Supporting information

L-Phenylalanine Potassium Catalyzed Aymmetric Formal [3 + 3] Annulation of 2-enoyl-Pyridine *N*-Oxides with Acetone

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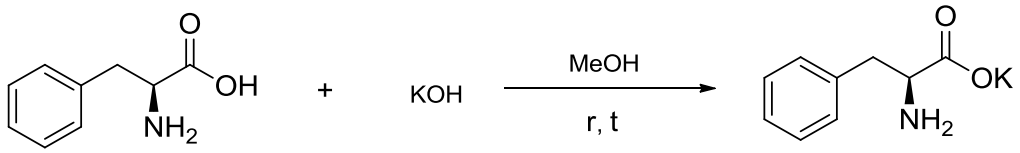
Table of Contents

Part I Experimental Section

1.1 General information

Commercially available compounds and solvents were used without further purification unless otherwise noted. Reactions were monitored by TLC analysis using Merck Silica Gel 60 F-254 thin layer plates. All the chromatographic separations were carried out by using silica gel (Acme's, 100–200 mesh). Optical rotations were measured on a PerKinElmer^{TM} Polarimeter (Model 343). ^{1}H NMR and ^{13}C NMR were recorded on a Bruker-400MHz Spectrometer (^{1}H NMR: 400MHz, ^{13}C NMR: 100MHz) and tetramethylsilane and $CDCl_3$ was used as internal standard for ^{1}H and ^{13}C NMR respectively. The chemical shifts (δ) and coupling constants (*J*) were expressed in ppm and Hz respectively. HPLC analysis was carried out on an Agilent 1100 series HPLC with a multiple wavelength detector. Chiralpak AD-H and IC columns were purchased form Daicel Chemical Industries, LTD. All Substrates was prepared according to the literature procedures.

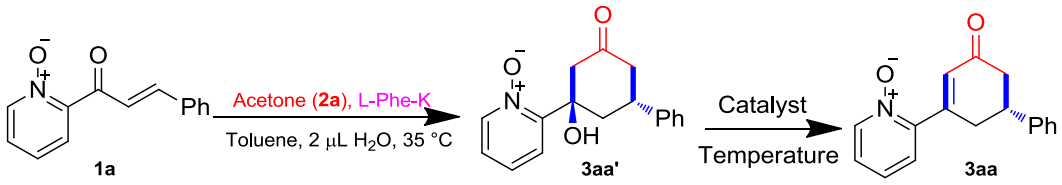
1.2 General procedure for the synthesis of catalyst L-phenylalanine potassium



Potassium hydroxide (20.0 mmol, 1.122 g) was dissolved in methanol (30 mL) in room temperature. L-phenylalanine (20.0 mmol, 3.304 g) were added to the solution and stirred for 30 min. The resulting mixture was filtered and concentrated in vacuo, to provide the catalyst L-phenylalanine potassium.

1.3 Optimization of reaction conditions

Table 1. Optimization of the dehydration process of the asymmetric formal [3 + 3] annulation[a]

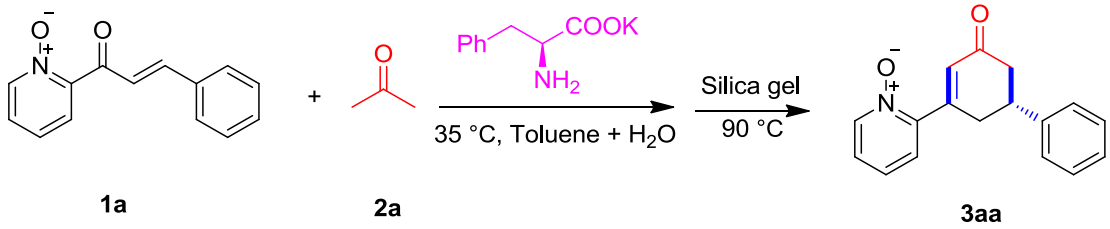


Entry	Catalyst	Temperature(˚C)	Yield(%)[b]	*ee*(%)[c]
1	**Silica gel**	90	82	86
2	**98% Sulfuric acid**	0	80	85
3	**Thionyl chloride + Pyridine**	0-r.t	78	86

[a] The dehydration process of asymmetric formal [3 + 3] annulations were conducted directly after the Michael addition and aldol condensation process of the reaction was completed (monitored by TLC). [b] Isolated yield. [c] The *ee* value was determined by chiral HPLC analysis (Chiralpak IC column).

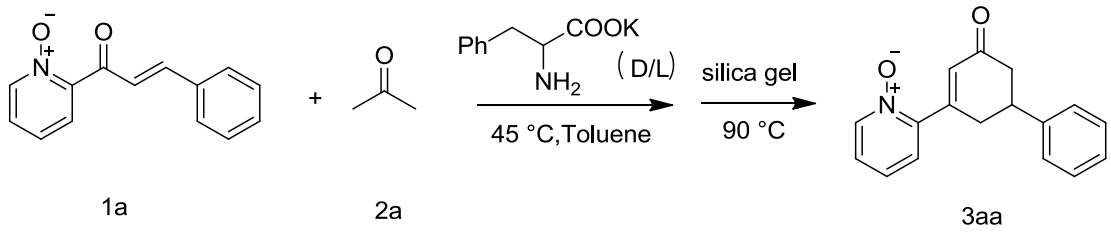
1.4 General working procedure for the asymmetric formal [3 + 3] annulation

a) Asymmetric formal [3 + 3] annulation catalyzed by L-phenylalanine potassium (3aa as an example)



A mixture of 2-alkenoyl pyridine *N*-oxide **1a** (67.5 mg, 0.3 mmol) and the L-phenylalanine potassium (12.3 mg, 0.06 mmol) was added in toluene (1.2 mL). Acetone **2a** (0.3 mL) and 5 μL water were then added and the resulting mixture was stirred at 35 ˚C. After the reaction was completed (monitored by TLC), the acetone was removed under vacuum. Silica gel (540 mg) was then added and the resulting mixture was stirred at 90 ˚C. After the reaction was completed (monitored by TLC) the resulting mixture was purified by silica gel column chromatography with methanol / ethyl acetate (1:100) as the eluent affording the product **3aa** as a yellow solid (65.1 mg, 82% yield, 86% *ee*). The enantiomeric excess of the product was determined by chiral HPLC analysis (Chiralpak IC column).

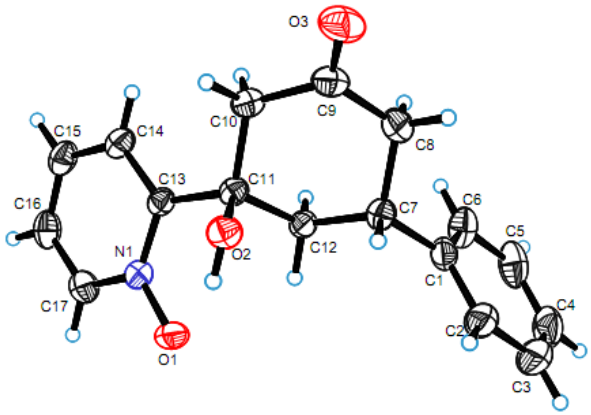
b) Preparation of the racemic product (3aa as an example)



2-Alkenoyl pyridine *N*-oxide **1a** (22.5 mg, 0.1 mmol) and (D/L)-phenylalanine potassium (4.1 mg) was added in toluene (0.4 mL). Acetone **2a** (0.1 mL) were then added and the mixture was stirred at 45 ˚C. After the reaction was completed (monitored by TLC), silica gel (180 mg) was then added and the resulting mixture was stirred at 90 ˚C. After the reaction was completed (monitored by TLC), the desired racemic product **3aa** was isolated by flash column chromatography.

1.5 Determination of absolute configurations of the product 3aa'

X-ray crystal structure of 3aa'

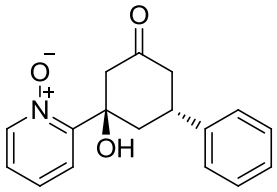


X-ray crystal structure analysis of 3aa':

Chemical formula	$C_{16}H_{16}N_2O_5$
Formula weight	283.31
Space group	$P2_12_12_1$
Z	4
a, Å	6.53370(10)
b, Å	8.45090(10)
c, Å	26.0879(3)
α, °	90
β, °	90
γ, °	90
V, Å^3	1440.46(3)
T,K	289(2)
ρ, g/cm^3	1.306
Goodness of fit	1.068

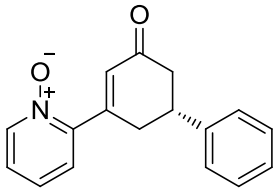
Reflections collected / unique: 12842 /2674 (R_{int} = 0.0217), number of observations [I > 2 σ (I)] 2618, parameters 192. Final R indexes [I >= 2 σ (I)]: R_1 = 0.0324, wR_2 = 0.0876; Final R indexes (all data): R_1 = 0.0349, wR_2 = 0.0987, Flack parameter = 0.01(6).

1.6 Experimental data of asymmetric formal [3 + 3] annulation products



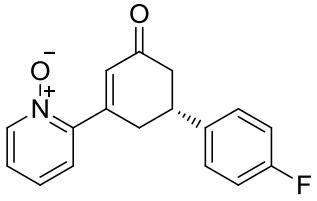
3aa'

2-((1R,5R)-1-hydroxy-3-oxo-5-phenylcyclohexyl)pyridine 1-oxide: yellow solid in 70% yield. mp 145-147 ˚C; $[\alpha]_D^{20}$ 2.72 (c = 0.565, $CHCl_3$); HPLC: Daicel Chiralpak AD-H, hexane: 2-propanol = 75:25, flow rate = 1.0 mL/min, T = 30 ˚C, UV = 215 nm, t_R = 15.6 min (minor), t_R= 17.2 min (major); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.18-8.16 (d, *J* = 6.2 Hz, 1H), 7.35-7.15 (m, 8H), 3.71-3.65 (m, 1H), 3.18-3.14 (d, *J* = 13.5 Hz, 1H), 2.74-2.65 (m, 3H), 2.55-2.48 (m, 1H), 2.23-2.16 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 207.0, 151.9, 143.3, 140.8, 128.7, 128.2, 126.8, 125.0, 122.5, 75.4, 49.0, 48.0, 40.9, 37.9; IR (film, ν/cm^{-1}): 3292, 3053, 2929, 1707, 1599, 1493, 1457, 1399, 759, 705; HRMS (ESI) m/z calcd for $C_{17}H_{17}NO_3$ [M+ Na]$^+$ 306.1106, found 306.1111.



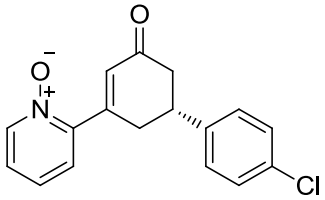
3aa

(R)-2-(5-oxo-1,2,5,6-tetrahydro-[1,1'-biphenyl]-3-yl)pyridine 1-oxide: yellow solid in 82% yield. mp 127-128 ˚C; $[\alpha]_D^{20}$ 0.413 (c = 0.990, $CHCl_3$); HPLC: Daicel Chiralpak IC, ethanol: hexane = 90:10, flow rate = 0.45 mL/min, T = 30 ˚C, UV = 231 nm, t_R = 22.7 min (minor), t_R= 27.6 min (major); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.17-8.15 (d, *J* = 4.7 Hz, 1H), 7.28-7.15 (m, 8H), 6.27 (s, 1H), 3.48-3.40 (m, 1H), 3.10-3.00 (m, 2H), 2.78-2.67 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 197.8, 153.6, 147.9, 141.7, 139.4, 129.0, 127.7, 126.0, 125.8, 125.0, 124.9, 124.8, 43.6, 40.1, 33.8; IR (film, ν/cm^{-1}): 3051, 2949, 2884, 2360, 1663, 1606, 1493, 1455, 1375, 734, 701; HRMS (ESI) m/z calcd for $C_{17}H_{15}NO_2$ [M+ Na]$^+$ 288.1000, found 288.1003.



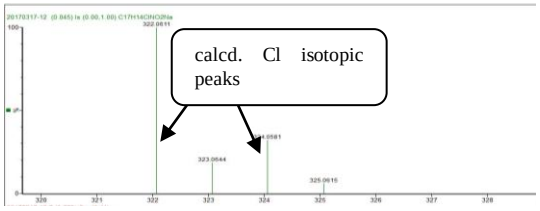
3ba

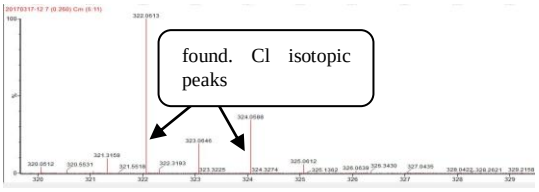
(R)-2-(4'-fluoro-5-oxo-1,2,5,6-tetrahydro-[1,1'-biphenyl]-3-yl)pyridine 1-oxide: yellow oil in 89% yield. $[\alpha]_D^{20}$ 0.0817 (c = 1.04, $CHCl_3$); HPLC: Daicel Chiralpak IC, ethanol = 100%, flow rate = 0.37 mL/min, T = 30 ˚C, UV = 231 nm, t_R = 22.8 min (minor), t_R= 24.0 min (major); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.25-8.24 (d, *J* = 3.7 Hz, 1H), 7.32-7.25 (m, 5H), 7.04-7.00 (m, 2H), 6.33 (s, 1H), 3.55-3.47 (m, 1H), 3.18-3.05 (m, 2H), 2.83-2.70 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 197.5, 161.9-159.5(d, *J* = 243.7 Hz), 153.5, 147.8, 139.4, 137.5 (d, *J* = 3.2 Hz), 129.0, 127.3(d, *J* = 7.9 Hz), 125.1, 125.0, 125.0, 114.6-114.4(d, *J* = 21.1 Hz), 43.8, 39.4, 33.8; IR (film, ν/cm^{-1}): 3053, 2922, 2361, 1666, 1604, 1508, 1368, 820; HRMS (ESI) m/z calcd for $C_{17}H_{14}FNO_2$ $[M+ Na]^+$ 306.0906, found 306.0913.

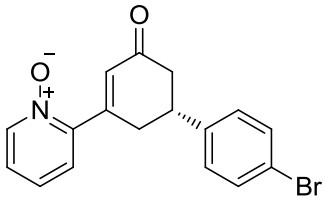


3ca

(R)-2-(4'-chloro-5-oxo-1,2,5,6-tetrahydro-[1,1'-biphenyl]-3-yl)pyridine 1-oxide: yellow oil in 87% yield. $[\alpha]_D^{20}$ 1.37 (c = 0.925, $CHCl_3$); HPLC: Daicel Chiralpak IC, ethanol: hexane = 90:10, flow rate = 0.45 mL/min, T = 30 ˚C, UV = 240 nm, t_R = 22.2 min (minor), t_R= 23.4 min (major); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.16 (s 1H), 7.24-7.14 (m, 7H), 6.25 (s, 1H), 3.46-3.38 (m, 1H), 3.10-2.97 (m, 2H), 2.75-2.61 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 197.3, 153.4, 147.8, 140.2, 139.4, 131.7, 129.0, 127.8, 127.2, 125.1, 125.0, 125.0, 43.5, 39.4, 33.5; IR (film, ν/cm^{-1}): 3050, 2923, 2360, 1664, 1491, 1366, 827; HRMS (ESI) m/z calcd for $C_{17}H_{14}ClNO_2$ $[M+ Na]^+$ 322.0611, 324.0581, found 322.0613, 324.0588.



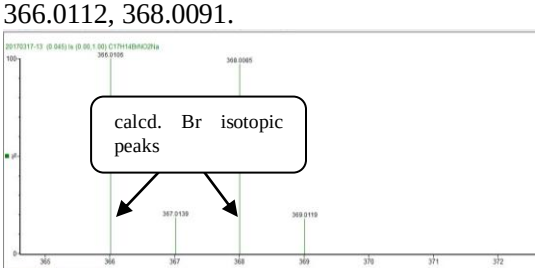


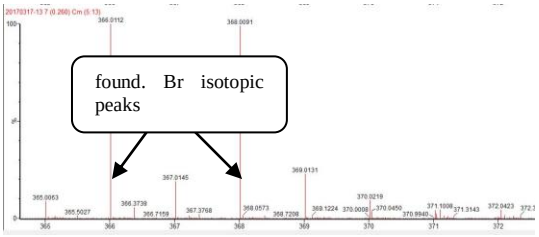


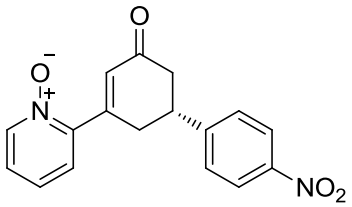
3da

(R)-2-(4'-bromo-5-oxo-1,2,5,6-tetrahydro-[1,1'-biphenyl]-3-yl)pyridine 1-oxide: white oil in 96% yield. $[\alpha]_D^{20}$ 1.47 (c = 0.980, $CHCl_3$); HPLC: Daicel Chiralpak IC, ethanol = 100%, flow rate = 0.37 mL/min, T = 30 ˚C, UV = 240 nm, t_R = 29.4 min (minor), t_R= 31.1 min (major); ^{1}H NMR

(400 MHz, $CDCl_3$): δ 8.18-8.17 (d, *J* = 4.2 Hz 1H), 7.37-7.35 (d, *J* = 8.2 Hz 2H), 7.25 (s, 3H), 7.10-7.08 (d, *J* = 8.2 Hz 1H), 6.24 (s, 1H), 3.44-3.36 (m, 1H), 3.08-2.95 (m, 2H), 2.73-2.61 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 197.3, 153.4, 147.7, 140.7, 139.4, 130.8, 129.0, 127.6, 125.3, 125.2, 125.1, 119.7, 43.4, 39.5, 33.4; IR (film, ν/cm^{-1}): 3077, 2921, 2853, 2361, 1666, 1488, 1431, 1365, 822; HRMS (ESI) m/z calcd for $C_{17}H_{14}BrNO_2$ $[M+ Na]^+$ 366.0106, 368.0085, found 366.0112, 368.0091.

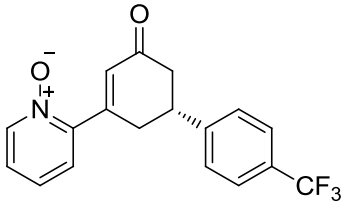






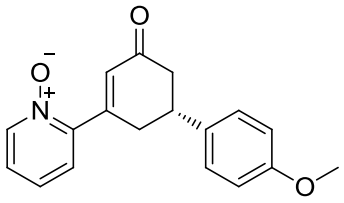
3ea

(R)-2-(4'-nitro-5-oxo-1,2,5,6-tetrahydro-[1,1'-biphenyl]-3-yl)pyridine 1-oxide: yellow oil in 39% yield. $[\alpha]_D^{20}$ 1.77 (c = 0.985, $CHCl_3$); HPLC: Daicel Chiralpak AD-H, hexane: 2-propanol = 75:25, flow rate = 1.0 mL/min, T = 30 ˚C, UV = 240 nm, t_R = 52.9 min (major), t_R= 58.3 min (minor); ^1H NMR (400 MHz, $CDCl_3$): δ 8.19-8.13 (m, 3H), 7.43-7.41 (d, *J* = 8.4 Hz, 2H), 7.27-7.21 (m, 3H), 6.27 (s, 1H), 3.63-3.56 (m, 1H), 3.19-3.05 (m, 2H), 2.81-2.68 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 196.4, 153.1, 149.0, 147.6, 146.0, 139.4, 129.1, 126.8, 125.3, 125.2, 125.1, 123.0, 43.1, 39.8, 32.9; IR (film, ν/cm^{-1}): 3047, 2962, 2921, 2360, 1667, 1597, 1515, 1484, 1343, 856; HRMS (ESI) m/z calcd for $C_{17}H_{14}N_2O_4$ $[M+ H]^+$ 311.1032, found 311.1032.



