Supporting Information

Iridium-Catalyzed Asymmetric Double Allylic Alkylation of Azlactone: Efficient Access to Chiral α-Amino Acid Derivatives

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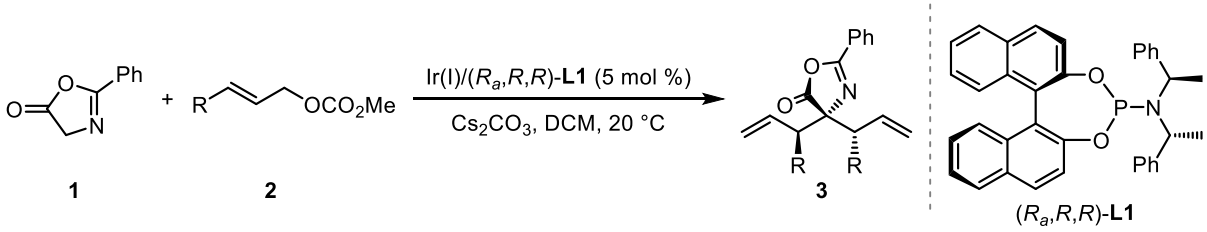
I. General Remarks

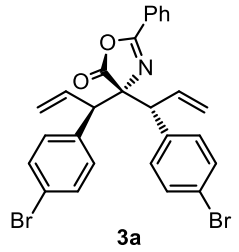
^{1}H NMR spectra were recorded on a Bruker 400 MHz spectrometer in $CDCl_3$. Chemical shifts are reported in ppm with the internal TMS signal at 0.0 ppm as a standard. ^{13}C NMR spectra were recorded on a Bruker 101 MHz spectrometer in $CDCl_3$. Chemical shifts are reported in ppm with the internal chloroform signal at 77.0 ppm as a standard. ^{19}F NMR spectra were recorded on a Bruker 376 MHz spectrometer in $CDCl_3$. Chemical shifts are reported in ppm with the internal CF_3COOH signal at -76.55 ppm. The data are reported as (s = single, d = double, t = triple, q = quarter, m = multiple or unresolved, br s = broad single, coupling constant(s) in Hz, integration). High resolution mass spectra (HR-MS) were recorded on a LTQ-Orbitrap Elite mass spectrometer with MeOH as solvent mixture for the measurements. Commercially obtained reagents were used without further purification. Solvents were purified prior to use according to the standard methods. Unless otherwise noted, all reactions were carried out under nitrogen atmosphere. The enantiomeric excesses (ee) of the products were determined by high-performance liquid chromatography (HPLC) analysis performed on Agilent 1200 and 1260 Series chromatographs using a Diacel chiral column (25 cm). Optical rotations were measured on a Rudolph Research Analytical Autopol VI polarimeter with $[\alpha]_D$ values reported in degrees; concentration (c) is in g/100 mL. All reactions were reacted under Ar_2 atmosphere. Substrates (2-phenyloxazol-5(4*H*)-one) **1** and (allylic carbonates) **2** were prepared according to the literature procedure.[1,2] Chiral ligands (*R*$_a$,*R*,*R*)-**L** was prepared according to the literature procedure.[3] The racemic products were obtained by running reactions with blending equal amount of two enantiomers. The absolute configuration of (*R*,*R*)-**8p** was determined by X-ray analysis, and those of other products were deduced on the basis of this result.

II. General Procedure for Iridium-Catalyzed Asymmetric Double Allylic Alkylation of Azlactone



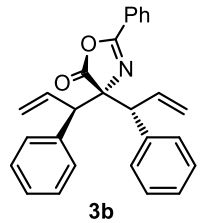
1 **2** **3**

A flame dried Schlenk tube was cooled to rt and evacuated and backfilled with argon for three times. To this Schlenk tube were added $[Ir(COD)Cl]_2$ (0.005 mmol, 2.5 mol %), phosphoramidite ligand (R_a,*R*,*R*)-**L1** (0.01 mmol, 5 mol %), degassed THF (0.5 mL) and degassed *n*-propylamine (0.5 mL). The reaction mixture was heated at 50 °C for 30 min and then the volatile solvents were removed under vacuum to give a pale yellow solid. 2-phenyloxazol-5(4*H*)-one **1** (0.20 mmol), allylic carbonates **2** (0.40 mmol), Cs_2CO_3 (0.20 mmol), DCM (2 mL) were then added, reacted at 20 ℃. After 12 hours of reaction, the mixture was added water, and extracted with dichloromethane (3×). The dichloromethane layers were dried over anhydrous Na_2SO_4, filtered, and evaporated to give crude **3**. The crude product was purified by silica-gel column chromatography to give **3**.



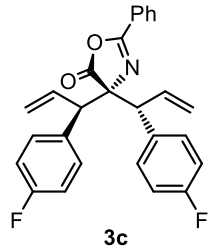
3a

4,4-bis((*R*)-1-(4-bromophenyl)allyl)-2-phenyloxazol-5(4*H*)-one (3a): Yield (102 mg, 92%); white solid, m.p. 82–84 °C; $[\alpha]^{22}_D$ = -64.6 (*c* 0.95, $CHCl_3$); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.74–7.66 (m, 2H), 7.55 – 7.48 (m, 1H), 7.43 – 7.36 (m, 2H), 7.29 – 7.27 (m, 2H), 7.26 – 7.25 (m, 2H), 7.11 – 6.98 (m, 4H), 6.55 (ddd, *J* = 16.8, 10.4, 10.0 Hz, 1H), 6.31 (ddd, *J* = 16.8, 10.4, 10.0 Hz, 1H), 5.38 – 5.18 (m, 4H), 3.96 (d, *J* = 10.0 Hz, 1H), 3.96 (d, *J* = 10.0 Hz, 1H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 177.8, 160.5, 137.6, 137.2, 134.2, 133.1, 132.7, 131.4, 131.2, 130.9, 130.5, 128.7, 127.7, 125.1, 121.6, 121.2, 120.1, 119.9, 78.6, 53.7, 53.6. HRMS (APCI+) Calcd. For $C_{27}H_{22}Br_2NO_2^+$ ($[M+H]^+$): 551.9993, found: 551.9987. The product was analyzed by HPLC to determine the enantiomeric excess: >99% ee (Chiralpak AD-H, *i*-propanol/hexane = 2/98, flow rate 1.0 mL/min, λ = 220 nm); t_r = 5.72 and 6.11 min.



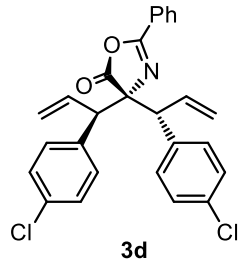
3b

2-phenyl-4,4-bis((*R*)-1-phenylallyl)oxazol-5(4*H*)-one (3b): Yield (69 mg, 88%); white solid, m.p. 119–121 °C; $[\alpha]^{22}_D$ = -133.5 (*c* 0.89, $CHCl_3$); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.69 – 7.57 (m, 2H), 7.50 – 7.42 (m, 1H), 7.38 – 7.29 (m, 2H), 7.21 – 7.17 (m, 2H), 7.16 – 7.08 (m, 7H), 7.08 – 7.02 (m, 1H), 6.68 (ddd, *J* = 17.2, 10.4, 10.0 Hz, 1H), 6.42 (ddd, *J* = 17.2, 10.4, 10.0 Hz, 1H), 5.39 – 5.22 (m, 4H), 4.05 (d, *J* = 10.0 Hz, 1H), 4.04 (d, *J* = 10.0 Hz, 1H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 178.1, 160.0, 138.8, 138.3, 134.9, 133.7, 132.3, 129.2, 128.8, 128.5, 128.2, 128.1, 127.6, 127.3, 127.1, 125.6, 119.5, 119.3, 79.1, 54.4, 54.2. HRMS (APCI+) Calcd. For $C_{27}H_{24}NO_2^+$ ($[M+H]^+$): 394.1802, found: 394.1800. The product was analyzed by HPLC to determine the enantiomeric excess: 99% ee (Chiralpak IE, *i*-propanol/hexane = 1/99, flow rate 0.5 mL/min, λ = 254 nm); t_r = 9.98 and 11.32 min.



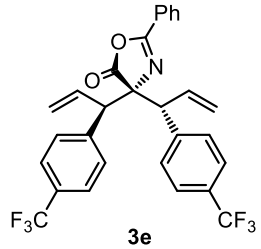
3c

4,4-bis((*R*)-1-(4-fluorophenyl)allyl)-2-phenyloxazol-5(4*H*)-one (3c): Yield (70 mg, 81%); white solid, m.p. 80–82 °C; $[\alpha]^{22}_D$ = -38.0 (*c* 0.94, $CHCl_3$); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.71 – 7.64 (m, 2H), 7.52 – 7.46 (m, 1H), 7.41 – 7.33 (m, 2H), 7.19 – 7.07 (m, 4H), 6.87 – 6.77 (m, 4H), 6.60 (ddd, *J* = 17.2, 10.4, 10.0 Hz, 1H), 6.35 (ddd, *J* = 17.2, 10.4, 10.0 Hz, 1H), 5.39 – 5.19 (m, 1H), 4.004 (d, *J* = 10.0 Hz, 1H), 3.999 (d, *J* = 10.0 Hz, 1H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 178.0, 162.0 (d, *J* = 246.4 Hz), 161.8 (d, *J* = 246.4 Hz), 160.3, 134.6, 134.4 (d, *J* = 3.0 Hz), 134.0 (d, *J* = 3.0 Hz), 133.3, 132.6, 130.7 (d, *J* = 8.1 Hz), 130.3 (d, *J* = 8.1 Hz), 128.6, 127.6, 125.2, 119.8, 119.6, 115.1 (d, *J* = 21.2 Hz), 114.9 (d, *J* = 21.2 Hz), 79.0, 53.5, 53.3. ^{19}F NMR (376 MHz, Chloroform-*d*) δ -114.88 – -115.05 (m), -115.18 – -115.33 (m). HRMS (APCI+) Calcd. For $C_{27}H_{22}F_2NO_2^+$ ($[M+H]^+$): 430.1613, found: 430.1612. The product was analyzed by HPLC to determine the enantiomeric excess: >99% ee (Chiralpak IE, *i*-propanol/hexane = 1/99, flow rate 0.5 mL/min, λ = 254 nm); t_r = 5.00 and 5.23 min.



3d

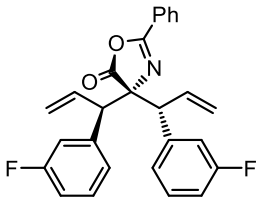
4,4-bis((*R*)-1-(4-chlorophenyl)allyl)-2-phenyloxazol-5(4*H*)-one (3d): Yield (84 mg, 91%); white solid, m.p. 91–93 °C; $[\alpha]^{22}_{D}$ = -65.4 (*c* 0.97, $CHCl_3$); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.73 – 7.65 (m, 2H), 7.56 – 7.48 (m, 1H), 7.43 – 7.35 (m, 2H), 7.14 – 7.06 (m, 8H), 6.56 (ddd, *J* = 17.2, 10.4, 10.0 Hz, 1H), 6.32 (ddd, *J* = 17.2, 10.4, 10.0 Hz, 1H), 5.40 – 5.18 (m, 4H), 3.98 (d, *J* = 10.0 Hz, 1H), 3.975 (d, *J* = 10.0 Hz, 1H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 177.8, 160.5, 137.1, 136.7, 134.3, 133.3, 133.1, 133.0, 132.7, 130.5, 130.2, 128.7, 128.4, 128.2, 127.7, 125.1, 120.0, 119.8, 78.8, 53.6, 53.5. HRMS (APCI+) Calcd. For $C_{27}H_{22}Cl_2NO_2^+$ ($[M+H]^+$): 462.1022, found: 462.1016. The product was analyzed by HPLC to determine the enantiomeric excess: 98% ee (Chiralpak AD-H, *i*-propanol/hexane = 2/98, flow rate 1.0 mL/min, λ = 220 nm); t_r = 5.25 and 5.71 min.



3e

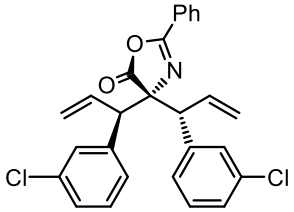
2-phenyl-4,4-bis((*R*)-1-(4-(trifluoromethyl)phenyl)allyl)oxazol-5(4*H*)-one (3e): Yield (97 mg, 92%); white syrupy liquid; $[\alpha]^{22}_{D}$ = -44.9 (*c* 0.85, $CHCl_3$); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.72 – 7.62 (m, 2H), 7.55 – 7.48 (m, 1H), 7.44 – 7.35 (m, 6H), 7.35 – 7.30 (m, 2H), 7.29 – 7.26 (m, 2H), 6.59 (ddd, *J* = 17.2, 10.4, 10.0 Hz, 1H), 6.36 (ddd, *J* = 17.2, 10.4, 10.0 Hz, 1H), 5.44 – 5.22 (m, 4H), 4.09 (d, *J* = 10.0 Hz, 1H), 4.07 (d, *J* = 10.0 Hz, 1H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 177.6, 160.7, 142.5, 142.1, 133.9, 132.9, 132.7, 129.64 (q, *J* = 32.3 Hz), 129.57, 129.48 (q, *J* = 32.3 Hz), 129.3, 128.7, 127.6, 125.2 (q, *J* = 4.0 Hz), 125.0 (q, *J* = 4.0 Hz), 124.9, 123.94 (q, *J* = 272.7 Hz), 123.91 (q, *J* = 272.7 Hz), 120.6, 120.4, 78.5, 54.2, 53.9. ^{19}F NMR (376 MHz, Chloroform-*d*) δ -62.64, -62.69. HRMS (APCI+) Calcd. For $C_{29}H_{22}F_6NO_2^+$ ($[M+H]^+$): 530.1549, found: 530.1553. The product

was analyzed by HPLC to determine the enantiomeric excess: >99% ee (Chiralpak IE, *i*-propanol/hexane = 1/99, flow rate 0.5 mL/min, λ = 254 nm); t_r = 7.76 and 8.12 min.



3f

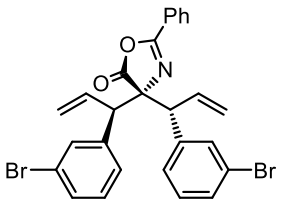
4,4-bis((*R*)-1-(3-fluorophenyl)allyl)-2-phenyloxazol-5(4*H*)-one (3f): Yield (82 mg, 96%); white solid, m.p. 77–79 °C; $[\alpha]^{22}_D$ = -35.2 (*c* 0.97, $CHCl_3$); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.73 – 7.66 (m, 2H), 7.53 – 7.45 (m, 1H), 7.43 – 7.34 (m, 2H), 7.14 – 7.07 (m, 2H), 7.00 – 6.74 (m, 6H), 6.55 (ddd, *J* = 17.2, 10.0, 10.0 Hz, 1H), 6.32 (ddd, *J* = 17.2, 10.0, 10.0 Hz, 1H), 5.38 – 5.22 (m, 4H), 4.01 (d, *J* = 10.0 Hz, 1H), 3.99 (d, *J* = 10.0 Hz, 1H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 177.7, 162.34 (d, *J* = 247.5 Hz), 162.29 (d, *J* = 247.5 Hz), 160.5, 141.0 (d, *J* = 8.1 Hz), 140.6 (d, *J* = 8.1 Hz), 134.2, 133.0, 132.6, 129.7 (d, *J* = 8.1 Hz), 129.5 (d, *J* = 8.1 Hz), 128.6, 127.7, 125.2, 124.9 (d, *J* = 3.0 Hz), 124.4 (d, *J* = 3.0 Hz), 120.1, 119.9, 116.2 (d, *J* = 22.2 Hz), 115.9 (d, *J* = 22.2 Hz), 114.3 (d, *J* = 21.2 Hz), 114.1 (d, *J* = 21.1 Hz), 78.7, 54.1 (d, *J* = 2.0 Hz), 53.8 (d, *J* = 2.0 Hz). ^{19}F NMR (376 MHz, Chloroform-*d*) δ -112.78 – -112.94 (m), -113.02 – -113.16 (m). HRMS (APCI+) Calcd. For $C_{27}H_{22}F_2NO_2^+$ ($[M+H]^+$): 430.1613, found: 430.1614. The product was analyzed by HPLC to determine the enantiomeric excess: >99% ee (Chiralpak IE, *i*-propanol/hexane = 1/99, flow rate 0.5 mL/min, λ = 254 nm); t_r = 9.53 and 10.45 min.



3g

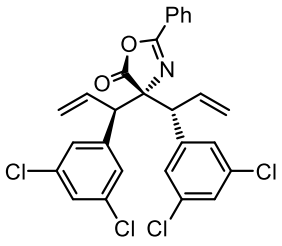
4,4-bis((*R*)-1-(3-chlorophenyl)allyl)-2-phenyloxazol-5(4*H*)-one (3g): Yield (84 mg, 91%); white syrupy liquid; $[\alpha]^{22}_D$ = -61.6 (*c* 0.92, $CHCl_3$); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.75 – 7.67 (m, 2H), 7.55 – 7.48 (m, 1H), 7.43 – 7.36 (m, 2H), 7.22 – 7.19 (m, 1H), 7.14 – 7.05 (m, 7H), 6.52 (ddd, *J* = 17.2, 10.4, 10.0 Hz, 1H), 6.29 (ddd, *J* = 17.2, 10.4, 10.0 Hz, 1H), 5.39 – 5.20 (m, 4H), 3.97 (d, *J* =

10.0 Hz, 1H), 3.94 (d, *J* = 10.0 Hz, 1H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 177.7, 160.6, 140.5, 140.0, 134.1, 133.9, 133.8, 133.0, 132.7, 129.5, 129.4, 128.6, 127.7, 127.6, 127.4, 127.3, 126.7, 125.2, 120.2, 120.0, 78.6, 54.0, 53.9. HRMS (APCI+) Calcd. For $C_{27}H_{22}Cl_2NO_2^+$ ([M+H]$^+$): 462.1022, found: 462.1021. The product was analyzed by HPLC to determine the enantiomeric excess: >99% ee (Chiralpak IE, *i*-propanol/hexane = 1/99, flow rate 0.5 mL/min, λ = 254 nm); t_r = 9.28 and 10.14 min.



3h

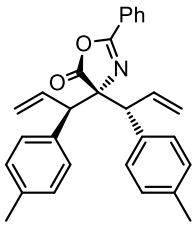
4,4-bis((*R*)-1-(3-bromophenyl)allyl)-2-phenyloxazol-5(4*H*)-one (3h): Yield (98 mg, 89%); white solid, m.p. 94–96 °C; $[\alpha]^{22}_D$ = -60.4 (*c* 0.98, $CHCl_3$); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.76 – 7.69 (m, 2H), 7.55 – 7.48 (m, 1H), 7.43 – 7.33 (m, 3H), 7.29 – 7.26 (m, 2H), 7.25 – 7.21 (m, 1H), 7.16 – 7.08 (m, 2H), 7.06 – 7.00 (m, 2H), 6.51 (ddd, *J* = 17.2, 10.4, 10.0 Hz, 1H), 6.28 (ddd, *J* = 17.2, 10.4, 10.0 Hz, 1H), 5.39 – 5.19 (m, 4H), 3.95 (d, *J* = 10.0 Hz, 1H), 3.93 (d, *J* = 10.0 Hz, 1H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 177.6, 160.7, 140.7, 140.3, 134.1, 132.9, 132.7, 132.4, 132.4, 130.5, 130.3, 129.8, 129.7, 128.6, 127.8, 127.7, 127.1, 125.1, 122.2, 122.0, 120.3, 120.1, 78.6, 53.93, 53.85. HRMS (APCI+) Calcd. For $C_{27}H_{22}Br_2NO_2^+$ ([M+H]$^+$): 551.9993, found: 551.9988. The product was analyzed by HPLC to determine the enantiomeric excess: >99% ee (Chiralpak IE, *i*-propanol/hexane = 1/99, flow rate 0.5 mL/min, λ = 254 nm); t_r = 9.37 and 10.59 min.



3i

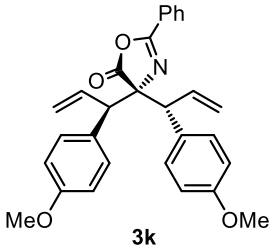
4,4-bis((*R*)-1-(3,5-dichlorophenyl)allyl)-2-phenyloxazol-5(4*H*)-one (3i): Yield (91 mg, 86%); white solid, m.p. 52–54 °C; $[\alpha]^{30}_D$ = -71.8 (*c* 1.02, $CHCl_3$); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.83 – 7.74 (m, 2H), 7.59 – 7.52 (m, 1H), 7.46 – 7.40 (m, 2H), 7.18 – 7.15 (m, 1H), 7.15 – 7.08 (m, 3H), 7.07 –

7.02 (m, 2H), 6.37 (ddd, *J* = 17.2, 10.0, 10.0 Hz, 1H), 6.18 (ddd, *J* = 17.2, 10.0, 10.0 Hz, 1H), 5.39 – 5.18 (m, 4H), 3.90 (d, *J* = 10.0 Hz, 1H), 4.86 (d, *J* = 10.0 Hz, 1H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 177.2, 161.2, 141.5, 141.1, 134.7, 134.6, 133.3, 133.1, 132.3, 128.8, 127.9, 127.79, 127.77, 127.6, 127.5, 124.8, 121.0, 120.8, 78.2, 53.69, 53.67. HRMS (APCI+) Calcd. For $C_{27}H_{20}Cl_4NO_2^+$ ([M+H]$^+$): 532.0216, found: 532.0209. The product was analyzed by HPLC to determine the enantiomeric excess: 99% ee (Chiralpak IE, *i*-propanol/hexane = 2/98, flow rate 1.0 mL/min, λ = 254 nm); t_r = 4.27 and 4.62 min.



3j

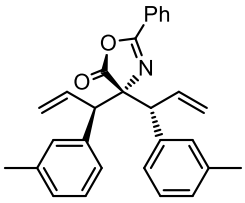
2-phenyl-4,4-bis((*R*)-1-(p-tolyl)allyl)oxazol-5(4*H*)-one (3j): Yield (69 mg, 82%); white solid, m.p. 96–98 °C; $[\alpha]^{22}_D$ = -59.9 (*c* 0.93, $CHCl_3$); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.70 – 7.62 (m, 2H), 7.49 – 7.43 (m, 1H), 7.39 – 7.30 (m, 2H), 7.10 – 6.99 (m, 4H), 6.96 – 6.88 (m, 4H), 6.67 (ddd, *J* = 17.2, 10.4, 10.0 Hz, 1H), 6.40 (ddd, *J* = 17.2, 10.4, 10.0 Hz, 1H), 5.37 – 5.19 (m, 4H), 4.00 (d, *J* = 10.0 Hz, 2H), 2.19 (s, 3H), 2.15 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 178.2, 160.0, 136.9, 136.6, 135.8, 135.4, 135.2, 133.9, 132.2, 128.94, 128.91, 128.7, 128.6, 128.4, 127.7, 125.8, 119.2, 119.0, 79.2, 54.0, 53.8, 21.0, 20.9. HRMS (APCI+) Calcd. For $C_{29}H_{28}NO_2^+$ ([M+H]$^+$): 422.2115, found: 422.2112. The product was analyzed by HPLC to determine the enantiomeric excess: 99% ee (Chiralpak AD-H, *i*-propanol/hexane = 2/98, flow rate 1.0 mL/min, λ = 360 nm); t_r = 4.93 and 7.26 min.



3k

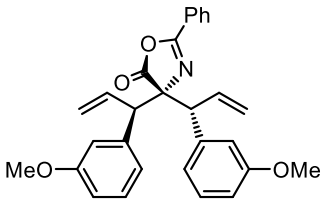
4,4-bis((*R*)-1-(4-methoxyphenyl)allyl)-2-phenyloxazol-5(4*H*)-one (3k): Yield (68 mg, 75%); white solid, m.p. 112–114 °C; $[\alpha]^{22}_D$ = -59.3 (*c* 0.93, $CHCl_3$); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.71 –

7.64 (m, 2H), 7.51 – 7.43 (m, 1H), 7.39 – 7.32 (m, 2H), 7.14 – 7.03 (m, 4H), 6.74 – 6.65 (m, 4H), 6.64 (ddd, *J* = 17.2, 10.0, 9.6 Hz, 1H), 6.37 (ddd, *J* = 17.2, 10.0, 9.6 Hz, 1H), 5.37 – 5.15 (m, 4H), 3.98 (d, *J* = 10 Hz, 2H), 3.68 (s, 3H), 3.66 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 178.3, 160.0, 158.6, 158.4, 135.2, 134.0, 132.3, 130.9, 130.5, 130.2, 129.8, 128.5, 127.7, 125.7, 119.1, 118.9, 113.6, 113.4, 79.4, 55.09, 55.06, 53.4, 53.3. HRMS (APCI+) Calcd. For $C_{29}H_{28}NO_4^+$ ($[M+H]^+$): 454.2013, found: 454.2012. The product was analyzed by HPLC to determine the enantiomeric excess: >99% ee (Chiralpak AD-H, *i*-propanol/hexane = 2/98, flow rate 1.0 mL/min, λ = 220 nm); t_r = 9.42 and 13.65 min.



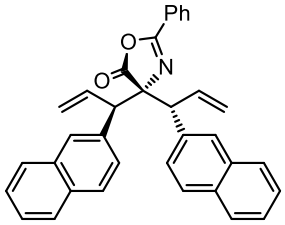
3l

2-phenyl-4,4-bis((*R*)-1-(m-tolyl)allyl)oxazol-5(4*H*)-one (3l): Yield (67 mg, 80%); white syrupy liquid; $[\alpha]^{22}_D$ = -52.0 (*c* 0.75, $CHCl_3$); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.69 – 7.61 (m, 2H), 7.50 – 7.43 (m, 1H), 7.40 – 7.30 (m, 2H), 7.06 – 6.84 (m, 8H), 6.67 (ddd, *J* = 17.2, 10.4, 10.0 Hz, 1H), 6.40 (ddd, *J* = 17.2, 10.4, 10.0 Hz, 1H), 5.39 – 5.21 (m, 4H), 4.01 (d, *J* = 9.6 Hz, 1H), 3.99 (d, *J* = 9.6 Hz, 1H), 2.17 (s, 3H), 2.11 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 178.1, 160.0, 138.7, 138.2, 137.6, 137.5, 135.1, 133.8, 132.2, 130.1, 129.8, 128.4, 128.1, 127.98, 127.96, 127.8, 127.6, 126.0, 125.8, 125.5, 119.3, 119.1, 79.0, 54.3, 54.2, 21.23, 21.20. HRMS (APCI+) Calcd. For $C_{29}H_{28}NO_2^+$ ($[M+H]^+$): 422.2115, found: 422.2108. The product was analyzed by HPLC to determine the enantiomeric excess: >99% ee (Chiralpak IE, *i*-propanol/hexane = 1/99, flow rate 0.5 mL/min, λ = 254 nm); t_r = 8.79 and 10.48 min.



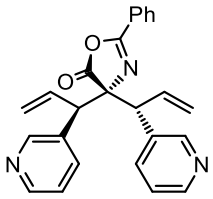
3m

4,4-bis((*R*)-1-(3-methoxyphenyl)allyl)-2-phenyloxazol-5(4*H*)-one (3m): Yield (74 mg, 82%); white syrupy liquid; $[\alpha]^{22}_{D}$ = -47.8 (*c* 0.94, $CHCl_3$); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.73 – 7.66 (m, 2H), 7.50 – 7.44 (m, 1H), 7.40 – 7.32 (m, 2H), 7.10 – 7.01 (m, 2H), 6.83 – 6.78 (m, 1H), 6.78 – 6.72 (m, 2H), 6.70 – 6.62 (m, 3H), 6.62 (ddd, *J* = 17.2, 10.0, 10.0 Hz, 1H), 6.37 (ddd, *J* = 17.2, 10.0, 10.0 Hz, 1H), 5.38 – 5.20 (m, 4H), 4.01 (d, *J* = 10.0 Hz, 1H), 4.00 (d, *J* = 10.0 Hz, 1H), 3.67 (s, 3H), 3.55 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 178.0, 160.2, 159.2, 159.1, 140.3, 139.78, 134.8, 133.7, 132.4, 129.2, 129.1, 128.5, 127.6, 125.7, 121.5, 121.0, 119.5, 119.3, 114.3, 114.0, 113.7, 113.0, 78.9, 55.1, 54.9, 54.4, 54.3. HRMS (ESI+) Calcd. For $C_{29}H_{27}NNaO_4^+$ ($[M+Na]^+$): 476.1832, found: 476.1839. The product was analyzed by HPLC to determine the enantiomeric excess: >99% ee (Chiralpak IE, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 254 nm); t_r = 5.64 and 8.10 min.



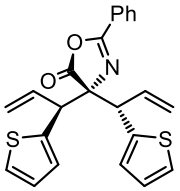
3n

4,4-bis((*R*)-1-(naphthalen-2-yl)allyl)-2-phenyloxazol-5(4*H*)-one (3n): Yield (83 mg, 84%); white solid, m.p. 134–136 °C; $[\alpha]^{22}_{D}$ = -133.5 (*c* 0.99, $CHCl_3$); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.71 – 7.57 (m, 10H), 7.42 – 7.28 (m, 8H), 7.26 – 7.23 (m, 1H), 6.76 (ddd, *J* = 17.2, 10.4, 10.0 Hz, 1H), 6.52 (ddd, *J* = 17.2, 10.0, 10.0 Hz, 1H), 5.43 – 5.23 (m, 4H), 4.25 (d, *J* = 10.0 Hz, 1H), 4.24 (d, *J* = 10.0 Hz, 1H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 178.1, 160.4, 136.3, 135.8, 134.9, 133.9, 133.2, 133.1, 132.6, 132.4, 132.3, 128.6, 128.4, 127.93, 127.90, 127.85, 127.7, 127.5, 127.4, 127.1, 126.9, 125.82, 125.79, 125.66, 125.4, 119.64, 119.62, 79.3, 54.5. HRMS (APCI+) Calcd. For $C_{29}H_{28}NO_4^+$ ($[M+H]^+$): 494.2115, found: 494.2114. The product was analyzed by HPLC to determine the enantiomeric excess: >99% ee (ChiralpakAD-H, *i*-propanol/hexane = 1/99, flow rate 0.5 mL/min, λ = 250 nm); t_r = 7.48 and 10.01 min.



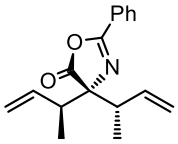
3o

2-phenyl-4,4-bis((*R*)-1-(pyridin-3-yl)allyl)oxazol-5(4*H*)-one (3o): Yield (59 mg, 74%); yellow syrupy liquid; $[\alpha]^{22}_{D}$ = -51.6 (*c* 0.90, $CHCl_3$); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.49 – 8.31 (m, 4H), 7.73 – 7.67 (m, 2H), 7.56 – 7.47 (m, 3H), 7.41 – 7.34 (m, 2H), 7.13 – 7.05 (m, 2H), 6.56 (ddd, *J* = 17.2, 10.4, 10.0 Hz, 1H), 6.35 (ddd, *J* = 17.2, 10.4, 10.0 Hz, 1H), 5.42 – 5.25 (m, 4H), 4.04 (d, *J* = 10.0 Hz, 1H), 4.02 (d, *J* = 10.0 Hz, 1H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 177.5, 160.8, 150.5, 150.4, 148.8, 148.6, 136.6, 135.7, 134.0, 133.7, 133.6, 133.0, 132.3, 128.7, 127.8, 124.7, 123.13, 123.09, 120.8, 120.6, 78.5, 51.9, 51.7; HRMS (APCI+) Calcd. For $C_{25}H_{22}N_3O_2^+$ ([M+H]$^+$): 396.1707, found: 396.1703. The product was analyzed by HPLC to determine the enantiomeric excess: >99% ee (Chiralpak IE, *i*-propanol/hexane = 30/70, flow rate 1.0 mL/min, λ = 250 nm); t_r = 14.78 and 17.99 min.



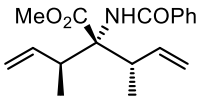
3p

2-phenyl-4,4-bis((*S*)-1-(thiophen-2-yl)allyl)oxazol-5(4*H*)-one (3p): Yield (53 mg, 65%); yellow solid, m.p. 90–92 °C;; $[\alpha]^{22}_{D}$ = -47.9 (*c* 0.97, $CHCl_3$); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.88 – 7.78 (m, 2H), 7.57 – 7.49 (m, 1H), 7.46 – 7.38 (m, 2H), 7.09 – 7.01 (m, 2H), 6.90 – 6.77 (m, 4H), 6.59 (ddd, *J* = 17.2, 10.4, 10.0 Hz, 1H), 6.29 (ddd, *J* = 17.2, 10.4, 10.0 Hz, 1H), 5.43 – 5.25 (m, 4H), 4.41 (d, *J* = 10.0 Hz, 1H), 4.30 (d, *J* = 10.0 Hz, 1H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 177.4, 161.9, 140.5, 140.4, 134.6, 133.1, 132.6, 128.6, 128.0, 126.4, 126.2, 125.8, 125.7, 125.3, 124.6, 120.0, 119.4, 78.9, 49.7, 49.3. HRMS (APCI+) Calcd. For $C_{23}H_{20}NO_2S_2^+$ ([M+H]$^+$): 406.0930, found: 406.0929. The product was analyzed by HPLC to determine the enantiomeric excess: 99% ee (Chiralpak AD-H, *i*-propanol/hexane = 1/99, flow rate 1.0 mL/min, λ = 220 nm); t_r = 12.43 and 13.86 min.



3q

4,4-di((*S*)-but-3-en-2-yl)-2-phenyloxazol-5(4*H*)-one (3q): $[Ir(DBCOT)Cl]_2$ was used instead of $[Ir(COD)Cl]_2$. Yield (50 mg, 92%); white syrupy liquid; $[\alpha]^{22}_D$ = -72.6 (*c* 1.25, $CHCl_3$); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.07 – 7.96 (m, 2H), 7.61 – 7.54 (m, 1H), 7.53 – 7.43 (m, 2H), 5.91 – 7.75 (m, 2H),, 5.18 –5.08 (m, 4H), 2.93 – 2.80 (m, 2H), 1.03 (d, *J* = 6.9 Hz, 6H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 178.9, 160.3, 137.2, 136.7, 132.6, 128.7, 128.0, 125.8, 117.67, 117.65, 78.4, 42.2, 41.9, 15.2, 14.7. HRMS (APCI+) Calcd. For $C_{17}H_{20}NO_2^+$ ($[M+H]^+$): 270.1489, found: 270.1481. The product was analyzed by ^{1}H NMR to determine the diastereomeric excess: 12:1 dr. The product was transformed to **8q** and then analyzed by HPLC to determine the enantiomeric excess: >99% ee.

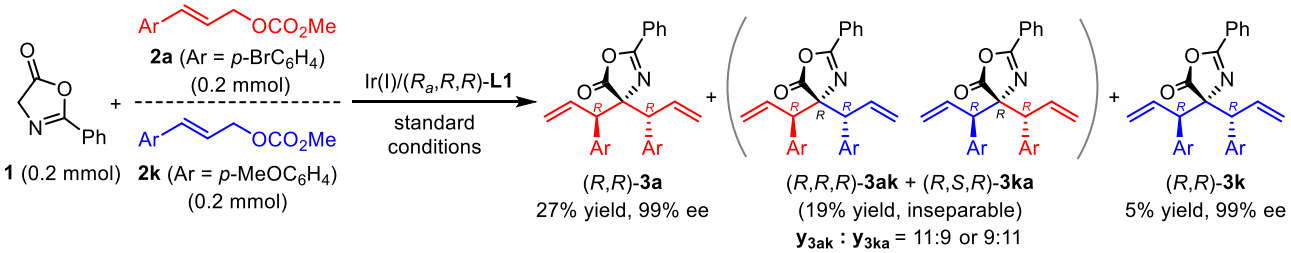


8q

methyl (*S*)-2-benzamido-2-((*S*)-but-3-en-2-yl)-3-methylpent-4-enoate (8q): Yield (48 mg, 80%); white syrupy liquid; $[\alpha]^{22}_D$ = -1.3 (*c* 1.21, $CHCl_3$); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.84 – 7.74 (m, 2H), 7.54 – 7.39 (m, 3H), 5.96 – 5.78 (m, 2H), 5.20 – 4.96 (m, 4H), 3.82 (s, 3H), 3.74 – 3.64 (m, 1H), 3.63 – 3.55 (m, 1H), 1.13 (d, *J* = 6.8 Hz, 3H), 1.12 (d, *J* = 6.8 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 173.7, 166.6, 139.3, 138.9, 135.9, 131.3, 128.6, 126.8, 116.6, 116.2, 71.1, 52.7, 41.7, 40.4, 16.06, 16.05. HRMS (APCI+) Calcd. For $C_{18}H_{24}NO_3^+$ ($[M+H]^+$): 302.1751, found: 302.1749. The product was analyzed by HPLC to determine the enantiomeric excess: >99% ee (Chiralpak AD-H, *i*-propanol/hexane = 1/99, flow rate 1.0 mL/min, λ = 226 nm); t_r = 5.90 and 6.20 min.

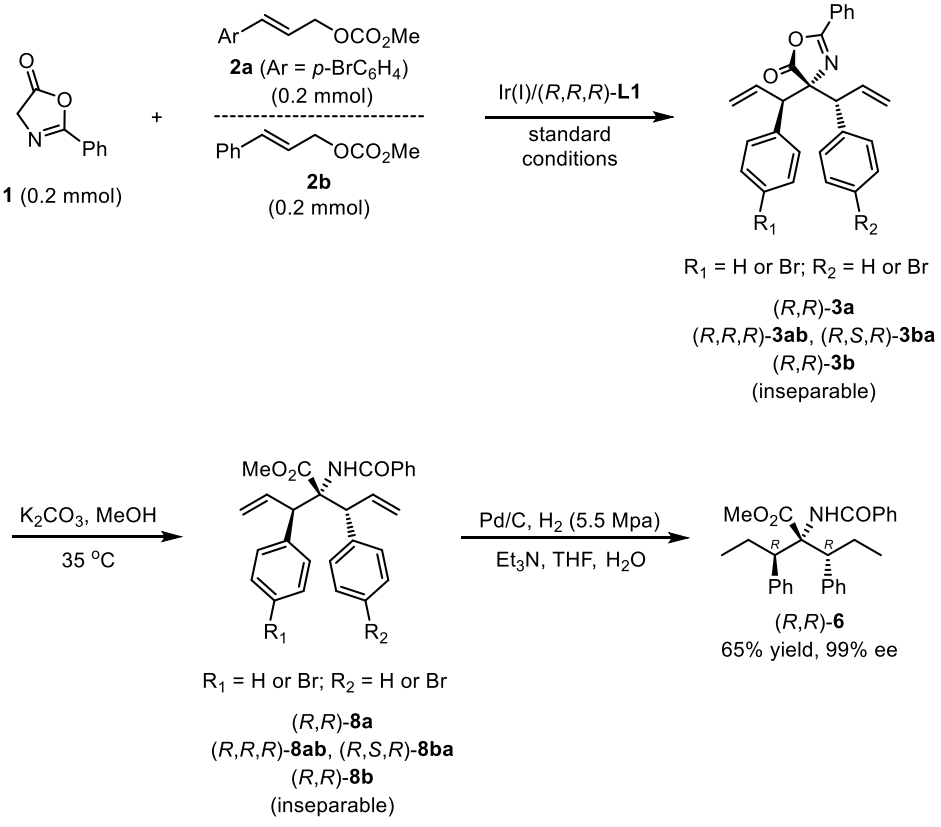
III. Control Experiment

(1) Reaction between **1** and **2a**, **2k**



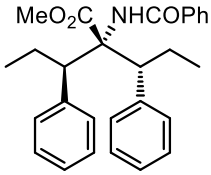
A flame dried Schlenk tube was cooled to rt and evacuated and backfilled with argon for three times. To this Schlenk tube were added $[Ir(COD)Cl]_2$ (0.005 mmol, 2.5 mol %), phosphoramidite ligand (R_a,*R*,*R*)-**L1** (0.01 mmol, 5 mol %), degassed THF (0.5 mL) and degassed *n*-propylamine (0.5 mL). The reaction mixture was heated at 50 °C for 30 min and then the volatile solvents were removed under vacuum to give a pale yellow solid. 2-phenyloxazol-5(4*H*)-one **1** (0.20 mmol), Allylic carbonates **2a** (0.20 mmol), **2k** (0.20 mmol), Cs_2CO_3 (0.20 mmol), DCM (2 mL) were then added, reacted at 20 ℃. After 12 hours of reaction, the mixture was added water, and extracted with dichloromethane (3×). The dichloromethane layers were dried over anhydrous Na_2SO_4, filtered, and evaporated to give crude **3**. The crude product was purified by silica-gel column chromatography to give pure **3a**, pure **3k,** mixture of **3ak** and **3ka**. Therefore, these above results indicated that these double allylic alkylation reactions should be proceeded in step-by-step manner. (*R*,*R*,*R*)-**3ak** and (*R*,*S*,*R*)-**3ka**: yield (19 mg, 19% yield, inseparable); HRMS (APCI+) Calcd. For $C_{28}H_{25}BrNO_3^+$ ($[M+H]^+$): 504.0996, found: 504.0987.

(2) Reaction between **1** and **2a**, **2b**



A flame dried Schlenk tube was cooled to rt and evacuated and backfilled with argon for three times. To this Schlenk tube were added $[Ir(COD)Cl]_2$ (0.005 mmol, 2.5 mol %), phosphoramidite ligand (*Ra,R,R*)-L (0.01 mmol, 5 mol %), degassed THF (0.5 mL) and degassed n-propylamine (0.5 mL). The reaction mixture was heated at 50 °C for 30 min and then the volatile solvents were removed under vacuum to give a pale yellow solid. 2-phenyloxazol-5(4H)-one **1** (0.20 mmol), Allylic carbonates **2a** (0.20 mmol), **2b** (0.20 mmol), Cs_2CO_3 (0.20 mmol), DCM (2 mL) were then added, reacted at 20 ℃. After 12 hours of reaction, the mixture was added water, and extracted with dichloromethane (3×). The dichloromethane layers were dried over anhydrous Na_2SO_4, filtered, and evaporated to give crude **3**. The crude product was purified by silica-gel column chromatography to give mixture of **3a**, **3b**, **3ab** and **3ba**. They can be detected by high resolution mass spectrometry: **3a** (R_1 = R_2 = Br): HRMS (APCI+) Calcd. For $C_{27}H_{22}Br_2NO_2^+$ ($[M+H]^+$): 551.9993, found: 551.9987. **3b** (R_1 = R_2 = H): HRMS (APCI+) Calcd. For $C_{27}H_{24}NO_2^+$ ($[M+H]^+$): 394.1802, found: 394.1797. **3ab/3ba** (R_1 = H, R_2 = Br or R_1 = Br, R_2 = H): HRMS (APCI+) Calcd. For $C_{27}H_{23}BrNO_2^+$ ($[M+H]^+$): 474.0890, found: 474.0887. To a solution of **3a/3b/3ab/3ba** in DCM (2.0 mL) was added K_2CO_3 (0.05 mol, 25 mol%) at 35 ℃ and the reaction mixture was stirred overnight before concentrated in vacuo and purified by silica-gel column chromatography to give **8a/8b/8ab/8ba**. They can be detected by high resolution mass spectrometry: **8a** (R_1 = R_2 = Br): HRMS (APCI+) Calcd. For $C_{28}H_{26}Br_2NO_3^+$ ($[M+H]^+$): 584.0256, found: 584.0249; **8b** (R_1 = R_2 = H): HRMS (APCI+) Calcd. For $C_{28}H_{28}NO_3^+$ ($[M+H]^+$): 426.2064, found: 426.2060; **8ab/8ba** (R_1 = H, R_2 = Br or R_1 = Br, R_2 = H): HRMS (APCI+) Calcd. For $C_{28}H_{27}BrNO_3^+$ ($[M+H]^+$): 506.1153, found: 506.1147.

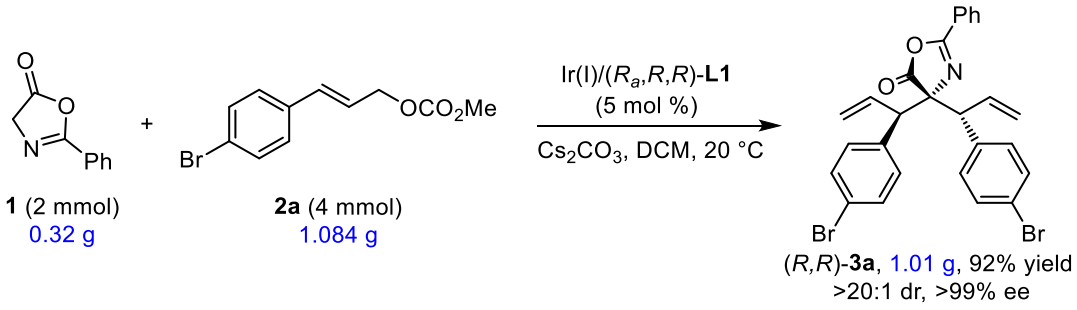
A flame dried 10 mL vial was cooled to rt and a magnetic stir bar was added. To this vial was added the mixture of **8a/8b/8ab/8ba**, Pd/C (10 mg), Et_3N (1.20 mmol), THF (2 mL), H_2O (1 mL). Then we put the vial into the autoclave and fill it with 5.5 MPa hydrogen. The reaction was stirred at room temperature for 24 hours, and then the remaining hydrogen was released. Remove palladium on carbon by filtration, then add water (20 mL) to the system and extract with dichloromethane (3 × 10 mL). The dichloromethane layers were dried over anhydrous Na_2SO_4, filtered, and evaporated to give crude **6**. The crude product was purified by silica-gel column chromatography to give pure **6**.



