Asymmetric Synthesis of Bicyclic Dihydropyrans *via* Organo-catalytic Inverse-Electron-Demand oxo-Diels-Alder Reactions of Enolizable Aliphatic Aldehydes

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Supporting Information

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1. General Information

General Procedures. All reactions were performed in oven-dried or flame-dried reaction vessels, modified Schlenk flasks, or round-bottom flasks. The flasks were fitted with Teflon screw caps and reactions were conducted under an atmosphere of argon if needed. Gas-tight syringes with stainless steel needles were used to transfer air- and moisture-sensitive liquids. All moisture and/or air sensitive solid compounds were manipulated inside normal desiccators. Flash column chromatography was performed using silica gel (40–63 μm, 230–400 mesh).

Analytical thin layer chromatography (TLC) was performed on silica gel 60 F_{254} aluminum plates (Merck) containing a 254 nm fluorescent indicator. TLC plates were visualized by exposure to short wave ultraviolet light (254 nm) and to a solution of $KMnO_4$ (1 g of $KMnO_4$, 6 g of K_2CO_3 and 0.1 g of KOH in 100 mL of H_2O) or vanillin (2 g of vanillin and 4 mL of concentrated H_2SO_4 in 100 mL of EtOH) followed by heating.

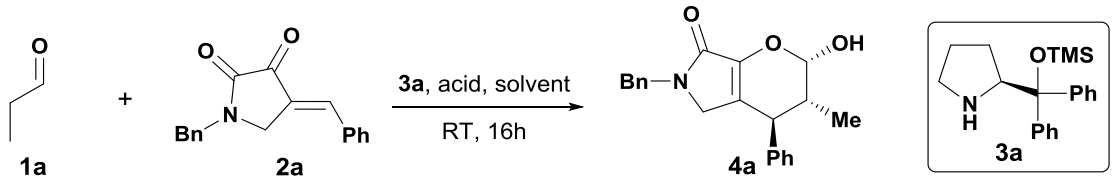
Organic solutions were concentrated at 30-50 ℃ on rotary evaporators at ~10 torr followed by drying on vacuum pump at ~1 torr. Reaction temperatures are reported as the temperature of the bath surrounding the vessel unless otherwise stated.

Materials. Commercial reagents and solvents were were obtained from Adamas-beta, Aldrich Chemical Co., Alfa Aesar, Macklin and Energy Chemical and used as received with the following exceptions: THF, Et_2O and toluene were purified by refluxing over Na-benzophenone under positive argon pressure followed by distillation. [1] The enone substrates were prepared according to literature procedure. [2]

Instrumentation.

- Proton nuclear magnetic resonance (^{1}H NMR) spectra were recorded with Bruker AV 400 MHz spectrometers. Proton chemical shifts are reported in parts per million (δ scale), and are referenced using residual protium in the NMR solvent ($CDCl_3$: δ 7.26 ($CHCl_3$)). Data are reported as follows: chemical shift [multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br s = broad singlet), coupling constant(s) (Hz), integration].
- Carbon-13 nuclear magnetic resonance (^{13}C NMR) spectra were recorded with Bruker AV 400 MHz spectrometers. Carbon chemical shifts are reported in parts per million (δ scale), and are referenced using the carbon resonances of the solvent (δ 77.0 ($CHCl_3$)). Data are reported as follows: chemical shift [multiplicity (if not singlet), assignment (C_q = fully substituted carbon)].
- High resolution mass spectra (HRMS) were recorded on a Waters SYNAPT G2 using an electrospray (ESI) ionization source.

2. Optimization Study of the Asymmetric *oxo*-IEDDA Reactions of Aldehyde 1a and the Cyclic Enone 2a[a]

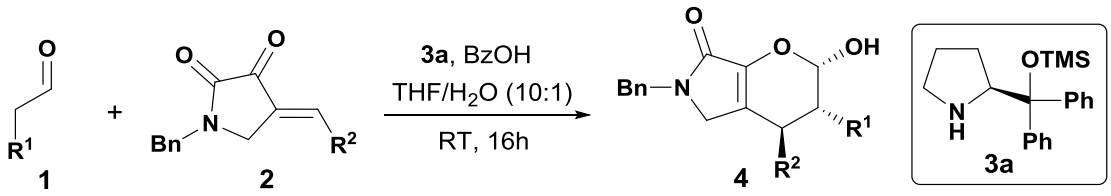


Entry	Solvent	Acid [b]	yield[%] [c]	*ee* [%] [d]
1	DCM	BA	70	98
2	THF	BA	40	>99
3	CH_3CN	BA	30	96
4	Toluene	BA	20	99
5	1,4-dioxane	BA	82	>99
6	Chloroform	BA	64	99
7	**THF:H_2O (10:1)**	**BA**	**90**	**>99**
8	CH_3CN:H_2O (10:1)	BA	75	98
9	1,4-dioxane:H_2O (10:1)	BA	80	>99
10	THF:H_2O (10:1)	None	N.D	trace
11	THF:H_2O (10:1)	PNBA	92	97
12	THF:H_2O (10:1)	ONBA	81	99
13	THF:H_2O (10:1)	SA	80	99
14	THF:H_2O (10:1)	AA	78	99
15 [e]	THF:H_2O (10:1)	BA	70	>99
16 [f]	THF:H_2O (10:1)	BA	82	99

[a] Unless otherwise noted, the title reacitons were performed with 0.15 mmol of **1a**, 0.1 mmol of **2a**, 0.02 mmol of **3a** and 0.02 mmol of the acid in 1 mL of the solvent for 16 hours; and the dr value of this reaction was generally >95:5 which was determined by ^{1}H-NMR analysis of the crude product. [b] BA: Benzoic acid; PNBA: *p*-Nitrobenzoc acid; ONBA: *o*-Nitrobenzoc acid; SA: Salicylic acid; AA: Acetic acid. [c] Isolated yield. [d] Determined by chiral HPLC analysis. [e] Reaction was performed in 0.5 ml of the solvent. [f] Reaction was performed in 2 ml of the solvent.

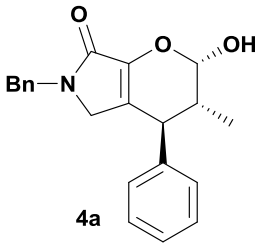
We firstly screened the solvent for the secondary amine catalyzed asymmetric *oxo*-IEDDA reaction of *n*-propanal **1a** and the cyclic enone **2a** under room temperature. As summarized in the above table (entries 1-9), the co-solvent of THF/H_2O (10:1) has demonstrated to be the optimal choice. Then, various kinds of acid additives including benzoic acid, *p*-nitrobenzoc acid, *o*-nitrobenzoc acid, salicylic acid and acetic acid, were also investigated (entries 10-14). Regarding the isolated yield, enantioselectivity as well as the cost of the material, we chose benzoic acid as the best additive for this reaction; it is noteworthy that no reaction happened in the absence of acid (entry 10). Finally, we studied the concentration of this reaction; however, lower yield was obtained by either increasing or reducing the amount of the solvent (entries 15-16). Thus, the desired product **4a** could be obtained in high yield (90% yield) and with excellent stereoselectivity (>95:5 dr, >99% ee) under the optimal condition (entry 7).

3. General Procedure for the Synthesis of Chiral Bicyclic Dihydropyranes 4 by using Normal Saturated Aldehydes



A glass tube was charged with pyrrolidine-2,3-dione **2** (0.2 mmol), amine catalyst **3a** (0.04 mmol, 13 mg) and benzoic acid (0.04 mmol, 4.9 mg) in THF/H_2O (*v*/*v* = 10:1, 0.1 M, 2 mL). The saturated aldehydes **1** (0.3 mmol) was added with a syringe, and the reaction was sealed with a Teflon cap and stirred at room temperature for about 16 hours. When the reaction was complete, the mixture was directly purified by column chromatography on silica gel eluting with petroleum ether/ethyl acetate (6:1 to 2:1) to afford the corresponding bicyclic dihydropyranes **4**, which was dried under vacuum and further analyzed by ^{1}H-NMR, ^{13}C-HMR, HRMS, chiral HPLC analysis, *etc*.

(2*S*,3*R*,4*S*)-6-benzyl-2-hydroxy-3-methyl-4-phenyl-3,4,5,6-tetrahydropyrano[2,3-*c*]pyrrol-7(2*H*)-one 4a



Prepared according to the general procedure using *n*-propanal (17.4 mg, 0.3 mmol, 1.5 equiv) and 1-benzyl-4-benzylidenepyrrolidine-2,3-dione **2a** (55.4 mg, 0.2 mmol, 1.0 equiv). Purification of the crude product via column chromatography delivered **4a** as a white solid with 90% yield. The diastereomeric ratio was determined to be >95:5 by crude ^{1}H-NMR analysis, and the enantiomeric excess of the major product was determined to be >99% by chiral HPLC analysis on Chiralpak AD-H column (25% 2-propanol/*n*-hexane, 1 mL/min), UV 220 nm, t_{major} = 6.48 min, t_{minor} = 12.19 min; $[\alpha]_D^{20}$ = 86.4 (*c* = 1.0 in CH_2Cl_2).

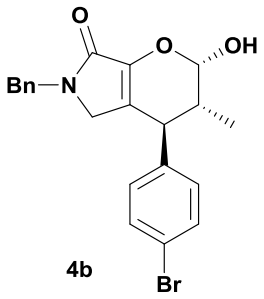
NMR and HRMS data for the product **4a**:

^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm): 7.32 – 7.26 (m, 4H), 7.25 – 7.22 (m, 2H), 7.19 – 7.16 (m, 2H), 7.14 – 7.12 (m, 2H), 5.66 (d, *J* = 2.4 Hz, 1H), 4.73 (d, *J* = 15.2 Hz, 1H), 4.41 (d, *J* = 15.2 Hz, 1H), 3.53 – 3.36 (m, 3H), 2.16 – 2.07 (m, 1H), 0.96 (d, *J* = 6.8 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm): 166.9, 142.4, 140.5, 136.7, 128.7, 128.6, 128.4, 128.0, 127.6, 127.1, 124.3, 96.9, 47.9, 46.5, 41.5, 39.7, 14.2

HRMS (ESI): *m/z* calculated for $C_{21}H_{21}NO_3Na^+$: 358.1419, found: 358.1417.

(2*S*,3*R*,4*S*)-6-benzyl-4-(4-bromophenyl)-2-hydroxy-3-methyl-3,4,5,6-tetrahydropyrano[2,3-*c*] pyrrol-7(2*H*)-one 4b



Prepared according to the general procedure using *n*-propanal (17.4 mg, 0.3 mmol, 1.5 equiv) and 1-benzyl-4-(4-bromobenzylidene)-pyrrolidine-2,3-dione **2b** (71.2 mg, 0.2 mmol, 1.0 equiv). Purification of the crude product via column chromatography delivered **4b** as a white solid with 92% yield. The diastereomeric ratio was determined to be 91:9 by crude ^{1}H-NMR analysis, and the enantiomeric excess of the major product was determined to be >99% by chiral HPLC analysis on Chiralpak AD-H column (25% 2-propanol/*n*-hexane, 1 mL/min), UV 220 nm, t_{major} = 6.43 min, t_{minor} = 11.10 min; $[\alpha]_D^{20}$ = +46.9 (*c* = 1.60 in CH_2Cl_2).

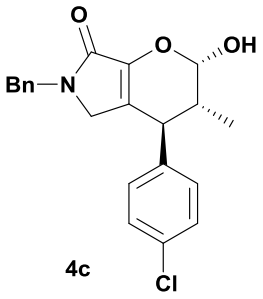
NMR and HRMS data for the product **4b**:

^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm): 7.43 (d, *J* = 8.4 Hz, 2H), 7.32 – 7.23 (m, 3H), 7.22 – 7.13 (m, 2H), 7.02 (d, *J* = 8.4 Hz, 2H), 6.24 (br s, 1H), 5.71 (br s, 1H), 4.69 (d, *J* = 14.8 Hz, 1H), 4.44 (d, *J* = 14.8 Hz, 1H), 3.50 – 3.44 (m, 2H), 3.35 (d, *J* = 18.0 Hz, 1H), 2.11 – 2.03 (m, 1H), 0.95 (d, *J* = 6.8 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm): 166.7, 142.7, 139.5, 136.6, 131.9, 130.2, 128.7, 128.1, 127.7, 123.3, 121.0, 96.8, 47.8, 46.6, 41.1, 39.8, 14.2

HRMS (ESI): *m/z* calculated for $C_{21}H_{20}BrNO_3Na^+$: 436.0524, found: 436.0527.

(2*S*,3*R*,4*S*)-6-benzyl-4-(4-chlorophenyl)-2-hydroxy-3-methyl-3,4,5,6-tetrahydropyrano[2,3-c] pyrrol-7(2*H*)-one 4c



4c

Prepared according to the general procedure using *n*-propanal (17.4 mg, 0.3 mmol, 1.5 equiv) and 1-benzyl-4-(4-chlorobenzylidene)-pyrrolidine-2,3-dione **2c** (62.2 mg, 0.2 mmol, 1.0 equiv). Purification of the crude product via column chromatography delivered **4c** as a white solid with 93% yield. The diastereomeric ratio was determined to be 90:10 by crude ^{1}H-NMR analysis, and the enantiomeric excess of the major product was determined to be 99% by chiral HPLC analysis on Chiralpak AD-H column (25% 2-propanol/*n*-hexane, 1 mL/min), UV 220 nm, t_{major} = 6.36 min, t_{minor} = 10.88 min; $[\alpha]_D^{20}$ = +68.3 (*c* = 1.14 in CH_2Cl_2).

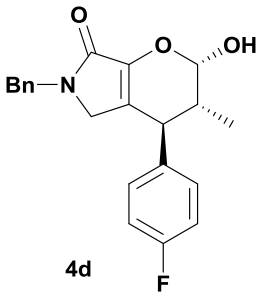
NMR and HRMS data for the product **4c**:

^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm): 7.24 – 7.18 (m, 5H), 7.12 – 7.09 (m, 2H), 7.02 – 6.98 (m, 2H), 6.11 (br s, 1H), 5.63 (d, *J* = 2.4 Hz, 1H), 4.62 (d, *J* = 14.8 Hz, 1H), 4.37 (d, *J* = 14.8 Hz, 1H), 3.44 – 3.37 (m, 2H), 3.28 (d, *J* = 18.4 Hz, 1H), 2.04 – 1.96 (m, 1H), 0.88 (d, *J* = 6.8 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm): 166.7, 142.6, 139.0, 136.6, 133.0, 129.8, 129.0, 128.7, 128.1, 127.7, 123.5, 96.8, 47.8, 46.6, 41.0, 39.8, 14.1

HRMS (ESI): *m/z* calculated for $C_{21}H_{20}ClNO_3Na^+$: 392.1029, found: 392.1029.

(2*S*,3*R*,4*S*)-6-benzyl-4-(4-fluorophenyl)-2-hydroxy-3-methyl-3,4,5,6-tetrahydropyrano[2,3-*c*] pyrrol-7(2*H*)-one 4d



4d

Prepared according to the general procedure using *n*-propanal (17.4 mg, 0.3 mmol, 1.5 equiv) and 1-benzyl-4-(4-fluorobenzylidene)-pyrrolidine-2,3-dione **2d** (59.0 mg, 0.2 mmol, 1.0 equiv). Purification of the crude product via column chromatography delivered **4d** as a white solid with 98% yield. The diastereomeric ratio was determined to be 89:11 by crude ^{1}H-NMR analysis, and the enantiomeric excess of the major product was determined to be 99% by

chiral HPLC analysis on Chiralpak AD-H column (25% 2-propanol/*n*-hexane, 1 mL/min), UV 220 nm, t_{major} = 6.45 min, t_{minor} = 11.48 min; $[\alpha]_D^{20}$ = +68.9 (*c* = 1.51 in CH_2Cl_2).

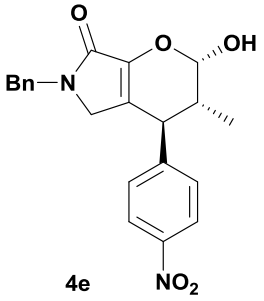
NMR and HRMS data for the product **4d**:

^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm): 7.31 – 7.23 (m, 3H), 7.21 – 7.17 (m, 2H), 7.12 – 7.08 (m, 2H), 7.02 – 6.97 (m, 2H), 6.31 (br s, 1H), 5.71 (d, *J* = 2.4 Hz, 1H), 4.69 (d, *J* = 14.8 Hz, 1H), 4.44 (d, *J* = 14.8 Hz, 1H), 3.53 – 3.45 (m, 2H), 3.35 (d, *J* = 18.4 Hz, 1H), 2.11 – 2.03 (m, 1H), 0.95 (d, *J* = 6.8 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm): 166.8, 163.1, 160.7, 142.5, 136.7, 136.1, 136.0, 129.8, 128.7, 128.1, 127.6, 123.8, 115.8, 115.6, 96.8, 47.8, 46.6, 40.9, 39.9, 14.2

HRMS (ESI): *m/z* calculated for $C_{21}H_{20}FNO_3Na^+$: 376.1325, found: 376.1325.

(2*S*,3*R*,4*S*)-6-benzyl-2-hydroxy-3-methyl-4-(4-nitrophenyl)-3,4,5,6-tetrahydropyrano[2,3-*c*] pyrrol-7(2*H*)-one 4e



Prepared according to the general procedure using *n*-propanal (17.4 mg, 0.3 mmol, 1.5 equiv) and 1-benzyl-4-(4-nitrobenzylidene)-pyrrolidine-2,3-dione **2e** (64.4 mg, 0.2 mmol, 1.0 equiv). Purification of the crude product via column chromatography delivered **4e** as a white solid with 98% yield. The diastereomeric ratio was determined to be >95:5 by crude ^{1}H-NMR analysis, and the enantiomeric excess of the major product was determined to be >99% by chiral HPLC analysis on Chiralpak AD-H column (25% 2-propanol/*n*-hexane, 1 mL/min), UV 220 nm, t_{major} = 9.68 min, t_{minor} = 15.75 min; $[\alpha]_D^{20}$ = +54.8 (*c* = 0.99 in CH_2Cl_2).

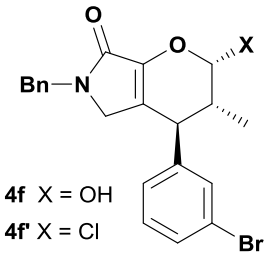
NMR and HRMS data for the product **4e**:

^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm): 8.18 (d, *J* = 8.8 Hz, 2H), 7.35 – 7.26 (m, 5H), 7.18 (d, *J* = 8.0 Hz, 2H), 6.74 (br s, 1H), 5.76 (br s, 1H), 4.67 (d, *J* = 14.8 Hz, 1H), 4.48 (d, *J* = 14.8 Hz, 1H), 3.69 (d, *J* = 10.8 Hz, 1H), 3.49 (d, *J* = 18.4 Hz, 1H), 3.34 (d, *J* = 18.4 Hz, 1H), 2.18 – 2.11 (m, 1H), 0.98 (d, *J* = 6.8 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm): 166.6, 148.4, 147.3, 143.1, 136.4, 129.3, 128.8, 128.1, 127.8, 124.1, 122.1, 96.6, 47.7, 46.6, 41.7, 39.9, 14.2

HRMS (ESI): *m/z* calculated for $C_{21}H_{20}N_2O_5Na^+$: 403.1270, found: 403.1269.

(2*S*,3*R*,4*S*)-6-benzyl-4-(3-bromophenyl)-2-hydroxy-3-methyl-3,4,5,6-tetrahydropyrano[2,3-*c*] pyrrol-7(2*H*)-one 4f



Prepared according to the general procedure using *n*-propanal (17.4 mg, 0.3 mmol, 1.5 equiv) and 1-benzyl-4-(3-bromobenzylidene)-pyrrolidine-2,3-dione **2f** (71.2 mg, 0.2 mmol, 1.0 equiv). Purification of the crude product via column chromatography delivered **4f** as a white solid with 94% yield. The diastereomeric ratio was determined to be 90:10 by crude ^{1}H-NMR analysis, and the diastereoisomers cannot be separated by simple column chromatography; in order to get clean NMR and HPLC spectrum, **4f** was transformed to its analogue **4f'** [3], thus the enantiomeric excess of **4f'** was determined to be >99% by chiral HPLC analysis on Chiralpak AD-H column (10% 2-propanol/*n*-hexane, 1 mL/min), UV 220 nm, t_{major} = 36.58 min, t_{minor} = 39.84 min; $[\alpha]_D^{20}$ = +110.5 (*c* = 1.76 in CH_2Cl_2, *data for* **4f`**).

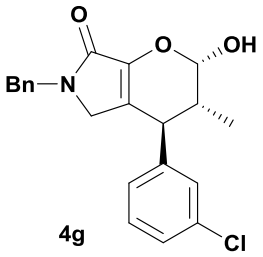
NMR and HRMS data for the product **4f'** :

^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm): 7.44 – 7.41 (m, 1H), 7.33 – 7.26 (m, 4H), 7.21 – 7.18 (m, 3H), 7.07 – 7.05 (m, 1H), 6.20 (d, *J* = 2.4 Hz, 1H), 4.75 (d, *J* = 14.8 Hz, 1H), 4.43 (d, *J* = 14.8 Hz, 1H), 3.56 – 3.47 (m, 2H), 3.36 (d, *J* = 18.4 Hz, 1H), 2.44 – 2.36 (m, 1H), 0.95 (d, *J* = 6.8 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm): 164.1, 141.9, 141.1, 136.5, 131.0, 130.6, 128.7, 127.9, 127.7, 125.1, 123.1, 94.1, 47.2, 46.5, 41.8, 41.7, 14.7

HRMS (ESI): *m/z* calculated for $C_{21}H_{19}BrClNO_2Na^+$: 454.0185, found: 454.0186.

(2*S*,3*R*,4*S*)-6-benzyl-4-(3-chlorophenyl)-2-hydroxy-3-methyl-3,4,5,6-tetrahydropyrano[2,3-*c*] pyrrol-7(2*H*)-one 4g



Prepared according to the general procedure using *n*-propanal (17.4 mg, 0.3 mmol, 1.5 equiv) and 1-benzyl-4-(3-chlorobenzylidene)-pyrrolidine-2,3-dione **2g** (62.2 mg, 0.2 mmol, 1.0 equiv). Purification of the crude product via column chromatography delivered **4g** as a white solid with 95% yield. The diastereomeric ratio was determined to be 91:9 by crude ^{1}H-NMR analysis, and the enantiomeric excess of the major product was determined to be >99% by chiral HPLC analysis on Chiralpak AD-H column (25% 2-propanol/*n*-hexane, 1 mL/min), UV 220 nm, t_{major} = 6.16 min, t_{minor} = 12.18 min; $[\alpha]_D^{20}$ = +57.1 (*c* = 1.38 in CH_2Cl_2).

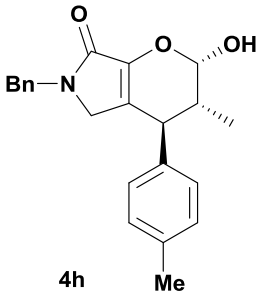
NMR and HRMS data for the product **4g**:

^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm): 7.32 – 7.23 (m, 5H), 7.20 – 7.17 (m, 2H), 7.13 – 7.12 (m, 1H), 7.04 – 7.01 (m, 1H), 5.90 (br s, 1H), 5.70 (br s, 1H), 4.74 (d, *J* = 14.8 Hz, 1H), 4.42 (d, *J* = 14.8 Hz, 1H), 3.50 – 3.46 (m, 2H), 3.36 (d, *J* = 18.4 Hz, 1H), 2.14 – 2.06 (m, 1H), 0.97 (d, *J* = 6.8 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm): 166.6, 142.7, 142.6, 136.7, 134.7, 130.1, 128.7, 128.1, 128.0, 127.9, 127.7, 127.5, 123.2, 96.7, 47.7, 46.5, 41.4, 39.7, 14.2

HRMS (ESI): *m/z* calculated for $C_{21}H_{20}ClNO_3Na^+$: 392.1029, found: 392.1023.

(2*S*,3*R*,4*S*)-6-benzyl-2-hydroxy-3-methyl-4-(*p*-tolyl)-3,4,5,6-tetrahydropyrano[2,3-*c*]pyrrol-7(2*H*)-one 4h



Prepared according to the general procedure using *n*-propanal (17.4 mg, 0.3 mmol, 1.5 equiv) and 1-benzyl-4-(4-methylbenzylidene)-pyrrolidine-2,3-dione **2h** (58.2 mg, 0.2 mmol, 1.0 equiv). Purification of the crude product via column chromatography delivered **4h** as a white solid with 96% yield. The diastereomeric ratio was determined to be 90:10 by crude ^{1}H-NMR analysis, and the enantiomeric excess of the major product was determined to be >99% by chiral HPLC analysis on Chiralpak AD-H column (25% 2-propanol/*n*-hexane, 1 mL/min), UV 220 nm, t_{major} = 5.87 min, t_{minor} = 9.94 min; $[\alpha]_D^{20}$ = +52.5 (*c* = 1.71 in CH_2Cl_2).

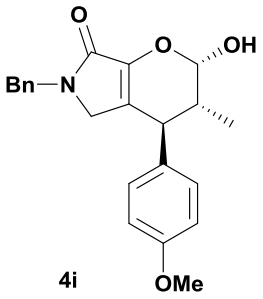
NMR and HRMS data for the product **4h**:

^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm): 7.51 – 7.44 (m, 3H), 7.39 – 7.37 (m, 2H), 7.31 (d, *J* = 8.0 Hz, 2H), 7.22 (d, *J* = 8.0 Hz, 2H), 5.92 (br s, 1H), 4.90 (d, *J* = 14.8 Hz, 1H), 4.64 (d, *J* = 14.8 Hz, 1H), 3.70 – 3.65 (m, 2H), 3.60 (d, *J* = 18.4 Hz, 1H), 2.53 (s, 3H), 2.33 – 2.28 (m, 1H), 1.16 (d, *J* = 6.8 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm): 166.9, 142.3, 137.4, 136.8, 136.7, 129.4, 128.6, 128.3, 128.0, 127.5, 124.5, 96.9, 48.0, 46.5, 41.1, 39.8, 21.0, 14.2

HRMS (ESI): *m/z* calculated for $C_{22}H_{23}NO_3Na^+$: 372.1576, found: 372.1575.

(2*S*,3*R*,4*S*)-6-benzyl-2-hydroxy-4-(4-methoxyphenyl)-3-methyl-3,4,5,6-tetrahydropyrano[2,3-*c*] pyrrol-7(2*H*)-one 4i



Prepared according to the general procedure using *n*-propanal (17.4 mg, 0.3 mmol, 1.5 equiv) and 1-benzyl-4-(4-methoxybenzylidene)-pyrrolidine-2,3-dione **2i** (61.4 mg, 0.2 mmol, 1.0 equiv). Purification of the crude product via column chromatography delivered **4i** as a white solid with 99% yield. The diastereomeric ratio was determined to be 92:8 by crude ^{1}H-NMR analysis, and the enantiomeric excess of the major product was determined to be >99% by chiral HPLC analysis on Chiralpak AD-H column (25% 2-propanol/*n*-hexane, 1 mL/min), UV 220 nm, t_{major} = 7.14 min, t_{minor} = 14.98 min; $[\alpha]_D^{20}$ = +58.2 (*c* = 1.66 in CH_2Cl_2).

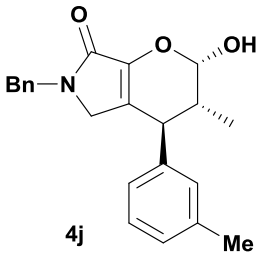
NMR and HRMS data for the product **4i**:

^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm): 7.51 – 7.45 (m, 3H), 7.39 – 7.37 (m, 2H), 7.25 (d, *J* = 8.4 Hz, 2H), 7.04 (d, *J* = 8.4 Hz, 2H), 5.91 (br s, 1H), 4.90 (d, *J* = 14.8 Hz, 1H), 4.64 (d, *J* = 14.8 Hz, 1H), 3.99 (s, 3H), 3.71 – 3.57 (m, 3H), 2.30 – 2.25 (m, 1H), 1.16 (d, *J* = 6.8 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm): 166.9, 158.6, 142.3, 136.8, 132.3, 129.3, 128.6, 128.0, 127.5, 124.6, 114.1, 96.9, 55.2, 47.9, 46.5, 40.7, 39.8, 14.2

HRMS (ESI): *m/z* calculated for $C_{22}H_{23}NO_4Na^+$: 388.1525, found: 388.1518.

(2*S*,3*R*,4*S*)-6-benzyl-2-hydroxy-3-methyl-4-(*m*-tolyl)-3,4,5,6-tetrahydropyrano[2,3-*c*]pyrrol-7(2*H*)-one 4h

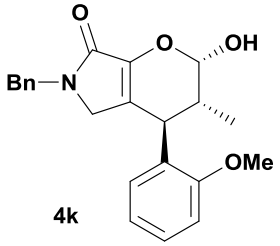


Prepared according to the general procedure using *n*-propanal (17.4 mg, 0.3 mmol, 1.5 equiv) and 1-benzyl-4-(3-methylbenzylidene)-pyrrolidine-2,3-dione **2j** (58.2 mg, 0.2 mmol, 1.0 equiv). Purification of the crude product via column chromatography delivered **4j** as a white solid with 86% yield. The diastereomeric ratio was determined to be 91:9 by crude ^{1}H-NMR analysis, and the enantiomeric excess of the major product was determined to be >99% by chiral HPLC analysis on Chiralpak AD-H column (25% 2-propanol/*n*-hexane, 1 mL/min), UV 220 nm, t_{major} = 5.52 min, t_{minor} = 9.56 min; $[\alpha]_D^{20}$ = +50.8 (*c* = 1.18 in CH_2Cl_2).

NMR and HRMS data for the product **4j**:

^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm): 7.30 – 7.24 (m, 3H), 7.20 – 7.16 (m, 3H), 7.05 (d, *J* = 7.6 Hz, 1H), 6.93 – 6.91 (m, 2H), 5.70 (br s, 1H), 4.71 (d, *J* = 14.8 Hz, 1H), 4.43 (d, *J* = 14.8 Hz, 1H), 3.50 – 3.36 (m, 3H), 2.31 (s, 3H), 2.15 – 2.08 (m, 1H), 0.96 (d, *J* = 6.8 Hz, 3H).
^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm): 166.9, 142.3, 137.4, 136.8, 136.7, 129.4, 128.6, 128.3, 128.0, 127.5, 124.5, 96.9, 48.0, 46.5, 41.1, 39.8, 21.0, 14.2
HRMS (ESI): *m/z* calculated for $C_{22}H_{23}NO_3Na^+$: 372.1576, found: 372.1575.

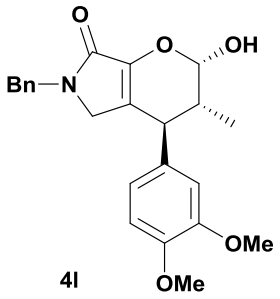
(2*S*,3*R*,4*S*)-6-benzyl-2-hydroxy-4-(2-methoxyphenyl)-3-methyl-3,4,5,6-tetrahydropyrano[2,3-*c*] pyrrol-7(2*H*)-one 4k



4k

Prepared according to the general procedure using *n*-propanal (17.4 mg, 0.3 mmol, 1.5 equiv) and 1-benzyl-4-(2-methoxybenzylidene)-pyrrolidine-2,3-dione **2k** (61.4 mg, 0.2 mmol, 1.0 equiv). Purification of the crude product via column chromatography delivered **4k** as a white solid with 94% yield. The diastereomeric ratio was determined to be 90:10 by crude ^{1}H-NMR analysis, and the enantiomeric excess of the major product was determined to be 96% by chiral HPLC analysis on Chiralpak AD-H column (25% 2-propanol/*n*-hexane, 1 mL/min), UV 220 nm, t_{major} = 8.00 min, t_{minor} = 17.83 min; $[\alpha]_D^{20}$ = +29.3 (*c* = 1.66 in CH_2Cl_2).
NMR and HRMS data for the product **4k**:
^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm): 7.51 – 7.43 (m, 4H), 7.39 – 7.37 (m, 2H), 7.25 (d, *J* = 7.6 Hz, 1H), 7.09 (t, *J* = 7.6 Hz, 1H), 7.04 (d, *J* = 8.0 Hz, 1H), 5.87 (br s, 1H), 4.78 (q, *J* = 15.2 Hz, 2H), 4.19 (d, *J* = 10.4 Hz, 1H), 3.91 (s, 3H), 3.70 (d, *J* = 18.4 Hz, 1H), 3.56 (d, *J* = 18.4 Hz, 1H), 2.48 – 2.44 (m, 1H), 1.15 (d, *J* = 6.8 Hz, 3H).
^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm): 166.9, 157.8, 141.8, 137.0, 130.0, 128.6, 128.3, 128.1, 127.9, 127.4, 125.0, 120.7, 110.7, 96.9, 60.4, 55.2, 48.0, 46.4, 14.1
HRMS (ESI): *m/z* calculated for $C_{22}H_{23}NO_4Na^+$: 388.1525, found: 388.1528.

(2*S*,3*R*,4*S*)-6-benzyl-4-(3,4-dimethoxyphenyl)-2-hydroxy-3-methyl-3,4,5,6-tetrahydropyrano[2,3-*c*]pyrrol-7(2*H*)-one 4l



4l

Prepared according to the general procedure using *n*-propanal (17.4 mg, 0.3 mmol, 1.5 equiv) and 1-benzyl-4-(3,4-dimethoxybenzylidene)pyrrolidine-2,3-dione **2l** (67.4 mg, 0.2 mmol, 1.0 equiv). Purification of the crude product via column chromatography delivered **4l** as a white solid with 81% yield. The diastereomeric ratio was determined to be 89:11 by crude ^{1}H-NMR analysis, and the enantiomeric excess of the major product was determined to be >99% by chiral HPLC analysis on Chiralpak AD-H column (25% 2-propanol/*n*-hexane, 1 mL/min), UV 220 nm, t_{major} = 7.89 min, t_{minor} = 16.68 min; $[\alpha]_D^{20}$ = 46.3 (*c* = 1.26 in CH_2Cl_2).

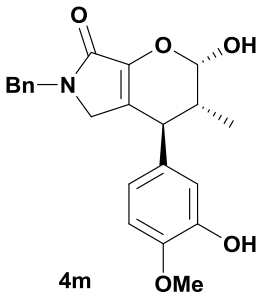
NMR and HRMS data for the product **4l**:

^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm): 7.30 – 7.22 (m, 3H), 7.19 – 7.17 (m, 2H), 6.79 (d, *J* = 8.0 Hz, 1H), 6.70 (dd, *J* = 8.4 Hz, *J* = 2.4 Hz, 1H), 6.57 (d, *J* = 2.4 Hz, 1H), 6.41 (br s, 1H), 5.73 (br s, 1H), 4.58 (q, *J* = 15.2 Hz, 2H), 3.86 (s, 3H), 3.79 (s, 3H), 3.52 – 3.38 (m, 3H), 2.12 – 2.04 (m, 1H), 0.98 (d, *J* = 6.8 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm): 166.9, 158.6, 142.3, 136.8, 132.3, 129.3, 128.6, 128.0, 127.5, 124.6, 114.1, 96.9, 55.2, 47.9, 46.5, 40.7, 39.8, 14.2

HRMS (ESI): *m/z* calculated for $C_{22}H_{23}NO_4Na^+$: 388.1525, found: 388.1518.

(2*S*,3*R*,4*S*)-6-benzyl-2-hydroxy-4-(3-hydroxy-4-methoxyphenyl)-3-methyl-3,4,5,6-tetrahydropyrano[2,3-*c*]pyrrol-7(2*H*)-one 4m



Prepared according to the general procedure using *n*-propanal (17.4 mg, 0.3 mmol, 1.5 equiv) and 1-benzyl-4-(3-hydroxy-4-methoxybenzylidene)-pyrrolidine-2,3-dione **2m** (64.6 mg, 0.2 mmol, 1.0 equiv). Purification of the crude product via column chromatography delivered **4m** as a white solid with 96% yield. The diastereomeric ratio was determined to be 88:12 by crude ^{1}H-NMR analysis, and the enantiomeric excess of the major product was determined to be >99% by chiral HPLC analysis on Chiralpak AD-H column (25% 2-propanol/*n*-hexane, 1 mL/min), UV 220 nm, t_{major} = 10.18 min, t_{minor} = 29.14 min; $[\alpha]_D^{20}$ = +154.6 (*c* = 0.58 in CH_2Cl_2).

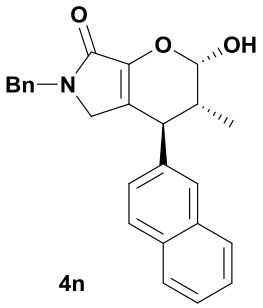
NMR and HRMS data for the product **4m**:

^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm): 7.22 – 7.15 (m, 3H), 7.11 – 7.08 (m, 2H), 6.69 (d, *J* = 8.4 Hz, 1H), 6.63 (d, *J* = 2.0 Hz, 1H), 6.54 (dd, *J* = 8.4 Hz, *J* = 2.0 Hz, 1H), 5.63 (br s, 1H), 4.65 (d, *J* = 14.8 Hz, 1H), 4.32 (d, *J* = 14.8 Hz, 1H), 3.78 (s, 3H), 3.42 – 3.30 (m, 3H), 2.02 – 1.99 (m, 1H), 0.88 (d, *J* = 6.8 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm): 166.8, 145.8, 145.6, 142.2, 136.8, 133.5, 128.7, 128.1, 128.0, 127.5, 124.5, 114.3, 110.7, 96.9, 55.9, 47.9, 46.5, 40.9, 39.6, 14.2

HRMS (ESI): *m/z* calculated for $C_{22}H_{23}NO_5Na^+$: 404.1474, found: 404.1474.

(2*S*,3*R*,4*S*)-6-benzyl-2-hydroxy-3-methyl-4-(naphthalen-2-yl)-3,4,5,6-tetrahydropyrano[2,3-*c*] pyrrol-7(2*H*)-one 4n



4n

Prepared according to the general procedure using *n*-propanal (17.4 mg, 0.3 mmol, 1.5 equiv) and 1-benzyl-4-(naphthalen-2-ylmethylene)-pyrrolidine-2,3-dione **2n** (77.0 mg, 0.2 mmol, 1.0 equiv). Purification of the crude product via column chromatography delivered **4n** as a white solid with 95% yield. The diastereomeric ratio was determined to be 92:8 by crude ^{1}H-NMR analysis, and the enantiomeric excess of the major product was determined to be 99% by chiral HPLC analysis on Chiralpak AD-H column (25% 2-propanol/*n*-hexane, 1 mL/min), UV 220 nm, t_{major} = 6.92 min, t_{minor} = 13.88 min; $[\alpha]_D^{20}$ = +65.4 (*c* = 1.72 in CH_2Cl_2).

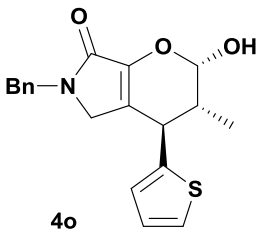
NMR and HRMS data for the product **4n**:

^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm): 8.01 – 7.94 (m, 3H), 7.81 (br s, 1H), 7.69 – 7.62 (m, 2H), 7.47 – 7.40 (m, 4H), 7.35 – 7.33 (m, 2H), 5.98 (br s, 1H), 4.89 (d, *J* = 14.8 Hz, 1H), 4.59 (d, *J* = 14.8 Hz, 1H), 3.89 (d, *J* = 10.8 Hz, 1H), 3.67 (d, *J* = 18.4 Hz, 1H), 3.55 (d, *J* = 18.4 Hz, 1H), 2.47 – 2.42 (m, 1H), 1.19 (d, *J* = 6.8 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm): 167.0, 142.5, 137.9, 136.6, 133.4, 132.6, 129.9, 128.6, 128.5, 128.1, 127.9, 127.6, 127.5, 127.4, 126.3, 125.8, 124.1, 96.9, 48.0, 46.5, 41.8, 39.6, 14.3

HRMS (ESI): *m/z* calculated for $C_{25}H_{23}NO_3Na^+$: 408.1576, found: 408.1574.

(2*S*,3*R*,4*R*)-6-benzyl-2-hydroxy-3-methyl-4-(thiophen-2-yl)-3,4,5,6-tetrahydropyrano[2,3-*c*] pyrrol-7(2*H*)-one 4o



4o

Prepared according to the general procedure using *n*-propanal (17.4 mg, 0.3 mmol, 1.5 equiv) and 1-benzyl-4-(thiophen-2-ylmethylene)-pyrrolidine-2,3-dione **2o** (56.6 mg, 0.2 mmol, 1.0 equiv). Purification of the crude product via column chromatography delivered **4o** as a white solid with 87% yield. The diastereomeric ratio was determined to be 85:15 by crude ^{1}H-NMR analysis, and the enantiomeric excess of the major product was determined to be 99% by chiral HPLC analysis on Chiralpak AD-H column (30% 2-propanol/*n*-hexane, 1 mL/min), UV 220 nm, t_{major} = 9.22 min, t_{minor} = 18.70 min; $[\alpha]_D^{20}$ = +55.8 (*c* = 0.53 in CH_2Cl_2).

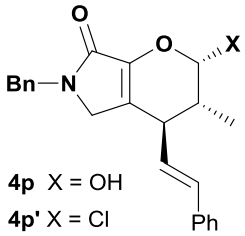
NMR and HRMS data for the product **4o**:

^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm): 7.24 – 7.17 (m, 3H), 7.13 – 7.10 (m, 3H), 6.87 – 6.84 (m, 1H), 6.80 – 6.79 (m, 1H), 6.34 (br s, 1H), 5.64 (br s, 1H), 4.63 (d, *J* = 14.8 Hz, 1H), 4.38 (d, *J* = 14.8 Hz, 1H), 3.81 (d, *J* = 10.8 Hz, 1H), 3.51 – 3.40 (m, 2H), 2.13 – 2.04 (m, 1H), 0.97 (d, *J* = 6.8 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm): 166.8, 143.6, 141.8, 136.7, 128.7, 128.0, 127.6, 126.7, 126.0, 124.4, 123.6, 96.8, 47.9, 46.5, 40.6, 36.9, 14.3

HRMS (ESI): *m/z* calculated for $C_{19}H_{19}NO_3SNa^+$: 364.0983, found: 364.0984.

(2*S*,3*R*,4*R*)-6-benzyl-2-hydroxy-3-methyl-4-((E)-styryl)-3,4,5,6-tetrahydropyrano[2,3-*c*]pyrrol-7(2*H*)-one 4p



Prepared according to the general procedure using *n*-propanal (17.4 mg, 0.3 mmol, 1.5 equiv) and 1-benzyl-4-((*E*)-3-phenylallylidene)-pyrrolidine-2,3-dione **2p** (60.6 mg, 0.2 mmol, 1.0 equiv). Purification of the crude product via column chromatography delivered **4p** as a white solid with 98% yield. The diastereomeric ratio was determined to be 85:15 by crude ^{1}H-NMR analysis, and the diastereoisomers cannot be separated by simple column chromatography; in order to get clean NMR and HPLC spectrum, **4p** was transformed to its analogue **4p'**[3], thus the enantiomeric excess of **4p'** was determined to be >99% by chiral HPLC analysis on Chiralpak AD-H column (25% 2-propanol/*n*-hexane, 1 mL/min), UV 254 nm, t_{major} = 9.08 min, t_{minor} = 10.79 min; $[\alpha]_D^{20}$ = +216.2 (*c* = 0.32 in CH_2Cl_2, *data for* **4p'**).

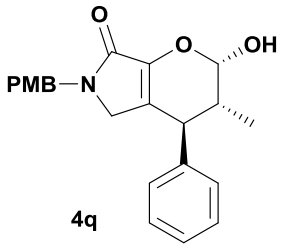
NMR and HRMS data for the product **4p'** :

^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm): 7.35 – 7.27 (m, 7H), 7.25 – 7.23 (m, 2H), 6.58 (d, *J* = 15.4 Hz, 1H), 6.19 (d, *J* = 2.4 Hz, 1H), 5.85 (dd, *J* = 15.4 Hz, *J* = 9.6 Hz, 1H), 4.61 (q, *J* = 14.8 Hz, 2H), 3.67 – 3.66 (m, 2H), 3.16 (d, *J* = 10.4 Hz, 1H), 2.26 – 2.18 (m, 1H), 1.11 (d, *J* = 6.8 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm): 164.4, 141.1, 136.8, 136.0, 135.1, 128.8, 128.7, 128.2, 128.1, 127.7, 126.6, 126.3, 125.4, 94.2, 47.7, 46.6, 39.8, 29.7, 15.0

HRMS (ESI): *m/z* calculated for $C_{23}H_{22}ClNO_2Na^+$: 402.1237, found: 402.1240.

(2*S*,3*R*,4*S*)-2-hydroxy-6-(4-methoxybenzyl)-3-methyl-4-phenyl-3,4,5,6-tetrahydropyrano[2,3-*c*] pyrrol-7(2*H*)-one 4q



4q

Prepared according to the general procedure using *n*-propanal (17.4 mg, 0.3 mmol, 1.5 equiv) and 4-benzylidene-1-(4-methoxybenzyl)-pyrrolidine-2,3-dione **2q** (61.4 mg, 0.2 mmol, 1.0 equiv). Purification of the crude product via column chromatography delivered **4q** as a white solid with 85% yield. The diastereomeric ratio was determined to be 90:10 by crude ^{1}H-NMR analysis, and the enantiomeric excess of the major product was determined to be 97% by chiral HPLC analysis on Chiralpak AD-H column (30% 2-propanol/*n*-hexane, 1 mL/min), UV 220 nm, t_{major} = 6.24 min, t_{minor} = 11.75 min; $[\alpha]_D^{20}$ = +71.0 (*c* = 2.32 in CH_2Cl_2).

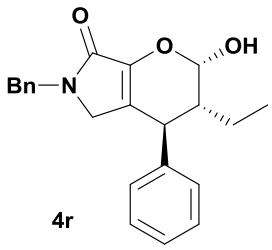
NMR and HRMS data for the product **4q**:

^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm): 7.24 – 7.20 (m, 2H), 7.18 – 7.14 (m, 1H), 7.06 – 7.00 (m, 4H), 6.74 – 6.70 (m, 2H), 5.66 (br s, 1H), 4.55 (d, *J* = 14.8 Hz, 1H), 4.28 (d, *J* = 14.8 Hz, 1H), 3.68 (s, 3H), 3.44 (d, *J* = 10.8 Hz, 1H), 3.40 – 3.25 (m, 2H), 2.07 – 1.99 (m, 1H), 0.88 (d, *J* = 6.8 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm): 166.9, 159.0, 142.4, 140.5, 129.4, 128.8, 128.6, 128.4, 127.0, 124.0, 114.0, 96.8, 55.2, 47.8, 45.9, 41.5, 39.8, 14.2

HRMS (ESI): *m/z* calculated for $C_{22}H_{23}NO_4Na^+$: 388.1525, found: 388.1528.

(2*S*,3*R*,4*S*)-6-benzyl-3-ethyl-2-hydroxy-4-phenyl-3,4,5,6-tetrahydropyrano[2,3-*c*]pyrrol-7(2*H*)-one 4r



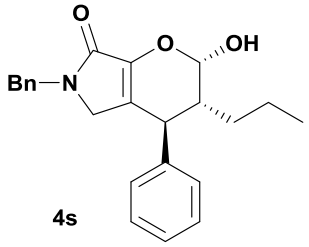
4r

Prepared according to the general procedure using *n*-butyraldehyde (21.6 mg, 0.3 mmol, 1.5 equiv) and 1-benzyl-4-benzylidenepyrrolidine-2,3-dione **2a** (55.4 mg, 0.2 mmol, 1.0 equiv). Purification of the crude product via column chromatography delivered **4r** as a white solid with 88% yield. The diastereomeric ratio was determined to be >95:5 by crude ^{1}H-NMR analysis, and the enantiomeric excess of the major product was determined to be 98% by chiral HPLC analysis on Chiralpak AD-H column (20% 2-propanol/*n*-hexane, 1 mL/min), UV 220 nm, t_{major} = 5.78 min, t_{minor} = 10.08 min; $[\alpha]_D^{20}$ = +79.8 (*c* = 0.94 in CH_2Cl_2).

NMR and HRMS data for the product **4r**:

^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm): 7.32 – 7.28 (m, 3H), 7.25 – 7.21 (m, 3H), 7.18 – 7.12 (m, 4H), 6.05 (br s, 1H), 5.88 (br s, 1H), 4.72 (d, *J* = 14.8 Hz, 1H), 4.40 (d, *J* = 14.8 Hz, 1H), 3.54 (d, *J* = 10.8 Hz, 1H), 3.49 – 3.31 (m, 2H), 1.97 – 1.90 (m, 1H), 1.57 – 1.47 (m, 2H), 0.87 (t, *J* = 7.2 Hz, 3H).
^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm): 166.8, 142.3, 140.7, 136.8, 128.7, 128.6, 128.1, 128.0, 127.6, 127.1, 124.4, 94.7, 47.9, 46.5, 46.0, 40.9, 21.6, 11.7
HRMS (ESI): *m/z* calculated for $C_{22}H_{23}NO_3Na^+$: 372.1576, found: 372.1578.

