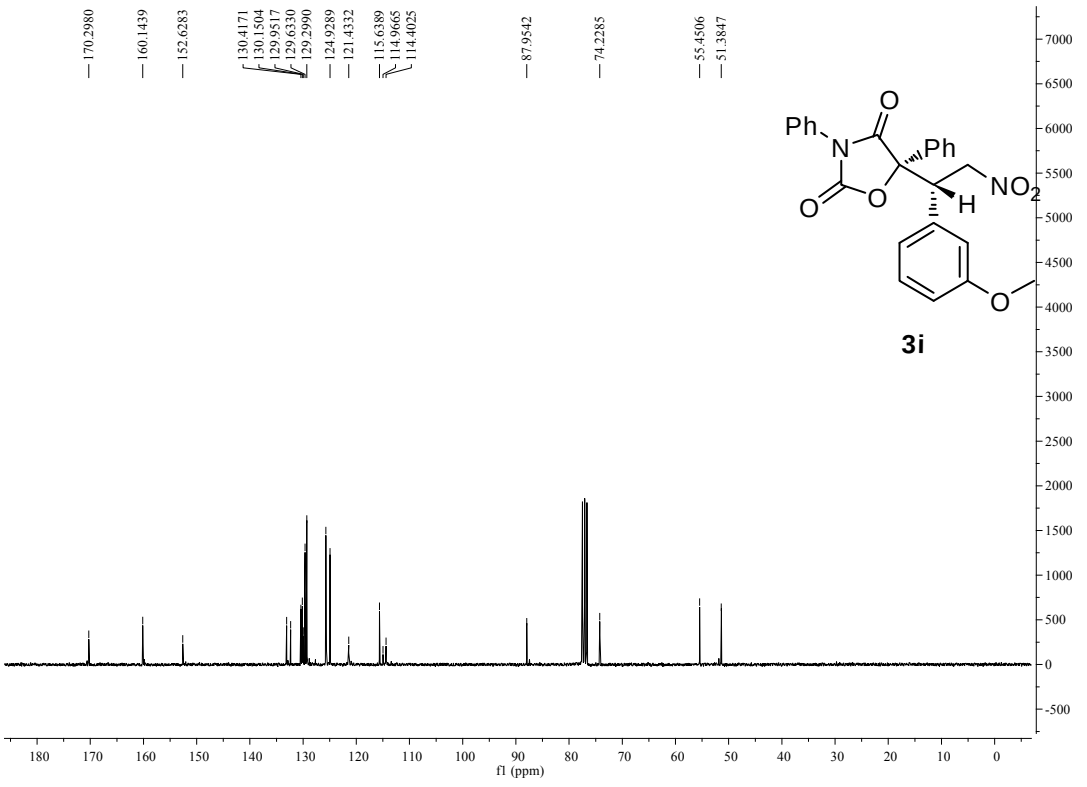
3.46
4.46
3.56
1.90
1.00
2.19
2.95
f1 (ppm)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130.1504
129.9517
129.6330
129.2990
124.9289
121.4332
115.6389
114.9665
114.4025
87.9542
74.2285
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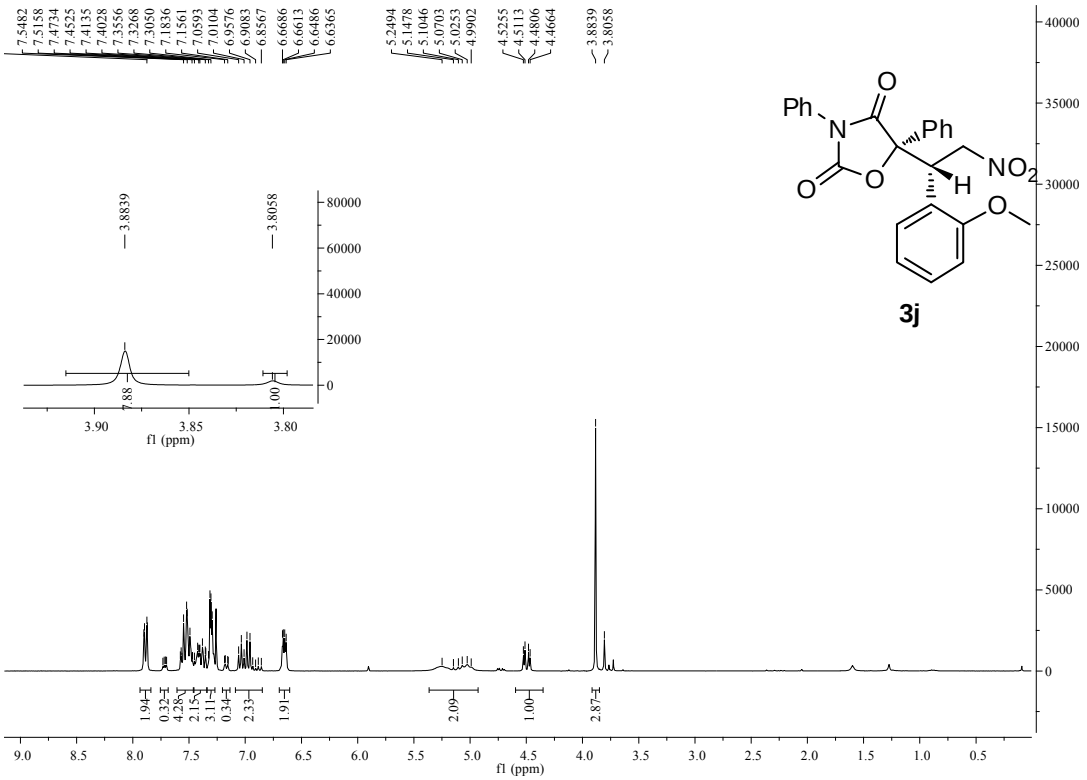
51.3847
Ph
N
O
O
O
Ph
NO2
H
O
3i
f1 (ppm)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7.1561
7.0593
7.0104
6.9576
6.9083
6.8567
6.6686
6.6613
6.6486
6.6365
5.2494
5.1478
5.1046
5.0703
5.0253
4.9902
4.5255
4.5113
4.4806
4.4664
3.8839
3.8058
Ph
N
O
O
O
Ph
NO2
H
O
3j
f1 (ppm)
1.94
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3.11
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2.09