6

methyl (*R*)-2-benzamido-3-phenyl-2-((*R*)-1-phenylpropyl)pentanoate (6): Yield (56 mg, 65%, the total yield of the three-step reaction); white solid, m.p. 157–159 °C;; $[\alpha]^{22}_{D}$ = -8.4 (*c* 1.12, $CHCl_3$); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.55 – 7.47 (m, 2H), 7.46 – 7.39 (m, 1H), 7.37 – 7.30 (m, 2H), 7.23 – 7.17 (m, 3H), 7.16 – 7.08 (m, 7H), 6.85 (bs, 1H), 4.68 (dd, *J* = 12.4, 2.8 Hz, 1H), 3.74 (s, 3H), 3.57 (dd, *J* = 12.4, 2.8 Hz, 1H), 2.45 – 2.32 (m, 1H), 2.30 – 2.17 (m, 2H), 2.15 – 2.05 (m, 1H), 0.81 (t, *J* = 7.2 Hz, 3H), 0.75 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 173.0, 167.4, 139.9, 139.6, 136.4, 131.1, 129.6, 128.7, 128.5, 128.1, 127.1, 126.9, 126.7, 73.2, 54.2, 52.5, 46.6, 23.5, 22.7, 13.1, 13.0. HRMS (APCI+) Calcd. For $C_{28}H_{32}NO_3^+$ ($[M+H]^+$): 430.2376, found: 430.2373. The product was analyzed by HPLC to determine the enantiomeric excess: >99% ee (Chiralpak AD-H, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 226 nm); t_r = 7.59 and 9.44 min.

IV. Gram-Scale of Double Allylic Alkylation of Azlactone and Synthetic Transformations



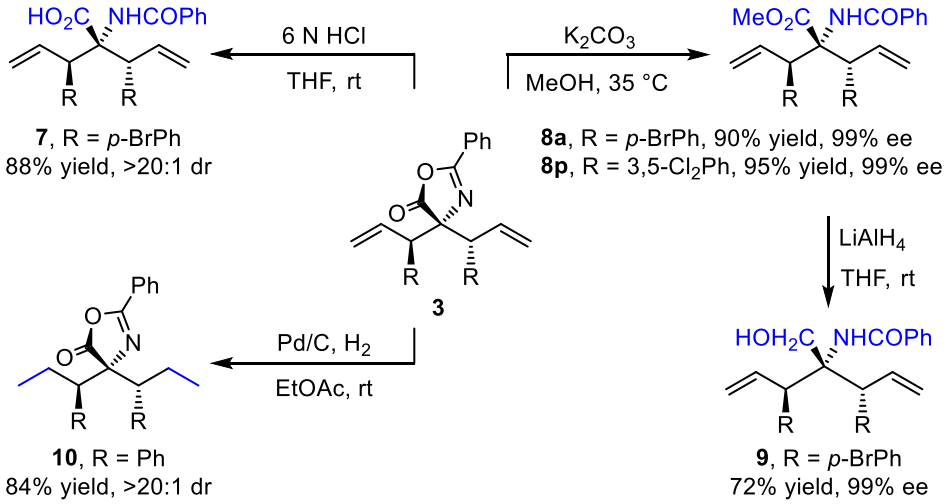
1 (2 mmol)
0.32 g

2a (4 mmol)
1.084 g

(*R,R*)-**3a**, 1.01 g, 92% yield
>20:1 dr, >99% ee

A flame dried Schlenk tube was cooled to rt and evacuated and backfilled with argon for three times. To this Schlenk tube were added $[Ir(COD)Cl]_2$ (0.05 mmol, 2.5 mol %), phosphoramidite ligand (*R*$_a$,*R*,*R*)-**L1** (0.10 mmol, 5.0 mol %), degassed THF (5.0 mL) and degassed *n*-propylamine (5.0 mL). The reaction mixture was heated at 50 °C for 30 min and then the volatile solvents were removed under vacuum to give a pale yellow solid. Allylic carbonate **2a** (4.00 mmol), 2-phenyloxazol-5(4H)-one **1** (2.00 mmol), Cs_2CO_3 (2.00 mmol), DCM (20 mL) were then added, reacted at 20 ℃. After 12 hours of reaction, the mixture was added water, and extracted with dichloromethane (3×). The

dichloromethane layers were dried over anhydrous Na_2SO_4, filtered, and evaporated to give crude **3a**. The crude product was purified by silica-gel column chromatography to give **3a** in 92% yield (1.01 g) with >99% ee.

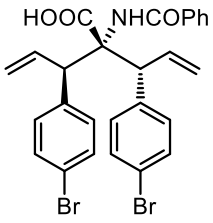


To a solution of **3a** (0.20 mmol) in THF (2.0 mL) was added 6 N HCl (1 mL), the reaction mixture was stirred at room temperature overnight. Then, water (30 mL) was added to the reaction system and extracted with CH_2Cl_2 (3 × 10 mL). The organics were combined and dried over Na_2SO_4 and concentrated under vacuum and purified by silica-gel column chromatography to give **7**.

To a solution of **3a/3p** (0.20 mmol) in DCM (2.0 mL) was added K_2CO_3 (0.05 mol, 25 mol%) at 35 ℃ and the reaction mixture was stirred overnight before concentrated in vacuo and purified by silica-gel column chromatography to give **8a/8p**.

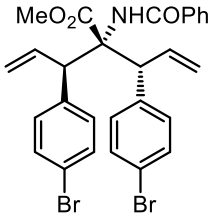
To a solution of **8a** (0.20 mmol) in THF (2.0 mL) was added $LiAlH_4$ (1.00 mmol, 5.0 equiv.) at 0 ℃ and the reaction mixture was stirred overnight. The reaction was quenched with sat. NH_4Cl a.q. and extracted with CH_2Cl_2 (3 x 10 mL) and the combined organics were washed with H_2O (1 x 10 mL), and brine (10 mL). The organics were combined and dried over Na_2SO_4 and concentrated under vacuum and purified by silica-gel column chromatography to give **9**.

A flame dried Schlenk tube was cooled to rt and evacuated and backfilled with hydrogen for three times. To this Schlenk tube were added **3b** (0.20 mmol), Pd/C (10 mg), CH_3CO_2Et (2 mL) and insert the hydrogen balloon below the liquid surface. The reaction mixture was stirred overnight before concentrated in vacuo and purified by silica-gel column chromatography to give **10**.



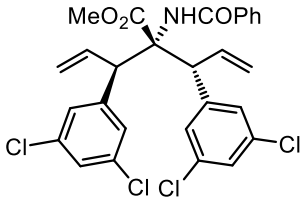
7

(*R*)-2-benzamido-3-(4-bromophenyl)-2-((*R*)-1-(4-bromophenyl)allyl)pent-4-enoic acid (7): Yield (100 mg, 88%); white solid, mp 208–210 °C; $[\alpha]^{22}_{D}$ = -94.9 (*c* 1.14, $CHCl_3$); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.56 – 7.48 (m, 3H), 7.43 – 7.32 (m, 4H), 7.24 (s, 1H), 7.17 – 7.04 (m, 5H), 6.85 (ddd, *J* = 16.8, 9.6, 9.6 Hz, 1H), 6.69 (ddd, *J* = 16.8, 9.6, 9.6 Hz, 1H), 5.37 – 5.21 (m, 4H), 5.12 (d, *J* = 16.8 Hz, 1H), 4.36 (d, *J* = 9.6 Hz, 1H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 174.9, 168.4, 139.0, 138.6, 136.1, 136.0, 135.0, 132.0, 131.51, 131.48, 131.0, 130.3, 128.9, 126.7, 121.4, 121.1, 119.6, 118.9, 70.9, 56.0, 49.7. HRMS (APCI+) Calcd. For $C_{27}H_{24}Br_2NO_3^+$ ($[M+H]^+$): 570.0099, found: 570.0086.



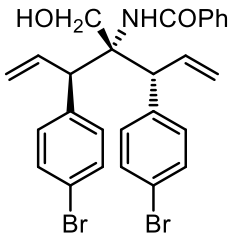
8a

methyl (*R*)-2-benzamido-3-(4-bromophenyl)-2-((*R*)-1-(4-bromophenyl)allyl)pent-4-enoate (8a): Yield (105 mg, 90%); white solid, m.p. 62–64 °C; $[\alpha]^{22}_{D}$ = -90.2 (*c* 0.97, $CHCl_3$); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.57 – 7.46 (m, 3H), 7.43 – 7.33 (m, 4H), 7.28 (s, 1H), 7.06 – 6.95 (m, 5H), 6.82 (ddd, *J* = 17.2, 10.0, 9.2 Hz, 1H), 6.59 (ddd, *J* = 17.2, 10.0, 9.2 Hz, 1H), 5.45 (d, *J* = 9.6 Hz, 1H), 5.33 – 5.23 (m, 3H), 5.12 – 5.02 (m, 1H), 4.32 (d, *J* = 9.6 Hz, 1H), 3.78 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 172.2, 167.5, 139.1, 138.7, 136.1, 135.8, 135.5, 131.7, 131.50, 131.47, 130.7, 130.0, 128.8, 126.6, 121.4, 121.0, 119.8, 118.6, 71.3, 56.3, 53.1, 49.2. HRMS (ESI+) Calcd. For $C_{28}H_{25}Br_2NNaO_3^+$ ($[M+Na]^+$): 606.0075, found: 606.0072. The product was analyzed by HPLC to determine the enantiomeric excess: 99% ee (Chiralpak AD-H, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 226 nm); t_r = 5.28 and 8.50 min.



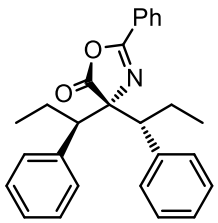
8p

methyl (*R*)-2-benzamido-3-(3,5-dichlorophenyl)-2-((*R*)-1-(3,5-dichlorophenyl)all-yl)pent-4-enoate (8p): Yield (107 mg, 95%); white solid, mp 146–148 °C; $[\alpha]^{30}_{D}$ = -57.5 (*c* 1.12, $CHCl_3$); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.64 – 7.57 (m, 2H), 7.54 – 7.47 (m, 1H), 7.46 – 7.38 (m, 2H), 7.26 – 7.23 (m, 1H), 7.13 – 7.11 (m, 1H), 7.10 – 7.06 (m, 2H), 7.04 – 6.99 (m, 2H), 6.97 – 6.88 (m, 1H), 6.75 (ddd, *J* = 16.8, 10.0, 9.6 Hz, 1H), 6.54 (ddd, *J* = 16.8, 10.0, 9.6 Hz, 1H), 5.47 (d, *J* = 9.6 Hz, 1H), 5.38 – 5.27 (m, 3H), 5.18 – 5.07 (m, 1H), 4.29 (d, *J* = 9.6 Hz, 1H), 3.84 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 171.6, 168.4, 143.2, 142.8, 135.5, 134.83, 134.78, 134.6, 131.8, 128.8, 127.7, 127.6, 127.4, 126.9, 126.6, 121.0, 119.7, 71.2, 56.4, 53.3, 49.1. HRMS (APCI+) Calcd. For $C_{28}H_{24}Cl_4NO_3^+$ ($[M+H]^+$): 564.0479, found: 564.0473. The product was analyzed by HPLC to determine the enantiomeric excess: 99% ee (Chiralpak IE, *i*-propanol/hexane = 2/98, flow rate 1.0 mL/min, λ = 254 nm); t_r = 6.78 and 7.17 min.



9

***N*-((3*R*,5*R*)-3,5-bis(4-bromophenyl)-4-(hydroxymethyl)hepta-1,6-dien-4-yl)benzamide (9):** Yield (80 mg, 72%); white solid, mp 106–108 °C; $[\alpha]^{22}_{D}$ = -71.8 (*c* 0.92, $CHCl_3$); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.63 – 7.56 (m, 2H), 7.55 – 7.48 (m, 1H), 7.46 – 7.38 (m, 4H), 7.34 – 7.26 (m, 3H), 7.25 – 7.20 (m, 3H), 6.61 – 6.49 (m, 2H), 6.47 (bs, 1H), 5.33 – 8.08 (m, 4H), 4.86 (t, *J* = 7.2 Hz, 1H), 4.15 (d, *J* = 10.4 Hz, 1H), 3.85 (d, *J* = 7.2 Hz, 2H), 3.81 (d, *J* = 10.4 Hz, 1H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 167.9, 139.9, 139.1, 137.7, 137.5, 135.1, 131.6, 129.9, 129.6, 128.8, 128.6, 128.5, 127.2, 127.1, 126.6, 119.2, 118.6, 65.8, 64.8, 55.1, 54.7. The product was analyzed by HPLC to determine the enantiomeric excess: 99% ee (Chiralpak IE, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 210 nm); t_r = 27.97 and 29.78 min.



10

2-phenyl-4,4-bis((*R*)-1-phenylpropyl)oxazol-5(4*H*)-one (10): Yield (67 mg, 84%); white solid, mp 134–136 °C; $[\alpha]^{22}_{D}$ = -94.3 (*c* 1.00, $CHCl_3$); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.80 – 7.67 (m, 2H), 7.52 – 7.44 (m, 1H), 7.41 – 7.33 (m, 2H), 7.21 – 6.98 (m, 10H), 3.17 (t, *J* = 11.6 Hz, 1H), 3.16 (t, *J* = 11.6 Hz, 1H), 2.11 – 1.98 (m, 1H), 1.95 – 1.77 (m, 3H), 0.71 (t, *J* = 7.2 Hz, 3H), 0.66 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 179.3, 159.6, 138.2, 137.9, 132.2, 130.2, 128.5, 127.7, 127.6, 127.1, 126.9, 125.8, 80.4, 51.9, 51.5, 22.3, 21.7, 12.2, 12.1. HRMS (APCI$^+$) Calcd. For $C_{27}H_{24}Br_2NO_3^+$ ([M+H]$^+$): 398.2115, found: 398.2107.

V. Absolute Configuration Determination of (*R*,*R*)-8p

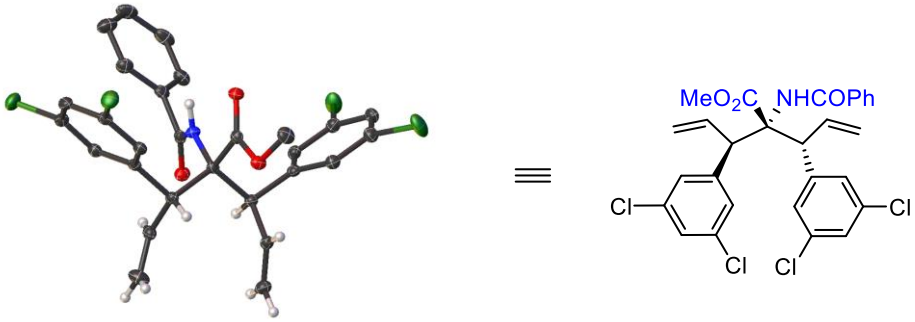


Figure S1. X-ray structure of (*R*,*R*)-**8p**

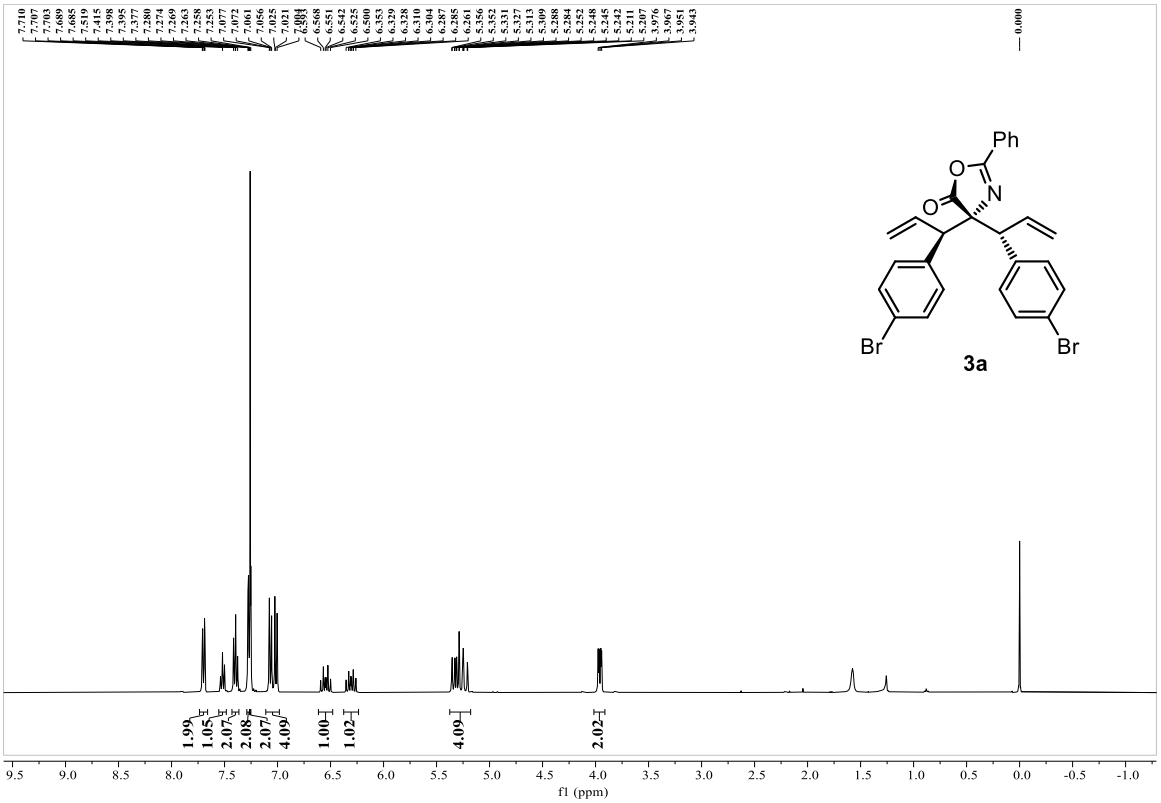
To a 10 mL oven-dried glass sample bottle was added 20 mg pure **8p** with 2 mL dichloromethane to get clear solution, then 7 mL *n*-hexane was slowly added. The mixture solution was sealed with filter paper to slowly grow crystals at room temperature. Crystal data for (*R*,*R*)-**8p**: $C_{28}H_{23}Cl_4NO_3$, M_r = 563.27, *T* = 100 K, Monoclinic, space group *P*2(1)2(1)2(1), *a*=11.76191(4) *b*=13.60474(5) *c*=16.49635(6) Å, *V* = 2639.710(17) Å^3, *Z* = 4, 5297 unique reflections, final R_1 = 0.0193 and wR_2 = 0.0514 for 5307 observed [*I*>2σ(*I*)] reflections, Flack = 0.003(2). CCDC 2130772 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge via www.ccdc.cam.ac.uk/conts/retrieving.html (or from the Cambridge Crystallographic Data Centre, 12,

Union Road, Cambridge CB21EZ, UK; fax: (+44) 1223-336-033; or deposit@ccdc.cam.ac.uk).

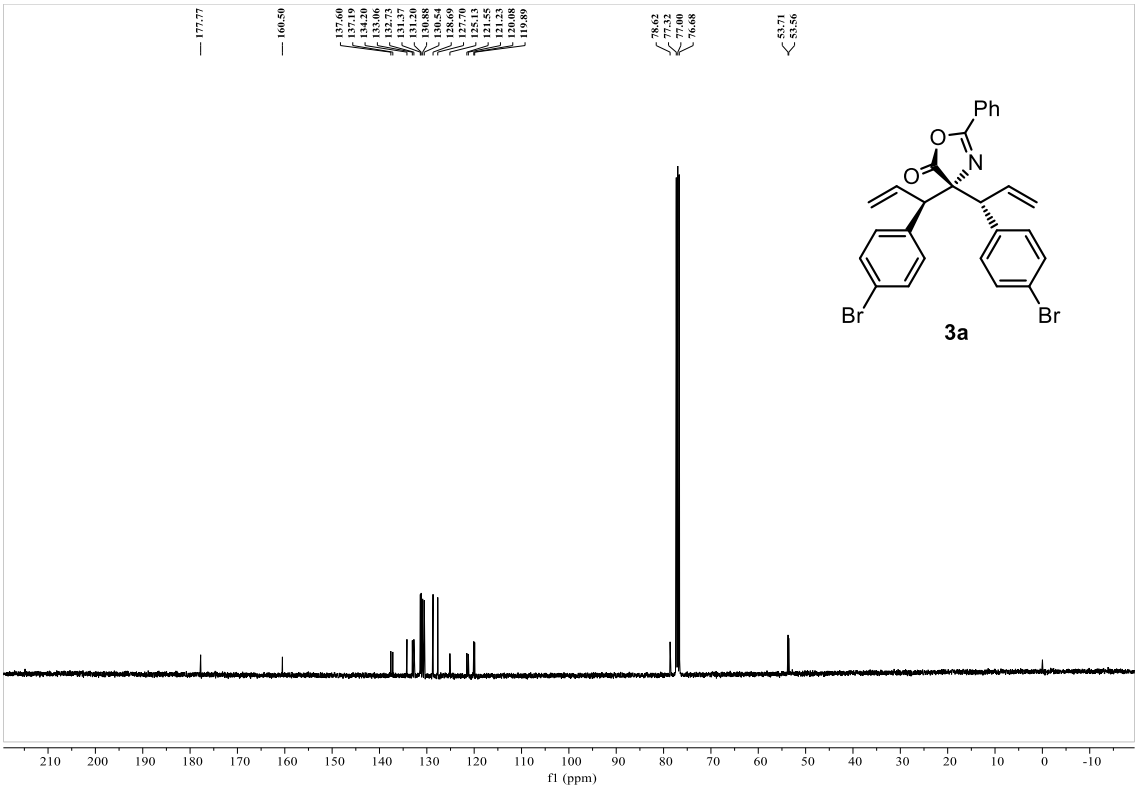
VI. References

1. Izumi, S.; Kobayashi, Y.; Takemoto, Y. Catalytic Asymmetric Synthesis of *anti*-α,β-Diamino Acid Derivatives. *Org. Lett.* **2016**, *18*, 696-699.
2. Trost, B. M.; Miller, J. R.; Hoffman, C. M. A Highly Enantio- and Diastereoselective Molybdenum-Catalyzed Asymmetric Allylic Alkylation of Cyanoesters. *J. Am. Chem. Soc.* **2011**, *133*, 8165-8167.
3. Smith, C. R.; Mans, D. J.; Rajanbabu, T. V. (*R*)-2,2'-Binaphthoyl-(*S*,*S*)-Di(1-Phenylethyl) Aminophosphine. Scalable Protocols for the Syntheses of Phosphoramidite (Feringa) Ligands. *Org. Synth.* **2008**, *85*, 238-247.

VII. NMR and HPLC Spectra



^{1}H NMR (400 MHz, $CDCl_3$) of **3a**



^{13}C NMR (101 MHz, $CDCl_3$) of **3a**

HPLC chromatogram of compound (*rac*)-3a

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Additional Info : Peak(s) manually integrated
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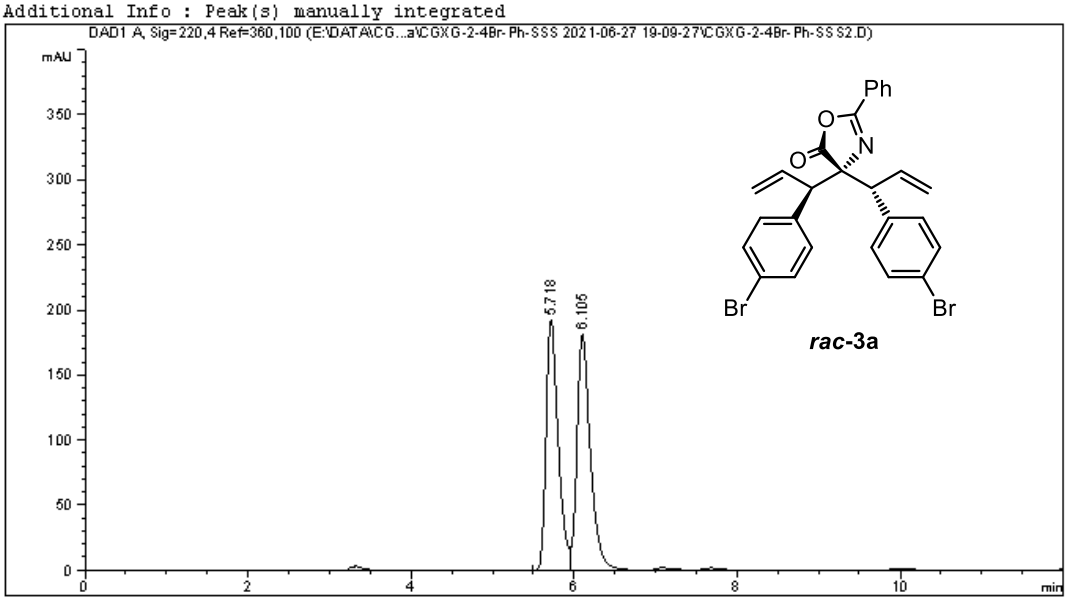


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Totals :                  4036.89941  374.10510
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HPLC chromatogram of compound 3a

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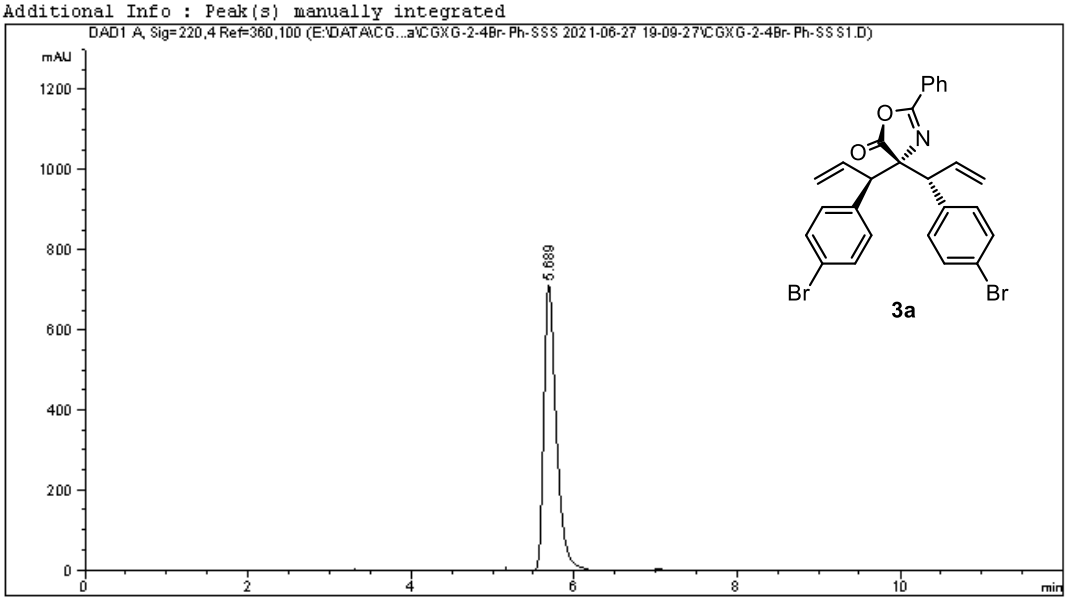


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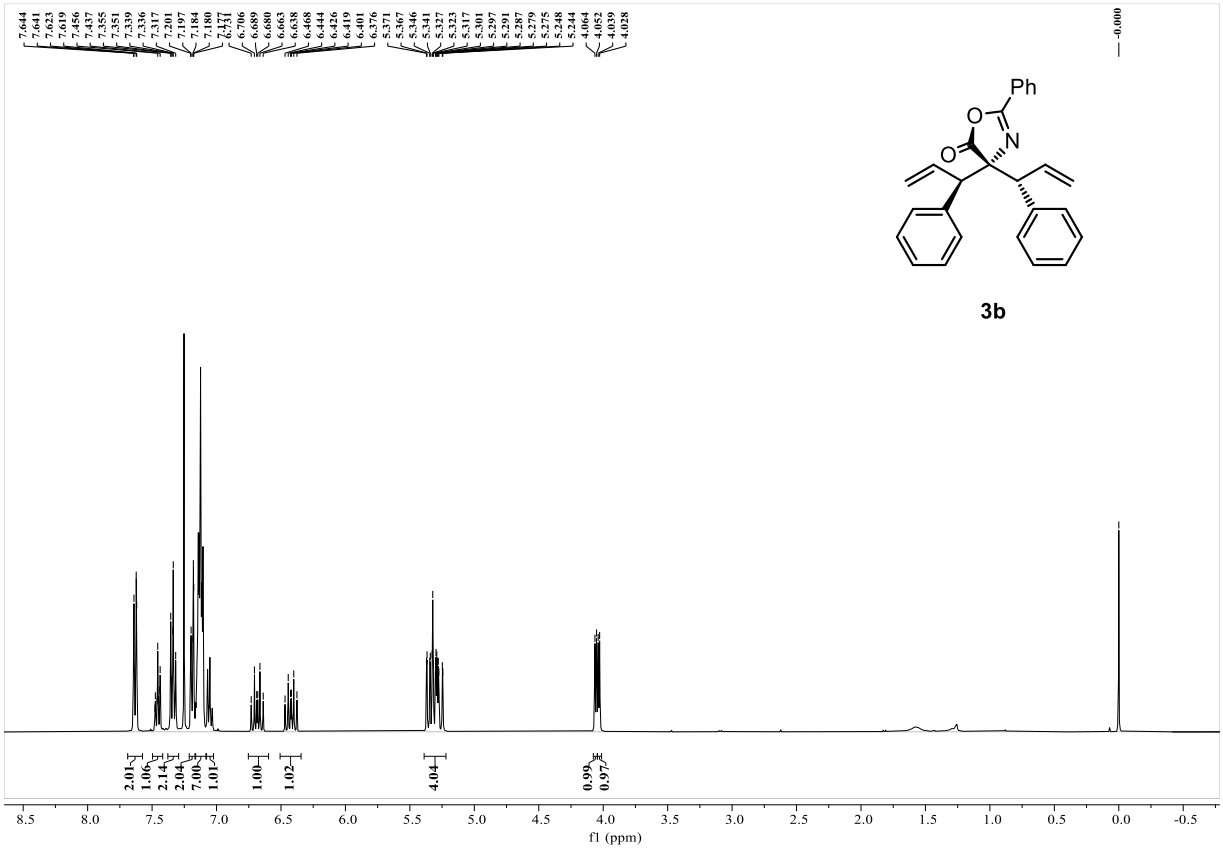
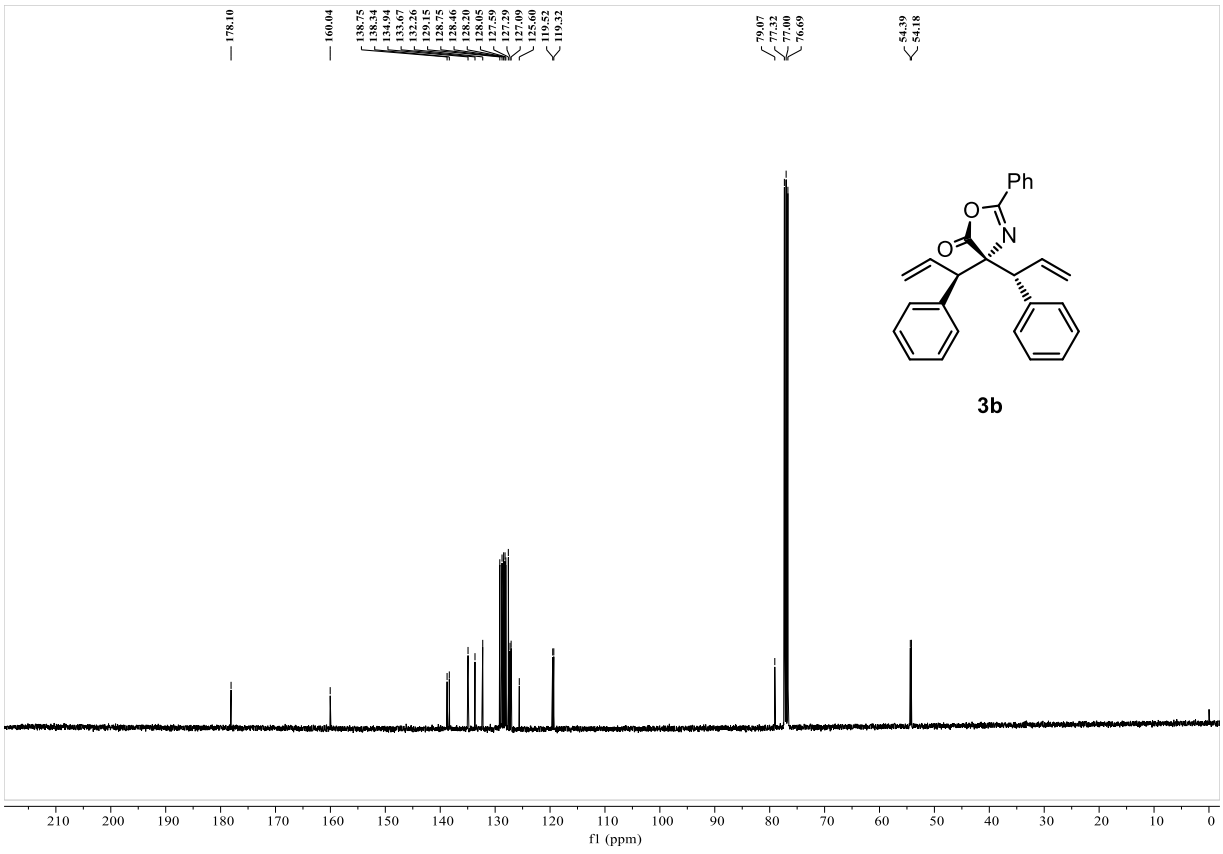


^{1}H NMR (400 MHz, $CDCl_3$) of **3b**



^{13}C NMR (101 MHz, $CDCl_3$) of **3b**

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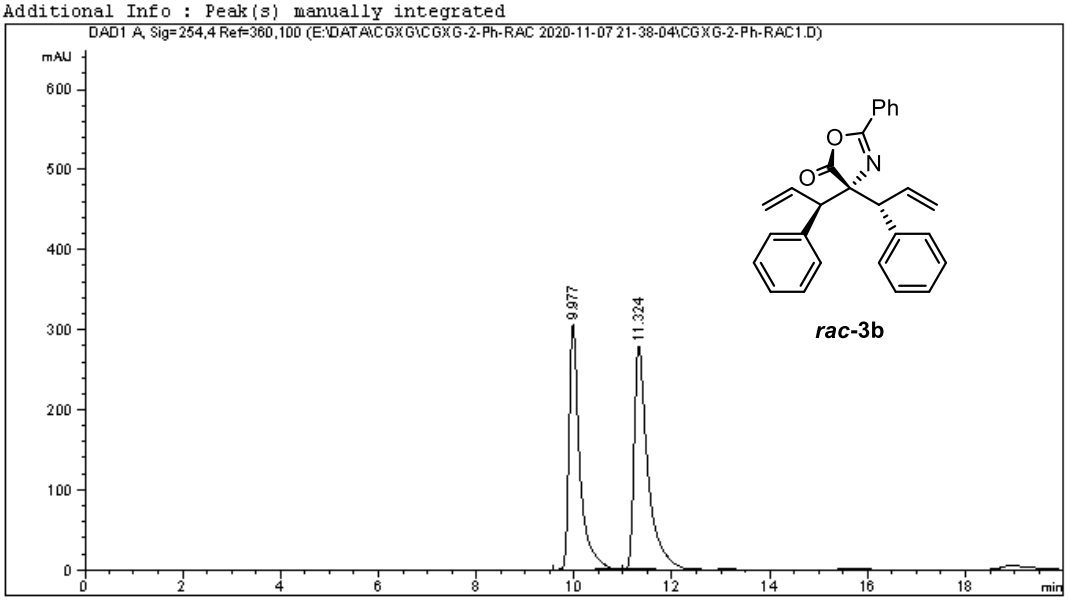


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Do not use Multiplier & Dilution Factor with ISTDs


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Totals :                  1.01068e4   585.11984
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HPLC chromatogram of compound 3b

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Sample Name: CGXG-2-Ph-R

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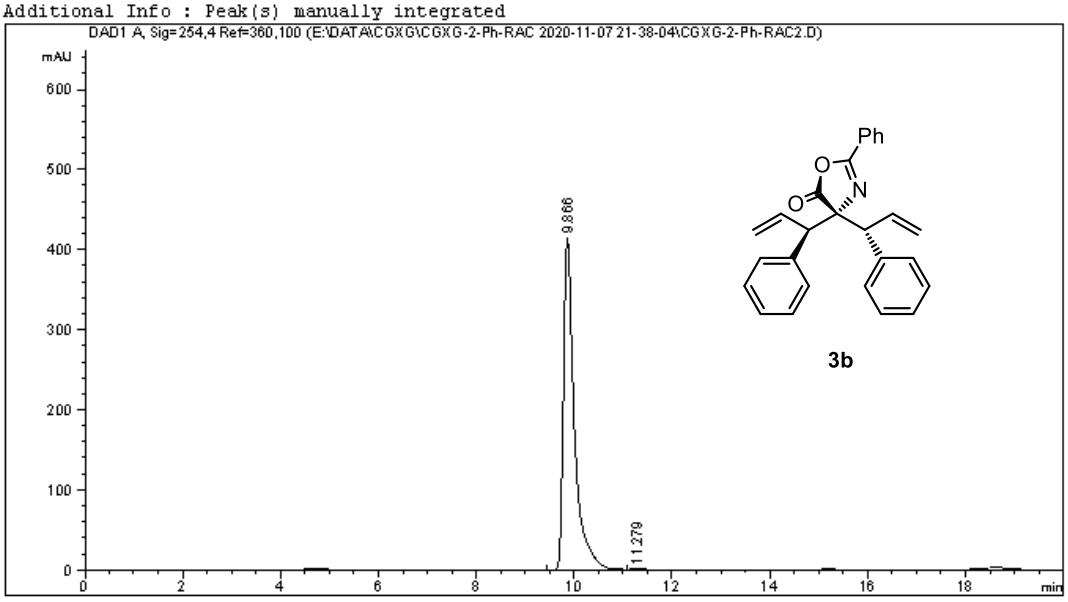

3b

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Do not use Multiplier & Dilution Factor with ISTDs


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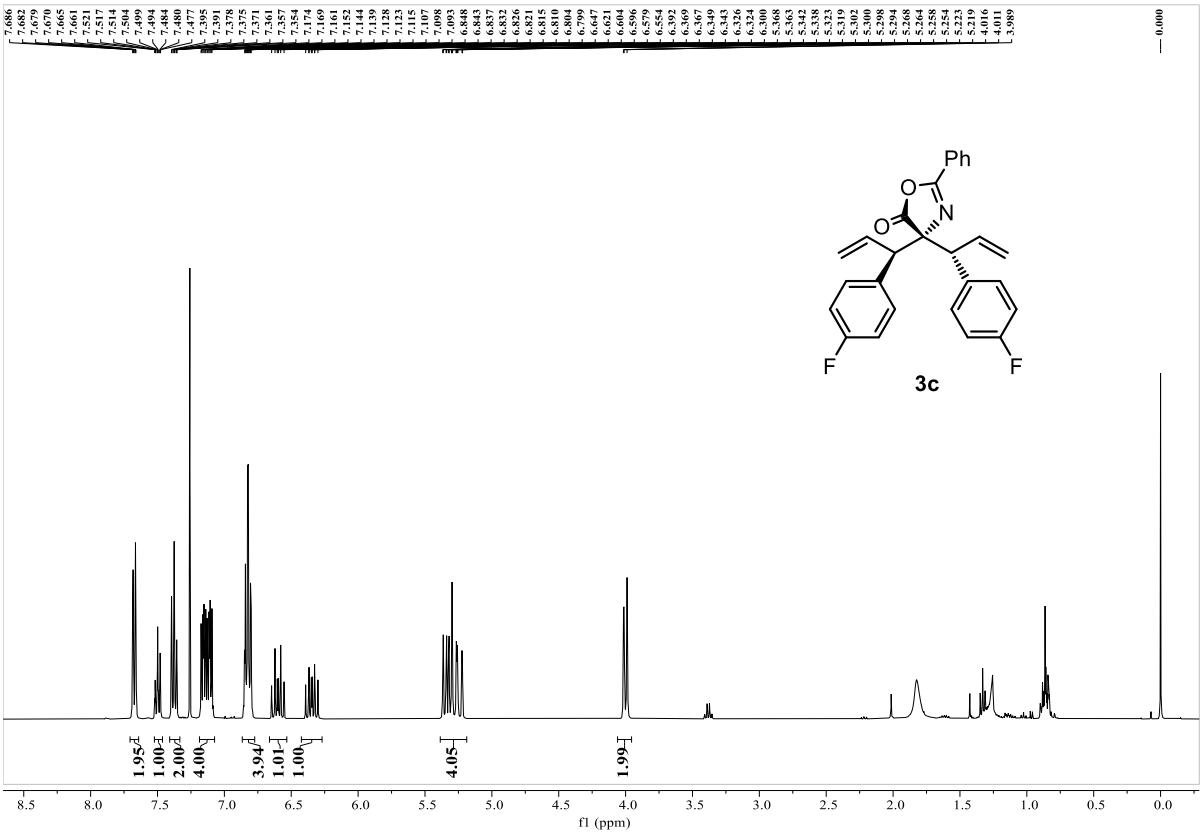
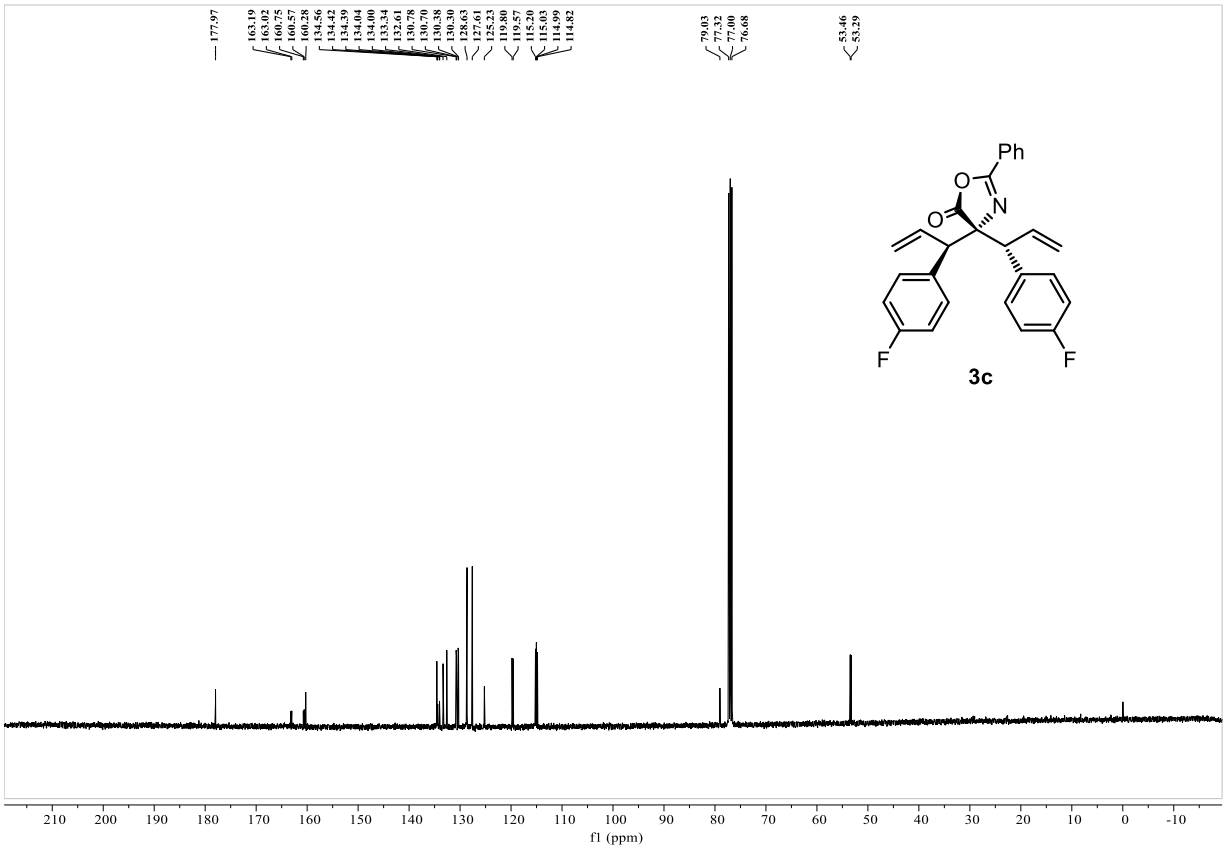
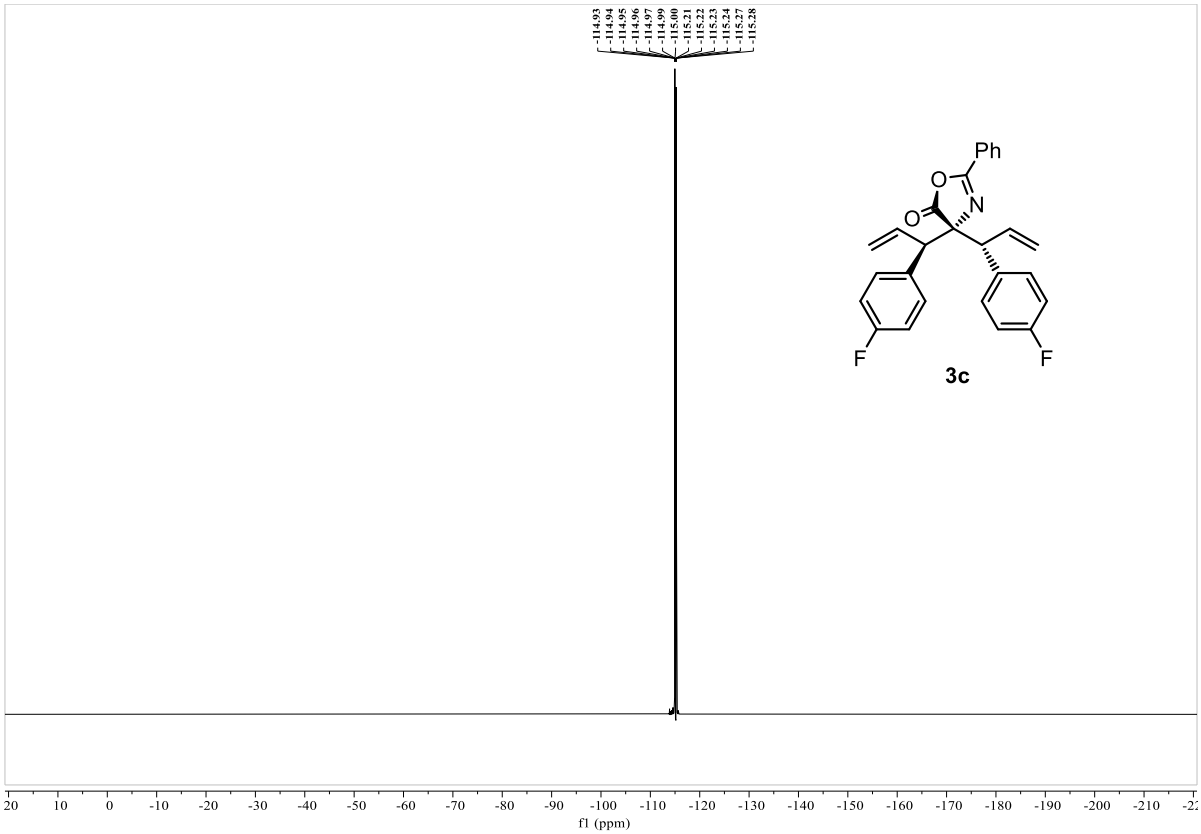