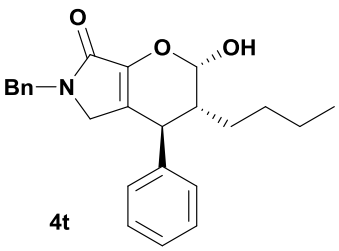
(2*S*,3*R*,4*S*)-6-benzyl-2-hydroxy-4-phenyl-3-propyl-3,4,5,6-tetrahydropyrano[2,3-*c*]pyrrol-7(2*H*)-one 4s



4s

Prepared according to the general procedure using *n*-pentanal (25.8 mg, 0.3 mmol, 1.5 equiv) and 1-benzyl-4-benzylidenepyrrolidine-2,3-dione **2a** (55.4 mg, 0.2 mmol, 1.0 equiv). Purification of the crude product via column chromatography delivered **4s** as a white solid with 92% yield. The diastereomeric ratio was determined to be 94:6 by crude ^{1}H-NMR analysis, and the enantiomeric excess of the major product was determined to be 96% by chiral HPLC analysis on Chiralpak AD-H column (25% 2-propanol/*n*-hexane, 1 mL/min), UV 220 nm, t_{major} = 5.23 min, t_{minor} = 8.47 min; $[\alpha]_D^{20}$ = +60.8 (*c* = 1.12 in CH_2Cl_2).
NMR and HRMS data for the product **4s**:
^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm): 7.33 – 7.21 (m, 6H), 7.18 – 7.12 (m, 4H), 6.60 (br s, 1H), 5.88 (br s, 1H), 4.71 (d, *J* = 14.8 Hz, 1H), 4.40 (d, *J* = 14.8 Hz, 1H), 3.55 (d, *J* = 10.8 Hz, 1H), 3.46 (d, *J* = 18.4 Hz, 1H), 3.32 (d, *J* = 18.4 Hz, 1H), 2.07 – 2.00 (m, 1H), 1.60 – 1.51 (m, 1H), 1.48 – 1.38 (m, 1H), 1.21 – 1.10 (m, 1H), 0.92 – 0.83 (m, 1H), 0.77 (t, *J* = 7.2 Hz, 3H).
^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm): 167.0, 142.2, 140.7, 136.7, 128.7, 128.6, 128.0, 127.9, 127.5, 127.1, 124.5, 94.9, 48.0, 46.5, 44.1, 40.9, 30.8, 20.1, 14.1
HRMS (ESI): *m/z* calculated for $C_{23}H_{25}NO_3Na^+$: 386.1732, found: 386.1736.

(2*S*,3*R*,4*S*)-6-benzyl-3-butyl-2-hydroxy-4-phenyl-3,4,5,6-tetrahydropyrano[2,3-*c*]pyrrol-7(2*H*)-one 4t



4t

Prepared according to the general procedure using *n*-hexanal (30.0 mg, 0.3 mmol, 1.5 equiv) and 1-benzyl-4-benzylidenepyrrolidine-2,3-dione **2a** (55.4 mg, 0.2 mmol, 1.0 equiv). Purification of the crude product via column chromatography delivered **4t** as a white solid with 82% yield. The diastereomeric ratio was determined to be 93:7 by crude ^{1}H-NMR analysis, and the enantiomeric excess of the major product was determined to be 97% by chiral HPLC analysis on Chiralpak AD-H column (25% 2-propanol/*n*-hexane, 1 mL/min), UV 220 nm, t_{major} = 5.08 min, t_{minor} = 7.40 min; $[\alpha]_D^{20}$ = +58.2 (*c* = 1.21 in CH_2Cl_2).

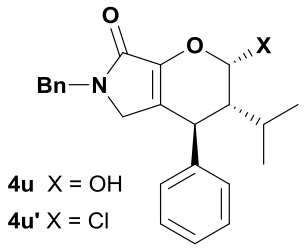
NMR and HRMS data for the product **4t**:

^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm): 7.32 – 7.27 (m, 4H), 7.24 – 7.21 (m, 2H), 7.18 – 7.16 (m, 2H), 7.14 – 7.12 (m, 2H), 5.83 (br s, 1H), 4.72 (d, *J* = 14.8 Hz, 1H), 4.40 (d, *J* = 14.8 Hz, 1H), 3.53 (d, *J* = 10.8 Hz, 1H), 3.46 (d, *J* = 18.4 Hz, 1H), 3.32 (d, *J* = 18.4 Hz, 1H), 2.04 – 1.97 (m, 1H), 1.58 – 1.48 (m, 1H), 1.40 – 1.32 (m, 1H), 1.26 – 1.05 (m, 4H), 0.77 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm): 166.8, 142.2, 140.6, 136.8, 130.1, 128.7, 128.6, 128.0, 127.6, 127.1, 124.5, 94.9, 47.9, 46.5, 44.2, 40.9, 29.1, 28.3, 22.7, 13.8

HRMS (ESI): *m/z* calculated for $C_{24}H_{27}NO_3Na^+$: 400.1889, found: 400.1892.

(2*S*,3*R*,4*S*)-6-benzyl-2-hydroxy-3-isopropyl-4-phenyl-3,4,5,6-tetrahydropyrano[2,3-*c*]pyrrol-7(2*H*)-one 4u



Prepared according to the general procedure using 3-methylbutanal (25.8 mg, 0.3 mmol, 1.5 equiv) and 1-benzyl-4-benzylidenepyrrolidine-2,3-dione **2a** (55.4 mg, 0.2 mmol, 1.0 equiv). Purification of the crude product via column chromatography delivered **4u** as a white solid with 70% yield. The diastereomeric ratio was determined to be 85:15 by crude ^{1}H-NMR analysis, and the diastereoisomers cannot be separated by simple column chromatography; in order to get clean NMR and HPLC spectrum, **4u** was transformed to its analogue **4u'**[3], thus the enantiomeric excess of **4u'** was determined to be 97% by chiral HPLC analysis on Chiralpak OD-H column (30% 2-propanol/*n*-hexane, 1 mL/min), UV 220 nm, t_{minor} = 8.74 min, t_{major} = 13.03 min; $[\alpha]_D^{20}$ = +175.0 (*c* = 0.26 in CH_2Cl_2, *data for* **4u'**).

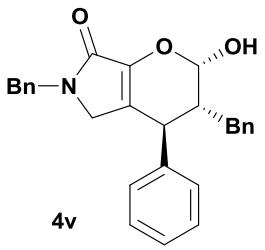
NMR and HRMS data for the product **4u'** :

^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm): 7.33 – 7.24 (m, 6H), 7.17 – 7.14 (m, 4H), 6.36 (d, *J* = 2.4 Hz, 1H), 4.75 (d, *J* = 14.8 Hz, 1H), 4.35 (d, *J* = 14.8 Hz, 1H), 3.81 (d, *J* = 11.2 Hz, 1H), 3.50 (d, *J* = 18.4 Hz, 1H), 3.19 (d, *J* = 18.4 Hz, 1H), 2.42 – 2.37 (m, 1H), 1.75 – 1.67 (m, 1H), 0.95 (d, *J* = 7.2 Hz, 3H), 0.79 (d, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm): 164.4, 140.9, 139.7, 136.8, 129.1, 128.7, 128.1, 127.9, 127.6, 127.5, 127.4, 91.7, 50.9, 47.4, 46.4, 39.9, 28.1, 21.1, 19.0

HRMS (ESI): *m/z* calculated for $C_{23}H_{24}ClNO_2Na^+$: 404.1393, found: 404.1397.

(2*S*,3*R*,4*S*)-3,6-dibenzyl-2-hydroxy-4-phenyl-3,4,5,6-tetrahydropyrano[2,3-*c*]pyrrol-7(2*H*)-one 4v



4v

Prepared according to the general procedure using 3-phenylpropanal (40.2 mg, 0.3 mmol, 1.5 equiv) and 1-benzyl-4-benzylidenepyrrolidine-2,3-dione **2a** (55.4 mg, 0.2 mmol, 1.0 equiv). Purification of the crude product via column chromatography delivered **4v** as a white solid with 85% yield. The diastereomeric ratio was determined to be 93:7 by crude ^{1}H-NMR analysis, and the enantiomeric excess of the major product was determined to be 99% by chiral HPLC analysis on Chiralpak AD-H column (25% 2-propanol/*n*-hexane, 1 mL/min), UV 220 nm, t_{major} = 6.25 min, t_{minor} = 7.78 min; $[\alpha]_D^{20}$ = +126.8 (*c* = 0.48 in CH_2Cl_2).

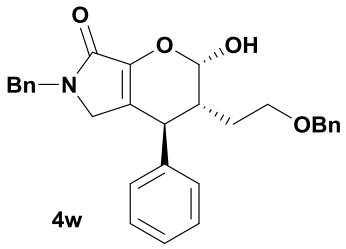
NMR and HRMS data for the product **4v**:

^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm): 7.35 – 7.19 (m, 8H), 7.18 – 7.16 (m, 4H), 7.14 – 7.09 (m, 1H), 7.06 – 7.04 (m, 2H), 5.45 (br s, 1H), 4.72 (d, *J* = 14.8 Hz, 1H), 4.40 (d, *J* = 14.8 Hz, 1H), 3.65 (d, *J* = 10.8 Hz, 1H), 3.48 (d, *J* = 18.4 Hz, 1H), 3.33 (d, *J* = 18.4 Hz, 1H), 2.77 – 2.71 (m, 1H), 2.56 (dd, *J* = 13.6 Hz, *J* = 3.6 Hz, 1H), 2.29 – 2.22 (m, 1H).

^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm): 167.0, 142.2, 140.7, 136.7, 128.7, 128.6, 128.0, 127.9, 127.5, 127.1, 124.5, 94.9, 48.0, 46.5, 44.1, 40.9, 30.8, 20.1, 14.1

HRMS (ESI): *m/z* calculated for $C_{23}H_{25}NO_3Na^+$: 386.1732, found: 386.1736.

(2*S*,3*R*,4*S*)-6-benzyl-3-(2-(benzyloxy)ethyl)-2-hydroxy-4-phenyl-3,4,5,6-tetrahydropyrano[2,3-*c*] pyrrol-7(2*H*)-one 4w



4w

Prepared according to the general procedure using 4-(benzyloxy)butanal (40.2 mg, 0.3 mmol, 1.5 equiv) and 1-benzyl-4-benzylidenepyrrolidine-2,3-dione **2a** (55.4 mg, 0.2 mmol, 1.0 equiv). Purification of the crude product via column chromatography delivered **4w** as a white solid with 80% yield. The diastereomeric ratio was determined to be 94:6 by crude ^{1}H-NMR analysis, and the enantiomeric excess of the major product was determined to be 99% by chiral HPLC analysis on Chiralpak AD-H column (25% 2-propanol/*n*-hexane, 1 mL/min), UV 220 nm, t_{major} = 7.11 min, t_{minor} = 10.56 min; $[\alpha]_D^{20}$ = +69.1 (*c* = 1.09 in CH_2Cl_2).

NMR and HRMS data for the product **4w**:

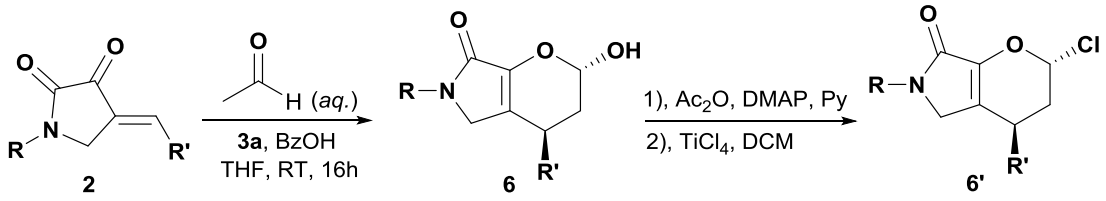
^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm): 7.33 – 7.24 (m, 8H), 7.23 – 7.21 (m, 3H), 7.18 – 7.12 (m, 4H), 6.37 (br s, 1H), 5.86 (br s, 1H), 4.70 (d, *J* = 14.8 Hz, 1H), 4.41 (d, *J* = 14.8 Hz, 1H),

4.33 (s, 2H), 3.59 (d, *J* = 10.8 Hz, 1H), 3.50 – 3.42 (m, 2H), 3.37 – 3.31 (m, 2H), 2.29 – 2.23 (m, 1H), 1.91 – 1.81 (m, 1H), 1.66 – 1.58 (m, 1H).

^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm): 166.8, 142.2, 140.4, 138.2, 136.7, 128.8, 128.6, 128.4, 128.3, 128.0, 127.6, 127.5, 127.4, 127.2, 124.1, 94.9, 72.6, 68.0, 47.9, 46.5, 41.6, 40.9, 28.7

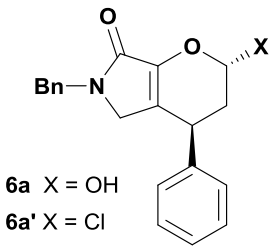
HRMS (ESI): *m/z* calculated for $C_{29}H_{29}NO_4Na^+$: 478.1994, found: 478.2000.

4. General Procedure for the Synthesis of Chiral Bicyclic Dihydropyranes 6 by Directly Using Aqueous Acetaldehyde



A glass tube was charged with pyrrolidine-2,3-dione **2** (0.2 mmol), amine catalyst **3a** (0.04 mmol, 13 mg) and benzoic acid (0.04 mmol, 4.9 mg) in 2 mL THF. The 40% aqueous acetaldehyde **5** (0.6 mmol, 66mg) was added with a syringe, and the reaction was sealed with a Teflon cap and stirred at room temperature for about 16 hours. When the reaction was complete, the mixture was directly purified by column chromatography on silica gel eluting with petroleum ether/ethyl acetate (4:1 to 1:1) to afford the corresponding bicyclic dihydropyranes **6**, which was analyzed by crude ^{1}H-NMR to determine the diastereoselectivity of the *oxo*-IEDDA reaction. Since the diastereoisomers of **6** cannot be separated by simple column chromatography in most cases, we transform **6** to its analogue **6'** by using the following sequential reactions: a glass tube was charged with **6**, Ac_2O (5 eq.) and DMAP (0.1 eq.) in 1 mL pyridine, and the reaction was stirred at room temperature for about 2 hours. When the reaction was completed, 2 mL of 20% hydrochloric acid was added to the reaction mixture, and the organic material was extracted with ethyl acetate, washed with brine and dried over anhydrous Na_2SO_4, then concentrated under reduced pressure to give the corresponding acetylated compound; such compound was dissolved in 2 mL DCM under argon, and $TiCl_4$ (5 eq.) was added into the reaction mixture. The reaction was stirred at room temperature for about 16 hours. When the reaction was completed, 2 mL DCM and 1mL water was added to the reaction mixture. The organic layer was separated, dried over anhydrous Na_2SO_4 and purified by column chromatography on silica gel eluting with petroleum ether/ethyl acetate (6:1 to 4:1) to afford the desired pure product **6'** (>95:5 d.r.) which was further analyzed by ^{1}H-NMR, ^{13}C-HMR, HRMS, chiral HPLC analysis, *etc*.

(2*S*,4*S*)-6-benzyl-2-hydroxy-4-phenyl-3,4,5,6-tetrahydropyrano[2,3-*c*]pyrrol-7(2*H*)-one 6a



According to the general procedure, the crude product was purified via column chromatography delivering **6a** as a white solid with 95% yield. The diastereomeric ratio was determined to be 88:12 by crude ^{1}H-NMR analysis. **6a** was transformed to its analogue **6a'** (only a single diastereoisomer was obtained) with 86% yield. The enantiomeric excess of **6a'** was determined to be 99% by chiral HPLC analysis on Chiralpak OD-H column (30% 2-propanol/*n*-hexane, 1 mL/min), UV 220 nm, t_{major} = 14.96 min, t_{minor} = 20.92 min; $[\alpha]_D^{20}$ = +124.8 (*c* = 0.32 in CH_2Cl_2, *data for* **6a'**).

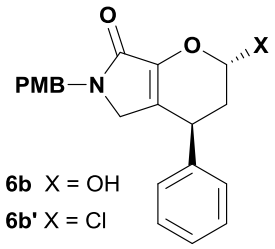
NMR and HRMS data for the product **6a'** :

^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm): 7.35 – 7.27 (m, 6H), 7.21 – 7.15 (m, 4H), 6.44 (t, *J* = 2.4 Hz, 1H), 4.75 (d, *J* = 14.8 Hz, 1H), 4.44 (d, *J* = 14.8 Hz, 1H), 4.07 (dd, *J* = 11.6 Hz, *J* = 6.0 Hz, 1H), 3.58 (dd, *J* = 18.4 Hz, *J* = 1.6 Hz, 1H), 3.46 (dd, *J* = 18.4 Hz, *J* = 1.6 Hz, 1H), 2.53 – 2.47 (m, 1H), 2.40 – 2.33 (m, 1H).

^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm): 164.3, 141.8, 139.6, 136.7, 129.1, 128.8, 128.0, 127.9, 127.7, 127.6, 125.3, 88.4, 47.4, 46.5, 39.6, 34.7

HRMS (ESI): *m/z* calculated for $C_{20}H_{18}ClNO_2Na^+$: 362.0924, found: 362.0927.

(2*S*,4*S*)-2-hydroxy-6-(4-methoxybenzyl)-4-phenyl-3,4,5,6-tetrahydropyrano[2,3-*c*]pyrrol-7(2*H*)-one 6b

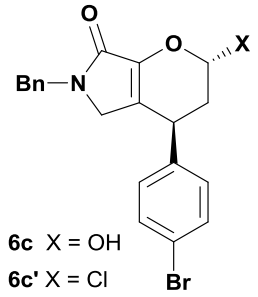


According to the general procedure, the crude product was purified via column chromatography delivering **6b** as a white solid with 90% yield. The diastereomeric ratio was determined to be 89:11 by crude ^{1}H-NMR analysis. **6b** was transformed to its analogue **6b'** (only a single diastereoisomer was obtained) with 81% yield. The enantiomeric excess of **6b'** was determined to be 95% by chiral HPLC analysis on Chiralpak AD-H column (20% 2-propanol/*n*-hexane, 1 mL/min), UV 220 nm, t_{major} = 22.87 min, t_{minor} = 26.50 min; $[\alpha]_D^{20}$ = +56.6 (*c* = 0.54 in CH_2Cl_2, *data for* **6b'**).

NMR and HRMS data for the product **6b'** :

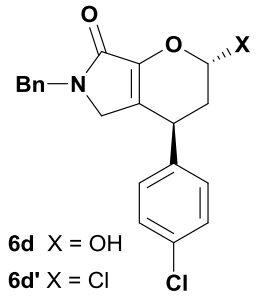
^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm): 7.29 – 7.21 (m, 3H), 7.10 – 7.05 (m, 4H), 6.76 (d, *J* = 8.4 Hz, 2H), 6.36 (t, *J* = 2.4 Hz, 1H), 4.63 (d, *J* = 14.8 Hz, 1H), 4.29 (d, *J* = 14.8 Hz, 1H), 3.98 (dd, *J* = 11.6 Hz, *J* = 6.0 Hz, 1H), 3.71 (s, 3H), 3.48 (dd, *J* = 18.4 Hz, *J* = 1.6 Hz, 1H), 3.36 (dd, *J* = 18.4 Hz, *J* = 1.6 Hz, 1H), 2.45 – 2.39 (m, 1H), 2.32 – 2.25 (m, 1H).
^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm): 164.2, 159.1, 141.9, 139.7, 129.5, 129.1, 128.8, 127.9, 127.7, 125.2, 114.1, 88.4, 55.3, 47.3, 45.9, 39.6, 34.8
HRMS (ESI): *m/z* calculated for $C_{21}H_{20}ClNO_3Na^+$: 392.1029, found: 392.1030.

(2*S*,4*S*)-6-benzyl-4-(4-bromophenyl)-2-hydroxy-3,4,5,6-tetrahydropyrano[2,3-*c*]pyrrol-7(2*H*)-one 6c



According to the general procedure, the crude product was purified via column chromatography delivering **6c** as a white solid with 88% yield. The diastereomeric ratio was determined to be 85:15 by crude ^{1}H-NMR analysis. **6c** was transformed to its analogue **6c'** (only a single diastereoisomer was obtained) with 80% yield. The enantiomeric excess of **6c'** was determined to be 97% by chiral HPLC analysis on Chiralpak OD-H column (30% 2-propanol/*n*-hexane, 1 mL/min), UV 220 nm, t_{major} = 16.79 min, t_{minor} = 22.55 min; $[\alpha]_D^{20}$ = +121.6 (*c* = 0.32 in CH_2Cl_2, *data for* **6c'**).
NMR and HRMS data for the product **6c'** :
^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm): 7.46 (d, *J* = 8.4 Hz, 2H), 7.34 – 7.27 (m, 3H), 7.21 – 7.19 (m, 2H), 7.05 (d, *J* = 8.4 Hz, 2H), 6.43 (t, *J* = 2.4 Hz, 1H), 4.74 (d, *J* = 14.8 Hz, 1H), 4.45 (d, *J* = 14.8 Hz, 1H), 4.04 (dd, *J* = 11.6 Hz, *J* = 6.0 Hz, 1H), 3.56 (dd, *J* = 18.4 Hz, *J* = 1.6 Hz, 1H), 3.43 (dd, *J* = 18.4 Hz, *J* = 1.6 Hz, 1H), 2.51 – 2.46 (m, 1H), 2.35 – 2.28 (m, 1H).
^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm): 164.1, 142.1, 138.6, 136.6, 132.3, 129.6, 128.8, 128.1, 127.8, 124.4, 121.7, 88.2, 47.3, 46.5, 39.5, 34.3
HRMS (ESI): *m/z* calculated for $C_{20}H_{17}BrClNO_2Na^+$: 440.0029, found: 440.0030.

(2*S*,4*S*)-6-benzyl-4-(4-chlorophenyl)-2-hydroxy-3,4,5,6-tetrahydropyrano[2,3-*c*]pyrrol-7(2*H*)-one 6d



According to the general procedure, the crude product was purified via column chromatography delivering **6d** as a white solid with 92% yield. The diastereomeric ratio was determined to be 85:15 by crude ^{1}H-NMR analysis. **6d** was transformed to its analogue **6d'** (only a single diastereoisomer was obtained) with 84% yield. The enantiomeric excess of **6c'** was determined to be 95% by chiral HPLC analysis on Chiralpak AD-H column (30% 2-propanol/*n*-hexane, 1 mL/min), UV 220 nm, t_{major} = 13.38 min, t_{minor} = 14.79 min; $[\alpha]_D^{20}$ = +56.8 (*c* = 0.94 in CH_2Cl_2, *data for* **6d'**).

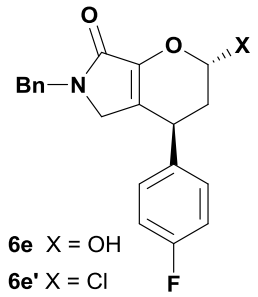
NMR and HRMS data for the product **6d'** :

^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm): 7.34 – 7.26 (m, 5H), 7.21 – 7.19 (m, 2H), 7.12 – 7.09 (m, 2H), 6.43 (t, *J* = 2.4 Hz, 1H), 4.74 (d, *J* = 14.8 Hz, 1H), 4.45 (d, *J* = 14.8 Hz, 1H), 4.06 (dd, *J* = 11.6 Hz, *J* = 6.0 Hz, 1H), 3.57 (dd, *J* = 18.4 Hz, *J* = 1.6 Hz, 1H), 3.43 (dd, *J* = 18.4 Hz, *J* = 1.6 Hz, 1H), 2.51 – 2.46 (m, 1H), 2.35 – 2.28 (m, 1H).

^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm): 164.1, 142.0, 138.1, 136.6, 133.6, 129.3, 129.2, 128.8, 128.1, 127.7, 124.6, 88.2, 47.3, 46.5, 39.5, 34.2

HRMS (ESI): *m/z* calculated for $C_{20}H_{17}Cl_2NO_2Na^+$: 396.0534, found: 396.0535.

(2*S*,4*S*)-6-benzyl-4-(4-fluorophenyl)-2-hydroxy-3,4,5,6-tetrahydropyrano[2,3-*c*]pyrrol-7(2*H*)-one 6e



According to the general procedure, the crude product was purified via column chromatography delivering **6e** as a white solid with 98% yield. The diastereomeric ratio was determined to be 87:13 by crude ^{1}H-NMR analysis. **6e** was transformed to its analogue **6e'** (only a single diastereoisomer was obtained) with 85% yield. The enantiomeric excess of **6e'** was determined to be 96% by chiral HPLC analysis on Chiralpak AD-H column (30% 2-propanol/*n*-hexane, 1 mL/min), UV 220 nm, t_{major} = 12.89 min, t_{minor} = 14.60 min; $[\alpha]_D^{20}$ = +58.3 (*c* = 1.21 in CH_2Cl_2, *data for* **6e'**).

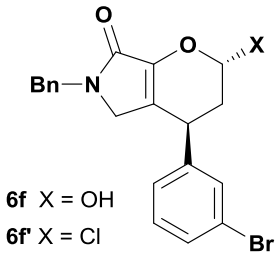
NMR and HRMS data for the product **6e'** :

^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm): 7.33 – 7.25 (m, 3H), 7.21 – 7.19 (m, 2H), 7.15 – 7.12 (m, 2H), 7.05 – 6.99 (m, 2H), 6.43 (t, *J* = 2.4 Hz, 1H), 4.74 (d, *J* = 14.8 Hz, 1H), 4.45 (d, *J* = 14.8 Hz, 1H), 4.07 (dd, *J* = 11.6 Hz, *J* = 6.0 Hz, 1H), 3.57 (dd, *J* = 18.4 Hz, *J* = 1.6 Hz, 1H), 3.43 (dd, *J* = 18.4 Hz, *J* = 1.6 Hz, 1H), 2.52 – 2.46 (m, 1H), 2.36 – 2.29 (m, 1H).

^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm): 164.2, 163.3, 160.9, 141.9, 136.6, 135.3, 135.2, 129.4, 129.3, 128.8, 128.0, 127.7, 124.9, 116.2, 116.0, 88.3, 47.3, 46.5, 39.7, 34.1

HRMS (ESI): *m/z* calculated for $C_{20}H_{17}ClFNO_2Na^+$: 380.0830, found: 380.0828.

(2*S*,4*S*)-6-benzyl-4-(3-bromophenyl)-2-hydroxy-3,4,5,6-tetrahydropyrano[2,3-*c*]pyrrol-7(2*H*)-one 6f



According to the general procedure, the crude product was purified via column chromatography delivering **6f** as a white solid with 97% yield. The diastereomeric ratio was determined to be 86:14 by crude ^{1}H-NMR analysis. **6f** was transformed to its analogue **6f'** (only a single diastereoisomer was obtained) with 82% yield. The enantiomeric excess of **6f'** was determined to be 96% by chiral HPLC analysis on Chiralpak AD-H column (30% 2-propanol/*n*-hexane, 1 mL/min), UV 220 nm, t_{minor} = 13.32 min, t_{major} = 15.70 min; $[\alpha]_D^{20}$ = +60.8 (*c* = 0.97 in CH_2Cl_2, *data for* **6f'**).

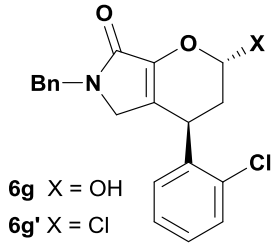
NMR and HRMS data for the product **6f'** :

^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm): 7.43 – 7.41 (m, 1H), 7.34 – 7.26 (m, 4H), 7.23 – 7.19 (m, 3H), 7.11 – 7.08 (m, 1H), 6.43 (t, *J* = 2.4 Hz, 1H), 4.77 (d, *J* = 14.8 Hz, 1H), 4.45 (d, *J* = 14.8 Hz, 1H), 4.04 (dd, *J* = 11.6 Hz, *J* = 6.0 Hz, 1H), 3.58 (dd, *J* = 18.4 Hz, *J* = 1.6 Hz, 1H), 3.45 (dd, *J* = 18.4 Hz, *J* = 1.6 Hz, 1H), 2.53 – 2.47 (m, 1H), 2.37 – 2.29 (m, 1H).

^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm): 164.1, 142.1, 142.0, 136.6, 131.0, 130.9, 130.7, 128.8, 128.0, 127.7, 126.5, 124.3, 123.1, 88.1, 47.3, 46.5, 39.4, 34.5

HRMS (ESI): *m/z* calculated for $C_{20}H_{17}BrClNO_2Na^+$: 440.0029, found: 440.0027.

(2*S*,4*R*)-6-benzyl-4-(2-chlorophenyl)-2-hydroxy-3,4,5,6-tetrahydropyrano[2,3-*c*]pyrrol-7(2*H*)-one 6g

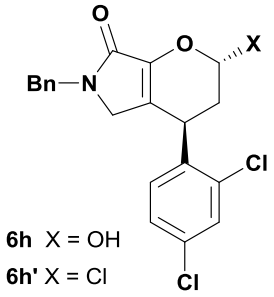


According to the general procedure, the crude product was purified via column chromatography delivering **6g** as a white solid with 95% yield. The diastereomeric ratio was determined to be 84:16 by crude ^{1}H-NMR analysis. **6g** was transformed to its analogue **6g'** (only a single diastereoisomer was obtained) with 80% yield. The enantiomeric excess of **6g'** was determined to be 95% by chiral HPLC analysis on Chiralpak OD-H column (30% 2-propanol/*n*-hexane, 1 mL/min), UV 220 nm, t_{major} = 12.99 min, t_{minor} = 18.38 min; $[\alpha]_D^{20}$ = +66.7 (*c* = 1.06 in CH_2Cl_2, *data for* **6g'**).

NMR and HRMS data for the product **6g'** :

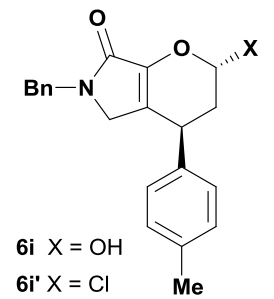
^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm): 7.39 – 7.37 (m, 1H), 7.34 – 7.26 (m, 3H), 7.24 – 7.19 (m, 4H), 7.14 – 7.11 (m, 1H), 6.43 (t, *J* = 2.4 Hz, 1H), 4.72 (d, *J* = 14.8 Hz, 1H), 4.63 (br s, 1H), 4.52 (d, *J* = 14.8 Hz, 1H), 3.64 (dd, *J* = 18.4 Hz, *J* = 1.6 Hz, 1H), 3.53 (dd, *J* = 18.4 Hz, *J* = 1.6 Hz, 1H), 2.55 – 2.50 (m, 1H), 2.36 (br s, 1H).
^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm): 164.3, 142.5, 137.1, 136.7, 134.1, 130.3, 129.0, 128.8, 128.0, 127.7, 127.6, 124.4, 88.3, 47.5, 46.5, 37.8, 29.7
HRMS (ESI): *m/z* calculated for $C_{20}H_{17}Cl_2NO_2Na^+$: 396.0534, found: 396.0531.

(2*S*,4*R*)-6-benzyl-4-(2,4-dichlorophenyl)-2-hydroxy-3,4,5,6-tetrahydropyrano[2,3-*c*]pyrrol-7(2*H*)-one 6h



According to the general procedure, the crude product was purified via column chromatography delivering **6h** as a white solid with 86% yield. The diastereomeric ratio was determined to be 82:18 by crude ^{1}H-NMR analysis. **6h** was transformed to its analogue **6h'** (only a single diastereoisomer was obtained) with 78% yield. The enantiomeric excess of **6h'** was determined to be 96% by chiral HPLC analysis on Chiralpak AD-H column (30% 2-propanol/*n*-hexane, 1 mL/min), UV 220 nm, t_{major} = 11.91 min, t_{minor} = 17.52 min; $[\alpha]_D^{20}$ = +48.7 (*c* = 1.22 in CH_2Cl_2, *data for* **6h'**).
NMR and HRMS data for the product **6h'** :
^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm): 7.40 (d, *J* = 2.4 Hz, 1H), 7.34 – 7.25 (m, 3H), 7.24 – 7.20 (m, 3H), 7.06 (d, *J* = 8.4 Hz, 1H), 6.43 (t, *J* = 2.4 Hz, 1H), 4.70 (d, *J* = 14.8 Hz, 1H), 4.59 (br s, 1H), 4.53 (d, *J* = 14.8 Hz, 1H), 3.63 (dd, *J* = 18.4 Hz, *J* = 1.6 Hz, 1H), 3.50 (dd, *J* = 18.4 Hz, *J* = 1.6 Hz, 1H), 2.53 – 2.48 (m, 1H), 2.32 (br s, 1H).
^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm): 164.0, 142.7, 136.5, 135.6, 134.7, 134.1, 130.1, 128.8, 128.2, 128.0, 127.9, 127.8, 123.6, 88.0, 47.3, 46.5, 37.8, 29.7
HRMS (ESI): *m/z* calculated for $C_{20}H_{16}Cl_3NO_2Na^+$: 430.0144, found: 430.0142.

(2*S*,4*S*)-6-benzyl-2-hydroxy-4-(*p*-tolyl)-3,4,5,6-tetrahydropyrano[2,3-*c*]pyrrol-7(2*H*)-one 6i



According to the general procedure, the crude product was purified via column chromatography delivering **6i** as a white solid with 91% yield. The diastereomeric ratio was determined to be 86:14 by crude ^{1}H-NMR analysis. **6i** was transformed to its analogue **6i'** (only a single diastereoisomer was obtained) with 83% yield. The enantiomeric excess of **6i'** was determined to be 95% by chiral HPLC analysis on Chiralpak AD-H column (25% 2-propanol/*n*-hexane, 1 mL/min), UV 220 nm, t_{major} = 11.76 min, t_{minor} = 12.88 min; $[\alpha]_D^{20}$ = +35.4 (*c* = 1.14 in CH_2Cl_2, *data for* **6i'**).

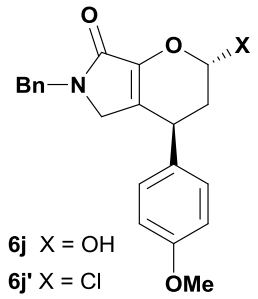
NMR and HRMS data for the product **6i'** :

^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm): 7.33 – 7.26 (m, 3H), 7.21 – 7.18 (m, 2H), 7.13 (d, *J* = 7.8 Hz, 2H), 7.04 (d, *J* = 7.8 Hz, 2H), 6.43 (t, *J* = 2.4 Hz, 1H), 4.74 (d, *J* = 14.8 Hz, 1H), 4.44 (d, *J* = 14.8 Hz, 1H), 4.03 (dd, *J* = 11.6 Hz, *J* = 6.0 Hz, 1H), 3.56 (dd, *J* = 18.4 Hz, *J* = 1.6 Hz, 1H), 3.46 (dd, *J* = 18.4 Hz, *J* = 1.6 Hz, 1H), 2.50 – 2.44 (m, 1H), 2.38 – 2.31 (m, 4H).

^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm): 164.3, 141.7, 137.5, 136.7, 136.5, 129.8, 128.7, 128.0, 127.7, 127.6, 125.6, 88.5, 47.5, 46.5, 39.7, 34.3, 21.0

HRMS (ESI): *m/z* calculated for $C_{21}H_{20}ClNO_2Na^+$: 376.1080, found: 376.1083.

(2*S*,4*S*)-6-benzyl-2-hydroxy-4-(4-methoxyphenyl)-3,4,5,6-tetrahydropyrano[2,3-*c*]pyrrol-7(2*H*)-one 6j



According to the general procedure, the crude product was purified via column chromatography delivering **6j** as a white solid with 95% yield. The diastereomeric ratio was determined to be 85:15 by crude ^{1}H-NMR analysis. **6j** was transformed to its analogue **6j'** (only a single diastereoisomer was obtained) with 84% yield. The enantiomeric excess of **6j'** was determined to be 95% by chiral HPLC analysis on Chiralpak AD-H column (30% 2-propanol/*n*-hexane, 1 mL/min), UV 220 nm, t_{minor} = 15.36 min, t_{major} = 16.89 min; $[\alpha]_D^{20}$ = +115.0 (*c* = 0.23 in CH_2Cl_2, *data for* **6j'**).

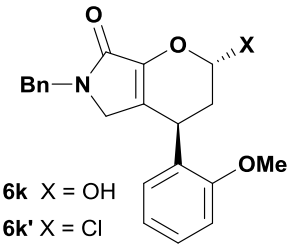
NMR and HRMS data for the product **6j'** :

^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm): 7.26 – 7.19 (m, 3H), 7.14 – 7.11 (m, 2H), 7.02 – 6.99 (m, 2H), 6.80 – 6.76 (m, 2H), 6.36 (t, *J* = 2.4 Hz, 1H), 4.67 (d, *J* = 14.8 Hz, 1H), 4.37 (d, *J* = 14.8 Hz, 1H), 3.95 (dd, *J* = 11.6 Hz, *J* = 6.0 Hz, 1H), 3.72 (s, 3H), 3.49 (dd, *J* = 18.4 Hz, *J* = 1.6 Hz, 1H), 3.37 (dd, *J* = 18.4 Hz, *J* = 1.6 Hz, 1H), 2.43 – 2.37 (m, 1H), 2.29 – 2.22 (m, 1H).

^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm): 164.4, 159.1, 141.7, 136.8, 131.5, 128.9, 128.7, 128.1, 127.7, 125.8, 114.5, 88.6, 55.3, 47.5, 46.5, 39.7, 33.9

HRMS (ESI): *m/z* calculated for $C_{21}H_{20}ClNO_3Na^+$: 392.1029, found: 392.1030.

(2*S*,4*S*)-6-benzyl-2-hydroxy-4-(2-methoxyphenyl)-3,4,5,6-tetrahydropyrano[2,3-*c*]pyrrol-7(2*H*)-one 6k



According to the general procedure, the crude product was purified via column chromatography delivering **6k** as a white solid with 90% yield. The diastereomeric ratio was determined to be 86:14 by crude ^{1}H-NMR analysis. **6k** was transformed to its analogue **6k'** (only a single diastereoisomer was obtained) with 81% yield. The enantiomeric excess of **6k'** was determined to be 96% by chiral HPLC analysis on Chiralpak AD-H column (30% 2-propanol/*n*-hexane, 1 mL/min), UV 220 nm, t_{major} = 16.28 min, t_{minor} = 21.60 min; $[\alpha]_D^{20}$ = +78.5 (*c* = 0.46 in CH_2Cl_2, *data for* **6k'**).

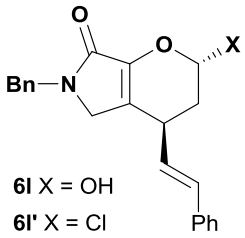
NMR and HRMS data for the product **6k'** :

^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm): 7.32 – 7.25 (m, 3H), 7.24 – 7.18 (m, 3H), 7.08 (dd, *J* = 7.2 Hz, *J* = 1.6 Hz, 1H), 6.91 (t, *J* = 7.2 Hz, 1H), 6.85 (d, *J* = 8.4 Hz, 1H), 6.44 (t, *J* = 2.4 Hz, 1H), 4.60 (dd, *J* = 17.2 Hz, *J* = 15.2 Hz, 1H), 4.42 (dd, *J* = 11.6 Hz, *J* = 6.0 Hz, 1H), 3.72 (s, 3H), 3.59 (dd, *J* = 18.4 Hz, *J* = 1.6 Hz, 1H), 3.43 (dd, *J* = 18.4 Hz, *J* = 1.6 Hz, 1H), 2.53 – 2.46 (m, 1H), 2.41 – 2.35 (m, 1H).

^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm): 164.6, 157.3, 141.1, 137.0, 129.2, 128.8, 128.7, 127.9, 127.6, 127.3, 126.3, 121.0, 110.9, 89.0, 55.2, 47.6, 46.4, 37.1, 29.7

HRMS (ESI): *m/z* calculated for $C_{21}H_{20}ClNO_3Na^+$: 392.1029, found: 392.1030.

(2*S*,4*S*)-6-benzyl-2-hydroxy-4-((*E*)-styryl)-3,4,5,6-tetrahydropyrano[2,3-*c*]pyrrol-7(2*H*)-one 6l

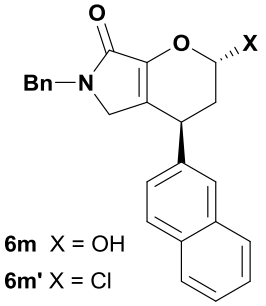


According to the general procedure, the crude product was purified via column chromatography delivering **6l** as a white solid with 89% yield. The diastereomeric ratio was determined to be 82:18 by crude ^{1}H-NMR analysis. **6l** was transformed to its analogue **6l'** (only a single diastereoisomer was obtained) with 70% yield. The enantiomeric excess of **6l'** was determined to be 90% by chiral HPLC analysis on Chiralpak AD-H column (20% 2-propanol/*n*-hexane, 1 mL/min), UV 220 nm, t_{major} = 9.83 min, t_{minor} = 18.29 min; $[\alpha]_D^{20}$ = +138.5 (*c* = 0.38 in CH_2Cl_2, *data for* **6l'**).

NMR and HRMS data for the product **6l'** :

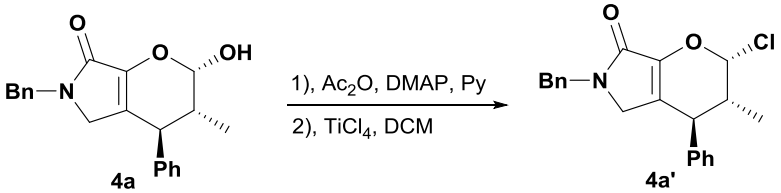
^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm): 7.34 – 7.28 (m, 7H), 7.25 – 7.23 (m, 3H), 6.60 (d, *J* = 15.6 Hz, 1H), 6.42 (t, *J* = 2.4 Hz, 1H), 5.96 (dd, *J* = 15.6 Hz, *J* = 8.8 Hz, 1H), 4.62 (q, *J* = 14.8 Hz, 2H), 3.71 – 3.70 (m, 3H), 2.43 – 2.37 (m, 1H), 2.23 – 2.16 (m, 1H).
^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm): 164.3, 141.0, 136.7, 136.0, 133.8, 128.8, 128.7, 128.2, 128.1, 127.7, 127.0, 126.3, 124.8, 88.3, 47.7, 46.6, 37.1, 32.4
HRMS (ESI): *m/z* calculated for $C_{22}H_{20}ClNO_2Na^+$: 388.1080, found: 388.1080.

(2*S*,4*S*)-6-benzyl-2-hydroxy-4-(naphthalen-2-yl)-3,4,5,6-tetrahydropyrano[2,3-*c*]pyrrol-7(2*H*)-one 6m



According to the general procedure, the crude product was purified via column chromatography delivering **6m** as a white solid with 83% yield. The diastereomeric ratio was determined to be 84:16 by crude ^{1}H-NMR analysis. **6m** was transformed to its analogue **6m'** (only a single diastereoisomer was obtained) with 82% yield. The enantiomeric excess of **6m'** was determined to be 96% by chiral HPLC analysis on Chiralpak AD-H column (25% 2-propanol/*n*-hexane, 1 mL/min), UV 220 nm, t_{minor} = 17.36 min, t_{major} = 33.53 min; $[\alpha]_D^{20}$ = +45.4 (*c* = 1.21 in CH_2Cl_2, *data for* **6m'**).
NMR and HRMS data for the product **6m'** :
^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm): 7.83 – 7.76 (m, 3H), 7.66 (br s, 1H), 7.53 – 7.46 (m, 2H), 7.31 – 7.22 (m, 4H), 7.19 – 7.17 (m, 2H), 6.48 (t, *J* = 2.4 Hz, 1H), 4.75 (d, *J* = 14.8 Hz, 1H), 4.42 (d, *J* = 14.8 Hz, 1H), 4.24 (dd, *J* = 11.6 Hz, *J* = 6.0 Hz, 1H), 3.59 (dd, *J* = 18.4 Hz, *J* = 1.6 Hz, 1H), 3.45 (dd, *J* = 18.4 Hz, *J* = 1.6 Hz, 1H), 2.59 – 2.45 (m, 2H).
^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm): 164.3, 141.9, 136.9, 136.6, 133.4, 132.7, 129.1, 128.7, 128.1, 127.7, 127.6, 127.5, 127.1, 126.6, 126.3, 125.3, 125.2, 88.4, 47.5, 46.5, 39.4, 34.9
HRMS (ESI): *m/z* calculated for $C_{24}H_{20}ClNO_2Na^+$: 412.1080, found: 412.1081.

5. Procedure for Synthetic Transformations of 4a



A dry glass tube was charged with **4a** (33.5 mg, 0.1 mmol), Ac_2O (51 mg, 0.5 mmol) and DMAP (1.2 mg, 0.01 mmol) in 1 mL pyridine, and the reaction was stirred at room

temperature for 2 hours. When the reaction was completed, 2 mL of 20% hydrochloric acid was added to the reaction mixture, and the organic material was extracted with ethyl acetate, washed with brine and dried over anhydrous Na_2SO_4, then concentrated under reduced pressure to give the corresponding acetylated compound; such compound was dissolved in 2 mL of DCM under argon, and $TiCl_4$ (94.5 mg, 0.5 mmol) was added into the reaction mixture. The reaction was stirred at room temperature for 16 hours. When the reaction was completed, 2 mL DCM and 1mL water was added to the reaction mixture. The organic layer was separated, dried over anhydrous Na_2SO_4 and purified by column chromatography on silica gel eluting with petroleum ether/ethyl acetate (6:1 to 4:1) to afford the desired pure product **4a'** as a white solid with 88% yield. The diastereomeric ratio was determined to be >95:5 by crude ^{1}H-NMR analysis, and the enantiomeric excess was determined to be 99% by chiral HPLC analysis on Chiralpak AD-H column (30% 2-propanol/*n*-hexane, 1 mL/min), UV 220 nm, t_{major} = 23.32 min, t_{minor} = 25.87 min; $[\alpha]_D^{20}$ = +190.2 (*c* = 0.67 in CH_2Cl_2).

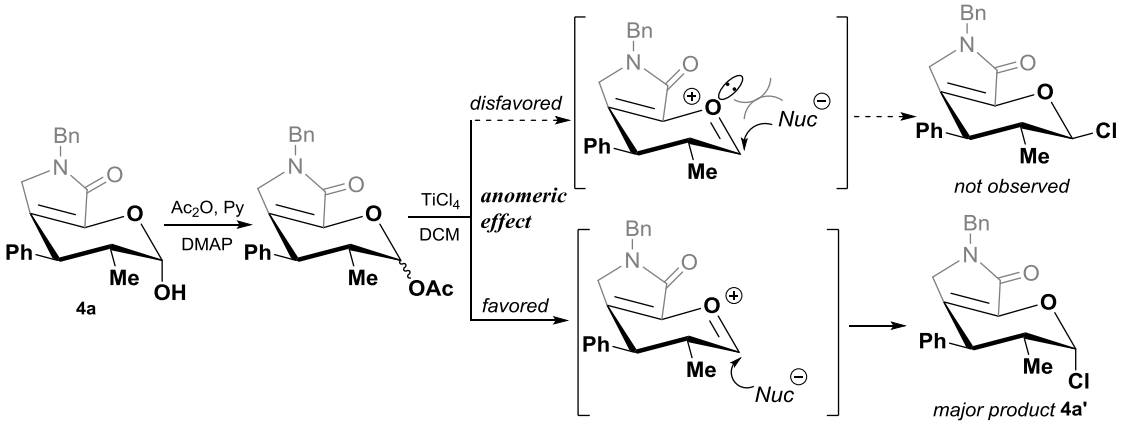
NMR and HRMS data for the product **4a'**:

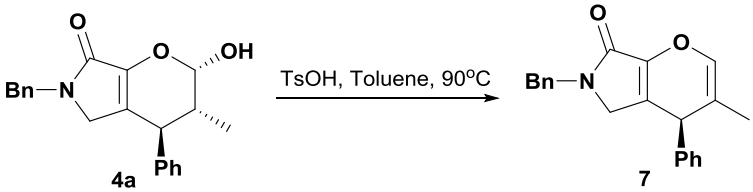
^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm): 7.35 – 7.23 (m, 6H), 7.20 – 7.17 (m, 2H), 7.13 – 7.11 (m, 2H), 6.21 (d, *J* = 2.4 Hz, 1H), 4.74 (d, *J* = 14.8 Hz, 1H), 4.42 (d, *J* = 14.8 Hz, 1H), 3.55 – 3.49 (m, 2H), 3.37 (d, *J* = 18.4 Hz, 1H), 2.47 – 2.38 (m, 1H), 0.94 (d, *J* = 6.8 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm): 164.4, 141.7, 138.7, 136.7, 129.0, 128.7, 128.4, 128.0, 127.7, 127.6, 126.1, 94.4, 47.3, 46.5, 42.0, 41.9, 14.8

HRMS (ESI): *m/z* calculated for $C_{21}H_{20}ClNO_2Na^+$: 376.1080, found: 376.1077.

- In order to rationalize the configuration of newly formed C-Cl bond in the chloro-compound **4a'**, a proposed reaction mechanism and transition state of the above synthetic transformation was described. As shown in the following scheme, the Ac protected hydroxyl group was firstly eliminated in the presence of Lewis acid. Then, because of anomeric effect of the dihydropyran, the nucloephilic chloride preferred to attack the oxonium intermediate from the bottom face, generating a configurationally favored axial bond. Thus, the corresponding product **4a'** with excellent stereoselectivity was obtained.





A dry glass tube was charged with **4a** (33.5 mg, 0.1 mmol), TsOH (86 mg, 0.5 mmol) in 1 mL toluene, and the reaction was stirred at 90 ^{o}C for 2 hours. When the reaction was completed, the reaction mixture was directly purified by column chromatography on silica gel eluting with petroleum ether/ethyl acetate (6:1 to 4:1) to afford the desired pure product **7** as a white solid with 85% yield. The enantiomeric excess was determined to be 99% by chiral HPLC analysis on Chiralpak AD-H column (20% 2-propanol/*n*-hexane, 1 mL/min), UV 220 nm, t_{major} = 16.47 min, t_{minor} = 35.97 min; $[\alpha]_D^{20}$ = +10.4 (*c* = 1.90 in CH_2Cl_2).