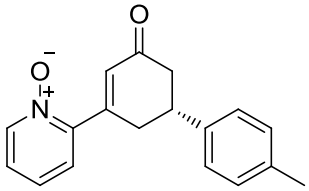
3fa

(R)-2-(5-oxo-4'-(trifluoromethyl)-1,2,5,6-tetrahydro-[1,1'-biphenyl]-3-yl)pyridine 1-oxide： yellow oil in 68% yield. $[\alpha]_D^{20}$ 0.550 (c = 0.917, $CHCl_3$); HPLC: Daicel Chiralpak IC, ethanol = 100%, flow rate = 0.3 mL/min, T = 30 ˚C, UV = 240 nm, t_R = 23.9 min (minor), t_R= 24.8 min (major); ^1H NMR (400 MHz, $CDCl_3$): δ 8.18 (s, 1H), 7.53-7.51 (d, *J* = 8.0 Hz, 2H), 7.36-7.34 (d, *J* = 8.0 Hz, 2H), 7.26-7.20 (m, 3H), 6.26 (s, 1H), 3.55-3.49 (m, 1H), 3.14-3.02 (m, 2H), 2.79-2.67 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 197.0, 153.3, 147.7, 145.6, 139.4, 129.1, 128.5-128.2 (d, *J* = 32.4 Hz), 126.3, 125.2 (d, *J* = 2.7 Hz), 125.1, 124.7 (d, *J* = 3.6 Hz), 124.4-121.7(d, *J* = 270.4 Hz), 43.3, 39.8, 33.2; IR (film, ν/cm^{-1}):3052, 2921, 2360, 1670, 1618, 1485, 835; HRMS (ESI) m/z calcd for $C_{18}H_{14}F_3NO_2$ $[M+ H]^+$ 334.1055, found 334.1056.



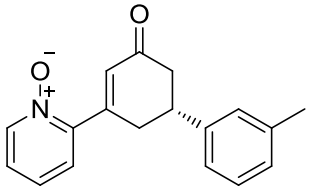
3ga

(R)-2-(4'-methoxy-5-oxo-1,2,5,6-tetrahydro-[1,1'-biphenyl]-3-yl)pyridine 1-oxide: yellow oil in 79% yield. $[\alpha]_D^{20}$ 1.44 (c = 1.16, $CHCl_3$); HPLC: Daicel Chiralpak AD-H, hexane: 2-propanol = 75:25, flow rate = 1.0 mL/min, T = 30 ˚C, UV = 240 nm, t_R = 29.7 min (major), t_R= 34.3 min (minor); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.26-8.24 (d, *J* = 5.0 Hz, 1H), 7.35-7.29 (m, 3H), 7.22-7.20 (d, *J* = 8.4 Hz, 2H), 6.88-6.86 (d, *J* = 8.4 Hz, 2H), 6.33 (s, 1H), 3.79 (s, 3H), 3.51-3.47 (m, 1H), 3.15-3.03 (m, 2H), 2.83-2.71 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 198.1, 157.5, 153.7, 148.0, 139.4, 133.9, 129.0, 126.8, 125.1, 125.0, 113.0, 54.3, 43.9, 39.3, 34.0; IR (film, ν/cm^{-1}): 3072, 2953, 2835, 2360, 1665, 1610, 1584, 1512, 1463, 1367, 1244, 1178, 829; HRMS (ESI) m/z calcd for $C_{18}H_{17}NO_3$ $[M+ Na]^+$ 318.1106, found 318.1108.



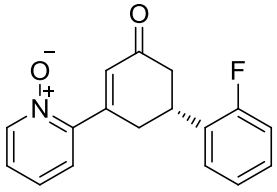
3ha

(R)-2-(4'-methyl-5-oxo-1,2,5,6-tetrahydro-[1,1'-biphenyl]-3-yl)pyridine 1-oxide: yellow solid in 99% yield. mp 108-109 ˚C; $[\alpha]_D^{20}$ 1.33 (c = 1.05, $CHCl_3$); HPLC: Daicel Chiralpak IC, ethanol = 100%, flow rate = 0.37 mL/min, T = 30 ˚C, UV = 231 nm, t_R = 28.2 min (minor), t_R= 31.8 min (major); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.25-8.24 (d, *J* = 4.2 Hz, 1H), 7.31 (m, 3H), 7.19-7.18 (d, *J* = 7.7 Hz, 2H), 7.15-7.13 (d, *J* = 7.6 Hz, 2H), 6.34 (s, 1H), 3.52-3.44 (m, 1H), 3.15-3.05 (m, 2H), 2.84-2.72 (m, 2H), 2.33 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 198.0, 153.7, 148.0, 139.4, 138.7, 135.6, 129.0, 128.3, 125.6, 125.0, 124.8, 43.8, 39.7, 33.8, 20.0; IR (film, ν/cm^{-1}): 3050, 3016, 2920, 2360, 1661, 1614, 1514, 1431, 1366, 856; HRMS (ESI) m/z calcd for $C_{18}H_{17}NO_2$ $[M+ Na]^+$ 302.1157, found 302.1154.



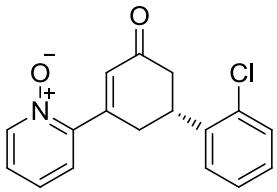
3ia

(R)-2-(3'-methyl-5-oxo-1,2,5,6-tetrahydro-[1,1'-biphenyl]-3-yl)pyridine 1-oxide： yellow solid in 99% yield. mp 109-110 ˚C; $[\alpha]_D^{20}$ 0.582 (c = 0.913, $CHCl_3$); HPLC: Daicel Chiralpak IC, ethanol = 100%, flow rate = 0.37 mL/min, T = 30 ˚C, UV = 231 nm, t_R = 26.8 min (minor), t_R= 29.2 min (major); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.25-8.24 (d, *J* = 4.2 Hz, 1H), 7.31 (s, 3H), 7.24-7.20 (m, 1H), 7.11-7.06 (m, 3H), 6.34 (s, 1H), 3.52-3.44 (m, 1H), 3.13-3.11 (d, *J* = 7.4 Hz, 2H), 2.84-2.73 (m, 2H), 2.34 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 198.0, 153.7, 148.0, 141.7, 139.4, 137.3, 129.0, 127.6, 126.8, 126.6, 125.0, 124.9, 122.8, 43.7, 40.1, 33.8, 20.4; IR (film, ν/cm^{-1}): 3068, 2961, 2360, 1666, 1607, 1483, 1370, 769, 699; HRMS (ESI) m/z calcd for $C_{18}H_{17}NO_2$ $[M+ H]^+$ 280.1338, found 280.1339.



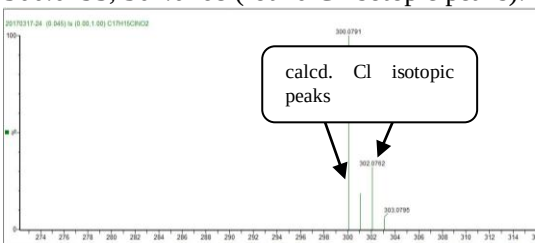
3ja

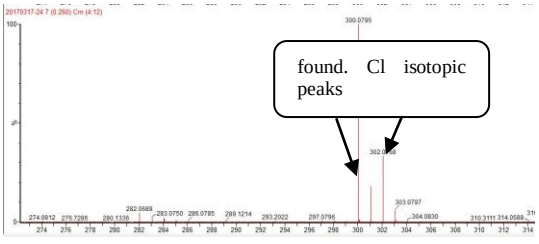
(R)-2-(2'-fluoro-5-oxo-1,2,5,6-tetrahydro-[1,1'-biphenyl]-3-yl)pyridine 1-oxide: yellow oil in 83% yield. $[\alpha]_D^{20}$ 1.05 (c = 1.12, $CHCl_3$); HPLC: Daicel Chiralpak IC, ethanol = 100%, flow rate = 0.37 mL/min, T = 30 ˚C, UV = 254 nm, t_R = 25.7 min (minor), t_R= 29.2 min (major); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.27-8.26 (d, *J* = 5.9 Hz, 1H), 7.36-7.31 (m, 4H), 7.27-7.21 (m, 1H), 7.15-7.11 (m, 1H), 7.07-7.02 (m, 1H), 6.36 (s, 1H), 3.84-3.76 (m, 1H), 3.31-3.23 (m, 1H), 3.11-3.06 (m, 1H), 2.89-2.77 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 197.5, 161.0-158.5 (d, *J* = 244.5 Hz), 153.5, 147.9, 139.4, 129.0, 128.4-128.3 (d, *J* = 13.7 Hz), 127.6 (d, *J* = 8.3 Hz), 127.0-126.9 (d, *J* = 4.6 Hz), 125.3, 125.1, 123.4 (d, *J* = 3.4 Hz), 114.8-114.6 (d, *J* = 22.3 Hz), 42.2, 33.9 (d, *J* = 1.8 Hz), 32.1; IR (film, ν/cm^{-1}): 3081, 2921, 2359, 1660, 1586, 1555, 1489, 1453, 1375, 750; HRMS (ESI) m/z calcd for $C_{17}H_{14}FNO_2$ $[M+H]^+$ 284.1087, found 284.1089.

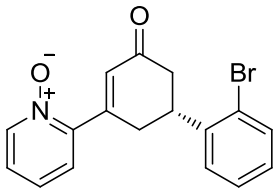


3ka

(R)-2-(2'-chloro-5-oxo-1,2,5,6-tetrahydro-[1,1'-biphenyl]-3-yl)pyridine 1-oxide: yellow oil in 88% yield. $[\alpha]_D^{20}$ 1.10 (c = 0.993, $CHCl_3$); HPLC: Daicel Chiralpak IC, ethanol = 100%, flow rate = 0.37 mL/min, T = 30 ˚C, UV = 231 nm, t_R = 26.7 min (minor), t_R= 28.3 min (major); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.26-8.24 (d, *J* = 6.0 Hz, 1H), 7.40-7.28 (m, 6H), 7.21-7.18 (m, 1H), 6.37 (s, 1H), 4.03-3.95 (m, 1H), 3.29-3.22 (m, 1H), 3.11-3.06 (m, 1H), 2.86-2.73 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 197.5, 153.4, 147.8, 139.4, 138.6, 132.6, 129.0, 128.9, 127.2, 126.3, 125.3, 125.1, 125.1, 42.3, 36.4, 32.2; IR (film, ν/cm^{-1}): 3059, 2959, 2360, 1664, 1551, 1475, 1371, 759; HRMS (ESI) m/z calcd. for $C_{17}H_{14}ClNO_2$ $[M+H]^+$ 300.0791, 302.0762 （calcd. Cl isotopic peaks）, found 300.0795, 302.0768 (found Cl isotopic peaks).

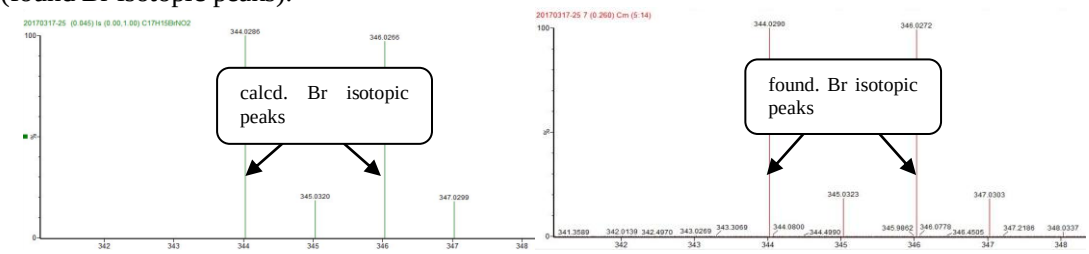


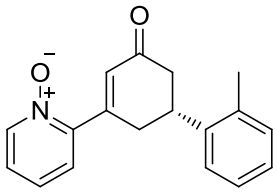




3la

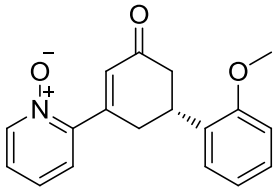
(R)-2-(2'-bromo-5-oxo-1,2,5,6-tetrahydro-[1,1'-biphenyl]-3-yl)pyridine 1-oxide: yellow oil in 77% yield. $[\alpha]_D^{20}$ 0.703 (c = 0.925, $CHCl_3$); HPLC: Daicel Chiralpak AD-H, hexane: 2-propanol = 75:25, flow rate = 1.0 mL/min, T = 30 ˚C, UV = 231 nm, t_R = 17.9 min (major), t_R = 21.0 min (minor); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.17-8.16 (d, *J* = 6.0 Hz, 1H), 7.48-7.46 (d, *J* = 7.9 Hz, 1H), 7.31-7.21 (m, 5H), 7.04-7.01 (m, 1H), 6.29 (s, 1H), 3.90-3.82 (m, 1H), 3.19-3.12 (m, 1H), 3.03-2.97 (m, 1H), 2.77-2.62 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 198.4, 154.3, 148.8, 141.2, 140.4, 133.3, 130.0, 128.6, 127.9, 127.4, 126.2, 126.2, 126.1, 124.3, 43.5, 40.0, 33.4; IR (film, ν/cm^{-1}): 3058, 2921, 2360, 1665, 1566, 1484, 1470, 1367, 754; HRMS (ESI) m/z calcd for $C_{17}H_{14}BrNO_2$ $[M+H]^+$ 344.0286, 346.0085（calcd. Br isotopic peaks）, found 344.0290, 346.0272 (found Br isotopic peaks).





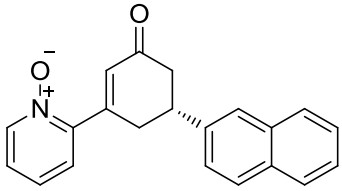
3ma

(R)-2-(2'-methyl-5-oxo-1,2,5,6-tetrahydro-[1,1'-biphenyl]-3-yl)pyridine 1-oxide： yellow oil in 99% yield. $[\alpha]_D^{20}$ -1.46 (c = 1.05, $CHCl_3$); HPLC: Daicel Chiralpak AD-H, hexane: 2-propanol = 75:25, flow rate = 1.0 mL/min, T = 30 ˚C, UV = 231 nm, t_R = 17.4 min (major), t_R= 24.8 min (minor); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.26-8.25 (d, *J* = 5.4 Hz, 1H), 7.32-7.29 (m, 4H), 7.21-7.13 (m, 3H), 6.35 (s, 1H), 3.79-3.73 (m, 1H), 3.15-3.11 (d, *J* = 14.8 Hz 1H), 3.03-2.96 (m, 1H), 2.80-2.72 (m, 2H), 2.39 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 198.2, 153.9, 147.9, 139.7, 139.4, 134.7, 129.8, 129.0, 125.8, 125.4, 125.1, 125.1, 125.0, 124.2, 43.1, 36.0, 33.0, 18.3; IR (film, ν/cm^{-1}): 3069, 2960, 2360, 1666, 1485, 1461, 1368, 758; HRMS (ESI) m/z calcd for $C_{18}H_{17}NO_2$ $[M+ H]^+$ 280.1338, found 280.1340.



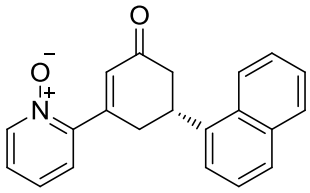
3na

(R)-2-(2'-methoxy-5-oxo-1,2,5,6-tetrahydro-[1,1'-biphenyl]-3-yl)pyridine 1-oxide: yellow oil in 83% yield. $[\alpha]_D^{20}$ 1.35 (c = 1.01, $CHCl_3$); HPLC: Daicel Chiralpak IC, ethanol = 100%, flow rate = 0.37 mL/min, T = 30 ˚C, UV = 231 nm, t_R = 25.8 min (minor), t_R= 27.5 min (major); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.26-8.24 (d, *J* = 5.8 Hz, 1H), 7.35-7.28 (m, 3H), 7.26-7.21 (m, 2H), 6.96-6.92 (m, 1H), 6.88-6.86 (d, *J* = 8.1 Hz, 1H), 6.37-6.36 (d, *J* = 1.9 Hz, 1H), 3.90-3.85 (m, 1H), 3.83-3.82 (s, 3H), 3.31-3.23 (m, 1H), 3.06-3.00 (m, 1H), 2.87-2.75 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 198.7, 156.0, 154.1, 148.2, 139.4, 129.7, 129.0, 126.9, 126.0, 125.0, 125.0, 124.9, 119.6, 109.5, 54.2, 42.4, 34.3, 32.0; IR (film, ν/cm^{-1}): 3107, 3063, 3009, 2950, 2844, 2361, 1665, 1598, 1584, 1493, 1465, 1373, 1223,753; HRMS (ESI) m/z calcd for $C_{18}H_{17}NO_3$ $[M+H]^+$ 296.1287, found 296.1291.



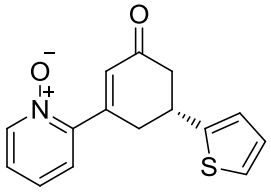
3oa

(R)-2-(5-(naphthalen-2-yl)-3-oxocyclohex-1-en-1-yl)pyridine 1-oxide: white solid in 67% yield. mp 175-177 ˚C; $[\alpha]_D^{20}$ 3.21 (c = 1.05, $CHCl_3$); HPLC: Daicel Chiralpak AD-H, hexane: 2-propanol = 75:25, flow rate = 1.0 mL/min, T = 30 ˚C, UV = 254 nm, t_R = 32.3 min (major), t_R= 36.6 min (minor); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.25-8.24 (d, *J* = 5.3 Hz, 1H), 7.83-7.79 (m, 3H), 7.72 (s, 1H), 7.49-7.42 (m, 3H), 7.30-7.28 (m, 3H), 6.37 (s, 1H), 3.73-3.65 (m, 1H), 3.28-3.18 (m, 2H) ,2.95-2.84 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 198.8, 154.7, 149.0, 140.4, 140.2, 133.5, 132.5, 130.1, 128.4, 127.8, 127.6, 126.3, 126.1, 126.0, 125.9, 125.8, 125.4, 125.2, 44.7, 41.2, 34.6; IR (film, ν/cm^{-1}): 3051, 2885, 1663, 1507, 1480, 1351; HRMS (ESI) m/z calcd for $C_{21}H_{17}NO_2$ $[M+H]^+$ 316.1338, found 316.1342.



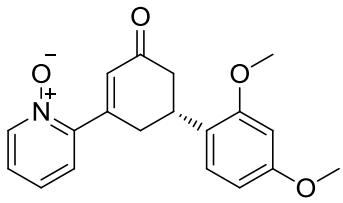
3pa

(R)-2-(5-(naphthalen-1-yl)-3-oxocyclohex-1-en-1-yl)pyridine 1-oxide： yellow oil in 63% yield. $[\alpha]_D^{20}$ -7.44 (c = 0.900, $CHCl_3$); HPLC: Daicel Chiralpak IC, ethanol = 100%, flow rate = 0.37 mL/min, T = 30 ˚C, UV = 240 nm, t_R = 32.6 min (minor), t_R= 34.2 min (major); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.18-8.16 (d, *J* = 5.6 Hz, 1H), 8.10-8.07 (d, *J* = 8.5 Hz, 1H), 7.79-7.77 (m, 1H), 7.70-7.66 (m, 1H), 7.49-7.45 (m, 1H), 7.42-7.35 (m, 3H), 7.26-7.18 (m, 3H), 6.32-6.31 (d, *J* = 2.1 Hz, 1H) 4.33-4.26 (m, 1H), 3.33-3.28 (m, 1H), 3.08-3.00 (m, 1H), 2.93-2.81 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 199.2, 155.0, 148.9, 140.4, 138.5, 134.0, 131.1, 130.1, 129.1, 127.7, 126.5, 126.2, 126.1, 126.0, 125.8, 125.4, 122.8, 122.8, 44.5, 36.3, 34.6; IR (film, ν/cm^{-1}): 3047, 2921, 2360, 1664, 1597, 1509, 1484, 1351; HRMS (ESI) m/z calcd for $C_{21}H_{17}NO_2$ $[M+H]^+$ 316.1338, found 316.1339.



3qa

(R)-2-(3-oxo-5-(thiophen-2-yl)cyclohex-1-en-1-yl)pyridine 1-oxide: yellow oil in 62% yield. $[\alpha]_D^{20}$ -1.03 (c = 1.08, $CHCl_3$); HPLC: Daicel Chiralpak IC, ethanol = 100%, flow rate = 0.37 mL/min, T = 30 ˚C, UV = 240 nm, t_R = 28.9 min (minor), t_R= 34.3 min (major); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.25 (s, 1H), 7.31 (s, 3H), 7.19-7.17 (d, *J* = 4.9 Hz, 1H), 6.96-6.92 (m, 2H), 6.32 (s, 1H), 3.85-3.79 (m, 1H), 3.38-3.33 (m, 1H), 3.14-3.07 (m, 1H) 3.00-2.95 (m, 1H) 2.82-2.74 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 196.8, 152.9, 147.8, 145.6, 139.4, 129.2, 125.8, 125.1, 125.1, 125.0, 122.6, 122.6, 44.5, 35.4, 34.4; IR (film, ν/cm^{-1}): 3101, 2886, 2360, 1663, 1523, 1484, 1367, 860; HRMS (ESI) m/z calcd for $C_{15}H_{13}NO_2S$ $[M+H]^+$ 272.0745, found 272.0750.