^{1}H NMR (400 MHz, $CDCl_3$) of **3c**



^{13}C NMR (101 MHz, $CDCl_3$) of **3c**



^{19}F NMR (376 MHz, $CDCl_3$) of **3c**

HPLC chromatogram of compound (*rac*)-3c

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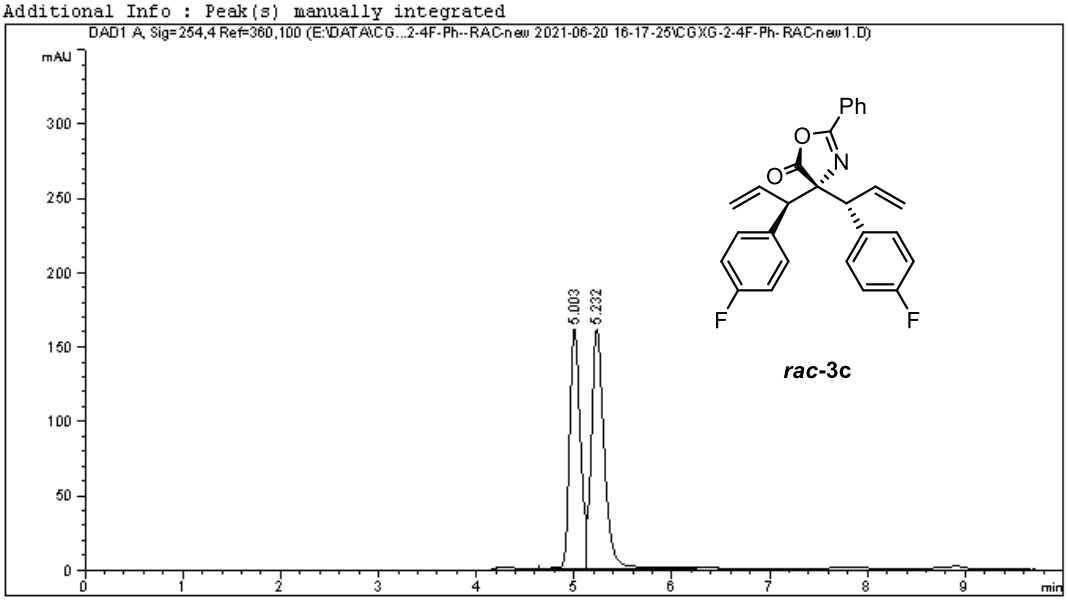
                  (modified after loading)
Analysis Method : E:\DATA\CGXG-2-Xibingjihua\CGXG-2-4F-Ph--RAC-new 2021-06-20 16-17-25\CGXG-2
                  -IE-99-1-DAD-0.5ML-20MIN-2UL.M (Sequence Method)
Last changed    : 12/30/2021 9:42:09 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```



rac-3c

```
==================================================================
                        Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=254,4 Ref=360,100

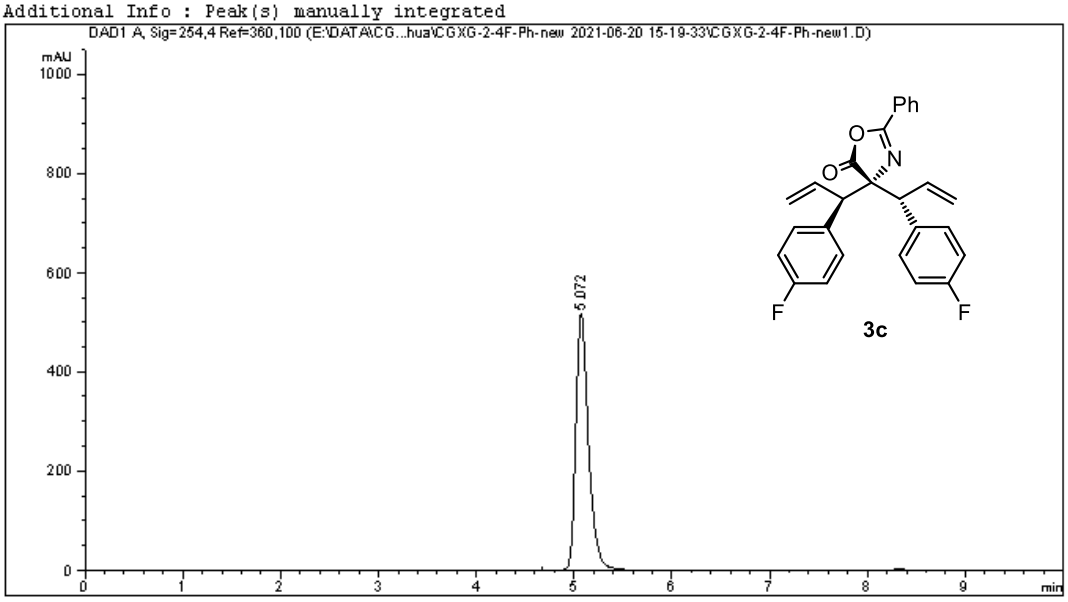
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   5.003 BV    0.1166 1227.07581  161.56740  45.8142
   2   5.232 VB    0.1350 1451.29846  161.26436  54.1858

Totals :                  2678.37427  322.83176
```

HPLC chromatogram of compound 3c

Data File E:\DATA\CG...ibingjihua\CGXG-2-4F-Ph-new 2021-06-20 15-19-33\CGXG-2-4F-Ph-new1.D
Sample Name: CGXG-2-4F-Ph-new-R

```
==================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   2
Acq. Instrument : 1260                           Location :   72
Injection Date  : 6/20/2021 3:56:05 PM               Inj :   1
                                                Inj Volume : 2.000 µl
Acq. Method     : E:\DATA\CGXG-2-Xibingjihua\CGXG-2-4F-Ph-new 2021-06-20 15-19-33\CGXG-2-IE-
                  99-1-DAD-0.5ML-20MIN-2UL.M
Last changed    : 6/20/2021 4:14:57 PM by SYSTEM
                  (modified after loading)
Analysis Method : E:\DATA\CGXG-2-Xibingjihua\CGXG-2-4F-Ph-new 2021-06-20 15-19-33\CGXG-2-IE-
                  99-1-DAD-0.5ML-20MIN-2UL.M (Sequence Method)
Last changed    : 12/30/2021 9:44:02 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```



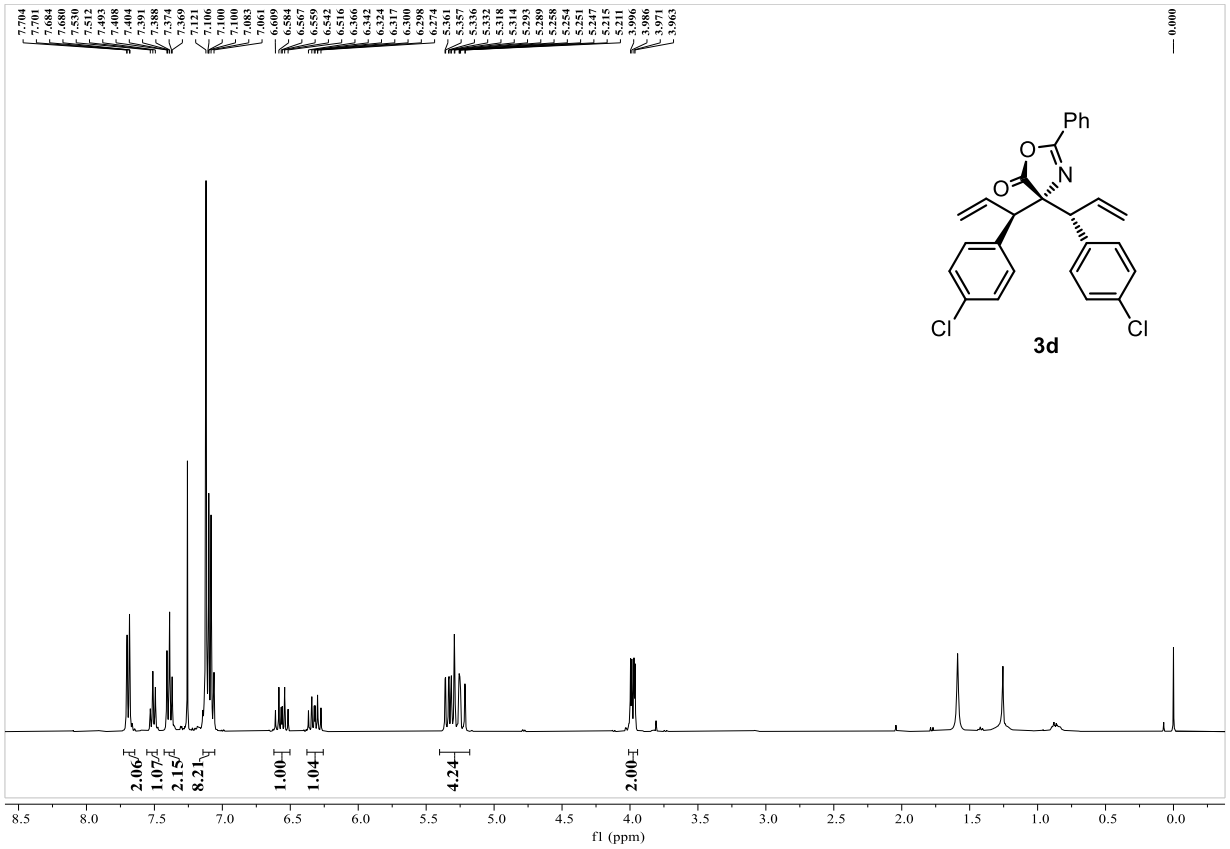

```
==================================================================
                        Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs

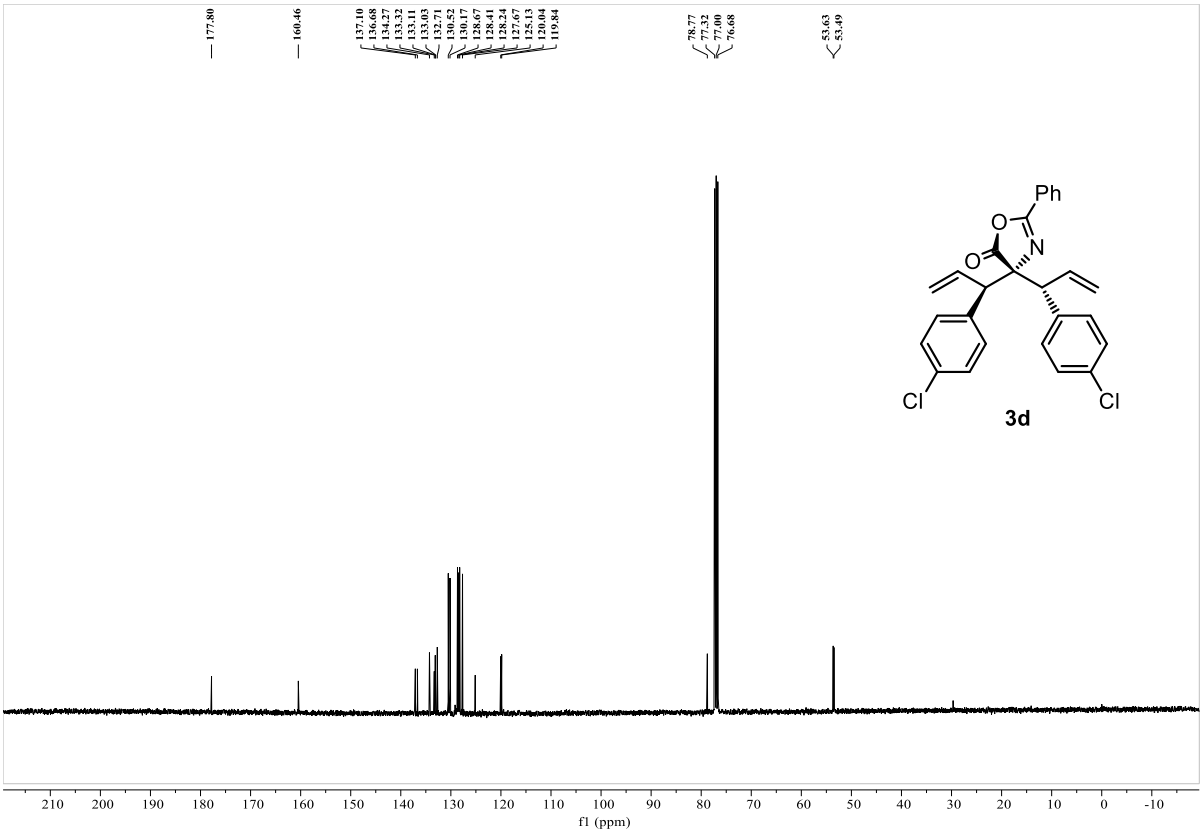

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]        %
----|-------|----|-------|----------|----------|--------|
   1   5.072 BB    0.1382 4705.96045  517.21686 100.0000

Totals :                  4705.96045  517.21686
```



^{1}H NMR (400 MHz, $CDCl_3$) of **3d**

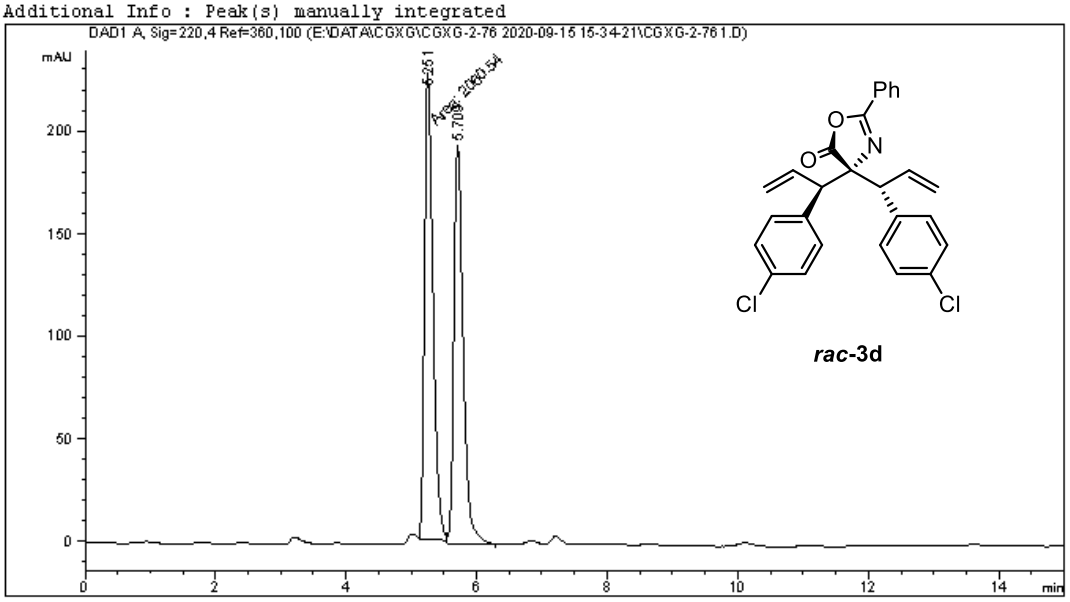


^{13}C NMR (101 MHz, $CDCl_3$) of **3d**

HPLC chromatogram of compound (*rac*)-3d

Data File E:\DATA\CGXG\CGXG-2-76 2020-09-15 15-34-21\CGXG-2-761.D
Sample Name: CGXG-2-76-RAC

```
======================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   2
Acq. Instrument : 1260                           Location :   57
Injection Date  : 9/15/2020 3:47:11 PM               Inj :   1
                                                Inj Volume : 2.000 µl
Acq. Method     : E:\DATA\CGXG\CGXG-2-76 2020-09-15 15-34-21\SC-2-ADH-98-2-DAD-1ML-30MIN-2UL.
                  M
Last changed    : 9/15/2020 3:34:21 PM by SYSTEM
Analysis Method : E:\DATA\CGXG\CGXG-2-76 2020-09-15 15-34-21\SC-2-ADH-98-2-DAD-1ML-30MIN-2UL.
                  M (Sequence Method)
Last changed    : 6/21/2021 12:54:05 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```



***rac*-3d**

```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=220,4 Ref=360,100

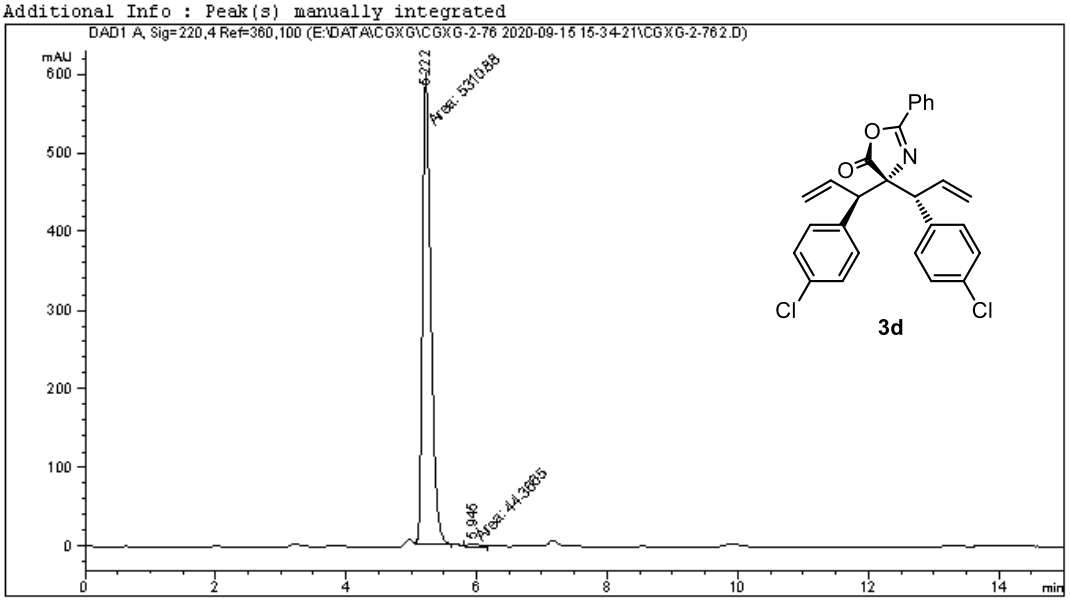
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   5.251 MM    0.1513 2060.54370  227.04514  51.7112
   2   5.709 VB    0.1495 1924.16699  194.32568  48.2888

Totals :                   3984.71069  421.37082
```

HPLC chromatogram of compound 3d

Data File E:\DATA\CGXG\CGXG-2-76 2020-09-15 15-34-21\CGXG-2-762.D
Sample Name: CGXG-2-76-R

```
==========================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   3
Acq. Instrument : 1260                           Location :   58
Injection Date  : 9/15/2020 4:18:44 PM               Inj :   1
                                                Inj Volume : 2.000 µl
Acq. Method     : E:\DATA\CGXG\CGXG-2-76 2020-09-15 15-34-21\SC-2-ADH-98-2-DAD-1ML-30MIN-2UL.
                  M
Last changed    : 9/15/2020 3:34:21 PM by SYSTEM
Analysis Method : E:\DATA\CGXG\CGXG-2-76 2020-09-15 15-34-21\SC-2-ADH-98-2-DAD-1ML-30MIN-2UL.
                  M (Sequence Method)
Last changed    : 6/21/2021 6:20:50 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```



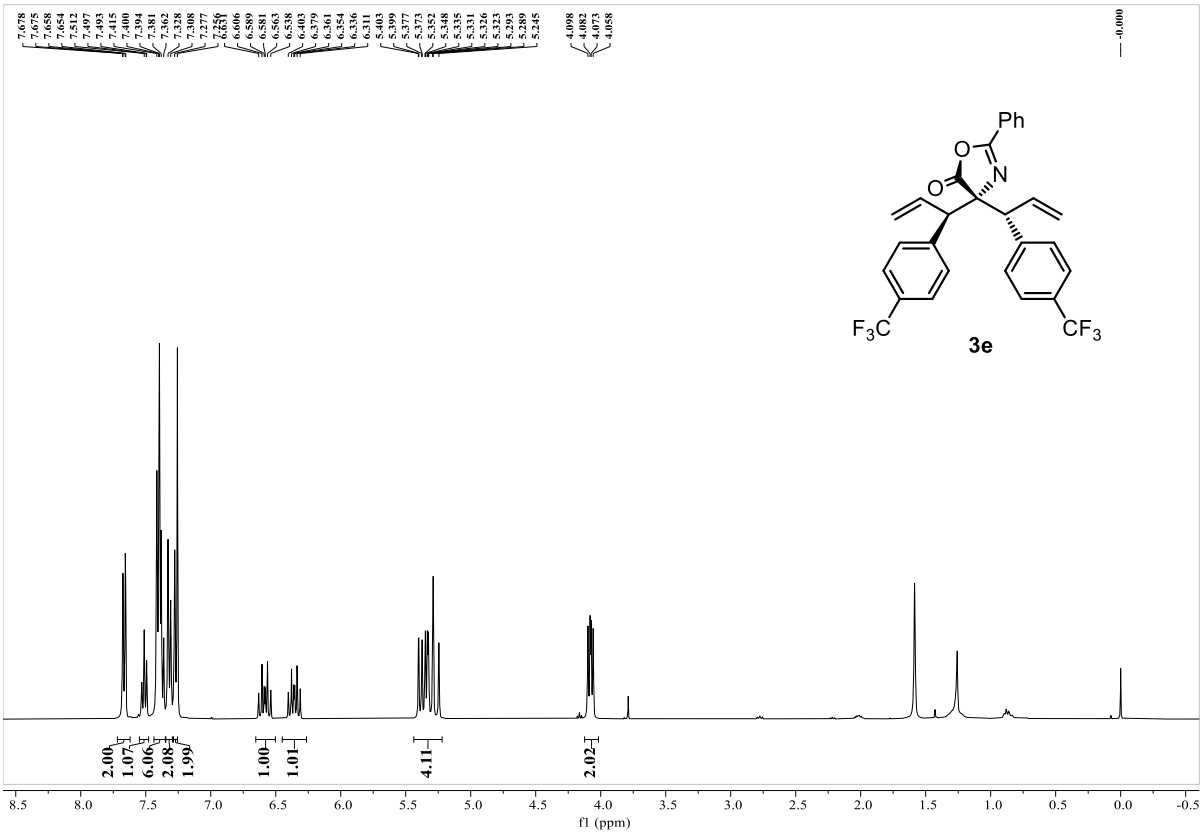

```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs

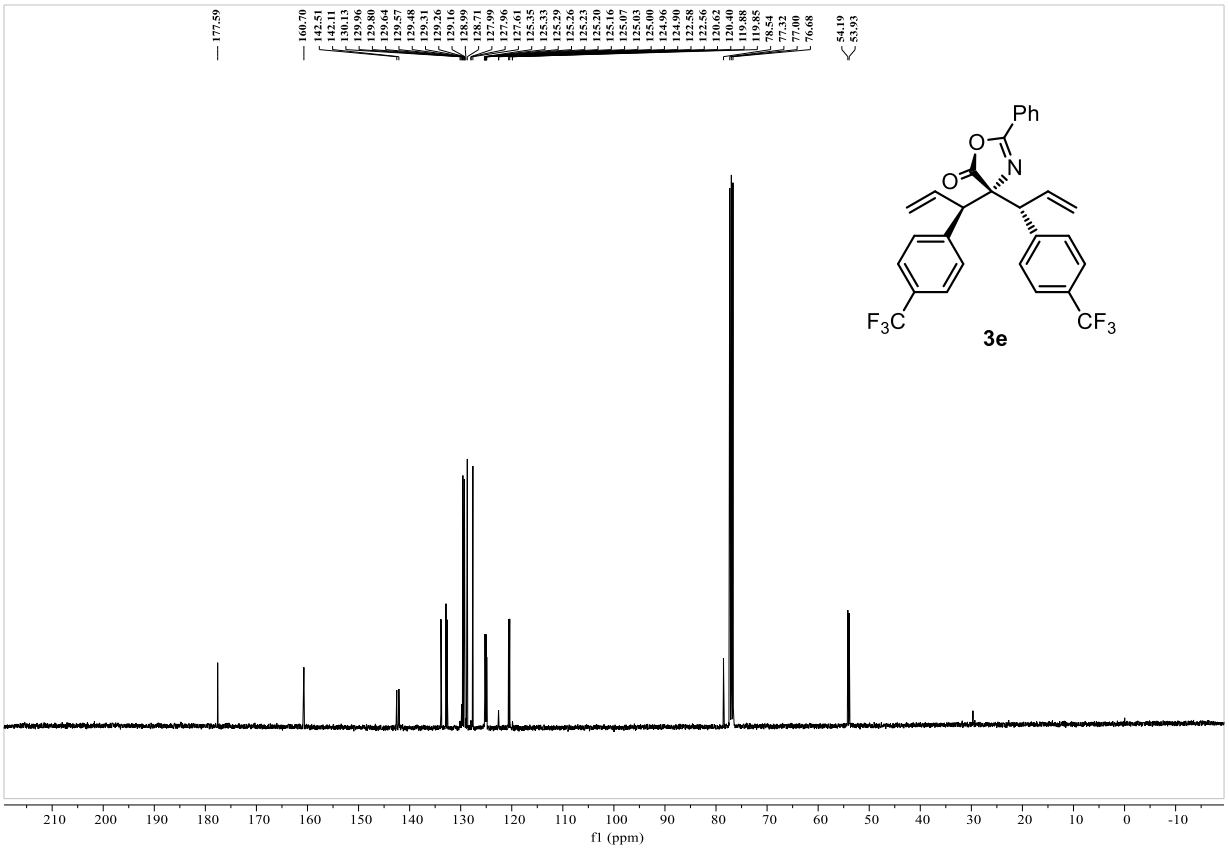

Signal 1: DAD1 A, Sig=220,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   5.222 MM    0.1480 5310.87744  597.91437  99.1715
   2   5.945 MM    0.1917   44.36651    3.85719   0.8285

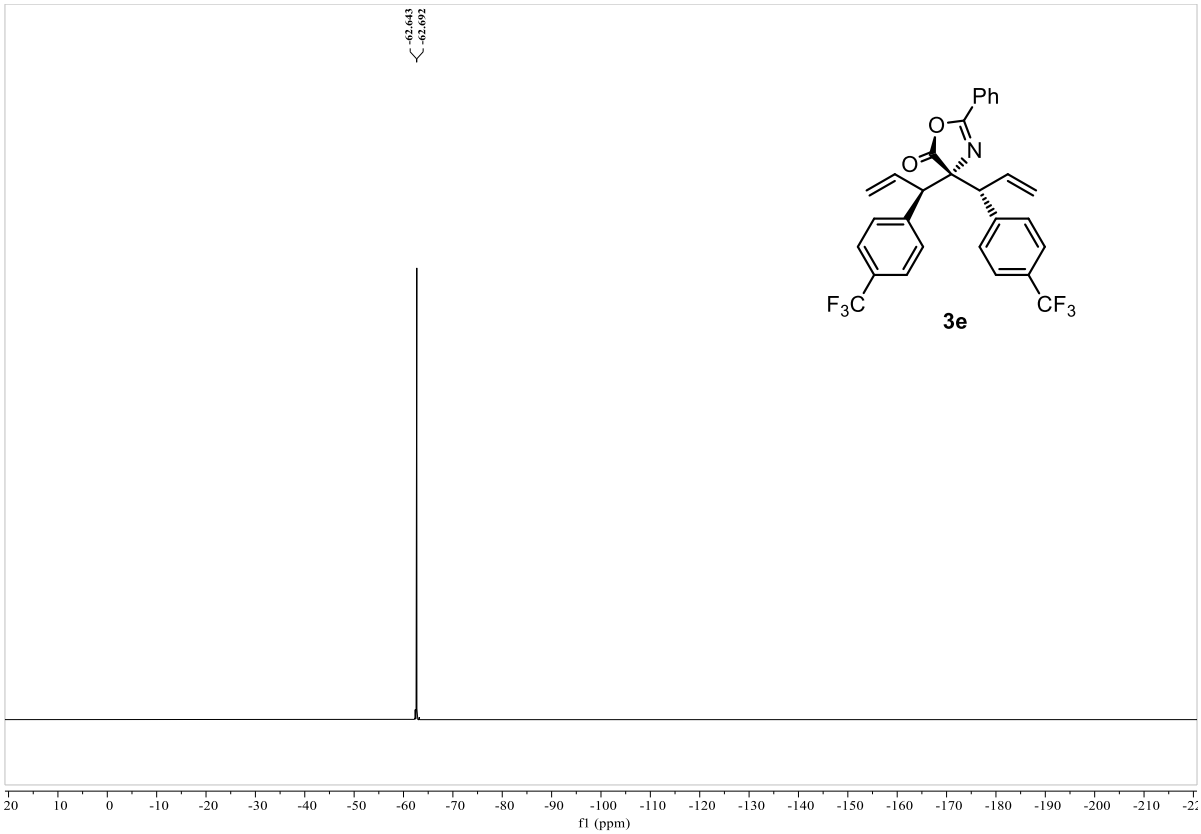
Totals :                  5355.24395  601.77156
```



^{1}H NMR (400 MHz, $CDCl_3$) of **3e**



^{13}C NMR (101 MHz, $CDCl_3$) of **3e**

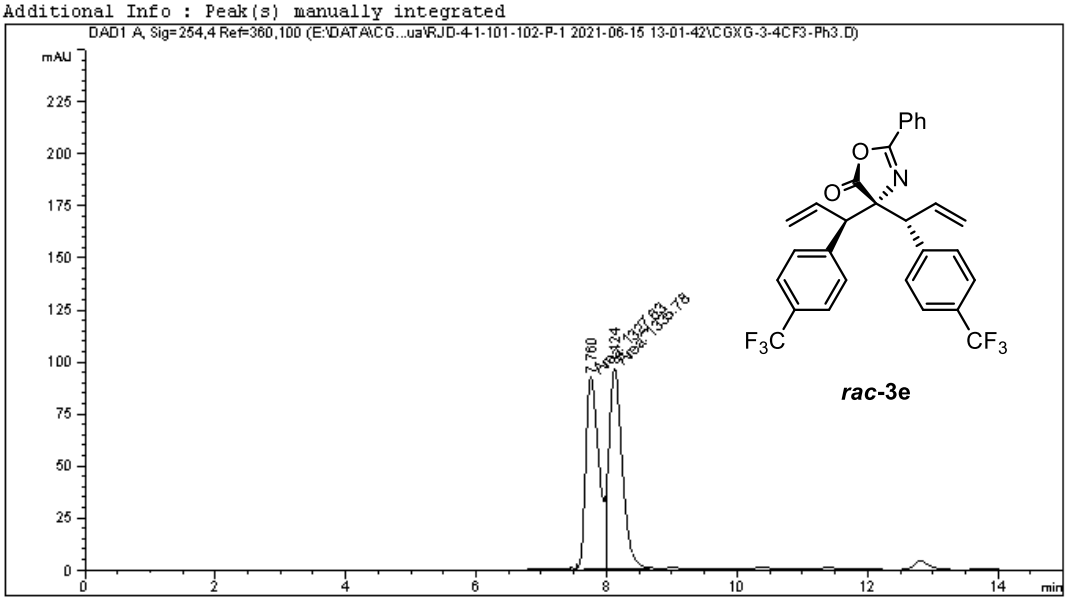


^{19}F NMR (376 MHz, $CDCl_3$) of **3e**

HPLC chromatogram of compound (*rac*)-3e

Data File E:\DATA\CG...bingjihua\RJD-4-1-101-102-P-1 2021-06-15 13-01-42\CGXG-3-4CF3-Ph3.D
Sample Name: CGXG-2-4CF3-Ph-RAC

```
==========================================================================
Acq. Operator   : SYSTEM                          Seq. Line :   4
Acq. Instrument : 1260                             Location :   71
Injection Date  : 6/15/2021 2:06:34 PM                 Inj :   1
                                                 Inj Volume : 2.000 µl
Acq. Method     : E:\DATA\CGXG-2-Xibingjihua\RJD-4-1-101-102-P-1 2021-06-15 13-01-42\CGXG-2-
                  IE-99-1-DAD-0.5ML-20MIN-2UL.M
Last changed    : 6/15/2021 2:27:45 PM by SYSTEM
                  (modified after loading)
Analysis Method : E:\DATA\CGXG-2-Xibingjihua\RJD-4-1-101-102-P-1 2021-06-15 13-01-42\CGXG-2-
                  IE-99-1-DAD-0.5ML-20MIN-2UL.M (Sequence Method)
Last changed    : 12/30/2021 9:52:41 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=254,4 Ref=360,100

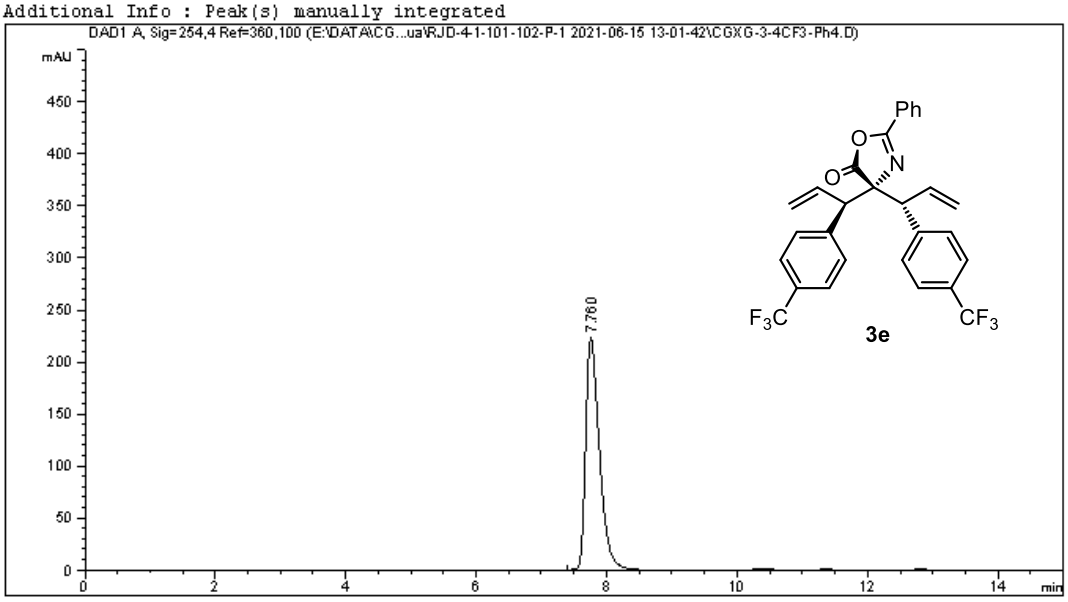
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]        %
----|-------|----|-------|----------|----------|--------|
   1   7.760 MF    0.2395 1327.63416   92.40102  49.8472
   2   8.124 FM    0.2310 1335.77539   96.36755  50.1528

Totals :                  2663.40955  188.76858
```

HPLC chromatogram of compound 3e

Data File E:\DATA\CG...bingjihua\RJD-4-1-101-102-P-1 2021-06-15 13-01-42\CGXG-3-4CF3-Ph4.D
Sample Name: CGCG-2-4CF3-Ph-RRR

```
==========================================================================
Acq. Operator   : SYSTEM                          Seq. Line :   5
Acq. Instrument : 1260                              Location :   72
Injection Date  : 6/15/2021 2:29:27 PM                  Inj :   1
                                                 Inj Volume : 2.000 µl
Acq. Method     : E:\DATA\CGXG-2-Xibingjihua\RJD-4-1-101-102-P-1 2021-06-15 13-01-42\CGXG-2-
                  IE-99-1-DAD-0.5ML-20MIN-2UL.M
Last changed    : 6/15/2021 2:32:09 PM by SYSTEM
                  (modified after loading)
Analysis Method : E:\DATA\CGXG-2-Xibingjihua\RJD-4-1-101-102-P-1 2021-06-15 13-01-42\CGXG-2-
                  IE-99-1-DAD-0.5ML-20MIN-2UL.M (Sequence Method)
Last changed    : 12/30/2021 9:51:45 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```



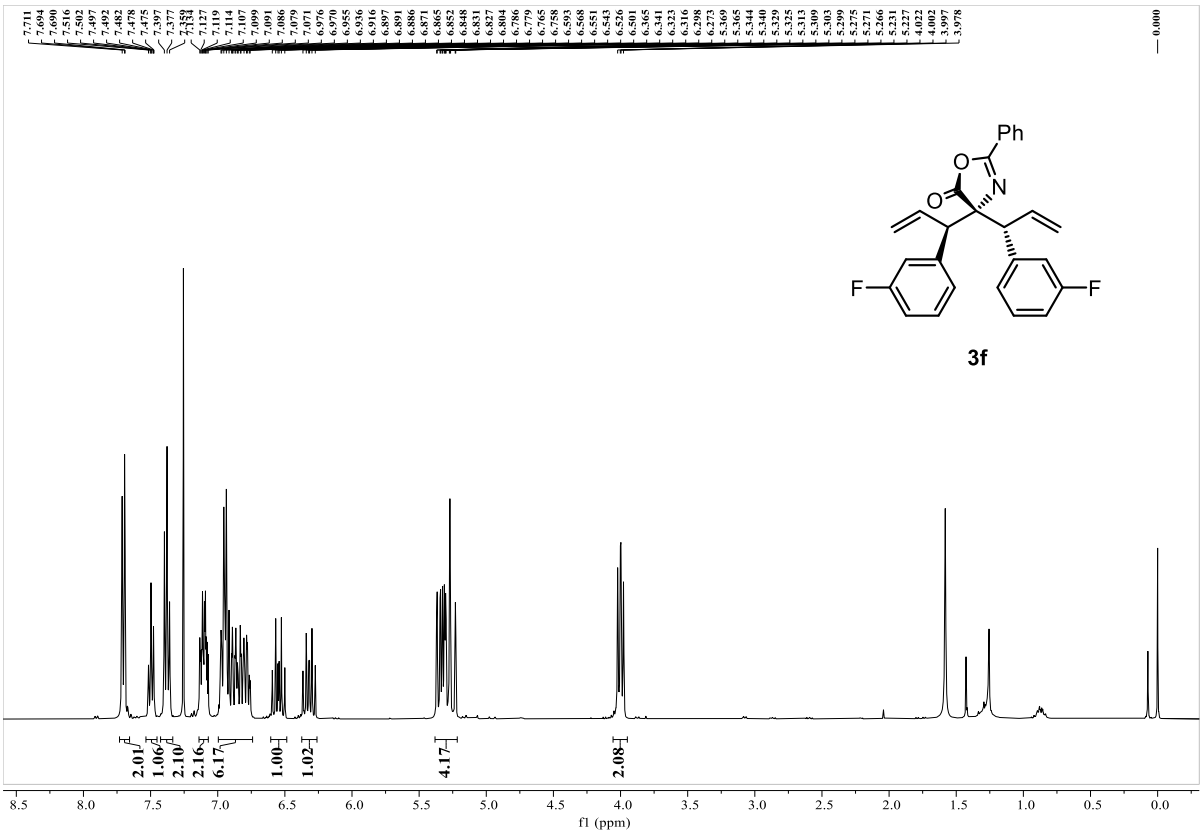

```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs

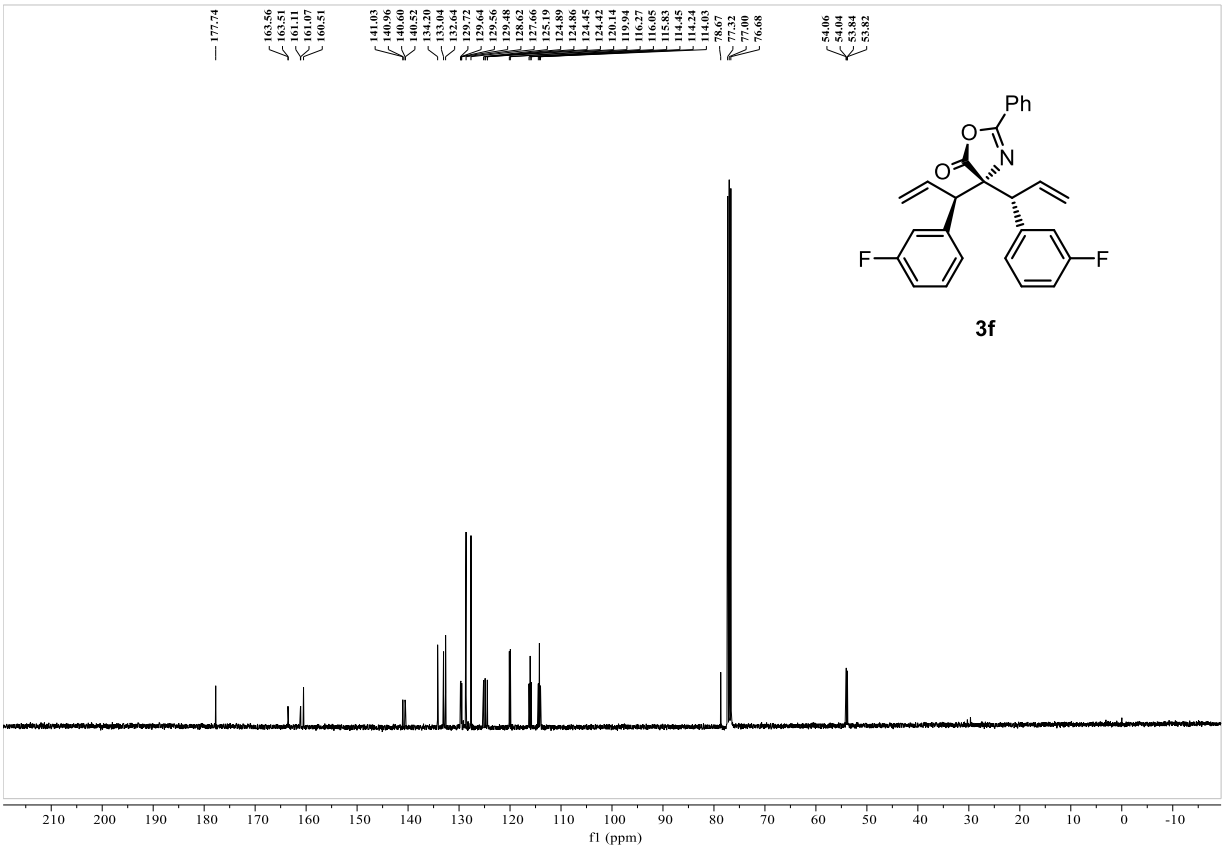

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]        %
----|-------|----|-------|----------|----------|--------|
   1   7.760 VB R  0.2169 3271.63086  223.33981 100.0000

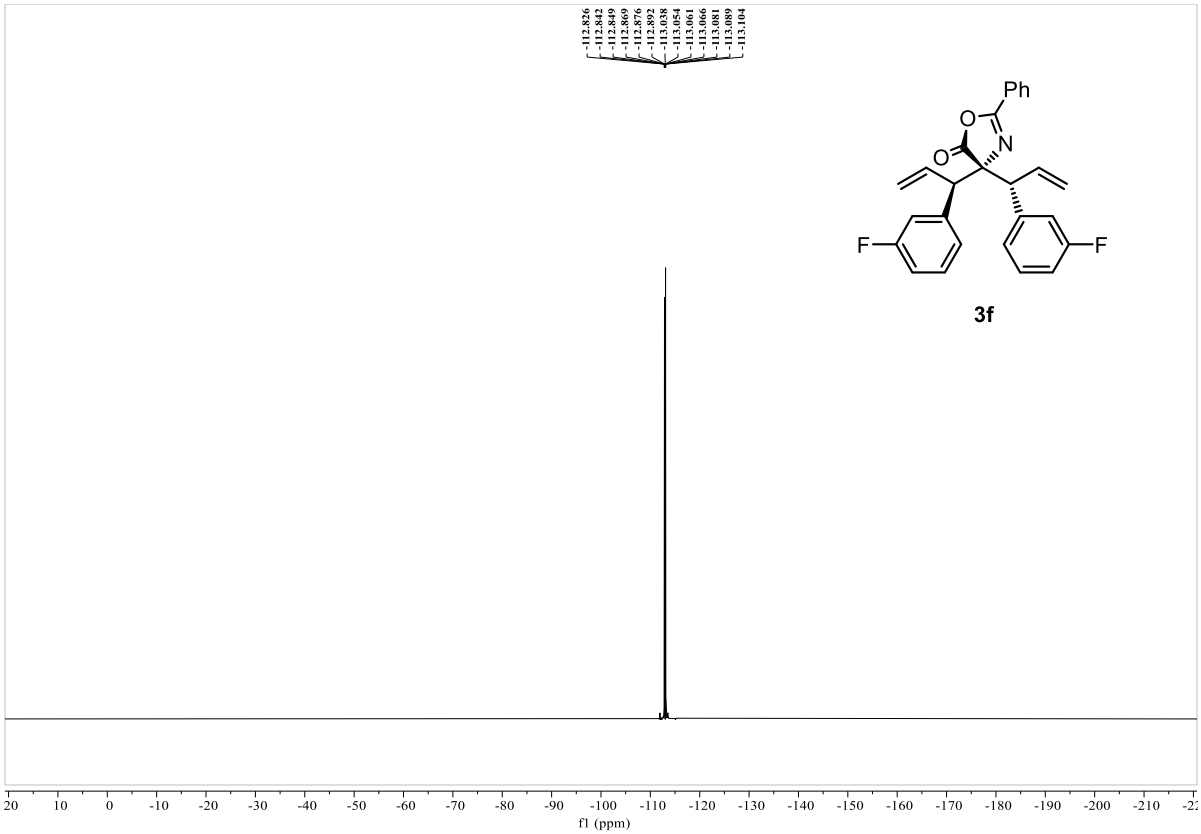
Totals :                  3271.63086  223.33981
```