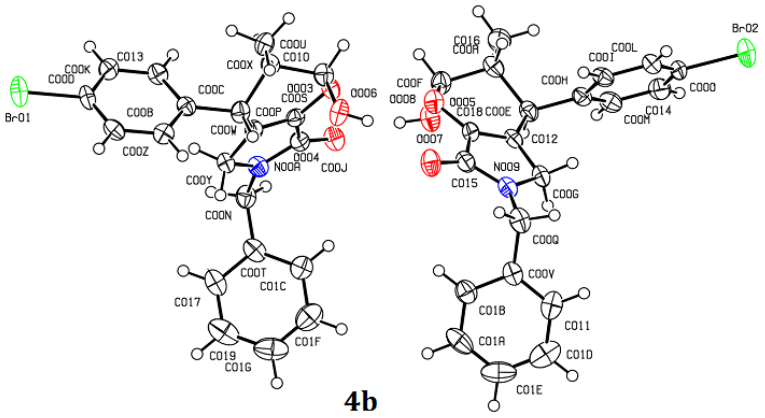
NMR and HRMS data for the product **7**:

^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm): 7.33 – 7.22 (m, 6H), 7.19 – 7.16 (m, 4H), 6.51 (s, 1H), 4.82 (d, *J* = 14.8 Hz, 1H), 4.31 (d, *J* = 14.8 Hz, 1H), 4.08 (s, 1H), 3.53 (d, *J* = 18.4 Hz, 1H), 3.34 (d, *J* = 18.4 Hz, 1H), 1.42 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm): 164.1, 141.4, 141.2, 136.7, 135.9, 128.8, 128.7, 128.0, 127.9, 127.6, 127.4, 123.1, 111.7, 47.3, 46.3, 43.2, 16.2

HRMS (ESI): *m/z* calculated for $C_{21}H_{19}NO_2Na^+$: 340.1313, found: 340.1316.

6. Crystal Data and Structure Refinement for the Enantiopure 4b



4b

Identification code	**4b**
Empirical formula	$C_{42}H_{40}Br_2N_2O_6$
Formula weight	828.58
Temperature/K	292.71(10)
Crystal system	monoclinic
Space group	$P2_1$
a/Å	9.5936(4)
b/Å	17.0134(7)
c/Å	12.4020(5)
α/°	90
β/°	111.095(5)
γ/°	90
Volume/Å^3	1888.61(14)
Z	2
ρ_{calc}g/cm^3	1.457
μ/mm^{-1}	3.132
F(000)	848.0
Crystal size/mm^3	0.5 × 0.3 × 0.2
Radiation	CuKα (λ = 1.54184)
2Θ range for data collection/°	9.244 to 134.144
Index ranges	$-10 \leq h \leq 11$, $-20 \leq k \leq 20$, $-13 \leq l \leq 14$
Reflections collected	20048
Independent reflections	6542 [R_{int} = 0.0568, R_{sigma} = 0.0594]

Data/restraints/parameters	6542/1/473
Goodness-of-fit on F^2	1.014
Final R indexes [I>=2σ (I)]	R_1 = 0.0546, wR_2 = 0.1360
Final R indexes [all data]	R_1 = 0.0616, wR_2 = 0.1437
Largest diff. peak/hole / e $Å^{-3}$	0.83/-0.52
Flack parameter	0.030(15)

- CCDC 1480846 (**4b**) contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from the Cambridge Crystallographic Data Centre *via* www.ccdc.cam.ac.uk/data_request/cif.

- From the crystallographic data, an intermolecular hydrogen bonding interaction was observed. The data of such hydrogen bonding interaction is listed as follows:

  Hydrogen bonds with H..A < r(A) + 2.000 Angstroms and <DHA > 110 deg.
  Appropriate HTAB instructions appended to .res file for future use.

D-H	d(D-H)	d(H..A)	<DHA	d(D..A)	A
O006-H006	0.840	2.321	144.94	3.047	O005
O006-H006	0.840	2.326	135.34	2.983	O007
O008-H008	0.840	2.052	155.10	2.836	O004
C00N-H00C	0.990	2.597	116.61	3.167	O006 [-x+1, y-1/2, -z+1]
C00N-H00C	0.990	2.472	170.48	3.452	O007 [-x+1, y-1/2, -z+1]

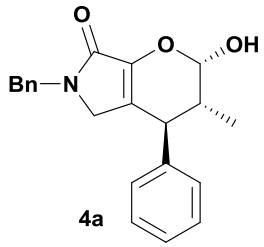
7. References and notes

[1] (a) E. Krell, *Handbook of Laboratory Distillation*, Elseriver Publishing Company,Amsterdam-London-New York, 1963; b) M. J. Rosengart, *The Technique of Distillation and Rectification in the Laboratory*, VEB Verlag Technik, Berlin, 1954; c) H. Stage *Columns for laboratory distillation*, *Angew. Chem.*, 1947, **B19**, 175.

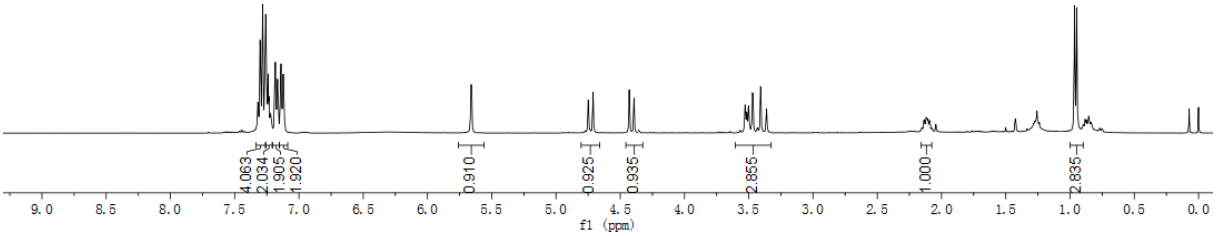
[2] P. L. Southwick, E. F. Barnas, *J. Org. Chem.*, 1962, **27**, 98.

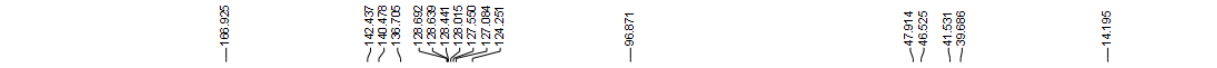
[3] For detail of the procedure for such function group transformation, see in page S27: *Procedure for Synthetic Transformations of **4a***.

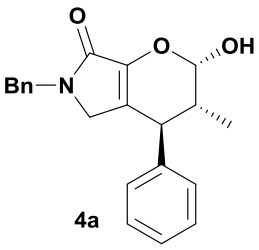
8. NMR and HPLC Spectra of the Chiral Bicyclic Dihydropyranes



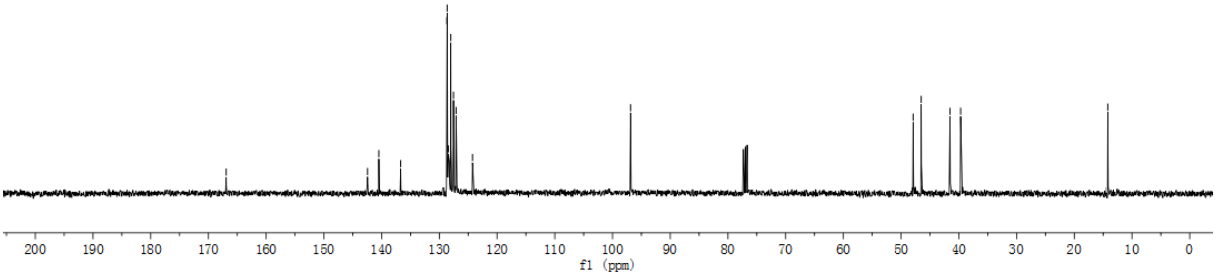
4a





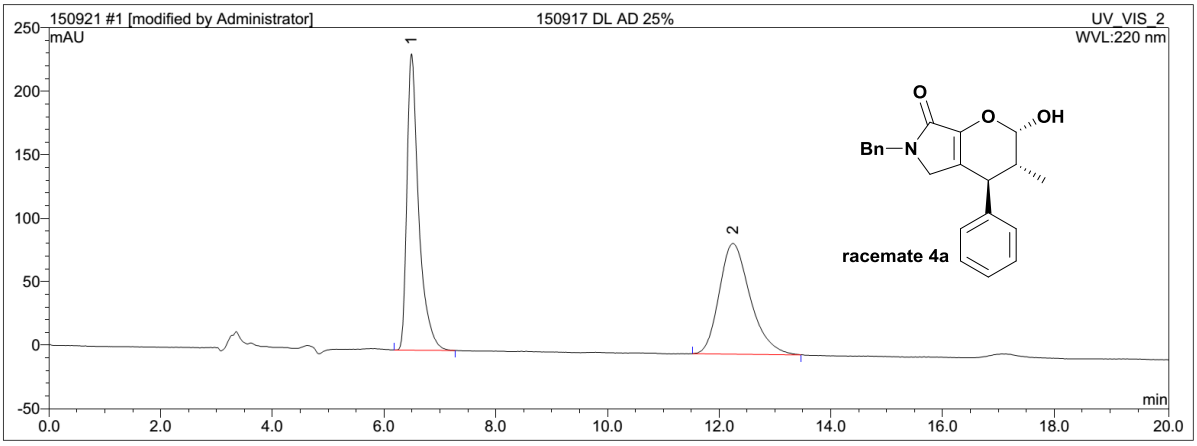


4a



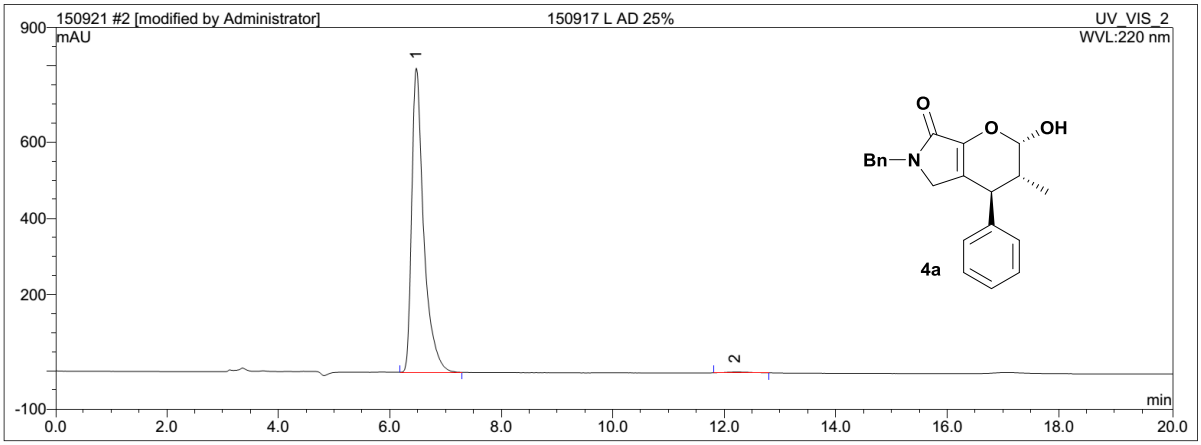
Peak Analysis Report

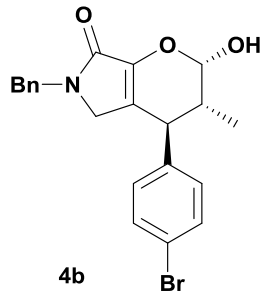
No.	Peak Name	Ret.Time (detected)	Area	Rel.Area	Height	Amount
		min	mAU*min	%	mAU	
1	n.a.	6.49	57.124	50.47	233.590	n.a.
2	n.a.	12.25	56.054	49.53	87.234	n.a.



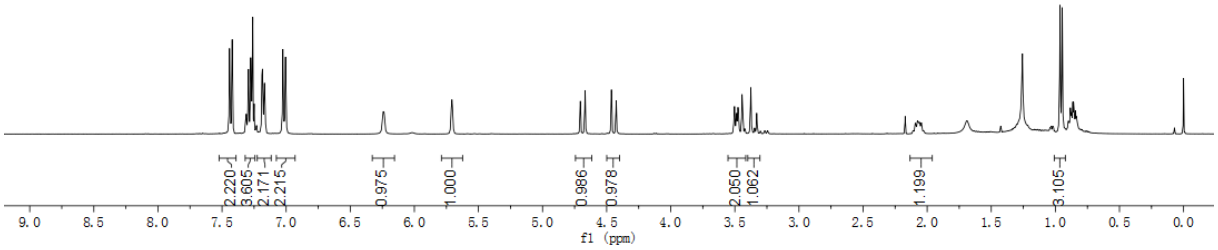
Peak Analysis Report

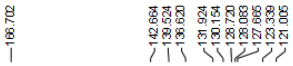
No.	Peak Name	Ret.Time (detected)	Area	Rel.Area	Height	Amount
		min	mAU*min	%	mAU	
1	n.a.	6.48	198.933	99.44	797.842	n.a.
2	n.a.	12.19	1.125	0.56	2.320	n.a.

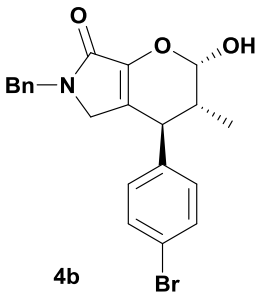



O
O
OH
Bn—N
Br

4b


2.220
3.605
2.171
2.215
0.975
1.000
0.986
0.978
2.050
1.062
1.199
3.105
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


166.702
142.664
139.524
136.620
131.924
130.154
128.720
128.083
127.695
123.339
121.005
96.771
47.771
46.567
41.131
39.757
14.166

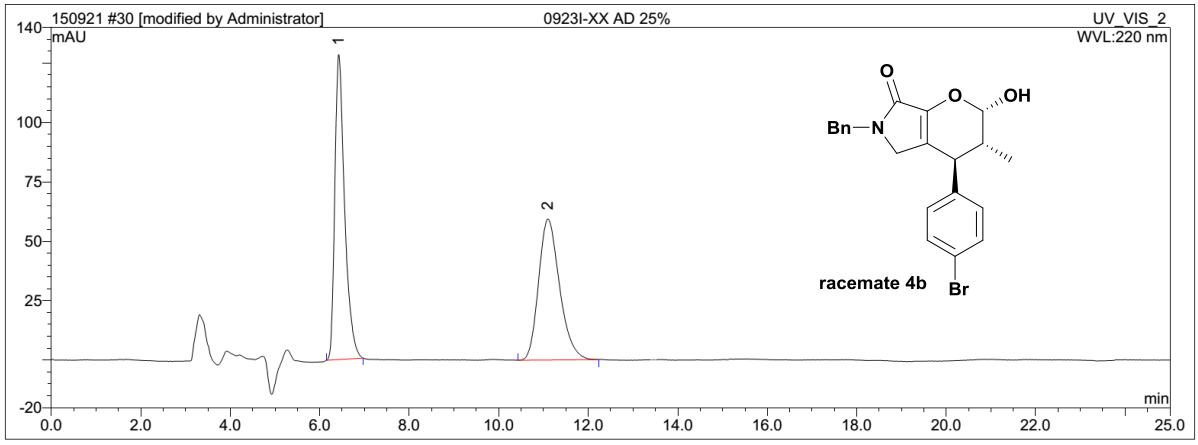

O
O
OH
Bn—N
Br

4b

200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

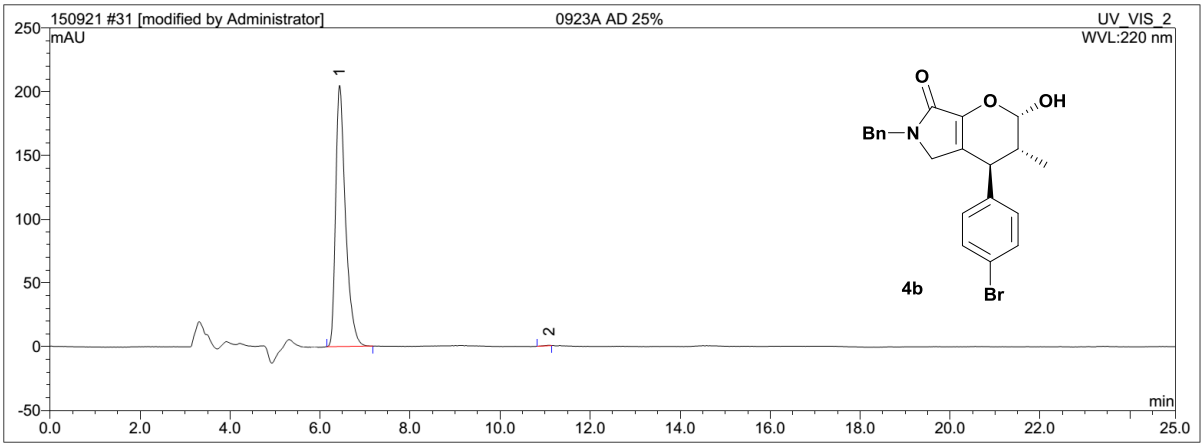
Peak Analysis Report

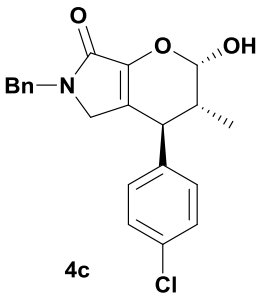
No.	Peak Name	Ret.Time (detected)	Area	Rel.Area	Height	Amount
		min	mAU*min	%	mAU	
1	n.a.	6.43	32.598	50.30	128.425	n.a.
2	n.a.	11.10	32.211	49.70	59.416	n.a.

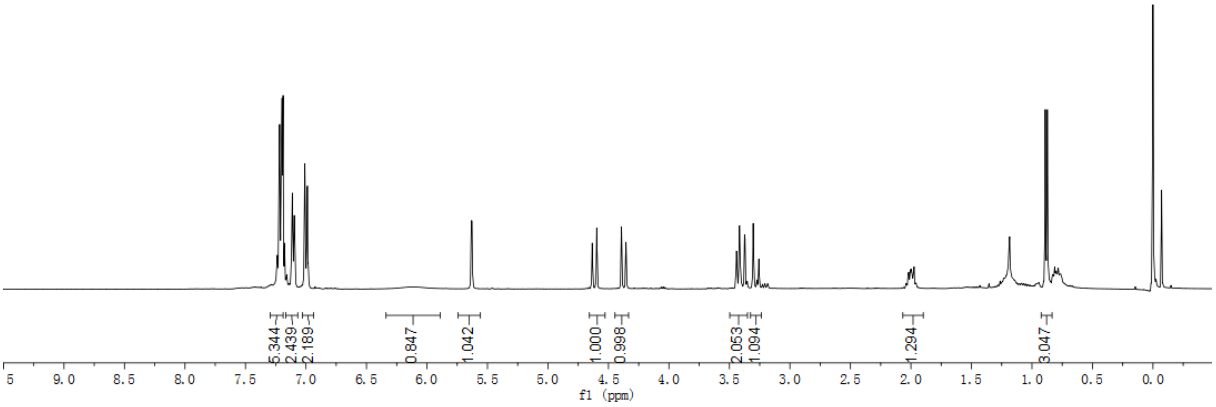


Peak Analysis Report

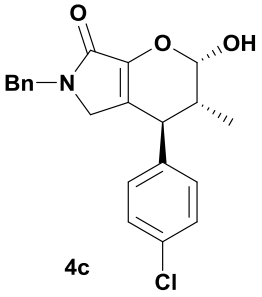
No.	Peak Name	Ret.Time (detected)	Area	Rel.Area	Height	Amount
		min	mAU*min	%	mAU	
1	n.a.	6.43	51.886	99.87	204.943	n.a.
2	n.a.	11.10	0.068	0.13	0.369	n.a.




O
O
OH
Bn—N
4c
Cl


5.344
2.439
2.189
0.847
1.042
1.000
0.998
2.053
1.094
1.294
3.047
f1 (ppm)

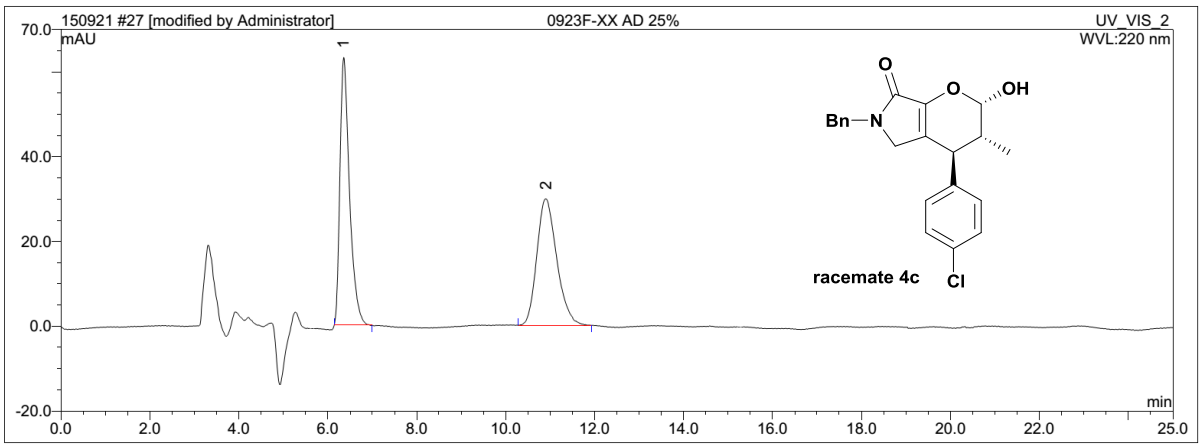

166.712
142.623
138.963
136.618
132.963
129.751
128.974
128.719
128.074
127.665
123.499
96.790
47.784
46.572
41.047
39.777
14.149


O
O
OH
Bn—N
4c
Cl

f1 (ppm)

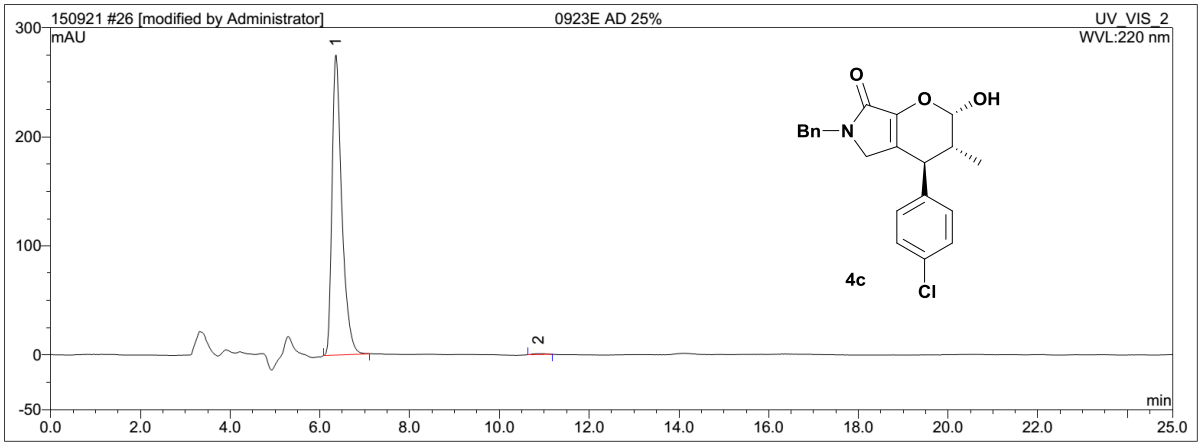
Peak Analysis Report

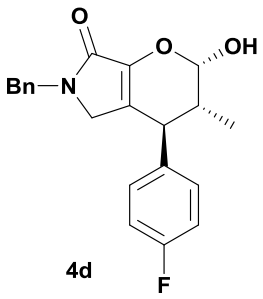
No.	Peak Name	Ret.Time (detected)	Area	Rel.Area	Height	Amount
		min	mAU*min	%	mAU	
1	n.a.	6.36	15.786	50.45	63.212	n.a.
2	n.a.	10.91	15.505	49.55	29.892	n.a.

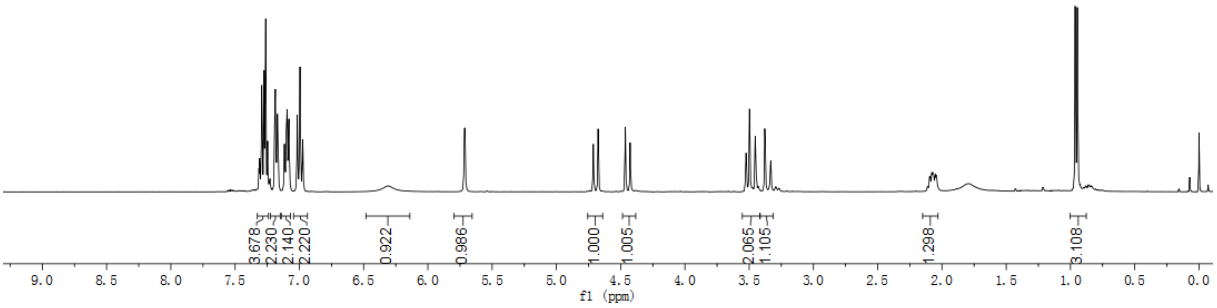


Peak Analysis Report

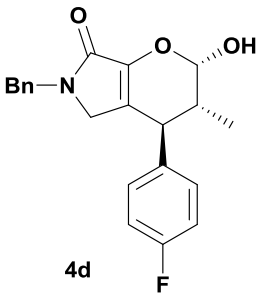
No.	Peak Name	Ret.Time (detected)	Area	Rel.Area	Height	Amount
		min	mAU*min	%	mAU	
1	n.a.	6.36	69.336	99.67	274.857	n.a.
2	n.a.	10.88	0.232	0.33	0.770	n.a.




O
O
OH
Bn—N
4d
F


3.678
2.230
2.140
2.220
0.922
0.986
1.000
1.005
2.065
1.105
1.298
3.108
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

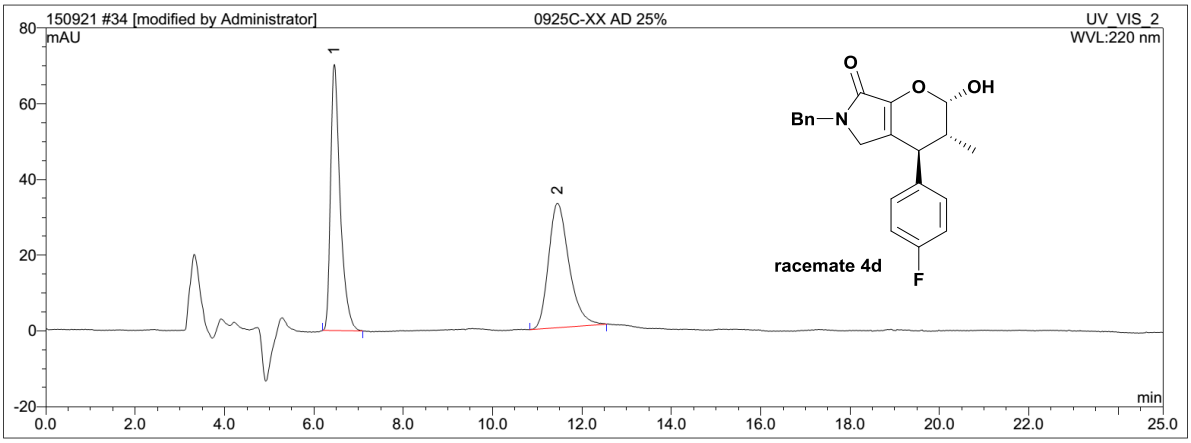

166.794
163.108
160.667
142.522
136.666
136.113
136.082
129.835
129.698
129.651
127.633
123.839
115.773
115.561
96.838
47.819
46.550
40.867
39.917
14.157


O
O
OH
Bn—N
4d
F

190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

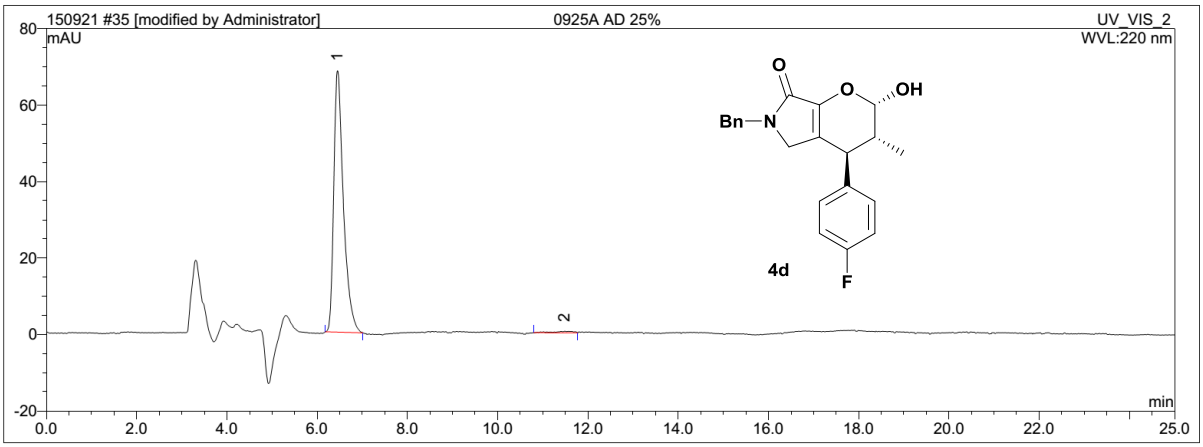
Peak Analysis Report

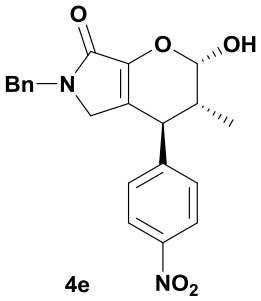
No.	Peak Name	Ret.Time (detected)	Area	Rel.Area	Height	Amount
		min	mAU*min	%	mAU	
1	n.a.	6.46	17.471	49.99	70.358	n.a.
2	n.a.	11.45	17.480	50.01	32.897	n.a.

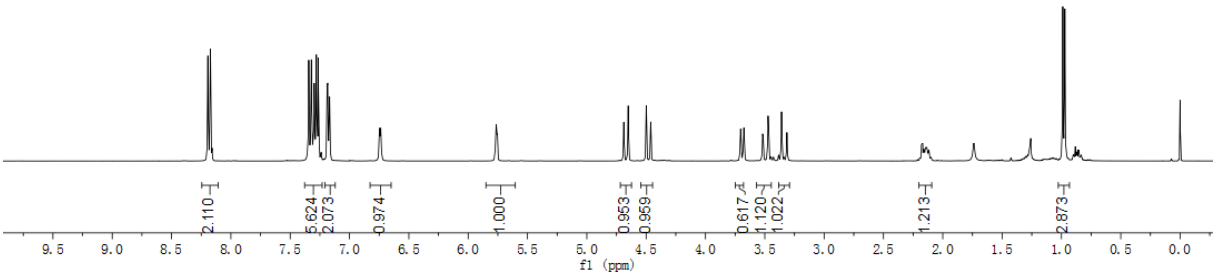


Peak Analysis Report

No.	Peak Name	Ret.Time (detected)	Area	Rel.Area	Height	Amount
		min	mAU*min	%	mAU	
1	n.a.	6.45	16.940	98.90	68.463	n.a.
2	n.a.	11.48	0.188	1.10	0.383	n.a.




O
O
OH
Bn—N
4e
NO2


2.110
5.624
2.073
0.974
1.000
0.953
0.959
0.617
1.120
1.022
1.213
2.873
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


166.632
148.386
147.251
143.112
136.368
129.339
128.770
128.076
127.784
124.070
122.060
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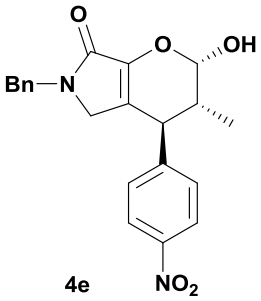
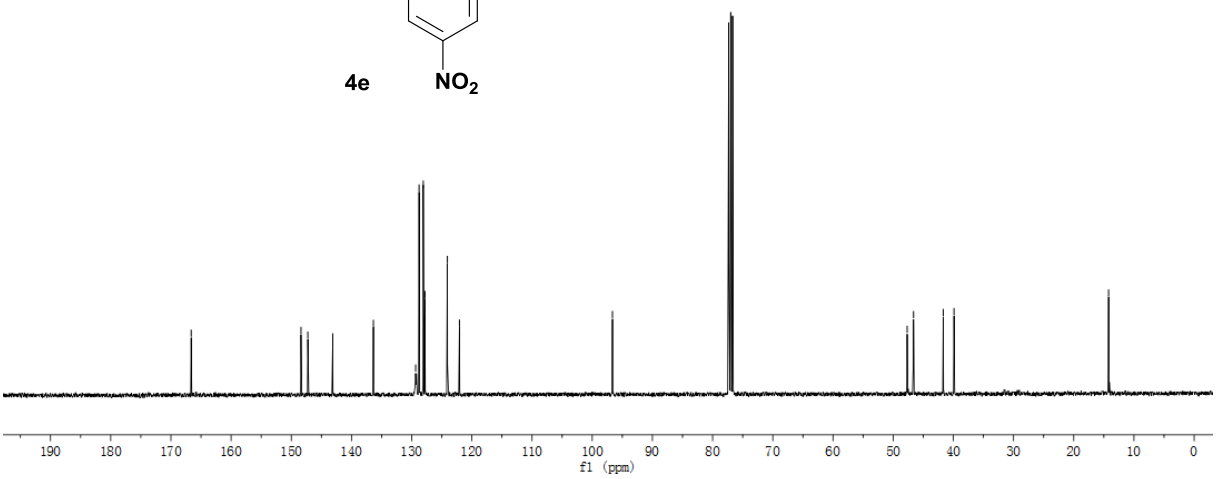
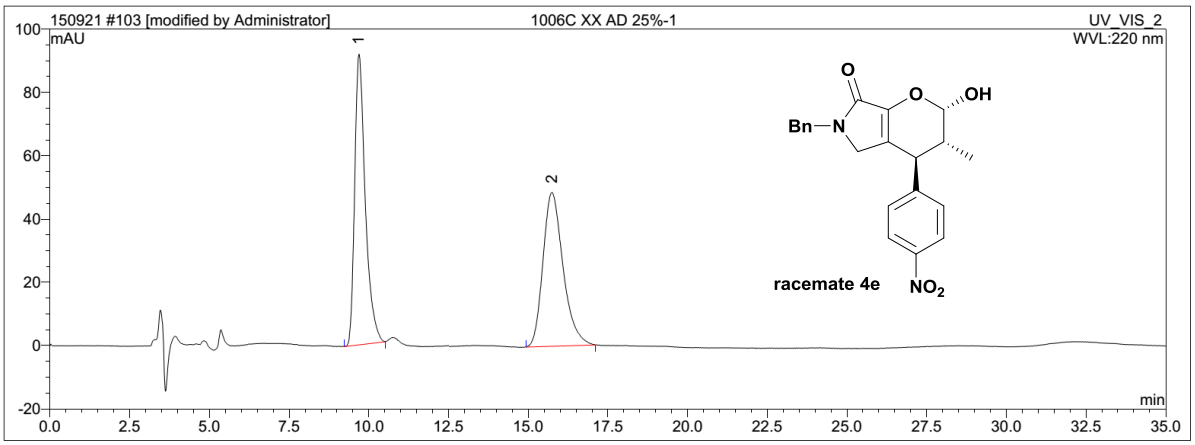
47.677
46.619
41.679
39.892
14.213


O
O
OH
Bn—N
4e
NO2


190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

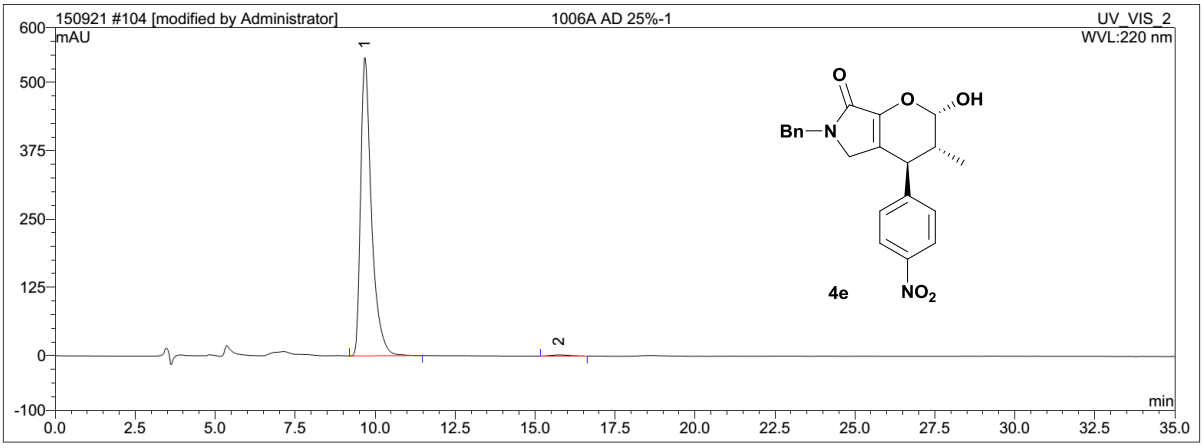
Peak Analysis Report

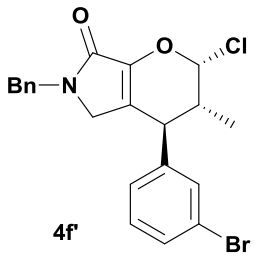
No.	Peak Name	Ret.Time (detected)	Area	Rel.Area	Height	Amount
		min	mAU*min	%	mAU	
1	n.a.	9.69	35.438	49.98	91.951	n.a.
2	n.a.	15.75	35.465	50.02	48.646	n.a.

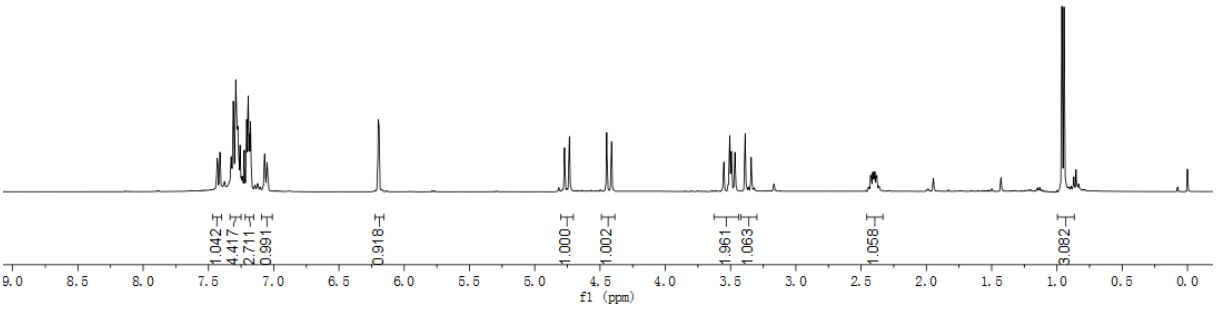


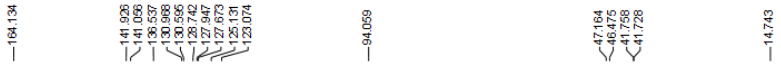
Peak Analysis Report

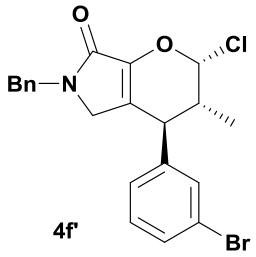
No.	Peak Name	Ret.Time (detected)	Area	Rel.Area	Height	Amount
		min	mAU*min	%	mAU	
1	n.a.	9.68	215.089	99.28	545.906	n.a.
2	n.a.	15.75	1.564	0.72	2.354	n.a.

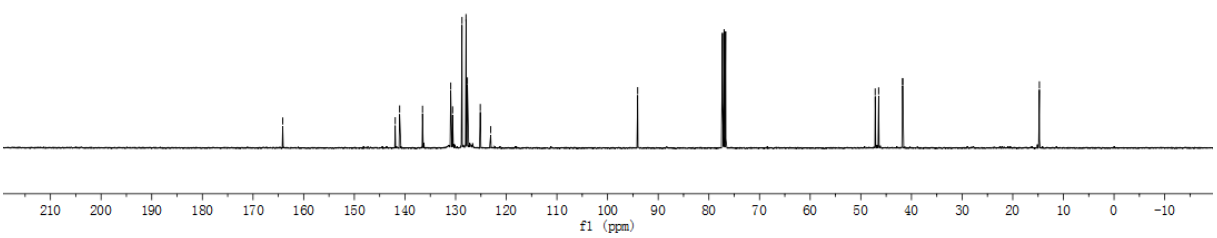



O
O
Cl
Bn
N
4f'
Br


1.042
4.417
2.711
0.991
0.918
1.000
1.002
1.961
1.063
1.058
3.082
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

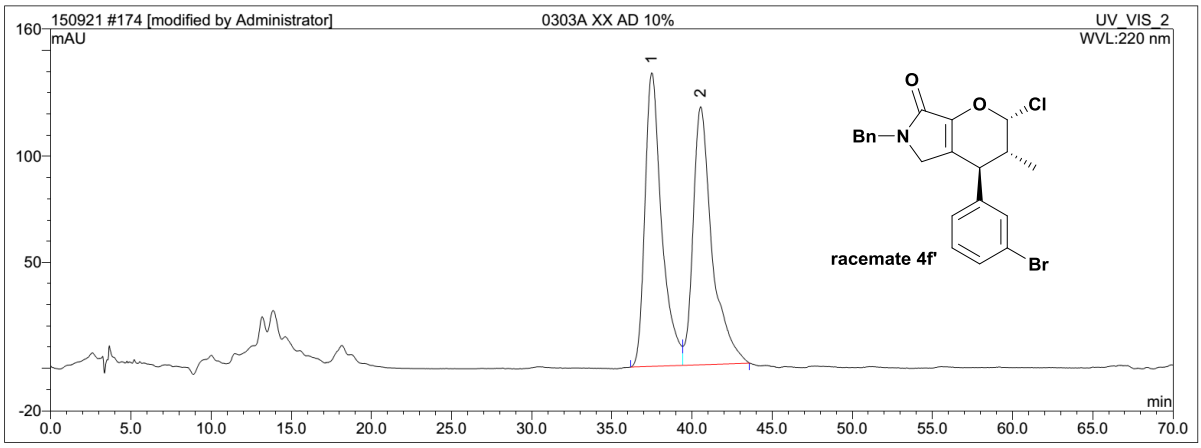

164.134
141.926
141.096
136.537
130.968
130.595
128.742
127.947
127.673
125.131
123.074
94.059
47.164
46.475
41.758
41.728
14.743


O
O
Cl
Bn
N
4f'
Br


210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

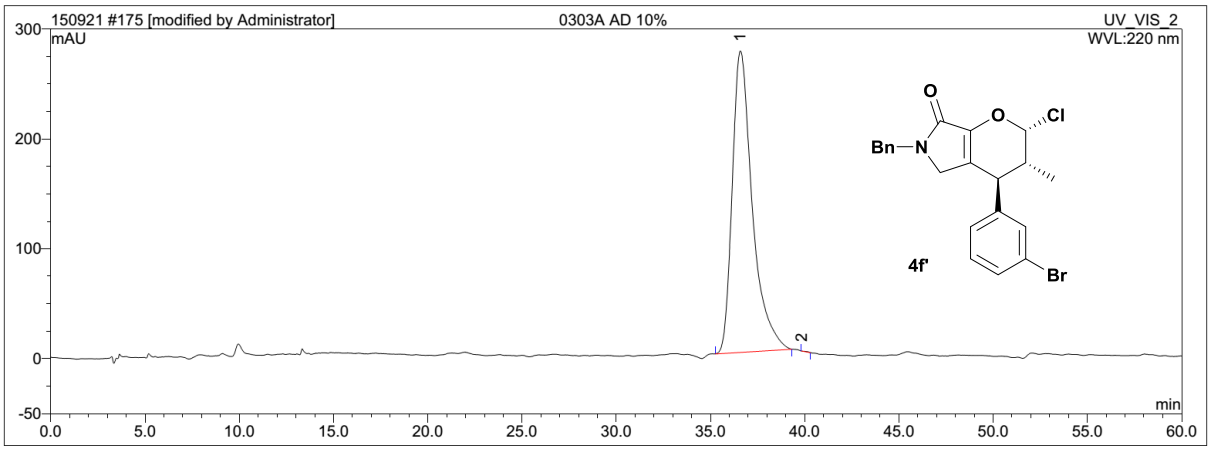
Peak Analysis Report

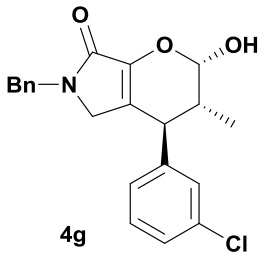
No.	Peak Name	Ret.Time (detected)	Area	Rel.Area	Height	Amount
		min	mAU*min	%	mAU	
1	n.a.	37.48	167.913	50.03	138.463	n.a.
2	n.a.	40.53	167.686	49.97	121.747	n.a.

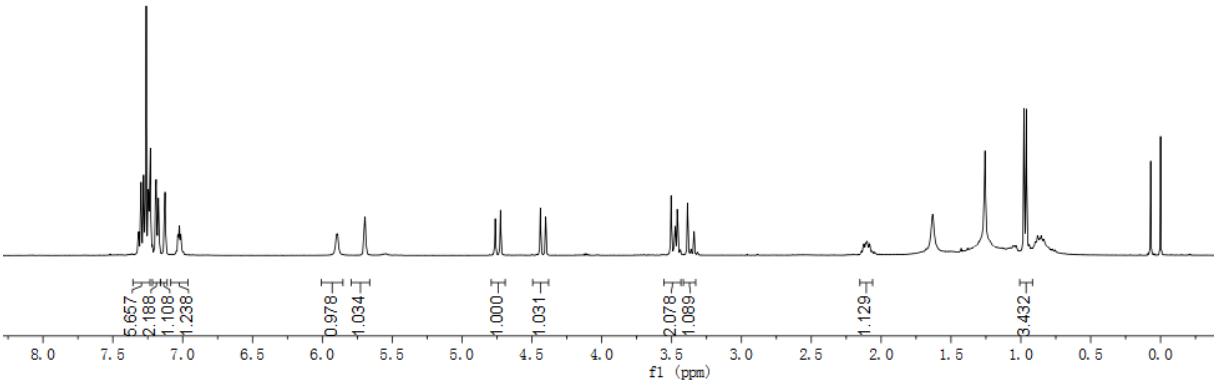


Peak Analysis Report

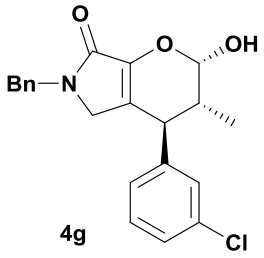
No.	Peak Name	Ret.Time (detected)	Area	Rel.Area	Height	Amount
		min	mAU*min	%	mAU	
1	n.a.	36.58	338.852	99.98	274.536	n.a.
2	n.a.	39.84	0.082	0.02	0.032	n.a.