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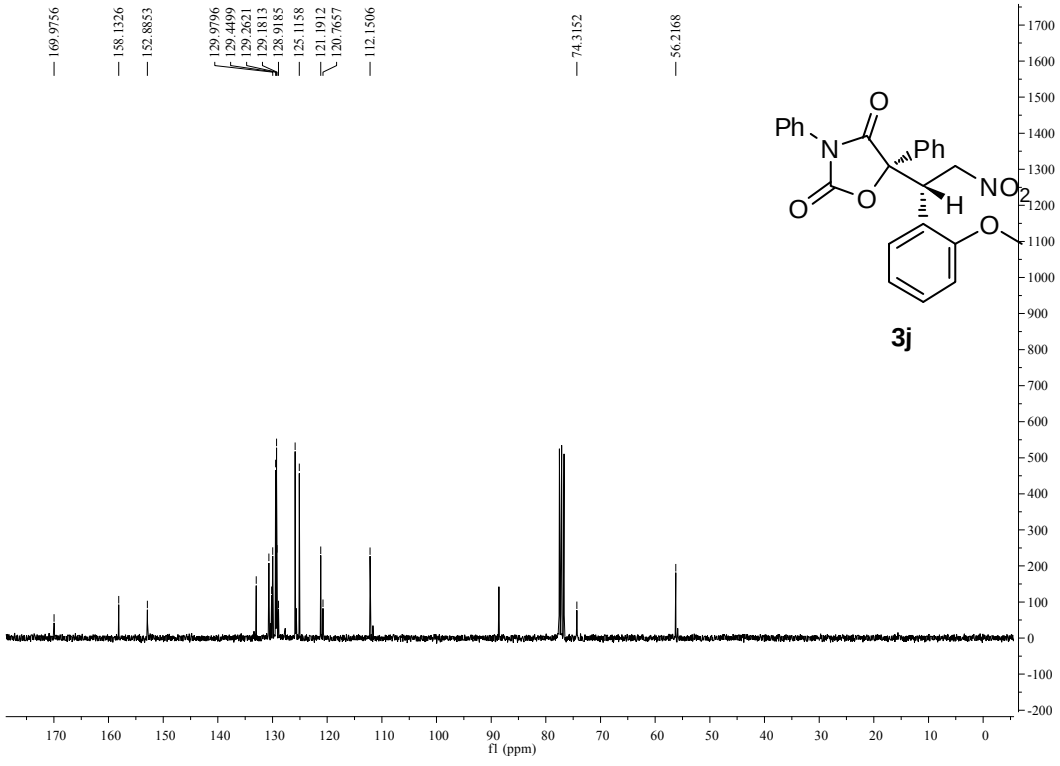
2.87
7.88
1.00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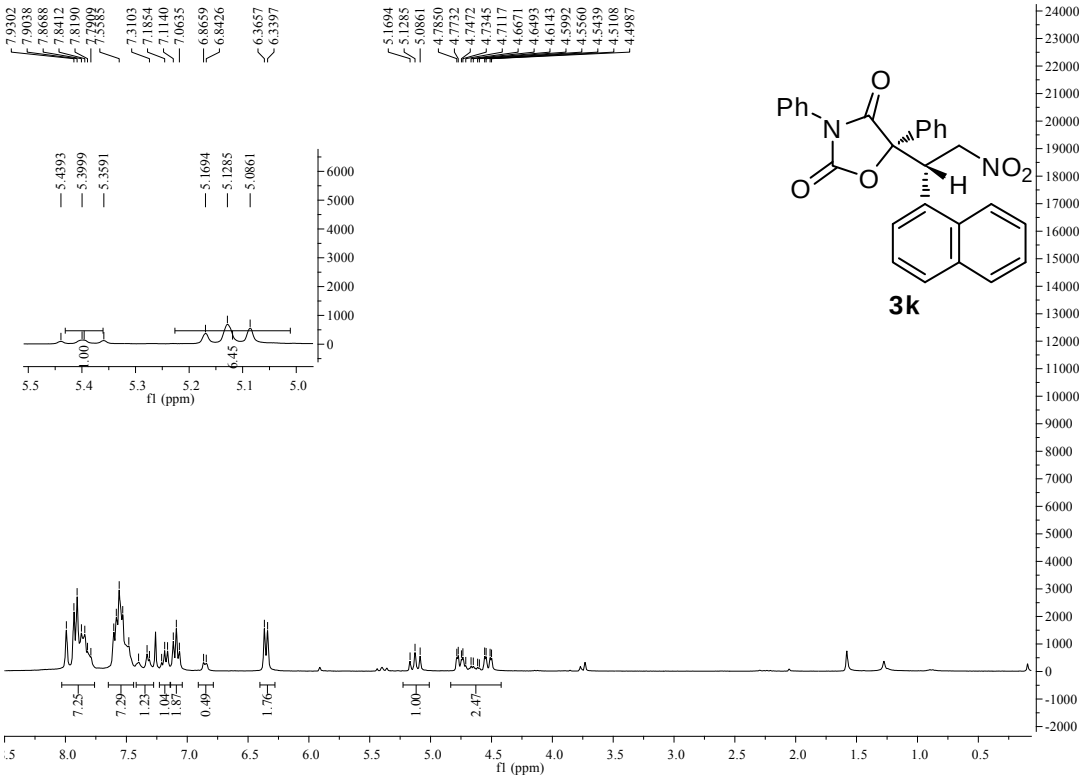
O
O
Ph
NO2
H
O
3j
f1 (ppm)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4.4987
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5.3999
5.3591
5.1694
5.1285
5.0861
1.00
6.45
f1 (ppm)
Ph
N
O
O
O
Ph
NO2
H
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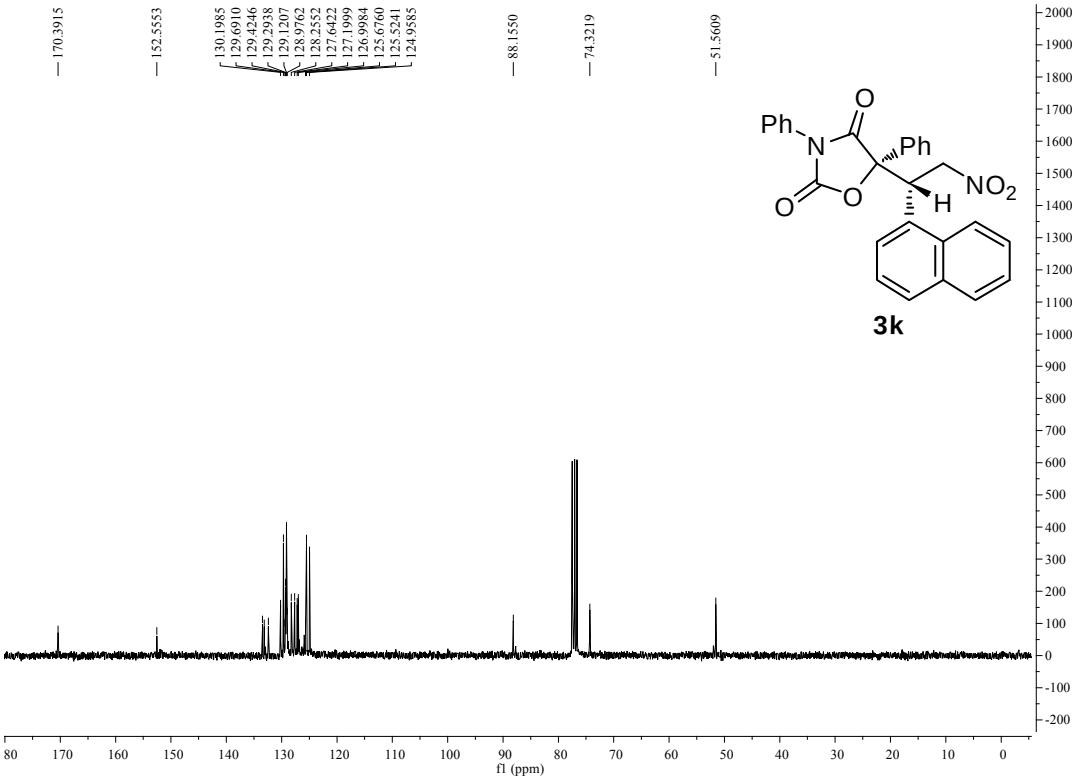
1.76
1.00
2.47
f1 (ppm)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126.9984
125.6760
125.5241
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74.3219
51.5609
Ph