^{1}H NMR (400 MHz, $CDCl_3$) of **3f**



^{13}C NMR (101 MHz, $CDCl_3$) of **3f**

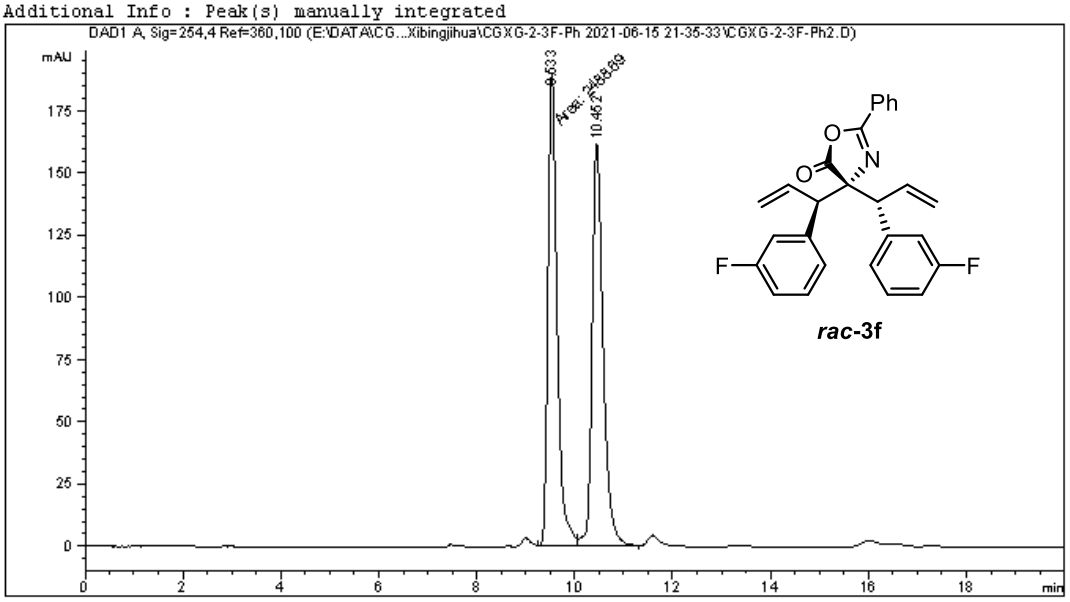


^{19}F NMR (376 MHz, $CDCl_3$) of **3f**

HPLC chromatogram of compound (*rac*)-3f

Data File E:\DATA\CGXG-2-Xibingjihua\CGXG-2-3F-Ph 2021-06-15 21-35-33\CGXG-2-3F-Ph2.D
Sample Name: CGXG-2-3F-Ph-RAC

```
======================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   3
Acq. Instrument : 1260                           Location :   71
Injection Date  : 6/15/2021 10:30:23 PM              Inj :   1
                                                Inj Volume : 2.000 µl
Acq. Method     : E:\DATA\CGXG-2-Xibingjihua\CGXG-2-3F-Ph 2021-06-15 21-35-33\CGXG-2-IE-99-1-
                  DAD-0.5ML-20MIN-2UL.M
Last changed    : 6/15/2021 10:28:25 PM by SYSTEM
Analysis Method : E:\DATA\CGXG-2-Xibingjihua\CGXG-2-3F-Ph 2021-06-15 21-35-33\CGXG-2-IE-99-1-
                  DAD-0.5ML-20MIN-2UL.M (Sequence Method)
Last changed    : 6/16/2021 11:29:05 AM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=254,4 Ref=360,100

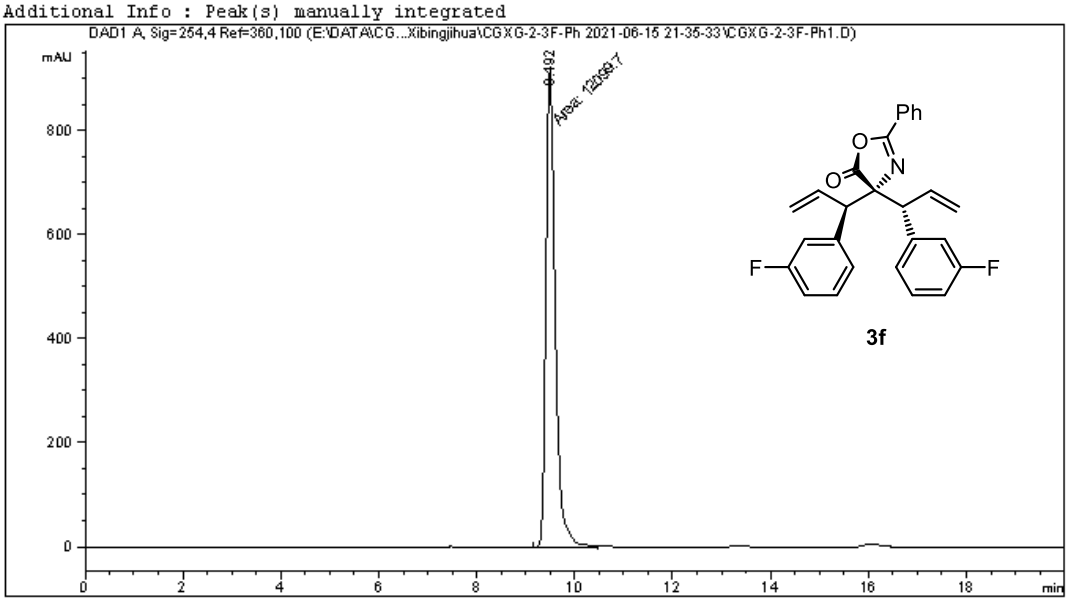
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   9.533 FM    0.2182 2488.69263  190.11781  49.8882
   2  10.452 VB    0.2364 2499.85181  161.64336  50.1118

Totals :                  4988.54443  351.76117
```

HPLC chromatogram of compound 3f

Data File E:\DATA\CGXG-2-Xibingjihua\CGXG-2-3F-Ph 2021-06-15 21-35-33\CGXG-2-3F-Ph1.D
Sample Name: CGXG-2-3F-Ph-RRR

```
======================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   2
Acq. Instrument : 1260                           Location :   72
Injection Date  : 6/15/2021 10:03:05 PM              Inj :   1
                                                Inj Volume : 2.000 µl
Acq. Method     : E:\DATA\CGXG-2-Xibingjihua\CGXG-2-3F-Ph 2021-06-15 21-35-33\CGXG-2-IE-99-1-
                  DAD-0.5ML-20MIN-2UL.M
Last changed    : 6/15/2021 10:28:25 PM by SYSTEM
                  (modified after loading)
Analysis Method : E:\DATA\CGXG-2-Xibingjihua\CGXG-2-3F-Ph 2021-06-15 21-35-33\CGXG-2-IE-99-1-
                  DAD-0.5ML-20MIN-2UL.M (Sequence Method)
Last changed    : 6/16/2021 11:30:15 AM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```



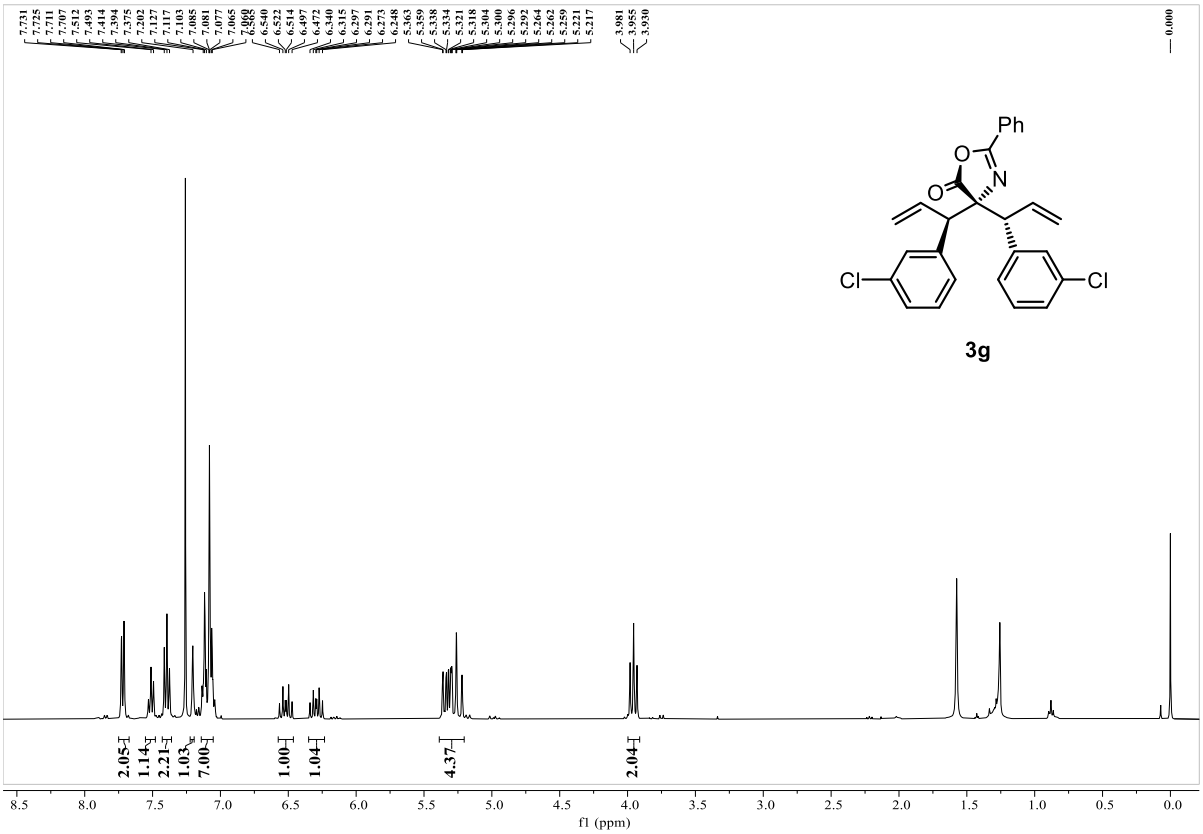

```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs

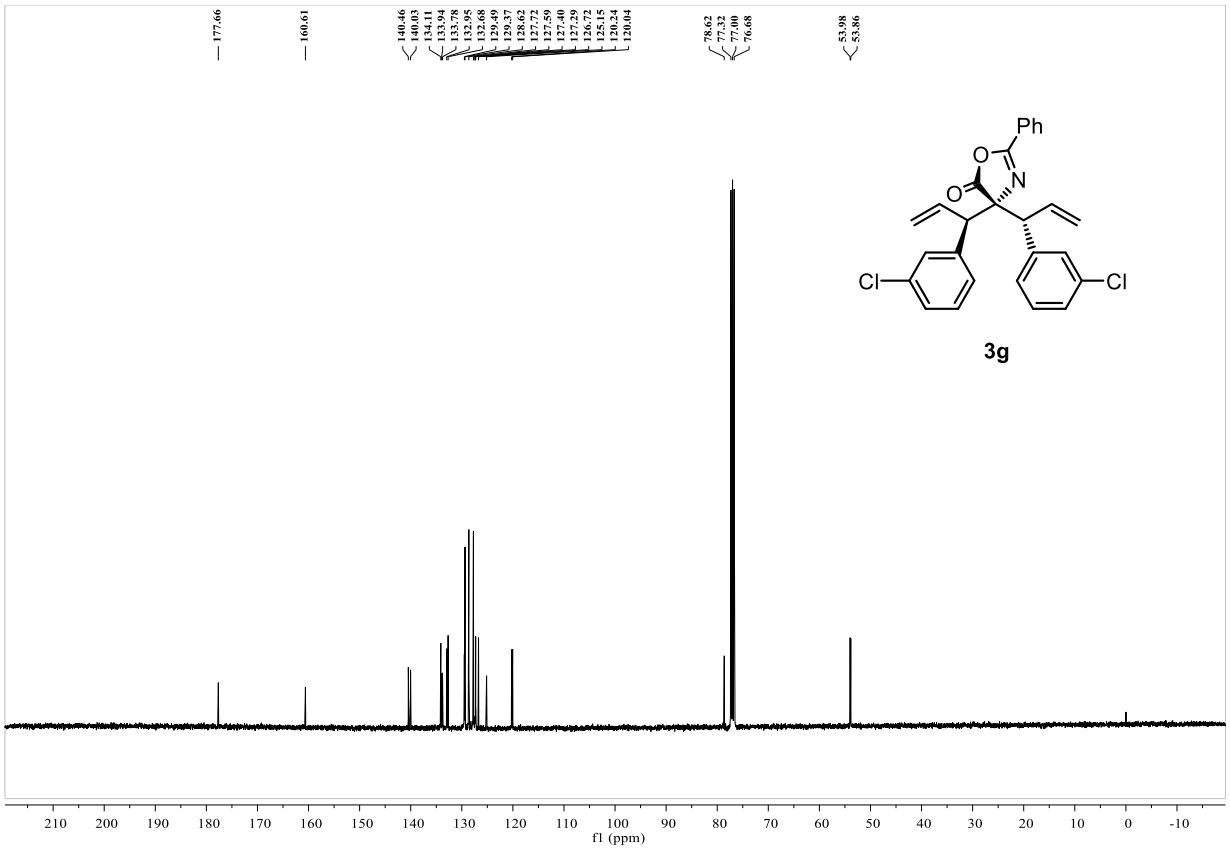

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]        %
----|-------|----|-------|----------|----------|--------|
   1   9.492 MF    0.2216 1.20997e4   910.00244 100.0000

Totals :                  1.20997e4   910.00244
```



^{1}H NMR (400 MHz, $CDCl_3$) of **3g**

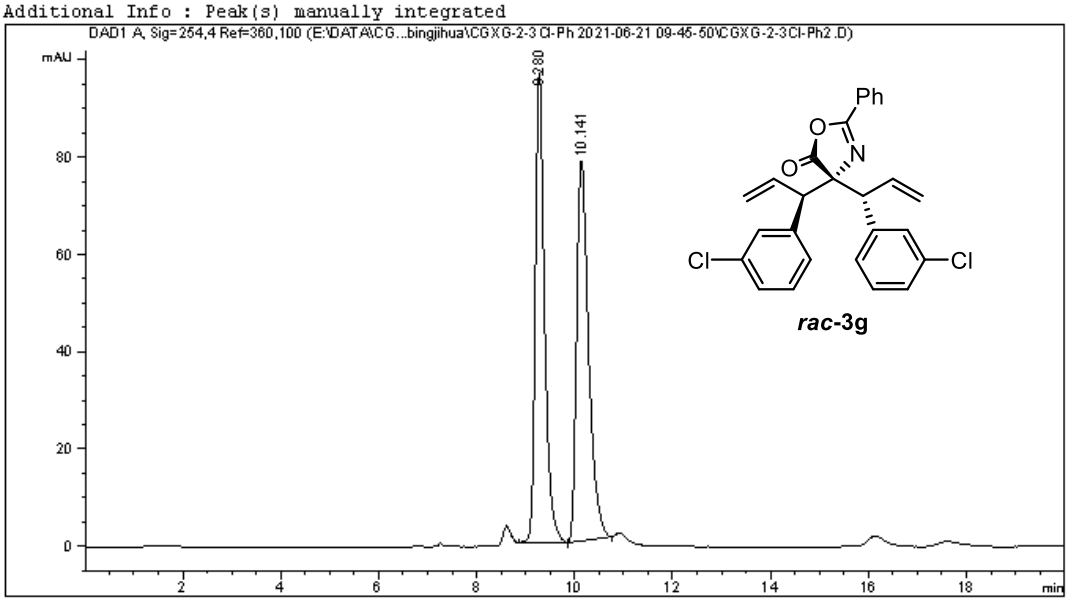


^{13}C NMR (101 MHz, $CDCl_3$) of **3g**

HPLC chromatogram of compound (*rac*)-3g

Data File E:\DATA\CGXG-2-Xibingjihua\CGXG-2-3Cl-Ph 2021-06-21 09-45-50\CGXG-2-3Cl-Ph2.D
Sample Name: CGXG-2-3Cl-Ph-Rac

```
======================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   3
Acq. Instrument : 1260                           Location :   71
Injection Date  : 6/21/2021 10:34:14 AM              Inj :   1
                                                Inj Volume : 2.000 µl
Acq. Method     : E:\DATA\CGXG-2-Xibingjihua\CGXG-2-3Cl-Ph 2021-06-21 09-45-50\CGXG-2-IE-99-1
                  -DAD-0.5ML-30MIN-2UL.M
Last changed    : 6/21/2021 10:34:23 AM by SYSTEM
                  (modified after loading)
Analysis Method : E:\DATA\CGXG-2-Xibingjihua\CGXG-2-3Cl-Ph 2021-06-21 09-45-50\CGXG-2-IE-99-1
                  -DAD-0.5ML-30MIN-2UL.M (Sequence Method)
Last changed    : 6/21/2021 1:13:58 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=254,4 Ref=360,100

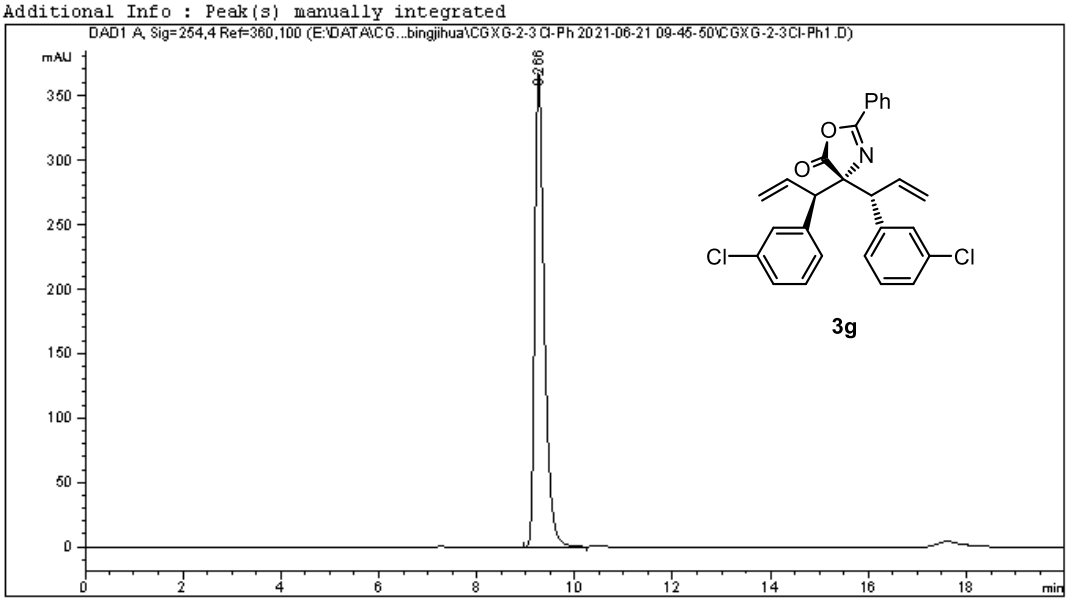
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]        %
----|-------|----|-------|----------|----------|--------|
   1   9.280 BB    0.1956 1270.39697   96.49085  48.9812
   2  10.141 BB    0.2584 1323.24561   78.19842  51.0188

Totals :                  2593.64258  174.68927
```

HPLC chromatogram of compound 3g

Data File E:\DATA\CGXG-2-Xibingjihua\CGXG-2-3Cl-Ph 2021-06-21 09-45-50\CGXG-2-3Cl-Phl.D
Sample Name: CGXG-2-3Cl-Ph-RRR

```
==================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   2
Acq. Instrument : 1260                           Location :   72
Injection Date  : 6/21/2021 10:08:26 AM              Inj :   1
                                                Inj Volume : 2.000 µl
Acq. Method     : E:\DATA\CGXG-2-Xibingjihua\CGXG-2-3Cl-Ph 2021-06-21 09-45-50\CGXG-2-IE-99-1
                  -DAD-0.5ML-30MIN-2UL.M
Last changed    : 6/21/2021 10:32:37 AM by SYSTEM
                  (modified after loading)
Analysis Method : E:\DATA\CGXG-2-Xibingjihua\CGXG-2-3Cl-Ph 2021-06-21 09-45-50\CGXG-2-IE-99-1
                  -DAD-0.5ML-30MIN-2UL.M (Sequence Method)
Last changed    : 6/21/2021 1:14:29 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```



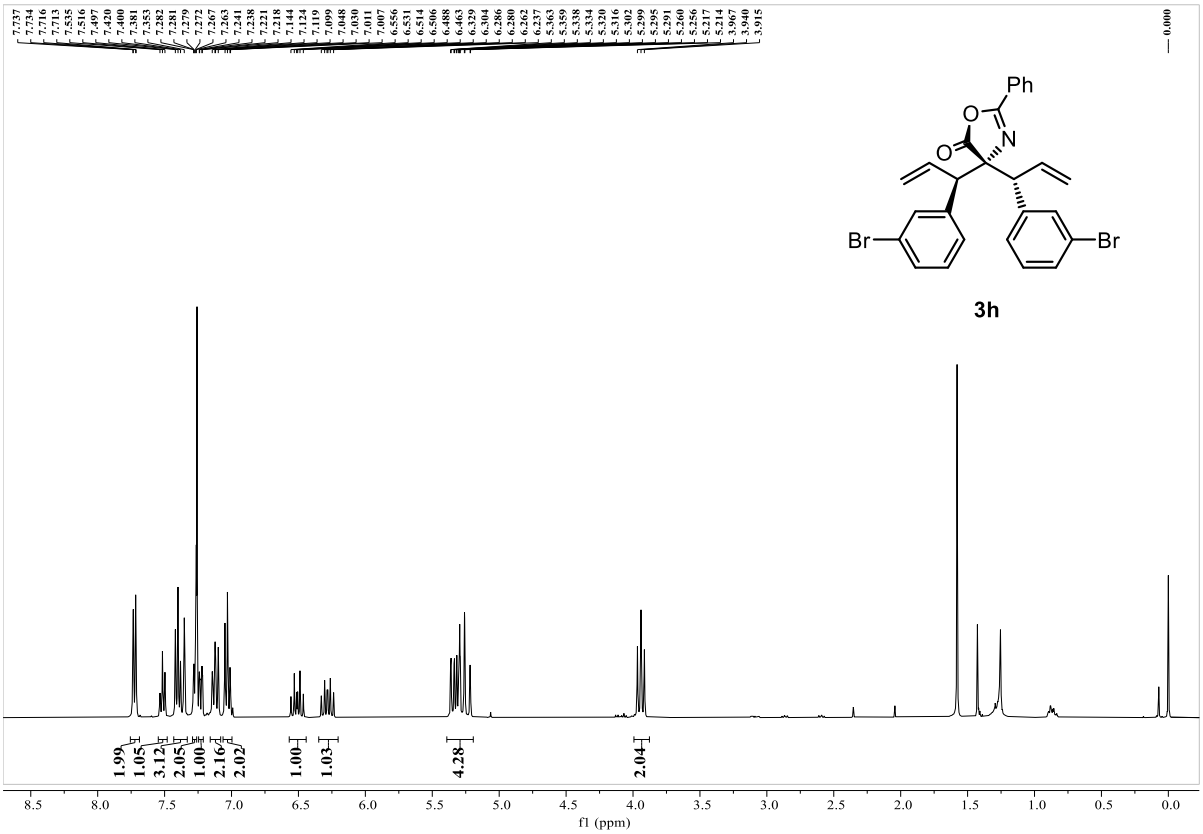

```
==================================================================
                        Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs

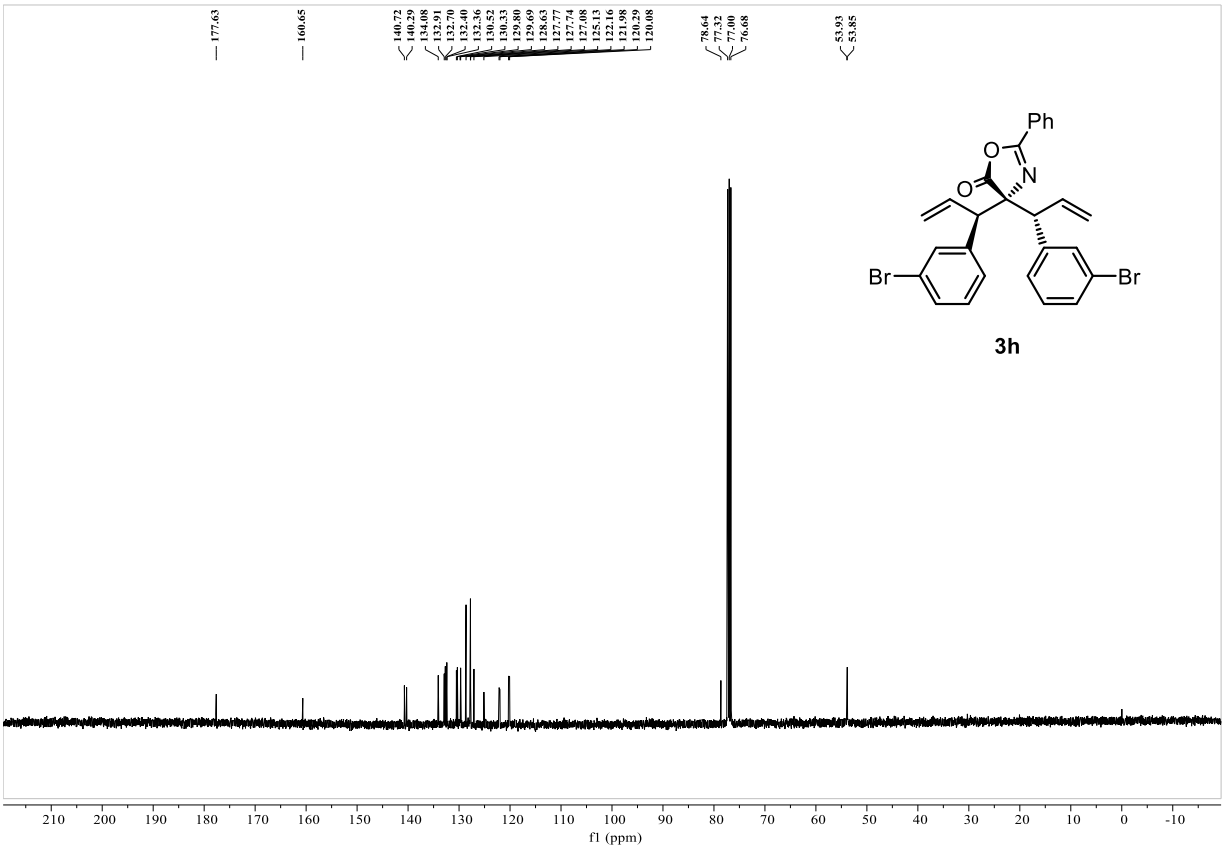

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]        %
----|-------|----|-------|----------|----------|--------|
   1   9.266 BB    0.2043 4943.42578  366.69754 100.0000

Totals :                  4943.42578  366.69754
```



^{1}H NMR (400 MHz, $CDCl_3$) of **3h**

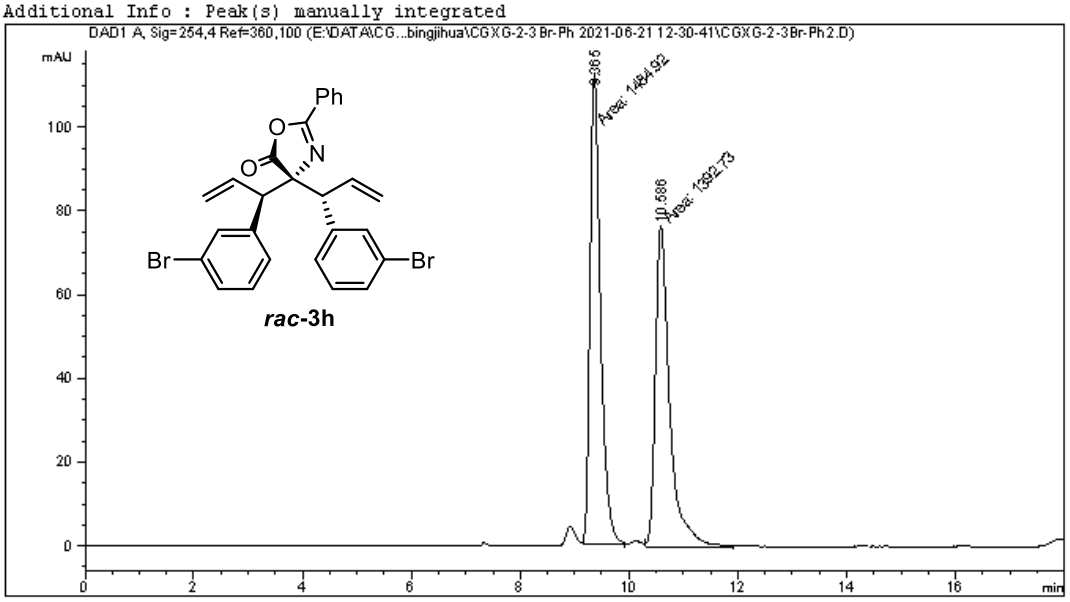


^{13}C NMR (101 MHz, $CDCl_3$) of **3h**

HPLC chromatogram of compound (*rac*)-3h

Data File E:\DATA\CGXG-2-Xibingjihua\CGXG-2-3Br-Ph 2021-06-21 12-30-41\CGXG-2-3Br-Ph2.D
Sample Name: CGXG-2-3Br-Ph-RAC

```
======================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   3
Acq. Instrument : 1260                           Location :   71
Injection Date  : 6/21/2021 1:25:32 PM               Inj :   1
                                                Inj Volume : 2.000 µl
Acq. Method     : E:\DATA\CGXG-2-Xibingjihua\CGXG-2-3Br-Ph 2021-06-21 12-30-41\CGXG-2-IE-99-1
                  -DAD-0.5ML-30MIN-2UL.M
Last changed    : 6/21/2021 1:23:44 PM by SYSTEM
Analysis Method : E:\DATA\CGXG-2-Xibingjihua\CGXG-2-3Br-Ph 2021-06-21 12-30-41\CGXG-2-IE-99-1
                  -DAD-0.5ML-30MIN-2UL.M (Sequence Method)
Last changed    : 6/21/2021 2:01:02 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=254,4 Ref=360,100

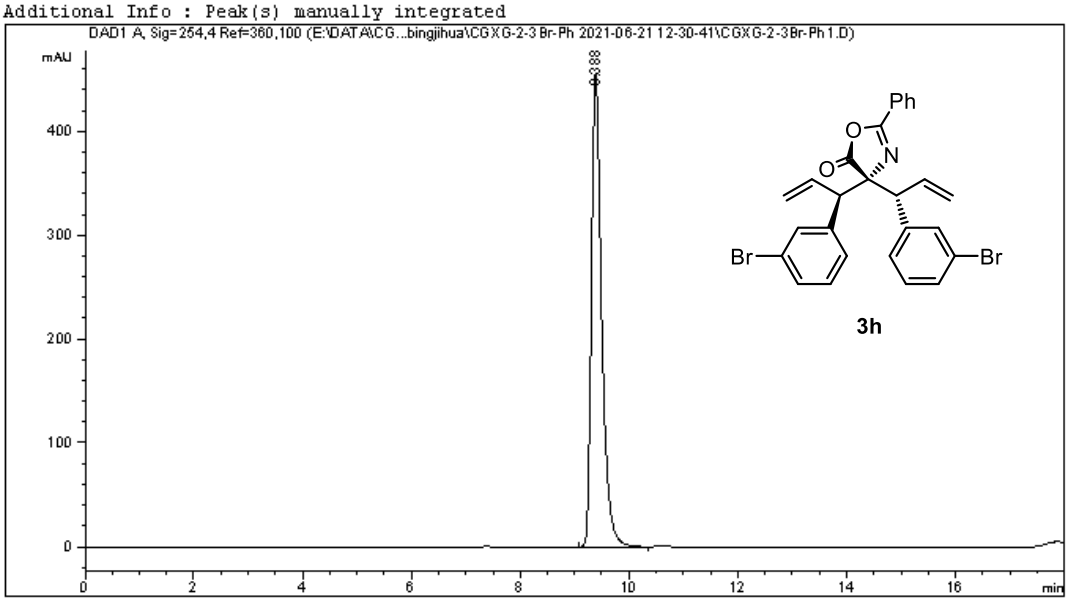
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   9.365 MM    0.2201 1484.92334  112.43555  51.6019
   2  10.586 MM    0.3037 1392.73071   76.43284  48.3981

Totals :                  2877.65405  188.86839
```

HPLC chromatogram of compound 3h

Data File E:\DATA\CGXG-2-Xibingjihua\CGXG-2-3Br-Ph 2021-06-21 12-30-41\CGXG-2-3Br-Ph1.D
Sample Name: CGXG-2-3Br-Ph-RRR

```
==========================================================================
Acq. Operator   : SYSTEM                          Seq. Line :   2
Acq. Instrument : 1260                             Location :   72
Injection Date  : 6/21/2021 12:58:17 PM                 Inj :   1
                                                Inj Volume : 2.000 µl
Acq. Method     : E:\DATA\CGXG-2-Xibingjihua\CGXG-2-3Br-Ph 2021-06-21 12-30-41\CGXG-2-IE-99-1
                  -DAD-0.5ML-30MIN-2UL.M
Last changed    : 6/21/2021 1:23:44 PM by SYSTEM
                  (modified after loading)
Analysis Method : E:\DATA\CGXG-2-Xibingjihua\CGXG-2-3Br-Ph 2021-06-21 12-30-41\CGXG-2-IE-99-1
                  -DAD-0.5ML-30MIN-2UL.M (Sequence Method)
Last changed    : 6/21/2021 1:59:44 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```



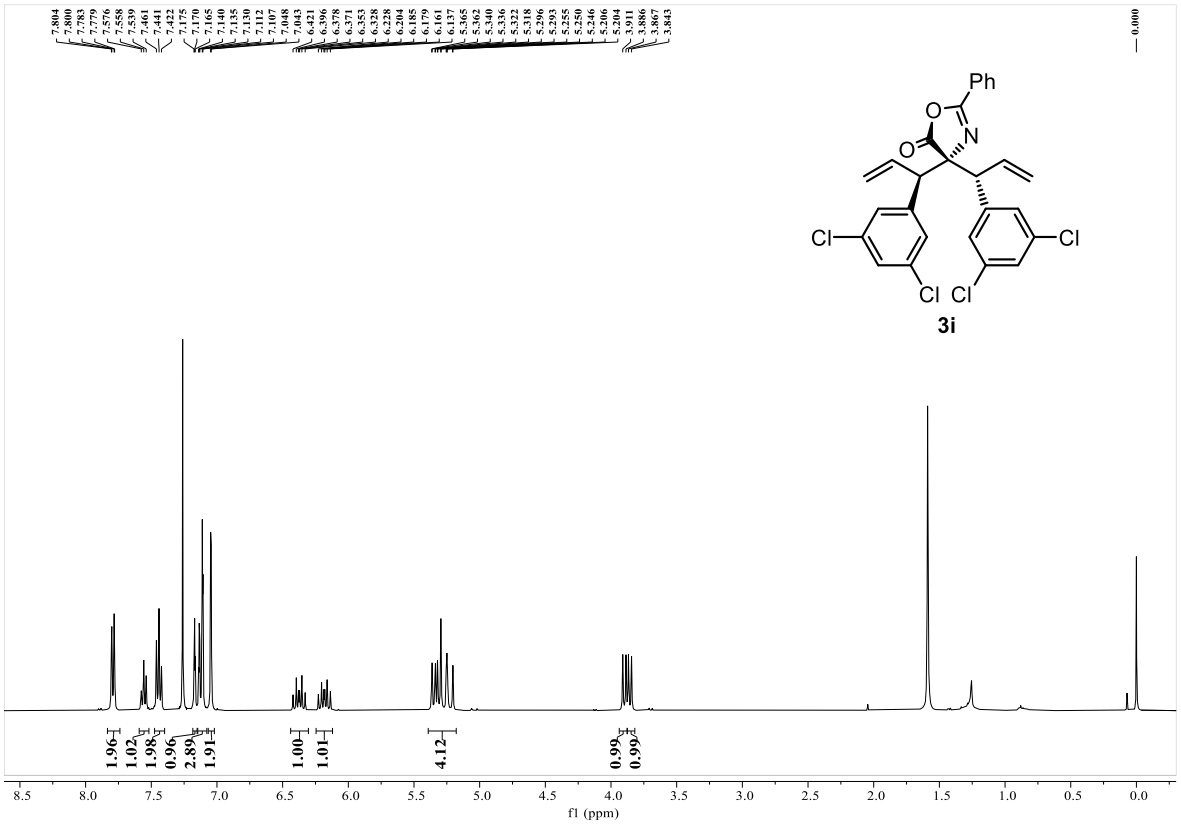

```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs

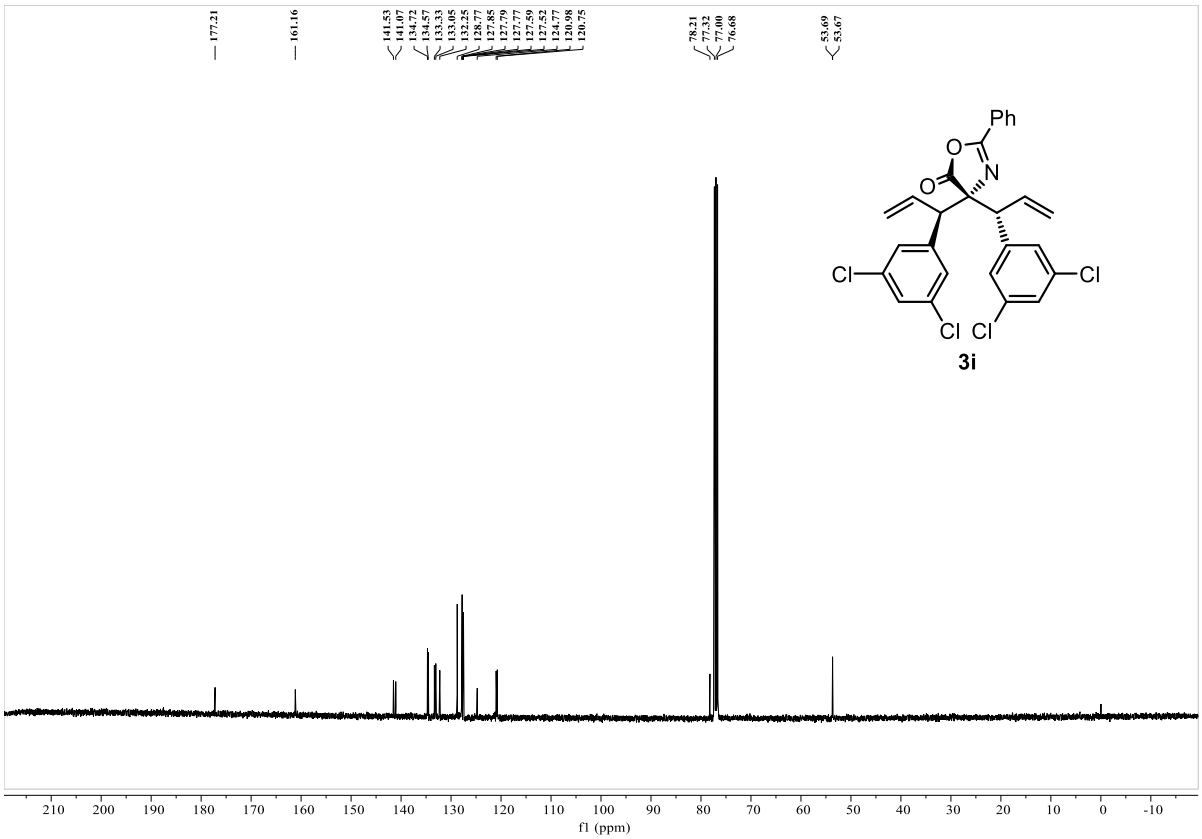

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]        %
----|-------|----|-------|----------|----------|--------|
   1   9.388 BB    0.1948 5929.81250  455.75357 100.0000

Totals :                  5929.81250  455.75357
```



^{1}H NMR (400 MHz, $CDCl_3$) of **3i**

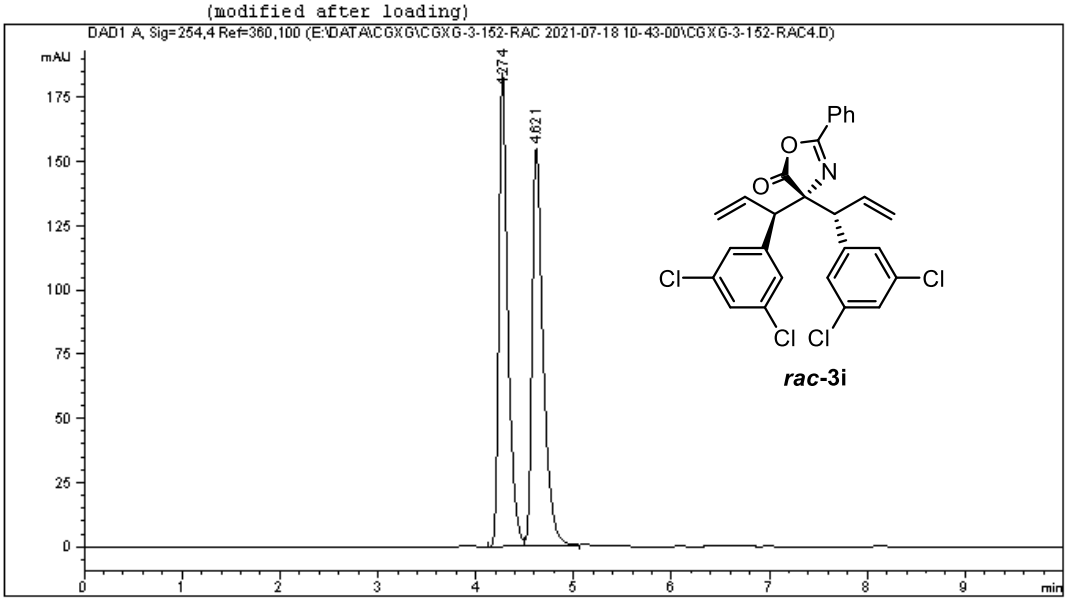


^{13}C NMR (101 MHz, $CDCl_3$) of **3i**

HPLC chromatogram of compound (*rac*)-3i

Data File E:\DATA\CGXG\CGXG-3-152-RAC 2021-07-18 10-43-00\CGXG-3-152-RAC4.D
Sample Name: CGXG-3-152-RAC

```
==========================================================================
Acq. Operator   : SYSTEM                          Seq. Line :   5
Acq. Instrument : 1260                              Location :   51
Injection Date  : 7/18/2021 11:51:05 AM                 Inj :   1
                                                 Inj Volume : 2.000 µl
Acq. Method     : E:\DATA\CGXG\CGXG-3-152-RAC 2021-07-18 10-43-00\CGXG-2-IE-98-2-254DAD-1ML-
                  2UL-30MIN.M
Last changed    : 7/18/2021 12:03:36 PM by SYSTEM
                  (modified after loading)
Analysis Method : E:\DATA\CGXG\CGXG-3-152-RAC 2021-07-18 10-43-00\CGXG-2-IE-98-2-254DAD-1ML-
                  2UL-30MIN.M (Sequence Method)
Last changed    : 7/18/2021 12:04:29 PM by SYSTEM
                  (modified after loading)
```



***rac*-3i**