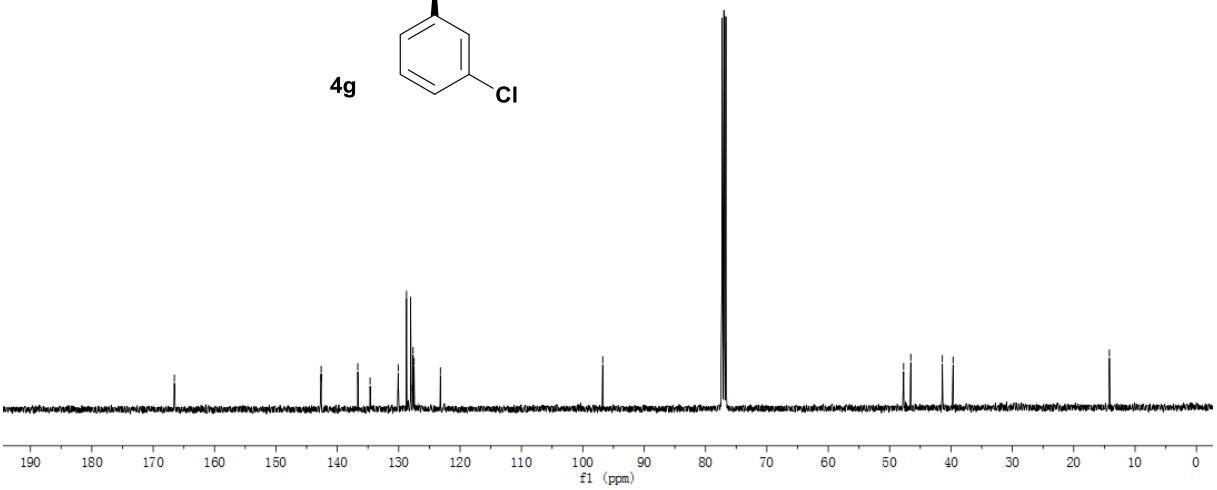



O
O
OH
Bn—N
4g
Cl


5.657
2.188
1.108
1.238
0.978
1.034
1.000
1.031
2.078
1.089
1.129
3.432
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

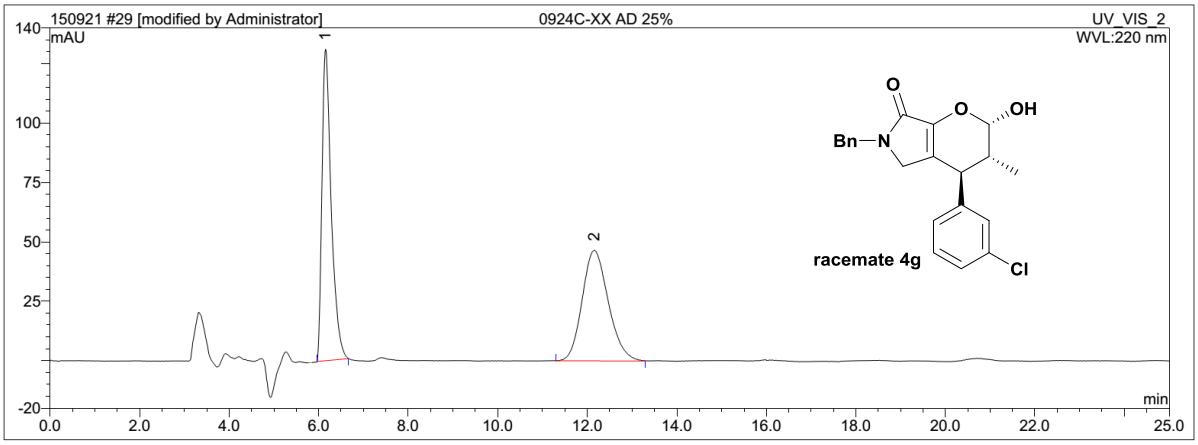

166.552
142.667
142.630
136.688
134.653
130.073
128.723
128.057
127.976
127.654
127.495
123.196
96.747
47.719
46.545
41.413
39.655
14.187


O
O
OH
Bn—N
4g
Cl


190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

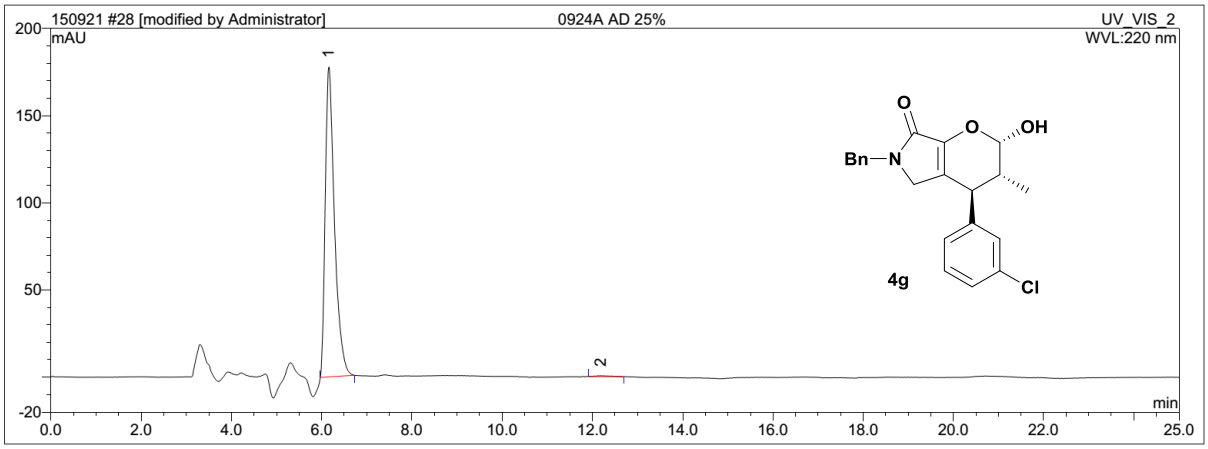
Peak Analysis Report

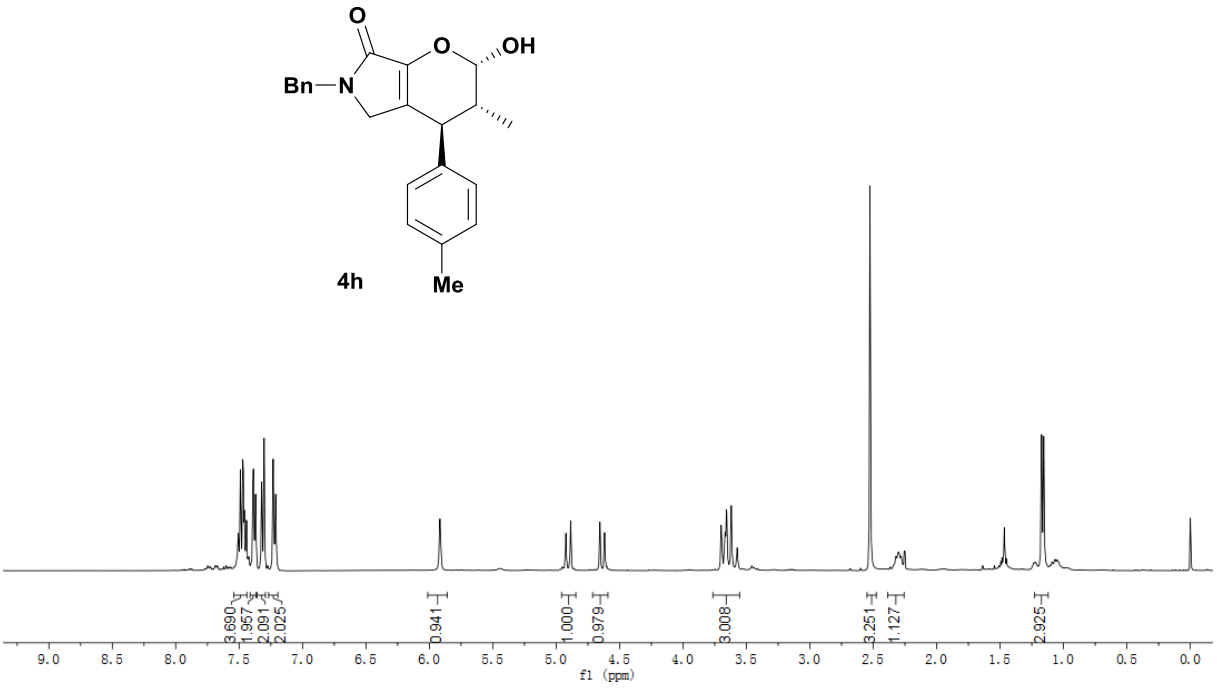
No.	Peak Name	Ret.Time (detected)	Area	Rel.Area	Height	Amount
		min	mAU*min	%	mAU	
1	n.a.	6.15	31.491	50.01	131.154	n.a.
2	n.a.	12.16	31.477	49.99	46.488	n.a.

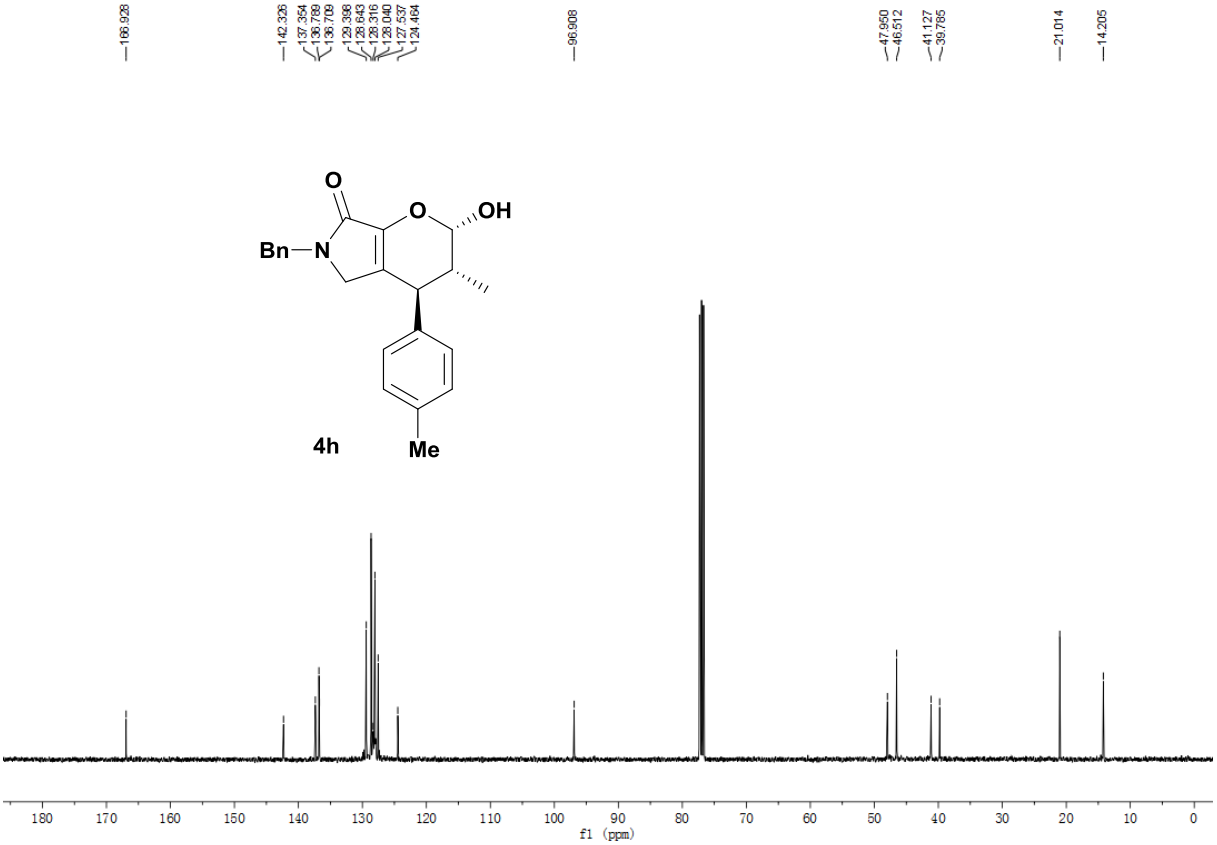


Peak Analysis Report

No.	Peak Name	Ret.Time (detected)	Area	Rel.Area	Height	Amount
		min	mAU*min	%	mAU	
1	n.a.	6.16	42.247	99.53	177.670	n.a.
2	n.a.	12.18	0.200	0.47	0.484	n.a.

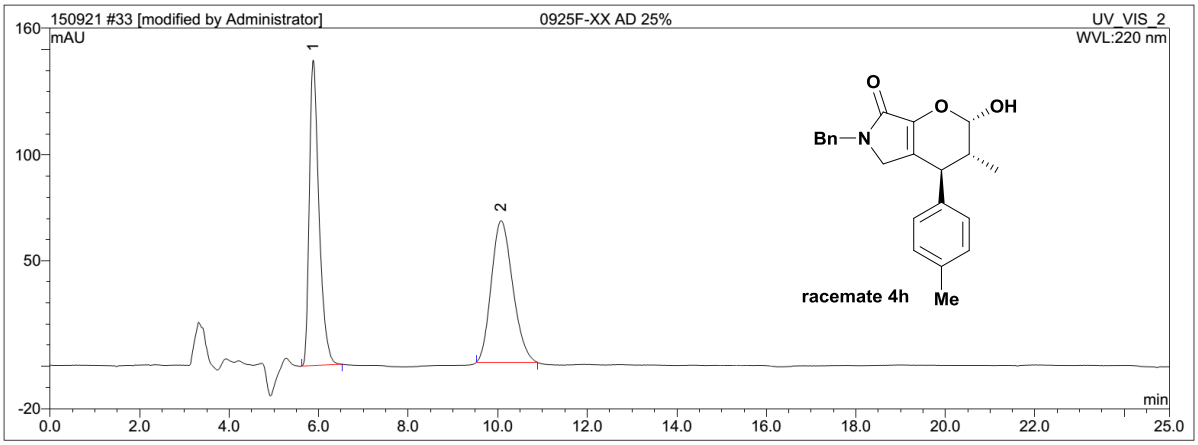



O
O
OH
Bn—N
4h
Me
3.690
1.957
2.091
2.025
0.941
1.000
0.979
3.008
3.251
1.127
2.925
f1 (ppm)


168.928
142.305
137.354
136.789
136.709
129.398
128.643
128.316
128.040
127.537
124.464
96.908
47.950
46.512
41.127
39.785
21.014
14.205
O
O
OH
Bn—N
4h
Me
f1 (ppm)

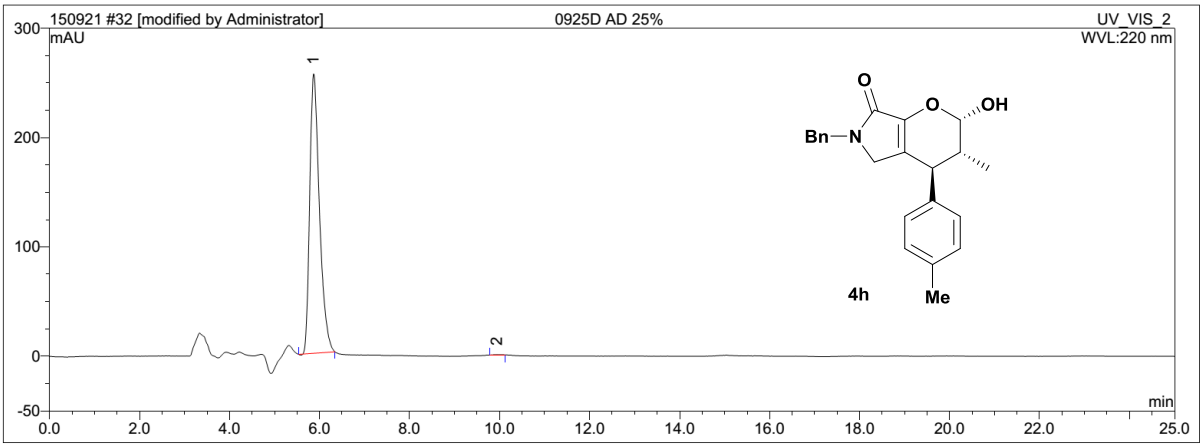
Peak Analysis Report

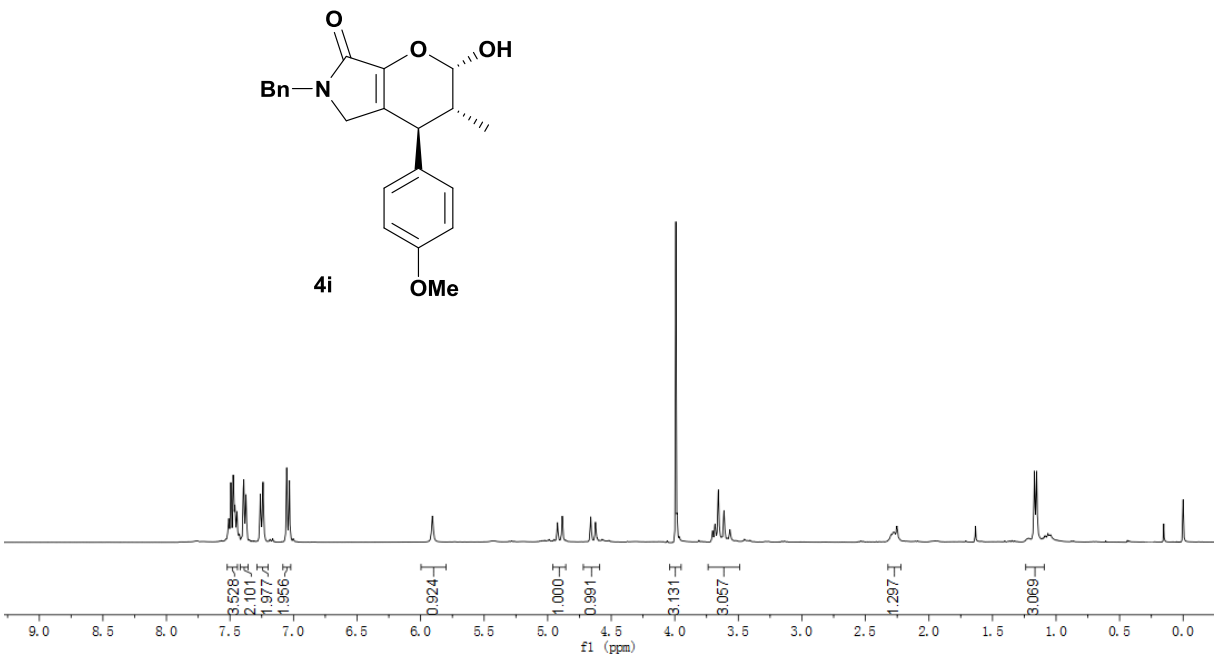
No.	Peak Name	Ret.Time (detected)	Area	Rel.Area	Height	Amount
		min	mAU*min	%	mAU	
1	n.a.	5.88	36.231	49.34	144.585	n.a.
2	n.a.	10.08	37.206	50.66	67.116	n.a.

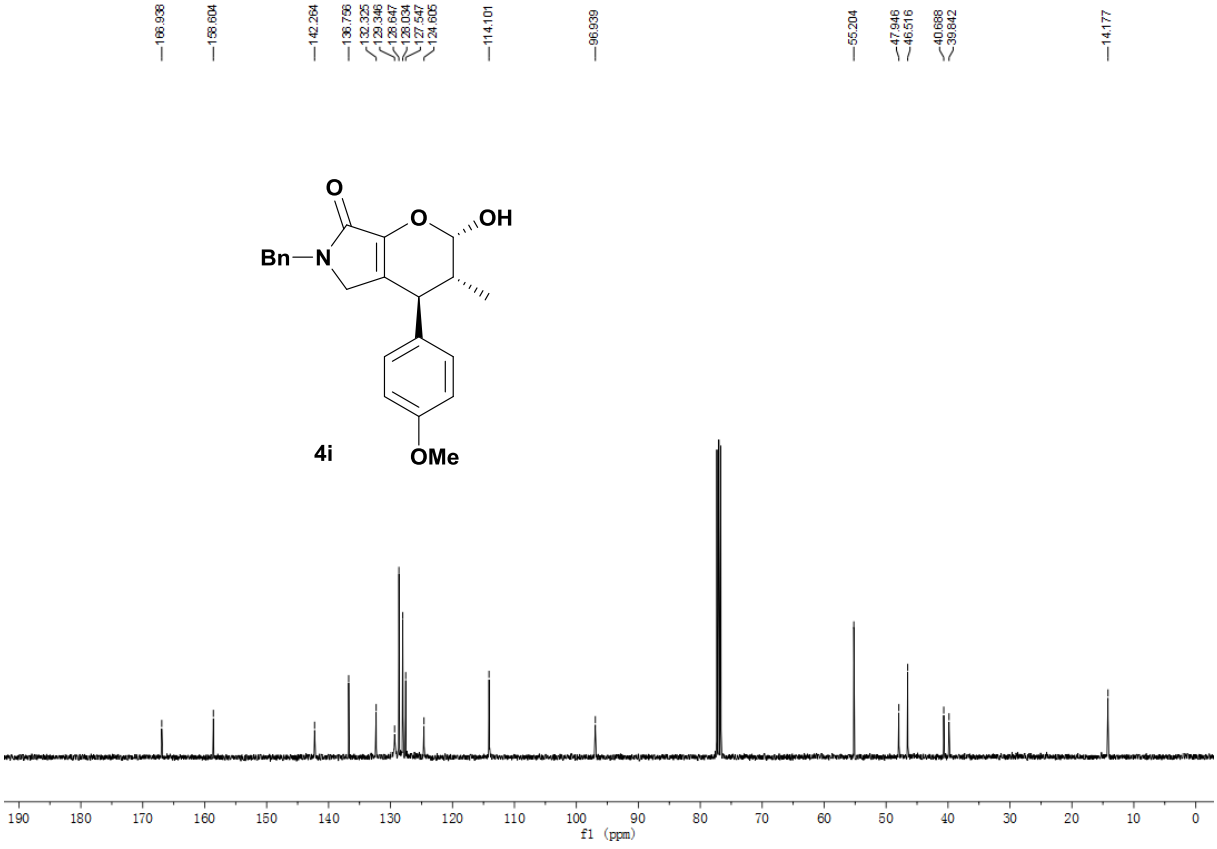


Peak Analysis Report

No.	Peak Name	Ret.Time (detected)	Area	Rel.Area	Height	Amount
		min	mAU*min	%	mAU	
1	n.a.	5.87	64.007	99.91	256.039	n.a.
2	n.a.	9.94	0.057	0.09	0.315	n.a.

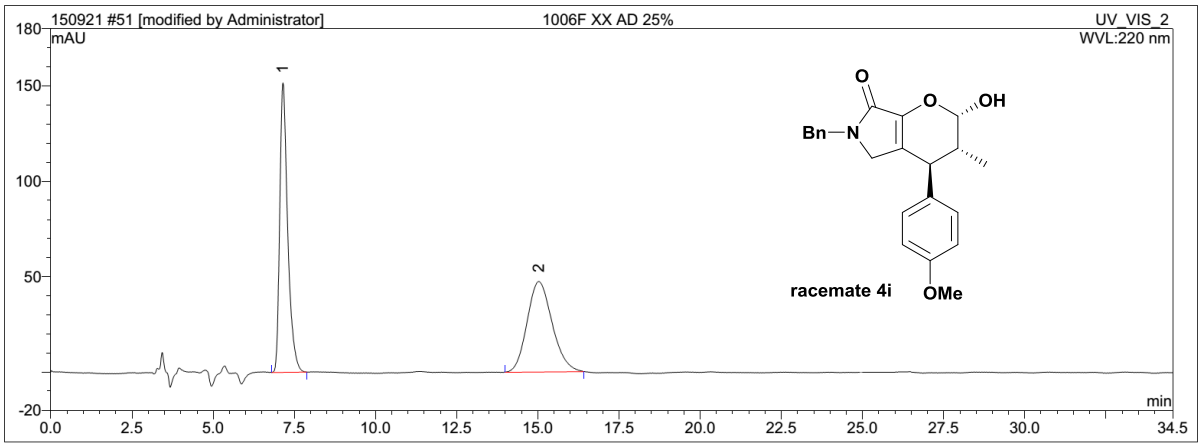



O
O
OH
Bn—N
4i
OMe
3.528
2.101
1.977
1.956
0.924
1.000
0.991
3.131
3.057
1.297
3.069
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


166.938
158.604
142.264
136.766
132.325
129.346
128.647
128.034
127.547
124.605
114.101
96.939
55.204
47.946
46.516
40.688
39.842
14.177
O
O
OH
Bn—N
4i
OMe
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

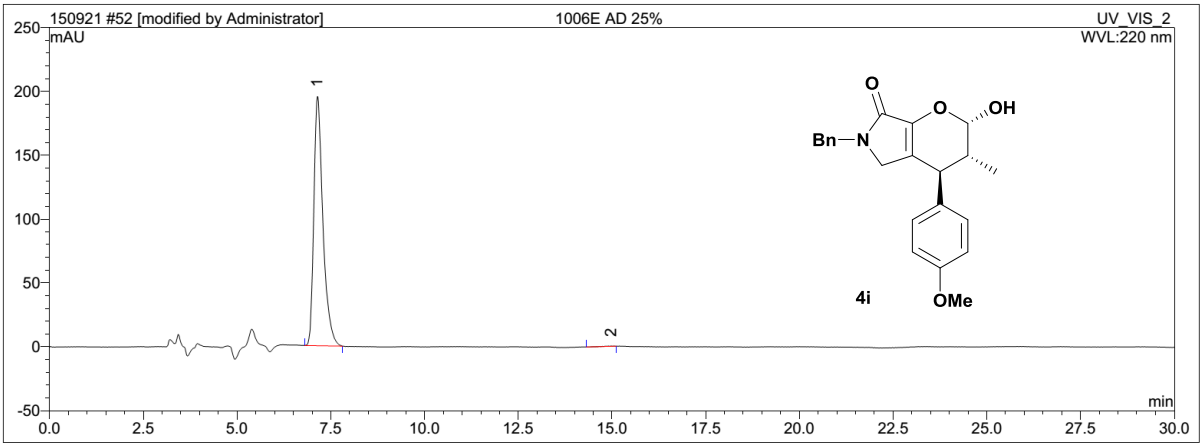
Peak Analysis Report

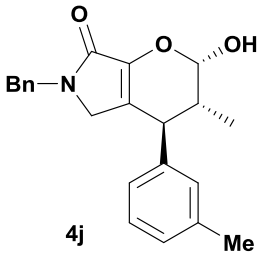
No.	Peak Name	Ret.Time (detected)	Area	Rel.Area	Height	Amount
		min	mAU*min	%	mAU	
1	n.a.	7.15	43.177	50.46	151.860	n.a.
2	n.a.	15.03	42.395	49.54	47.389	n.a.

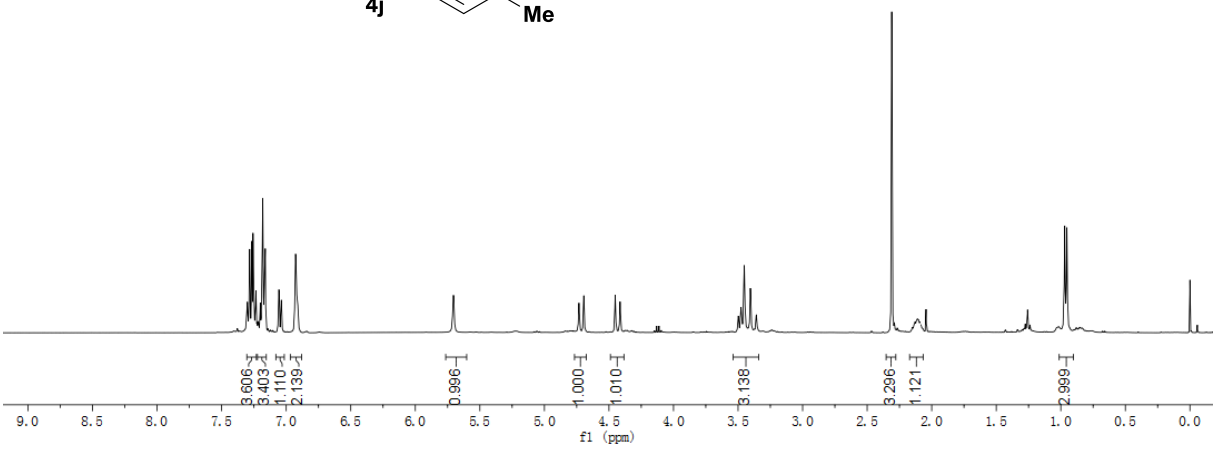


Peak Analysis Report

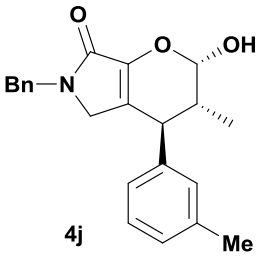
No.	Peak Name	Ret.Time (detected)	Area	Rel.Area	Height	Amount
		min	mAU*min	%	mAU	
1	n.a.	7.14	55.097	99.86	195.301	n.a.
2	n.a.	14.98	0.078	0.14	0.214	n.a.

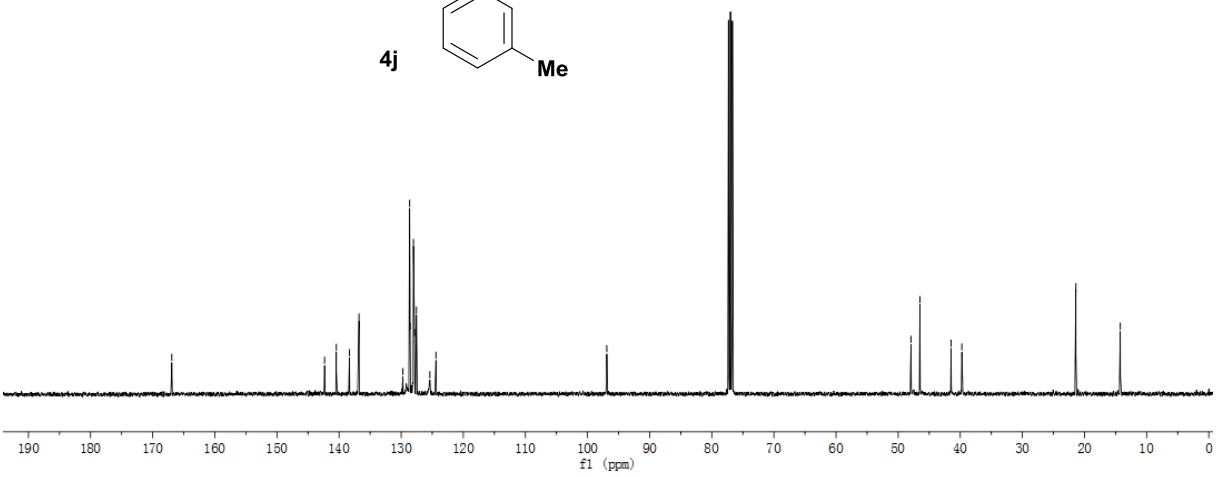



O
O
OH
Bn—N
4j
Me


3.606
3.403
1.110
2.139
0.996
1.000
1.010
3.138
3.296
1.121
2.999
f1 (ppm)

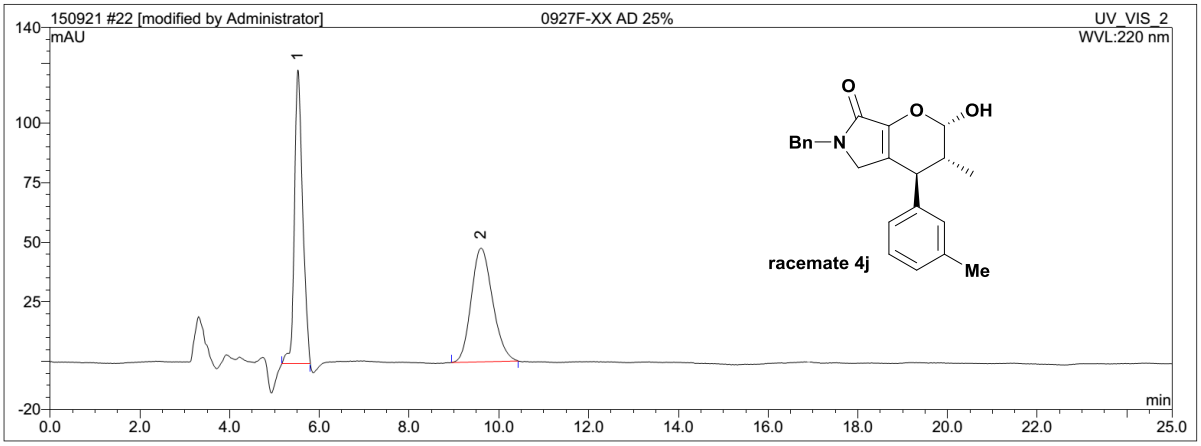

166.931
142.324
140.446
138.295
136.797
129.729
128.641
128.561
128.022
127.846
127.548
125.384
124.405
96.901
47.935
46.496
41.485
39.746
21.395
14.247


O
O
OH
Bn—N
4j
Me


f1 (ppm)

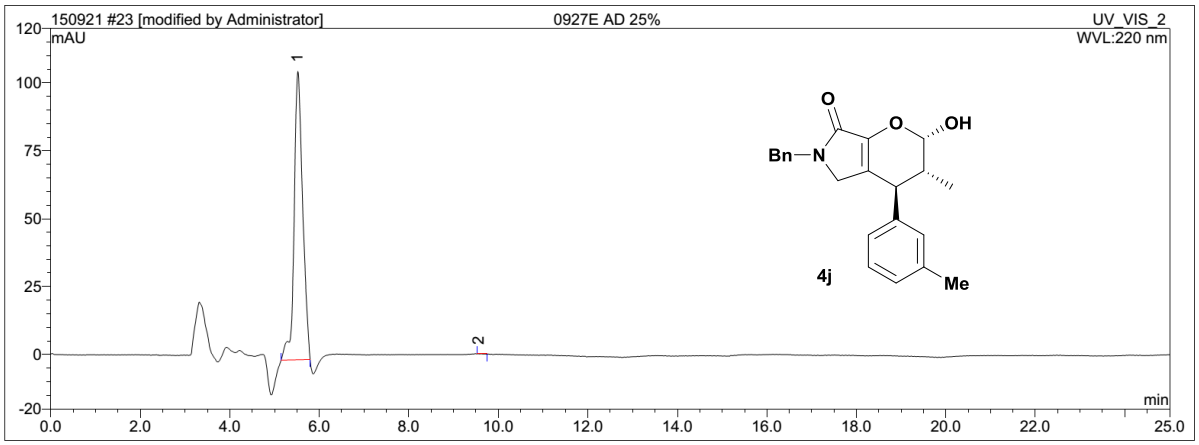
Peak Analysis Report

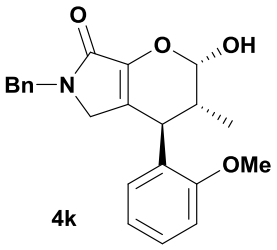
No.	Peak Name	Ret.Time (detected)	Area	Rel.Area	Height	Amount
		min	mAU*min	%	mAU	
1	n.a.	5.52	26.015	49.75	123.359	n.a.
2	n.a.	9.60	26.272	50.25	47.637	n.a.



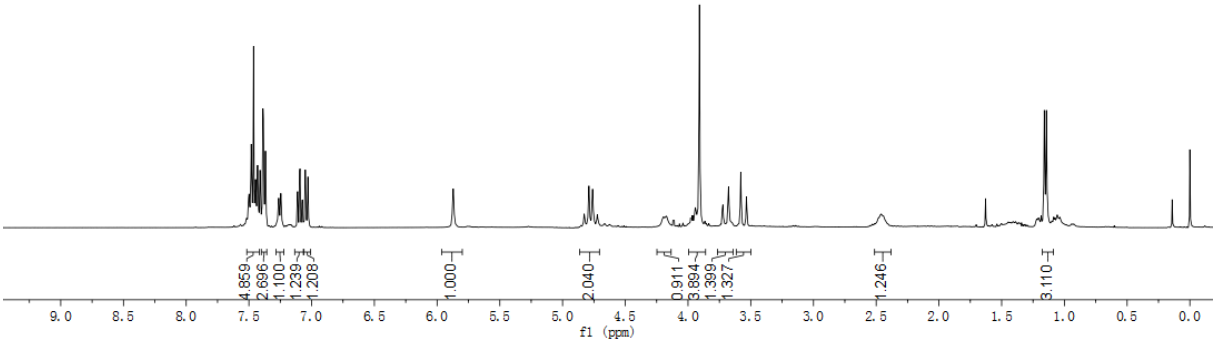
Peak Analysis Report

No.	Peak Name	Ret.Time (detected)	Area	Rel.Area	Height	Amount
		min	mAU*min	%	mAU	
1	n.a.	5.52	23.436	99.95	106.157	n.a.
2	n.a.	9.56	0.011	0.05	0.024	n.a.

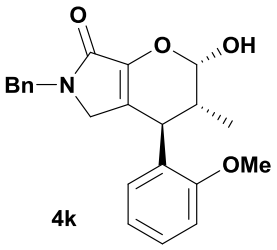



O
O
OH
Bn—N
OMe

4k


4.859
2.696
1.100
1.239
1.208
1.000
2.040
0.911
3.894
1.399
1.327
1.246
3.110
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


166.936
157.781
141.821
137.023
129.969
128.596
128.273
128.066
127.920
127.448
125.011
120.688
110.728
96.938
60.383
55.158
48.033
46.400
14.085

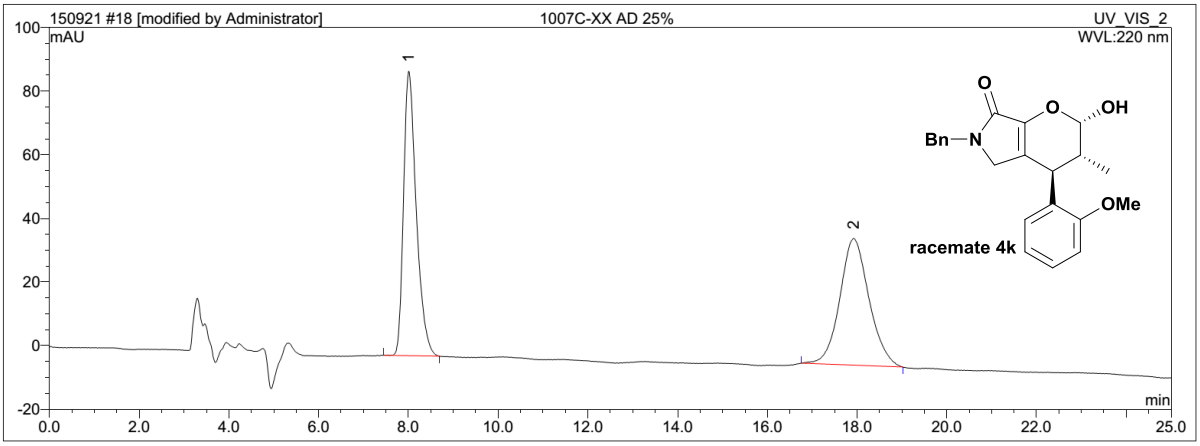

O
O
OH
Bn—N
OMe

4k

190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

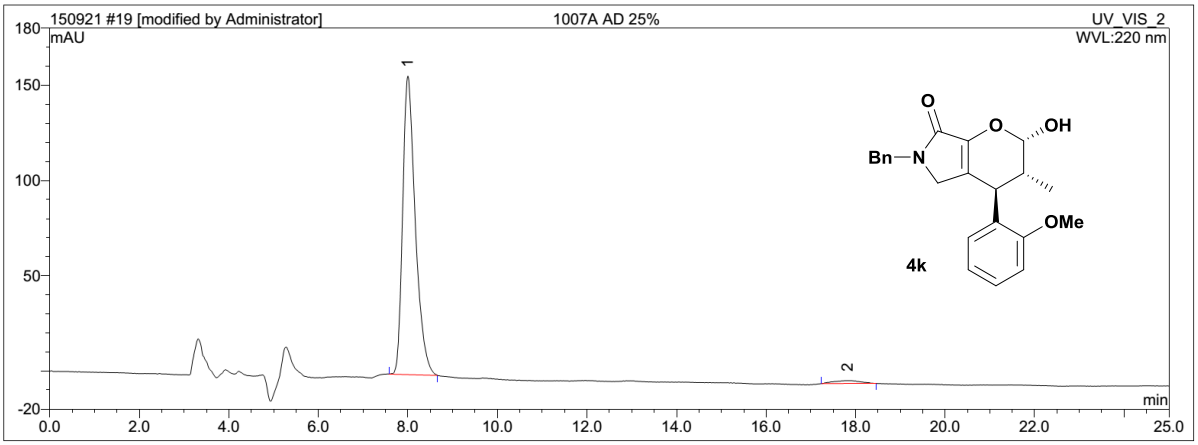
Peak Analysis Report

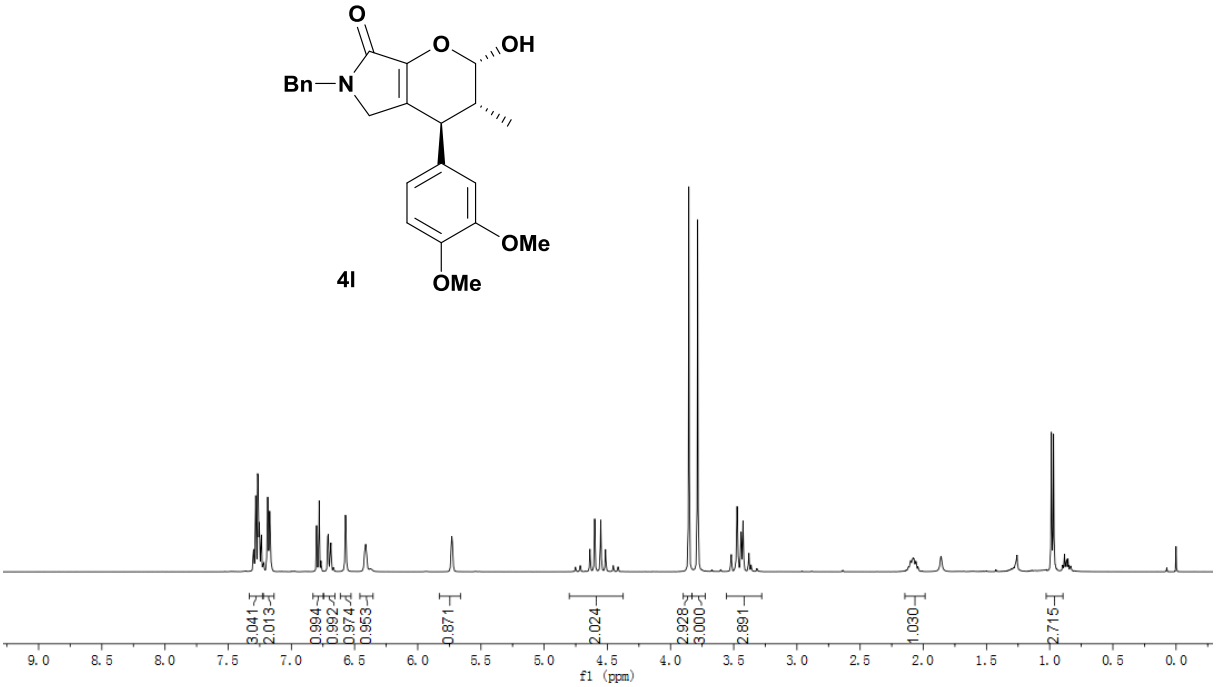
No.	Peak Name	Ret.Time (detected)	Area	Rel.Area	Height	Amount
		min	mAU*min	%	mAU	
1	n.a.	8.01	29.332	49.45	89.465	n.a.
2	n.a.	17.93	29.986	50.55	39.869	n.a.



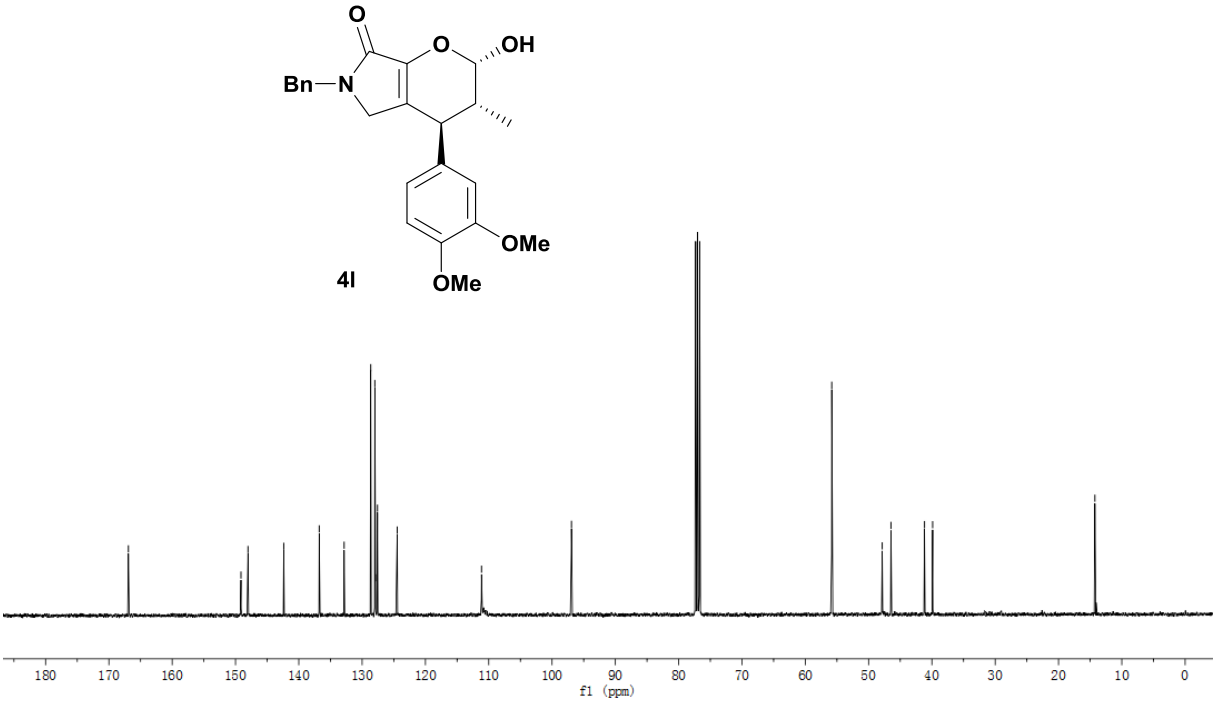
Peak Analysis Report

No.	Peak Name	Ret.Time (detected)	Area	Rel.Area	Height	Amount
		min	mAU*min	%	mAU	
1	n.a.	8.00	51.262	98.08	156.814	n.a.
2	n.a.	17.83	1.002	1.92	1.467	n.a.



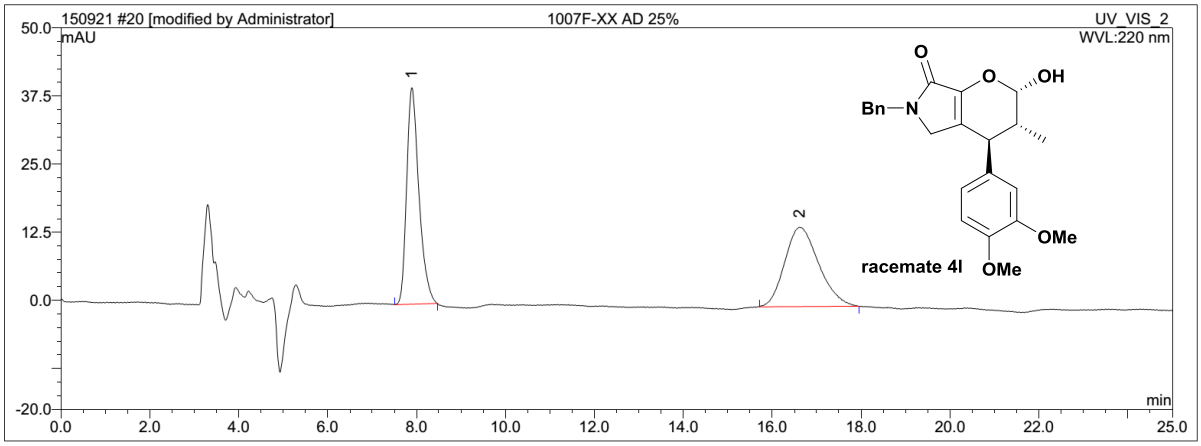

O
O
OH
Bn—N
OMe
OMe
4l
3.041
2.013
0.994
0.992
0.974
0.953
0.871
2.024
2.928
3.000
2.891
1.030
2.715
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


166.921
149.138
148.022
142.378
136.772
132.844
128.668
128.640
127.969
127.892
127.573
124.491
111.139
96.932
55.818
47.851
46.462
41.167
39.881
14.254


O
O
OH
Bn—N
OMe
OMe
4l
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

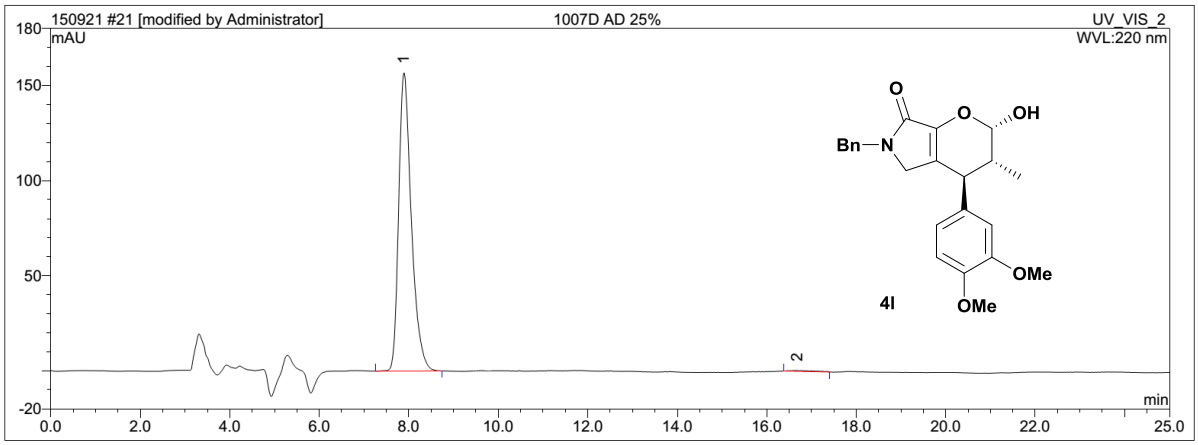
Peak Analysis Report

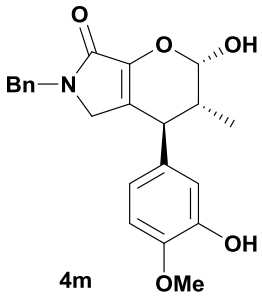
No.	Peak Name	Ret.Time (detected)	Area	Rel.Area	Height	Amount
		min	mAU*min	%	mAU	
1	n.a.	7.89	12.796	49.79	39.780	n.a.
2	n.a.	16.62	12.905	50.21	14.569	n.a.

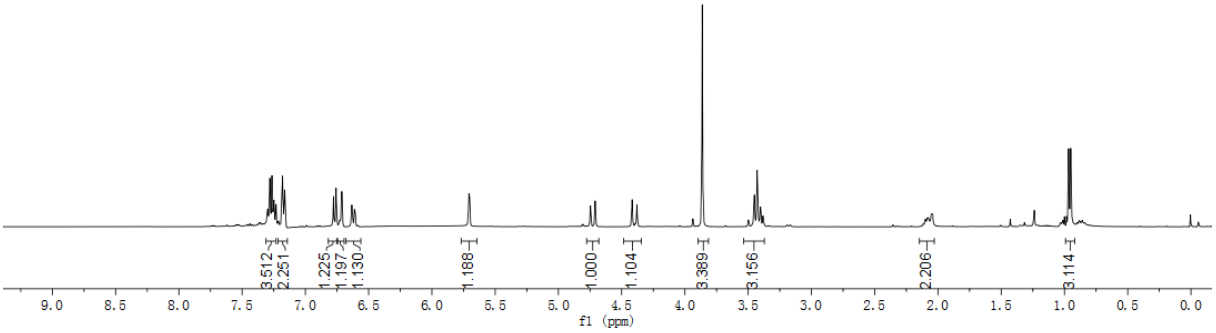


Peak Analysis Report

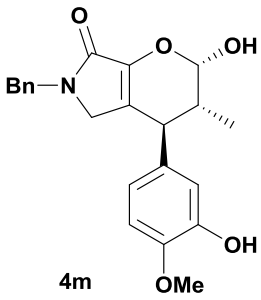
No.	Peak Name	Ret.Time (detected)	Area	Rel.Area	Height	Amount
		min	mAU*min	%	mAU	
1	n.a.	7.89	51.472	99.55	156.894	n.a.
2	n.a.	16.68	0.232	0.45	0.411	n.a.

