Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Supporting Information

Zinc-catalyzed asymmetric [3 + 2] annulations for construction of chiral spiro[1-indanone-γ-butyrolactones] via a C−N bond cleavage process

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Table of Contents

General Information

All reactions were carried out under an atmosphere of argon using oven-dried glassware. Super dry solvents, metal catalysts, were purchased from chemical companies and used without further treatment. Flash column chromatography was performed using silica gel (300-400 mesh). ^{1}H NMR ,^{13}C NMR, ^{19}F NMR spectra were recorded in $CDCl_3$ or DMSO-d_6 on a 400 MHz spectrometer; chemical shifts are reported in ppm with the solvent signals as reference, and coupling constants (*J*) are given in Hertz. The peak information is described as: s = singlet, d = doublet, t = triplet, q = quartet, m= multiplet. High-resolution mass spectra (HRMS) were obtained using an Agilent LC-MSAD-Trap-XCT instrument using electrospray ionization time-of-flight (ESI-TOF). High performance liquid chromatography (HPLC) was performed on instrument consisted of JASCO model PU-1580 intelligent HPLC pump and JASCO model UV-1575 intelligent UV-vis detector (254 nm) using Daicel Chiralpak IC, IE, IF (4.6 mm × 250 mm) columns. Melting points were determined using YRT-3 melting point apparatus. Optical rotations were measured with Perkin Elmer, model 341 Polarimeter. The instrumentation used for the crystal measurement is Oxford Gemini E X-ray single-crystal diffractometer. α-Hydroxy-1-indanone[1] and methyleneindolinone[2] were synthesized according to the literature.

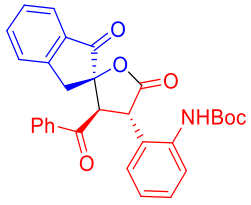
General Procedure for optimization of the reaction conditions.

Under a nitrogen atmosphere, a solution of diethylzinc (20 μL, 1.0 M in hexane, 0.02 mmol) was added dropwise to a solution of **L** (0.01 mmol) in solvent (2 mL). After the mixture was stirred for 30 min at 20 °C, then, α-hydroxy-1-indanone **1a** (0.1 mmol, 14.8 mg), methyleneindolinone **2a** (0.1 mmol, 34.9 mg), and additives were added. The reaction mixture was stirred for corresponding time at the same temperature. The reaction was quenched with NH_4Cl solution (2 mL), and the organic layer was extracted with CH_2Cl_2 (3 × 5 mL). The combined organic layer was washed with brine and dried over Na_2SO_4. The solvent was removed under reduced pressure by using a rotary evaporator. The residue was purified by flash chromatography with petroleum ether/ethyl acetate (4/1) to afford the desired product **3a**.

Synthesis of chiral spiro[1-indanone-γ-butyrolactones]

Under a nitrogen atmosphere, a solution of diethylzinc (40 μL, 1.0 M in hexane, 0.04 mmol) was added dropwise to a solution of **L4** (0.02 mmol, 19.0 mg) in THF (2 mL). After the mixture was stirred for 30 min at 20 °C. Then, α-hydroxy-1-indanone **1a** (0.2 mmol, 29.6 mg), methyleneindolinone **2a** (0.2 mmol, 69.8 mg) and 2-Br-4-ClPhOH (2.0 eq, 82.8 mg) were added. The reaction mixture was stirred for 24 h at the same temperature. The reaction was quenched with NH_4Cl solution (4 mL), and the organic layer was extracted with CH_2Cl_2 (3 × 5 mL). The combined organic layer was washed with brine and dried over Na_2SO_4. The solvent was removed under reduced pressure by using a rotary evaporator. The residue was purified by flash chromatography with petroleum ether/ethyl acetate (5/1) to afford the desired product **3**.

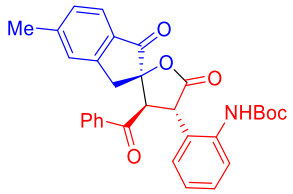
***tert*-Butyl(2-((2*R*,3*R*,4*S*)-3-benzoyl-1',5-dioxo-1',3',4,5-tetrahydro-3*H*-spiro[furan-2,2'-inden]-4-yl)phenyl)carbamate (3a):**



3a

Followed the general procedure, using **1a** (0.2 mmol, 29.6 mg), **2a** (0.2 mmol, 69.8 mg) and **L4** (0.02 mmol, 19.0 mg). Purified by flash column chromatography (petroleum ether/ethyl acetate 4:1) to afforded **3a** as a white solid (64.6 mg, 65% yield, 10:1 dr); $[\alpha]_D^{20}$ = -19.9 (c = 1.0, THF, 95% ee); **m.p.** = 108.0-109.2 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.75 – 7.64 (m, 1H), 7.64 – 7.56 (m, 3H), 7.54 – 7.44 (m, 2H), 7.39 – 7.28 (m, 3H), 7.24 (d, *J* = 7.4 Hz, 2H), 7.20 – 7.13 (m, 3H), 5.02 – 4.75 (m, 2H), 3.37 (dd, *J* = 44.2, 17.7 Hz, 2H), 1.60 (s, 9H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 199.2, 196.1, 174.0, 153.9, 150.0, 136.9, 136.8, 135.2, 134.5, 133.8, 129.1, 129.0, 128.7, 128.5, 126.4, 125.4, 125.0, 84.7, 80.6, 56.5, 44.2, 35.9, 28.4; **HRMS** (ESI): m/z $[M + Na]^+$ calcd for $[C_{30}H_{27}NNaO_6]^+$: 520.1731, found: 520.1726; **HPLC**: Daicel Chiralpak IF, *n*-hexane/*i*-PrOH = 70/30, flow rate = 1 mL/min, λ = 254 nm, major product: t_{major} = 21.16 min and t_{minor} = 15.95 min; minor product: t_{major} = 12.29 min and t_{minor} = 11.00 min.

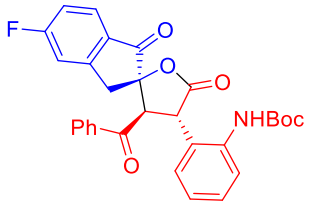
***tert*-Butyl(2-((*2R,3R,4S*)-3-benzoyl-5'-methyl-1',5-dioxo-1',3',4,5-tetrahydro-3*H*-spiro[furan-2,2'-inden]-4-yl)phenyl)carbamate (3b):**



3b

Followed the general procedure, using **1b** (0.2 mmol, 32.8 mg), **2a** (0.2 mmol, 70.2 mg) and **L4** (0.02 mmol, 19.0 mg). Purified by flash column chromatography (petroleum ether/ethyl acetate 4:1) to afforded **3b** as a light yellow oil (54.2 mg, 53% yield, 10:1 dr); $[\alpha]_D^{20}$ = -23.6 (c = 1.0, THF, 90% ee); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.74 – 7.65 (m, 1H), 7.63 (d, *J* = 7.4 Hz, 2H), 7.58 – 7.47 (m, 2H), 7.41 – 7.34 (m, 1H), 7.34 – 7.27 (m, 2H), 7.25 – 7.11 (m, 3H), 7.09 – 6.99 (m, 2H), 5.00 – 4.91 (m, 2H), 3.31 (dd, *J* = 39.6, 17.7 Hz, 2H), 2.33 (s, 3H), 1.59 (s, 9H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 198.4, 196.2, 174.1, 153.8, 150.5, 148.6, 136.9, 135.2, 134.5, 131.5, 129.8, 129.1, 128.9, 128.7, 128.6, 126.6, 125.3, 124.9, 85.0, 80.6, 56.3, 44.2, 35.8, 28.4, 22.2; **HRMS** (ESI): m/z $[M + Na]^+$ calcd for $[C_{31}H_{29}NNaO_6]^+$: 534.1887, found: 534.1885; **HPLC**: Daicel Chiralpak IF, *n*-hexane/*i*-PrOH = 70/30, flow rate = 1 mL/min, λ = 254 nm, t_{major} = 29.73 min and t_{minor} = 17.43 min.

***tert*-Butyl(2-((*2R,3R,4S*)-3-benzoyl-5'-fluoro-1',5-dioxo-1',3',4,5-tetrahydro-3*H*-spiro[furan-2,2'-inden]-4-yl)phenyl)carbamate (3c):**

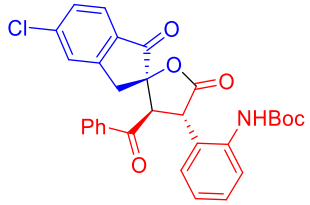


3c

Followed the general procedure, using **1c** (0.2 mmol, 33.6 mg), **2a** (0.2 mmol, 69.5 mg) and **L4** (0.02 mmol, 19.0 mg). Purified by flash column chromatography (petroleum ether/ethyl acetate 4:1) to afforded **3c** as a light yellow oil (65.9 mg, 64% yield, 17:1 dr); $[\alpha]_D^{20}$ = -131.0 (c = 1.0, THF, 90% ee); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.75 – 7.57 (m, 4H), 7.45 (s, 1H), 7.42 – 7.36 (m, 1H), 7.34 – 7.27 (m, 2H), 7.25 – 7.14 (m, 3H), 7.00 – 6.87 (m, 2H), 5.00 – 4.90 (m, 2H), 3.36 (dd, *J* = 45.0, 18.0 Hz, 2H), 1.60 (s, 9H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 197.3, 196.0, 173.8, 168.1 (d, *J* = 260.9 Hz), 153.9, 153.0 (d, *J* = 10.8 Hz), 136.9, 135.1, 134.7, 130.2, 129.2, 129.1, 128.8, 128.5, 127.7, 127.6, 125.5, 117.1 (d, *J* = 23.9 Hz), 113.3 (d, *J* = 22.9 Hz), 84.7, 80.6, 56.4, 44.2, 35.8, 28.4; **^{19}F NMR** (376 MHz, $CDCl_3$) δ -

97.7; **HRMS** (ESI): m/z [M + Na]$^+$ calcd for [$C_{30}H_{26}FNNaO_6$]$^+$: 538.1636, found: 538.1632; **HPLC**: Daicel Chiralpak IF, *n*-hexane/*i*-PrOH = 70/30, flow rate = 1 mL/min, λ = 254 nm, major product: t_{major} = 18.87 min and t_{minor} = 14.53 min; minor product: t_{major} = 11.05 min and t_{minor} = 12.35 min.

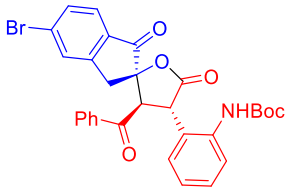
***tert*-Butyl(2-((*2R,3R,4S*)-3-benzoyl-5'-chloro-1',5-dioxo-1',3',4,5-tetrahydro-3*H*-spiro[furan-2,2'-inden]-4-yl)phenyl)carbamate (3d):**



3d

Followed the general procedure, using **1d** (0.2 mmol, 37.2 mg), **2a** (0.2 mmol, 69.8 mg) and **L4** (0.02 mmol, 19.0 mg). Purified by flash column chromatography (petroleum ether/ethyl acetate 4:1) to afforded **3d** as a light yellow oil (64.8 mg, 61% yield 16:1 dr); $[\alpha]_D^{20}$ = -121.0 (c = 1.0, THF, 92% ee); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.74 – 7.64 (m, 1H), 7.64 – 7.57 (m, 2H), 7.54 (d, *J* = 8.2 Hz, 1H), 7.46 – 7.36 (m, 2H), 7.35 – 7.27 (m, 2H), 7.25 – 7.12 (m, 5H), 5.03 – 4.84 (m, 2H), 3.34 (dd, *J* = 43.8, 17.9 Hz, 2H), 1.60 (s, 9H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 197.8, 196.0, 173.8, 153.9, 151.4, 143.5, 136.9, 135.1, 134.7, 132.2, 129.4, 129.2, 129.1, 128.8, 128.6, 126.6, 126.1, 125.5, 84.6, 80.7, 56.4, 44.2, 35.7, 28.4; **HRMS** (ESI): m/z [M + Na]$^+$ calcd for [$C_{30}H_{26}ClNNaO_6$]$^+$: 554.1341, found: 554.1340; **HPLC**: Daicel Chiralpak IF, *n*-hexane/*i*-PrOH = 70/30, flow rate = 1 mL/min, λ = 254 nm, t_{major} = 23.70 min and t_{minor} = 15.24 min.

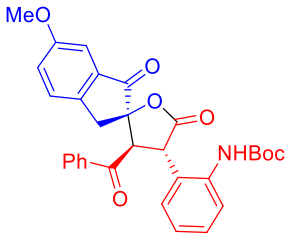
***tert*-Butyl(2-((*2R,3R,4S*)-3-benzoyl-5'-bromo-1',5-dioxo-1',3',4,5-tetrahydro-3*H*-spiro[furan-2,2'-inden]-4-yl)phenyl)carbamate (3e):**



3e

Followed the general procedure, using **1e** (0.2 mmol, 45.5 mg), **2a** (0.2 mmol, 70.3 mg) and **L4** (0.02 mmol, 19.0 mg). Purified by flash column chromatography (petroleum ether/ethyl acetate 4:1) to afforded **3e** as a light yellow oil (51.8 mg, 45% yield, 15:1 dr); $[\alpha]_D^{20}$ = -98.6 (c = 1.0, THF, 93% ee); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.72 – 7.64 (m, 1H), 7.60 (d, *J* = 7.4 Hz, 2H), 7.51 – 7.35 (m, 5H), 7.34 – 7.28 (m, 2H), 7.25 – 7.14 (m, 3H), 4.99 – 4.89 (m, 2H), 3.35 (dd, *J* = 42.8, 17.9 Hz, 2H), 1.60 (s, 9H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 198.1, 196.0, 173.8, 153.9, 151.4, 136.9, 135.1, 134.7, 132.6, 132.5, 132.3, 129.7, 129.2, 129.1, 128.8, 128.6, 126.1, 125.5, 84.5, 80.7, 56.4, 44.2, 35.6, 28.4; **HRMS** (ESI): m/z [M + Na]$^+$ calcd for [$C_{30}H_{26}BrNNaO_6$]$^+$: 598.0836, found: 598.0835; **HPLC**: Daicel Chiralpak IF, *n*-hexane/*i*-PrOH = 80/20, flow rate = 1 mL/min, λ = 254 nm, t_{major} = 44.15 min and t_{minor} = 25.05 min.

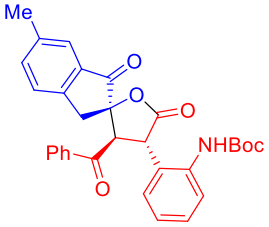
***tert*-Butyl(2-((*2R,3R,4S*)-3-benzoyl-6'-methoxy-1',5-dioxo-1',3',4,5-tetrahydro-3*H*-spiro[furan-2,2'-inden]-4-yl)phenyl)carbamate (3f):**



3f

Followed the general procedure, using **1f** (0.2 mmol, 36.8 mg), **2a** (0.2 mmol, 68.3 mg) and **L4** (0.02 mmol, 19.0 mg). Purified by flash column chromatography (petroleum ether/ethyl acetate 2:1) to afforded **3f** as a light yellow oil (61.2 mg, 58% yield, 10:1 dr); $[\alpha]_D^{20}$ = -103.0 (c = 1.0, THF, 91% ee); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.76 – 7.65 (m, 1H), 7.58 (d, *J* = 8.1 Hz, 2H), 7.52 (s, 1H), 7.39 – 7.28 (m, 3H), 7.24 – 7.14 (m, 3H), 7.13 – 7.04 (m, 2H), 6.99 – 6.94 (m, 1H), 4.99 – 4.87 (m, 2H), 3.72 (s, 3H), 3.28 (dd, *J* = 51.6, 17.3 Hz, 2H), 1.59 (s, 9H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 199.1, 196.1, 174.0, 160.0, 153.9, 142.8, 136.9, 135.3, 135.0, 134.4, 129.2, 129.0, 128.7, 128.4, 127.2, 127.2, 126.2, 125.4, 105.9, 85.4, 80.6, 56.7, 55.6, 44.2, 35.1, 28.4; **HRMS** (ESI): m/z [M + Na]$^+$ calcd for $[C_{31}H_{29}NNaO_7]^+$: 550.1836, found: 550.1837; **HPLC**: Daicel Chiralpak IF, *n*-hexane/*i*-PrOH = 80/20, flow rate = 1 mL/min, λ = 254 nm, t_{major} = 47.80 min and t_{minor} = 30.66 min.

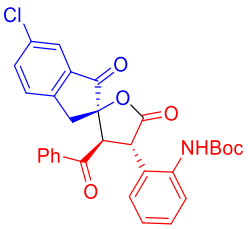
***tert*-Butyl(2-((*2R,3R,4S*)-3-benzoyl-6'-methyl-1',5-dioxo-1',3',4,5-tetrahydro-3*H*-spiro[furan-2,2'-inden]-4-yl)phenyl)carbamate (3g):**



3g

Followed the general procedure, using **1g** (0.2 mmol, 31.6mg), **2a** (0.2 mmol, 71.2 mg) and **L4** (0.02 mmol, 19.0 mg). Purified by flash column chromatography (petroleum ether/ethyl acetate 4:1) to afforded **3g** as a light yellow oil (61.3 mg, 60% yield, 11:1 dr); $[\alpha]_D^{20}$ = -26.3 (c = 1.0, THF, 93% ee); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.74 – 7.64 (m, 1H), 7.62 – 7.57 (m, 2H), 7.53 (s, 1H), 7.40 – 7.28 (m, 5H), 7.21 – 7.13 (m, 3H), 7.11 (d, *J* = 7.8 Hz, 1H), 4.95 (s, 2H), 3.30 (dd, *J* = 44.2, 17.6 Hz, 2H), 2.27 (s, 3H), 1.60 (s, 9H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 199.1, 196.2, 174.1, 153.9, 147.4, 138.7, 138.1, 136.9, 135.2, 134.3, 133.9, 129.1, 129.0, 128.7, 128.5, 126.1, 125.4, 124.8, 85.1, 80.6, 56.5, 44.2, 35.5, 28.4, 21.0; **HRMS** (ESI): m/z [M + Na]$^+$ calcd for $[C_{31}H_{29}NNaO_6]^+$: 534.1887, found: 534.1888; **HPLC**: Daicel Chiralpak IF, *n*-hexane/*i*-PrOH = 80/20, flow rate = 1 mL/min, λ = 254 nm, major product: t_{major} = 35.26 min and t_{minor} = 26.62 min; minor product: t_{major} = 17.84 min and t_{minor} = 24.76 min.

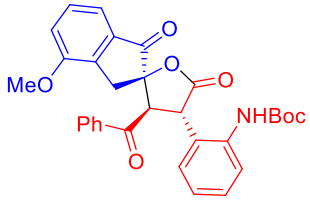
***tert*-Butyl(2-((*2R,3R,4S*)-3-benzoyl-6'-chloro-1',5-dioxo-1',3',4,5-tetrahydro-3*H*-spiro[furan-2,2'-inden]-4-yl)phenyl)carbamate (3h):**



3h

Followed the general procedure, using **1h** (0.2 mmol, 37.8 mg), **2a** (0.2 mmol, 67.2 mg) and **L4** (0.02 mmol, 19.0 mg). Purified by flash column chromatography (petroleum ether/ethyl acetate 4:1) to afforded **3h** as a white solid (53.1 mg, 50% yield, 10:1 dr); $[\alpha]_D^{20}$ = -69.5 (c = 1.0, THF, 90% ee); **m.p.** = 87.5-88.3 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.72 – 7.62 (m, 1H), 7.61 – 7.55 (m, 2H), 7.54 – 7.49 (m, *J* = 1.8 Hz, 1H), 7.48 – 7.35 (m, 3H), 7.33 – 7.28 (m, 2H), 7.25 – 7.13 (m, 4H), 5.00 – 4.87 (m, 2H), 3.33 (dd, *J* = 50.7, 17.8 Hz, 2H), 1.60 (s, 9H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 198.2, 196.0, 173.80, 154.0, 147.9, 136.9, 136.7, 135.2, 134.6, 129.2, 129.1, 128.9, 128.5, 127.6, 125.6, 124.6, 84.9, 80.7, 56.6, 44.2, 35.5, 28.4; **HRMS** (ESI): m/z [M + Na]$^+$ calcd for $[C_{30}H_{26}ClNNaO_6]^+$: 554.1341, found: 554.1340; **HPLC**: Daicel Chiralpak IF, *n*-hexane/*i*-PrOH = 70/30, flow rate = 1 mL/min, λ = 254 nm, major product: t_{major} = 20.27 min and t_{minor} = 15.94 min; minor product: t_{major} = 10.74 min and t_{minor} = 14.87 min.

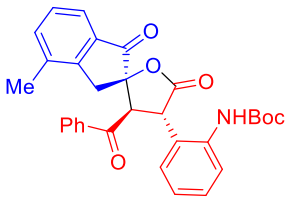
***tert*-Butyl(2-((*2R,3R,4S*)-3-benzoyl-4'-methoxy-1',5-dioxo-1',3',4,5-tetrahydro-3*H*-spiro[furan-2,2'-inden]-4-yl)phenyl)carbamate (3i):**



3i

Followed the general procedure, using **1i** (0.2 mmol, 36.2 mg), **2a** (0.2 mmol, 71.4 mg) and **L4** (0.02 mmol, 19.0 mg). Purified by flash column chromatography (petroleum ether/ethyl acetate 2:1) to afforded **3i** as a light yellow oil (61.2 mg, 58% yield, 9:1 dr); $[\alpha]_D^{20}$ = -15.8 (c = 1.0, THF, 95% ee); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.76 – 7.65 (m, 1H), 7.62 (d, *J* = 7.5 Hz, 2H), 7.59 (s, 1H), 7.38 – 7.27 (m, 3H), 7.24 – 7.12 (m, 5H), 6.95 – 6.87 (m, 1H), 5.01 – 4.88 (m, 2H), 3.79 (s, 2H), 3.23 (dd, *J* = 54.4, 18.2 Hz, 2H), 1.60 (s, 9H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 199.4, 196.2, 174.1, 156.3, 153.8, 139.1, 137.0, 135.2, 135.0, 134.4, 130.1, 129.1, 128.9, 128.7, 128.6, 128.5, 127.1, 125.3, 116.8, 116.4, 84.6, 80.6, 56.66, 55.6, 44.0, 32.8, 28.4; **HRMS** (ESI): m/z [M + Na]$^+$ calcd for $[C_{31}H_{29}NNaO_7]^+$: 550.1836, found: 550.1838; **HPLC**: Daicel Chiralpak IF, *n*-hexane/*i*-PrOH = 70/30, flow rate = 1 mL/min, λ = 254 nm, t_{major} = 21.19 min and t_{minor} = 13.89 min.

***tert*-Butyl(2-((*2R,3R,4S*)-3-benzoyl-4'-methyl-1',5-dioxo-1',3',4,5-tetrahydro-3*H*-spiro[furan-2,2'-inden]-4-yl)phenyl)carbamate (3j):**

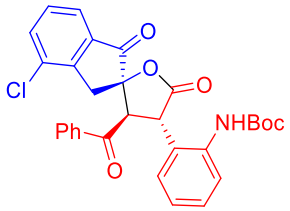


3j

Followed the general procedure, using **1j** (0.2 mmol, 32.8 mg), **2a** (0.2 mmol, 70.4 mg) and **L4** (0.02 mmol, 19.0 mg). Purified by flash column chromatography (petroleum ether/ethyl acetate 3:1) to afforded **3j** as a light yellow oil (56.2 mg, 55% yield, 11:1 dr); $[\alpha]_D^{20}$ = -47.3 (c = 1.0, THF, 92% ee); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.68 (m, 1H), 7.60 (d, *J* = 7.5 Hz, 2H), 7.52 (s, 1H), 7.49 – 7.44 (m, 1H), 7.36 – 7.31 (m, 2H), 7.29 (d, *J* = 7.4 Hz, 2H), 7.21 – 7.12 (m, 4H), 5.02 – 4.93 (m, 2H), 3.22 (dd, *J* = 39.7, 17.7 Hz, 2H), 2.18 (s, 3H), 1.59 (s, 9H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 199.5, 196.3, 174.1, 153.9, 149.0, 137.2, 136.9, 135.7, 135.2, 134.5, 133.6, 129.1, 129.0, 128.8, 128.7, 128.6, 128.4, 127.2, 125.4, 122.4, 84.8, 80.6, 56.4, 44.2, 34.8, 28.4, 17.7; **HRMS** (ESI): m/z [M + Na]$^+$ calcd for

$[C_{31}H_{29}NNaO_6]^+$: 534.1887, found: 534.1887; **HPLC**: Daicel Chiralpak IE, *n*-hexane/*i*-PrOH = 80/20, flow rate = 1 mL/min, λ = 254 nm, major product: t_{major} = 48.91 min and t_{minor} = 41.19 min; minor product: t_{major} = 34.80 min and t_{minor} = 45.42 min.

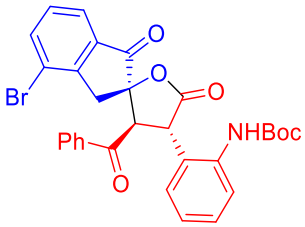
***tert*-Butyl(2-((*2R,3R,4S*)-3-benzoyl-4'-chloro-1',5-dioxo-1',3',4,5-tetrahydro-3*H*-spiro[furan-2,2'-inden]-4-yl)phenyl)carbamate (3k):**



3k

Followed the general procedure, using **1k** (0.2 mmol, 37.5 mg), **2a** (0.2 mmol, 68.8 mg) and **L4** (0.02 mmol, 19.0 mg). Purified by flash column chromatography (petroleum ether/ethyl acetate 4:1) to afforded **3k** as a light yellow oil (46.7 mg, 44% yield, 20:1 dr); $[\alpha]_D^{20}$ = -174.0 (c = 1.0, THF, 90% ee); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.74 – 7.65 (m, 1H), 7.64 – 7.57 (m, 2H), 7.56 – 7.52 (m, 1H), 7.51 – 7.42 (m, 2H), 7.38 – 7.28 (m, 3H), 7.25 – 7.14 (m, 4H), 5.01 – 4.92 (m, 2H), 3.33 (dd, *J* = 43.8, 18.3 Hz, 2H), 1.60 (s, 9H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 198.7, 196.1, 173.8, 153.9, 147.9, 136.9, 136.2, 135.6, 135.2, 134.6, 132.6, 129.9, 129.2, 129.1, 128.8, 128.4, 125.5, 123.2, 84.2, 80.7, 56.6, 44.0, 35.1, 28.4; **HRMS** (ESI): m/z $[M + Na]^+$ calcd for $[C_{30}H_{26}ClNNaO_6]^+$: 554.1341, found: 554.1344; **HPLC**: Daicel Chiralpak IF, *n*-hexane/*i*-PrOH = 80/20, flow rate = 1 mL/min, λ = 254 nm, t_{major} = 25.85 min and t_{minor} = 18.19 min.

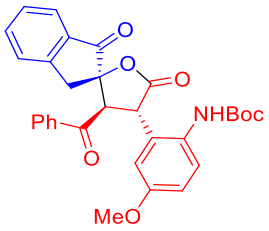
***tert*-Butyl(2-((*2R,3R,4S*)-3-benzoyl-4'-bromo-1',5-dioxo-1',3',4,5-tetrahydro-3*H*-spiro[furan-2,2'-inden]-4-yl)phenyl)carbamate (3l):**



3l

Followed the general procedure, using **1l** (0.2 mmol, 46.5 mg), **2a** (0.2 mmol, 69.8 mg) and **L4** (0.02 mmol, 19.0 mg). Purified by flash column chromatography (petroleum ether/ethyl acetate 4:1) to afforded **3l** as a light yellow oil (52.9 mg, 46% yield, 8:1 dr); $[\alpha]_D^{20}$ = -41.5 (c = 1.0, THF, 90% ee); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.75 – 7.63 (m, 2H), 7.63 – 7.54 (m, 3H), 7.45 (s, 1H), 7.39 – 7.28 (m, 3H), 7.24 – 7.11 (m, 4H), 5.01 – 4.91 (m, 2H), 3.36 – 3.20 (m, 2H), 1.60 (s, 9H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 198.9, 196.1, 173.8, 153.9, 149.9, 139.3, 136.9, 135.7, 135.2, 134.5, 130.9, 130.1, 129.2, 129.1, 128.8, 128.4, 127.3, 125.5, 123.7, 121.7, 84.3, 80.7, 56.7, 44.0, 37.2, 28.4; **HRMS** (ESI): m/z $[M + Na]^+$ calcd for $[C_{30}H_{26}BrNNaO_6]^+$: 598.0836, found: 598.0837; **HPLC**: Daicel Chiralpak IF, *n*-hexane/*i*-PrOH = 70/30, flow rate = 1 mL/min, λ = 254 nm, t_{major} = 18.12 min and t_{minor} = 13.14 min.

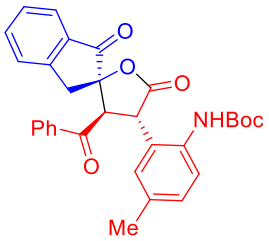
***tert*-Butyl(2-((*2R,3R,4S*)-3-benzoyl-1',5-dioxo-1',3',4,5-tetrahydro-3*H*-spiro[furan-2,2'-inden]-4-yl)-4-methoxyphenyl)carbamate (3m):**



3m

Followed the general procedure, using **1a** (0.2 mmol, 30.5 mg), **2b** (0.2 mmol, 76.8 mg) and **L4** (0.02 mmol, 19.0 mg). Purified by flash column chromatography (petroleum ether/ethyl acetate 2.5:1) to afforded **3m** as a light yellow oil (53.8 mg, 51% yield, 5:1 dr); $[\alpha]_D^{20}$ = -35.6 (c = 1.0, THF, 88% ee); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.63 – 7.55 (m, 3H), 7.52 – 7.41 (m, 2H), 7.38 – 7.32 (m, 1H), 7.26 – 7.14 (m, 4H), 6.99 (s, 1H), 6.89 – 6.80 (m, 2H), 5.03 – 4.84 (m, 2H), 3.78 (s, 3H), 3.36 (dd, *J* = 56.0, 17.7 Hz, 2H), 1.59 (s, 9H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 199.1, 195.7, 174.1, 157.5, 154.6, 149.9, 136.7, 135.4, 134.3, 133.8, 130.1, 129.5, 128.7, 128.7, 128.5, 128.5, 126.4, 125.0, 114.6, 85.0, 80.5, 55.8, 55.6, 45.1, 35.8, 28.4; **HRMS** (ESI): m/z [M + Na]$^+$ calcd for $[C_{31}H_{29}NNaO_7]^+$: 550.1836, found: 550.1841; **HPLC**: Daicel Chiralpak IF, *n*-hexane/*i*-PrOH = 70/30, flow rate = 1 mL/min, λ = 254 nm, t_{major} = 12.27 min and t_{minor} = 17.89 min.

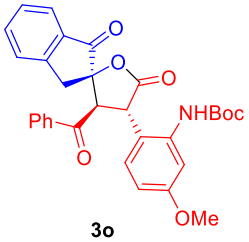
***tert*-Butyl(2-((*2R,3R,4S*)-3-benzoyl-1',5-dioxo-1',3',4,5-tetrahydro-3*H*-spiro[furan-2,2'-inden]-4-yl)-4-methylphenyl)carbamate (3n):**



3n

Followed the general procedure, using **1a** (0.2 mmol, 31.2 mg), **2c** (0.2 mmol, 71.5 mg) and **L4** (0.02 mmol, 19.0 mg). Purified by flash column chromatography (petroleum ether/ethyl acetate 3:1) to afforded **3n** as a light yellow oil (49.1mg, 48% yield, 7:1 dr); $[\alpha]_D^{20}$ = +27.7 (c = 1.0, THF, 96% ee); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.59 (d, *J* = 7.9 Hz, 3H), 7.54 – 7.43 (m, 2H), 7.38 – 7.29 (m, 2H), 7.25 – 7.20 (m, 2H), 7.20 – 7.14 (m, 2H), 7.13 – 7.06 (m, 2H), 5.00 – 4.86 (m, 2H), 3.35 (dd, *J* = 49.6, 17.7 Hz, 2H), 2.31 (s, 3H), 1.59 (s, 9H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 199.2, 196.2, 174.2, 154.1, 150.0, 136.7, 135.4, 135.3, 134.4, 134.2, 133.8, 129.8, 129.7, 128.7, 128.5, 128.5, 127.6, 126.4, 125.8, 125.0, 84.7, 80.4, 56.7, 44.3, 35.8, 28.4, 20.9; **HRMS** (ESI): m/z [M + Na]$^+$ calcd for $[C_{31}H_{29}NNaO_6]^+$: 534.1887, found: 534.1883; **HPLC**: Daicel Chiralpak IF, *n*-hexane/*i*-PrOH = 80/20, flow rate = 1 mL/min, λ = 254 nm, major product: t_{major} = 38.37 min and t_{minor} = 24.81 min; minor product: t_{major} = 15.45 min and t_{minor} = 22.03 min.

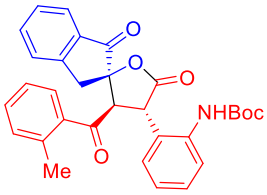
***tert*-Butyl(2-((*2R,3R,4S*)-3-benzoyl-1',5-dioxo-1',3',4,5-tetrahydro-3*H*-spiro[furan-2,2'-inden]-4-yl)-5-methoxyphenyl)carbamate (3o):**



3o

Followed the general procedure, using **1a** (0.2 mmol, 30.8 mg), **2d** (0.2 mmol, 74.9 mg) and **L4** (0.02 mmol, 19.0 mg). Purified by flash column chromatography (petroleum ether/ethyl acetate 2:1) to afforded **3o** as a light yellow oil (61.2 mg, 58% yield, 6 :1 dr); $[\alpha]_D^{20}$ = -28.8 (c = 1.0, THF, 95% ee); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.60 (d, *J* = 8.1 Hz, 4H), 7.52 – 7.44 (m, 1H), 7.41 – 7.31 (m, 2H), 7.26 – 7.13 (m, 5H), 6.82 – 6.68 (m, 1H), 5.03 – 4.73 (m, 2H), 3.78 (s, 3H), 3.35 (dd, *J* = 45.0, 17.7 Hz, 2H), 1.60 (s, 9H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 199.3, 196.4, 174.3, 159.9, 153.6, 150.0, 138.2, 136.8, 135.2, 134.5, 133.8, 129.9, 128.7, 128.5, 126.4, 125.0, 118.4, 111.6, 84.5, 80.6, 56.8, 55.4, 43.5, 35.8, 28.4; **HRMS** (ESI): m/z $[M + Na]^+$ calcd for $[C_{31}H_{29}NNaO_7]^+$:550.1836, found: 550.1834; **HPLC**: Daicel Chiralpak IC, *n*-hexane/*i*-PrOH = 70/30, flow rate = 1 mL/min, λ = 254 nm, major product: t_{major} = 25.14 min and t_{minor} = 35.06 min; minor product: t_{major} = 21.82 min and t_{minor} = 16.68 min.

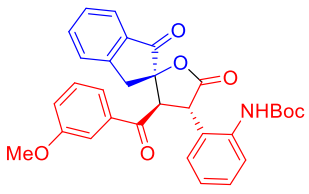
***tert*-butyl(2-((*2R,3R,4S*)-3-(2-methylbenzoyl)-1',5-dioxo-1',3',4,5-tetrahydro-3*H*-spiro[furan-2,2'-inden]-4-yl)phenyl)carbamate (3p):**



3p

Followed the general procedure, using **1a** (0.2 mmol, 32.4 mg), **2e** (0.2 mmol, 72.6 mg) and **L4** (0.02 mmol, 19.0 mg). Purified by flash column chromatography (petroleum ether/ethyl acetate 4:1) to afforded **3p** as a white solid (76.7 mg, 75% yield, 16:1 dr); $[\alpha]_D^{20}$ = -41.3 (c = 1.0, THF, 98% ee); **m.p.** = 136.4-137.7 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.82 – 7.68 (m, 1H), 7.66 (s, 1H), 7.56 – 7.42 (m, 2H), 7.39 – 7.27 (m, 3H), 7.27 – 7.23 (m, 1H), 7.22 – 7.15 (m, 2H), 7.09 (t, *J* = 7.5 Hz, 1H), 6.95 (t, *J* = 7.6 Hz, 1H), 6.84 (d, *J* = 7.6 Hz, 1H), 5.00 – 4.73 (m, 2H), 3.36 (dd, *J* = 48.6, 17.8 Hz, 2H), 2.25 (s, 3H), 1.60 (s, 9H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 199.1, 198.3, 174.2, 153.9, 149.5, 138.9, 137.0, 136.5, 134.6, 133.9, 133.0, 132.0, 129.6, 129.3, 129.0, 128.5, 125.9, 125.8, 125.4, 124.7, 84.6, 80.6, 59.4, 43.9, 35.7, 28.4, 21.2; **HRMS** (ESI): m/z $[M + Na]^+$ calcd for $[C_{31}H_{29}NNaO_6]^+$: 534.1887, found: 534.1884; **HPLC**: Daicel Chiralpak IE, *n*-hexane/*i*-PrOH = 80/20, flow rate = 1 mL/min, λ = 254 nm, t_{major} = 52.92 min and t_{minor} = 40.45 min.

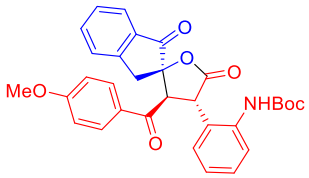
***tert*-Butyl(2-((*2R,3R,4S*)-3-(3-methoxybenzoyl)-1',5-dioxo-1',3',4,5-tetrahydro-3*H*-spiro[furan-2,2'-inden]-4-yl)phenyl)carbamate (3q):**



3q

Followed the general procedure, using **1a** (0.2 mmol, 30.1 mg), **2f** (0.2 mmol, 76.4 mg) and **L4** (0.02 mmol, 19.0 mg). Purified by flash column chromatography (petroleum ether/ethyl acetate 2:1) to afforded **3q** as a light yellow oil (55.9 mg, 53% yield, 7:1 dr); $[\alpha]_D^{20}$ = -34.4 (c = 1.0, THF, 96% ee); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.76 – 7.66 (m, 1H), 7.63 (d, *J* = 7.7 Hz, 1H), 7.57 – 7.45 (m, 2H), 7.42 – 7.27 (m, 3H), 7.23 (s, 1H), 7.21 – 7.14 (m, 2H), 7.12 – 7.01 (m, 2H), 6.96 – 6.80 (m, 1H), 5.02 – 4.86 (m, 2H), 3.71 (s, 3H), 3.45 – 3.26 (m, 2H), 1.60 (s, 9H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 199.3, 196.0, 174.0, 159.6, 153.9, 150.1, 136.9, 136.8, 136.4, 133.8, 129.8, 129.6, 129.2, 129.0, 128.6, 127.3, 126.4, 125.4, 125.0, 121.7, 121.2, 112.0, 84.7, 80.6, 56.7, 55.4, 44.2, 36.0, 28.4; **HRMS** (ESI): m/z [M + Na]$^+$ calcd for $[C_{31}H_{29}NNaO_7]^+$: 550.1836, found: 550.1836;**HPLC**: Daicel Chiralpak IF, *n*-hexane/*i*-PrOH = 80/20, flow rate = 1 mL/min, λ = 254 nm, major product: t_{major} = 35.93 min and t_{minor} = 25.07 min; minor product: t_{major} = 19.43 min and t_{minor} = 23.55 min.

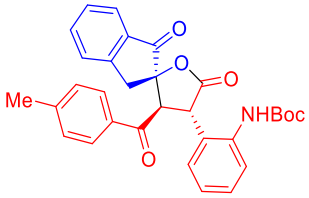
***tert*-Butyl(2-((*2R,3R,4S*)-3-(4-methoxybenzoyl)-1',5-dioxo-1',3',4,5-tetrahydro-3*H*-spiro[furan-2,2'-inden]-4-yl)phenyl)carbamate (3r):**



3r

Followed the general procedure, using **1a** (0.2 mmol, 32.2 mg), **2g** (0.2 mmol, 75.6 mg) and **L4** (0.02 mmol, 19.0 mg). Purified by flash column chromatography (petroleum ether/ethyl acetate 2:1) to afforded **3r** as a light yellow oil (65.4 mg, 62% yield, 7:1 dr); $[\alpha]_D^{20}$ = -47.7 (c = 1.0, THF, 96% ee); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.77 – 7.58 (m, 5H), 7.51 (t, *J* = 7.5 Hz, 1H), 7.41 – 7.27 (m, 3H), 7.26 – 7.21 (m, 1H), 7.15 (t, *J* = 7.5 Hz, 1H), 6.65 (d, *J* = 8.9 Hz, 2H), 5.01 – 4.78 (m, 2H), 3.72 (s, 3H), 3.36 (dd, *J* = 56.1, 17.8 Hz, 2H), 1.59 (s, 9H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 199.4, 194.0, 174.2, 164.7, 153.8, 150.4, 137.0, 136.8, 133.8, 131.2, 129.1, 128.9, 128.4, 128.1, 126.5, 125.2, 125.1, 114.0, 84.8, 80.5, 56.1, 55.6, 44.2, 35.9, 28.4; **HRMS** (ESI): m/z [M + Na]$^+$ calcd for $[C_{31}H_{29}NNaO_7]^+$: 550.1836, found: 550.1839; **HPLC**: Daicel Chiralpak IF, *n*-hexane/*i*-PrOH = 80/20, flow rate = 1 mL/min, λ = 254 nm, major product: t_{major} = 40.97 min and t_{minor} = 31.13 min; minor product: t_{major} = 25.51 min and t_{minor} = 33.82 min.

***tert*-Butyl(2-((*2R,3R,4S*)-3-(4-methylbenzoyl)-1',5-dioxo-1',3',4,5-tetrahydro-3*H*-spiro[furan-2,2'-inden]-4-yl)phenyl)carbamate (3s):**

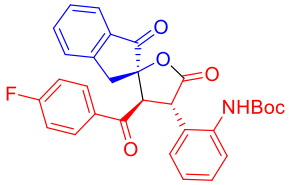


3s

Followed the general procedure, using **1a** (0.2 mmol, 30.5 mg), **2h** (0.2 mmol, 73.9 mg) and **L4** (0.02 mmol, 19.0 mg). Purified by flash column chromatography (petroleum ether/ethyl acetate 3:1) to afforded **3s** as a light yellow oil (49.1 mg, 48% yield, 6:1 dr); $[\alpha]_D^{20}$ = -33.7 (c = 1.0, THF, 95% ee); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.76 – 7.67 (m, 1H), 7.63 (d, *J* = 7.7 Hz, 1H), 7.58 (s, 1H), 7.55 – 7.46 (m, 3H), 7.37 – 7.28 (m, 2H), 7.27 – 7.20 (m, 2H), 7.15 (t, *J* = 7.7 Hz, 1H), 6.97 (d, *J* = 8.1 Hz, 2H), 5.01 – 4.84 (m, 2H), 3.35 (dd, *J* = 46.4, 17.8 Hz, 2H), 2.22 (s, 3H), 1.59 (s, 9H); **^{13}C NMR** (101 MHz, $CDCl_3$)

δ 199.3, 195.6, 174.1, 153.8, 150.2, 145.9, 137.0, 136.8, 133.8, 132.7, 129.4, 129.1, 128.9, 128.7, 128.6, 128.4, 127.0, 126.4, 125.3, 125.1, 84.8, 80.5, 56.3, 44.2, 35.9, 28.4, 21.6; **HRMS** (ESI): m/z [M + Na]$^+$ calcd for $[C_{31}H_{29}NNaO_6]^+$: 534.1887, found: 534.1882; **HPLC**: Daicel Chiralpak IF, *n*-hexane/*i*-PrOH = 80/20, flow rate = 1 mL/min, λ = 254 nm, major product: t_{major} = 33.88 min and t_{minor} = 24.33 min; minor product: t_{major} = 21.60 min and t_{minor} = 26.31 min.