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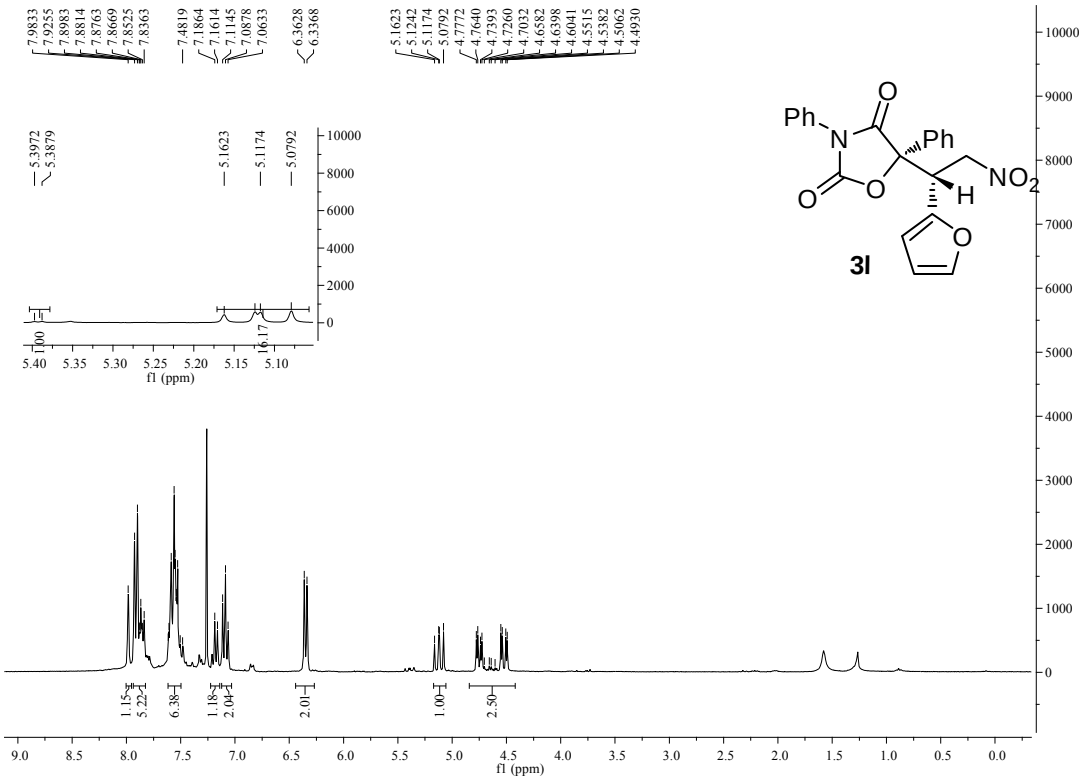
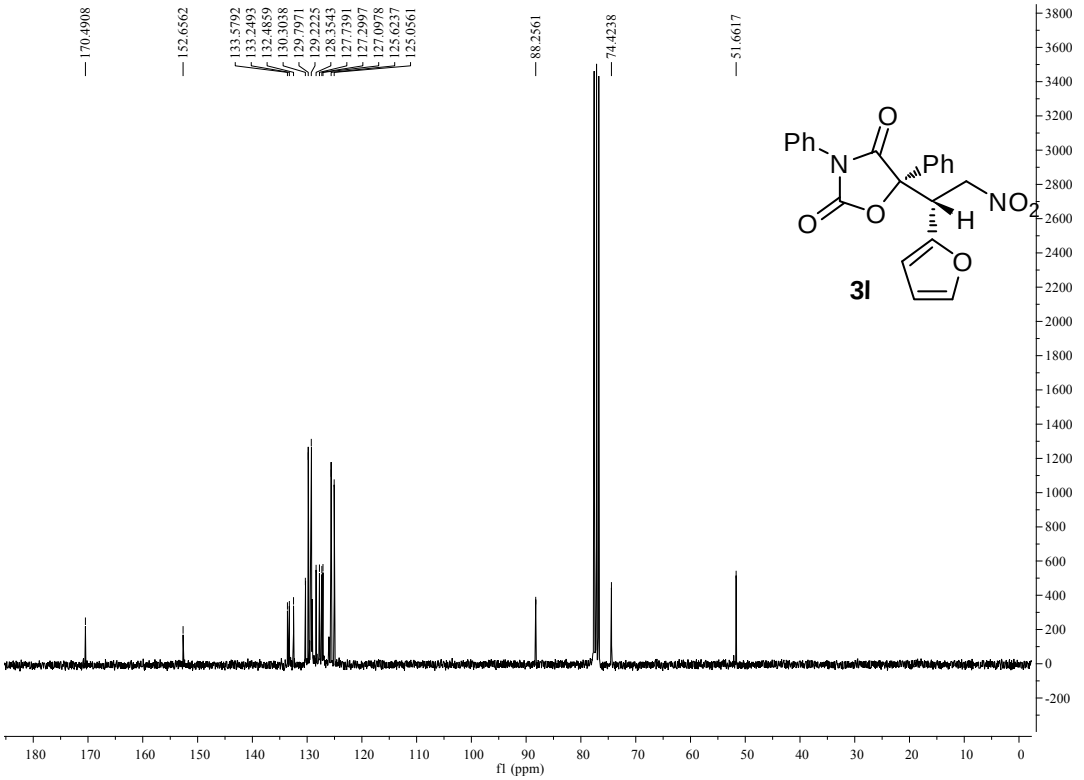
O
O
Ph
NO2
H
3k
f1 (ppm)


3l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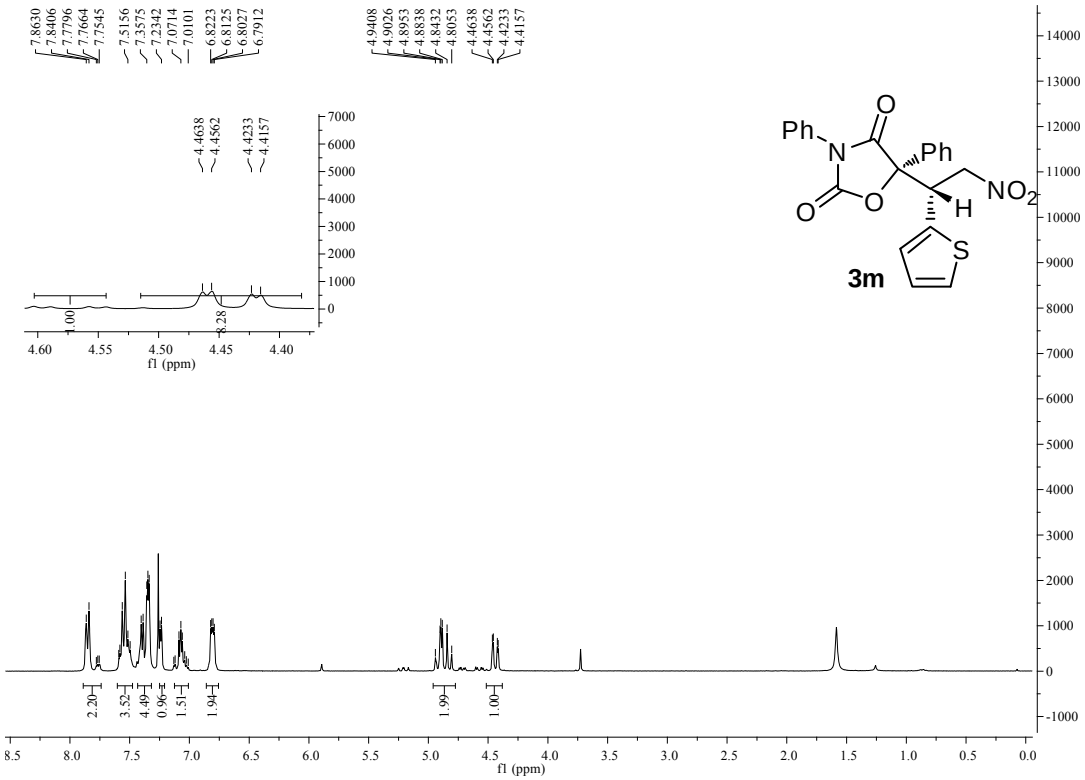
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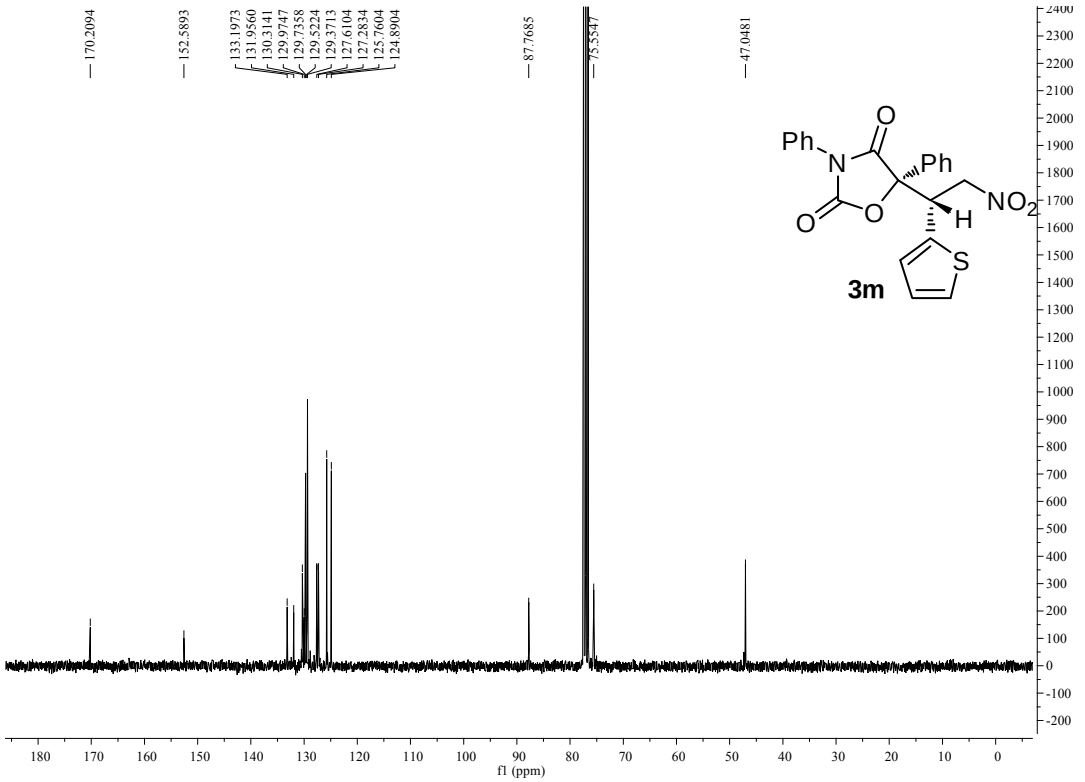
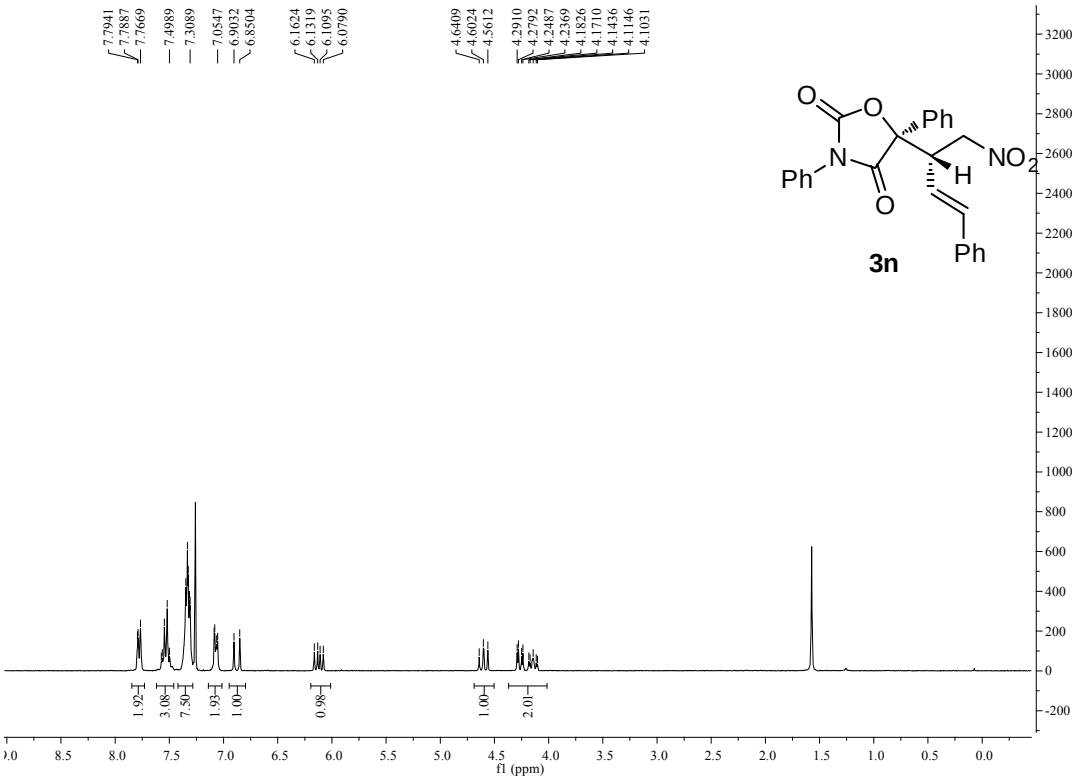
8.28
f1 (ppm)
Ph
N
O
O
Ph
O
NO2
H
S
3m
2.20
3.52
4.40
0.96
1.51
1.94
1.99
1.00
f1 (ppm)


170.2094
152.5893
133.1973
131.9560
130.3141
129.9747
129.7358
129.5224
129.3713
127.6104
127.2834
125.7604
124.8904
87.7685
75.5547
47.0481
Ph
N
O
O
Ph
O
NO2
H
S
3m
f1 (ppm)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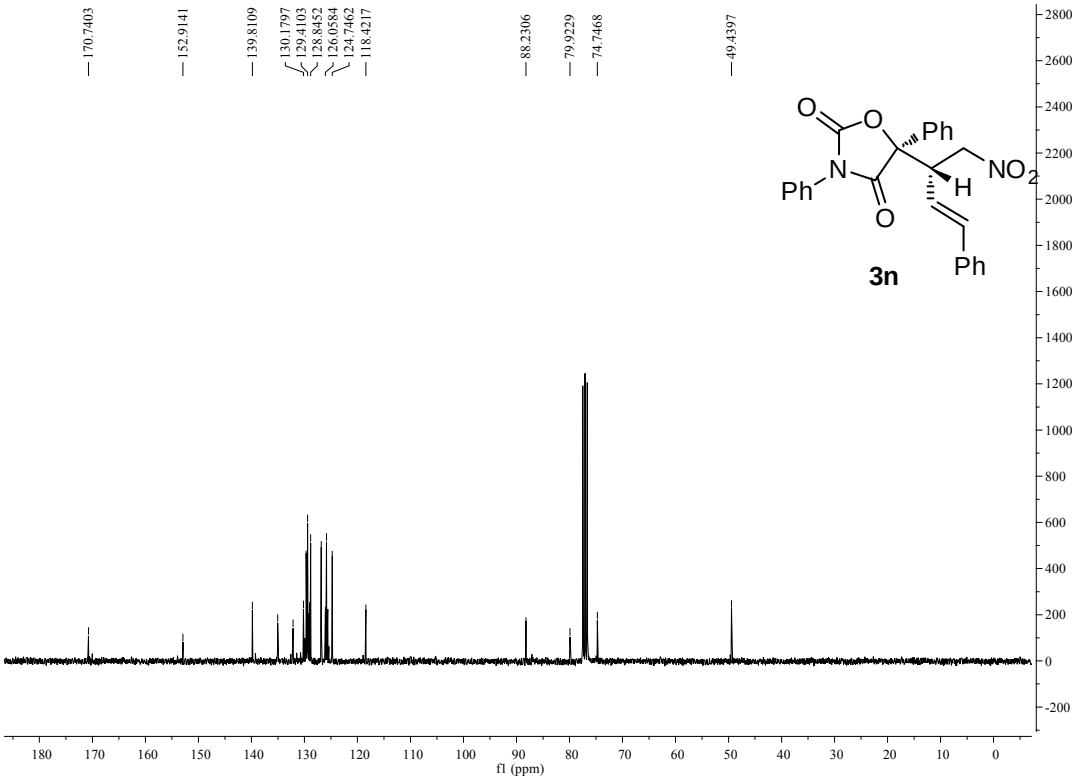