```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=254,4 Ref=360,100

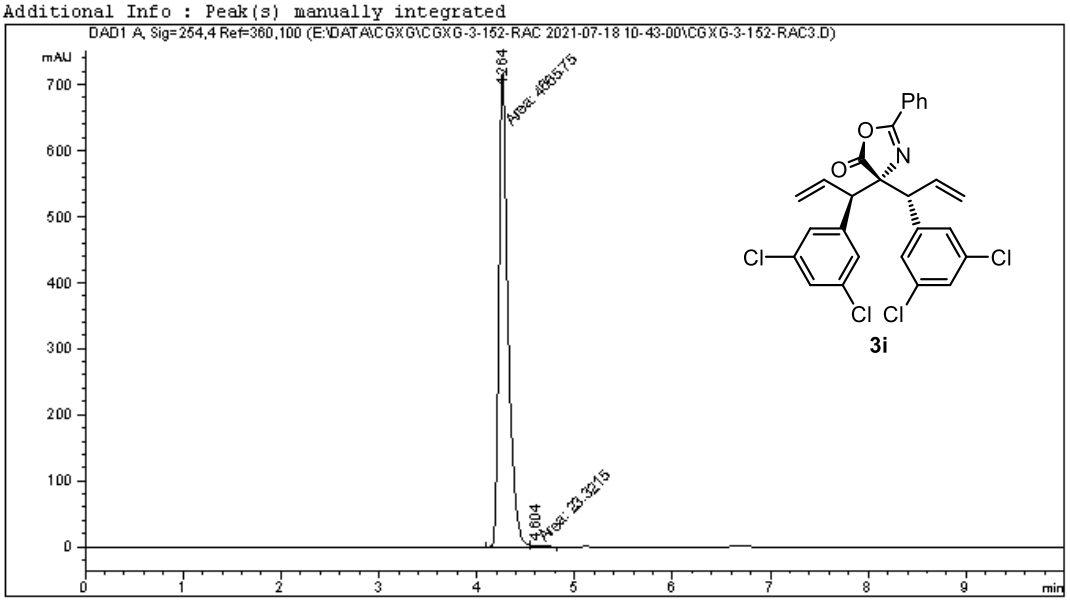
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   4.274 BV    0.0948 1181.81873  184.24092  49.4684
   2   4.621 VB    0.1160 1207.21973  154.72073  50.5316

Totals :                  2389.03845  338.96165
```

HPLC chromatogram of compound 3i

Data File E:\DATA\CGXG\CGXG-3-152-RAC 2021-07-18 10-43-00\CGXG-3-152-RAC3.D
Sample Name: CGXG-3-152-R

```
======================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   4
Acq. Instrument : 1260                           Location :   52
Injection Date  : 7/18/2021 11:31:39 AM              Inj :   1
                                                Inj Volume : 2.000 µl
Acq. Method     : E:\DATA\CGXG\CGXG-3-152-RAC 2021-07-18 10-43-00\CGXG-2-IE-98-2-254DAD-1ML-
                  2UL-30MIN.M
Last changed    : 7/18/2021 11:49:32 AM by SYSTEM
                  (modified after loading)
Analysis Method : E:\DATA\CGXG\CGXG-3-152-RAC 2021-07-18 10-43-00\CGXG-2-IE-98-2-254DAD-1ML-
                  2UL-30MIN.M (Sequence Method)
Last changed    : 7/18/2021 11:52:12 AM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```



3i

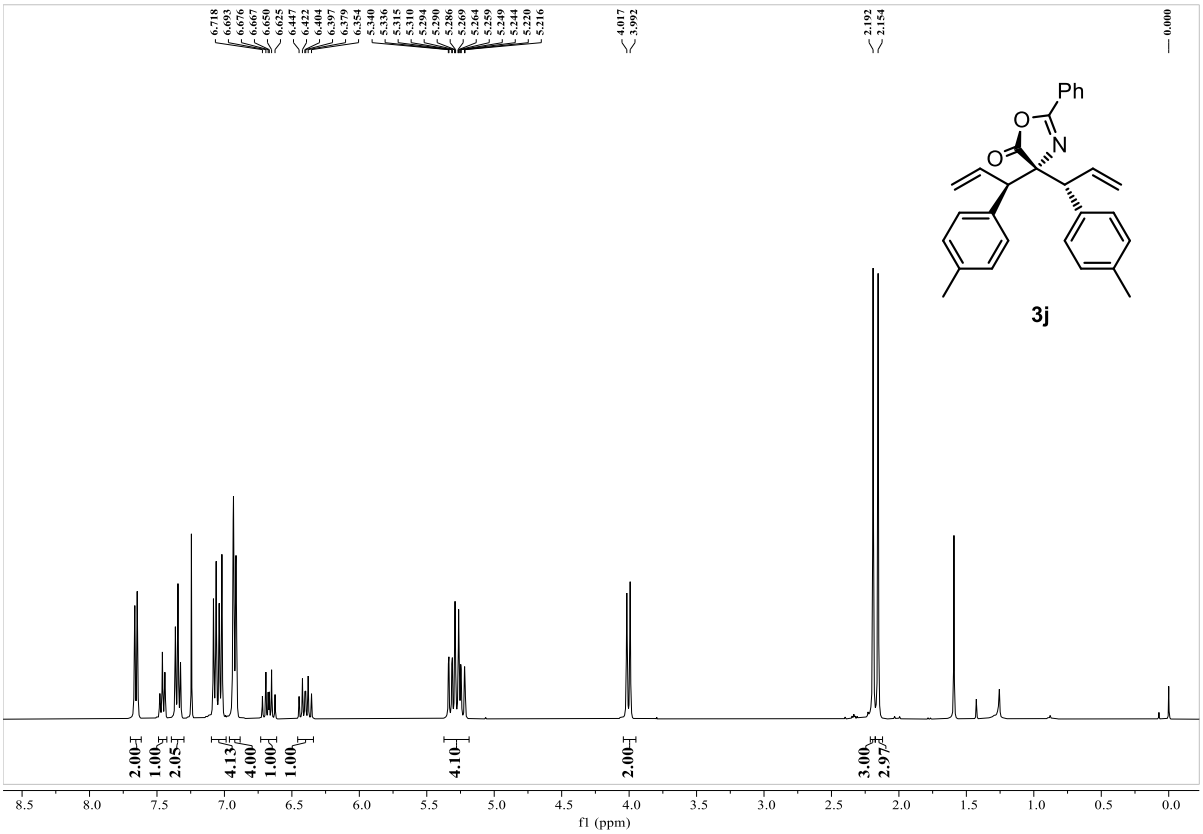
```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs

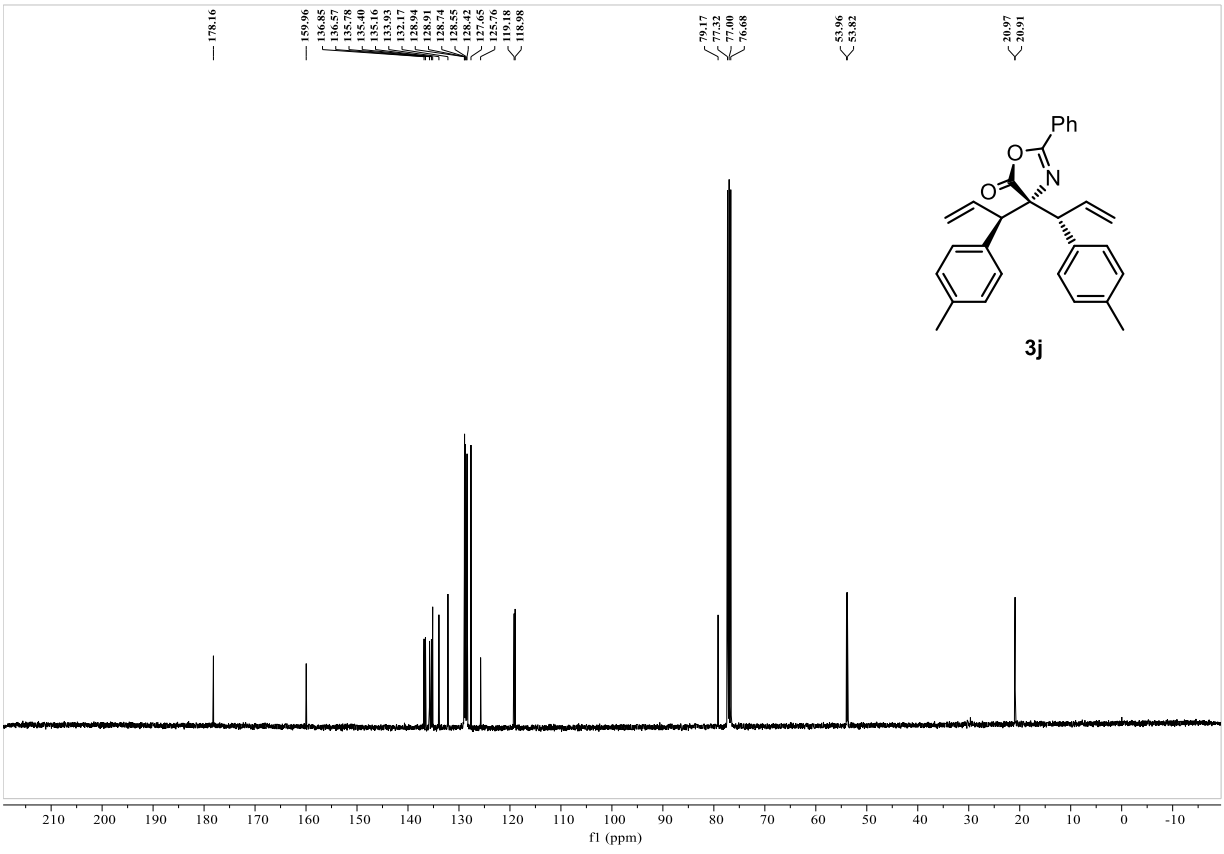

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]        %
----|-------|----|-------|----------|----------|--------|
   1   4.264 MF    0.1085 4665.75293  716.83716  99.5026
   2   4.604 FM    0.1499   23.32147    2.59236   0.4974

Totals :                  4689.07440  719.42952
```



^{1}H NMR (400 MHz, $CDCl_3$) of **3j**

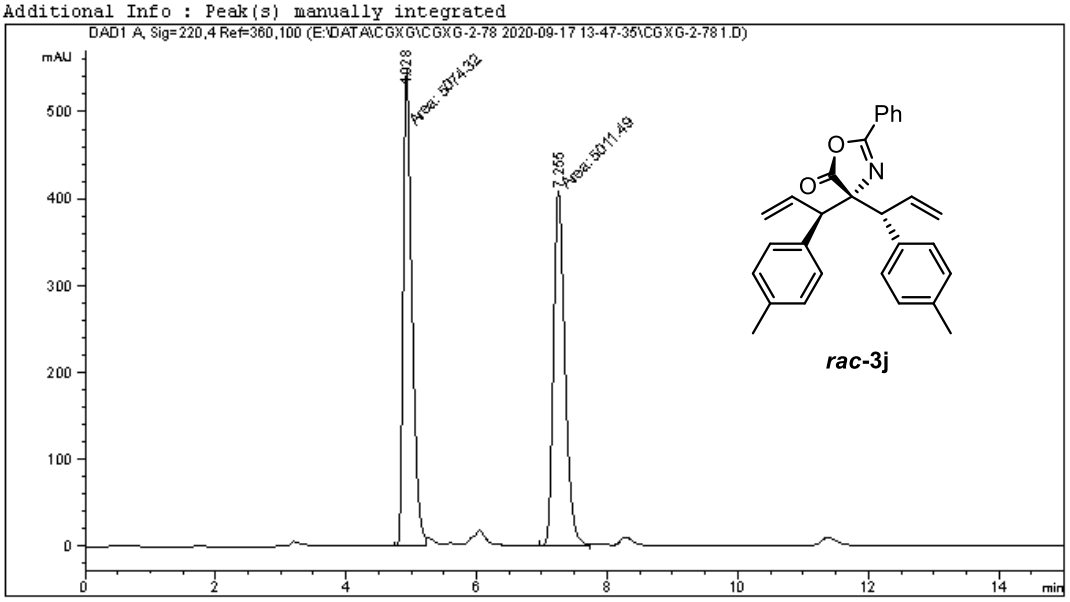


^{13}C NMR (101 MHz, $CDCl_3$) of **3j**

HPLC chromatogram of compound (*rac*)-3j

Data File E:\DATA\CGXG\CGXG-2-78 2020-09-17 13-47-35\CGXG-2-781.D
Sample Name: CGXG-2-78-RAC

```
======================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   2
Acq. Instrument : 1260                           Location :   57
Injection Date  : 9/17/2020 2:00:24 PM               Inj :   1
                                                Inj Volume : 2.000 µl
Acq. Method     : E:\DATA\CGXG\CGXG-2-78 2020-09-17 13-47-35\SC-2-ADH-98-2-DAD-1ML-30MIN-2UL.
                  M
Last changed    : 9/17/2020 1:47:35 PM by SYSTEM
Analysis Method : E:\DATA\CGXG\CGXG-2-78 2020-09-17 13-47-35\SC-2-ADH-98-2-DAD-1ML-30MIN-2UL.
                  M (Sequence Method)
Last changed    : 6/12/2021 9:46:28 AM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=220,4 Ref=360,100

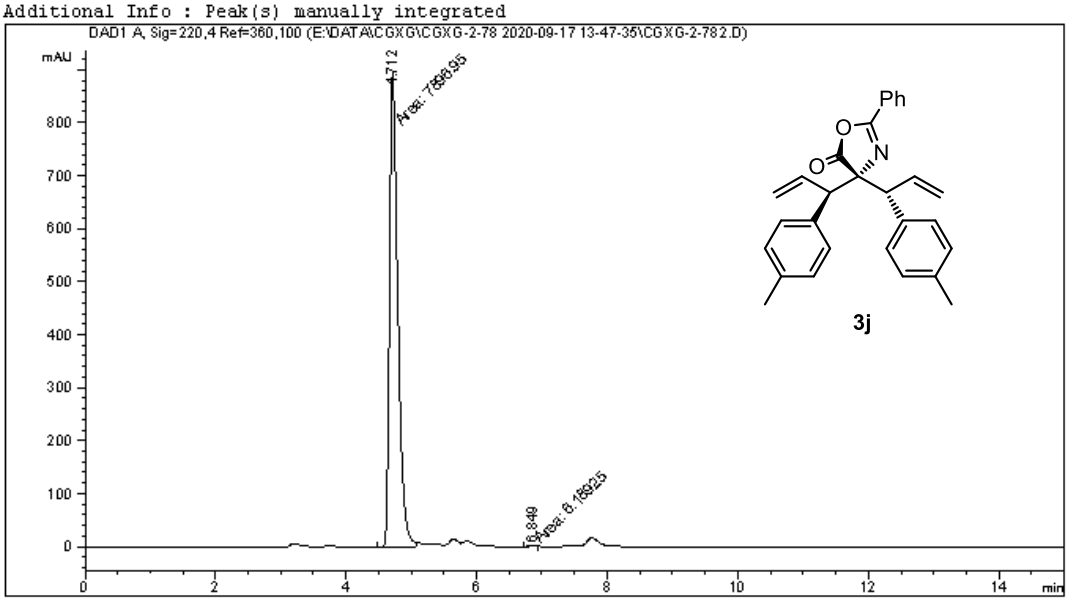
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   4.928 MF    0.1554 5074.31787  544.10046  50.3115
   2   7.255 MF    0.2051 5011.49268  407.31024  49.6885

Totals :                  1.00858e4   951.41071
```

HPLC chromatogram of compound 3j

Data File E:\DATA\CGXG\CGXG-2-78 2020-09-17 13-47-35\CGXG-2-782.D
Sample Name: CGXG-2-78-R

```
======================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   3
Acq. Instrument : 1260                           Location :   58
Injection Date  : 9/17/2020 2:31:54 PM               Inj :   1
                                                Inj Volume : 2.000 µl
Acq. Method     : E:\DATA\CGXG\CGXG-2-78 2020-09-17 13-47-35\SC-2-ADH-98-2-DAD-1ML-30MIN-2UL.
                  M
Last changed    : 9/17/2020 1:47:35 PM by SYSTEM
Analysis Method : E:\DATA\CGXG\CGXG-2-78 2020-09-17 13-47-35\SC-2-ADH-98-2-DAD-1ML-30MIN-2UL.
                  M (Sequence Method)
Last changed    : 6/12/2021 9:53:05 AM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```



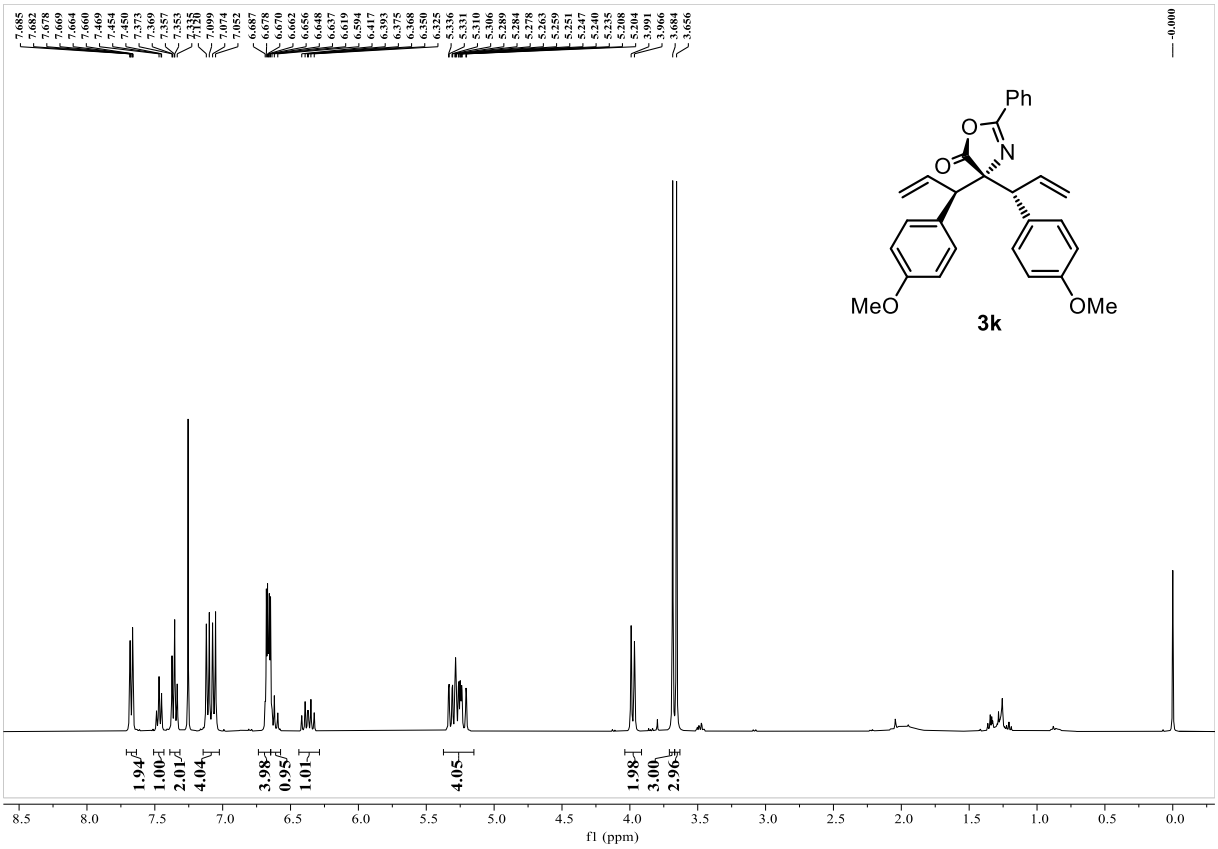

```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs

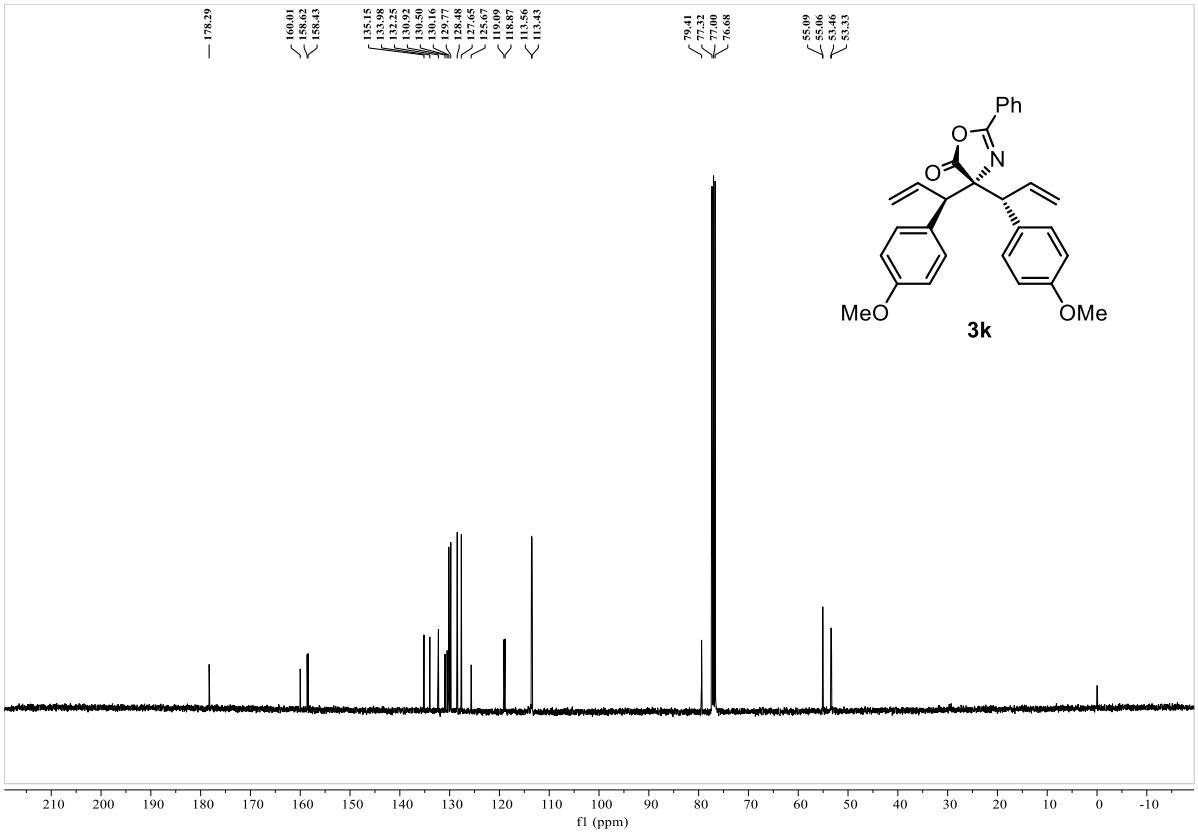

Signal 1: DAD1 A, Sig=220,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   4.712 MF    0.1473 7896.94922  893.49457  99.9217
   2   6.849 MM    0.1481    6.18925 6.96556e-1   0.0783

Totals :                  7903.13847  894.19112
```



^{1}H NMR (400 MHz, $CDCl_3$) of **3k**

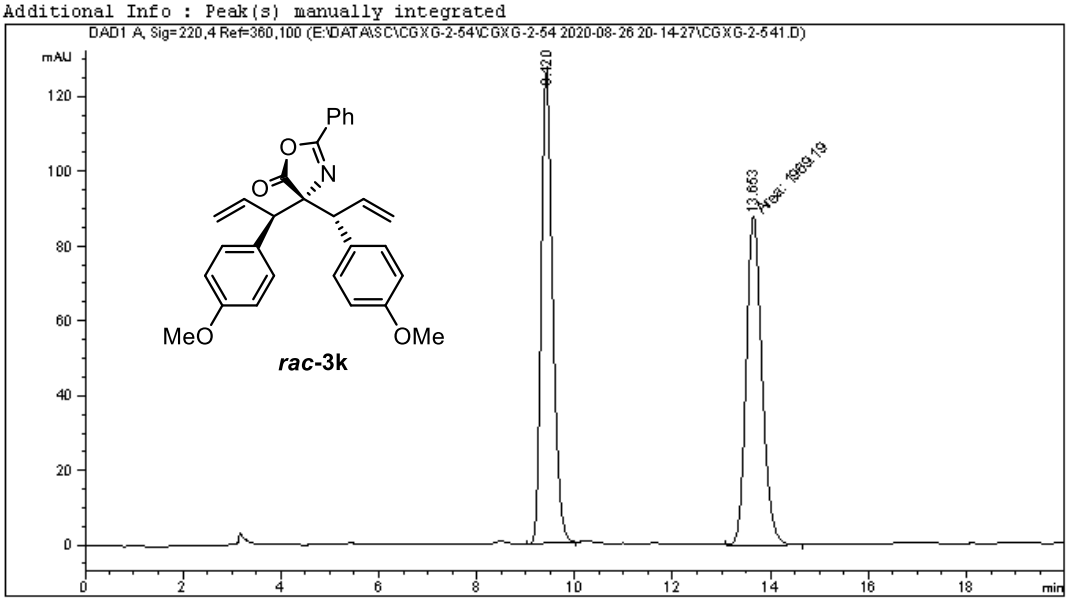


^{13}C NMR (101 MHz, $CDCl_3$) of **3k**

HPLC chromatogram of compound (*rac*)-3k

Data File E:\DATA\SC\CGXG-2-54\CGXG-2-54 2020-08-26 20-14-27\CGXG-2-541.D
Sample Name: CGXG-2-54

```
=====================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   2
Acq. Instrument : 1260                           Location :   43
Injection Date  : 8/26/2020 8:26:59 PM               Inj :   1
                                                Inj Volume : 2.000 µl
Acq. Method     : E:\DATA\SC\CGXG-2-54\CGXG-2-54 2020-08-26 20-14-27\SC-2-ADH-98-2-DAD-1ML-
                  30MIN-2UL.M
Last changed    : 8/26/2020 8:14:27 PM by SYSTEM
Analysis Method : E:\DATA\SC\CGXG-2-54\CGXG-2-54 2020-08-26 20-14-27\SC-2-ADH-98-2-DAD-1ML-
                  30MIN-2UL.M (Sequence Method)
Last changed    : 6/12/2021 9:24:33 AM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
=====================================================================
                        Area Percent Report
=====================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=220,4 Ref=360,100

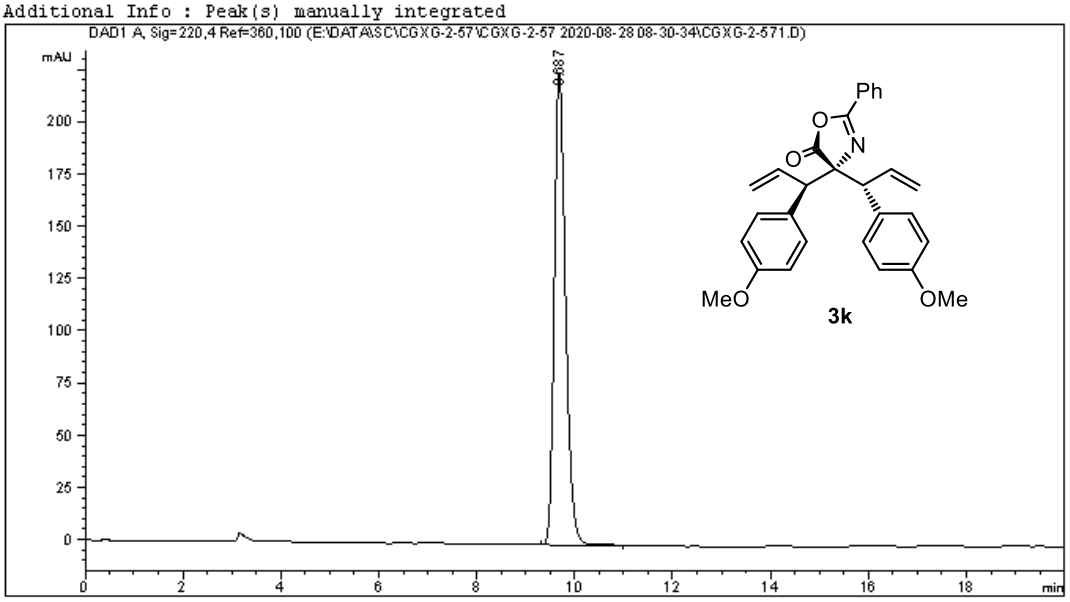
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   9.420 BB    0.2443 2029.50867  125.71338  50.7543
   2  13.653 MM    0.3740 1969.18555   87.74489  49.2457

Totals :                  3998.69421  213.45827
```

HPLC chromatogram of compound 3k

Data File E:\DATA\SC\CGXG-2-57\CGXG-2-57 2020-08-28 08-30-34\CGXG-2-571.D
Sample Name: CGXG-2-57-R

```
======================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   2
Acq. Instrument : 1260                           Location :   43
Injection Date  : 8/28/2020 8:43:46 AM               Inj :   1
                                                Inj Volume : 2.000 µl
Acq. Method     : E:\DATA\SC\CGXG-2-57\CGXG-2-57 2020-08-28 08-30-34\SC-2-ADH-98-2-DAD-1ML-
                  30MIN-2UL.M
Last changed    : 8/28/2020 8:30:34 AM by SYSTEM
Analysis Method : E:\DATA\SC\CGXG-2-57\CGXG-2-57 2020-08-28 08-30-34\SC-2-ADH-98-2-DAD-1ML-
                  30MIN-2UL.M (Sequence Method)
Last changed    : 6/12/2021 9:26:57 AM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```



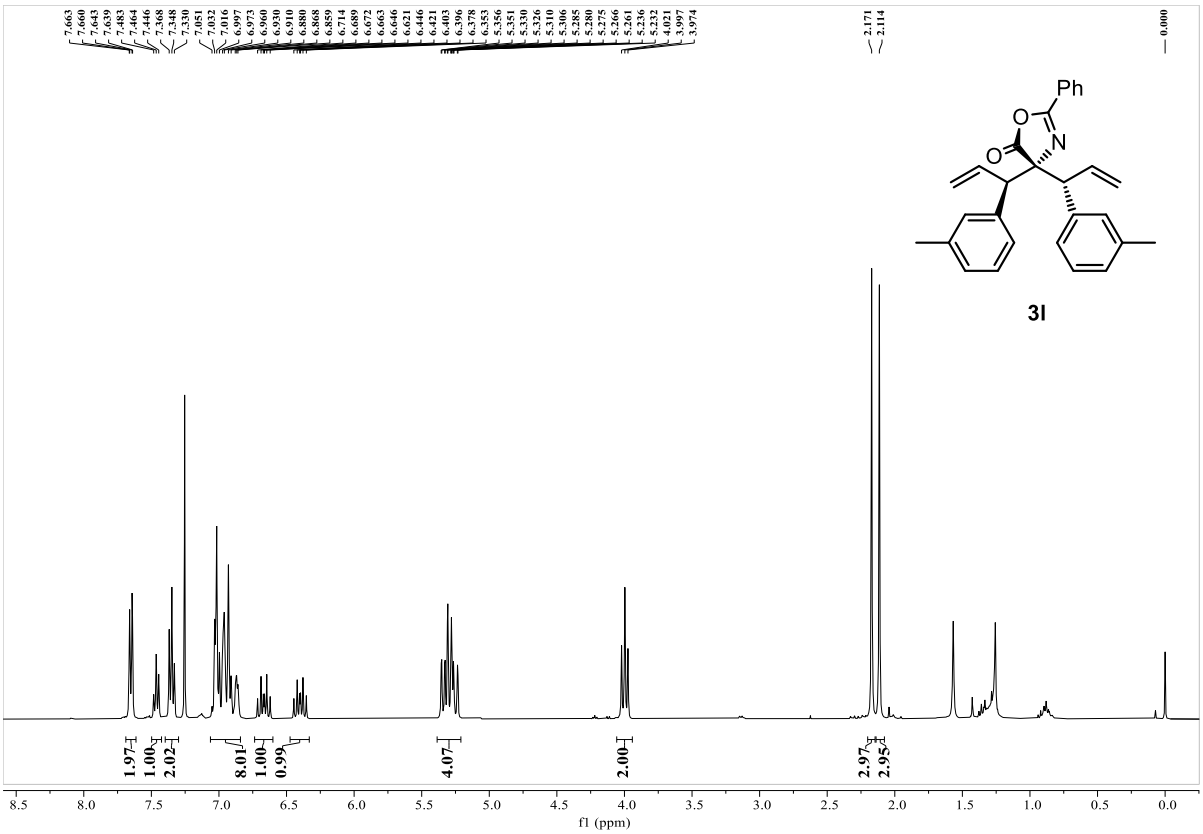

```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs

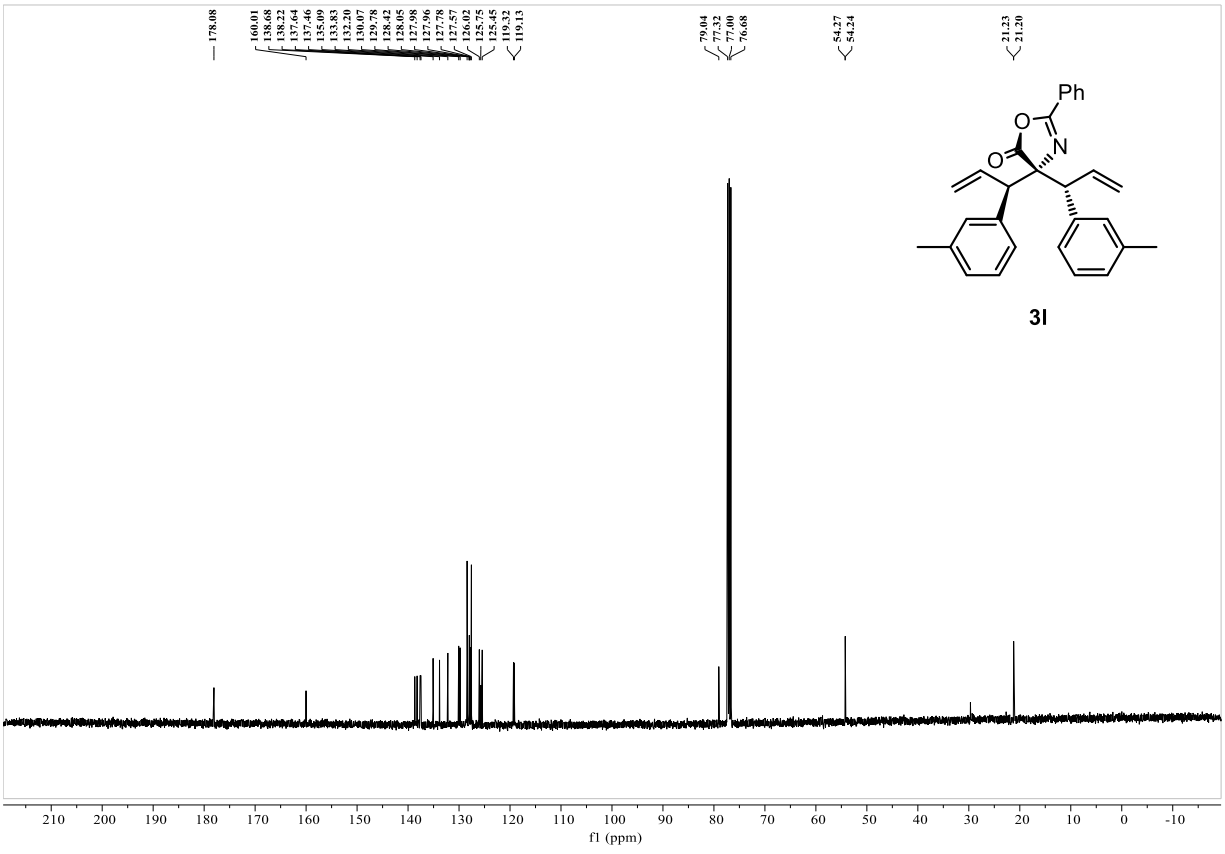

Signal 1: DAD1 A, Sig=220,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   9.687 BB    0.2471 3637.84521  225.66730 100.0000

Totals :                  3637.84521  225.66730
```



^{1}H NMR (400 MHz, $CDCl_3$) of **3l**

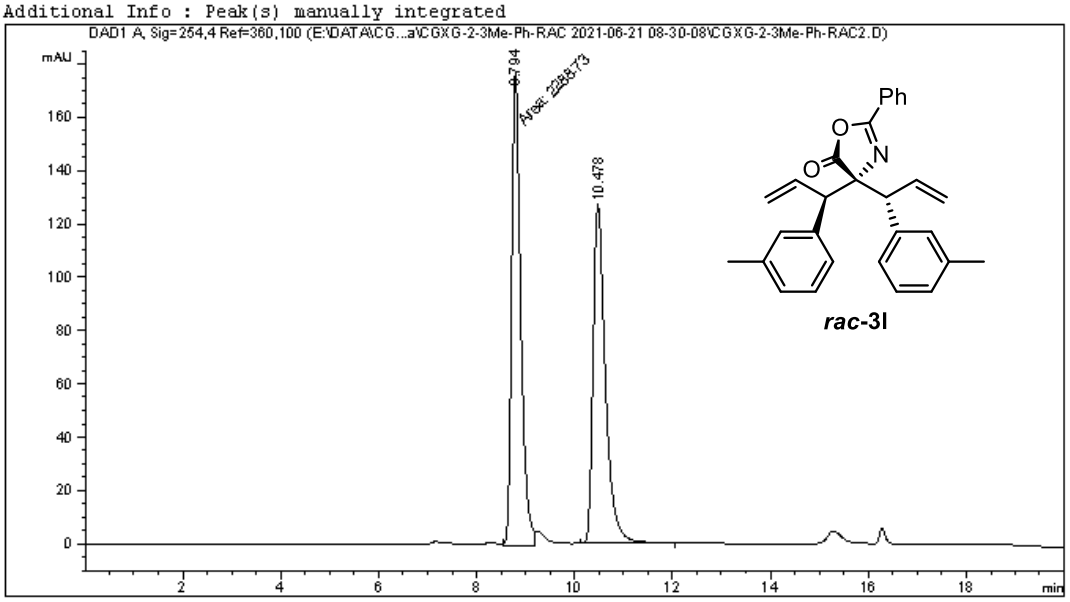


^{13}C NMR (101 MHz, $CDCl_3$) of **3l**

HPLC chromatogram of compound (*rac*)-3l

Data File E:\DATA\CG...ingjihua\CGXG-2-3Me-Ph-RAC 2021-06-21 08-30-08\CGXG-2-3Me-Ph-RAC2.D
Sample Name: CGXG-2-3Me-Ph-RAC

```
======================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   3
Acq. Instrument : 1260                           Location :   71
Injection Date  : 6/21/2021 9:24:06 AM               Inj :   1
                                               Inj Volume : 2.000 µl
Acq. Method     : E:\DATA\CGXG-2-Xibingjihua\CGXG-2-3Me-Ph-RAC 2021-06-21 08-30-08\CGXG-2-IE-
                  99-1-DAD-0.5ML-30MIN-2UL.M
Last changed    : 6/21/2021 9:42:15 AM by SYSTEM
                  (modified after loading)
Analysis Method : E:\DATA\CGXG-2-Xibingjihua\CGXG-2-3Me-Ph-RAC 2021-06-21 08-30-08\CGXG-2-IE-
                  99-1-DAD-0.5ML-30MIN-2UL.M (Sequence Method)
Last changed    : 6/21/2021 1:33:14 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=254,4 Ref=360,100

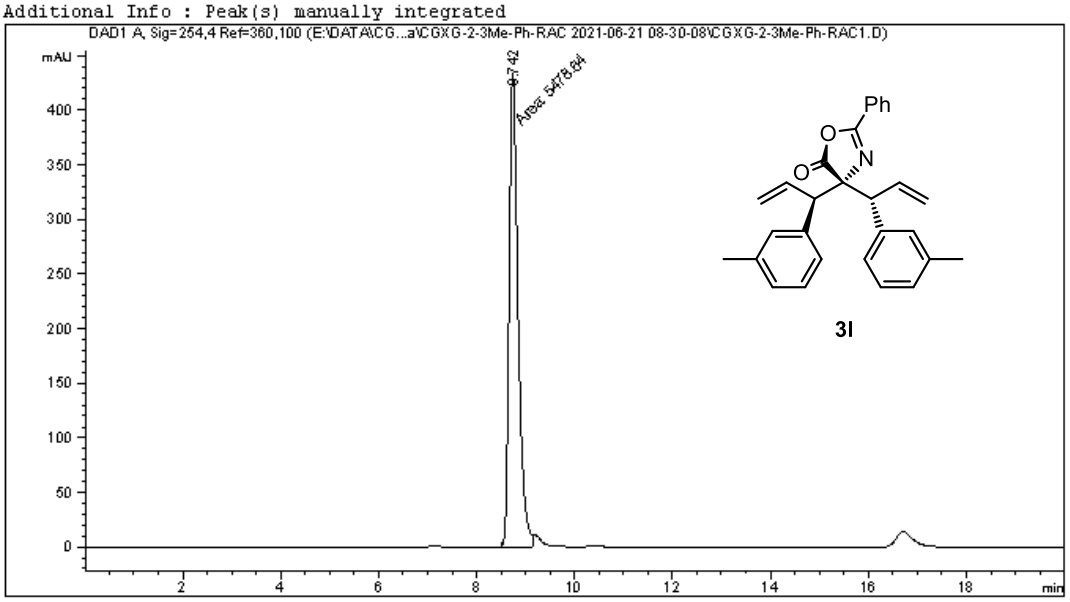
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]        %
----|-------|----|-------|----------|----------|--------|
   1   8.794 MF    0.2151 2288.72900  177.36864  50.8823
   2  10.478 BB    0.2599 2209.35156  127.04240  49.1177

Totals :                  4498.08057  304.41103
```

HPLC chromatogram of compound 3l

Data File E:\DATA\CG...ingjihua\CGXG-2-3Me-Ph-RAC 2021-06-21 08-30-08\CGXG-2-3Me-Ph-RAC1.D
Sample Name: CGXG-2-3Me-Ph-RRR

```
======================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   2
Acq. Instrument : 1260                           Location :   72
Injection Date  : 6/21/2021 9:02:47 AM               Inj :   1
                                                Inj Volume : 2.000 µl
Acq. Method     : E:\DATA\CGXG-2-Xibingjihua\CGXG-2-3Me-Ph-RAC 2021-06-21 08-30-08\CGXG-2-IE-
                  99-1-DAD-0.5ML-30MIN-2UL.M
Last changed    : 6/21/2021 9:18:31 AM by SYSTEM
                  (modified after loading)
Analysis Method : E:\DATA\CGXG-2-Xibingjihua\CGXG-2-3Me-Ph-RAC 2021-06-21 08-30-08\CGXG-2-IE-
                  99-1-DAD-0.5ML-30MIN-2UL.M (Sequence Method)
Last changed    : 6/21/2021 1:30:37 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```



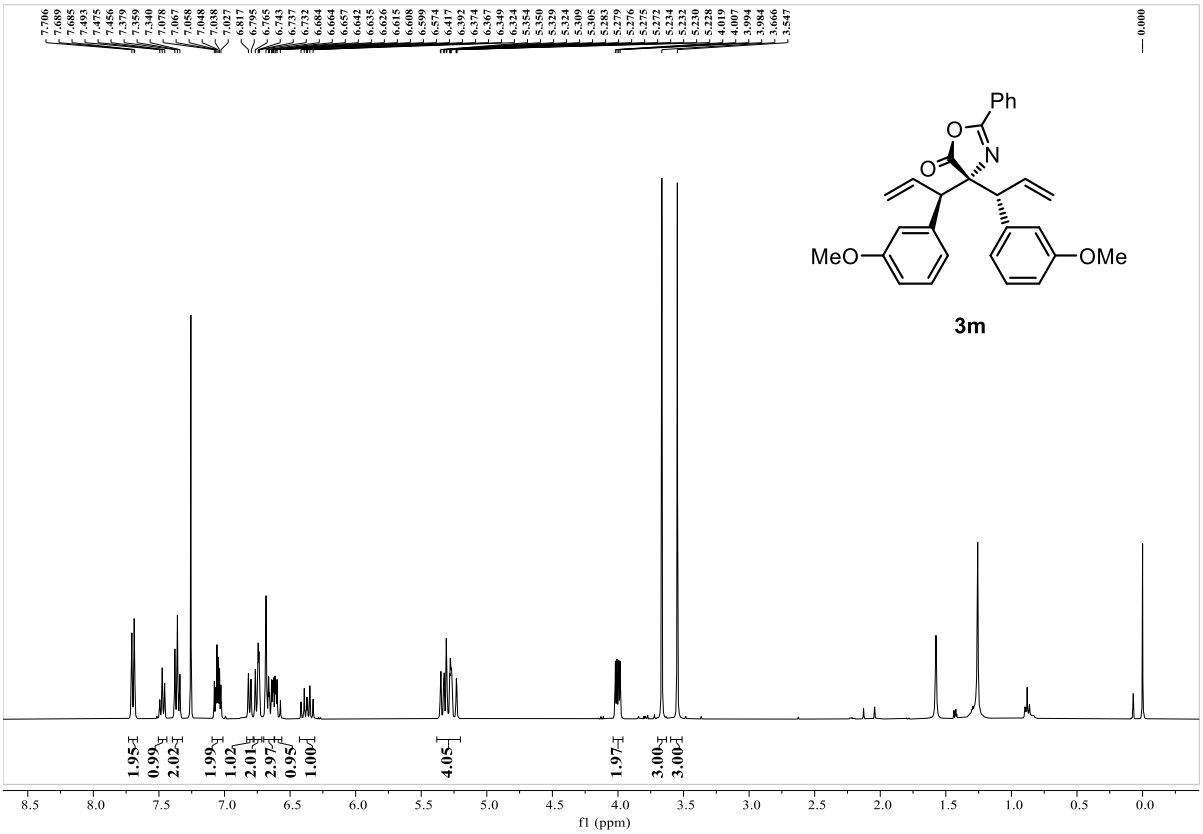