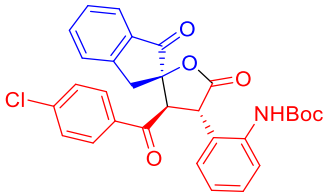
***tert*-Butyl(2-((*2R,3R,4S*)-3-(4-fluorobenzoyl)-1',5-dioxo-1',3',4,5-tetrahydro-3*H*-spiro[furan-2,2'-inden]-4-yl)phenyl)carbamate (3t):**



3t

Followed the general procedure, using **1a** (0.2 mmol, 28.9 mg), **2i** (0.2 mmol, 74.1 mg) and **L4** (0.02 mmol, 19.0 mg). Purified by flash column chromatography (petroleum ether/ethyl acetate 4:1) to afforded **3t** as a light yellow oil (51.5 mg, 50% yield, 5:1 dr); $[\alpha]_D^{20}$ = -49.3 (c = 1.0, THF, 93% ee); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.73 – 7.58 (m, 4H), 7.57 – 7.48 (m, 1H), 7.38 (s, 1H), 7.35 – 7.27 (m, 3H), 7.26 – 7.21 (m, 1H), 7.17 (t, *J* = 8.0 Hz, 1H), 6.86 (t, *J* = 8.5 Hz, 2H), 4.93 (s, 2H), 3.37 (dd, *J* = 41.5, 17.7 Hz, 2H), 1.60 (s, 9H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 199.1, 194.4, 173.9, 166.4 (d, *J* = 258.5 Hz), 154.0, 145.0, 137.0, 136.9, 133.7, 131.7, 131.7, 131.5, 131.4, 129.3, 129.1, 128.6, 127.4, 126.4, 125.6, 125.1, 116.1, 115.9, 84.7, 80.7, 56.3, 44.5, 35.8, 28.4; **^{19}F NMR** (376 MHz, $CDCl_3$) δ -101.6; **HRMS** (ESI): m/z [M + Na]$^+$ calcd for $[C_{30}H_{26}FNNaO_6]^+$: 538.1636, found: 538.1633; **HPLC**: Daicel Chiralpak IE, *n*-hexane/*i*-PrOH = 80/20, flow rate = 1 mL/min, λ = 254 nm, major product: t_{major} =35.97 min and t_{minor} = 30.34 min; minor product: t_{major} = 21.17 min and t_{minor} = 32.06 min.

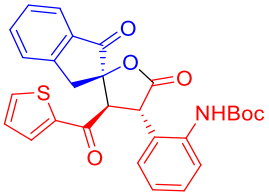
***tert*-Butyl(2-((*2R,3R,4S*)-3-(4-chlorobenzoyl)-1',5-dioxo-1',3',4,5-tetrahydro-3*H*-spiro[furan-2,2'-inden]-4-yl)phenyl)carbamate (3u):**



3u

Followed the general procedure, using **1a** (0.2 mmol, 29.5 mg), **2j** (0.2 mmol, 77.3 mg) and **L4** (0.02 mmol, 19.0 mg). Purified by flash column chromatography (petroleum ether/ethyl acetate 4:1) to afforded **3u** as a light yellow oil (58.4 mg, 55% yield, 6:1 dr); $[\alpha]_D^{20}$ = -29.1 (c = 1.0, THF, 90 % ee); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.73 – 7.60 (m, 2H), 7.58 – 7.50 (m, 3H), 7.36 – 7.27 (m, 4H), 7.26 – 7.22 (m, 1H), 7.20 – 7.09 (m, 3H), 4.92 (s, 2H), 3.36 (dd, *J* = 16.0 Hz, 2H), 1.60 (s, 9H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 199.0, 194.9, 173.9, 154.0, 149.9, 141.2, 137.0, 136.8, 133.7, 133.6, 129.9, 129.3, 129.1, 129.1, 128.6, 127.5, 126.4, 125.9, 125.7, 125.1, 84.7, 80.7, 56.3, 44.6, 35.8, 28.4; **HRMS** (ESI): m/z [M + Na]$^+$ calcd for $[C_{30}H_{26}ClNNaO_6]^+$: 554.1341, found: 554.1343; **HPLC**: Daicel Chiralpak IE, *n*-hexane/*i*-PrOH = 80/20, flow rate = 1 mL/min, λ = 254 nm, major product: t_{major} = 36.25 min and t_{minor} = 29.07 min; minor product: t_{major} = 20.69 min and t_{minor} = 31.57 min.

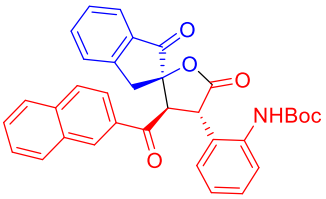
***tert*-Butyl(2-((*2R,3R,4S*)-1',5-dioxo-3-(thiophene-2-carbonyl)-1',3',4,5-tetrahydro-3*H*-spiro[furan-2,2'-inden]-4-yl)phenyl)carbamate (3v):**



3v

Followed the general procedure, using **1a** (0.2 mmol, 30.6 mg), **2k** (0.2 mmol, 71.8 mg) and **L4** (0.02 mmol, 19.0 mg). Purified by flash column chromatography (petroleum ether/ethyl acetate 3:1) to afforded **3v** as a light yellow oil (60.4 mg, 60% yield, 11:1 dr); $[\alpha]_D^{20}$ = -29.8 (c = 1.0, THF, 91% ee); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.72 (d, *J* = 7.7 Hz, 1H), 7.70 – 7.62 (m, 1H), 7.60 – 7.51 (m, 2H), 7.45 (d, *J* = 3.4 Hz, 1H), 7.40 – 7.27 (m, 5H), 7.22 – 7.09 (m, 1H), 6.93 – 6.81 (m, 1H), 5.02 – 4.67 (m, 2H), 3.45 (dd, *J* = 78.3, 17.8 Hz, 2H), 1.59 (s, 9H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 199.1, 187.6, 173.8, 153.9, 150.4, 142.4, 137.0, 137.0, 136.9, 134.7, 133.7, 129.1, 129.0, 128.7, 128.6, 127.0, 126.6, 125.5, 125.1, 84.9, 80.6, 57.0, 44.2, 35.9, 28.4; **HRMS** (ESI): m/z [M + Na]$^+$ calcd for $[C_{28}H_{25}NNaO_6S]^+$: 526.1295, found: 526.1298; **HPLC**: Daicel Chiralpak IF, *n*-hexane/*i*-PrOH = 70/30, flow rate = 1 mL/min, λ = 254 nm, major product: t_{major} = 21.57 min and t_{minor} = 17.36 min; minor product: t_{major} = 15.40 min and t_{minor} = 18.07 min.

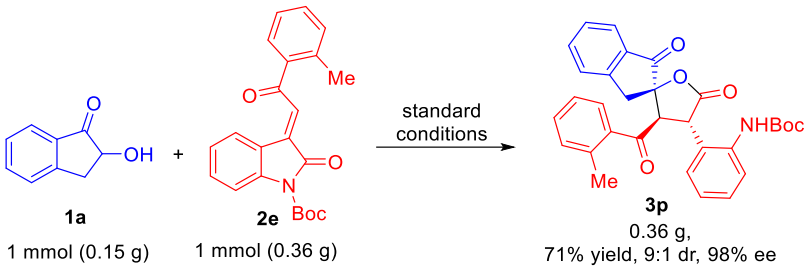
***tert*-Butyl(2-((*2R,3R,4S*)-3-(2-naphthoyl)-1',5-dioxo-1',3',4,5-tetrahydro-3*H*-spiro[furan-2,2'-inden]-4-yl)phenyl)carbamate (3w):**



3w

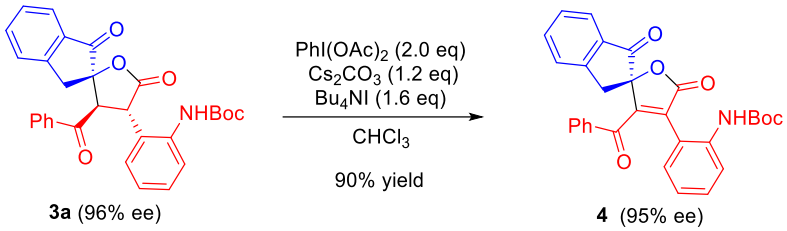
Followed the general procedure, using **1a** (0.2 mmol, 31.1 mg), **2l** (0.2 mmol, 80.6 mg) and **L4** (0.02 mmol, 19.0 mg). Purified by flash column chromatography (petroleum ether/ethyl acetate 3:1) to afforded **3w** as a light yellow oil (71.1 mg, 65% yield, 9:1 dr); $[\alpha]_D^{20}$ = -28.3 (c = 1.0, THF, 93% ee); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.09 (s, 1H), 7.78 (d, *J* = 8.1 Hz, 1H), 7.74 – 7.64 (m, 3H), 7.60 (d, *J* = 8.7 Hz, 1H), 7.58 – 7.51 (m, 2H), 7.51 – 7.44 (m, 2H), 7.41 – 7.28 (m, 3H), 7.22 – 7.11 (m, 2H), 7.00 (t, *J* = 7.5 Hz, 1H), 5.17 – 4.77 (m, 2H), 3.37 (dd, *J* = 48.5, 17.7 Hz, 2H), 1.62 (s, 9H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 199.4, 195.9, 174.2, 154.0, 150.0, 137.0, 136.6, 135.8, 133.8, 132.4, 131.9, 131.5, 129.7, 129.5, 129.3, 129.0, 128.8, 128.3, 127.6, 127.1, 126.2, 125.5, 124.7, 123.2, 84.9, 80.6, 56.6, 44.3, 36.0, 28.5; **HRMS** (ESI): m/z [M + Na]$^+$ calcd for $[C_{34}H_{29}NNaO_6]^+$: 570.1887, found: 570.1892; **HPLC**: Daicel Chiralpak IE, *n*-hexane/*i*-PrOH = 70/30, flow rate = 1 mL/min, λ = 254 nm, major product: t_{major} = 35.56 min and t_{minor} = 28.23 min; minor product: t_{major} = 21.55 min and t_{minor} = 31.18 min.

Mmol-scale reaction



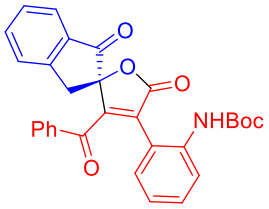
Under a nitrogen atmosphere, a solution of diethylzinc (200 μL, 1.0 M in hexane, 0.2 mmol) was added dropwise to a solution of **L4** (0.1 mmol, 90 mg) in THF (5 mL). After the mixture was stirred for 30 min at room temperature. Then, α-hydroxy-1-indanone **1a** (1.0 mmol, 0.15 g), methyleneindolinone **2e** (1.0 mmol, 0.36 g) and 2-Br-4-ClPhOH (2.0 eq, 0.41 g) were added. The reaction mixture was stirred for 24 h at the same temperature. The reaction was quenched with NH_4Cl solution (10 mL), and the organic layer was extracted with CH_2Cl_2 (3 × 5 mL). The combined organic layer was washed with brine and dried over Na_2SO_4. The solvent was removed under reduced pressure by using a rotary evaporator. The residue was purified by flash chromatography with petroleum ether/ethyl acetate (5/1) to afford the desired product **3p** (0.36g) as a white solid.

Derivatization



Synthesis of **4**[3]: The mixture of **3a** (50.0 mg, 0.1 mmol, 1.0 equiv), $PhI(OAc)_2$ (65.0 mg, 0.2 mmol, 2.0 equiv), and Cs_2CO_3 (39.5 mg, 0.12 mmol, 1.2 equiv) in $CHCl_3$ (2 mL) was treated with Bu_4NI (59.0 mg, 0.16 mmol, 1.6 equiv) at 0 °C. The reaction was allowed to stir at same temperature for 48 h. Upon completion as shown by TLC, the reaction mixture was washed with saturated $Na_2S_2O_3$ (1 mL) and extracted using dichloromethane (3 x 1 mL). The organic layer was dried over Na_2SO_4 and concentrated in vacuo. The residue was purified purified by flash chromatography with petroleum ether/ethyl acetate (5/1) to provide the product **4** as a light yellow oil.

***tert*-Butyl (*R*)-(2-(3-benzoyl-1',5-dioxo-1',3'-dihydro-5*H*-spiro[furan-2,2'-inden]-4-yl)phenyl)carbamate (4):**

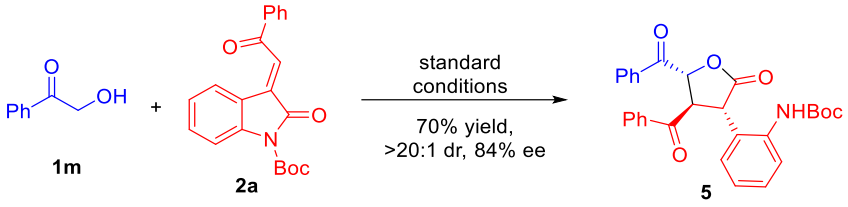


4

Light yellow oil (44.6 mg) in 90% isolated yield; $[\alpha]_D^{20}$ = -20.2 (c = 1.0, THF, 95% ee); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.84 (d, *J* = 7.7 Hz, 1H), 7.70 – 7.60 (m, 1H), 7.49 (d, *J* = 8.2 Hz, 1H), 7.47 – 7.39 (m, 4H), 7.32 – 7.25 (m, 1H), 7.16 – 7.04 (m, 4H), 6.98 (s, 1H), 6.90 – 6.80 (m, 1H), 3.84 – 3.56 (m, 2H), 1.47 (s, 9H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 195.4, 190.2, 171.0, 155.0, 153.0, 150.8, 136.7, 136.6, 134.5, 133.9, 133.6, 131.0, 130.7, 129.3, 128.6, 128.5, 126.7, 125.9, 124.0, 123.4, 120.8, 90.0, 80.5, 35.0, 28.3; **HRMS** (ESI): m/z $[M + Na]^+$ calcd for $[C_{30}H_{25}NNaO_6]^+$: 518.1574, found: 518,1578; **HPLC**:

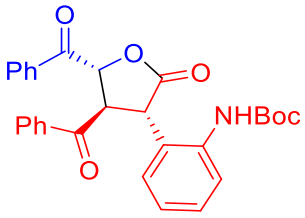
Daicel Chiralpak IC, *n*-hexane/*i*-PrOH = 80/20, flow rate = 1 mL/min, λ = 254 nm, t_{major} = 20.10 min and t_{minor} = 17.88 min.

Further applications



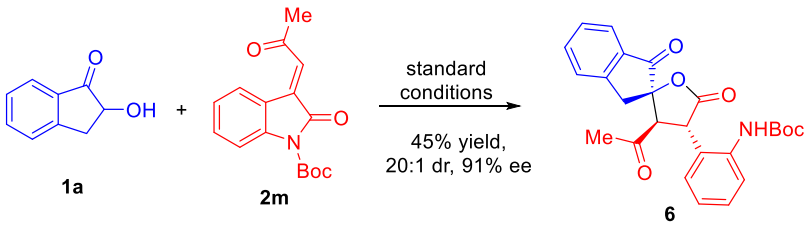
Under a nitrogen atmosphere, a solution of diethylzinc (40 μL, 1.0 M in hexane, 0.04 mmol) was added dropwise to a solution of **L4** (0.02 mmol, 19.0 mg) in THF (2 mL). After the mixture was stirred for 30 min at 20 °C. Then, α-hydroxyacetophenone **1m** (0.2 mmol, 27.5 mg), methyleneindolinone **2** (0.2 mmol, 69.8 mg) and 2-Br-4-ClPhOH (2.0 eq, 83.0 mg) were added. The reaction mixture was stirred for 24 h at the same temperature. The reaction was quenched with NH_4Cl solution (4 mL), and the organic layer was extracted with CH_2Cl_2 (3 × 5 mL). The combined organic layer was washed with brine and dried over Na_2SO_4. The solvent was removed under reduced pressure by using a rotary evaporator. The residue was purified by flash chromatography with petroleum ether/ethyl acetate (5/1) to afford the desired product **5**.

***tert*-Butyl (2-((*3S,4R,5R*)-4,5-dibenzoyl-2-oxotetrahydrofuran-3-yl)phenyl)carbamate (5):**



5

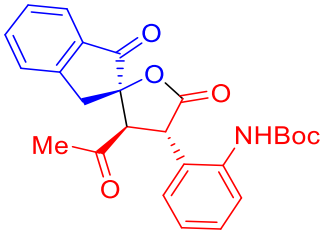
Light yellow oil (67.9 mg, >20:1dr) in 70% isolated yield; $[\alpha]_D^{20}$ = -4.2 (c = 1.0, THF, 84% ee); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.74 – 7.60 (m, 5H), 7.53 (s, 1H), 7.50 – 7.42 (m, 2H), 7.35 – 7.28 (m, 3H), 7.27 – 7.22 (m, 2H), 7.21 – 7.16 (m, 1H), 7.16 – 7.10 (m, 1H), 6.25 (d, *J* = 8.5 Hz, 1H), 5.01 – 4.86 (m, 1H), 4.77 (d, *J* = 10.4 Hz, 1H), 1.57 (s, 9H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 195.0, 193.7, 175.3, 154.2, 136.8, 135.9, 134.3, 134.3, 134.1, 129.0, 129.0, 128.9, 128.8, 128.6, 128.2, 126.2, 125.7, 80.6, 76.6, 55.6, 43.0, 28.4; **HRMS** (ESI): m/z [M + Na]$^+$ calcd for $[C_{29}H_{27}NNaO_6]^+$: 508.1731, found: 508.1736; **HPLC**: Daicel Chiralpak AD, *n*-hexane/*i*-PrOH = 80/20, flow rate = 1 mL/min, λ = 254 nm, t_{major} = 21.03 min and t_{minor} = 42.17 min.



Under a nitrogen atmosphere, a solution of diethylzinc (40 μL, 1.0 M in hexane, 0.04 mmol) was added dropwise to a solution of **L4** (0.02 mmol, 19.0 mg) in THF (2 mL). After the mixture was stirred for 30 min at 20 ℃. Then, α-hydroxyacetophenone **1a** (0.2 mmol, 31.1 mg), methyleneindolinone **2m** (0.2 mmol, 57.8 mg) and 2-Br-4-ClPhOH (2.0 eq, 83.0 mg) were added. The reaction mixture was stirred for

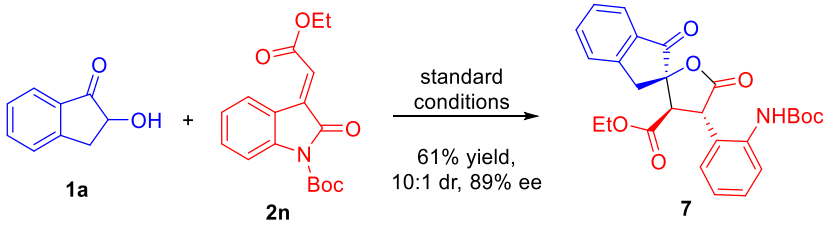
24 h at the same temperature. The reaction was quenched with NH_4Cl solution (4 mL), and the organic layer was extracted with CH_2Cl_2 (3 × 5 mL). The combined organic layer was washed with brine and dried over Na_2SO_4. The solvent was removed under reduced pressure by using a rotary evaporator. The residue was purified by flash chromatography with petroleum ether/ethyl acetate (5/1) to afford the desired product **6**.

***tert*-butyl(2-(3-acetyl-1',5-dioxo-1',3',4,5-tetrahydro-3*H*-spiro[furan-2,2'-inden]-4-yl)phenyl)carbamate (6):**



6

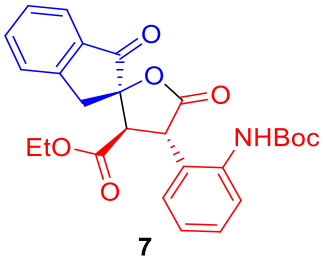
Light yellow oil (39.2 mg, >20:1dr) in 45% isolated yield; $[\alpha]_D^{20}$ = -26.0 (c = 1.0, THF, 91% ee); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.85 (d, *J* = 7.7 Hz, 1H), 7.71 – 7.57 (m, 2H), 7.50 – 7.38 (m, 3H), 7.27 – 7.20 (m, 1H), 7.17 – 7.05 (m, 2H), 4.56 (d, *J* = 12.0 Hz, 1H), 4.12 (d, *J* = 12.0 Hz, 1H), 3.39 – 3.23 (m, 2H), 1.66 (s, 3H), 1.49 (s, 9H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 204.0, 198.6, 173.7, 153.8, 150.3, 137.4, 136.9, 133.6, 129.1, 129.0, 129.0, 127.1, 126.9, 125.5, 125.4, 83.7, 80.6, 62.0, 43.4, 36.1, 30.4, 28.4; **HRMS** (ESI): m/z [M + Na]$^+$ calcd for $[C_{25}H_{25}NNaO_6]^+$: 458.1574, found: 458.1579; **HPLC**: Daicel Chiralpak IF, *n*-hexane/*i*-PrOH = 70/30, flow rate = 1 mL/min, λ = 254 nm, t_{major} = 21.08 min and t_{minor} = 10.35 min.



1a **2n** **7**

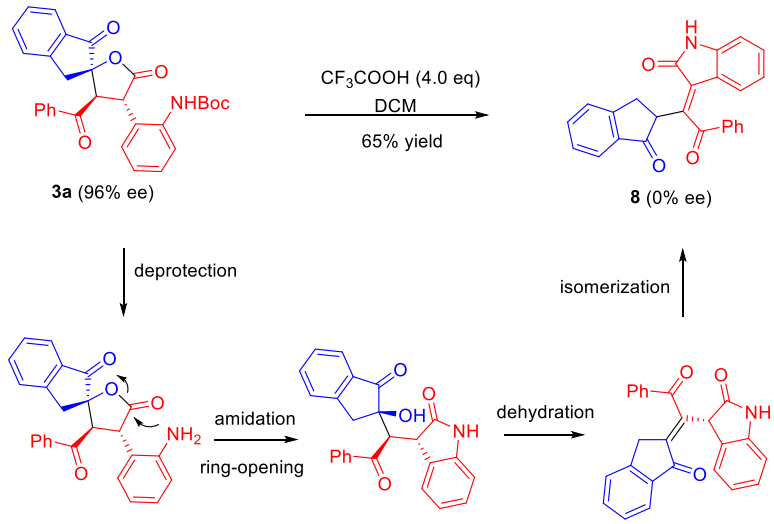
Under a nitrogen atmosphere, a solution of diethylzinc (40 μL, 1.0 M in hexane, 0.04 mmol) was added dropwise to a solution of **L4** (0.02 mmol, 19.0 mg) in THF (2 mL). After the mixture was stirred for 30 min at 20 ℃. Then, α-hydroxyacetophenone **1a** (0.2 mmol, 31.1 mg), methyleneindolinone **2n** (0.2 mmol, 63.4 mg) and 2-Br-4-ClPhOH (2.0 eq, 83.0 mg) were added. The reaction mixture was stirred for 24 h at the same temperature. The reaction was quenched with NH_4Cl solution (4 mL), and the organic layer was extracted with CH_2Cl_2 (3 × 5 mL). The combined organic layer was washed with brine and dried over Na_2SO_4. The solvent was removed under reduced pressure by using a rotary evaporator. The residue was purified by flash chromatography with petroleum ether/ethyl acetate (5/1) to afford the desired product **7**.

ethyl 4-(2-((*tert*-butoxycarbonyl)amino)phenyl)-1',5-dioxo-1',3',4,5-tetrahydro-3*H*-spiro[furan-2,2'-indene]-3-carboxylate (7):



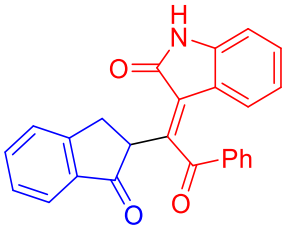
7

Light yellow oil (55.8 mg, 10:1dr) in 61% isolated yield; $[\alpha]_D^{20}$ = -42.6 (c = 1.0, THF, 89% ee); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.89 (d, *J* = 7.7 Hz, 1H), 7.76 – 7.65 (m, 3H), 7.53 – 7.42 (m, 2H), 7.38 – 7.29 (m, 1H), 7.27 – 7.15 (m, 2H), 4.62 (d, *J* = 12.3 Hz, 1H), 3.94 (d, *J* = 12.3 Hz, 1H), 3.90 – 3.74 (m, 2H), 3.54 – 3.36 (m, 2H), 1.56 (s, 9H), 0.62 (t, *J* = 7.1 Hz, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 198.5, 173.5, 169.4, 153.7, 150.2, 137.1, 136.8, 134.1, 128.9, 128.8, 128.7, 127.0, 126.4, 125.3, 125.1, 83.6, 80.5, 62.2, 55.8, 43.5, 36.5, 28.4, 13.1; **HRMS** (ESI): m/z $[M + Na]^+$ calcd for $[C_{26}H_{27}NNaO_7]^+$: 488.1680, found: 488.1691; **HPLC**: Daicel Chiralpak IF, *n*-hexane/*i*-PrOH = 70/30, flow rate = 1 mL/min, λ = 254 nm, major product: t_{major} = 21.30 min and t_{minor} = 11.35 min; minor product: t_{major} = 10.32 min and t_{minor} = 12.68 min.



Synthesis of **8**: The mixture of **3a** (50.0 mg, 0.1 mmol, 1.0 equiv) in DCM (2 mL) was treated with CF_3COOH (45.5 mg, 0.4 mmol, 4.0 equiv). The reaction was allowed to stir at rt for 24 h. Upon completion as shown by TLC, the reaction mixture was washed with saturated $NaHCO_3$ (1 mL) and extracted using dichloromethane (3 x 1 mL). The organic layer was dried over Na_2SO_4 and concentrated in vacuo. The residue was purified purified by flash chromatography with petroleum ether/ethyl acetate (10/1) to provide the product **8** as a colourless oil.

(E)-3-(2-oxo-1-(1-oxo-2,3-dihydro-1H-inden-2-yl)-2-phenylethylidene)indolin-2-one (8):



8

Colourless oil (25.0 mg) in 60% isolated yield; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.15 (d, *J* = 8.4 Hz, 1H),

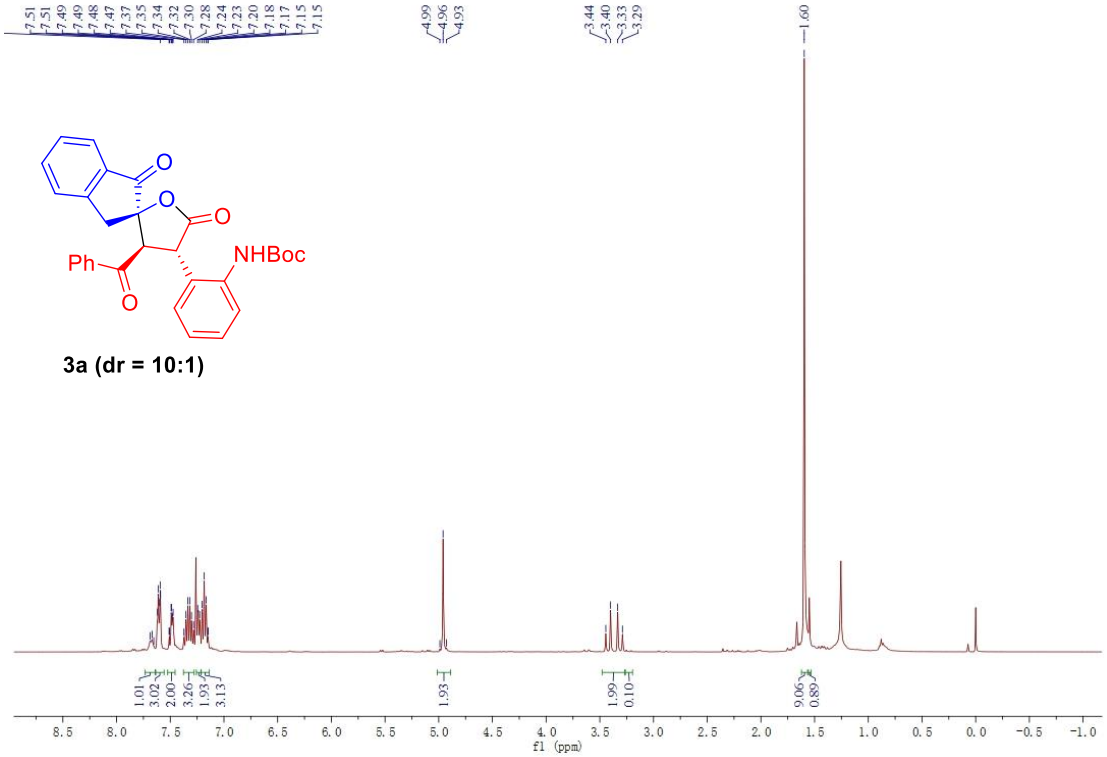
7.87 (s, 1H), 7.84 (d, *J* = 7.6 Hz, 1H), 7.75 – 7.55 (m, 5H), 7.53 – 7.32 (m, 6H), 4.40 – 4.14 (m, 1H), 3.73 – 3.46 (m, 1H), 3.29 – 3.08 (m, 1H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 206.3, 160.9, 153.2, 146.7, 140.4, 136.4, 135.3, 135.0, 132.6, 129.6, 129.4, 129.3, 128.5, 128.4, 128.0, 127.6, 127.2, 126.7, 126.4, 124.4, 50.2, 37.7; **HRMS** (ESI): m/z $[M + H]^+$ calcd for $[C_{25}H_{18}NO_3]^+$: 380.1281, found: 380.1284.

References

1. K. Matsuo and M. Shindo, *Org. Lett.,* 2010, **12**, 5346–5349.
2. J. Guo, Y. Liu, X. Li, X. Liu, L. Lin and X. Feng, *Chem. Sci.*, 2016, **7**, 2717-2721.
3. V. S. Raut, M. Jean, N. Vanthuyne, C. Roussel, T. Constantieux, C. Bressy, X. Bugaut, D. Bonne and J. Rodriguez, *J. Am. Chem. Soc.,* 2017, **139**, 2140–2143.

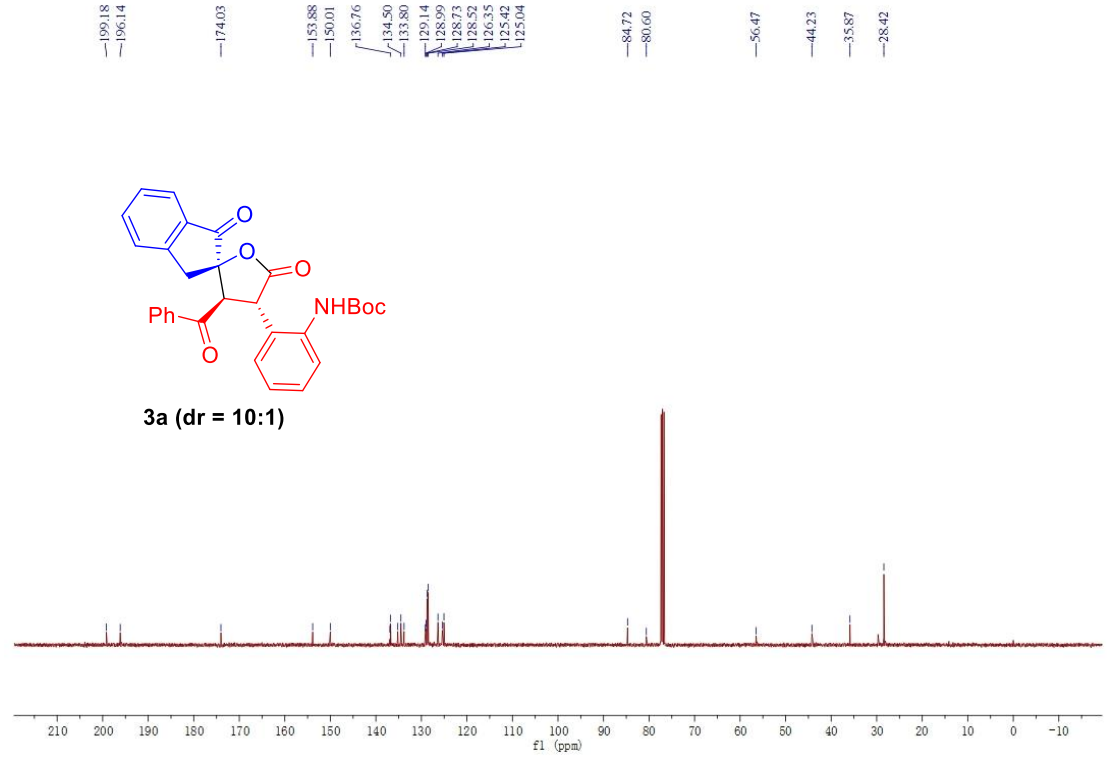
NMR Spectra of compounds

^{1}H NMR (400 MHz, $CDCl_3$)



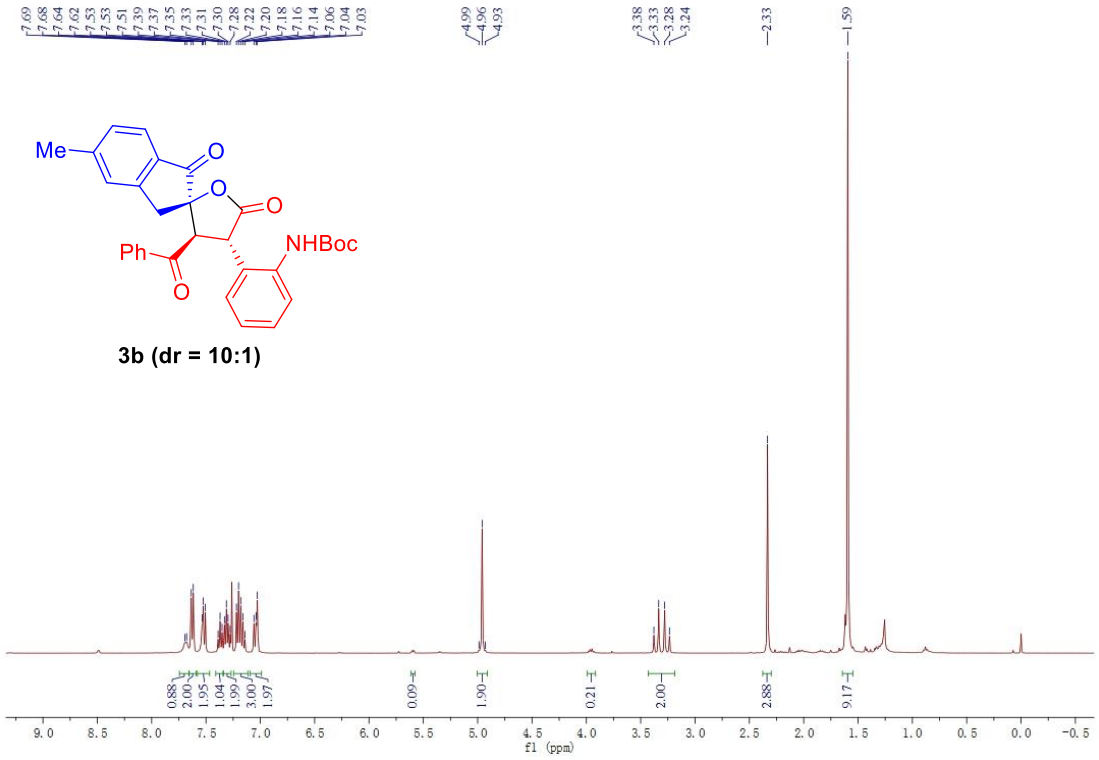
3a (dr = 10:1)

^{13}C NMR (101 MHz, $CDCl_3$)



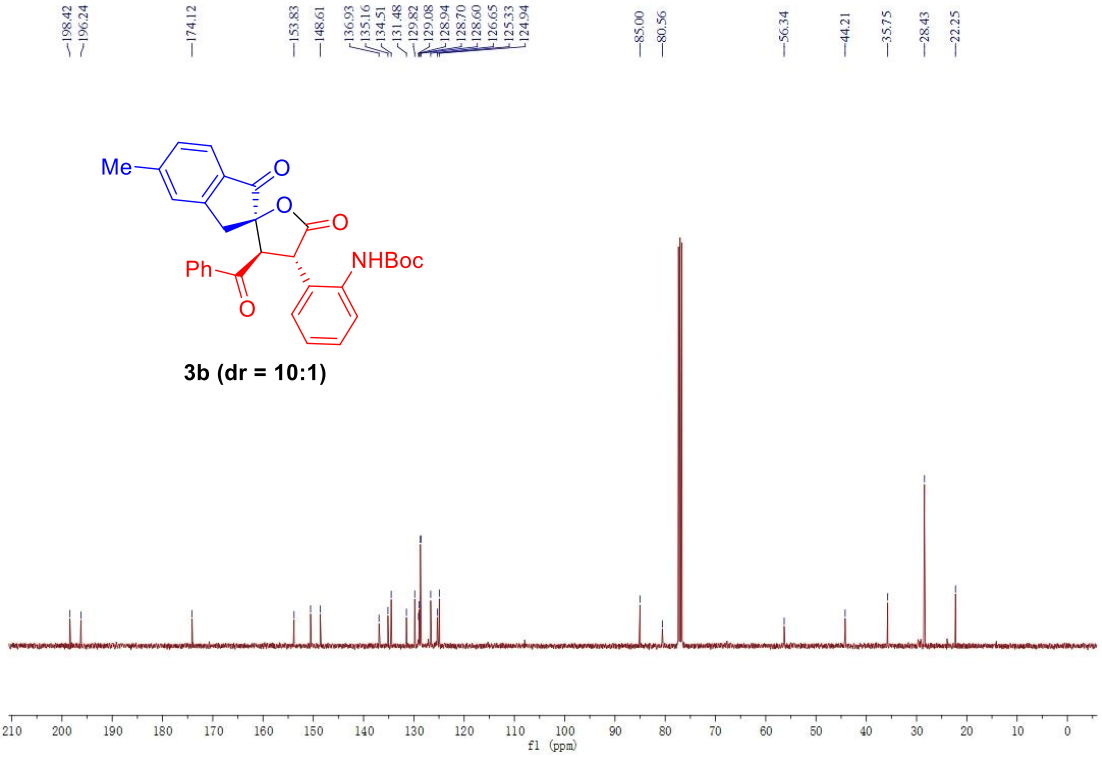
3a (dr = 10:1)

^{1}H NMR (400 MHz, $CDCl_3$)



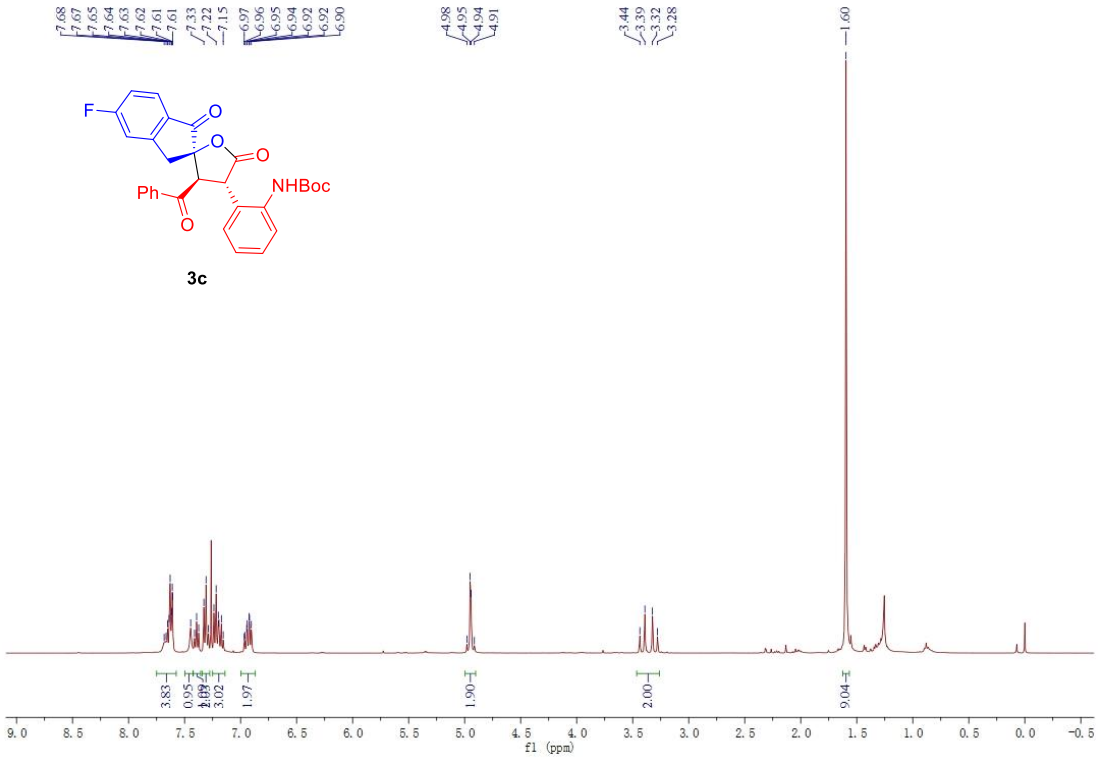
3b (dr = 10:1)

^{13}C NMR (101 MHz, $CDCl_3$)