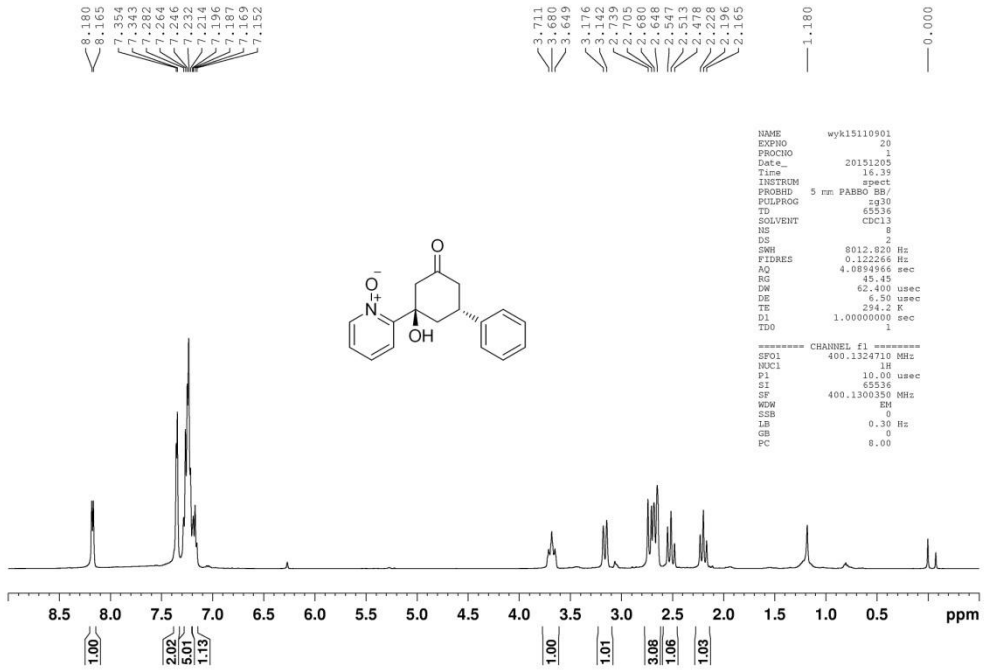
3ra

(R)-2-(2',4'-dimethoxy-5-oxo-1,2,5,6-tetrahydro-[1,1'-biphenyl]-3-yl)pyridine 1-oxide: yellow oil in 68 % yield. $[\alpha]_D^{20}$ 2.55 (c = 0.990, $CHCl_3$); HPLC: Daicel Chiralpak AD-H hexane: 2-propanol = 75:25, flow rate = 1.0 mL/min, T = 30 ˚C, UV = 206 nm, t_R = 21.3 min (major), t_R= 25.2 min (minor); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.25-8.23 (d, *J* = 5.8 Hz, 1H), 7.34-7.27 (m, 3H), 7.15-7.13 (d, *J* = 9.0 Hz, 1H), 6.46-6.45 (m, 2H), 6.35 (s, 1H), 3.86-3.70 (m, 7H), 3.30-3.20 (m, 1H), 3.02-2.97 (m, 1H), 2.85-2.71 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 199.0, 158.7, 157.0, 154.2, 148.2, 139.4, 128.9, 126.5, 125.1, 125.0, 124.9, 122.3, 102.9, 97.7, 54.3, 54.2, 42.7, 33.9, 32.3; IR (film, ν/cm^{-1}): 3055, 2923, 2835, 2360, 1663, 1609, 1585, 1505, 1463, 1370, 1205, 1156, 862, 820; HRMS (ESI) m/z calcd for $C_{19}H_{19}NO_4$ $[M+H]^+$ 326.1392, found 326.1392.

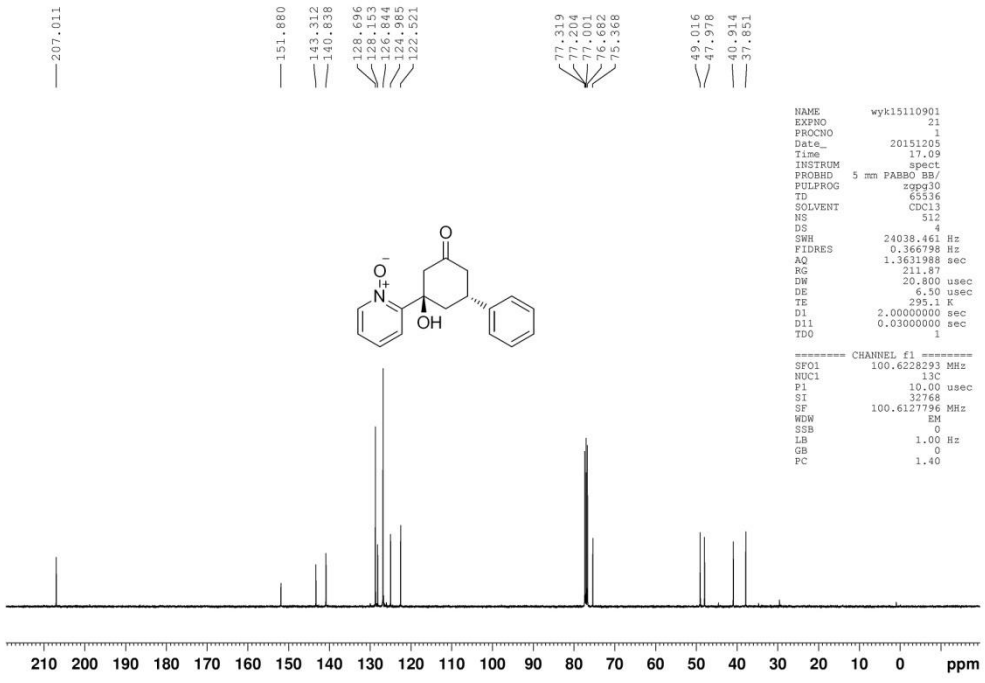
References:

[1] J. Gao, H. Wu, B. Xiang, W. Yu, L. Han, and Y. Jia, *J. Am. Chem. Soc*., 2013, **135**, 2983.
[2] S. Rout, S. Ray, R. Unhale, V. Singh, *Org. Lett.*, 2014, **16**, 5568.
[3] L. Li, S. Zhang, Y. Hu, Y. Li, C. Li, Z. Zha, and Z. Wang, *Chem. Eur. J*., 2015, **21**, 12885.

Part II NMR Spectra
3aa'-^{1}H NMR



3aa'-^{13}C NMR



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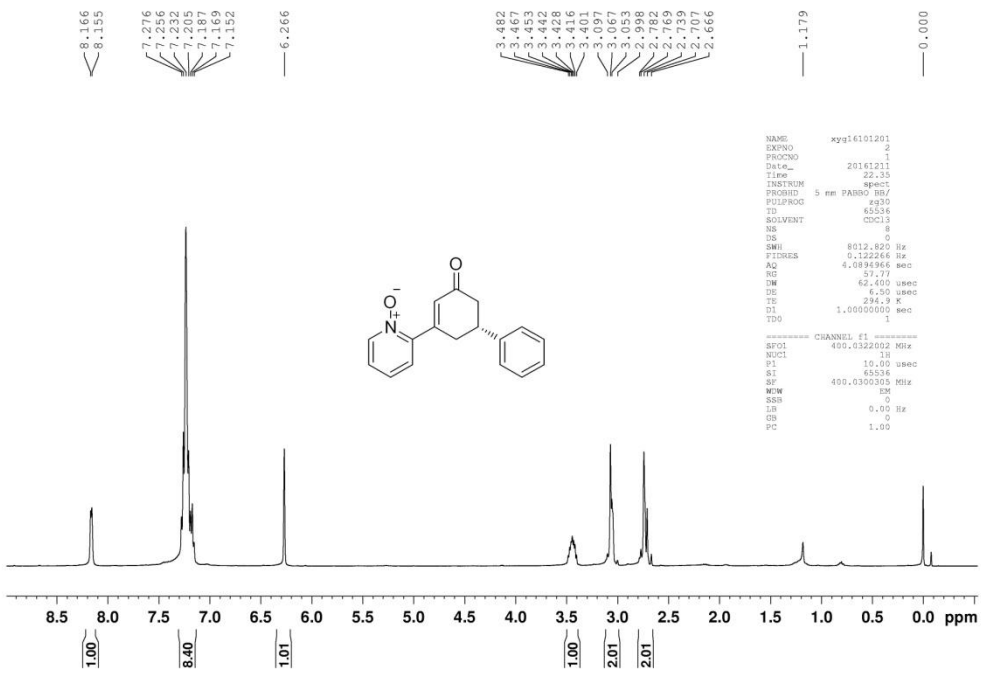

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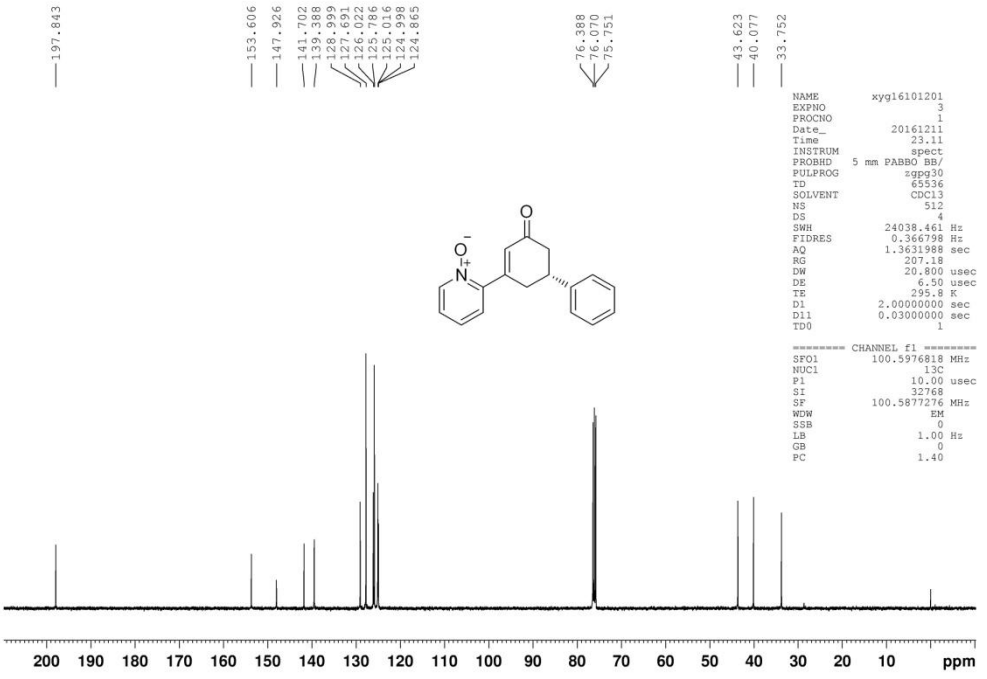

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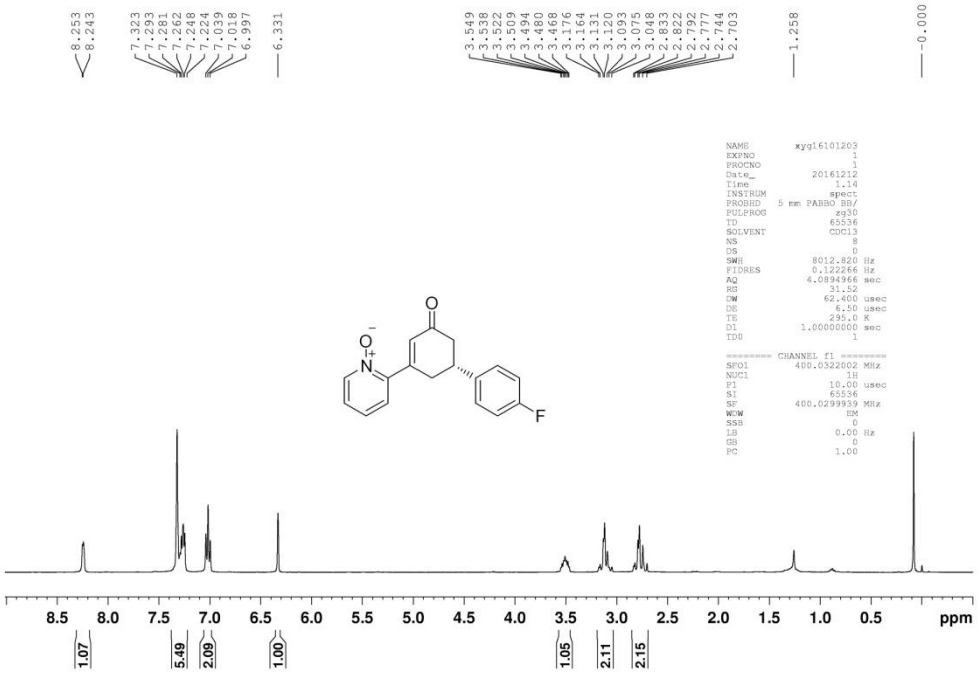

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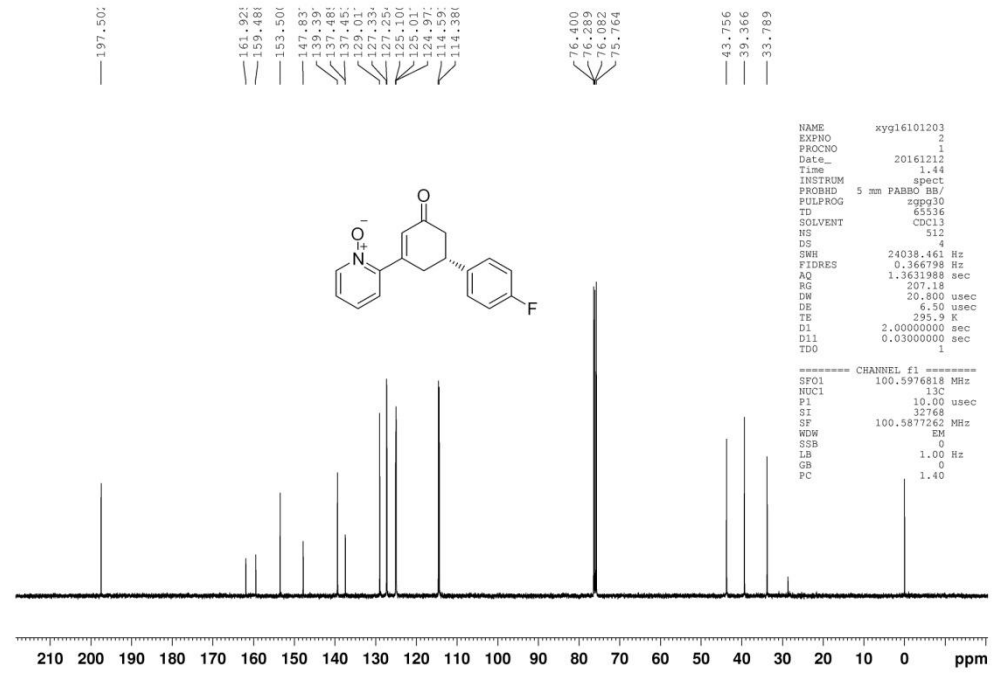

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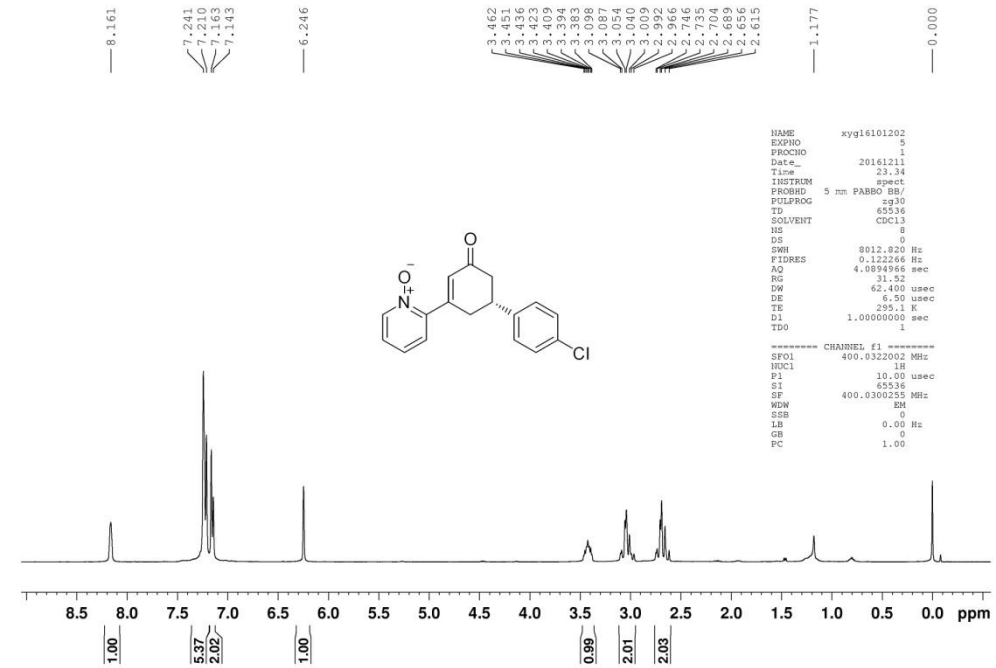

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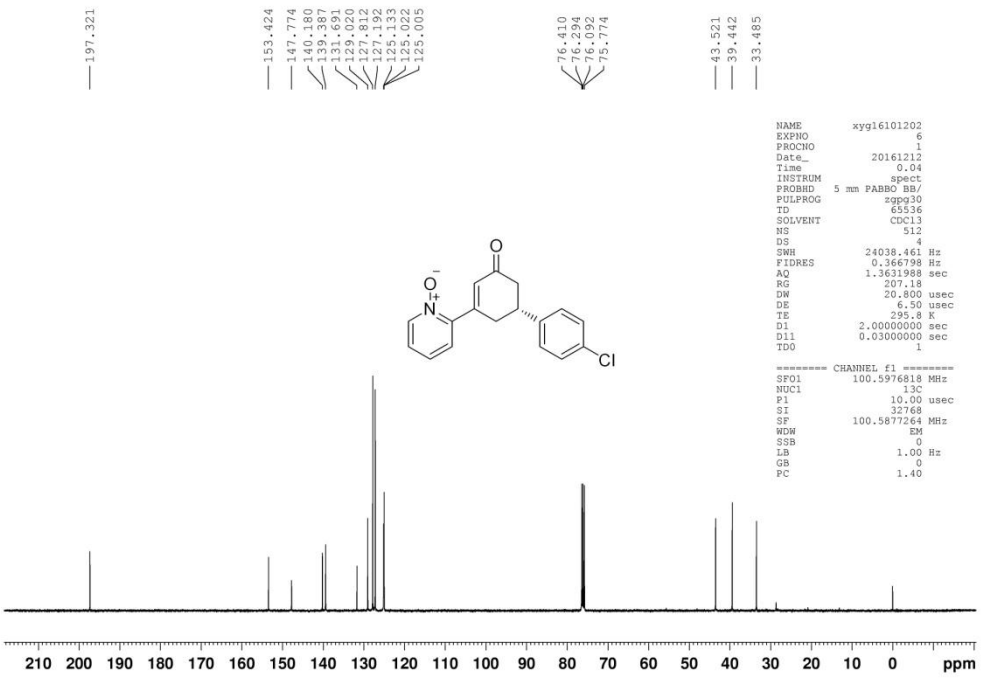