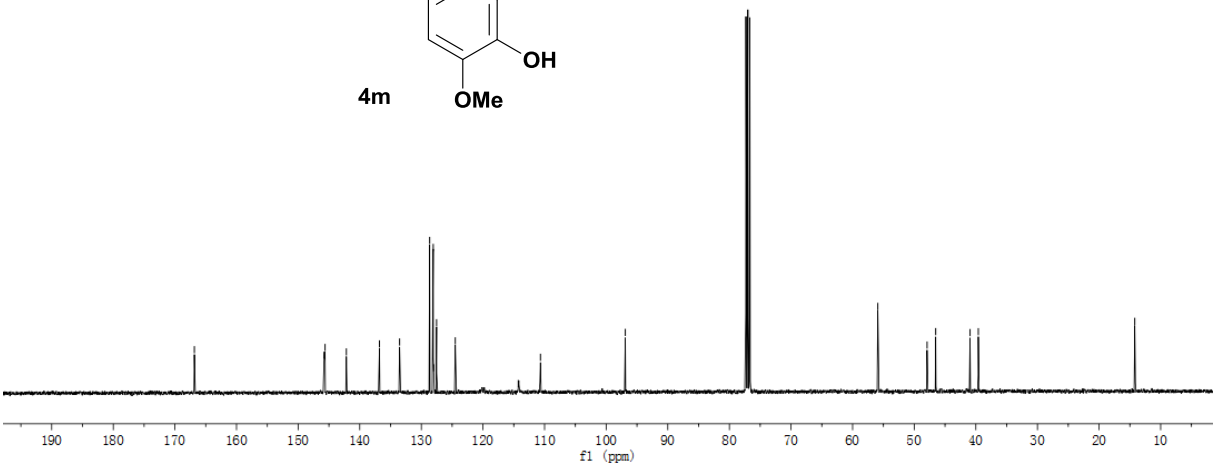



O
O
OH
Bn—N
OH
4m
OMe


3.512
2.251
1.225
1.197
1.130
1.188
1.000
1.104
3.389
3.156
2.206
3.114
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

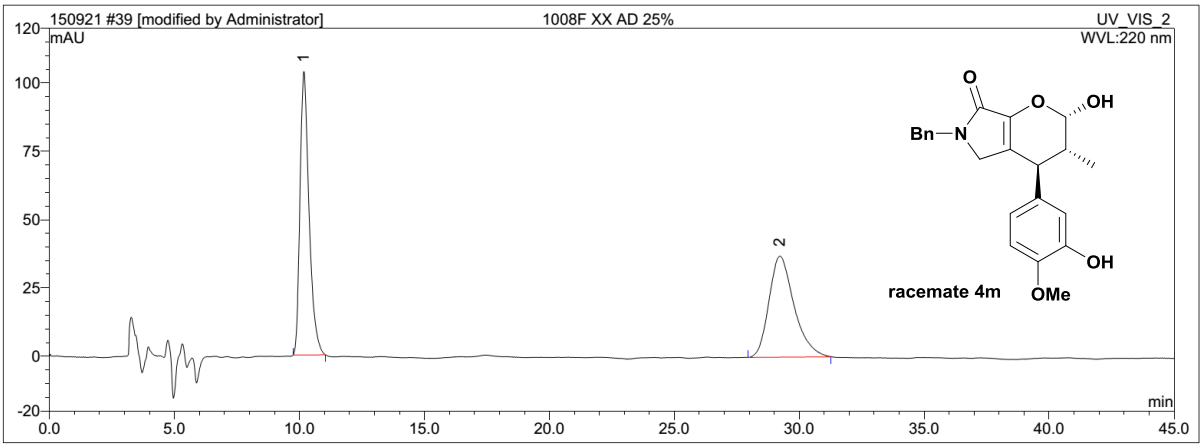

166.842
145.799
145.631
142.172
136.799
133.532
128.650
128.072
127.986
127.528
124.494
114.267
110.666
96.897
55.917
47.915
46.517
40.940
39.596
14.208


O
O
OH
Bn—N
OH
4m
OMe


190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

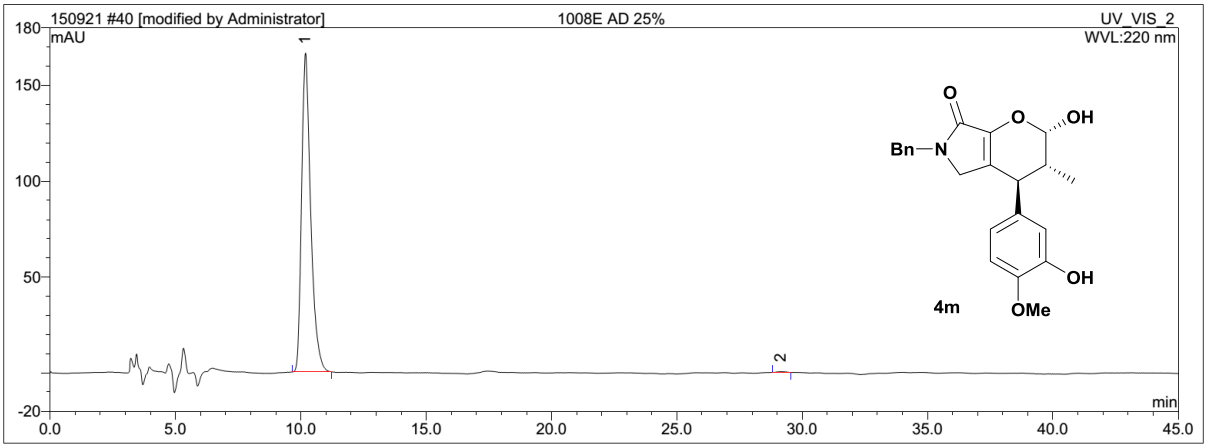
Peak Analysis Report

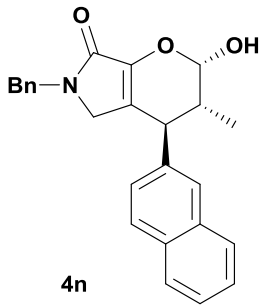
No.	Peak Name	Ret.Time (detected)	Area	Rel.Area	Height	Amount
		min	mAU*min	%	mAU	
1	n.a.	10.17	43.631	50.37	103.764	n.a.
2	n.a.	29.23	42.997	49.63	37.062	n.a.



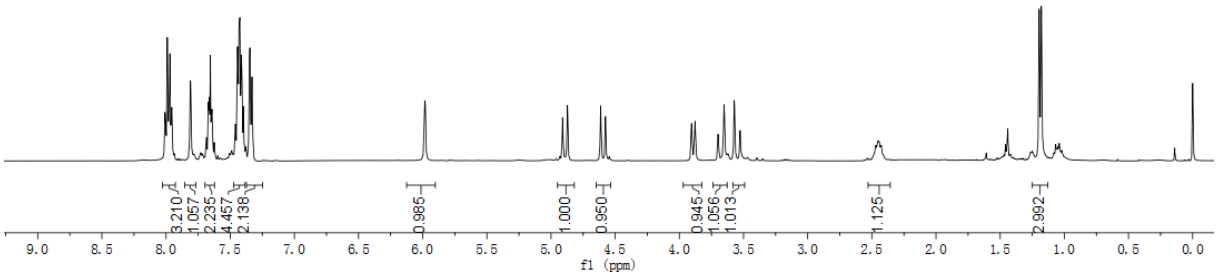
Peak Analysis Report

No.	Peak Name	Ret.Time (detected)	Area	Rel.Area	Height	Amount
		min	mAU*min	%	mAU	
1	n.a.	10.18	70.343	99.80	166.457	n.a.
2	n.a.	29.14	0.140	0.20	0.400	n.a.




O
O
OH
Bn—N

4n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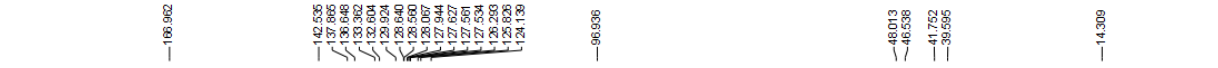
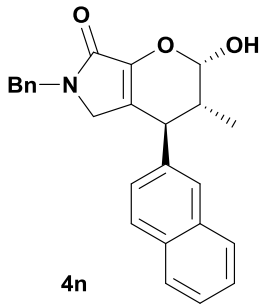
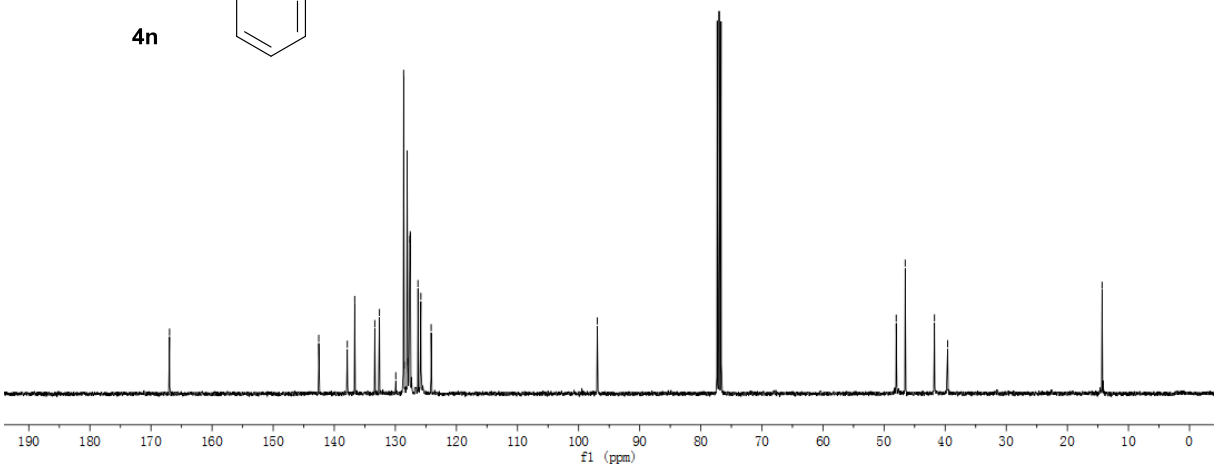
1.057
2.235
4.457
2.138
0.985
1.000
0.950
0.945
1.056
1.013
1.125
2.992
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


166.962
142.536
137.865
136.648
133.362
132.604
129.924
128.640
128.560
128.067
127.944
127.627
127.561
127.534
126.293
125.826
124.139
96.936
48.013
46.538
41.752
39.595
14.309

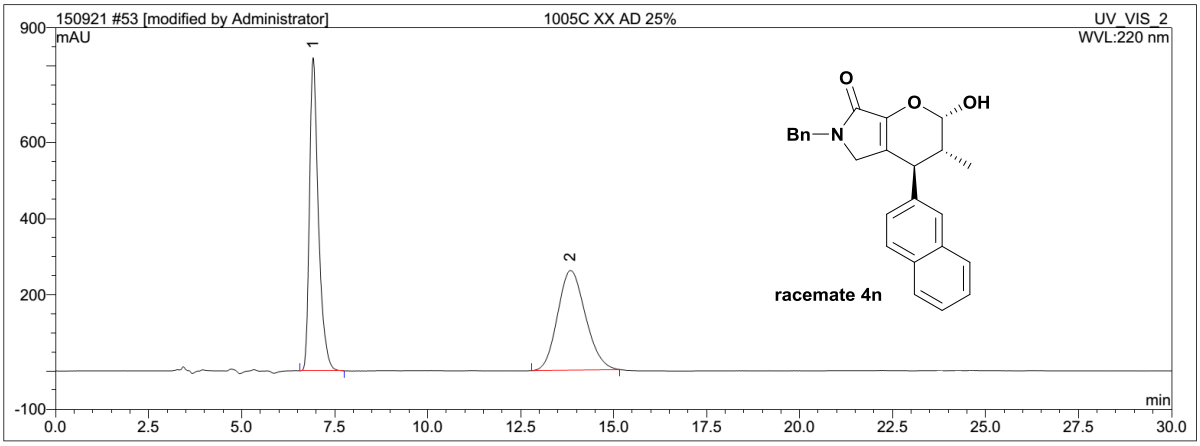

O
O
OH
Bn—N

4n


190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

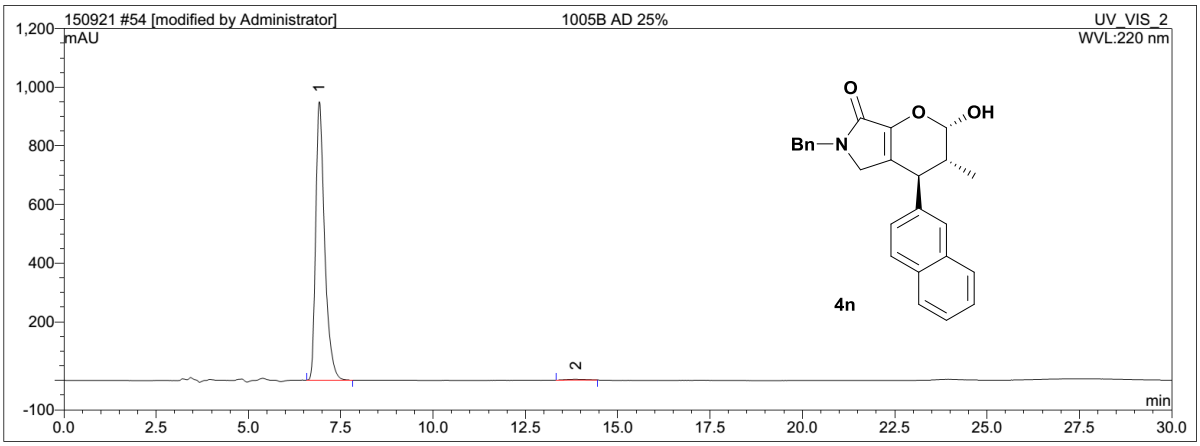
Peak Analysis Report

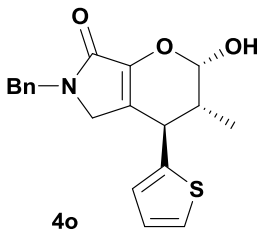
No.	Peak Name	Ret.Time (detected)	Area	Rel.Area	Height	Amount
		min	mAU*min	%	mAU	
1	n.a.	6.93	229.390	50.20	821.440	n.a.
2	n.a.	13.84	227.523	49.80	261.424	n.a.



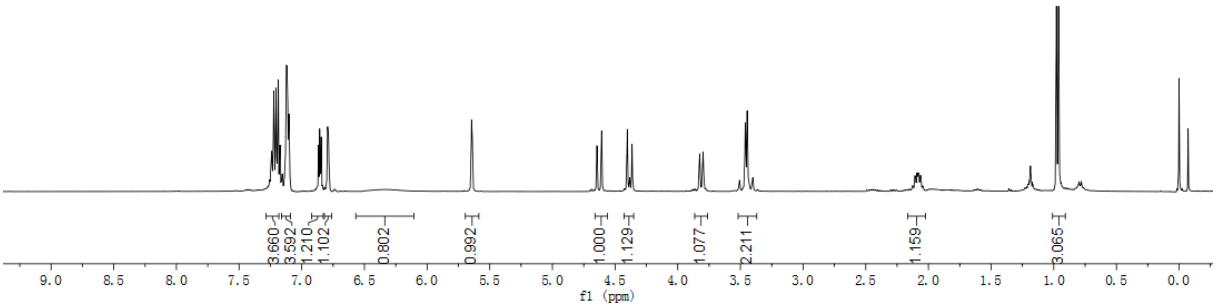
Peak Analysis Report

No.	Peak Name	Ret.Time (detected)	Area	Rel.Area	Height	Amount
		min	mAU*min	%	mAU	
1	n.a.	6.92	266.413	99.50	950.072	n.a.
2	n.a.	13.88	1.328	0.50	2.175	n.a.

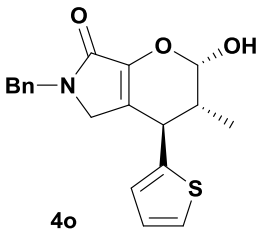



O
O
OH
Bn—N
S

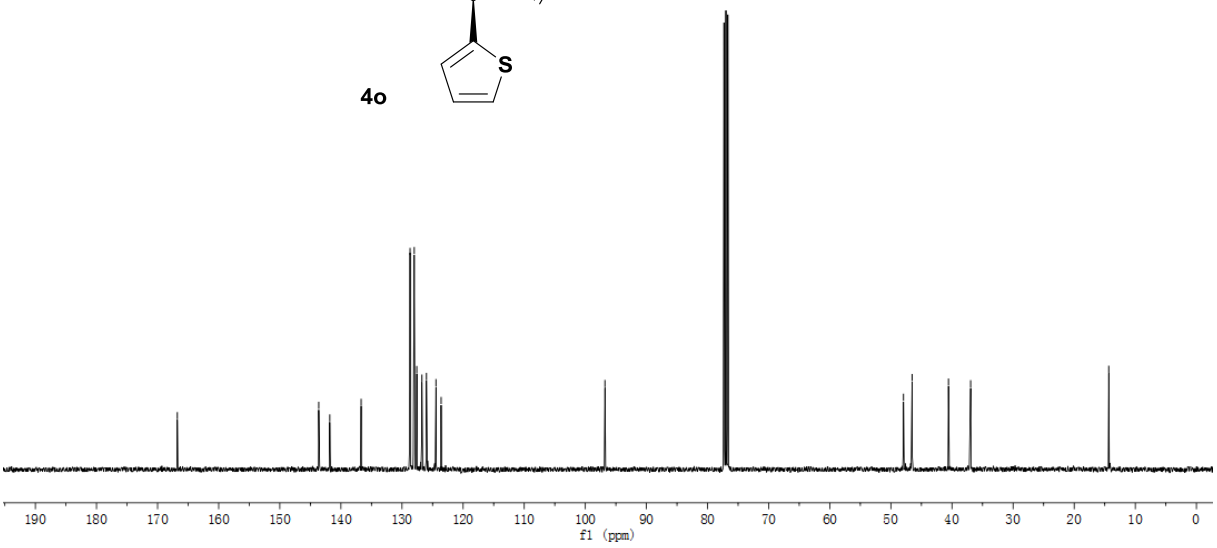
4o


3.660
3.592
1.210
1.102
0.802
0.992
1.000
1.129
1.077
2.211
1.159
3.065
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


166.775
143.625
141.822
136.691
128.682
128.000
127.583
126.746
126.005
124.441
123.599
96.790
47.937
46.523
40.572
36.945
14.340

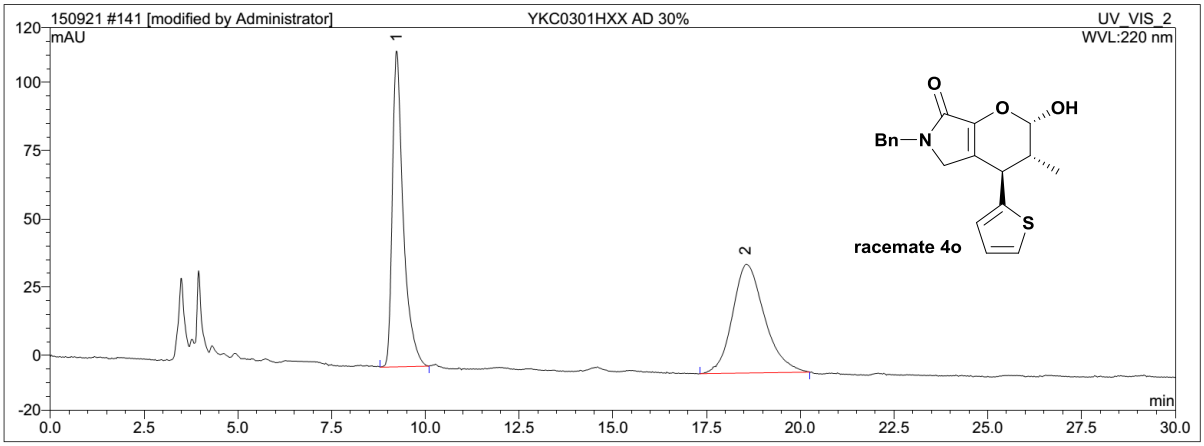

O
O
OH
Bn—N
S

4o


190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

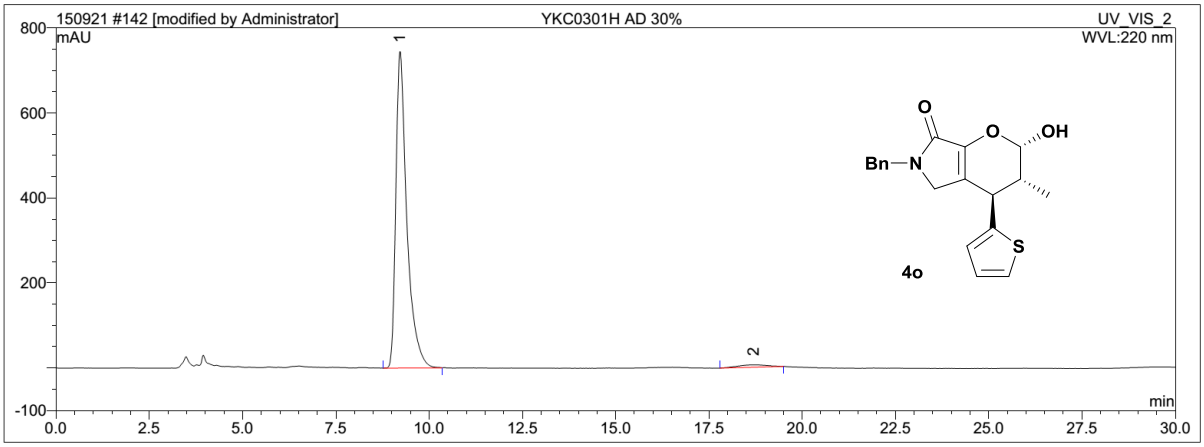
Peak Analysis Report

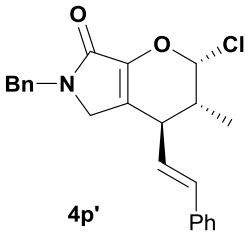
No.	Peak Name	Ret.Time (detected)	Area	Rel.Area	Height	Amount
		min	mAU*min	%	mAU	
1	n.a.	9.23	39.271	49.83	115.766	n.a.
2	n.a.	18.54	39.532	50.17	39.788	n.a.



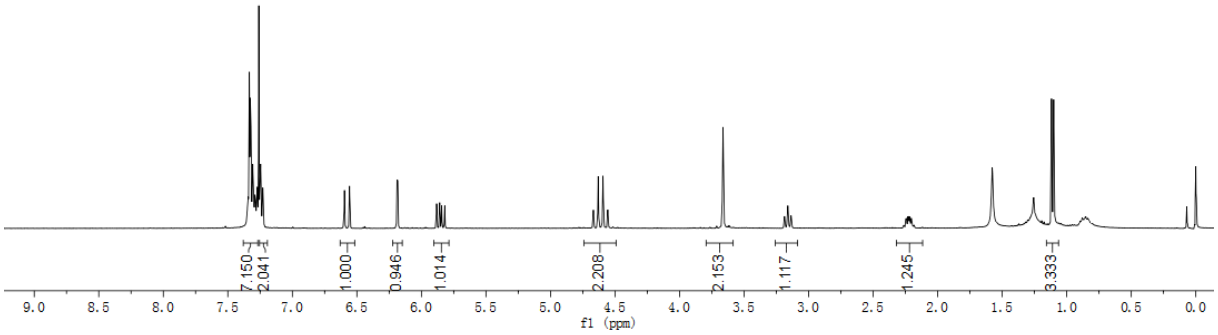
Peak Analysis Report

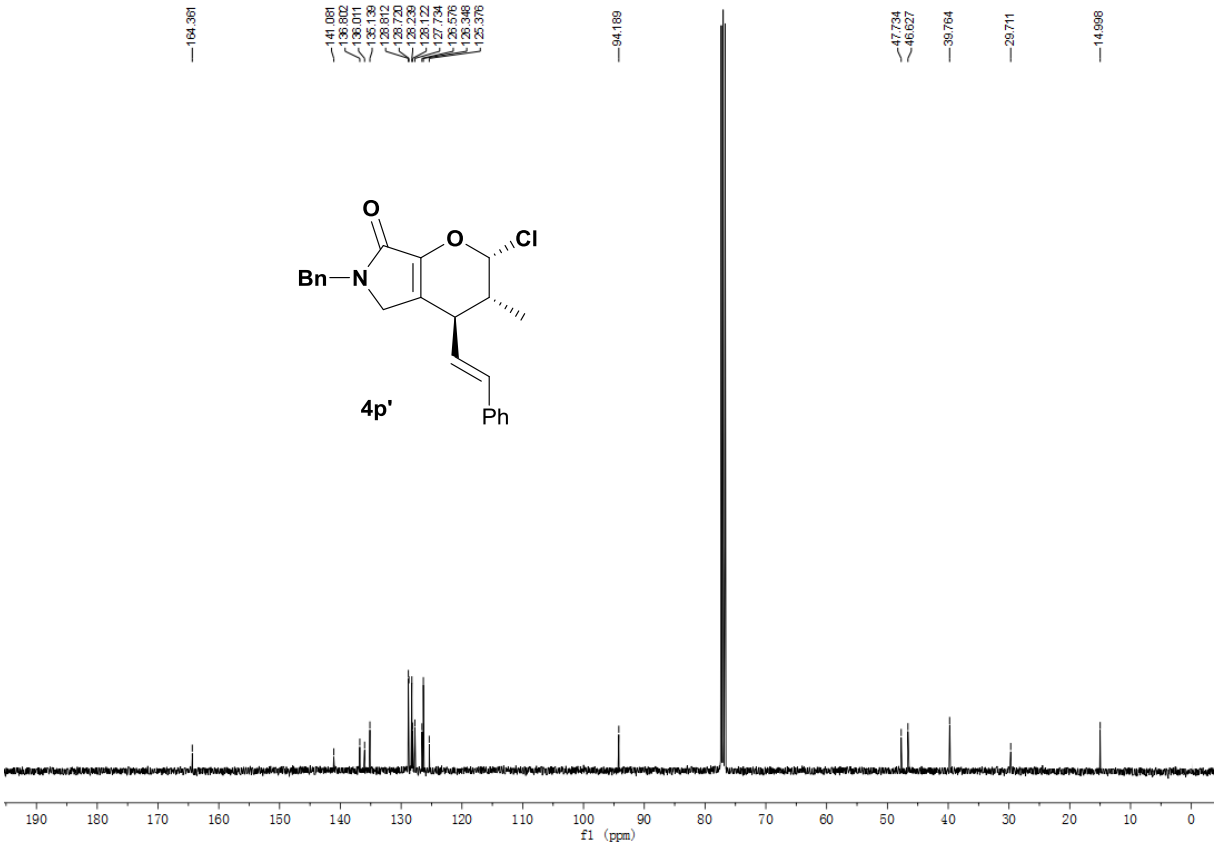
No.	Peak Name	Ret.Time (detected)	Area	Rel.Area	Height	Amount
		min	mAU*min	%	mAU	
1	n.a.	9.22	255.400	98.11	744.620	n.a.
2	n.a.	18.70	4.922	1.89	5.471	n.a.




O
O
Cl
Bn—N
Ph

4p'

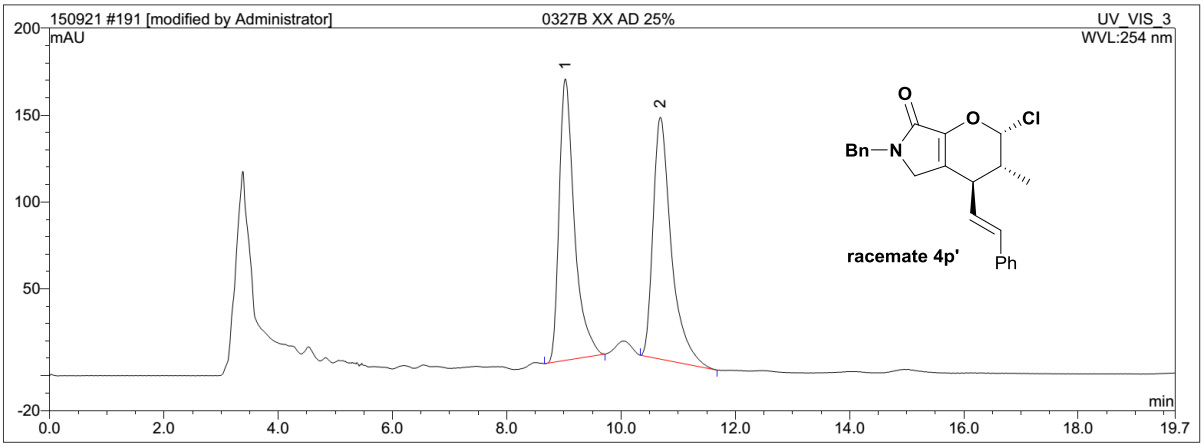

7.150
2.041
1.000
0.946
1.014
2.208
2.153
1.117
1.245
3.333
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


164.361
141.081
136.802
136.011
135.139
128.812
128.720
128.239
128.122
127.734
126.576
126.348
125.376
94.189
47.734
46.627
39.764
29.711
14.996
O
O
Cl
Bn—N
Ph
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

4p'

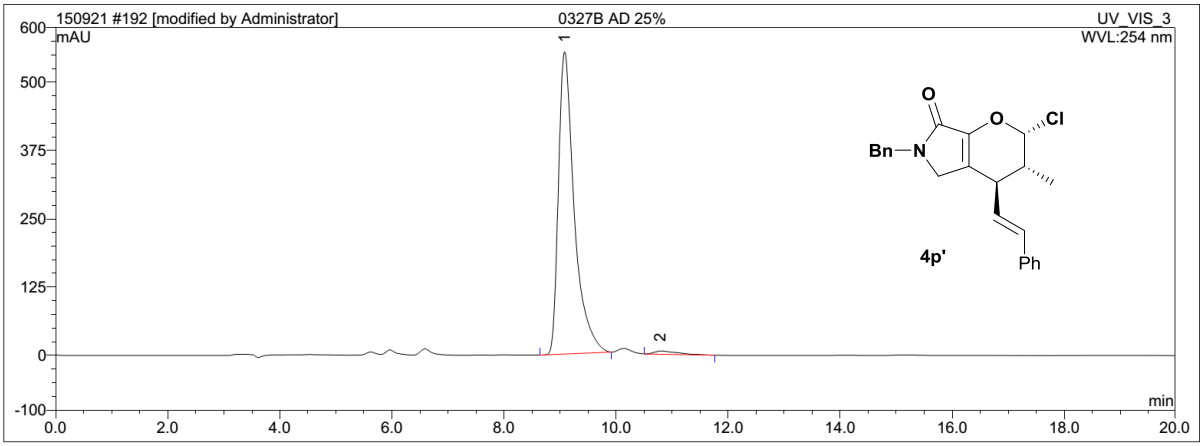
Peak Analysis Report

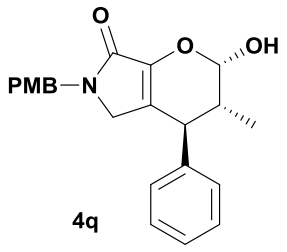
No.	Peak Name	Ret.Time (detected)	Area	Rel.Area	Height	Amount
		min	mAU*min	%	mAU	
1	n.a.	9.03	50.282	49.44	162.113	n.a.
2	n.a.	10.68	51.425	50.56	139.236	n.a.



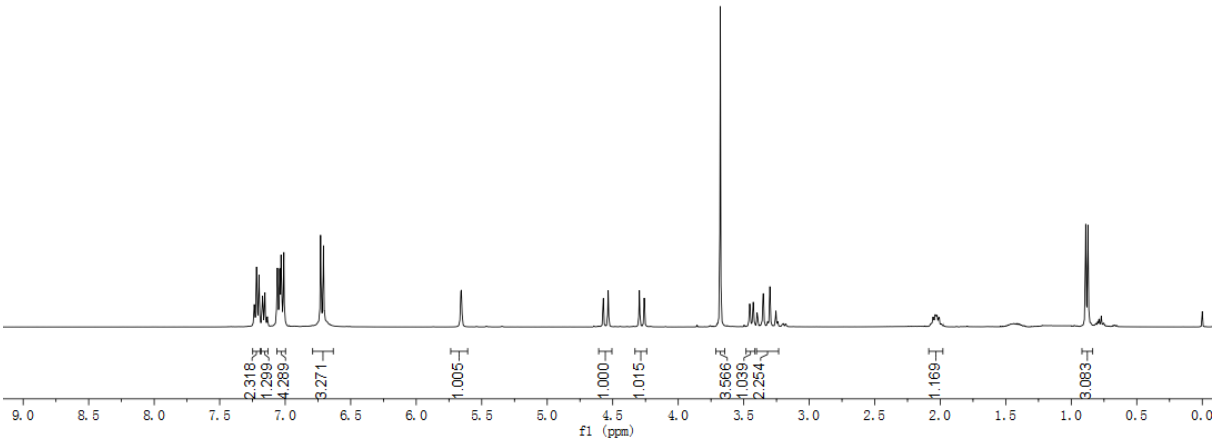
Peak Analysis Report

No.	Peak Name	Ret.Time (detected)	Area	Rel.Area	Height	Amount
		min	mAU*min	%	mAU	
1	n.a.	9.08	180.875	98.44	553.349	n.a.
2	n.a.	10.79	2.874	1.56	5.842	n.a.




O
O
OH
PMB—N

4q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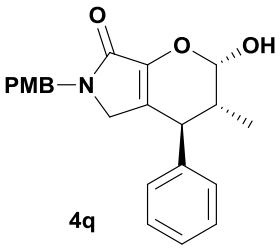
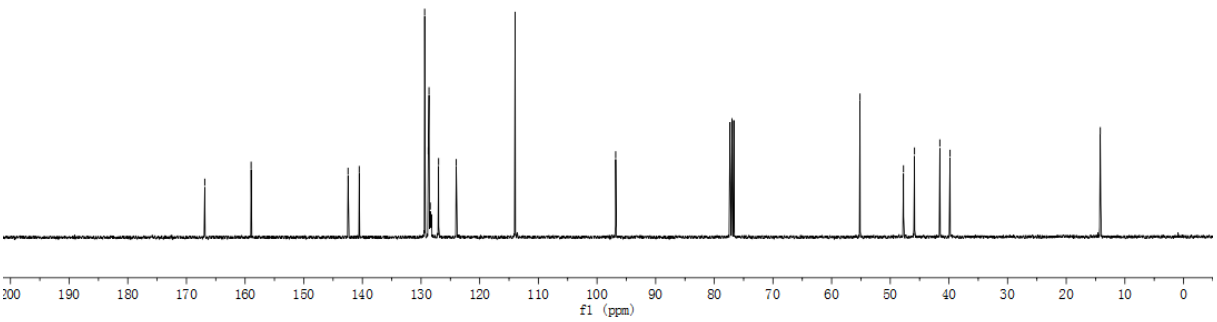
1.299
4.289
3.271
1.005
1.000
1.015
3.566
1.039
2.254
1.169
3.083
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


166.872
158.970
142.446
140.538
129.381
128.789
128.645
128.427
127.014
124.005
113.959
96.815
55.167
47.751
45.871
41.544
39.812
14.196

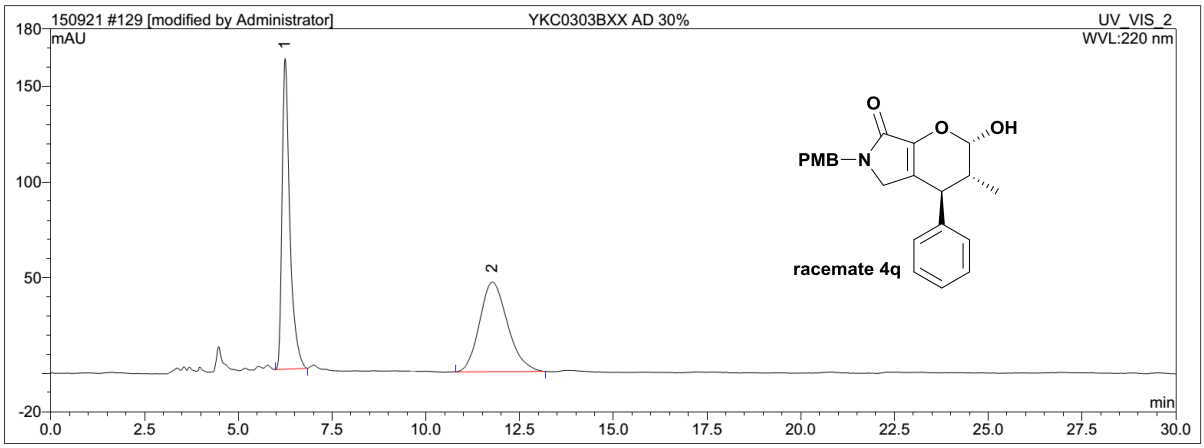

O
O
OH
PMB—N

4q


200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

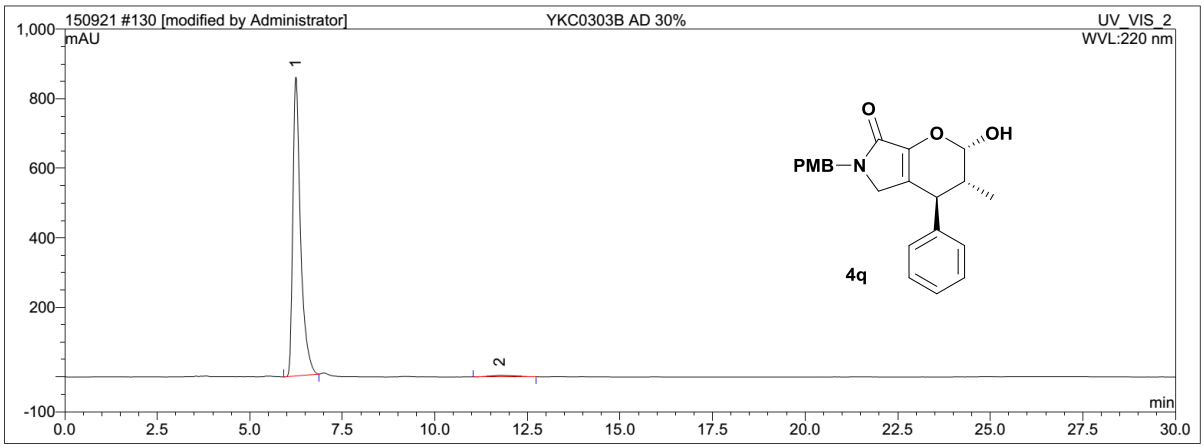
Peak Analysis Report

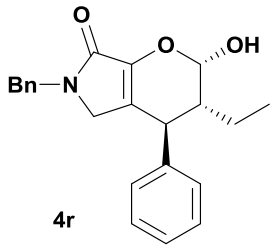
No.	Peak Name	Ret.Time (detected)	Area	Rel.Area	Height	Amount
		min	mAU*min	%	mAU	
1	n.a.	6.24	39.011	49.02	162.274	n.a.
2	n.a.	11.77	40.565	50.98	46.928	n.a.



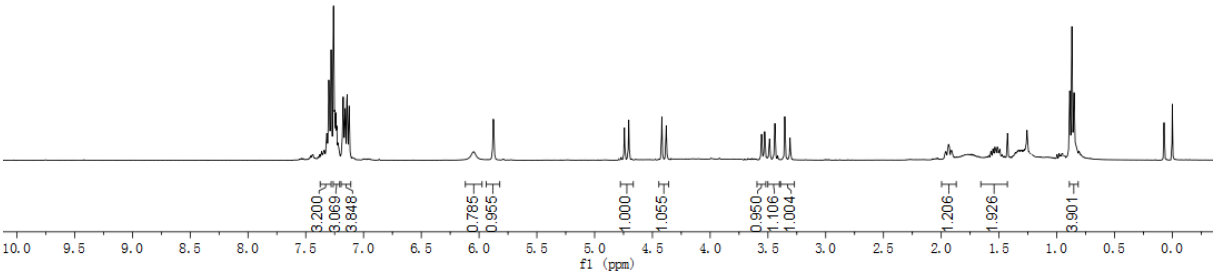
Peak Analysis Report

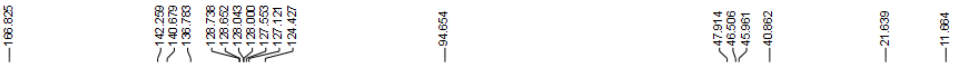
No.	Peak Name	Ret.Time (detected)	Area	Rel.Area	Height	Amount
		min	mAU*min	%	mAU	
1	n.a.	6.24	207.676	98.46	859.816	n.a.
2	n.a.	11.75	3.238	1.54	4.163	n.a.

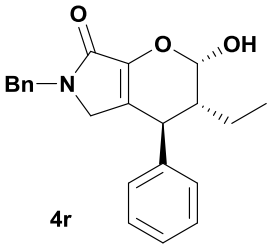



O
O
OH
Bn—N

4r


3.200
3.069
3.848
0.785
0.955
1.000
1.055
0.950
1.106
1.004
1.206
1.926
3.901
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


166.825
142.259
140.679
136.783
128.738
128.652
128.043
128.000
127.553
127.121
124.427
94.654
47.914
46.506
45.961
40.862
21.639
11.664

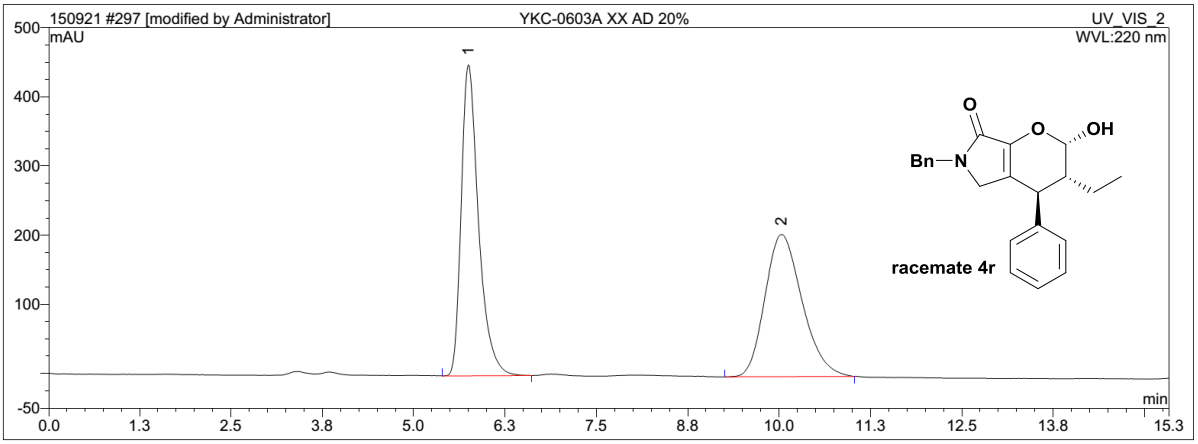

O
O
OH
Bn—N

4r

190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

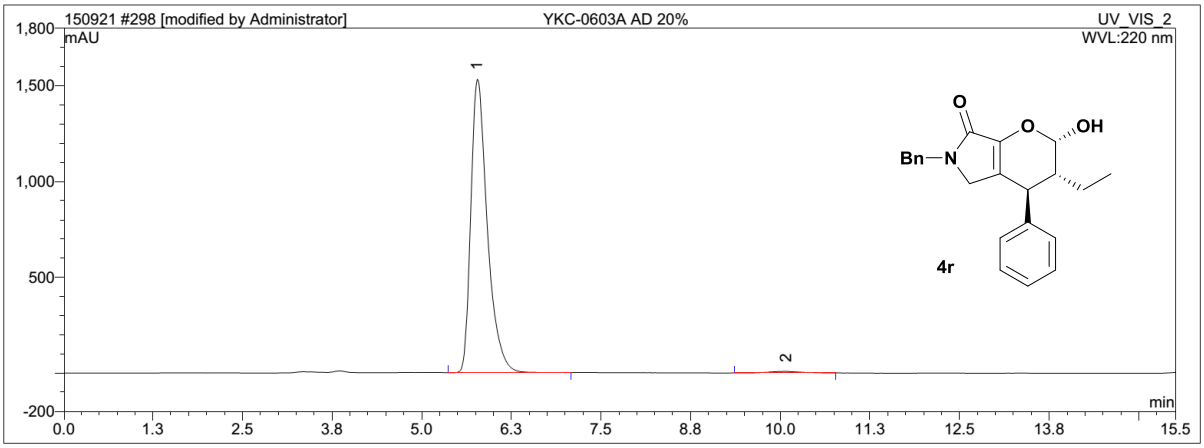
Peak Analysis Report

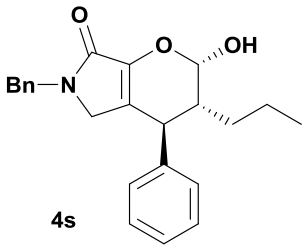
No.	Peak Name	Ret.Time (detected)	Area	Rel.Area	Height	Amount
		min	mAU*min	%	mAU	
1	n.a.	5.75	123.644	50.54	450.007	n.a.
2	n.a.	10.03	120.991	49.46	205.762	n.a.



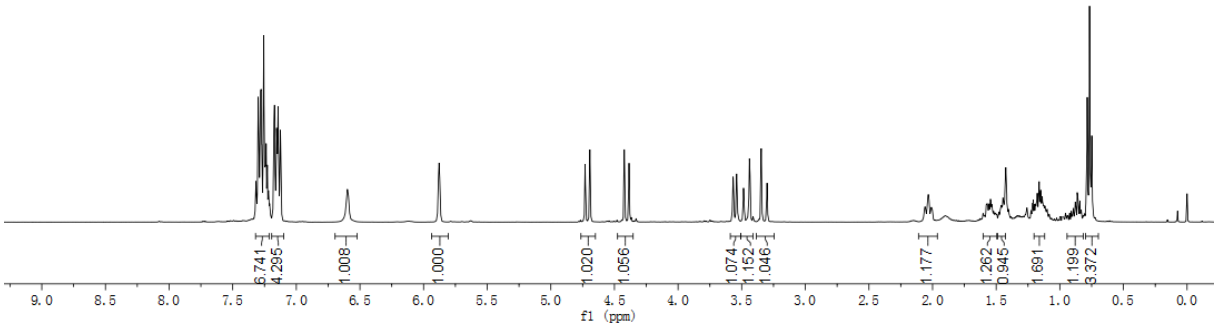
Peak Analysis Report

No.	Peak Name	Ret.Time (detected)	Area	Rel.Area	Height	Amount
		min	mAU*min	%	mAU	
1	n.a.	5.78	408.317	98.99	1532.134	n.a.
2	n.a.	10.08	4.172	1.01	7.760	n.a.

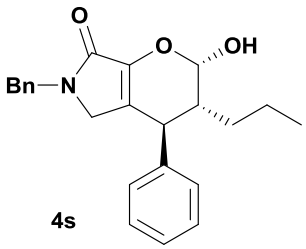



O
O
OH
Bn—N

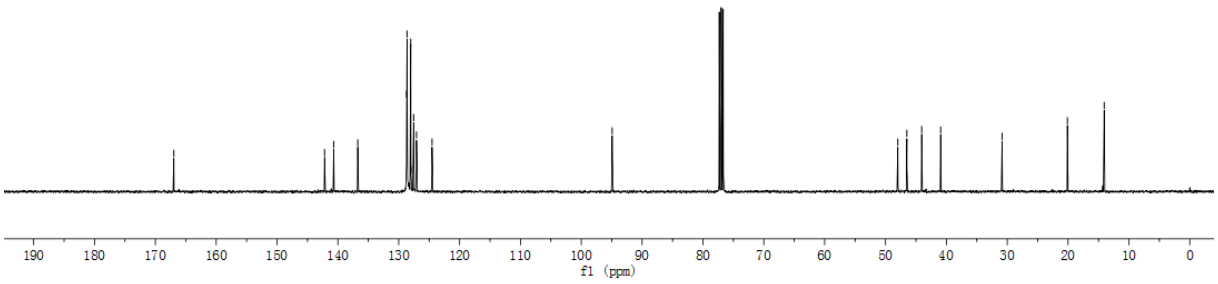
4s


6.741
4.295
1.008
1.000
1.020
1.056
1.074
1.152
1.046
1.177
1.262
0.945
1.691
1.199
3.372
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


166.996
142.185
140.687
136.733
128.715
128.640
128.030
127.969
127.544
127.081
124.526
94.926
47.982
46.499
44.056
40.941
30.843
20.109
14.061

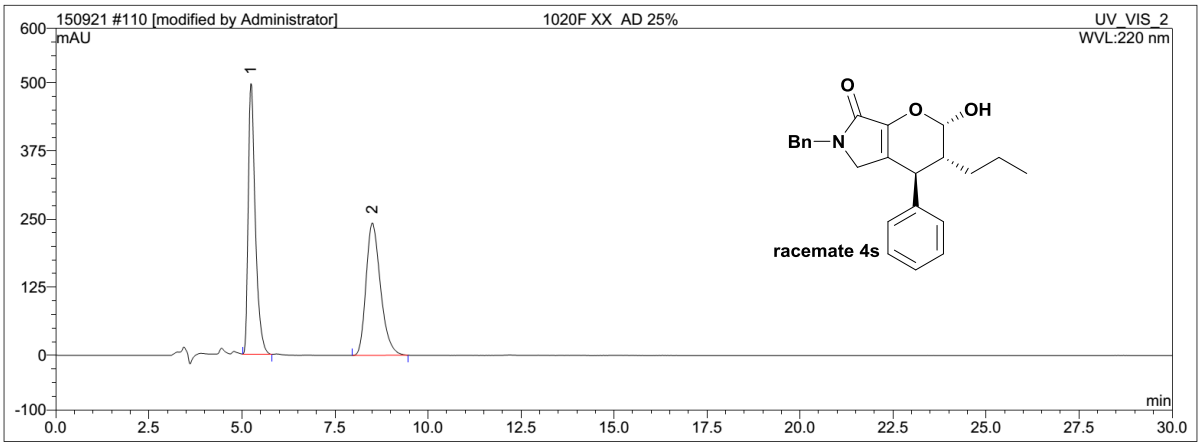

O
O
OH
Bn—N

4s


190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

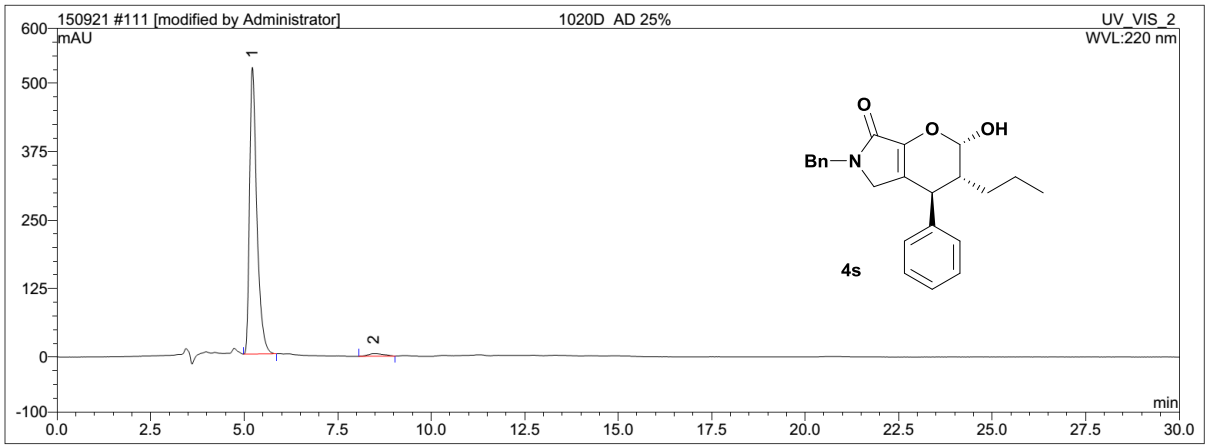
Peak Analysis Report

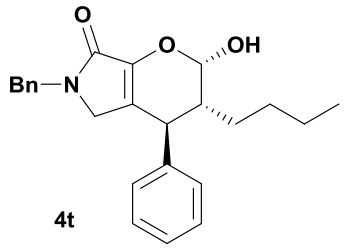
No.	Peak Name	Ret.Time (detected)	Area	Rel.Area	Height	Amount
		min	mAU*min	%	mAU	
1	n.a.	5.24	109.014	49.91	497.026	n.a.
2	n.a.	8.51	109.404	50.09	242.985	n.a.