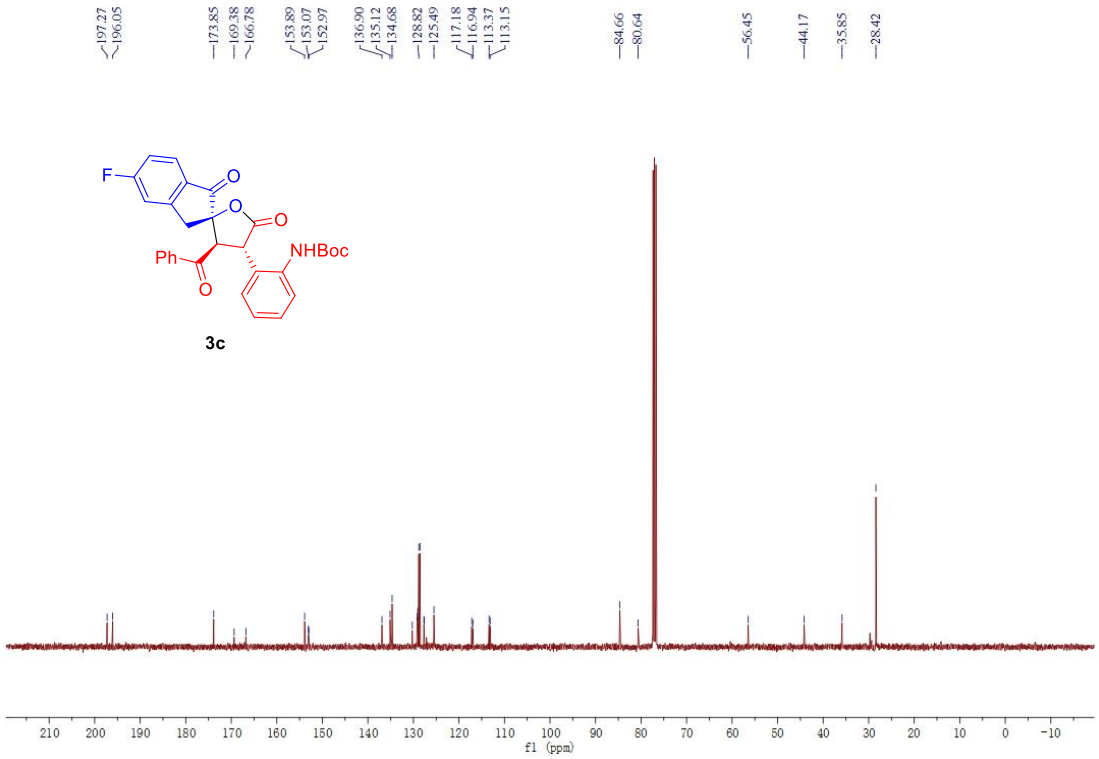


3b (dr = 10:1)

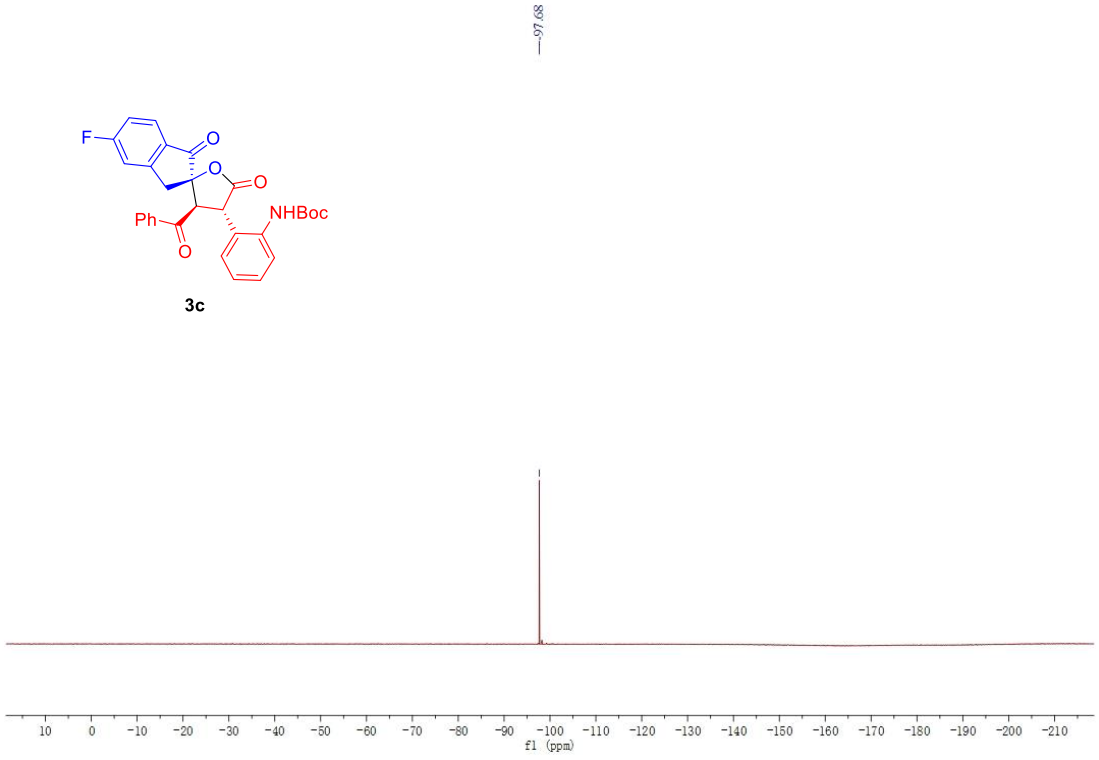
^{1}H NMR (400 MHz, $CDCl_3$)



^{13}C NMR (101 MHz, $CDCl_3$)

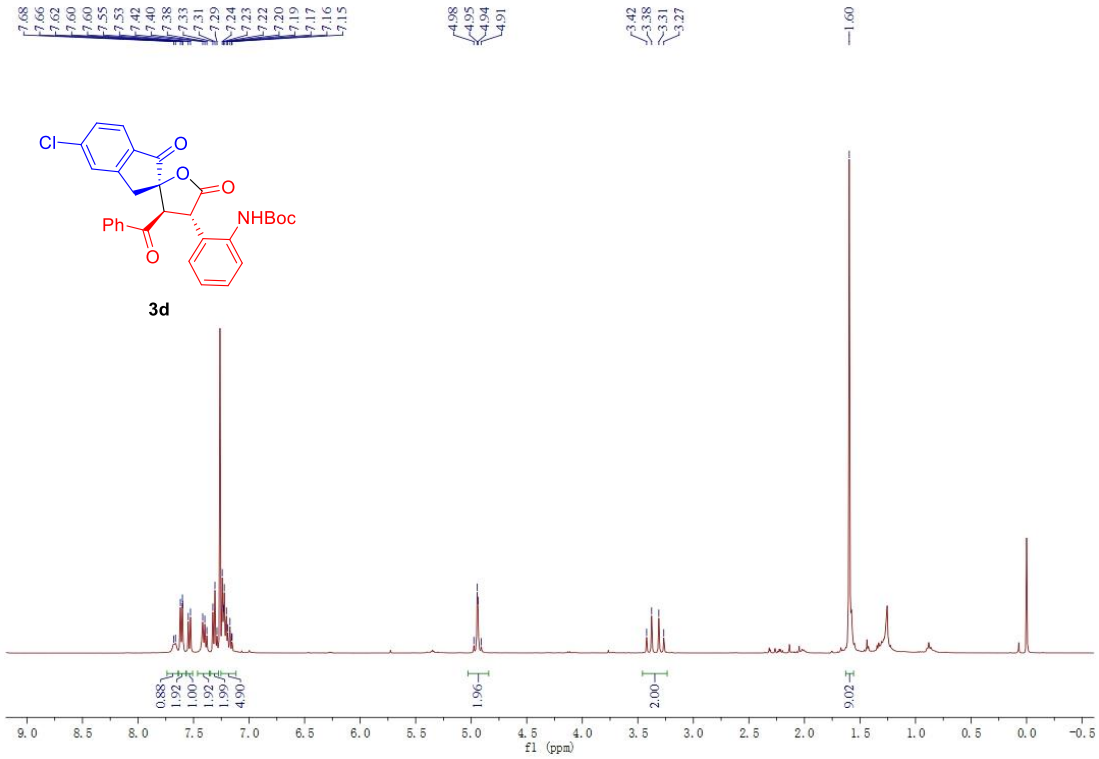


^{19}F NMR (376 MHz, $CDCl_3$)



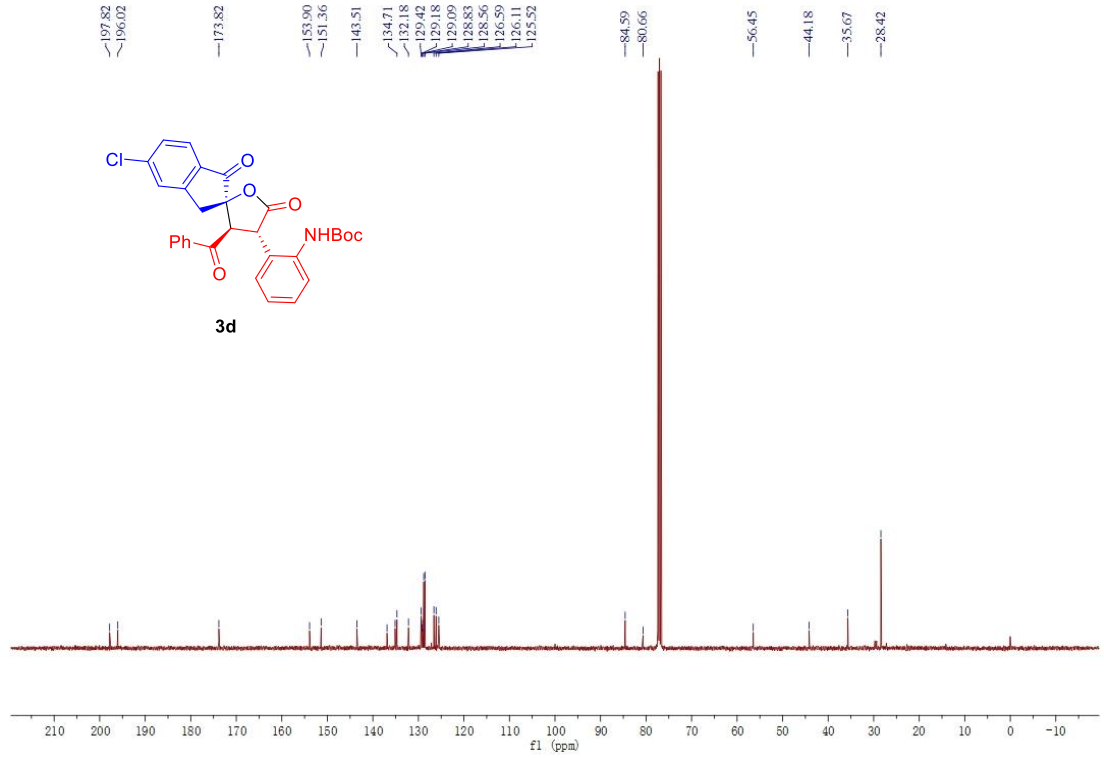
3c

^{1}H NMR (400 MHz, $CDCl_3$)



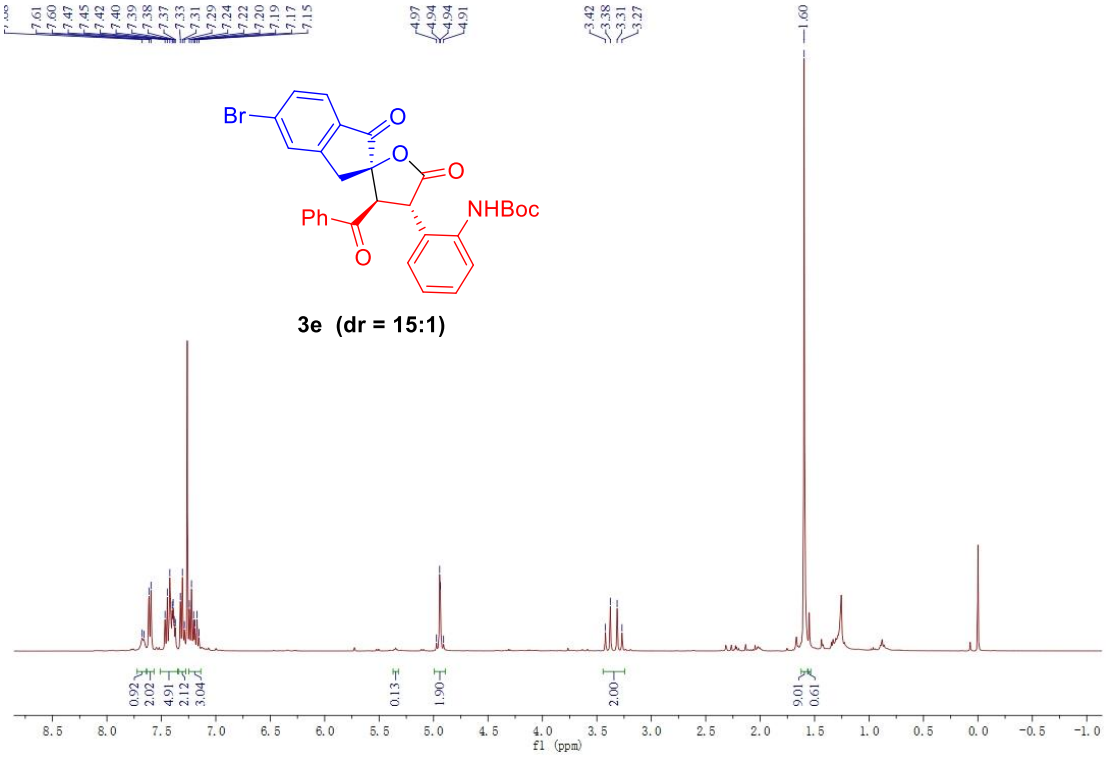
3d

^{13}C NMR (101 MHz, $CDCl_3$)



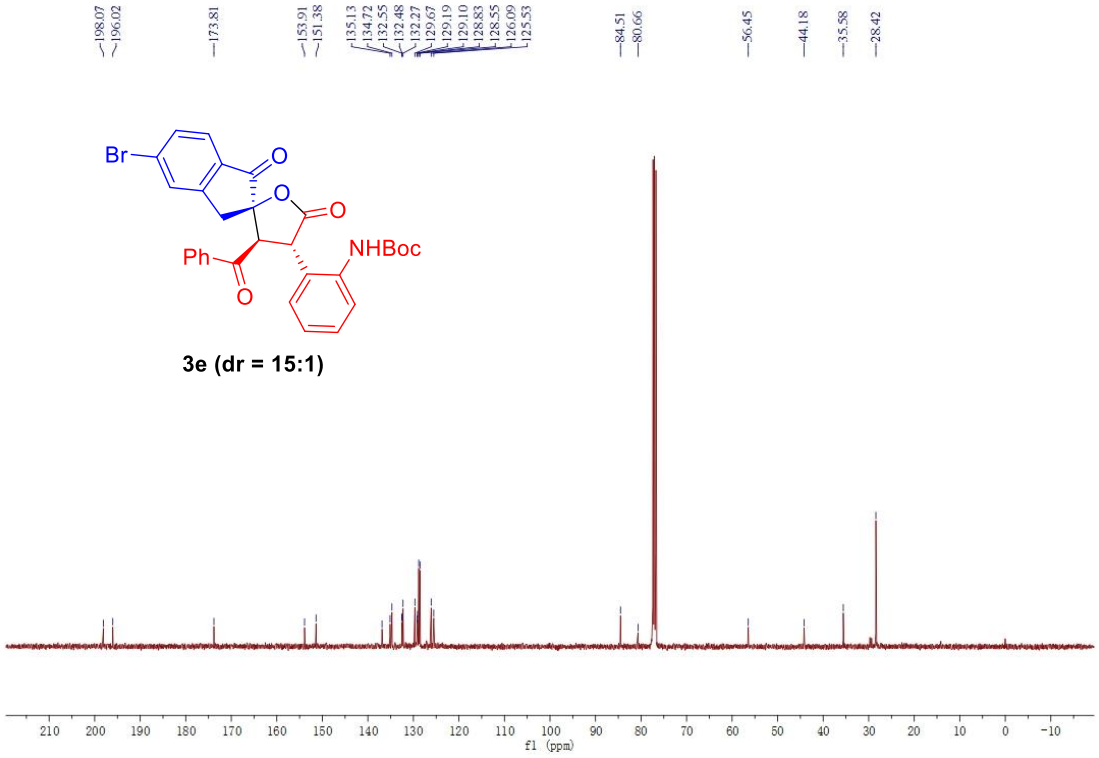
3d

^{1}H NMR (400 MHz, $CDCl_3$)



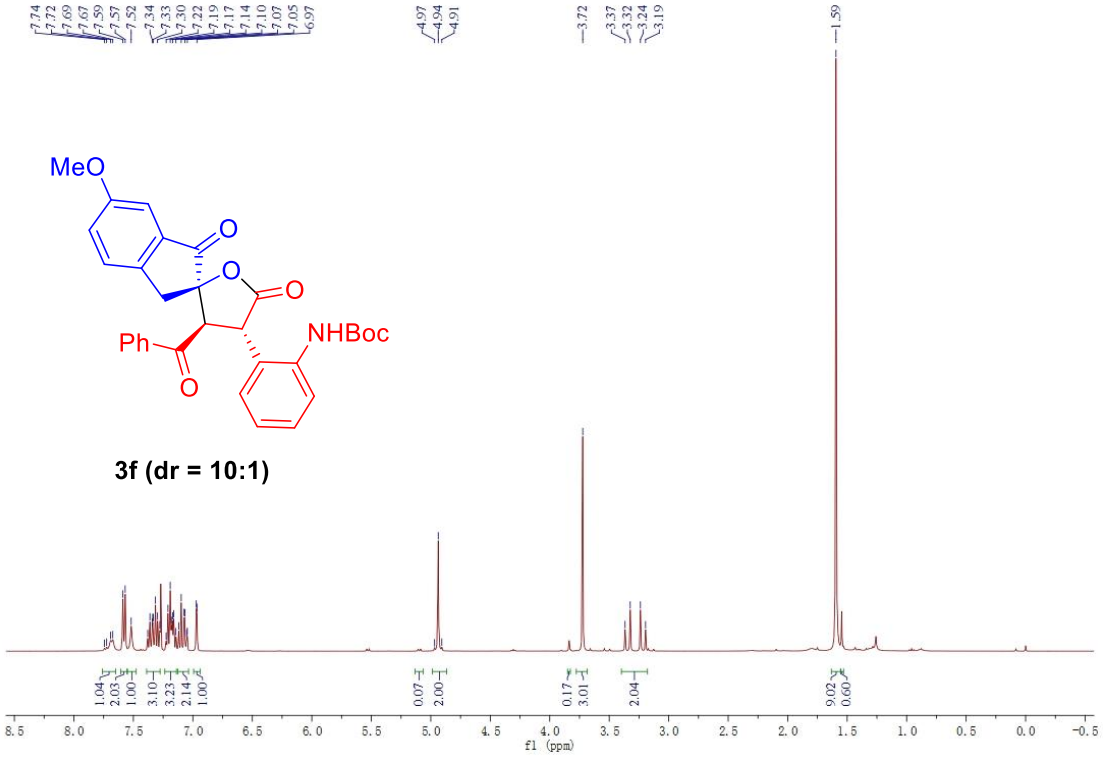
3e (dr = 15:1)

^{13}C NMR (101 MHz, $CDCl_3$)



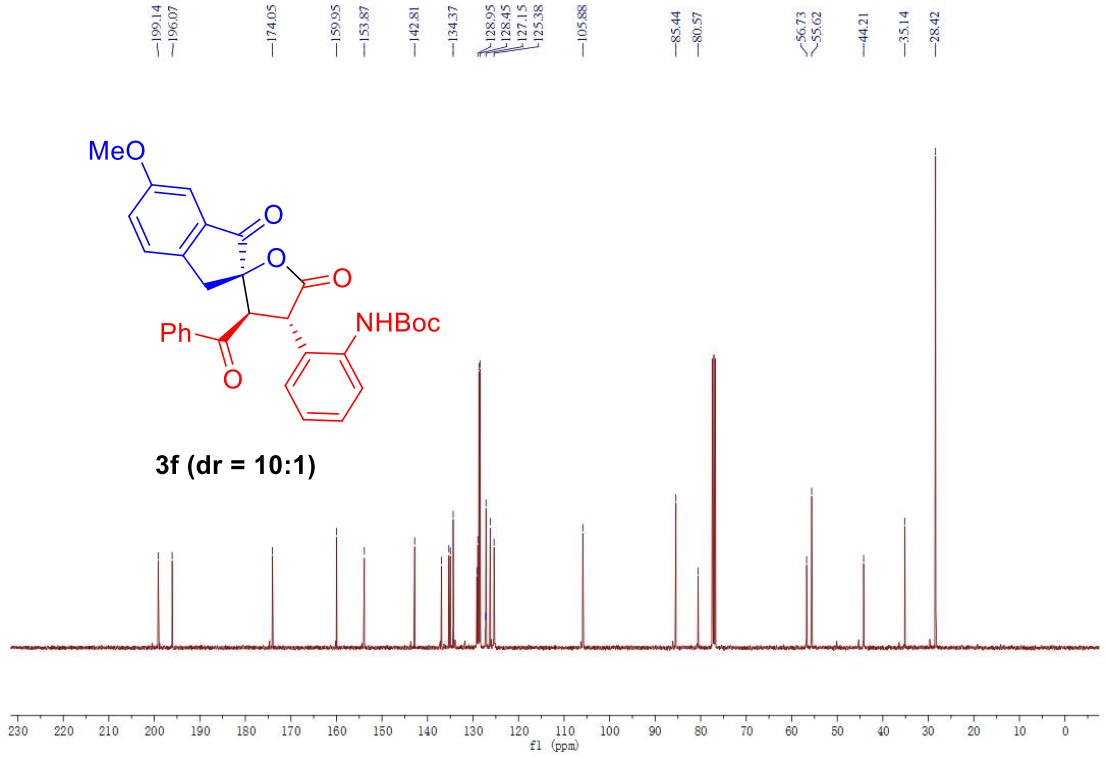
3e (dr = 15:1)

^{1}H NMR (400 MHz, $CDCl_3$)

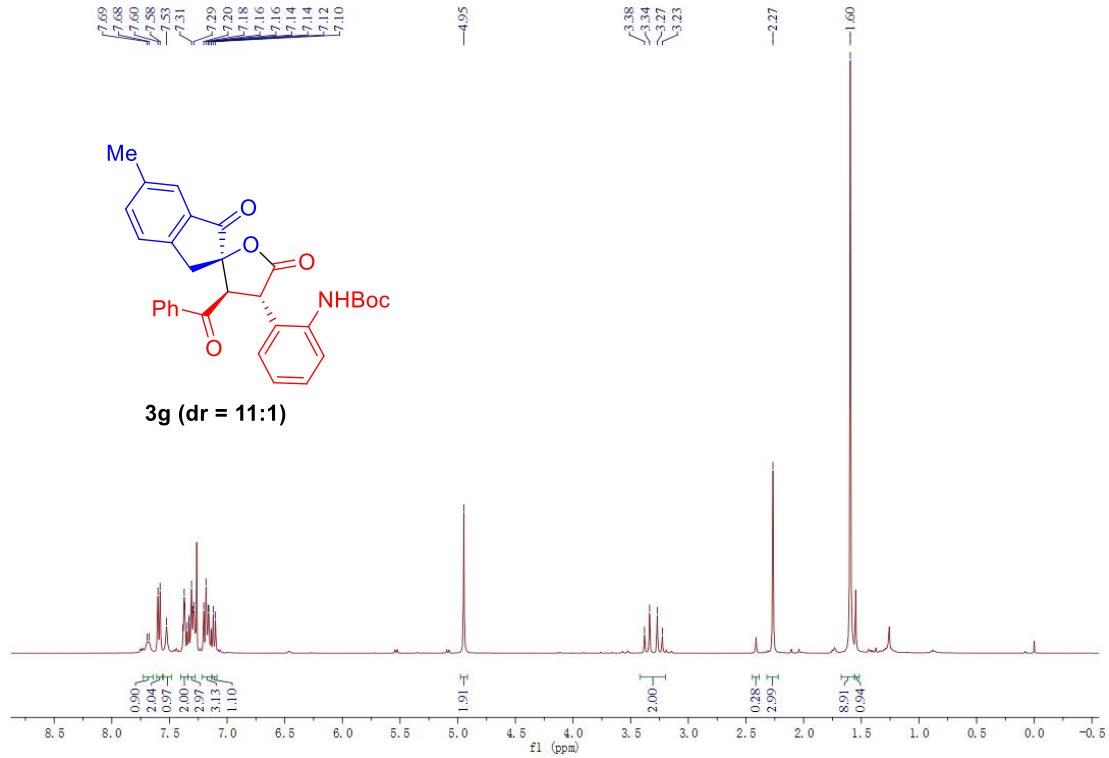


3f (dr = 10:1)

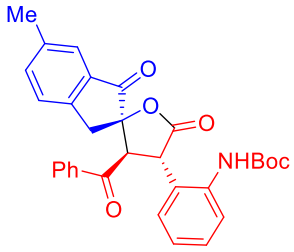
^{13}C NMR (101 MHz, $CDCl_3$)



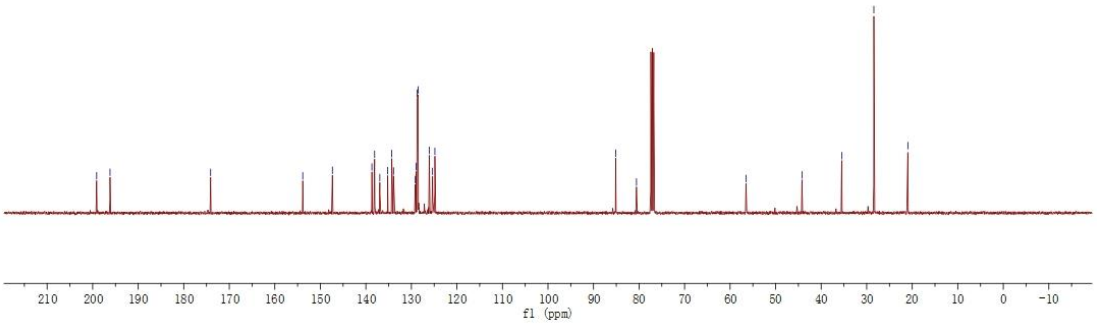
^{1}H NMR (400 MHz, $CDCl_3$)



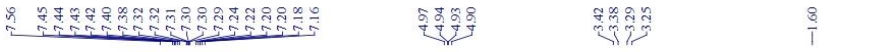
^{13}C NMR (101 MHz, $CDCl_3$)

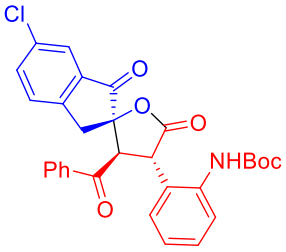


3g (dr = 11:1)



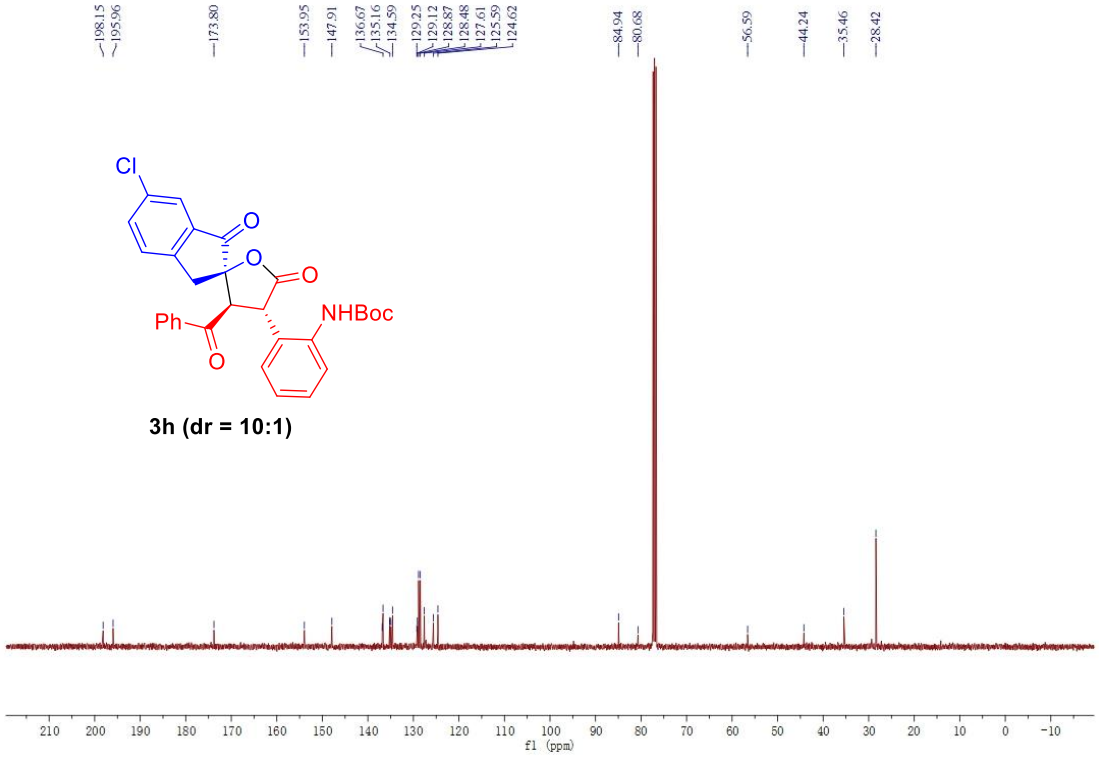
^{1}H NMR (400 MHz, $CDCl_3$)





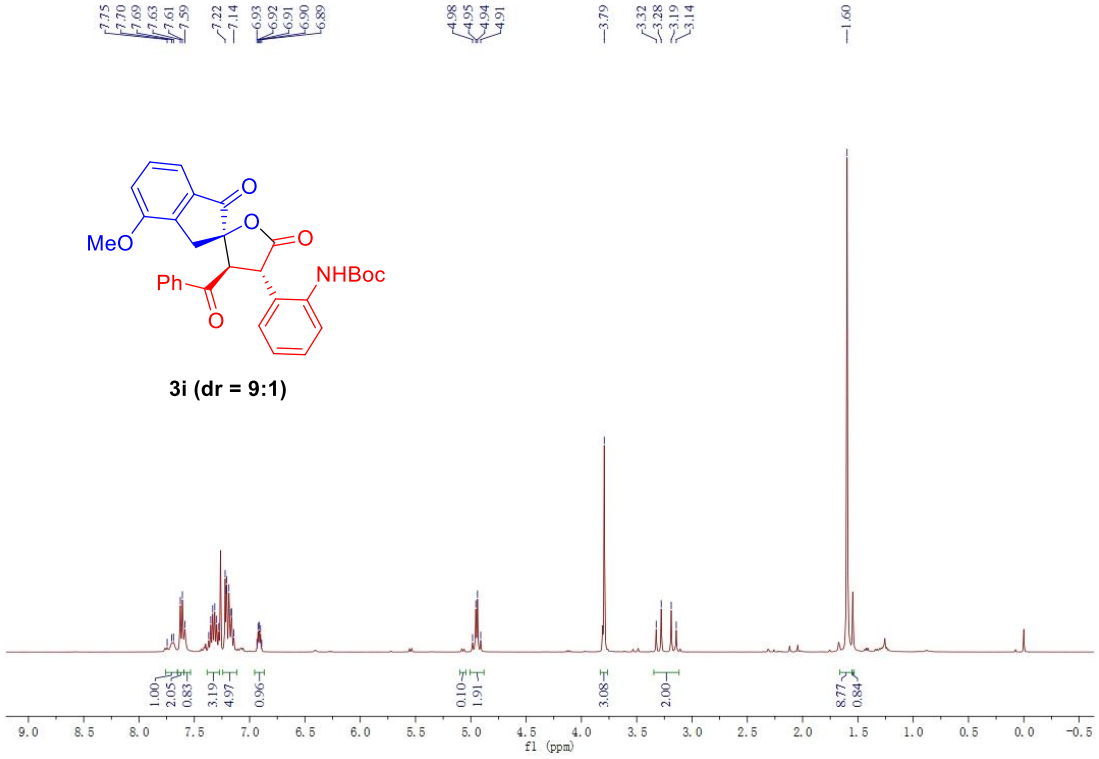
3h (dr = 10:1)

^{13}C NMR (101 MHz, $CDCl_3$)



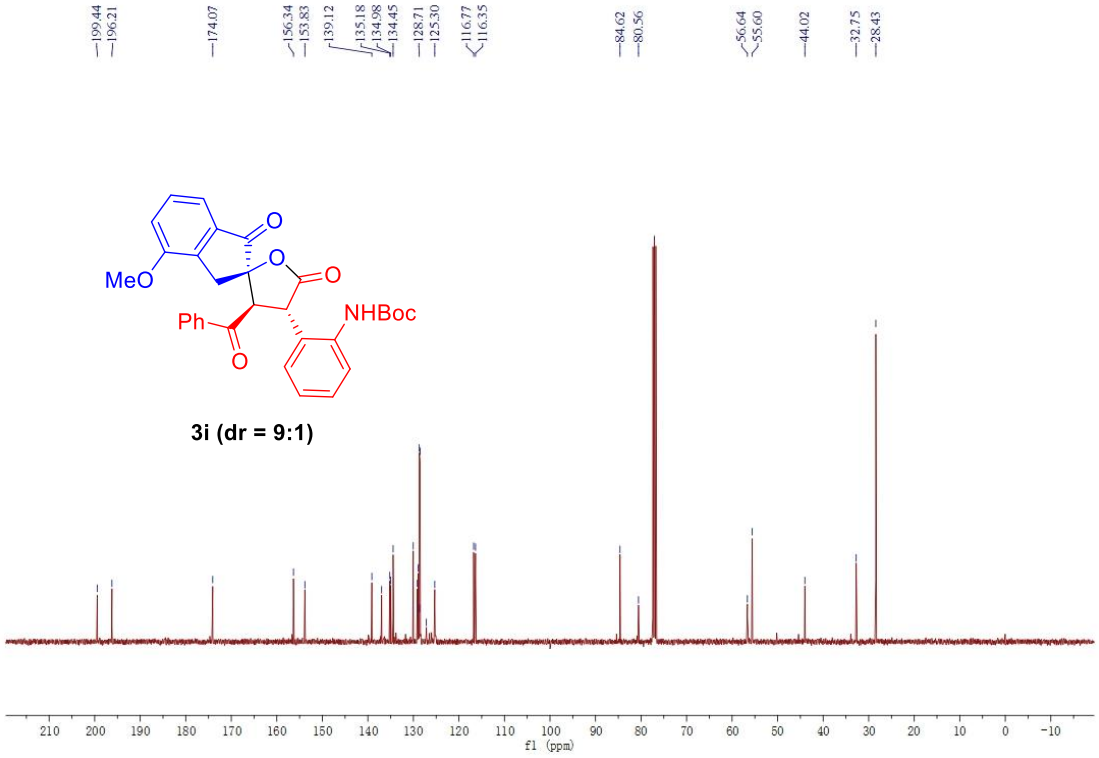
3h (dr = 10:1)

^{1}H NMR (400 MHz, $CDCl_3$)

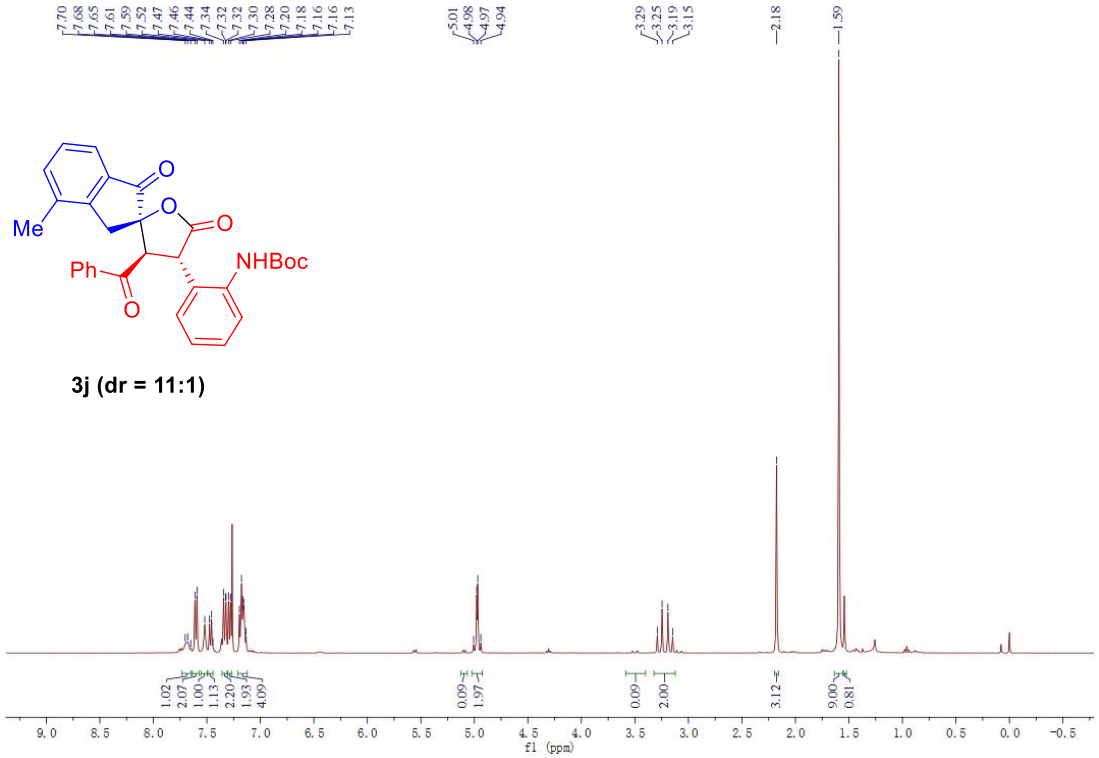


3i (dr = 9:1)

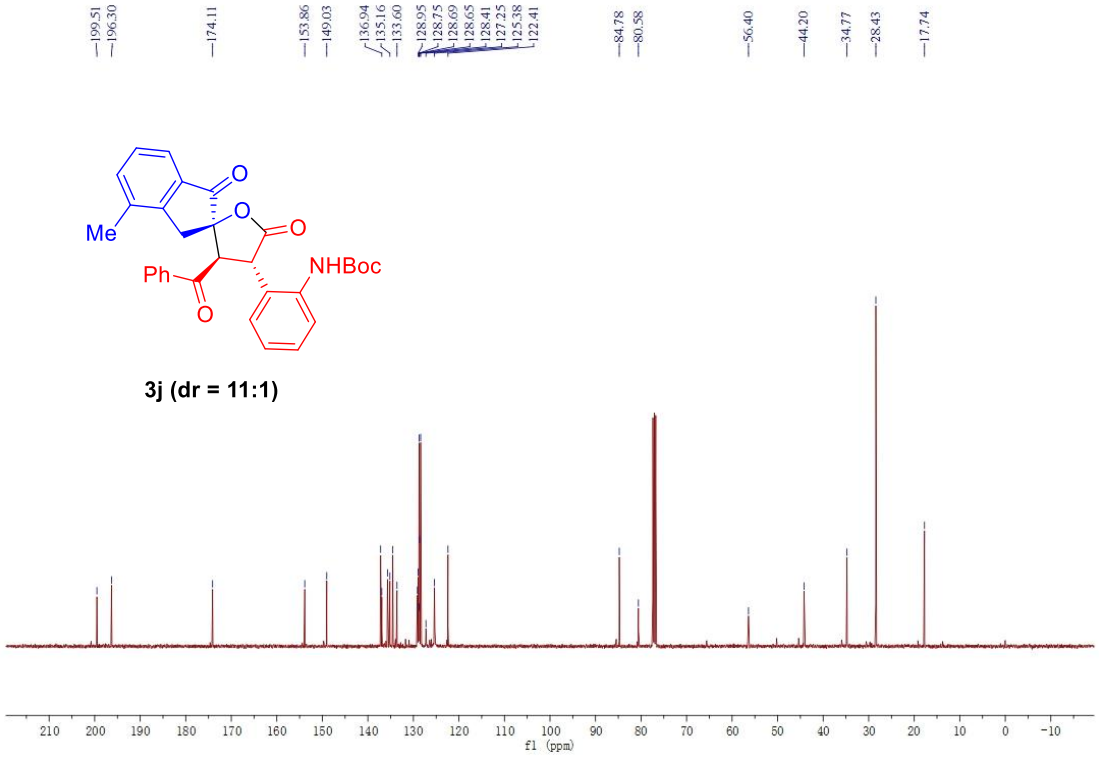
^{13}C NMR (101 MHz, $CDCl_3$)



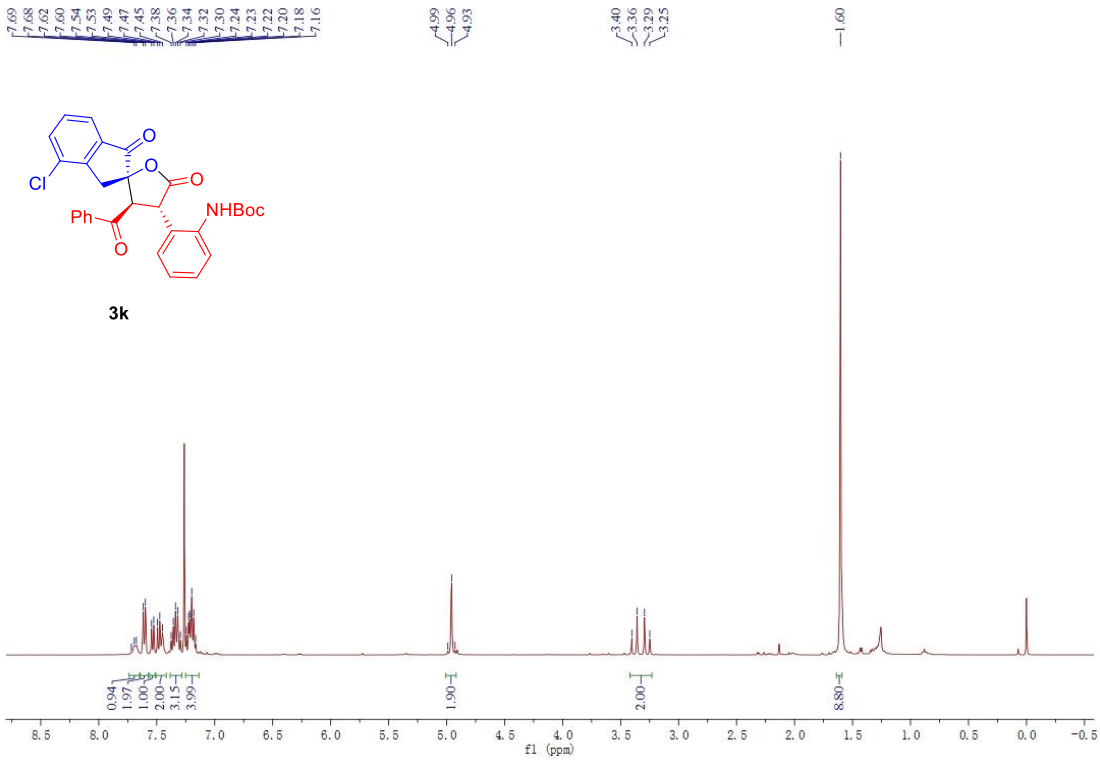
^{1}H NMR (400 MHz, $CDCl_3$)