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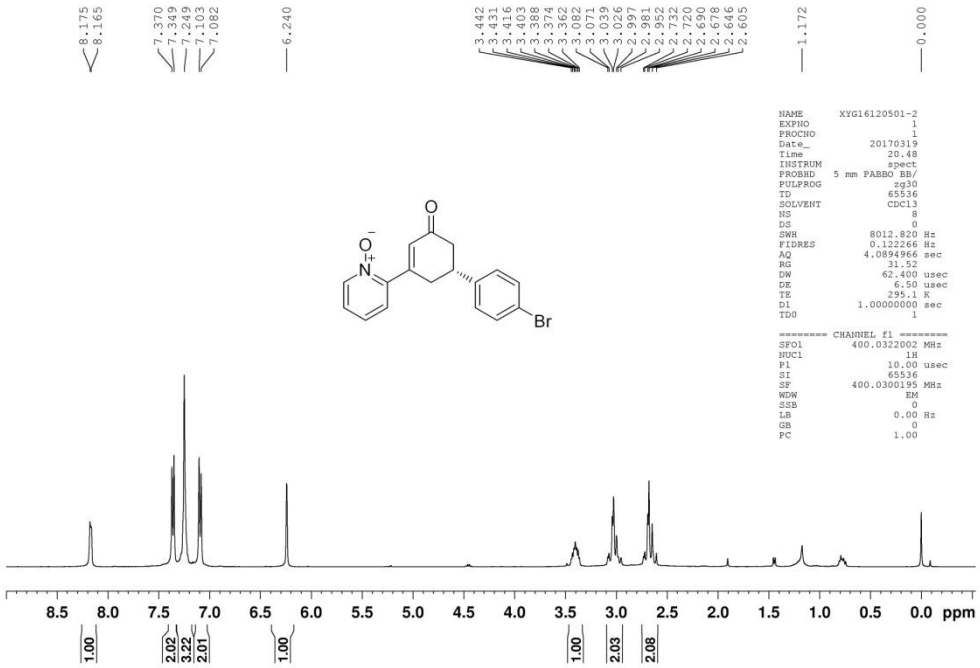

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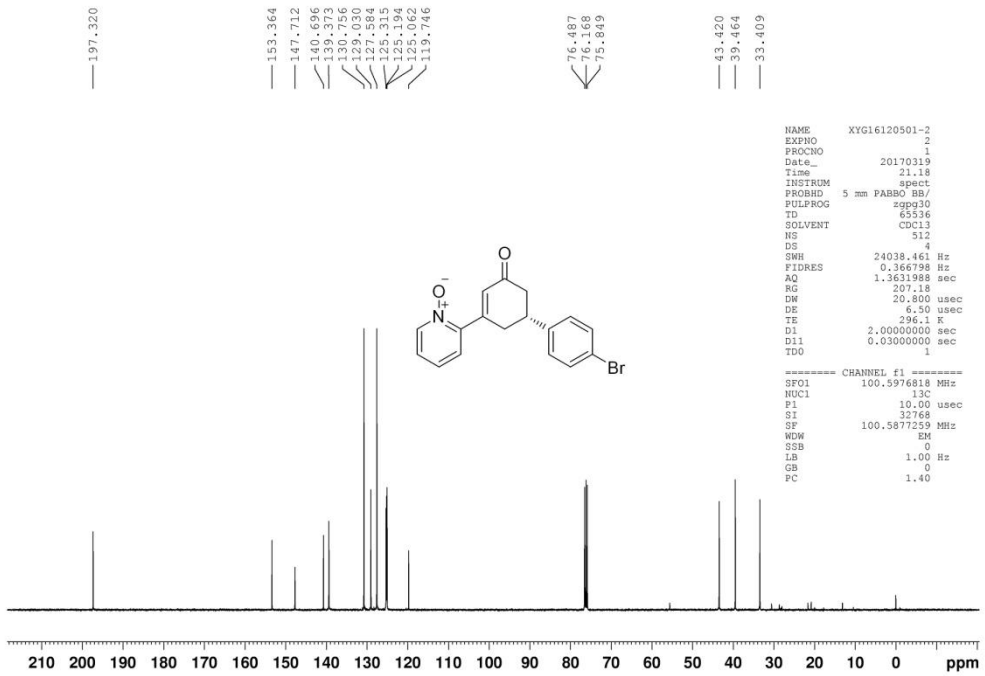

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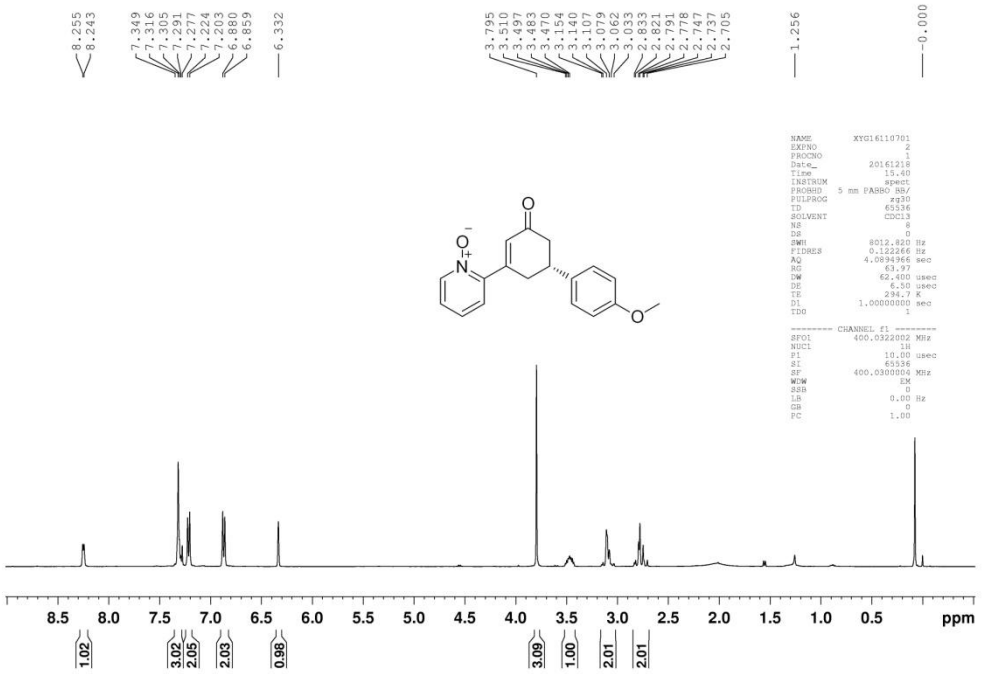
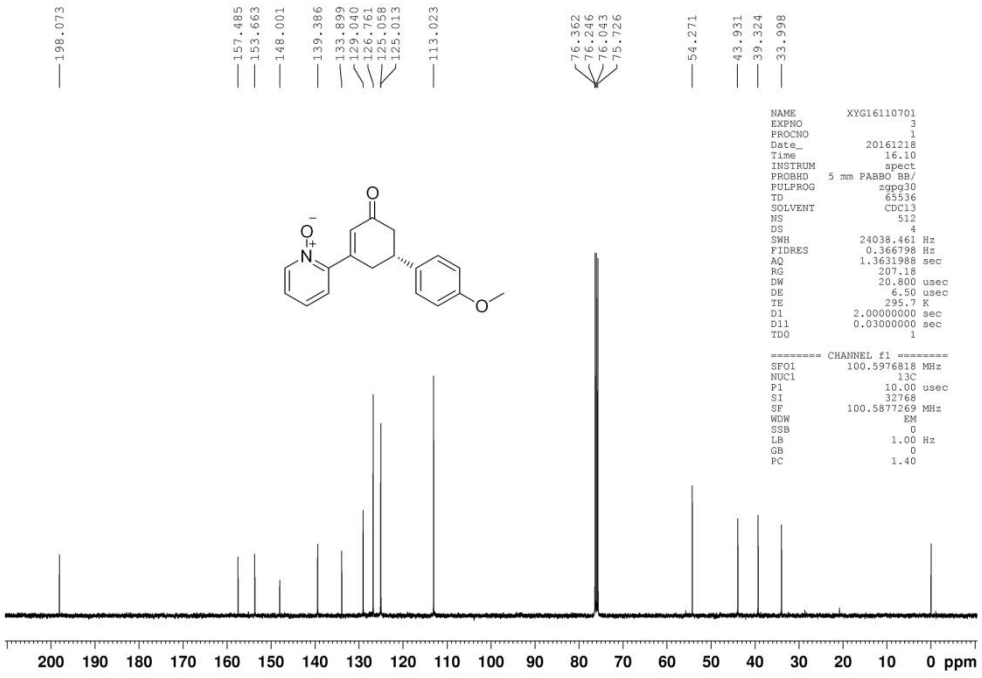
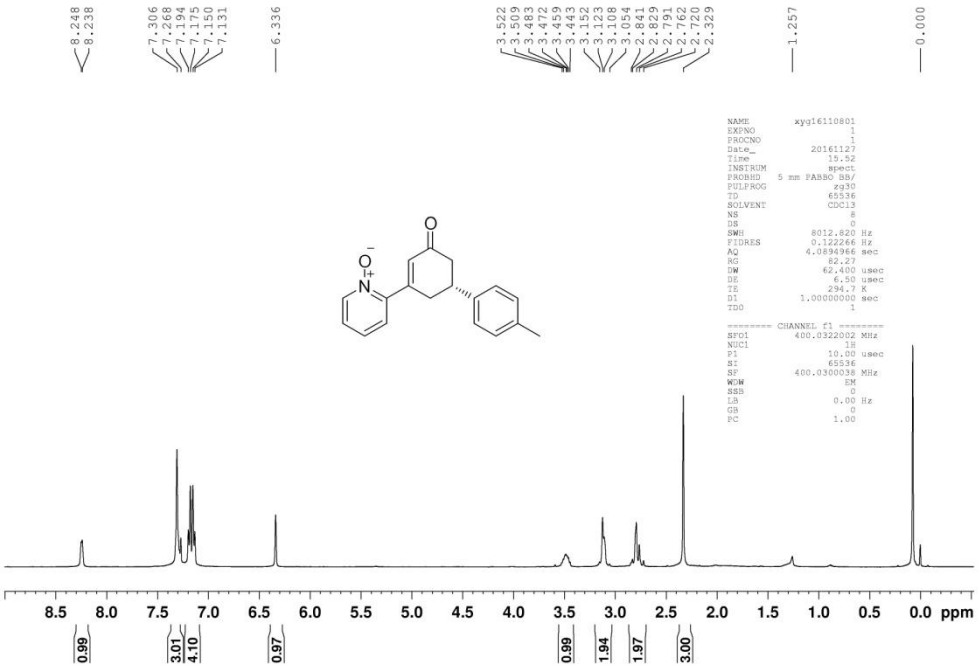