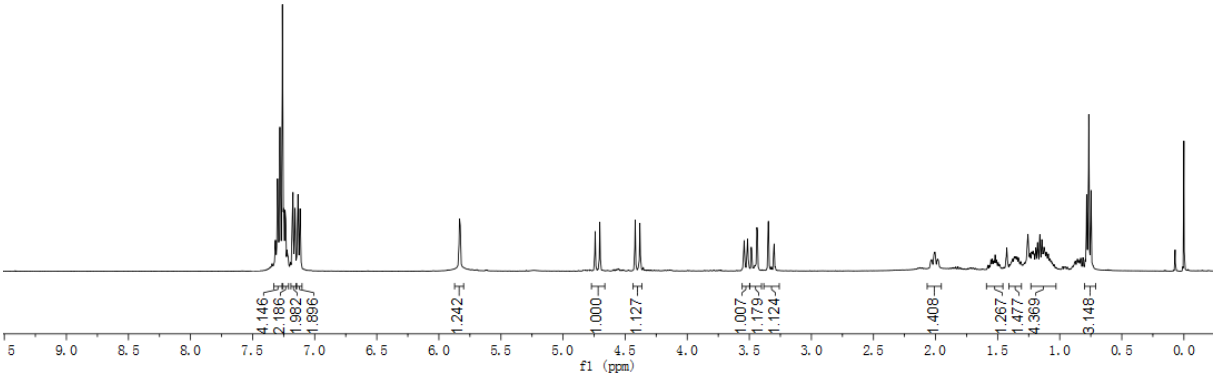
Peak Analysis Report

No.	Peak Name	Ret.Time (detected)	Area	Rel.Area	Height	Amount
		min	mAU*min	%	mAU	
1	n.a.	5.23	117.676	98.09	524.121	n.a.
2	n.a.	8.47	2.292	1.91	4.770	n.a.

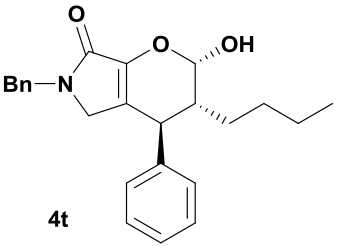



O
O
OH
Bn—N

4t


4.146
2.186
1.982
1.896
1.242
1.000
1.127
1.007
1.179
1.124
1.408
1.267
1.477
4.369
3.148
f1 (ppm)


166.758
142.176
140.619
136.791
130.119
128.737
128.655
128.039
127.555
127.126
124.530
94.938
47.923
46.507
44.176
40.927
29.140
28.306
22.655
13.841

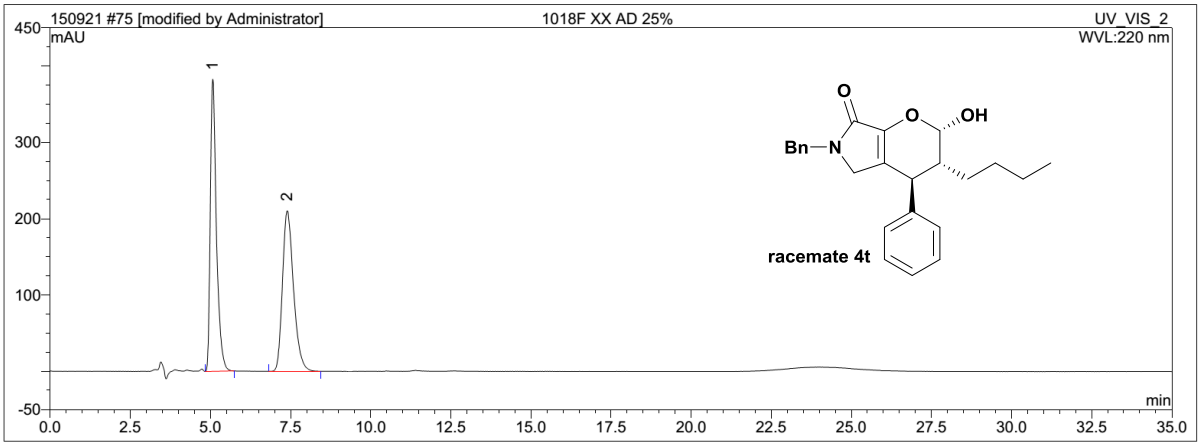

O
O
OH
Bn—N

4t

f1 (ppm)

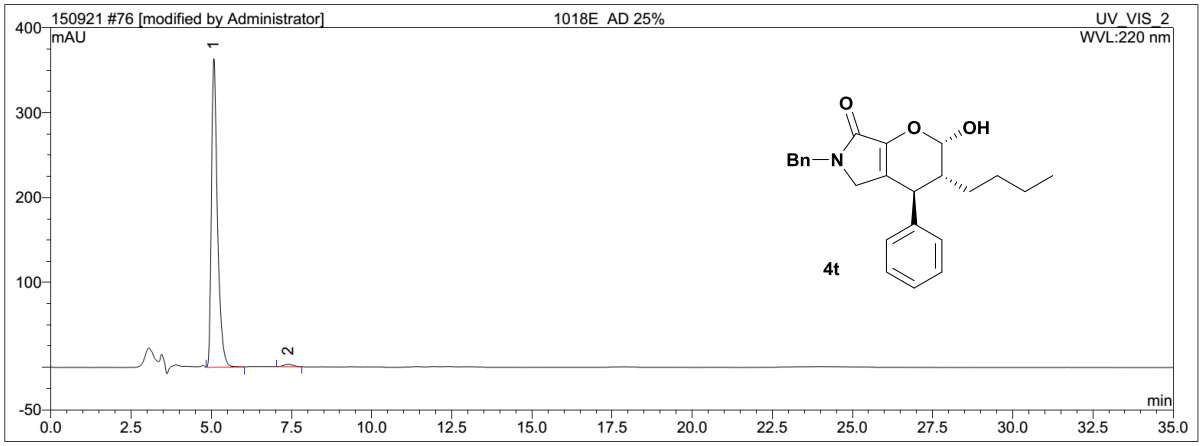
Peak Analysis Report

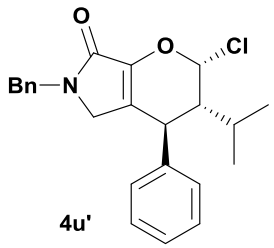
No.	Peak Name	Ret.Time (detected)	Area	Rel.Area	Height	Amount
		min	mAU*min	%	mAU	
1	n.a.	5.07	84.441	50.04	382.909	n.a.
2	n.a.	7.39	84.305	49.96	210.589	n.a.



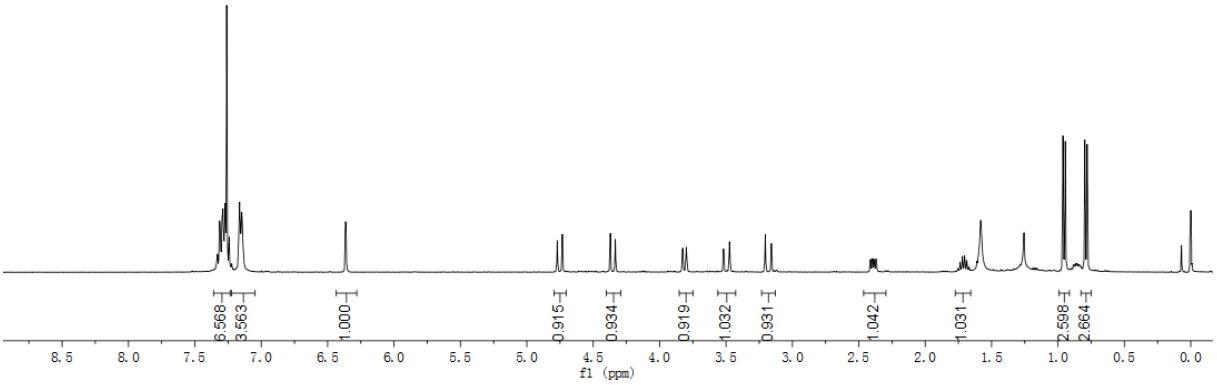
Peak Analysis Report

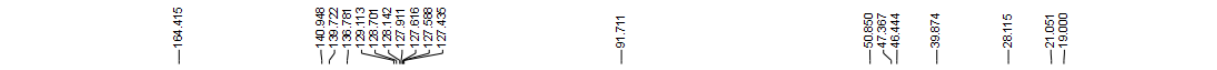
No.	Peak Name	Ret.Time (detected)	Area	Rel.Area	Height	Amount
		min	mAU*min	%	mAU	
1	n.a.	5.08	79.573	98.70	363.694	n.a.
2	n.a.	7.40	1.047	1.30	2.868	n.a.

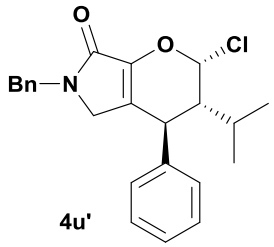



O
O
Cl
Bn—N

4u'


6.568
3.563
1.000
0.915
0.934
0.919
1.032
0.931
1.042
1.031
2.598
2.664
f1 (ppm)


164.415
140.948
138.722
138.176
130.113
128.701
128.142
127.911
127.616
127.588
127.435
91.711
50.650
47.367
46.444
39.874
28.115
21.051
19.000

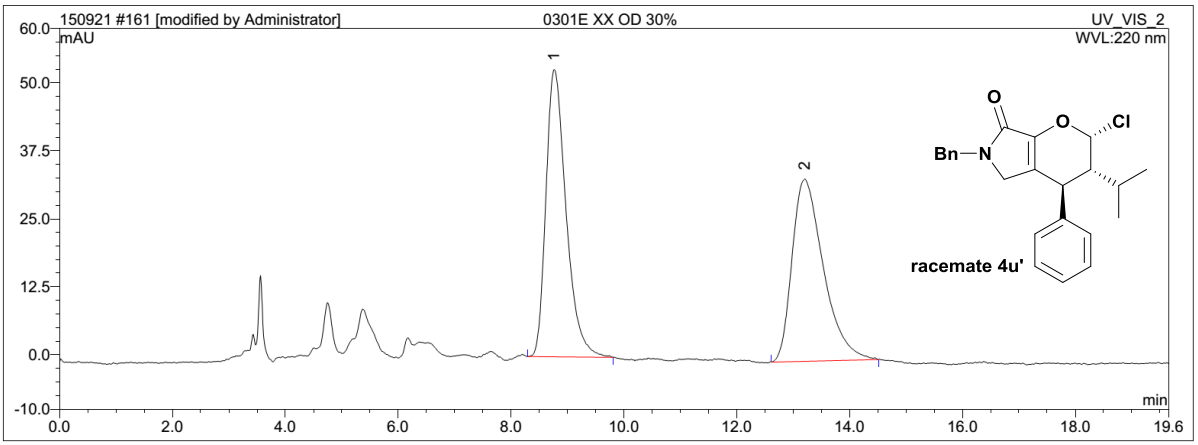

O
O
Cl
Bn—N

4u'

f1 (ppm)

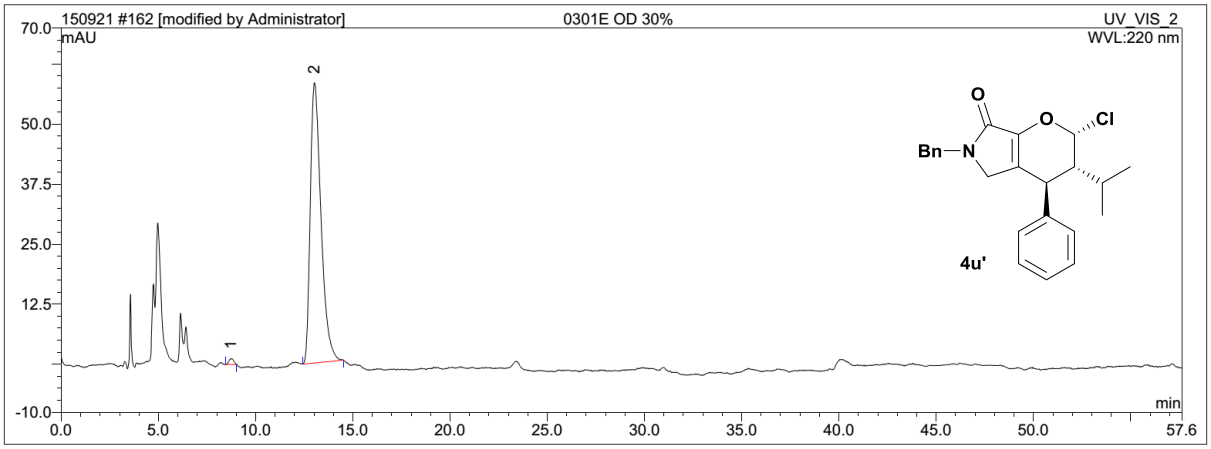
Peak Analysis Report

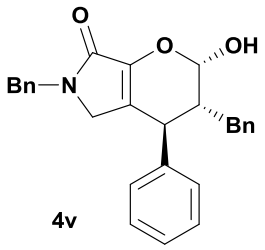
No.	Peak Name	Ret.Time (detected)	Area	Rel.Area	Height	Amount
		min	mAU*min	%	mAU	
1	n.a.	8.77	21.854	50.37	52.847	n.a.
2	n.a.	13.21	21.529	49.63	33.538	n.a.



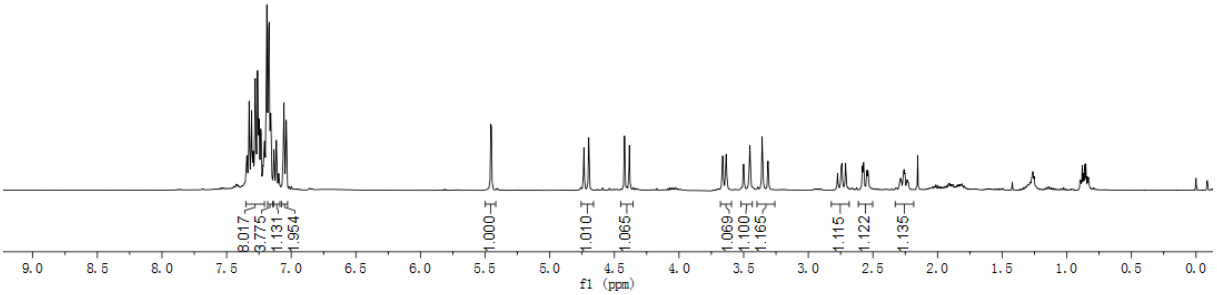
Peak Analysis Report

No.	Peak Name	Ret.Time (detected)	Area	Rel.Area	Height	Amount
		min	mAU*min	%	mAU	
1	n.a.	8.74	0.358	0.96	1.294	n.a.
2	n.a.	13.03	37.169	99.04	58.390	n.a.

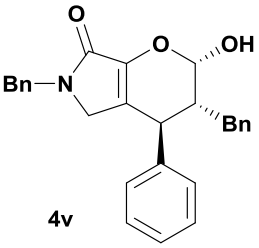



O
O
OH
Bn—N
Bn

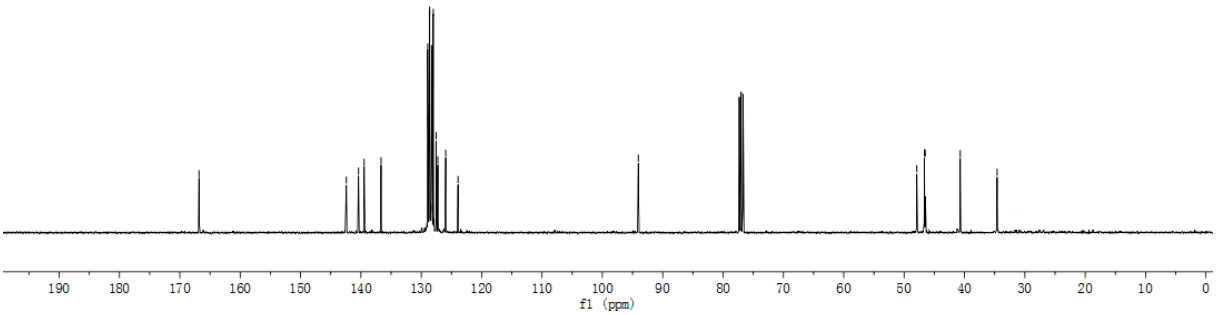
4v


8.017
3.775
1.131
1.954
1.000
1.010
1.065
1.069
1.100
1.165
1.115
1.122
1.135
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


166.822
142.414
140.398
139.488
136.662
128.938
128.854
128.612
128.291
128.158
128.015
127.525
127.255
125.954
123.909
94.036
47.903
46.642
46.501
40.704
34.593

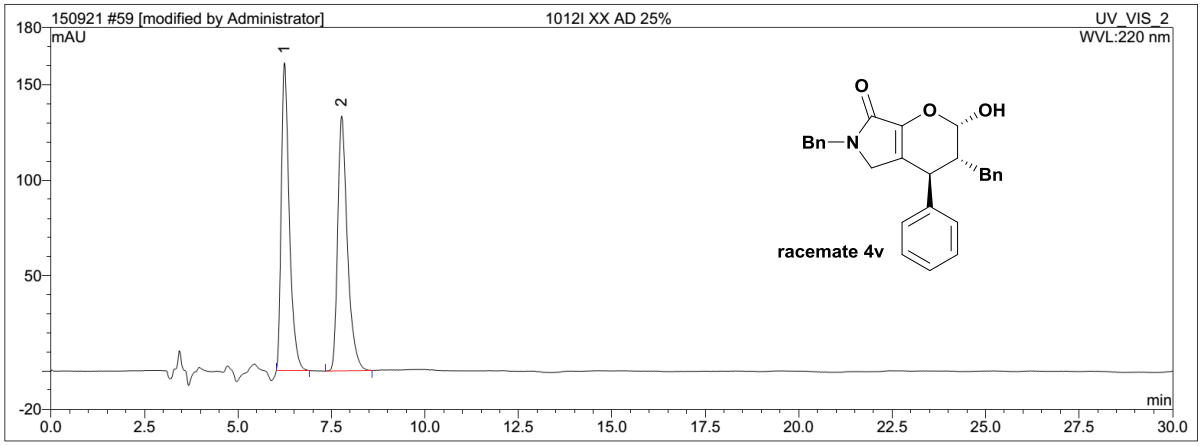

O
O
OH
Bn—N
Bn

4v


190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

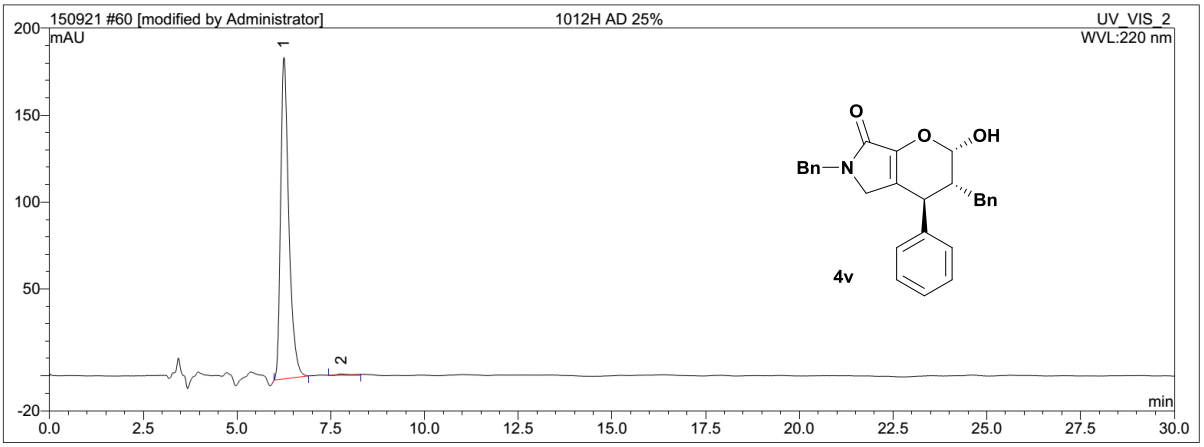
Peak Analysis Report

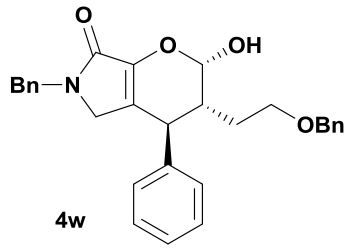
No.	Peak Name	Ret.Time (detected)	Area	Rel.Area	Height	Amount
		min	mAU*min	%	mAU	
1	n.a.	6.24	40.178	49.75	161.247	n.a.
2	n.a.	7.78	40.578	50.25	133.644	n.a.



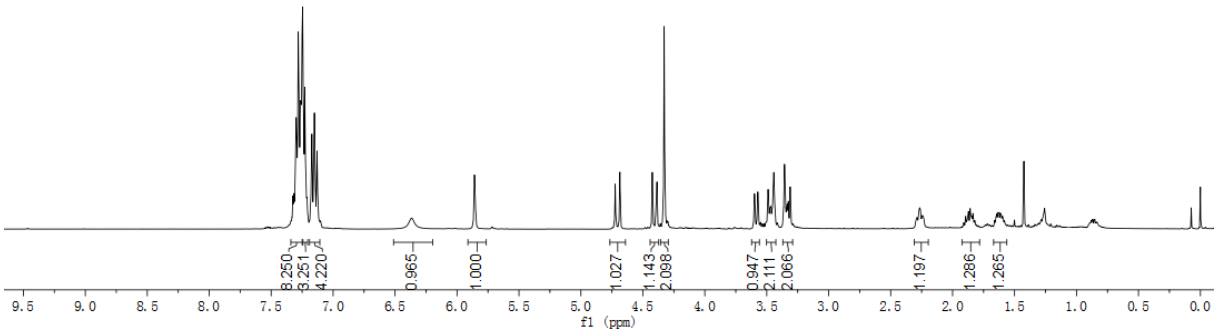
Peak Analysis Report

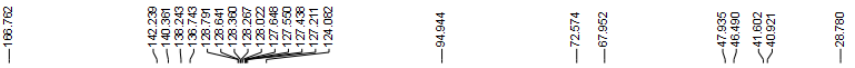
No.	Peak Name	Ret.Time (detected)	Area	Rel.Area	Height	Amount
		min	mAU*min	%	mAU	
1	n.a.	6.25	46.878	99.27	184.965	n.a.
2	n.a.	7.78	0.345	0.73	0.832	n.a.

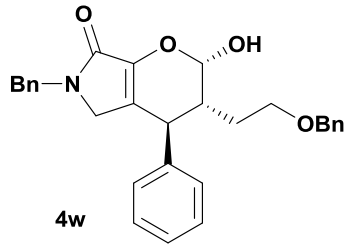



O
O
OH
Bn—N
OBn

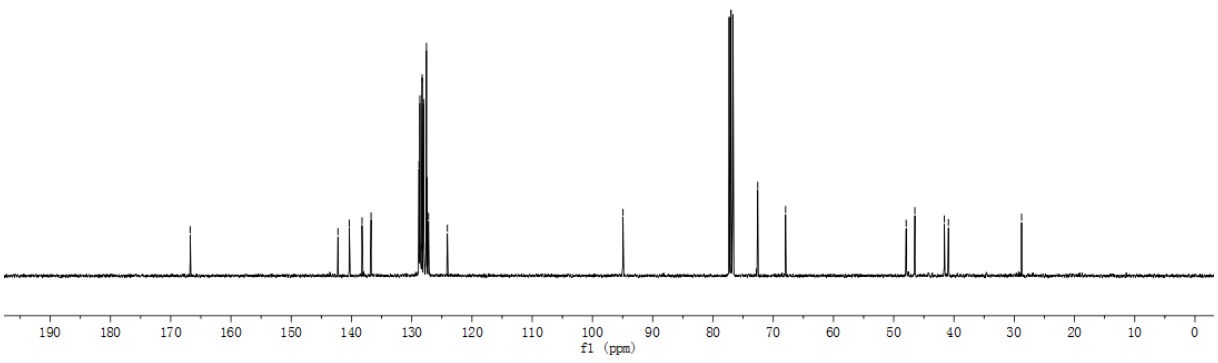
4w


8.250
3.251
4.220
0.965
1.000
1.027
1.143
2.098
0.947
2.111
2.066
1.197
1.286
1.265
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


166.762
142.239
140.361
138.243
136.743
128.791
128.641
128.360
128.267
128.022
127.648
127.550
127.438
127.211
124.062
94.944
72.574
67.952
47.935
46.490
41.602
40.921
28.780

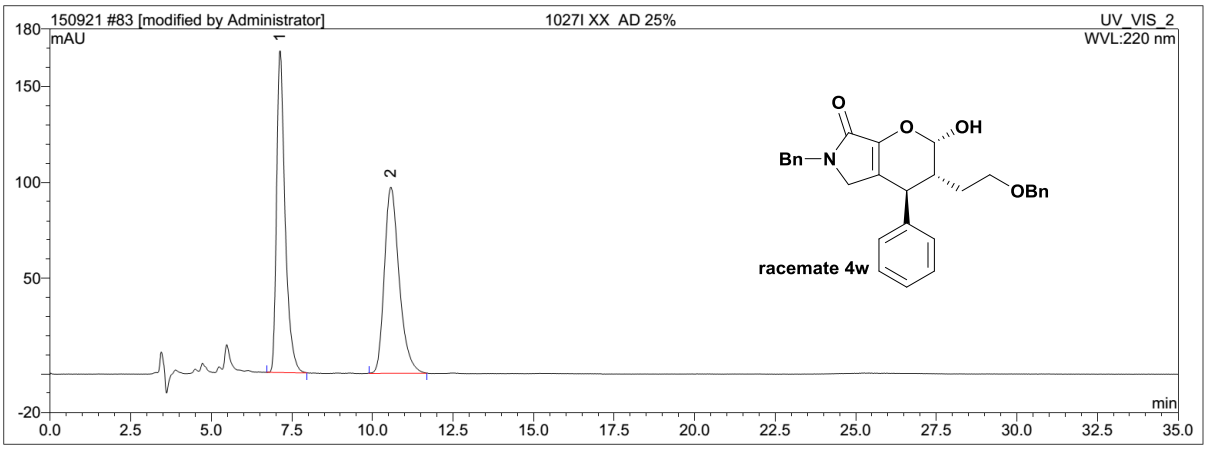

O
O
OH
Bn—N
OBn

4w


190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

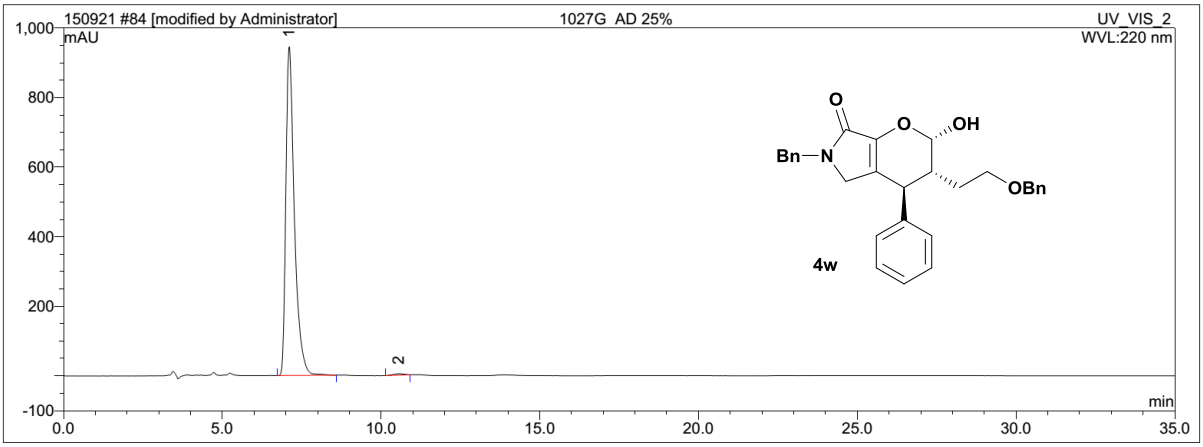
Peak Analysis Report

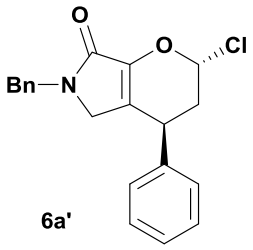
No.	Peak Name	Ret.Time (detected)	Area	Rel.Area	Height	Amount
		min	mAU*min	%	mAU	
1	n.a.	7.13	51.490	49.94	167.911	n.a.
2	n.a.	10.57	51.610	50.06	97.191	n.a.



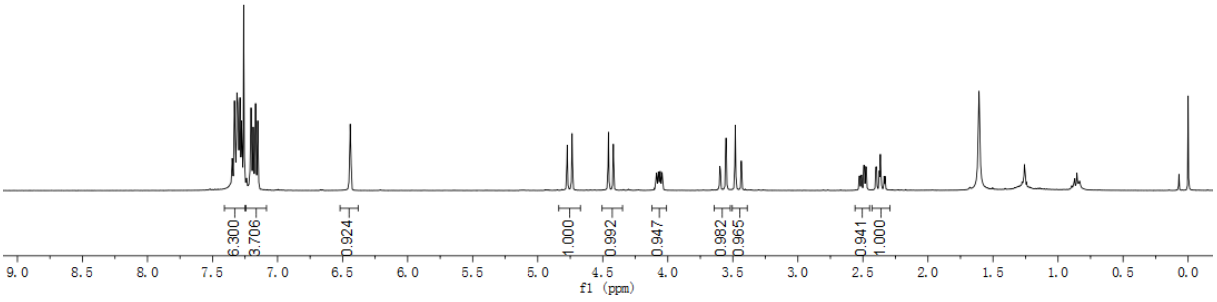
Peak Analysis Report

No.	Peak Name	Ret.Time (detected)	Area	Rel.Area	Height	Amount
		min	mAU*min	%	mAU	
1	n.a.	7.11	294.961	99.44	945.491	n.a.
2	n.a.	10.56	1.673	0.56	4.329	n.a.

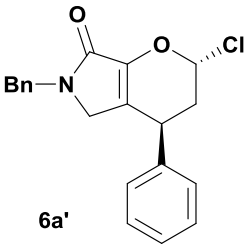



O
O
Cl
Bn—N

6a'

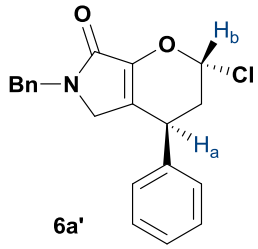

6.300
3.706
0.924
1.000
0.992
0.947
0.982
0.965
0.941
1.000
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


164.309
141.841
139.642
136.706
129.143
128.751
128.045
127.898
127.728
127.677
125.325
88.431
47.441
46.496
39.626
34.770

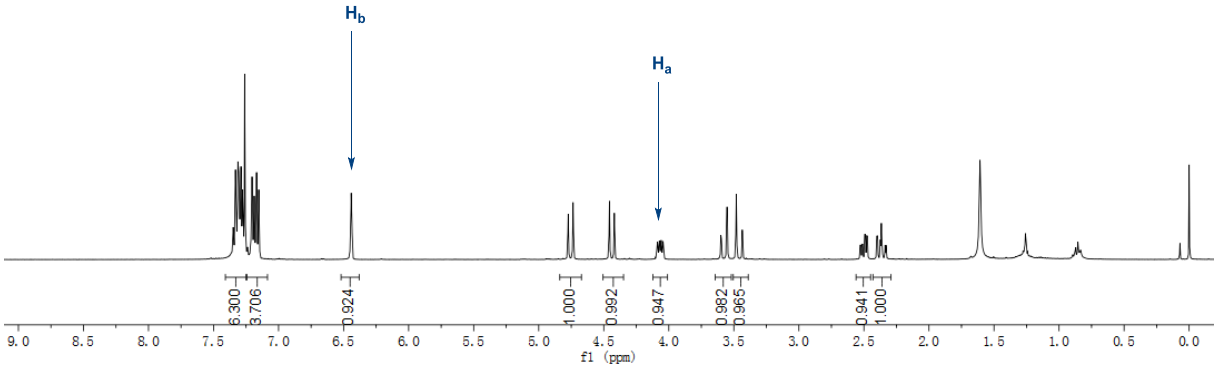

O
O
Cl
Bn—N

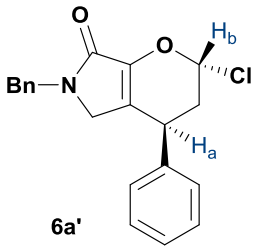
6a'

190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

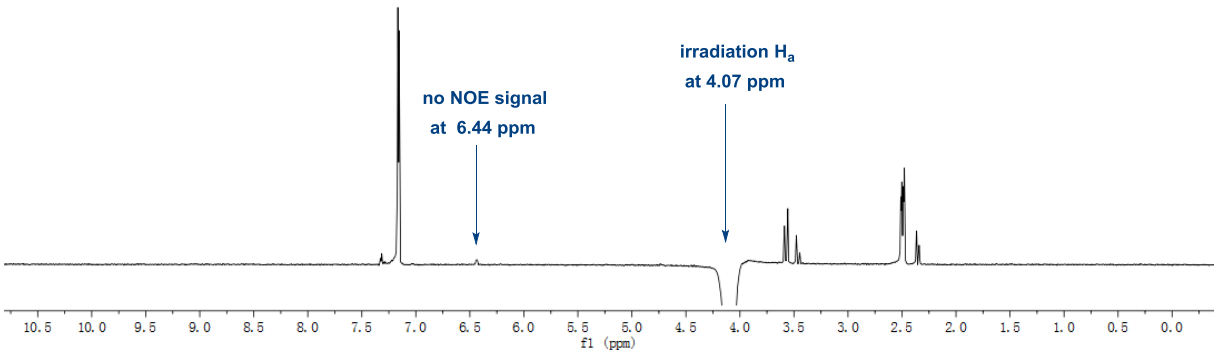

O
O
Hb
Bn—N
Cl
Ha

6a'


Hb
Ha
6.300
3.706
0.924
1.000
0.992
0.947
0.982
0.965
0.941
1.000
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

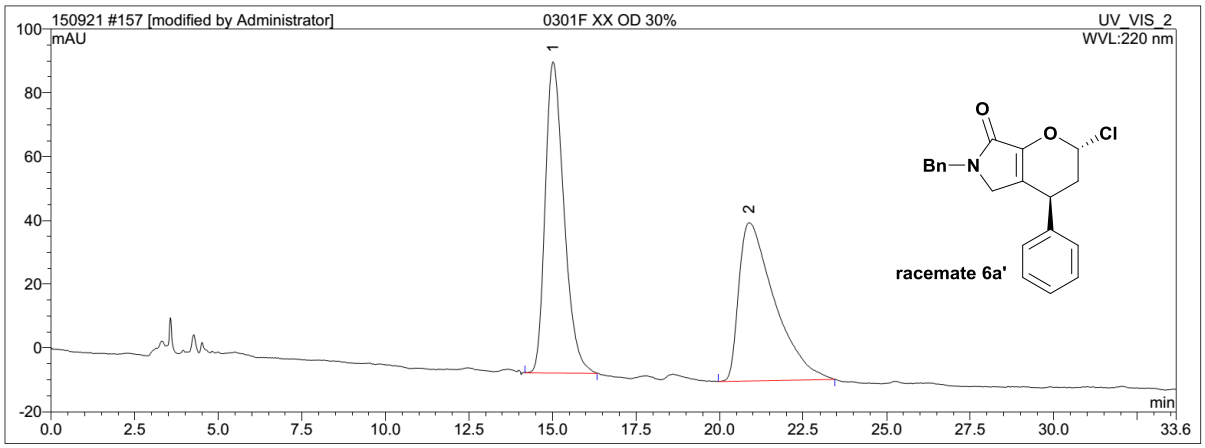

O
O
Hb
Bn—N
Cl
Ha

6a'


no NOE signal
at 6.44 ppm
irradiation Ha
at 4.07 ppm
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

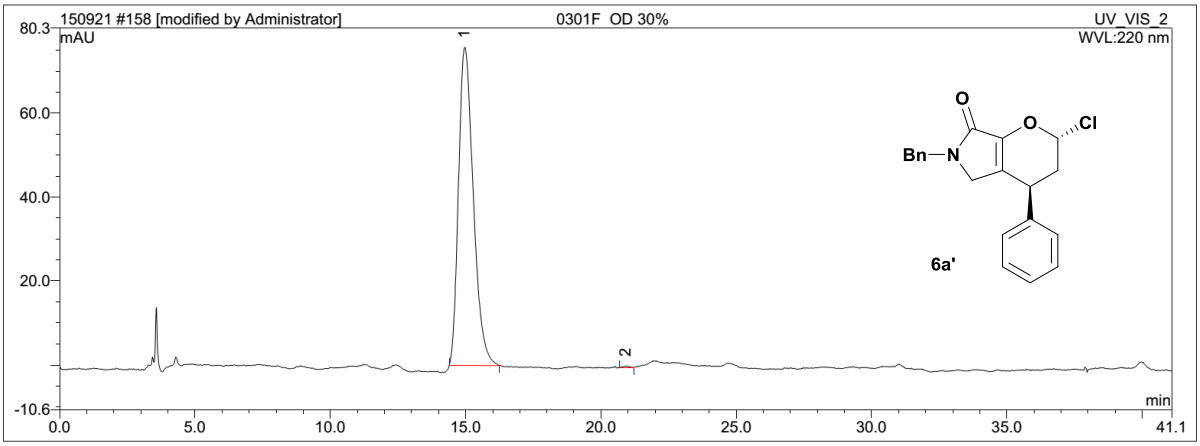
Peak Analysis Report

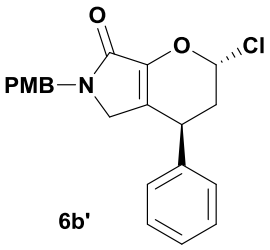
No.	Peak Name	Ret.Time (detected)	Area	Rel.Area	Height	Amount
		min	mAU*min	%	mAU	
1	n.a.	15.02	63.622	50.88	97.648	n.a.
2	n.a.	20.89	61.411	49.12	49.688	n.a.



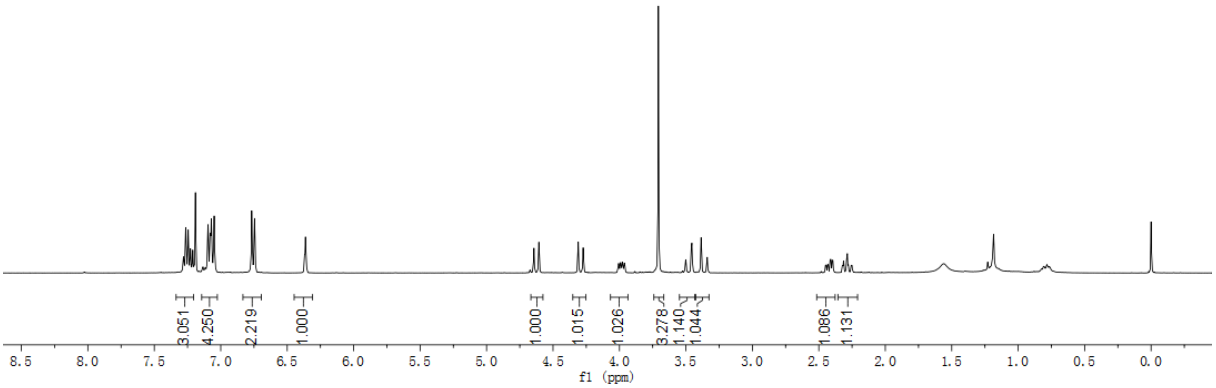
Peak Analysis Report

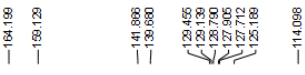
No.	Peak Name	Ret.Time (detected)	Area	Rel.Area	Height	Amount
		min	mAU*min	%	mAU	
1	n.a.	14.96	48.052	99.79	75.858	n.a.
2	n.a.	20.92	0.101	0.21	0.455	n.a.

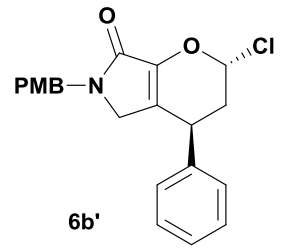



O
O
Cl
PMB—N

6b'


3.051
4.250
2.219
1.000
1.000
1.015
1.026
3.278
1.140
1.044
1.086
1.131
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


164.199
159.129
141.866
139.680
129.455
129.139
128.790
127.905
127.712
125.188
114.098
88.438
55.260
47.284
45.905
39.643
34.769

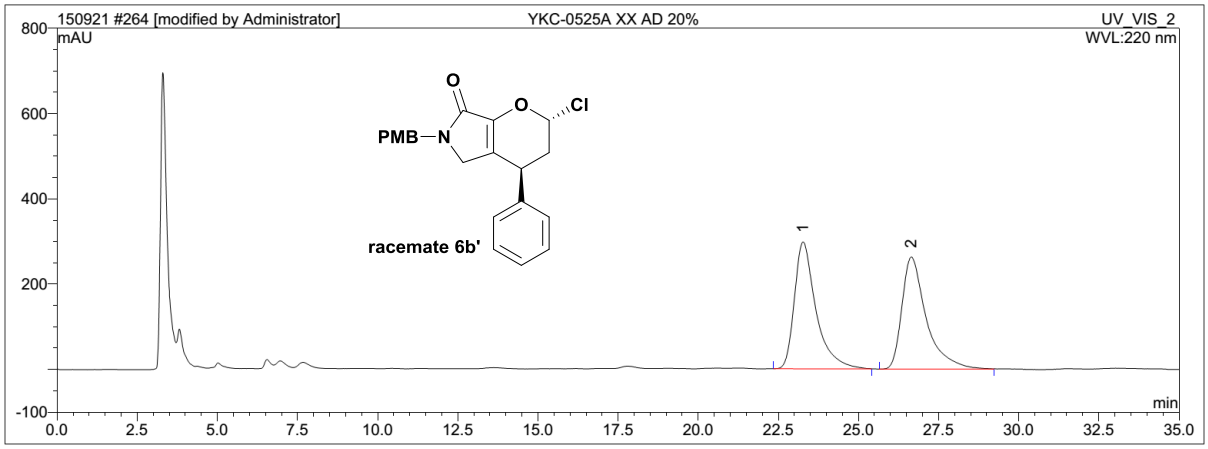

O
O
Cl
PMB—N

6b'

200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

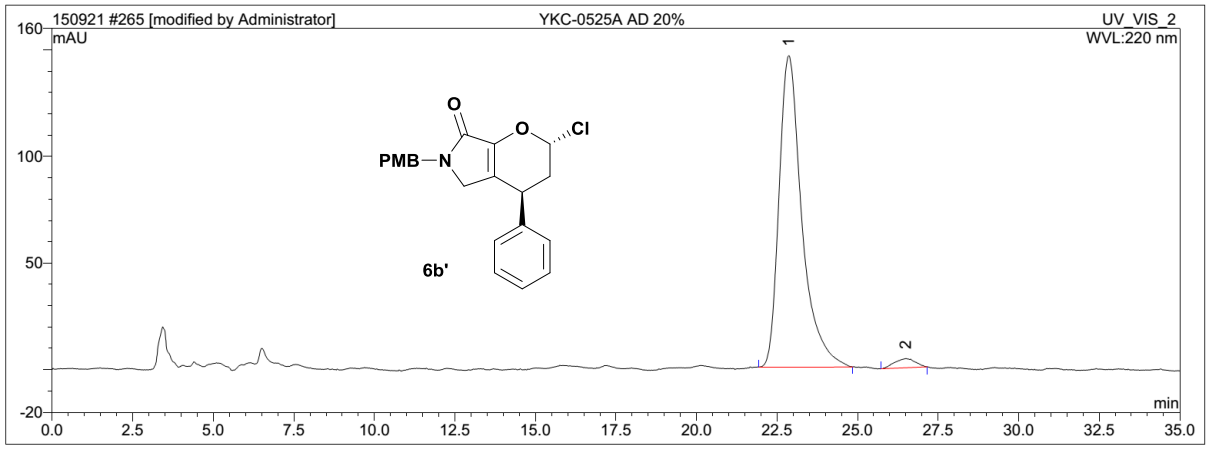
Peak Analysis Report

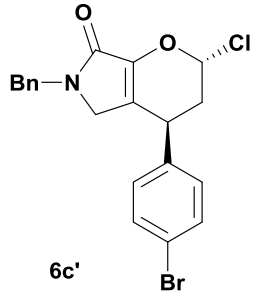
No.	Peak Name	Ret.Time (detected)	Area	Rel.Area	Height	Amount
		min	mAU*min	%	mAU	
1	n.a.	23.28	232.660	50.14	297.723	n.a.
2	n.a.	26.65	231.351	49.86	262.973	n.a.

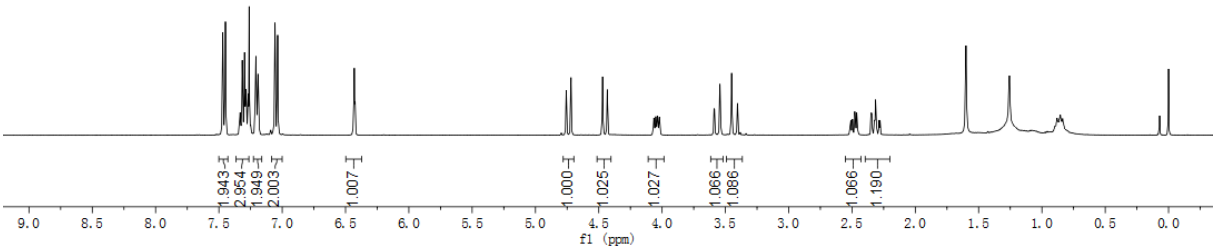


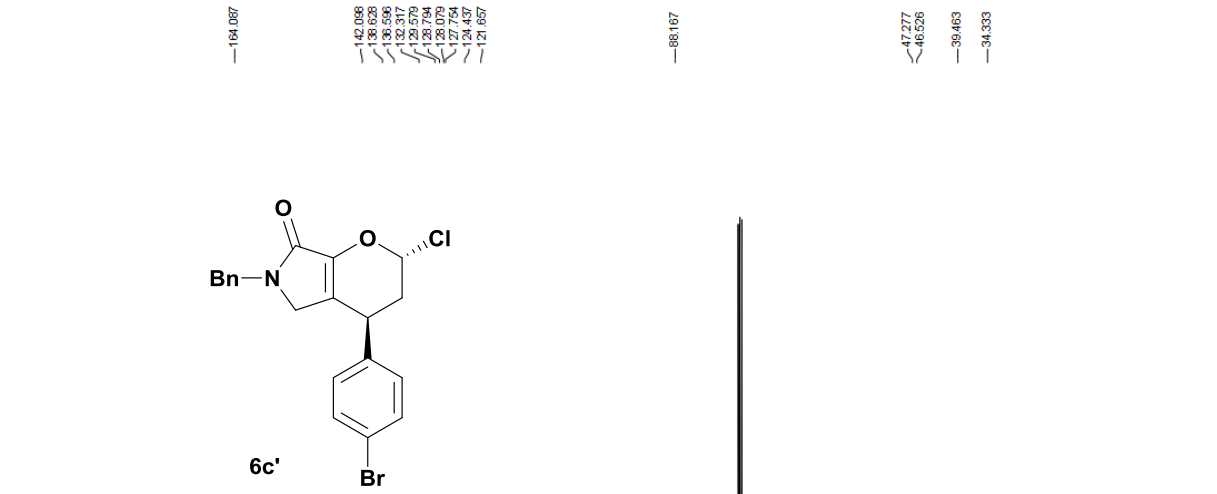
Peak Analysis Report

No.	Peak Name	Ret.Time (detected)	Area	Rel.Area	Height	Amount
		min	mAU*min	%	mAU	
1	n.a.	22.87	119.965	97.41	146.121	n.a.
2	n.a.	26.50	3.192	2.59	4.370	n.a.