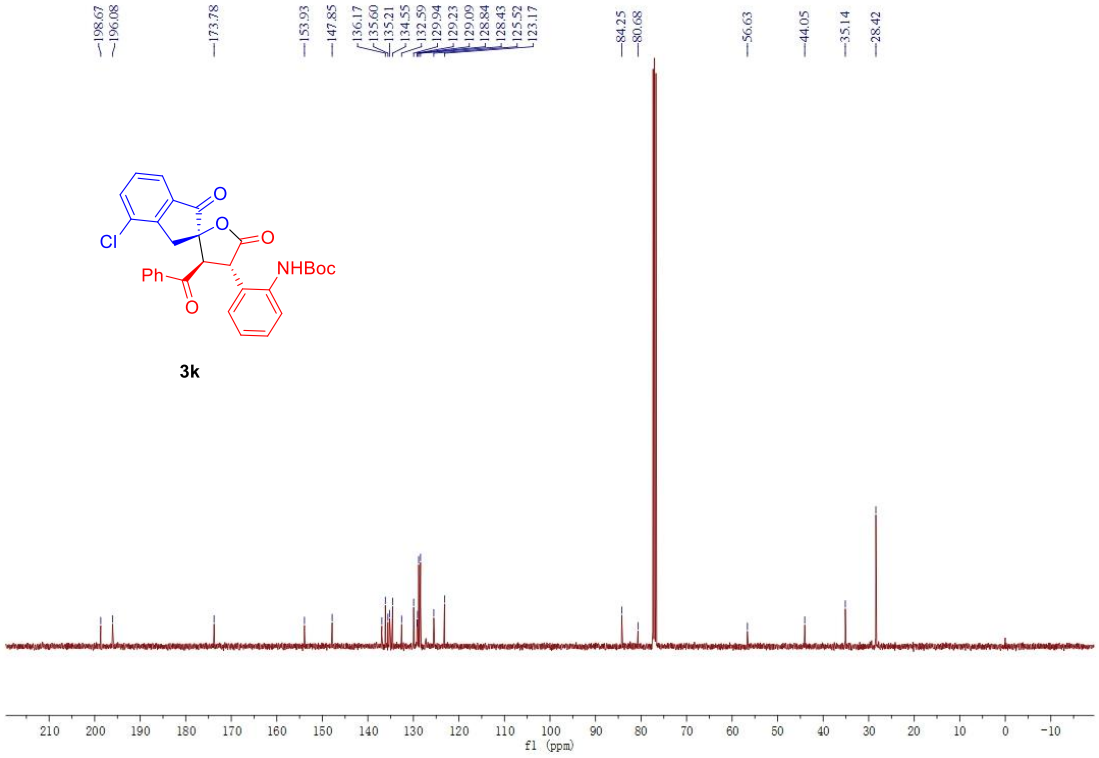
^{13}C NMR (101 MHz, $CDCl_3$)



^{1}H NMR (400 MHz, $CDCl_3$)

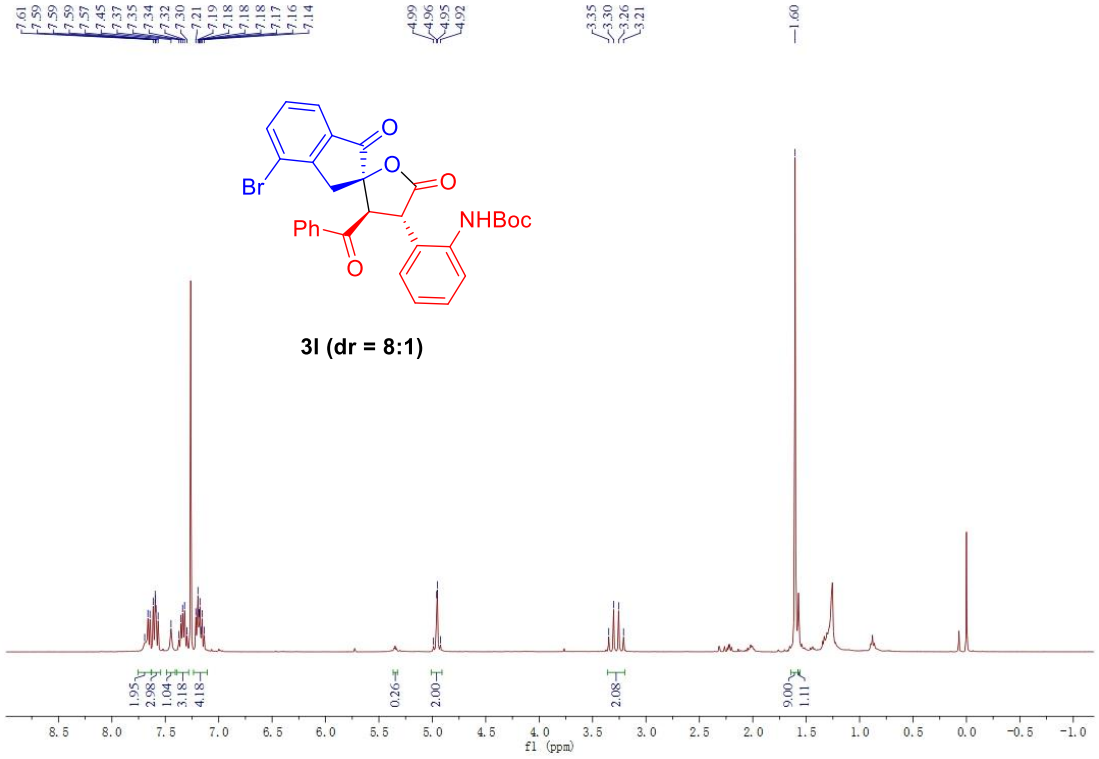


^{13}C NMR (101 MHz, $CDCl_3$)



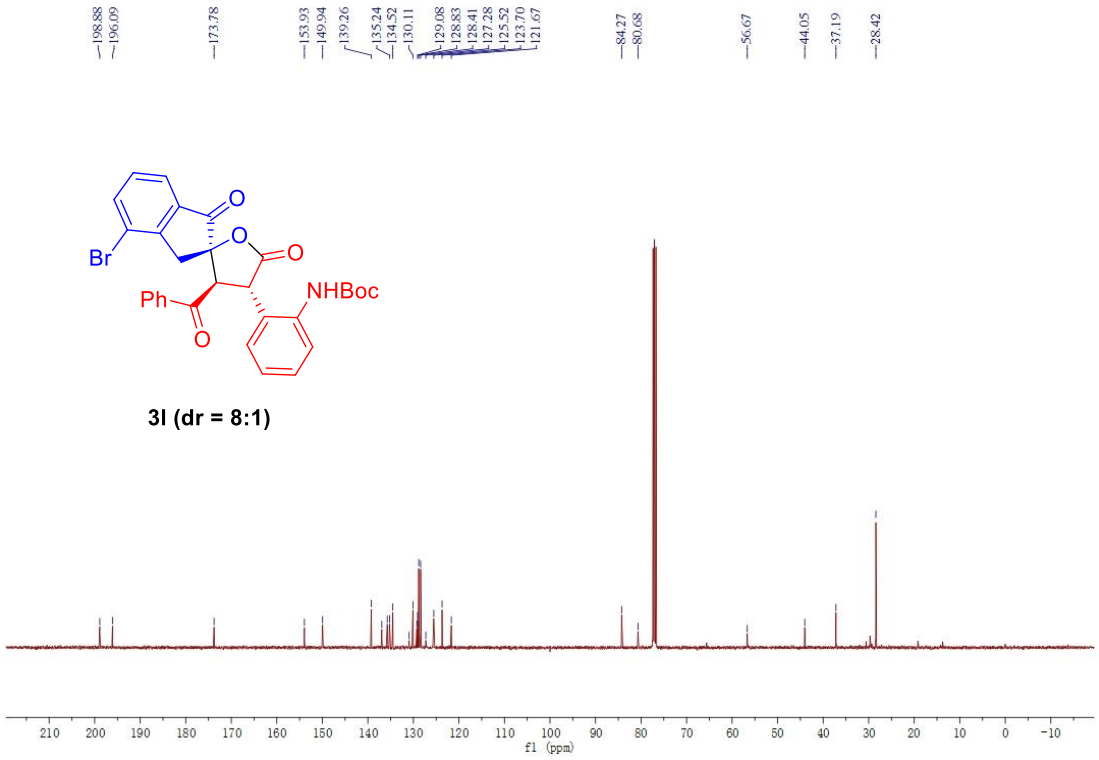
3k

^{1}H NMR (400 MHz, $CDCl_3$)



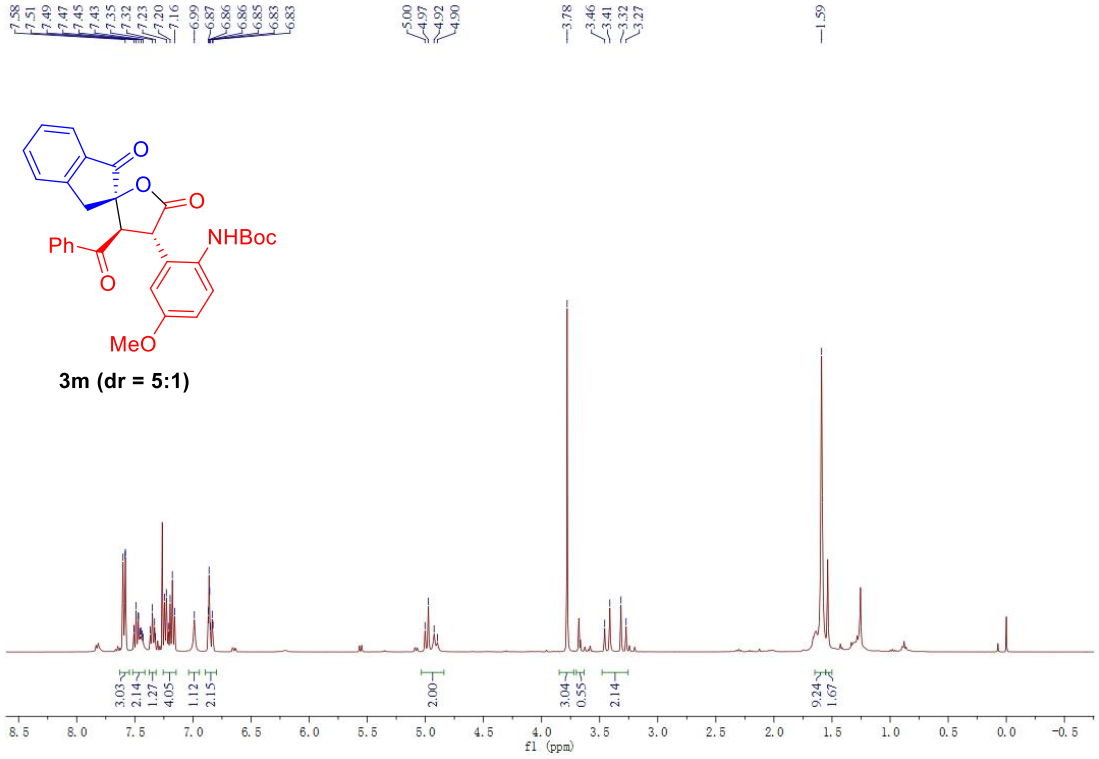
3l (dr = 8:1)

^{13}C NMR (101 MHz, $CDCl_3$)



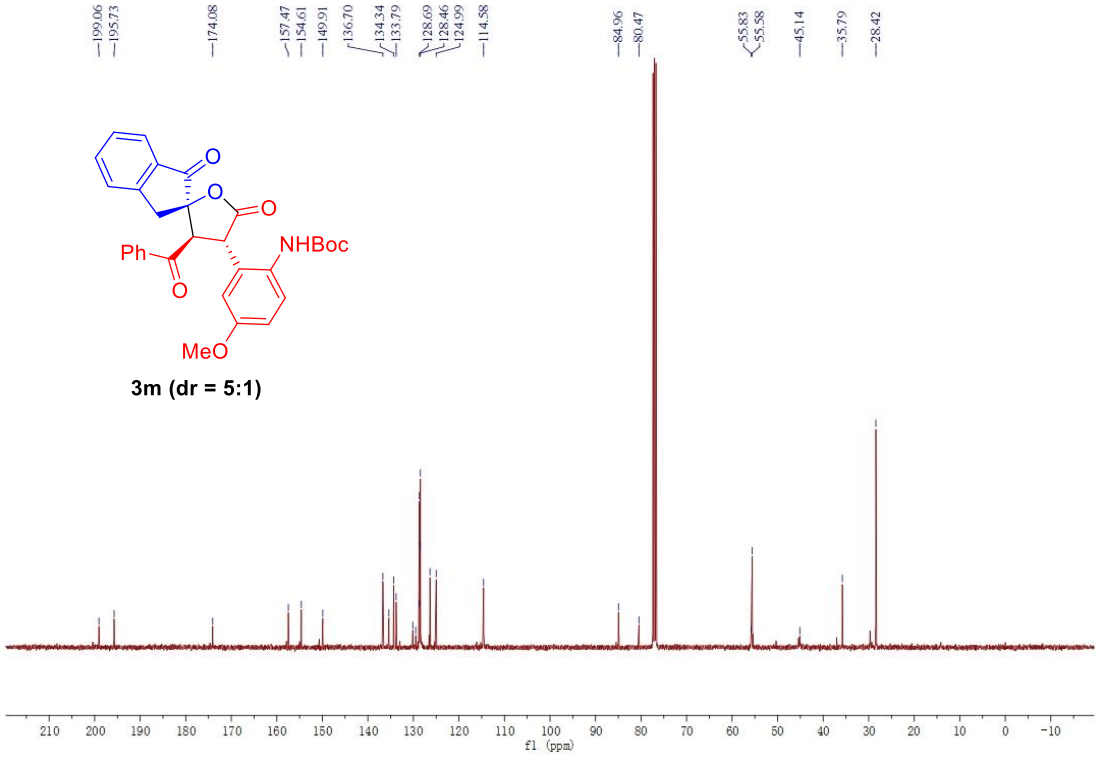
3l (dr = 8:1)

^{1}H NMR (400 MHz, $CDCl_3$)

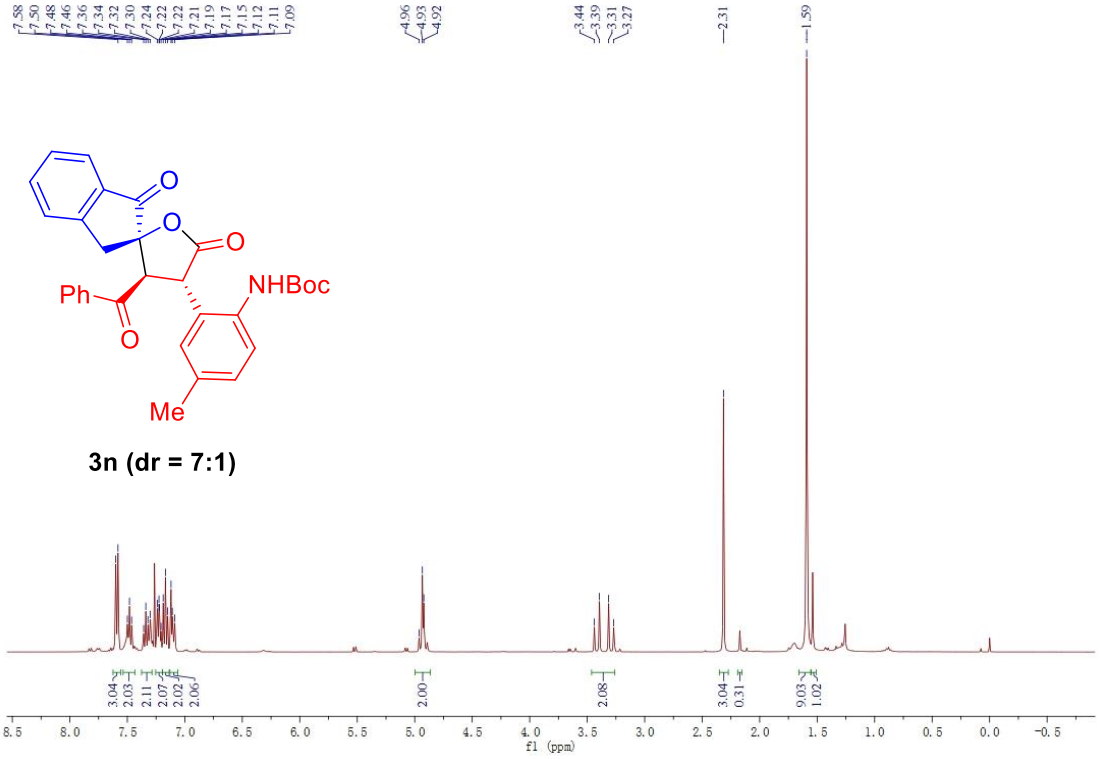


3m (dr = 5:1)

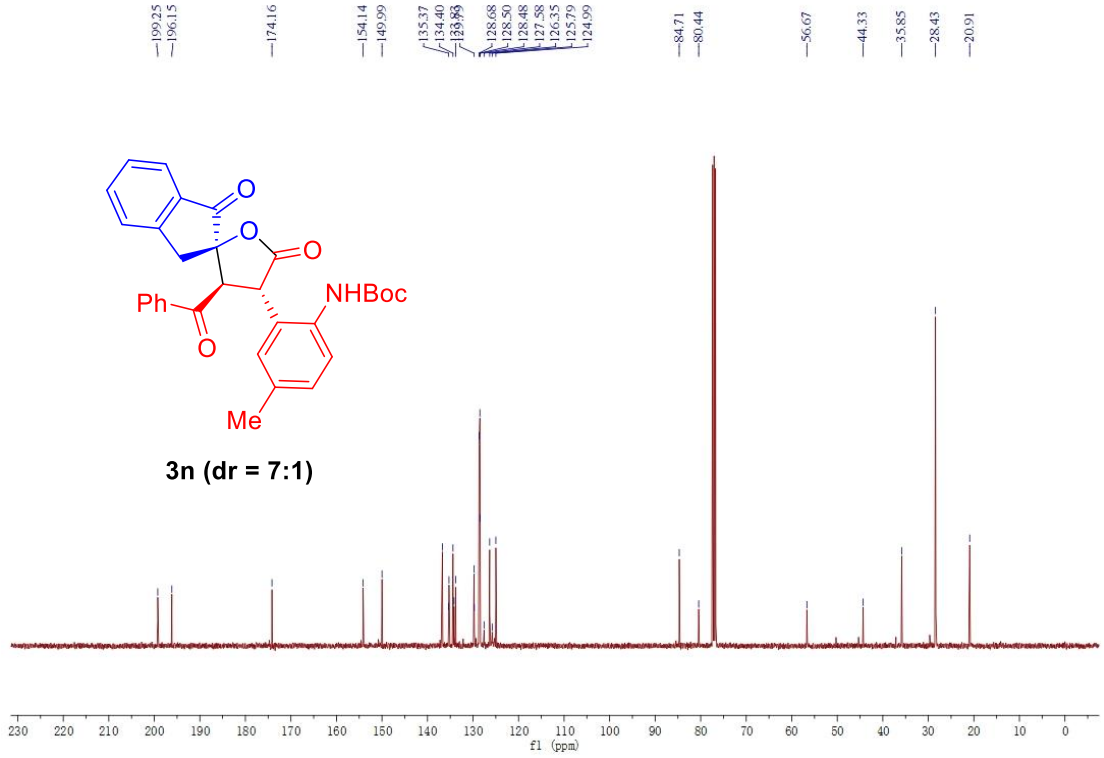
^{13}C NMR (101 MHz, $CDCl_3$)



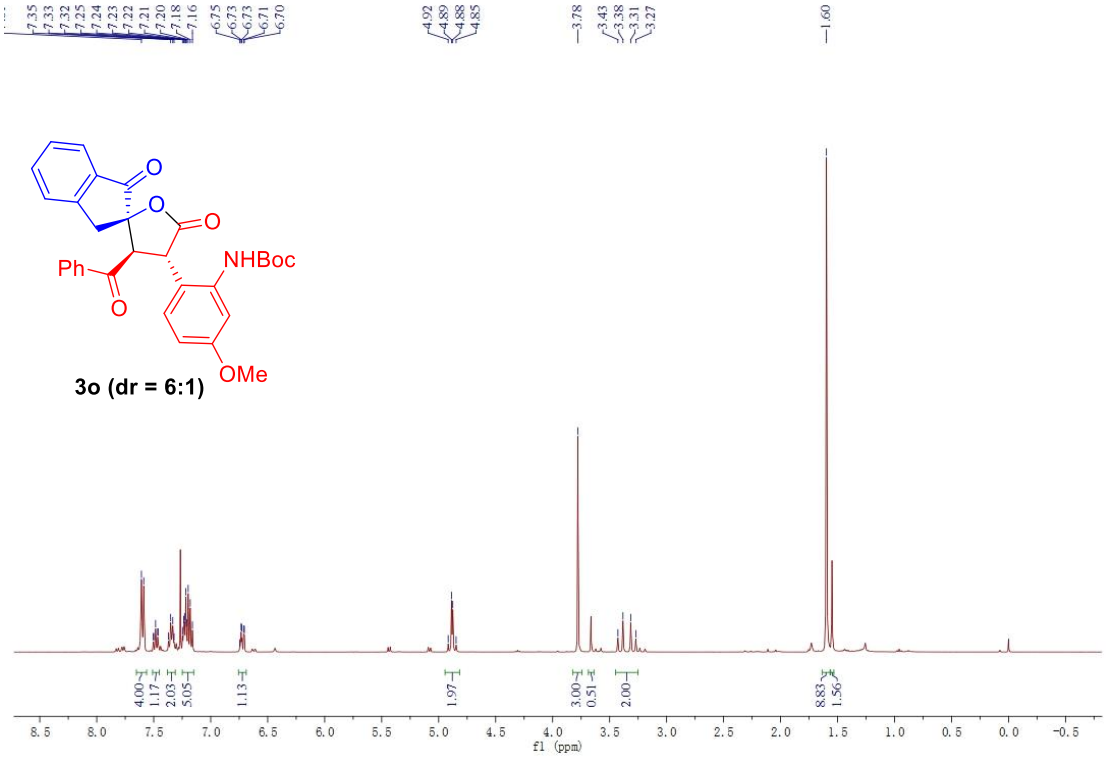
^{1}H NMR (400 MHz, $CDCl_3$)



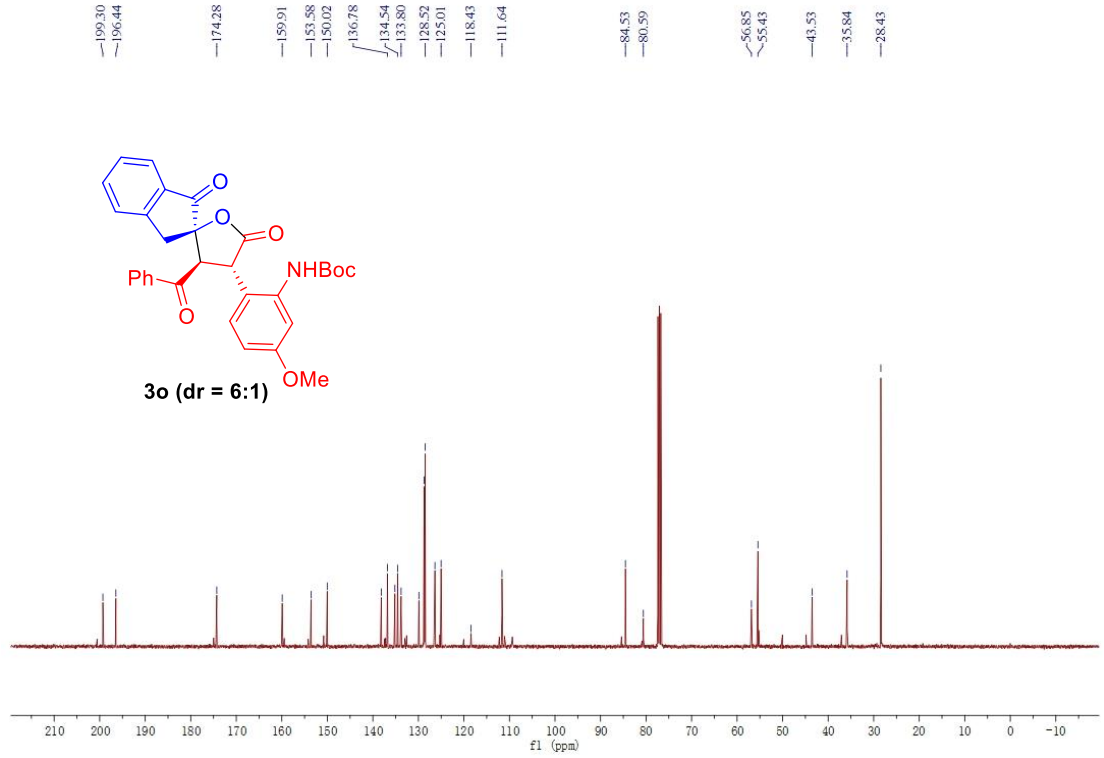
^{13}C NMR (101 MHz, $CDCl_3$)



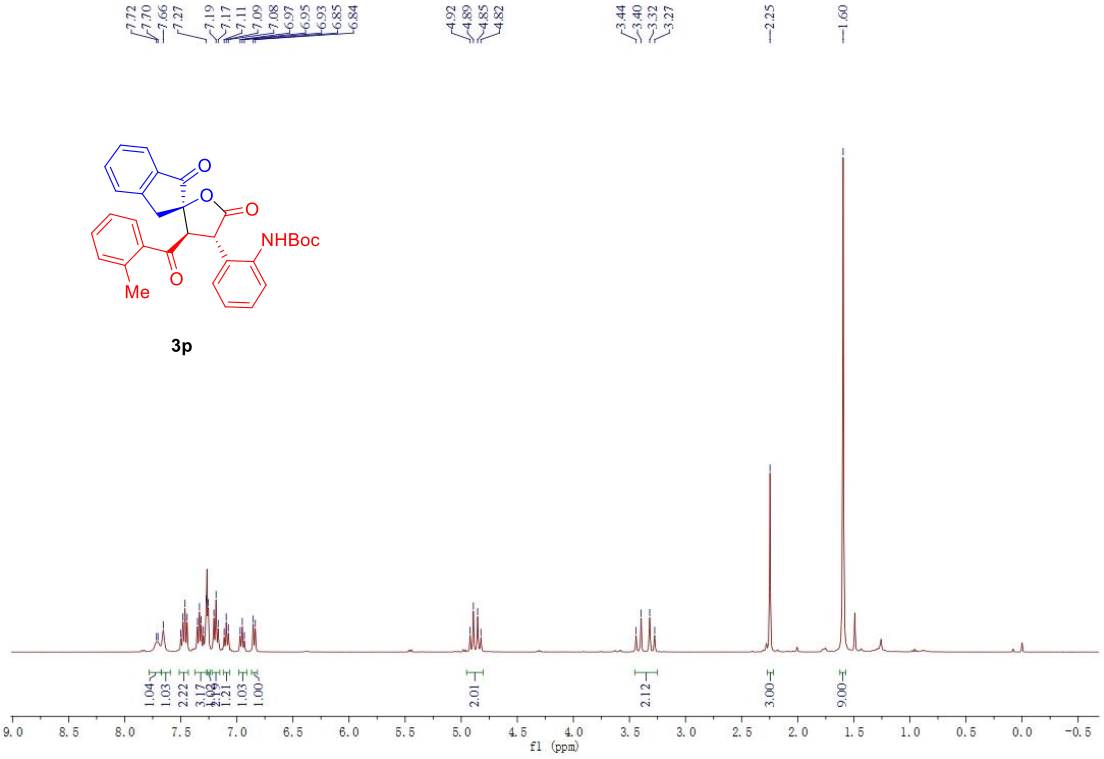
^{1}H NMR (400 MHz, $CDCl_3$)



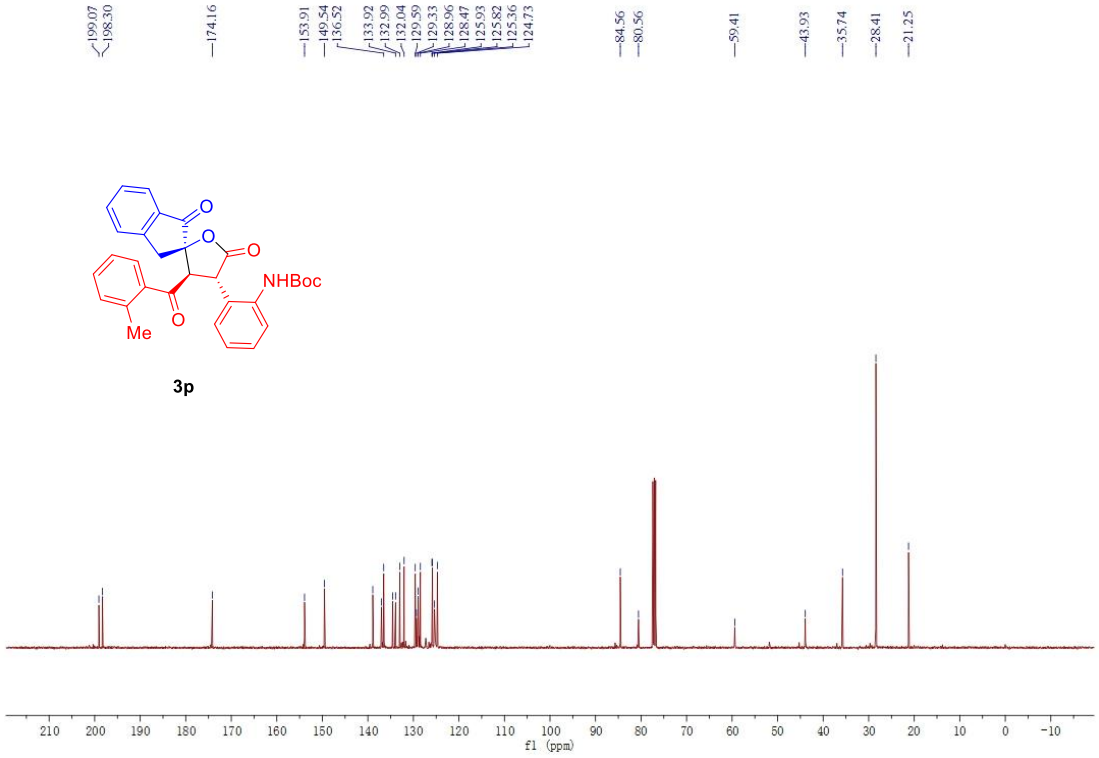
^{13}C NMR (101 MHz, $CDCl_3$)



^{1}H NMR (400 MHz, $CDCl_3$)

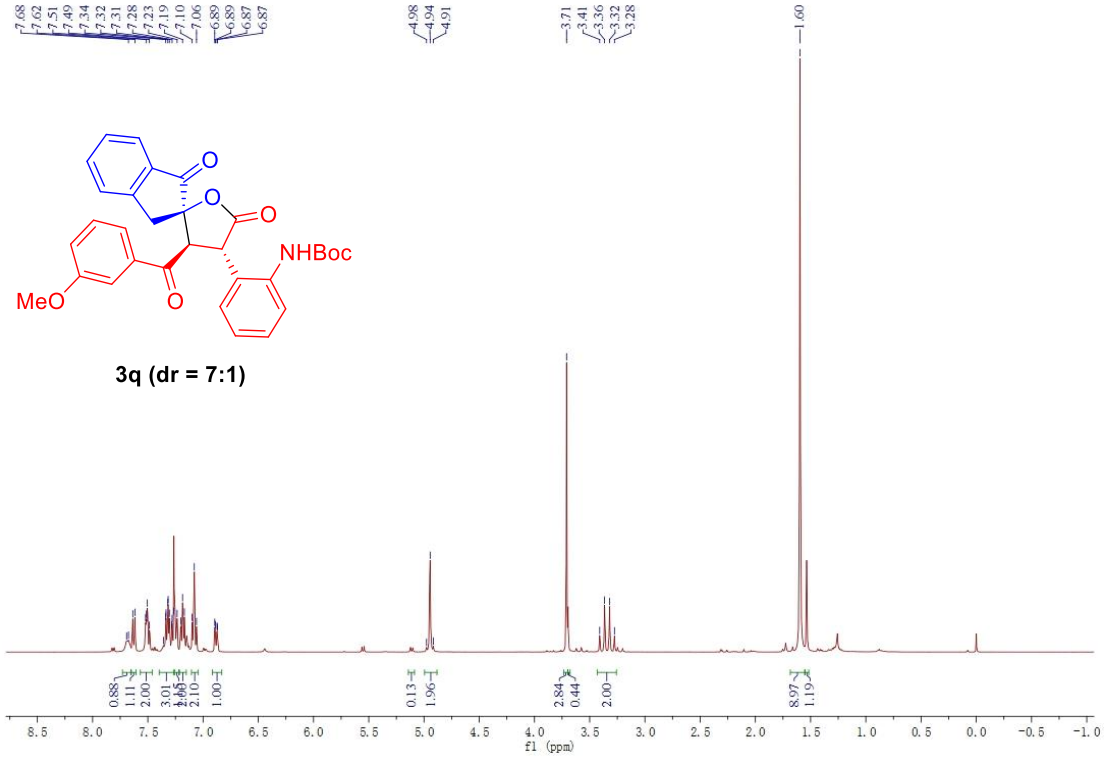


^{13}C NMR (101 MHz, $CDCl_3$)



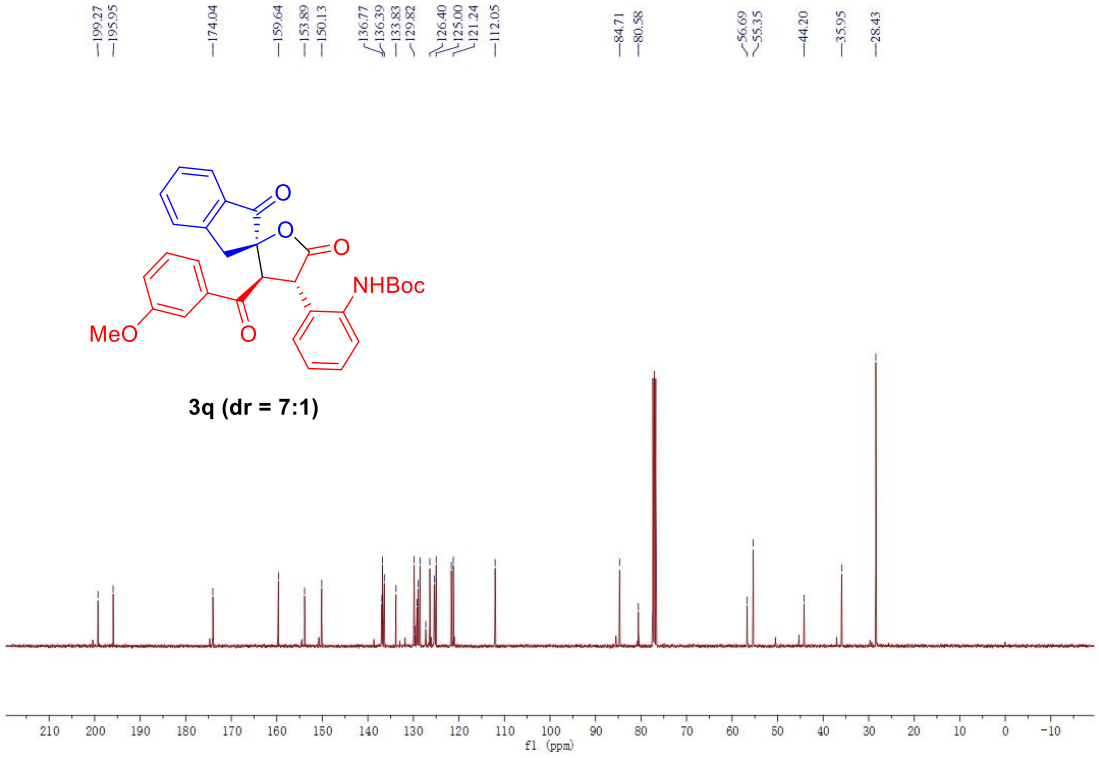
3p

^{1}H NMR (400 MHz, $CDCl_3$)



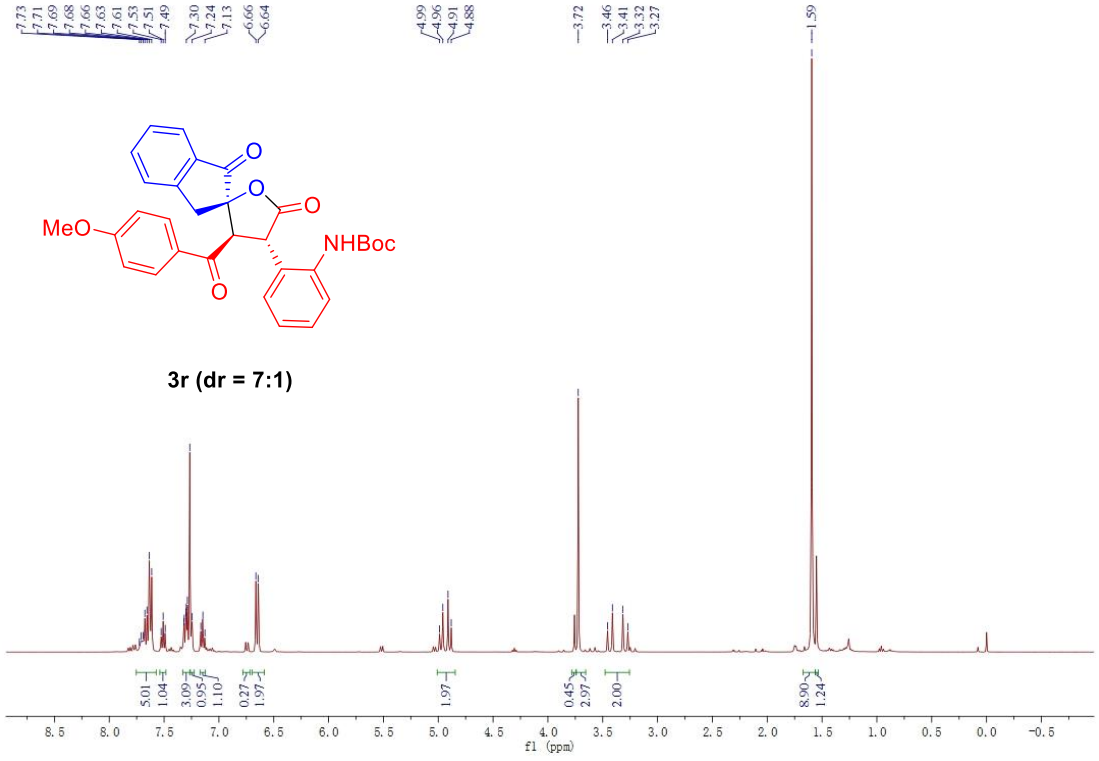
3q (dr = 7:1)

^{13}C NMR (101 MHz, $CDCl_3$)



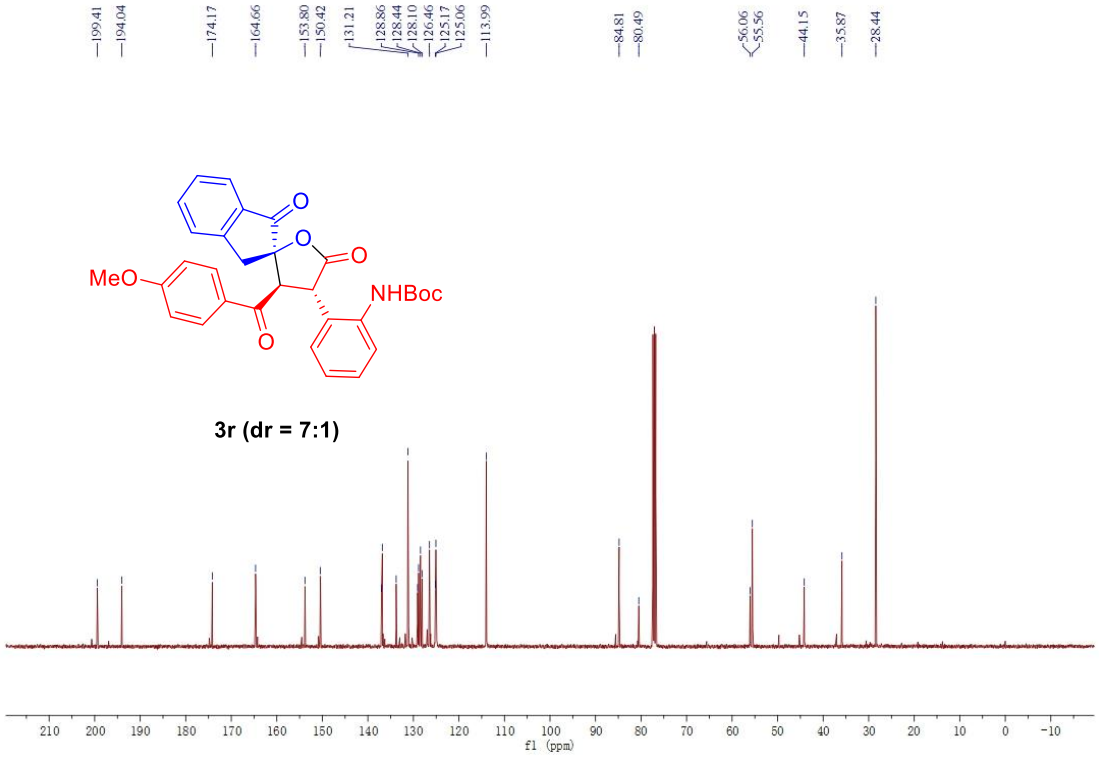
3q (dr = 7:1)

^{1}H NMR (400 MHz, $CDCl_3$)