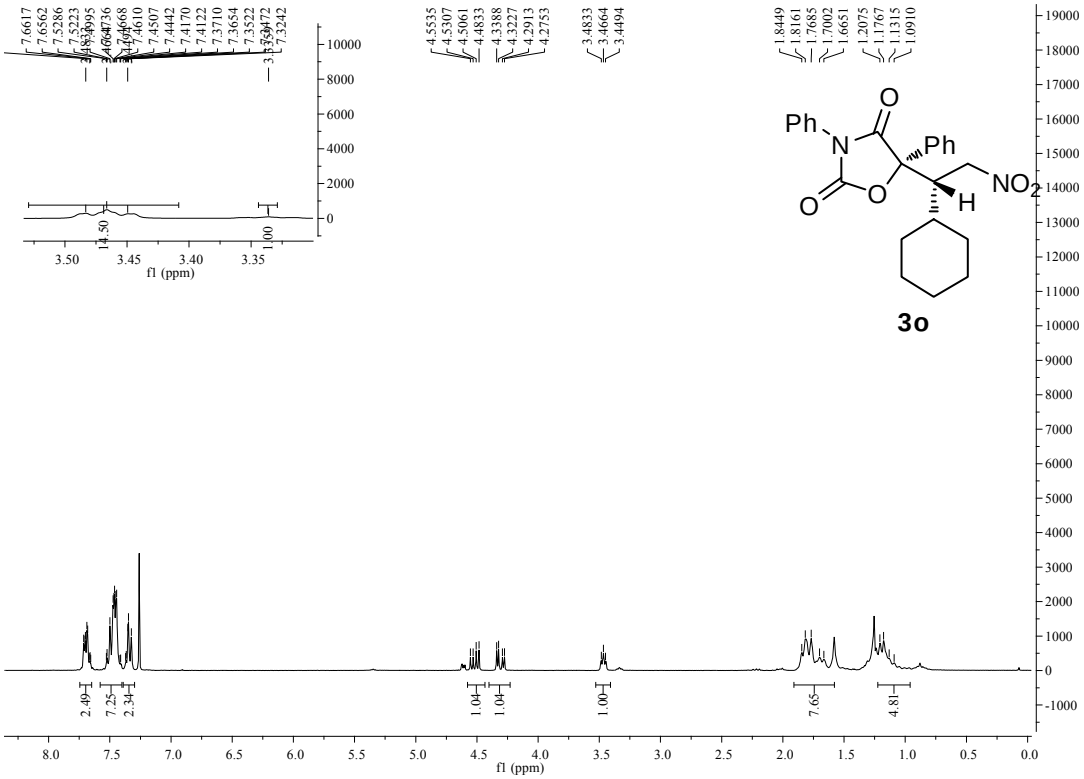
7.7887
7.7669
7.4989
7.3089
7.0547
6.9032
6.8504
6.1624
6.1319
6.1095
6.0790
4.6409
4.6024
4.5612
4.2910
4.2792
4.2487
4.2369
4.1826
4.1710
4.1436
4.1146
4.1031
1.92
3.08
7.50
1.93
1.00
0.98
1.00
2.01
f1 (ppm)
O
O
Ph
N
NO2
Ph
H
O
Ph
3n


170.7403
152.9141
139.8109
130.1797
129.4103
128.8452
126.0584
124.7462
118.4217
88.2306
79.9229
74.7468
49.4397
f1 (ppm)
O
O
Ph
N
NO2
Ph
H
O
Ph
3n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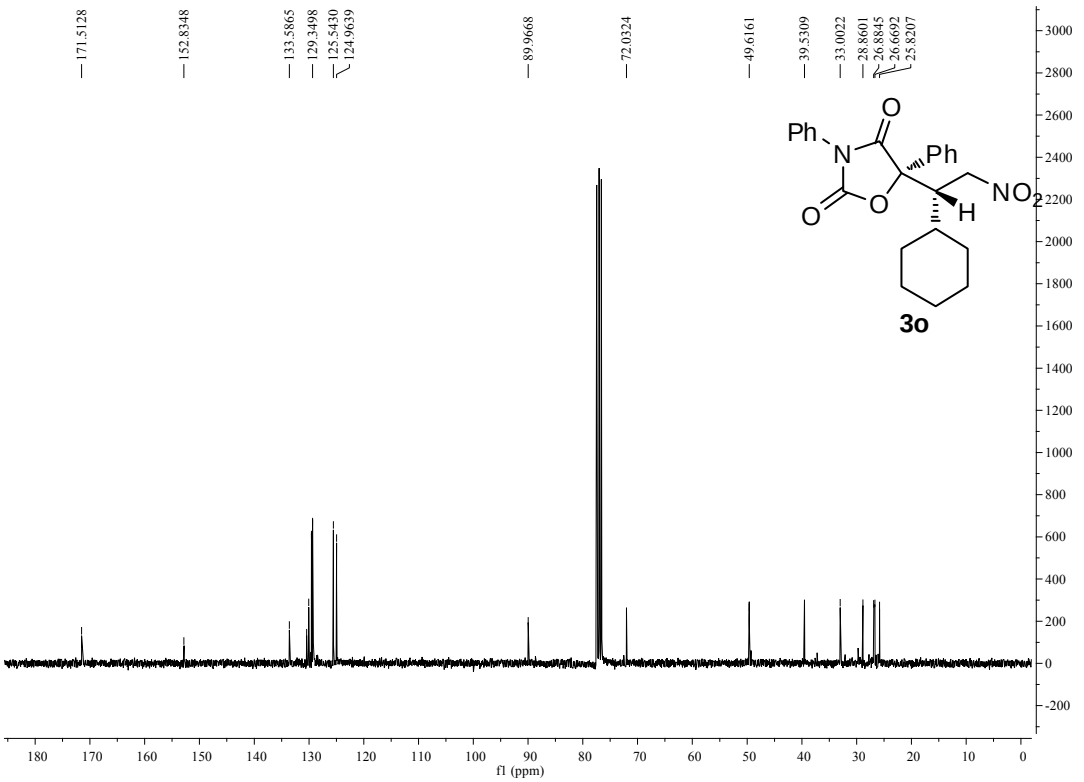
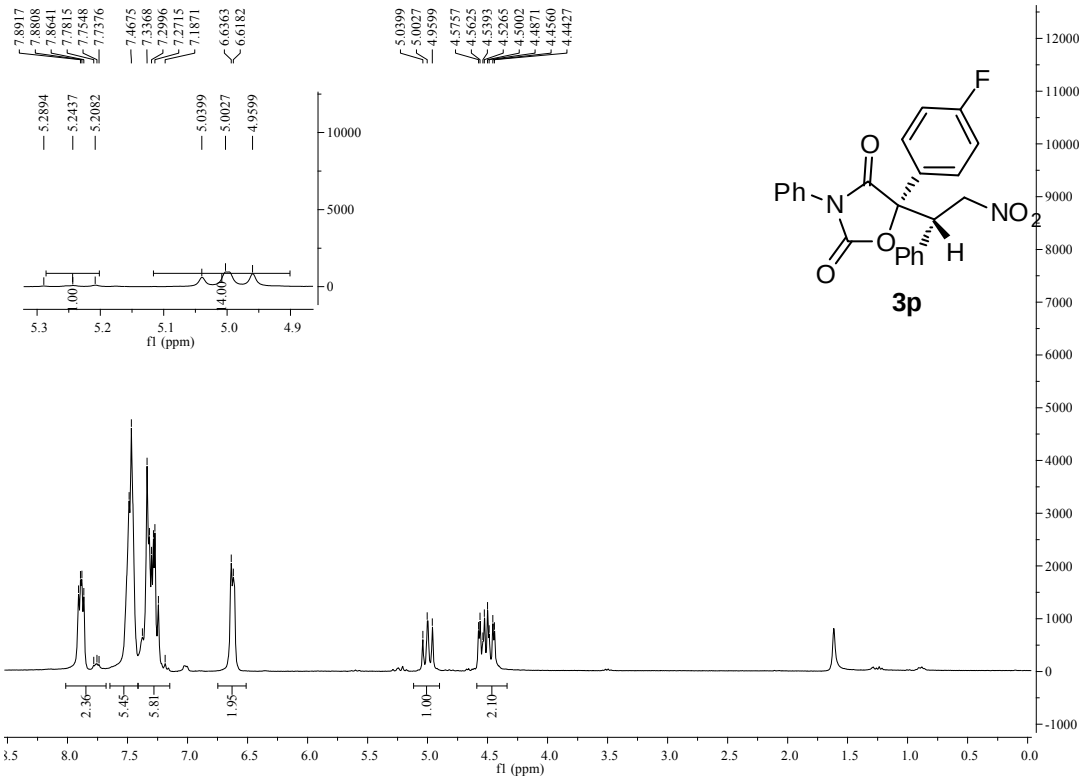
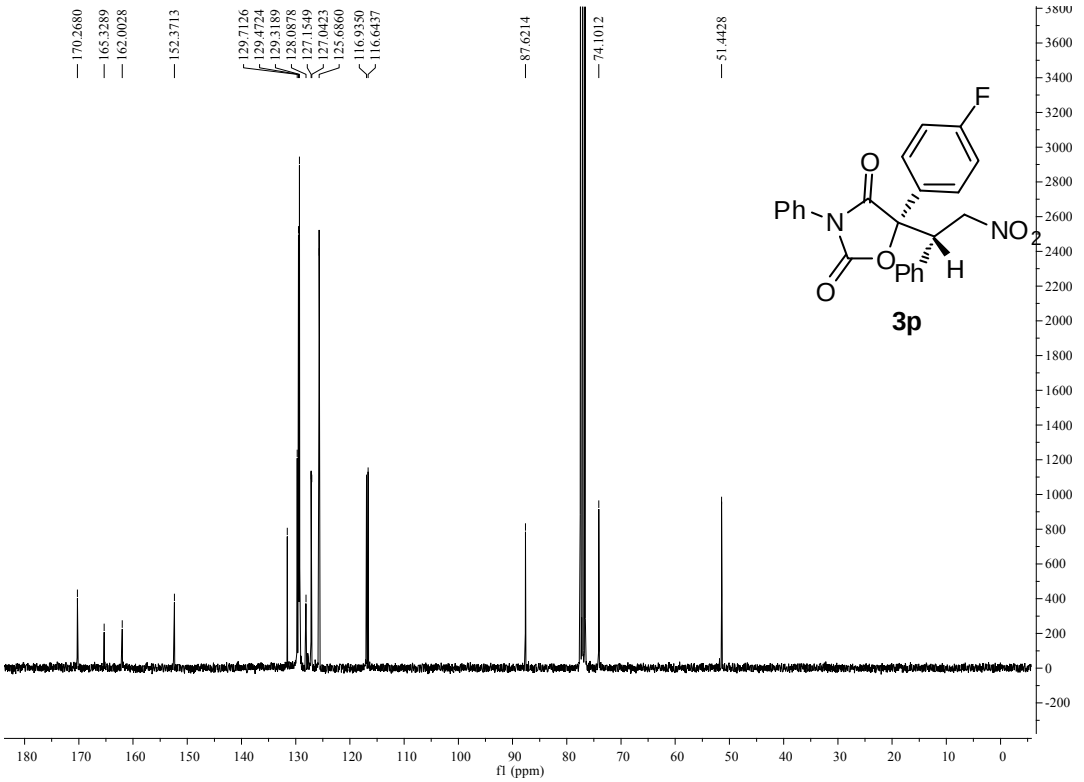
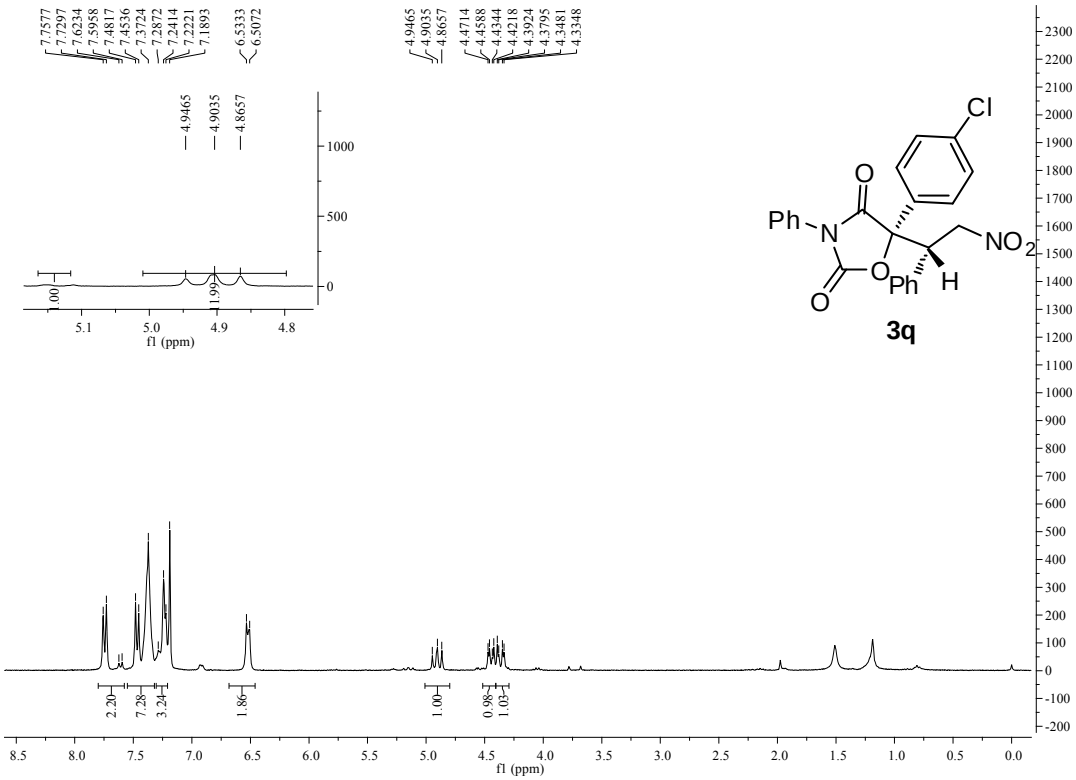
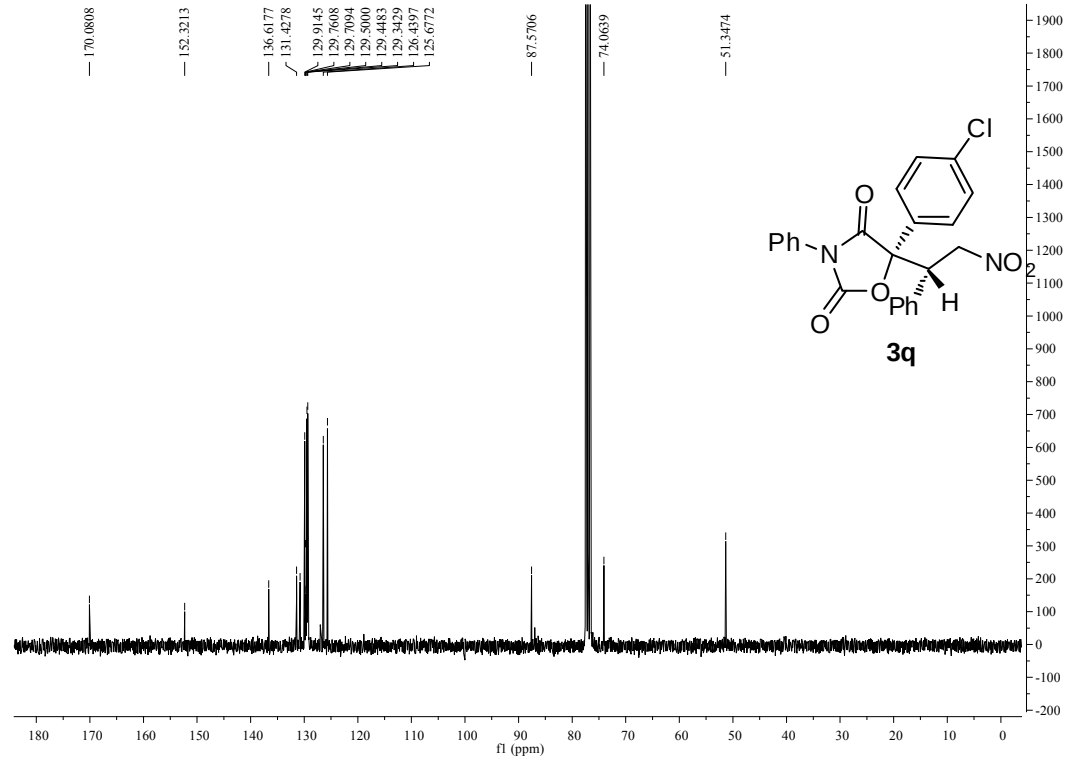
7.6562
7.5286
7.5223
7.4995
7.4736
7.4668
7.4610