O
O
Cl
Bn—N
6c'
Br


1.943
2.954
1.949
2.003
1.007
1.000
1.025
1.027
1.066
1.086
1.066
1.190
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

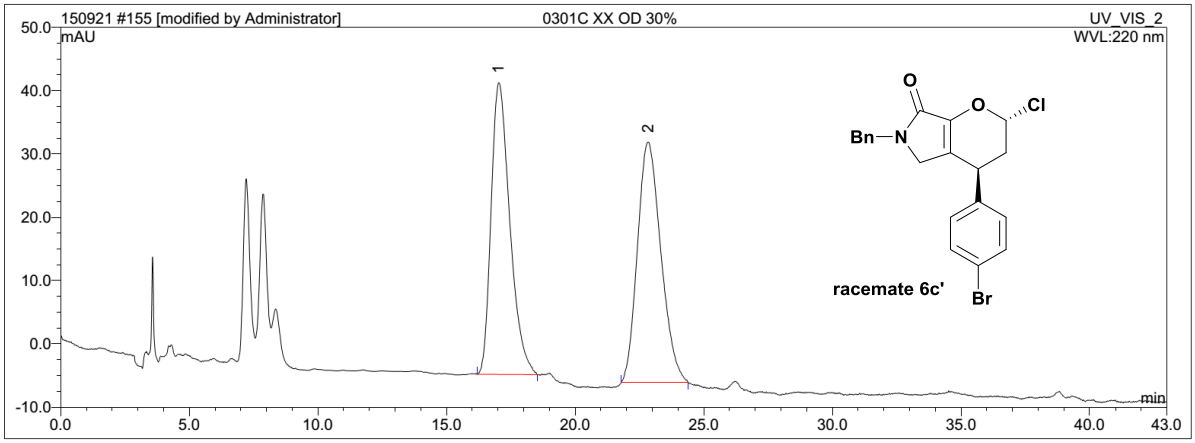

164.087
142.088
136.628
136.596
132.317
129.579
128.794
128.079
127.754
124.437
121.657
88.167
47.277
46.526
39.463
34.333

O
O
Cl
Bn—N
6c'
Br

200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

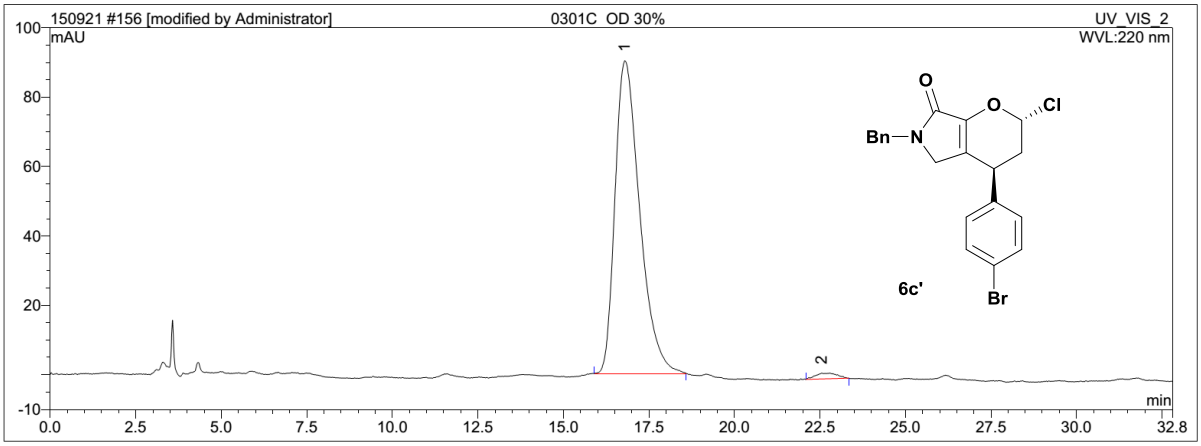
Peak Analysis Report

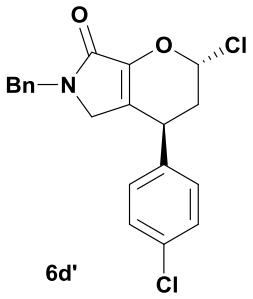
No.	Peak Name	Ret.Time (detected)	Area	Rel.Area	Height	Amount
		min	mAU*min	%	mAU	
1	n.a.	17.03	38.916	49.28	46.102	n.a.
2	n.a.	22.84	40.048	50.72	37.979	n.a.



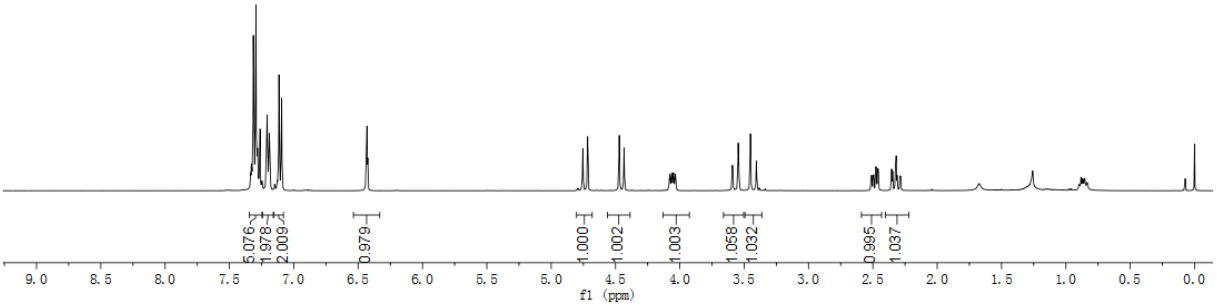
Peak Analysis Report

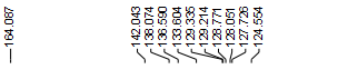
No.	Peak Name	Ret.Time (detected)	Area	Rel.Area	Height	Amount
		min	mAU*min	%	mAU	
1	n.a.	16.79	75.433	98.44	90.166	n.a.
2	n.a.	22.55	1.195	1.56	1.712	n.a.

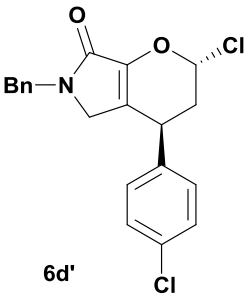



O
O
Cl
Bn—N
Cl

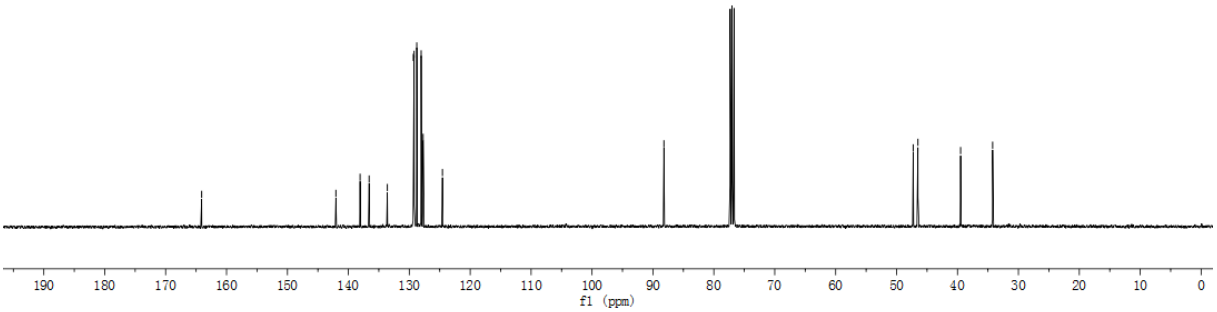
6d'


5.076
1.978
2.009
0.979
1.000
1.002
1.003
1.058
1.032
0.995
1.037
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


164.087
142.043
138.074
136.590
133.604
129.335
129.214
128.771
128.051
127.726
124.554
88.192
47.270
46.498
39.488
34.230

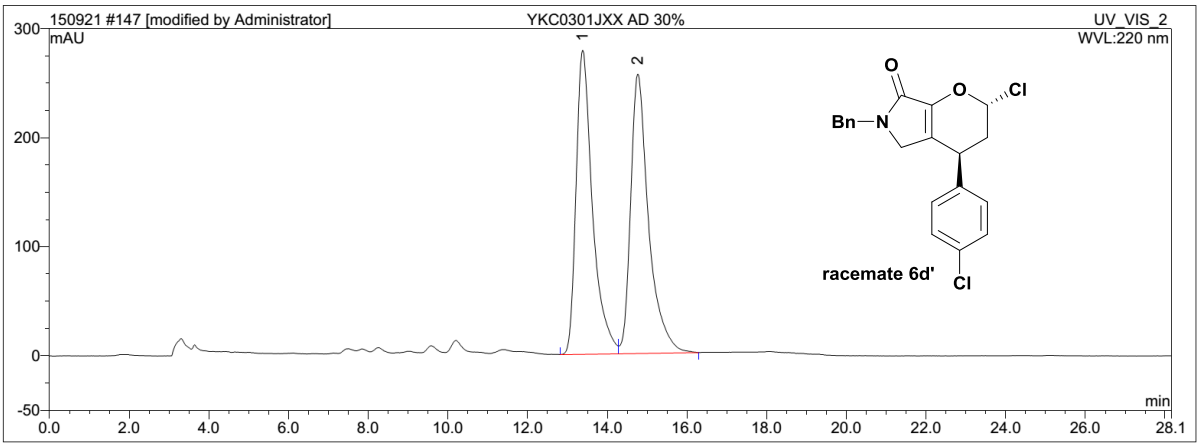

O
O
Cl
Bn—N
Cl

6d'


190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

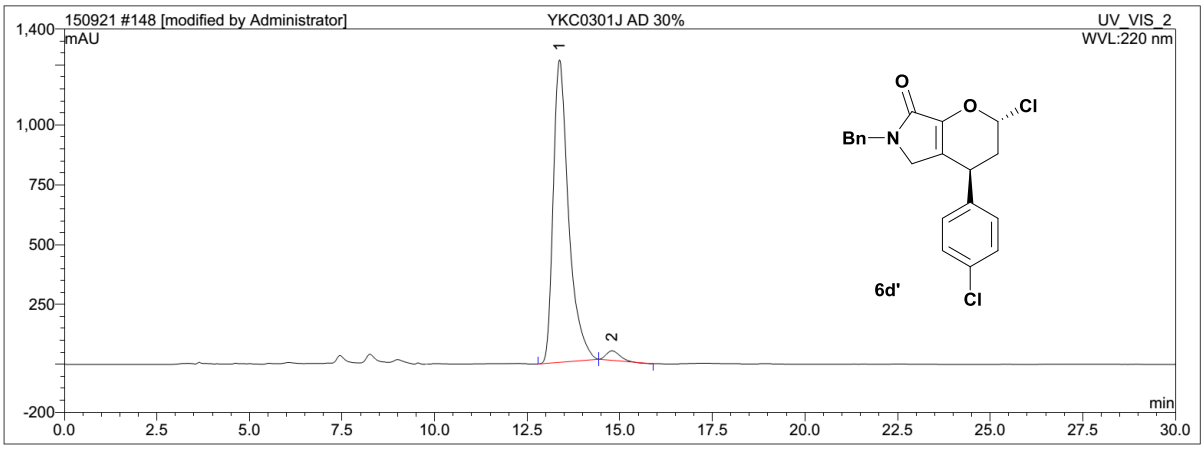
Peak Analysis Report

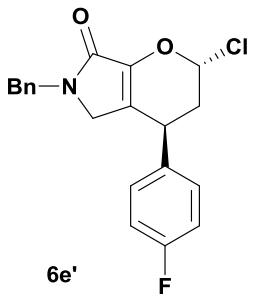
No.	Peak Name	Ret.Time (detected)	Area	Rel.Area	Height	Amount
		min	mAU*min	%	mAU	
1	n.a.	13.38	133.239	49.87	278.990	n.a.
2	n.a.	14.77	133.922	50.13	256.485	n.a.



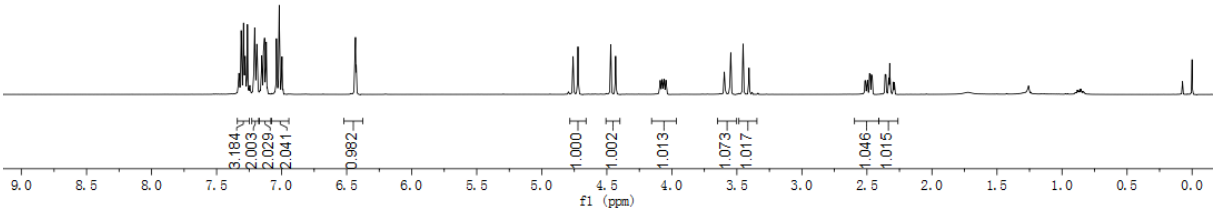
Peak Analysis Report

No.	Peak Name	Ret.Time (detected)	Area	Rel.Area	Height	Amount
		min	mAU*min	%	mAU	
1	n.a.	13.38	608.404	97.39	1263.769	n.a.
2	n.a.	14.79	16.332	2.61	39.536	n.a.

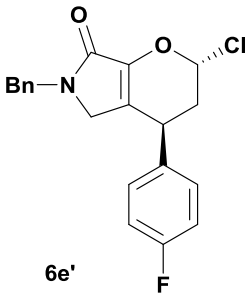



O
O
Cl
Bn—N
F

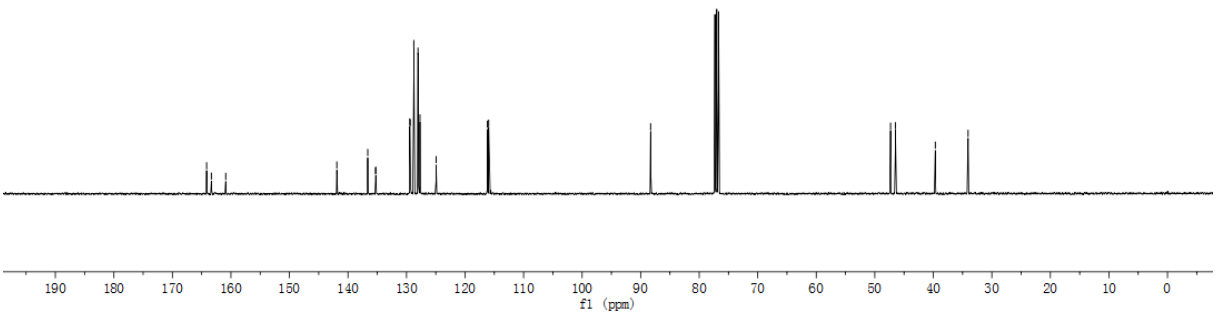
6e'


3.184
2.003
2.029
2.041
0.982
1.000
1.002
1.013
1.073
1.017
1.046
1.015
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


164.151
163.339
160.885
141.906
136.623
135.262
135.249
129.443
129.363
128.751
128.031
127.698
124.942
116.184
115.970
88.294
47.302
46.479
39.658
34.060

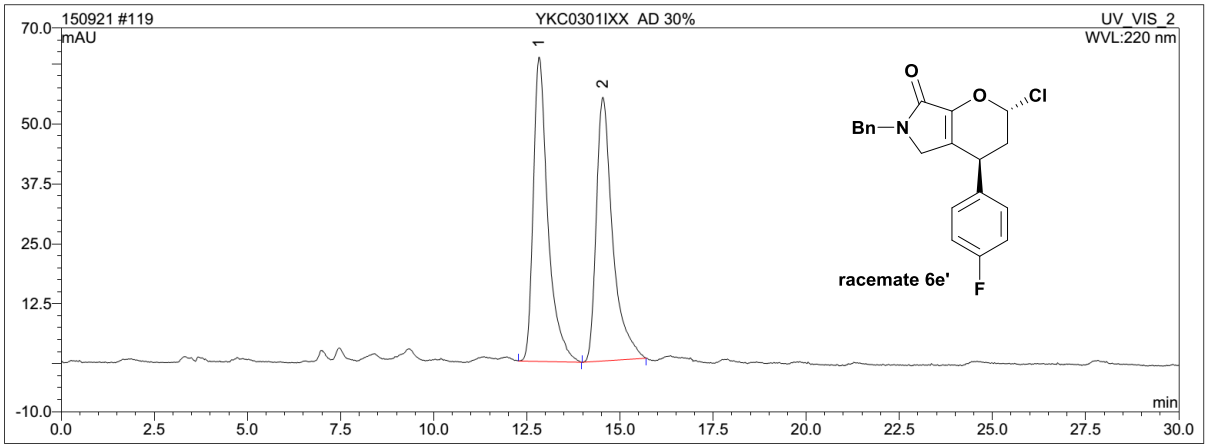

O
O
Cl
Bn—N
F

6e'


190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

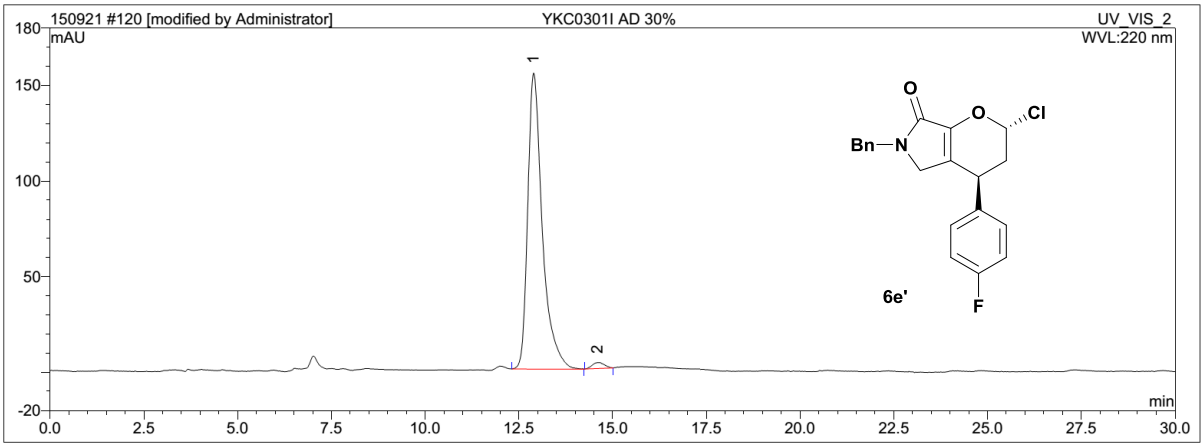
Peak Analysis Report

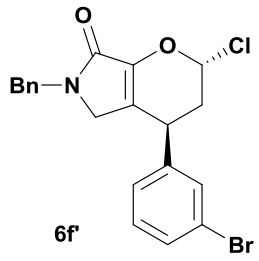
No.	Peak Name	Ret.Time (detected)	Area	Rel.Area	Height	Amount
		min	mAU*min	%	mAU	
1	n.a.	12.83	28.779	50.64	63.502	n.a.
2	n.a.	14.54	28.048	49.36	55.059	n.a.

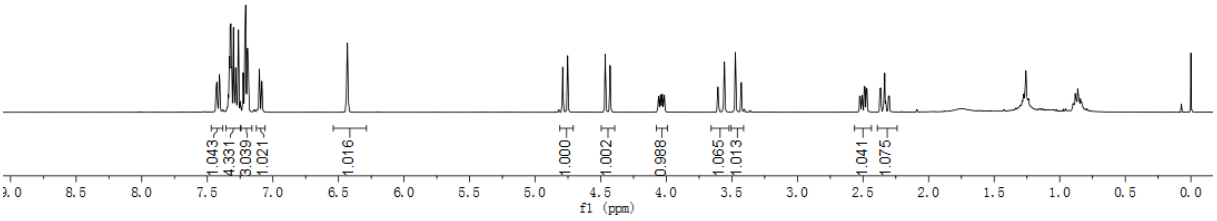


Peak Analysis Report

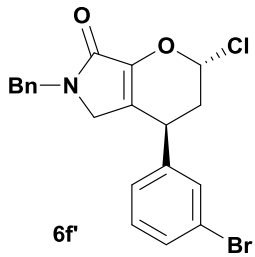
No.	Peak Name	Ret.Time (detected)	Area	Rel.Area	Height	Amount
		min	mAU*min	%	mAU	
1	n.a.	12.89	69.731	98.34	154.877	n.a.
2	n.a.	14.60	1.179	1.66	3.162	n.a.

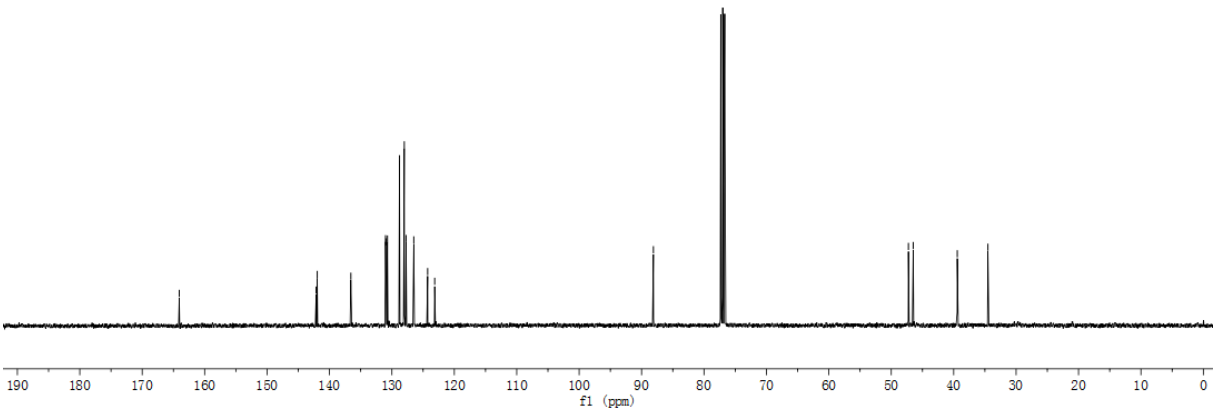



O
O
Cl
Bn—N
6f'
Br


1.043
4.331
3.039
1.021
1.016
1.000
1.002
0.988
1.065
1.013
1.041
1.075
f1 (ppm)

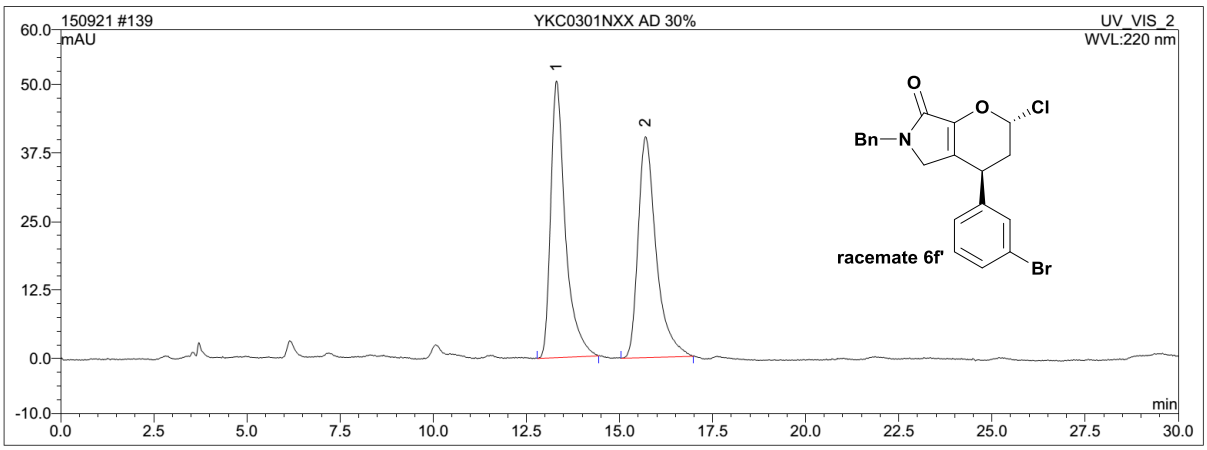

164.086
142.137
141.962
136.574
131.020
130.948
130.714
128.783
128.706
127.725
126.492
124.272
123.140
88.109
47.251
46.482
39.425
34.519


O
O
Cl
Bn—N
6f'
Br


f1 (ppm)

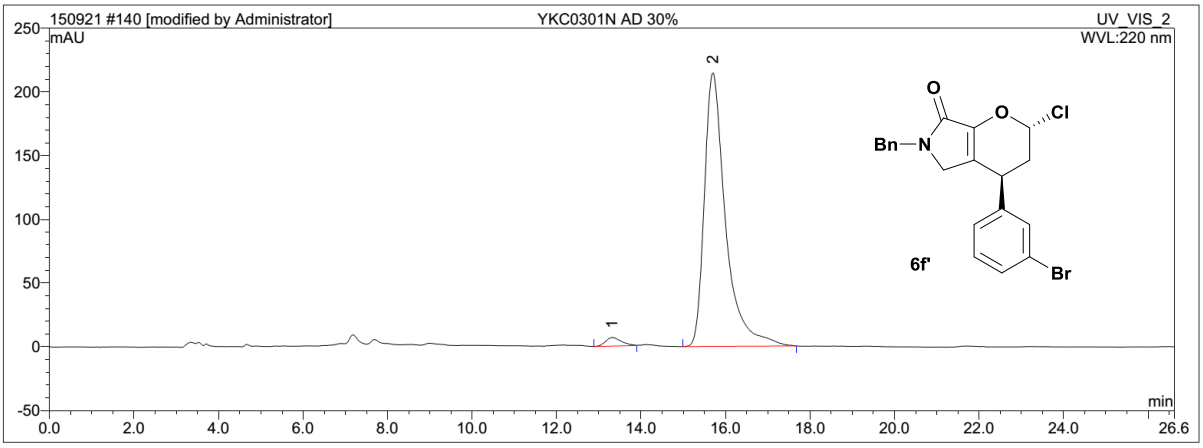
Peak Analysis Report

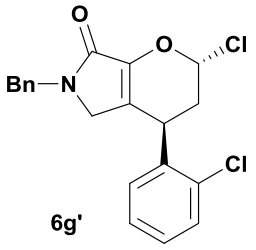
No.	Peak Name	Ret.Time (detected)	Area	Rel.Area	Height	Amount
		min	mAU*min	%	mAU	
1	n.a.	13.31	23.058	50.41	50.524	n.a.
2	n.a.	15.70	22.684	49.59	40.345	n.a.



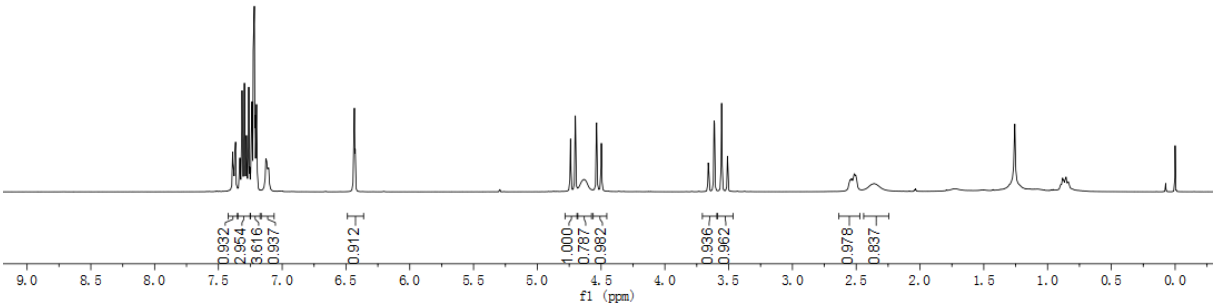
Peak Analysis Report

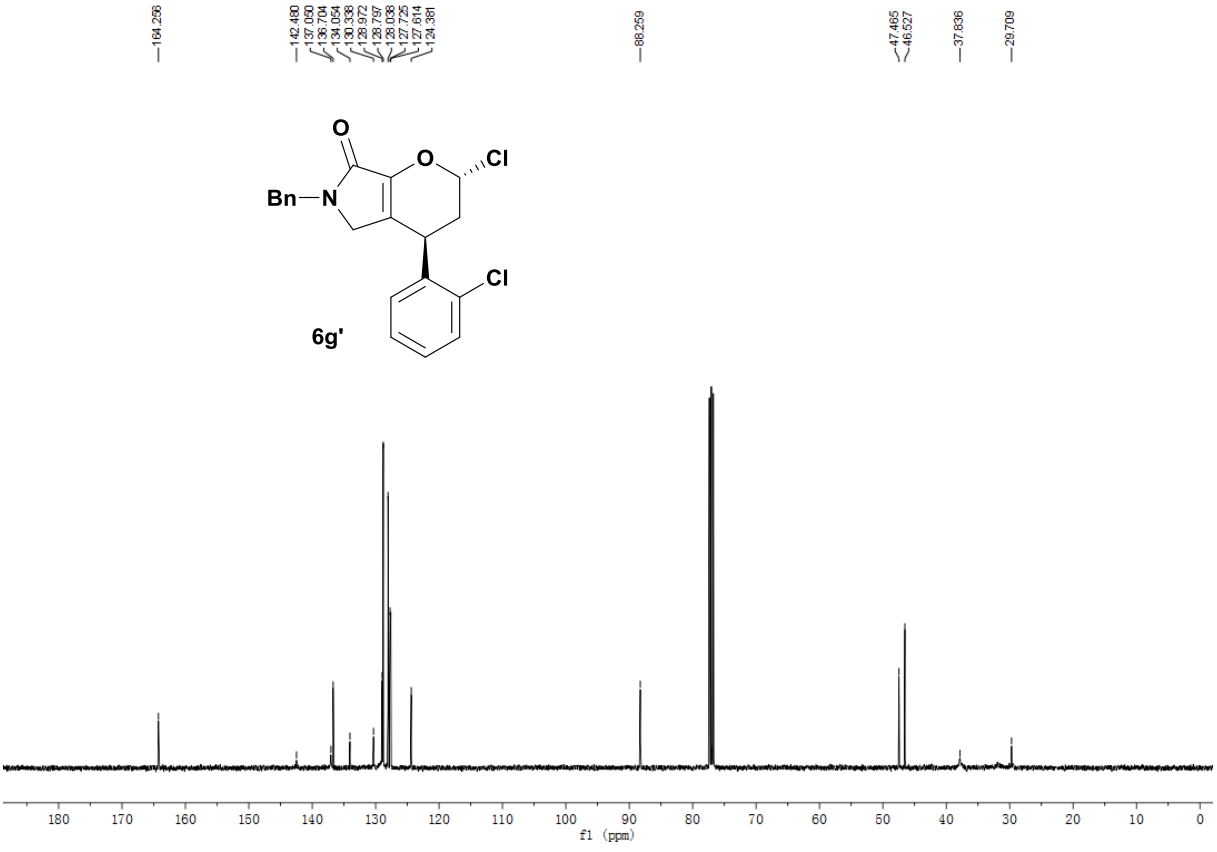
No.	Peak Name	Ret.Time (detected)	Area	Rel.Area	Height	Amount
		min	mAU*min	%	mAU	
1	n.a.	13.32	2.858	2.23	6.726	n.a.
2	n.a.	15.70	125.070	97.77	214.826	n.a.




O
O
Cl
Bn—N
Cl

6g'

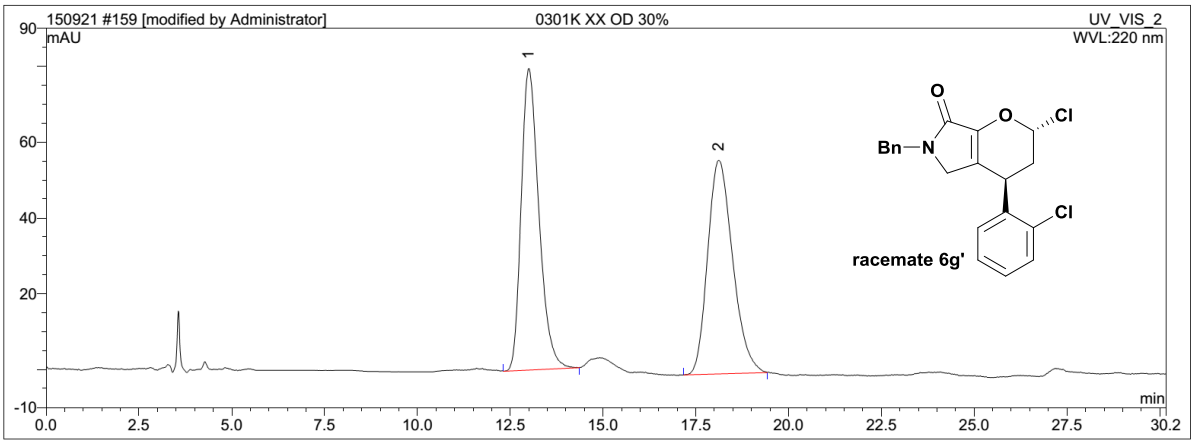

0.932
2.954
3.616
0.937
0.912
1.000
0.787
0.982
0.936
0.962
0.978
0.837
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


164.256
142.490
137.050
136.704
134.054
130.338
128.972
128.797
128.038
127.725
127.614
124.381
88.259
47.465
46.527
37.836
29.709
O
O
Cl
Bn—N
Cl
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

6g'

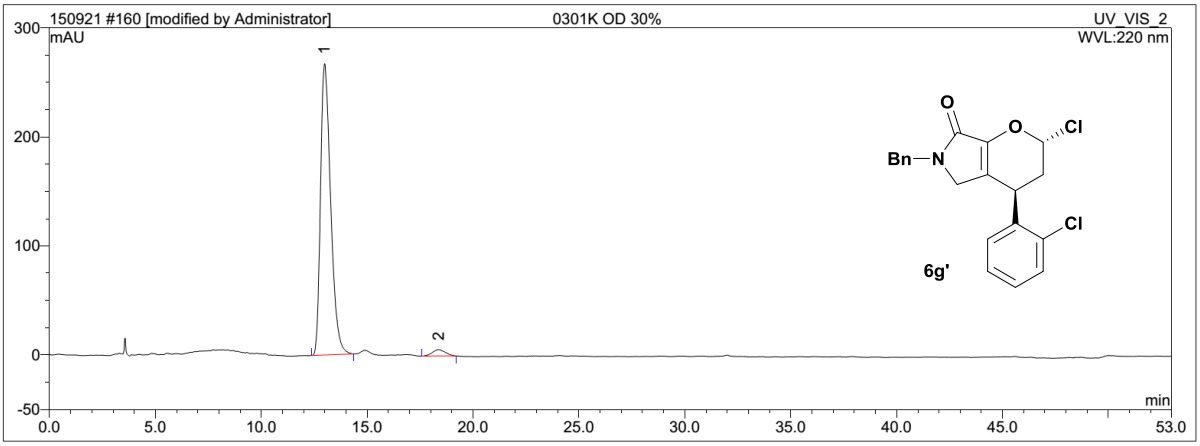
Peak Analysis Report

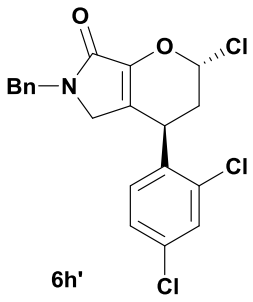
No.	Peak Name	Ret.Time (detected)	Area	Rel.Area	Height	Amount
		min	mAU*min	%	mAU	
1	n.a.	13.00	43.992	50.00	79.581	n.a.
2	n.a.	18.12	43.997	50.00	56.322	n.a.

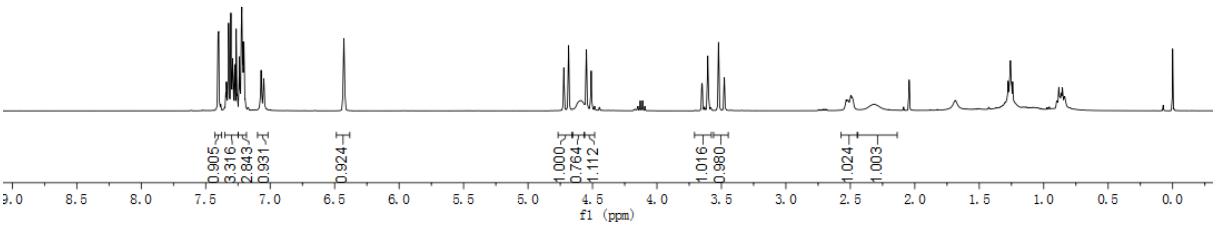


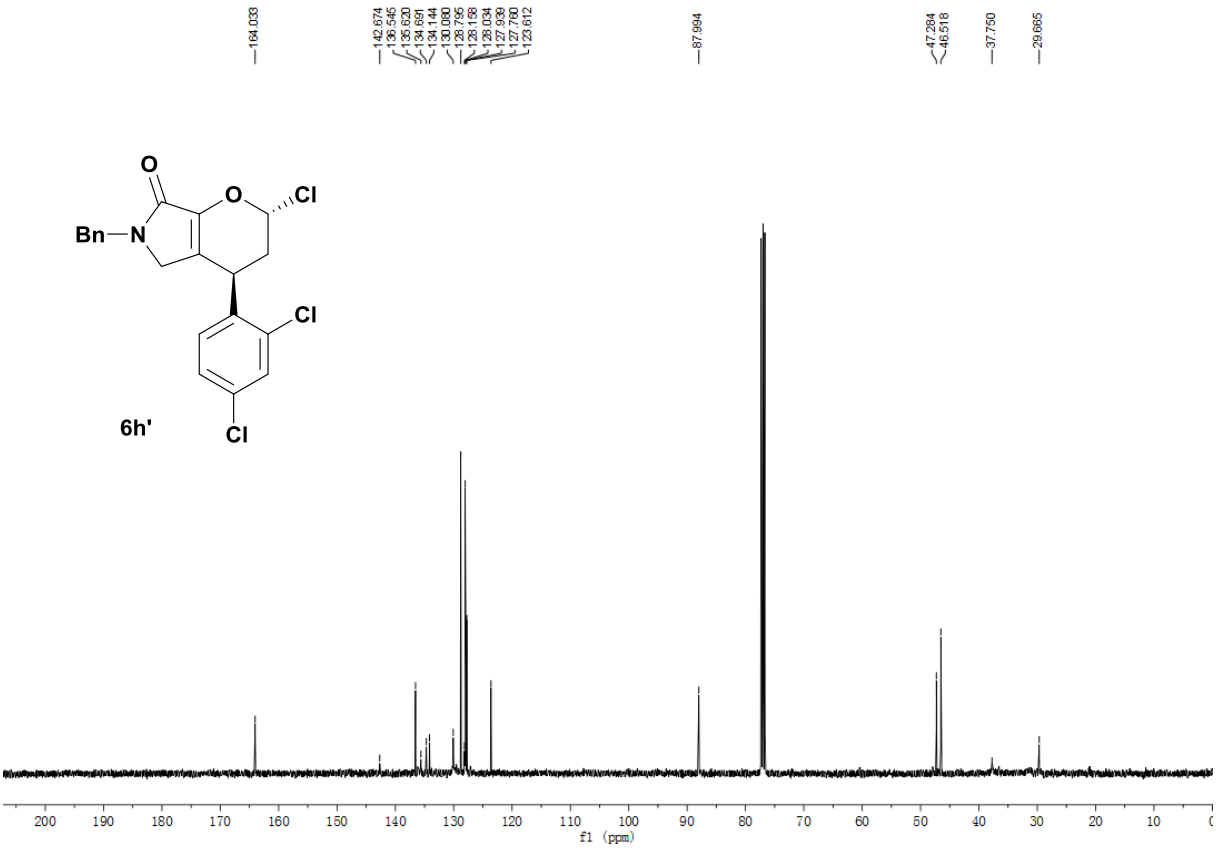
Peak Analysis Report

No.	Peak Name	Ret.Time (detected)	Area	Rel.Area	Height	Amount
		min	mAU*min	%	mAU	
1	n.a.	12.99	148.530	97.28	267.407	n.a.
2	n.a.	18.38	4.146	2.72	5.801	n.a.



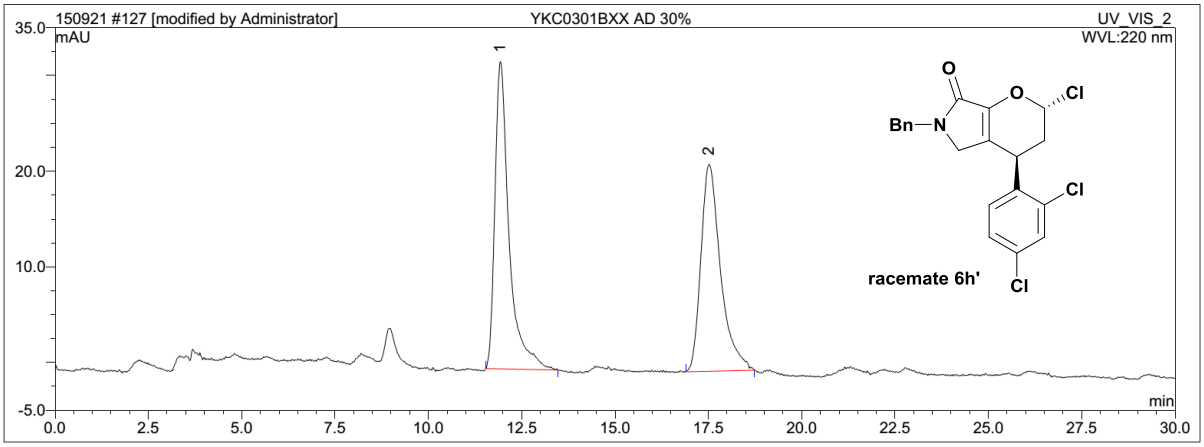

O
O
Cl
Bn—N
Cl
Cl
6h'


0.905
3.316
2.843
0.931
0.924
1.000
0.764
1.112
1.016
0.980
1.024
1.003
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


164.033
142.674
136.545
135.620
134.691
134.144
130.080
128.795
128.158
128.034
127.938
127.780
123.612
87.994
47.284
46.518
37.750
29.665
O
O
Cl
Bn—N
Cl
Cl
6h'
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

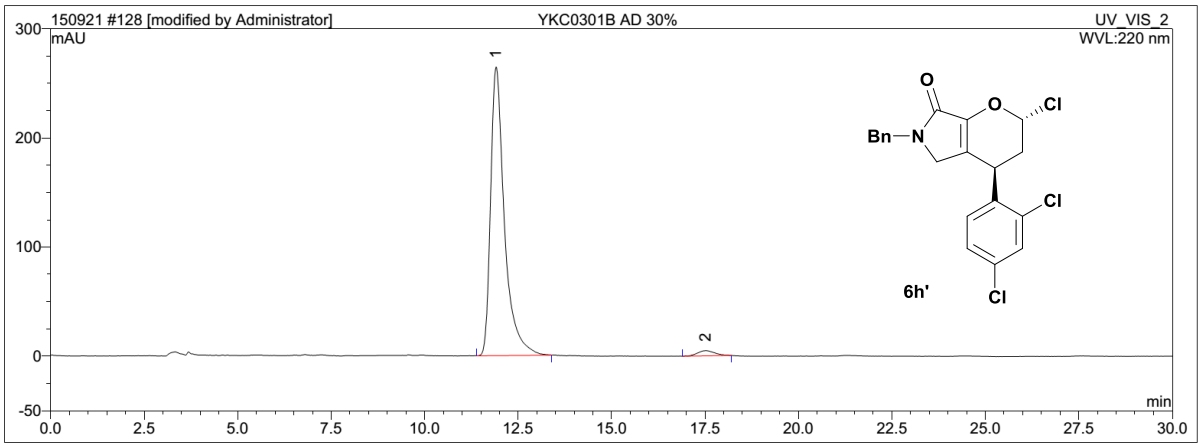
Peak Analysis Report

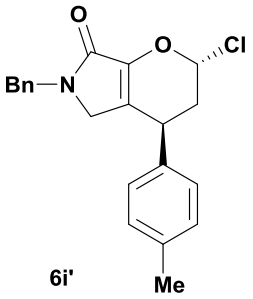
No.	Peak Name	Ret.Time (detected)	Area	Rel.Area	Height	Amount
		min	mAU*min	%	mAU	
1	n.a.	11.93	14.096	51.58	32.150	n.a.
2	n.a.	17.52	13.235	48.42	21.654	n.a.



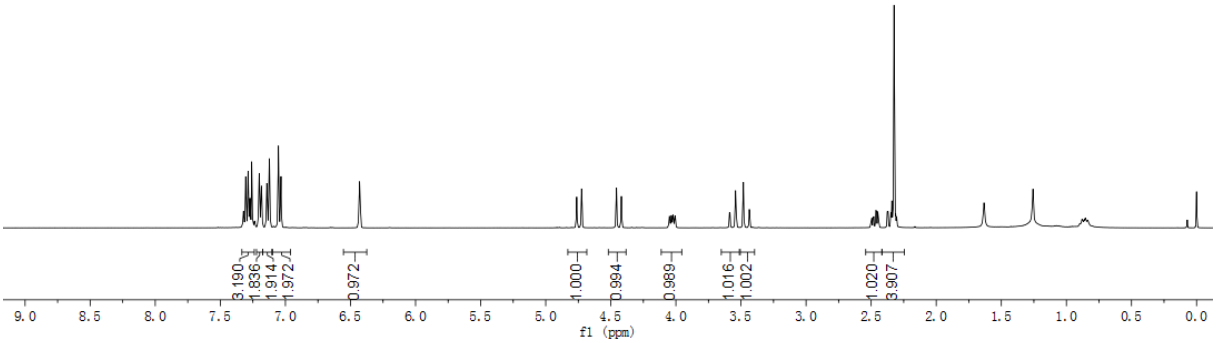
Peak Analysis Report

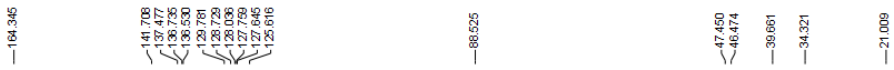
No.	Peak Name	Ret.Time (detected)	Area	Rel.Area	Height	Amount
		min	mAU*min	%	mAU	
1	n.a.	11.91	113.162	97.80	264.559	n.a.
2	n.a.	17.52	2.547	2.20	4.776	n.a.