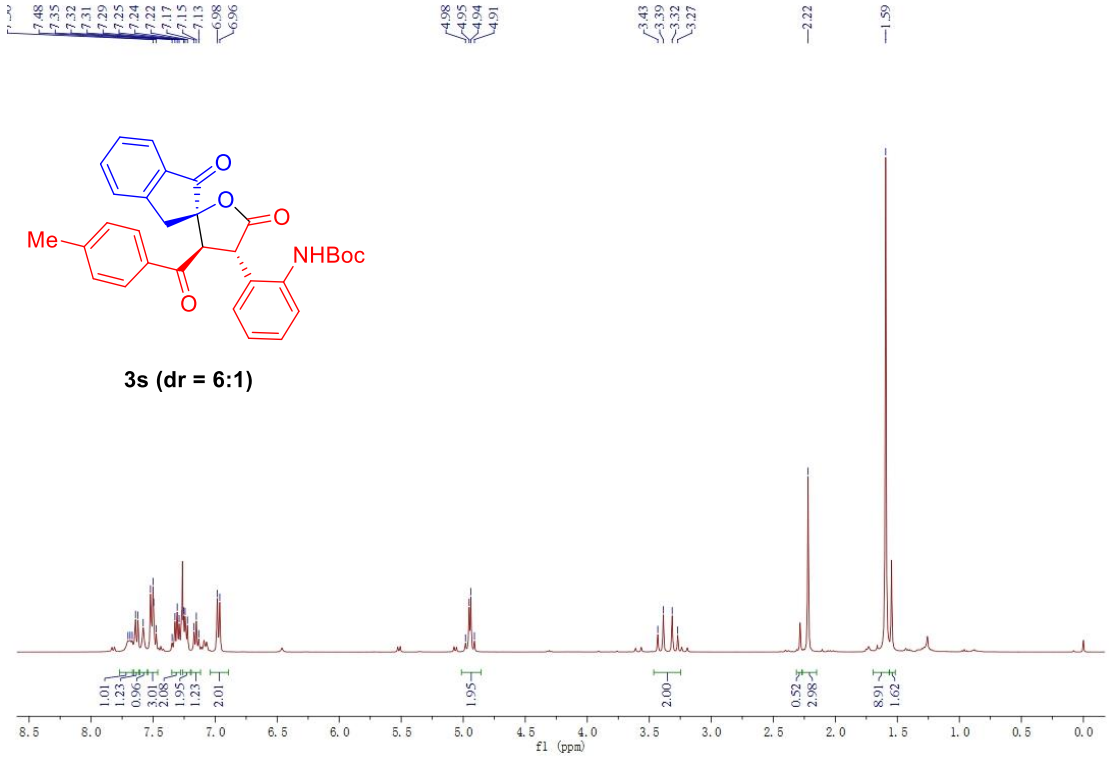


3r (dr = 7:1)

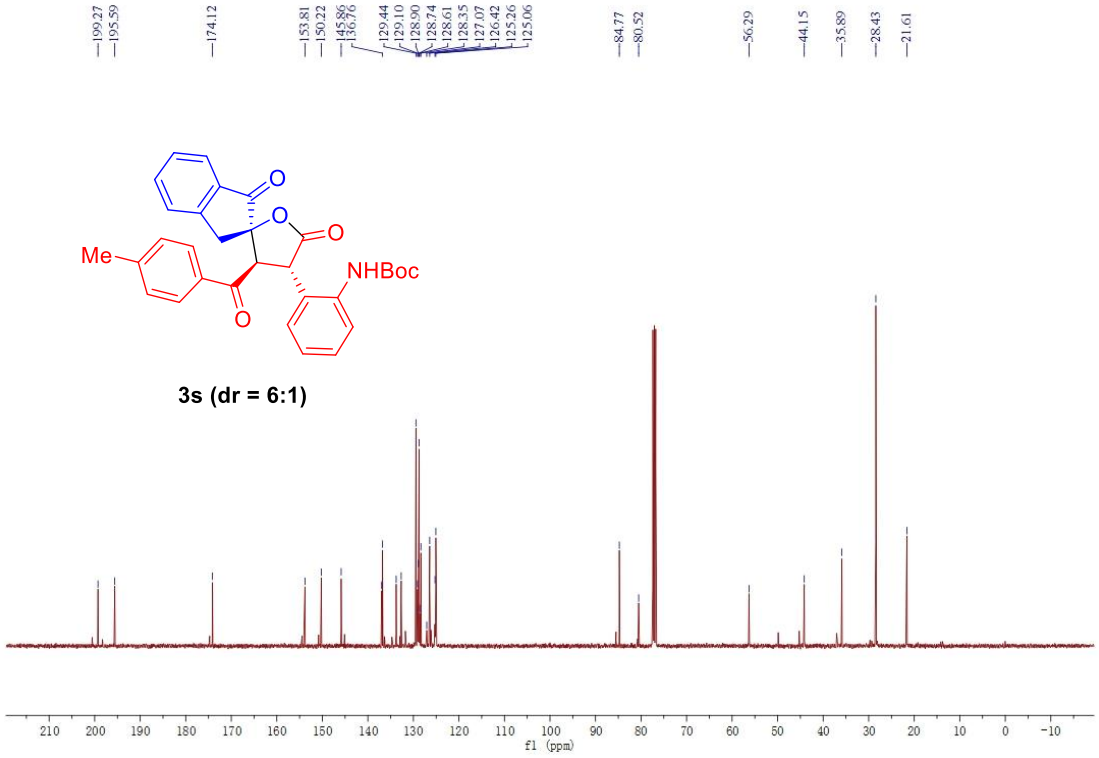
^{13}C NMR (101 MHz, $CDCl_3$)



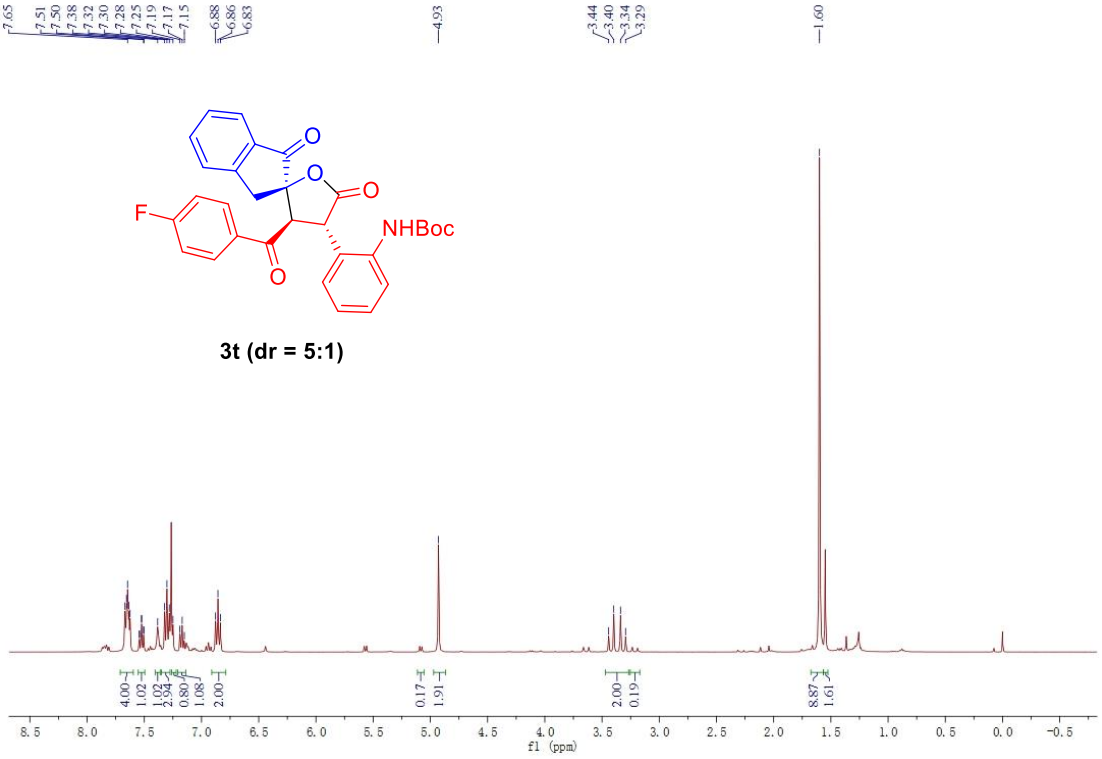
^{1}H NMR (400 MHz, $CDCl_3$)



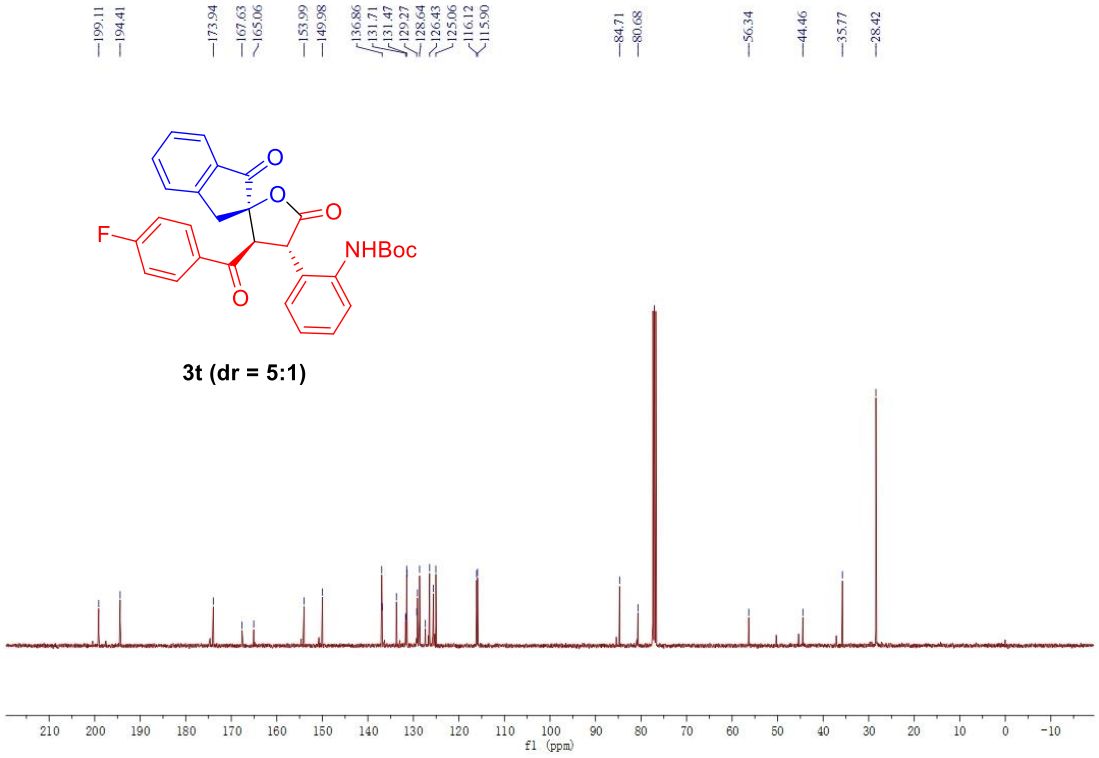
^{13}C NMR (101 MHz, $CDCl_3$)



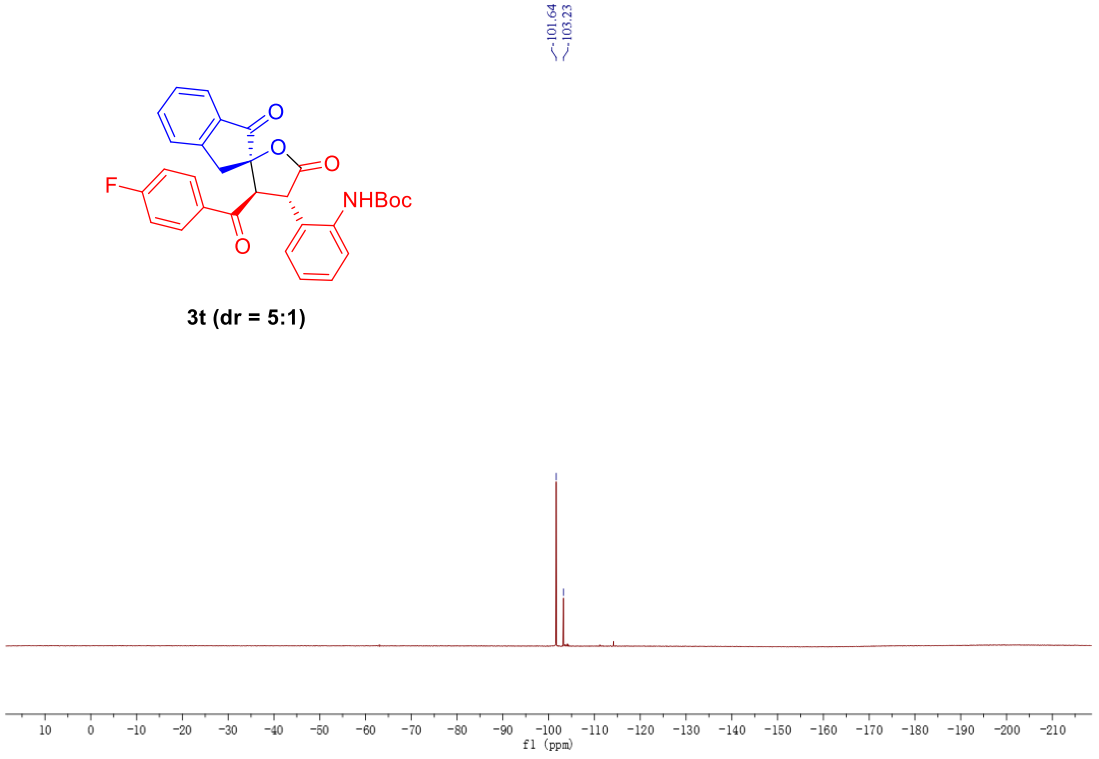
^{1}H NMR (400 MHz, $CDCl_3$)



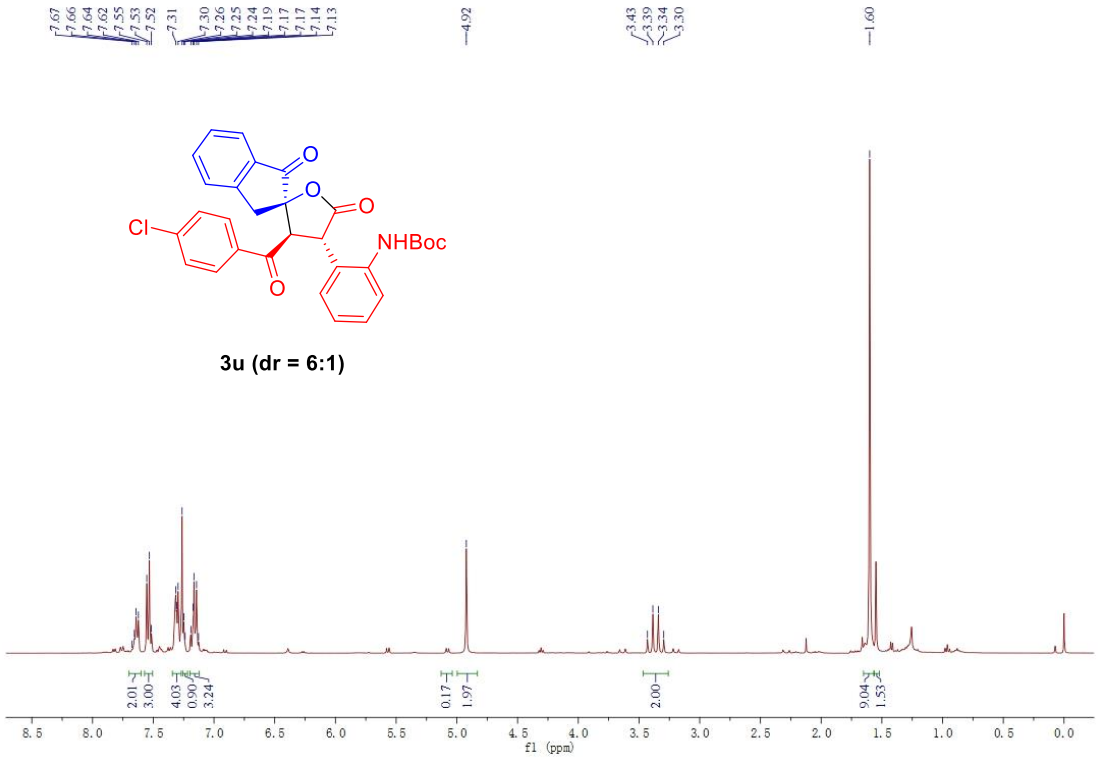
^{13}C NMR (101 MHz, $CDCl_3$)



^{19}F NMR (376 MHz, $CDCl_3$)

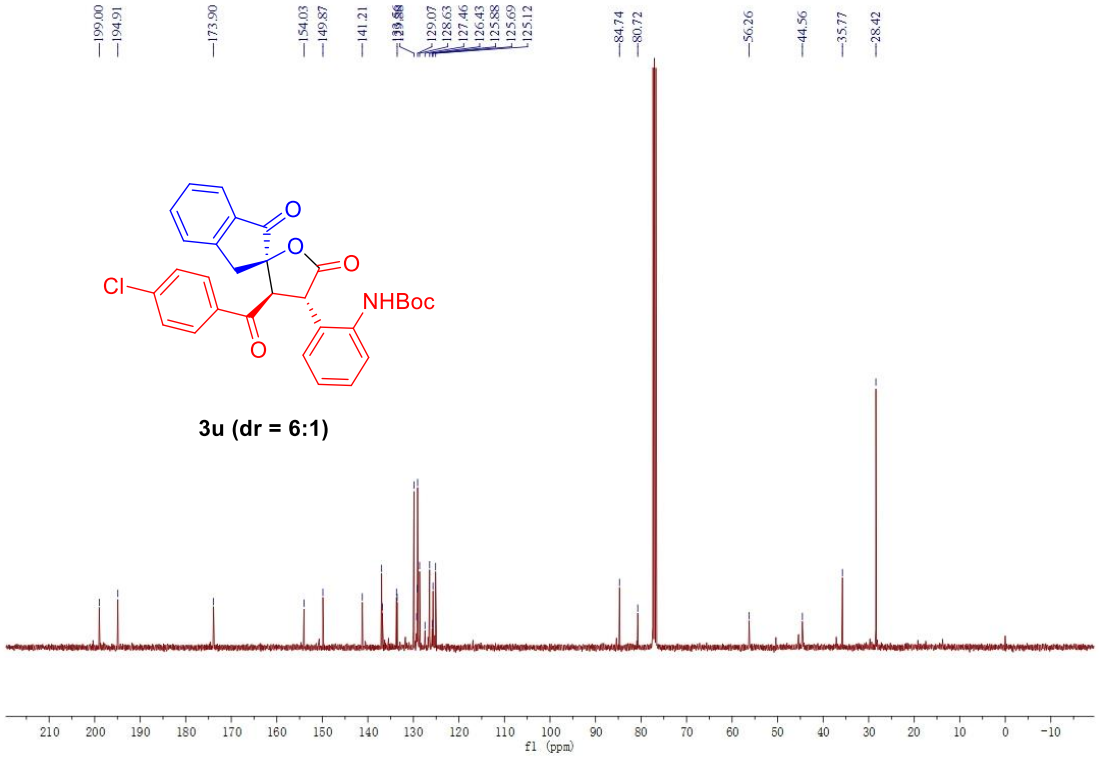


^{1}H NMR (400 MHz, $CDCl_3$)



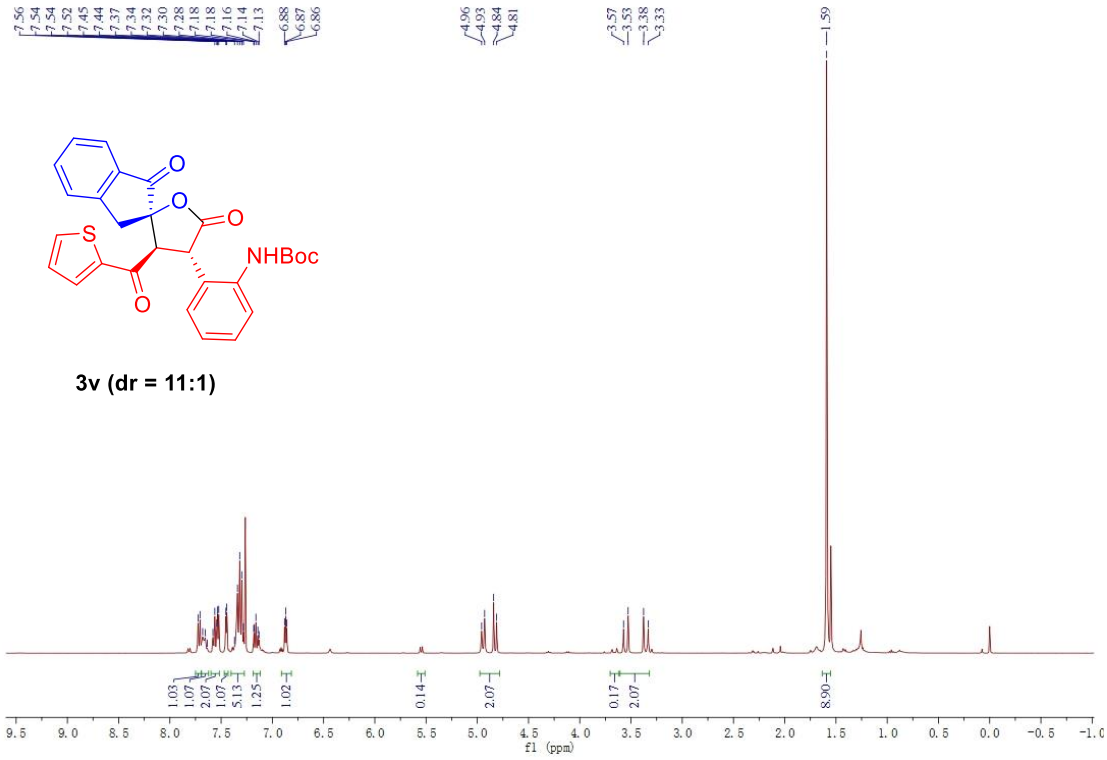
3u (dr = 6:1)

^{13}C NMR (101 MHz, $CDCl_3$)



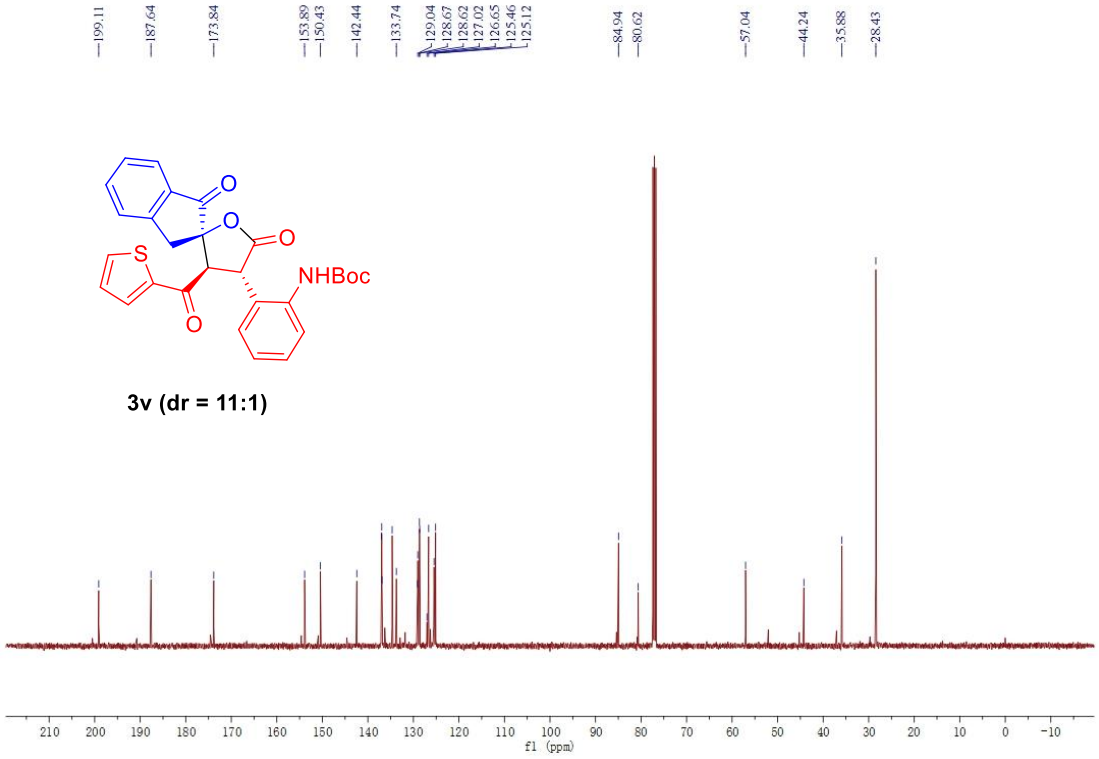
3u (dr = 6:1)

^{1}H NMR (400 MHz, $CDCl_3$)



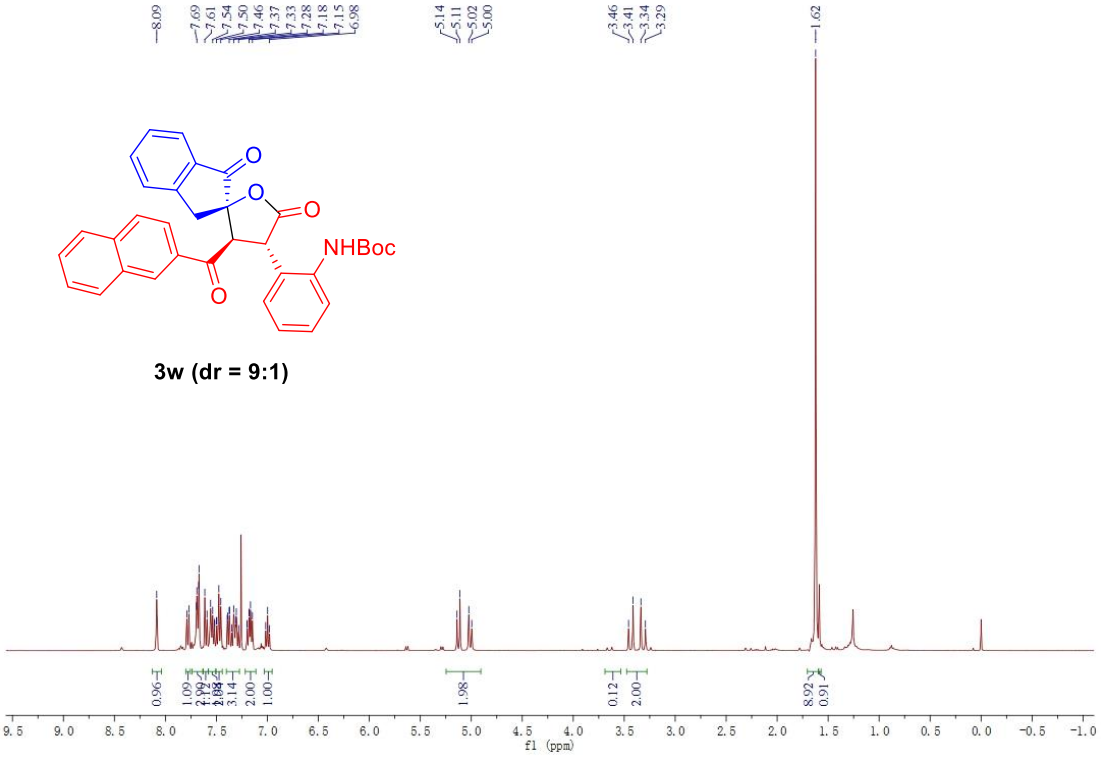
3v (dr = 11:1)

^{13}C NMR (101 MHz, $CDCl_3$)

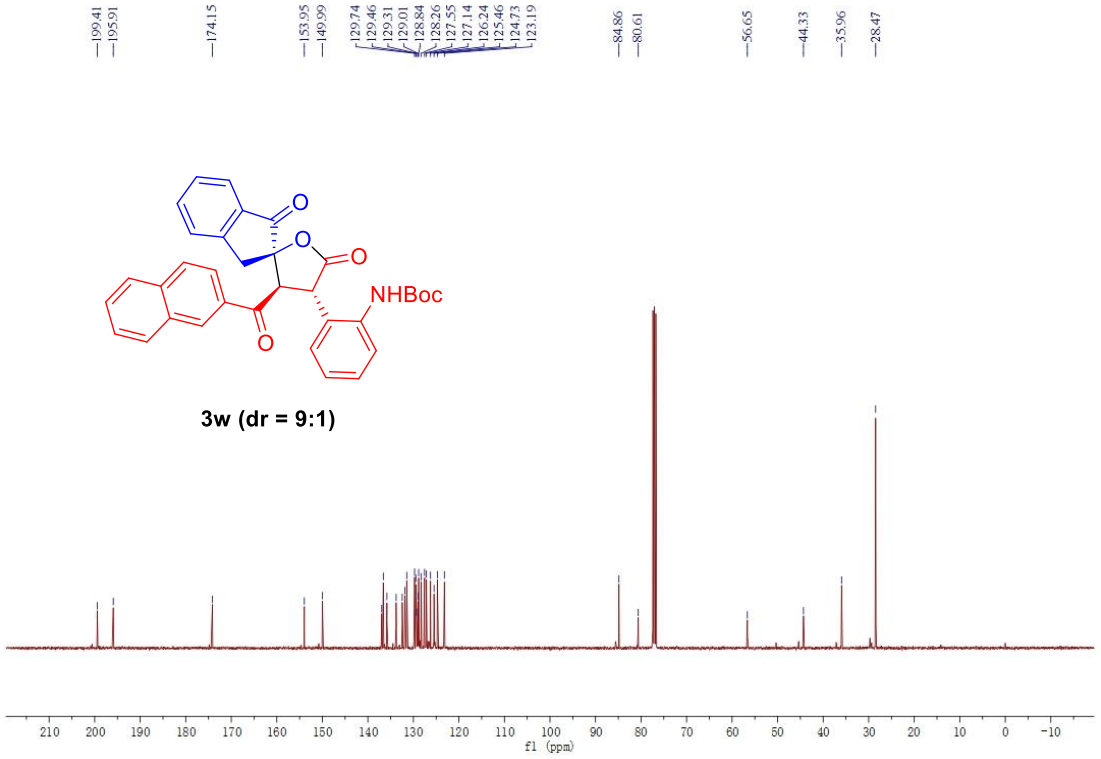


3v (dr = 11:1)

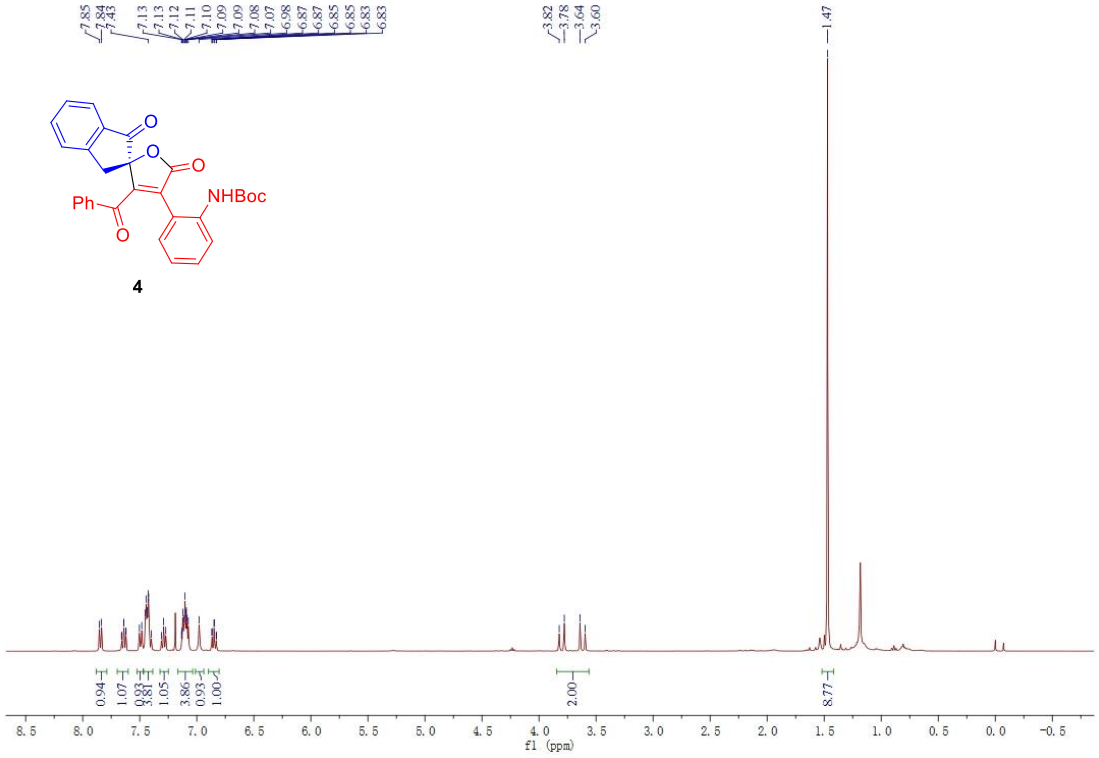
^{1}H NMR (400 MHz, $CDCl_3$)



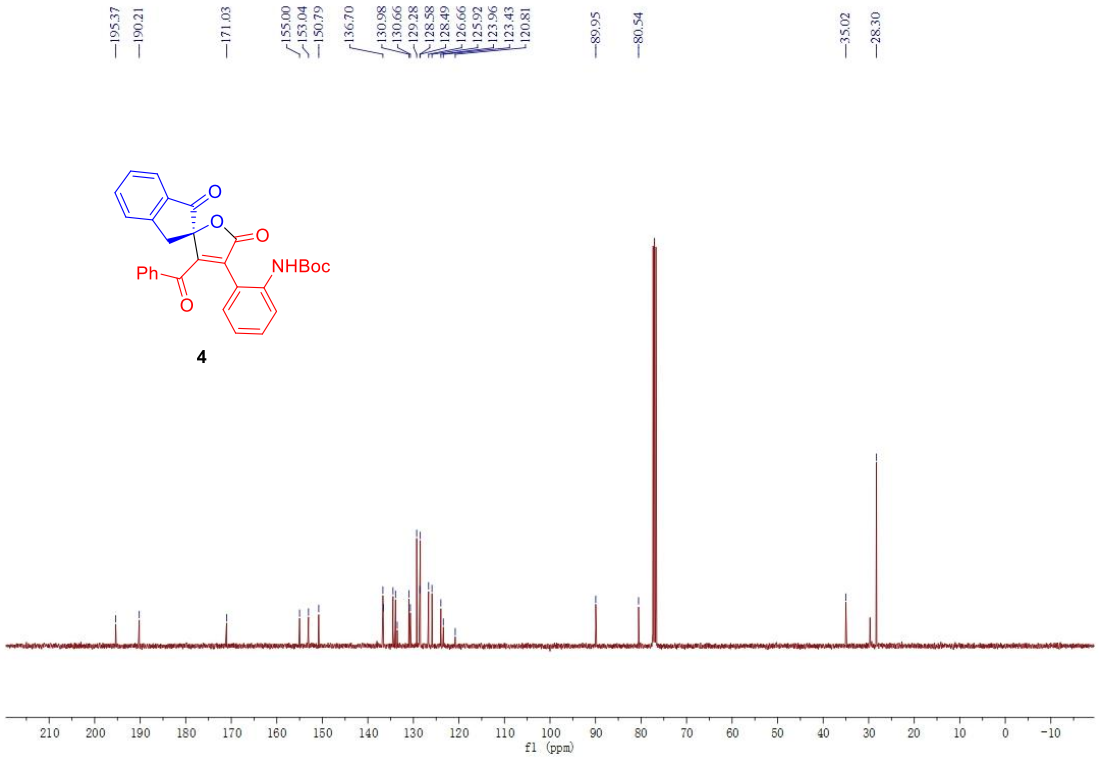
^{13}C NMR (101 MHz, $CDCl_3$)



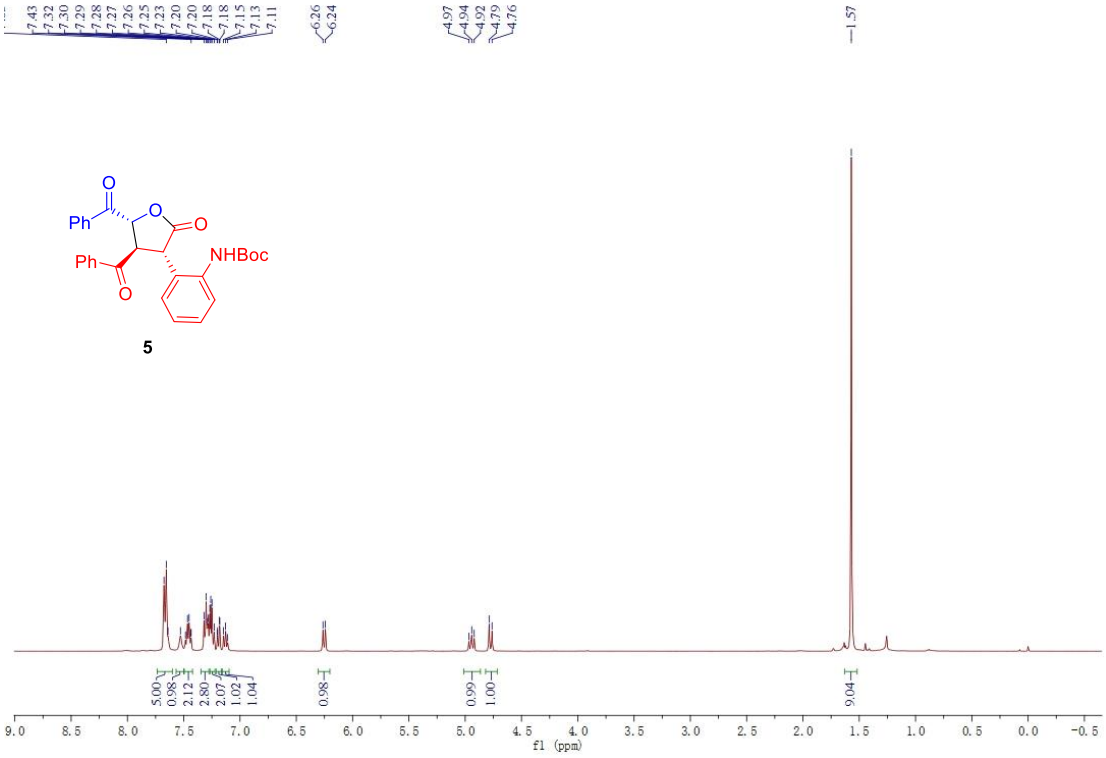
^{1}H NMR (400 MHz, $CDCl_3$)



^{13}C NMR (101 MHz, $CDCl_3$)



^{1}H NMR (400 MHz, $CDCl_3$)



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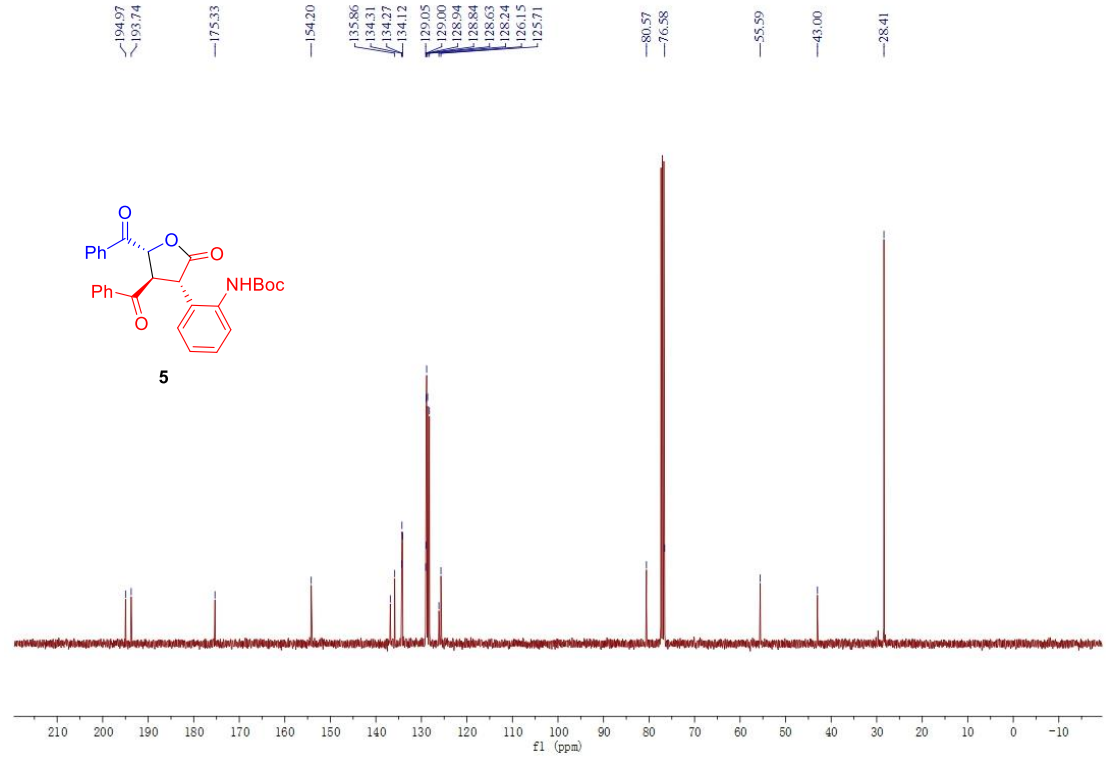
^{13}C NMR (101 MHz, $CDCl_3$)