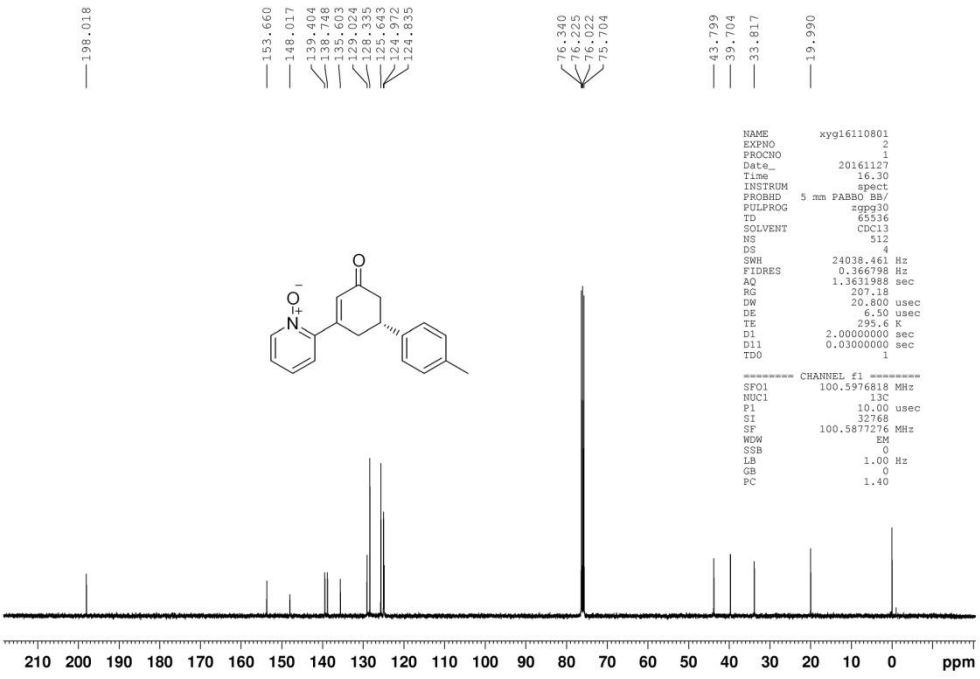
3ea-^{13}C NMR



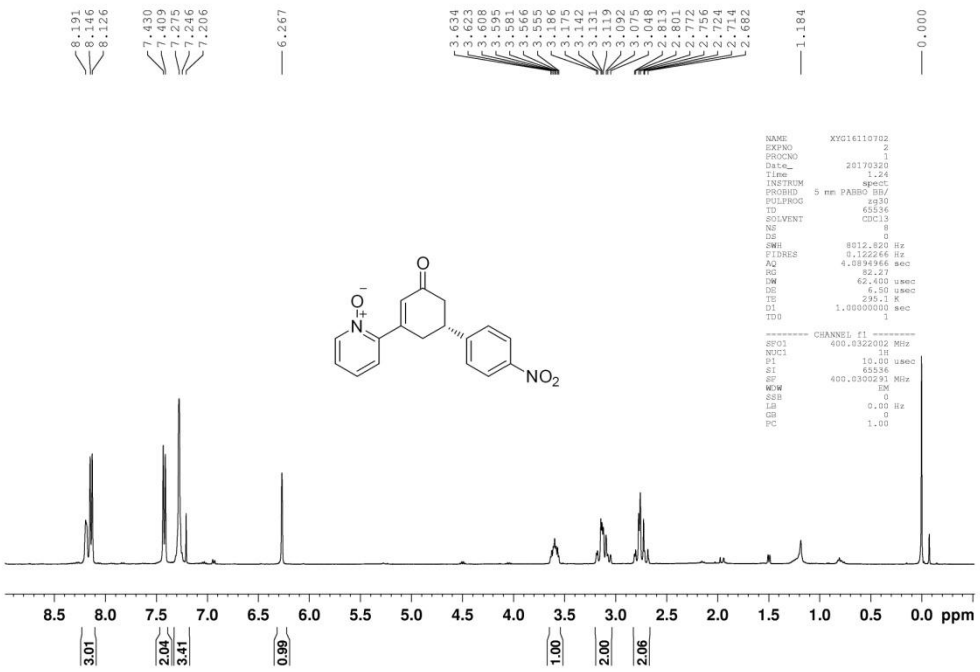
3fa-^{1}H NMR



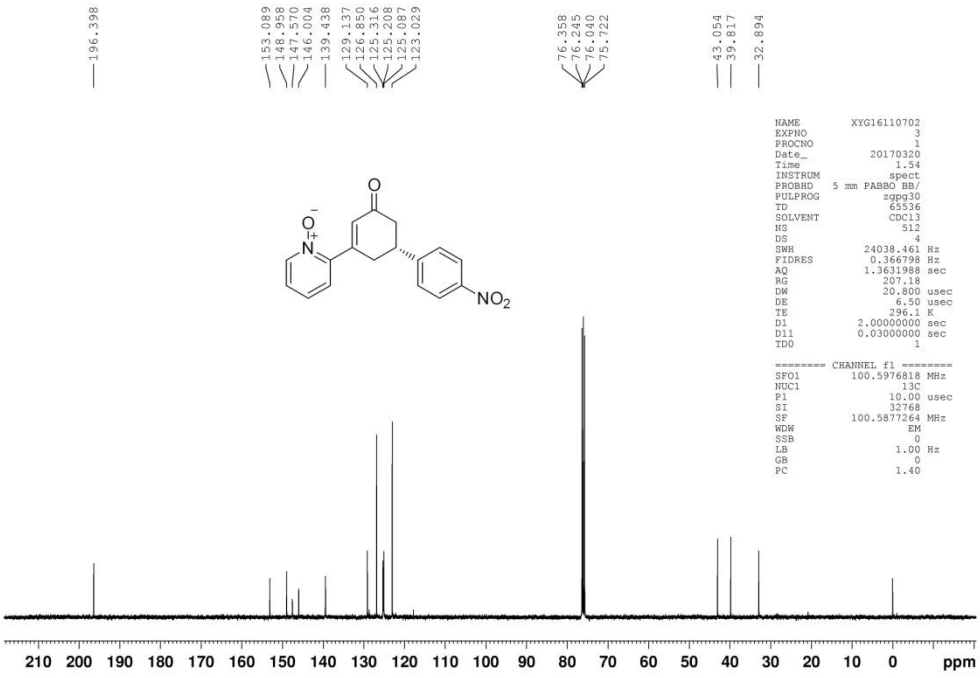
3fa-^{13}C NMR



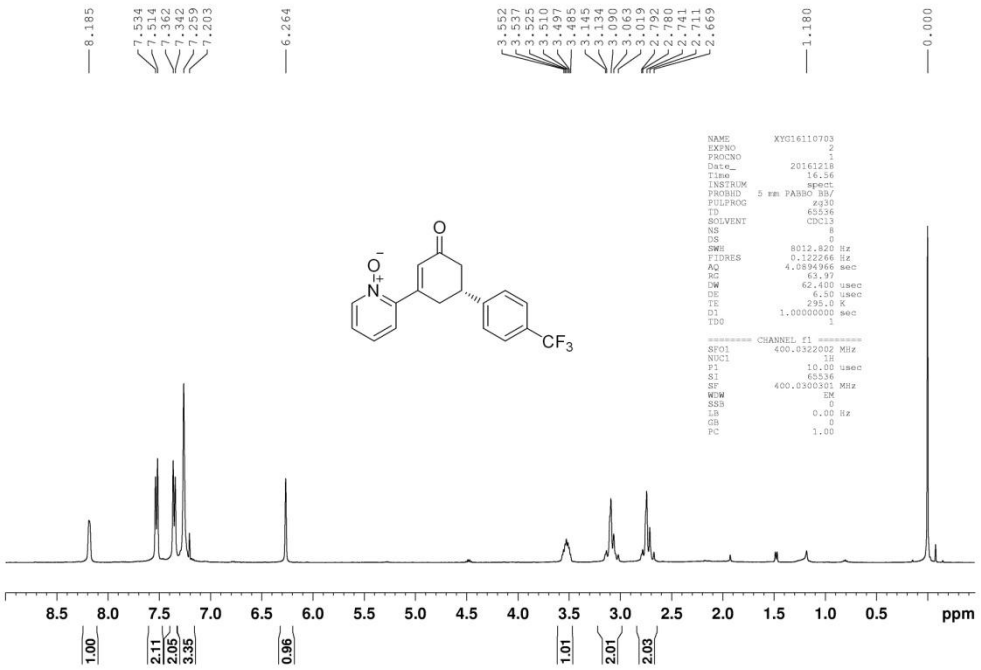
3ga-^{1}H NMR



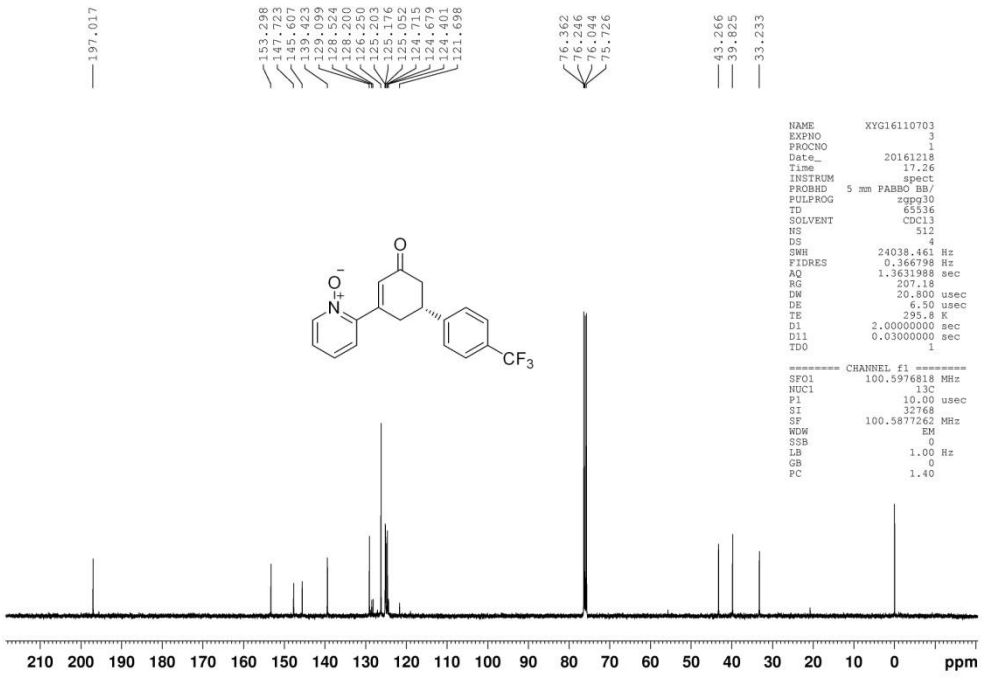
3ga-^{13}C NMR



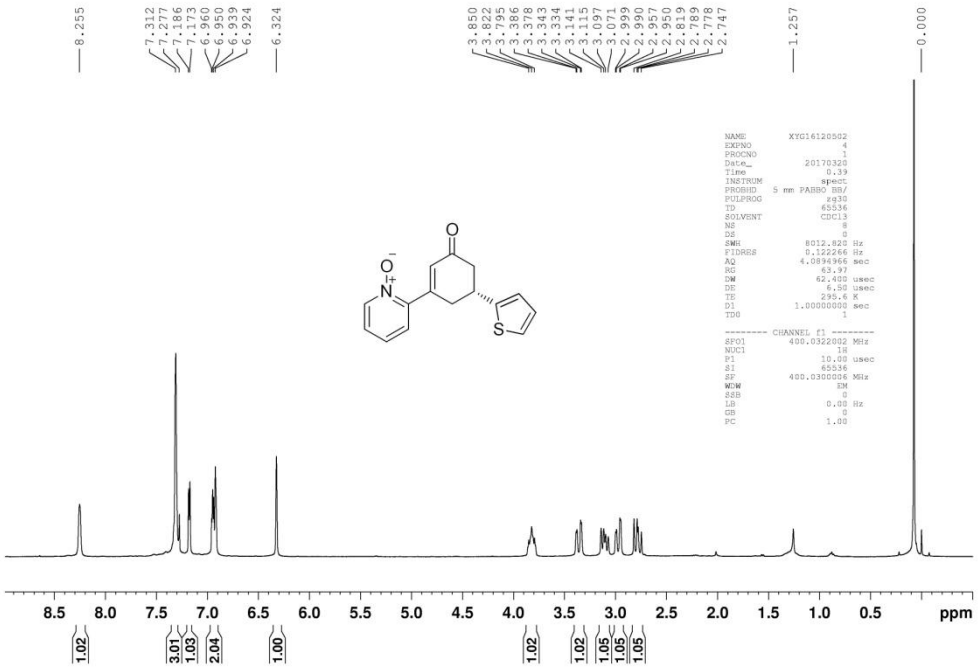
3ha-^{1}H NMR



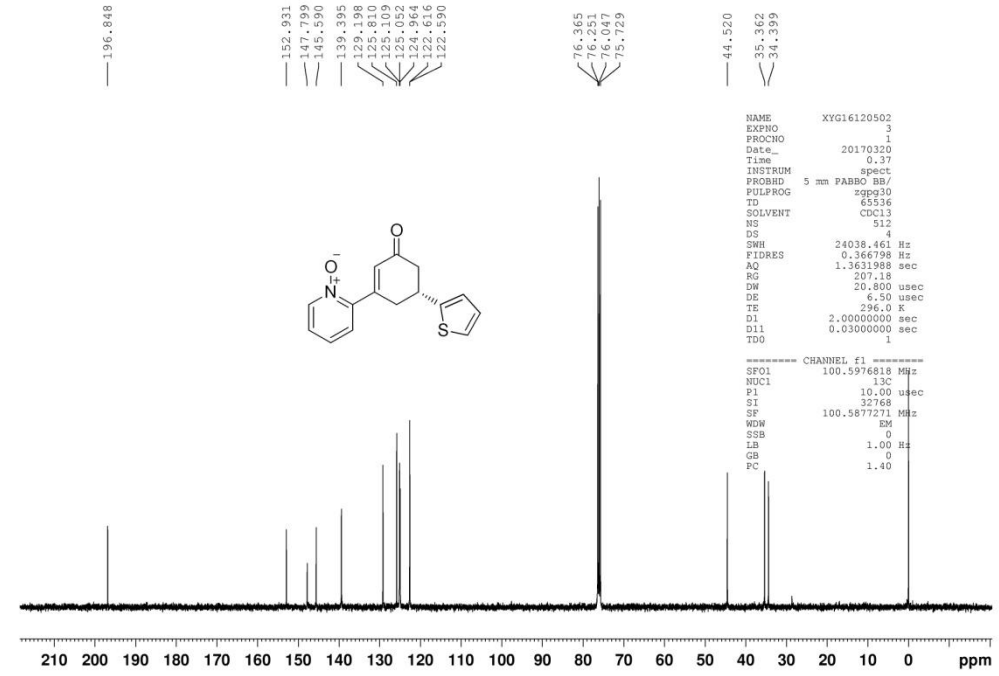
3ha-^{13}C NMR



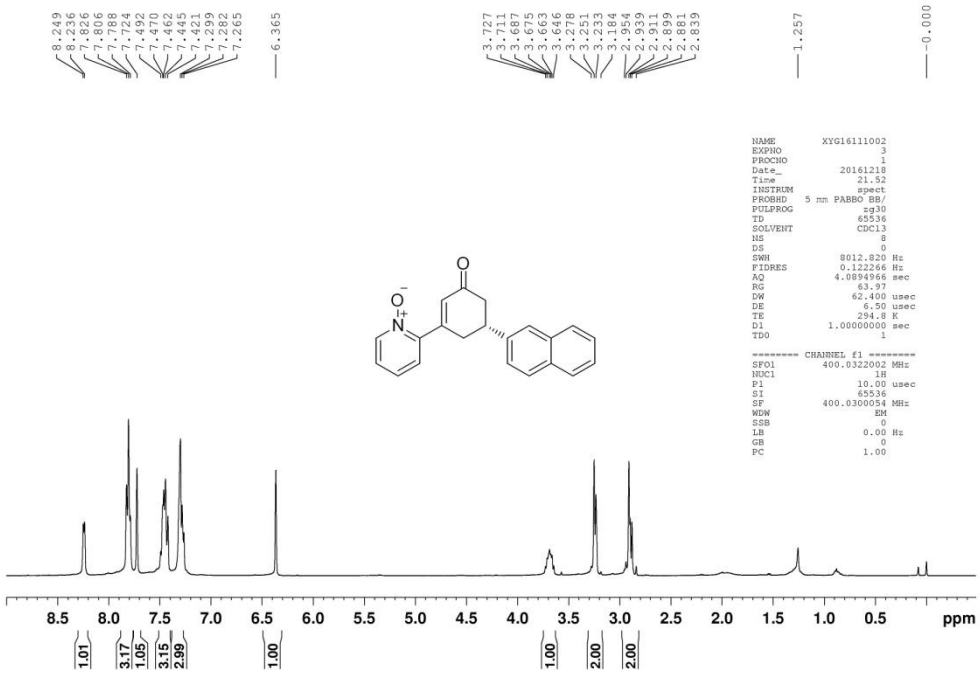
3ia-^{1}H NMR



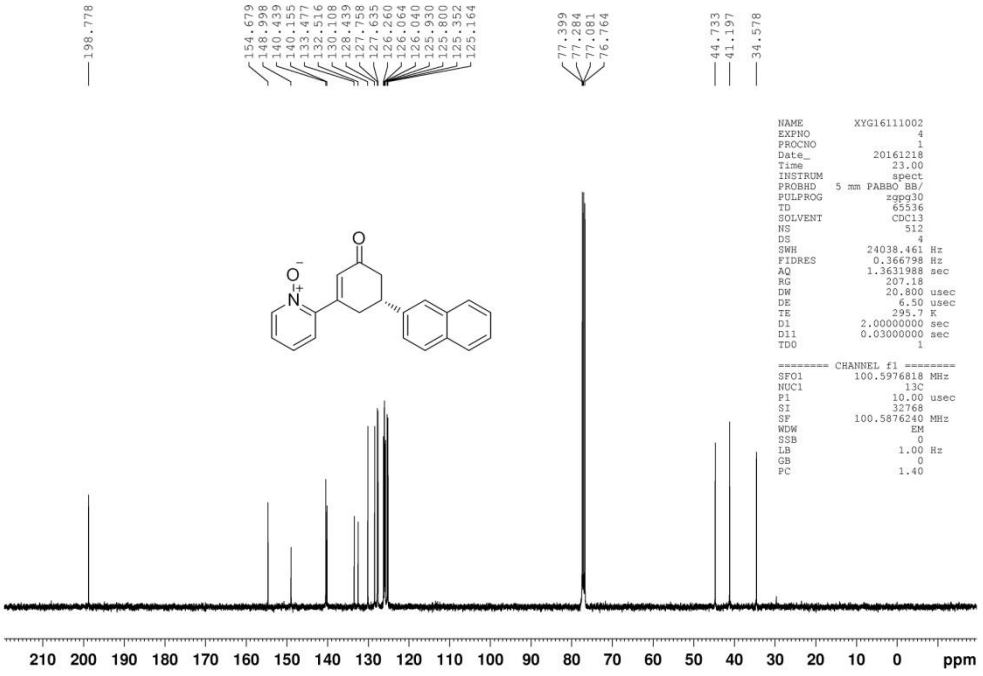
3ia-^{13}C NMR



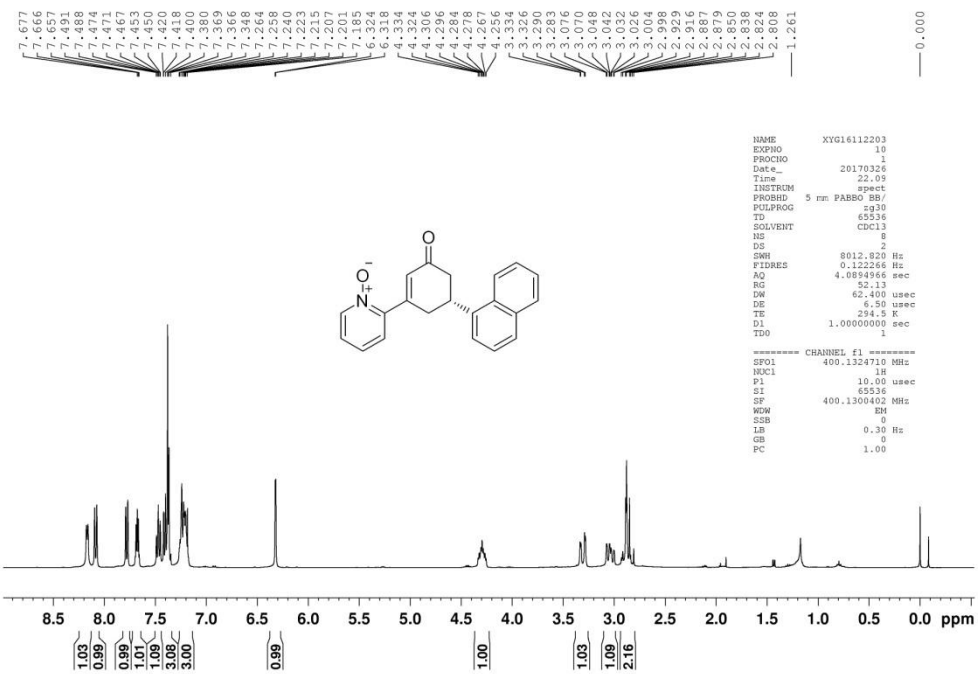
3ja-^{1}H NMR



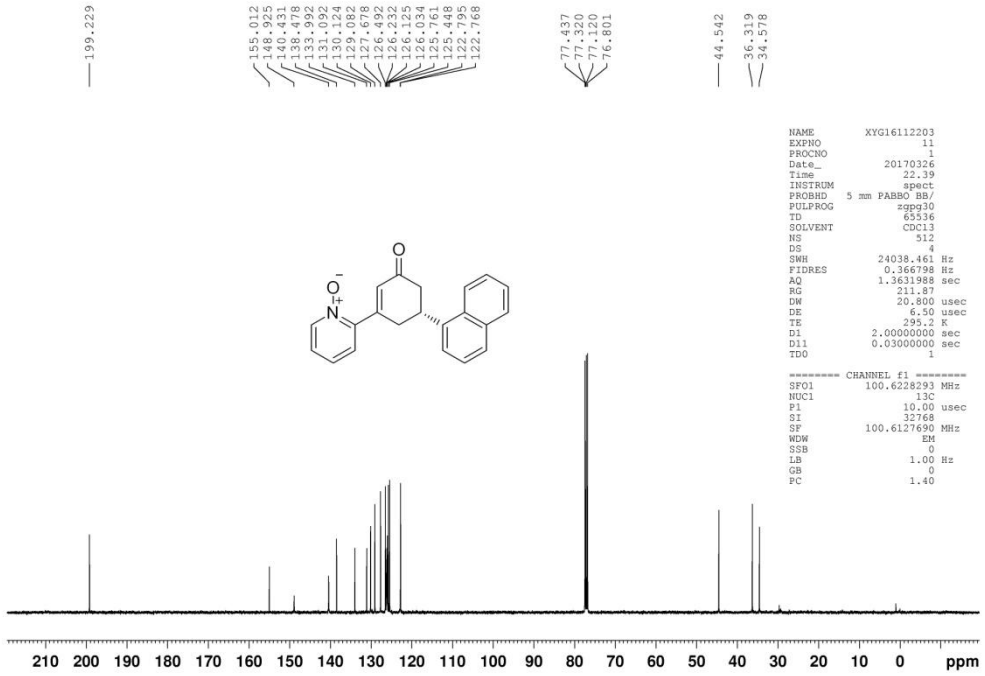
3ja-^{13}C NMR



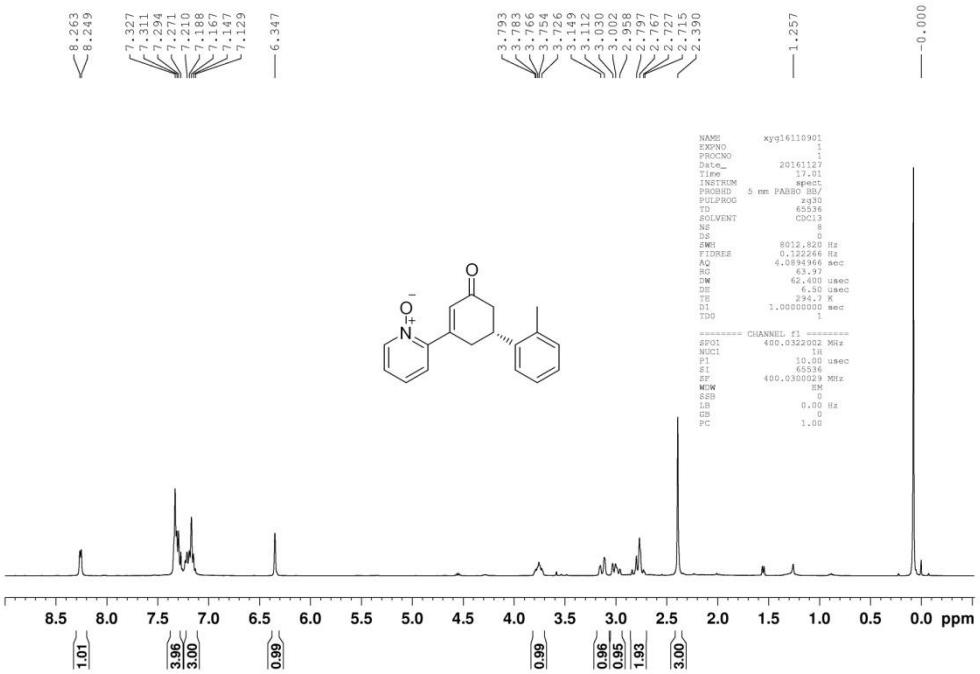
3ka-^{1}H NMR



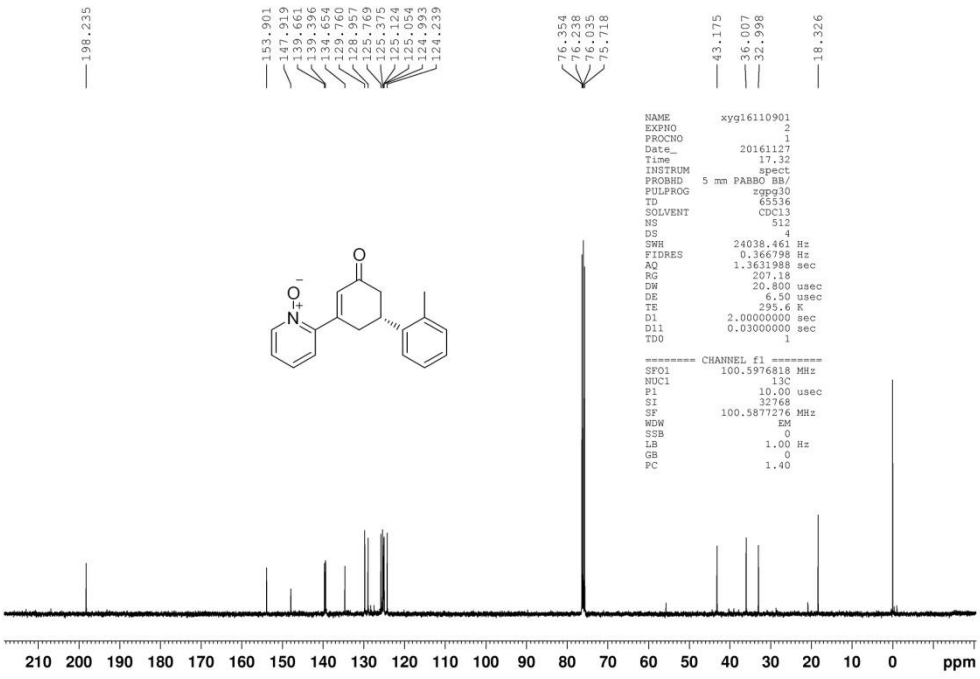
3ka-^{13}C NMR



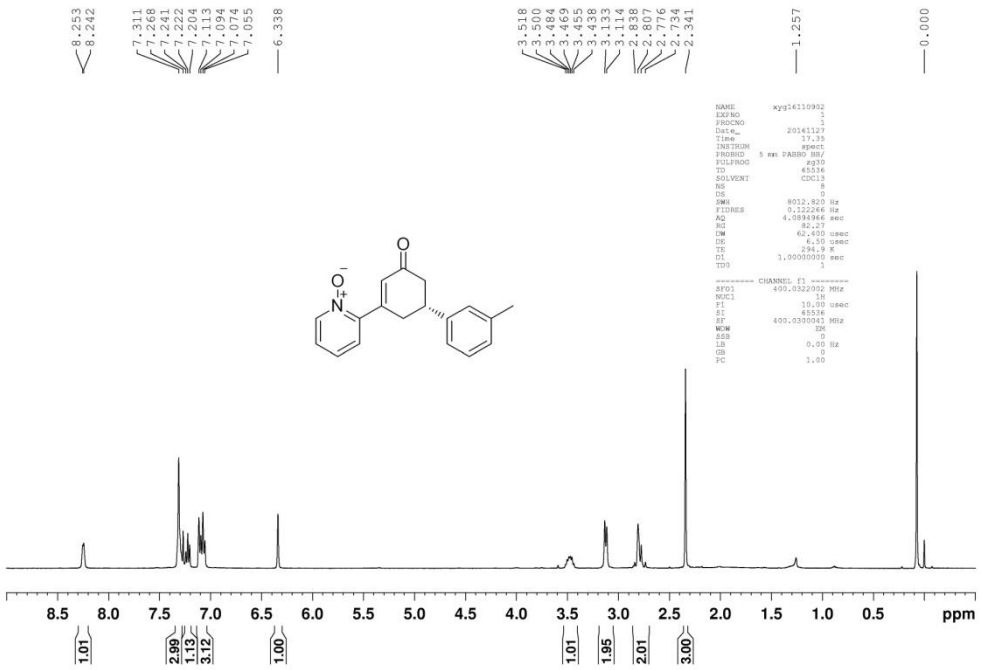
3la-^{1}H NMR



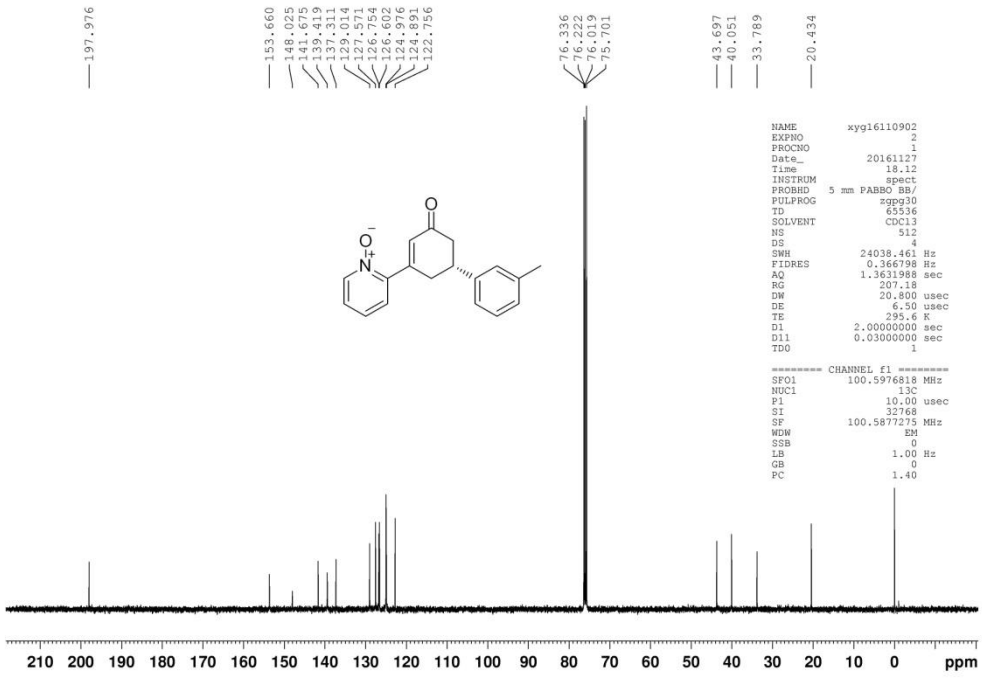
3la-^{13}C NMR



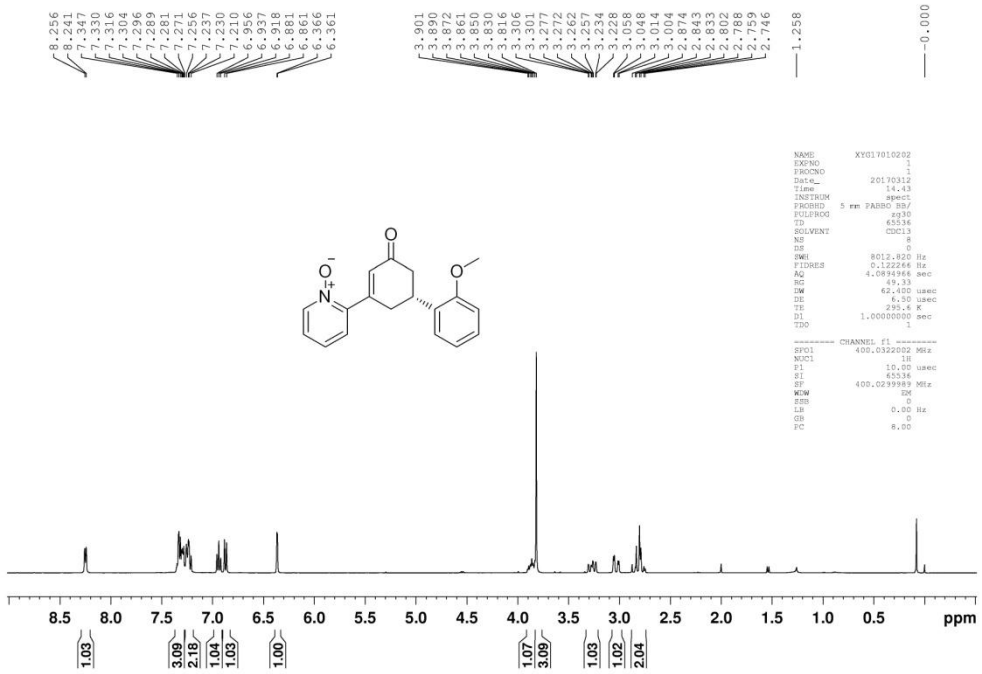
3ma-^{1}H NMR



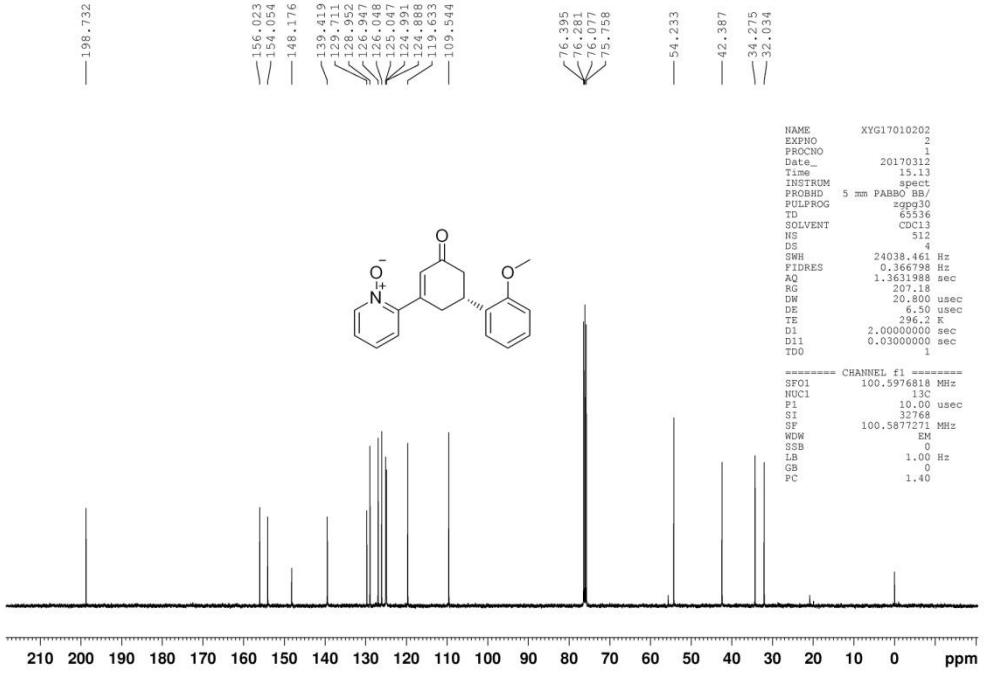
3ma-^{13}C NMR



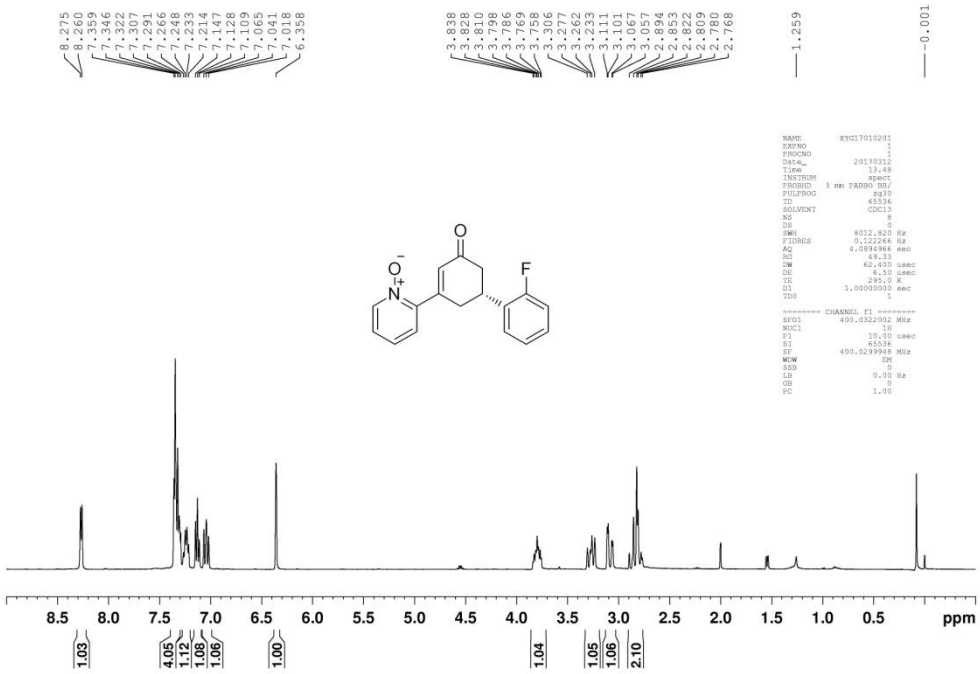
3na-^{1}H NMR



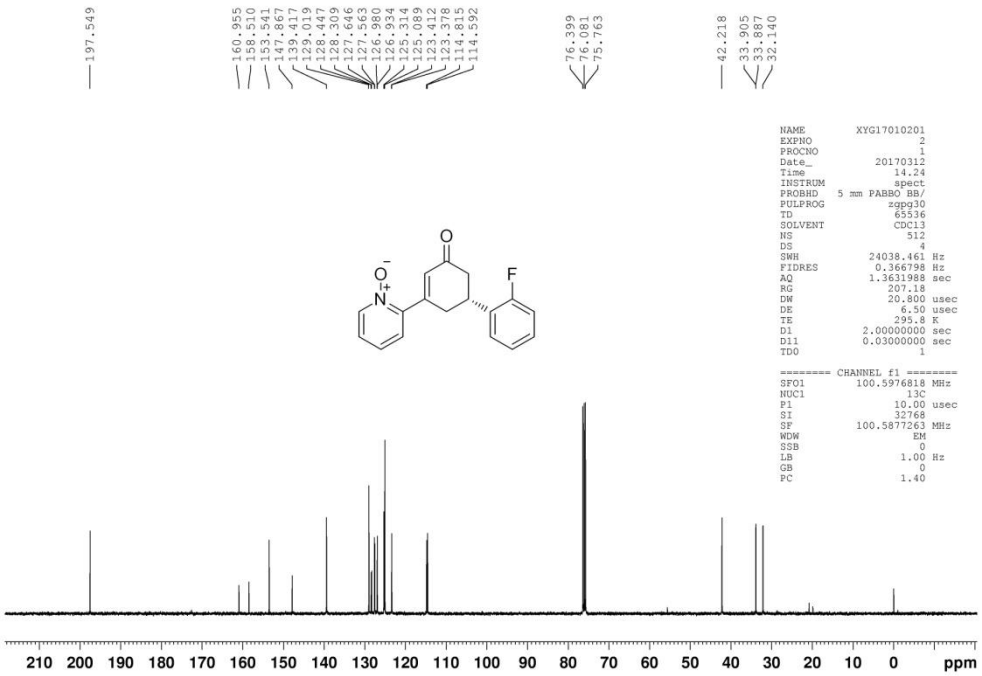
3na-^{13}C NMR



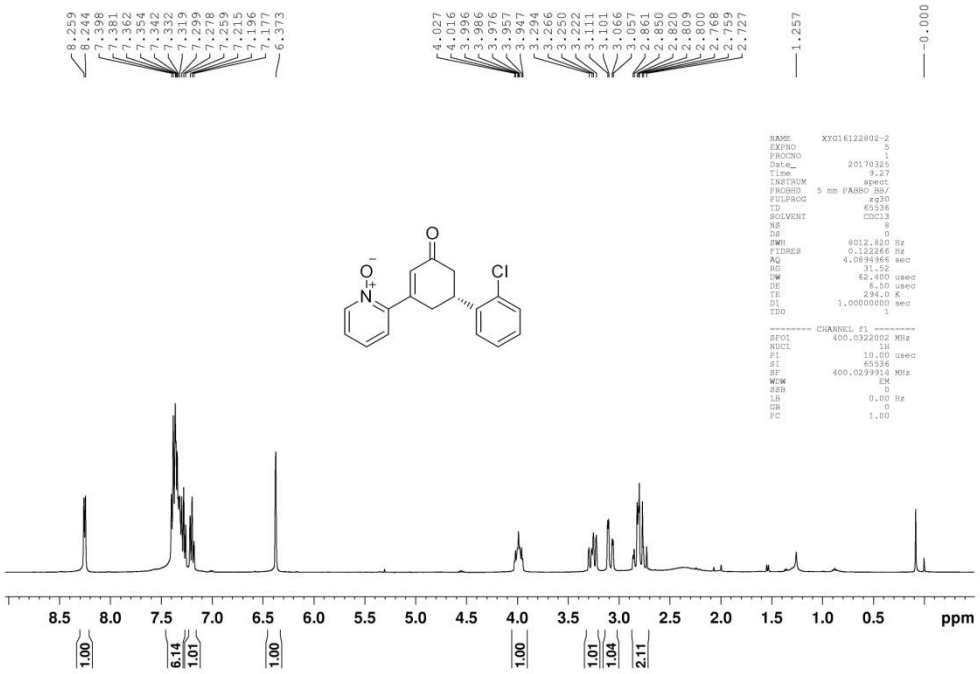
3oa-^{1}H NMR



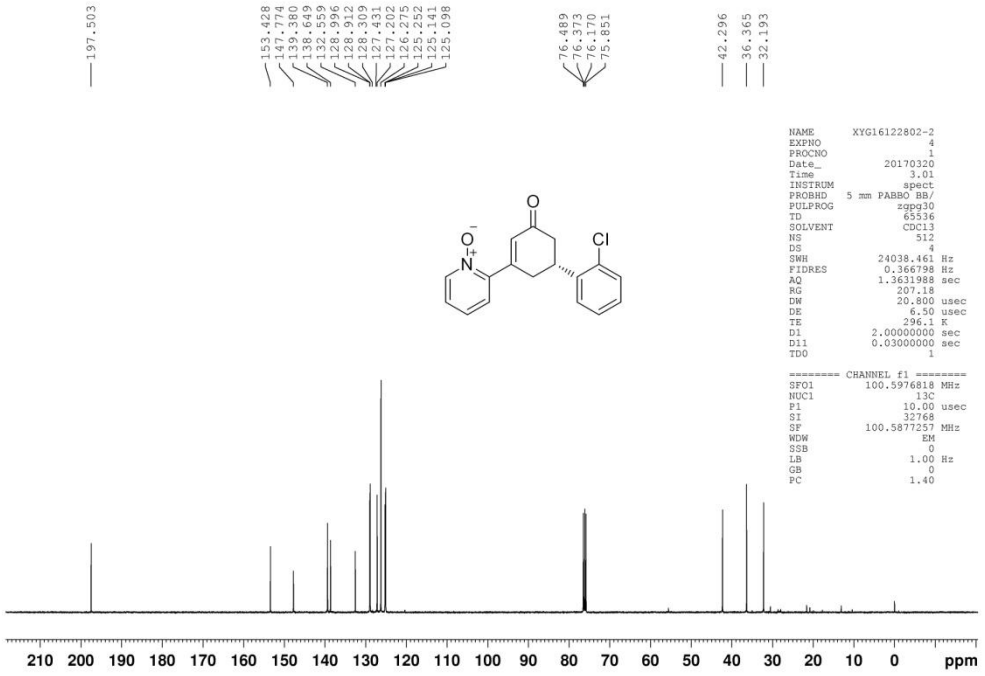
3oa-^{13}C NMR



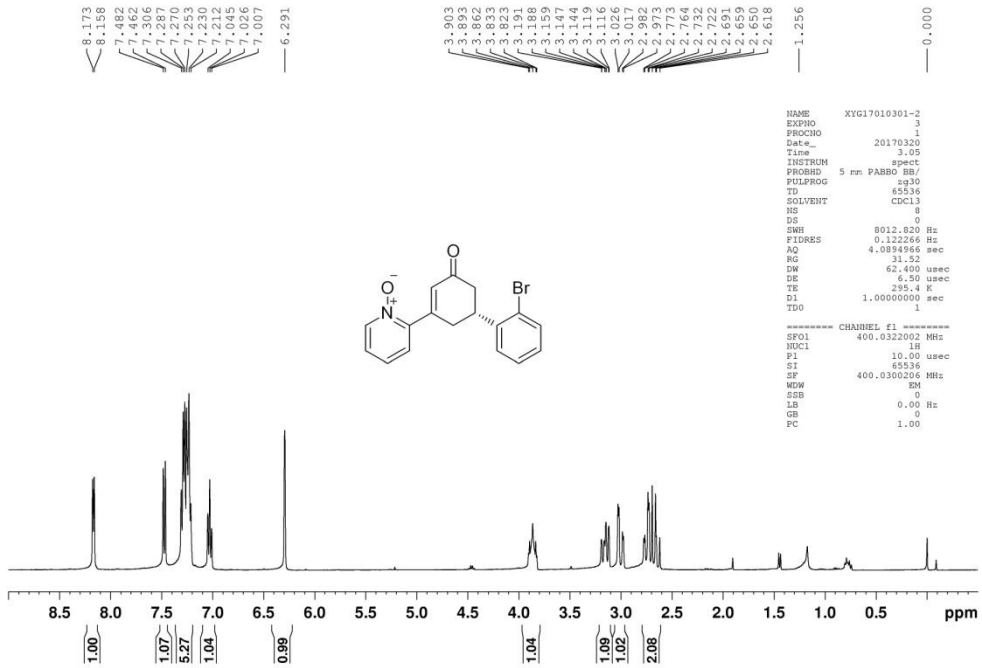
3pa-^{1}H NMR



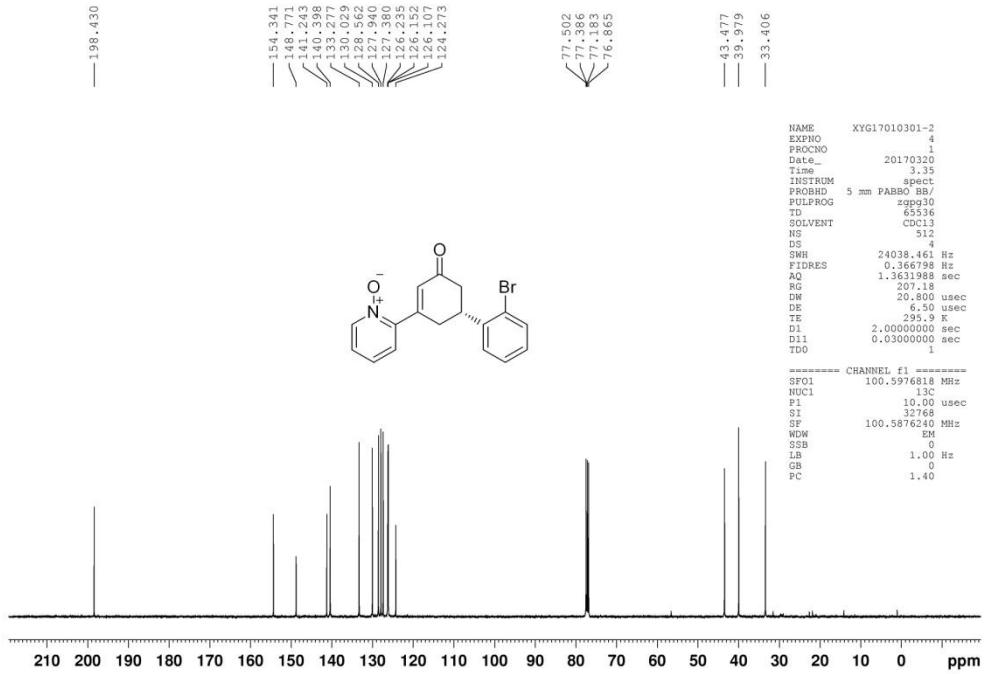
3pa-^{13}C NMR



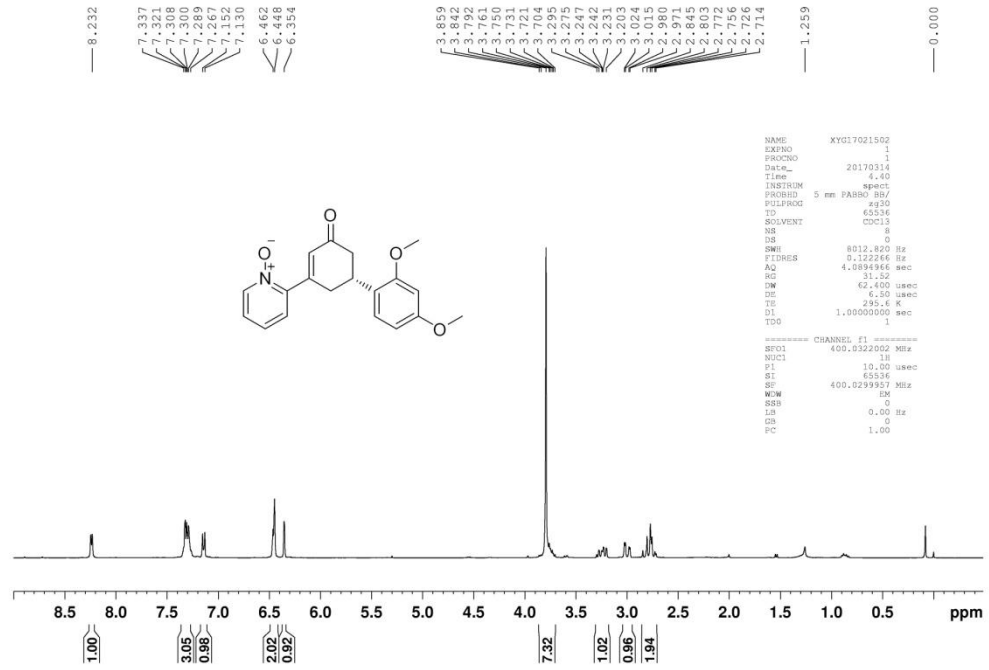
3qa-^{1}H NMR



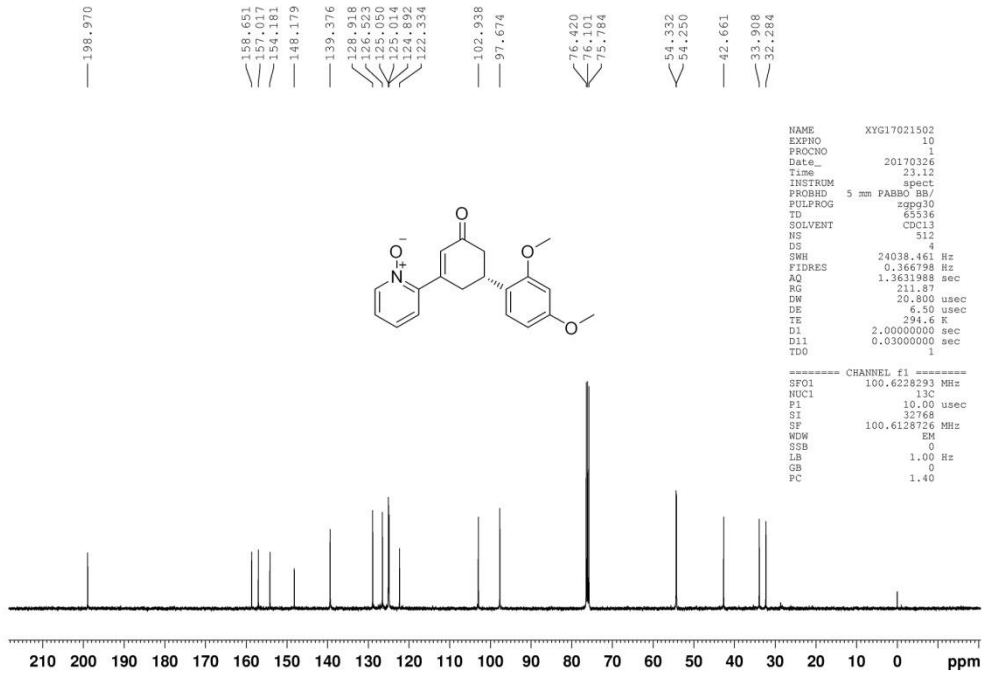
3qa-^{13}C NMR



3ra-^{1}H NMR

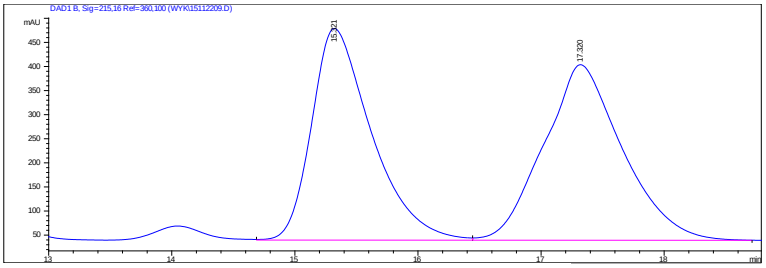


3ra-^{13}C NMR



Part III HPLC Spectra

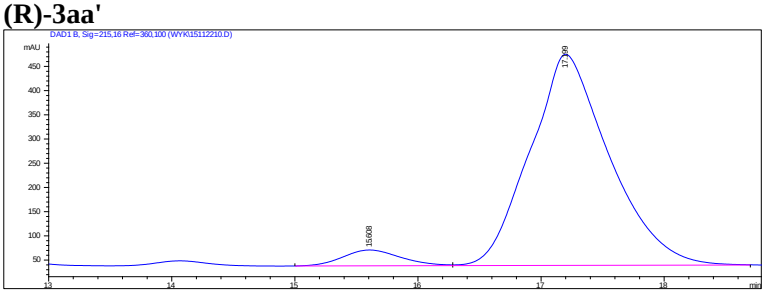
3aa' racemic



Signal 2: DAD1 B, Sig=215,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.321	VV	0.4991	1.48723e4	439.13617	47.9078
2	17.320	VB	0.6142	1.61713e4	364.11127	52.0922
Totals :				3.10436e4	803.24744	

(R)-3aa'

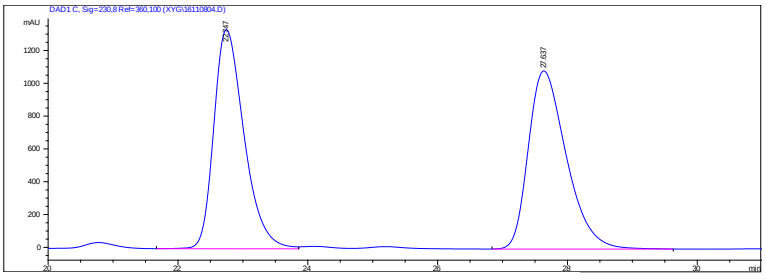


Signal 2: DAD1 B, Sig=215,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.608	BV	0.4964	1072.78931	32.73037	5.1995