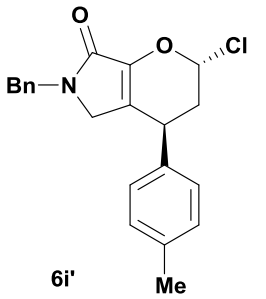



O
O
Cl
Bn—N
Me

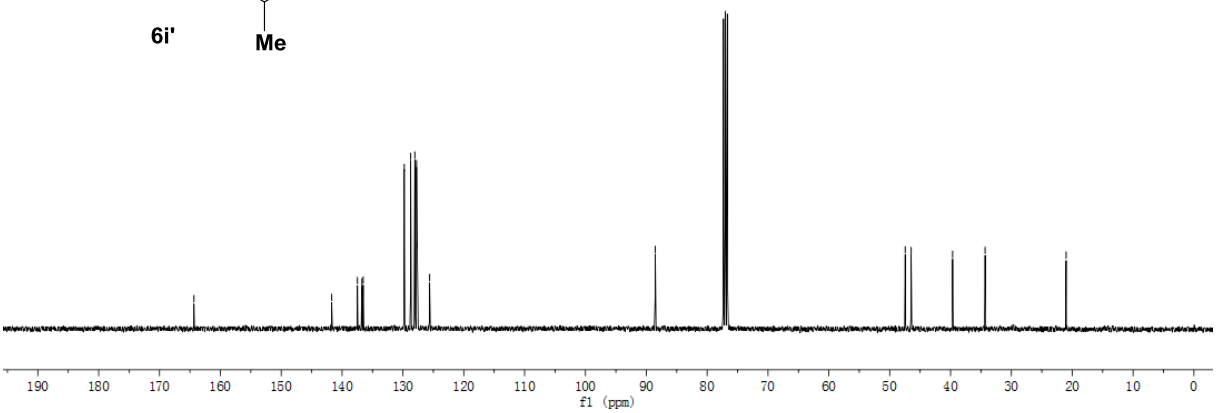
6i'


3.190
1.836
1.914
1.972
0.972
1.000
0.994
0.989
1.016
1.002
1.020
3.907
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


164.345
141.708
137.477
136.735
136.530
129.781
128.729
128.036
127.759
127.645
125.616
88.525
47.450
46.474
39.661
34.321
21.009

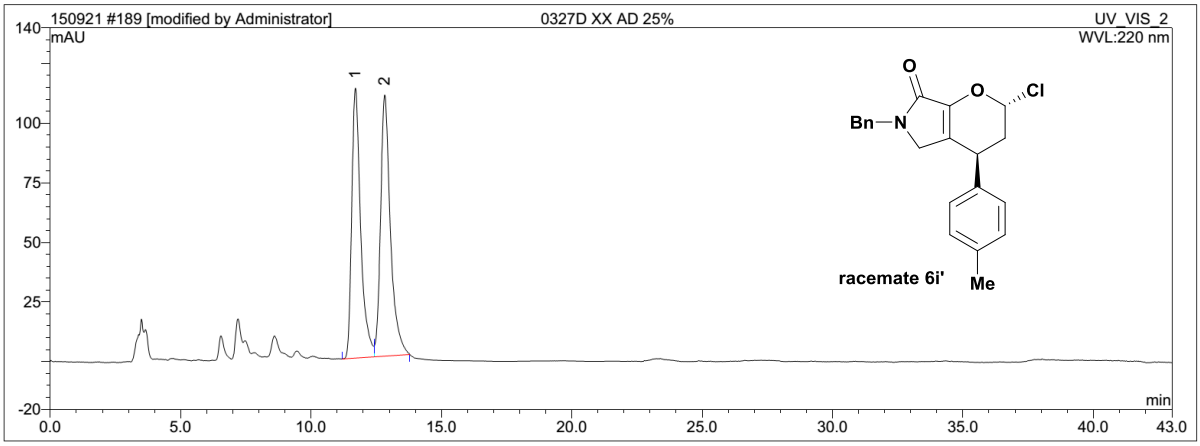

O
O
Cl
Bn—N
Me

6i'


190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

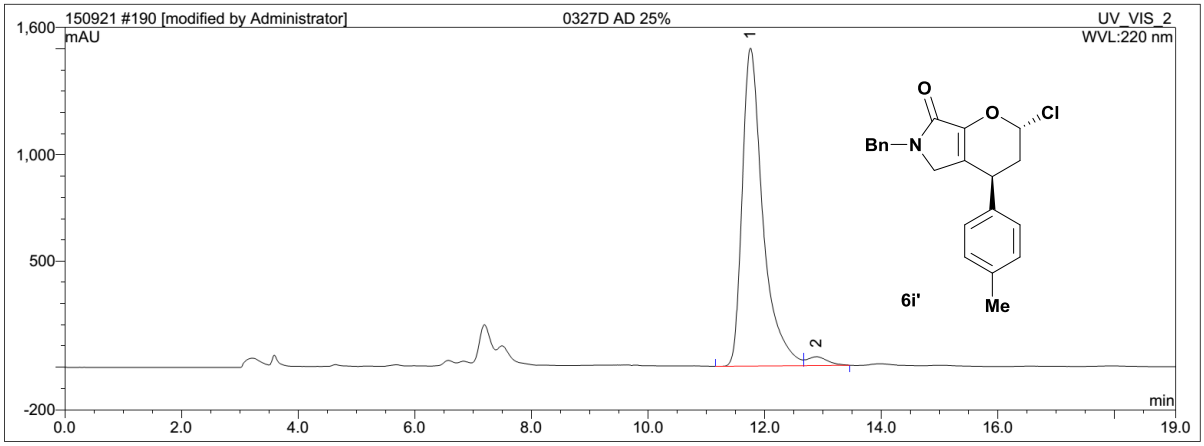
Peak Analysis Report

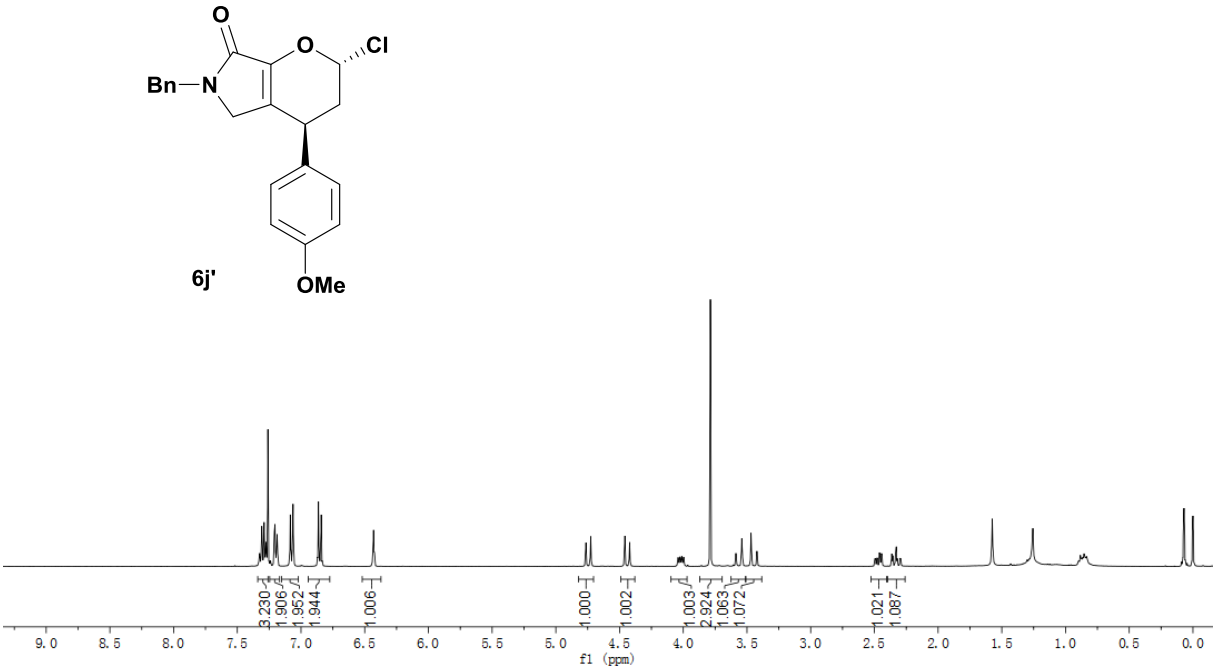
No.	Peak Name	Ret.Time (detected)	Area	Rel.Area	Height	Amount
		min	mAU*min	%	mAU	
1	n.a.	11.70	44.845	48.20	113.276	n.a.
2	n.a.	12.83	48.189	51.80	109.593	n.a.

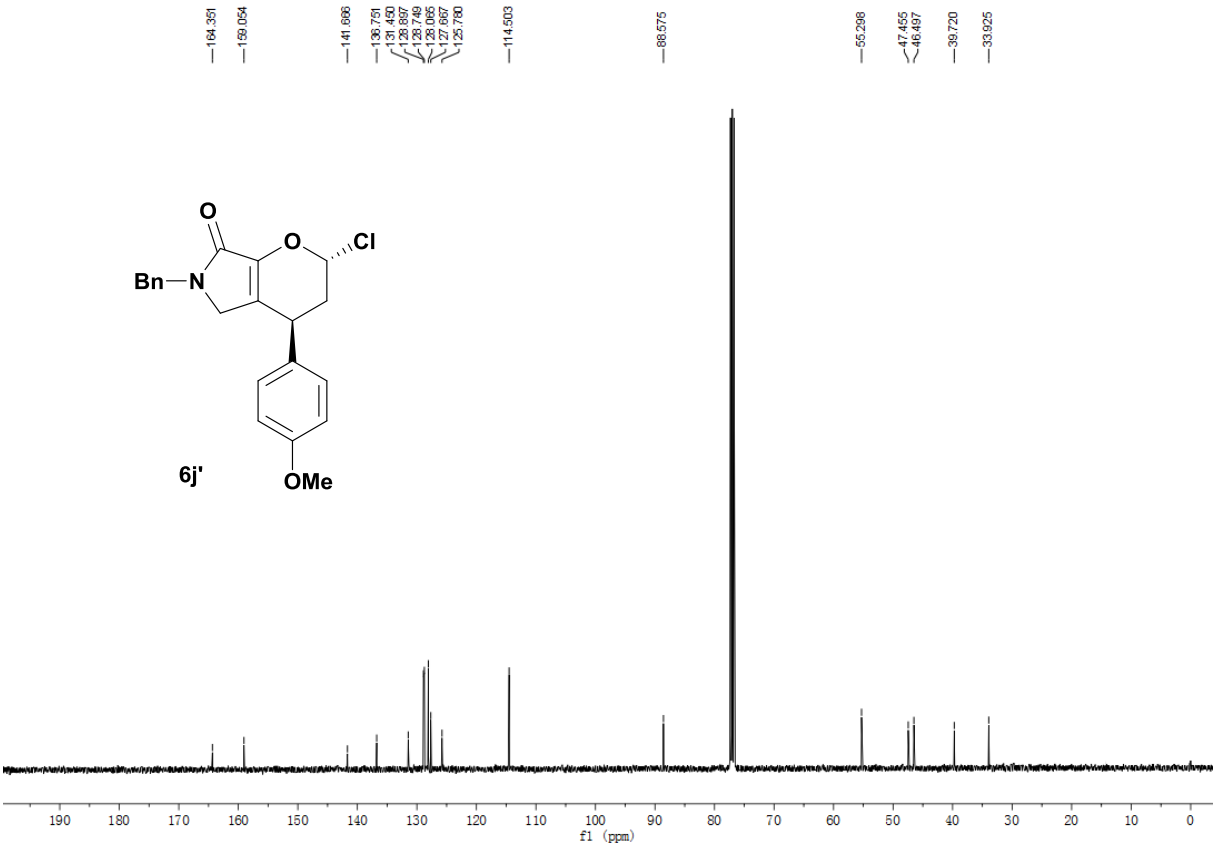


Peak Analysis Report

No.	Peak Name	Ret.Time (detected)	Area	Rel.Area	Height	Amount
		min	mAU*min	%	mAU	
1	n.a.	11.76	616.364	97.37	1497.797	n.a.
2	n.a.	12.88	16.675	2.63	41.349	n.a.

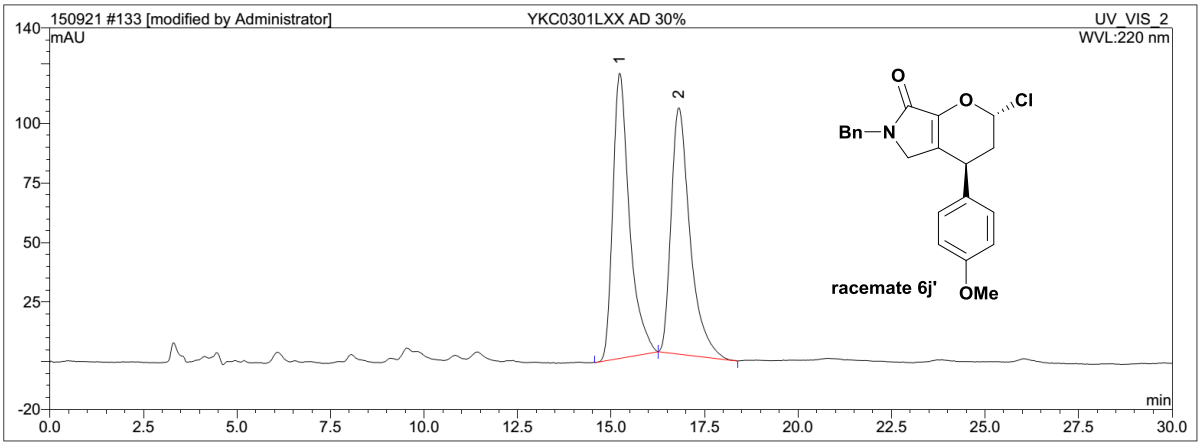



O
O
Cl
Bn—N
6j'
OMe
3.230
1.906
1.952
1.944
1.006
1.000
1.002
1.003
2.924
1.063
1.072
1.021
1.087
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


164.351
159.054
141.686
136.751
131.450
128.997
128.749
128.085
127.667
125.780
114.503
88.575
55.298
47.455
46.497
39.720
33.925
O
O
Cl
Bn—N
6j'
OMe
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

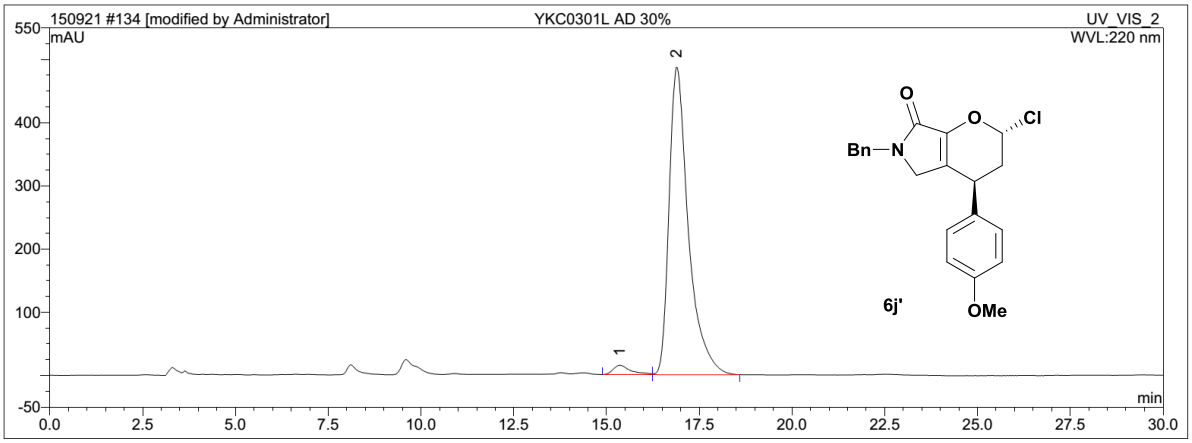
Peak Analysis Report

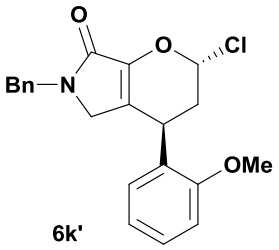
No.	Peak Name	Ret.Time (detected)	Area	Rel.Area	Height	Amount
		min	mAU*min	%	mAU	
1	n.a.	15.23	61.865	50.47	119.659	n.a.
2	n.a.	16.81	60.712	49.53	103.407	n.a.



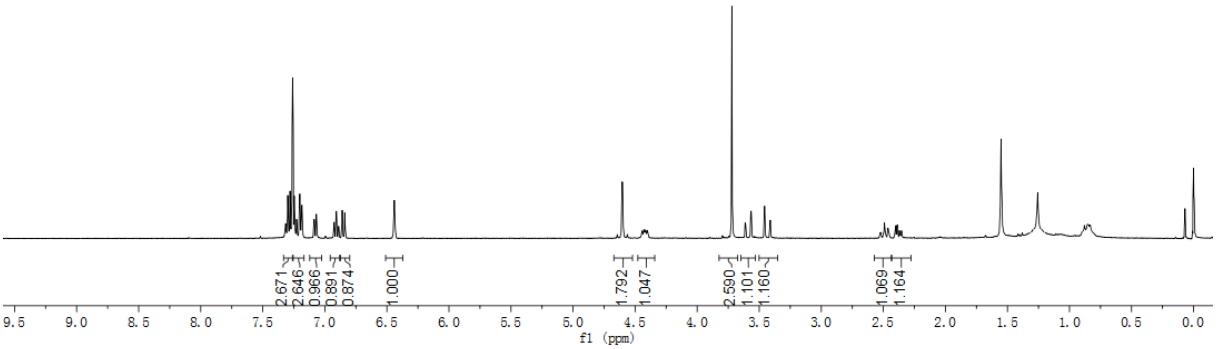
Peak Analysis Report

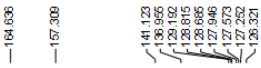
No.	Peak Name	Ret.Time (detected)	Area	Rel.Area	Height	Amount
		min	mAU*min	%	mAU	
1	n.a.	15.36	8.069	2.68	14.739	n.a.
2	n.a.	16.89	293.470	97.32	487.195	n.a.

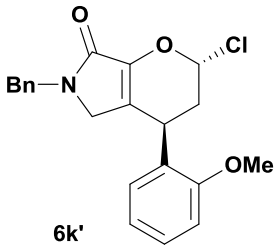



O
O
Cl
Bn—N
OMe

6k'


2.671
2.646
0.966
0.891
0.874
1.000
1.792
1.047
2.590
1.101
1.160
1.069
1.164
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


164.636
157.309
141.123
130.955
129.192
128.815
128.685
127.946
127.573
127.252
126.321
120.952
110.892
88.966
55.192
47.634
46.372
37.098
29.691

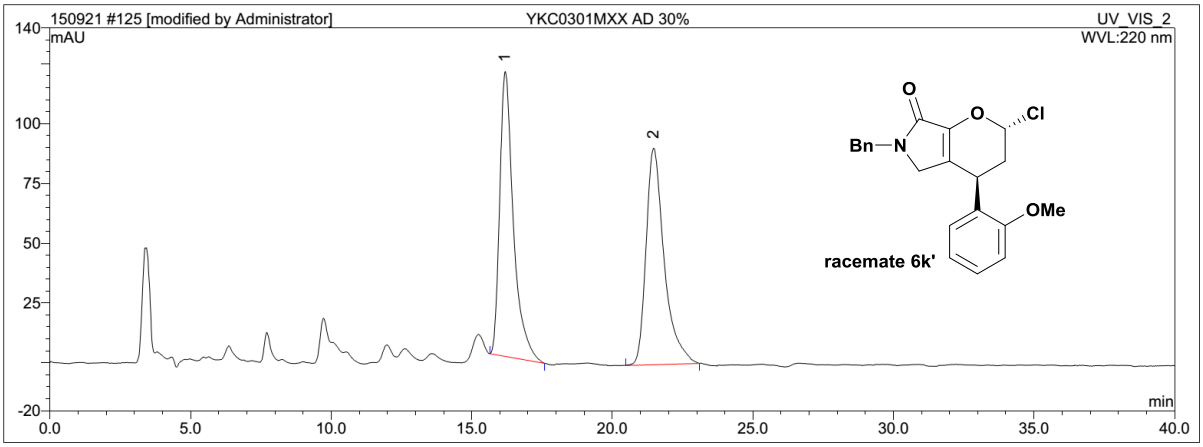

O
O
Cl
Bn—N
OMe

6k'

190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

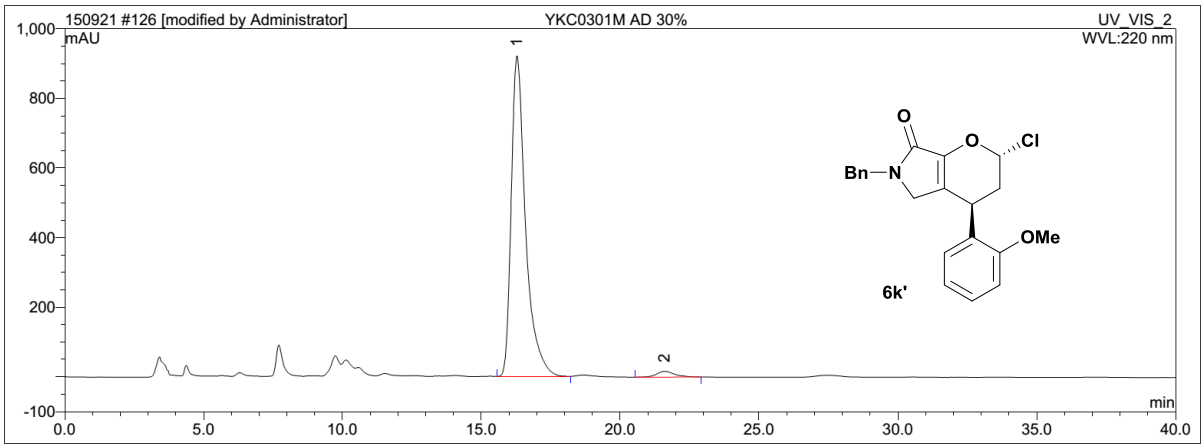
Peak Analysis Report

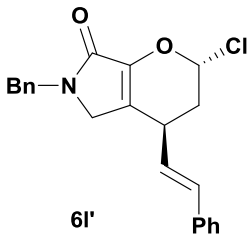
No.	Peak Name	Ret.Time (detected)	Area	Rel.Area	Height	Amount
		min	mAU*min	%	mAU	
1	n.a.	16.18	67.047	49.70	119.105	n.a.
2	n.a.	21.47	67.867	50.30	90.616	n.a.



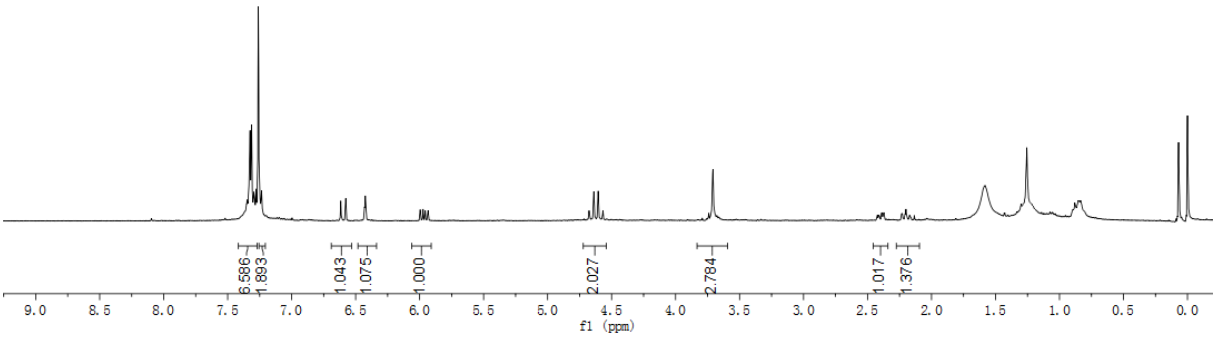
Peak Analysis Report

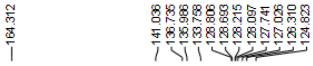
No.	Peak Name	Ret.Time (detected)	Area	Rel.Area	Height	Amount
		min	mAU*min	%	mAU	
1	n.a.	16.28	551.896	97.82	921.409	n.a.
2	n.a.	21.60	12.319	2.18	16.597	n.a.

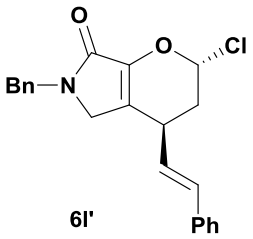



O
O
Cl
Bn—N
Ph

6l'


6.586
1.893
1.043
1.075
1.000
2.027
2.784
1.017
1.376
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


164.312
141.036
136.735
135.996
133.758
128.806
128.693
128.215
128.097
127.741
127.026
126.310
124.823
88.277
47.736
46.573
37.139
32.355

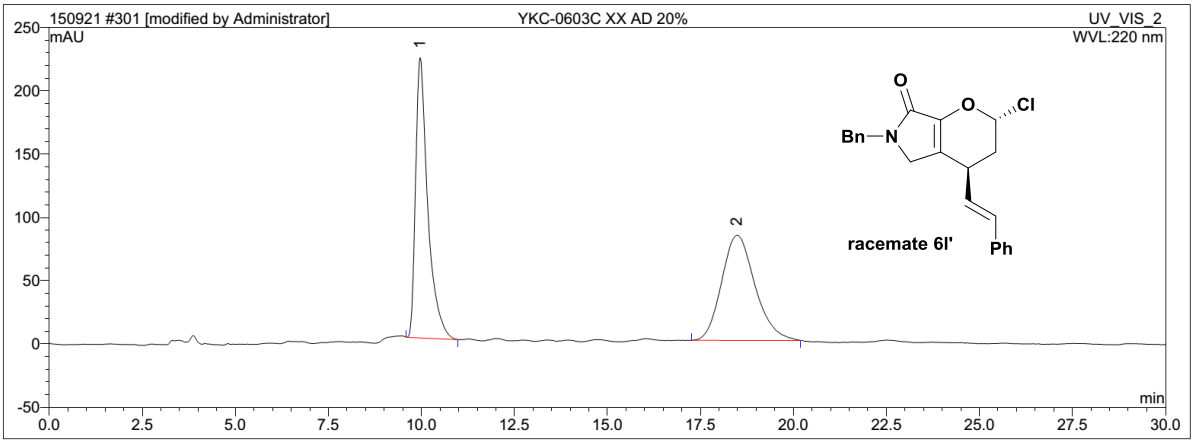

O
O
Cl
Bn—N
Ph

6l'

190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

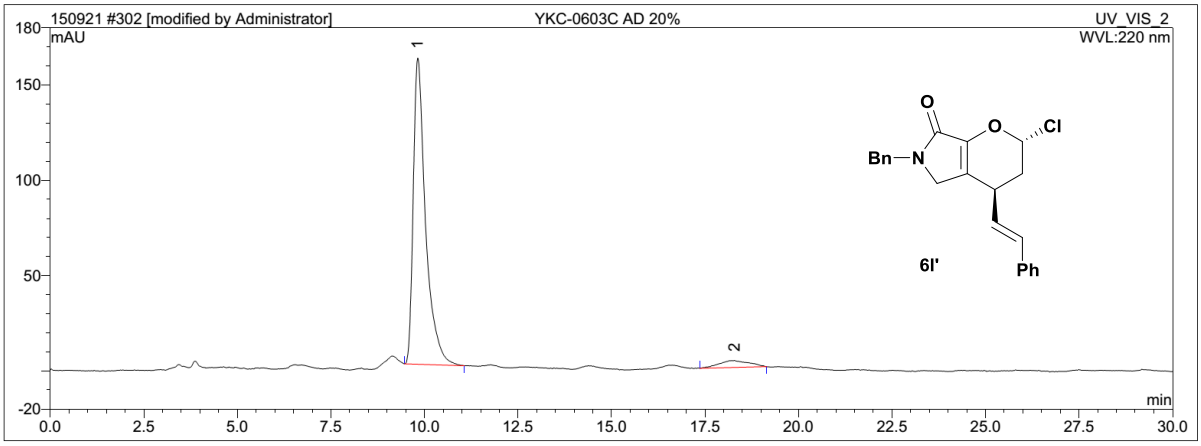
Peak Analysis Report

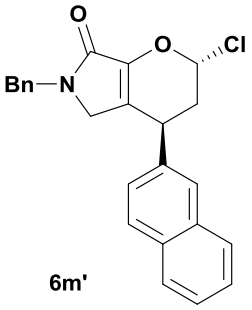
No.	Peak Name	Ret.Time (detected)	Area	Rel.Area	Height	Amount
		min	mAU*min	%	mAU	
1	n.a.	9.97	83.829	49.24	221.498	n.a.
2	n.a.	18.48	86.407	50.76	83.172	n.a.



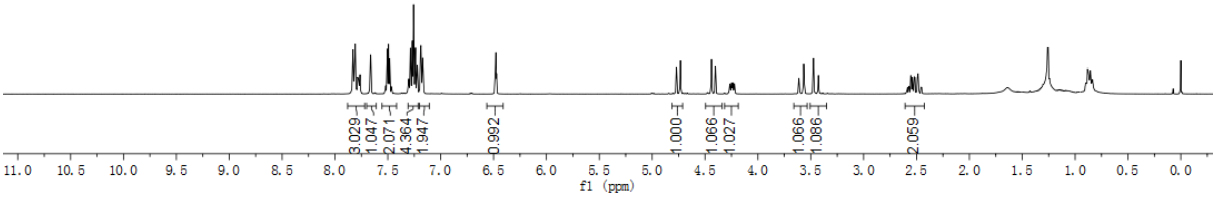
Peak Analysis Report

No.	Peak Name	Ret.Time (detected)	Area	Rel.Area	Height	Amount
		min	mAU*min	%	mAU	
1	n.a.	9.83	61.177	94.81	160.809	n.a.
2	n.a.	18.29	3.352	5.19	3.624	n.a.

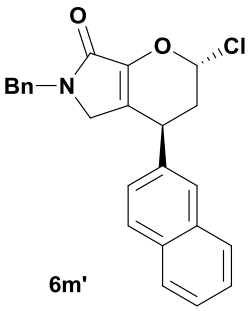



O
O
Cl
Bn—N

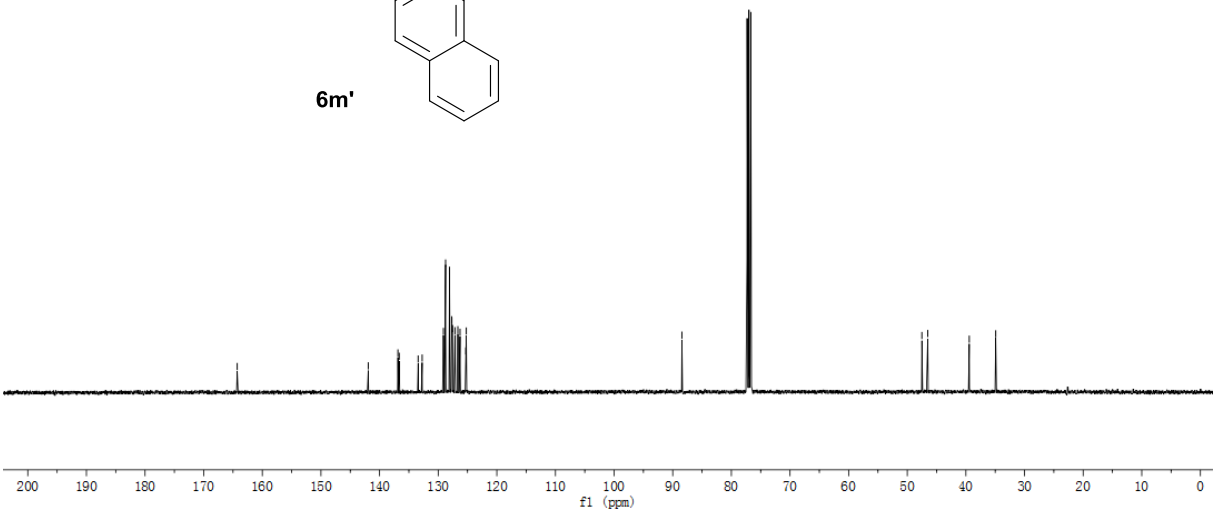
6m'


3.029
1.047
2.071
4.364
1.947
0.992
1.000
1.066
1.027
1.066
1.086
2.059
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


164.284
141.926
136.868
136.648
133.418
132.736
129.113
128.739
128.074
127.698
127.677
127.549
127.142
126.649
126.275
125.292
125.215
88.433
47.507
46.508
39.427
34.919

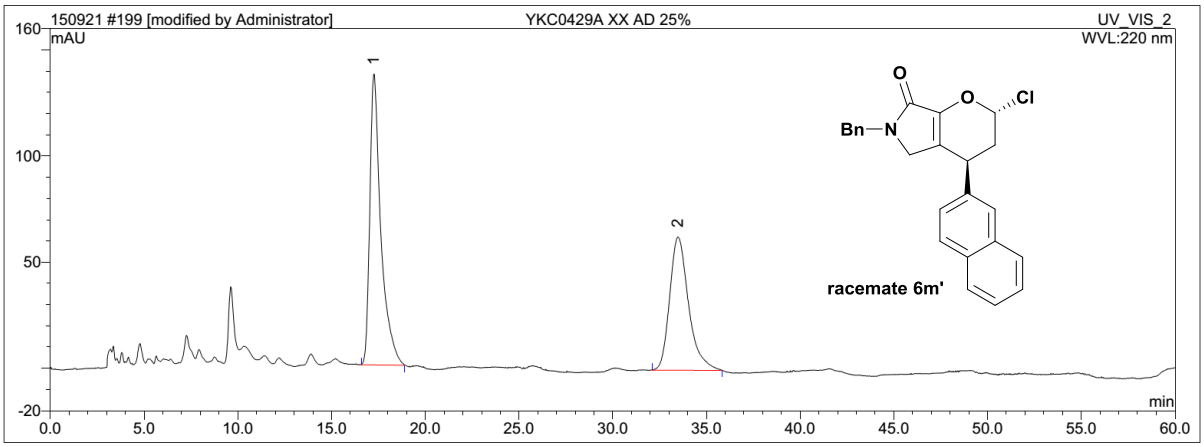

O
O
Cl
Bn—N

6m'


200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

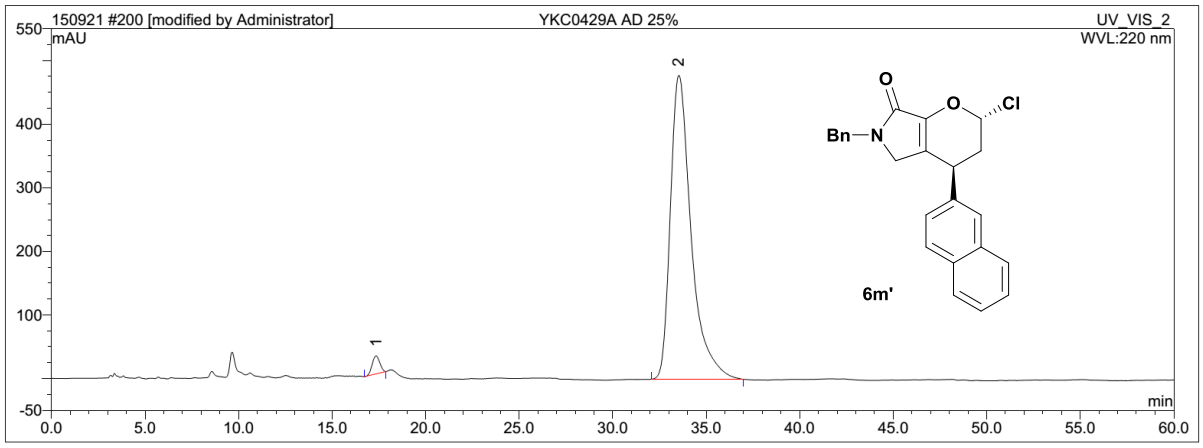
Peak Analysis Report

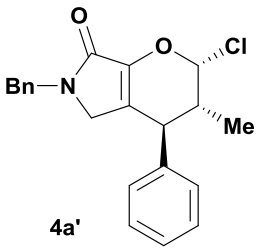
No.	Peak Name	Ret.Time (detected)	Area	Rel.Area	Height	Amount
		min	mAU*min	%	mAU	
1	n.a.	17.26	93.486	56.08	137.216	n.a.
2	n.a.	33.48	73.211	43.92	62.841	n.a.



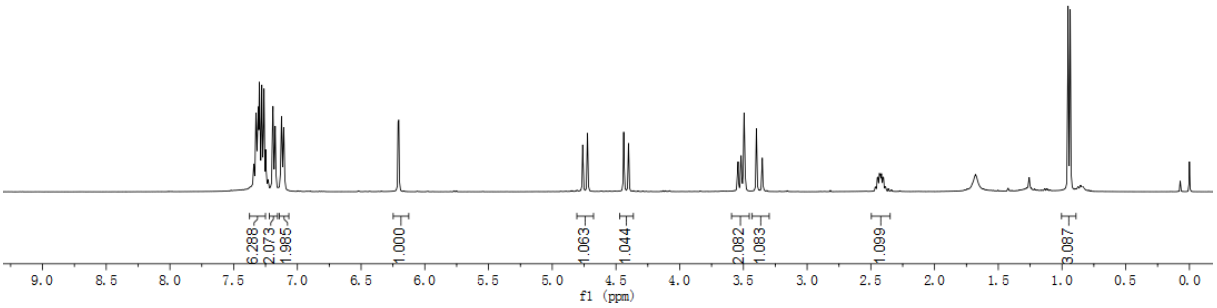
Peak Analysis Report

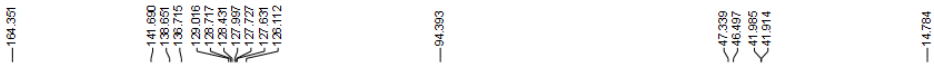
No.	Peak Name	Ret.Time (detected)	Area	Rel.Area	Height	Amount
		min	mAU*min	%	mAU	
1	n.a.	17.36	13.336	2.12	28.206	n.a.
2	n.a.	33.53	615.426	97.88	478.321	n.a.

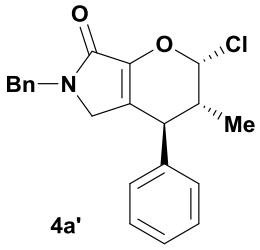



O
O
Cl
Bn—N
Me

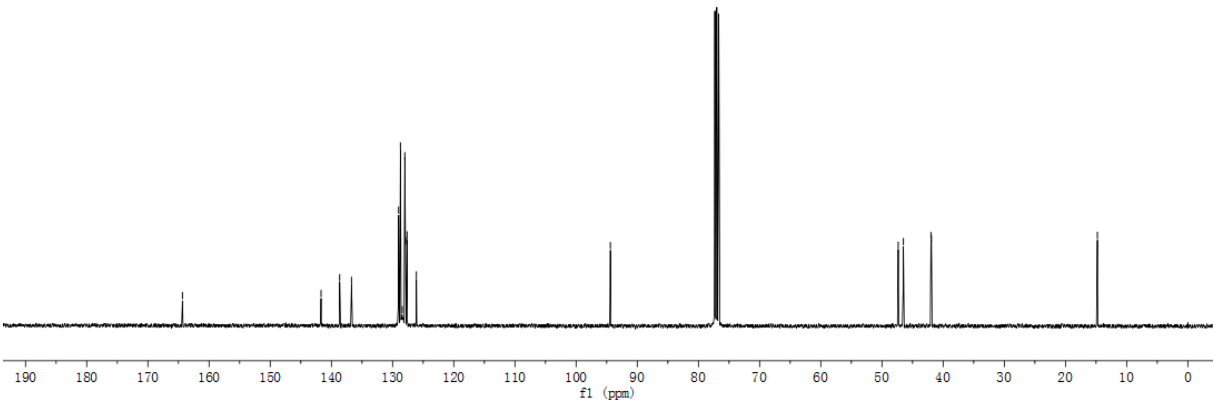
4a'

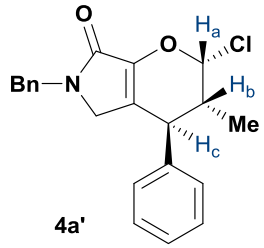

6.288
2.073
1.985
1.000
1.063
1.044
2.082
1.083
1.099
3.087
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


164.351
141.690
138.651
136.715
129.016
128.717
128.431
127.997
127.727
127.631
126.112
94.393
47.339
46.497
41.985
41.914
14.784

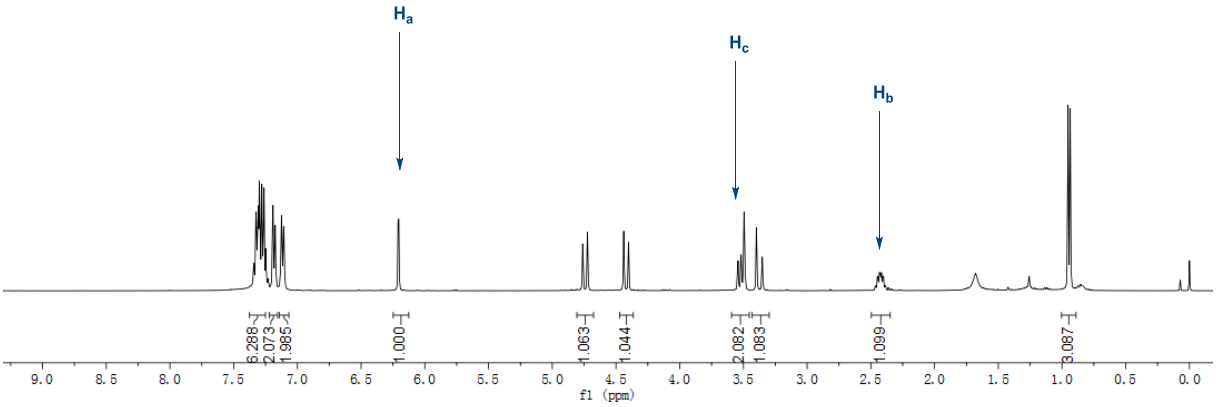

O
O
Cl
Bn—N
Me

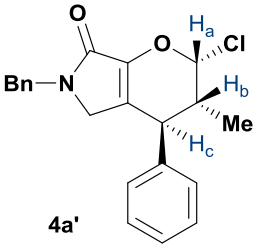
4a'


190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

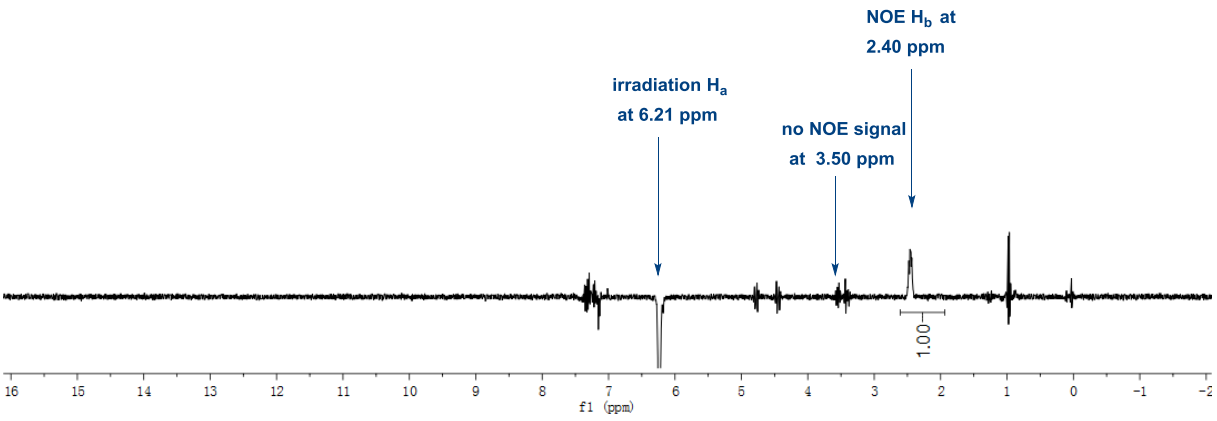

O
O
Ha
Cl
Bn—N
Hb
Me
Hc

4a'


Ha
Hc
Hb
6.288
2.073
1.985
1.000
1.063
1.044
2.082
1.083
1.099
3.087
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

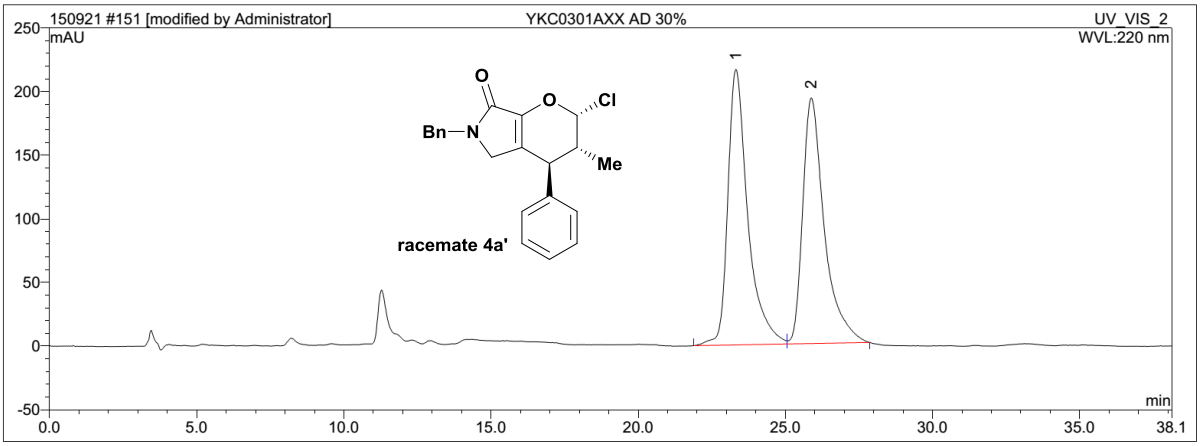

O
O
Ha
Cl
Bn—N
Hb
Me
Hc

4a'


NOE Hb at
2.40 ppm
irradiation Ha
at 6.21 ppm
no NOE signal
at 3.50 ppm
1.00
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2
f1 (ppm)

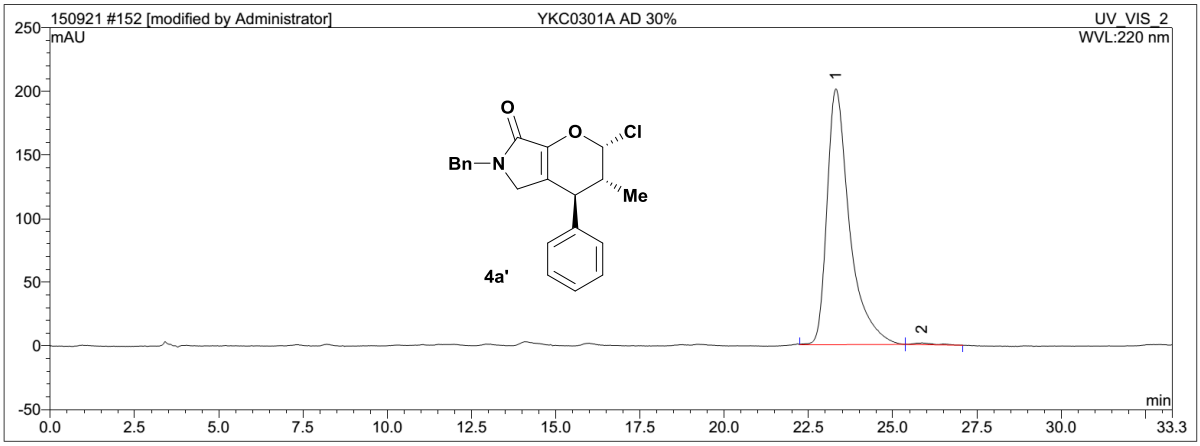
Peak Analysis Report

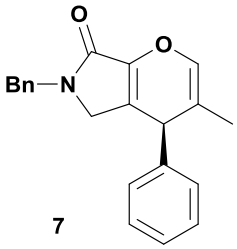
No.	Peak Name	Ret.Time (detected)	Area	Rel.Area	Height	Amount
		min	mAU*min	%	mAU	
1	n.a.	23.33	170.423	50.94	216.712	n.a.
2	n.a.	25.88	164.104	49.06	193.056	n.a.



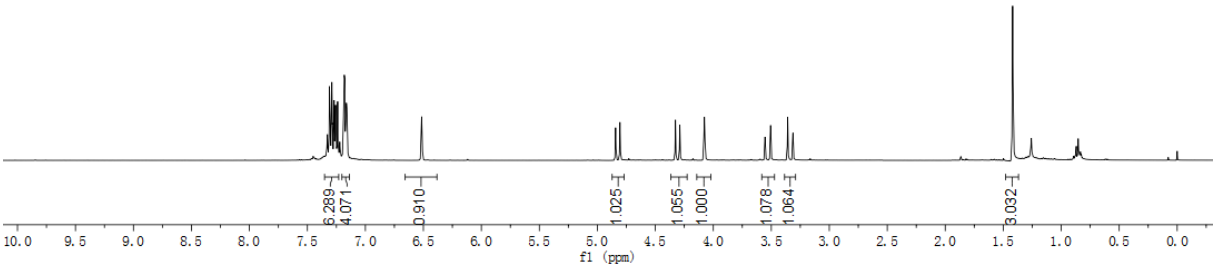
Peak Analysis Report

No.	Peak Name	Ret.Time (detected)	Area	Rel.Area	Height	Amount
		min	mAU*min	%	mAU	
1	n.a.	23.32	156.207	99.41	201.097	n.a.
2	n.a.	25.87	0.920	0.59	1.311	n.a.

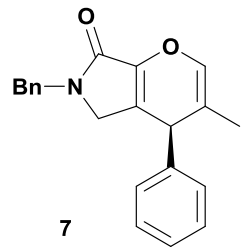



O
O
Bn—N

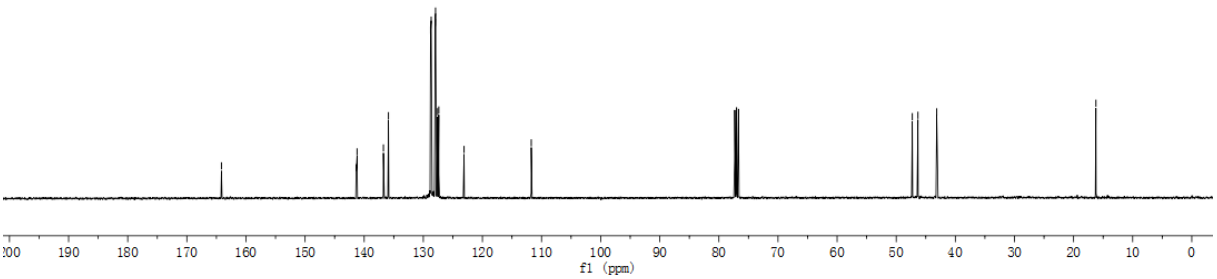
7


6.289
4.071
0.910
1.025
1.055
1.000
1.078
1.064
3.032
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


164.144
141.364
141.204
136.749
135.898
128.775
128.672
127.967
127.931
127.576
127.361
123.110
111.739
47.283
46.328
43.154
16.222

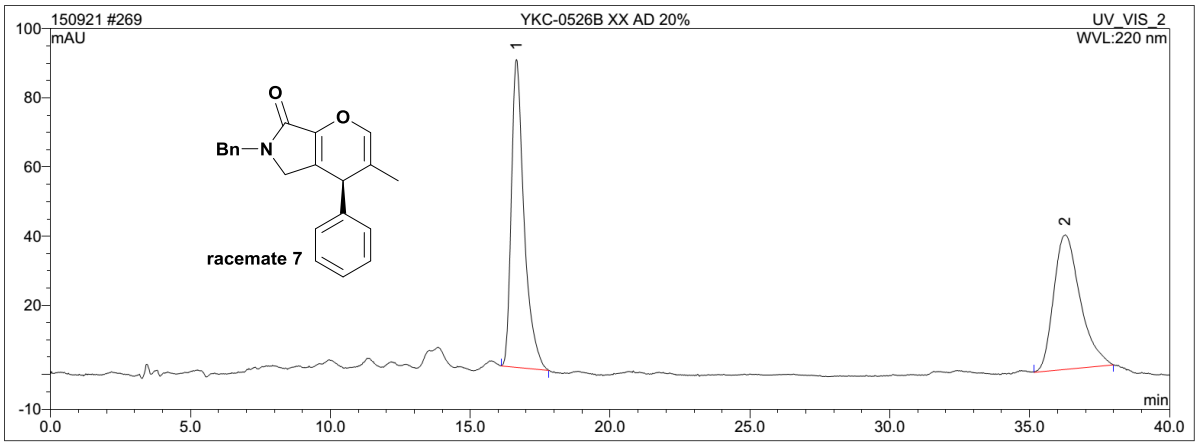

O
O
Bn—N

7


200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

Peak Analysis Report

No.	Peak Name	Ret.Time (detected)	Area	Rel.Area	Height	Amount
		min	mAU*min	%	mAU	
1	n.a.	16.66	46.973	52.38	89.026	n.a.
2	n.a.	36.28	42.711	47.62	38.931	n.a.



Peak Analysis Report

No.	Peak Name	Ret.Time (detected)	Area	Rel.Area	Height	Amount
		min	mAU*min	%	mAU	
1	n.a.	16.47	256.309	99.26	443.999	n.a.
2	n.a.	35.97	1.900	0.74	2.700	n.a.