7.4507
7.4442
7.4170
7.4122
7.3710
7.3654
7.3522
7.3472
7.3242
4.5535
4.5307
4.5061
4.4833
4.3388
4.3227
4.2913
4.2753
3.4833
3.4664
3.4494
1.8449
1.8161
1.7685
1.7002
1.6651
1.2075
1.1767
1.1315
1.0910
Ph
N
O
O
O
Ph
NO2
H
3o
14.50
1.00
3.50
3.45
3.40
3.35
f1 (ppm)
2.49
7.25
2.34
1.04
1.04
1.00
7.65
4.81
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

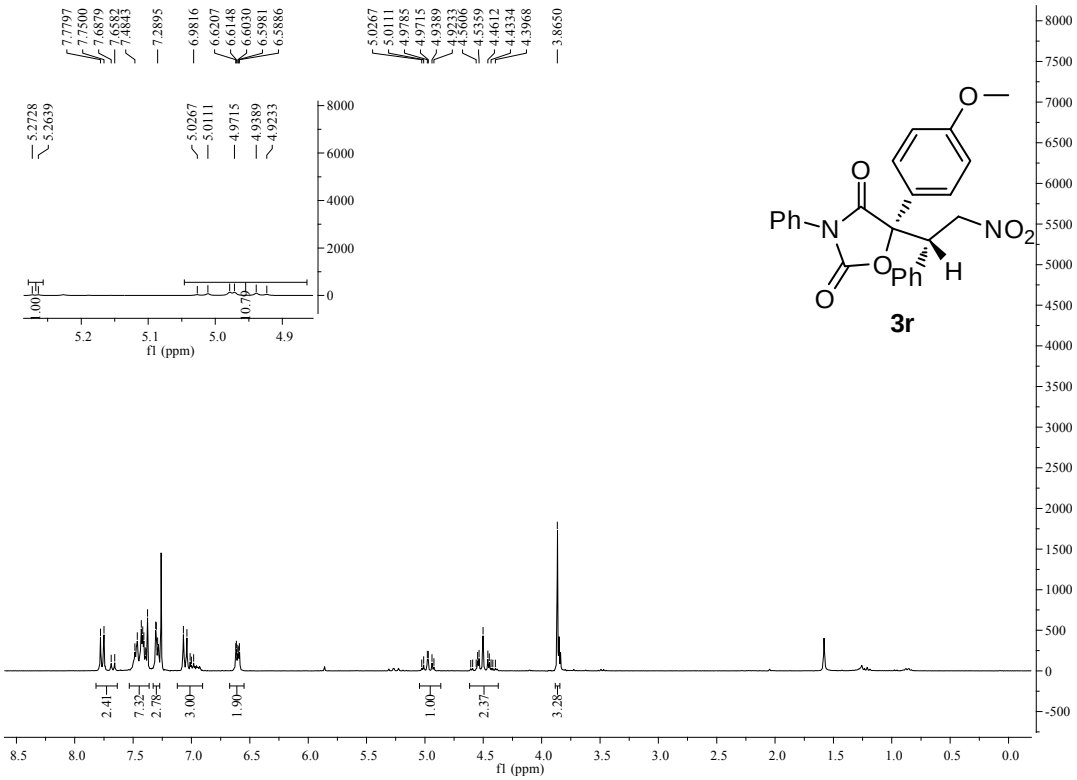

171.5128
152.8348
133.5865
129.3498
125.5430
124.9639
89.9668
72.0324
49.6161
39.5309
33.0022
28.8601
26.8845
26.6692
25.8207
Ph
N
O
O
O
Ph
NO2
H
3o
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

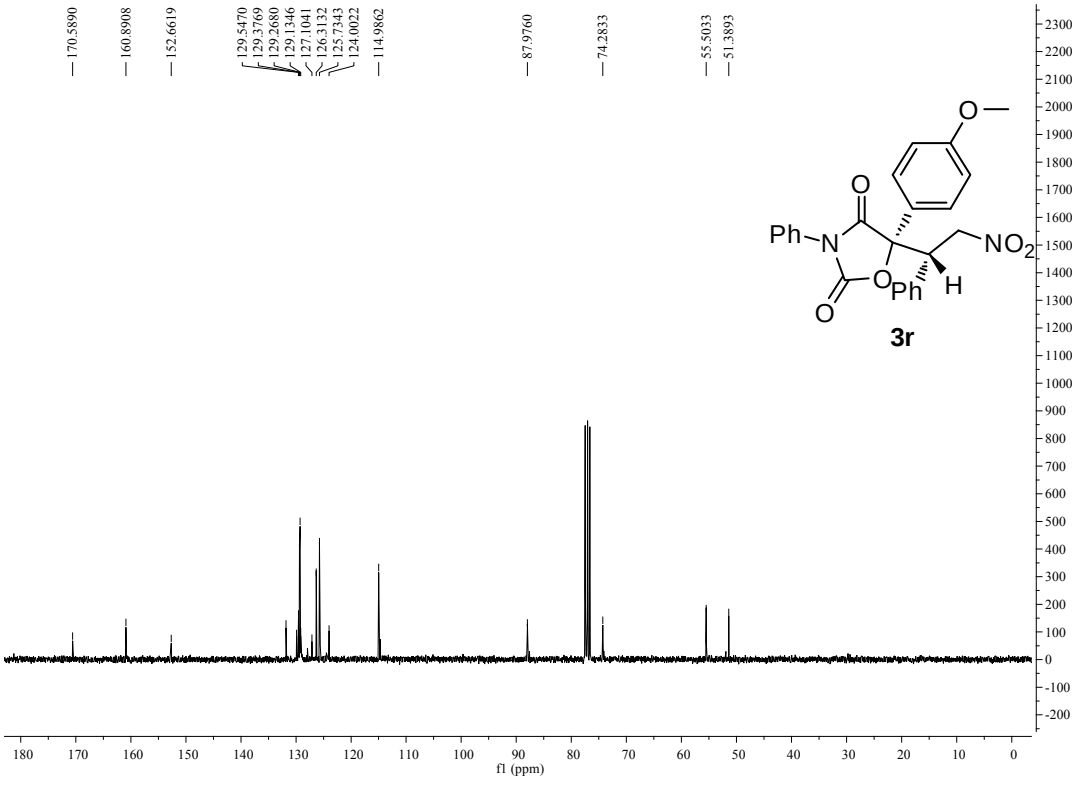

7.8917
7.8808
7.8641
7.7815
7.7548
7.7376
7.4675
7.3368
7.2996
7.2715
7.1871