```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs

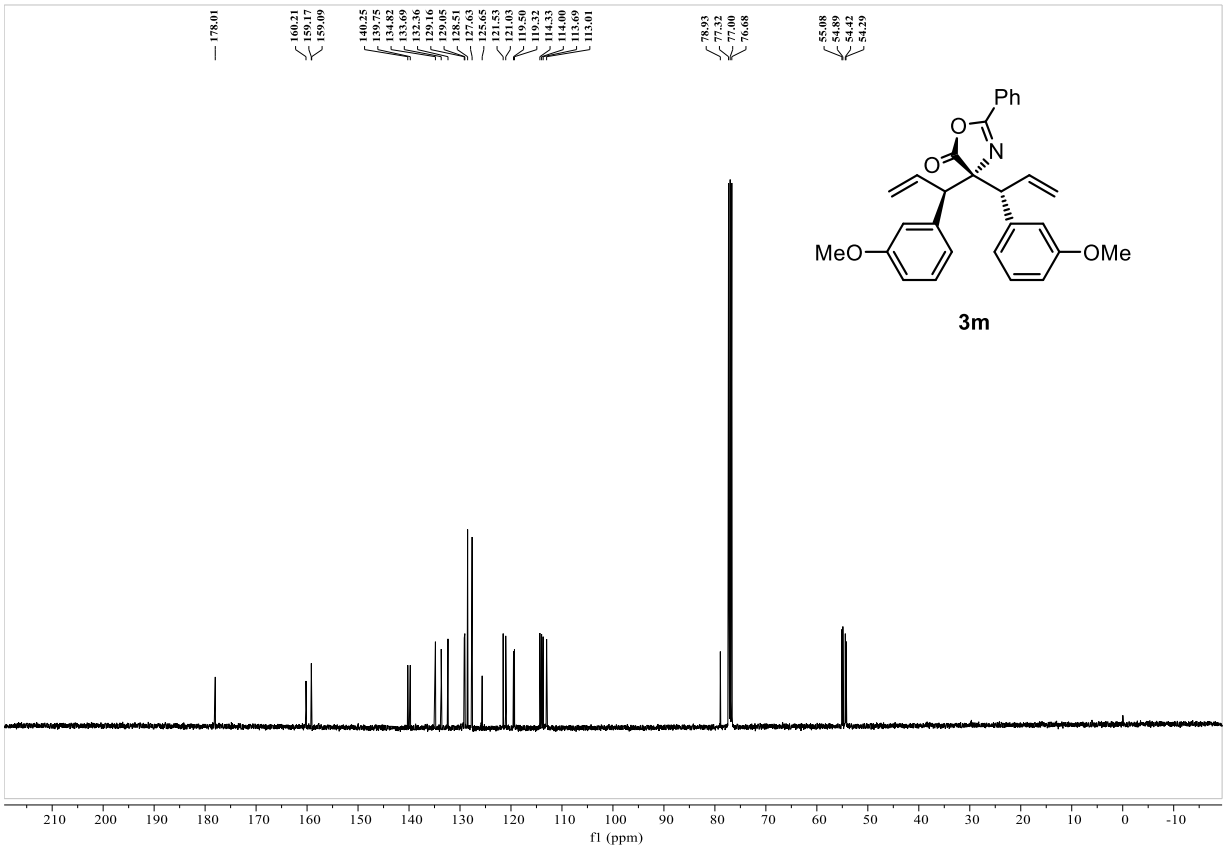

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]        %
----|-------|----|-------|----------|----------|--------|
   1   8.742 MF    0.2105 5478.64063  433.76361 100.0000

Totals :                  5478.64063  433.76361
```



^{1}H NMR (400 MHz, $CDCl_3$) of **3m**

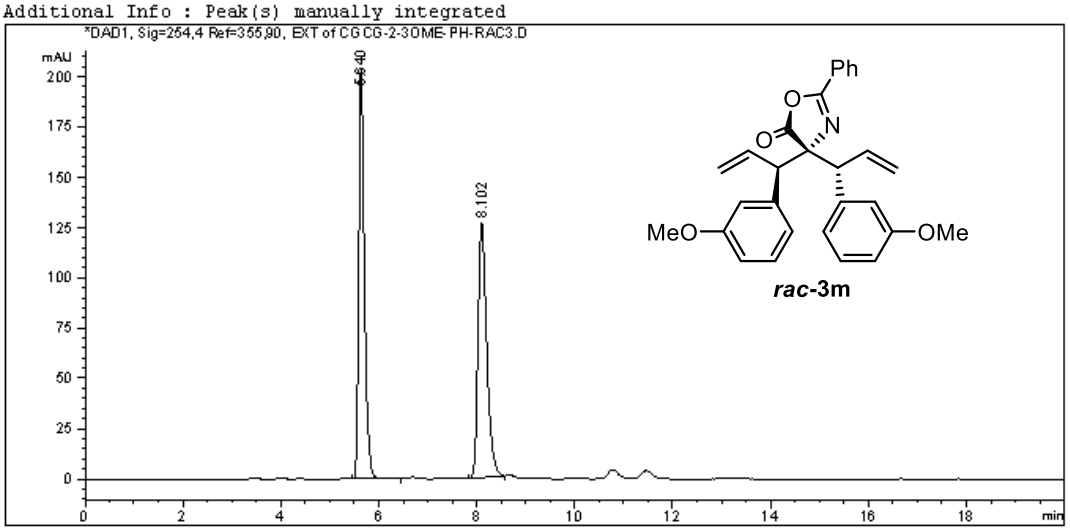


^{13}C NMR (101 MHz, $CDCl_3$) of **3m**

HPLC chromatogram of compound (*rac*)-3m

Data File E:\DATA\CG...GJIHUA\CGXG-2-30ME-PH-RAC 2021-06-20 11-16-34\CGCG-2-30ME-PH-RAC3.D
Sample Name: CGXG-2-30Me-Ph-RAC

```
======================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   4
Acq. Instrument : 1260                           Location :   73
Injection Date  : 6/20/2021 12:25:52 PM              Inj :   1
                                                Inj Volume : 2.000 µl
Acq. Method     : E:\DATA\CGXG-2-Xibingjihua\CGXG-2-30Me-Ph-RAC 2021-06-20 11-16-34\CGXG-2-IE
                  -90-10-DAD-1ML-30MIN-2UL.M
Last changed    : 6/20/2021 12:42:34 PM by SYSTEM
                  (modified after loading)
Analysis Method : E:\DATA\CGXG-2-Xibingjihua\CGXG-2-30Me-Ph-RAC 2021-06-20 11-16-34\CGXG-2-IE
                  -90-10-DAD-1ML-30MIN-2UL.M (Sequence Method)
Last changed    : 6/21/2021 1:37:19 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1, Sig=254,4 Ref=355,90, EXT
 Signal has been modified after loading from rawdata file!

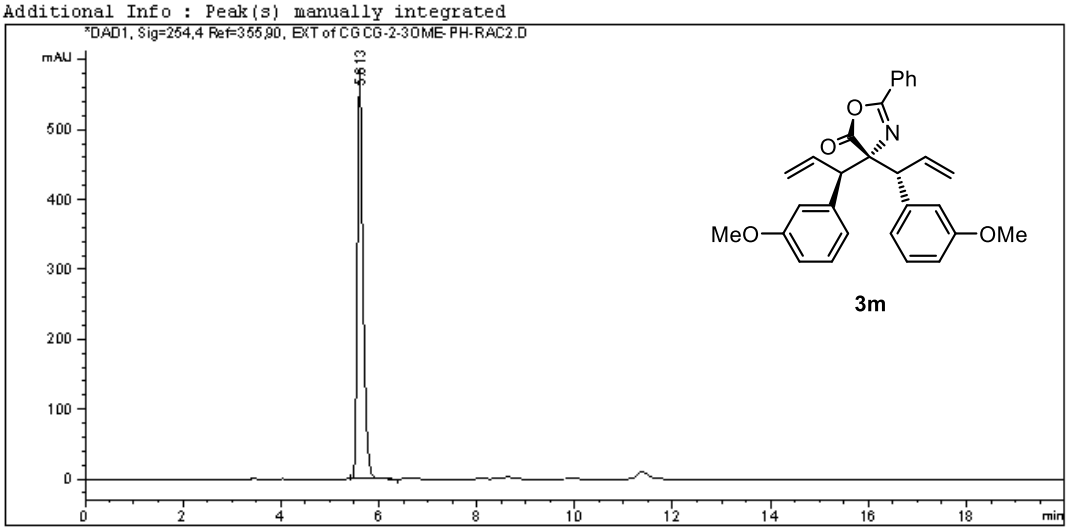
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   5.640 BB    0.1190 1618.64404  203.13576  50.8733
   2   8.102 BB    0.1878 1563.07043  126.81268  49.1267

Totals :                  3181.71448  329.94844
```

HPLC chromatogram of compound 3m

Data File E:\DATA\CG...GJIHUA\CGXG-2-30ME-PH-RAC 2021-06-20 11-16-34\CGCG-2-30ME-PH-RAC2.D
Sample Name: CGXG-2-30Me-Ph-R

```
======================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   3
Acq. Instrument : 1260                           Location :   72
Injection Date  : 6/20/2021 11:59:58 AM              Inj :   1
                                                Inj Volume : 2.000 µl
Acq. Method     : E:\DATA\CGXG-2-Xibingjihua\CGXG-2-30Me-Ph-RAC 2021-06-20 11-16-34\CGXG-2-IE
                  -90-10-DAD-1ML-30MIN-2UL.M
Last changed    : 6/20/2021 12:19:18 PM by SYSTEM
                  (modified after loading)
Analysis Method : E:\DATA\CGXG-2-Xibingjihua\CGXG-2-30Me-Ph-RAC 2021-06-20 11-16-34\CGXG-2-IE
                  -90-10-DAD-1ML-30MIN-2UL.M (Sequence Method)
Last changed    : 6/21/2021 1:38:06 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```



3m

```
======================================================================
                        Area Percent Report
======================================================================

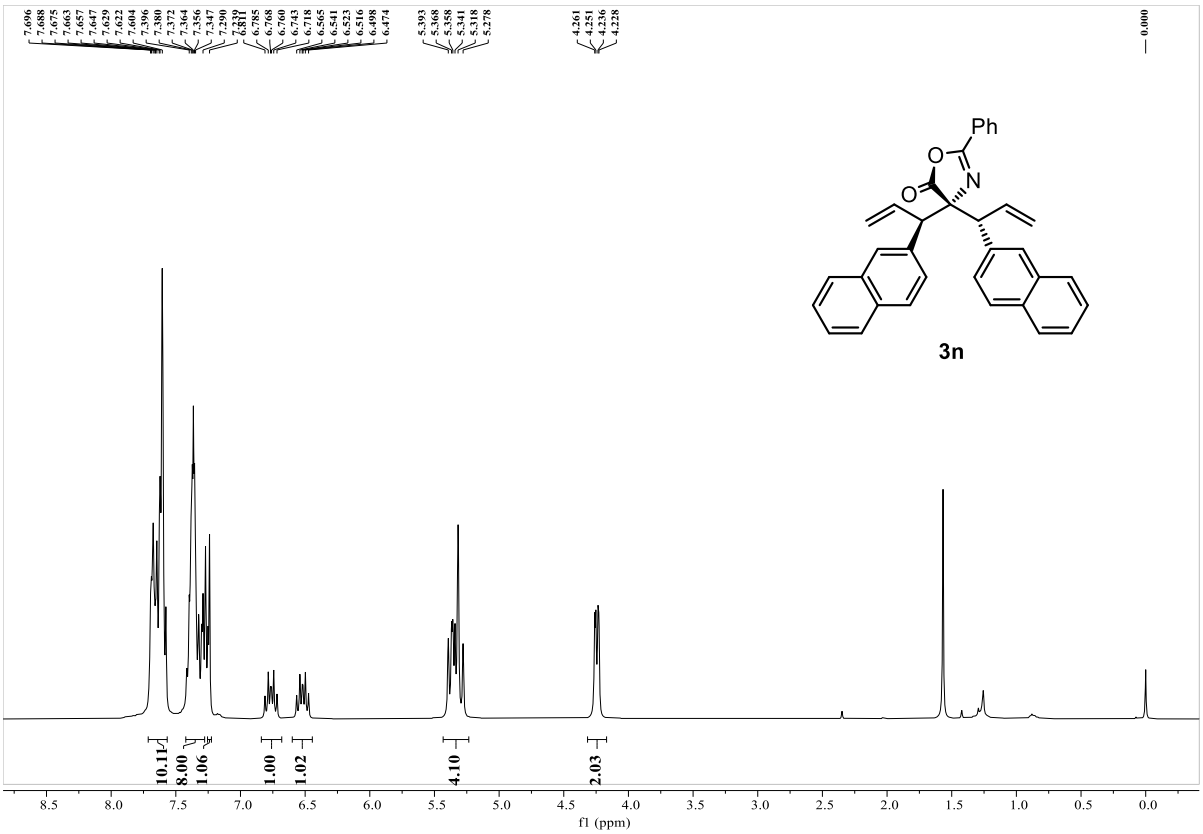
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1, Sig=254,4 Ref=355,90, EXT
 Signal has been modified after loading from rawdata file!

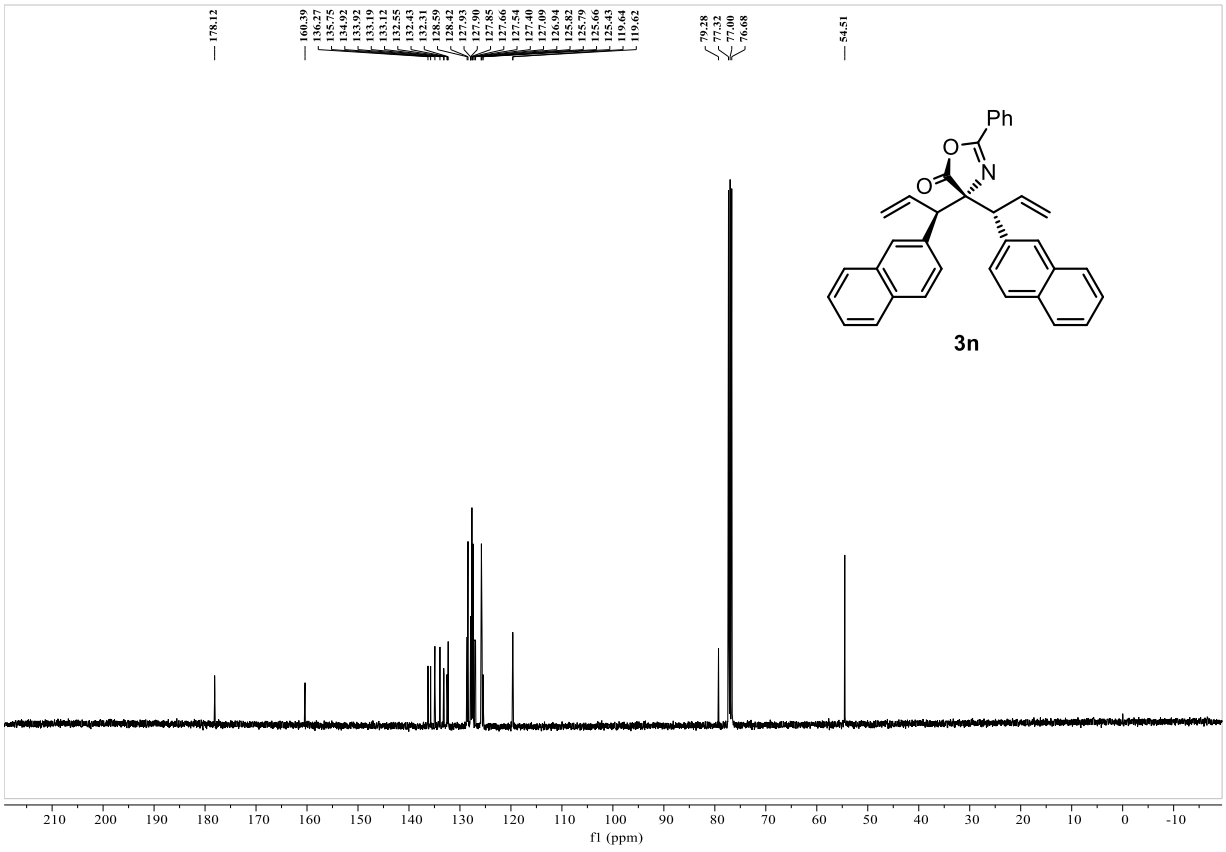
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   5.613 BB    0.1186 4641.52246  584.79779 100.0000

Totals :                  4641.52246  584.79779

======================================================================
```



^{1}H NMR (400 MHz, $CDCl_3$) of **3n**

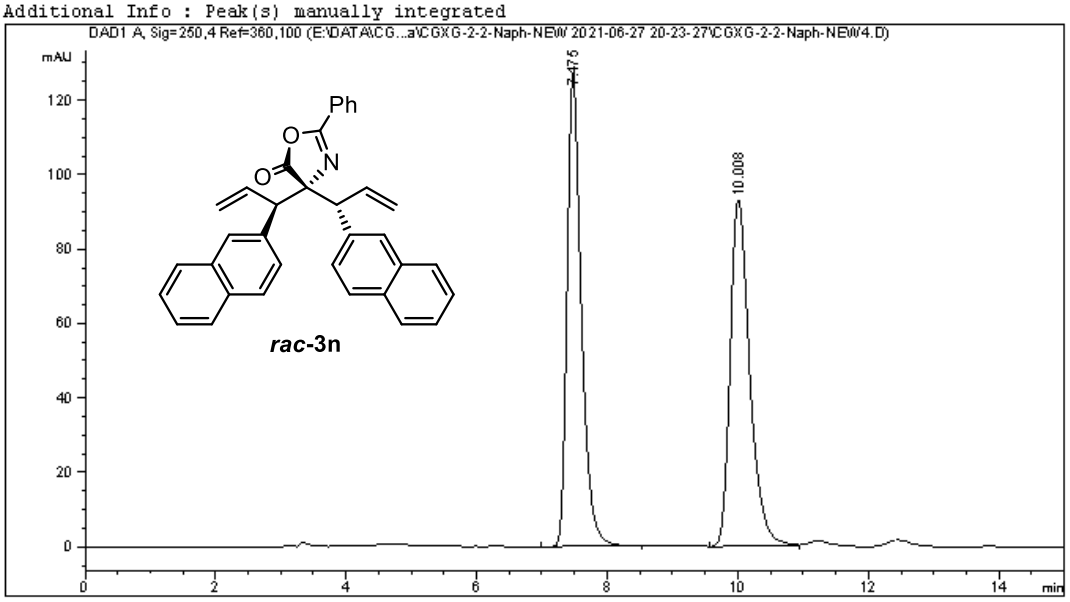


^{13}C NMR (101 MHz, $CDCl_3$) of **3n**

HPLC chromatogram of compound (*rac*)-3n

Data File E:\DATA\CG...ingjihua\CGXG-2-2-Naph-NEW 2021-06-27 20-23-27\CGXG-2-2-Naph-NEW4.D
Sample Name: CGXG-2-2-Naph-RAC

```
==================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   5
Acq. Instrument : 1260                           Location :   71
Injection Date  : 6/27/2021 10:08:18 PM              Inj :   1
                                                Inj Volume : 5.000 µl
Acq. Method     : E:\DATA\CGXG-2-Xibingjihua\CGXG-2-2-Naph-NEW 2021-06-27 20-23-27\CGXG-2-ADH
                  -99-1-DAD-0.5ML-30MIN-5UL.M
Last changed    : 6/27/2021 10:07:11 PM by SYSTEM
Analysis Method : E:\DATA\CGXG-2-Xibingjihua\CGXG-2-2-Naph-NEW 2021-06-27 20-23-27\CGXG-2-ADH
                  -99-1-DAD-0.5ML-30MIN-5UL.M (Sequence Method)
Last changed    : 7/11/2021 1:11:52 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==================================================================
                        Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=250,4 Ref=360,100

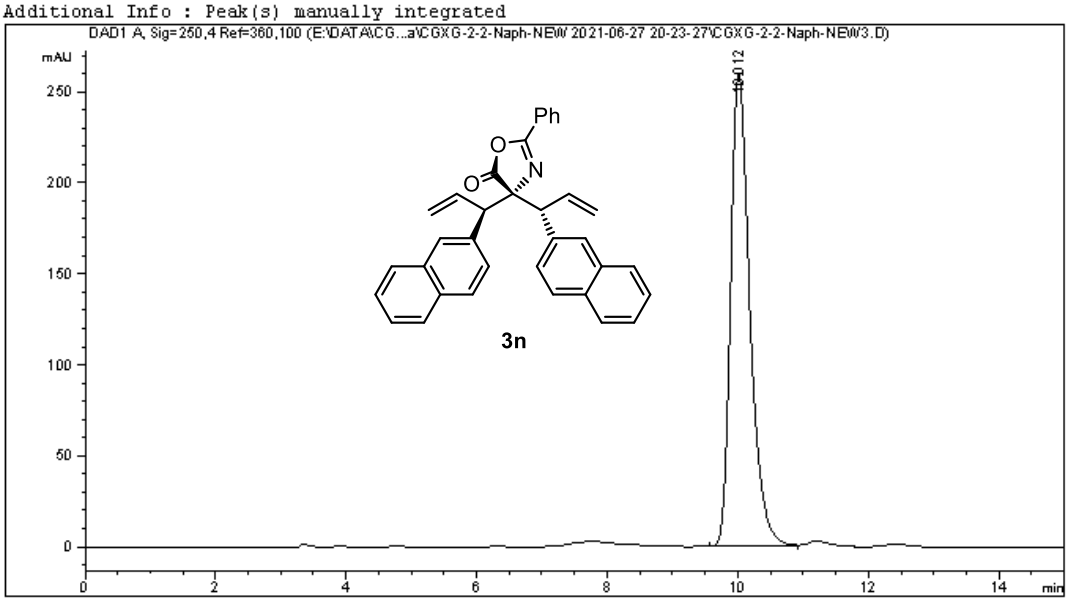
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   7.475 BB    0.2293 1932.90430  127.19111  50.0497
   2  10.008 BB    0.3158 1929.06519   93.09113  49.9503

Totals :                  3861.96948  220.28224
```

HPLC chromatogram of compound 3n

Data File E:\DATA\CG...ingjihua\CGXG-2-2-Naph-NEW 2021-06-27 20-23-27\CGXG-2-2-Naph-NEW3.D
Sample Name: CGXG-2-2-Naph-RRR

```
==================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   4
Acq. Instrument : 1260                           Location :   72
Injection Date  : 6/27/2021 9:45:23 PM               Inj :   1
                                                Inj Volume : 5.000 µl
Acq. Method     : E:\DATA\CGXG-2-Xibingjihua\CGXG-2-2-Naph-NEW 2021-06-27 20-23-27\CGXG-2-ADH
                  -99-1-DAD-0.5ML-30MIN-5UL.M
Last changed    : 6/27/2021 10:07:11 PM by SYSTEM
                  (modified after loading)
Analysis Method : E:\DATA\CGXG-2-Xibingjihua\CGXG-2-2-Naph-NEW 2021-06-27 20-23-27\CGXG-2-ADH
                  -99-1-DAD-0.5ML-30MIN-5UL.M (Sequence Method)
Last changed    : 7/11/2021 1:12:23 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```



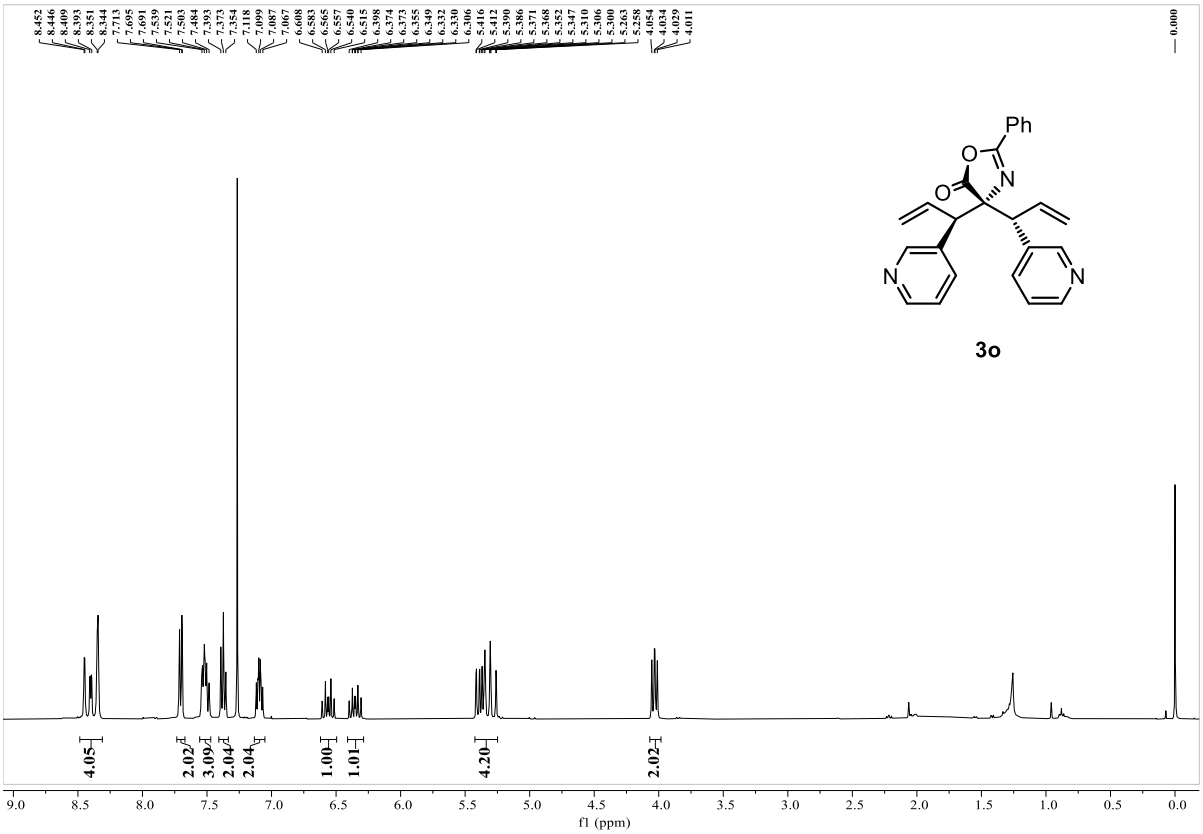

```
==================================================================
                        Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs

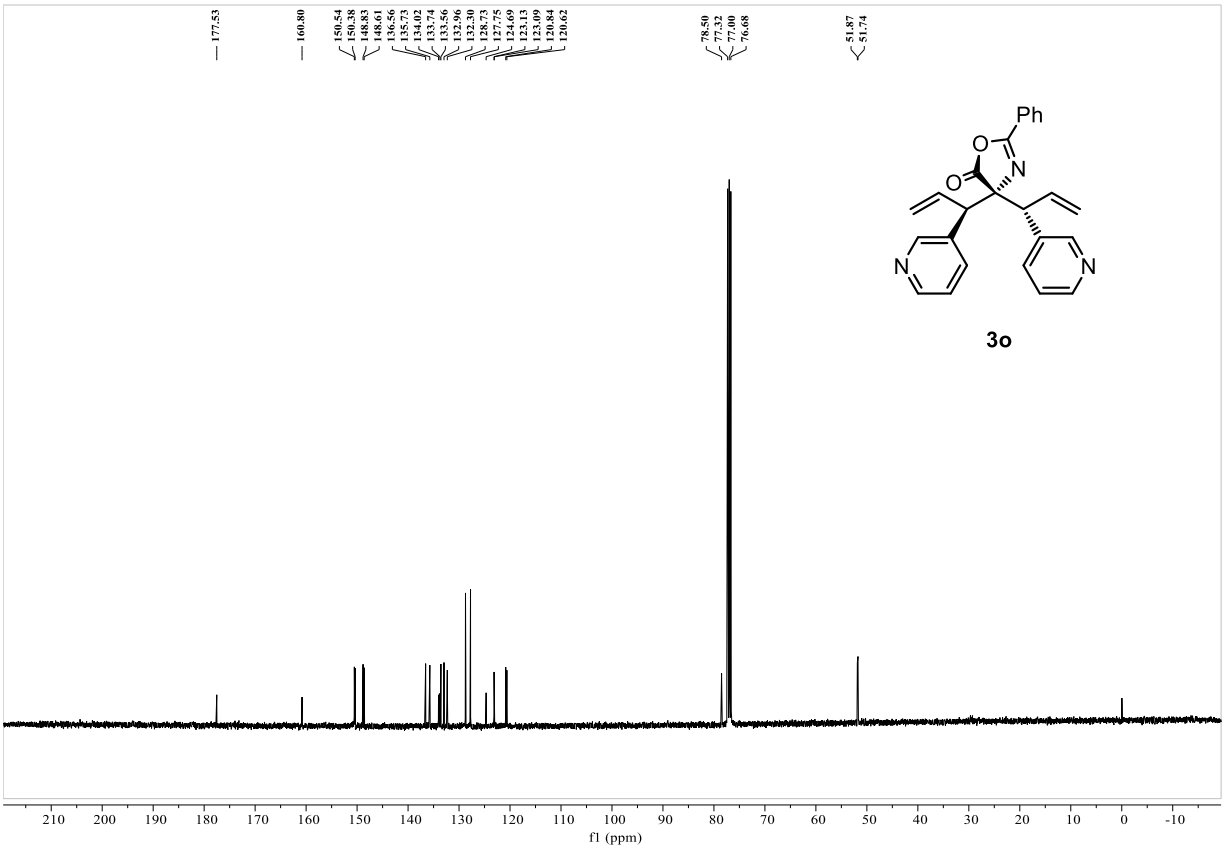

Signal 1: DAD1 A, Sig=250,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]        %
----|-------|----|-------|----------|----------|--------|
   1  10.012 BB    0.3082 5299.16260  259.54733 100.0000

Totals :                  5299.16260  259.54733
```



^{1}H NMR (400 MHz, $CDCl_3$) of **3o**

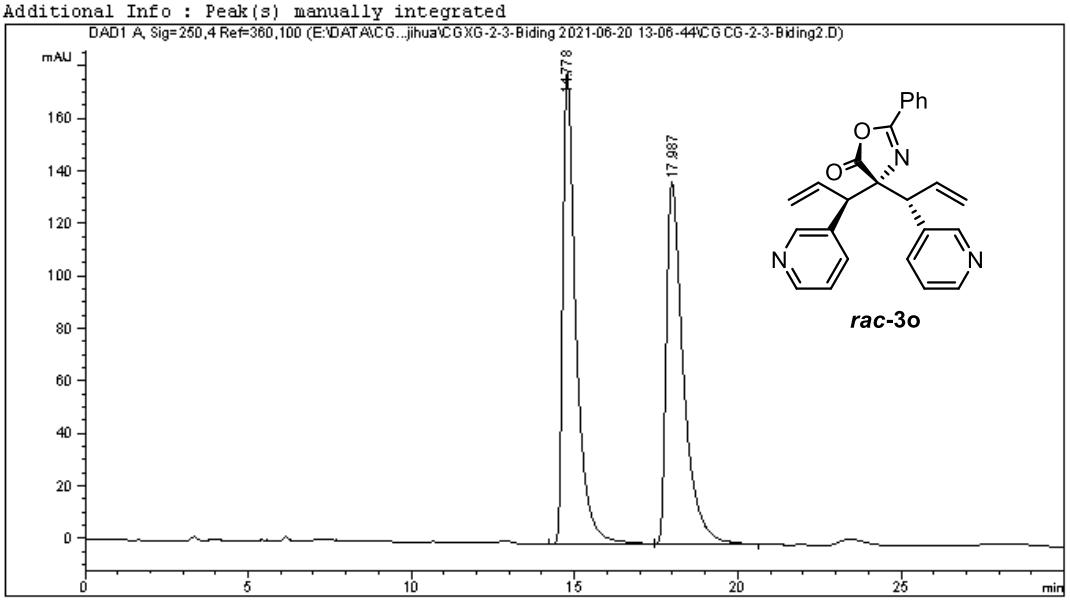


^{13}C NMR (101 MHz, $CDCl_3$) of **3o**

HPLC chromatogram of compound (*rac*)-3o

Data File E:\DATA\CG...-Xibingjihua\CGXG-2-3-Biding 2021-06-20 13-06-44\CGCG-2-3-Biding2.D
Sample Name: CGXG-2-3-Biding-RAC

```
==================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   3
Acq. Instrument : 1260                           Location :   73
Injection Date  : 6/20/2021 2:10:08 PM               Inj :   1
                                                Inj Volume : 5.000 µl
Acq. Method     : E:\DATA\CGXG-2-Xibingjihua\CGXG-2-3-Biding 2021-06-20 13-06-44\CGXG-2-IE-70
                  -30-DAD-1ML-30MIN-5UL.M
Last changed    : 6/20/2021 2:09:04 PM by SYSTEM
Analysis Method : E:\DATA\CGXG-2-Xibingjihua\CGXG-2-3-Biding 2021-06-20 13-06-44\CGXG-2-IE-70
                  -30-DAD-1ML-30MIN-5UL.M (Sequence Method)
Last changed    : 6/21/2021 2:08:55 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==================================================================
                        Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=250,4 Ref=360,100

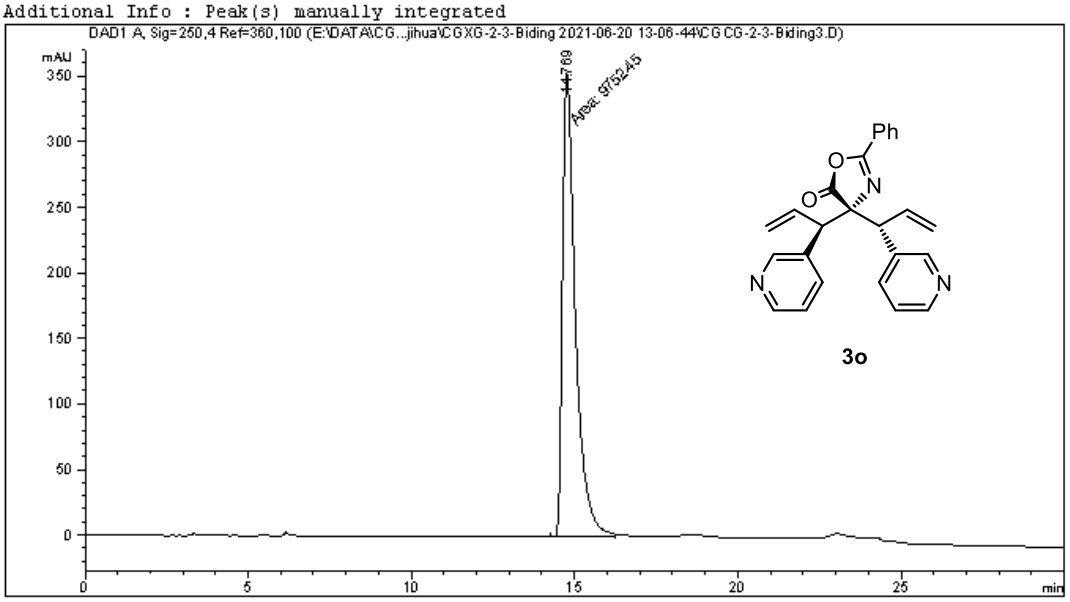
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  14.778 BB    0.4131 5018.15283  179.00841  50.0481
   2  17.987 BB    0.5448 5008.50391  137.55748  49.9519

Totals :                  1.00267e4   316.56589
```

HPLC chromatogram of compound 3o

Data File E:\DATA\CG...-Xibingjihua\CGXG-2-3-Biding 2021-06-20 13-06-44\CGCG-2-3-Biding3.D
Sample Name: CGXG-2-3-Biding-R

```
======================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   4
Acq. Instrument : 1260                           Location :   71
Injection Date  : 6/20/2021 2:41:44 PM               Inj :   1
                                                Inj Volume : 5.000 µl
Acq. Method     : E:\DATA\CGXG-2-Xibingjihua\CGXG-2-3-Biding 2021-06-20 13-06-44\CGXG-2-IE-70
                  -30-DAD-1ML-30MIN-5UL.M
Last changed    : 6/20/2021 3:11:47 PM by SYSTEM
                  (modified after loading)
Analysis Method : E:\DATA\CGXG-2-Xibingjihua\CGXG-2-3-Biding 2021-06-20 13-06-44\CGXG-2-IE-70
                  -30-DAD-1ML-30MIN-5UL.M (Sequence Method)
Last changed    : 6/21/2021 2:09:38 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
======================================================================
                        Area Percent Report
======================================================================

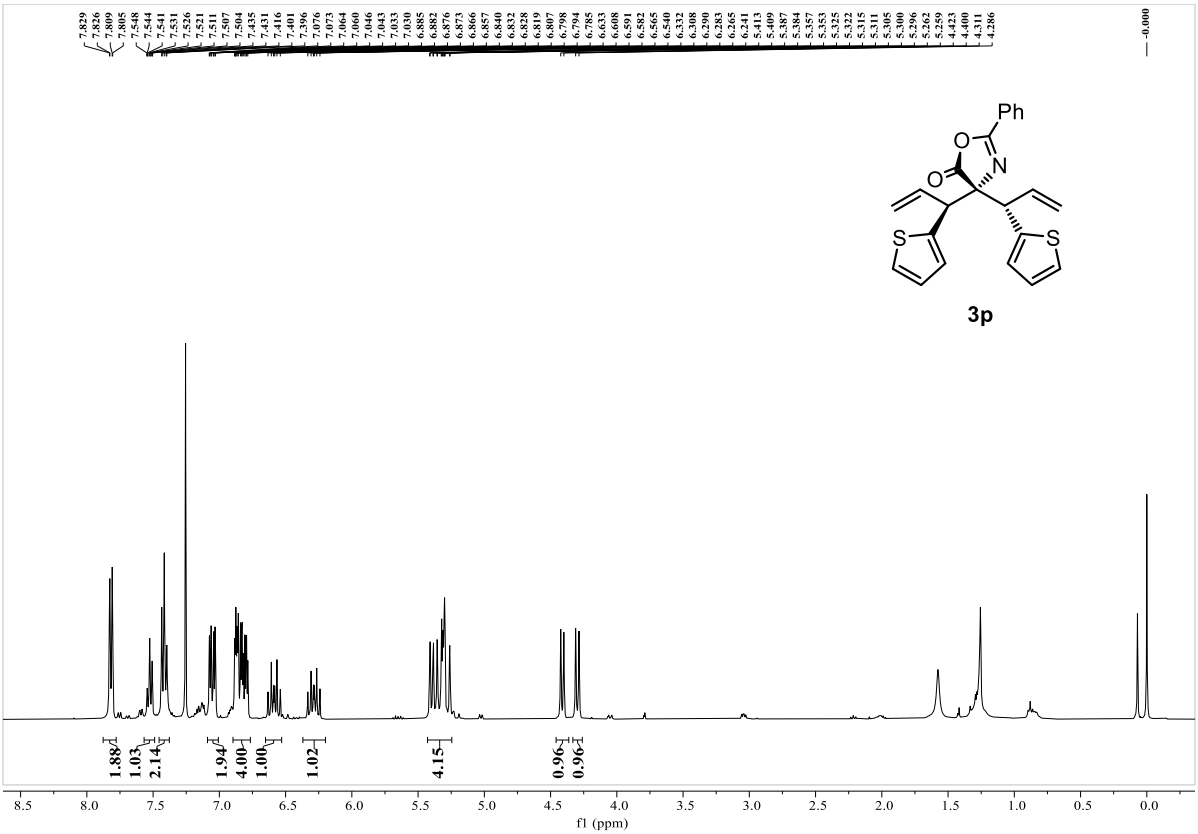
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=250,4 Ref=360,100

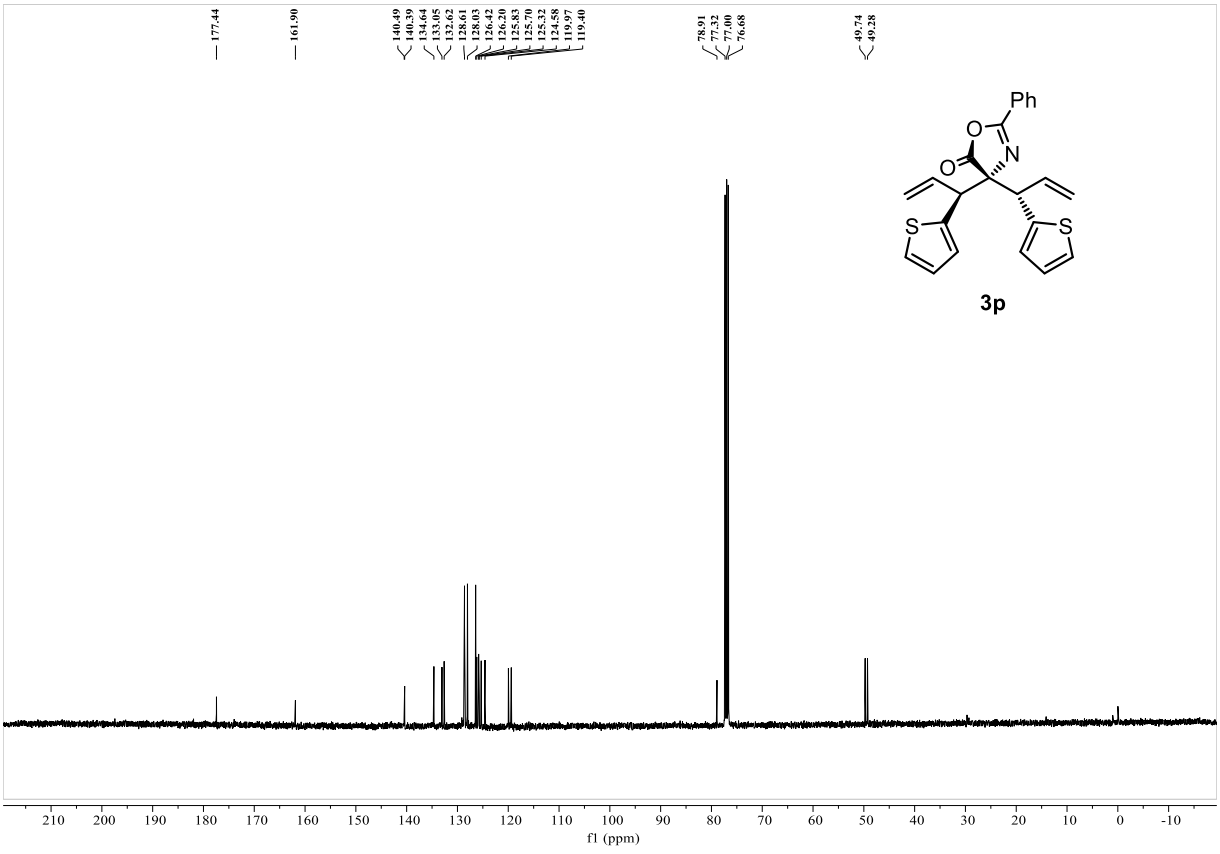
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]        %
----|-------|----|-------|----------|----------|--------|
   1  14.769 MF    0.4599 9752.44629  353.40515 100.0000

Totals :                  9752.44629  353.40515
```

1260 6/21/2021 2:09:40 PM SYSTEM



^{1}H NMR (400 MHz, $CDCl_3$) of **3p**

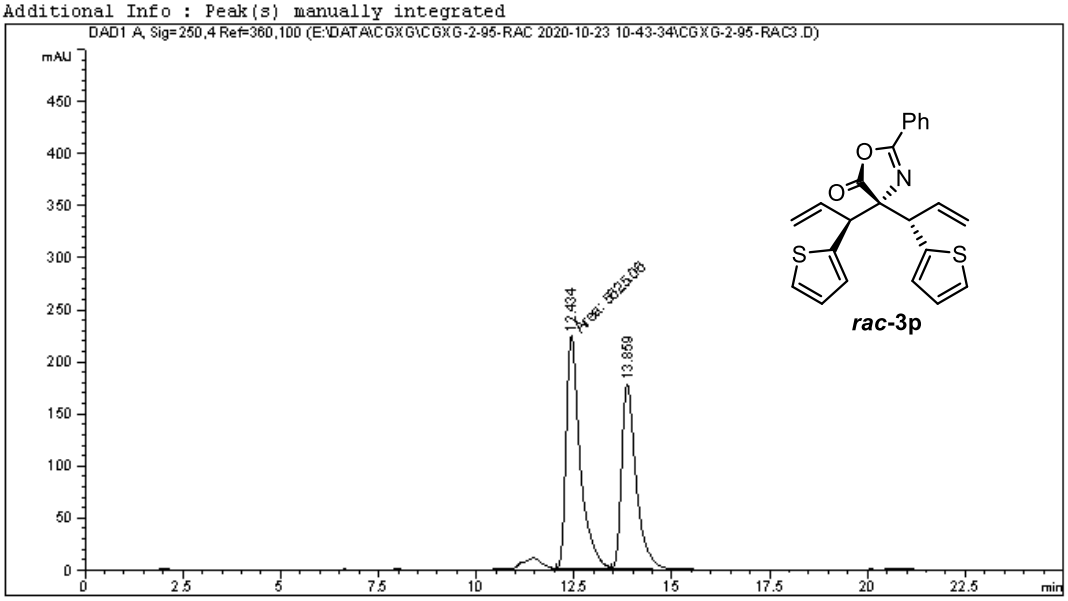


^{13}C NMR (101 MHz, $CDCl_3$) of **3p**

HPLC chromatogram of compound (*rac*)-3p

Data File E:\DATA\CGXG\CGXG-2-95-RAC 2020-10-23 10-43-34\CGXG-2-95-RAC3.D
Sample Name: CGXG-2-95-RAC