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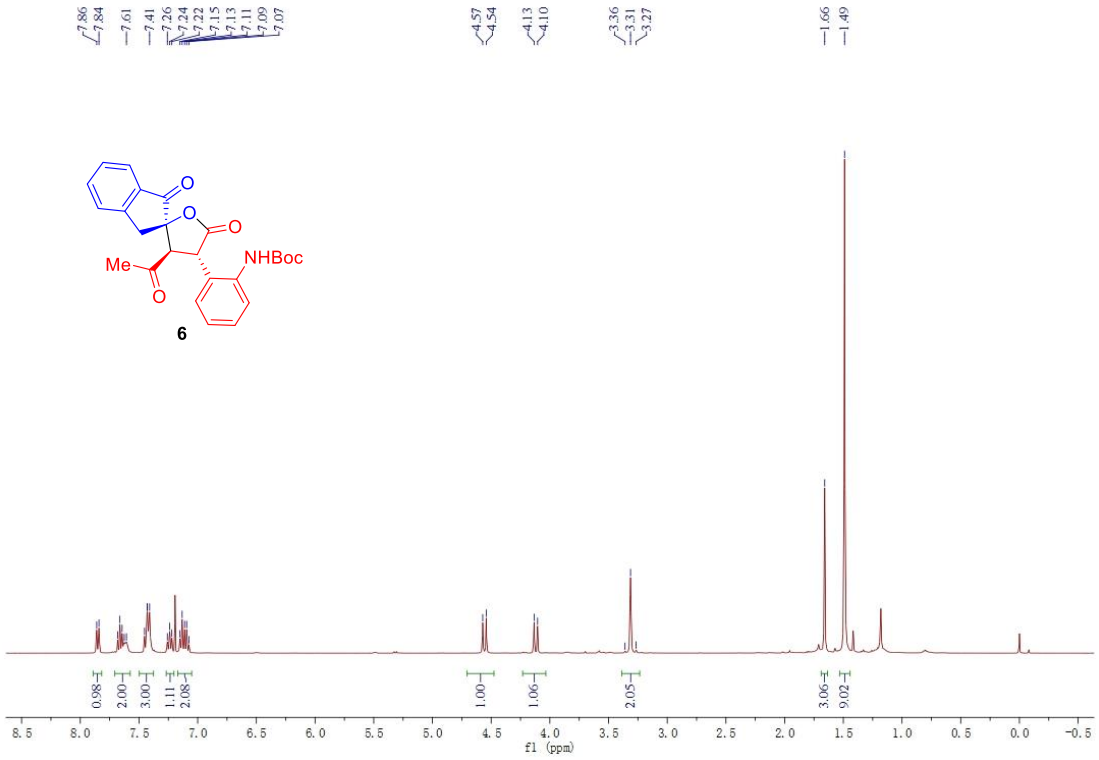
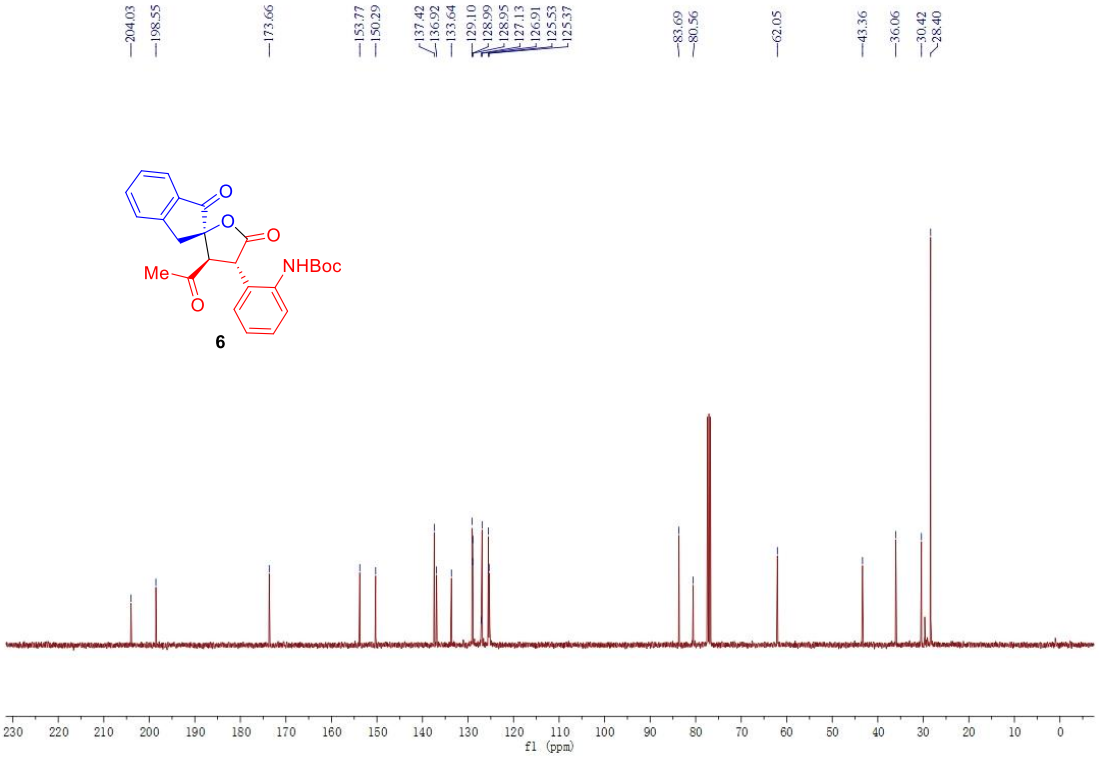
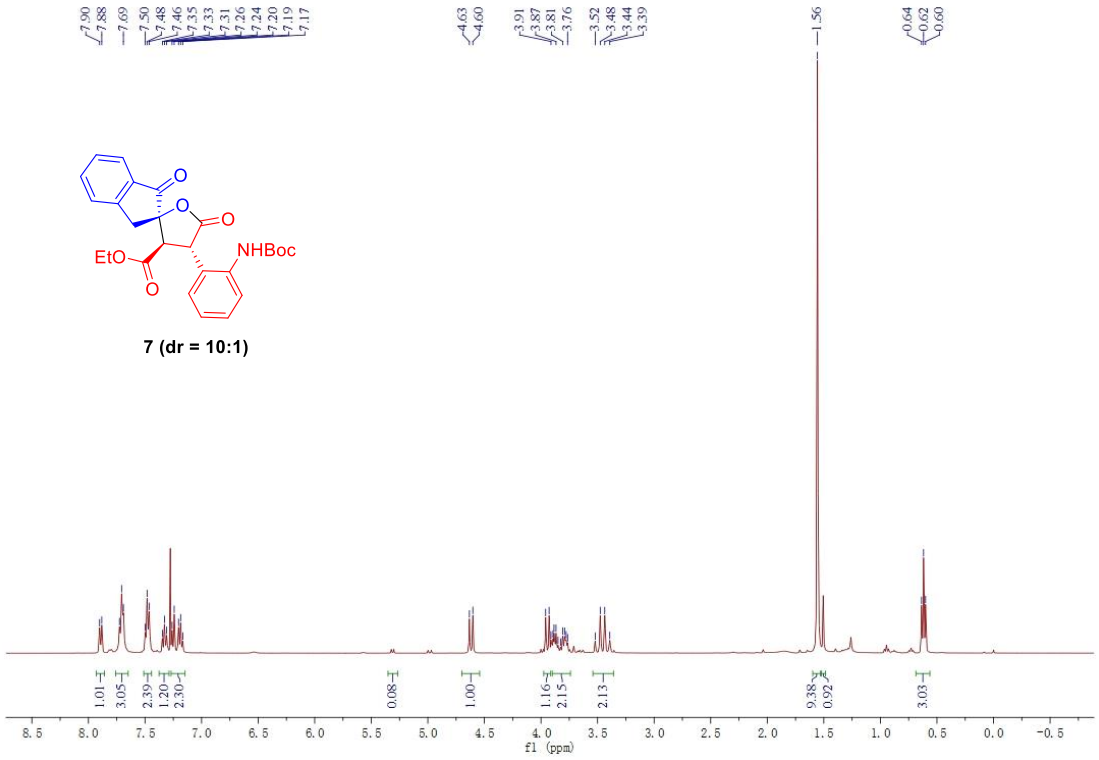
^{1}H NMR (400 MHz, $CDCl_3$)



^{13}C NMR (101 MHz, $CDCl_3$)

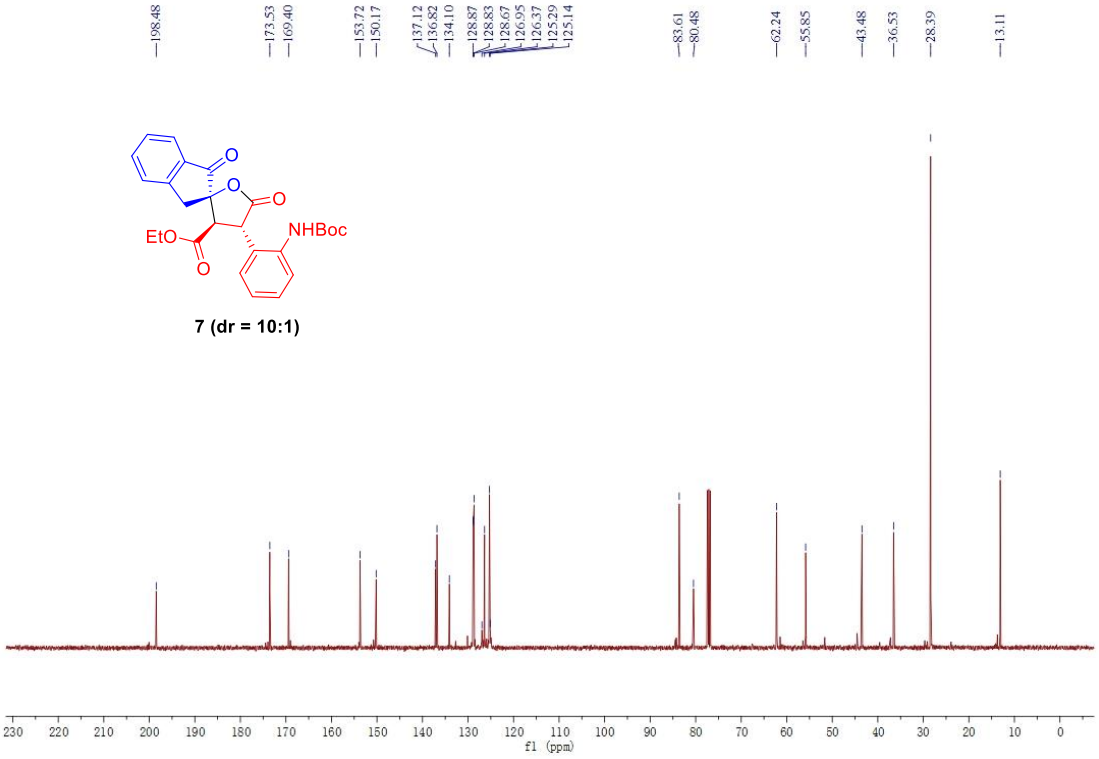


^{1}H NMR (400 MHz, $CDCl_3$)



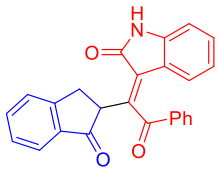
7 (dr = 10:1)

^{13}C NMR (101 MHz, $CDCl_3$)



7 (dr = 10:1)

^{1}H NMR (400 MHz, $CDCl_3$)



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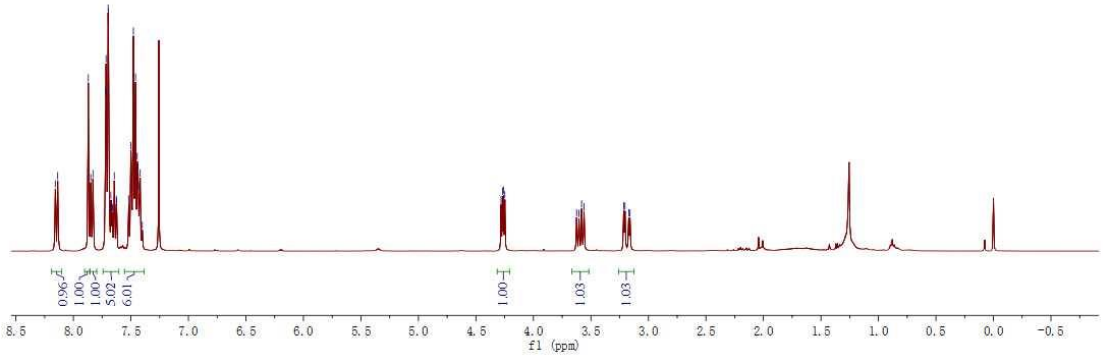
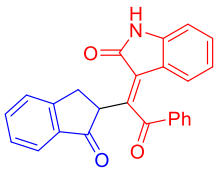


^{13}C NMR (101 MHz, $CDCl_3$)



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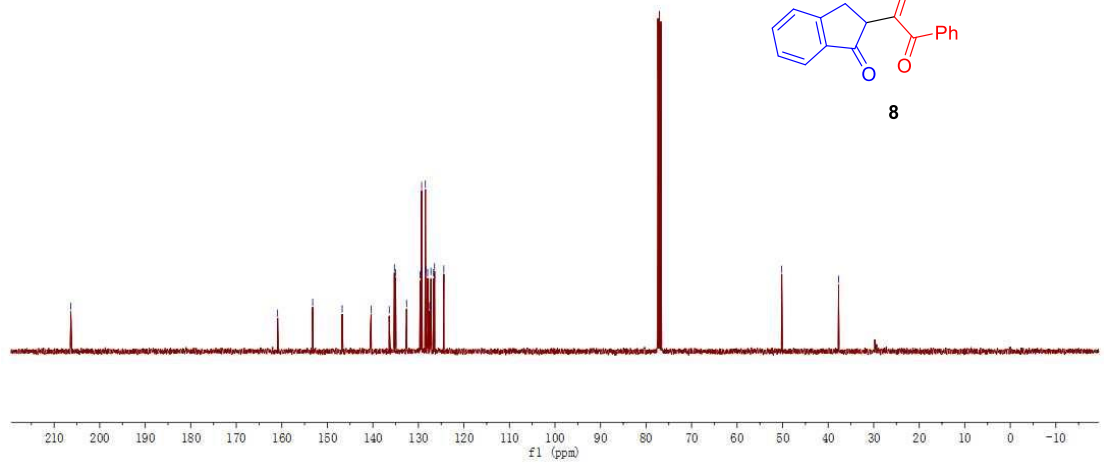
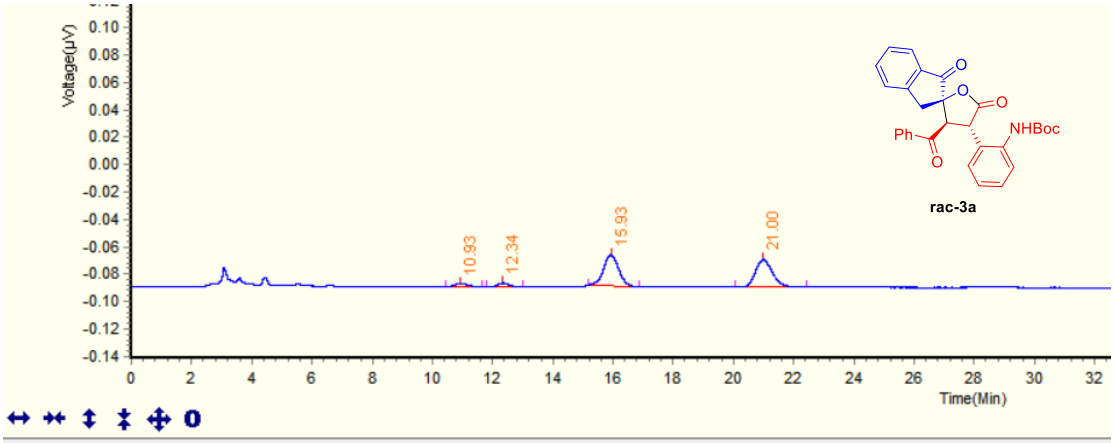
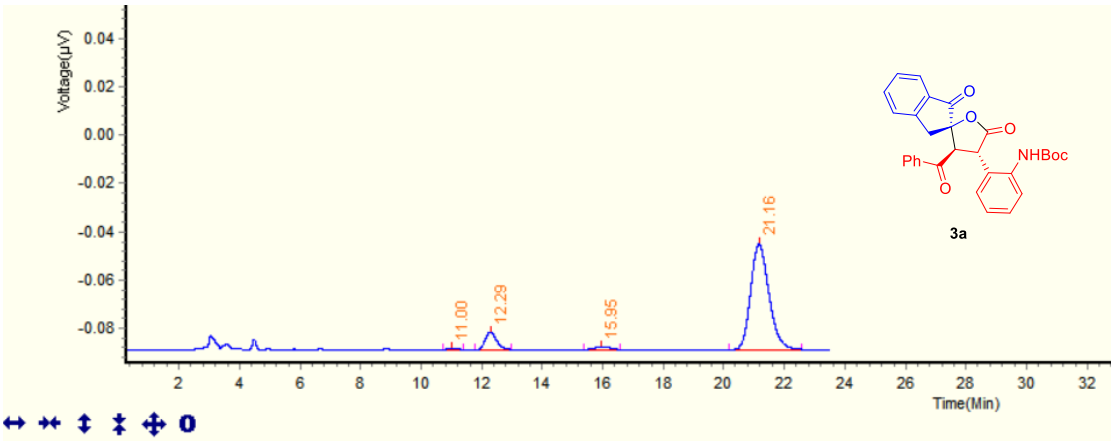


HPLC spectra of compounds



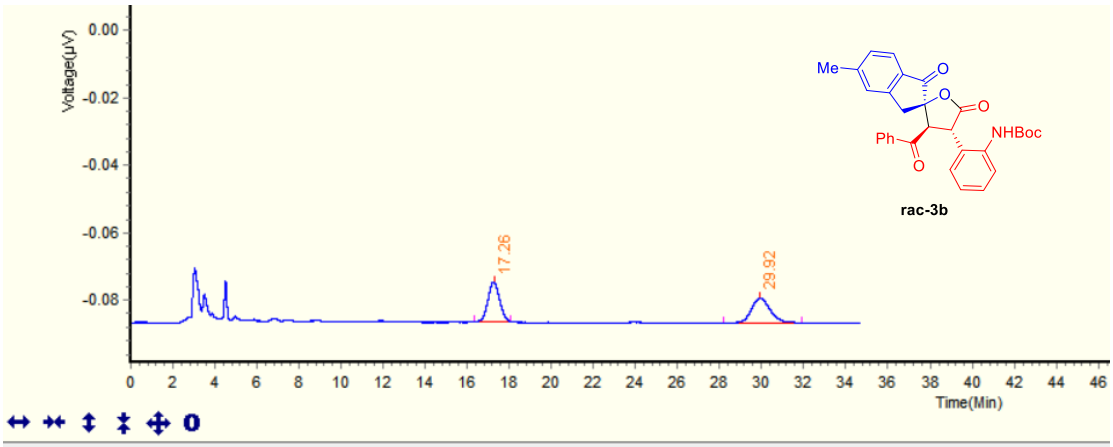
Integration Result | Calculation Result | TimeTable

No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	
1	10.93	35899	1176	4.15%	1.208	BB
2	12.34	35307	1445	4.08%	1.203	BB
3	15.93	395138	11343	45.70%	1.707	BB
4	21.00	398314	10135	46.07%	2.377	BB
Total		864,658	24,099	100.00%		



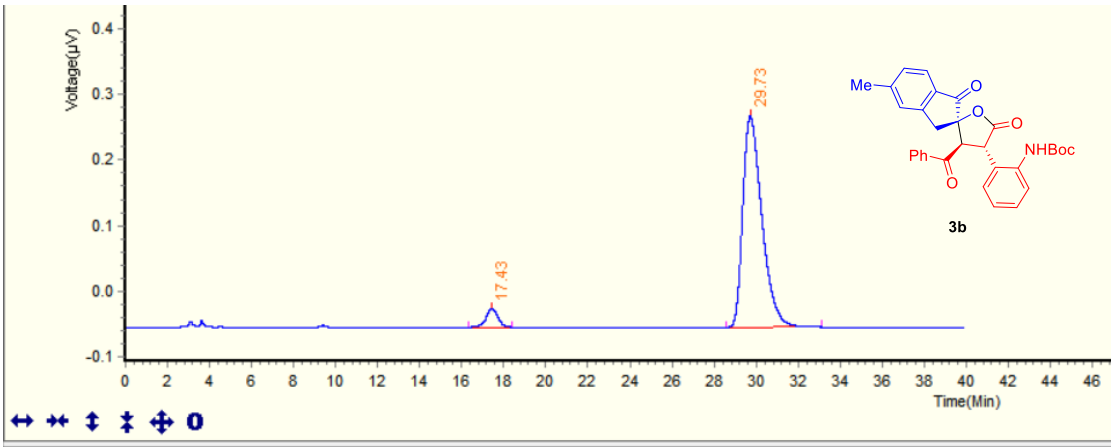
Integration Result | Calculation Result | TimeTable

No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	
1	11.00	4006	193	0.39%	0.658	BB
2	12.29	94743	3669	9.18%	1.168	BB
3	15.95	20752	627	2.01%	1.193	BB
4	21.16	912930	21715	88.43%	2.386	BB
Total		1,032,431	26,204	100.00%		



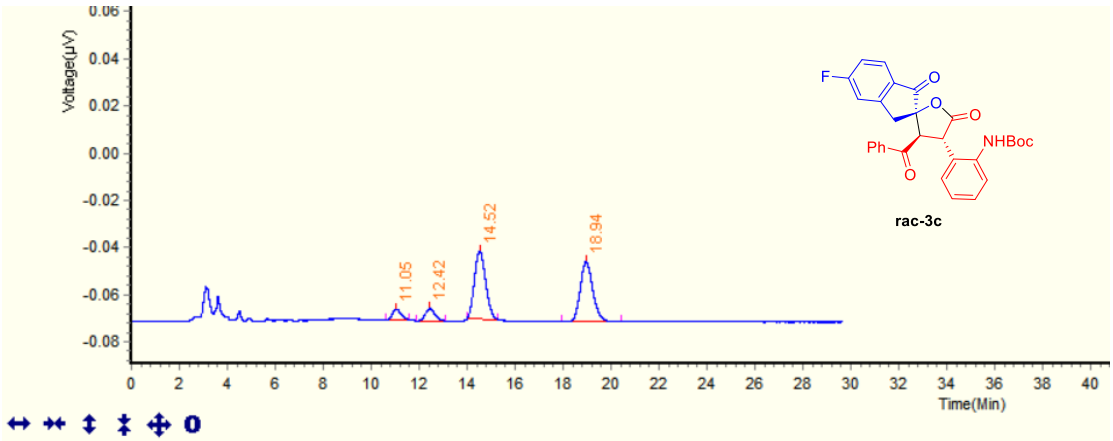
Integration Result | Calculation Result | TimeTable

No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	
1	17.26	220606	5860	50.11%	1.725	BB
2	29.92	219648	3628	49.89%	3.739	BB
Total		440,254	9,488	100.00%		



Integration Result | Calculation Result | TimeTable

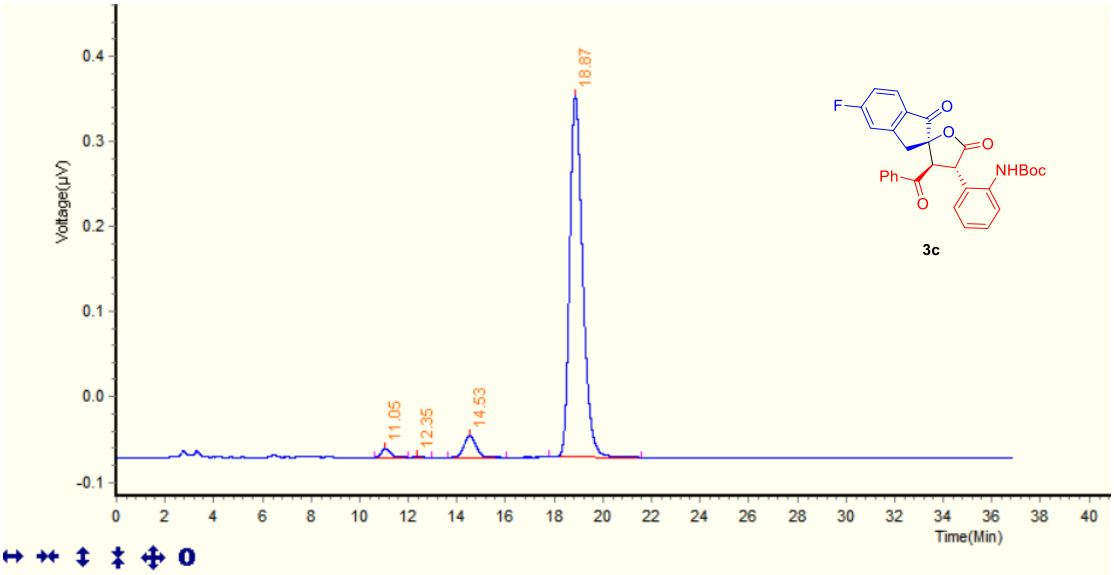
No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	
1	17.43	546885	14076	5.11%	2.028	BB
2	29.73	10161298	160430	94.89%	4.552	BB
Total		10,708,183	174,506	100.00%		



rac-3c

Integration Result | Calculation Result | TimeTable

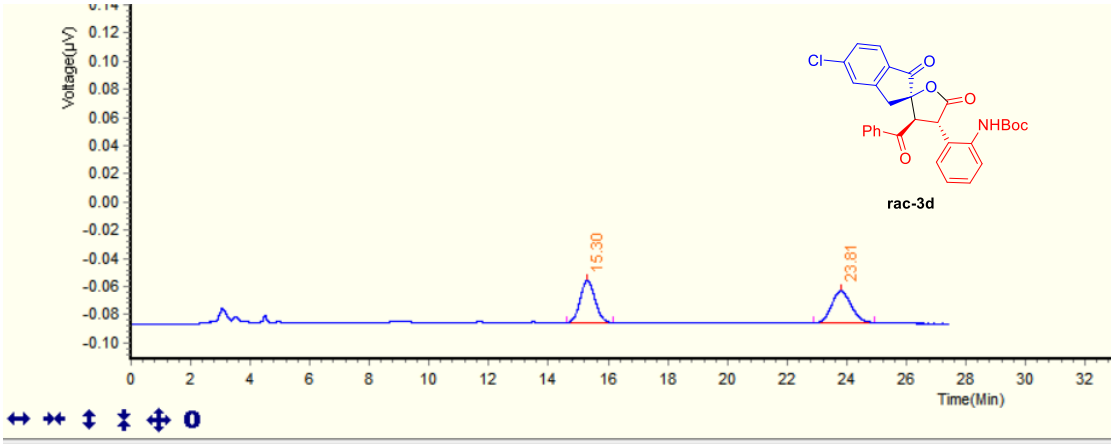
No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	
1	11.05	57370	2308	5.44%	0.984	BB
2	12.42	69579	2588	6.59%	1.199	BB
3	14.52	467101	14450	44.27%	1.292	BB
4	18.94	461041	12631	43.70%	2.459	BB
Total		1,055,091	31,977	100.00%		



3c

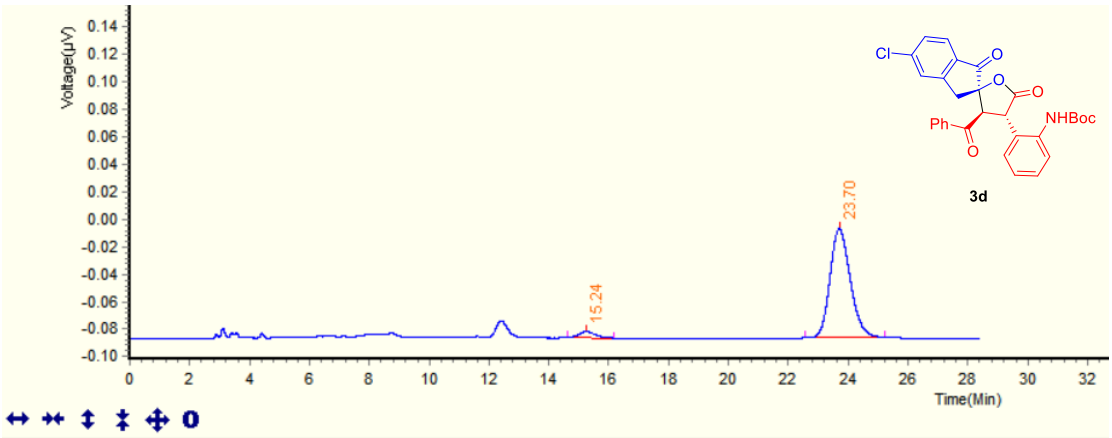
Integration Result | Calculation Result | TimeTable

No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	
1	11.05	132681	5186	1.58%	1.375	BV
2	12.35	5810	245	0.07%	0.954	VB
3	14.53	429239	12244	5.11%	2.407	BB
4	18.87	7833922	212194	93.24%	3.82	BB
Total		8,401,652	229,869	100.00%		



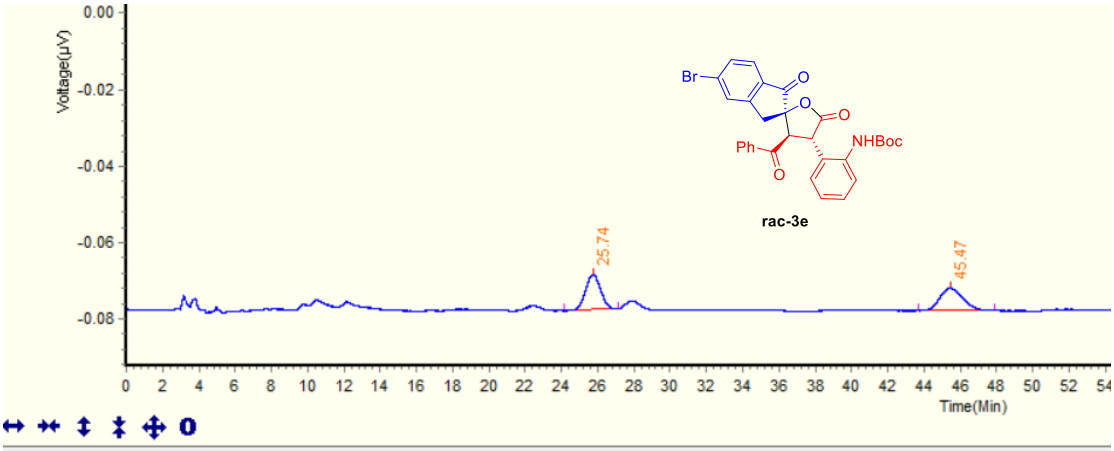
Integration Result | Calculation Result | TimeTable

No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	
1	15.30	519902	15167	50.05%	1.563	BB
2	23.81	518802	11323	49.95%	2.075	BB
Total		1,038,704	26,490	100.00%		



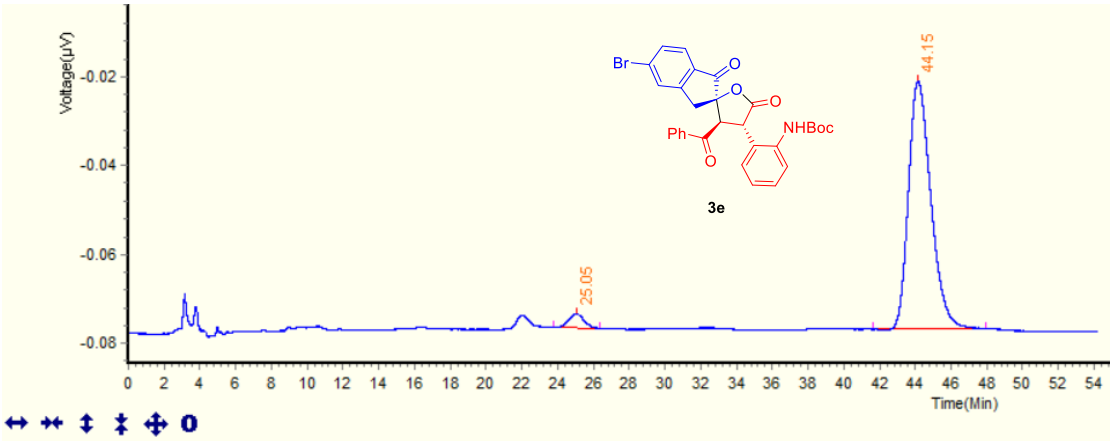
Integration Result | Calculation Result | TimeTable

No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	
1	15.24	82695	2241	4.26%	1.53	BB
2	23.70	1858803	39546	95.74%	2.677	BB
Total		1,941,498	41,787	100.00%		



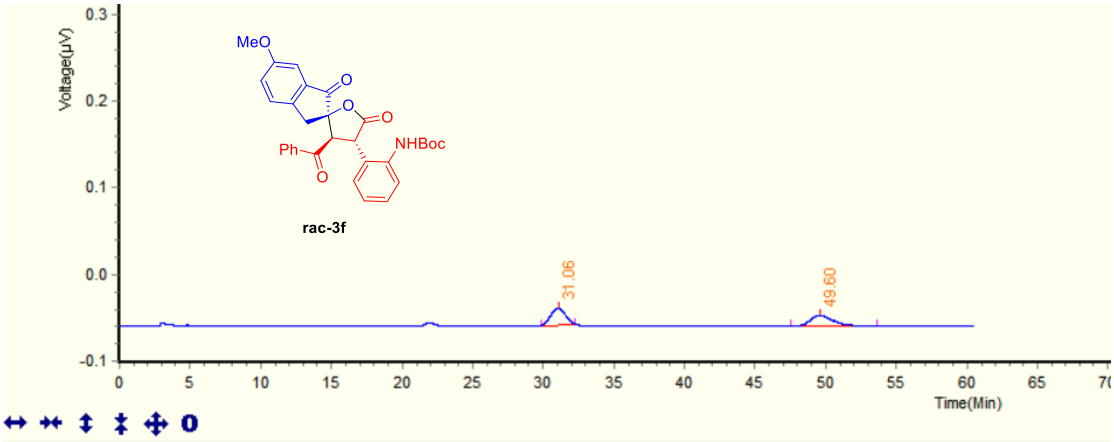
Integration Result | Calculation Result | TimeTable

No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	
1	25.74	263785	4606	50.28%	2.945	BB
2	45.47	260887	2938	49.72%	4.229	BB
Total		524,672	7,544	100.00%		



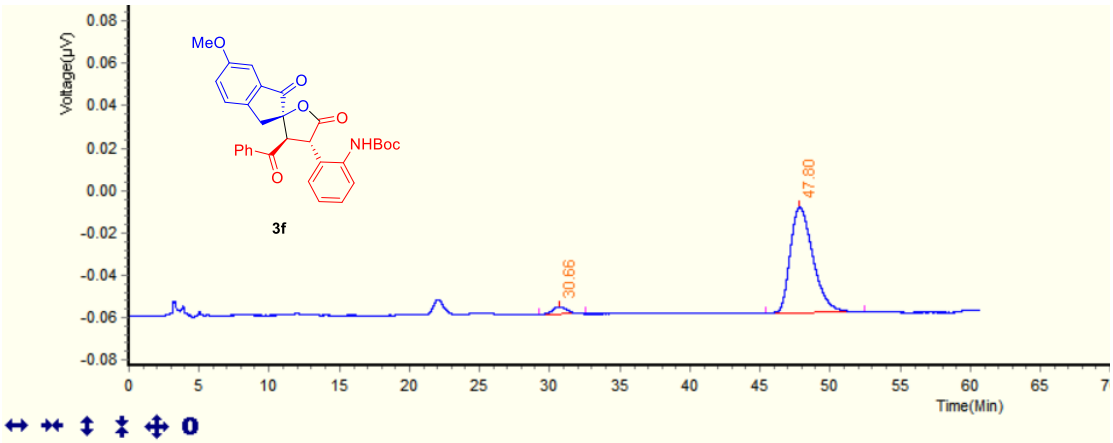
Integration Result | Calculation Result | TimeTable

No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	
1	25.05	91393	1571	3.64%	2.639	BB
2	44.15	2418425	27904	96.36%	6.344	BB
Total		2,509,818	29,475	100.00%		



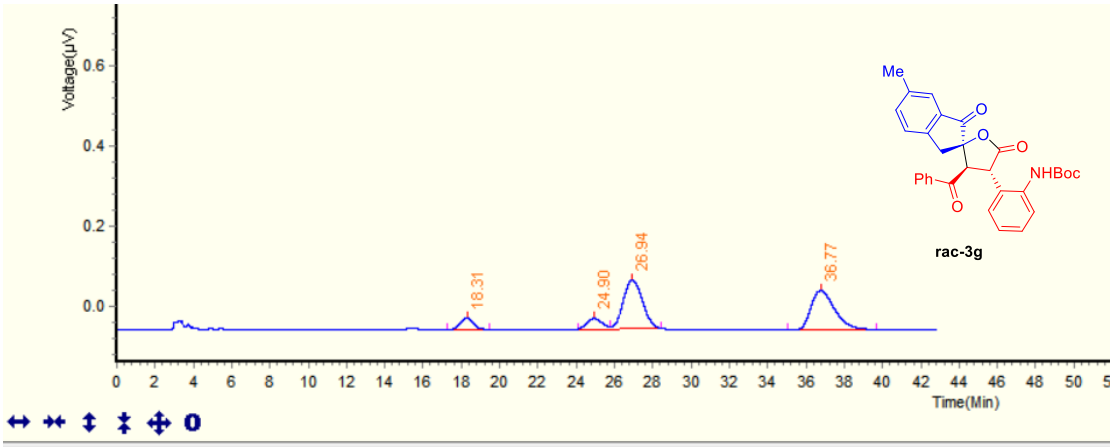
Integration Result | Calculation Result | TimeTable

No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	
1	31.06	681996	9273	50.31%	2.445	BB
2	49.60	673534	5994	49.69%	6.144	BB
Total		1,355,530	15,267	100.00%		



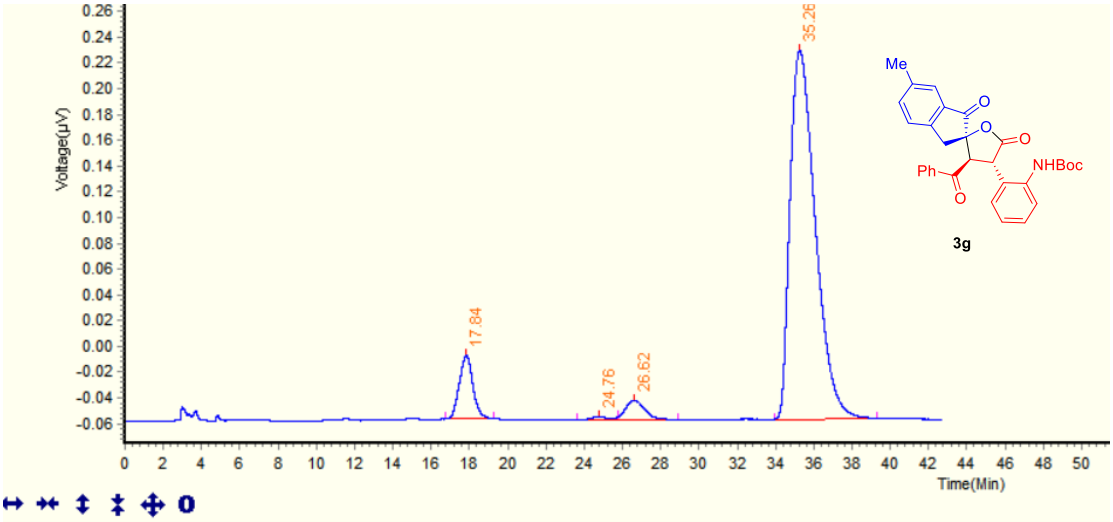
Integration Result | Calculation Result | TimeTable

No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	
1	30.66	127520	1721	4.36%	3.333	BB
2	47.80	2794709	24796	95.64%	6.981	BB
Total		2,922,229	26,517	100.00%		



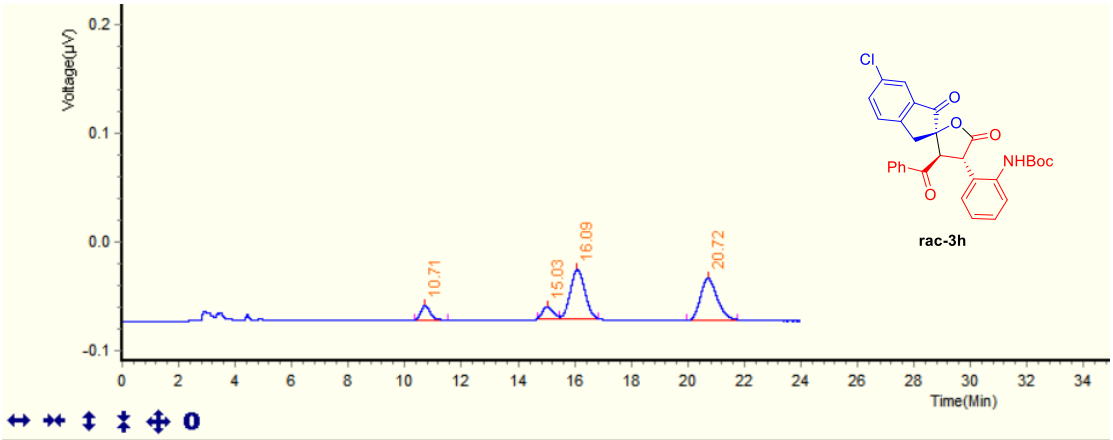
Integration Result | Calculation Result | TimeTable

No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	
1	18.31	661936	14690	6.80%	2.174	BB
2	24.90	720978	12940	7.40%	1.685	BV
3	26.94	4186376	60281	43.00%	2.663	VB
4	36.77	4167107	48713	42.80%	4.659	BB
Total		9,736,397	136,624	100.00%		



Integration Result | Calculation Result | TimeTable

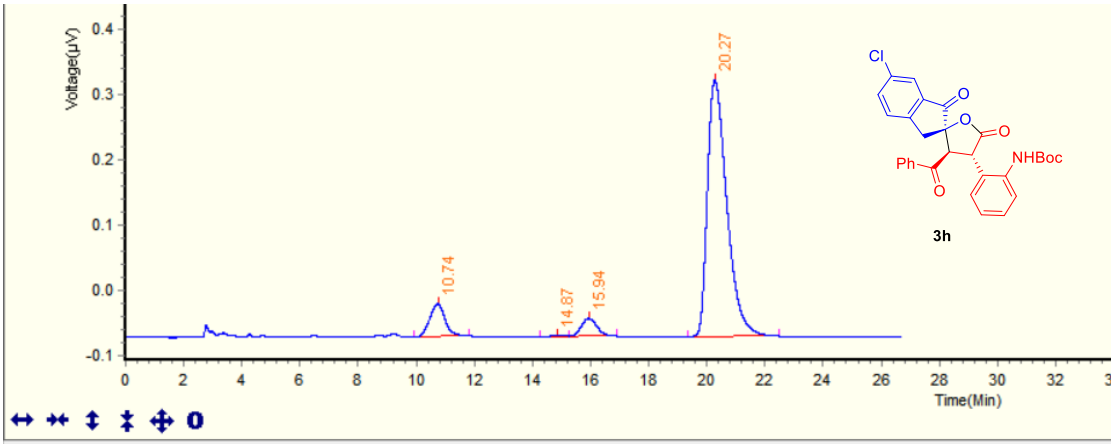
No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	
1	17.84	1218831	24647	8.14%	2.522	BB
2	24.76	83713	1194	0.56%	2.123	BV
3	26.62	531627	7530	3.55%	3.142	VB
4	35.26	13133310	142859	87.75%	5.397	BB
Total		14,967,481	176,230	100.00%		



rac-3h

Integration Result | Calculation Result | TimeTable

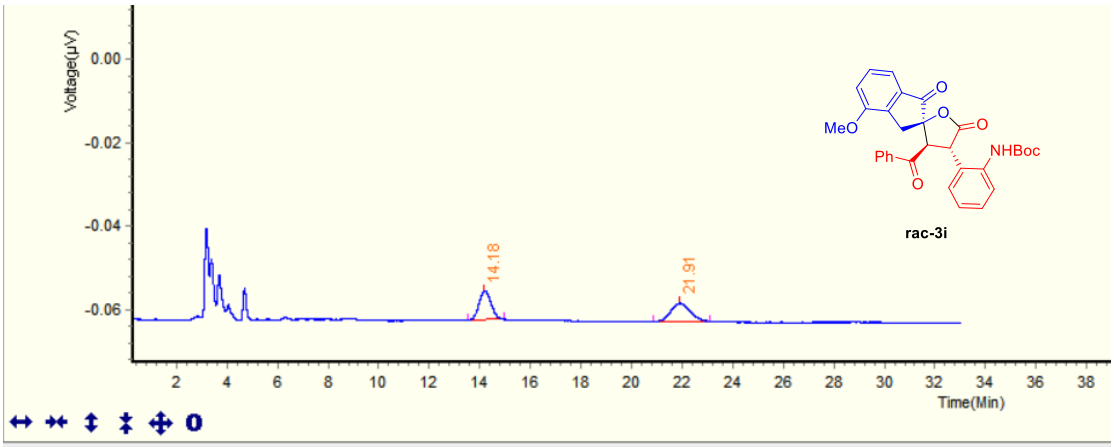
No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	
1	10.71	165683	6810	8.78%	1.184	BB
2	15.03	138725	5212	7.35%	0.785	BV
3	16.09	802525	22520	42.51%	1.386	VB
4	20.72	780879	19178	41.36%	1.802	BB
Total		1,887,812	53,720	100.00%		