6.6363
6.6182
5.0399
5.0027
4.9599
4.5757
4.5625
4.5393
4.5265
4.5002
4.4871
4.4560
4.4427
5.2894
5.2437
5.2082
5.0399
5.0027
4.9599
1.00
4.00
fl (ppm)
2.36
5.45
5.81
1.95
1.00
2.10
fl (ppm)
F
O
Ph
N
O
O
Ph
H
NO2
3p


170.2680
165.3289
162.0028
152.3713
129.7126
129.4724
129.3189
128.0878
127.1549
127.0423
125.6860
116.9350
116.6437
87.6214
74.1012
51.4428
fl (ppm)
F
O
Ph
N
O
O
Ph
H
NO2
3p


7.7577
7.7297
7.6234
7.5958
7.4817
7.4536
7.3724
7.2872
7.2414
7.2221
7.1893
6.5333
6.5072
4.9465
4.9035
4.8657
4.4714
4.4588
4.4344
4.4218
4.3924
4.3795
4.3481
4.3348
Cl
O
Ph
N
NO2
O
O
Ph
H
3q
fl (ppm)


170.0808
152.3213
136.6177
131.4278
129.9145
129.7608
129.7094
129.5000
129.4483
129.3429
126.4397
125.6772
87.5706
74.0639