```
==========================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   4
Acq. Instrument : 1260                           Location :   74
Injection Date  : 10/23/2020 11:25:32 AM             Inj :   1
                                               Inj Volume : 2.000 µl
Acq. Method     : E:\DATA\CGXG\CGXG-2-95-RAC 2020-10-23 10-43-34\CGXG-2-ADH-99-1-DAD-0.5ML-
                  30MIN-2UL.M
Last changed    : 10/23/2020 10:43:34 AM by SYSTEM
Analysis Method : E:\DATA\CGXG\CGXG-2-95-RAC 2020-10-23 10-43-34\CGXG-2-ADH-99-1-DAD-0.5ML-
                  30MIN-2UL.M (Sequence Method)
Last changed    : 12/30/2021 9:56:47 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=250,4 Ref=360,100

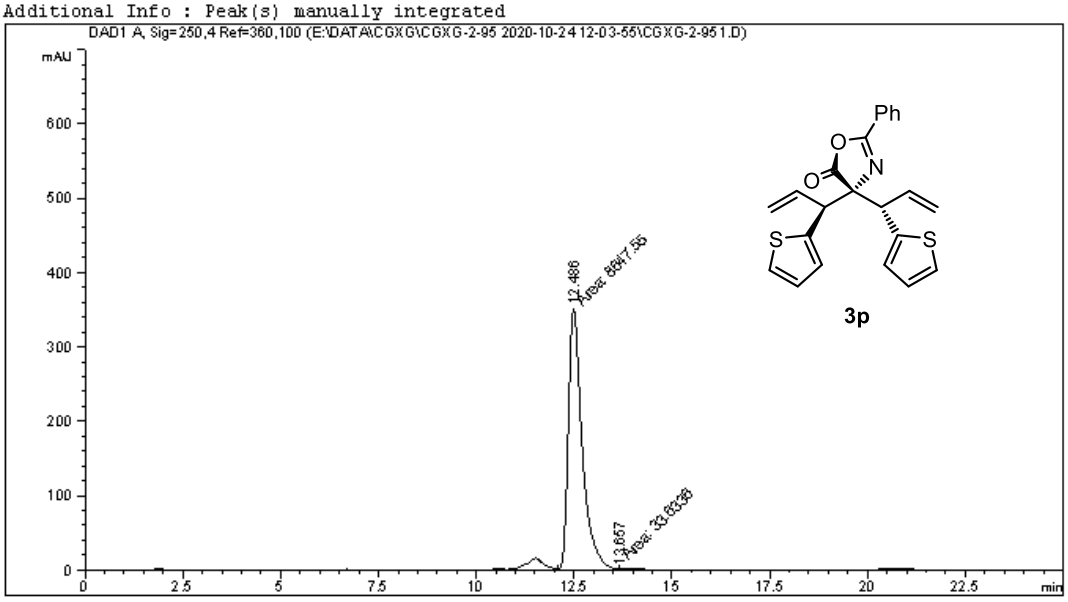
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]        %
----|-------|----|-------|----------|----------|--------|
   1  12.434 FM    0.4182 5625.06299  224.15239  54.9699
   2  13.859 VB    0.3876 4607.93018  178.26364  45.0301

Totals :                  1.02330e4   402.41603
```

HPLC chromatogram of compound 3p

Data File E:\DATA\CGXG\CGXG-2-95 2020-10-24 12-03-55\CGXG-2-951.D
Sample Name: CGXG-2-95-R

```
======================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   2
Acq. Instrument : 1260                           Location :   33
Injection Date  : 10/24/2020 12:15:52 PM             Inj :   1
                                                Inj Volume : 2.000 µl
Acq. Method     : E:\DATA\CGXG\CGXG-2-95 2020-10-24 12-03-55\CGXG-2-ADH-99-1-DAD-0.5ML-30MIN-
                  2UL.M
Last changed    : 10/24/2020 12:03:55 PM by SYSTEM
Analysis Method : E:\DATA\CGXG\CGXG-2-95 2020-10-24 12-03-55\CGXG-2-ADH-99-1-DAD-0.5ML-30MIN-
                  2UL.M (Sequence Method)
Last changed    : 12/30/2021 10:00:36 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```



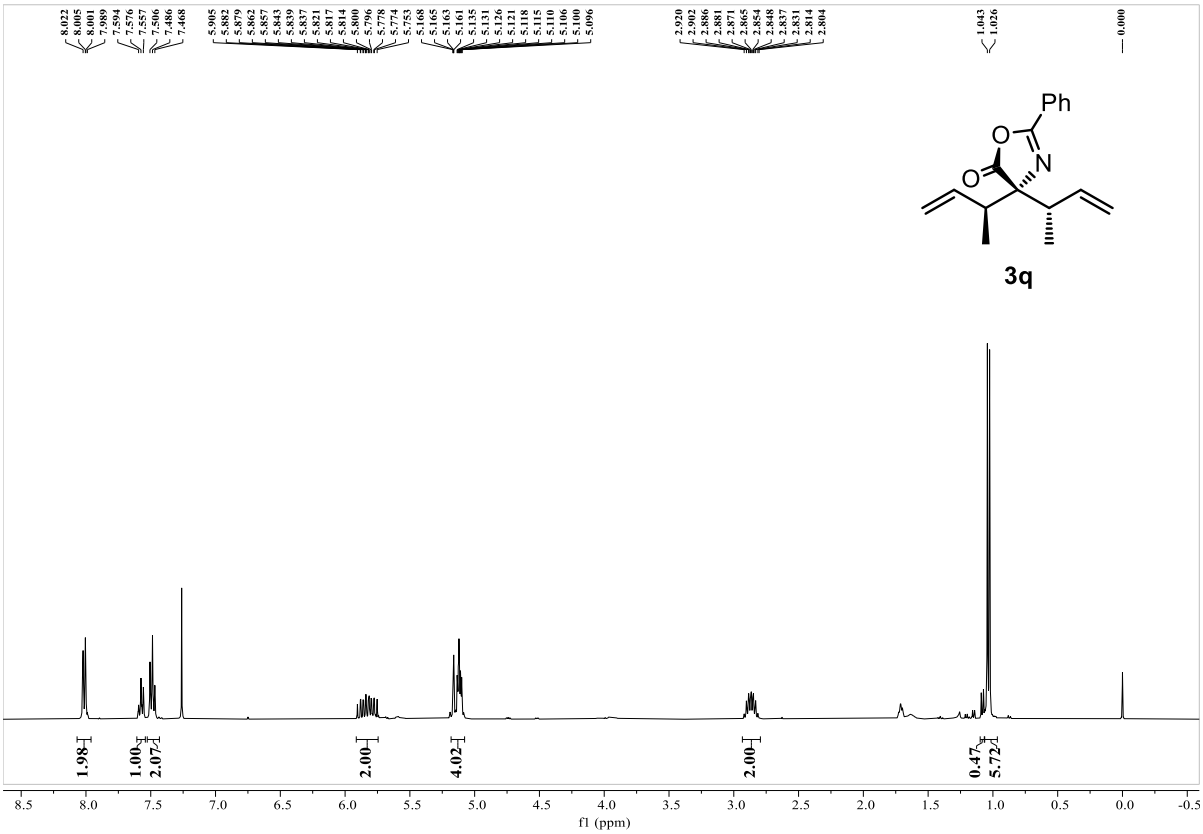

```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs

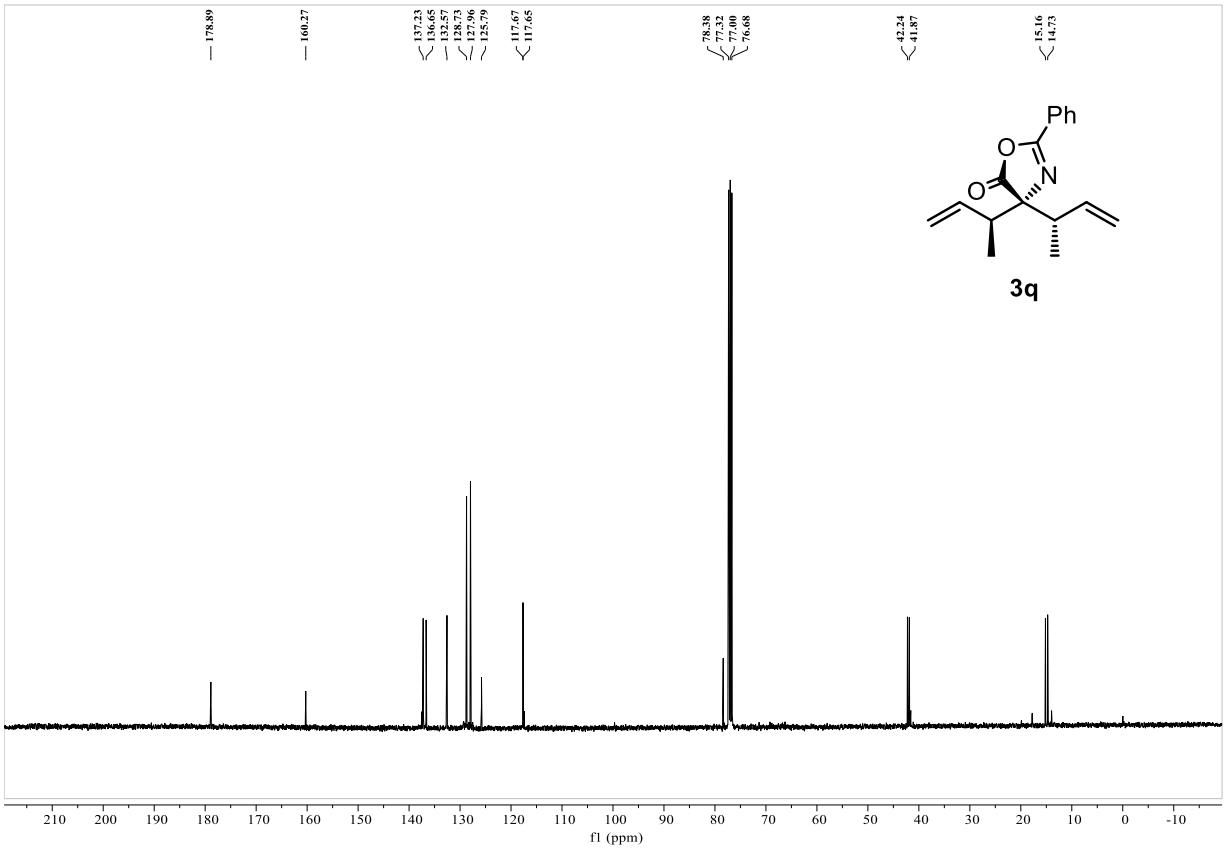

Signal 1: DAD1 A, Sig=250,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]        %
----|-------|----|-------|----------|----------|--------|
   1  12.486 MF    0.4109 8647.55273  350.77515  99.6126
   2  13.657 FM    0.4604   33.63358    1.21767   0.3874

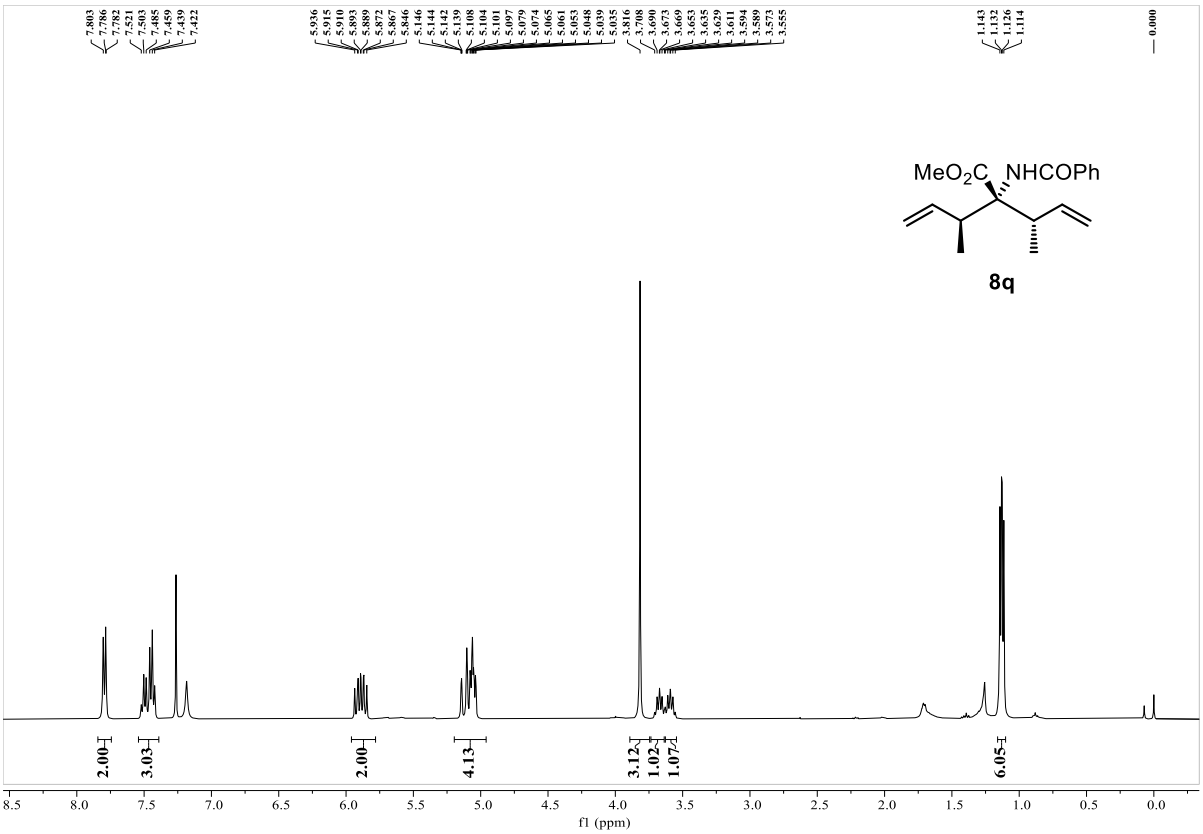
Totals :                  8681.18631  351.99281
```



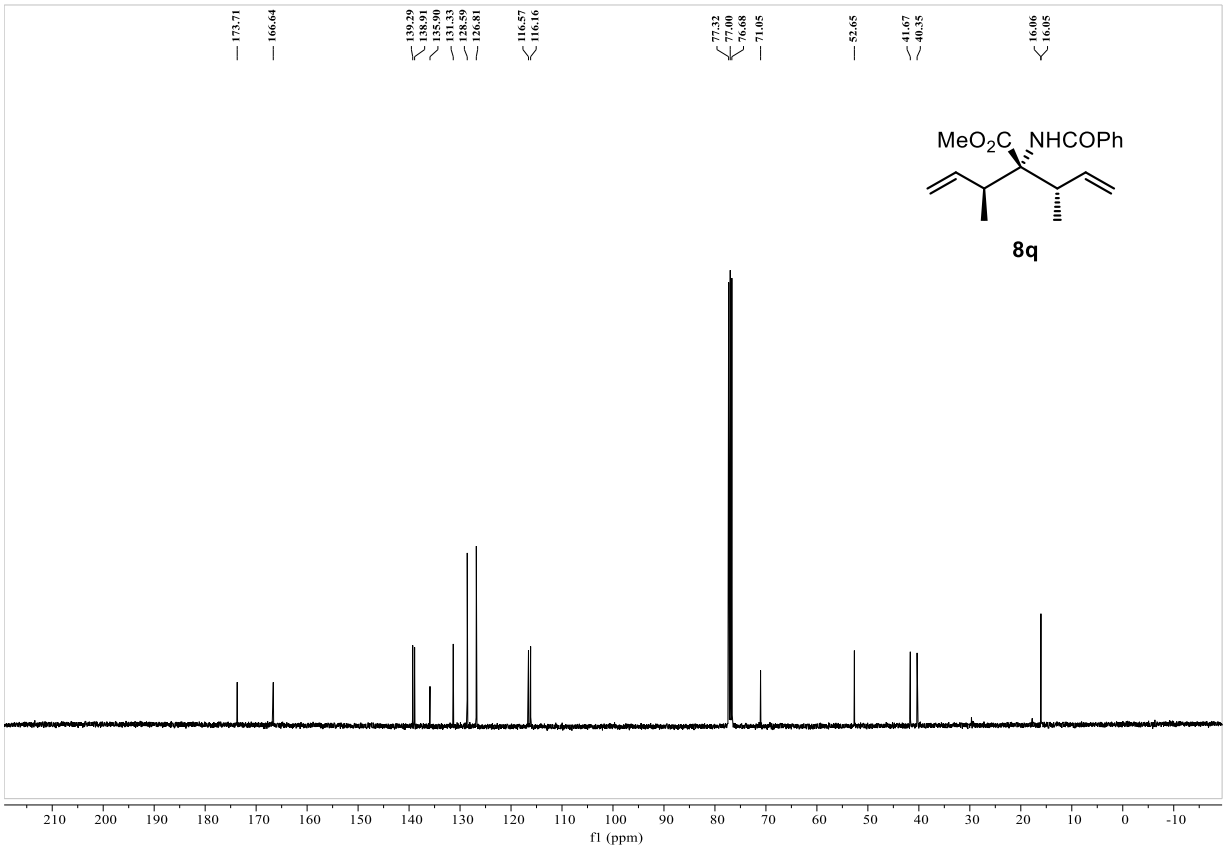
^{1}H NMR (400 MHz, $CDCl_3$) of **3q**



^{13}C NMR (101 MHz, $CDCl_3$) of **3q**



^{1}H NMR (400 MHz, $CDCl_3$) of **8q**

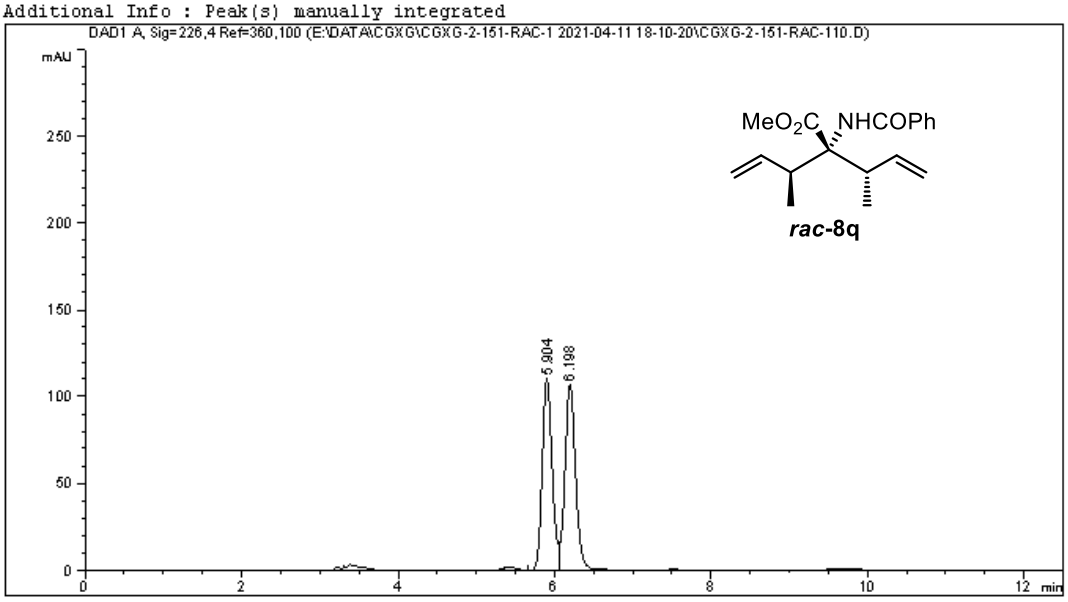


^{13}C NMR (101 MHz, $CDCl_3$) of **8q**

HPLC chromatogram of compound (*rac*)-8q

Data File E:\DATA\CGXG\CGXG-2-151-RAC-1 2021-04-11 18-10-20\CGXG-2-151-RAC-110.D
Sample Name: CGXG-2-151-RAC

```
======================================================================
Acq. Operator   : SYSTEM                       Seq. Line :  11
Acq. Instrument : 1260                          Location :  74
Injection Date  : 4/11/2021 8:34:46 PM              Inj :  1
                                               Inj Volume : 2.000 µl
Acq. Method     : E:\DATA\CGXG\CGXG-2-151-RAC-1 2021-04-11 18-10-20\CGXG-2-ODH-99-1-226NMDAD-
                  1ML-20MIN-3UL.M
Last changed    : 4/11/2021 8:42:56 PM by SYSTEM
                  (modified after loading)
Analysis Method : E:\DATA\CGXG\CGXG-2-151-RAC-1 2021-04-11 18-10-20\CGXG-2-ODH-99-1-226NMDAD-
                  1ML-20MIN-3UL.M (Sequence Method)
Last changed    : 12/30/2021 10:02:36 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=226,4 Ref=360,100

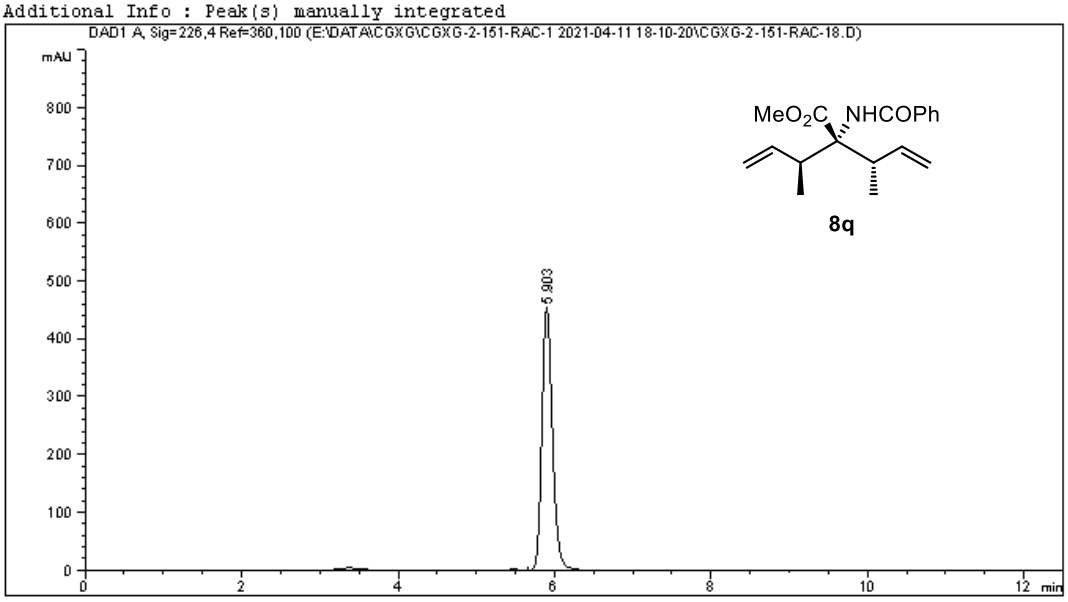
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]        %
----|-------|----|-------|----------|----------|--------|
   1   5.904 BV    0.1350  967.49609  110.62539  48.7660
   2   6.198 VB    0.1448 1016.46075  107.05552  51.2340

Totals :                   1983.95685  217.68091
```

HPLC chromatogram of compound 8q

Data File E:\DATA\CGXG\CGXG-2-151-RAC-1 2021-04-11 18-10-20\CGXG-2-151-RAC-18.D
Sample Name: CGXG-2-151-R

```
==================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   9
Acq. Instrument : 1260                           Location :   72
Injection Date  : 4/11/2021 8:00:01 PM                Inj :   1
                                                Inj Volume : 3.000 µl
Acq. Method     : E:\DATA\CGXG\CGXG-2-151-RAC-1 2021-04-11 18-10-20\CGXG-2-ODH-99-1-226NMDAD-
                  1ML-20MIN-3UL.M
Last changed    : 4/11/2021 8:11:13 PM by SYSTEM
                  (modified after loading)
Analysis Method : E:\DATA\CGXG\CGXG-2-151-RAC-1 2021-04-11 18-10-20\CGXG-2-ODH-99-1-226NMDAD-
                  1ML-20MIN-3UL.M (Sequence Method)
Last changed    : 12/30/2021 10:06:17 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```



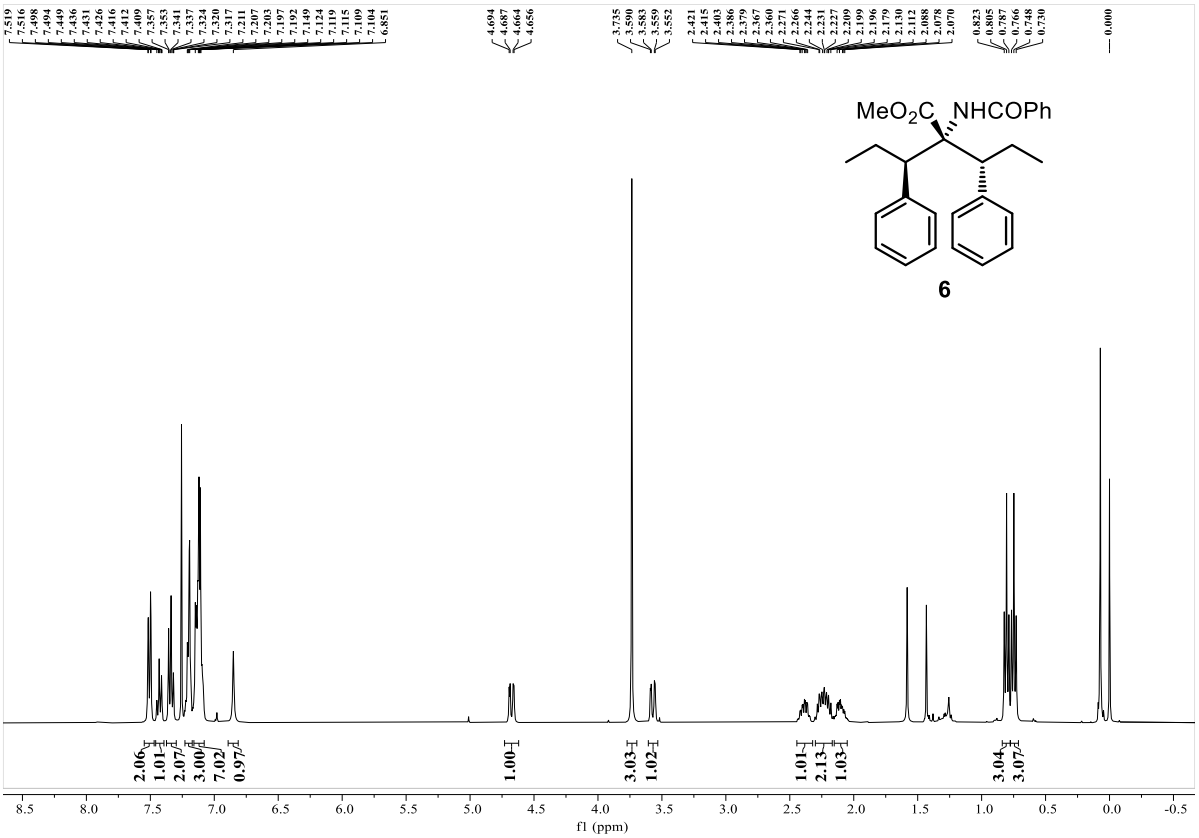

```
==================================================================
                        Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs

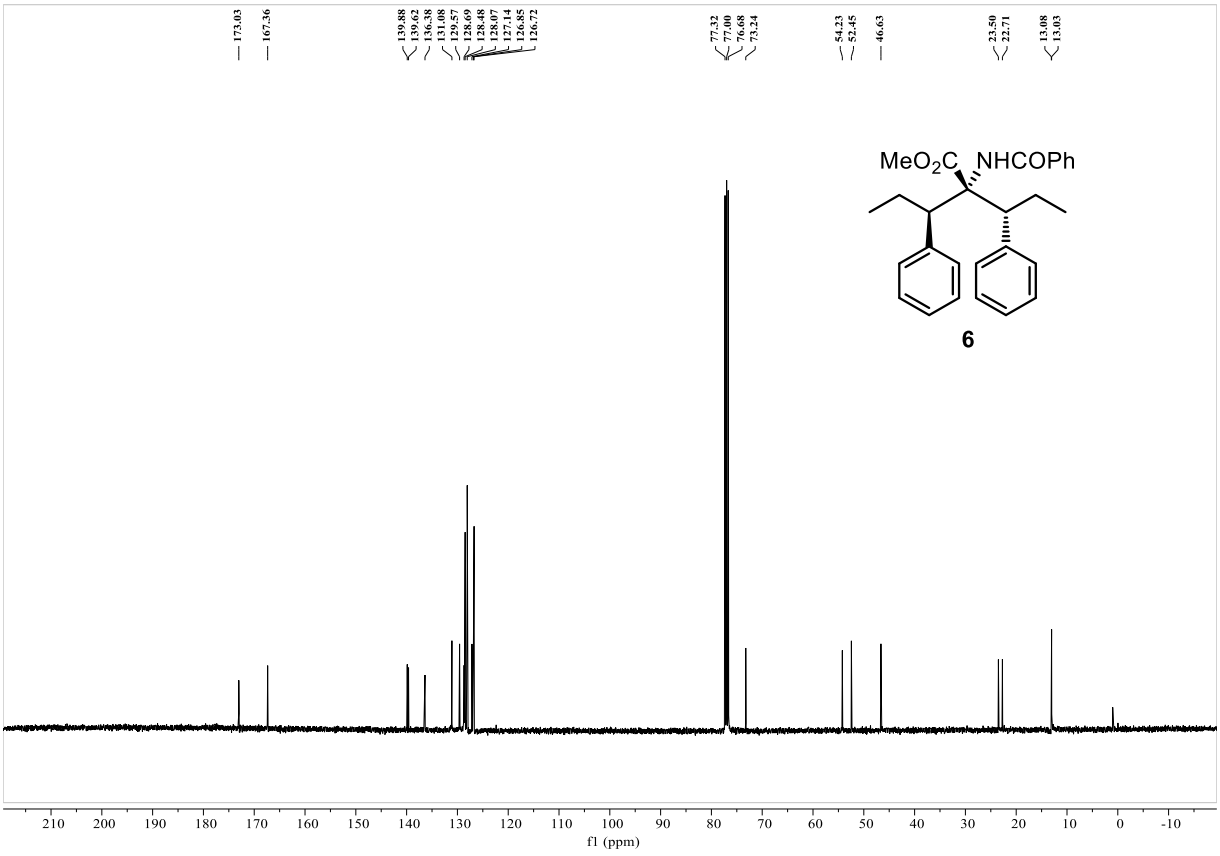

Signal 1: DAD1 A, Sig=226,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]        %
----|-------|----|-------|----------|----------|--------|
   1   5.903 BB    0.1429 4234.09619  453.60251 100.0000

Totals :                  4234.09619  453.60251
```



^{1}H NMR (400 MHz, $CDCl_3$) of **6**

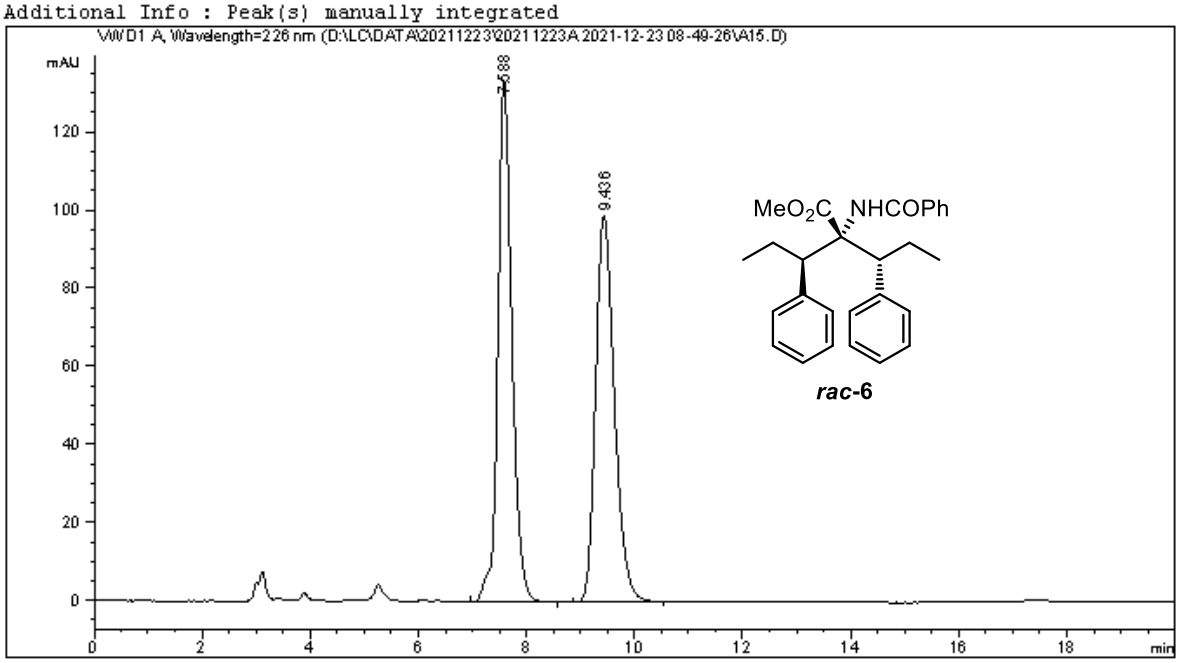


^{13}C NMR (101 MHz, $CDCl_3$) of **6**

HPLC chromatogram of compound (*rac*)-6

Data File D:\LC\DATA\20211223\20211223A 2021-12-23 08-49-26\A15.D
Sample Name: CGXG-4-152RAC

```
==================================================================
Acq. Operator   : 系统                          Seq. Line :  16
Sample Operator : 系统
Acq. Instrument : 1200                           Location :   51
Injection Date  : 12/23/2021 2:46:38 PM               Inj :   1
                                                Inj Volume : 2.000 µl
Acq. Method     : D:\LC\DATA\20211223\20211223A 2021-12-23 08-49-26\CGXG-ADH-90-10-226NM-1ML-
                  30MIN-2UL.M
Last changed    : 12/23/2021 3:14:07 PM by 系统
                  (modified after loading)
Analysis Method : D:\LC\DATA\20211223\20211223A 2021-12-23 08-49-26\CGXG-ADH-90-10-226NM-1ML-
                  30MIN-2UL.M (Sequence Method)
Last changed    : 12/23/2021 5:07:29 PM by 系统
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==================================================================
                        Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs

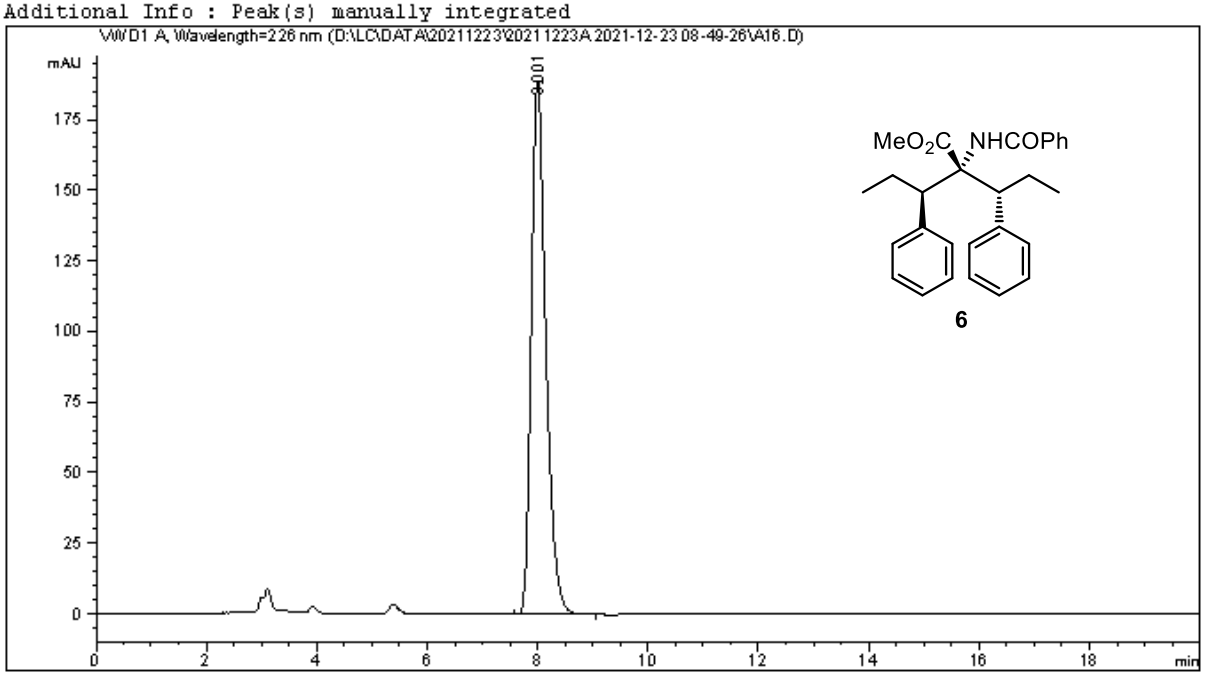

Signal 1: VWD1 A, Wavelength=226 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   7.588 BB    0.2725 2419.72827  133.60234  50.3470
   2   9.436 BB    0.3685 2386.37061   99.01464  49.6530
```

HPLC chromatogram of compound 6

Data File D:\LC\DATA\20211223\20211223A 2021-12-23 08-49-26\A16.D
Sample Name: CGXG-4-152R

```
======================================================================
Acq. Operator   : 系统                          Seq. Line :  17
Sample Operator : 系统
Acq. Instrument : 1200                           Location :   52
Injection Date  : 12/23/2021 3:15:19 PM              Inj :   1
                                                 Inj Volume : 2.000 µl
Acq. Method     : D:\LC\DATA\20211223\20211223A 2021-12-23 08-49-26\CGXG-ADH-90-10-226NM-1ML-
                  30MIN-2UL.M
Last changed    : 12/23/2021 3:15:35 PM by 系统
                  (modified after loading)
Analysis Method : D:\LC\DATA\20211223\20211223A 2021-12-23 08-49-26\CGXG-ADH-90-10-226NM-1ML-
                  30MIN-2UL.M (Sequence Method)
Last changed    : 12/23/2021 5:08:20 PM by 系统
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```



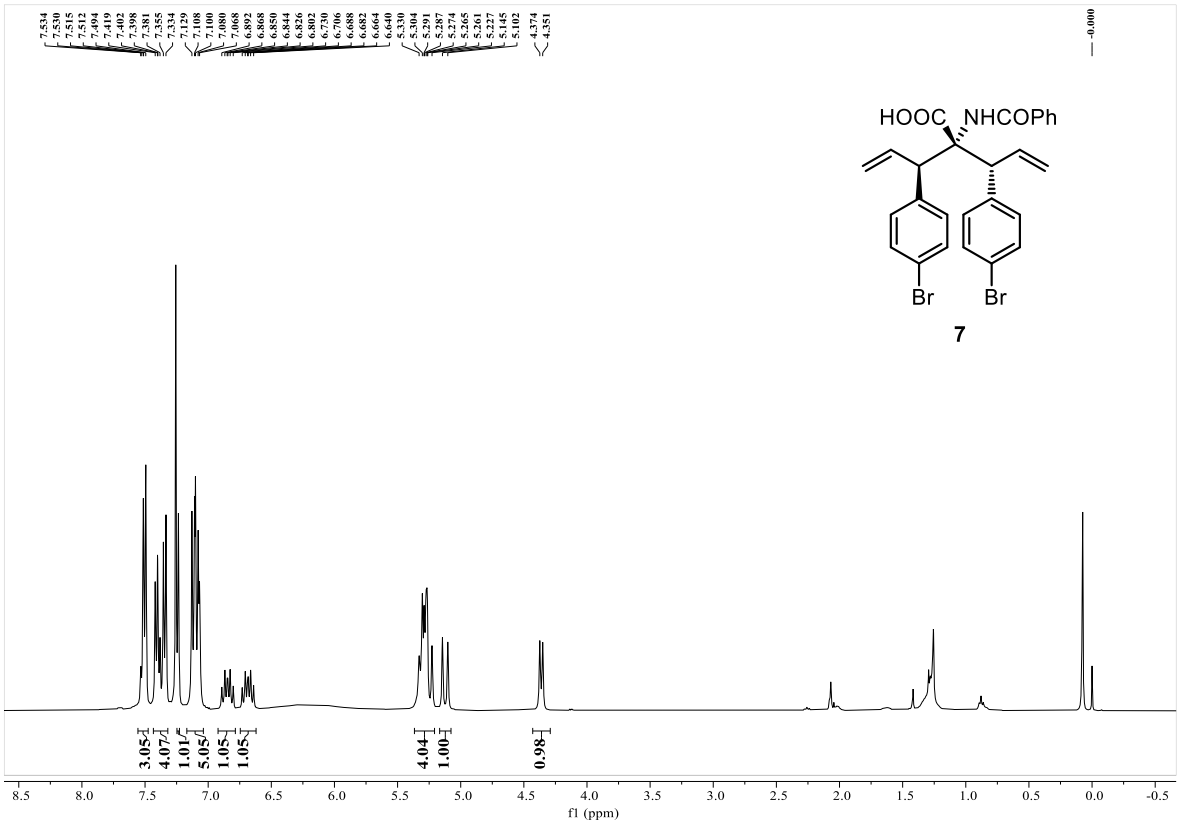

```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs

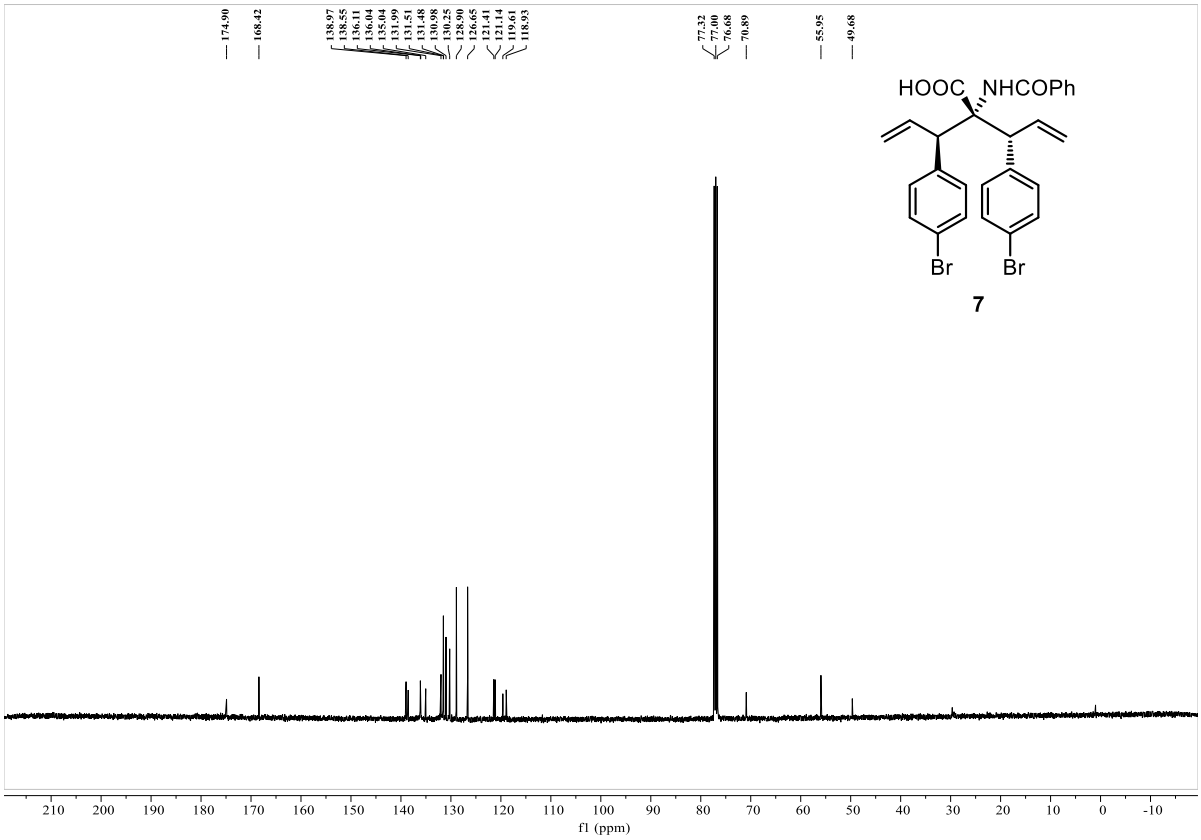

Signal 1: VWD1 A, Wavelength=226 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   8.001 BB    0.2788 3478.61865  189.01076 100.0000

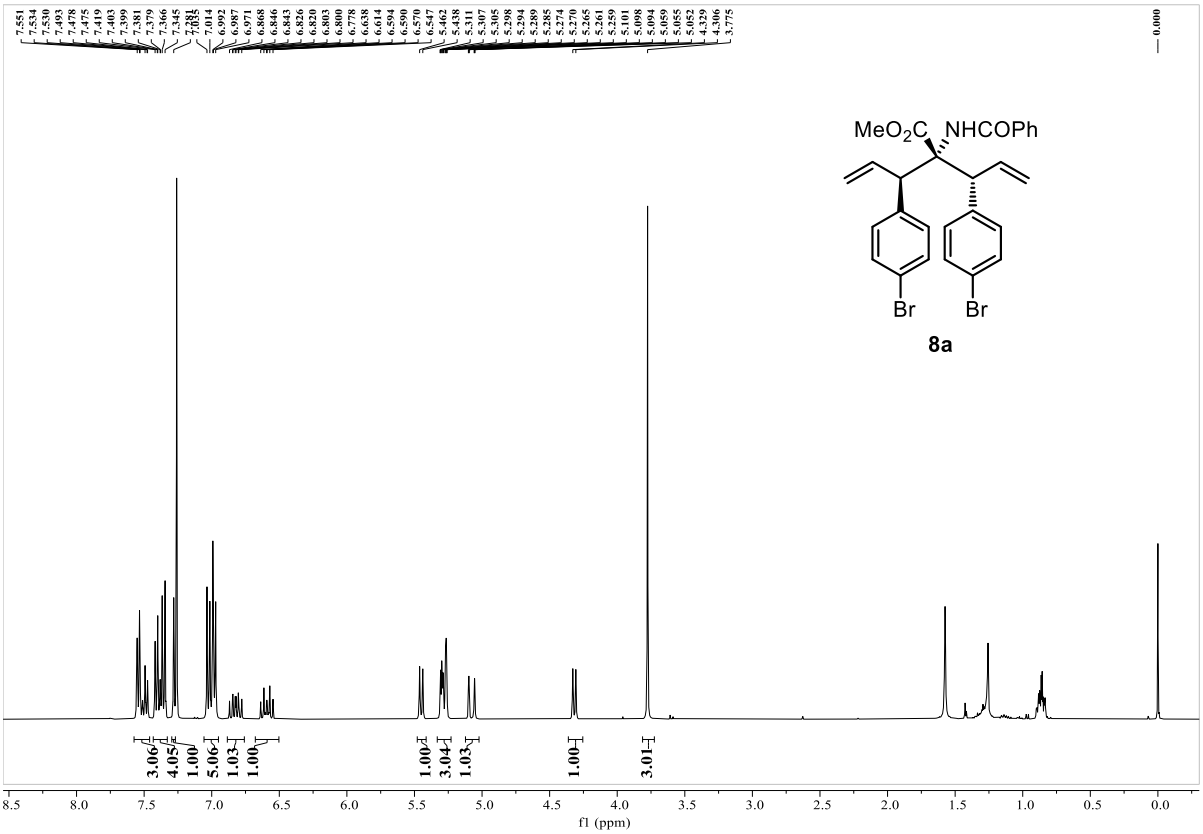
Totals :                  3478.61865  189.01076
```