3h

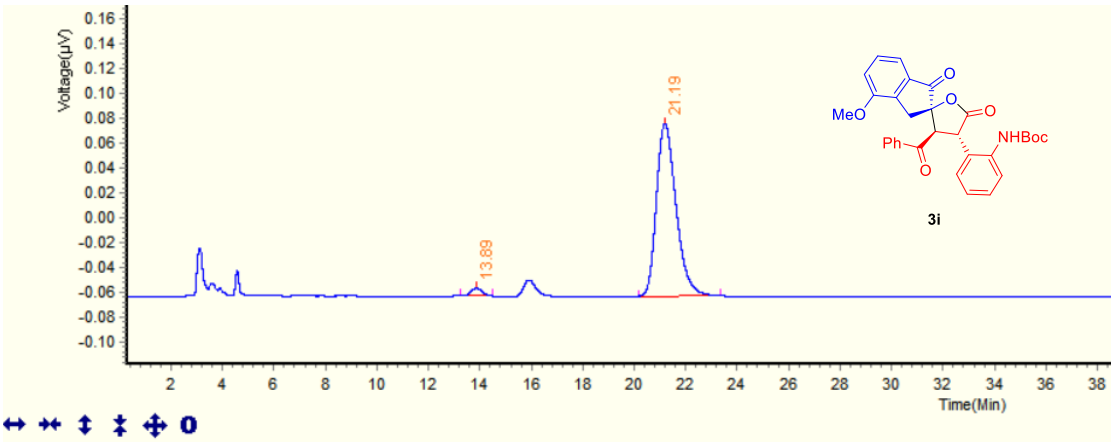
Integration Result | Calculation Result | TimeTable

No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	
1	10.74	908114	25082	8.57%	1.882	BB
2	14.87	39141	1373	0.37%	0.995	BV
3	15.94	517449	13716	4.88%	1.605	VB
4	20.27	9130856	196962	86.18%	3.127	BB
Total		10,595,560	237,133	100.00%		



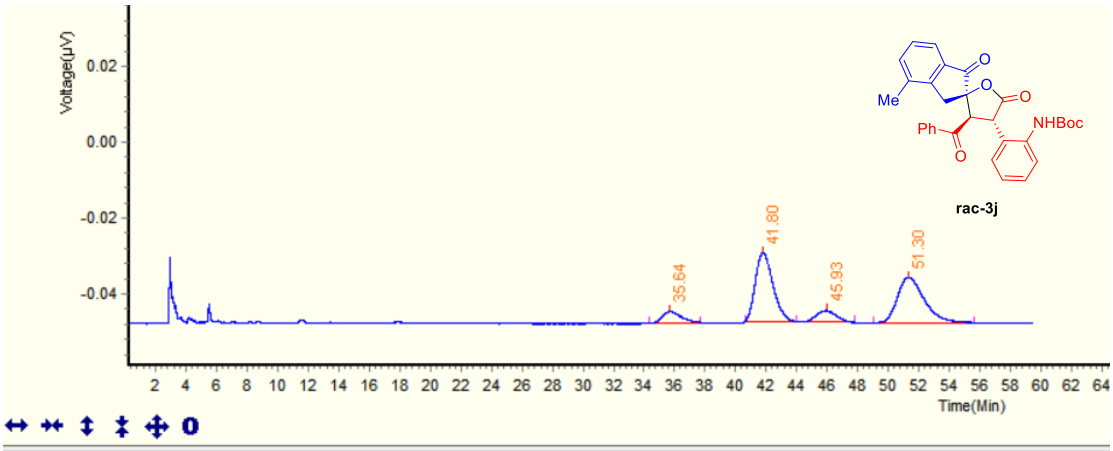
Integration Result | Calculation Result | TimeTable

No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	
1	14.18	113851	3456	50.11%	1.438	BB
2	21.91	113370	2178	49.89%	2.227	BB
Total		227,221	5,634	100.00%		



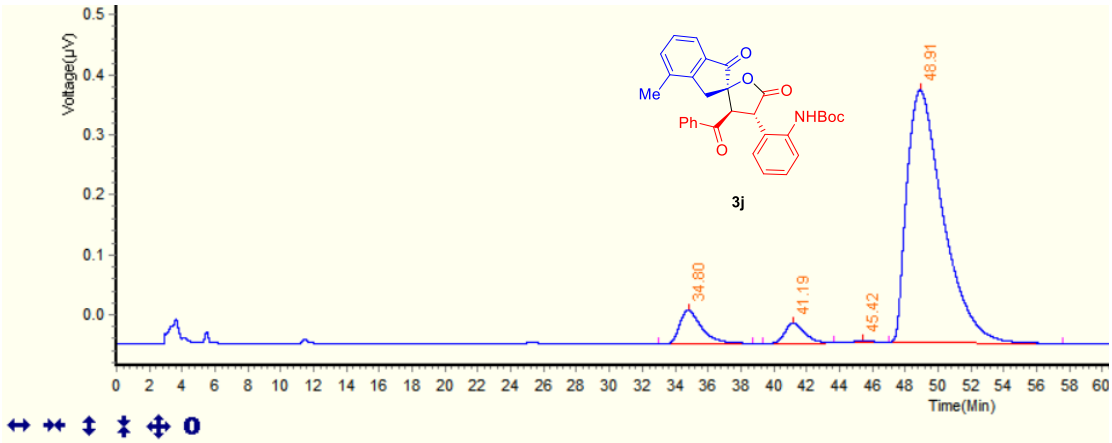
Integration Result | Calculation Result | TimeTable

No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	
1	13.89	93450	3041	2.49%	1.267	BB
2	21.19	3662554	69302	97.51%	3.188	BB
Total		3,756,004	72,343	100.00%		



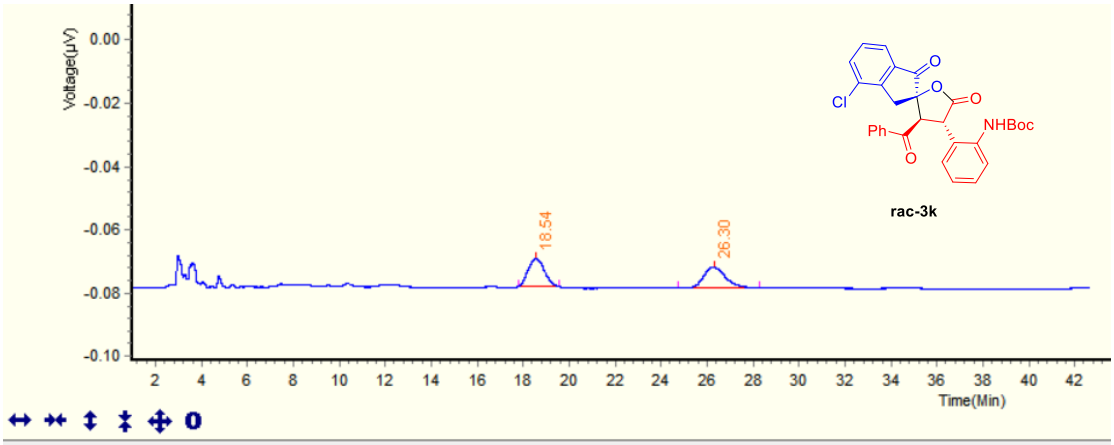
Integration Result | Calculation Result | TimeTable

No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	
1	35.64	128186	1495	7.50%	3.265	BB
2	41.80	724649	9048	42.39%	3.327	BV
3	45.93	133610	1498	7.82%	3.819	VB
4	51.30	722954	5940	42.29%	6.592	BB
Total		1,709,399	17,981	100.00%		



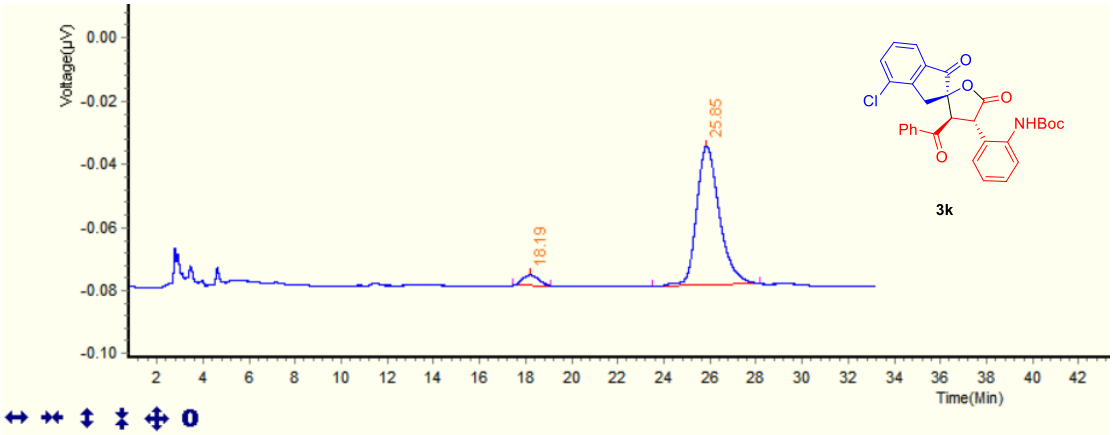
Integration Result | Calculation Result | TimeTable

No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	
1	34.80	2538715	27577	7.02%	5.693	BB
2	41.19	1368276	16609	3.79%	4.336	BV
3	48.91	32142677	209587	88.94%	10.586	BB
4	45.42	90400	1419	0.25%	3.313	VB
Total		36,140,068	255,192	100.00%		



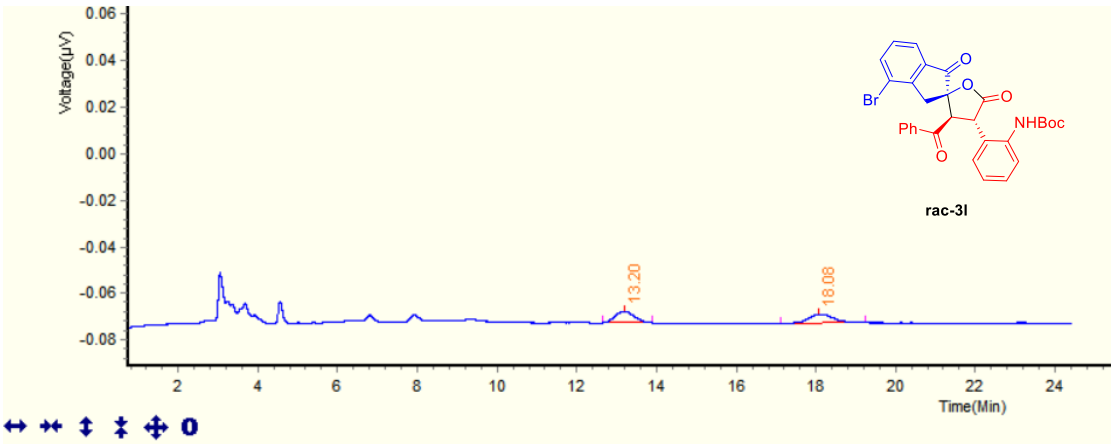
Integration Result | Calculation Result | TimeTable

No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	
1	18.54	217675	4393	50.41%	1.768	BB
2	26.30	214122	3272	49.59%	3.535	BB
Total		431,797	7,665	100.00%		



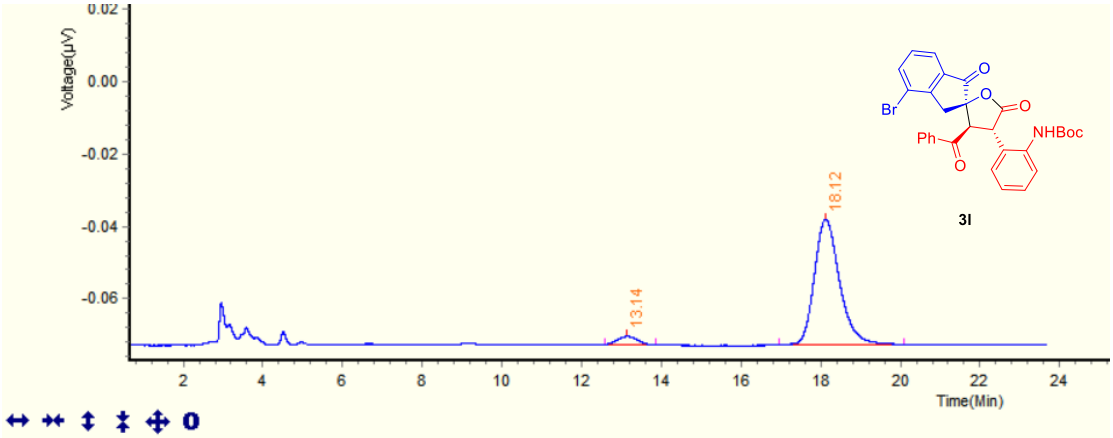
Integration Result | Calculation Result | TimeTable

No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	
1	18.19	79888	1640	4.99%	1.609	BB
2	25.85	1521508	22053	95.01%	4.705	BB
Total		1,601,396	23,693	100.00%		



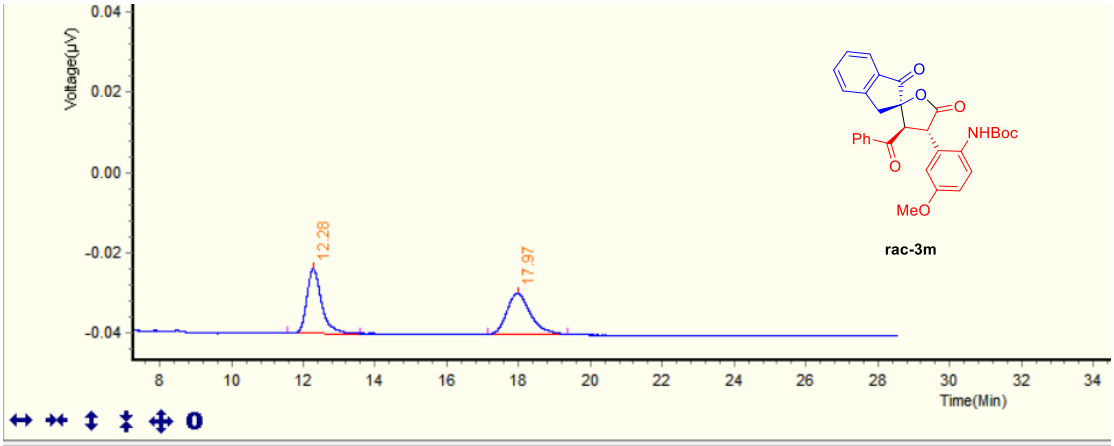
Integration Result | Calculation Result | TimeTable

No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	
1	13.20	82244	2415	50.46%	1.227	BB
2	18.08	80742	1904	49.54%	2.151	BB
Total		162,986	4,319	100.00%		



Integration Result | Calculation Result | TimeTable

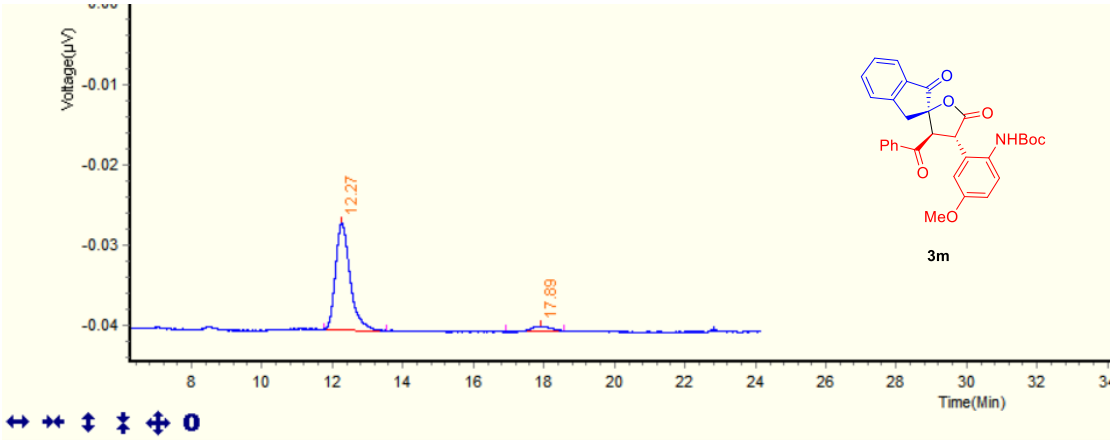
No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	
1	13.14	39838	1162	5.02%	1.301	BB
2	18.12	754441	17312	94.98%	3.141	BB
Total		794,279	18,474	100.00%		



rac-3m

Integration Result | Calculation Result | TimeTable

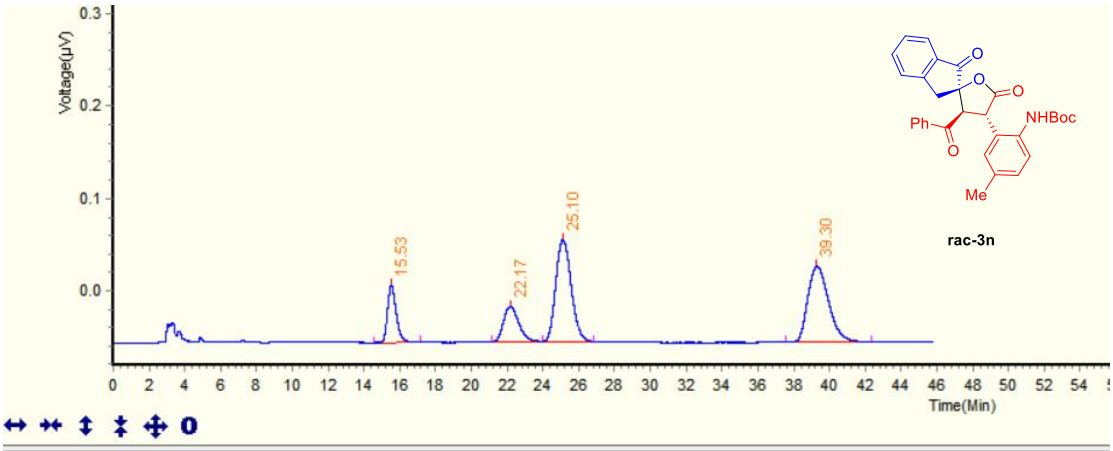
No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	
1	12.28	226671	8025	49.63%	2.01	BB
2	17.97	230087	5065	50.37%	2.217	BB
Total		456,758	13,090	100.00%		



3m

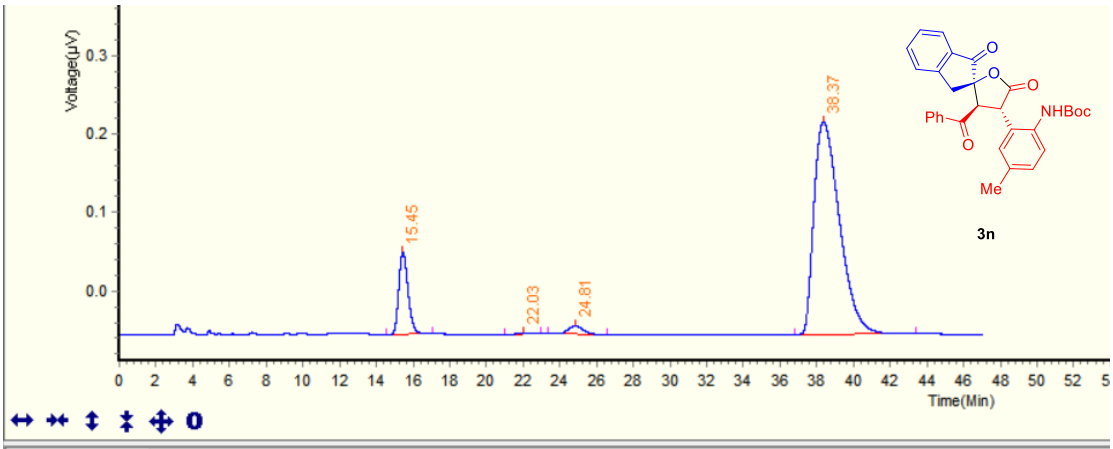
Integration Result | Calculation Result | TimeTable

No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	
1	12.27	189560	6642	93.91%	1.802	BB
2	17.89	12294	321	6.09%	1.652	BB
Total		201,854	6,963	100.00%		



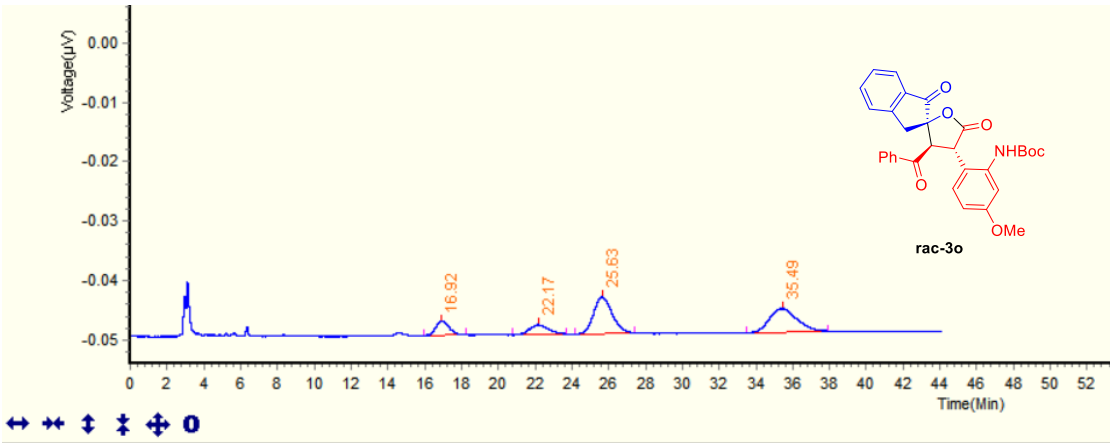
Integration Result | Calculation Result | TimeTable

No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	
1	15.53	1040376	30944	11.82%	2.609	BB
2	22.17	1136507	19065	12.92%	2.781	BV
3	25.10	3294864	54867	37.45%	2.864	VB
4	39.30	3326796	41164	37.81%	4.793	BB
Total		8,798,543	146,040	100.00%		



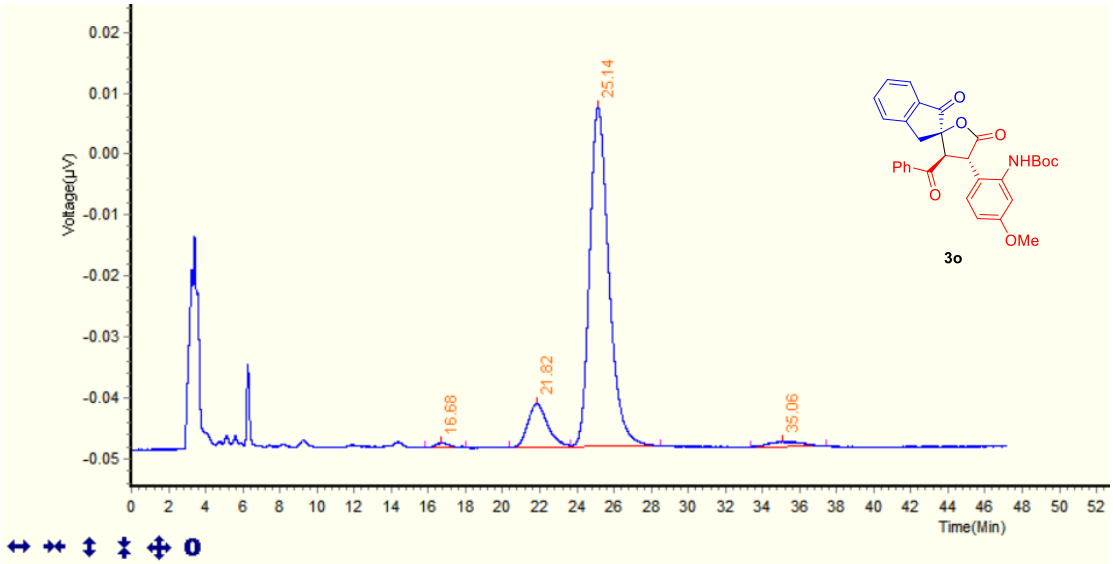
Integration Result | Calculation Result | TimeTable

No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	
1	15.45	1861274	52414	12.21%	2.487	BB
2	22.03	16693	391	0.11%	1.999	BB
3	24.81	298678	5081	1.96%	3.219	BB
4	38.37	13070024	134671	85.72%	6.584	BB
Total		15,246,669	192,557	100.00%		



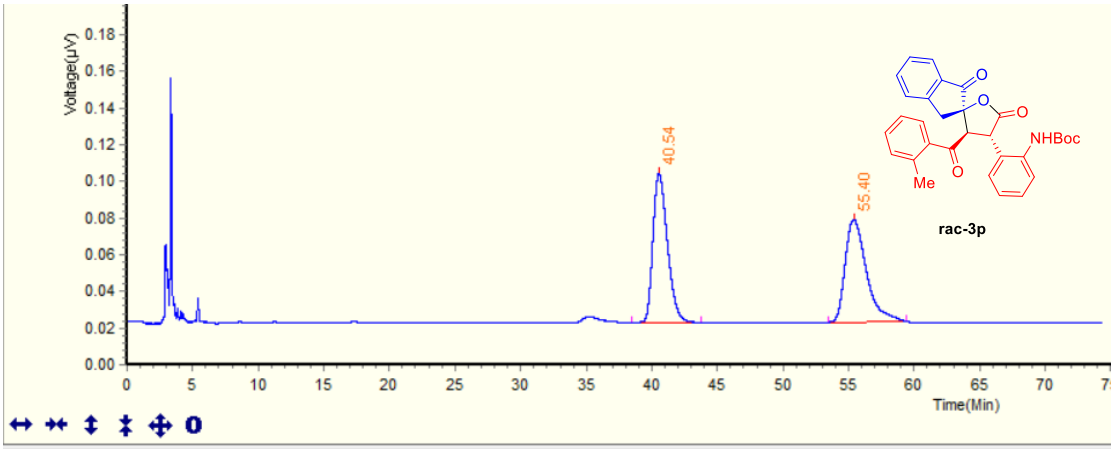
Integration Result | Calculation Result | TimeTable

No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	
1	16.92	60536	1224	11.22%	2.285	BB
2	22.17	58132	827	10.78%	2.97	BB
3	25.63	213424	3081	39.57%	3.291	BB
4	35.49	207268	1988	38.43%	4.479	BB
Total		539,360	7,120	100.00%		



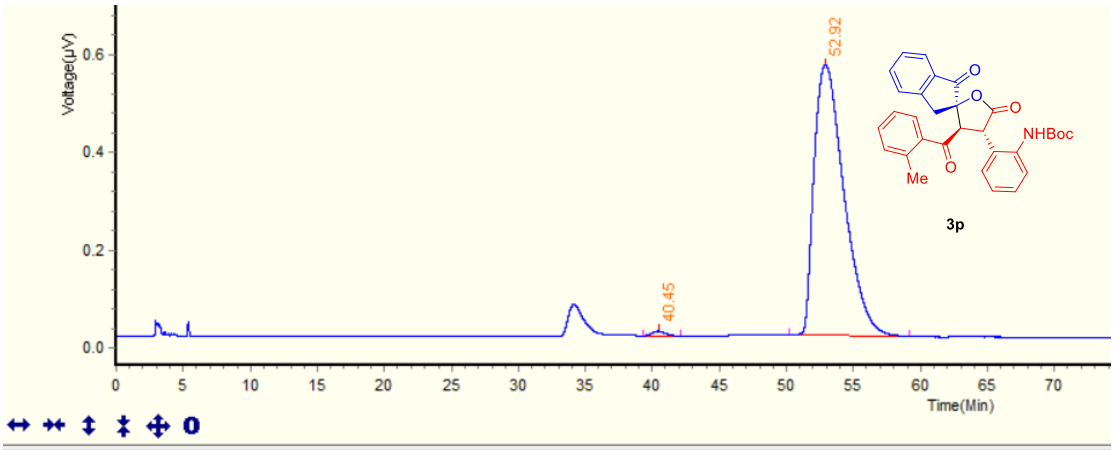
Integration Result | Calculation Result | TimeTable

No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	
1	16.68	15724	356	0.67%	2.201	BB
2	21.82	264115	3557	11.29%	3.243	BV
3	25.14	2010331	27912	85.94%	4.874	VB
4	35.06	49035	386	2.10%	4.058	BB
Total		2,339,205	32,211	100.00%		



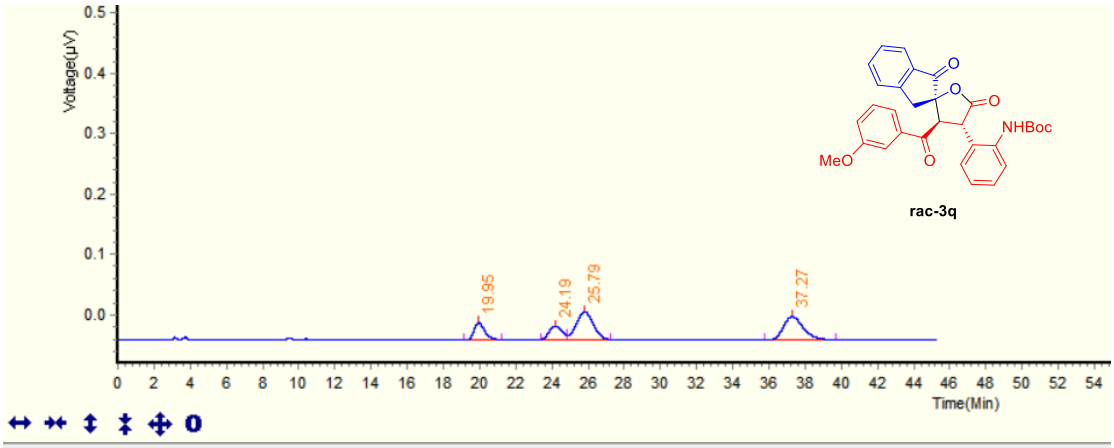
Integration Result | Calculation Result | TimeTable

No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	
1	40.54	3124129	40656	49.80%	5.295	BB
2	55.40	3149727	27998	50.20%	5.986	BB
Total		6,273,856	68,654	100.00%		



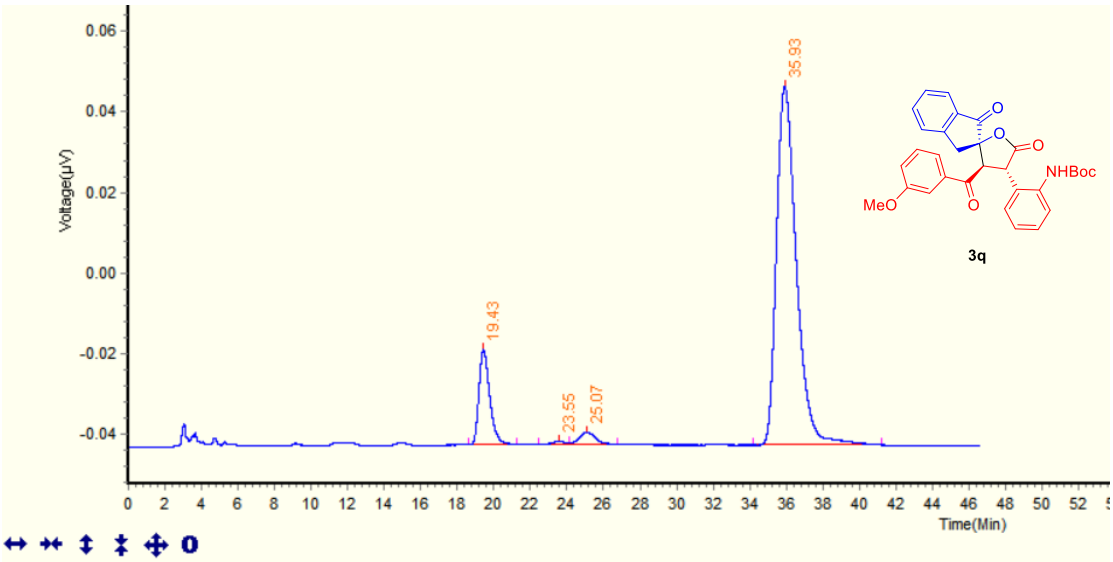
Integration Result | Calculation Result | TimeTable

No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	
1	40.45	352219	5041	0.81%	2.902	BB
2	52.92	42901779	276350	99.19%	8.947	BB
Total		43,253,998	281,391	100.00%		



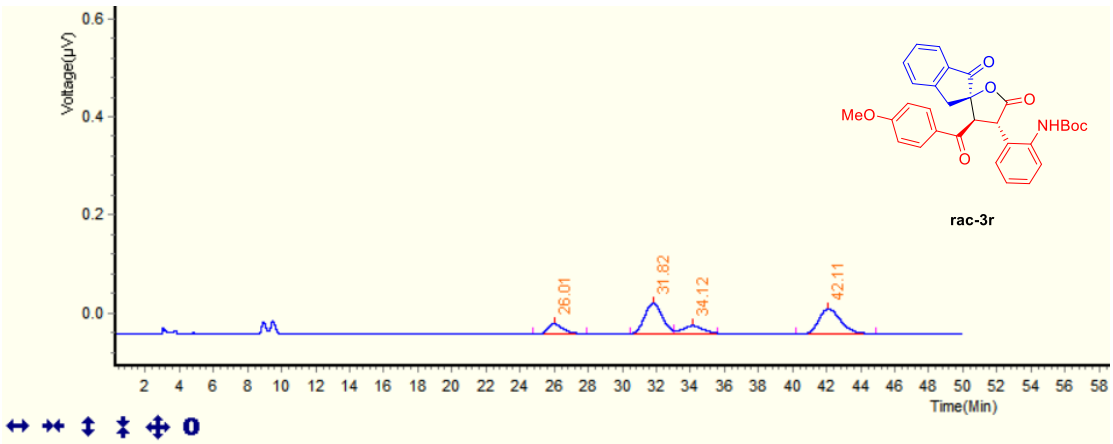
Integration Result | Calculation Result | TimeTable

No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	
1	19.95	582550	14051	14.37%	2.139	BB
2	24.19	537351	10996	13.26%	1.419	BV
3	25.79	1479481	22934	36.51%	2.427	VB
4	37.27	1453260	19069	35.86%	3.906	BB
Total		4,052,642	67,050	100.00%		



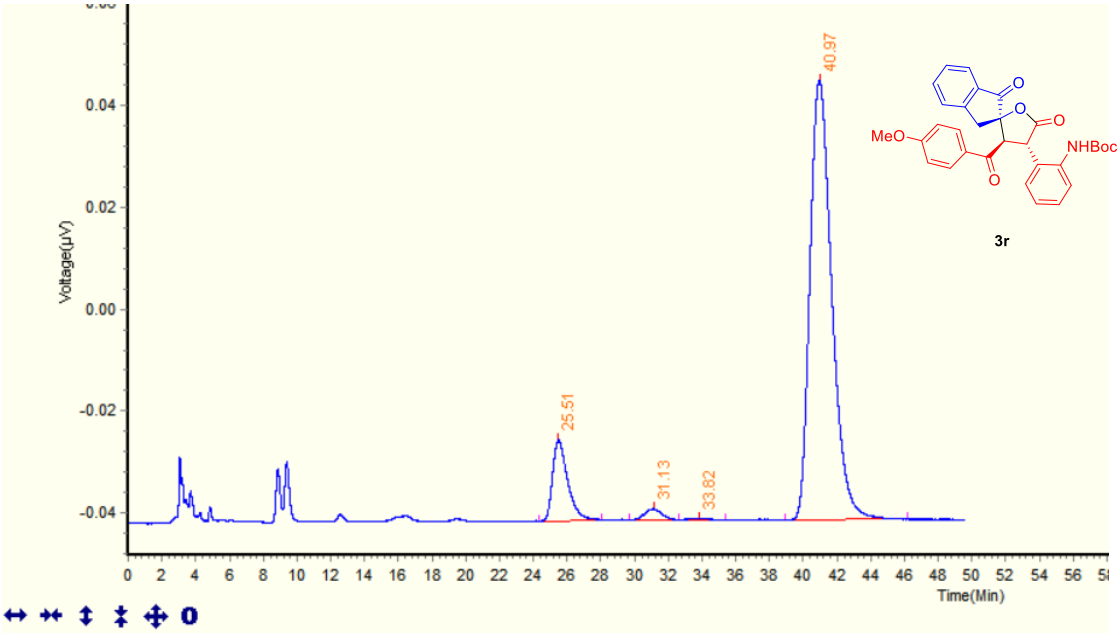
Integration Result | Calculation Result | TimeTable

No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	
1	19.43	480477	11735	12.11%	2.703	BB
2	23.55	17354	365	0.44%	1.688	BV
3	25.07	89433	1470	2.25%	2.607	VB
4	35.93	3380964	44525	85.20%	7.007	BB
Total		3,968,228	58,095	100.00%		



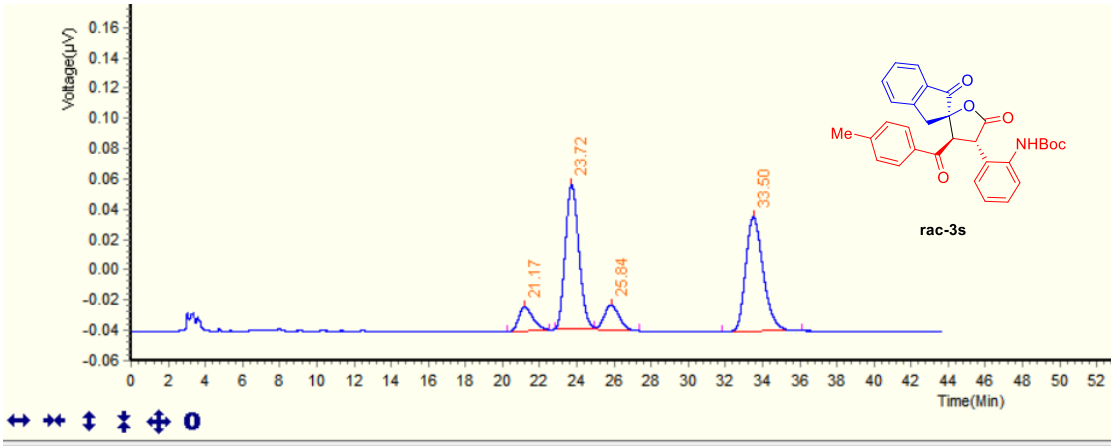
Integration Result | Calculation Result | TimeTable

No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	
1	26.01	645970	10421	11.14%	3.157	BB
2	31.82	2243085	30912	38.67%	2.612	BV
3	34.12	645739	7832	11.13%	2.562	VB
4	42.11	2265945	25469	39.06%	4.658	BB
Total		5,800,739	74,634	100.00%		



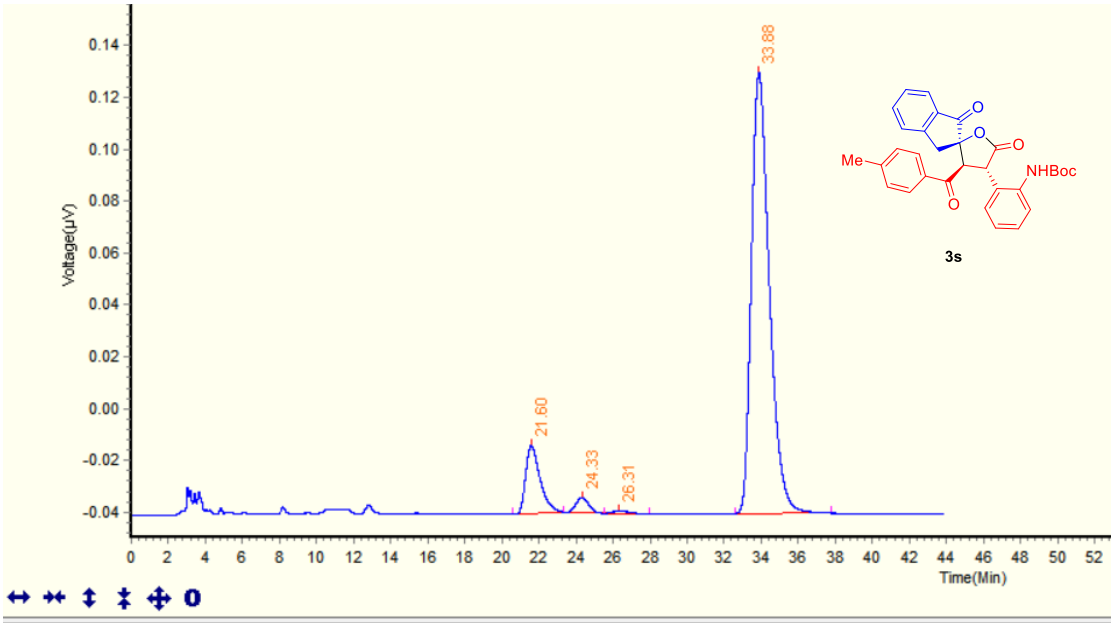
Integration Result | Calculation Result | TimeTable

No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	
1	25.51	487420	7875	10.95%	3.754	BB
2	31.13	73649	1077	1.65%	2.946	BV
3	33.82	12025	174	0.27%	2.71	VB
4	40.97	3877620	43182	87.12%	7.237	BB
Total		4,450,714	52,308	100.00%		



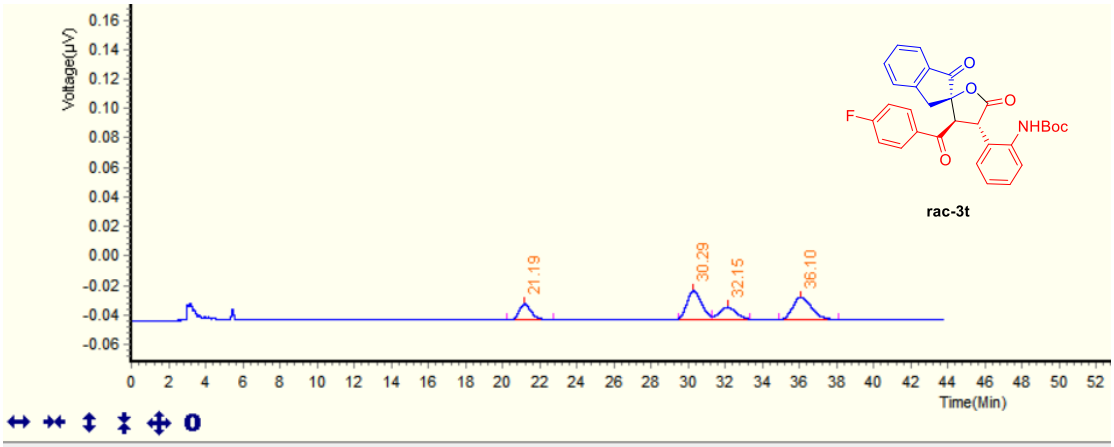
Integration Result | Calculation Result | TimeTable