51.3474
Cl
O
Ph
N
NO2
O
O
Ph
H
3q
fl (ppm)


7.7797
7.7500
7.6879
7.6582
7.4843
7.2895
6.9816
6.6207
6.6148
6.6030
6.5981
6.5886
5.0267
5.0111
4.9785
4.9715
4.9389
4.9233
4.5606
4.5359
4.4612
4.4334
4.3968
3.8650
5.2728
5.2639
1.00
10.70
f1 (ppm)
2.41
7.32
2.78
3.00
1.90
1.00
2.37
3.28
f1 (ppm)
Ph
N
O
O
O
Ph
H
NO2
O
3r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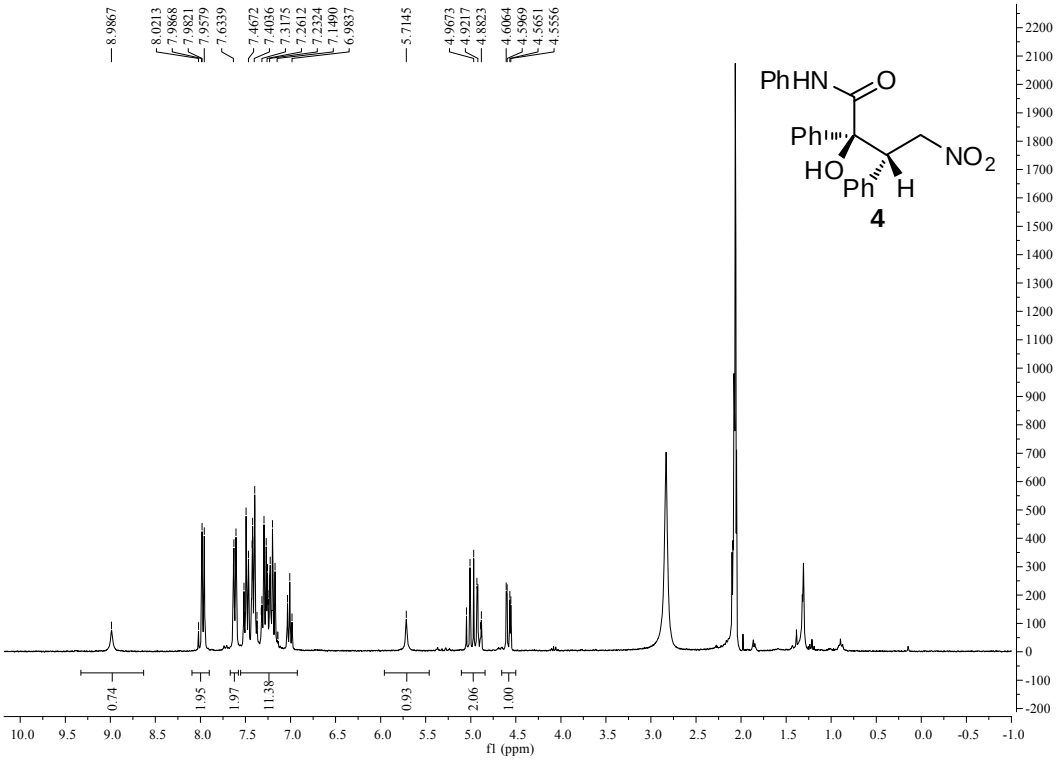
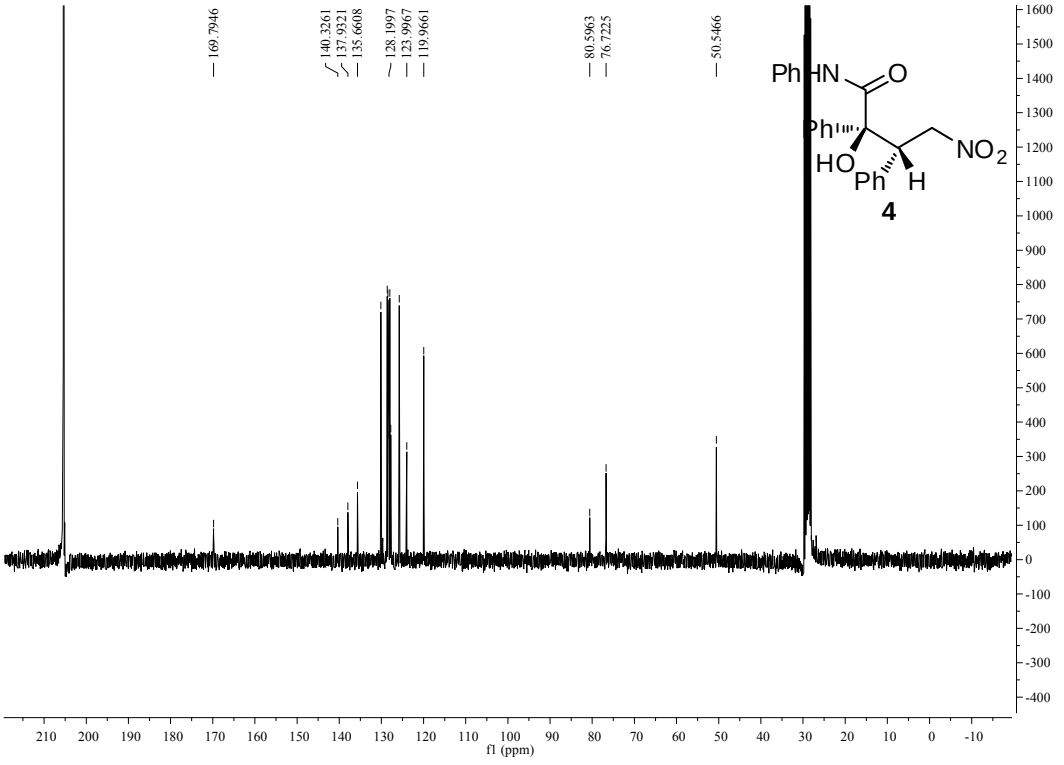
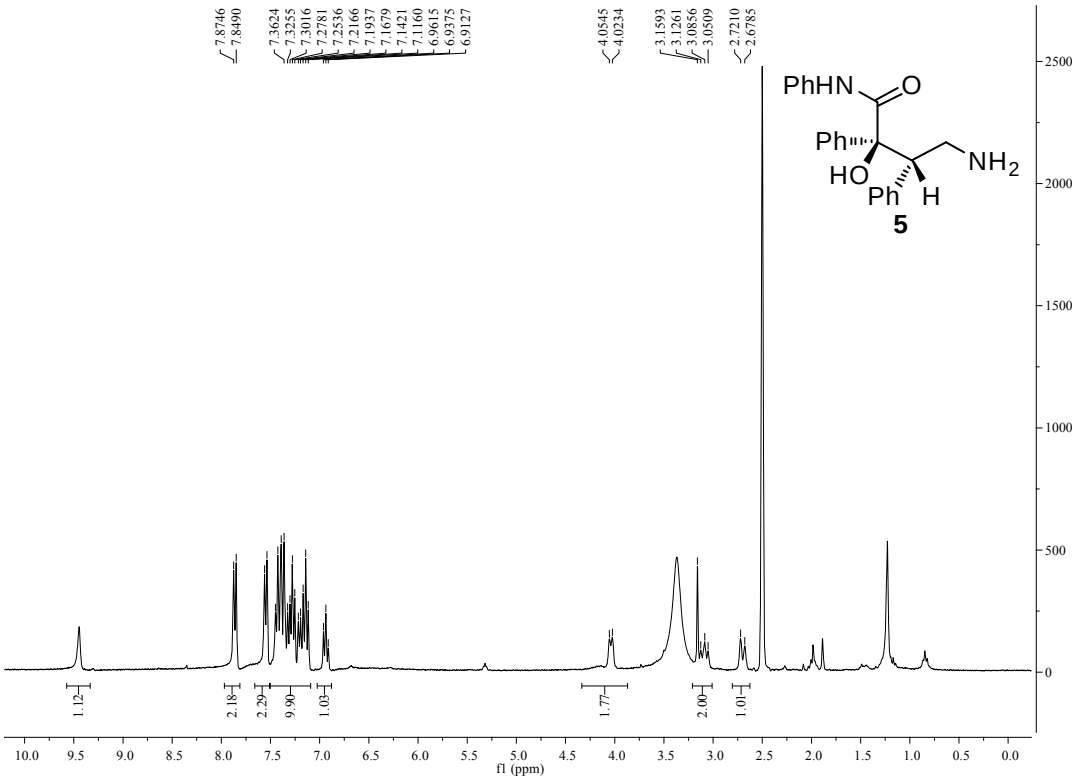
160.8908
152.6619
129.5470
129.3769
129.2680
129.1346
127.1041
126.3132
125.7343
124.0022
114.9862
87.9760
74.2833
55.5033
51.3893
f1 (ppm)
Ph
N
O
O
O
Ph
H
NO2
O
3r


8.9867
8.0213
7.9868
7.9821
7.9579
7.6339
7.4672
7.4036
7.3175