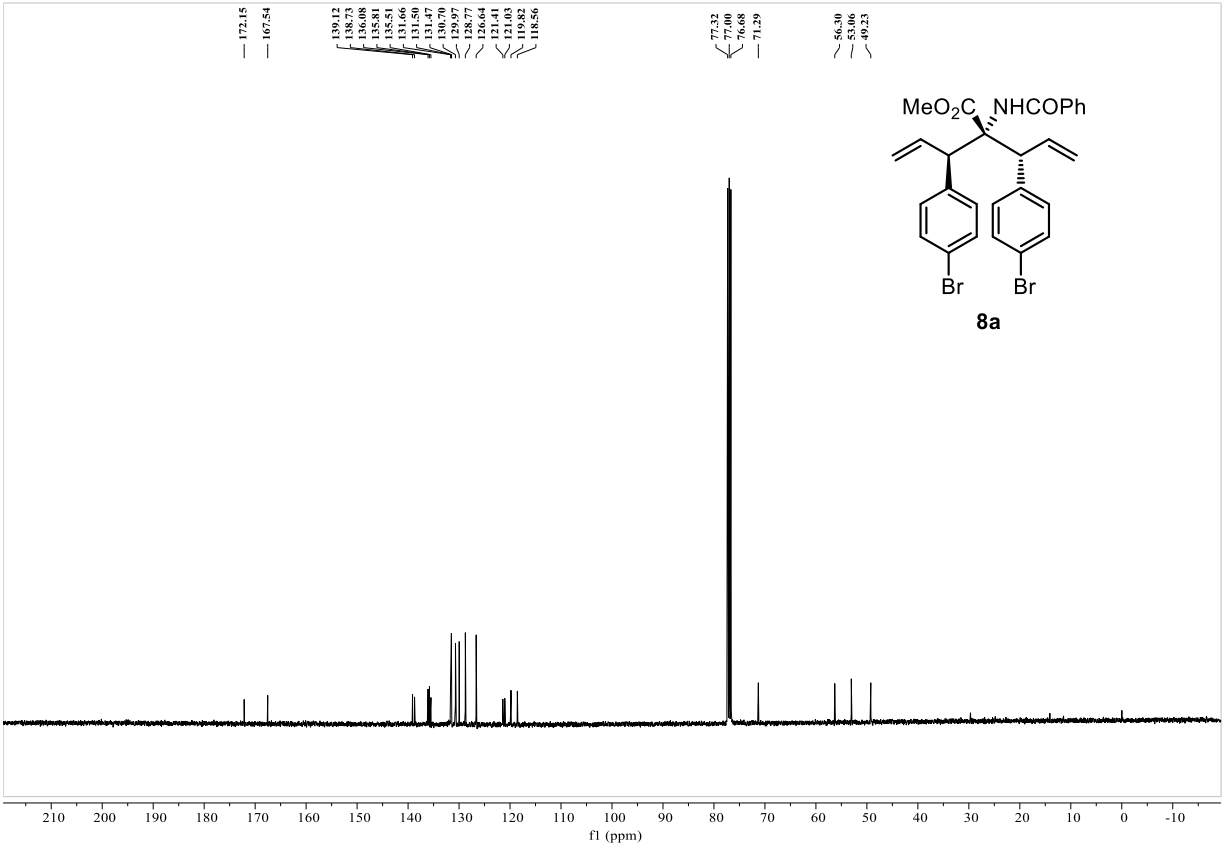
^{1}H NMR (400 MHz, $CDCl_3$) of **7**



^{13}C NMR (101 MHz, $CDCl_3$) of **7**



^{1}H NMR (400 MHz, $CDCl_3$) of **8a**

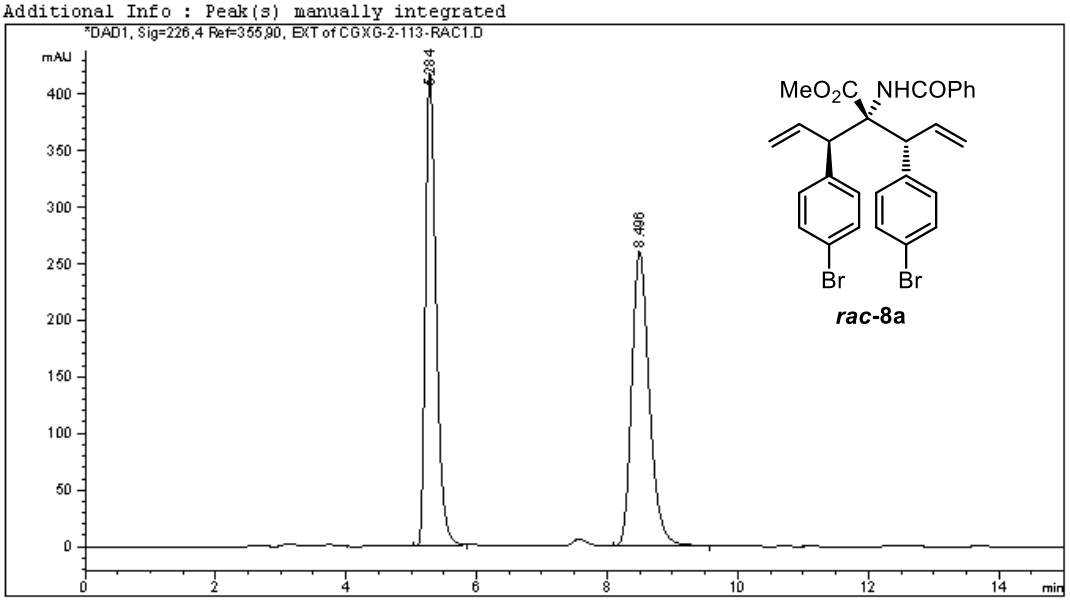


^{13}C NMR (101 MHz, $CDCl_3$) of **8a**

HPLC chromatogram of compound (*rac*)-8a

Data File E:\DATA\CGXG\CGXG-2-113-RAC 2020-11-20 10-48-07\CGXG-2-113-RAC1.D
Sample Name: CGXG-2-113-RAC

```
======================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   2
Acq. Instrument : 1260                           Location :   41
Injection Date  : 11/20/2020 11:00:27 AM             Inj :   1
                                                Inj Volume : 2.000 µl
Acq. Method     : E:\DATA\CGXG\CGXG-2-113-RAC 2020-11-20 10-48-07\CGXG-2-ADH-90-10-DAD-1ML-
                  30ML-2UL.M
Last changed    : 11/20/2020 10:48:07 AM by SYSTEM
Analysis Method : E:\DATA\CGXG\CGXG-2-113-RAC 2020-11-20 10-48-07\CGXG-2-ADH-90-10-DAD-1ML-
                  30ML-2UL.M (Sequence Method)
Last changed    : 6/12/2021 10:25:45 AM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1, Sig=226,4 Ref=355,90, EXT
 Signal has been modified after loading from rawdata file!

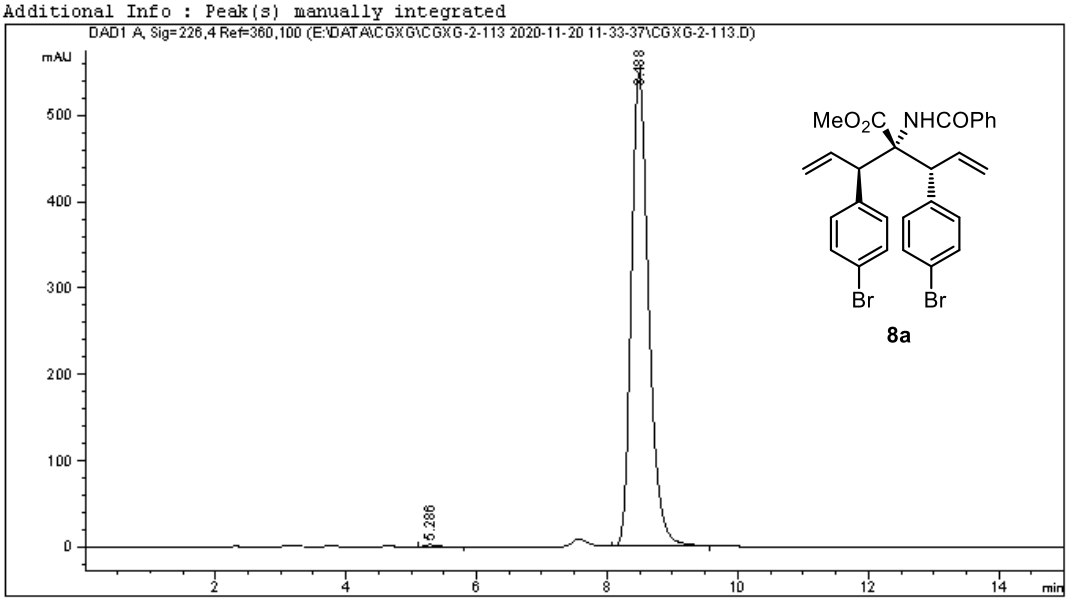
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   5.284 BB    0.1741 4726.63916  417.40366  49.3044
   2   8.496 BB    0.2857 4860.00879  260.43353  50.6956

Totals :                  9586.64795  677.83719
```

HPLC chromatogram of compound 8a

Data File E:\DATA\CGXG\CGXG-2-113 2020-11-20 11-33-37\CGXG-2-113.D
Sample Name: CGXG-2-113-R

```
======================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   1
Acq. Instrument : 1260                           Location :   41
Injection Date  : 11/20/2020 11:35:02 AM             Inj :   1
                                                Inj Volume : 2.000 µl
Acq. Method     : E:\DATA\CGXG\CGXG-2-113 2020-11-20 11-33-37\CGXG-2-ADH-90-10-DAD-226NM-1ML-
                  15ML-2UL.M
Last changed    : 11/20/2020 11:33:37 AM by SYSTEM
Analysis Method : E:\DATA\CGXG\CGXG-2-113 2020-11-20 11-33-37\CGXG-2-ADH-90-10-DAD-226NM-1ML-
                  15ML-2UL.M (Sequence Method)
Last changed    : 6/12/2021 10:27:31 AM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```



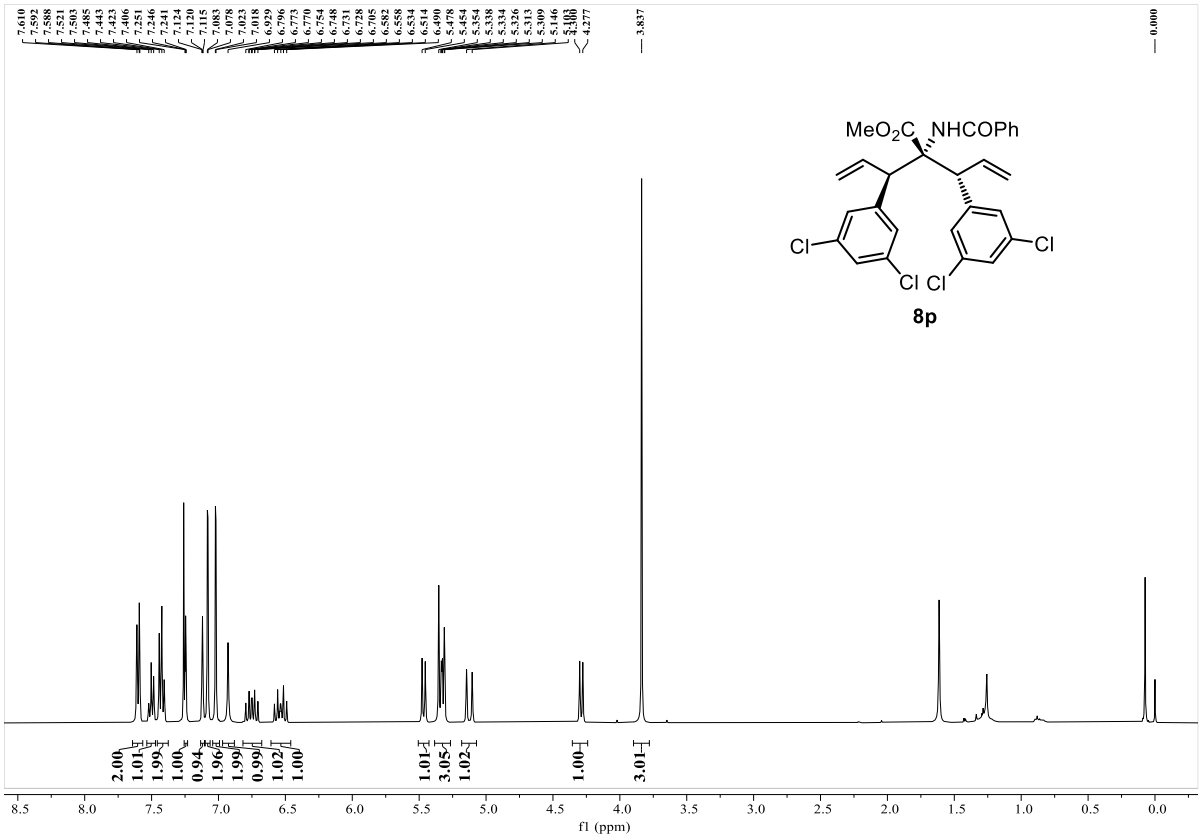

```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs

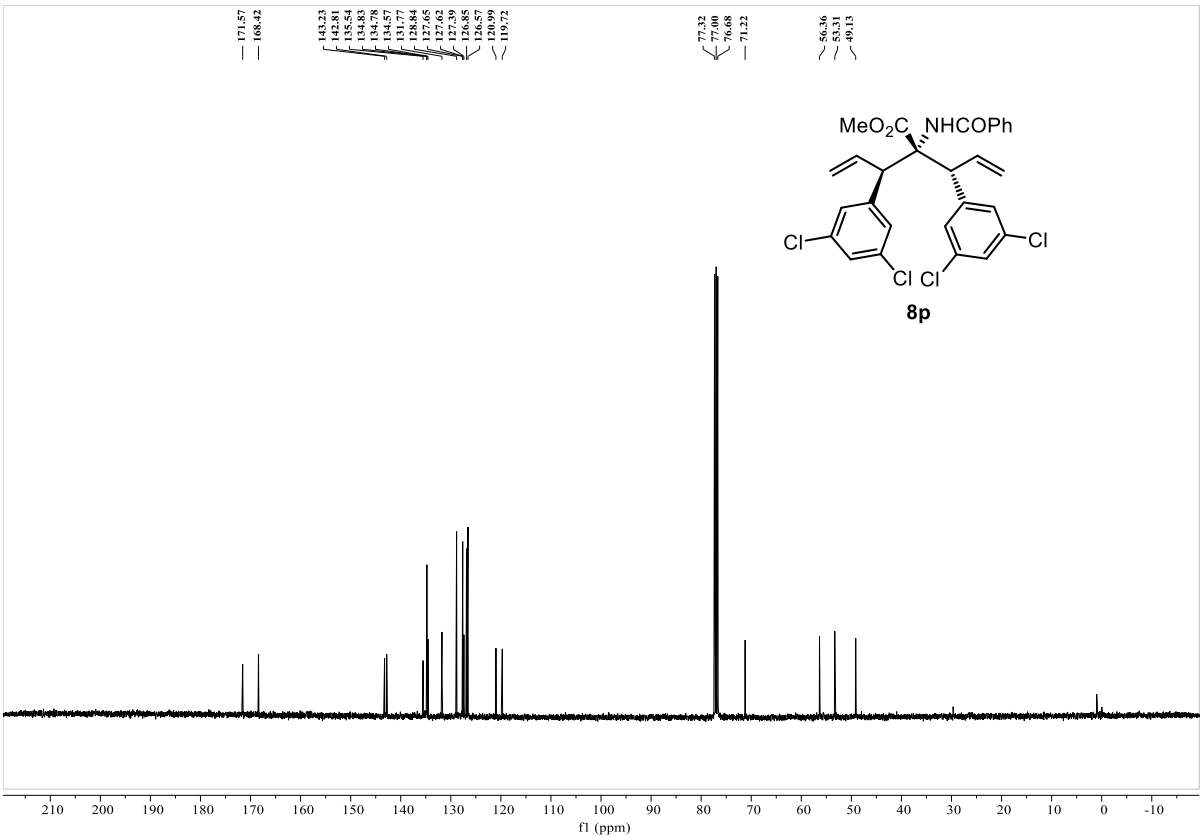

Signal 1: DAD1 A, Sig=226,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   5.286 BB    0.1438   21.78297    1.87201   0.2132
   2   8.488 BB    0.2859 1.01960e4   548.37531  99.7868

Totals :                  1.02178e4   550.24731
```



^{1}H NMR (400 MHz, $CDCl_3$) of **8p**

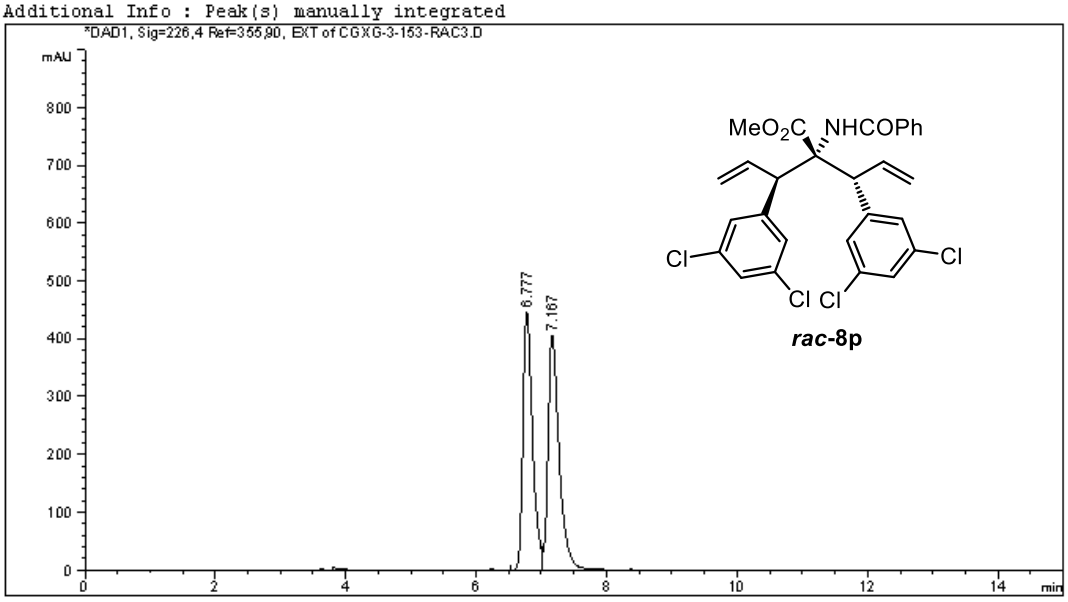


^{13}C NMR (101 MHz, $CDCl_3$) of **8p**

HPLC chromatogram of compound (*rac*)-8p

Data File E:\DATA\CGXG\CGXG-3-153-RAC 2021-07-18 20-21-55\CGXG-3-153-RAC3.D
Sample Name: CGXG-3-153-RAC

```
======================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   4
Acq. Instrument : 1260                           Location :   51
Injection Date  : 7/18/2021 9:25:33 PM                Inj :   1
                                               Inj Volume : 2.000 µl
Acq. Method     : E:\DATA\CGXG\CGXG-3-153-RAC 2021-07-18 20-21-55\CGXG-2-IE-98-2-254DAD-1ML-
                  2UL-30MIN.M
Last changed    : 7/18/2021 9:45:20 PM by SYSTEM
                  (modified after loading)
Analysis Method : E:\DATA\CGXG\CGXG-3-153-RAC 2021-07-18 20-21-55\CGXG-2-IE-98-2-254DAD-1ML-
                  2UL-30MIN.M (Sequence Method)
Last changed    : 12/30/2021 10:23:08 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs

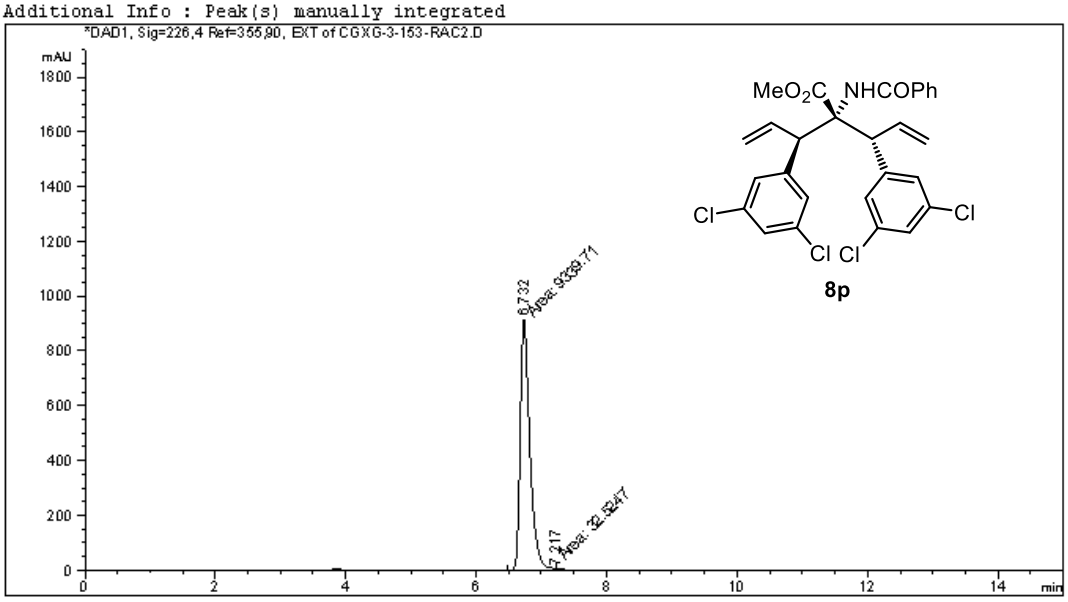

Signal 1: DAD1, Sig=226,4 Ref=355,90, EXT
 Signal has been modified after loading from rawdata file!

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   6.777 BV    0.1539 4547.00439  446.05301  48.7357
   2   7.167 VB    0.1745 4782.91162  405.70493  51.2643
```

HPLC chromatogram of compound 8p

```
Data File E:\DATA\CGXG\CGXG-3-153-RAC 2021-07-18 20-21-55\CGXG-3-153-RAC2.D
Sample Name: CGXG-3-153-R

    ======================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :   3
    Acq. Instrument : 1260                           Location :   51
    Injection Date  : 7/18/2021 9:06:05 PM               Inj :   1
                                                  Inj Volume : 2.000 µl
    Acq. Method     : E:\DATA\CGXG\CGXG-3-153-RAC 2021-07-18 20-21-55\CGXG-2-IE-98-2-254DAD-1ML-
                      2UL-30MIN.M
    Last changed    : 7/18/2021 9:24:07 PM by SYSTEM
                      (modified after loading)
    Analysis Method : E:\DATA\CGXG\CGXG-3-153-RAC 2021-07-18 20-21-55\CGXG-2-IE-98-2-254DAD-1ML-
                      2UL-30MIN.M (Sequence Method)
    Last changed    : 12/30/2021 10:25:46 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```



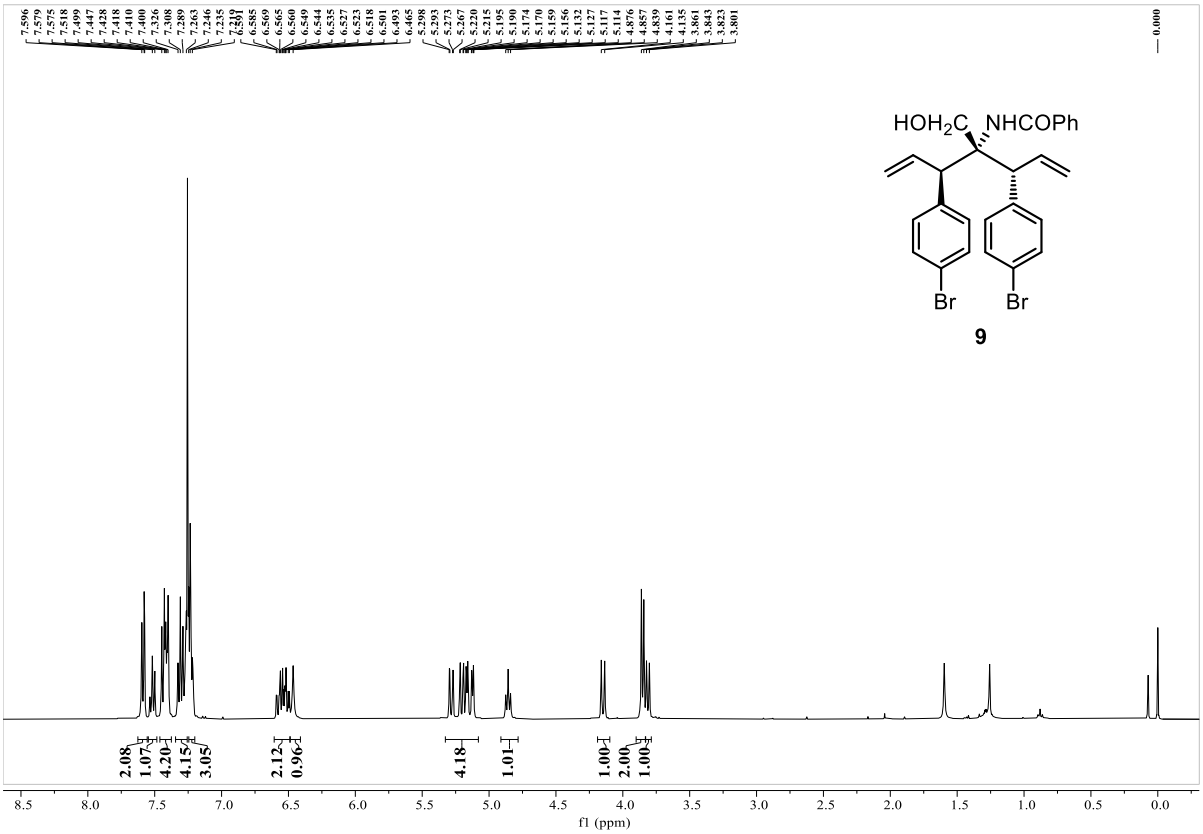

```
    ======================================================================
                              Area Percent Report
    ======================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Do not use Multiplier & Dilution Factor with ISTDs

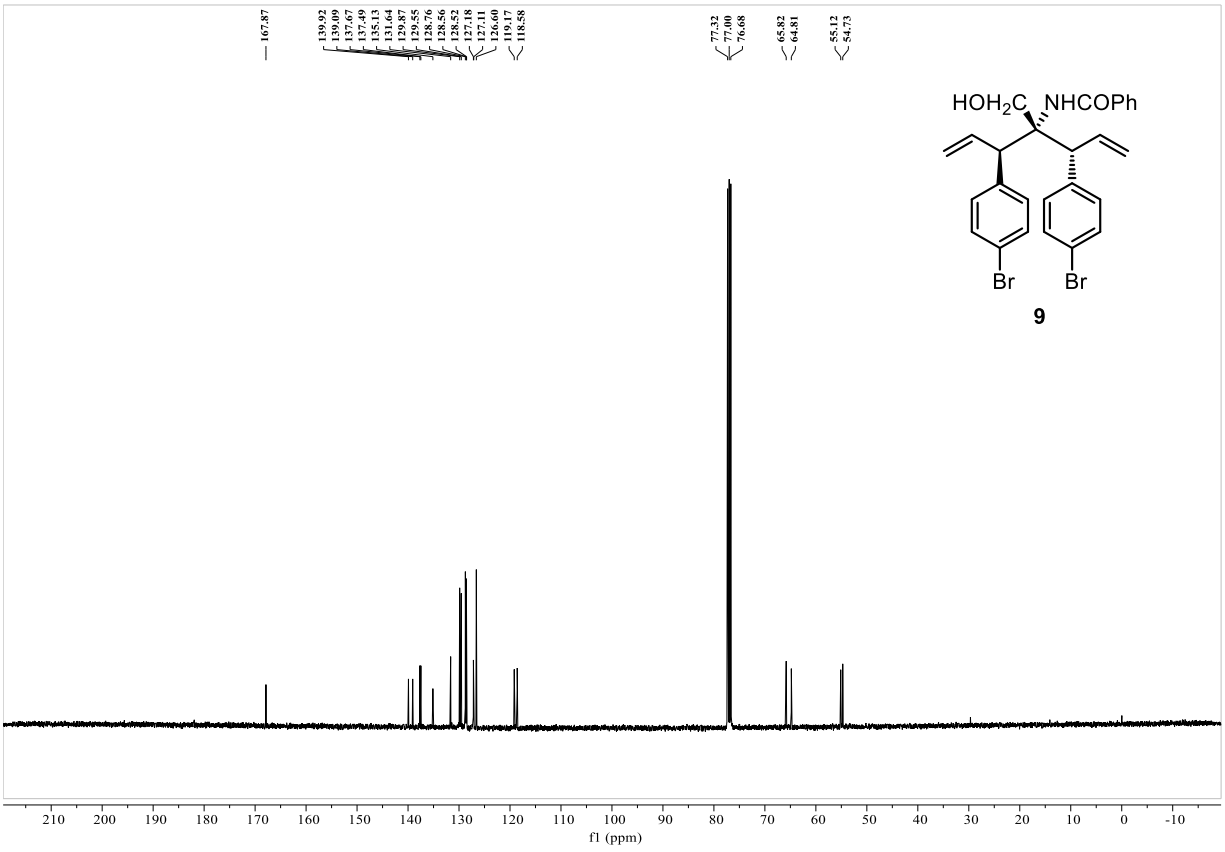

    Signal 1: DAD1, Sig=226,4 Ref=355,90, EXT
     Signal has been modified after loading from rawdata file!

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1   6.732 MF    0.1704 9339.71484  913.69348  99.6530
       2   7.217 FM    0.1202   32.52470    4.51154   0.3470

1260 12/30/2021 10:25:49 PM SYSTEM                          Page 1 of 2
```



^{1}H NMR (400 MHz, $CDCl_3$) of **9**

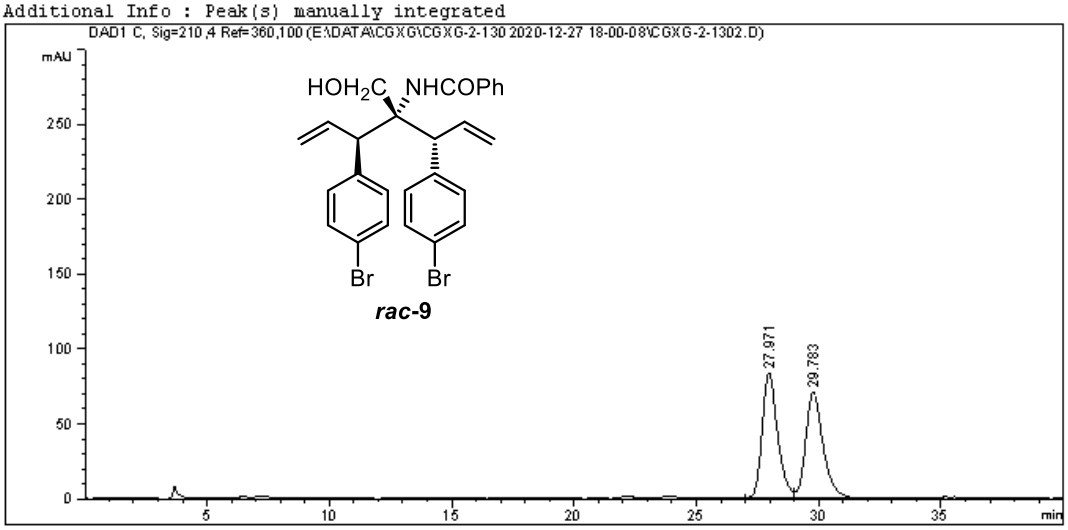


^{13}C NMR (101 MHz, $CDCl_3$) of **9**

HPLC chromatogram of compound (*rac*)-9

Data File E:\DATA\CGXG\CGXG-2-130 2020-12-27 18-00-08\CGXG-2-1302.D
Sample Name: CGXG-2-130-RAC

```
==================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   3
Acq. Instrument : 1260                           Location :   11
Injection Date  : 12/27/2020 7:22:45 PM              Inj :   1
                                                Inj Volume : 2.000 µl
Acq. Method     : E:\DATA\CGXG\CGXG-2-130 2020-12-27 18-00-08\CGXG-2-IE-90-10-DAD-1ML-40MIN-
                  2UL.M
Last changed    : 12/27/2020 6:00:08 PM by SYSTEM
Analysis Method : E:\DATA\CGXG\CGXG-2-130 2020-12-27 18-00-08\CGXG-2-IE-90-10-DAD-1ML-40MIN-
                  2UL.M (Sequence Method)
Last changed    : 12/31/2021 12:30:53 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==================================================================
                        Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=210,4 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  27.971 BV    0.6280 3665.38330   83.52290  51.6449
   2  29.783 VB    0.6694 3431.90039   70.56400  48.3551

Totals :                  7097.28369  154.08690

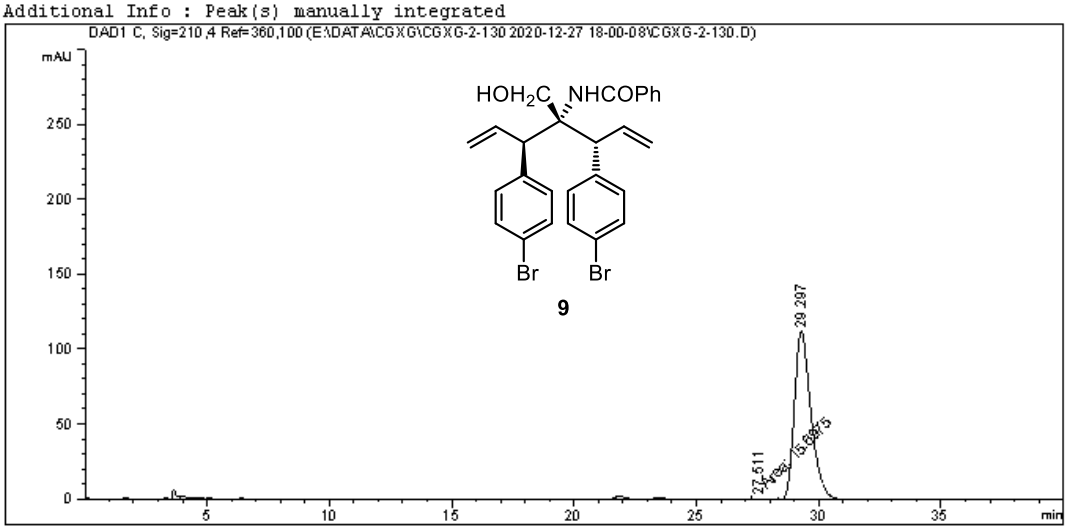

==================================================================
                        *** End of Report ***

1260 12/31/2021 12:31:13 PM SYSTEM                    Page 1 of 1
```

HPLC chromatogram of compound 9

```
Data File E:\DATA\CGXG\CGXG-2-130 2020-12-27 18-00-08\CGXG-2-130.D
Sample Name: CGXG-2-130-R

    ======================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :   1
    Acq. Instrument : 1260                           Location :   12
    Injection Date  : 12/27/2020 6:01:00 PM              Inj :   1
                                                  Inj Volume : 2.000 µl
    Acq. Method     : E:\DATA\CGXG\CGXG-2-130 2020-12-27 18-00-08\CGXG-2-IE-90-10-DAD-1ML-40MIN-
                      2UL.M
    Last changed    : 12/27/2020 6:00:08 PM by SYSTEM
    Analysis Method : E:\DATA\CGXG\CGXG-2-130 2020-12-27 18-00-08\CGXG-2-IE-90-10-DAD-1ML-40MIN-
                      2UL.M (Sequence Method)
    Last changed    : 12/31/2021 12:38:54 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```

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    ======================================================================
                            Area Percent Report
    ======================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Do not use Multiplier & Dilution Factor with ISTDs


    Signal 1: DAD1 C, Sig=210,4 Ref=360,100

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  27.511 MM    0.3533   15.69747 7.40587e-1   0.2900
       2  29.297 BB    0.6907 5397.67090  113.12232  99.7100

    Totals :                  5413.36837  113.86291


    ======================================================================
                            *** End of Report ***

1260 12/31/2021 12:38:59 PM SYSTEM                          Page 1 of 1
```
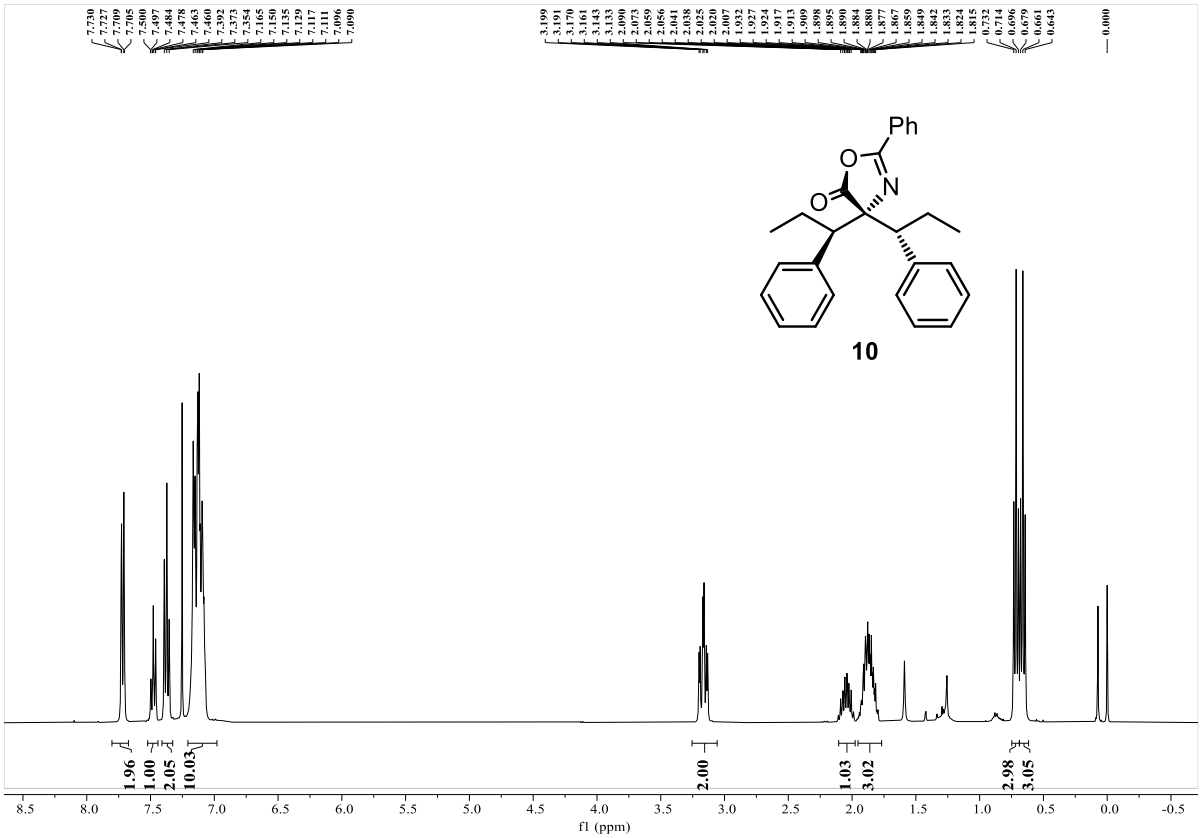
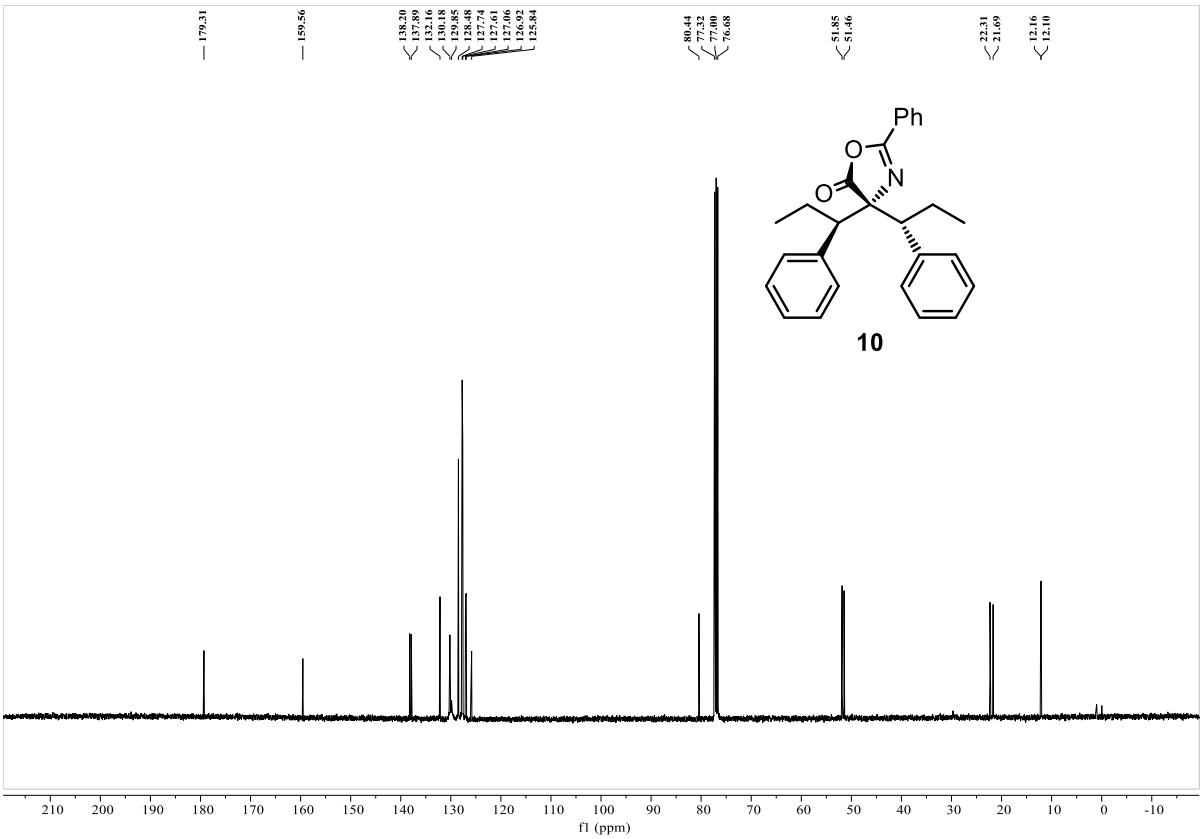



^{1}H NMR (400 MHz, $CDCl_3$) of **10**



^{13}C NMR (101 MHz, $CDCl_3$) of **10**