No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	
1	21.17	418910	8064	7.19%	2.307	BB
2	23.72	2458389	48002	42.19%	2.172	BV
3	25.84	470947	8430	8.08%	2.351	VB
4	33.50	2478666	37781	42.54%	4.297	BB
Total		5,826,912	102,277	100.00%		



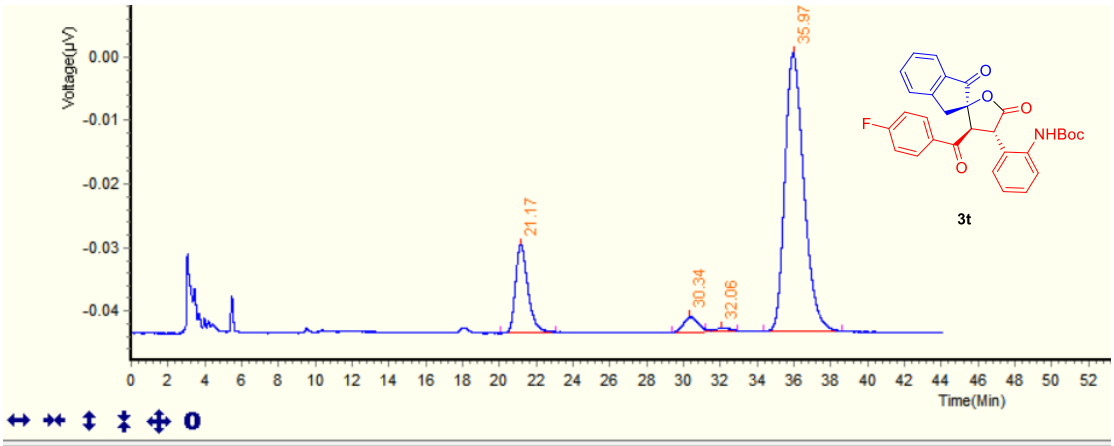
Integration Result | Calculation Result | TimeTable

No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	
1	24.33	133012	2850	2.02%	2.227	BV
2	26.31	22850	498	0.35%	2.408	VB
3	21.60	703654	13047	10.71%	2.728	BB
4	33.88	5710590	84980	86.92%	5.182	BB
Total		6,570,106	101,375	100.00%		



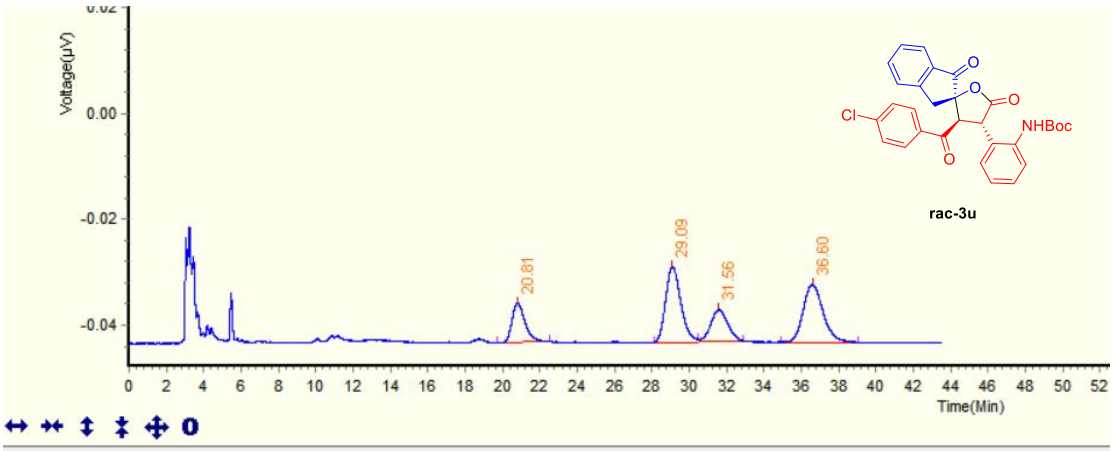
Integration Result | Calculation Result | TimeTable

No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	
1	21.19	245623	5409	15.84%	2.542	BB
2	30.29	523410	9612	33.75%	1.861	BV
3	32.15	248239	4066	16.01%	2.042	VB
4	36.10	533602	7649	34.41%	3.177	BB
Total		1,550,874	26,736	100.00%		



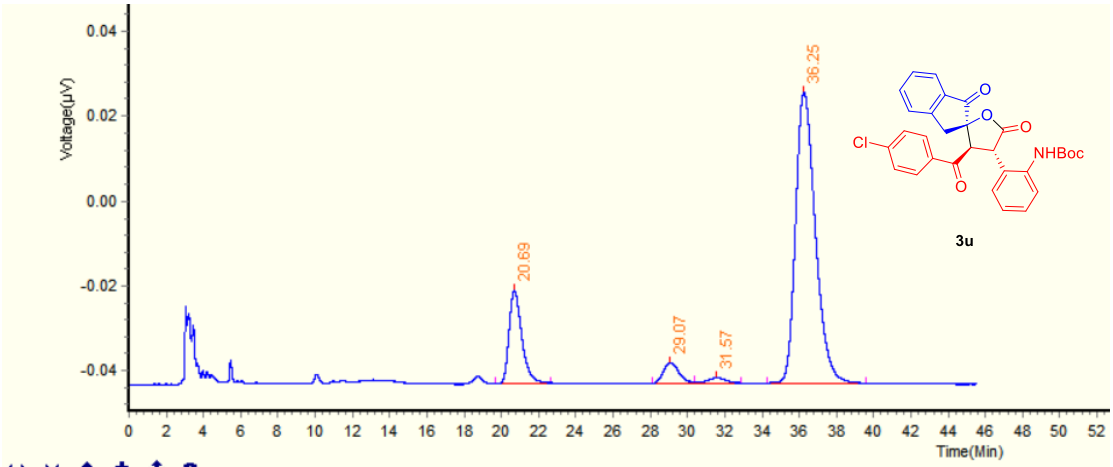
Integration Result | Calculation Result | TimeTable

No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	
1	21.17	321809	6896	16.36%	3.015	BB
2	30.34	64131	1188	3.26%	1.885	BV
3	32.06	15850	280	0.81%	1.723	VB
4	35.97	1564979	21874	79.57%	4.249	BB
Total		1,966,769	30,238	100.00%		



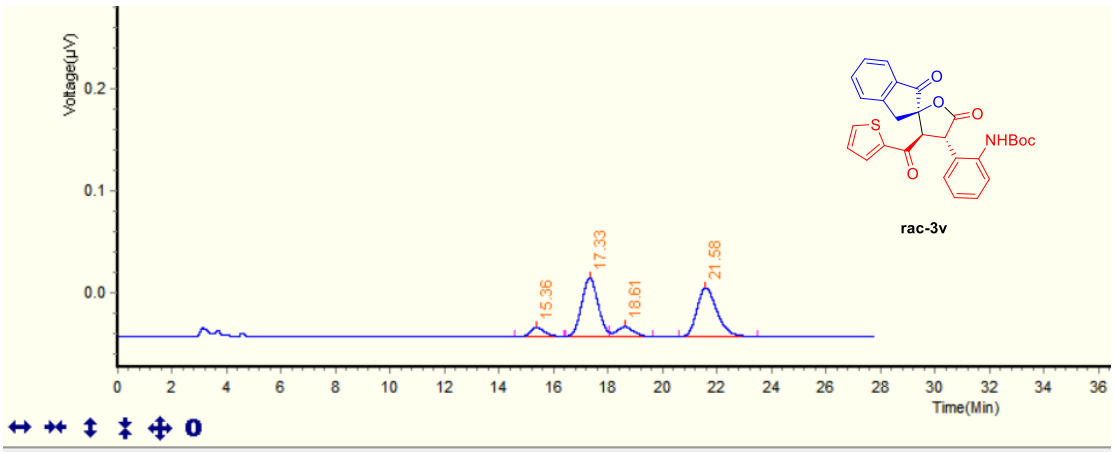
Integration Result | Calculation Result | TimeTable

No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	
1	20.81	178370	3716	15.27%	2.796	BB
2	29.09	398490	7065	34.12%	2.39	BV
3	31.56	188887	2982	16.18%	2.434	VB
4	36.60	402001	5467	34.43%	4.149	BB
Total		1,167,748	19,230	100.00%		



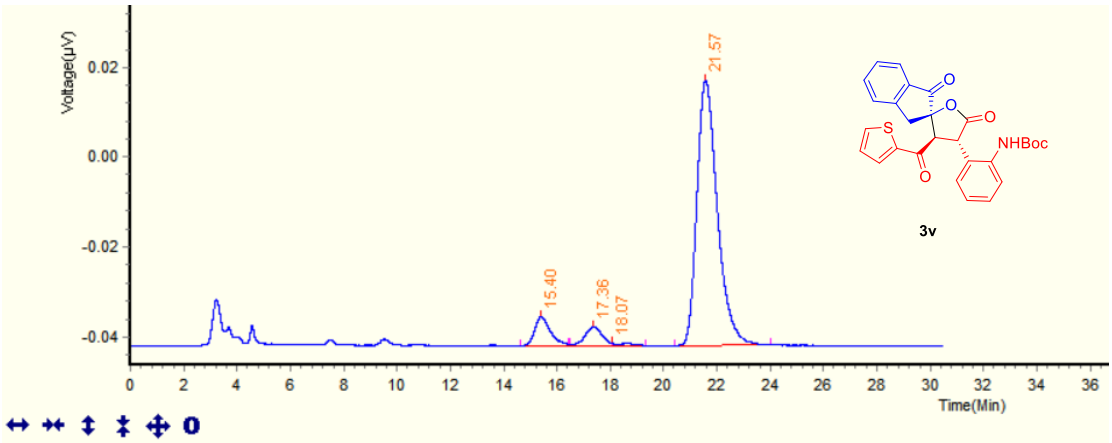
Integration Result | Calculation Result | TimeTable

No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	
1	20.69	521943	10986	16.17%	2.972	BB
2	29.07	139588	2457	4.33%	2.264	BV
3	31.57	41391	653	1.28%	2.499	VB
4	36.25	2524127	34294	78.22%	5.331	BB
Total		3,227,049	48,390	100.00%		



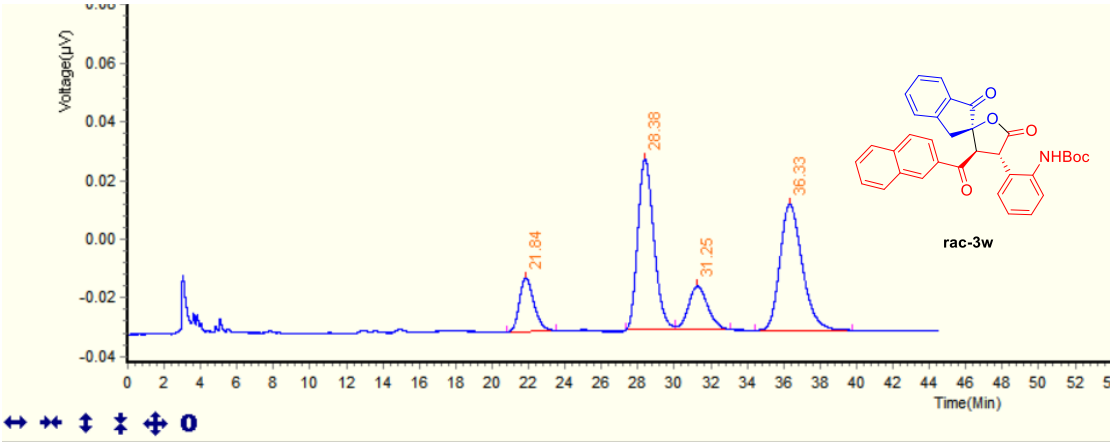
Integration Result | Calculation Result | TimeTable

No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	
1	15.36	159594	4286	5.76%	1.843	BB
2	17.33	1192607	28664	43.02%	1.635	BV
3	18.61	205375	4725	7.41%	1.59	VB
4	21.58	1214873	23750	43.82%	2.908	BB
Total		2,772,449	61,425	100.00%		



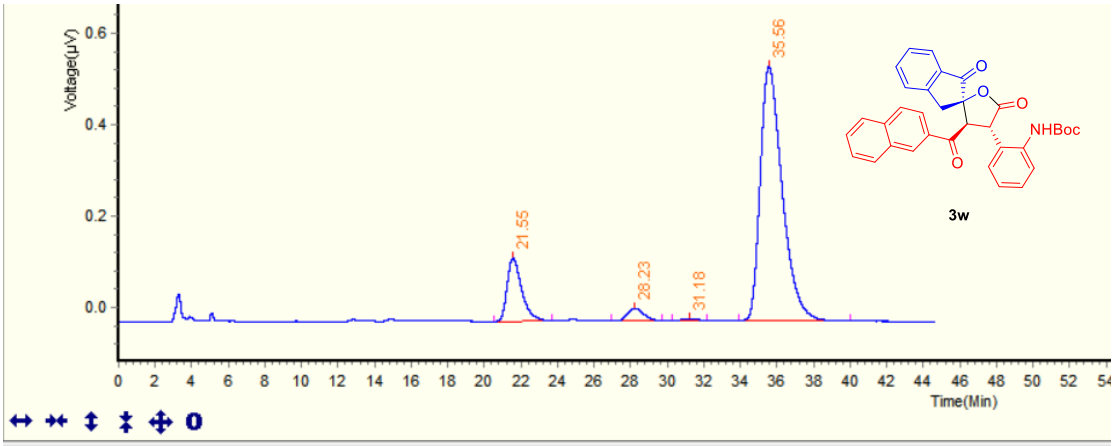
Integration Result | Calculation Result | TimeTable

No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	
1	15.40	131961	3187	7.31%	1.832	BV
2	17.36	93024	2119	5.15%	1.61	VV
3	18.07	10738	249	0.59%	1.231	VB
4	21.57	1570018	29537	86.95%	3.632	BB
Total		1,805,741	35,092	100.00%		



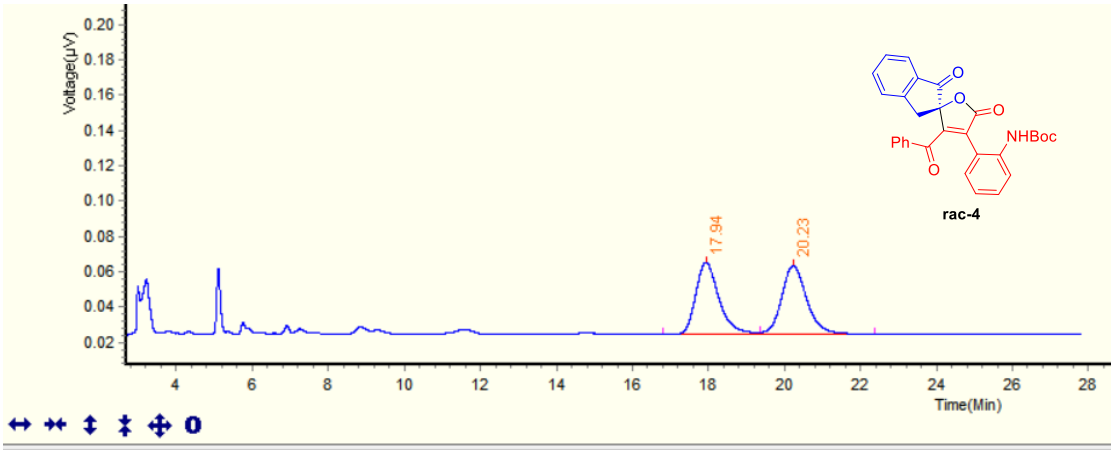
Integration Result | Calculation Result | TimeTable

No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	
1	21.84	504067	9147	10.81%	2.674	BB
2	28.38	1790573	29089	38.41%	2.721	BV
3	31.25	541589	7420	11.62%	2.996	VB
4	36.33	1825346	21519	39.16%	5.396	BB
Total		4,661,575	67,175	100.00%		



Integration Result | Calculation Result | TimeTable

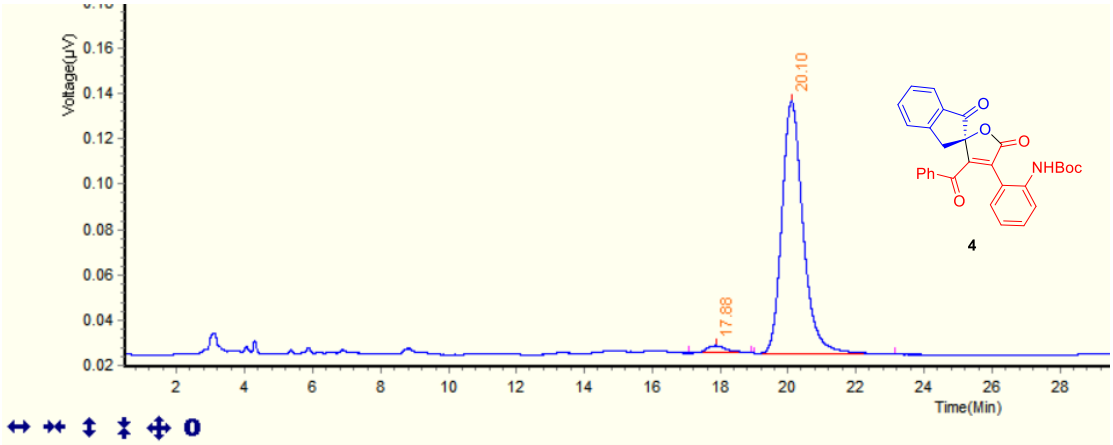
No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	
1	21.55	3808388	68699	13.60%	3.208	BB
2	28.23	848654	13672	3.03%	2.751	BB
3	31.18	126477	2099	0.45%	1.925	BB
4	35.56	23219116	277784	82.92%	6.098	BB
Total		28,002,635	362,254	100.00%		



rac-4

Integration Result | Calculation Result | TimeTable

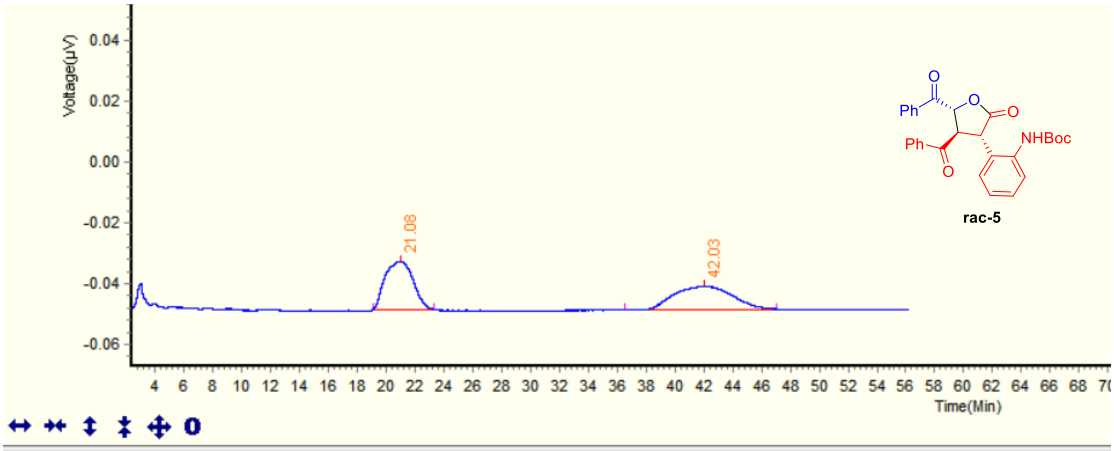
No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	
1	17.94	879243	20446	49.41%	2.53	BV
2	20.23	900149	19438	50.59%	3.026	VB
Total		1,779,392	39,884	100.00%		



4

Integration Result | Calculation Result | TimeTable

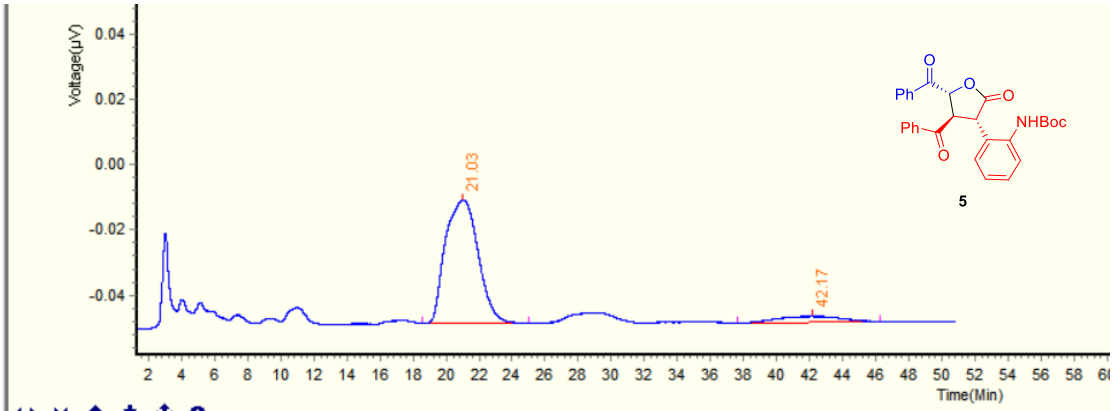
No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	
1	17.88	66038	1616	2.67%	1.838	BB
2	20.10	2409260	55636	97.33%	4.113	BB
Total		2,475,298	57,252	100.00%		



rac-5

Integration Result | Calculation Result | TimeTable

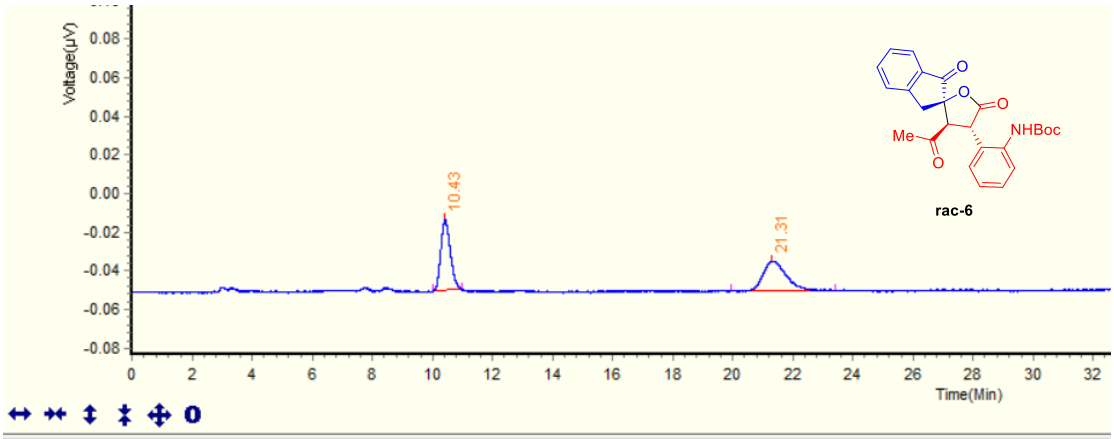
No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	
1	21.08	1072314	7947	49.95%	4.226	BB
2	42.03	1074340	3808	50.05%	10.566	BB
Total		2,146,654	11,755	100.00%		



5

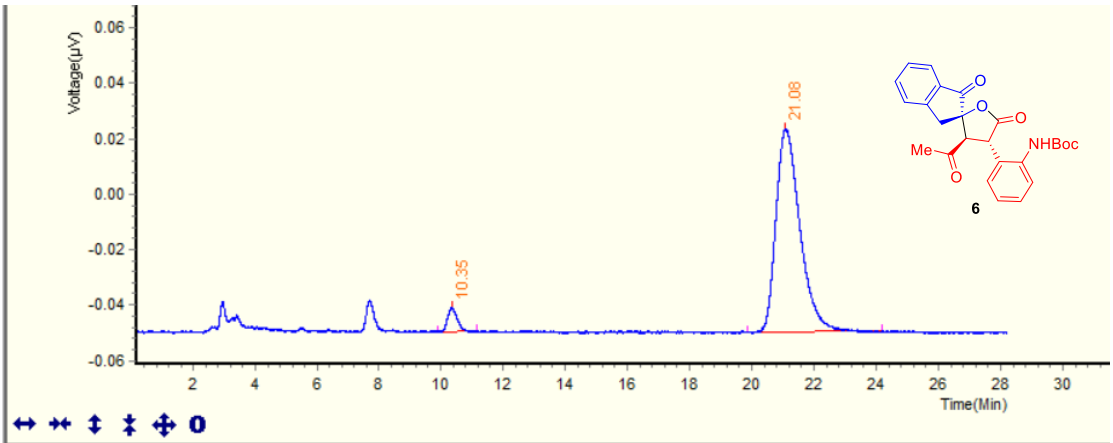
Integration Result | Calculation Result | TimeTable

No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	
1	21.03	2598600	18878	92.00%	6.458	BB
2	42.17	225967	895	8.00%	8.64	BB
Total		2,824,567	19,773	100.00%		



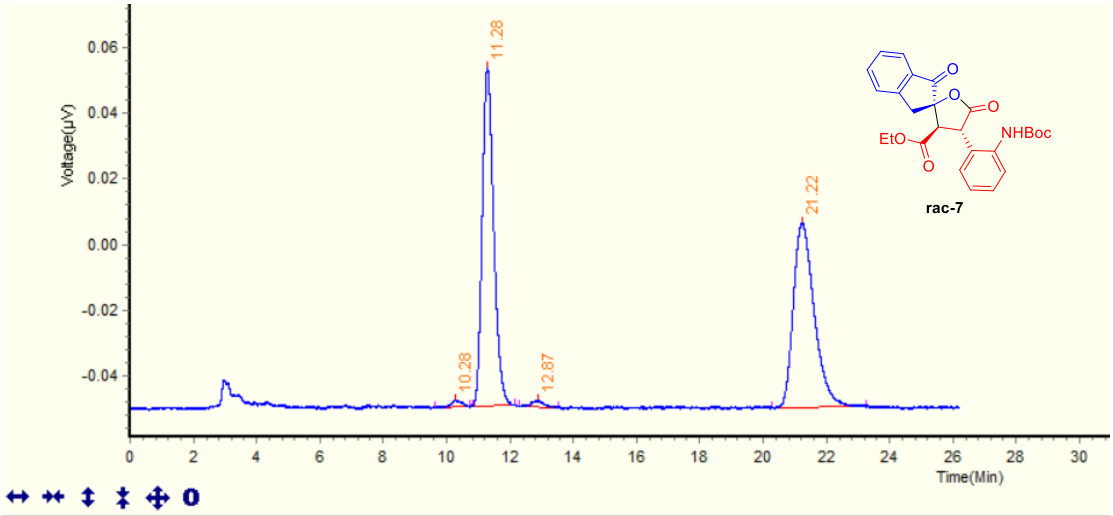
Integration Result | Calculation Result | TimeTable

No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	
1	10.43	399168	18272	50.05%	0.924	BB
2	21.31	398301	7577	49.95%	3.453	BB
Total		797,469	25,849	100.00%		



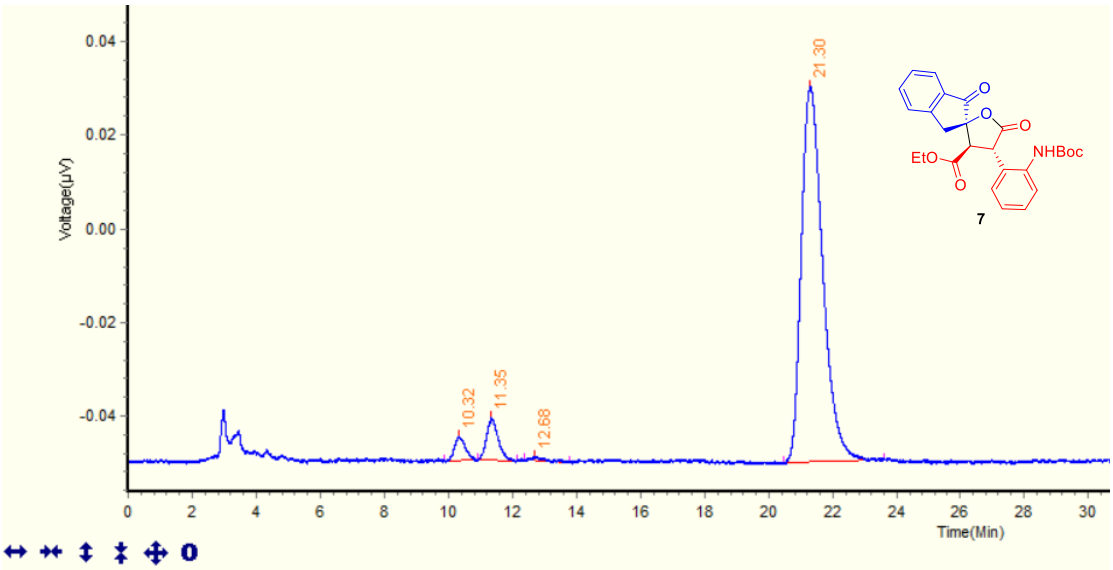
Integration Result | Calculation Result | TimeTable

No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	
1	10.35	90714	4242	4.42%	1.257	BB
2	21.08	1961083	36535	95.58%	4.353	BB
Total		2,051,797	40,777	100.00%		



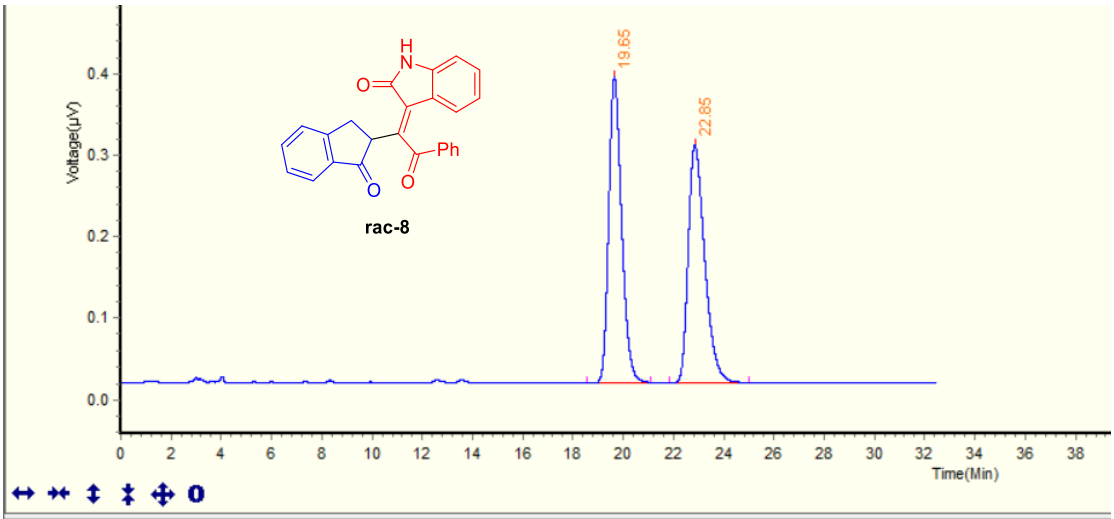
Integration Result | Calculation Result | TimeTable

No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	
1	10.28	18226	955	0.70%	1.086	BB
2	11.28	1293800	51500	49.37%	1.331	BB
3	12.87	23245	955	0.89%	1.277	BB
4	21.22	1285550	27940	49.05%	2.987	BB
Total		2,620,821	81,350	100.00%		



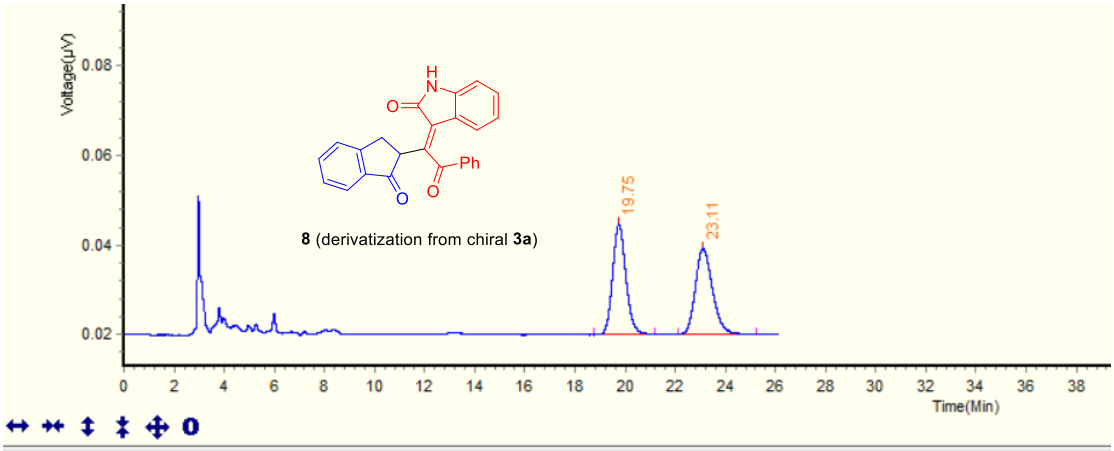
Integration Result | Calculation Result | TimeTable

No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	
1	10.32	60230	2598	2.94%	1.005	BB
2	11.35	110178	4485	5.39%	1.2	BB
3	12.68	11636	455	0.57%	1.428	BB
4	21.30	1863354	40266	91.10%	3.147	BB
Total		2,045,398	47,804	100.00%		



Integration Result | Calculation Result | TimeTable

No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	
1	19.65	6667527	187146	50.06%	2.535	BB
2	22.85	6651082	145560	49.94%	3.168	BB
Total		13,318,609	332,706	100.00%		



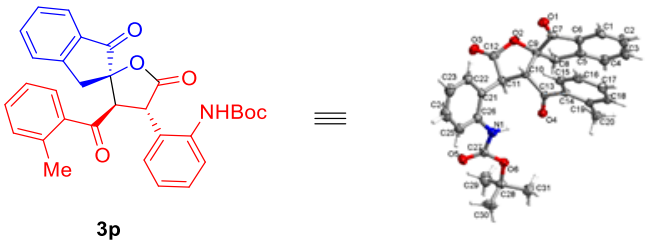
Integration Result | Calculation Result | TimeTable

No.	Retention Time	Peak Area	Peak Height	Peak Area(%)	Peak Width	
1	19.75	451674	12291	49.70%	2.43	BB
2	23.11	457178	9609	50.30%	3.125	BB
Total		908,852	21,900	100.00%		

Single-crystal X-ray diffraction of 3p (CCDC 2167416)

X-ray analysis was carried out using the single crystal which was grown in Hexane/$CHCl_3$.

The instrumentation used for the crystal measurement is Oxford Gemini E X-ray single-crystal diffractometer (ellipsoid contour at 30% probability level).



3p

checkCIF/PLATON report

Structure factors have been supplied for datablock(s) 20220464_sq

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found. CIF dictionary Interpreting this report

Datablock: 20220464_sq

Bond precision: C-C = 0.0073 A Wavelength=1.54184

Cell: a=15.8569(2) b=17.1648(2) c=35.0258(4)
alpha=90 beta=90 gamma=90
Temperature: 293 K

	Calculated	Reported
Volume	9533.3(2)	9533.3(2)
Space group	P 21 21 21	P 21 21 21
Hall group	P 2ac 2ab	P 2ac 2ab
Moiety formula	3(C31 H29 N O6), C H Cl3 [+ solvent]	3(C31 H29 N O6), C H Cl3
Sum formula	C94 H88 Cl3 N3 O18 [+ solvent]	C94 H88 Cl3 N3 O18
Mr	1654.02	1654.02
Dx,g cm-3	1.152	1.152
Z	4	4
Mu (mm-1)	1.394	1.394
F000	3472.0	3472.0
F000'	3486.70	
h,k,lmax	19,21,42	19,20,42
Nref	18387[10016]	17862
Tmin,Tmax	0.818,0.946	0.745,1.000
Tmin'	0.736	

Correction method= # Reported T Limits: Tmin=0.745 Tmax=1.000
AbsCorr = MULTI-SCAN

Data completeness= 1.78/0.97 Theta(max)= 70.884

R(reflections)= 0.0537(12582) wR2(reflections)= 0.1577(17862)

S = 1.016 Npar= 1064

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

Alert level C

PLAT234_ALERT_4_C Large Hirshfeld Difference C4' --C5' . 0.17 Ang.
PLAT241_ALERT_2_C High 'MainMol' Ueq as Compared to Neighbors of C1' Check
PLAT241_ALERT_2_C High 'MainMol' Ueq as Compared to Neighbors of C2' Check
PLAT241_ALERT_2_C High 'MainMol' Ueq as Compared to Neighbors of C17' Check
PLAT241_ALERT_2_C High 'MainMol' Ueq as Compared to Neighbors of C2" Check
PLAT241_ALERT_2_C High 'MainMol' Ueq as Compared to Neighbors of C17" Check
PLAT242_ALERT_2_C Low 'MainMol' Ueq as Compared to Neighbors of C28 Check
PLAT242_ALERT_2_C Low 'MainMol' Ueq as Compared to Neighbors of C28" Check
PLAT260_ALERT_2_C Large Average Ueq of Residue Including O1' 0.109 Check
PLAT260_ALERT_2_C Large Average Ueq of Residue Including C11 0.201 Check
PLAT260_ALERT_2_C Large Average Ueq of Residue Including C11A 0.201 Check
PLAT334_ALERT_2_C Small Aver. Benzene C-C Dist C1" -C6" 1.37 Ang.
PLAT340_ALERT_3_C Low Bond Precision on C-C Bonds ..... 0.00733 Ang.
PLAT911_ALERT_3_C Missing FCF Refl Between Thmin & STh/L= 0.600 4 Report
PLAT918_ALERT_3_C Reflection(s) with I(obs) much Smaller I(calc) . 1 Check
PLAT987_ALERT_1_C The Flack x is >> 0 - Do a BASF/TWIN Refinement Please Check

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Alert level G

PLAT002_ALERT_2_G Number of Distance or Angle Restraints on AtSite 6 Note
PLAT007_ALERT_5_G Number of Unrefined Donor-H Atoms ..... 3 Report
PLAT033_ALERT_4_G Flack x Value Deviates > 3.0 * sigma from Zero . 0.037 Note
PLAT171_ALERT_4_G The CIF-Embedded .res File Contains EADP Records 4 Report
PLAT172_ALERT_4_G The CIF-Embedded .res File Contains DFIX Records 4 Report
PLAT199_ALERT_1_G Reported _cell_measurement_temperature ..... (K) 293 Check
PLAT200_ALERT_1_G Reported _diffrn_ambient_temperature ..... (K) 293 Check
PLAT302_ALERT_4_G Anion/Solvent/Minor-Residue Disorder (Resd 4) 100% Note
PLAT302_ALERT_4_G Anion/Solvent/Minor-Residue Disorder (Resd 5) 100% Note
PLAT304_ALERT_4_G Non-Integer Number of Atoms in ..... (Resd 4) 3.49 Check
PLAT304_ALERT_4_G Non-Integer Number of Atoms in ..... (Resd 5) 1.51 Check
PLAT432_ALERT_2_G Short Inter X...Y Contact O3' ..C32 . 3.00 Ang.
1/2+x,3/2-y,1-z = 4_566 Check
PLAT432_ALERT_2_G Short Inter X...Y Contact O5' ..C12" . 2.93 Ang.
-1/2+x,3/2-y,1-z = 4_466 Check
PLAT606_ALERT_4_G Solvent Accessible VOID(S) in Structure ..... ! Info
PLAT720_ALERT_4_G Number of Unusual/Non-Standard Labels ..... 6 Note
PLAT791_ALERT_4_G Model has Chirality at C9 (Sohnke SpGr) R Verify
PLAT791_ALERT_4_G Model has Chirality at C9' (Sohnke SpGr) R Verify
PLAT791_ALERT_4_G Model has Chirality at C9" (Sohnke SpGr) R Verify
PLAT791_ALERT_4_G Model has Chirality at C10 (Sohnke SpGr) R Verify
PLAT791_ALERT_4_G Model has Chirality at C10' (Sohnke SpGr) R Verify
PLAT791_ALERT_4_G Model has Chirality at C10" (Sohnke SpGr) R Verify
PLAT791_ALERT_4_G Model has Chirality at C11 (Sohnke SpGr) S Verify
PLAT791_ALERT_4_G Model has Chirality at C11' (Sohnke SpGr) S Verify
PLAT791_ALERT_4_G Model has Chirality at C11" (Sohnke SpGr) S Verify

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PLAT860_ALERT_3_G Number of Least-Squares Restraints .............          4 Note
PLAT869_ALERT_4_G ALERTS Related to the Use of SQUEEZE  Suppressed          ! Info
PLAT910_ALERT_3_G Missing # of FCF Reflection(s) Below Theta(Min).          3 Note
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L=  0.600         98 Note
PLAT941_ALERT_3_G Average HKL Measurement Multiplicity ...........        3.9 Low
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density.          0 Info
PLAT992_ALERT_5_G Repd & Actual _reflns_number_gt Values Differ by          4 Check
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  0 ALERT level A = Most likely a serious problem - resolve or explain
  0 ALERT level B = A potentially serious problem, consider carefully
 16 ALERT level C = Check. Ensure it is not caused by an omission or oversight
 31 ALERT level G = General information/check it is not something unexpected

  3 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
 15 ALERT type 2 Indicator that the structure model may be wrong or deficient
  6 ALERT type 3 Indicator that the structure quality may be low
 21 ALERT type 4 Improvement, methodology, query or suggestion
  2 ALERT type 5 Informative message, check
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It is advisable to attempt to resolve as many as possible of the alerts in all categories. Often the minor alerts point to easily fixed oversights, errors and omissions in your CIF or refinement strategy, so attention to these fine details can be worthwhile. In order to resolve some of the more serious problems it may be necessary to carry out additional measurements or structure refinements. However, the purpose of your study may justify the reported deviations and the more serious of these should normally be commented upon in the discussion or experimental section of a paper or in the "special_details" fields of the CIF. checkCIF was carefully designed to identify outliers and unusual parameters, but every test has its limitations and alerts that are not important in a particular case may appear. Conversely, the absence of alerts does not guarantee there are no aspects of the results needing attention. It is up to the individual to critically assess their own results and, if necessary, seek expert advice.

Publication of your CIF in IUCr journals

A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (*Acta Crystallographica*, *Journal of Applied Crystallography*, *Journal of Synchrotron Radiation*); however, if you intend to submit to *Acta Crystallographica Section C* or *E* or *IUCrData*, you should make sure that full publication checks are run on the final version of your CIF prior to submission.

Publication of your CIF in other journals

Please refer to the *Notes for Authors* of the relevant journal for any special instructions relating to CIF submission.

PLATON version of 19/02/2022; check.def file version of 19/02/2022