2	17.199	VB	0.6191	1.95597e4	436.20685	94.8005
Totals :				2.06325e4	468.93722	

90%

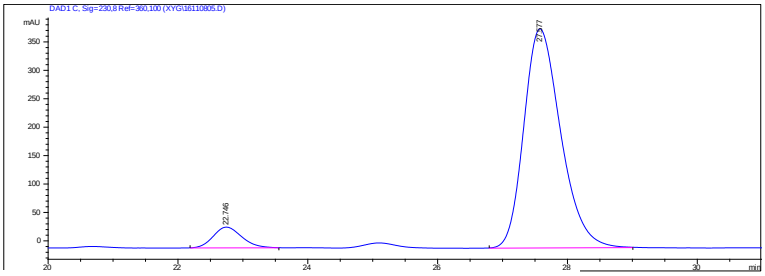
3aa racemic



Signal 3: DAD1 C, Sig=230,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	22.747	VV	0.4979	4.32726e4	1335.79871	49.5214
2	27.637	BB	0.6240	4.41090e4	1085.76160	50.4786
Totals :				8.73816e4	2421.56030	

(R)-3aa

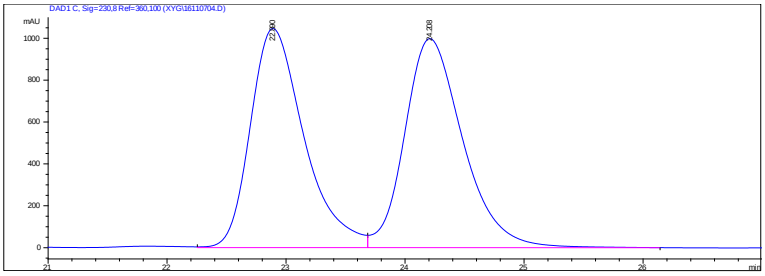


Signal 3: DAD1 C, Sig=230,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	22.746	BB	0.4737	1121.22156	36.57402	6.8730
2	27.577	BB	0.6080	1.51923e4	385.27209	93.1270
Totals :				1.63135e4	421.84611	

86%

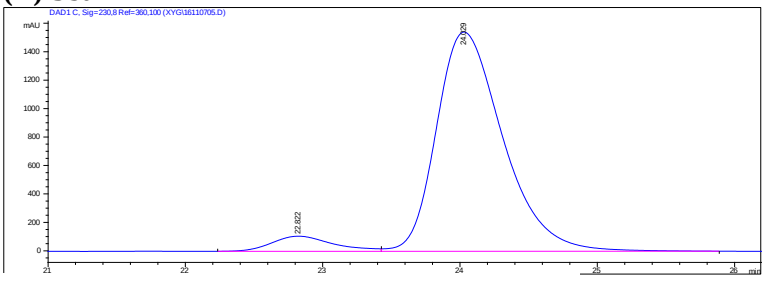
3ba racemic



Signal 3: DAD1 C, Sig=230,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	22.890	VV	0.4776	3.27900e4	1046.23901	49.1391
2	24.208	VB	0.5206	3.39389e4	998.34503	50.8609
Totals :				6.67289e4	2044.58405	

(R)-3ba

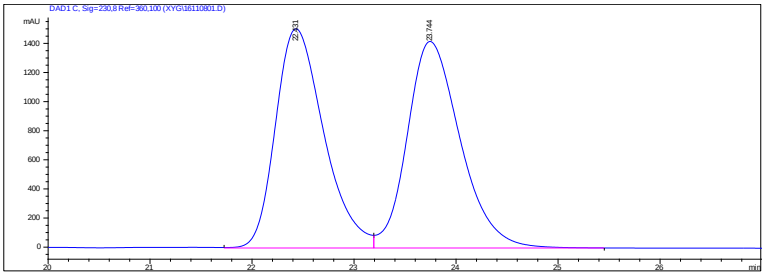


Signal 3: DAD1 C, Sig=230,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	22.822	BV	0.4694	3288.16797	106.14540	5.9593
2	24.029	VB	0.5146	5.18892e4	1541.81946	94.0407
Totals :				5.51773e4	1647.96486	

88%

3ca racemic

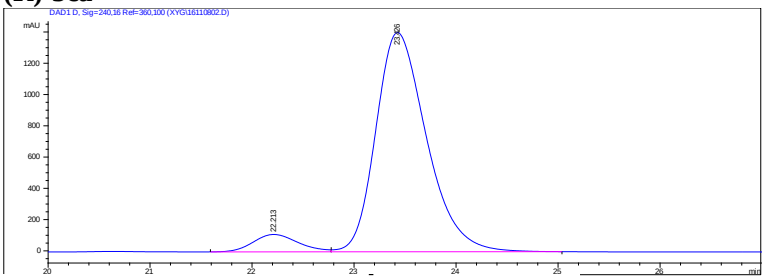


Signal 3: DAD1 C, Sig=230,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	22.431	VV	0.5082	4.96082e4	1506.26306	49.1938
2	23.744	VB	0.5531	5.12342e4	1419.52588	50.8062

Totals : 1.00842e5 2925.78894

(R)-3ca



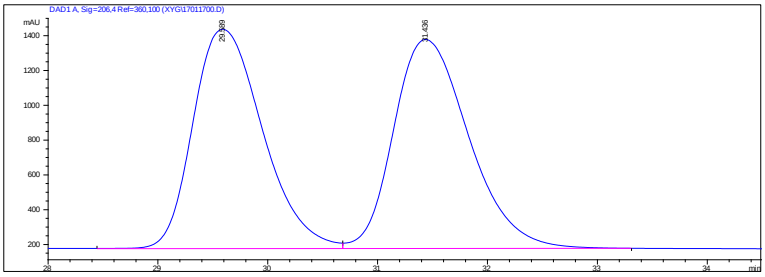
Signal 4: DAD1 D, Sig=240,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	22.213	BV	0.4739	3410.24609	111.17516	6.5818
2	23.426	VB	0.5306	4.84027e4	1402.63623	93.4182

Totals : 5.18130e4 1513.81139

87%

3da racemic

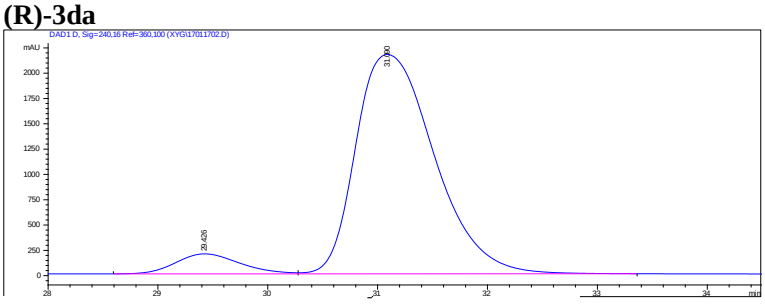


Signal 1: DAD1 A, Sig=206,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	29.589	BV	0.6699	5.55325e4	1260.75708	49.2744
2	31.436	VB	0.7270	5.71681e4	1201.72400	50.7256