7.2612
7.2324
7.1490
6.9837
5.7145
4.9673
4.9217
4.8823
4.6064
4.5969
4.5651
4.5556
PhHN
O
Ph
HO
Ph
H
NO2
4
0.74
1.95
1.97
11.38
0.93
2.06
1.00
f1 (ppm)


169.7946
140.3261
137.9321
135.6608
128.1997
123.9967
119.9661
80.5963
76.7225
50.5466
PhHN
O
Ph
HO
Ph
H
NO2
4
f1 (ppm)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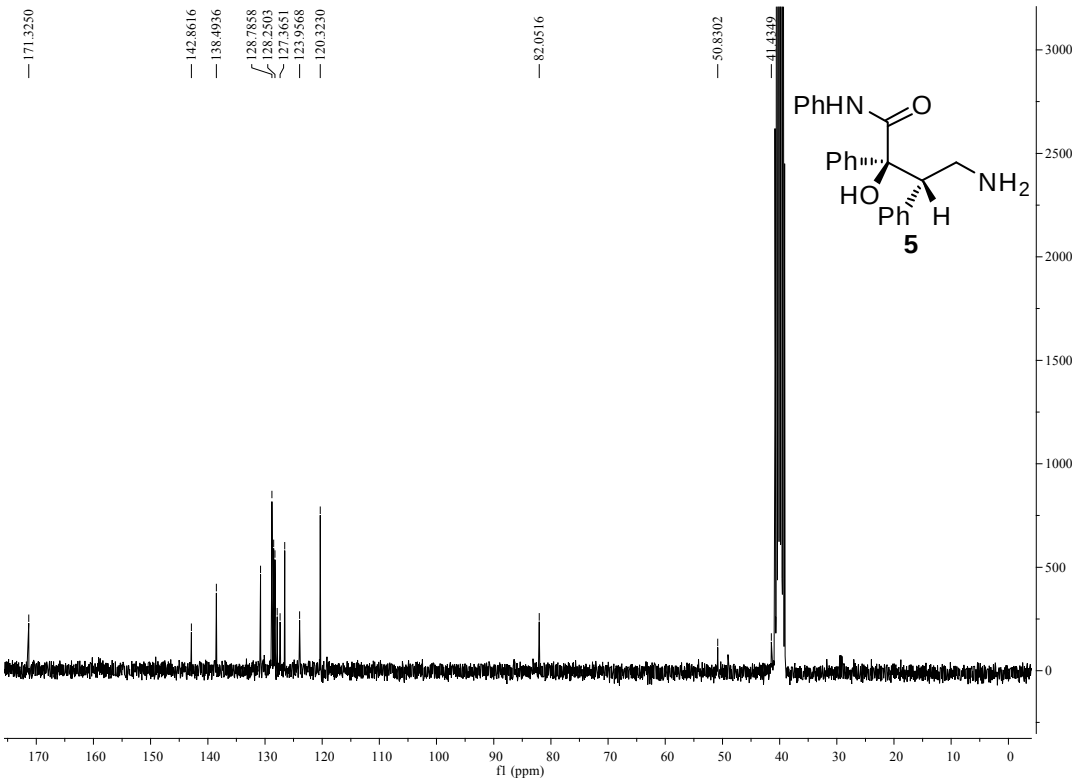
7.8490
7.3624
7.3255
7.3016
7.2781
7.2536
7.2166
7.1937
7.1679
7.1421
7.1160
6.9615
6.9375
6.9127
4.0545
4.0234
3.1593
3.1261
3.0856
3.0509
2.7210
2.6785
PhHN
O
Ph
HO
NH2
Ph
H
5
1.12
2.18
2.29
9.90
1.03
1.77
2.00
1.01
f1 (ppm)


171.3250
142.8616
138.4936
128.7858
128.2503
127.3651
123.9568
120.3230
82.0516
50.8302
41.4349
PhHN
O
Ph
HO
NH2
Ph
H
5
f1 (ppm)