Totals : 1.12701e5 2462.48108

(R)-3da



Signal 4: DAD1 D, Sig=240,16 Ref=360,100

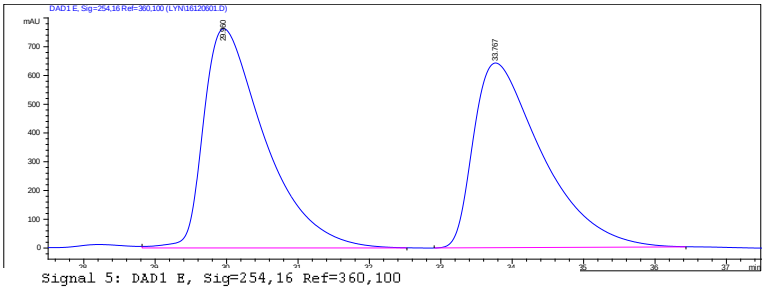
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	29.426	BV	0.6270	7997.13965	197.23593	6.9193
2	31.090	VB	0.7813	1.07580e5	2166.51807	93.0807

Totals : 1.15578e5 2363.75400

Results obtained with enhanced integrator!

86%

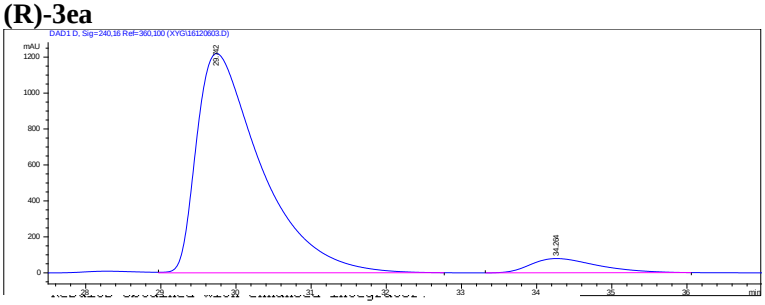
3ea racemic



Signal 5: DAD1 E, Sig=254,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	29.960	VB	0.8956	4.54453e4	762.21069	51.4219
2	33.767	PB	1.0020	4.29321e4	642.13495	48.5781
Totals :				8.83774e4	1404.34564	

(R)-3ea

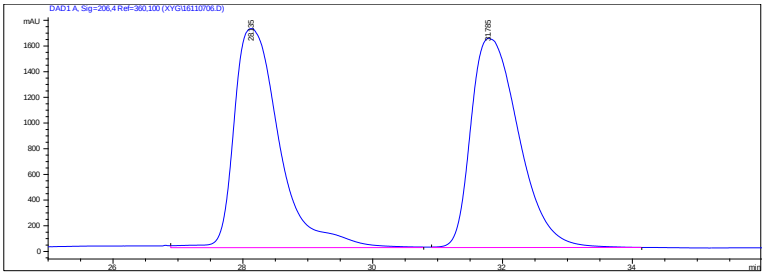


Signal 4: DAD1 D, Sig=240,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	29.742	VB	0.8893	7.27165e4	1220.18311	93.6696
2	34.264	BB	0.9499	4914.35547	79.20382	6.3304
Totals :				7.76309e4	1299.38692	

87%

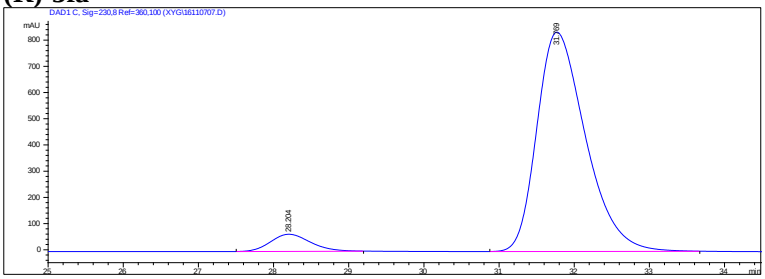
3fa racemic



Signal 1: DAD1 A, Sig=206,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	28.135	VB	0.6667	8.28670e4	1705.85767	49.8324
2	31.785	BB	0.6947	8.34243e4	1630.97766	50.1676
Totals :				1.66291e5	3336.83533	

(R)-3fa

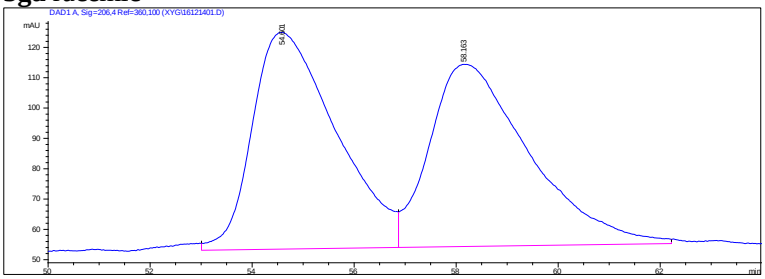


Signal 3: DAD1 C, Sig=230,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	28.204	BB	0.5719	2423.63452	65.46365	6.0507
2	31.769	BB	0.6906	3.76320e4	836.89313	93.9493
Totals :				4.00556e4	902.35678	

88%

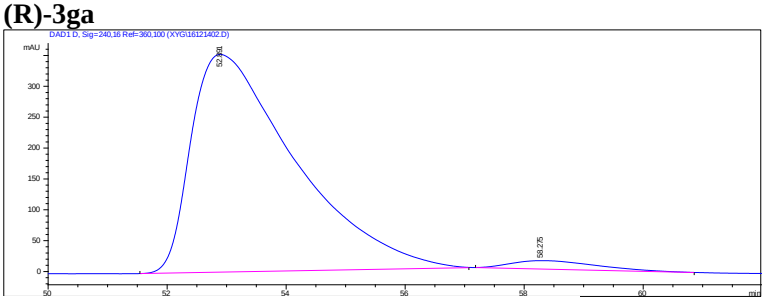
3ga racemic



Signal 1: DAD1 A, Sig=206,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	54.601	BV	1.3554	8168.49121	71.47189	50.0574
2	58.163	VV	1.6032	8149.76172	60.05766	49.9426
Totals :				1.63183e4	131.52955	

(R)-3ga

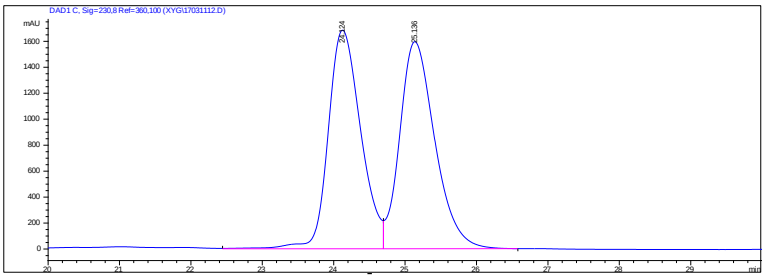


Signal 4: DAD1 D, Sig=240,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	52.891	PB	1.7359	4.27516e4	352.76523	96.9191
2	58.275	PB	1.1909	1358.99487	13.49427	3.0809
Totals :				4.41106e4	366.25950	

94%

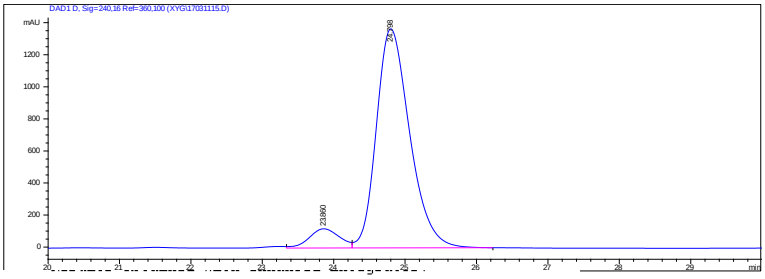
3ha racemic



Signal 3: DAD1 C, Sig=230,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	24.124	VV	0.4870	5.32445e4	1683.29224	49.4414
2	25.136	VP	0.5205	5.44477e4	1594.15417	50.5586
Totals :				1.07692e5	3277.44641	

(R)-3ha

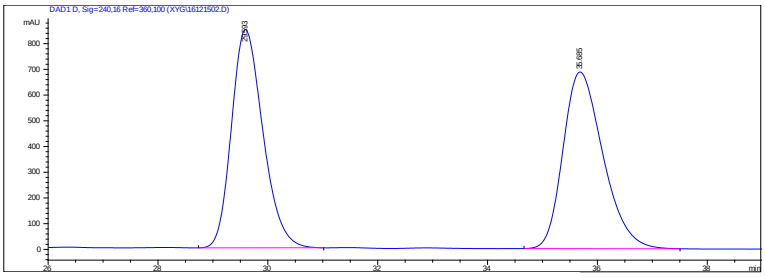


Signal 4: DAD1 D, Sig=240,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	23.860	VV	0.4521	3510.99121	119.74337	7.3437
2	24.798	VB	0.4982	4.42988e4	1366.34387	92.6563
Totals :				4.78098e4	1486.08724	

85%

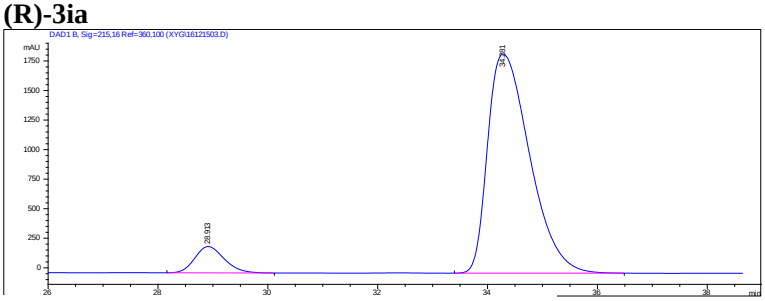
3ia racemic



Signal 4: DAD1 D, Sig=240,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	29.593	PP	0.6143	3.37572e4	848.19000	49.3021
2	35.685	BB	0.7775	3.47129e4	686.98505	50.6979
Totals :				6.84701e4	1535.17505	

(R)-3ia

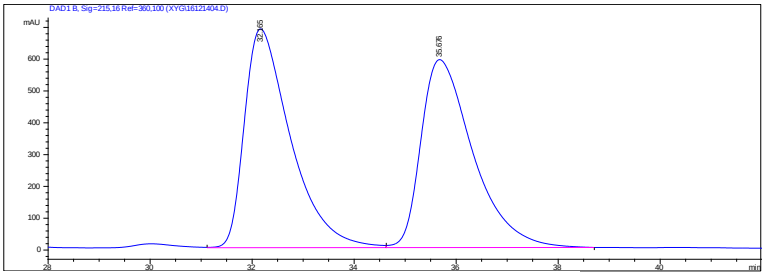


Signal 2: DAD1 B, Sig=215,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	28.913	BB	0.5783	8325.38379	222.61223	7.7857
2	34.281	BB	0.7260	9.86059e4	1855.47852	92.2143
Totals :				1.06931e5	2078.09074	

84%

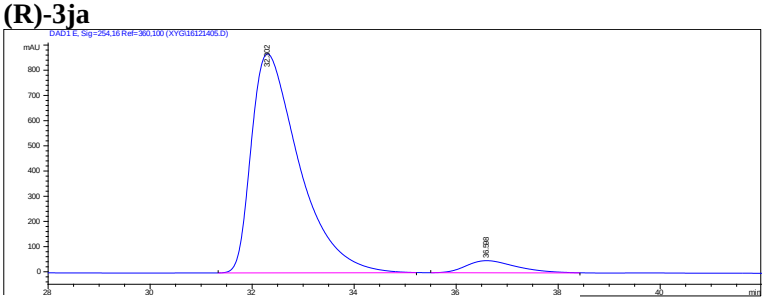
3ja racemic



Signal 2: DAD1 B, Sig=215,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	32.165	VB	0.9552	4.33530e4	687.98425	50.8602
2	35.676	BB	1.0810	4.18865e4	590.90735	49.1398
Totals :				8.52395e4	1278.89160	

(R)-3ja

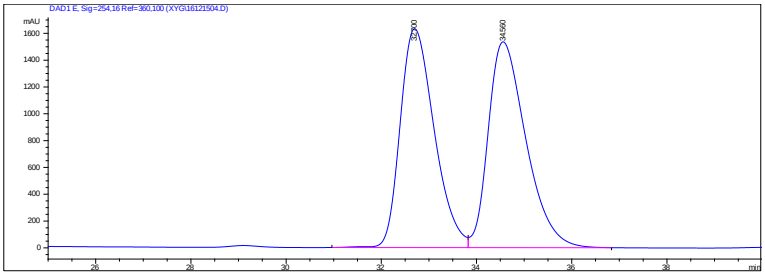


Signal 5: DAD1 E, Sig=254,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	32.302	BB	0.9937	5.65892e4	869.04218	94.5369
2	36.598	PB	1.0062	3270.15332	48.04095	5.4631
Totals :				5.98593e4	917.08313	

89%

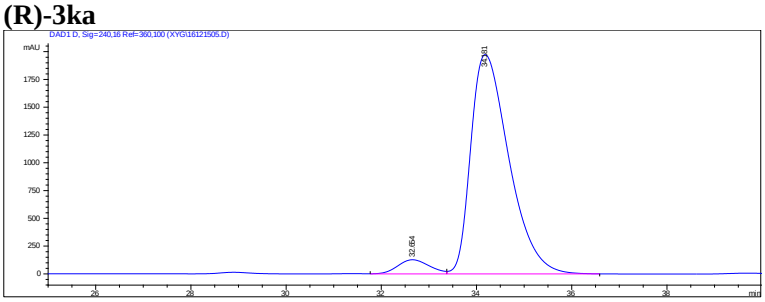
3ka racemic



Signal 5: DAD1 E, Sig=254,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	32.700	BV	0.7696	8.11329e4	1632.99890	49.0009
2	34.560	VB	0.8458	8.44414e4	1534.94373	50.9991
Totals :				1.65574e5	3167.94263	

(R)-3ka

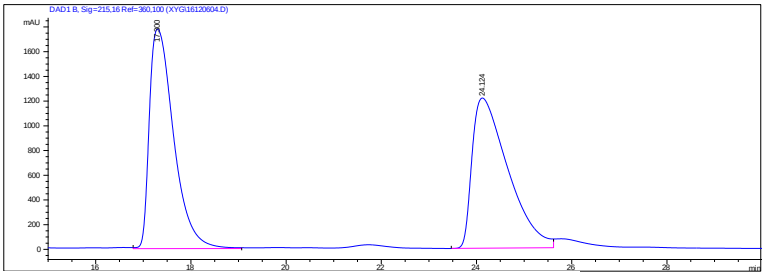


Signal 4: DAD1 D, Sig=240,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	32.654	BV	0.7197	5857.86914	127.11130	5.0550
2	34.181	VB	0.8525	1.10025e5	1973.31018	94.9450
Totals :				1.15883e5	2100.42148	

90%

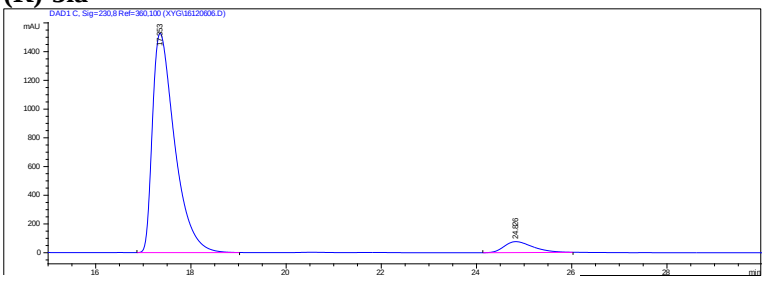
3la racemic



Signal 2: DAD1 B, Sig=215,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	17.300	VB	0.5491	6.29238e4	1776.90527	49.7256
2	24.124	PV	0.7944	6.36183e4	1215.97546	50.2744
Totals :				1.26542e5	2992.88074	

(R)-3la

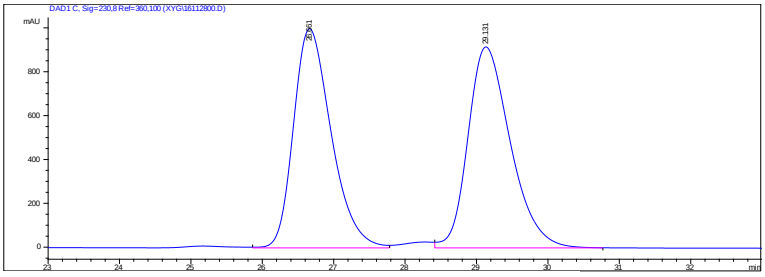


Signal 3: DAD1 C, Sig=230,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	17.353	PB	0.4915	4.94019e4	1526.86975	93.9093
2	24.826	PB	0.6419	3204.06299	76.29687	6.0907
Totals :				5.26060e4	1603.16662	

88%

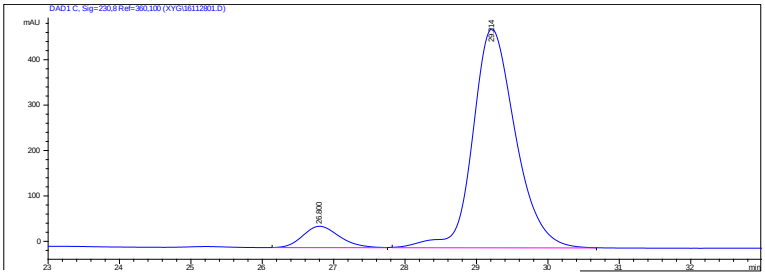
3ma racemic



Signal 3: DAD1 C, Sig=230,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	26.661	VV	0.5631	3.65694e4	998.85596	49.1720
2	29.131	VB	0.6335	3.78011e4	915.95386	50.8280
Totals :				7.43705e4	1914.80981	

(R)-3ma

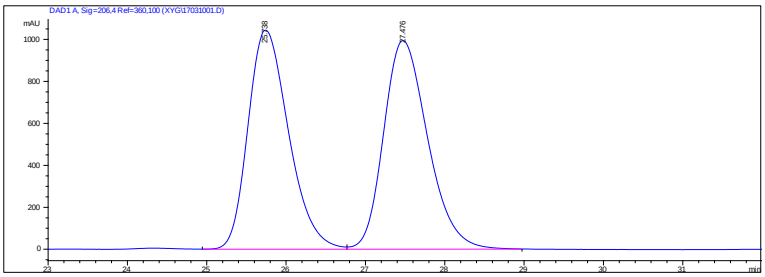


Signal 3: DAD1 C, Sig=230,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	26.800	BB	0.5460	1667.37976	46.98969	7.6923
2	29.214	BB	0.6321	2.00086e4	482.18597	92.3077
Totals :				2.16759e4	529.17566	

85%

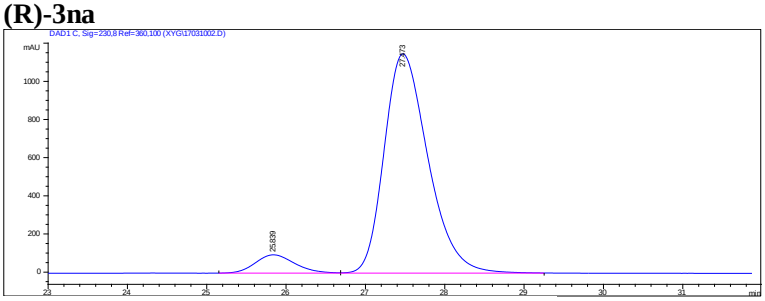
3na racemic



Signal 1: DAD1 A, Sig=206,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	25.738	VV	0.5413	3.71480e4	1043.61462	49.2132
2	27.476	VB	0.6001	3.83358e4	993.68890	50.7868
Totals :				7.54838e4	2037.30353	

(R)-3na

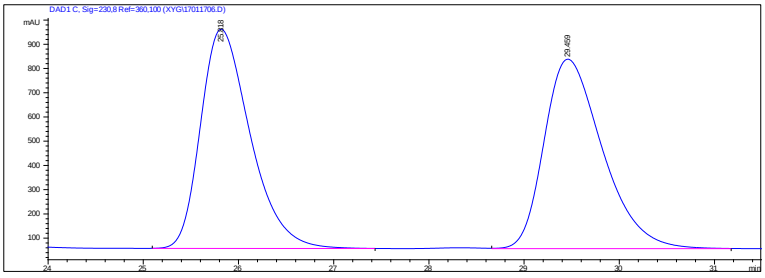


Signal 3: DAD1 C, Sig=230,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	25.839	BV	0.5363	3327.06055	96.01756	7.0123
2	27.473	VB	0.5879	4.41192e4	1149.20386	92.9877
Totals :				4.74463e4	1245.22141	

86%

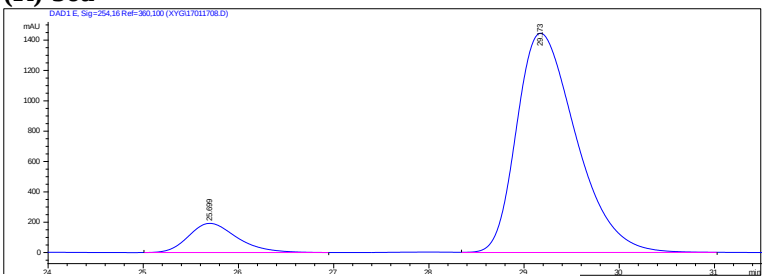
3oa racemic



Signal 3: DAD1 C, Sig=230,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	25.818	BB	0.5567	3.27718e4	904.54584	49.6236
2	29.459	VB	0.6566	3.32690e4	781.55206	50.3764
Totals :				6.60407e4	1686.09790	

(R)-3oa

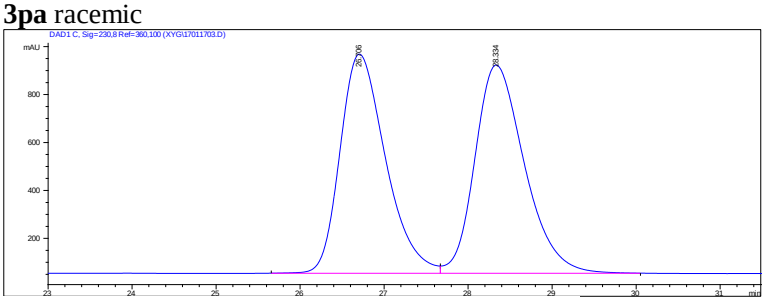


Signal 5: DAD1 E, Sig=254,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	25.699	BB	0.5437	6859.64404	192.50371	9.8804
2	29.173	VB	0.6712	6.25672e4	1444.77173	90.1196
Totals :				6.94269e4	1637.27544	

80%

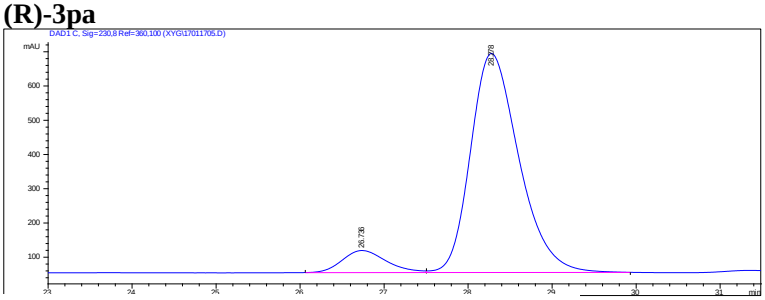
3pa racemic



Signal 3: DAD1 C, Sig=230,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	26.706	BV	0.5805	3.43644e4	914.23547	49.1503
2	28.334	VB	0.6295	3.55525e4	868.65320	50.8497
Totals :				6.99169e4	1782.88867	

(R)-3pa

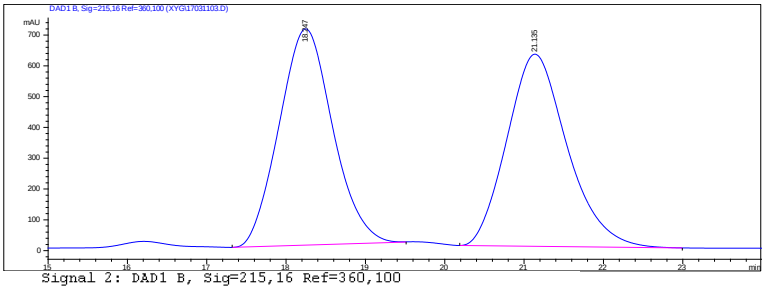


Signal 3: DAD1 C, Sig=230,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	26.736	BV	0.5584	2362.33154	64.62790	8.4552
2	28.278	VB	0.6144	2.55771e4	639.80115	91.5448
Totals :				2.79394e4	704.42905	

83%

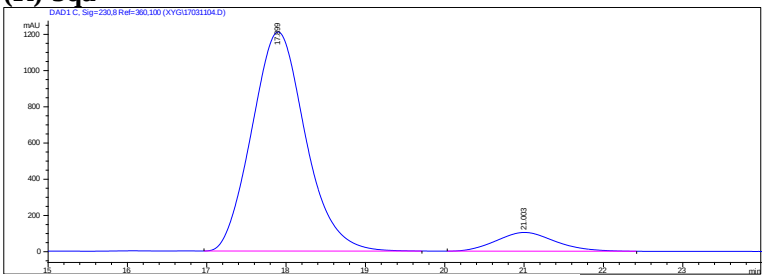
3qa racemic



Signal 2: DAD1 B, Sig=215,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	18.247	PB	0.7219	3.29713e4	702.08234	50.0175
2	21.135	BB	0.7942	3.29482e4	623.79816	49.9825
Totals :				6.59195e4	1325.88049	

(R)-3qa

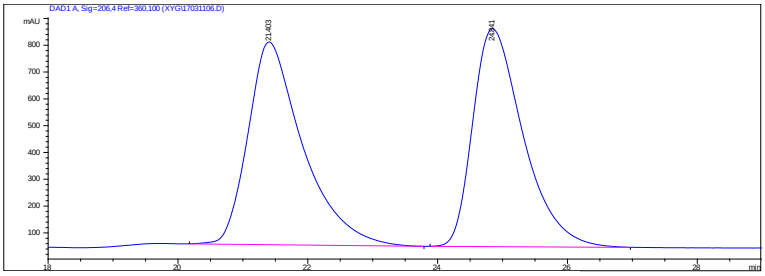


Signal 3: DAD1 C, Sig=230,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	17.899	PB	0.7232	5.67148e4	1209.24841	91.3810
2	21.003	PB	0.7803	5349.32324	103.60892	8.6190
Totals :				6.20642e4	1312.85733	

83%

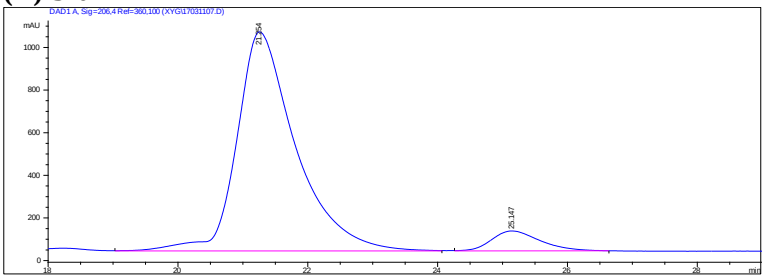
3ra racemic



Signal 1: DAD1 A, Sig=206,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	21.403	BP	0.8101	4.44532e4	755.90295	50.3268
2	24.841	BB	0.7956	4.38759e4	813.00232	49.6732
Totals :				8.83291e4	1568.90527	

(R)-3ra



Signal 1: DAD1 A, Sig=206,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	21.254	VB	0.8226	6.47181e4	1027.80640	93.0412
2	25.147	VB	0.6480	4840.41650	93.48956	6.9588
Totals :				6.95585e4	1121.29596	

86%